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PENTA(METHOXYCARBONYL)CYCLOPENTADIENYL-SUBSTITUTED GERMANIUM(II) AND TIN(II) COMPOUNDS. X-RAY CRYSTAL STRUCTURE OF BIS[PENTA(METHOXYCARBONYL)CYCLOPENTADIENYL]TIN(II) *

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Summary

Reaction of decamethyl-germanocene or -stannocene with equimolar amounts of penta(methoxycarbonyl)cyclopentadiene leads to the ionic compounds pentamethylcyclopentadienylgermanium-penta(methoxycarbonyl)cyclopentadienide (**3**) and pentamethylcyclopentadienyltin-penta(methoxycarbonyl)cyclopentadienide (**4**), in which pentamethylcyclopentadienyl-germanium and -tin cations are present together with the penta(methoxy)cyclopentadienide anion. A molar ratio of 1/2 yields the covalent bis[penta(methoxycarbonyl)cyclopentadienyl]-germanium(II) (**5**) or -tin(II) (**6**). Crystals of the tin compound **6** are orthorhombic, space group $P2_12_12$ with a 15.656(2), b 15.424(2), c 7.033(1) Å and $Z = 2$. The structure of **6** was solved via the heavy-atom method and refined to $R = 0.037$ using 1098 diffractometer data with $I \geqslant 1.5\sigma(I)$. The tin atom, which lies on the two-fold axis, has four-fold oxygen coordination from carbonyl oxygen atoms of two methoxy carbonyl groups on each of two ligands, with Sn–O distances of 2.240(9) and 2.271(9) Å. The compounds **5** and **6** are fluxional in solution.

In recent years it has been shown that germanium(II) and tin(II) compounds (germylenes and stannylenes) can be stabilized in a monomeric form by a variety of substituted cyclopentadienyl ligands. In these compounds the cyclopentadienyl rings are π-bonded to a Main Group element, and they therefore belong to a class which is of considerable interest. The substituents on the cyclopentadienyl ring influence the thermodynamic and kinetic stability of the corresponding Main Group metallocenes.

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* Dedicated to Professor M. Kumada.

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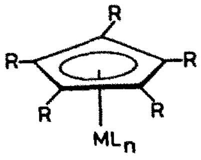
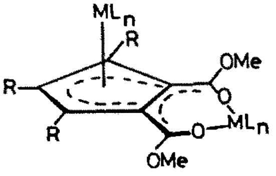
So far the unsubstituted [1,2], the partially and fully methylated [3,4], the partially trimethylsilylated [5] germanocenes and stannocenes and the perphenylated stannocene [6] have been investigated.

The penta(methoxycarbonyl)cyclopentadienyl ligand shows interesting behaviour in its bonding to transition elements. According to reports by Bruce [7] and his group, this ring system can act as a pure π-ligand (**A**), as a partially π- and oxygen-bonded ligand (**B**), as an oxygen-only bonded ligand (**C**), or as a purely ionic ligand (**D**):



A



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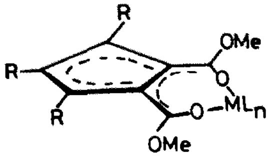
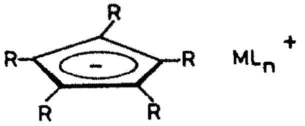


C



D

We were interested in studying a system containing a penta(methoxycarbonyl)cyclopentadienyl ligand and a Main Group element, for which, in principle, all the bonding patterns **A**–**D** are possible. We have therefore investigated appropriate germanium(II) and tin(II) species. In this context it is of interest to note that in the only known example in tin(IV) chemistry the penta(methoxycarbonyl)cyclopentadienyl ligand is ionically bonded [8].

