Inorganica Chimica Acta 369 (2011) 40–44

Contents lists available at ScienceDirect

Inorganica Chimica Acta

journal homepage: www.elsevier.com/locate/ica

C–H activation by a terminal imidoiron(III) complex to form a cyclopentadienyliron(II) product

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ARTICLE INFO

Article history:
Available online 26 November 2010

This article is dedicated to Robert G. Bergman, a teacher, researcher, and mentor of the highest order.

Keywords:
Imido complexes
Iron
Half-sandwich complexes
C–H activation
Crystallography

ABSTRACT

Imido complexes of the late transition metals have the ability to activate C–H bonds through the abstraction of hydrogen atoms from hydrocarbons. This paper describes the apparent hydrogen atom transfer from cyclopentadiene (CpH) to the isolable imidoiron(III) complex $L^{Me}FeNAd$ (L^{Me} = 2,4-bis(2,6-diisopropylphenylimido)pent-3-yl; Ad = 1-adamantyl) in the presence of 4-*tert*-butylpyridine (^{t}Bupy). The isolated product is not the amidoiron(II) complex, but instead it is $L^{Me}Fe(Cp)(^{t}Bupy)$, the result of amido protonation by additional CpH. The crystal structures of both $L^{Me}FeCp$ and $L^{Me}Fe(Cp)(^{t}Bupy)$ are presented, and in the latter compound the Fe–C bonds are longer than in any previously characterized cyclopentadienyliron complex.

1. Introduction

Reactions that result in the removal of a hydrogen atom from a hydrocarbon play an important role in organometallic chemistry [1]. Though the term "C–H activation" is most often used to describe the single-step oxidative addition of a hydrocarbon to a metal complex as elucidated by Bergman and others [2,3], there are other ways to activate C–H bonds. For example, nature often uses enzymatic iron-oxo (Fe=O) species to break a C–H bond in hydrocarbons by abstracting a hydrogen atom; the resulting organic radical reacts with the iron hydroxide in a "rebound" step, with the ultimate result being formal insertion of an oxygen atom into the R–H bond to give ROH [4,5]. These natural hydroxylation reactions have inspired chemists to create synthetic iron-oxo complexes that hydroxylate hydrocarbons [6]. In an effort to design and understand the analogous insertion of an NR fragment into hydrocarbon C–H bonds, we have synthesized $L^{Me}Fe{=}NAd$ (L^{Me} = 2,4-bis(2,6-diisopropylphenylimido)pent-3-yl; Ad = 1-adamantyl), a rare example of an imidoiron(III) complex [7]. This complex has been isolated and characterized using crystallography, NMR, EPR, magnetism, and X-ray absorption spectroscopy [8]. In the presence of 4-*tert*-butylpyridine, it abstracts hydrogen atoms from hydrocarbons such as 1,4-cyclohexadiene and indene, giving an iron(II) amido complex and an organic radical. This C–H activation is the first step of a hypothetical catalytic cycle for hydrocarbon amination.

In this paper, we describe the reaction of $L^{Me}Fe{=}NAd$ with cyclopentadiene, a reaction in which the iron product has gained a cyclopentadienyl ligand. Cyclopentadienylmetal complexes are an important class of organometallic complexes, and have found many applications including olefin polymerization [9], asymmetric catalysis [10], and polymer science [11]. Protonation of metal–amido ($M–NR_2$) complexes by cyclopentadiene (CpH) is a useful route to metal–Cp complexes, especially for early transition metals [12–17]. The reaction of cyclopentadiene with metal–imido (M=NR) complexes, however, has received little attention, despite a renaissance in late metal–imido chemistry [18]. In literature reactions, CpH reacts with complexes containing both amido and imido ligands, but reacts only with the amido moiety [17]. Here, we demonstrate that an imidoiron(III) complex reacts with CpH to afford a cyclopentadienyliron(II) product with complete loss of the NR group. We give evidence that C–H activation of CpH through hydrogen atom abstraction is a key step in this reaction.

2. Experimental

2.1. General considerations

All manipulations were carried out under an N_2 atmosphere in an MBraun glovebox maintained at or below 1 ppm of O_2 and H_2O.

