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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ (P–O) = 0.004 Å
H-atom completeness 61%
R factor = 0.036
wR factor = 0.086
Data-to-parameter ratio = 25.6

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

Barium dihydrogen phosphite hemihydrate

$\text{Ba}(\text{H}_2\text{PO}_3)_2 \cdot 0.5\text{H}_2\text{O}$ contains ninefold-coordinated Ba ions connected by dihydrogen phosphite anions to form a three-dimensional network. The asymmetric unit contains two Ba^{2+} ions, four $[\text{H}_2\text{PO}_3]^-$ ions and one water molecule.

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Comment

Crystal structure analyses of alkaline-earth dihydrogen phosphites have been undertaken for several decades (Ouarsal *et al.*, 2002, and references therein). Continuing these investigations, we report here the structure of $\text{Ba}(\text{H}_2\text{PO}_3)_2 \cdot 0.5\text{H}_2\text{O}$. Fig. 1 shows the asymmetric unit. The Ba ions occupy two different ninefold-coordinated sites. The coordination of Ba1 is made up of seven monodentate dihydrogen phosphite groups and two O atoms from two water molecules. Ba2 is surrounded by six monodentate and one bidentate dihydrogen phosphite ligands. The average $d_{\text{Ba}-\text{O}}$ is 2.83 (11) and 2.86 (10) Å, respectively. In $\text{BaZn}_3(\text{HPO}_3)_4 \cdot 6\text{H}_2\text{O}$, Ba is twelvefold-coordinated with $d_{\text{Ba}-\text{O}}$ ranging from 2.876 to 3.053 Å (Ortiz-Avila *et al.*, 1989). Two neighbouring BaO_9 units, related by a centre of symmetry, share an O–O edge, to form a Ba_2O_{16} dimer. Two such dimers are joined through an edge (O33–O43) and a face (O41–O23–O32) to build up a double channel parallel to [010] (Fig. 2). There are four crystallographically independent phosphorus centres, each

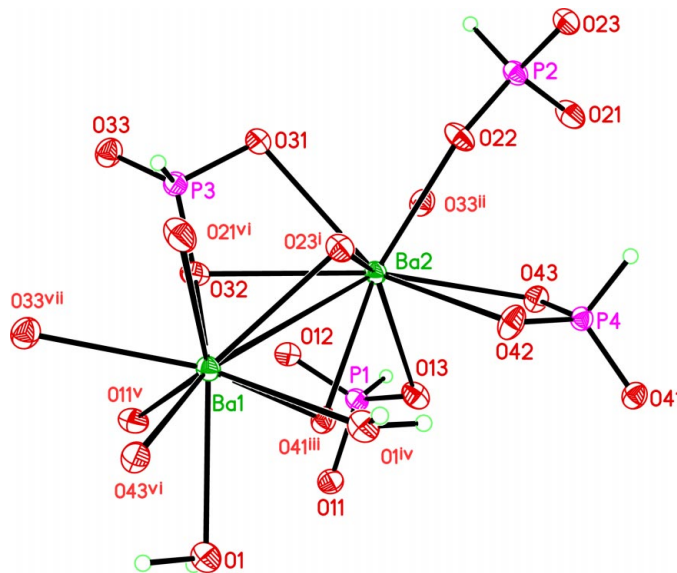


Figure 1

Perspective view of the asymmetric unit of the title compound, with the atom numbering; displacement ellipsoids are at the 50% probability level. Symmetry operators for equivalent atoms: (i) $3/2-x, -1/2+y, 3/2-z$; (ii) $1-x, 1-y, 1-z$; (iii) $2-x, 1-y, 1-z$; (iv) $2-x, -y, 1-z$; (v) $3/2-x, -1/2+y, 1/2-z$; (vi) $x, -1+y, z$; (vii) $1-x, -y, 1-z$.

