Journal of Organometallic Chemistry, 417 (1991) 389–396
Elsevier Sequoia S.A., Lausanne
JOM 21957

The crystal structure and properties of di-μ-[bis(2-phenylethyl) *m*-phthalate]octachlorodititanium(IV) bis(dichloromethane)

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(Received April 7th, 1991)

Abstract

The dimeric complex $[Ti_2\{\mu\text{-m-}C_6H_4(CO_2CH_2CH_2Ph)_2\}_2Cl_8]$, is, in the presence of activators, a good catalyst for olefin polymerization, its crystal structure has been determined by X-ray diffraction methods and refined by block-diagonal least-squares to $R = 0.041$ for 3027 independent non-zero reflections. The crystals are monoclinic, space group $P2_1/c$ with $Z = 2$, in a unit cell of dimensions: $a = 13.145(9)$, $b = 10.816(8)$, $c = 21.530(14)$ Å, $\beta = 108.84(6)°$. The crystals consist of dimers possessing a crystallographic symmetry centre and dichloromethane molecules. Titanium atoms are octahedrally coordinated by four chlorine atoms and two carbonyl oxygen atoms of two bis(2-phenylethyl) *m*-phthalate in the *cis* position. Two ester ligands and two titanium atoms form a sixteen-membered ring.

Introduction

To understand the enhancement of the isospecific activity of aromatic esters with bulky substituents in the propylene polymerization process [1–6] the reactions of $TiCl_4$ with *m*-$C_6H_4(CO_2R)_2$ were studied.

Here we present the crystal structure of the complex $[Ti_2[\mu\text{-}m\text{-}C_6H_4(CO_2CH_2\text{-}CH_2Ph)_2\}_2Cl_8] \cdot 2CH_2Cl_2$ (I) and compare it with that of the previously described dimeric compound $[Ti_2\{\mu\text{-}m\text{-}C_6H_4(CO_2Et)_2\}_2Cl_8]$ (II) [7].

Experimental

All reactions were carried out under nitrogen in dried solvents by Schlenk-tube techniques. Anhydrous $TiCl_4$ was of commercial grade. Bis(2-phenylethyl) *m*-phthalate was obtained by a standard procedure viz. reaction of isophthalic acid with 2-phenylethyl alcohol [8]. IR spectra were recorded on a Perkin–Elmer 180 spectrometer.

Di-μ-[bis(2-phenylethyl) m-phthalate]octachlorodititanium(IV) bis(dichloromethane)

To 1.65 cm^3 of $TiCl_4$ (2.8 g; 15 mmol) was added 5.6 g (15 mmol) of *m*-$C_6H_4(CO_2CH_2CH_2Ph)_2$. The mixture was stirred into 100 cm^3 n-hexane under N_2.

0022-328X/91/$03.50

Table 1

Final atomic parameters with esd's in parentheses for $[Ti_2\{\mu\text{-}m\text{-}C_6H_4(CO_2CH_2CH_2Ph)_2\}_2Cl_8]\cdot 2CH_2Cl_2$

