Marchand, A. P., Goodin, D. B., Hossain, M. B. & van der Helm, D. (1984). *J. Org. Chem.* **49**, 2897–2900.
Nicolet Instrument Corporation (1986). *SHELXTL for Desktop* 30 (*Microeclipse*). Report PN269–1040340, April. Nicolet Instrument Corp., WI, USA.
Shvo, Y. & Hazum, E. (1974). *J. Chem. Soc. Chem. Commun.* pp. 336–337.
Watson, W. H. (1983). Editor. *Stereochemistry and Reactivity of Systems Containing Pi-Electrons*. Deerfield Beach, Florida: Verlag Chemie International.

Acta Cryst. (1988). C**44**, 942–944

Structure of an Imidazoline Ribonucleoside

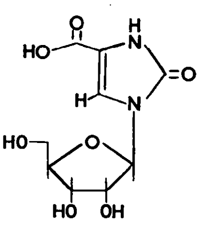
By Steven B. Larson, Howard B. Cottam and Roland K. Robins

Nucleic Acid Research Institute, 3300 *Hyland Avenue, Costa Mesa, CA* 92626, *USA*

(*Received* 30 *November* 1987; *accepted* 15 *January* 1988)

Abstract. 1-(β-D-Ribofuranosyl)-2-oxo-4-imidazoline-4-carboxylic acid dihydrate, $C_9H_{12}N_2O_7.2H_2O$, $M_r = 296{\cdot}23$, monoclinic, $P2_1$, $a = 9{\cdot}3754\,(10)$, $b = 5{\cdot}0542\,(4)$, $c = 13{\cdot}3075\,(10)$ Å, $\beta = 103{\cdot}155\,(9)°$, $V = 614{\cdot}03\,(10)$ Å^3, $Z = 2$, $D_x = 1{\cdot}602$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54178$ Å, $\mu = 12{\cdot}175$ cm^{-1}, $F(000) = 312$, $T = 295$ K, $R = 0{\cdot}0311$ for 2458 reflections ($F \geq 4\sigma_F$). The sugar bond lengths and bond angles are normal. The sugar ring is C_3, *endo* (conformation $^3T_4$) with angle of pseudorotation 26·9° and $\tau_m = 32{\cdot}0°$. The conformation around C4′—C5′ is *gt* [torsion angles: O4′, 67·1 (2); C3′, −175·94 (13)°]. The glycosidic torsion angle, χ = O4′—C1′—N1—C2, is −122·68 (14)°. Nine hydrogen bonds with $d(\text{H}\cdots A)$ ranging from 1·64 (3) to 2·20 (3) Å exist in the crystal structure. The imidazoline ring is planar [r.m.s.d. 0·003 (3) Å]. The carboxylic acid function is twisted 1·14 (8)° out of the plane of the imidazoline ring.

Experimental. The title compound, 1-(β-D-ribofuranosyl)-2-oxo-4-imidazoline-4-carboxylic acid dihydrate (1) was synthesized by the base-induced ring contraction of uridine previously reported by Otter, Falco & Fox (1969). Transparent crystals were grown from an acetone/water solution. The data collection and refinement are summarized in Table 1.



(1)

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

(*A*) Data collection (295 K)*†	
Mode	ω–2θ scan
Scan range (°)	$0{\cdot}80 + 0{\cdot}15\tan\theta$
Background	Scan 0·25 times scan range before and after scan
Scan rate (° min^{-1})	1·5–8·3
Exposure time (h)	17·1
Stability correction range on I	1·000–1·003
2θ range (°)	3·0–152·0
Range in *hkl*, min.	0, −6, −16
max.	11, 6, 16
Total reflections measured, unique	2722, 2482
R_{int}	0·0093
Crystal dimensions (mm)	0·36 × 0·20 × 0·13
Crystal volume (mm^3)	0·00912
Crystal faces	{001}; {100}; $(0\bar{1}0)$; (011); $(01\bar{1})$
Transmission-factor range	0·672–0·866
(*B*) Structure refinement‡	
Reflections used ($F \geq 4\sigma_F$)	2458
Number of variables	245
Extinction parameter	1·86 (5) × 10^{-5}
Goodness of fit, S	1·878
R, wR	0·0311, 0·0441
R for all data	0·0315
Max., av. Δ/σ	0·0033, 0·0006
Max., min. density in $\Delta\rho$ map (e Å^{-3})	0·49, −0·27

* Unit-cell parameters were obtained by least-squares refinement of the setting angles of 25 reflections with $54{\cdot}9 < 2\theta < 59{\cdot}5°$.

† Enraf–Nonius CAD-4 diffractometer with a graphite monochromator was used. Crystal and instrument stability was monitored by re-measurement of 3 check reflections every hour. A linear fit of the intensities of these reflections was used to correct the data.

