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Zoledronic acid: monoclinic and triclinic polymorphs from powder diffraction data

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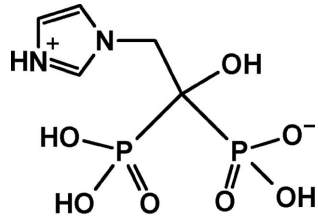
The crystal structures of the monoclinic and triclinic polymorphs of zoledronic acid, $C_5H_{10}N_2O_7P_2$, have been established from laboratory powder X-ray diffraction data. The molecules in both polymorphs are described as zwitterions, namely 1-(2-hydroxy-2-phosphonato-2-phosphonoethyl)-1*H*-imidazol-3-ium. Strong intermolecular hydrogen bonds (with donor–acceptor distances of 2.60 Å or less) link the molecules into layers, parallel to the (100) plane in the monoclinic polymorph and to the $(1\bar{1}0)$ plane in the triclinic polymorph. The phosphonic acid groups form the inner side of each layer, while the imidazolium groups lie to the outside of the layer, protruding in opposite directions. In both polymorphs, layers related by translation along [100] interact through weak hydrogen bonds (with donor–acceptor distances greater than 2.70 Å), forming three-dimensional layered structures. In the monoclinic polymorph, there are hydrogen-bonded centrosymmetric dimers linked by four strong O—H···O hydrogen bonds, which are not present in the triclinic polymorph.

Comment

Zoledronic acid belongs to the class of bisphosphonic acids, which are excellent therapeutic agents for the treatment of a number of diseases characterized by abnormal calcium metabolism. In particular, zoledronic acid acts as a bone 'shield' incorporated into the skeleton, attaining therapeutic concentrations and thus inhibiting bone resorption by cellular effects on osteoclasts. Zoledronic acid has also been shown to be effective in the treatment of early-stage breast cancer (Gnant, 2012) and castration-resistant prostate cancer (Marech *et al.*, 2012). Several polymorphs of zoledronic acid and zoledronate sodium salts and their hydrates have been described by Aronhime & Lifshitz-Liron (2009). However, a search for the crystal structure of zoledronic acid in the Cambridge Structural Database (CSD, Version 5.33 with updates; Allen, 2002) gave no hits. Herewith, we present the crystal structures of its monoclinic, (I*M*), and triclinic, (I*T*), polymorphs determined from laboratory X-ray powder diffraction data.



(I*M*)/(I*T*)

The molecular conformations in (I*M*) and (I*T*) are closely comparable (Fig. 1), differing only in the opposite orientation of the imidazole ring. Although the laboratory powder pattern does not allow localization of the O- and N-bound H atoms reliably, one can estimate their most probable positions based on analysis of short intermolecular contacts. Particularly, in (I*M*) and (I*T*), intermolecular H···H distances not shorter than 2 Å can be attained only by assuming that the molecules in both forms are zwitterions, namely 1-(2-hydroxy-2-phosphonato-2-phosphonoethyl)-1*H*-imidazol-3-ium. Thus, all H atoms were geometrically positioned to form zwitterions, and we discuss the hydrogen-bonding patterns in (I*M*) and (I*T*) (Tables 1 and 2) on this basis.

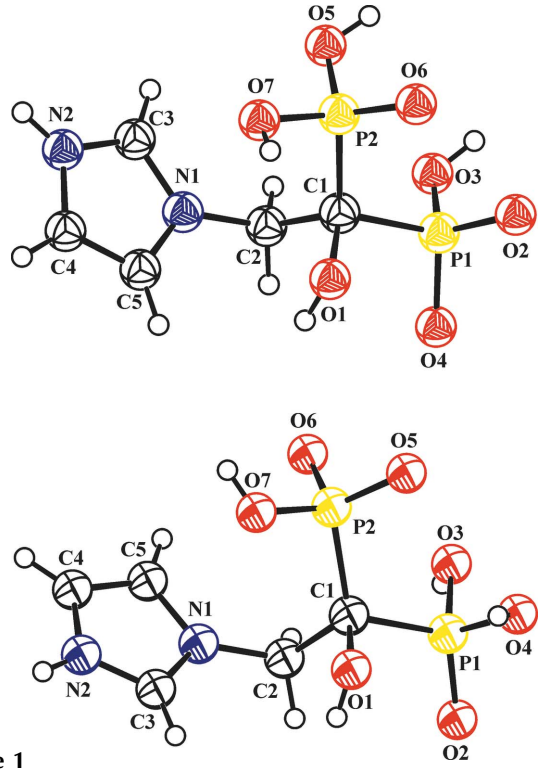


Figure 1
The molecular structure of the zoledronate zwitterion in polymorph (I*M*) (top) and polymorph (I*T*) (bottom), showing the atomic numbering. Displacement spheres are drawn at the 50% probability level.

