Table 2. *Intramolecular distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

S1—C4	1·752 (3)	S1—O1	1·576 (2)
S1—O2	1·419 (3)	S1—O3	1·414 (3)
C1—C1^{i}	1·380 (5)	C1—C2	1·182 (5)
C2—C3	1·453 (5)	C3—O1	1·455 (5)
C3—H31	0·99 (6)	C3—H32	0·96 (4)
C4—C5	1·379 (4)	C4—C9	1·392 (4)
C5—C6	1·378 (5)	C5—H51	0·89 (3)
C6—C7	1·392 (5)	C6—C11	1·502 (6)
C7—C8	1·378 (5)	C7—H71	0·95 (3)
C8—C9	1·379 (5)	C8—C10	1·513 (7)
C9—H91	0·92 (3)	C10—H101	1·05 (6)
C10—H102	0·75 (6)	C10—H103	0·91 (6)
C11—H111	0·98 (6)	C11—H112	0·95 (5)
C11—H113	0·96 (6)		
O1—S1—C4	103·56 (12)	O2—S1—C4	109·48 (14)
O3—S1—C4	110·74 (14)	C5—C4—S1	118·97 (23)
C9—C4—S1	119·46 (23)	O2—S1—O1	108·95 (13)
O3—S1—O1	103·19 (13)	C3—O1—S1	117·03 (21)
O3—S1—O2	119·56 (14)	C2—C1—C1^{i}	178·99 (40)
C3—C2—C1	177·24 (40)	O1—C3—C2	107·25 (32)
H31—C3—C2	114·9 (30)	H32—C3—C2	112·3 (27)
H31—C3—O1	95·8 (33)	H32—C3—O1	109·2 (26)
H32—C3—H31	115·7 (42)	C9—C4—C5	121·41 (29)
C6—C5—C4	120·10 (30)	H51—C5—C4	117·2 (19)
C8—C9—C4	119·38 (30)	H91—C9—C4	115·5 (20)
H51—C5—C6	122·7 (19)	C7—C6—C5	117·57 (31)
C11—C6—C5	121·39 (34)	C11—C6—C7	121·01 (35)
C8—C7—C6	123·31 (33)	H71—C7—C6	116·5 (20)
H111—C11—C6	109·9 (33)	H112—C11—C6	112·0 (28)
H113—C11—C6	107·5 (32)	H71—C7—C8	120·1 (20)
C9—C8—C7	118·23 (32)	C10—C8—C7	121·42 (38)
C10—C8—C9	120·35 (37)	H91—C9—C8	124·8 (20)
H101—C10—C8	106·9 (35)	H102—C10—C8	118·3 (47)
H103—C10—C8	108·5 (32)	H102—C10—H101	108·9 (57)
H103—C10—H101	100·3 (48)	H103—C10—H102	112·4 (61)
H112—C11—H111	96·3 (40)	H113—C11—H111	126·5 (47)
H113—C11—H112	103·6 (42)		

Symmetry code: (i) 1·0 − *x*, −*y*, 1·0 − *z*.

acetylenes was by Bloor, Ando, Norman, Motevalli, Hursthouse, Milburn, Werninck & Blair (1986). In the present structure the molecules pack together along *y* with a 5·54 Å repeat distance. However, the crucial distance for 1,4-*trans* polymerization is between C2···C2 (at 1 − *x*, 1 − *y*, 1 − *z*), and this is 5·62 Å. The angle of inclination between the diacetylene axis and the stacking direction (the *b* axis) is 71·6°. These parameters are outside the normally accepted limits for facile solid-state polymerization according to the solid-state criterion (Schmidt, 1967) and the least-motion criterion (Baughman, 1974).

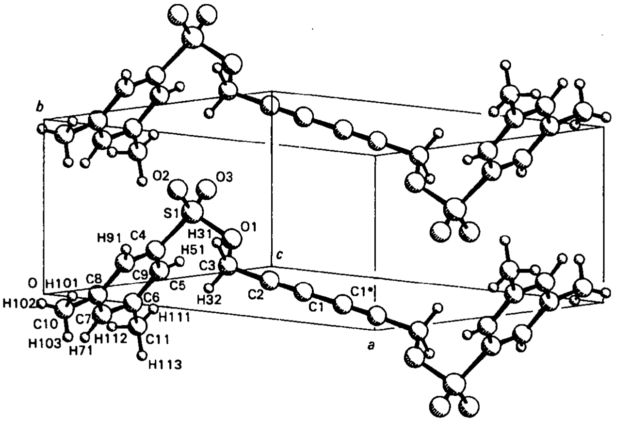

Fig. 1. View to show stacking of molecules in the *y* direction and to indicate the atomic labelling scheme.