‡ Function minimized was $\sum w(F_o - F_c)^2$, where $w^{-1} = (\sigma_F^2 + 0{\cdot}0004F^2)$. $\sigma_F = F\sigma_I/2I$; $\sigma_I = [N_{\text{pk}} + N_{\text{bg1}} + N_{\text{bg2}}]^{1/2}$.

The positions of the 20 non-H atoms were obtained by direct methods (*SHELXS*86; Sheldrick, 1986); the 16 H atoms were located in a difference map as peaks of 0·45–0·89 e Å^{-3} at $R = 0{\cdot}053$. All atomic positions were varied as well as all anisotropic thermal parameters for non-H atoms and isotropic thermal parameters for H atoms. Refinement by full-matrix least squares was carried out with *SHELX*76 (Sheldrick, 1976). Scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray*

0108-2701/88/050942-03$03.00

Table 2. *Positional and equivalent isotropic thermal parameters for non*-H *atoms in* (1)

	x	*y*	*z*	U_{eq}(Å^2)
N1	0·55716 (12)	0·388088	0·29924 (9)	0·0251 (3)
C2	0·70209 (13)	0·3128 (4)	0·33122 (10)	0·0240 (4)
N3	0·77812 (12)	0·4805 (4)	0·28359 (9)	0·0277 (3)
C4	0·68245 (15)	0·6562 (4)	0·22187 (11)	0·0270 (4)
C5	0·5455 (2)	0·5984 (4)	0·23159 (11)	0·0281 (4)
O6	0·75003 (11)	0·1267 (4)	0·38979 (9)	0·0339 (3)
C7	0·7336 (2)	0·8549 (4)	0·15905 (11)	0·0302 (4)
O8	0·86137 (13)	0·8806 (4)	0·15749 (10)	0·0432 (4)
O9	0·62646 (13)	1·0047 (4)	0·10575 (10)	0·0424 (4)
C1′	0·43743 (13)	0·2557 (4)	0·33086 (10)	0·0234 (3)
C2′	0·35471 (13)	0·4333 (4)	0·39250 (10)	0·0230 (3)
C3′	0·21124 (13)	0·4987 (4)	0·31283 (9)	0·0218 (3)
C4′	0·18988 (13)	0·2581 (4)	0·24186 (9)	0·0211 (3)
C5′	0·1081 (2)	0·3214 (4)	0·13309 (11)	0·0305 (4)
O4′	0·33550 (10)	0·1693 (4)	0·24063 (8)	0·0302 (3)
O2′	0·32812 (12)	0·2790 (4)	0·47536 (8)	0·0327 (3)
O3′	0·08782 (11)	0·5259 (3)	0·35560 (8)	0·0302 (3)
O5′	0·07955 (13)	0·0937 (4)	0·06895 (9)	0·0387 (4)
O*W*1	1·05048 (15)	−0·0216 (4)	0·44389 (11)	0·0387 (4)
O*W*2	0·3139 (2)	−0·1288 (4)	0·01349 (12)	0·0525 (5)

* $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*A_{ij}$, where A_{ij} is the dot product of the *i*th and *j*th direct-space unit-cell vectors.

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

1	2	3	1–2	1–2–3
C2	N1	C5	1·382 (2)	109·82 (12)
C5	N1	C1′	1·381 (2)	126·38 (11)
C1′	N1	C2	1·449 (2)	123·77 (10)
N3	C2	O6	1·354 (2)	127·98 (12)
N3	C2	N1		105·61 (13)
O6	C2	N1	1·239 (2)	126·40 (14)
C4	N3	C2	1·390 (2)	109·80 (12)
C5	C4	C7	1·351 (2)	130·42 (14)
C5	C4	N3		107·7 (2)
C7	C4	N3	1·456 (3)	121·84 (13)
N1	C5	C4		107·04 (13)
O8	C7	O9	1·210 (2)	124·2 (2)
O8	C7	C4		122·7 (2)
O9	C7	C4	1·327 (2)	113·10 (14)
C2′	C1′	O4′	1·539 (2)	107·80 (10)
C2′	C1′	N1		113·76 (14)
O4′	C1′	N1	1·422 (2)	108·22 (11)
C3′	C2′	O2′	1·547 (2)	111·78 (11)
C3′	C2′	C1′		102·95 (10)
O2′	C2′	C1′	1·418 (2)	107·0 (2)
C4′	C3′	O3′	1·525 (2)	108·91 (12)
C4′	C3′	C2′		103·03 (13)
O3′	C3′	C2′	1·408 (2)	114·22 (11)
C5′	C4′	O4′	1·510 (2)	109·32 (11)
C5′	C4′	C3′		113·03 (14)
O4′	C4′	C3′	1·441 (2)	105·32 (11)
O5′	C5′	C4′	1·422 (3)	112·9 (2)
C1′	O4′	C4′		110·56 (12)

