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THE STRUCTURE OF DI-μ-PENTAFLUOROBENZOATOBIS-[BIS(PENTAFLUOROPHENYL)(TRIPHENYLPHOSPHINE OXIDE)THALLIUM(III)]

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Summary

An X-ray crystallographic study has shown that the complex $(C_6H_5)_2TlO_2C$-$C_6F_5(OPPh_3)$ has a dimeric structure with unsymmetrical pentafluorobenzoate bridging (Tl—O 2.531 and 2.789 Å) but an exact crystallographic centre of symmetry. The pentafluorobenzoate groups are also unsymmetrically chelated to thallium (Tl—O 2.389 and 2.531 Å), which overall has irregular six coordination.

Introduction

Recent crystal structures of two halogenobis(polyfluorophenyl)thallium(III) compounds, viz. $(p\text{-}HC_6F_4)_2TlBr$ [1] and $(p\text{-}HC_6F_4)_2TlCl(OPPh_3)$ [2], have led to a complete revision [2,3] of earlier structural proposals [4,5] for halogenobis(pentafluorophenyl)thallium(III) complexes. For example, the complexes $(C_6F_5)_2TlX(L)$ (X = Br or Cl; L = Ph_3PO or 2,2′-bipyridyl (bpy)) do not have the monomeric structures with four (L = Ph_3PO) or five (L = bpy) coordinate thallium originally suggested [4,5] but are dimeric with five (L = Ph_3PO) or six (L = bpy) coordinate thallium [2,3]. A wide range of carboxylatobis(pentafluorophenyl)thallium(III) complexes has also been prepared [4—6], and it is likely that their proposed structures also need revision. Indeed, it has recently been suggested [7] that $(C_6F_5)_2TlO_2CC_6F_5$ may not be dimeric and four coordinate as initially proposed [6], but may be a six coordinate polymer similar to dimethylthallium(III) acetate [8]. We now report the crystal structure of the complex $(C_6F_5)_2TlO_2CC_6F_5(OPPh_3)$. The results are not only relevant to complexes of this stoichiometric class (four coordinate monomeric structures have been proposed for analogous trifluoroacetato complexes [5]), but also provide a basis for inferring the structure of $(C_6F_5)_2TlO_2CC_6F_5$, which cannot be obtained in a form suitable for crystallographic examination.

Experimental

To a solution of pentafluorobenzoatobis(pentafluorophenyl)thallium(III) (0.45 mmol) and triphenylphosphine oxide (0.90 mmol) in ether (10 ml) was added 20 ml of boiling petroleum ether (b.p. <40°C). After cooling and standing for several hours, large transparent crystals formed. They were collected after 2 days, contained 67% by mass of the reactants, and analysed as $(C_6F_5)_2TlO_2CC_6F_5(OPPh_3)_2$, m.p. 143—180°C (Found: C, 50.4; H, 2.4; F, 21.3. $C_{55}H_{30}F_{15}O_4P_2Tl$ calcd.: C, 50.6; H, 2.3; F, 21.8%). The infrared spectrum and X-ray powder photograph were identical with those of an equimolar mixture of authentic $(C_6F_5)_2TlO_2CC_6F_5(OPPh_3)$ [9] and triphenylphosphine oxide. Microscopic examination and hand picking achieved separation into large rectangular prisms of the former, one of which was used for the X-ray study, and thin plates of the latter (infrared identification). Separation could also be achieved by flotation in carbon tetrachloride (density 1.59 g/ml, cf. Ph_3PO, 1.21 g/ml, $(C_6F_5)_2TlO_2CC_6F_5(OPPh_3)$, 1.84 g/ml).

Crystal structure determination

Crystals of $C_{74}H_{30}F_{30}O_6P_2Tl_2$ are triclinic, space group $C\bar{1}$, a 24.305(4), b 12.802(3), c 12.543(4) Å, α 88.99(3), β 107.50(2), γ 93.27(3)°, V 3716.1 Å^3, Z 2, D_c 1.84 g cm^{-3}, Mo-K_α radiation, μ = 43.5 cm^{-1}. Dimensions for the standard space group $P\bar{1}$ are a = 13.408(4), b = 12.543(4), c = 12.802(3) Å, α = 91.01(3), β = 115.19(2), γ = 105.31(3)°.

