SDP.* Table 1 gives the atomic coordinates and Table 2 selected bond distances and angles. Fig. 1 shows the molecule with the numbering scheme.

Related literature. The structural parameters of this molecule can be compared with those found in the six-membered Se-containing rings of $(PhC)_2N_3(SeCl)$ (Oakley, Reed, Cordes, Craig & Graham, 1987) and $[(Ph_2P)(PhC)N_3Se]_2$ (Bestari, Cordes, Oakley & Young, 1990) as well as those of the sulfur analog $(Ph_2P)_2N_3SCl$ (Burford, Chivers, Hojo, Laidlaw, Richardson & Trsic, 1985).

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* Tables of distances and angles in the phenyl groups, anisotropic temperature factors, H-atom positions and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52342 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1990). C**46**, 506–508

Structure of 7-Methyl-8-thioxo-7,8-dihydroguanosine Monohydrate

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Abstract. 2-Amino-7-methyl-9-(β-D-ribofuranosyl)-8(9*H*)-thioxopurin-6(1*H*)-one (1) monohydrate, $C_{11}H_{15}N_5O_5S.H_2O$, $M_r = 347{\cdot}34$, orthorhombic, $P2_12_12_1$, $a = 6{\cdot}9207\ (2)$, $b = 10{\cdot}5170\ (9)$, $c = 20{\cdot}315\ (2)$ Å, $V = 1478{\cdot}6\ (2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}560$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54178$ Å, $\mu = 22{\cdot}866$ cm^{-1}, $F(000) = 728$, $T = 295$ K, $R = 0{\cdot}0286$ for 2874 reflections ($F \geq 4\sigma_F$). The sugar conformation and puckering parameters are $^2T_1$ (C2′-*endo*), $P = 156{\cdot}3°$ and $\tau_m = 38{\cdot}7°$. The side chain is *gauche–gauche*. The glycosidic torsion angle is 63·6 (2)° corresponding to the *syn* conformation which is stabilized by an O5′—H···N3 intramolecular hydrogen bond [d(H···N3) = 2·13 (3) Å; ∠O5′—H···N3 = 153 (3)°]. The purine ring is nearly planar [r.m.s. deviation: 0·011 (2) Å]; the dihedral angle between the pyrimidine and imidazole rings is 0·24 (7)°. All possible hydrogen donors participate in hydrogen bonding as do all possible hydrogen acceptors including the 8-thioxo group [d(H10*B*···S13) = 2·79 (2) Å; ∠N10—H10*B*···S13 = 111 (2)°].

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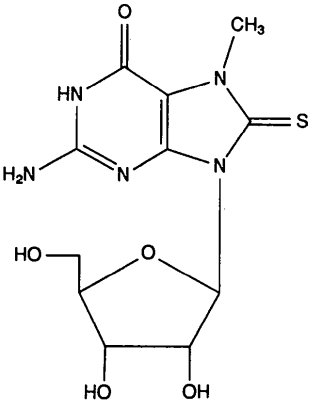
Experimental. The title compound (1).H_2O was synthesized by the procedure of Henry, Kini, Larson, Robins, Alaghamandan & Smee (1989). Colorless, transparent prismatic crystals grew from an ethyl acetate/methanol/acetone/water solution (18:1:1:1) following chromatography. Table 1 sumarizes data collection and refinement.



(1)

0108-2701/90/030506-03$03.00

Table 1. *Summary of data collection and refinement for* (1).H_2O

(*a*) Data collection (295 K)[i,ii]

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})	1·4–16·49
Exposure time (h)	65·0
Stability correction range on *I*	1·000–1·006
2θ range (°)	3·0–152·0
Range in *hkl*, min.	0, −13, −25
max.	8,13,25
Total reflections, measured, unique	6628, 3070
R_{int}	0·0164
Crystal dimensions (mm)	0·26 × 0·24 × 0·13
Crystal volume (mm^3)	0·00613
Crystal faces	{001}; {011}; {101}
Transmission-factor range	0·609–0·774

(*b*) Structure refinement[iii]

Reflections used ($F \geq 4\sigma_F$)	2874
No. of variables	277
Extinction parameter	1·10 (8) × 10^{-6}
Goodness of fit, *S*	1·466
R, *wR*	0·0286, 0·0391
R for all data	0·0322
Max., av. Δ/σ	0·0072, 0·0006
Max., min. $\Delta\rho$ in ΔF map (e Å^{-3})	0·31, −0·29

Notes: (i) Unit-cell parameters were obtained by least-squares refinement of the setting angles of 25 reflections with $51{\cdot}3 < 2\theta < 59{\cdot}8°$. (ii) Enraf–Nonius CAD-4 diffractometer with a graphite monochromator was used. Crystal and instrument stability were monitored by remeasurement of three check reflections (1,2,11, $16\bar{4}$, $4\bar{3}1$) every hour. A linear fit of the intensities of these reflections was used to correct the data. (iii) Function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w^{-1} = (\sigma_F^2 + 0{\cdot}0004F^2)$. $\sigma_F = F\sigma_I/2I$; $\sigma_I = [N_{pk} + N_{bg1} + N_{bg2}]^{1/2}$.

