Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Tetrakis[4-(trifluoromethyl)phenyl]stannane

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Received 13 April 2005
Accepted 19 April 2005
Online 27 April 2005

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(n–C) = 0.007 Å
Disorder in main residue
R factor = 0.051
wR factor = 0.157
Data-to-parameter ratio = 10.0

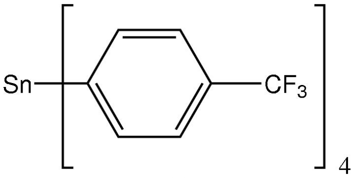
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The Sn atom in the title compound, [Sn($C_7H_4F_3$)$_4$], 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 & Lebuis, 2003). A number of such compounds crystallize with the Sn atom located on a site of symmetry, $\bar{4}$, *e.g.* $(C_6H_5)_4Sn$ (Engelhardt *et al.*, 1982) and (p-$CH_3C_6H_4$)$_4$Sn (Karipides & Wolfe, 1975). By contrast, (p-ClC_6H_4)$_4$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-$CF_3C_6H_4$)$_4$Sn, (I), a known compound (King *et al.*, 1986), was investigated.



(I)

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 $(C_6H_5)_4Sn$ (Engelhardt *et al.*, 1982) and (p-$CH_3C_6H_4$)$_4$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-ClC_6H_4)$_4$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.

doi:10.1107/S1600536805012213

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

$[Sn(C_7H_4F_3)_4]$
$M_r = 699.12$
Tetragonal, $I\bar{4}$
$a = 14.748$ (5) Å
$c = 6.465$ (8) Å
$V = 1406.1$ (18) Å^3
$Z = 2$
$D_x = 1.651$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 9.5$–$12.0°$
$\mu = 1.00$ mm^{-1}
$T = 293$ (2) K
Needle, colourless
$0.40 \times 0.15 \times 0.07$ mm

Data collection

Rigaku AFC-7*R* diffractometer
ω–2θ 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 $I > 2\sigma(I)$
$R_{\rm int} = 0.039$
$\theta_{\max} = 27.5°$
$h = 0 \rightarrow 19$
$k = 0 \rightarrow 19$
$l = 0 \rightarrow 8$
3 standard reflections every 150 reflections intensity decay: 1.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.157$
$S = 0.98$
883 reflections
88 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1052P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.72$ e Å^{-3}
$\Delta\rho_{\min} = -0.73$ e 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 $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. High thermal motion was noted for the CF_3 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 CF_3 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$\cdots$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.

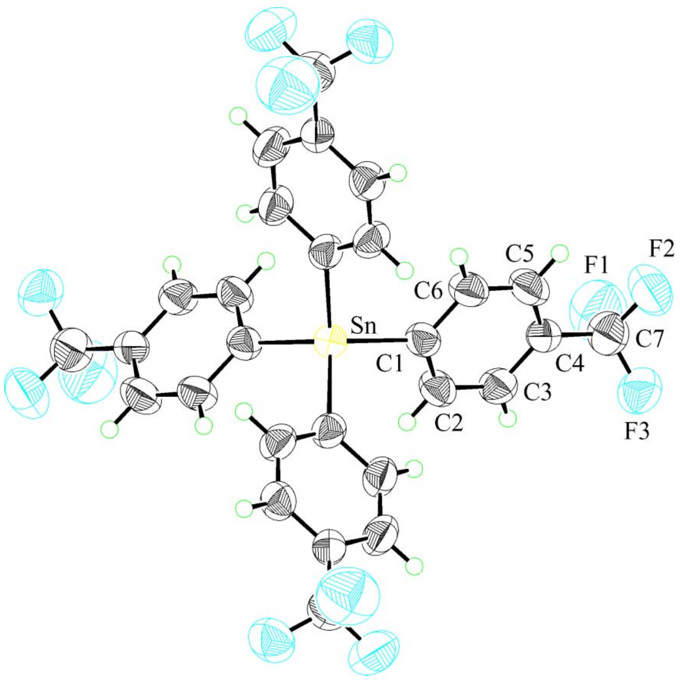


Figure 1
The molecular structure and atomic numbering scheme. Displacement ellipsoids are shown at the 35% probability level. Only the major components of the disordered F atoms are shown. Unlabelled atoms are related to labelled atoms by the $\bar{4}$ symmetry.

AusAID (PM) and the Queensland Government, for the award of a Smart Returns Fellowship (ERTT), are thanked.

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