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Bis(tetramethylguanidinium) hexachloridotellurate(IV)

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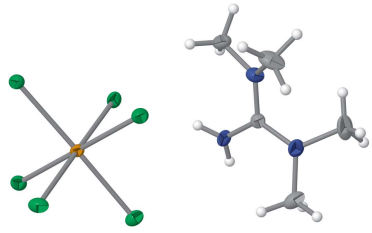
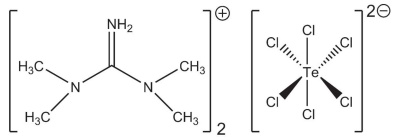
The title compound, $2C_5H_{14}N_3^+\cdot TeCl_6^{2-}$, is an easily accessible salt with a relatively low melting point. The asymmetric unit consists of a $Te_{0.25}Cl_{1.5}$ fragment and half a cation. Weak hydrogen bonds of the type C—H···Cl and N—H···Cl are present in the crystal structure.

3D view



Chemical scheme



Structure description

The asymmetric unit of the title salt consists of a $Te_{0.25}Cl_{1.5}$ unit (with the tellurium atom located on the 8*a* Wyckoff site of the space group *Fddd* with 222 symmetry) and of one half of the TMG cation (the C1 and N1 atoms are located on a twofold rotation axis, site 16*g*). The molecular structure is shown in Fig. 1.

The distances between the tellurium atom and the surrounding chlorine atoms of the $[TeCl_6]^{2-}$ anion are 2.5363 (4) and 2.5394 (3) Å, in agreement with published Te—Cl bond lengths (Allen *et al.*, 1987). The angle between the carbon atom C1 and the two nitrogen atoms N2 in the cation is 120.14 (6)°. The distance between N1 and C1 is 1.323 (2) Å, while that between C1 and N2 is 1.342 (1) Å, indicating that C1 is involved in a double bond. The bond lengths between N2 and atoms C2 and C3 are longer, 1.464 (2) and 1.459 (1) Å, respectively, as expected for N—C single bonds. Consistent with the *d* glide planes of the space group *Fddd*, the tetramethylguanidinium cations and the hexachloridotellurate(IV) anions are arranged in chains with an alternating orientation of the cations in the three unit-cell directions (see Fig. 2).

Weak hydrogen bonds (Table 1) are found between the NH_2 groups of the cation and the chlorine atoms of the anion with a shortest Cl···N distance of 3.3875 (8) Å.

The number of published X-ray structures of tetramethylguanidinium (TMG) metal salts is limited. The first publication is from the 1960s (Longhi & Drago, 1965). Different metal salts with the TMG cation have been published since then (Snaith *et al.*, 1970; Bujak *et al.*, 1999; Jones & Thonnessen, 2006; Bujak & Zaleski, 2007; Due-Hansen *et al.*,

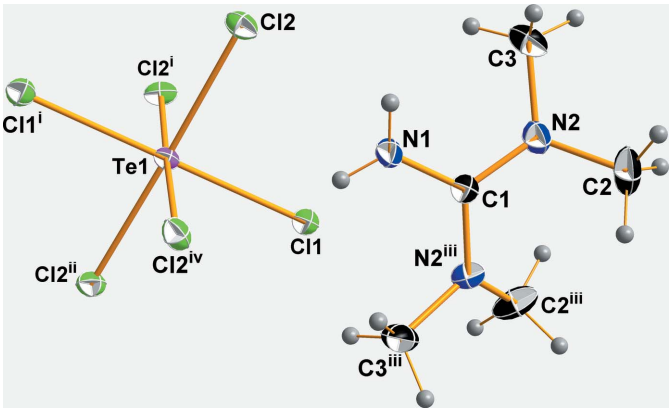


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level. Symmetry codes: (i) $-x+\frac{7}{4}, y, -z+\frac{3}{4}$; (ii) $-x+\frac{7}{4}, -y+\frac{3}{4}, z$; (iii) $-x+\frac{3}{4}, -y+\frac{3}{4}, z$; (iv) $x, -y+\frac{3}{4}, -z+\frac{3}{4}$.

