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Hydrocarbon bridged metal complexes
XLV. Dinuclear polyene-bridged Fischer carbene complexes and a star-shaped benzene-bridged tris(ferrocenyl-decapentaenyl) compound☆

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Dedicated to Professor Ernst Otto Fischer on the occasion of his 80th birthday

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Abstract

Condensation of carotinoid polyene dialdehydes, 1,1′-ferrocene dialdehydes and of 9-ferrocenyl-2,7-dimethylnonatetraenal with the Fischer carbene complexes $(OC)_5W{=}C(NMe_2)CH_2SiMe_3$ or $(OC)_5M{=}C(Me)(OMe)$ (M=Cr, W) in the presence of *n*-BuLi or $SiMe_3Cl/NEt_3$ yields the bis(carbene) complexes **1**–**4** and the donor acceptor substituted complexes **5**, **6**. The star-shaped trinuclear molecules **7** and **8** were obtained under Wittig conditions from 1,3,5-tris[(triphenylphosphonio)methyl]benzene tribromide and ferrocene aldehyde or 9-ferrocenyl-2,7-dimethyl-nonatetraenal. © 1999 Elsevier Science S.A. All rights reserved.

Keywords: Ferrocenyl; Polyene; Carbene

The interest in symmetrical bimetallic or hetero bimetallic complexes with electron conducting hydrocarbon bridges is growing rapidly due to their potential applications in material science (e.g. compounds with metal-metal-communication [2a] or for non-linear optics [2b]). Several methods to introduce Fischer type carbene complexes into π-systems have been described [3]. Coupling reactions of $(OC)_5Cr{=}C(OMe)CH_2Li$ with Cu^I- or Ag^I-salts and subsequent oxidation of the formed σ–C–C-bond led to symmetrical polyene bridged bis(carbene) complexes [4,5]. In many cases reactions of dilithiated aromatic precursors with $M(CO)_6$ (M=Cr, W) lead to symmetrical bis(carbene) complexes [6]. Also formation of μ-bis(aminocarbene) dimetal complexes of chromium and iron by reaction of tertiary diamides and $Cr(CO)_5^{2-}$ and $Fe(CO)_4^{2-}$ in the presence of chlorotrimethylsilane was reported [7]. Photolysis reactions of $W(CO)_6$ with dialkynol derivates provide bis(aminocarbene) complexes [8]. Metathesis reactions of polyenes with Schrock-type metal carbenes yielded symmetrical bis(carbene) complexes [9]. Conjugated polyene bis(carbene) complexes were also synthesized by oxidation of diiron–carbon-σ-bound compounds [10] or from a chromium Fischer carbene complex and tetrachlorocyclopropene [11]. Oxidation of acetylide bridged complexes gave a series of interesting bis(carbene) compounds $M{=}C{=}C{=}(C)_n{=}C{=}C{=}M$ [12, 3b,c]. Other bimetallic complexes with π-conjugated carbon chains contain metal-carbene, metal-carbyne and C≡C bonds [13].

Another approach to synthesize polyene bridged bis(carbene) complexes is the use of polyene dialdehydes as precursors. Aumann [14] and Macomber [15] developed

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☆ For part XLIV see Ref. [1].

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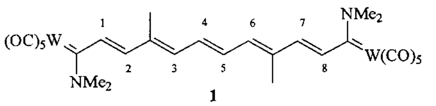
synthetic routes which allow condensation of aldehyde groups with the methyl group of the Fischer carbene complex. In continuation of our studies on polyene bridged Schiff base complexes [16] and of Fischer bis-(carbene) complexes with conjugated C=C-bonds [5] we used slightly modified techniques of Aumann [14] and Macomber [15] for the synthesis of symmetrical bimetallic bis(carbene) complexes and of trinuclear bis-(carbene) complexes of the form $L_mM{=}C(R){-}C_{conj}{-}M'{-}C_{conj}{-}(R)C{=}ML_m$ (M=W, M′=Fe, R=OMe, NMe_2), which are rare [13a].

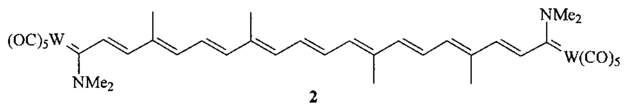
Several star-shaped trimetallic acetylene bridged complexes have been described [17,18] where the electronic metal–metal interaction was of interest. We synthesized two new star-shaped ferrocene containing polyene bridged trimetallic complexes using Wittig conditions.