A Philips PW1100 diffractometer with a θ—2θ scan mode was used for data collection and reflections with $3.0 < \theta < 27.0°$ were examined. Weak reflections which gave $I_t - 2(I_t)^{1/2} < I_b$ on the first scan were not further examined (I_t is the count rate at the top of the reflection peak and I_b is the mean count rate from two preliminary 5 s background measurements on either side). Of the remaining reflections, those for which the total intensity recorded in the first scan (I_i) was <300 counts were scanned twice to increase their accuracy. A constant scan speed of 0.05° s^{-1} and a scan width of 0.70° were used, with a background measuring time proportional to I_b/I_i. Three standard reflections were measured every 5 h during data collection and showed no significant variations in intensity. The intensities were calculated from the peak and background measurements with a program written for the PW1100 diffractometer [10]. The variance of intensity, I, was calculated as $[\{\sigma_c(I)\}^2 + (0.04I)^2]^{1/2}$, where $\{\sigma_c(I)\}$ is the variance due to counting statistics, and the term in I^2 was introduced to allow for other sources of error [11]. I and $\sigma(I)$ were corrected for Lorentz and polarisation factors. Absorption corrections were applied with an empirical psi-scan technique on 29 reflections using the program X: EMPABS [12a]. The final data set consisted of 3724 independent reflections of which 121 for which $I < 3\sigma(I)$ were rejected. The structure was solved by standard Patterson and Fourier methods and refined by blocked-matrix least-squares cal-

TABLE 1

ATOMIC FRACTIONAL COORDINATES (Tl × 10^5, OTHERS × 10^4) FOR THE COMPOUND $[(C_6F_5)_2TlO_2CC_6F_5(OPPh_3)]_2$ (e.s.d.'s are given in parentheses)

Atom	x	y	z
Tl	3721(2)	11260(3)	13324(3)
P(1)	874(1)	3576)2)	3010(2)
O(31)	−431(3)	2195(5)	601(6)
O(32)	−460(3)	815(5)	−435(6)
O(1)	730(3)	2454(5)	2704(5)
C(37)	−666(5)	1631(9)	−231(9)
C(11)	949(5)	1553(7)	426(6)
C(12)	1483(6)	1092(9)	724(9)
C(13)	1891(5)	1240(10)	183(11)
C(14)	1778(6)	1871(11)	−715(11)
C(15)	1274(6)	2372(10)	−1060(10)
C(16)	878(5)	2186(9)	−476(10)
C(111)	1459(5)	3648(9)	4332(9)
C(112)	1767(7)	4592(13)	4683(15)
C(113)	2215(8)	4493(15)	5824(16)
C(114)	2273(9)	3625(18)	6284(18)
C(115)	2025(8)	2670(16)	5957(17)
C(116)	1574(6)	2707(11)	4891(13)
C(121)	1101(5)	4299(8)	1975(9)
C(122)	1659(7)	4272(11)	2005(13)
C(123)	1850(8)	4822(13)	1140(15)
C(124)	1453(7)	5259(11)	303(13)
C(125)	918(8)	5324(12)	283(14)
C(126)	720(6)	4839(11)	1182(12)
C(131)	262(5)	4189(9)	3197(9)
C(132)	−238(6)	3568(10)	3041(10)
C(133)	−744(7)	4061(13)	3147(13)
C(134)	−668(7)	5052(13)	3448(13)
C(135)	−204(7)	5694(13)	3648(13)
C(136)	306(6)	5248(11)	3466(12)
C(21)	128(6)	203(10)	2544(10)
C(22)	−421(7)	−3(9)	2573(10)
C(23)	−567(8)	−653(12)	3365(14)
C(24)	−133(12)	−1101(12)	4127(14)
C(25)	416(9)	−901(13)	4160(14)
C(26)	546(8)	−262(10)	3360(11)
C(31)	−1234(5)	1935(8)	−1042(10)
C(32)	−1729(7)	1822(10)	−765(13)
C(33)	−2262(8)	2029(15)	−1503(18)
C(34)	−2285(8)	2360(14)	−2531(20)
C(35)	−1790(8)	2514(10)	−2841(12)
C(36)	−1273(6)	2272(9)	−2082(11)
F(12)	1610(3)	446(5)	1634(5)
F(13)	2390(3)	764(6)	522(6)
F(14)	2160(3)	2033(7)	−1287(6)
F(15)	1157(3)	2982(6)	−1952(6)
F(16)	379(3)	2691(5)	−840(6)
F(22)	−874(4)	386(7)	1778(7)
F(23)	−1131(5)	−813(7)	3327(7)
F(24)	−283(4)	−1706(6)	4913(7)
F(25)	826(5)	−1347(8)	4963(8)
F(26)	1092(4)	−111(6)	3398(7)
F(32)	−1732(4)	1435(8)	234(8)
F(33)	−2753(4)	1888(9)	−1243(10)
F(34)	−2787(4)	2614(8)	−3295(9)
F(35)	−1812(4)	2850(8)	−3862(8)
F(36)	−797(4)	2405(6)	−2410(6)

