inorganic compounds

The Mg member of the isotypic series MTe_6O_{13}

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$MgTe_6O_{13}$, magnesium hexatellurate(IV), is isotypic with the structures of divalent first-row transition metal analogues MTe_6O_{13} (M = Mn, Fe, Co, Ni and Zn). The asymmetric unit contains one Mg, two Te and five O atoms of which the Mg and one O atom lie on a threefold rotation axis. The structure is made up from slightly distorted $[MgO_6]$ octahedra (isolated from each other), distorted $[TeO_4]$ bisphenoids and $[TeO_{4 + 1}]$ tetragonal pyramids sharing corners and edges. This arrangement leads to the formation of a dense three-dimensional structure.

Related literature

The title compound is isotypic with its Co, Mn, Ni (Irvine *et al.*, 2003), Fe (van der Lee & Astier, 2007; van der Lee, 2013) and Zn (Nawash *et al.*, 2007) analogues. For other phases in the system Mg–Te–O, see: Trömel & Ziethen-Reichnach (1970). For structure determinations of $MgTe_2O_5$ and $Mg_2Te_3O_8$, see: Weil (2005) and Lin *et al.* (2013), respectively. The crystal chemistry of oxotellurates(IV) has been reviewed by Zemann (1971).

Experimental

Crystal data

$MgTe_6O_{13}$	Z = 6
M_r = 997.91	Mo $K\alpha$ radiation
Trigonal, $R\bar{3}$	μ = 15.38 mm^{-1}
a = 10.1676 (2) Å	T = 295 K
c = 18.9701 (3) Å	0.22 × 0.14 × 0.09 mm
V = 1698.39 (5) Å^3	

Data collection

Bruker APEXII CCD diffractometer	50742 measured reflections
	2875 independent reflections
Absorption correction: multi-scan ($SADABS$; Bruker, 2012)	2764 reflections with $I > 2\sigma(I)$
T_{min} = 0.437, T_{max} = 0.749	R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.017	62 parameters
$wR(F^2)$ = 0.034	$\Delta\rho_{max}$ = 1.17 e Å^{-3}
S = 1.36	$\Delta\rho_{min}$ = −1.40 e Å^{-3}
2875 reflections	

Table 1
Structural data of isotypic MTe_6O_{13} compounds (Å, Å^3) in space group $R\bar{3}$.

M^{2+}	a	c	V
Mg^a	10.1676 (2)	18.9701 (3)	1698.39 (5)
Mn^b	10.2505 (5)	19.2195 (9)	1748.89 (14)
Fe^c	10.16630 (10)	18.9330 (3)	1694.63 (4)
Co^b	10.1641 (5)	18.9814 (9)	1698.23 (14)
Ni^b	10.1522 (5)	18.8669 (9)	1684.30 (14)
Zn^d	10.1283 (9)	18.948 (3)	1683.3 (3)

Notes: (*a*) this work; (*b*) Irvine *et al.* (2003); (*c*) van der Lee & Astier (2007); (*d*) Nawash *et al.* (2007).

Table 2
Selected bond lengths (Å).

Mg1—O5	2.0676 (12)	Te2—O5	1.8489 (11)
Mg1—O2^i	2.1596 (12)	Te2—O4	1.9186 (11)
Te1—O3	1.8524 (12)	Te2—O4^{iii}	2.0286 (12)
Te1—O2	1.9324 (11)	Te2—O3	2.2118 (12)
Te1—O1	2.1314 (2)	Te2—O5^{iv}	2.5908 (12)
Te1—O2^{ii}	2.1842 (11)		

Symmetry codes: (i) $-y+\frac{1}{3}, x-y+\frac{2}{3}, z-\frac{1}{3}$; (ii) $-x+\frac{2}{3}, -y+\frac{1}{3}, -z+\frac{1}{3}$; (iii) $-y+1, x-y+1, z$; (iv) $-x+1, -y+1, -z$.

Data collection: *APEX2* (Bruker, 2012); cell refinement: *SAINT* (Bruker, 2012); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ATOMS for Windows* (Dowty, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PJ2001).

