New M^+, M^{3+}-arsenates – the framework structures of $AgM^{3+}(HAsO_4)_2$ (M^{3+} = Al, Ga) and $M^+GaAs_2O_7$ (M^+ = Na, Ag)

Karolina Schwendtner[a]* and Uwe Kolitsch[b,c]

[a]Institute for Chemical Technology and Analytics, Division of Structural Chemistry, TU Wien, Getreidemarkt 9/164-SC, 1060 Wien, Austria, [b]Naturhistorisches Museum Wien, Burgring 7, 1010 Wien, Austria, and [c]Institute for Mineralogy and Crystallography, University of Vienna, Althanstrasse 14, 1090 Wien, Austria. *Correspondence e-mail: karolina.schwendtner@tuwien.ac.at

The crystal structures of hydrothermally synthesized silver(I) aluminium bis[hydrogen arsenate(V)], $AgAl(HAsO_4)_2$, silver(I) gallium bis[hydrogen arsenate(V)], $AgGa(HAsO_4)_2$, silver gallium diarsenate(V), $AgGaAs_2O_7$, and sodium gallium diarsenate(V), $NaGaAs_2O_7$, were determined from single-crystal X-ray diffraction data collected at room temperature. The first two compounds are representatives of the MCV-3 structure type known for $KSc(HAsO_4)_2$, which is characterized by a three-dimensional anionic framework of corner-sharing alternating $M^{3+}O_6$ octahedra (M = Al, Ga) and singly protonated AsO_4 tetrahedra. Intersecting channels parallel to [101] and [110] host the Ag^+ cations, which are positionally disordered in the Ga compound, but not in the Al compound. The hydrogen bonds are relatively strong, with O···O donor–acceptor distances of 2.6262 (17) and 2.6240 (19) Å for the Al and Ga compounds, respectively. The two diarsenate compounds are representatives of the $NaAlAs_2O_7$ structure type, characterized by an anionic framework topology built of $M^{3+}O_6$ octahedra (M = Al, Ga) sharing corners with diarsenate groups, and M^+ cations (M = Ag) hosted in the voids of the framework. Both structures are characterized by a staggered conformation of the As_2O_7 groups.

1. Chemical context

Metal arsenates often form tetrahedral–octahedral framework structures with potentially interesting properties, such as ion conductivity, ion exchange and catalytic properties (Masquelier *et al.*, 1990, 1994*a*,*b*, 1995, 1996; Medvedev *et al.*, 2003; Ouerfelli *et al.*, 2007*a*, 2008; Pintard-Scrépel *et al.*, 1983; Rousse *et al.*, 2013). A detailed study of the system $M^+–M^{3+}–$As–O–(H) was therefore conducted, and a wide variety of new compounds and structure types were found and have been published (Schwendtner, 2006; Schwendtner & Kolitsch, 2004*a*,*b*, 2005, 2007*a*,*b*,*c*,*d*). Thus far, three different structure types for $M^{1+}M^{3+}(HAsO_4)_2$ compounds have been reported. Two of them are very common and were first described for isotypic phosphate compounds. The $(H_3O)Fe(HPO_4)_2$ type (Vencato *et al.*, 1989) is also adopted by β-$CsSc(HAsO_4)_2$ (Schwendtner & Kolitsch, 2004*b*), while the $(NH_4)Fe(HPO_4)_2$ type (Yakubovich, 1993) is adopted by α-$CsSc(HAsO_4)_2$ (Schwendtner & Kolitsch, 2004) and $(NH_4)Fe(HAsO_4)_2$ (Ouerfelli *et al.*, 2014). The $KSc(HAsO_4)_2$ type (Schwendtner & Kolitsch, 2004*a*) has so far been the only known example; the two new protonated arsenates presented here are two further representatives of this structure type.
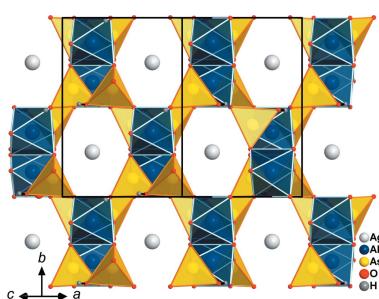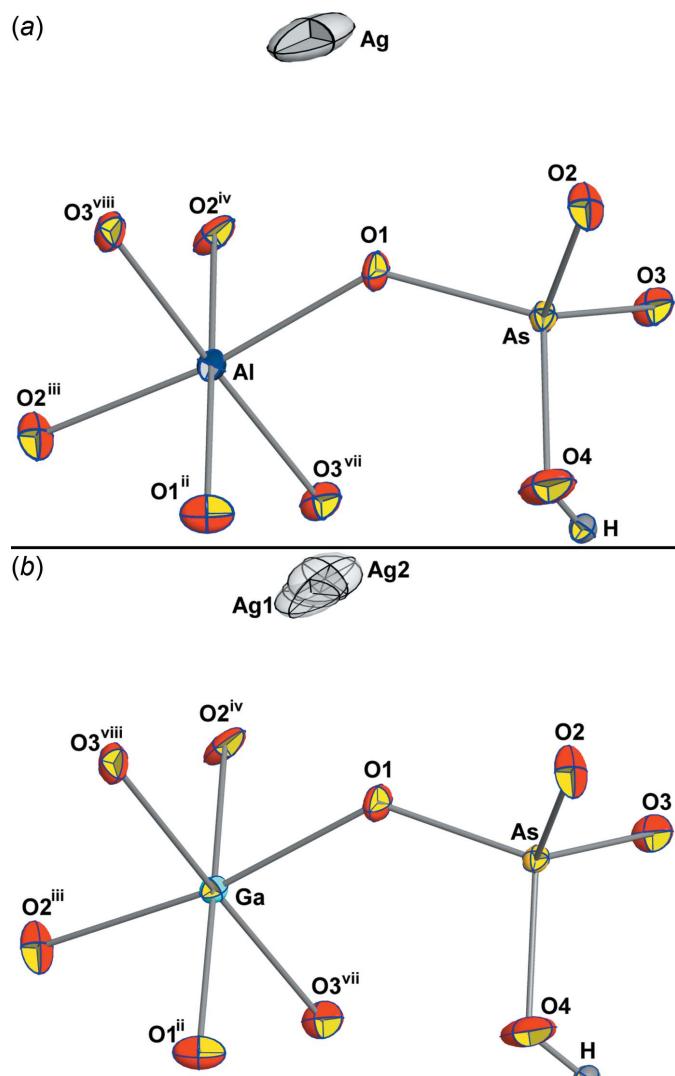
Figure 1
The principal building units of (a) AgAl(HAsO$_4$)$_2$ and (b) AgGa-(HAsO$_4$)$_2$, shown as displacement ellipsoids at the 70% probability level. The H atom is shown as a sphere with arbitrary radius. Part (b) shows the split position for Ag in AgGa(HAsO$_4$)$_2$. [Symmetry codes: (ii) $-x, y, -z + \frac{1}{2}$; (iii) $x + \frac{1}{2}, y + \frac{1}{2}, z$; (iv) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$; (vii) $-x + \frac{1}{2}, -y + \frac{1}{2}, -z$; (viii) $x + \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$.]

