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Synthesis of new thiophenolato hydrido iron(II) complexes and their substitution reactions with alkynes

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1. Introduction

Hydrido compounds of transition metals are not only important materials in many stoichiometric reactions, but also decisive intermediates in catalytic processes. Therefore, they play an important role in synthetic and industrial chemistry. Literature reports on transition metal hydrides are mainly concentrated on precious metals such as Ru, Rh, Ir, Pt, Pd [1–13]. For their lighter congeners Fe, Co and Ni, there are rather few reports with regard to catalytic properties of their metal hydrides, which would be of great significance from the perspective of economic practicality.

A [M–H] moiety in hydrido compounds of transition metals can react with alkenes or alkynes giving rise to σ -alkyl or σ -alkenyl metal compounds. It has been demonstrated that hydrides of cobalt and nickel afforded the expected products, while the family iron hydrides has received less attention [14,15].

More recently, vinyl sulfides have shown great potential in organic synthesis with the useful feature of a unique selectivity control. They are easily obtained by reaction of thiols with alkynes [16–18], and it was recognized that the addition of thiol to alkyne in the presence of a metal catalyst occurs with high regioselectivity and good yields [19,20].

 $\begin{array}{ccc} \text{Co}(\text{PMe}_3)_4 & & \text{SPh} \\ \text{Co}(\text{PMe}_3)_3\text{Cl} & + & \text{PhSH} & & & \text{Me}_3\text{P}_{\text{Me}_3} \\ \text{Co}(\text{PMe}_3)_4\text{Me} & & & \text{H} & \text{Co}_{\text{PMe}_3} \\ \end{array}$ (1)

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ABSTRACT

The novel thiophenolato hydrido iron(II) complexes [*cis*-Fe(H)(SAr)(PMe₃)₄] (**4**–**6**) (Ar = *p*-BrC₆H₄ (**4**), *p*-MeOC₆H₄ (**5**) and *o*-MeC₆H₄ (**6**)) were prepared through the reaction of Fe(PMe₃)₄ with thiophenols ArSH (**1**–**3**). Reaction of **6** with trimethylsilylacetylene and phenylacetylene afforded bisalkynyl iron(II) complexes [Fe(PMe₃)₄(C \equiv CSiMe₃)₂] (**7**) and [Fe(PMe₃)₄(C \equiv CPh)₂] (**9**) through elimination of dihydrogen and the formation of thiophenol. The reaction of **5** with 2-methyl-3-butyn-2-ol gave [Fe(PMe₃)₄(C \equiv CC-Me₂OH)₂] (**10**). The crystal structures of complexes **4**, **7** and **10** were determined by X-ray diffraction. A mechanism for the formation of **7** is proposed.

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We have reported on the S–H bond activation of thiophenols at cobalt(0) and cobalt(I) centres [21]. A novel thiophenolato hydrido cobalt(III) complex was obtained (Eq. (1)). As continuation of this work we here report on progress in the S–H bond activation by iron(0). The novel hydrido iron complexes (**4–6**) formed by oxidative addition of S–H moieties were isolated and characterized. Reaction of the hydrido iron complexes with alkynes invariably resulted in bisalkynyl iron(II) complexes. No insertion products have been observed that dominated reactions of hydrido cobalt [22] and hydrido nickel [15] complexes.

2. Experimental

2.1. General procedures and materials

Standard vacuum techniques were used in manipulations of volatile and air-sensitive materials. The literature method was used in the preparation of tetrakis(trimethylphosphane)iron(0) [23]. Other chemicals were used as purchased. Infrared spectra (4000–400 cm⁻¹), as obtained from Nujol mulls between KBr disks, were recorded on a Nicolet 5700. ¹H, ¹³C, and ³¹P NMR (300, 75, and 121 MHz, respectively) spectra were recorded on a Bruker Avance 300 spectrometer. ¹³C and ³¹P NMR resonances were obtained with broadband proton decoupling. X-ray crystallography was performed with a Bruker Smart 1000 diffractometer.

