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Caesium diuranium hexatelluride

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Key indicators: single-crystal X-ray study; T = 100 K; mean σ(Te–Te) = 0.001 Å; disorder in main residue; R factor = 0.022; wR factor = 0.053; data-to-parameter ratio = 32.2.

Single crystals of CsU_2Te_6 were synthesized from the reaction of U, Te, and Cs_2Te_3 at 1273 K. CsU_2Te_6 crystallizes in the space group *Cmcm* in the $CsTh_2Te_6$ structure type. The asymmetric unit comprises one U (site symmetry *m*2*m*), one Cs (*m*2*m*; half-occupancy) and two Te atoms (*m*.. and *m*2*m*). The structure of CsU_2Te_6 consists of infinite $[U_2Te_6]$ layers perpendicular to [010] separated by Cs atoms. There are infinite Te—Te—Te linear chains along [001].

Related literature

For related structures, see: Narducci & Ibers (1998); Chan *et al.* (2004); Bugaris *et al.* (2010); Choi *et al.* (1998); Cody & Ibers (1996); Mizoguchi *et al.* (2006); Tougait *et al.* (1997); Krönert & Plieth (1965); Wu *et al.* (1997). For synthetic details, see: Bugaris & Ibers (2008); Haneveld & Jellinek (1969). For standardization of structural data, see: Gelato & Parthé (1987).

Experimental

Crystal data

CsU_2Te_6
M_r = 1374.57
Orthorhombic, *Cmcm*
a = 4.2129 (2) Å
b = 25.6317 (11) Å
c = 6.0385 (2) Å
V = 652.06 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 40.65 mm^{-1}
T = 100 K
0.21 × 0.03 × 0.02 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: numerical face-indexed (*SADABS*; Sheldrick, 2008*a*)
T_{min} = 0.043, T_{max} = 0.482
5645 measured reflections
611 independent reflections
574 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.053
S = 1.22
611 reflections
19 parameters
$\Delta\rho_{max}$ = 3.89 e Å^{-3}
$\Delta\rho_{min}$ = −1.87 e Å^{-3}

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008*b*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008*b*); 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: BR2209).

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S1. Comment

CsU_2Te_6 (Figure 1) belongs to the AAn_2Q_6 (A= K, Rb, Cs, or Tl; An = U, Th, or Np; Q = S, Se, or Te) family. Compounds in this family crystallize in two different structures types: $CsTh_2Te_6$ (Cody & Ibers, 1996) (space group *Cmcm*) and KTh_2Se_6 (Choi *et al.*, 1998; Wu *et al.*, 1997) (space group *Immm*). Both structure types have AnQ_3 layers intercalated with A atoms. The difference between the two structure types is that each successive AnQ_3 layer in the *Cmcm* structure type is shifted by a/2, whereas each successive AnQ_3 layer in the *Immm* structure type is shifted by (a + b)/2 (Mizoguchi *et al.*, 2006). The AnQ_3 layers are analogous to those in the structure of $ZrSe_3$ (Krönert & Plieth, 1965). The $CsTh_2Te_6$ structure type is adopted by KTh_2Te_6 (Wu *et al.*, 1997) and $Tl_{1.12}UTe_6$ (Tougait *et al.*, 1997). The KTh_2Se_6 structure type is adopted by $RbTh_2Se_6$ (Choi *et al.*, 1998), $K_{0.91}U_{1.79}S_6$ (Mizoguchi *et al.*, 2006), KU_2Se_6 (Chan *et al.*, 2004; Mizoguchi *et al.*, 2006), CsU_2Se_6 (Choi *et al.*, 1998), $CsTh_2Se_6$, $Rb_{0.85}U_{1.74}S_6$, RbU_2Se_6, $Cs_{0.88}(La_{0.68}U_{1.32})Se_6$, KNp_2Se_6, $CsNp_2Se_6$, and TlU_2Se_6 (Bugaris *et al.*, 2010). The structures of the two last compounds are modulated and were refined in 5a *x* 5b *x* 5c and 4a *x* 4b superlattices, respectively.

