Supporting Information for

NHC and nucleophile chelation effects on reactive iron(II) species in alkyl-alkyl cross-coupling

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Table of Contents

1. General Considerations and Synthetic Procedures

Commercially available chemicals were purchased from the specified vendors and used without further purification. Iron(II) acetate was purchased from Sigma Aldrich (95 %; Lot#MKBN5638V); 1,3-Bis(2,4,6-trimethylphenyl)imidazolium chloride and 1,3-Bis(2,6-diisopropylphenyl)imidazolinium chloride from TCI; (2-(1,3-dioxan-2-yl)ethyl)magnesium bromide (0.5 M), 1-iodo-3-phenylpropane, 1,2-dibromoethane, (2-bromoethyl)cyclohexane, 2-hydroxybenzaldehyde phenylhydrazone and dodecane from Sigma Aldrich; 1-bromo-3-methoxypropane from Oakwood Chemical; 3-bromo-1,1-dimethoxypropane from Acros Organics; 2-(2-bromoethyl)pyridine hydrobromide from Accela and magnesium from Strem. . $^{57}Fe(OAc)_2$ was synthesized according to literature[1] procedure using ^{57}Fe metal powder purchased from Isoflex USA. All anhydrous solvents were purchased from Sigma Aldrich, dried with alumina and stored over molecular sieves in inert atmosphere.

All air and moisture sensitive chemistry was carried out in an MBraun inert atmosphere N_2 dry box with a direct nitrogen inlet hose. The alkyl-alkyl cross-coupling reaction for in situ Mössbauer spectroscopic studies was performed as reported in the literature.[2]

^1H and $^{13}C\{^1H\}$ NMR spectra were recorded on a Bruker 400 MHz or 500 mHz spectrometer and were recorded at ambient temperature. Chemical shifts (δ) are recorded in parts per million (ppm). Coupling constants (*J*) are recorded in Hertz (Hz). Chemical shifts are referenced relative to residual solvent and solvent peaks, respectively ($CDCl_3$ 7.27 ppm, 77.2 ppm; d^8-THF 1.73/3.58 ppm, 25.37/67.57 ppm).

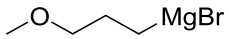
Gas Chromatography was carried out using a Shimadzu GC-2010 Plus with an RTX-1 column, 60 m, 0.25 mm internal diameter. Data workup carried out on Lab Solutions Lite. Details of method used: injection volume 1 µl; injection temperature 300 °C, 100 °C (hold 2 min); ramp rate 15 °C/min ending and holding at 300 °C for 3 min.

Gas chromatography mass spectrometry was carried out using a Shimadzu GCMS-QP2010 equipped with an RTX-XLB column, 30 m, 0.25 mm internal diameter with a quadrupole mass analyzer using helium as the carrier gas. Details of method used: injection volume 5 µl; injection temperature 225 °C with a 25:1 split ratio. The gas flow was held at 1.0 ml/min and 7.8 psi for the duration of the run. The interface temperature was held at 250 °C and the ion source (EI, 30 eV) was held at 250 °C. The initial oven temperature was held at 50 °C for 3 min with the detector off, followed by a temperature ramp, with the detector on, to 280 °C at 40 °C/min, before holding the temperature at 280 °C for 3 min. The total run time was 11.75 min.

General Procedure 1 (Table 2)

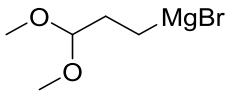
A suspension of $Fe(OAc)_2$ (1.8 mg; 0.010 mmol) and carbene salt (0.024 mmol) in anhydrous THF (2 ml) was placed to stir at the relevant temperature for 15 minutes after which time (2-(1,3-dioxan-2-yl)ethyl)magnesium bromide (0.5 M; 245 µl; 0.123 mmol) was added dropwise to the stirring suspension. After the relevant amount of time, the resulting solution was removed to stir at room temperature for 15 minutes if previously at elevated temperature. 1-iodo-3-phenylpropane (65 µl; 0.404 mmol) was then added dropwise to the stirring solution and (2-(1,3-dioxan-2-yl)ethyl)magnesium bromide (0.5 M; 1 ml; 0.5 mmol) added using a syringe pump at a rate of 0.16 ml/hour. On completion of the addition of (2-(1,3-dioxan-2-yl)ethyl)magnesium bromide, dodecane (46 µl; 0.203 mmol) was added followed by saturated aqueous NH_4Cl (~2 ml) and additional THF (~3 ml). The organic phase was filtered through a plug of Na_2SO_4 (~3 cm in a pasteur pipette) and the crude reaction mixture analyzed by GC.

(2-methoxypropyl)magnesium bromide



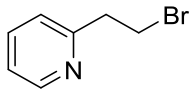
A suspension of magnesium (226.4 mg; 9.3 mmol) in anhydrous THF (15 ml) was stirred at 20 °C for 20 minutes, after which time 1,2-dibromoethane (4 drops) was added and the suspension stirred for an additional 10 minutes. 1-bromo-3-methoxypropane (0.84 ml; 7.5 mmol) was then added dropwise over 17 minutes. After stirring for an additional 90 minutes at 20 °C before filtration. The concentration of (2-methoxypropyl)magnesium bromide was determined to be 0.46 M by titration using 2-hydroxybenzaldehyde phenylhydrazone.

(3,3-dimethoxypropyl)magnesium bromide



A suspension of magnesium (244.6 mg; 10.1 mmol) in anhydrous THF (14.5 ml) was stirred at 20 °C for 20 minutes, after which time 1,2-dibromoethane (4 drops) was added and the suspension stirred for an additional 20 minutes. 3-bromo-1,1-dimethoxypropane (1 ml; 7.3 mmol) was then added dropwise over 22 minutes. After stirring for an additional 105 minutes at 20 °C,the suspension was filtered and the concentration of (2-methoxypropyl)magnesium bromide was determined to be 0.44 M by titration using 2-hydroxybenzaldehyde phenylhydrazone.

2-(2-bromoethyl)pyridine



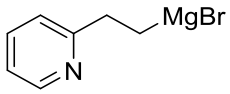
2-(2-bromoethyl)pyridine hydrobromide (1.5027 g; 5.63 mmol) was dissolved in 20 % aqueous potassium hydroxide (3 ml) and transferred to a separating funnel and extracted with dichloromethane (4 × 10 ml). The combined organic extracts were then washed with water (5 ml), dried over sodium sulphate, filtered and concentrated *in vacuo* to afford 2-(2-bromoethyl)pyridine as a pale yellow oil (919.2 mg; 4.94 mmol; 88 %).

^{1}H NMR (400 MHz, CDCl3)

8.57 (d, *J* = 5.0 Hz, 1H), 7.63 (t, *J* = 7.6 Hz, 1H), 7.24 – 7.11 (m, 2H), 3.78 (t, *J* = 7.1 Hz, 2H), 3.34 (t, *J* = 7.1 Hz, 2H).

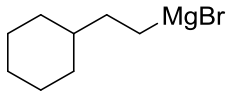
Data in accordance with that previously reported.[3]

(2-(pyridine-2-yl)ethyl)magnesium bromide



A suspension of magnesium (365 mg; 15 mmol) in anhydrous THF (8 ml) was stirred for 15 minutes at 20 °C, after which time 1,2-dibromoethane (4 drops) was added and the suspension stirred for a further 20 minutes. A solution of 2-(2-bromoethyl)pyridine (919.2 mg; 4.94 mmol) in anhydrous THF (1.9 ml) was added dropwise over 15 minutes, during which time the suspension rapidly turned dark orange/red. After stirring for 90 minutes, the suspension was filtered and the concentration of (2-(pyridine-2-yl)ethyl)magnesium bromide determined to be 0.34 M by titration using 2-hydroxybenzaldehyde phenylhydrazone.

(2-cyclohexylethyl)magnesium bromide



A suspension of magnesium (389 mg; 16 mmol) in anhydrous THF (15 ml) was stirred for 15 minutes at 20 °C, after which time 1,2-dibromoethane (4 drops) was added and the suspension stirred for a further 20 minutes. A solution of (2-bromoethyl)cyclohexane (2.68 g; 14 mmol) in anhydrous THF (5 ml) was added dropwise over 15 minutes. After stirring for 90 minutes, the suspension was filtered and the concentration of (2-cyclohexylethyl)magnesium bromide determined to be 0.49 M by titration using 2-hydroxybenzaldehyde phenylhydrazone.

General procedure for catalytic reactions with alternative nucleophiles (Scheme 2)

A suspension of $Fe(OAc)_2$ (1.8 mg; 0.010 mmol) and 1,3-bis(2,4,6-trimethylphenyl)imidazolium chloride (8.3 mg; 0.024 mmol) in anhydrous THF (2 ml) was placed to stir at 54 °C for 15 minutes, after which time Grignard reagent (0.123 mmol) was added dropwise to the stirring suspension. After 1 minute, the resulting solution was removed to stir at room temperature for 15 minutes. 1-iodo-3-phenylpropane (65 µl; 0.404 mmol) was then added dropwise to the stirring solution and additional Grignard reagent (0.5 mmol) added using a syringe pump at a rate of 0.08 mmol/hour. On completion of the addition of Grignard reagent, dodecane (46 µl; 0.203 mmol) was added followed by saturated aqueous NH_4Cl (~2 ml) and additional THF (~3 ml). The organic phase was filtered through a plug of Na_2SO_4 (~3 cm in a pasteur pipette) and the crude reaction mixture analyzed by both GC and GCMS.

2. Supplementary Spectral Data

2.1 EPR

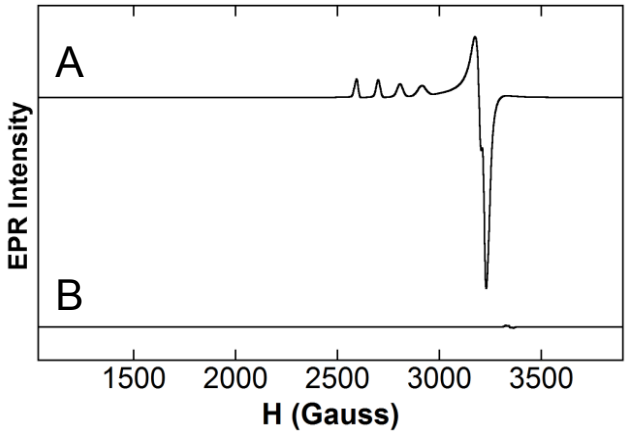


Figure S1. (A) 10 K EPR spectrum of 5 mM $CuSO_4$•$5H_2O$ in 9:1methanol:ethanol. (B) 10 K EPR spectrum of 5 mM Fe solution for pre-catalyst formation (**1**) by the addition of 12 equiv (2-(1,3-dioxan-2-yl)ethyl)MgBr to 2.1 equiv IMes•HCl and 1.0 equiv $Fe(OAc)_2$ in THF at 54 °C. Spectra A and B were both recorded at 0.10 mW (low power is necessary for the Cu sample signal to not saturate) and are on the same y-axis scale for direct comparison of EPR signal intensity.

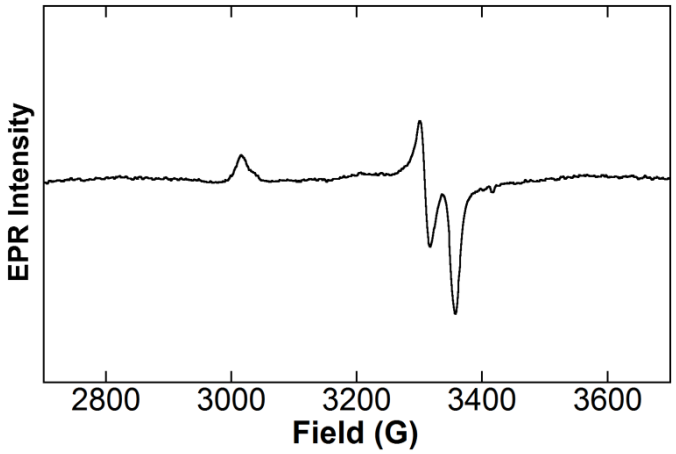


Figure S2. 100 K EPR spectrum of the reported pre-catalyst formation step (formation of **1**) of the catalytic reaction, replicated from reported methods (modulation frequency 100 kHz, modulation amplitude 5 G, power 6.32 kW).[2] 12 equiv (2-(1,3-dioxan-2-yl)ethyl)magnesium bromide was added to 1.0 equiv $Fe(OAc)_2$ and 2.1 equiv IMes•HCl in THF at 54 °C and stirred for 20 min. The sample was frozen in liquid nitrogen after 20 minutes of stirring. Spin integration is consistent with 10 K data with *S* = 1/2 iron representing< 0.1 % of all iron in solution (see Figure S6).

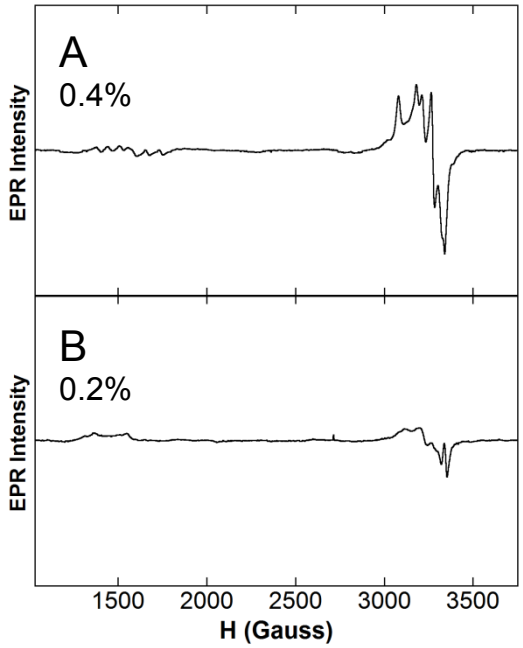


Figure S3. (A) 10 K EPR spectrum of *in situ* formed iron species in 5 mM Fe solution for pre-catalyst (**1**) formation by the addition of 12 equiv (2-(1,3-dioxan-2-yl)ethyl)MgBr to 2.1 equiv SIPr•HCl and 1.0 equiv $Fe(OAc)_2$ in THF at 54 °C for 20 min (B) 10 K EPR spectrum of 5 mM Fe solution for pre-catalyst (**1**) formation by the addition of 12 equiv (2-(1,3-dioxan-2-yl)ethyl)MgBr to 2.1 equiv SIPr•HCl and 1.0 equiv $Fe(OAc)_2$ in THF at 23 °C for 1 h. Spectra A and B are on the same y-axis scale for direct comparison of EPR signal intensity; percentages given are the relative percent of all $S = 1/2$ iron as a result of spin integration with $CuSO_4•5H_2O$.

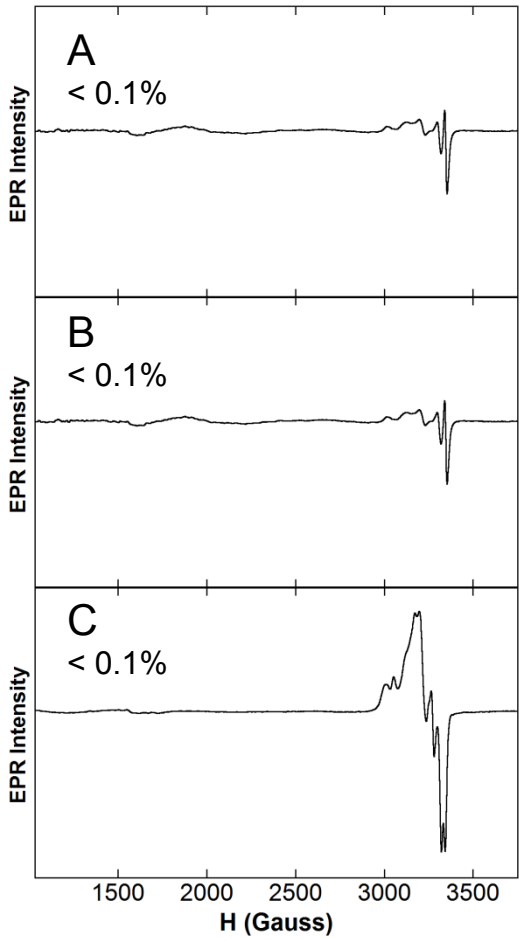


Figure S4. 10 K EPR spectra of *in situ* formed iron species in THF during the pre-catalyst formation step where 12 equiv (2-(1,3-dioxan-2-yl)ethyl)magnesium bromide was added to 1.0 equiv $Fe(OAc)_2$ and 2.1 equiv IMes•HCl in THF at 23 °C and stirred for 1 h (A) and 2 h (B). Spectra C shows the addition of 40 equivalents of 1-iodo-3-phenylpropane to the 1 h time point mixture, followed by the slow addition of 0.5 M (2-(1,3-dioxan-2-yl)ethyl)magnesium bromide at 0.16 mL/hr at 23 °C for 2 h. Spectra A and B are on the same y-axis scale for direct comparison of EPR signal intensity; percentages given are the relative percent of all $S = 1/2$ iron as a result of spin integration with $CuSO_4•5H_2O$.

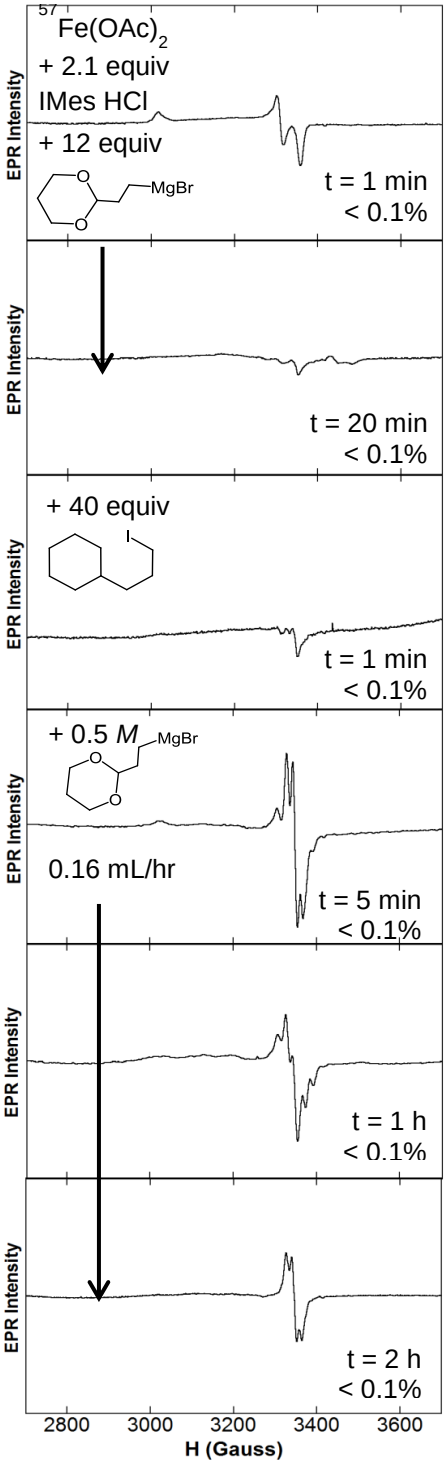


Figure S5. 10 K EPR spectra of *in situ* iron species as a function of time throughout the reported catalytic reaction by Cárdenas in THF.[2] Spin quantitation indicates < 0.1% of all iron in solution is S = 1/2 iron. Note that some small variations in the trace S = 1/2 signal are observed during slow Grignard addition, indicating that additional off-cycle S = 1/2 species can form during catalysis. However, these species are present in exceptionally low amounts and, hence, could not be further analyzed. Percentages given are the relative percent of all S = 1/2 iron as a result of spin integration with $CuSO_4 \bullet 5H_2O$.

2.2 Mössbauer

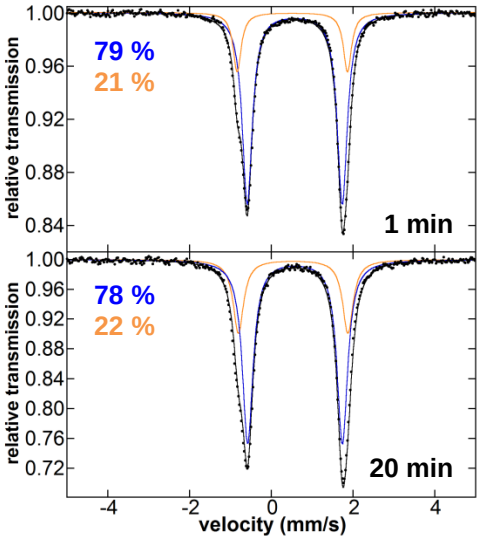
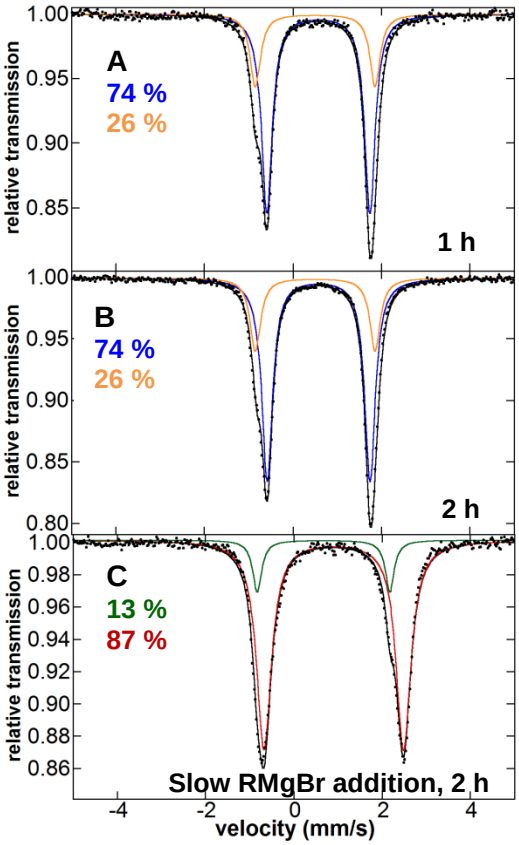


Figure S6. 5 K ^{57}Fe Mössbauer spectra of *in situ* formed iron species in THF during the reported pre-catalyst formation step where 12 equiv (2-(1,3-dioxan-2-yl)ethyl)magnesium bromide is added to 1.0 equiv $^{57}Fe(OAc)_2$ and 2.1 equiv IMes•HCl in THF at 54 °C and stirred for 20 min. Data (black dots), total fit (black line), and fit components are shown. The blue component represents **1** with Mössbauer parameters of δ = 0.57 mm/s and ΔE_Q = 2.33 mm/s and the orange component represents **1-THF** with δ = 0.50 mm/s and ΔE_Q = 2.70 mm/s.



2.8

Figure S7. 5 K ^{57}Fe Mössbauer spectra of *in situ* formed iron species in THF during the reported pre-catalyst formation step where 12 equiv (2-(1,3-dioxan-2-yl)ethyl)magnesium bromide is added to 1.0 equiv $^{57}Fe(OAc)_2$ and 2.1 equiv IMes•HCl in THF at 23 °C and stirred for 1 h (A) and 2 h (B). Data (black dots), total fit (black line), and fit components are shown. The blue component represents **1** with Mössbauer parameters of δ = 0.57 mm/s and ΔE_Q = 2.33 mm/s and the orange component represents **1-THF** with δ = 0.50 mm/s and ΔE_Q = 2.70 mm/s. Spectra C shows the addition of 40 equiv of 1-iodo-3-phenylpropane to the 1 h time point mixture, followed by the slow addition of 0.5 M (2-(1,3-dioxan-2-yl)ethyl)magnesium bromide at 0.16 mL/hr at 23 °C for 2 h. The green fit represents **2-Br** with Mössbauer parameters of δ = 0.67 mm/s and ΔE_Q = 2.98 mm/s and red represents **3** with δ = 0.90 mm/s and ΔE_Q = 3.19 mm/s.

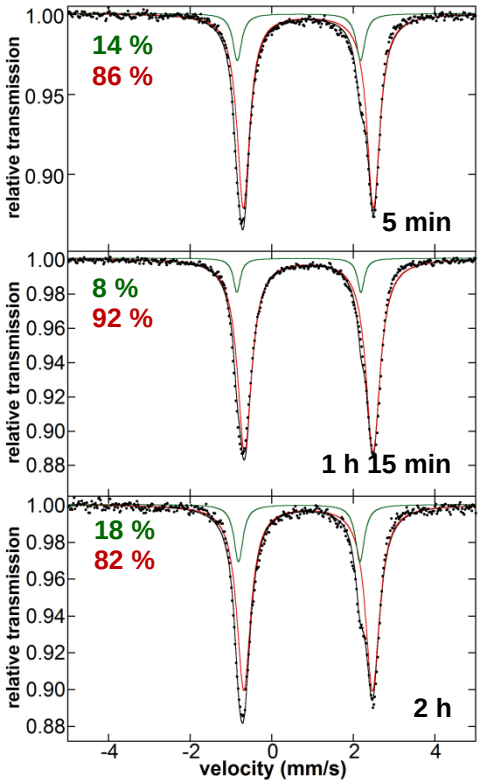


Figure S8. 5 K ^{57}Fe Mössbauer spectra of *in situ* formed iron species in THF following pre-catalyst formation, addition of 40 equiv of 1-iodo-3-phenylpropane, during the slow addition of 0.5 M (2-(1,3-dioxan-2-yl)ethyl)magnesium bromide at 0.16 mL/hr at 23 °C. Data (black dots), total fit (black line), and fit components are shown. The green fit represents **2-Br** with Mössbauer parameters of δ = 0.67 mm/s and ΔE_Q = 2.98 mm/s and red represents **3** with δ = 0.90 mm/s and ΔE_Q = 3.19 mm/s.

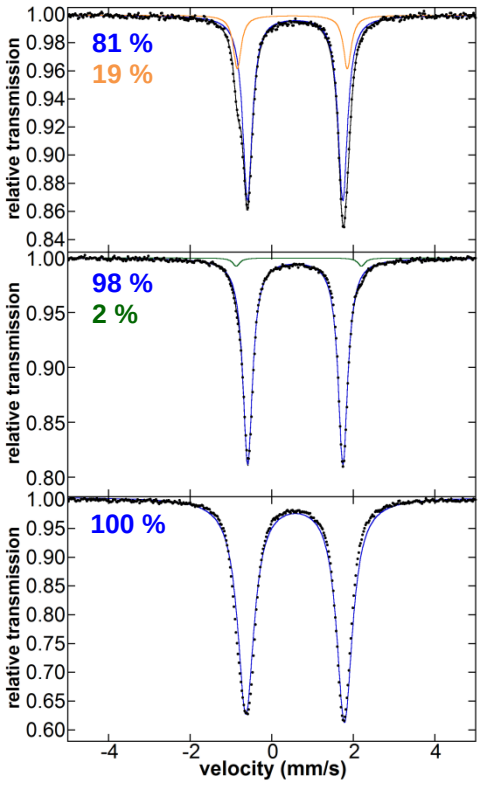


Figure S9. 5 K ^{57}Fe Mössbauer spectra of isolated (IMes)^{57}Fe((1,3-dioxan-2-yl)ethyl)$_2$ (**1**) in THF (top), methyl-THF (middle), and as a solid diluted with boron nitride (bottom). The frozen THF solution sample shows the presence of **1** and **1-THF**. Dissolving **1** in 2-MeTHF shows no THF adduct, but has a small impurity of **2-Br**, and the solid shows **1** with some asymmetry and distortion from solution parameters as previously reported for iron(II)-SciOPP solid samples.[4]

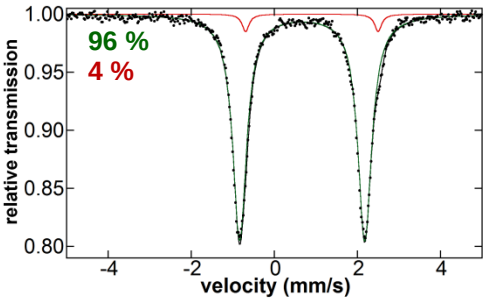


Figure S10. Solid 5 K ^{57}Fe Mössbauer spectrum of isolated (IMes)^{57}FeBr((1,3-dioxan-2-yl)ethyl) (**2-Br**, green) and(IMes)^{57}FeBr$_2$(THF) (**3**, red) diluted with boron nitride, no change in individual component parameters is observed compared to frozen solution though less **3** is observed in this particular crystalline sample.

