

Fig. 2. The arrangement of molecules in the unit cell, viewed along *a*, showing hydrogen bonding.

C(2') [C(1')—C(2')—C(3')] 100.5 (2)°. This agrees with the observation made by Sundaralingam (1975), to the effect that 'in the arabinosides the puckered atoms always assume the smallest ring valency angle'. The *N*-glycosidic torsion angle χ [C(2)—N(1)—C(1')—O(4')] is 164.0 (2)°, in the usual *anti* range. The sugar pucker, as defined by the pseudorotational phase angles $P = 165.4$ (2)°, $\psi_m = 37.3$ (2)°, is 2E , C(2')-*endo*, which is in the same conformational region as all arabinosides studied to date.

The conformation about C(4')—C(5') as defined by the angle γ [C(3')—C(4')—C(5')—O(5')] is *ap*. In the structures of arabinosyl nucleosides an intramolecular hydrogen bond is sometimes formed between C(2') and O(5'). Formation of this intramolecular hydrogen bond in *ara*-FC is precluded by the *ap* conformation of the C(4')—C(5') bond.

The possible intermolecular hydrogen-bond contacts are given in Table 3 (deposited). All possible donor H atoms are involved in these contacts. There is one bifurcated (three-centred) contact involving H(N4) and F and O(5') (Fig. 2).

As has been noted in previous halogenated nucleosides (Wilson, Low & Young, 1983), the halogen atom stacks above a symmetry-related base. The perpendicular distance of the F atom above the mean plane of the base ring atoms is 3.015 (4) Å.

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Structure of 5-Methylthio-1,3,4-thiadiazole-2-thione*

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Abstract. C₃H₄N₂S₃, $M_r = 164.3$, monoclinic, $P2_1/n$, $a = 4.126$ (1), $b = 9.507$ (1), $c = 16.701$ (2) Å, $\beta =$

92.12 (1)°, $V = 654.7$ Å³, $Z = 4$, $D_m = 1.68$, $D_x = 1.67$ Mg m⁻³, Mo $K\alpha$, $\lambda = 0.7107$ Å, $\mu = 0.98$ mm⁻¹, $F(000) = 336.0$, $T = 293$ K, $R = 0.037$ for 900 observed reflections. The thiadiazole ring is planar with S—C(2) and C(2)—N(3) partial double bonds. The

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compound crystallizes as 5-methylthiothiadiazole-2-thione due to proton transfer in an N—H...S hydrogen bond. The crystal structure is also stabilized by two short S...S interactions.

Introduction. The title compound was prepared by suitable modifications of the general procedure for the preparation of thiadiazoles (Thorn & Ludwig, 1958). The study was undertaken mainly to clarify the bonding features around the sulfur atoms.

Experimental. Crystal (transparent yellow, needle shape) approximate dimensions 0.35 × 0.25 × 0.48 mm used for data collection; lattice parameters from 22 reflections ($22 \leq 2\theta \leq 38^\circ$); Enraf-Nonius CAD-4F-11M diffractometer; graphite-monochromated Mo K α radiation; three standard reflections ($\bar{1}, 5, \bar{1}\bar{1}$, 075, 247), 5% intensity variation; $\omega/2\theta$ scan mode; scan speed 1° min^{-1} ; $\theta \leq 23.5^\circ$; 1203 reflections measured, 900 significant ($|F_o| \geq 3\sigma|F_o|$); index range $h 0-4$, $k 0-10$, $l \pm 18$; no correction for absorption. Structure solved by direct methods using MULTAN78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); full-matrix refinement of scale factor, positional and anisotropic thermal parameters (positional and isotropic thermal parameters for H atoms, located from difference map) converged to $R = 0.037$ and $wR = 0.035$; $\sum w(|F_o| - |F_c|)^2$ minimized, $w = (3.0 + 1.0|F_o| + 0.02|F_o|^2)^{-1}$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Max. $\Delta/\sigma = 0.1$, final $\Delta\rho$ excursions $< |0.2| \text{ e } \text{Å}^{-3}$; LALS (Gantzel, Sparks & Trueblood, 1961) used for refinement.

