Notes

Group VI Allyl Complexes of Dibenzo[c,g]fluorenide

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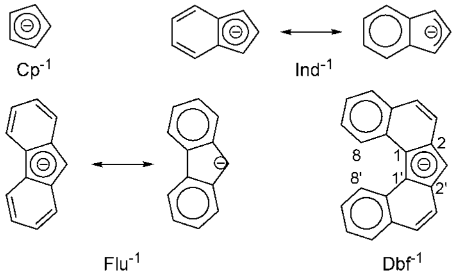
Summary: The synthesis of group VI complexes of the type (η^5-Dbf)M(CO)$_2$(η^3-allyl) (Dbf = dibenzo[c,g]fluorenide; M = Mo, W) is reported, which are the first examples of molybdenum and tungsten allyl complexes bearing a benzannealed fluorenide ligand. The molybdenum complex could be characterized by means of X-ray crystallography.

Introduction

Annulation of aromatic rings to the cyclopentadienide anion (Cp$^-$) enlarges the conjugated π system of the ligand. The most prominent examples of such ligands are indenide (Ind$^-$) and fluorenide (Flu$^-$). However, the coordination chemistry of these ligands differs from that of Cp$^-$. This is due to the fact that it is energetically favorable to avoid the perturbation of the six-membered aromatic ring by an annulated five-membered-ring system. Thus, for the indenide anion the negative charge is expected to be localized in an allylic fragment instead of being more or less symmetrically distributed on all carbon atoms of the five-membered ring. For fluorenide a high concentration of negative charge density is expected to be localized at the tertiary carbon atom of the five-membered ring (Scheme 1). Such an unbalanced charge distribution will facilitate a haptotropic $\eta^5 \rightarrow \eta^1$ shift of the metal center, thus leading to a decreased stability of the transition-metal complexes compared to the analogous systems with coordinating Cp ligands.[1] This becomes manifest in the fact that (Flu)$_2$Fe, an analogue to ferrocene, has not been described up to now. Further annulation of aromatic rings may thus result in an enhanced stability of the anionic ligand and its transition-metal complexes with respect to Flu$^-$, depending on the position at which the annulation takes place. One example for such a ligand is the dibenzo[*c,g*]fluorenide anion (Dbf$^-$, **1**$^-$). We are interested in this ligand due to its electronic structure (Scheme 1), its coordination chemistry, and especially its twisted chiral molecular geometry caused by the steric hindrance of the protons in the 8- and 8′-positions. The latter feature can simply be understood by considering Dbf-H a 2,2′-methylene-bridged 1,1′-binaphthyl.

Scheme 1



We recently were able to prove by means of DFT calculations that Dbf$^-$ indeed shows a more balanced charge distribution than Flu$^-$. This was corroborated by the synthesis of stable (Dbf)$_2$Fe.[2] Thus, Dbf$^-$ complexes can in terms of their electronic situation be considered as links between Flu$^-$ and Cp$^-$ complexes.

In the present publication we report the synthesis, spectroscopic data, and molecular structures of group VI metal complexes of the type (η^5-Dbf)M(CO)$_2$(η^3-allyl) (**3a**, M = Mo; **3b**, M = W). Dbf-H is accessible by following procedures published by Martin and Seki et al.,[3] which give access to the compound on a multigram scale. According to the synthesis of the fluorenide complex (η^5-Flu)Mo(CO)$_2$(η^3-pentadienyl) published by Peng and Liu et al.,[4] we reacted the precursor complexes (η^3-allyl)MBr(CO)$_2$(NCMe)$_2$ (**2a**, M = Mo;[5] **2b**, M = W[6]) with (Dbf)Li (Scheme 2).

