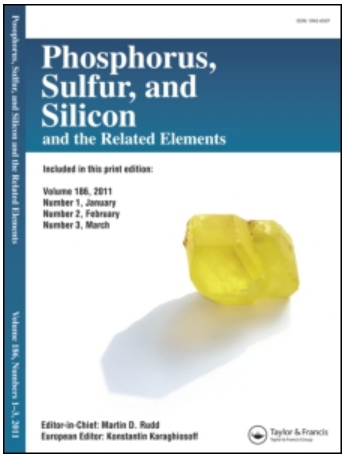

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Crystal Structure and Spectroscopic Studies of a New Organic Dihydrogenmonophosphate $[2\text{-}NH_2\text{-}6\text{-}CH_3\text{-}C_4H_3N_2O]_2(H_2PO_4)_2$

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Crystal Structure and Spectroscopic Studies of a New Organic Dihydrogenmonophosphate $[2\text{-}NH_2\text{-}6\text{-}CH_3\text{-}C_4H_3N_2O]_2(H_2PO_4)_2$

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Physicochemical properties of a new dihydrogenmonophosphate $[2\text{-}NH_2\text{-}6\text{-}CH_3\text{-}C_4H_3N_2O]_2(H_2PO_4)_2$ are described on the basis of X-ray crystal structure investigation. This compound crystallizes in the triclinic space group P-1. The unit cell parameters are: $a = 7.667(3)$ Å, $b = 8.204(5)$ Å, $c = 14.761(6)$ Å, $\alpha = 98.85(4)^\circ$, $\beta = 99.23(3)^\circ$, $\gamma = 90.50(4)^\circ$, $V = 905.0$ Å^3, and $Z = 2$. The crystal structure was solved and refined to $R = 0.037$, using 4351 independent reflections. The atomic arrangement of this compound is built up by $(H_2PO_4)_n^{n-}$ chains. Each chain aggregates with organic molecules to form an open framework architecture through hydrogen bond interactions. The structure includes four types of hydrogen bonds. The first one, O—H—O, links the H_2PO_4 groups to form $(H_2PO_4)_n^{n-}$ infinite inorganic chains parallel to the a axis. The three other types, O—H—O(carbonylic), N—H—O(carbonylic), and N—H—O, assemble the inorganic chains so as to build up a three-dimensional arrangement. This compound has also been investigated by IR, and solid-state ^{13}C and ^{31}P MAS NMR spectroscopies combined to ab initio calculations.

Keywords Ab initio calculations; IR spectroscopy; NMR spectroscopy; X-ray diffraction

INTRODUCTION

Organic-cation monophosphates have been widely studied due to their numerous practical uses in different areas of chemistry.[1] In these materials, the phosphate anions are interconnected by strong hydrogen

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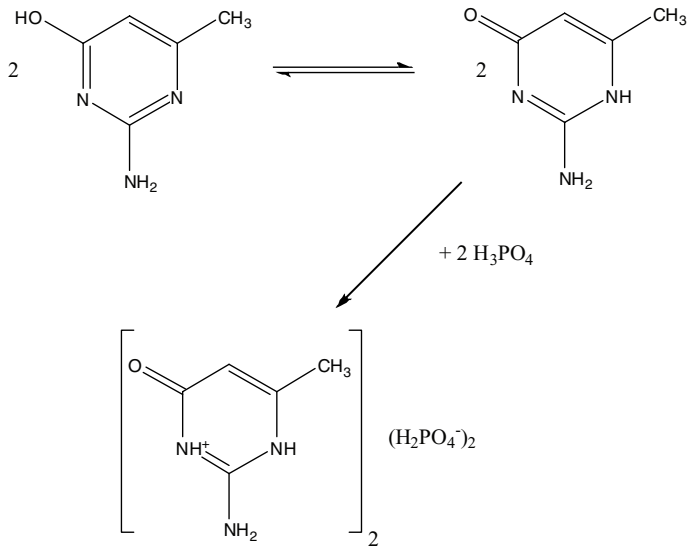
bonds so as to build infinite networks with various geometries: ribbons,[2] chains[3,4] or layers.[5,6] Until now, the monohydrogenomonophosphate [2-NH_2-6-CH_3-$C_4H_3N_2O]_2HPO_4$ is the only monophosphate complex of 2-amino-6-methylpyrimidin-4(1H)-one known.[7] In this article, we report the synthesis and the crystal structure investigation of a new hybrid organic–inorganic compound [2-NH_2-6-CH_3-$C_4H_3N_2O]_2(H_2PO_4)_2$. The characterization of the title compound by solid state ^{31}P and ^{13}C NMR combined with ab initio calculations and infrared spectroscopies is also reported.