1. Results and discussion

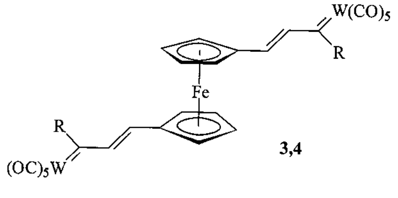
The bis(carbene) complexes **1** and **2** were obtained by a two step reaction, beginning with the reaction of $(OC)_5W{=}C(NMe_2)CH_2SiMe_3$ with *n*-BuLi in THF at − 78°C. Then the dialdehydes 2,7-dimethyloctatrienedial and crocetine dialdehyde were added to the resulting solution. Subsequent purification by chromatography lead to the orange or red air sensitive products. To our knowledge complex **2** is the longest polyene bridged bis(carbene) complex up to date. Use of 1,1′-ferrocene dialdehyde as aldehyde component leads to formation of the symmetrical π-bridged trinuclear bis(carbene) complex **3**. The complexes **4**–**6** were obtained by one pot reactions of $(OC)_5M{=}C(OMe)CH_3$ (M=Cr, W) with 9-ferrocenyl-2,7-dimethyl-nonatetraenal [19] or 1,1′-ferrocene dialdehyde and NEt_3/Me_3SiCl in diethyl ether. The complexes were isolated in moderate yields after purification by column chromatography as air sensitive black microcrystalline products.



1

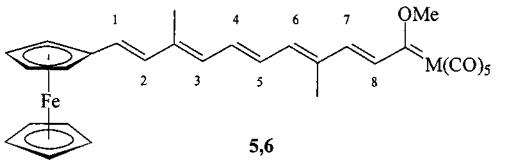


2



3,4

	R
3	NMe_2
4	OMe

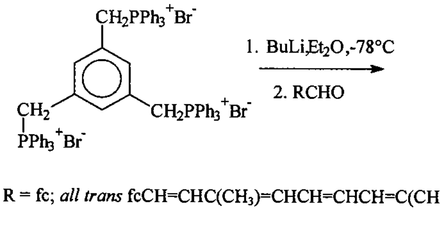


5,6

	M
5	Cr
6	W

In the infrared spectra the A_1–CO-absorptions of **1-6** are shifted to lower wavenumbers due to the electron pushing effect of the polyene ligands. This effect is marked for the methoxy substituted complexes **4**, **5** and **6** with shifts of 13, 10 and 13 cm^{-1} compared to 5 cm^{-1} for **1**, **2** and **3**, since the $(OC)_5M{=}C(OMe)$ complex fragment is a better electron acceptor than its NMe_2 substituted analogue. The NMR spectra of the complexes **1**–**4** are very simple due to the high symmetry of the molecules. For example, the ^{13}C-NMR spectrum of **1** only shows five resonances for the olefinic carbon atoms. Unexpectedly in the ^1H-NMR spectra the Cp protons of **3** and **4** appear as singuletts.

Furthermore, we synthesized new olefin bridged complexes combining the cyclopentadienylring of ferrocene with polyene systems. The synthesis of the complexes **7** and **8** was achieved by Wittig reactions of ferrocenemonoaldehyde and 9-ferrocenyl-2,7-di-methyl-nonatetraenal [19] with 1,3,5-tris[(triphenylphosphonio)methyl]benzene tribromide [20]. The products were purified by column chromatography and were obtained as *cis*/*trans* isomers with rates of 2:3 for **7** and 1:2 for **8**. These mixtures could not be separated. Probably all possible isomer combinations (e.g. *cis*, *trans*, *trans*; *cis*, *cis*, *trans* etc.) were present. **7** and **8** were characterized by NMR and mass spectra. Hopes that the metal centers would electronically communicate with each other were not fulfilled. Cyclovoltammetric studies of **7** and **8** showed only one redox wave for both compounds (for **7**: redoxpotential = 530 mV, CH_2Cl_2, nBu_4NPF_6, scan rate 50 mV s^{-1}, ferrocene as internal reference, 440 mV). A single step oxidation process which implies that the iron centres do not electronically communicate with each other over the benzene bridge was also observed for 1,3,5-tris(ferrocenyl-ethynyl)benzene [17a]. However significant electronic communication between the metal centres was observed, e.g. for $[(Cp^*)(dppe)Fe(C{\equiv}C{-})]_3(1,3,5\text{-}C_6H_3)$ [18a] and for $[(Cp)(Ph_3P)_2Ru(C{\equiv}C{-})]_3(1,3,5\text{-}C_6H_3)$ [18b].



