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$Zn(AsO_3)_2$, a unique structure among arsenates with composition $M^{II}As^{V}_2O_6$

Maximilian Pfeiffer,[a] Berthold Stöger[a] and Matthias Weil[b]*

[a]TU Wien, X-Ray Centre, Getreidemarkt 9/E057, 1060 Vienna, Austria, and [b]TU Wien, Institute for Chemical Technologies and Analytics, Division of Applied Solid State Chemistry, Getreidemarkt 9/E164-05-1, 1060 Vienna, Austria. *Correspondence e-mail: matthias.weil@tuwien.ac.at

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Zinc bis[*catena*-arsenate(V)] or *catena*-poly[zinc-di-μ-arsenato(V)-κ^4O:O], $[Zn(AsO_3)_2]_n$, is isotypic with the low-temperature modification of $Zn(PO_3)_2$. The asymmetric unit comprises one Zn atom situated on a twofold rotation axis and one As and three O atoms in general positions. In the crystal structure, the principal building units, ZnO_6 octahedra and AsO_4 tetrahedra, are condensed into $^1_\infty[ZnO_{4/2}O_{2/1}]$ zigzag chains and $(AsO_3)_2$ polyarsenate chains with a periodicity of two AsO_4 tetrahedra, respectively. The two types of chains extend parallel to [001] and are fused into a compact framework structure. Compared with divalent transition metal arsenates and other arsenates of the formula $M^{II}As_2O_6$, $Zn(AsO_3)_2$ is unique in that arsenic is present exclusively in a tetrahedral coordination and not in a mixed tetrahedral/octahedral or purely octahedral coordination.

1. Chemical context

The zinc arsenate $Zn(AsO_3)_2$ has been reported as one of the phases existing in the *pseudo*-binary system $ZnO–As_2O_5$ (Kasenov *et al.*, 1996). Whereas for the other phases reported in this system complete crystal structure determinations have been carried out in the meantime (details are given in the database survey), for $Zn(AsO_3)_2$ only unit-cell parameters were given on basis of a powder X-ray diffraction study: crystal system orthorhombic with a = 8.625, b = 9.035, c = 12.494 Å, V = 933 Å^3 (Mustafin & Kasenov, 1994, 1996). Recent results of crystal structure prediction methods claimed the possible existence of $Zn(AsO_3)_2$ (or as stated there of $ZnAs_2O_6$). Whereas one study states that this phase has an existence probability of 0.9105 with a polar structure (Gake *et al.*, 2025; data given in the supporting information) the other states that it crystallizes in the centrosymmetric $PbSb_2O_6$ structure type with trigonal symmetry (Griesemer *et al.*, 2025; supplementary material of this article: OQMD ID 1348395).

We sought to investigate these conflicting claims and carried out experiments on the crystal growth of $Zn(AsO_3)_2$. Using a reaction similar to that described by Kasenov *et al.* (1996), we were able to isolate single crystals of $Zn(AsO_3)_2$ (**I**) and report here on the results of the crystal structure analysis.

2. Structural commentary

Our study does not confirm any of the previous statements made regarding the orthorhombic unit cell or possible crystal structures. Compound (**I**) crystallizes in the monoclinic system in the centrosymmetric space group $C2/c$ and is isotypic with the corresponding phosphate, *i.e.* low-temperature $Zn(PO_3)_2$ (Averbuch-Pouchot *et al.*, 1983). The unit cell of $Zn(AsO_3)_2$

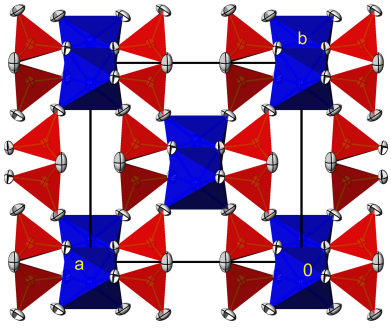

Table 1
Selected bond lengths (Å).

Zn1—O3	2.0276 (10)	As1—O2	1.6518 (10)
Zn1—O2^{i}	2.0786 (10)	As1—O1^{iii}	1.7319 (10)
Zn1—O2^{ii}	2.1640 (10)	As1—O1^{iv}	1.7379 (10)
As1—O3	1.6336 (10)		

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

Table 2
Bond-valence-sum calculations (in valence units) for $Zn(AsO_3)_2$.

