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Bis[(dimethyl-λ^4-sulfanylidene)oxonium] hexabromidotellurate(IV) dimethyl sulfoxide disolvate

Martin D. Rudd,[a]* Gregory Kokke[a] and Sergey V. Lindeman[b]

[a]University of Wisconsin–Fox Valley, Menasha, Wisconsin 54952, USA, and [b]Marquette University, Milwaukee, Wisconsin 53201, USA
Correspondence e-mail: martin.rudd@uwc.edu

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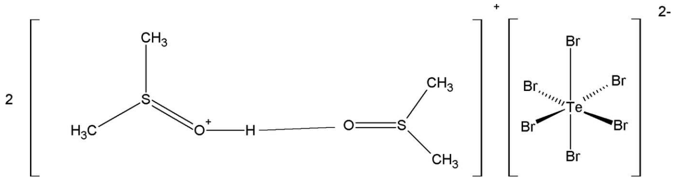
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(S–C) = 0.005 Å; R factor = 0.021; wR factor = 0.057; data-to-parameter ratio = 13.3.

The structure of the title salt, $2C_2H_7OS^+\cdot Br_6Te^{2-}\cdot 2C_2H_6OS$, displays O—H⋯O hydrogen bonding between one protonated dimethyl sulfoxide molecule and a neighboring dimethyl sulfoxide molecule, and an octahedral geometry for the Te atom; the latter is situated on a center of inversion.

Related literature

For the structure of the related compound $[(dmso\text{-}H)_2][TeCl_6]$, see: Laitinen *et al.* (2002); Viossat *et al.* (1981). For related literature, see Abriel (1987); Abriel & du Bois (1989); Borgias *et al.* (1985); Jaswal *et al.* (1990); Keefer *et al.* (1988).



Experimental

Crystal data

$2C_2H_7OS^+\cdot Br_6Te^{2-}\cdot 2C_2H_6OS$
M_r = 921.59
Triclinic, $P\bar{1}$
a = 8.0087 (2) Å
b = 9.2428 (2) Å
c = 10.5249 (3) Å
α = 66.280 (1)°
β = 70.732 (1)°
γ = 66.340 (1)°
V = 639.98 (3) Å^3
Z = 1
Cu $K\alpha$ radiation
μ = 23.30 mm^{-1}
T = 100 (2) K
0.23 × 0.20 × 0.16 mm

Data collection

Bruker APEX2 CCD detector diffractometer
Absorption correction: numerical [based on real shape of the crystal; absorption correction followed by the application of *SADABS* (Bruker, 2005)]
T_{min} = 0.075, T_{max} = 0.118
5232 measured reflections
2112 independent reflections
2112 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.056
S = 1.15
2112 reflections
159 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 1.02 e Å^{-3}
$\Delta\rho_{min}$ = −0.68 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O2—H2*O*⋯O1	0.83 (7)	1.62 (8)	2.448 (4)	175 (7)

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *XS* in *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* (Bruker, 1998); software used to prepare material for publication: *XCIF* in *SHELXTL* (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2262).

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Bis[(dimethyl-λ^4-sulfanylidene)oxonium] hexabromidotellurate(IV) dimethyl sulfoxide disolvate

Martin D. Rudd, Gregory Kokke and Sergey V. Lindeman

S1. Comment

The structure of (I) consists of two units of two H^+ hydrogen bonded dimethylsulfoxide molecules, Fig. 1, and a centrosymmetric hexabromotellurate(IV) anion, Fig. 2. At 2.448 (4) Å, the O1···O2 distance is relatively short, and is consistent with the presence of a moderately strong hydrogen bond (Keefer *et al.*, 1988). The IR spectrum reveals peaks typical for the $[(dmso)_2H]^+$ cation with a strong band at 731 cm^{-1}. This is in line with similar samples in which the same cation has been analyzed (Jaswal *et al.*, 1990). A closely related tellurium complex, $[(dmso)_2H]_2[TeCl_6]$ has been structurally reported at room temperature (Viossat *et al.*, 1981) and at low temperature (Laitinen *et al.*, 2002). The cation in the latter experiment shows a O1···O2 distance of 2.435 (3) Å and the authors describe this as a "relatively strong hydrogen bond".