R = fc; *all trans* $fcCH{=}CHC(CH_3){=}CHCH{=}CHCH{=}C(CH_3)$

7

8

2. Experimental

All operations were carried out under nitrogen using Schlenk techniques. Solvents were dried by distillation from sodium/benzophenone or calcium hydride. NMR spectra: Jeol GSX 270 (^{1}H: 270.17 MHz; ^{13}C: 67.94 MHz; ^{31}P: 109.38 MHz) or Jeol EX 400 (^{1}H: 399.78 MHz; ^{13}C: 100.53 MHz). -IR: Perkin-Elmer 841, Nicolet 520 FT-IR.-UV/vis Philips PU 8710. Since the described compounds had to be purified by column chromatography the yields are moderate.

2.1. General procedure for the synthesis of the complexes ***1–3***

An equimolar amount of *n*-BuLi (1.6 M in hexane) is added dropwise to a solution of $(OC)_5W{=}C(N(CH_3)_2)CH_2Si(CH_3)_3$ [14] in 15 ml of THF at −78°C. After stirring for 1 h 0.5 equivalents of the aldehyde compound are added. After stirring for 1.5 h at −78°C the mixture is warmed up to room temperature and the solvent is evaporated in vacuo. The crude residue is dissolved in a minimum of dichloromethane and purified by chromatography using a silica gel column (20 × 2 cm) with *n*-pentane/dichloromethane (4:1) as eluent. The second band yields an oily product after evaporation. Stirring in *n*-pentane at −20°C and centrifugation gives fine powders.

2.1.1. Reaction of 2,7-dimethyl-octatriene-dial with $(OC)_5W{=}C(N(CH_3)_2)CH_2Si(CH_3)_3$

1: Orange powder; yield 110 mg (24%). -IR (KBr, cm^{-1}): $\tilde{\nu}$(CO) = 2059 m (A_1), 1972 w (sh) (B_1), 1931 s (sh), 1902 versus (E).-^{1}H-NMR (400 MHz, $CDCl_3$): δ6.64 (dd, 2H, $H_{3,6.}$), 6.58 (d, 2H, $^3J = 16.1$, $H_{2,7.}$), 6.27–6.25 (m, 2H, $H_{4,5.}$), 5.81 (d, 2H, $^3J = 15.6$, $H_{1,8.}$), 3.81 (s, 6H, NCH_3), 3.38 (s, 6H, NCH_3), 1.95 (s, 6H, CH_3).-^{13}C-NMR: (67.8 MHz, $CDCl_3$) δ252.19 (W=C), 203.56 (CO_{eq}), 198.53 (CO_{ax}),137.78, 134.89, 134.34, 130.43, 129.84 ($C_{olef.}$),53.56, 44.14 ($N(CH_3)_2$), 12.55 (CH_3).-UV/vis (CH_2Cl_2, nm, lg ε): $\lambda_{max} = 397$ (5.06), 431 sh (4.99).-$C_{28}H_{26}N_2O_{10}W_2$ (918.1): Calc. C 36.62, H 2.85, N 3.05; Found C 36.46, H 3.25, N 2.76.

2.1.2. Reaction of crocetinedial with $(OC)_5W{=}C(N(CH_3)_2)CH_2Si(CH_3)_3$

2: Red powder; yield 50 mg (20%).-IR (KBr, cm^{-1}): $\tilde{\nu}$(CO) = 2059 s (A_1), 1967 m (B_1), 1908 versus br (E).-^{1}H-NMR (270 MHz, $CDCl_3$) δ6.71–6.54 (m, 6H, $H_{olef.}$), 6.41 (d, 2H, $^3J = 15.1$, $H_{olef.}$), 6.31–6.18 (m, 4H, $H_{olef.}$), 5.85 (d, 2H, $^3J = 15.8$, $H_{olef.}$), 3.82 (s, 6H, NCH_3), 3.39 (s, 6H, NCH_3), 1.99–1.95 (m, 12H, CH_3). -UV/vis (CH_2Cl_2, nm, lg ε): $\lambda_{max} = 461$ (4.92), 576 sh (4.02). −$C_{38}H_{38}N_2O_{10}W_2$ (1050.3): Calc. C 43.45, H 3.64, N 2.66; Found C 44.47, H 4.41, N 2.07. A molecular ion could not be detected in the FAB MS.

