Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

myo-Inositol dihydrate: a redetermination

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Key indicators

Single-crystal X-ray study
T = 180 K
Mean σ(C–C) = 0.002 Å
R factor = 0.032
wR factor = 0.101
Data-to-parameter ratio = 13.1

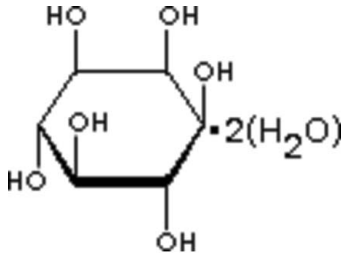
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 22 May 2006
Accepted 9 June 2006

The crystal structure of *myo*-inositol dihydrate, $C_6H_{12}O_6\cdot 2H_2O$, previously reported by Lomer, Miller & Beevers [*Acta Cryst.* (1963), **16**, 264–268], has been redetermined, and the positions of the H atoms of the hydroxyl groups were located, showing an ordered hydrogen-bonding scheme.

Comment

myo-Inositol (Fig. 1) is a biological molecule of nutritional and medical importance, which has been extracted from both plant and animal sources (Posternak, 1965). The crystal structure of the anhydrous form has previously been determined (Rabinovich & Kraut, 1964) and that of the dihydrate by Lomer *et al.* (1963).



(I)

In a series of experiments aimed at inhibiting the crystallization of *myo*-inositol from solution, we evaporated aqueous solutions of varying concentrations of *myo*-inositol and polyvinylpyrrolidone. Needle-shaped crystals formed as the solutions became more concentrated at room temperature. We

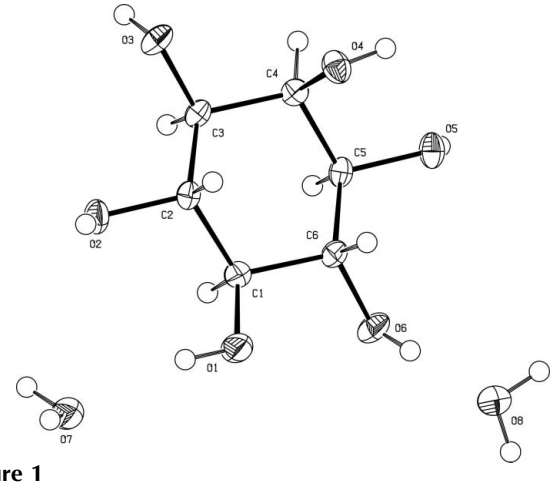


Figure 1
The aymmetric unit of *myo*-inositol. Displacement ellipsoids are drawn at the 50% probability level.

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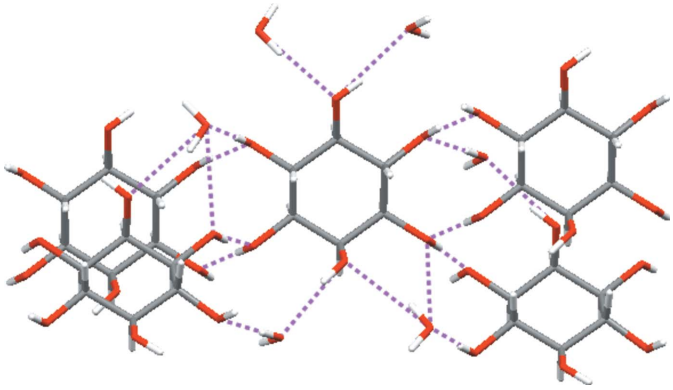

Figure 2
myo-Inositol dihydrate, showing hydrogen-bonds (dashed lines) to the neighbouring molecules.

