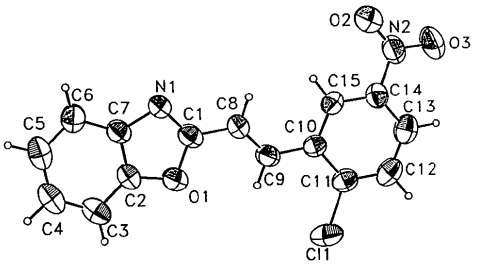

Fig. 1. Labeling of atoms in 2-(2-chloro-5-nitrostyryl)benzoxazole (50% probability ellipsoids).

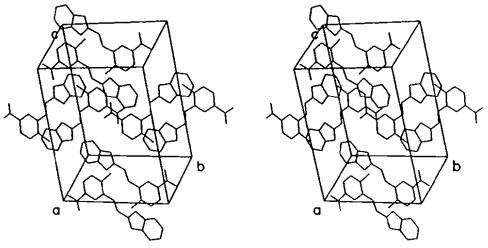

Fig. 2. Packing diagram of 2-(2-chloro-5-nitrostyryl)benzoxazole in the unit cell.

nsbo has a somewhat smaller C—O—C angle than that in a 2-benzoxazolethiolato complex of gold(I) [105.4 (7)°; Muir, Cuadrado & Muir, 1989]. The average C—N, C—S and C—C distances and bond angles in (1) are similar to those found in the other compounds.

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Structure of a Hexenopyranosid-4-ulose

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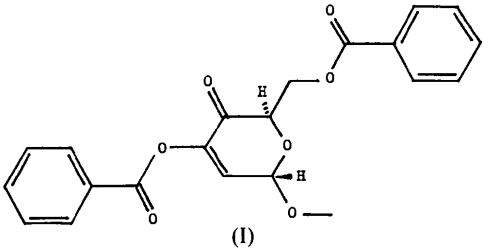
Abstract. Methyl 3,6-di-*O*-benzoyl-2-deoxy-α-D-*glycero*-hex-2-enopyranosid-4-ulose. $C_{21}H_{18}O_7$, M_r = 382.4, monoclinic, $P2_1$, a = 4.206 (1), b = 27.601 (6), c = 8.145 (1) Å, β = 95.31 (2)°, V = 941.5 (3) Å^3, Z = 2, D_x = 1.349 Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, μ = 0.10 mm^{-1}, $F(000)$ = 400, T = 297 K, final R = 0.034 for 1375 observed reflections. The pyranoid conformation is $^OH_5$ with Q = 45.6 pm, θ = 57.8° and φ = 11.5° [Cremer & Pople (1975). *J. Am. Chem. Soc.* **97**, 1354–1358]. The two benzoyl groups are equatorial, pointing away from each other with the phenyl rings approximately parallel. All bond lengths and angles are within expected ranges.

Experimental. The title compound (I) was synthesized by treating methyl 2,3,6-tri-*O*-benzoyl-α-D-glucopyranoside with P_2O_5–dimethyl sulfoxide in *N*,*N*-dimethylformamide at 338–343 K for 2.5 h. The



(I)

0108-2701/92/030585-02$03.00

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement coefficients* ($Å^2 \times 10^3$)

