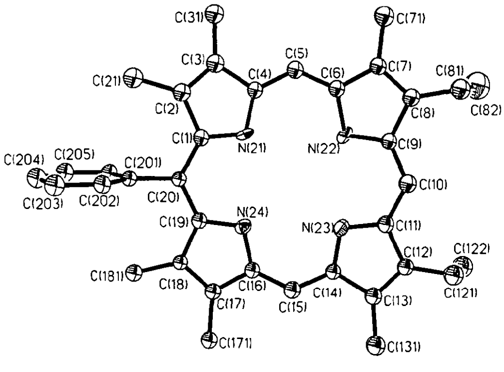

Fig. 1. Molecular structure and numbering scheme for the title compound.

structure is shown in Fig. 1. Fig. 2 gives a stereoview of the molecular packing in the unit cell. Weighting scheme defined as $w^{-1} = \sigma^2(F) + 0.0005F^2$, Δ/σ 0.180, data/parameter ratio 7.9:1, $(\Delta\rho)_{\max} = 0.49$, $(\Delta\rho)_{\min} = -0.36$ e Å^{-1}. Atomic scattering factors from Cromer & Waber (1974).

Related literature. This study presents part of an accumulating body of information on conformational details of the porphyrin macrocycle (Scheidt & Lee, 1987; Barkigia, Berber, Fajer, Medforth, Renner & Smith, 1990). The conformational flexibility is of central importance for the biological role of these chromophores in photosynthesis and electron transport (Barkigia, Chantranupong, Smith & Fajer, 1988). Although the β-substituents are small and only one *meso* position is substituted the molecule shows a slight degree of conformational distortion with the β-pyrrole atoms pushed alternately above and below the mean plane. The *meso* C atom bearing the phenyl substituent is displaced slightly above the mean plane and the plane of the phenyl substituent deviates 5.4° from an orthogonal orientation with respect to the plane of the four N atoms. The molecules are tightly packed in parallel layers with each layer connected to the neighboring one by $\pi - \pi$ stacking of one of the pyrrole rings.

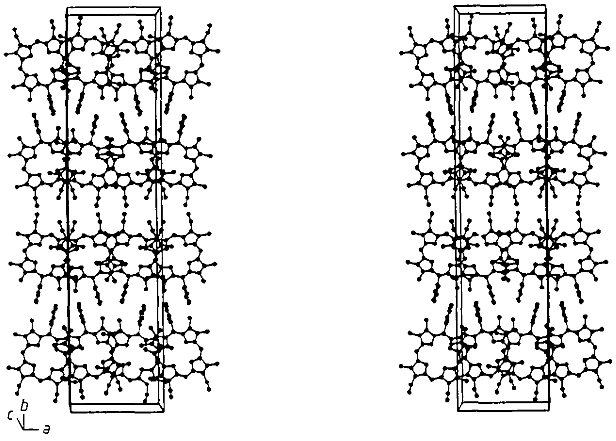

Fig. 2. View of the molecular packing.

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Structure of a Styrylbenzoxazole Derivative

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Abstract. 2-(2-Chloro-5-nitrostyryl)benzoxazole (nsbo), $C_{15}H_9ClN_2O_3$, $M_r = 300.70$, monoclinic, $P2_1/c$, $a = 7.030$ (3), $b = 13.55$ (1), $c = 15.40$ (1) Å, $\beta = 114.77$ (3)°, $V = 1332$ (3) Å^3, $Z = 4$, $D_x = 1.498$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 2.94$ cm^{-1}, $F(000) = 616$, $T = 298$ K, final $R = 0.043$ for 1244 unique observed reflections. The molecule is almost planar, with a dihedral angle of 6.8 (6)° between the benzoxazole and phenyl rings.

Experimental. The oxazole derivative nsbo (1) (X = O) was prepared as part of a study of substitution in

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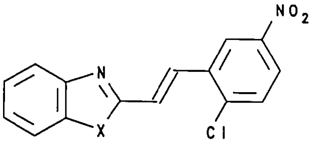
the azole ring ($X = O$, S, Se and NR) (Muir, Cox, Rivera, Cádiz & Medina, 1992). The preparation and characterization of the compound have been described elsewhere (Cox, Jackson, Vargas, Báez, Colón, González & de León, 1982). Analysis for $C_{15}H_9ClN_2O_3$: calculated C 59.91, H 3.00, N 9.32%; found C 59.68, H 3.00, N 9.29%. The solid, m.p. 450–451 K, was recrystallized from dioxane; crystals obtained by slow evaporation from acetonitrile.