M^+ScAs$_2$O$_7$ with M^+ = NH$_4$ (Kolitsch, 2004), Rb (Schwendtner & Kolitsch, 2004a), and Tl (Baran *et al.*, 2006), as well as CsCrAs$_2$O$_7$ (Bouhassine & Boughzala, 2015), and the mixed (Al/Cr) compound K(Al$_{0.75}$Cr$_{0.25}$)As$_2$O$_7$ (Bouhassine & Boughzala, 2017). The NaAlAs$_2$O$_7$ type (Driss & Jouini, 1994) is also known for AgFeAs$_2$O$_7$ and NaFeAs$_2$O$_7$ (Ouerfelli *et al.*, 2004), and the $M1^{2+}M2^{2+}$ representative CaCuAs$_2$O$_7$ (Chen & Wang, 1996). The two diarsenates presented here also adopt the NaAlAs$_2$O$_7$ structure type.

2. Structural commentary

AgAl(HAsO$_4$)$_2$ and AgGa(HAsO$_4$)$_2$ are isotypic and crystallize in the monoclinic ($C2/c$) microporous framework structure of KSc(HAsO$_4$)$_2$ (Schwendtner & Kolitsch, 2004a). The asymmetric unit contains one Ag, one Ga/Al, one As, four O and one H sites (Fig. 1). The Al/Ga atoms lie on a special position (twofold rotation axis) and the Ag$^+$ cation is situated

M^+M^{3+}As$_2$O$_7$ compounds crystallize in six known structure types, some of them isotypic to phosphates or silicates. The CaZrSi$_2$O$_7$ type (mineral gittinsite; Roelofsen-Ahl & Peterson, 1989) is also adopted by LiFeAs$_2$O$_7$ (Wang *et al.*, 1994), LiM^{3+}As$_2$O$_7$ (M^{3+} = Al, Ga, Sc) and NaScAs$_2$O$_7$ (Schwendtner & Kolitsch, 2007d). The NaInAs$_2$O$_7$ type (Belam *et al.*, 1997) has no other known members. The TlInAs$_2$O$_7$ type (M^+ = Tl, Rb, NH$_4$) (Schwendtner, 2006) is also adopted by KFeAs$_2$O$_7$ (Ouerfelli *et al.*, 2007b). The RbAlAs$_2$O$_7$ type (Boughzala *et al.*, 1993) is also known for M^{1+} = Tl, Cs (Boughzala & Jouini, 1992), M^{1+} = K (Boughzala & Jouini, 1995), and is further represented by KGaAs$_2$O$_7$ (Lin & Lii, 1996), KCrAs$_2$O$_7$ (Siegfried *et al.*, 2004) and the mixed (Al/Fe) compound TlAl$_{0.78}$Fe$_{0.22}$As$_2$O$_7$ (Ouerfelli *et al.*, 2007a). The KAlP$_2$O$_7$ type is extremely common for phosphates and also has five examples that are arsenates,

Figure 2
View of the framework structure of (a) AgAl(HAsO$_4$)$_2$ along [101] and (b) isotypic AgGa(HAsO$_4$)$_2$ along [110]. The M^{3+}O$_6$ octahedra (M = Al, Ga) are corner-linked to HAsO$_4$ tetrahedra. Both views show small micropores in which the Ag$^+$ cations are situated; the split Ag position (see text) and hydrogen bonds (dashed lines) are indicated in (b).

Table 1
Selected bond lengths (Å) for AgAl(HAsO₄)₂.

Ag1—O1	2.4040 (11)	Al—O3^{iv}	1.9167 (10)
Ag1—O3^{i}	2.6362 (11)	As—O2	1.6683 (9)
Ag1—O4^{ii}	2.8202 (13)	As—O1	1.6697 (10)
Ag1—O2	3.1259 (11)	As—O3	1.6710 (11)
Al—O1	1.8920 (10)	As—O4	1.7161 (10)
Al—O2^{iii}	1.8955 (11)		

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

Table 2
Hydrogen-bond geometry (Å, °) for AgAl(HAsO₄)₂.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O4—H$\cdots$O2^{v}	0.89 (1)	1.81 (2)	2.6212 (17)	151 (3)

Symmetry code: (v) $x, -y+1, z+\frac{1}{2}$.

Table 3
Selected bond lengths (Å) for AgGa(HAsO₄)₂.

Ag1—O1	2.3600 (12)	Ag2—O2^{iii}	3.116 (18)
Ag1—O3^{i}	2.5864 (12)	Ag2—O2	3.185 (17)
Ag1—O4^{ii}	3.0574 (15)	Ga—O1	1.9591 (11)
Ag1—O2	3.1352 (12)	Ga—O2^{vi}	1.9641 (11)
Ag2—O1	2.357 (17)	Ga—O3^{vii}	1.9799 (12)
Ag2—O1^{iii}	2.402 (18)	As—O2	1.6719 (11)
Ag2—O3^{i}	2.48 (2)	As—O3	1.6753 (12)
Ag2—O3^{iv}	2.73 (3)	As—O1	1.6773 (11)
Ag2—O4^{v}	2.83 (2)	As—O4	1.7096 (12)

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

Table 4
Hydrogen-bond geometry (Å, °) for AgGa(HAsO₄)₂.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O4—H$\cdots$O2^{viii}	0.80 (3)	1.89 (3)	2.6240 (19)	153 (3)

Symmetry code: (viii) $x, -y+1, z+\frac{1}{2}$.

on an inversion centre. In the case of AgGa(HAsO₄)₂, the Ag$^+$ cation occupies a split position (Fig. 1b), where Ag1 sits on the inversion centre, with a freely refined occupancy of 0.75 (2). The second site (Ag2) is only 0.31 (3) Å away from Ag1 and has a freely refined occupancy of 0.123 (10). In total, this leads to a composition of one Ag atom per formula unit. The slightly distorted M^{3+}O₆ octahedra share corners with six hydrogen arsenate tetrahedra to form a three-dimensional, anionic framework structure with narrow irregular channels parallel to [101] and [110] (Fig. 2), which host the Ag$^+$ cations. The latter show a rather irregular [6 + 2]-coordination in both compounds. However, it is well known that Ag atoms tolerate a broad spectrum of coordination spheres (Müller-Buschbaum, 2004).

