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Bis(diisobutylammonium) tetrachloridobis[3-(trifluoromethyl)phenyl]stannate

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Keywords: stannate; ammonium cation; crystal structure; hydrogen bonding; salt.

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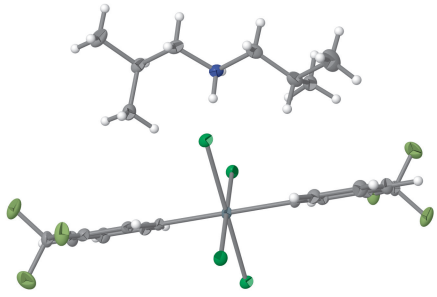
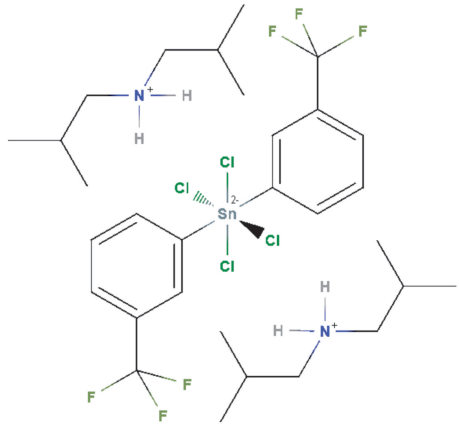
Structural data: full structural data are available from iucrdata.iucr.org

The asymmetric unit in the title salt, $(C_8H_{20}N)_2[SnCl_4(C_7H_4Cl_2F_3)_2]$, features a di-*iso*butylammonium cation in a general position and a diorganotin tetrachloride dianion, *i.e.* tetrachloridobis(3-trifuoromethylphenyl)stannate(IV), located on a centre of inversion; the Sn^{IV} atom is octahedrally coordinated. In the crystal, charge-assisted $N^+—H\cdots Cl$ hydrogen bonds along with $C—H\cdots F$ contacts occur within supramolecular layers that interdigitate along the *a*-axis direction.

3D view



Chemical scheme



Structure description

The title salt, bis(di-*iso*butylammonium) tetrachloridobis(3-trifluoromethylphenyl)stannate, was obtained as a by-product in a reaction of tris(3-trifluoromethylphenyl)tin chloride with acetic acid in the presence of di-*iso*butylamine. An interesting Sn—C cleavage occurred during this reaction.

The crystal comprises di-*iso*butylammonium cations and tetrachloridobis-(3-trifluoromethylphenyl)stannate(IV) anions, with the Sn^{IV} atom of the latter located on a centre of inversion, Fig. 1. The coordination geometry about the Sn^{IV} atom is based on an octahedron, Table 1. This observation resembles literature precedents, *e.g.* Teoh *et al.* (1992) and Hazell *et al.* (1998).

In the crystal, charge-assisted $N^+—H\cdots Cl$ hydrogen bonds along with $C—H\cdots F$ contacts link molecules into a supramolecular layer parallel to (011). As noted from Table 2, the Cl1 atom accepts two $N^+—H\cdots Cl$ hydrogen bonds, each of which is significantly shorter than the $N^+—H\cdots Cl$ hydrogen bond involving the Cl2 atom. This observation accounts for the disparity in the Sn—Cl bond lengths, Table 1. The supramolecular layers interdigitate along [100], Fig. 2.

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Table 1
Selected geometric parameters (Å, °).

Sn1—Cl1	2.5845 (4)	Sn1—C9	2.147 (2)
Sn1—Cl2	2.5719 (4)		
C9—Sn1—Cl1	89.36 (6)	Cl1—Sn1—Cl2	90.308 (14)
C9—Sn1—Cl2	90.11 (5)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*C*···Cl1^{i}	0.91	2.31	3.1877 (17)	161
N1—H1*D*···Cl1	0.91	2.44	3.1771 (17)	138
N1—H1*D*···Cl2	0.91	2.75	3.4094 (17)	130
C7—H7*A*···F1^{ii}	0.98	2.56	3.227 (3)	125

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

Synthesis and crystallization

The crystal was obtained as a by-product in an attempt to poduce tris(3-trifluoromethylphenyl)tin acetate in a reaction involving tris(3-trifluoromethylphenyl)tin chloride and acetic acid in the presence of di-isobutylamine. The anticipated tris(3-trifluoromethylphenyl)tin acetate was isolated as the major product along with a few smaller crystals of the title compound in the mother liquid, comprising a mixture of dichloromethane and hexane.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3.

