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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(n-C) = 0.007$ Å
Disorder in main residue
 R factor = 0.051
 wR factor = 0.157
Data-to-parameter ratio = 10.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

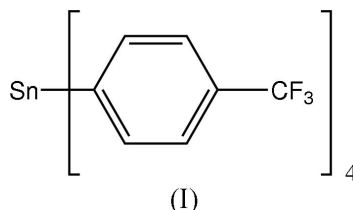
Tetrakis[4-(trifluoromethyl)phenyl]stannane

The Sn atom in the title compound, $[\text{Sn}(\text{C}_7\text{H}_4\text{F}_3)_4]$, is located on a site of symmetry $\bar{4}$ and has tetrahedral geometry, with an Sn—C bond distance of 2.149 (4) Å, and C—Sn—C bond angles of 107.89 (19) and 112.7 (4)°.

Comment

Interest in the structural chemistry of tetraaryltin(IV) compounds largely rests in delineating the principles of their crystal packing (e.g. Wharf & Bélanger-Gariépy, 2003; Wharf & Lehuis, 2003). A number of such compounds crystallize with the Sn atom located on a site of symmetry $\bar{4}$, e.g. $(\text{C}_6\text{H}_5)_4\text{Sn}$ (Engelhardt *et al.*, 1982) and $(p\text{-CH}_3\text{C}_6\text{H}_4)_4\text{Sn}$ (Karipides & Wolfe, 1975). By contrast, $(p\text{-ClC}_6\text{H}_4)_4\text{Sn}$ crystallizes in space group $P\bar{1}$, with no molecular symmetry (Ng, 1997).

In order to add to the database of relevant structures available for comparison, the title structure, $(p\text{-CF}_3\text{C}_6\text{H}_4)_4\text{Sn}$, (I), a known compound (King *et al.*, 1986), was investigated.



The Sn atom in (I) (Fig. 1) is located on a site of symmetry $\bar{4}$, and indeed the structure is isostructural with the aforementioned $(\text{C}_6\text{H}_5)_4\text{Sn}$ (Engelhardt *et al.*, 1982) and $(p\text{-CH}_3\text{C}_6\text{H}_4)_4\text{Sn}$ (Karipides & Wolfe, 1975) structures. The unique Sn—C bond distance is 2.149 (4) Å and the two independent C—Sn—C bond angles are 107.89 (19) and 112.7 (4)°, indicating relatively minor distortions from an ideal tetrahedral geometry.

It has been proposed (Schürmann *et al.*, 1999) that very weak C—H...Cl interactions are responsible for the deviation from $\bar{4}$ symmetry in the structure of $(p\text{-ClC}_6\text{H}_4)_4\text{Sn}$ (Ng, 1997). This conclusion is consistent with previous combined experimental/theoretical studies that point to weak intermolecular interactions as being responsible for significant distortions of molecular structure in organotin systems (e.g. Buntine *et al.*, 1998, 2003).

Experimental

The compound was prepared by reaction of activated magnesium (2 g) and *p*-bromotrifluoromethylphenyl bromide (17.5 g, 0.08 mol) in THF (150 ml). To this cold (273 K) mixture, anhydrous tin tetrachloride (4.56 g, 0.018 mol) in benzene (50 ml) was added dropwise.

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The reaction mixture was refluxed for 1 h and filtered hot. The filtrate was evaporated under vacuum to give a precipitate of the compound, which was recrystallized from an ethanol solution to give colourless needles. Yield 9.17 g, 75%. M.p. 422–423 K (literature 423–424 K; King *et al.*, 1986).