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Structure of 7-Amino-2-chloro[1,3]thiazolo[4,5-*d*]pyrimidine, an Adenine Analog

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Abstract. $C_5H_3ClN_4S$, $M_r = 186{\cdot}62$, orthorhombic, *Pccn*, $a = 29{\cdot}874\,(6)$, $b = 7{\cdot}1932\,(9)$, $c = 12{\cdot}9913\,(14)$ Å, $V = 2791{\cdot}7\,(7)$ Å^3, $Z = 16$, $D_x = 1{\cdot}776$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54178$ Å, $\mu = 71{\cdot}31$ cm^{-1}, $F(000) = 1504$, $T = 295$ K, $R = 0{\cdot}0389$ for 2101 reflections ($F \geq 4\sigma_F$). The asymmetric unit is

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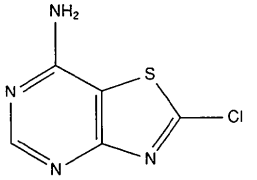
a dimer of independent molecules A and B formed through reciprocated N10—H102···N6 hydrogen bonds [d(N···N) = 3·063 (4), 2·956 (4) Å; d(H··N) = 2·25 (4), 2·15 (3) Å; ∠N—H···N = 170 (4), 175 (3)° for $A \rightarrow B$ and $B \rightarrow A$, respectively]. The dihedral angle between the essentially planar thiazolopyrimidine rings is 6·94 (6)°. The thiazole rings are planar [r.m.s.d.'s: A, 0·0017 (11); B, 0·002 (2) Å] while the pyrimidine rings are slightly nonplanar [r.m.s.d.'s: A, 0·0045 (12); B, 0·0047 (12) Å]; the dihedral angles between thiazole and pyrimidine rings in each molecule are 0·82 (10) and 0·42 (9)° for A and B, respectively. All C—S bond distances are equivalent [1·728 (3) and 1·726 (3) Å in A; 1·733 (3) and 1·718 (3) Å in B]. The C—S—C angles are 86·94 (14) and 87·29 (14)° for A and B. All bond lengths and angles between molecules are equivalent within experimental error. Each independent molecule interacts with a symmetry-related molecule *via* an N10—H101···N4 hydrogen bond [d(N···N) = 3·038 (4), 3·100 (4) Å; d(H··N) = 2·30 (4), 2·32 (4) Å; ∠(N—H··N) = 158 (4), 172 (3)° for A and B, respectively].

Experimental. The title compound (1) was synthesized by the procedure of Nagahara, Anderson, Kini, Dalley, Larson, Smee, Sharma, Jolley, Robins & Cottam (1989). A hot aqueous solution of (1) was slowly cooled to give faint-amber rectangular crystalline plates. The data collection and refinement are summarized in Table 1.

Table 1. *Summary of data collection and structure refinement for* (1)

(A) Data collection (295 K)[a,b]	
Mode	ω–2θ scan
Scan range (°)	$0·80 + 0·15 \tan \theta$
Background	Scan 0·25 times scan range before and after scan
Scan rate (° min^{-1})	1·4–8·3
Exposure time (h)	36·2
Stability correction range on I	1·000–1·016
2θ range (°)	3·0–152·0
Range in hkl, min.	0,0,0
max.	37,9,16
Total reflections, measured, unique	2908, 2908
Crystal dimensions (mm)	0·34 × 0·145 × 0·03
Crystal volume (mm^3)	0·00148
Crystal faces	{100};{010};{001}
Transmission-factor range	0·291–0·810
(B) Structure refinement[c]	
Reflections used ($F \geq 4\sigma_F$)	2101
No. of variables	224
Extinction parameter	7 (2) × 10^{-8}
Goodness of fit, S	1·465
R, wR	0·0389, 0·0495
R for all data	0·0693
Max. Δ/σ	0·0034
Max., min. density in ΔF map (e Å^{-3})	0·29, −0·31

Notes: (a) Unit-cell parameters were obtained by least-squares refinement of the setting angles of 25 reflections with $46·5 < 2\theta < 57·0°$. (b) Enraf–Nonius CAD-4 diffractometer with a graphite monochromator was used. Crystal and instrument stability were monitored by remeasurement of three check reflections (227, 517, 541) every 2 h. A linear fit of the intensities of these reflections was used to correct the data. (c) Function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w^{-1} = (\sigma_F^2 + 0·0004F^2)$. $\sigma_F = F\sigma_I/2I$; $\sigma_I = (N_{pk} + N_{bg1} + N_{bg2})^{1/2}$.