(Table 1 continued)

Atom	*x*	*y*	*z*
H(112)	1695(7)	5311(13)	4209(15)
H(113)	2481(8)	5165(15)	6222(16)
H(114)	2581(9)	3634(18)	7110(18)
H(115)	2146(8)	1960(16)	6426(17)
H(116)	1328(6)	2000(11)	4538(13)
H(122)	1959(7)	3852(11)	2661(13)
H(123)	2300(8)	4871(13)	1176(15)
H(124)	1581(7)	5579(11)	—395(13)
H(125)	620(8)	5729(12)	—390(14)
H(126)	284(6)	4914(11)	1207(12)
H(132)	—253(6)	2747(10)	2846(10)
H(133)	—1158(7)	3637(13)	2990(13)
H(134)	—1045(7)	5400(13)	3542(13)
H(135)	—198(7)	6489(13)	3925(13)
H(136)	698(6)	5722(11)	3537(12)

TABLE 2

BOND LENGTHS (Å) AND ANGLES (°) FOR THE COMPOUND $[(C_6F_5)_2TlO_2CC_6F_5(OPPh_3)]_2$ (with e.s.d.'s in parentheses) [a]

a) Bond lengths

Tl—O(31)	2.389(7)	C(24)—F(24)	1.365(23)
Tl—O(32)	2.531(6)	C(25)—F(25)	1.333(20)
Tl—O(1)	2.375(6)	C(26)—F(26)	1.317(22)
Tl—C(11)	2.099(12)	C(31)—C(32)	1.349(22)
Tl—C(21)	2.111(14)	C(32)—C(33)	1.384(22)
Tl—O(32)^{i}	2.791(6)	C(33)—C(34)	1.336(34)
Tl...Tl^{i}	4.343	C(34)—C(35)	1.374(32)
P(1)—O(1)	1.488(7)	C(35)—C(36)	1.377(21)
C(37)—O(31)	1.249(12)	C(36)—C(31)	1.344(19)
C(37)—O(32)	1.247(14)	C(32)—F(32)	1.340(19)
C(37)—C(31)	1.515(14)	C(33)—F(33)	1.330(25)
P(1)—C(111)	1.830(10)	C(34)—F(34)	1.359(21)
P(1)—C(121)	1.782(13)	C(35)—F(35)	1.331(18)
P(1)—C(131)	1.800(13)	C(36)—F(36)	1.340(19)
C(11)—C(12)	1.399(17)	C(111)—C(112)	1.389(19)
C(12)—C(13)	1.364(21)	C(112)—C(113)	1.524(23)
C(13)—C(14)	1.343(19)	C(113)—C(114)	1.240(30)
C(14)—C(15)	1.365(20)	C(114)—C(115)	1.345(29)
C(15)—C(16)	1.381(22)	C(115)—C(116)	1.454(22)
C(16)—C(11)	1.354(16)	C(116)—C(111)	1.383(18)
C(12)—F(12)	1.365(13)	C(121)—C(122)	1.349(21)
C(13)—F(13)	1.337(14)	C(122)—C(123)	1.458(26)
C(14)—F(14)	1.340(18)	C(123)—C(124)	1.333(21)
C(15)—F(15)	1.322(14)	C(124)—C(125)	1.300(27)
C(16)—F(16)	1.358(14)	C(125)—C(126)	1.468(26)
C(21)—C(22)	1.358(23)	C(126)—C(121)	1.348(17)
C(22)—C(23)	1.398(23)	C(131)—C(132)	1.377(18)
C(23)—C(24)	1.341(26)	C(132)—C(133)	1.455(24)
C(24)—C(25)	1.330(37)	C(133)—C(134)	1.317(24)
C(25)—C(26)	1.382(24)	C(134)—C(135)	1.318(24)
C(26)—C(21)	1.361(18)	C(135)—C(136)	1.475(25)
C(22)—F(22)	1.356(15)	C(136)—C(131)	1.393(18)
C(23)—F(23)	1.361(23)		

