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N-Isopropyl-*α*-glucoheptonamide

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Key indicators

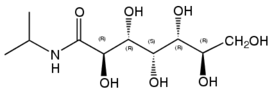
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.044
wR factor = 0.113
Data-to-parameter ratio = 9.1

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

The structure of *N*-isopropyl-D-*glycero*-D-*gulo*-heptonamide, $C_{10}H_{21}NO_7$, shows a bent C chain conformation in the glucoheptonic acid moiety. The crystal packing comprises a two-dimensional arrangement of intermolecular hydrogen bonds involving all O atoms. A feature of the crystal packing is a hydrogen-bonded $(OH)_4$ ring, which might be responsible for the observed conformation.

Comment

The aim of this study was to examine the crystal packing, the hydrogen-bonding arrangement and the conformation of *N*-isopropyl-*α*-glucoheptonamide, (I). Compound (I) was obtained from D-*glycero*-D-*gulo*-heptonic γ-lactone, which in turn was synthesized by Killiani's reaction with glucose. The stereochemistry of (I) was determined by correlation with glucose and was found to be 2*R*, 3*R*, 4*S*, 5*R* and 6*R*.



(I)

By analogy with other gluconamides (Darbon *et al.*, 1984; Darbon-Meyssonnier *et al.*, 1985; Oddon *et al.*, 1986), it was expected that the conformation of the glucoheptonic acid moiety would be a straight chain. Instead, the observed conformation is a bent chain in which the C atoms lie in two planes, C1/C2/C3 and C3/C4/C5/C6/C7, with an interplanar angle of 87.63 (8)° (Fig. 1 and Table 1). This conformation might be adopted as a result of the extended hydrogen-

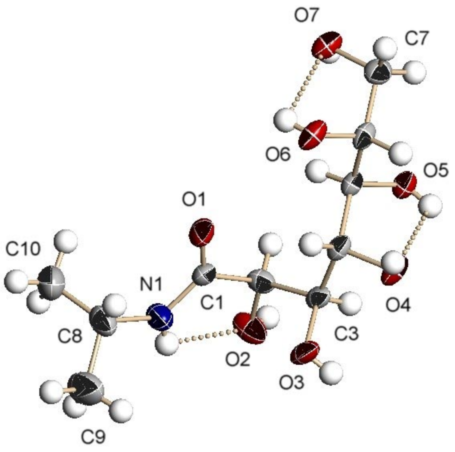


Figure 1
Perspective view of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Dashed lines indicate hydrogen bonds.

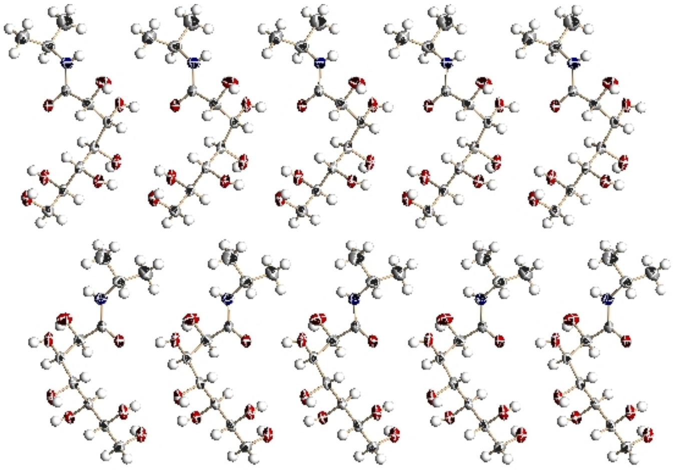


Figure 2
Crystal packing of (I), projected down the *a* axis, with the *c* axis horizontal. The non-polar edge between the layers can be seen.

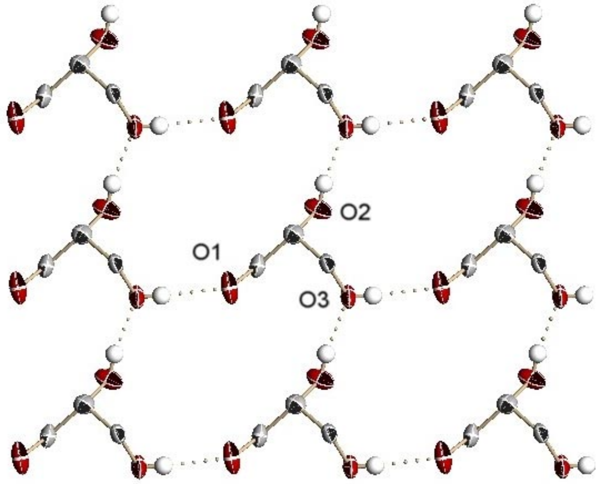


Figure 3
HB net formed by atoms O1, O2 and O3. The view is along the *b* axis, with the *c* axis horizontal. Some atoms have been omitted for clarity.