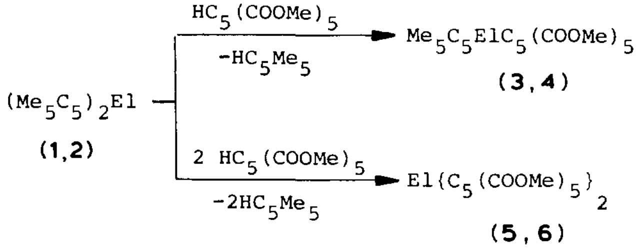
Synthesis and structure

In earlier experiments we were able to show that decamethyl-germanocene and -stannocene are attacked by electrophiles at the π-system of the cyclopentadienyl rings. In reactions with proton acids one or two cyclopentadienyl rings are split off, according to the following equations [4,9]:

$$(Me_5C_5)_2El \xrightarrow[-Me_5C_5H]{+HX} \begin{matrix} Me_5C_5El^+X^- \\ \text{or} \\ Me_5C_5ElX \end{matrix} \xrightarrow[-Me_5C_5H]{+HX} ElX_2$$

(El = Ge, Sn)

Using this type of reaction sequence we synthesized penta(methoxycarbonyl)cyclopentadienyl compounds of germanium(II) and tin(II) [10]. Reaction of one equivalent of dacemethylmetallocene **1** or **2** with one equivalent of the strong acid penta(methoxycarbonyl)cyclopentadiene [11] in benzene solution leads to the colourless crystalline substitution products **3** and **4**, respectively. Use of a 1/2 molar ratio of the above reactants and longer reaction times in methylene chloride allowed the isolation of the bis-penta(methoxycarbonyl)cyclopentadienyl substituted compounds **5** and **6**, respectively, as colourless crystals.

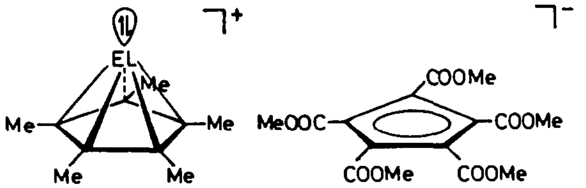


Ge	**1**	**3**	**5**
Sn	**2**	**4**	**6**

The stepwise substitution of penta(methoxycarbonyl)cyclopentadienyl rings can easily be followed by NMR spectroscopy, especially in the case of the germanium compounds, where the second substitution takes several weeks.

Compounds **3** and **4** are very air sensitive, but thermally stable. Compounds **5** and **6** are less air sensitive. The identities of these compounds were confirmed by analytical and spectroscopic data.

Molecular weight determinations, solubility criteria and spectroscopic data indicate that the compounds **3** and **4** have an ionic structure **E**, in which the known [4] pentamethylcyclopentadienyl-germanium and -tin cations are present, with the also well-known [7,8] penta(methoxycarbonyl)cyclopentadienide as counter ion:



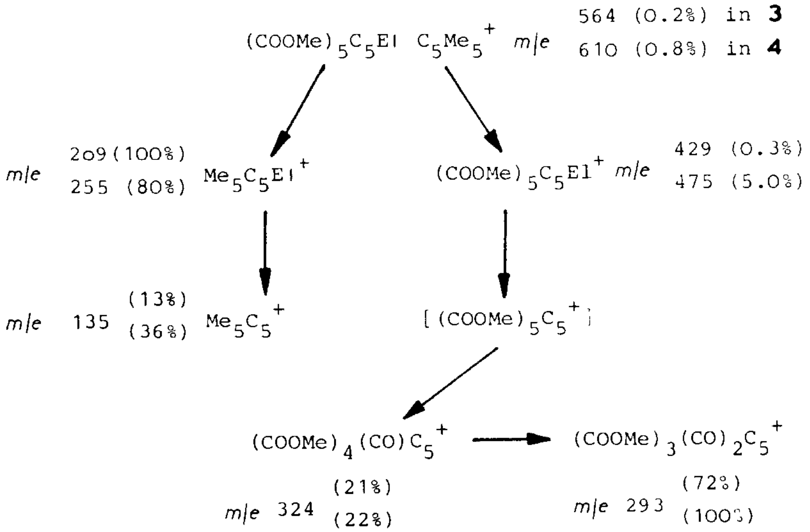
E

El	Ge	Sn
	3	**4**

The molecular weights of **3** and **4** in nitrobenzene are consistent with those expected for 1 : 1 electrolytes; the ^{1}H, ^{13}C and ^{119}Sn NMR data correspond to those found for the *nido*-cluster cation [4] and for the penta(methoxycarbonyl)cyclopentadienide anion [7]. The salts are very slightly soluble in benzene, insoluble in hexane, but readily soluble in methylene chloride and chloroform.

The mass spectra of **3** and **4** show that these salts exist as covalent species in the gas phase. This is demonstrated by the observation of the M^{+} peaks and by typical fragmentation patterns, which are illustrated in Scheme 1.

