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5,5-Dimethyl-2,2-bis(pentafluorophenyl)-4,4,6,6-tetrakis(trimethylsilyl)-1,3-dioxa-4,5,6-trisila-2-titanacyclohexane

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean *σ*(C–C) = 0.004 Å
R factor = 0.036
wR factor = 0.098
Data-to-parameter ratio = 17.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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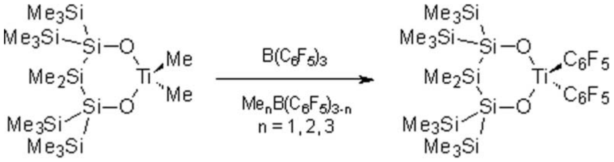
The title compound, [Ti(C_6F_5)$_2$($C_{14}H_{42}O_2Si_7$)], was readily synthesized from B(C_6F_5)$_3$ and SiMe$_2$[(Me$_3$Si)$_2$SiO]$_2$TiMe$_2$ in *n*-pentane. The compound contains one titanium metal centre with a distorted tetrahedral geometry.

Comment

We have investigated the reactivity of compound (1) (see scheme) towards the Lewis acid B(C_6F_5)$_3$ that is known to abstract alkyl groups from the metal centre to form cationic metal species active in olefin polymerization. Complex (1) reacted cleanly with B(C_6F_5)$_3$ (molar ratio 1:1) within minutes, resulting in the formation of Me$_2$Si[(Me$_3$Si)$_2$SiO]$_2$Ti(C_6F_5)$_2$, (2), the quantitative product of C_6F_5 group transfer. The fact that both methyl groups have been replaced smoothly by C_6F_5 groups indicates the high electrophilicity of the titanium centre caused by the electron-withdrawing ligand environment.



(1) (2)

The structure of (2) contains a titanium centre with a distorted tetrahedral coordination geometry (Fig. 1 and Table 1). The titanium is coordinated in a chelate fashion [O1—Ti1—O2 106.33 (7)°] by the siloxide ligand, giving a nearly planar six-membered ring. The Ti—O distances [1.7503 (15), 1.7408 (15) Å] in the ring are similar to those found in closely related cyclic siloxide complexes (Krempner *et al.*, 2006).

Experimental

All manipulations were carried out under an atmosphere of argon using standard Schlenk and glove-box techniques. Benzene-d_6 was dried over activated molecular sieves and stored in the glove box and *n*-pentane was distilled under argon from alkali metals prior to use. B(C_6F_5)$_3$ (Chernega *et al.*, 1997) and (1) (Krempner *et al.*, 2006) were prepared as previously described. NMR: Bruker AC 250. A Schlenk flask was charged with (1) (0.28 g, 0.54 mmol) and B(C_6F_5)$_3$ (0.27 g, 0.52 mmol) and dissolved in *n*-pentane (10 ml) at *ca* 253K. The stirred yellow solution was allowed to warm to room temperature within 30 min, after which time the solution was concentrated. Crystallization at 233K afforded SiMe$_2$[(Me$_3$Si)$_2$SiO]$_2$Ti(C_6F_5)$_2$ (2) as orange crystals [yield 0.18 g (41%); m.p. 395–396K]. ^{1}H-NMR (C_6D_6, 250 MHz): *d* = 0.41 (*s*, Si(CH$_3$)$_2$, 6 H), 0.21 (*s*, Si(CH$_3$)$_3$, 36 H) p.p.m.. ^{13}C-NMR (C_6D_6, 75.5 MHz): *d* = −0.7 (Si(CH$_3$)$_3$), −1.1 (Si(CH$_3$)$_2$), 134,4–149,6 (C_6F_5) p.p.m. ^{29}Si-NMR (C_6D_6, 59.6 MHz): d = 34.4 (SiO), −11.7 ($Si(CH_3)_3$), −28.5 ($Si(CH_3)_2$) p.p.m. ^{19}F (235.4 MHz): d −158.8, −146.8, −116.6 (*m*-, *p*-, *o*-F, $Ti(C_6F_5)_2$) p.p.m. Analysis calculated for $C_{26}H_{42}F_{10}O_2Si_7Ti$ (821.07): C, 38.03; H 5.16%. Found: C, 34.79; H 5.04%.