S2. Experimental

Black needles of CsU_2Te_6 were obtained by direct combination of ^{238}U (30 mg, 12.9 mmol), Te (20.9 mg, 16.4 mmol, Aldrich, 99.8%) and Cs_2Te_3 (24.6 mg, 37.9 mmol). Cs_2Te_3 was prepared by the stoichiometric reaction of Cs (Alfa Aesar, 99.8%) and Te in liquid NH_3 at 194 K. U powder obtained by hydridization and decomposition of turnings (depleted, ORNL) by heating under vacuum, in a modification (Bugaris & Ibers, 2008) of a previous literature method (Haneveld & Jellinek, 1969). The starting reagents were loaded in a carbon-coated fused-silica tube under an Ar atmosphere in a glove box, then evacuated to 10^{-4} Torr, and flame sealed. The tube was placed in computer-controlled furnace, heated to 1273 K in 48 h, held there for 4 h, cooled to 1223 K in 12 h and kept there for 8 d, then cooled to 293 K at 3 K/ h. Black needles were selected and analyzed by EDX and showed the formation of Cs:U:Te in a 1:2:6 ratio. The yield, based on U, was about 15% of the product.

S3. Refinement

The highest peak (3.9 e^- $Å^{-3}$) is 0.76 Å from atom Te1 and the deepest hole (1.9 e^- $Å^{-3}$) is 0.78 Å from atom U1. These should be compared with the height of 225 e^- $Å^{-3}$ of atom Te(1) in an electron density map.

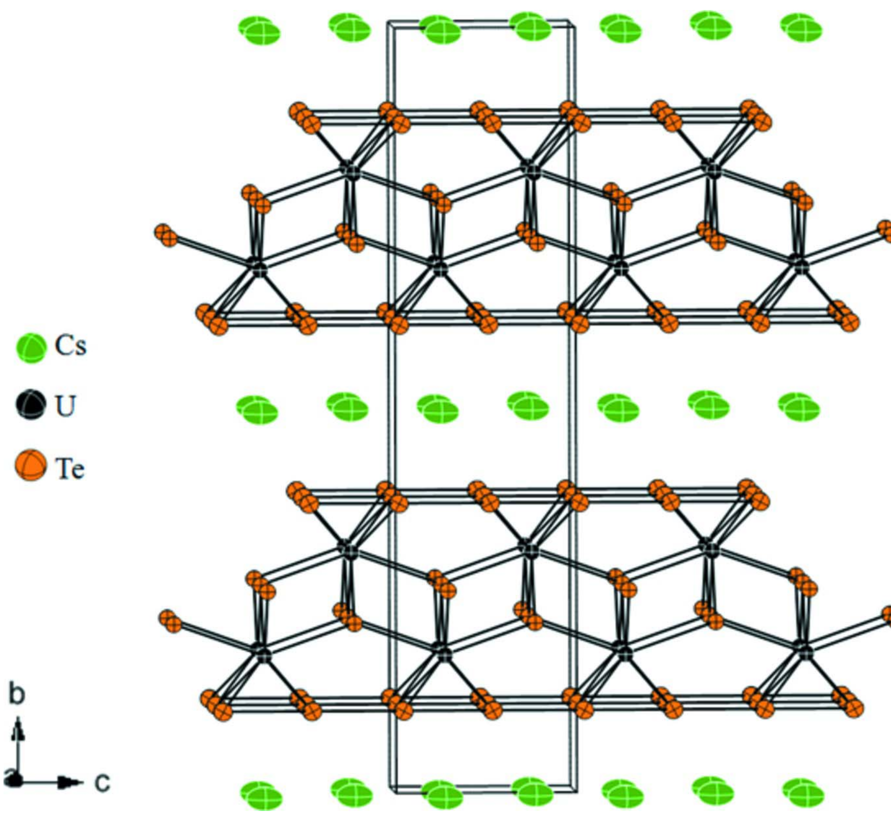

Figure 1

Structure of CsU_2Te_6 viewed approximately down [100]. Displacement ellipsoids are drawn at the 95% probability level.

