six Pu neighbors for a total of 14. No distances are unusually short and all atoms are fairly uniformly surrounded, a structural feature which is reflected in the nearly isotropic thermal motion.

All calculations were performed on a CDC-7600 computer using the LASL crystal structure codes developed by A. C. Larson. Thanks are due to V. O. Struebing for preparation of the alloys.

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p-Nitrophenyl-β-D-xylopyranoside

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Abstract. $C_{11}H_{13}O_7N$, $M=271{\cdot}2$; orthorhombic, $P2_12_12_1$; $a=5{\cdot}502$ (1), $b=9{\cdot}110$ (1), $c=22{\cdot}859$ (5) Å; $Z=4$, $D_m=1{\cdot}568$ (flotation), $D_c=1{\cdot}573$ g cm^{-3}. $R=0{\cdot}042$ for 915 reflexions. The xylopyranoside is in the C_1 chair conformation. The valence angle of the oxygen atom linking with the *p*-nitrophenyl group is 118·5°. The anomeric C–O bond is twisted by 23·6° against the benzene plane.

Introduction. The needle-like crystals were obtained from an ethanol solution. The space group $P2_12_12_1$ was deduced from systematic absences of $h=2n+1$ for $h00$, $k=2n+1$ for $0k0$, and $l=2n+1$ for $00l$. Intensity data were collected on a Rigaku automatic four-circle diffractometer with a specimen 0·2 × 0·2 × 0·2 mm, using graphite-monochromated Mo $K\alpha$ radiation ($\lambda=0{\cdot}70926$ Å) and the 2θ–ω scanning mode.

Table 1. *Final atomic parameters* ($\times 10^4$) *for the non-hydrogen atoms*

The temperature factor expression used is $\exp[-(h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+hk\beta_{12}+hl\beta_{13}+kl\beta_{23})]$. Values in parentheses are estimated standard deviations.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	5478 (10)	2769 (5)	6602 (2)	238 (19)	47 (5)	11 (1)	−13 (18)	4 (8)	3 (4)
C(2)	7555 (9)	2854 (5)	7037 (2)	157 (17)	56 (5)	12 (1)	−4 (18)	1 (7)	−6 (4)
C(3)	7906 (9)	4440 (5)	7236 (2)	170 (18)	56 (5)	14 (1)	7 (18)	−1 (8)	−1 (4)
C(4)	7958 (9)	5520 (5)	6726 (2)	187 (19)	53 (6)	16 (1)	11 (17)	−17 (8)	−12 (4)
C(5)	5983 (11)	5174 (6)	6276 (2)	292 (24)	74 (7)	16 (1)	−46 (21)	34 (9)	−11 (5)
O(1)	5322 (7)	1306 (3)	6425 (2)	237 (14)	54 (4)	16 (1)	−7 (14)	17 (6)	9 (3)
O(2)	6993 (6)	2014 (4)	7547 (2)	189 (13)	48 (4)	15 (1)	39 (13)	−16 (6)	−8 (3)
O(3)	117 (7)	4563 (4)	7542 (2)	242 (13)	66 (4)	19 (1)	−62 (13)	58 (6)	−21 (3)
O(4)	7665 (7)	6989 (4)	6940 (2)	223 (15)	43 (4)	19 (1)	8 (14)	−2 (7)	0 (3)
O(5)	6062 (7)	3660 (4)	6114 (1)	316 (16)	68 (4)	12 (1)	−39 (14)	3 (6)	3 (3)
C(6)	3290 (9)	861 (5)	6121 (2)	200 (17)	65 (6)	10 (1)	−25 (17)	−3 (8)	6 (4)
C(7)	2843 (10)	−637 (5)	6136 (2)	314 (22)	58 (6)	13 (1)	16 (20)	−8 (9)	1 (4)
C(8)	897 (10)	−1209 (5)	5842 (2)	301 (22)	51 (6)	16 (1)	−33 (19)	6 (9)	−1 (5)
C(9)	−523 (10)	−276 (5)	5516 (2)	256 (19)	77 (7)	12 (1)	−25 (19)	−10 (8)	10 (4)
C(10)	−117 (10)	1223 (5)	5487 (2)	255 (20)	70 (6)	15 (1)	13 (21)	10 (9)	−10 (5)
C(11)	1827 (10)	1789 (5)	5804 (2)	269 (21)	50 (5)	16 (1)	−17 (18)	13 (9)	1 (4)
O(6)	−3271 (7)	−2124(4)	5320 (2)	335 (16)	80 (4)	20 (1)	−126 (16)	−6 (6)	8 (3)
O(7)	−3494 (8)	−165 (4)	4797 (2)	457 (19)	127 (6)	24 (1)	−143 (20)	101 (8)	−26 (4)
N	−2575 (8)	−893 (4)	5186 (2)	229 (17)	91 (6)	15 (1)	−46 (17)	−6 (7)	14 (4)

