GILMORE, C. J. (1984). *J. Appl. Cryst.* **17**, 42–46.
IBERS, J. A. & HAMILTON, W. C. (1964). *Acta Cryst.* **17**, 781–782.
JEFFREY, G. A., POPLE, J. A., BINKLEY, J. S. & VISHVESHWARA, S. (1978). *J. Am. Chem. Soc.* **100**, 373–379.
JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
KALLMERTEN, J. (1984). *Tetrahedron Lett.* **25**, 2843–2846.
Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200A Research Forest Drive, The Woodlands, TX 77381, USA.
PFLUGER, C. E., KALLMERTEN, J. & PLATA, D. J. (1989). *Acta Cryst.* C**45**, 1031–1034.
PLATA, D. J. & KALLMERTEN, J. (1988). *J. Am. Chem. Soc.* **110**, 4041–4042.
ROSSANO, L. T., PLATA, D. J. & KALLMERTEN, J. (1988). *J. Org. Chem.* **53**, 5189–5191.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
WHALEY, H. A., CHIDESTER, C. G., MIZSAK, S. A. & WNUK, R. J. (1980). *Tetrahedron Lett.* **21**, 3569–3662.
WHALEY, H. A. & COATES, J. H. (1981). 21st Intersci. Conf. Antimicrob. Agents Chemother. Abstract No. 187.
WITTMAN, M. D. & KALLMERTEN, J. (1988). *J. Org. Chem.* **13**, 4631–4633.

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Structure of 8-Amino-3,4-dihydro-2*H*,6*H*-pyrimido[2,1-*b*][1,3]thiazin-6-one

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Abstract. $C_7H_9N_3OS$, M_r = 183·23, monoclinic, $P2_1/c$, a = 7·152 (2), b = 15·407 (3), c = 7·493 (2) Å, β = 101·36 (3)°, V = 809·5 (9) Å^3, Z = 4, D_x = 1·50 g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, μ = 3·0 cm^{-1}, $F(000)$ = 384, T = 293 K, R = 0·040 for 1500 unique reflections with $I \geq 3\sigma(I)$. The structure consists of statistically disordered molecules with occupancy factors of 0·815 and 0·185. The disorder arises from two alternative half-chair conformations of the thiazine ring. The crystal packing is determined by intermolecular hydrogen-bonding interactions.

Introduction. The title compound was obtained from the reaction of 6-amino-2-thiouracil with 1,3-dibromopropane, and is a useful intermediate in the synthesis of thiazinopurine and triazolothiazinopyrimidine derivatives (Pecorari, Rinaldi & Costi, 1989). Their biological activity, on which little information is available, is one area of our current interest.

Experimental. Colourless prismatic crystals obtained at room temperature from ethanol–ether solution, dimensions 0·40 × 0·28 × 0·12 mm. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Measurements carried out at room temperature; 25 ($11 \leq \theta \leq 18°$) reflections for cell-parameter determination; $\omega/2\theta$ scan, scan width $(1·05 + 0·35 \tan\theta)°$; two standard reflections ($\bar{6}\bar{1}0$ and $\bar{3}\bar{6}0$) measured at 1 h intervals showed no significant intensity decay; 2088 reflections measured in the range $2 \leq \theta \leq 28°$, $-9 \leq h \leq 9$, $0 \leq k \leq 20$, $0 \leq l \leq 9$, 1934 unique ($R_{\rm int}$ = 0·031), 1500 with $I \geq 3\sigma(I)$ used for structure determination; intensities corrected for Lorentz–polarization effects and for absorption based on empirical ψ scan [$0·909 \leq T$ factor $\leq 0·999$]; space group from systematic absences. Structure solved by direct methods (*SHELXS*86; Sheldrick, 1986), and refined by full-matrix least squares with $\sum w\Delta F^2$ being minimized (*SHELX*76; Sheldrick, 1976). After isotropic refinement of all non-H atoms, an unrealistically high thermal parameter for the C(2) atom and a close residual in the ΔF map showed evidence of disorder, due to two positions in the crystal. The model was therefore adjusted to include two sites for this atom, for its H atoms and for those bonded to adjacent C atoms; preliminary least-squares refinement of their occupancy factors led to the values of 0·815 and 0·185, which were subsequently held fixed. All heavy atoms except C(2*b*) were then refined anisotropically, and H atoms with full or major occupancy (previously located in ΔF maps) isotropically; the H atoms at minor sites were added in calculated positions (C—H = 1·08 Å) with B values equal to those of the major sites; 146 parameters refined. R = 0·040, wR = 0·047, S = 1·44,

