Supporting Information for

Multinuclear Iron-Phenyl Species in Reactions of Simple Iron Salts with PhMgBr: Identification of $Fe_4(\mu\text{-}Ph)_6(THF)_4$ as a Key Reactive Species for Cross-Coupling Catalysis

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1. Experimental Procedures

1.1 Handling temperature sensitive crystals for X-ray diffraction. Temperature sensitive crystals were transferred to a nitrogen purged glovebag in an aluminum pie block containing dry ice. A customized aluminum block containing inlet and outlet nozzles allowed for the passage of liquid nitrogen. The cold aluminum block was placed on the base of the microscope, and was used to cool microscope slides containing SilOil. Aliquots of the cold crystalline material were placed on the cold microscope slides using liquid nitrogen cooled pipettes. Single crystals were transferred from the microscope slide using a goniometer pin that was encased in liquid nitrogen cooled aluminum.

1.2 Collection of temperature sensitive crystalline material for spectroscopic characterization. *Collection of **1a**, **1c**, and **4** for spectroscopic characterization.* Crystalline material was grown in mother liquor as described in the experimental section. A fritted funnel was frozen in liquid nitrogen and placed in a -80 °C cold well. Crystals of **1a** were collected on the glass frit using cold utensils.

*Collection of **1b** and **2a** for spectroscopic characterization.* Crystalline material was grown as described in the experimental section. The mother liquor was removed from the scintillation vial at -30 °C using cold pipettes.

1.3 Preparation of Mössbauer samples. *Preparation of solid Mössbauer samples of **1a**, **1c**, and **4***. Crystalline material was placed in a mortar via cold spatula, where the mortar was placed on top of a pie block sitting in liquid nitrogen. The crystals were crushed in the cold mortar by a pestle that had been cooled in liquid nitrogen. The ground crystalline material was collected by a cold spatula and placed in a Delrin Mössbauer sample cup; the Delrin Mössbauer sample was cooled by placing on top of a pie block sitting in liquid nitrogen. The sample was held in placed by a smaller, inner Delrin cup, which was sealed by grease. Samples were then frozen and stored in liquid nitrogen.

*Preparation of solid Mössabuer samples of **1b** and **2a***. Using a cold spatula, the crystals were then placed in a mortar, which was placed on an aluminum pie block sitting in liquid nitrogen. Crushing of the crystalline material and collection into a Delrin Mössbauer sample cup was performed as described above.

*Preparation of solution Mössbauer samples of **1b** and **2a**.* Crystalline material collected as previously described. Crystalline material was transferred via a cold spatula to a scintillation vial containing a known amount of THF at -30. The crystals were allowed to redissolve in THF, and were then transferred to a Delrin Mössbauer sample cup, which had been cooled by liquid nitrogen. Samples were immediately frozen and stored in liquid nitrogen.

1.4 Preparation of MCD samples. *Preparation of solution MCD samples of **1b** and **2a**.* Crystalline material was collected as previously described. The crystalline material was transferred to a vial containing 2-Me-THF at -80 °C for **1b** and -30 °C for **2a** using a cold spatula. A copper MCD cell was assembled with two quartz discs and a 3 mm rubber gasket. The cell was briefly chilled in liquid nitrogen, and placed at -80 °C. Once the crystalline material was redissolved in 2-Me-THF, the solution was injected into the cell using a cold needle and syringe. The cell was immediately frozen in liquid nitrogen, and a glass was formed. AAS was used to determine the concentration of each complex in solution.