1592 independent reflexions were collected up to 50° in 2θ, but 677 reflexions with $F_o < 3\sigma(F)$ were treated as unobserved. No corrections were made for absorption or extinction.

The structure was solved by the direct method, using the program *MULTAN* (Main, Woolfson & Germain, 1971) and refined by the full-matrix least-squares method. The thermal factors of the hydrogen atoms were fixed as the isotropic factors of the carbon or oxygen atoms to which they are bonded. Each cycle of refinement was carried out in two stages because of the limited core-storage capacity of the computer; in the first stage the parameters of the xyloside group were refined and in the second stage those of the *p*-nitrophenyl group. The quantity minimized was $\sum w(|F_o|-|F_c|)^2$ with $w=1{\cdot}0$. The final R ($R=\sum||F_o|-|F_c||/\sum|F_o|$) was 0·042.* The atomic scattering factors

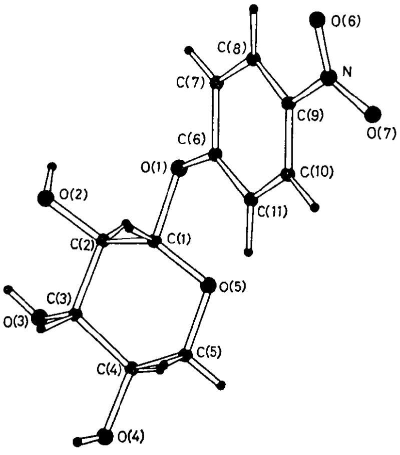

Fig. 1. Molecular conformation and numbering of atoms in *p*-nitrophenyl-*β*-D-xylopyranoside.

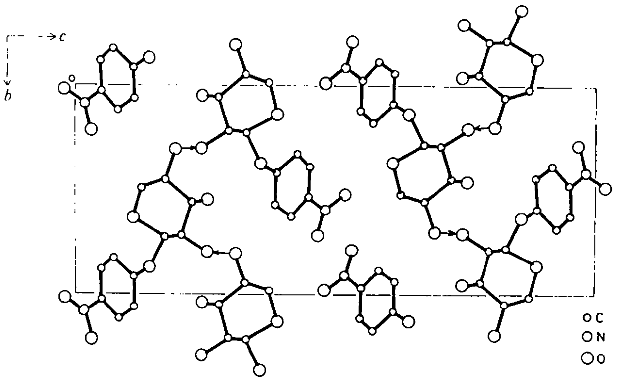

Fig. 2. The crystal structure viewed along the *a* axis. Arrows indicate hydrogen bonds.

