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Molybdenum complexes containing ferrocenyl-functionalised arylimido ligands; crystal structure of 2,6- Pr_2^i -4-Fc-C=C-C₆H₂N(SiMe₃)₂

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Abstract

The ferrocene-functionalised anilines $2,6-Pr_2^i-4-Fc-C_6H_2NH_2$ (**3a**), $2,6-Pr_2^i-4-Fc-C_6H_2N(SiMe_3)_2$ (**3b**), $2,6-Pr_2^i-4-Fc-C \equiv C-C_6H_2NH_2$ (**5a**) and $2,6-Pr_2^i-4-Fc-C \equiv C-C_6H_2N(SiMe_3)_2$ (**5b**) (Fc=ferrocenyl) have been prepared by Pd-catalysed cross-coupling reactions. The crystal structure of **5b** has been determined. The NSi₂ plane and the aryl ring plane form a dihedral angle of 89.8 °. The N atom is trigonal planar [C–N distance 1.459(4) Å]. The Cp ring and the aryl ring planes, which are connected by the C=C spacer, form a dihedral angle of 85.8 °. **3a** and **5a** have been utilised for the preparation of the novel redox-functionalised imido complexes [Mo(N-2,6-Pr_2^i-4-Fc-C_6H_2)_2Cl_2(DME)] (**6**) and [Mo(N-2,6-Pr_2^i-4-Fc-C=C-C_6H_2)_2Cl_2(DME)] (**7**) (DME=1,2-dimethoxyethane). Electrochemical investigations by cyclic voltammetry revealed a pronounced interaction between the respective *N* functional group and the ferrocenyl moiety for all six compounds, which is largest for **6**. © 1999 Elsevier Science Ltd. All rights reserved.

Keywords: Crystal structure; Cyclic voltammetry; Ferrocene, functionalised; Imido ligand, redox-functionalised; Iron; Molybdenum

1. Introduction

The great current interest in ferrocene-functionalised ligands is mainly due to the special stereo- and redoxchemical properties of the ferrocene moiety. Such ligands have found widespread application for asymmetric catalysis [1–3] and redox-chemical sensors [4–7], and their potential for a variety of areas such as, for example, thermotropic liquid crystals [8] and redox-tunable catalysts [9] is starting to emerge. The vast majority of ferrocene-functionalised ligands known to date are phosphanes and oxygen-, nitrogen- or sulfur-containing macrocycles. Investigations concerning further important ligand platforms are still rare. We are therefore currently investigating the use of ferrocene-functionalised (oligo-)pyridine and organoimido ligands in transition metal chemistry, and here report on results from our imido chemistry program.

2. Experimental

All manipulations were performed in an inert atmosphere (purified argon or dinitrogen) by using standard Schlenk and cannula techniques or a conventional glovebox. Solvents and reagents were appropriately dried and purified by using standard procedures. NMR spectra were recorded at 300 K with a Bruker DRX 500 spectrometer operating at 500.13 MHz for ¹H; TMS was used as external reference. Cyclic voltammograms were recorded with an EG&G Princeton Applied Research Potentiostat/Galvanostat Model 273A; ferrocene or decamethylferrocene was used as internal reference. Elemental analyses were performed by the Microanalytical Laboratory of the Universität Bielefeld. 2,6-Pr₂ⁱ-4-I-C₆H₂NH₂ (2a) was prepared from commercially available $2,6-Pr_2^i$ -C₆H₃NH₂ and iodine in a degassed aqueous solution of NaHCO₃ [19]. 2,6- Pr_2^i -4-I-C₆H₂N(SiMe₃)₂ (**2b**) was obtained from 2a by a standard lithiation/silvlation sequence using lithium diisopropylamide and chlorotrimethylsilane. Ethinylferrocene (4) [20], $Pd(PPh_3)_4$ [21] and $Pd(dba)_2$ [22] were prepared according to published procedures.