The protonated apex of the AsO₄ tetrahedron is involved in a relatively strong hydrogen bond with O4$\cdots$O2(x, $-y+1$, $z+\frac{1}{2}$) = 2.6212 (17) and 2.6240 (19) Å for the Al (Table 2) and Ga compounds (Table 4), respectively, which runs roughly perpendicular to the (101) plane (Fig. 2). The As—OH bond lengths (Tables 1 and 3) are considerably elongated in comparison to the three remaining As—O bonds (Ferraris & Ivaldi, 1984), but at 1.7161 (10) Å for the Al and 1.7096 (12) Å for the Ga compound are slightly shorter than the average As—OH bond length in HAsO₄²⁻ anions of 1.72 (3) Å (Schwendtner, 2008). The bonds to non-protonated O ligands are shorter than the average mean As—O bond length for inorganic arsenates (1.686 Å; Schwendtner, 2008), and fit well with the data for As bonds to non-protonated O atoms in H₁₋₃AsO₄ groups [average 1.669 Å, Schwendtner, 2008; 1.670 Å, AgAl(HAsO₄)₂; 1.675 Å, AgGa(HAsO₄)₂]. The average bond lengths for the AlO₆ and GaO₆ octahedra agree well with published averages (Baur, 1981; Overweg et al., 1999). Bond-valence sums were calculated using the bond-valence parameters of Brown & Altermatt (1985), and amount to 0.88/0.90 (Ag1), 0.89 (Ag2), 3.05/3.16 (Al/Ga), 5.05/5.02 (As), 2.04/2.06 (O1), 1.85/1.85 (O2), 1.90/2.01 (O3) and 1.22/1.21 (O4 = OH; H atom not considered for calculation) valence units for AgAl(HAsO₄)₂ and AgGa(HAsO₄)₂, respectively. These results are reasonably close to the ideal values; the underbonded O2 is an acceptor of the strong hydrogen bond. Compared to isotypic KSc(HAsO₄)₂, the cell lengths and the hydrogen bonds of the two new compounds are considerably shorter. As a result of the similar ionic radii of Al³⁺ and Ga³⁺, these two compounds have similar unit-cell parameters. The Ga compound is slightly compressed along the a axis (smaller β), but elongated along the b axis; the c axis is not affected.

The two diarsenates AgGaAs₂O₇ and NaGaAs₂O₇ crystallize in space group $P2_1/c$ and are isotypic to NaAlAs₂O₇ (Driss & Jouini, 1994). The asymmetric unit contains one Ag/Na, one Ga, two As and seven O sites, all of which occupy general positions (Fig. 3). The basic building block is an As₂O₇ group, which is connected to the GaO₆ octahedra by two corners. The other four free O ligands are connected to different GaO₆ octahedra to form a three-dimensional framework structure (Fig. 4). The As2—O$_{bridge}$ distances [1.755 (3), 1.756 (3) Å] are nearly identical to the literature value of 1.755 Å for the average As—O$_{bridge}$ bond length in diarsenates (Schwendtner & Kolitsch, 2007d), whereas the As1—O$_{bridge}$ distances [1.781 (3), 1.777 (3) Å] are further elongated. The average bond lengths of both AsO₄ tetrahedra are slightly longer than the literature value of 1.688 Å (Schwendtner & Kolitsch, 2007d) for the average As—O bond length in As₂O₇ groups.
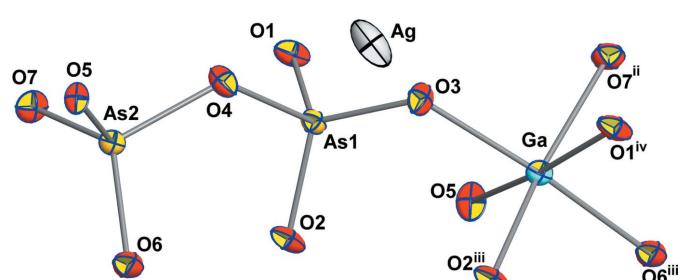

Figure 3
The principal building units of AgGaAs₂O₇ shown as displacement ellipsoids at the 70% probability level. [Symmetry codes: (ii) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x, -y, -z$; (iv) $x, -y-\frac{1}{2}, z-\frac{1}{2}$.]

(a)

(b)
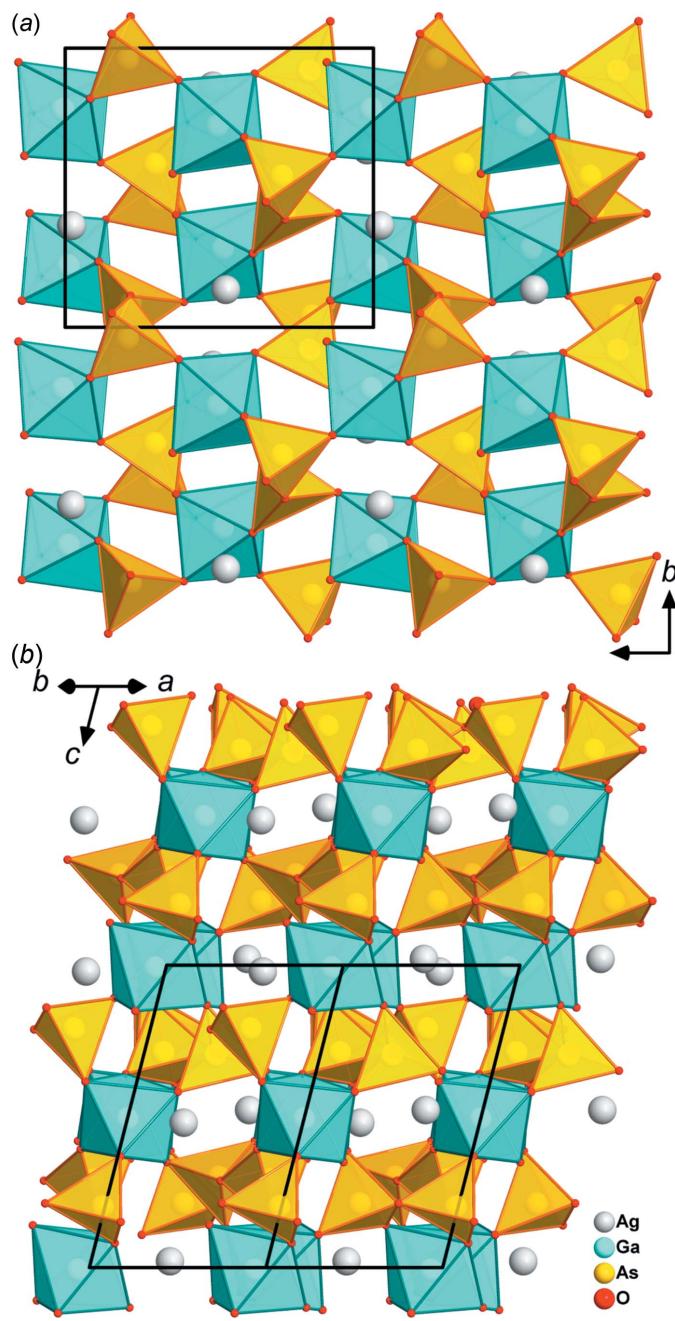
Figure 4
View of the framework structure of AgGaAs$_2$O$_7$ along (*a*) [100] and (*b*) [110].

The As—O—As angle is very close to the average value of As$_2$O$_7$ groups in a staggered conformation (124.2°, Schwendtner & Kolitsch, 2007*d*) for both compounds (see Tables 5 and 6).

The GaO$_6$ octahedra are slightly distorted and the average Ga—O bond lengths are close to the literature values (~1.96 Å; Overweg *et al.*, 1999). The Na$^+$ and Ag$^+$ cations show a strongly distorted octahedral coordination. The calculated bond-valence sums using the parameters of Brown & Altermatt (1985) for Ag, Ga and As, and Wood & Palenik (1999) for Na, amount to 1.06/1.00 (Ag/Na), 3.11/3.11 (Ga),

Table 5
Selected geometric parameters (Å, °) for AgGa(As$_2$O$_7$).