Table 3
Experimental details.

Crystal data	
Chemical formula	$(C_8H_{20}N)_2[SnCl_4(C_7H_4Cl_2F_3)_2]$
M_r	811.19
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, b, c (Å)	12.2614 (1), 10.8318 (1), 14.6297 (1)
β (°)	108.523 (1)
V (Å^3)	1842.36 (3)
Z	2
Radiation type	Cu $K\alpha$
μ (mm^{-1})	8.64
Crystal size (mm)	0.1 × 0.07 × 0.03
Data collection	
Diffractometer	XtaLAB Synergy, Single source at home/near, HyPix
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2023)
T_{min}, T_{max}	0.293, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	18706, 3822, 3652
R_{int}	0.057
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.634
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.027, 0.075, 1.08
No. of reflections	3822
No. of parameters	200
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.66, −0.90

Computer programs: *CrysAlis PRO* (Rigaku OD, 2023), *SHELXT2018/2* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

Funding information

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References

Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.

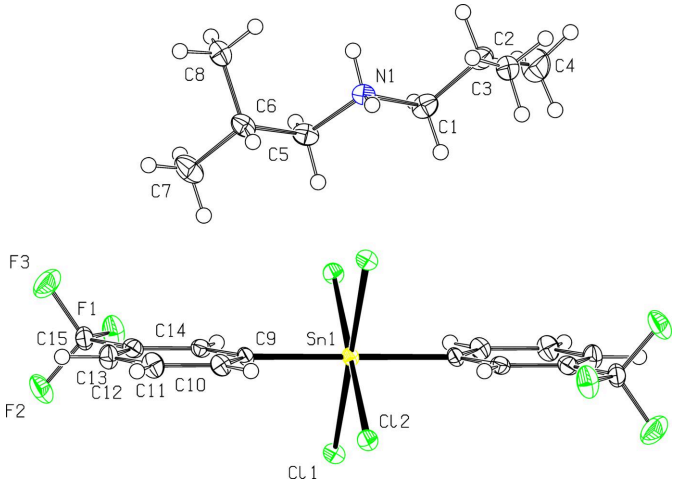

Figure 1
A view of the molecular structures of the di-*iso*butylammonium cation and the tetrachloridobis-(3-trifuoromethylphenyl)stannate(IV) anion, showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level. The unlabelled atoms for the anion are related by $1-x, 1-y, 1-z$.

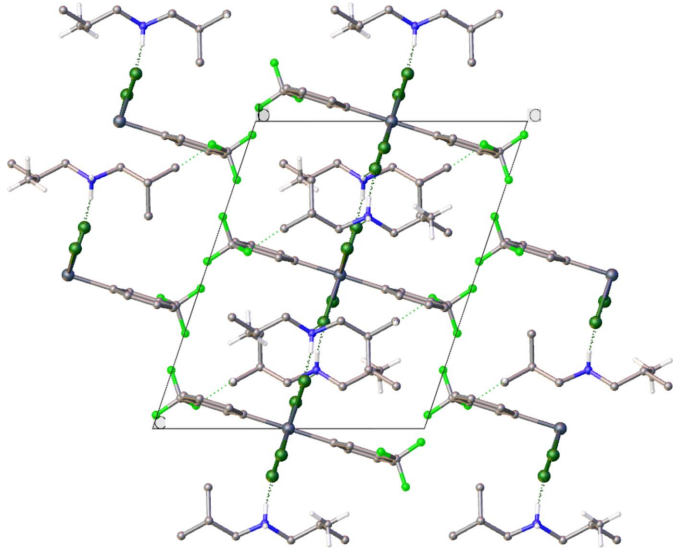

Figure 2
A packing diagram viewed along [010] with intermolecular hydrogen bonding shown as dashed lines.

