Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0209$
$wR(F^2) = 0.0549$
$S = 1.132$
1231 reflections
81 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0220P)^2 + 0.2637P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.001$
$\Delta\rho_{max} = 0.386$ e Å^{-3}
$\Delta\rho_{min} = -0.264$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0345 (34)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
S(1)	0.18274 (7)	0.11136 (3)	0.91024 (2)	0.01213 (10)
C(2)	0.3190 (3)	0.28744 (11)	0.95345 (8)	0.0112 (2)
N(2)	0.2736 (3)	0.32888 (11)	1.04858 (7)	0.0159 (2)
N(3)	0.4649 (2)	0.37589 (10)	0.88679 (7)	0.0115 (2)
C(4)	0.4930 (3)	0.30606 (11)	0.79469 (8)	0.0104 (2)
O(4)	0.6290 (2)	0.36192 (9)	0.72118 (6)	0.0146 (2)
C(5)	0.3437 (3)	0.14997 (12)	0.78708 (8)	0.0113 (2)

Table 2. *Selected geometric parameters* (Å, °)

S(1)—C(2)	1.7509 (11)	N(3)—C(4)	1.3642 (13)
S(1)—C(5)	1.7953 (10)	C(4)—O(4)	1.2329 (13)
C(2)—N(2)	1.3167 (13)	C(4)—C(5)	1.5206 (14)
C(2)—N(3)	1.3333 (13)		
C(2)—S(1)—C(5)	89.70 (5)	O(4)—C(4)—N(3)	124.01 (10)
N(2)—C(2)—N(3)	122.29 (10)	O(4)—C(4)—C(5)	120.62 (9)
N(2)—C(2)—S(1)	120.04 (8)	N(3)—C(4)—C(5)	115.37 (9)
N(3)—C(2)—S(1)	117.66 (8)	C(4)—C(5)—S(1)	106.05 (7)
C(2)—N(3)—C(4)	111.17 (9)		

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H···A	H···A	D···A	D—H···A
N(2)—H(2*A*)···O(4^{i})	2.09 (2)	2.911 (1)	163 (1)
N(2)—H(2*B*)···N(3^{ii})	2.08 (2)	2.947 (1)	177 (1)

Symmetry codes: (i) $x - \frac{1}{2}, \frac{1}{2} - y, \frac{1}{2} + z$; (ii) $1 - x, 1 - y, 2 - z$.

Data collection, cell refinement and data reduction: Siemens *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1121). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Anti-Inflammatory Drugs: 1-(2-Hydroxyethyl)pyrrolidinium Salt of Diclofenac

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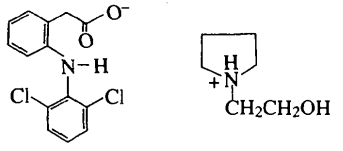
Abstract

In the solid-state structure of *N*-(2-hydroxyethyl)pyrrolidinium {2-[(2,6-dichlorophenyl)amino]phenyl}acetate, $C_6H_{14}NO^+.C_{14}H_{10}Cl_2NO_2^-$, the asymmetric unit contains two independent ion pairs differing mainly in the conformation of the cation. A complex network of inter- and intramolecular hydrogen bonds is present.

Comment

Non-steroidal anti-inflammatory drugs (NSAID's) have been receiving considerable attention during the last few decades. Their mechanism of action has been recognized (Vane, 1971) as inhibition of the activity of the cyclo-oxygenase enzyme. Different hypotheses have been made about the binding site, but all indicate that the carboxyl groups of the NSAID's are responsible for the interactions (Moser, Sallmann & Wiesenberg, 1990). Diclofenac, {2-[2,6-dichlorophenyl)amino]phenyl}acetic acid, is a potent and widely used anti-inflammatory agent and is usually

used in the form of a soluble salt. Here we report the structure of *N*-(2-hydroxyethyl)pyrrolidinium {2-[(2,6-dichlorophenyl)amino]phenyl}acetate, (I).



