C(6)—C(7)	1.401 (9)	C(28)—C(29)	1.362 (8)
C(7)—C(8)	1.387 (8)	C(29)—C(30)	1.345 (8)
C(8)—C(9)	1.457 (8)	C(30)—C(31)	1.402 (8)
C(9)—C(10)	1.466 (8)	C(31)—C(32)	1.413 (9)
C(10)—C(11)	1.462 (8)	C(32)—C(33)	1.343 (9)
C(12)—C(13)	1.406 (8)	C(33)—C(34)	1.419 (8)
C(12)—C(17)	1.436 (8)		
C(17)—S(1)—S(2)	104.3 (2)	C(16)—C(17)—S(1)	127.3 (5)
C(18)—S(2)—S(1)	104.7 (2)	C(12)—C(17)—S(1)	115.0 (5)
C(11)—N(1)—C(12)	118.5 (5)	C(19)—C(18)—S(2)	123.7 (5)
C(24)—N(2)—C(23)	120.8 (6)	C(23)—C(18)—S(2)	114.4 (5)
O(1)—C(1)—C(10)	119.6 (6)	N(2)—C(23)—C(22)	128.4 (6)
O(1)—C(1)—C(2)	119.1 (6)	N(2)—C(23)—C(18)	116.9 (6)
N(1)—C(11)—C(10)	119.8 (5)	N(2)—C(24)—C(25)	121.2 (6)
C(13)—C(12)—N(1)	128.2 (6)	O(2)—C(34)—C(25)	122.3 (6)
N(1)—C(12)—C(17)	113.5 (6)	O(2)—C(34)—C(33)	118.1 (6)

The structure was solved by direct phase determination. The parameters of the complete structure were refined by full-matrix least squares. All phenyl rings were refined without constraint. Values of distances and angles in the rings show no significant differences from those of an ideal benzene ring. All H-atom positions were calculated using a riding model and were included with fixed isotropic U_{iso} in all refinements.

Data collection: *Enraf–Nonius SDP-Plus Structure Determination Package* (Frenz, 1985). Cell refinement: *Enraf–Nonius SDP-Plus Structure Determination Package*. Data reduction: *Enraf–Nonius SDP-Plus Structure Determination Package*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *PLATON* (Spek, 1992). Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1033). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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7-Hydroxyflavanone (I) and 7-Ethoxycarbonylmethoxyflavanone (II)

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Abstract

The γ-pyrone rings in both of the title molecules, 2,3-dihydro-7-hydroxy-2-phenyl-4*H*-1-benzopyran-4-one, $C_{15}H_{12}O_3$, (I), and ethyl (2,3-dihydro-2-phenyl-4-oxo-4*H*-1-benzopyran-7-yl)oxyacetate, $C_{19}H_{18}O_5$, (II), adopt a C(2)-sofa conformation. In (I), the phenyl ring is twisted 103.0 (1)° from the plane of the fused rings and the hydroxy group at C(7) forms an intermolecular hydrogen bond to O(4) of the adjacent molecule. In (II), the phenyl ring is twisted 121.5 (2)° from the plane of the fused rings and the acetyl group is oriented slightly out of the plane of the benzene ring.

Comment

As a result of pharmacological interest in the flavanoids, a number of crystal structures have been resolved. The most studied are the flavones. In contrast, only a few single-crystal X-ray studies of flavanones have been reported so far (Cantrell, Stalzer & Becker, 1974; Mariezcurrena, 1978; Tomlin & Cantrell, 1990). The spasmolytic activity of a series of flavanone ether derivatives, including (I) and (II), was investigated using efloxate (Ertan, 1981*a*) as the reference compound. According to the results obtained, most of the compounds have an antagonistic activity comparable with that of the reference compound against the agonistic acetylcholine, histamine and $BaCl_2$ contractions, but no advantage over efloxate was found. Only compound (I) showed more potent activity than efloxate against histamine contractions (Ertan, 1981*b*). The determination of the structures of (I) and (II) was undertaken in order to study the conformations of the molecules and to establish any structure–activity relationship.

