

(Fair, 1990). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, including bond distances and angles involving H atoms, and torsion angles have been deposited with the IUCr (Reference: TA1022). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## *o*-Anisidinium Dihydrogenmonophosphate

MOHAMED OULD ABDELLAHI AND TAHAR JOUINI

*Département de Chimie, Faculté des Sciences,  
1060 Campus Universitaire, Tunis, Tunisia*

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## Abstract

The structure of *o*-anisidinium dihydrogenmonophosphate, C<sub>7</sub>H<sub>10</sub>NO<sup>+</sup>·H<sub>2</sub>PO<sub>4</sub><sup>−</sup>, consists of layers containing H<sub>2</sub>PO<sub>4</sub><sup>−</sup> anions alternating with layers built up of C<sub>7</sub>H<sub>10</sub>NO<sup>+</sup> cations, both layers being parallel to the *ab* plane. In the first type of layer, the H<sub>2</sub>PO<sub>4</sub><sup>−</sup> tetrahedra are connected through strong hydrogen bonds forming infinite (H<sub>2</sub>PO<sub>4</sub>)<sub>n</sub> chains with rather short internal P··P distances of 4.16 and 4.27 Å.

## Comment

The present study is part of a program aimed at examining the packing characteristics of compounds resulting from the interaction of various phosphoric acids and aromatic amines. To our knowledge, the anisidine–H<sub>3</sub>PO<sub>4</sub>

system has not yet been investigated. In the present work, we describe the chemical preparation and structure of *o*-anisidinium dihydrogenmonophosphate, (I), an example of such a compound.

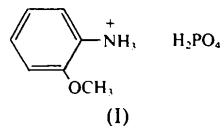


Fig. 1 shows the general atomic arrangement of (I) projected along the *c* axis. It can be described as an alternate stacking of planes containing H<sub>2</sub>PO<sub>4</sub><sup>−</sup> tetrahedra and planes built up of organic cations, the two types of layer being parallel to the *ab* plane. In the inorganic layers, the phosphoric entities form infinite zigzag (H<sub>2</sub>PO<sub>4</sub>)<sub>n</sub> chains parallel to the *a* direction. The H<sub>2</sub>PO<sub>4</sub><sup>−</sup> tetrahedra are connected through strong hydrogen bonds with O··O distances of 2.573 and 2.627 Å. Consequently, the P··P distance appears relatively short (4.16 and 4.27 Å). All bond distances and angles are normal and correspond well with those observed in similar structures (Averbuch-Pouchot & Durif, 1987; Averbuch-Pouchot, Durif & Guitel, 1988; Kamoun, Jouini, Kamoun & Daoud, 1989; Averbuch-Pouchot, 1993). The benzene ring is practically planar with the greatest deviation from the six-atom least-squares plane being 0.005 Å. The methoxy group makes an angle of 10° with the plane of the phenyl ring. The stability of the structure results from two types of hydrogen bond: the O—H··O type connecting the phosphoric entities in the (H<sub>2</sub>PO<sub>4</sub>)<sub>n</sub> chains and the N—H··O type connecting the NH<sub>3</sub><sup>+</sup> radicals, originating from the organic cation, to the external O atoms of the phosphoric entities of the inorganic chain. The first type of hydrogen bond is strong but the second is relatively weak according to the criterion of the donor–acceptor distance being less than or greater than 2.7 Å (Brown, 1976).

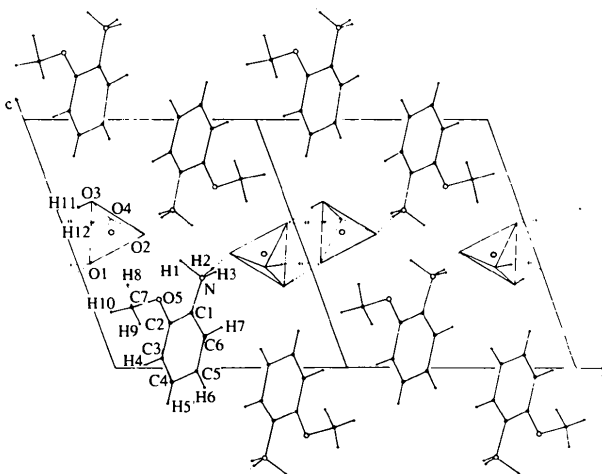
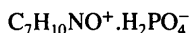


Fig. 1. Projection of the title structure onto the (100) plane.

## Experimental

*o*-Anisidinium dihydrogenmonophosphate was prepared by slow evaporation at room temperature of a mixture containing an aqueous solution of monophosphoric acid (84%,  $d = 1.70$ ) and an *o*-anisidine solution (97%,  $d = 1.092$ ) with the required stoichiometry. Several dissolutions followed by recrystallization led to well formed crystals.