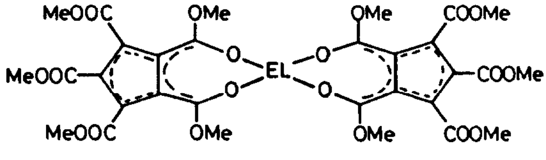
SCHEME 1. Fragmentation processes in the mass spectra of **3** and **4**.



Not only the cluster species $Me_5C_5El^+$, but also the species $(COOMe)_5C_5El^+$ appear in the mass spectra of **3** and **4**. The radical cation $(COOMe)_5C_5{}^+$ is not observed, but there are ions at *m/e* 324 and 293, with high intensity, which correspond to species formed by loss of one or two methoxy groups from this cation. The loosening of steric constraints after loss of one or two methoxy groups presumably allows those remaining to adopt an orientation whereby their $-M$-effects can stabilize the anti-aromatic cation by resonance.

NMR data and solubility criteria provide no clear evidence for assigning the structures of **5** and **6**. Both compounds are soluble in methylene chloride and more so in acetonitrile. The ^1H and ^{13}C NMR spectra indicate that there is only one type of methoxycarbonyl group and ring carbon present. The chemical shift in the ^{119}Sn NMR of **6** (-1739 ppm) is quite different from those found in other tin(II) compounds with π-bonded cyclopentadienyl rings (-2199 for $(C_5H_5)_2Sn$ [12] and -2129 ppm for $(C_5Me_5)_2Sn$ [13]).

The assumption that the cyclopentadienyl rings are not π-bonded in the compound **6** was confirmed by an X-ray crystal structure analysis, which revealed the bonding situation **F**, corresponding, as the first approximation, to type **C**. It is assumed that **5** has an analogous structure.



F

El	Ge	Sn
	5	6

Thus, two penta(methoxycarbonyl)cyclopentadienyl groups act as bidentate ligands using the carbonyl oxygen atoms from two neighbouring groups on each ring (see Fig. 1). The four-fold oxygen coordination of the tin atom, which lies on a crystallographic two-fold axis, is one-sided, but not "pyramidal" in the simplest sense of the word. In fact, probably the best description is to regard the tin-coordination as based on a φ-trigonal bipyramid, with the two ligands spanning two axial/equatorial edges and with the third equatorial site occupied by a lone pair. Distortions from normal bond angles then occur (a) due to some restrictions in the chelate "bite", which gives an O_{ax}–Sn–O_{eq} angle of 77.2(3)°, and (b) due to "lone-pair" repulsions which reduce to O_{ax}–Sn–O_{ax} angle from an ideal 180 to 146.4(2)° and the O_{eq}–Sn–O_{eq} angle from 120 to 79.4 (3)° (see Table 2). This description also correlates with a difference between the unique Sn–O distances, the "axial" Sn–O(21) bond being longer than the "equatorial" Sn–O(11) bond (see Table 1). Detailed analysis of the crystal structure, however, reveals a further complication in that as the SnL_2 molecules stack along the crystallographic two-fold axis, the metal atom also forms two intermolecular Sn · · · O interactions, of length 2.925(10) Å, to a carbonyl oxygen on each of two (symmetry-related) ligands in the next molecule. These interactions thus complete a very distorted octahedral SnO_6 coordination. Apart from the obvious distortions from octahedral geometry in the Sn–O bond lengths and O–Sn–O angles (Tables 1 and 2), there is a dihedral twist angle between the planes formed by O(51b), Sn, O(51c) and O(11), Sn, O(11a) (which might be considered to constitute the "equator" of an octahedron) of 12.6°.

