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FURTHER STUDY OF ESTERTIN TRICHLORIDES, $Cl_3SnCH_2CH_2CO_2R$. LEWIS ACIDITY TOWARDS ACETONITRILE. CRYSTAL STRUCTURE OF $Cl_3SnCH_2CH_2CO_2Pr$-i

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Summary

Crystals of $Cl_3SnCH_2CH_2CO_2Pr$-i are orthorhombic, space group $P2_12_12_1$ with a 9.638(6), b 10.004(7) and c 12.848(8) Å. The tin atom is five-coordinate with two chlorines and carbon equatorial and the remaining chlorine and the carbonyl oxygen axial, in a distorted trigonal-bipyramidal arrangement: $(Sn–Cl)_{ax}$ 2.389(3), average $(Sn–Cl)_{eq}$ 2.320(2), Sn–C 2.142(9), Sn–O 2.337(5) Å. Apart from the equatorial chlorine and the terminal carbons in the isopropyl group, all non-hydrogen atoms are essentially coplanar. The molecule approaches C_{2v} symmetry although not constrained to do so by the crystallographic space group.

In MeCN solution, the compounds $Cl_3SnCH_2CH_2CO_2R$ (I, R = Me, Pr-i, C_6H_4X (X = *p*-MeO, H, *p*-Cl, *o*-MeO) or $C_6H_3Cl_2$-2,4) form as equilibrium mixtures of 1/1 and 2/1 MeCN/I complexes; the chelate ring is broken in the 2/1 complexes. Equilibrium constants indicate that the strength of the intramolecular Sn–O coordination in I increases with the electron releasing ability of the R group.

Introduction

The so-called estertin chlorides, $Cl_3SnCH_2CH_2CO_2R$ (I), $Cl_2Sn(CH_2CH_2CO_2R)_2$ etc. have attracted considerable attention ever since their syntheses were first reported [1–10]. Included among the aspects studied have been their structural and coordination chemistry. A crystal structure determination of I (R = Me) revealed a penta-coordinate tin atom and tin-carbonyl oxygen coordination [8] (Fig. 1). The ν(C=O) value of I (R = Me) in the solid state is 1658 cm^{-1}, compared to an

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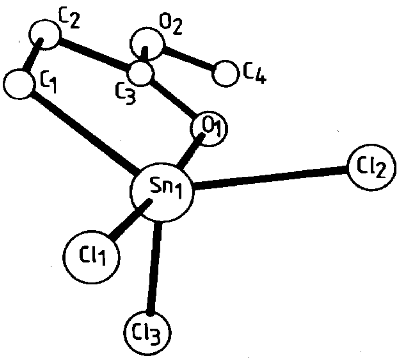
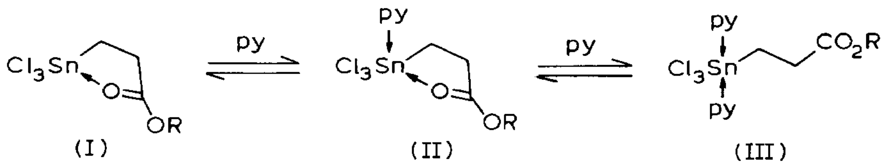


Fig. 1. Molecular structure of $Cl_3SnCH_2CH_2CO_2Me$ [8].

expected value of ca. 1740 cm^{-1} for an uncomplexed ester group. As reported earlier, values of ν(C=O) for I (R = Me) [7] and I (R = Et) [10] in CH_2Cl_2 solution indicated that the ester groups remain completely complexed in this non-coordinating solvent. Such intramolecular coordination within I has an impact on the Lewis acidity or acceptor ability towards external donors. This is especially so when the intramolecular Sn–O interaction has to be broken to accommodate the external donor molecule. Previous work reported similar acceptor strengths for I (R = Me) and I (R = Et) [7,10] in CH_2Cl_2 solution towards nitrogen donors, e.g. pyridine, as shown by equilibrium constants K_1 and K_2 at 25°C:



(1)

	$K_1 = [II]/[I][py]$;	$K_2 = [III]/[II][py]$
R = Me	$>10^5$	$10^{0.80}$
R = Et	$>10^5$	$10^{0.29}$

The similarity of the K_2 values for I (R = Me) and I (R = Et) suggests that differences in the electronic effects (or combinations of the electronic and steric effects) of the Me and Et groups are too slight to result in any significant differences in the strengths of the Sn–O intramolecular interactions.

