Refinement

Refinement on F	$w = 1/[\sigma^2(F) + 0.008F^2]$
Final $R = 0.051$	$(\Delta/\sigma)_{max} = 0.026$
$wR = 0.067$	$\Delta\rho_{max} = 0.16$ e Å^{-3}
$S = 1.52$	$\Delta\rho_{min} = -0.20$ e Å^{-3}
2789 reflections	Atomic scattering factors
385 parameters	from *International Tables*
All H-atom parameters re- fined	*for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$B_{eq} = \tfrac{4}{3}\Sigma_i\Sigma_j B_{ij} \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
O(1)	0.1191 (2)	0.7973 (3)	0.00320 (3)	3.65
O(2)	0.0826 (2)	0.4245 (3)	0.03029 (3)	3.64
C(1)	0.1444 (2)	0.6478 (4)	0.02559 (4)	3.13
C(2)	0.2510 (3)	0.7824 (5)	0.04530 (4)	3.70
C(3)	0.3193 (2)	0.5856 (4)	0.06672 (4)	3.54
C(4)	0.4078 (2)	0.7535 (4)	0.08820 (4)	3.47
C(5)	0.4865 (3)	0.5683 (5)	0.10940 (4)	3.69
C(6)	0.5707 (2)	0.7428 (5)	0.13103 (4)	3.61
C(7)	0.6521 (3)	0.5628 (5)	0.15237 (4)	3.77
C(8)	0.7356 (2)	0.7399 (5)	0.17382 (4)	3.66
C(9)	0.8175 (3)	0.5618 (5)	0.19522 (4)	3.88
C(10)	0.9012 (3)	0.7391 (5)	0.21665 (4)	3.76
C(11)	0.9837 (3)	0.5591 (5)	0.23792 (4)	4.03
C(12)	1.0693 (3)	0.7350 (5)	0.25909 (4)	4.09
C(13)	1.1618 (3)	0.5516 (5)	0.27798 (4)	4.06
C(14)	1.1719 (3)	0.5543 (5)	0.30481 (4)	4.23
C(15)	1.0942 (3)	0.7413 (5)	0.32366 (4)	4.44
C(16)	1.0213 (3)	0.5700 (5)	0.34495 (5)	4.60
C(17)	0.9557 (3)	0.7565 (5)	0.36622 (4)	4.39
C(18)	0.8856 (3)	0.5860 (5)	0.38793 (5)	4.74
C(19)	0.8198 (3)	0.7724 (5)	0.40912 (5)	4.65
C(20)	0.7521 (3)	0.6045 (6)	0.43126 (5)	5.05
C(21)	0.6847 (3)	0.7931 (6)	0.45203 (5)	5.53
C(22)	0.6150 (4)	0.6246 (9)	0.47396 (7)	7.1

Table 2. *Selected geometric parameters* (Å, °)

C(1)—O(1)	1.317 (2)	C(12)—C(13)	1.507 (3)
C(1)—C(2)	1.497 (3)	C(13)—C(14)	1.322 (3)
C(1)—O(2)	1.227 (2)	C(14)—C(15)	1.504 (3)
C(2)—C(1)—O(1)	123.1 (2)	C(12)—C(13)—C(14)	128.1 (2)
O(1)—C(1)—O(2)	123.1 (2)	C(13)—C(14)—C(15)	128.2 (2)
C(10)—C(11)—C(12)	113.8 (2)	C(14)—C(15)—C(16)	112.6 (2)
C(2)—C(1)—O(2)	113.8 (2)	C(15)—C(16)—C(17)	113.5 (2)
C(11)—C(12)—C(13)	112.5 (2)		

O(1)—C(1)—C(2)—C(3)	−161.1 (2)
O(2)—C(1)—C(2)—C(3)	21.5 (3)
C(1)—C(2)—C(3)—C(4)	−171.1 (2)
C(10)—C(11)—C(12)—C(13)	−173.8 (2)
C(11)—C(12)—C(13)—C(14)	−129.1 (2)
C(12)—C(13)—C(14)—C(15)	−0.1 (4)
C(13)—C(14)—C(15)—C(16)	127.5 (2)
C(14)—C(15)—C(16)—C(17)	173.6 (2)