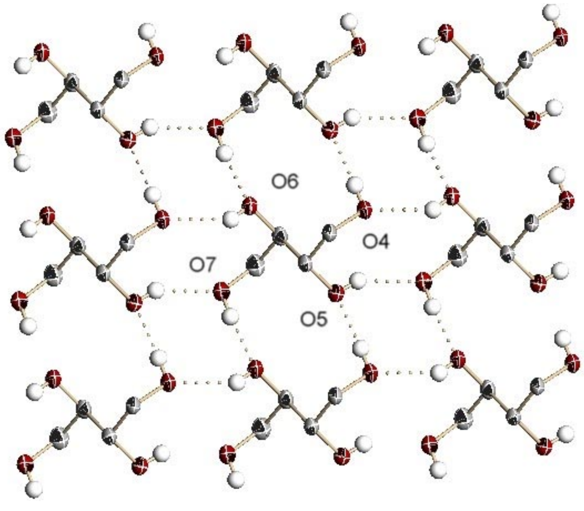


Figure 4
HB system generated by atoms O4, O5, O7 and O6, viewed along the *b* axis, with the *c* axis horizontal. Some atoms have been omitted for clarity.

bonding network stabilizing the crystal structure.

The crystal packing in (I) is based on monolayers stabilized by van der Waals forces. There is a non-polar edge between adjacent layers, created by directing the OH6 and OH7 groups away from the hydrophobic groups of neighboring molecules and leaving the lipophilic methylene H atoms facing the *N*-isopropyl groups (Fig. 2).

All hydroxyl groups of (I) participate in the formation of the two-dimensional intermolecular hydrogen-bond (HB) network, where all O atoms of these groups act both as donors and as acceptors, with the exception of atom O2, which acts only as an intermolecular HB donor; atom O2 also forms an intramolecular interaction (Table 2). This two-dimensional network lies parallel to the crystallographic *ac* plane. In the crystal packing, two motifs can be distinguished. One, formed by atoms O1, O2 and O3, generates 18-membered rings where four hydrogen bonds can be clearly identified (Fig. 3). The second motif, formed by atoms O4, O5, O6 and O7, leads to well defined $(OH)_4$ rings, where all the O atoms involved are derived from different molecules. Because of this, each molecule participates in the formation of four rings (Fig. 4). The eight-membered ring $(OH)_4$ has pseudo-C_i symmetry. This ring may be considered as a planar square according to the data in Table 3. A close resemblance of this motif to the most stable structure found for a water tetramer can be observed (Cruzan *et al.*, 1997). Since the latter was found to be a very stable structure ($E_{\rm int}$ = -25.93 kcal mol^{-1}) the rings found here might be considered as responsible for the observed packing. The average O···O distances are similar, 2.75 Å for this structure and 2.79 Å for the water ring, thus confirming the stability of the observed motif in (I). Furthermore, Supriya & Das (2003) recently reported a structure containing four water molecules, similarly arranged, where the O···O distance was found to be 2.77 Å.

Additionally, the structure exhibits three intramolecular HB interactions, *viz.* N1···O2, O5···O4 and O6···O7 (Table 2).

Experimental

A mixture of the γ-lactone of glucoheptonic acid (10.00 g, 48 mmol) and isopropylamine (4.1 ml, 48 mmol) in ethanol (30 ml) was heated to reflux for one hour. The crystalline product was collected by filtration and recrystallized from water/acetone to yield 6.00 g (47%) of (I) as colorless trigonal prismatic crystals (m.p. 434–435 K). $[\alpha]^{20}_D$ = +8.1° (*c* = 1.3, water).

