diastereomer. The phenyl ring defined by C(1), C(2), C(3), C(4), C(5), C(6) is planar within 0·003 Å; phenyl ring C(11), C(12), C(13), C(14), C(15), C(16) is planar within 0·02 Å; the imidazole ring defined by C(18), C(19), C(20), N(2), N(3) is planar within 0·003 Å.

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Structure of 6-(Iodomethyl)-2-oxo-2-phenoxy-1,2-oxaphosphorinane

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Abstract. $C_{11}H_{14}IO_3P$, $M_r=352\cdot11$, monoclinic, $P2_1/n$, $a=8\cdot300\,(3)$, $b=14\cdot081\,(2)$, $c=11\cdot326\,(4)$ Å, $\beta=104\cdot32\,(3)°$, $V=1282\cdot6\,(13)$ Å^3, $Z=4$, $D_x=1\cdot823$ Mg m^{-3}, $\lambda(\text{Mo }K\alpha)=0\cdot71073$ Å, $\mu=25\cdot8$ cm^{-1}, $F(000)=688$, $R=0\cdot035$ for 2364 observed reflections. The six-membered ring O(1), P(2), C(3), C(4), C(5), C(6) is in a chair conformation with phenoxy and iodomethyl groups adopting axial and equatorial orientations, respectively. The bond distances and angles are unexceptional.

Introduction. The phosphonate ester 6-(aminomethyl)-2-oxo-2-phenoxy-1,2-oxaphosphorinane was synthesized to resemble a product-like intermediate for the cyclization of phenyl 6-acetamido-5-hydroxyhexanoate. The phosphonate ester was used as a hapten to generate a series of monoclonal antibodies that were then screened for their ability to act as abzyme catalysts for the cyclization reaction (Napper, Benkovic, Tramontano & Lerner, 1987).

The synthesis of the phosphonate ester was accomplished by a stereospecific cyclization of phenyl isopropyl-4-pentenylphosphonate induced by iodine (Zhao & Yan, 1985) followed by conversion of the iodomethyl derivative to the desired compound. Definitive evidence for a 1,3-*trans* orientation (axial–equatorial) of the phenoxy and aminomethyl substituents was sought by obtaining the X-ray crystal structure of the precursor iodo compound. The results of that study are presented herein.

Experimental. A large crystal of the title compound, (1), from a hot acetone solution by slow cooling was cut to an approximate size 0·54 × 0·70 × 0·70 mm and was used for data collection.

Accurate cell constants and a crystal orientation matrix were determined on an Enraf–Nonius CAD-4 diffractometer by a least-squares refinement of the setting angles of 25 reflections with θ in the range 10–15°. Intensity data were collected by the $\omega/2\theta$-scan method and variable scan speed using monochromatized radiation in the range $2<\theta<27°$. The intensities of three reflections, chosen as standards, were monitored at 2 h exposure intervals and decreased in a linear fashion by 2·4% over the course of data collection; this decay was corrected for by appropriate scaling. Intensities of 2788 independent reflections (h 0→10, k 0→17, l −14→14) were measured, of which 2364 had $I>3\sigma(I)$ and were used in the structure solution and refinement. The data were corrected for Lorentz–polarization effects and for empirical absorption (North, Phillips & Mathews, 1968); minimum and maximum correction factors were 0·854 and 1·000, respectively.

The structure was solved by the heavy-atom method and refined by full-matrix least-squares calculations on F's, initially with isotropic and finally with anisotropic temperature factors for the non-H atoms. At an intermediate stage in the refinement, a difference map revealed all H atoms which were included in the subsequent cycles in geometrically idealized positions

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(C—H 0·95 Å) with an overall isotropic thermal parameter $B_{\rm iso} = 5{\cdot}0$ Å^2. Refinement converged with $R = 0{\cdot}035$ and $wR = 0{\cdot}075$; (Δ/σ) in the last cycle $<0{\cdot}05$ and $S = 2{\cdot}216$. In the refinement cycles, weights were derived from the counting statistics, $w = 1/[\sigma^2(F) + 0{\cdot}060F^2]$. Scattering factors were those of Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965) and allowance was made for anomalous dispersion (Cromer & Liberman, 1970). A difference map calculated at the conclusion of the refinement showed electron density (0·50 e Å^{-3}) close to the I atom with no other chemically significant features; the minimum peak was −0·37 e Å^{-3}. The computer programs used in this study were from the Enraf–Nonius *Structure Determination Package* (B. A. Frenz & Associates, Inc., 1985) and *ORTEP* (Johnson, 1976).