Data collection: Rigaku AFC-5 with software *AFCM* from the Protein Research Center, Osaka University. Programs used to refine structure: *HBLS*-V and *FMLS* (Ashida, 1979). Programs used to plot structure: *ORTEP*II (Johnson, 1971). Software used to prepare material for publication: *POTP* (Yasuoka, Kimura & Mizuma, 1979) and *DAPH* (Ashida, 1979). The following two types of ω-scan width were used in order to avoid the overlapping of neighbouring reflections, with an ω-scan rate of 4.0° min^{-1} and background counts for 4 s on each side of every scan: $(1.6-0.17\tan\theta)°$ for reflections with $\theta \leq 100°$ and

$(1.6-0.4\tan\theta)°$ for reflections with $100 < \theta < 120°$. The molecular model was initially built for 13 C and two O atoms by reference to the structure of the γ phase of oleic acid. Refinement was by full-matrix least-squares methods.

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Structure of a Coupled Carbohydrate and Terpene†

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Abstract

In $(1S\text{-}\{1\alpha,1[3R^*,5R^*(R^*)],2\alpha,5\beta\})$-5-[1,2-bis(benzoyloxy)ethyl]-4,5-dihydro-3-[1-hydroxy-5-methyl-2-(1-methylethyl)cyclohexyl]-2(3H)-furanone, the tetrahydrofuran (THF) ring exhibits a shallow conformation with atom C3 (C-4 by IUPAC numbering)

† Part 2. Part 1: Abboud, Jiang & Enholm (1993).

occupying the flap position. The cyclohexyl ring, on the other hand, exhibits a chair conformation. Molecules of the title compound (I) are not involved in intermolecular hydrogen bonding, but each has an intramolecular hydrogen bond between the hydroxyl group and carbonyl atom O1 [O1···H5 = 2.17 (6), H5—O5 = 0.83 (6),   O1···O5 = 2.863 (5) Å   and O1···H5—O5 = 142 (6)°].

Comment

Compound (I) was first purified by flash chromatography over silica gel (Still, Khan & Mitra, 1978) to produce fine colorless needles. Next, these were collected and filtered, and dried and recrystallized with hexanes–dichloromethane (20:1). An additional recrystallization from methanol at 296 K over 3 days gave colorless rods. The crystals had an m.p. of 141–143 °C (uncorrected).

(I)
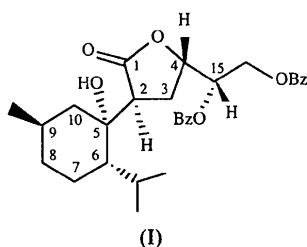
Although only the relative stereochemistry is indicated in Fig. 1, the actual chirality was assigned on the basis of asymmetric centers inherent to each of the two components coupled to make compound (I). The carbohydrate lactone, L-gulonic-γ-lactone, was utilized as a precursor to the THF ring and the dibenzoate appendage. In a similar manner, the terpene ketone (−)-menthone was the precursor to the cyclohexanol ring. The absolute stereochemistry of the molecule was established from the fixed C atoms C4 and C15 on the THF and its two-C-atom appendage, respectively (right side of Fig. 1), and C atoms C6 and C9 bearing the isopropyl and methyl groups on the cyclohexanol ring, respectively (left side of Fig. 1). The X-ray studies reported herein were needed to establish the stereochemistry of the single C2—C5 bond which connects the two ring moieties. The THF ring exhibits an envelope conformation with C3 occupying the flap position. The two bulky groups on C2 and C4 occupy *trans* positions with respect to the ring. O1 is within one e.s.d. (0.01) of the envelope plane (containing atoms C1, C2, C4 and O4), while C3 lies at a distance of 0.37 (1) Å above the plane. Puckering parameters (Cremer & Pople, 1975) of the THF ring are $Q(2) = 0.229$ Å and $\Phi(2) = 108.2°$. The cyclohexyl ring exhibits a chair conformation with atoms C6 and C9 at distances of 0.70 and 0.64 Å, respectively, above