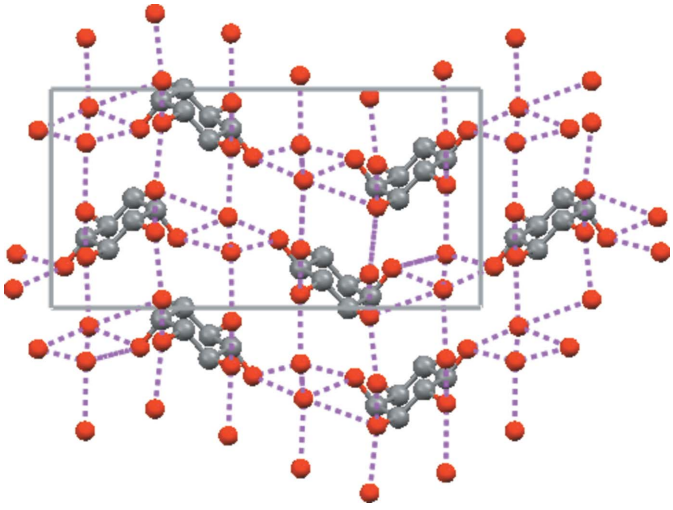

Figure 3
Packing diagram viewed in *a* axis projection, with *b* horizontal and *c* vertical. H atoms have been omitted for clarity. Hydrogen bonds are shown as dashed lines.

obtained the structure by single-crystal X-ray diffraction analysis at 180 K, confirming the dihydrate structure, with well located H atoms and a rational hydrogen-bonding scheme.

Each *myo*-inositol molecule forms a total of 13 intermolecular hydrogen bonds, defined as having O···O less than 3.04 Å. All 13 hydrogen bonds have normal bond lengths and geometry. The immediate hydrogen-bonded neighbours are six water molecules and four *myo*-inositol molecules (Fig. 2). Both water molecules show an optimal hydrogen-bond environment of two donor and two acceptor bonds. Each hydroxyl group on the inositol has a donor and acceptor hydrogen bond, with one (O3) forming (as an acceptor) a third hydrogen bond. The packing diagram (Fig. 3) shows an interesting feature where the water molecules link the inositol molecules in the *b*-axis direction, forming four-membered ring motifs $H_2O\cdots OH\cdots H_2O\cdots OH$.

Experimental

An aqueous solution (10 ml) of *myo*-inositol (0.426 g) and polyvinylpyrrolidone (0.631 g) was prepared. The colourless solution was allowed to evaporate at room temperature. When the solution had reduced to about half its initial volume, white needle-shaped crystals were observed and analysed by single-crystal X-ray diffraction. The crystals dehydrate prior to melting at 469 K.

Crystal data

$C_6H_{12}O_6\cdot 2H_2O$
M_r = 216.19
Monoclinic, $P2_1/n$
a = 6.6099 (2) Å
b = 16.6009 (4) Å
c = 9.0264 (2) Å
β = 110.751 (1)°
V = 926.22 (4) Å^3
Z = 4
D_x = 1.550 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.15 mm^{-1}
T = 180 (2) K
Block cut from needle, white
0.46 × 0.35 × 0.23 mm

Data collection

Nonius KappaCCD diffractometer
Thin-slice ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.905, T_{max} = 0.968
7754 measured reflections
2104 independent reflections
1907 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.101
S = 1.14
2104 reflections
161 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 0.2568P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*A*···O7	0.82 (2)	2.02 (2)	2.8259 (13)	168 (2)
O2—H2*A*···O6^{i}	0.81 (2)	1.92 (2)	2.7275 (12)	175 (2)
O3—H3*A*···O5^{ii}	0.82 (2)	1.83 (2)	2.6454 (12)	173 (2)
O4—H4*A*···O7^{iii}	0.81 (2)	2.02 (2)	2.8207 (12)	170 (2)
O5—H5*A*···O3^{iv}	0.84 (2)	1.80 (2)	2.6359 (12)	172 (2)
O6—H6*A*···O8	0.81 (2)	1.93 (2)	2.7365 (13)	176 (2)
O7—H7*A*···O8^{v}	0.82 (2)	2.18 (2)	2.9903 (14)	169 (2)
O7—H7*B*···O2^{vi}	0.84 (2)	2.01 (2)	2.8442 (13)	179 (2)
O8—H8*A*···O4^{vii}	0.82 (2)	2.23 (2)	3.0141 (13)	160 (2)
O8—H8*A*···O3^{vii}	0.82 (2)	2.45 (2)	3.0272 (13)	128 (2)
O8—H8*B*···O1^{viii}	0.83 (2)	2.03 (2)	2.8525 (13)	172 (2)

