organic compounds

Bis(ethyleneglycolato-κ^2O,O')-tellurium(IV)

Neil R. Brooks,[a] Minxian Wu,[b] Luc Van Meervelt,[a]* Koen Binnemans[a] and Jan Fransaer[b]

[a]KU Leuven–Universtiy of Leuven, Department of Chemistry, Celestijnenlaan 200F, B-3001 Leuven, Belgium, and [b]KU Leuven–Universtiy of Leuven, Department of Metallurgy and Materials Engineering, Kasteelpark Arenberg 44, B-3001 Leuven, Belgium
Correspondence e-mail: Luc.VanMeervelt@chem.kuleuven.be

The title compound, $C_4H_8O_4Te$, crystallized from a solution of Te^{4+} in ethylene glycol. The Te^{IV} atom is in a distorted seesaw coordination defined by four O atoms from two different ethyleneglycate ligands. The C atoms of the ethyleneglycate ligands are disorderd over two positions, with population parameters of 50.3 (6) and 49.7 (6)% indicating a statistical distribution. Due to the possibility to transform the primitive monoclinic unit cell into a metrically orthorhombic C unit cell, the data are twinned and were refined with the twin law $\bar{1}00/0\bar{1}0/101$ with the relative scale factor refining to 1.82 (4)% for the minor component.

Related literature

For the use of Te^{4+} ethylene glycol solutions in electro-deposition of Te and Te compounds, see: Nguyen *et al.* (2012); Wu *et al.* (2013). For crystal structures of related four-coordinate Te^{4+} complexes with oxo ligands, see: Day & Holmes (1981); Yosef *et al.* (2007); Annan *et al.* (1992); Fleischer & Schollmeyer (2001); Betz *et al.* (2008); Lindqvist (1967).
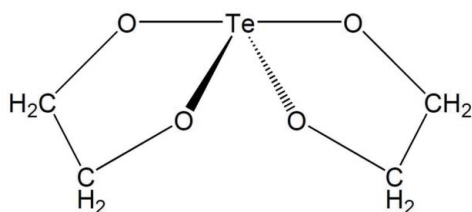
Experimental

Crystal data

$C_4H_8O_4Te$
$M_r = 247.70$
Monoclinic, $P2_1/n$
$a = 6.4838$ (7) Å
$b = 6.4978$ (8) Å
$c = 15.3633$ (15) Å
$\beta = 102.168$ (11)°
$V = 632.72$ (12) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 4.64$ mm^{-1}
$T = 100$ K
$0.20 \times 0.10 \times 0.08$ mm

Data collection

Agilent SuperNova (Single source at offset, Eos) diffractometer
Absorption correction: numerical (*CrysAlis PRO*; Agilent, 2012)
$T_{min} = 0.540$, $T_{max} = 0.710$
2841 measured reflections
1501 independent reflections
1291 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.079$
$S = 1.05$
1501 reflections
96 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 3.93$ e Å^{-3}
$\Delta\rho_{min} = -0.97$ e Å^{-3}

Table 1
Selected bond lengths (Å).

Te1—O4	1.940 (3)	Te1—O5	2.027 (3)
Te1—O8	1.942 (3)	Te1—O1	2.032 (4)

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* (Sheldrick, 2008); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010).

References

Agilent (2012). *CrysAlis PRO*. Agilent Technologies, Yarnton, England.
Annan, T. A., Ozarowski, A., Tian, Z. & Tuck, D. G. (1992). *J. Chem. Soc. Dalton Trans.* pp. 2931–2938.
Betz, R., Stapel, M., Pfister, M., Roessner, F. W., Reichvilser, M. M. & Klufers, P. (2008). *Z. Anorg. Allg. Chem.* **634**, 2391–2396.
Day, R. O. & Holmes, R. R. (1981). *Inorg. Chem.* **20**, 3071–3075.
Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
Fleischer, H. & Schollmeyer, D. (2001). *Inorg. Chem.* **40**, 324–328.
Lindqvist, O. (1967). *Acta Chem. Scand.* **21**, 1473–1483.
Nguyen, H. P., Wu, M., Su, J., Vullers, R. J. M., Vereecken, P. M. & Fransaer, J. (2012). *Electrochim. Acta*, **68**, 9–17.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
Wu, M., Nguyen, H. P., Vullers, R. J. M., Vereecken, P. M., Binnemans, K. & Fransaer, J. (2013). *J. Electrochem. Soc.* **160**, D196–D201.
Yosef, S., Brodsky, M., Sredni, B., Albeck, A. & Albeck, M. (2007). *ChemMedChem*, **2**, 1601–1606.