The hexabromotellurate(IV) anion in (I) shows an approximately octahedral geometry as expected. A review of some related structures shows that there are packing factors that slightly distort the geometry. One example where $[TeBr_6]^{2-}$ shows deviations away from the regular octahedral geometry indicates that there is a 0.024Å difference between the longest and shortest bond Te—Br bond lengths (Borgias *et al.*, 1985). In that report, the Te atom is located in a general position. In other literature, the Te is located at a center of inversion and displays a larger angular deviation from 90° [87.56 (3) - 92.44 (3)°] (Abriel & du Bois, 1989) which is greater than those reported here [less than 0.9° away from 90°]. A review of structural data for MX_6E^{2-} compounds (M = Se, Te and X = Cl, Br, I) was published to provide an explanation of the stereochemistry of the lone pair electrons (Abriel, 1987).

The unit cell shows, Fig. 3, the pairs of hydrogen bonded dmso molecule and dmso-H ions and anions, Table 1.

S2. Experimental

Compound (I) was prepared by the slow cooling to room temperature of a hot solution (333 K) of tellurium dioxide (0.30 g, 0.19 mmol) dissolved in hydrobromic acid (1 mL) to which dimethylsulfoxide (5 mL) had been added. After 2 weeks, a crop of orange crystals formed although they are prone to solvent loss and decomposition. Analysis found: C 10.57; H 2.91; $C_8H_{26}Br_6O_4S_4Te$ requires: C 10.42, H 2.84. The IR spectrum showed strong bands at 3392, 1056, 731 cm^{-1}.

S3. Refinement

The maximum and minimum electron density peaks of 1.01 and -0.68 e $Å^{-3}$, respectively, are located 0.88 and 1.53 Å, respectively, from the Te atom. Hydrogen atoms positions were refined freely with C-H = 0.83 (7) - 1.03 (6) Å.