The geometry of the $C_5(COOMe)_5$ ligand is normal, except that there are

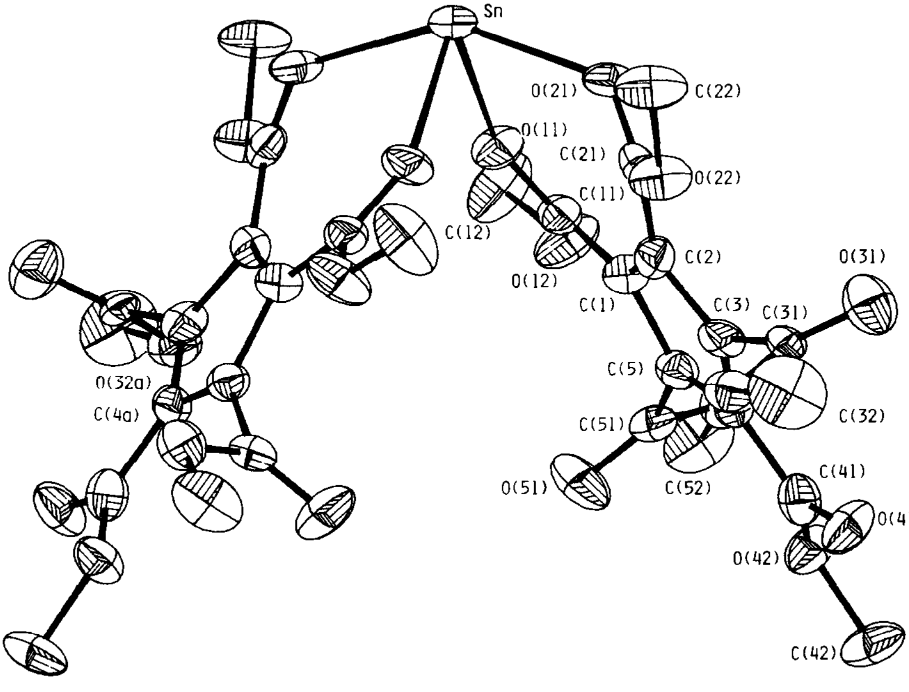


Fig. 1. A single molecule of $Sn[C_5(COOMe)_5]_2$ (6), showing atom numbering scheme. Atoms labelled (a) are related to the parent atom by the two-fold axis which passes through the tin atom.

TABLE 1

BOND LENGTHS (Å) IN **6**

Sn–O (11)	2.240(9)	Sn–O(11a)	2.240(9)
Sn–O(21)	2.271(9)	Sn–O(21a)	2.271(9)
Sn–O(51b)	2.925(10)	Sn–O(51c)	2.925(10)
C(2)–C(1)	1.448(14)	C(5)–C(1)	1.408(15)
C(11)–C(1)	1.441(17)	C(3)–C(2)	1.390(15)
C(21)–C(3)	1.436(16)	C(4)–C(3)	1.410(15)
C(31)–C(3)	1.515(17)	C(5)–C(4)	1.398(15)
C(41)–C(4)	1.434(16)	C(51)–C(5)	1.498(15)
O(11)–C(11)	1.212(13)	O(12)–C(11)	1.323(13)
O(12)–C(12)	1.455(13)	O(21)–C(21)	1.242(13)
O(22)–C(21)	1.321(12)	O(22)–C(22)	1.458(13)
O(31)–C(31)	1.172(14)	O(32)–C(31)	1.355(14)
O(32)–C(32)	1.451(14)	O(41)–C(41)	1.231(13)
O(42)–C(41)	1.347(13)	O(42)–C(42)	1.446(15)
O(51)–C(51)	1.204(14)	O(52)–C(51)	1.321(12)
O(52)–C(52)	1.465(15)		

Key to symmetry operations relating designated atoms to reference atoms at (x,y,z):
(a) $1.0-x,\ 1.0-y, z$
(b) $x, y, -1.0+z$
(c) $1.0-x, 1.0-y, -1.0+z$