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Ti	0.34882(7)	0.19490(8)	0.12712(4)	3.78(4)	2.02(3)	2.60(3)	0.12(3)	1.66(3)	−0.06(3)
Cl1	0.39954(13)	0.39288(12)	0.13964(7)	6.77(9)	2.27(5)	5.54(7)	−0.66(6)	2.78(7)	−0.35(5)
Cl2	0.28092(11)	0.20756(13)	0.01420(6)	5.13(7)	4.01(6)	2.71(5)	0.46(6)	1.26(5)	0.54(5)
Cl3	0.18971(11)	0.22421(14)	0.14061(7)	4.26(7)	4.69(8)	4.99(7)	0.23(6)	2.64(6)	−0.71(6)
Cl4	0.43753(12)	0.14829(13)	0.23545(6)	5.07(7)	4.75(7)	2.52(5)	−0.14(6)	1.68(5)	0.30(5)
Cl5	0.90013(19)	0.09800(22)	0.43044(10)	10.28(15)	8.75(14)	7.07(11)	−0.38(12)	2.70(11)	−0.63(11)
Cl6	0.95000(20)	0.34895(23)	0.47855(11)	11.15(17)	8.26(14)	7.77(12)	−1.49(12)	0.72(11)	0.19(11)
O1	0.31603(28)	0.00640(28)	0.11377(15)	5.62(20)	2.11(14)	3.22(15)	0.19(14)	2.45(15)	−0.24(12)
O2	0.50340(26)	−0.14326(31)	−0.11350(14)	3.70(17)	3.67(16)	2.88(14)	0.54(14)	2.07(13)	0.24(13)
O3	0.29089(29)	−0.17906(29)	0.15141(15)	6.20(21)	2.49(16)	3.40(15)	0.13(15)	3.27(15)	0.15(12)
O4	0.37363(25)	−0.01842(29)	0.10581(14)	3.61(16)	3.07(15)	2.49(14)	0.67(13)	1.45(12)	0.71(12)
C1	0.3233(4)	−0.1062(5)	0.11299(20)	3.82(25)	2.35(20)	2.11(19)	−0.05(19)	1.29(18)	−0.08(17)
C2	0.4402(4)	−0.1101(5)	−0.08592(20)	2.98(23)	2.52(21)	1.88(18)	0.39(18)	0.66(17)	−0.09(16)
C3	0.2455(5)	−0.1257(5)	0.20030(23)	5.90(32)	3.76(27)	3.01(23)	1.07(24)	3.07(23)	0.45(20)
C4	0.3081(5)	−0.1789(6)	0.26487(26)	6.78(37)	5.78(36)	3.23(25)	2.35(30)	2.14(25)	−0.15(24)
C5	0.3720(5)	−0.0476(5)	0.16616(22)	4.29(28)	3.29(24)	2.58(21)	0.13(21)	1.23(20)	0.94(19)
C6	0.2754(5)	−0.1331(5)	0.18225(25)	4.53(29)	2.89(25)	3.98(25)	0.08(22)	1.01(22)	1.14(21)
C7	1.0020(8)	0.2010(9)	0.46859(50)	7.14(49)	8.37(58)	15.69(82)	0.72(45)	1.41(52)	2.54(57)
C11	0.3695(4)	0.1738(4)	0.06834(21)	3.19(23)	1.93(20)	2.30(19)	0.23(17)	1.25(17)	−0.11(15)
C12	0.4058(5)	−0.2952(5)	0.08216(22)	4.58(27)	2.42(20)	2.64(20)	0.17(21)	1.80(19)	0.48(18)
C13	0.4547(5)	−0.3542(5)	0.04207(25)	5.39(31)	2.00(21)	4.26(26)	0.90(21)	2.45(23)	0.75(20)

C14	0.4677(4)	−0.2920(5)	−0.01205(22)	4.33(28)	2.39(21)	2.84(21)	0.23(20)	1.97(20)	−0.09(18)
C15	0.4293(4)	−0.1732(4)	−0.02601(20)	2.71(22)	2.03(20)	2.18(19)	−0.19(17)	0.96(17)	0.01(15)
C16	0.3791(4)	−0.1118(5)	0.01359(20)	2.82(22)	2.21(20)	2.09(18)	−0.16(17)	0.93(16)	−0.14(16)
C21	0.2685(5)	−0.1321(5)	0.32029(22)	4.69(29)	4.17(28)	2.09(20)	1.28(23)	0.90(20)	0.13(20)
C22	0.3001(5)	−0.0183(6)	0.34765(25)	5.27(32)	4.41(29)	3.31(24)	−0.50(25)	1.93(23)	−0.26(22)
C23	0.2669(6)	0.0237(6)	0.39884(27)	7.06(38)	4.61(31)	3.52(26)	−0.11(28)	1.96(26)	−0.82(24)
C24	0.2024(6)	−0.0494(7)	0.42301(27)	7.11(39)	6.41(37)	3.68(27)	1.12(31)	3.09(27)	−0.59(27)
C25	0.1711(6)	−0.1629(7)	0.39579(31)	7.47(41)	6.33(38)	5.83(34)	−0.71(32)	4.37(32)	1.31(29)
C26	0.2040(6)	−0.2052(6)	0.34459(29)	6.68(37)	3.72(28)	5.11(30)	−0.67(28)	2.22(27)	−0.32(26)
C31	0.1701(5)	0.0666(5)	−0.19846(25)	3.88(27)	3.59(27)	4.00(25)	1.29(22)	0.95(21)	1.85(21)
C32	0.1197(5)	0.0456(6)	−0.15119(28)	5.00(32)	5.18(32)	4.60(28)	0.90(26)	1.87(25)	0.12(26)
C33	0.0249(5)	−0.0196(7)	−0.16550(30)	4.37(32)	6.75(39)	5.65(33)	0.27(29)	2.30(27)	0.43(29)
C34	−0.0215(5)	−0.0674(8)	−0.22707(33)	3.94(32)	8.18(47)	6.89(39)	−1.08(32)	1.09(28)	1.14(35)
C35	0.0279(6)	−0.0490(8)	−0.27487(28)	6.04(37)	8.74(48)	3.60(29)	−1.10(35)	0.22(27)	0.61(31)
C36	0.1222(5)	0.0174(6)	−0.26089(26)	5.31(33)	6.16(37)	3.43(27)	−1.11(29)	0.79(24)	0.75(25)