Hazell, A., Khoo, L. E., Ouyang, J., Rausch, B. J. & Tavares, Z. M. (1998). *Acta Cryst.* C**54**, 728–732.
Rigaku OD (2023). *CrysAlis PRO*. Rigaku Oxford Diffraction, Yarnton, England.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Teoh, S. G., Teo, S. B., Yeap, G. Y. & Declercq, J. P. (1992). *Polyhedron*, **11**, 2351–2356.

full crystallographic data

IUCrData (2023). **8**, x230913 [https://doi.org/10.1107/S2414314623009136]

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Crystal data

$(C_8H_{20}N)_2[SnCl_4(C_7H_4Cl_2F_3)_2]$
M_r = 811.19
Monoclinic, $P2_1/c$
a = 12.2614 (1) Å
b = 10.8318 (1) Å
c = 14.6297 (1) Å
β = 108.523 (1)°
V = 1842.36 (3) Å^3
Z = 2
$F(000)$ = 828
D_x = 1.462 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 14287 reflections
θ = 3.8–77.8°
μ = 8.64 mm^{-1}
T = 100 K
Block, clear brown-orange
0.1 × 0.07 × 0.03 mm

Data collection

XtaLAB Synergy, Single source at home/near, HyPix diffractometer
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2023)
T_{min} = 0.293, T_{max} = 1.000
18706 measured reflections
3822 independent reflections
3652 reflections with $I > 2\sigma(I)$
R_{int} = 0.057
θ_{max} = 78.0°, θ_{min} = 3.8°
h = −15→15
k = −13→11
l = −18→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.075
S = 1.08
3822 reflections
200 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.041P)^2 + 0.7503P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −0.90 e Å^{-3}

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.35270 (19)	0.5478 (2)	0.13703 (15)	0.0239 (4)
H1A	0.358554	0.457309	0.130324	0.029*
H1B	0.326308	0.583776	0.071441	0.029*
C2	0.26367 (19)	0.5751 (2)	0.18694 (15)	0.0260 (5)
H2	0.295430	0.546221	0.255225	0.031*
C3	0.23739 (19)	0.7127 (2)	0.18845 (16)	0.0275 (5)
H3A	0.306651	0.756553	0.226965	0.041*
H3B	0.175849	0.724982	0.216927	0.041*
H3C	0.212799	0.744875	0.122425	0.041*
C4	0.1551 (3)	0.5004 (2)	0.1380 (3)	0.0411 (8)
H4A	0.121094	0.528525	0.071310	0.062*
H4B	0.099624	0.511991	0.172948	0.062*
H4C	0.174867	0.412669	0.138369	0.062*
C5	0.56054 (19)	0.55525 (19)	0.14964 (15)	0.0222 (4)
H5A	0.537390	0.577720	0.080518	0.027*
H5B	0.565458	0.464099	0.154047	0.027*
C6	0.67905 (19)	0.60917 (19)	0.20024 (15)	0.0218 (4)
H6	0.674857	0.700698	0.190829	0.026*
C7	0.7618 (2)	0.5571 (2)	0.15105 (19)	0.0328 (5)
H7A	0.765195	0.467094	0.157844	0.049*
H7B	0.838600	0.591970	0.181228	0.049*
H7C	0.734647	0.578953	0.082497	0.049*
C8	0.72259 (19)	0.5828 (2)	0.30827 (16)	0.0274 (5)
H8A	0.669735	0.619158	0.338881	0.041*
H8B	0.799278	0.618867	0.336388	0.041*
H8C	0.726737	0.493329	0.318920	0.041*
N1	0.46944 (15)	0.59824 (15)	0.19037 (12)	0.0177 (3)
H1C	0.466464	0.682178	0.188412	0.021*
H1D	0.488962	0.574815	0.253256	0.021*
C9	0.68457 (19)	0.50380 (16)	0.54647 (16)	0.0146 (4)
C10	0.74444 (18)	0.6150 (2)	0.56702 (15)	0.0178 (4)
H10	0.702813	0.690201	0.560520	0.021*
C11	0.86397 (19)	0.6180 (2)	0.59687 (16)	0.0220 (4)
H11	0.903295	0.694773	0.610510	0.026*
C12	0.9258 (2)	0.50866 (18)	0.60672 (18)	0.0215 (5)
H12	1.007476	0.509922	0.627119	0.026*
C13	0.86647 (18)	0.3971 (2)	0.58629 (14)	0.0180 (4)
C14	0.74712 (18)	0.39424 (19)	0.55628 (14)	0.0157 (4)
H14	0.707913	0.317433	0.542374	0.019*
C15	0.93450 (17)	0.2801 (2)	0.59848 (15)	0.0205 (4)
Cl1	0.50101 (4)	0.38477 (4)	0.34558 (3)	0.01643 (11)
Cl2	0.49391 (4)	0.70843 (4)	0.41432 (3)	0.01725 (11)
F1	0.86867 (11)	0.17902 (12)	0.57535 (11)	0.0291 (3)
F2	1.00453 (12)	0.27762 (13)	0.54416 (10)	0.0306 (3)
F3	1.00321 (13)	0.26451 (14)	0.69011 (10)	0.0364 (3)