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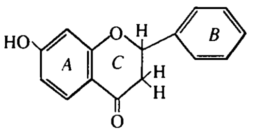
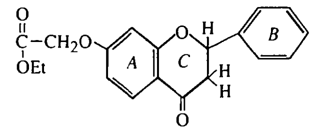


(I)



(II)

In (I) and (II), the pyrone ring is puckered as a result of the C(2)—C(3) single bond. The puckering causes the γ-pyrone ring to be distorted into a C(2)-sofa conformation. The plane through C(3), C(2) and O(1) makes an angle with the least-squares plane through O(1), C(9), C(10) and C(4) of 47.9 (2)° in (I) and 146.4 (5)° in (II). C(2) deviates from the pyrone-ring plane by −0.36 (1) and −0.30 (1) Å in (I) and (II), respectively. The dihedral angle between the *A* and *C* rings is 1.7 (1)° in (I) and 5.0 (2)° and (II). The phenyl ring is twisted out of the plane of the benzo-γ-pyrone ring, the dihedral angle between these planes being 103.0 (1)° in (I) and 121.5 (2)° in (II). The corresponding angle is 5.3 (7)° in 5-hydroxyflavone (Shoja, 1990) and 6.7 and 11° in two flavanoids isolated from *Physalis minima* (Koh & Ng, 1993). In other flavanone derivatives, the phenyl ring is also rotated as much as in (I) and (II). In 5,7,4′-trimethoxyflavanone, the reported dihedral angle is 70.8° (Mariezcurrena, 1978) and in 3-chloroflavanone the C(3)—C(2)—C(20)—C(21) torsion angle is 74.3 (4)°; the corresponding torsion angles are −133.2 (2) and −99.8 (7)° in compounds (I) and (II), respectively. The hydroxy group in compound (I) is almost planar with a C(5)—C(6)—C(7)—O(7) torsion angle of −177.9 (3)°.

In compound (II), the acetyl group is oriented slightly out of the plane of the benzene ring with a torsion angle of −177.3 (5)° for C(11)—O(7)—C(7)—C(6). The methylenoxy C(11) atom is displaced from the C(7)—O(7)—C(11)—C(12) plane by 0.051 (6) Å. The ester methyl group is nearly perpendicular to this plane with an O(12*B*)—C(13)—C(14) bond angle of 110.1 (6)°.

The bond lengths and angles in (I) and (II) are similar and within the expected ranges. The C(2)—O(1) and C(9)—O(1) bond lengths in (II) [1.389 (6) and 1.367 (5) Å, respectively] are very similar to those reported for similar flavanoid structures (Cantrell & Stalzer, 1982). C(2)—C(3) in ring *C* is a single bond with bond lengths of 1.502 (5) and 1.442 (9) Å in (I) and (II), respectively. This bond is 1.511 (5) Å in 3-chloroflavanone, 1.496 (11) Å in 5,7,4′-trimethoxyflavanone and 1.514 (7) Å in 4′-bromoflavanone. In flavones, C(2)—C(3) is a double bond with an average bond length of 1.34 Å (Kaneda, Iitaka & Shibata, 1973).

Compound (I) is stabilized by an intermolecular hydrogen bond between O(7) and O(4)$(x-1, -y+\frac{1}{2}, z+\frac{1}{2})$ with O(7)···O(4) 2.678 (4) [O(7)—H(7O) 0.93 (4), H(7O)···O(4) 1.75 (4) Å and O(7)—H(7O)···O(4) 179 (4)°]. There are no intermolecular hydrogen bonds in (II). All intermolecular contacts correspond to normal van der Waals interactions.