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The Mg member of the isotypic series MTe$_6$O$_{13}$

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S1. Comment

Single crystals of Mg$_2$Te$_3$O$_8$ (Lin *et al.*, 2013) and of the title compound, MgTe$_6$O$_{13}$, were obtained during hydrothermal phase formation studies in the system Mg–Se–Te–O. These two phases, along with MgTe$_2$O$_5$ have already been described as existing phases in the system Mg–Te–O and were characterized by their X-ray diffraction patterns at that time (Trömel & Ziethen-Reichnach, 1970). Whereas for MgTe$_2$O$_5$ (Weil, 2005) and Mg$_2$Te$_3$O$_8$ (Lin *et al.*, 2013) structure determinations from single-crystal data were reported, detailed structure data for MgTe$_6$O$_{13}$ were missing up to now.

   MgTe$_6$O$_{13}$ is a member of the isotypic series MTe$_6$O$_{13}$ crystallizing in space group $R\overline{3}$. Structure determinations were reported for the Co, Mn, Ni (Irvine *et al.*, 2003), Fe (van der Lee & Astier, 2007) and Zn (Nawash *et al.*, 2007) members. It should be mentioned that the given space group $R\overline{3}m$ for FeTe$_6$O$_{13}$ (van der Lee & Astier, 2007) is incorrect. The correct space group in fact is $R\overline{3}$ (van der Lee, 2013). Lattice parameters and volumes of the several MTe$_6$O$_{13}$ phases are in a narrow range (Table 1), as one expects from the similar ionic radii of Mg and the first row transition metals.

   The magnesium cation is located on a threefold rotation axis and is surrounded by six oxygen atoms in a slightly distored octahedral environment. Because of the high tellurium content in the structure, the [MgO$_6$] octahedra are isolated from each other. The two distinct tellurium(IV) atoms exhibit different oxygen environments. Te1 is bonded to four oxygen atoms in form of a bisphenoid with distances ranging from 1.8524 (12) to 2.1842 (11) Å. Considering Te···O separations less than 3.1 Å, two remote O atoms (O3, O5) are also present, leading to a pentagonal pyramid [TeO$_{4+2}$] as the resulting coordination polyhedron. Atom Te2 is bonded to four O atoms with distances ranging from 1.8489 (11) to 2.2118 (12) Å, with an additional O atom 2.5908 (12) Å away. The resulting [TeO$_{4+1}$] polyhedron is a distorted tetragonal pyramid. It is augmented to a distorted octahedron if the remote O4 atom at a distance of 3.0387 (12) Å is also considered. For both [TeO$_x$] polyhedra (Fig. 1, Table 2) the rules derived by Zemann for the crystal chemistry of oxotellurates(IV) are valid (Zemann, 1971).

   The [MgO$_6$], [Te1O$_4$] and [Te2O$_{4+1}$] building units share corners and edges, thereby forming a dense three-dimensional structure (Fig. 2). In a simpler view, the structure can be described as being built up from distorted hexagonal layers of the Mg and Te atoms extending parallel to (001) and stacked in an *ABCA'B'C'* sequence along [001]. The oxygen atoms are situated in the voids of this arrangement.

S2. Experimental

Magnesium oxide, tellurium dioxide and selenic acid (conc.; 96%$_{wt}$) were loaded in the stoichiometric ratio 3:2:1 in a Teflon lined stainless steel autoclave (overall volume 10 ml) that was filled up to two-thirds of its volume with water. The autoclave was then heated at 503 K for one week. Few colourless single crystals of Mg$_2$Te$_3$O$_8$ (Lin *et al.*, 2013) and MgTe$_6$O$_{13}$, both with a platy habit, were isolated from the colourless solid reaction product. X-ray powder diffraction (XRPD) of the ground bulk material revealed Mg$_2$Te$_3$O$_8$ and TeO$_2$ as main products. Se-containing phases could not be identified by XRPD in the solid reaction product.

S3. Refinement

The atomic coordinates of isotypic $ZnTe_6O_{13}$ (Nawash *et al.*, 2007) were used as starting parameters for the refinement. Reflection 003 was affected from the beamstop and was omitted from the refinement. The highest positive and negative residual electron densities are located 0.68 and 0.48 Å, respectively, from atom Te1.

