Table 3. *Positional parameters* ($\times 10^4$) *and equivalent isotropic temperature factors* ($Å^2 \times 10^3$) *for* (2)

	x	y	z	U_{eq}
Cl(1)	−2188 (1)	−1347 (1)	5317 (1)	68 (1)*
O(1)	−3598 (3)	−2000 (3)	1111 (2)	48 (1)*
O(4)	1553 (3)	−3687 (3)	2810 (2)	59 (1)*
C(2)	−3901 (4)	−2333 (4)	2478 (3)	45 (1)*
C(3)	−2298 (4)	−2863 (4)	3102 (2)	39 (1)*
C(4)	10 (4)	−3153 (4)	2291 (3)	41 (1)*
C(4a)	347 (4)	−2797 (3)	805 (2)	36 (1)*
C(5)	2463 (4)	−3019 (4)	−122 (3)	46 (1)*
C(6)	2736 (5)	−2713 (4)	−1502 (3)	55 (1)*
C(7)	896 (6)	−2167 (4)	−1999 (3)	57 (1)*
C(8)	−1194 (5)	−1927 (4)	−1125 (3)	50 (1)*
C(8a)	−1454 (4)	−2242 (4)	272 (2)	40 (1)*
C(9)	−2879 (5)	−3201 (5)	4626 (3)	51 (1)*

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

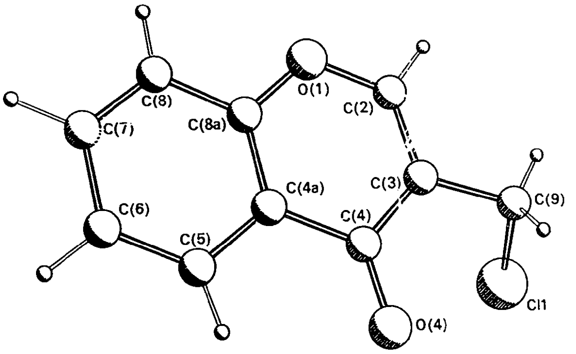

Fig. 2. Drawing showing the atom-numbering scheme of (2).

Table 4. *Bond lengths* (Å) *and bond angles* (°) *for* (2)

Cl(1)—C(9)	1·807 (4)	O(1)—C(2)	1·355 (3)
O(1)—C(8a)	1·384 (3)	O(4)—C(4)	1·234 (4)
C(2)—C(3)	1·339 (5)	C(3)—C(4)	1·460 (3)
C(3)—C(9)	1·496 (4)	C(4)—C(4a)	1·472 (4)
C(4a)—C(5)	1·404 (4)	C(4a)—C(8a)	1·392 (4)
C(5)—C(6)	1·371 (4)	C(6)—C(7)	1·392 (6)
C(7)—C(8)	1·369 (4)	C(8)—(C8a)	1·390 (4)

C(2)—O(1)—C(8a)	117·9 (2)	O(1)—C(2)—C(3)	125·7 (2)
C(9)—C(3)—C(4)	120·3 (3)	C(9)—C(3)—C(2)	119·9 (2)
C(2)—C(3)—C(4)	119·8 (2)	O(4)—C(4)—C(3)	122·6 (2)
O(4)—C(4)—C(4a)	122·7 (2)	C(3)—C(4)—C(4a)	114·7 (2)
C(4)—C(4a)—C(5)	122·0 (3)	C(4)—C(4a)—C(8a)	120·4 (2)
C(5)—C(4a)—C(8a)	117·6 (2)	C(4a)—C(5)—C(6)	121·0 (3)
C(5)—C(6)—C(7)	119·9 (3)	C(6)—C(7)—C(8)	120·8 (3)
C(7)—C(8)—C(8a)	118·9 (3)	O(1)—C(8a)—C(4a)	121·5 (2)
O(1)—C(8a)—C(8)	116·7 (3)	C(4a)—C(8a)—C(8)	121·9 (2)
Cl(1)—C(9)—C(3)	111·4 (2)		