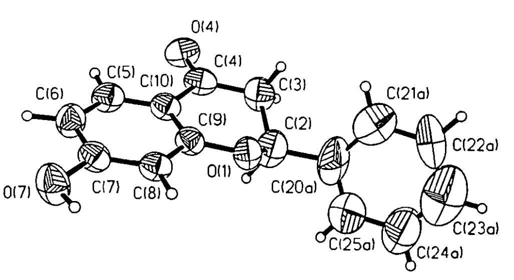


Fig. 1. A view of (I) showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 30% probability level for non-H atoms; H atoms are represented by spheres of arbitrary radii. The position of the disordered phenyl ring '*a*' is shown.

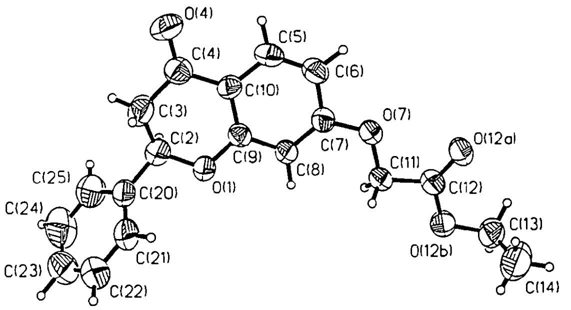


Fig. 2. A view of (II) showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 30% probability level for non-H atoms; H atoms are represented by spheres of arbitrary radii.

Experimental

Compound (I) was prepared by mixing 2′,4′-dihydroxychalcone (2,4-dihydroxyphenyl styryl ketone, 4 g, 0.017 mol), EtOH (20 ml), aqueous NaOH (60 ml, 1.5%) and sodium acetate (20 g, $CH_3COONa.3H_2O$). The mixture was heated for 2 h at 373 K, then left overnight at room temperature. Water (80 ml) was added, and the solution was acidified with acetic acid (20%). A precipitate started to form at pH 7 and the final pH was regulated to 5–6. The mixture was left in a refrigerator for 2 h, then the precipitate was filtered off, washed and dried. The crude product was decoloured and crystallized from boiling MeOH with aid of active carbon. Yield 48%, m.p. 457–460 K.

Compound (II) was prepared by the addition of anhydrous K_2CO_3 (0.276 g, 0.002 mol) to a mixture of 7-hydroxyflavanone (0.48 g, 0.002 mol) and ethyl α-bromoacetate (0.334 g, 0.002 mol) in acetone (30 ml, freshly distilled on P_2O_5). The mixture was stirred and refluxed for 8 h at 333–338 K using a magnetic stirrer. Anhydrous $CaCl_2$ was used to protect the mixture from moisture. The solvent was distilled off and water (30 ml) was added. The oily residue which separated was extracted three times with 50 ml ether. The combined ether extracts were extracted twice with aqueous NaOH (20 ml, 10%) and once with water, then dried over anhydrous Na_2SO_4 and concentrated. The precipitate formed on the addition of petroleum ether was crystallized from ether–ethanol. Yield 76.9%, m.p. 364–365 K.

Compound (I)

Crystal data

$C_{15}H_{12}O_3$
$M_r = 240.2$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Monoclinic
$P2_1/c$
$a = 5.333\ (2)$ Å
$b = 23.891\ (6)$ Å
$c = 9.876\ (2)$ Å
$\beta = 101.68\ (2)^\circ$
$V = 1232.3\ (6)$ Å^3
$Z = 4$
$D_x = 1.295$ Mg m^{-3}

Cell parameters from 25 reflections
$\theta = 5$–12.5°
$\mu = 0.090$ mm^{-1}
$T = 298$ K
Prismatic
0.50 × 0.44 × 0.30 mm
Colourless

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: ψ scans
$T_{\min} = 0.870$, $T_{\max} = 0.940$
3056 measured reflections
2779 independent reflections
1639 observed reflections $[F > 4\sigma(F)]$

$R_{\rm int} = 0.0124$
$\theta_{\max} = 27.5^\circ$
$h = 0 \rightarrow 6$
$k = 0 \rightarrow 31$
$l = -12 \rightarrow 12$
2 standard reflections monitored every 200 reflections
intensity decay: <1%

