

Cette configuration avait été envisagée avec une forte présomption suite à l'analyse RMN ¹H et suite à la discussion des différentes configurations possibles des états de transition conduisant aux aziridines esters α -halogénés (El Gadi, 1987).

Le fait que pour Ar¹ = *m*-NO₂Ph, Ar² = Ph, R = CH₃, la configuration *trans* des aryles Ar¹ et Ar² liée à la relation *cis* entre Ar¹ et Cl soit établie sans ambiguïté par ce travail conforte les résultats précédents.

Il est en effet peu probable que la réaction de type Darzens qui permet la synthèse de ces produits puisse conduire à des stéréoisomères uniques de configuration différente selon les imines utilisées.

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Structure of 1-Benzoyl-3-propylthiourea

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Abstract. C₁₁H₁₄N₂OS, *M_r* = 222.1, monoclinic, *P*2₁/*n*, *a* = 10.951 (5), *b* = 20.52 (2), *c* = 5.724 (4) Å, γ = 110.85 (5)°, *V* = 1202 (5) Å³, *Z* = 4, *D_x* = 1.23 g cm⁻³, λ (Mo *K* α) = 0.71069 Å, μ = 2.4 cm⁻¹, *T* = 298 K, *F*(000) = 472, final *R* = 0.065 for 1471 reflections. The crystal structure is built up of C₁₁H₁₄N₂OS units linked into dimers by N–H...S intermolecular hydrogen bonds.

only. In both states the molecule has a structure with an intramolecular hydrogen bond between the H atom of the NH group in position 3 and the O atom of the carbonyl group. In the solid state the 1-(*p*-chlorobenzoyl)-3-fenylthiourea has a dimeric structure (Dago *et al.*, 1988). It is interesting to investigate whether these structural characteristics remain when the aryl group in position 3 is substituted by an aliphatic group.

Introduction. It is known that N-substituted thioureas in general possess a wide spectrum of bioactivity: antituberculose, hypnotic, antiviral and pesticide properties are known (Schroeder, 1955). In particular, some acylthioureas have potent fungicidal action (Macías, Rodríguez & Rivero, 1986). It has been shown that 1-aryloyl-3-arylthioureas in solution (Otazo, Gra & Macías, 1979; Macías, Otazo, Gra & Beletskaya, 1982) and in the solid state (Dago, Simonov, Pobedinskaya, Macías & Martin, 1987, 1988) are in thioamidic form

Experimental. Single crystals of the title compound were obtained by the reaction of propylamine with benzoyl isothiocyanate obtained *in situ* by a published method (Macías, Otazo & Beletskaya, 1982): reaction of benzoyl chloride with ammonium isothiocyanate in acetone. Crystal 0.2 × 0.2 × 0.1 mm. All data collected on Syntex *P*2₁ diffractometer with graphite-monochromatized Mo *K* α radiation. Lattice parameters were refined by least squares utilizing 15 medium-angle reflections (10 ≤ 2 θ ≤ 18°). Data were collected using

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Table 1. Fractional atomic coordinates with equivalent isotropic thermal parameters (\AA^2) for non-hydrogen atoms

$$U_{\text{eq}} = \frac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	U_{eq}
S	0.9715 (1)	0.5826 (1)	-0.1869 (3)	0.067 (1)
O	0.5655 (3)	0.4431 (2)	0.0441 (6)	0.074 (2)
N(1)	0.7888 (3)	0.4843 (2)	0.0461 (1)	0.048 (2)
N(2)	0.7162 (4)	0.5591 (2)	-0.1725 (7)	0.069 (2)
C(1)	0.6692 (4)	0.3831 (2)	0.2829 (7)	0.050 (2)
C(2)	0.5797 (4)	0.3167 (2)	0.2530 (8)	0.057 (2)
C(3)	0.5741 (4)	0.2633 (2)	0.4088 (9)	0.069 (3)
C(4)	0.6583 (5)	0.2779 (2)	0.5986 (9)	0.066 (3)
C(5)	0.7483 (5)	0.3446 (3)	0.6310 (8)	0.068 (3)
C(6)	0.7557 (4)	0.3978 (20)	0.4709 (7)	0.057 (2)
C(7)	0.6677 (3)	0.4387 (2)	0.1162 (8)	0.052 (2)
C(8)	0.8160 (4)	0.5407 (2)	-0.1058 (7)	0.049 (2)
C(9)	0.7291 (5)	0.6219 (3)	-0.3109 (9)	0.079 (3)
C(10)	0.6869 (9)	0.6035 (4)	-0.5514 (11)	0.117 (5)
C(11)	0.6925 (8)	0.6709 (3)	-0.6874 (10)	0.103 (5)

