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C_2-Bridged sandwich and half-sandwich entities with Ti(IV) and Cr(0) centers

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Abstract

The intense purple colored bi- and trimetallic complexes $\{Ti\}(CH_2SiMe_3)[C{\equiv}C(\eta^6\text{-}C_6H_5)Cr(CO)_3]$ (**3**) ($\{Ti\} = (\eta^5\text{-}C_5H_5)_2Ti$) and $[Ti][C{\equiv}C(\eta^6\text{-}C_6H_5)Cr(CO)_3]_2$ (**5**) $\{[Ti] = (\eta^5\text{-}C_5H_4SiMe_3)_2Ti\}$, in which next to a Ti(IV) center a Cr(0) atom is present, are accessible by the reaction of $Li[C{\equiv}C(\eta^6\text{-}C_6H_5)Cr(CO)_3]$ (**2**) with $\{Ti\}(CH_2SiMe_3)Cl$ (**1**) or $[Ti]Cl_2$ (**4**) in a 1:1 or 2:1 molar ratio. The chemical and electrochemical properties of **3**, **5**, $\{Ti\}(CH_2SiMe_3)(C{\equiv}CFc)$ $[Fc = (\eta^5\text{-}C_5H_5)Fe(\eta^5\text{-}C_5H_4)]$ and $[Ti][(C{\equiv}C)_nMc][(C{\equiv}C)_mM'c]$ [n, $m = 1, 2$; $n = m$; $n \neq m$; $Mc = (\eta^5\text{-}C_5H_5)Fe(\eta^5\text{-}C_5H_4)$; $M'c = (\eta^5\text{-}C_5H_5)Ru(\eta^5\text{-}C_5H_4)$; $Mc = M'c$; $Mc \neq M'c$] will be comparatively discussed. © 2002 Elsevier Science B.V. All rights reserved.

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1. Introduction

Recently, mono- and bis(alkynyl) titanocenes of type $[Ti](R)(C{\equiv}CR')$ $\{[Ti] = (\eta^5\text{-}C_5H_5)_2Ti$, $(\eta^5\text{-}C_5H_4SiMe_3)_2Ti$, …; $R = Cl$, CH_2SiMe_3; R' = singly bonded organic group, Fc; $Fc = (\eta^5\text{-}C_5H_5)Fe(\eta^5\text{-}C_5H_4)\}$, $[Ti](C{\equiv}CR')(C{\equiv}CR'')$, $[Ti][(C{\equiv}C)_nR']_2$ (R', R'' = singly bonded organic group; $R' = R''$; $R' \neq R''$) and $[Ti][(C{\equiv}C)_nMc][(C{\equiv}C)_mM'c]$ [n, $m = 1, 2$; $n = m$; $n \neq m$; $Mc = M'c$; $Mc \neq M'c$; $Mc = (\eta^5\text{-}C_5H_5)Fe(\eta^5\text{-}C_5H_4)$; $M'c = (\eta^5\text{-}C_5H_5)Ru(\eta^5\text{-}C_5H_4)$] have been the subject of interest, due to their potential use, both, as organometallic π-tweezers [1,2], and as model compounds to study intra- and/or intermolecular electron transfer reactions [1,3].

In this respect, the heterometallic early–late transition metal complexes $[Ti](R)(C{\equiv}CFc)$ and $[Ti][(C{\equiv}C)_nMc][(C{\equiv}C)_mM'c]$ in which linear π-conjugated alkynyl units span different organometallic entities have attracted much attention [2,3]. It was found that by remote electron transfer activation the latter type of molecules undergo a reductive elimination of the corresponding $(C{\equiv}C)_nMc$ or $(C{\equiv}C)_mM'c$ units producing the coupling products $Mc(C{\equiv}C)_n(C{\equiv}C)_mM'c$ ($Mc = M'c$, $Mc \neq M'c$) in which a carbon wire spans the terminal metallocene building blocks Mc and M′c, respectively. [2,3] In these species two sandwich building blocks, [Ti] and Mc/M′c, are present. However, to the best of our knowledge no examples exist in which a sandwich unit like $(\eta^5\text{-}C_5H_4R)_2Ti$ and a half-sandwich entity such as $(\eta^6\text{-}C_6H_5)Cr(CO)_3$ are bridged by a π-conjugated organic building block, which may provide electronic communication between the appropriate transition metal centers.

