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(1*R*,2*R*,3*S*,5*R*,6*R*,7*S*,9a*R*,10a*R*)-4a,8a-Diazaperhydroanthracene-1,2,3,5,6,7-hexaol

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The title compound, $C_{12}H_{22}N_2O_6$, is a linearly fused di(trihydroxypiperido)piperazine. The three six-membered rings adopt chair conformations with all the hydroxyl groups equatorial, and the ring fusion is the unusual *trans–cisoid–cis*.

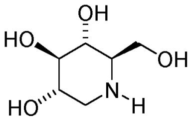
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.005 Å
R factor = 0.046
wR factor = 0.094
Data-to-parameter ratio = 8.0

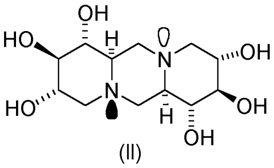
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The title compound, (II), which is a condensation dimer of deoxynojirimycin, (I) (1-deoxy-D-*gluco*pyranose with nitrogen in place of oxygen as the ring atom), was formed in low yield as a by-product on reaction of (I) with 2-bromobenzoylchloride in the presence of base. Presumably, the primary hydroxyl groups of the starting material were selectively esterified and the nitrogen nucleophiles of pairs of the product molecules then displaced the acyloxy groups to generate the central ring of the dimeric product.



(I)



(II)

The bond lengths and angles in (II) (Fig. 1) are normal (Table 1). The crystal lattice is bound tightly by five intermolecular O—H···O bonds and one O—H···N hydrogen bond, which involve all the hydroxyl H atoms [*e.g.* O3—H3O···O1(1 + *x*, *y*, *z*), with O···O = 2.782 (4) Å and O—H···O = 162°; Spek, 1990].

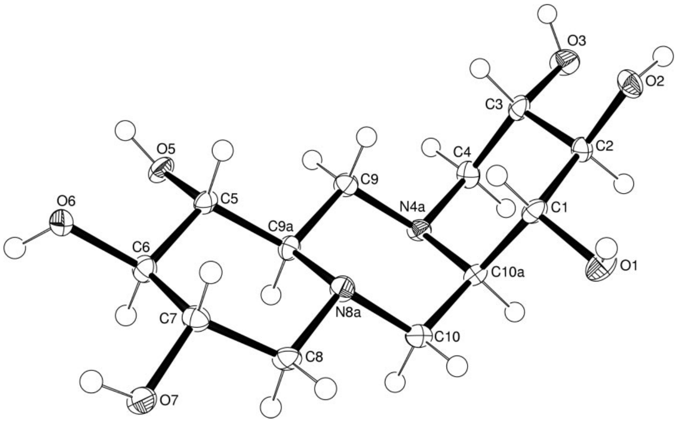


Figure 1
ORTEP-3 (Farrugia, 1997) view of (II), showing the atom-numbering scheme and displacement ellipsoids at the 50% probability level. H atoms are shown as circles of arbitrary radii.

There are no reported crystal structures of perhydrodiazaanthracene ring systems with ring fusion through the N atoms; the closest relatives are two sugar-derived fused-ring pyridopyrimidine compounds, with the former ring carrying three equatorial hydroxyl groups (Berges *et al.*, 1999, and references therein). There are a few reported structures of piperazine compounds in which the N atoms are linked *via* aliphatic carbon chains [*e.g.* 1,7(1,4)-bis(2,5-diphenylpiperizina)dodecacyclophane; Fuji *et al.*, 1996].

The six-membered rings of (II) adopt chair conformations and the ring fusion is unusual in being *trans–cisoid–cis*. This conformation is also observed in a perhydroanthracene derivative with one N atom at a ring-junction position, a structure that occurs in the Veratrum alkaloids (Brambilla *et al.*, 1982). The structures of perhydroanthracenes with the *cis–cisoid–cis* (van Koningsveld *et al.*, 1984) and *cis–transoid–cis* (Hjortaas, 1967) configurations have also been reported.

Experimental

A suspension of deoxynojirimycin (50 mg, 0.31 mmol), potassium carbonate (100 mg) and 2-bromobenzoyl chloride (200 µl) in water (1 ml) was sonicated for 15 min and then stirred at room temperature overnight. The solvent was removed under reduced pressure and the residue was chromatographed on a column of silica gel eluted with $CH_2Cl_2/CH_3OH/NH_4OH$ (5:8:2) to give first deoxynojirimycin, (I), and then the title compound as an oil that gave suitable crystals on standing.

Crystal data

$C_{12}H_{22}N_2O_6$
$M_r = 290.32$
Orthorhombic, $P2_12_12_1$
$a = 6.702$ (2) Å
$b = 13.099$ (5) Å
$c = 14.893$ (3) Å
$V = 1307.5$ (7) Å^3
$Z = 4$
$D_x = 1.475$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 24 reflections
$\theta = 4.8–23.8°$
$\mu = 0.12$ mm^{-1}
$T = 173$ (2) K
Needle, colourless
0.42 × 0.30 × 0.15 mm

Data collection

Siemens/Nicolet *R*3*m* four-circle diffractometer
ω scans
1608 measured reflections
1504 independent reflections
1146 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.039$

$\theta_{\rm max} = 25.9°$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 18$
3 standard reflections every 97 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.094$
$S = 0.98$
1504 reflections
187 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0441P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.23$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.24$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N4*A*—C4	1.480 (4)	N4*A*—C10*A*	1.480 (4)
N4*A*—C9	1.480 (4)	N8*A*—C9*A*	1.472 (4)
C9—N4*A*—C10*A*	113.0 (3)	C8—N8*A*—C10	107.3 (3)

All H atoms were constrained to ride on their parent atoms (C—H = 0.99 or 1.00 Å), with $U_{\rm iso}$ values of $1.2U_{\rm eq}$ of the parent atoms.

Data collection: *SHELXTL* (Siemens, 1983); cell refinement: *SHELXTL*; data reduction: *SHELXTL*; program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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