

An Icosahedral 55-Atom Iron Hydride Cluster Protected by tri-*tert*-Butylphosphines

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ABSTRACT: Nanoclusters are nanometer-sized molecular compounds characterized by significant metal-metal bonding and low average oxidation states, and they exhibit unique properties distinct from those of small metal complexes or nanoparticles. Unlike noble metals stable in metallic forms, the synthesis of nanometer-sized iron clusters has been precluded by the relatively weak iron-iron bonds and the high reactivity of low oxidation state iron, despite the extensive history of molecular iron compounds. Here we report the synthesis and characterization of a cationic 55-atom iron cluster with a 1.2 nm icosahedral core. Its twelve vertices are occupied by tri-*tert*-butylphosphines, while multiple hydrides cover the surface. This core can be viewed as a substructure of the face-centered cubic (fcc), in contrast to the body-centered cubic (bcc) for the stable bulk phase. This work reveals a stable structure and fundamental properties of a nanometer-sized iron cluster, which have remained elusive for decades, and the simple synthetic protocol provides a route to explore molecular nanochemistry.

INTRODUCTION

Atomically precise synthesis of metal nanoclusters has facilitated the exploration of unique structures and functionalities that are absent in bulk materials or conventional nanoparticles.¹⁻⁹ Unlike conventional nanoparticles, which exhibit size distributions (*e.g.*, 5% in a histogram, even for highly monodispersed samples), metal nanoclusters possess identical structures with no variation in molecular composition. This uniformity enables scientists to investigate the fundamental relationships between the sizes and structures of nanoclusters and their properties, such as catalysis, optical properties, and magnetism, at single-atom resolution.¹⁰⁻¹⁶ Significant progress in the field has been driven by structure determination through X-ray crystallography, as structural elucidation at the nanoscale is typically elusive using conventional techniques, particularly for the interface between the metal core and the surface ligands.¹⁷ Since the seminal work on the structure determination of thiolate-protected gold and silver nanoclusters,¹⁸⁻²² substantial efforts have been made to acquire valuable information on their structural construction and evolution rules. This has enabled the development of a comprehensive library of crystal structures, facilitating the mapping of size- and structure-dependent properties. Such advancements have further led to the discovery of coinage metal nanoclusters (*i.e.*, gold, silver, and copper) protected by a wide range of ligands.²³⁻²⁵

In contrast to the diverse range of sizes and structures discovered for coinage metal nanoclusters, the family of iron (Fe) clusters remains limited to the subnanometer scale (*i.e.* < 1 nm)²⁶⁻²⁸ owing to the relatively weak iron-iron bonds²⁹ and the high reactivity of low oxidation state iron.³⁰ For example, Fe carbonyl clusters have been studied since the 1970s, with the largest known cluster being [Fe₉C(CO)₁₆]²⁻ featuring a central C atom connecting six Fe atoms.²⁶ In 2015, Betley *et al.* reported [Fe₈(^{trien}L)₂(PMe₂Ph)₂] supported by multidentate ligands (^{trien}LH₉ = [*o*-H₂NC₆H₄NH(CH₂)₂]₃N, Me = methyl, Ph = phenyl), which exhibits metal-metal exchange interactions with antiferromagnetic coupling.²⁷ More recently, Agapie *et al.* reported the synthesis of [Tp*₄W₄Fe₁₃S₁₂] (Tp* = tris(3,5-dimethylpyrazolyl)borate), which features a central Fe₁₃ core.²⁸

Fe nanoclusters are expected to exhibit intriguing properties in catalysis and magnetism.³¹ While bulk Fe displays ferromagnetism at room temperature, the ferromagnetic phase in Fe nanoparticles diminishes as the particle size decreases below a certain threshold, known as the superparamagnetic limit.³² Fundamental investigations into such quantum size effects in ultrasmall Fe nanoparticles require detailed structural elucidation at the atomic level. Therefore, uncovering the structures of Fe nanoclusters in the >1 nm size region is crucial. Here we report the synthesis and characterization of a cationic 55-atom Fe nanocluster containing forty-six hydrides and twelve phosphines (referred to as [Fe₅₅]), which is paired with an anionic 6-atom Fe cluster protected by eight hydrides and six amides

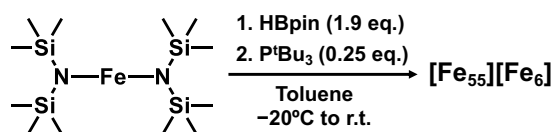
(referred to as $[\text{Fe}_6]$). This $[\text{Fe}_{55}][\text{Fe}_6]$ ionic pair is formulated as $[\text{Fe}_{55}\text{H}_{46}(\text{P}^t\text{Bu}_3)_{12}][\text{Fe}_6\text{H}_8\{\text{N}(\text{SiMe}_3)_2\}_6]$ ('Bu = *tert*-butyl).

RESULTS AND DISCUSSION

Synthesis and crystal structure. As shown in Scheme 1, the highly reproducible synthesis of the ionic pair $[\text{Fe}_{55}][\text{Fe}_6]$ involves a simple one-pot reaction of an Fe^{II} bis-amide complex, $\text{Fe}\{\text{N}(\text{SiMe}_3)_2\}_2$,³³ with pinacolborane (HBpin) and tri-*tert*-butylphosphine (P^tBu_3) in toluene. Before the addition of P^tBu_3 , the mixture of $\text{Fe}\{\text{N}(\text{SiMe}_3)_2\}_2$ and HBpin at -20°C is expected to form “Fe-hydride” intermediates for cluster growth through metathesis between Fe-N and H-B bonds.^{34,35} An analogous metathesis is possible by using diisobutylaluminium hydride instead of HBpin, as demonstrated by Jacobi von Wangelin *et al.*³⁶ The resulting reaction mixture was gradually warmed to room temperature and filtered to remove insoluble materials. From the filtrate, $[\text{Fe}_{55}][\text{Fe}_6]$ was obtained in the form of dark brown crystals in 13% yield (see Supporting Information for details). Notably, the aggregation of Fe atoms is typically an irreversible reaction step, making the 13% isolated yield sufficiently high, given that it follows at least 55 irreversible reaction steps.

The product $[\text{Fe}_{55}][\text{Fe}_6]$ is highly air-sensitive and decomposes instantly upon exposure to air, both in solution and in the solid state. These results highlight the high reactivity of $[\text{Fe}_{55}][\text{Fe}_6]$, which may originate from the ionic pair formed by the $[\text{Fe}_{55}]$ cation and the $[\text{Fe}_6]$ anion. To eliminate the influence of the iron atoms in the $[\text{Fe}_6]$ anion, further anion exchange was attempted. However, the reactions of $[\text{Fe}_{55}][\text{Fe}_6]$ with $\text{NaBAR}^{\text{F}}_4$, $\text{LiB}(\text{C}_6\text{F}_5)_4$, and $[\text{NEt}_4][\text{PF}_6]$ resulted in complex mixtures. The addition of an excess (10 equiv.) of PMe_3 to a toluene solution of isolated $[\text{Fe}_{55}][\text{Fe}_6]$ caused gradual decomposition at ambient temperature, while an analogous reaction with PEt_3 resulted in the partial degradation accompanied by partial ligand exchange between P^tBu_3 (see Supporting Information for details).

Using the same $\text{Fe}\{\text{N}(\text{SiMe}_3)_2\}_2$ precursor, Chaudret *et al.* synthesized cube-shaped Fe nanoparticles of ~ 7 nm diameter through hydrogenolysis (3 bar H_2) at 150°C in the presence of a long-chain acid and a long-chain amine.³⁷ A major difference in the present reaction is the use of HBpin (Scheme 1), which operates at low temperatures and enables the trapping of smaller, molecular products with suitable protecting ligands.



Scheme 1. Synthesis of $[\text{Fe}_{55}][\text{Fe}_6]$.

Single-crystal X-ray crystallography confirmed the structure of $[\text{Fe}_{55}][\text{Fe}_6]$ (Figure 1), which crystallized in the $R\bar{3}$ space group (Figure 1(b) and Table S3). The Fe atoms of $[\text{Fe}_{55}]$ exhibit a Mackay icosahedron³⁸ with a shell-by-shell growth pattern, while $[\text{Fe}_6]$ displays an octahedral core with each Fe atom bound to a silylamide. In the core of $[\text{Fe}_{55}]$ (Figure 2), the central Fe atom is covered by the first Fe_{12} icosahedral shell, forming an Fe_{13} icosahedron. The Fe_{13} core is further enclosed by a second Fe_{42} icosahedral layer, leading to the formation of the Fe_{55} icosahedron with a diameter of 1.2 nm. The twelve vertices of the Fe_{55} icosahedron are occupied by P^tBu_3 ligands.

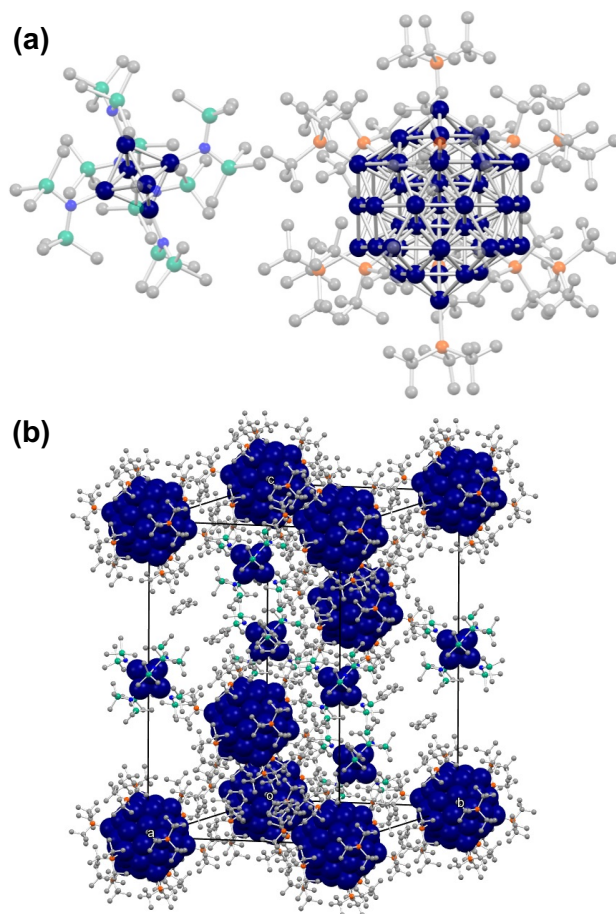


Figure 1. (a) Crystal structure of $[\text{Fe}_{55}][\text{Fe}_6]$ depicted in a ball-and-stick model. Hydrogen atoms and the crystal solvent (toluene) are omitted for clarity. Color legend: dark blue, Fe; orange, P; teal, Si; light blue, N; gray, C. (b) The unit cell of $[\text{Fe}_{55}][\text{Fe}_6]$ in the $R\bar{3}$ space group.

The inner Fe_{13} atoms, comprising the central Fe atom and the first Fe_{12} shell, also form an ideal icosahedron, as evaluated by the ratio of averaged radial (r) and transverse (d) Fe-Fe distances. The mean radial Fe-Fe distance between the central Fe atom and the first Fe_{12} shell is $r = 2.4418 \pm 0.006 \text{ \AA}$, while the transverse Fe-Fe distances within the first Fe_{12} shell are averaging $d = 2.569 \pm 0.065 \text{ \AA}$. Their ratio ($2.4409/2.568 = 0.950$) closely matches the geometry requirement ($r = 0.95d$) for an icosahedron. It is interesting to note that the Mackay icosahedral arrangement of Fe atoms in $[\text{Fe}_{55}]$ can be considered a substructure of face-centered cubic (fcc), whereas the

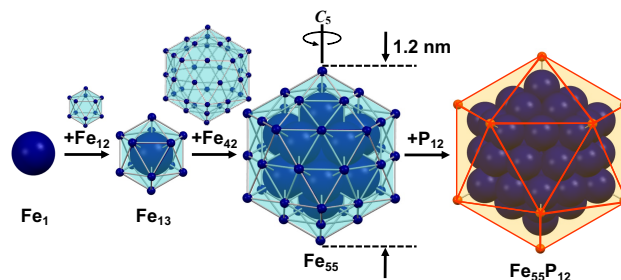


Figure 2. Detailed structural dissection of $[\text{Fe}_{55}]$, illustrating its icosahedral layer-by-layer configuration.

stable bulk phase of Fe at room temperature is known to be body-centered cubic (bcc).

In the crystallographic analysis, we were unable to refine the positions of the forty-six hydrides in $[\text{Fe}_{55}]$. However, the short inner Fe-Fe distances within the Fe_{55} core suggest that the hydrides are located on the surface. The inner Fe-Fe distances, including the mean radial and transverse distances discussed above, as well as the averaged radial distance between the first Fe_{12} shell and the second Fe_{42} shell ($r = 2.511 \pm 0.031 \text{ \AA}$), are short and close to the Fe-Fe distances in bulk Fe (*i.e.*, $2.48 \text{ \AA}$). On the other hand, the transverse Fe-Fe distances within the surface Fe_{42} layer are longer and averaging $d = 2.617 \pm 0.029 \text{ \AA}$. These Fe-Fe distances are shorter than the average Fe-Fe distance ($2.795(2) \text{ \AA}$) of the hydride-encapsulating Fe_4H moiety of an Au_3Fe_4 cluster,³⁹ undermining the possibility of hydride penetration in $[\text{Fe}_{55}]$. Computational studies suggested the locations of surface hydrides of $[\text{Fe}_{55}]$ and $[\text{Fe}_6]$. For $[\text{Fe}_{55}]$, several energetically close structures were found (Table S5). This is reasonable as the positions of hydrides in polyhydride complexes are typically flexible and thus exchangeable.^{40,41} In the lowest energy structure of $[\text{Fe}_{55}]$, 39 hydrides are localized around the phosphine-bound Fe atoms (Figure S3; 27 triply bridging and 12 doubly bridging hydrides), leaving 7 hydrides to occupy the centers of the triangular surfaces of the icosahedron (6 triply bridging and 1 doubly bridging hydrides). In the case of $[\text{Fe}_6]$, the eight hydrides in the lowest energy structure each cap one of the triangular Fe_3 surfaces of the octahedron (Table S6).

Some compounds relevant to $[\text{Fe}_{55}]$ should also be noted.^{42–46} The gas-phase detection of a bare Fe_{55} nanocluster was reported by Schooss *et al.* in 2013, where a polytetrahedron was proposed as the ground state structure.⁴² This is in stark contrast to the Mackay icosahedron observed in $[\text{Fe}_{55}]$. Structurally related nanoclusters featuring a Mackay icosahedral 55-metal-atom core have been synthesized in the forms of $\text{Pd}_{55}(\text{P}^i\text{Pr}_3)_{12}(\mu_3\text{-CO})_{20}$ (^iPr = isopropyl), $\text{Au}_{133}(\text{SPh-}p\text{-Bu})_{52}$ ($\text{SPh-}p\text{-Bu}$ = *para-tert-butyl* thiophenolate), and $[\text{Cu}_{43}\text{Al}_{12}](\text{C}_5\text{Me}_5)_{12}$.^{43–46}

Mass spectrometry and deuterium labeling experiments. Electrospray ionization (ESI) mass spectrometric analysis was performed to determine the formulae of the $[\text{Fe}_{55}]$ cation and the $[\text{Fe}_6]$ anion. In a positive-mode spectrum, a single strong peak was observed at mass/charge ratio (m/z) of 2773.0 (Figure 3), corresponding to $[\text{Fe}_{55}\text{H}_{46}(\text{P}^i\text{Bu}_3)_{12}]^{2+}$ (calculated $m/z = 2773.0$). The negative-mode spectrum shows a single peak at $m/z = 1276.3$, which can be assigned to $[\text{Fe}_6\text{H}_8\{\text{N}(\text{SiMe}_3)_2\}_5 + 2\text{H}_2\text{O} + 3\text{O}_2]^-$ (calculated $m/z =$

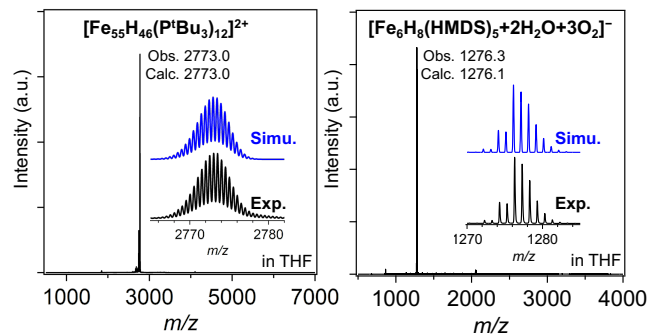


Figure 3. Positive-mode (left) and negative-mode (right) ESI mass spectra of $[\text{Fe}_{55}][\text{Fe}_6]$ in THF. Insets show the observed and simulated isotope patterns of the major peaks. HMDS = $\text{N}(\text{SiMe}_3)_2$.

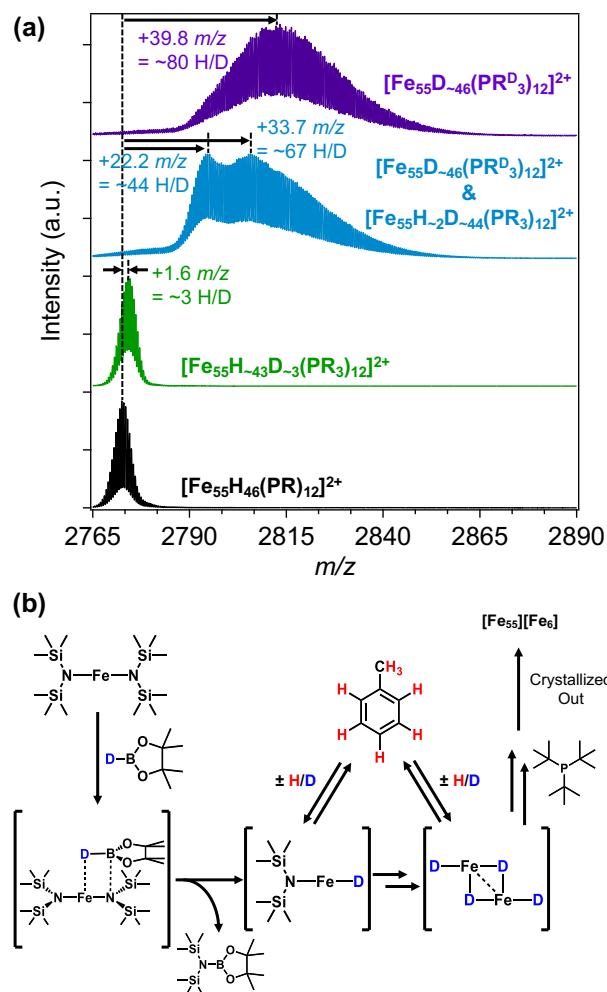


Figure 4. Deuterium labeling in the synthesis of $[\text{Fe}_{55}][\text{Fe}_6]$ and the ESI-MS spectra. (a) Positive-mode ESI mass spectra of $[\text{Fe}_{55}][\text{Fe}_6]$ in the absence and presence of deuterium sources. (b) Schematic illustration of H/D exchange during the synthesis of $[\text{Fe}_{55}][\text{Fe}_6]$ in the presence of DBpin in toluene. R = ^iBu , R^D = deuterated ^iBu .

