

The isolation and structure of titanium bromide compounds containing 2,6-diphenylphenoxide ligation; terminal and bridging ligand effects on Ti–Ti interactions

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The titanium aryloxy bromides $[\text{Ti}(\text{OC}_6\text{H}_3\text{Ph}_2\text{-2,6})_2\text{Br}_2]$ **1** and $[(2,6\text{-Ph}_2\text{C}_6\text{H}_3\text{O})_2\text{Ti}(\mu\text{-Br})_2\text{Ti}(\text{OC}_6\text{H}_3\text{Ph}_2\text{-2,6})_2]$ **2** have been isolated and structurally characterized. Compound **1** adopts a pseudo-tetrahedral environment about titanium while **2** contains an edge-shared bis(tetrahedral) coordination geometry. The Ti–Ti distance is 3.0308(8) Å in **2**. A comparison of these compounds with their chloride counterparts as well as cyclopentadiene analogues has been made. The Ti–Ti distance in the compounds $[\text{Cp}'_2\text{Ti}(\mu\text{-X})_2\text{TiCp}'_2]$ (Cp' = methylcyclopentadienyl) increases as the bridging ligand X is changed from Cl to Br. In contrast this parameter is almost identical in the aryloxy compounds $[(\text{ArO})_2\text{Ti}(\mu\text{-X})_2\text{Ti}(\text{OAr})_2]$ (ArO = 2,6-diphenylphenoxide), due to the presence of a titanium–titanium bonding interaction.

Introduction

The structure and reactivity of both titanium(IV) and titanium(III) compounds are important aspects of modern inorganic and organometallic chemistry.¹ In the case of the d^1 -derivatives discrete paramagnetic, mononuclear compounds with coordination numbers as low as three (bulky ligands) are common.² In the case of dinuclear compounds containing two Ti(III) metal centers there are a spectrum of possible Ti–Ti interactions and corresponding magnetic behaviour. To date there has been no molecular species isolated containing an unsupported Ti–Ti bond.³ Hence the nature of the associated bridging ligands is an important variable in probing the nature of the underlying Ti–Ti interaction. During our studies of the organometallic chemistry associated with substrates $[(\text{ArO})_2\text{TiCl}_2]$ (ArO = bulky aryloxy ligand)^{4,5} we have isolated and characterized the d^1 - d^1 dimer $[(2,6\text{-Ph}_2\text{C}_6\text{H}_3\text{O})_2\text{Ti}(\mu\text{-Cl})_2\text{Ti}(\text{OC}_6\text{H}_3\text{Ph}_2)_2]$.⁶ In contrast to their paramagnetic, cyclopentadienyl analogues,^{7,8} this diamagnetic compound contains a short Ti–Ti distance of 2.9827(7) Å. Recent work has also led to the isolation of the “hybrid”, mixed cyclopentadienyl aryloxides which have intermediate Ti–Ti distances.^{9,10} In this paper we report on the synthesis and characterization of mixed bromo aryloxides of Ti(IV) and Ti(III). This study focuses on the possible influence of both bridging and terminal ligands upon the nature of Ti(III)–Ti(III) interactions.

Results and discussion

Synthesis and characterization of compounds

The addition of 2,6-diphenylphenol (2 equiv per Ti) to a benzene solution of $[\text{TiBr}_4]$ leads to the elimination of HBr and formation of the bis(aryloxy) $[\text{Ti}(\text{OC}_6\text{H}_3\text{Ph}_2\text{-2,6})_2\text{Br}_2]$ **1** as deep red crystals in good yield (Scheme 1). The solid-state structure of **1** (Fig. 1, Table 1) consists of a pseudo-

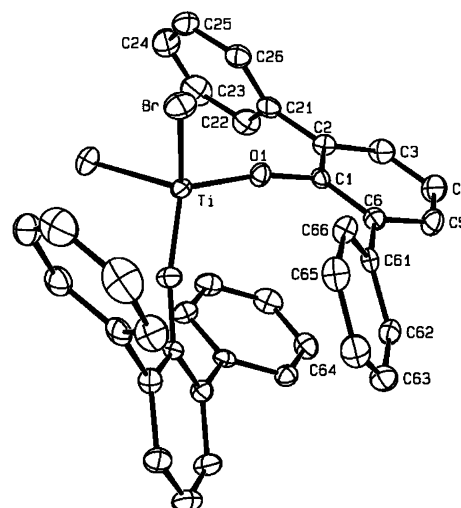


Fig. 1 Molecular structure of $[(2,6\text{-Ph}_2\text{C}_6\text{H}_3\text{O})_2\text{TiBr}_2]$ **1**.

tetrahedral titanium atom with a crystallographically imposed C_2 -axis leading to equivalent bromide and aryloxy ligands.

