Reaction of the Organometallic Planar Tetracoordinate Carbon Compound $[(Cp_2Zr)_2(\mu\text{-}C{\equiv}CCH_3)(\mu\text{-}(\eta^1\text{-}C{:}\eta^2\text{-}C,C)\text{-}CH_3CCCH_3)]^+[BPh_4]^-$ with Alkyl Isocyanides

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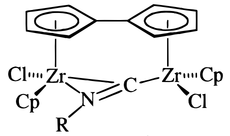
The $[(Cp_2Zr)_2(\mu\text{-}CH_3CCCH_3)(\mu\text{-}C{\equiv}CCH_3)]^+BPh_4^-$ complex (**4**) reacts readily with alkyl isocyanides $RN{\equiv}C$ by displacement of the bridging 2-butyne ligand to give the cationic $[(\mu$-acetylide)(μ-isonitrile)$(Cp_2Zr)_2]$ complexes **6a**−**c** in high yield (∼80%). The X-ray crystal structure analysis of an example (**6c**) revealed the μ-(η^1-C:η^2-C,N) coordination of the isonitrile ligand.

Introduction

Like carbon monoxide, isonitrile ligands can bridge unsymmetrically between two transition metal centers, formally serving as a 4-electron ligand.[1] The μ-(η^1-C:η^2-C,N) (or σ,π) bonding mode of a variety of substituted RNC ligands has been established, mostly employing metal combinations from the center of the periodic table (mostly groups 6 and 7).[2] However, the unsymmetrical μ-isonitrile bonding mode is encountered much less frequently than its "semibridging" μ-CO analogue in metal carbonyl chemistry. Examples from the left side of the periodic table also are extremely sparse. To our knowledge, μ-(η^1-C:η^2-C,N)isonitrile group 4 metal complexes have only very rarely been observed and characterized by X-ray diffraction, with a few examples of dinuclear fulvalene zirconium complexes (**1**) being notable exceptions.[3]



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Most σ,π-bridging isonitrile complexes are synthesized by means of an effective RNC addition to low-valent dimetallic systems or to a metal−metal bond. This makes it, of course, difficult to prepare μ-(η^1-C:η^2-C,N)-isonitrile metal complexes when such organometallic precursors are not generally available or are difficult to generate. We have now found an alternative way of preparing such μ-isonitrile early transition metal complexes that makes use of a ligand substitution reaction. This has, to our knowledge, for the first time allowed the preparation of stable cationic dinuclear σ,π-isonitrile zirconocene complexes. Several typical examples are described in this account.

Results and Discussion

We used the planar tetracoordinate carbon complex **4** as the starting material for this study. The dinuclear hydrocarbon-bridged metallocene cation system is readily available by the reaction of the mononuclear methylzirconocene cation reagent **3** with dipropynylzirconocene. Complex **4** contains a σ-bridging μ-propynyl ligand, as shown by X-ray diffraction and IR ($\tilde{\nu}_{C\equiv C}$ = 2070 cm^{-1}), and a μ-(η^1-C2:η^2-C2,C3)-2-butyne ligand. Carbon atom C2 is planar tetracoordinate.[4] We had previously demonstrated that the μ-2-butyne ligand can easily be displaced by heating the system with, for example, benzonitrile. 2-Butyne is liberated and the dinuclear μ-$N{\equiv}CR$ metallocene cation **5** is formed.[5] In complex **5**, the nitrile ligand is σ,π-bridging (Scheme 1).