1276.1) (Figure 3). In both spectra, the simulated isotope patterns match well with the experimentally observed ones, further confirming the peak assignments.

To gain further insight, we synthesized deuterium (D) labeled $[\text{Fe}_{55}][\text{Fe}_6]$ using deuterated pinacolborane (DBpin)⁴⁷ instead of HBpin, followed by ESI-MS analysis to observe the m/z shift. If all 46 hydrides in $[\text{Fe}_{55}\text{H}_{46}(\text{P}^i\text{Bu}_3)_{12}]^{2+}$ are replaced by deuterides, the mass number should increase by 46, resulting in a $+23 m/z$ shift for the dicationic peak of $[\text{Fe}_{55}\text{D}_{46}(\text{P}^i\text{Bu}_3)_{12}]^{2+}$. However, the positive-mode ESI mass spectrum of the deuterated sample showed a peak shift of only $1.6 m/z$ compared to $[\text{Fe}_{55}\text{H}_{46}(\text{P}^i\text{Bu}_3)_{12}]^{2+}$ (Figure 4(a), green). This shift corresponds to approximately ~ 3 H/D exchanges, indicating that the deuterated $[\text{Fe}_{55}]$ contains significantly fewer D than expected due to the exchange between D atoms on Fe and H atoms in toluene (Figure 4(b)). To test this hypothesis, we synthesized $[\text{Fe}_{55}][\text{Fe}_6]$ using HBpin in toluene- d_8 . The resultant ESI mass spectrum displayed two broad peaks with increased m/z of $+22.2$ and $+33.7$, respectively (Figure 4(a), blue). These can be assigned to $[\text{Fe}_{55}\text{H}_{-2}\text{D}_{-44}(\text{P}^i\text{Bu}_3)_{12}]^{2+}$ and $[\text{Fe}_{55}\text{D}_{-46}(\text{PR}^D_3)_{12}]^{2+}$ (R^D = deuterated ^iBu groups), supporting the incorporation of deuterium from

toluene- d_8 in this reaction. Further ESI-MS analysis was performed for $[\text{Fe}_{55}][\text{Fe}_6]$ synthesized using DBpin in toluene- d_8 . The positive-mode spectrum exhibited a broader peak with a larger shift of 39.8 m/z (Figure 4(a), purple), indicating the exchange of approximately 80 hydrogen atoms in $[\text{Fe}_{55}]$ with deuterium atoms during the synthesis. This number of incorporated deuterium atoms exceeds the 46 hydrides originally present in $[\text{Fe}_{55}]$, suggesting that the H/D exchange occurs not only with toluene but also with the $t\text{Bu}$ groups of P^tBu_3 through the reversible C-H(D) bond cleavage. Thus, the broad signals observed in Figure 4(a) (blue and purple) indicate the presence of $[\text{Fe}_{55}]$ cluster species with varying numbers of deuterium atoms, while we cannot entirely rule out the possibility of solvent (THF) binding to the cluster during measurements.

In contrast to the high activity of intermediary Fe-hydride species toward C-H(D) bond cleavage, the absence of H/D exchange between the product $[\text{Fe}_{55}][\text{Fe}_6]$ and toluene- d_8 indicates that the protecting ligands (P^tBu_3 and $\text{N}(\text{SiMe}_3)_2$) prevent toluene- d_8 from approaching the Fe centers once $[\text{Fe}_{55}]$ and $[\text{Fe}_6]$ are formed. The small molecular size of D_2 enables its approach to the Fe centers. However, $[\text{Fe}_{55}][\text{Fe}_6]$ gradually decomposed in the presence of D_2 in toluene (Figure S12), giving a nearly colorless solution and a black precipitate. Dissociation of P^tBu_3 would account for this phenomenon.

Negative-mode ESI-MS was also performed for the $[\text{Fe}_{55}][\text{Fe}_6]$ sample prepared in the presence of DBpin in toluene, revealing a prominent peak with minimal shift compared to the original $[\text{Fe}_6\text{H}_8\{\text{N}(\text{SiMe}_3)_2\}_2 + 2\text{H}_2\text{O} + 3\text{O}_2]^-$ anion (Figure S4). This result suggests that the deuterated $[\text{Fe}_6]$ contains significantly fewer D atoms than expected, mirroring the observations from the positive-mode analysis of $[\text{Fe}_{55}]$. The negative-mode spectra of samples synthesized using HBpin in toluene- d_8 and DBpin in toluene- d_8 exhibited peak shifts of approximately 10 m/z compared to non-deuterated $[\text{Fe}_6]$ in both cases. This shift is analogous to the theoretically predicted number of eight hydrides arranged in a face-capping manner on the octahedral Fe_6 core (*vide supra*), a structural type of which is precedent with a phosphine-protected cobalt hydride cluster.³⁵

To gain further insight into the number of hydrides in $[\text{Fe}_{55}][\text{Fe}_6]$, we quantified the labile hydrogen atoms by thermolysis of crystalline $[\text{Fe}_{55}][\text{Fe}_6]$ at 180 °C (Tables S1-S2 and Figures S5-S7). The amount of released H_2 was analyzed as 27.7(2) equivalents relative to $[\text{Fe}_{55}][\text{Fe}_6]$, consistent with the presence of 46+8 hydrides (27 equiv. of H_2 in total) in $[\text{Fe}_{55}][\text{Fe}_6]$.

NMR spectra. The ^1H NMR spectrum of $[\text{Fe}_{55}][\text{Fe}_6]$ in toluene- d_8 exhibited two broad signals corresponding to the $t\text{Bu}$ groups of P^tBu_3 at 1.24 ppm and the methyl groups of $\text{N}(\text{SiMe}_3)_2$ at 0.07 ppm (Figures S15 and S16). However, signals for hydrides were not detected, likely due to broadening from their proximity to the magnetic Fe centers. Despite the magnetism exhibited by $[\text{Fe}_{55}][\text{Fe}_6]$ (*vide infra*), the $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum still allowed the detection of carbon signals from the $t\text{Bu}$ and SiMe_3 groups. The successful observation of both ^1H and ^{13}C signals enabled the acquisition of 2D ^1H - ^{13}C correlation NMR spectra. Additionally, combined with distortionless enhancement by polarization transfer (DEPT)-135 ^{13}C NMR, the ^1H and ^{13}C signals from the $t\text{Bu}$ and SiMe_3 groups were unambiguously assigned. Detailed discussion on peak assignments is provided in the Supporting Information.

Electronic characterization of iron centers. In order to assess the charge state of Fe atoms of $[\text{Fe}_{55}][\text{Fe}_6]$, Fe K-edge X-ray absorption fine structure (XAFS) measurements were conducted at a synchrotron facility. The solid samples were sealed in plastic films, placed in aluminum bags, and the spectra were measured in

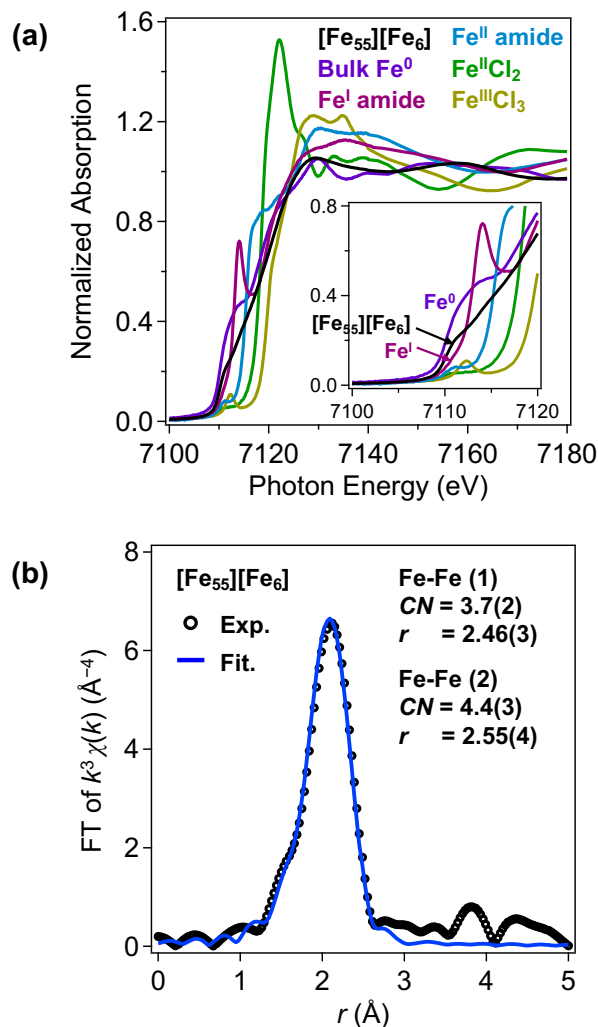


Figure 5. XAFS analysis of $[\text{Fe}_{55}][\text{Fe}_6]$. (a) Fe K-edge XANES spectra of $[\text{Fe}_{55}][\text{Fe}_6]$ and reference samples. The inset shows enlarged spectra of the onset region. (b) FT-EXAFS analysis of $[\text{Fe}_{55}][\text{Fe}_6]$. Fe^{II} amide = $\text{Fe}\{\text{N}(\text{SiMe}_3)_2\}_2$, Fe^{I} amide = $[\text{K}(\text{18-crown-6})][\text{Fe}\{\text{N}(\text{SiMe}_3)_2\}_2]$, CN = coordination number, r = distance (Å).

transmission mode at room temperature. For comparison, several reference samples were also measured, including Fe^0 powder (bulk Fe^0), $[\text{K}(\text{18-crown-6})][\text{Fe}^{\text{I}}\{\text{N}(\text{SiMe}_3)_2\}_2]$ (Fe^{I} amide),⁴⁸ $\text{Fe}^{\text{II}}\{\text{N}(\text{SiMe}_3)_2\}_2$ (Fe^{II} amide),³³ $\text{Fe}^{\text{II}}\text{Cl}_2$, and $\text{Fe}^{\text{III}}\text{Cl}_3$. The onset of the X-ray absorption near edge structure (XANES) spectra, which corresponds to the oxidation state of Fe, shifts to higher energy region in the order from bulk Fe^0 , $[\text{Fe}_{55}][\text{Fe}_6]$, Fe^{I} amide, Fe^{II} amide, FeCl_2 to FeCl_3 (Figure 5(a)). This result indicates that the average oxidation state of Fe in $[\text{Fe}_{55}][\text{Fe}_6]$ lies between 0 and +1. The observation is consistent with the formal charge of +60/61, which was calculated from the formula of $[\text{Fe}_{55}\text{H}_{46}(\text{P}^t\text{Bu}_3)_{12}][\text{Fe}_6\text{H}_8\{\text{N}(\text{SiMe}_3)_2\}_6]$. The absorption onset of $[\text{Fe}_{55}][\text{Fe}_6]$ closely resembles that of bulk Fe, indicating the metallic state of the majority of Fe in $[\text{Fe}_{55}][\text{Fe}_6]$.

Extended X-ray absorption fine structure (EXAFS) analysis of these samples was also conducted to gain insights into the local environment of Fe atoms in $[\text{Fe}_{55}][\text{Fe}_6]$ (Figure S18). The EXAFS of Fe^{I} amide and Fe^{II} amide, which corresponds to the Fe-N (and remote Fe-Si) bonds, displayed oscillations with different periodicities

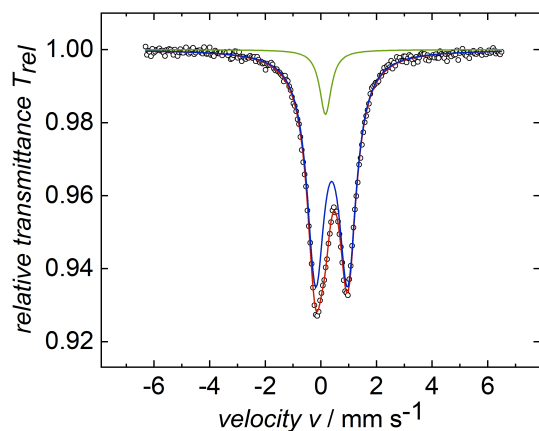


Figure 6. The zero-field ^{57}Fe Mössbauer spectrum of $[\text{Fe}_{55}][\text{Fe}_6]$ at 77 K. Collected data are represented by black circles. The blue trace represents the best fit of Fe species 1, and the green trace represents the best fit of Fe species 2. The red trace represents the best combined fit with the parameters given in the text.

compared to $[\text{Fe}_{55}][\text{Fe}_6]$, indicating minimal contributions from Fe-N bonds in the EXAFS of $[\text{Fe}_{55}][\text{Fe}_6]$. Curve fitting analysis of the Fourier-transformed (FT) EXAFS of $[\text{Fe}_{55}][\text{Fe}_6]$ confirmed adequate fitting results without the inclusion of Fe-P bonds (Figure S5(b)). The EXAFS spectra of $\text{Fe}^{\text{II}}\text{Cl}_2$ and $\text{Fe}^{\text{III}}\text{Cl}_3$ showed oscillations related to Fe-Cl, which are expected to be similar to Fe-P due to the comparable electron density of the two elements in terms of XAFS. Comparison of the EXAFS spectra with each reference sample revealed that the EXAFS of Fe powder exhibited a periodicity similar to that of $[\text{Fe}_{55}][\text{Fe}_6]$, suggesting that its EXAFS oscillations primarily arise from Fe-Fe bonds. Indeed, FT-EXAFS of $[\text{Fe}_{55}][\text{Fe}_6]$ could be fitted with two Fe-Fe bonds having coordination numbers of 3.7(2) and 4.4(3) and r values (distances) of 2.46(3) and 2.55(4) Å, respectively (Figure S5(b)). These values are consistent with the crystal structure of $[\text{Fe}_{55}][\text{Fe}_6]$, where average coordination numbers of 3.5/4.5 can be calculated for Fe-Fe bonds shorter/longer than 2.57 Å, respectively. The r values of the fitting (*i.e.*, 2.46(3) and 2.55(4) Å) are also close to the Fe-Fe bond distances in the crystal structure of $[\text{Fe}_{55}][\text{Fe}_6]$ (see Figure S1 for details), indicating the reliability of the data collection and the preceding discussion. The curve fitting analysis achieved a satisfactory R value (7.6%) without including Fe-P bonds, possibly due to the small coordination number in the crystal structure (*i.e.*, $12/(55+6) = 0.20$).

To further assess the electronic state of the Fe atoms, zero-field ^{57}Fe Mössbauer spectra of $[\text{Fe}_{55}][\text{Fe}_6]$ were recorded on different batches of microcrystalline samples at 77 K with varying velocity scales (Figure S19). As seen in Figures 1 and 2, $[\text{Fe}_{55}][\text{Fe}_6]$ features at least four types of Fe sites: one from the Fe atoms of $[\text{Fe}_6]$, and three from $[\text{Fe}_{55}]$; namely, the inner Fe_{13} icosahedron, the phosphine-free surface Fe atoms, and the phosphine-protected vertices of the surface Fe atoms. Despite these Fe atoms' different local environments and symmetries, the reproducible Mössbauer spectra displayed no magnetic splitting but a single yet asymmetric doublet (Figure 6).⁴⁹ This doublet could not be satisfactorily fitted with different line widths but equal integrated areas. Instead, a second Fe species had to be invoked. The resulting fit features two Fe species in a 91:9 ratio (Figure S19), corresponding to the $[\text{Fe}_{55}]$ and $[\text{Fe}_6]$ cluster units, respectively. Thus, the fit results indicated the superposition of a doublet signal from chemically inequivalent Fe atoms in

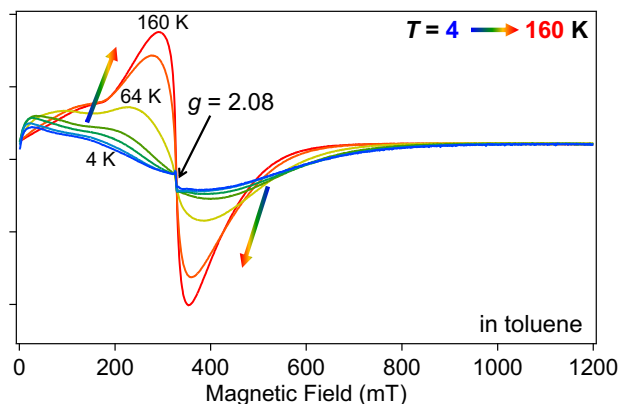


Figure 7. Variable-temperature ESR spectra of $[\text{Fe}_{55}][\text{Fe}_6]$ in toluene from 4 to 160 K.

$[\text{Fe}_{55}]$ (isomer shift $\delta = 0.39(1) \text{ mm s}^{-1}$, quadrupole splitting of $\Delta E_Q = 1.15(2) \text{ mm s}^{-1}$, line width $\Gamma_{\text{FWHM}} = 0.78(2) \text{ mm s}^{-1}$) and a singlet for the $[\text{Fe}_6]$ cluster ($\delta = 0.17(1) \text{ mm s}^{-1}$ and $\Gamma_{\text{FWHM}} = 0.52(2) \text{ mm s}^{-1}$).

