

Related literature. Urea–oxalic acid (2/1) is the second known structure of complexes between urea and oxalic acid (Harkema, ter Brake & Helmholtz, 1979) and was studied in the framework of the accurate electron density of oxalic acid complexes (Coppens, 1984). The aim is to determine the accurate hydrogen positions in combination with the thermal parameters. Non-H-atom positions and thermal parameters are in good agreement with those obtained by a new X-ray diffraction experiment (Harkema, van Hummel, Peerdeman, Feil & Helmholtz, 1990).

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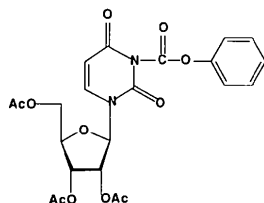
Structure of 2',3',5'-Tri-*O*-acetyl-*N*³-phenoxycarbonyluridine

BY NORIAKI HIRAYAMA

Tokyo Research Laboratories, Kyowa Hakko Kogyo Co. Ltd, 3-6-6 Asahimachi, Machida, Tokyo 194, Japan

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Abstract. C₂₂H₂₂N₂O₁₁, *M_r* = 490.41, monoclinic, *P*2₁, *a* = 9.870 (1), *b* = 15.421 (2), *c* = 8.059 (1) Å, β = 110.28 (1)°, *V* = 1150.6 (3) Å³, *Z* = 2, *D_x* = 1.42 g cm⁻³, Cu *Kα*, λ = 1.54184 Å, μ = 9.42 cm⁻¹, *F*(000) = 512, *T* = 293 K, *wR* = 0.057 for 2345 observed reflections with *I* > 3σ(*I*). The sugar conformation and puckering parameters are ³*E* (C3'-endo), *P* = 16.0° and τ_{*m*} = 38.8°. The C4'-C5' side-chain orientation is *gauche-gauche* [φ_{OO} = -57.5 (2); φ_{OC} = 59.7 (2)°]. The glycosidic torsion angle is -160.0 (2)° corresponding to the *anti* conformation. The dihedral angle between the pyrimidine and benzene rings is 15.1 (3)°.



Experimental. Crystal dimensions 0.40 × 0.30 × 0.25 mm. Enraf–Nonius CAD-4 diffractometer,

graphite-monochromated Cu *Kα* radiation. Cell dimensions from setting angles of 25 independent reflections with 47 ≤ θ ≤ 55°. ω–2θ scans. Reflections surveyed in the range 2 ≤ 2θ ≤ 150° (Cu *Kα*); -12 ≤ *h* ≤ 11, 0 ≤ *k* ≤ 19, 0 ≤ *l* ≤ 9; 2352 reflections were unique, 2345 observed with *I* > 3σ(*I*). Three reference reflections monitored periodically showed no significant variation in intensity. Absorption correction was not applied. Structure solved using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier-map recycling. Refinement using the *SDP* package (Frenz, 1985), full-matrix least-squares refinement on *F*, with non-H atoms having anisotropic temperature factors. Most of the H atoms were located from difference Fourier syntheses and were refined with isotropic temperature parameters. *w* = 4*F_o*²/[σ²(*I_o*) + (0.04*I_o*)²]^{1/2}/Lp, final *R* = 0.040, *wR* = 0.057, *S* = 2.22, maximum shift/e.s.d. in the final least-squares cycle 0.30, maximum peak in the final difference map 0.27 (4) e Å⁻³. Secondary-extinction coefficient refined (final value of 9.5 × 10⁻⁶). Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final fractional coordinates and