2.1. Synthesis of 2,6- Pr_2^i -4-Fc- $C_6H_2NH_2$ (**3***a*)

Bu^tLi (10.0 cm³ of a 1.70 M solution in pentane, 17.0 mmol) was added slowly to a stirred solution of ferrocene (1.30 g, 7.00 mmol) in THF (50 cm³) at -20° C. The mixture was allowed to warm to room temperature and was stirred for 30 min. Volatile components were removed in vacuo. The residue was dissolved in THF (30 cm³).

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Subsequently, a solution of ZnCl₂ (0.95 g, 7.00 mmol) in THF (10 cm³) was added. After stirring of the mixture for 30 min it was added to a solution containing 2a (1.73 g, 5.70 mmol) and a few mg of $Pd(PPh_3)_4$ in THF (35 cm³). The resulting solution was stirred for 15 h at room temperature and was refluxed for 1 h. It was allowed to cool to room temperature and was then quenched with 0.10 M HCl (50 cm³) and extracted with CHCl₃ (3×30 cm³). The combined organic layers were dried with Na₂SO₄. Volatile components (solvents and ferrocene) were removed in vacuo. The crude product was purified by column chromatography (silica gel, eluent CHCl₃) to yield **3a** (2.60 g, 74%) as an orange viscous oil. ¹H-NMR (CDCl₃): δ 1.30 (d, ³J_{HH}=6.8 Hz,12 H, CHMe₂), 2.93 (sept, ³J_{HH}=6.8 Hz, 2 H, CHMe₂), 3.67 (br s, 2 H, NH₂), 4.03 (s, 5 H, C₅H₅), 4.23 (s, 2 H, C₅H₄), 4.53 (s, 2 H, C_5H_4), 7.12 (s, 2 H, ArH). ¹³C{¹H} NMR (CDCl₃): δ 22.5 (CHMe₂), 27.9 (CHMe₂), 66.4 (C₅H₄), 68.0 (C₅H₄), 69.4 (C₅H₅), 88.8 (C₅H₄), 121.5 (Ar CH), 128.5 (quart Ar C), 132.2 (quart Ar C), 138.7 (Cipso). Anal. Calcd for C₂₂H₂₇NFe (361.3): C,73.14; H, 7.53; N, 3.88. Found: C, 72.82; H, 7.44; N, 3.68.

2.2. Synthesis of 2,6- Pr_2^i -4-Fc- $C_6H_2N(SiMe_3)_2$ (**3b**)

In analogy to the preparation of **3a**, compound **3b** (2.60 g, 46%) was obtained from **2b** (5.00 g, 11.2 mmol) after one crystallisation of the crude product from hot methanol. ¹H NMR (CDCl₃): δ 0.10 (s, 18 H, SiMe₃), 1.23 (d, ³J_{HH}=6.9 Hz, 12 H, CHMe₂), 3.45 (sept, ³J_{HH}=6.9 Hz, 2 H, CHMe₂), 3.98 (s, 5 H, C₅H₅), 4.26 ('t', apparent J_{HH}=1.7 Hz, 2 H, C₅H₄), 4.62 ('t', apparent J_{HH}=1.7 Hz, 2 H, C₅H₄), 7.12 (s, 2 H, ArH). ¹³C{¹H} NMR (CDCl₃): δ 2.5 (SiMe₃), 25.1 (CHMe₂), 27.4 (CHMe₂), 66.3 (C₅H₄), 68.5 (C₅H₄), 69.6 (C₅H₅), 86.5 (C₅H₄), 121.4 (Ar CH), 133.9 (quart Ar C), 141.5 (quart Ar C), 146.5 (C_{ipso}). Anal. Calcd for C₂₈H₄₃NSi₂Fe (505.3): C, 66.56; H, 8.58; N, 2.77. Found: C, 66.61; H, 8.74; N, 2.72.

