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2-(Indol-3-yl)ethylammonium dihydrogenphosphate

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.104
Data-to-parameter ratio = 13.2

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

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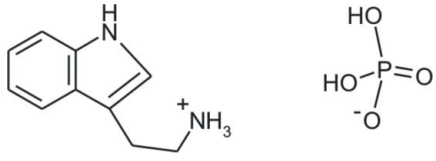
Ions of the title compound, tryptaminium dihydrogenphosphate, $C_{10}H_{13}N_2^+\cdot H_2PO_4^-$, are connected by intermolecular N—H···O═P [N···O = 3.028 (3) Å], NH_3···O═P [N···O = 2.720 (3) and 2.950 (3) Å], NH_3···OH—P [N···O = 2.993 (2) Å] and P—OH···O═P [O···O = 2.562 (2) and 2.619 (2) Å] hydrogen bonds between the indole –NH and ammonium —NH_3 groups of the cation and the dihydrogenphosphate anions into an infinite three-dimensional network.

Comment

Phosphate is an essential nutrient for all organisms used in the biosynthesis of diverse cellular components, such as nucleic acids, proteins, lipids, and sugars. It is essential for organisms to have evolved regulatory mechanisms for the acquisition, storage and release of this amino acid (Torriani-Gorini *et al.*, 1994). Present evidence indicates that regulation of the overall renal tubular phosphate transport by dietary, hormonal or metabolic factors occurs at the level of the proximal tubular brush border membrane of the Na/phosphate co-transport system (Dousa, 1996; Kempson, 1996; Lotscher *et al.*, 1996; Levi *et al.*, 1996). Phosphate is also a structural fragment in a series of important biologically active enzymes. The role of intestinal phosphate absorption in providing phosphate to buffer protons and to compensate for loss from bone during metabolic acidosis has not yet been clarified (Christian *et al.*, 1999). The risk factors that contribute to the higher prevalence of atherosclerotic lesions in chronic renal failure include dyslipidemia, hyperhomocysteinemia, and hypertension. In addition, hyperphosphatemia and increased calcium phosphate production are important contributors to vascular calcifications in patients with uremia (Block *et al.*, 1998).



(I)

There are seven known families of serotonin receptors, which are tryptamine derivatives. All of them are neurotransmitters. Hallucinogens all have a high affinity for certain serotonin receptor subtypes and the relative hallucinogenic potencies of various drugs can be gauged by their affinities for these receptors (Glennon *et al.*, 1984; Nichols *et al.*, 2001; Johnson *et al.*, 1987; Krebs-Thomson *et al.*, 1998). The structures of many hallucinogens are similar to serotonin and have a tryptamine core.

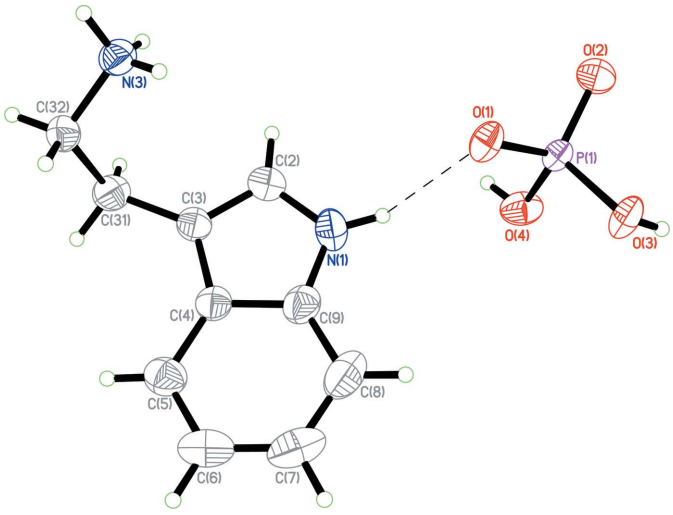


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. The dashed line indicated a hydrogen bond.

