

Interaction of bis(trimethylsilyl)acetylene complex of titanocene with tris(pentafluorophenyl)borane. Synthesis and structure of a new type of zwitterionic metallocene

$$(\eta^5\text{-C}_5\text{H}_5)\{\eta^5\text{-}[\text{C}_5\text{H}_4\text{B}(\text{C}_6\text{F}_5)_3]\}\text{Ti}$$

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Abstract

In the interaction of the bis(trimethylsilyl)acetylene complex of titanocene $\text{Cp}_2\text{Ti}(\text{Me}_3\text{SiC}_2\text{SiMe}_3)$ with an equimolar amount of $\text{B}(\text{C}_6\text{F}_5)_3$ in toluene at 20°C, electrophilic substitution of a hydrogen atom in one of the $\eta^5\text{-C}_5\text{H}_5$ rings by a $\text{B}(\text{C}_6\text{F}_5)_3$ group takes place and the paramagnetic zwitterionic titanium complex $(\eta^5\text{-C}_5\text{H}_5)\{\eta^5\text{-}[\text{C}_5\text{H}_4\text{B}(\text{C}_6\text{F}_5)_3]\}\text{Ti}$ (**I**) is produced. The formation of **I** is accompanied by liberation of molecular hydrogen. Bis(trimethylsilyl)acetylene and 1,2-bis(trimethylsilyl)ethane were found in organic products of the reaction. An X-ray diffraction study of complex **I** revealed the coordination of the *ortho*-fluorine atoms of two C_6F_5 groups with the positively charged titanium centre. In contrast to other known titanium, zirconium and hafnium zwitterionic metallocenes, complex **I** contains a metal in the +3 oxidation state and does not contain any σ -bonded organic radicals at the metal atom. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Zwitterionic metallocenes; Acetylene complexes; Titanium; Tris(pentafluorophenyl)borane; Electrophilic substitution; X-ray structure

1. Introduction

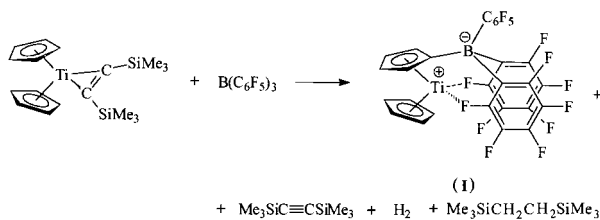
Zwitterionic cyclopentadienyl complexes of the Group IVB metals have received considerable attention in the past decade due to their ability to catalyse the polymerization of olefinic monomers in the absence of Lewis-acidic cocatalysts (see e.g. Ref. [1]). The greatest progress in this important area has been achieved in studying the synthesis and reactivity of zwitterionic zirconocene derivatives. For titanium and hafnium, only few examples of similar betaine metallocenes have been described [2–4]. In all presently known zwitter-

ionic titanocene, zirconocene and hafnocene complexes, the metal atom is in the +4 oxidation state and contains at least one σ -bonded organic group.

In the present paper, we wish to report on the synthesis and structure of a novel type of zwitterionic metallocene, which is formed in the interaction of the bis(trimethylsilyl)acetylene complex of titanocene $\text{Cp}_2\text{Ti}(\text{Me}_3\text{SiC}_2\text{SiMe}_3)$ [5,6] with tris(pentafluorophenyl)borane $\text{B}(\text{C}_6\text{F}_5)_3$. It turned out that in the course of this reaction, electrophilic substitution of a hydrogen atom in one of the $\eta^5\text{-C}_5\text{H}_5$ rings by a $\text{B}(\text{C}_6\text{F}_5)_3$ group takes place and a zwitterionic complex of trivalent titanium, $(\eta^5\text{-C}_5\text{H}_5)\{\eta^5\text{-}[\text{C}_5\text{H}_4\text{B}(\text{C}_6\text{F}_5)_3]\}\text{Ti}$ (**I**), containing no σ -bonded organic radicals at the metal atom is produced. For preliminary communication, see Ref. [7].