Figure 1
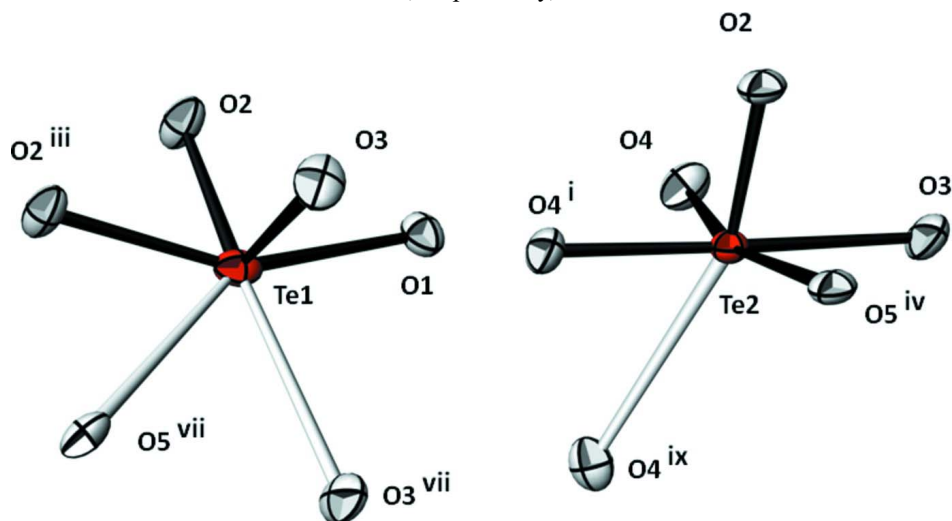
The coordination spheres of the two tellurium(IV) atoms. Te—O bonds < 2.6 Å are given as solid lines, Te—O bonds between 2.6 and 3.1 Å as open lines; probability level of the displacement ellipsoids is 74%. [Symmetry codes: i) $-y + 1$, x-y+1, z; iii) $-x + 2/3$, $-y + 1/3$, $-z + 1/3$; iv) $-x + 1$, $-y + 1$, $-z$; vii) y, $-x$+y, $-z$; ix) x-y+2/3, $x + 1/3$, $-z + 1/3$.]
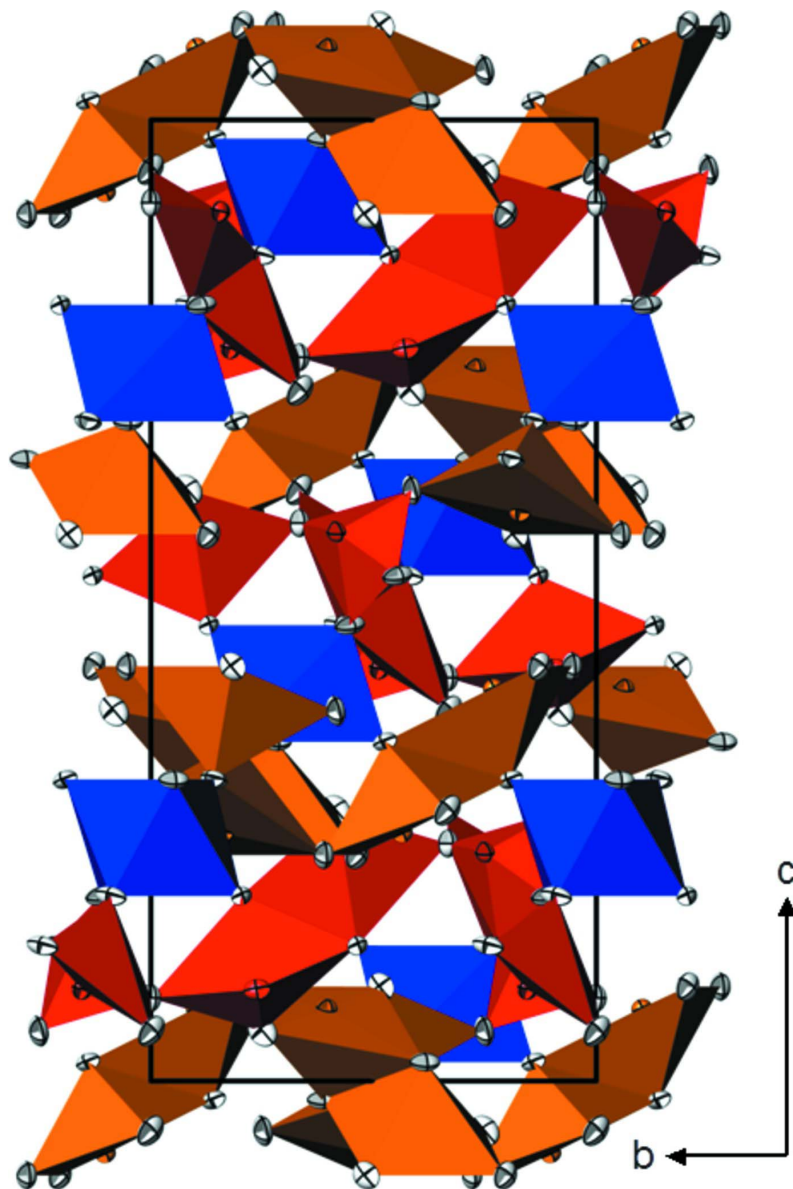
Figure 2

