

2-Amino-4,6-dimethylpyrimidinium dihydrogenphosphate

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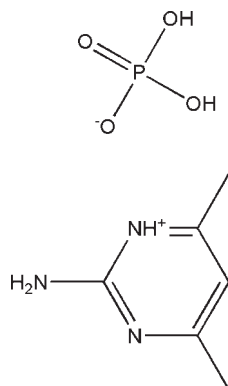
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.035; wR factor = 0.108; data-to-parameter ratio = 16.5.

In the crystal structure of the title compound, $\text{C}_6\text{H}_{10}\text{N}_3^{+}\cdot\text{H}_2\text{PO}_4^{-}$, the cations and anions are linked by intermolecular $\text{O}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, forming a two-dimensional network. Additional stabilization is provided by weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ interactions. $\text{N}-\text{H}\cdots\text{N}$ interactions are also present.

Related literature

Five and six-membered heterocyclic compounds often exist in biologically active natural products and synthetic compounds of medicinal interest, see: Gilchrist (1998). For methylpyrimidines as precursors to potentially bioactive pyrimidine derivatives, see: Xue *et al.* (1993). For Ru complexes of pyrimidine with an $-\text{NH}_2$ substituent, see: Zhu *et al.* (2008). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$\text{C}_6\text{H}_{10}\text{N}_3^{+}\cdot\text{H}_2\text{PO}_4^{-}$
 $M_r = 221.16$
 Monoclinic, $P2_1/n$
 $a = 11.743$ (2) Å
 $b = 4.8266$ (10) Å
 $c = 16.940$ (3) Å
 $\beta = 95.55$ (3)°

$V = 955.6$ (3) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.28$ mm⁻¹
 $T = 293$ K
 $0.20 \times 0.15 \times 0.11$ mm

Data collection

Bruker SMART CCD diffractometer
 Absorption correction: none
 8743 measured reflections

2193 independent reflections
 1973 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.108$
 $S = 1.08$
 2193 reflections

133 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.40$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.41$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O1}$	0.93	1.75	2.667 (2)	168
$\text{O2}-\text{H2A}\cdots\text{O3}^{\text{i}}$	0.87	1.70	2.560 (2)	171
$\text{N3}-\text{H3A}\cdots\text{O3}$	0.82	2.02	2.831 (2)	170
$\text{N3}-\text{H3B}\cdots\text{N2}^{\text{ii}}$	0.93	2.07	3.004 (2)	178
$\text{O4}-\text{H4A}\cdots\text{O1}^{\text{iii}}$	0.92	1.68	2.594 (2)	171
$\text{C3}-\text{H3D}\cdots\text{O2}^{\text{iv}}$	0.93	2.40	3.319 (2)	171

Symmetry codes: (i) $x, y + 1, z$; (ii) $-x, -y - 1, -z + 2$; (iii) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$; (iv) $x + \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2909).

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supporting information

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2-Amino-4,6-dimethylpyrimidinium dihydrogenphosphate

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S1. Comment

Five and six-membered heterocyclic compounds are important constituents that often exist in biologically active natural products and synthetic compounds of medicinal interest (Gilchrist, 1998). As useful precursors to potentially bioactive pyrimidine derivatives, methylpyrimidine has attracted considerable attention for many years (Xue *et al.*, 1993). In recent years, Ru complexes of pyrimidine with an $-NH_2$ substituent have been synthesized (Zhu *et al.*, 2008). The title compound(I), was synthesized and we report herein its crystal structure.

The molecular structure of the title compound is shown in Fig. 1. There is one 2-Amino-4,6-dimethylpyrimidine cation and one dihydrogenphosphate anion in the asymmetric unit. All bond lengths are within the normal ranges (Allen *et al.*, 1987). In the crystal structure, cations and anions are linked by intermolecular O-H \cdots O and N-H \cdots O hydrogen bonds to form a two-dimensional network. Additional stabilization is provided by weak intermolecular C—H \cdots O interactions.

S2. Experimental

A mixture of guanidine hydrochloride (0.1 mol), acetyl acetone (0.2 mol), sodium carbonate (0.03 mol) and phosphoric acid (0.1 mol) was stirred with water (30 mL) for 5 h to afford the title compound (yield 78%). Single crystals suitable for X-ray measurements were obtained by recrystallization of the title compound from water at room temperature.