Crystal data

$[Ti(C_6F_5)_2(C_{14}H_{42}O_2Si_7)]$
M_r = 821.13
Monoclinic, $P2_1/c$
a = 11.3299 (2) Å
b = 19.1104 (3) Å
c = 19.7235 (4) Å
β = 102.039 (1)°
V = 4176.58 (13) Å^3
Z = 4
D_x = 1.306 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.47 mm^{-1}
T = 173 (2) K
Prism, orange
0.60 × 0.48 × 0.45 mm

Data collection

Bruker X8-APEX CCD diffractometer
φ and ω scans
Absorption correction: none
45244 measured reflections
7359 independent reflections
6528 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.027
$\theta_{\rm max}$ = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.099
S = 1.08
7359 reflections
429 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0379P)^2 + 3.8726P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max}$ = 0.001
$\Delta\rho_{\rm max}$ = 0.59 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.55 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ti1—O2	1.7408 (15)	Si2—O1	1.7127 (15)
Ti1—O1	1.7503 (15)	Si2—Si3	2.3869 (9)
Ti1—C15	2.135 (2)	Si3—Si4	2.3753 (9)
Ti1—C21	2.136 (2)	Si4—O2	1.7125 (15)
Si1—Si2	2.3540 (8)		
O2—Ti1—O1	106.33 (7)	O1—Ti1—C15	111.75 (8)
O2—Ti1—C15	109.77 (8)	O1—Ti1—C21	111.43 (9)
O2—Ti1—C21	108.35 (8)	C15—Ti1—C21	109.12 (9)
O1—Si2—Si3—Si4	−9.90 (7)	Si2—Si3—Si4—O2	6.83 (7)

All H atoms were initially located in a difference Fourier map. They were then constrained to an ideal geometry with a C—H distance of 0.98 Å and $U_{\rm iso}$ (H) = $1.5U_{\rm eq}$(C), but each group was allowed to rotate freely about its C—Si bond.

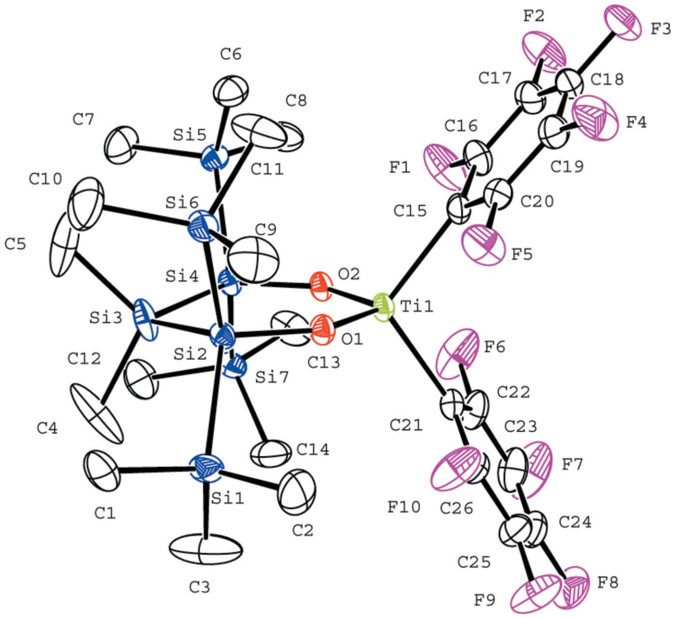


Figure 1
Fig. 1. The molecular structure of (2), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are omitted for clarity.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Acta Cryst. (2007). E**63**, m356–m357 Krempner *et al.* • $[Ti(C_6F_5)_2(C_{14}H_{42}O_2Si_7)]$ **m357**