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Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters*

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

	x	y	z	B_{eq}(Å^2)
S	0·48546 (7)	0·57128 (3)	0·25217 (6)	3·75 (2)
C(1)	0·7179 (4)	0·5439 (2)	0·3818 (3)	4·42 (9)
C(2*a*)†	0·8690 (4)	0·5976 (2)	0·3241 (4)	4·5 (1)
C(3)	0·8744 (3)	0·5869 (2)	0·1290 (4)	5·2 (1)
N(1)	0·7022 (2)	0·6206 (1)	0·0070 (2)	3·03 (5)
C(4)	0·5271 (2)	0·6148 (1)	0·0483 (2)	2·52 (5)
N(2)	0·3702 (2)	0·64204 (9)	−0·0564 (2)	2·67 (5)
C(5)	0·3845 (2)	0·6782 (1)	−0·2193 (2)	2·60 (6)
N(3)	0·2197 (2)	0·7062 (1)	−0·3211 (2)	3·93 (7)
C(6)	0·5560 (3)	0·6845 (1)	−0·2757 (3)	3·09 (6)
C(7)	0·7227 (2)	0·6555 (1)	−0·1632 (3)	3·46 (7)
O	0·8844 (2)	0·6572 (2)	−0·1984 (3)	6·13 (9)
C(2*b*)†	0·876 (2)	0·5335 (8)	0·254 (2)	3·9 (3)‡

† The alternative occupancy factors of *a* and *b* labelled atoms are 0·815 and 0·185, respectively.

‡ Isotropic *B*.

Table 2. *Bond distances* (Å) *and bond angles* (°)

S—C(1)	1·803 (2)	C(1)—C(2*a*)	1·491 (4)
C(2*a*)—C(3)	1·479 (3)	C(3)—N(1)	1·476 (2)
N(1)—C(4)	1·351 (2)	C(4)—S	1·747 (1)
C(4)—N(2)	1·305 (2)	N(2)—C(5)	1·364 (2)
C(5)—N(3)	1·343 (2)	C(5)—C(6)	1·377 (2)
C(6)—C(7)	1·392 (2)	C(7)—N(1)	1·418 (2)
C(7)—O	1·236 (2)		
C(4)—S—C(1)	105·3 (1)	S—C(1)—C(2*a*)	111·0 (2)
C(1)—C(2*a*)—C(3)	112·8 (2)	C(2*a*)—C(3)—N(1)	113·1 (2)
C(3)—N(1)—C(4)	122·2 (1)	C(3)—N(1)—C(7)	118·0 (1)
C(7)—N(1)—C(4)	119·7 (1)	N(1)—C(4)—S	123·4 (1)
N(1)—C(4)—N(2)	124·5 (1)	S—C(4)—N(2)	112·0 (1)
C(4)—N(2)—C(5)	117·5 (1)	N(2)—C(5)—C(6)	122·1 (1)
N(2)—C(5)—N(3)	115·3 (1)	C(6)—C(5)—N(3)	122·6 (1)
C(5)—C(6)—C(7)	120·0 (1)	C(6)—C(7)—N(1)	116·0 (1)
C(6)—C(7)—O	126·2 (2)	N(1)—C(7)—O	117·8 (2)

$w = 1·77/(\sigma^2F + 0·00018F^2)$, max. $\Delta/\sigma = 0·21$, $-0·27 \leq \Delta\rho \leq 0·34$ e Å^{-3}. No correction for secondary extinction. Complex neutral-atom scattering factors from *SHELX*76 (Sheldrick, 1976). All calculations were carried out on a VAX 6310 computer.

