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Pyridoxinium nitrate

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In the title compound, $C_8H_{12}NO_3^+\cdot NO_3^-$, the protonated pyridoxinium cations and the nitrate anions are arranged nearly parallel to each other. The aggregation of cations and anions through hydrogen bonds forms a sheet-like structure in parallel planes. An intramolecular hydrogen bond links the phenol OH and the $-CH_2OH$ group, characteristic feature of pyridoxine structures.

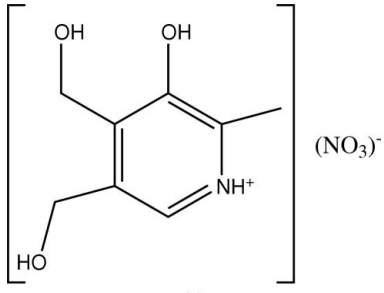
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.041
wR factor = 0.129
Data-to-parameter ratio = 12.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Pyridoxine (vitamin B6) is involved in the production of antibodies, which protect humans against bacterial diseases. Furthermore, the combination of pyridoxine with immunosuppressive drugs improves the efficiency of that therapy (Trakatellis *et al.*, 1992). The crystal structures of pyridoxinium chloride (Bacon & Plant, 1980), pyridoxamine monohydrochloride (Longo & Richardson, 1980), copper complexes of neutral pyridoxamine (Franklin & Richardson, 1980), pyridoxine (Longo *et al.*, 1982), *cis*-(oxalato-*O*,*O*′)-bis(pyridoxine-*N*)palladium, (II) (Dey *et al.*, 2003), and 6-dimethyl aminopyridoxine-α^4-(*t*-butyldimethylsilyl ether) (Culbertson *et al.*, 2003) are already known. In the present investigation, pyridoxine was reacted with nitric acid and the structure of the product, (I), is reported.



(I)

The asymmetric unit of (I) consists of a protonated pyridoxinium cation and a nitrate anion (Fig. 1). Both of these are planar with r.m.s. deviations 0.0942 and 0.0003 Å for the cation and anion, respectively. The presence of CH_2OH groups decreases the planarity of the cation overall, particularly in comparison with that of the pyridinium ring for which the r.m.s. deviation is only 0.0019 Å. Twisting of the CH_2OH groups is a characteristic feature of all pyridoxine complexes. The deviations of atoms O42 and O52 from the plane of the ring are 0.354 (4) and 0.273 (4) Å, respectively. With the exception of pyridoxinium chloride, pyridoxine is generally found in the unprotonated or zwitterionic form (Cambridge Structural Database; Version 5.26; Allen, 2002). In the zwit-

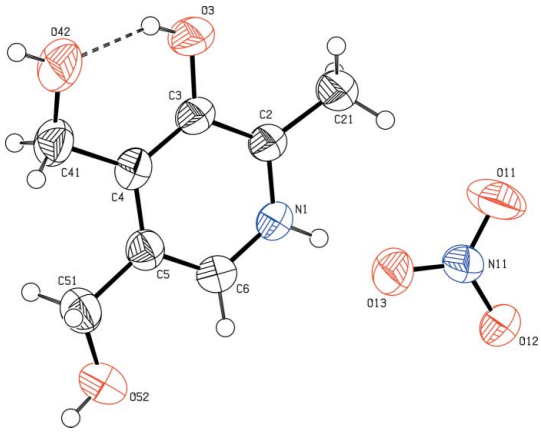


Figure 1
The structure of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids.

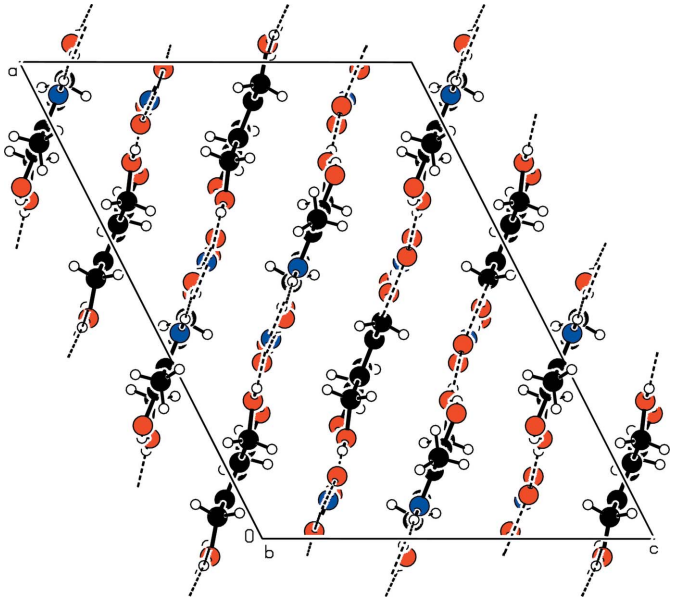


Figure 2
Packing diagram of the molecules viewed down the *b* axis. H atoms have been omitted unless they are involved in hydrogen bonds (dashed lines).

terionic form, the phenol group is deprotonated and the pyridine N atom is protonated, a form generally found in metal–pyridoxine complexes such as bis(μ_2-pyridoxinato)diaquatetrachlorodiiron(III) (Sabirov *et al.*, 1993). Here pyridoxine is observed in the protonated form, as evidenced by the C3—O3 and C—N1 bond distances (Table 1).