Discussion. Final fractional coordinates and equivalent isotropic thermal parameters with e.s.d.'s for (1) are given in Table 1.* Table 2 contains molecular dimensions. Fig. 1 is a perspective drawing of the molecular structure of (1) with our numbering scheme. The six-membered ring O(1), P(2), C(3), C(4), C(5), C(6) has a chair conformation with iodomethyl and phenoxy groups adopting equatorial and axial orientations, respectively. A CONNECTIVITY search through the Cambridge Structural Database (1987) yielded only one structure, (2*R*)-6-(1′,2′-dihydroxyethyl)-3,4,5-trihydroxy-1′,2′;4,5-di-isopropylidene-2-methoxy-D-*glycero*-D-*talo*-1,2-oxaphosphorinane-2-one-3-*p*-toluenesulfonate (2) (Appel & Milker, 1977), which contains the 1,2-oxaphosphorinane moiety. An asymmetric unit of (2) contains two independent molecules with several substituents present on the 1,2-oxaphosphorinane ring; therefore, the molecular dimensions of the two structures, *i.e.* (1) and (2) cannot be compared with each other.

The P(2)—O(3) bond in (1) is certainly a double bond with a bond distance of 1·458 (2) Å. The endocyclic P—O distance of 1·573 (2) Å is significantly shorter than the exocyclic P—O distance of 1·606 (2) Å. The P—C bond length in (1), 1·774 (3) Å, is significantly shorter than the corresponding lengths in (2) [1·828 (6) and 1·815 (7) Å], a difference which seems to be a consequence of the substituents present in (2). The remaining molecular dimensions in (1) are unexceptional with bond lengths: I—C 2·137 (3), C_{sp^3}—O 1·456 (4), C_{sp^2}—O 1·397 (3), C_{sp^3}—C_{sp^3} average 1·510 (4) and aromatic C—C average 1·371 (4) Å. The P atom has tetrahedral geometry with the angles

Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$$B_{\rm eq} = \tfrac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	$B_{\rm eq}$
I	0·74665 (5)	0·44299 (2)	1·05693 (3)	6·379 (9)
P(2)	0·72202 (9)	0·11036 (6)	0·97161 (8)	3·00 (1)
O(1)	0·7030 (3)	0·2064 (2)	1·0378 (2)	3·06 (4)
O(2)	0·8730 (3)	0·1275 (2)	0·9086 (2)	3·70 (5)
O(3)	0·5663 (3)	0·0838 (2)	0·8863 (3)	4·34 (6)
C(3)	0·8094 (4)	0·0288 (3)	1·0895 (3)	3·64 (7)
C(4)	0·9408 (4)	0·0746 (3)	1·1914 (4)	3·90 (7)
C(5)	0·8801 (5)	0·1645 (3)	1·2376 (3)	4·12 (8)
C(6)	0·8351 (4)	0·2405 (3)	1·1388 (3)	3·31 (6)
C(7)	0·7730 (5)	0·3289 (3)	1·1847 (3)	4·04 (7)
C(21)	0·8520 (4)	0·1833 (3)	0·8039 (3)	3·71 (7)
C(22)	0·7923 (5)	0·1423 (3)	0·6933 (4)	4·44 (8)
C(23)	0·7853 (6)	0·1954 (4)	0·5889 (4)	5·5 (1)
C(24)	0·8332 (5)	0·2885 (4)	0·5979 (4)	5·60 (9)
C(25)	0·8886 (5)	0·3281 (3)	0·7095 (5)	5·4 (1)
C(26)	0·8996 (5)	0·2778 (3)	0·8152 (4)	4·48 (8)

