

1D-1,2,5,6-Tetra-O-methyl-3,4-di-O-phosphinoyl-*chiro*-inositol 2.5-hydrateGraeme J. Gainsford,* Cornelis
Lensink and Andrew FalshawIndustrial 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
 $T = 168\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.018\text{ \AA}$
 R factor = 0.066
 wR factor = 0.186
Data-to-parameter ratio = 10.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.In the title compound, $\text{C}_{34}\text{H}_{38}\text{O}_8\text{P}_2 \cdot 2.5\text{H}_2\text{O}$, intermolecular interactions consist of strong $\text{O}-\text{H}(\text{water}) \cdots \text{O}$ hydrogen bonds, as well as $\text{C}-\text{H} \cdots \text{O}$ interactions.

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Comment

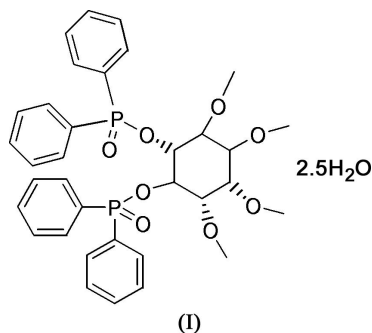
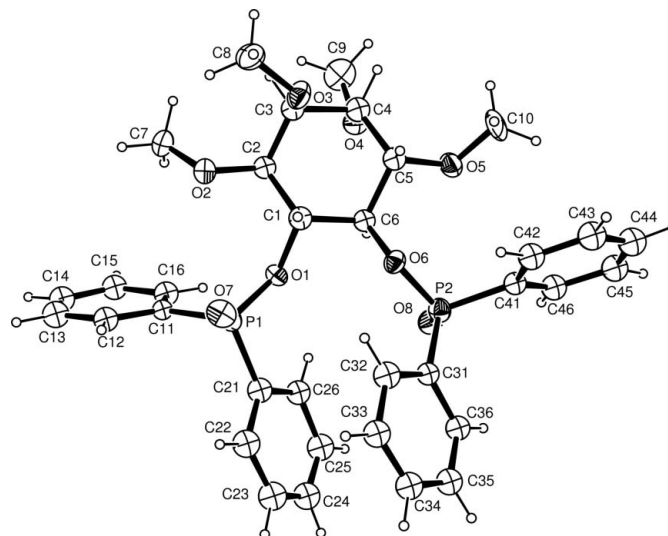
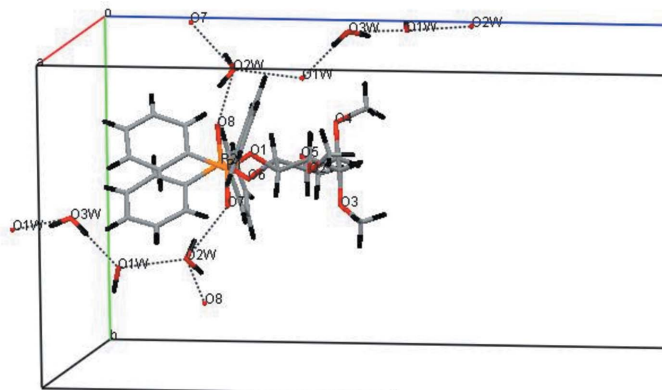
The title compound, (I), was prepared in research aimed at generating new hydrogenation catalyst ligands from inositols (Falshaw *et al.*, 1999, 2006; Gainsford *et al.*, 2000).The asymmetric unit contains the independent molecule shown in Fig. 1 and three water molecules, one of which (O3W) is on a twofold rotation axis. Based on four units per cell, the composition is $2\text{C}_{34}\text{H}_{38}\text{O}_8\text{P}_2 \cdot 5\text{H}_2\text{O}$; one of the water H atoms was not located (or refined). The absolute configuration was known from the synthesis and is confirmed here by the anomalous dispersion effects.

Figure 1
Molecular structure of (I), with displacement ellipsoids drawn at the 20% probability level. Water molecules have been omitted.


Figure 2

Mercury (Bruno *et al.*, 2002) plot of part of the unit cell showing the strong intermolecular hydrogen bonding (dotted lines) between the water molecules and diphenylphosphinate atoms O7 and O8. Colours black, grey, orange and red correspond to H, C, P and O atoms, respectively. Only O and P atoms are labelled (see Table 2 and text).

The inositol ring adopts the chair form, with Cremer & Pople (1975) puckering parameters $Q = 0.569$ (12) Å, $\theta = 3.3$ (12)° and $\varphi = 91$ (17)°. There are few related diphenylphosphinate structures reported [CSD (Version 5.27; Allen, 2002) refcodes CESJIT (Mazhar-ul-Haque *et al.*, 1984), HANBUU (Grice *et al.*, 2004), HOFLOD (Falshaw *et al.*, 1999) and VANQUW (Francio *et al.*, 1998)]. No significant deviations in geometry are found from the ranges reported.