2011; Ndiaye *et al.*, 2016*a*,*b*; Şendıl *et al.*, 2016). Tetramethylguanidinium salts find applications in the capture of SO_2 or the removal of sulfur-carrying organic materials (Berg *et al.*, 2013; Meng *et al.*, 2017). An example of the properties of ionic liquids with the $[TeCl_6]^{2-}$ anion was published recently (Shen *et al.*, 2018).

Synthesis and crystallization

N,*N*,*N*′,*N*′-tetramethylguanidinium chloride (0.5156 g, 0.0034 mol) and tellurium tetrachloride (0.458 g, 0.0017 mol) were dissolved in ethanol (10 ml). The yellow liquid was stirred at ambient temperature for one day. The reaction mixture was filtered and the solvent was removed with a rotary evaporator. A yellow solid was obtained in nearly stoichiometrical yields. The melting point was determined using DSC to be 134°C. A stoichiometric amount of the compound was dissolved in ethanol (approx. 10 mg per ml of ethanol) and crystals were grown through slow diffusion of diethyl ether into the solution.

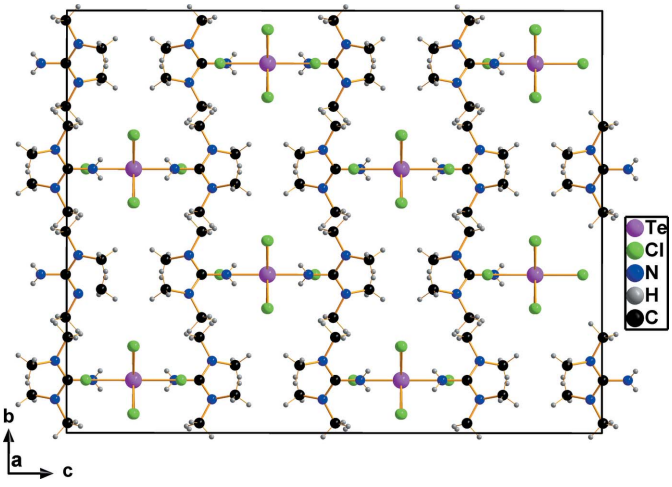


Figure 2
View along the *a* axis of the unit cell showing the arrangement of the ions of the title compound.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···Cl2^{ii}	0.83 (2)	2.57 (2)	3.3875 (8)	167 (1)
C2—H2B···Cl2^{v}	0.98	2.84	3.712 (1)	149
C3—H3B···Cl1^{v}	0.98	2.82	3.644 (1)	142

Symmetry codes: (ii) $x, -y+\frac{3}{4}, -z+\frac{3}{4}$; (v) $-x+1, -y+1, -z+1$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$2C_5H_{14}N_3^+\cdot Cl_6Te^{2-}$
M_r	572.68
Crystal system, space group	Orthorhombic, *Fddd*
Temperature (K)	123
a, b, c (Å)	7.3899 (5), 22.447 (2), 28.512 (2)
V (Å^3)	4729.6 (6)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	1.92
Crystal size (mm)	0.45 × 0.35 × 0.12
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2017)
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	47585, 2158, 2057
R_{int}	0.027
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.757
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.013, 0.034, 1.26
No. of reflections	2158
No. of parameters	62
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.39, −0.37

Computer programs: *APEX3* and *SAINT* (Bruker, 2017), *SHELXS2014* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *DIAMOND* (Brandenburg & Putz, 2014) and *publCIF* (Westrip, 2010).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The reflection 022 was omitted from the refinement because its intensity was affected by the beam stop.

Acknowledgements

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References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, S1–S19.

Berg, R. W., Harris, P., Riisager, A. & Fehrmann, R. (2013). *J. Phys. Chem. A*, **117**, 11364–11373.

Brandenburg, K. & Putz, H. (2014). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.

Bruker (2017). *APEX3*, *SADABS* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Bujak, M., Osadczuk, P. & Zaleski, J. (1999). *Acta Cryst.* C**55**, 1443–1447.

Bujak, M. & Zaleski, J. (2007). *Acta Cryst.* E**63**, m102–m104.