Atom	x	y	z	B_{iso}	Atom	x	y	z	B_{iso}	Atom	x	y	z	B_{iso}
H3	0.162	−0.151	0.188	6	H7	1.053	0.211	0.439	12	H24	0.177	−0.017	0.463	7
H31	0.254	−0.026	0.202	6	H71	1.047	0.165	0.516	12	H25	0.120	−0.220	0.414	7
H4	0.391	−0.153	0.276	6	H12	0.396	−0.343	0.124	5	H26	0.179	−0.295	0.324	7
H41	0.300	−0.278	0.262	6	H13	0.483	−0.448	0.053	5	H32	0.156	0.082	−0.102	7
H5	0.363	−0.017	−0.206	6	H14	0.507	−0.337	0.043	5	H33	−0.013	−0.034	−0.128	7
H51	0.445	0.101	−0.158	6	H16	0.349	−0.019	0.002	5	H34	−0.096	−0.119	−0.239	7
H6	0.274	0.189	−0.224	6	H22	0.351	0.039	0.329	7	H35	−0.008	−0.087	−0.323	7
H61	0.284	0.192	−0.140	6	H23	0.292	0.114	0.420	7	H36	0.159	0.032	−0.299	7

After 1 h the yellow precipitate was filtered off and washed with n-hexane (3×15 cm^3). Yield 7.8 g: 92%. A portion of the compound (4 g) was heated in 20 cm^3 CH_2Cl_2 under reflux and left to crystallize. The species usually precipitates as needles. Crystals suitable for the X-ray data were grown by slow diffusion of n-hexane into a solution of the compound in dichloromethane.

X-Ray crystal structure determination

Crystal data. $C_{48}H_{44}Cl_8O_8Ti_2 \cdot 2CH_2Cl_2$, $M = 1298.17$, $a = 13.145(9)$, $b = 10.816(8)$, $c = 21.530(14)$ Å, $\beta = 108.84(6)°$, $U = 2897(4)$ $Å^3$, $D_m = 1.52$, $Z = 2$, $D_c = 1.488(2)$ g cm^{-3}, $F(000) = 1320$, space group $P2_1/c$, Mo-K_α radiation, $\lambda = 0.71069$ Å, $\mu = 8.86$ cm^{-1}, $T = 303(2)$ K.

Preliminary Weissenberg photographs revealed the space group. A sample of dimensions $0.5 \times 0.5 \times 0.5$ mm was cut from a large crystal and sealed in a capillary. A Syntex $P2_1$ four-circle diffractometer was used. Cell parameters were obtained from a least-squares fit of 15 reflections in the range $17 < 2\theta < 24°$. The diffraction data were collected by $\theta/2\theta$ scan technique with graphite monochromatized Mo-K_α radiation. Of the 5408 reflections collected up to $2\theta = 52°$, 3027 with $I > 3\sigma(I)$ were used for the structure analysis. Two check reflections measured after each 50 reflections showed ±5% variation. The structure was solved by the heavy-atom technique and refined by block-diagonal least squares [9]. The H-atoms were included in geometrically calculated positions with $d(C-H) = 1.08$ Å. Neutral atom scattering factors were taken from ref. 10. The Ti, Cl, O and C scattering factors were corrected for real and imaginary components. The function minimized was $\sum w(|F_o| - |F_c|)^2$ where $w = 1/\sigma^2(F_o)$. The final R and R_w values were 0.041 and 0.045 for the observed reflections. For the last cycle of the refinement the maximal value of the Δ/σ ratio was 0.01 and the final difference map showed a general background within −0.31 and 0.63 e $Å^{-3}$. No absorption and extinction corrections were made.

The final atomic parameters are summarized in Table 1.

