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myo-Inositol 1,3,5-Bicyclic Phosphate

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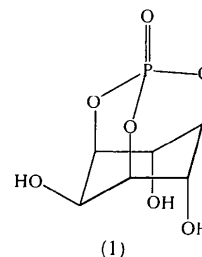
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Abstract

In the crystal structure of the title compound (4,6,10-trihydroxy-2,8,9-trioxo-1-phosphatricyclo[3.3.1.1^{3,7}]decane *P*-oxide, C₆H₉O₇P), the cyclohexane ring is in a chair conformation. Two hydroxy substituents are in axial orientations and the third is equatorial. There is an intramolecular hydrogen bond involving the two axial hydroxy groups.

Comment

A number of phosphate esters of *D*-*myo*-inositol are of considerable importance inasmuch as they display profound biological activity in the cell as second messengers (Berridge, 1987). Our studies directed towards the synthesis of these phosphate esters (Gaffney & Reese, 1997) led us to the preparation of the title compound, (1), which has an adamantane-like structure of particular interest. The preparation of compound (1) has also been reported by Chinese workers (Yuan & Zhai, 1992). This is the first reported crystal structure of a cyclic phosphate of inositol; the structures of three other non-cyclic phosphates are available in the literature (Spiers *et al.*, 1995).



The cyclohexane ring adopts the expected chair conformation, with the 2-, 4- and 6-hydroxy substituents being axial, equatorial and axial, respectively. There is an intramolecular hydrogen bond between the O6 atom as donor and O5 as acceptor (Table 2). All three hydroxy groups, as well as the phosphate oxygen atom O1, participate in intermolecular hydrogen bonding in the crystal lattice. Atom O5 participates in an asymmetric bifurcated hydrogen bond with acceptors O6 and O7. The H atom donated from O5 is evidently closer to O7, as judged by the angles involved.

The bicyclic phosphate/cyclohexane moiety has approximate threefold symmetry. The three P—O single bonds, of average length 1.580 Å, are all equivalent within significant error, as are the O—P—O bond angles (average 104.5°). The P—O1 bond is of pure double-bond character (Allen *et al.*, 1987).

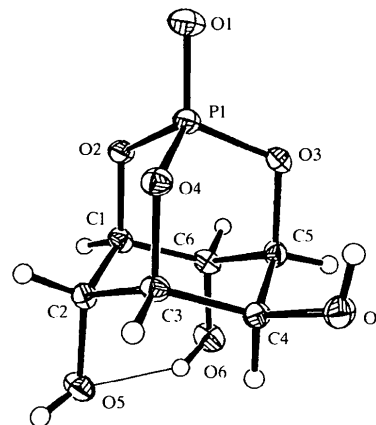


Fig. 1. View of the title structure. Displacement ellipsoids are shown at the 50% probability level. H atoms have been drawn as small circles of arbitrary radii.

Experimental

The title compound was crystallized by slow evaporation from a methanol/2-propanol solution.

Crystal data

C₆H₉O₇P
M_r = 224.10

Mo K α radiation
 λ = 0.7107 Å

Monoclinic

 $P2_1/c$ $a = 9.2892(6) \text{ \AA}$ $b = 9.0724(5) \text{ \AA}$ $c = 9.5328(12) \text{ \AA}$ $\beta = 96.150(6)^\circ$ $V = 798.76(12) \text{ \AA}^3$ $Z = 4$ $D_x = 1.864 \text{ Mg m}^{-3}$ D_m not measured

Data collection

Enraf–Nonius FAST area-detector diffractometer

 ω scans

Absorption correction: none

3136 measured reflections

1194 independent reflections

Refinement

Refinement on F^2 $R[F^2 > 2\sigma(F^2)] = 0.039$ $wR(F^2) = 0.098$ $S = 0.576$

1194 reflections

163 parameters

H atoms freely refined with

 U_{iso}

Cell parameters from 250 reflections

 $\theta = 2-22^\circ$ $\mu = 0.357 \text{ mm}^{-1}$ $T = 293(2) \text{ K}$

Square plate

 $0.30 \times 0.20 \times 0.07 \text{ mm}$

Colourless

989 reflections with

 $I > 2\sigma(I)$ $R_{int} = 0.081$ $\theta_{max} = 24.88^\circ$ $h = -10 \rightarrow 9$ $k = -10 \rightarrow 10$ $l = -10 \rightarrow 10$ $w = 1/[\sigma^2(F_o^2) + (0.1222P)^2 + 0.4645P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{max} < 0.001$ $\Delta\rho_{max} = 0.308 \text{ e \AA}^{-3}$ $\Delta\rho_{min} = -0.493 \text{ e \AA}^{-3}$

Extinction correction: none

Scattering factors from

International Tables for Crystallography (Vol. C)Table 1. Selected geometric parameters (\AA , $^\circ$)

P1—O1	1.448 (2)	P1—O3	1.581 (2)
P1—O4	1.574 (2)	P1—O2	1.584 (2)
O1—P1—O4	113.74 (9)	O1—P1—O2	113.28 (9)
O1—P1—O3	115.09 (9)	O4—P1—O2	104.45 (9)
O4—P1—O3	104.68 (8)	O3—P1—O2	104.50 (8)

Table 2. Hydrogen-bonding geometry (\AA , $^\circ$)

$D-H \cdots A$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O6—HO6 \cdots O5	2.07 (2)	2.779 (3)	148.3 (12)
O5—HO5 \cdots O6 ⁱ	2.70 (3)	2.909 (3)	97.3 (13)
O5—HO5 \cdots O7 ⁱⁱ	2.02 (3)	2.797 (3)	167.5 (13)
O7—HO7 \cdots O1 ⁱⁱⁱ	2.07 (3)	2.760 (3)	162.5 (14)

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

Data collection: *MADNES* (Pflugrath & Messerschmidt, 1990). Cell refinement: *MADNES*. Data reduction: *MADNES*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEX* (McArdle, 1993). Software used to prepare material for publication: *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1164). Services for accessing these data are described at the back of the journal.

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