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Acta Cryst. (1995). C**51**, 758–759

p-Chlorophenyl 3,4,6-Tri-*O*-benzyl-2-deoxy-2-methylene-β-D-glucopyranoside

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(*Received* 14 *March* 1994; *accepted* 19 *September* 1994)

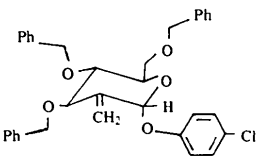
Abstract

The title compound, $C_{34}H_{33}ClO_5$, has a β configuration at the anomeric centre C1. The pyranose ring adopts a chair conformation with all the substituents in equatorial positions. The molecular packing is achieved through van der Waals interactions.

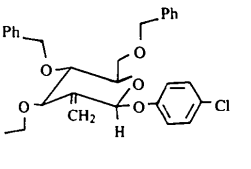
Comment

The title compound was prepared as an intermediate to prove the mechanism of the reaction of a new Ferrier system (Booma & Balasubramanian, 1993*a*) with phenols (Booma & Balasubramanian, 1993*b*). Structure (I) was originally suggested for this compound but NMR NOE experiments indicated structure (II) with a β configuration at the anomeric centre C1 (Fig. 1). The present paper reports the X-ray structure, confirming this configuration in the crystal. A perspective view of the molecule is given in Fig. 1.

† DCB contrubution No. 840.



(I)



(II)

The pyranose ring adopts a chair conformation (ΔC_m = 8.2°, ΔC_2 = 10.1°) (Duax, Weeks & Rohrer, 1976). All the phenyl rings are planar [χ^2 = 1.0, 19.0, 3.6 and 2.8 for rings *A* (C8–C13), *B* (C14–C19), *C* (C22–C27) and *D* (C29–C34), respectively]; the angles between the planes are *A*–*B* 121.0 (3)°, *B*–*C* 137.0 (2)°, *C*–*D* 147.1 (2)°, *A*–*D* 128.9 (3)°, *A*–*C* 18.5 (2)° and *B*–*D* 16.4 (2)°. The moiety C6—O2—C7—C8 is planar and makes an angle of 27.7 (4)° with ring *A*.

The C28—O5 bond is synplanar with respect to the C29—C34 bond. The substituents at C1, C3, C4 and C5 are all in equatorial positions. The packing in the unit cell is governed only by van der Waals interactions.

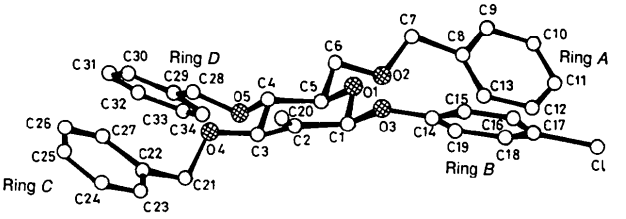


Fig. 1. A perspective view of the title compound.

Experimental

Crystal data

$C_{34}H_{33}ClO_5$	Cu $K\alpha$ radiation
M_r = 558.093	λ = 1.5418 Å
Orthorhombic	Cell parameters from 25 reflections
$P2_12_12_1$	θ = 15–25°
a = 8.236 (3) Å	μ = 1.4 mm^{-1}
b = 14.967 (4) Å	T = 293 K
c = 24.032 (5) Å	Needle
V = 2962 (1) Å^3	0.24 × 0.12 × 0.10 mm
Z = 4	Colourless
D_x = 1.24 Mg m^{-3}	Crystal source: recrystallized from $CHCl_3$–EtOH
D_m = 1.25 (2) Mg m^{-3}	
D_m measured by flotation	

Data collection

Enraf–Nonius diffractometer	θ_{max} = 60°
$\omega/2\theta$ scans	h = 0 → 8
Absorption correction: none	k = 0 → 16
2538 measured reflections	l = 0 → 26
2538 independent reflections	3 standard reflections monitored every 200 reflections
2214 observed reflections [$I > 3\sigma(I)$]	intensity decay: <3%