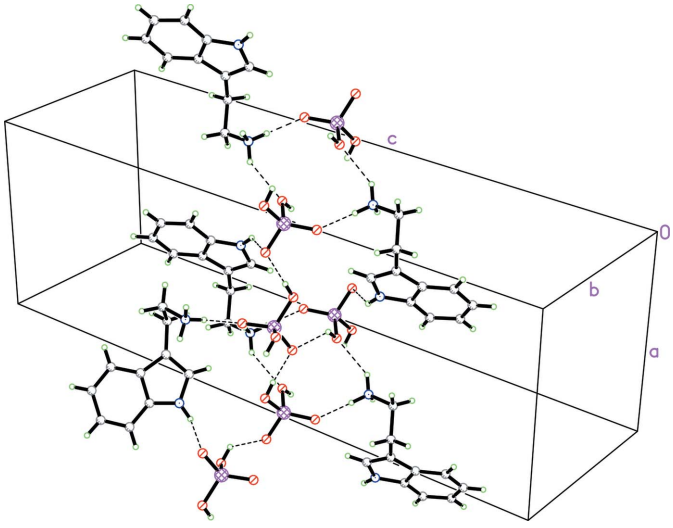


Figure 2
Linkage of the ions of (I) into a three-dimensional network through intermolecular hydrogen bonds (dashed lines).

The present investigation on the synthesis and structural characterization of tryptaminium dihydrogenphosphate, (I), is part of our continuing studies on the structure of biologically active amines (Kolev, Yancheva *et al.*, 2006; Kolev, Spiteller *et al.*, 2006) and was prompted by the biological significance of the component ions. It is believed that the dihydrogenphosphates of bioactive compounds or drugs may be more readily assimilated by living organisms.

The asymmetric unit of (I) is depicted in Fig. 1, and a view along the *b* axis is given in Fig. 2. A polymeric network is formed by the tryptaminium cations and dihydrogenphosphate anions by means of N—H···O and O—H···O hydrogen bonds (Table 1) with participation of the indole NH and the protonated primary amino group as well as the dihydrogenphosphate P═O and P—OH functions. As can be seen in Fig. 2, the dihydrogenphosphate anions are linked into infinite hydrogen-bonded sheets through a network of O—H···O hydrogen bonds. These are separated from one another by a hydrophobic region containing tryptaminium cations which link a total of four dihydrogenphosphate anions within a sheet through an N—H···O hydrogen bond from the indole NH function and N—H···O hydrogen bonds from the ammonium group (Table 1).

Experimental

Tryptamine was received from Fluka (Switzerland) and recrystallized from cyclohexane, giving a yellowish polycrystalline sample. Compound (I) was synthesized in high yield by mixing 20 ml of a methanolic solution of tryptamine (467 mg, 6 mmol) with an equimolar amount of 50% phosphoric acid. The reaction mixture was stirred and heated for 8 h at 333 K and the reaction monitored by thin layer chromatography. The resulting solution was filtered and the filtrate set aside to precipitate (I) after 24 h. Suitable crystals for X-ray analysis were grown from a water–ethanol (1:1) solution over a period of three weeks. The product was characterized by elemental analysis and mass spectrometry as well as IR, Raman and NMR spectroscopy. The IR spectrum of (I) (KBr pellet) exhibits the characteristic frequencies (Ivanova, 2006) for an indole ring with ν NH at 3402 cm^{-1}, the ring in-plane modes at 1602 and 1554 cm^{-1}, as well as the intense peak at 744 cm^{-1} belonging to the out-of-plane vibration. The broad maximum at 3200–2900 cm^{-1} corresponds to asymmetric and symmetric stretching frequencies of the protonated primary amino group overlapped with ν OH of the hydrogen-bonded dihydrogenphosphate anions. ν P═O and ν PO(H) are assigned to the intense peaks at 1273 and 1067 cm^{-1}, respectively. A full theoretical vibrational analysis and experimental assignment of (I) by means of solid-state linear polarized IR spectroscopy is now in progress and will be published at a later date.