Table 2. *Fractional coordinates* (× 10³) *and isotropic thermal factors* (Å²) *for the hydrogen atoms*

The thermal factors are those of the corresponding carbon or oxygen atoms to which the hydrogen atoms are bonded.

	x	*y*	*z*	*B*
H(C1)	371 (8)	312 (5)	682 (2)	2·5
H(C2)	913 (9)	246 (6)	684 (2)	2·3
H(C3)	644 (8)	476 (5)	753 (2)	2·3
H(C4)	956 (9)	550 (6)	650 (2)	2·6
H(C5)	613 (9)	576 (5)	588 (2)	2·9
H′(C5)	427 (9)	537 (5)	646 (2)	2·9
H(O2)	765 (10)	135 (6)	750 (2)	2·6
H(O3)	21 (13)	400 (7)	782 (2)	3·2
H(O4)	656 (13)	713 (8)	715 (3)	2·9
H(C7)	399 (9)	−126 (6)	639 (2)	3·0
H(C8)	49 (9)	−221 (5)	587 (2)	2·8
H(C10)	−116 (10)	186 (6)	526 (2)	3·3
H(C11)	222 (9)	286 (5)	579 (2)	2·7

Table 3. *Bond distances* (Å), *angles* (°) *and conformation angles* (°)

Bond distances

C(1)—C(2)	1·517 (7)	C(9)—N	1·470 (6)
C(2)—C(3)	1·526 (7)	O(7)—N	1·218 (6)
C(3)—O(3)	1·437 (6)	C(2)—H(C2)	1·04 (5)
C(5)—O(5)	1·428 (7)	C(5)—H(C5)	1·05 (5)
C(6)—C(11)	1·373 (7)	O(3)—H(O3)	0·81 (6)
C(9)—C(10)	1·384 (7)	C(8)—H(C8)	0·94 (5)
O(6)—N	1·223 (6)	C(1)—O(5)	1·415 (6)
C(1)—H(C1)	1·14 (4)	C(3)—C(4)	1·524 (7)
C(4)—H(C4)	1·02 (5)	C(4)—O(4)	1·425 (6)
O(2)—H(O2)	0·72 (5)	C(6)—C(7)	1·385 (7)
C(7)—H(C7)	1·03 (5)	C(8)—C(9)	1·374 (7)
C(11)-H(C11)	1·00 (5)	C(10)-C(11)	1·391 (7)
C(1)—O(1)	1·393 (6)	C(3)—H(C3)	1·09 (5)
C(2)—O(2)	1·426 (6)	C(5)—H′(C5)	1·05 (5)
C(4)—C(5)	1·529 (7)	O(4)—H(O4)	0·80 (7)
C(6)—O(1)	1·379 (6)	C(10)-H(C10)	0·97 (5)
C(7)—C(8)	1·369 (7)		

Bond angles

C(2)—C(1)—O(1)	106·6 (4)	C(2)—C(1)—O(5)	108·3 (4)
O(1)—C(1)—O(5)	109·4 (4)	C(1)—C(2)—C(3)	109·8 (4)
C(1)—C(2)—O(2)	110·1 (4)	C(3)—C(2)—O(2)	106·9 (4)
C(2)—C(3)—C(4)	112·6 (4)	C(2)—C(3)—O(3)	109·2 (4)
C(4)—C(3)—O(3)	107·7 (4)	C(3)—C(4)—C(5)	111·5 (4)
C(3)—C(4)—O(4)	110·7 (4)	C(5)—C(4)—O(4)	109·4 (4)
C(4)—C(5)—O(5)	110·6 (4)	C(1)—O(5)—C(5)	110·0 (4)
C(1)—O(1)—C(6)	118·5 (4)	C(7)—C(6)—O(1)	114·9 (4)
C(11)-C(6)—O(1)	124·2 (4)	C(6)—C(7)—C(8)	120·1 (5)
C(7)—C(8)—C(9)	118·5 (5)	C(8)—C(9)—C(10)	122·9 (5)
C(8)—C(9)—N	118·7 (4)	C(10)-C(9)—N	118·5 (4)
C(9)—C(10)-C(11)	117·7 (5)	C(6)—C(11)-C(10)	119·9 (5)
C(9)—N——O(6)	117·6 (4)	C(9)—N——O(7)	119·0 (4)
O(6)—N——O(7)	123·4 (4)		