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

C(1)–C(2)	1.376 (5)	C(7)–N(1)	1.383 (4)
C(2)–C(3)	1.398 (7)	C(8)–N(1)	1.393 (5)
C(3)–C(4)	1.387 (7)	C(8)–S	1.678 (4)
C(4)–C(5)	1.386 (6)	C(8)–N(2)	1.332 (7)
C(5)–C(6)	1.405 (7)	N(2)–C(9)	1.477 (7)
C(6)–C(1)	1.394 (6)	C(9)–C(10)	1.459 (9)
C(1)–C(7)	1.492 (6)	C(10)–C(11)	1.569 (11)
C(7)–O	1.226 (5)		
C(2)–C(1)–C(6)	120.0 (4)	S–C(8)–N(2)	124.1 (3)
C(3)–C(2)–C(1)	120.9 (4)	C(8)–N(2)–C(9)	124.5 (4)
C(2)–C(3)–C(4)	119.3 (4)	H(7)–N(2)–C(9)	129 (5)
C(3)–C(4)–C(5)	120.2 (5)	N(2)–C(9)–C(10)	110.9 (5)
C(4)–C(5)–C(6)	120.3 (4)	C(9)–C(10)–C(11)	109.3 (3)
C(1)–C(6)–C(5)	119.2 (4)	N(2)–C(9)–C(10)	110.9 (5)
C(2)–C(1)–C(7)	118.7 (4)		
C(6)–C(1)–C(7)	121.3 (3)		
C(1)–C(7)–O	122.0 (3)		
C(1)–C(7)–N(1)	115.8 (4)		
O–C(7)–N(1)	122.2 (4)		
C(7)–N(1)–C(8)	127.9 (4)		
N(1)–C(8)–S	118.5 (3)		
N(1)–C(8)–N(2)	117.4 (3)		
C(2)–C(1)–C(7)–N(1)	139.3 (4)		
C(8)–N(2)–C(9)–C(10)	105.9 (4)		
N(2)–C(9)–C(10)–C(11)	176.0 (6)		

the $\theta/2\theta$ scanning technique, scan rate $2.0\text{--}2.4^\circ \text{ min}^{-1}$, depending on intensity; space group $P2_1/n$ based on conditions $hk0$, $h+k=2n$ and $00l$, $l=2n$; 1612 independent reflections, $2\theta_{\text{max}} = 50^\circ$, index range ($h\ 0\text{--}13$, $k\ -24\text{--}24$, $l\ 0\text{--}6$); standards measured every 50 reflections, no significant change; correction for Lorentz and polarization but not for absorption; 1471 reflections with $I > 1.96\sigma(I)$ used for structure determination. The structure was solved by direct methods; difference electron density maps revealed positions for all H atoms. Least-squares refinement with anisotropic thermal parameters for the non-H atoms, isotropic for the H atoms, gave $R = 0.065$, $wR = 0.081$ where $w_i = 1.51/[\sigma^2(F_o) + 0.00070F_o^2]$. $(\Delta/\sigma)_{\text{max}} = 0.05$ in

final cycle of refinement, in final difference Fourier map $\Delta\rho$ within $+0.7$ and -0.4 e \AA^{-3} . Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). The program *SHELX76* (Sheldrick, 1976) was used for all calculations.

Discussion. The atomic fractional coordinates and equivalent isotropic thermal parameters for non-H atoms are listed in Table 1.* Bond lengths, bond angles and some selected torsion angles are shown in Table 2. A perspective view of the molecule and the atom-labeling scheme are shown in Fig. 1. The structure of the title compound was shown to be a dimer, formed through two intermolecular hydrogen bonds $\text{N}(1)\text{--H}(6)\cdots\text{S}$ [$\text{N}(1)\cdots\text{S}$, $3.461(5)$, $\text{N}(1)\text{--H}(6)$, $0.95(6)$ and $\text{S}\cdots\text{H}(6)$, $2.55(6)\text{ \AA}$; $\text{N}(1)\text{--H}(6)\cdots\text{S}$, $161(5)^\circ$]. Each monomer shows an intramolecular hydrogen bond $\text{N}(2)\text{--H}(7)\cdots\text{O}$ [$\text{N}(2)\cdots\text{O}$, $2.672(5)$, $\text{N}(2)\text{--H}(7)$, $0.98(6)$ and $\text{O}\cdots\text{H}(7)$, $1.80(6)\text{ \AA}$; $\text{N}(2)\text{--H}(7)\cdots\text{O}$, $146(5)^\circ$]. The bond lengths and angles given here agree with those reported by Donohue (1968, 1969) for $\text{N}\text{--H}\cdots\text{O}$ and $\text{N}\text{--H}\cdots\text{S}$ interactions.

The present compound, as well as the recently published 1-(*p*-chlorobenzoyl)-3-fenylthiourea (Dago *et al.*, 1988), is characterized by the formation of dimers. In both compounds the values of the bond distances C–S and C–N indicate that the compounds are stabilized by resonance (Hyun, 1984). The fact that the C(8)–N(2) distance is $1.332(7)\text{ \AA}$ in the present compound and $1.346(6)\text{ \AA}$ in 1-(*p*-chlorobenzoyl)-3-fenylthiourea indicates that resonance form II is

*Lists of structure factors, anisotropic thermal parameters, H-atom parameters, and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51706 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