Sn1	0.500000	0.500000	0.500000	0.01258 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0260 (11)	0.0198 (11)	0.0220 (9)	−0.0067 (9)	0.0021 (8)	−0.0019 (8)
C2	0.0229 (10)	0.0294 (11)	0.0233 (10)	−0.0069 (9)	0.0038 (8)	0.0065 (9)
C3	0.0221 (11)	0.0338 (13)	0.0268 (10)	0.0007 (9)	0.0080 (8)	0.0042 (9)
C4	0.0291 (15)	0.0419 (19)	0.0480 (18)	−0.0161 (10)	0.0061 (13)	0.0051 (10)
C5	0.0301 (11)	0.0173 (10)	0.0218 (9)	0.0001 (8)	0.0118 (8)	−0.0026 (8)
C6	0.0270 (10)	0.0160 (9)	0.0269 (10)	0.0026 (8)	0.0151 (8)	0.0001 (8)
C7	0.0383 (13)	0.0285 (13)	0.0404 (13)	0.0049 (10)	0.0249 (11)	−0.0024 (10)
C8	0.0212 (10)	0.0331 (12)	0.0300 (11)	−0.0014 (9)	0.0112 (8)	0.0022 (10)
N1	0.0220 (8)	0.0135 (8)	0.0181 (7)	−0.0022 (6)	0.0071 (6)	−0.0008 (6)
C9	0.0117 (10)	0.0175 (11)	0.0161 (10)	−0.0006 (6)	0.0063 (8)	−0.0003 (6)
C10	0.0190 (10)	0.0152 (10)	0.0204 (9)	0.0002 (8)	0.0081 (7)	−0.0026 (8)
C11	0.0199 (10)	0.0188 (11)	0.0275 (10)	−0.0063 (8)	0.0077 (8)	−0.0041 (8)
C12	0.0155 (11)	0.0232 (12)	0.0254 (11)	−0.0015 (7)	0.0057 (9)	0.0001 (7)
C13	0.0189 (9)	0.0194 (10)	0.0176 (9)	0.0016 (8)	0.0086 (7)	0.0013 (8)
C14	0.0170 (9)	0.0158 (10)	0.0155 (8)	0.0007 (7)	0.0070 (7)	0.0017 (7)
C15	0.0136 (9)	0.0234 (10)	0.0250 (9)	0.0015 (8)	0.0068 (7)	0.0033 (8)
Cl1	0.0205 (2)	0.0118 (2)	0.0188 (2)	−0.00014 (15)	0.00893 (16)	−0.00113 (15)
Cl2	0.0188 (2)	0.0129 (2)	0.0212 (2)	0.00085 (15)	0.00785 (16)	0.00294 (16)
F1	0.0222 (6)	0.0172 (6)	0.0500 (8)	0.0021 (5)	0.0143 (6)	0.0042 (6)
F2	0.0271 (7)	0.0289 (7)	0.0442 (8)	0.0067 (5)	0.0235 (6)	0.0043 (6)
F3	0.0351 (8)	0.0389 (8)	0.0284 (6)	0.0168 (6)	0.0005 (6)	0.0061 (6)
Sn1	0.01231 (11)	0.01048 (12)	0.01599 (11)	0.00097 (5)	0.00599 (8)	0.00084 (5)

Geometric parameters (Å, º)