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Scheme 1.

2. Results and discussion

In the interaction of Cp₂Ti(Me₃SiC₂SiMe₃) with an equimolar amount of B(C₆F₅)₃ in toluene at room temperature under Ar, the colour of the reaction mixture gradually turns from dark yellow to blue and after 24 h dark-blue crystals of complex **I** can be isolated from the solution. The reaction is accompanied by liberation of molecular hydrogen (0.11 mol per mol of the starting acetylene complex). Bis(trimethylsilyl)acetylene and 1,2-bis(trimethylsilyl)ethane (in 4:1 ratio) were found in organic products of the reaction (Scheme 1).

Electrophilic substitution of a hydrogen atom in the η⁵-C₅H₅ ring by a B(C₆F₅)₃ group has been previously observed by Erker and co-workers in studying the interaction of B(C₆F₅)₃ with 1,1-bis(cyclopentadienyl)-2,3,4,5-tetramethylzirconacyclopentadiene metallacycle (**II**) [8]. The reaction results in the formation of the zwitterionic zirconocene complex (η⁵-C₅H₅){η⁵-[C₅H₄B(C₆F₅)₃]}ZrC(Me)=C(Me)C(Me)=CHMe, which contains a trimethylpentadienyl group bonded to Zr(IV) atom. Similar zwitterionic metallocene (η⁵-C₅H₅){η⁵-[C₅H₄BH(C₆F₅)₂]}ZrC(Et)=CH(Et) contain-

ing σ-alkenyl ligand attached to Zr(IV) centre has been isolated recently by Piers from the reaction of BH(C₆F₅)₂ with a mixed phosphine–acetylene zirconium complex Cp₂Zr(EtC₂Et)(PMe₃) [1].

Complex **I** is a dark-blue paramagnetic crystalline substance readily soluble in THF and less readily in toluene. In the solid state, the complex is stable at room temperature in Ar atmosphere but it rapidly decomposes on contact with air oxygen and moisture. According to ¹H-NMR spectra and elemental analysis, the complex contains (after drying for 2 h at 20°C in vacuum) half a mole of toluene per mole of **I**. If the time of drying is shortened to 10 min, the content of toluene in complex **I** increases to 1 mol. The crystals of this toluene solvate, **I**·C₆H₅CH₃, were used for carrying out the X-ray diffraction study of the complex (see below).

The ESR spectrum of **I** in toluene at 20°C exhibits an intense broadened (8 gauss) singlet signal with *g* = 1.962. Dilution and/or cooling of the solution to –30°C do not affect practically a pattern of the signal. A similar broadened (7.5 gauss) singlet but with different value of the *g*-factor (1.970) is observed in the ESR spectrum of a dark-blue solution of **I** in THF. Such a change in the position of the singlet in the spectrum on passing from toluene to THF is evidently due to a complexation of THF with titanium atom in the molecule of **I**.

Fig. 1 shows the structure of **I**. The selected bond distances and angles for **I** are presented in Table 1. The complex is indeed a bis(η⁵-cyclopentadienyl)titanium(III) derivative in which one of the hydrogen atoms of the η⁵-C₅H₅ ring is substituted by a B(C₆F₅)₃ group. A characteristic feature of **I** is the presence of coordinative bonds between the *ortho*-fluorine atoms (F(1) and F(6)) of two C₆F₅ substituents and the

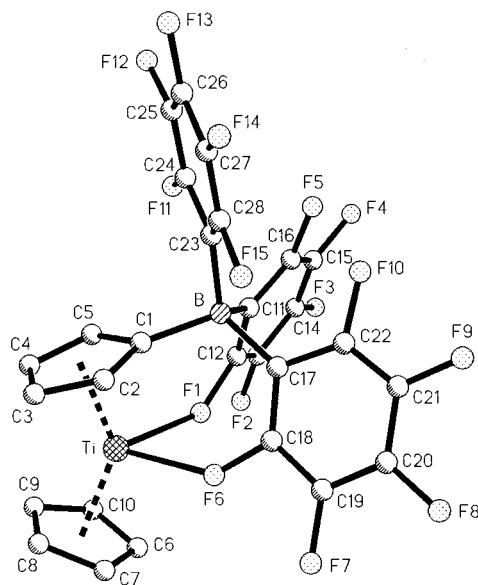


Fig. 1. Structure of the zwitterionic complex (η⁵-C₅H₅){η⁵-[C₅H₄B(C₆F₅)₃]}Ti (**I**).