RESULTS AND DISCUSSION

Structure Description

Interatomic distances, bond angles, and the hydrogen bonds scheme of [2-NH_2-6-CH_3-$C_4H_3N_2O]_2(H_2PO_4)_2$ are given in Table I.

An examination of Table I data shows that the distances values of C2-O9 [1.230 (2) Å] and C9-O10 [1.222 (2) Å] clearly indicate two double bonds. This allows us to confirm that the first step of the preparation of the title compound consists in the transformation of the 2-amino-6-methyl-4-pyrimidinol into 2-amino-6-methylpyrimidin-4-(1H)-one (Scheme 1):



SCHEME 1

TABLE I Main Interatomic Distances (Å) and Bond Angles (°) in the $[C_5H_8N_3O]_2$ $[H_2PO_4]_2$Atomic Arrangement

$P(1)O_4$ tetrahedron				
P(1)	O(5)	O(6)	O(7)	O(8)
O(5)	1.567(16)	2.509(2)	2.529(2)	2.459(3)
O(6)	106.38(9)	1.564(15)	2.474(3)	2.545(4)
O(7)	110.40(9)	107.13(8)	1.510(14)	2.519(3)
O(8)	106.61(9)	112.37(9)	113.74(8)	1.497(14)
O(5)-H(O5)	0.908Å	P(1)-O(5)-H(O5)	115.25°	
O(6)-H(O6)	0.888Å	P(1)-O(6)-H(O6)	116.49°	
$P(2)O_4$ tetrahedron				
P(2)	O(1)	O(2)	O(3)	O(4)
O(1)	1.568(15)	2.507(2)	2.546(3)	2.461(2)
O(2)	105.87(9)	1.572(15)	2.461(4)	2.541(3)
O(3)	111.72(8)	106.03(8)	1.5077(14)	2.525(3)
O(4)	106.98(8)	111.66(9)	114.33(8)	1.495(15)
O(1)-H(O1)		0.881Å	P(2)-O(1)-H(O1)	112.45°
O(2)-H(O2)		0.893Å	P(2)-O(2)-H(O2)	111.79°
$[C_5H_8N_3O]_2^+$ cation				
N(1)-C(1)	1.343(2)		C(1)-N(1)-C(4)	122.12(15)
N(1)-C(4)	1.378(2)		C(1)-N(3)-C(2)	123.55(15)
N(2)-C(1)	1.309(2)		N(3)-C(2)-C(3)	115.60(15)
N(3)-C(1)	1.343(2)		O(9)-C(2)-C(3)	126.26(15)
N(3)-C(2)	1.389(2)		O(9)-C(2)-N(3)	118.13(12)
N(4)-C(6)	1.347(2)		C(2)-C(3)-C(4)	120.47(16)
N(4)-C(9)	1.402(2)		C(3)-C(4)-N(1)	119.50(16)
N(5)-C(6)	1.316(2)		C(3)-C(4)-C(5)	124.92(18)
N(6)-C(6)	1.342(2)		C(5)-C(4)-N(1)	115.58(17)
N(6)-C(7)	1.382(2)		N(1)-C(1)-N(3)	118.63(15)
C(2)-C(3)	1.435(3)		N(1)-C(1)-N(2)	121.23(16)
C(2)-O(9)	1.230(2)		N(3)-C(1)-N(2)	120.13(16)
C(3)-C(4)	1.348(3)		C(6)-N(4)-C(9)	124.12(15)
C(4)-C(5)	1.490(3)		N(4)-C(6)-N(6)	118.26(16)
C(7)-C(8)	1.342(3)		N(5)-C(6)-N(4)	120.47(16)
C(7)-C(10)	1.489(3)		N(5)-C(6)-N(6)	121.26(16)
C(8)-C(9)	1.438(3)		C(6)-N(6)-C(7)	121.89(15)
C(9)-O(10)	1.222(2)		N(6)-C(7)-C(8)	120.33(16)
			C(6)-C(7)-C(10)	115.11(16)
			C(10)-C(7)-C(8)	124.55(18)
			C(7)-C(8)-C(9)	120.44(17)
			C(8)-C(9)-N(4)	114.86(16)
			C(8)-C(9)-O(10)	126.40(19)
			N(4)-C(9)-O(10)	118.74(18)
Hydrogen bonds				
O(N)-H···O	O(N)···O(Å)	O(N)-H (Å)	H···O(Å)	O(N)-H···O(°)
O(1)-H(1)···O8	1.69	0.88	2.572(3)	176
O(2)-H(2)···O7	1.61	0.89	2.504(2)	173

(Continued on next page)