A series of compounds I, containing both R = aryl and alkyl groups, has now been studied with a view to ascertaining how the strength of the tin–O coordination in I varies with the R group. We also report on the crystal and molecular structure of I (R = Pr-i).

Experimental

Aryl acrylates. Trifluoroacetic anhydride (0.18 *M*) was added with stirring and cooling to acrylic acid (0.18 *M*) at such a rate as to maintain the temperature below

15°C, and stirring was then continued for a further 15 min. The phenol (0.18 *M*) was added slowly with stirring, and the mixture was left for 1.5 h. Water was added, the organic layer separated, washed successively with 5% NaOH solution and H_2O, dried over $MgSO_4$, and distilled under reduced pressure.

CH_2=$CHCO_2Ph$. b.p. 43–45°C/1 mmHg [lit. 11, 59–60°C/1.4 mmHg]. Anal. Found: C, 72.8; H, 5.4. $C_9H_8O_2$ calcd.: C, 73.0; H, 5.4%. IR (neat) 1725 cm^{-1} (C=O). MS (20 eV) *m/e*.: 148 (M^+, 10%), 94 (35%), 55 (100%).

CH_2=$CHCO_2C_6H_4OMe$-p. b.p. 104°C/1 mmHg [lit. 11, 91-92°C/1 mmHg]. Anal. Found: C, 67.6; H, 5.6. $C_{10}H_{10}O_3$ calcd.: C, 67.4; H, 5.6%. IR (neat) 1720 cm^{-1} (C=O). MS (20 eV) *m/e*: 178 (M^+, 25%), 124 (100%), 109 (38%), 55 (50%).

CH_2=$CHCO_2C_6H_4Cl$-p. b.p. 62–65°C/0.5 mmHg [lit. 11, 101–102°C/3.5 mmHg]. Anal. Found: C, 59.1; H, 3.9; Cl, 19.3. $C_9H_7ClO_2$ calcd.: C, 59.2; H, 3.8; Cl, 19.5%. IR (neat) 1720cm^{-1} (C=O). MS (20 eV) *m/e*: 182 (M^+, 10%), 128 (30%), 55 (100%).

CH_2=$CHCO_2C_6H_3Cl_2$-2,4. b.p. 68–70°C/0.6 mmHg [lit. 12, 105°C/13 mmHg]. Anal. Found: C, 49.7; H, 2.7; Cl, 33.1. $C_9H_6Cl_2O_2$ calcd.: C, 49.8; H, 2.8; Cl, 32.7%. IR (neat) 1728 cm^{-1} (C=O). MS (20 eV) *m/e*: 216 (M^+, 0.5%), 162 (100%), 126 (15%), 98 (25%), 63 (25%).

CH_2=$CHCO_2C_6H_4OMe$-o. b.p. 96–98°C/0.7 mmHg. Anal. Found: C, 67.4; H, 5.6. $C_{10}H_{10}O_3$ calcd.: C, 67.4; H, 5.6%. IR (neat) 1735 cm^{-1} (C=O). MS (20 eV) *m/e*: 178 (M^+, 10%), 124 (100%), 109 (85%), 81 (40%), 55 (35%).

Estertin trichlorides, $Cl_3SnCH_2CH_2CO_2R$. $Cl_3SnCH_2CH_2CO_2Me$ was a freshly recrystallised sample (from $CHCl_3$) prepared for a previous study [7]. $Cl_3SnCH_2CH_2CO_2Pr$-i was a gift from Akzo Chemie (UK) Ltd; it was recrystallised from $CHCl_3$/hexane, m.p. 125–127°C. Anal. Found: C, 21.6; H, 3.4; Cl, 31.3. $C_6H_{11}Cl_3O_2Sn$ calcd.: C, 21.2; H, 3.3; Cl, 31.2%. IR (CH_2Cl_2 soln.) 1650 cm^{-1}. NMR (100 MHz in CD_2Cl_2): δ 1.34 (d, 6H, $(CH_3)_2C$, *J* 6 Hz), 2.10 (t, 2H, CH_2Sn, *J* 7 Hz), 2.88 (t, 2H, CH_2CO, *J* 7 Hz), 5.20 (septet, 1H, $CH(CH_3)_2$, *J* 6 Hz).