supplementary materials

Bis(ethyleneglycolato-*κ²O,O′*)tellurium(IV)

Neil R. Brooks, Minxian Wu, Luc Van Meervelt, Koen Binnemans and Jan Fransaer

Comment

Solutions of Te^{4+} in ethylene glycol are interesting for the purposes of electrodeposition of Te and Te compounds (Nguyen *et al.*, 2012, Wu *et al.*, 2013). During such electrodeposition experiments we have noticed a large number of colourless crystals on the walls of the glass flask. We report here the crystal structure of these crystals.

The title compound, $Te(C_2H_4O_2)_2$, crystallized with one molecule in the asymmetric unit. The Te atom is in a distorted seesaw coordination defined by four O atoms from two different ethyleneglycato ligands (Fig. 1, Table 1). The carbon atoms of the ethyleneglycato ligands are disorderd over two positions, with the major component comprising 50.3 (6)% of the total. The angle between the best planes through atoms Te1—O1—C2—C3—O4 and Te1—O5—C6—C7—O8 is 85.4 (3)°. For the second position the angle between the best planes through Te1—O1—C7′—C6′—O8 and Te1—O4—C2′—C3′—O5 is 85.6 (3)°.

Crystal structures of seven similar four coordinate Te^{4+} complexes with oxo ligands have previously been reported by Day & Holmes (1981), Yosef *et al.* (2007), Annan *et al.* (1992), Fleischer & Schollmeyer (2001), Betz *et al.* (2008) and Lindqvist (1967), all of which have a distorted seesaw geometry of the Te centre. The geometries or the Te—O bond lengths in these structures do not differ remarkably from those in the title compound. The most closely related compound is the octamethyl derivative bis(1,1,2,2-tetramethyleneglycolato-*O,O′*)tellurium(IV) (Day & Holmes, 1981), in which the Te—O bond lengths are, within error, the same as in the title compound. The O—Te—O bond angles that define the seesaw are 105.59° and 153.53° compared to 94.83 (14)° and 159.65 (13)° in the title compound. In tetrakis(methoxy)-tellurium(IV) (Betz *et al.*, 2008), the O—Te—O bond angles are 89.99° and 171.42°.

Experimental

Equal volumes of a 1 *M* solution of $TeCl_4$ in ethylene glycol and a 4 *M* solution of $AgNO_3$ in ethylene glycol were mixed together, ensuring that $AgNO_3$ was in a slight excess. The resulting precipitate of AgCl was removed by filtration. Excess Ag^+ ions in the remaining solution were removed by electrodeposition on a Pt working electrode at a constant potential of -0.1 V *versus* Ag and the solution used in electrodeposition experiments (Wu *et al.*, 2013). When the solution was left to stand for a period of two months, a large number of colourless crystals of the title compound slowly appeared on the walls of the glass flask.

Refinement

The H atoms were included using a riding model, with C—H distances of 0.99 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. The disorder of the ligands was modelled with two atomic positions for each C atom and the relative occupancy of these two positions refined as a least-squares parameter. The population parameters of 50.3 (6) and 49.7 (6)% indicate a statistical distribution. Constraints were applied to the displacement parameters of the C atoms to keep the respective atoms from the two disorder components the same. The unit-cell dimensions are such that it is possible to transform the primitive

monoclinic unit cell into a metrically orthorhombic *C* unit cell, however the high R_{int} for the higher Laue class shows that the Laue symmetry of the data is unequivocally monoclinic. There is however slight twinning due to these metrics and the data were refined with the twin law $\bar{1}\ 0\ 0\ /\ 0\ \bar{1}\ 0\ /\ 1\ 0\ 1$ with the relative scale factor refining to 1.82 (4)% for the minor component.. At the end of the refinement there was a residual difference electron density peak of 3.93 e Å^{-3}, which was located close to the Te atom. Although careful consideration was given to the unit cell determination and the absorption correction, this peak could not be eliminated.

