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8,11-Dihydroxypentacycloundecane-8,11-lactam

Hendrik G. Kruger, Melanie Rademeyer,* Thavendran Govender and Vikash Gokul

School of Chemistry, Howard College Campus, University of KwaZulu–Natal, Durban 4041, South Africa

Correspondence e-mail: rademeyerm@ukzn.ac.za

Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.039
wR factor = 0.107
Data-to-parameter ratio = 15.0

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

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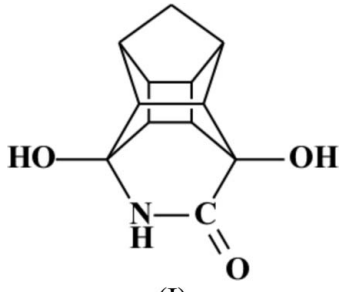
The title molecule, $C_{12}H_{13}NO_3$, is a pentacycloundecane cage derivative that exhibits C—C bond lengths deviating from the normal values. Neighbouring molecules interact *via* N—H···O and O—H···O hydrogen bonds, forming two-dimensional hydrogen-bonded sheets.

Comment

The present study is part of an ongoing investigation of the chemical reactivity and solid-state structures of polycyclic pentacycloundecane (PCU) cage derivatives.

The title compound, (I), was first synthesized by Martins *et al.* (1993). It forms a hydrate (1:1) complex with water, and the crystal structure of the hydrate was reported by Kruger *et al.* (1996). The crystal structure of the lactam hydrate was, however, disordered and more precise data for the structure of the lactam were required for our computational studies. The crystal structure of the pure lactam without water is reported here.



(I)

The molecule of (I) consists of a large apolar (lipophilic) hydrocarbon skeleton and more polar dihydroxy and lactam units (Fig. 1). A number of publications have focused on the molecular geometries of PCU cage derivatives (Flippen-Anderson *et al.*, 1991; Linden *et al.*, 2005; Kruger *et al.*, 2005), and it has been reported that these compounds exhibit C—C bond lengths deviating from the normal value of 1.54 Å.

In (I), the C9—C10 bond, as well as the bonds constituting the cyclobutane ring, C1—C2, C2—C6, C6—C7 and C7—C1, are longer than expected, while the bonds involving atoms C4 and C11 (Table 1) are shorter than expected. On the other hand, the C8—C12 bond is very short.

In the crystal structure of (I), molecules are arranged to form a bilayer consisting of alternating lipophilic and polar layers. The lipophilic layer contains the PCU cage groups, while the hydroxy, ketone and amino groups constitute the polar layer. In the polar layer, a complex intermolecular hydrogen-bonding network, involving atoms N, O2 and O3 as

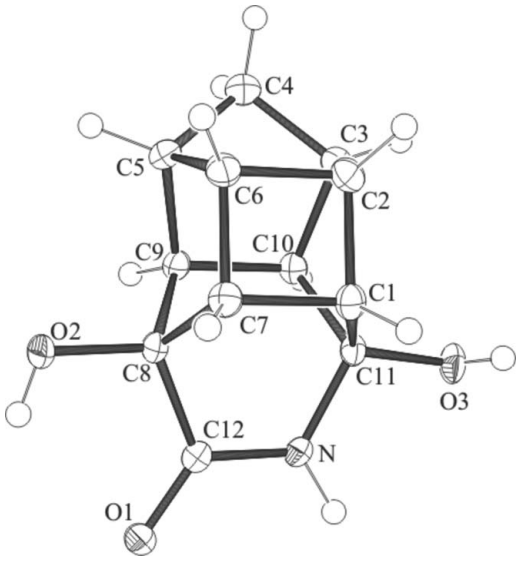


Figure 1
An *ORTEP-3* (Farrugia, 1997) drawing of the title compound, (I), with displacement ellipsoids drawn at the 50% probability level.

