

Fig. 1. CHEMGRAF (Davies, 1981) drawing of the asymmetric unit content showing the numbering scheme. (a) Molecule 1. (b) Molecule 2.

1987b). The synthesis and chemistry of the title compound will be reported elsewhere (Bodalski *et al.*, 1987b).

We thank Professor R. Bodalski, Łódź, for the gift of crystals, Drs C. K. Prout and D. J. Watkin, Oxford, for providing us with facilities and for useful discussions. TJB wishes to thank Oriel College, University of Oxford, and the Ministry of National Education, Warsaw (project RP-II-10), for financial support.

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3a,4,5-Trihydroxy-5,7,7-trimethylperhydro-4-phosphaindene 4-Oxide

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Abstract. C₁₁H₂₁O₄P.2H₂O, *M_r* = 284.28, monoclinic, *P*2₁/*n*, *a* = 6.818 (4), *b* = 12.956 (3), *c* = 32.854 (7) Å, β = 91.01 (3)°, *V* = 2901.81 Å³, *Z* = 8, *D_x* = 1.301 g cm⁻³, λ(Cu Kα) = 1.54178 Å, μ = 18.4 cm⁻¹, *F*(000) = 1232, *T* = 293 K, *R_F* = 0.045, *wR_F* = 0.049 for 3421 observed reflections [*F_o* ≥

3σ(*F_o*)] and 327 parameters. The asymmetric unit consists of two independent molecules existing in almost identical conformations. Molecule 1: The six-membered phosphorinane ring exists in a distorted chair conformation [the asymmetry parameters are Δ*C_s*^{P(1)} = 5.66 (6), Δ*C_s*^{C(1)} = 6.37 (6), Δ*C₂*^{C(1),C(2)} = 11.26 (6)°]. The five-membered cyclopentane ring is an envelope with C(4) at the flap [Δ*C_s*^{C(4)} = 10.07 (6)°]. Molecule 2: The six-membered phosphorinane ring is a distorted chair [Δ*C_s*^{P(2)} = 3.25 (6), Δ*C_s*^{C(21)} = 7.10 (6), Δ*C₂*^{C(22)} = 12.71 (6)°]. The five-

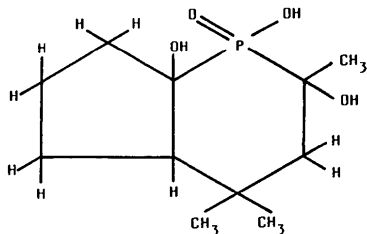
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membered cyclopentane ring is an envelope with C(24) at the flap [$\Delta C_s^{C(24)} = 7.10(6)^\circ$]. The rings in both molecules are *trans*-fused and the hydroxyl groups O(4) and O(24) are *trans* to the phosphoryl groups. There are a large number of intra- and intermolecular hydrogen bonds in the structure formed between water molecules, phosphoryl and hydroxyl groups.

Experimental. Colourless soft crystals of (I) from water/acetonitrile (1/1), $0.06 \times 0.20 \times 0.35$ mm; CAD-4 diffractometer with graphite monochromator, ω scans; lattice parameters from the least-squares refinement of the setting angles of 20 reflections in the θ range $10\text{--}35^\circ$. Intensity data were collected by the $\omega\text{--}2\theta$ scan method with an ω -scan width of $(1.80 + 0.14 \tan \theta)^\circ$ and a variable scan speed of $0.9\text{--}5.5^\circ \text{ min}^{-1}$ using graphite-monochromatized Cu $K\alpha$ radiation with indices $h - 1$ to 8, $k - 1$ to 15 and $l - 38$ to 38 in the θ range $0\text{--}65^\circ$. The intensities of three standard reflections showed no evidence of crystal decay. Intensities of 4908 unique reflections were measured of which 3518 had $I \geq 3\sigma(I)$, and were used in the structure solution and refinement. Lorentz-polarization corrections and ψ -scan-based empirical absorption corrections were applied.



