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Crystallographic report

Dipyridium mercaptoacetotrichlorophenylstannate

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The tin atom in the anion of $[C_5H_5NH]_2[PhSn(SCH_2COO)Cl_3]$ is six-coordinated within an octahedral geometry defined by a CCl_3OS donor set; the cations and anion interact via hydrogen bonds. Copyright © 2005 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; organotin; hydrogen bonding

COMMENT

The study of ionic organotin compounds is of current interest owing to their diversified molecular structures and wide range of applications, such as biological activities[1,2] and as catalysts in organic synthesis.[3] Recently, we studied the reaction of mercaptoacetic acid with $PhSnCl_3$ in the presence of organic amines.[4] The products were generally formulated as $[R_3NH][PhSn(\mu^2\text{-}SCH_2COO)_2]$ for most amines (e.g. $HNEt_2$, NEt_3, $HN(i\text{-}Pr)_2$, and $N(n\text{-}Pr)_3$) in which the tin atom exists in a trigonal bipyramid geometry. Repeating this reaction in the presence of pyridine resulted in the isolation of $[C_5H_5NH]_2[PhSn(SCH_2COO)Cl_3]$; Fig. 1. The tin atom in the dianion is six-coordinated in an octahedral geometry and interacts with the cations via hydrogen bonds.

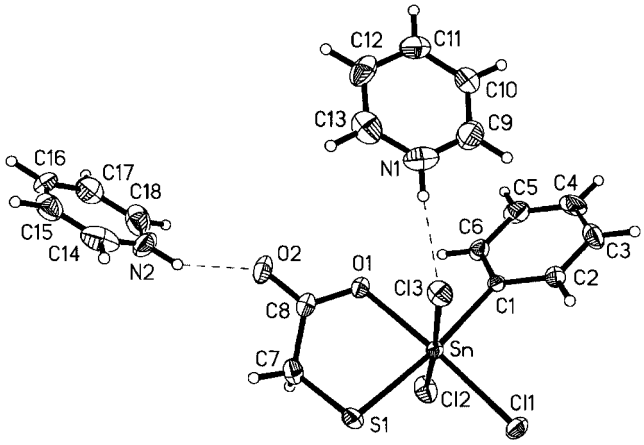

Figure 1. Molecular structure of $[C_5H_5NH]_2[PhSn(SCH_2COO)Cl_3]$. Key geometric parameters: Sn–C1 2.145(4), Sn–O1 2.174(3), Sn–S1 2.413(14), Sn–Cl1 2.4713(13), Sn–Cl2 2.5299(15), Sn–Cl3 2.6333(14) Å; C1–Sn–S1 172.82(11), O1–Sn–Cl1 172.38(9), Cl2–Sn–Cl3 174.89(5)°. Hydrogen bonds: N1–H1···Cl3 2.33, N1···Cl3 3.182(5) Å and angle at H 171°; N2–H···O2 1.92, N2···O2 2.710(6) Å and angle 152°.

EXPERIMENTAL

The title compound was synthesized according to Ref. 4. Crystals were isolated from an acetone/ether (1/3) solution; m.p. 118–120 °C. Anal. Found: C, 38.97; H, 3.51; N, 5.07. Calc. for $C_{18}H_{19}Cl_3N_2O_2SSn$: C, 39.13; H, 3.60; N, 5.03%. IR (cm^{-1}): 455 (νSn–O), 559 (νSn–C), 1586/1359 (ν_{as}/ν_sC=O). Intensity data were collected at 293 K on a Bruker Smart 1000 diffractometer for a colorless block 0.14 × 0.24 × 0.26 mm^3. $C_{18}H_{19}Cl_3N_2O_2SSn$, $M = 552.45$, monoclinic, $P2_1/n$, $a = 8.817(3)$, $b = 8.418(3)$, $c = 28.868(10)$ Å, $\beta = 96.195(6)°$, $V = 2130.3(12)$ $Å^3$, $Z = 4$, 4364 unique data (θ_{max} 26.4°), 3333 data with $I > 2\sigma(I)$. $R = 0.040$ (obs. data), $wR = 0.095$ (all data). Programs used: SHELXL and ORTEP. CCDC deposition number: 239505.

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