Abstract: A homoleptic organometallic Fe\textsuperscript{IV} complex that is stable in both solution and in the solid state at ambient conditions has been synthesized and isolated as [Fe(phtmeimb)\textsubscript{2}](PF\textsubscript{6})\textsubscript{2} (phtmeimb = [phenyl(tris(3-methylimidazo-2-ylidene))borate]). This Fe\textsuperscript{IV} N-heterocyclic carbene (NHC) complex was characterized by \textsuperscript{1}H NMR, HR-MS, elemental analysis, scXRD analysis, electrochemistry, Mössbauer spectroscopy, and magnetic susceptibility. The two latter techniques unequivocally demonstrate that [Fe(phtmeimb)\textsubscript{2}](PF\textsubscript{6})\textsubscript{2} is a triplet Fe\textsuperscript{IV} low-spin \textit{S} = 1 complex in the ground state, in agreement with quantum chemical calculations. The electronic absorption spectrum of [Fe(phtmeimb)\textsubscript{2}](PF\textsubscript{6})\textsubscript{2} in acetonitrile shows an intense absorption band in the red and near IR, due to LMCT (ligand-to-metal charge transfer) excitation. For the first time the excited state dynamics of a Fe\textsuperscript{IV} complex was studied and revealed a \textit{\sim}0.8 ps lifetime of the \textsuperscript{3}LMCT excited state of [Fe(phtmeimb)\textsubscript{2}](PF\textsubscript{6})\textsubscript{2} in acetonitrile.

High-valent iron complexes, both heme- and non-heme, function as active key intermediates in various biological catalytic cycles and important organic transformations.\textsuperscript{[1]} This has inspired the development of synthetic chemistry of Fe\textsuperscript{IV} complexes.\textsuperscript{[1–15]} Several heteroleptic high-valent Fe\textsuperscript{IV} complexes have been reported, where the high oxidation state is stabilized by terminal \pi-donating auxiliary ligands (PDALs) such as oxide, nitride, imide, isocyanide, and ketimide, with various stabilities.\textsuperscript{[16–17]} In particular, Fe\textsuperscript{IV}-oxo complexes have been found capable of various oxidative transformations.\textsuperscript{[18]} Examples of Fe\textsuperscript{IV} coordination compounds without stabilizing PDALs are scarce however. Besides Fe\textsubscript{IV} that could be isolated in a matrix,\textsuperscript{[9]} there are only few examples of more traditional Werner complexes using for example, electron-donating dithiocarbamate ligands to stabilize the iron center in oxidation state Fe\textsuperscript{IV}\textsuperscript{[10–12]} or Fe\textsuperscript{IV} complexes containing multidentate macrocyclic tetraamide ligands.\textsuperscript{[13]} a Fe\textsuperscript{IV} cyclam–azide complex\textsuperscript{[14]} and a Fe\textsuperscript{IV} cyano complex based on a tridentate imino-ligand.\textsuperscript{[15]} The latter was electrochemically generated and studied in situ. To date, the exceptional example of an entirely stable Fe\textsuperscript{IV} complex belongs to the class of coordination cage compounds; Fe\textsuperscript{IV} hexahydrazone clathrochelate.\textsuperscript{[16–17]} This compound shows infinite stability in both aqueous and non-aqueous solutions as well as in the solid state, unique to synthetic Fe\textsuperscript{IV} complexes.