The compounds **3a**,**b** are obtained as yellow microcrystalline solids in yields of about 40–50%. It turned out that it is favorable to prepare the lithium salt in toluene solution and add it to a solution of the η^3-allyl precursor **2a**,**b** in THF. To our knowledge a tungsten complex containing a fluorenide ligand is not known up to now. IR spectroscopy clearly proves the local C_{2v} symmetry of the M(CO)$_2$ unit (ν_{CO} in CH$_2$Cl$_2$ solution: **3a**, 1964, 1866 cm^{-1}; **3b**, 1939, 1853 cm^{-1}). These data correlate with the IR data of the corresponding CpM(CO)$_2$(η^3-allyl) and IndM(CO)$_2$(η^3-allyl) complexes.[7]

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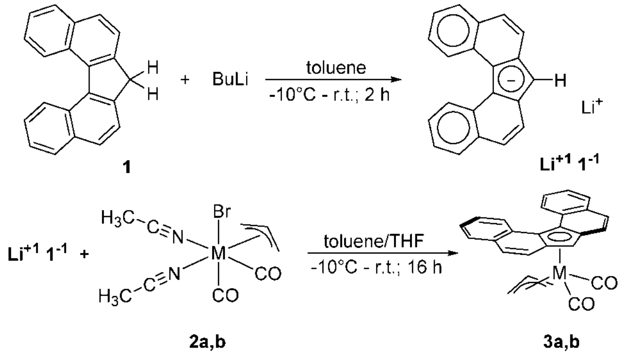
10.1021/om701022q CCC: $40.75

Scheme 2



While the ^{1}H and ^{13}C NMR resonances of the naphthyl systems of the Dbf fragment of **3a**,**b** could not be assigned individually, the resonances of the CH unit of the five-membered η^5-C_5H ring exhibits characteristic chemical shifts in the NMR spectra: it appears shifted to lower field ($CDCl_3$: **3a**, 6.52 ppm; **3b**, 6.57 ppm) compared to Dbf-H (**1**, 4.17 ppm). Additionally the position of the resonance of this proton is solvent dependent, as it is for $(Dbf)_2Fe$:[2] in C_6D_6 it is observed at 5.96 ppm (**3a**), indicating π-interaction of the Dbf site with the aromatic solvent. In contrast, the corresponding ^{13}C resonances are almost independent of the nature of the solvent (**3a**, 74.2 (C_6D_6), 74.0 ppm ($CDCl_3$); **3b**, 71.4 ppm ($CDCl_3$)). Dbf is a 1,1′-binaphthyl derivative, it thus possesses axial chirality due to steric repulsion of the protons in the 8- and 8′-position (for numbering see Scheme 1). Although the ^{1}H and ^{13}C NMR resonances of the binaphthyl system could not completely be assigned, it is obvious that only one set of signals is observed for the two formally different naphthyl sites of the Dbf ligand. We explain this by a rapid process of racemization with respect to the NMR time scale, a feature which was also observed for $(Dbf)_2Fe$ and $(Dbf)Mn(CO)_3$.[2] By means of quantum-chemical calculations we were able to show that the racemization barrier for Dbf^- is just about 19 kJ mol^{-1}, which makes it impossible to slow down the equilibration on the NMR time scale by cooling the sample. The only way to do this will be to introduce substituents at the 8- and 8′-positions of the Dbf backbone. We are currently working on such modified ligands.