and below the plane of C5, C7, C8 and C10 (mean e.s.d. = 0.006). Puckering parameters (Cremer & Pople, 1981) of the cyclohexyl ring are $Q(2) = 0.041$, $Q(3) = -0.567$ Å, $\Phi(2) = 252.0°$ and a puckering amplitude of $Q = 0.569$ Å, with $\theta = 175.8°$ and $\Phi = 252.0°$.

Simple methods of coupling achiral esters/lactones and ketones have been established (Enholm & Jiang, 1992a) which result in β-hydroxy esters. One of the authors has also studied several other examples of lactone carbohydrates such as δ-gluconolactone and γ-ribonolactone in couplings with the terpene ketone (−)-menthone (Enholm & Jiang, 1992b). Compound (I) can also contain a cyclohexanol unit derived from (+)-dihydrocarvone (Abboud, Jiang & Enholm, 1993).
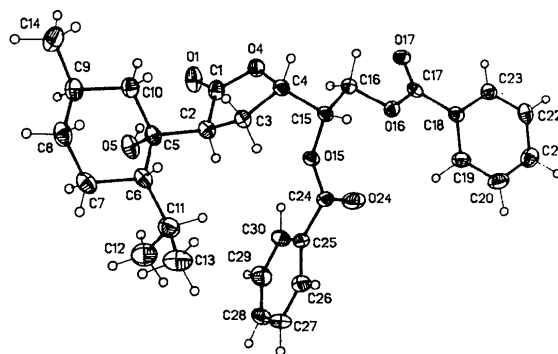
Fig. 1. Molecular structure of (I), with 50% probability ellipsoids, showing the atom-numbering scheme.

Experimental

Crystal data

$C_{30}H_{36}O_7$	Mo $K\alpha$ radiation
$M_r = 508.6$	$\lambda = 0.71073$ Å
Orthorhombic	Cell parameters from 50
$P2_12_12_1$	reflections
$a = 9.145$ (1) Å	$\theta = 10–11°$
$b = 16.756$ (2) Å	$\mu = 0.09$ mm^{-1}
$c = 18.201$ (2) Å	$T = 293$ K
$V = 2789.0$ (5) Å^3	Needles
$Z = 4$	0.50 × 0.38 × 0.23 mm
$D_x = 1.211$ Mg m^{-3}	Colorless

Data collection

Seimens $P3m/V$ diffractometer	$R_{int} = 0.021$
	$\theta_{max} = 27.5°$
ω scans	$h = 0 \rightarrow 11$
Absorption correction:	$k = 0 \rightarrow 21$
analytical	$l = 0 \rightarrow 23$
$T_{min} = 0.974$, $T_{max} = 0.983$	4 standard reflections
	monitored every 96
3438 measured reflections	reflections
3420 independent reflections	intensity variation: 1%
2279 observed reflections	
[$F > 6\sigma(F)$]	

Refinement

Refinement on F	All H-atom parameters re-
Final $R = 0.0527$	fined except those of C12
$wR = 0.0503$	$w = 1/[\sigma^2(F)+0.0004F]^2$
$S = 1.60$	$(\Delta/\sigma)_{max} = 0.001$
2279 reflections	$\Delta\rho_{max} = 0.19$ e Å^{-3}
466 parameters	$\Delta\rho_{min} = -0.22$ e Å^{-3}