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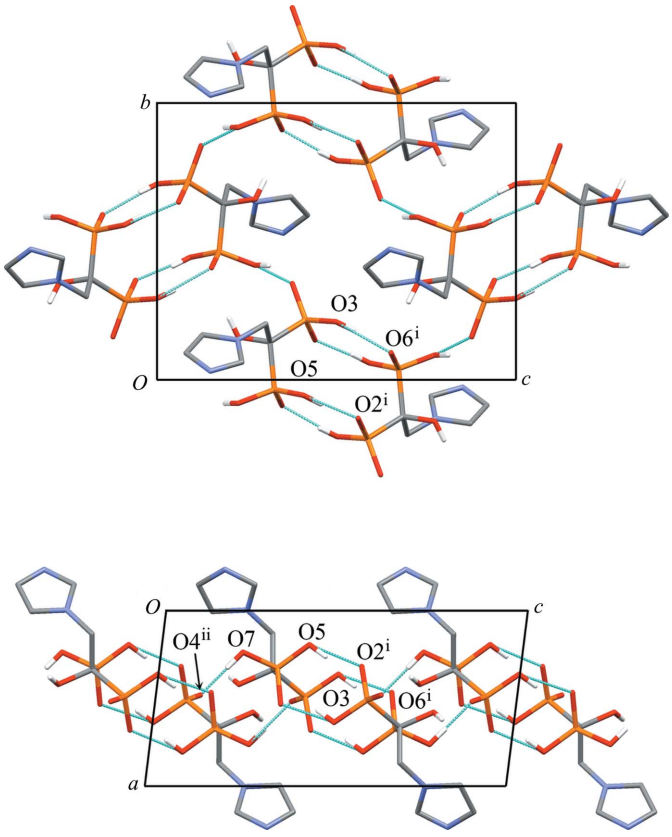

Figure 2
The hydrogen-bonded layer in (I*M*), viewed along [100] (top) and [010] (bottom). Thin lines (blue in the electronic version of the paper) denote strong O—H···O hydrogen bonds. The centrosymmetric dimers discussed in the *Comment* are visible in the top figure. H atoms not involved in hydrogen bonding have been omitted. Symmetry codes are as in Table 1.

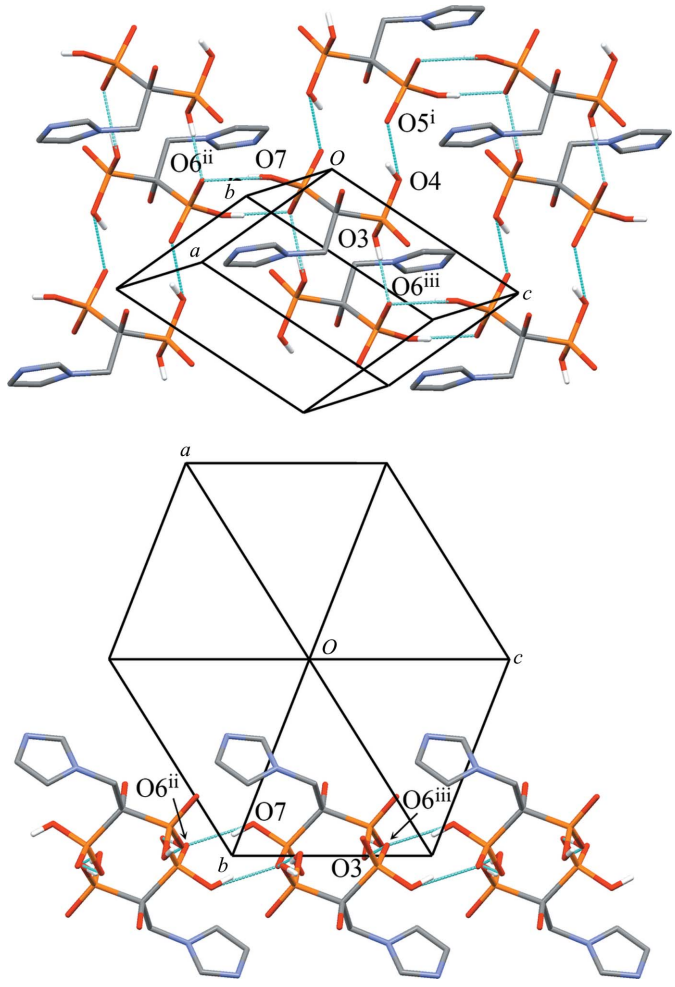

Figure 3
The hydrogen-bonded layer in (I*T*), viewed approximately along the normal to the layer (top) and along [111] (bottom). Thin lines (blue in the electronic version of the paper) denote strong O—H···O hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted. Symmetry codes are as in Table 2.

Following the idea of strong and weak hydrogen bonds (Desiraju & Steiner, 1999), we define here a strong hydrogen bond as an interaction with a donor–acceptor distance ($D\cdots A$ in Tables 1 and 2) of 2.60 Å or less. For a weak hydrogen bond, this distance is greater than 2.70 Å. Using these definitions, we can describe the general features of the hydrogen-bonding patterns that are common for (I*M*) and (I*T*). In both crystal structures, strong intermolecular hydrogen bonds (O—H···O) are generated by the phosphonic acid groups. They link the molecules into layers in such a way that the phosphonic acid groups form the central part of each layer, while the imidazolium groups are on the outside of the layer, protruding in opposite directions. These layers are parallel to the (100) plane in (I*M*) (Fig. 2) and the $(1\bar{1}0)$ plane in (I*T*) (Fig. 3). Layers related by translation along [100] interact through weak N—H···O and O—H···O hydrogen bonds in (I*M*) (Table 1) and through weak N—H···O hydrogen bonds only in (I*T*) (Table 2) to form three-dimensional layered structures.