We here describe the synthesis as well as chemical and electrochemical behavior of these species.

2. Results and discussion

When $\{Ti\}(CH_2SiMe_3)Cl$ (**1**) [4] is reacted with one equivalent of $Li[C{\equiv}C(\eta^6\text{-}C_6H_5)Cr(CO)_3]$ (**2**) [5a] in diethyl ether at −78 °C, the heterobimetallic early–late complex $\{Ti\}(CH_2SiMe_3)[C{\equiv}C(\eta^6\text{-}C_6H_5)Cr(CO)_3]$ (**3**) is produced in 56% yield (Eq. (1)).

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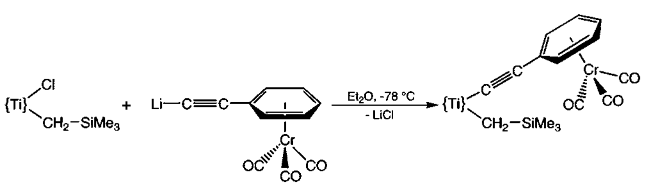
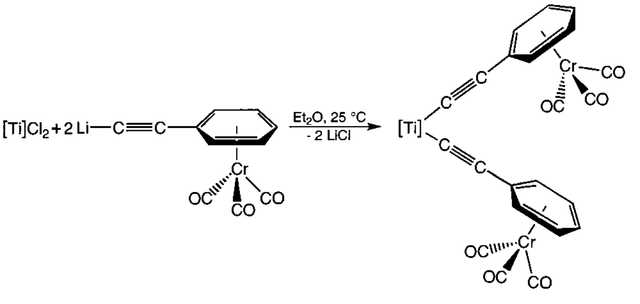


1 **2** **3**

(1)

The reaction of **1** with **2** is accompanied by a color change from orange to intense purple. One distinguished feature of **3** is its high reactivity even at low temperature, both, in solution as well as in the solid-state leading to decomposition products, which, however could not be unequivocally characterized. Nevertheless, the decomposition route postulated for [Ti](CH$_2$SiMe$_3$)(C≡CFc) [2a] which finally produces HC≡CFc is not common for **3**.

A significant higher stability of heteronuclear complexes with both, the [Ti] and (η^6-benzene)Cr(CO)$_3$ entities can be reached by using the titanocenedichloride [Ti]Cl$_2$ (**4**) [6] as the respective starting material. The latter complex reacts with two equivalents of Li[C≡C(η^6-C$_6$H$_5$)Cr(CO)$_3$] (**2**) in diethyl ether at 25 °C to produce trinuclear [Ti][C≡C(η^6-C$_6$H$_5$)Cr(CO)$_3$]$_2$ (**5**) in 65% isolated yield (Eq. 2).



4 **2** **5**

(2)

After appropriate work-up complex **5** can be obtained as a dark purple solid, which is stable for short periods of time at ambient temperatures (−30 °C), while at 25 °C a visible decomposition to products which cannot be further characterised is observed as it is typical for **3**. However, on exposure to air both complexes, **3** and **5**, rapidly decompose. In addition, **3** and **5** are sensitive to light and therefore they should be handled and stored in the dark. Complexes **3** and **5** are highly soluble in polar organic solvents, such as diethyl ether, tetrahydrofuran and dichloromethane.

