FRENZ, B. A. (1985). *Enraf–Nonius SDP-Plus Structure Determination Package*. Version 3·0. Enraf–Nonius, Delft, The Netherlands.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
LARSON, S. B. (1980). PhD Dissertation, Brigham Young Univ., USA.
LARSON, S. B., ANDERSON, J. D., COTTAM, H. B. & ROBINS, R. K. (1989). *Acta Cryst.* C**45**, 1073-1076.
LARSON, S. B., COTTAM, H. B. & ROBINS, R. K. (1989). *Acta Cryst.* C**45**, 1825–1827.
MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN*82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
NAGAHARA, K., ANDERSON, J. D., KINI, G. D., DALLEY, N. K., LARSON, S. B., SMEE, D. F., SHARMA, B. S., JOLLEY, W. B., ROBINS, R. K. & COTTAM, H. B. (1989). *J. Med. Chem.* In the press.
SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1989). C**45**, 1825–1827

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

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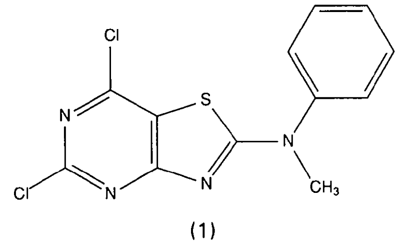
(*Received* 12 *May* 1989; *accepted* 22 *June* 1989)

Abstract. $C_{12}H_8Cl_2N_4S$, $M_r = 311{\cdot}19$, monoclinic, $P2_1/c$, $a = 7{\cdot}2579\,(8)$, $b = 14{\cdot}512\,(2)$, $c = 12{\cdot}905\,(2)$ Å, $\beta = 98{\cdot}608\,(10)^\circ$, $V = 1343{\cdot}9\,(3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}538$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 57{\cdot}932$ cm^{-1}, $F(000) = 632$, $T = 295$ K, $R = 0{\cdot}0343$ 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 $R = 0{\cdot}046$ 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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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 tanθ
Background	Scan 0·25 times scan range before and after scan
Scan rate (° min^{-1})	1·4–8·3
Exposure time (h)	32·7
Stability correction range on *I*	1·000–1·001
2θ range (°)	3·0–152·0
Range in *hkl*, min.	0,0, − 16
max.	9,18,16
Total reflections, measured, unique	3008, 2795
$R_{\rm int}$	0·0119
Crystal dimensions (mm)	0·41 × 0·185 × 0·17
Crystal volume (mm^3)	0·0123
Crystal faces	$\{100\}$; $\{010\}$; $\{01\bar{1}\}$; $\{011\}$
Transmission-factor range	0·238–0·464
(*B*) Structure refinement^c	
Reflections used ($F \geq 4\sigma_F$)	2352
No. of variables	215
Extinction parameter	8·5 (5) × 10^{-7}
Goodness of fit, *S*	1·822
R, *wR*	0·0343, 0·0527
R for all data	0·0435
Max. Δ/σ	0·0031
Max., min. density in ΔF map (e Å^{-3})	0·23, −0·31

Notes: (*a*) Unit-cell parameters were obtained by least-squares refinement of the setting angles of 25 reflections with $50{\cdot}1 < 2\theta < 59{\cdot}8°$. (*b*) Enraf–Nonius CAD-4 diffractometer with a graphite monochromator was used. Crystal and instrument stability were monitored by remeasurement of three check reflections ($24\bar{7}$, $2\bar{8}\bar{3}$, 271) every hour. 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 $w^{-1} = (\sigma_F^2 + 0{\cdot}0004F^2)$. $\sigma_F = F\sigma_I/2I$; $\sigma_I = (N_{\rm pk} + N_{\rm bg1} + N_{\rm bg2})^{1/2}$.

dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974); those for H were taken from Stewart, Davidson & Simpson (1965). Data were reduced with *SDP-Plus* (Frenz, 1985); least-squares planes were calculated with the program *PLANES* (Cordes, 1983); thermal-ellipsoid plots were 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 molecule illustrating atom labeling; Fig. 2 illustrates the molecular packing.

Related literature. The synthesis of the title compound was first reported by Nagahara *et al.* (1989).

