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Poly[(μ_2-2,2′-bi-1*H*-imidazole)bis(μ_3-hydrogenphosphato)dizinc(II)]

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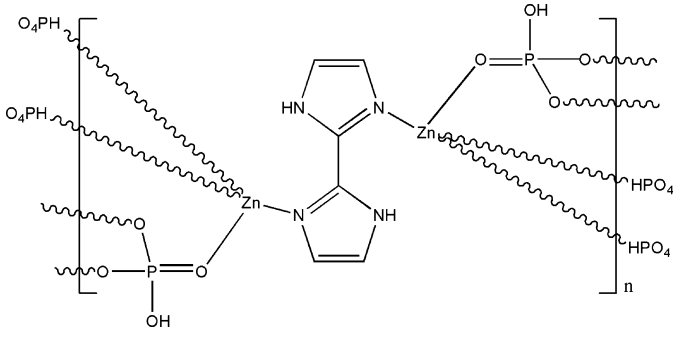
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.032; wR factor = 0.076; data-to-parameter ratio = 12.0.

The Zn^{II} ion in the title compound, $[Zn_2(HPO_4)_2(C_6H_6N_4)]$, exhibits a tetrahedral geometry. It is coordinated by three O atoms from three hydrogenphosphate ions of three different structure units and one N atom of a centrosymmetric bridging 2,2′-biimidazole molecule. In this way, a three-dimensional polymer is built. The crystal packing is stabilized by O—H···O, N—H···O and C—H···O hydrogen bonds.

Related literature

For related literature, see: Xia *et al.* (2006); Kamar *et al.* (2004); Xiao & Shreeve (2005).



Experimental

Crystal data

$[Zn_2(HPO_4)_2(C_6H_6N_4)]$
M_r = 456.88
Orthorhombic, *Pbca*
a = 8.8050 (5) Å
b = 8.8051 (7) Å
c = 16.6614 (14) Å
V = 1291.75 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.01 mm^{-1}
T = 296 (2) K
0.20 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.501, T_{max} = 0.501
(expected range = 0.448–0.448)
7551 measured reflections
1275 independent reflections
1167 reflections with $I > 2\sigma(I)$
R_{int} = 0.094

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.076
S = 1.08
1275 reflections
106 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.62 e Å^{-3}
$\Delta\rho_{min}$ = −0.64 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2B···O3^{i}	0.80 (4)	1.84 (4)	2.615 (3)	165 (4)
N2—H2A···O1^{ii}	0.77 (4)	2.05 (4)	2.804 (3)	168 (4)
C2—H2···O2^{iii}	0.93	2.50	3.303 (4)	145

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

The authors thank Xianggao Meng for assistance with the refinement of the structure.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2492).

References

Bruker (2001). *SMART*, *SAINT*, *SADABS* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Kamar, K. K., Falvello, L. R., Fanwick, P. E., Kim, J. & Goswami, S. (2004). *Dalton Trans.* pp. 1827–1831.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Xia, C.-K., Lu, C.-Z., Yuan, D.-Q., Zhang, Q.-Z., Wu, X.-Y., Xiang, S.-C., Zhang, J.-J. & Wu, D.-M. (2006). *CrystEngComm*, **8**, 281–291.
Xiao, J.-C. & Shreeve, J. M. (2005). *J. Org. Chem.* **70**, 3072–3078.

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supplementary materials

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Poly[(μ_2-2,2'-bi-1*H*-imidazole)bis(μ_3-hydrogenphosphato)dizinc(II)]

M.-N. Cao, M. Wang, F.-H. Luo, C.-X. Cheng and Z.-Q. Hu

Comment

Biimidazole (H_2biim) is a bidentate chelating ligand with multiple proton-donor sites which can coordinate to a transition metal in its neutral (H_2biim), singly-deprotonated ($Hbiim^-$) and doubly-deprotonated ($biim^{2-}$) forms. Coordinated H_2biim usually forms hydrogen bonds with counteranions or solvent molecules (Xia *et al.*, 2006); Kamar *et al.*, 2004).

Here, we report the synthesis and crystal structure of the title compound, which contains Zn(II) ions, neutral H_2biim molecules and hydrogen phosphate ions. The Zn^{II} ion exhibits a distorted tetrahedral geometry. It is coordinated to three O atoms from a hydrogen phosphate ion and one N atom of a 2,2'-biimidazole.