S3. Refinement

H atoms bonded to C atoms were fixed geometrically and included in a riding-model approximation with C—H = 0.93–0.96 Å and $U_{iso}(H)=1.2-1.5U_{eq}(C)$. H atoms bonded to N and O atoms were included in their 'as found' locations with refined isotropic displacement parameters,

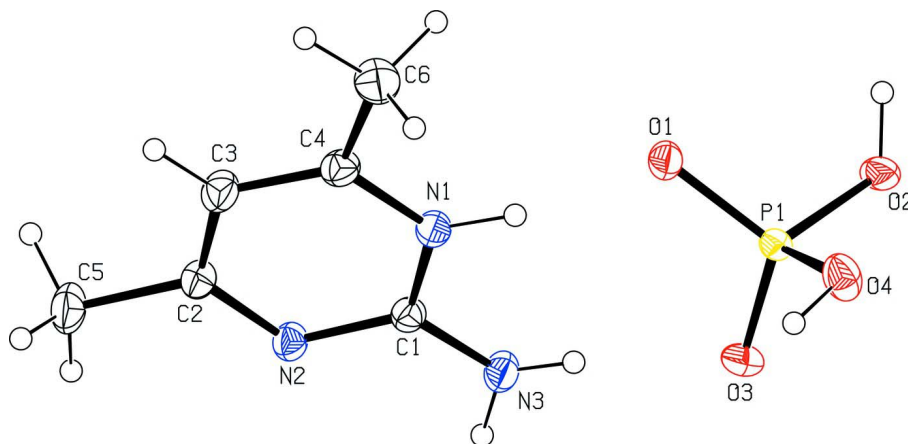


Figure 1

The molecular structure of the title compound showing 30% probability displacement ellipsoids and the atom-numbering scheme.

2-Amino-4,6-dimethylpyrimidinium dihydrogenphosphate

Crystal data

$C_6H_{10}N_3^+ \cdot H_2PO_4^-$

$M_r = 221.16$

Monoclinic, $P2_1/n$

Hall symbol: $-P 2_1n$

$a = 11.743 (2) \text{ \AA}$

$b = 4.8266 (10) \text{ \AA}$

$c = 16.940 (3) \text{ \AA}$

$\beta = 95.55 (3)^\circ$

$V = 955.6 (3) \text{ \AA}^3$

$Z = 4$

$F(000) = 464$

$D_x = 1.537 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1973 reflections

$\theta = 3.5\text{--}27.5^\circ$

$\mu = 0.28 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Block, colorless

$0.20 \times 0.15 \times 0.11 \text{ mm}$

Data collection

Bruker SMART CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

8743 measured reflections

2193 independent reflections

1973 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$

$\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 3.5^\circ$

$h = -15 \rightarrow 15$

$k = -6 \rightarrow 5$

$l = -21 \rightarrow 21$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.035$

$wR(F^2) = 0.108$

$S = 1.08$

2193 reflections

133 parameters

0 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0686P)^2 + 0.3094P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 0.40 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.41 \text{ e \AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
P1	0.09666 (3)	0.22149 (8)	0.74781 (2)	0.02327 (14)
O1	0.17203 (9)	0.3129 (2)	0.82063 (6)	0.0302 (3)
O2	0.01084 (9)	0.4557 (2)	0.71860 (7)	0.0337 (3)
H2A	0.0243	0.6265	0.7326	0.085 (9)*
O3	0.02931 (10)	-0.0362 (2)	0.76133 (7)	0.0339 (3)
N1	0.20023 (11)	0.0034 (3)	0.95145 (7)	0.0270 (3)
H1A	0.1936	0.0900	0.9021	0.052 (6)*
O4	0.16964 (11)	0.1887 (3)	0.67571 (7)	0.0381 (3)
H4A	0.2241	0.0508	0.6822	0.084 (9)*
N3	0.02982 (11)	-0.2279 (3)	0.91928 (8)	0.0322 (3)
H3B	-0.0198	-0.3692	0.9308	0.032 (4)*
H3A	0.0243	-0.1573	0.8748	0.044 (6)*
C1	0.12099 (12)	-0.1846 (3)	0.96966 (8)	0.0252 (3)
N2	0.13194 (11)	-0.3247 (3)	1.03924 (7)	0.0282 (3)
C4	0.29458 (13)	0.0550 (3)	1.00208 (9)	0.0300 (3)
C2	0.22244 (14)	-0.2691 (3)	1.09017 (9)	0.0294 (3)
C3	0.30667 (13)	-0.0793 (4)	1.07340 (9)	0.0341 (3)
H3D	0.3695	-0.0453	1.1099	0.041*
C6	0.37991 (16)	0.2522 (4)	0.97449 (12)	0.0417 (4)
H6A	0.3427	0.4240	0.9594	0.063*
H6B	0.4128	0.1748	0.9297	0.063*
H6C	0.4391	0.2851	1.0166	0.063*
C5	0.23093 (16)	-0.4213 (4)	1.16774 (9)	0.0429 (4)
H5A	0.1664	-0.5429	1.1690	0.064*
H5B	0.2315	-0.2905	1.2105	0.064*
H5C	0.3002	-0.5280	1.1734	0.064*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0252 (2)	0.0188 (2)	0.0253 (2)	0.00191 (12)	-0.00052 (14)	0.00042 (12)
O1	0.0312 (6)	0.0308 (6)	0.0274 (5)	-0.0050 (4)	-0.0039 (4)	0.0044 (4)
O2	0.0333 (6)	0.0214 (5)	0.0437 (6)	0.0054 (4)	-0.0103 (4)	-0.0035 (4)
O3	0.0404 (6)	0.0207 (5)	0.0400 (6)	-0.0048 (4)	0.0005 (5)	0.0003 (4)
N1	0.0281 (6)	0.0277 (6)	0.0250 (6)	-0.0035 (5)	0.0012 (4)	0.0012 (5)
O4	0.0470 (7)	0.0380 (7)	0.0305 (6)	0.0135 (5)	0.0099 (5)	0.0051 (5)

