Table 2. *Selected geometric parameters (Å, °)*

P1—O4	1.582 (3)	P3—O8	1.593 (3)
P1—O5	1.609 (3)	P3—O11	1.599 (4)
P1—O6	1.475 (3)	P3—O12	1.484 (3)
P1—O7	1.484 (3)	P3—O13	1.475 (4)
P2—O5	1.585 (3)	P4—O11	1.606 (4)
P2—O8	1.595 (3)	P4—O14	1.584 (3)
P2—O9	1.483 (4)	P4—O15	1.473 (4)
P2—O10	1.478 (4)	P4—O16	1.480 (3)
O4—P1—O5	102.4 (2)	O11—P3—O12	108.9 (2)
O4—P1—O6	105.4 (2)	O11—P3—O13	107.8 (2)
O4—P1—O7	111.2 (2)	O12—P3—O13	119.8 (2)
O5—P1—O6	111.2 (2)	O11—P4—O14	101.1 (2)
O5—P1—O7	105.7 (2)	O11—P4—O15	111.7 (2)
O6—P1—O7	119.6 (2)	O11—P4—O16	107.8 (2)
O5—P2—O8	99.1 (2)	O14—P4—O15	105.4 (2)
O5—P2—O9	108.4 (2)	O14—P4—O16	110.8 (2)
O5—P2—O10	110.5 (2)	O15—P4—O16	118.7 (2)
O8—P2—O9	109.5 (2)	P1—O4—C10	121.1 (3)
O8—P2—O10	110.5 (2)	P1—O5—P2	130.7 (2)
O9—P2—O10	117.3 (2)	P2—O8—P3	128.4 (2)
O8—P3—O11	100.3 (2)	P3—O11—P4	129.2 (2)
O8—P3—O12	109.7 (2)	P4—O14—C20	122.2 (3)
O8—P3—O13	108.5 (2)		
P1—O4—C10—C9	165.9 (3)	P3—O11—P4—O14	−159.4 (3)
P1—O5—P2—O8	74.7 (3)	P4—O11—P3—O8	105.4 (3)
P2—O5—P1—O4	−135.0 (3)	P4—O14—C20—C19	157.8 (3)
P2—O8—P3—O11	−124.8 (3)	O5—P1—O4—C10	−67.3 (4)
P3—O8—P2—O5	−164.3 (3)	O11—P4—O14—C20	−65.3 (4)

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1993). Program(s) used to solve structure: *TEXSAN*; *SAPI*91 (Fan, 1991). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *TEXSAN*; *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN FINISH*.

We thank Dr Motoo Shiro (Rigaku X-ray Research Laboratory) for his assistance in the preparation of this manuscript.

References

Altona, C. & Sundaralingam, M. (1972). *J. Am. Chem. Soc.* **94**, 8205–8212.

Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2A, pp. 72–98. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Fan, H.-F. (1991). *SAPI*91. *Structure Analysis Programs with Intelligent Control*. Rigaku Corporation, Tokyo, Japan.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Lüthje, J. (1989). *Klin. Wochenschr.* **67**, 317–327.

Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation (1993). *TEXSAN. Single-Crystal Structure Analysis Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Nakajima, H., Tomioka, I., Kitabatake, S., Dombou, M. & Tomita, K. (1989). *Agric. Biol. Chem.* **53**, 615–623.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.

Trimethylammonium Trichlorostannate(II)

JOAN HALFPENNY

Department of Chemistry and Physics, Nottingham Trent University, Clifton Lane, Nottingham NG11 8NS, England

Abstract

The $NHMe_3^+$ and $SnCl_3^-$ ions both have approximate threefold symmetry and lie on a mirror plane. The anion is pyramidal with Sn—Cl 2.515 (4) and 2.500 (3) Å and Cl—Sn—Cl 90.6 (2) and 89.1 (1)°. The pseudo-threefold axes of the ions are superimposed with close $Cl\cdots H$ contacts of 2.61 (7) and 2.71 (6) Å.