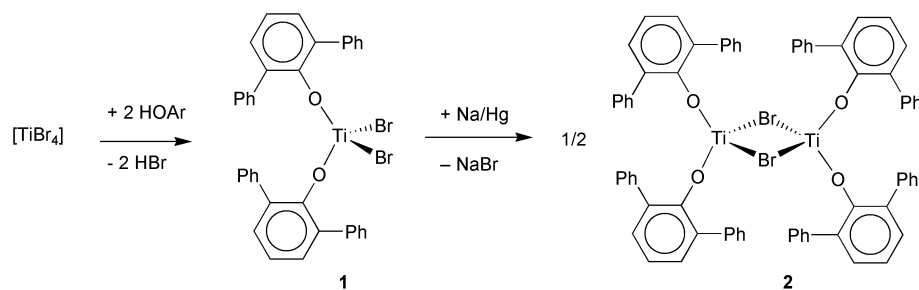
The sodium amalgam reduction of hydrocarbon solutions of **1** occurs smoothly over a number of hours to yield a dark, highly air and moisture sensitive solution. Recrystallization by layering a saturated toluene solution with pentane yielded pure crystals of titanium(III) compound $[(2,6\text{-Ph}_2\text{C}_6\text{H}_3\text{O})_2\text{Ti}(\mu\text{-Br})_2\text{Ti}(\text{OC}_6\text{H}_3\text{Ph}_2\text{-2,6})_2]$ **2** containing 1/2 toluene per Ti in the unit cell (Scheme 1). The solid state structure of **2** (Fig. 2, Table 2) is isostructural to that of the previously isolated chloride, with

Table 2 Selected bond distances (Å) and angles (°) for $[(2,6\text{-Ph}_2\text{C}_6\text{H}_3\text{O})_2\text{Ti}(\mu\text{-Br})_2]$ **2**

Ti(1)–O(2)	1.840(2)	Ti(1)–O(3)	1.826(3)
Ti(2)–O(1)	1.818(2)	Ti(2)–O(4)	1.833(3)
Ti(1)–Br(1)	2.5155(7)	Ti(1)–Br(2)	2.5171(7)
Ti(2)–Br(1)	2.5180(8)	Ti(2)–Br(2)	2.5205(7)
Ti(1)–Ti(2)	3.0308(8)		
O(2)–Ti(1)–O(3)	145.6(1)	O(1)–Ti(2)–O(4)	146.2(1)
Br(1)–Ti(1)–Br(2)	106.05(2)	Br(1)–Ti(2)–Br(2)	105.87(2)
Ti(1)–O(2)–C(11)	156.8(2)	Ti(1)–O(3)–C(31)	153.3(3)
Ti(2)–O(1)–C(21)	156.8(2)	Ti(1)–O(4)–C(41)	153.1(3)

Table 1 Selected bond distances (Å) and angles (°) for $[(2,6\text{-Ph}_2\text{C}_6\text{H}_3\text{O})_2\text{TiBr}_2]$ **1**

Ti–Br	2.3719(4)	Ti–O(1)	1.746(2)
O(1)–Ti–O(1)	112.6(1)	Br–Ti–Br	108.48(3)
O(1)–Ti–Br	111.38(5)	O(1)–Ti–Br	106.50(6)
Ti–O(1)–C(1)	168.3(2)		



