APPLIED ORGANOMETALLIC CHEMISTRY
Appl. Organometal. Chem. 2005; **19**: 179–180
Published online in Wiley InterScience (www.interscience.wiley.com). DOI:10.1002/aoc.670

Crystallographic report

L-(−)-Dichloro(β-menthoxycarbonylethyl)tin *N,N*-diethyldithiocarbamate

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Received 9 March 2004; Revised 9 April 2004; Accepted 12 April 2004

The tin atom in the title compound adopts a distorted octahedral geometry within a CCl_2OS_2 donor set. Copyright © 2004 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; organotin; dithiocarbamate

COMMENT

The structural chemistry of estertin and organotin dithiocarbamates continues to be the focus of much research.[1–3] The title complex exists as a discrete molecule and features a distorted octahedral geometry. The structure is comparable to the reported compound $CH_3OCOCH_2CH_2SnCl_2(S_2CN(CH_3)_2)$.[4]

EXPERIMENTAL

A solution of $NaS_2CNEt_2{\cdot}3H_2O$ (1.12 g, 6 mmol) dissolved in ethanol (40 ml) was added dropwise to a solution of β-menthoxycarbonylethyltin trichloride (2.18 g, 6 mmol) in the solvent (40 ml) at room temperature. The reaction mixture was stirred for 1 h. The NaCl that formed was removed by filtration. The filtrate, after distilling off the excess solvent, yielded a crystalline solid, which was recrystallized from a dichloromethane/*n*-hexane (1 : 1, v/v) mixture. Yield 81%, m.p. 145–146 °C, $[\alpha]_D^{25}$ − 49.2°. IR, *ν*: 1640 (vs, C═O), 1521 (vs, C–N), 998 cm^{-1} (m, C–S). ^{1}H NMR (500 MHz, $CDCl_3$) *δ*: 5.07 (1H, dt, J_{aa} = 10.9 Hz, J_{ae} = 4.4 Hz, CHO), 3.75 (4H, q, *J* = 7.2 Hz, $2NCH_2$), 2.89 (2H, t, *J* = 7.5 Hz, $J(^{119}Sn-^1H)$ = 205.9 Hz, $COCH_2$), 1.87 (2H, t, *J* = 7.5 Hz, $J(^{119}Sn-^1H)$ = 111.4 Hz, CH_2Sn), 1.34 (6H, t, *J* = 7.2 Hz, $N(CH_2CH_3)_2$), 2.09–0.89 (9H, m), 0.93 (3H, d, *J* = 6.7 Hz, CH_3), 0.91 (3H, d, *J* = 7.0 Hz, CH_3), 0.80 ppm (3H, d, *J* = 6.9 Hz, CH_3) (Men). ^{13}C NMR (125 MHz, $CDCl_3$) *δ*: 194.62 (C═S), 180.58 (C═O), 78.33 (OCH), 52.11 (NCH_2), 32.26 ($SnCH_2$, $J(^{119/117}Sn-C)$ = 959.6/916.0 Hz), 29.31 (CH_2CO, $J(^{119}Sn-C)$ = 75.0 Hz), 12.14 (NCH_2CH_3), 46.99, 40.40, 33.91, 31.30, 26.54, 23.29, 22.03, 20.80, 16.40 ppm (Men). Anal. Found: C, 39.28; H, 5.89; N, 2.40. Calc. for $C_{18}H_{33}Cl_2O_2S_2Sn$: C, 39.36; H, 6.06; N, 2.55%. Intensity data were collected at 293 K on a Bruker SMART CCD diffractometer using a colorless crystal 0.22 × 0.38 × 0.46 mm^3. $C_{18}H_{33}Cl_2NO_2S_2Sn$, *M* = 549.16, monoclinic, $P2_1$, *a* = 10.314(3), *b* = 12.095(3), *c* = 20.359(6) Å, *β* = 90.121(4)°, *V* = 2539.8(13) $Å^3$, *Z* = 5, 9457 unique data (θ_{max}28.3°), *R* = 0.045, *wR* = 0.077. The Flack parameter is −0.020(17). The compound crystallizes with two independent molecules in the crystallographic asymmetric unit that do not differ from each other

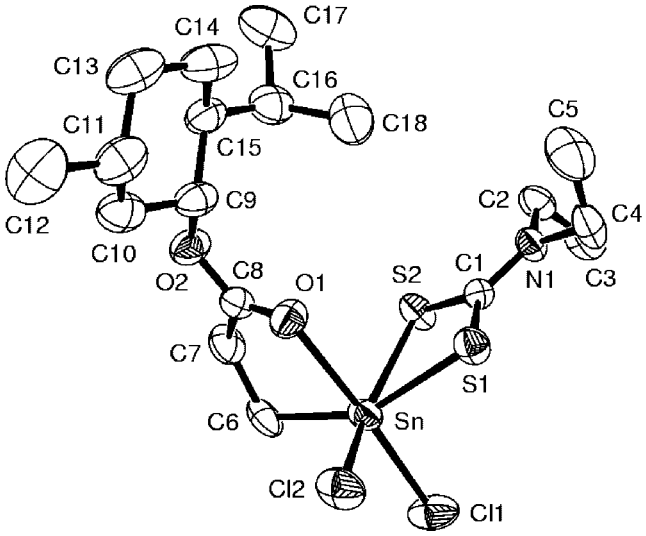

Figure 1. Molecular structure of one of the independent molecules of $MenOCOCH_2CH_2SnCl_2(S_2CN(CH_2CH_3)_2)$; hydrogen atoms are omitted for clarity. Selected geometric parameters: Sn–Cl1 2.3998(15), Sn–Cl2 2.4050(15), Sn–S1 2.4623(14), Sn–S2 2.6940(14), Sn–O1 2.372(4), Sn–C6 2.136(5) Å; Cl1–Sn1–Cl2 93.68(5), Cl1–Sn–O1 176.69(10), Cl2–Sn1–S2 159.68(5), S1–Sn1–C6 152.52(15)°.

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Contract/grant sponsor: National Science Foundation of China; Contract/grant number: 20173050.

significantly. For clarity, only one molecule is shown in Fig. 1. Programs used: SHELXTL, WINGX, ORTEP. CCDC deposition number: 233185.

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