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Crystallographic report

Tri(*p*-fluorobenzyl)tin *N*-methylpiperazinyldithiocarbamate

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The tin atom is a distorted trigonal bipyramid geometry defined by sulfur donors derived from the asymmetrically binding dithiocarbamate ligand and three *ipso*-carbon atoms from the *p*-fluorobenzyl substituents. Copyright © 2004 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; *p*-fluorobenzyltin; *N*-methylpiperazinyldithiocarbamate

COMMENT

A distorted trigonal bipyramidal geometry about the tin atom was found in the title structure that was investigated during the study of the structural diversity in these and analogous organotin dithiocarbamates.[1–4] The structure Fig. 1, is similar, for example, to those reported for compounds $(4\text{-}FC_6H_4CH_2)_3SnS_2CNC_5H_{10}$[5] and $(2\text{-}FC_6H_4CH_2)_3SnS_2CN(CH_2CH_2)_2O$.[6]

EXPERIMENTAL

$Na[S_2CN(CH_2CH_2)NCH_3]$ (2.0 mmol) was added to a CH_2Cl_2 solution (30 ml) of $(4\text{-}FC_6H_4CH_2)_3SnCl$ (2.0 mmol) and stirred for 8 h at 30 °C. The precipitated NaCl was removed by filtration and the filtrate was concentrated to about 5 ml under reduced pressure. Hexane (5 ml) were added to this solution, immediately a precipitate was formed. The product was recrystallized from CH_2Cl_2–hexane to give colorless crystals; m.p. 111–112 °C; IR (KBr), ν: 1487, 1140, 1002, 547, 466 cm^{-1}. Intensity data were collected at 298 K on a Bruker Smart 1000 CCD for a block 0.19 × 0.41 × 0.45 mm^3. $C_{27}H_{29}F_3N_2S_2Sn$, $M = 621.33$, monoclinic, $P2_1/n$, $a = 18.881(4)$, $b = 8.2353(17)$, $c = 19.035(4)$ Å, $\beta = 108.886(3)°$, $V = 2800.4(10)$ $Å^3$, $Z = 4$, 4932 unique data ($\theta_{max} = 25.0°$), $R = 0.028$ (3986 data with $I \geq 2\sigma(I)$), $wR = 0.081$ (all data). Programs used: SHELXL and ORTEP. CCDC deposition number: 237869.

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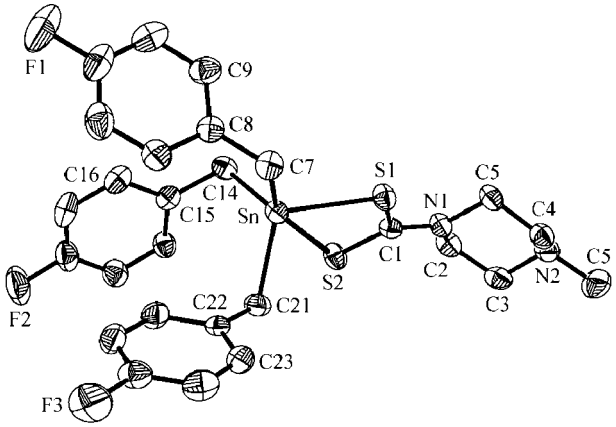

Figure 1. The molecular structure of $(4\text{-}FC_6H_4CH_2)_2SnS_2 - CN(CH_2CH_2)NCH_3$; the hydrogen atoms and solvent CH_2Cl_2 molecule have been removed for clarity. Key geometric parameters: Sn–S1 2.4718(9), Sn–S2 3.1261(10), Sn–C7 2.174(3), Sn–C14 2.159(3), Sn–C21 2.154(3), S1–C1 1.765(3), S2–C1 1.688(3) Å; S1–Sn–S2 62.96(2), S1–Sn–C7 94.15(9), S1–Sn–C14 117.18(8), S1–Sn–C21 109.66(9), S2–Sn–C7 157.08(9), S2–Sn–C14 82.80(9), S2–Sn–C21 80.29(9), C7–Sn–C14 108.79(12), C7–Sn–C21 110.77(13), C14–Sn–C21 114.42(12)°.

REFERENCES

1. Tiekink ERT. *Main Group Met. Chem.* 1992; **15**: 161.
2. Dakternieks D, Zhu H, Masi D, Mealli C. *Inorg. Chem.* 1992; **31**: 3601.
3. Sheldrick GM, Sheldrick WS. *J Chem Soc A* 1970; 490.
4. Yin HD, Wang CH. *Appl. Organometal. Chem.* 2004; **18**: 145.
5. Yin HD, Wang CH, Ma CL. *Chin. J. Org. Chem.* 2004; **24**: 145.
6. Yin HD, Wang CH, Hong M, Wang DQ. *J. Organometal. Chem.* 2004; **689**: 1277.