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO* (Agilent, 2012); data reduction: *CrysAlis PRO* (Agilent, 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* (Sheldrick, 2008); software used to prepare material for publication: OLEX2 (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010).

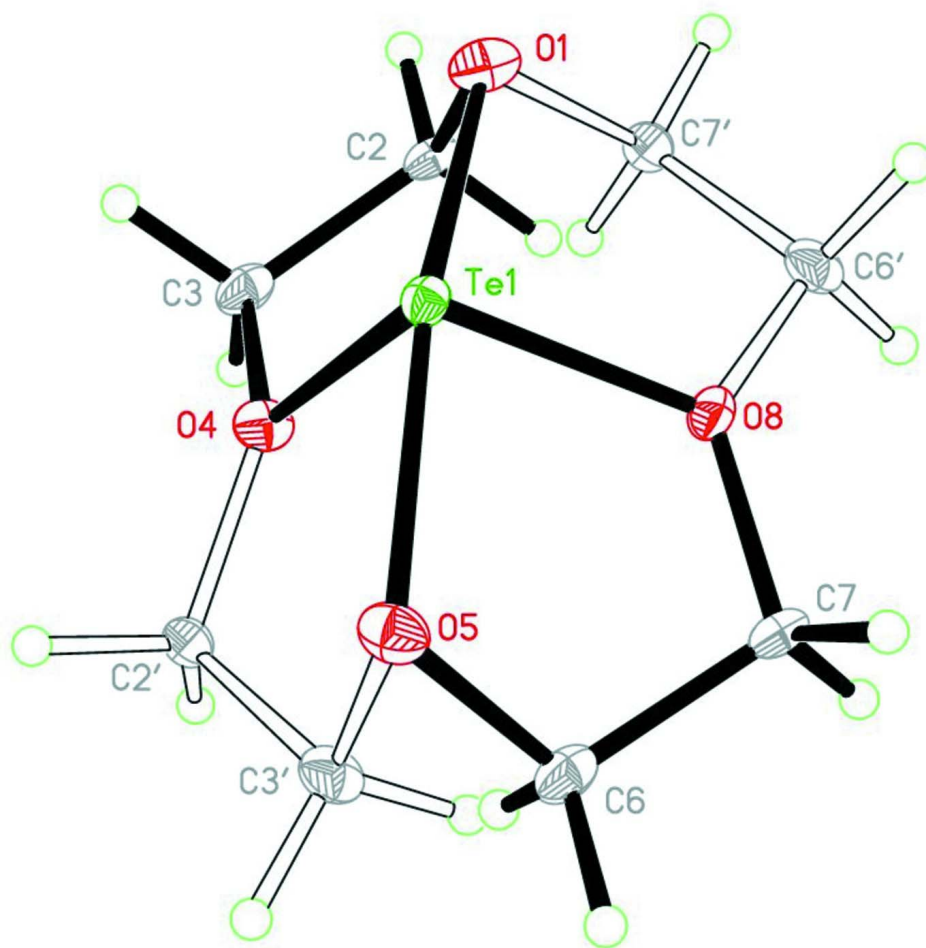**Figure 1**

View of the asymmetric unit of Te(OCH$_2$CH$_2$O)$_2$ showing the molecular structure and the disorder of the ligands. Solid bonds belong to one disorder component and open bonds to the other component (Te—O bonds are common to both components). For emphasis and to avoid confusion it should be noted that the (OCH$_2$CH$_2$O) ligands are disordered and the ligand is not macrocyclic.