Results and discussion

The addition of bis(2-phenylethyl) *m*-phthalate to titanium tetrachloride, in a 1 : 1 ratio, in dichloromethane gave a yellow air-sensitive compound formulated as $[Ti_2\{\mu\text{-}m\text{-}C_6H_4(CO_2CH_2CH_2Ph)_2\}_2Cl_8] \cdot 2CH_2Cl_2$ (1).

$$2TiCl_4 + 2\ m\text{-}C_6H_4(CO_2CH_2CH_2Ph)_2 \xrightarrow{CH_2Cl_2}$$

$$\left[Cl_4Ti\{\mu\text{-}m\text{-}C_6H_4(CO_2CH_2CH_2Ph)_2\}_2TiCl_4\right] \cdot 2CH_2Cl_2$$

(1)

The new compound is diamagnetic. The IR spectrum shows stretching $\nu(C{=}O)$ modes at 1620vs, 1648vs and 1660sh cm^{-1}, and those of $\nu(Ti-Cl)$ at 306m, 370vs and 390sh cm^{-1}, and of the phenyl ring at 1582m and 1600m cm^{-1}.

The complex compound in the crystalline state is dimeric. The structure of di-μ-[bis(2-phenylethyl) *m*-phthalate]octachlorodititanium(IV) molecule is depicted in Fig. 1 and principal interatomic distances, bond angles, and torsion angles are listed in Table 2. In the complex molecule four Cl atoms and two O atoms from two bis(2-phenylethyl) *m*-phthalate ligands (in the *cis* position) form a distorted oc-