For both forms, the main difference in the hydrogen-bonding motifs is found within the layers. In the layers of (I*M*), centrosymmetric dimers linked by four strong O—H···O hydrogen bonds are observed (Fig. 2). These dimers are not present in (I*T*) (Fig. 3). The CSD contains seven single-crystal structures of bisphosphonates with analogous dimers linked by four strong O—H···O hydrogen bonds with O···O distances less than 2.60 Å, namely, 10-{[(2,2-bisphosphonoethyl)hydroxyphosphoryl]methyl}-1,4,7,10-tetraazacyclododecane-1,4,7-triacetic acid hydrate (Vitha *et al.*, 2009), 1-hydroxy-1-phosphono-3-(1-piperidinio)propyl-1-phosphonate (Fernández & Vega, 2003), pyridinium trihydrogen benzyldiphosphonate and *p*-xylylenediammonium pentahydrogen 1,4-phenylenebis(methylidyne)tetraphosphonate sesquihydrate (Plabst *et al.*, 2009), bis[tris(1,10-phenanthroline-κ^2N,N')nickel(II)] bis[1-hydroxyethane-1-(phosphonic acid)-1-phosphonate] bis(1-hydroxyethane-1,1-diphosphonate) tetrahydrate (Sergienko *et al.*, 2000), 2,2′-bipyridinium hydrogen 1-aminopropane-1,1,3-triphosphonate dihydrate (Wu *et al.*, 2007), and {phosphono[(pyridin-1-ium-3-yl)amino]methyl}-phosphonate monohydrate (Matczak-Jon & Ślepokura, 2011). In spite of the presence of such strongly bonded dimers in (I*M*), its packing with ρ = 1.80 Mg m^{-3} is less dense than the packing of (I*T*) with ρ = 1.90 Mg m^{-3}.

Table 1
Hydrogen-bond geometry (Å, °) for polymorph (I*M*).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O6^{i}	0.82	1.71	2.481 (11)	156
O5—H5···O2^{i}	0.82	1.57	2.359 (11)	160
O7—H7···O4^{ii}	0.82	1.67	2.436 (10)	156
N2—H2···O4^{iii}	0.86	1.88	2.740 (11)	172
O1—H1···O6^{iv}	0.82	2.19	2.899 (11)	145

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

Table 2
Hydrogen-bond geometry (Å, °) for polymorph (I*T*).

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O5^{i}	0.82	1.69	2.406 (9)	144
O7—H7···O6^{ii}	0.82	1.80	2.604 (10)	166
O3—H3···O6^{iii}	0.82	1.76	2.559 (10)	166
O1—H1···O2^{iv}	0.82	1.87	2.682 (12)	172
N2—H2···O5^{v}	0.86	2.02	2.877 (13)	171

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

Experimental

Zoledronic acid monosodium salt was prepared according to a known procedure (Kieczykowski *et al.*, 1995). The crude product (30 g) was suspended in distilled water (450 ml) and the pH was adjusted to 1.5 with concentrated HCl. The suspension was heated to 363 K with stirring and complete dissolution occurred after 15 min at this temperature. The solution was then cooled to 283 K with stirring and a precipitate of zoledronic acid formed slowly. After overnight incubation at 278 K, the precipitate was filtered off, washed with methanol and placed in a flask fitted with a Dean–Stark trap. To obtain (I*M*), benzene (80 ml) was added and the reaction mixture was boiled for 2 h. To obtain (I*T*), toluene (80 ml) was added and the reaction mixture was boiled for 4 h (isolation of water ended after 1 h). The product was cooled, filtered and dried *in vacuo* (1 mbar; 1 bar = 100 000 Pa) at 323 K. For (I*M*), 11.2 g of zoledronic acid was recovered as a white crystalline powder. For (I*T*), 10.5 g of zoledronic acid was recovered as a white crystalline powder.

Polymorph (I*M*)

Crystal data

$C_5H_{10}N_2O_7P_2$
M_r = 272.09
Monoclinic, $P2_1/c$
a = 6.8162 (12) Å
b = 10.6307 (11) Å
c = 13.9240 (14) Å
β = 96.954 (18)°
V = 1001.5 (2) Å^3
Z = 4
Cu $K\alpha$ radiation, λ = 1.5418 Å
T = 298 K
Flat sheet, 15 × 1 mm

Data collection

PANanalytical EMPYREAN diffractometer
Specimen mounting: thin layer on the non-diffracting silicon plate
Data collection mode: reflection
Scan method: continuous
$2\theta_{min}$ = 7.009°, $2\theta_{max}$ = 79.973°, $2\theta_{step}$ = 0.017°

Refinement

R_p = 0.033
R_{wp} = 0.048
R_{exp} = 0.030
R_{Bragg} = 0.061
χ^2 = 2.570
4293 data points
121 parameters
43 restraints
H-atom parameters not refined