Table 1
Selected bond distances (Å) and angles (°) for **I**

Bond distances			
Ti–C(1)	2.258(5)	Ti–C(6)	2.308(5)
Ti–C(2)	2.303(5)	Ti–C(7)	2.341(5)
Ti–C(3)	2.358(5)	Ti–C(8)	2.356(5)
Ti–C(4)	2.343(4)	Ti–C(9)	2.345(5)
Ti–C(5)	2.285(4)	Ti–C(10)	2.328(5)
Ti–Cp(1)	1.977(3)	Ti–Cp(2)	2.015(3)
Ti–F(1)	2.248(2)	Ti–F(6)	2.223(3)
F(1)–C(12)	1.401(4)	F(6)–C(18)	1.382(5)
B–C(1)	1.626(7)	B–C(17)	1.656(7)
B–C(11)	1.646(7)	B–C(23)	1.662(7)
Bond angles			
Cp(1)–Ti–Cp(2)	139.5(2)	F(1)–Ti–F(6)	74.0(1)
Cp(1)–Ti–F(1)	105.1(2)	Cp(2)–Ti–F(6)	107.7(2)
Cp(1)–Ti–F(6)	104.9(2)	Cp(2)–Ti–F(1)	106.4(2)
Ti–F(1)–C(12)	139.7(2)	Ti–F(6)–C(18)	139.9(2)
C(1)–B–C(11)	111.1(3)	C(11)–B–C(17)	103.6(3)
C(1)–B–C(17)	113.3(3)	C(11)–B–C(23)	113.9(3)
C(1)–B–C(23)	101.6(4)	C(17)–B–C(23)	113.8(4)

positively charged titanium centre (the Ti⋯F(1) and Ti⋯F(6) distances are 2.248(2) and 2.223(3) Å, respectively). As a result of the coordination of the F(1) and F(6) atoms with titanium, the C(12)–F(1) and C(18)–F(6) bonds become longer (1.401(4) and 1.382(5) Å, respectively) as compared with other C–F bonds in the molecule of **I** (1.330–1.364 Å, av. 1.348 Å).

Similar Ti⋯F–C interaction is realized in the structure of the cationic titanium(III) complex with fluorobenzene, $[\text{Cp}_2^*\text{Ti}(\text{C}_6\text{H}_5\text{F})]^+ \text{BPh}_4^-$ [9]. Here the Ti⋯F distance (2.151 Å) is even shorter than the corresponding distances in **I**. The C–F bond length in $[\text{Cp}_2^*\text{Ti}(\text{C}_6\text{H}_5\text{F})]^+$ is 1.402 Å. The coordination of two C–F bonds of a $\text{B}(\text{C}_6\text{F}_5)_3$ unit to Zr(IV) centre has been found in the structure of complex $[\text{Cp}_2^*\text{ZrH}]^+ [\text{HB}(\text{C}_6\text{F}_5)_3]^-$ [10]. However, in this complex, in contrast to **I**, the *ortho*- and *meta*-fluorine atoms of the same C_6F_5 ring are involved in the bonding to the metal. There are also examples of zirconocene complexes with the coordination of one C–F bond of a $\text{B}(\text{C}_6\text{F}_5)_3$ group to Zr(IV) atom [3,8]. In all of the above zirconium complexes, the lengths of the coordinated C–F bonds (1.379–1.402 Å) are close to those in **I**.