(1)

All non-H atomic coordinates were obtained by direct methods (*MULTAN*82; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982); the six H-atom positions were obtained from a difference map calculated at $R = 0·048$ as peaks of density 0·28–0·66 e Å^{-3}. All atomic positions, anisotropic thermal parameters for non-H atoms and isotropic thermal parameters for H atoms were varied by a full-matrix least-squares refinement procedure (*SHELX*76; Sheldrick, 1976). Scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974) except those for H which were taken from Stewart, Davidson & Simpson (1965). Data were reduced with *SDP-Plus* (Frenz, 1985) which included Lorentz, polarization, decay and absorption corrections. Least-squares planes were calculated with program *PLANES* (Cordes, 1983); thermal-ellipsoid plots produced with *ORTEP*II (Johnson, 1976). Parameter, geometry and structure-factor-amplitude tables were prepared with programs *FUER* and *LISTFC* (Larson, 1980).

Atomic coordinates are listed in Table 2;* bond lengths and bond angles are given in Table 3. Fig. 1 is a perspective drawing of the asymmetric unit illustrating atom labeling; Fig. 2 illustrates the packing of the hydrogen-bonded dimeric units.

Related literature. The synthesis of the title compound was first reported by Nagahara *et al.* (1989). In that report, the crystal structure of 5-amino-3-β-D-ribofuranosyl-7(6*H*)-thioxothiazolo[4,5-*d*]pyrimidin-2(3*H*)-one, a 6-thioguanosine analog, was presented. Recently, we reported the structure of the 8-aminoguanine analog, 2,5-diamino[1,3]thiazolo-[4,5-*d*]pyrimidin-7(6*H*)-one (Larson, Anderson, Cottam & Robins, 1989), and in the following paper

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* Tables of anisotropic thermal parameters, torsion angles, least-squares planes and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52061 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

we report the structure of 5,7-dichloro-2-(*N*-methylanilino)[1,3]thiazolo[4,5-*d*]pyrimidine (Larson, Cottam & Robins, 1989). These four reports constitute the only structural determinations of the thiazolo[4,5-*d*]pyrimidine ring system reported to date (Cambridge Structural Database, 1989).

Table 2. *Positional and isotropic thermal parameters for all atoms in* (1)

For non-H atoms, U is $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\,\mathbf{A}_{ij}$, where $\mathbf{A}_{ij}$ is the dot product of the ith and jth direct-space unit-cell vectors.

	x	*y*	*z*	U/U_{eq} (Å^2)
S1*A*	0·51423 (2)	0·69766 (13)	0·33413 (6)	0·0419 (3)
C2*A*	0·54392 (10)	0·6815 (5)	0·4479 (2)	0·0388 (9)
N3*A*	0·52435 (8)	0·7233 (4)	0·5336 (2)	0·0395 (8)
N4*A*	0·45224 (9)	0·8258 (4)	0·5862 (2)	0·0424 (9)
C5*A*	0·41222 (11)	0·8741 (5)	0·5511 (2)	0·0431 (10)
N6*A*	0·39736 (8)	0·8773 (4)	0·4536 (2)	0·0407 (8)
C7*A*	0·42631 (10)	0·8259 (4)	0·3785 (2)	0·0354 (9)
C8*A*	0·46941 (9)	0·7711 (4)	0·4075 (2)	0·0340 (9)
C9*A*	0·48096 (10)	0·7756 (5)	0·5112 (2)	0·0364 (9)
Cl*A*	0·59893 (3)	0·6134 (2)	0·44215 (7)	0·0551 (3)
N10*A*	0·41209 (10)	0·8324 (5)	0·2823 (2)	0·0463 (10)
H5*A*	0·3884 (10)	0·909 (4)	0·596 (2)	0·026 (8)
H10*A*1	0·4282 (12)	0·788 (6)	0·242 (3)	0·052 (12)
H10*A*2	0·3873 (14)	0·867 (6)	0·269 (3)	0·058 (12)
S1*B*	0·69622 (3)	0·55915 (13)	0·61253 (6)	0·0429 (2)
C2*B*	0·66434 (10)	0·5920 (5)	0·7225 (2)	0·0415 (10)
N3*B*	0·68313 (9)	0·5696 (4)	0·8114 (2)	0·0408 (8)
N4*B*	0·75532 (9)	0·4862 (4)	0·8744 (2)	0·0427 (8)
C5*B*	0·79630 (11)	0·4399 (5)	0·8447 (2)	0·0429 (10)
N6*B*	0·81300 (8)	0·4256 (4)	0·7491 (2)	0·0403 (8)
C7*B*	0·78512 (10)	0·4605 (4)	0·6698 (2)	0·0347 (9)
C8*B*	0·74037 (10)	0·5080 (4)	0·6924 (2)	0·0351 (9)
C9*B*	0·72728 (10)	0·5200 (4)	0·7950 (2)	0·0357 (9)
Cl*B*	0·60975 (3)	0·6543 (2)	0·70713 (7)	0·0599 (3)
N10*B*	0·80063 (10)	0·4471 (5)	0·5742 (2)	0·0472 (10)
H5*B*	0·8152 (10)	0·408 (4)	0·896 (2)	0·024 (7)
H10*B*1	0·7844 (12)	0·462 (5)	0·527 (3)	0·047 (11)
H10*B*2	0·8273 (12)	0·436 (5)	0·567 (2)	0·038 (9)