Comment

The structure of the title compound, (I), is ionic with $SnCl_3^-$ and $NHMe_3^+$ lying on a mirror plane. The molecular dimensions (Table 2) show that the trimethylammonium ion has the usual tetrahedral shape and that the trichlorostannate ion is pyramidal. Both ions possess non-crystallographic threefold symmetry, and an unusual feature of the structure is the coincidence of their threefold axes (Fig. 1). The axial hydrogen of the trimethylammonium ion is aligned in the direction of the Cl atoms (Fig. 2).

(I)
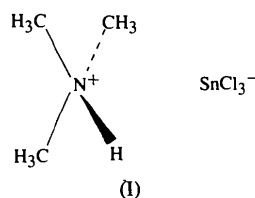
The $Cl\cdots H$ distances (Table 3) are significantly less than the sum of the van der Waals distances, indicating weak bond formation. A search of the Cambridge Structural Database (CSD) (Allen *et al.*, 1991) revealed 11 other organometallic structures (CSD codenames ENCOSN, PECOTB20, PPECOT20, BITXUX, DOWCIJ, DUSWOD, GEHTUI, KAHWAR, SIGMEA, SIGNIF and VOGRAK) involving the $SnCl_3^-$ ion. In
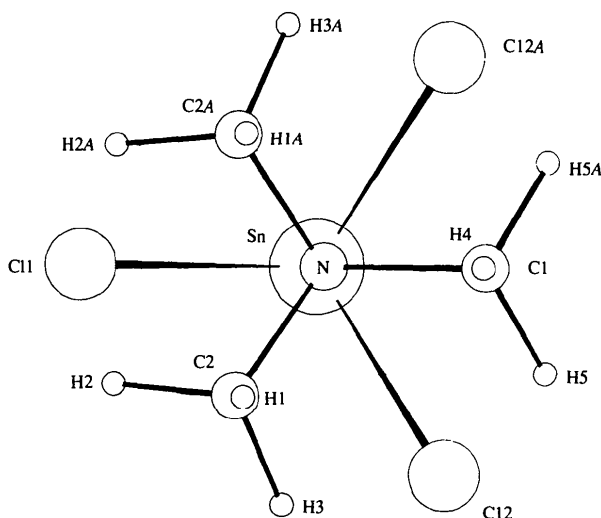
Fig. 1. Projection of the structure along the N–Sn direction showing superposition of the ions. H6 is wholly obscured by N. Atoms bearing the suffix A are related to those without this suffix by the symmetry operation $-x, y, z$. The mirror plane passes through Cl1, Sn, N, H6, Cl and H4.
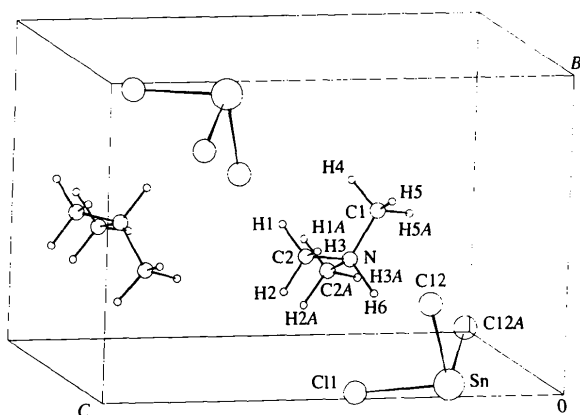

Fig. 2. Projection of the structure onto (100) showing the unit cell with half the contents omitted for clarity.

all of these structures except one, the mean Sn—Cl distance was significantly less and the Cl—Sn—Cl angle was significantly greater than in the present structure, the mean values over the ten structures being Sn—Cl = 2.47 Å and Cl—Sn—Cl = 93.5°. The one exception was GEHTUI, *tert*-butylammonium trichlorotin (Veith, Jarczyk & Huch, 1988): although there is no coincidence of the threefold axes of the ions as found in the present structure, in both structures the formation of Cl···H interionic bonds is coupled with lengthening of the Sn—Cl distances due to electron withdrawal. *tert*-Butylammonium trichlorotin has one extremely short Cl···H(ammonium) distance (2.29 Å) and a further short contact of 2.42 Å between this H atom and Cl from a different anion. The Sn—Cl bonds are long (2.578, 2.527, 2.521 Å). In the present structure the attraction of

the three Cl atoms to the axial H6 may contribute to the reduction of Cl—Sn—Cl angles; other slightly longer Cl···H approaches (Table 3) may also be significant. It is interesting that the Cl—Sn—Cl angles in the present structure are all similar whereas in *tert*-butylammonium trichlorotin (where unsymmetrical Cl···H interactions are present) the Cl—Sn—Cl angles vary between 88.2 and 93.5°.

