

The β -polymorph of uranium phosphide selenide

George N. Oh and James A. Ibers*

Department of Chemistry, Northwestern University, 2145 Sheridan Rd., Evanston, IL 60208-3113, USA

Correspondence e-mail: ibers@chem.northwestern.edu

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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{U-Se}) = 0.001$ Å; disorder in main residue; R factor = 0.024; wR factor = 0.052; data-to-parameter ratio = 10.7.

β -UPSe was synthesized from the reaction of U_2Se_3 , P and Se in a CsCl flux in a fused-silica tube. It crystallizes with four formula units in the tetragonal space group $I4/mmm$ in the UGeTe structure type. The asymmetric unit comprises one U (site symmetry $4mm$), one Se ($4mm$), and one P (mmm) atom. The U atom is coordinated in a monocapped square-antiprismatic arrangement, where the square face is formed by P atoms and the other five vertices are Se atoms. The P site is disordered about a mirror plane, showing half-occupancy for each of the two resulting P atoms. The title structure is related to that of α -UPSe, which crystallizes with two formula units in the tetragonal space group $P4/nmm$ in the PbFCl structure type.

Related literature

Whereas β -UPSe crystallizes in the UGeTe structure type (Haneveld & Jellinek, 1969), α -UPSe (Hulliger, 1968; Zymunt *et al.*, 1974a) crystallizes in the PbFCl structure type (Nieuwenkamp & Bijvoet, 1932). Isostructural compounds UTQ have been synthesized ($T = \text{P-Bi}$, $Q = \text{Se-Te}$) (Hulliger, 1968; Leciejewicz & Zymunt, 1972; Haneveld & Jellinek, 1969; Zymunt *et al.*, 1974a,b; Pietraszko & Lukaszewicz, 1975; Pearson, 1985). Magnetic studies have been performed on single crystals of β -UPSe synthesized by the vapor-transport method (Kaczorowski *et al.*, 1995), and other compounds with formula UTQ (Troc, 1987). For synthetic details, see: Bugaris & Ibers (2008) and Haneveld & Jellinek (1969). For standardization of structural data, see: Gelato & Parthé (1987).

Experimental

Crystal data

UPSe	$c = 16.2836$ (17) Å
$M_r = 347.96$	$V = 253.33$ (4) Å ³
Tetragonal, $I4/mmm$	$Z = 4$
$a = 3.9443$ (4) Å	Mo $K\alpha$ radiation

$$\mu = 78.66 \text{ mm}^{-1}$$

$$T = 298 \text{ K}$$

$$0.41 \times 0.39 \times 0.03 \text{ mm}$$

Data collection

Bruker APEXII CCD diffractometer	1506 measured reflections
Absorption correction: numerical face-indexed (SADABS; Shel-drick, 2008a)	128 independent reflections
$T_{\min} = 0.013$, $T_{\max} = 0.175$	128 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.034$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.024$	12 parameters
$wR(F^2) = 0.052$	$\Delta\rho_{\text{max}} = 1.89 \text{ e } \text{Å}^{-3}$
$S = 1.31$	$\Delta\rho_{\text{min}} = -4.77 \text{ e } \text{Å}^{-3}$
128 reflections	

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008b); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008b); molecular graphics: CrystalMaker (Palmer, 2009); software used to prepare material for publication: SHELXL97.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2560).

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supporting information

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S1. Comment

The compounds UTQ ($T = \text{P, As, Bi}; Q = \text{S, Se, Te}$) crystallize in one of two structure types. UBiTe , UTQ ($T = \text{As, Sb}; Q = \text{S, Se, Te}$), and UPQ ($Q = \text{S, Se}$) (Hulliger, 1968; Leciejewicz & Zygmunt, 1972; Haneveld & Jellinek, 1969; Zygmunt *et al.*, 1974a; Pietraszko & Lukaszewicz, 1975; Pearson, 1985) crystallize in the tetragonal space group $P4/nmm$ in the PbFCl (Nieuwenkamp & Bijvoet, 1932) structure type. UPTe and UAsTe (Pietraszko & Lukaszewicz, 1975; Pearson, 1985; Zygmunt *et al.*, 1974b) crystallize in the tetragonal space group $I4/mmm$ in the UGeTe (Haneveld & Jellinek, 1969) structure type. Magnetic studies have been performed on single crystals of β -UPSe crystallizing in the UGeTe structure type, but no structural data were reported. β -UPSe orders antiferromagnetically at low temperature (Troc, 1987; Kaczorowski *et al.*, 1995).