Other estertin trichlorides, listed below, were obtained from $SnCl_2$, HCl and acrylic esters, CH_2=$CHCO_2R$, using a general route [1–4].

$Cl_3SnCH_2CH_2CO_2Ph$. m.p. 176°C [lit. 3, m.p. 177°C]. Anal. Found: C, 28.8; H, 2.3; Cl, 28.2. $C_9H_9Cl_3O_2Sn$ calcd.: C, 28.8; H, 2.4; Cl, 28.5%. IR (KBr) 1685 cm^{-1} (C=O). ^1H NMR (220 MHz in CD_3COCD_3): δ 2.41 (t, 2H, CH_2Sn, *J* 7 Hz), 3.36 (t, 2H, CH_2CO, *J* 7 Hz), 7.31 (s, 2H, *o*-protons of Ph), 7.41 (m, 3H, *m*- and *p*-protons of Ph). ^{13}C NMR (20 MHz in CD_3COCD_3) δ 181.31 (C=O); 151.38, 130.47, 127.57 and 121.66 (aryl), 29.40 (CH_2CO), 27.92 (CH_2Sn). MS (20 eV) *m/e*: M^+ (10%).

$Cl_3SnCH_2CH_2CO_2C_6H_4OMe$-p. m.p. 172°C. Anal. Found: C, 29.5; H, 2.7; Cl, 26.2. $C_{10}H_{11}Cl_3O_3Sn$ calcd.: C, 29.7; H, 2.7; Cl, 26.3%. IR (KBr) 1658 cm^{-1} (C=O). ^1H NMR (220 MHz in CD_3COCD_3): δ 2.46 (t, 2H, CH_2Sn *J* 7 Hz), 3.40 (t, CH_2CO_2, 2H, *J* 7 Hz), 3.84 (3H, OCH_3), 7.18 (4H, C_6H_4). ^{13}C NMR (20 MHz in CD_3COCD_3): 181.88 (C=O), 158.84, 144.78, 122.44 and 115.33 (aryl), 55.67 (OCH_3), 29.29 (*CH_2*CO), 27.92 (CH_2Sn). MS (20 eV) *m/e*: M^+ (2%).

$Cl_3SnCH_2CH_2CO_2C_6H_4$-Cl-p. m.p. 159–161°C. Anal. Found: C, 26.3; H, 1.8; Cl, 34.9. $C_9H_8Cl_4O_2Sn$ calcd.: C, 26.4; H, 2.0; Cl, 34.7%. IR (KBr) 1678 cm^{-1} (C=O). ^1H NMR (220 MHz in CD_3COCD_3): δ 2.50 (t, 2H, CH_2Sn, *J* 7 Hz), 3.43 (t, 2H, CH_2CO, *J* 7 Hz), 7.45 (4H, C_6H_4). ^{13}C NMR (20 MHz in CD_3COCD_3): δ 180.75 (C=O), 149.93, 132.41, 130.37 and 123.49 (aryl), 31.09 (*C*H_2CO), 29.44 (CH_2Sn). MS (20 eV) *m/e*: M^+ (2%).

$Cl_3SnCH_2CH_2CO_2C_6H_4Cl_2$-2,4. m.p. 127–128°C. Anal. Found: C, 24.4; H, 1.4; Cl, 39.7. $C_9H_7Cl_5O_2Sn$ calcd.: C, 24.4; H, 1.6; Cl, 40.0%. IR (KBr) 1659 cm^{-1} (C=O). ^1H NMR (220 MHz in CD_3COCD_3): δ 2.36 (t, 2H, CH_2Sn, J 7 Hz), 3.32 (t, 2H, CH_2CO, J 7 Hz), 7.27 and 7.52 (m, 3H, C_6H_3). ^{13}C NMR (20 MHz in CD_3COCD_3): δ 179.16 (C=O), 146.26, 131.19, 130.61, 129.30, 127.78 and 125.33 (aryl), 31.42 (CH_2CO), 29.24 (CH_2Sn). MS (20 eV) m/e: M^+ (4%).