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Structure of 5-Bromouridine: a Redetermination

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Abstract. $C_9H_{11}BrN_2O_6$, $M_r = 323·10$, monoclinic, $P2_1$, $a = 7·722$ (6), $b = 5·809$ (1), $c = 13·259$ (1) Å, $\beta = 101·17$ (7)°, $V = 583·6$ (2) Å^3, $Z = 2$, $D_x = 1·839$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 50·84$ cm^{-1}, $F(000) = 324$, $T = 296$ K, $R = 0·043$, $wR = 0·063$ for 1290 independent reflections with $I > 3\sigma(I)$. The precision of this structure is improved (averaged coordinate e.s.d.'s are smaller by a factor of three) by comparison with the previous report [$R = 0·074$, for 1312 reflections; Iball, Morgan & Wilson (1966). *Proc. R. Soc. London*, **295**, 320–333]. This study is a pre-requisite for using the sample as a test case for anomalous-dispersion studies at a synchrotron.

Experimental. 5-Bromouridine was obtained from Aldrich Chemical Co. Thin colourless plates, typically 1·5 × 0·5 × 0·1 mm, grown from aqueous solution by slow evaporation at room temperature. Sample glued on a glass fibre for data collection. Space group determined from systematic absences, $0k0$: $k = 2n$, Rigaku AFC5 diffractometer on an Ru200 rotating anode, graphite monochromator, Cu $K\alpha$ radiation, 50 kV 180 mA, 23 reflections (77 <

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$2\theta < 80°$) for accurate unit-cell parameters, 1416 measurements, 1324 independent reflections ($R_{\rm int}$ = 0·015), $-8 \le h \le 9$, $-6 \le k \le 6$, $-16 \le l \le 15$ from $\omega/2\theta$ scans, width (1·37 + 0·30 tanθ)°, $2\theta_{\rm max}$ = 160°, 1290 reflections with $I > 3\sigma(I)$ considered observed, $26\bar{1}$ and 1,5,10 reflections omitted during refinement due to suspected extinction, intensity decline by 0·20% on three standard reflections measured every 150 reflections, linear absorption correction applied, transmission factors (0·36 to 1·0), secondary-extinction coefficient corrections applied. Patterson method (*PHASE*; Calabrese, 1972) for initial determination of Br-atom position and direct methods for structure expansion (*DIRDIF*; Beurskens *et al.*, 1984) and (*MITHRIL*, Gilmore, 1984); full-matrix least-squares refinement (Busing, Martin & Levy, 1962), anisotropic parameters for non-H atoms; $\sum w|\Delta F^2|$ minimized; all H atoms located from difference Fourier syntheses and refined isotropically; final R = 0·043, wR = 0·063; $w = [4F_o^2/\sigma^2(F_o)^2]$; $\Delta\rho_{\rm max}$ = 0·87, $\Delta\rho_{\rm min}$ = −0·80 e Å^{-3}, maximum density in proximity of Br atom; neutral atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2A); anomalous-dispersion correction included in F_c (Ibers & Hamilton, 1964); calculations performed using *TEXSAN* (Molecular Structure Corporation; 1985); diagrams obtained with *PLUTO* (Motherwell & Clegg, 1978) and *ORTEP* (Johnson, 1976).

Final atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.* Bond distances are in Table 2. A perspective view of the molecule is shown in Fig. 1.

Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

	x	y	z	$B_{\rm eq}$
Br(1)	0·18893 (8)	0·7284	0·54214 (5)	3·11 (3)
O(1′)	−0·1985 (6)	1·099 (1)	0·2121 (4)	3·1 (2)
O(2)	0·2348 (8)	1·424 (1)	0·2256 (4)	3·8 (2)
O(2′)	0·0797 (7)	0·923 (1)	0·0431 (4)	3·4 (2)
O(3′)	−0·2729 (7)	1·021 (1)	−0·0244 (3)	3·4 (2)
O(5′)	−0·239 (1)	0·622 (2)	0·247 (6)	7·2 (4)
O(6)	0·4312 (6)	1·1553 (9)	0·5468 (3)	2·6 (2)
N(1)	0·3337 (7)	1·276 (1)	0·3841 (4)	2·5 (2)
N(3)	0·105 (7)	1·095 (1)	0·2730 (4)	2·3 (2)
C(1′)	−0·0344 (8)	1·10 (1)	0·1796 (5)	2·3 (2)
C(2)	0·2236 (8)	1·275 (1)	0·2892 (4)	2·4 (2)
C(2′)	−0·0353 (9)	0·888 (1)	0·1121 (5)	2·5 (2)
C(3′)	−0·2314 (9)	0·871 (1)	0·0612 (5)	2·8 (2)
C(4)	0·0996 (8)	0·937 (1)	0·3455 (5)	2·1 (2)
C(4′)	−0·3236 (9)	0·956 (2)	0·1454 (5)	3·4 (3)
C(5)	0·2074 (7)	0·944 (1)	0·4370 (4)	2·0 (2)
C(5′)	−0·385 (1)	0·767 (3)	0·2099 (7)	6·4 (6)
C(6)	0·3340 (7)	1·128 (2)	0·4637 (4)	2·1 (2)

Table 2. *Bond lengths* (Å) *with e.s.d.'s given in parentheses*

Br(1)—C(5)	1·901 (6)	N(3)—C(1′)	1·475 (7)
O(1′)—C(1′)	1·416 (7)	N(3)—C(2)	1·380 (9)
O(1′)—C(4′)	1·439 (9)	N(3)—C(4)	1·337 (9)
O(2)—C(2)	1·223 (9)	C(1′)—C(2′)	1·52 (1)
O(2′)—C(2′)	1·408 (8)	C(2′)—C(3′)	1·538 (9)
O(3′)—C(3′)	1·415 (9)	C(3′)—C(4′)	1·52 (1)
O(5′)—C(5′)	1·41 (2)	C(4)—C(5)	1·333 (8)
O(6)—C(6)	1·217 (7)	C(4′)—C(5′)	1·52 (2)
N(1)—C(2)	1·376 (7)	C(5)—C(6)	1·44 (1)
N(1)—C(6)	1·363 (9)		

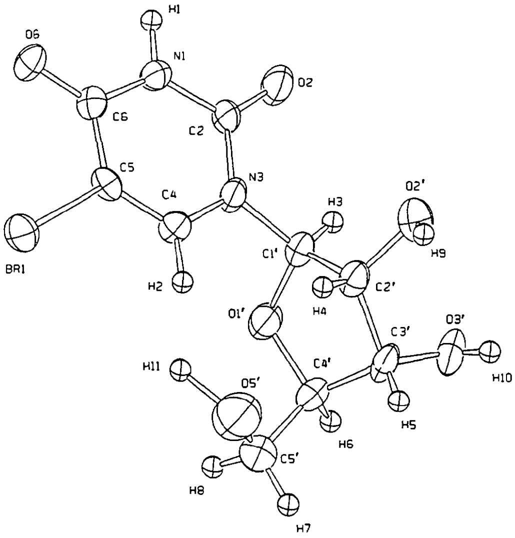

Fig. 1. The molecular structure of the bromouridine molecule with 50% probability ellipsoids for the non-H atoms.