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

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

	x	*y*	*z*	$U/U_{\rm eq}$
S1	0·76096 (7)	0·39059 (3)	0·46519 (4)	0·0543 (2)
C2	0·7726 (3)	0·39745 (14)	0·32914 (15)	0·0552 (6)
N3	0·7716 (2)	0·48070 (12)	0·28801 (13)	0·0594 (5)
N4	0·7517 (3)	0.63536 (13)	0·34440 (15)	0·0643 (6)
C5	0·7344 (3)	0·6862 (2)	0·4270 (2)	0·0645 (7)
N6	0·7261 (2)	0·66239 (13)	0·5249 (2)	0·0637 (6)
C7	0·7348 (3)	0·57253 (15)	0·5427 (2)	0·0561 (6)
C8	0·7513 (2)	0·50928 (13)	0·46493 (14)	0·0513 (6)
C9	0·7591 (3)	0·54444 (13)	0·3640 (2)	0·0536 (6)
Cl1	0·71788 (11)	0·80401 (4)	0·40265 (7)	0·0941 (3)
Cl2	0·72303 (8)	0·53490 (5)	0·66808 (4)	0·0736 (2)
N10	0·7858 (3)	0·32011 (13)	0·27488 (13)	0·0639 (6)
C11	0·8058 (4)	0·3253 (2)	0·1638 (2)	0·0929 (12)
C12	0·7735 (3)	0·23161 (15)	0·3221 (2)	0·0603 (6)
C13	0·6050 (4)	0·2004 (2)	0·3452 (2)	0·0736 (9)
C14	0·5960 (6)	0·1139 (2)	0·3880 (2)	0·0916 (12)
C15	0·7519 (7)	0·0599 (2)	0·4071 (2)	0·103 (2)
C16	0·9180 (7)	0·0908 (2)	0·3824 (3)	0·1050 (14)
C17	0·9309 (4)	0·1758 (2)	0·3401 (2)	0·0834 (10)
H13	0·495 (4)	0·240 (2)	0·326 (2)	0·102 (9)
H14	0·484 (5)	0·097 (2)	0·401 (3)	0·101 (11)
H15	0·745 (3)	0·003 (2)	0·446 (2)	0·089 (8)
H16	1·026 (6)	0·057 (3)	0·402 (3)	0·141 (15)
H17	1·038 (5)	0·195 (2)	0·321 (3)	0·102 (11)
H11*A**	0·878 (14)	0·274 (4)	0·145 (3)	0·122 (10)
H11*B**	0·868 (15)	0·382 (4)	0·150 (2)	0·122 (10)
H11*C**	0·6843 (13)	0·324 (8)	0·121 (2)	0·122 (10)
H11*D**	0·936 (2)	0·329 (9)	0·1567 (13)	0·122 (10)
H11*E**	0·75 (2)	0·271 (4)	0·128 (2)	0·122 (10)
H11*F**	0·74 (2)	0·379 (5)	0·133 (3)	0·122 (10)

* H11*A–C* constitute one methyl orientation; occupancy is 0·53 (3). H11*D–F* constitute the other orientation; occupancy is 0·47 (3). All methyl H atoms were given a common thermal parameter which was refined.

Table 3. *Bond lengths* (Å) *and bond angles* (°) *in* (1)

1	2	3	1—2	1—2—3
C2	S1	C8	1·773 (2)	87·13 (9)
N3	C2	N10	1·319 (3)	123·8 (2)
N3	C2	S1		116·8 (2)
N10	C2	S1	1·334 (3)	119·4 (2)
C9	N3	C2	1·361 (3)	109·2 (2)
C5	N4	C9	1·318 (3)	113·8 (2)
N6	C5	N4	1·319 (3)	130·7 (2)
N6	C5	Cl1		114·7 (2)
Cl1	C5	N4	1·740 (2)	114·6 (2)
C7	N6	C5	1·324 (3)	114·6 (2)
C8	C7	N6	1·379 (3)	122·4 (2)
C8	C7	Cl2		119·7 (2)
Cl2	C7	N6	1·721 (2)	117·9 (2)
C9	C8	S1	1·408 (3)	110·89 (14)
C9	C8	C7		116·9 (2)
S1	C8	C7	1·724 (2)	132·2 (2)
N3	C9	N4		122·5 (2)
N3	C9	C8		115·9 (2)
N4	C9	C8	1·343 (3)	121·6 (2)
C11	N10	C12	1·464 (3)	119·0 (2)
C11	N10	C2		119·7 (2)
C12	N10	C2	1·430 (3)	121·2 (2)
C13	C12	C17	1·378 (4)	120·4 (2)
C13	C12	N10		120·1 (2)
C17	C12	N10	1·391 (4)	119·5 (2)
C14	C13	C12	1·378 (4)	118·9 (3)
C15	C14	C13	1·368 (6)	120·4 (4)
C16	C15	C14	1·367 (7)	120·3 (3)
C17	C16	C15	1·358 (5)	120·5 (4)
C12	C17	C16		119·4 (3)

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* Tables of anisotropic thermal parameters, bond lengths and angles involving H atoms, torsion angles, least-squares planes and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52062 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