Bis(ethyleneglycolato-$\kappa^2 O,O'$)tellurium(IV)

Crystal data

$C_4H_8O_4Te$	$F(000) = 464$
$M_r = 247.70$	$D_x = 2.600$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 6.4838$ (7) Å	Cell parameters from 1495 reflections
$b = 6.4978$ (8) Å	$\theta = 2.8–29.0°$
$c = 15.3633$ (15) Å	$\mu = 4.64$ mm^{-1}
$\beta = 102.168$ (11)°	$T = 100$ K
$V = 632.72$ (12) Å^3	Block, colourless
$Z = 4$	$0.20 \times 0.10 \times 0.08$ mm

Data collection

Agilent SuperNova (Single source at offset, Eos) diffractometer	$T_{min} = 0.540$, $T_{max} = 0.710$
	2841 measured reflections
	1501 independent reflections
Radiation source: SuperNova (Mo) X-ray Source	1291 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.031$
Mirror monochromator	$\theta_{max} = 28.9°$, $\theta_{min} = 3.1°$
Detector resolution: 15.9631 pixels mm^{-1}	$h = -8 \rightarrow 7$
ω scans	$k = -8 \rightarrow 8$
Absorption correction: numerical (*CrysAlis PRO*; Agilent, 2012)	$l = -20 \rightarrow 18$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.034$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.079$	
$S = 1.05$	H-atom parameters constrained
1501 reflections	$w = 1/[\sigma^2(F_o^2) + (0.034P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
96 parameters	
0 restraints	$(\Delta/\sigma)_{max} = 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 3.93$ e Å^{-3}
	$\Delta\rho_{min} = -0.97$ 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)
Te1	0.47847 (4)	0.12922 (6)	0.711596 (17)	0.00914 (12)	
O1	0.2476 (5)	0.3458 (6)	0.6857 (2)	0.0152 (8)	
C2	0.1002 (15)	0.2905 (17)	0.6034 (6)	0.0116 (15)	0.499 (4)
H2A	0.1543	0.3389	0.5513	0.014*	0.499 (4)

H2B	−0.0385	0.3561	0.6016	0.014*	0.499 (4)
C3	0.0770 (15)	0.0595 (17)	0.6007 (6)	0.0132 (15)	0.499 (4)
H3A	−0.0115	0.0147	0.6424	0.016*	0.499 (4)
H3B	0.0080	0.0146	0.5399	0.016*	0.499 (4)
C2′	0.3599 (14)	−0.2166 (17)	0.5966 (6)	0.0116 (15)	0.501 (4)
H2′A	0.2925	−0.2444	0.5336	0.014*	0.501 (4)
H2′B	0.3273	−0.3326	0.6332	0.014*	0.501 (4)
C3′	0.5943 (15)	−0.1981 (18)	0.6060 (6)	0.0132 (15)	0.501 (4)
H3′A	0.6578	−0.3368	0.6067	0.016*	0.501 (4)
H3′B	0.6259	−0.1211	0.5548	0.016*	0.501 (4)
O4	0.2792 (5)	−0.0285 (6)	0.6251 (2)	0.0114 (7)	
O5	0.6852 (6)	−0.0891 (6)	0.6898 (2)	0.0139 (8)	
C6	0.7477 (16)	−0.0408 (18)	0.6043 (6)	0.0136 (15)	0.499 (4)
H6A	0.6412	−0.0956	0.5538	0.016*	0.499 (4)
H6B	0.8853	−0.1056	0.6030	0.016*	0.499 (4)
C7	0.7640 (15)	0.1902 (16)	0.5960 (6)	0.0101 (14)	0.499 (4)
H7A	0.8976	0.2397	0.6340	0.012*	0.499 (4)
H7B	0.7635	0.2280	0.5336	0.012*	0.499 (4)
C6′	0.4899 (15)	0.4712 (18)	0.5992 (6)	0.0136 (15)	0.501 (4)
H6′A	0.5056	0.5106	0.5387	0.016*	0.501 (4)
H6′B	0.5554	0.5796	0.6413	0.016*	0.501 (4)
C7′	0.2586 (15)	0.4511 (17)	0.6011 (6)	0.0101 (14)	0.501 (4)
H7′A	0.1920	0.5887	0.5987	0.012*	0.501 (4)
H7′B	0.1843	0.3694	0.5496	0.012*	0.501 (4)
O8	0.5902 (5)	0.2822 (6)	0.6232 (2)	0.0122 (7)	

