

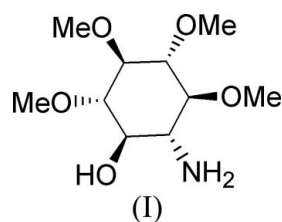
Graeme J. Gainsford,* Cornelis
Lensink, Andrew Falshaw and
Simon P.H. MeeIndustrial Research Limited, PO Box 31-310,
Lower Hutt, New Zealand

Correspondence e-mail: g.gainsford@irl.cri.nz

Key indicators

Single-crystal X-ray study
 $T = 88\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$
Disorder in main residue
 R factor = 0.033
 wR factor = 0.065
Data-to-parameter ratio = 9.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.1L-1-Amino-1-deoxy-2,3,4,5-tetra-*O*-methyl-
scyllo-inositolIn the crystal structure of the title compound, $\text{C}_{10}\text{H}_{21}\text{NO}_5$,
molecules are linked by strong $\text{N}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{N}$
hydrogen bonds into infinite zigzag chains.Received 26 October 2006
Accepted 30 October 2006

Comment

This study is part of a programme aimed at generating new
hydrogenation catalyst ligands (Gainsford *et al.*, 2006, and
references therein). The asymmetric unit of the title
compound, (I), contains one independent 1L-1-amino-1-de-
oxy-2,3,4,5-tetra-*O*-methyl-scyllo-inositol molecule (Fig. 1).
The absolute configuration shown (1*R*,2*S*,3*R*,4*R*,5*S*, 6*R*) is that
determined from the chemical synthesis.

Two related *scyllo*-inositol compounds have been reported, *viz.* BEKNAG (Solomons *et al.*, 1998) and ZOSSEF (Anderson *et al.*, 1995) [CSD refcodes; Version 5.27, with August 2006 updates; Allen, 2002]. Three others based on *scyllo*-inositol involve fused ring structures, *e.g.* the isopropylidene adduct TERCIC (Lampe *et al.*, 1996). The inositol ring adopts a slightly flattened chair conformation with $Q = 0.556(3)\text{ \AA}$, $\theta = 174.4(3)^\circ$ and $\varphi = 168(2)^\circ$ (Cremer & Pople, 1975). The flattening is seen in the ring torsion angles involving C4 [average absolute value is $50.5(3)^\circ$] and was also noted in BEKNAG. One other minor perturbation involves the normally eclipsed methyl on C3, which has a significant twist about the C3—O3 bond; see Table 1 ($>18^\circ$; Anderson *et al.*, 1995). Other distances and angles are normal.

The crystal structure (Fig. 2) is dominated by strong $\text{N}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds, linking molecules by way of an $R_2^2(10)$ motif (Bernstein *et al.*, 1995). A weaker $\text{C}-\text{H}\cdots\text{O}$ interaction provides some linking along the *a* axis (Table 2).

Experimental

1D-3,4,5,6-Tetra-*O*-methyl-*myo*-inositol (2.60 g, 11.0 mmol), prepared in a similar manner to the tetra-*O*-benzyl analogue (Wewers *et al.*, 2005), was dissolved in toluene (130 ml) and dibutyltin(IV) oxide (3.29 g, 13.2 mmol) was added. The solution was heated to reflux using a Dean–Stark apparatus for 2 h and then the solvent was

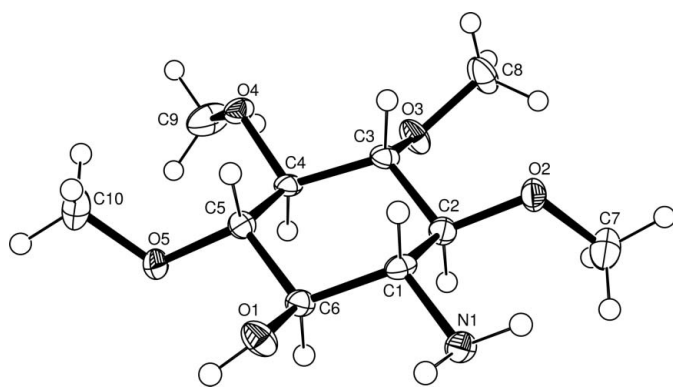


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level.

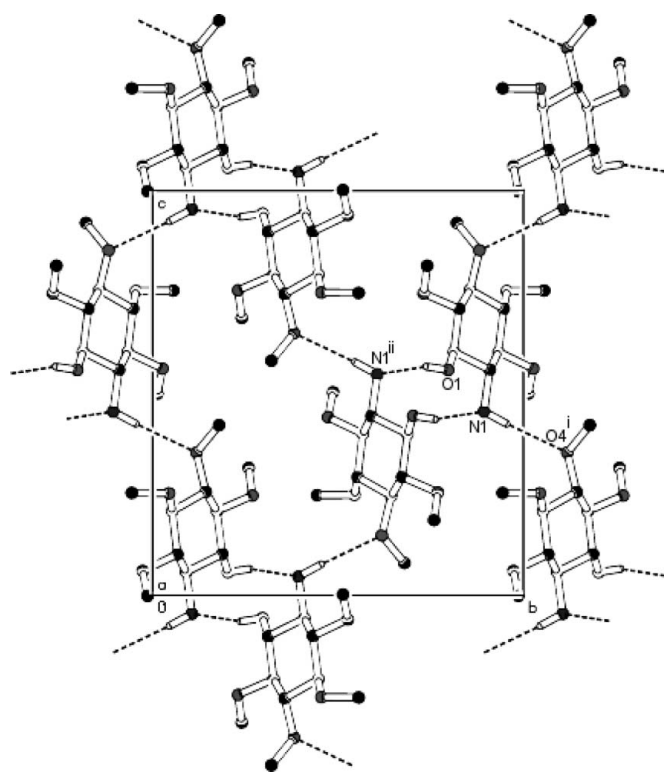


Figure 2
Packing diagram of (I), viewed down the *a* axis. Only H atoms involved in hydrogen bonds (dashed lines) are shown. For symmetry codes see Table 2.