As expected **3** and **5** exhibit one distinguished C≡C stretching vibration at 2088 (**3**) or 2070 cm^{-1} (**5**) in the IR spectrum. These absorptions appear in a region typical for C$_2$-bridged transition metal complexes [1–3]. The ν_{CO} absorptions, characteristic for Cr(CO)$_3$ moieties are found at 1964 and 1885 cm^{-1} for **3** as well as 1966 and 1885 cm^{-1} for **5** [5,7].

The ^{1}H-NMR spectra of **3** and **5** consists of well-resolved resonance signals for each of the organic groups C$_6$H$_5$, C$_5$H$_5$ and C$_5$H$_4$ present. The most informative feature in the ^{1}H-NMR spectrum for trinuclear **5** is the appearance of a AA′XX′ resonance pattern with two pseudotriplets centered at 6.38 and 6.76 ppm with $J_{HH} = 2.3$ Hz. For **3**, where only η^5-C$_5$H$_5$ units at the titanium center are present, a singlet at 6.27 ppm is found. The C$_6$H$_5$ building blocks of the half-sandwich entities (η^6-C$_6$H$_5$)Cr(CO)$_3$ appear at higher field (**3**: 5.0–5.4 ppm, **5**: 5.1–5.3 ppm), when compared with, e.g. the bis(alkinyl) titanocene [Ti](C≡CPh)$_2$ (7.2–7.4 ppm) [8a]. This shifting is in accordance with the observation generally made by changing form *non*-coordinated to η^6-bonded phenyl entities [5].

Due to the high instability of **3** no expressive ^{13}C{^{1}H}-NMR spectra could be obtained. For the tweezer molecule [Ti][C≡C(η^6-C$_6$H$_5$)Cr(CO)$_3$]$_2$ (**5**) only the required resonance signals for the C$_5$H$_4$SiMe$_3$ and C$_6$H$_5$ groups are found, while the carbon resonance signals for the bridging acetylide unit as well as for the CO ligands of the Cr(CO)$_3$ fragments could not be detected.

As a result of the high instability of **3** and **5** also no satisfactory elemental analyses are available. However, ESI-TOF studies clearly demonstrate that these complexes were formed, since for **3** and **5** the molecular ion [M$^+$ + Na] is observed. Further very characteristic ions are [M$^+$ − CO], [M$^+$ − 2Cr(CO)$_3$]. While for **3** as basis peak [M$^+$ − CO] is typical, for **5** [M$^+$ − 2C$_5$H$_4$SiMe$_3$ − Cr(CO)$_3$ − CO] is the ion with the highest intensity (Section 3).

As reported previously, the organometallic π-tweezer complexes [Ti][(C≡C)$_n$Mc][(C≡C)$_m$M′c] (vide supra) readily release Mc(C≡C)$_n$(C≡C)$_m$M′c via initial electrochemical or chemical oxidation of the appropriate starting material [2,3]. The oxidative coupling of the corresponding acetylide-ferrocenyl or -ruthenocenyl entities to produce the appropriate all-carbon butadiin, hexatriin or octatetrain species occurs instantaneously by an electron transfer from the Ti–C$_{C≡C}$ fragments to the Mc or M′c building blocks through the π-conjugated carbon wires (C≡C)$_n$ and (C≡C)$_m$. In this respect, we have been interested to demonstrate, if complexes **3** and **5** possess similar electrochemical properties as the afore mentioned Ti–Fc and Ti–Rc complexes. This is of interest, since in the latter heterometallic species the electron-donating groups Fc or Rc are replaced by more electron-withdrawing units, e.g. the half-sandwich entity (η^6-benzene)Cr(CO)$_3$. However, cyclic voltammetric studies could only be carried out on trinuclear **5**, since **3** is, as described earlier, even at ambient temperatures not stable in solution. The cyclic voltammogram for **5** recorded at −20 °C is depicted in Fig. 1.