Ag—O1^i	2.353 (3)	Ga—O6^v	1.985 (3)
Ag—O6^{ii}	2.361 (3)	Ga—O7^i	2.015 (3)
Ag—O3	2.440 (3)	As1—O2	1.653 (3)
Ag—O7^{iii}	2.529 (3)	As1—O1	1.668 (3)
Ag—O7^{iv}	2.600 (3)	As1—O3	1.679 (3)
Ag—O4	2.766 (3)	As1—O4	1.781 (3)
Ga—O2^v	1.939 (3)	As2—O5^{vi}	1.655 (3)
Ga—O3	1.957 (3)	As2—O7	1.674 (3)
Ga—O1^{iii}	1.973 (3)	As2—O6	1.675 (3)
Ga—O5	1.975 (3)	As2—O4	1.755 (3)
As2—O4—As1	124.65 (15)		

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

Table 6
Selected geometric parameters (Å, °) for NaGa(As$_2$O$_7$).

Na—O1^i	2.289 (4)	Ga—O6^v	1.987 (3)
Na—O6^{ii}	2.360 (4)	Ga—O7^i	2.042 (3)
Na—O3	2.364 (4)	As1—O2	1.655 (3)
Na—O7^{iii}	2.468 (4)	As1—O1	1.664 (3)
Na—O7^{iv}	2.479 (4)	As1—O3	1.676 (3)
Na—O4	2.624 (4)	As1—O4	1.777 (3)
Ga—O2^v	1.940 (3)	As2—O5^{vi}	1.661 (3)
Ga—O1^{iii}	1.952 (3)	As2—O6	1.671 (3)
Ga—O3	1.960 (3)	As2—O7	1.678 (3)
Ga—O5	1.967 (3)	As2—O4	1.756 (3)
As2—O4—As1	124.73 (19)		

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

4.90/4.93 (As1), 4.96/4.93 (As2), 2.08/2.11 (O1), 1.93/1.92 (O2), 2.01/2.01 (O3), 2.08/2.10 (O4), 1.87/1.86 (O5), 2.03/1.99 (O6), and 2.03/1.99 (O7) valence units for AgGaAs$_2$O$_7$ and NaGaAs$_2$O$_7$, respectively.

3. Synthesis and crystallization

The compounds were grown by hydrothermal synthesis at 493 K (7 d, autogeneous pressure, slow furnace cooling) using Teflon-lined stainless-steel autoclaves with an approximate filling volume of 2 cm^3. Reagent-grade Ag$_2$CO$_3$, Ga$_2$O$_3$, α-Al$_2$O$_3$, Na$_2$CO$_3$, and H$_3$AsO$_4$·0.5H$_2$O were used as starting reagents in approximate volume ratios of M^+:M^{3+}:As of 1:1:2. The vessels were filled with distilled water to about 70% of their inner volumes, which led to final pH values of < 1 for all synthesis batches except NaGaAs$_2$O$_7$ (pH 1.5). The reaction products were washed thoroughly with distilled water, filtered and dried at room temperature.

The two hydrogen arsenates formed pseudo-dipyramidal colourless crystals up to 2 mm in length (yield > 95% for the Al compound with minor amounts of AgCl, explained by the reaction of Ag$^+$ with remnants of concentrated hot HCl used for standard cleaning of the Teflon vessels before each new experiment; Fig. 5*a*). The crystals of AgGa(HAsO$_4$)$_2$ (Fig. 5*c*) were accompanied by about 20% of AgGaAs$_2$O$_7$ as pseudo-orthorhombic platelets (Fig. 5*d*). NaGaAs$_2$O$_7$ crystallized as small, colourless platelets with a diamond-shaped outline (yield 100%) (Fig. 5*b*).

Table 7
Experimental details.

	AgAl(HAsO$_4$)$_2$	AgGa(HAsO$_4$)$_2$	AgGa(AsO$_7$)	NaGa(As$_2$O$_7$)
Crystal data				
M_r	414.71	457.45	354.55	439.43
Crystal system, space group	Monoclinic, $C2/c$	Monoclinic, $C2/c$	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/c$
Temperature (K)	293	293	293	293
a, b, c (Å)	7.842 (2), 9.937 (2), 8.686 (2)	7.826 (2), 10.216 (2), 8.694 (2)	6.987 (1), 8.266 (2), 9.677 (2)	7.049 (1), 8.368 (2), 9.735 (2)
β (°)	108.45 (3)	107.77 (3)	107.50 (3)	108.47 (3)
V (Å^3)	642.1 (3)	661.9 (3)	533.0 (2)	544.7 (2)
Z	4	4	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	13.51	16.96	17.55	20.58
Crystal size (mm)	0.15 × 0.08 × 0.07	0.18 × 0.10 × 0.10	0.07 × 0.07 × 0.02	0.10 × 0.08 × 0.02
Data collection				
Diffractometer	Nonius KappaCCD single-crystal four-circle	Nonius KappaCCD single-crystal four-circle	Nonius KappaCCD single-crystal four-circle	Nonius KappaCCD single-crystal four-circle
Absorption correction	Multi-scan (*HKL SCALEPACK*; Otwinowski *et al.*, 2003)	Multi-scan (*HKL SCALEPACK*; Otwinowski *et al.*, 2003)	Multi-scan (*HKL SCALEPACK*; Otwinowski *et al.*, 2003)	Multi-scan (*HKL SCALEPACK*; Otwinowski *et al.*, 2003)
T_{min}, T_{max}	0.236, 0.451	0.150, 0.282	0.373, 0.720	0.233, 0.684
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	2751, 1408, 1354	2852, 1460, 1402	3015, 1557, 1336	3849, 1982, 1775
R_{int}	0.011	0.012	0.023	0.022
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.806	0.806	0.704	0.757
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.014, 0.037, 1.10	0.015, 0.037, 1.15	0.033, 0.090, 1.04	0.033, 0.089, 1.05
No. of reflections	1408	1460	1557	1982
No. of parameters	62	73	100	100
No. of restraints	1	0	0	0
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	All H-atom parameters refined	–	–
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.55, −0.60	0.66, −0.50	1.54, −1.49	2.29, −2.38

Computer programs: *COLLECT* (Nonius, 2003), *HKL DENZO* and *SCALEPACK* (Otwinowski *et al.*, 2003), *SHELXS97* (Sheldrick, 2008), *SHELXL2016* (Sheldrick, 2015), *DIAMOND* (Brandenburg, 2005) and *publCIF* (Westrip, 2010).

Measured X-ray powder diffraction diagrams of the NaGaAs$_2$O$_7$ and AgAl(HAsO$_4$)$_2$ synthesis batches were deposited at the International Centre for Diffraction Data under PDF number 57-0162 (Prem *et al.*, 2005) for NaGaAs$_2$O$_7$ and 57-0161 (Prem *et al.*, 2005) for AgAl(HAsO$_4$)$_2$.

4. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 7.