	Zn1	As1	Σ
O1		1.10, 1.08	2.18
O2	0.36 (1×→, 2×↓), 0.29 (1×→, 2×↓)	1.38	2.03
O3	0.41 (1×→, 2×↓)	1.45	1.86
Σ	2.12	5.01	

reported by Mustafin & Kasenov (1994, 1996) has a doubled volume compared to the unit cell determined here, however, with no apparent metrical relation beyond this.

In the crystal structure of $Zn(AsO_3)_2$, the Zn atom is located on a twofold rotation axis (multiplicity 4, Wyckoff letter *e*), and the As and the three O atom sites on general positions (8*f*) of space group *C*2/*c*. The zinc atom in (**I**) exhibits a distorted octahedral coordination environment with an average Zn—O bond length of 2.090 Å (Table 1), in good agreement with the literature value of 2.110 (86) Å derived from 193 [ZnO_6] coordination polyhedra (Gagné & Hawthorne, 2020). The [ZnO_6] octahedron shares two of its edges with neighbouring octahedra, forming $^{1}_{\infty}$[$ZnO_{4/2}O_{2/1}$] zigzag-chains extending parallel to [001] (Fig. 1*a*). The arsenate tetrahedron condenses into a polyarsenate chain with a periodicity of two tetrahedra (Fig. 1*b*). The bond length distribution (Table 1) in this chain follows the usual pattern, with significantly longer As—O distances to the bridging oxygen atoms (O1, average 1.735 Å) than to the terminal O atoms (O2, O3; average 1.646 Å). The mean average As—O bond length within the AsO_4 tetrahedron (1.690 Å) complies with the literature value [1.687 (27) Å from 508 arsenate tetrahedra; Gagné & Hawthorne, 2018]. The terminal oxygen atoms of the polyarsenate chain are also the ones that bind to the zinc atoms in adjacent zinc oxide chains, so that the polyarsenate chains are aligned in the same direction as the zinc oxide chains, which leads to a very compact packing in this type of structure (Durif, 1995). In the crystal structure of (**I**) (Fig. 2), each zinc oxide chain is surrounded by six polyarsenate chains, and each polyarsenate chain is surrounded by three zinc oxide chains, corresponding to the 1:2 ratio of Zn: As in the formula.

The structure model was confirmed using bond-valence-sum calculations (Brown, 2002) performed with the *ECoN21* program (Ilinca, 2022). The calculated bond-valence sums (Table 2) are close to the expected values of 2, 5 and 2 valence units for Zn, As and O atoms, respectively, and the global instability index (GII) of 0.11 valence units indicates a stable and not particularly strained crystal structure (Salinas-Sanchez *et al.*, 1992; Brown, 2009).

Using the *compstru* program (de la Flor *et al.*, 2016) that is available at the Bilbao Crystallographic Server (Aroyo *et al.*, 2006), a quantitative comparison of the isotypic structures of low-temperature $Zn(PO_3)_2$ and $Zn(AsO_3)_2$ was made. The arithmetic mean of the distance between paired atoms is 0.1148 Å, and the absolute distances between paired atoms are 0.0925 Å for Zn1, 0.0598 Å for As1/P1, 0.0901 Å for O1, 0.1146 Å for O2, and the largest distance of 0.2057 Å for O3. The latter O atoms are those which, in both structures, have both the shortest Zn—O and the shortest *X*—O (*X* = As, P) bond lengths. The degree of lattice distortion (0.0264), and the low value for the measure of similarity (0.023) indicate a high degree of similarity for the two crystal structures.

