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Structure of 2-[2-(Thymin-1-yl)ethylthio]acetic Acid

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Abstract. $C_9H_{12}N_2O_4S$, $M_r = 244{\cdot}26$, monoclinic, $P2_1$, $a = 8{\cdot}610$ (2), $b = 12{\cdot}427$ (2), $c = 5{\cdot}070$ (1) Å, $\beta = 92{\cdot}69$ (2)°, $V = 541{\cdot}9$ (2) Å^3, $Z = 2$, $D_m = 1{\cdot}48$, $D_x = 1{\cdot}497$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 29{\cdot}9$ cm^{-1}, $F(000) = 256$, $T = 293$ K, $R = 0{\cdot}064$ for 461 observed reflexions. The molecule takes a folded conformation. Bond distances and angles are normal. The carboxyl group is doubly hydrogen-bonded to O(4) and N(3) of the thymine moiety of the adjacent molecule related by 2_1 with an O···O distance of 2·61 (1) Å and an O···N distance of 2·84 (1) Å.

Introduction. As part of studies on elementary patterns in protein–nucleic acid interactions, we have found hydrogen-bonding schemes between carboxyl groups and nucleic acid bases (Ohki, Takenaka, Shimanouchi & Sasada, 1975, 1976, 1977; Takenaka, Ohki & Sasada, 1980; Takenaka & Sasada, 1982*a*; Fujita, Takenaka & Sasada, 1982, 1984), using model crystals that contain both components. The present paper deals with hydrogen bonds between a carboxyl group and thymine.

Experimental. 1-(2-Chloroethyl)thymine, derived from its 2-hydroxyethyl derivative (Shibata, Takenaka, Sasada & Ohki, 1985), was reacted with mercaptoacetic acid ethyl ester. After de-esterification, plate crystals of the title compound were obtained from an aqueous solution. D_m by flotation in a mixture of bromoform and cyclohexane. Rigaku four-circle diffractometer; graphite-monochromated Mo $K\alpha$ radiation; crystal size 0·5 × 0·2 × 0·05 mm; unit-cell dimensions determined with 38 reflexions ($11 < 2\theta < 25°$); 589 independent reflexions measured in the range $2 < 2\theta < 42°$, h −8~8, k 0~12, l 0~5; 461 reflexions with $F_o > 3\sigma$ considered observed; ω-scan mode, scan rate 2° min^{-1}, scan width 1·75° in ω. Five reference reflexions monitored every 50 reflexions showed no significant intensity deterioration. Corrections for Lorentz and polarization factors, but not for absorption. Standard deviations $\sigma^2(F_o) = \sigma_p^2(F_o) + qF_o^2$, where σ_p was evaluated by counting statistics and q was estimated to be $4{\cdot}67 \times 10^{-4}$ from measurement of monitored reflexions (McCandlish & Stout, 1975).

Structure solved by the heavy-atom method and refined by the full-matrix least-squares method; geometrically assigned H atoms included in structure-factor calculations isotropically; $\sum w(|F_o| - |F_c|)^2$ minimized, where $w = 1/\sigma^2(F_o)$; final R value 0·064 for 461 reflexions with $F_o > 3\sigma$ ($wR = 0{\cdot}056$, $S = 1{\cdot}48$); maximum shift of parameters $0{\cdot}08\sigma$, $\Delta\rho$ peak 0·27 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974); programs used: *TPAT* (Takenaka & Sasada, 1971), *LSAP*80 (Takenaka & Sasada, 1980), *DCMS*82 (Takenaka & Sasada, 1982*b*) and *LISTUP* (Takenaka & Sasada, 1983). Final atomic parameters are given in Table 1.*

Discussion. Fig. 1 shows the molecular structure with the atom numbering. The molecule as a whole takes a folded conformation; the torsion angles are given in the figure. The distance between the centres of the pyrimidine ring and the carboxyl group is 5·16 Å. Bond distances and angles of the thymine moiety are in agreement with those of thymine derivatives, *e.g.*

* Lists of structure factors, anisotropic thermal parameters of non-H atoms, H-atom coordinates and least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42822 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1-methylthymine (Kvick, Koetzle & Thomas, 1974) and thymidine (Young, Tollin & Wilson, 1969). The carboxyl group exhibits typical dimensions for an undissociated group.