Experimental

Crystal data

[NH(CH₃)₃][SnCl₃]	Mo $K\alpha$ radiation
$M_r = 285.17$	$\lambda = 0.71073$ Å
Orthorhombic	Cell parameters from 15
$Cmc2_1$	reflections
$a = 9.53\,(1)$ Å	$\theta = 5-25°$
$b = 8.31\,(1)$ Å	$\mu = 3.11$ mm^{-1}
$c = 12.39\,(1)$ Å	$T = 286$ K
$V = 981\,(3)$ Å³	Thin plate, tabular on (010)
$Z = 4$	$0.56 \times 0.44 \times 0.039$ mm
$D_x = 1.930$ Mg m^{-3}	Pale yellow
$D_m = 1.921$ Mg m^{-3}	

Data collection

Stoe Stadi-2 two-circle	$R_{int} = 0.017$
diffractometer	$\theta_{max} = 30.0°$
Variable ω scans	$h = 0 \to 11$
Absorption correction:	$k = 0 \to 10$
by integration from	$l = 0 \to 16$
crystal shape (*SHELX*76;	1 standard reflection per
Sheldrick, 1976)	layer monitored every
$T_{min} = 0.330$, $T_{max} =$	20 reflections
0.886	intensity decay: $\leq 5\%$
583 measured reflections	(corrected by interpo-
580 independent reflections	lation)
552 observed reflections	
$[I > 3\sigma(I)]$	

Refinement

Refinement on F	Unit weights applied
$R = 0.031$	$(\Delta/\sigma)_{max} < 0.001$
$wR = 0.031$	$\Delta\rho_{max} = 0.65$ e Å^{-3}
$S = 0.906$	$\Delta\rho_{min} = -0.45$ e Å^{-3}
552 reflections	Extinction correction: none
50 parameters	Atomic scattering factors
H-atom treatment: see below	from *SHELX*76

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* ($\mathring{A}^2$)

U_{iso} for N and C; $U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for Sn and Cl atoms.

	x	y	z	U_{iso}/U_{eq}
Sn	0	0.0390 (1)	0.2500†	0.0596 (5)
Cl1	0	0.0193 (5)	0.4525 (3)	0.077 (4)
Cl2	0.1840 (3)	0.2532 (3)	0.2572 (5)	0.085 (2)

N	0	0.565 (1)	−0.027 (1)	0.052 (3)
C2	0.117 (2)	0.578 (2)	0.043 (1)	0.12 (1)
C1	0	0.411 (3)	−0.082 (2)	0.11 (1)

† Coordinate fixed to define origin.

Table 2. *Selected bond lengths (Å) and angles (°)*

Sn—Cl1	2.515 (4)	N—C1	1.45 (2)
Sn—Cl2	2.500 (3)	N—C2	1.42 (2)
Cl1—Sn—Cl2	90.6 (2)	C1—N—C2	111 (1)
Cl2—Sn—Cl2ⁱ	89.1 (1)	C2—N—C2ⁱ	104 (2)

Symmetry code: (i) −x, y, z.

Table 3. *Contact distances (Å)*

Cl1ⁱ···H6ⁱⁱ	2.61 (5)	Cl1ⁱ···H4ⁱⁱⁱ	2.78 (5)
Cl2ⁱ···H6ⁱⁱ	2.71 (6)	Cl2···H1ⁱⁱ	2.78 (6)

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

Intensity measurements were made with a two-circle diffractometer employing equi-inclination Weissenberg geometry. The angle θ' is the projection of θ onto the equatorial plane. The crystal was set to rotate around the a axis. A separate standard reflection was chosen for each layer. During structure refinement, interlayer scale factors were refined and thereafter fixed. The structure was solved by the use of Patterson and difference syntheses. During refinement H atoms were included at calculated positions with individually refined U_{iso}. Anisotropic displacement parameters were allowed for Sn and Cl only, with isotropic refinement of N and C.