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

	x	y	z	U_{eq}
O(1)	7399 (6)	4343	11048 (3)	47 (1)
O(3)	5073 (8)	3891 (1)	5750 (3)	53 (1)
O(4)	4123 (7)	4849 (1)	5719 (3)	52 (1)
O(5)	9979 (5)	4773 (1)	9098 (3)	41 (1)
O(6)	9209 (7)	5751 (1)	9197 (3)	54 (1)
O(7)	3061 (7)	3300 (2)	7254 (3)	61 (1)
O(8)	11679 (9)	6299 (2)	7796 (3)	68 (1)
C(1)	9376 (8)	4316 (2)	9759 (4)	41 (1)
C(2)	7832 (9)	3984 (2)	8483 (4)	44 (1)
C(3)	6263 (9)	4157 (2)	7108 (5)	43 (1)
C(4)	5729 (8)	4683 (2)	6875 (4)	39 (1)
C(5)	7212 (8)	4997 (2)	8271 (4)	38 (1)
C(6)	8245 (10)	5488 (2)	7705 (4)	42 (1)
C(7)	3625 (9)	3457 (2)	5942 (4)	43 (1)
C(8)	10990 (9)	6145 (2)	9077 (4)	43 (1)
C(1*M*)	8891 (12)	4587 (2)	12476 (5)	65 (2)
C(71)	2804 (9)	3215 (2)	4339 (4)	39 (1)
C(72)	3733 (9)	3401 (2)	2872 (5)	46 (1)
C(73)	2993 (11)	3148 (2)	1416 (5)	56 (2)
C(74)	1283 (11)	2722 (2)	1416 (5)	59 (1)
C(75)	356 (11)	2535 (2)	2873 (6)	57 (1)
C(76)	1064 (10)	2783 (2)	4334 (5)	48 (1)
C(81)	11979 (10)	6354 (2)	10732 (5)	46 (1)
C(82)	11262 (13)	6140 (2)	12171 (5)	67 (2)
C(83)	12279 (15)	6348 (2)	13663 (6)	83 (2)
C(84)	13968 (13)	6770 (2)	13734 (7)	78 (2)
C(85)	14654 (13)	6989 (2)	12295 (7)	71 (2)
C(86)	13730 (10)	6782 (2)	10786 (5)	57 (1)

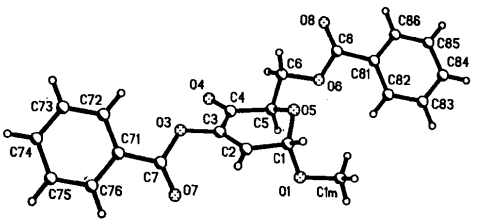


Fig. 1. A perspective view of the molecule with atomic numbering.

resulting solution was poured into ice–water, extracted with dichloroethane, dried, evaporated, and the product recrystallized from ether–hexane (4/1). Colourless prism, $0.5 \times 0.4 \times 0.25$ mm, Siemens *R3m/V* diffractometer, ω scan, lattice parameters obtained from 20 reflections with 2θ values from 8 to 20°. 1805 reflections with $\sin\theta/\lambda < 0.59$ Å^{-1} measured, 1683 independent ($R_{int} = 0.011$), 1375 classified as observed [$I_o > 3\sigma(I)$], $-5 < h < 4$, $0 < k < 30$, $0 < l < 9$. Two standard reflections measured every 98 reflections showed no significant intensity variation. No absorption correction was applied. The structure was solved by direct method using *SHELXTL-Plus* (Siemens Analytical X-ray Instruments Inc., 1990). Refinement was carried out by full-matrix least-squares (on F) method with anisotropic temperature coefficients for non-H atoms. H-atom positions were calculated and fixed with isotropic thermal parameters. Function minimized $\sum w(F_o - F_c)^2$, with $w^{-1} = \sigma^2(F) + 0.0015F^2$, $\sigma(F)$ from counting statistics. The

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

O(1)—C(1)	1.400 (4)	O(1)—C(1*M*)	1.436 (5)
O(3)—C(3)	1.383 (5)	O(3)—C(7)	1.360 (6)
O(4)—C(4)	1.198 (4)	O(5)—C(1)	1.404 (6)
O(5)—C(5)	1.430 (4)	O(6)—C(6)	1.441 (5)
O(6)—C(8)	1.329 (6)	O(7)—C(7)	1.197 (5)
O(8)—C(8)	1.187 (5)	C(1)—C(2)	1.490 (5)
C(2)—C(3)	1.335 (5)	C(3)—C(4)	1.476 (7)
C(4)—C(5)	1.518 (5)	C(5)—C(6)	1.509 (6)
C(7)—C(71)	1.478 (5)	C(8)—C(81)	1.489 (5)
C(71)—C(72)	1.389 (6)	C(71)—C(76)	1.401 (6)
C(72)—C(73)	1.387 (6)	C(73)—C(74)	1.378 (7)
C(74)—C(75)	1.383 (7)	C(75)—C(76)	1.381 (6)
C(81)—C(82)	1.371 (6)	C(81)—C(86)	1.392 (7)
C(82)—C(83)	1.376 (7)	C(83)—C(84)	1.363 (9)
C(84)—C(85)	1.373 (8)	C(85)—C(86)	1.378 (7)

