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Tricaesium tetraselenidoarsenate(V) monohydrate

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Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(Se–As) = 0.004 Å
H-atom completeness 0%
R factor = 0.068
wR factor = 0.201
Data-to-parameter ratio = 27.7

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

The title compound, $Cs_3AsSe_4\cdot H_2O$, contains discrete tetrahedral tetraselenidoarsenate(V) anions. The solvent water O atom participates in the coordination spheres of all three independent Cs cations, as do, in each case, seven Se atoms.

Comment

The discrete tetrahedral $[AsSe_4]^{3-}$ anion has been characterized by X-ray structural analysis in $[Li(NH_3)_4]_3AsSe_4$ (Korber & Grothe, 2001), $Na_3AsSe_4\cdot 9H_2O$ (Krebs *et al.*, 1990), Rb_3AsSe_4 and Cs_3AsSe_4 (Wachhold & Sheldrick, 1996), $Rb_3AsSe_4\cdot 2Se_6$ and $Cs_3AsSe_4.2\,C\,s_2As_2Se_4\cdot 6Te_4Se_2$ (Wachhold & Sheldrick, 1997), and $Ba_2AsSe_4(OH)\cdot 2H_2O$ (Kaub, 1986). For instance, red crystals of M_3AsSe_4 (M = Rb or Cs) can be obtained by methanolothermal reaction of M_2CO_3 with As_2Se_3 and Se in appropriate molar ratio at 453 K.

We have now discovered that, on changing the solvent from methanol to an equimolar H_2O/CH_3OH mixture for M = Cs, the title monohydrate, $Cs_3AsSe_4\cdot H_2O$, (I), is formed, rather than Cs_3AsSe_4.

The discrete tetraselenidoarsenate(V) anions of (I) exhibit As—Se distances between 2.308 (4) and 2.316 (4) Å (Fig. 1 and Table 1), which are similar to those in the range 2.306–2.336 Å found in Rb_3AsSe_4 and Cs_3AsSe_4. As in these selenidoarsenates(V), seven Se atoms participate in the coordination spheres of each of the alkali metal cations, which are sited between the tetrahedral $[AsSe_4]^{3-}$ anions (Fig. 2). Additional Cs···O contacts of 3.09 (2) (Cs1), 3.54 (2) (Cs2) and 3.34 (2)/3.39 (2) Å (Cs3) lead to total coordination numbers of, respectively, 8, 8 and 9 for the Cs^+ cations of (I).

Experimental

As_2Se_3 (193.4 mg, 0.5 mmol), Se (79.0 mg, 1.0 mmol) and Cs_2CO_3 (977.5 mg, 3.0 mmol) were heated to 393 K in an H_2O–CH_3OH

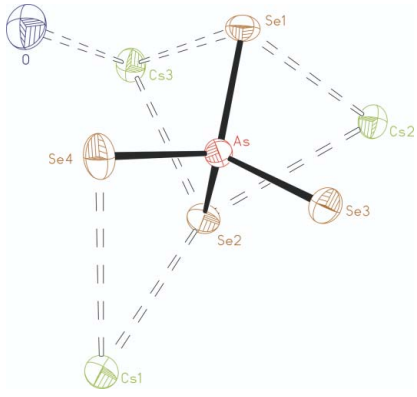

Figure 1
The asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level.

mixture (1:1 *v*/*v*, 0.8 ml) in a sealed glass tube. After 2 d, the contents were cooled to room temperature to afford red crystals of $Cs_3AsSe_4{\cdot}H_2O$ in 32% yield.

Crystal data

$Cs_3AsSe_4{\cdot}H_2O$
M_r = 807.51
Monoclinic, $P2_1/n$
a = 9.994 (3) Å
b = 10.541 (4) Å
c = 12.372 (6) Å
β = 91.73 (2)°
V = 1302.8 (9) Å^3
Z = 4
D_x = 4.117 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 18 reflections
θ = 5.5–12.7°
μ = 22.00 mm^{-1}
T = 292 (2) K
Block, red
0.15 × 0.13 × 0.11 mm