Scheme 1

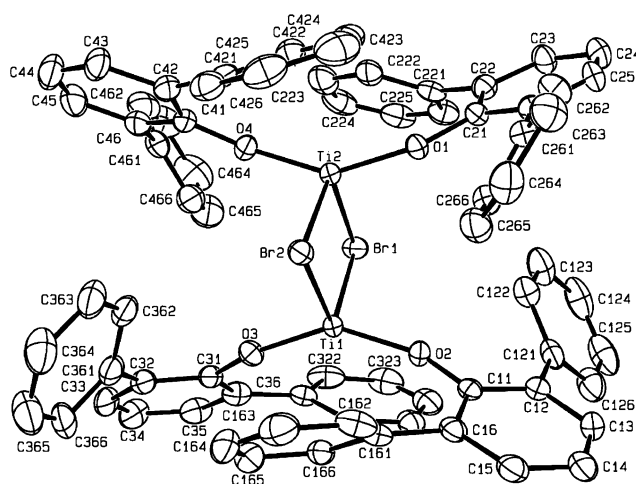


Fig. 2 Molecular structure of $[(2,6\text{-Ph}_2\text{C}_6\text{H}_3\text{O})_2\text{Ti}(\mu\text{-Br})_2]$.

two bromide ligands bridging two pseudo-tetrahedral titanium atoms. In both compounds there are large Ti–O–Ar angles, which are a common feature of early d-block metal aryloxides. Previous work has shown that the magnitude of these angles is not particularly informative.

The nature of the aryloxy ligands in **1** and **2** precludes any significant structural information being obtained from their ^1H NMR spectra. However, both 2,6-diphenylphenoxide compounds exhibit a sharp set of overlapping multiplets in the aromatic region of the spectrum. In the case of **2** this result indicates that the molecule is diamagnetic in C_6D_6 solution under ambient conditions.

Discussion of structural parameters

All of the molecules of interest in this study contain the titanium metal centers in a pseudo-tetrahedral environment. In Table 3 are contained some key structural parameters for a series of titanium(IV) halides containing cyclopentadienyl and aryloxy ligands.^{11–15} For the three chloride compounds it can be seen that replacement of cyclopentadiene by aryloxy leads to a significant shortening of the Ti–Cl bond lengths. This can be rationalized in terms of a corresponding increase in the electron deficiency of the metal center. The Ti–Br bond distances are, as expected, longer than the corresponding Ti–Cl bonds. Based upon organic structures one expects an increase of ~ 0.15 Å for E–Br over corresponding E–Cl distances. For the titanocene compounds the increase is ~ 0.13 Å while it is ~ 0.16 Å for the two aryloxy compounds (Table 3). A significant observation is that the X–Ti–X angle does not vary as X changes from Cl to Br.

In the case of the dinuclear compounds, the molecular geometry is best described as edge-shared bis(tetrahedral) with a $[\text{Ti}(\mu\text{-X})_2\text{Ti}]$ core. This is a very common structural motif in titanium chemistry. In titanium(IV) chemistry the shortest Ti–Ti distances occur when dianionic groups such as oxo, imido or alkylidene ligands bridge the d^0 -metals. Examples include $[\{\text{C}_5\text{H}_2(\text{SiMe}_3)\}_2\text{ClTi}(\mu\text{-O})_2\text{TiCl}\{\text{C}_5\text{H}_2(\text{SiMe}_3)\}_2]$ 2.7071(4) Å,¹⁶

Table 3 Structural parameters for $[(\text{L})(\text{L}')\text{TiX}_2]$; L, L' = Cp or ArO; X = Cl, Br

[Compound] ^{ref.}	Ti–X/Å	L–Ti–L'/°	X–Ti–X/°
$[\text{Cp}_2\text{TiCl}_2]$ ¹¹	2.367(2) 2.361(1)	131	94
$[\text{Cp}_2\text{TiBr}_2]$ ¹⁵	2.489(2) 2.491(2)	131	95
$[\text{CpTi}(\text{OC}_6\text{HPh}_4\text{-}2,3,5,6)\text{Cl}_2]$ ^{9c}	2.255(1) 2.240(1)	115	102
$[\text{Ti}(\text{OC}_6\text{H}_3\text{Ph}_2\text{-}2,6)_2\text{Cl}_2]$ ¹³	2.206(1)	113	109
$[\text{Ti}(\text{OC}_6\text{H}_3\text{Ph}_2\text{-}2,6)_2\text{Br}_2]$ ^a	2.3719(4) 2.3720(4)	113	108

^a This work.