C(1)—O(1)—C(1*M*)	112.8 (3)	C(3)—O(3)—C(7)	120.6 (3)
C(1)—O(5)—C(5)	113.6 (3)	C(6)—O(6)—C(8)	117.8 (3)
O(1)—C(1)—O(5)	112.4 (3)	O(1)—C(1)—C(2)	107.8 (3)
O(5)—C(1)—C(2)	111.6 (3)	C(1)—C(2)—C(3)	120.9 (4)
O(3)—C(3)—C(2)	126.4 (4)	O(3)—C(3)—C(4)	112.3 (3)
C(2)—C(3)—C(4)	121.2 (4)	O(4)—C(4)—C(3)	123.1 (4)
O(4)—C(4)—C(5)	122.2 (4)	C(3)—C(4)—C(5)	114.7 (3)
O(5)—C(5)—C(4)	111.5 (3)	O(5)—C(5)—C(6)	106.7 (3)
C(4)—C(5)—C(6)	113.2 (3)	O(6)—C(6)—C(5)	105.1 (3)
O(3)—C(7)—O(7)	123.3 (4)	O(3)—C(7)—C(71)	111.5 (3)
O(7)—C(7)—C(71)	125.2 (4)	O(6)—C(8)—O(8)	122.9 (4)
O(6)—C(8)—C(81)	111.3 (3)	O(8)—C(8)—C(81)	125.8 (4)
C(7)—C(71)—C(72)	122.2 (4)	C(7)—C(71)—C(76)	117.8 (4)
C(72)—C(71)—C(76)	120.0 (4)	C(71)—C(72)—C(73)	119.5 (4)
C(72)—C(73)—C(74)	120.4 (4)	C(73)—C(74)—C(75)	120.5 (4)
C(74)—C(75)—C(76)	119.9 (5)	C(71)—C(76)—C(75)	119.8 (4)
C(8)—C(81)—C(82)	122.8 (4)	C(8)—C(81)—C(86)	117.4 (4)
C(82)—C(81)—C(86)	119.8 (4)	C(81)—C(82)—C(83)	120.0 (5)
C(82)—C(83)—C(84)	120.9 (5)	C(83)—C(84)—C(85)	119.3 (5)
C(84)—C(85)—C(86)	121.0 (5)	C(81)—C(86)—C(85)	119.0 (4)

final R values are $R = 0.034$ and $wR = 0.051$; $S = 1.12$; 253 parameters refined. Maximum $\Delta/\sigma = 0.64$, $(\Delta\rho)_{max} = 0.16$, $(\Delta\rho)_{min} = -0.12$ e Å^{-3}. All calculations performed on a MicroVAX 2000 computer with *SHELXTL-Plus*. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atomic coordinates, bond lengths and bond angles are given in Tables 1 and 2.* The molecule and atom labelling are shown in Fig. 1.

Related literature. The title compound has been synthesized before by a slightly different procedure (Lichtenthaler, Ogawa & Heidel, 1977). It was proposed that the oxidation of methyl 2,3,6-tri-*O*-benzoyl-α-D-glucopyranoside gave initially the intermediate 4-*xylo*-hexuloside which could not be isolated as a pure sample because it rapidly underwent β-elimination of benzoic acid to yield the title compound.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54688 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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