For organometallic iron complexes the electron-donating effect of carbanions can be exploited to stabilize higher metal oxidation states which has led to a current interest in homoleptic Fe\textsuperscript{IV} organometallic complexes (Figure 1).\textsuperscript{[17]} Three of the reported complexes constitute Fe\textsuperscript{IV} tetralkyl species (Figure 1, 1–3, Table S4).\textsuperscript{[18–c]} The Fe\textsuperscript{IV} state of these compounds is however only accessible via disproportionation reactions and suffers from thermal instability and air sensitivity at ambient conditions (Figure 1, Table S4).\textsuperscript{[18–c]} Similarly, also the recently described organometallic Fe\textsuperscript{IV} complex [Fe(Cp\textsubscript{2})\textsuperscript{2}+ (Cp\textsuperscript{+} = pentamethylcyclopentadienyl)] (Figure 1, 4) is thermally stable at room temperature only in the solid state under inert atmosphere.\textsuperscript{[18d]} We have recently reported that the strongly \pi-donating N-heterocyclic carbene (NHC) ligands form homoleptic Fe\textsuperscript{IV} and Fe\textsuperscript{IV} complexes with a hexa-NHC coordination sphere leading to long photo-induced charge-transfer states.\textsuperscript{[19–21]} The complexes being [Fe(btz)\textsubscript{2}](PF\textsubscript{6})\textsubscript{2}\textsuperscript{[20]} and [Fe(btz)\textsubscript{2}](PF\textsubscript{6})\textsubscript{2}\textsuperscript{[22]} (btz\textsuperscript{[22]} = 3,3’-dimethyl-1,1’-bis(p-tolyl)-4,4’-bis(1,2,3-triazol-5-ylidene), and [Fe(phtmeimb)\textsubscript{2}](PF\textsubscript{6})\textsubscript{2}\textsuperscript{[23]} (phtmeimb\textsuperscript{[23]} = [phenyl-tris(3-

(tris(3-methylimidazolin-2-ylidene)borate)\(^{−}\), the latter complex building on Felhammer’s scorpionate ligand (htmeimb = [hydrodi(tris(3-methylimidazolin-2-ylidene)borate)]) and corresponding Fe\(^{II}\) complex, [Fe(htmeimb)]\(_2\)X (X = BF\(_4\), BPh\(_4\)).\(^{[24]}\) Using the analogous phtmeimb ligand developed by Smith,\(^{[22]}\) another effect of the strong \(\sigma\)-donor NHC ligands above is a pronounced stabilization of higher metal oxidation states apparent from the remarkably low Fe\(^{II}\)\(^\text{III} /\text{II}\) redox potential.\(^{[20, 21, 24b]}\) In case of the scorpionate NHC ligand (phtmeimb), we obtained indications for the formation of a stable Fe\(^{IV}\) complex at modest oxidizing potentials,\(^{[21]}\) something also observed by Felhammer for the Fe\(^{IV}\) complex of htmeimb.\(^{[24b]}\)

These observations attracted our attention as examples of Fe\(^{IV}\)-NHCs, outside the electrochemical observation of the Fe\(^{IV}\) scorpionates above, are exclusively found among heteroleptic complexes, having PDALs as additionally stabilizing groups (Table S3), resulting in complexes of various stabilities.\(^{[14a, c, 5b, 23]}\)

Here, we present the synthesis and isolation of the stable homoleptic hexa-coordinated, [Fel(ptmeimb)]\(_2\)\(_{\text{PF}}\_6\) (Figure 1). The combined spectroscopic, electrochemical, magnetic and theoretical investigations characterize this complex as a Fe\(^{III}\) complex. Its \(S = 1\) ground state undergoes a ligand-to-metal charge transfer (LMCT) transition and we present the dynamics of the \(^{1}\)LMCT excited state that complements our previous observations of \(^{1}\)MLCT and \(^{1}\)LMCT states with Fe\(^{IV}\) and Fe\(^{III}\) NHC complexes, respectively.\(^{[19–21]}\)

