Table 2. *Bond lengths* (Å) *and angles* (°) (*e.s.d.'s are* 0·005 Å *and* 0·4°, *respectively*)

N–C(1)	1·282	C(8')–C(9')	1·386
N–C(3)	1·469	C(12)–C(13)	1·388
O–C(10)	1·224	C(12)–C(17)	1·391
C(1)–C(4)	1·506	C(13)–C(14)	1·390
C(1)–C(4')	1·496	C(14)–C(15)	1·386
C(3)–C(18)	1·528	C(15)–C(16)	1·382
C(3)–C(18')	1·518	C(16)–C(17)	1·374
C(4)–C(5)	1·404	C(18)–C(19)	1·380
C(4)–C(9)	1·397	C(18)–C(23)	1·380
C(5)–C(6)	1·398	C(19)–C(20)	1·395
C(5)–C(10)	1·502	C(20)–C(21)	1·372
C(6)–C(7)	1·376	C(21)–C(22)	1·373
C(7)–C(8)	1·372	C(22)–C(23)	1·400
C(8)–C(9)	1·388	C(18')–C(19')	1·383
C(10)–C(12)	1·489	C(18')–C(23')	1·392
C(4')–C(5')	1·384	C(19')–C(20')	1·391
C(4')–C(9')	1·394	C(20')–C(21')	1·364
C(5')–C(6')	1·384	C(21')–C(22')	1·353
C(6')–C(7')	1·383	C(22')–C(23')	1·404
C(7')–C(8')	1·366		

C(1)–N–C(3)	121·1	C(7')–C(8')–C(9')	121·3
N–C(1)–C(4')	117·6	C(4')–C(9')–C(8')	119·8
N–C(1)–C(4)	125·3	C(10)–C(12)–C(17)	118·8
C(4)–C(1)–C(4')	117·1	C(10)–C(12)–C(13)	121·7
N–C(3)–C(18')	110·8	C(13)–C(12)–C(17)	119·4
N–C(3)–C(18)	108·3	C(12)–C(13)–C(14)	120·3
C(18)–C(3)–C(18')	110·4	C(13)–C(14)–C(15)	119·7
C(1)–C(4)–C(9)	116·7	C(14)–C(15)–C(16)	119·8
C(1)–C(4)–C(5)	124·2	C(15)–C(16)–C(17)	120·7
C(5)–C(4)–C(9)	118·8	C(12)–C(17)–C(16)	120·1
C(4)–C(5)–C(10)	123·9	C(3)–C(18)–C(23)	121·6
C(4)–C(5)–C(6)	119·4	C(3)–C(18)–C(19)	120·0
C(6)–C(5)–C(10)	116·5	C(19)–C(18)–C(23)	118·5
C(5)–C(6)–C(7)	120·8	C(18)–C(19)–C(20)	120·5
C(6)–C(7)–C(8)	120·1	C(19)–C(20)–C(21)	120·8
C(7)–C(8)–C(9)	120·2	C(20)–C(21)–C(22)	119·0
C(4)–C(9)–C(8)	120·6	C(21)–C(22)–C(23)	120·4
O–C(10)–C(5)	118·1	C(18)–C(23)–C(22)	120·8
C(5)–C(10)–C(12)	122·1	C(3)–C(18')–C(23')	120·0
O–C(10)–C(12)	119·8	C(3)–C(18')–C(19')	121·6
C(1)–C(4')–C(9')	120·9	C(19')–C(18')–C(23')	118·4
C(1)–C(4')–C(5')	120·3	C(18')–C(19')–C(20')	120·8
C(5')–C(4')–C(9')	118·8	C(19')–C(20')–C(21')	120·0
C(4')–C(5')–C(6')	120·6	C(20')–C(21')–C(22')	120·6
C(5')–C(6')–C(7')	120·4	C(21')–C(22')–C(23')	120·4
C(6')–C(7')–C(8')	119·2	C(18')–C(23')–C(22')	119·8

Discussion. The final positional parameters are listed in Table 1. Fig. 1 is a computer-generated perspective drawing of the molecule. Table 2 shows bond lengths and angles; these are normal. Table 3* shows the angles between least-squares planes for various portions of the molecule and principal torsion angles, computed by *PARST* 5 (Nardelli, Musatti, Domiano & Andreetti, 1965).