TABLE I Main Interatomic Distances (Å) and Bond Angles (°) in the $[C_5H_8N_3O]_2$ $[H_2PO_4]_2$Atomic Arrangement *(Continued)*

$P(1)O_4$ tetrahedron				
O(6)-H(3)···O3	1.71	0.89	2.593(2)	173
O(5)-H(4)···O9	1.73	0.91	2.635(3)	176
N(2) -H(5)···O4	1.93	0.89	2.798(3)	167
N(2)-H(6)···O10	2.25	0.90	2.815(3)	120
N(3)-H(7)···O3	1.81	0.90	2.702(3)	175
N(1)-H(9)···O2	1.96	0.86	2.804(3)	168
N(5)-H(13)···O1	2.06	0.95	3.003(3)	171
N(5)-H(14)···O8	1.71	1.06	2.770(3)	174
N(6)-H(15)···O4	1.80	0.87	2.669(3)	178
N(4)-H(17)···O7	1.96	0.87	2.821(3)	171

Esd are given in parentheses.

Figure 1 shows the ORTEP plot of the structure, including the atoms labels and their vibrational ellipsoids at 40% probability. The asymmetric unit of the crystal structure consists of two phosphate anions $H_2PO_4^-$ and two organic cations $[2\text{-}NH_2\text{-}6\text{-}CH_3\text{-}C_4H_3N_2O]^+$.

The structure can be described as being built up by chains of $H_2PO_4^-$ anions spreading with planes $y = 1/2$ or $z = (2n + 1)/4$ aggregates with organic molecules, leading to an open framework architecture through hydrogen bond interactions. The structure in projection on the (b, c)

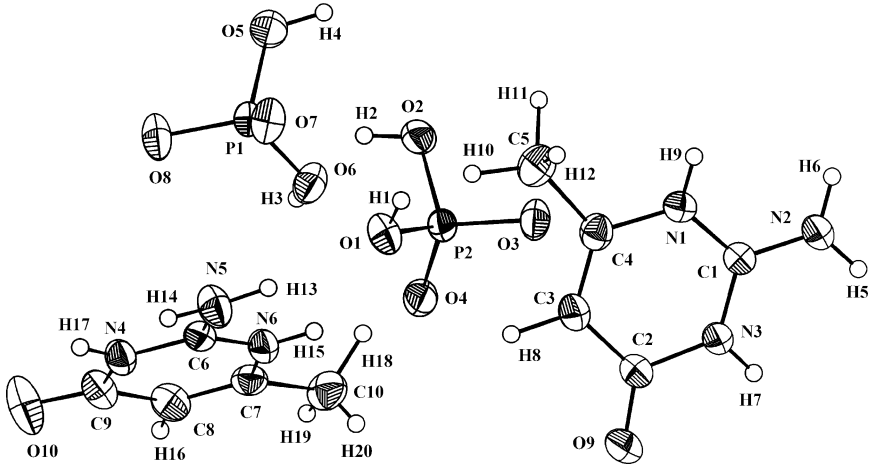


FIGURE 1 Asymmetric unit of $[2\text{-}NH_2\text{-}6\text{-}CH_3\text{-}C_4H_3N_2O]_2(H_2PO_4)_2$. Thermal ellipsoids are shown at 40% probability.

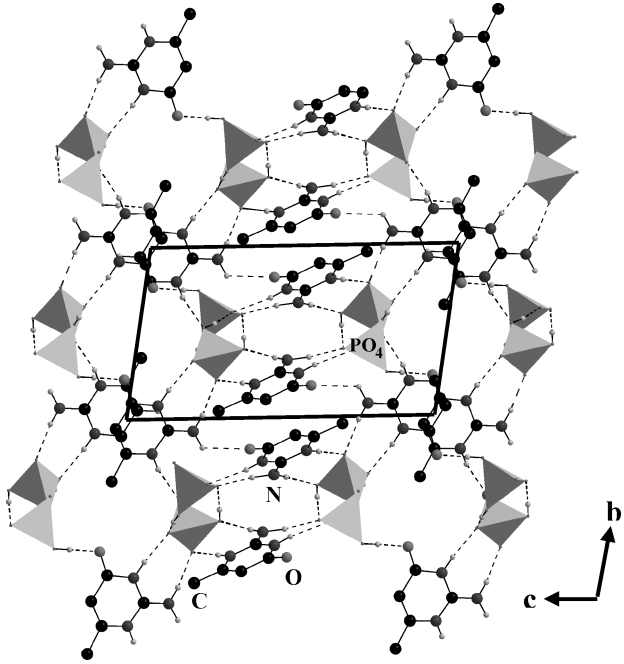


FIGURE 2 Projection of the structure of $[2\text{-}NH_2\text{-}6\text{-}CH_3\text{-}C_4H_3N_2O]_2(H_2PO_4)_2$ along the *a* axis. A polyhedral representation is used for the PO_4 tetrahedron.

