

Sodium α -glucoheptonate dihydrate

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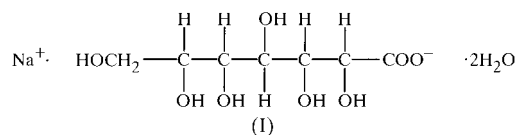
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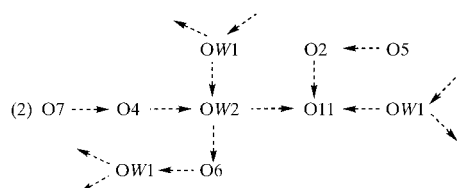
In the structure of sodium *D*-glycero-*D*-gulo-heptonate dihydrate, $\text{Na}^+\cdot\text{C}_7\text{H}_{13}\text{O}_8^-\cdot 2\text{H}_2\text{O}$, the glucoheptonate anion has a bent carbon chain conformation. There are extensive intermolecular hydrogen bonds involving all the hydroxy and water H atoms. The Na^+ cation has a distorted octahedral coordination to six O atoms, with $\text{Na}^+\cdots\text{O}$ distances ranging from 2.316 (2) to 2.645 (2) Å.

Comment

The primary aim of this study was to examine the acyclic carbon-chain conformation in the *D*-glycero-*D*-gulo-heptonate anion of the title compound, (I). Crystal structure studies of *meso*-*L*-glycero-*L*-gulo-heptitol (Nimgirawath *et al.*, 1976) and of other alditols (Park *et al.*, 1971, 1989; Panagiotopoulos *et al.*, 1974) led to the expectation that, because of its DDLDD configuration, the *D*-glycero-*D*-gulo-heptonate anion should exist as the bent conformer. This conformational preference has its origin in the repulsion of *syn*-axially related hydroxyl groups, parallel to C3—OH and C5—OH, in the straight-chain conformer.



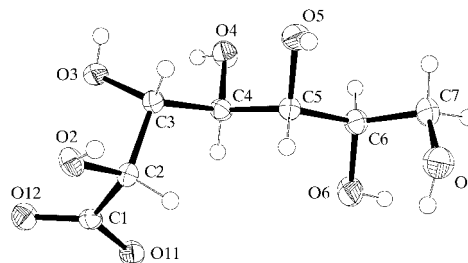
The anion of (I) has a bent-chain conformation. This conformation can be derived from the zigzag planar conformation by two consecutive rotations around the C3—C4 and C2—C3 bonds. This bent-chain conformation was predicted

 (1) O3 \cdots O12


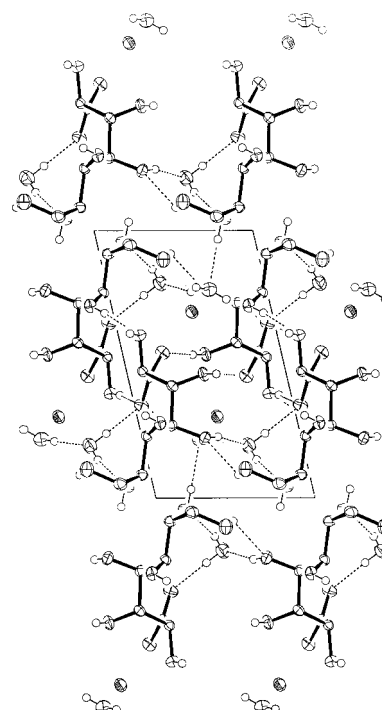
for the polyhydroxy alkyl chain of *D*-glycero-*D*-gulo-heptitol by Jeffrey & Kim (1970). In the bent conformer of (I), the C atoms lie in two planes, C1—C3 and C3—C7, with an interplanar angle of 71.2 (2)°.

The Na^+ cations occupy the voids between the hydrogen-bonded anions. The sixfold coordination of each Na^+ cation consists of O2, O3, O5, O7, O12 and OW1, with $\text{Na}^+\cdots\text{O}$ distances ranging from 2.316 (2) to 2.645 (2) Å, making a distorted octahedral geometry.

The molecular packing in the crystal of (I) appears to be determined mainly by hydrogen bonds, since there are ten hydrogen bonds per asymmetric unit. The hydrogen-bond distances and angles are listed in Table 2. The molecular packing projected down the *b* axis is shown in Fig. 2. All six hydroxy groups and the water molecules act as hydrogen-bond donors. Atoms O2, O4 and O6 are hydrogen-bond acceptors; O3, O5 and O7 do not accept any hydrogen bonds. Atom OW2 accepts two hydrogen bonds, while OW1 accepts one. The O11 carboxylate accepts three hydrogen bonds and O12 one. This may account for the small difference in bond length of 0.037 Å by which the C1—O11 distance is longer than C1—O12. Each molecule is linked by two kinds of hydrogen bonds, one donor–acceptor hydrogen bond and one branched hydrogen bond.


Figure 1

A view of the glucoheptonate anion of (I) showing the atomic numbering. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.


Figure 2

The crystal packing of (I) projected down the *b* axis, with the *c* axis horizontal. Hydrogen bonds are shown as thin dashed lines.

Experimental

Crystals of (I) (m.p. 437 K) were obtained from Aldrich Chemical Company Inc. and no further recrystallization was carried out.