* See deposition footnote.

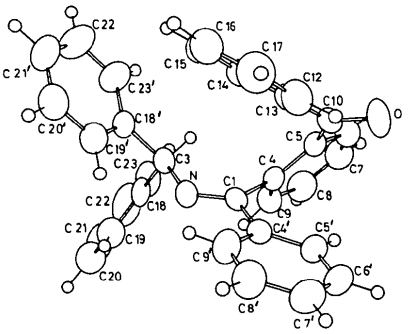

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecule, showing the atom numbering.

The photochemical transformation of (I) into (II) can be interpreted as a 1,5 acyl migration.

References

ARMESTO, D. & GOMEZ-GALLEGO (1982). Private communication. Departamento de Química Orgánica, Facultad de Ciencias Químicas, Madrid-3, Spain.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 72–98. Birmingham: Kynoch Press.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

MAIN, P., LESSINGER, L., WOOLFSON, M. M., GERMAIN, G. & DECLERCQ, J. P. (1977). *MULTAN. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

MARTÍNEZ-RIPOLL, M. & CANO, F. H. (1975). *PESOS* program. Instituto 'Rocasolano', CSIC, Serrano 119, Madrid-6, Spain.

NARDELLI, M., MUSATTI, A., DOMIANO, P. & ANDREETTI, G. D. (1965). *Ric. Sci. Parte* 2: *Sez. A*, **8**, 807–810.

RUIZ-VALERO, C., GUTIÉRREZ-PUEBLA, E. & MONGE, A. (1983). *Acta Cryst.* C**39**, 793–794.

Acta Cryst. (1983). C**39**, 796–798

Structure of 3',5'-Di-*O*-acetyl-5-bromo-2'-deoxyuridine, $C_{13}H_{15}BrN_2O_7$

BY J. N. LOW

*Carnegie Laboratory of Physics, The University, Dundee DD*1 4*HN, Scotland*

(*Received* 20 *December* 1982; *accepted* 10 *February* 1983)

Abstract. $M_r = 391{\cdot}2$, monoclinic, $C2$, $a = 16{\cdot}444$ (10), $b = 6{\cdot}651$ (6), $c = 17{\cdot}246$ (10) Å, $\beta = 56{\cdot}9$ (1)°, $U = 1580$ Å^3, $Z = 4$, $D_x = 1{\cdot}64$ Mg m^{-3}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71069$ Å, $\mu = 2{\cdot}552$ mm^{-1}, $T = 293$ K, $F(000) = 792$, $R = 0{\cdot}077$ for 1185 observed reflexions (Friedel pairs not merged). The sugar-ring pucker in

0108-2701/83/060796-03$01.50

terms of the phase angle of pseudorotation is $P = 166°$, $\chi_{CN} = -113\ (1·4)°$, and the C(4′)–C(5′) conformation is g^+.

Introduction. The structure determination was undertaken as part of a series of conformational studies of nucleic acid components, with particular reference to the glycosyl torsion angle χ_{CN} and the sugar-ring pucker (Low, Tollin & Wilson, 1982). The atomic numbering is shown in Fig. 1.

Experimental. Structure determined using intensities measured on a Stoe STADI 2 diffractometer (two-circle); all calculations performed on the Dundee University DEC-10 computer using the *SHELX*76 (Sheldrick, 1976), *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO* (Motherwell & Clegg, 1978) program packages. Cell dimensions measured from zero-level Weissenberg photographs of crystals mounted along **a** and **b**, and on the diffractometer; intensities collected using the *b*-axis crystal (dimensions 0·78 × 0·34 × 0·2 mm elongated along **b**) in the range $k = 0$ to 6 and $0° < 2\theta \leq 60°$; absorption corrections applied; 2102 reflexions measured; $R_{int} = 0·017$; a small number of reflexions which had inconsistent values for symmetrically related pairs were rejected as were all planes for which $F < 5\sigma F$; the latter were regarded as unobserved; refinement carried out using 1185 reflexions. Structure solved by the heavy-atom method and refined by least squares; Br atom was refined using anisotropic thermal parameters; H atoms included as 'riding atoms' at calculated positions. Final refinement: 103 refined parameters, $wR = 0·079$, $w = 2·766 \times [\sigma^2(F) + 0·001331F^2]$, maximum shift/e.s.d. = 0·04, residual electron density = 0·8 e Å^{-3}.