Related literature. The structure consists of a pyrimidine base and a furanose ring. The mean deviation from the best plane through the six-membered ring and attached Br and O atoms is 0·0245 Å. The sugar ring is puckered with C2′ lying 0·55 Å out of the plane C1′ O1′ C3′ C4′. The bond lengths and angles agree well with literature values (Saenger, 1984, and references therein). The hydrogen-bonding network is as previously determined by Iball, Morgan & Wilson (1966). Here, distances between O(2′)···O(3′), O(3′)···O(2) and N(1)···O(6) are 2·811, 2·797 and 2·889 Å, respectively; angle O(2′)—H(9)···O(3′) is 149·27, O(3′)—H(10)···O(2) 152·50 and N(1)—H(1)···O(6) 161·66°. In this work, the H atom attached to O(5′) of the sugar ring [H(11)] was located in a region of relatively high flexibility. The R factor was reduced, by comparison with the earlier study, using a comparable number of reflections; the average coordinate e.s.d.'s were improved by a factor of three.

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* Lists of structure factors, anisotropic thermal parameters, intermolecular and intramolecular distances involving H atoms, intermolecular distances and intramolecular bond angles involving non-H atoms, torsion angles, and H-atom parameters and a unit-cell packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53793 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Désoxy-6 *N*,*N*-Diméthyldithiocarbamoyl-6 Di-*O*-isopropylidène-1,2:3,4 α-D-Galactopyrannose

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(*Reçu le* 14 *juin* 1990, *accepté le* 3 *décembre* 1990)

Abstract. $C_{15}H_{25}NO_5S_2$, $M_r = 363{\cdot}5$, monoclinic, $P2_1$, $a = 9{\cdot}803$ (5), $b = 10{\cdot}50$ (1), $c = 10{\cdot}090$ (3) Å, $\beta = 116{\cdot}38$ (3)°, $V = 930$ (2) Å^3, $Z = 2$, $D_x = 1{\cdot}297$ Mg m^{-3}, $\lambda(\text{Mo } K\bar{\alpha}) = 0{\cdot}7107$ Å, $\mu = 0{\cdot}295$ mm^{-1}, $F(000) = 388$, $T = 294$ (1) K, $R = 0{\cdot}042$ for 1380 independent observed reflections. As expected, the dimethyldithiocarbamoyl group is approximately planar. In this group, the shortest C—S and C—N distances, 1·658 (5) and 1·321 (6) Å respectively, indicate the presence of a delocalized orbital. One of the two five-membered rings (*A* ring) has an envelope form and the other (*C* ring) a half-chair form. The pyranosyl ring has a skew-boat conformation. The cohesion of the structure is due to van der Waals interactions.

Partie expérimentale. Cristal approximativement parallélépipédique: 0,24 × 0,32 × 0,40 mm. Diffractomètre Enraf–Nonius CAD-4. Dimensions de la maille déterminées sur monocristal avec 25 réflexions telles que $7{,}08 \le \theta \le 13{,}01°$. $0{,}039 \le (\sin\theta)/\lambda \le 0{,}639$ Å^{-1}. Balayage $\theta/2\theta$ d'amplitude $s(°) = 0{,}80 + 0{,}35\text{tg}\theta$. $0 \le h \le 12$, $0 \le k \le 13$, $-12 \le l \le 11$. Réflexions de contrôle de l'intensité: $30\bar{4}$, $\bar{1}2\bar{2}$ et $\bar{4}12$. Variations non significatives de I au cours des mesures. $\sigma(I)/I$ moyen (contrôle): 0,0068. 2144 réflexions indépendantes mesurées, 764 inobservées $[I < 2\sigma(I)]$. Méthodes directes, programme *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) et série de Fourier des ΔF. Affinement basé sur les F, programme à matrice complète. Facteurs de diffusion des *International Tables for X-ray Crystallography* (1974, Tome IV, pp. 99, 149). Paramètres affinés: coordonnées de tous les atomes et β_{ij} de S, O, N et C. $B(\text{H}) = B_{\text{éq}}$ de l'atome lié à H + 1 Å^2. $R = 0{,}042$, $wR = 0{,}040$, $w = 1/\sigma^2(F)$, $S = 1{,}19$, $(\Delta/\sigma)_{\max} = 0{\cdot}05$, $|\Delta\rho|_{\max} = 0{,}21$ (5) e Å^{-3}. Le pic résiduel le plus élevé se trouve

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