Caesium diuranium hexatelluride

Crystal data

CsU_2Te_6
M_r = 1374.57
Orthorhombic, *Cmcm*
Hall symbol: -C 2c 2
a = 4.2129 (2) Å
b = 25.6317 (11) Å
c = 6.0385 (2) Å
V = 652.06 (5) Å^3
Z = 2
F(000) = 1102
D_x = 7.001 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2738 reflections
θ = 6.4–60.9°
μ = 40.65 mm^{-1}
T = 100 K
Needle, black
0.21 × 0.03 × 0.02 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, 2008*a*)
T_{min} = 0.043, T_{max} = 0.482
5645 measured reflections
611 independent reflections
574 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 30.7°, θ_{min} = 3.2°
h = −5→3
k = −36→36
l = −8→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.053$
$S = 1.22$
611 reflections
19 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
$[1.00000]/[\sigma^2(F_o^2) + (0.0298F_o^2)^2]$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 3.89$ e Å^{-3}
$\Delta\rho_{min} = -1.87$ e Å^{-3}

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.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
U1	0.0000	0.685078 (14)	0.2500	0.00916 (11)	
Cs1	0.0000	0.49825 (7)	0.2500	0.0556 (7)	0.50
Te1	0.0000	0.116539 (18)	0.00249 (7)	0.01098 (13)	
Te2	0.0000	0.27322 (2)	0.2500	0.00877 (14)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
U1	0.0072 (2)	0.01045 (17)	0.00982 (17)	0.000	0.000	0.000
Cs1	0.100 (2)	0.0194 (9)	0.0475 (12)	0.000	0.000	0.000
Te1	0.0087 (3)	0.0118 (2)	0.0125 (2)	0.000	0.000	−0.00052 (15)
Te2	0.0079 (3)	0.0090 (3)	0.0095 (3)	0.000	0.000	0.000

Geometric parameters (Å, º)

U1—Te2^{i}	3.0890 (5)	Cs1—Te1^{iii}	3.9830 (15)
U1—Te2^{ii}	3.0890 (5)	Cs1—Te1^{ii}	3.9830 (15)
U1—Te1^{ii}	3.1237 (4)	Cs1—Cs1^{xi}	4.2129 (2)
U1—Te1^{iii}	3.1237 (4)	Cs1—Cs1^{xii}	4.2129 (2)
U1—Te1^{iv}	3.1237 (4)	Te1—Te1^{xiii}	2.9892 (8)
U1—Te1^{i}	3.1237 (4)	Te1—Te1^{xiv}	3.0493 (8)
U1—Te2^{v}	3.2028 (3)	Te1—U1^{xv}	3.1237 (4)
U1—Te2^{vi}	3.2028 (3)	Te1—U1^{xvi}	3.1237 (4)
U1—Cs1	4.7888 (19)	Te1—Cs1^{vii}	3.9265 (14)
Cs1—Cs1^{v}	3.0206 (2)	Te1—Cs1^{ix}	3.9265 (14)
Cs1—Cs1^{vi}	3.0206 (2)	Te1—Cs1^{xvi}	3.9830 (15)
Cs1—Te1^{vii}	3.9266 (14)	Te1—Cs1^{xv}	3.9830 (15)

Cs1—Te1^{viii}	3.9266 (14)	Te2—U1^{xvi}	3.0889 (5)
Cs1—Te1^{ix}	3.9266 (14)	Te2—U1^{xv}	3.0889 (5)
Cs1—Te1^{x}	3.9266 (14)	Te2—U1^{v}	3.2028 (3)
Cs1—Te1^{iv}	3.9830 (15)	Te2—U1^{vi}	3.2028 (3)
Cs1—Te1^{i}	3.9830 (15)		