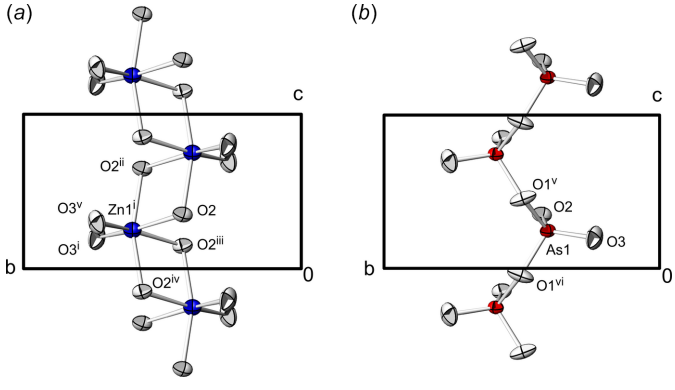

Figure 1
(*a*) One $^{1}_{\infty}$[$ZnO_{4/2}O_{2/1}$] zigzag chain, and (*b*) one polyarsenate chain crossing the unit cell of (**I**), as shown in projections down [$\bar{1}$00]. Displacement ellipsoids are drawn at the 97% probability level. [Symmetry codes: (i) $x+\frac{1}{2}, y+\frac{1}{2}, z$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x+1, y, -z+\frac{1}{2}$; (iv) $x, -y+1, z-\frac{1}{2}$; (v) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (vi) $-x+\frac{1}{2}, -y+\frac{1}{2}, -z$.]

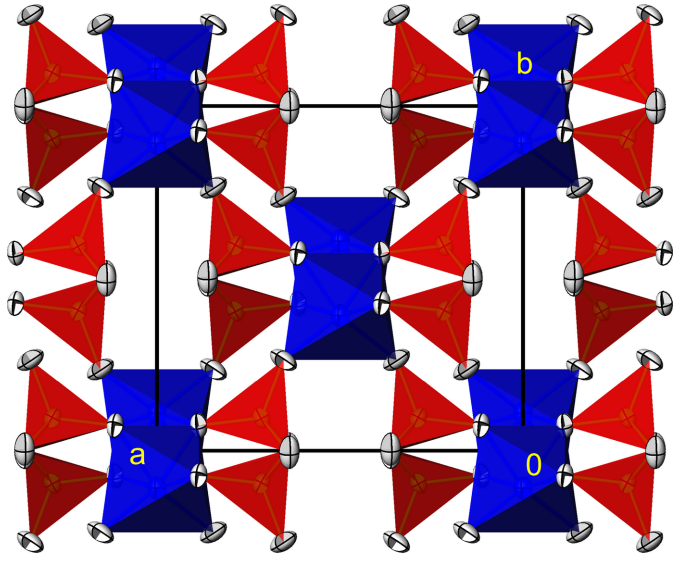

Figure 2
The crystal structure of (**I**) in polyhedral representation (ZnO_6 octahedra blue, AsO_4 tetrahedra red), viewed down [00$\bar{1}$]. Displacement ellipsoids are drawn at the 97% probability level.

3. Database survey

The Inorganic Crystal Structure Database (ICSD, data release 2025.1; Zagorac *et al.*, 2019) was used for the database search. Known structurally characterized phases in the $ZnO–As_2O_5$ system include the high- and low-temperature modifications of tetrazinc bis(arsenate(V)) oxide, $Zn_4(AsO_4)_2O$ (Frerichs *et al.*, 1996*a*), dimorphic trizinc orthoarsenate, $Zn_3(AsO_4)_2$ (Frerichs *et al.*, 1996*b*), and the pyroarsenate $Zn_2As_2O_7$ with three modifications existing at different temperatures, one of which is commensurately and one is incommensurately modulated (Weil & Stöger, 2010).

As already mentioned, $Zn(AsO_3)_2$ is isotypic with $Zn(PO_3)_2$, in its low-temperature modification (Averbuch-Pouchot *et al.*, 1983). The high-temperature modification of $Zn(PO_3)_2$ can be obtained by quenching from the recrystallized melt and contains a similar polyphosphate chain but in contrast to the low-temperature modification with two unique zinc cations in tetrahedral coordination (Weil, 2004). According to the information on the system $ZnO–As_2O_5$ provided by Kasenov *et al.* (1996), there are no indications of a possible high-temperature form of $Zn(AsO_3)_2$. Other $M(PO_3)_2$ polyphosphates isotypic with low-temperature $Zn(PO_3)_2$ and $Zn(AsO_3)_2$ can only be prepared by using high pressure (∼80 kbar) and temperatures (∼1270 K) and are reported for M = Ni, Mg, Cu, Co, Fe, Mn, and Cd (Bagieu-Beucher *et al.*, 1976).