We have now found that the cationic complex **4** reacts in a similar manner with alkyl isocyanides. *tert*-Butyl isocyanide (1.5 molar equiv) was added to a solution of **4** in CD_2Cl_2. ^1H NMR spectroscopy revealed that a displacement reaction started instantaneously, at ambient temperature, that led to the liberation of 1 equiv of 2-butyne and the formation of a single new metallocene

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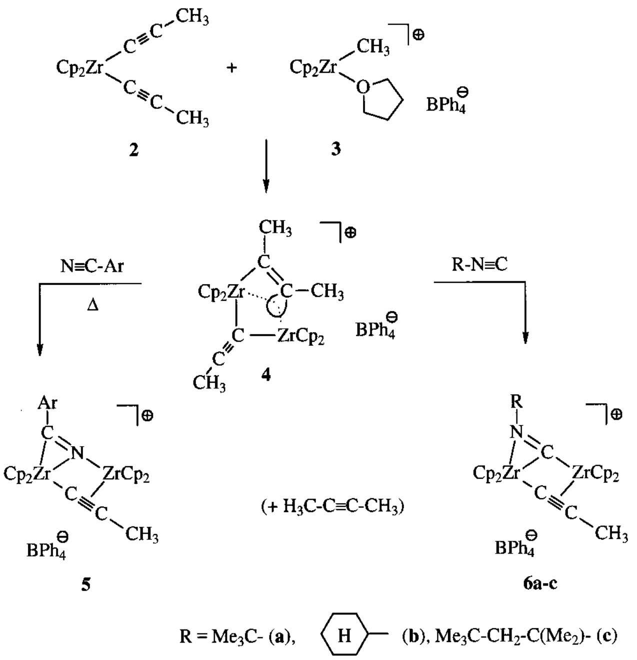
Scheme 1



complex.[6] The reaction was complete within minutes. The reaction was then carried out on a preparative scale (as described in the Experimental Section), and the organometallic product **6a** was isolated (84% yield). The cation of **6a** contains two slightly different metallocene moieties exhibiting ^{1}H/^{13}C NMR signals (in CD_2Cl_2) at δ 5.67, 5.66/106.5, 105.8. The quaternary carbon resonances of the two bridging ligands are at δ 280.1 (CN) and δ 155.4 and 125.7 (CC), respectively. Consequently, there are no IR bands observed in the C≡N or C≡C triple bond regions, but there are two medium intensity IR features at $\tilde{\nu} = 1706$ and 1678 cm^{-1} (in KBr). These tentatively may be assigned to the μ-(η^1-C:η^2-C,N)- and μ-(η^1-C:η^2-C,C)-bridging ligand systems of complex **6a**.

The reaction of **4** with cyclohexyl isocyanide and 1,1,3,3-tetramethylbutyl isocyanide proceeds analogously to give **6b** and **6c**, respectively. Complex **6b** also shows two inequivalent metallocene moieties (^{1}H/^{13}C NMR Cp resonances at δ 5.67, 5.65/106.6, 105.8) (**6c**: δ 5.67, 5.66/106.5, 105.9). In contrast to **6a**, these two complexes only exhibit a broad medium intensity IR band in the typical region (**6b**, 1700 cm^{-1}; **6c**, 1692 cm^{-1}).

Complex **6c** was characterized by an X-ray crystal structure analysis. In the crystal the organometallic cation is well separated from the BPh_4^- anion. The cation of **6c** contains two typical bent metallocene units in chemically different environments. Each of the zirconocene units is distorted tetrahedral around the zirconium, but each zirconium binds to three atoms in the σ-ligand plane. The Zr1···Zr2 separation is 3.53 Å. The zirconium centers are connected by means of an acetylide and an isonitrile ligand. Both of these bridging ligand systems are arranged in the typical bent metallocene σ-ligand plane, i.e., the plane bisecting the Cp—Zr—Cp angles (see Figure 1).

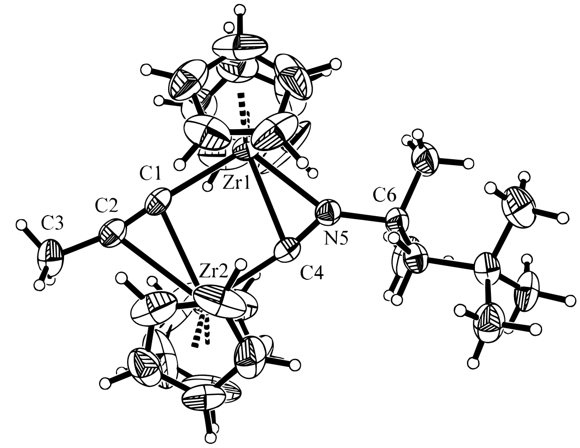


Figure 1. View of the molecular structure of **6c** (only the cationic part is depicted).