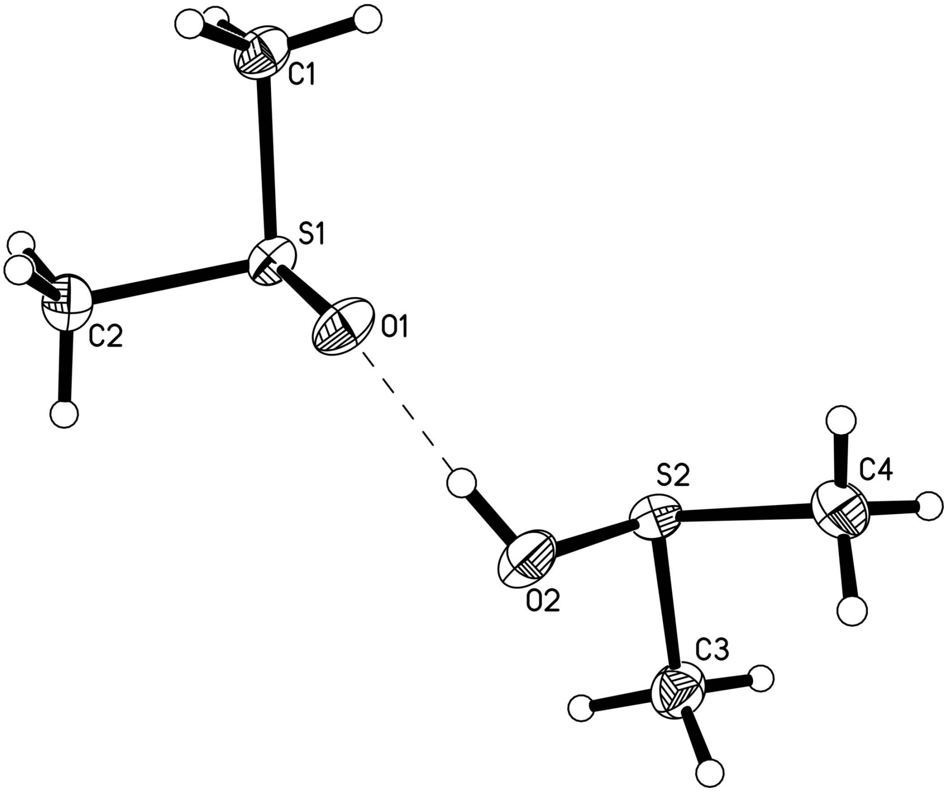

Figure 1

Numbering Scheme for $[(DMSO)_2H]^+$ (the hydrogen bond is shown as a dashed line). Displacement ellipsoids are shown at the 50% level.

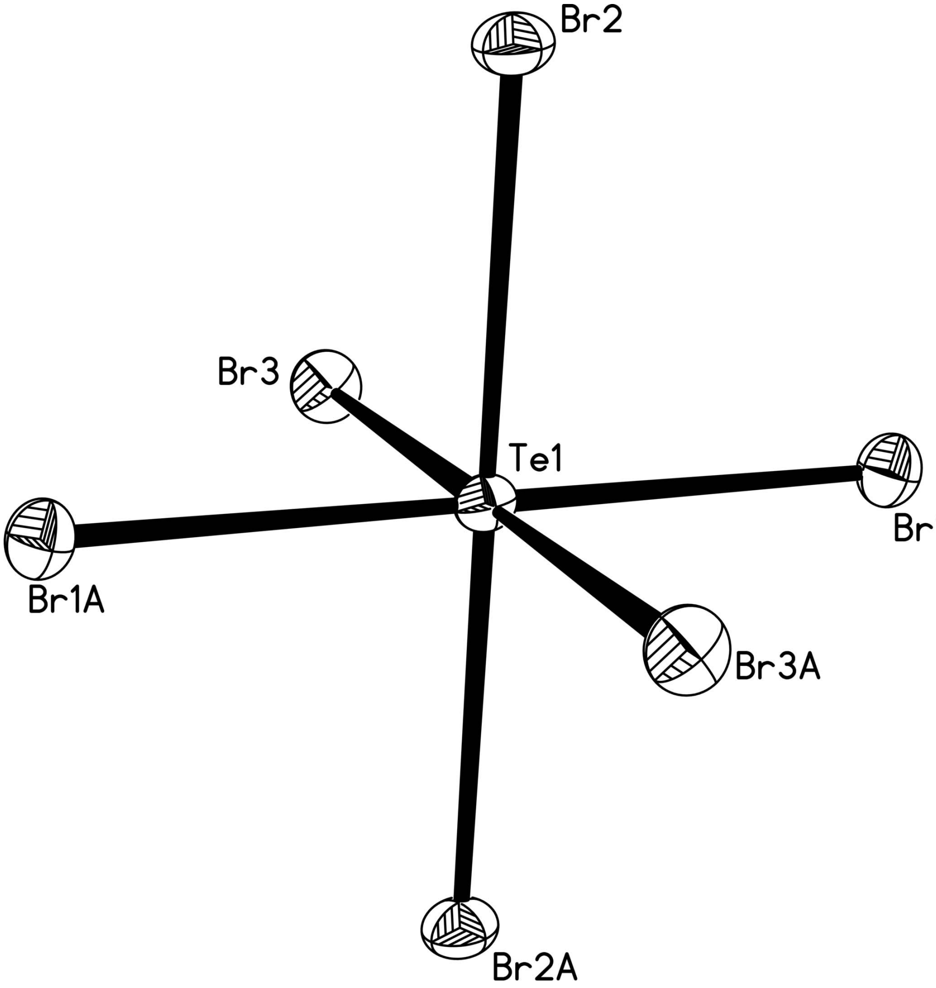

Figure 2

Numbering Scheme for $[TeBr_6]^{2-}$. Displacement ellipsoids are shown at the 50% level.