Table 4. *Hydrogen bonding in* (1)

D—H···*A*			Symmetry of *A* relative to *D*	*d*(*D*···*A*) (Å)	*d*(H···*A*) (Å)	∠(*D*—H···*A*) (°)
N3	H3	O3′	1+*x*, *y*, *z*	2·8528 (15)	1·96 (2)	169· (2)
O9	H9	O*W*2	1−*x*, 1·5+*y*, −*z*	2·582 (2)	1·64 (3)	166· (3)
O2′	HO2′	O6	1−*x*, 0·5+*y*, 1−*z*	2·729 (2)	1·85 (3)	176· (3)
O3′	HO3′	O*W*1	*x*−1, 1·0+*y*, *z*	2·631 (2)	1·77 (4)	163· (3)
O5′	HO5′	O8	*x*−1, *y*−1·0, *z*	2·798 (2)	1·91 (3)	169· (3)
O*W*1	H*W*1*A*	O6	*x*, *y*, *z*	2·845 (2)	1·98 (3)	172· (3)
O*W*1	H*W*1*B*	O2′	1+*x*, *y*, *z*	2·961 (2)	2·18 (3)	161· (3)
O*W*2	H*W*2*A*	O5′	*x*, *y*, *z*	2·716 (2)	1·86 (3)	170· (3)
O*W*2	H*W*2*B*	O9	*x*, *y*−1·0, *z*	2·986 (2)	2·20 (3)	159· (3)

Crystallography (1974) except for H which were taken from Stewart, Davidson & Simpson (1965). Data were reduced with *SDP-Plus* (Frenz, 1985); least-squares planes calculated with program *PLANES* from Cordes (1983); thermal-ellipsoid plots produced with *ORTEP*II (Johnson, 1976). Parameter, geometry and structure-factor-amplitude tables were prepared with programs *FUER* and *LISTFC* (Larson, 1980).

Atomic coordinates are listed in Table 2;* bond lengths and bond angles are in Table 3. Fig. 1 is a perspective drawing of the molecule illustrating atom

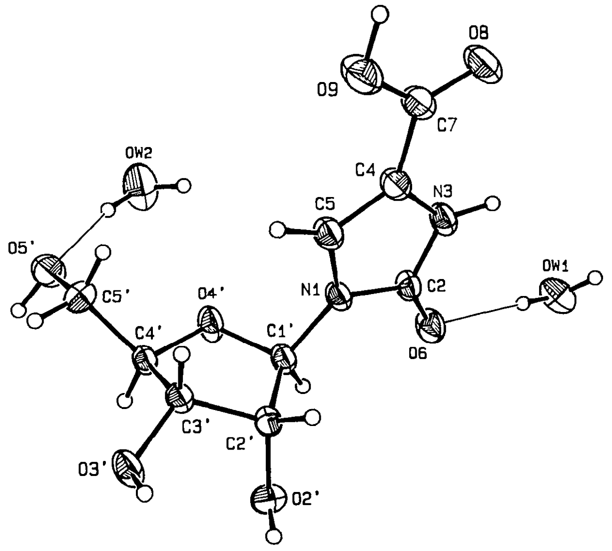


Fig. 1. Perspective drawing of (1) indicating atom labeling. Thermal ellipsoids are drawn at the 50% probability level.

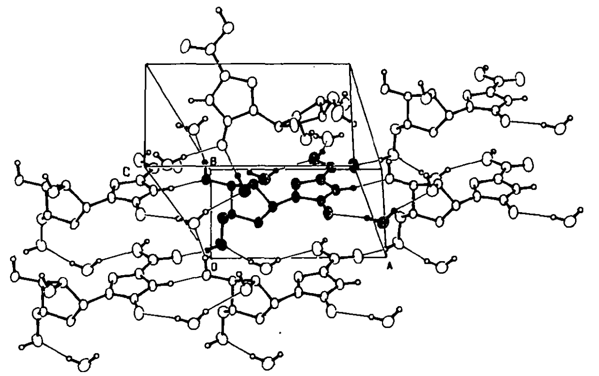


Fig. 2. Perspective drawing of the unit cell and hydrogen-bonding environment of the asymmetric unit (shaded atoms) with hydrogen bonds indicated by thin lines. Every –OH group (including the solvent) participates as both a donor and an acceptor.

---

* Tables of anisotropic thermal parameters, H-atom 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 44689 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

labeling; Fig. 2 illustrates the intermolecular hydrogen bonding which is detailed in Table 4.