TABLE 2

BOND ANGLES (deg.) IN **6**

O(21)–Sn–O(11)	77.2(3)	O(21)–Sn–O(11a)	77.1(3)
O(11)–Sn–O(11a)	79.4(3)	O(21)–Sn–O(21a)	146.4(2)
O(11)–Sn–O(51b)	69.1(3)	O(11a)–Sn–O(51b)	146.1(2)
O(21)–Sn–O(51b)	83.8(3)	O(21a)–Sn–O(51b)	106.6(3)
O(51b)–Sn–O(51c)	144.2(2)		
C(5)–C(1)–C(2)	107.0(9)	C(11)–C(1)–C(2)	128.8(10)
C(11)–C(1)–C(5)	124.2(10)	C(3)–C(2)–C(1)	106.9(10)
C(21)–C(2)–C(1)	127.9(11)	C(21)–C(2)–C(3)	124.2(10)
C(4)–C(3)–C(2)	109.7(10)	C(31)–C(3)–C(2)	125.9(11)
C(31)–C(3)–C(4)	123.8(11)	C(5)–C(4)–C(3)	107.4(10)
C(41)–C(4)–C(3)	123.2(11)	C(41)–C(4)–C(5)	129.3(11)
C(4)–C(5)–C(1)	109.1(10)	C(51)–C(5)–C(1)	123.9(10)
C(51)–C(5)–C(4)	126.2(11)	O(11)–C(11)–C(1)	128.7(12)
O(12)–C(11)–C(1)	111.8(10)	O(12)–C(11)–O(11)	119.5(13)
C(11)–O(11)–Sn	147.9(9)	C(12)–O(12)–C(11)	117.9(11)
O(21)–C(21)–C(2)	126.3(10)	O(22)–C(21)–C(2)	112.9(11)
O(22)–C(21)–O(21)	120.8(10)	C(21)–O(21)–Sn	132.5(6)
C(22)–O(22)–C(21)	116.9(10)	O(31)–C(31)–C(3)	124.6(13)
O(32)–C(31)–C(3)	110.1(11)	O(32)–C(31)–O(31)	125.0(14)
C(32)–O(32)–C(31)	112.2(12)	O(41)–C(41)–C(4)	124.4(12)
O(42)–C(41)–C(4)	114.4(10)	O(42)–C(41)–O(41)	121.1(11)
C(42)–O(52)–C(41)	116.5(9)	O(51)–C(51)–C(5)	124.4(10)
O(52)–C(51)–C(5)	111.8(10)	O(52)–C(51)–O(51)	123.8(11)
C(52)–O(52)–C(51)	116.2(11)		

indications that the five-fold symmetry of the cyclopentadienyl ring is slightly disturbed by the effect of the coordination, since the bond C(1)–C(2), which joins the two coordinating functions, is marginally longer than the other four ring bonds. However, the differences are on the borderline of significance, and must not be given too much emphasis.

The molecules **5** and **6** must be fluxional in solution; they therefore resemble the complex $(COOMe)_5C_5Ag(PPh_3)_2$ [7]. In the low temperature NMR spectra only one sharp resonance is found for each of the four sets of magnetically active nuclei (CH_3, ring C, CO and CH_3).

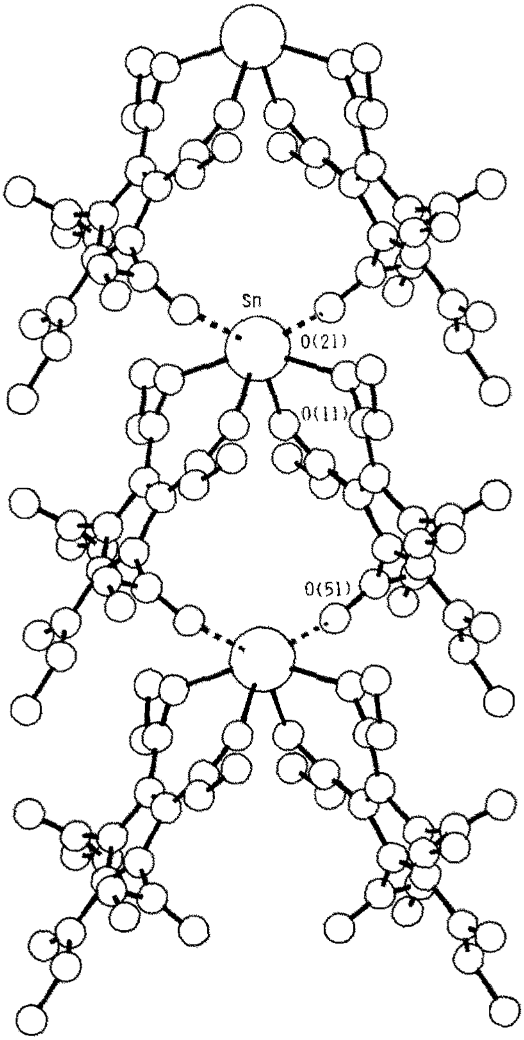


Fig. 2. A stack of three molecules **6** along the crystallographic *c*-axis, showing the intermolecular Sn ··· O interactions.

