An anomalous feature of the physical properties of γ-HMX has been the difficulty in establishing conditions for the interconversion with other polymorphs (apart from the high-temperature conversion to δ-HMX). This structure determination indicates that water must be regarded as a component in the equilibrium. It is intended to pursue further experimental investigations into the thermodynamics of the HMX–H_2O system, in particular to measure vapour pressure and to determine the extent to which the H_2O composition can be varied.

We are indebted to Dr J. E. Davies for the IR spectra.

References

CADY, H. H., LARSON, A. C. & CROMER, D. T. (1963). *Acta Cryst.* **16**, 617–623.

CADY, H. H. & SMITH, L. C. (1961). *Studies on the Polymorphs of HMX*. Report No. LAMS-2652. Los Alamos Scientific Laboratory, Univ. of California.

CHOI, C. S. & BOUTIN, H. P. (1970). *Acta Cryst.* B**26**, 1235–1240.

COBBLEDICK, R. E. & SMALL, R. W. H. (1974). *Acta Cryst.* B**30**, 1918–1922.

COBBLEDICK, R. E. & SMALL, R. W. H. (1975). *Acta Cryst.* B**31**, 2805–2808.

EILAND, P. F. & PEPINSKY, R. (1955). *Z. Kristallogr.* **103**, 273–298.

HALLER, T. M., RHEINGOLD, A. L. & BRILL, T. B. (1983). *Acta Cryst.* C**39**, 1559–1563.

International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

MARSH, R. E. (1984). *Acta Cryst.* C**40**, 1632–1633.

SHELDRICK, G. M. (1976). *SHELX*76. A program for crystal structure determination. Univ. of Cambridge, England.

SMALL, R. W. H. (1947). PhD Thesis, Univ. of Birmingham.

Acta Cryst. (1985). C**41**, 1354–1356

Structure of 1-(2-Hydroxyethyl)cytosine

BY MASAYUKI SHIBATA, AKIO TAKENAKA AND YOSHIO SASADA

Laboratory of Chemistry for Natural Products, Tokyo Institute of Technology, Nagatsuta 4259, *Midori-ku, Yokohama* 227, *Japan*

AND MINORU OHKI

Research Institute, Wakamoto Pharmaceutical Co. Ltd, Kanate, Ohimachi, Ashigara-kamigun, Kanagawa 258, *Japan*

(*Received* 25 *April* 1985; *accepted* 24 *May* 1985)

Abstract. $C_6H_9N_3O_2$, $M_r = 155{\cdot}16$, orthorhombic, *Pbca*, $a = 12{\cdot}376$ (1), $b = 7{\cdot}554$ (1), $c = 15{\cdot}407$ (1) Å, $V = 1440{\cdot}3$ (1) Å^3, $Z = 8$, $D_m = 1{\cdot}435$, $D_x = 1{\cdot}431$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 9{\cdot}43$ cm^{-1}, $F(000) = 656$, room temperature, $R = 0{\cdot}049$ for 1109 observed reflexions. The cytosine moieties form a pair through the N(4)–H···N(3) hydrogen bonds around the inversion centre. The hydroxyl group is hydrogen-bonded with the amino group, to form a sheet parallel to the *ac* plane, and with O(2) of an adjacent molecule, which links the sheets. The pyrimidine ring is planar within ±0·027 Å.

Introduction. To reveal elementary binding patterns in protein–nucleic acid interactions, we have investigated the structures of model crystals that contain nucleic acid base and an amino-acid side group: carboxyl group (Fujita, Takenaka & Sasada, 1982, 1983, 1984*a*; Takenaka & Sasada, 1982*b*), carbamoyl group (Fujita, Takenaka & Sasada, 1984*b*,*c*, 1985; Takimoto, Takenaka & Sasada, 1981, 1982), indolyl group (Ohki, Takenaka, Shimanouchi & Sasada, 1977) and imidazolyl group (Takenaka, Takimoto & Sasada, 1984). The hydroxyl group is also a functional group which could play a role in the protein–nucleic acid interactions. We have synthesized several model compounds having both nucleic acid base and a hydroxyl group and examined their crystal structures by X-ray analysis. The present paper deals with the hydrogen-bonding patterns between cytosine and the hydroxyl group.

