

hydrogen bonds. The amino group is also hydrogen-bonded to O(9) of the hydroxyl group at  $(\frac{1}{2}-x, -y, \frac{1}{2}+z)$  to form a sheet parallel to the *ac* plane, and the O—H...O hydrogen bond between the hydroxyl group at  $(1-x, -\frac{1}{2}+y, \frac{1}{2}-z)$  and O(2) of cytosine links the sheets.

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## Structure of 1-(2-Hydroxyethyl)thymine Monohydrate

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**Abstract.**  $C_7H_{10}N_2O_3 \cdot H_2O$ ,  $M_r = 188.18$ , monoclinic,  $C2/c$ ,  $a = 12.210$  (1),  $b = 9.266$  (1),  $c = 16.338$  (1) Å,  $\beta = 105.39$  (1)°,  $V = 1782.2$  (1) Å<sup>3</sup>,  $Z = 8$ ,  $D_m = 1.396$ ,  $D_x = 1.403$  g cm<sup>-3</sup>,  $\lambda(\text{Cu K}\alpha) = 1.54184$  Å,  $\mu = 9.98$  cm<sup>-1</sup>,  $F(000) = 800$ , room temperature,  $R = 0.045$  for 1328 observed reflexions. The hydroxyl group is hydrogen-bonded to O(4) of an adjacent thymine to form a dimer around the inversion centre. There is no hydrogen bond between thymine moieties, and the thymine moiety is surrounded by water molecules, O(2), N(3) and O(4) making hydrogen bonds with water. The pyrimidine ring is planar within  $\pm 0.010$  Å.

**Introduction.** In a series of studies on hydrogen bonds between the hydroxyl group and nucleic acid bases (Shibata, Takenaka, Sasada & Ohki, 1985), the present paper deals with the structure of 1-(2-hydroxyethyl)-thymine.

**Experimental.** 1-(2-Hydroxyethyl)thymine, synthesized from thymine with ethylene carbonate in dimethylformamide in the presence of a trace of sodium hydroxide (Ueda, Kondo, Kono, Takemoto & Imoto, 1968). Colourless prism from aqueous solution.  $D_m$  by flotation in a mixture of chloroform and cyclohexane. Rigaku four-circle diffractometer, graphite-

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 principal components of the mean-square displacement matrix  $U$ . Values in  $\langle \rangle$  are anisotropy defined by  $[\sum(B_{eq} - 8\pi^2 U_i)^2/3]^{1/2}$  and those in  $( )$  are e.s.d.'s; they refer to last decimal places.

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{eq}(\text{\AA}^2)$
N(1)	0.3118 (1)	0.0029 (2)	-0.0868 (1)	2.42(59)
C(2)	0.3108 (2)	0.1322 (2)	-0.1275 (1)	2.52(44)
O(2)	0.2750 (1)	0.1465 (2)	-0.20451 (9)	3.78(206)
N(3)	0.3543 (1)	0.2467 (2)	-0.0758 (1)	2.51(76)
C(4)	0.3955 (2)	0.2437 (2)	0.0112 (1)	2.39(35)
O(4)	0.4312 (1)	0.3563 (2)	0.04977 (9)	3.43(126)
C(5)	0.3917 (2)	0.1057 (2)	0.0506 (1)	2.49(39)
C(5M)	0.4321 (3)	0.0940 (3)	0.1457 (1)	3.9(14)
C(6)	0.3510 (2)	-0.0073 (2)	0.0005 (1)	2.65(31)
C(7)	0.2622 (2)	-0.1252 (2)	-0.1364 (1)	3.04(91)
C(8)	0.1356 (2)	-0.1357 (3)	-0.1467 (2)	3.5(9)
O(9)	0.1120 (1)	-0.1562 (2)	-0.0666 (1)	3.97(116)
O(W)	0.3606 (2)	0.5206 (2)	-0.1469 (1)	4.82(310)