As previously reported, the Fe\(^{III}\) precursor [Fel(ptmeimb)]\(_2\)\(_{\text{PF}}\_6\) undergoes a reversible one-electron electrochemical oxidation (\(E_{1/2} = 0.25\) V vs. ferrocene) that was assigned to the Fe\(^{IV}\)\(^\text{III} /\text{II}\) redox couple based on the spectroelectrochemical changes.\(^{[21]}\) Chemical oxidation of the red Fe\(^{III}\) complex of [Fel(ptmeimb)]\(_2\)\(_{\text{PF}}\_6\) in acetonitrile with thianthrenylhexafluorophosphate radical (\(E_{1/2} = 0.85\) V vs. ferrocene)\(^{[26]}\) produced a dark green colored solution of the Fe\(^{IV}\) compound [Fel(phtmeimb)]\(_2\)\(_{\text{PF}}\_6\), which could be isolated pure in 95% yield as green crystals (see Scheme 1), as proven by the combination of \(^1\)H NMR spectroscopy, HR-MS, IR, elemental analysis, scXRD analysis, electrochemistry, \(^{57}\)Fe Mößbauer spectroscopy, magnetic susceptibility and quantum chemical calculations. [Fel(phtmeimb)]\(_2\)\(_{\text{PF}}\_6\) is stable in the solid state exposed to air as well as in acetonitrile solution for days at ambient temperature. In addition, the crystals of [Fel(ptmeimb)]\(_2\)\(_{\text{PF}}\_6\) are stable when washed with water, however slow decomposition was observed when water was added to a solution of the complex in acetonitrile.

The \(^1\)H spectrum of [Fel(ptmeimb)]\(_2\)\(_{\text{PF}}\_6\) shows highly deshielded resonances in comparison to its Fe\(^{III}\) congener and the resonances are rather well-resolved (See Supporting Information).\(^{[21]}\) Dark-green single crystals of [Fel(ptmeimb)]\(_2\)\(_{\text{PF}}\_6\) suitable for scXRD analysis were grown in a saturated anhydrous acetonitrile solution of [Fel(ptmeimb)]\(_2\)\(_{\text{PF}}\_6\) by slow diffusion of anhydrous diethyl ether at room temperature exposed to air (see section S4, Supporting Information, for details). The molecular structure shows an octahedral iron center surrounded by two fac-tridentate phtmeimb ligands (Figure 2).

\(\text{Fe}–\text{C}\) bond lengths are 1.99 to 2.01 Å, which are close to that of the Fe\(^{III}\) congener (1.96 to 2.01 Å).\(^{[21]}\) Similarly, the Fe–C bite angles (87.3° to 87.7°) for [Fel(ptmeimb)]\(_2\)\(_{\text{PF}}\_6\) are similar to the Fe\(^{III}\) congener (86.2° to 87.6°).\(^{[21]}\) Thus, [Fel(ptmeimb)]\(_2\)\(_{\text{PF}}\_6\) exhibits a close to perfect octahedral geometry that is virtually identical to its Fe\(^{III}\) salt (details in section S4, Supporting Information). This indicates that very little structural re-organization energy is needed when altering between the Fe\(^{IV}\)/Fe\(^{III}\) oxidation states in [Fel(ptmeimb)]\(_2\), thus facilitating rapid electron transfer processes involving this couple. The very similar Fe–C distances between the Fe\(^{IV}\) and Fe\(^{III}\) oxidation states, as well as the Fe–C distances themselves, have also been observed for a heteroleptic macrocyclic tetra-NHC complex in the two oxidation states, as Fe\(^{IV}\)-O and Fe\(^{III}\)-O–Fe\(^{III}\) complexes.\(^{[20b]}\) We suggest that the very minor difference in Fe–C bond lengths is due to the rigidity of the tridentate phtmeimb ligand and/or the covalent nature of this bond, where the influence of the formal charge differences on iron is of little importance for this bond length in the latter case.

The \(^{57}\)Fe Mößbauer spectrum of [Fel(ptmeimb)]\(_2\)\(_{\text{PF}}\_6\) is shown in Figure 3a). The isomer shift \(\delta\) and electric quadrupole moment \(\epsilon Q\) are

![Figure 1](https://example.com/figure1.png)

**Figure 1.** All reported organometallic homoleptic Fe\(^{IV}\) complexes.

![Figure 2](https://example.com/figure2.png)

**Figure 2.** scXRD Molecular structure of the cation in [Fel(ptmeimb)]\(_2\)\(_{\text{PF}}\_6\). Thermal ellipsoids are shown at 50% probability. Hydrogen atoms, counter ions and solvent molecules are omitted for clarity. Orange = Fe; purple = B; blue = N, and grey = C.