0108-2701/85/091354-03$01.50

Experimental. 1-(2-Hydroxyethyl)cytosine, synthesized from cytosine and ethylene carbonate in dimethylformamide in the presence of a trace of sodium hydroxide (Ueda, Kondo, Kono, Takemoto & Imoto, 1968). Columnar crystals from aqueous methanol or methanol solution. D_m by flotation in a mixture of chloroform and cyclohexane. Rigaku four-circle diffractometer, graphite-monochromated Cu $K\alpha$ radiation, crystal size $0{\cdot}7 \times 0{\cdot}4 \times 0{\cdot}2$ mm; unit-cell dimensions determined with 35 reflexions ($32 < 2\theta < 45°$). Intensities measured for $3 < 2\theta < 125°$, h 0–14, k 0–8, l 0–17; ω-scan mode, scan rate $4°\,(\omega)\,\text{min}^{-1}$, scan width $1{\cdot}5°\,(\omega)$. Five reference reflexions monitored showed no significant intensity deterioration. Corrections for Lorentz and polarization factors, not for absorption; 1151 independent reflexions, 39 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 $2{\cdot}59 \times 10^{-5}$ 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 $|F_c|>F_{\text{lim}}$ ($F_{\text{lim}}=0{\cdot}579$) included in refinement assuming $F_o=F_{\text{lim}}$ and $w=w(F_{\text{lim}})$; final R value 0·049 for 1109 reflexions with $F_o>3\sigma$ ($wR=0{\cdot}057$, $S=6{\cdot}53$); maximum shift of parameters $0{\cdot}04\sigma$ for non-H atoms, $\Delta\rho$ peak 0·31 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974); programs used: *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), *LSAP*80 (Takenaka & Sasada, 1980), *DCMS*82 (Takenaka & Sasada, 1982*a*) and *LISTUP* (Takenaka & Sasada, 1983). Final atomic parameters are given in Table 1.*

Discussion. Fig. 1 shows bond distances and angles. The dimensions of the cytosine moiety are in good agreement with those of 1-methylcytosine (Rossi & Kistenmacher, 1977), 3-(1-cytosinyl)propionamide (Fujita *et al.*, 1984*b*) and cytidine (Furberg, Petersen & Rømming, 1965). The pyrimidine ring is planar within ±0·027 Å. The torsion angles are 74·5 (2)° for C(2)–N(1)–C(7)–C(8) and 68·3 (2)° for N(1)–C(7)–C(8)–O(9).

The crystal structure is shown in Fig. 2(*a*) and the hydrogen-bond scheme of the cytosine moiety in Fig. 2(*b*). The cytosine moieties form a pair around the inversion centre at ($\frac{1}{2}$, 0, $\frac{1}{2}$) through the N(4)–H···N(3)

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

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

	x	y	z	B_{eq}(Å^2)
N(1)	0·3644 (1)	0·1999 (2)	0·2745 (1)	2·33⟨33⟩
C(2)	0·4544 (2)	0·1816 (3)	0·3278 (1)	2·22⟨40⟩
O(2)	0·5426 (1)	0·2422 (2)	0·30247 (9)	2·88⟨81⟩
N(3)	0·4441 (1)	0·0996 (2)	0·4051 (1)	2·38⟨67⟩
C(4)	0·3468 (2)	0·0447 (3)	0·4323 (1)	2·35⟨48⟩
N(4)	0·3416 (2)	−0·0380 (3)	0·5082 (1)	3·14⟨115⟩
C(5)	0·2520 (2)	0·0740 (3)	0·3816 (1)	2·80⟨70⟩
C(6)	0·2650 (2)	0·1489 (3)	0·3034 (1)	2·67⟨71⟩
C(7)	0·3782 (2)	0·2801 (3)	0·1881 (1)	2·9⟨6⟩
C(8)	0·4319 (2)	0·1564 (3)	0·1245 (2)	3·0⟨6⟩
O(9)	0·3636 (1)	0·0113 (2)	0·1033 (1)	3·76⟨146⟩

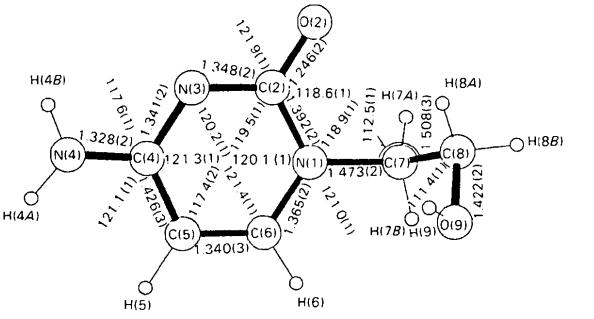

Fig. 1. Bond distances (Å) and angles (°) in 1-(2-hydroxyethyl)cytosine. E.s.d.'s are shown in parentheses.

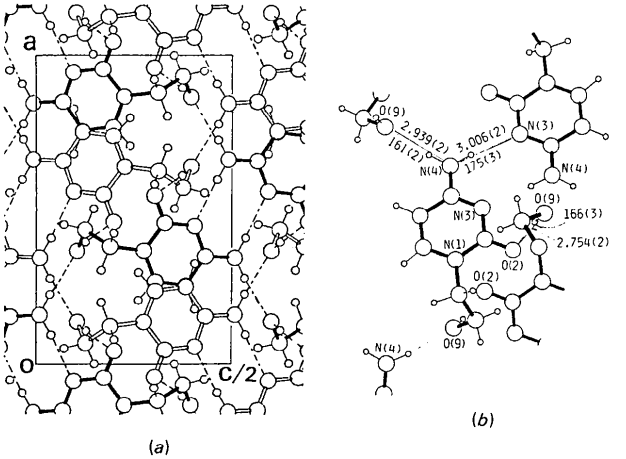

Fig. 2. (*a*) Crystal structure of 1-(2-hydroxyethyl)cytosine, projected along **b**. Broken lines indicate the hydrogen bonds. (*b*) Hydrogen-bonding patterns between the hydroxyl group and cytosine moiety. Hydrogen-bond distances (Å) and angles (°) are shown with e.s.d.'s in parentheses.

