Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Redetermination of $SrTe_3O_8$ from a hydrothermally grown single crystal

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Received 11 March 2007
Accepted 28 March 2007

Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(e–Te) = 0.001 Å
R factor = 0.023
wR factor = 0.058
Data-to-parameter ratio = 15.1

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

The crystal structure of the mixed-valence tellurium compound strontium ditellurate(IV) tellurate(VI) was redetermined from single-crystal data. The previous structural model, determined *ab initio* from powder X-ray diffraction data [Barrier *et al.* (2006). *J. Solid State Chem.* **179**, 3484–3488], was confirmed, but with all atoms refined with anisotropic displacement parameters and with differences in individual Te—O bond lengths up to 0.05 Å. The structure is composed of $[Te_3O_8]_\infty^{2-}$ chains that extend parallel to [001] and [110] to form a channel structure. Whereas one half of the channel is occupied by the Sr atoms in an eightfold coordination, the other half remains unoccupied, with the electron lone pairs of Te^{IV} protruding into the empty space. Except for one O atom, all other atoms are on special positions: Sr ($\bar{4}$.. symmetry), Te1 (*m*..), Te2 (2/*m*..), O1 (*m*..) and O2 (*m*..).

Comment

In connection with studies of the preparation and crystal chemistry of mixed-valence $Te^{IV/VI}$ oxocompounds, *viz.* $Hg_2Te_2O_7$ (Weil, 2003), $Cd_2Te_2O_7$, $Cd_3Te_2O_9$ (Weil, 2004) and $Ag_2Te_2O_6$ (Weil, 2007), we turned our attention from d^{10} transition metals to alkaline earth metals, where the crystal structure of the rare mineral carlfriesite, $CaTe_3O_8$, has already been described (Effenberger *et al.*, 1978). During hydrothermal phase formation experiments, we obtained single

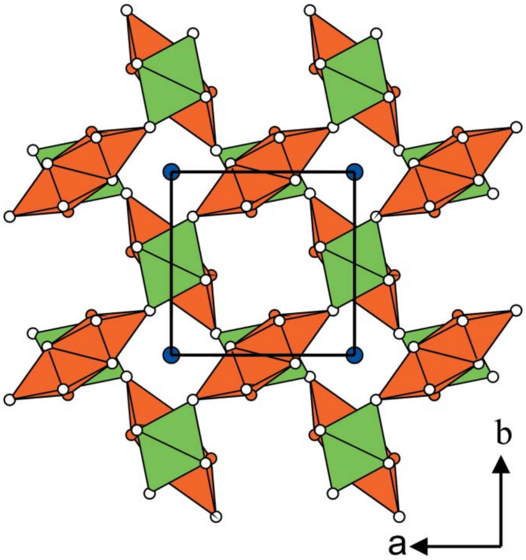

Figure 1
The crystal structure of $SrTe_3O_8$ in projection along [001]. TeO_6 octahedra are green and TeO_5 pyramids are orange, with Te^{IV} atoms shown as orange, O atoms as white and Sr atoms as blue spheres.

crystals of $SrTe_3O_8$ and determined its crystal structure. More or less simultaneously with our study, this structure was determined independently *ab initio* from powder X-ray diffraction data (Barrier *et al.*, 2006). In comparison with the powder refinement, all atoms are described with anisotropic displacement parameters instead of U_{iso} values, and with differences in individual Te—O bond lengths up to 0.05 Å.

$SrTe_3O_8$ consists of $[Te_3O_8]_\infty{}^{2-}$ chains running parallel to [001] and [110] to establish a framework with two types of square channels. The smaller channels run along [00*z*] and are occupied by Sr atoms, whereas the larger channels run along $[\frac{1}{2}\frac{1}{2}z]$ and are partly filled with the stereoactive electron lone pairs of Te^{IV} (Fig. 1).