### Crystal data



$$M_r = 221.15$$

Triclinic

$P\bar{1}$

$$a = 5.6560 (10) \text{ \AA}$$

$$b = 9.0660 (10) \text{ \AA}$$

$$c = 10.4200 (10) \text{ \AA}$$

$$\alpha = 109.694 (10)^\circ$$

$$\beta = 103.062 (10)^\circ$$

$$\gamma = 90.688 (10)^\circ$$

$$V = 487.77 (11) \text{ \AA}^3$$

$$Z = 2$$

$$D_x = 1.506 \text{ Mg m}^{-3}$$

### Data collection

Enraf–Nonius CAD-4 diffractometer

$\omega/2\theta$  scans

Absorption correction: none

1819 measured reflections

1715 independent reflections

1501 observed reflections

$$[I > 2\sigma(I)]$$

### Refinement

Refinement on  $F^2$

$$R(F) = 0.0408$$

$$wR(F^2) = 0.1644$$

$$S = 0.253$$

1715 reflections

175 parameters

All H-atom parameters refined

$$w = 1/[\sigma^2(F_o^2) + (0.4751P)^2 + 7.8262P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

Mo  $K\alpha$  radiation

$$\lambda = 0.7107 \text{ \AA}$$

Cell parameters from 22 reflections

$$\theta = 14\text{--}16^\circ$$

$$\mu = 0.279 \text{ mm}^{-1}$$

$$T = 293 (2) \text{ K}$$

Prism

$$0.24 \times 0.20 \times 0.16 \text{ mm}$$

Colorless

$$R_{\text{int}} = 0.0111$$

$$\theta_{\text{max}} = 25.02^\circ$$

$$h = -6 \rightarrow 6$$

$$k = -10 \rightarrow 10$$

$$l = 0 \rightarrow 12$$

3 standard reflections

frequency: 60 min

intensity decay: 0.021%

$$(\Delta/\sigma)_{\text{max}} = -0.002$$

$$\Delta\rho_{\text{max}} = 0.265 \text{ e \AA}^{-3}$$

$$\Delta\rho_{\text{min}} = -0.383 \text{ e \AA}^{-3}$$

Atomic scattering factors

from *International Tables for Crystallography* (1992),

Vol. C, Tables 4.2.6.8 and 6.1.1.4)

C5	0.2996 (9)	0.6677 (6)	1.0104 (4)	0.0673 (11)
C6	0.2349 (7)	0.5696 (5)	0.8716 (4)	0.0539 (9)
C7	0.8464 (9)	0.8348 (5)	0.7549 (6)	0.0636 (11)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

P—O2	1.487 (2)	C1—C6	1.370 (5)
P—O1	1.501 (2)	C1—C2	1.382 (4)
P—O4	1.561 (2)	C2—C3	1.386 (5)
P—O3	1.564 (2)	C3—C4	1.371 (6)
O5—C2	1.357 (4)	C4—C5	1.370 (7)
O5—C7	1.427 (5)	C5—C6	1.379 (6)
N1—C1	1.454 (4)		
O2—P—O1	116.4 (1)	C2—C1—N1	117.8 (3)
O2—P—O4	106.3 (1)	O5—C2—C1	115.3 (3)
O1—P—O4	110.2 (1)	O5—C2—C3	126.0 (3)
O2—P—O3	109.4 (1)	C1—C2—C3	118.7 (3)
O1—P—O3	109.5 (1)	C4—C3—C2	119.5 (4)
O4—P—O3	104.2 (1)	C5—C4—C3	121.5 (4)
C2—O5—C7	118.3 (3)	C4—C5—C6	119.5 (4)
C6—C1—C2	121.5 (3)	C1—C6—C5	119.3 (4)
C6—C1—N1	120.7 (3)		
C7—O5—C2—C1	170.1 (3)	C7—O5—C2—C3	−9.8 (6)

Table 3. Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ )

D—H...A	D—H	H...A	D...A	D—H...A
N1—H1...O2 <sup>i</sup>	0.87 (5)	1.88 (5)	2.716 (4)	160 (4)
N1—H2...O2 <sup>ii</sup>	0.92 (5)	1.84 (5)	2.730 (4)	164 (4)
O3—H11...O1 <sup>iii</sup>	0.86 (6)	1.77 (6)	2.627 (3)	174 (5)
O4—H12...O1 <sup>iv</sup>	0.86 (5)	1.71 (5)	2.573 (3)	180 (5)

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

The  $\theta$ -scan width was  $(0.90 + 0.35 \tan \theta)^\circ$ . Data were corrected for Lorentz and polarization effects.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, including bond distances and angles involving H atoms, and torsion angles have been deposited with the IUCr (Reference: DU1096). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

$$U_{\text{eq}} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$U_{\text{eq}}$
P	0.23372 (12)	0.81665 (7)	0.45225 (7)	0.0295 (3)
O1	0.2716 (3)	0.9614 (2)	0.5815 (2)	0.0346 (5)
O2	0.1615 (5)	0.6660 (3)	0.4670 (3)	0.0524 (7)
O3	0.0428 (4)	0.8423 (3)	0.3292 (2)	0.0446 (6)
O4	0.4719 (4)	0.7920 (2)	0.3988 (2)	0.0428 (6)
O5	0.6702 (5)	0.7056 (3)	0.7232 (3)	0.0531 (6)
N1	0.2948 (6)	0.4899 (3)	0.6323 (3)	0.0410 (6)
C1	0.3633 (5)	0.5880 (3)	0.7802 (3)	0.0368 (7)
C2	0.5581 (6)	0.7015 (4)	0.8243 (3)	0.0413 (7)
C3	0.6217 (8)	0.7995 (5)	0.9639 (4)	0.0569 (9)
C4	0.4911 (8)	0.7815 (5)	1.0545 (4)	0.0629 (10)