(Table 2 continued)

b) Inter-bond angles

O(1)—Tl—O(32)^{i}	151.0(2)	F(22)—C(22)—C(21)	120.7(12)
P(1)—O(1)—Tl	150.5(4)	F(22)—C(22)—C(23)	115.4(14)
C(37)—O(31)—Tl	95.1(6)	F(23)—C(23)—C(22)	119.6(14)
C(37)—O(32)—Tl	88.5(5)	F(23)—C(23)—C(24)	123.3(18)
O(31)—C(37)—O(32)	123.4(9)	F(24)—C(24)—C(23)	116.1(23)
O(31)—C(37)—C(31)	119.3(10)	F(24)—C(24)—C(25)	122.1(17)
O(32)—C(37)—C(31)	117.4(8)	F(25)—C(25)—C(24)	118.3(17)
C(11)—Tl—C(21)	151.6(5)	F(25)—C(25)—C(26)	121.7(19)
C(11)—Tl—C(31)	105.1(3)	F(26)—C(26)—C(21)	119.9(14)
C(11)—Tl—O(32)	92.2(3)	F(26)—C(26)—C(25)	118.1(14)
C(11)—Tl—O(1)	94.8(3)	C(37)—C(31)—C(32)	120.8(12)
C(11)—Tl—O(32)^{i}	80.2(3)	C(37)—C(31)—C(36)	121.8(12)
C(21)—Tl—O(31)	103.3(4)	C(32)—C(31)—C(36)	117.3(12)
C(21)—Tl—O(32)	103.9(4)	C(31)—C(32)—C(33)	122.7(16)
C(21)—Tl—O(1)	88.6(4)	C(32)—C(33)—C(34)	118.4(19)
C(21)—Tl—O(32)^{i}	83.3(3)	C(33)—C(34)—C(35)	120.8(17)
O(31)—Tl—O(32)	53.0(2)	C(34)—C(35)—C(36)	118.3(16)
O(31)—Tl—O(1)	85.6	C(35)—C(36)—C(31)	122.3(15)
O(31)—Tl—O(32)^{i}	123.4(2)	F(32)—C(32)—C(31)	120.9(12)
O(32)—Tl—O(1)	138.3(2)	F(32)—C(32)—C(33)	116.3(16)
O(32)—Tl—O(32)^{i}	70.7(2)	F(33)—C(33)—C(32)	122.8(19)
O(1)—P(1)—C(111)	108.3(4)	F(33)—C(33)—C(34)	118.7(16)
O(1)—P(1)—C(121)	112.3(5)	F(34)—C(34)—C(33)	122.3(20)
O(1)—P(1)—C(131)	111.0(5)	F(34)—C(34)—C(35)	116.8(20)
C(111)—P(1)—C(121)	108.9(5)	F(35)—C(35)—C(34)	120.9(18)
C(111)—P(1)—C(131)	108.5(5)	F(35)—C(35)—C(36)	120.7(16)
C(121)—P(1)—C(131)	107.7(6)	F(36)—C(36)—C(35)	117.4(13)
Tl—C(11)—C(12)	118.0(8)	F(36)—C(36)—C(31)	120.3(11)
Tl—C(11)—C(16)	129.8(9)	P(1)—C(111)—C(112)	120.2(9)
C(12)—C(11)—C(16)	112.1(11)	P(1)—C(111)—C(116)	115.0(8)
C(11)—C(12)—C(13)	125.2(11)	C(112)—C(111)—C(116)	124.8(11)
C(12)—C(13)—C(14)	118.4(12)	C(111)—C(112)—C(113)	111.8(14)
C(13)—C(14)—C(15)	120.7(15)	C(112)—C(113)—C(114)	118.9(17)
C(14)—C(15)—C(16)	117.9(12)	C(113)—C(114)—C(115)	132.4(19)
C(15)—C(16)—C(11)	125.6(11)	C(114)—C(115)—C(116)	111.9(17)
F(12)—C(12)—C(11)	117.8(12)	C(115)—C(116)—C(111)	120.0(13)
F(12)—C(12)—C(13)	116.9(11)	P(1)—C(121)—C(122)	117.8(9)
F(13)—C(13)—C(12)	121.4(11)	P(1)—C(121)—C(126)	120.6(11)
F(13)—C(13)—C(14)	120.2(13)	C(122)—C(121)—C(126)	121.7(14)
F(14)—C(14)—C(13)	120.9(12)	C(121)—C(122)—C(123)	118.8(13)
F(14)—C(14)—C(15)	118.4(12)	C(122)—C(123)—C(124)	118.2(17)
F(15)—C(15)—C(14)	121.0(14)	C(123)—C(124)—C(125)	123.4(18)
F(15)—C(15)—C(16)	121.0(12)	C(124)—C(125)—C(126)	119.6(14)
F(16)—C(16)—C(11)	117.8(12)	C(125)—C(126)—C(121)	117.9(15)
F(16)—C(16)—C(15)	116.6(11)	P(1)—C(131)—C(132)	116.8(9)
Tl—C(21)—C(22)	125.6(9)	P(1)—C(131)—C(136)	119.8(10)
Tl—C(21)—C(26)	118.8(12)	C(132)—C(131)—C(136)	123.4(13)
C(22)—C(21)—C(26)	115.5(13)	C(131)—C(132)—C(133)	117.5(12)
C(21)—C(22)—C(23)	123.9(13)	C(132)—C(133)—C(134)	116.2(15)
C(22)—C(23)—C(24)	117.1(19)	C(133)—C(134)—C(135)	130.3(19)
C(23)—C(24)—C(25)	121.6(18)	C(134)—C(135)—C(136)	115.2(15)
C(24)—C(25)—C(26)	119.9(16)	C(135)—C(136)—C(131)	117.1(13)
C(25)—C(26)—C(21)	122.0(8)		