Magnetic properties. Superconducting quantum interference device (SQUID) measurement was performed to study the magnetic properties of $[\text{Fe}_{55}][\text{Fe}_6]$. The magnetization versus field plot of $[\text{Fe}_{55}][\text{Fe}_6]$ collected at 1.8 K shows a nearly closed hysteresis with a coercive field of 165 Oe (Figure S20). Saturation magnetization of *ca.* 155 Bohr magnetons (μ_B) indicates a large magnetic moment in $[\text{Fe}_{55}]$. The magnetization versus field plot recorded at 300 K shows no linear region within the field range probably due to partial magnetization saturation. The saturation prevented us from applying a strong magnetic field for dc magnetic susceptibility measurement. Therefore, variable-temperature dc magnetic susceptibility data were collected for $[\text{Fe}_{55}][\text{Fe}_6]$ at a weak field of 10 Oe, showing a $\chi_M T$ product of $33531 \text{ cm}^3 \text{ K/mol}$ at 300 K (Figure S21A). With decreasing temperature, the $\chi_M T$ product of $[\text{Fe}_{55}][\text{Fe}_6]$ monotonically decreases, suggesting antiferromagnetic interactions. The relaxation dynamics of $[\text{Fe}_{55}][\text{Fe}_6]$ were explored through ac magnetic susceptibility measurements with a 3 Oe (1 Hz) or 2 Oe (for 10, 100, and 1000 Hz) oscillating ac field and zero dc field (Figure S21B). From 20 to 100 K, the out-of-phase (χ_M'') susceptibility data show no peak in the range of 1 to 1000 Hz. The non-zero values of χ_M'' below 20 K indicate slow magnetic relaxation in $[\text{Fe}_{55}][\text{Fe}_6]$, which is out of the detectable range.

Further analysis was performed using electron spin resonance (ESR) spectroscopy on $[\text{Fe}_{55}][\text{Fe}_6]$ in toluene at 300 K, revealing a relatively narrow and sharp line at $g_{\text{eff}} \approx 2$ (Figure S22A). At 4 K, a sharp $S = 1/2$ signal appeared at $g_{\text{eff}} \approx 2.08$, which may be assignable to $[\text{Fe}_6]$. Additionally, a broad spectrum with significantly increased linewidth appeared (Figure 7 and Figure S22B). Upon warming the frozen solution to 160 K, the intensity of the broad spectrum increased, accompanied by a shift in the apparent resonance field and a decrease in linewidth. Similar spectra were observed in ESR measurements of powdered samples. These results deviate from Curie's law and sharply contrast with conventional paramagnetic Fe complexes, indicating a property unique to our compound.

CONCLUSIONS

We synthesized and isolated a 1.2 nm diameter 55-atom Fe cluster and characterized it through crystallographic and spectroscopic analyses. The straightforward one-pot synthetic protocol, involving the

reaction of a metal-amide complex with HBpin and phosphines, has proven effective. As demonstrated in our previous studies on Fe and Co clusters^{34,35}, the choice of protecting ligands can significantly change the resultant product structures. Therefore, by employing appropriate ligands to stabilize nanoclusters, it is possible to expand the scope of synthetically accessible nanoclusters of Fe and Co. These nanoclusters have the potential to open new scientific avenues by elucidating their properties and reactivities.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website.

Experimental details, supporting figures, supporting tables, supplementary discussion, and Cartesian coordinates of the optimized structures (PDF)

Optimized coordinates of the 55-iron cluster (.xyz)

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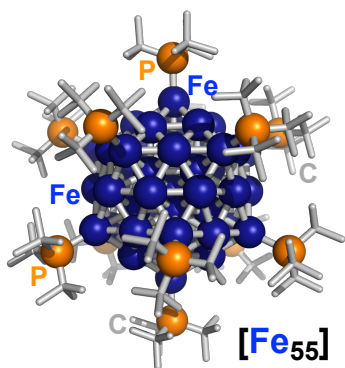
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Supporting information for
An Icosahedral 55-Atom Iron Hydride Cluster Protected by tri-*tert*-Butylphosphines

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This file contains

Experimental Details,

additional references,

Figures S1-S25 and accompanying supplementary discussion,

Tables S1-S7 and accompanying supplementary discussion,

and Cartesian coordinates of the optimized structures

Experimental Details

General considerations. Unless otherwise noted, all reactions were performed under an inert nitrogen or argon atmosphere using Schlenk techniques and glove boxes. Hexane, toluene, and tetrahydrofuran (THF) were dried and deoxygenated by passing through columns of activated alumina and supported copper catalyst purchased from Hansen & Co., Ltd. NMR spectra, including ^1H , $^{13}\text{C}\{^1\text{H}\}$, HSQC, and HMBC, were recorded on a Bruker Avance 800 spectrometer. Signals were referenced to the residual peaks of the deuterated solvents. Fourier-transform infrared spectroscopy (FT-IR) was performed in a glove box using a Bruker Optics ALPHA FT-IR equipped with a Diamond ATR accessory. Electrospray ionization mass spectrometry (ESI-MS) was performed using a Bruker MicroTOF-II instrument. Elemental analyses were carried out using a J-Science Micro Corder JM11, with crystalline samples sealed in tin capsules under an inert atmosphere. Cyclic voltammograms were recorded on a BAS ALS-660A electrochemical analyzer in a glove box. The electrolyte, tetra-*n*-butylammonium hexafluorophosphate, $[\text{nBu}_4\text{N}][\text{PF}_6]$, was recrystallized from THF prior to use. Gas chromatographic analysis (GC) was carried out using Shimadzu GC-2014s. Low-temperature experiments in glove boxes were carried out using a TechnoSigma UCR-150GB. Deuterated solvents (*e.g.*, toluene- d_8) were dried over sodium metal before use. Fe powder (FEE14PB, >99%(2N), fine powder *ca.* 5~10 μm) was purchased from Kojundo Chemical Laboratory Co. Ltd. for use as the Fe(0) reference sample for XAFS analysis. $\text{Fe}\{\text{N}(\text{SiMe}_3)_2\}_2$ ³³, $[\text{K}(18\text{-crown-6})][\text{Fe}\{\text{N}(\text{SiMe}_3)_2\}_2]$ ⁴⁸, and DBpin⁴⁷ were prepared according to previously reported procedures. All other chemicals were purchased from commercial sources and used without further purification.

Synthesis of $[\text{Fe}_{55}\text{H}_{46}(\text{P}^t\text{Bu}_3)_{12}][\text{Fe}_6\text{H}_8\{\text{N}(\text{SiMe}_3)_2\}_6]\cdot(\text{toluene})$. $\text{Fe}\{\text{N}(\text{SiMe}_3)_2\}_2$ (0.500 g, 1.33 mmol) was dissolved in toluene (1.5 mL) to give a clear green solution. This solution was stirred for 30 min at -20°C . HBpin (0.365 mL, 2.52 mmol) was added dropwise, and the color of the mixture quickly turned to black. After removing the reaction vessel from the cooling device, the mixture was stirred for an additional 1 min. A toluene solution of P^tBu_3 (0.664 M, 0.5 mL, 0.332 mmol) was added to the black solution, followed by stirring for 1 minute. The mixture was then filtered through a syringe filter (Whatman 25 mm, 0.45 μm) to remove insoluble materials. The solution was allowed to stand at room temperature for 3 days, furnishing black rod-shaped (often dendritic-shaped) crystals. The supernatant was removed, and the crystals were washed with hexane (*ca.* 1 $\times$ 5 mL). The crystals were gently dried in the glovebox without evacuation (18.1 mg, 12%). Note that the crystalline sample seems to degrade slowly under vacuum, possibly due to the loss of hydrides as H_2 . The synthesis can be scaled up by 10 times, where $\text{Fe}\{\text{N}(\text{SiMe}_3)_2\}_2$ (5.00 g, 13.3 mmol) was used, resulting in 199 mg of the product in 13% yield. ^1H NMR (800 MHz, toluene-

d_8 , r.t.): δ 1.24 ($w_{1/2}$ = 64 Hz, $PC(CH_3)_3$), 0.07 ($w_{1/2}$ = 54 Hz, $N(SiMe_3)_2$), $^{13}C\{^1H\}$ NMR (800 MHz, toluene- d_8 , r.t.): δ 34.39 ($PC(CH_3)_3$), 32.50 ($PC(CH_3)_3$), 2.59 ($N(SiMe_3)_2$). UV-vis (THF): λ_{max} = 270 (ϵ 1.0×10^4 cm $^{-1}$ M $^{-1}$) nm. Deuterated cluster samples were prepared by using DBpin and/or toluene- d_8 . Elem. Anal. Found: C, 31.20; H, 6.75; N, 1.24%. Calcd for $[Fe_{55}H_{46}(P^tBu_3)_{12}][Fe_6H_8\{N(SiMe_3)_2\}_6]$: C, 31.56; H, 7.15; N, 1.23%. FT-IR (ATR, cm $^{-1}$): ν 2991, 2940, 2863, 1469, 1440, 1389, 1387, 1363, 1238, 1173, 1020, 961, 930, 836, 800, 751, 663, 612, 588, 561, 494, 410.

X-ray Crystallography. Crystal data and refinement parameters of $[Fe_{55}][Fe_6] \bullet toluene$ and $[Fe_{55}H_{46}(P^tBu_3)_{12}][Fe\{N(SiMe_3)_2\}_3]$, $[Fe_{55}][Fe\{N(SiMe_3)_2\}_3]$, are listed in Table S1. On rare occasions, crystals of $[Fe_{55}][Fe\{N(SiMe_3)_2\}_3]$ precipitated in trace amounts (<1% yield) in addition to the crystals of $[Fe_{55}][Fe_6] \bullet toluene$. Therefore, diffraction data for $[Fe_{55}][Fe\{N(SiMe_3)_2\}_3]$ were collected for structural identification. Single crystals were covered with immersion oil (Type-B: code 1248, Cargile Laboratories, Inc.) and mounted on a MiTeGen MicroLoop. Diffraction data were collected under a cold N $_2$ stream (–100/–150 °C for $[Fe_{55}][Fe_6] \bullet toluene/[Fe_{55}][Fe\{N(SiMe_3)_2\}_3]$), on a Rigaku RA-Micro 7 equipped with a PILATUS 200K detector, using graphite-monochromated Mo K α radiation (λ = 0.710690 Å). Eighteen preliminary images were taken at 0.5° increments of ω to evaluate crystal quality and determine pre-refined unit cell parameters, followed by full data collection with 0.5° increments of ω . The raw frame data was processed using CrystalClear program, and the data sets were corrected for absorption using the REQAB program. Calculations were performed with the Olex2 program package and ShelX programs. The structures were solved with Intrinsic Phasing using SHELXT and refined by full-matrix least-square techniques. All non-hydrogen atoms were refined anisotropically. All hydrogen atoms were refined isotropically. The relatively high-quality data for $[Fe_{55}][Fe\{N(SiMe_3)_2\}_3]$ prompted us to collect diffraction data at as low a temperature as possible (at –150 °C) for our attempt to determine the hydride positions. However, we were unable to locate all hydrides, possibly due to the flexibility of their positions. For the analysis of $[Fe_{55}][Fe_6] \bullet toluene$, SIMU, RIGU, DELU and SADI were applied to the disordered SiMe $_3$ unit on $[Fe_6]$. FLAT, SADI, DFIX were applied to some H, P, and C atoms. SIMU, RIGU, DFIX, SADI, and FLAT were applied to the disordered crystal solvent (toluene). Other solvent molecules were SQUEEZED from the lattice using the Olex2 solvent mask command due to unresolvable disorder. For the analysis of $[Fe_{55}][Fe\{N(SiMe_3)_2\}_3]$, SIMU was applied to some C atoms on a P t Bu $_3$. FLAT was applied to some H, P, and C atoms. Olex2 solvent mask command was used to model disordered solvents. The atomic coordinates were deposited with the Cambridge Crystallographic Data Centre, Cambridge CB2 1EK, UK under reference numbers CCDC 2366180 and 2366181.

Fe K-Edge X-ray Absorption Fine Structure (XAFS). The Fe K-edge XAFS measurements were conducted at beamline BL01B1 of the SPring-8 facility, operated by the Japan Synchrotron Radiation Research Institute (JASRI). The incident X-ray beam was monochromatized by a Si(111) double-crystal monochromator. XAFS spectra of $[\text{Fe}_{55}][\text{Fe}_6]$ and reference samples were measured in transmission mode at room temperature using an ionization chamber as a detector. The X-ray energies were calibrated using Fe foil. The obtained extended X-ray absorption fine structure (EXAFS) spectra of $[\text{Fe}_{55}][\text{Fe}_6]$ were analyzed with the xTunes program⁵⁰. The k^3 -weighted EXAFS spectra in the k range 3.0–12.0 Å⁻¹ for the Fe-K edge were Fourier transformed into r space for structural analysis. In the curve fitting analysis, the phase shifts and backscattering amplitude functions for Fe-Fe, Fe-Cl, Fe-P, Fe-Si, and Fe-N were calculated using the FEFF8.5L program. The results of the curve fitting analysis are summarized in Table S2.

Magnetic Measurements. The microcrystalline sample of $[\text{Fe}_{55}][\text{Fe}_6]$ was sealed in a quartz tube with quartz wool, and the tube was flame-sealed before measurement. Susceptibility data were collected using a Quantum Design MPMS-5XL SQUID magnetometer. Magnetic data were corrected for the diamagnetism of the sample using Pascal's constants⁵¹. The diamagnetism of the sample holder and quartz wool was corrected using a blank sample tube without a compound. Variable-temperature dc magnetization data for $[\text{Fe}_{55}][\text{Fe}_6]$ were collected under applied dc magnetic fields at 10 Oe over the temperature range of 2 to 300 K. AC magnetic susceptibility data were collected using a 3 Oe (1 Hz) or 2 Oe (for 10, 100 and 1000 Hz) oscillating ac field under static magnetic fields of 0 Oe. Magnetic hysteresis data were collected at 1.8 K with an average sweep rate of 3 Oe/s.

Zero-Field ⁵⁷Fe-Mössbauer Spectroscopy. ⁵⁷Fe Mössbauer spectra were recorded on a WissEl Mössbauer spectrometer (MRG-500) at 77 K in constant acceleration mode. A ⁵⁷Co/Rh source was used for γ-radiation. The WinNormos for Igor Pro software was used for the quantitative evaluation of the spectral parameters through least-squares fitting to Lorentzian peaks. The minimum experimental line width was determined to be 0.21 mm/s (full width at half maximum, FWHM). The sample temperature was controlled by an MBBC-HE0106 Mössbauer He/N₂ cryostat with an accuracy of ±0.3 K. Least-squares fitting of the Lorentzian signals was carried out with the "Mfit" software developed by Dr. Eckhard Bill (MPI CEC, Mülheim/Ruhr)^{52,53}. The isomer shifts are reported relative to α-iron reference at 300 K.

Electron Spin Resonance (ESR) Spectroscopy. For ESR measurements, a powder or a toluene solution (ca. 0.4 mM) of $[\text{Fe}_{55}][\text{Fe}_6]$ was loaded into quartz tubes (o.d. 3.0 mm) in an Ar-filled glovebox and sealed with a J-Young valve. Low-temperature ESR spectra were recorded at the Institute for Molecular Science. Continuous wave (CW) X-band (9.4 GHz) spectra were collected using a Bruker E580 spectrometer

equipped with a super high Q resonator (ER 4122SHQ). Cryogenic temperatures were controlled and maintained using an ESR900 liquid helium cryostat in conjunction with a temperature controller and a gas flow controller. The room-temperature spectrum was collected using a Bruker E500 spectrometer.

Computational Methods. The structures of the $[\text{Fe}_{55}]$ cation and the $[\text{Fe}_6]$ anion were optimized using the GFN1-xTB method⁵⁴, as implemented in the density functional-based tight-binding (DFTB) module of AMS2024 (version 2024.102)⁵⁵. The 46 hydrides of $[\text{Fe}_{55}]$ and the 8 hydrides of $[\text{Fe}_6]$ were randomly placed and the structures were then optimized, while the positions of Fe, P, and N atoms were frozen. The lowest energy structure of $[\text{Fe}_6]$ was fully optimized without any constraints using density functional theory (DFT), as implemented in the ORCA program (Version 4.2.1)⁵⁶. The BP86 functional^{57,58}, incorporating the D3 version of Grimme's dispersion with Becke-Johnson damping⁵⁹, was used. The ZORA-def2-TZVP basis sets and SARC/J auxiliary basis sets were employed for all atoms⁶⁰⁻⁶².

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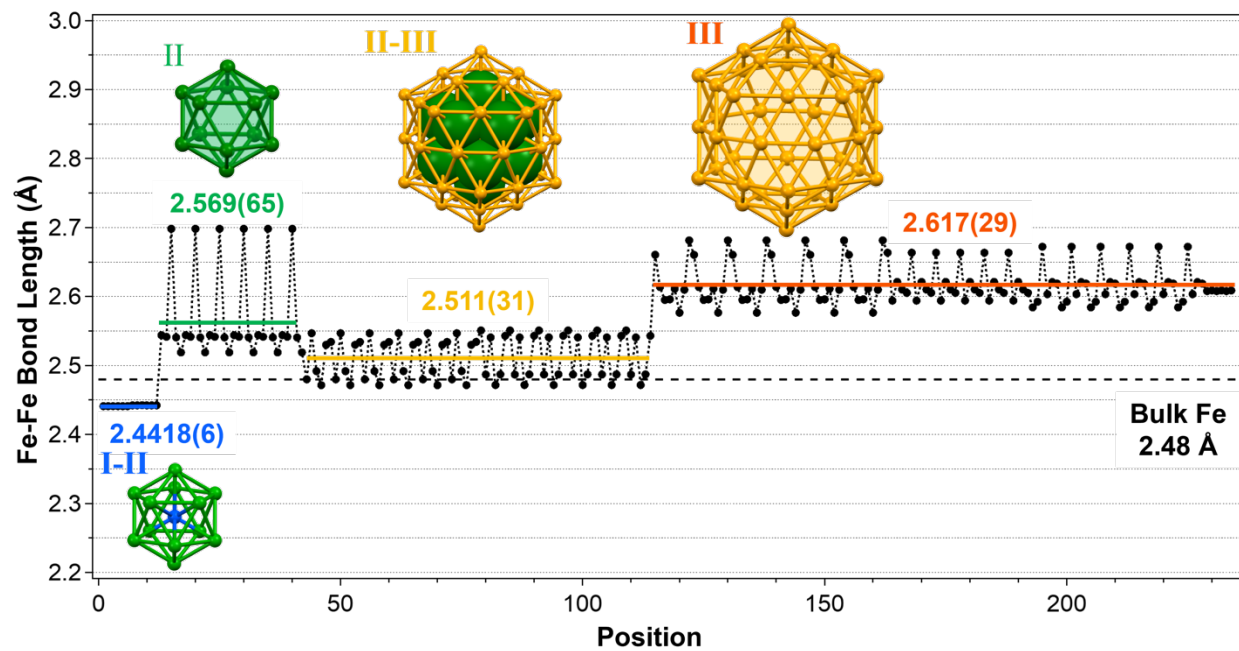


Figure S1. Shell-by-shell analysis of the Fe-Fe distances in the Fe_{55} core of $[\text{Fe}_{55}][\text{Fe}_6]$.