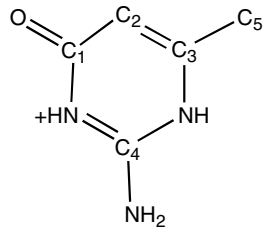
plane shows two types of columns constructed by anions and cations running along the *a* axis, the first formed by four $H_2PO_4^-$ and four $[2\text{-}NH_2\text{-}6\text{-}CH_3\text{-}C_4H_3N_2O]^+$, the second by four $H_2PO_4^-$ and two $[2\text{-}NH_2\text{-}6\text{-}CH_3\text{-}C_4H_3N_2O]^+$ (Figure 2).

In the chain organization of the $H_2PO_4^-$ entity, it is worth noticing that the O···O distances involved in hydrogen bonds (2.504(2) to 2.635(3) Å) are of the same magnitude as the O—O edges in the PO_4 tetrahedron (2.459(3) to 2.546(3) Å). The strength of O—H···O hydrogen bonds and the shortest P—P distance (4.959Å) between phosphorus atoms of adjacent chains are in favor of the general formation of $[H_2PO_4]_n^{n-}$ polyanions in the crystal structure, and not of the individualization of $H_2PO_4^-$ groups. This polymeric structure is in accordance with what has been observed in other crystal structures.[11]

When looking at the $H_2PO_4^-$ geometrical features (Table I), it can be noticed the existence of two types of P···O distances. The longest ones (1.567(16), (1.564(15), 1.568(15), and 1.572(15)Å) correspond to the P—OH groups, and the shortest ones (1.510(14), 1.497(14), 1.507(7),

TABLE II Comparison of Calculated and Experimental Carbon-13 Chemical Shifts for the Organic Ligand in the $[C_5H_8N_3O]_2$ $[H_2PO_4]_2$ Compound

Atom number	Full optimization	No optimization		Optimization of protons (L2)			
		L1	L2	δ(ppm)	d(C—H) nm Before	d(C—H) nm After	Exp.
1	144.9	149.3	147.5	147.7	—	—	163.2 and 164.0 ?
2	105.6	104.6	101.4	104.3	0.987	1.082	106.1 and 106.7
3	147.0	141.2	141.5	141.6	—	—	signals in the 151–155 range?
4	142.0	138.2	141.1	139.6	—	—	
5	20.6	19.5	−0.9	20.6	1.089 0.934 0.763	1.098 1.098 1.091	20.7 and 21.2



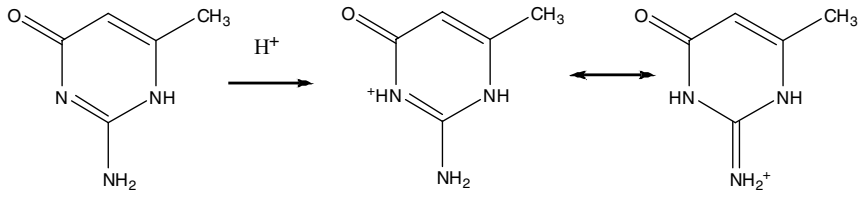
and 1.495(15) Å) correspond to the classical P=O bonds. The average of the P—O distances and the O—P—O angles are [1.534 Å and 109.43°] and [1.535 Å, and 109.43°], respectively for phosphorus atoms P(1) and P(2). These values agree perfectly with what is generally observed for other phosphate anions.[12] The O—P—O angles spread in the range 105.87(9) to 114.33(8)°. This distortion from the ideal tetrahedral value has been regularly noted in literature.[12]

An examination of the organic moiety geometrical features shows that the bond length values of C2-C3 [1.435 (2) Å] and C8-C9 [1.438 (3) Å], which cannot be considered as double bonds, are longer than C3-C4 [1.348 (3) Å] and C7-C8 [1.342 (3) Å]. Similarly, the bond length values of C2-N3 [1.389 (2) Å] and C9-N4 [1.402 (2) Å], which cannot be considered as double bonds, are longer than C1-N3 [1.343 (2) Å] and C6-N4 [1.347 (2) Å]. Furthermore, the organic molecule is protonated in acidic solution. The bonding of the H atom to the ring N atom gives

TABLE III Crystal Data and Structure Refinement of $[C_5H_8N_3O]_2$ $[H_2PO_4]_2$