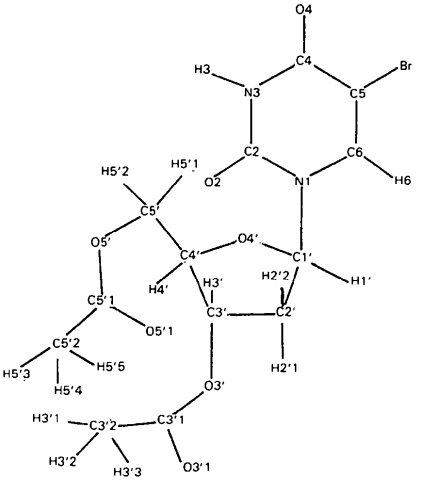

Fig. 1. The atomic numbering for the title compound.

Table 1. *Coordinates* (×10⁴) *and temperature factors* (Å² × 10³) *for non-hydrogen atoms, with e.s.d.'s in parentheses*

	x	*y*	*z*	*U*
Br(1)	3841 (1)	5223	−395 (1)	*
N(1)	854 (7)	5100 (28)	1401 (6)	56 (3)
C(2)	489 (8)	5223 (40)	844 (8)	39 (3)
O(2)	−360 (6)	5019 (21)	1145 (6)	26 (2)
N(3)	1183 (7)	5161 (30)	−108 (7)	57 (3)
C(4)	2183 (8)	5188 (41)	−524 (8)	38 (3)
O(4)	2721 (7)	5341 (31)	−1372 (7)	39 (2)
C(5)	2495 (8)	5186 (39)	114 (8)	33 (3)
C(6)	1852 (8)	5197 (37)	1019 (8)	36 (3)
C(1′)	183 (8)	5058 (30)	2415 (8)	37 (3)
C(2′)	384 (11)	3361 (26)	2849 (11)	34 (4)
C(3′)	54 (12)	4197 (27)	3785 (12)	36 (4)
O(3′)	−952 (6)	3969 (15)	4474 (6)	18 (2)
C(3′)1	−1258 (10)	2117 (27)	4843 (11)	46 (4)
C(3′)2	−2307 (13)	2072 (33)	5503 (12)	66 (5)
O(3′)1	−742 (9)	694 (21)	4614 (8)	50 (3)
C(4′)	261 (10)	6414 (24)	3622 (10)	28 (4)
C(5′)	1175 (10)	7188 (25)	3538 (10)	43 (4)
O(5′)	1985 (6)	5832 (14)	2883 (6)	26 (3)
C(5′)1	2829 (12)	6134 (27)	2840 (12)	54 (4)
C(5′)2	3589 (13)	4832 (34)	2130 (12)	75 (6)
O(5′)1	2916 (9)	7293 (22)	3297 (9)	55 (4)
O(4′)	298 (8)	6808 (19)	2777 (9)	27 (3)

* Refined anisotropically with parameters:

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Br(1)	41 (1)	30 (2)	56 (1)	2 (1)	−10 (1)	−4 (1)

Table 2. *Interatomic distances* (Å), *angles* (°) *and selected torsion angles* (°)