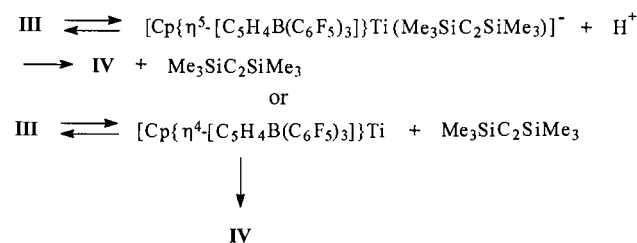
The geometry of the bent sandwich in **I** is not exceptional. The planes of the substituted and unsubstituted C_5 rings form a dihedral angle of 36.2°. The titanium atom and coordinated C(12)–F(1) and C(18)–F(6) bonds are located approximately in the bisector plane of this dihedral angle. Coordination environment at titanium can be described as pseudotetrahe-

dral with the large Cp(1)–Ti–Cp(2) angle (139.5°) and small F(1)–Ti–F(6) angle (74.0(1)°) owing to the size difference between Cp ligand and fluorine atom (Cp(1) and Cp(2) are the centroids of the substituted and unsubstituted C_5 rings, respectively). The Ti–Cp(1) distance (1.977 Å) is somewhat shorter than Ti–Cp(2) (2.015 Å), which is caused, apparently, by the bonding of the substituted C_5 ring to titanium via the C–B–C–C–F bridges. The Ti–F(1)–C(12) and Ti–F(6)–C(18) angles are virtually the same (139.7(2) and 139.9(2)°). The Cp ligands are in eclipsed conformation. The Ti–C distances are in the range of 2.26–2.36 Å with slightly shorter Ti–C(1) and Ti–C(6) distances because of the small slippage of the Cp rings. Despite the bonding of two C_6F_5 groups to the titanium centre, the boron atom is located nearly exactly in the plane of the C_5 ring; its displacement from this plane (in the direction opposite to the Ti atom) is only 0.045 Å. Coordination at the boron atom is essentially tetrahedral with the B–C bond lengths in the range of 1.626–1.662 Å and the C–B–C bond angles of 102–114°.

The proposed mechanism of the reaction of $\text{B}(\text{C}_6\text{F}_5)_3$ with $\text{Cp}_2\text{Ti}(\text{Me}_3\text{SiC}_2\text{SiMe}_3)$ is shown in Scheme 2.

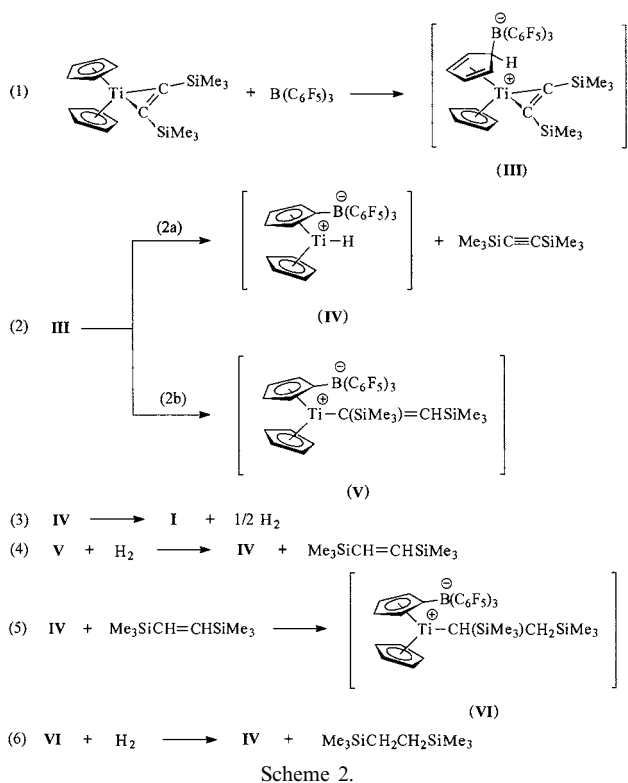
Stages (1) and (2b) in the Scheme are analogous to those postulated by Erker and co-workers [8] for the above-mentioned reaction of $\text{B}(\text{C}_6\text{F}_5)_3$ with zirconacyclopentadiene metallacycle **II**. In stage (1), a highly electrophilic $\text{B}(\text{C}_6\text{F}_5)_3$ attacks one of the $\eta^5\text{-C}_5\text{H}_5$ rings of the starting acetylene complex to afford the intermediate η^4 -cyclopentadiene titanium derivative **III** containing a $\text{B}(\text{C}_6\text{F}_5)_3$ group in an *exo*-position of the $\eta^4\text{-C}_5\text{H}_5\text{B}(\text{C}_6\text{F}_5)_3$ moiety and, correspondingly, a hydrogen atom in an *endo*-position.