Refinement

Refinement on F
$R = 0.0601$
$wR = 0.0766$
$S = 2.07$
1639 reflections
213 parameters
$w = 1/[\sigma^2(F) + 0.0006F^2]$
$(\Delta/\sigma)_{\max} = 0.024$

$\Delta\rho_{\max} = 0.58$ e Å^{-3}
$\Delta\rho_{\min} = -0.22$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Compound (II)

Crystal data

$C_{19}H_{18}O_5$
$M_r = 326.3$
Monoclinic
$P2_1/c$
$a = 7.238\ (3)$ Å
$b = 7.822\ (3)$ Å
$c = 29.518\ (13)$ Å
$\beta = 90.48\ (4)^\circ$
$V = 1671.1\ (12)$ Å^3
$Z = 4$
$D_x = 1.297$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 5$–12.5°
$\mu = 0.094$ mm^{-1}
$T = 298$ K
Needle
1.00 × 0.12 × 0.10 mm
Colourless

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: refined from ΔF (*ABSORB*; Ugozzoli, 1987)
$T_{\min} = 0.943$, $T_{\max} = 1.000$
2444 measured reflections
2199 independent reflections

1375 observed reflections $[F > 4\sigma(F)]$
$R_{\rm int} = 0.012$
$\theta_{\max} = 22.5^\circ$
$h = -7 \rightarrow 7$
$k = 0 \rightarrow 8$
$l = 0 \rightarrow 31$
2 standard reflections monitored every 100 reflections
intensity decay: 3.3%

