inorganic papers

$Cs_2(SO_4)_{0.57}(SeO_4)_{0.43}\cdot Te(OH)_6$, an adduct between dicaesium sulfate selenate and telluric acid

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The title compound, dicaesium sulfate selenate–telluric acid adduct, $Cs_2(SO_4)_{0.57}(SeO_4)_{0.43}\cdot Te(OH)_6$, is a solid solution in the series $Cs_2(SO_4)\cdot Te(OH)_6/Cs_2(SeO_4)\cdot Te(OH)_6$. It crystallizes in the same structure as the end member $Cs_2(SeO_4)\cdot Te(OH)_6$ in space group $P2_1/c$, whereas the corresponding sulfate adopts another structure type and crystallizes in space group $R3$. The structure contains planes of statistically distributed SO_4/SeO_4 tetrahedra alternating with planes of $Te(OH)_6$ octahedra, and with Cs^+ cations situated between the planes. Both Te atoms lie on centres of symmetry.

Comment

Pursuing our study of adducts between sulfate and/or selenate salts with telluric acid, among which we have solved the crystal structures of $Cs_2SO_4\cdot Te(OH)_6$, $Cs_2SeO_4\cdot Te(OH)_6$ (Dammak *et al.*, 2001) and $Rb_2(SO_4)_{0.5}(SeO_4)_{0.5}\cdot Te(OH)_6$ (Abdelhedi *et al.*, 2005), we have grown crystals of $Cs_2(SO_4)_{0.57}(SeO_4)_{0.43}\cdot Te(OH)_6$, (I), from a solution of Cs_2SO_4, Cs_2SeO_4 and telluric acid.

Compound (I) is a solid solution in the series $Cs_2(SO_4)\cdot Te(OH)_6/Cs_2(SeO_4)\cdot Te(OH)_6$ and crystallizes in the same space group as the end member $Cs_2(SeO_4)\cdot Te(OH)_6$, whereas the sulfate end member adopts another structure type in space group $R3$ (Dammak *et al.*, 2001). In (I), the S and Se atoms are statistically distributed over the same site. The crystal structure can be regarded as being built up of planes of $Te(OH)_6$ octahedra (at $x = 0$ and $\frac{1}{2}$) alternating with planes of XO_4 tetrahedra (at $x = \frac{1}{4}$ and $\frac{3}{4}$; X = S and Se), with Cs^+ cations intercalated between the planes (Figs. 1 and 2). In the XO_4 tetrahedra, the X—O distances range from 1.511 (9) Å to
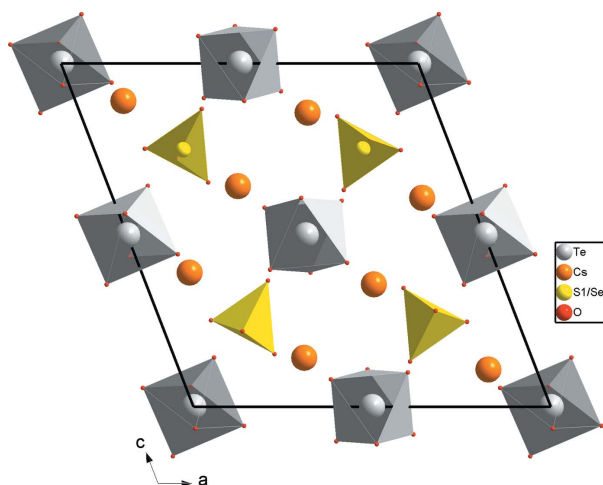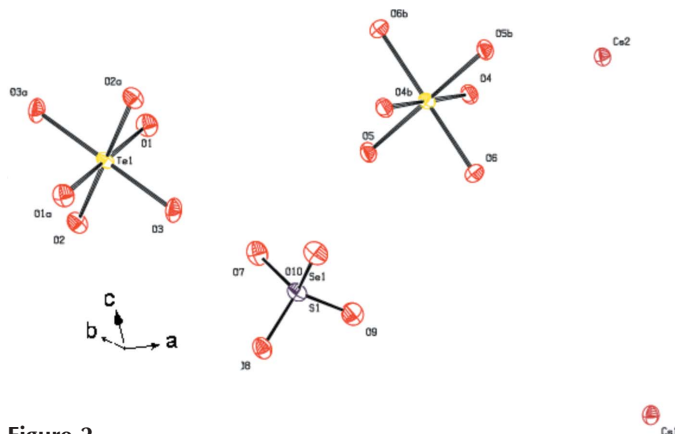

Figure 1
Projection of the crystal structure of (I) on to the *ac* plane.