Table 2. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles* (°)

I—C(7)	2·137 (3)	C(5)—C(6)	1·526 (3)
P(2)—O(1)	1·573 (2)	C(6)—C(7)	1·489 (4)
P(2)—O(2)	1·606 (2)	C(21)—C(22)	1·358 (4)
P(2)—O(3)	1·458 (2)	C(21)—C(26)	1·385 (4)
P(2)—C(3)	1·774 (3)	C(22)—C(23)	1·388 (4)
O(1)—C(6)	1·456 (3)	C(23)—C(24)	1·366 (6)
O(2)—C(21)	1·397 (3)	C(24)—C(25)	1·353 (5)
C(3)—C(4)	1·522 (4)	C(25)—C(26)	1·374 (4)
C(4)—C(5)	1·504 (4)		

O(1)—P(2)—O(2)	105·3 (1)	O(1)—C(6)—C(7)	106·9 (2)
O(1)—P(2)—O(3)	111·0 (1)	C(5)—C(6)—C(7)	111·9 (2)
O(1)—P(2)—C(3)	105·6 (1)	I—C(7)—C(6)	112·2 (2)
O(2)—P(2)—O(3)	113·9 (1)	O(2)—C(21)—C(22)	119·0 (3)
O(2)—P(2)—C(3)	102·3 (1)	O(2)—C(21)—C(26)	119·3 (3)
O(3)—P(2)—C(3)	117·6 (1)	C(22)—C(21)—C(26)	121·6 (3)
P(2)—O(1)—C(6)	120·8 (1)	C(21)—C(22)—C(23)	119·1 (3)
P(2)—O(2)—C(21)	120·9 (1)	C(22)—C(23)—C(24)	120·2 (3)
P(2)—C(3)—C(4)	112·3 (2)	C(23)—C(24)—C(25)	119·4 (3)
C(3)—C(4)—C(5)	112·4 (2)	C(24)—C(25)—C(26)	122·4 (3)
C(4)—C(5)—C(6)	112·2 (2)	C(21)—C(26)—C(25)	117·3 (3)
O(1)—C(6)—C(5)	110·2 (2)		

C(3)—P(2)—O(1)—C(6)	43·2 (1)	C(4)—C(5)—C(6)—O(1)	60·4 (3)
O(3)—P(2)—O(2)—C(21)	47·4 (1)	O(1)—C(6)—C(7)—I	−68·5 (2)
O(1)—P(2)—C(3)—C(4)	−39·3 (2)	C(26)—C(21)—C(22)—C(23)	2·6 (4)
P(2)—O(1)—C(6)—C(5)	−54·8 (2)	C(22)—C(21)—C(26)—C(25)	−1·8 (4)
P(2)—O(2)—C(21)—C(22)	−86·2 (3)	C(21)—C(22)—C(23)—C(24)	−2·0 (4)
P(2)—O(2)—C(21)—C(26)	96·8 (2)	C(22)—C(23)—C(24)—C(25)	0·5 (4)
P(2)—C(3)—C(4)—C(5)	51·0 (2)	C(23)—C(24)—C(25)—C(26)	0·4 (4)
C(3)—C(4)—C(5)—C(6)	−61·9 (3)	C(24)—C(25)—C(26)—C(21)	0·2 (4)

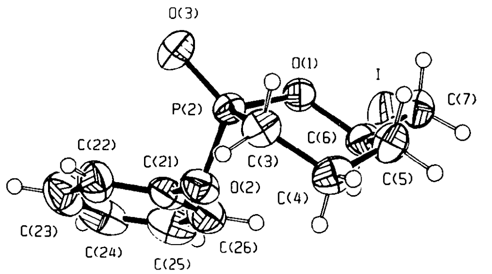

Fig. 1. An *ORTEP* (Johnson, 1976) drawing of (1) with crystallographic numbering scheme.