Polymorph (I*T*)

Crystal data

$C_5H_{10}N_2O_7P_2$
M_r = 272.09
Triclinic, $P\overline{1}$
a = 8.4217 (13) Å
b = 8.6039 (11) Å
c = 8.1818 (12) Å
α = 92.778 (16)°
β = 112.415 (17)°
γ = 116.072 (19)°
V = 475.33 (12) Å^3
Z = 2
Cu $K\alpha_1$ radiation, λ = 1.5406 Å
μ = 4.50 mm^{-1}
T = 298 K
Flat sheet, 15 × 1 mm

Data collection

Huber G670 Guinier camera diffractometer
Specimen mounting: thin layer in the specimen holder of the camera
Data collection mode: transmission
Scan method: continuous
$2\theta_{min}$ = 7.00°, $2\theta_{max}$ = 80.00°, $2\theta_{step}$ = 0.01°

Refinement

R_p = 0.026
R_{wp} = 0.033
R_{exp} = 0.015
R_{Bragg} = 0.050
χ^2 = 4.550
7301 data points
102 parameters
43 restraints
H-atom parameters not refined

X-ray powder diffraction data for (I*M*) were collected using a Panalytical EMPYREAN instrument with a linear X'celerator detector using nonmonochromated Cu $K\alpha$ radiation. Data for (I*T*) were collected using a Huber G670 Guinier camera with an imaging-plate detector using monochromated Cu $K\alpha_1$. The latter instrument was used for (I*T*) in order to minimize strong texture effects observed in the pattern measured with the EMPYREAN instrument. The unit-cell dimensions were determined using three indexing programs: *TREOR90* (Werner *et al.*, 1985), *ITO* (Visser, 1969) and *AUTOX* (Zlokazov, 1992, 1995). Based on systematic extinctions, the space group for (I*M*) was determined as $P2_1/c$, whereas the space group of (I*T*) was assumed to be $P\overline{1}$. The unit-cell parameters and space groups were further tested using a Pawley fit (Pawley, 1981) and confirmed by the successful crystal structure solution and refinement. The powder pattern of (I*M*) contains three weak peaks (d spacings = 4.886, 3.552 and 3.345 Å) that are assumed to arise from another polymorphic form of zoledronic acid.

The structures were solved using a simulated annealing technique (Zhukov *et al.*, 2001). The geometry of the anion (without H atoms) from the crystal structure of cytosimium zoledronate trihydrate (Sridhar & Ravikumar, 2011) was used as the initial molecular model. In the simulated annealing runs, six external and four internal degrees of freedom were varied.

The solutions found were fitted using the program *MRIA* (Zlokazov & Chernyshev, 1992) in the bond-restrained Rietveld refinement using a split-type pseudo-Voigt peak-profile function (Toraya, 1986). In the refinement of (I*M*), anisotropic line broadening was taken into account with the use of nine variables (Popa, 1998), and symmetrized harmonics expansion up to the sixth order (Ahtee *et al.*, 1989; Järvinen, 1993) was used for correction of the texture effect (the minimum and maximum texture multipliers for the calculated intensities were 0.61 and 1.22, respectively). In the refinement of (I*T*), the March–Dollase (Dollase, 1986) formalism was used for correction of preferred orientation in the $[01\overline{1}]$ direction (the minimum and maximum texture multipliers for the calculated intensities were 0.96 and 1.01, respectively). Restraints were applied to the intramolecular bond lengths and contacts (<2.8 Å). The geometric parameters for

