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X-ray structure of $Et_4NNO_3 \cdot SnPh_2Cl_2$

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Comment

The structure of $Et_4NNO_3 \cdot SnPh_2Cl_2$ (Figure 1) consists of a nitrate mono-coordinating $SnPh_2Cl_2$ at tin center. The coordination sphere around tin is irregular as evidenced by the value of Cl(2)-Sn-O(3)=175° (the Sn-O and Sn-Cl distances are 2.292 Å and 2.556 Å, respectively). The sum of the bond angles C(1)-Sn(1)-Cl(1) (106.2°), Cl(1)-Sn(1)-C(7) (109.8°) and C(7)-Sn(1)-C(1) (143.7°) is 359.7°, which indicates that Sn, Cl(1), C(7), and C(1) atoms are in the same plane.

The trigonal bipyramidal geometry around tin in $Et_4NNO_3 \cdot SnPh_2Cl_2$ is similar to the pyrazine, benzthiazole, and triphenylphosphine oxide adducts of diphenyltin dichloride reported by Harrison and Molloy (1978) and Cunningham et al. (1998, 2000), respectively.

The C-Sn-C bond angle difference (131.05° and 143.7° for $Et_4NNO_3 \cdot SnPh_2Cl_2$) outlines the steric difference effects between the nitrate and the triphenylphosphine. The Sn-O bond of this studied adduct (2.292 Å) and the bond of $SnPh_2Cl_2 \cdot OPPh_3$ (2.278 Å) are in the range of the Sn-O bonds lengths reported (Cunningham et al., 1998). Between $[NO_3 \cdot SnPh_2Cl_2]^-$ and Et_4N^+ the interactions are electrostatic type.

Crystal structure

A crystal of approximate dimension 0.21×0.13×0.11 mm was used for data collection. Experimental details relating to the crystal class, method of data collection, and refinement results are given in Table 1. Selected geometric data are given in Table 2.

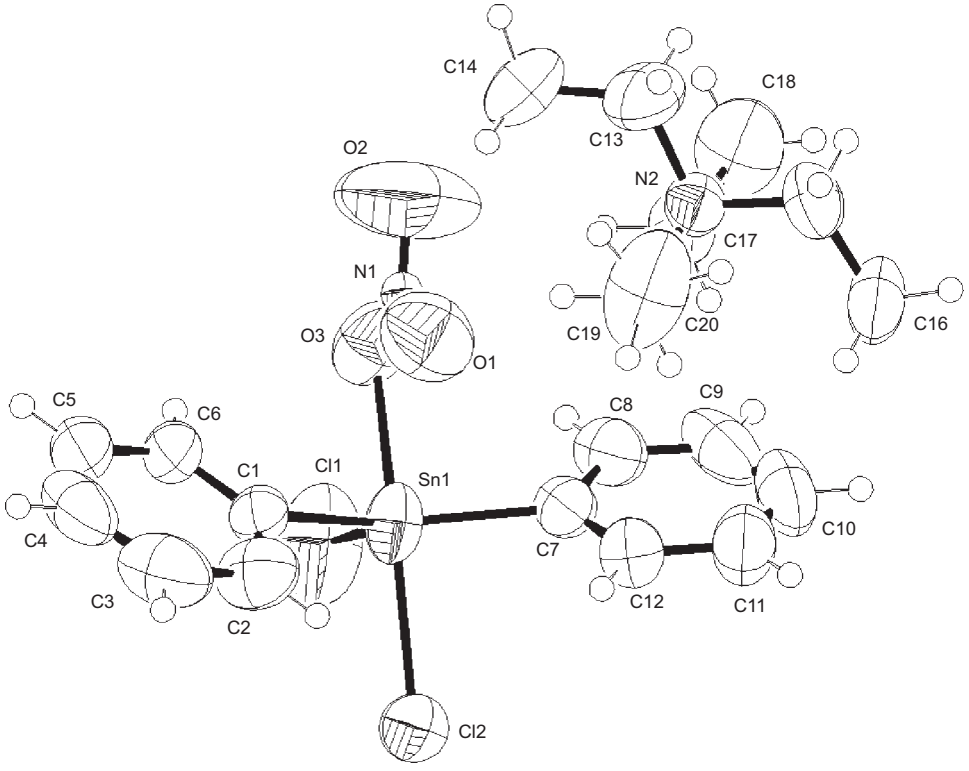

Figure 1 An ORTEP view of the asymmetric unit of the title compound, showing the atom numbering scheme. Displacement ellipsoids are plotted at the 50% probability level.

Table 1 Crystal data and structure refinement.

Empirical formula: $C_{20}H_{30}Cl_2N_2O_3Sn$; formula weight: 536.05; crystal system: monoclinic; crystal size (mm): 0.21×0.13×0.11; space group: $P_{21/c}$; a=11.6958(10) Å; b=15.8062(9) Å; c=13.9616(12) Å; β°=111.185(9); V=2406.6 (3) $Å^3$; Z=4; diffractometer: X-calibur 2CCD4; temperature: 297(2) K; η(cm^{-1})=1.305; transmission factors: 0.831 and 0.907; Dcal=1.479 g cm^3; F(000) 1088; θ_{max}=24.7; Refl. meas./unique: 15,939/4098; Refl. unique R(int): 4098, 0.0480; reflection with I>nσ (I) 2662; R(F), R_w 0.0905, 0.2411; weighting scheme w=1/[s^2 (F_o^2)+(0.1337P)^2+9.6121P] where P=(F_o^2+2F_c^2)/3; ρ, e.A^{-3} 1.524 and 2.083; programs used: SHELXS, SHELXL (Sheldrick, 1997), SIR92 (Altomare et al., 1993), ORTEP3 (Farrugia, 1997); deposition number: CCDC 771510.

Table 2 Selected bond distances.

(Å) C(1)-C(6) 1.355(15); C(1)-C(2) 1.379(17); C(1)-Sn(1) 2.129(10); C(2)-C(3) 1.405(19); C(7)-C(8) 1.372(16); C(7)-C(12) 1.379(16); C(7)-Sn(1) 2.123(10); C(8)-C(9) 1.382(18); Cl(1)-Sn(1) 2.387(4); Cl(2)-Sn(1) 2.556(4); Sn(1)-O(3) 2.292(11); N(1)-O(3) 1.052(12); N(1)-O(2) 1.155(16), and angles (°) C(12)-C(7)-Sn(1) 120.9(8); C(7)-C(8)-C(9) 119.6(14); C(11)-C(12)-H(12) 120.2; C(7)-Sn(1)-C(1) 143.7(4); C(7)-Sn(1)-O(3) 90.3(4); C(1)-Sn(1)-O(3) 90.3(4); C(7)-Sn(1)-Cl(1) 109.8(3); C(1)-Sn(1)-Cl(1) 106.2(3); O(3)-Sn(1)-Cl(1) 82.1(4); C(7)-Sn(1)-Cl(2) 90.3(3); C(1)-Sn(1)-Cl(2) 92.2(3); O(3)-Sn(1)-Cl(2) 175.0(3); equivalent positions: x, y, z; -x+2, +y-1/2, -z+1/2; x, -y+1/2, +z+1/2; x-1, +y, +z.

Experimental

Regular crystals of the title compound have been obtained as a white crystalline solid by reacting dichloro(diphenyl)tin(IV) with tetramethylammonium nitrate in methanol in 1/2 ratio. After a slow solvent evaporation, regular colorless crystals suitable for X-ray work were obtained.

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