Experimental

All reactions were carried out under an inert atmosphere with exclusion of moisture. C,H analyses: Perkin–Elmer 240; melting points: Büchi 510, Mettler FP 61; mass spectra: Varian 311 A (70 eV, 300 μA, 200 °C (**3**, **4**), 260 °C (**5**, **6**); NMR spectra: Varian EM 360 L, Bruker AM 300; chemical shifts (δ) in ppm.

Pentamethylcyclopentadienylgermanium-penta(methoxycarbonyl)cyclopentadienide (3)

A solution of 0.97 g (2.83 mmol) **1** in 8 ml benzene was treated with 1.00 g (2.83 mmol) penta(methoxycarbonyl)cyclopentadiene. Within a few minutes the clear solution changed to a colourless suspension. After 24 h of stirring the precipitate was filtered off. Recrystallization from acetonitrile yielded the salt **3** as colourless cubes.

Compound **3**: m.p. 142 °C (decomp.) yield 0.2 g (12%); Found: C, 52.99; H, 5.82. $C_{25}H_{30}GeO_{10}$ (562.9) calcd.: C, 53.30; H, 5.33%. MS 564 (M^+, 0.2%); Mol. weight determination (cryoscopy in nitrobenzene): 269; ^{1}H NMR (CD_3CN, TMS): δ (CH_3) 1.91 (s, 15H); δ (OCH_3) 3.59 (s, 15H); ^{13}C NMR (CD_3CN, TMS): δ (CH_3) 12.05; δ (OCH_3) 56.32; δ (C ring) 121.43, 124.07; δ (C=O) 167.98.

Pentamethylcyclopentadienyltin-penta(methoxycarbonyl)cyclopentadienide (4)

A procedure similar to that used for **3** was employed, starting from 1.31 g (3.36 mmol) **2** in 8 ml benzene and 1.20 g (3.36 mmol) penta(methoxycarbonyl)cyclopentadiene. Recrystallization from methylene chloride yielded the salt **4** as colourless rhombs. Compound **4**: m.p. 135 °C (decomp.); yield 0.77 g (38%); Found: C, 49.25; H, 4.83. $C_{25}H_{30}O_{10}$ Sn (609.2) calcd.: C, 49.29; H, 4.96%, MS 610 (M^+, 0.8%); Mol. weight determination (cryoscopy in nitrobenzene): 367; ^{1}H NMR ($CDCl_3$, TMS): δ (CH_3) 2.07 (s, 15H), δ (OCH_3) 3.75 (s, 15H). ^{13}C NMR ($CDCl_3$, TMS): δ (CH_3) 10.95; δ (OCH_3) 55.06, δ (C ring) 120.92, 122.49; δ(C=O) 169.24. ^{119}Sn NMR ($CDCl_3$, $(CH_3)_4Sn$): δ −2185 (s).

Bis[penta(methoxycarbonyl)cyclopentadienyl]germanium (5)

A mixture of 0.28 g (2.40 mmol) **1** and 1.71 g (4.80 mmol) penta(methoxycarbonyl)cyclopentadiene in 40 ml of methylene chloride was stirred until a clear solution was formed. After a few weeks the solution changed in colour from yellow to brown. After 3 months compound **5** crystallized out as colourless needles.

Compound **5**: m.p. 166 °C (decomp.); yield 0.26 g (14%); Found: C, 45.21; H, 4.20. $C_{30}H_{30}O_{20}Ge$ (782.9) calcd.: C, 45.98; H, 3.83%. MS: 429 ($M^+ - C_5(COOMe)_5$, 1.5%), 324($C_5(COOMe)_5 -$ OMe, 25%), 293 ($C_5(COOMe)_5 -$ 2OMe, 100%); ^{1}H NMR (CD_3CN, TMS): δ (CH_3) 3.59 (s); ^{13}C NMR (CD_3CN, TMS): δ (CH_3) 52.88; δ (C ring) 122.62; δ (C=O) 169.22.

Bis[penta(methoxycarbonyl)cyclopentadienyl]tin (6)

A procedure similar to that used for **5** was employed, starting from 0.94 g (2.34 mmol) of **2** and 1.72 g (4.83 mmol) penta(methoxycarbonyl)cyclopentadiene in 15 ml methylene chloride; after two days the colour of the solution changed from yellow to green. Compound **6** crystallized out within seven days, and was recrystallized from acetonitrile to give colourless cubic crystals.