The two phenylacetate anions and two *N*-(2-hydroxyethyl)pyrrolidinium cations present in the asymmetric unit form ion pairs. The main difference between the two independent pairs lies in the opposite spatial orientation of the cations with respect to the anions. A complex network of inter- and intramolecular hydrogen bonds is present in (I) with two kinds of interactions: *D*—H···*A* and C—H···*A* (*D* = N or O and *A* = Cl or O). *D*—H···*A* bonds are responsible for the formation of the two ion pairs and the cation and anion in each pair are held together by very short hydrogen bonds connecting the carboxylic O atoms with the N—H and O—H groups of the cations. Moreover, in both anions, two intramolecular *D*—H···*A* bonds involve the diphenylamine H atom H(1) with Cl(1) [2.61 (3), 2.68 (3) Å] and with O(1) [2.22 (3), 2.05 (3) Å]. O(1) in molecule 1 is connected *via* a hydrogen bond to N(2) [O(1)···H(11) 1.78 (3) Å], while in molecule 2 it is connected to the hydroxyl O atom O(3) [O(1*A*)···H(24*A*) 1.85 (3) Å]. Consequently, the second contact occurs between O(2) and O(3) in molecule 1 and between O(2*A*) and N(2*A*) in molecule 2 [O(2)···H(24) 1.78 (3), O(2*A*)···H(11*A*) 1.84 (3) Å]. Within the two ion pairs, the cations adopt both possible relative orientations with respect to the anions and with nearly opposite torsion angles [*e.g.* N(2)—C(19)—C(20)—O(3) −73.4 (3) in molecule 1, N(2*A*)—C(19*A*)—C(20*A*)—O(3*A*) 64.0 (3)° in molecule 2].

In conclusion, the *D*—H···*A* interactions give rise to strong hydrogen bonds within the asymmetric unit, while the weaker C—H···*A* interactions are responsible for the crystal packing. This is consistent with the results reported by Desiraju (1991) for the crystal engineering of carboxylic acids. The delocalized character of the carboxyl groups is shown by the O—C bond lengths, which are similar in both molecules and average 1.255 (3) Å, compared with 1.304 and 1.226 Å in the free acid (II) (Moser, Sallmann & Wiesenberg, 1990). The different angle of twist between the two aromatic rings in (I) [73.6 (1) and 66.3 (1)° in molecules 1 and 2, respectively] appears to be of some interest because it seems to be directly related to the therapeutic activity of the drug and is comparable with the corresponding value found in (II) (69.4°). The inter-planar angle between the carboxyl group [O(1), O(2), C(13), C(14)] and the dichloro-substituted phenyl ring differs in the two molecules [9.9 (1) and 24.0 (1)° in molecules 1 and 2, respectively, while the corresponding value in (II) is 1.5°]. Again this structural difference appears to be of some interest since it is assumed that the drug activity is based on the interaction of the carboxyl group with the receptor site of arachidonic acid cyclo-oxygenase, inhibiting the prostaglandin synthetase. Bond distances and angles in both the anions and cations fall within their standard ranges.

The strong ion-pair interactions shown in the solid state could be related to the particular properties of (I) observed in solution (*i.e.* higher solubility than predicted, supersaturation phenomena and unexpected detergent ability) (Fini, Fazio, Orienti, Zecchi & Rapaport, 1991), if the presence of solvated ion pairs is allowed for.