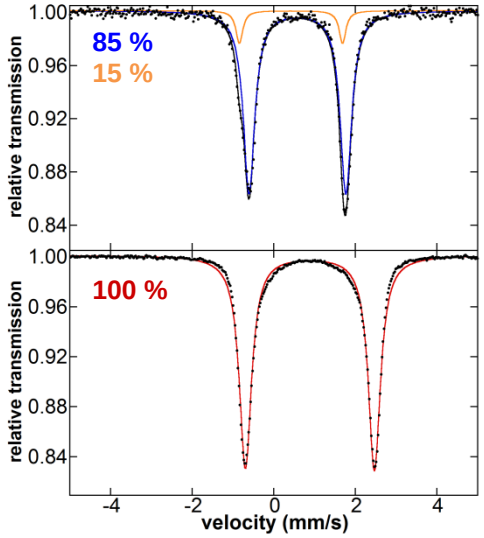


Figure S11. 5 K ^{57}Fe Mössbauer spectra of isolated (IMes)^{57}FeBr((1,3-dioxan-2-yl)ethyl) and (IMes)^{57}FeBr$_2$(THF) (85:15 mixture) reacted with 1.1 equiv of (2-(1,3-dioxan-2-yl)ethyl)magnesium bromide at 23 °C for 2 min (top). The resulting iron species mixture shows the reformation of **1** (blue) and **1-THF** (orange). Subsequent reaction with 20 equiv of 1-iodo-3-phenylpropane shows the exclusive formation of **3** as expected along with the formation of 2 equiv of product (with respect to iron) (see Table S1).

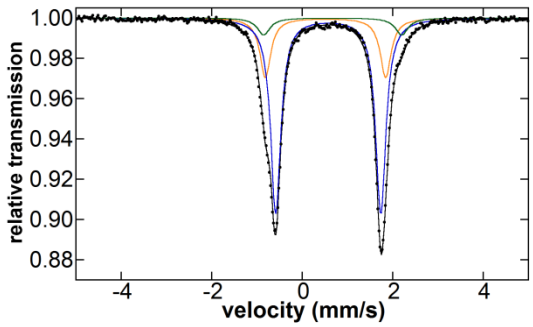


Figure S12. 5 K ^{57}Fe Mössbauer spectrum of isolated (IMes)^{57}Fe((1,3-dioxan-2-yl)ethyl)$_2$ (**1**) in THF 2 min after reaction with 0.7 equiv 1-bromo-3-phenylpropane at 23 °C. Reactivity occurs at a much slower rate than with alkyl iodide producing only 7 % **2-Br** (δ = 0.67 mm/s and ΔE_Q = 3.00 mm/s), 71 % **1 (**δ = 0.57 mm/s and ΔE_Q = 2.33 mm/s), and 22 % **1-THF (**δ = 0.50 mm/s and ΔE_Q = 2.70 mm/s).

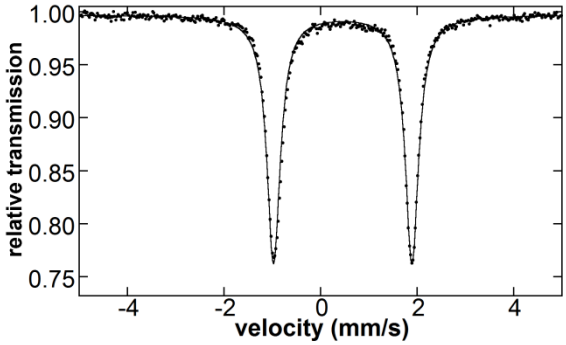


Figure S13. 5 K ^{57}Fe Mössbauer spectrum of *in situ* formed iron species in THF during the reported pre-catalyst formation step where 12 equiv (2-(1,3-dioxan-2-yl)ethyl)magnesium bromide was added to 1.0 equiv Fe(OAc)$_2$ and 2.1 equiv SIPr•HCl in THF at 54 °C and stirred for 20 min. Data (black dots), total fit (black line), and fit components are shown. The black component represents **4** with Mössbauer parameters of δ = 0.44 mm/s and ΔE_Q = 2.91 mm/s.

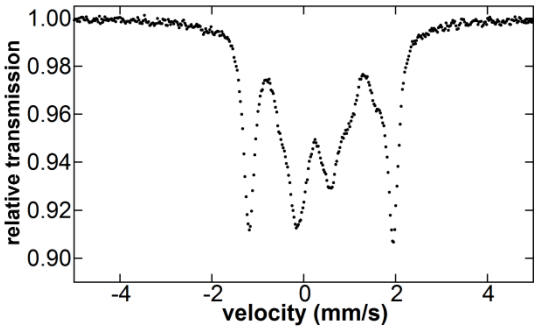


Figure S14. 80 K ^{57}Fe Mössbauer spectrum of *in situ* iron speciation formed 20 m after the addition of 12 equiv (2-cyclohexylethyl)magnesium bromide to 1.0 equiv ^{57}Fe(OAc)$_2$ and 2.1 equiv of IMes•HCl in THF at 54 °C. The iron distribution is complex due to the presence of a larger number of iron species compared to using (2-(1,3-dioxan-2-yl)ethyl)magnesium bromide which generates **1** and **1-THF** exclusively. Note that the data was not fit due to the spectral complexity.

2.3 NMR

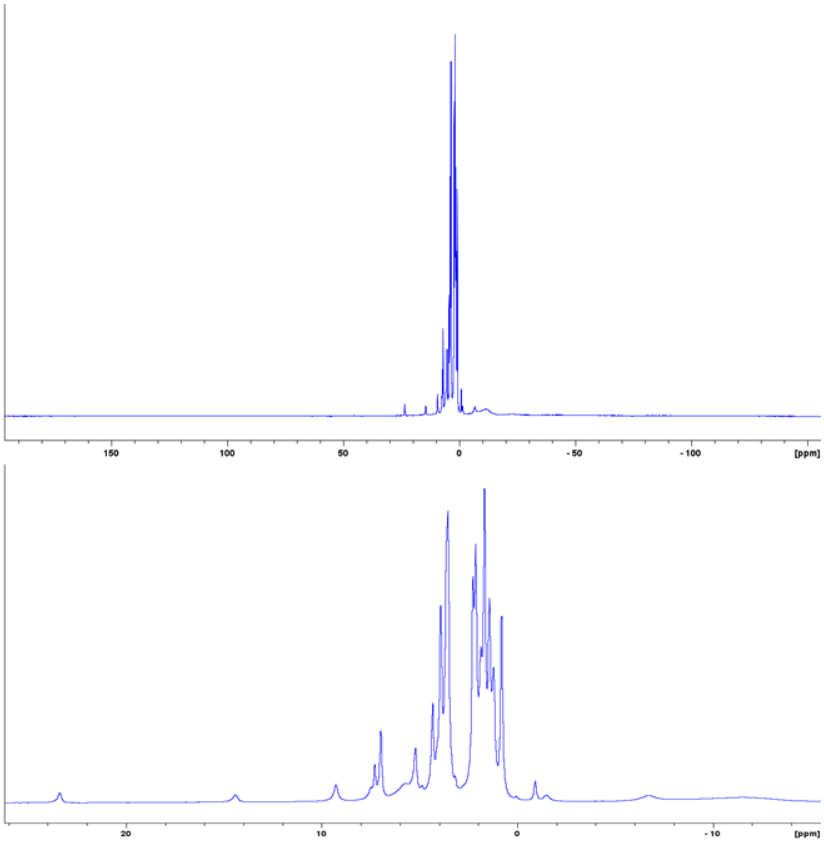


Figure S15. Full paramagnetic ^{1}H NMR (400 MHz) spectrum of isolated, crystalline (IMes)Fe((1,3-dioxan-2-yl)ethyl)$_2$ in THF-d_8 (top), and selected region (bottom).

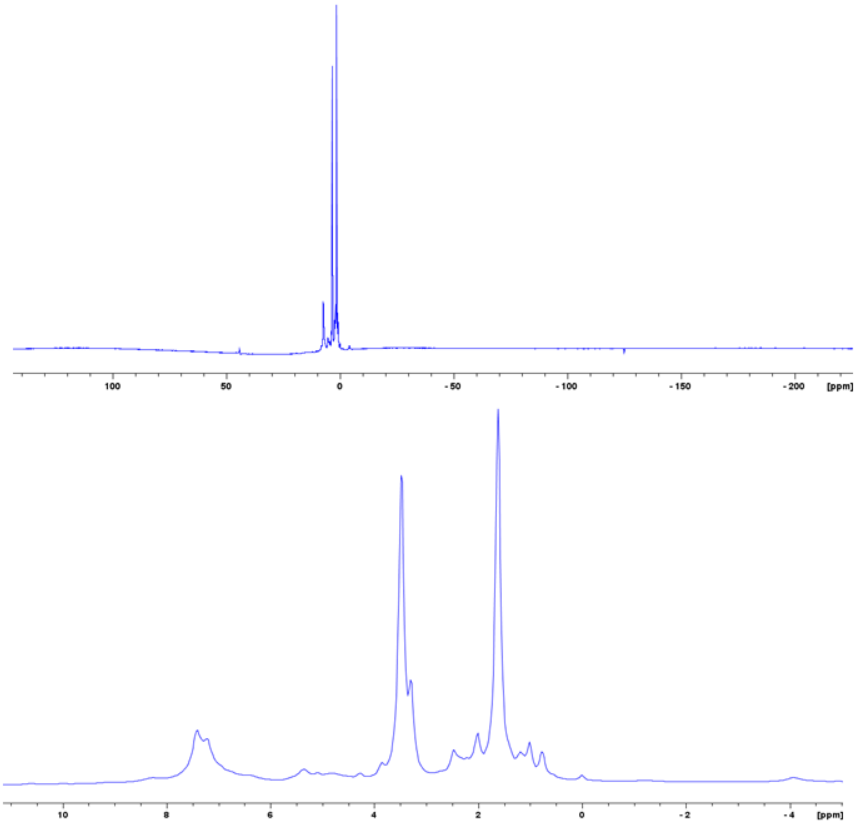


Figure S16. Full paramagnetic ^{1}H NMR (400 MHz) spectrum of isolated crystalline material of co-crystallized (IMes)FeBr((1,3-dioxan-2-yl)ethyl) and (IMes)$FeBr_2$ in THF-d_8 (top), and selected region (bottom).

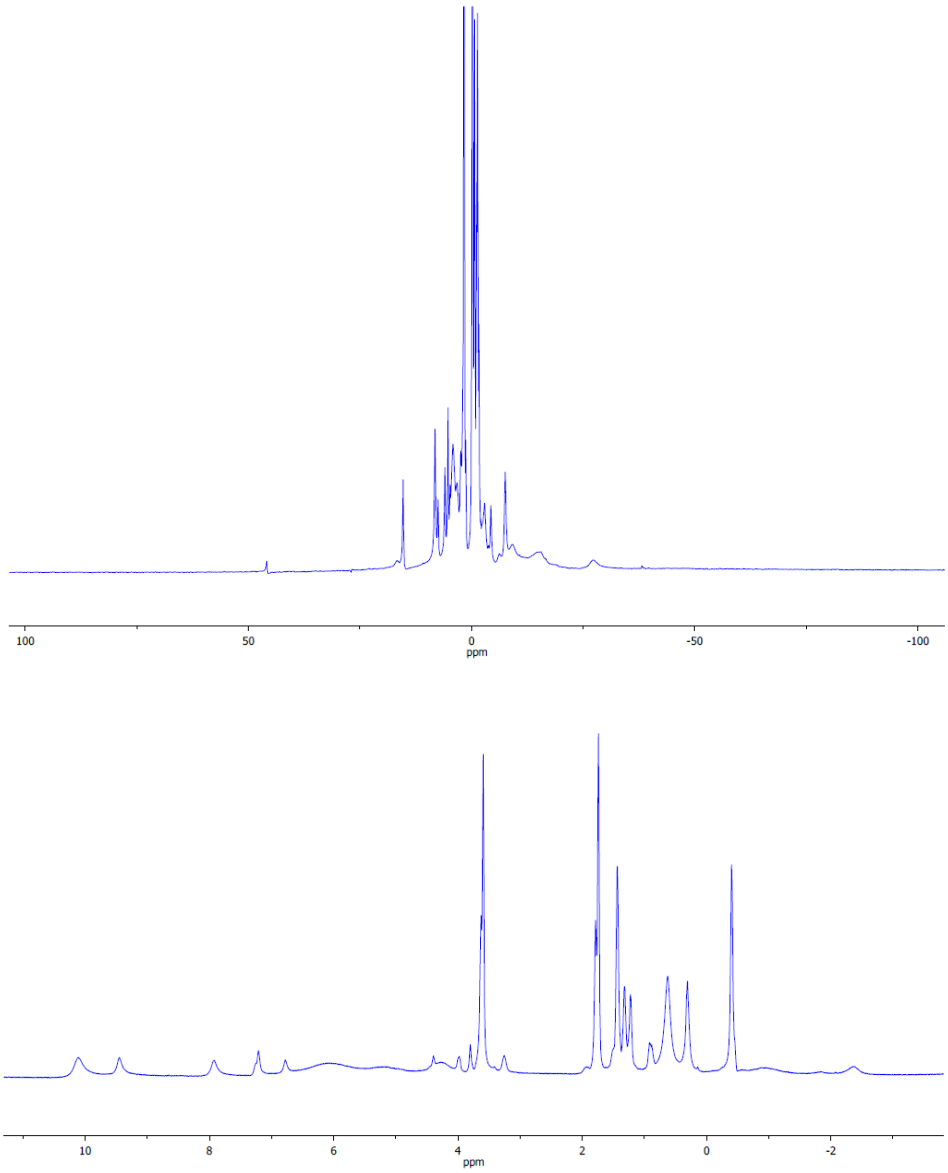


Figure S17. Full paramagnetic ^{1}H NMR (400 MHz) spectrum of isolated crystalline material of (SIPr)Fe((1,3-dioxan-2-yl)ethyl)$_2$ in THF-d_8 (top), and selected region (bottom).

3. Reaction/GC Data

Table S1. Stoichiometric reactivity of **1** and **2-Br** with 1-iodo-3-phenylpropane at 23 °C in THF.

			% yield with respect to iron			
Catalyst	**x equiv R – I**	**Time (s)**	**cross-coupled product**	**allylbenzene**	**1-phenylpropene**	**1-propylbenzene**
1 (in situ)	40	60	360	320	0	0
1 (isolated)	20	5 60	160 160	20 20	0 0	20 20
1 (isolated)	0.7	5 60	42 42	0 0	11 11	17 17
2-Br	20	300	0	0	0	0
***2-Br** + 1.1 equiv RMgBr	20	12 60	170 170	35 35	0 0	0 0

* **2-Br** first reacted with 1.1 equiv (2-(1,3-dioxan-2-yl)ethyl)magnesium bromide at 23 ^{0}C for 1 m before addition of 1-iodo-3-phenylpropane.

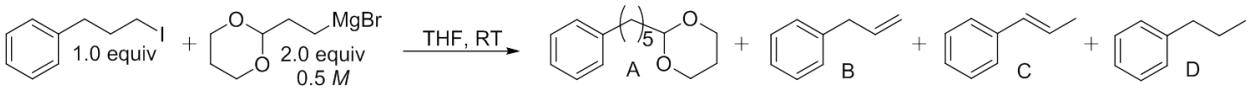
Table S2. Time monitoring of alkyl-alkyl cross-coupling reaction with $Fe(OAc)_2$, IMes•HCl, 1-iodo-3-phenylpropane, and slow addition of (2-(1,3-dioxan-2-yl)ethyl)magnesium bromide at 23 °C in THF. All time points were recorded defining t = 0 min as the point of addition of electrophile.

		% yield with respect to electrophile				
time	**equiv RMgBr added**	**cross-coupled product**	**allylbenzene**	**n-propylbenzene**	**1-phenylpropene**	**TON**
1 m	0.13	9	8	0	0	8
5 m	0.67	9	8	0	0	8
20 m	2.67	13	9	0	0	9
45 m	6	14	9	0	0	10
1 h 15 m	10	34	12	7	0	18
2 h	16	55	14	8	0	29

Table S3. Comparison of alkyl-alkyl cross-coupling reaction yields using the reported procedure by Cárdenas, **2-Br** as a pre-catalyst, and (2-cyclohexylethyl)magnesium bromide as a nucleophile in the reported catalytic reaction.

	% yield with respect to electrophile				
	cross-coupled product	allylbenzene	n-propylbenzene	1-phenylpropene	TON
Reported Reaction	75 (75)	9 (12)	7 (9)	9 (1)	40
2-Br precatalyst	78	4	6	12	40
(2-cyclohexylethyl)magnesium bromide	9	0	62	29	40

Table S4. GC analysis of 1-iodo-3-phenylpropane with (2-(1,3-dioxan-2-yl)ethyl)magnesium bromide at 23 °C in THF using dodecane as an internal standard.



		% Yield			
Time (s)	% Remaining R – I	A	B	C	D
5	100	0	0	0	0
300	99	1	0	0	0

References

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4. Supplementary DFT Analysis

4.1 Calculated MO Energy Level Diagrams

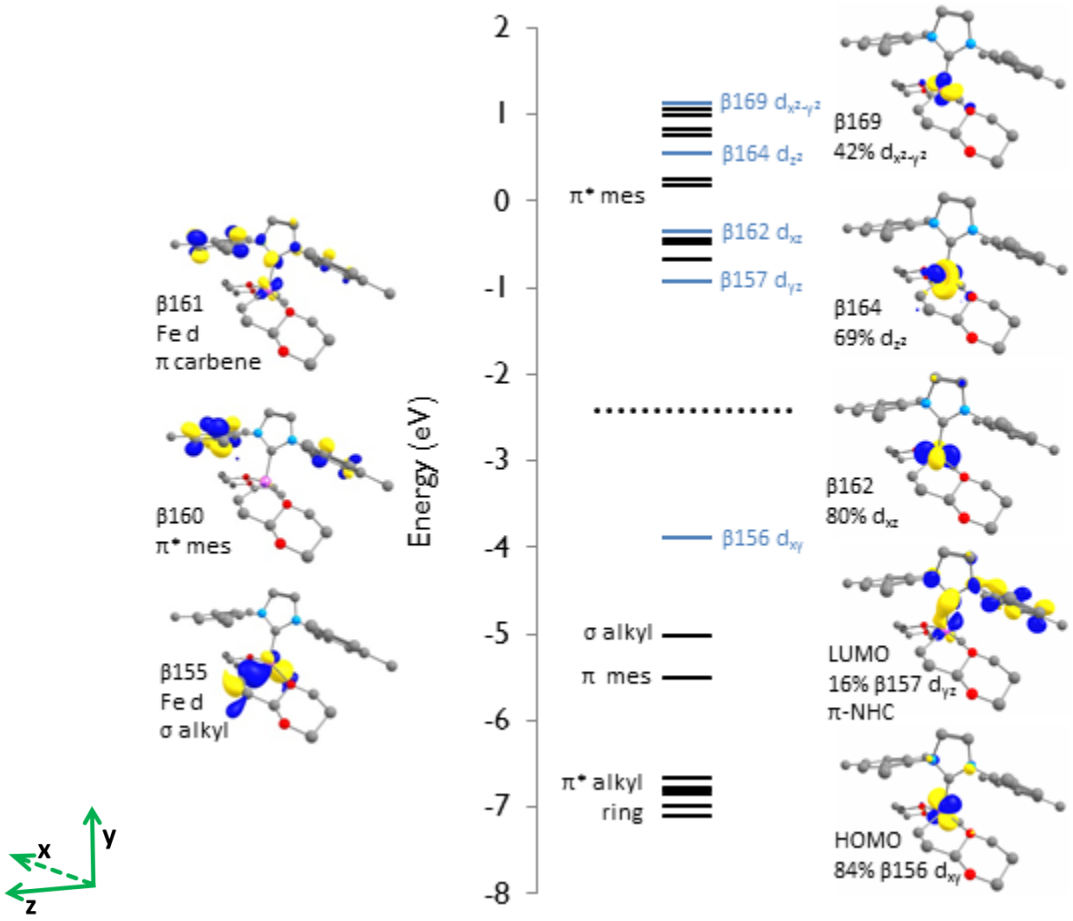


Figure S18. Calculated FMO energy level diagram for (IMes)Fe((1,3-dioxan-2-yl)ethyl)$_2$ (**1**) from gas phase optimized geometric coordinates. Select orbital depictions are given.

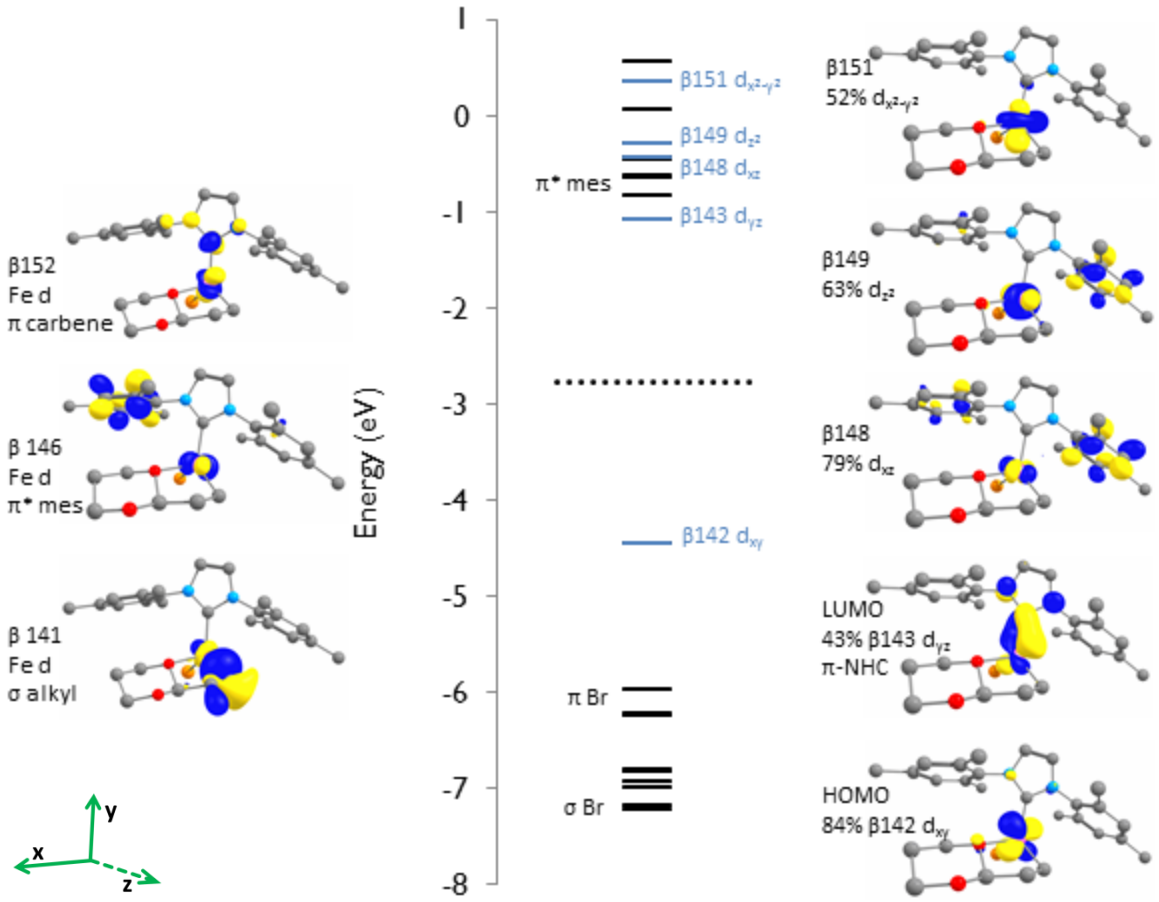


Figure S19. Calculated FMO energy level diagram for (IMes)FeBr((1,3-dioxan-2-yl)ethyl) (**2-Br**) from gas phase optimized geometric coordinates. Select orbital depictions are given.

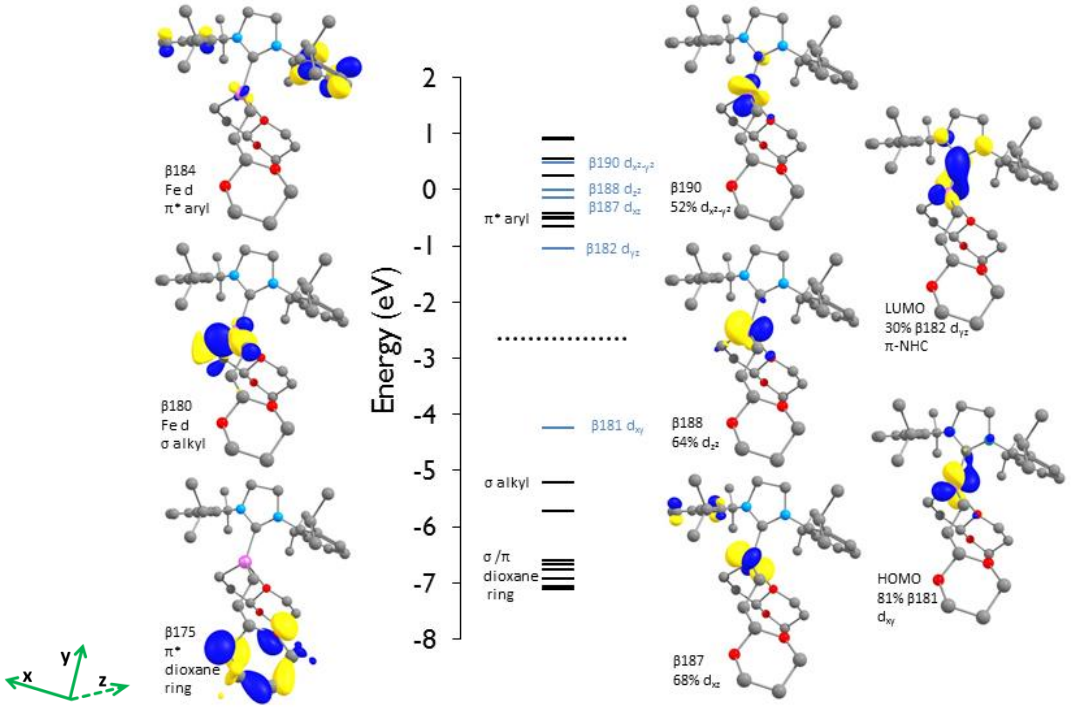


Figure S20. Calculated FMO energy level diagram for (SIPr)Fe((1,3-dioxan-2-yl)ethyl)$_2$ (**4**) from gas phase optimized geometric coordinates. Select orbital depictions are given. Analogous calculations to **1** and **2-Br** were performed on **4**. Geometry optimizations are in good agreement with crystallographic data and coordinates can be found *vide infra*. Additionally, molecular orbital analysis results in an orbital trend similar to the other two complexes studied. Dominant Fe d orbital character is found in β181 (HOMO, 81% d_{xy}), β187 (LUMO+5, 68% d_{xz}), β188 (LUMO+6, 64% d_{z2}) and β190 (LUMO+8, 52% d_{x2-y2}). β182 (LUMO) shows 30% d_{yz} character with a π interaction with the carbene carbon of IPr, similar to those found in **1** and **2-Br**. Ligand based unoccupied orbitals between LUMO and LUMO+5 are composed largely of diffuse iron d character and π* orbitals on the NHC aryl rings (e.g. β184). The highest occupied ligand based orbital (HOMO-1) shows strong σ overlap with the alkyl carbon of the bis-chelated alkyl group, remaining consistent with the other molecules studies here. When the Mayer bond order analysis was carried out for **4** in solution and gas phases, it was found that the coordinated O-Fe interaction has a bond order of 0.074 (gas phase, see Table 1). This is similar to the results found in **1** where this Fe-O coordination is very weak.