$Cl_3SnCH_2CH_2CO_2C_6H_4OMe$-o. m.p. 133–135°C. Anal. Found: C, 29.5; H, 2.7; Cl, 26.4. $C_{10}H_{11}Cl_3O_3Sn$ calcd.: C, 29.7; H, 2.7; Cl, 26.3%. IR (KBr) 1684 cm^{-1} (C=O). ^1H NMR (220 MHz in CD_3COCD_3): δ 2.35 (t, 2H, CH_2Sn, J 7 Hz), 3.55 (t, 2H, CH_2CO, J 7 Hz), 3.85 (s, 3H, $-OCH_3$), 7.00 (m, 4H aryl). ^{13}C NMR (20 MHz in CD_3COCD_3): δ 181.14 (C=O), 151.41, 140.48, 129.16, 122.53, 121.46 and 113.91 (aryl) 56.60 (OCH_3), 31.59 (CH_2CO), 30.49 ($SnCH_2$). MS (20 eV) m/e: M^+ (1%).

Solvents. Acetonitrile was purified by a standard procedure [13].

Equilibrium constants. These were determined from IR data at 25 ± 1°C in CH_3CN. Solutions of I (ca. 10^{-2} M) were made up and IR cells filled in a glove box. Absorptions were recorded in the carbonyl region (1600–1800 cm^{-1}) and equilibrium constants were calculated from the intensities of the frequencies for complexed and free ester groups.

Determination of the crystal structures of $Cl_3SnCH_2CH_2CO_2Pr$-i

Crystal data. $Cl_3SnCH_2CH_2CO_2Pr$-i is orthorhombic (oscillation photographs about all three axes) with a 9.638(6), b 10.004(7), c 12.848(8) Å. The space group is $P2_12_12_1$ [from systematic absences $h00$ ($0k0$, $00l$) present only for $h(k, l) = 2n$]. The cell contains four molecules to give an X-ray density of 1.823 g cm^{-3} and linear absorption coefficient (μ) 2.69 mm^{-1} for the molecular weight of 340.2.

The unit cell and intensity data were obtained on a Nicolet P3 four circle diffractometer using graphite monochromated Mo-K_α radiation (λ 0.71069 Å). The crystal, of dimensions 1.0 × 0.55 × 0.3 mm, with the $\omega/2\theta$ scan procedure, yielded

TABLE 1

FINAL ATOMIC PARAMETERS FOR $Cl_3SnCH_2CH_2CO_2Pr$-i (Estimated standard deviations applicable to the least significant figures are given in parenthesis. Fractional coordinates are $\times 10^5$ for Sn and $\times 10^4$ for all other atoms and B_{eq} are $\times 10$)

Atom	x	y	z	B_{eq} (Å^2) [a]
Sn	4348(4)	34019(5)	10660(4)	42
Cl(1)	1054(3)	5318(3)	2075(2)	68
Cl(2)	−1916(2)	3480(2)	1455(2)	49
Cl(3)	682(2)	4344(2)	−573(2)	54
O(1)	−169(5)	1426(5)	207(4)	44
O(2)	442(6)	−702(5)	132(5)	52
C(1)	1949(9)	2025(10)	1651(7)	54
C(2)	1813(7)	661(9)	1188(6)	47
C(3)	590(7)	494(7)	467(5)	39
C(4)	−719(9)	−1046(8)	−560(8)	52
C(5)	−1524(16)	−2211(13)	31(7)	103
C(6)	−125(14)	−1259(25)	−1598(10)	157

[a] Calculated as $B_{eq} = 4/3(a^2B_{11} + b^2B_{22} + c^2B_{33})$ for an anisotropic thermal vibration correction of the form $\exp = -(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + B_{23}kl + B_{13}hl + B_{12}hk)$.

