C10	0.40803 (14)	0.0483 (9)	0.5692 (3)	0.028 (2)
C11	0.40125 (13)	0.2583 (8)	0.6226 (4)	0.045 (3)
C12	0.41792 (15)	0.3057 (8)	0.7409 (4)	0.043 (3)
C13	0.44138 (13)	0.1431 (11)	0.8058 (3)	0.050 (3)
C14	0.44816 (14)	−0.0669 (10)	0.7523 (5)	0.053 (3)
C15	0.4315 (2)	−0.1143 (7)	0.6341 (5)	0.053 (3)
C16	0.2703 (2)	0.4061 (16)	0.2554 (6)	0.028 (2)
C17	0.23979 (12)	0.3290 (9)	0.3233 (4)	0.028 (2)
C18	0.22739 (13)	0.4785 (8)	0.4051 (4)	0.040 (2)
C19	0.19832 (13)	0.4124 (9)	0.4624 (4)	0.044 (2)
C20	0.18165 (11)	0.1967 (10)	0.4379 (4)	0.044 (3)
C21	0.19405 (13)	0.0472 (8)	0.3562 (4)	0.038 (2)
C22	0.22312 (13)	0.1133 (8)	0.2989 (4)	0.042 (2)

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

C1—O1	1.395 (10)	O4—C16	1.414 (9)
C1—O5	1.402 (9)	C5—O5	1.450 (8)
C1—C2	1.547 (10)	C5—C6	1.489 (11)
O1—C7	1.427 (9)	C6—O6	1.439 (8)
C2—O2	1.459 (9)	O6—C16	1.417 (8)
C2—C3	1.527 (10)	S1—O7	1.409 (6)
O2—S1	1.548 (5)	S1—O8	1.409 (7)
C3—O3	1.458 (9)	S1—C8	1.812 (11)
C3—C4	1.519 (10)	C8—F3	1.250 (10)
O3—C9	1.378 (11)	C8—F1	1.328 (11)
C4—O4	1.438 (8)	C8—F2	1.360 (12)
C4—C5	1.549 (10)	C9—O9	1.189 (11)
O1—C1—O5	113.1 (8)	C6—C5—C4	107.1 (7)
O1—C1—C2	104.8 (7)	C1—O5—C5	113.2 (7)
O5—C1—C2	111.3 (7)	O6—C6—C5	108.7 (7)
C1—O1—C7	112.4 (7)	C16—O6—C6	110.8 (6)
O2—C2—C3	107.7 (6)	O7—S1—O8	123.0 (4)
O2—C2—C1	108.9 (7)	O7—S1—O2	111.8 (4)
C3—C2—C1	109.8 (7)	O8—S1—O2	107.4 (4)
C2—O2—S1	119.8 (5)	O7—S1—C8	108.2 (5)
O3—C3—C4	107.9 (7)	O8—S1—C8	103.4 (5)
O3—C3—C2	109.2 (7)	O2—S1—C8	100.4 (4)
C4—C3—C2	109.0 (7)	F3—C8—F1	111.2 (9)
C9—O3—C3	115.4 (7)	F3—C8—F2	106.8 (9)
O4—C4—C3	109.6 (6)	F1—C8—F2	103.9 (10)
O4—C4—C5	104.2 (6)	F3—C8—S1	115.1 (9)
C3—C4—C5	108.7 (7)	F1—C8—S1	109.7 (7)
C16—O4—C4	110.0 (6)	F2—C8—S1	109.5 (7)
O5—C5—C6	110.8 (7)	O9—C9—O3	124.0 (8)
O5—C5—C4	109.3 (6)	O6—C16—O4	111.3 (6)

An area-detector system was used. Cell dimensions were refined from 250 reflections selected from 2 regions 90° apart and 5° wide at $\kappa = 0°$. The phenyl groups were refined as rigid groups with individual isotropic atomic displacement parameters for the C atoms and $U(H) = 1.5U(C)$.

Although there was no intention to derive the absolute configuration from the relatively poor quality data obtained from crystals which were far from ideal, the automatic Flack (1983) calculation in *SHELXL*93 (Sheldrick, 1993) confirmed the expected configuration.

The high value of $R_{\rm int}$ reflects the poor quality and weak diffraction of the available crystals.

Data collection: *MADNESS* (Enraf–Nonius, 1990). Cell refinement: *MADNESS*. Data reduction: *MADNESS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *PLATON*92 (Spek, 1992*a*), *PLUTON*92 (Spek, 1992*b*). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: LI1139). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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6-Amino-4-methoxy-2-methylthiopyrimidine

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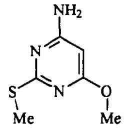
Abstract

Molecules of the title compound, $C_6H_9N_3OS$, form base-paired N—H···N hydrogen-bonded dimers in the solid state, with N···N 3.060 (3) Å.