Empirical formula	C10H20N6O10P2
Formula weight	446.26
Temperature (K)	293
Crystal system	Triclinic
Space group	P-1
a	7.667(3) Å
b	8.204(5) Å
c	14.761(6) Å
α	98.85(4)°
β	99.23 (3)°
γ	90.50(4)°
V	905.0(7) A°3
Z	2
.ρ_{cal}	1.638 g.cm^{-3}
F(000)	464
Crystal size	0.3 × 0.2 × 0.2 [mm]
μ(MoKα)	0.307 (mm^{-1})
Index range: ±h, ±k, l	($h_{max} = \pm 10$, $k_{max} = \pm 10$, $l_{max} = 19$)
Collected reflections	4516
Independent reflections	4351
Unique reflections included	3905
R_{int}	0.01
Refined parameters	273
R (I> 2.σ(I))	0.036
Rw	0.105
Goodness of fit	1.14

an ion, for which an additional resonance structure can be written as depicted in Scheme 2:[13]



SCHEME 2

The present investigation clearly shows that the positive charge is on the pyrimidinium ring N atom: the N2-C1 bond length (1.309 (2) Å) is slightly shorter than the N1-C1 (1.343(2) Å) and N3-C1 (1.343 (2) Å) ones. Similarly, the N5-C6 bond length (1.316(2) Å) is slightly

shorter than the N4-C6 (1.347(2) Å) and N6-C6 (1.342 (2) Å) ones. The sum of the angles around N1, N3, N4, and N6 are 360.00, 359.45, 359.80, and 359.00°, respectively. All these bond lengths and angles features are consistent with the imino resonance, and suggest a great contribution from it to the title compound. The N2-C1 (1.309 (2) Å) and N5-C6 (1.316(2) Å) distances are approximately equal to that of a C=N double bond length, indicating that N2 and N5 nitrogen atoms of the amino group are probably in a sp^2 hybridization. This is also supported by the values of the N1-C1-N2, N1-C1-N3, and N2-C1-N3 angles (121.23(16), 118.63(16), and 120.13(16)°, respectively) and of the N4-C6-N5, N4-C6-N6, and N5-C6-N6 angles (120.47(16), 118.26(16), and 121.26(16)°, respectively). Similar bond distances and angles were observed in 2-amino-4-methyl-6-oxo-3,6-dihydropyrimidin-1-ium hydrogenmonophosphate,[7] 2-aminopyridinium adipate monoadipic acid,[14] in 2-aminopyridinium benzoate,[15] and in some 2-aminopyridine-containing molecules.[16–18]

It may be noted that controlling packing modes in crystalline solids using suitable functional groups could be achieved through well-defined directional interactions such as hydrogen bonds. The organic molecules are similarly linked into centrosymmetric dimers by hydrogen bond and $\pi - \pi$ interactions. So this structure includes ten hydrogen bond donors (four O and six N atoms) and eight hydrogen bond acceptors (eight O atoms). The oxygen acceptor atoms O(3), O(4), O(7), and O(8) are twofold acceptors.

NMR Results

The proton decoupled ^{31}P MAS NMR spectrum of the crystalline dihydrogenmonophosphate $[2\text{-}NH_2\text{-}6\text{-}CH_3\text{-}C_4H_3N_2O]_2(H_2PO_4)_2$ is shown in Figure 3. It exhibits two sharp resonance peaks at 2.15 and −3.9 ppm. These chemical shift values agree with those corresponding to monophosphates (between −10 and + 5 ppm), depending on the compound.[19–25] The existence of two peaks in the spectrum clearly indicates the presence of two crystallographic sites in the unit cell of this compound, in agreement with the X-ray structure results. When recording the spectrum at a low spinning rate, numerous spinning sidebands appear (Figure 3). By deconvolution of the corresponding spectra with the DMFIT software, it is then possible to determine, for each phosphorus signal, not only its isotropic chemical shift (obtained also with a high spinning rate) but also its anisotropic parameters. This calculation allows also us to calculate the relative amounts of the two nuclei, a non-negligible part of the intensity being in the spinning sidebands.