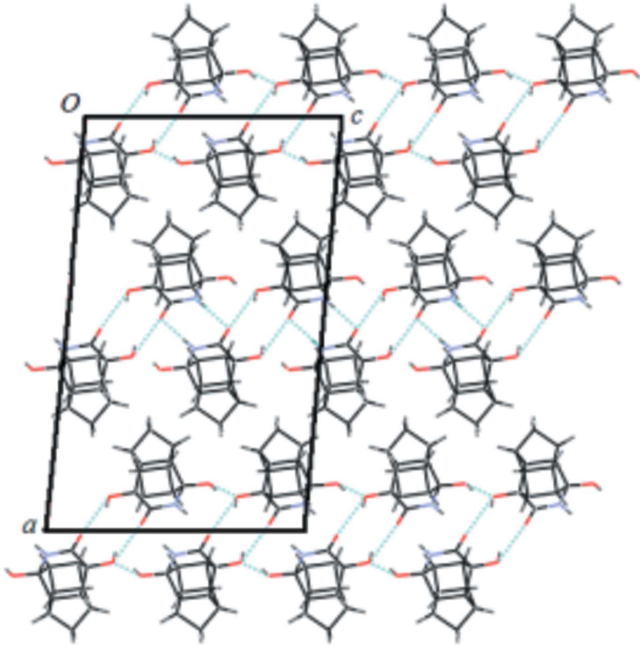


Figure 2
A packing diagram of (I), viewed down the *b* axis. The dashed lines denote hydrogen bonds.

hydrogen-bond donors and atoms O1 and O2 as hydrogen-bond acceptors, is formed (Table 2). These interactions result in two-dimensional hydrogen-bonded sheets of molecules (Fig. 2).

Experimental

The PCU lactam was synthesized as described by Martins *et al.* (1993) and recrystallized from dioxane.

Crystal data

$C_{12}H_{13}NO_3$
M_r = 219.23
Monoclinic, $C2/c$
a = 22.0040 (11) Å
b = 6.3299 (3) Å
c = 13.6153 (7) Å
β = 95.426 (3)°
V = 1887.88 (16) Å^3
Z = 8
D_x = 1.543 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 957 reflections
θ = 3.0–28.3°
μ = 0.11 mm^{-1}
T = 173 (2) K
Block, colourless
0.38 × 0.29 × 0.29 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
7588 measured reflections
2354 independent reflections
1973 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 28.3°
$h = -26 \rightarrow 29$
$k = -7 \rightarrow 8$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.107
S = 1.06
2354 reflections
157 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0585P)^2 + 0.9852P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Selected bond lengths (Å).

C12—C8	1.5189 (16)	C9—C5	1.5539 (16)
C8—C9	1.5442 (16)	C9—C10	1.5719 (15)
C8—C7	1.5455 (17)	C5—C4	1.5365 (17)
C7—C1	1.5654 (15)	C5—C6	1.5430 (18)
C7—C6	1.5655 (17)	C6—C2	1.5738 (16)
C1—C11	1.5341 (17)	C2—C3	1.5489 (17)
C1—C2	1.5588 (17)	C3—C4	1.5376 (17)
C11—C10	1.5368 (16)	C3—C10	1.5431 (16)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N—H1H···O1^{i}	0.92 (2)	1.99 (2)	2.9094 (13)	174 (2)
O2—H2H···O1^{ii}	0.83 (2)	2.19 (2)	2.9214 (13)	147 (2)
O2—H2H···O1	0.83 (2)	2.24 (2)	2.7213 (12)	118 (2)
O3—H3H···O2^{iii}	0.89 (2)	1.92 (2)	2.7844 (13)	164 (2)

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

Atoms H1H, H2H and H3H were located in a difference map and refined isotropically [N—H = 0.924 (17) Å and O—H = 0.83 (2) and 0.89 (2) Å]. The remaining H atoms were positioned geometrically [C—H = 1.00 (CH) and 0.99 Å (CH_2)] and constrained to ride on their parent atoms with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART-NT* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *MERCURY* (Bruno *et al.*, 2002) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2003).

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