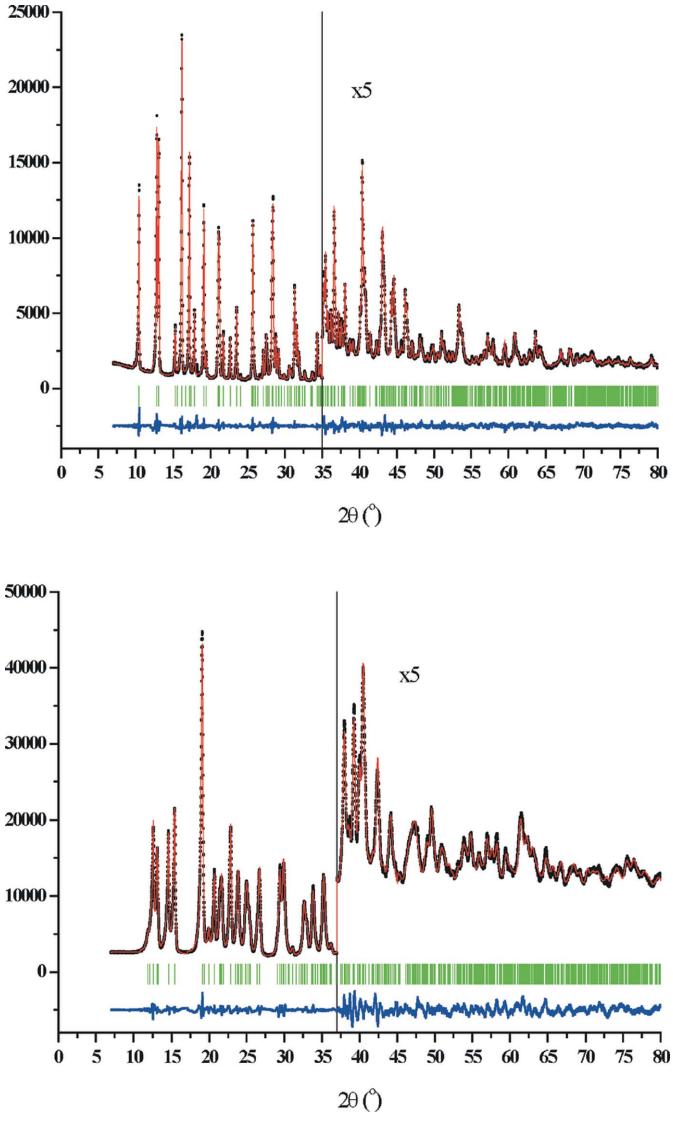

Figure 4
The final Rietveld plots for (I*M*) (top) and (I*T*) (bottom). The experimental diffraction profiles are indicated by black dots. The calculated diffraction profiles are shown as the upper solid lines (red in the electronic version of the paper), the difference profiles are shown as the bottom solid lines (blue) and the vertical bars (green) correspond to the positions of the Bragg peaks.

the restraints were taken from the crystal structure of zoledronic acid trihydrate (Ruscica *et al.*, 2010), and the strength of the restraints was a function of interatomic separation and corresponded to an r.m.s. deviation of 0.02 Å for intramolecular bond lengths. Additional restraints were applied to the planarity of the imidazole ring with attached atom C2, with the maximum allowed deviation from the mean plane being 0.02 Å. All non-H atoms were refined isotropically. H atoms were positioned geometrically (C—H = 0.93 or 0.97 Å, O—H = 0.82 Å and N—H = 0.86 Å) and not refined. The diffraction profiles for both compounds after the final bond-restrained Rietveld refinements are shown in Fig. 4.

Data collection: *DataCollector* (PANalytical, 2010) for (I*M*); *Software for G670 Imaging-Plate Guinier Camera* (Huber, 2002) for (I*T*). For both polymorphs, cell refinement: *MRIA* (Zlokazov & Chernyshev, 1992). Data reduction: *DataCollector* for (I*M*); *Software for G670 Imaging-Plate Guinier Camera* for (I*T*). For both polymorphs, structure solution: simulated annealing (Zhukov *et al.*, 2001); program(s) used to refine structure: *MRIA*; molecular graphics: *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *MRIA* and *SHELXL97* (Sheldrick, 2008).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BI3053). Services for accessing these data are described at the back of the journal.

References

Ahtee, M., Nurmela, M., Suortti, P. & Järvinen, M. (1989). *J. Appl. Cryst.* **22**, 261–268.
Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Aronhime, J. & Lifshitz-Liron, R. (2009). US Patent No. 7582768 B2.
Desiraju, G. R. & Steiner, T. (1999). In *The Weak Hydrogen Bond in Structural Chemistry and Biology*. Oxford University Press.
Dollase, W. A. (1986). *J. Appl. Cryst.* **19**, 267–272.
Fernández, D. & Vega, D. (2003). *Acta Cryst.* C**59**, o661–o663.
Gnant, M. (2012). *Curr. Oncol. Rep.* **14**, 35–43.
Huber (2002). *Software for G670 Imaging Plate Guinier Camera*. Huber Diffraktionstechnik GmbH, Rimsting, Germany.
Järvinen, M. (1993). *J. Appl. Cryst.* **26**, 525–531.
Kieczykowski, G. R., Jobson, R. B., Melilo, D. G., Reinhold, D. F., Grenda, V. J. & Shinkai, I. (1995). *J. Org. Chem.* **60**, 8310–8312.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Marech, I., Vacca, A., Ranieri, G., Gnoni, A. & Dammacco, F. (2012). *Int. J. Oncol.* **40**, 1313–1320.
Matczak-Jon, E. & Ślepokura, K. (2011). *Acta Cryst.* C**67**, o450–o456.
PANalytical (2010). *DataCollector*. PANalytical BV, Almelo, The Netherlands.
Pawley, G. S. (1981). *J. Appl. Cryst.* **14**, 357–361.
Plabst, M., Stock, N. & Bein, T. (2009). *Cryst. Growth Des.* **9**, 5049–5060.
Popa, N. C. (1998). *J. Appl. Cryst.* **31**, 176–180.
Ruscica, R., Bianchi, M., Quintero, M., Martinez, A. & Vega, D. R. (2010). *J. Pharm. Sci.* **99**, 4962–4972.
Sergienko, V. S., Aleksandrov, G. G. & Afonin, E. G. (2000). *Crystallogr. Rep.* **45**, 432–438.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Sridhar, B. & Ravikumar, K. (2011). *Acta Cryst.* C**67**, o115–o119.
Toraya, H. (1986). *J. Appl. Cryst.* **19**, 440–447.
Visser, J. W. (1969). *J. Appl. Cryst.* **2**, 89–95.
Vitha, T., Kubíček, V., Kotek, J., Hermann, P., Vander Elst, L., Muller, R. N., Lukeš, I. & Peters, J. A. (2009). *Dalton Trans.* pp. 3204–3214.
Werner, P.-E., Eriksson, L. & Westdahl, M. (1985). *J. Appl. Cryst.* **18**, 367–370.
Wu, S., Chen, S., Li, M., Xiang, J., Xiao, Y. & Yuan, L. (2007). *CrystEngComm*, **9**, 907–914.
Zhukov, S. G., Chernyshev, V. V., Babaev, E. V., Sonneveld, E. J. & Schenk, H. (2001). *Z. Kristallogr.* **216**, 5–9.
Zlokazov, V. B. (1992). *J. Appl. Cryst.* **25**, 69–72.
Zlokazov, V. B. (1995). *Comput. Phys. Commun.* **85**, 415–422.
Zlokazov, V. B. & Chernyshev, V. V. (1992). *J. Appl. Cryst.* **25**, 447–451.