Due-Hansen, J., Ståhl, K., Boghosian, S., Riisager, A. & Fehrmann, R. (2011). *Polyhedron*, **30**, 785–789.

Jones, P. G. & Thonnessen, H. (2006). Private communication (refcode CEPXIF). CCDC, Cambridge, England.

Longhi, R. & Drago, R. S. (1965). *Inorg. Chem.* **4**, 11–14.

Meng, X., Wang, J., Jiang, H., Zhang, X., Liu, S. & Hu, Y. (2017). *J. Chem. Technol. Biotechnol.* **92**, 767–774.

Ndiaye, M., Samb, A., Diop, L. & Maris, T. (2016*a*). *Acta Cryst.* E**72**, 1–3.

Ndiaye, M., Samb, A., Diop, L. & Maris, T. (2016*b*). *Acta Cryst.* E**72**, 1047–1049.

Şendıl, K., Özgün, H. B. & Üstün, E. (2016). *J. Chem.* pp. 1–7.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

Shen, N.-N., Cai, M.-L., Song, Y., Wang, Z.-P., Huang, F.-Q., Li, J.-R. & Huang, X.-Y. (2018). *Inorg. Chem.* **57**, 5282–5291.

Snaith, R., Wade, K. & Wyatt, B. K. (1970). *J. Chem. Soc. A*, pp. 380.

Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

full crystallographic data

IUCrData (2018). **3**, x181488 [https://doi.org/10.1107/S2414314618014888]

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Crystal data

$2C_5H_{14}N_3^+\cdot Cl_6Te^{2-}$
M_r = 572.68
Orthorhombic, *Fddd*
a = 7.3899 (5) Å
b = 22.447 (2) Å
c = 28.512 (2) Å
V = 4729.6 (6) Å^3
Z = 8
F(000) = 2272
D_x = 1.609 Mg m^{-3}
Melting point: 407 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9942 reflections
θ = 2.3–32.5°
μ = 1.92 mm^{-1}
T = 123 K
Stick, yellow
0.45 × 0.35 × 0.12 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: microfocus sealed tube
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2017)
47585 measured reflections
2158 independent reflections
2057 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 32.6°, θ_{min} = 2.9°
h = −11→11
k = −34→33
l = −42→42

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.013
$wR(F^2)$ = 0.034
S = 1.26
2158 reflections
62 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0103P)^2 + 6.2116P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.000156 (17)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. All non-hydrogen atoms were refined anisotropically. The methyl H atoms were positioned with idealized geometry and refined isotropically with $U_{iso}(H) = 1.5\ U_{eq}(C)$ using a riding model. The positions of the hydrogen atoms of the NH_2 group of the guanidinium cation were taken from the electron density map and refined isotropically.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Te1	0.8750	0.3750	0.3750	0.01545 (4)
Cl1	0.8750	0.3750	0.46396 (2)	0.02182 (6)
Cl2	0.63226 (3)	0.45506 (2)	0.37388 (2)	0.02254 (5)
N1	0.3750	0.3750	0.45159 (4)	0.0222 (2)
H1	0.447 (2)	0.3533 (6)	0.4372 (5)	0.030 (4)*
C1	0.3750	0.3750	0.49798 (5)	0.0184 (2)
N2	0.3004 (1)	0.42051 (4)	0.52161 (3)	0.0241 (2)
C2	0.1980 (2)	0.41017 (6)	0.56481 (4)	0.0381 (3)
H2A	0.0721	0.4226	0.5602	0.057*
H2B	0.2517	0.4333	0.5904	0.057*
H2C	0.2017	0.3677	0.5727	0.057*
C3	0.2619 (2)	0.47675 (5)	0.49795 (4)	0.0307 (2)
H3A	0.3543	0.4842	0.4740	0.046*
H3B	0.2629	0.5092	0.5209	0.046*
H3C	0.1426	0.4746	0.4831	0.046*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Te1	0.01545 (5)	0.01243 (5)	0.01847 (6)	0	0.00	0.00
Cl1	0.0257 (1)	0.0190 (1)	0.0207 (1)	0.0020 (1)	0.00	0.00
Cl2	0.0214 (1)	0.01873 (9)	0.0274 (1)	0.00211 (7)	−0.00547 (8)	−0.00431 (7)
N1	0.0221 (5)	0.0269 (5)	0.0178 (5)	0.0099 (4)	0.00	0.00
C1	0.0161 (5)	0.0192 (5)	0.0200 (5)	−0.0008 (4)	0.00	0.00
N2	0.0272 (4)	0.0224 (4)	0.0226 (4)	0.0008 (3)	0.0022 (3)	−0.0052 (3)
C2	0.0439 (7)	0.0409 (6)	0.0296 (5)	−0.0080 (5)	0.0141 (5)	−0.0145 (5)
C3	0.0316 (5)	0.0213 (4)	0.0391 (6)	0.0060 (4)	−0.0049 (4)	−0.0062 (4)