Table 1. Selected Bond Lengths (Å) and Angles (deg) of 6c

Zr(1)—C(1)	2.271(4)	Zr(2)—C(4)	2.148(4)
Zr(1)—C(4)	2.413(4)	C(1)—C(2)	1.220(5)
Zr(1)—N(5)	2.280(3)	C(2)—C(3)	1.477(6)
Zr(2)—C(1)	2.468(4)	C(4)—N(5)	1.218(4)
Zr(2)—C(2)	2.564(4)	N(5)—C(6)	1.528(5)
C(1)—Zr(1)—C(4)	80.9(2)	Zr(2)—C(2)—C(1)	71.6(2)
C(1)—Zr(1)—N(5)	110.8(2)	Zr(2)—C(2)—C(3)	121.3(3)
C(4)—Zr(1)—N(5)	29.9(2)	C(1)—C(2)—C(3)	167.1(4)
C(1)—Zr(2)—C(2)	28.0(2)	Zr(1)—C(4)—Zr(2)	101.1(2)
C(1)—Zr(2)—C(4)	82.0(2)	Zr(1)—C(4)—N(5)	69.0(2)
C(2)—Zr(2)—C(4)	110.0(2)	Zr(2)—C(4)—N(5)	170.1(3)
Zr(1)—C(1)—Zr(2)	96.0(2)	Zr(1)—N(5)—C(4)	81.1(2)
Zr(1)—C(1)—C(2)	176.4(3)	Zr(1)—N(5)—C(6)	149.1(2)
Zr(2)—C(1)—C(2)	80.4(3)	C(4)—N(5)—C(6)	129.7(3)

The propynyl ligand is σ-bonded to Zr1 [C1—Zr1 2.271(4) Å] and unsymmetrically π-bonded to Zr2 [C1—Zr2 2.468(4) Å, C2—Zr2 2.564(4) Å, C1—C2 1.220(5) Å, C2—C3 1.477(6) Å; angles Zr1—C1—C2 176.4(3)°, C1—C2—C3 167.1(4)°].[7] The alkyl isocyanide ligand is also σ,π-bonded, with typical bond lengths and angles of 2.148(4) (Zr2—C4), 1.218(4) (C4—N5), 2.413(4) (Zr1—C4), 2.280(3) (Zr1—N5), and 1.528(5) Å (N5—C6) and 170.1(3)° (Zr2—C4—N5). The C4—N5—C6 angle is 129.7(3)°; together with the adjacent angles of 81.1(2)° (Zr1—N5—C4) and 149.1(2)° (Zr1—N5—C6) this makes a perfect trigonal planar coordination geometry at N5. The binding of three neighboring atoms in the Cp—Zr—Cp bisecting plane results in rather large maximal bond angles in the major plane of each metallocene unit [C1—Zr1—N5 110.8(2)°, C2—Zr2—C4 110.0(2)°].

The results of this study provide additional evidence for the high ability of triple bond-containing bridging ligand systems to stabilize electron-deficient early transition metal centers. The examples **6** are especially noteworthy in this respect as these very stable complexes contain not only two electrophilic group 4 metal centers but also a positive charge. We find it remarkable that the μ-(η^1-C:η^2-C,N)isonitrile complexes **6** can so easily be formed by a simple ligand displacement reaction starting from the readily available precursors **4**. Although a description of the detailed mechanistic features of this transformation must await further detailed investigation, it appears that we have opened a simple synthetic route for preparing dinuclear μ-(σ,π-

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(6) In this case, C—C coupling between the isonitrile and the coordinated π-ligand was not observed. For an example of such a reaction, see: Hessen, B.; Blenkers, J.; Teuben, J. H.; Helgesson, G.; Jagner, S. *Organometallics* **1989**, *8*, 830.