Comment

Derivatives of 6-aminopyrimidines are of interest both because of their biological activity and their use as intermediates in the synthesis of other nucleoside derivatives (Low, Ferguson, Cobo, Nogueras & Sánchez, 1996; Low, Ferguson, Cobo, Melguizo, Nogueras & Sánchez, 1996*a*,*b*; Cobo, Low, Ferguson, Melguizo, Sánchez & Nogueras, 1996).

In our preparation of novel compounds with potential antiviral activity, we synthesized 6-amino-4-methoxy-2-methylthiopyrimidine, (I). This compound has been used to prepare N-coordinated Hg^{II} dimeric and C-5 organomercurial species, which are currently under further examination (López, Godino, Gutiérrez, Arranz & Moreno, 1995).



(I)

Molecules of (I) form base-paired hydrogen-bonded dimers in the solid state. Fig. 1 shows an *ORTEP*II (Johnson, 1976) view of the dimer of (I) with our numbering scheme. Self base-pairing between nucleobases is a very common feature in the crystal structures of compounds in which an amine group is attached to a ring C atom adjacent to an unsubstituted ring N atom where neither group is sterically hindered. This is well demonstrated in the structure of the triclinic form of 5-bromo-2′-deoxycytidine (Low, Tollin & Young, 1981) in which four conformationally distinct molecules form two self base-paired dimers. In that case, there are four individual N4···N3 hydrogen bonds with an average bond length of 2.96 (3) Å. In (I), there is a single hydrogen bond of length 3.060 (3) Å. The other amino H atom appears to take no part in hydrogen bonding.

The molecular dimensions (Table 2) are unexceptional and comparable with those of related structures of nucleoside derivatives (see references listed above). The six-membered ring is essentially planar with a maximum deviation of 0.010 (4) Å and the methoxy and methylthio groups are almost coplanar with the pyrimidine ring [torsion angles N3—C4—O4—C41 5.5 (4) and N3—C2—S2—C21 1.4 (2)°].

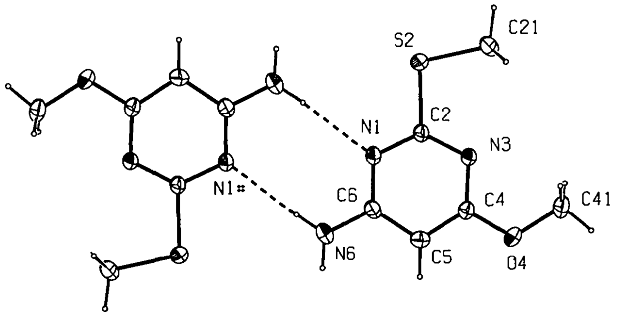


Fig. 1. A view of the dimer of (I) with our numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Atom N1# and all other atoms in the same molecule are at equivalent position 2 − *x*, 1 − *y*, 1 − *z*.

Experimental

The title compound was prepared as described by Bretschneider, Klotzer & Spiteller (1961); recrystallization from methanol produced colourless needles.

Crystal data

$C_6H_9N_3OS$
$M_r = 171.22$
Monoclinic
$P2_1/n$
$a = 7.519\ (4)$ Å
$b = 14.202\ (3)$ Å
$c = 7.885\ (3)$ Å
$\beta = 109.26\ (4)°$
$V = 794.9\ (5)$ Å^3
$Z = 4$
$D_x = 1.431$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 9.30–13.25°$
$\mu = 0.351$ mm^{-1}
$T = 294\ (1)$ K
Plate
0.43 × 0.43 × 0.10 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
1832 measured reflections
1709 independent reflections
1211 observed reflections [$I > 2\sigma(I)$]

$R_{int} = 0.042$
$\theta_{max} = 26.89°$
$h = -9 \rightarrow 9$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 10$
3 standard reflections frequency: 120 min intensity variation: 1.8%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0636$
$wR(F^2) = 0.1636$
$S = 1.003$
1709 reflections
103 parameters
H atoms riding (C—H 0.93–0.98, N—H 0.86 Å)
$w = 1/[\sigma^2(F_o^2) + (0.1214P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$

$\Delta\rho_{max} = 0.969$ e Å^{-3}
$\Delta\rho_{min} = -0.514$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0105 (60)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	U_{eq}
N1	0.8816 (3)	0.49733 (15)	0.2535 (3)	0.0378 (6)
C2	0.7805 (4)	0.5295 (2)	0.0917 (3)	0.0318 (5)
S2	0.70902 (10)	0.64720 (5)	0.09177 (9)	0.0418 (3)