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O1	0.8335 (4)	0.4949 (2)	0.6164 (2)	0.0833 (14)
O4	0.8642 (3)	0.3981 (2)	0.53583 (13)	0.0583 (11)
O5	0.6189 (4)	0.6096 (2)	0.5723 (2)	0.0742 (13)
O15	0.9711 (3)	0.46655 (14)	0.39259 (12)	0.0481 (9)
O16	1.1918 (3)	0.3463 (2)	0.38514 (13)	0.0530 (10)
O17	1.3476 (3)	0.2974 (2)	0.4686 (2)	0.0632 (11)
O24	0.9188 (5)	0.4460 (2)	0.27360 (14)	0.083 (2)
C1	0.8064 (5)	0.4675 (3)	0.5570 (2)	0.0576 (14)
C2	0.7098 (5)	0.5021 (3)	0.4979 (2)	0.0500 (14)
C3	0.6881 (5)	0.4318 (2)	0.4453 (2)	0.0485 (14)
C4	0.8169 (5)	0.3769 (3)	0.4615 (2)	0.0494 (14)
C5	0.5715 (5)	0.5436 (2)	0.5279 (2)	0.0545 (14)
C6	0.4768 (5)	0.5770 (3)	0.4655 (2)	0.0590 (14)
C7	0.3408 (6)	0.6174 (3)	0.4980 (3)	0.072 (2)
C8	0.2507 (6)	0.5601 (4)	0.5438 (3)	0.075 (2)
C9	0.3393 (5)	0.5234 (3)	0.6058 (3)	0.067 (2)
C10	0.4792 (5)	0.4870 (3)	0.5750 (3)	0.059 (2)
C11	0.5545 (7)	0.6279 (3)	0.4063 (3)	0.078 (2)
C12	0.5847 (9)	0.7127 (3)	0.4279 (3)	0.113 (3)
C13	0.4722 (11)	0.6238 (6)	0.3341 (4)	0.112 (4)
C14	0.2507 (8)	0.4612 (5)	0.6475 (4)	0.093 (3)
C15	0.9455 (5)	0.3834 (2)	0.4091 (2)	0.0463 (14)
C16	1.0806 (5)	0.3480 (3)	0.4419 (2)	0.0512 (14)
C17	1.3232 (5)	0.3186 (2)	0.4066 (2)	0.0467 (13)
C18	1.4314 (5)	0.3186 (2)	0.3462 (2)	0.0494 (13)
C19	1.4202 (6)	0.3706 (3)	0.2877 (2)	0.066 (2)
C20	1.5315 (7)	0.3720 (3)	0.2361 (3)	0.082 (2)
C21	1.6485 (7)	0.3221 (3)	0.2416 (3)	0.078 (2)
C22	1.6581 (6)	0.2690 (3)	0.2983 (3)	0.069 (2)
C23	1.5494 (6)	0.2679 (3)	0.3511 (3)	0.059 (2)
C24	0.9439 (5)	0.4911 (2)	0.3230 (2)	0.0518 (15)
C25	0.9474 (5)	0.5795 (2)	0.3163 (2)	0.0506 (14)
C26	0.8807 (7)	0.6133 (3)	0.2553 (3)	0.074 (2)
C27	0.8822 (8)	0.6946 (3)	0.2463 (3)	0.094 (3)
C28	0.9503 (8)	0.7426 (3)	0.2968 (4)	0.088 (2)
C29	1.0125 (7)	0.7101 (3)	0.3569 (4)	0.084 (2)
C30	1.0110 (5)	0.6283 (3)	0.3674 (2)	0.061 (2)

Table 2. *Geometric parameters* (Å, °)