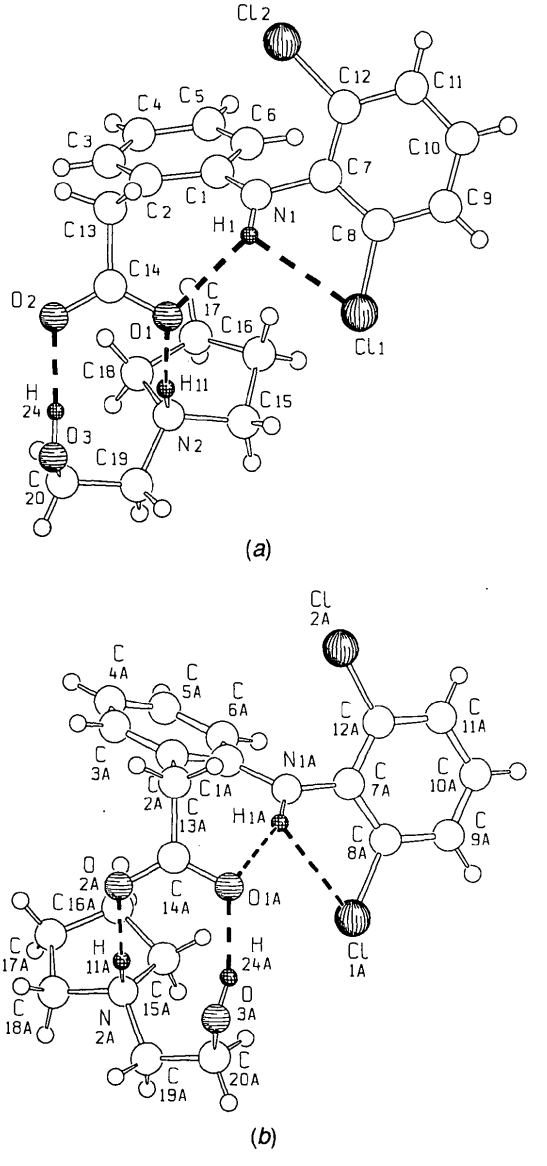


Fig. 1. Perspective view of (I) showing the atom labelling and anion–cation interactions in (*a*) molecule 1 and (*b*) molecule 2.

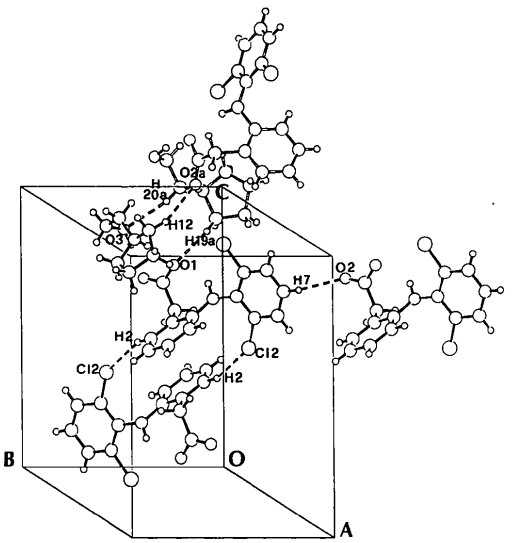


Fig. 2. Crystal packing of (I) showing the intermolecular interactions. Filled bonds indicate the anions and cations of molecule 1.

Experimental

The compound was supplied by IBSA, Lugano, Switzerland. Crystals were obtained from a solution of triacetin and PEG 400.

Crystal data

$C_6H_{14}NO^+.C_{14}H_{10}Cl_2NO_2^-$
M_r = 411.31
Triclinic
$P\bar{1}$
a = 11.663 (2) Å
b = 11.909 (10) Å
c = 15.306 (10) Å
α = 76.01 (6)°
β = 84.67 (3)°
γ = 87.14 (4)°
V = 2053.2 (22) Å^3
Z = 4
D_x = 1.331 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 8–12°
μ = 0.339 mm^{-1}
T = 293 (2) K
Prismatic
0.5 × 0.4 × 0.3 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
Profile data from ω scans
Absorption correction: none
7173 measured reflections
6881 independent reflections
5238 observed reflections [$I > 2\sigma(I)$]

R_{int} = 0.0104
θ_{max} = 24.97°
$h = -13 \rightarrow 13$
$k = -13 \rightarrow 14$
$l = 0 \rightarrow 15$
3 standard reflections
frequency: 160 min
intensity variation: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0443
$wR(F^2)$ = 0.1080
S = 1.147
6871 reflections
510 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0465P)^2 + 0.7749P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.076
$\Delta\rho_{max}$ = 0.284 e Å^{-3}
$\Delta\rho_{min}$ = −0.290 e Å^{-3}