2.2. Synthesis of the complexes

2.2.1. Synthesis of 4

To a solution of 1.10 g (3.05 mmol) of Fe(PMe₃)₄ in 50 mL of pentane was slowly injected 0.58 g (3.07 mmol) of *p*-bromothiophenol





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1 at -80 °C. The reaction started immediately with formation of a yellow solid. The mixture was allowed to warm to ambient temperature. During this period the color turned yellow-brown. After stirring at room temperature for 16 h a dark brown solution was obtained. After a slight turbidity had been filtered off, the red filtrate at 4 °C gave red crystals of 4 suitable for X-ray diffraction. Yield: 0.28 g (17%). *Anal.* Calc. for C₁₈H₄₁BrFeP₄S (549.21 g/mol): C, 39.36; H, 7.52. Found: C, 39.02; H, 8.01%. IR (Nujol, cm⁻¹): 1866 v(Fe–H), 1580 v(C=C), 939 v(PMe₃). ¹H NMR (300 MHz, C₆D₆, 296 K, ppm): δ 0.99 (m, 36H, PCH₃), 7.16–8.09 (m, 4H, Ar–H), -10.31 (m, 1H, Fe–H); ³¹P NMR (121 MHz, C₆D₆, 300 K, ppm): δ 10.1 (m, 1P), 16.6 (m, 2P), 27.6 (m, 1P).

2.2.2. Synthesis of 5

To a solution of 1.24 g (3.44 mmol) of Fe(PMe₃)₄ in 50 mL of pentane was slowly injected 0.48 g (3.44 mmol) of *p*-methoxylthiophenol **2** at -80 °C. The reaction started immediately with formation of a yellow solid. The reaction mixture was allowed to warm to ambient temperature. During this period the color turned yellow-brown. After stirring at room temperature for 16 h a dark brown solution was obtained. A slight turbidity was filtered off, and from the red filtrate at 4 °C red crystals of **5** were obtained. Yield: 0.43 g (25%). *Anal.* Calc. for C₁₉H₄₄FeOP₄S (500.36 g/mol): C, 45.61; H, 8.86. Found: C, 45.46; H, 9.00%. IR (Nujol, cm⁻¹): 1818 ν (Fe-H), 1591 ν (C=C), 942 ν (PMe₃). ¹H NMR (300 MHz, C₆D₆, 296 K, ppm): δ 1.12 (m, 36H, PCH₃), 3.40 (s, 3H, OCH₃), 7.15–8.22 (m, 4H, Ar-H), -14.20 (m, 1H, Fe-H); ³¹P NMR (121 MHz, C₆D₆, 300 K, ppm): δ 10.8 (m, 1P), 16.5 (m, 2P), 24.2 (m, 1P).

2.2.3. Synthesis of 6

To a solution of 1.37 g (3.80 mmol) of Fe(PMe₃)₄ in 50 mL of pentane were slowly injected 0.47 g (3.79 mmol) of o-methylthiophenol 3 at -80 °C. The reaction started immediately with formation of a yellow solid. The reaction mixture was allowed to warm to ambient temperature. During this period the color turned yellow-brown. After stirring at room temperature for 16 h a dark brown solution was obtained. A slight turbidity was filtered off. and from the red filtrate at 4 °C red crystals of 6 were obtained. Yield: 0.51 g (28%). Anal. Calc. for C₁₉H₄₄FeP₄S (484.36 g/mol): C, 47.12; H, 9.16. Found: C, 46.80; H, 8.88%. IR (Nujol, cm⁻¹): 1834 v(Fe-H), 1580 v(C=C), 937 v(PMe₃). ¹H NMR (300 MHz, C₆D₆, 296 K, ppm): δ 1.13 (m, 36H, PCH₃), 2.87 (s, 3H, CH₃), 7.16-8.66 (m, 4H, Ar-H), -9.99 (m, 1H, Fe-H); ³¹P NMR (121 MHz, C₆D₆, 300 K, ppm): δ 10.4 (m, 1P), 16.4 (m, 2P), 27.5 (m, 1P); ¹³C NMR (75 MHz, C_6H_6 , 297 K, ppm): δ 24.5 (m, PCH₃), 30.5 (s, Ar–C), 119.3 (s, Ar-C), 125.0 (s, Ar-C), 131.3 (s, Ar-C).

2.2.4. Synthesis of 7

To a solution of 0.50 g (1.03 mmol) of complex **6** in 50 mL of pentane were slowly injected 0.21 g (2.14 mmol) of trimethylsilylacetylene at -80 °C. After stirring at room temperature for 18 h a dark brown solution was obtained. A slight turbidity was filtered off, and from the brown filtrate at 4 °C yellow cubic crystals of **7** were obtained. Yield: 0.17 g (30%). *Anal.* Calc. for C₂₂H₅₄FeP₄Si₂ (554.55 g/mol): C, 47.65; H, 9.81. Found: C, 47.86; H, 10.01%. IR (Nujol, cm⁻¹): 1968 v(C=C), 939 v(PMe₃). ¹H NMR (300 MHz, C₆D₆, 296 K, ppm): δ 1.42 (m, 36H, PCH₃), 0.11 (d, 6H, CH₃); ³¹P NMR (121 MHz, C₆D₆, 300 K, ppm): δ 20.0 (s, *P*CH₃).