Although two conformations of the allyl moiety are possible, only one set of signals was observed. The X-ray structure of **3a** (see below) confirms the exo structure proposed in Scheme 2. It seems that the allyl ligand prefers the central CH unit to be oriented between the two naphthyl units of Dbf due to steric reasons. Alternatively, in the endo structure, two of the terminal CH_2 protons would have to point directly toward the wings of Dbf. Three groups of resonances were observed in the ^{1}H NMR spectrum for the protons of the η^3-allyl ligand: the central CH groups provide symmetrical multiplets at 2.25 (**3a**) and 1.76 ppm (**3b**) in $CDCl_3$. The terminal CH_2 protons could be identified by their characteristic $^3J_{HH}$ coupling constants: 7.1 Hz for the coupling to the proton in the syn position and 10.3 Hz for the coupling to the proton in the anti position.[6a] The corresponding chemical shifts are 1.26 and 0.55 ppm for compound **3a** and 1.22 and 0.79 ppm for compound **3b**. The resonances of the syn-CH_2 protons as well as of the CH proton are shifted about 1.5 ppm toward high field compared to those in $CpM(CO)_2(\eta^3$-allyl),[7a] indicating a shielding by the ring current of the Dbf ligand. By means of CH-COSY NMR spectroscopy all carbon atoms of the η^5-C_5H unit and the η^3-allyl ligand could be assigned. The allyl ligands give one signal at 77.8 (**3a**) and 70.3 ppm (**3b**) for the central carbon atom and one signal at 50.0 (**3a**) and 42.0 ppm (**3b**) for the two terminal atoms. This again corroborates the rapid racemization of the binaphthyl unit of Dbf. The carbonyl ligands show a single ^{13}C NMR resonance at 236.9 (**3a**) and 224.1 ppm (**3b**).

Although solid-state structures of group VI metal complexes of the type $(\eta^5$-Cp$)M(CO)_2(\eta^3$-allyl) frequently have been reported in the literature,[8] the situation is quite different for the corresponding systems bearing η^5-Ind or η^5-Flu as ligands instead of η^5-Cp. To the best of our knowledge, no solid-state structure of the fluorenide complexes $(\eta^5$-Flu$)M(CO)_2(\eta^3$-allyl) (M = Cr, Mo, W) has been published up to now.[9] For the corresponding indenide systems $(\eta^5$-Ind$)M(CO)_2(\eta^3$-allyl) (M = Cr, Mo, W), only the solid-state structure of the molybdenum complex has been reported.[10] Here we report the first structure of a molybdenum complex bearing a η^5-coordinated benzannealed fluorenide ligand.

Yellow crystals of **3a** suitable for X-ray diffraction studies were obtained from a concentrated toluene solution by slow vapor diffusion of *n*-pentane. Figure 1 (left) shows the molecular structure of **3a** in the solid state. The complex crystallizes as a racemate in the triclinic centric space group $P\bar{1}$ with two heterochiral molecules in the unit cell. In the solid-state structure, the equilibration by torsion around the bond between C1 and C11 obviously is frozen. The corresponding torsion angle C10−C1−C11−-C20 is only 15.6°, which is much smaller than the corresponding angles in derivatives of the uncoordinated Dbf ligand. Here torsion angles of about 22–25° were found.[11] As already observed for $(\eta^5$-Dbf$)Mn(CO)_3$, where we found a torsion angle of 10.7°, the η^5 coordination of the Dbf ligand forces the five-membered ring to be as planar as possible, which makes the protons in the 8- and 8′-positions of the two naphthyl rings to approach each other (1.985 vs 2.110 Å in the uncoordinated Dbf-H). As predicted by means of theoretical calculations,[2] the bonds C3−C4 and C13−C14 (1.342 and 1.344 Å) are by far the shortest C−C bonds in the Dbf system, implying more or less localized C−C double bonds due to the dominance of the resonance structure shown in Scheme 1 (bottom, right side).

From Figure 1 (right), presenting a top view perpendicular to the plane of the five-membered ring, it becomes clear that the molybdenum atom undergoes a ring slippage from the center of the ring to the direction of C21. This leads to a significant variation of the bond distances between the molybdenum and the carbon atoms of the five-membered ring (maximum variation: 0.172 Å). This observation also has been made for (η^5-

