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The First Example of a Discrete Di(carboxylato)triorganostannate: Tetramethylammonium Triphenylbis(trifluoroacetato)stannate

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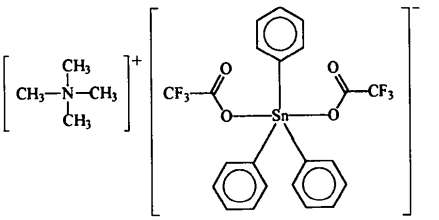
Abstract

Tetramethylammonium triphenylbis(trifluoroacetato-*O*)-stannate crystallizes as non-interacting tetrahedral $[(CH_3)_4N]^+$ cations and *trans*-trigonal bipyramidal $[Sn(C_2F_3O_2)_2(C_6H_5)_3]^-$ anions. The anion is the first example of a discrete triorganostannate covalently bonded to two unidentate monocarboxylato anions.

Comment

As a result of their preference for forming carboxyl bridges that link adjacent molecules into five-coordinate polymeric chains (Ng, Chen & Kumar Das, 1988), triphenyltin alkanoates, with rare exceptions (Ng, 1995), do not yield complexes with Lewis bases. The appearance of Lewis acidity in triphenyltin trifluoroacetate (Ford & Sams, 1971), which forms a hydrate that is hydrogen bonded to 1,10-phenanthroline (Ng, Kumar Das & Kennard, 1996), is unambiguously proven in tetramethylammonium triphenylbis(trifluoroacetato)-stannate, (I). The Lewis acidity of (I) is comparable to that of triphenyltin chloride as it can accept an essentially non-basic trifluoroacetato anion (Brown, 1980). The triphenylbis(trifluoroacetato)stannate ion constitutes the first example of a di(carboxylato)triorganostannate (Tiekink, 1991, 1994).



(I)

The discrete nature of this triphenylbis(trifluoroacetato)stannate contrasts with that of (dicarboxylato)-triphenylstannates, such as the oxalato (Ng, Kumar Das, Luo & Mak, 1994) and succinato (Ng, Kumar Das, Xiao, van der Helm, Holecek & Lycka, 1991) derivatives, which adopt linear polyanionic chain structures.

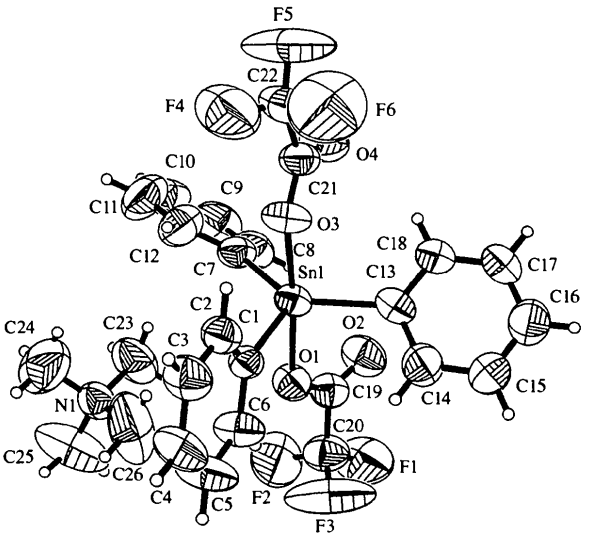


Fig. 1. *ORTEP*II (Johnson, 1976) plot of tetramethylammonium triphenylbis(trifluoroacetato)stannate with ellipsoids at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

Experimental

Tetramethylammonium hydroxide, triphenyltin hydroxide and trifluoroacetic acid (molar ratio 1:1:2) were heated in a small volume of ethanol. The filtered solution when cooled slowly

yielded large (>1 cm) colorless crystals of the title ammonium stannate.

Crystal data

$(C_4H_{12}N)[Sn(C_2F_3O_2)_2(C_6H_5)_3]$
M_r = 650.18
Orthorhombic
Pbca
a = 16.700 (1) Å
b = 17.781 (1) Å
c = 19.708 (1) Å
V = 5852.1 (6) Å^3
Z = 8
D_x = 1.476 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 12–13°
μ = 0.941 mm^{-1}
T = 298 (2) K
Block
0.50 × 0.50 × 0.50 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
T_{min} = 0.550, T_{max} = 0.625
5134 measured reflections
5134 independent reflections
3195 reflections with $I > 2\sigma(I)$
θ_{max} = 25°
$h = -19 \rightarrow 0$
$k = -21 \rightarrow 0$
$l = -23 \rightarrow 0$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R(F)$ = 0.0509
$wR(F^2)$ = 0.1310
S = 0.990
5134 reflections
307 parameters
H atoms: U(H) = 1.5U_{eq}(C)
$w = 1/[\sigma^2(F_o^2) + (0.0717P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.572 e Å^{-3}
$\Delta\rho_{min}$ = −0.481 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Sn1—C1	2.152 (3)	Sn1—O1	2.255 (4)
Sn1—C7	2.131 (3)	Sn1—O3	2.219 (3)
Sn1—C13	2.137 (3)		
C1—Sn1—C7	118.7 (2)	C7—Sn1—O1	88.9 (1)
C1—Sn1—C13	111.9 (1)	C7—Sn1—O3	90.6 (1)
C1—Sn1—O1	87.4 (1)	C13—Sn1—O1	90.7 (1)
C1—Sn1—O3	88.6 (1)	C13—Sn1—O3	93.4 (2)
C7—Sn1—C13	129.3 (2)	O1—Sn1—O3	175.1 (1)

The CF_3 and $(CH_3)_4N$ groups are disordered but the disorder could not be resolved. The disorder affected the phenyl rings which had to be refined as rigid hexagons.

Data collection: *CAD-4 VAX/PC* (Enraf–Nonius, 1988). Cell refinement: *CAD-4 VAX/PC*. Data reduction: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *DIRDIF*94 (Beurskens *et al.*, 1994). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976) in *SHELXTL/PC* (Sheldrick, 1990). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, atomic coordinates and complete geometry have been deposited with the IUCr (Reference: SK1049). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Pteridin-6-yl Alkene-1,2-dithiolate Complex of Cobalt

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Abstract

The crystal structure of the title compound, (η^5-cyclopentadienyl)[6-(1,2-dimercaptoethen-1-yl)-2-(*N*,*N*′-dimethylaminomethylideneamino-3-methylpteridin-4(3*H*)-