Crystal packing is dominated by linking hydrogen bonds utilizing the water molecules *via* two O—H(water)···O(inositol), two O—H(water)···O(water), two P=O···H(water) and weaker C—H···O types (most are shown in Table 2). Fig. 2 illustrates how the chains of water molecules link the organic molecules running approximately along the *c* axis. Note that the (expected) interaction O2W···O1W [2.774 (10) Å] is shown in Fig. 2 but not in Table 2 as the H atom on O1W was not located.

Experimental

Crystals of (I) settled out of a solution of 1*D*-3,4-bis(*o*-diphenylphosphino)-1,2,5,6-tetra-*O*-methyl-*chiro*-inositol (Falshaw *et al.*, 2006) in CDCl₃ after exposure to air for several days.

Crystal data

C₃₄H₃₈O₈P₂·2.5H₂O
 $M_r = 680.62$
 Tetragonal, $P4_12_12$
 $a = 13.115$ (5) Å
 $c = 39.441$ (15) Å
 $V = 6784$ (4) Å³
 $Z = 8$

$D_x = 1.333$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.19$ mm⁻¹
 $T = 168$ (2) K
 Plate, colourless
 0.40 × 0.37 × 0.19 mm

Data collection

Siemens SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Blessing, 1995; Sheldrick, 1996)
 $T_{\min} = 0.629$, $T_{\max} = 0.965$

22268 measured reflections
 4694 independent reflections
 1666 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.245$
 $\theta_{\text{max}} = 26.1^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.066$
 $wR(F^2) = 0.186$
 $S = 0.85$
 4694 reflections
 437 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0828P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.36$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.31$ e Å⁻³
 Extinction correction: SHELXL97
 Extinction coefficient: 0.061 (3)
 Absolute structure: Flack (1983),
 783 Friedel pairs
 Flack parameter: 0.0 (2)

Table 1

Selected geometric parameters (Å, °).

P1—O7	1.478 (6)	P2—O6	1.576 (6)
P1—O1	1.577 (6)	P2—C31	1.753 (10)
P2—O8	1.485 (6)	P2—C41	1.769 (10)
O7—P1—O1	116.4 (4)	O7—P1—C21	113.9 (5)
O7—P1—C11	112.4 (5)	C1—O1—P1	125.3 (6)
O1—P1—C11	105.1 (5)	C6—O6—P2	121.5 (6)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O1W—H1W1···O5 ⁱ	0.87 (6)	2.29 (6)	3.123 (8)	160 (11)
O1W—H1W1···O4 ⁱ	0.87 (6)	2.55 (12)	3.136 (9)	126 (9)
O2W—H2W1···O7	0.87 (7)	1.95 (9)	2.732 (10)	149 (9)
O2W—H2W2···O8 ⁱ	0.90 (8)	1.91 (8)	2.678 (10)	142 (7)
O3W—H3W···O1W ⁱⁱ	0.91 (12)	1.92 (12)	2.791 (10)	160 (12)
C23—H23···O3W ⁱⁱⁱ	0.95	2.52	3.385 (19)	152
C44—H44···O2W ^{iv}	0.95	2.47	3.406 (15)	168

Symmetry codes: (i) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{4}$; (ii) $-y + \frac{1}{2}, x - \frac{1}{2}, z + \frac{1}{4}$; (iii) $y + \frac{1}{2}, -x + \frac{1}{2}, z - \frac{1}{4}$; (iv) $x - 1, y, z$.

H atoms on C atoms were constrained to their expected geometries with C—H = 0.95–1.0 Å. Only one of the two H atoms on water atom O1W could be located in difference maps; the water H atoms on O2W and O1W were restrained to O—H = 0.85 (3) Å and H···H = 1.3 (1) Å. For all H atoms, $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$. Examination of the full sphere of collected data showed that a section of data taken late in the collection was badly measured, probably from crystal shifting (from consideration of equivalent reflections and very high statistical errors). These data were the major contributors to the high value for R_{int} .

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97 and PLATON (Spek, 2003).

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References

Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.

- Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.
- Bruno, I. J., Cole, J. C., Edgington, P. R., Kessler, M., Macrae, C. F., McCabe, P., Pearson, J. & Taylor, R. (2002). *Acta Cryst.* **B58**, 389–397.
- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- Falshaw, A., Gainsford, G. J. & Lensink, C. (1999). *Acta Cryst.* **C55**, 1353–1355.
- Falshaw, A., Gainsford, G. J., Lensink, C., Slade, A. T. & Wright, L. J. (2006). *Polyhedron*. Submitted.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Francio, G., Arena, C. G., Panzalorto, M., Bruno, G. & Faraone, F. (1998). *Inorg. Chim. Acta*, **277**, 119–126.
- Gainsford, G. J., Lensink, C., Hart, J. B. & Falshaw, A. (2000). *Acta Cryst.* **C56**, 1396–1398.
- Grice, I. D., Jenkins, I. D., Busfield, W. K., Byriel, K. A. & Kennard, C. H. L. (2004). *Acta Cryst.* **E60**, o2384–o2385.
- Ul-Haque, M., Ahmed, J. & Horne, W. (1984). *J. Crystallogr. Spectrosc. Res.* **14**, 169–177.
- 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.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.