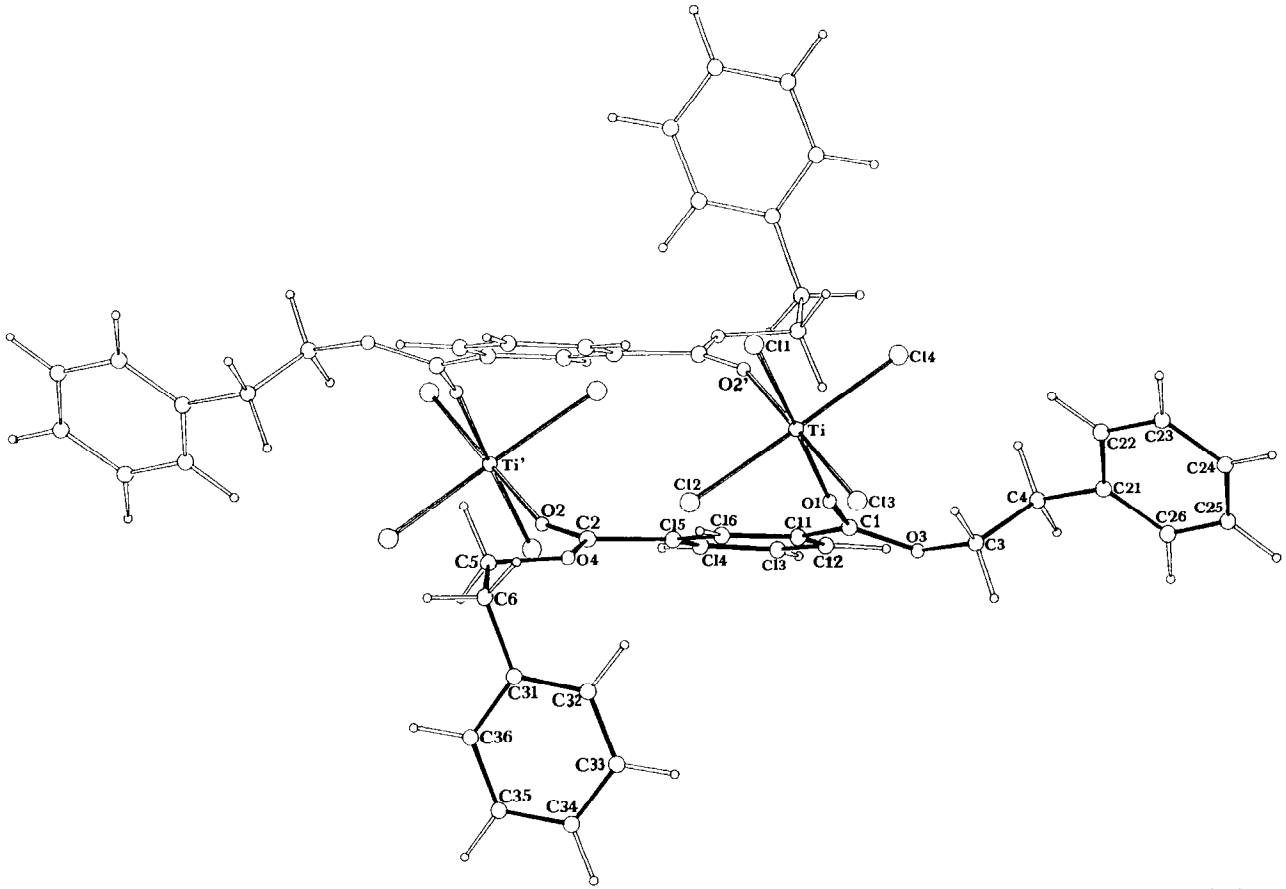

Fig. 1. View of $[Ti_2\{\mu\text{-}m\text{-}C_6H_4(CO_2CH_2CH_2Ph)_2\}_2Cl_8]$ molecule. Primed and unlabelled atoms are related to labelled atoms by the inversion centre.

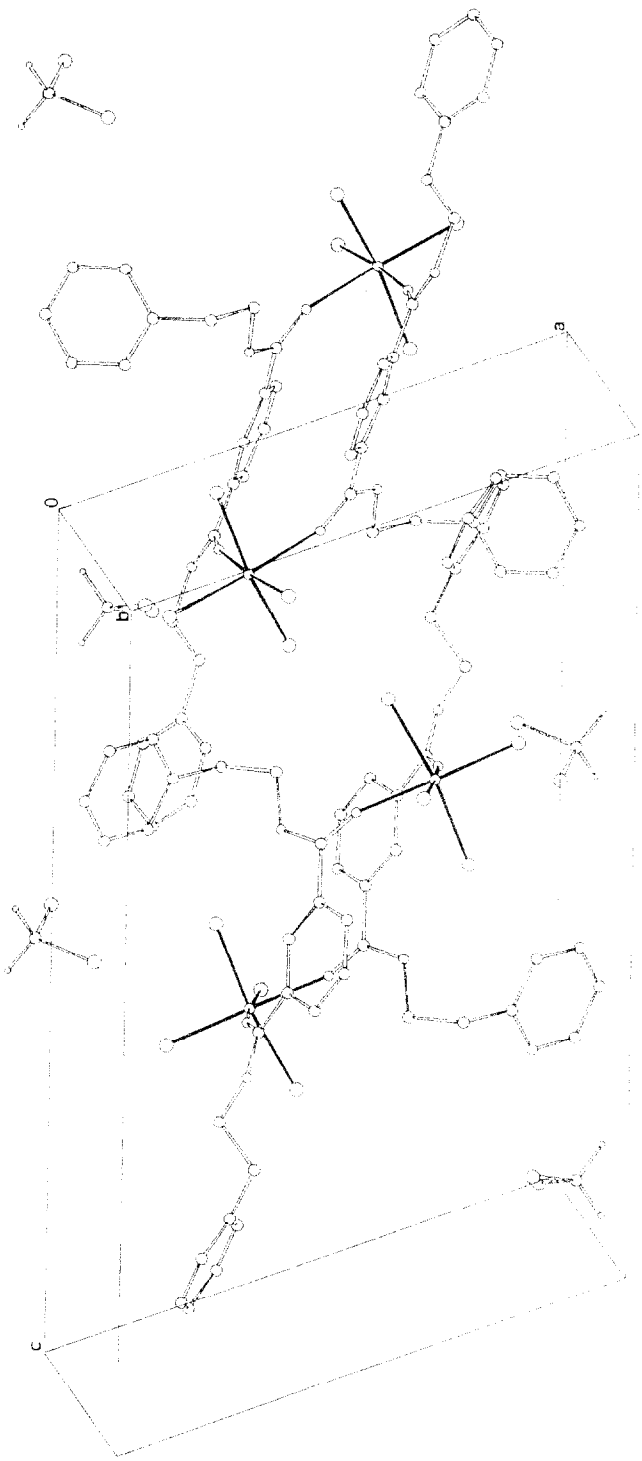

Fig. 2. The packing arrangement in the $[Tl_2\{\mu\text{-}m\text{-}C_6H_4(OCH_2C_6H_5Ph)_2\}_2(I_8)]\cdot 2CH_2Cl_2$ crystal