Compound **6**: m.p. 205 °C (decomp.); yield 1.22 g (43%); Found: C, 43.22; H, 3.46. $C_{30}H_{30}O_{20}Sn$ (828.7) calcd.: C, 43.45; H, 3.65%. MS: 475 ($M^+ - C_5(COOMe)_5$, 1.0%), 324 ($C_5(COOMe)_5 - OMe$, 20%), 293 ($C_5(COOMe)_5 - 2OMe$, 100%); ^1H NMR (CD_3CN, TMS): δ (CH_3) 3.73 (s); ^{13}C NMR (CD_3CN, TMS): δ (CH_3) 53.17; δ(C ring) 120.27; δ (C=O) 169.22; ^{119}Sn NMR (CD_3CN, $(CH_3)_4Sn$): δ -1739 (s).

Crystal data

$(C_{15}H_{15}O_{10})_2Sn$, $M = 828.7$, orthorhombic, a 15.656(2), b 15.424(2), c 7.033(1) Å; V 1698.3 Å^3; space group $P2_12_12$; $Z = 2$; D_c 1.62 g cm^{-3}; $F(000) = 840$, λ 0.71069 Å, μ(Mo-K_α) 7.48 cm^{-1}.

Data collection

Unit cell parameters were determined, and intensity data collected, on an Enraf-Nonius CAD 4 diffractometer using graphite-monochromated Mo-K_α radiation and an ω-2θ scan procedure [14]. 1473 data ($1.5 \leqslant \theta \leqslant 23°$) were measured, of which 1385 were unique and 1098 were considered observed ($I \geqslant 1.5\sigma(I)$).

Structure solution and refinement

The heavy-atom method was used to solve the structure and an absorption correction using the DIFABS [15] method was applied to the data after refinement with isotropic thermal parameters for all the non-hydrogen atoms. Difference Fouriers obtained after refinement with anisotropic temperature factors for all atoms showed some, but not all, of the methyl hydrogen atoms. Consequently all hydrogen

TABLE 3

FRACTIONAL ATOMIC COORDINATES ($\times 10^4$)

Atom	*x*	*y*	*z*
Sn	5000	5000	296(1)
C(1)	3838(6)	4078(6)	5019(15)
C(2)	4400(6)	3369(6)	4515(16)
C(3)	4321(6)	2749(7)	5938(14)
C(4)	3754(6)	3049(7)	7349(15)
C(5)	3451(6)	3858(6)	6760(15)
C(11)	3694(6)	4882(10)	4023(14)
C(12)	2792(8)	6121(8)	3812(22)
O(11)	4108(4)	5201(4)	2747(10)
O(12)	3006(4)	5289(4)	4658(12)
C(21)	4830(6)	3226(6)	2738(15)
C(22)	5823(8)	2359(9)	1050(17)
O(21)	4702(4)	3624(4)	1230(10)
O(22)	5391(4)	2586(5)	2818(11)
C(31)	4673(9)	1834(8)	5872(17)
C(32)	5855(10)	960(8)	6493(26)
O(31)	4334(5)	1247(5)	5130(15)
O(32)	5465(6)	1811(6)	6646(13)
C(41)	3530(7)	2551(7)	8997(17)
C(42)	2688(7)	2446(9)	11797(18)
O(41)	3837(5)	1839(5)	9394(12)
O(42)	2915(4)	2909(4)	10082(12)
C(51)	2928(6)	4474(7)	7924(14)
C(52)	1536(7)	5005(12)	8521(16)
O(51)	3222(4)	4997(8)	9016(10)
O(52)	2101(5)	4358(5)	7659(12)

atoms were included in the refinement with the application of geometric constraints and a single isotropic temperature factor for each group of methyl hydrogens. The full-matrix least-squares refinements, using unit weights, gave final R factors of $R = \Sigma|\Delta F|\Sigma|F_o| = 0.037$ and $R_w = [\Sigma|\Delta F|^2\Sigma|F_o|^2]^{1/2} = 0.041$. All computations were made using the SHELX-76 [16] program on a DEC VAX 11/750 computer. Final atomic coordinates are given in Table 3. Tables of thermal parameters, hydrogen atom coordinates and F_o/F_c values can be obtained from the authors at Queen Mary College.

Acknowledgement

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