2.2.5. Synthesis of 9

To a solution of 0.80 g (1.65 mmol) of complex **6** in 50 mL of pentane were slowly injected 0.34 g (3.33 mmol) of phenylethyne at -80 °C. After stirring at room temperature for 18 h a dark brown solution was obtained. A slight turbidity was fil-

tered off, and from the brown filtrate at 4 °C yellow cubic crystals of **9** were obtained. Yield: 0.23 g (25%). *Anal.* Calc. for C₂₈H₄₆FeP₄ (562.41 g/mol): C, 59.80; H, 8.24. Found: C, 59.90; H, 8.01%. IR (Nujol, cm⁻¹): 2037 v(C=C), 1589 v(C=C), 936 $v(PMe_3)$. ¹H NMR (300 MHz, C₆D₆, 296 K, ppm): δ 1.41 (m, 36H, PCH₃), 6.96–7.41 (m, 10H, Ar–H); ³¹P NMR (121 MHz, C₆D₆, 300 K, ppm): δ 20.9 (s, PCH₃).

2.2.6. Synthesis of 10

To a solution of 0.82 g (1.64 mmol) of complex **5** in 50 mL of pentane at -80 °C were slowly injected 0.28 g (3.33 mmol) of 2-methyl-3-butyn-2-ol. The reaction mixture was allowed to warm to ambient temperature. After stirring at room temperature for 16 h the color of the solution changed to yellow-brown. A slight turbidity was filtered off, and from the yellow filtrate at 4 °C yellow cubic crystals of **10** were obtained. Yield: 0.43 g (25%). *Anal.* Calc. for C₂₂H₅₀FeO₂P₄ (526.38 g/mol): C, 50.20; H, 9.57. Found: C, 50.50; H, 9.55%. IR (Nujol, cm⁻¹): 1922 ν (C==C), 938 ν (PMe₃). ¹H NMR (300 MHz, C₆D₆, 296 K, ppm): δ 1.22 (m, 36H, PCH₃), 5.00(s br, 1H, OH); ³¹P NMR (121 MHz, C₆D₆, 300 K, ppm): δ 20.8 (s, *P*CH₃).

2.3. X-ray structure determinations

Intensity data were collected on a Bruker SMART diffractometer with graphite-monochromated Mo K α radiation (λ = 0.71073 Å). Crystallographic data for complexes **4**, **7**, and **10** are summarized in Table 1. The structures were solved by direct methods and refined with full matrix least-squares on all F^2 (*shelxl*-97) with non-hydrogen atoms anisotropic.

3. Results and discussion

3.1. Synthesis of hydrido phenylthiolato iron(II) complexes

Reaction of $Fe(PMe_3)_4$ with thiophenols **1–3** to gave rise to the hydrido iron complexes **4–6** (Eq. (2)) via an oxidative addition of the S–H bond.

At -27 °C crystalline solids were obtained with yields of 17–28%. In the solid state complexes **4–6** rapidly decompose when exposed to air.

Table 1Crystallographic data for complexes 4, 7 and 10.

Complex	4	7	10×2
Empirical formula	C18H41BrFeP4S	C ₂₂ H ₅₄ FeP ₄ Si ₂	$C_{44}H_{100}Fe_2O_4P_8$
Formula weight	549.21	554.55	1052.70
T (K)	100(2)	293(2)	293(2)
Crystal system	monoclinic	monoclinic	monoclinic
Space group	$P2_1/c$	$P2_1/c$	C2/c
a (Å)	13.8039(7)	16.024	24.009
b (Å)	28.913(2)	10.587	19.446
c (Å)	12.8157(7)	20.696	17.018
α(°)	90	90	90
β (°)	90.327(4)	112.05	134.38
γ (°)	90	90	90
$V(Å^3)$	5114.8(5)	3254.2	5678.6
Ζ	8	4	4
D_{Calc} (g cm ⁻³)	1.426	1.132	1.231
Reflections measured	24688	17323	12078
Unique reflections	8974	6821	4880
R _{int}	0.2316	0.0916	0.0349
θ_{\max} (°)	25.00	27.04	25.00
$R_1 \left(I > 2\sigma \left(I \right) \right)$	0.0830	0.0563	0.0919
wR_2 (all data)	0.1321	0.1709	0.2435