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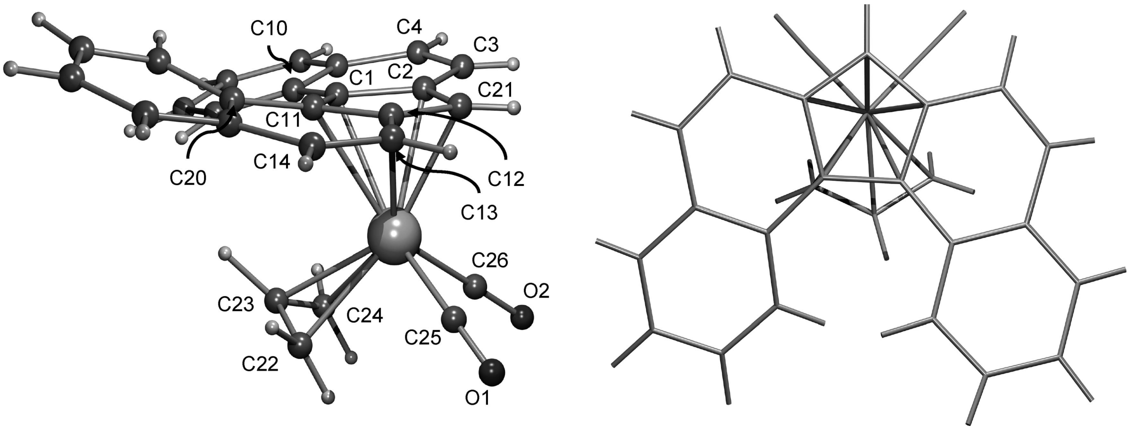

Figure 1. Molecular structure[12] of **3a** in the solid state (left) and top view of the five-membered ring (right). Selected distances (Å), angles (deg), and torsion angles (deg): Mo1−C1 = 2.459(7), Mo1−C2 = 2.345(7), Mo1−C11 = 2.428(7), Mo1−C12 = 2.394(7), Mo1−C21 = 2.287(7), Mo1−C22 = 2.320(9), Mo1−C23 = 2.225(8), Mo1−C24 = 2.322(8), Mo1−C25 = 1.941(7), Mo1−C26 = 1.946(7), O1−C25 = 1.164(9), O2−C26 = 1.167(9), C1−C2 = 1.439(10), C1−C11 = 1.472(10), C2−C21 = 1.433(10), C11−C12 = 1.441(10), C12−C21 = 1.419(10), C3−C4 = 1.339(11), C13−C14 = 1.343(10); C22−Mo1−C25 = 71.5(3), C24−Mo1−C26 = 74.3(3), C25−Mo1−C26 = 77.8(3); C10−C1−C11−C20 = –15.6(13).

Ind)Mo(CO)$_2$(η^3-allyl) type structures such as (η^5-Ind)Mo(CO)$_2$(η^3-C$_4$H$_7$), where η^3-C$_4$H$_7$ is η^3-crotyl.[10a,b] The allyl moiety in **3a** is slightly rotated; thus, H23 moves to a position where the steric hindrance with the Dbf fragment is reduced due to the internal twist of the Dbf.

Experimental Section

All synthetic procedures were carried out under an atmosphere of nitrogen. Solvents were purified and dried by standard methods.