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doi:10.1016/j.ica.2010.11.031

Proton NMR spectra were recorded using a Bruker Avance 500 instrument at 25 °C, and chemical shifts were referenced to residual C_6D_5H at δ 7.16 ppm. IR spectra were recorded on a Shimadzu 8400S FTIR spectrometer as KBr pellets. UV–Vis spectra were recorded on a Cary 50 spectrometer using screw-cap cuvettes. Solution magnetic susceptibilities were determined using Evans' method [19]. Microanalysis samples were sealed into airtight cups in a VAC Atmospheres glovebox under argon and analyzed with a PerkinElmer 2400 Series II Analyzer. L^{Me}FeNAd [8], L^{Me}Fe(NHAd)(^{t}Bupy) [7] and NaCp [20] were synthesized using literature methods. $[L^{Me}FeBr]_2$ was synthesized analogously to $[L^{Me}FeCl]_2$ [27]. 4-*tert*-Butylpyridine (Aldrich) was distilled from CaH_2 and stored over activated 3 Å molecular sieves. Diethyl ether and pentane were purified by passage through activated alumina and Q5 columns from Glass Contour Co. (Laguna Beach, CA). Benzene-d_6 was dried over flame-activated alumina. Before use, an aliquot of each solvent was tested with a drop of sodium benzophenone ketyl in THF. All glassware was dried overnight at 150 °C, and Celite was dried at 250 °C overnight under vacuum.

2.2. Synthesis of L^{Me}Fe(Cp)(^{t}Bupy)(**1**·^{t}**Bupy**)

A resealable NMR tube was loaded with L^{Me}FeNAd (23.4 mg, 37.6 μmol), 4-*tert*-butylpyridine (22 μL, 150 μmol), and 0.2 mL C_6D_6. A solution of freshly cracked CpH in C_6D_6 (0.68 mL of a 0.11 M solution, 75 μmol) was added, causing an immediate color change from red to orange. The ^{1}H NMR spectrum showed that L^{Me}Fe(Cp)(^{t}Bupy) (**1**·^{t}**Bupy**) was formed in 50% yield versus an internal integration standard. At room temperature, **1**·^{t}**Bupy** exists in equilibrium with pyridine-free **1**, precluding full solution characterization. ^{1}H NMR (C_6D_6, 4 equiv ^{t}Bupy): δ 44 (1H, α-*H*), 12 (6H, C*H*$_3$), 9 (^{t}Bupy), 8 (^{t}Bupy), 1 (^{t}Bupy), −4 (12H, CH(C*H*$_3$)$_2$), −7 (4H, *m*-Ar or C*H*(CH$_3$)$_2$), −23 (5H, Cp-*H*), −27 (2H, *p*-Ar), −47 (12H, CH(C*H*$_3$)$_2$), −96 (4H, *m*-Ar or C*H*(CH$_3$)$_2$).

2.3. Synthesis of L^{Me}FeCp (**1**)

To a slurry of $[L^{Me}FeBr]_2$ (423 mg, 0.382 mmol) in Et_2O (10 mL) was added a slurry of NaCp (67 mg, 0.76 mmol) in Et_2O (2 mL). The mixture was stirred for 16 h, and the volatile materials were removed under vacuum. The residue was extracted into pentane (10 mL), and a white solid was removed by filtration over a pad of Celite. The orange supernatant solution was concentrated to 5 mL, and orange-brown crystals were deposited upon storing the solution at −45 °C. The mother liquor was decanted from the crystals and concentrated to 2 mL, affording a second crop of crystals at −45 °C. The crystalline products were combined and dried under vacuum (163 mg + 135 mg, total yield 73%). ^{1}H NMR (C_6D_6): δ 58 (1H, α-*H*), 19 (6H, C*H*$_3$), −5 (12H, CH(C*H*$_3$)$_2$), −10 (4H, *m*-Ar or C*H*(CH$_3$)$_2$), −13 (5H, Cp-*H*), −27 (2H, *p*-Ar), −51 (12H, CH(C*H*$_3$)$_2$), −105 (4H, *m*-Ar or C*H*(CH$_3$)$_2$). μ_{eff} (C_6D_6, 25 °C): 4.9 ± 0.1 μ_B. IR (KBr): 3057 (w), 3018 (w), 2962 (s), 2927 (m), 2868 (m), 1528 (s), 1433 (s), 1388 (s), 1317 (s), 1267 (m), 1252 (w), 1231 (w), 1175 (m), 1100 (w), 1055 (w), 1030 (w), 1009 (w), 933 (w) cm^{-1}. UV–Vis (pentane): 330 (10 000 M^{-1} cm^{-1}), 360 (sh, ~5500 M^{-1} cm^{-1}), 390 (sh, ~3300 M^{-1} cm^{-1}), 500 (sh, ~400 M^{-1} cm^{-1}), 880 (25 M^{-1} cm^{-1}) nm. *Anal.* Calc. for $C_{34}H_{46}FeN_2$: C, 75.82; H, 8.61; N, 5.20. Found: C, 75.66; H, 8.73; N, 5.14%.

