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Crystallographic report

A two-dimensional zinc phosphate framework: $[H_3N(CH_2)_3NH_3]_{0.5}[Zn_2(PO_4)(HPO_4)]$

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The two-dimensional zinc phosphate $[H_3N(CH_2)_3NH_3]_{0.5}[Zn_2(PO_4)(HPO_4)]$, has been synthesized hydrothermally using 1,3-diaminopropane as the template. Its structure contains an inorganic framework with three-, four-, or six-membered rings, built from PO_4, $PO_3(OH)$ and ZnO_4 tetrahedral moieties sharing vertexes. The protonated 1,3-diaminopropane molecules interact with the framework through hydrogen bonds. Copyright © 2004 John Wiley & Sons, Ltd.

KEYWORDS: hydrothermal synthesis; zinc phosphate; inorganic framework; crystal structure

COMMENT

A vast number of organically templated zincophosphates containing inorganic frameworks have been synthesized in the last decade.[1–3] Some of them display interesting features, such as three-membered rings and 'infinite' sequences of –Zn–O–Zn–O–bonds.[4,5] The crystal structure of a layered two-dimensional zinc phosphate, $[H_3N(CH_2)_3NH_3]_{0.5}[Zn_2(PO_4)(HPO_4)]$ (**1**), is shown to contain three-membered rings. The structure, Fig. 1, is built from PO_4, $PO_3(OH)$ and ZnO_4 tetrahedral moieties by sharing vertexes via Zn–O–P and Zn–O–Zn links. The connectivity between PO_4, $PO_3(OH)$ and ZnO_4 blocks gives rise to inorganic layers with three-, four-, or six-membered rings along the *b* axis. These layers are held together by protonated 1,3-diaminopropane through hydrogen bonds, as shown in Fig. 2.

EXPERIMENTAL

(**1**) was prepared hydrothermally from a reaction mixture of $Zn(OAc)_2$, H_3PO_4, H_3PO_3, 1,3-diaminopropane and distilled water with a molar composition of 1:2:2:3.5:900. The mixture was

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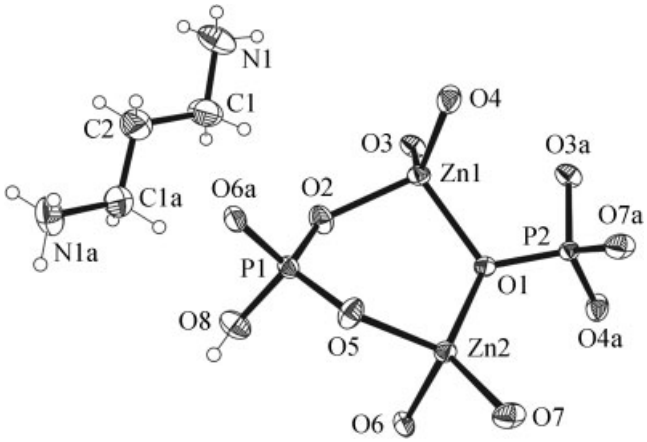

Figure 1. ORTEP plot of **1**. Thermal ellipsoids are given at 50% probability. Key geometry parameters: Zn1–O1 2.036(2), Zn1–O2 1.948(3), Zn1–O3 1.932(2), Zn1–O4 1.923(2), Zn2–O1 1.962(3), Zn2–O5 1.943(2), Zn2–O6 1.983(2), Zn2–O7 1.898(3), P1–O2 1.509(3), P1–O5 1.520(3), P1–O6c 1.529(3), P1–O8 1.582(3), P2–O1 1.567(3), P2–O3a 1.531(3), P2–O4b 1.511(3), P2–O7d 1.518(3) Å; O1–Zn1–O2 100.01(11), O1–Zn1–O3 111.66(10), O1–Zn1–O4 108.96(10), O2–Zn1–O3 101.59(11), O2–Zn1–O4 115.13(11), O3–Zn1–O4 117.96(12), O1–Zn2–O5 106.45(11), O1–Zn2–O6 109.06(10), O1–Zn2–O7 123.57(11), O5–Zn2–O6 103.24(11), O5–Zn2–O7 108.09(11), O6–Zn2–O7 104.69(11)°. Symmetry codes: a = $1-x$, $-y$, $1-z$; b = x, $-1+y$, z; c = x, $1+y$, z; d = $0.5-x$, $-0.5-y$, $1-z$.

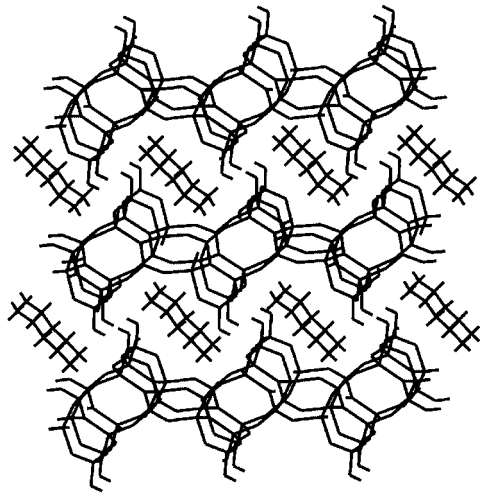

Figure 2. The two-dimensional structure of **1** viewed along the *b* axis.

stirred for 20 min at room temperature and then crystallized in a 23 ml capacity Teflon-lined stainless-steel autoclave at 160 °C for 120 h. Data collection was performed at 293(2) K on a Siemens Smart CCD diffractometer for a colorless crystal $0.15 \times 0.15 \times 0.30\ mm^3$. $C_{1.5}H_7NO_8P_2Zn_2$, $M = 359.76$, monoclinic, $C2/c$, $a = 17.2338(12)$ Å, $b = 5.1936(3)$ Å, $c = 20.0783(11)$ Å, $\beta = 92.549(7)°$, $V = 1795.34(19)$ Å^3, $Z = 8$, 1543 unique reflections ($\theta_{max} = 25.0°$), $R = 0.026$ for 1284 data with $I > 2\sigma(I)$; $wR = 0.059$ (all data). Programs used: SHELXS-97, SHELXL-97 and ORTEP. CCDC deposition number: 235629.

Acknowledgements

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