2.3. Synthesis of 2,6- Pr_2^i -4-Fc- $C \equiv C$ - $C_6H_2NH_2$ (5a)

Pd(dba)₂ (0.17 g, 0.3 mmol) was added to a degassed solution of ethinylferrocene (**4**) (3.17 g, 15.1 mmol), **2a** (4.57 g, 15.1 mmol), PPh₃ (0.24 g, 0.9 mmol) and CuI (30 mg, 0.2 mmol) in triethylamine (150 cm³) placed in a thick-walled Rotaflo ampoule. The solution was stirred at 65°C for 6 h. The precipitate was filtered off. Volatile components were removed in vacuo from the filtrate. The residue was recrystallised from *n*-hexane to give **5a** (2.97 g, 51%) as an orange solid. ¹H NMR (CDCl₃): δ 1.27 (d, ³*J*_{HH}=6.9 Hz,12 H, CH*Me*₂), 2.86 (sept, ³*J*_{HH}=6.9 Hz, 2 H, C*HMe*₂), 3.85 (br s, 2 H, N*H*₂), 4.19 (s, 2 H, C₅H₄), 4.23 (s, 5 H, C₅H₅), 4.46 (s, 2 H, C₅H₄), 7.16 (s, 2 H, ArH). ¹³C{¹H} NMR (CDCl₃): δ 22.3 (CH*Me*₂), 27.9 (CHMe₂), 66.5 (C₅H₄), 68.4 (C₅H₄), 69.9 (C₅H₅), 72.2

(C₅H₄), 84.9 (C≡C), 87.2 (C≡C), 113.0 (C), 126.4 (Ar CH), 132.2 (quart Ar C), 140.4 (C_{ipso}). Anal. Calcd for C₂₄H₂₇NFe (385.3): C, 74.82; H, 7.06; N, 3.64. Found: C, 74.78; H, 7.14; N, 3.50.

2.4. Synthesis of 2,6- Pr_2^i -4-Fc- $C \equiv C$ - $C_6H_2N(SiMe_3)_2$ (5b)

In analogy to the preparation of **5a**, compound **5b** (4.15 g, 74%) was obtained as a yellow solid from **2b** (3.86 g, 10.6 mmol). ¹H NMR (CDCl₃): δ 0.07 (s, 18 H, SiMe₃), 1.18 (d, ³J_{HH}=6.9 Hz,12 H, CHMe₂), 3.42 (sept, ³J_{HH}=6.9 Hz, 2 H, CHMe₂), 4.21 ('t', apparent J_{HH}=1.8 Hz, 2 H, C₅H₄), 4.23 (s, 5 H, C₅H₅), 4.48 ('t', apparent J_{HH}=1.5 Hz, 2 H, C₅H₄), 7.17 (s, 2 H, ArH). ¹³C{¹H} NMR (CDCl₃): δ 2.5 (SiMe₃), 25.0 (CHMe₂), 27.4 (CHMe₂), 65.9 (C₅H₄), 68.6 (C₅H₄), 69.9 (C₅H₅), 71.3 (C₅H₄), 86.5 (C=C), 119.1 (quart Ar C), 127.2 (Ar CH), 143.8 (quart Ar C), 147.2 (C_{ipso}). Anal. Calcd for C₃₀H₄₃NSi₂Fe (529.7): C, 68.03; H, 8.18; N, 2.64. Found: C, 67.75; H, 8.32; N, 2.56.