(1)

Colorless rectangular plate, D_m not determined, 0.45 × 0.25 × 0.20 mm, Enraf–Nonius CAD-4 diffractometer, monochromated Mo $K\alpha$ radiation, ω–2θ scan, scan width $(0.9 + 0.34\tan\theta)^\circ$, $2\theta_{\max} = 53^\circ$, lattice parameters from least-squares fit of reflections in the range $10 < \theta < 15^\circ$. Systematic absences $h0l$ ($h + l = 2n + 1$), $0k0$ ($k = 2n + 1$); $+h$, $+k$, $\pm l$, $h \leq 8$, $k \leq 17$, $-19 \leq l \leq 17$; three standard reflections (055, 064 and 26$\bar{4}$), total intensity loss 0.4% during 54.7 h exposure, no equivalent reflections measured, total unique reflections measured 2647, 1244 with $F^2 > 3\sigma^2(F_o)$, $\sigma(F_o) = [\sigma^2(I_{\rm raw}) + (0.06F_o^2)^2]^{1/2}/2F_o$. Structure was solved with the Enraf–Nonius *SDP* programs (Frenz, 1986), heavy-atom and difference-Fourier methods; isotropic, then anisotropic refinement on F of all non-H atoms, H atoms at calculated positions, riding model [C—H 0.95 Å, $U({\rm H}) = 1.3U({\rm C})$]; 190 parameters refined, $R = 0.043$, $wR = 0.053$, $w = 1/[\sigma^2(F_o)]$, $S = 1.286$; difference syntheses showed no densities above 0.18 e Å^{-3} (minimum −0.23); maximum $\Delta/\sigma = 0.01$. Neutral-atom scattering factors were used (*International Tables for X-ray Crystallography*, 1974, Vol. IV, Tables 2.2B and 2.3.1), and were corrected for anomalous dispersion (Cromer & Liberman, 1970). No corrections for absorption or secondary extinction.

The atomic coordinates and equivalent isotropic temperature factors are listed in Table 1, and selected bond lengths and angles in Table 2.* An *ORTEP*II plot (Johnson, 1976) of the complex with the atom numbering is shown in Fig. 1, and the packing of the molecules (Motherwell & Clegg, 1976) in Fig. 2.

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (Å^2) *for* 2-(2-*chloro*-5-*nitrostyryl*)-*benzoxazole*

$$B_{\rm eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	$B_{\rm eq}$
Cl1	0.1857 (2)	0.08334 (8)	0.60887 (6)	6.00 (3)
O1	0.2647 (3)	−0.1053 (2)	0.3494 (2)	4.38 (6)
O2	0.2657 (5)	0.4435 (2)	0.3398 (2)	7.52 (8)
O3	0.2485 (4)	0.5285 (2)	0.4532 (2)	7.37 (9)
N1	0.2680 (4)	−0.0351 (2)	0.2178 (2)	3.96 (7)
N2	0.2524 (5)	0.4499 (2)	0.4153 (2)	5.20 (8)
C1	0.2607 (5)	−0.0217 (2)	0.2994 (2)	4.07 (8)
C2	0.2737 (5)	−0.1800 (2)	0.2907 (2)	3.83 (8)
C3	0.2774 (5)	−0.2804 (3)	0.3053 (3)	4.9 (1)
C4	0.2904 (5)	−0.3380 (3)	0.2338 (3)	5.4 (1)
C5	0.2964 (5)	−0.2968 (3)	0.1525 (3)	5.2 (1)
C6	0.2908 (5)	−0.1958 (3)	0.1386 (2)	4.53 (9)
C7	0.2771 (5)	−0.1371 (2)	0.2095 (2)	3.84 (8)
C8	0.2501 (5)	0.0733 (2)	0.3405 (2)	4.06 (8)
C9	0.2532 (5)	0.0863 (2)	0.4259 (2)	3.85 (8)
C10	0.2406 (4)	0.1818 (2)	0.4668 (2)	3.51 (8)
C11	0.2070 (5)	0.1887 (3)	0.5505 (2)	4.13 (8)
C12	0.1877 (5)	0.2785 (3)	0.5884 (2)	5.1 (1)
C13	0.2059 (5)	0.3655 (3)	0.5464 (2)	5.00 (9)
C14	0.2377 (5)	0.3591 (3)	0.4641 (2)	4.08 (9)
C15	0.2558 (5)	0.2703 (2)	0.4253 (2)	3.78 (8)

Table 2. *Selected bond lengths* (Å) *and angles* (°) *in* 2-(2-*chloro*-5-*nitrostyryl*)*benzoxazole*