Table 3. *Bond lengths* (Å) *and bond angles* (°) *in the two independent molecules* (*A and B*) *of* (1)

			Molecule *A*		Molecule *B*	
1	2	3	1—2	1—2—3	1—2	1—2—3
C2	S1	C8	1·728 (3)	86·94 (14)	1·733 (3)	87·29 (14)
N3	C2	Cl	1·293 (4)	122·5 (2)	1·294 (4)	123·6 (2)
N3	C2	S1		119·3 (2)		118·7 (2)
Cl	C2	S1	1·716 (3)	118·2 (2)	1·703 (3)	117·7 (2)
C9	N3	C2	1·381 (4)	107·8 (2)	1·383 (4)	107·9 (2)
C5	N4	C9	1·326 (4)	113·3 (3)	1·326 (4)	113·3 (3)
N6	C5	N4	1·343 (4)	128·8 (3)	1·343 (4)	129·2 (3)
C7	N6	C5	1·355 (4)	117·6 (3)	1·348 (4)	117·6 (3)
C8	C7	N10	1·398 (4)	124·1 (3)	1·411 (4)	122·9 (3)
C8	C7	N6		118·1 (3)		118·1 (3)
N10	C7	N6	1·321 (4)	117·8 (3)	1·329 (4)	119·0 (3)
C9	C8	S1	1·392 (4)	110·4 (2)	1·391 (4)	110·5 (2)
C9	C8	C7		118·8 (3)		118·7 (3)
S1	C8	C7	1·726 (3)	130·7 (2)	1·718 (3)	130·8 (2)
N3	C9	N4		121·2 (3)		121·3 (3)
N3	C9	C8		115·5 (3)		115·6 (3)
N4	C9	C8	1·347 (4)	123.3 (3)	1·351 (4)	123·1 (3)
H5	C5	N6	0·95 (3)	109· (2)	0·91 (3)	116· (2)
H5	C5	N4		122· (2)		115· (2)
H101	N10	H102	0·78 (4)	124. (4)	0·79 (4)	122· (3)
H101	N10	C7		115· (3)		120· (3)
H102	N10	C7	0·80 (4)	121· (3)	0·81 (3)	117· (2)

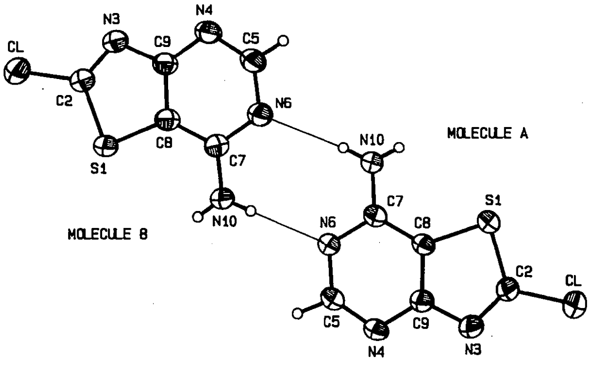
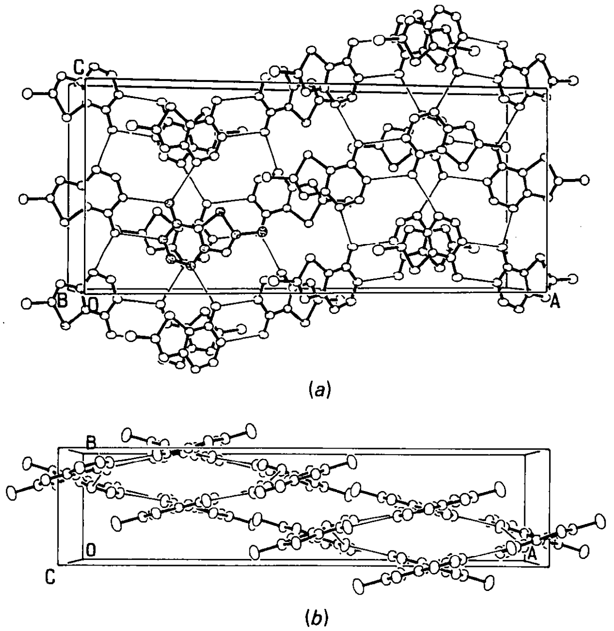


Fig. 1. Perspective drawing of (1) as the hydrogen-bonded dimer indicating atom labeling. Thermal ellipsoids are drawn at the 50% probability level.