Te2^{i}—U1—Te2^{ii}	85.989 (18)	Te1^{ix}—Cs1—Te1^{iii}	178.90 (3)
Te2^{i}—U1—Te1^{ii}	150.600 (9)	Te1^{x}—Cs1—Te1^{iii}	135.105 (5)
Te2^{ii}—U1—Te1^{ii}	87.220 (10)	Te1^{iv}—Cs1—Te1^{iii}	63.86 (3)
Te2^{i}—U1—Te1^{iii}	150.600 (9)	Te1^{i}—Cs1—Te1^{iii}	80.85 (4)
Te2^{ii}—U1—Te1^{iii}	87.220 (10)	Cs1^{v}—Cs1—Te1^{ii}	110.64 (7)
Te1^{ii}—U1—Te1^{iii}	57.172 (15)	Cs1^{vi}—Cs1—Te1^{ii}	66.56 (5)
Te2^{i}—U1—Te1^{iv}	87.220 (10)	Te1^{vii}—Cs1—Te1^{ii}	98.104 (9)
Te2^{ii}—U1—Te1^{iv}	150.600 (9)	Te1^{viii}—Cs1—Te1^{ii}	115.619 (7)
Te1^{ii}—U1—Te1^{iv}	111.555 (18)	Te1^{ix}—Cs1—Te1^{ii}	135.105 (5)
Te1^{iii}—U1—Te1^{iv}	84.808 (13)	Te1^{x}—Cs1—Te1^{ii}	178.90 (3)
Te2^{i}—U1—Te1^{i}	87.220 (10)	Te1^{iv}—Cs1—Te1^{ii}	80.85 (4)
Te2^{ii}—U1—Te1^{i}	150.600 (9)	Te1^{i}—Cs1—Te1^{ii}	63.86 (3)
Te1^{ii}—U1—Te1^{i}	84.808 (13)	Te1^{iii}—Cs1—Te1^{ii}	44.08 (2)
Te1^{iii}—U1—Te1^{i}	111.555 (18)	Cs1^{v}—Cs1—Cs1^{xi}	90.0
Te1^{iv}—U1—Te1^{i}	57.172 (15)	Cs1^{vi}—Cs1—Cs1^{xi}	90.0
Te2^{i}—U1—Te2^{v}	75.873 (8)	Te1^{vii}—Cs1—Cs1^{xi}	57.556 (13)
Te2^{ii}—U1—Te2^{v}	75.873 (8)	Te1^{viii}—Cs1—Cs1^{xi}	57.556 (13)
Te1^{ii}—U1—Te2^{v}	129.697 (9)	Te1^{ix}—Cs1—Cs1^{xi}	122.442 (13)
Te1^{iii}—U1—Te2^{v}	74.730 (11)	Te1^{x}—Cs1—Cs1^{xi}	122.442 (13)
Te1^{iv}—U1—Te2^{v}	74.730 (11)	Te1^{iv}—Cs1—Cs1^{xi}	121.930 (13)
Te1^{i}—U1—Te2^{v}	129.697 (9)	Te1^{i}—Cs1—Cs1^{xi}	121.930 (13)