Extinction correction:
$F_c^* = kF_c[1 + (0.001\chi \times F_c^2\lambda^3/\sin 2\theta)]^{-1/4}$
Extinction coefficient:
χ = 0.0381 (14)

Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* (Å^2)

U_{iso} for H atoms (fixed); $U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for others.

	x	y	z	U_{iso}/U_{eq}
Cl(1)	0.24399 (8)	0.12785 (7)	0.85379 (6)	0.0956 (3)
Cl(2)	0.54078 (7)	0.16902 (6)	0.55385 (6)	0.0877 (3)
N(1)	0.3926 (2)	0.2623 (2)	0.6933 (2)	0.0604 (5)
C(1)	0.3557 (2)	0.3470 (2)	0.6186 (2)	0.0555 (6)
C(2)	0.3891 (2)	0.4614 (2)	0.6076 (2)	0.0597 (7)
C(3)	0.3455 (3)	0.5454 (2)	0.5374 (2)	0.0780 (9)
C(4)	0.2748 (3)	0.5189 (3)	0.4790 (2)	0.0860 (9)
C(5)	0.2464 (3)	0.4061 (2)	0.4885 (2)	0.0770 (8)
C(6)	0.2862 (2)	0.3210 (2)	0.5583 (2)	0.0676 (7)
C(7)	0.3855 (2)	0.1431 (2)	0.7000 (2)	0.0568 (6)
C(8)	0.3201 (2)	0.0706 (2)	0.7705 (2)	0.0648 (7)
C(9)	0.3162 (3)	−0.0475 (2)	0.7781 (2)	0.0754 (8)
C(10)	0.3767 (3)	−0.0963 (2)	0.7151 (2)	0.0790 (9)
C(11)	0.4446 (3)	−0.0295 (2)	0.6458 (2)	0.0736 (8)
C(12)	0.4492 (2)	0.0887 (2)	0.6391 (2)	0.0629 (7)
C(13)	0.4682 (2)	0.4940 (2)	0.6691 (2)	0.0702 (8)
C(14)	0.4029 (2)	0.5142 (2)	0.7547 (2)	0.0563 (6)
O(1)	0.34654 (14)	0.43105 (13)	0.80411 (12)	0.0621 (4)
O(2)	0.4053 (2)	0.61214 (14)	0.77057 (13)	0.0727 (5)
N(2)	0.1295 (2)	0.50538 (15)	0.84132 (12)	0.0468 (4)
C(15)	0.0612 (2)	0.3974 (2)	0.8585 (2)	0.0577 (6)
C(16)	0.0584 (3)	0.3738 (2)	0.7671 (2)	0.0752 (8)
C(17)	0.0518 (3)	0.4908 (3)	0.7038 (2)	0.0796 (8)
C(18)	0.1025 (2)	0.5748 (2)	0.7490 (2)	0.0577 (6)
C(19)	0.1068 (2)	0.5682 (2)	0.9149 (2)	0.0538 (6)
C(20)	0.1781 (2)	0.6740 (2)	0.9036 (2)	0.0635 (6)
O(3)	0.2938 (2)	0.6483 (2)	0.92028 (13)	0.0733 (5)
H(1)	0.3790 (28)	0.2781 (27)	0.7457 (19)	0.100