C1—O1	1.200 (5)	C15—C4	1.518 (6)
C1—O4	1.334 (6)	C6—C5	1.534 (6)
C4—O4	1.464 (5)	C10—C5	1.532 (6)
C5—O5	1.437 (5)	C7—C6	1.535 (7)
C15—O15	1.444 (4)	C11—C6	1.546 (7)
C24—O15	1.355 (4)	C8—C7	1.515 (8)
C16—O16	1.449 (5)	C9—C8	1.519 (8)
C17—O16	1.346 (5)	C10—C9	1.525 (7)
C17—O17	1.204 (5)	C14—C9	1.522 (10)
C24—O24	1.196 (5)	C12—C11	1.501 (8)
C2—C1	1.509 (6)	C13—C11	1.516 (10)
C3—C2	1.531 (6)	C16—C15	1.495 (6)
C5—C2	1.542 (6)	C18—C17	1.479 (6)
C4—C3	1.524 (6)	C25—C24	1.487 (5)
C1—O4—C4	111.2 (3)	C11—C6—C5	117.2 (4)
C15—O15—C24	117.2 (3)	C8—C7—C6	111.9 (4)
C16—O16—C17	115.2 (3)	C9—C8—C7	112.1 (5)
C2—C1—O1	128.0 (4)	C10—C9—C14	110.9 (5)
C2—C1—O4	111.2 (3)	C10—C9—C8	109.6 (4)
O1—C1—O4	120.8 (4)	C14—C9—C8	111.3 (5)
C3—C2—C5	117.5 (4)	C5—C10—C9	114.9 (4)
C3—C2—C1	103.1 (3)	C12—C11—C13	111.1 (6)
C5—C2—C1	113.6 (3)	C12—C11—C6	115.1 (4)
C4—C3—C2	104.1 (3)	C13—C11—C6	110.5 (5)
C15—C4—O4	109.5 (3)	C16—C15—O15	109.4 (3)
C15—C4—C3	115.7 (3)	C16—C15—C4	111.2 (3)
O4—C4—C3	105.1 (3)	O15—C15—C4	109.0 (3)
C6—C5—C10	109.2 (4)	O16—C16—C15	107.7 (3)
C6—C5—O5	107.8 (3)	C18—C17—O16	112.5 (3)
C6—C5—C2	111.4 (3)	C18—C17—O17	124.9 (4)
C10—C5—O5	109.2 (3)	O16—C17—O17	122.6 (4)
C10—C5—C2	111.8 (3)	C25—C24—O15	112.0 (3)
O5—C5—C2	107.4 (3)	C25—C24—O24	124.8 (3)
C7—C6—C11	113.5 (4)	O15—C24—O24	123.1 (3)
C7—C6—C5	109.5 (4)		

Programs used: *SHELXTL-Plus* (Sheldrick, 1990) for cell refinement, data collection, data reduction, structure solution (direct methods) and molecular graphics; *SHELX76* (Sheldrick, 1976) for structure refinement (full-matrix least squares); *FUER* (S. B. Larson, personal communication) for preparation of computer-typed tables of atomic coordinates, bond lengths, bond angles and torsion angles. The ω-scan width was symmetrical over 1.2° about the $K\alpha_{1,2}$ maximum and the background was offset 1.0 and -1.0 in ω from the $K\alpha_{1,2}$ maximum. The scan speed was variable over 3-6° min^{-1} (depending on intensity). All H atoms, except those of C12, were located in a difference Fourier map. The H atoms were refined with isotropic thermal parameters; those bonded to C12 were fixed in idealized positions and their isotropic thermal parameters were fixed at 0.08 Å^2. The absolute configuration was chosen to correspond to the known configurations of the precursors used to synthesize the title compound. The linear absorption coefficient was calculated using values from *International Tables for X-ray Crystallography* (1974, Vol. 4, p. 55). Scattering factors for non-H atoms were taken from Cromer & Mann (1968) with anomalous-dispersion corrections from Cromer & Liberman (1970), while those of H atoms were from Stewart, Davidson & Simpson (1965).

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Structure Determination of a Monohydrated Piperazinium Hydrogenphosphate

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Abstract

Piperazinium hydrogenphosphate monohydrate, $(N_2C_4H_{12}).HPO_4.H_2O$, is composed of a network of isolated PO_4 tetrahedra and organic six-membered rings of piperazine. The two amino groups of the piperazine are protonated and the rings are stacked along the [010] direction. The stability of the framework is ensured by strong O—H linkages between the O atoms from the PO_4 tetrahedra and H atoms from the diamine and water molecules.