Te2^{i}—U1—Te2^{vi}	75.873 (8)	Te1^{iii}—Cs1—Cs1^{xi}	58.072 (13)
Te2^{ii}—U1—Te2^{vi}	75.873 (8)	Te1^{ii}—Cs1—Cs1^{xi}	58.072 (13)
Te1^{ii}—U1—Te2^{vi}	74.730 (11)	Cs1^{v}—Cs1—Cs1^{xii}	90.0
Te1^{iii}—U1—Te2^{vi}	129.697 (9)	Cs1^{vi}—Cs1—Cs1^{xii}	90.0
Te1^{iv}—U1—Te2^{vi}	129.697 (9)	Te1^{vii}—Cs1—Cs1^{xii}	122.442 (13)
Te1^{i}—U1—Te2^{vi}	74.730 (11)	Te1^{viii}—Cs1—Cs1^{xii}	122.442 (13)
Te2^{v}—U1—Te2^{vi}	141.01 (2)	Te1^{ix}—Cs1—Cs1^{xii}	57.556 (13)
Te2^{i}—U1—Cs1	137.005 (9)	Te1^{x}—Cs1—Cs1^{xii}	57.556 (13)
Te2^{ii}—U1—Cs1	137.005 (9)	Te1^{iv}—Cs1—Cs1^{xii}	58.072 (13)
Te1^{ii}—U1—Cs1	55.777 (9)	Te1^{i}—Cs1—Cs1^{xii}	58.072 (13)
Te1^{iii}—U1—Cs1	55.777 (9)	Te1^{iii}—Cs1—Cs1^{xii}	121.930 (13)
Te1^{iv}—U1—Cs1	55.777 (9)	Te1^{ii}—Cs1—Cs1^{xii}	121.930 (13)
Te1^{i}—U1—Cs1	55.777 (9)	Cs1^{xi}—Cs1—Cs1^{xii}	180.0
Te2^{v}—U1—Cs1	109.494 (12)	Te1^{xiii}—Te1—Te1^{xiv}	180.00 (3)
Te2^{vi}—U1—Cs1	109.494 (12)	Te1^{xiii}—Te1—U1^{xv}	61.414 (8)
Cs1^{v}—Cs1—Cs1^{vi}	176.59 (14)	Te1^{xiv}—Te1—U1^{xv}	118.586 (8)
Cs1^{v}—Cs1—Te1^{vii}	114.23 (7)	Te1^{xiii}—Te1—U1^{xvi}	61.414 (8)
Cs1^{vi}—Cs1—Te1^{vii}	68.54 (5)	Te1^{xiv}—Te1—U1^{xvi}	118.586 (8)
Cs1^{v}—Cs1—Te1^{viii}	68.54 (5)	U1^{xv}—Te1—U1^{xvi}	84.806 (13)
Cs1^{vi}—Cs1—Te1^{viii}	114.23 (7)	Te1^{xiii}—Te1—Cs1^{vii}	112.848 (11)
Te1^{vii}—Cs1—Te1^{viii}	45.70 (2)	Te1^{xiv}—Te1—Cs1^{vii}	67.152 (11)