The crystal packing is stabilized by O—H···O, N—H···O and C—H···O hydrogen. bonds.

Experimental

2,2'-biimidazole was synthesized according to the literature procedure (Xiao & Shreeve, 2005). A mixture of $Zn(NO_3)_2{\cdot}4H_2O$, 2,2'-biimidazole in 1:1 molar ratio with 0.2 ml H_3PO_4 and 10 ml water was sealed into a Teflon-lined pressure vessel and heated at 433 K for 72 h. After the mixture cooled to room temperature, colourless crystals were formed, collected by filtration, washed in deionized water, and finally dried in air.

Refinement

After having located them in a difference map, H-atoms bonded to C were fixed geometrically at ideal positions and allowed to ride on their parent atoms with C–H=0.93 Å and U_{iso}(H)=1.2U_{eq}(C), 1.2U_{eq}(N), or 1.5U_{eq}(O). The coordinates of the H atoms bonded to N and O were refined.

Figures

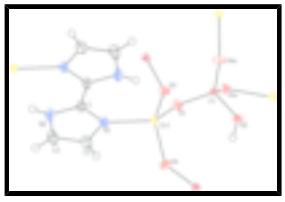

Fig. 1. View of the molecular structure of (I), showing 50% probability displacement ellipsoids for the non-hydrogen atoms. [Symmetry codes:(*a*) 3/2 − *x*, −*y*, 1/2 + *z*]

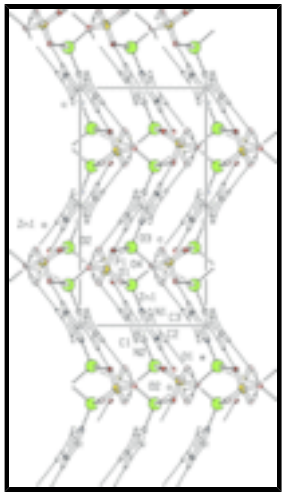

Fig. 2. Part of the crystal structure of the title compound. Hydrogen bonds are shown as dashed lines.

Poly[(μ$_2$-2,2'-bi-1*H*-imidazole)bis(μ$_3$-hydrogenphosphato)dizinc(II)]

Crystal data

$[Zn_2(HPO_4)_2(C_6H_6N_4)]$	F_{000} = 904
M_r = 456.88	D_x = 2.349 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 4008 reflections
a = 8.8050 (5) Å	θ = 2.6–28.2º
b = 8.8051 (7) Å	μ = 4.01 mm^{-1}
c = 16.6614 (14) Å	*T* = 296 (2) K
V = 1291.75 (18) Å^3	Block, colourless
Z = 4	0.20 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer	1275 independent reflections
Radiation source: fine-focus sealed tube	1167 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.094
T = 296(2) K	θ_{max} = 26.0º
φ and ω scans	θ_{min} = 2.4º
Absorption correction: multi-scan (SADABS; Bruker, 2001)	*h* = −10→10
T_{min} = 0.501, T_{max} = 0.501	*k* = −10→10
7551 measured reflections	*l* = −20→19

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.033$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.076$	$w = 1/[\sigma^2(F_o^2) + (0.0384P)^2 + 0.9211P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{max} < 0.001$
1275 reflections	$\Delta\rho_{max} = 0.62$ e Å^{-3}
106 parameters	$\Delta\rho_{min} = -0.63$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Zn1	0.32416 (4)	0.09443 (4)	0.670742 (19)	0.01604 (15)
C1	0.4213 (3)	−0.0248 (3)	0.50171 (16)	0.0182 (6)
C2	0.2054 (4)	−0.1293 (4)	0.4651 (2)	0.0295 (7)
H2	0.1320	−0.1817	0.4361	0.035*
C3	0.1870 (3)	−0.0582 (4)	0.5357 (2)	0.0270 (7)
H3	0.0966	−0.0540	0.5645	0.032*
N1	0.3221 (3)	0.0079 (3)	0.55921 (14)	0.0198 (5)
N2	0.3541 (3)	−0.1087 (3)	0.44472 (17)	0.0258 (6)
H2A	0.387 (4)	−0.139 (4)	0.405 (2)	0.031*
O1	0.4899 (2)	0.2367 (2)	0.68551 (11)	0.0201 (5)
O2	0.4137 (2)	0.3524 (3)	0.81871 (12)	0.0204 (5)
H2B	0.344 (4)	0.389 (4)	0.795 (3)	0.031*
O3	0.3408 (2)	−0.0739 (2)	0.74787 (13)	0.0190 (4)
O4	0.1231 (2)	0.1699 (3)	0.68453 (13)	0.0240 (5)
P1	0.55013 (7)	0.29324 (8)	0.76628 (4)	0.01433 (19)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.0150 (2)	0.0183 (2)	0.0149 (2)	0.00045 (13)	0.00130 (12)	−0.00147 (12)
C1	0.0196 (14)	0.0219 (14)	0.0131 (13)	−0.0020 (12)	0.0005 (11)	−0.0027 (11)
C2	0.0226 (15)	0.0432 (18)	0.0227 (17)	−0.0135 (15)	0.0019 (14)	−0.0065 (15)
C3	0.0175 (15)	0.0409 (18)	0.0224 (17)	−0.0056 (14)	0.0043 (13)	−0.0038 (14)