The crystal packing of the MgTe$_6$O$_{13}$ structure in a view along [100]. [MgO$_6$] polyhedra are blue, [Te1O$_4$] polyhedra are red and [Te2O$_{4+1}$] polyhedra are orange. Probability level as in Fig. 1.

Magnesium hexatellurate(IV)

Crystal data

MgTe$_6$O$_{13}$

$M_r = 997.91$

Trigonal, $R\bar{3}$

Hall symbol: -R 3

$a = 10.1676 (2)$ Å

$c = 18.9701 (3)$ Å

$V = 1698.39 (5)$ Å^3

$Z = 6$

$F(000) = 2568$

$D_x = 5.854$ Mg m^{-3}

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9908 reflections

$\theta = 3.2–43.5°$

$\mu = 15.38$ mm^{-1}

$T = 295$ K

Plate, colourless

$0.22 \times 0.14 \times 0.09$ mm

Data collection

Bruker APEXII CCD diffractometer	50742 measured reflections
Radiation source: fine-focus sealed tube	2875 independent reflections
Graphite monochromator	2764 reflections with $I > 2\sigma(I)$
ω– and φ–scans	$R_{int} = 0.044$
Absorption correction: multi-scan	$\theta_{max} = 43.6°$, $\theta_{min} = 2.6°$
($SADABS$; Bruker, 2012)	$h = -19\rightarrow19$
$T_{min} = 0.437$, $T_{max} = 0.749$	$k = -19\rightarrow19$
	$l = -36\rightarrow36$

Refinement

Refinement on F^2	Primary atom site location: isomorphous structure methods
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.017$	$w = 1/[\sigma^2(F_o^2) + 7.6037P]$
$wR(F^2) = 0.034$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.36$	$(\Delta/\sigma)_{max} = 0.002$
2875 reflections	$\Delta\rho_{max} = 1.17$ e Å^{-3}
62 parameters	$\Delta\rho_{min} = -1.40$ e Å^{-3}
0 restraints	Extinction correction: $SHELXL97$ (Sheldrick, 2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
	Extinction coefficient: 0.000970 (18)

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}
Mg1	0.3333	0.6667	−0.07728 (5)	0.00655 (14)
Te1	0.237607 (11)	0.082435 (11)	0.095241 (5)	0.00766 (2)
Te2	0.394752 (11)	0.496265 (11)	0.075896 (5)	0.00633 (2)
O1	0.0000	0.0000	0.08633 (11)	0.0085 (3)
O2	0.20309 (13)	0.11297 (14)	0.19252 (6)	0.01008 (18)
O3	0.25075 (15)	0.25178 (13)	0.05181 (7)	0.01187 (19)
O4	0.20451 (14)	0.48320 (14)	0.09819 (6)	0.00998 (17)
O5	0.38463 (14)	0.52546 (14)	−0.01971 (6)	0.00969 (17)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mg1	0.0066 (2)	0.0066 (2)	0.0065 (3)	0.00329 (11)	0.000	0.000
Te1	0.01113 (4)	0.00635 (4)	0.00675 (4)	0.00530 (3)	0.00084 (2)	0.00065 (2)
Te2	0.00718 (4)	0.00727 (4)	0.00516 (3)	0.00408 (3)	−0.00023 (2)	0.00038 (2)
O1	0.0068 (4)	0.0068 (4)	0.0118 (7)	0.0034 (2)	0.000	0.000
O2	0.0065 (4)	0.0145 (5)	0.0060 (4)	0.0028 (4)	0.0005 (3)	−0.0017 (3)