N3	0.0327 (7)	0.0363 (7)	0.0263 (7)	-0.0087 (5)	-0.0031 (5)	0.0042 (5)
C1	0.0265 (7)	0.0256 (7)	0.0236 (7)	-0.0009 (5)	0.0026 (5)	-0.0008 (5)
N2	0.0303 (6)	0.0310 (7)	0.0233 (6)	-0.0038 (5)	0.0020 (5)	0.0020 (5)
C4	0.0277 (7)	0.0296 (7)	0.0322 (7)	-0.0034 (6)	0.0008 (5)	-0.0019 (6)
C2	0.0327 (8)	0.0313 (8)	0.0239 (7)	0.0003 (6)	0.0011 (5)	-0.0007 (5)
C3	0.0310 (8)	0.0391 (9)	0.0308 (7)	-0.0049 (7)	-0.0040 (6)	0.0005 (6)
C6	0.0346 (9)	0.0423 (10)	0.0475 (10)	-0.0125 (7)	-0.0002 (7)	0.0068 (7)
C5	0.0479 (10)	0.0532 (11)	0.0261 (7)	-0.0066 (8)	-0.0032 (6)	0.0089 (7)

Geometric parameters (Å, °)

P1—O3	1.5033 (12)	N2—C2	1.330 (2)
P1—O1	1.5128 (12)	C4—C3	1.366 (2)
P1—O2	1.5626 (11)	C4—C6	1.490 (2)
P1—O4	1.5665 (12)	C2—C3	1.397 (2)
O2—H2A	0.8679	C2—C5	1.500 (2)
N1—C1	1.3566 (19)	C3—H3D	0.9300
N1—C4	1.3568 (19)	C6—H6A	0.9600
N1—H1A	0.9317	C6—H6B	0.9600
O4—H4A	0.9225	C6—H6C	0.9600
N3—C1	1.3195 (19)	C5—H5A	0.9600
N3—H3B	0.9302	C5—H5B	0.9600
N3—H3A	0.8243	C5—H5C	0.9600
C1—N2	1.3542 (19)		
O3—P1—O1	113.08 (6)	C3—C4—C6	124.41 (15)
O3—P1—O2	108.30 (7)	N2—C2—C3	122.50 (14)
O1—P1—O2	110.84 (7)	N2—C2—C5	116.78 (14)
O3—P1—O4	111.77 (7)	C3—C2—C5	120.72 (15)
O1—P1—O4	110.13 (7)	C4—C3—C2	118.42 (14)
O2—P1—O4	102.18 (7)	C4—C3—H3D	120.8
P1—O2—H2A	120.4	C2—C3—H3D	120.8
C1—N1—C4	121.00 (13)	C4—C6—H6A	109.5
C1—N1—H1A	120.3	C4—C6—H6B	109.5
C4—N1—H1A	118.5	H6A—C6—H6B	109.5
P1—O4—H4A	113.9	C4—C6—H6C	109.5
C1—N3—H3B	117.8	H6A—C6—H6C	109.5
C1—N3—H3A	120.9	H6B—C6—H6C	109.5
H3B—N3—H3A	119.9	C2—C5—H5A	109.5
N3—C1—N2	119.12 (14)	C2—C5—H5B	109.5
N3—C1—N1	119.36 (13)	H5A—C5—H5B	109.5
N2—C1—N1	121.51 (13)	C2—C5—H5C	109.5
C2—N2—C1	117.81 (13)	H5A—C5—H5C	109.5
N1—C4—C3	118.72 (14)	H5B—C5—H5C	109.5
N1—C4—C6	116.85 (14)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1A \cdots O1	0.93	1.75	2.667 (2)	168
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C3—H3D \cdots O2 ^{iv}	0.93	2.40	3.319 (2)	171

Symmetry codes: (i) $x, y+1, z$; (ii) $-x, -y-1, -z+2$; (iii) $-x+1/2, y-1/2, -z+3/2$; (iv) $x+1/2, -y+1/2, z+1/2$.