(*a*)

(*b*)

Fig. 2. Perspective drawings of the molecular packing. The hydrogen bonding is indicated by thin lines. Molecules *A* lie along *x* = 0 and 0·5 and molecules *B* lie along *x* = 0·25 and 0·75. (*a*) View perpendicular to the plane of the shaded molecule *B* showing the stacking parallel to the *b* axis. Molecules *A* (which stack around centers of symmetry) have partial overlap of the atoms Cl, C2, N3, N4, C5 and C9; molecules *B* (which stack around twofold axes) have complete overlap of the pyrimidine rings. The interplanar spacings are 3·39 and 3·43 Å between planes in the *A* stacks and 3·42 and 3·53 Å between planes in the *B* stacks. (*b*) The molecular planes form a criss-cross pattern.

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Structure of 5,7-Dichloro-2-(*N*-methylanilino)[1,3]thiazolo[4,5-*d*]pyrimidine

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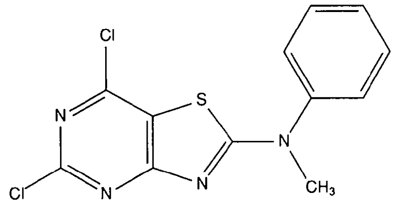
Abstract. $C_{12}H_8Cl_2N_4S$, $M_r = 311·19$, monoclinic, $P2_1/c$, $a = 7·2579\ (8)$, $b = 14·512\ (2)$, $c = 12·905\ (2)$ Å, $\beta = 98·608\ (10)°$, $V = 1343·9\ (3)$ Å^3, $Z = 4$, $D_x = 1·538$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 57·932$ cm^{-1}, $F(000) = 632$, $T = 295$ K, $R = 0·0343$ for 2352 reflections ($F \geq 4\sigma_F$). The thiazole and pyrimidine rings are planar [r.m.s.d.: 0·003 (2) and 0·003 (2) Å, respectively]; the dihedral angle between these planes is 1·44 (9)°. The C—S bond lengths are significantly different [1·773 (2) and 1·724 (2) Å]; the C—S—C angle is 87·13 (9)°. The C2, N10, C11, C12 fragment is nearly planar and rotated 3·97 (9)° with respect to the thiazolopyrimidine system. The dihedral angle between the phenyl ring and the thiazolopyrimidine ring is 73·00 (8)°. The thiazolopyrimidine rings are layered approximately parallel to the *bc* plane with spacings between adjacent rings of about 3·49 and 3·60 Å. The overlap involves the Cl atom at C7 which is sandwiched between thiazole rings of adjacent molecules, 3·48 Å from one and 3·61 Å from the other. There is no hydrogen bonding. The shortest contacts involve the disordered H atoms of the methyl group with Cl1 [2·757 (12) and 2·904 (9) Å].

Experimental. The title compound (1) was synthesized by the procedure outlined by Nagahara, Anderson, Kini, Dalley, Larson, Smee, Sharma, Jolley, Robins & Cottam (1989). Long, colorless, transparent needles were grown from ethanol and cut to the appropriate size. The data collection and refinement are summarized in Table 1.



(1)

The positions of the S and one Cl atom were determined from a sharpened Patterson map. Positions of 12 more atoms were determined from an electron density map and the remaining five non-H atoms were located in a difference map. A difference map calculated at $R = 0·046$ revealed the five phenyl H atoms and positions for two sets of disordered methyl H atoms (0·17–0·48 e Å^{-3}). During the final cycles, all atomic positions, anisotropic thermal parameters for non-H atoms and isotropic thermal parameters for H atoms were varied except for the methyl H atoms. These were idealized to a tetrahedral geometry with all d(C—H) equal, all d(N10···H) equal, and all H—C—H angles in each methyl group equal to 109·5°; all methyl H atoms had a common isotropic thermal parameter which was varied; the methyl-H-atom occupancies refined to 0·53:0·47. Refinement was accomplished by a full-matrix least-squares procedure (*SHELX*76; Sheldrick, 1976). Scattering factors and anomalous-

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