Discussion. The atomic parameters and their standard deviations for non-H atoms are given in Table 1.* Bond lengths and bond angles are given in Table 2. Fig. 1 shows a perspective view of the molecule and the numbering of the atoms. Bond lengths S(2)—C(1) and C(1)—N(2) [1.665 (4) and 1.330 (5) Å respectively] indicate partial double-bond character, while C(2)—N(1) [1.282 (5) Å] is a pure double bond. This type of resonance structure also occurs in 5-mercapto-1,3,4-thiadiazole-2-thione (Bats, 1976). The bond N(1)—N(2) [1.381 (4) Å], however, is significantly longer than in 5-mercapto-1,3,4-thiadiazole-2-thione [1.366 (2) Å]. This feature may be attributed to the fact that in the present structure N(1) is not involved in hydrogen bonding.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42598 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The molecule, with the exception of the side groups, is essentially planar. The largest torsion angle within the five-membered ring is $-1.0 (4)^\circ$. N(2) is protonated as in 5-mercaptothiadiazole-2-thione (Bats, 1976), which makes the ring system asymmetric compared with 2,5-dimethylthiadiazole (Povet'eva & Zvonkova, 1972) and 1,3,4-thiadiazole (La Cour, 1974), which both have approximate C_{2v} symmetry. The molecules are held together by an intermolecular N(2)...S(2) hydrogen bond {S(2)...N(2)(2-x, -y, 2-z) = 3.299 (6) Å, S(2)...H[N(2)] = 2.29 (16) Å and S(2)...H[N(2)]—N(2) = 174 (1)°}.

There are two short S...S interactions [S(1)...S(3)($\frac{3}{2}-x, y-\frac{1}{2}, \frac{3}{2}-z$) = 3.520 (5) Å; S(1)...S(3)($\frac{5}{2}-x, y-\frac{1}{2}, \frac{3}{2}-z$) = 3.489 (5) Å]. These are attributed as being due to orbital interactions around sulfur and are also responsible for intermolecular interactions (Guru Row & Parthasarathy, 1981).

Table 1. Atomic coordinates ($\times 10^4$) with e.s.d.'s in parentheses and equivalent isotropic temperature factors for non-H atoms

$$B_{eq} = \frac{2}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	$B_{eq}(\text{Å}^2)$
S(1)	8630 (2)	2280 (1)	8004 (1)	2.76
S(2)	7546 (3)	-540 (1)	8791 (1)	3.91
S(3)	11612 (2)	5155 (1)	8187 (1)	3.17
N(1)	11599 (9)	3111 (3)	9292 (2)	3.13
N(2)	10567 (9)	1744 (3)	9398 (2)	3.40
C(1)	8986 (9)	1096 (4)	8795 (2)	2.79
C(2)	10749 (8)	3523 (4)	8585 (2)	2.36
C(3)	13379 (12)	6004 (5)	9057 (3)	4.04

Table 2. Bond distances (Å) and bond angles ($^\circ$) with e.s.d.'s in parentheses

S(1)—C(1)	1.738 (4)	C(1)—S(1)—C(2)	89.4 (2)
S(1)—C(2)	1.744 (4)	C(2)—S(3)—C(3)	100.1 (2)
S(2)—C(1)	1.665 (4)	N(2)—N(1)—C(2)	109.3 (3)
S(3)—C(2)	1.730 (4)	N(1)—N(2)—C(1)	118.9 (3)
S(3)—C(3)	1.794 (5)	S(1)—C(1)—S(2)	125.5 (2)
N(1)—N(2)	1.381 (4)	S(1)—C(1)—N(2)	107.6 (3)
N(1)—C(2)	1.282 (5)	S(2)—C(1)—N(2)	126.9 (3)
N(2)—C(1)	1.330 (5)	S(1)—C(2)—S(3)	120.0 (2)
		S(1)—C(2)—N(1)	114.8 (3)
		S(3)—C(2)—N(1)	125.2 (3)