Table 2

Principal interatomic distances (Å), bond angles (°) and torsion angles (°) for $[Ti_2\{\mu\text{-}m\text{-}C_6H_4(CO_2CH_2CH_2Ph)_2\}_2Cl_8]\cdot 2CH_2Cl_2$ [a]

Ti–Cl(1)	2.233(2)	Ti–Cl(2)	2.307(2)
Ti–Cl(3)	2.225(2)	Ti–Cl(4)	2.302(2)
Ti–O(1)	2.085(3)	Ti–O(2^i)	2.131(4)
O(1)–C(1)	1.222(6)	O(2)–C(2)	1.220(6)
C(1)–O(3)	1.351(6)	O(4)–C(2)	1.350(6)
O(3)–C(3)	1.484(6)	O(4)–C(5)	1.477(6)
C(3)–C(4)	1.484(7)	C(5)–C(6)	1.518(8)
C(4)–C(21)	1.534(8)	C(6)–C(31)	1.497(8)
C(21)–C(22)	1.370(8)	C(31)–C(32)	1.400(8)
C(22)–C(23)	1.386(8)	C(32)–C(33)	1.377(9)
C(23)–C(24)	1.379(9)	C(33)–C(34)	1.369(9)
C(24)–C(25)	1.366(9)	C(34)–C(35)	1.398(9)
C(25)–C(26)	1.385(9)	C(35)–C(36)	1.378(10)
C(26)–C(21)	1.380(9)	C(36)–C(31)	1.393(8)
C(1)–C(11)	1.485(6)	C(2)–C(15)	1.507(6)
C(11)–C(16)	1.397(6)	C(15)–C(16)	1.402(6)
C(11)–C(12)	1.396(6)	C(15)–C(14)	1.378(6)
C(12)–C(13)	1.387(7)	C(14)–C(13)	1.403(7)
C(7)–Cl(5)	1.731(10)	C(7)–Cl(6)	1.780(10)
Cl(1)–Ti–Cl(2)	94.0(1)	Cl(1)–Ti–Cl(3)	96.0(1)
Cl(1)–Ti–Cl(4)	93.1(1)	Cl(2)–Ti–Cl(3)	94.0(1)
Cl(2)–Ti–Cl(4)	167.9(1)	Cl(3)–Ti–Cl(4)	95.1(1)
O(1)–Ti–Cl(1)	174.2(2)	O(2^i)–Ti–Cl(3)	173.0(2)
O(1)–Ti–Cl(2)	85.4(2)	O(2^i)–Ti–Cl(2)	86.0(2)
O(1)–Ti–Cl(4)	86.6(2)	O(2^i)–Ti–Cl(4)	84.0(2)
O(1)–Ti–Cl(3)	89.8(2)	O(2^i)–Ti–Cl(1)	91.0(2)
O(1)–Ti–O(2^i)	83.2(2)	Cl(5)–C(7)–Cl(6)	111.5(6)
Ti–O(1)–C(1)	164.3(4)	Ti^i–O(2)–C(2)	159.7(4)
C(1)–O(3)–C(3)	120.1(4)	C(2)–O(4)–C(5)	118.4(4)
O(1)–C(1)–O(3)	123.2(5)	O(2)–C(2)–O(4)	123.7(5)
O(1)–C(1)–C(11)	123.4(5)	O(2)–C(2)–C(15)	123.5(5)
O(3)–C(1)–C(11)	113.4(4)	O(4)–C(2)–C(15)	112.8(4)
O(3)–C(3)–C(4)	106.5(5)	O(4)–C(5)–C(6)	105.6(4)
C(3)–C(4)–C(21)	112.0(5)	C(5)–C(6)–C(31)	113.8(5)
C(4)–C(21)–C(22)	119.9(5)	C(6)–C(31)–C(32)	121.8(5)
C(4)–C(21)–C(26)	120.7(5)	C(6)–C(31)–C(36)	120.3(5)
C(22)–C(21)–C(26)	119.3(6)	C(32)–C(31)–C(36)	117.8(6)
C(21)–C(22)–C(23)	120.4(6)	C(31)–C(32)–C(33)	121.8(6)
C(22)–C(23)–C(24)	120.1(6)	C(32)–C(33)–C(34)	119.9(7)
C(23)–C(24)–C(25)	119.4(6)	C(33)–C(34)–C(35)	119.4(7)
C(24)–C(25)–C(26)	120.7(7)	C(34)–C(35)–C(36)	120.8(7)
C(25)–C(26)–C(21)	120.0(6)	C(35)–C(36)–C(31)	120.3(6)
C(1)–C(11)–C(12)	120.6(5)	C(2)–C(15)–C(14)	118.6(4)
C(1)–C(11)–C(16)	118.5(4)	C(2)–C(15)–C(16)	120.0(4)
C(12)–C(11)–C(16)	120.9(5)	C(14)–C(15)–C(16)	121.3(5)
C(11)–C(12)–C(13)	119.7(5)	C(15)–C(14)–C(13)	119.6(5)
C(11)–C(16)–C(15)	118.3(5)	C(12)–C(13)–C(14)	120.1(5)
Ti–O(1)–C(1)–C(11)	−72.4(17)	Ti^i–O(2)–C(2)–C(15)	−52.8(16)
Ti–O(1)–C(1)–O(3)	107.8(16)	Ti^i–O(2)–C(2)–O(4)	128.7(17)
O(1)–C(1)–O(3)–C(3)	−1.9(7)	O(2)–C(2)–O(4)–C(5)	1.7(8)
C(1)–O(3)–C(3)–C(4)	−125.9(6)	C(2)–O(4)–C(5)–C(6)	170.9(7)
O(3)–C(3)–C(4)–C(21)	−179.4(6)	O(4)–C(5)–C(6)–C(31)	−64.0(7)