For easier comparison, the atomic positions of the isotypic compounds KSc(HAsO$_4$)$_2$ (Schwendtner & Kolitsch, 2004) and NaAlAs$_2$O$_7$ (Driss & Jouini, 1994) were used for the final refinement. The O—H distance was restrained to 0.9 (2) Å for AgAl(HAsO$_4$)$_2$ and refined freely for AgGa(HAsO$_4$)$_2$.

Remaining electron densities are below 1 e Å^{-3} for the two hydrogen arsenates. The remaining maximum and minimum electron densities in the final difference-Fourier maps are located close to the As1 atom (0.87/0.78 Å) for NaGaAs$_2$O$_7$, and close to the Ag atom (0.73/0.66 Å) for AgGaAs$_2$O$_7$.

Figure 5
SEM micrographs of hydrothermally synthesized crystals of (*a*) AgAl(HAsO$_4$)$_2$, (*b*) NaGaAs$_2$O$_7$, (*c*) AgGa(HAsO$_4$)$_2$ and (*d*) AgGaAs$_2$O$_7$.

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research communications

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New M^+, M^{3+}-arsenates – the framework structures of $AgM^{3+}(HAsO_4)_2$ (M^{3+} = Al, Ga) and $M^+GaAs_2O_7$ (M^+ = Na, Ag)

Karolina Schwendtner and Uwe Kolitsch

Computing details

For all compounds, data collection: *COLLECT* (Nonius, 2003); cell refinement: *HKL SCALEPACK* (Otwinowski *et al.*, 2003); data reduction: *HKL DENZO* and *SCALEPACK* (Otwinowski *et al.*, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2016* (Sheldrick, 2015); molecular graphics: *DIAMOND* (Brandenburg, 2005); software used to prepare material for publication: *publCIF* (Westrip, 2010).

(AgAlHAsO42) Silver(I) aluminium bis[hydrogen arsenate(V)]

Crystal data

$AgAl(HAsO_4)_2$	$F(000) = 768$
$M_r = 414.71$	$D_x = 4.290$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 7.842$ (2) Å	Cell parameters from 1477 reflections
$b = 9.937$ (2) Å	$\theta = 2.0$–35.0°
$c = 8.686$ (2) Å	$\mu = 13.51$ mm^{-1}
$\beta = 108.45$ (3)°	$T = 293$ K
$V = 642.1$ (3) Å^3	Pseudo-dipyramidal glassy, colourless
$Z = 4$	$0.15 \times 0.08 \times 0.07$ mm

Data collection

Nonius KappaCCD single-crystal four-circle diffractometer	1408 independent reflections
	1354 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.011$
φ and ω scans	$\theta_{max} = 34.9°$, $\theta_{min} = 3.4°$
Absorption correction: multi-scan (HKL SCALEPACK; Otwinowski *et al.*, 2003)	$h = -12 \rightarrow 12$
	$k = -16 \rightarrow 16$
$T_{min} = 0.236$, $T_{max} = 0.451$	$l = -13 \rightarrow 13$
2751 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.014$	Hydrogen site location: difference Fourier map
$wR(F^2) = 0.037$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.10$	
1408 reflections	$w = 1/[\sigma^2(F_o^2) + (0.018P)^2 + 0.890P]$ where $P = (F_o^2 + 2F_c^2)/3$
62 parameters	
1 restraint	$(\Delta/\sigma)_{max} = 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.55$ e Å^{-3}
	$\Delta\rho_{min} = -0.60$ e Å^{-3}

Extinction correction: SHELXL2016
(Sheldrick, 2015),
$Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0114 (3)

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}
Ag1	0.250000	0.250000	0.000000	0.03518 (7)
Al	0.000000	0.13916 (5)	0.250000	0.00554 (9)
As	0.27443 (2)	0.40001 (2)	0.35945 (2)	0.00508 (5)
O1	0.17964 (13)	0.26607 (10)	0.24940 (12)	0.00972 (16)
O2	0.32668 (13)	0.50141 (10)	0.22800 (11)	0.00973 (16)
O3	0.44856 (12)	0.35479 (10)	0.51919 (11)	0.00870 (15)
O4	0.12010 (13)	0.48544 (12)	0.42496 (12)	0.01492 (19)
H	0.153 (4)	0.487 (3)	0.5324 (12)	0.047 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.06317 (16)	0.02460 (10)	0.03540 (12)	−0.01037 (9)	0.04068 (11)	−0.00702 (8)
Al	0.0059 (2)	0.0053 (2)	0.0052 (2)	0.000	0.00145 (16)	0.000
As	0.00555 (6)	0.00526 (7)	0.00416 (6)	−0.00071 (3)	0.00115 (4)	0.00007 (3)
O1	0.0112 (4)	0.0086 (4)	0.0102 (4)	−0.0057 (3)	0.0045 (3)	−0.0046 (3)
O2	0.0113 (4)	0.0100 (4)	0.0069 (3)	−0.0043 (3)	0.0014 (3)	0.0027 (3)
O3	0.0087 (3)	0.0117 (4)	0.0045 (3)	0.0024 (3)	0.0004 (3)	0.0009 (3)
O4	0.0113 (4)	0.0234 (5)	0.0104 (4)	0.0070 (4)	0.0038 (3)	−0.0026 (4)

Geometric parameters (Å, º)

Ag1—O1[i]	2.4040 (11)	Al—O2[vii]	1.8955 (11)
Ag1—O1	2.4040 (11)	Al—O2[v]	1.8955 (11)
Ag1—O3[ii]	2.6362 (11)	Al—O3[viii]	1.9167 (10)
Ag1—O3[iii]	2.6362 (11)	Al—O3[iii]	1.9167 (10)
Ag1—O4[iv]	2.8202 (13)	As—O2	1.6683 (9)
Ag1—O4[v]	2.8202 (13)	As—O1	1.6697 (10)
Ag1—O2	3.1259 (11)	As—O3	1.6710 (11)
Ag1—O2[i]	3.1259 (11)	As—O4	1.7161 (10)
Al—O1	1.8920 (10)	O4—H	0.886 (10)
Al—O1[vi]	1.8920 (10)		
O1—Al—O1[vi]	96.40 (7)	O1[vi]—Al—O3[iii]	94.10 (5)
O1—Al—O2[vii]	172.66 (4)	O2[vii]—Al—O3[iii]	90.59 (5)

O1^{vi}—Al—O2^{vii}	88.33 (5)	O2^{v}—Al—O3^{iii}	92.00 (5)
O1—Al—O2^{v}	88.33 (5)	O3^{viii}—Al—O3^{iii}	176.41 (7)
O1^{vi}—Al—O2^{v}	172.66 (4)	O2—As—O1	104.53 (5)
O2^{vii}—Al—O2^{v}	87.54 (7)	O2—As—O3	114.68 (5)
O1—Al—O3^{viii}	94.10 (5)	O1—As—O3	111.09 (5)
O1^{vi}—Al—O3^{viii}	83.49 (5)	O2—As—O4	106.25 (6)
O2^{vii}—Al—O3^{viii}	92.00 (5)	O1—As—O4	110.56 (5)
O2^{v}—Al—O3^{viii}	90.59 (5)	O3—As—O4	109.54 (5)
O1—Al—O3^{iii}	83.49 (5)		

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

Hydrogen-bond geometry (Å, º)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O4—H⋯O2^{ix}	0.89 (1)	1.81 (2)	2.6212 (17)	151 (3)

Symmetry code: (ix) x, −y+1, z+1/2.