2.4. Crystal structures of **1** and **1**·^{t}**Bupy**

Crystals were placed onto the tip of a ~0.1 mm diameter glass fiber and mounted on a Bruker SMART APEX II CCD Platform diffractometer [21] for a data collection at 100.0(1) K using Mo Kα radiation and a graphite monochromator. Randomly oriented regions of reciprocal space were surveyed: four major sections of frames were collected with 0.50° steps in ω at four different φ settings and a detector position of −33° in 2θ. The intensity data were corrected for absorption [22]. Final cell constants were calculated from the *xyz* centroids of *ca.* 4000 strong reflections from the actual data collection. The structures were solved using SIR97 [23] and refined using SHELXL-97 [24]. The space groups were determined by systematic absences ($P2_1/c$, **1**·^{t}**Bupy**) and intensity statistics ($P\bar{1}$, **1**). All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. In the structure of **1**·^{t}**Bupy**, a single non-negligible density peak near the Cp ligand (1.27 *e* Å^{-3}, ~2.25 Å from Fe1) was observed, consistent with a minor occupational disorder of Cp with a chloride ligand. When Cl was included in the model, the occupancy refined to 0.979:0.021 (Cp:Cl), i.e., ~2% occupancy by Cl. Since the overall statistics were not significantly improved, Cl was omitted from the final model. The structure of **1** was refined as a pseudomerohedral twin: application of twin law [1 0 0/0 −1 0/ 0 0 −1], a rotation around direct lattice [1 0 0], improved the *R*1 residual from 0.1747 to 0.0565. The final full matrix least squares refinement for **1** converged to *R*1 = 0.0565 (F^2, $I > 2\sigma(I)$) and *wR*2 = 0.1335 (F^2, all data), and for **1**·^{t}**Bupy** converged to *R*1 = 0.0449 (F^2, $I > 2\sigma(I)$) and *wR*2 = 0.1240 (F^2, all data).

3. Results and discussion

3.1. Reaction of imidoiron complex with cyclopentadiene

We recently showed that in the presence of 4-*tert*-butylpyridine (^{t}Bupy) the iron(III) imido complex L^{Me}FeNAd reacts with indene to cleanly form the iron(II) amido complex L^{Me}Fe(NHAd)(^{t}Bupy) through H-atom (H·) transfer [7,25]. Here we investigate the reaction of L^{Me}FeNAd with cyclopentadiene (CpH), a similar but smaller substrate. Two equivalents of freshly cracked CpH were added to a pentane solution of L^{Me}FeNAd and 4 equiv of ^{t}Bupy. The color changed from bright red to orange-brown within 10 s, and the ^{1}H NMR spectrum showed a new paramagnetic species as the major product. Interestingly, this ^{1}H NMR spectrum showed no amidoiron(II) complex L^{Me}Fe(NHAd)(^{t}Bupy), though this was the sole product of the reaction of L^{Me}FeNAd with indene [25]. The product of the CpH reaction was identified by X-ray crystallography as the half-sandwich iron(II) complex L^{Me}Fe(Cp)(^{t}Bupy) (**1**·^{t}**Bupy**) (Fig. 1b). The Fe–$N_{diketiminate}$ bond lengths (2.076(1) and 2.090(1) Å) are the longest of any (diketiminate)–iron complex in the Cambridge Structural Database (CSD) [26], perhaps as a result of steric pressure from the Cp and ^{t}Bupy ligands. The iron–Cp distance is also unusually long (Fe–$Cp_{centroid}$ = 2.11 Å; Fe–Cp_{plane} = 2.09 Å). This feature is discussed in further detail below.

3.2. Proposed mechanism

Based on the precedent of H-atom abstraction reactions of L^{Me}Fe(NAd)(^{t}Bupy) (e.g., with indene) [25], homolytic cleavage of the Cp–H bond by the imide is reasonable, thus affording the iron(II) amide L^{Me}Fe(NHAd)(^{t}Bupy) and Cp·. We have shown that the anilido complex L^{tBu}Fe(NHdipp) (dipp = 2,6-^{i}Pr$_2$C$_6$H$_3$; L^{tBu} = HC[C(^{t}Bu)N(2,6-^{i}Pr$_2$C$_6$H$_3$)]$_2$) is protonated by the weak acids HO^{t}Bu (pK_a = 32 in DMSO) and HCCPh (pK_a = 29 in DMSO) to give H_2Ndipp and the corresponding alkoxo and acetylide complexes [27]. Thus, if formed in the reaction, the amido complex L^{Me}Fe(NHAd)(^{t}Bupy) is expected to be protonated by CpH (pK_a = 18 in DMSO). To test this hypothesis, we added 1 equiv CpH to an independently prepared sample of L^{Me}Fe(NHAd)(^{t}Bupy) [7].