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* Tables of structure factors, anisotropic temperature factors and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44874 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

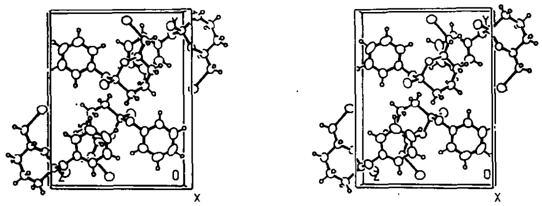

Fig. 2. A stereoview of the unit cell showing molecular packing.

around P(2) in the range 102·3 (1)–117·6 (1)°. Moreover, the endocyclic angles at C atoms in the 1,2-oxaphosphorinane ring are slightly enlarged from the tetrahedral values [range 110·2–112·4 (2)°] as is the angle I—C(7)—C(6) [112·1 (2)°]. The phenyl ring is essentially planar with maximum deviation of any atom from the least-squares plane being 0·01 Å.

The crystal structure (Fig. 2) consists of discrete molecules separated by normal van der Waals distances.

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Structure of Ethanolammonium *cyclo*-Tetraphosphate

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Abstract. $4C_2H_8NO^+.P_4O_{12}^{4-}$, $M_r = 564·25$, tetragonal, $I\bar{4}$, $a = 10·746$ (8), $c = 10·071$ (8) Å, $V = 1163$ (3) Å^3, $Z = 2$, $D_x = 1·611$ Mg m^{-3}, $\lambda(\mathrm{Ag}\,K\bar{\alpha}) = 0·5608$ Å, $\mu = 0·219$ mm^{-1}, $F(000) = 592$, $T = 293$ K, final $R = 0·034$ for 1112 independent observed reflexions. The P_4O_{12} ring anions have $\bar{4}$ symmetry; they alternate with the $[OH—(CH_2)_2—NH_3]^+$ groups in planes $z \sim 0·25$ and 0·75. A three-dimensional network of strong hydrogen bonds connects the hydroxyl and the NH_3 groups to the external O atoms of the ring anion.

Introduction. The present work is part of a systematic investigation of condensed phosphates of amines, amino acids and aminoalcohols. In this practically unexplored domain we have previously described tris(methylammonium) *cyclo*-triphosphate (Averbuch-Pouchot, Durif & Guitel, 1988*a*), glycine *cyclo*-tri- and tetraphosphate (Averbuch-Pouchot, Durif & Guitel, 1988*b,c*) and calcium ethylenediammonium *cyclo*-tetraphosphate monohydrate (Averbuch-Pouchot, Durif & Guitel, 1988*d*). The title compound is the first evidence for the existence of condensed phosphates of aminoalcohols.

Experimental. The title compound has been prepared by adding slowly a stoichiometric amount of P_4O_{10} to an aqueous solution of ethanolamine kept at a temperature close to 273 K. The reaction is

$$P_4O_{10} + 2H_2O \rightarrow H_4P_4O_{12}$$

$$H_4P_4O_{12} + 4NH_2—(CH_2)_2—OH \rightarrow P_4O_{12}[NH_3—(CH_2)_2—OH]_4.$$

The resulting solution is then kept at room temperature until crystallization occurs. Many careful recrystallizations are then necessary to obtain good quality crystals. They appear as multifaceted short stout tetragonal prisms. The compound is stable at room temperature.

Density not measured. Prism fragment 0·35 × 0·35 × 0·40 mm. Philips PW 1100 diffractometer, graphite monochromator. Systematic absences: hkl, $h+k+l = 2n$. 19 reflexions ($10·2 < \theta < 13·3°$) for refining unit-cell dimensions. ω scan, 1494 reflexions measured ($3 < \theta < 30°$), h,k,l, $h_{\max} = k_{\max} = 16$, $l_{\max} = 17$. Scan width: 1·20°, scan speed: 0·03° s^{-1}, total background measuring time: 6 s. Two intensity and orientation reference reflexions (112 and $\bar{1}\bar{1}\bar{2}$) measured every 2 h, no variation. Lorentz and polarization

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