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Table 2. Atomic Coordinates and Equivalent Isotropic Displacement Parameters (Å^2) for 6c

	x	y	z	U(eq)[a]
Zr(1)	0.6985(1)	0.1852(1)	0.4403(1)	0.035(1)
Zr(2)	0.6505(1)	0.4116(1)	0.3660(1)	0.032(1)
C(1)	0.7444(2)	0.3363(3)	0.4652(2)	0.035(1)
C(2)	0.7661(2)	0.4191(3)	0.4753(2)	0.037(1)
C(3)	0.8050(3)	0.5106(3)	0.5013(2)	0.051(1)
C(4)	0.6109(2)	0.2668(3)	0.3449(2)	0.032(1)
N(5)	0.5967(2)	0.1816(2)	0.3424(2)	0.034(1)
C(6)	0.5364(2)	0.1238(3)	0.2885(2)	0.037(1)
C(7)	0.4524(2)	0.1659(3)	0.2863(2)	0.035(1)
C(8)	0.3708(2)	0.1184(3)	0.2478(2)	0.040(1)
C(9)	0.5475(3)	0.0206(3)	0.3092(3)	0.067(2)
C(10)	0.5611(3)	0.1403(4)	0.2228(2)	0.059(1)
C(11)	0.3510(3)	0.0301(3)	0.2852(3)	0.065(1)
C(12)	0.3678(3)	0.0904(3)	0.1752(2)	0.054(1)
C(13)	0.3041(2)	0.1911(3)	0.2454(2)	0.055(1)
C(20)	0.7719(4)	0.1149(8)	0.3606(3)	0.123(4)
C(21)	0.8236(4)	0.1855(5)	0.3975(4)	0.084(2)
C(22)	0.8493(3)	0.1587(4)	0.4619(3)	0.065(1)
C(23)	0.8184(3)	0.0742(4)	0.4699(3)	0.073(2)
C(24)	0.7704(3)	0.0446(4)	0.4103(4)	0.092(2)
C(25)	0.6269(4)	0.2323(4)	0.5289(3)	0.065(2)
C(26)	0.6963(3)	0.1841(5)	0.5616(2)	0.070(2)
C(27)	0.6869(5)	0.0931(5)	0.5417(3)	0.086(2)
C(28)	0.6145(5)	0.0821(4)	0.4953(3)	0.092(2)
C(29)	0.5752(3)	0.1697(5)	0.4870(2)	0.071(2)
C(30)	0.6635(5)	0.5044(5)	0.2665(4)	0.095(2)
C(31)	0.7350(7)	0.5192(6)	0.3171(4)	0.114(3)
C(32)	0.7765(3)	0.4360(8)	0.3254(3)	0.105(3)
C(33)	0.7310(4)	0.3705(4)	0.2838(3)	0.075(2)
C(34)	0.6614(3)	0.4135(5)	0.2476(2)	0.069(2)
C(35)	0.5358(3)	0.5288(3)	0.3460(2)	0.057(1)
C(36)	0.5879(3)	0.5503(3)	0.4089(3)	0.062(1)
C(37)	0.5851(3)	0.4776(4)	0.4521(2)	0.066(2)
C(38)	0.5335(3)	0.4098(4)	0.4174(3)	0.068(2)
C(39)	0.5030(3)	0.4412(4)	0.3515(3)	0.062(1)

[a] U(eq) is defined as one-third of the trace of the orthogonalized $\mathbf{U}_{ij}$ tensor.

isonitrile) early transition metal complexes, even in cases where suitable metal−metal bond-containing starting materials are lacking.