4.2 Optimized Geometry Coordinates

Table S5. Comparison of Select Calculated and Experimental Bonding Parameters for (NHC)-Iron(II)-Alkyl Complexes. Experimental Mayer bond orders calculated from crystallographic coordinates.

		bond lengths (Å) and angles (°)			Mayer bond order		
		exptl	gas phase calcd	solution calcd	exptl	gas phase calcd	solution calcd
1	**Fe-C13**	2.1222(12)	2.00291	2.01152	0.761	0.969	0.767
	Fe-C1	2.0994(13)	2.08277	2.08771	0.816	0.840	0.803
	Fe-C7	2.1062(12)	2.08488	2.09046	0.804	0.784	0.788
	Fe-O1	2.4632(9)	2.41927	2.42506	0.042	0.071	0.042
	Fe-O3	2.4428(9)	2.39385	2.40521	0.046	0.099	0.047
2-Br	**Fe-C7**	2.096(4)	2.01088	2.01929	0.890	0.885	0.906
	Fe-Br1	2.4184(8)	2.41058	2.4544	0.709	0.704	0.711
	Fe-C1	2.016(12)	2.0522	2.05861	0.910	0.911	0.865
	Fe-O1	2.181(4)	2.24683	2.2555	0.224	0.224	0.176
4	**Fe-C13**	2.0920(10)	2.02529	1.99340	0.918	1.053	1.032
	Fe-C1	2.0885(12)	2.07123	2.07261	0.800	0.789	0.801
	Fe-C7	2.0707(12)	2.06514	2.05259	0.789	0.738	0.793
	Fe-O1	2.3130(9)	2.32068	2.24317	0.063	0.047	0.074

4.2.1 (IMes)Fe((1,3-Dioxan-2-yl)ethyl)$_2$ (1) optimized with uPBEPBE/TZVP (gas phase, EmpiricalDispersion=ED2)

26	-0.600706000	7.829864000	9.815452000
8	-2.431974000	8.818055000	10.998861000
8	-3.584930000	10.863114000	11.023503000
8	0.686727000	7.069954000	7.913375000
8	0.488849000	5.463144000	6.218318000
7	0.408932000	6.544408000	12.395085000
7	2.068516000	7.291087000	11.245544000
6	-0.613732000	9.752237000	9.008591000
1	0.405890000	10.155349000	8.878026000
1	-1.062028000	9.682017000	7.998126000
6	-1.457312000	10.690987000	9.881444000
1	-0.939007000	10.941074000	10.823925000
1	-1.720476000	11.651171000	9.397903000
6	-2.756314000	10.011770000	10.255244000
1	-3.312421000	9.687508000	9.344985000
6	-3.624185000	8.045170000	11.237407000
1	-3.312550000	7.142255000	11.773303000
1	-4.055320000	7.747610000	10.261680000
6	-4.615507000	8.889007000	12.034657000
1	-5.572457000	8.351458000	12.144042000
1	-4.203127000	9.075659000	13.038158000
6	-4.836864000	10.216832000	11.306314000
1	-5.374845000	10.032709000	10.352659000
1	-5.421813000	10.926152000	11.908311000
6	-1.853291000	6.368552000	9.019506000
1	-2.356742000	5.779645000	9.805105000
1	-2.647737000	6.886768000	8.446964000
6	-1.061335000	5.447694000	8.081497000
1	-0.368542000	4.795164000	8.642408000
1	-1.692276000	4.783164000	7.460495000
6	-0.225480000	6.277203000	7.130948000
1	-0.867228000	6.986329000	6.558152000
6	1.378133000	8.008762000	7.067988000
1	2.022085000	8.605648000	7.722936000
1	0.632929000	8.678810000	6.596498000
6	2.161418000	7.238501000	6.008866000
1	2.963877000	6.667494000	6.499855000
1	2.621214000	7.936786000	5.289671000
6	1.210017000	6.282994000	5.283424000
1	0.484905000	6.867057000	4.678455000
1	1.746683000	5.592700000	4.617638000
6	0.683292000	7.249711000	11.238976000
6	1.562731000	6.160921000	13.073141000
1	1.525120000	5.605537000	14.003628000
6	2.610751000	6.636433000	12.349457000
1	3.680710000	6.584814000	12.516993000
6	-0.905727000	6.352749000	12.930265000
6	-1.585482000	5.154285000	12.666444000

6	-2.837519000	4.966588000	13.267513000
1	-3.385364000	4.041702000	13.066473000
6	-3.397499000	5.927615000	14.119918000
6	-2.684187000	7.110600000	14.356469000
1	-3.110815000	7.874530000	15.012631000
6	-1.442597000	7.353994000	13.758498000
6	-0.983201000	4.121453000	11.754688000
1	-1.685702000	3.294032000	11.584531000
1	-0.052469000	3.708596000	12.176627000
1	-0.727102000	4.575565000	10.785384000
6	-4.759941000	5.720719000	14.729452000
1	-4.811896000	6.143728000	15.743967000
1	-5.016983000	4.652739000	14.782614000
1	-5.537065000	6.220542000	14.126014000
6	-0.723140000	8.659467000	13.947571000
1	0.337187000	8.506985000	14.201756000
1	-1.198546000	9.257404000	14.737452000
1	-0.758504000	9.228393000	13.004120000
6	2.872251000	7.967162000	10.274643000
6	2.888412000	9.372851000	10.268544000
6	3.712443000	10.014073000	9.334759000
1	3.723345000	11.107232000	9.308403000
6	4.528289000	9.294868000	8.451368000
6	4.485180000	7.895120000	8.493700000
1	5.092851000	7.317828000	7.791051000
6	3.650531000	7.207718000	9.382673000
6	2.048007000	10.155606000	11.237686000
1	2.286869000	9.879878000	12.277673000
1	0.980960000	9.929086000	11.080699000
1	2.209272000	11.234917000	11.110629000
6	5.439706000	10.008267000	7.486207000
1	5.051453000	11.006262000	7.234675000
1	5.560470000	9.435676000	6.554112000
1	6.444631000	10.143806000	7.920742000
6	3.518862000	5.710781000	9.320015000
1	4.248575000	5.286779000	8.615859000
1	2.504560000	5.451702000	8.973921000
1	3.662044000	5.236065000	10.302553000

Energy: -2959.206748 A.U.

4.2.2 (IMes)Fe((1,3-Dioxan-2-yl)ethyl)$_2$ (1) optimized with uPBEPBE/TZVP (THF PCM, EmpiricalDispersion=ED2)

26	-0.600500000	7.824521000	9.808479000
8	-2.433196000	8.824717000	11.002586000
8	-3.580472000	10.872449000	11.035744000
8	0.681683000	7.056761000	7.898643000
8	0.477647000	5.456820000	6.196848000
7	0.411692000	6.557183000	12.404299000
7	2.070041000	7.302559000	11.254987000
6	-0.610881000	9.757469000	9.012471000
1	0.415751000	10.148647000	8.900130000
1	-1.047868000	9.703859000	7.995758000
6	-1.453834000	10.694025000	9.888945000
1	-0.931293000	10.936078000	10.831498000
1	-1.715565000	11.658320000	9.410720000
6	-2.754842000	10.016035000	10.262565000
1	-3.317034000	9.700514000	9.353737000
6	-3.628755000	8.053005000	11.245337000
1	-3.314841000	7.150236000	11.779801000
1	-4.063818000	7.759564000	10.270555000
6	-4.614606000	8.896747000	12.047429000
1	-5.571942000	8.361656000	12.157791000
1	-4.200947000	9.078702000	13.051298000
6	-4.838183000	10.225082000	11.323768000
1	-5.376503000	10.046845000	10.370796000
1	-5.418602000	10.933583000	11.930256000
6	-1.855960000	6.359174000	9.011530000
1	-2.345828000	5.769431000	9.805319000
1	-2.659129000	6.874937000	8.448586000
6	-1.069574000	5.441538000	8.065099000
1	-0.378177000	4.786054000	8.624761000
1	-1.703402000	4.780176000	7.442161000
6	-0.232693000	6.273518000	7.115904000
1	-0.872087000	6.983398000	6.542556000
6	1.381370000	7.995401000	7.055763000
1	2.027561000	8.584519000	7.715320000
1	0.640014000	8.669252000	6.584404000
6	2.162308000	7.225059000	5.996438000
1	2.961645000	6.647858000	6.485287000
1	2.626316000	7.923078000	5.280652000
6	1.208135000	6.280628000	5.263228000
1	0.485000000	6.869308000	4.662805000
1	1.742351000	5.590954000	4.595584000
6	0.687262000	7.250466000	11.243177000
6	1.564812000	6.192082000	13.094076000
1	1.527797000	5.645230000	14.029682000
6	2.612923000	6.667079000	12.369948000
1	3.682327000	6.623610000	12.543702000
6	-0.904956000	6.355696000	12.933143000
6	-1.580788000	5.157355000	12.657673000

6	-2.837336000	4.963989000	13.248264000
1	-3.382391000	4.039660000	13.037476000
6	-3.405411000	5.919211000	14.103333000
6	-2.692947000	7.099837000	14.356636000
1	-3.124926000	7.858103000	15.015596000
6	-1.446658000	7.347705000	13.769086000
6	-0.967868000	4.125477000	11.751458000
1	-1.668832000	3.298577000	11.573007000
1	-0.043347000	3.712080000	12.186235000
1	-0.698019000	4.579688000	10.785846000
6	-4.775116000	5.708629000	14.694456000
1	-4.857123000	6.173828000	15.687954000
1	-5.012129000	4.638510000	14.784795000
1	-5.547545000	6.166446000	14.052968000
6	-0.724340000	8.648493000	13.981533000
1	0.325567000	8.488202000	14.272159000
1	-1.221751000	9.249632000	14.755046000
1	-0.720268000	9.218195000	13.037754000
6	2.872614000	7.973435000	10.278030000
6	2.892122000	9.378806000	10.266536000
6	3.712269000	10.015418000	9.325234000
1	3.726024000	11.108454000	9.294193000
6	4.520843000	9.290384000	8.438759000
6	4.477538000	7.890281000	8.489448000
1	5.080140000	7.308853000	7.786159000
6	3.648200000	7.208742000	9.388438000
6	2.061573000	10.167362000	11.240387000
1	2.311672000	9.897757000	12.279348000
1	0.993089000	9.939806000	11.097361000
1	2.222079000	11.245800000	11.105185000
6	5.423589000	9.997293000	7.460834000
1	5.037432000	10.996914000	7.213287000
1	5.530458000	9.420700000	6.529753000
1	6.434156000	10.127996000	7.883406000
6	3.528237000	5.709993000	9.348275000
1	4.230410000	5.285272000	8.617453000
1	2.502588000	5.434560000	9.052928000
1	3.721788000	5.251376000	10.329958000

Energy: -2959.221098 A.U.

4.2.3 (IMes)FeBr((1,3-Dioxan-2-yl)ethyl) (2-Br) optimized with uPBEPBE/TZVP (gas phase, EmpiricalDispersion=ED2)

26	5.039310000	9.603011000	7.916717000
35	7.367142000	8.923592000	7.977534000
8	4.926100000	10.849596000	9.827372000
8	4.378900000	13.053735000	10.435881000
6	4.515210000	11.397168000	7.064021000
1	5.433407000	11.812805000	6.611573000
1	3.771753000	11.293599000	6.257337000
6	3.992824000	12.317173000	8.174130000
1	3.941131000	13.382300000	7.880402000
1	2.975164000	12.030872000	8.490540000
6	4.879788000	12.240555000	9.400117000
1	5.926581000	12.538318000	9.167454000
6	5.844673000	10.689169000	10.935912000
1	6.865167000	10.934602000	10.588253000
1	5.820446000	9.626698000	11.204991000
6	5.415416000	11.592590000	12.088516000
1	4.460727000	11.232594000	12.502109000
1	6.169905000	11.556796000	12.891252000
6	5.249655000	13.023110000	11.579558000
1	4.790180000	13.679041000	12.331167000
1	6.235603000	13.448316000	11.302050000
7	2.983265000	7.529519000	7.095397000
7	3.479614000	7.160787000	9.159690000
6	3.768725000	8.016379000	8.118983000
6	2.238575000	6.422444000	7.487885000
1	1.567007000	5.907391000	6.810402000
6	2.555769000	6.186016000	8.790085000
1	2.216032000	5.423562000	9.481880000
6	2.967141000	8.081026000	5.763875000
6	1.960445000	9.002527000	5.424094000
6	1.952914000	9.510638000	4.120238000
1	1.179312000	10.231572000	3.841399000
6	2.911485000	9.129768000	3.172118000
6	3.893001000	8.206569000	3.552112000
1	4.646933000	7.896281000	2.823514000
6	3.945627000	7.664317000	4.842846000
6	0.943210000	9.458066000	6.437305000
1	1.435526000	9.944716000	7.293864000
1	0.355537000	8.618779000	6.839716000
1	0.245525000	10.177458000	5.989336000
6	2.905926000	9.726469000	1.787857000
1	3.492172000	10.659507000	1.762420000
1	1.886225000	9.972180000	1.458895000
1	3.349697000	9.041323000	1.051993000
6	5.025653000	6.687819000	5.226009000
1	4.612425000	5.788466000	5.707036000
1	5.733338000	7.139928000	5.940569000
1	5.593655000	6.373959000	4.340529000

6	4.020990000	7.252541000	10.492015000
6	5.196374000	6.543140000	10.802496000
6	5.666302000	6.611457000	12.120971000
1	6.582378000	6.072084000	12.377062000
6	4.997786000	7.339696000	13.113208000
6	3.831582000	8.029986000	12.759164000
1	3.298759000	8.608575000	13.519172000
6	3.322733000	8.005334000	11.453966000
6	5.947942000	5.765155000	9.757551000
1	5.279188000	5.132910000	9.155387000
1	6.705212000	5.122029000	10.224727000
1	6.462219000	6.454051000	9.065891000
6	5.504555000	7.353059000	14.533420000
1	5.168692000	8.249735000	15.072947000
1	6.602852000	7.320929000	14.569121000
1	5.134901000	6.477135000	15.091214000
6	2.092970000	8.796807000	11.094835000
1	2.368272000	9.678236000	10.494013000
1	1.580191000	9.148364000	11.999970000
1	1.379596000	8.206871000	10.501205000

Energy: -5147.963211

4.2.4 (IMes)FeBr(1,3-Dioxan-2-ylethyl) (2-Br) optimized with uPBEPBE/TZVP (THF PCM, EmpiricalDispersion=ED2)

26	5.122461000	9.455876000	7.861609000
35	7.511705000	8.920287000	8.031196000
8	4.844983000	10.629446000	9.767652000
8	4.187940000	12.791032000	10.411767000
6	4.551221000	11.240252000	7.008637000
1	5.453912000	11.739565000	6.611552000
1	3.849626000	11.083827000	6.173276000
6	3.914130000	12.089724000	8.117790000
1	3.802510000	13.157629000	7.851712000
1	2.908760000	11.716208000	8.379514000
6	4.758302000	12.026949000	9.374624000
1	5.799481000	12.371120000	9.186252000
6	5.739450000	10.473551000	10.899083000
1	6.753261000	10.786236000	10.588780000
1	5.752299000	9.403711000	11.136565000
6	5.231194000	11.321973000	12.059276000
1	4.276568000	10.910900000	12.420757000
1	5.956671000	11.291824000	12.887984000
6	5.032534000	12.759782000	11.584806000
1	4.531648000	13.379423000	12.340248000
1	6.010073000	13.217894000	11.334929000
7	2.935276000	7.532708000	7.121130000
7	3.431764000	7.191441000	9.182784000
6	3.795395000	7.953156000	8.102984000
6	2.062738000	6.551306000	7.573851000
1	1.312224000	6.103461000	6.932370000
6	2.382049000	6.329943000	8.880471000
1	1.970312000	5.645422000	9.613626000
6	2.956065000	8.086037000	5.796749000
6	2.040030000	9.103331000	5.479221000
6	2.087689000	9.645935000	4.191470000
1	1.385858000	10.442758000	3.929496000
6	3.023735000	9.207410000	3.241762000
6	3.927836000	8.202218000	3.606559000
1	4.667936000	7.857052000	2.879584000
6	3.917062000	7.625015000	4.884492000
6	1.082784000	9.624698000	6.516336000
1	1.638912000	10.001484000	7.390633000
1	0.406825000	8.835877000	6.882242000
1	0.475983000	10.443333000	6.106891000
6	3.059256000	9.821152000	1.865904000
1	3.306368000	10.893646000	1.921281000
1	2.078773000	9.740206000	1.370294000
1	3.809736000	9.329890000	1.230649000
6	4.918750000	6.571801000	5.273885000
1	4.431972000	5.713361000	5.761941000
1	5.647778000	6.978890000	5.995212000
1	5.467497000	6.214878000	4.391668000

6	4.023140000	7.310614000	10.484982000
6	5.221820000	6.631631000	10.752993000
6	5.770101000	6.765217000	12.036590000
1	6.710921000	6.255789000	12.261600000
6	5.143459000	7.525103000	13.033296000
6	3.942286000	8.178544000	12.722470000
1	3.449005000	8.789011000	13.483648000
6	3.369627000	8.098625000	11.448074000
6	5.899270000	5.806891000	9.694023000
1	5.194068000	5.102930000	9.225229000
1	6.735594000	5.238678000	10.123492000
1	6.293877000	6.458872000	8.897254000
6	5.738715000	7.631227000	14.413378000
1	5.617870000	8.646393000	14.820856000
1	6.809279000	7.381139000	14.404806000
1	5.238522000	6.937797000	15.109983000
6	2.138339000	8.886454000	11.094760000
1	2.402991000	9.673345000	10.369222000
1	1.713140000	9.363781000	11.988156000
1	1.365956000	8.255536000	10.628961000

Energy: -5147.963026

4.2.5 (SIPr)Fe((1,3-Dioxan-2-yl)ethyl)$_2$ (4) optimized with uPBEPBE/TZVP (gas phase, EmpiricalDispersion=ED2)

26	11.163540000	7.655434000	12.128136000
8	12.241777000	8.316379000	14.073924000
8	14.174021000	9.515110000	14.681574000
8	12.856681000	3.724891000	14.307633000
8	14.182860000	3.137824000	12.449471000
7	8.724142000	8.816259000	10.794719000
7	8.175884000	8.041885000	12.770604000
6	12.799978000	8.612977000	11.294375000
1	13.493911000	7.835744000	10.923399000
1	12.534003000	9.239881000	10.426946000
6	13.487049000	9.460041000	12.373655000
1	14.513183000	9.776073000	12.106595000
1	12.921639000	10.384377000	12.581347000
6	13.587965000	8.707262000	13.684508000
1	14.179344000	7.770298000	13.578395000
6	12.259972000	7.525368000	15.286862000
1	12.780974000	6.570614000	15.087650000
1	11.211046000	7.306675000	15.523263000
6	12.951066000	8.303358000	16.403805000
1	13.070457000	7.656402000	17.288190000
1	12.334123000	9.167685000	16.693565000
6	14.313496000	8.788212000	15.913491000
1	14.787622000	9.478397000	16.624391000
1	14.993447000	7.926018000	15.756660000

6	11.339302000	5.638510000	12.535494000
1	11.054521000	5.525693000	13.596867000
1	10.580976000	5.089496000	11.946391000
6	12.728357000	5.042879000	12.306546000
1	12.956701000	4.975462000	11.230783000
1	13.521927000	5.671105000	12.746421000
6	12.923038000	3.647985000	12.882428000
1	12.123593000	2.949776000	12.537607000
6	13.000076000	2.429355000	14.899158000
1	12.149133000	1.784595000	14.593026000
1	12.943334000	2.575705000	15.987000000
6	14.320165000	1.786640000	14.473587000
1	15.160736000	2.357867000	14.897012000
1	14.384085000	0.750866000	14.845682000
6	14.401406000	1.812837000	12.947214000
1	15.390000000	1.502714000	12.580987000
1	13.642825000	1.124190000	12.517815000
6	9.249775000	8.293477000	11.948721000
6	7.247765000	8.787858000	10.750033000
1	6.902033000	7.975666000	10.085908000
1	6.845912000	9.737649000	10.367550000
6	6.890623000	8.528666000	12.214510000
1	6.578199000	9.449510000	12.737150000
1	6.096036000	7.779315000	12.333677000
6	9.462319000	9.144013000	9.608210000
6	9.800472000	8.131864000	8.680104000
6	10.482813000	8.507493000	7.514176000
1	10.761681000	7.740768000	6.787907000
6	10.810547000	9.838142000	7.264885000
1	11.345519000	10.107569000	6.351663000
6	10.457828000	10.824294000	8.184067000
1	10.720013000	11.865452000	7.982227000
6	9.774829000	10.502134000	9.364472000
6	9.438757000	6.668164000	8.894743000
1	9.015223000	6.572891000	9.906114000
6	10.677560000	5.758758000	8.833857000
1	11.442419000	6.083145000	9.553952000
1	11.129720000	5.759270000	7.830324000
1	10.401808000	4.720907000	9.074554000
6	8.372480000	6.205134000	7.883980000
1	7.469355000	6.832433000	7.925150000
1	8.076807000	5.164172000	8.086175000
1	8.759528000	6.249557000	6.854291000
6	9.403201000	11.605356000	10.345141000
1	8.856042000	11.132839000	11.174701000
6	10.654268000	12.268942000	10.944677000
1	11.294484000	11.525769000	11.439547000
1	10.368036000	13.029577000	11.687554000
1	11.253260000	12.768012000	10.167658000
6	8.474513000	12.651585000	9.704305000
1	8.163536000	13.397669000	10.451520000

1	7.568531000	12.188295000	9.285405000
1	8.978532000	13.190946000	8.887715000
6	8.272081000	7.794568000	14.182486000
6	8.571846000	8.865250000	15.061982000
6	8.632987000	8.594113000	16.436491000
1	8.869646000	9.404695000	17.129682000
6	8.382632000	7.316095000	16.933250000
1	8.436219000	7.126225000	18.007598000
6	8.051088000	6.283752000	16.057835000
1	7.839448000	5.289414000	16.456206000
6	7.978211000	6.499351000	14.674025000
6	8.754056000	10.298942000	14.577709000
1	8.788966000	10.277370000	13.478997000
6	10.070966000	10.931404000	15.053968000
1	10.936668000	10.338648000	14.731190000
1	10.100461000	11.023003000	16.151166000
1	10.173599000	11.946176000	14.639751000
6	7.553834000	11.167739000	15.004772000
1	6.596217000	10.742664000	14.668494000
1	7.645353000	12.182112000	14.586776000
1	7.505327000	11.260124000	16.100829000
6	7.534544000	5.369437000	13.754134000
1	7.776473000	5.677320000	12.725121000
6	8.256612000	4.041040000	14.030035000
1	9.347585000	4.156222000	13.983911000
1	7.960846000	3.291258000	13.280300000
1	7.991323000	3.633746000	15.017767000
6	6.008466000	5.165514000	13.851478000
1	5.451554000	6.094130000	13.658086000
1	5.727521000	4.817910000	14.857977000
1	5.672843000	4.406890000	13.127656000

Energy: -3194.245850

4.2.6 (SIPr)Fe((1,3-Dioxan-2-yl)ethyl)$_2$ (4) optimized with uPBEPBE/TZVP (THF PCM, EmpiricalDispersion=ED2)

26	11.099783000	7.700809000	11.921059000
8	12.075861000	8.335851000	13.838299000
8	13.946032000	9.549146000	14.574450000
8	12.507633000	3.933932000	14.394887000
8	14.144808000	3.289645000	12.827258000
7	8.615822000	8.920030000	10.801120000
7	8.195808000	8.024765000	12.752125000
6	12.733092000	8.687961000	11.112628000
1	13.455524000	7.932707000	10.748173000
1	12.457887000	9.327571000	10.256534000
6	13.371760000	9.533068000	12.227516000
1	14.403284000	9.868992000	12.006361000
1	12.771812000	10.438262000	12.422277000
6	13.429709000	8.754647000	13.527362000
1	14.042838000	7.830583000	13.433783000
6	12.042353000	7.499655000	15.022484000
1	12.600159000	6.568736000	14.814585000
1	10.988087000	7.251942000	15.188698000
6	12.650670000	8.259696000	16.195441000
1	12.738489000	7.589555000	17.065702000
1	11.996349000	9.101288000	16.469009000
6	14.029584000	8.780854000	15.795571000
1	14.454546000	9.452875000	16.553098000
1	14.724266000	7.931497000	15.637973000
6	11.270045000	5.705940000	12.373408000
1	10.866596000	5.624651000	13.396730000
1	10.607099000	5.110578000	11.719365000
6	12.690104000	5.150529000	12.337905000
1	13.043892000	5.041852000	11.299241000
1	13.403146000	5.827145000	12.841805000
6	12.818979000	3.792533000	13.007128000
1	12.104570000	3.057170000	12.568666000
6	12.542941000	2.655397000	15.051996000
1	11.766853000	1.997764000	14.608490000
1	12.288304000	2.838140000	16.105212000
6	13.919986000	2.012382000	14.895064000
1	14.670368000	2.607858000	15.438301000
1	13.915130000	0.991688000	15.310293000
6	14.269148000	1.977642000	13.407812000
1	15.304724000	1.654705000	13.233371000
1	13.585762000	1.280802000	12.879972000
6	9.203177000	8.312293000	11.869894000
6	7.140736000	8.892665000	10.861096000
1	6.756116000	8.062691000	10.241037000
1	6.709737000	9.838080000	10.502752000
6	6.912548000	8.651842000	12.353760000
1	6.772476000	9.600085000	12.900371000
1	6.057817000	7.996322000	12.561705000