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

All OH H atoms were located in the final difference map without any difficulty. The positions of the H atoms were refined independently and successfully, with a single O—H bond-length restraint [O—H = 0.807 (16)–0.844 (17) Å] and common U_{iso}(H) values for similar atoms [U_{iso}(H) = 0.44 (2) Å^2 for OH and U_{iso}(H) = 0.52 (3) Å^2 for H_2O]. The remaining H atoms were positioned geometrically, with C—H = 1.00 Å, and refined as riding with a common displacement parameter [U_{iso}(H) = 0.206 (14) Å^2].

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *MERCURY* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97*.

The authors are grateful to Dr John Davies (Department of Chemistry, University of Cambridge) for determining the crystal structure, and Drs A. Trask and L. Fabian for help with CIF preparation. The Pfizer Institute for Pharmaceutical Materials Science is acknowledged for funding the work.

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supporting information

Acta Cryst. (2006). E**62**, o2902–o2904 [https://doi.org/10.1107/S1600536806022033]

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$c = 9.0264$ (2) Å
$\beta = 110.751$ (1)°
$V = 926.22$ (4) Å^3
$Z = 4$
$F(000) = 464$
$D_x = 1.550$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5712 reflections
$\theta = 1.0$–$27.5°$
$\mu = 0.15$ mm^{-1}
$T = 180$ K
Needle, white
0.46 × 0.35 × 0.23 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
thin slice ω and φ scans
Absorption correction: multi-scan (SORTAV; Blessing, 1995)
$T_{min} = 0.905$, $T_{max} = 0.968$
7754 measured reflections
2104 independent reflections
1907 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.7°$
$h = -8 \rightarrow 8$
$k = -21 \rightarrow 21$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.101$
$S = 1.14$
2104 reflections
161 parameters
10 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 0.2568P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}

Special details

Experimental. Previous report: MYTOLD in CSD.
Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.28308 (15)	0.46905 (5)	0.31146 (11)	0.0298 (2)
H1A	0.367 (3)	0.4968 (11)	0.285 (2)	0.044 (2)*
C1	0.16681 (18)	0.41915 (6)	0.18041 (13)	0.0186 (2)
H1	0.0799	0.4544	0.0905	0.0206 (14)*
O2	0.44085 (14)	0.42063 (5)	0.06268 (10)	0.0227 (2)
H2A	0.563 (3)	0.4227 (11)	0.126 (2)	0.044 (2)*
C2	0.32068 (17)	0.36849 (6)	0.12653 (12)	0.0167 (2)
H2	0.4226	0.3388	0.2194	0.0206 (14)*
O3	0.34860 (13)	0.25832 (5)	−0.03785 (10)	0.0215 (2)
H3A	0.315 (3)	0.2566 (11)	−0.135 (2)	0.044 (2)*
C3	0.19698 (17)	0.30839 (6)	−0.00098 (12)	0.0168 (2)
H3	0.1095	0.3387	−0.0984	0.0206 (14)*
O4	0.17370 (13)	0.20684 (5)	0.18073 (10)	0.0223 (2)
H4A	0.092 (3)	0.1746 (10)	0.198 (2)	0.044 (2)*
C4	0.04578 (17)	0.25630 (6)	0.05213 (13)	0.0168 (2)
H4	−0.0400	0.2209	−0.0379	0.0206 (14)*
O5	−0.24375 (14)	0.25828 (5)	0.15365 (10)	0.0215 (2)
H5A	−0.370 (3)	0.2604 (11)	0.086 (2)	0.044 (2)*
C5	−0.10927 (17)	0.30913 (6)	0.10039 (12)	0.0168 (2)
H5	−0.2021	0.3403	0.0060	0.0206 (14)*
O6	−0.13972 (14)	0.41902 (5)	0.26532 (11)	0.0238 (2)
H6A	−0.119 (3)	0.4181 (11)	0.359 (2)	0.044 (2)*
C6	0.01239 (17)	0.36792 (6)	0.23186 (12)	0.0170 (2)
H6	0.0964	0.3370	0.3294	0.0206 (14)*
O7	0.56987 (15)	0.58094 (5)	0.25510 (12)	0.0272 (2)
H7A	0.698 (3)	0.5847 (12)	0.310 (2)	0.052 (3)*
H7B	0.566 (3)	0.5798 (12)	0.162 (2)	0.052 (3)*
O8	−0.05058 (16)	0.41219 (6)	0.58513 (12)	0.0300 (2)
H8A	−0.119 (3)	0.3725 (10)	0.595 (2)	0.052 (3)*
H8B	−0.107 (3)	0.4492 (13)	0.620 (2)	0.052 (3)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0305 (5)	0.0288 (5)	0.0372 (5)	−0.0131 (4)	0.0207 (4)	−0.0156 (4)
C1	0.0193 (5)	0.0172 (5)	0.0210 (5)	−0.0006 (4)	0.0094 (4)	−0.0016 (4)
O2	0.0193 (4)	0.0267 (4)	0.0249 (4)	−0.0054 (3)	0.0114 (3)	0.0009 (3)
C2	0.0151 (5)	0.0192 (5)	0.0171 (5)	−0.0010 (4)	0.0074 (4)	0.0007 (4)
O3	0.0172 (4)	0.0296 (4)	0.0192 (4)	0.0036 (3)	0.0081 (3)	−0.0044 (3)
C3	0.0144 (5)	0.0207 (5)	0.0162 (5)	0.0019 (4)	0.0067 (4)	−0.0005 (4)