Synthesis of the Dbf Complexes 3a,b. A 266 mg (1.00 mmol) portion of Dbf[3] was dissolved in 10 mL of dry toluene. The pale yellow solution was cooled to −10 °C, and 690 μL (1.1 mmol) of a 1.6 M solution of *n*-butyllithium in hexane was added dropwise. While it was stirred for at least another 2 h, the solution was slowly warmed to ambient temperature. The colorless precipitate of (Dbf)Li formed during this time was then dissolved by the addition of 3 mL of dry THF, resulting in a greenish black solution. In a second Schlenk tube 1.00 mmol of (η^3-allyl)M(CO)$_2$(NCMe)$_2$)Br was dissolved in 15 mL of dry THF. The resulting orange solution was cooled to 0 °C, and the solution of (Dbf)Li was added dropwise over 20 min. The solution was stirred at 0 °C for another 4 h, warmed to room temperature, and stirred for a further 16 h. After that time, the solvent was removed under vacuum and the yellowish brown residue was washed twice with 10 mL of dry Et$_2$O. For **3a** the residue was extracted three times with 10 mL of dry toluene. Removing the solvents from the unified extracts yielded the analytically pure product as a bright yellow microcrystalline solid (yield 224 mg, 49%). For **3b** the residue was extracted with dry toluene until the toluene extract became colorless. The combined solutions were evaporated to dryness, and the residue was extracted again three times with 10 mL of dry toluene. The unified extracts were concentrated and brought to crystallization at −40 °C to yield a first crop of **3b** as a bright orange-yellow microcrystalline solid. Further concentration and crystallization of the mother liquor yielded a second crop of the analytically pure product (yield 196 mg, 36%). Both compounds are sensitive to air. Analytical data for **3a** are as follows. ^{1}H NMR (C$_6$D$_6$): δ 8.73 (d, 2H, $^3J_{HH}$ = 8.1 Hz, Ar H), 7.49 (d, 2H, $^3J_{HH}$ = 7.3 Hz, Ar H), 7.25 (m, 4H, Ar H), 7.17 (d, 2H, $^3J_{HH}$ = 9.2 Hz, Ar H), 7.06 (d, 2H, $^3J_{HH}$ = 8.8 Hz, Ar H), 5.96 (s, 1H, 9-H), 2.23 (m, 1H, η^3-CH$_2$*C*HCH$_2$), 1.23 (d, 2H, $^3J_{HH}$ = 7.2 Hz, syn-η^3-C*H*$_2$CHC*H*$_2$), 0.49 (d, 2H, $^3J_{HH}$ = 10.6 Hz, anti-η^3-C*H*$_2$CHC*H*$_2$). ^{1}H NMR (CDCl$_3$): δ 8.81 (d, 2H, $^3J_{HH}$ = 8.2 Hz, Ar H), 7.85 (d, 2H, $^3J_{HH}$ = 7.8 Hz, Ar H), 7.66 (t, 2H, $^3J_{HH}$ = 7.2 Hz, Ar H), 7.57 (t, 2H, $^3J_{HH}$ = 7.3 Hz, Ar H), 7.54 (d, 2H, $^3J_{HH}$ = 8.8 Hz, Ar H), 7.43 (d, 2H, $^3J_{HH}$ = 8.8 Hz, Ar H), 6.52 (s, 1H, 9-H), 2.25 (m, 1H, η^3-CH$_2$C*H*CH$_2$), 1.26 (d, 2H, $^3J_{HH}$ = 7.1 Hz, syn-η^3-C*H*$_2$CHC*H*$_2$), 0.55 (d,2 H, $^3J_{HH}$ = 10.3 Hz, anti-η^3-C*H*$_2$CHC*H*$_2$). ^{13}C NMR (C$_6$D$_6$): δ 235.9 (CO), 132.3, 129.7, 129.3, 126.8, 126.3, 125.3, 123.2, 109.2 (η^5-C), 102.8 (η^5-C), 74.2 (9-C), 78.0 (η^3-CH$_2$*C*HCH$_2$), 49.8 (η^3-*C*H$_2$CH*C*H$_2$). ^{13}C NMR (CDCl$_3$): δ 236.9 (CO), 131.9, 129.6, 129.0, 126.9, 126.3, 126.3, 125.2, 123.6, 109.0 (η^5-C), 102.7 (η^5-C), 77.8 (η^3-CH$_2$*C*HCH$_2$), 73.95 (9-C), 50.03 (η^3-*C*H$_2$CH*C*H$_2$). IR (toluene, cm^{-1}): 1948, 1874 (2 × ν_{CO}). IR (CH$_2$Cl$_2$, cm^{-1}): 1964, 1866 (2 × ν_{CO}). Anal. Calcd for C$_{26}$H$_{18}$MoO$_2$ (458.36): C, 68.13; H, 3.96. Found: C, 68.10; H, 3.87. Analytical data for **3b** are as follows. ^{1}H NMR (CDCl$_3$): 8.79 (d, 2H, $^3J_{HH}$ = 8.2 Hz, Ar H), 7.85 (d, 2H, $^3J_{HH}$ = 7.7 Hz, Ar H), 7.66 (t, 2H, $^3J_{HH}$ = 7.4 Hz, Ar H), 7.60 (t, 2H, $^3J_{HH}$ = 7.4 Hz, Ar H), 7.54 (d, 2H, $^3J_{HH}$ = 9.0 Hz, Ar H), 7.43 (d, 2H, $^3J_{HH}$ = 8.9 Hz, Ar H), 6.57 (s, 1H, 9-H), 1.76 (m, 1H, η^3-CH$_2$C*H*CH$_2$), 1.22 (d, 2H, $^3J_{HH}$ = 7.1 Hz, syn-η^3-C*H*$_2$CHC*H*$_2$), 0.79 (d, 2H, $^3J_{HH}$ = 10.3 Hz, anti-η^3-C*H*$_2$CHC*H*$_2$). ^{13}C NMR (CDCl$_3$): δ 224.1 (CO), 131.9, 129.8, 128.8, 127.6, 126.7, 126.7, 125.1, 123.5, 107.4 (η^5-C), 101.1 (η^5-C), 71.4 (9-C), 70.3 (η^3-CH$_2$*C*HCH$_2$), 42.0 (η^3-