6	9.299709000	9.193704000	9.577031000
6	9.628199000	8.143572000	8.697076000
6	10.278963000	8.471813000	7.497202000
1	10.545300000	7.673278000	6.800996000
6	10.586524000	9.794945000	7.185045000
1	11.092517000	10.030477000	6.246053000
6	10.252999000	10.822352000	8.073286000
1	10.505369000	11.853930000	7.822563000
6	9.604966000	10.540911000	9.281717000
6	9.293009000	6.692999000	8.998126000
1	8.915649000	6.633018000	10.030009000
6	10.542636000	5.802692000	8.917787000
1	11.323103000	6.163773000	9.606031000
1	10.951952000	5.787070000	7.895467000
1	10.288710000	4.769214000	9.200603000
6	8.195816000	6.180994000	8.049910000
1	7.283981000	6.793812000	8.124023000
1	7.937248000	5.138340000	8.293277000
1	8.544479000	6.213128000	7.005171000
6	9.313329000	11.623392000	10.306395000
1	8.378258000	11.351336000	10.823279000
6	10.434301000	11.632974000	11.361868000
1	10.570889000	10.632418000	11.800331000
1	10.202122000	12.347248000	12.167882000
1	11.386156000	11.931782000	10.893889000
6	9.127470000	13.018172000	9.701408000
1	8.822237000	13.725619000	10.487253000
1	8.359309000	13.019584000	8.912885000
1	10.067705000	13.393338000	9.267241000
6	8.455847000	7.723453000	14.128922000
6	8.723724000	8.779431000	15.027794000
6	8.908429000	8.466135000	16.383177000
1	9.111696000	9.272118000	17.091965000
6	8.855659000	7.146085000	16.827546000
1	9.011372000	6.917726000	17.884283000
6	8.622012000	6.111472000	15.917822000
1	8.605132000	5.079482000	16.270764000
6	8.411724000	6.376107000	14.557046000
6	8.801954000	10.231207000	14.583362000
1	8.758214000	10.262850000	13.485080000
6	10.130853000	10.882496000	14.994976000
1	10.982840000	10.325180000	14.582581000
1	10.229540000	10.913392000	16.092002000
1	10.168544000	11.918693000	14.624415000
6	7.616285000	11.034859000	15.145195000
1	6.650089000	10.591700000	14.858243000
1	7.649658000	12.072048000	14.776005000
1	7.661685000	11.061598000	16.245733000
6	8.121440000	5.251829000	13.575575000
1	8.570747000	5.544552000	12.614629000
6	8.723227000	3.902807000	13.990365000

1	9.799060000	3.984518000	14.204282000
1	8.588871000	3.179091000	13.171607000
1	8.212907000	3.493654000	14.877675000
6	6.604578000	5.092189000	13.366768000
1	6.141694000	6.020153000	13.005567000
1	6.117691000	4.811661000	14.314818000
1	6.405365000	4.298050000	12.629701000

Energy: -3194.417544

5. X-ray Crystallographic Data

5.1 (IMes)Fe((1,3-Dioxan-2-yl)ethyl)$_2$ (1)

REFERENCE NUMBER: neism35

CRYSTAL STRUCTURE REPORT

C_{33} H_{46} Fe N_2 O_4

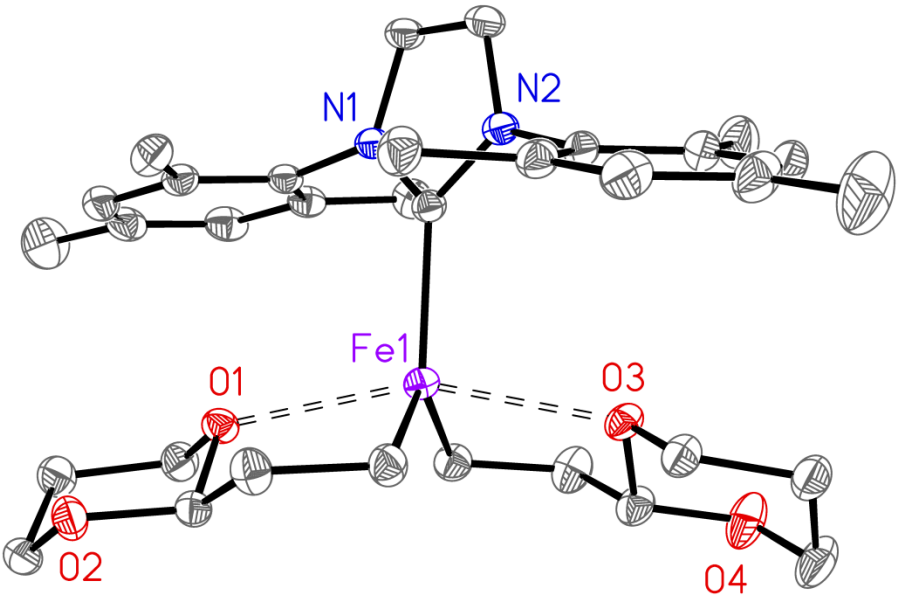
or

(IMes)Fe((1,3-dioxan-2-yl)ethyl)$_2$ · xSolvent

Report prepared for:

Dr. S. Muñoz, Prof. M. Neidig

December 01, 2016



William W. Brennessel

X-ray Crystallographic Facility

Department of Chemistry, University of Rochester

120 Trustee Road

Rochester, NY 14627

Data collection

A crystal (0.40 x 0.35 x 0.32 mm^3) was placed onto the tip of a thin glass optical fiber and mounted on a Bruker SMART APEX II CCD platform diffractometer for a data collection at 100.0(5) K.[1] A preliminary set of cell constants and an orientation matrix were calculated from reflections harvested from three orthogonal wedges of reciprocal space. The full data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 30 seconds and a detector distance of 3.99 cm. A randomly oriented region of reciprocal space was surveyed: seven major sections of frames were collected with 0.50º steps in ω at seven different ϕ settings and a detector position of -38º in 2θ. The intensity data were corrected for absorption.[2] Final cell constants were calculated from the xyz centroids of 3961 strong reflections from the actual data collection after integration.[3] See Table S6 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SHELXT-2014/5[4] and refined using SHELXL-2016/6.[5] The space group *Cc* was determined based on systematic absences and intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters.

Reflection contributions from highly disordered solvent, which appeared to be a mixture of THF and pentane, were fixed and added to the calculated structure factors using the SQUEEZE routine from program Platon,[6] which determined there to be 309 electrons in 1234 $Å^3$ per unit cell associated with this solvent. Because the exact identity and amount of solvent were not determined, no solvent was included in the atom list or molecular formula. Thus all calculated quantities that derive from the molecular formula (i.e., F(000), density, molecular weight, etc.) are known to be incorrect.

The final full matrix least squares refinement converged to $R1 = 0.0341$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.0808$ (F^2, all data).

Structure description

The structure is the one suggested. The asymmetric unit contains one iron complex and two solvent sites, all in general positions. The solvent was not modeled (see above).

Unless noted otherwise all structural diagrams containing thermal displacement ellipsoids are drawn at the 50 % probability level.

Data collection, structure solution, and structure refinement were conducted at the X-ray Crystallographic Facility, B04 Hutchison Hall, Department of Chemistry, University of Rochester. All publications arising from this report MUST either 1) include William W. Brennessel as a coauthor or 2) acknowledge William W. Brennessel and the X-ray Crystallographic Facility of the Department of Chemistry at the University of Rochester.

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[1] *APEX3*, version 2016.5-0; Bruker AXS: Madison, WI, 2016.

[2] Sheldrick, G. M. *SADABS*, version 2016/2; *J. Appl. Cryst.* **2015**, *48*, 3-10.

[3] *SAINT*, version 8.34A; Bruker AXS: Madison, WI, 2013.

[4] Sheldrick, G. M. *SHELXT-2014/5*; University of Göttingen: Göttingen, Germany, 2014.

[5] Sheldrick, G. M. *SHELXL-2016/6*; *Acta. Cryst.* **2015**, C*71*, 3-8.

[6] Spek, A. L. *PLATON*, version 07052015; *Acta. Cryst.* **2015**, C*71*, 9-18.

Some equations of interest:

$$R_{int} = \Sigma\, |F_o^2 - < F_o^2 >| \,/\, \Sigma |F_o^2|$$

$$R1 = \Sigma ||F_o| - |F_c|| \,/\, \Sigma |F_o|$$

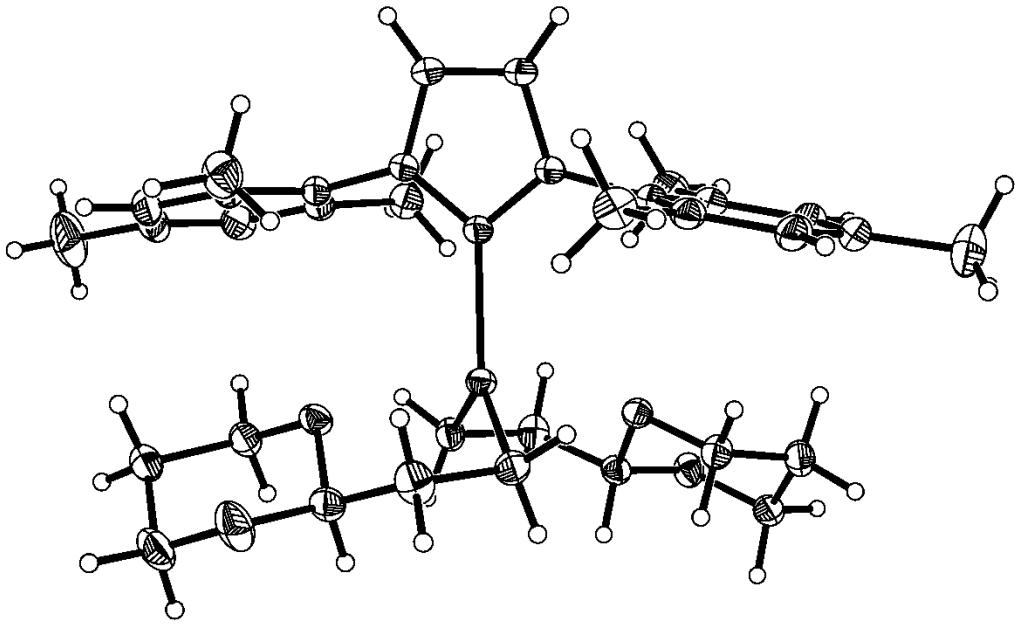
$$wR2 = [\Sigma [w(F_o^2 - F_c^2)^2] \,/\, \Sigma\, [w(F_o^2)^2]]^{1/2}$$

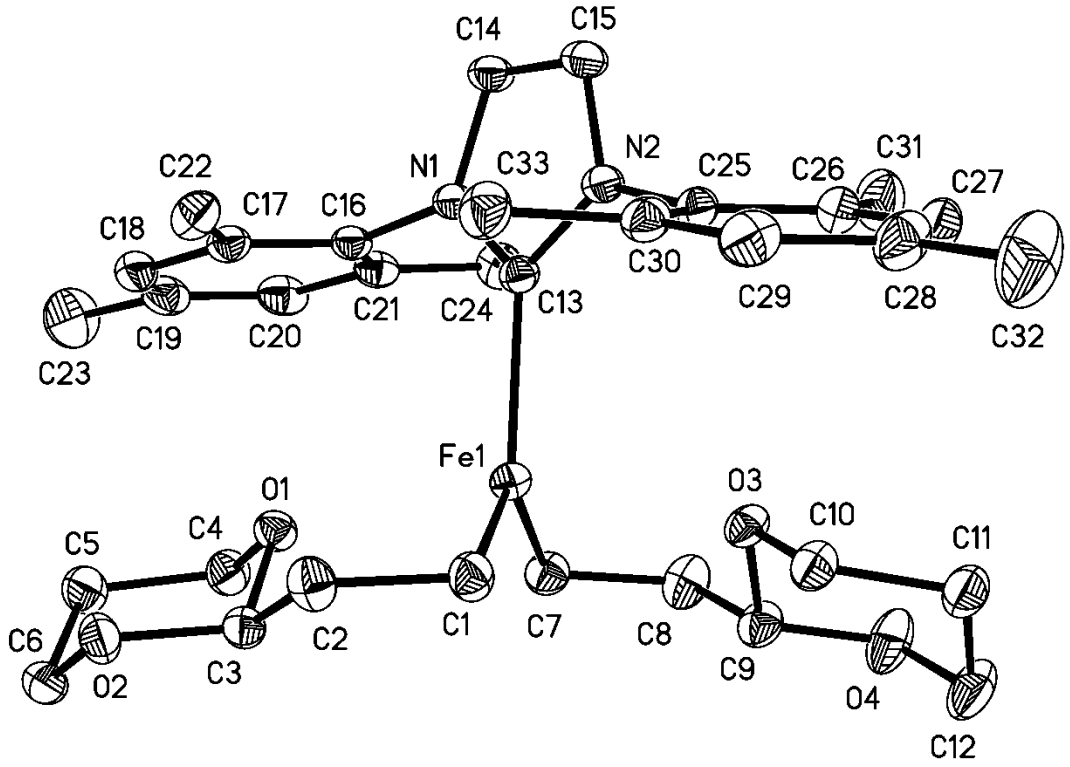
where $w = 1 \,/\, [\sigma^2 (F_o^2) + (aP)^2 + bP]$ and

$$P = 1/3 \max (0, F_o^2) + 2/3\, F_c^2$$

$$\text{GOF} = S = [\Sigma [w(F_o^2 - F_c^2)^2] \,/\, (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters

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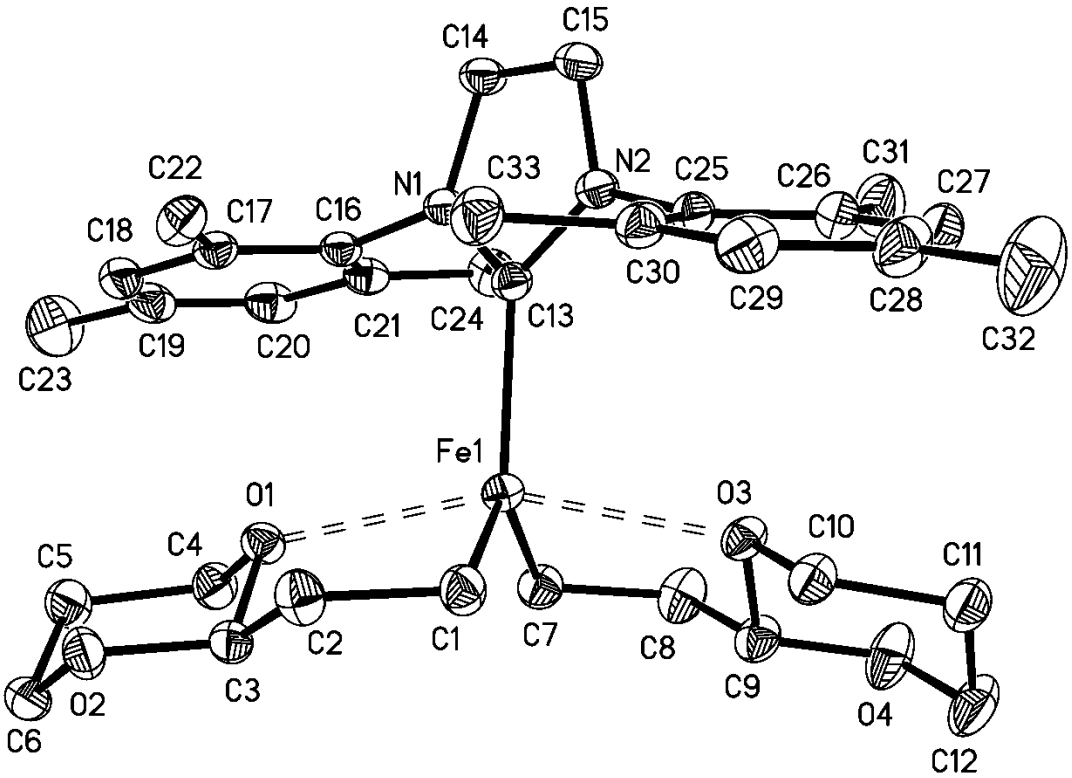
C22
N1
C33
N2
C25
C26
C31
C27
C18
C17
C16
C30
C29
C28
C32
C19
C20
C21
C24
C13
C23
Fe1
O1
O3
C10
C11
C4
C5
C1
C7
C8
C6
C2
C9
O2
C3
O4
C12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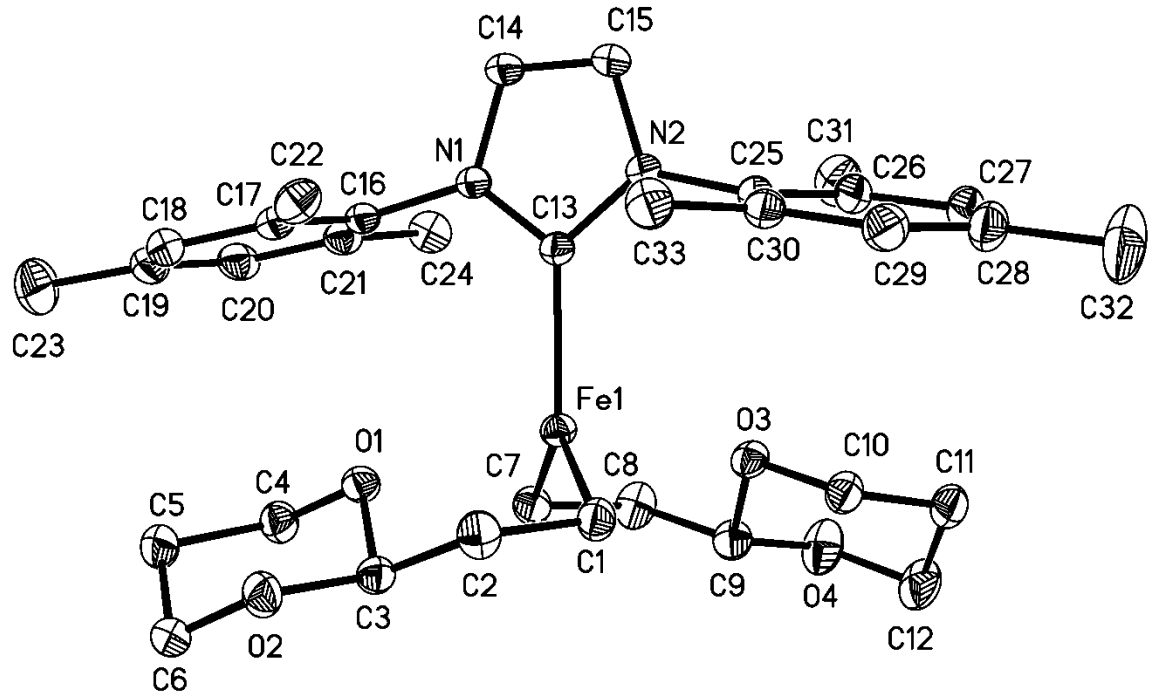
C32
C19
C20
C21
C24
C13
C23
Fe1
O1
O3
C10
C11
C4
C5
C1
C7
C8
C2
C9
O2
C3
O4
C6
C12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N2
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C16
C17
C18
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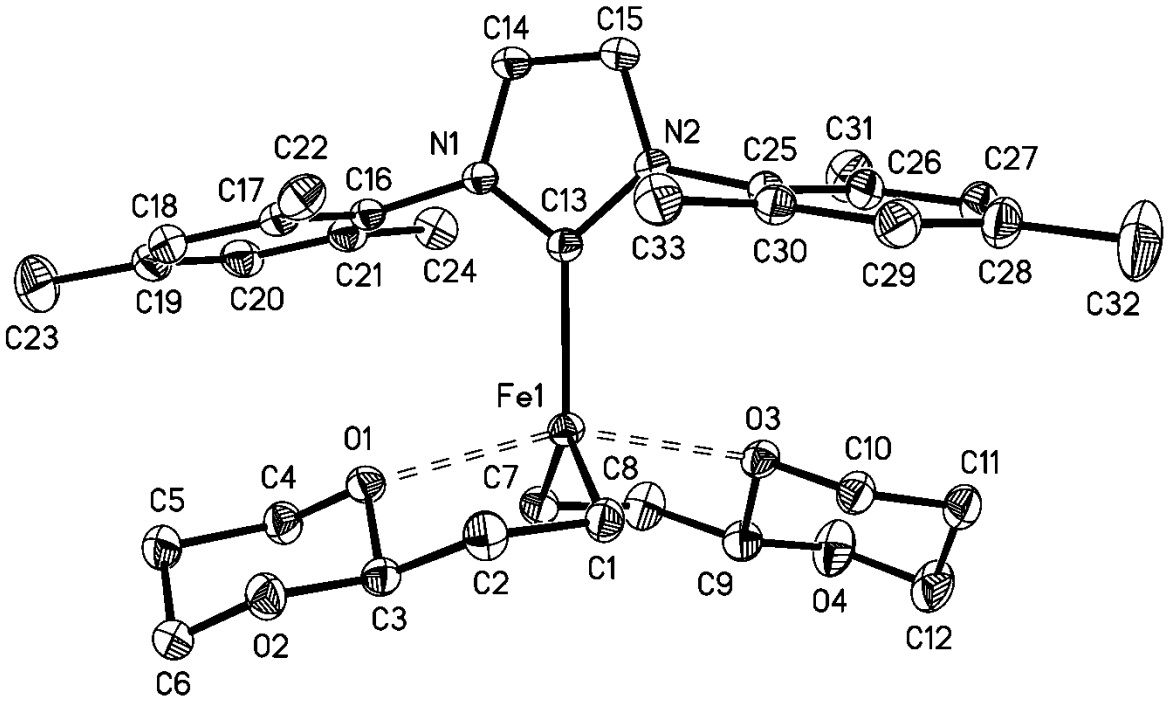
C28
C32
C24
C21
C19
C20
C23
Fe1
O1
O3
C10
C11
C7
C8
C4
C5
C1
C2
C9
O4
C3
O2
C12
C6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N2
C22
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C25
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C27
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C29
C28
C32
C24
C21
C19
C20
C23
Fe1
O1
O3
C10
C11
C7
C8
C4
C5
C1
C2
C9
O4
C3
O2
C12
C6

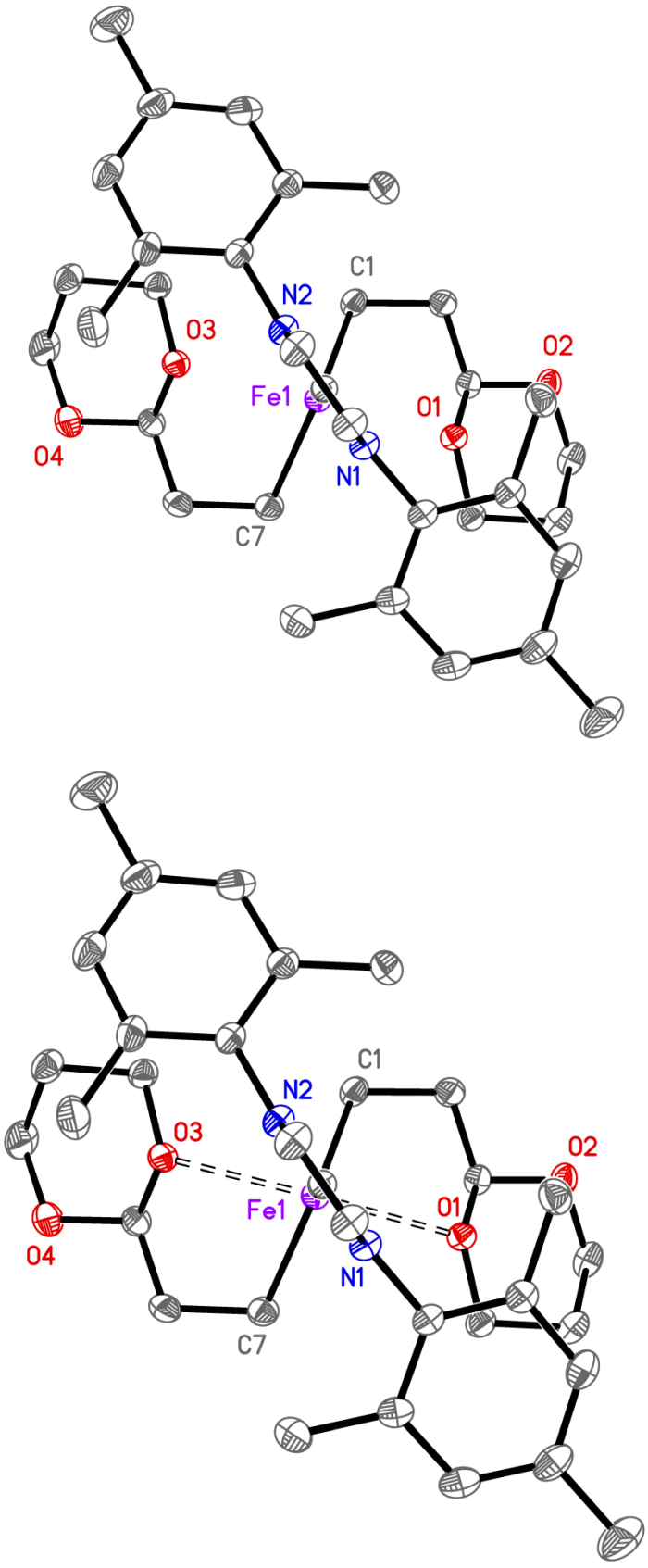

C1
N2
O3
O2
Fe1
O1
O4
N1
C7
C1
N2
O3
O2
Fe1
O1
O4
N1
C7

Table S6. Crystal data and structure refinement for neism35.

Identification code	neism35	
Empirical formula	C33 H46 Fe N2 O4	
Formula weight	590.57	
Temperature	100.0(5) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	*Cc*	
Unit cell dimensions	a = 11.2603(6) Å	α = 90°
	b = 22.9235(12) Å	β = 103.8779(11)°
	c = 16.1864(9) Å	γ = 90°
Volume	4056.2(4) Å^3	
Z	4	
Density (calculated)	0.967 Mg/m^3	
Absorption coefficient	0.401 mm^{-1}	
F(000)	1264	
Crystal color, morphology	yellow, block	
Crystal size	0.40 x 0.35 x 0.32 mm^3	
Theta range for data collection	1.777 to 38.797°	
Index ranges	$-19 \le h \le 19$, $-39 \le k \le 39$, $-28 \le l \le 27$	
Reflections collected	89669	
Independent reflections	22458 [R(int) = 0.0358]	
Observed reflections	18509	
Completeness to theta = 37.785°	100.0%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.7476 and 0.7065	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	22458 / 2 / 367	
Goodness-of-fit on F^2	0.981	
Final R indices [I>2sigma(I)]	R1 = 0.0341, wR2 = 0.0753	
R indices (all data)	R1 = 0.0481, wR2 = 0.0808	
Absolute structure parameter	0.017(3)	
Largest diff. peak and hole	0.363 and -0.245 e.Å^{-3}	

Table S7. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for neism35. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Fe1	5492(1)	6036(1)	4581(1)	17(1)
O1	4831(1)	5010(1)	4554(1)	19(1)
O2	5304(1)	4128(1)	3990(1)	24(1)
O3	5852(1)	6988(1)	4007(1)	21(1)
O4	4689(1)	7698(1)	3162(1)	34(1)
N1	5731(1)	6068(1)	6555(1)	18(1)
N2	7205(1)	6551(1)	6276(1)	19(1)
C1	6625(1)	5623(1)	3902(1)	22(1)
C2	6616(1)	4957(1)	4015(1)	24(1)
C3	5322(1)	4744(1)	3911(1)	20(1)
C4	3562(1)	4861(1)	4438(1)	23(1)
C5	3444(1)	4201(1)	4483(1)	26(1)
C6	4070(1)	3920(1)	3850(1)	26(1)
C7	3694(1)	6332(1)	4071(1)	23(1)
C8	3703(1)	6982(1)	3854(1)	28(1)
C9	4726(1)	7111(1)	3420(1)	24(1)
C10	6856(1)	7058(1)	3610(1)	23(1)
C11	6876(1)	7680(1)	3292(1)	28(1)
C12	5634(2)	7823(1)	2721(1)	36(1)
C13	6220(1)	6216(1)	5891(1)	17(1)
C14	6379(1)	6306(1)	7325(1)	22(1)
C15	7310(1)	6607(1)	7147(1)	23(1)
C16	4739(1)	5666(1)	6522(1)	18(1)
C17	5000(1)	5068(1)	6586(1)	21(1)
C18	4046(1)	4689(1)	6638(1)	25(1)
C19	2877(1)	4893(1)	6623(1)	28(1)
C20	2652(1)	5490(1)	6540(1)	26(1)
C21	3573(1)	5888(1)	6495(1)	21(1)
C22	6253(1)	4838(1)	6593(1)	26(1)
C23	1882(2)	4472(1)	6709(1)	46(1)
C24	3339(1)	6533(1)	6432(1)	28(1)

C25	8114(1)	6780(1)	5872(1)	20(1)
C26	8152(1)	7383(1)	5728(1)	23(1)
C27	9101(1)	7595(1)	5392(1)	29(1)
C28	9975(1)	7230(1)	5190(1)	31(1)
C29	9899(1)	6638(1)	5339(1)	28(1)
C30	8988(1)	6400(1)	5692(1)	21(1)
C31	7194(2)	7784(1)	5917(1)	34(1)
C32	10998(2)	7476(1)	4825(2)	51(1)
C33	8966(1)	5760(1)	5885(1)	27(1)

Table S8. Bond lengths [Å] and angles [°] for neism35.