Further transformations of the resulting **III** may occur in two ways. One way (stage (2a)) is the *endo*-proton transfer to titanium atom with the formation of the intermediate titanium hydride complex **IV** and free bis(trimethylsilyl)acetylene. This process could, in principle, proceed stepwise, for example:



Another way (stage (2b)) is the *endo*-proton transfer to a carbon atom of the acetylene ligand in **III** to yield the σ -alkenyl titanium derivative **V**.

The transient hydride complex **IV** formed in stage (2a) can be considered as a product of the reaction of the Cp-substituted titanocene $[\text{Cp}\{\eta^5\text{-}[\text{C}_5\text{H}_4\text{B}(\text{C}_6\text{F}_5)_3]\}\text{Ti}]^-$ with H^+ . Therefore, it must readily decompose producing molecular hydrogen and the final zwitter-



Scheme 2.

ionic complex **I** (stage (3)). Such a mechanism for the formation of **I** is well consistent with the isolation of the cationic titanium(III) complex $[\text{Cp}_2\text{Ti}(\text{THF})_2]^+ \text{BPh}_4^-$ from the interaction of $\text{Cp}_2\text{Ti}(\text{Me}_3\text{SiC}_2\text{SiMe}_3)$ in THF with $\text{Me}_3\text{NH}^+ \text{BPh}_4^-$ as a proton source [11]. One may suggest that this reaction proceeds via the intermediate formation of the protonated titanocene $[\text{Cp}_2\text{TiH}]^+$, the subsequent decomposition of which with liberation of dihydrogen gives the end Ti(III) derivative. Electrophilic substitution of a hydrogen atom in the $\eta^5\text{-C}_5\text{H}_5$ ring by Lewis acid, accompanied by a proton transfer to a metal centre, has been observed earlier by Braunschweig and Wagner [12] in the interaction of Cp_2WH_2 with RBCl_2 ($\text{R} = i\text{-Pr}, t\text{-Bu}$). The reaction results in the zwitterionic metallocenes $(\eta^5\text{-C}_5\text{H}_5)\{\eta^5\text{-}[\text{C}_5\text{H}_4\text{BCl}_2(\text{R})]\}\text{WH}_3$ which, in contrast to the hypothetical **IV**, are quite stable.

The mechanism of the formation of 1,2-bis(trimethylsilyl)ethane in the reaction of $\text{Cp}_2\text{Ti}(\text{Me}_3\text{SiC}_2\text{SiMe}_3)$ with $\text{B}(\text{C}_6\text{F}_5)_3$ is described by stages (2b) and (4–6) in Scheme 2. An alternative mechanism consisting in the catalytic hydrogenation of bis(trimethylsilyl)acetylene with dihydrogen under the action of **I** is not supported by the experimental data, which show that $\text{Me}_3\text{SiC}\equiv\text{CSiMe}_3$ does not react with H_2 in toluene at 20°C in the presence of **I**.

Complex **I** is also inactive in polymerization of ethylene, styrene and 1,3-butadiene in toluene at $40\text{--}60^\circ\text{C}$. This fact is in a good agreement with the generally accepted idea that for catalysis of the olefin polymerization the Group IVB cationic metallocene should have a metal in the +4 oxidation state and contain a σ -bonded organic or hydride ligand.

