Fig. 2. Drawing of compound $C_9H_7NO_3$ with thermal ellipsoids at the 50% probability level.

final difference Fourier synthesis 0·62, $-0·50$ e Å^{-3} for (I), 0·26, $-0·35$ e Å^{-3} for (II). Scattering factors from *International Tables for X-ray Crystallography* (1974). All standard calculations were performed with *CRYSRULER* (Rizzoli, Sangermano, Calestani & Andreetti, 1987) on an XT IBM 286 computer. Final atomic coordinates are given in Tables 1 and 2,* selected bond distances and angles are in Tables 3 and 4. Figs. 1 and 2 are *ORTEP* (Johnson, 1976) drawings of the molecules for (I) and (II), respectively.

Related literature. With the aim to understand better the auxin activity of several classes of compounds (3-indolylacetic, 1-naphthaleneacetic, 3-indolylbutyric and 2,4-chlorophenoxyacetic acids), an intensive search of the Cambridge Structural Database System (CSDS) (Allen *et al.*, 1979), accessed through the Servizio Italiano di Diffusione Dati Cristallografici (CNR, Parma), was performed. Since neither benzisothiazole nor benzisoxazole derivatives of acetic acid were found from this search, the crystal

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52311 (33 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

structure determinations of the title compounds were undertaken in order to gain more information so that a complete series of structural data could be correlated. Details on the correlation between structure and biological activity on six different auxins in *in vitro* regeneration of tomato plants are reported elsewhere (Branca, Bucci, Domiano, Ricci, Torelli & Bassi, 1989).

Other structurally similar molecules have been studied by Allen, Kennard, Watson, Brammer, Orpen & Taylor (1987); Gaetani, Vitali, Mangia, Nardelli & Pelizzi (1972); Lisgarten & Palmer (1988).

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Structure of Sulfonosine

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Abstract. 2-Amino-9-(β-D-ribofuranosyl)-9*H*-purine-6-sulfonamide (1) (sulfonosine), $C_{10}H_{14}N_6O_6S$, $M_r =$ 346·32, monoclinic, $P2_1$, $a = 6·2899$ (12), $b = 13·276$ (3), $c = 8·4161$ (14) Å, $\beta = 105·686$ (16)°, $V = 676·6$ (2) Å^3, $Z = 2$, $D_x = 1·700$ g cm^{-3}, Mo $K\alpha$ ($\lambda = 0·71073$ Å), $\mu = 2·723$ cm^{-1}, $F(000) = 360$, $T =$

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

(a) Data collection (295 K)[a,b]

Mode	ω scan
Scan range (°)	1·00
Background	Scan 0·25 times scan range before and after scan
Scan rate (° min⁻¹)	1·4–5·5
Exposure time (h)	12·6
Stability correction range on I	1·000–1·003
2θ range (°)	2·0–60·0
Range in hkl, min.	0,0,–11
max.	8,18,11
Total reflections measured, unique	2185, 2034
R_{int}	0·0095
Crystal dimensions (mm)	0·40 × 0·225 × 0·12
Crystal volume (mm³)	0·00980
Crystal faces	{001},{010},021,~4Ī0,~521
Transmission factor range	0·902–0·970

(b) Structure refinement[c]

Reflections used ($F \geq 4\sigma_F$)	1713
No. of variables	264
Extinction parameter	4·5(12) × 10⁻⁷
Goodness of fit, S	1·197
R, wR	0·0386, 0·0397
R for all data	0·0554
Max. Δ/σ	0·0021
Max., min. $\Delta\rho$ in ΔF map (e Å⁻³)	0·34, −0·30

(a) Unit-cell parameters were obtained by least-squares refinement of the setting angles of 25 reflections with $20\cdot4 < 2\theta < 29\cdot4°$. (b) Enraf–Nonius CAD-4 diffractometer with a graphite monochromator was used. Data reduction, which included Lorentz, polarization, decay and absorption corrections, was accomplished with the *SDP-Plus* software (Frenz, 1985). Crystal and instrument stability were monitored by re-measurement of four check reflections (12 $\bar{4}$, 16 $\bar{2}$, 42 $\bar{1}$, 23 $\bar{4}$) every hour. A linear fit of the intensities of these reflections was used to correct the data. Absorption corrections based on crystal face measurements were applied. (c) Function minimized was $\Sigma w(|F_o| - |F_c|)^2$, where $w^{-1} = (\sigma_F^2 + 0\cdot0004F^2)$. $\sigma_F = F\sigma_I/2I$; $\sigma_I = [N_{pk} + N_{bg1} + N_{bg2}]^{1/2}$.