C(5)–Br(1)	1·884 (11)	C(3′)–C(2′)	1·502 (22)
C(2)–N(1)	1·385 (15)	O(3′)–C(3′)	1·424 (18)
C(6)–N(1)	1·398 (13)	C(4′)–C(3′)	1·505 (18)
C(1′)–N(1)	1·474 (14)	C(3′)1–O(3′)	1·351 (20)
O(2)–C(2)	1·202 (14)	C(3′)2–C(3′)1	1·460 (21)
N(3)–C(2)	1·398 (15)	O(3′)1–C(3′)1	1·186 (19)
C(4)–N(3)	1·389 (14)	C(5′)–C(4′)	1·519 (21)
O(4)–C(4)	1·232 (14)	O(4′)–C(4′)	1·449 (18)
C(5)–C(4)	1·446 (17)	O(5′)–C(5′)	1·491 (17)
C(6)–C(5)	1·324 (15)	C(5′)1–O(5′)	1·362 (18)
C(2′)–C(1′)	1·487 (23)	C(5′)2–C(5′)1	1·463 (23)
O(4′)–C(1′)	1·381 (22)	O(5′)1–C(5′)1	1·167 (19)
C(6)–N(1)–C(2)	120·9 (10)	C(3′)–C(2′)–C(1′)	102·1 (13)
C(1′)–N(1)–C(2)	119·8 (9)	O(3′)–C(3′)–C(2′)	115·6 (13)
C(1′)–N(1)–C(6)	119·1 (9)	C(4′)–C(3′)–C(2′)	104·5 (14)
O(2)–C(2)–N(1)	122·3 (11)	C(4′)–C(3′)–O(3′)	107·5 (13)
N(3)–C(2)–N(1)	115·2 (10)	C(3′)1–O(3′)–C(3′)	117·0 (12)
N(3)–C(2)–O(2)	121·1 (11)	C(3′)2–C(3′)1–O(3′)	111·3 (15)
C(4)–N(3)–C(2)	125·8 (10)	O(3′)1–C(3′)1–O(3′)	123·7 (13)
O(4)–C(4)–N(3)	119·7 (11)	O(3′)1–C(3′)1–C(3′)2	124·8 (17)
C(5)–C(4)–N(3)	114·7 (10)	C(5′)–C(4′)–C(3′)	117·4 (14)
C(5)–C(4)–O(4)	125·4 (11)	O(4′)–C(4′)–C(3′)	104·2 (14)
C(4)–C(5)–Br(1)	117·4 (8)	O(4′)–C(4′)–C(5′)	110·3 (12)
C(6)–C(5)–Br(1)	121·9 (9)	O(5′)–C(5′)–C(4′)	107·2 (12)
C(6)–C(5)–C(4)	120·8 (11)	C(5′)1–O(5′)–C(5′)	114·5 (11)
C(5)–C(6)–N(1)	122·1 (11)	C(5′)2–C(5′)1–O(5′)	109·8 (15)
C(2′)–C(1′)–N(1)	112·5 (13)	O(5′)1–C(5′)1–O(5′)	123·8 (16)
O(4′)–C(1′)–N(1)	108·8 (14)	O(5′)1–C(5′)1–C(5′)2	126·4 (17)
O(4′)–C(1′)–C(2′)	106·9 (9)	C(4′)–O(4′)–C(1′)	111·2 (12)
C(4′)–O(4′)–C(1′)–C(2′)	−18·1 (15)	O(3′)–C(3′)–C(4′)–C(5′)	136·7 (14)
O(4′)–C(1′)–C(2′)–C(3′)	31·2 (15)	C(3′)–C(4′)–C(5′)–O(5′)	47·7 (14)
C(1′)–C(2′)–C(3′)–C(4′)	−32·2 (15)	O(5′)–C(5′)–C(4′)–O(4′)	−71·5 (12)
C(2′)–C(3′)–C(4′)–O(4′)	22·3 (14)	C(4′)–C(5′)–O(5′)–C(5′)1	−170·4 (16)
C(3′)–C(4′)–O(4′)–C(1′)	−2·9 (14)	C(5′)–O(5′)–C(5′)1–C(5′)2	−175·8 (18)
C(2)–N(1)–C(1′)–O(4′)	−113·0 (14)	C(4′)–C(3′)–O(3′)–C(3′)1	−168·5 (13)
		C(3′)–O(3′)–C(3′)1–C(3′)2	−177·1 (18)

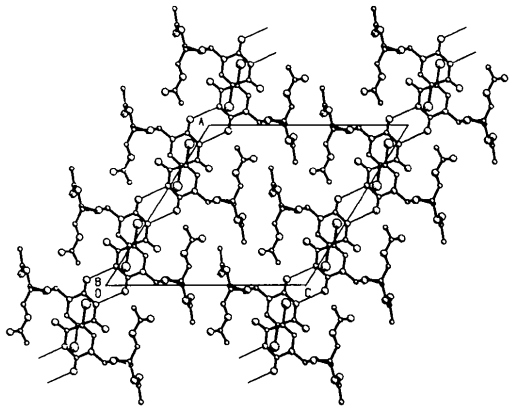

Fig. 2. A view along **b** of the unit-cell contents, showing hydrogen bonding and stacking.