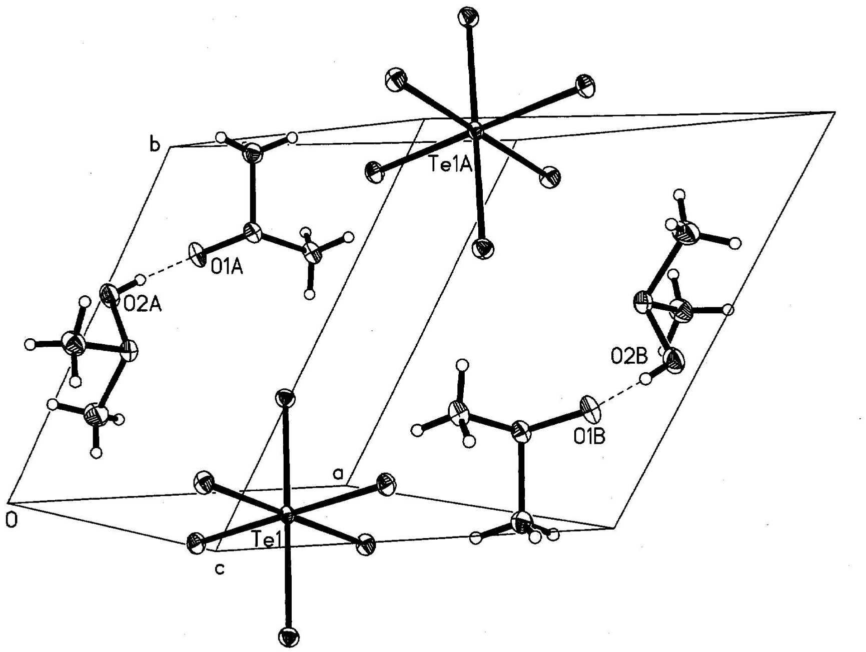

Figure 3

Hydrogen-bond formation and projection of the unit cell content of [(DMSO)$_2$H]$_2$[TeBr$_6$]. Symmetry operators: Te1A [x, y+1, z]; O1A and O2A [1-x, 1-y, -z]; O1B and O2B [x, y, z+1]

Bis[(dimethyl-λ^4-sulfanylidene)oxonium] hexabromidotellurate(IV) dimethyl sulfoxide disolvate

Crystal data

2C$_2$H$_7$OS$^+$·Br$_6$Te^{2-}·2C$_2$H$_6$OS
M_r = 921.59
Triclinic, $P\bar{1}$
Hall symbol: -P 1
a = 8.0087 (2) Å
b = 9.2428 (2) Å
c = 10.5249 (3) Å
α = 66.280 (1)°
β = 70.732 (1)°
γ = 66.340 (1)°
V = 639.98 (3) Å^3

Z = 1
F(000) = 432
D_x = 2.391 Mg m^{-3}
Melting point: 343 K
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 4736 reflections
θ = 5–66°
μ = 23.30 mm^{-1}
T = 100 K
Block, orange
0.23 × 0.20 × 0.16 mm

Data collection

Bruker APEX2 CCD detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: numerical [based on real shape of the crystal; absorption correction followed by the application of *SADABS* (Bruker, 2005)]

$T_{\min}$ = 0.075, $T_{\max}$ = 0.118
5232 measured reflections
2112 independent reflections
2112 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
$\theta_{\max}$ = 66.8°, $\theta_{\min}$ = 4.7°
$h = -8 \rightarrow 9$
$k = -9 \rightarrow 10$
$l = 0 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.021$
$wR(F^2) = 0.056$
$S = 1.15$
2112 reflections
159 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0268P)^2 + 1.1408P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.004$
$\Delta\rho_{\max} = 1.02$ e Å^{-3}
$\Delta\rho_{\min} = -0.68$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00507 (17)