295 K, $R = 0\cdot0386$ for 1713 reflections ($F \geq 4\sigma_F$). The sugar conformation and puckering parameters are $^2T_3$ ($C_{2'}$-*endo*), $P = 167\cdot0$ and $\tau_m = 36\cdot4°$. The C5′—O5′ side chain orientation is *gauche–gauche* [$\varphi_{OO} = -71\cdot1$ (4); $\varphi_{CO} = 48\cdot1$ (5)°] which, combined with a glycosidic torsion angle (C4—N9—C1′—O4′) of 54·0 (4)° (*syn* conformation), results in an intramolecular O5′—H···N3 hydrogen bond [d(H···N3) = 2·05 (4) Å; ∠O5′—H···N3 = 165 (5)°]. The purine ring is nearly planar [r.m.s. deviation: 0·013 (3) Å; the dihedral angle between the pyrimidine and imidazole rings is 1·7 (2)°. All possible hydrogen donors participate in hydrogen bonding although the hydrogens of the amino groups generally have long H···acceptor distances (2·19–2·50 Å). A C8—H8···O12 intermolecular interaction may exist [d(H8···O12) = 2·43 Å]. One O atom of the sulfonamido group is in the plane of the heterocycle *cis* to the imidazole ring. The amino groups are nonplanar; the N atoms deviate from the plane of the three adjoining atoms by 0·128 (3) Å for N10 and 0·199 (4) Å for N14 (sulfonamide). There is no base stacking; the ribose and sulfonamide moieties are sandwiched between heterocycles.

Experimental. The title compound (1) was synthesized by successive amination (with chloramine) and

Table 2. *Positional and isotropic thermal parameters* ($Å^2$) *for atoms in* (1)

	x	y	z	U/U_{eq}*
N1	0·4064 (5)	0·9307 (3)	0·4727 (3)	0·0262 (9)
C2	0·2981 (6)	0·9378 (3)	0·5922 (4)	0·0246 (10)
N3	0·2011 (5)	0·860383	0·6496 (3)	0·0251 (9)
C4	0·2210 (6)	0·7721 (3)	0·5797 (4)	0·0222 (10)
C5	0·3320 (6)	0·7552 (3)	0·4582 (4)	0·0241 (10)
C6	0·4226 (5)	0·8408 (3)	0·4094 (3)	0·0237 (9)
N7	0·3281 (5)	0·6530 (3)	0·4182 (3)	0·0300 (10)
C8	0·2175 (6)	0·6121 (3)	0·5113 (4)	0·0291 (11)
N9	0·1471 (5)	0·6791 (3)	0·6133 (3)	0·0262 (9)
N10	0·2888 (6)	1·0302 (3)	0·6550 (4)	0·0360 (12)
S11	0·57190 (13)	0·8410 (2)	0·25597 (9)	0·0249 (2)
O12	0·7859 (4)	0·8827 (3)	0·3270 (3)	0·0379 (9)
O13	0·5552 (5)	0·7414 (3)	0·1879 (3)	0·0352 (9)
N14	0·4415 (7)	0·9164 (3)	0·1164 (4)	0·0338 (12)
C1′	0·0237 (6)	0·6513 (3)	0·7290 (4)	0·0250 (10)
C2′	0·1461 (6)	0·6705 (3)	0·9103 (4)	0·0244 (10)
C3′	−0·0450 (6)	0·6843 (3)	0·9879 (4)	0·0278 (11)
C4′	−0·2155 (6)	0·7376 (3)	0·8525 (4)	0·0303 (11)
C5′	−0·2163 (6)	0·8516 (4)	0·8652 (5)	0·0358 (12)
O2′	0·2858 (5)	0·5882 (3)	0·9728 (4)	0·0378 (9)
O3′	−0·1332 (5)	0·5897 (3)	1·0185 (4)	0·0404 (10)
O4′	−0·1683 (4)	0·7109 (3)	0·6980 (3)	0·0318 (8)
O5′	−0·0027 (5)	0·8944 (3)	0·8941 (3)	0·0329 (9)
H8	0·188 (6)	0·540 (3)	0·520 (5)	0·034 (10)
H10A	0·249 (6)	1·035 (3)	0·733 (5)	0·023 (10)
H10B	0·372 (7)	1·068 (3)	0·635 (5)	0·031 (11)
H14A	0·319 (7)	0·906 (3)	0·077 (5)	0·031 (13)
H14B	0·469 (7)	0·974 (4)	0·134 (5)	0·046 (14)
H1′	−0·006 (6)	0·581 (3)	0·709 (4)	0·023 (9)
H2′	0·229 (5)	0·732 (3)	0·922 (4)	0·014 (8)
H3′	0·009 (6)	0·716 (3)	1·084 (5)	0·022 (9)
H4′	−0·357 (7)	0·711 (3)	0·844 (5)	0·035 (10)
H5′A	−0·310 (7)	0·878 (3)	0·758 (5)	0·052 (13)
H5′B	−0·253 (7)	0·871 (3)	0·957 (5)	0·039 (12)
H(O2′)	0·373 (7)	0·605 (3)	1·053 (5)	0·035 (12)
H(O3′)	−0·032 (8)	0·552 (4)	1·048 (6)	0·046 (14)
H(O5′)	0·029 (7)	0·885 (3)	0·820 (5)	0·032 (12)