Refinement

Refinement on F	$w = 1/[\sigma^2(F) + 0.009(F)^2]$
$R = 0.042$	$(\Delta/\sigma)_{max} = 0.113$
$wR = 0.047$	$\Delta\rho_{max} = 0.18$ e Å^{-3}
$S = 0.62$	$\Delta\rho_{min} = -0.17$ e Å^{-3}
1548 reflections	Extinction correction: none
361 parameters	Atomic scattering factors from *SHELXS*86 (Sheldrick, 1985)
All H-atom parameters refined	

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

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

	x	y	z	U_{eq}
Cl	0.1544 (3)	0.2906 (1)	0.9885 (1)	0.109 (1)
O1	0.3531 (5)	0.7406 (2)	0.9660 (1)	0.058 (2)
O2	0.3093 (6)	0.7285 (3)	0.8489 (1)	0.079 (3)
O3	0.3344 (5)	0.6593 (3)	1.0454 (1)	0.067 (2)
O4	0.2687 (5)	0.9776 (3)	1.0469 (1)	0.072 (2)
O5	0.2428 (5)	0.9662 (2)	0.9254 (1)	0.064 (2)
C1	0.2655 (7)	0.7331 (4)	1.0169 (2)	0.058 (3)
C2	0.2866 (7)	0.8164 (4)	1.0498 (2)	0.062 (3)
C3	0.2341 (7)	0.8980 (4)	1.0181 (2)	0.060 (3)
C4	0.3122 (7)	0.8997 (4)	0.9604 (2)	0.059 (3)
C5	0.2880 (6)	0.8113 (3)	0.9320 (2)	0.053 (3)
C6	0.3733 (8)	0.8036 (4)	0.8769 (2)	0.061 (3)
C7	0.4147 (8)	0.6887 (4)	0.8102 (2)	0.070 (4)
C8	0.3332 (8)	0.6070 (4)	0.7858 (2)	0.058 (3)
C9	0.3783 (9)	0.5775 (4)	0.7340 (2)	0.081 (4)
C10	0.3079 (12)	0.5008 (6)	0.7116 (3)	0.098 (5)
C11	0.1987 (12)	0.4550 (5)	0.7400 (3)	0.099 (5)
C12	0.1537 (12)	0.4822 (5)	0.7911 (3)	0.103 (6)
C13	0.2186 (9)	0.5578 (4)	0.8148 (2)	0.077 (4)
C14	0.2927 (7)	0.5745 (4)	1.0283 (2)	0.059 (3)
C15	0.2171 (8)	0.5549 (4)	0.9785 (2)	0.065 (3)
C16	0.1769 (8)	0.4680 (4)	0.9663 (2)	0.069 (4)
C17	0.2155 (8)	0.4011 (4)	1.0025 (2)	0.076 (4)
C18	0.2975 (10)	0.4202 (5)	1.0518 (3)	0.091 (5)
C19	0.3340 (8)	0.5074 (5)	1.0636 (2)	0.078 (4)
C20	0.3489 (9)	0.8195 (5)	1.1015 (2)	0.091 (5)
C21	0.1348 (8)	1.0160 (5)	1.0762 (2)	0.077 (4)
C22	0.1986 (7)	1.0756 (4)	1.1204 (2)	0.062 (3)
C23	0.1587 (10)	1.0599 (4)	1.1756 (2)	0.082 (4)
C24	0.2198 (12)	1.1161 (5)	1.2160 (2)	0.098 (5)
C25	0.3134 (9)	1.1867 (5)	1.2040 (3)	0.079 (4)
C26	0.3545 (10)	1.2030 (4)	1.1500 (3)	0.086 (5)
C27	0.2986 (10)	1.1480 (4)	1.1088 (2)	0.079 (4)
C28	0.3165 (11)	1.0506 (5)	0.9274 (2)	0.095 (5)
C29	0.2799 (9)	1.1044 (4)	0.8758 (2)	0.069 (4)
C30	0.3445 (10)	1.1887 (4)	0.8714 (3)	0.078 (4)
C31	0.3156 (12)	1.2408 (4)	0.8246 (4)	0.102 (6)
C32	0.2216 (12)	1.2070 (5)	0.7818 (4)	0.099 (6)
C33	0.1592 (10)	1.1250 (5)	0.7870 (3)	0.087 (5)
C34	0.1829 (8)	1.0717 (4)	0.8327 (2)	0.071 (4)