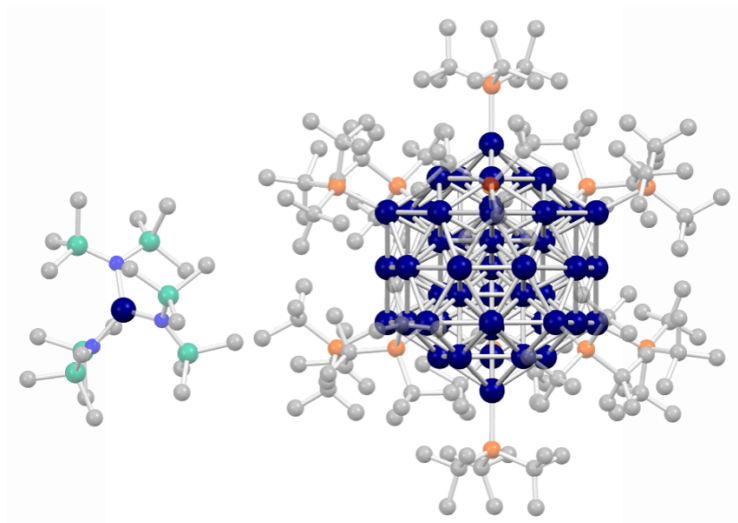


Figure S2. Crystal structure of $[\text{Fe}_{55}][\text{Fe}\{\text{N}(\text{SiMe}_3)_2\}_3]$ depicted in a ball-and-stick model. Hydrogen atoms are omitted for clarity. On rare occasions, crystals of this compound precipitated in trace amounts (<1% yield) in addition to the crystals of $[\text{Fe}_{55}][\text{Fe}_6]\bullet\text{toluene}$. Color legend: dark blue, Fe; orange, P; teal, Si; light blue, N; gray, C.

The cationic charge (1+) of the $[\text{Fe}_{55}]$ moiety is further supported by this crystal structure, as the $[\text{Fe}\{\text{N}(\text{SiMe}_3)_2\}_3]$ unit of $[\text{Fe}_{55}][\text{Fe}\{\text{N}(\text{SiMe}_3)_2\}_3]$ is in an anionic form in the Fe^{II} state. The Fe-N distance of the $[\text{Fe}\{\text{N}(\text{SiMe}_3)_2\}_3]$ unit (1.968(12) Å) is comparable to those reported for the anionic Fe^{II} complex $[\text{Fe}\{\text{N}(\text{SiMe}_3)_2\}_3]^-$ (Fe-N: 1.979(5)-1.999(4) Å) without cation-N interaction⁶³⁻⁶⁷, and is longer than those in the neutral $\text{Fe}\{\text{N}(\text{SiMe}_3)_2\}_3$ complex in the Fe^{III} state (Fe-N: 1.8917(12)-1.917(4) Å)⁶⁸⁻⁷⁰.

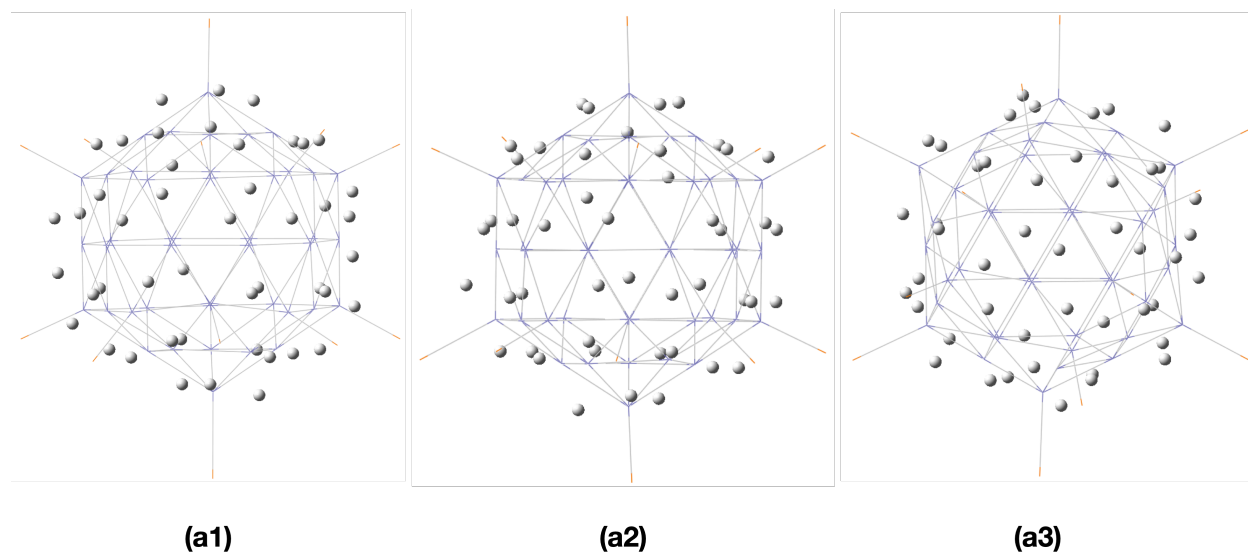


Figure S3. The three lowest energy structures of **[Fe₅₅]** (**a1-a3**) with 46 hydrides. Their relative energies are 0 kcal/mol (**a1**), 3.4 kcal/mol (**a2**), and 3.9 kcal/mol (**a3**) (Table S3). For simplicity, the ^tBu groups and the inner Fe atoms of the Fe₅₅ core have been omitted. The Fe and P atoms are drawn in a wireframe model. Optimized coordinates are given as separate Supporting Information files in a .xyz format.

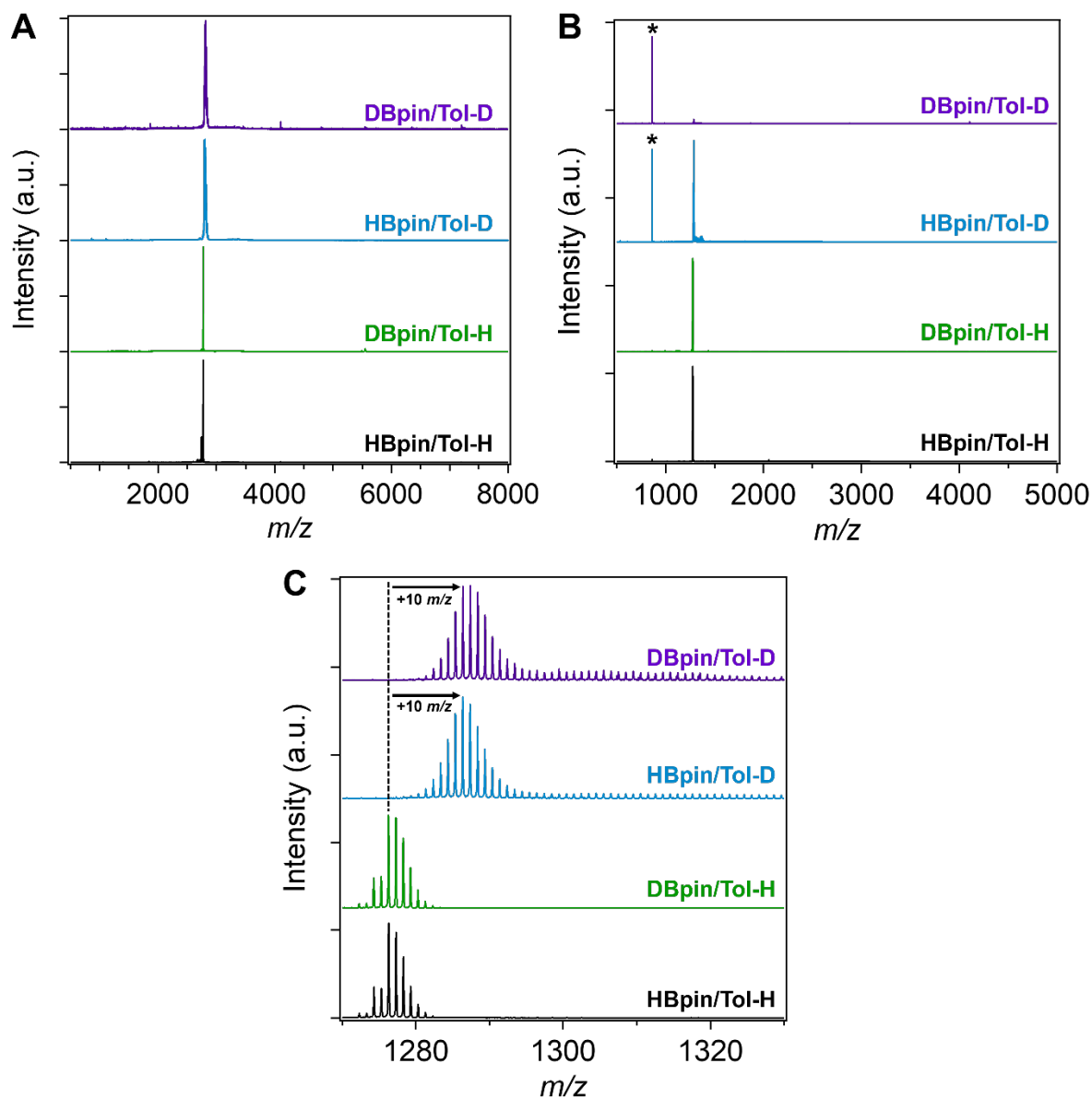


Figure S4. (A) Positive-mode and (B,C) negative-mode ESI mass spectra of $[\text{Fe}_{55}][\text{Fe}_6]$ synthesized from HBpin/toluene, DBpin/toluene, HBpin/toluene- d_8 , or DBpin/toluene- d_8 . An asterisk indicates the presence of the $[\text{BAr}^{\text{F}}_4]^-$ anion ($\text{Ar}^{\text{F}} = 3,5\text{-(CF}_3)_2\text{C}_6\text{H}_3$) due to instrument contamination.

Quantification of H₂ evolved from [Fe₅₅][Fe₆] via thermolysis

Setup and calibration curve. All procedures were carried out using the same setup (Figure S5 and S7). The GC conditions are summarized in Table S1, and the calibration curve is shown in Figure S6.

For the preparation of the calibration curve, a 20 mL vial was charged with Ar and sealed with a rubber septum and an aluminum cap in a glove box. Ar (100, 200, 300, 400, 500 μ L) was taken from the sealed vial and known volumes of H₂ (100, 200, 300, 400, 500 μ L, respectively) were injected using a gas-tight syringe, and the vial was heated at 180 °C in an oil bath for 4 hours. A 0.5 mL sample of the gas phase was then withdrawn with a gas-tight syringe and analyzed by GC. The retention time for H₂ was 0.96 min.

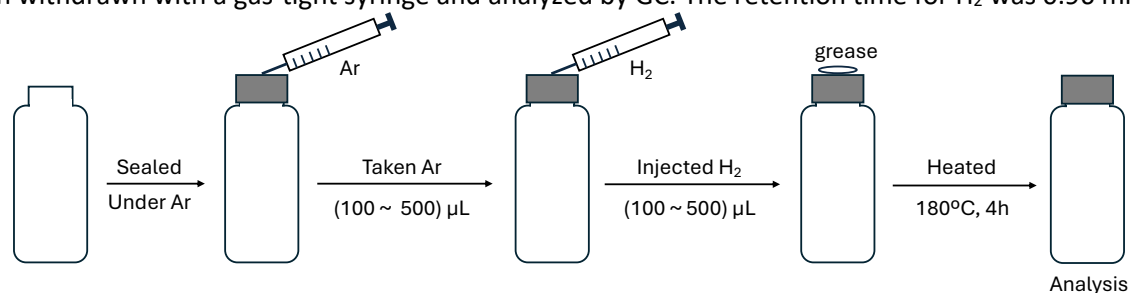


Figure S5. Setup for the calibration curve.

Table S1. Conditions for the GC analysis.

Instrument	Shimadzu GC-2014s
Column	Molecular Sieve 13X SUS 2m x 3.0mm I.D.
Col. Temp.	40 °C
Carrier Gas	Ar, 25 mL/min
Detector	TCD, 70 mA
Sample Volume	0.5 mL

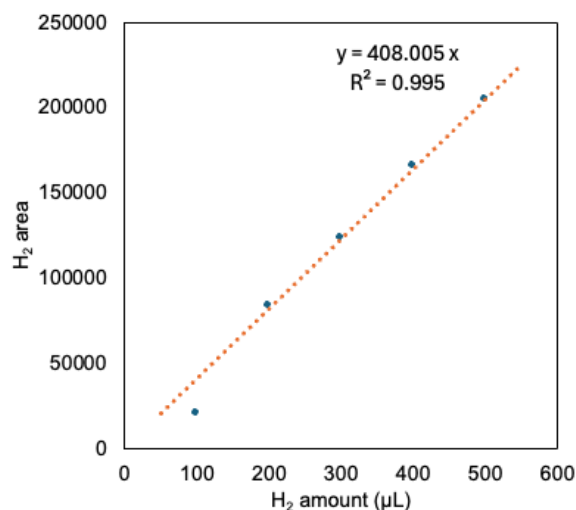


Figure S6. Calibration curve for H₂ quantification.

Quantification of H₂. For the H₂ quantification experiment, the same vial used for the preparation of the calibration curve was charged with **[Fe₅₅][Fe₆]** (4.37 mg, 0.629 μmol; 4.21 mg, 0.606 μmol; 4.40 mg, 0.634 mmol for three runs) under an Ar atmosphere and sealed with a rubber septum and an aluminum cap. The vial was heated at 180 °C in an oil bath for 4 hours. A 0.5 mL sample of the gas phase was withdrawn using a gas-tight syringe and analyzed by GC. The yields of H₂ per **[Fe₅₅][Fe₆]** were 27.1, 27.7, and 28.4 equivalents for three runs, with an average of 27.7(2) equivalents. These results are in agreement with the presence of 46 and 8 hydrides (27 equivalents of H₂ in total) in **[Fe₅₅]** and **[Fe₆]** moieties, respectively.

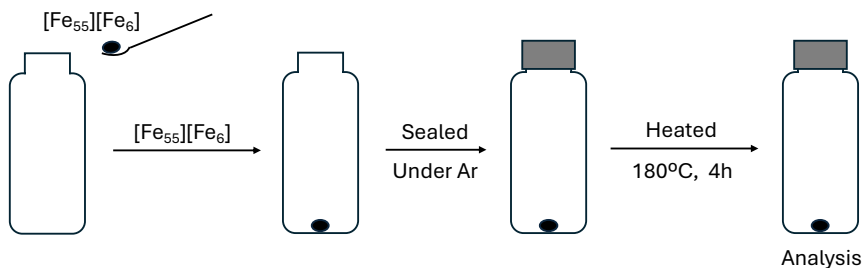


Figure S7. Set up for H₂ quantification.

As listed in Table S2, we additionally carried out analogous reactions for 1 hour and 5 hours, respectively. Based on these results, we concluded that heating for 4 hours at 180 °C is sufficient for eliminating labile hydrogen atoms.

Table S2. Reaction time vs evolved H₂.

time / hour	average H ₂ yield per [Fe₅₅][Fe₆] / equivalent
1	23.2(5)
4	27.7(2)
5	28.3(22)

Reactions of $[\text{Fe}_{55}][\text{Fe}_6]$ with phosphines (PMe_3 or PEt_3)

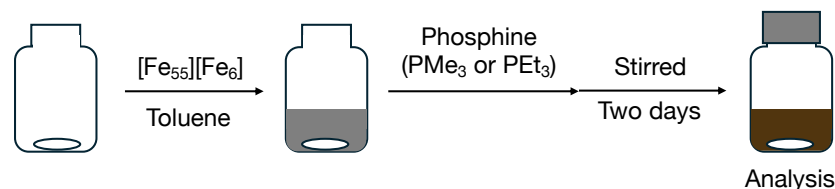


Figure S8. A schematic overview of the experiments. The reactions were carried out in an argon-filled glove box.

The reaction with PMe_3 . A vial was charged with $[\text{Fe}_{55}][\text{Fe}_6]$ (5.0 mg, 0.72 μmol) and toluene (2 mL) to yield a black solution with a small amount of undissolved material. A toluene solution of PMe_3 (7.2 μL , 1 M, 10 equiv.) was then added to the black suspension, which was stirred at room temperature for two days. After this period, the solution changed to a brownish-black color, and the formation of a black precipitate was observed. Subsequently, 1 mL of the supernatant was carefully removed and evaporated to dryness. The residue was dissolved in THF (0.5 mL), and the resulting solution was analyzed by the ESI-MS (Figure S9). No signals were observed in either the positive or negative mode. This result suggests the decomposition of $[\text{Fe}_{55}][\text{Fe}_6]$ or the formation of species undetectable by mass spectrometry.

The reaction with PEt_3 . A vial was charged with $[\text{Fe}_{55}][\text{Fe}_6]$ (5.0 mg, 0.72 μmol) and toluene (2 mL) to yield a black solution with a small amount of undissolved material. A toluene solution of PEt_3 (4.3 mg, 20 wt%, 10 equiv.) was then added to the black suspension, which was stirred at room temperature for two days. After this period, the solution turned brownish-black, and the formation of a black precipitate was observed. Subsequently, 1 mL of the supernatant was carefully removed and evaporated to dryness. The residue was dissolved in THF (0.5 mL), and the resulting solution was analyzed by the ESI-MS.