Special details

Experimental. Analysis found: C 10.57; H 2.91; C~8~H~26~Br~6Õ~4~S~4~Te requires: C 10.42, H 2.84

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}
Te1	0.5000	0.0000	0.5000	0.01240 (12)
Br1	0.47203 (5)	0.30103 (4)	0.30479 (4)	0.01875 (12)
Br2	0.13998 (5)	0.11843 (5)	0.62389 (4)	0.01970 (12)
Br3	0.39388 (5)	−0.09389 (5)	0.33507 (4)	0.02004 (12)
S1	0.72250 (12)	0.24280 (11)	−0.11647 (9)	0.0176 (2)
O1	0.8060 (4)	0.2997 (3)	−0.0395 (3)	0.0229 (6)
C1	0.4776 (5)	0.3040 (5)	−0.0455 (4)	0.0213 (8)
H1A	0.459 (7)	0.246 (6)	0.054 (5)	0.030 (12)*
H1B	0.440 (7)	0.421 (7)	−0.066 (5)	0.032 (13)*
H1C	0.424 (6)	0.263 (6)	−0.088 (5)	0.026 (12)*
C2	0.7743 (6)	0.0237 (5)	−0.0301 (4)	0.0215 (8)
H2A	0.700 (7)	−0.016 (6)	−0.060 (5)	0.027 (11)*
H2B	0.741 (6)	0.002 (6)	0.070 (5)	0.027 (12)*
H2C	0.907 (7)	−0.020 (6)	−0.066 (5)	0.025 (11)*
S2	1.03170 (12)	0.54942 (12)	−0.30476 (10)	0.0219 (2)
O2	1.0726 (4)	0.4039 (4)	−0.1636 (3)	0.0256 (6)
H2O	0.985 (10)	0.364 (9)	−0.123 (8)	0.07 (2)*
C3	1.2525 (6)	0.5183 (6)	−0.4190 (5)	0.0266 (9)
H3A	1.340 (7)	0.519 (6)	−0.369 (5)	0.029 (12)*
H3B	1.245 (7)	0.614 (7)	−0.496 (6)	0.040 (14)*
H3C	1.284 (8)	0.410 (7)	−0.440 (6)	0.049 (15)*
C4	1.0161 (7)	0.7251 (6)	−0.2660 (6)	0.0321 (10)

H4A	1.015 (7)	0.822 (7)	−0.346 (6)	0.040 (14)*
H4B	0.913 (8)	0.747 (7)	−0.207 (6)	0.043 (15)*
H4C	1.125 (7)	0.695 (6)	−0.222 (5)	0.035 (13)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Te1	0.01221 (17)	0.01490 (18)	0.01250 (17)	−0.00478 (12)	−0.00205 (12)	−0.00641 (12)
Br1	0.0217 (2)	0.0180 (2)	0.0169 (2)	−0.00800 (15)	−0.00436 (15)	−0.00352 (15)
Br2	0.0143 (2)	0.0229 (2)	0.0215 (2)	−0.00556 (15)	0.00073 (15)	−0.01025 (16)
Br3	0.0237 (2)	0.0221 (2)	0.0211 (2)	−0.00733 (16)	−0.00826 (16)	−0.01003 (16)
S1	0.0187 (4)	0.0208 (4)	0.0158 (4)	−0.0096 (4)	−0.0020 (3)	−0.0059 (3)
O1	0.0228 (14)	0.0314 (15)	0.0234 (13)	−0.0161 (12)	−0.0018 (11)	−0.0118 (11)
C1	0.0215 (19)	0.026 (2)	0.022 (2)	−0.0115 (17)	−0.0024 (16)	−0.0095 (17)
C2	0.025 (2)	0.0208 (19)	0.021 (2)	−0.0087 (17)	−0.0077 (17)	−0.0047 (16)
S2	0.0167 (4)	0.0226 (5)	0.0279 (5)	−0.0070 (4)	−0.0058 (4)	−0.0075 (4)
O2	0.0243 (15)	0.0295 (15)	0.0261 (14)	−0.0158 (13)	−0.0052 (12)	−0.0042 (12)
C3	0.026 (2)	0.028 (2)	0.028 (2)	−0.0117 (18)	0.0002 (18)	−0.0107 (18)
C4	0.024 (2)	0.025 (2)	0.049 (3)	−0.0039 (18)	−0.002 (2)	−0.021 (2)