Refinement

Refinement on F
$R = 0.0528$
$wR = 0.0474$
$S = 1.06$
1375 reflections
266 parameters
Unit weights applied
$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.35$ e Å^{-3}
$\Delta\rho_{\min} = -0.28$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (I) *and* (II)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
(I)				
O(1)	0.2153 (4)	0.3387 (1)	0.1995 (2)	0.053 (1)
O(4)	0.6045 (4)	0.2682 (1)	−0.0747 (2)	0.060 (1)
O(7)	−0.1521 (4)	0.1646 (1)	0.2772 (3)	0.067 (1)
C(3)	0.5050 (7)	0.3501 (1)	0.0409 (3)	0.063 (1)
C(4)	0.4895 (5)	0.2888 (1)	0.0090 (3)	0.051 (1)
C(5)	0.2918 (5)	0.1989 (1)	0.0593 (3)	0.054 (1)
C(6)	0.1309 (6)	0.1686 (1)	0.1234 (3)	0.056 (1)
C(7)	−0.0015 (5)	0.1963 (1)	0.2125 (3)	0.049 (1)
C(8)	0.0267 (5)	0.2530 (1)	0.2346 (3)	0.047 (1)
C(9)	0.1928 (5)	0.2827 (1)	0.1708 (3)	0.044 (1)
C(10)	0.3275 (5)	0.2562 (1)	0.0813 (3)	0.046 (1)
C(2)	0.4536 (6)	0.3626 (1)	0.1818 (3)	0.731 (1)
C(20*a*)†	0.4437	0.4261	0.2119	0.101 (9)
C(21*a*)	0.3003	0.4660	0.1262	0.115 (5)
C(22*a*)	0.2981	0.5215	0.1699	0.122 (6)
C(23*a*)	0.4392	0.5371	0.2991	0.156 (10)
C(24*a*)	0.5826	0.4973	0.3847	0.135 (6)
C(25*a*)	0.5849	0.4418	0.3411	0.118 (5)
C(20*b*)	0.4471	0.4238	0.2129	0.054 (5)
C(21*b*)	0.2219	0.4551	0.1823	0.091 (4)
C(22*b*)	0.2281	0.5125	0.2087	0.109 (5)
C(23*b*)	0.4594	0.5386	0.2657	0.077 (4)
C(24*b*)	0.6846	0.5073	0.2963	0.116 (5)
C(25*b*)	0.6784	0.4499	0.2699	0.096 (4)
(II)				
O(1)	0.0618 (4)	0.8364 (4)	0.6043 (1)	0.060 (1)
O(4)	−0.4596 (5)	1.0285 (5)	0.5864 (1)	0.080 (2)
O(7)	0.0912 (4)	0.6765 (4)	0.4490 (1)	0.058 (1)
O(12*A*)	0.2536 (5)	0.5653 (5)	0.3751 (1)	0.079 (2)
O(12*B*)	0.5021 (5)	0.4816 (5)	0.4141 (1)	0.071 (1)
C(2)	−0.0470 (7)	0.8722 (10)	0.6416 (2)	0.095 (3)
C(3)	−0.1998 (7)	0.9883 (8)	0.6355 (2)	0.080 (2)
C(4)	−0.3055 (7)	0.9673 (7)	0.5919 (2)	0.058 (2)
C(5)	−0.2796 (7)	0.8589 (6)	0.5124 (2)	0.055 (2)
C(6)	−0.1808 (7)	0.7923 (7)	0.4776 (2)	0.057 (2)
C(7)	0.0005 (6)	0.7392 (6)	0.4861 (1)	0.050 (2)
C(8)	0.0797 (7)	0.7507 (6)	0.5286 (1)	0.051 (2)
C(9)	−0.0257 (6)	0.8224 (6)	0.5632 (1)	0.047 (2)
C(10)	−0.2051 (6)	0.8794 (6)	0.5559 (1)	0.048 (2)
C(11)	0.2730 (7)	0.6131 (7)	0.4551 (2)	0.052 (2)
C(12)	0.3379 (7)	0.5514 (6)	0.4098 (2)	0.054 (2)
C(13)	0.5869 (9)	0.4153 (10)	0.3731 (2)	0.078 (3)
C(14)	0.7160 (12)	0.5410 (13)	0.3547 (3)	0.112 (4)
C(20)	0.0718 (7)	0.8929 (7)	0.6832 (2)	0.064 (2)
C(21)	0.2379 (8)	0.9761 (8)	0.6811 (2)	0.074 (2)
C(22)	0.3464 (9)	0.9910 (10)	0.7198 (2)	0.100 (3)
C(23)	0.2867 (11)	0.9243 (12)	0.7594 (2)	0.106 (3)
C(24)	0.1236 (13)	0.8460 (11)	0.7618 (2)	0.106 (4)
C(25)	0.0145 (9)	0.8280 (9)	0.7240 (2)	0.089 (3)

† For atoms C(20*a*)–C(25*a*) and C(20*b*)–C(25*b*) the site occupancy factors are each 0.5 (disordered phenyl rings).

Table 2. *Selected geometric parameters* (Å, °) *for* (I) *and* (II)