![Scheme 1](https://example.com/scheme1.png)

**Scheme 1.** Synthesis of complex [Fel(ptmeimb)]\(_2\)\(_{\text{PF}}\_6\) from [Fel(phtmeimb)]\(_2\)\(_{\text{PF}}\_6\).
are shown in Figure 5. The pronounced and differ from the doublet at 87 K of the Fe

The combination of an unusual large energy at 80 K. This assignment can now be supported from the experimental data of the isolated complex and computational data (vide infra) that the transition occurs from the triplet ground state (\( ^3 \text{GS} \)) with a \((t_{2g})^6\) electronic configuration to a \(^1\text{LMCT} \) state.

In previous studies of complexes \([\text{Fe(btz)}]^{2+}\) and \([\text{Fe(btz)}]^{3+}\), we showed that the same NHC ligand set furnishes both the \( \text{Fe}^{II} \) and \( \text{Fe}^{III} \) oxidation states with exceptional lifetimes (hundreds of picoseconds) of their \(^1\text{LMCT} \) and \(^3\text{MLCT} \) excited states, respectively. With the relative photostability of \([\text{Fe(phthmeimb)}]^{2+}\) in acetonitrile solution (Section S8, Supporting Information), we have the opportunity to compare the excited state dynamics following LMCT excitation of a \( \text{Fe}^{II} \) complex to the recently reported record 2.0 ns lifetime of the \(^2\text{LMCT} \) state of a Fe complex, featured by the \( \text{Fe}^{III} \) congener. Transient absorption spectra following 800 nm excitation of \([\text{Fe(phthmeimb)}]^{2+}\), shown in Figure 5. The pronounced ground state bleach (GSB), peaking at 715 nm, and the excited state absorption (ESA) at wavelengths below around 600 nm are readily rationalized in terms of the spectral differences arising from the \( \text{Fe}^{III} \) to \( \text{Fe}^{II} \) reduction. The additional excited state absorption at wavelengths above around 900 nm can be attributed to the oxidation of the phthmeimb ligand in analogy to the spectrum of the LMCT excited state of \([\text{Fe(phthmeimb)}]^{2+}\) that involves the same ligand oxidation.\cite{21}

Figure 3. (a) \(^{57}\text{Fe} \) Möllbauer spectrum of \([\text{Fe(phthmeimb)}]^{2+}\) at 80 K. (b) Experimental magnetic data and model fits for \([\text{Fe(phthmeimb)}]^{2+}\). Main panel: magnetic susceptibility, represented by the \(x'/x\) product in the temperature range 2–300 K. Insert: magnetization data for the same sample recorded at \( T = 2–10 \) K and \( B = 0.5, 1.0, 2.0, 3.0, 4.0, \) and 5.0 T.

Figure 4. Electronic absorption spectra of \([\text{Fe(phthmeimb)}]^{2+} \) and \([\text{Fe(phthmeimb)}]^{3+} \) in deaerated acetonitrile, and the \( \text{Fe}^{II} - \text{Fe}^{III} \) differential spectrum.\( (-\cdots-) \).
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the transient absorption decays with a similarly fast ground state recovery. Only a minor component with a 16 ps lifetime that contributes to the ground state recovery and the transient absorption between 850 and 1000 nm points to the involvement of additional states in the deactivation of the LMCT excited state.

Quantum chemical calculations of different relaxed spin states corroborate the nature and structure of the ground state as a triplet state. Figure 6 shows the calculated ground state spin density of the \([\text{Fe(phtmeimb)}]^{2+}\) complex, which, together with the calculated Mulliken spin density on the iron for this state (Table 1) supports an assignment of the ground state as a triplet \(\text{Fe}^{3+}\) complex with \(t_{2g}^6\) character, with some admixing of the frontier molecular orbitals with NHC-\(\pi\) contributions.