3. Experimental

Experiments were conducted under Ar or in vacuum with careful exclusion of air and moisture. Toluene was purified in the usual manner and twice freshly distilled before use over sodium under Ar. The starting acetylene complex $\text{Cp}_2\text{Ti}(\text{Me}_3\text{SiC}_2\text{SiMe}_3)$ was prepared by the method described in Refs. [5,6]. Commercial $\text{B}(\text{C}_6\text{F}_5)_3$ was used without additional purification. GLC–MS analyses were performed on a VG 7070E instrument (ionization energy 70 eV, temperature of ion source 150°C) using the capillary SE-54 column (temperature programming $35\text{--}230^\circ\text{C}$, 8°C min^{-1}). The NMR spectra were recorded on a Bruker AMX-400 spectrometer. The ESR spectra were registered on a Varian E12A instrument.

3.1. Reaction of $\text{Cp}_2\text{Ti}(\text{Me}_3\text{SiC}_2\text{SiMe}_3)$ with $\text{B}(\text{C}_6\text{F}_5)_3$

A total of 0.54 g (1.5 mmol) of $\text{Cp}_2\text{Ti}(\text{Me}_3\text{SiC}_2\text{SiMe}_3)$ was dissolved in 5 ml of toluene under Ar and the

resulting solution was added to a solution of 0.76 g (1.5 mmol) of $\text{B}(\text{C}_6\text{F}_5)_3$ in 10–12 ml of toluene. After 5–10 min, the reaction mixture was filtered off and then kept overnight at room temperature (r.t.) in an Ar atmosphere. After 24 h, trituration and shaking led to deposition of dark-blue crystals of complex **I** from the solution. The crystals were separated, washed with a small amount of toluene and dried for 2 h at 20°C in vacuum. Despite drying, the obtained complex contained a half mole of toluene per 1 mol of **I** ($^1\text{H-NMR}$ and elemental analysis data). The yield of **I** is 0.73 g (70%), m.p. $161\text{--}163^\circ\text{C}$ (dec.) under Ar. Analysis: Found: C, 51.16; H, 1.94%. $\text{C}_{28}\text{H}_9\text{TiBF}_{15}\cdot 1/2\text{C}_6\text{H}_5\text{CH}_3$ requires: C, 51.47; H, 1.78%. For identification of organic products of the reaction, the solution obtained after separation of crystals of **I** was analysed by the GLC–MS method. The analysis revealed the presence of bis(trimethylsilyl)acetylene and 1,2-bis(trimethylsilyl)ethane in the solution. The ratio of $\text{Me}_3\text{SiC}\equiv\text{CSiMe}_3$ to $\text{Me}_3\text{SiCH}_2\text{CH}_2\text{SiMe}_3$ was 4:1.

In the other experiment, the reaction of $\text{Cp}_2\text{Ti}(\text{Me}_3\text{SiC}_2\text{SiMe}_3)$ with $\text{B}(\text{C}_6\text{F}_5)_3$ was conducted in a sealed two-chamber glass vessel in vacuum. A solution of 0.41 g (1.2 mmol) of $\text{Cp}_2\text{Ti}(\text{Me}_3\text{SiC}_2\text{SiMe}_3)$ in 3–4 ml of toluene was placed in one chamber of the vessel while a solution of 0.59 g (1.15 mmol) of $\text{B}(\text{C}_6\text{F}_5)_3$ in 5 ml of toluene was placed in another chamber. After freezing of both solutions with liquid dinitrogen, the vessel was evacuated and sealed off. Then the solutions were heated to r.t. and mixed with each other. After 24 h, the vessel was connected to a high vacuum unit (equipped with a gas burette and a Toepler pump) and opened in vacuum. The reaction mixture was frozen and dihydrogen liberated in the course of the reaction was transferred by the Toepler pump into the gas burette for the volume determination. The amount of H_2 formed is 2.4 ml (STP) or 0.11 mol per mol of $\text{B}(\text{C}_6\text{F}_5)_3$ (19% of the theoretical value).