Related literature. A single imidazoline structure has been reported (Ellestad *et al.*, 1978). Sugar conformations in nucleosides and nucleotides were discussed by Altona & Sundaralingam (1972). A recent summary of nucleoside and nucleotide structures has been given by Jeffrey & Sundaralingam (1985).

References

ALTONA, C. & SUNDARALINGAM, M. (1972). *J. Am. Chem. Soc.* **94**, 8205–8212.

CORDES, A. W. (1983). Personal communication.

ELLESTAD, G. A., COSULICH, D. B., BROSCHARD, R. W., MARTIN, J. H., KUNSTMANN, M. P., MORTON, G. O., LANCASTER, J. E., FULMOR, W. & LOVELL, F. M. (1978). *J. Am. Chem. Soc.* **100**, 2515–2524.

FRENZ, B. A. (1985). *Enraf–Nonius SDP-Plus Structure Determination Package*, version 3.0. Enraf–Nonius, Delft, The Netherlands.

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

JEFFREY, G. A. & SUNDARALINGAM, M. (1985). *Adv. Carbohydr. Chem. Biochem.* **43**, 203–422.

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138 (3rd revision). Oak Ridge National Laboratory, Tennessee, USA.

LARSON, S. B. (1980). PhD Dissertation, Brigham Young Univ.

OTTER, B. A., FALCO, E. A. & FOX, J. J. (1969). *J. Org. Chem.* **34**, 2636–2642.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

SHELDRICK, G. M. (1986). *SHELXS86*. Program for crystal structure determination. Univ. of Göttingen, Federal Republic of Germany.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1988). C**44**, 944–946

Structure of 5,5-Dimethyl-1,3,4-triphenylhexane-1,2-dione

BY J. L. BALCAZAR

Department of Geology, University of Alcala de Henares, Spain

M. S. ARIAS AND E. MOYA

Department of Organic Chemistry, University of Alcala de Henares, Spain

AND F. FLORENCIO

X-ray Department, Instituto Rocasolano, CSIC, 28006-Madrid, Spain

(*Received* 18 *June* 1987; *accepted* 21 *January* 1988)

Abstract. $C_{26}H_{26}O_2$, $M_r = 370{\cdot}490$, triclinic, $P\bar{1}$, $a = 8{\cdot}716\,(3)$, $b = 11{\cdot}077\,(8)$, $c = 12{\cdot}182\,(4)$ Å, $\alpha = 109{\cdot}17\,(4)$, $\beta = 105{\cdot}88\,(3)$, $\gamma = 91{\cdot}39\,(4)°$, $V = 1060{\cdot}1\,(9)$ Å^3, $D_x = 1{\cdot}1607$ g cm^{-3}, $\lambda(\text{Mo}\,K\alpha) = 0{\cdot}7107$ Å, $\mu = 0{\cdot}669$ cm^{-1}, $F(000) = 396$, $T = 293$ K, $R = 0{\cdot}055$ for 2321 observed diffractometer data and 357 refined parameters. The existence of the 1,2-dione group is verified. The molecule displays a steric interaction between the different groups attached to the central chain.

Experimental. Crystal 0·4 × 0·4 × 0·5 mm. Automated Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation. 25 centered reflections within $5 < \theta \leq 20°$ used for determining lattice parameters. Data corrected for Lorentz and polarization effects, absorption ignored. $2\theta_{\max} = 52°$, range of *hkl*: $0 \leq h \leq 10$, $-13 \leq k \leq 13$, $-15 \leq l \leq 15$. Two check reflections measured every 100 reflections showed no significant variation over data collection. $\omega/2\theta$ scans, 4139 reflections measured, 2678 independent and 2321 observed with $I > 2\sigma(I)$. $R_{\text{int}} = 0{\cdot}007$.

Structure solved by direct methods with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); refined by full-matrix least squares minimizing $\sum w(|F_o| - |F_c|)^2$; anisotropic thermal parameters for all non-H atoms and isotropic thermal parameters for H atoms, located from difference Fourier maps. w from an empirical weighting scheme so as to give no trends in $\langle w\Delta^2F\rangle$ *vs* $\langle|F_o|\rangle$ and $\langle(\sin\theta)/\lambda\rangle$. $(\Delta/\sigma)_{\max} = 0{\cdot}01$, $R = 0{\cdot}055$, $wR = 0{\cdot}052$, $S = 3{\cdot}97$. Final difference Fourier excursions 0·17 and −0·22 e Å^{-3}. Atomic scattering factors and anomalous-dispersion coefficients were taken from *International Tables for X-ray Crystallography* (1974). Calculations performed with *XRAY*76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976), *PARST* (Nardelli, 1983) and *PESOS* (Martinez-Ripoll & Cano, 1975) on a VAX11/750 computer.

0108-2701/88/050944-03$03.00