monochromated Cu  $K\alpha$  radiation, crystal size  $0.3 \times 0.3 \times 0.2$  mm; unit-cell dimensions determined with 47 reflexions ( $34 < 2\theta < 47^\circ$ ). Intensities measured for  $3 < 2\theta < 125^\circ$ ,  $h - 13$ – $13$ ,  $k 0$ – $10$ ,  $l 0$ – $18$ ;  $\omega$ -scan mode, scan rate  $8^\circ (\omega) \text{ min}^{-1}$ , scan width  $1.5^\circ (\omega)$ . Five reference reflexions monitored showed no significant intensity deterioration. Corrections for Lorentz and polarization factors, not for absorption; 1428 independent reflexions, 86 weak reflexions below background considered zero reflexions. Standard deviations  $\sigma^2(F_o) = \sigma_p^2(F_o) + qF_o^2$ , where  $\sigma_p(F_o)$  was evaluated by counting statistics and  $q$  estimated to be  $4.61 \times 10^{-5}$  from measurement of monitored reflexions (McCandlish & Stout, 1975).

Structure solved by direct methods, full-matrix least-squares refinement; all H atoms found on difference map and refined isotropically;  $\sum w(|F_o| - |F_c|)^2$  minimized, where  $w = 1/\sigma^2(F_o)$ ; zero reflexions with  $|F_c| > F_{lim}$  ( $F_{lim} = 1.537$ ) included in refinement assuming  $F_o = F_{lim}$  and  $w = w(F_{lim})$ ; final  $R$  value 0.045 for 1328 reflexions with  $F_o > 3\sigma$  ( $wR = 0.045$ ,  $S = 2.790$ ); maximum shift of parameters  $0.05\sigma$  for non-H atoms,  $\Delta\rho$  peak  $0.26 \text{ e \AA}^{-3}$ . Atomic scattering factors from *International Tables for X-ray Crystallography* (1974); programs used: *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), *LSAP80* (Takenaka & Sasada, 1980), *DCMS82* (Takenaka & Sasada, 1982) and *LISTUP* (Takenaka & Sasada, 1983). Final atomic parameters are given in Table 1.\*

\* Lists of structure factors, anisotropic thermal parameters and atomic parameters for H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42250 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

**Discussion.** Fig. 1 shows bond distances and angles. The dimensions of the thymine moiety are in good agreement with those of thymine monohydrate (Gerdil, 1961), 1-methylthymine (Hoogsteen, 1963) and thymidine (Young, Tollin & Wilson, 1969). The pyrimidine ring is planar within  $\pm 0.010 \text{ \AA}$ . Torsion angles are  $273.9 (2)^\circ$  for C(2)–N(1)–C(7)–C(8) and  $295.7 (2)^\circ$  for N(1)–C(7)–C(8)–O(9).

The crystal structure is shown in Fig. 2(a) and the hydrogen-bond scheme of thymine in Fig. 2(b). The hydroxyl group is hydrogen-bonded to O(4) of an adjacent thymine to form a dimer around the inversion centre at  $(\frac{1}{4}, \frac{1}{4}, 0)$ . These dimers are stacked with the pyrimidine planes around the inversion centre at (0,0,0) to form a column along [110]. There are no hydrogen-bond interactions between thymine moieties. The thymine moiety is surrounded by three water molecules, hydrogen bonds with water being formed at O(2), O(4) and N(3). These hydrogen bonds link the columns.

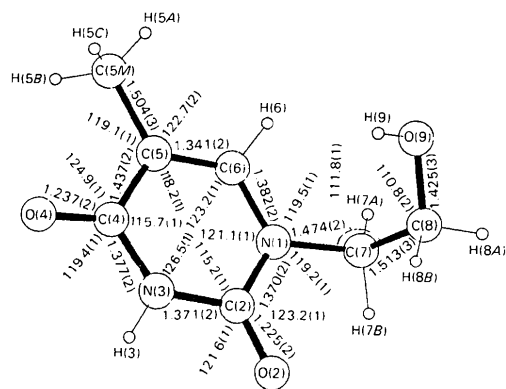


Fig. 1. Bond distances (Å) and angles ( $^\circ$ ) in 1-(2-hydroxyethyl)thymine. E.s.d.'s are shown in parentheses.