The polarity of the structure was established by competitive refinement. The chosen polarity had a significantly lower conventional R value (0.0314 *versus* 0.0365), smaller residuals in the final ΔF synthesis and lower uncertainties in the refined parameters.

Data reduction: *DATR* (Small, 1977). Program(s) used to solve structure: *SHELX76* (Sheldrick, 1976), Patterson. Program(s) used to refine structure: *SHELX76*. Molecular graphics: *SNOOPI* (Davies, 1983).

I acknowledge the use of the Cambridge Structural Database (Allen *et al.*, 1991).

References

Allen, F. H., Davies, J. E., Galloy, J. J., Johnson, O., Kennard, O., Macrae, C. F., Mitchell, E. M., Mitchell, G. F., Smith, J. M. & Watson, D. G. (1991). *J. Chem. Inf. Comput. Sci.* **31**, 187–204.
Davies, K. (1983). *SNOOPI. Program for Drawing Crystal and Molecular Diagrams.* University of Oxford, England.
Dillon, K. B., Halfpenny, J. & Marshall, A. (1983). *J. Chem. Soc. Dalton Trans.* pp. 1091–1094.
Dillon, K. B., Halfpenny, J. & Marshall, A. (1985). *J. Chem. Soc. Dalton Trans.* pp. 1399–1403.
Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination.* University of Cambridge England.
Small, R. W. H. (1977). *DATR. Program for Reducing Raw Stadi-2 Data to Intensities.* University of Lancaster, England.
Veith, M., Jarczyk, H. M. & Huch, V. (1988). *Chem. Ber.* **121**, 347–355.

The Role of C—H···O Bonds in Stabilizing 3-Furancarboxylic Acid and its Complexes with Calcium and Strontium

BEATA PALUCHOWSKA,[a] JAN K. MAURIN[a] AND JANUSZ LECIEJEWICZ[b]

[a]*Institute of Atomic Energy, 05-400 Otwock-Świerk, Poland,* and [b]*Institute of Nuclear Chemistry and Technology, Dorodna 16, 03-195 Warsaw, Poland*

Abstract

The crystal structures of 3-furancarboxylic acid, $C_5H_4O_3$, and both its calcium and strontium complexes, *catena*-poly[diaquacalcium-μ-(3-furancarboxylato-$O,O':O$)-μ-aqua 3-furancarboxylate], $[Ca(C_5H_3O_3)-(H_2O)_3](C_5H_3O_3)$, and *catena*-poly[diaquastrontium-μ-(3-furancarboxylato-$O,O':O$)-μ-aqua 3-furancarboxylate], $[Sr(C_5H_3O_3)(H_2O)_3](C_5H_3O_3)$, respectively, are presented. The differences and similarities in the hydrogen-bonding schemes of the title acid and 2-furancarboxylic acid are discussed, and the importance of C—H···O hydrogen bonds in the crystal packing of 3-furancarboxylic acid and its strontium and calcium complexes is highlighted.

Comment

The determination of the crystal structure of 3-furancarboxylic acid, (I), has been performed as an introductory step in the study of coordination compounds in which it acts as a ligand or an anion.

(I)

(II) M = Ca
(III) M = Sr
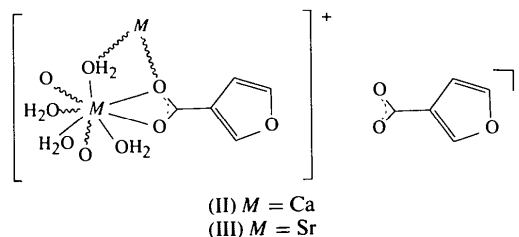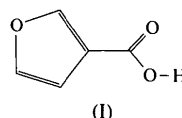
The crystals of 3-furancarboxylic acid are composed of centrosymmetric hydrogen-bonded dimers, forming approximately planar layers of molecules parallel to the (10$\bar{4}$) plane (Fig. 1). The two O3—H3···O2 hydrogen bonds between the carboxy O atoms, which are responsible for the formation of the dimer, have a