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

O1—C1	1.425 (6)	O3—C14	1.378 (7)
O1—C5	1.440 (5)	O4—C3	1.407 (7)
O2—C6	1.412 (7)	O4—C21	1.429 (7)
O2—C7	1.405 (7)	O5—C4	1.423 (6)
O3—C1	1.418 (7)	O5—C28	1.402 (9)
C1—O1—C5	110.9 (4)	O5—C4—C5	107.1 (4)
C6—O2—C7	115.0 (5)	O1—C5—C4	109.9 (4)
C1—O3—C14	118.3 (4)	C4—C5—C6	113.9 (4)
C3—O4—C21	115.3 (4)	O1—C5—C6	105.6 (4)
C4—O5—C28	115.8 (4)	O2—C6—C5	107.8 (4)
O1—C1—O3	105.9 (4)	O2—C7—C8	109.0 (5)
O3—C1—C2	110.5 (4)	O3—C14—C19	115.6 (5)
O1—C1—C2	109.3 (4)	O3—C14—C15	124.5 (5)
O4—C3—C2	112.4 (4)	O4—C21—C22	108.7 (5)
O4—C3—C4	110.3 (4)	O5—C28—C29	111.5 (5)
O5—C4—C3	112.3 (4)		
C1—O1—C5—C6	−171.9 (4)	C1—C2—C3—O4	−174.1 (4)
C1—O1—C5—C4	64.8 (5)	C20—C2—C3—O4	4.4 (8)
C5—O1—C1—O3	177.2 (4)	C20—C2—C3—C4	128.2 (6)
C5—O1—C1—C2	−63.8 (5)	O4—C3—C4—O5	−66.1 (6)
C6—O2—C7—C8	177.2 (5)	C2—C3—C4—O5	169.0 (4)
C7—O2—C6—C5	−153.9 (4)	O4—C3—C4—C5	174.7 (4)
C14—O3—C1—O1	−77.3 (5)	O5—C4—C5—O1	−178.9 (4)
C1—O3—C14—C15	14.6 (8)	C3—C4—C5—O1	−56.6 (5)
C1—O3—C14—C19	−166.8 (5)	O5—C4—C5—C6	62.8 (6)
C14—O3—C1—C2	164.5 (4)	O1—C5—C6—O2	73.7 (5)
C3—O4—C21—C22	158.4 (5)	C4—C5—C6—O2	−165.7 (4)
C21—O4—C3—C2	−99.4 (6)	O2—C7—C8—C9	154.7 (5)
C21—O4—C3—C4	136.7 (5)	O2—C7—C8—C13	−28.2 (8)
C4—O5—C28—C29	158.6 (5)	O3—C14—C19—C18	179.1 (6)
C28—O5—C4—C3	90.1 (6)	O3—C14—C15—C16	−178.2 (5)
C28—O5—C4—C5	−149.1 (5)	O4—C21—C22—C23	−121.3 (6)
O3—C1—C2—C3	172.9 (4)	O4—C21—C22—C27	58.8 (7)
O1—C1—C2—C3	56.7 (6)	O5—C28—C29—C30	−179.6 (6)
O3—C1—C2—C20	−5.7 (8)	O5—C28—C29—C34	0.5 (9)
O1—C1—C2—C20	−121.8 (6)		

Data were corrected for Lorentz and polarization effects. The structure was solved by direct methods. H atoms were found by difference Fourier synthesis and were refined isotropically. Data collection: Enraf–Nonius diffractometer software. Cell refinement and data reduction: *SDP* (Frenz, 1978). Program used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program used to refine structure: *SHELX*76 (Sheldrick, 1976). Calculation of geometric parameters: *PARST* (Nardelli, 1983). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978). Most of the calculations were performed on a MicroVAX II computer system.

The authors thank Professor Vasantha Pattabhi for useful discussions. One of us (VK) thanks the University of Madras for the award of a fellowship.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, bond distances and angles involving non-H atoms and torsion angles have been deposited with the IUCr (Reference: LI1117). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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