O4	0.0194 (4)	0.0205 (4)	0.0268 (4)	0.0015 (3)	0.0080 (3)	0.0057 (3)
C4	0.0147 (5)	0.0190 (5)	0.0161 (5)	−0.0005 (4)	0.0046 (4)	−0.0016 (4)
O5	0.0146 (4)	0.0281 (4)	0.0221 (4)	−0.0037 (3)	0.0070 (3)	0.0031 (3)
C5	0.0134 (5)	0.0211 (5)	0.0168 (5)	−0.0003 (4)	0.0063 (4)	0.0018 (4)
O6	0.0200 (4)	0.0290 (5)	0.0258 (4)	0.0038 (3)	0.0124 (4)	−0.0044 (3)
C6	0.0148 (5)	0.0195 (5)	0.0184 (5)	0.0014 (4)	0.0079 (4)	−0.0007 (4)
O7	0.0265 (5)	0.0271 (5)	0.0306 (5)	−0.0033 (3)	0.0134 (4)	−0.0051 (4)
O8	0.0356 (5)	0.0245 (5)	0.0354 (5)	−0.0028 (4)	0.0194 (4)	−0.0063 (4)

Geometric parameters (Å, º)

O1—C1	1.4255 (13)	O4—H4A	0.813 (16)
O1—H1A	0.820 (16)	C4—C5	1.5250 (14)
C1—C6	1.5221 (14)	C4—H4	1.0000
C1—C2	1.5261 (14)	O5—C5	1.4271 (13)
C1—H1	1.0000	O5—H5A	0.844 (17)
O2—C2	1.4271 (12)	C5—C6	1.5267 (14)
O2—H2A	0.807 (16)	C5—H5	1.0000
C2—C3	1.5237 (14)	O6—C6	1.4273 (13)
C2—H2	1.0000	O6—H6A	0.809 (16)
O3—C3	1.4292 (13)	C6—H6	1.0000
O3—H3A	0.824 (17)	O7—H7A	0.821 (17)
C3—C4	1.5218 (14)	O7—H7B	0.836 (17)
C3—H3	1.0000	O8—H8A	0.820 (15)
O4—C4	1.4287 (13)	O8—H8B	0.83 (2)
C1—O1—H1A	107.8 (13)	O4—C4—C3	108.47 (8)
O1—C1—C6	107.11 (9)	O4—C4—C5	111.26 (8)
O1—C1—C2	111.13 (9)	C3—C4—C5	110.24 (9)
C6—C1—C2	112.58 (9)	O4—C4—H4	108.9
O1—C1—H1	108.6	C3—C4—H4	108.9
C6—C1—H1	108.6	C5—C4—H4	108.9
C2—C1—H1	108.6	C5—O5—H5A	107.8 (13)
C2—O2—H2A	107.7 (13)	O5—C5—C4	108.54 (8)
O2—C2—C3	108.58 (8)	O5—C5—C6	109.61 (8)
O2—C2—C1	108.88 (8)	C4—C5—C6	111.56 (8)
C3—C2—C1	111.20 (8)	O5—C5—H5	109.0
O2—C2—H2	109.4	C4—C5—H5	109.0
C3—C2—H2	109.4	C6—C5—H5	109.0