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

oxidation (with 3-chloroperoxybenzoic acid) of 6-thioguanosine (Revankar, Hanna, Imamura, Lewis, Larson, Finch, Avery & Robins, 1990). Colorless, transparent prismatic crystals grew from a water/ethanol (8:2) solution. Table 1 summarizes data collection and refinement.

(1)
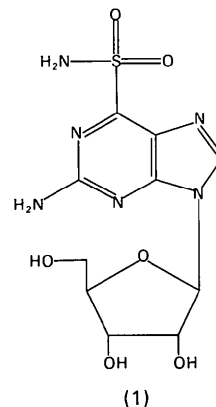
The position of the S atom, determined from a Patterson map, was input as a known fragment to *MULTAN82* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) from which coordinates of 12 additional atoms of the base

moiety were obtained. A subsequent Fourier and two subsequent difference Fourier maps provided positions for the remaining non-H atoms. All H atoms were located in a difference map as peaks of density $0.33-0.53$ e Å^{-3} at $R = 0.055$. All positional parameters, anisotropic thermal parameters for non-H atoms, isotropic thermal parameters for H atoms and an extinction parameter 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. Bond lengths and bond angles are given in Table 3. Hydrogen bonding is detailed in Table 4. Fig. 1
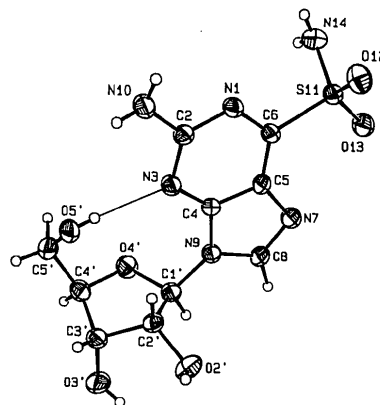

Fig. 1. Perspective drawing of (1) illustrating atom labeling, molecular conformation and intramolecular hydrogen bonding. The thermal ellipsoids are drawn at the 50% probability level.
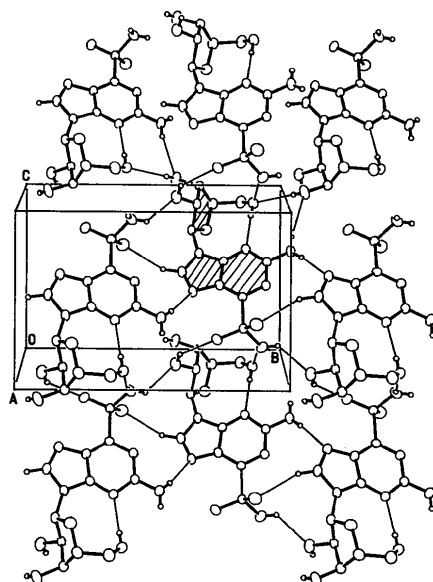

Fig. 2. Crystal packing diagram of (1) with sugar C—H hydrogens omitted and hydrogen bonds drawn as thin lines. There is no base stacking; sulfonamide and ribose moieties are sandwiched between bases. The shaded molecule has hydrogen bonding interactions with six symmetry-related molecules *via* two or three hydrogen bonds (all hydrogens are indicated).