We have synthesized β -UPSe from a CsCl flux, in contrast to previous reported synthetic methods. This compound crystallizes with four formula units in the tetragonal space group $I4/mmm$ (Figure 1) in the UGeTe structure type. The asymmetric unit comprises atoms U1 (site symmetry $4mm$), Se1 ($4mm$), and P1 (mmm) (Figure 2). U is coordinated in a monocapped square-antiprismatic arrangement, where the square face is formed by P atoms and the other five vertices are Se atoms. These moieties face-share along the four triangular faces formed by two Se atoms and one P atom with adjacent moieties. They also face-share along the square faces formed by P atoms. Finally, each of the four edges on the cap are shared with another moiety so that the U atoms are staggered when viewed down $[001]$. Viewed on the side of the basal plane, each atom type lies on its own plane.

The structure is related to that of α -UPSe (Zygmunt *et al.*, 1974a; Hulliger, 1968). The only difference is that instead of face-sharing along the square faces formed by the P atoms, the moieties edge-share with four other moieties, such that the U atoms are staggered in a checkerboard pattern when viewed down $[001]$.

Interatomic distances are typical. The U-P distance is 2.8514 (7) Å, comparable to 2.91 Å in α -UPSe (Zygmunt *et al.*, 1974a). The U-Se distances are 2.9605 (5) Å and 3.0633 (11) Å, compared to 2.901 Å and 3.131 Å in α -UPSe.

S2. Experimental

U filings (Oak Ridge National Laboratory) were powdered as previously described (Bugaris & Ibers, 2008; Haneveld & Jellinek, 1969), and U_2Se_3 was synthesized by the stoichiometric reaction of U and Se (Cerac 99.999%) in a fused-silica tube at 1273 K. Black square plates of β -UPSe were synthesized in the reaction of U_2Se_3 (0.215 mmol), red P (Aldrich 99%, 0.064 mmol), Se (0.215 mmol), and CsCl (MP Biomedical 99.9%, 1.21 mmol) in a carbon-coated fused-silica tube. Reagents were loaded in an argon-filled glove box, and the tube was flame-sealed under 10^{-4} Torr vacuum. The tube was placed in a computer-controlled furnace and heated to 1273 K in 96 h, held there for 4 h, cooled to 1223 K in 12 h, held there for 96 h, and then cooled to 298 K in 350 h. The solidified flux was washed off with water. Qualitative EDS analysis using a Hitachi S-3400 SEM showed the presence of U , P , and Se , with no detectable Cs or Cl content in the crystals.

S3. Refinement

The structure was standardized with the use of *STRUCTURE TIDY* (Gelato & Parthé, 1987). The highest peak in the difference electron density map is $1.9(6) \text{ e}/\text{\AA}^3$, 0.03 \AA from atom U1, and the deepest hole is $-4.8(6) \text{ e}/\text{\AA}^3$, 0.91 \AA from atom U1.

The refinement results in the placement of a half-occupied P1 atom in the $8j$ position. The resultant disorder at $+x, 1/2, 0$ (P1a) and $-x, 1/2, 0$ (P1b) leads to impossible P1a–P1b distances of 0.52 \AA and 2.42 \AA . The P1 atoms are arranged in a square net 2.813 \AA on a side. The distribution of P1a squares and P1b squares within a structure is random, as no supercell reflections were observed.