The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1985). Refinement of the structure was by full-matrix least-squares calculations. At an intermediate stage in the refinement, a difference map revealed two O atoms of water and all H atoms. The latter were included in the subsequent cycles of refinement at fixed C—H distances of 0.95 \AA but only H atoms belonging to the hydroxyl groups and to the water molecules were allowed to refine. The non-H atoms were refined anisotropically and an overall isotropic temperature factor was allowed for the H atoms bonded to C atoms. Refinement converged with $R = 0.045$ and $wR = 0.049$. Maximum shift/e.s.d. was less than 0.1; final weighting scheme (Carruthers & Watkin, 1979), $w = A_0 T_0(x) + A_1 T_1(x) + A_2 T_2(x)$ where T_i is the i th Chebyshev series, $x = F_o/F_o(\text{max})$; minimum and maximum electron density in the final difference map $ca -0.3$ and 0.3 e \AA^{-3} ; scattering factors from Cromer & Mann (1968). Anomalous-dispersion parameters from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^3$)

Molecule 1	x	y	z	U_{iso}^*
P(1)	4588 (1)	1650.9 (7)	1731.3 (3)	266
O(1)	3708 (4)	1379 (2)	2127.5 (8)	374
O(2)	5792 (4)	2683 (2)	1741.7 (8)	364
O(3)	8101 (3)	783 (2)	1757.9 (8)	346
O(4)	1588 (4)	939 (2)	1310.6 (8)	349
C(1)	6260 (5)	653 (3)	1540 (1)	287
C(2)	6747 (5)	861 (3)	1099 (1)	363
C(3)	5123 (6)	1060 (3)	772 (1)	378
C(4)	3817 (6)	1960 (3)	917 (1)	349
C(5)	2130 (7)	2345 (4)	639 (1)	528
C(6)	981 (7)	3076 (4)	908 (1)	538
C(7)	1563 (7)	2821 (3)	1350 (1)	450
C(8)	2800 (5)	1844 (3)	1325 (1)	302
C(9)	5418 (6)	-416 (3)	1623 (1)	392
C(10)	3986 (7)	76 (4)	657 (1)	506
C(11)	6175 (7)	1423 (5)	388 (1)	584
Molecule 2				
P(2)	9432 (1)	6249.1 (7)	1634.6 (3)	275
O(21)	8525 (4)	6506 (2)	2029.4 (8)	380
O(22)	10613 (4)	5214 (2)	1643.6 (8)	364
O(23)	12971 (4)	7085 (2)	1677.2 (8)	357
O(24)	6468 (4)	7004 (2)	1219.9 (8)	347
C(21)	11142 (5)	7243 (3)	1454 (1)	295
C(22)	11647 (5)	7051 (3)	1012 (1)	357
C(23)	10027 (6)	6908 (3)	681 (1)	386
C(24)	8685 (6)	6015 (3)	809 (1)	366
C(25)	6965 (7)	5694 (4)	526 (1)	529
C(26)	5852 (7)	4909 (4)	778 (2)	555
C(27)	6399 (7)	5107 (3)	1228 (1)	464
C(28)	7670 (5)	6089 (3)	1220 (1)	299
C(29)	10354 (6)	8315 (3)	1546 (1)	394
C(30)	8945 (7)	7915 (4)	579 (1)	510
C(31)	11098 (7)	6570 (5)	295 (1)	573
O(100)	1702 (4)	2861 (2)	2540.6 (8)	406
O(101)	7287 (4)	7946 (2)	2572.8 (8)	372
O(102)	2458 (4)	-81 (2)	2663.8 (8)	390
O(103)	8447 (4)	10037 (2)	2540.1 (8)	419

* U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

tallography (1974, Vol. IV). All calculations carried out on the University of Oxford Chemical Crystallography Laboratory VAX 11/750 computer using *SHELXS86* (Sheldrick, 1985), *CRYSTALS* (Watkin, Carruthers & Betteridge, 1985) and *CHEMGRAF* (Davies, 1981), and on an Amstrad PC1512SD microcomputer in Łódź using the *CSU* program (Vićković, 1988).