2.5. Synthesis of $Mo(N-2,6-Pr_2^i-4-Fc-C_6H_2)_2Cl_2(DME)$ (6)

Triethylamine (0.58 g, 5.76 mmol), chlorotrimethylsilane (1.04 g, 9.60 mmol) and a solution of 3a (0.60 g, 1.92 mmol) in DME (10 cm^3) were added sequentially to a suspension of sodium molybdate (0.20 g, 0.96 mmol) in DME (20 cm³) placed in a thick-walled Rotaflo ampoule. The mixture was stirred at 60°C for 15 h. Insoluble material was filtered off and washed with DME (2×5 cm³). The red solution was reduced to dryness in vacuo. The remaining solid was washed with *n*-hexane (3×5) cm³) affording 6 (0.71 g, 76%) as a deep red, microcrystalline solid. ¹H NMR (CDCl₃): δ 1.11 (d, ³J_{HH}= 6.3 Hz, 24 H, CHMe₂), 3.80 (s, 6 H, OMe), 3.94 (m, 8 H, CHMe₂ and OCH₂), 4.01 (s, 10 H, C₅H₅), 4.30 (s, 4 H, C_5H_4 , 4.61 (s, 4 H, C_5H_4), 7.13 (s, 4 H, ArH). ¹³C{¹H} NMR (CDCl₃): δ 24.9 (CHMe₂), 27.2 (CHMe₂), 62.1 (OMe), 66.3 (C₅H₄), 68.8 (C₅H₄), 69.3 (C₅H₅), 71.2 (OCH₂), 85.2 (C₅H₄), 120.2 (Ar CH), 137.8 (quart Ar C), 144.1 (quart Ar C), 151.6 (Cipso). Anal. Calcd for C₄₈H₆₀N₂Cl₂Fe₂MoO₂ (975.6): C, 59.09; H, 6.20; N, 2.87. Found: C, 59.82; H, 6.44; N, 2.90.

2.6. Synthesis of $Mo(N-2,6-Pr_2^i-4-Fc-C \equiv C-C_6H_2)_2Cl_2(DME)$ (7)

In analogy to the preparation of **6**, complex **7** (1.67 g, 79%) was obtained as a deep red, microcrystalline solid from triethylamine (1.26 g, 11.4 mmol), chlorotrimethyl-silane (2.25 g, 20.7 mmol), **3b** (1.60 g, 4.14 mmol), and sodium molybdate (0.42 g, 2.07 mmol) in DME (55 cm³). ¹H NMR (CDCl₃): δ 1.06 (d, ³*J*_{HH}=6.6 Hz, 24 H, CH*Me*₂), 3.51 (s, 6 H, OMe), 3.66 (s, 4 H, OCH₂), 3.84

Table 1 Crystallographic data for compound **5b**

Empirical formula C ₃₀ H ₄₃ FeNSi ₂		Index ranges	$0 \le h \le 10, \ 0 \le k \le 19, \ -22 \le l \le 22$	
Formula weight	529.68	μ , mm ⁻¹	0.610	
Space group	Monoclinic, $P2_1/c$	F(000)	1136	
<i>a</i> , Å	9.185(7)	θ range, deg	2.12-24.99	
<i>b</i> , Å	16.734(11)	Reflections collected	5543	
<i>c</i> , Å	19.273(12)	Unique reflections	5197 ($R_{int} = 0.0375$)	
β , deg	94.25(6)	Data/restraints/parameters	5197/0/317	
$V, \text{ Å}^3$	2954(3)	R_F/R_F^2 [I>2 $\sigma(I)$]	0.0481/0.1148 (3934 reflections)	
Ζ	4	Goodness-of-fit on F^2	1.014	
$\rho_{\rm calcd}, {\rm g/cm}^3$	1.191	Largest diff. peak and hole, $e/Å^3$	0.293 and -0.323	

(sept, ${}^{3}J_{\rm HH}$ =6.6 Hz, 4 H, CHMe₂),4.01 (s, 10 H, C₅H₅), 4.23 (s, 14 H, C₅H₅ and C₅H₄), 4.47 (s, 4 H, C₅H₄), 7.13 (s, 4 H, ArH). ${}^{13}C{}^{1}H{}$ NMR (CDCl₃): δ 24.1 (CHMe₂), 27.3 (CHMe₂), 62.6 (br, OMe), 64.8 (C₅H₄), 68.5 (C₅H₄), 69.5 (C₅H₅), 71.0 (C₅H₄), 86.1 (C=C), 89.2 (C=C), 121.9 (quart Ar C), 125.6 (Ar CH), 144.4 (quart Ar C), 152.2 (C_{ipso}). Anal. Calcd for C₅₂H₆₀N₂Cl₂ Fe₂MoO₂ (1023.6): C, 61.02; H, 5.91; N, 2.74. Found: C, 61.63; H, 6.05; N, 2.99.