2.1.3. Reaction of 1,1′-ferrocene-dialdehyde with $(OC)_5W{=}C(N(CH_3)_2)CH_2Si(CH_3)_3$

3: Orange powder; yield 100 mg (40%). -IR (KBr, cm^{-1}): $\tilde{\nu}$(CO) = 2059 m (A_1), 1971 w (B_1), 1903 versus (E). -^{1}H-NMR (400 MHz, $CDCl_3$) δ6.67 (d, 1H, $^3J = 16.2$, $H_{olef.}$), 6.06 (d, 1H, $^3J = 16.3$, $H_{olef.}$), 4.39 (s, 8H, C_5H_4), 3.81 (s, 6H, NCH_3), 3.35 (s, 6H, NCH_3). -UV/vis (CH_2Cl_2, nm, lg ε): $\lambda_{max} = 333.4$ (4.13), 365.2 (4.01), 476.4 (3.17).-$C_{30}H_{24}FeN_2O_{10}W_2$ (996.0): Calc. C 36.17, H 2.43, N 2.81; Found C 36.23, H 2.34, N 2.61.

2.2. General procedure for the synthesis of the complexes ***4–6***

A total of 0.3 mmol of $(OC)_5M{=}C(OCH_3)CH_3$ (M=Cr, W) and 0.3 mmol (for **4**: 0.15 mmol) of the aldehyde component are combined with 150 µl Me_3SiCl and 200 µl NEt_3 in 10 ml diethylether. The Schlenk tube is closed with a glass cap and stirred gently for 3–5 d. The mixture is then centrifugated, and after separation of the precipitate the solvent is evaporated in vacuo. The residue is dissolved in a minimum of toluene, and chromatographed using a silica gel column (30 × 2 cm) with *n*-pentane until all unreacted $(OC)_5M{=}C(OCH_3)CH_3$ is removed. The polarity of the mobile phase is slowly increased up to *n*-pentane/diethylether (4:1). The second fractions (purple) contain the products, which are dried after removing the solvent in vacuo.

2.2.1. Reaction of 1,1′-ferrocene-dialdehyde with $(OC)_5W{=}C(OCH_3)CH_3$

4: Dark red powder; yield 100 mg (18%). -IR (KBr, cm^{-1}): $\tilde{\nu}$(CO) = 2061 s (A_1), 1980 w (B_1), 1913 versus (E). -^{1}H-NMR (400 MHz, $CDCl_3$) δ7.40 (d, 1H, ^3J = 15.0, $H_{olef.}$), 7.22 (d, 1H, ^3J = 15.5, $H_{olef.}$), 4.60 (s, 8H, C_5H_4), 4.56 (s, 6H, OCH_3). -UV/vis (CH_2Cl_2, nm, lg ε): λ_{max} = 347.6 (4.65), 445.6 (4.57), 565.4 (4.30). –$C_{28}H_{18}FeO_{12}W_2$ (969.7) Calc. C 34.67, H 1.87; Found C 35.05, H 2.12.

2.2.2. Reaction of 9-ferrocenyl-2,7-dimethyl-nona-tetraenal [18] and $(OC)_5Cr{=}C(OCH_3)CH_3$

5: Black powder; yield 25 mg (14%). -IR (KBr, cm^{-1}): $\tilde{\nu}$(CO) = 2054 m (A_1), 1986 s (B_1), 1944 s (E). -^{1}H-NMR (400 MHz, $CDCl_3$) δ7.38 (d, 1H, ^3J = 14.7, H_8), 6.91-6.84 (m, 2H, $H_{5,7}$), 6.74-6.65 (m, 2H, $H_{3,4}$), 6.53 (d, 1H, ^3J = 15.8, H_2), 6.49 (d, 1H, ^3J = 15.8, H_1), 6.29 (d, 1H, ^3J = 11.6, H_6), 4.71 (s, 3H, OCH_3), 4.44 (t, 2H, C_5H_4), 4.32 (t, 2H, C_5H_4), 4.13 (s, 5H, C_5H_5), 2.04/1.97 (each s, each 3H, CH_3). -^{13}C-NMR (100 MHz, $CDCl_3$) δ217.36 (CO), 145.52, 140.41, 138.0, 135.81, 133.91, 130.76, 129.98, 129.30, 128.45 ($C_{olef.}$), 83.24, 69.51, 65.70 (C_5H_4), 69.39 (C_5H_5), 67.06 (OCH_3), 12.86, 12.37 (CH_3). –$C_{29}H_{26}CrFeO_6$ (578.3) Calc. C 60.17, H 4.52; Found C 59.31, H 4.30.