$Cs1^{v}$—Cs1—$Te1^{ix}$	114.23 (7)	$U1^{xv}$—Te1—$Cs1^{vii}$	165.69 (2)
$Cs1^{vi}$—Cs1—$Te1^{ix}$	68.54 (5)	$U1^{xvi}$—Te1—$Cs1^{vii}$	104.208 (12)
$Te1^{vii}$—Cs1—$Te1^{ix}$	64.89 (3)	$Te1^{xiii}$—Te1—$Cs1^{ix}$	112.848 (11)
$Te1^{viii}$—Cs1—$Te1^{ix}$	82.94 (4)	$Te1^{xiv}$—Te1—$Cs1^{ix}$	67.152 (11)
$Cs1^{v}$—Cs1—$Te1^{x}$	68.54 (5)	$U1^{xv}$—Te1—$Cs1^{ix}$	104.208 (12)
$Cs1^{vi}$—Cs1—$Te1^{x}$	114.23 (7)	$U1^{xvi}$—Te1—$Cs1^{ix}$	165.69 (2)
$Te1^{vii}$—Cs1—$Te1^{x}$	82.94 (4)	$Cs1^{vii}$—Te1—$Cs1^{ix}$	64.89 (3)
$Te1^{viii}$—Cs1—$Te1^{x}$	64.89 (3)	$Te1^{xiii}$—Te1—$Cs1^{xvi}$	67.961 (10)
$Te1^{ix}$—Cs1—$Te1^{x}$	45.70 (2)	$Te1^{xiv}$—Te1—$Cs1^{xvi}$	112.039 (10)
$Cs1^{v}$—Cs1—$Te1^{iv}$	66.56 (5)	$U1^{xv}$—Te1—$Cs1^{xvi}$	127.246 (13)
$Cs1^{vi}$—Cs1—$Te1^{iv}$	110.64 (7)	$U1^{xvi}$—Te1—$Cs1^{xvi}$	83.80 (2)
$Te1^{vii}$—Cs1—$Te1^{iv}$	178.90 (3)	$Cs1^{vii}$—Te1—$Cs1^{xvi}$	44.895 (5)
$Te1^{viii}$—Cs1—$Te1^{iv}$	135.105 (5)	$Cs1^{ix}$—Te1—$Cs1^{xvi}$	81.896 (9)
$Te1^{ix}$—Cs1—$Te1^{iv}$	115.619 (7)	$Te1^{xiii}$—Te1—$Cs1^{xv}$	67.961 (10)
$Te1^{x}$—Cs1—$Te1^{iv}$	98.104 (9)	$Te1^{xiv}$—Te1—$Cs1^{xv}$	112.039 (10)
$Cs1^{v}$—Cs1—$Te1^{i}$	110.64 (7)	$U1^{xv}$—Te1—$Cs1^{xv}$	83.80 (2)
$Cs1^{vi}$—Cs1—$Te1^{i}$	66.56 (5)	$U1^{xvi}$—Te1—$Cs1^{xv}$	127.246 (13)
$Te1^{vii}$—Cs1—$Te1^{i}$	135.105 (5)	$Cs1^{vii}$—Te1—$Cs1^{xv}$	81.896 (9)
$Te1^{viii}$—Cs1—$Te1^{i}$	178.90 (3)	$Cs1^{ix}$—Te1—$Cs1^{xv}$	44.895 (5)
$Te1^{ix}$—Cs1—$Te1^{i}$	98.104 (9)	$Cs1^{xvi}$—Te1—$Cs1^{xv}$	63.86 (3)
$Te1^{x}$—Cs1—$Te1^{i}$	115.619 (7)	$U1^{xvi}$—Te2—$U1^{xv}$	85.992 (18)
$Te1^{iv}$—Cs1—$Te1^{i}$	44.08 (2)	$U1^{xvi}$—Te2—$U1^{v}$	104.126 (8)
$Cs1^{v}$—Cs1—$Te1^{iii}$	66.56 (5)	$U1^{xv}$—Te2—$U1^{v}$	104.126 (8)
$Cs1^{vi}$—Cs1—$Te1^{iii}$	110.64 (7)	$U1^{xvi}$—Te2—$U1^{vi}$	104.126 (8)
$Te1^{vii}$—Cs1—$Te1^{iii}$	115.619 (7)	$U1^{xv}$—Te2—$U1^{vi}$	104.126 (8)
$Te1^{viii}$—Cs1—$Te1^{iii}$	98.104 (9)	$U1^{v}$—Te2—$U1^{vi}$	141.01 (2)

Symmetry codes: (i) *x*+1/2, *y*+1/2, *z*; (ii) *x*−1/2, *y*+1/2, *z*; (iii) *x*−1/2, *y*+1/2, −*z*+1/2; (iv) *x*+1/2, *y*+1/2, −*z*+1/2; (v) −*x*, −*y*+1, −*z*+1; (vi) −*x*, −*y*+1, −*z*; (vii) −*x*−1/2, −*y*+1/2, −*z*; (viii) −*x*−1/2, −*y*+1/2, *z*+1/2; (ix) −*x*+1/2, −*y*+1/2, −*z*; (x) −*x*+1/2, −*y*+1/2, *z*+1/2; (xi) *x*−1, *y*, *z*; (xii) *x*+1, *y*, *z*; (xiii) *x*, *y*, −*z*+1/2; (xiv) *x*, *y*, −*z*−1/2; (xv) *x*+1/2, *y*−1/2, *z*; (xvi) *x*−1/2, *y*−1/2, *z*.