Comment

The $AlPO_4$-n family has been studied extensively over a few years. These compounds possess great similarities to the known zeolites whose frameworks are characterized by their large porosities. This property is fundamental for their activity in catalysis or as molecular sieves. One route to the synthesis of microporous structures uses an organic amine as a template. However, Tapp, Milestone & Bibby (1988) noted that this type of preparation sometimes leads to a simple amine phosphate at low temperatures. This paper describes the synthesis and X-ray structure determination of the piperazinium hydrogenphosphate $(N_2C_4H_{12}).HPO_4.H_2O$.

The title compound consists of a framework of isolated PO_4 tetrahedra interleaved with piperazine and H_2O molecules. The two amino groups of each piperazine molecule are protonated giving the formula $(N_2C_4H_{12})^{2+}$.

The organic rings thus constituted are stacked perpendicularly to the [010] direction at approximatively the $y = 0$ and $y = \frac{1}{2}$ levels, whereas the PO_4 tetrahedra and the H_2O molecules are at the $y = \frac{1}{4}$ and $y = \frac{3}{4}$ levels (Fig. 1). The P atoms are distributed at one site in a tetrahedral coordination with three P—O bond lengths around 1.52 Å and a fourth of 1.589 (3) Å. This difference has previously been observed in $(C_2H_{10}N_2).Na_2.(HPO_4)_2.6H_2O$ (Averbuch-Pouchot, Durif & Guitel, 1987) and $(C_2H_{10}N_2).HPO_4$ (Averbuch-Pouchot & Durif, 1987); in each case it is characteristic of the existence of an $(HPO_4)^{2-}$ hydrogenphosphate species. No residual electronic density is detected in the vicinity of the O(4) atom in $(N_2C_4H_{12}).HPO_4.H_2O$, but to satisfy at the electroneutrality of the compound it is reasonable to locate a hydroxyl group at this site. Each O atom [except O(4)] of the PO_4 tetrahedra makes two strong O—H linkages with distances ranging from 1.72 (4) to 1.92 (6) Å; this network of hydrogen bonds ensures the stability of the framework.
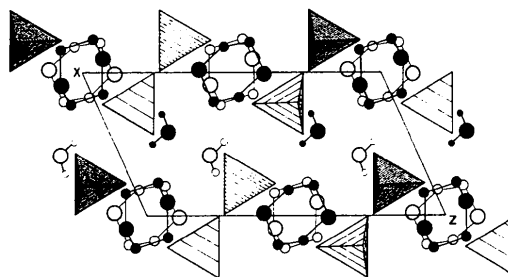

Fig. 1. Projection of $(N_2C_4H_{12}).HPO_4.H_2O$ along [010]; the piperazine rings and the H_2O molecules are drawn.

Experimental

Crystal data

$C_4H_{12}N_2^{2+}.HO_4P^{2-}.H_2O$	Mo $K\alpha$ radiation
$M_r = 202.16$	$\lambda = 0.71069$ Å
Monoclinic	Cell parameters from 34
$P2_1/c$	reflections
$a = 6.4176$ (9) Å	$\theta = 15$–$16°$
$b = 12.296$ (2) Å	$\mu = 0.294$ mm^{-1}
$c = 12.215$ (2) Å	$T = 293$ K
$\beta = 114.30$ (1)°	Plate
$V = 878.5$ (3) Å^3	$0.228 \times 0.171 \times 0.027$ mm
$Z = 4$	Colorless
$D_x = 1.529$ Mg m^{-3}	

Data collection

Stoe Siemens AED-2	$R_{int} = 0.0$
diffractometer	$\theta_{max} = 27.5°$
ω/θ scans	$h = -8 \rightarrow 8$
Absorption correction:	$k = 0 \rightarrow 15$
none	$l = 0 \rightarrow 15$
4232 measured reflections	3 standard reflections
4232 independent reflections	frequency: 60 min
891 observed reflections	intensity variation: 2.1%
$[I > 3.0\sigma(I)]$	