2.7. X-ray crystal structure analysis of 2,6- Pr_2^i -4-Fc- $C \equiv C-C_6H_2N(SiMe_3)_2$ (5b)

A yellow crystal with dimensions $0.40 \times 0.20 \times 0.20$ mm was used for data collection at 173(2) K on a Siemens P3 four-circle diffractometer with graphite-monochromated Mo K α radiation (λ =0.71073 Å). The structure was solved by direct methods. Programs used were Siemens SHELXTL PLUS [23] and SHELXL 97 [24]. Full-matrix least-squares refinement on F^2 was carried out anisotropically for the non-hydrogen atoms. Hydrogen atoms were included at calculated positions using a riding model. Further details are given in Table 1.

3. Results and discussion

We have investigated a number of ferrocene-bearing 2,6-diisopropylanilines as arylimido ligand precursors for the synthesis of redox-functionalised organoimido complexes. The synthesis of the anilines is outlined in Scheme 1.

Negishi coupling [10] of FcZnCl (1) with 2,6-Pr₂ⁱ-4-I-C₆H₂NH₂ (2a) and 2,6-Pr₂ⁱ-4-I-C₆H₂N(SiMe₃)₂ (2b) yielded 2,6-Pr₂ⁱ-4-Fc-C₆H₂NH₂ (3a) and 2,6-Pr₂ⁱ-4-Fc-C₆H₂N(SiMe₃)₂ (3b), respectively. Similarly, Sonogashira coupling [11] of FcC=CH (4) with 2a and 2b afforded 2,6-Pr₂ⁱ-4-Fc-C=C-C₆H₂NH₂ (5a) and 2,6-Pr₂ⁱ-4-Fc-C=C-C₆H₂NH₂ (5b), respectively. It is noteworthy that the presence of an unprotected primary amino group in 2a and 3a does not affect the product yields.

5b has been investigated by a single-crystal X-ray



Scheme 1. Synthesis of the ferrocenyl-functionalised anilines **3** and **5** by Pd-catalysed cross-coupling reactions.

structure analysis. A view of the molecule is shown in Fig. 1.

The aryl unit and the ferrocenyl unit are undistorted. Their respective C–C bond lengths are identical within experimental error, their average value being 1.40 Å for the C₆ ring and 1.42 Å for the cyclopentadienyl rings. The Cp rings are arranged in an eclipsed orientation and exhibit an average Fe–C bond length of 2.04 Å. The acetylene group is almost linear $[C(1)-C(11)-C(12), 176.8(3); C(11)-C(12)-C(13), 176.5(3)^{\circ}]$ and has a C–C distance of 1.207(4) Å; the distance between each acetylene carbon



Fig. 1. ORTEP drawing of **5b**. Selected bond lengths (Å) and angles (deg): Si(1)-N(1) 1.749(3), Si(2)-N(1) 1.747(3), C(15)-C(19) 1.522(4), C(17)-C(28) 1.522(4); Si(1)-N(1)-Si(2) 125.02(16), C(14)-C(15)-C(19) 118.8(3), C(18)-C(17)-C(28) 118.6(3).

atom and the sp² carbon atom attached to it is 1.431(4)[C(1)-C(11)] and 1.439(4) Å [C(12)-C(13)], respectively. These values are unexceptional and compare well with those of other structurally characterised (phenylethynyl)ferrocenes [12-14]. A striking feature of the structure of **5b** is the fact that the Cp ring plane and the aryl ring plane, which are connected by the acetylene spacer, are almost perpendicular to each other, their dihedral angle being 85.8°. This may be the result of crystal packing forces, but it may also reflect an interaction of each aromatic ring with a different one of the two orthogonal π bonds of the acetylene unit. Although such a perpendicular orientation is not unprecedented [12], we note that in the majority of related compounds a more or less coplanar arrangement of the rings has been observed [12–14]. Owing to the severe steric congestion around the amino group, the NSi₂ plane is orthogonal to the aryl ring plane (dihedral angle 89.8 °). The N atom is trigonal planar (sum of angles 360°), and the C-N distance has a value of 1.459(4) Å. In comparison, the sterically less crowded N,N,N',N'-tetrakis(trimethylsilyl)-para-phenylenediamine exhibits slightly smaller values for the C-N bond length (1.44 Å) and the NSi_2-C_6 dihedral angle (83°) [15].