supplementary materials

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(IM) 1-(2-Hydroxy-2-phosphonato-2-phosphonoethyl)-1*H*-imidazol-3-ium

Crystal data

$C_5H_{10}N_2O_7P_2$
M_r = 272.09
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 6.8162 (12) Å
b = 10.6307 (11) Å
c = 13.9240 (14) Å
β = 96.954 (18)°
V = 1001.5 (2) Å^3
Z = 4
$F(000)$ = 560
D_x = 1.805 Mg m^{-3}
Melting point: 503 K
Cu $K\alpha$ radiation, λ = 1.5418 Å
T = 298 K
Particle morphology: plate
white
flat sheet, 15 × 1 mm
Specimen preparation: Prepared at 298 K and 101 kPa

Data collection

PANanalytical EMPYREAN diffractometer
Radiation source: line-focus sealed tube
None monochromator
Specimen mounting: thin layer on the non-diffracting silicon plate
Data collection mode: reflection
Scan method: continuous
$2\theta_{min}$ = 7.009°, $2\theta_{max}$ = 79.973°, $2\theta_{step}$ = 0.017°

Refinement

Refinement on I_{net}
Least-squares matrix: full with fixed elements per cycle
R_p = 0.033
R_{wp} = 0.048
R_{exp} = 0.030
R_{Bragg} = 0.061
χ^2 = 2.570
4293 data points
Excluded region(s): none
Profile function: split-type pseudo-Voigt (Toraya, 1986)
121 parameters
43 restraints
0 constraints
H-atom parameters not refined
Weighting scheme based on measured s.u.'s
$(\Delta/\sigma)_{max}$ = 0.002
Background function: Chebyshev polynomial up to the fifth order
Preferred orientation correction: spherical harmonics expansion up to the sixth order (Ahtee *et al.*, 1989; Järvinen, 1993)

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
P1	0.4912 (5)	0.2154 (3)	0.4136 (3)	0.0551 (14)*
P2	0.3277 (5)	−0.0379 (3)	0.3270 (3)	0.0542 (15)*
O1	0.4207 (10)	0.1527 (6)	0.2262 (6)	0.076 (3)*
H1	0.3811	0.2192	0.2011	0.114*
O2	0.6808 (12)	0.1419 (7)	0.4400 (6)	0.077 (3)*
O3	0.3630 (10)	0.2083 (7)	0.4980 (6)	0.070 (3)*
H3	0.4286	0.1786	0.5459	0.104*
O4	0.5183 (10)	0.3460 (6)	0.3771 (5)	0.063 (3)*
O5	0.1945 (11)	−0.0584 (6)	0.4078 (6)	0.072 (3)*
H5	0.2600	−0.0895	0.4553	0.108*
O6	0.5327 (10)	−0.0958 (7)	0.3482 (6)	0.073 (3)*
O7	0.2373 (10)	−0.0873 (6)	0.2264 (5)	0.066 (3)*
H7	0.3224	−0.0879	0.1896	0.099*
N1	−0.0030 (13)	0.1423 (9)	0.2245 (7)	0.068 (4)*
N2	−0.2341 (14)	0.0293 (8)	0.1485 (7)	0.071 (4)*
H2	−0.3289	−0.0240	0.1365	0.084*
C1	0.3363 (15)	0.1350 (11)	0.3146 (9)	0.064 (5)*
C2	0.1328 (16)	0.2003 (11)	0.3027 (9)	0.068 (5)*
H2A	0.0754	0.1948	0.3630	0.082*
H2B	0.1496	0.2886	0.2882	0.082*
C3	−0.1481 (16)	0.0617 (11)	0.2363 (9)	0.077 (5)*
H3A	−0.1830	0.0332	0.2951	0.093*
C4	−0.1468 (16)	0.0945 (11)	0.0809 (8)	0.065 (5)*
H4	−0.1848	0.0936	0.0144	0.080*
C5	0.0051 (16)	0.1610 (10)	0.1276 (9)	0.074 (5)*
H5A	0.0965	0.2095	0.0997	0.090*

Geometric parameters (Å, º)