In the anodic region, the cyclic voltammogram of **5** exhibits a quasi-reversible wave at $E = -1.60$ V ($\Delta E =$

100 mV), which can be assigned to the Ti(IV)/Ti(III) redox couple. In comparison with [Ti](C≡CFc)$_2$ in which the Ti(IV)/Ti(III) redox couple appears at $E_{red} = -1.99$ V ($\Delta E = 150$ mV) complex **5** is more difficult to reduce [2d]. This finding points to the fact that the ferrocenyl moieties in the tweezer molecule [Ti](C≡CFc)$_2$ in comparison to [Ti][C≡C(η^6-C_6H_5)Cr(CO)$_3$]$_2$ (**5**) resemble a more electron-donating organometallic group. Thus, the electrochemical response of the Ti(IV) center is shifted to a more positive potential.

Compared to the Ti(IV)/Ti(III) redox potential of the *non*-coordinated tweezer molecule [Ti](C≡CPh)$_2$, which possesses a reversible wave at $E = -1.76$ V ($\Delta E = 150$ mV) [9] this potential is shifted to a more positive value in **5**, indicating a facilitation of the reduction of the titanium center in **5**, and also can be interpreted in means of the more electron-withdrawing effect of the (η^6-benzene)Cr(CO)$_3$ entity.

As typical for other organometallic species in which (η^6-benzene)Cr(CO)$_3$ groups are present, **5** shows no reduction of the chromium(0) center [5c].

In contrast to the reversible Ti(IV)/Ti(III) reduction wave, two irreversible oxidations for the (η^6-benzene)Cr(CO)$_3$ entities are found at +0.57 and +0.95 V. The irreversibility of these waves has been demonstrated by multicyclic experiments in the region of −0.5 to +1.0 V. This indicates that **5** shows a different electrochemical behavior than [Ti](C≡CFc)$_2$ in which a reductive coupling, induced by a remote-side oxidation, produces FcC≡C–C≡CFc [2d]. However, the applied measurement conditions did not result in the formation of (CO)$_3$Cr(η^6-C_6H_5)C≡C–C≡C(η^6-C_6H_5)Cr(CO)$_3$, a compound first synthesized by Müller et al. [5a].

The appearance of irreversible oxidation waves for **5** is not unusual, since other (η^6-C_6H_5)Cr(CO)$_3$ containing organometallic transition metal fragments linked by π-conjugated organic groups such as acetylides display similar features [5c,10]. While for (CO)$_3$Cr(η^6-C_6H_5)CH=CH(η^6-C_6H_5)Cr(CO)$_3$ two quasi-reversible one-electron oxidations (+0.782 and +0.900 V) are predicted, the homobi- and -trinuclear species (CO)$_3$Cr(η^6-C_6H_5)CH=CHC$_6$H$_4$CH=CH(η^6-C_6H_5)Cr(CO)$_3$ as well as (CO)$_3$Cr(η^6-C_6H_5)CH=CH(η^6-C_6H_4)Cr(CO)$_3$CH=CH(η^6-C_6H_5)Cr(CO)$_3$ possess irreversible oxidation potentials [5c].

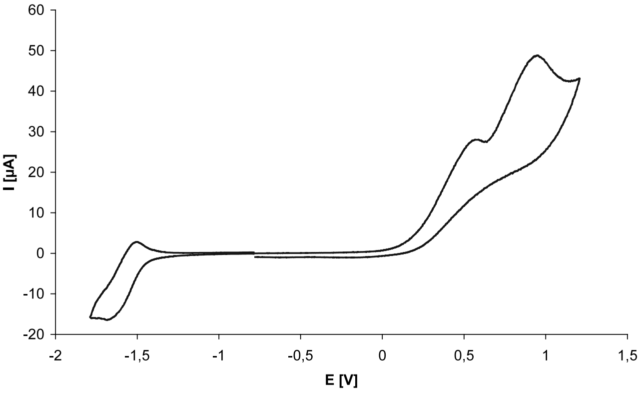


Fig. 1. Cyclic voltammogram of **5** in tetrahydrofuran solution in the presence of [n-Bu$_4$N][PF$_6$] ($c = 0.1$ M) at −20 °C under N$_2$ (scan rate 100 mV s^{-1}); potentials are referenced to FcH/FcH$^+$ as an internal standard ($E_0 = 0.00$ V).