Cl1—C11	1,728 (4)	N2—C14	1.467 (5)
O1—C1	1.363 (4)	C1—C8	1.449 (5)
O1—C2	1.377 (4)	C8—C9	1.319 (5)
N1—C1	1.292 (5)	C9—C10	1.458 (5)
N1—C7	1.393 (4)	$\rm C—C_{phenyl,\ av.}$	1.383 (8)
$\rm C—C_{benzo,\ av.}$	1.385 (5)	$\rm N—O_{nitro,\ av.}$	1.214 (5)
C1—O1—C2	103.7 (3)	C1—C8—C9	124.9 (3)
C1—N1—C7	104.5 (3)	C8—C9—C10	124.8 (3)
O1—C1—N1	115.6 (3)	O1—C1—C8	118.9 (3)
N1—C7—C2	108.3 (3)	N1—C1—C8	125.4 (3)
O1—C2—C7	107.8 (3)	O2—N2—O3	123.2 (3)
$\rm C—C—C_{phenyl,\ av.}$	120.0 (4)	$\rm C—C—C_{benzo,\ av.}$	120.0 (3)
$\rm C14—N2—O_{av.}$	118.4 (3)		

Related literature. Derivatives of 2-styrylbenzazoles have been studied because of their importance as precursors to the biologically active benzazolo[3,2-*a*]-quinolinium salts (Cox, Jackson, Vargas, Báez, Colón, González & de León, 1982). Derivatives include S, O, Se and N analogues. Moreover, two platinum(II) complexes of the type $[{\rm Pt}(L){\rm Br_3}]{\rm NEt_4}$, L = 2-(2-hydroxystyryl)- and 2-(2,5-dichloro-5-nitrostyryl)benzothiazole, respectively, show good cytotoxic activity against CHO-K cells *in vitro* (Muir, Cádiz & Báez, 1988). The X-ray crystal structures of several Pt$^{\rm II}$ styrylbenzothiazole complexes have been determined (Muir, Gómez, Muir, Cox & Cádiz, 1987; Gómez, Muir, Muir & Cox, 1988; Muir, Gómez, Muir, Cádiz, Cox & Barnes, 1988; Muir, Gómez, Muir & Cádiz, 1990). The average C—S—C angle in the thiazole rings of those compounds was 90.2 (4)°, which is considerably smaller than the C—O—C angle of 103.7 (3)° in nsbo. The oxazole in

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* Lists of structure factors, anisotropic thermal factors, H-atom parameters, bond lengths and angles, and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54660 (41 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

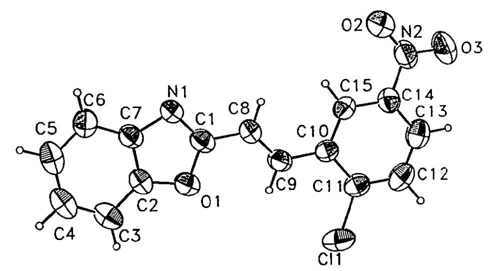

Fig. 1. Labeling of atoms in 2-(2-chloro-5-nitrostyryl)benzoxazole (50% probability ellipsoids).

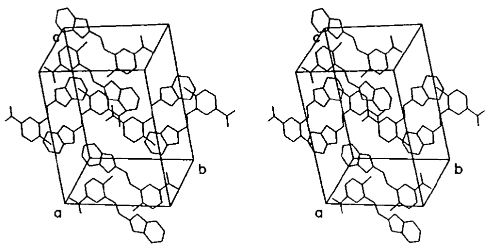

Fig. 2. Packing diagram of 2-(2-chloro-5-nitrostyryl)benzoxazole in the unit cell.

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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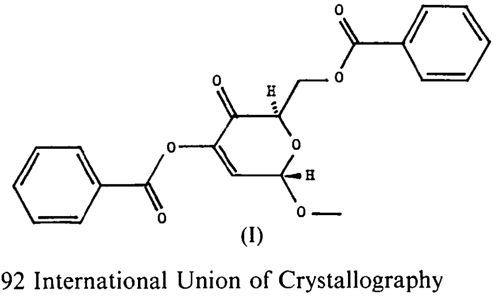
Abstract. Methyl 3,6-di-*O*-benzoyl-2-deoxy-α-D-*glycero*-hex-2-enopyranosid-4-ulose. $C_{21}H_{18}O_7$, M_r = 382.4, monoclinic, $P2_1$, $a = 4.206$ (1), $b = 27.601$ (6), $c = 8.145$ (1) Å, $\beta = 95.31$ (2)°, $V = 941.5$ (3) Å^3, $Z = 2$, $D_x = 1.349$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.10$ mm^{-1}, $F(000) = 400$, $T = 297$ K, final $R = 0.034$ for 1375 observed reflections. The pyranoid conformation is $^{O}H_5$ with $Q = 45.6$ pm, $\theta = 57.8°$ and $\varphi = 11.5°$ [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)