C1—C2	1.523 (3)	C11—C12	1.389 (3)
C1—N1	1.499 (3)	C12—C13	1.393 (3)
C2—C3	1.527 (3)	C13—C14	1.388 (3)
C2—C4	1.528 (3)	C13—C15	1.497 (3)
C5—C6	1.522 (3)	C15—F1	1.338 (3)
C5—N1	1.497 (3)	C15—F2	1.343 (2)
C6—C7	1.526 (3)	C15—F3	1.348 (2)
C6—C8	1.526 (3)	Sn1—Cl1	2.5845 (4)
C9—C10	1.393 (3)	Sn1—Cl2	2.5719 (4)
C9—C14	1.396 (3)	Sn1—C9	2.147 (2)
C10—C11	1.390 (3)		
N1—C1—C2	113.03 (17)	F1—C15—F2	106.35 (17)
C1—C2—C3	112.52 (18)	F1—C15—F3	106.55 (17)
C1—C2—C4	108.9 (2)	F2—C15—C13	112.55 (17)
C3—C2—C4	111.5 (2)	F2—C15—F3	105.72 (16)
N1—C5—C6	113.87 (16)	F3—C15—C13	111.92 (17)
C5—C6—C7	107.69 (18)	C9—Sn1—C9^{i}	180.0

C5—C6—C8	113.43 (18)	C9^{i}—Sn1—Cl1^{i}	89.36 (6)
C7—C6—C8	110.67 (19)	C9—Sn1—Cl1	89.36 (6)
C5—N1—C1	112.90 (16)	C9—Sn1—Cl1^{i}	90.64 (6)
C10—C9—C14	118.6 (2)	C9^{i}—Sn1—Cl1	90.64 (6)
C10—C9—Sn1	121.02 (14)	C9—Sn1—Cl2^{i}	89.89 (5)
C14—C9—Sn1	120.40 (14)	C9^{i}—Sn1—Cl2^{i}	90.11 (5)
C11—C10—C9	121.3 (2)	C9^{i}—Sn1—Cl2	89.89 (5)
C12—C11—C10	119.9 (2)	C9—Sn1—Cl2	90.11 (5)
C11—C12—C13	119.1 (2)	Cl1^{i}—Sn1—Cl1	180.0
C12—C13—C15	118.4 (2)	Cl1—Sn1—Cl2	90.308 (14)
C14—C13—C12	120.9 (2)	Cl2^{i}—Sn1—Cl1^{i}	90.308 (14)
C14—C13—C15	120.68 (19)	Cl2^{i}—Sn1—Cl1	89.692 (14)
C13—C14—C9	120.20 (19)	Cl2—Sn1—Cl1^{i}	89.693 (14)
F1—C15—C13	113.22 (17)	Cl2^{i}—Sn1—Cl2	180.00 (3)
C2—C1—N1—C5	171.26 (17)	C12—C13—C14—C9	−0.3 (3)
C6—C5—N1—C1	177.79 (17)	C12—C13—C15—F1	−178.98 (19)
N1—C1—C2—C3	66.3 (2)	C12—C13—C15—F2	−58.3 (3)
N1—C1—C2—C4	−169.55 (19)	C12—C13—C15—F3	60.6 (3)
N1—C5—C6—C7	179.98 (17)	C14—C9—C10—C11	−0.1 (3)
N1—C5—C6—C8	57.2 (2)	C14—C13—C15—F1	1.7 (3)
C9—C10—C11—C12	−0.1 (3)	C14—C13—C15—F2	122.3 (2)
C10—C9—C14—C13	0.2 (3)	C14—C13—C15—F3	−118.8 (2)
C10—C11—C12—C13	0.0 (4)	C15—C13—C14—C9	179.09 (19)
C11—C12—C13—C14	0.1 (4)	Sn1—C9—C10—C11	−179.81 (16)
C11—C12—C13—C15	−179.2 (2)	Sn1—C9—C14—C13	179.96 (15)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*C*···Cl1^{ii}	0.91	2.31	3.1877 (17)	161
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C7—H7*A*···F1^{iii}	0.98	2.56	3.227 (3)	125

Symmetry codes: (ii) −*x*+1, *y*+1/2, −*z*+1/2; (iii) *x*, −*y*+1/2, *z*−1/2.