(AgGaHAsO42) Silver(I) gallium bis[hydrogen arsenate(V)]

Crystal data

AgGa(HAsO$_4$)$_2$	$F(000) = 840$
$M_r = 457.45$	$D_x = 4.590$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 7.826$ (2) Å	Cell parameters from 1519 reflections
$b = 10.216$ (2) Å	$\theta = 2.0$–35.0°
$c = 8.694$ (2) Å	$\mu = 16.96$ mm^{-1}
$\beta = 107.77$ (3)°	$T = 293$ K
$V = 661.9$ (3) Å^3	Large pseudo-dipyramidal glassy, colourless
$Z = 4$	0.18 × 0.10 × 0.10 mm

Data collection

Nonius KappaCCD single-crystal four-circle diffractometer	1460 independent reflections
	1402 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.012$
φ and ω scans	$\theta_{max} = 35.0°$, $\theta_{min} = 3.4°$
Absorption correction: multi-scan (HKL SCALEPACK; Otwinowski *et al.*, 2003)	$h = -12 \rightarrow 12$
	$k = -16 \rightarrow 16$
$T_{min} = 0.150$, $T_{max} = 0.282$	$l = -14 \rightarrow 13$
2852 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.015$	Hydrogen site location: difference Fourier map
$wR(F^2) = 0.037$	All H-atom parameters refined
$S = 1.15$	$w = 1/[\sigma^2(F_o^2) + (0.015P)^2 + 0.940P]$
1460 reflections	where $P = (F_o^2 + 2F_c^2)/3$
73 parameters	$(\Delta/\sigma)_{max} = 0.037$
0 restraints	$\Delta\rho_{max} = 0.66$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{min} = -0.50$ e Å^{-3}

Extinction correction: SHELXL2016 (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$ Extinction coefficient: 0.0133 (3)

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
Ag1	0.250000	0.250000	0.000000	0.0335 (9)	0.75 (2)
Ag2	0.284 (3)	0.2351 (14)	0.019 (2)	0.0316 (18)	0.123 (10)
Ga	0.000000	0.13670 (2)	0.250000	0.00500 (5)	
As	0.27567 (2)	0.39556 (2)	0.35733 (2)	0.00482 (5)	
O1	0.18942 (15)	0.26190 (10)	0.24943 (13)	0.00983 (18)	
O2	0.32045 (15)	0.49738 (11)	0.22405 (13)	0.01055 (19)	
O3	0.45491 (14)	0.35629 (11)	0.51333 (13)	0.00948 (18)	
O4	0.11608 (16)	0.46896 (14)	0.42534 (15)	0.0169 (2)	
H	0.148 (4)	0.487 (3)	0.519 (4)	0.033 (8)*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.051 (2)	0.0368 (14)	0.0271 (9)	−0.0173 (12)	0.0327 (12)	−0.0125 (9)
Ag2	0.049 (4)	0.0249 (16)	0.035 (3)	−0.015 (2)	0.034 (3)	−0.0064 (14)
Ga	0.00555 (9)	0.00478 (9)	0.00482 (9)	0.000	0.00182 (6)	0.000
As	0.00564 (7)	0.00525 (7)	0.00364 (7)	−0.00073 (4)	0.00153 (5)	−0.00003 (4)
O1	0.0114 (4)	0.0093 (4)	0.0101 (5)	−0.0057 (3)	0.0053 (4)	−0.0045 (3)
O2	0.0122 (4)	0.0110 (4)	0.0068 (4)	−0.0060 (3)	0.0003 (3)	0.0036 (4)
O3	0.0085 (4)	0.0135 (4)	0.0053 (4)	0.0032 (3)	0.0004 (3)	0.0011 (4)
O4	0.0116 (5)	0.0299 (6)	0.0094 (5)	0.0081 (4)	0.0035 (4)	−0.0047 (4)

Geometric parameters (Å, º)

Ag1—O1[i]	2.3600 (12)	Ag2—O2	3.185 (17)
Ag1—O1	2.3600 (12)	Ga—O1	1.9591 (11)
Ag1—O3[ii]	2.5864 (12)	Ga—O1[vi]	1.9591 (11)
Ag1—O3[iii]	2.5864 (12)	Ga—O2[vii]	1.9641 (11)
Ag1—O4[iv]	3.0574 (15)	Ga—O2[v]	1.9641 (11)
Ag1—O4[v]	3.0574 (15)	Ga—O3[viii]	1.9799 (12)
Ag1—O2	3.1352 (12)	Ga—O3[iii]	1.9799 (12)
Ag2—O1	2.357 (17)	As—O2	1.6719 (11)
Ag2—O1[i]	2.402 (18)	As—O3	1.6753 (12)
Ag2—O3[ii]	2.48 (2)	As—O1	1.6773 (11)
Ag2—O3[iii]	2.73 (3)	As—O4	1.7096 (12)

Ag2—O4^{v}	2.83 (2)	O4—H	0.80 (3)
Ag2—O2^{i}	3.116 (18)		
O1—Ga—O1^{vi}	98.48 (7)	O3^{viii}—Ga—O3^{iii}	175.86 (6)
O1—Ga—O2^{vii}	171.19 (5)	O2—As—O3	114.16 (6)
O1^{vi}—Ga—O2^{vii}	87.59 (5)	O2—As—O1	104.63 (6)
O1—Ga—O2^{v}	87.59 (5)	O3—As—O1	110.64 (6)
O1^{vi}—Ga—O2^{v}	171.19 (5)	O2—As—O4	107.25 (7)
O2^{vii}—Ga—O2^{v}	87.12 (7)	O3—As—O4	110.16 (6)
O1—Ga—O3^{viii}	94.82 (5)	O1—As—O4	109.80 (6)
O1^{vi}—Ga—O3^{viii}	82.46 (5)	O2—As—O3	114.16 (6)
O2^{vii}—Ga—O3^{viii}	92.29 (5)	O2—As—O1	104.63 (6)
O2^{v}—Ga—O3^{viii}	90.71 (5)	O3—As—O1	110.64 (6)
O1—Ga—O3^{iii}	82.46 (5)	O2—As—O4	107.25 (7)
O1^{vi}—Ga—O3^{iii}	94.82 (5)	O3—As—O4	110.16 (6)
O2^{vii}—Ga—O3^{iii}	90.71 (5)	O1—As—O4	109.80 (6)
O2^{v}—Ga—O3^{iii}	92.29 (5)		

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

Hydrogen-bond geometry (Å, º)

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O4—H$\cdots$O2^{ix}	0.80 (3)	1.89 (3)	2.6240 (19)	153 (3)

Symmetry code: (ix) x, −y+1, z+1/2.