In the ESI-MS, two distinct cationic signals were observed (Figure S10 top-left, marked with *). The signal centered at $m/z = 2550.2$ may be assignable as $[\text{Fe}_{55}\text{H}_{47}(\text{P}^t\text{Bu}_3)_7(\text{PEt}_3)_4(\text{C}_7\text{H}_8)]^{2+}$ (calc. $m/z = 2550.3$, C_7H_8 = toluene), while another prominent signal at $m/z = 2562.8$ corresponds to $[\text{Fe}_{55}\text{H}_{46}(\text{P}^t\text{Bu}_3)_7(\text{PEt}_3)_5]^{2+}$ (calc. $m/z = 2562.8$) (Figure S10, bottom). These results suggest that the P^tBu_3 ligands in $[\text{Fe}_{55}][\text{Fe}_6]$ can dissociate for ligand exchange with PEt_3 . However, partial decomposition of $[\text{Fe}_{55}][\text{Fe}_6]$ also occurs, leading to the formation of an insoluble material.

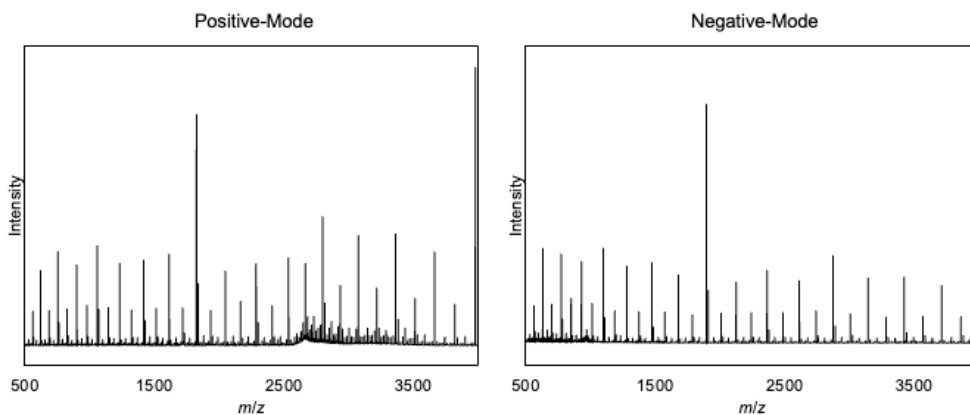


Figure S9. ESI-MS spectra of the solution from the reaction of $[\text{Fe}_{55}][\text{Fe}_6]$ with excess PMe_3 .

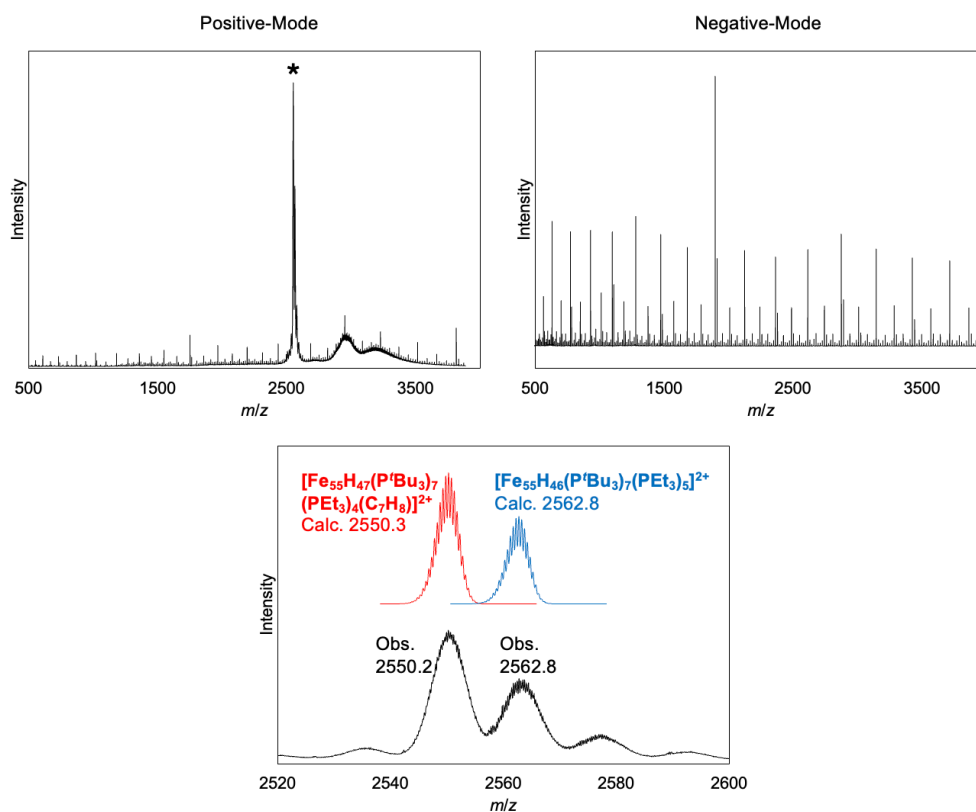


Figure S10. (Top) ESI-MS spectra of the solution from the reaction of $[\text{Fe}_{55}][\text{Fe}_6]$ with excess PET_3 . (Bottom) Expanded spectrum of the peaks marked by *.

Reaction of $[\text{Fe}_{55}][\text{Fe}_6]$ with D_2 (1 atm) leading to decomposition.

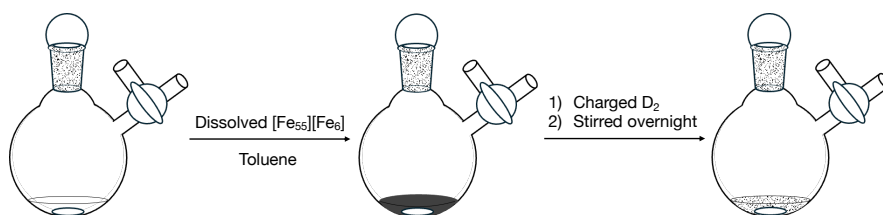


Figure S11. A schematic overview of the experiment.

Protocol and observation. In an argon-filled glove box, a 100 mL round-bottom flask was charged with $[\text{Fe}_{55}][\text{Fe}_6]$ (10.0 mg, 1.44 μmol) and toluene (5 mL) to yield a black solution with a small amount of undissolved material. The flask was taken out from the glove box, and the headspace of the flask was purged with D_2 . The resultant brownish-black solution was stirred at room temperature overnight. After this period, the mixture turned into a colorless solution with a black precipitate. Subsequently, 1 mL of the supernatant was carefully taken out and evaporated. The residue was dissolved in THF (0.5 mL), and the resulting solution was analyzed by the ESI-MS. No cluster signals were observed (Figure S12). Dissociation of P^tBu_3 would account for this phenomenon.

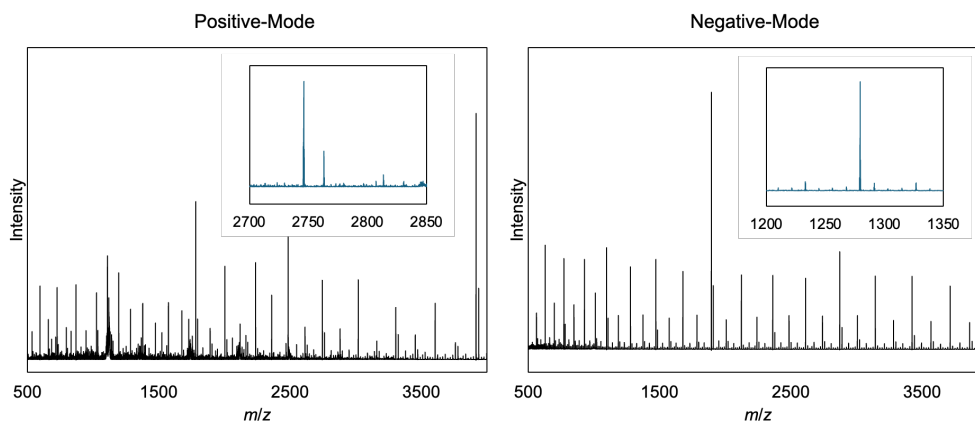


Figure S12. ESI-MS spectra of the solution from the reaction of $[\text{Fe}_{55}][\text{Fe}_6]$ with D_2 (1 atm). Insets are the expanded spectra where the $[\text{Fe}_{55}]$ and $[\text{Fe}_6]$ signals appear.

Analysis of the reaction solution after crystallization of $[\text{Fe}_{55}][\text{Fe}_6]$.

The entitled compound $[\text{Fe}_{55}][\text{Fe}_6]$ was isolated after crystallization, followed by the removal of the supernatant. In order to find the remaining $[\text{Fe}_{55}][\text{Fe}_6]$ and some other products in the discarded mother liquor, we analyzed the solution by the ESI-MS measurement.

Protocol and observation. $\text{Fe}\{\text{SiMe}_3\}_2$ (0.500 g, 1.33 mmol) was dissolved in toluene (1.5 mL) at room temperature, and the solution was stirred for 30 min at -20°C . HBpin (0.365 mL, 2.52 mmol) was then added dropwise to the light green solution, causing a quick color change to black. After stirring for 1 min without cooling, a toluene solution of P^tBu_3 (0.7 mL, 0.335 mmol) was added, and the mixture was stirred for 5 min. The resulting mixture was filtered, and the solution was allowed to stand for two days, yielding black crystals of $[\text{Fe}_{55}][\text{Fe}_6]$. A few drops of the supernatant were carefully taken out and diluted by THF (0.5 mL). The resulting solution was analyzed by the ESI-MS (Figure S13), where no prominent cluster signals were observed. We were unable to assign the broad cationic signals between $m/z = 500$ and 5000.

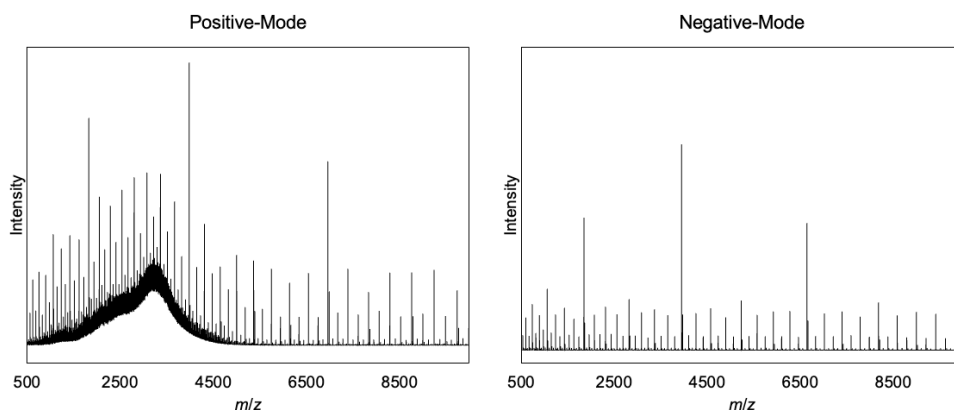


Figure S13. ESI-MS spectra of the supernatant after crystallization of $[\text{Fe}_{55}][\text{Fe}_6]$.

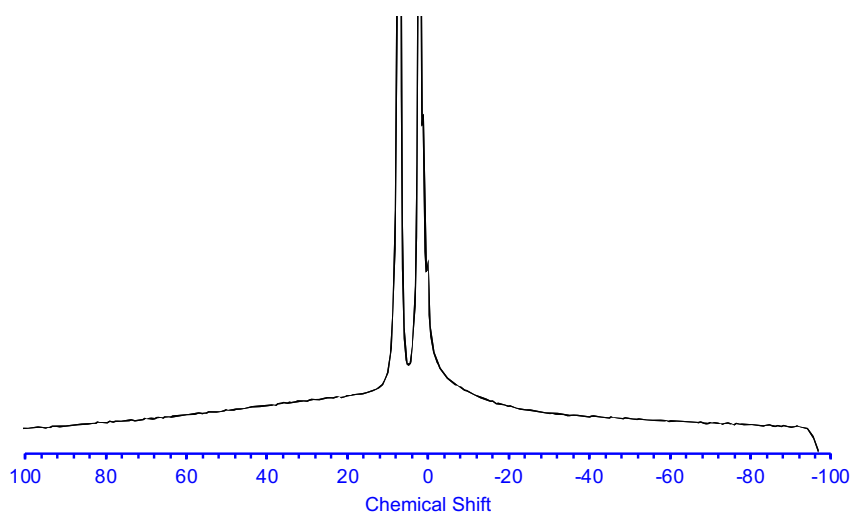


Figure S14. The ^1H NMR spectrum of $[\text{Fe}_{55}][\text{Fe}_6]$ in toluene- d_8 from +100 to -100 ppm. We were unable to find hydride signals.

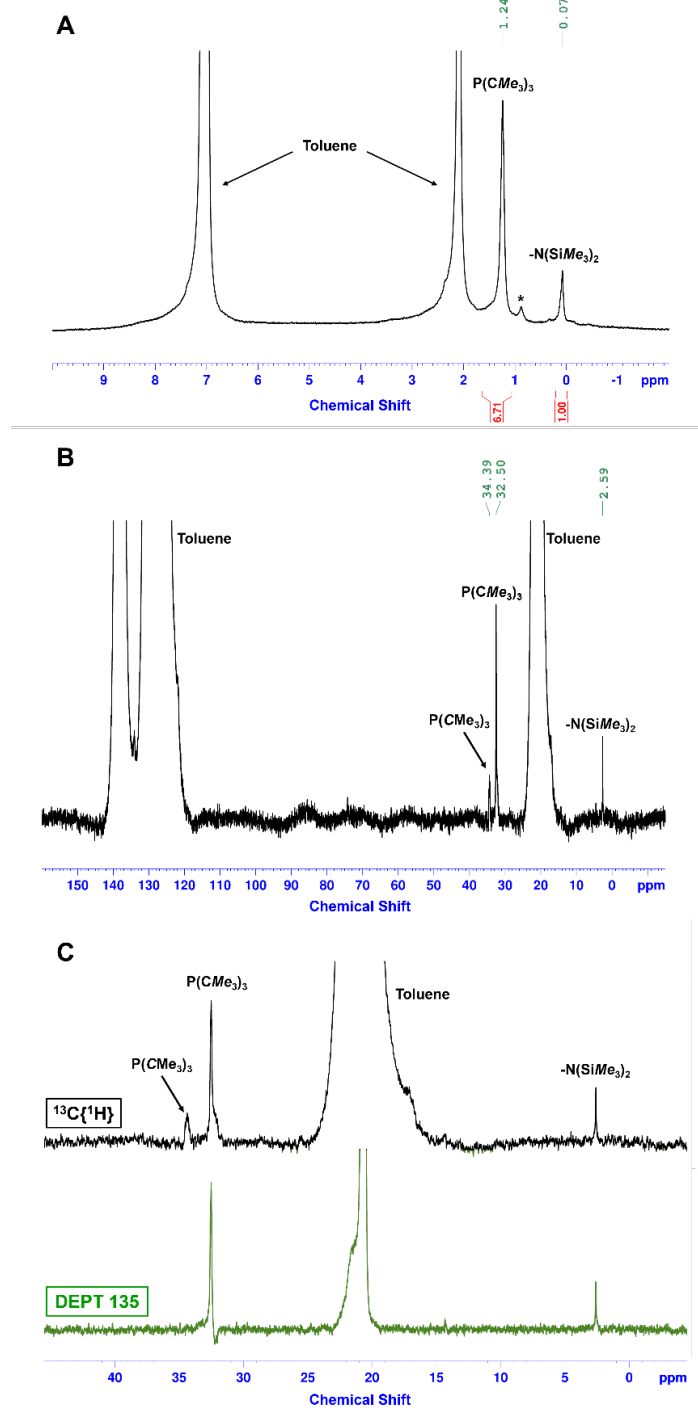


Figure S15. (A) ^1H and (B) $^{13}\text{C}\{^1\text{H}\}$ NMR spectra of $[\text{Fe}_{55}][\text{Fe}_6]$ in toluene- d_8 . (C) DEPT-135 NMR and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra. An asterisk indicates an impurity signal.

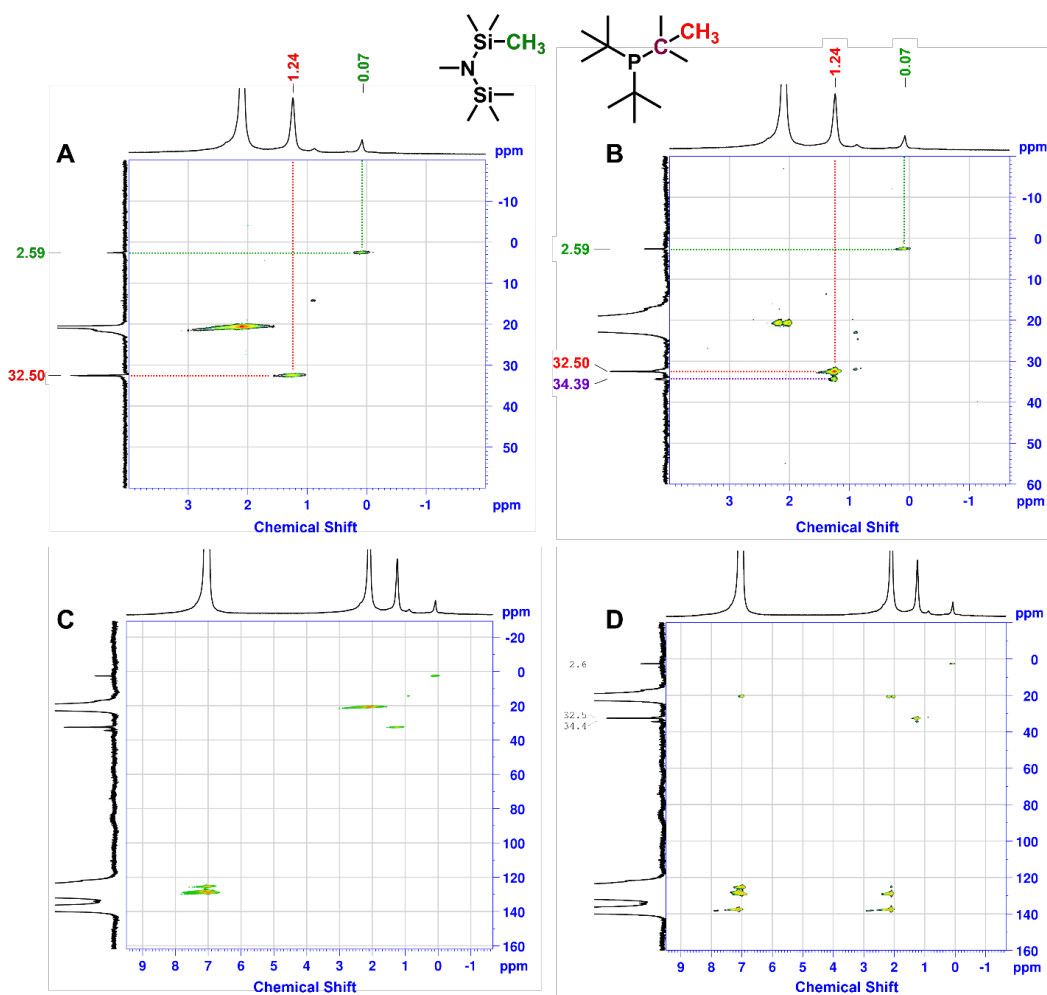


Figure S16. (A, C) ^1H - ^{13}C (DEPT 135) HSQC and (B, D) ^1H - ^{13}C HMBC NMR spectra of $[\text{Fe}_{55}][\text{Fe}_6]$ in $\text{toluene-}d_8$.