O3	0.0137 (5)	0.0063 (4)	0.0146 (5)	0.0042 (4)	−0.0030 (4)	0.0017 (3)
O4	0.0090 (4)	0.0112 (4)	0.0123 (4)	0.0069 (4)	0.0017 (3)	0.0017 (3)
O5	0.0143 (5)	0.0116 (4)	0.0055 (4)	0.0083 (4)	−0.0002 (3)	0.0014 (3)

Geometric parameters (Å, º)

Mg1—O5[i]	2.0676 (12)	Te1—O2[vi]	2.1842 (11)
Mg1—O5[ii]	2.0676 (12)	Te1—O3[vii]	2.9337 (13)
Mg1—O5	2.0676 (12)	Te1—O5[vii]	3.0387 (12)
Mg1—O2[iii]	2.1596 (12)	Te2—O5	1.8489 (11)
Mg1—O2[iv]	2.1596 (12)	Te2—O4	1.9186 (11)
Mg1—O2[v]	2.1596 (12)	Te2—O4[i]	2.0286 (12)
Te1—O3	1.8524 (12)	Te2—O3	2.2118 (12)
Te1—O2	1.9324 (11)	Te2—O5[viii]	2.5908 (12)
Te1—O1	2.1314 (2)	Te2—O4[ix]	3.0560 (12)
O5[i]—Mg1—O5[ii]	94.67 (5)	Te1[x]—O1—Te1[xi]	119.377 (15)
O5[i]—Mg1—O5	94.67 (5)	Te1—O2—Mg1[xii]	130.51 (6)
O5[ii]—Mg1—O5	94.67 (5)	Te1—O2—Te1[vi]	105.30 (5)
O5[i]—Mg1—O2[iii]	99.09 (5)	Mg1[xii]—O2—Te1[vi]	122.67 (6)
O5[ii]—Mg1—O2[iii]	76.38 (5)	Te1—O2—Te2[vi]	94.79 (5)
O5—Mg1—O2[iii]	164.10 (5)	Mg1[xii]—O2—Te2[vi]	84.08 (4)
O5[i]—Mg1—O2[iv]	76.38 (5)	Te1[vi]—O2—Te2[vi]	78.72 (4)
O5[ii]—Mg1—O2[iv]	164.10 (5)	Te1—O2—Te1[xi]	75.79 (4)
O5—Mg1—O2[iv]	99.09 (5)	Mg1[xii]—O2—Te1[xi]	74.86 (4)
O2[iii]—Mg1—O2[iv]	91.88 (5)	Te1[vi]—O2—Te1[xi]	140.31 (5)
O5[i]—Mg1—O2[v]	164.10 (5)	Te2[vi]—O2—Te1[xi]	140.94 (4)
O5[ii]—Mg1—O2[v]	99.09 (5)	Te1—O2—Te2[xiii]	124.64 (5)
O5—Mg1—O2[v]	76.38 (5)	Mg1[xii]—O2—Te2[xiii]	77.64 (3)
O2[iii]—Mg1—O2[v]	91.88 (5)	Te1[vi]—O2—Te2[xiii]	81.17 (4)
O2[iv]—Mg1—O2[v]	91.88 (5)	Te2[vi]—O2—Te2[xiii]	139.31 (3)
O3—Te1—O2	102.12 (6)	Te1[xi]—O2—Te2[xiii]	67.54 (2)
O3—Te1—O1	82.59 (5)	Te1—O3—Te2	130.72 (6)
O2—Te1—O1	82.98 (6)	Te1—O3—Te1[xiv]	120.60 (6)
O3—Te1—O2[vi]	90.88 (5)	Te2—O3—Te1[xiv]	105.58 (4)
O2—Te1—O2[vi]	74.70 (5)	Te1—O3—Te1[xi]	91.57 (5)
O1—Te1—O2[vi]	154.89 (5)	Te2—O3—Te1[xi]	107.62 (5)
O3—Te1—O3[vii]	81.28 (3)	Te1[xiv]—O3—Te1[xi]	87.42 (3)
O2—Te1—O3[vii]	172.70 (4)	Te2—O4—Te2[ii]	136.27 (7)
O1—Te1—O3[vii]	91.12 (6)	Te2—O4—Te2[xiii]	106.42 (5)
O2[vi]—Te1—O3[vii]	111.91 (4)	Te2[ii]—O4—Te2[xiii]	103.43 (4)
O3—Te1—O5[vii]	89.65 (5)	Te2—O4—Te1[xi]	115.96 (5)
O2—Te1—O5[vii]	130.80 (4)	Te2[ii]—O4—Te1[xi]	98.97 (4)
O1—Te1—O5[vii]	146.21 (6)	Te2[xiii]—O4—Te1[xi]	82.34 (3)
O2[vi]—Te1—O5[vii]	57.32 (4)	Te2—O4—Te2[i]	71.27 (4)
O3[vii]—Te1—O5[vii]	55.14 (3)	Te2[ii]—O4—Te2[i]	70.62 (3)
O5—Te2—O4	95.29 (5)	Te2[xiii]—O4—Te2[i]	104.98 (3)
O5—Te2—O4[i]	94.31 (5)	Te1[xi]—O4—Te2[i]	168.27 (4)