The $[Te_3O_8]_\infty{}^{2-}$ chains are made up of a nearly undistorted $Te^{VI}O_6$ octahedron (2/*m*.. symmetry) and a Te_2O_8 double polyhedron, made up of two symmetry-related $Te^{IV}O_5$ pyramids (*m*.. symmetry). These principal structural units are linked *via* the edge of a TeO_6 octahedron and two parallel apices of the Te_2O_8 double polyhedron into a polymeric chain structure (Fig. 2). The connection of chains that run perpendicular to this direction is accomplished by corner-sharing with the terminal O atoms of the TeO_6 octahedra. In this way, each TeO_6 octahedron is linked to four Te_2O_8 double polyhedra, two by edge-sharing and two by corner-sharing, and each Te_2O_8 double polyhedron is linked in the same way by four TeO_6 octahedra. The different Te—O bond lengths and O—Te—O angles for the tetravalent Te1 and the hexavalent Te2 atoms (Table 1) are in the typical ranges observed for other Te^{IV} and Te^{VI} oxocompounds (for reviews see, for example, Zemann, 1971; Dolgikh, 1991; Loub, 1993; Levason, 1997; Weil, 2006), with an average Te^{IV}—O distance of 2.066 Å and a Te^{VI}—O distance of 1.930 Å.

The Sr^{2+} cation exhibits $\overline{4}$ symmetry in a distorted cubic environment with four Sr—O distances of 2.509 (3) Å and four distances of 2.798 (4) Å. The average Sr—O distance (2.654 Å) is in good agreement with the sum of the ionic radii (2.62 Å; Shannon, 1976).

$CaTe_3O_8$ is the only other representative with mixed-valence $Te^{IV/VI}$ and the same formula type $M^{II}Te_3O_8$. Consequently, the crystal structure of the lighter homologue is made up of similar $[Te_3O_8]_\infty{}^{2-}$ chains forming a framework with a channel structure, however, with an entirely different arrangement of the chains and a different filling of the channels. In contrast to $SrTe_3O_8$, the polyanionic chains run along [110] and $[1\overline{1}0]$, and all channels are occupied by Ca atoms in an eightfold coordination, with an average distance of Ca—O = 2.453 Å (Fig. 3).

Experimental

An aqueous $Te(OH)_6$ (1.71 g) solution (10 ml) was added dropwise to a 20 ml solution containing $SrCl_2\cdot 6H_2O$ (1.68 g) and KOH (0.83 g). The white precipitate was filtered off, washed with a water–acetone mixture (1/1) and dried. The resulting material (0.4 g) was placed in a Teflon inlay with 10 ml capacity, which was charged to two-thirds capacity with the mother liquor of the precipitation experiment, sealed, placed in a steel autoclave and heated at 503 K for 100 h.

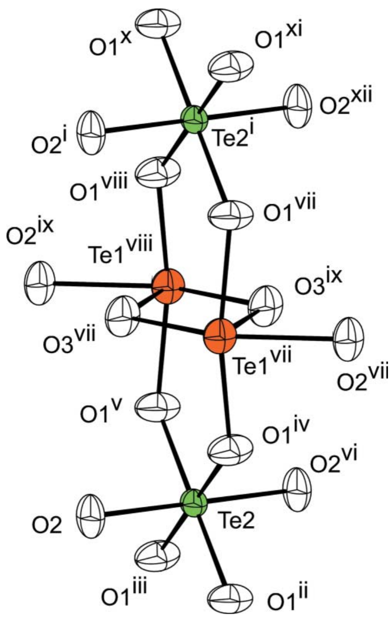

Figure 2
Details of a $[Te_3O_8]_\infty{}^{2-}$ chain with the atom numbering scheme and anisotropic displacement ellipsoids displayed at the 97% probability level. [Symmetry operators: (i) *x*, *y*, *z* + 1; (ii) *y*, 1 − *x*, $\frac{1}{2}$ − *z*; (iii) −*y*, *x*, *z* − $\frac{1}{2}$; (iv) *y*, 1 − *x*, *z* − $\frac{1}{2}$; (v) −*y*, *x*, $\frac{1}{2}$ − *z*; (vi) −*x*, 1 − *y*, −*z*; (vii) *y*,1 − *x*, *z* + $\frac{1}{2}$; (viii) −*y*, *x*, *z* + $\frac{1}{2}$; (ix) *y*, *x*, *z* + $\frac{1}{2}$; (*x*) −*y*, *x*, $\frac{3}{2}$ − *z*; (xi) *y*, 1 − *x*, $\frac{3}{2}$ − *z*; (xii) −*x*, *y* + $\frac{1}{2}$, *z* + 1.]