![Image](https://example.com/fig5.png)

**Figure 5.** Transient absorption results for \([\text{Fe(phtmeimb)}]^{+}\) in deaerated acetonitrile—full spectral decays (top), and kinetics at 400 nm, 540 nm, 650 nm, and 1100 nm (bottom).

The transient-absorption spectrum hence corroborates the assignment of the 715 nm absorption band of \([\text{Fe-(phtmeimb)}]^{+}\) to an LMCT transition from the \(t_{2g}\) low-spin ground state \((^3\text{GS})\) to a \(t_{2g}^5\ \pi^1\ \text{LMCT}\) excited state. Most of the transient absorption decays with a \(\approx 0.8\) ps lifetime, indicating fast deactivation of the \(\text{LMCT}\) state accompanied by similarly fast ground state recovery. Only a minor component with a 16 ps lifetime that contributes to the ground state recovery and the transient absorption between 850 and 1000 nm points to the involvement of additional states in the deactivation of the \(t_{2g}\) LMCT excited state.

Quantum chemical calculations of different relaxed spin states corroborate the nature and structure of the ground state as a triplet state. Figure 6 shows the calculated ground state spin density of the \([\text{Fe(phtmeimb)}]^{2+}\) complex, which, together with the calculated Mulliken spin density on the iron for this state (Table 1) supports an assignment of the ground state as a triplet \(\text{Fe}^{3+}\) complex with \(t_{2g}^6\) character, with some admixing of the frontier molecular orbitals with NHC-\(\pi\) contributions.

![Image](https://example.com/fig6.png)

**Figure 6.** Calculated spin density for the optimized lowest energy state of \([\text{Fe(phtmeimb)}]^{2+}\). Spin-up density is in blue and spin down in green.

| State | \(E_{av}\) [eV] | \(R_{av}(\text{Fe-C})\) [Å] | Fe Spin |
|-------|----------------|-----------------|---------|
| \([\text{Fe(phtmeimb)}]^{2+}\)| 1.26 | 2.00 | 0.00 |
| \([\text{Fe(phtmeimb)}]^{2+}\)| 0.00 | 2.021 | 2.00 |
| \([\text{Fe(phtmeimb)}]^{2+}\)| 1.84 | 2.129 | 3.41 |

The synthesized and isolated \([\text{Fe(phtmeimb)}]^{+}\)(PF\(_6\))\(_2\) complex, is stable in the solid state and acetonitrile solution at ambient conditions. The facile tunability of the NHC ligand systems provide potential for stabilization of high-valent metal complexes in general. We have built on Fehlhammer’s initial observation,\(^{24}\) and developed an organometallic high-valent \(\text{Fe}^{3+}\) NHC complex without additional stabilizing \(\pi\)-donating ligands,\(^{34c,1c,10,21,2b,6}\) stable at ambient conditions, utilizing the strongly \(\pi\)-donating mono-anionic facial \(\text{tris-NHC scorpionate}\) ligand phtmeimb developed by Smith.\(^{23}\) The excited state dynamics of homoleptic \([\text{Fe(phtmeimb)}]^{+}\)(PF\(_6\))\(_2\) was studied, constituting the first of its kind study of a \(\text{Fe}^{3+}\) complex. We observed the fundamental difference in excited state lifetimes for \(\text{Fe}^{3+}\)-LMCT (2 ns, 2.1 eV) vs. \(\text{Fe}^{3+}\)-LMCT (0.8 ps, < 1.5 eV) states, which deserves further investigation beyond the scope of the present paper. Moreover, while advanced designs and studies are needed for a better understanding of \(\text{Fe}^{3+}\) NHC complexes, modifications that aim at further prolonging the \(t_{2g}\) LMCT state through subtle tuning of the energy levels are also in progress. Finally, having access to stable \(\text{Fe}^{3+}\) species, with long lived \(t_{2g}\) LMCT state that can take part in redox/photocatalytic processes/cycles, is an appealing area for future exploration.

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**Conflict of interest**

The authors declare no conflict of interest.
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