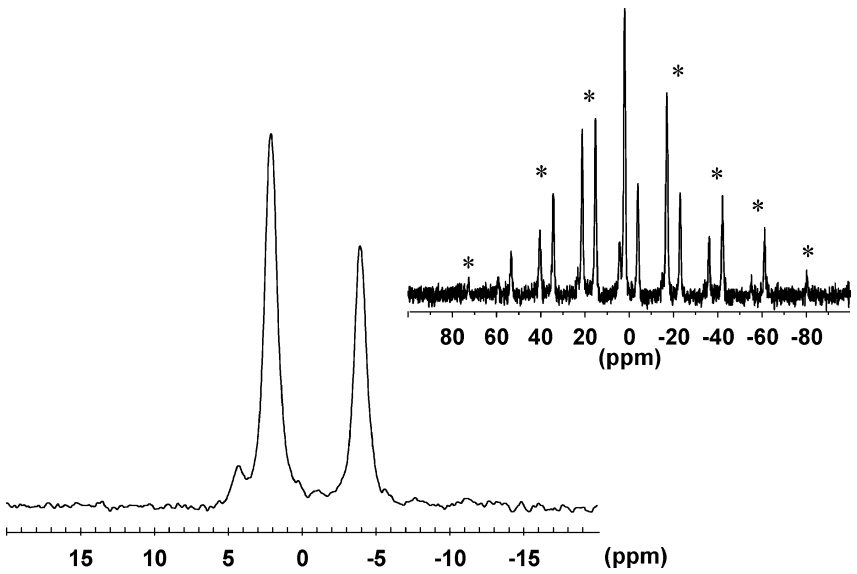


FIGURE 3 ^{31}P MAS-NMR spectrum of $[2\text{-}NH_2\text{-}6\text{-}CH_3\text{-}C_4H_3N_2O]_2(H_2PO_4)_2$ (* = spinning side bands).

The following results were obtained:

$$\text{Site 1} : \delta_{iso} = 2.15 \text{ ppm} \quad \Delta\delta = 45.8 \text{ ppm}$$
$$\eta = 0.85 \quad 52.1\% \text{ of the intensity}$$
$$\text{Site 2} : \delta_{iso} = -3.90 \text{ ppm} \quad \Delta\delta = -78.3 \text{ ppm}$$
$$\eta = 0.70 \quad 45.6\% \text{ of the intensity}$$

The small signal at 4.35 ppm corresponds to an impurity (ca. 0.1% of the phosphorus in the solid as determined by integration of all peaks in the +100/−100 ppm range) that was not identified.

The main difference between the two sites is their chemical shift anisotropy. This could be used for their attribution to the two crystallographically unequivalent phosphorus atoms. For this purpose, theoretical calculations of the NMR chemical shifts of the phosphorus atoms in the two PO_4 groups were made with the GIAO method. Unfortunately, the results do not allow us to discriminate between the two sites, as not only the isotropic chemical shifts but also the anisotropy coefficients are quite comparable for the two phosphorus atoms (isotropic chemical shifts 359.8 and 359.4 ppm for P(1) and P(2), respectively, anisotropy 23 and 25 ppm). This can be due to the fact that for these calculations only the PO_4 tetrahedra were taken into account. It should be better to use

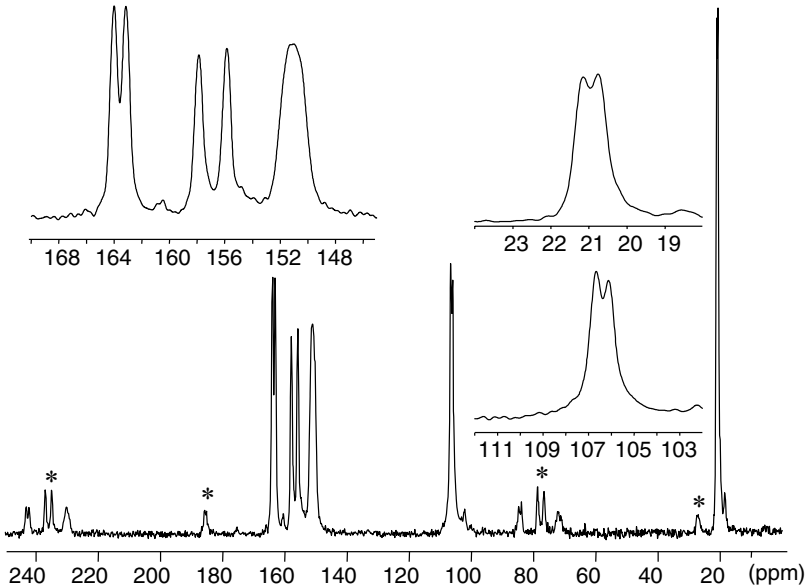


FIGURE 4 ^{13}C CP-MAS-NMR spectrum of $[2\text{-}NH_2\text{-}6\text{-}CH_3\text{-}C_4H_3N_2O]_2(H_2PO_4)_2$ (* = spinning ide bands).

a model that takes into account the interactions between these tetrahedra and the organic moieties, but this could be highly time-consuming.