Table 1. *Fractional coordinates and equivalent isotropic temperature factors*

$B_{eq} = 8\pi^2(U_1 + U_2 + U_3)/3$, where U_1, U_2 and U_3 are the principal components of the mean-square displacement matrix **U**. Values in ⟨ ⟩ are the anisotropicity defined by $[\Sigma(B_{eq}-8\pi^2U_i)^2/3]^{1/2}$ and those in ( ) are e.s.d.'s, referring to the last decimal places.

	x	*y*	*z*	B_{eq}(Å²)
N(1)	0·516 (2)	0·496 (1)	0·062 (2)	5·6 ⟨25⟩
C(2)	0·589 (2)	0·591 (2)	0·105 (4)	5·9 ⟨16⟩
N(3)	0·706 (2)	0·588 (1)	0·314 (3)	5·3 ⟨20⟩
C(4)	0·740 (2)	0·499 (2)	0·471 (3)	6·0 ⟨42⟩
C(5)	0·663 (2)	0·401 (1)	0·404 (3)	4·8 ⟨25⟩
C(6)	0·550 (2)	0·404 (2)	0·205 (4)	5·9 ⟨28⟩
O(2)	0·567 (1)	0·672 (1)	−0·021 (2)	6·5 ⟨22⟩
O(4)	0·841 (1)	0·513 (1)	0·658 (2)	6·3 ⟨17⟩
C(7)	0·701 (2)	0·300 (1)	0·560 (2)	5·8 ⟨25⟩
C(8)	0·391 (2)	0·493 (2)	−0·149 (3)	5·4 ⟨13⟩
C(9)	0·243 (2)	0·553 (1)	−0·068 (2)	5·5 ⟨21⟩
S	0·1584 (5)	0·50517*	0·2239 (7)	6·0 ⟨22⟩
C(10)	0·070 (2)	0·382 (1)	0·101 (3)	6·1 ⟨20⟩
C(11)	0·117 (2)	0·283 (2)	0·254 (4)	7·0 ⟨40⟩
O(5)	0·068 (1)	0·1960 (9)	0·132 (2)	7·0 ⟨22⟩
O(6)	0·181 (1)	0·285 (1)	0·472 (2)	7·8 ⟨47⟩

* Fixed.

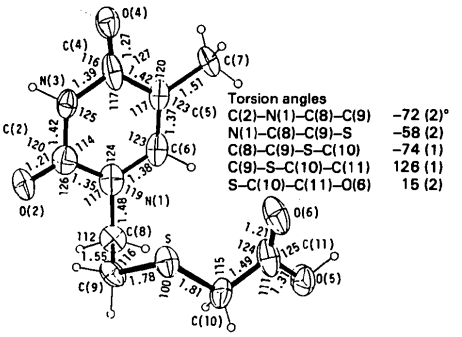

Fig. 1. Molecular structure of the title compound, with thermal ellipsoids at 30% probability. E.s.d.'s are 0·01–0·03 Å for bond distances and 0·6–1·8° for bond angles.

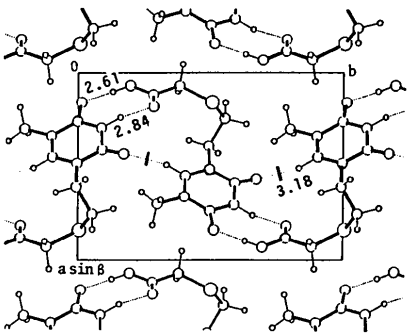

Fig. 2. The crystal structure projected along **c**. Hydrogen bonds are indicated by dotted lines.

The crystal structure is shown in Fig. 2. The carboxyl group is doubly hydrogen-bonded to O(4) and N(3) of the thymine moiety of the adjacent molecule related by 2_1. The OH···O and O···HN distances are 2·61 (1) and 2·84 (1) Å, respectively, and the dihedral angle between the planar groups composing the double hydrogen-bond system is 24·1 (6)°. The molecules are connected *via* these hydrogen bonds to form an infinite chain along **b**. In addition there are CH···O interactions between the molecular chains with a C(6)···O(2) distance of 3·18 (2) Å, which is slightly longer than that of 1-methylthymine (Kvick *et al.*, 1974). The hydrogen-bonding scheme in the present crystal is similar to that found in 5-carboxymethyl-6-methyluracil (Destro & Marsh, 1972).

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