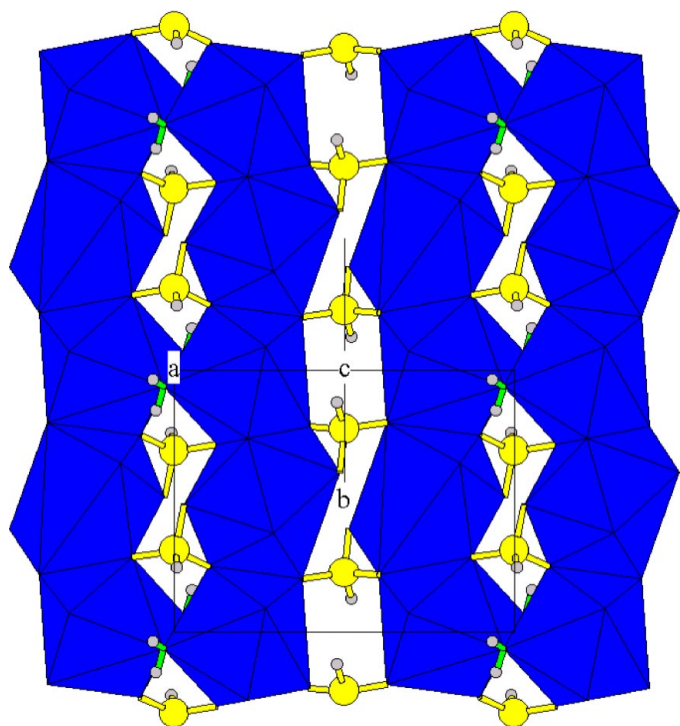


Figure 2
Packing diagram of the title compound.

tetrahedrally coordinated by one H and three O atoms. The P—O bond distances are in the usual range, similar to those reported in $\text{Sr}(\text{H}_2\text{PO}_3)_2$ (Ouarsal *et al.*, 2002).

Experimental

25 ml of $\text{BaCl}_2 \cdot 6\text{H}_2\text{O}$ (0.1 M) and 25 ml of phosphorous acid (1 M) were mixed in water. The mixture was stirred for 8 h at 333 K, before being left at room temperature. After a few days, colourless crystals were deposited. These were filtered off and washed with a solution of ethanol (80%).

Crystal data

$\text{Ba}(\text{H}_2\text{PO}_3)_2 \cdot 0.5\text{H}_2\text{O}$
 $M_r = 308.32$
 Monoclinic, $P2_1/n$
 $a = 11.2351$ (8) Å
 $b = 8.7244$ (5) Å
 $c = 14.0518$ (9) Å
 $\beta = 90.493$ (6)°
 $V = 1377.30$ (15) Å³
 $Z = 8$

$D_x = 2.974$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 32287 reflections
 $\theta = 4.0\text{--}32.7^\circ$
 $\mu = 6.21$ mm⁻¹
 $T = 173$ (2) K
 Plate, colourless
 $0.28 \times 0.18 \times 0.05$ mm

Data collection

Stoe IPDS-II two-circle diffractometer
 ω scans
 Absorption correction: multi-scan (MULABS; Spek, 1990; Blessing, 1995)
 $T_{\min} = 0.275$, $T_{\max} = 0.747$
 38 630 measured reflections

4582 independent reflections
 3635 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.097$
 $\theta_{\max} = 31.5^\circ$
 $h = -16 \rightarrow 16$
 $k = -12 \rightarrow 12$
 $l = -20 \rightarrow 20$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.086$
 $S = 0.93$
 4582 reflections
 179 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0533P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.002$
 $\Delta\rho_{\max} = 2.01$ e Å⁻³
 $\Delta\rho_{\min} = -1.79$ e Å⁻³
 Extinction correction: SHELXL97
 Extinction coefficient: 0.0045 (3)

Table 1

Hydrogen-bonding geometry (Å, °).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
$\text{O1—H1A}\cdots\text{O13}^i$	0.8400 (10)	2.17 (3)	2.947 (5)	153 (7)
$\text{O1—H1A}\cdots\text{O43}^i$	0.8400 (10)	2.41 (6)	2.909 (5)	118 (6)
$\text{O1—H1B}\cdots\text{O12}^{ii}$	0.8400 (10)	1.986 (12)	2.818 (5)	171 (7)

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

The water H atoms were located in a difference Fourier synthesis and were refined freely, constraining the O—H distance to 0.84 Å. H atoms bonded to P were refined with fixed individual displacement parameters [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{P})$], using a riding model with P—H = 1.30 Å. The H atoms bonded to the O atoms of the dihydrogen phosphite ions could not be located and were omitted from the refinement. The ten highest peaks in the difference electron-density map are located close (~ 1 Å) to the Ba atoms and the deepest hole is 0.69 Å from Ba1.

Data collection: X-Area (Stoe & Cie, 2001); cell refinement: X-Area; data reduction: X-Area; program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: XP in SHELXTL-Plus (Sheldrick, 1991).

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