C1—C2—H2	109.4	C6—O6—H6A	110.0 (13)
C3—O3—H3A	108.6 (13)	O6—C6—C1	109.34 (9)
O3—C3—C4	109.62 (9)	O6—C6—C5	109.14 (8)
O3—C3—C2	108.89 (8)	C1—C6—C5	110.21 (8)
C4—C3—C2	111.76 (8)	O6—C6—H6	109.4
O3—C3—H3	108.8	C1—C6—H6	109.4
C4—C3—H3	108.8	C5—C6—H6	109.4
C2—C3—H3	108.8	H7A—O7—H7B	105 (2)
C4—O4—H4A	107.1 (13)	H8A—O8—H8B	103 (2)

O1—C1—C2—O2	−66.81 (11)	O4—C4—C5—O5	−57.95 (11)
C6—C1—C2—O2	173.04 (8)	C3—C4—C5—O5	−178.33 (8)
O1—C1—C2—C3	173.60 (9)	O4—C4—C5—C6	62.93 (11)
C6—C1—C2—C3	53.45 (11)	C3—C4—C5—C6	−57.46 (11)
O2—C2—C3—O3	64.86 (11)	O1—C1—C6—O6	63.29 (11)
C1—C2—C3—O3	−175.37 (8)	C2—C1—C6—O6	−174.27 (8)
O2—C2—C3—C4	−173.89 (8)	O1—C1—C6—C5	−176.74 (8)
C1—C2—C3—C4	−54.12 (11)	C2—C1—C6—C5	−54.30 (11)
O3—C3—C4—O4	54.92 (11)	O5—C5—C6—O6	−63.34 (11)
C2—C3—C4—O4	−65.91 (11)	C4—C5—C6—O6	176.42 (8)
O3—C3—C4—C5	176.97 (8)	O5—C5—C6—C1	176.57 (8)
C2—C3—C4—C5	56.14 (11)	C4—C5—C6—C1	56.33 (11)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
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O7—H7*A*···O8^{v}	0.82 (2)	2.18 (2)	2.9903 (14)	169 (2)
O7—H7*B*···O2^{vi}	0.84 (2)	2.01 (2)	2.8442 (13)	179 (2)
O8—H8*A*···O4^{vii}	0.82 (2)	2.23 (2)	3.0141 (13)	160 (2)
O8—H8*A*···O3^{vii}	0.82 (2)	2.45 (2)	3.0272 (13)	128 (2)
O8—H8*B*···O1^{viii}	0.83 (2)	2.03 (2)	2.8525 (13)	172 (2)

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) *x*+1/2, −*y*+1/2, *z*−1/2; (iii) −*x*+1/2, *y*−1/2, −*z*+1/2; (iv) *x*−1, *y*, *z*; (v) −*x*+1, −*y*+1, −*z*+1; (vi) −*x*+1, −*y*+1, −*z*; (vii) *x*−1/2, −*y*+1/2, *z*+1/2; (viii) −*x*, −*y*+1, −*z*+1.