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Te1	0.00895 (18)	0.0106 (2)	0.00793 (17)	−0.00114 (12)	0.00182 (13)	−0.00028 (12)
O1	0.0134 (18)	0.020 (2)	0.0122 (16)	0.0023 (16)	0.0029 (14)	−0.0021 (16)
C2	0.010 (3)	0.015 (4)	0.009 (3)	0.003 (3)	0.001 (3)	0.000 (3)
C3	0.011 (3)	0.015 (4)	0.012 (3)	0.003 (3)	0.000 (3)	−0.002 (3)
C2′	0.010 (3)	0.015 (4)	0.009 (3)	0.003 (3)	0.001 (3)	0.000 (3)
C3′	0.011 (3)	0.015 (4)	0.012 (3)	0.003 (3)	0.000 (3)	−0.002 (3)
O4	0.0091 (16)	0.014 (2)	0.0099 (15)	−0.0011 (15)	−0.0006 (13)	−0.0037 (15)
O5	0.0164 (18)	0.012 (2)	0.0121 (16)	0.0035 (16)	0.0014 (14)	0.0012 (15)
C6	0.010 (3)	0.016 (4)	0.016 (3)	−0.006 (3)	0.005 (3)	−0.006 (3)
C7	0.009 (3)	0.009 (4)	0.013 (3)	0.001 (3)	0.005 (3)	0.002 (3)
C6′	0.010 (3)	0.016 (4)	0.016 (3)	−0.006 (3)	0.005 (3)	−0.006 (3)
C7′	0.009 (3)	0.009 (4)	0.013 (3)	0.001 (3)	0.005 (3)	0.002 (3)
O8	0.0137 (17)	0.011 (2)	0.0143 (16)	−0.0001 (16)	0.0077 (14)	0.0024 (16)

Geometric parameters (Å, º)

Te1—O4	1.940 (3)	C3′—O5	1.478 (10)
Te1—O8	1.942 (3)	C3′—H3′A	0.9900
Te1—O5	2.027 (3)	C3′—H3′B	0.9900
Te1—O1	2.032 (4)	O5—C6	1.487 (10)
O1—C2	1.460 (10)	C6—C7	1.512 (14)

