

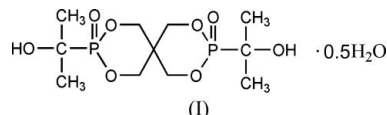
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Key indicators

Single-crystal X-ray study
 $T = 296$ K
Mean $\sigma(\text{C}-\text{C}) = 0.008$ Å
Some non-H atoms missing
 R factor = 0.069
 wR factor = 0.191
Data-to-parameter ratio = 16.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.3,9-Bis(2-hydroxy-2-propyl)-2,4,8,10-
tetraoxa-3,9-diphosphaspiro[5.5]undecane
3,9-dioxide hemihydrateIn the crystal structure of the title compound, $\text{C}_{11}\text{H}_{22}\text{O}_8\text{P}_2 \cdot 0.5\text{H}_2\text{O}$, the asymmetric unit contains two spiro[5.5]undecane molecules and one water molecule. The structure is stabilized by intra- and intermolecular $\text{C}-\text{H} \cdots \text{O}$ and $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds.Received 26 May 2006
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Comment

A pentaerythritol diphosphonate compound is capable of being used as a fire retardant agent and as a plasticizer (Tanabe *et al.*, 2005). The title compound, (I), has particular utility for flame retardant materials, which provide phosphorus as a component to reduce flammability (Levchik & Weil, 2005). We report here the crystal structure of (I).The asymmetric unit of (I) contains two spiro[5.5]undecane molecules and one water molecule (Fig. 1). The bond lengths and angles are within normal ranges (Allen *et al.*, 1987).The rings *A* (P1/O3/O4/C4–C6), *B* (P2/O5/O6/C6–C8), *C* (P3/O11/O12/C15–C17) and *D* (P4/O13/O14/C17–C19) have chair conformations with puckering parameters of $Q_T = 0.6168$ (6) Å, $\theta = 61.76$ (5)°, $\varphi = -64.80$ (8)° for ring *A*, $Q_T = 1.2407$ (25) Å, $\theta = 175.58$ (5)°, $\varphi = 17.55$ (55)° for ring *B*, $Q_T = 0.6142$ (9) Å, $\theta = 112.61$ (8)°, $\varphi = 115.65$ (13)° for ring *C*, and $Q_T = 2.4984$ (9) Å, $\theta = 89.89$ (2)°, $\varphi = -90.35$ (2)° for ring *D* (Cremer & Pople, 1975).The crystal structure is stabilized by intra- and intermolecular $\text{C}-\text{H} \cdots \text{O}$ and $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds (Table 1).

Experimental

The title compound was prepared in a one-step reaction of pentaerythritol dichlorophosphite (50.0 g, 189 mmol) (Hechenbleikner & Enlow, 1983) with formic acid (22.0 g, 478 mmol) and acetone (80 ml), heated with stirring at 300–323 K for 2 h (Brium, 1978). The reaction mixture was filtered and the resulting solid was washed with acetonitrile and acetone. Recrystallization from a mixture of glacial acetic acid/acetone (1:5) afforded a white powder. Crystals were obtained by dissolving the diphosphonate (0.4 g) in ethyl acetate (10 ml) and evaporating it slowly at room temperature for about 7 d.

Crystal data

 $\text{C}_{11}\text{H}_{22}\text{O}_8\text{P}_2 \cdot 0.5\text{H}_2\text{O}$
 $M_r = 353.24$
Triclinic, $P\bar{1}$
 $a = 7.5500$ (15) Å
 $b = 10.539$ (2) Å
 $c = 21.068$ (4) Å
 $\alpha = 86.28$ (3)°
 $\beta = 82.40$ (3)°
 $\gamma = 77.11$ (3)° $V = 1618.7$ (6) Å³
 $Z = 4$
 $D_x = 1.449$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 0.31$ mm⁻¹
 $T = 296$ (2) K
Block, colorless
 $0.40 \times 0.20 \times 0.20$ mm

Data collection

Enraf–Nonius CAD-4
diffractometer
 $\omega/2\theta$ scans
Absorption correction: ψ scan
(North *et al.*, 1968)
 $T_{\min} = 0.929$, $T_{\max} = 0.941$
6846 measured reflections

6337 independent reflections
3535 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.040$
 $\theta_{\max} = 26.0^\circ$
3 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.069$
 $wR(F^2) = 0.191$
 $S = 1.01$
6337 reflections
388 parameters
H atoms treated by a mixture of
independent and constrained
refinement

$w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 2P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.44 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.42 \text{ e } \text{\AA}^{-3}$

Table 1

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$OW-HWA\cdots O15^i$	0.79 (6)	2.06 (7)	2.823 (7)	163 (6)
$O1-H1A\cdots O15$	0.82	2.00	2.818 (5)	175
$OW-HWB\cdots O7^{ii}$	0.82 (6)	2.34 (6)	2.706 (8)	108 (5)
$O8-H8A\cdots O2^{iii}$	0.82	1.97	2.785 (5)	175
$O9-H9A\cdots OW$	0.82	2.27	2.599 (8)	105
$O16-H16A\cdots O10^{iv}$	0.82	1.90	2.716 (5)	179
$C5-H5A\cdots O8^v$	0.97	2.44	3.405 (7)	173
$C5-H5B\cdots O1$	0.97	2.51	3.106 (7)	120
$C7-H7C\cdots O2^{vi}$	0.97	2.47	3.332 (7)	148
$C8-H8B\cdots O8$	0.97	2.59	3.155 (7)	117
$C16-H16B\cdots O9$	0.97	2.50	3.056 (7)	117

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

The H atoms of water molecules were located in difference syntheses and refined [$O_{\text{water}}-H = 0.791(19)-0.826(19) \text{ \AA}$; $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(O)$]. The remaining H atoms were positioned geometrically, with $O-H = 0.82 \text{ \AA}$ and $C-H = 0.96$ and 0.97 \AA for methyl and methylene H, respectively, and constrained to ride on their parent

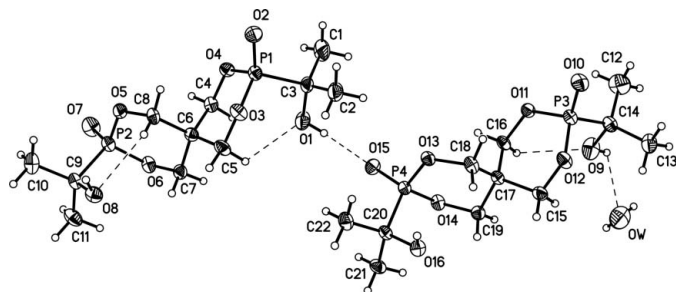


Figure 1

The asymmetric unit of the title molecule, with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines.

atoms with $U_{\text{iso}}(H) = xU_{\text{eq}}(C,O)$, where $x = 1.2$ for methylene H and $x = 1.5$ for all other H.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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