 $Ar = p-BrC_6H_4$, $p-MeOC_6H_4$, $o-MeC_6H_4$

In the infrared spectra of complexes **4–6**, the characteristic v(Fe–H) bands are found at 1866, 1818 and 1834 cm⁻¹ because the electron-withdrawing Br group at the para-position makes the Fe-H bond stronger comparing with the electron-donating methoxy group at the para-position. The electron-donating methyl group on the ortho-position has less influence upon the Fe-H bond. The resonances of the Fe-H group in the ¹H NMR spectra at room temperature are registered between -9.99 and -14.20 ppm as a multiplet. The protons of the PMe₃ groups resonate in the range of 0.99-1.13 ppm. In the ³¹P NMR spectra three multiplets between 10.1-10.8 ppm, 16.4-16.6 ppm and 24.2-27.6 ppm are found with the intensity ratio of 1:2:1. This confirms that two of the four PMe₃ ligands have the same chemical surrounding. The spectroscopic data are compatible with an octahedral configuration around the Fe atom consisting of a P-Fe-P axis and equatorial coordination of P, P, S and H atoms. The expected molecular structure was confirmed by X-ray diffraction analysis of complex 4 (Fig. 1).

The molecular structure of complex **4** shows an octahedral geometry around the iron atom with atoms P5 and P6 in axial positions. The angle P5–Fe2–P6 of 162.75(16)° (Fig. 1) indicates the position of the hydrido ligand. H200, S2, P7, P6 are located in an equatorial plane. Owing to the stronger *trans*-influence of the hydr-



Fig. 1. Molecular structure of **4** and selected bond distances (Å) and angles (°): P5– Fe2 2.248(3), P6–Fe2 2.279(3), P7–Fe2 2.222(4), P8–Fe2 2.184(4), Fe2–H200 1.5091, S2–Fe2 2.355(4); P8–Fe2–P7 99.0(2), P8–Fe2–P5 93.6(1), P7–Fe2–P5 95.1(1), P8– Fe2–P6 99.5(1), P7–Fe2–P6 93.9(1), P5–Fe2–P6 162.8(2), P8–Fe2–S2 165.8(2), P7– Fe2–S2 95.0(1), P5–Fe2–S2 83.3(1), P6–Fe2–S2 81.3(1), P8–Fe2–H200 81.5, P7–Fe2– H200 179.4, P5–Fe2–H200 85.1, P6–Fe2–H200 85.8, S2–Fe2–H200 84.5.

3.2. Reaction of 6 with trimethylsilylacetylene

Hydrido iron(II) complex **6** reacts with trimethylsilylacetylene to give the bis(trimethylsilylethynyl)iron(II) complex **7** according to Eq. (3).



Complex **7** is an entirely different reaction product where the expected result [15,22] would be a vinyl compound. It can be proposed that the stronger basicity of the hydrido hydrogen in complex **6** is beneficial to the formation of H₂ and complex **7**. In the IR spectra the characteristic $v(C \equiv C)$ band was found at 1968 cm⁻¹. No absorption characteristic of a phenyl group was identified. In the ¹H NMR spectrum the singlet resonance of SiMe₃ protons is registered at 0.11 ppm while those of the PMe₃ ligands at 1.42 ppm also appear as singlet. In the ³¹P NMR spectrum, the PMe₃ group gave rise to a singlet signal at 20.0 ppm. Spectroscopic data are compatible with an octahedral configuration around the iron atom consisting of a C-Fe-C axis and equatorial P1, P2, P3, and P4 donor atoms. The molecular structure of complex **7** was confirmed by single crystal X-ray diffraction analysis.

The molecular structure of complex **7** is shown in Fig. 2, which shows an octahedral geometry around the iron atom with two alkynyl-C atoms in axial positions and four equatorial P-donor atoms.