TABLE 2

BOND LENGTHS (Å) AND ANGLES (°) FOR $Cl_3SnCH_2CH_2CO_2Pr$-i (Values in parentheses are estimated standard deviations associated with the least significant digits)

Sn–Cl(1)	2.389(3)	O(2)–C(3)	1.280(9)
Sn–Cl(2)	2.321(2)	O(2)–C(4)	1.470(11)
Sn–Cl(3)	2.319(2)	C(1)–C(2)	1.495(13)
Sn–O(1)	2.337(5)	C(2)–C(3)	1.508(10)
Sn–C(1)	2.142(9)	C(4)–C(5)	1.593(18)
O(1)–C(3)	1.231(8)	C(4)–C(6)	1.467(17)
Cl(1)–Sn–Cl(2)	95.8(1)	Sn–O(1)–C(3)	111.4(4)
Cl(1)–Sn–Cl(3)	98.1(1)	Sn–C(1)–C(2)	112.8(6)
Cl(1)–Sn–O(1)	175.3(1)	C(1)–C(2)–C(3)	114.4(7)
Cl(1)–Sn–C(1)	98.9(2)	C(2)–C(3)–O(1)	123.2(6)
Cl(2)–Sn–Cl(3)	106.4(1)	C(2)–C(3)–O(2)	113.4(6)
Cl(2)–Sn–O(1)	83.5(1)	O(1)–C(3)–O(2)	123.4(6)
Cl(2)–Sn–C(1)	127.7(2)	C(3)–O(2)–C(4)	120.5(6)
Cl(3)–Sn–O(1)	86.6(1)	O(2)–C(4)–C(5)	104.7(8)
Cl(3)–Sn–C(1)	120.7(2)	O(2)–C(4)–C(6)	106.6(9)
O(1)–Sn–C(1)	78.0(3)	C(5)–C(4)–C(6)	121.2(11)

2048 unique reflections for $2\theta \leqslant 60^0$ of which 19 with $I < 2\sigma(I)$ were classed as unobserved and excluded from all further calculations. No absorption correction was applied. The calculations were performed on the Honeywell 66/80 computer of the Computing Centre of the University of Aberdeen, using MULTAN 80 [14] and the NRC crystallographic package [15]. Atomic scattering factors were taken from the International Tables for X-ray crystallography [16].

The position of Sn was readily obtained by the application of MULTAN 80. Thereafter the positions of the remaining non-hydrogen atoms were established by

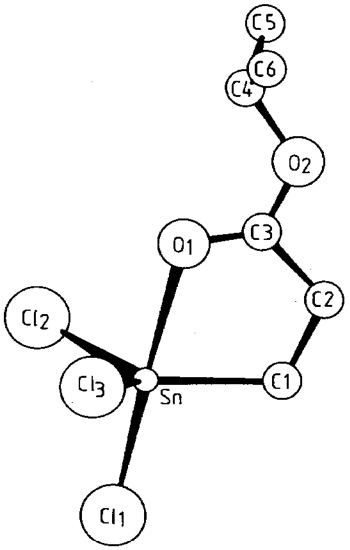


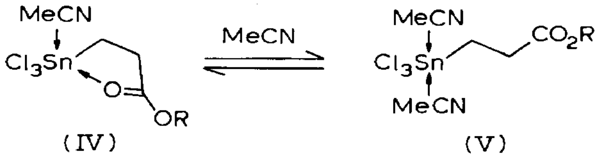
Fig. 2. Molecular structure and atomic labelling in $Cl_3SnCH_2CH_2CO_2Pr$-i.

standard Fourier methods. The structure was refined by block-diagonal least-squares minimising $\Sigma w(|F_0|-|F_c|)^2$, where w was given by $1/\{1+[|F_0|-P2)/P1]^2\}$ and $P1=11$ and $P2=20$ down to 0.055 ($wR=0.057$) with all atoms vibrating anisotropically. At this stage, shifts in parameters (Δ) were, in a few cases, comparable with the corresponding standard deviations (σ) but generally Δ/σ was $\leqslant 0.5$. The correctness of the structure was confirmed by the satisfactory distribution of $\Sigma w\Delta^2$ against $\sin\theta$ and against $|F_0|$ and the essentially featureless difference map. No attempt was made to position the H atoms, or to determine the absolute configuration.