Crystal data

$C_{10}H_{21}NO_7$
M_r = 267.28
Monoclinic, $P2_1$
a = 4.866 (2) Å
b = 22.435 (9) Å
c = 6.040 (2) Å
β = 90.664 (7)°
V = 659.3 (5) Å^3
Z = 2

D_x = 1.346 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1024 reflections
θ = 3.5–27.3°
μ = 0.11 mm^{-1}
T = 293 (2) K
Trigonal prism, colorless
0.40 × 0.20 × 0.05 mm

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Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
3915 measured reflections
1504 independent reflections
1361 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.037$
$\theta_{\rm max} = 27.3°$
$h = -6 \rightarrow 6$
$k = -28 \rightarrow 24$
$l = -7 \rightarrow 6$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.113$
$S = 1.00$
1504 reflections
165 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0755P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.24$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.18$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.227 (3)	C5—O5	1.435 (3)
C1—N1	1.328 (4)	C5—C6	1.530 (4)
C1—C2	1.521 (4)	C6—O6	1.427 (3)
C2—O2	1.415 (3)	C6—C7	1.512 (4)
C2—C3	1.528 (4)	C7—O7	1.408 (4)
C3—O3	1.414 (3)	C8—N1	1.461 (4)
C3—C4	1.532 (4)	C8—C9	1.508 (5)
C4—O4	1.427 (3)	C8—C10	1.510 (6)
C4—C5	1.514 (4)	N1—H1N	0.7671
O1—C1—N1	124.0 (3)	C4—C5—C6	111.1 (2)
O1—C1—C2	120.0 (2)	C7—C6—C5	113.1 (2)
N1—C1—C2	116.0 (2)	N1—C8—C9	108.6 (3)
C1—C2—C3	109.9 (2)	C1—N1—C8	124.4 (3)
C2—C3—C4	116.0 (2)	C1—N1—H1N	116.4
C5—C4—C3	115.7 (2)	C8—N1—H1N	118.8
O5—C5—C6	109.2 (2)		
N1—C1—C2—O2	8.3 (3)	C3—C4—C5—C6	172.9 (2)
N1—C1—C2—C3	−113.3 (3)	C4—C5—C6—C7	179.5 (2)
C1—C2—C3—C4	−64.1 (3)	O1—C1—N1—C8	−1.6 (5)
C2—C3—C4—C5	−55.6 (3)	C2—C1—N1—C8	176.5 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O2	0.77	2.17	2.581 (3)	114
O5—H5O···O4	0.77	2.40	2.784 (3)	113
O6—H6O···O7	0.75	2.55	2.878 (3)	109
O2—H2O···O3$^{\rm i}$	0.80	1.91	2.704 (3)	172
O3—H3O···O1$^{\rm ii}$	0.72	1.96	2.663 (3)	168
O4—H4O···O5$^{\rm iii}$	0.82	1.93	2.746 (3)	175
O5—H5O···O7$^{\rm ii}$	0.77	2.08	2.773 (3)	150
O6—H6O···O4$^{\rm iv}$	0.75	2.09	2.741 (3)	145
O7—H7O···O6$^{\rm i}$	0.82	1.93	2.734 (3)	167

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

Table 3
Deviations from the O7/O6$^{\rm i}$/O5$^{\rm ii}$/O4$^{\rm iii}$ plane for the atoms present in the $(OH)_4$ ring (Å).

r.m.s. deviation of fitted atoms = 0.033 Å.

O7	0.0324 (11)	H7O	0.1427 (13)	C7	−0.004 (4)
O6$^{\rm i}$	−0.0328 (11)	H6O$^{\rm i}$	0.0012 (28)	C6$^{\rm i}$	0.749 (4)
O5$^{\rm iv}$	−0.0323 (11)	H5O$^{\rm iv}$	−0.1727 (18)	C5$^{\rm iv}$	−0.820 (4)
O4$^{\rm v}$	0.0327 (11)	H4O$^{\rm v}$	−0.0405 (28)	C4$^{\rm v}$	−0.059 (4)

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

The positions of the non-methyl H atoms were obtained from a difference Fourier map and restrained to ride on the parent atoms in the final refinement. The range of C—H bond distances was 0.88–1.04 Å, with a mean value of 0.95 Å. The O—H distances are in the range 0.72–0.82 Å, with a mean value of 0.78 Å. The N—H bond distance was 0.77 Å. A common $U_{\rm iso}$(H) value was adopted for C-bound H atoms. The methyl H atoms were included in the riding-model approximation, with C—H distances of 0.96 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm iso}({\rm C})$. For the set of O- and N-bound H atoms, $U_{\rm iso}({\rm H}) = 1.5U_{\rm iso}({\rm O,N})$. The absolute configuration was assigned on the basis of that of the starting material. Friedel pairs were merged.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL* and *enCIFer* (Allen *et al.*, 2004).

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