The cations and anions are oriented nearly parallel to each other, the angle between the pyridinium and nitrate planes being only 2.52 (1)°. Intramolecular hydrogen bonds form between the phenol OH and the adjacent –CH_2OH group, generating an *S*(6) hydrogen-bonded graph-set motif (Etter *et al.*, 1990). This is a characteristic feature found in all pyridoxine complexes. Cations and anions are arranged in column-like structures and linked through intermolecular hydrogen bonds (Table 2). This leads to aggregation of the cations and anions, forming a sheet-like structure (Fig. 3) parallel to the ($\bar{8}08$) and ($80\bar{8}$) planes.

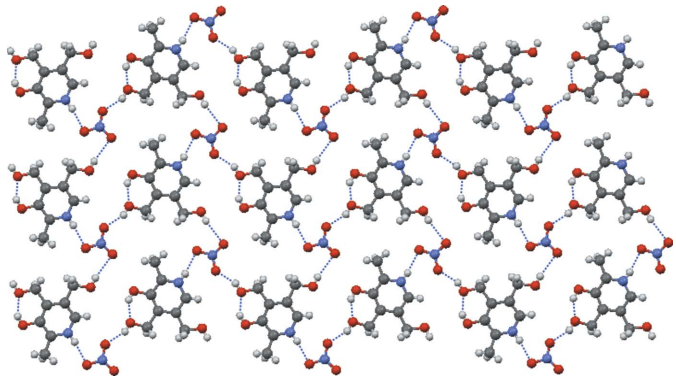


Figure 3
The sheet-like structure of cations and anions generated by hydrogen bonds (shown as dashed lines).

Experimental

Compound (I) was crystallized from a liquid mixture containing pyridoxine and nitric acid, in the stoichiometric ratio 1:1, at room temperature by the technique of slow evaporation.

Crystal data

$C_8H_{12}NO_3^+\cdot NO_3^-$
M_r = 232.20
Monoclinic, *C*2/*c*
a = 18.6810 (11) Å
b = 9.0430 (7) Å
c = 13.6950 (9) Å
β = 117.046 (9)°
V = 2060.5 (2) Å^3
Z = 8
D_x = 1.497 Mg m^{-3}
D_m = 1.49 Mg m^{-3}
D_m measured by flotation in a mixture of xylene and bromoform
Mo *K*α radiation
μ = 0.13 mm^{-1}
T = 293 (2) K
Block, colourless
0.24 × 0.21 × 0.19 mm

Data collection

Nonius MACH3 sealed-tube diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.958, T_{max} = 0.996
2125 measured reflections
1813 independent reflections
1385 reflections with $I > 2\sigma(I)$
R_{int} = 0.014
θ_{max} = 25.0°
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.129
S = 1.09
1813 reflections
150 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.061P)^2 + 1.481P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0009 (3)

Table 1
Selected geometric parameters (Å, °).

N1—C6	1.340 (3)	C3—O3	1.354 (2)
N1—C2	1.343 (2)		
C6—N1—C2	124.28 (17)	O52—C51—C5	108.90 (18)
O42—C41—C4	110.16 (19)		
C3—C4—C41—O42	−16.8 (3)	C6—C5—C51—O52	−11.6 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O42	0.82	1.82	2.535 (2)	146
N1—H1···O12^{i}	0.86	2.02	2.869 (2)	170
O42—H42···O13^{ii}	0.82	1.86	2.626 (2)	155
O52—H52···O11^{iii}	0.82	2.27	3.077 (3)	170

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

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93, 0.96 and 0.97 Å, O—H = 0.82 Å, and N—H = 0.86 Å and U_{iso}(H) = 1.2–1.5U_{eq}(parent atom).

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXTL/PC* (Bruker, 2000); program(s) used to refine structure: *SHELXTL/PC*; molecular graphics: *MERCURY* (Macrae *et al.*, 2006) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL/PC*.

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References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Bacon, G. E. & Plant, J. S. (1980). *Acta Cryst.* B**36**, 1130–1136.
Bruker (2000). *SHELXTL/PC*. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Culbertson, S. M., Enright, G. D. & Ingold, K. U. (2003). *Org. Lett.* **5**, 2659–2662.
Dey, S., Banerjee, P., Gangopadhyay, S. & Vijtisek, P. (2003). *Transition Met. Chem.* **28**, 765–771.
Enraf–Nonius (1994). *CAD-4 EXPRESS*. Version 5.1/1.2. Enraf–Nonius, Delft, The Netherlands.
Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* B**46**, 256–262.
Franklin, K. J. & Richardson, M. F. (1980). *Inorg. Chem.* **19**, 2107–2113.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Longo, J., Franklin, K. J. & Richardson, M. F. (1982). *Acta Cryst.* B**38**, 2721–2724.
Longo, J. & Richardson, M. F. (1980). *Acta Cryst.* B**36**, 2456–2458.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sabirov, V. K., Porai-Koshitz, M. A. & Struchkov, Yu. T. (1993). *Acta Cryst.* C**49**, 1611–1614.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Trakatellis, A., Dimitriadou, A., Exindari, M., Scountzou, J., Koliakos, G., Christodoulou, D., Malissiovas, N., Antoniadis, A. & Polyzoni, T. (1992). *Postgrad. Med. J.* **68**, S70–77.