3. Experimental

3.1. General methods

All reactions were carried out under an atmosphere of purified nitrogen (O$_2$ traces: Cu catalyst, BASF AG, Ludwigshafen; H$_2$O: molecular sieve 4 Å, Roth) using standard Schlenk techniques. Tetrahydrofuran, Et$_2$O and n-pentane were purified by distillation from sodium/benzophenone ketyl. Infrared spectra were recorded in a Perkin–Elmer FT-IR 1000 spectrometer. ^{1}H-NMR spectra were recorded in a Bruker Avance 250 spectrometer operating at 250.123 MHz in the Fourier transform mode; ^{13}C{^{1}H}-NMR spectra were recorded at 62.860 MHz. Chemical shifts are reported in δ units (parts per million) downfield from tetramethylsilane with the solvent as the reference signal (CDCl$_3$: ^{1}H-NMR, $\delta = 7.26$; ^{13}C{^{1}H}-NMR, $\delta = 77.0$). Melting points (M.p.) were determined using sealed nitrogen purged capillaries in a Gallenkamp melting point apparatus. Microanalyses were performed by the Department of Organic Chemistry at Chemnitz, the Technical University. Electrochemical measurements were carried out by cyclic voltammetry in [n-Bu$_4$N][PF$_6$] solutions ($c = 0.1$ M) at −20 °C, using a standard three-electrode cell in a Radiometer Copenhagen DEA 101 Digital Electrochemical Analyser with a IMT 102 Electrochemical Interface. All potentials were referenced to the ferrocene/ferrocenium couple, which was used as an internal standard with $E_0 = 0.00$ V. ESI-TOF mass spectra were recorded in a Mariner ESI-TOF mass spectrometer (Applied Biosystems) operating in the positive-ion mode in a CH$_2$Cl$_2$–MeCN solution.

3.2. General remarks

The starting materials {Ti}(CH$_2$SiMe$_3$)Cl (**1**) [4], HC≡C(η^6-C_6H_5)Cr(CO)$_3$ [5a] and [Ti]Cl$_2$ (**4**) [6] were prepared following published procedures. All other chemicals were purchased from commercial sources and were used without any further purification.

3.3. Synthesis of {Ti}(CH$_2$SiMe$_3$)[C≡C(η^6-C$_6$H$_5$)-Cr(CO)$_3$] (**3**)[1]

At −78 °C, 360 mg (1.5 mmol) of $HC\equiv C(\eta^6\text{-}C_6H_5)Cr(CO)_3$ was dissolved in 30 ml of Et_2O under exclusion of light and 0.6 ml (1.5 mmol) of ^{n}BuLi (2.5 M in hexane) was added in one portion. After 15 min of stirring at this temperature 455 mg (1.5 mmol) of $\{Ti\}(CH_2SiMe_3)Cl$ (**1**) was added. After 15 min all volatiles were evaporated in oil-pump vacuo. Afterwards the dark brown residue was extracted with *n*-pentane (2 × 15 ml) and Et_2O (3 × 15 ml). The solvent from the combined Et_2O fractions was evaporated in oil-pump vacuo to produce **3** (350 mg, 56% based on $\{Ti\}(CH_2SiMe_3)Cl$) as a deep purple solid. *Caution:* **3** should be kept at low temperature (−30 °C) and in the dark. Otherwise rapid decomposition is observed.