3.2. X-ray diffraction analysis of complex **I**

The needle-like crystal ($0.25 \times 0.08 \times 0.05 \text{ mm}^3$) of the solvate $\text{I}\cdot\text{C}_6\text{H}_5\text{CH}_3$ was placed under Ar into a glass capillary and sealed off. The X-ray data were collected at 150 K on an IPDS diffractometer (Stoe) using Mo-K_α radiation ($\lambda = 0.71073 \text{ \AA}$, graphite monochromator, $\theta_{\text{max}} = 25^\circ$). The crystals of $\text{I}\cdot\text{C}_6\text{H}_5\text{CH}_3$ are monoclinic, space group $P2_1/n$, $a = 10.216(3)$, $b = 14.813(6)$, $c = 19.924(7) \text{ \AA}$, $\beta = 91.40(5)^\circ$, $V = 3014(2) \text{ \AA}^3$, $Z = 4$, $D_{\text{calc}} = 1.721 \text{ g cm}^{-3}$. From 20 960 collected reflections, 5043 were independent. The structure was solved by direct methods [13] and refined anisotropically for all non-hydrogen atoms [14]. Hydrogen atoms were placed into calculated positions and refined in the riding mode.

The refinement with 4090 reflections and 469 parameters converged to the final values $wR_2 = 0.0830$ and $R_1 = 0.0484$ (for 2644 reflections with $I > 2\sigma(I)$).

4. Supplementary material

Atomic coordinates, bond lengths and angles, and thermal parameters have been deposited at the Cambridge Crystallographic Data Centre under registration no. 125202. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

Acknowledgements

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References

- [1] W.E. Piers, Chem. Eur. J. 4 (1998) 13.
- [2] W. Ahlers, B. Temme, G. Erker, R. Fröhlich, T. Fox, J. Organomet. Chem. 527 (1997) 191.
- [3] B. Temme, G. Erker, J. Karl, H. Luftmann, R. Fröhlich, S. Kotila, Angew. Chem. Int. Ed. Engl. 34 (1995) 1755.
- [4] M. Bochmann, S.J. Lankaster, O.B. Robinson, J. Chem. Soc. Chem. Commun. (1995) 2081.
- [5] V.V. Burlakov, U. Rosenthal, P.V. Petrovskii, V.B. Shur, M.E. Vol'pin, Metalloorg. Khim. 1 (1988) 953 (Organomet. Chem. USSR, 1 (1988) 526 (Engl. Transl.)).
- [6] V.V. Burlakov, A.V. Polyakov, A.I. Yanovsky, Y.T. Struchkov, V.B. Shur, M.E. Vol'pin, U. Rosenthal, H. Görls, J. Organomet. Chem. 476 (1994) 197.
- [7] V.V. Burlakov, S.I. Troyanov, A.V. Letov, E.I. Mysov, G.G. Furin, V.B. Shur, Izvest. Akad. Nauk, Ser. Khim. (1999) 1022 (Russ. Chem. Bull. 48 (1999) 1012 (Engl. Transl.)).
- [8] J. Ruwwe, G. Erker, R. Fröhlich, Angew. Chem. Int. Ed. Engl. 35 (1996) 80.
- [9] J.M. deWolf, J.M. Gercama, S.I. Troyanov, A. Meetsma, J.H. Teuben, Proceedings of the XVIIth International Conference on Organometallic Chemistry, July 7–12, 1966, Brisbane, Australia.
- [10] X. Yang, C.L. Stern, T.J. Marks, J. Am. Chem. Soc. 116 (1994) 10015.
- [11] A. Ohff, R. Kempe, W. Baumann, U. Rosenthal, J. Organomet. Chem. 520 (1996) 241.
- [12] H. Braunschweig, T. Wagner, Chem. Ber. 127 (1994) 1613.
- [13] G.M. Sheldrick, SHELXS-86, Program for the Solution of Crystal Structures, University of Göttingen, Germany, 1986.
- [14] G.M. Sheldrick, SHELXL-93, Program for the Refinement of Crystal Structures, University of Göttingen, Germany, 1993.