P1—O4	1.498 (7)	N1—C3	1.333 (15)
P1—O2	1.516 (9)	N1—C5	1.370 (16)
P1—O3	1.549 (9)	N1—C2	1.476 (14)
P1—C1	1.842 (12)	N2—C3	1.336 (15)
P2—O5	1.544 (9)	N2—C4	1.362 (15)
P2—O6	1.522 (8)	N2—H2	0.86
P2—O7	1.552 (8)	C1—C2	1.542 (15)
P2—C1	1.847 (12)	C2—H2A	0.97
O1—C1	1.434 (15)	C2—H2B	0.97
O1—H1	0.82	C3—H3A	0.93
O3—H3	0.82	C4—C5	1.354 (15)
O5—H5	0.82	C4—H4	0.93

O7—H7	0.82	C5—H5A	0.93
O4—P1—O2	115.0 (5)	O1—C1—C2	107.3 (9)
O4—P1—O3	114.0 (4)	O1—C1—P1	109.2 (7)
O2—P1—O3	109.5 (5)	C2—C1—P1	107.4 (8)
O4—P1—C1	105.0 (5)	O1—C1—P2	103.4 (7)
O2—P1—C1	109.7 (5)	C2—C1—P2	114.8 (8)
O3—P1—C1	102.7 (5)	P1—C1—P2	114.4 (6)
O5—P2—O6	114.1 (5)	N1—C2—C1	111.5 (9)
O5—P2—O7	114.0 (4)	N1—C2—H2A	109.3
O6—P2—O7	107.0 (4)	C1—C2—H2A	109.3
O5—P2—C1	103.6 (5)	N1—C2—H2B	109.3
O6—P2—C1	112.4 (5)	C1—C2—H2B	109.4
O7—P2—C1	105.4 (5)	H2A—C2—H2B	107.9
C1—O1—H1	109.5	N1—C3—N2	107.6 (11)
P1—O3—H3	109.5	N1—C3—H3A	126.2
P2—O5—H5	109.5	N2—C3—H3A	126.2
P2—O7—H7	109.4	C5—C4—N2	107.8 (10)
C3—N1—C5	109.5 (9)	C5—C4—H4	126.0
C3—N1—C2	125.8 (10)	N2—C4—H4	126.1
C5—N1—C2	124.7 (9)	C4—C5—N1	106.1 (10)
C3—N2—C4	108.7 (9)	C4—C5—H5A	126.9
C3—N2—H2	125.6	N1—C5—H5A	127.0
C4—N2—H2	125.7		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O6^{i}	0.82	1.71	2.481 (11)	156
O5—H5···O2^{i}	0.82	1.57	2.359 (11)	160
O7—H7···O4^{ii}	0.82	1.67	2.436 (10)	156
N2—H2···O4^{iii}	0.86	1.88	2.740 (11)	172
O1—H1···O6^{iv}	0.82	2.19	2.899 (11)	145

Symmetry codes: (i) −*x*+1, −*y*, −*z*+1; (ii) −*x*+1, *y*−1/2, −*z*+1/2; (iii) −*x*, *y*−1/2, −*z*+1/2; (iv) −*x*+1, *y*+1/2, −*z*+1/2.

(IT) 1-(2-Hydroxy-2-phosphonato-2-phosphonoethyl)-1*H*-imidazol-3-ium

Crystal data

$C_5H_{10}N_2O_7P_2$
M_r = 272.09
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 8.4217 (13) Å
b = 8.6039 (11) Å
c = 8.1818 (12) Å
α = 92.778 (16)°
β = 112.415 (17)°
γ = 116.072 (19)°
V = 475.33 (12) Å^3
Z = 2

$F(000)$ = 280
D_x = 1.901 Mg m^{-3}
Melting point: 495 K
Cu $K\alpha_1$ radiation, λ = 1.5406 Å
μ = 4.50 mm^{-1}
T = 298 K
Particle morphology: no specific habit
white
flat sheet, 15 × 1 mm
Specimen preparation: Prepared at 298 K and 101 kPa

Data collection

Huber Guinier Camera G670 diffractometer
Radiation source: line-focus sealed tube
Curved Germanium(111) monochromator
Specimen mounting: thin layer in the specimen holder of the camera
Data collection mode: transmission
Scan method: continuous
$2\theta_{min}$ = 7.00°, $2\theta_{max}$ = 80.00°, $2\theta_{step}$ = 0.01°

Refinement

Refinement on I_{net}
Least-squares matrix: full with fixed elements per cycle
R_p = 0.026
R_{wp} = 0.033
R_{exp} = 0.015
R_{Bragg} = 0.050
χ^2 = 4.550
7301 data points
Excluded region(s): none
Profile function: split-type pseudo-Voigt (Toraya, 1986)
102 parameters
43 restraints
0 constraints
H-atom parameters not refined
Weighting scheme based on measured s.u.'s
$(\Delta/\sigma)_{max}$ = 0.002
Background function: Chebyshev polynomial up to the fifth order
Preferred orientation correction: March–Dollase (Dollase, 1986) texture correction