2.2.3. Reaction of 9-ferrocenyl-2,7-dimethyl-nona-tetraenal [18] with $(OC)_5W{=}C(OCH_3)CH_3$

6: Black powder; yield 50 mg (23%). -IR (KBr, cm^{-1}): $\tilde{\nu}$(CO) = 2061 s (A_1), 1976 w (B_1), 1921 versus (E). -^{1}H-NMR (400 MHz, $CDCl_3$) δ7.31 (d, 1H, ^3J = 14.4, H_8), 7.08 (d, 1H, ^3J = 14.4, H_7), 6.93 (dd, 1H, ^3J = 14.0, ^3J = 11.8, H_5), 6.78 (d, 1H, ^3J = 11.9, H_3), 6.66 (dd, 1H, ^3J = 14.0, ^3J = 12.1, H_4), 6.52 (s, 2H, $H_{1,2}$), 6.28 (d, 1H, ^3J = 11.8, H_6), 4.57 (s, 3H, OCH_3), 4.44 (t, 2H, C_5H_4), 4.33 (t, 2H, C_5H_4), 4.14 (s, 5H, C_5H_5), 2.03, 2.00 (each s, each 3H, CH_3). ^{13}C-NMR (67.8 MHz, $CDCl_3$) δ302.89 (W=C), 204.30 (CO_{eq}), 198.16 (t, ^1J = 63.6, CO_{ax}), 145.45, 142.03, 141.36, 140.59, 136.12, 134.19, 130.85, 130.15, 129.46, 128.68 ($C_{olef.}$), 83.23,69.53, 67.05 (C_5H_4), 69.39 (C_5H_5), 68.40 (OCH_3), 12.89, 12.30 (CH_3).-$C_{29}H_{26}FeO_6W$ (710.3): Calc. C 49.20, H 3.41; Found C 50.05, H 4.13.

2.2.4. Reaction of 1,3,5-{tris-[(triphenylphosphonio)methyl]}-benzene tribromide [19] with FcCHO under Wittig conditions

7: 800 mg (0.69 mmol) of 1,3,5-tris-[(triphenylphosphonio)methyl]-benzene tribromide were suspended in 15 ml of diethylether at − 78°C. 1.38 ml (2.2 mmol) of *n*-BuLi (1.6 M in hexane) was added dropwise. Then the mixture was warmed up to room temperature. After 4 h the resulting deep red suspension was conveyed into a drop funnel and slowly added to a solution of 443 mg (2.1 mmol) of ferrocenemonoaldehyde. After stirring over night and evaporation of the solvent, the residue was chromatographed on a silica gel column with CH_2Cl_2 as eluent. The first fraction contained the product. The solvent was evaporated and the crude product was dried in vacuo.

Red powder; yield 200 mg (0.28 mmol) 41%. ^{1}H-NMR (400 MHz, $CDCl_3$): δ7.31 (s, 3H, C_6H_3), 6.81 (d, 2H, ^3J = 16.2, H_{trans}), 6.62 (d, 2H, ^3J = 16.2, H_{trans}), 6.39 (d, 1H, ^3J = 11.9, H_{cis}), 6.30 (d, 1H, ^3J = 11.9, H_{cis}), 4.46 (t, 4H, $C_5H_{4\text{-}trans}$), 4.28 (t, 4H, $C_5H_{4\text{-}trans}$), 4.26 (t, 2H, $C_5H_{4\text{-}cis}$), 4.20 (t, 2H, $C_5H_{4\text{-}cis}$), 4.17–4.13 (m, 15H, C_5H_5). MS (FAB, mNBA) m/z (%) = 708(6.9)[M]$^+$. –$C_{42}H_{36}Fe_3$ (708.22): Calc. C 71.22, H 5.12; Found C 71.22, H 5.31.

2.2.5. Reaction of 1,3,5-tris[(triphenylphosphonio)-methyl]-benzene tribromide with 9-ferrocenyl-2,7-dimethylnonatetraenal [18] under Wittig conditions

8: The same reaction procedure as described for **7** was carried out. Dark red powder; yield 80 mg (36%). ^{1}H-NMR (400 MHz, $CDCl_3$): δ7.38 (s, 3H, C_6H_3), 6.96 (d, 3H, ^3J = 16.2, H_{trans}), 6.72–6.28 (m, 21H, H_{olef}), 4.47–4.42 (m, 6H, C_5H_4), 4.32–4.28 (m, 6H, C_5H_4), 4.16–4.13 (m, 15H, C_5H_5), 2.07, 2.05 (s, jew. 9H, CH_3). MS (FAB, mNBA) m/z(%) = 1105(1.1)[M]$^+$. –$C_{72}H_{72}Fe_3$ (1104.7): Calc. C 78.27, H 6.56; Found C 77.60, H 6.93.

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