Geometric parameters (Å, º)

Te1—Cl1	2.5363 (4)	C1—N2	1.342 (1)
Te1—Cl1^{i}	2.5364 (4)	N2—C3	1.459 (1)
Te1—Cl2^{ii}	2.5394 (3)	N2—C2	1.464 (2)
Te1—Cl2	2.5394 (3)	C2—H2A	0.9800
Te1—Cl2^{i}	2.5394 (3)	C2—H2B	0.9800
Te1—Cl2^{iii}	2.5394 (3)	C2—H2C	0.9800
N1—C1	1.323 (2)	C3—H3A	0.9800
N1—H1	0.83 (2)	C3—H3B	0.9800
C1—N2^{iv}	1.342 (1)	C3—H3C	0.9800
Cl1—Te1—Cl1^{i}	180.0	N1—C1—N2	120.14 (6)

Cl1—Te1—Cl2^{ii}	89.282 (5)	N2^{iv}—C1—N2	119.7 (1)
Cl1^{i}—Te1—Cl2^{ii}	90.718 (5)	C1—N2—C3	120.43 (9)
Cl1—Te1—Cl2	90.718 (5)	C1—N2—C2	120.93 (9)
Cl1^{i}—Te1—Cl2	89.282 (5)	C3—N2—C2	115.15 (9)
Cl2^{ii}—Te1—Cl2	90.12 (1)	N2—C2—H2A	109.5
Cl1—Te1—Cl2^{i}	89.283 (5)	N2—C2—H2B	109.5
Cl1^{i}—Te1—Cl2^{i}	90.717 (5)	H2A—C2—H2B	109.5
Cl2^{ii}—Te1—Cl2^{i}	178.57 (1)	N2—C2—H2C	109.5
Cl2—Te1—Cl2^{i}	89.90 (1)	H2A—C2—H2C	109.5
Cl1—Te1—Cl2^{iii}	90.717 (5)	H2B—C2—H2C	109.5
Cl1^{i}—Te1—Cl2^{iii}	89.283 (5)	N2—C3—H3A	109.5
Cl2^{ii}—Te1—Cl2^{iii}	89.90 (1)	N2—C3—H3B	109.5
Cl2—Te1—Cl2^{iii}	178.57 (1)	H3A—C3—H3B	109.5
Cl2^{i}—Te1—Cl2^{iii}	90.12 (1)	N2—C3—H3C	109.5
C1—N1—H1	120 (1)	H3A—C3—H3C	109.5
N1—C1—N2^{iv}	120.14 (6)	H3B—C3—H3C	109.5

Symmetry codes: (i) $-x+7/4, y, -z+3/4$; (ii) $x, -y+3/4, -z+3/4$; (iii) $-x+7/4, -y+3/4, z$; (iv) $-x+3/4, -y+3/4, z$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···Cl2^{ii}	0.83 (2)	2.57 (2)	3.3875 (8)	167 (1)
C2—H2*B*···Cl2^{v}	0.98	2.84	3.712 (1)	149
C3—H3*B*···Cl1^{v}	0.98	2.82	3.644 (1)	142

Symmetry codes: (ii) $x, -y+3/4, -z+3/4$; (v) $-x+1, -y+1, -z+1$.