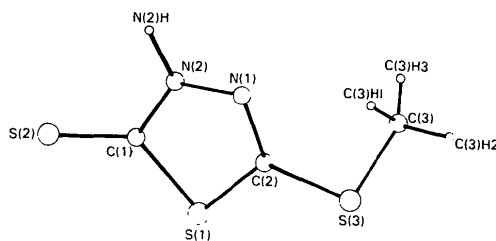


Fig. 1. A perspective view of the molecule showing the numbering scheme.

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The Structure of 24-Methylene-9,19-cyclolanostan-3 β -yl Acetate*

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Abstract. C₃₃H₅₄O₂, $M_r = 482.7$, monoclinic, $P2_1$, $a = 12.658$ (1), $b = 7.222$ (1), $c = 16.604$ (2) Å, $\beta = 97.52$ (1)°, $V = 1504.8$ Å³, $Z = 2$, $D_m = 1.06$ (1), $D_x = 1.07$ Mg m⁻³, Mo $K\alpha$, $\lambda = 0.7107$ Å, $\mu = 0.068$ mm⁻¹, $F(000) = 536$, $T = 293$ K, $R = 0.054$ for 1259 observed reflections. The four *trans*-fused rings *A*, *B*, *C* and *D* of the triterpene nucleus have chair, half-chair, 1:3 diplanar and half-chair conformations, respectively. Ring *B* is distorted by the cyclopropane ring fused to it. The molecules are loosely packed in the crystal as is evident from the very low density.

Introduction. Currently, a great deal of interest centres around the latex of the species *Euphorbia* as a possible petroleum substitute. The Himalayan plant *Euphorbia royleana* Boiss contains a large quantity of latex. The triterpene in the present study is obtained from source; it constitutes less than 0.3% of the latex and this precluded its structure determination by chemical methods and therefore X-ray crystal structure analysis was carried out.

Experimental. Compound isolated as its acetate by IDCC (inverted dry column chromatography) technique and crystallized from chloroform–acetone mixture, m.p. 386–387 K and $[\alpha]_D^{25} = 53.09^\circ$. Crystal $\sim 0.60 \times 0.20 \times 1.00$ mm; D_m by flotation in NaCl solution; Nonius CAD-4F-11M diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scan

mode, scan speed 1° min⁻¹, $\theta \leq 23.5^\circ$, h 0 to 14, k 0 to 8, l -18 to 18. 2668 reflections measured, 1259 significant ($|F_o| > 3\sigma|F_o|$), lattice parameters from 20 reflections ($22 \leq 2\theta \leq 34^\circ$), three standard reflections (020, 400 and 008) every 2000 s, 4% variation in intensity. No correction for absorption. Structure solved by direct methods with *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Full-matrix least-squares refinement (on F) of scale factor, positional and anisotropic thermal parameters (isotropic thermal parameters for H atoms), H atoms in calculated positions (but not refined); convergence at $R = 0.054$, $wR = 0.054$, $S = 0.92$. $\sum w(|F_o| - |F_c|)^2$ minimized, $w = (3.5 + 1.0|F_o| + 0.014|F_o|^2)^{-1}$. $(\Delta/\sigma)_{\max} = 0.1$. Final $\Delta\rho$ excursions < 10.3 e Å⁻³. No correction for secondary extinction. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). *LALS* (Gantzel, Sparks & Trueblood, 1961) for refinement.

Discussion. The atomic parameters with their e.s.d.'s and equivalent isotropic temperature factors are given in Table 1.‡ Bond lengths and angles are in Table 2. The chemical formula with the atomic numbering is shown in Fig. 1, and a perspective drawing of the molecule in Fig. 2.

‡ Lists of structure factors, anisotropic thermal parameters, H-atom parameters and selected torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42597 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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