In our earlier study cobalt and nickel hydrides reacted with alkynes affording products of an insertion reaction [22,24]. With



Fig. 2. Molecular structure of **7** and selected bond distances (Å) and angles (°): C6– Fe1 1.921(4), C1–Fe1 1.918(4), Fe1–P2 2.2359(8), Fe1–P1 2.2368(7), Fe1–P3 2.2410(8), Fe1–P4 2.2414(8), C2–C1 1.240(5), C6–C7 1.238(5); C1–Fe1–C6 179.8(1), C1–Fe1–P2 98.5(1) C6–Fe1–P2 81.3(1), C1–Fe1–P1 99.7(1), C6–Fe1–P1 80.5(1), P2–Fe1–P1 61.8(1), C1–Fe1–P3 81.2(1), C6–Fe1–P3 98.8(1), P2–Fe1–P3 91.5(1), P1–Fe1–P3 91.0(1), C1–Fe1–P4 80.5(1), C6–Fe1–P3 99.5(1), P2–Fe1–P4 91.7(1), P1–Fe1–P4 91.5(1), P3–Fe1–P4 161.7(1).



Scheme 1. Proposed mechanism of reaction (3).

iron hydrides reactivity is turned towards substituted products. A possible reaction mechanism is proposed in Scheme 1. In the first step, trimethylsilylacetylene reacts with a [Fe–H] function by elimination of dihydrogen yielding intermediate (**8**) which can react with another trimethylsilylacetylene molecule through elimination of thiophenol to give the bisalkynyl iron(II) complex **7** as end product.

3.3. Reaction of 6 with phenylacetylene

When phenylethyne was used in the reaction of complex **6**, complex **9** was directly obtained from pentane as yellow cubic crystals (Eq. (4)). Complex **9** is stable in the air for several hours at room temperature.



In the IR spectrum of complex **9** the characteristic $v(C \equiv C)$ band is registered at 2037 cm⁻¹. The PMe₃ protons resonate at 1.41 ppm. In the ³¹P NMR spectrum a singlet for the PMe₃ ligands was found at 20.9 ppm. The spectroscopic data are compatible with an octahedral configuration around the iron atom.

3.4. Reaction of 5 with 2-methyl-3-butyn-2-ol

By reaction of complex **5** with 2-methyl-3-butyn-2-ol under similar conditions complex **10** was obtained as yellow cubic crystals (Eq. (5)). In the IR spectrum of **10** the typical $v(C \equiv C)$ absorption appears at 1922 cm⁻¹, and the v(O-H) band is registered at 3442 cm⁻¹. The ¹H NMR spectrum shows PMe₃ protons as multiplet at 1.22 ppm. In the ³¹P NMR spectrum a singlet was found at 20.8 ppm for the PMe₃ ligands. The spectroscopic data are compatible with an octahedral configuration around the iron atom consisting of a C–Fe–C axis and equatorial coordination of P1, P2, P3, and P4 donor atoms. The molecular structure of complex **10** was confirmed by X-ray diffraction analysis.



The molecular structure of complex **10** is shown in Fig. 3, which is similar with complex **7**. The bending of [C=C-C]-chain away from 180° and some differences in Fe–P distances in complex **7** and **10** should be attributable to packing effects in the crystal structures.



Fig. 3. Molecular structure of **10** and selected bond distances (Å) and angles (°): Fe1-C1 1.955(8), Fe1-C1A 1.955(8), Fe1-P1 2.224(2), Fe1-P1A 2.224(2), Fe1-P2A 2.232(2), Fe1-P2 2.232(2), P1-C9 1.820(9), P1-C8 1.834(9), P1-C7 1.848(9), P2-C10 1.833(9), P2-C11 1.840(9), P2-C12 1.855(10); C1-Fe1-C1A 176.5(4), C1-Fe1-P1 98.2(2) C1A-Fe1-P1 79.4(2), C1-Fe1-P1A 79.4(2), C1A-Fe1-P1A 98.2(2), P1-Fe1-P1A 92.1(1), C1-Fe1-P2A 101.6(2), C1A-Fe1-P2A 80.9(2), P1-Fe1-P2A 160.3(1), P1A-Fe1-P2A 91.6(1), C1-Fe1-P2 80.9(2), C1A-Fe1-P2 101.6(2), P1-Fe1-P2 91.6(1), P1A-Fe1-P2 160.3(1), P2A-Fe1-P2 91.5(1).

4. Conclusion

The novel hydrido iron complexes **4–6** are formed by oxidative addition of S-H bond of the thiophenols. The structures of these hydrido iron complexes as derived from solution spectroscopy were confirmed for the solid state by single crystal X-ray diffraction. The reaction of the hydrido iron complexes with alkynes resulted in bisalkynyl iron(II) complexes. No products arising from an expected insertion reaction have been observed.

Supplementary data

CCDC 711798, 711796, and 711797 contain the supplementary crystallographic data for **4**, **7** and **10**. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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