Crystal data

$C_{10}H_{13}N_2^+\cdot H_2O_4P^-$
M_r = 258.21
Orthorhombic, *Pbca*
a = 9.4345 (13) Å
b = 8.607 (3) Å
c = 28.973 (5) Å
V = 2352.6 (11) Å^3
Z = 8
D_x = 1.458 Mg m^{-3}
Mo *K*α radiation
μ = 0.24 mm^{-1}
T = 294 (2) K
Prism, colourless
0.49 × 0.40 × 0.34 mm

Data collection

Siemens P4 four-circle diffractometer
ω scans
Absorption correction: ψ scan (*XPREP* in *SHELXTL*; Sheldrick, 1995)
T_{min} = 0.872, T_{max} = 0.921
2747 measured reflections
2064 independent reflections
1594 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 25.0°
3 standard reflections
every 97 reflections
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.104
S = 1.04
2064 reflections
156 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0524P)^2 + 0.6354P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0036 (9)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.86	2.35	3.028 (3)	136
N3—H31···O4^{i}	0.89	2.24	2.993 (2)	142
N3—H32···O2^{ii}	0.89	1.83	2.720 (3)	175
N3—H33···O2^{iii}	0.89	2.09	2.950 (3)	163
O3—H3···O1^{iv}	0.82	1.80	2.619 (2)	172
O4—H4···O1^{v}	0.82	1.99	2.562 (2)	126

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

The H atoms were constrained to idealized positions and refined using a riding model, with C—H = 0.97 Å for the methylene groups and 0.93 Å for the aromatic H. N—H distances of 0.89 and 0.86 Å were employed for the indole and ammonium groups, respectively, and O—H distances of 0.82 Å were used for the dihydrogenphosphate anions. Isotropic displacement parameters U_{iso}(H) = 1.2U_{iso}(C), 1.2U_{iso}(indole N), 1.5U_{iso}(ammonium N) and 1.5U_{iso}(O) were employed for the respective H atoms.

Data collection: Siemens *R3m/V* (Siemens, 1989); cell refinement: Siemens *R3m/V*; data reduction: *XDISK* (Siemens, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1995); software used to prepare material for publication: *SHELXL97*.

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References

Block, G. A., Hulbert-Shearon, T. E., Levin, N. W. & Port, F. K. (1998). *Am. J. Kidney Dis.* **31**, 607–617.
Christian, R. C. & Fitzpatrick, L. A. (1999). *Curr. Opin. Nephrol. Hypertens.* **8**, 443–448.
Dousa, T. (1996). *Kidney Int.* **49**, 997–1004.
Glennon, R. A., Titeler, M. & McKenney, J. D. (1984). *Life Sci.* **35**, 2505–2511.
Ivanova, B. B. (2006). *Spectrochim. Acta Part A*, **64**, 931–938.
Johnson, M. P., Hoffman, A. J., Nichols, D. E. & Mathis, C. A. (1987). *Neuropharmacology*, **26**, 1803–1806.
Kempson, S. (1996). *Kidney Int.* **49**, 1005–1009.
Kolev, T., Cherneva, E., Spiteller, M., Sheldrick, W. S. & Mayer-Figge, H. (2006). *Acta Cryst.* E**62**, o1390–o1392.
Kolev, T., Yancheva, D., Spiteller, M., Sheldrick, W. S. & Mayer-Figge, H. (2006). *Acta Cryst.* E**62**, o463–o465.
Krebs-Thomson, K., Paulus, M. P. & Geyer, M. A. (1998). *Neuropsychopharmacology*, **18**, 339–351.
Levi, M., Kempson, S., Lotscher, M., Biber, J. & Murer, H. (1996). *J. Membr. Biol.* **154**, 1–9.
Lotscher, M., Wilson, P., Nguyen, S., Kaissling, B., Biber, J., Murer, H. & Levi, M. (1996). *Kidney Int.* **49**, 1012–1018.
Nichols, C. D. & Sanders-Bush, E. (2001). *Heffter Rev. Psychedel. Res.* **2**, 73–79.
Sheldrick, G. M. (1995). *SHELXTL*. Release 5.03. Siemens Analytical X-ray Instruments Inc., Madison, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Siemens (1989). *R3m/V Crystallographic-Systems User's Guide*. Version 3.2. Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.
Torriani-Gorini, A., Silver, S. & Yagil, E. (1994). *Phosphate in Microorganisms: Cellular and Molecular Biology*. Washington, DC: American Society for Microbiology.