Conformation angles

C(1)-C(2)-C(3)-C(4)	−47·9
C(2)-C(3)-C(4)-C(5)	43·2
C(3)-C(4)-C(5)-O(5)	−50·7
C(4)-C(5)-O(5)-C(1)	65·2
C(5)-O(5)-C(1)-C(2)	−70·1
O(5)-C(1)-C(2)-C(3)	60·3
O(1)-C(1)-C(2)-O(2)	−64·7
O(2)-C(2)-C(3)-O(3)	73·2
O(3)-C(3)-C(4)-O(4)	−74·4
C(2)-C(1)-O(1)-C(6)	167·2
O(5)-C(1)-O(1)-C(6)	−75·1
C(1)-O(1)-C(6)-C(7)	159·6
C(1)-O(1)-C(6)-C(11)	−23·6

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31727 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

were taken from *International Tables for X-ray Crystallography* (1962). Atomic parameters are given in Tables 1 and 2. Bond distances, angles and conformation angles are shown in Table 3.

Table 4. *Least-squares planes and deviations of atoms* (Å)

An asterisk indicates atoms not included in the plane.

The plane through the nitro group

$$0\cdot6396X-0\cdot3981Y+0\cdot6602Z=6\cdot6715$$

C(9)	0·000	O(7)	0·000
O(6)	0·000	N	0·000

The plane through the benzene ring

$$0\cdot5879X-0\cdot1341Y+0\cdot7977Z=8\cdot0217$$

C(6)	0·001	C(9)	0·005
C(7)	0·011	C(10)	0·007
C(8)	−0·014	C(11)	−0·010
O(1)*	0·051	O(6)*	−0·303
N*	0·016	O(7)*	0·338

The dihedral angle between the two planes is 17·4°.

Discussion. The xyloside group is in the C_1 chair conformation. The bond distances, angles and conformation angles in the xyloside group are in agreement with those observed in other β-D-xyloside derivatives (Brown, Cox & Llewellyn, 1966; Moran & Richards, 1973). The bond distances and angles in the *p*-nitrophenyl group are also in agreement with those in *p*-nitrophenol (Coppens & Schmidt, 1965*a*,*b*) and its derivatives (Guttormson & Robertson, 1972; Brehm & Moult, 1975). The benzene ring shows good planarity (Table 4). The plane of the nitro group makes an angle of 17·4° with the benzene plane. The valence angle of the oxygen atom linking the *p*-nitrophenyl group with the xyloside group is similar to that of *p*-nitrophenyl-β-D-*N*-acetylglucosaminide (Brehm & Moult, 1975). The O(1)–C(6) bond is *gauche* to the O(5)–C(1) bond and *trans* to the C(2)–C(1) bond. The C(1)–O(1) bond is twisted by 23·6° against the benzene plane. The conformation around the C(1)–O(1)–C(6) linkage (Fig. 1) is very similar to that observed in *p*-nitrophenyl-β-D-*N*-acetylglucosaminide. Only one hydrogen bond with the O···O distance of 2·832 Å is observed between O(2) and O(4) $(1-x, -\frac{1}{2}+y, \frac{1}{2}-z)$ (Fig. 2).

The author wishes to thank Dr H. Uedaira for supporting this work and for helpful discussions. The program *FMLS* (Ashida, 1973) was used with some modifications for the full-matrix least-squares calculations. The computation was done on a HITAC 8450 computer in our laboratory.

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Dinitrosylbis(triphenylphosphine)cobalt Hexafluorophosphate

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Abstract. $C_{36}H_{30}N_2O_2F_6P_3Co$, $M=788\cdot49$, monoclinic, $C2/c$, $a=17\cdot431$ (9), $b=12\cdot352$ (6), $c=20\cdot256$ (10) Å, $\beta=125\cdot27°$, $U=3560\cdot7$ Å^3, $Z=4$, $D_c=1\cdot70$ g cm^{-3}. The structure consists of discrete cations and anions each possessing crystallographic twofold axes. The coordination about Co in the cation is tetrahedral distorted towards square planar, the N–Co–N angle being 136·7 (4)°.

Introduction. Cell dimensions were obtained from a least-squares fit to the settings for 25 reflexions on a Philips PW 1100 automated four-circle diffractometer

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