Fe(1)-C(1)	2.0994(13)	C(7)-C(8)	1.532(2)
Fe(1)-C(7)	2.1062(12)	C(7)-H(7A)	0.9900
Fe(1)-C(13)	2.1222(12)	C(7)-H(7B)	0.9900
Fe(1)-O(3)	2.4428(9)	C(8)-C(9)	1.515(2)
Fe(1)-O(1)	2.4632(9)	C(8)-H(8A)	0.9900
O(1)-C(3)	1.4288(15)	C(8)-H(8B)	0.9900
O(1)-C(4)	1.4376(15)	C(9)-H(9)	1.0000
O(2)-C(3)	1.4190(16)	C(10)-C(11)	1.516(2)
O(2)-C(6)	1.4350(18)	C(10)-H(10A)	0.9900
O(3)-C(9)	1.4187(15)	C(10)-H(10B)	0.9900
O(3)-C(10)	1.4384(16)	C(11)-C(12)	1.515(2)
O(4)-C(9)	1.4058(17)	C(11)-H(11A)	0.9900
O(4)-C(12)	1.446(2)	C(11)-H(11B)	0.9900
N(1)-C(13)	1.3625(16)	C(12)-H(12A)	0.9900
N(1)-C(14)	1.3951(16)	C(12)-H(12B)	0.9900
N(1)-C(16)	1.4393(16)	C(14)-C(15)	1.3437(19)
N(2)-C(13)	1.3704(15)	C(14)-H(14)	0.9500
N(2)-C(15)	1.3930(16)	C(15)-H(15)	0.9500
N(2)-C(25)	1.4383(16)	C(16)-C(21)	1.3976(18)
C(1)-C(2)	1.5372(19)	C(16)-C(17)	1.4013(18)
C(1)-H(1A)	0.9900	C(17)-C(18)	1.3991(19)
C(1)-H(1B)	0.9900	C(17)-C(22)	1.5033(19)
C(2)-C(3)	1.5080(18)	C(18)-C(19)	1.393(2)
C(2)-H(2A)	0.9900	C(18)-H(18)	0.9500
C(2)-H(2B)	0.9900	C(19)-C(20)	1.393(2)
C(3)-H(3)	1.0000	C(19)-C(23)	1.509(2)
C(4)-C(5)	1.522(2)	C(20)-C(21)	1.3961(19)
C(4)-H(4A)	0.9900	C(20)-H(20)	0.9500
C(4)-H(4B)	0.9900	C(21)-C(24)	1.502(2)
C(5)-C(6)	1.519(2)	C(22)-H(22A)	0.9800
C(5)-H(5A)	0.9900	C(22)-H(22B)	0.9800
C(5)-H(5B)	0.9900	C(22)-H(22C)	0.9800
C(6)-H(6A)	0.9900	C(23)-H(23A)	0.9800
C(6)-H(6B)	0.9900	C(23)-H(23B)	0.9800

C(23)-H(23C)	0.9800	C(4)-O(1)-Fe(1)	121.11(7)
C(24)-H(24A)	0.9800	C(3)-O(2)-C(6)	110.49(10)
C(24)-H(24B)	0.9800	C(9)-O(3)-C(10)	110.46(10)
C(24)-H(24C)	0.9800	C(9)-O(3)-Fe(1)	103.25(7)
C(25)-C(30)	1.3961(19)	C(10)-O(3)-Fe(1)	119.36(8)
C(25)-C(26)	1.4042(18)	C(9)-O(4)-C(12)	111.31(12)
C(26)-C(27)	1.397(2)	C(13)-N(1)-C(14)	112.58(10)
C(26)-C(31)	1.504(2)	C(13)-N(1)-C(16)	126.00(10)
C(27)-C(28)	1.389(2)	C(14)-N(1)-C(16)	121.14(10)
C(27)-H(27)	0.9500	C(13)-N(2)-C(15)	112.03(10)
C(28)-C(29)	1.385(2)	C(13)-N(2)-C(25)	125.79(10)
C(28)-C(32)	1.524(2)	C(15)-N(2)-C(25)	121.93(10)
C(29)-C(30)	1.3995(19)	C(2)-C(1)-Fe(1)	111.35(8)
C(29)-H(29)	0.9500	C(2)-C(1)-H(1A)	109.4
C(30)-C(33)	1.501(2)	Fe(1)-C(1)-H(1A)	109.4
C(31)-H(31A)	0.9800	C(2)-C(1)-H(1B)	109.4
C(31)-H(31B)	0.9800	Fe(1)-C(1)-H(1B)	109.4
C(31)-H(31C)	0.9800	H(1A)-C(1)-H(1B)	108.0
C(32)-H(32A)	0.9800	C(3)-C(2)-C(1)	109.99(11)
C(32)-H(32B)	0.9800	C(3)-C(2)-H(2A)	109.7
C(32)-H(32C)	0.9800	C(1)-C(2)-H(2A)	109.7
C(33)-H(33A)	0.9800	C(3)-C(2)-H(2B)	109.7
C(33)-H(33B)	0.9800	C(1)-C(2)-H(2B)	109.7
C(33)-H(33C)	0.9800	H(2A)-C(2)-H(2B)	108.2
C(1)-Fe(1)-C(7)	126.02(5)	O(2)-C(3)-O(1)	110.11(10)
C(1)-Fe(1)-C(13)	118.21(5)	O(2)-C(3)-C(2)	110.25(11)
C(7)-Fe(1)-C(13)	115.70(5)	O(1)-C(3)-C(2)	108.23(10)
C(1)-Fe(1)-O(3)	91.56(4)	O(2)-C(3)-H(3)	109.4
C(7)-Fe(1)-O(3)	78.18(4)	O(1)-C(3)-H(3)	109.4
C(13)-Fe(1)-O(3)	98.45(4)	C(2)-C(3)-H(3)	109.4
C(1)-Fe(1)-O(1)	77.24(4)	O(1)-C(4)-C(5)	109.00(11)
C(7)-Fe(1)-O(1)	92.43(4)	O(1)-C(4)-H(4A)	109.9
C(13)-Fe(1)-O(1)	104.19(4)	C(5)-C(4)-H(4A)	109.9
O(3)-Fe(1)-O(1)	157.35(3)	O(1)-C(4)-H(4B)	109.9
C(3)-O(1)-C(4)	110.44(10)	C(5)-C(4)-H(4B)	109.9
C(3)-O(1)-Fe(1)	104.64(7)	H(4A)-C(4)-H(4B)	108.3

C(6)-C(5)-C(4)	109.29(11)	C(12)-C(11)-C(10)	108.89(12)
C(6)-C(5)-H(5A)	109.8	C(12)-C(11)-H(11A)	109.9
C(4)-C(5)-H(5A)	109.8	C(10)-C(11)-H(11A)	109.9
C(6)-C(5)-H(5B)	109.8	C(12)-C(11)-H(11B)	109.9
C(4)-C(5)-H(5B)	109.8	C(10)-C(11)-H(11B)	109.9
H(5A)-C(5)-H(5B)	108.3	H(11A)-C(11)-H(11B)	108.3
O(2)-C(6)-C(5)	109.96(11)	O(4)-C(12)-C(11)	109.65(12)
O(2)-C(6)-H(6A)	109.7	O(4)-C(12)-H(12A)	109.7
C(5)-C(6)-H(6A)	109.7	C(11)-C(12)-H(12A)	109.7
O(2)-C(6)-H(6B)	109.7	O(4)-C(12)-H(12B)	109.7
C(5)-C(6)-H(6B)	109.7	C(11)-C(12)-H(12B)	109.7
H(6A)-C(6)-H(6B)	108.2	H(12A)-C(12)-H(12B)	108.2
C(8)-C(7)-Fe(1)	110.17(8)	N(1)-C(13)-N(2)	102.55(10)
C(8)-C(7)-H(7A)	109.6	N(1)-C(13)-Fe(1)	127.40(8)
Fe(1)-C(7)-H(7A)	109.6	N(2)-C(13)-Fe(1)	129.76(9)
C(8)-C(7)-H(7B)	109.6	C(15)-C(14)-N(1)	106.12(11)
Fe(1)-C(7)-H(7B)	109.6	C(15)-C(14)-H(14)	126.9
H(7A)-C(7)-H(7B)	108.1	N(1)-C(14)-H(14)	126.9
C(9)-C(8)-C(7)	110.08(11)	C(14)-C(15)-N(2)	106.73(11)
C(9)-C(8)-H(8A)	109.6	C(14)-C(15)-H(15)	126.6
C(7)-C(8)-H(8A)	109.6	N(2)-C(15)-H(15)	126.6
C(9)-C(8)-H(8B)	109.6	C(21)-C(16)-C(17)	122.49(11)
C(7)-C(8)-H(8B)	109.6	C(21)-C(16)-N(1)	118.89(11)
H(8A)-C(8)-H(8B)	108.2	C(17)-C(16)-N(1)	118.44(11)
O(4)-C(9)-O(3)	110.32(11)	C(18)-C(17)-C(16)	117.51(12)
O(4)-C(9)-C(8)	110.60(12)	C(18)-C(17)-C(22)	120.93(13)
O(3)-C(9)-C(8)	107.88(11)	C(16)-C(17)-C(22)	121.55(12)
O(4)-C(9)-H(9)	109.3	C(19)-C(18)-C(17)	121.75(13)
O(3)-C(9)-H(9)	109.3	C(19)-C(18)-H(18)	119.1
C(8)-C(9)-H(9)	109.3	C(17)-C(18)-H(18)	119.1
O(3)-C(10)-C(11)	109.48(11)	C(18)-C(19)-C(20)	118.77(12)
O(3)-C(10)-H(10A)	109.8	C(18)-C(19)-C(23)	120.25(16)
C(11)-C(10)-H(10A)	109.8	C(20)-C(19)-C(23)	120.98(15)
O(3)-C(10)-H(10B)	109.8	C(19)-C(20)-C(21)	121.78(13)
C(11)-C(10)-H(10B)	109.8	C(19)-C(20)-H(20)	119.1
H(10A)-C(10)-H(10B)	108.2	C(21)-C(20)-H(20)	119.1

C(20)-C(21)-C(16)	117.69(13)	C(26)-C(27)-H(27)	118.9
C(20)-C(21)-C(24)	121.72(12)	C(29)-C(28)-C(27)	118.21(13)
C(16)-C(21)-C(24)	120.58(12)	C(29)-C(28)-C(32)	120.92(16)
C(17)-C(22)-H(22A)	109.5	C(27)-C(28)-C(32)	120.87(16)
C(17)-C(22)-H(22B)	109.5	C(28)-C(29)-C(30)	122.32(14)
H(22A)-C(22)-H(22B)	109.5	C(28)-C(29)-H(29)	118.8
C(17)-C(22)-H(22C)	109.5	C(30)-C(29)-H(29)	118.8
H(22A)-C(22)-H(22C)	109.5	C(25)-C(30)-C(29)	117.73(12)
H(22B)-C(22)-H(22C)	109.5	C(25)-C(30)-C(33)	121.23(12)
C(19)-C(23)-H(23A)	109.5	C(29)-C(30)-C(33)	121.03(13)
C(19)-C(23)-H(23B)	109.5	C(26)-C(31)-H(31A)	109.5
H(23A)-C(23)-H(23B)	109.5	C(26)-C(31)-H(31B)	109.5
C(19)-C(23)-H(23C)	109.5	H(31A)-C(31)-H(31B)	109.5
H(23A)-C(23)-H(23C)	109.5	C(26)-C(31)-H(31C)	109.5
H(23B)-C(23)-H(23C)	109.5	H(31A)-C(31)-H(31C)	109.5
C(21)-C(24)-H(24A)	109.5	H(31B)-C(31)-H(31C)	109.5
C(21)-C(24)-H(24B)	109.5	C(28)-C(32)-H(32A)	109.5
H(24A)-C(24)-H(24B)	109.5	C(28)-C(32)-H(32B)	109.5
C(21)-C(24)-H(24C)	109.5	H(32A)-C(32)-H(32B)	109.5
H(24A)-C(24)-H(24C)	109.5	C(28)-C(32)-H(32C)	109.5
H(24B)-C(24)-H(24C)	109.5	H(32A)-C(32)-H(32C)	109.5
C(30)-C(25)-C(26)	121.83(12)	H(32B)-C(32)-H(32C)	109.5
C(30)-C(25)-N(2)	118.76(11)	C(30)-C(33)-H(33A)	109.5
C(26)-C(25)-N(2)	119.25(12)	C(30)-C(33)-H(33B)	109.5
C(27)-C(26)-C(25)	117.68(13)	H(33A)-C(33)-H(33B)	109.5
C(27)-C(26)-C(31)	121.30(13)	C(30)-C(33)-H(33C)	109.5
C(25)-C(26)-C(31)	121.01(12)	H(33A)-C(33)-H(33C)	109.5
C(28)-C(27)-C(26)	122.21(14)	H(33B)-C(33)-H(33C)	109.5
C(28)-C(27)-H(27)	118.9		

Table S9. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for neism35. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U_{11} + ... + 2\ h\ k\ a^*\ b^*\ U_{12}\ ]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Fe1	16(1)	19(1)	15(1)	1(1)	3(1)	0(1)
O1	20(1)	20(1)	18(1)	-2(1)	6(1)	-1(1)
O2	32(1)	17(1)	26(1)	-2(1)	11(1)	-2(1)
O3	19(1)	23(1)	19(1)	5(1)	2(1)	2(1)
O4	29(1)	29(1)	44(1)	18(1)	8(1)	8(1)
N1	18(1)	21(1)	14(1)	0(1)	3(1)	-2(1)
N2	20(1)	21(1)	15(1)	1(1)	2(1)	-3(1)
C1	24(1)	21(1)	23(1)	0(1)	9(1)	-3(1)
C2	25(1)	22(1)	28(1)	-2(1)	12(1)	1(1)
C3	25(1)	18(1)	18(1)	-1(1)	7(1)	-1(1)
C4	21(1)	24(1)	25(1)	1(1)	6(1)	-3(1)
C5	30(1)	25(1)	26(1)	-1(1)	10(1)	-7(1)
C6	36(1)	23(1)	20(1)	-2(1)	7(1)	-8(1)
C7	19(1)	26(1)	23(1)	2(1)	2(1)	-1(1)
C8	20(1)	29(1)	34(1)	8(1)	7(1)	6(1)
C9	20(1)	24(1)	24(1)	6(1)	0(1)	2(1)
C10	22(1)	25(1)	23(1)	5(1)	5(1)	-1(1)
C11	28(1)	28(1)	26(1)	7(1)	6(1)	-4(1)
C12	36(1)	35(1)	34(1)	18(1)	4(1)	0(1)
C13	16(1)	18(1)	16(1)	1(1)	3(1)	0(1)
C14	24(1)	26(1)	15(1)	-2(1)	4(1)	-4(1)
C15	25(1)	27(1)	16(1)	-2(1)	2(1)	-6(1)
C16	19(1)	22(1)	13(1)	-1(1)	4(1)	-3(1)
C17	23(1)	24(1)	15(1)	0(1)	4(1)	-1(1)
C18	32(1)	24(1)	20(1)	0(1)	5(1)	-7(1)
C19	28(1)	34(1)	21(1)	-2(1)	6(1)	-12(1)
C20	20(1)	37(1)	23(1)	-3(1)	6(1)	-5(1)
C21	20(1)	27(1)	17(1)	-2(1)	5(1)	-1(1)
C22	28(1)	26(1)	24(1)	4(1)	7(1)	5(1)
C23	36(1)	49(1)	53(1)	4(1)	11(1)	-19(1)
C24	25(1)	28(1)	31(1)	-2(1)	6(1)	4(1)

C25	19(1)	21(1)	18(1)	2(1)	2(1)	-3(1)
C26	26(1)	21(1)	22(1)	3(1)	3(1)	-3(1)
C27	31(1)	27(1)	27(1)	9(1)	3(1)	-7(1)
C28	23(1)	40(1)	29(1)	10(1)	6(1)	-6(1)
C29	20(1)	36(1)	26(1)	3(1)	5(1)	-1(1)
C30	19(1)	25(1)	19(1)	3(1)	1(1)	-1(1)
C31	42(1)	23(1)	39(1)	2(1)	12(1)	4(1)
C32	36(1)	56(1)	67(1)	23(1)	23(1)	-6(1)
C33	24(1)	24(1)	31(1)	4(1)	3(1)	3(1)

Table S10. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for neism35.

	x	y	z	U(eq)
H1A	6342	5719	3290	27
H1B	7472	5769	4105	27
H2A	6985	4767	3586	29
H2B	7111	4851	4587	29
H3	4821	4860	3337	24
H4A	3088	5004	3879	28
H4B	3230	5047	4888	28
H5A	3829	4063	5066	32
H5B	2570	4090	4348	32
H6A	4071	3491	3917	31
H6B	3615	4015	3263	31
H7A	3323	6107	3551	28
H7B	3194	6268	4490	28
H8A	2908	7092	3473	33
H8B	3819	7216	4381	33
H9	4638	6853	2910	28
H10A	6765	6783	3127	28
H10B	7636	6970	4024	28
H11A	7064	7952	3781	33
H11B	7517	7722	2972	33
H12A	5604	8240	2561	43
H12B	5498	7588	2194	43
H14	6197	6263	7865	26
H15	7922	6818	7540	28
H18	4201	4281	6684	30
H20	1850	5631	6515	32
H22A	6860	5032	7045	39
H22B	6438	4916	6042	39
H22C	6276	4417	6698	39
H23A	1993	4102	6435	69

H23B	1082	4638	6434	69
H23C	1924	4405	7313	69
H24A	3884	6730	6915	42
H24B	2486	6610	6441	42
H24C	3494	6681	5900	42
H27	9149	8002	5298	35
H29	10486	6384	5196	33
H31A	7292	8174	5695	51
H31B	7285	7805	6534	51
H31C	6379	7633	5645	51
H32A	10819	7884	4659	77
H32B	11060	7248	4325	77
H32C	11774	7455	5256	77
H33A	9671	5570	5744	40
H33B	8209	5588	5546	40
H33C	9005	5706	6491	40

Table S11. Torsion angles [°] for neism35.

Fe1-C1-C2-C3	45.59(13)	C15-N2-C13-Fe1	173.82(9)
C6-O2-C3-O1	63.44(13)	C25-N2-C13-Fe1	-11.84(18)
C6-O2-C3-C2	-177.21(11)	C13-N1-C14-C15	-0.65(15)
C4-O1-C3-O2	-64.38(13)	C16-N1-C14-C15	173.56(12)
Fe1-O1-C3-O2	163.77(8)	N1-C14-C15-N2	0.48(15)
C4-O1-C3-C2	175.05(10)	C13-N2-C15-C14	-0.18(16)
Fe1-O1-C3-C2	43.19(11)	C25-N2-C15-C14	-174.77(12)
C1-C2-C3-O2	178.34(11)	C13-N1-C16-C21	-102.55(14)
C1-C2-C3-O1	-61.18(14)	C14-N1-C16-C21	84.06(15)
C3-O1-C4-C5	59.39(13)	C13-N1-C16-C17	82.21(15)
Fe1-O1-C4-C5	-177.94(8)	C14-N1-C16-C17	-91.18(14)
O1-C4-C5-C6	-53.92(15)	C21-C16-C17-C18	-1.23(18)
C3-O2-C6-C5	-58.32(14)	N1-C16-C17-C18	173.84(11)
C4-C5-C6-O2	53.57(15)	C21-C16-C17-C22	178.50(12)
Fe1-C7-C8-C9	43.42(14)	N1-C16-C17-C22	-6.44(17)
C12-O4-C9-O3	62.67(16)	C16-C17-C18-C19	0.35(19)
C12-O4-C9-C8	-178.05(12)	C22-C17-C18-C19	-179.38(13)
C10-O3-C9-O4	-63.59(14)	C17-C18-C19-C20	1.1(2)
Fe1-O3-C9-O4	167.70(9)	C17-C18-C19-C23	-178.07(15)
C10-O3-C9-C8	175.49(11)	C18-C19-C20-C21	-1.7(2)
Fe1-O3-C9-C8	46.78(11)	C23-C19-C20-C21	177.43(15)
C7-C8-C9-O4	175.77(11)	C19-C20-C21-C16	0.9(2)
C7-C8-C9-O3	-63.49(15)	C19-C20-C21-C24	-178.10(13)
C9-O3-C10-C11	59.59(14)	C17-C16-C21-C20	0.64(18)
Fe1-O3-C10-C11	178.97(8)	N1-C16-C21-C20	-174.41(11)
O3-C10-C11-C12	-54.38(16)	C17-C16-C21-C24	179.62(12)
C9-O4-C12-C11	-57.77(18)	N1-C16-C21-C24	4.58(18)
C10-C11-C12-O4	53.13(18)	C13-N2-C25-C30	-74.36(16)
C14-N1-C13-N2	0.51(13)	C15-N2-C25-C30	99.45(15)
C16-N1-C13-N2	-173.36(11)	C13-N2-C25-C26	110.18(14)
C14-N1-C13-Fe1	-173.70(9)	C15-N2-C25-C26	-76.01(16)
C16-N1-C13-Fe1	12.42(18)	C30-C25-C26-C27	0.10(19)
C15-N2-C13-N1	-0.20(14)	N2-C25-C26-C27	175.42(12)
C25-N2-C13-N1	174.14(11)	C30-C25-C26-C31	179.30(13)

N2-C25-C26-C31	-5.38(19)
C25-C26-C27-C28	1.0(2)
C31-C26-C27-C28	-178.17(15)
C26-C27-C28-C29	-0.7(2)
C26-C27-C28-C32	-179.99(16)
C27-C28-C29-C30	-0.9(2)
C32-C28-C29-C30	178.47(16)
C26-C25-C30-C29	-1.51(18)
N2-C25-C30-C29	-176.85(11)
C26-C25-C30-C33	177.34(12)
N2-C25-C30-C33	2.00(18)
C28-C29-C30-C25	1.9(2)
C28-C29-C30-C33	-176.94(13)

5.2 (IMes)FeBr((1,3-Dioxan-2-yl)ethyl) (2-Br) and (IMes)FeBr$_2$(THF) (3)

REFERENCE NUMBER: neivf10

CRYSTAL STRUCTURE REPORT

$C_{30.71}\ H_{44.57}\ Br_{1.14}\ Fe\ N_2\ O_{2.86}$

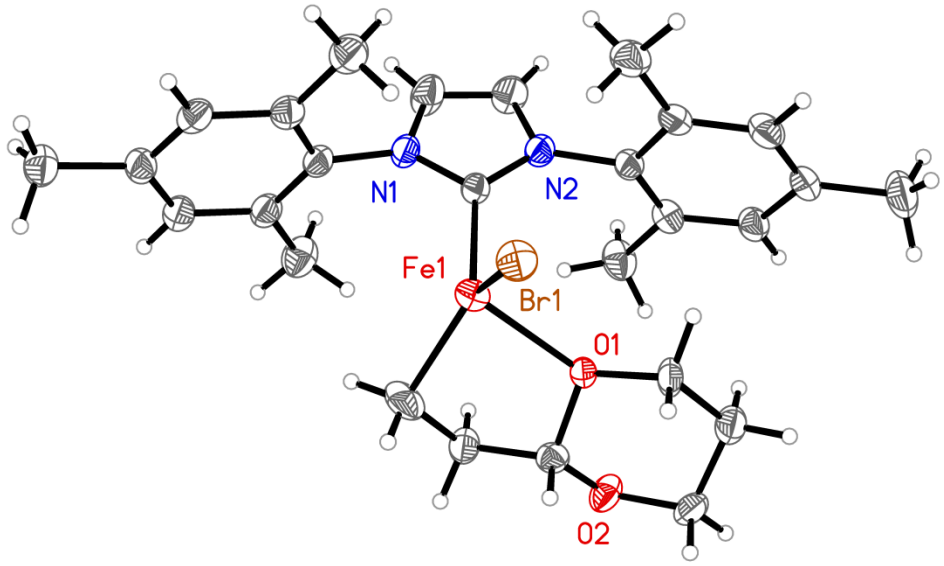
or

$[(IMes)FeBr(C_6H_{11}O_2)]_{0.86} \cdot [(IMes)FeBr_2(THF)]_{0.14} \cdot Et_2O$

Report prepared for:

V. Fleischauer, Prof. M. Neidig

February 29, 2016



William W. Brennessel

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Department of Chemistry, University of Rochester

120 Trustee Road

Rochester, NY 14627

Data collection

A crystal (0.25 x 0.20 x 0.18 mm^3) was placed onto the tip of a thin glass optical fiber and mounted on a Bruker SMART APEX II CCD platform diffractometer for a data collection at 100(2) K.[1] A preliminary set of cell constants and an orientation matrix were calculated from reflections harvested from three orthogonal wedges of reciprocal space. The full data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 60 seconds and a detector distance of 4.00 cm. A randomly oriented region of reciprocal space was surveyed: four major sections of frames were collected with 0.50º steps in ω at four different ϕ settings and a detector position of -38º in 2θ. The intensity data were corrected for absorption.[2] Final cell constants were calculated from the xyz centroids of 4019 strong reflections from the actual data collection after integration.[3] See Table S12 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SHELXT-2014/5[4] and refined using SHELXL-2014/7.[5] The space group *Ia* was determined based on systematic absences and intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0512$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1148$ (F^2, all data).

Structure description

The structure contains the one suggested. The structure is modeled as a disorder of (IMes)FeBr($C_6H_{11}O_2$) and (IMes)$FeBr_2$(THF) (0.86:0.14). The asymmetric unit contains one iron complex (of either type) and a cocrystallized diethyl ether molecule, both in general positions.

Unless noted otherwise all structural diagrams containing thermal displacement ellipsoids are drawn at the 50 % probability level.

Data collection, structure solution, and structure refinement were conducted at the X-ray Crystallographic Facility, B51 Hutchison Hall, Department of Chemistry, University of Rochester. All publications arising from this report MUST either 1) include William W. Brennessel as a coauthor or 2) acknowledge William W. Brennessel and the X-ray Crystallographic Facility of the Department of Chemistry at the University of Rochester.

[1] *APEX3*, version 2015.9-0; Bruker AXS: Madison, WI, 2015.

[2] Sheldrick, G. M. *SADABS*, version 2014/5; *J. Appl. Cryst.* **2015**, *48*, 3-10.

[3] *SAINT*, version 8.34A; Bruker AXS: Madison, WI, 2013.

[4] Sheldrick, G. M. *SHELXT-2014/5*; University of Göttingen: Göttingen, Germany, 2014.

[5] Sheldrick, G. M. *SHELXL-2014/7*; *Acta. Cryst.* **2015**, C*71*, 3-8.