O1—C7′	1.484 (10)	C6—H6A	0.9900
C2—C3	1.509 (15)	C6—H6B	0.9900
C2—H2A	0.9900	C7—O8	1.414 (9)
C2—H2B	0.9900	C7—H7A	0.9900
C3—O4	1.407 (10)	C7—H7B	0.9900
C3—H3A	0.9900	C6′—O8	1.402 (12)
C3—H3B	0.9900	C6′—C7′	1.511 (12)
C2′—O4	1.434 (10)	C6′—H6′A	0.9900
C2′—C3′	1.500 (12)	C6′—H6′B	0.9900
C2′—H2′A	0.9900	C7′—H7′A	0.9900
C2′—H2′B	0.9900	C7′—H7′B	0.9900
O4—Te1—O8	94.84 (14)	C3—O4—C2′	130.1 (6)
O4—Te1—O5	83.41 (15)	C3—O4—Te1	114.6 (5)
O8—Te1—O5	83.41 (15)	C2′—O4—Te1	115.2 (4)
O4—Te1—O1	82.81 (15)	C3′—O5—C6	57.7 (6)
O8—Te1—O1	82.92 (14)	C3′—O5—Te1	108.8 (4)
O5—Te1—O1	159.66 (13)	C6—O5—Te1	108.1 (5)
C2—O1—C7′	59.9 (5)	O5—C6—C7	108.7 (7)
C2—O1—Te1	108.7 (5)	O5—C6—H6A	110.0
C7′—O1—Te1	108.7 (4)	C7—C6—H6A	110.0
O1—C2—C3	108.2 (8)	O5—C6—H6B	110.0
O1—C2—H2A	110.1	C7—C6—H6B	110.0
C3—C2—H2A	110.1	H6A—C6—H6B	108.3
O1—C2—H2B	110.1	O8—C7—C6	108.7 (7)
C3—C2—H2B	110.1	O8—C7—H7A	109.9
H2A—C2—H2B	108.4	C6—C7—H7A	109.9
O4—C3—C2	108.4 (8)	O8—C7—H7B	109.9
O4—C3—H3A	110.0	C6—C7—H7B	109.9
C2—C3—H3A	110.0	H7A—C7—H7B	108.3
O4—C3—H3B	110.0	O8—C6′—C7′	109.1 (8)
C2—C3—H3B	110.0	O8—C6′—H6′A	109.9
H3A—C3—H3B	108.4	C7′—C6′—H6′A	109.9
O4—C2′—C3′	109.2 (8)	O8—C6′—H6′B	109.9
O4—C2′—H2′A	109.8	C7′—C6′—H6′B	109.9
C3′—C2′—H2′A	109.8	H6′A—C6′—H6′B	108.3
O4—C2′—H2′B	109.8	O1—C7′—C6′	106.7 (7)
C3′—C2′—H2′B	109.8	O1—C7′—H7′A	110.4
H2′A—C2′—H2′B	108.3	C6′—C7′—H7′A	110.4
O5—C3′—C2′	109.5 (7)	O1—C7′—H7′B	110.4
O5—C3′—H3′A	109.8	C6′—C7′—H7′B	110.4
C2′—C3′—H3′A	109.8	H7′A—C7′—H7′B	108.6
O5—C3′—H3′B	109.8	C6′—O8—C7	130.3 (6)
C2′—C3′—H3′B	109.8	C6′—O8—Te1	114.3 (4)
H3′A—C3′—H3′B	108.2	C7—O8—Te1	115.4 (5)
O4—Te1—O1—C2	−16.1 (5)	O4—Te1—O5—C3′	17.1 (5)
O8—Te1—O1—C2	79.7 (5)	O8—Te1—O5—C3′	−78.5 (5)
O5—Te1—O1—C2	31.6 (7)	O1—Te1—O5—C3′	−30.5 (7)

O4—Te1—O1—C7′	−79.7 (5)	O4—Te1—O5—C6	78.3 (5)
O8—Te1—O1—C7′	16.1 (5)	O8—Te1—O5—C6	−17.4 (5)
O5—Te1—O1—C7′	−32.0 (7)	O1—Te1—O5—C6	30.7 (7)
C7′—O1—C2—C3	138.3 (9)	C3′—O5—C6—C7	138.1 (10)
Te1—O1—C2—C3	37.0 (7)	Te1—O5—C6—C7	36.9 (8)
O1—C2—C3—O4	−45.4 (9)	O5—C6—C7—O8	−43.0 (10)
O4—C2′—C3′—O5	40.2 (10)	C2—O1—C7′—C6′	−138.4 (10)
C2—C3—O4—C2′	−147.6 (8)	Te1—O1—C7′—C6′	−37.2 (8)
C2—C3—O4—Te1	32.9 (8)	O8—C6′—C7′—O1	46.5 (9)
C3′—C2′—O4—C3	154.0 (8)	C7′—C6′—O8—C7	148.3 (8)
C3′—C2′—O4—Te1	−26.5 (8)	C7′—C6′—O8—Te1	−34.5 (8)
O8—Te1—O4—C3	−92.2 (5)	C6—C7—O8—C6′	−153.6 (8)
O5—Te1—O4—C3	−175.0 (5)	C6—C7—O8—Te1	29.2 (9)
O1—Te1—O4—C3	−9.9 (5)	O4—Te1—O8—C6′	92.7 (5)
O8—Te1—O4—C2′	88.2 (5)	O5—Te1—O8—C6′	175.5 (5)
O5—Te1—O4—C2′	5.5 (5)	O1—Te1—O8—C6′	10.6 (5)
O1—Te1—O4—C2′	170.5 (5)	O4—Te1—O8—C7	−89.7 (5)
C2′—C3′—O5—C6	−135.5 (11)	O5—Te1—O8—C7	−6.9 (5)
C2′—C3′—O5—Te1	−35.5 (9)	O1—Te1—O8—C7	−171.8 (5)