O4—Te2—O4[i]	93.39 (7)	Te2—O5—Mg1	133.10 (7)
O5—Te2—O3	85.36 (5)	Te2—O5—Te2[viii]	105.65 (5)
O4—Te2—O3	83.67 (5)	Mg1—O5—Te2[viii]	111.82 (5)
O4[i]—Te2—O3	177.00 (5)	Te2—O5—Te1[xiv]	112.67 (5)
O5—Te2—O5[viii]	74.35 (5)	Mg1—O5—Te1[xiv]	94.65 (4)
O4—Te2—O5[viii]	166.09 (5)	Te2[viii]—O5—Te1[xiv]	90.25 (3)
O4[i]—Te2—O5[viii]	96.57 (5)	Te2—O5—Te2[i]	71.20 (4)
O3—Te2—O5[viii]	86.22 (5)	Mg1—O5—Te2[i]	68.01 (4)
O5—Te2—O4[ix]	160.15 (4)	Te2[viii]—O5—Te2[i]	114.99 (4)
O4—Te2—O4[ix]	72.54 (5)	Te1[xiv]—O5—Te2[i]	153.01 (4)
O4[i]—Te2—O4[ix]	71.41 (4)	Te2—O5—Te2[ii]	71.12 (4)
O3—Te2—O4[ix]	108.16 (4)	Mg1—O5—Te2[ii]	67.95 (4)
O5[viii]—Te2—O4[ix]	119.92 (3)	Te2[viii]—O5—Te2[ii]	172.20 (5)
Te1—O1—Te1[x]	119.377 (15)	Te1[xiv]—O5—Te2[ii]	97.55 (3)
Te1—O1—Te1[xi]	119.377 (15)	Te2[i]—O5—Te2[ii]	57.381 (17)

Symmetry codes: (i) −*y*+1, *x*−*y*+1, *z*; (ii) −*x*+*y*, −*x*+1, *z*; (iii) −*y*+1/3, *x*−*y*+2/3, *z*−1/3; (iv) *x*+1/3, *y*+2/3, *z*−1/3; (v) −*x*+*y*+1/3, −*x*+2/3, *z*−1/3; (vi) −*x*+2/3, −*y*+1/3, −*z*+1/3; (vii) *y*, −*x*+*y*, −*z*; (viii) −*x*+1, −*y*+1, −*z*; (ix) *x*−*y*+2/3, *x*+1/3, −*z*+1/3; (x) −*x*+*y*, −*x*, *z*; (xi) −*y*, *x*−*y*, *z*; (xii) *x*−1/3, *y*−2/3, *z*+1/3; (xiii) *y*−1/3, −*x*+*y*+1/3, −*z*+1/3; (xiv) *x*−*y*, *x*, −*z*.