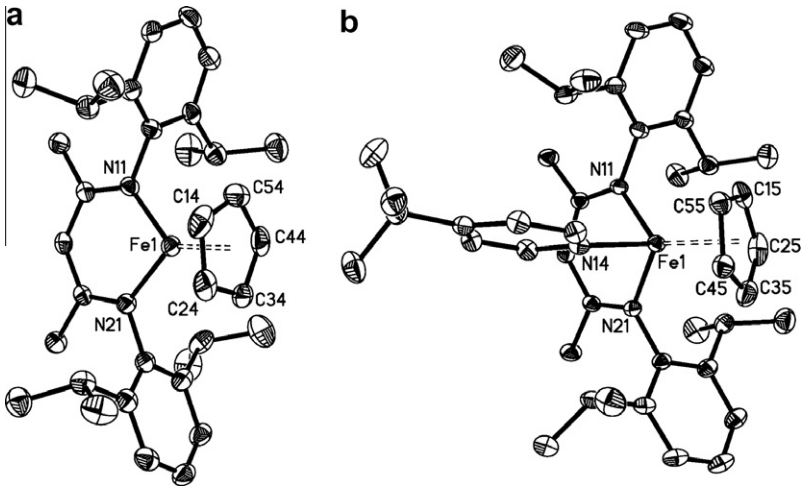
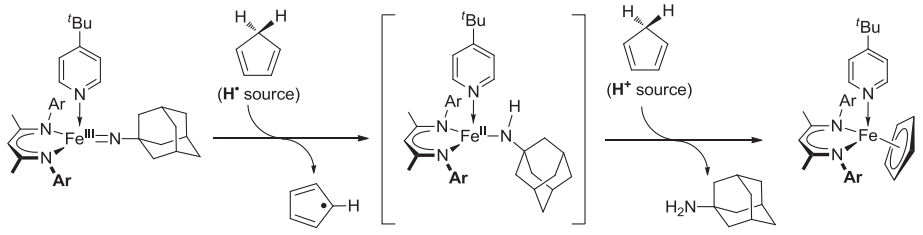


Fig. 1. Structures of (a) $L^{Me}FeCp$ (**1**) and (b) $L^{Me}Fe(Cp)(^{t}Bupy)$ (**1**·**^{t}Bupy**) using 50% probability ellipsoids. Hydrogen atoms are removed for clarity, and only one unique molecule in the asymmetric unit of **1** is shown. Selected bond distances (Å) and angles (°) for **1**: Fe1–N11 1.902(2); Fe1–N21 1.904(2); Fe1–Cp_{cent} 1.68, average C_{Cp}–C_{Cp} 1.402(6); N11–Fe1–N21 93.19(9); N11–Fe1–Cp_{cent} 133.3; N21–Fe1–Cp_{cent} 133.5. For **1**·**^{t}Bupy**: Fe1–N11 2.076(1); Fe1–N21 2.090(1); Fe–N14 2.221(1); Fe1–Cp_{cent} 2.11, average C_{Cp}–C_{Cp} 1.405(2); N11–Fe1–N21 89.61(4); N11–Fe1–N14 94.27(4); N21–Fe1–N14 99.33(4); N11–Fe1–Cp_{cent} 131.3; N21–Fe1–Cp_{cent} 123.3; N14–Fe1–Cp_{cent} 111.8.



Scheme 1.

The ^{1}H NMR spectrum immediately showed complete conversion to **1**·**^{t}Bupy** (91% yield), showing that $L^{Me}Fe(NHAd)(^{t}Bupy)$ is a kinetically competent intermediate in the conversion of $L^{Me}FeNAd$ to **1**·**^{t}Bupy**. Together, the data are consistent with the proposed mechanism in Scheme 1.

An alternative description of the second step in Scheme 1 could be a second H· transfer from CpH, giving iron(I) and Cp·, which would then form the observed iron(II)/Cp^{-} product via electron transfer. However, less acidic substrates such as 1,4-cyclohexadiene do not react with $L^{Me}Fe(NHAd)(^{t}Bupy)$, thus arguing against this alternative mechanism.

