Crystal structure of 2-amino-3-nitro-pyridinium dihydrogenmonophosphate, $C_5H_8N_3O_6P$

S. Akriche and M. Rzaigui*

Faculté des Sciences de Bizerte, Laboratoire de Chimie des Matériaux, 7021 Zarzouna Bizerte, Tunisia

Received May 12, 2000, CCDC-No. 1267/460

Abstract

$C_5H_8N_3O_6P$, monoclinic, $P12_1/c1$ (No. 14), $a = 9.710(3) \text{ \AA}$, $b = 9.254(1) \text{ \AA}$, $c = 10.373(6) \text{ \AA}$, $\beta = 100.36(5)^\circ$, $V = 916.9 \text{ \AA}^3$, $Z = 4$, $R_{\text{gt}}(F) = 0.040$, $wR_{\text{ref}}(F) = 0.057$, $T = 296 \text{ K}$.

Source of material

The 2-amino-3-nitropyridine was dissolved in 20 cm^3 of an acidic solution containing an excess of H_3PO_4 ($d = 1.5$) heated to 373 K . The so obtained solution is immediately concentrated by evaporation in air-circulation for some hours then maintained for many days at room temperature till formation of prismatic yellow crystals.

Discussion

The structure of $(C_5H_6N_3O_2)H_2PO_4$ can be described as a stacking of thick layer centred by (b, c) plane. Inside one layer, the $H_2PO_4^-$ anions, connected by strong hydrogen bonds, are assembled to build infinite layers perpendicular to the a direction. The organic groups $(C_5H_6N_3O_2)^+$, are anchored onto two phosphoric layers through multiple hydrogen bonds to make up into two dimensions infinite network.

Table 1. Data collection and handling.

Crystal:	yellow prism, size $0.30 \times 0.30 \times 0.50 \text{ mm}$
Wavelength:	Ag $K\alpha$ radiation ($0.5608 \text{ \AA}$)
μ :	1.71 cm^{-1}
Diffractometer, scan mode:	CAD4, $\omega/2\theta$
$2\theta_{\text{max}}$:	59.9°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	7483, 4051
Criterion for I_{obs} , $N(hkl)_{\text{gi}}$:	$I_{\text{obs}} > 4 \sigma(I_{\text{obs}})$, 3760
$N(\text{param})_{\text{refined}}$:	168
Programs:	teXsan [1], DIRDIF92 ORIENT [2]

Table 2. Atomic coordinates and displacement parameters (in $\text{\AA}^2$).

Atom	Site	x	y	z	U_{iso}
H(1)	4e	0.755(1)	0.308(1)	0.903(1)	0.042(3)
H(2)	4e	0.604(1)	0.367(1)	1.048(1)	0.064(4)
H(3)	4e	0.547(1)	0.180(1)	1.184(1)	0.038(3)
H(4)	4e	0.841(1)	-0.138(1)	0.925(1)	0.075(4)
H(5)	4e	0.786(1)	-0.207(1)	1.034(1)	0.043(3)
H(6)	4e	0.824(1)	0.095(1)	0.874(1)	0.062(4)
H(7)	4e	0.985(1)	0.218(1)	1.221(1)	0.058(3)
H(8)	4e	1.110(2)	0.046(2)	1.513(2)	0.084(5)

* Correspondence author (e-mail: Mohamed.Rzaigui@fsb.rnu.tn)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
P(1)	4e	1.02892(2)	0.02214(2)	1.30687(2)	0.03621(7)	0.01849(6)	0.02186(6)	−0.00007(5)	0.01204(5)	−0.00063(5)
O(1)	4e	1.13906(6)	0.07549(6)	1.42371(5)	0.0457(3)	0.0362(3)	0.0287(2)	−0.0126(2)	0.0084(2)	−0.0019(2)
O(2)	4e	1.08088(6)	−0.10801(5)	1.24177(5)	0.0521(3)	0.0190(2)	0.0344(2)	0.0009(2)	0.0223(2)	−0.0021(2)
O(3)	4e	1.01591(7)	0.14413(6)	1.20022(5)	0.0591(3)	0.0224(2)	0.0284(2)	0.0077(2)	0.0189(2)	0.0047(2)
O(4)	4e	0.88969(6)	−0.00257(6)	1.34866(5)	0.0347(2)	0.0408(3)	0.0297(2)	0.0003(2)	0.0115(2)	0.0033(2)
O(5)	4e	0.65210(8)	−0.20782(7)	1.17964(7)	0.0721(4)	0.0359(3)	0.0527(3)	−0.0067(3)	0.0272(3)	0.0080(3)
O(6)	4e	0.54782(9)	−0.04831(9)	1.27655(8)	0.0840(4)	0.0641(5)	0.0740(4)	−0.0029(4)	0.0607(3)	0.0095(4)
N(1)	4e	0.79138(8)	−0.12989(8)	0.99031(7)	0.0575(4)	0.0330(3)	0.0398(3)	0.0103(3)	0.0240(3)	0.0064(2)
N(2)	4e	0.76704(7)	0.11084(7)	0.94129(6)	0.0354(3)	0.0315(3)	0.0274(2)	0.0003(2)	0.0122(2)	0.0034(2)
N(3)	4e	0.61529(7)	−0.08389(8)	1.19336(7)	0.0399(3)	0.0435(3)	0.0362(3)	−0.0100(3)	0.0168(2)	0.0033(2)
C(1)	4e	0.72031(8)	0.24532(8)	0.95468(7)	0.0432(4)	0.0292(3)	0.0338(3)	−0.0022(3)	0.0116(3)	0.0024(2)
C(2)	4e	0.63535(9)	0.27694(9)	1.04263(8)	0.0467(4)	0.0314(3)	0.0422(4)	0.0033(3)	0.0151(3)	−0.0023(3)
C(3)	4e	0.60150(8)	0.16488(9)	1.12001(7)	0.0341(3)	0.0390(3)	0.0343(3)	−0.0003(3)	0.0136(2)	−0.0049(3)
C(4)	4e	0.65261(7)	0.02786(8)	1.10793(6)	0.0291(3)	0.0331(3)	0.0266(2)	−0.0046(2)	0.0094(2)	0.0002(2)
C(5)	4e	0.73858(7)	−0.00378(7)	1.01410(6)	0.0305(3)	0.0305(3)	0.0253(2)	−0.0009(2)	0.0086(2)	0.0015(2)

References

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