The final atoms parameters are given in Table 1, selected bond lengths and angles in Table 2. Tables of observed and calculated structure factors and of anisotropic thermal vibration frequencies parameters are available from the authors. The structure of $Cl_3SnCH_2CH_2CO_2Pr$-i and the atom numbering scheme are shown in Fig. 2.

Results and discussion

The effects of the R groups on the strength of the chelation in I were clearly indicated in the IR spectra of I in acetonitrile solution. The IR spectra showed two carbonyl absorptions for each I. The ν(C=O) values indicate the presence of both a free and a complexed ester group with the relative amounts of each varying with the R group. Equilibrium 2, with equilibrium constants K_3, is evidently established in the coordinating solvent, MeCN. The values of K_3 reflect the ability of MeCN to compete with the intramolecular ester for coordination to tin.



(2)

K_3 = [V]/[IV] [MeCN]

Acetonitrile is a good donor and has been found to coordinate with tin(IV) chlorides. For example, a solid 2/1 MeCN/$SnCl_4$ complex has been isolated [17] and the formation of a 1/1 MeCN/Me_3SnCl (VI) complex [18] has been studied in CCl_4 solution: the formation constant for VI at 26°C in CCl_4 solution had a value of 1.8 M^{-1}.

In view of the donor ability of MeCN and its concentration, it is reasonable to assume the presence of the mono-coordination complex IV in MeCN solutions. Previous work with strong monodentate donors has indicated that 1/1 species, e.g. II, are readily formed, and have large stability constants; ν(C=O) values for 1/1 complexes, such as II, clearly lie in the region expected for a coordinated ester group [7,10].

As shown in Table 3, values of K_3 decrease with the electron withdrawing ability of the group R, for example see K_3 values for I (R = p-$MeOC_6H_4$, Ph and p-ClC_6H_4). Thus the strength of the tin–carbonyl interaction increases with the greater electron releasing ability of the R group. The stronger the Sn–O interaction, the more difficult it is to replace it by MeCN–Sn complexation, and hence the lower will be the value of K_3.

TABLE 3

ν(C=O) VALUES FOR I IN MeCN SOLUTION AND EQUILIBRIUM CONSTANTS (K_3) IN MeCN SOLUTION (K_3 = [V]/[IV][MeCN])

Compound	ν(C=O)		k_3 (×10² M^{-1})
	[V]	[IV]	
$Cl_3SnCH_2CH_2CO_2Me$	1670	1740	1.7
$Cl_3SnCH_2CH_2CO_2Pr$-i	1645	1724	0.61
$Cl_3SnCH_2CH_2CO_2C_6H_4OMe$-*p*	1691	1753	1.5
$Cl_3SnCH_2CH_2CO_2C_6H_5$	1690	1757	2.2
$Cl_3SnCH_2CH_2CO_2C_6H_4Cl$-*p*	1692	1758	3.2
$Cl_3SnCH_2CH_2CO_2C_6H_4OMe$-*o*	1691	1758	1.5
$Cl_3SnCH_2CH_2CO_2C_6H_3Cl_2$-2,4	1686	1758	13.0

While electronic effects could also account for the fact that K_3 is larger for I (R = Me) than for I (R = Pr-i), steric effects could also play a part. It was therefore decided to determine the crystal structure of I (R = Pr-i) and to compare its structure with that of I (R = Me) previously determined by Harrison et al. [8].

There are great similarities between these structures, see Fig. 1 for I (R = Me) and Fig. 2 for I (R = Pr-i). The structure of I (R = Pr-i) is a distorted bipyramid with two chlorines and the alkyl group in equatorial sites and the remaining chlorine and oxygen in axial positions. The axial Sn–Cl bond length is longer than the equatorial bonds. There are no intermolecular contacts less than 3:538(7) Å. The atoms in the chelate ring as well as the oxygen and α-carbon of the OPr-i group are essentially coplanar. (A similar situation pertained for I (R = Me) [8]) The I (R = Pr-i) molecule approaches C_{2v} symmetry, although not constrained to do so by the crystallographic space group. Of interest, the C_3–O_2 bond lengths in I are shorter than in simple esters, e.g. CH_3CO_2Me (1.36 Å). Comparisons of bond lengths for I (R = Pr-i) and I (R = Me) [8] are made in Fig. 3.