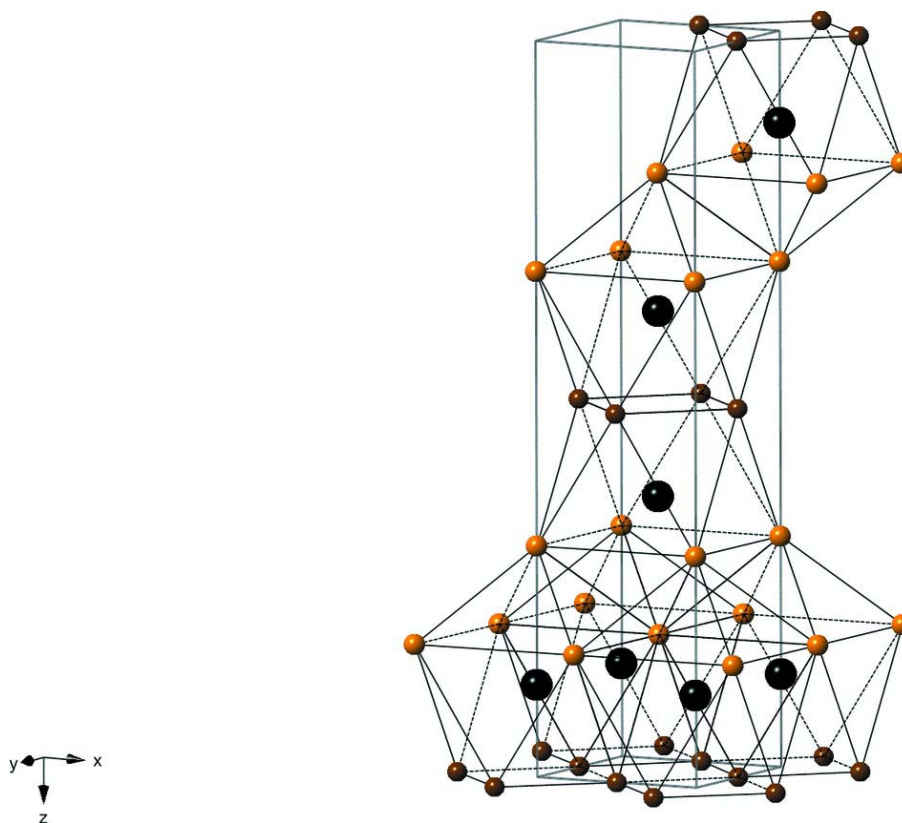


Figure 1

Structure of β -UPSe. U atoms are black, P atoms are brown, and Se atoms are orange.

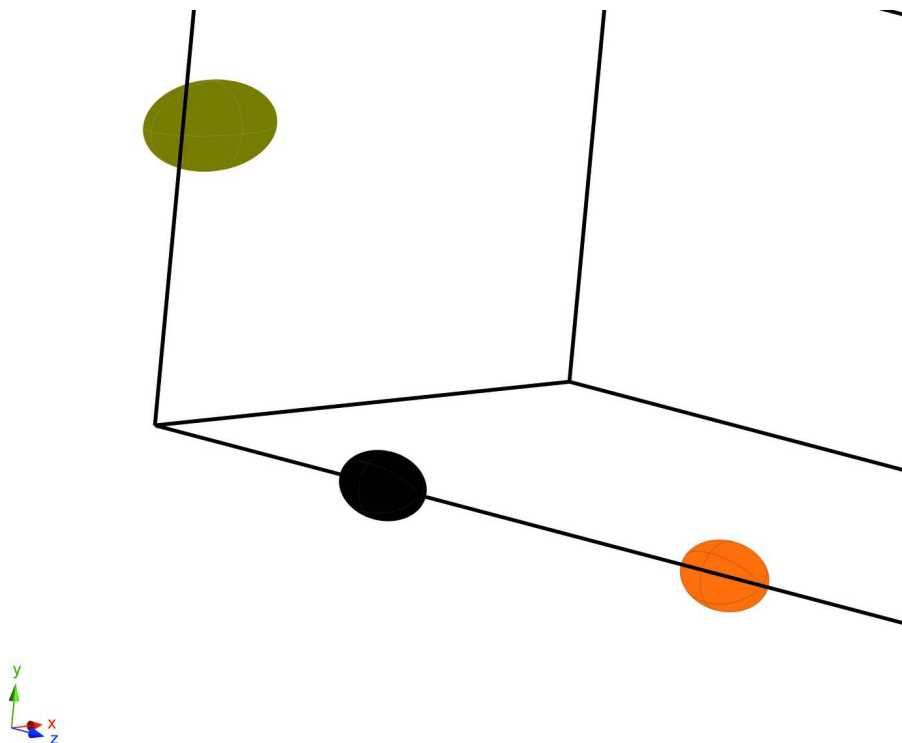


Figure 2

Asymmetric unit of β -UPSe. Displacement ellipsoids are drawn at the 95% probability level. Color code as in Fig. 1.