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Table 1. Positional parameters and equivalent isotropic temperature factors of non-H atoms with *e.s.d.*'s in parentheses

$$B_{eq} = (4/3) \sum_i \beta_{ij} a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq} (Å ²)
O(2)	0.8995 (1)	0.4261 (1)	1.1150 (2)	4.57 (3)
O(3)	0.8429 (2)	0.3907 (1)	1.4212 (2)	4.23 (3)
O(4)	0.8289 (1)	0.235	1.0906 (2)	3.75 (3)
O(5)	0.7257 (2)	0.1501 (1)	1.3359 (2)	4.51 (3)
O(2)	0.6063 (2)	0.3858 (1)	0.6761 (2)	5.01 (3)
O(4)	0.2165 (2)	0.2089 (2)	0.5406 (3)	5.29 (4)
O(21')	0.7853 (2)	0.5510 (2)	1.0223 (4)	7.30 (6)
O(31')	0.6512 (2)	0.4786 (1)	1.3401 (2)	4.82 (3)
O(51')	0.7057 (3)	0.1861 (3)	1.5935 (3)	7.91 (6)
O(31)	0.2732 (2)	0.4054 (2)	0.4391 (2)	6.24 (4)
O(32)	0.3725 (2)	0.3022 (1)	0.3203 (2)	4.78 (3)
N(1)	0.6106 (1)	0.2776 (1)	0.8706 (2)	3.19 (3)
N(3)	0.4128 (2)	0.2959 (1)	0.6130 (2)	3.71 (3)
C(1)	0.7613 (2)	0.3030 (1)	0.9755 (2)	3.35 (3)
C(2)	0.7657 (2)	0.3825 (1)	1.0906 (3)	3.50 (3)
C(3)	0.7758 (2)	0.3400 (1)	1.2659 (2)	3.43 (3)
C(4)	0.8732 (2)	0.2626 (1)	1.2719 (2)	3.48 (3)
C(5)	0.8683 (2)	0.1874 (2)	1.3872 (3)	4.45 (4)
C(2)	0.5482 (2)	0.3241 (1)	0.7175 (2)	3.58 (3)
C(4)	0.3353 (2)	0.2255 (2)	0.6472 (3)	3.92 (4)
C(5)	0.4101 (2)	0.1819 (2)	0.8112 (3)	4.29 (4)
C(6)	0.5420 (2)	0.2078 (1)	0.9131 (2)	3.66 (3)
C(21')	0.8961 (2)	0.5113 (1)	1.0814 (3)	4.06 (4)
C(22')	1.0459 (3)	0.5451 (2)	1.1157 (5)	6.15 (6)
C(31')	0.7659 (2)	0.4593 (1)	1.4449 (3)	3.65 (3)
C(32')	0.8420 (3)	0.5054 (2)	1.6137 (3)	4.79 (5)
C(51')	0.6581 (3)	0.1502 (2)	1.4550 (3)	4.58 (4)
C(52')	0.5184 (3)	0.1016 (2)	1.3876 (4)	6.07 (6)
C(31)	0.3436 (2)	0.3427 (2)	0.4492 (3)	4.23 (4)
C(32)	0.2991 (2)	0.3306 (2)	0.1451 (3)	4.30 (4)
C(33)	0.3515 (3)	0.3982 (2)	0.0779 (4)	5.55 (6)
C(34)	0.2821 (4)	0.4173 (3)	-0.1001 (4)	7.14 (8)
C(35)	0.1647 (3)	0.3702 (3)	-0.2017 (3)	7.14 (7)
C(36)	0.1143 (3)	0.3045 (3)	-0.1292 (4)	7.33 (8)
C(37)	0.1808 (3)	0.2819 (3)	0.0464 (4)	5.86 (6)

equivalent *B* values are listed in Table 1. Distances and angles are listed in Table 2.* Fig. 1 shows an ORTEPII (Johnson, 1976) drawing of the molecule with the atomic numbering.

Related literature. Although diisopropylethylamine is effective as a base for acylation of 2',3',5'-tri-*O*-acetyluridine with various acid chlorides, it was not certain whether the acyl groups are attached to N3 or O4 of the uracil moiety. The ¹³C NMR spectra of the products and related compounds showed that the acyl groups are attached to N3 (Kamimura, Masegi, Sekine & Hata, 1984). We have undertaken the present analysis to verify the assignment and assess the effect of the large substituent at N3 on the geometry of the uridine moiety.