Experimental Section

All experiments were performed under argon by standard glove box and Schlenk techniques. Solvents were purified by distillation under argon from appropriate drying agents before use. The NMR spectra were obtained with Bruker AC 200 P or AM 360 instruments. IR spectra were recorded with a Nicolet 5 DXC FT-IR spectrometer. Melting points were determined by DSC (DuPont 2910 DSC, STA Instruments). Complex **4** was prepared according to a literature procedure.[4a]

(μ-(η^1-C:η^2-C,N)-*tert*-Butyl isocyanide)(μ-(η^1-C:η^2-C,C)-propynyl)bis(zirconocene) Tetraphenylborate (6a). To a solution of **4** (230 mg, 0.27 mmol) in 10 mL of CH_2Cl_2 was added *tert*-butyl isocyanide (80 mg, 0.96 mmol) via syringe. After 1 h, pentane (10 mL) was added dropwise, and a yellow solid precipitated. The solid was collected by filtration, washed with pentane (10 mL), and dried in vacuo to yield 200 mg (84%) of **6a**: mp 199 °C (dec); ^{1}H NMR (360 MHz, 300 K, CD_2Cl_2) δ 5.67 (s, 10 H, Cp), 5.66 (s, 10 H, Cp), 2.44 (s, 3 H, $\equiv CCH_3$), 1.67 (s, 9 H, $C(CH_3)_3$); ^{1}H NMR BPh_4^- δ 7.40−7.30 (m, 8 H, Ph), 7.10−7.00 (m, 8 H, Ph), 7.00−6.85 (m, 4 H, Ph); ^{13}C NMR (50 MHz, 300 K, CD_2Cl_2) δ 280.1 (*C*N), 154.4, 125.7 ($C\equiv C$), 106.5 (Cp), 105.8 (Cp), 67.4 ($C(CH_3)_3$), 31.2 ($^1J_{CH}$ = 127 Hz, $C(CH_3)_3$), 13.5 ($^1J_{CH}$ = 133 Hz, $\equiv CCH_3$); ^{13}C NMR BPh_4^- δ 163.5 ($^1J_{CB}$ = 50 Hz), 136.4 ($^1J_{CH}$ = 153 Hz), 126.0 ($^1J_{CH}$ = 154 Hz), 122.1 ($^1J_{CH}$ = 157 Hz); IR (KBr) $\tilde{\nu}$ 3109, 3054, 3029, 2981, 1706, 1678, 1580, 1479, 1427, 1366, 1172, 1015, 809, 742, 706 cm^{-1}. Anal. Calcd for $C_{52}H_{52}BNZr_2$: C, 70.63; H, 5.93. Found: C, 69.75; H, 5.86.

(μ-(η^1-C:η^2-C,N)Cyclohexyl isocyanide)(μ-(η^1-C:η^2-C,C)-propynyl)bis(zirconocene) Tetraphenylborate (6b). To a solution of **4** (227 mg, 0.27 mmol) in 15 mL of CH_2Cl_2 was added cyclohexyl isocyanide (90 mg, 0.82 mmol). After 15 min, pentane (20 mL) was added and the solution was stirred for 12 h, during which time a yellow solid precipitate formed. The solid was collected by filtration, washed with toluene (10 mL) and pentane (10 mL), and dried in vacuo to yield 196 mg (81%) of **6b**: mp 187 °C (dec); ^{1}H NMR (200 MHz, 300 K, CD_2Cl_2) δ 5.67 (s, 10 H, Cp), 5.65 (s, 10 H, Cp), 3.88 (m, 1 H, cyclohexyl-CH), 2.46 (s, 3 H, $\equiv CCH_3$), 2.10−1.25 (m, 10 H, cyclohexyl-CH_2); ^{1}H NMR BPh_4^- δ 7.40−7.30 (m, 8 H, Ph), 7.10−7.00 (m, 8 H, Ph), 7.00−6.85 (m, 4 H, Ph); ^{13}C NMR (91 MHz, 300 K, CD_2Cl_2) δ 278.4 (*C*N), 154,5, 125.7 ($C\equiv C$), 106.6 (Cp), 105.8 (Cp), 70.5 (N−*C*H), 35.3, 25.7, 25.6 (CH_2), 13.6 ($\equiv CCH_3$); ^{13}C NMR BPh_4^- δ 164.6 ($^1J_{CB}$ = 50 Hz), 136.4, 125.9, 122.1; IR (KBr) $\tilde{\nu}$ 3110, 3033, 2931, 2855, 1700, 1578, 1480, 1446, 1425, 1262, 1098, 1016, 803, 734, 707, 612 cm^{-1}. Anal. Calcd for $C_{54}H_{54}BNZr_2$: C, 71.25; H, 5.98. Found: C, 70.54; H, 6.10.