(I)			
O(1)—C(9)	1.367 (3)	O(1)—C(2)	1.436 (4)
O(4)—C(4)	1.228 (4)	O(7)—C(7)	1.353 (4)
C(3)—C(4)	1.497 (4)	C(3)—C(2)	1.502 (5)
C(4)—C(10)	1.454 (4)	C(5)—C(6)	1.371 (5)
C(5)—C(10)	1.393 (4)	C(6)—C(7)	1.401 (4)
C(7)—C(8)	1.377 (4)	C(8)—C(9)	1.382 (4)
C(9)—C(10)	1.399 (4)		
(II)			
O(1)—C(2)	1.389 (6)	C(2)—C(20)	1.501 (7)
O(4)—C(4)	1.223 (6)	C(4)—C(10)	1.464 (6)
O(7)—C(11)	1.416 (6)	C(5)—C(10)	1.396 (7)
O(12*B*)—C(12)	1.313 (6)	C(7)—C(8)	1.379 (6)
C(2)—C(3)	1.442 (9)	C(9)—C(10)	1.389 (6)
C(3)—C(4)	1.501 (7)	C(11)—C(12)	1.499 (7)
C(5)—C(6)	1.361 (7)	C(20)—C(21)	1.369 (8)
C(6)—C(7)	1.397 (7)	C(21)—C(22)	1.385 (9)
C(8)—C(9)	1.397 (6)	C(23)—C(24)	1.332 (12)
O(1)—C(9)	1.367 (5)	C(13)—C(14)	1.464 (11)
O(7)—C(7)	1.372 (5)	C(20)—C(25)	1.376 (8)
O(12*A*)—C(12)	1.194 (6)	C(22)—C(23)	1.356 (10)
O(12*B*)—C(13)	1.456 (7)	C(24)—C(25)	1.369 (10)
O(7)—C(11)—C(12)	107.3 (4)	O(12*A*)—C(12)—O(12*B*)	125.1 (4)
O(12*A*)—C(12)—C(11)	125.1 (5)	O(12*B*)—C(12)—C(11)	109.8 (4)
O(12*B*)—C(13)—C(14)	110.1 (6)	C(2)—C(20)—C(21)	120.8 (5)
C(2)—C(20)—C(25)	120.1 (5)	C(21)—C(20)—C(25)	119.1 (5)
C(20)—C(21)—C(22)	119.6 (5)	C(21)—C(22)—C(23)	119.8 (6)
C(22)—C(23)—C(24)	120.8 (7)	C(23)—C(24)—C(25)	120.6 (7)
C(20)—C(25)—C(24)	120.0 (6)		

The structures of (I) and (II) were solved by direct methods and refined by full-matrix least squares. The phenyl ring in (I) shows disorder over two positions, denoted *a* and *b*, with an assumed occupancy of 0.5 for each. All H-atom parameters in (I) and (II) were refined, with the exception of the H atoms attached to the disordered phenyl rings in (I) and the H atoms bonded to C(2) and C(3) in (II), which were refined with isotropic displacement parameters using a riding model. The absorption correction applied to the data for (II) was not calculated using a crystal-based method due to a lack of strong reflections at high values of χ suitable for ψ-scans, and as the crystal used was in the form of a thin needle, it was difficult to index the faces for an analytical correction.

Data collection, cell refinement and data reduction: Siemens *P*3/*P*4 data collection software (PC version). Structure solution and refinement, molecular graphics and preparation of material for publication: *SHELXTL*/*PC* (Sheldrick, 1990).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1213). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Orthophénylènediammonium Bis(dihydrogénophosphate)

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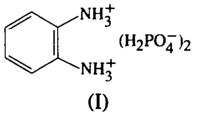
Abstract

The structure of 2-ammonioanilinium bis(dihydrogenphosphate), $C_6H_{10}N_2^{2+}.2H_2PO_4^-$, consists of alternating and imbricated sheets of phosphate anions and organic cations linked by hydrogen bonds.

Commentaire

L'étude de l'intéraction de la base 1,2-$NH_2C_6H_4NH_2$ avec les différents acides phosphoriques, nous a permis de préparer les phosphates organiques (1,2-$NH_3C_6H_4NH_3$)$H_2P_2O_7$ et (1,2-$NH_3C_6H_4NH_3$)$_2P_4O_{12}$,-$2H_2O$ (Soumhi & Jouini, 1995*a*,*b*). Dans ce travail, nous décrivons la structure cristalline d'un nouveau phosphate de la même base, (1,2-$NH_3C_6H_4NH_3$)$2H_2PO_4$, (I).



(I)

La Fig. 1 montre en projection parallèlement à l'axe *a*, une alternance de couches inorganiques $(H_2PO_4)^-$ et organiques $(NH_3C_6H_4NH_3)^{2+}$ imbriquées. Chaque anion $(H_2PO_4)^-$ est connecté à trois de ces voisins dans la couche par des liaisons hydrogène fortes [O···O 2,546 (2), 2,568 (2), 2,584 (2) et 2,667 (2) Å]. Les caractéristiques géométriques de ces anions concordent avec celles de $(H_2PO_4)^-$ dans la structure de (1,4-