Table 1. Crystallographic Summary for 3a

formula	C$_{26}$H$_{18}$MoO$_2$
formula weight	458.34
crystal color, habit; size, mm	yellow block; 0.19 × 0.08 × 0.07
temp, K	150(2)
space group	$P\bar{1}$
a, Å	6.988(2)
b, Å	11.666(12)
c, Å	11.997(5)
α, deg	100.97(6)
β, deg	93.00(3)
γ, deg	90.64(4)
V, Å^3	958.6(11)
Z	2
D(calcd), g cm^{-3}	1.588
μ(Cu Kα(λ = 1.541 84 Å)), mm^{-1}	5.749
2θ range, deg	3.86−62.50
no. of reflns collected	7131
no. of indep rflns	2949
no. of params	334
R1, wR2 ($I > 2\sigma(I)$)[a]	0.0426, 0.1544
R1, wR2 (all data)[b]	0.0444, 0.1548
GOF[c]	1.195
$\Delta\rho_{max}/\Delta\rho_{min}$, e Å^{-3}	2.158/−0.863

[a] $R1 = \sum||F_o| - |F_c||/\sum|F_o|$. [b] $wR2 = (\sum[w(F_o^2 - F_c^2)^2]/\sum[w(F_o^2)^2])^{1/2}$. [c] $GOF = (\sum[w(F_o^2 - F_c^2)^2]/(n - p))^{1/2}$.

CH_2CH*C*H_2). IR (CH_2Cl_2, cm^{-1}): 1939, 1853 ($2 \times \nu_{CO}$). Anal. Calcd. for $C_{26}H_{18}WO_2$ (546.26): C, 57.17; H, 3.32. Found: C, 56.63; H, 3.16.

Structure Determination of Compound 3a. Crystal data and refinement parameters are collected in Table 1. The structure was solved using direct methods,[13] completed by subsequent difference Fourier syntheses, and refined by full-matrix least-squares procedures.[14] Semiempirical absorption correction from equivalents (Multiscan) was carried out. All hydrogen atom positions were found in the difference map calculated from the model containing all non-hydrogen atoms. The hydrogen positions were refined with individual isotropic displacement parameters.

Acknowledgment. We gratefully acknowledge the measurement of the NMR spectra by Christiane Müller.

Supporting Information Available: Tables and figures giving X-ray structure data and NMR spectra. This material is available free of charge via the Internet at http://pubs.acs.org. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as Supplementary Publication No. CCDC-676102 (**3a**). Copies of the data can be obtained free of charge on application to the CCDC, 12 Union Road, Cambridge CB2 1EZ, U.K. (fax, (+44)1223-336-033; e-mail, deposit@ccdc.cam.ac.uk).

OM701022Q

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