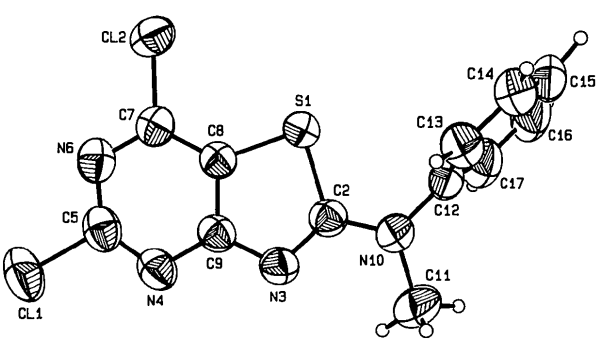

Fig. 1. Perspective drawing of (1) indicating atom labeling. Methyl H atoms H11*A*, H11*B* and H11*C* are shown. The other orientation is rotated approximately 60° with respect to the orientation shown. Thermal ellipsoids are drawn at the 50% probability level.

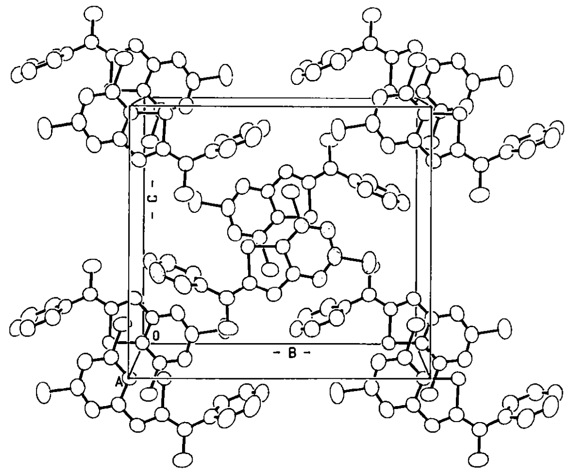

Fig. 2. Perspective drawing of the molecular packing as viewed perpendicular to the *bc* plane. The H atoms have been omitted for clarity. The thiazolopyrimidine rings form layers parallel to the *bc* plane with neighbors 3·49 and 3·60 Å apart. There is essentially no overlap of the thiazolopyrimidine rings although Cl2 is sandwiched between thiazole rings of adjacent molecules.

In the preceding paper we presented the structure of the 8-chloroadenine analog (7-amino-2-chloro-[1,3]thiazolo[4,5-*d*]pyrimidine) (Larson, Anderson, Cottam & Robins, 1989*b*) and we have recently reported the structure of the sodium salt of the 8-aminoguanine analog {2,5-diamino[1,3]thiazolo-[4,5-*d*]pyrimidin-7(6*H*)-one} (Larson, Anderson, Cottam & Robins, 1989*a*). The nucleoside 5-amino-3-β-D-ribofuranosyl-7(6*H*)-thioxothiazolo[4,5-*d*]pyrimidin-2(3*H*)-one, a 6-thioguanosine analog, has been reported (Nagahara *et al.*, 1989). No other thiazolo[4,5-*d*]pyrimidine crystal structures have been reported (Cambridge Structural Database, 1989).

References

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.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Larson, S. B. (1980). PhD Dissertation, Brigham Young Univ., USA.

Larson, S. B., Anderson, J. D., Cottam, H. B. & Robins, R. K. (1989*a*). *Acta Cryst.* C**45**, 1073–1076.

Larson, S. B., Anderson, J. D., Cottam, H. B. & Robins, R. K. (1989*b*). *Acta Cryst.* C**45**, 1822–1825.

Nagahara, K., Anderson, J. D., Kini, G. D., Dalley, N. K., Larson, S. B., Smee, D. F., Sharma, B. S., Jolley, W. B., Robins, R. K. & Cottam, H. B. (1989). *J. Med. Chem.* In the press.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1989). C**45**, 1827–1829

Structure of the Flavone Centaureidin

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(*Received* 13 *December* 1988; *accepted* 29 *June* 1989)

Abstract. 5,7-Dihydroxy-2-(3-hydroxy-4-methoxyphenyl)-3,6-dimethoxy-4*H*-1-benzopyran-4-one, $C_{18}H_{16}O_8$, $M_r = 360·3$, monoclinic, $P2_1/c$, $a = 8·393\,(2)$, $b = 18·356\,(3)$, $c = 10·297\,(2)$ Å, $\beta = 97·964\,(13)°$, $V = 1571·1\,(8)$ Å^3, $Z = 4$, $D_x = 1·523$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1·54184$ Å, $\mu = 9·85$ cm^{-1}, $F(000) = 752$, $T = 295$ K, $R = 0·041$ for 2241 observations (of 3235 unique data). The *A* ring exhibits maximum deviation, 0·013 (2) Å, from planarity, the heterocyclic *B* ring 0·017 (2) Å, and phenyl *C* ring 0·006 (2) Å. The *B* and *C* rings form a dihedral angle of 27·6 (1)°. The methoxy substitution of ring *B* is

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