Some equations of interest:

$$R_{int} = \Sigma\, |F_o^2 - < F_o^2 >| \,/\, \Sigma |F_o^2|$$

$$R1 = \Sigma ||F_o| - |F_c|| \,/\, \Sigma |F_o|$$

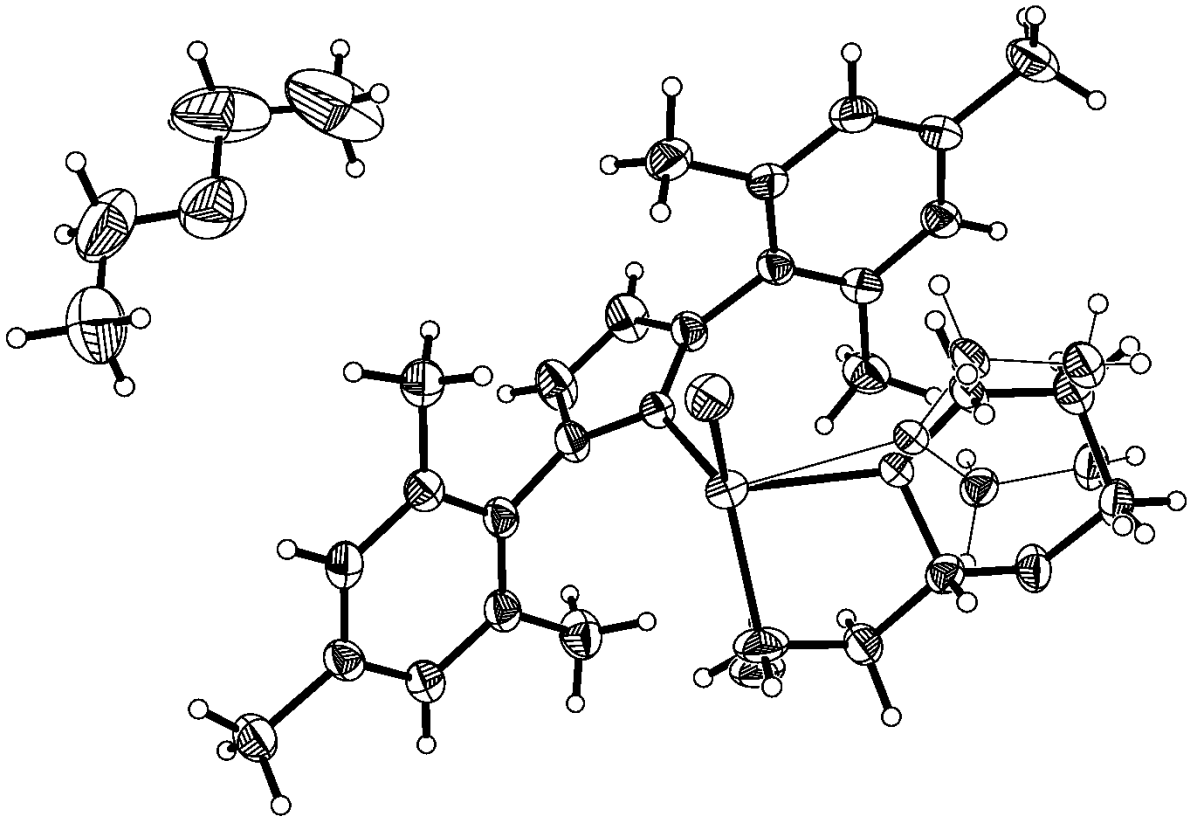
$$wR2 = [\Sigma [w(F_o^2 - F_c^2)^2] \,/\, \Sigma\, [w(F_o^2)^2]]^{1/2}$$

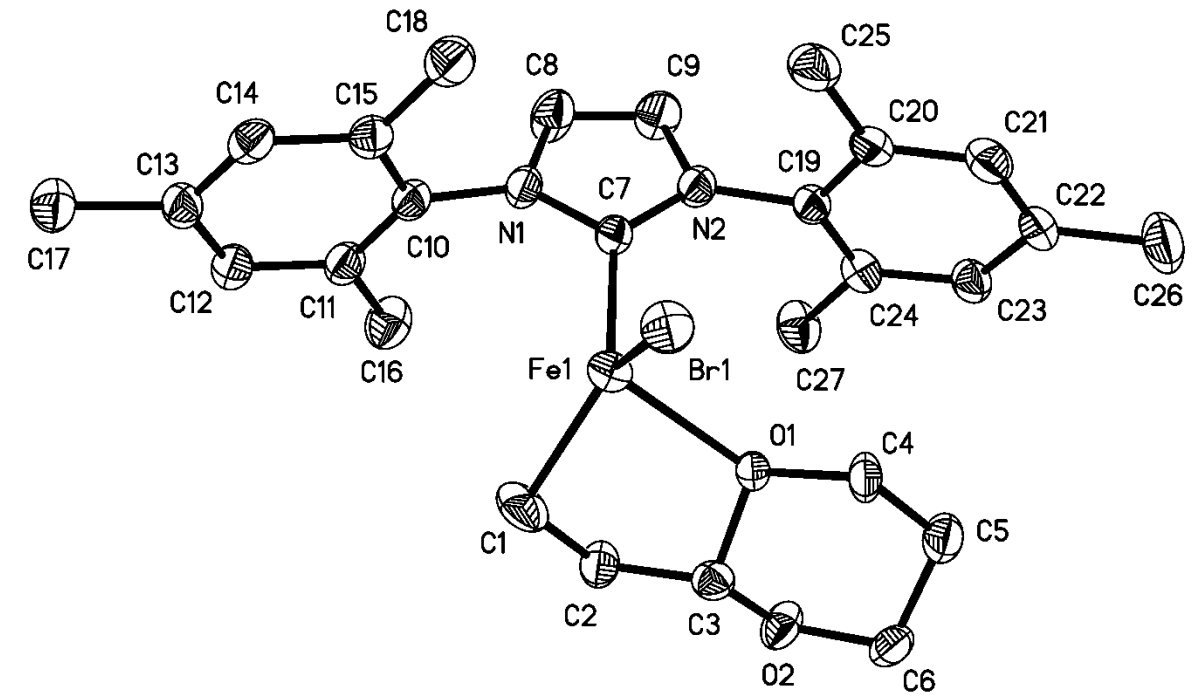
where $w = 1 \,/\, [\sigma^2 (F_o^2) + (aP)^2 + bP]$ and

$$P = 1/3 \max (0, F_o^2) + 2/3\, F_c^2$$

$$\text{GOF} = S = [\Sigma [w(F_o^2 - F_c^2)^2] \,/\, (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters

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C8
C9
C14
C15
C20
C21
C13
C19
C7
C22
C17
C10
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N2
C26
C12
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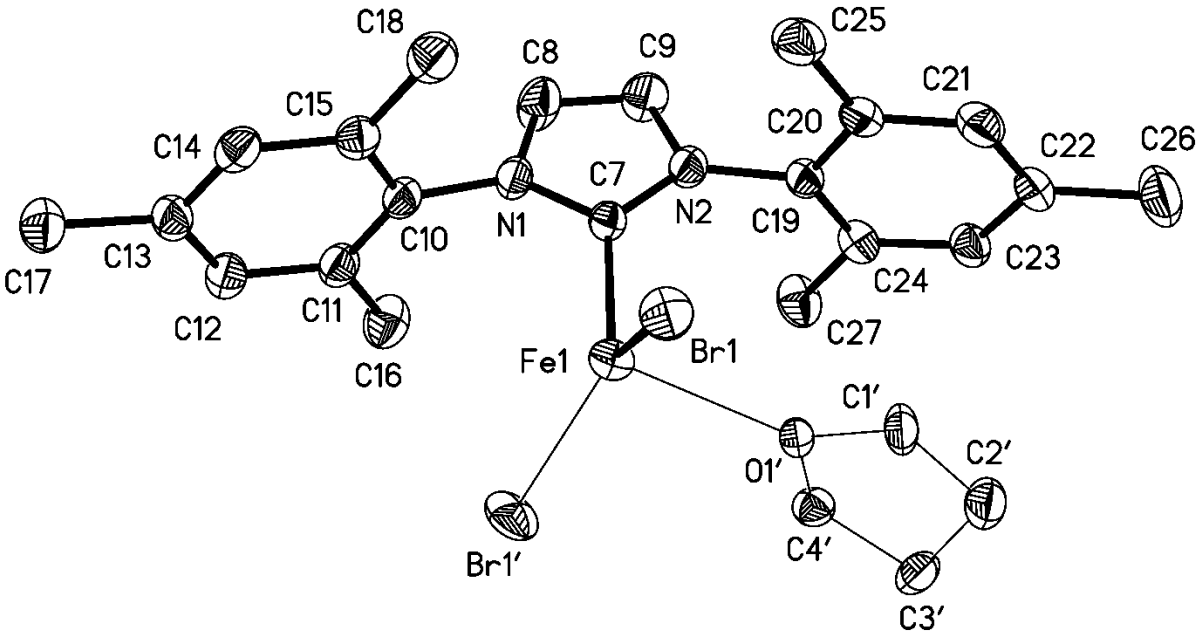
O1
C4
C1
C5
C2
C3
O2
C6


C18
C25
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C9
C21
C15
C20
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C26
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N2
C10
N1
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C17
C13
C24
C23
C12
C11
C27
C16
Fe1
Br1
C1′
O1′
C2′
Br1′
C4′
C3′

Table S12. Crystal data and structure refinement for neivf10.

Identification code	neivf10	
Empirical formula	C30.71 H44.57 Br1.14 Fe N2 O2.86	
Formula weight	634.83	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	*Ia*	
Unit cell dimensions	a = 15.186(2) Å	α = 90°
	b = 13.4178(13) Å	β = 112.4487(14)°
	c = 17.1643(16) Å	γ = 90°
Volume	3232.4(7) Å^3	
Z	4	
Density (calculated)	1.304 Mg/m^3	
Absorption coefficient	1.912 mm^{-1}	
F(000)	1327	
Crystal color, morphology	pale yellow, block	
Crystal size	0.25 x 0.20 x 0.18 mm^3	
Theta range for data collection	1.988 to 32.567°	
Index ranges	$-22 \leq h \leq 22$, $-20 \leq k \leq 20$, $-25 \leq l \leq 25$	
Reflections collected	27844	
Independent reflections	11571 [R(int) = 0.0463]	
Observed reflections	8330	
Completeness to theta = 32.567°	99.7%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.7464 and 0.6218	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	11571 / 8 / 369	
Goodness-of-fit on F^2	1.005	
Final R indices [I>2sigma(I)]	R1 = 0.0512, wR2 = 0.1039	
R indices (all data)	R1 = 0.0822, wR2 = 0.1148	
Absolute structure parameter	0.032(5)	
Largest diff. peak and hole	1.154 and -0.540 e.Å^{-3}	

Table S13. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for neivf10. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Fe1	5519(1)	7358(1)	4982(1)	27(1)
Br1	7015(1)	6897(1)	4890(1)	32(1)
O1	6000(3)	8036(3)	6231(2)	24(1)
O2	5689(3)	9437(3)	6880(2)	33(1)
C1	4854(14)	8679(13)	4625(12)	40(2)
C2	4774(4)	9137(4)	5420(3)	30(1)
C3	5728(4)	9073(4)	6139(3)	26(1)
C4	6949(5)	7909(5)	6870(4)	30(1)
C5	6988(5)	8359(5)	7698(4)	34(1)
C6	6617(4)	9413(5)	7557(4)	31(1)
Br1'	4678(7)	8836(6)	4514(6)	40(2)
O1'	6270(20)	7880(20)	6431(17)	24(1)
C1'	7240(20)	7690(30)	6960(20)	30(1)
C2'	7430(30)	8270(30)	7740(20)	34(1)
C3'	6640(20)	9000(30)	7560(20)	31(1)
C4'	5840(20)	8530(20)	6848(17)	26(1)
N1	3872(3)	5841(3)	4459(2)	27(1)
N2	4736(3)	5574(3)	5758(2)	27(1)
C7	4694(3)	6152(3)	5087(3)	24(1)
C8	3424(4)	5106(4)	4738(3)	40(1)
C9	3972(4)	4936(4)	5547(3)	39(1)
C10	3541(3)	6136(3)	3590(3)	28(1)
C11	2785(3)	6800(3)	3274(3)	30(1)
C12	2420(4)	7000(3)	2404(3)	31(1)
C13	2795(4)	6532(4)	1873(3)	30(1)
C14	3566(3)	5894(3)	2217(3)	30(1)
C15	3959(3)	5676(3)	3083(3)	28(1)
C16	2345(4)	7291(4)	3833(4)	42(1)
C17	2361(4)	6715(4)	938(3)	39(1)
C18	4775(4)	4961(4)	3437(3)	36(1)
C19	5499(3)	5574(3)	6580(3)	26(1)

C20	6313(4)	5019(3)	6706(3)	29(1)
C21	7013(4)	4987(4)	7521(3)	33(1)
C22	6895(4)	5467(4)	8195(3)	34(1)
C23	6065(4)	6011(4)	8037(3)	30(1)
C24	5355(3)	6082(3)	7237(3)	29(1)
C25	6457(4)	4485(4)	5994(3)	38(1)
C26	7641(5)	5419(5)	9075(3)	50(1)
C27	4472(4)	6689(4)	7082(3)	39(1)
C28	4531(6)	3124(6)	1456(5)	73(2)
C29	4521(6)	2231(6)	1958(6)	80(3)
O4	4650(4)	2493(4)	2770(4)	75(2)
C30	4677(7)	1631(7)	3276(8)	104(4)
C31	4796(9)	1999(9)	4151(8)	125(5)

Table S14. Bond lengths [Å] and angles [°] for neivf10.

Fe(1)-C(1)	2.016(12)
Fe(1)-C(7)	2.096(4)
Fe(1)-O(1)	2.181(4)
Fe(1)-Br(1')	2.329(6)
Fe(1)-O(1')	2.41(3)
Fe(1)-Br(1)	2.4184(8)
O(1)-C(3)	1.443(6)
O(1)-C(4)	1.450(7)
O(2)-C(3)	1.384(6)
O(2)-C(6)	1.445(7)
C(1)-C(2)	1.54(2)
C(1)-H(1A)	0.9900
C(1)-H(1B)	0.9900
C(2)-C(3)	1.505(8)
C(2)-H(2A)	0.9900
C(2)-H(2B)	0.9900
C(3)-H(3A)	1.0000
C(4)-C(5)	1.524(8)
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(5)-C(6)	1.507(9)
C(5)-H(5A)	0.9900
C(5)-H(5B)	0.9900
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
O(1')-C(1')	1.429(14)
O(1')-C(4')	1.430(14)
C(1')-C(2')	1.49(3)
C(1')-H(1C)	0.9900
C(1')-H(1D)	0.9900
C(2')-C(3')	1.49(3)
C(2')-H(2C)	0.9900
C(2')-H(2D)	0.9900
C(3')-C(4')	1.49(3)
C(3')-H(3B)	0.9900
C(3')-H(3C)	0.9900

C(4')-H(4C)	0.9900
C(4')-H(4D)	0.9900
N(1)-C(7)	1.366(6)
N(1)-C(8)	1.383(6)
N(1)-C(10)	1.437(6)
N(2)-C(7)	1.369(5)
N(2)-C(9)	1.375(6)
N(2)-C(19)	1.443(6)
C(8)-C(9)	1.338(7)
C(8)-H(8)	0.9500
C(9)-H(9)	0.9500
C(10)-C(11)	1.390(7)
C(10)-C(15)	1.401(6)
C(11)-C(12)	1.406(7)
C(11)-C(16)	1.512(7)
C(12)-C(13)	1.395(7)
C(12)-H(12)	0.9500
C(13)-C(14)	1.387(7)
C(13)-C(17)	1.504(7)
C(14)-C(15)	1.405(6)
C(14)-H(14)	0.9500
C(15)-C(18)	1.499(7)
C(16)-H(16A)	0.9800
C(16)-H(16B)	0.9800
C(16)-H(16C)	0.9800
C(17)-H(17A)	0.9800
C(17)-H(17B)	0.9800
C(17)-H(17C)	0.9800
C(18)-H(18A)	0.9800
C(18)-H(18B)	0.9800
C(18)-H(18C)	0.9800
C(19)-C(20)	1.387(7)
C(19)-C(24)	1.405(6)
C(20)-C(21)	1.397(7)
C(20)-C(25)	1.503(7)
C(21)-C(22)	1.394(7)

C(21)-H(21)	0.9500
C(22)-C(23)	1.390(7)
C(22)-C(26)	1.505(7)
C(23)-C(24)	1.387(7)
C(23)-H(23)	0.9500
C(24)-C(27)	1.503(7)
C(25)-H(25A)	0.9800
C(25)-H(25B)	0.9800
C(25)-H(25C)	0.9800
C(26)-H(26A)	0.9800
C(26)-H(26B)	0.9800
C(26)-H(26C)	0.9800
C(27)-H(27A)	0.9800
C(27)-H(27B)	0.9800
C(27)-H(27C)	0.9800
C(28)-C(29)	1.479(13)
C(28)-H(28A)	0.9800
C(28)-H(28B)	0.9800
C(28)-H(28C)	0.9800
C(29)-O(4)	1.377(11)
C(29)-H(29A)	0.9900
C(29)-H(29B)	0.9900
O(4)-C(30)	1.437(10)
C(30)-C(31)	1.525(18)
C(30)-H(30A)	0.9900
C(30)-H(30B)	0.9900
C(31)-H(31A)	0.9800
C(31)-H(31B)	0.9800
C(31)-H(31C)	0.9800
C(1)-Fe(1)-C(7)	117.3(7)
C(1)-Fe(1)-O(1)	82.7(6)
C(7)-Fe(1)-O(1)	102.97(16)
C(7)-Fe(1)-Br(1')	114.9(3)
C(7)-Fe(1)-O(1')	101.5(8)
Br(1')-Fe(1)-O(1')	95.2(7)
C(1)-Fe(1)-Br(1)	125.4(6)

C(7)-Fe(1)-Br(1)	114.55(12)
O(1)-Fe(1)-Br(1)	101.55(11)
Br(1')-Fe(1)-Br(1)	126.9(3)
O(1')-Fe(1)-Br(1)	93.1(6)
C(3)-O(1)-C(4)	110.9(4)
C(3)-O(1)-Fe(1)	108.7(3)
C(4)-O(1)-Fe(1)	123.9(3)
C(3)-O(2)-C(6)	111.1(4)
C(2)-C(1)-Fe(1)	106.3(9)
C(2)-C(1)-H(1A)	110.5
Fe(1)-C(1)-H(1A)	110.5
C(2)-C(1)-H(1B)	110.5
Fe(1)-C(1)-H(1B)	110.5
H(1A)-C(1)-H(1B)	108.7
C(3)-C(2)-C(1)	109.0(7)
C(3)-C(2)-H(2A)	109.9
C(1)-C(2)-H(2A)	109.9
C(3)-C(2)-H(2B)	109.9
C(1)-C(2)-H(2B)	109.9
H(2A)-C(2)-H(2B)	108.3
O(2)-C(3)-O(1)	110.6(4)
O(2)-C(3)-C(2)	111.6(4)
O(1)-C(3)-C(2)	106.9(4)
O(2)-C(3)-H(3A)	109.3
O(1)-C(3)-H(3A)	109.3
C(2)-C(3)-H(3A)	109.3
O(1)-C(4)-C(5)	109.4(5)
O(1)-C(4)-H(4A)	109.8
C(5)-C(4)-H(4A)	109.8
O(1)-C(4)-H(4B)	109.8
C(5)-C(4)-H(4B)	109.8
H(4A)-C(4)-H(4B)	108.2
C(6)-C(5)-C(4)	110.1(5)
C(6)-C(5)-H(5A)	109.6
C(4)-C(5)-H(5A)	109.6
C(6)-C(5)-H(5B)	109.6

C(4)-C(5)-H(5B)	109.6
H(5A)-C(5)-H(5B)	108.2
O(2)-C(6)-C(5)	109.9(5)
O(2)-C(6)-H(6A)	109.7
C(5)-C(6)-H(6A)	109.7
O(2)-C(6)-H(6B)	109.7
C(5)-C(6)-H(6B)	109.7
H(6A)-C(6)-H(6B)	108.2
C(1')-O(1')-C(4')	111(3)
C(1')-O(1')-Fe(1)	124.5(19)
C(4')-O(1')-Fe(1)	124.4(18)
O(1')-C(1')-C(2')	106(3)
O(1')-C(1')-H(1C)	110.6
C(2')-C(1')-H(1C)	110.6
O(1')-C(1')-H(1D)	110.6
C(2')-C(1')-H(1D)	110.6
H(1C)-C(1')-H(1D)	108.8
C(3')-C(2')-C(1')	107(3)
C(3')-C(2')-H(2C)	110.3
C(1')-C(2')-H(2C)	110.3
C(3')-C(2')-H(2D)	110.3
C(1')-C(2')-H(2D)	110.3
H(2C)-C(2')-H(2D)	108.5
C(2')-C(3')-C(4')	103(3)
C(2')-C(3')-H(3B)	111.1
C(4')-C(3')-H(3B)	111.1
C(2')-C(3')-H(3C)	111.1
C(4')-C(3')-H(3C)	111.1
H(3B)-C(3')-H(3C)	109.1
O(1')-C(4')-C(3')	107(3)
O(1')-C(4')-H(4C)	110.4
C(3')-C(4')-H(4C)	110.4
O(1')-C(4')-H(4D)	110.4
C(3')-C(4')-H(4D)	110.4
H(4C)-C(4')-H(4D)	108.6
C(7)-N(1)-C(8)	111.5(4)

C(7)-N(1)-C(10)	126.0(4)
C(8)-N(1)-C(10)	122.1(4)
C(7)-N(2)-C(9)	111.5(4)
C(7)-N(2)-C(19)	125.8(4)
C(9)-N(2)-C(19)	122.6(4)
N(1)-C(7)-N(2)	103.1(3)
N(1)-C(7)-Fe(1)	124.3(3)
N(2)-C(7)-Fe(1)	132.2(3)
C(9)-C(8)-N(1)	106.7(4)
C(9)-C(8)-H(8)	126.7
N(1)-C(8)-H(8)	126.7
C(8)-C(9)-N(2)	107.2(4)
C(8)-C(9)-H(9)	126.4
N(2)-C(9)-H(9)	126.4
C(11)-C(10)-C(15)	122.9(4)
C(11)-C(10)-N(1)	119.4(4)
C(15)-C(10)-N(1)	117.6(4)
C(10)-C(11)-C(12)	117.9(4)
C(10)-C(11)-C(16)	122.1(4)
C(12)-C(11)-C(16)	120.0(4)
C(13)-C(12)-C(11)	121.0(5)
C(13)-C(12)-H(12)	119.5
C(11)-C(12)-H(12)	119.5
C(14)-C(13)-C(12)	119.2(4)
C(14)-C(13)-C(17)	120.6(4)
C(12)-C(13)-C(17)	120.1(5)
C(13)-C(14)-C(15)	121.8(4)
C(13)-C(14)-H(14)	119.1
C(15)-C(14)-H(14)	119.1
C(10)-C(15)-C(14)	117.1(4)
C(10)-C(15)-C(18)	122.0(4)
C(14)-C(15)-C(18)	120.9(4)
C(11)-C(16)-H(16A)	109.5
C(11)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
C(11)-C(16)-H(16C)	109.5

H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5
C(13)-C(17)-H(17A)	109.5
C(13)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5
C(13)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
C(15)-C(18)-H(18A)	109.5
C(15)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5
C(15)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5
C(20)-C(19)-C(24)	122.5(4)
C(20)-C(19)-N(2)	119.2(4)
C(24)-C(19)-N(2)	118.2(4)
C(19)-C(20)-C(21)	117.7(4)
C(19)-C(20)-C(25)	121.7(4)
C(21)-C(20)-C(25)	120.6(4)
C(22)-C(21)-C(20)	121.8(4)
C(22)-C(21)-H(21)	119.1
C(20)-C(21)-H(21)	119.1
C(23)-C(22)-C(21)	118.3(4)
C(23)-C(22)-C(26)	119.8(5)
C(21)-C(22)-C(26)	121.8(5)
C(24)-C(23)-C(22)	122.2(4)
C(24)-C(23)-H(23)	118.9
C(22)-C(23)-H(23)	118.9
C(23)-C(24)-C(19)	117.5(4)
C(23)-C(24)-C(27)	121.0(4)
C(19)-C(24)-C(27)	121.5(4)
C(20)-C(25)-H(25A)	109.5
C(20)-C(25)-H(25B)	109.5
H(25A)-C(25)-H(25B)	109.5
C(20)-C(25)-H(25C)	109.5

H(25A)-C(25)-H(25C)	109.5
H(25B)-C(25)-H(25C)	109.5
C(22)-C(26)-H(26A)	109.5
C(22)-C(26)-H(26B)	109.5
H(26A)-C(26)-H(26B)	109.5
C(22)-C(26)-H(26C)	109.5
H(26A)-C(26)-H(26C)	109.5
H(26B)-C(26)-H(26C)	109.5
C(24)-C(27)-H(27A)	109.4
C(24)-C(27)-H(27B)	109.5
H(27A)-C(27)-H(27B)	109.5
C(24)-C(27)-H(27C)	109.5
H(27A)-C(27)-H(27C)	109.5
H(27B)-C(27)-H(27C)	109.5
C(29)-C(28)-H(28A)	109.5
C(29)-C(28)-H(28B)	109.5
H(28A)-C(28)-H(28B)	109.5
C(29)-C(28)-H(28C)	109.5
H(28A)-C(28)-H(28C)	109.5
H(28B)-C(28)-H(28C)	109.5
O(4)-C(29)-C(28)	110.8(6)
O(4)-C(29)-H(29A)	109.5
C(28)-C(29)-H(29A)	109.5
O(4)-C(29)-H(29B)	109.5
C(28)-C(29)-H(29B)	109.5
H(29A)-C(29)-H(29B)	108.1
C(29)-O(4)-C(30)	111.5(8)
O(4)-C(30)-C(31)	107.4(8)
O(4)-C(30)-H(30A)	110.2
C(31)-C(30)-H(30A)	110.2
O(4)-C(30)-H(30B)	110.2
C(31)-C(30)-H(30B)	110.2
H(30A)-C(30)-H(30B)	108.5
C(30)-C(31)-H(31A)	109.5
C(30)-C(31)-H(31B)	109.5
H(31A)-C(31)-H(31B)	109.5

C(30)-C(31)-H(31C)	109.5
H(31A)-C(31)-H(31C)	109.5
H(31B)-C(31)-H(31C)	109.5

Table S15. Anisotropic displacement parameters ($Å^2$x 10^3) for neivf10. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U_{11} + ... + 2 h k a^* b^* U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Fe1	28(1)	23(1)	30(1)	1(1)	11(1)	2(1)
Br1	32(1)	34(1)	33(1)	-2(1)	16(1)	5(1)
O1	26(2)	22(2)	18(2)	0(2)	3(2)	4(2)
O2	28(2)	37(2)	28(2)	-12(2)	6(2)	4(2)
C1	44(4)	30(3)	45(3)	9(2)	16(3)	19(2)
C2	29(3)	33(3)	24(2)	-3(2)	6(2)	7(2)
C3	31(3)	23(2)	25(2)	-2(2)	13(2)	1(2)
C4	30(4)	35(3)	18(2)	0(2)	0(3)	9(2)
C5	35(4)	38(3)	25(3)	-3(2)	5(3)	2(3)
C6	33(3)	30(3)	28(2)	-8(3)	8(2)	-8(3)
Br1'	44(4)	30(3)	45(3)	9(2)	16(3)	19(2)
O1'	26(2)	22(2)	18(2)	0(2)	3(2)	4(2)
C1'	30(4)	35(3)	18(2)	0(2)	0(3)	9(2)
C2'	35(4)	38(3)	25(3)	-3(2)	5(3)	2(3)
C3'	33(3)	30(3)	28(2)	-8(3)	8(2)	-8(3)
C4'	31(3)	23(2)	25(2)	-2(2)	13(2)	1(2)
N1	30(2)	29(2)	20(2)	0(1)	6(2)	-8(2)
N2	28(2)	27(2)	24(2)	0(1)	7(2)	-5(2)
C7	27(2)	24(2)	20(2)	0(2)	9(2)	1(2)
C8	38(3)	47(3)	30(2)	0(2)	9(2)	-19(2)
C9	43(3)	37(3)	34(3)	3(2)	13(2)	-19(2)
C10	30(2)	27(2)	23(2)	-2(2)	6(2)	-6(2)
C11	29(2)	31(2)	26(2)	-3(2)	7(2)	-4(2)
C12	31(2)	33(2)	27(2)	-1(2)	9(2)	-1(2)
C13	33(2)	30(2)	25(2)	3(2)	9(2)	-7(2)
C14	35(2)	30(2)	27(2)	-5(2)	15(2)	-8(2)
C15	26(2)	27(2)	28(2)	-1(2)	9(2)	-7(2)
C16	38(3)	50(3)	35(3)	-6(2)	13(2)	2(2)
C17	45(3)	42(3)	28(2)	1(2)	12(2)	-2(2)
C18	38(3)	37(3)	33(3)	-3(2)	13(2)	0(2)
C19	27(2)	24(2)	23(2)	3(2)	7(2)	-2(2)