Reaction of **3a** and **5a** under standard 'one-pot' conditions [16] with sodium molybdate in dimethoxyethane (DME) in the presence of triethylamine and chlorotrimethylsilane gave the bis(imido)molybdenum complexes [Mo(N-2,6-Pr₂ⁱ-4-Fc-C₆H₂)₂Cl₂(DME)] (**6**) and [Mo(N-2,6-Pr₂ⁱ-4-Fc-C=C-C₆H₂)₂Cl₂(DME)] (**7**), respectively, in high yields (Scheme 2). Their spectroscopic data are fully



Scheme 2. Synthesis of complexes 6 and 7.

Table 2

Formal electrode potentials (in V, vs ferrocene/ferrocenium) for the oxidation process of the present ferrocenyl-functionalised compounds in dichloromethane solution^a

Compound	3a	5a	3b	5b	6	7
$\frac{E_{0/+}^{0}}{\Delta E^{0,b}}$	-0.10	+0.05	-0.03 + 0.07	+0.10 +0.05	+0.03 +0.13	+0.12 +0.07

^a Conditions: 298 K; supporting electrolyte, NBu₄PF₆ (0.1 M); Pt disk working electrode, Ag wire pseudo-reference electrode, Pt wire counter electrode; scan rate, 0.1 V/s; solute concentration, 1 mM; confidence limit 0.01 V.

^b Referenced to the respective parent aniline (3a or 5a).

in accord with a distorted octahedral core geometry with *cis* coordinating DME and imido ligands and a *trans* arrangement of the chloro ligands, as is commonly observed for bis(imido) complexes of the type [Mo(NR)₂Cl₂(DME)].

6 and **7** are rare examples of ferrocenyl-functionalised transition-metal imido complexes, the only previous example being, to the best of our knowledge, the polyoxo-ymetallate $[(FcN)Mo_6O_{18}]^{2^-}$ published recently by Maatta and coworkers [17,18].

The electrochemical behavior of the new ferrocenefunctionalised compounds has been investigated by cyclic voltammetry (Table 2).

The $\Delta E^{0'}$ values, referenced to the respective parent aniline, range from 0.05 (5b) to 0.13 V (6) and prove an interaction between the ferrocenyl unit and the N functional group. Bearing in mind that the electronic influence of the MoCl₂(DME) fragment is shared between two ferrocenyl groups in the case of the bis(imido) complexes, one can conclude that the interaction is quite pronounced for 7 and especially for 6, which shows the largest ΔE^{0} value altogether. The fact that in comparison to the parent anilines 3a and 5a the potentials of the silvlated derivatives 3b and 5b are shifted anodically is most likely due to the orthogonal arrangement of the NSi₂ plane and the aromatic C_6 ring plane, which effectively prevents electron-donating n_N/π -delocalisation, as is the case in the closely related N,N,N',N'-tetrakis(trimethylsilyl)-paraphenylenediamine [15].

We are currently exploring the use of **6** and **7** as precursors for redox-tunable olefin metathesis catalysts of the type $[Mo(NR^1)(CHR^2)(OR^3)_2]$ [16] and will report on our findings in due course.

Supplementary data

Supplementary data concerning the crystal structure determination of **5b** are available from the CCDC, 12 Union Road, Cambridge CB2 1 EZ, UK on request, quoting the deposition number 111895.

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