$[(2,6\text{-Me}_2\text{C}_6\text{H}_3)_2\text{Ti}(\mu\text{-NBu}^t)_2\text{Ti}(\text{OC}_6\text{H}_3\text{Me}_2\text{-}2,6)_2]$ 2.7909(7) Å,¹⁷ $[(\text{cb})_2\text{ClTi}(\mu\text{-CHSiMe}_3)_2\text{Ti}(\text{cb})_2]$ 2.9504(8) Å,¹⁸ and $[(\text{Cy}_2\text{N})_2\text{-ClTi}(\mu\text{-CH}_2)_2\text{Ti}(\text{NCy}_2)_2]$ 2.934(2) Å.¹⁹

In the case of di-titanium(III) compounds, there exists a great deal of variation in Ti–Ti distances for compounds containing the $[\text{Ti}(\mu\text{-Cl})_2\text{Ti}]$. This is highlighted by the titanium(III) halides containing cyclopentadienyl and aryloxy ligands shown in Table 4. Very few compounds contain Ti–Ti distances < 3 Å for $[\text{Ti}(\mu\text{-Cl})_2\text{Ti}]$ central cores. An important class of compounds are the edge-shared bis-octahedral formamidinate complexes $[\text{Ti}_2\{\text{RNC}(\text{H})\text{CNR}\}_2\{\text{RNC}(\text{H})\text{CNR}\}_2(\mu\text{-Cl})_2]$ isolated by Cotton *et al.* (R = C_6H_5 , $\text{C}_6\text{H}_4\text{Ph}$ -4)²⁰ and Gambarotta *et al.* (R = $\text{c-C}_6\text{H}_{11}$).²¹ In these diamagnetic compounds the Ti–Ti distances of 2.8890(8), 2.916(3) and 2.942(2) Å respectively were taken as indicative of the presence of a metal–metal single bond. The aryloxy $[(2,6\text{-Ph}_2\text{C}_6\text{H}_3\text{O})_2\text{Ti}(\mu\text{-Cl})_2]$ is therefore unusual. It can be seen for the chlorides in Table 4 that the Ti–Ti distance shortens dramatically as cyclopentadienyl ligands are replaced by aryloxides. It may be argued that this is simply a consequence of the corresponding decrease in Ti–Cl distances. Furthermore there is an increase in Cl–Ti–Cl angle noticed for the Ti(IV) compounds (Table 3). Both of these effects will lead to the metal centers being pulled closer together as we substitute ArO for Cp in the dinuclear, halide bridged species. However, the data obtained in this study for the bromides indicate that the Ti–Ti interaction in the aryloxy compounds is not simply a function of the bridging ligands. It can be seen that as Cl is replaced by Br in the compounds $[\text{Cp}'_2\text{Ti}(\mu\text{-X})_2\text{TiCp}'_2]$ (Cp' = methylcyclopentadienyl) the Ti–Ti distance increases from 3.926(3) to 4.125(4) Å. This increase of ~ 0.2 Å can be accounted for by the underlying increase in Ti–μ–X distances, ~ 0.16 Å. However, in the $[(\text{ArO})_2\text{Ti}(\mu\text{-X})_2\text{Ti}(\text{OAr})_2]$ (ArO = 2,6-diphenylphenoxide) the increase in Ti–Ti distance is only ~ 0.04 Å. This is despite the expected increase in Ti–μ–X distances of ~ 0.15 Å. Instead of increasing the Ti–Ti distance, the aryloxy compounds compensate by moving the halide atoms further apart with a corresponding increase in the X–Ti–X angle and closing of the Ti–X–Ti angle on replacing Cl by Br (Table 4). The key structural changes for the four $[\text{Ti}(\mu\text{-X})_2\text{Ti}]$ trapezoids are shown exaggerated in the radar plots shown in Fig. 3. In the case of the methylcyclopentadienyl (Cp') compounds it can be clearly that the trapezoid

Table 4 Structural parameters for [(L)(L')Ti(μ -X)₂Ti(L)(L')]; L, L' = Cp or ArO; X = Cl, Br