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
P1	−0.2088 (4)	0.6438 (4)	0.4230 (4)	0.0380 (15)*
P2	−0.1622 (4)	0.7888 (4)	0.0929 (5)	0.0431 (15)*
O1	−0.1430 (9)	0.4938 (9)	0.1814 (10)	0.050 (3)*
H1	−0.0674	0.4638	0.2498	0.075*
O2	−0.1274 (10)	0.5710 (10)	0.5770 (10)	0.057 (3)*
O3	−0.1769 (9)	0.8325 (9)	0.4878 (9)	0.055 (3)*
H3	−0.0935	0.8775	0.5952	0.083*
O4	−0.4320 (8)	0.5176 (9)	0.2965 (9)	0.047 (3)*
H4	−0.4462	0.4633	0.2023	0.071*
O5	−0.3872 (9)	0.7015 (8)	−0.0065 (9)	0.052 (3)*
O6	−0.0680 (9)	0.9850 (9)	0.1807 (10)	0.051 (3)*
O7	−0.0814 (9)	0.7559 (9)	−0.0395 (10)	0.053 (3)*
H7	−0.0488	0.8415	−0.0837	0.080*
N1	0.2623 (11)	0.8163 (11)	0.3117 (12)	0.068 (4)*
N2	0.4221 (11)	0.8120 (11)	0.1616 (12)	0.058 (4)*
H2	0.4672	0.7747	0.0999	0.070*
C1	−0.0909 (16)	0.6710 (15)	0.2673 (16)	0.067 (5)*
C2	0.1331 (14)	0.7658 (15)	0.4031 (15)	0.064 (5)*
H2A	0.1670	0.8728	0.4849	0.077*
H2B	0.1578	0.6865	0.4768	0.077*
C3	0.3042 (16)	0.7112 (15)	0.2292 (15)	0.062 (5)*
H3A	0.2588	0.5897	0.2210	0.074*

C4	0.4610 (14)	0.9836 (14)	0.2045 (16)	0.063 (5)*
H4A	0.5462	1.0816	0.1795	0.076*
C5	0.3532 (15)	0.9854 (16)	0.2901 (16)	0.058 (5)*
H5	0.3423	1.0826	0.3274	0.070*

Geometric parameters (Å, º)

P1—O2	1.498 (9)	N1—C3	1.34 (2)
P1—O4	1.548 (6)	N1—C5	1.372 (15)
P1—O3	1.554 (9)	N1—C2	1.472 (18)
P1—C1	1.858 (16)	N2—C3	1.324 (16)
P2—O6	1.505 (8)	N2—C4	1.359 (16)
P2—O5	1.527 (7)	N2—H2	0.86
P2—O7	1.555 (11)	C1—C2	1.556 (14)
P2—C1	1.856 (14)	C2—H2A	0.97
O1—C1	1.443 (16)	C2—H2B	0.97
O1—H1	0.82	C3—H3A	0.93
O3—H3	0.82	C4—C5	1.35 (2)
O4—H4	0.82	C4—H4A	0.93
O7—H7	0.82	C5—H5	0.93
O2—P1—O4	112.7 (4)	O1—C1—C2	106.3 (11)
O2—P1—O3	113.6 (4)	O1—C1—P2	110.9 (8)
O4—P1—O3	108.2 (5)	C2—C1—P2	115.3 (8)
O2—P1—C1	112.5 (6)	O1—C1—P1	106.9 (8)
O4—P1—C1	104.3 (5)	C2—C1—P1	102.6 (8)
O3—P1—C1	104.9 (5)	P2—C1—P1	114.1 (9)
O6—P2—O5	111.2 (5)	N1—C2—C1	113.4 (10)
O6—P2—O7	112.2 (5)	N1—C2—H2A	108.9
O5—P2—O7	110.4 (4)	C1—C2—H2A	108.9
O6—P2—C1	110.5 (5)	N1—C2—H2B	108.8
O5—P2—C1	109.6 (5)	C1—C2—H2B	108.8
O7—P2—C1	102.6 (6)	H2A—C2—H2B	107.8
C1—O1—H1	109.5	N2—C3—N1	107.5 (11)
P1—O3—H3	109.5	N2—C3—H3A	126.4
P1—O4—H4	109.5	N1—C3—H3A	126.2
P2—O7—H7	109.5	C5—C4—N2	107.3 (10)
C3—N1—C5	108.6 (11)	C5—C4—H4A	126.3
C3—N1—C2	128.2 (10)	N2—C4—H4A	126.4
C5—N1—C2	123.1 (12)	C4—C5—N1	106.9 (12)
C3—N2—C4	109.6 (12)	C4—C5—H5	126.6
C3—N2—H2	125.2	N1—C5—H5	126.5
C4—N2—H2	125.2		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O5^{i}	0.82	1.69	2.406 (9)	144
O7—H7···O6^{ii}	0.82	1.80	2.604 (10)	166
O3—H3···O6^{iii}	0.82	1.76	2.559 (10)	166

O1—H1···O2^{iv}	0.82	1.87	2.682 (12)	172
N2—H2···O5^{v}	0.86	2.02	2.877 (13)	171

Symmetry codes: (i) −*x*−1, −*y*+1, −*z*; (ii) −*x*, −*y*+2, −*z*; (iii) −*x*, −*y*+2, −*z*+1; (iv) −*x*, −*y*+1, −*z*+1; (v) *x*+1, *y*, *z*.