* 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 42249 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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.

The present work was supported in part by a Grant-in-Aid for Scientific Research, No. 5910600 from the Ministry of Education, Science and Culture, to which the authors' thanks are due. Part of the calculation was performed on an HITAC M200 computer at the Computer Centre of the Institute for Molecular Science.

References

FUJITA, S., TAKENAKA, A. & SASADA, Y. (1982). *Acta Cryst.* B**38**, 2936–2938.

FUJITA, S., TAKENAKA, A. & SASADA, Y. (1983). *Bull. Chem. Soc. Jpn*, **56**, 2234–2237.

FUJITA, S., TAKENAKA, A. & SASADA, Y. (1984*a*). *Bull. Chem. Soc. Jpn*, **57**, 1707–1712.

FUJITA, S., TAKENAKA, A. & SASADA, Y. (1984*b*). *Acta Cryst.* C**40**, 1467–1469.

FUJITA, S., TAKENAKA, A. & SASADA, Y. (1984*c*). *Acta Cryst.* C**40**, 1605–1607.

FUJITA, S., TAKENAKA, A. & SASADA, Y. (1985). *Biochemistry*, **24**, 508–512.

FURBERG, S., PETERSEN, C. S. & RØMMING, C. (1965). *Acta Cryst.* **18**, 313–320.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

MCCANDLISH, L. E. & STOUT, G. H. (1975). *Acta Cryst.* A**31**, 245–249.

MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN*78. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

OHKI, M., TAKENAKA, A., SHIMANOUCHI, H. & SASADA, Y. (1977). *Bull. Chem. Soc. Jpn*, **50**, 2573–2578.

ROSSI, M. & KISTENMACHER, T. J. (1977). *Acta Cryst.* B**33**, 3962–3965.

TAKENAKA, A. & SASADA, Y. (1980). *LSAP*80. Tokyo Institute of Technology, Japan.

TAKENAKA, A. & SASADA, Y. (1982*a*). *DCMS*82. Tokyo Institute of Technology, Japan.

TAKENAKA, A. & SASADA, Y. (1982*b*). *Bull. Chem. Soc. Jpn*, **55**, 680–686.

TAKENAKA, A. & SASADA, Y. (1983). *LISTUP*. Tokyo Institute of Technology, Japan.

TAKENAKA, A., TAKIMOTO, M. & SASADA, Y. (1984). *Biochim. Biophys. Acta*, **797**, 239–246.

TAKIMOTO, M., TAKENAKA, A. & SASADA, Y. (1981). *Bull. Chem. Soc. Jpn*, **54**, 1635–1639.

TAKIMOTO, M., TAKENAKA, A. & SASADA, Y. (1982). *Bull. Chem. Soc. Jpn*, **55**, 2734–2738.

UEDA, N., KONDO, K., KONO, M., TAKEMOTO, K. & IMOTO, M. (1968). *Makromol. Chem.* **120**, 13–20.

Acta Cryst. (1985). C**41**, 1356–1358

Structure of 1-(2-Hydroxyethyl)thymine Monohydrate

BY MASAYUKI SHIBATA, AKIO TAKENAKA AND YOSHIO SASADA

Laboratory of Chemistry for Natural Products, Tokyo Institute of Technology, Nagatsuta 4259, *Midori-ku, Yokohama* 227, *Japan*

AND MINORU OHKI

Research Institute, Wakamoto Pharmaceutical Co. Ltd, Kanate, Ohimachi, Ashigara-kamigun, Kanagawa 258, *Japan*

(*Received* 25 *April* 1985; *accepted* 28 *May* 1985)

Abstract. $C_7H_{10}N_2O_3.H_2O$, $M_r = 188{\cdot}18$, monoclinic, $C2/c$, $a = 12{\cdot}210$ (1), $b = 9{\cdot}266$ (1), $c = 16{\cdot}338$ (1) Å, $\beta = 105{\cdot}39$ (1)°, $V = 1782{\cdot}2$ (1) Å^3, $Z = 8$, $D_m = 1{\cdot}396$, $D_x = 1{\cdot}403$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 9{\cdot}98$ cm^{-1}, $F(000) = 800$, room temperature, $R = 0{\cdot}045$ 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 ±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-

0108-2701/85/091356-03$01.50