Discussion. The atomic parameters are given in Table 1.* Interatomic distances and angles are given in Table 2. These bonds and angles compare well with those of similar compounds, *e.g.* 3′,5′-di-*O*-acetyluridine (de Graaff, Admiraal, Koen & Romers, 1977). The value of the phase angle of pseudorotation $P = 166°$ (Altona & Sundaralingam 1972), and $\chi_{CN} = -113°$ [C(2)–N(1)–C(1′)–O(4′)]. The C(4′)–C(5′) conformation is g^+ (see Table 2). Figs. 2 and 3† illustrate the packing of the molecules in the cell. The only hydrogen bond present in the structure is that between O(2) and N(3) of bases related to one another by the twofold axis. The interatomic distance for these two atoms is 2·77 (2) Å.

The author wishes to thank Dr P. Tollin and Dr H. R. Wilson for their encouragement and interest in this work.

* Lists of structure factors, H-atom parameters and Fig. 3 [a view perpendicular to the (100) plane] have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38425 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

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

GRAAFF, R. A. G. DE, ADMIRAAL, G., KOEN, E. H. & ROMERS, C. (1977). *Acta Cryst.* B**33**, 2459–2464.

LOW, J. N., TOLLIN, P. & WILSON, H. R. (1982). *Nucl. Acid. Res.* **10**, 5599–5604.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*78. A program for plotting molecular and crystal structures. Univ. of Cambridge, England.

ROBERTS, P. & SHELDRICK, G. M. (1975). *XANADU*. A program for torsion-angle, mean-plane and libration-correction calculations. Univ. of Cambridge, England.

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

Acta Cryst. (1983). C**39**, 798–801

Structure of (*Z*)-1,3,4,5,6-Penta-*O*-acetyl-*keto*-D-fructose (2,4-Dinitrophenyl)hydrazone, $C_{22}H_{26}N_4O_{14}$

BY BISERKA KOJIĆ-PRODIĆ AND ŽIVA RUŽIĆ-TOROŠ

'Rudjer Bošković' Institute, PO Box 1016, 41001 *Zagreb, Yugoslavia*

AND KAZIMIR LINEK

Institute of Chemistry, Slovak Academy of Sciences, Dúbravská cesta, 84238 *Bratislava, Czechoslovakia*

(*Received* 25 *October* 1982; *accepted* 9 *February* 1983)

Abstract. $M_r = 570·46$, orthorhombic, $P2_12_12_1$, $a = 7·942$ (2), $b = 12·737$ (3), $c = 27·328$ (8) Å, $Z = 4$, $U = 2764·43$ Å^3, $D_x = 1·371$ Mg m^{-3}, Cu $K\alpha$, $\lambda = 1·5418$ Å, $\mu = 1·01$ mm^{-1}, $F(000) = 1192$, room temperature; final $R = 0·057$ for 1664 observed reflexions [$I \geq 3\sigma(I)$]. The sugar occurs in the open-chain form and the compound is a true hydrazone. The C atoms of the sugar backbone [from C(2) to C(6)] form a nearly planar zigzag chain. N(1)–N(2)–C(1)–C(2)–C(3) are roughly planar and coplanar with the benzene ring. The sugar conformation is stabilized by an intramolecular hydrogen bond between the hydrazone moiety and the nitro group [N(1)–H···O(1′) 2·613 (7) Å].

Introduction. Phenylhydrazones of the sugars may appear in cyclic or acyclic forms. The crystal structures of arabinose (4-bromophenyl)hydrazone (Furberg & Petersen, 1962), glucose (4-bromophenyl)hydrazone (Dukefos & Mostad, 1965), ribose (4-bromophenyl)hydrazone (Bjåmer, Furberg & Petersen, 1964) and mannose (4-bromophenyl)hydrazone (Furberg & Solbakk, 1969) have been reported. The

0108-2701/83/060798-04$01.50