3.3. Synthesis of pyridine-free $L^{Me}FeCp$ (**1**)

For comparison, we also synthesized the pyridine-free complex $L^{Me}FeCp$ (**1**). Complex **1** was synthesized as a bright orange solid in 73% yield from $[L^{Me}FeBr]_2$ by anion metathesis with NaCp. Eight paramagnetically shifted peaks are observed in the ^{1}H NMR spectrum of **1**, consistent with an average C_{2v} symmetry on the NMR timescale. A broad resonance in the ^{1}H NMR spectrum of **1** can be assigned to the Cp hydrogens based on its integration of five protons. The ability to observe these protons by ^{1}H NMR spectroscopy is surprising because α and β protons in the alkyl group of (diketiminate)Fe(alkyl) are too broad to be observed [28,29]. The solution effective magnetic moment of **1** (4.9 μ_B) is consistent with a high-spin iron(II) ground state ($S = 2$). Complex **1** was also characterized by X-ray crystallography (Fig. 1a). The complex crystallizes with two independent molecules in the asymmetric unit, and both show very similar structural features. In contrast to **1**·**^{t}Bupy**, compound **1** has a more typical Fe–Cp distance (Fe–$Cp_{centroid}$ = 1.68 Å; Fe–Cp_{plane} = 1.68 Å).

We also investigated the conversion of **1** to **1**·**^{t}Bupy**. Interestingly, the ^{1}H NMR chemical shifts of **1** are dependent on the concentration of ^{t}Bupy between 70 mM and 3.1 M, indicating that ^{t}Bupy binds **1** rapidly, reversibly, and weakly. An equilibrium

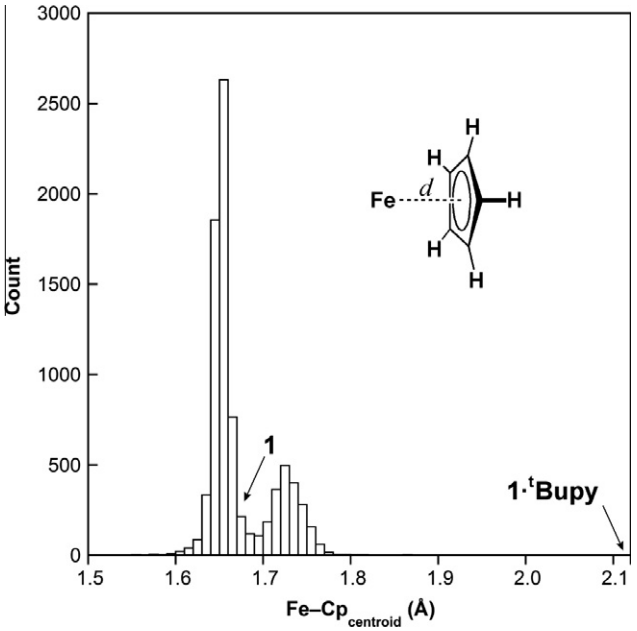


Fig. 2. Histogram of all iron–Cp centroid distances for iron–C_5H_5 in the CSD (n = 8187) [26]. The locations of compounds **1** and **1**·**^{t}Bupy** are indicated.

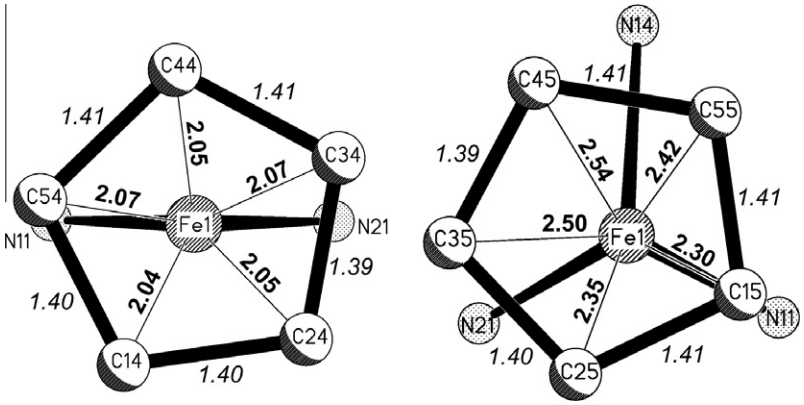


Fig. 3. Comparison of the cores of **1** (left) and **1**·^{t}**Bupy** (right), looking perpendicular to the C_5H_5 plane. Fe–C (in bold) and C–C (in italic) distances are given in Å. The atoms N11 and N21 are the ligating atoms from the diketiminate ligands, and N14 is the ligating atom from ^{t}Bupy.