uranium phosphide selenide

Crystal data

UPSe

$M_r = 347.96$

Tetragonal, $I4/mmm$

Hall symbol: $-I\ 4\ 2$

$a = 3.9443\ (4)\ \text{\AA}$

$c = 16.2836\ (17)\ \text{\AA}$

$V = 253.33\ (4)\ \text{\AA}^3$

$Z = 4$

$F(000) = 564$

$D_x = 9.123\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1519 reflections

$\theta = 5.0\text{--}28.4^\circ$

$\mu = 78.66\ \text{mm}^{-1}$

$T = 298\ \text{K}$

Square plate, black

$0.41 \times 0.39 \times 0.03\ \text{mm}$

Data collection

Bruker APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: numerical

face-indexed (*SADABS*; Sheldrick, 2008b)

$T_{\min} = 0.013$, $T_{\max} = 0.175$

1506 measured reflections

128 independent reflections

128 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.034$

$\theta_{\max} = 28.4^\circ$, $\theta_{\min} = 2.5^\circ$

$h = -5 \rightarrow 5$

$k = -5 \rightarrow 5$

$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.024$
 $wR(F^2) = 0.052$
 $S = 1.31$
 128 reflections
 12 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 $[1.00000 + 0.00000\exp(0.00(\sin\theta/\lambda)^2)] / [\sigma^2(F_o^2)$
 $+ 0.0000 + 0.0000*P + (0.0377P)^2 +$
 $0.0000\sin\theta/\lambda]$
 where $P = 1.00000F_o^2 + 0.00000F_c^2$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 1.89 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -4.77 \text{ e } \text{\AA}^{-3}$
 Extinction correction: *SHELXL97* (Sheldrick,
 2008a), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0139 (12)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
U1	0.0000	0.0000	0.125455 (18)	0.0090 (3)	
Se1	0.0000	0.0000	0.31358 (6)	0.0093 (4)	
P1	0.0660 (18)	0.5000	0.0000	0.021 (2)	0.50

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
U1	0.0066 (3)	0.0066 (3)	0.0138 (4)	0.000	0.000	0.000
Se1	0.0068 (4)	0.0068 (4)	0.0142 (6)	0.000	0.000	0.000
P1	0.040 (7)	0.0114 (18)	0.0107 (13)	0.000	0.000	0.000

Geometric parameters (\AA , $^\circ$)

U1—P1 ⁱ	2.8514 (7)	U1—U1 ^{iv}	3.9443 (4)
U1—P1 ⁱⁱ	2.8514 (7)	U1—U1 ⁱⁱ	4.0858 (7)
U1—P1 ⁱⁱⁱ	2.8514 (7)	P1—P1 ^{vii}	0.520 (14)
U1—P1 ^{iv}	2.8514 (7)	P1—P1 ^{xv}	2.421 (10)
U1—P1 ^v	2.8514 (7)	P1—P1 ^{vi}	2.421 (10)
U1—P1 ^{vi}	2.8514 (7)	P1—P1 ^{xvi}	2.8132 (13)
U1—P1 ^{vii}	2.8514 (7)	P1—P1 ⁱⁱⁱ	2.8132 (13)
U1—P1	2.8514 (7)	P1—P1 ^{xvii}	2.8132 (13)
U1—Se1 ^{viii}	2.9605 (5)	P1—P1 ^v	2.8132 (13)
U1—Se1 ^{ix}	2.9605 (5)	P1—U1 ⁱⁱ	2.8514 (7)
U1—Se1 ^x	2.9605 (5)	P1—U1 ^{xiv}	2.8514 (7)
U1—Se1 ^{xi}	2.9605 (5)	P1—U1 ^{vii}	2.8514 (7)
U1—Se1	3.0633 (11)	P1—P1 ⁱ	3.157 (10)
U1—U1 ^{xii}	3.9443 (4)	P1—P1 ^{xviii}	3.157 (10)