Data collection

Siemens P4 four-circle diffractometer
ω scans
Absorption correction: ψ-scan (*XPREP* in *SHELXTL*; Sheldrick, 1995)
T_{min} = 0.054, T_{max} = 0.092
2449 measured reflections
2295 independent reflections
1399 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 25.0°
$h = 0 \rightarrow 11$
$k = -12 \rightarrow 0$
$l = -14 \rightarrow 14$
3 standard reflections every 97 reflections
intensity decay: 0.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.201
S = 1.15
2295 reflections
83 parameters
H atoms not located
$w = 1/[\sigma^2(F_o^2) + (0.0949P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.95 e Å^{-3}
$\Delta\rho_{min}$ = −2.41 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cs1—O^{i}	3.09 (2)	Cs2—Se4^{vi}	4.002 (4)
Cs1—Se1^{ii}	3.533 (3)	Cs3—O	3.34 (2)
Cs1—Se2	3.620 (3)	Cs3—O^{ix}	3.39 (2)
Cs1—Se3^{iii}	3.743 (3)	Cs3—Se1^{vi}	3.635 (3)
Cs1—Se4^{iv}	3.756 (4)	Cs3—Se2	3.717 (3)
Cs1—Se2^{v}	3.928 (4)	Cs3—Se3^{vi}	3.718 (3)
Cs1—Se3^{iv}	4.024 (4)	Cs3—Se1	3.737 (3)
Cs1—Se4	4.024 (4)	Cs3—Se4^{ix}	3.933 (4)
Cs2—O^{vi}	3.54 (2)	Cs3—Se2^{ix}	4.079 (3)
Cs2—Se4^{vii}	3.588 (3)	Cs3—Se3^{x}	4.130 (4)
Cs2—Se1^{vi}	3.650 (3)	As—Se4	2.308 (4)
Cs2—Se1	3.668 (3)	As—Se2	2.313 (4)
Cs2—Se2^{viii}	3.729 (3)	As—Se3	2.316 (3)
Cs2—Se2	3.822 (3)	As—Se1	2.316 (4)
Cs2—Se3^{viii}	3.929 (4)		
Se4—As—Se2	109.08 (14)	Se4—As—Se1	108.04 (14)
Se4—As—Se3	110.24 (15)	Se2—As—Se1	109.26 (14)
Se2—As—Se3	110.12 (14)	Se3—As—Se1	110.07 (13)

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$; (ii) $x+1, y, z$; (iii) $x+\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$; (iv) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (v) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (vi) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (vii) $x-\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$; (viii) $-x+1, -y+2, -z+1$; (ix) $-x+1, -y+2, -z$; (x) $x-\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$.

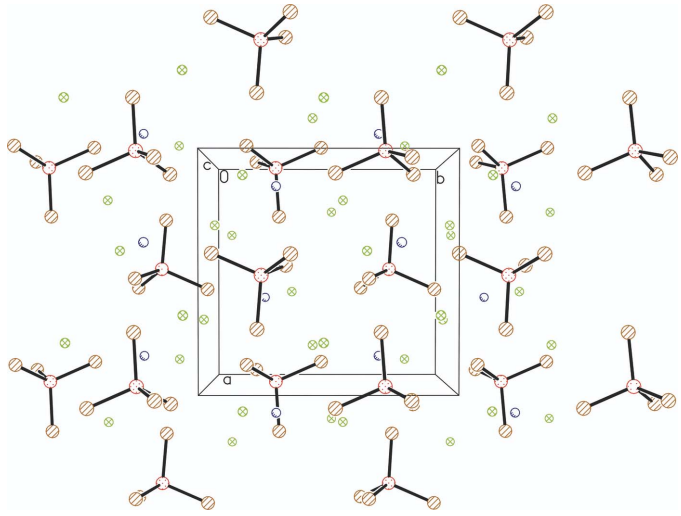

Figure 2
A projection of the structure of (I), perpendicular to the *ab* plane. Atom colour codes: Cs green cross-hatched circles, Se orange hatched circles, As red dotted circles, O blue semi-hatched circles.

The water H atoms could not be located in a final difference synthesis and were not, therefore, included in the refinement. The highest peak in the final difference Fourier synthesis is 2.30 Å from Cs3 and the deepest hole is 0.96 Å from the same atom.

Data collection: *R3m*/*V* (Siemens, 1989); cell refinement: *R3m*/*V*; data reduction: *XDISK* (Siemens, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1995); software used to prepare material for publication: *SHELXL97*.

References

Kaub, J. (1986). *Z. Naturforsch. Teil B*, **41**, 436–438.
Korber, N. & Grothe, M. (2001). *Z. Kristallogr. New Cryst. Struct.* **216**, 177–178.
Krebs, B., Hürter, H.-U., Enax, J. & Frölich, R. (1990). *Z. Anorg. Allg. Chem.* **581**, 141–152.
Sheldrick, G. M. (1995). *SHELXTL*. Release 5.03. Siemens Analytical X-ray Instruments Inc., Madison, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Siemens (1989). *R3m*/*V* and *XDISK*. Versions 3.2. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Wachhold, M. & Sheldrick, W. S. (1996). *Z. Naturforsch. Teil B*, **51**, 32–36.
Wachhold, M. & Sheldrick, W. S. (1997). *Z. Naturforsch. Teil B*, **52**, 169–175.