C21	0.5789 (4)	0.6643 (2)	−0.1409 (4)	0.0465 (7)
N3	0.7313 (3)	0.48445 (15)	−0.0650 (3)	0.0346 (5)
C4	0.7909 (4)	0.3954 (2)	−0.0537 (3)	0.0363 (6)
O4	0.7428 (3)	0.34416 (13)	−0.2055 (3)	0.0488 (6)
C41	0.6450 (5)	0.3919 (2)	−0.3714 (4)	0.0560 (9)
C5	0.8979 (4)	0.3527 (2)	0.1026 (4)	0.0408 (6)
C6	0.9429 (4)	0.4081 (2)	0.2578 (3)	0.0376 (6)
N6	1.0505 (4)	0.3744 (2)	0.4193 (3)	0.0569 (8)

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

N1—C2	1.332 (3)	C4—O4	1.344 (3)
N1—C6	1.345 (3)	C4—C5	1.371 (4)
C2—N3	1.331 (3)	O4—C41	1.441 (4)
C2—S2	1.756 (3)	C5—C6	1.399 (4)
S2—C21	1.788 (3)	C6—N6	1.351 (3)
N3—C4	1.335 (3)		
C2—N1—C6	115.4 (2)	N3—C4—C5	124.3 (2)
N3—C2—N1	128.3 (2)	O4—C4—C5	117.8 (3)
N3—C2—S2	117.8 (2)	C4—O4—C41	117.4 (2)
N1—C2—S2	113.8 (2)	C4—C5—C6	116.0 (2)
C2—S2—C21	101.14 (12)	N1—C6—N6	116.8 (2)
C2—N3—C4	114.1 (2)	N1—C6—C5	121.7 (2)
N3—C4—O4	117.9 (2)	N6—C6—C5	121.4 (3)
C6—N1—C2—N3	0.7 (4)	N3—C4—O4—C41	5.5 (4)
C6—N1—C2—S2	−178.4 (2)	C5—C4—O4—C41	−174.6 (3)
N3—C2—S2—C21	1.4 (2)	N3—C4—C5—C6	0.7 (4)
N1—C2—S2—C21	−179.4 (2)	O4—C4—C5—C6	−179.2 (2)
N1—C2—N3—C4	1.0 (4)	C2—N1—C6—N6	178.1 (3)
S2—C2—N3—C4	−180.0 (2)	C2—N1—C6—C5	−1.8 (4)
C2—N3—C4—O4	178.2 (2)	C4—C5—C6—N1	1.1 (4)
C2—N3—C4—C5	−1.7 (4)	C4—C5—C6—N6	−178.7 (3)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N6—H6A···N1^{i}	0.86	2.20	3.060 (3)	175.2

Symmetry code: (i) $2 - x, 1 - y, 1 - z$.

The diagram was prepared using *ORTEP*II (Johnson, 1976) as implemented in *PLATON* (Spek, 1995*a*). Examination of the structure with *PLATON* showed that there were no solvent accessible voids in the crystal lattice.

Data collection: *CAD*-4/*PC Software* (Enraf–Nonius 1992). Cell refinement: *SET*4 and *CELDIM* in *CAD*-4/*PC Software*. Data reduction: *DATRD*2 in *NRCVAX*94 (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SOLVER* in *NRCVAX*. Program(s) used to refine structure: *NRCVAX*94; *SHELXL*93 (Sheldrick, 1993). Software used to prepare material for publication: *NRCVAX*94; *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1323). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tris(4-methoxyphenyl)methanol

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Abstract

Tris(4-methoxyphenyl)methanol, $C_{22}H_{22}O_4$, crystallizes in space group $P2_1$ with two molecules in the asymmetric unit. The molecules are linked into dimers by a weak O—H···O hydrogen bond [O···O 3.035 (3) Å].

Comment

Substituted diphenylmethanols, *R*CPh$_2$OH, exhibit a very wide range of intermolecular aggregation patterns in the solid state. When the *R* group carries no functionality, the aggregation usually depends upon O—H···O hydrogen-bond formation and examples are now known of cyclic (OH)$_2$ dimers [*R* = $(C_5H_5)Fe(C_5H_4)$; Ferguson, Gallagher, Glidewell & Zakaria, 1993], cyclic (OH)$_3$ trimers (*R* = Me$_2$CH; Ferguson, Carroll, Glidewell, Zakaria & Lough, 1995), cyclic (OH)$_4$ tetramers (*R* = CH$_3$; Sultanov, Shnulin & Mamedov, 1985), tetrahedral tetramers (*R* = Ph; Ferguson, Gallagher, Glidewell, Low & Scrimgeour, 1992), cyclic (OH)$_6$ hexamers (Ferguson, Carroll, Glidewell, Zakaria & Lough, 1995) and extended chains (*R* = H; Ferguson, Carroll, Glidewell,

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