[Compound] ^{ref.}	L–Ti–L'/ $^\circ$	X–Ti–X'/ $^\circ$	Ti–X–Ti/ $^\circ$	Ti–X/Å	Ti–Ti/Å
[(C ₅ H ₄ Me) ₂ Ti(μ -Cl)] ₂ ⁷	131	79	101 101	2.535(2) 2.566(2) 2.562(2) 2.526(2)	3.926(3)
[(C ₅ H ₄ Me) ₂ Ti(μ -Br)] ₂ ⁷	134	81	99	2.722(2) 2.705(3)	4.125(4)
[Cp(2,4-Bu ^t -Np-6-C ₆ H ₃ O)Ti(μ -Cl)] ₂ ^{9c}	125	92	88	2.400(1) 2.406(1)	3.336(1)
[(2,6-Ph ₂ C ₆ H ₃ O) ₂ Ti(μ -Cl)] ₂ ⁶	144	102	78	2.3660(9) 2.3761(9)	2.9827(7)
[(2,6-Ph ₂ C ₆ H ₃ O) ₂ Ti(μ -Br)] ₂ ^a	147 146	106 106	74 74	2.5155(7) 2.5180(8) 2.5171(7) 2.5205(7)	3.0308(8)

Np = 1-naphthyl.^a This work.

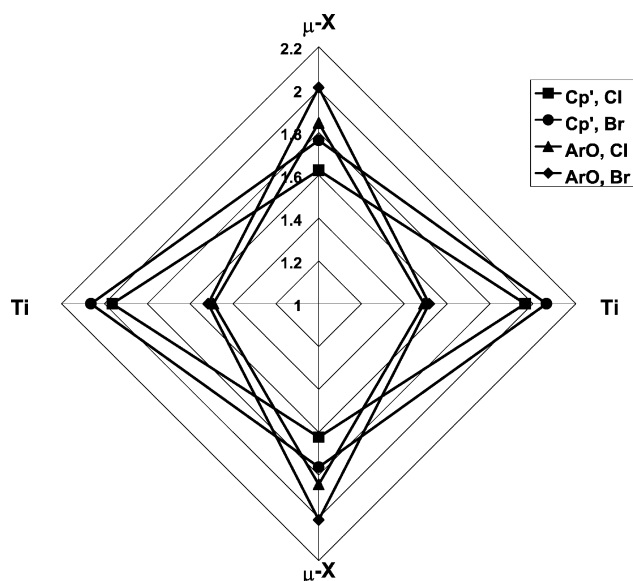


Fig. 3 “Radar” plot of the distances of the titanium and halide atoms about the centroid of the [Ti(μ -X)₂Ti] (X = Cl, Br) core in the four compounds [Cp'₂Ti(μ -X)₂TiCp'₂] (Cp' = methylcyclopentadienyl) and [(ArO)₂Ti(μ -X)₂Ti(OAr)₂] (ArO = 2,6-diphenylphenoxide). The change in atomic positions is exaggerated by using a plot scale of 1.0–2.2 Å from the centroid.

expands evenly as Br replaces Cl. However, in the case of the 2,6-diphenylphenoxides, the central core accommodates the change from Cl to Br by increasing the halide–halide distance. The Ti–Ti distance does not increase proportionately. This analysis leads to the conclusion that the short Ti–Ti interaction in the aryloxo compounds is less sensitive to the nature of the bridging groups and implies the existence of a Ti–Ti single bond.

Experimental

General remarks

All manipulations were carried out using standard syringe, Schlenk line, and glovebox techniques.²² Hydrocarbon solvents were dried by distillation from sodium/benzophenone and stored under dry nitrogen. All reagents were dried over 3 Å molecular sieves prior to use. The ¹H NMR spectra were recorded on a Varian Associates Gemini 200 and an Inova 300 spectrometer and were referenced using protio impurities of commercial benzene-*d*₆ as an internal standard. Micro-analytical data were obtained in-house at Purdue. The X-ray diffraction studies were completed “in-house” at Purdue University.