C20	35(3)	22(2)	28(2)	3(2)	11(2)	3(2)
C21	34(3)	29(2)	37(2)	4(2)	13(2)	9(2)
C22	40(3)	28(2)	27(2)	6(2)	5(2)	6(2)
C23	37(3)	30(2)	24(2)	3(2)	11(2)	5(2)
C24	28(2)	30(2)	29(2)	4(2)	11(2)	4(2)
C25	49(3)	32(2)	36(3)	0(2)	18(2)	9(2)
C26	53(4)	55(4)	30(3)	9(2)	4(3)	16(3)
C27	34(3)	47(3)	35(3)	3(2)	14(2)	14(2)
C28	65(5)	86(6)	61(5)	-15(4)	18(4)	-13(4)
C29	62(5)	63(5)	101(7)	-40(5)	15(5)	-1(4)
O4	70(4)	51(3)	89(4)	-7(3)	14(3)	-6(3)
C30	64(5)	59(5)	158(11)	38(6)	6(6)	-12(4)
C31	92(8)	123(9)	114(10)	59(8)	-12(7)	-33(7)

Table S16. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for neivf10.

	x	y	z	U(eq)
H1A	5227	9121	4404	48
H1B	4214	8583	4179	48
H2A	4571	9842	5313	36
H2B	4291	8770	5565	36
H3A	6205	9466	5996	31
H4A	7421	8242	6689	36
H4B	7108	7191	6949	36
H5A	6598	7953	7927	41
H5B	7653	8356	8114	41
H6A	6571	9673	8080	37
H6B	7063	9843	7414	37
H1C	7673	7904	6675	36
H1D	7346	6965	7083	36
H2C	7454	7820	8206	41
H2D	8048	8622	7909	41
H3B	6813	9651	7392	37
H3C	6467	9089	8056	37
H4C	5427	8144	7062	31
H4D	5457	9044	6454	31
H8	2840	4783	4417	48
H9	3854	4464	5908	46
H12	1911	7461	2174	37
H14	3835	5596	1857	36
H16A	2849	7588	4323	62
H16B	2001	6791	4023	62
H16C	1901	7812	3515	62
H17A	2437	7418	824	58
H17B	1682	6547	723	58
H17C	2682	6298	658	58
H18A	4619	4457	3777	54

H18B	5348	5324	3790	54
H18C	4890	4634	2974	54
H21	7584	4628	7618	40
H23	5982	6346	8491	37
H25A	5891	4085	5684	57
H25B	7014	4048	6221	57
H25C	6559	4973	5612	57
H26A	7691	6070	9348	74
H26B	8257	5243	9052	74
H26C	7462	4913	9400	74
H27A	4218	6923	6497	58
H27B	4630	7263	7464	58
H27C	3994	6277	7183	58
H28A	4446	2921	883	109
H28B	5141	3471	1719	109
H28C	4011	3571	1432	109
H29A	3905	1880	1691	96
H29B	5035	1771	1969	96
H30A	5217	1195	3312	125
H30B	4079	1246	3023	125
H31A	4769	1431	4500	187
H31B	4283	2468	4103	187
H31C	5413	2335	4412	187

Table S17. Torsion angles [°] for neivf10.

Fe1-C1-C2-C3	48.2(12)
C6-O2-C3-O1	63.7(6)
C6-O2-C3-C2	-177.5(5)
C4-O1-C3-O2	-62.8(6)
Fe1-O1-C3-O2	157.8(3)
C4-O1-C3-C2	175.6(4)
Fe1-O1-C3-C2	36.2(4)
C1-C2-C3-O2	-177.6(8)
C1-C2-C3-O1	-56.6(9)
C3-O1-C4-C5	56.1(6)
Fe1-O1-C4-C5	-171.9(4)
O1-C4-C5-C6	-51.7(7)
C3-O2-C6-C5	-59.1(6)
C4-C5-C6-O2	52.6(7)
C4'-O1'-C1'-C2'	-1(5)
Fe1-O1'-C1'-C2'	172(3)
O1'-C1'-C2'-C3'	-15(5)
C1'-C2'-C3'-C4'	24(4)
C1'-O1'-C4'-C3'	17(4)
Fe1-O1'-C4'-C3'	-156(2)
C2'-C3'-C4'-O1'	-25(4)
C8-N1-C7-N2	0.5(5)
C10-N1-C7-N2	-172.9(4)
C8-N1-C7-Fe1	-173.0(3)
C10-N1-C7-Fe1	13.6(6)
C9-N2-C7-N1	0.0(5)
C19-N2-C7-N1	177.0(4)
C9-N2-C7-Fe1	172.8(4)
C19-N2-C7-Fe1	-10.2(6)
C7-N1-C8-C9	-0.9(6)
C10-N1-C8-C9	172.8(5)
N1-C8-C9-N2	0.8(6)
C7-N2-C9-C8	-0.5(6)
C19-N2-C9-C8	-177.6(5)

C7-N1-C10-C11	-106.0(5)
C8-N1-C10-C11	81.3(6)
C7-N1-C10-C15	78.6(6)
C8-N1-C10-C15	-94.1(5)
C15-C10-C11-C12	1.0(7)
N1-C10-C11-C12	-174.1(4)
C15-C10-C11-C16	-179.8(5)
N1-C10-C11-C16	5.0(7)
C10-C11-C12-C13	1.2(7)
C16-C11-C12-C13	-178.0(5)
C11-C12-C13-C14	-2.8(7)
C11-C12-C13-C17	176.8(4)
C12-C13-C14-C15	2.4(7)
C17-C13-C14-C15	-177.2(4)
C11-C10-C15-C14	-1.5(6)
N1-C10-C15-C14	173.8(4)
C11-C10-C15-C18	-179.9(4)
N1-C10-C15-C18	-4.7(6)
C13-C14-C15-C10	-0.3(7)
C13-C14-C15-C18	178.2(4)
C7-N2-C19-C20	-82.2(5)
C9-N2-C19-C20	94.5(6)
C7-N2-C19-C24	102.3(5)
C9-N2-C19-C24	-81.0(6)
C24-C19-C20-C21	-0.9(7)
N2-C19-C20-C21	-176.2(4)
C24-C19-C20-C25	-179.7(4)
N2-C19-C20-C25	5.0(6)
C19-C20-C21-C22	1.8(7)
C25-C20-C21-C22	-179.3(5)
C20-C21-C22-C23	-1.7(7)
C20-C21-C22-C26	179.1(5)
C21-C22-C23-C24	0.6(7)
C26-C22-C23-C24	179.9(5)
C22-C23-C24-C19	0.3(7)
C22-C23-C24-C27	-179.1(5)

C20-C19-C24-C23	-0.2(7)
N2-C19-C24-C23	175.2(4)
C20-C19-C24-C27	179.2(5)
N2-C19-C24-C27	-5.5(6)
C28-C29-O4-C30	-177.9(7)
C29-O4-C30-C31	-178.6(8)

5.3 (SIPr)Fe((1,3-Dioxan-2-yl)ethyl)$_2$ (4)

REFERENCE NUMBER: neism54

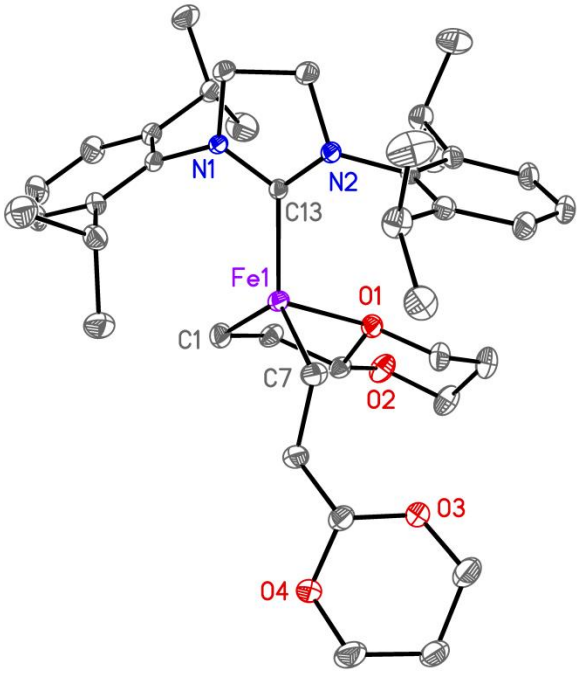
CRYSTAL STRUCTURE REPORT

C_{39} H_{60} Fe N_2 O_4

Report prepared for:

Dr. S. Muñoz, Prof. M. Neidig

June 16, 2017



William W. Brennessel

X-ray Crystallographic Facility

Department of Chemistry, University of Rochester

120 Trustee Road

Rochester, NY 14627

Data collection

A crystal (0.45 x 0.34 x 0.20 mm^3) was placed onto the tip of a thin glass optical fiber and mounted on a Bruker SMART APEX II CCD platform diffractometer for a data collection at 100.0(5) K.[1] A preliminary set of cell constants and an orientation matrix were calculated from reflections harvested from three orthogonal wedges of reciprocal space. The full data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 40 seconds and a detector distance of 4.03 cm. A randomly oriented region of reciprocal space was surveyed: five major sections of frames were collected with 0.50º steps in ω at five different ϕ settings and a detector position of -38º in 2θ. The intensity data were corrected for absorption.[2] Final cell constants were calculated from the xyz centroids of 4082 strong reflections from the actual data collection after integration.[3] See Table 18 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SHELXT-2014/5[4] and refined using SHELXL-2016/6.[5] The space group *P*-1 was determined based on intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0484$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1459$ (F^2, all data).

Structure description

The structure is the one suggested. The asymmetric unit contains one molecule in a general position. Additional non-bonding distances and angles follow (see corresponding figures):

Fe1…C23: 3.5400(15) Å
Fe1…H23A: 2.68 Å
H23A…Fe1—O1: 149.1º
H23A…Fe1—C1: 75.9º
H23A…Fe1—C7: 80.5º
H23A…Fe1—C13: 97.5º

Unless noted otherwise all structural diagrams containing thermal displacement ellipsoids are drawn at the 50 % probability level.

Data collection, structure solution, and structure refinement were conducted at the X-ray Crystallographic Facility, B04 Hutchison Hall, Department of Chemistry, University of Rochester. All publications arising from this report MUST either 1) include William W. Brennessel as a coauthor or 2) acknowledge William W. Brennessel and the X-ray Crystallographic Facility of the Department of Chemistry at the University of Rochester.

[1] *APEX3*, version 2016.5-0; Bruker AXS: Madison, WI, 2016.

[2] Krause, L.; Herbst-Irmer, R.; Sheldrick, G. M.; Stalke, D. *SADABS*, version 2016/2; *J. Appl. Cryst.* **2015**, *48*, 3-10.

[3] *SAINT*, version 8.34A; Bruker AXS: Madison, WI, 2013.

[4] Sheldrick, G. M. *SHELXT*, version 2014/5; *Acta. Cryst.* **2015**, A*71*, 3-8.

[5] Sheldrick, G. M. *SHELXL*, version 2016/6; *Acta. Cryst.* **2015**, C*71*, 3-8.

Some equations of interest:

$$R_{int} = \Sigma\, |F_o^2 - < F_o^2 >| \,/\, \Sigma |F_o{}^2|$$

$$R1 = \Sigma ||F_o| - |F_c|| \,/\, \Sigma |F_o|$$

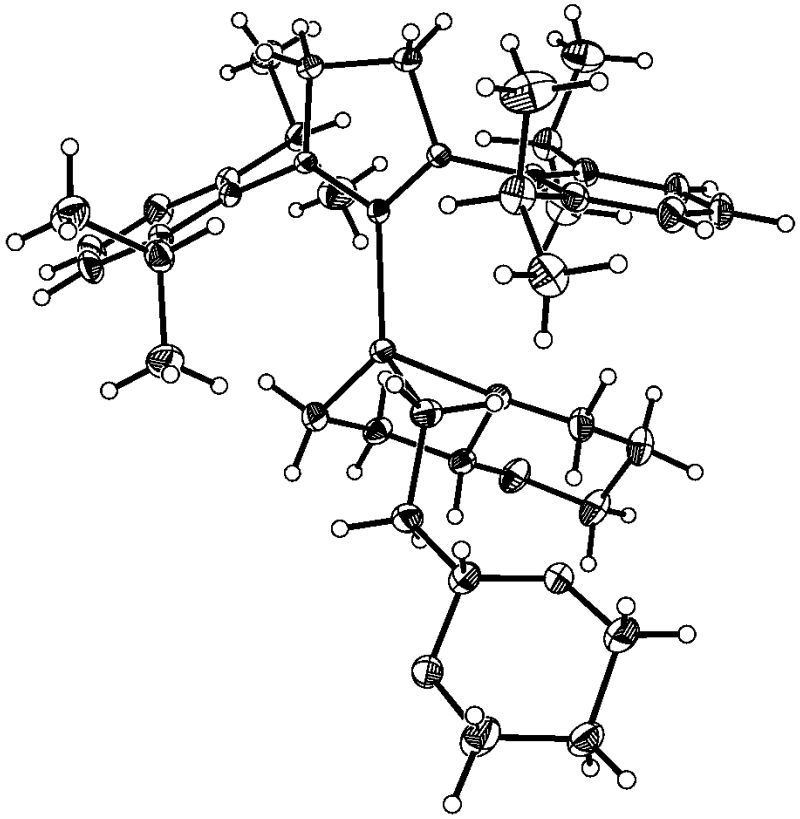
$$wR2 = [\Sigma [w(F_o^2 - F_c{}^2)^2] \,/\, \Sigma\, [w(F_o{}^2)^2]]^{1/2}$$

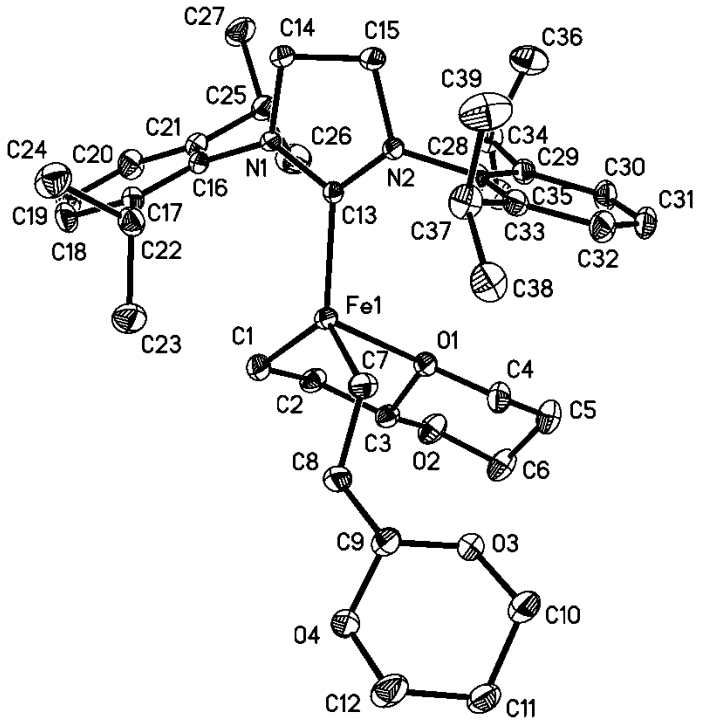
where $w = 1 \,/\, [\sigma^2 (F_o{}^2) + (aP)^2 + bP]$ and

$$P = 1/3 \max (0, F_o{}^2) + 2/3\, F_c{}^2$$

$$\text{GOF} = S = [\Sigma [w(F_o^2 - F_c{}^2)^2] \,/\, (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters

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C14
C15
C36
C39
C25
C21
C24
C20
C26
C28
C34
C29
C30
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N2
C16
C35
C31
C19
C17
C13
C37
C33
C18
C22
C32
C38
Fe1
C23
C1
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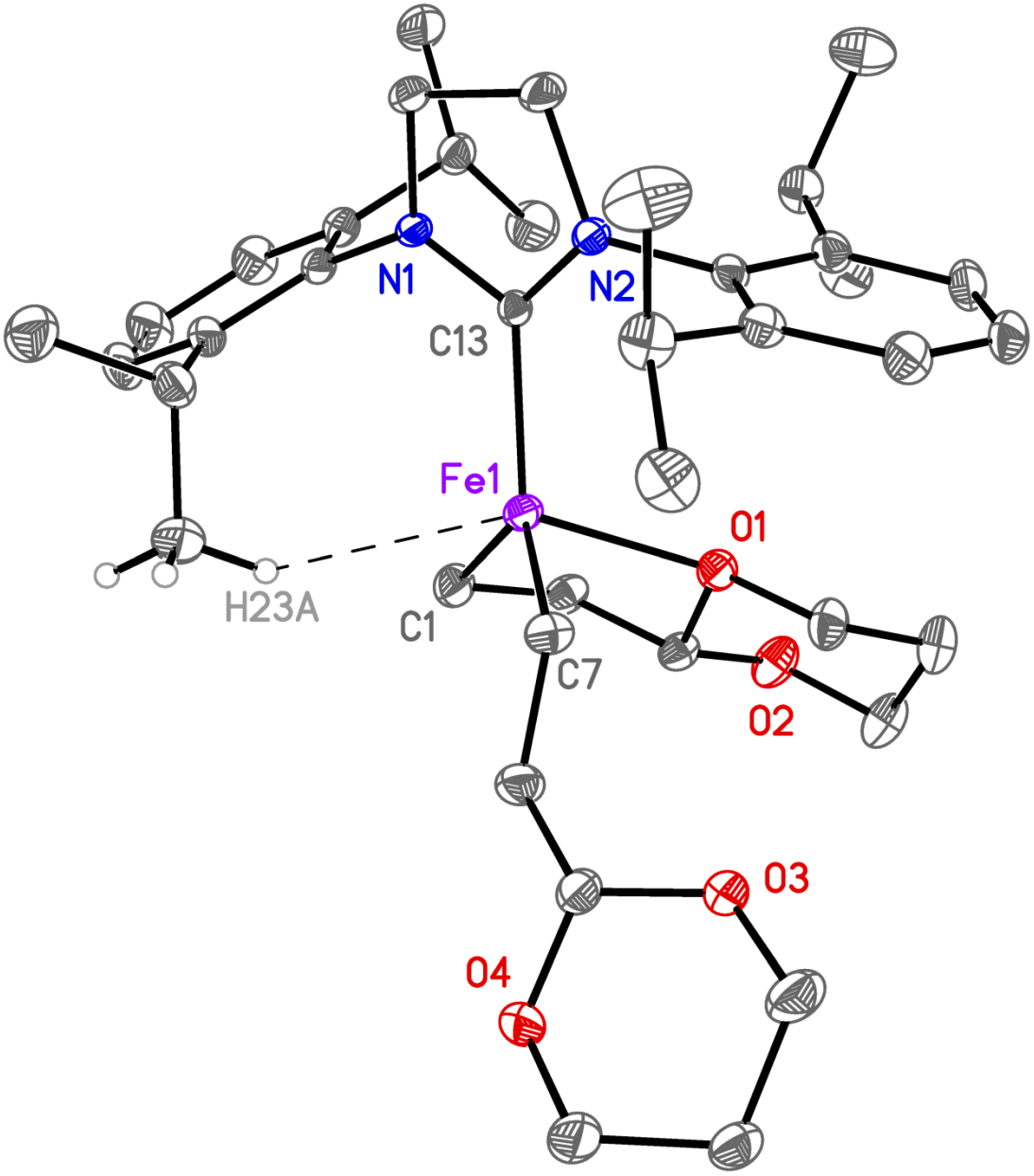
C8
C6
C9
O3
C10
O4
C12
C11


N1
N2
C13
Fe1
O1
H23A
C1
C7
O2
O3
O4

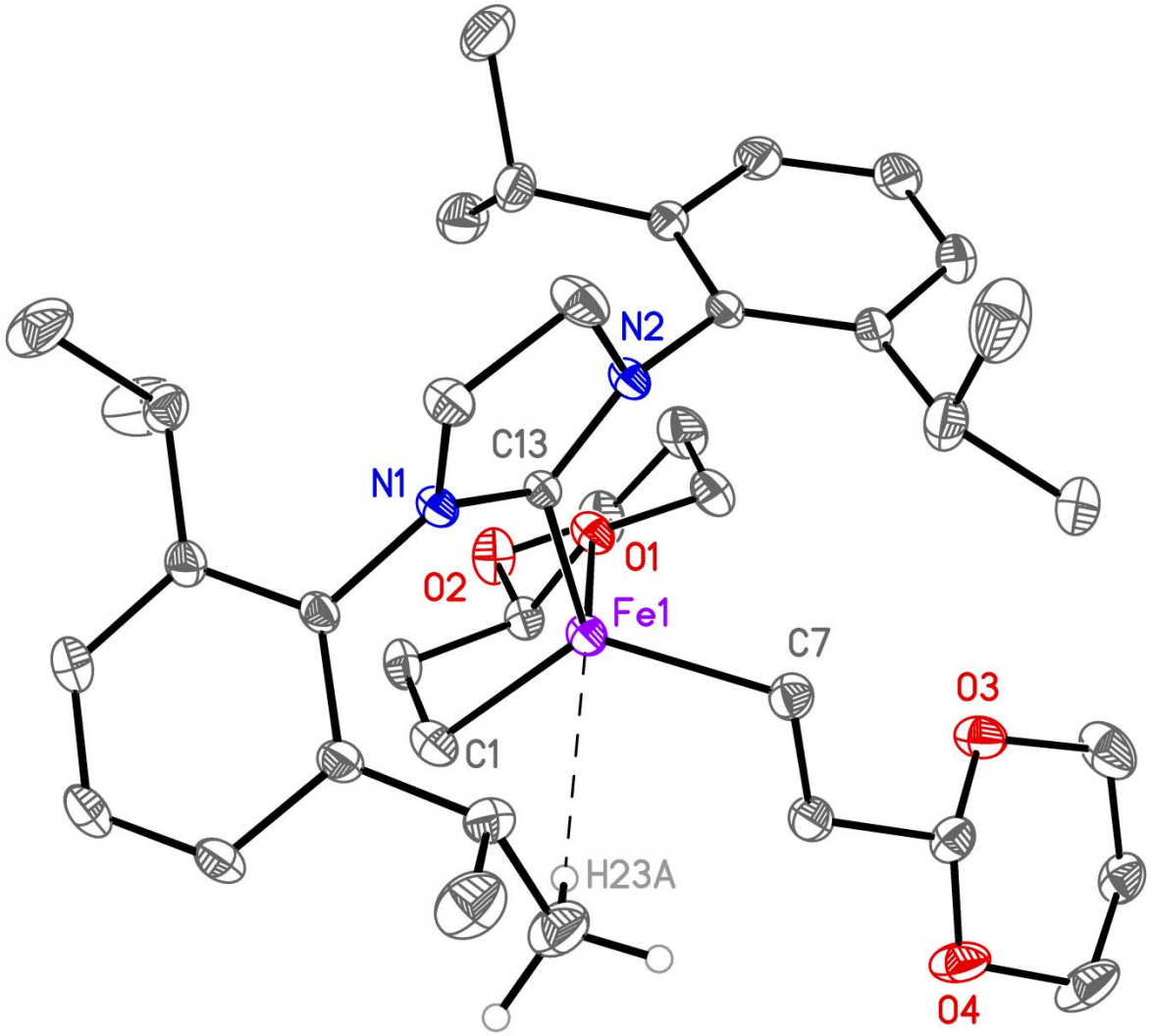

N2
C13
N1
O1
O2
Fe1
C7
O3
C1
H23A
O4

Table 18. Crystal data and structure refinement for neism54.

Identification code	neism54	
Empirical formula	C39 H60 Fe N2 O4	
Formula weight	676.74	
Temperature	100.0(5) K	
Wavelength	0.71073 Å	
Crystal system	triclinic	
Space group	*P*-1	
Unit cell dimensions	*a* = 10.0953(8) Å	α = 88.7501(16)°
	b = 10.9483(8) Å	β = 75.1691(16)°
	c = 17.6570(13) Å	γ = 88.9205(16)°
Volume	1885.9(2) Å^3	
Z	2	
Density (calculated)	1.192 Mg/m^3	
Absorption coefficient	0.440 mm^{-1}	
F(000)	732	
Crystal color, morphology	light yellow, block	
Crystal size	0.45 x 0.34 x 0.20 mm^3	
Theta range for data collection	1.861 to 38.663°	
Index ranges	$-17 \le h \le 17$, $-19 \le k \le 19$, $-30 \le l \le 30$	
Reflections collected	59534	
Independent reflections	20320 [*R*(int) = 0.0356]	
Observed reflections	14762	
Completeness to theta = 37.785°	98.0%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.7476 and 0.6883	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	20320 / 0 / 423	
Goodness-of-fit on F^2	1.047	
Final *R* indices [*I*>2sigma(*I*)]	*R*1 = 0.0484, *wR*2 = 0.1339	
R indices (all data)	*R*1 = 0.0725, *wR*2 = 0.1459	
Largest diff. peak and hole	0.602 and -0.735 e.Å^{-3}	

Table 19. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for neism54. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Fe1	7742(1)	6639(1)	7032(1)	16(1)
O1	8353(1)	7363(1)	8114(1)	19(1)
O2	10173(1)	8414(1)	8377(1)	26(1)
O3	8887(1)	3324(1)	8520(1)	25(1)
O4	10380(1)	2454(1)	7444(1)	32(1)
N1	5597(1)	7843(1)	6297(1)	14(1)
N2	4603(1)	7092(1)	7446(1)	14(1)
C1	9574(1)	7484(1)	6462(1)	22(1)
C2	9966(1)	8311(1)	7055(1)	21(1)
C3	9770(1)	7662(1)	7839(1)	18(1)
C4	8092(1)	6666(1)	8842(1)	23(1)
C5	8549(2)	7392(1)	9452(1)	29(1)
C6	10040(2)	7795(1)	9115(1)	29(1)
C7	7649(1)	4823(1)	7392(1)	20(1)
C8	9064(1)	4214(1)	7261(1)	23(1)
C9	9104(1)	3040(1)	7725(1)	21(1)
C10	8892(2)	2250(2)	8992(1)	36(1)
C11	10233(2)	1548(1)	8720(1)	26(1)
C12	10469(2)	1333(1)	7857(1)	39(1)
C13	5837(1)	7332(1)	6949(1)	13(1)
C14	4131(1)	7941(1)	6310(1)	18(1)
C15	3438(1)	7523(1)	7142(1)	23(1)
C16	6633(1)	8265(1)	5632(1)	15(1)
C17	7271(1)	7441(1)	5048(1)	18(1)
C18	8275(1)	7902(1)	4408(1)	24(1)
C19	8616(1)	9128(1)	4352(1)	28(1)
C20	7958(1)	9929(1)	4930(1)	24(1)
C21	6952(1)	9517(1)	5581(1)	17(1)
C22	6938(1)	6090(1)	5089(1)	21(1)
C23	8171(2)	5305(1)	5175(1)	30(1)
C24	6496(2)	5694(1)	4363(1)	32(1)

C25	6250(1)	10408(1)	6213(1)	19(1)
C26	7261(2)	10973(2)	6604(1)	40(1)
C27	5446(2)	11395(2)	5888(1)	37(1)
C28	4410(1)	6825(1)	8265(1)	14(1)
C29	4484(1)	7782(1)	8771(1)	17(1)
C30	4260(1)	7509(1)	9571(1)	23(1)
C31	3943(1)	6334(1)	9860(1)	26(1)
C32	3852(1)	5412(1)	9357(1)	24(1)
C33	4081(1)	5625(1)	8550(1)	18(1)
C34	4709(1)	9105(1)	8490(1)	21(1)
C35	5915(1)	9685(1)	8723(1)	28(1)
C36	3392(2)	9855(1)	8802(1)	32(1)
C37	3884(1)	4607(1)	8021(1)	24(1)
C38	4395(2)	3357(1)	8243(1)	29(1)
C39	2351(2)	4510(2)	8049(1)	41(1)

Table 20. Bond lengths [Å] and angles [°] for neism54.