Crystal data

$\text{Na}^+ \cdot \text{C}_7\text{H}_{13}\text{O}_8^- \cdot 2\text{H}_2\text{O}$	$D_x = 1.593 \text{ Mg m}^{-3}$
$M_r = 284.20$	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 25 reflections
$a = 6.181 (1) \text{ \AA}$	$\theta = 15.7\text{--}24.7^\circ$
$b = 9.200 (1) \text{ \AA}$	$\mu = 0.18 \text{ mm}^{-1}$
$c = 10.691 (1) \text{ \AA}$	$T = 292 (2) \text{ K}$
$\beta = 102.94 (1)^\circ$	Prism, colourless
$V = 592.4 (1) \text{ \AA}^3$	$0.65 \times 0.50 \times 0.28 \text{ mm}$
$Z = 2$	

Data collection

Enraf-Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.024$
$\omega/2\theta$ scans	$\theta_{\text{max}} = 25.98^\circ$
Absorption correction: via ψ scans (North <i>et al.</i> , 1968)	$h = 0 \rightarrow 7$
$T_{\text{min}} = 0.80, T_{\text{max}} = 0.95$	$k = 0 \rightarrow 11$
1359 measured reflections	$l = -13 \rightarrow 12$
1243 independent reflections	3 standard reflections
1206 reflections with $I > 2\sigma(I)$	frequency: 60 min
	intensity decay: 2.2%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0568P)^2 + 0.0911P]$
$R[F^2 > 2\sigma(F^2)] = 0.032$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.090$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.28$	$\Delta\rho_{\text{max}} = 0.32 \text{ e \AA}^{-3}$
1243 reflections	$\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$
231 parameters	
All H-atom parameters refined	

Table 1

Selected geometric parameters (\AA , $^\circ$).

Na—O5 ⁱ	2.316 (2)	C2—C3	1.532 (3)
Na—O7 ⁱⁱ	2.382 (3)	C3—O3	1.423 (3)
Na—O12	2.416 (2)	C3—C4	1.545 (4)
Na—O2	2.420 (2)	C4—O4	1.425 (3)
Na—OW1	2.421 (3)	C4—C5	1.521 (4)
Na—O3	2.645 (2)	C5—O5	1.425 (3)
C1—O12	1.235 (3)	C5—C6	1.537 (3)
C1—O11	1.272 (3)	C6—O6	1.428 (4)
C1—C2	1.538 (4)	C6—C7	1.517 (4)
C2—O2	1.434 (3)	C7—O7	1.429 (4)
O12—C1—O11	125.5 (3)	O4—C4—C3	110.1 (2)
O12—C1—C2	118.7 (2)	C5—C4—C3	110.1 (2)
O11—C1—C2	115.8 (2)	O5—C5—C4	107.6 (2)
O2—C2—C3	109.7 (2)	O5—C5—C6	108.6 (2)
O2—C2—C1	107.3 (2)	C4—C5—C6	114.6 (2)
C3—C2—C1	110.3 (2)	O6—C6—C7	110.6 (2)
O3—C3—C2	107.5 (2)	O6—C6—C5	106.6 (2)
O3—C3—C4	112.4 (2)	C7—C6—C5	112.0 (2)
C2—C3—C4	113.0 (2)	O7—C7—C6	112.5 (2)
O4—C4—C5	107.3 (2)		
O12—C1—C2—C3	−95.4 (3)	C3—C4—C5—C6	175.6 (2)
O11—C1—C2—C3	84.6 (3)	C4—C5—C6—C7	173.4 (2)
C1—C2—C3—C4	−78.2 (3)	C5—C6—C7—O7	60.4 (3)
C2—C3—C4—C5	−81.9 (3)		

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

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
O2—H2O \cdots O11 ⁱ	0.79 (5)	1.93 (5)	2.714 (4)	173 (4)
O3—H3O \cdots O12 ⁱⁱ	0.73 (5)	2.04 (5)	2.771 (4)	172 (5)
O4—H4O \cdots OW2 ⁱⁱⁱ	0.78 (5)	2.11 (5)	2.872 (4)	165 (4)
O5—H5O \cdots O2 ⁱ	0.73 (3)	2.01 (3)	2.730 (3)	172 (4)
O6—H6O \cdots OW1 ^{iv}	0.82 (4)	2.02 (4)	2.837 (3)	176 (3)
O7—H7O \cdots O4 ^v	0.85 (6)	2.31 (5)	2.854 (4)	122 (4)
OW1—H11W \cdots O11 ⁱⁱ	0.79 (4)	2.09 (4)	2.865 (3)	166 (4)
OW1—H12W \cdots OW2 ⁱ	0.83 (5)	2.06 (5)	2.887 (3)	175 (4)
OW2—H21W \cdots O11	0.85 (4)	1.90 (5)	2.747 (3)	177 (5)
OW2—H22W \cdots O6	0.73 (5)	2.20 (5)	2.893 (4)	160 (5)

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

We did not determine the absolute configuration but assumed the D-form, corresponding to the configuration as supplied by the Aldrich Chemical Company Inc. The C—H and O—H bond distances range from 0.92 (4) to 1.00 (4) \AA and from 0.73 (5) to 0.85 (4) \AA , respectively.

Data collection: *CAD-4-PC Software* (Enraf-Nonius, 1992); cell refinement: *SET4* and *CELDIM* in *CAD-4-PC Software*; data reduction: *WinGX* (Farrugia, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1543). Services for accessing these data are described at the back of the journal.

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