The ^{13}C CP-MAS-NMR spectrum of crystalline dihydrogenmonophosphate $[2\text{-}NH2\text{-}6\text{-}CH_3\text{-}C_4H_3N_2O]_2(H_2PO_4)_2$ given in Figure 4 shows nine resonances, all peaks being doublets (with the exception of the signal at ca. 151 ppm). These results are in full agreement with the presence of two organic molecules in the unit cell. The attribution of the signals at 20.7 and 21.2 ppm to the unequivalent methyl carbons C5 and C10 is relatively easy and proves the presence of two organic entities in the asymmetric unit, which agrees perfectly with the X-ray structure. In order to have more information from the carbon-13 NMR spectrum, ab initio calculations were performed (at the B3LYP/6-31+G* level), and the carbon-13 NMR chemical shifts were calculated (by use of the GIAO method). The following different calculations were made:

- Full optimization of the organic ligand
- Calculation of the chemical shifts for the two unequivalent organic molecules of the unit cell by taking the X-ray coordinates
- For one of the two ligands (that for which the discrepancy experiment/theory was the higher), optimization of the positions of the

protons, the coordinates of the C, N and O atoms being taken from the X-ray determination

The results are given in Table II, which gives the calculated chemical shifts (after subtraction from the chemical shift of tetramethylsilane calculated at the same level of theory) for the three calculations. The d(C—H) distances before and after protons optimization are also given, together with the experimental chemical shifts and the possible attributions. The first conclusion is that the position of the proton has a great influence on the chemical shift of the carbon to which it is directly bonded. However, it has no influence on the chemical shifts of the other carbon atoms. The calculated d(C—H) values are more realistic after optimization and lead to chemical shifts quite in good agreement with the experiment. However, it must be pointed out that all chemical shifts, whatever the calculation method, are always lower then 150 ppm, while experimental chemical shifts are found until 164 ppm. There are two possible explanations for these discrepancies: the location of the C and N atoms is not exactly at the good position (for example due to a jump between two positions), or, more probably, the hydrogen bonds were not taken into account and very probably the bigger effect will be on the carbonyl group. It is for this reason that we have proposed to attribute this signal to the most deshielded peaks.

IR Absorption Spectroscopy

The IR spectrum of crystalline $[2\text{-}NH_2\text{-}6\text{-}CH_3\text{-}C_4H_3N_2O]_2(H_2PO_4)_2$ is shown in Figure 5. The most representative and characteristic vibrational modes of this compound can be compared to those of similar monophosphates.[26] Some aspects of the performed assignments are briefly commented on here:

- The high-frequency region, between 3500–2700 cm^{-1}, corresponds to the valence vibrations of C-H, N-H, and O—H groups interconnected by a system of hydrogen bonds in the crystal.[27]
- The very strong band at 1700 cm^{-1} is related to the valence vibration of the C=O group and confirms the transformation of 2-amino-6-methyl-4-pyrimidinol into 2-amino-6-methylpyrimidin-4-(1H)-one when preparing the title compound.
- The bands between 1600 and 1350 cm^{-1} correspond to the ν(N-H), ν(C—H), and ν(O—H) stretching vibrations.[28]

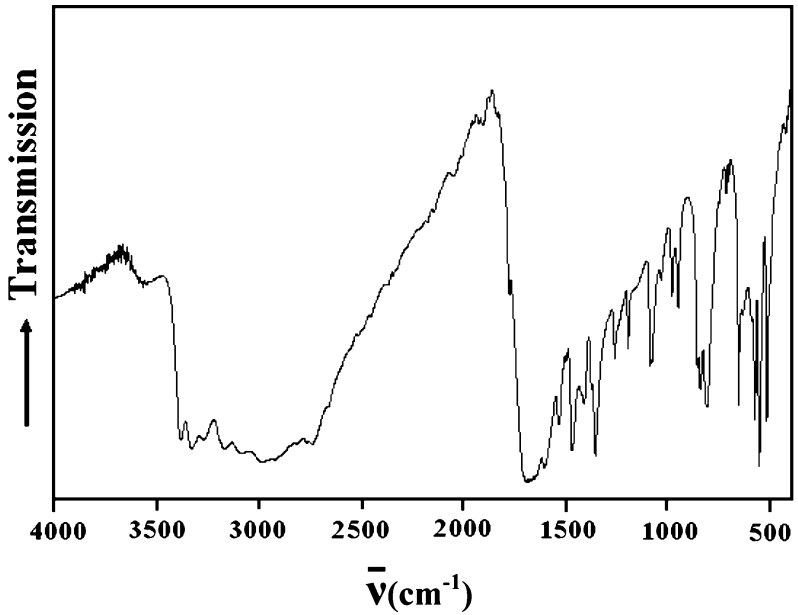


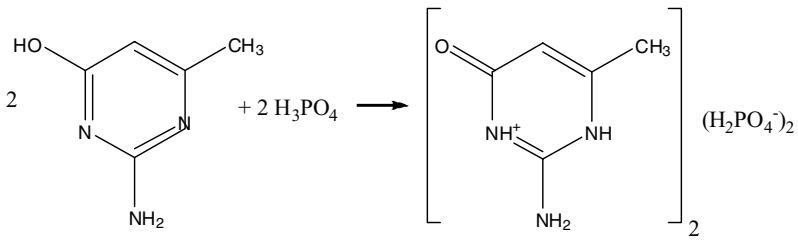
FIGURE 5 Infrared spectra of $[2\text{-}NH_2\text{-}6\text{-}CH_3\text{-}C_4H_3N_2O]_2(H_2PO_4)_2$.