H(11)	0.2077 (23)	0.4834 (26)	0.8384 (21)	0.100
H(24)	0.3313 (28)	0.6316 (28)	0.8730 (20)	0.100
Cl(1A)	−0.23232 (8)	−0.14466 (6)	−0.39426 (5)	0.0858 (3)
Cl(2A)	−0.00655 (8)	0.24323 (7)	−0.40157 (5)	0.0894 (3)
N(1A)	−0.1161 (2)	0.0157 (2)	−0.30978 (13)	0.0570 (5)
C(1A)	−0.1605 (2)	0.0774 (2)	−0.2450 (2)	0.0519 (6)
C(2A)	−0.1249 (2)	0.0453 (2)	−0.1582 (2)	0.0516 (5)
C(3A)	−0.1684 (2)	0.1079 (2)	−0.0960 (2)	0.0679 (7)
C(4A)	−0.2449 (3)	0.2007 (3)	−0.1178 (2)	0.0799 (8)
C(5A)	−0.2808 (3)	0.2300 (2)	−0.2031 (2)	0.0804 (8)
C(6A)	−0.2399 (2)	0.1697 (2)	−0.2669 (2)	0.0666 (7)
C(7A)	−0.1251 (2)	0.0542 (2)	−0.4025 (2)	0.0520 (6)
C(8A)	−0.1747 (2)	−0.0131 (2)	−0.4514 (2)	0.0571 (6)
C(9A)	−0.1777 (3)	0.0199 (2)	−0.5441 (2)	0.0717 (7)
C(10A)	−0.1335 (3)	0.1240 (3)	−0.5904 (2)	0.0791 (8)
C(11A)	−0.0848 (3)	0.1934 (2)	−0.5454 (2)	0.0743 (8)
C(12A)	−0.0795 (2)	0.1581 (2)	−0.4531 (2)	0.0600 (6)
C(13A)	−0.0419 (2)	−0.0567 (2)	−0.1309 (2)	0.0587 (6)
C(14A)	−0.1007 (2)	−0.1711 (2)	−0.1213 (2)	0.0549 (6)
O(1A)	−0.0963 (2)	−0.21164 (14)	−0.19024 (12)	0.0677 (5)
O(2A)	−0.1535 (2)	−0.2157 (2)	−0.04699 (12)	0.0711 (5)
N(2A)	−0.3622 (2)	−0.2920 (2)	−0.07608 (15)	0.0641 (6)
C(15A)	−0.3930 (2)	−0.1957 (2)	−0.1528 (2)	0.0683 (7)
C(16A)	−0.4211 (3)	−0.0946 (3)	−0.1102 (2)	0.0872 (9)
C(17A)	−0.4680 (4)	−0.1446 (4)	−0.0182 (3)	0.136 (2)
C(18A)	−0.4410 (3)	−0.2714 (3)	0.0033 (2)	0.0978 (11)
C(19A)	−0.3704 (3)	−0.4100 (2)	−0.0913 (3)	0.0883 (10)
C(20A)	−0.2908 (3)	−0.4312 (3)	−0.1676 (3)	0.1035 (12)
O(3A)	−0.1738 (2)	−0.4272 (2)	−0.1507 (2)	0.1009 (8)
H(1A)	−0.1108 (27)	−0.0594 (23)	−0.2864 (21)	0.100
H(11A)	−0.2883 (24)	−0.2781 (27)	−0.0639 (21)	0.100
H(24A)	−0.1507 (28)	−0.3584 (25)	−0.1782 (22)	0.100