[a] Symmetry code: (i) $1-x, -y, -z$.

tahedron around the titanium atom. Each of the *m*-$C_6H_4(CO_2CH_2CH_2Ph)_2$ ligands is coordinated to two titanium atoms via two carbonyl oxygen atoms. At first glance the main features of the compound (dimeric complex, octahedral titanium, isophthalic acid phenyl rings parallel, sixteen-membered ring, etc.,) are similar to the previously known complex $[Ti_2\{\mu\text{-}m\text{-}C_6H_4(CO_2Et)_2\}_2Cl_8]$ (II) [7]. The corresponding dihedral angles Ti–O(1)–C(1)–C(11) of $-72.4(17)°$ [Ti–O(1)–C(11)–C(6) of $-60.5(16)°$] and Ti'–O(2)–C(2)–C(15) of $-52.8(16)°$ [Ti'–O(2)–C(12)–C(10) of $143.2(14)°$], in I [II] and the angles Ti–O(1)–C(1) and Ti–O(2)–C(2) of 164.3(4) and 159.7(4)° in I [156.0(4) and 173.9(4)°, in II] distinguish these two compounds.

The titanium atom is placed 2.085(3) and 2.131(4) Å from carbonyl oxygens O(1) and O(2), respectively. The Ti–O and Ti–Cl distances are similar to those in II. The isophthalic acid phenyl rings are planar in dimer I and are parallel and shifted mutually, so that they do not stack. As a consequence there are two short contacts C(16) · · · O(4') of 3.510(6) Å and C(16) · · · C(2') of 3.382(6) Å, only. The planes of ester groups and of the isophthalic acid phenyl ring in I form angles of ca. 20°. The carbon atom C(21) is *trans* to O(3). Instead, the carbon atom C(31) is in *gauche* position to O(4). The carbon atom C(1) is eclipsed to C(4) but C(2) is staggered in relation to C(6). The large differences between the torsion angles of C(1)–O(3)–C(3)–C(4) and C(2)–O(4)–C(5)–C(6) as well as between those of O(3)–C(3)–C(4)–C(21) and O(4)–C(5)–C(6)–C(31) (see Table 2) shows the different positions of the 2-phenylethyl alcohol rings towards the isophthalic acid ring. In the alcohol residue, with the C(21) atom, an extended chain is formed. In the other residue the phenyl ring is perpendicular to the isophthalate ring. In spite of this the location of the phenyl rings of both alcohol residues towards C(3) and C(5) atoms is similar.

A comparison of Ti–O–C–C and Ti–O–C angles of the dimeric compounds I and II under discussion shows that bulky substituents R in $C_6H_4(CO_2R)_2$, which are oriented away from the titanium atom, change the coordination geometries around the metal. Similar changes were observed also in the monomeric complexes with *o*-diesters [*o*-$C_6H_4(CO_2R)_2TiCl_4$], where R = Et and ^{t}Bu [11,12].

Supplementary material available. The tables of observed and calculated structure factors for crystals I and II are available from the authors.

Acknowledgement

This work was supported financially by MEN, grant 255.

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