Gougeon, P., Gall, P. & Sergent, M. (1991). *Acta Cryst.* C**47**, 421–423.
Gougeon, P. & McCarley, R. E. (1991). *Acta Cryst.* C**47**, 241–244.
Hibble, S. J., Cheetham, A. K., Bogle, A. R. L., Wakerley, H. R. & Cox, D. E. (1988). *J. Am. Chem. Soc.* **110**, 3295–3296.
Leligny, H., Ledesert, M., Labbe, Ph., Raveau, B. & McCarroll, W. H. (1990). *J. Solid State Chem.* **87**, 35–43.
Lii, K. H., Wang, C. C. & Wang, S. L. (1988). *J. Solid State Chem.* **77**, 407–411.
Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
Moini, A., Subramanian, M. A., Clearfield, A., Di Salvo, F. J. & McCarroll, W. H. (1987). *J. Solid State Chem.* **66**, 136–143.
Stout, G. & Jensen, L. H. (1968). *X-ray Structure Determination.* London: MacMillan.
Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.

Acta Cryst. (1993). C**49**, 1584–1585

Structure of UOSe

Michael F. Mansuetto, Stéphane Jobic, Huey P. Ng and James A. Ibers*

Department of Chemistry, Northwestern University, Evanston, IL 60208-3113, USA

(*Received* 12 *January* 1993; *accepted* 5 *March* 1993)

Abstract

The structure of UOSe, uranium oxyselenide, has been determined from a single crystal at 113 K. UOSe is isostructural with UOS and is of the PbFCl structure type. The structure contains a nine-coordinate U atom bonded to five Se atoms and four O atoms in a distorted tricapped trigonal prismatic arrangement. The U—Se distances are 3.012 (5) and 3.080 (5) Å; the U—O distance is 2.346 (4) Å.

Comment

A single crystal of UOSe was discovered in the reaction products of a combination of K_2Se, Cu, U and Se. In an attempt to explore further the synthesis of quaternary chalcogenides that are related to the recently reported series of compounds $KCuZrQ_3$ (Mansuetto, Keane & Ibers, 1992) and $NaCuMQ_3$ (Mansuetto, Keane & Ibers, 1993) K_2Se, Cu, U, and Se were combined in a molar ratio of 1:1:1:3. The mixture was loaded into a quartz tube under an argon atmosphere and evacuated to approximately 10^{-4} Torr (1 Torr = 133.322 Pa) and then sealed. The tube was heated in a furnace from room temperature to 1223 K over 7 h and held there for 1 week before being cooled back to room temperature at 4 K h^{-1}. A flat needle of UOSe was extracted from the melt by washing away the excess flux with water. The oxygen is presumed to have come from the quartz.

The structure of UOSe was first determined from powder data (Ferro, 1954) and found to be isostructural with UOS (Zachariasen, 1949). In the present work, this structure of UOSe has been confirmed and refined from data collected from a single crystal at 113 K. The structure is of the PbFCl type (Nieuwenkamp & Bijvoet, 1932) and comprises a nine-coordinate U atom in a distorted tricapped trigonal prism of five Se atoms and four O atoms. A view down the *b* axis is given in Fig. 1. The U—Se distances are comparable to those found in α-USe_2 [2.880–3.133 Å (Beck & Dausch, 1989)] and U_3Se_5 [2.80–3.18 Å (Moseley, Brown & Whittaker, 1972)], while the U—O distance agrees with that of 2.363 Å in UO_2 (Rundle, Baenziger, Wilson & McDonald, 1948).

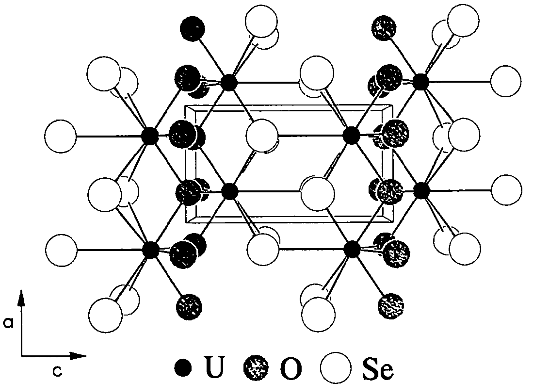

Fig. 1. View of UOSe down [010] with atoms labeled.

Experimental

Crystal data

UOSe
$M_r = 332.99$
Tetragonal
$P4/nmm$
$a = 3.885$ (4) Å
$c = 6.947$ (4) Å
$V = 104.9$ (2) Å^3
$Z = 2$
$D_x = 10.547$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7093$ Å
Cell parameters from 21 reflections
$\theta = 7.43$-$17.80°$
$\mu = 94.31$ mm^{-1}
$T = 113$ K
Flat needle
0.372 × 0.048 × 0.002 mm
Black

Data collection

Picker diffractometer
θ-2θ scans

$R_{\rm int} = 0.0644$
$\theta_{\rm max} = 30.07°$

Absorption correction: analytical
$T_{min} = 0.061$, $T_{max} = 0.827$
1169 measured reflections
116 independent reflections
109 observed reflections $[I > 2\sigma(I)]$