The final fractional coordinates are given in Table 1* and bond distances and angles for non-H atoms are in Table 2. The molecular structure with the crystallographic numbering scheme is depicted in Fig. 1.

Related literature. The title compound was synthesized by Bodalski, Krawczyk & Majewski (1987a). The synthesis and chemistry of the title compound will be reported elsewhere (Bodalski, Krawczyk & Majewski, 1987b). The crystal and molecular struc-

* Lists of anisotropic thermal parameters, H-atom positions, bond distances and angles involving H atoms, torsion angles and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53915 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths (Å) and angles (°)

Molecule 1		Molecule 2	
P(1)—O(1)	1.485 (3)	P(2)—O(21)	1.485 (3)
P(1)—O(2)	1.569 (3)	P(2)—O(22)	1.564 (3)
P(1)—C(1)	1.842 (4)	P(2)—C(21)	1.842 (4)
P(1)—C(8)	1.810 (4)	P(2)—C(28)	1.813 (4)
O(3)—C(1)	1.444 (4)	O(23)—C(21)	1.449 (4)
O(4)—C(8)	1.434 (4)	O(24)—C(28)	1.441 (4)
C(1)—C(2)	1.515 (5)	C(21)—C(22)	1.520 (5)
C(1)—C(9)	1.526 (5)	C(21)—C(29)	1.521 (5)
C(2)—C(3)	1.550 (5)	C(22)—C(23)	1.548 (5)
C(3)—C(4)	1.547 (6)	C(23)—C(24)	1.538 (6)
C(3)—C(10)	1.535 (6)	C(23)—C(30)	1.533 (6)
C(3)—C(11)	1.536 (6)	C(23)—C(31)	1.538 (6)
C(4)—C(5)	1.540 (5)	C(24)—C(25)	1.540 (5)
C(4)—C(8)	1.526 (5)	C(24)—C(28)	1.531 (5)
C(5)—C(6)	1.523 (7)	C(25)—C(26)	1.523 (7)
C(6)—C(7)	1.534 (6)	C(26)—C(27)	1.539 (6)
C(7)—C(8)	1.524 (5)	C(27)—C(28)	1.540 (5)
O(2)—P(1)—O(1)	113.7 (2)	O(22)—P(2)—O(21)	113.5 (2)
C(1)—P(1)—O(1)	113.2 (2)	C(21)—P(2)—O(21)	113.5 (2)
C(1)—P(1)—O(2)	106.2 (2)	C(21)—P(2)—O(22)	106.1 (2)
C(8)—P(1)—O(1)	113.7 (2)	C(28)—P(2)—O(21)	113.7 (2)
C(8)—P(1)—O(2)	104.1 (2)	C(28)—P(2)—O(22)	104.5 (2)
C(8)—P(1)—C(1)	105.0 (2)	C(28)—P(2)—C(21)	104.7 (2)
O(3)—C(1)—P(1)	106.7 (2)	O(23)—C(21)—P(2)	106.6 (2)
C(2)—C(1)—P(1)	110.4 (3)	C(22)—C(21)—P(2)	110.4 (3)
C(2)—C(1)—O(3)	104.5 (3)	C(22)—C(21)—O(23)	104.6 (3)
C(9)—C(1)—P(1)	109.8 (2)	C(29)—C(21)—P(2)	110.3 (3)
C(9)—C(1)—O(3)	110.1 (3)	C(29)—C(21)—O(23)	109.4 (3)
C(9)—C(1)—C(2)	115.0 (3)	C(29)—C(21)—C(22)	115.2 (3)
C(3)—C(2)—C(1)	121.7 (3)	C(23)—C(22)—C(21)	121.4 (3)
C(4)—C(3)—C(2)	108.7 (3)	C(24)—C(23)—C(22)	108.6 (3)
C(10)—C(3)—C(2)	112.6 (4)	C(30)—C(23)—C(22)	112.7 (4)
C(10)—C(3)—C(4)	114.2 (3)	C(30)—C(23)—C(24)	114.4 (3)
C(11)—C(3)—C(2)	106.4 (3)	C(31)—C(23)—C(22)	105.7 (3)
C(11)—C(3)—C(4)	107.5 (4)	C(31)—C(23)—C(24)	107.7 (4)
C(11)—C(3)—C(10)	107.1 (4)	C(31)—C(23)—C(30)	107.2 (4)
C(5)—C(4)—C(3)	119.3 (3)	C(25)—C(24)—C(23)	119.3 (4)
C(8)—C(4)—C(3)	117.9 (3)	C(28)—C(24)—C(23)	118.3 (3)
C(8)—C(4)—C(5)	101.9 (3)	C(28)—C(24)—C(25)	101.4 (3)
C(6)—C(5)—C(4)	104.1 (4)	C(26)—C(25)—C(24)	103.5 (4)
C(7)—C(6)—C(5)	106.8 (4)	C(27)—C(26)—C(25)	107.3 (4)
C(8)—C(7)—C(6)	105.1 (4)	C(28)—C(27)—C(26)	104.3 (4)
O(4)—C(8)—P(1)	106.9 (2)	O(24)—C(28)—P(2)	106.0 (2)
C(4)—C(8)—P(1)	110.5 (3)	C(24)—C(28)—P(2)	111.5 (2)
C(4)—C(8)—O(4)	108.8 (3)	C(24)—C(28)—O(24)	108.5 (3)
C(7)—C(8)—P(1)	116.1 (3)	C(27)—C(28)—P(2)	116.3 (3)
C(7)—C(8)—O(4)	111.2 (3)	C(27)—C(28)—O(24)	111.1 (3)
C(7)—C(8)—C(4)	103.1 (3)	C(27)—C(28)—C(24)	103.2 (3)