The ^1H NMR spectrum of $[\text{Fe}_{55}][\text{Fe}_6]$ in $\text{toluene-}d_8$ exhibits two broad paramagnetic signals at 1.24 and 0.07 ppm, which were assigned to P^tBu_3 and $\text{N}(\text{SiMe}_3)_2$, respectively (Figure S15). The $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum shows three signals at 34.4, 32.5, and 2.6 ppm, with the signals at 34.4 and 32.5 ppm assigned to P^tBu_3 . Distortionless enhancement by polarization transfer (DEPT)-135 was performed to confirm these assignments. Notably, the DEPT-135 spectra reveal no quaternary carbons, with methylene carbons phased negative and methine and methyl carbons phased positive. DEPT-135 ^{13}C NMR of $[\text{Fe}_{55}][\text{Fe}_6]$ shows no signals at 34.4 ppm, indicating that this signal originates from a quaternary carbon in P^tBu_3 . The signals at 32.5 and 2.6 ppm exhibit positive phases, assigned to methyl carbons in P^tBu_3 and $\text{N}(\text{SiMe}_3)_2$, respectively. The 2D ^1H - ^{13}C correlation HSQC/HMBC spectrum supports these assignments (Figure S16).

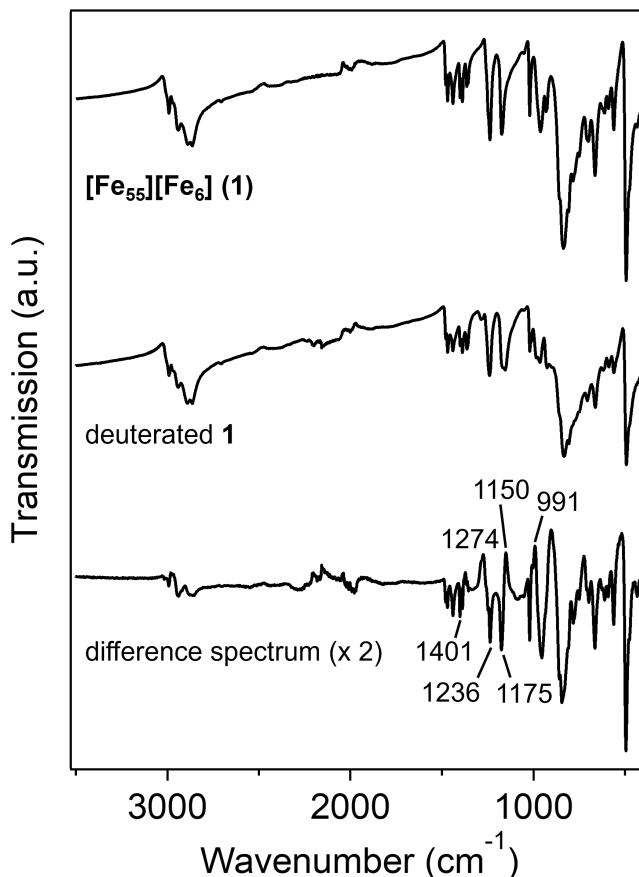


Figure S17. Infrared spectra of $[\text{Fe}_{55}][\text{Fe}_6]$, its deuterated form (obtained using DBpin in toluene- d_8), as well as the corresponding difference spectrum.

The IR spectrum of $[\text{Fe}_{55}][\text{Fe}_6]$ was compared with its deuterated form, which was synthesized using DBpin in toluene- d_8 . Although the difference spectrum exhibits some bands phased positively and negatively, we were unable to assign bands for bridging μ_2 - or μ_3 -hydrides. Typically, metal complexes with terminal hydrides show a M–H stretching and bending modes in the ranges of approximately 2300–1600 and 900–600 cm^{-1} , while those with bridging hydrides (*e.g.*, μ_2 - or μ_3 -hydrides) display broader bands in the lower energy region⁷⁰. A commonly used strategy to identify the stretching bands for the bridging hydrides is the synthesis of the corresponding deuterides and the observation of the isotope shift [*i.e.*, $\nu(\text{Fe–H})/\nu(\text{Fe–D}) \sim 1.4$]⁷¹. For instance, a $\nu(\text{Fe–H})$ band of μ/μ_3 -hydrides in $\text{Fe}_4(\mu\text{-H})_4(\mu_3\text{-H})_2\{\text{N}(\text{SiMe}_3)_2\}_2(\text{PMe}_2\text{Ph})_4$ was observed at 1524 cm^{-1} , which shifted to 1108 cm^{-1} upon deuteration³⁴.

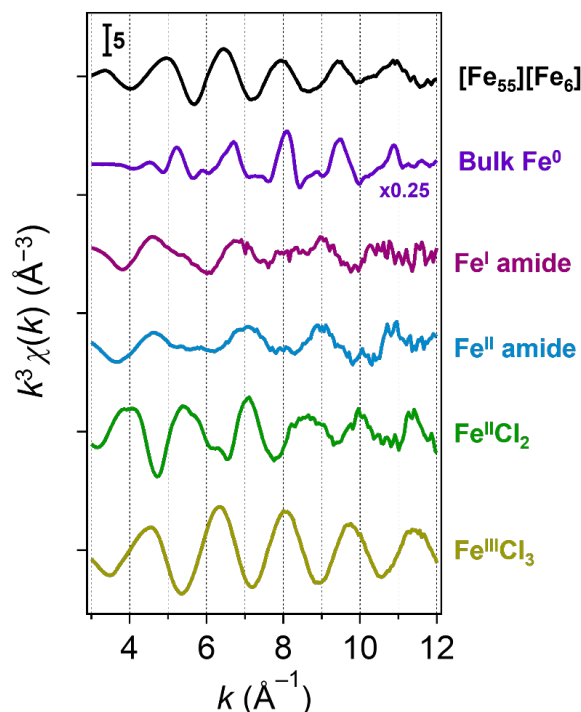


Figure S18. Fe K-edge EXAFS oscillations of $[\text{Fe}_{55}][\text{Fe}_6]$ and reference samples.

Curve fitting analysis of FeCl_2 and FeCl_3 yielded coordination numbers of 4.7(3) and 4.0(2) for Fe-Cl bonds, respectively. These values are smaller than expected considering the six-coordinate Fe in their crystal structures. This discrepancy may be attributed to partial hydration of the commercial samples and the presence of Fe-O bonds as a minor component.

The EXAFS of $[\text{K}(18\text{-crown-6})][\text{Fe}\{\text{N}(\text{SiMe}_3)_2\}_2]$ can be fitted with a Fe-N bond as a first shell component, providing a coordination number of 2.2(3) and an r value of 1.94(5) Å. These values are consistent with the coordination environment in the crystal structure, where two N atoms are directly coordinated to Fe with a bond distance of 1.9213(6) Å. The second shell component reveals the presence of the band for the Fe-Si distance with a coordination number of 2.8(3) and an r value of 3.06(6) Å. In the crystal structure, $\text{N}(\text{SiMe}_3)_2$ ligands have two Si coordinated to N, thereby there should be four Si in the second coordination shell of Fe. The Fe-Si distances in the crystal structure are 3.037 and 3.057 Å, which closely match the r value from the curve fitting analysis.

The EXAFS of $\text{Fe}\{\text{N}(\text{SiMe}_3)_2\}_2$ was fitted in the r range of 1.2-2.3 Å. Decent fitting results were obtained for two different Fe-N bonds with coordination numbers of 1.8(2) and 1.7(3), respectively. In the crystal structure, $\text{Fe}\{\text{N}(\text{SiMe}_3)_2\}_2$ dimerizes, forming three-coordinate Fe sites with one terminal and two bridging $\mu_2\text{-N}(\text{SiMe}_3)_2$ units (33). The sum of Fe-N coordination numbers is almost three in total, which is consistent with the coordination environment in the crystal structure. The Fe-N bond distances in the crystal structure are 1.927(3) and 1.923(3) Å for terminal $\text{N}(\text{SiMe}_3)_2$ and 2.086(2) and 2.083(2) Å for bridging $\mu_2\text{-N}(\text{SiMe}_3)_2$, respectively. This observation is also consistent with the r values of 1.92(4) and 2.24(8) Å obtained for each Fe-N bond from the curve fitting analysis of EXAFS.

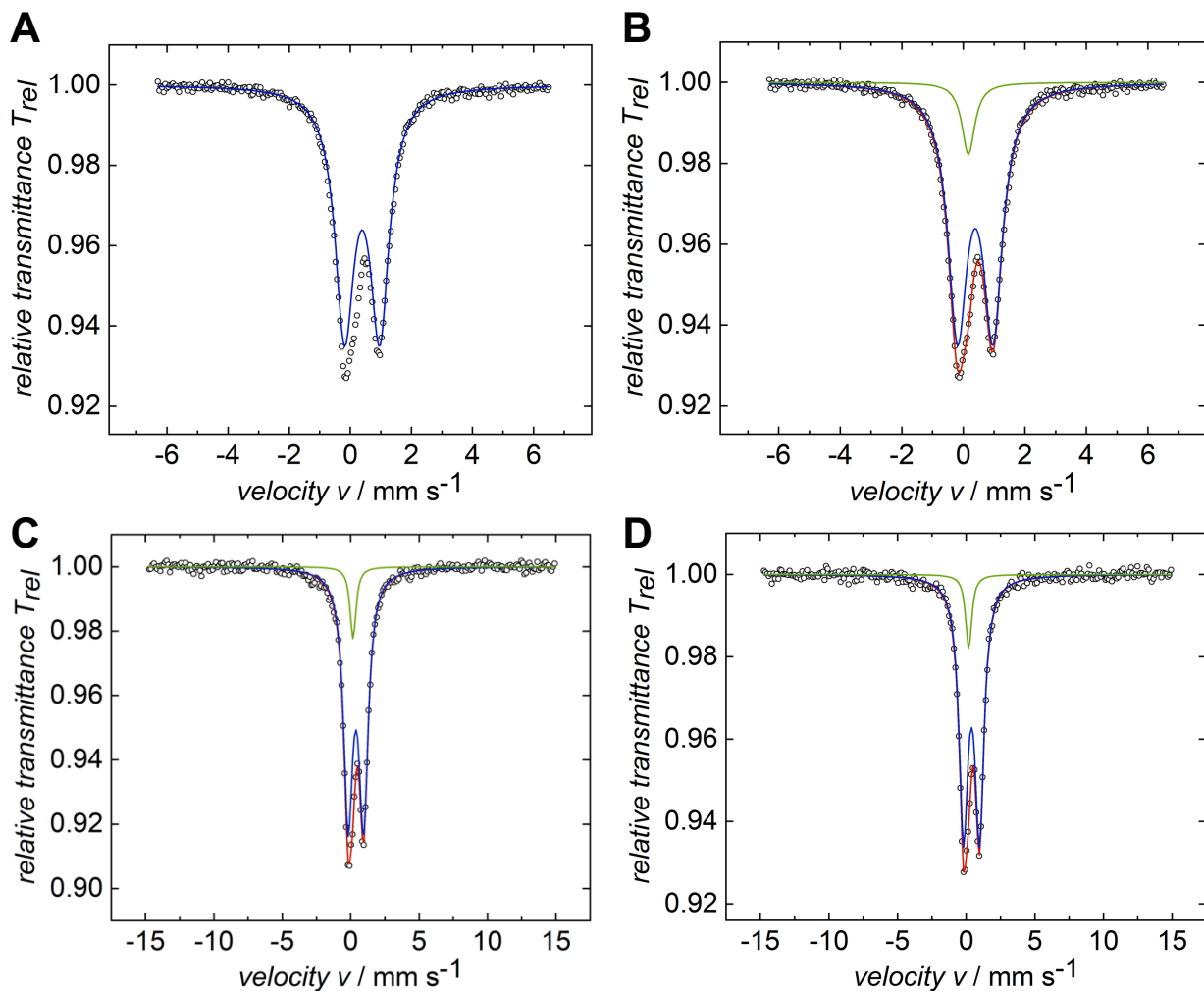


Figure S19. The zero-field ^{57}Fe Mössbauer spectra of $[\text{Fe}_{55}][\text{Fe}_6]$ were recorded at 77 K for three independently synthesized batches of microcrystalline samples. The spectra were obtained using two different velocity scales: ± 6 mm/s (**A**, **B**) and ± 15 mm/s (**C**, **D**). The collected data are represented by black circles. The results demonstrate the reproducibility of sample synthesis and data acquisition. Simulation (**A**) with a doublet for a single Fe species shows a misfit for the low-energy absorption line, indicating that a second Fe species needs to be invoked to account for the residual intensity. Thus, the best fit to the experimental data (in red) is obtained with Fe species 1, $[\text{Fe}_{55}]$ (91%), and a second Fe species 2, $[\text{Fe}_6]$ (9%). The red traces in **B**, **C**, and **D** represent the best combined fits using the parameters listed below. The relative content of 91-to-9% fits the iron stoichiometry in $[\text{Fe}_{55}][\text{Fe}_6]$:

(B): Species 1 ($[\text{Fe}_{55}]$, blue trace): $\delta = 0.39 \text{ mm s}^{-1}$, $\Delta E_Q = 1.15 \text{ mm s}^{-1}$, $\Gamma_{\text{FWHM}} = 0.76 \text{ mm s}^{-1}$, relative content = 91%. Species 2 ($[\text{Fe}_6]$, green trace): $\delta = 0.17 \text{ mm s}^{-1}$, $\Delta E_Q = 0.00 \text{ mm s}^{-1}$, $\Gamma_{\text{FWHM}} = 0.50 \text{ mm s}^{-1}$, relative content = 9%.

(C): Species 1 ($[\text{Fe}_{55}]$, blue trace): $\delta = 0.38 \text{ mm s}^{-1}$, $\Delta E_Q = 1.13 \text{ mm s}^{-1}$, $\Gamma_{\text{FWHM}} = 0.80 \text{ mm s}^{-1}$, relative content = 91%. Species 2 ($[\text{Fe}_6]$, green trace): $\delta = 0.17 \text{ mm s}^{-1}$, $\Delta E_Q = 0.00 \text{ mm s}^{-1}$, $\Gamma_{\text{FWHM}} = 0.52 \text{ mm s}^{-1}$, relative content = 9%.

(D): Species 1 ($[\text{Fe}_{55}]$, blue trace): $\delta = 0.39 \text{ mm s}^{-1}$, $\Delta E_Q = 1.16 \text{ mm s}^{-1}$, $\Gamma_{\text{FWHM}} = 0.78 \text{ mm s}^{-1}$, relative content = 91%. Species 2 ($[\text{Fe}_6]$, green trace): $\delta = 0.18 \text{ mm s}^{-1}$, $\Delta E_Q = 0.00 \text{ mm s}^{-1}$, $\Gamma_{\text{FWHM}} = 0.52 \text{ mm s}^{-1}$, relative content = 9%.

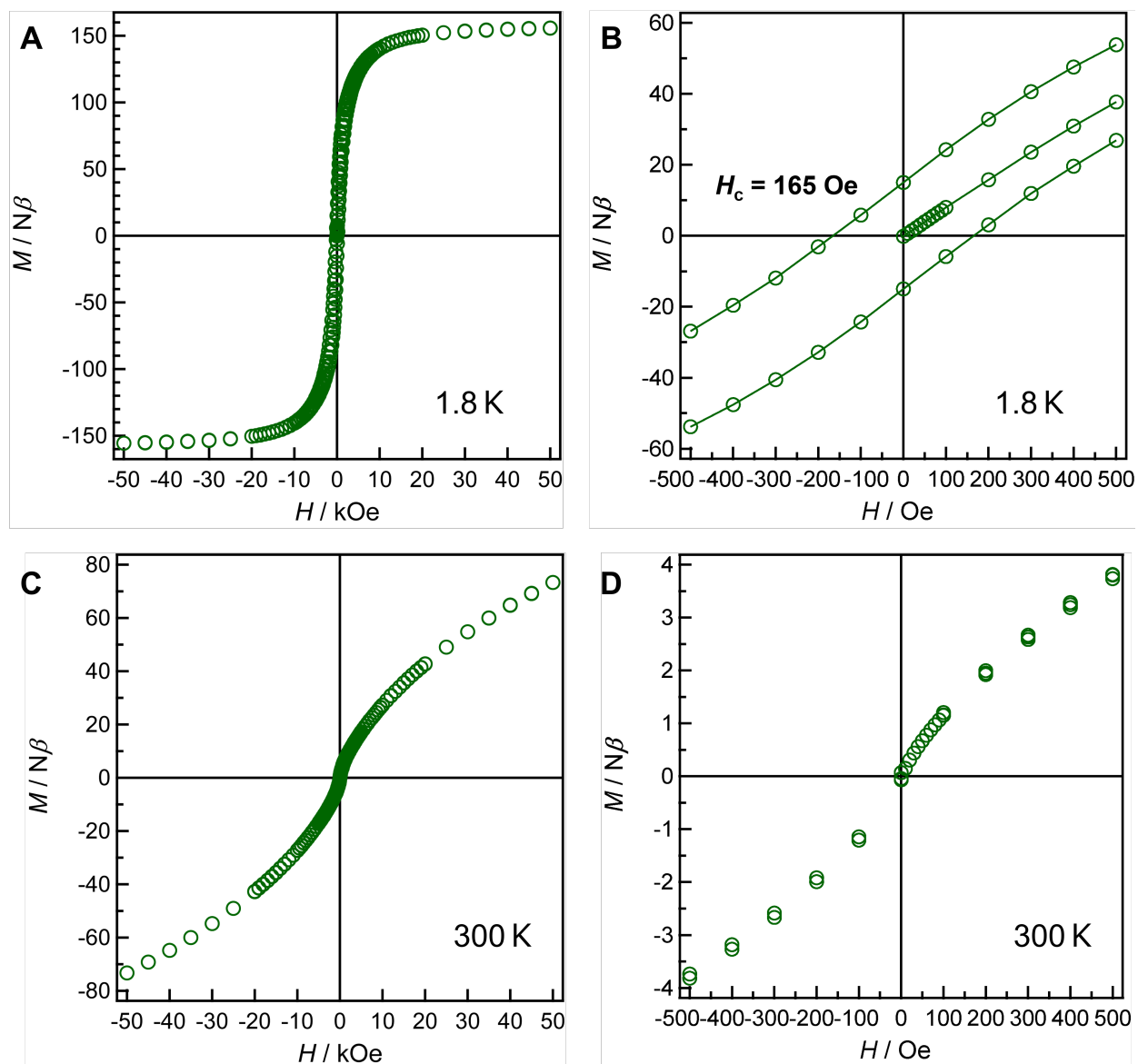


Figure S20. Magnetization versus field plots of $[\text{Fe}_{55}][\text{Fe}_6]$ at (A, B) 1.8 K and (C, D) 300 K.

