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1,3:4,6-Di-*O*-benzylidene-*N*-cinnamyl-2,5-dideoxy-2,5-imino-L-iditol

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Key indicators

Single-crystal X-ray study
T = 113 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.094
Data-to-parameter ratio = 11.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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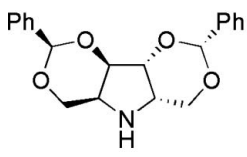
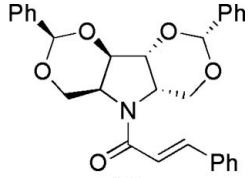
In the crystal structure of the title compound, $C_{29}H_{27}NO_5$, the molecules assemble into a network structure by both intermolecular C—H···O hydrogen bonding and C—H···π stacking interactions.

Comment

The C_2-symmetric pyrrolidine, 1,3:4,6-di-*O*-benzylidene-2,5-dideoxy-2,5-imino-L-iditol, (I), has been shown (Shing, 1988; Masaki *et al.*, 1992) to be an efficient chiral auxiliary in a variety of reactions, *e.g.* in the Diels–Alder cycloaddition reaction (Defoin *et al.*, 1991), radical addition reaction (Veit *et al.*, 1993) or in photochemical reactions (Giese *et al.*, 1996). We are also interested in asymmetric synthesis, employing (I) as a chiral auxiliary. Thus, we prepared the title compound, 1,3:4,6-di-*O*-benzylidene-2,5-dideoxy-2,5-imino-N-cinnamyl-L-iditol, (II), as a major reaction intermediate.



(I)



(II)

An intramolecular C—H···O hydrogen bond is present (Table 1). The crystal packing of the title compound is stabilized by intermolecular C—H···O hydrogen bonding, as well as C—H···π stacking interactions (Fig. 2). The phenyl ring

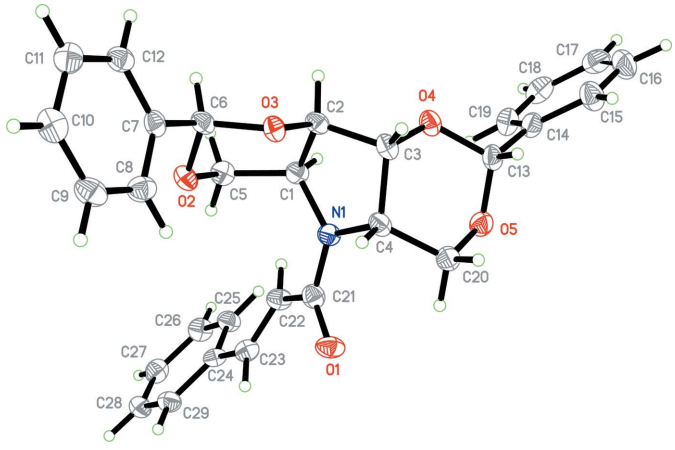


Figure 1
View of the molecular structure of (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size.

C14–C19 at (x, y, z) makes a dihedral angle of 86.8 (7)° with the phenyl ring C7–C12 at $(x-1, y, z)$. The distance between C16 in the molecule at (x, y, z) and the centroid of phenyl ring C7–C12 at $(x-1, y, z)$ is 3.683 Å, indicative of C—H···π bonding.

Experimental

The title compound was prepared by adding cinnamyl chloride (1.5 equivalents) to a solution of (I) (1.0 mmol) in CH_2Cl_2 (15 ml) in the presence of triethylamine (3.0 equivalents). The pure product was obtained in 83% yield after purification by column chromatography, using ethyl acetate–petroleum ether (1:3) as the eluant. Suitable crystals were obtained by evaporation of an ethyl acetate solution (m.p. 387 K). $[\alpha]_D^{24}$ = + 244.31 (*c* 0.83 $CHCl_3$). ESI–MS calculated for $C_{29}H_{28}NO_5$: $[M + H]^+$: 470.2; found: 470.4.

Crystal data

$C_{29}H_{27}NO_5$	Z = 2
M_r = 469.52	D_x = 1.139 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation
a = 13.373 (5) Å	μ = 0.08 mm^{-1}
b = 5.7653 (18) Å	T = 113 (2) K
c = 17.964 (6) Å	Block, colourless
β = 98.653 (5)°	0.32 × 0.22 × 0.20 mm
V = 1369.2 (8) Å^3	

Data collection

Rigaku Saturn diffractometer	8139 measured reflections
ω scans	3555 independent reflections
Absorption correction: multi-scan (Jacobson, 1998)	2777 reflections with $I > 2\sigma(I)$
T_{min} = 0.976, T_{max} = 0.985	R_{int} = 0.038
	θ_{max} = 27.9°

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0496P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.040	$(\Delta/\sigma)_{max}$ = 0.002
$wR(F^2)$ = 0.094	$\Delta\rho_{max}$ = 0.23 e Å^{-3}
S = 0.96	$\Delta\rho_{min}$ = −0.21 e Å^{-3}
3555 reflections	Extinction correction: *SHELXL97*
318 parameters	Extinction coefficient: 0.022 (2)
H-atom parameters constrained	

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C20—H20B···O1	0.99	2.36	2.990 (3)	121
C27—H27···O2^{i}	0.95	2.55	3.427 (3)	154

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

In the absence of significant anomalous dispersion effects, Friedel pairs were averaged. All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95–1.00 Å and U_{iso}(H) = 1.2U_{eq}(C).

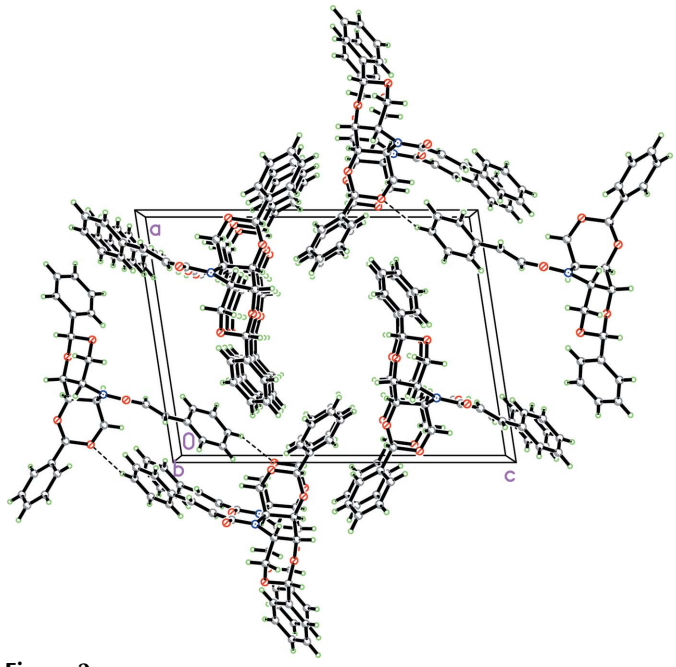


Figure 2
The molecular packing of (II), viewed along the *b* axis.

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *CrystalStructure* (Rigaku/MSC, 2005).

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