N1	0.0174 (13)	0.0265 (13)	0.0153 (12)	−0.0030 (10)	0.0008 (9)	−0.0017 (10)
N2	0.0233 (13)	0.0360 (16)	0.0179 (14)	−0.0068 (12)	0.0047 (11)	−0.0104 (11)
O1	0.0189 (10)	0.0256 (11)	0.0157 (10)	−0.0058 (9)	0.0003 (8)	−0.0038 (8)
O2	0.0130 (10)	0.0300 (12)	0.0182 (10)	0.0073 (10)	0.0001 (8)	−0.0007 (8)
O3	0.0155 (10)	0.0213 (10)	0.0202 (11)	0.0008 (8)	0.0011 (8)	0.0039 (8)
O4	0.0167 (10)	0.0240 (11)	0.0313 (12)	0.0046 (9)	0.0017 (9)	−0.0075 (9)
P1	0.0108 (3)	0.0179 (4)	0.0143 (4)	0.0012 (3)	−0.0001 (3)	−0.0012 (3)

Geometric parameters (Å, °)

Zn1—O4	1.905 (2)	C3—N1	1.381 (4)
Zn1—O1	1.939 (2)	C3—H3	0.9300
Zn1—O3	1.967 (2)	N2—H2A	0.77 (4)
Zn1—N1	2.009 (2)	O1—P1	1.530 (2)
C1—N1	1.328 (4)	O2—P1	1.574 (2)
C1—N2	1.340 (4)	O2—H2B	0.80 (4)
C1—C1^{i}	1.454 (5)	O3—P1^{ii}	1.532 (2)
C2—C3	1.343 (5)	O4—P1^{iii}	1.505 (2)
C2—N2	1.365 (4)	P1—O4^{iv}	1.505 (2)
C2—H2	0.9300	P1—O3^{v}	1.532 (2)
O4—Zn1—O1	117.29 (9)	C1—N1—Zn1	138.0 (2)
O4—Zn1—O3	104.68 (9)	C3—N1—Zn1	115.46 (19)
O1—Zn1—O3	110.36 (9)	C1—N2—C2	108.7 (3)
O4—Zn1—N1	103.63 (9)	C1—N2—H2A	130 (3)
O1—Zn1—N1	111.68 (9)	C2—N2—H2A	122 (3)
O3—Zn1—N1	108.61 (9)	P1—O1—Zn1	125.65 (12)
N1—C1—N2	109.9 (2)	P1—O2—H2B	116 (3)
N1—C1—C1^{i}	126.2 (3)	P1^{ii}—O3—Zn1	121.44 (12)
N2—C1—C1^{i}	123.9 (3)	P1^{iii}—O4—Zn1	135.61 (14)
C3—C2—N2	105.8 (3)	O4^{iv}—P1—O1	113.12 (13)
C3—C2—H2	127.1	O4^{iv}—P1—O3^{v}	111.54 (12)
N2—C2—H2	127.1	O1—P1—O3^{v}	109.30 (12)
C2—C3—N1	109.9 (3)	O4^{iv}—P1—O2	105.22 (12)
C2—C3—H3	125.0	O1—P1—O2	109.35 (12)
N1—C3—H3	125.0	O3^{v}—P1—O2	108.13 (12)
C1—N1—C3	105.7 (2)		
N2—C2—C3—N1	0.6 (4)	C1^{i}—C1—N2—C2	−177.4 (4)
N2—C1—N1—C3	−0.6 (3)	C3—C2—N2—C1	−1.0 (4)
C1^{i}—C1—N1—C3	177.8 (4)	O4—Zn1—O1—P1	76.82 (17)
N2—C1—N1—Zn1	168.2 (2)	O3—Zn1—O1—P1	−42.89 (18)
C1^{i}—C1—N1—Zn1	−13.4 (6)	N1—Zn1—O1—P1	−163.81 (14)
C2—C3—N1—C1	0.0 (4)	O4—Zn1—O3—P1^{ii}	153.03 (13)
C2—C3—N1—Zn1	−171.8 (2)	O1—Zn1—O3—P1^{ii}	−79.90 (15)
O4—Zn1—N1—C1	157.1 (3)	N1—Zn1—O3—P1^{ii}	42.83 (16)
O1—Zn1—N1—C1	30.0 (3)	O1—Zn1—O4—P1^{iii}	−29.4 (2)