U1—U1 ^{xiii}	3.9443 (4)	P1—P1 ^{xix}	3.424 (14)
U1—U1 ^{xiv}	3.9443 (4)		
P1 ⁱ —U1—P1 ⁱⁱ	50.2 (2)	P1 ⁱⁱⁱ —U1—Se1 ^{xi}	139.62 (13)
P1 ⁱⁱ —U1—P1 ⁱⁱⁱ	59.116 (17)	P1 ^{iv} —U1—Se1 ^{xi}	73.66 (10)
P1 ⁱ —U1—P1 ^{iv}	59.116 (17)	P1 ^v —U1—Se1 ^{xi}	73.66 (10)
P1 ⁱⁱⁱ —U1—P1 ^{iv}	67.2 (2)	P1 ^{vi} —U1—Se1 ^{xi}	80.81 (10)
P1 ⁱ —U1—P1 ^v	87.52 (3)	P1 ^{vii} —U1—Se1 ^{xi}	139.62 (13)
P1 ⁱⁱ —U1—P1 ^v	59.116 (17)	P1—U1—Se1 ^{xi}	129.81 (13)
P1 ⁱⁱⁱ —U1—P1 ^v	88.48 (3)	Se1 ^{viii} —U1—Se1 ^{xi}	83.543 (13)
P1 ^{iv} —U1—P1 ^v	50.2 (2)	Se1 ^{ix} —U1—Se1 ^{xi}	83.543 (13)
P1 ⁱ —U1—P1 ^{vi}	88.48 (3)	Se1 ^x —U1—Se1 ^{xi}	140.81 (4)
P1 ⁱⁱ —U1—P1 ^{vi}	67.2 (2)	P1 ⁱ —U1—Se1	135.762 (15)
P1 ⁱⁱⁱ —U1—P1 ^{vi}	87.52 (3)	P1 ⁱⁱ —U1—Se1	135.762 (15)
P1 ^{iv} —U1—P1 ^{vi}	59.116 (17)	P1 ⁱⁱⁱ —U1—Se1	135.762 (15)
P1 ⁱ —U1—P1 ^{vii}	59.116 (17)	P1 ^{iv} —U1—Se1	135.762 (15)
P1 ⁱⁱ —U1—P1 ^{vii}	87.52 (3)	P1 ^v —U1—Se1	135.762 (15)
P1 ⁱⁱⁱ —U1—P1 ^{vii}	50.2 (2)	P1 ^{vi} —U1—Se1	135.762 (15)
P1 ^{iv} —U1—P1 ^{vii}	88.48 (3)	P1 ^{vii} —U1—Se1	135.762 (15)
P1 ^v —U1—P1 ^{vii}	67.2 (2)	P1—U1—Se1	135.762 (15)
P1 ^{vi} —U1—P1 ^{vii}	59.116 (17)	Se1 ^{viii} —U1—Se1	70.41 (2)
P1 ⁱ —U1—P1	67.2 (2)	Se1 ^{ix} —U1—Se1	70.41 (2)
P1 ⁱⁱ —U1—P1	88.48 (3)	Se1 ^x —U1—Se1	70.41 (2)
P1 ⁱⁱⁱ —U1—P1	59.116 (17)	Se1 ^{xi} —U1—Se1	70.41 (2)
P1 ^{iv} —U1—P1	87.52 (3)	P1 ⁱ —U1—P1 ^{xx}	47.14 (19)
P1 ^v —U1—P1	59.116 (17)	P1 ⁱⁱ —U1—P1 ^{xx}	25.69 (12)
P1 ^{vi} —U1—P1	50.2 (2)	P1 ⁱⁱⁱ —U1—P1 ^{xx}	57.61 (9)
P1 ⁱ —U1—Se1 ^{viii}	139.62 (13)	P1 ^{iv} —U1—P1 ^{xx}	34.53 (13)
P1 ⁱⁱ —U1—Se1 ^{viii}	139.62 (13)	P1 ^v —U1—P1 ^{xx}	84.63 (11)
P1 ⁱⁱⁱ —U1—Se1 ^{viii}	129.81 (13)	P1 ^{vi} —U1—P1 ^{xx}	92.92 (11)
P1 ^{iv} —U1—Se1 ^{viii}	129.81 (13)	P1 ^{vii} —U1—P1 ^{xx}	101.21 (11)
P1 ^v —U1—Se1 ^{viii}	80.81 (10)	P1—U1—P1 ^{xx}	105.774 (19)
P1 ^{vi} —U1—Se1 ^{viii}	73.66 (10)	Se1 ^{viii} —U1—P1 ^{xx}	163.21 (3)
P1 ^{vii} —U1—Se1 ^{viii}	80.81 (10)	Se1 ^{ix} —U1—P1 ^{xx}	48.49 (3)
P1—U1—Se1 ^{viii}	73.66 (10)	Se1 ^x —U1—P1 ^{xx}	113.11 (3)
P1 ⁱ —U1—Se1 ^{ix}	73.66 (10)	Se1 ^{xi} —U1—P1 ^{xx}	84.38 (5)
P1 ⁱⁱ —U1—Se1 ^{ix}	73.66 (10)	Se1—U1—P1 ^{xx}	116.05 (3)
P1 ⁱⁱⁱ —U1—Se1 ^{ix}	80.81 (10)	Se1 ^{viii} —U1—P1 ^{xxi}	163.21 (3)
P1 ^{iv} —U1—Se1 ^{ix}	80.81 (10)	Se1 ^{ix} —U1—P1 ^{xxi}	48.49 (2)
P1 ^v —U1—Se1 ^{ix}	129.81 (13)	Se1 ^x —U1—P1 ^{xxi}	84.38 (5)
P1 ^{vi} —U1—Se1 ^{ix}	139.62 (13)	Se1 ^{xi} —U1—P1 ^{xxi}	113.11 (3)
P1 ^{vii} —U1—Se1 ^{ix}	129.81 (13)	Se1—U1—P1 ^{xxi}	116.05 (3)
P1—U1—Se1 ^{ix}	139.62 (13)	U1 ^{viii} —Se1—U1 ^{ix}	140.82 (4)
Se1 ^{viii} —U1—Se1 ^{ix}	140.81 (4)	U1 ^{viii} —Se1—U1 ^x	83.544 (13)
P1 ⁱ —U1—Se1 ^x	80.81 (10)	U1 ^{ix} —Se1—U1 ^x	83.544 (13)
P1 ⁱⁱ —U1—Se1 ^x	129.81 (13)	U1 ^{viii} —Se1—U1 ^{xi}	83.544 (13)
P1 ⁱⁱⁱ —U1—Se1 ^x	73.66 (10)	U1 ^{ix} —Se1—U1 ^{xi}	83.544 (13)
P1 ^{iv} —U1—Se1 ^x	139.62 (13)	U1 ^x —Se1—U1 ^{xi}	140.82 (4)