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[a] Atoms indicated with the superscript (i) are related by the transformation, $-x$, $-y$, $-z$.

culations using the SHELX program [12b]. The C—H hydrogen atom coordinates were estimated geometrically (C—H 1.08 Å) and in refinement were allowed to ride on their respective C atom coordinates. Neutral-atom scattering factors were used [13], those for Tl, P, O and F being corrected for anomalous dispersion ($\Delta f'$, $\Delta f''$). For the final stages of refinement the Tl, P, O and F atoms were assigned anisotropic thermal parameters. The final $R = \Sigma||F_0| - |F_c||/\Sigma|F_0| = 0.0448$ and $R_w = [\Sigma w(|F_0| - |F_c|)^2/\Sigma w|F_0|^2]^{1/2} = 0.0450$ where $w = 1.186\ (\sigma^2|F_0|)^{-1}$. The final atomic parameters are listed in Table 1 and bond lengths and angles are in Table 2.

Lists of structure factors, thermal parameters and equations of least-squares planes are available from the authors (KH).

Discussion

The molecular structure of the complex consists of two $(C_6F_5)_2TlO_2CC_6F_5(OPPh_3)$ units (Fig. 1) linked by unsymmetrical carboxylate bridges to give the discrete centrosymmetric dimers illustrated in Figure 2. The thallium atoms have irregular six coordination. The pentafluorobenzoate ligand is asymmetrically bidentate with Tl—O(31) 2.389(7) and Tl—O(32) 2.531(6) Å. The dimeric units, which have an exact crystallographic centre of symmetry, are formed by longer bonds from O(32) to Tl^i of 2.761(6) Å. There is no indication of further association, as all the shortest inter-dimer contacts involve the phenyl fluorine and hydrogen atoms.

The irregular geometry of the six donor atoms around thallium is in part due to the small bite of the carboxylate ligand [O(31)—Tl—O(32) 53.0(2)°] and in part to the large C(11)—Tl—C(21) angle [151.6(5)°] between the pentafluorophenyl groups. The latter is larger than the corresponding angles in $(C_6F_5)_2TlOH$ (139°) [14], $(p\text{-}HC_6F_4)_2TlBr$ (144 and 150°C) [1], $(p\text{-}HC_6F_4)_2$-

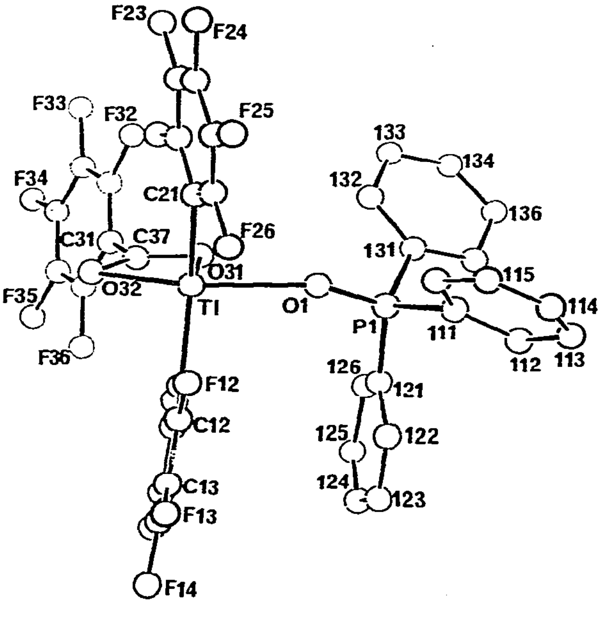

Fig. 1. The structure of the monomeric unit, $(C_6F_5)_2TlO_2CC_6F_5(OPPh_3)$. Bonded F and C atoms have the same numbers.