removed. Dimethylformamide (80 ml) was added and the solution was cooled to 273 K. Caesium fluoride (3.35 g, 22.0 mmol) and benzyl bromide (2.63 ml, 22.0 mmol) were added; the suspension was stirred at 273 K for 10 min and then at room temperature for 3 h to give 1-*O*-benzyl-3,4,5,6-tetra-*O*-methyl-*myo*-inositol (3.47 g, 97%). The benzyl inositol (3.34 g, 10.2 mmol) was treated with methanesulfonyl chloride (3.99 ml, 51.2 mmol) in pyridine (50 ml) overnight at room temperature to give the mesylate (3.73 g, 90%). The mesylate (3.73 g, 9.23 mmol) was then subjected to a displacement reaction using sodium azide (4.20 g, 64.6 mmol) in dimethylformamide (37 ml) overnight at 383 K to give the azide (2.44 g, 75%). The azide (2.44 g, 7.51 mmol) was stirred in methanol (100 ml) with 10% palladium on carbon (1.00 g) under one atmosphere of hydrogen at room

temperature for 4 d to give the amino alcohol (1.31 g, 80%). Crystals of the title compound were isolated from dichloromethane.

Crystal data

$C_{10}H_{21}NO_5$
 $M_r = 235.28$
Orthorhombic, $P2_12_12_1$
 $a = 6.7256$ (14) Å
 $b = 12.920$ (3) Å
 $c = 14.091$ (3) Å
 $V = 1224.4$ (5) Å³

$Z = 4$
 $D_x = 1.276$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 88$ (2) K
Needle, colourless
 $0.50 \times 0.10 \times 0.05$ mm

Data collection

Bruker–Nonius APEX2 CCD area-detector diffractometer
 φ and ω scans
Absorption correction: multi-scan (Blessing, 1995)
 $T_{\min} = 0.888$, $T_{\max} = 1.0$

10832 measured reflections
1471 independent reflections
913 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.064$
 $\theta_{\max} = 26.4^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.065$
 $S = 0.83$
1471 reflections
161 parameters

H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0338P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.15$ e Å⁻³
 $\Delta\rho_{\min} = -0.18$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

O1–C6	1.431 (3)	N1–C1	1.475 (3)
O1–H1O	0.83 (3)	N1–H1N	0.92 (3)
C6–O1–H1O	113 (2)	C1–N1–H1N	111.0 (16)
N1–C1–C2–O2	–61.4 (3)	C2–C3–C4–C5	49.8 (3)
C6–C1–C2–C3	58.1 (3)	C3–C4–C5–C6	–51.1 (3)
C1–C2–C3–C4	–53.1 (3)	C8–O3–C3–H3	36

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1–H1N \cdots O4 ⁱ	0.92 (3)	2.35 (3)	3.258 (3)	171 (2)
O1–H1O \cdots N1 ⁱⁱ	0.83 (3)	2.02 (3)	2.819 (3)	162 (3)
C10–H10A \cdots O3 ⁱⁱⁱ	0.98	2.59	3.496 (3)	154

Symmetry codes: (i) $-x + \frac{1}{2}, -y + 2, z - \frac{1}{2}$; (ii) $x + \frac{1}{2}, -y + \frac{3}{2}, -z + 1$; (iii) $x + 1, y, z$.

In the absence of significant anomalous scattering effects, Friedel pairs were merged. All carbon-bound H atoms were constrained to their expected geometries (methyl C–H = 0.98 Å and methine C–H = 1.00 Å). Amino and hydroxyl H atoms were freely refined, yielding N–H = 0.92 (3)–0.94 (3) Å and O–H = 0.83 (3) Å. All methyl H atoms were free to rotate, except those on C7; the latter were disordered over two sites with refined occupancies of 0.79 (3) and 0.21 (3). $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{parent atom})$, where $x = 1.5$ for methyl and $x = 1.2$ for other H atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT* and *SADABS* (Sheldrick, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick,

1997); molecular graphics: *ORTEP-3* in *WinGX* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

The authors thank Professor Ward T. Robinson and Dr J. Wikaira of the University of Canterbury for their assistance.

References

- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
- Anderson, J. E., Angyal, S. J. & Craig, D. C. (1995). *Carbohydr. Res.* **272**, 141–148.
- Bernstein, J., Davis, R. E., Shimon, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.
- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Gainsford, G. J., Lensink, C. & Falshaw, A. (2006). *Acta Cryst.* **C62**, o650–o652.
- Lampe, D., Liu, C., Mahon, M. F. & Potter, B. V. L. (1996). *J. Chem. Soc. Perkin Trans. 1*, pp. 1717–1727.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
- Siemens (1996). *SMART* and *SAINT*. Versions 4.0. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Solomons, K. R. H., Freeman, S., Schwalbe, C. H., Shears, S. B., Nelson, D. J., Xie, W., Bruzik, K. S. & Kaetzel, M. A. (1998). *Carbohydr. Res.* **309**, 337–343.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Wewers, W., Gillandt, H. & Traub, H. S. (2005). *Tetrahedron Asymmetry*, **16**, 1723–1728.