P1 ^v —U1—Se1 ^x	139.62 (13)	U1 ^{viii} —Se1—U1	109.59 (2)
P1 ^{vi} —U1—Se1 ^x	129.81 (13)	U1 ^{ix} —Se1—U1	109.59 (2)
P1 ^{vii} —U1—Se1 ^x	73.66 (10)	U1 ^x —Se1—U1	109.59 (2)
P1—U1—Se1 ^x	80.81 (10)	U1 ^{xi} —Se1—U1	109.59 (2)
Se1 ^{viii} —U1—Se1 ^x	83.543 (13)	U1 ^{xxii} —P1—U1 ^{xxiii}	176.41 (10)
Se1 ^{ix} —U1—Se1 ^x	83.543 (13)	U1 ^{xii} —P1—U1 ^{xxiii}	131.945 (10)
P1 ⁱ —U1—Se1 ^{xi}	129.81 (13)	U1 ^{xix} —P1—U1 ^{xxiii}	130.116 (13)
P1 ⁱⁱ —U1—Se1 ^{xi}	80.81 (10)	U1 ^{xxiv} —P1—U1 ^{xxiii}	108.344 (10)

Symmetry codes: (i) $y-1, -x, -z$; (ii) $-x, -y, -z$; (iii) $-y, x, z$; (iv) $x, y-1, z$; (v) $y, -x, -z$; (vi) $-y+1, x, z$; (vii) $-x, -y+1, -z$; (viii) $-x+1/2, -y+1/2, -z+1/2$; (ix) $-x-1/2, -y-1/2, -z+1/2$; (x) $-x-1/2, -y+1/2, -z+1/2$; (xi) $-x+1/2, -y-1/2, -z+1/2$; (xii) $x+1, y, z$; (xiii) $x-1, y, z$; (xiv) $x, y+1, z$; (xv) $y, -x+1, -z$; (xvi) $-y+1, x+1, z$; (xvii) $y-1, -x+1, -z$; (xviii) $-y, x+1, z$; (xix) $-x+1, -y+1, -z$; (xx) $-y, x-1, z$; (xxi) $x-1, y-1, z$; (xxii) $x+1, y+1, z$; (xxiii) $-x-1, -y, -z$; (xxiv) $-x+1, -y, -z$.