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

1	2	3	1—2	1—2—3
C2	N1	C6	1·361 (5)	118·0 (3)
N3	C2	N10	1·350 (4)	118·8 (3)
N3	C2	N1		125·2 (3)
N10	C2	N1	1·343 (6)	116·0 (4)
C4	N3	C2	1·333 (4)	113·9 (3)
C5	C4	N9	1·403 (5)	105·6 (3)
C5	C4	N3		126·0 (4)
N9	C4	N3	1·375 (5)	128·4 (3)
C6	C5	N7	1·383 (6)	135·1 (4)
C6	C5	C4		114·3 (4)
N7	C5	C4	1·397 (6)	110·6 (3)
S11	C6	N1	1·790 (3)	113·8 (3)
S11	C6	C5		123·6 (3)
N1	C6	C5	1·322 (5)	122·6 (3)
C8	N7	C5	1·299 (5)	103·8 (3)
N9	C8	N7	1·389 (5)	114·7 (4)
C1′	N9	C4	1·448 (5)	129·7 (3)
C1′	N9	C8		124·8 (3)
C4	N9	C8		105·4 (3)
O12	S11	O13	1·429 (3)	118·9 (2)
O12	S11	N14		107·7 (3)
O12	S11	C6		108·5 (2)
O13	S11	N14	1·434 (4)	108·5 (2)
O13	S11	C6		106·9 (2)
N14	S11	C6	1·592 (4)	105·5 (2)
C2′	C1′	O4′	1·533 (4)	105·4 (3)
C2′	C1′	N9		114·3 (3)
O4′	C1′	N9	1·407 (5)	108·8 (3)
C3′	C2′	O2′	1·526 (6)	114·6 (3)
C3′	C2′	C1′		101·8 (3)
O2′	C2′	C1′	1·411 (5)	109·7 (3)
C4′	C3′	O3′	1·513 (5)	108·5 (3)
C4′	C3′	C2′		102·4 (3)
O3′	C3′	C2′	1·425 (6)	111·2 (3)
C5′	C4′	O4′	1·518 (7)	108·1 (3)
C5′	C4′	C3′		115·4 (3)
O4′	C4′	C3′	1·454 (5)	106·5 (3)
O5′	C5′	C4′	1·417 (5)	113·0 (3)
C1′	O4′	C4′		110·1 (2)

Table 4. *Hydrogen bonding and close intermolecular contacts in* (1) (Å *and* °)

D	H	A	Symmetry of A relative to D	d(D···A)	d(H···A)	∠(D—H···A)
C8	H8	O12	$1·0 - x, y - 0·5, 1·0 - z$	3·338 (5)	2·43 (4)	153 (3)
N10	H10A	O3′	$-x, 0·5 + y, 2·0 - z$	3·254 (5)	2·50 (4)	168 (4)
N10	H10B	N7	$1·0 - x, 0·5 + y, 1·0 - z$	3·106 (5)	2·34 (4)	168 (4)
N14	H14A	O5′	$x, y, z - 1·0$	2·927 (5)	2·19 (5)	162 (4)
N14	H14B	O2′	$1·0 - x, 0·5 + y, 1·0 - z$	3·067 (6)	2·49 (5)	130 (4)
O2′	HO2′	O13	$x, y, 1·0 + z$	2·937 (4)	2·28 (5)	143 (4)
O3′	HO3′	O2′	x, y, z	2·765 (5)	2·30 (5)	118 (4)
O3′	HO3′	O5′	$-x, y - 0·5, 2·0 - z$	2·767 (5)	2·15 (5)	135 (5)
O5′	HO5′	N3	x, y, z	2·741 (3)	2·05 (4)	165 (5)

illustrates the atom labeling and molecular conformation; Fig. 2 illustrates the unit-cell packing and hydrogen bonding.*

Related literature. The title compound has shown antitumor activity (Revankar *et al.*, 1990). The structure of 6-thioguanine (Bugg & Thewalt, 1970) and 6-thioguanosine (Thewalt & Bugg, 1972) have been reported. 6-Thioguanosine has an *anti* conformation. The *syn* conformation observed in (1) is characteristic of the 8-substituted guanosines such as 7-methyl-8-oxoguanosine (Larson, Cottam & Robins, 1989), 7-methyl-8-thioxoguanosine (Larson, Henry, Kini & Robins, 1990), 8-bromoguanosine (Tavale & Sobell, 1970), 8-chloroguanosine (Birnbaum, Lassota & Shugar, 1984) and 8-methylguanosine (Hamada, Honda, Fujii, Fujiwara & Tomita, 1985). Conformational parameters follow the conventions of Altona & Sundaralingam (1972).

* 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 52343 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Tetrahydrofuniculosin

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Abstract. rel-(2′S,5′R,6′R,1″R,3″R,5″S,2‴R,3‴R,-4‴S,5‴S)-4-Hydroxy-1-methyl-3-[5′-methyl-6′-(1″,-3″,5″-trimethylheptyl)tetrahydro-2′H-pyran-2′-yl]-5-(2‴,3‴,4‴,5‴-tetrahydroxy-1‴-cyclopentyl)-2-pyridone, $C_{27}H_{45}NO_7$, $M_r = 495.66$, monoclinic, $P2_1$, $a = 19.275$ (2), $b = 6.968$ (1), $c = 10.717$ (2) Å, $\beta = 104.10$ (1)°, $V = 1395.9$ Å^3, $Z = 2$, $D_x = 1.179$ g cm^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 6.474$ cm^{-1}, $F(000) = 540$, $T = 298$ K, final $R = 0.038$ for 1788 unique reflections [$F_o^2 > 1\sigma(F_o^2)$]. The cyclopentyl ring takes an envelope conformation and the four hydroxy groups are attached on the same side of the ring. Intra- and intermolecular hydrogen bonds are observed in this moiety.

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