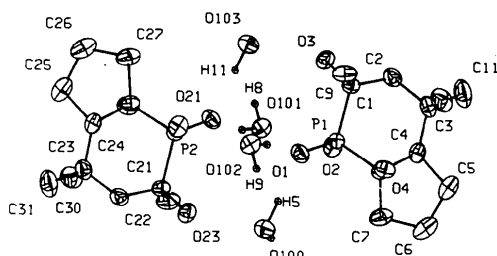


Fig. 1. CHEMGRAF (Davies, 1981) drawing of the asymmetric unit content showing the numbering scheme.

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ture of 1,2,7 α -trihydroxy-2-methylperhydro-1-phosphaindene 1-oxide has been reported previously (Bartczak & Yagbasan, 1991).

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Structure of the Triclinic Modification of 17 β -Hydroxy-19-nor-7 α -methyl-17 α -pregn-5(10)-en-20-yn-3-one (ORG OD14)

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Abstract. (I) C₂₁H₂₈O₂, $M_r = 312.45$, triclinic, $P1$, $a = 6.542$ (2), $b = 6.6773$ (8), $c = 10.287$ (2) Å, $\alpha = 87.05$ (1), $\beta = 80.09$ (2), $\gamma = 79.17$ (2)°, $V = 434.7$ (2) Å³, $Z = 1$, $D_x = 1.194$ g cm⁻³, $\lambda(\text{Cu } K\alpha) =$

1.54184 Å, $\mu = 5.5$ cm⁻¹, $F(000) = 170$, room temperature, $R = 0.066$ for 1495 reflections with $I \geq 2.5\sigma(I)$. The A ring has a 2 α ,3 β half-chair conformation, the B ring a 7 α ,8 β half-chair conformation.