- The band at 1230 cm^{-1} corresponds to the δ(P—O—H) in-plane bending, while the out-of-plane bending vibrations are observed at 880 and 840 cm^{-1}.[28]
- Bands ranging from 650 to 400 cm^{-1} are attributed to γ_{as} and γ_s out-of-plane bending vibrations of PO_4 groups.[29,30]

EXPERIMENTAL

Chemical Preparation

Crystals of the title compound were prepared according to the following chemical reaction:



by adding in a crystallizer, 25 mmol of concentrated orthophosphoric acid (Fluka, 85%, d = 1.7) to a solution of acetone and distilled water (V/V) containing 25 mmol of 2-amino-6-methyl-4-pyrimidinol (Acros). The so-obtained solution was slowly evaporated at room temperature until the formation of a single crystal of 2-amino-6-methylpyrimidin-4(1H) dihydrogenmonophosphate (5.9 g, 53% yield). Anal. calcd. for $C_{10}H_{20}N_6O_{10}P_2$: C, 26.89; H, 4.48; N, 18.26; P, 13.89; Found: C, 26.78; H, 4.32; N, 18.02; P, 13.76. The crystals remain stable for months in normal conditions of temperature and humidity.

Investigation Techniques

The title compound has been studied by various physicochemical methods: X-ray diffraction, solid-state NMR spectroscopy combined with ab initio calculations, infrared spectroscopy.

X-Ray Diffraction

A single crystal was carefully selected under a polarizing microscope in order to perform its structural analysis by X-ray diffraction. The intensity data were collected on an Enraf-Nonius MACH3 automated four-circle diffractometer using graphite monochromated Mo$K\bar{\alpha}$ radiation, $\lambda = 0.7107$ Å. The structure was solved by direct methods using the SIR92[8] program and refined by the full matrix least-squares techniques using SHELX-L.[9] The drawings were made with Diamond.[10] All nonhydrogen atoms were refined anisotropically. The hydrogen atoms position were located by difference-Fourier synthesis and refined. Crystal data and experimental parameters used for the intensity data collection are summarized in Table III.

Physical Measurement

All NMR spectra were recorded on a Bruker DSX-300 spectrometer operating at 75.49 MHz for ^{13}C and 121.51 MHz for ^{31}P with a classical 4 mm probehead allowing spinning rates up to 10 kHz. ^{13}C NMR chemical shifts are given relative to tetramethylsilane and ^{31}P one relative to H_3PO_4 (external references, precision 0.5 ppm). The carbon spectrum was recorded by use of cross-polarization (CP) from protons (contact time 5 ms) and MAS, while the phosphorus one was obtained under classical MAS conditions. In all cases it was checked that there was a sufficient delay between the scans allowing a full relaxation of the nuclei. Theoretical calculations on the two organic molecules present in the crystal were made by use of the Gaussian 98 software at the B3LYP/6-31+G* level.

Spectra were recorded in the range 4000–400 cm^{-1} with a Perkin-Elmer FTIR spectrophotometer 1000 using a sample dispersed in spectroscopically pure KBr pellet.

Supplementary Material

Crystallographic data for the title compound has been deposited at the Cambridge Crystallographic Data Center as supplementary publication CCDC-666269. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223/336 033; e-mal mail to: deposit@ccdc.cam.ac.uk).

CONCLUSION

The title compound was obtained by the reaction of orthophosphoric acid with 2-amino-6-methyl-4-pyrimidinol. From the single crystal X-ray diffraction, IR and ^{13}C CP-MAS-NMR spectra, it can be concluded that the 2-amino-6-methyl-4-pyrimidinol was transferred into 2-amino-6-methylpyrimidin-4-(1H)-one. The atomic arrangement of this compound is built up by by $(H_2PO_4)_n^{n-}$ chains. Each chain aggregates with organic molecules to form an open framework architecture through multiple hydrogen bond interactions. Solid-state MAS NMR and infrared spectroscopies are in full agreement with the X-ray structure.

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