O3—Zn1—N1—C1	−92.0 (3)	O3—Zn1—O4—P1^{iii}	93.3 (2)
O4—Zn1—N1—C3	−34.8 (2)	N1—Zn1—O4—P1^{iii}	−153.0 (2)
O1—Zn1—N1—C3	−161.9 (2)	Zn1—O1—P1—O4^{iv}	65.95 (18)
O3—Zn1—N1—C3	76.1 (2)	Zn1—O1—P1—O3^{v}	−169.13 (14)
N1—C1—N2—C2	1.0 (4)	Zn1—O1—P1—O2	−50.94 (18)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2B···O3^{vi}	0.80 (4)	1.84 (4)	2.615 (3)	165 (4)
N2—H2A···O1^{i}	0.77 (4)	2.05 (4)	2.804 (3)	168 (4)
C2—H2···O2^{vii}	0.93	2.50	3.303 (4)	145

Symmetry codes: (vi) −*x*+1/2, *y*+1/2, *z*; (i) −*x*+1, −*y*, −*z*+1; (vii) −*x*+1/2, −*y*, *z*−1/2.

Fig. 1

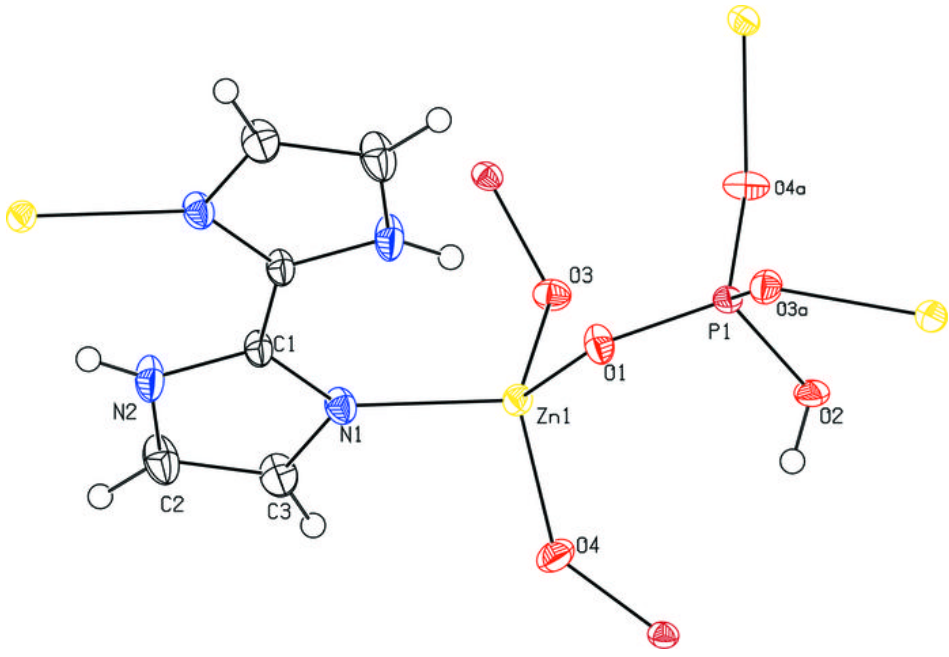

Fig. 2