(AgGaAs2O7) Silver(I) gallium diarsenate(V)

Crystal data

AgGa(As$_2$O$_7$)	$F(000) = 800$
$M_r = 439.43$	$D_x = 5.359$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 7.049$ (1) Å	Cell parameters from 2105 reflections
$b = 8.368$ (2) Å	$\theta = 2.0–32.6°$
$c = 9.735$ (2) Å	$\mu = 20.58$ mm^{-1}
$\beta = 108.47$ (3)°	$T = 293$ K
$V = 544.7$ (2) Å^3	Pseudo-orthorhombic platelets, colourless
$Z = 4$	0.10 × 0.08 × 0.02 mm

Data collection

Nonius KappaCCD single-crystal four-circle diffractometer	1982 independent reflections
	1775 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.022$
φ and ω scans	$\theta_{max} = 32.5°$, $\theta_{min} = 3.1°$
Absorption correction: multi-scan	$h = -10\rightarrow10$
(HKL SCALEPACK; Otwinowski *et al.*, 2003)	$k = -12\rightarrow12$
$T_{min} = 0.233$, $T_{max} = 0.684$	$l = -14\rightarrow14$
3849 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.033$	Secondary atom site location: difference Fourier map
$wR(F^2) = 0.089$	
$S = 1.05$	$w = 1/[\sigma^2(F_o^2) + (0.0619P)^2 + 0.6339P]$
1982 reflections	where $P = (F_o^2 + 2F_c^2)/3$
100 parameters	$(\Delta/\sigma)_{max} < 0.001$
0 restraints	$\Delta\rho_{max} = 2.29$ e Å^{-3}
	$\Delta\rho_{min} = -2.38$ e Å^{-3}

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Ag	0.81152 (5)	0.13552 (4)	0.47918 (4)	0.02028 (11)
Ga	0.28442 (6)	0.28038 (5)	0.49133 (4)	0.00810 (11)
As1	0.52020 (5)	0.41622 (4)	0.28916 (4)	0.00783 (10)
As2	0.94310 (5)	0.54112 (4)	0.29927 (4)	0.00759 (10)
O1	0.3828 (4)	0.4083 (3)	0.1148 (3)	0.0108 (5)
O2	0.5231 (4)	0.5961 (3)	0.3601 (3)	0.0118 (5)
O3	0.4848 (4)	0.2646 (3)	0.3914 (3)	0.0107 (5)
O4	0.7697 (4)	0.3871 (3)	0.2876 (3)	0.0118 (5)
O5	0.1621 (4)	0.4491 (3)	0.3489 (3)	0.0117 (5)
O6	0.9278 (4)	0.6807 (3)	0.4187 (3)	0.0126 (5)
O7	0.8992 (4)	0.6146 (3)	0.1321 (3)	0.0113 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag	0.01549 (17)	0.01302 (15)	0.0269 (2)	−0.00362 (10)	−0.00097 (13)	0.00458 (11)
Ga	0.0088 (2)	0.00743 (18)	0.00749 (19)	0.00004 (12)	0.00177 (15)	0.00004 (12)
As1	0.00867 (19)	0.00705 (17)	0.00701 (17)	−0.00073 (11)	0.00144 (13)	−0.00004 (11)
As2	0.00788 (18)	0.00726 (17)	0.00707 (18)	0.00035 (11)	0.00158 (13)	0.00016 (11)
O1	0.0143 (13)	0.0076 (11)	0.0076 (11)	0.0007 (9)	−0.0008 (10)	0.0002 (8)
O2	0.0133 (13)	0.0080 (11)	0.0113 (12)	−0.0024 (9)	0.0002 (10)	−0.0026 (9)
O3	0.0125 (12)	0.0096 (11)	0.0114 (11)	0.0030 (9)	0.0056 (10)	0.0040 (9)
O4	0.0104 (12)	0.0080 (11)	0.0164 (13)	−0.0007 (9)	0.0033 (10)	0.0003 (9)
O5	0.0075 (11)	0.0128 (11)	0.0144 (13)	0.0043 (9)	0.0030 (10)	0.0060 (10)
O6	0.0122 (13)	0.0141 (12)	0.0120 (12)	−0.0042 (10)	0.0043 (10)	−0.0052 (10)
O7	0.0129 (13)	0.0124 (12)	0.0074 (11)	0.0015 (9)	0.0013 (10)	0.0020 (9)

Geometric parameters (Å, º)

Ag—O1^i	2.353 (3)	Ga—O6^v	1.985 (3)
Ag—O6^{ii}	2.361 (3)	Ga—O7^i	2.015 (3)
Ag—O3	2.440 (3)	As1—O2	1.653 (3)
Ag—O7^{iii}	2.529 (3)	As1—O1	1.668 (3)
Ag—O7^{iv}	2.600 (3)	As1—O3	1.679 (3)
Ag—O4	2.766 (3)	As1—O4	1.781 (3)
Ga—O2^v	1.939 (3)	As2—O5^{vi}	1.655 (3)
Ga—O3	1.957 (3)	As2—O7	1.674 (3)
Ga—O1^{iii}	1.973 (3)	As2—O6	1.675 (3)
Ga—O5	1.975 (3)	As2—O4	1.755 (3)
O1^i—Ag—O6^{ii}	165.93 (10)	O3—Ga—O7^i	94.90 (12)
O1^i—Ag—O3	81.53 (10)	O1^{iii}—Ga—O7^i	81.24 (11)
O6^{ii}—Ag—O3	112.42 (10)	O5—Ga—O7^i	91.08 (12)
O1^i—Ag—O7^{iii}	64.14 (9)	O6^v—Ga—O7^i	86.83 (11)
O6^{ii}—Ag—O7^{iii}	106.19 (10)	O2—As1—O1	112.81 (13)
O3—Ag—O7^{iii}	126.87 (9)	O2—As1—O3	115.14 (14)
O2^v—Ga—O3	87.83 (12)	O1—As1—O3	115.19 (13)
O2^v—Ga—O1^{iii}	86.80 (11)	O2—As1—O4	104.27 (13)
O3—Ga—O1^{iii}	94.54 (11)	O1—As1—O4	104.04 (14)
O2^v—Ga—O5	100.89 (12)	O3—As1—O4	103.55 (13)
O3—Ga—O5	85.56 (11)	O5^{vi}—As2—O7	108.84 (14)
O1^{iii}—Ga—O5	172.30 (11)	O5^{vi}—As2—O6	112.38 (15)
O2^v—Ga—O6^v	91.74 (12)	O7—As2—O6	112.71 (14)
O3—Ga—O6^v	173.63 (11)	O5^{vi}—As2—O4	104.10 (13)
O1^{iii}—Ga—O6^v	91.78 (12)	O7—As2—O4	107.22 (13)
O5—Ga—O6^v	88.28 (11)	O6—As2—O4	111.12 (14)
O2^v—Ga—O7^i	167.90 (11)	As2—O4—As1	124.65 (15)

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

(NaGaAs2O7) Sodium gallium diarsenate(V)

Crystal data

NaGa(AsO$_7$)
$M_r = 354.55$
Monoclinic, $P2_1/c$
$a = 6.987$ (1) Å
$b = 8.266$ (2) Å
$c = 9.677$ (2) Å
$β = 107.50$ (3)°
$V = 533.0$ (2) Å^3
$Z = 4$
$F(000) = 656$