Table 2. *Selected geometric parameters* (Å, °)

C(14)—O(2)	1.250 (3)	C(14A)—O(2A)	1.251 (3)
C(14)—O(1)	1.263 (3)	C(14A)—O(1A)	1.258 (3)
C(7)—N(1)—C(1)	122.4 (2)	C(7A)—N(1A)—C(1A)	124.3 (2)
C(2)—C(13)—C(14)	111.7 (2)	C(2A)—C(13A)—C(14A)	111.1 (2)
O(2)—C(14)—C(13)	118.4 (2)	O(2A)—C(14A)—C(13A)	118.1 (2)
N(2)—C(19)—C(20)	114.8 (2)	C(20A)—C(19A)—N(2A)	113.3 (3)
O(3)—C(20)—C(19)	113.7 (2)	O(3A)—C(20A)—C(19A)	111.8 (3)
C(1)—N(1)—C(7)—C(12)	64.6 (3)		
N(2)—C(19)—C(20)—O(3)	−73.4 (3)		
C(1A)—N(1A)—C(7A)—C(12A)	59.6 (3)		
N(2A)—C(19A)—C(20A)—O(3A)	64.0 (3)		

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N(2)—H(11)···O(1)	0.94 (3)	1.78 (3)	2.696 (3)	164 (3)
N(2A)—H(11A)···O(2A)	0.93 (3)	1.84 (3)	2.751 (3)	167 (3)
O(3)—H(24)···O(2)	0.87 (3)	1.78 (3)	2.645 (3)	174 (3)
O(3A)—H(24A)···O(1A)	0.87 (3)	1.85 (3)	2.674 (3)	158 (3)
N(1)—H(1)···Cl(1)	0.87 (3)	2.61 (3)	3.031 (3)	111 (2)
N(1A)—H(1A)···Cl(1A)	0.88 (3)	2.68 (3)	2.989 (3)	102 (2)
N(1)—H(1)···O(1)	0.87 (3)	2.22 (3)	2.929 (3)	139 (3)
N(1A)—H(1A)···O(1A)	0.88 (3)	2.05 (3)	2.890 (3)	159 (3)
C(3)—H(2)···Cl(2^{i})		2.74	3.618 (4)	157
C(10)—H(7)···O(2^{ii})		2.49	3.376 (4)	160
C(15)—H(12)···O(2A^{iii})		2.48	3.390 (4)	156
C(18A)—H(19A)···O(1^{iii})		2.44	3.347 (5)	154
C(19A)—H(20A)···O(3^{iii})		2.56	3.506 (5)	165

Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $x, 1+y, z$; (iii) $-x, -y, 1-z$.

The H atoms bonded to N and O atoms [H(1), H(1A), H(11), H(11A), H24, H(24A)] were located experimentally and refined with distance restraints. The remaining H atoms were placed in calculated positions and refined riding on the parent atoms (aromatic C—H = 0.93, C_{sp^3}—H = 0.97 Å) with U_{iso} fixed at 1.2 × U_{eq} of attached atom. Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1994). Molecular graphics: *SCHAKAL92* (Keller, 1992). Software used to prepare material for publication: *SHELXL93* (Sheldrick, 1994).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1099). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1-*O*-(2-Iodophenyl)-β-D-galactopyranose

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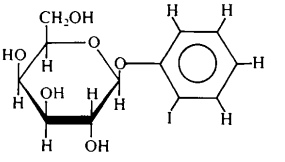
Abstract

The sugar ring in 1-*O*-(2-iodophenyl)-β-D-galactopyranose ($C_{12}H_{15}IO_6$) has the expected $^4C_1$ chair conformation. The molecules form hydrogen-bonded layers which stack together so that the crystal structure contains alternate polar and non-polar slices along the *c* axis.

Comment

The title compound, (1), which was synthesized in this laboratory, was used to prepare a heavy-atom derivative of peanut lectin (Banerjee *et al.*, 1994). As it was also used to locate the sugar binding site in the lectin, its structure was determined.



(1)

The sugar ring in the title structure has an almost ideal $^4C_1$ chair conformation (Stoddart, 1971) with θ = 0.02 (2)° (Pickett & Strauss, 1970). C(1) and C(4) are displaced by −0.668 (3) and 0.683 (3) Å, respectively, from the mean plane through the remaining ring atoms. The phenyl ring is inclined at 45.1 (3)° to this plane. The I atom is displaced from the plane of the phenyl ring by 0.091 (1) Å. The torsion angle between the two rings, H(1)—C(1)—O(1)—C(11), is 34 (1)°. The torsion angle C(4)—C(5)—C(6)—O(6), which defines the position of the CH_2OH group with respect to the sugar ring, can assume three sterically favourable values corresponding to three staggered conformations. The angle in the title compound is −58.9 (4)° compared to 176.3° in β-D-galactose (Sheldrick, 1976*a*).

The molecules in the crystal form hydrogen-bonded layers parallel to the *ab* plane (Fig. 2). The hydrogen bonds spiral around 2_1 screw axes and the