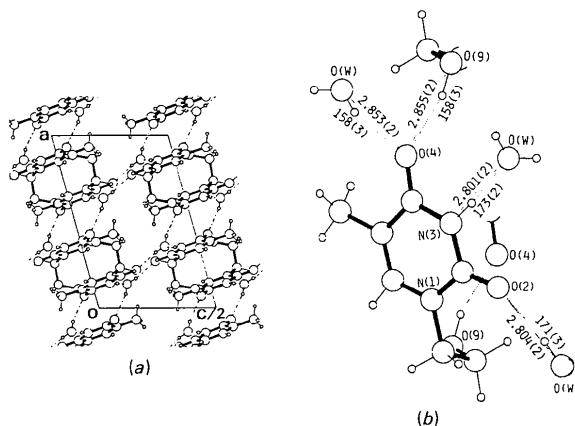


Fig. 2. (a) Crystal structure of 1-(2-hydroxyethyl)thymine monohydrate, projected along *b*. Broken lines indicate the hydrogen bonds. (b) Hydrogen-bonding patterns between the hydroxyl group and thymine moiety. Hydrogen-bond distances (Å) and angles ( $^\circ$ ) are shown with e.s.d.'s in parentheses.

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## Structure of a 1:2 Adduct between 1,4,7,10,13,16-Hexaoxacyclooctadecane (18-Crown-6) and Thiourea, C<sub>12</sub>H<sub>24</sub>O<sub>6</sub>·2CH<sub>4</sub>N<sub>2</sub>S

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**Abstract.**  $M_r = 416.4$ , triclinic,  $P\bar{1}$ ,  $a = 8.368$  (7),  $b = 8.691$  (9),  $c = 8.680$  (9) Å,  $\alpha = 114.9$  (1),  $\beta = 106.8$  (1),  $\gamma = 71.5$  (1)°,  $V = 534.0$  Å<sup>3</sup>,  $Z = 1$ ,  $D_x = 1.29$ ,  $D_m = 1.26$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.7107$  Å,  $\mu = 2.81$  cm<sup>-1</sup>,  $F(000) = 224$ , room temperature, final  $R = 0.061$  for 1829 independent observed reflections. The 18-crown-6 molecule contains a crystallographic centre of symmetry. Two thioureas are hydrogen-bonded on either side of the 18-crown-6 molecule *via* three N–H···O interactions. These crown.2thiourea adducts are connected *via* N–H···S hydrogen bonds to form a polymeric chain along the  $y$  axis.

**Introduction.** There has been an enormous amount of interest in adducts of the crown ethers particularly 18-crown-6 and over 300 structures containing this molecule have been reported so far. Of particular relevance to the present study are the 1:5 adduct with urea (Harkema, Van Hummel, Daasvatn & Reinhoudt, 1981), the 1:4 adduct with thiourea (Weber, 1984) and

the 1:2 adduct with  $N,N'$ -dimethylthiourea (Weber, 1983). We are also studying adducts between crown ethers and thiourea. We found that methanol solutions containing ratios of 18-crown-6 to thiourea of 1:1.25 and greater gave 1:4 adducts isomorphous with those of Weber. However, with a ratio of 1:1 we produced triclinic crystals with a 1:2 ratio of 18-crown-6 to thiourea. Accordingly we report here the crystal structure of this adduct (1).

**Experimental.** The 1:2 adduct was prepared by dissolving thiourea (0.76 g) in hot methanol (10 ml) and adding a hot solution of 18-crown-6 (2.64 g) in methanol (10 ml). The complex crystallized out after several hours and crystals suitable for diffraction work were washed in 2-propanol in order to remove the excess crown ether which adhered to them. The 1:4 adduct was prepared similarly, but with 3.20 g of 18-crown-6 and a total of 10 ml methanol. Crystals suitable for X-ray diffraction were formed overnight.