Discussion. Atomic coordinates are listed in Table 1, and bond lengths and angles in Table 2.* *ORTEP* (Johnson, 1965) drawings of the molecule with atom-numbering scheme and of the unit-cell contents are depicted in Figs. 1 and 2, respectively. Five of the six atoms of the thiazine ring, and those of the pyrimidinone ring are coplanar (maximum deviations 0·036 and 0·014 Å, respectively). The dihedral angle between their mean planes is 1·4°. Bond distances and angles are normal (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987), and compare well with those of the 7-nitroso-3-hydroxy derivative (Pecorari, Rinaldi, Costi & Antolini, 1991). As evidenced by the short C(5)—N(3) bond distance of 1·343 (2) Å, there is strong conjugation between the amino group and the ring. The N(2)—C(4) bond [1·305 (2) Å in the present case and 1·304 (4) Å in the above-cited compound] shows a stronger double-bond order, when compared with corresponding values observed in other pyrimidine [1·329 (1) to 1·358 (2) Å] (Furberg, Grøgaard & Smedsrud, 1979) or uracil [mean value 1·379 (2) Å] (Taylor & Kennard, 1982) derivatives. The lengthening of the carbonyl C(7)—O bond distance with respect to the previously reported mean value of 1·227 (2) Å (Taylor & Kennard, 1982) may be due to a strong hydrogen-bonding interaction which involves the O atom. The crystal packing is mainly determined by two hydrogen-bonding interactions. The stronger one, which joins the molecules so as to form linear

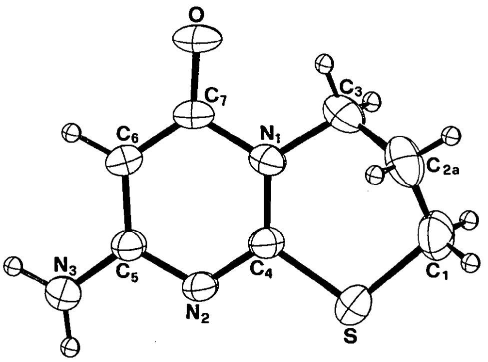

Fig. 1. *ORTEP* plot of the molecule with atom-numbering scheme. Thermal ellipsoids for non-H atoms enclose 50% probability.

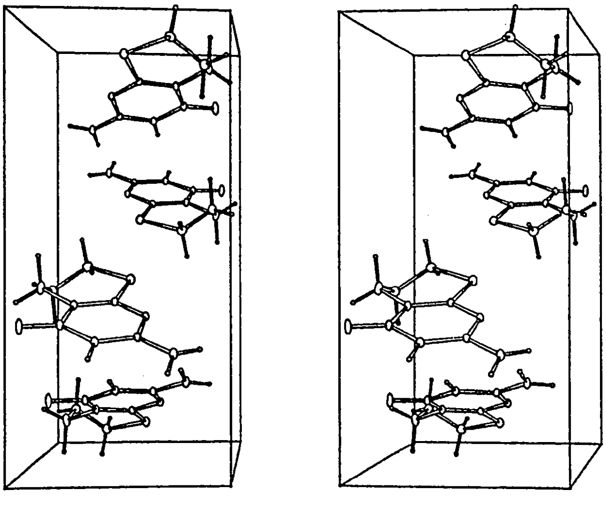

Fig. 2. Stereoview of the unit-cell contents approximately down the *c* axis, with *b* vertical.

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* Lists of structure factors, anisotropic temperature factors, positional and thermal parameters for H atoms, bond lengths and angles for H atoms, selected least-squares planes, and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54284 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

chains parallel to the *a* axis, occurs between the amino group and the carbonyl oxygen, with N···O and H···O separations of 2·834 (2) and 1·97 (3) Å, and a subtended N—H···O angle of 168 (2)°. These chains interact through a weaker hydrogen-bonding contact which involves the amino function and the N(2) ring atom [N(3)···N(2) = 3·238 (2), H···N(2) = 2·41 (3) Å, ∠N(3)—H···N(2) = 149 (2)°].