Figure 2
Part of the crystal structure of (I), with displacement parameters drawn at the 50% probability level. [Symmetry codes: (a) $-x, -y, 1-z$; (b) $1-x, 1-y, 1-z$.]

1.558 (9) Å, intermediate between those observed in the end members $Cs_2SO_4 \cdot Te(OH)_6$ [1.399 (10)–1.405 (7) Å] and $Cs_2SeO_4 \cdot Te(OH)_6$ [1.630 (4)–1.649 (4) Å; Dammak *et al.*, 2001]. The Te atoms occupy two sites, giving rise to two kinds of octahedra, *viz.* $Te1O_6$ and $Te2O_6$, having bond lengths and angles similar to those in $K_2SO_4 \cdot Te(OH)_6$ (Zilber *et al.*, 1980) and $Tl_2SO_4 \cdot Te(OH)_6$ (Zilber *et al.*, 1982) (Table 1). The coordination environments around both Cs cations are slightly different, with Cs1 having ten coordination partners and Cs2 having 11 coordination partners (Table 1; Figs. 3 and 4). In contrast, the Rb cations in $Rb_2(SO_4)_{0.5}(SeO_4)_{0.5} \cdot Te(OH)_6$ (Abdelhedi *et al.*, 2005) and the Cs cations in $Cs_2SeO_4 \cdot Te(OH)_6$ (Dammak *et al.*, 2001) are ninefold coordinate.
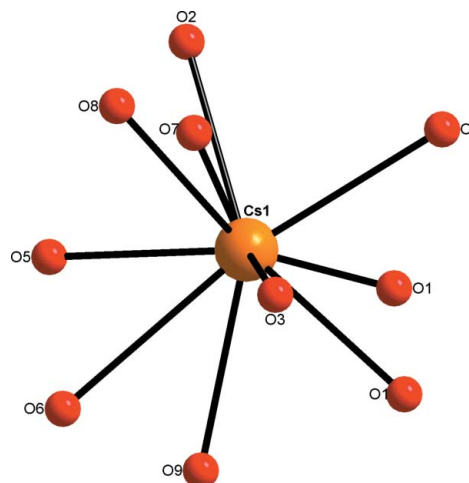
Experimental

Crystals were grown at room temperature by evaporating an aqueous solution of telluric acid, H_6TeO_6 (Aldrich, 99%), caesium carbonate, Cs_2CO_3 (Aldrich 99.9%), selenic acid, H_2SeO_4 (Aldrich 94%) and caesium sulfate Cs_2SO_4, (Aldrich 99.999%) (in the stoichiometric ratio 1:0.5:0.5:0.5). After a few days, colourless single crystals of $Cs_2(SO_4)_{0.57}(SeO_4)_{0.43} \cdot Te(OH)_6$ were obtained.

Crystal data

$Cs_2(SO_4)_{0.57}(SeO_4)_{0.43} \cdot Te(OH)_6$
$M_r = 611.68$
Monoclinic, $P2_1/c$
$a = 12.2646 (4)$ Å
$b = 7.3926 (4)$ Å
$c = 12.6354 (6)$ Å
$\beta = 111.095 (3)°$
$V = 1068.84 (9)$ Å^3
$Z = 4$

$D_x = 3.765$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 986 reflections
$\theta = 1.8–25.8°$
$\mu = 11.12$ mm^{-1}
$T = 298$ K
Prism, colourless
$0.10 \times 0.05 \times 0.05$ mm

Data collection

Nonius KappaCCD diffractometer
φ scans
Absorption correction: multi-scan (using intensity measurements) MULABS in *PLATON* (Spek, 2003)
$T_{min} = 0.541$, $T_{max} = 0.573$
2044 measured reflections

1881 independent reflections
1216 reflections with $I > 3\sigma(I)$
$R_{int} = 0.042$
$\theta_{max} = 25.8°$
$h = -14 \rightarrow 14$
$k = -8 \rightarrow 0$
$l = 0 \rightarrow 14$

Figure 3
The coordination of Cs1.