Crystal data

[Sn(C₇H₄F₃)₄]
M_r = 699.12
 Tetragonal, $I\bar{4}$
a = 14.748 (5) Å
c = 6.465 (8) Å
V = 1406.1 (18) Å³
Z = 2
D_x = 1.651 Mg m⁻³

Mo *K*α radiation
 Cell parameters from 25 reflections
 θ = 9.5–12.0°
 μ = 1.00 mm⁻¹
T = 293 (2) K
 Needle, colourless
 0.40 × 0.15 × 0.07 mm

Data collection

Rigaku AFC-7R diffractometer
 ω -2 θ scans
 Absorption correction: ψ scan
 (North *et al.*, 1968)
T_{min} = 0.695, *T_{max}* = 0.934
 949 measured reflections
 883 independent reflections
 567 reflections with *I* > 2 σ (*I*)

R_{int} = 0.039
 θ_{max} = 27.5°
h = 0 → 19
k = 0 → 19
l = 0 → 8
 3 standard reflections
 every 150 reflections
 intensity decay: 1.3%

Refinement

Refinement on *F*²
R[*F*² > 2 σ (*F*²)] = 0.051
wR(*F*²) = 0.157
S = 0.98
 883 reflections
 88 parameters
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.1052P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$

(Δ/σ)_{max} < 0.001
 $\Delta\rho_{\text{max}} = 0.72 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.73 \text{ e } \text{Å}^{-3}$
 Extinction correction: none
 Absolute structure: Flack (1983), no Friedel pairs
 Flack parameter: 0.12 (12)

H atoms were included in the riding-model approximation, with C–H distances of 0.93 Å and with *U*_{iso}(H) = 1.2*U*_{eq}(C). High thermal motion was noted for the CF₃ residue. This was modelled so that two sites were resolved for the F atoms, with site occupancies of 0.69 (4) and 0.31 (4) for the major and minor components, respectively. While this protocol resulted in *R*(*F*², all data) = 0.082, the data/parameter ratio was low, *viz.* 7.3, for the non-centrosymmetric space group. Another protocol, suggested by a referee, was one with considerably more restraints. Here, the six F components of the CF₃ group were restrained to be coplanar, the displacement parameters of each pair of opposite disordered F atoms were made equal, and the C–F and F···F' distances were made approximately equal; the displacement parameters were restrained to be approximately equal. The ratio of the major-to-minor components of the F atoms refined to 0.681 (17):0.319 (17). The result of this refinement strategy was a higher value of *R*(*F*², all data) of 0.099 but a higher data/parameter ratio, *viz.* 10.0, with a consequent improvement in the standard uncertainty values. The results of the latter refinement are reported here.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1996); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *TEXSAN for Windows*.

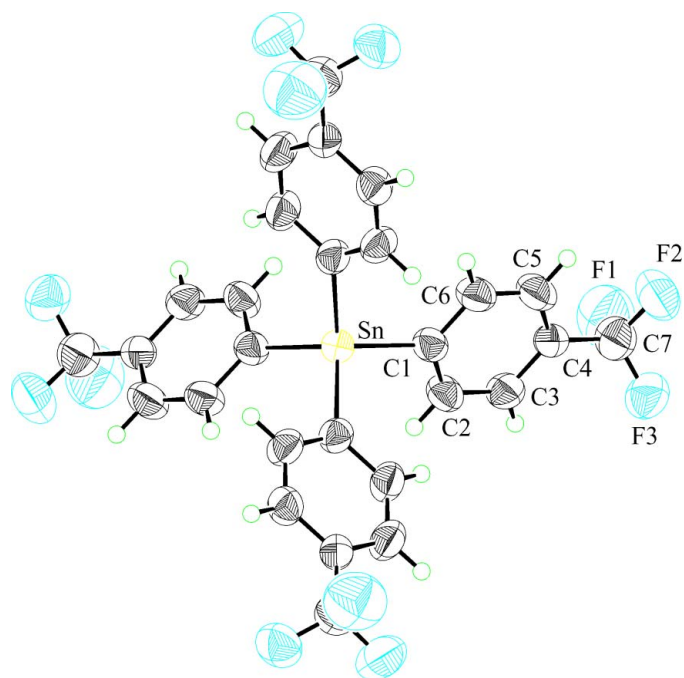


Figure 1

The molecular structure and atomic numbering scheme. Displacement ellipsoids are shown at the 35% probability level. Only the major components of the disordered F atoms are shown. Unlabelled atoms are related to labelled atoms by the $\bar{4}$ symmetry.

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