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References

ALLEN, F. H., KENNARD, O., WATSON, D. G., BRAMMER, L., ORPEN, G. & TAYLOR, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, S1–S19.
FURBERG, S., GRØGAARD, J. & SMEDSRUD, B. (1979). *Acta Chem. Scand. Ser. B*, **33**, 715–724.
JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
PECORARI, P., RINALDI, M. & COSTI, M. P. (1989). *J. Heterocyl. Chem.* **26**, 1701–1705.
PECORARI, P., RINALDI, M., COSTI, M. P. & ANTOLINI, L. (1991). *J. Heterocycl. Chem.* In the press.
SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
SHELDRICK, G. M. (1986). *SHELXS86*. Program for crystal structure solution. Univ. of Göttingen, Germany.
TAYLOR, R. & KENNARD, O. (1982). *J. Mol. Struct.* **78**, 1–28.

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Tris[(2-pyridinium)methyl]amine Perchlorate

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Abstract. Nitrilotrismethylenetri-2-pyridinium perchlorate, $C_{18}H_{21}N_4^{3+}.3ClO_4^-$, $M_r = 591·74$, cubic, $P2_13$, $a = 13·289$ (3) Å, $V = 2347$ (2) Å^3, $Z = 4$, $D_x = 1·675$ (2) g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 4·60$ cm^{-1}, $F(000) = 1216$, $T = 189$ (3) K, $R = 0·056$ for 1073 unique observed reflections with $I > \sigma(I)$. All four ions lie on threefold axes. The perchlorate ions are nearly regular tetrahedra. The bond lengths and angles in the ions are normal. As the name implies, the cation is protonated on the pyridine N atoms and not on the amine N atom. Each H atom attached to an N atom is part of a three-centered hydrogen bond in which the H-atom acceptors are O atoms on two different perchlorate ions.

Introduction. $[Fe_2TPA_2\{O_2P(OPh)_2\}](ClO_4)_3$, where TPA = tris(2-pyridylmethyl)amine and $O_2P(OPh)_2$ = diphenylphosphate, was synthesized by the reaction of $TPA.HClO_4$, $Fe(ClO_4)_3.10H_2O$, the diphenyl ester of phosphoric acid and triethylamine in methanol, yielding dark-green crystals (Norman, Yan, Que, Backes, Ling, Sanders-Loehr, Zhang, & O'Connor, 1990). Upon further standing a second pale-yellow crystalline product formed in low yield. These crystals were thought, at first, to be a different iron-containing complex and an X-ray diffraction study was begun. It quickly became clear that they did not contain iron but that they were probably a salt of the ligand. The cubic symmetry, which is relatively rare for crystals of large molecules, and the question of the location of the protons on the cation were the reasons for completing the structure determination. This determination, which also served as the chemical analysis, showed the compound to be the perchlorate salt of the triply protonated ligand.

Experimental. Crystals suitable for X-ray diffraction were found in the original sample. D_m was not measured. A light-yellow crystal, 0·2 × 0·2 × 0·6 mm, elongated along [111] was used for the data collection. Data were collected on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite monochromator. 25 reflections with $12 < \theta < 19°$ were used to determine the cell parameters. Systematic extinctions ($h00$, h odd) plus the Laue symmetry, $m3$, uniquely determined the space group. Data were collected, using ω scans, in the range $0 < \theta < 30°$ for most of one octant (h and k, 0 to 18; l, 0 to 11). The intensities of 2885 different reflections were measured. Three check reflections measured every 6000 s of exposure time showed no systematic change with time. After absorption corrections were made using *DIFABS* (Walker & Stuart, 1983; transmission factors 0·84–1·06), equivalent reflections were combined to give 1281 independent reflections ($R_{\rm int} = 0·052$) of which the 1073 with $I > \sigma(I)$ were used in the calculations. The structure was solved by direct methods (*MITHRIL*; Gilmore, 1984) and refined with full-

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