Refinement

Refinement on F
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.035$
$S = 0.94$
1216 reflections
114 parameters
H-atom parameters not refined
Weighting scheme: Chebychev polynomial (Watkin, 1994; Prince, 1982)
$w = [1 - (F_o^2 - F_c^2)^2/36\sigma^2(F_o)]^2/[2.38T_0(x) - 2.95T_1(x) + 2.50T_2(x) - 1.02T_3(x) + 0.340T_4(x)]$, where $x = F/F_{max}$; robust weighting (Prince, 1982)

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 2.43$ e Å^{-3}
$\Delta\rho_{min} = -2.54$ e Å^{-3}
Extinction correction: Larson (1970)
Extinction coefficient: 17.2 (14)

Table 1
Selected geometric parameters (Å, °).

X is disordered S/Se on a single site.

Te1—O1^i	1.935 (8)	Cs1—O8^{iv}	3.334 (9)
Te1—O2^i	1.917 (8)	Cs1—O1^{vii}	3.413 (9)
Te1—O3^i	1.896 (8)	Cs1—O6^{viii}	3.460 (9)
Te2—O4^{ii}	1.937 (8)	Cs1—O7^{iii}	3.698 (9)
Te2—O6^{ii}	1.925 (8)	Cs2—O6^{ix}	3.098 (8)
Te2—O5^{ii}	1.905 (8)	Cs2—O4	3.109 (8)
X1—O7	1.511 (9)	Cs2—O2^x	3.133 (9)
X1—O8	1.526 (8)	Cs2—O3^{ii}	3.153 (9)
X1—O9	1.558 (9)	Cs2—O9^{iv}	3.165 (9)
X1—O10	1.546 (8)	Cs2—O8^{xi}	3.248 (9)
Cs1—O5^{iii}	2.988 (9)	Cs2—O4^{iv}	3.301 (8)
Cs1—O1^{iii}	3.065 (9)	Cs2—O5^{iv}	3.337 (8)
Cs1—O3^{iv}	3.116 (9)	Cs2—O10^{ii}	3.404 (8)
Cs1—O2^v	3.147 (9)	Cs2—O10^{ix}	3.639 (10)
Cs1—O7^v	3.255 (9)	Cs2—O6^{iv}	3.675 (9)
Cs1—O9^{vi}	3.292 (9)		
O1^i—Te1—O2^i	86.5 (4)	O6^{ii}—Te2—O4	92.1 (4)
O1^i—Te1—O3^i	91.3 (4)	O5^{ii}—Te2—O4	91.5 (3)
O2^i—Te1—O3^i	88.5 (4)	O6^{ii}—Te2—O5	92.2 (4)
O2^i—Te1—O1	93.5 (4)	O7—X1—O8	110.8 (5)
O3^i—Te1—O1	88.7 (4)	O7—X1—O9	107.3 (4)
O3^i—Te1—O2	91.5 (4)	O8—X1—O9	108.6 (5)
O4^{ii}—Te2—O6^{ii}	87.9 (4)	O7—X1—O10	108.1 (5)
O4^{ii}—Te2—O5^{ii}	88.5 (3)	O8—X1—O10	108.6 (5)
O6^{ii}—Te2—O5^{ii}	87.8 (4)	O9—X1—O10	113.4 (5)

Symmetry codes: (i) $-x, -y, -z+1$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$; (iv) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (v) $x+1, y, z$; (vi) $-x+1, -y, -z$; (vii) $x+1, -y+\frac{1}{2}, z-\frac{1}{2}$; (viii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (ix) $x, y+1, z$; (x) $x+1, y+1, z$; (xi) $-x+1, y+\frac{3}{2}, -z+\frac{1}{2}$.
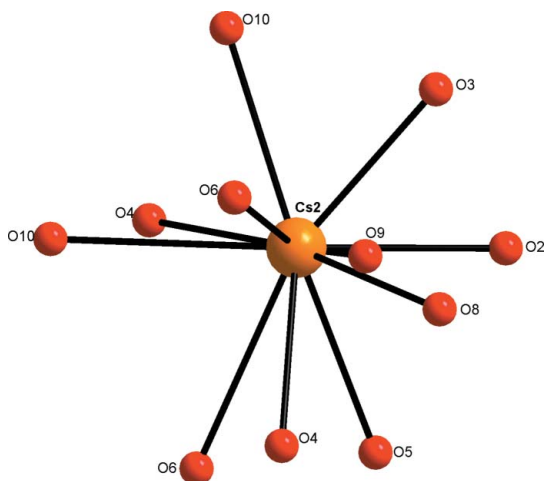
Figure 4
The coordination of Cs2.

For the refinement of the occupation factors for S and Se, their sum was restrained to be equal to 1. The highest peak is situated 1.00 Å from Cs2, and the deepest hole 0.63 Å from S/Se. H atoms could not be located.

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*;

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