$Zn(AsO_3)_2$ is the only arsenate phase with an M:As^V ratio of 1:2 where solely tetrahedral AsO_4 units are present in the polymeric arsenate anion. Except for M = Ba with a *catena*-arsenate anion consisting of AsO_4 tetrahedra and AsO_6 octahedra (Weil, 2016), all other MAs_2O_6 phases crystallize in the $PbSb_2O_6$ type of structure (Hill, 1987) in the trigonal space group type $P\bar{3}1m$. This structure type is also referred to as the rosiaite structure type after the mineral of this name (Basso *et al.*, 1996) and is based on a hexagonal array of O atoms with the As atom octahedrally coordinated in form of honeycomb layers. This structure type is realized for M = Ni, Co, Mn (Nakua & Greedan, 1995), Cd (Weil, 2001), Hg (Weil, 2000), Pd (Orosel & Jansen, 2006), Ca, Pb (Losilla *et al.*, 1995), and Sr. The strontium phase is dimorphic and as well as in a rosiaite-type structure also crystallizes in a hexagonal superstructure with a doubled unit cell (Weil *et al.*, 2009).

4. Synthesis and crystallization

Crystal growth procedures were carried out in sealed and evacuated silica ampoules using commercially available chemicals as received. A molar 1:1 mixture of ZnO (≥ 99%, Carl Roth GmbH) and As_2O_5 [99.9% (metals basis), abcr] was heated at 750 K for about 336 h. X-ray powder diffraction of the bulk product revealed $Zn(AsO_3OH)·H_2O$ (mineral name koritnigite; Keller *et al.*, 1980) and $Zn(AsO_3)_2$ as the main phases in an approximate ratio of 1:2. From this mixture, yellowish crystals of $Zn(AsO_3)_2$ were isolated for the single-crystal X-ray diffraction study. The presence of the hydrous phase $Zn(AsO_3OH)·H_2O$ indicates that the hygroscopic starting material As_2O_5 was indeed not free of water.

Table 3
Experimental details.

Crystal data	
Chemical formula	$[Zn(AsO_3)_2]$
M_r	311.21
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	300
a, *b*, *c* (Å)	10.1061 (10), 9.0308 (8), 5.2801 (5)
β (°)	108.374 (7)
V (Å^3)	457.33 (8)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	19.68
Crystal size (mm)	0.04 × 0.02 × 0.01
Data collection	
Diffractometer	Stoe Stadivari
Absorption correction	Multi-scan (*LANA*; Koziskova *et al.*, 2016)
T_{min}, T_{max}	0.492, 0.654
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	3846, 797, 770
R_{int}	0.017
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.762
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.010, 0.028, 1.13
No. of reflections	797
No. of parameters	43
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.40, −0.37

Computer programs: *X-AREA* (Folkers-Karlsson *et al.*, 2026), *SHELXL* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009), *ATOMS* (Dowty, 2006) and *publCIF* (Westrip, 2010).

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. Atomic coordinates and labelling were adapted from the isotypic low-temperature phase of $Zn(PO_3)_2$ (Averbuch-Pouchot *et al.*, 1983).

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$Zn(AsO_3)_2$, a unique structure among arsenates with composition $M^{II}As^{V}_2O_6$

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Computing details

***catena*-Poly[zinc-di-μ-arsenato(V)-κ^4O:O]**

Crystal data

$[Zn(AsO_3)_2]$
M_r = 311.21
Monoclinic, $C2/c$
a = 10.1061 (10) Å
b = 9.0308 (8) Å
c = 5.2801 (5) Å
β = 108.374 (7)°
V = 457.33 (8) Å^3
Z = 4
$F(000)$ = 576
D_x = 4.520 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5088 reflections
θ = 3.1–32.8°
μ = 19.68 mm^{-1}
T = 300 K
Shard, yellow
0.04 × 0.02 × 0.01 mm