$h = -5 \rightarrow 5$
$k = -5 \rightarrow 5$
$l = -9 \rightarrow 9$
6 standard reflections monitored every 100 reflections
intensity variation: none

Refinement

Refinement on F^2
Final $R(F) = 0.0215$
$wR(F^2) = 0.0481$
$S = 1.094$
116 reflections
10 parameters
Calculated weights, $w = 1/[\sigma^2(F_o^2)+0.0016F^2]$
$(\Delta/\sigma)_{max} = 0.113$
$\Delta\rho_{max} = 4.59$ e Å^{-3}
$\Delta\rho_{min} = -1.53$ e Å^{-3}

Extinction correction: *SHELXL92* (Sheldrick, 1992)
Extinction coefficient: 0.0341 (43)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Refinement on F^2 for all reflections; the observed threshold $I > 2\sigma(I)$ is used only for calculating R_{obs} *etc.* given here for comparison with refinements on F. Cell refinement: *CELREF*, local program. Data reduction: *NUPICK*, local program. Program used to solve structure: *XS* subroutine of *SHELXTL/PC* (Sheldrick, 1990). Program used to refine structure: *SHELXL92* (Sheldrick, 1992). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990). Software used to prepare material for publication: *SHELXL92*; *DATABL*, local program.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i\cdot\mathbf{a}_j.$$

	x	y	z	U_{eq}
U	$\frac{1}{4}$	$\frac{1}{4}$	0.18946 (7)	0.0034 (3)
Se	$\frac{1}{4}$	$\frac{1}{4}$	0.6328 (2)	0.0043 (4)
O	$\frac{3}{4}$	$\frac{1}{4}$	0	0.007 (2)

Table 2. *Geometric parameters* (Å, °)

U—O^{i}	2.346 (4)	U—Se	3.080 (5)
U—Se^{ii}	3.012 (5)		
O^{i}—U—O^{iii}	71.66 (9)	Se^{ii}—U—Se^{v}	131.60 (9)
O^{i}—U—O^{iv}	111.76 (9)	Se^{ii}—U—Se^{vi}	80.33 (9)
O^{i}—U—Se^{ii}	139.81 (6)	O^{i}—U—Se	124.12 (8)
O^{iv}—U—Se^{ii}	72.30 (9)	Se^{ii}—U—Se	65.80 (9)

Symmetry codes: (i) $1-x, -y, -z$; (ii) $1-x, 1-y, 1-z$; (iii) $x-1, y, z$; (iv) $1-x, 1-y, -z$; (v) $-x, -y, 1-z$; (vi) $-x, 1-y, 1-z$.

This work was supported by the US National Science Foundation through grant DMR91-14934.

Lists of structure factors, anisotropic thermal parameters and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71186 (5 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST1058]

References

Beck, H. P. & Dausch, W. (1989). *J. Solid State Chem.* **80**, 32-39.
Ferro, R. (1954). *Z. Anorg. Allg. Chem.* **275**, 320-326.
Mansuetto, M. F., Keane, P. M. & Ibers, J. A. (1992). *J. Solid State Chem.* **101**, 257-264.
Mansuetto, M. F., Keane, P. M. & Ibers, J. A. (1993). *J. Solid State Chem.* In the press.
Moseley, P. T., Brown, D. & Whittaker, B. (1972). *Acta Cryst.* B**28**, 1816-1821.
Nieuwenkamp, W. & Bijvoet, J. M. (1932). *Z. Kristallogr. Kristallgeom. Kristallphys. Kristallchem.* **81**, 469-474.
Rundle, R. E., Baenziger, N. C., Wilson, A. S. & McDonald, R. A. (1948). *J. Am. Chem. Soc.* **70**, 99-105.
Sheldrick, G. M. (1990). *SHELXTL/PC. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1992). *SHELXL92*. Beta Test Version. Univ. of Göttingen, Germany.
Zachariasen, W. H. (1949). *Acta Cryst.* **2**, 291-296.

Acta Cryst. (1993). C**49**, 1585–1588

Structure of $Ba_4NaSb_3O_{12}$ by Neutron Powder Diffraction

Kenneth P. Reis and Allan J. Jacobson*

Department of Chemistry, University of Houston, Houston, TX 77204, USA

(*Received* 3 *November* 1992; *accepted* 5 *March* 1993)

Abstract

The structure of the ordered perovskite triantimony tetrabarium sodium dodecaoxide, $Ba_4NaSb_3O_{12}$, was determined by Rietveld analysis of powder time-of-flight neutron diffraction data. The Na and Sb atoms form a 1:3 ordered arrangement on the *B* sites similar to that observed for $Ba_4LiSb_3O_{12}$.

Comment

The ABO_3 perovskite structure can accommodate more than one cation on both the *A* and *B* sites in ordered or disordered arrangements depending on stoichiometry and on the specific *A* and *B* cations present (Galasso, 1969). Ordering of the *A* cations is quite rare for perovskite compounds though some important cases, for example $YBa_2Cu_3O_7$, are well known. Many more examples of compounds exist where two different *B* cations form an ordered structure, usually when the metal cations differ significantly in either size or charge. The most common ordered arrangements correspond to *B*/*B*′ ratios