Fe(1)-C(7)	2.0707(12)	C(7)-H(7A)	0.9900
Fe(1)-C(1)	2.0885(12)	C(7)-H(7B)	0.9900
Fe(1)-C(13)	2.0920(10)	C(8)-C(9)	1.5141(16)
Fe(1)-O(1)	2.3130(9)	C(8)-H(8A)	0.9900
O(1)-C(3)	1.4305(14)	C(8)-H(8B)	0.9900
O(1)-C(4)	1.4465(15)	C(9)-H(9)	1.0000
O(2)-C(3)	1.4097(15)	C(10)-C(11)	1.516(2)
O(2)-C(6)	1.4318(16)	C(10)-H(10A)	0.9900
O(3)-C(9)	1.4050(15)	C(10)-H(10B)	0.9900
O(3)-C(10)	1.4279(17)	C(11)-C(12)	1.504(2)
O(4)-C(9)	1.4050(16)	C(11)-H(11A)	0.9900
O(4)-C(12)	1.4272(18)	C(11)-H(11B)	0.9900
N(1)-C(13)	1.3443(13)	C(12)-H(12A)	0.9900
N(1)-C(16)	1.4330(13)	C(12)-H(12B)	0.9900
N(1)-C(14)	1.4769(13)	C(14)-C(15)	1.5214(16)
N(2)-C(13)	1.3535(13)	C(14)-H(14A)	0.9900
N(2)-C(28)	1.4343(14)	C(14)-H(14B)	0.9900
N(2)-C(15)	1.4779(14)	C(15)-H(15A)	0.9900
C(1)-C(2)	1.5291(18)	C(15)-H(15B)	0.9900
C(1)-H(1A)	0.9900	C(16)-C(17)	1.4033(15)
C(1)-H(1B)	0.9900	C(16)-C(21)	1.4092(15)
C(2)-C(3)	1.5102(16)	C(17)-C(18)	1.4035(16)
C(2)-H(2A)	0.9900	C(17)-C(22)	1.5191(17)
C(2)-H(2B)	0.9900	C(18)-C(19)	1.3869(19)
C(3)-H(3)	1.0000	C(18)-H(18)	0.9500
C(4)-C(5)	1.5195(19)	C(19)-C(20)	1.3865(19)
C(4)-H(4A)	0.9900	C(19)-H(19)	0.9500
C(4)-H(4B)	0.9900	C(20)-C(21)	1.3981(16)
C(5)-C(6)	1.539(2)	C(20)-H(20)	0.9500
C(5)-H(5A)	0.9900	C(21)-C(25)	1.5218(16)
C(5)-H(5B)	0.9900	C(22)-C(24)	1.5341(19)
C(6)-H(6A)	0.9900	C(22)-C(23)	1.5373(18)
C(6)-H(6B)	0.9900	C(22)-H(22)	1.0000
C(7)-C(8)	1.5299(16)	C(23)-H(23A)	0.9800

C(23)-H(23B)	0.9800	C(37)-H(37)	1.0000
C(23)-H(23C)	0.9800	C(38)-H(38A)	0.9800
C(24)-H(24A)	0.9800	C(38)-H(38B)	0.9800
C(24)-H(24B)	0.9800	C(38)-H(38C)	0.9800
C(24)-H(24C)	0.9800	C(39)-H(39A)	0.9800
C(25)-C(26)	1.5182(19)	C(39)-H(39B)	0.9800
C(25)-C(27)	1.5283(17)	C(39)-H(39C)	0.9800
C(25)-H(25)	1.0000	C(7)-Fe(1)-C(1)	122.78(5)
C(26)-H(26A)	0.9800	C(7)-Fe(1)-C(13)	112.28(4)
C(26)-H(26B)	0.9800	C(1)-Fe(1)-C(13)	121.91(4)
C(26)-H(26C)	0.9800	C(7)-Fe(1)-O(1)	95.51(4)
C(27)-H(27A)	0.9800	C(1)-Fe(1)-O(1)	80.96(4)
C(27)-H(27B)	0.9800	C(13)-Fe(1)-O(1)	112.08(4)
C(27)-H(27C)	0.9800	C(3)-O(1)-C(4)	111.34(9)
C(28)-C(29)	1.4067(16)	C(3)-O(1)-Fe(1)	105.20(6)
C(28)-C(33)	1.4106(15)	C(4)-O(1)-Fe(1)	121.33(7)
C(29)-C(30)	1.3993(16)	C(3)-O(2)-C(6)	111.25(10)
C(29)-C(34)	1.5226(16)	C(9)-O(3)-C(10)	111.40(11)
C(30)-C(31)	1.3861(18)	C(9)-O(4)-C(12)	111.75(12)
C(30)-H(30)	0.9500	C(13)-N(1)-C(16)	125.04(8)
C(31)-C(32)	1.380(2)	C(13)-N(1)-C(14)	114.09(8)
C(31)-H(31)	0.9500	C(16)-N(1)-C(14)	120.88(8)
C(32)-C(33)	1.3986(17)	C(13)-N(2)-C(28)	123.69(9)
C(32)-H(32)	0.9500	C(13)-N(2)-C(15)	113.13(9)
C(33)-C(37)	1.5174(18)	C(28)-N(2)-C(15)	120.33(9)
C(34)-C(36)	1.5330(19)	C(2)-C(1)-Fe(1)	107.32(8)
C(34)-C(35)	1.5338(19)	C(2)-C(1)-H(1A)	110.3
C(34)-H(34)	1.0000	Fe(1)-C(1)-H(1A)	110.3
C(35)-H(35A)	0.9800	C(2)-C(1)-H(1B)	110.3
C(35)-H(35B)	0.9800	Fe(1)-C(1)-H(1B)	110.3
C(35)-H(35C)	0.9800	H(1A)-C(1)-H(1B)	108.5
C(36)-H(36A)	0.9800	C(3)-C(2)-C(1)	110.73(10)
C(36)-H(36B)	0.9800	C(3)-C(2)-H(2A)	109.5
C(36)-H(36C)	0.9800	C(1)-C(2)-H(2A)	109.5
C(37)-C(38)	1.5318(18)	C(3)-C(2)-H(2B)	109.5
C(37)-C(39)	1.541(2)	C(1)-C(2)-H(2B)	109.5

H(2A)-C(2)-H(2B)	108.1	H(8A)-C(8)-H(8B)	107.5
O(2)-C(3)-O(1)	110.59(10)	O(3)-C(9)-O(4)	110.71(10)
O(2)-C(3)-C(2)	110.56(9)	O(3)-C(9)-C(8)	108.69(10)
O(1)-C(3)-C(2)	107.80(9)	O(4)-C(9)-C(8)	109.36(10)
O(2)-C(3)-H(3)	109.3	O(3)-C(9)-H(9)	109.4
O(1)-C(3)-H(3)	109.3	O(4)-C(9)-H(9)	109.4
C(2)-C(3)-H(3)	109.3	C(8)-C(9)-H(9)	109.4
O(1)-C(4)-C(5)	109.35(10)	O(3)-C(10)-C(11)	110.76(12)
O(1)-C(4)-H(4A)	109.8	O(3)-C(10)-H(10A)	109.5
C(5)-C(4)-H(4A)	109.8	C(11)-C(10)-H(10A)	109.5
O(1)-C(4)-H(4B)	109.8	O(3)-C(10)-H(10B)	109.5
C(5)-C(4)-H(4B)	109.8	C(11)-C(10)-H(10B)	109.5
H(4A)-C(4)-H(4B)	108.3	H(10A)-C(10)-H(10B)	108.1
C(4)-C(5)-C(6)	109.37(11)	C(12)-C(11)-C(10)	107.75(12)
C(4)-C(5)-H(5A)	109.8	C(12)-C(11)-H(11A)	110.2
C(6)-C(5)-H(5A)	109.8	C(10)-C(11)-H(11A)	110.2
C(4)-C(5)-H(5B)	109.8	C(12)-C(11)-H(11B)	110.2
C(6)-C(5)-H(5B)	109.8	C(10)-C(11)-H(11B)	110.2
H(5A)-C(5)-H(5B)	108.2	H(11A)-C(11)-H(11B)	108.5
O(2)-C(6)-C(5)	109.95(11)	O(4)-C(12)-C(11)	110.74(11)
O(2)-C(6)-H(6A)	109.7	O(4)-C(12)-H(12A)	109.5
C(5)-C(6)-H(6A)	109.7	C(11)-C(12)-H(12A)	109.5
O(2)-C(6)-H(6B)	109.7	O(4)-C(12)-H(12B)	109.5
C(5)-C(6)-H(6B)	109.7	C(11)-C(12)-H(12B)	109.5
H(6A)-C(6)-H(6B)	108.2	H(12A)-C(12)-H(12B)	108.1
C(8)-C(7)-Fe(1)	112.90(8)	N(1)-C(13)-N(2)	107.12(8)
C(8)-C(7)-H(7A)	109.0	N(1)-C(13)-Fe(1)	124.41(7)
Fe(1)-C(7)-H(7A)	109.0	N(2)-C(13)-Fe(1)	126.67(7)
C(8)-C(7)-H(7B)	109.0	N(1)-C(14)-C(15)	102.12(8)
Fe(1)-C(7)-H(7B)	109.0	N(1)-C(14)-H(14A)	111.3
H(7A)-C(7)-H(7B)	107.8	C(15)-C(14)-H(14A)	111.3
C(9)-C(8)-C(7)	115.06(10)	N(1)-C(14)-H(14B)	111.3
C(9)-C(8)-H(8A)	108.5	C(15)-C(14)-H(14B)	111.3
C(7)-C(8)-H(8A)	108.5	H(14A)-C(14)-H(14B)	109.2
C(9)-C(8)-H(8B)	108.5	N(2)-C(15)-C(14)	102.94(8)
C(7)-C(8)-H(8B)	108.5	N(2)-C(15)-H(15A)	111.2

C(14)-C(15)-H(15A)	111.2	H(24A)-C(24)-H(24B)	109.5
N(2)-C(15)-H(15B)	111.2	C(22)-C(24)-H(24C)	109.5
C(14)-C(15)-H(15B)	111.2	H(24A)-C(24)-H(24C)	109.5
H(15A)-C(15)-H(15B)	109.1	H(24B)-C(24)-H(24C)	109.5
C(17)-C(16)-C(21)	122.23(10)	C(26)-C(25)-C(21)	111.78(11)
C(17)-C(16)-N(1)	119.75(10)	C(26)-C(25)-C(27)	111.00(12)
C(21)-C(16)-N(1)	118.01(9)	C(21)-C(25)-C(27)	111.34(10)
C(16)-C(17)-C(18)	117.49(11)	C(26)-C(25)-H(25)	107.5
C(16)-C(17)-C(22)	123.13(10)	C(21)-C(25)-H(25)	107.5
C(18)-C(17)-C(22)	119.38(10)	C(27)-C(25)-H(25)	107.5
C(19)-C(18)-C(17)	121.14(11)	C(25)-C(26)-H(26A)	109.5
C(19)-C(18)-H(18)	119.4	C(25)-C(26)-H(26B)	109.5
C(17)-C(18)-H(18)	119.4	H(26A)-C(26)-H(26B)	109.5
C(20)-C(19)-C(18)	120.37(11)	C(25)-C(26)-H(26C)	109.5
C(20)-C(19)-H(19)	119.8	H(26A)-C(26)-H(26C)	109.5
C(18)-C(19)-H(19)	119.8	H(26B)-C(26)-H(26C)	109.5
C(19)-C(20)-C(21)	120.78(12)	C(25)-C(27)-H(27A)	109.5
C(19)-C(20)-H(20)	119.6	C(25)-C(27)-H(27B)	109.5
C(21)-C(20)-H(20)	119.6	H(27A)-C(27)-H(27B)	109.5
C(20)-C(21)-C(16)	117.97(11)	C(25)-C(27)-H(27C)	109.5
C(20)-C(21)-C(25)	120.02(11)	H(27A)-C(27)-H(27C)	109.5
C(16)-C(21)-C(25)	122.00(10)	H(27B)-C(27)-H(27C)	109.5
C(17)-C(22)-C(24)	111.60(11)	C(29)-C(28)-C(33)	121.65(10)
C(17)-C(22)-C(23)	111.00(11)	C(29)-C(28)-N(2)	118.92(9)
C(24)-C(22)-C(23)	109.42(11)	C(33)-C(28)-N(2)	119.34(10)
C(17)-C(22)-H(22)	108.2	C(30)-C(29)-C(28)	118.11(10)
C(24)-C(22)-H(22)	108.2	C(30)-C(29)-C(34)	119.06(11)
C(23)-C(22)-H(22)	108.2	C(28)-C(29)-C(34)	122.72(10)
C(22)-C(23)-H(23A)	109.5	C(31)-C(30)-C(29)	120.98(12)
C(22)-C(23)-H(23B)	109.5	C(31)-C(30)-H(30)	119.5
H(23A)-C(23)-H(23B)	109.5	C(29)-C(30)-H(30)	119.5
C(22)-C(23)-H(23C)	109.5	C(32)-C(31)-C(30)	120.02(11)
H(23A)-C(23)-H(23C)	109.5	C(32)-C(31)-H(31)	120.0
H(23B)-C(23)-H(23C)	109.5	C(30)-C(31)-H(31)	120.0
C(22)-C(24)-H(24A)	109.5	C(31)-C(32)-C(33)	121.63(11)
C(22)-C(24)-H(24B)	109.5	C(31)-C(32)-H(32)	119.2

C(33)-C(32)-H(32)	119.2	H(39A)-C(39)-H(39B)	109.5
C(32)-C(33)-C(28)	117.59(11)	C(37)-C(39)-H(39C)	109.5
C(32)-C(33)-C(37)	120.11(10)	H(39A)-C(39)-H(39C)	109.5
C(28)-C(33)-C(37)	122.18(10)	H(39B)-C(39)-H(39C)	109.5
C(29)-C(34)-C(36)	109.81(10)		
C(29)-C(34)-C(35)	112.73(10)		
C(36)-C(34)-C(35)	110.41(11)		
C(29)-C(34)-H(34)	107.9		
C(36)-C(34)-H(34)	107.9		
C(35)-C(34)-H(34)	107.9		
C(34)-C(35)-H(35A)	109.5		
C(34)-C(35)-H(35B)	109.5		
H(35A)-C(35)-H(35B)	109.5		
C(34)-C(35)-H(35C)	109.5		
H(35A)-C(35)-H(35C)	109.5		
H(35B)-C(35)-H(35C)	109.5		
C(34)-C(36)-H(36A)	109.5		
C(34)-C(36)-H(36B)	109.5		
H(36A)-C(36)-H(36B)	109.5		
C(34)-C(36)-H(36C)	109.5		
H(36A)-C(36)-H(36C)	109.5		
H(36B)-C(36)-H(36C)	109.5		
C(33)-C(37)-C(38)	113.49(11)		
C(33)-C(37)-C(39)	109.52(12)		
C(38)-C(37)-C(39)	108.39(11)		
C(33)-C(37)-H(37)	108.4		
C(38)-C(37)-H(37)	108.4		
C(39)-C(37)-H(37)	108.4		
C(37)-C(38)-H(38A)	109.5		
C(37)-C(38)-H(38B)	109.5		
H(38A)-C(38)-H(38B)	109.5		
C(37)-C(38)-H(38C)	109.5		
H(38A)-C(38)-H(38C)	109.5		
H(38B)-C(38)-H(38C)	109.5		
C(37)-C(39)-H(39A)	109.5		
C(37)-C(39)-H(39B)	109.5		

Table 21. Anisotropic displacement parameters ($Å^2$x 10^3) for neism54. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U_{11} + ... + 2 h k a^* b^* U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Fe1	14(1)	18(1)	15(1)	2(1)	-4(1)	1(1)
O1	15(1)	24(1)	17(1)	2(1)	-3(1)	-1(1)
O2	32(1)	24(1)	25(1)	3(1)	-14(1)	-8(1)
O3	26(1)	31(1)	18(1)	2(1)	-4(1)	9(1)
O4	43(1)	32(1)	19(1)	-1(1)	-5(1)	22(1)
N1	11(1)	19(1)	12(1)	3(1)	-3(1)	1(1)
N2	11(1)	19(1)	13(1)	3(1)	-2(1)	-1(1)
C1	18(1)	29(1)	17(1)	3(1)	-3(1)	-1(1)
C2	18(1)	22(1)	22(1)	7(1)	-7(1)	-3(1)
C3	16(1)	20(1)	19(1)	2(1)	-6(1)	-1(1)
C4	24(1)	28(1)	16(1)	3(1)	-3(1)	-4(1)
C5	36(1)	34(1)	17(1)	0(1)	-8(1)	-3(1)
C6	36(1)	32(1)	24(1)	4(1)	-16(1)	-6(1)
C7	18(1)	20(1)	23(1)	2(1)	-6(1)	1(1)
C8	22(1)	23(1)	22(1)	5(1)	-3(1)	5(1)
C9	26(1)	18(1)	20(1)	-1(1)	-10(1)	2(1)
C10	30(1)	48(1)	27(1)	16(1)	-4(1)	7(1)
C11	33(1)	22(1)	25(1)	3(1)	-12(1)	4(1)
C12	66(1)	25(1)	28(1)	-6(1)	-20(1)	22(1)
C13	13(1)	14(1)	11(1)	1(1)	-2(1)	-1(1)
C14	14(1)	24(1)	16(1)	2(1)	-5(1)	1(1)
C15	12(1)	38(1)	18(1)	6(1)	-4(1)	1(1)
C16	14(1)	18(1)	11(1)	3(1)	-2(1)	1(1)
C17	18(1)	20(1)	14(1)	1(1)	-2(1)	2(1)
C18	24(1)	28(1)	15(1)	2(1)	3(1)	4(1)
C19	28(1)	31(1)	19(1)	8(1)	5(1)	-1(1)
C20	27(1)	23(1)	21(1)	7(1)	-2(1)	-3(1)
C21	19(1)	18(1)	14(1)	2(1)	-4(1)	0(1)
C22	23(1)	19(1)	19(1)	-2(1)	-3(1)	2(1)
C23	35(1)	25(1)	30(1)	-3(1)	-12(1)	10(1)
C24	40(1)	29(1)	33(1)	-7(1)	-18(1)	4(1)

C25	25(1)	18(1)	17(1)	0(1)	-7(1)	2(1)
C26	34(1)	49(1)	42(1)	-21(1)	-20(1)	7(1)
C27	50(1)	36(1)	28(1)	-3(1)	-16(1)	23(1)
C28	13(1)	15(1)	13(1)	2(1)	0(1)	1(1)
C29	19(1)	16(1)	15(1)	1(1)	-2(1)	2(1)
C30	30(1)	22(1)	14(1)	-1(1)	-1(1)	5(1)
C31	31(1)	28(1)	14(1)	5(1)	0(1)	6(1)
C32	27(1)	20(1)	20(1)	7(1)	0(1)	1(1)
C33	17(1)	16(1)	18(1)	4(1)	-1(1)	0(1)
C34	28(1)	15(1)	21(1)	0(1)	-6(1)	0(1)
C35	26(1)	23(1)	33(1)	-4(1)	-6(1)	-2(1)
C36	30(1)	19(1)	47(1)	0(1)	-10(1)	4(1)
C37	29(1)	17(1)	27(1)	2(1)	-7(1)	-4(1)
C38	34(1)	18(1)	32(1)	3(1)	-3(1)	-3(1)
C39	36(1)	30(1)	63(1)	6(1)	-24(1)	-9(1)

Table 22. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for neism54.

	x	y	z	U(eq)
H1A	10303	6863	6274	26
H1B	9452	7973	6006	26
H2A	10936	8551	6858	25
H2B	9391	9063	7118	25
H3	10336	6894	7771	21
H4A	8600	5877	8757	28
H4B	7102	6492	9028	28
H5A	8482	6881	9929	34
H5B	7946	8118	9596	34
H6A	10308	8347	9485	35
H6B	10659	7071	9044	35
H7A	7174	4769	7956	24
H7B	7101	4372	7102	24
H8A	9411	4037	6696	27
H8B	9697	4800	7398	27
H9	8360	2491	7661	25
H10A	8123	1722	8959	43
H10B	8758	2480	9546	43
H11A	10994	2025	8819	32
H11B	10186	759	9009	32
H12A	11387	956	7652	46
H12B	9777	761	7772	46
H14A	3905	7402	5919	21
H14B	3866	8793	6209	21
H15A	2940	8208	7454	27
H15B	2790	6855	7145	27
H18	8729	7365	4005	29
H19	9305	9420	3916	33
H20	8193	10768	4882	29
H22	6161	5940	5560	25

H23A	8473	5576	5628	44
H23B	8922	5393	4700	44
H23C	7904	4446	5251	44
H24A	5653	6134	4338	49
H24B	6330	4813	4396	49
H24C	7222	5883	3891	49
H25	5578	9935	6624	23
H26A	7716	10326	6841	59
H26B	6771	11530	7013	59
H26C	7948	11428	6213	59
H27A	4920	11896	6318	55
H27B	4817	11009	5628	55
H27C	6084	11913	5509	55
H30	4325	8138	9921	28
H31	3789	6164	10406	31
H32	3627	4612	9563	28
H34	4916	9118	7904	25
H35A	6760	9228	8489	42
H35B	5747	9662	9294	42
H35C	6010	10535	8533	42
H36A	2631	9473	8650	48
H36B	3515	10687	8582	48
H36C	3188	9886	9375	48
H37	4396	4818	7472	29
H38A	5362	3410	8246	43
H38B	4298	2754	7860	43
H38C	3852	3108	8765	43
H39A	2006	5305	7912	61
H39B	1840	4264	8579	61
H39C	2233	3899	7675	61

Table 23. Torsion angles [°] for neism54.

Fe1-C1-C2-C3	44.02(11)
C6-O2-C3-O1	62.79(13)
C6-O2-C3-C2	-177.89(10)
C4-O1-C3-O2	-62.77(12)
Fe1-O1-C3-O2	164.02(7)
C4-O1-C3-C2	176.26(10)
Fe1-O1-C3-C2	43.05(10)
C1-C2-C3-O2	178.19(9)
C1-C2-C3-O1	-60.82(12)
C3-O1-C4-C5	57.73(13)
Fe1-O1-C4-C5	-177.70(8)
O1-C4-C5-C6	-52.45(14)
C3-O2-C6-C5	-58.01(15)
C4-C5-C6-O2	52.85(16)
Fe1-C7-C8-C9	162.07(9)
C10-O3-C9-O4	-60.76(14)
C10-O3-C9-C8	179.11(11)
C12-O4-C9-O3	60.84(15)
C12-O4-C9-C8	-179.43(12)
C7-C8-C9-O3	-70.32(14)
C7-C8-C9-O4	168.72(11)
C9-O3-C10-C11	57.91(16)
O3-C10-C11-C12	-53.21(18)
C9-O4-C12-C11	-58.00(18)
C10-C11-C12-O4	53.06(19)
C16-N1-C13-N2	178.25(10)
C14-N1-C13-N2	-2.02(12)
C16-N1-C13-Fe1	-16.19(15)
C14-N1-C13-Fe1	163.54(8)
C28-N2-C13-N1	-164.29(9)
C15-N2-C13-N1	-3.45(13)
C28-N2-C13-Fe1	30.58(14)
C15-N2-C13-Fe1	-168.59(8)
C13-N1-C14-C15	6.21(13)

C16-N1-C14-C15	-174.05(10)
C13-N2-C15-C14	7.10(13)
C28-N2-C15-C14	168.65(10)
N1-C14-C15-N2	-7.33(12)
C13-N1-C16-C17	84.96(14)
C14-N1-C16-C17	-94.75(12)
C13-N1-C16-C21	-96.41(12)
C14-N1-C16-C21	83.87(13)
C21-C16-C17-C18	1.23(16)
N1-C16-C17-C18	179.79(10)
C21-C16-C17-C22	-179.69(10)
N1-C16-C17-C22	-1.12(16)
C16-C17-C18-C19	-0.39(18)
C22-C17-C18-C19	-179.51(12)
C17-C18-C19-C20	-0.6(2)
C18-C19-C20-C21	0.7(2)
C19-C20-C21-C16	0.10(19)
C19-C20-C21-C25	179.46(12)
C17-C16-C21-C20	-1.09(16)
N1-C16-C21-C20	-179.68(10)
C17-C16-C21-C25	179.56(10)
N1-C16-C21-C25	0.97(15)
C16-C17-C22-C24	123.88(13)
C18-C17-C22-C24	-57.04(15)
C16-C17-C22-C23	-113.77(12)
C18-C17-C22-C23	65.31(15)
C20-C21-C25-C26	-61.86(16)
C16-C21-C25-C26	117.48(14)
C20-C21-C25-C27	62.92(16)
C16-C21-C25-C27	-117.74(13)
C13-N2-C28-C29	74.33(13)
C15-N2-C28-C29	-85.20(13)
C13-N2-C28-C33	-108.93(12)
C15-N2-C28-C33	91.54(13)
C33-C28-C29-C30	1.80(16)
N2-C28-C29-C30	178.46(10)

C33-C28-C29-C34	-174.30(10)
N2-C28-C29-C34	2.36(15)
C28-C29-C30-C31	-1.41(18)
C34-C29-C30-C31	174.84(12)
C29-C30-C31-C32	0.3(2)
C30-C31-C32-C33	0.5(2)
C31-C32-C33-C28	-0.09(18)
C31-C32-C33-C37	-176.16(12)
C29-C28-C33-C32	-1.06(16)
N2-C28-C33-C32	-177.71(10)
C29-C28-C33-C37	174.92(11)
N2-C28-C33-C37	-1.73(15)
C30-C29-C34-C36	-66.55(15)
C28-C29-C34-C36	109.52(13)
C30-C29-C34-C35	57.01(15)
C28-C29-C34-C35	-126.93(12)
C32-C33-C37-C38	-39.52(16)
C28-C33-C37-C38	144.60(11)
C32-C33-C37-C39	81.74(14)
C28-C33-C37-C39	-94.14(14)