constant of $K_{eq} = 1.3 \pm 0.1\ M^{-1}$ was calculated from the [^{t}Bupy] dependence of the chemical shifts using an iterative method [30]. This binding constant is ~10^4 smaller than the binding affinity of pyridine to the iron(II) complex $L^{tBu}FeCl$ ($K_{eq} = 41\,000 \pm 6000$) [31]. This is most likely a steric effect, in which the bulkier Cp ligand creates a much smaller pyridine binding pocket. This steric effect is also manifested in the Fe–N_{py} bond length of 2.221(1) Å in **1**·^{t}**Bupy**, which is significantly longer than iron–pyridine distances in other complexes of the type (diketiminate)Fe^{II}(X)(py) (average Fe–N_{py} = 2.12(2) Å, n = 9) [26].

3.4. Structural comparison of Fe–Cp complexes

The presence of ^{t}Bupy significantly changes the Fe–Cp interaction, as evidenced by the lengthening of the Fe–$Cp_{centroid}$ distance from **1** (1.68 Å) to **1**·^{t}**Bupy** (2.11 Å). To compare these distances to literature values, we surveyed the Cambridge Structural Database [26] for all crystallographically characterized Fe–C_5H_5 distances (Fig. 2). The Fe–Cp centroid distance in compound **1** is unremarkable, lying near the mean Fe–C_5H_5 distance of 1.68 ± 0.04 Å. In contrast, however, **1**·^{t}**Bupy** lies significantly outside the range of previously observed distances.

A closer inspection of the coordination sphere of **1**·^{t}**Bupy** shows that although the Cp ring is planar (largest deviation from the least-squares plane is 0.0018 Å), the Cp ring is slightly tilted (6.9°) with respect to the Fe–$Cp_{centroid}$ vector, and pushed away from the crowding pyridine (Fig. 3). As a result, the two Fe···C interactions abutting the pyridine ligand (2.54 and 2.50 Å) are slightly longer than the other three (2.30, 2.35, and 2.42 Å). This could be described as partial ring slippage, resulting in a resonance description lying between η^3-C_5H_5 and η^5-C_5H_5 coordination modes. However, there is no apparent contribution from the "ene-allyl" resonance form of Cp^- [32], since there is no significant contraction in any of the C–C bonds in the Cp. In summary, the remarkable change in the Fe–Cp distance between **1** and **1**·^{t}**Bupy** demonstrates the ability of exogenous donors to modulate the metal–Cp interaction.

4. Concluding remarks

We have demonstrated that a late-metal imido complex can be a useful synthon for assembling half-sandwich compounds. In particular, the reaction of L^{Me}Fe(NAd)(^{t}Bupy) with CpH likely proceeds through a stepwise reaction in which the first step is hydrogen atom abstraction to form the metal amide L^{Me}Fe(NHAd)(^{t}Bupy). This is similar to the reactions of **1**·^{t}**Bupy** with other H· sources such as 1,4-cyclohexadiene and indene [25], in which the weak C–H bond is homolyzed. Cyclopentadiene is also acidic enough to protonate the amido ligand in the amidoiron(II) species, affording the observed final product, L^{Me}Fe(Cp)(^{t}Bupy) (**1**·^{t}**Bupy**). The deprotonation of weak acids by a group 8 amido complex has been characterized thoroughly by Bergman and coworkers [33]. The structural changes between **1** and **1**·^{t}**Bupy** are also remarkable, with the Cp ligand pushed 0.43 Å further away from Fe in the presence of ^{t}Bupy. The greatly decreased Fe–Cp bonding in **1**·^{t}**Bupy** causes the overall binding of the pyridine to **1** to be surprisingly weak.

Acknowledgements

P.L.H. acknowledges funding from the National Science Foundation (CHE-0911314), and R.E.C. acknowledges the NSF and the University of Rochester for fellowships. Microanalytical data were obtained at the CENTC Elemental Analysis Facility funded by the NSF (CHE-0650456). We thank Dr. William Brennessel for assistance with X-ray crystallography and for obtaining microanalytical data.

Appendix A. Supplementary material

CCDC 785971 and 785972 contain the supplementary crystallographic data for **1**·^{t}**Bupy** and **1**, respectively. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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