Table 5 Crystal data and data collection parameters

	1	2·1/2toluene
Formula	C ₃₆ H ₂₆ Br ₂ O ₂ Ti	C _{75.50} H ₅₆ Br ₂ O ₄ Ti ₂
Formula weight	698.33	1282.90
Space group	<i>P</i> 2 ₁ 2 ₁ 2 (No. 18)	<i>P</i> $\bar{1}$ (No. 2)
<i>a</i> /Å	10.4945(3)	14.6298(3)
<i>b</i> /Å	15.3372(4)	15.9823(4)
<i>c</i> /Å	9.5602(6)	16.0980(5)
α / $^\circ$	90	115.1707(11)
β / $^\circ$	90	92.5252(13)
γ / $^\circ$	90	115.7050(14)
<i>V</i> /Å ³	1538.77(17)	2946.5(3)
<i>Z</i>	2	2
ρ_{calc} /g cm ⁻³	1.507	1.446
Temperature/K	150	150
Radiation (wavelength)	Mo K α (0.71073 Å)	Mo K α (0.71073 Å)
<i>R</i>	0.032	0.055
<i>R</i> _w	0.061	0.139

Synthesis of [Ti(OC₆H₃Ph₂-2,6)₂Br₂] (1)

A sample of [TiBr₄] (5.92 g, 0.016 mol) was dissolved in benzene (250 mL) and stirred until an orange/yellow solution was obtained. To this solution, solid 2,6-diphenylphenol (7.87 g, 0.032 mol) was added. The red/brown mixture was left to stir for 2 hours. The mixture was then evacuated to dryness under vacuum giving an orange solid which could be recrystallised from toluene/pentane. Yield = 10.1 g (88%). Anal. Calcd. for C₃₆H₂₆Br₂O₂Ti: C, 61.92; H, 3.75; Br, 22.89. Found: C, 61.93; H, 3.89; Br, 22.72%. ¹H NMR (C₆D₆, 30 °C): δ 6.60–7.60 (aromatics).

Synthesis of [(2,6-Ph₂C₆H₃O)₂Ti(μ -Br)₂Ti(OC₆H₃Ph₂-2,6)] (2)

A sample of [Ti(OC₆H₃Ph₂-2,6)₂Br₂] **1** (1.00 g, 1.43 mmol) was added to benzene (25 mL) above sodium (excess, 0.050 g, 2.14 mmol) as a mercury amalgam pool. The red solution was stirred vigorously overnight. The resulting solution was decanted from the mercury pool, filtered to remove sodium salts and then evacuated to dryness, affording a dark solid. This crude solid was recrystallised from toluene/pentane as crystals containing 1/2 toluene molecule per Ti in the unit cell. Yield = 0.40 g (45%). Anal. Calcd. for C_{75.50}H₅₆Br₂O₄Ti₂: C, 70.55; H, 4.42; Br, 12.52. Found: C, 70.92; H, 4.41; Br, 12.27%. ¹H NMR (C₆D₆, 30 °C): δ 6.50–7.50 (aromatics).

X-Ray data collection and reduction

Crystal data and data collection parameters are contained in Table 5. A suitable crystal was mounted on a glass fiber in a random orientation under a cold stream of dry nitrogen. Preliminary examination and final data collection were performed

with MoK α radiation ($\lambda = 0.71073 \text{ \AA}$) on a Nonius Kappa CCD. Lorentz and polarization corrections were applied to the data.²³ An empirical absorption correction using SCALEPACK was applied.²⁴ Intensities of equivalent reflections were averaged. The structure was solved using the structure solution program PATTY in DIRDIF92.²⁵ The remaining atoms were located in succeeding difference Fourier syntheses. Hydrogen atoms were included in the refinement but restrained to ride on the atom to which they are bonded. The structure was refined in full-matrix least-squares where the function minimized was $\sum w(|F_o|^2 - |F_c|^2)^2$ and the weight w is defined as $w = 1/[\sigma^2(F_o^2) + (0.0585P)^2 + 1.4064P]$ where $P = (F_o^2 + 2F_c^2)/3$. Scattering factors were taken from the "International Tables for Crystallography".²⁶ Refinement was performed on a AlphaServer 2100 using SHELX-97.²⁷ Crystallographic drawings were done using programs ORTEP.²⁸

CCDC reference numbers 200530 and 200531.

See <http://www.rsc.org/suppdata/dt/b2/b212909d/> for crystallographic files in CIF or other electronic format.

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