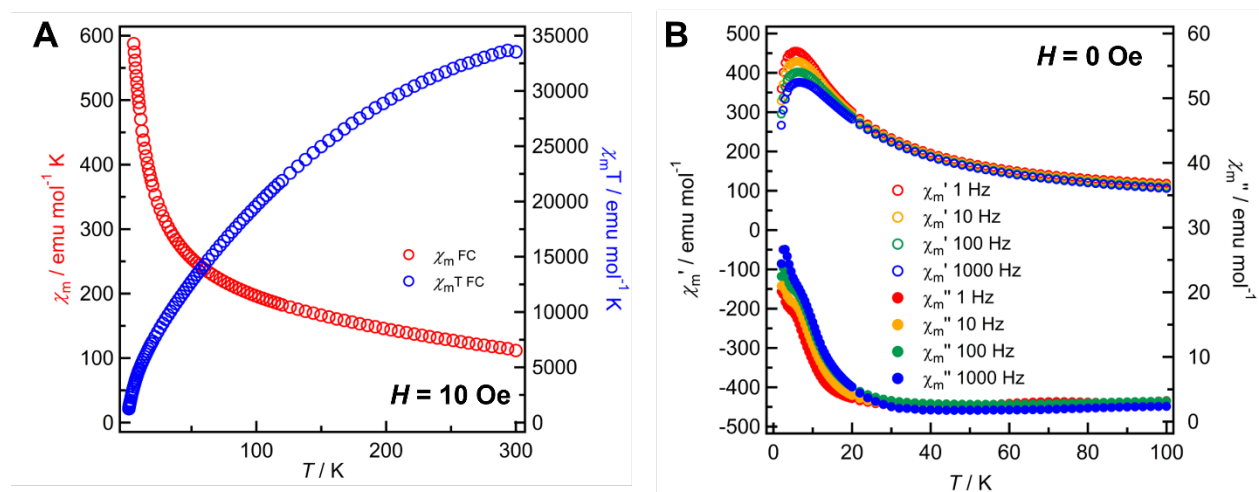


Figure S21. Variable-temperature (A) dc and (B) ac magnetic susceptibility of $[\text{Fe}_{55}][\text{Fe}_6]$.

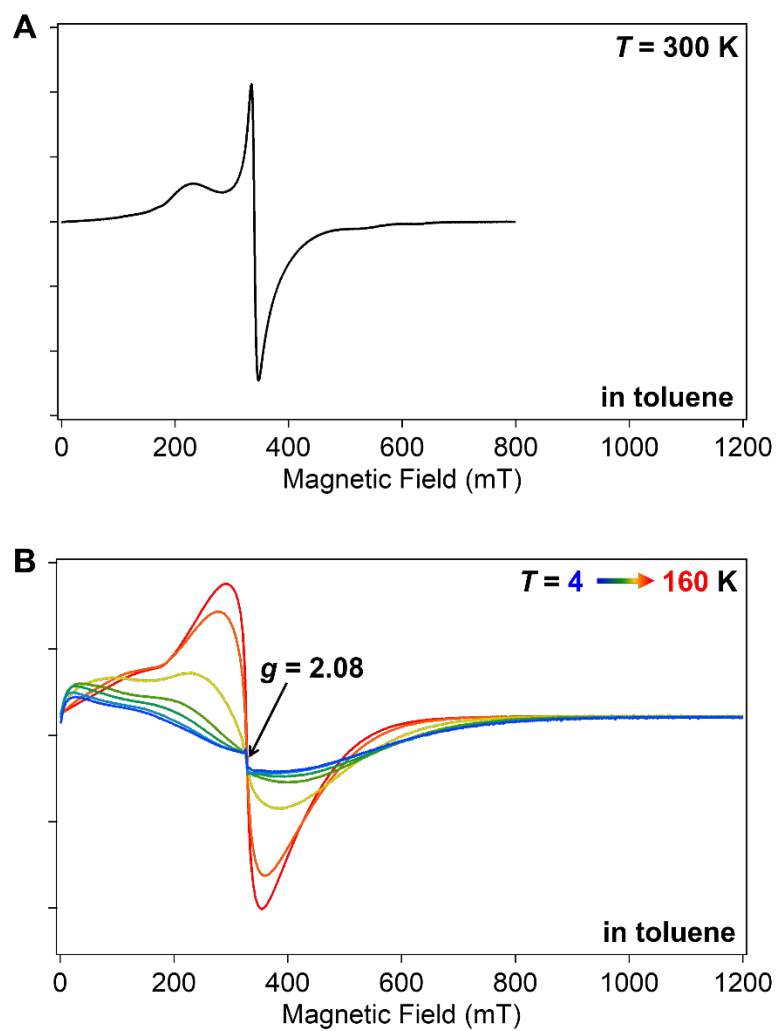


Figure S22. ESR spectra of $[\text{Fe}_{55}][\text{Fe}_6]$ in toluene collected (A) at 300 K and (B) from 4 to 160 K.

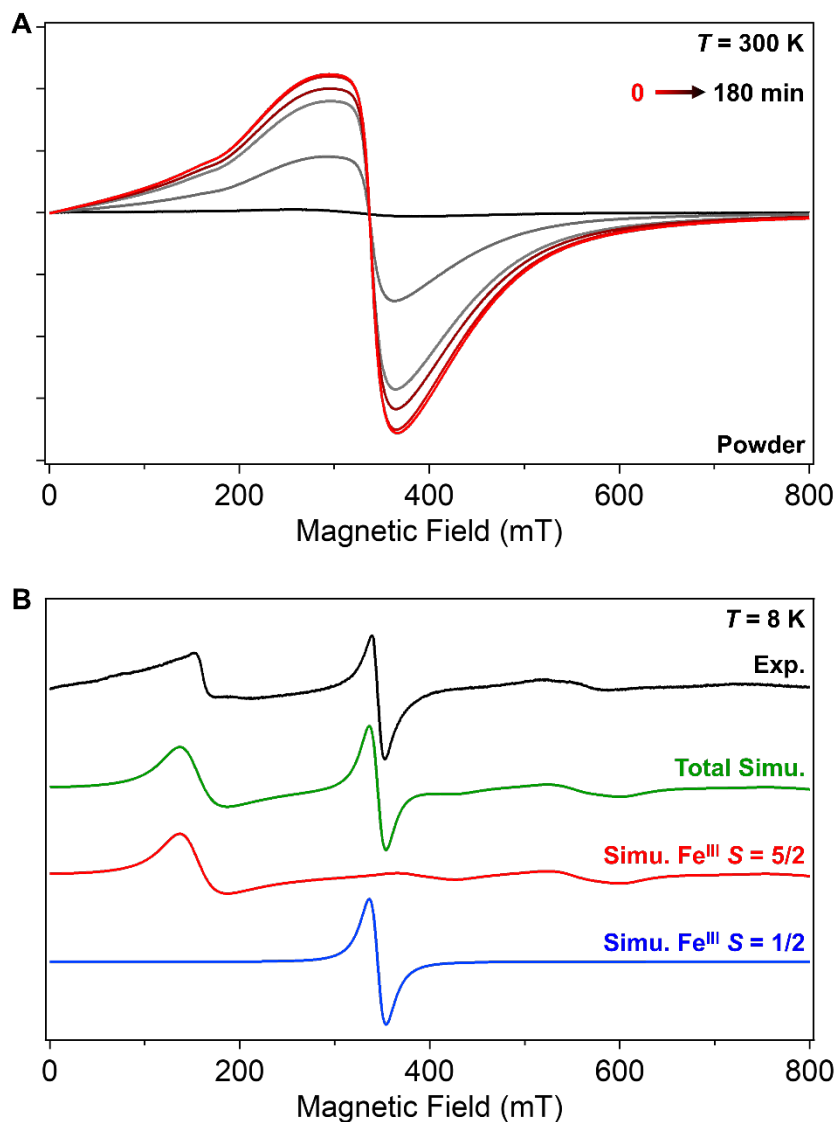


Figure S23. (A) Time course of ESR spectra during air-exposure of powder $[\text{Fe}_{55}][\text{Fe}_6]$. (B) ESR spectrum of air-exposed $[\text{Fe}_{55}][\text{Fe}_6]$ at 8 K and simulated spectra for high-spin and low-spin Fe^{III} . Parameters for the simulation are $g = 1.926$, line width = 31 mT, weight = 1.00 for Fe^{III} ($S = 1/2$), and $g = 1.545$, line width = 60 mT, $D = 450\text{ mT}$, $E = 5.0\text{ mT}$, weight = 6.45 for Fe^{III} ($S = 5/2$), respectively.

After data collection, the extremely air-sensitive $[\text{Fe}_{55}][\text{Fe}_6]$ was exposed to air by opening the J-Young valve, and the time course of ESR was measured. As the exposure to atmospheric conditions progressed, the intensity of the ESR signal decreased, and eventually, no signal was detected at 300 K (Figure S23). The color of the powder sample changed from black to red upon exposure. Upon re-cooling the exposed sample to 8 K, the ESR spectrum exhibited peaks at $g_{\text{eff}} \approx 4.2$ and 2.0 , which could be simulated by a mixture of Fe^{III} ions in both high ($S = 5/2$) and low ($S = 1/2$) spin states.

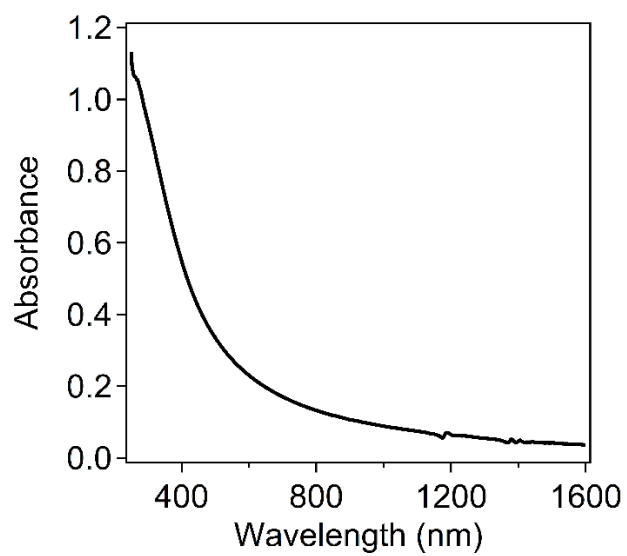


Figure S24. Absorption spectrum of $[\text{Fe}_{55}][\text{Fe}_6]$ in THF at room temperature (conc. = 0.1 mM).

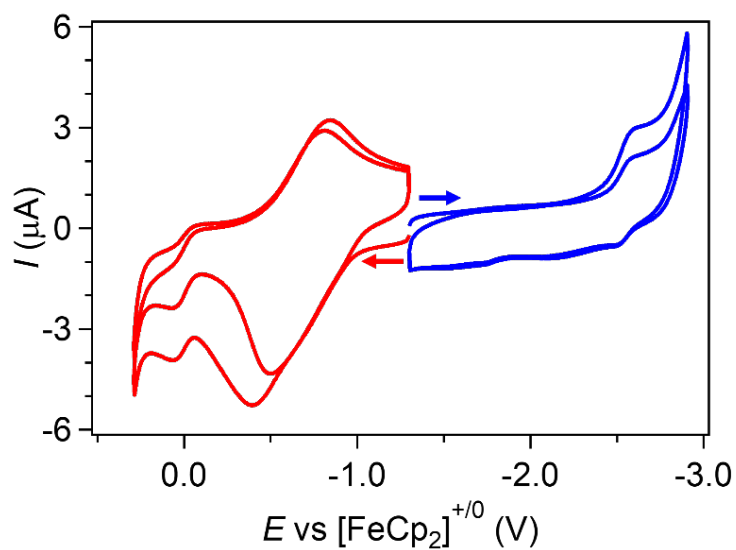


Figure S25. Cyclic voltammogram of $[\text{Fe}_{55}][\text{Fe}_6]$ in THF at room temperature; conditions: scan rate = 0.1 V/s; concentration of analyte = 0.2 mM; concentration of electrolyte ($[\text{nBu}_4\text{N}][\text{PF}_6]$) = 0.2 M; working electrode = glassy carbon; reference electrode = Ag/AgCl; counter electrode = Pt. The potentials were referenced to the ferrocene/ferrocenium ($[\text{FeCp}_2]^{+/0}$) redox couple.

Table S3. Crystal data and refinement summary of **[Fe₅₅][Fe₆•toluene]** and **[Fe₅₅][Fe{N(SiMe₃)₂}]₃**.

Sample Name	[Fe₅₅][Fe₆•toluene]	[Fe₅₅][Fe{N(SiMe₃)₂}]₃
Empirical formula	C ₂₂₂ H ₄₈₀ Fe ₆₁ N ₆ P ₁₂ Si ₁₂	C ₁₆₂ H ₃₇₈ Fe ₅₆ N ₃ P ₁₂ Si ₆
Formula weight	7349.66	6036.43
Temperature/K	173.15	123
Crystal system	trigonal	trigonal
Space group	$R\bar{3}$	$R\bar{3}c$
$a/\text{\AA}$	25.177(3)	19.6745(8)
$b/\text{\AA}$	25.177(3)	19.6745(8)
$c/\text{\AA}$	42.584(15)	112.652(8)
$\alpha/^\circ$	90	90
$\beta/^\circ$	90	90
$\gamma/^\circ$	120	120
Volume/ $\text{\AA}^3$	23377(10)	37764(4)
Z	3	6
$\rho_{\text{calc}}/\text{g/cm}^3$	1.566	1.593
μ/mm^{-1}	2.881	3.247
$F(000)$	11364.0	18546.0
Crystal size/ mm^3	$0.2 \times 0.02 \times 0.02$	$0.12 \times 0.12 \times 0.04$
Radiation	Mo K α ($\lambda = 0.71073$)	Mo K α ($\lambda = 0.71075$)
2 θ range for data collection/ $^\circ$	6.07 to 50.692	6.814 to 54.858
Index ranges	$-30 \leq h \leq 30, -30 \leq k \leq 30, -51 \leq l \leq 51$	$-25 \leq h \leq 25, -25 \leq k \leq 25, -145 \leq l \leq 145$
Reflections collected	74744	153581
Independent reflections	9525 [$R_{\text{int}} = 0.0850, R_{\text{sigma}} = 0.0659$]	9556 [$R_{\text{int}} = 0.0919, R_{\text{sigma}} = 0.0401$]
Data/restraints/parameters	9525/193/484	9556/24/404
Goodness-of-fit on F^2	0.858	1.027
Final R indexes [$I \geq 2\sigma(I)$]	$R_1 = 0.0722, wR_2 = 0.2097$	$R_1 = 0.0484, wR_2 = 0.1418$
Final R indexes [all data]	$R_1 = 0.1291, wR_2 = 0.2384$	$R_1 = 0.0727, wR_2 = 0.1527$
Largest diff. peak/hole / $\text{e \AA}^{-3}$	1.78/-0.59	0.77/-0.81

Table S4. Fitting results of Fe K-edge EXAFS of **[Fe₅₅][Fe₆]** and reference samples.

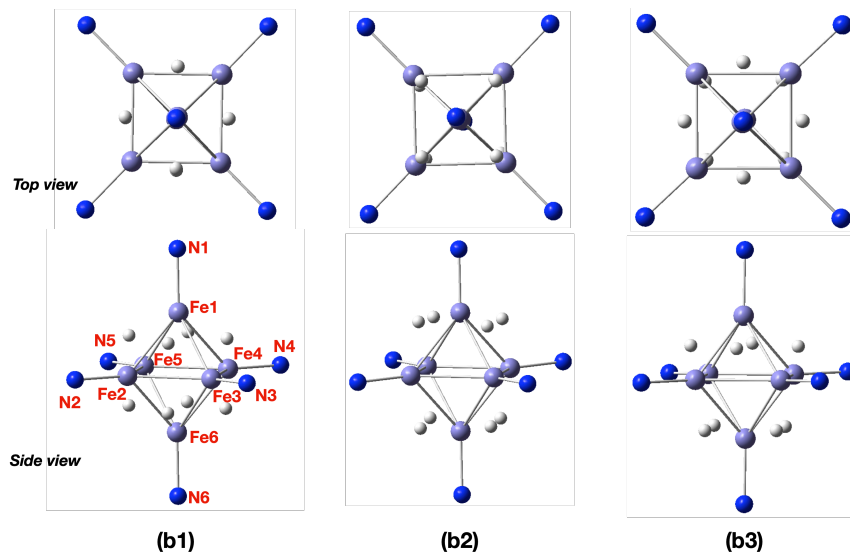
Sample	r Range (Å)	Bond	CN ^a	r (Å) ^b	σ ² (Å) ^c	R factor (%) ^d
[Fe ₅₅][Fe ₆]	1.3 – 2.6	Fe-Fe	3.7(2)	2.46(3)	0.011(4)	7.6
		Fe-Fe	4.4(3)	2.55(4)	0.015(5)	
[K(18-crown-6)]	1.2 – 1.9 (1 st shell)	Fe-N	2.2(3)	1.94(5)	0.006(5)	5.3
[Fe ^I {N(SiMe ₃) ₂ } ₂]	2.4 – 2.9 (2 nd shell)	Fe-Si	2.8(3)	3.06(6)	0.012(10)	14.5
Fe ^{II} {N(SiMe ₃) ₂ } ₂	1.2 – 2.3	Fe-N	1.8(2)	1.92(4)	0.003(2)	11.3
		Fe-N	1.7(3)	2.24(8)	0.011(10)	
Fe ^{II} Cl ₂	1.5 – 2.4	Fe-Cl	4.7(3)	2.48(3)	0.106(29)	14.6
Fe ^{III} Cl ₃	1.4 – 2.1	Fe-Cl	4.0(2)	2.20(3)	0.084(26)	14.1

a: Coordination number, b: Bond length, c: Debye-Waller factor, d: $[\sum(\chi^{\text{Expt}} - \chi^{\text{fit}})^2 / \sum(\chi^{\text{Expt}})^2]^{1/2}$

Table S5. Relative energies of the optimized structures of **[Fe₅₅]** calculated from the GFN1-xTB method. The positions of hydrides in the energetically most stable structures are shown in Figure S3. $\mu_3\text{-H}$ = hydrides bridging three Fe atoms, $\mu_2\text{-H}$ = hydrides bridging two Fe atoms.