(μ-(η^1-C:η^2-C,N)-1,1,3,3-Tetramethylbutyl isocyanide)(μ-(η^1-C:η^2-C,C)propynyl)bis(zirconocene) Tetraphenylborate (6c). To a solution of **4** (206 mg, 0.24 mmol) in 15 mL of CH_2Cl_2 was added 1,1,3,3-tetramethylbutyl isocyanide. After 2 h, pentane (20 mL) was added dropwise, and a yellow solid precipitated. The solid was collected by filtration, washed with toluene (10 mL) and pentane (10 mL), and dried in vacuo to yield 169 mg (76%) of **6c**: mp 154 °C (dec); ^{1}H NMR (360 MHz, 300 K, CD_2Cl_2) δ 5.67 (s, 10 H, Cp), 5.66 (s, 10 H, Cp), 2.43 (s, 3H, $\equiv CCH_3$), 1.97 (s, 2H, CH_2), 1.74 (s, 6 H, CH_3), 1.23 (s, 9 H, CH_3); ^{1}H NMR BPh_4^- δ 7.40−7.30 (m, 8 H, Ph), 7.10−7.00 (m, 8 H, Ph), 7.00−6.85 (m, 4 H, Ph); ^{13}C NMR (91 MHz, 300 K, CD_2Cl_2) δ 279.5 (*C*N), 154.5, 125.8 ($C\equiv C$), 106.5 (Cp), 105.9 (Cp), 72.4 (N−$C(CH_3)_2$), 58.2 ($C(CH_3)_2CH_2C(CH_3)_3$), 32.5 ($C(CH_3)_2CH_2C(CH_3)_3$), 32.0 ($C(CH_3)_2CH_2C(CH_3)_3$), 30.2 ($C(CH_3)_2CH_2C(CH_3)_3$), 13.5 ($\equiv CCH_3$); ^{13}C NMR BPh_4^- δ 164.5 ($^1J_{CB}$ = 50 Hz), 136.4, 126.0, 122.1; IR (KBr) $\tilde{\nu}$ 3112, 3054, 3029, 2961, 1692, 1580, 1480, 1427, 1369, 1262, 1242, 1114, 1018, 806, 733, 704, 613, 463 cm^{-1}.

X-ray crystal structure analysis of 6c: $C_{56}H_{60}BNZr_2$, M = 940.30; yellow plates, crystal dimensions 0.50 × 0.40 × 0.25 mm; monoclinic, space group $P2_1/n$ (No. 14), T = 223(2) K, a = 17.003(2) Å, b = 14.031(1) Å, c = 20.547(6) Å, β = 105.37(2)°, V = 4727(2) Å^3, Z = 4, d_{calc} = 1.321 g cm^{-3}; μ(Mo Kα) = 4.78 cm^{-1}, F(000) = 1952, Enraf-Nonius Mach 3 diffractometer, λ = 0.710 73 Å, graphite monochromator, $2\theta-\omega$ scan mode, θ-range 2.32−25.00°, index ranges $-20 \le h \le 21$, $-17 \le k \le 0$, $-25 \le l \le 0$; 8555 reflections collected, 8309 independent reflections (R_{int} = 0.057); semiempirical absorption correction from Ψ-scans (max and min corrections 0.999 and 0.965); structure solution by direct methods (SHELXS-86),[8] refinement by full-matrix least-squares on F^2 (SHELXL-93);[8] final R indices for 5366 observed [$I > 2\sigma(I)$] reflections and 547 parameters R = 0.040 and wR^2 = 0.096; largest difference peak and hole 0.98 and −0.50 e Å^{-3}. The figure was drawn with the XP program.[8]

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Supporting Information Available: Details of the X-ray crystal structure analysis of **6c**, including listings of the atomic coordinates and bond lengths and angles (16 pages). Ordering information is given on any current masthead page.

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