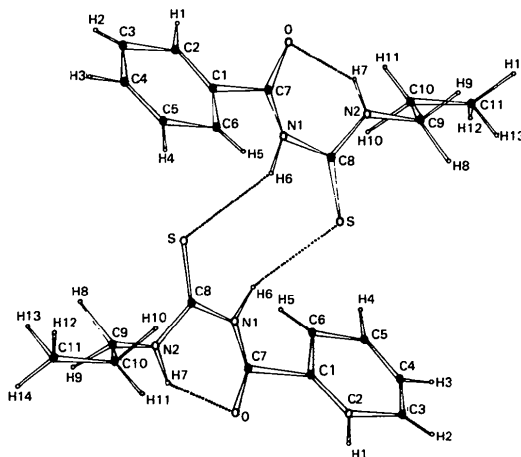
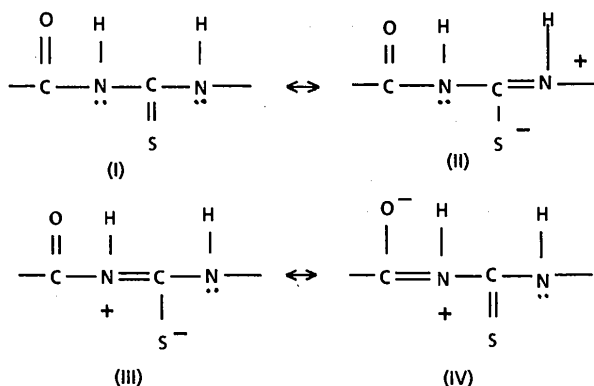


Fig. 1. Perspective view of the molecule, with the numbering scheme. The hydrogen bonds are indicated by dotted lines.

contributing since these two C—N distances are shorter than the others.



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Structures of Two 8-(2,3-*O*-Isopropylidene- β -D-ribofuranosylamino)pyrimido-[5,4-*d*]pyrimidines

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Abstract. 2,4,6-Trichloro-8-(2,3-*O*-isopropylidene- β -D-ribofuranosylamino)pyrimido[5,4-*d*]pyrimidine (1) ethanol solvate, $C_{14}H_{14}Cl_3N_5O_4 \cdot C_2H_5OH$, $M_r = 468.72$, triclinic, $P1$, $a = 5.1578$ (6), $b = 9.3348$ (15), $c = 11.115$ (2) Å, $\alpha = 106.08$ (2), $\beta = 96.713$ (9), $\gamma = 93.731$ (12)°, $V = 508.02$ (15) Å³, $Z = 1$, $D_x = 1.532$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu = 45.244$ cm⁻¹, $F(000) = 242$, $T = 295$ K, $R = 0.0276$ for 2045 reflections ($F \geq 4\sigma_F$). 4-Amino-6-chloro-8-(2,3-*O*-isopropylidene- β -D-ribofuranosylamino)pyrimido[5,4-*d*]pyrimidine (2), $C_{14}H_{17}ClN_6O_4$, $M_r = 368.78$, monoclinic, $P2_1$, $a = 5.0427$ (8), $b = 9.994$ (2), $c = 15.713$ (5) Å, $\beta = 99.13$ (2)°, $V = 781.8$ (4) Å³, $Z = 2$, $D_x = 1.566$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu = 25.084$ cm⁻¹, $F(000) = 384$, $T = 295$ K, $R = 0.0365$ for 1413 reflections ($F \geq 4\sigma_F$). The two structures are nearly identical despite the presence of ethanol in the lattice of (1). Each compound exhibits an intramolecular hydrogen bond from the bridging N atom

(N11) to O5' [$d(\text{H}\cdots\text{O}) = 2.25$ (7), $d(\text{N}\cdots\text{O}) = 2.841$ (4) Å, $\angle(\text{N}-\text{H}\cdots\text{O}) = 133$ (6)° for (1); $d(\text{H}\cdots\text{O}) = 2.37$ (5), $d(\text{N}\cdots\text{O}) = 3.034$ (5) Å, $\angle(\text{N}-\text{H}\cdots\text{O}) = 134$ (4)° for (2)]. The sugar conformations and puckering parameters are 4T_0 ($C_{4'}$ -endo), $P = 243.1^\circ$ and $\tau_m = 31.8^\circ$ for (1) and 3T ($C_{4'}$ -endo- $C_{3'}$ -exo), $P = 214.6^\circ$ and $\tau_m = 30.2^\circ$ for (2). The pyrimidopyrimidine rings are nearly planar [r.m.s. deviations: 0.0180 (9) Å for (1) and 0.0215 (6) Å for (2)]. The dihedral angles between the fused pyrimidine rings are 1.58 (11)° for (1) and 2.09 (13)° for (2).

Introduction. The exocyclic aminonucleoside 4-amino-8-(β -D-ribofuranosylamino)pyrimido[5,4-*d*]pyrimidine [ARPP (3)] was first reported as the product of the rearrangement of the purine ring in the aqueous ammonia adduct of 9-(2,3,5-tri-*O*-acetyl- β -D-ribofuranosyl)purine-6-carbonitrile (Berman, Rousseau, Mancuso, Kreishman & Robins, 1973). ARPP possesses broad-spectrum antiviral activity (Westover *et al.*, 1981) and immunosuppressive activity and inhibits

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