Data collection

Stoe Stadivari diffractometer
Radiation source: Axo_Mo
Graded multilayer mirror monochromator
Detector resolution: 13.33 pixels mm^{-1}
rotation method, ω scans
Absorption correction: multi-scan (*LANA*; Koziskova *et al.*, 2016)
T_{min} = 0.492, T_{max} = 0.654
3846 measured reflections
797 independent reflections
770 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 32.8°, θ_{min} = 3.1°
h = −14→15
k = −12→13
l = −7→4

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.010
$wR(F^2)$ = 0.028
S = 1.13
797 reflections
43 parameters
0 restraints
Primary atom site location: isomorphous structure methods
$w = 1/[\sigma^2(F_o^2) + (0.0127P)^2 + 0.509P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}
Extinction correction: SHELXL (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0035 (2)

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}
Zn1	0.000000	0.10801 (2)	0.250000	0.00841 (6)
As1	0.21468 (2)	0.40539 (2)	0.24523 (3)	0.00640 (5)
O1	0.36157 (10)	−0.00460 (12)	0.04492 (19)	0.0121 (2)
O2	0.38566 (10)	0.42724 (11)	0.35206 (19)	0.00870 (17)
O3	0.15428 (11)	0.23634 (11)	0.1990 (2)	0.0132 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.00662 (11)	0.00985 (11)	0.00828 (10)	0.000	0.00168 (8)	0.000
As1	0.00579 (7)	0.00774 (8)	0.00560 (7)	−0.00148 (4)	0.00169 (5)	−0.00034 (4)
O1	0.0078 (4)	0.0209 (5)	0.0074 (4)	0.0001 (4)	0.0021 (3)	0.0056 (4)
O2	0.0057 (4)	0.0118 (4)	0.0077 (4)	−0.0008 (3)	0.0008 (3)	0.0008 (3)
O3	0.0139 (5)	0.0094 (4)	0.0172 (5)	−0.0052 (4)	0.0061 (4)	−0.0020 (4)

Geometric parameters (Å, º)

Zn1—O3	2.0276 (10)	Zn1—O2^{v}	2.1640 (10)
Zn1—O3^{i}	2.0276 (10)	As1—O3	1.6336 (10)
Zn1—O2^{ii}	2.0786 (10)	As1—O2	1.6518 (10)
Zn1—O2^{iii}	2.0786 (10)	As1—O1^{vi}	1.7319 (10)
Zn1—O2^{iv}	2.1640 (10)	As1—O1^{vii}	1.7379 (10)
O3—Zn1—O3^{i}	110.28 (6)	O2^{iii}—Zn1—O2^{v}	88.05 (3)
O3—Zn1—O2^{ii}	99.15 (4)	O2^{iv}—Zn1—O2^{v}	82.06 (5)
O3^{i}—Zn1—O2^{ii}	90.92 (4)	O3—As1—O2	117.65 (5)
O3—Zn1—O2^{iii}	90.92 (4)	O3—As1—O1^{vi}	108.08 (5)
O3^{i}—Zn1—O2^{iii}	99.15 (4)	O2—As1—O1^{vi}	111.48 (5)
O2^{ii}—Zn1—O2^{iii}	162.38 (6)	O3—As1—O1^{vii}	108.20 (5)
O3—Zn1—O2^{iv}	163.24 (4)	O2—As1—O1^{vii}	110.65 (5)
O3^{i}—Zn1—O2^{iv}	84.54 (4)	O1^{vi}—As1—O1^{vii}	99.15 (3)
O2^{ii}—Zn1—O2^{iv}	88.05 (3)	As1^{vi}—O1—As1^{v}	130.01 (6)
O2^{iii}—Zn1—O2^{iv}	78.64 (4)	As1—O2—Zn1^{iii}	121.02 (5)
O3—Zn1—O2^{v}	84.54 (4)	As1—O2—Zn1^{viii}	126.04 (5)
O3^{i}—Zn1—O2^{v}	163.24 (4)	Zn1^{iii}—O2—Zn1^{viii}	101.35 (4)
O2^{ii}—Zn1—O2^{v}	78.64 (4)	As1—O3—Zn1	141.36 (6)

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