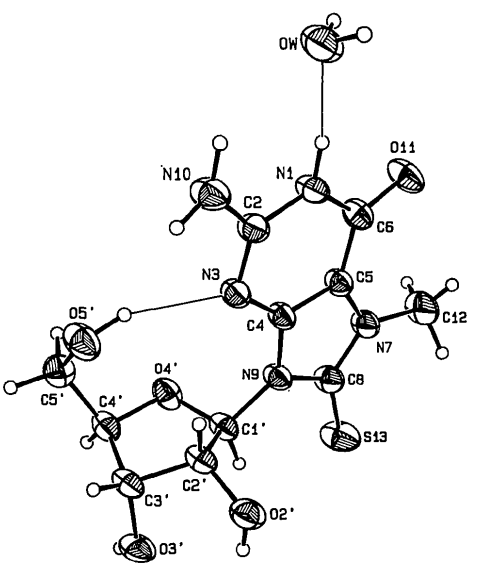


Fig. 1. Thermal-ellipsoid plot of (1).H_2O illustrating atom labeling, molecular conformation and intramolecular hydrogen bonding. The ellipsoids are drawn at the 50% probability level.

All non-H atomic positions were obtained with *SHELXS*86 (Sheldrick, 1986). All H atoms were located in a difference map as peaks of density

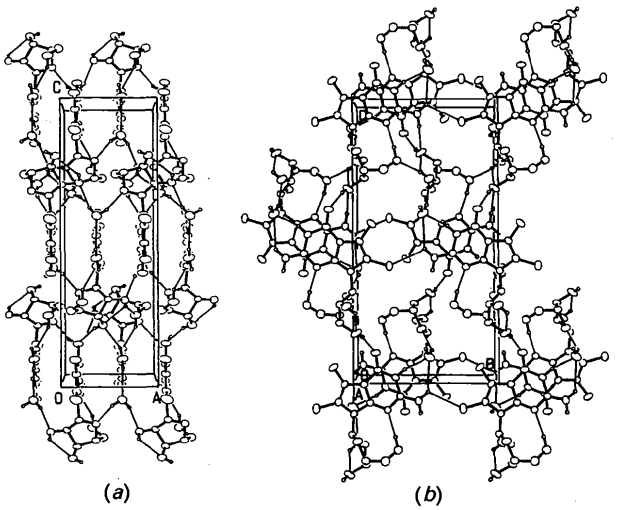
Table 2. *Positional and isotropic thermal parameters for all atoms in* (1).H_2O

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

	x	*y*	*z*	U/U_{eq}(Å^2)
N1	0·8577 (2)	0·79043 (12)	0·47442 (6)	0·0339 (3)
C2	0·8575 (2)	0·81961 (13)	0·54016 (7)	0·0319 (4)
N3	0·8556 (2)	0·73288 (11)	0·58735 (5)	0·0328 (3)
C4	0·8637 (2)	0·61329 (13)	0·56385 (6)	0·0297 (4)
C5	0·8678 (2)	0·57576 (14)	0·49905 (6)	0·0319 (4)
C6	0·8612 (2)	0·66804 (14)	0·44823 (7)	0·0334 (4)
N7	0·8743 (2)	0·44314 (13)	0·49790 (6)	0·0358 (4)
C8	0·8739 (3)	0·39822 (14)	0·56013 (7)	0·0360 (4)
N9	0·8702 (2)	0·50416 (11)	0·60137 (6)	0·0327 (3)
N10	0·8561 (3)	0·94355 (12)	0·55639 (7)	0·0443 (4)
O11	0·8602 (2)	0·65245 (12)	0·38803 (5)	0·0459 (4)
C12	0·8825 (4)	0·3644 (2)	0·43862 (9)	0·0529 (6)
S13	0·87662 (9)	0·24674 (4)	0·58374 (2)	0·0565 (2)
C1′	0·8505 (2)	0·49395 (13)	0·67233 (6)	0·0323 (4)
C2′	0·9881 (2)	0·57502 (14)	0·71339 (7)	0·0328 (4)
C3′	0·8749 (3)	0·58196 (15)	0·77828 (6)	0·0359 (4)
C4′	0·6632 (2)	0·5871 (2)	0·75565 (7)	0·0362 (4)
C5′	0·5775 (3)	0·7195 (2)	0·75268 (9)	0·0453 (5)
O2′	1·1747 (2)	0·52403 (13)	0·71744 (6)	0·0423 (3)
O3′	0·9216 (2)	0·46946 (13)	0·81424 (6)	0·0487 (4)
O4′	0·6616 (2)	0·53547 (11)	0·68913 (5)	0·0393 (3)
O5′	0·7010 (2)	0·80766 (11)	0·72034 (6)	0·0495 (4)
O*W*	0·8648 (2)	1·01860 (15)	0·40363 (6)	0·0521 (4)
H1	0·854 (3)	0·853 (2)	0·4454 (10)	0·040 (5)
H10*A*	0·884 (4)	0·956 (3)	0·5990 (13)	0·065 (7)
H10*B*	0·854 (3)	1·007 (2)	0·5252 (12)	0·054 (6)
H12*A*	0·946 (4)	0·403 (3)	0·4071 (14)	0·069 (8)
H12*B*	0·963 (5)	0·285 (3)	0·4521 (14)	0·089 (9)
H12*C*	0·752 (5)	0·352 (3)	0·4238 (14)	0·093 (10)
H1′	0·878 (3)	0·406 (2)	0·6842 (10)	0·039 (5)
H2′	1·002 (3)	0·656 (2)	0·6945 (9)	0·037 (5)
H3′	0·913 (3)	0·661 (2)	0·8050 (10)	0·049 (6)
H4′	0·578 (3)	0·535 (2)	0·7802 (11)	0·047 (5)
H5′*A*	0·545 (3)	0·752 (2)	0·7973 (10)	0·052 (6)
H5′*B*	0·451 (4)	0·716 (2)	0·7287 (11)	0·055 (6)
HO2′	1·181 (3)	0·466 (2)	0·7422 (11)	0·049 (6)
HO3′	0·826 (4)	0·432 (3)	0·8277 (14)	0·075 (9)
HO5′	0·707 (5)	0·789 (4)	0·675 (2)	0·107 (11)
H*WA*	0·787 (11)	0·994 (7)	0·374 (3)	0·22 (2)
H*WB*	0·977 (7)	1·035 (4)	0·383 (2)	0·128 (14)