Geometric parameters (Å, º)

Te1—Br1^{i}	2.6865 (4)	C2—H2B	0.95 (5)
Te1—Br1	2.6865 (4)	C2—H2C	0.97 (5)
Te1—Br3	2.6956 (4)	S2—O2	1.576 (3)
Te1—Br3^{i}	2.6956 (4)	S2—C3	1.767 (4)
Te1—Br2^{i}	2.7103 (4)	S2—C4	1.776 (4)
Te1—Br2	2.7103 (4)	O2—H2O	0.83 (7)
S1—O1	1.537 (3)	C3—H3A	1.00 (5)
S1—C2	1.787 (4)	C3—H3B	0.92 (6)
S1—C1	1.791 (4)	C3—H3C	1.03 (6)
C1—H1A	0.96 (5)	C4—H4A	0.95 (6)
C1—H1B	0.95 (5)	C4—H4B	0.86 (6)
C1—H1C	0.97 (5)	C4—H4C	1.01 (5)
C2—H2A	0.98 (5)		

Br1^{i}—Te1—Br1	180.0	H1B—C1—H1C	117 (4)
Br1^{i}—Te1—Br3	89.604 (11)	S1—C2—H2A	107 (3)
Br1—Te1—Br3	90.395 (11)	S1—C2—H2B	110 (3)
Br1^{i}—Te1—Br3^{i}	90.397 (11)	H2A—C2—H2B	110 (4)
Br1—Te1—Br3^{i}	89.604 (11)	S1—C2—H2C	103 (3)
Br3—Te1—Br3^{i}	179.999 (1)	H2A—C2—H2C	112 (4)
Br1^{i}—Te1—Br2^{i}	89.151 (11)	H2B—C2—H2C	114 (4)
Br1—Te1—Br2^{i}	90.849 (11)	O2—S2—C3	102.38 (19)
Br3—Te1—Br2^{i}	89.485 (12)	O2—S2—C4	102.5 (2)
Br3^{i}—Te1—Br2^{i}	90.515 (11)	C3—S2—C4	100.2 (2)
Br1^{i}—Te1—Br2	90.848 (11)	S2—O2—H2O	112 (5)
Br1—Te1—Br2	89.151 (11)	S2—C3—H3A	106 (3)

Br3—Te1—Br2	90.515 (11)	S2—C3—H3B	107 (3)
Br3^{i}—Te1—Br2	89.485 (12)	H3A—C3—H3B	104 (4)
Br2^{i}—Te1—Br2	180.0	S2—C3—H3C	107 (3)
O1—S1—C2	103.95 (17)	H3A—C3—H3C	115 (4)
O1—S1—C1	104.47 (17)	H3B—C3—H3C	116 (5)
C2—S1—C1	98.62 (19)	S2—C4—H4A	114 (3)
S1—C1—H1A	108 (3)	S2—C4—H4B	107 (4)
S1—C1—H1B	106 (3)	H4A—C4—H4B	108 (5)
H1A—C1—H1B	113 (4)	S2—C4—H4C	108 (3)
S1—C1—H1C	106 (3)	H4A—C4—H4C	110 (4)
H1A—C1—H1C	108 (4)	H4B—C4—H4C	111 (5)

Symmetry code: (i) −*x*+1, −*y*, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*O*···O1	0.83 (7)	1.62 (8)	2.448 (4)	175 (7)