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* Lists of structure factors, anisotropic thermal parameters, torsion angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53323 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Selected bond lengths (Å) and angles (°)

O(2)—C(2)	1.433 (2)	N(1)—C(6)	1.378 (3)
O(3)—C(3)	1.429 (2)	N(3)—C(2)	1.380 (2)
O(4)—C(1)	1.409 (3)	N(3)—C(4)	1.409 (4)
O(4)—C(4)	1.438 (2)	N(3)—C(31)	1.451 (3)
O(5)—C(5)	1.443 (3)	C(1)—C(2)	1.528 (3)
O(2)—C(2)	1.215 (3)	C(2)—C(3)	1.530 (3)
O(4)—C(4)	1.216 (2)	C(3)—C(4)	1.524 (3)
O(31)—C(31)	1.176 (3)	C(4)—C(5)	1.496 (3)
O(32)—C(31)	1.326 (4)	C(4)—C(5)	1.438 (3)
N(1)—C(1)	1.486 (2)	C(5)—C(6)	1.337 (3)
N(1)—C(2)	1.374 (2)		
C(1)—O(4)—C(4)	110.7 (1)	O(4)—C(4)—C(3)	103.6 (1)
C(1)—N(1)—C(2)	115.0 (2)	O(4)—C(4)—C(5)	109.6 (2)
C(1)—N(1)—C(6)	123.1 (1)	C(3)—C(4)—C(5)	118.1 (2)
C(2)—N(1)—C(6)	121.6 (1)	O(5)—C(5)—C(4)	111.7 (2)
C(2)—N(3)—C(4)	126.9 (2)	O(2)—C(2)—N(1)	123.2 (2)
C(2)—N(3)—C(31)	116.8 (2)	O(2)—C(2)—N(3)	121.9 (2)
C(4)—N(3)—C(31)	116.4 (2)	N(1)—C(2)—N(3)	115.0 (2)
O(4)—C(1)—N(1)	109.2 (2)	O(4)—C(4)—N(3)	118.7 (2)
O(4)—C(1)—C(2)	107.1 (1)	O(4)—C(4)—C(5)	127.8 (2)
N(1)—C(1)—C(2)	111.7 (1)	N(3)—C(4)—C(5)	113.5 (2)
O(2)—C(2)—C(1)	106.9 (2)	C(4)—C(5)—C(6)	120.4 (2)
O(2)—C(2)—C(3)	108.2 (1)	N(1)—C(6)—C(5)	122.6 (2)
C(1)—C(2)—C(3)	101.3 (2)	O(31)—C(31)—O(32)	128.0 (3)
O(3)—C(3)—C(2)	115.8 (2)	O(31)—C(31)—N(3)	123.5 (2)
O(3)—C(3)—C(4)	107.8 (1)	O(32)—C(31)—N(3)	108.6 (2)
C(2)—C(3)—C(4)	101.8 (2)		

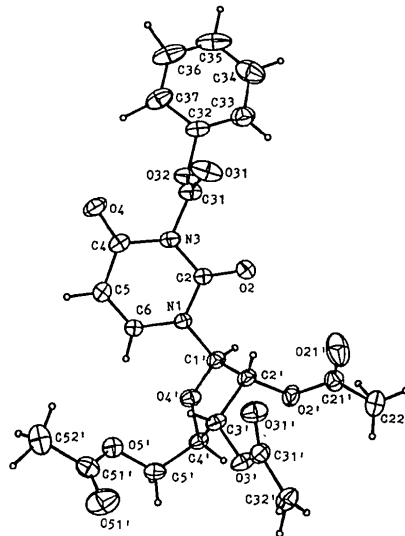


Fig. 1. An ORTEPII (Johnson, 1976) drawing of the molecule with the numbering system.

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