0·34–0·87 e Å^{-3} at $R = 0{\cdot}050$. All positional parameters, anisotropic thermal parameters for non-H atoms and isotropic thermal parameters for H atoms were refined with *SHELX*76 (Sheldrick, 1976). Scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974) except those of H which were taken from Stewart, Davidson & Simpson (1965). Data were reduced with *SDP-Plus* (Frenz, 1985); least-squares-planes program from Cordes (1983); figures were drawn with *ORTEP*II (Johnson, 1976); parameter and geometry tables were produced with *FUER* and structure-factor tables were produced with *LISTFC* (Larson, 1980). The atomic coordinates are listed in Table 2. Fig. 1 illustrates the atom labeling and molecular conformation; Fig. 2 illustrates the unit-cell packing.*

* Tables of anisotropic thermal parameters, bond lengths and angles, bond lengths and angles involving H atoms, torsion angles, hydrogen-bonding geometries, least-squares planes and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52224 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.



(*a*) (*b*)

Fig. 2. Crystal packing diagrams of (1).H_2O with C—H H atoms omitted and hydrogen bonds drawn as thin lines. (*a*) View along the *b* axis, illustrating the base stacking and the hydrogen-bond linking of molecules through the water along the *a* axis. The interplanar spacings of the bases are 3·38 and 3·54 Å. (*b*) View along the *a* axis showing the partial base overlap.

Related literature. Henry *et al.* (1989) report the synthesis of the title compound and show that the immune system stimulatory effects are similar to those of 7-methyl-8-oxo-7,8-dihydroguanosine. The crystal structure of (1) is isomorphous to that of the 8-oxo nucleoside (Larson, Cottam & Robins, 1989). The C8—S13 bond length is 1·664 (2) Å; the r.m.s.d. of all other bond lengths involving non-H atoms is 0·006 Å. The r.m.s.d. of all bond angles is 0·54°. Structures of 8-substituted guanosines, most of which have immunomodulatory properties, show a predominance of *syn* conformation [*e.g.* 8-bromoguanosine (Tavale & Sobell, 1970), 8-chloroguanosine (Birnbaum, Lassota & Shugar, 1984), and 8-methylguanosine (Hamada, Honda, Fujii, Fujiwara & Tomita, 1985)] which may be partially responsible for such activity (Katze, 1985). Conformational parameters follow the conventions of Altona & Sundaralingam (1972).

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3-Hydroxy-1,3-bis(2-thienyl)prop-2-en-1-one

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Abstract. $C_{11}H_8O_2S_2$, $M_r = 236{\cdot}31$, monoclinic, *Cc*, $a = 27{\cdot}172$ (18), $b = 5{\cdot}282$ (3), $c = 21{\cdot}835$ (17) Å, $\beta = 136{\cdot}23$ (3)°, $V = 2168$ Å^3, $Z = 8$, $D_x = 1{\cdot}448$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}450$ mm^{-1}, $F(000) = 976$, $T = 298$ K, $R = 0{\cdot}0687$ for 786 unique observed reflections. The two independent molecules in the asymmetric unit are essentially identical: both are approximately planar, with no non-H atom deviating by more than 0·10 Å from the molecular mean plane. The thiophene S atoms are *cis* to the O atoms of the central moiety, whose molecular parameters indicate

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