$D_x = 4.418$ Mg m^{-3}
Mo $Kα$ radiation, $λ = 0.71073$ Å
Cell parameters from 2065 reflections
$θ = 3–30°$
$μ = 17.55$ mm^{-1}
$T = 293$ K
Small platelets with diamond-shaped outline, colourless
$0.07 × 0.07 × 0.02$ mm

Data collection

Nonius KappaCCD single-crystal four-circle diffractometer	1557 independent reflections
Radiation source: fine-focus sealed tube	1336 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.023$
Absorption correction: multi-scan (HKL SCALEPACK; Otwinowski *et al.*, 2003)	$\theta_{max} = 30.0°$, $\theta_{min} = 3.1°$
	$h = -9 \rightarrow 9$
	$k = -11 \rightarrow 11$
$T_{min} = 0.373$, $T_{max} = 0.720$	$l = -13 \rightarrow 13$
3015 measured reflections	

Refinement

Refinement on F^2	0 restraints
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.060P)^2 + 1.1P]$
$R[F^2 > 2\sigma(F^2)] = 0.033$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.090$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.04$	$\Delta\rho_{max} = 1.54$ e Å^{-3}
1557 reflections	$\Delta\rho_{min} = -1.49$ e Å^{-3}
100 parameters	

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 (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
Na	0.8070 (3)	0.1389 (2)	0.4691 (2)	0.0212 (4)
Ga	0.28177 (7)	0.27455 (6)	0.49377 (5)	0.00989 (14)
As1	0.51759 (7)	0.41477 (5)	0.29095 (5)	0.00962 (13)
As2	0.94401 (7)	0.53707 (5)	0.29682 (5)	0.00931 (13)
O1	0.3798 (5)	0.4129 (4)	0.1176 (3)	0.0128 (6)
O2	0.5235 (5)	0.5967 (4)	0.3635 (4)	0.0132 (6)
O3	0.4850 (5)	0.2586 (4)	0.3921 (4)	0.0126 (6)
O4	0.7683 (5)	0.3827 (4)	0.2891 (4)	0.0137 (6)
O5	0.1637 (5)	0.4423 (4)	0.3491 (3)	0.0134 (6)
O6	0.9305 (5)	0.6834 (4)	0.4127 (4)	0.0140 (6)
O7	0.9038 (5)	0.6014 (4)	0.1260 (3)	0.0117 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Na	0.0190 (10)	0.0122 (9)	0.0286 (10)	−0.0006 (8)	0.0013 (8)	0.0005 (8)
Ga	0.0130 (3)	0.0074 (2)	0.0096 (2)	0.00006 (16)	0.00367 (19)	0.00009 (16)
As1	0.0125 (2)	0.0070 (2)	0.0090 (2)	−0.00080 (15)	0.00274 (17)	0.00001 (15)
As2	0.0120 (2)	0.0073 (2)	0.0086 (2)	0.00045 (15)	0.00308 (17)	0.00032 (15)
O1	0.0175 (17)	0.0087 (14)	0.0102 (14)	−0.0003 (12)	0.0011 (12)	0.0012 (12)
O2	0.0167 (16)	0.0064 (14)	0.0164 (15)	−0.0015 (11)	0.0049 (13)	−0.0028 (12)
O3	0.0166 (16)	0.0087 (14)	0.0146 (15)	0.0022 (12)	0.0080 (13)	0.0035 (12)
O4	0.0098 (15)	0.0121 (14)	0.0198 (16)	0.0002 (12)	0.0055 (12)	0.0001 (12)

O5	0.0130 (16)	0.0124 (14)	0.0142 (15)	0.0039 (12)	0.0029 (13)	0.0043 (12)
O6	0.0188 (17)	0.0120 (15)	0.0139 (14)	−0.0035 (12)	0.0088 (13)	−0.0043 (12)
O7	0.0157 (16)	0.0116 (14)	0.0082 (13)	0.0022 (12)	0.0042 (12)	0.0033 (12)

Geometric parameters (Å, °)

Na—O1[i]	2.289 (4)	Ga—O6[v]	1.987 (3)
Na—O6[ii]	2.360 (4)	Ga—O7[i]	2.042 (3)
Na—O3	2.364 (4)	As1—O2	1.655 (3)
Na—O7[iii]	2.468 (4)	As1—O1	1.664 (3)
Na—O7[iv]	2.479 (4)	As1—O3	1.676 (3)
Na—O4	2.624 (4)	As1—O4	1.777 (3)
Ga—O2[v]	1.940 (3)	As2—O5[vi]	1.661 (3)
Ga—O1[iii]	1.952 (3)	As2—O6	1.671 (3)
Ga—O3	1.960 (3)	As2—O7	1.678 (3)
Ga—O5	1.967 (3)	As2—O4	1.756 (3)
O1[i]—Na—O6[ii]	163.76 (16)	O1[iii]—Ga—O6[v]	91.80 (14)
O1[i]—Na—O3	80.93 (13)	O3—Ga—O6[v]	173.31 (13)
O6[ii]—Na—O3	114.82 (15)	O5—Ga—O6[v]	89.46 (14)
O1[i]—Na—O7[iii]	65.73 (13)	O2[v]—Ga—O7[i]	168.13 (13)
O6[ii]—Na—O7[iii]	99.98 (14)	O1[iii]—Ga—O7[i]	80.66 (14)
O3—Na—O7[iii]	126.13 (14)	O3—Ga—O7[i]	95.74 (13)
O1[i]—Na—O7[iv]	101.56 (14)	O5—Ga—O7[i]	91.80 (13)
O6[ii]—Na—O7[iv]	69.89 (12)	O6[v]—Ga—O7[i]	87.01 (13)
O3—Na—O7[iv]	137.74 (14)	O2—As1—O1	111.67 (16)
O7[iii]—Na—O7[iv]	91.40 (12)	O2—As1—O3	116.27 (16)
O1[i]—Na—O4	116.75 (14)	O1—As1—O3	116.25 (16)
O6[ii]—Na—O4	75.75 (13)	O2—As1—O4	103.93 (16)
O3—Na—O4	64.59 (11)	O1—As1—O4	105.16 (17)
O7[iii]—Na—O4	168.81 (15)	O3—As1—O4	101.46 (16)
O7[iv]—Na—O4	77.43 (12)	O5[vi]—As2—O6	111.76 (17)
O2[v]—Ga—O1[iii]	87.52 (13)	O5[vi]—As2—O7	108.37 (16)
O2[v]—Ga—O3	86.31 (14)	O6—As2—O7	113.90 (16)
O1[iii]—Ga—O3	94.67 (14)	O5[vi]—As2—O4	103.91 (16)
O2[v]—Ga—O5	100.04 (14)	O6—As2—O4	112.00 (16)
O1[iii]—Ga—O5	172.28 (14)	O7—As2—O4	106.27 (16)
O3—Ga—O5	84.37 (14)	As2—O4—As1	124.73 (19)
O2[v]—Ga—O6[v]	92.25 (14)		

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