M.p. (dec.): 92° C. IR (KBr, cm^{-1}): 2088 (w) ($\nu_{C\equiv C}$), 1964 (s) (ν_{CO}), 1885 (s) (ν_{CO}). ^{1}H-NMR ($CDCl_3$): δ 0.00 (s, 9H, Si*Me*$_3$), 1.71 (s, 2H, C*H*$_2$), 5.1 (m, 1H, C$_6$*H*$_5$), 5.2 (m, 1H, C$_6$*H*$_5$), 5.4 (m, 3H, C$_6$*H*$_5$), 6.27 (s, 10H, C$_5$*H*$_5$). ESI-TOF MS; *m*/*z* (relative intensity): 525 (20) [$M^+ + Na$], 473 (100) [$M^+ - CO$], 301 (55) [$M^+ - C_5H_5 - Cr(CO)_3$]. Anal. Calc. for $C_{25}H_{26}CrO_3TiSi$: 502.42.

3.4. Synthesis of [Ti][C≡C(η^6-C$_6$H$_5$)Cr(CO)$_3$]$_2$ (**5**)[2]

To 120 mg (0.5 mmol) of $HC\equiv C(\eta^6\text{-}C_6H_5)Cr(CO)_3$ dissolved in 30 ml of Et_2O, 0.2 ml (0.5 mmol) of ^{n}BuLi (2.5 M in hexane) was added in one portion at 25 °C in the dark. After 15 min of stirring at this temperature 100 mg (0.25 mmol) of $[Ti]Cl_2$ (**4**) was added. This reaction mixture was stirred for 2 h and afterwards filtered through a pad of Celite. After evaporation of all volatiles in oil-pump vacuo complex **5** (139 mg, 65% based on $[Ti]Cl_2$) could be obtained as a deep purple solid.

M.p. (dec.): 96 °C. IR (KBr, cm^{-1}) 2070 (w) ($\nu_{C\equiv C}$), 1966 (s) (ν_{CO}), 1885 (s) (ν_{CO}). ^{1}H-NMR ($CDCl_3$): δ 0.32 (s, 18H, Si*Me*$_3$), 5.2 (m, 2H, C$_6$*H*$_5$), 5.3 (m, 8H, C$_6$*H*$_5$), 6.38 (pt, $J_{HH} = 2.3$ Hz, 4H, C$_5$*H*$_4$), 6.76 (pt, $J_{HH} = 2.3$ Hz, 4H, C$_5$*H*$_4$). ^{13}C{^{1}H}-NMR ($CDCl_3$): δ −0.1 (Si*Me*$_3$), 90.1 (*CH*/C$_6$H$_5$), 92.4 (*CH*/C$_6$H$_5$), 94.1 (*CH*/C$_6$H$_5$), 115.2 (*CH*/C$_5$H$_4$), 122.7 (*CH*/C$_5$H$_4$), 125.5 (^{i}*C*/C$_5$H$_4$), 127.9 (^{i}*C*/C$_6$H$_5$).[1] ESI-TOF MS; *m*/*z* (relative intensity): 819 (20) [$M^+ + Na$], 767 (30) [$M^+ - CO$], 547 (15) [$M^+ - 2Cr(CO)_3 + Na$], 438 (40) [$M^+ - 2C_5H_4SiMe_3 - 3CO$], 358 (100) [$M^+ - 2C_5H_4SiMe_3 - Cr(CO)_3 - CO$], 302.2 (70) [$M - 2C_5H_4SiMe_3 - Cr(CO)_3 - 3CO$]$^+$. Anal. Calc. for $C_{38}H_{36}Cr_2O_6Si_2Ti$: 796.72.

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[1] Due to the high instability of **3** (vide supra) following its decomposition, the signals for the carbon atoms in the ^{13}C{^{1}H}-NMR spectrum could not be unambiguously identified. In addition, no satisfactory elemental analyses could be obtained.

[2] Due to the instability of **5** (vide supra) following its decomposition the signals for the carbonyl- and acetylide carbon atoms could not be detected. In addition, no satisfactory elemental analyses could be obtained.

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