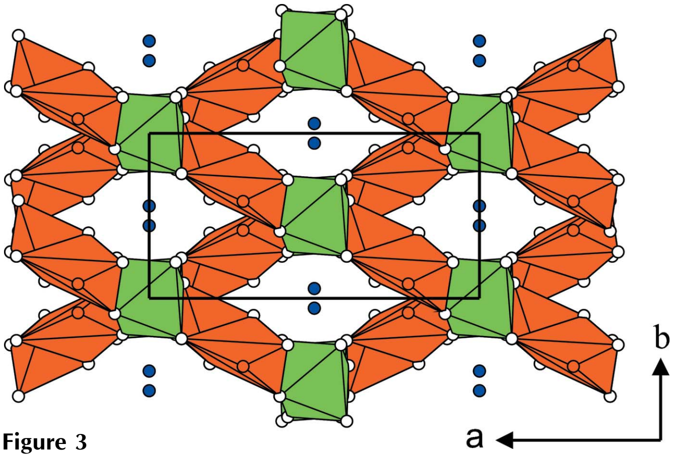

Figure 3
The crystal structure of $CaTe_3O_8$ (Effenberger *et al.*, 1978) in projection along [001]. TeO_6 octahedra are green and TeO_5 pyramids are orange, with Te^{IV} atoms shown as orange, O atoms as white and Ca atoms as blue spheres.

Besides microcrystalline $SrTeO_4$ (Lapasset & Moret, 1985) as the main phase, a few spheroidal transparent crystals of the title compound were obtained, indicating that a part of the hexavalent tellurium was reduced to oxidation state +IV.

Crystal data

$SrTe_3O_8$
M_r = 598.42
Tetragonal, $P4_2/m$
a = 6.8321 (3) Å
c = 6.7605 (5) Å
V = 315.56 (3) Å^3

Z = 2
Mo *Kα* radiation
μ = 22.14 mm^{-1}
T = 295 (2) K
0.07 × 0.06 × 0.06 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002) $T_{\min} = 0.306$, $T_{\max} = 0.350$ (expected range = 0.232–0.265)
3678 measured reflections
530 independent reflections
525 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.023$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.058$
$S = 1.20$
530 reflections
35 parameters
$\Delta\rho_{\max} = 1.04$ e Å^{-3}
$\Delta\rho_{\min} = -2.08$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Te1—O3$^{\rm i}$	1.951 (5)	Te1—O2	2.180 (4)
Te1—O3	2.024 (4)	Te2—O2	1.922 (4)
Te1—O1	2.086 (3)	Te2—O1$^{\rm ii}$	1.934 (3)
O3$^{\rm i}$—Te1—O3	77.6 (2)	O2—Te2—O2$^{\rm iv}$	180.0
O3$^{\rm i}$—Te1—O1	82.93 (10)	O2—Te2—O1$^{\rm ii}$	89.33 (14)
O3—Te1—O1	90.69 (9)	O2$^{\rm iv}$—Te2—O1$^{\rm ii}$	90.67 (14)
O3$^{\rm i}$—Te1—O1$^{\rm iii}$	82.93 (10)	O1$^{\rm ii}$—Te2—O1$^{\rm v}$	94.62 (19)
O1—Te1—O1$^{\rm iii}$	165.14 (19)	O1$^{\rm vi}$—Te2—O1$^{\rm v}$	85.38 (19)
O3$^{\rm i}$—Te1—O2	71.70 (18)	Te2$^{\rm vii}$—O1—Te1	135.24 (17)
O3—Te1—O2	149.32 (18)	Te2—O2—Te1	119.7 (2)
O1—Te1—O2	85.63 (9)	Te1$^{\rm i}$—O3—Te1	102.4 (2)

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

The crystal data were standardized by means of the program *STRUCTURETIDY* (Gelato & Parthé, 1987). The highest remaining peak in the final difference Fourier map is coincident with Te2 and the deepest hole is 0.35 Å away from Sr.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ATOMS* (Dowty, 2006); software used to prepare material for publication: *SHELXL97*.

Financial support of the FWF (Fonds zur Förderung der wissenschaftlichen Forschung), project P19099-N17, is gratefully acknowledged.

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