Clearly the Me and Pr-i groups in I (R = Me) and I (R = Pr-i) have little influence on the structures; specificially there are no additional steric problems at the tin

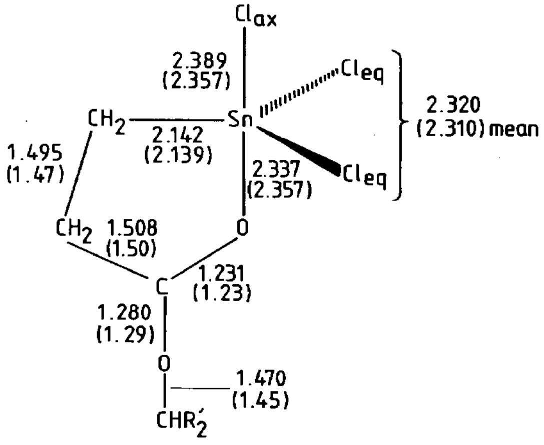


Fig. 3. Comparison of bond lengths for $Cl_3SnCH_2CH_2CO_2Me$ [8] (data in brackets) and $Cl_3SnCH_2CH_2CO_2Pr$-i.

centre in the Pr-i compound. It thus appears that the K_3 values for I (R = Me) and I (R = Pr-i) reflect only electronic effects of the R groups.

References

1 R.E. Hutton and V. Oakes, in J.J. Zuckermann (Ed.), Organotin Compounds: New Chemistry and Applications, Advances in Chemistry Series, Vol. 157 American Chemistry Society, Washington DC, 1976, 123.
2 J.W. Burley, R.E. Hutton and V. Oakes, J. Chem. Soc., Chem. Commun., (1976) 803.
3 R.E. Hutton, J.W. Burley and V. Oakes, J. Organomet. Chem., 156 (1978) 369.
4 J.W. Burley, P. Hope, R.E. Hutton and C.J. Groenenbaum, J. Organomet. Chem., 170 (1979) 21.
5 J.W. Burley, R.E. Hutton, M.R.J. Jolly and C.J. Groenenbaum, J. Organomet. Chem., 251 (1983) 189.
6 J.W. Burley, R.E. Hutton and R.D. Dworkin, J. Organomet. Chem., 284 (1985) 171.
7 D. Maughan, J.L. Wardell and J.W. Burley, J. Organomet. Chem., 212 (1981) 59.
8 P.G. Harrison, T.J. King and M.A. Healy, J. Organomet. Chem., 182 (1979) 17.
9 R.M. Haigh, A.G. Davies and M-W. Tse, J. Organomet. Chem., 174 (1979) 163.
10 R.A. Howie, E.S. Paterson, J.L. Wardell and J.W. Burley, J. Organomet. Chem., 259 (1983) 71.
11 P.L. Magagnini and G. Pizzirani, Gazz. Chim. Ital., 96 (1966) 1035.
12 E. Walling, G. Faerber and W. Schilling, Ger., 1,044, 802.
13 J.F. Coetzee, Pure and Appl. Chem., 13 (1966) 429.
14 P. Main, S.J. Fiske, S.E. Hull, L. Lessinger, G. Germain, J.-P. Declerq and M.M. Woolfson, MULTAN 80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data. Universities of York, England and Louvain, Belgium, 1980.
15 F.R. Ahmed, S.R. Hall, M.E. Pippy and C.P. Huber, NRC Crystallographic Programs for the IBM/360 system. National Research Council of Canada, Ottawa, 1966.
16 International Tables for X-ray Crystallography, Volume IV, Kynoch Press, Birmingham, 1974.
17 I.R. Beattie, G.P. McQuillan, L. Rule and M. Webster, J. Chem. Soc., (1963) 15.
18 T.F. Bolles and R.S. Drago, J. Am. Chem. Soc., 88 (1966) 3921.