Structure	Relative energy/kcal mol ⁻¹	Hydrides around the Fe- P ^t Bu ₃ units		Hydrides at the centers of the triangular surfaces	
		$\mu_3\text{-H}$	$\mu_2\text{-H}$	$\mu_3\text{-H}$	$\mu_2\text{-H}$
a1	0.0	27	12	6	1
a2	3.4	25	12	7	2
a3	3.9	28	11	5	2
a4	3.9	25	13	6	2
a5	4.9	23	14	8	1
a6	7.2	22	12	8	4
a7	7.3	25	13	5	3
a8	12.3	27	15	3	1
a9	12.9	23	15	6	2
a10	19.0	23	13	9	1

Table S6. Relative energies of the optimized structures of $[\text{Fe}_6]$, calculated from the GFN1-xTB method. Relative energies in parathesis are from the DFT single-point energy of the optimized structures. For simplicity, the SiMe_3 groups have been omitted from the structures of **b1-b3** shown below.

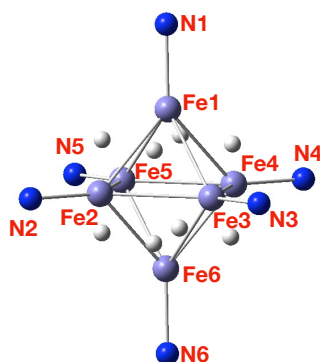


	$S = 1/2$	$S = 1/2$	$S = 1/2$
Relative energy/kcal mol ⁻¹	0.0 (0.0)	3.6 (27.6)	10.5 (61.3)
$\langle S^2 \rangle$	9.29	9.19	9.37
Spin density			
Fe1	3.03	-2.26	-2.80
N1	0.15	-0.20	-0.12
Fe2	-2.75	2.58	3.10
N2	-0.07	0.12	0.13
Fe3	-2.76	3.18	2.92
N3	-0.12	0.12	0.13
Fe4	3.03	-2.25	2.90
N4	0.15	-0.20	0.11
Fe5	2.99	3.08	-2.27
N5	0.13	0.16	-0.16
Fe6	-2.76	-3.17	-2.83
N6	-0.12	-0.14	-0.11

DFT single-point energy computations were performed for the $S = 1/2$ state, which is the most stable state (Table S7). The computed spin densities of the $S = 1/2$ state indicated antiferromagnetic coupling. According to GFN1-xTB or DFT relative energies, the most stable structure is **b1**, where all eight hydrides occupy the centers of the triangular Fe_3 surfaces, forming three bonds with the neighboring Fe

atoms (*i.e.*, $\mu_3\text{-H}$). In the case of **b2**, four hydrides form $\mu_3\text{-H}$ bonds, while the remaining four hydrides form $\mu_2\text{-H}$ bonds with neighboring Fe atoms. In **b3**, all eight hydrides appear as $\mu_2\text{-H}$.

Table S7. Relative energies of the $S = 1/2$, $3/2$, and $5/2$ states of $[\text{Fe}_6]$, along with their $\langle S^2 \rangle$ values, spin densities, and interatomic distances. Atomic labeling is shown below.



	$S = 1/2$	$S = 3/2$	$S = 5/2$	
Relative energy/kcal mol⁻¹	0.0	2.8	0.6	
$\langle S^2 \rangle$	9.58	11.35	15.41	
Spin density				
Fe1	3.23	3.19	2.74	
N1	0.18	0.16	0.13	
Fe2	2.95	3.25	-3.08	
N2	0.17	0.16	-0.16	
Fe3	-2.59	-2.08	-3.04	
N3	-0.15	-0.12	-0.16	
Fe4	-3.01	-2.84	2.83	
N4	-0.14	-0.13	0.13	
Fe5	2.95	3.25	2.7	
N5	0.17	0.17	0.11	
Fe6	-2.68	-2.08	2.64	
N6	-0.15	-0.11	0.11	
Bond distances/Å	$S = 1/2$	$S = 3/2$	$S = 5/2$	X-ray
Fe1-Fe2	2.63	2.66	2.72	2.67
Fe1-Fe3	2.72	2.68	2.68	2.67
Fe1-Fe4	2.63	2.66	2.55	2.65
Fe1-Fe5	2.73	2.68	2.6	2.64
Fe6-Fe2	2.73	2.74	2.71	2.65
Fe6-Fe3	2.56	2.6	2.72	2.65
Fe6-Fe4	2.73	2.75	2.57	2.67
Fe6-Fe5	2.56	2.57	2.56	2.67
AVG (neighboring Fe-Fe)	2.66	2.67	2.64	2.66
Fe1-Fe6	3.78	3.78	3.6	3.76

Fe2-Fe4	3.81	3.86	3.84	3.76
Fe3-Fe5	3.79	3.66	3.84	3.76
AVG (diagonal Fe-Fe)	3.79	3.77	3.76	3.76
Fe1-N1	1.91	1.92	1.92	2.00
Fe1-N2	1.92	1.92	1.91	2.00
Fe1-N3	1.91	1.9	1.92	2.00
Fe1-N4	1.91	1.92	1.92	2.00
Fe1-N5	1.92	1.91	1.92	2.00
Fe1-N6	1.94	1.93	1.92	2.00
AVG(Fe-N)	1.92	1.92	1.92	2.00

The ground state is $S = 1/2$, with the $S = 3/2$ and $5/2$ states being 2.8 and 0.6 kcal/mol higher in energy, respectively. The key structural parameters in the optimized ground state ($S = 1/2$) are in agreement with the crystal structure.

Cartesian coordinates of the optimized structures

a1

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H 9.404561883645121 4.794959392920509 17.67304173081842
H 11.77428776661326 5.94587244578175 18.93146782038145
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H 11.85042427225881 8.640957363814953 18.95163961136932
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b1

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N	1.13170000	11.59750000	9.36850000
C	3.39831046	10.60463731	11.16095326
C	3.17389892	13.57438350	10.62066372
C	1.98926614	9.02947202	8.00875502
C	0.99129426	12.22480670	12.32788227

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C	-0.94702430	9.90516679	7.78747380
Fe	0.94790000	15.74900000	8.17360000
Si	3.65277078	17.30542526	8.96101879
Si	1.14423376	17.63041563	10.76846480
N	1.97890000	16.98530000	9.36850000
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Fe	-1.52450000	14.75040000	8.17360000
Si	-4.22581386	16.30517750	8.93286423
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C	-4.16916148	12.21573232	7.03040957
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Si	2.19461968	11.94275493	10.72876689
N	1.13170000	11.59750000	9.36850000
C	3.39299431	10.47441386	11.13347071
C	3.43785813	13.39902198	10.54880950
C	1.84226999	8.86066447	8.26577597
C	1.14486759	12.32621185	12.30025196
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C	-1.06495589	9.73537031	8.05747863
Fe	0.94790000	15.74900000	8.17360000
Si	3.61425746	17.26968912	8.84164634
Si	1.10876148	17.56796336	10.75974750
N	1.97890000	16.98530000	9.36850000
C	1.56014086	19.40265001	11.15322967
C	-0.78232542	17.51236000	10.47261878
C	3.75174162	18.91744712	7.85367907
C	1.50944269	16.51101909	12.31796634
C	4.83647486	17.38546159	10.33150713
C	4.24884367	15.85541208	7.71780207
Fe	-1.52450000	14.75040000	8.17360000
Si	-4.26483833	16.30757650	9.00653087
Si	-3.32553809	13.99236532	10.78012063
N	-3.11060000	15.02510000	9.36850000
C	-5.19204433	13.59828267	11.12270538
C	-2.56254676	12.22844548	10.73383428
C	-5.80883253	15.58360510	8.10103402
C	-2.57407493	14.83211130	12.34599522

C	-4.84643731	17.22663945	10.61008372
C	-3.69691552	17.76842079	7.89199842
Fe	-0.57660000	15.96340000	6.02110000
Si	-0.63155927	19.11872542	5.20430459
Si	-2.13867887	17.15094334	3.41793631
N	-1.13170000	17.47440000	4.82620000
C	-3.99862684	17.37695439	3.88020369
C	-2.02431627	15.41559816	2.59346242
C	-2.05987296	20.39566522	4.91400678
C	-1.72460651	18.31032473	1.92176017
C	0.89634523	19.59981923	4.12834357
C	-0.11335079	19.52917050	7.01110335
Fe	-0.94790000	13.32290000	6.02110000
Si	-3.66810487	11.92468495	5.23808090
Si	-1.06025700	11.29675376	3.56948277
N	-1.97890000	12.08660000	4.82620000
C	-0.46396467	9.54692756	4.11109702
C	0.49048585	12.32529320	3.12358380
C	-4.28474399	10.10798044	5.03697660
C	-2.09321634	11.09866645	1.95198473
C	-4.78006486	13.05205500	4.14290339
C	-3.97184199	12.41235760	7.06595764
Fe	1.52450000	14.32150000	6.02110000
Si	4.27837410	12.76250836	5.11653639
Si	3.37064844	15.25101937	3.56010082
N	3.11060000	14.04680000	4.82620000
C	4.24109909	16.81762444	4.26973010
C	1.83181911	15.89070646	2.59811924
C	6.08054813	13.45981625	5.24374098
C	4.47678494	14.60394357	2.10570795
C	4.21400277	11.43269001	3.71653448
C	4.09628875	11.72756932	6.72424706
H	2.92377916	9.50365567	11.30805160

H	4.12631036	10.31952462	10.33461009
H	4.13155693	13.39434784	11.39898451
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H	2.99202667	14.39609804	10.53340891
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H	2.82219018	8.85764618	8.75027920
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H	4.62937078	18.22034792	11.00803802
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H	-5.29698220	13.01523762	12.04528286
H	-2.88505093	11.66985316	11.62139396
H	-2.88592670	11.63795439	9.87251991
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H	-6.57718256	16.34811449	7.93896193
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H	-5.62638217	17.96017603	10.37473029
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H	-4.02794580	17.79307407	11.06746537
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H	-2.77927383	18.25673736	8.22968803
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H	-4.31797816	16.66066374	4.64485065
H	-2.58361235	15.42462640	1.64929101
H	-1.00526213	15.11704852	2.33594165
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H	1.22756438	20.62570345	4.32419504
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H	0.73049905	19.53013428	3.04958407

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H	-1.27320936	8.89542005	4.45575313
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H	6.80908578	12.64850716	5.35156622
H	6.41410779	14.05658412	4.39049844
H	0.25576265	13.19271919	2.50123869
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H	-3.39093122	13.30482927	7.35921495
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H	-1.24943503	14.79949516	5.19460761
H	-1.80804704	15.86843006	7.02710911
H	-0.76226325	13.54079910	8.93506661
H	-0.28372949	12.24194492	7.16807445
H	1.29238513	14.35115572	8.89981086
H	1.74593330	13.12125091	7.09328118

b3

Fe	0.57660000	13.10850000	8.17360000
Si	0.57131287	9.95032268	9.11660713
Si	2.12346053	12.05850193	10.74290617
N	1.13170000	11.59750000	9.36850000
C	3.43941663	10.70784363	11.17529284
C	3.18021283	13.64124066	10.51473557
C	1.95186617	8.87699418	8.29946922
C	1.02895821	12.35565225	12.30134633
C	0.06168799	9.09796944	10.77815527
C	-0.97416203	9.71100347	8.00114544
Fe	0.94790000	15.74900000	8.17360000
Si	3.63781435	17.30438210	8.90604757
Si	1.13903978	17.64380750	10.75506422
N	1.97890000	16.98530000	9.36850000
C	1.64171532	19.48057531	11.08757859

C	-0.77142831	17.69604636	10.60061185
C	3.76074650	18.96785656	7.93848672
C	1.54466942	16.61708840	12.33478458
C	4.79722294	17.42570977	10.44806747
C	4.46111615	15.97425259	7.79495894
Fe	-1.52450000	14.75040000	8.17360000
Si	-4.24325841	16.28415634	8.92351670
Si	-3.23714658	13.97669026	10.76576691
N	-3.11060000	15.02510000	9.36850000
C	-5.07456366	13.59489302	11.23402586
C	-2.44229210	12.24640737	10.54094888
C	-5.85607502	15.55600081	8.15487760
C	-2.41332787	14.79737835	12.30274733
C	-4.74321692	17.34078715	10.46278154
C	-3.58249060	17.54518307	7.64015365
Fe	-0.57660000	15.96340000	6.02110000
Si	-0.45344877	19.03222721	5.27309091
Si	-2.20320083	17.26188737	3.45045270
N	-1.13170000	17.47440000	4.82620000
C	-4.00965170	17.77792426	3.89036010
C	-2.34255663	15.48591094	2.73901702
C	-1.71286032	20.47799444	5.01280006
C	-1.62868834	18.31320926	1.93110064
C	1.12163115	19.41318307	4.22907454
C	0.06138365	19.23162511	7.10969122
Fe	-0.94790000	13.32290000	6.02110000
Si	-3.63583489	11.63009797	5.19512208
Si	-1.10931497	11.33025106	3.49264370
N	-1.97890000	12.08660000	4.82620000
C	-0.19885659	9.74314150	4.10226150
C	0.21437282	12.38494730	2.57991152
C	-3.88305033	9.71010395	5.14188797
C	-2.27352446	10.83080012	2.02604364

C	-4.85563003	12.46941033	3.95593205
C	-4.32559130	12.08730966	6.92770785
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Si	3.30982514	15.23387437	3.55299490
N	3.11060000	14.04680000	4.82620000
C	4.33852231	16.73703618	4.18233754
C	1.66884955	15.91129177	2.82646334
C	5.99802302	13.20939770	5.17937672
C	4.24744468	14.51950296	2.02002451
C	3.91862400	11.28949380	3.83417293
C	3.83599472	11.83155499	6.85088092
H	3.03235086	9.72598630	11.43085727
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H	3.70487802	13.88875314	11.44528407
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H	2.15050986	9.17768393	7.26559748
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H	-1.22385973	16.73419594	10.34130620
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H	1.19132388	15.58363765	12.25719471
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H	4.80857609	16.50014598	11.03301253
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H	5.43371066	16.32670213	7.43192414
H	4.65472393	15.03014097	8.31123322
H	3.88163262	15.71806827	6.90280315
H	-5.67784361	14.47665128	11.46859803
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H	0.55452464	9.96704382	4.86492357
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H	0.56215364	11.84646647	1.68949018
H	-4.93237155	9.44661156	5.31868928
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H	5.31293327	16.45218921	4.59190837
H	1.85358861	16.77669358	2.17989935
H	1.14422145	15.17775983	2.20783706
H	0.94679295	16.24454211	3.57902926
H	4.37429232	15.28932956	1.24973387
H	5.24997542	14.13993624	2.23685202
H	3.70502233	13.69836862	1.53983454
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H	4.59275240	10.44451727	4.01494604
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H	2.79322432	11.52950209	7.00281475
H	4.43314254	10.91735900	6.95172975
H	-0.79170973	9.59398426	11.25213401
H	4.04399808	11.01932657	12.03522380
H	6.19958063	13.96668009	5.94411784
H	6.65541020	12.36276515	5.40800013
H	6.35767214	13.62844519	4.23532121
H	-0.17206023	13.34227097	2.21960572
H	-2.69264179	11.70809974	1.52171215
H	-4.41874138	13.16086633	7.10790585
H	1.07794779	15.90443104	6.30296235
H	0.47540624	13.37828697	5.29141663
H	-1.29183408	14.71262361	5.31969881
H	-0.75588760	16.13809184	7.66228941
H	-2.05083017	13.68728274	7.12308774
H	-0.28706350	12.32400897	7.06028333
H	-0.11219208	14.37424230	9.00431703
H	1.77754430	14.09942533	7.63786989

b1 optimized structures without any constrain

S=1/2

Fe	0.63888544105142	13.11185999338003	8.20021322832033
Si	0.78405254963119	10.00752499712685	8.98663509516993
Si	2.24037618961421	12.00327479434543	10.72084333722693
N	1.23182354269241	11.67610553735290	9.31803236362835
C	3.56995986379667	10.67419642021577	11.01090940119805
C	3.22039037876019	13.60230939525063	10.56543143052578
C	2.18165542077551	9.02568286922309	8.16321192238390
C	1.20414666279626	12.12800452166445	12.30228728262322
C	0.27325325807983	9.06837236212494	10.56019630602265
C	-0.70579289990038	9.87093027023886	7.85124309927744
Fe	0.95882450355941	15.72319075205695	8.27301160239113
Si	3.53563329995456	17.37804619654990	9.19439522332198
Si	1.00850848357014	17.55943155868288	10.86426878809225
N	1.85725363830566	16.92826416116963	9.46022611919658
C	1.65057536335023	19.25046434683587	11.45417972632194
C	-0.81873881715915	17.86694411018672	10.52729599823840
C	3.74630465504904	19.18499304770537	8.65944939856054
C	1.15454488543623	16.38080984092359	12.33877080610934
C	4.60868144007040	17.12821920559254	10.74461211532845
C	4.35067795452879	16.34107927388759	7.85116870320750
Fe	-1.50703877548030	14.77776169628187	8.11744240411592
Si	-4.23766961741974	16.31336794607203	8.90398403409498
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