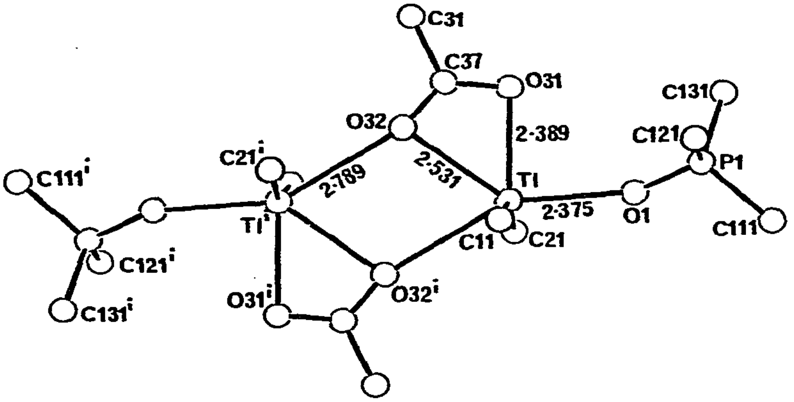

Fig. 2. The structure of the dimeric $[(C_6F_5)_2TlO_2CC_6F_5(OPPh_3)]_2$ molecule. The first C atoms only of the phenyl rings are shown.

$TlCl(OPPh_3)$ (141°) [2], and $(C_6F_5)_2Tldpa$ (dpa = 2,2'-dipyridylamide) (127°) [15]. The C—P—C angles in the triphenylphosphine oxide ligand are in the range 107.7 to 108.9°, and the Tl—$OPPh_3$ distance of 2.375(6) Å is shorter than the carboxylate Tl—O bonds (Table 2). Although the Tl—O(1)—P(1) angle [150.5(4)°] is very large (even with considerable $p\pi$(O)—$d\pi$(P) bonding, an angle of 120° would be expected), it is smaller than the corresponding angle (168.0°) of $(p\text{-}HC_6F_4)_2TlCl(OPPh_3)$ [2]. In the present case, the large angle can be attributed to steric factors, as there are close intramolecular contacts between the phenyl hydrogen atoms and the pentafluorophenyl fluorine atoms.

Superficially, the irregular six coordinate geometry of $[(C_6F_5)_2TlO_2CC_6F_5(OPPh_3)]_2$ bears little relationship to the structures of other bis(polyfluorophenyl)thallium(III) compounds [1,2,14,15], which are all five coordination site distorted trigonal bipyramidal stereochemistry. However, because of the small bite, a carboxylate ligand can be viewed as occupying only one coordinated site [16]. On this basis, the stereochemistry of $[(C_6F_5)_2TlO_2CC_6F_5(OPPh_3)]_2$ may be regarded as distorted trigonal bipyramidal with two carbon atoms and the carboxylate group in the equatorial plane.

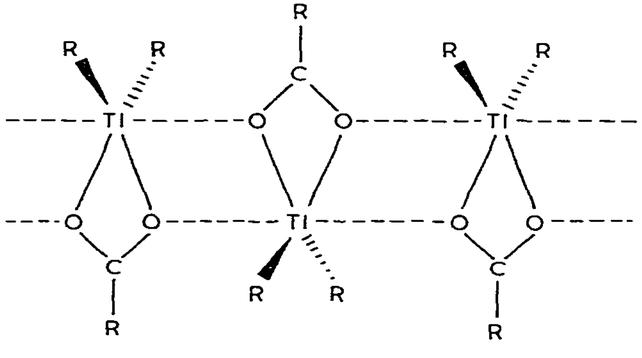

Fig. 3. The structure of some diorganothallium(III) carboxylates.

The present structure is closely related to that of dimethylthallium(III) acetate (Fig. 3, R = Me) [8], providing strong support for the proposal [7] that $(C_6F_5)_2TlO_2CC_6F_5$ has a similar polymeric structure (Fig. 3, R = C_6F_5). This can readily be converted into the structure of $[(C_6F_5)_2TlO_2CC_6F_5(OPPh_3)]_2$ by replacement of alternate pairs of carboxylate bridges by triphenylphosphine oxide.

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