2. Supplementary Data

2.1 ^{57}Fe Mössbauer spectra

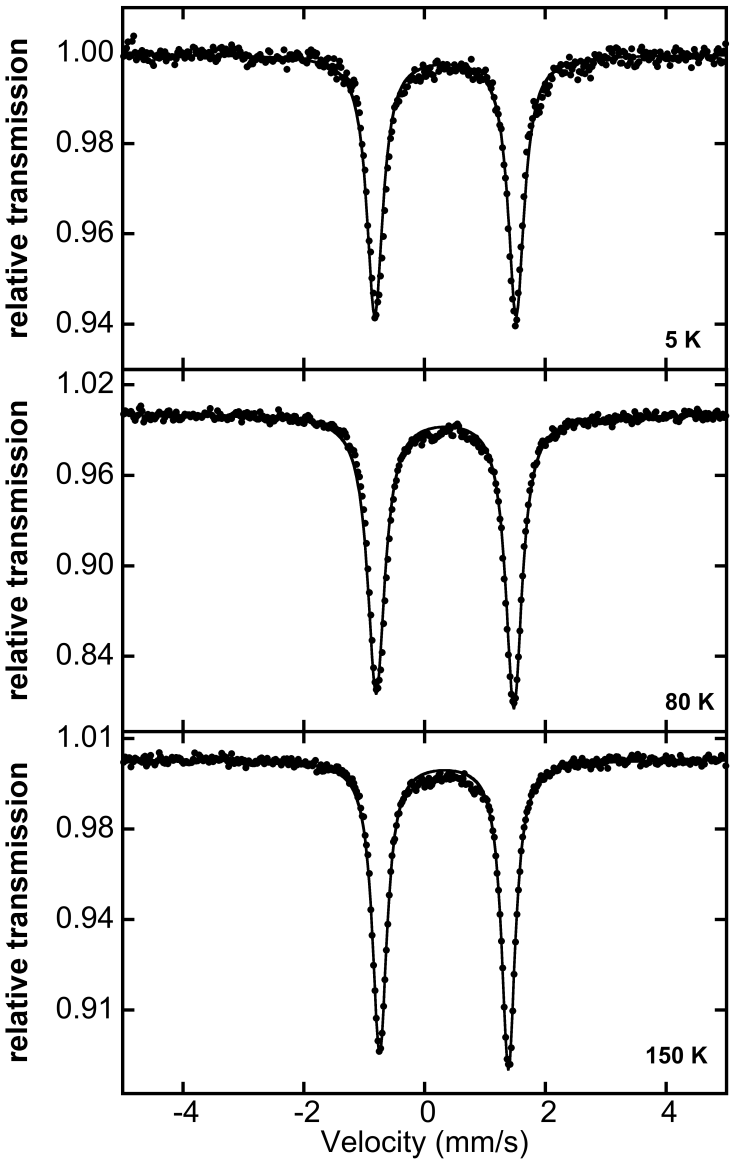


Figure S1. Solution ^{57}Fe Mössbauer spectra of $[Mg(NMP)_6][FePh_2(\mu\text{-}Ph)]_2 \cdot 3.5$ THF (**1b**) in THF at 5 K (top), 80 K (middle), and 150 K (bottom).

Table S1. ^{57}Fe Mössbauer parameters (isomer shift, δ, and quadrupole splitting ΔE_Q) of $[Mg(NMP)_6][FePh_2(\mu\text{-}Ph)]_2 \cdot 3.5$ THF (**1b**) at various temperatures.

Temperature (K)	δ (mm/s)	ΔE_Q (mm/s)
5	0.35	2.34
80	0.34	2.28
150	0.32	2.13

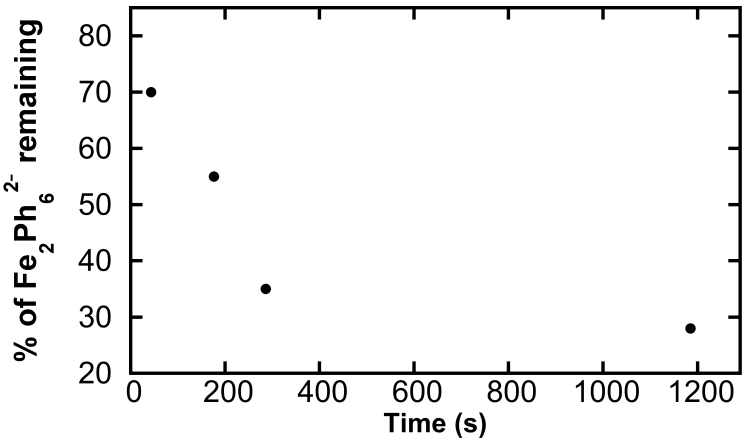


Figure S2. Thermal decay of $[Mg(NMP)_6][FePh_2(\mu\text{-}Ph)]_2 \cdot 3.5$ THF (**1b**) at -20 °C in THF using time-resolved, freeze-quenced ^{57}Fe Mössbauer spectroscopy. ^{57}Fe Mössbauer spectra were collected at 80 K. After 45 seconds, approximately 70% of **1b** remains in solution.

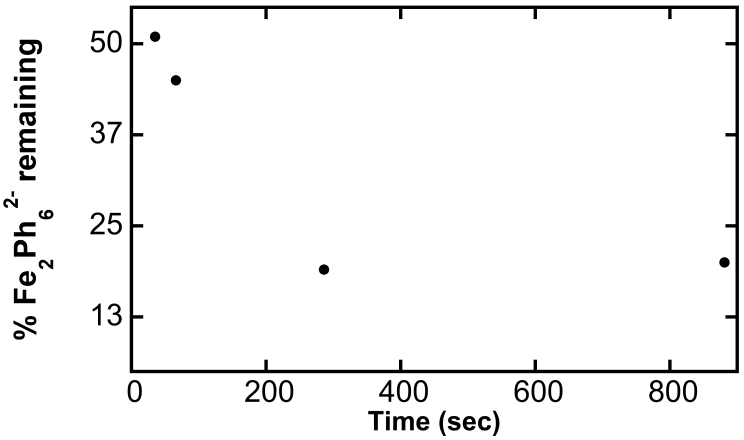


Figure S3. Thermal decay of $[Mg(NMP)_6][FePh_2(\mu\text{-}Ph)]_2 \cdot 3.5$ THF (**1b**) at 0 °C in THF using solution ^{57}Fe Mössbauer spectroscopy. ^{57}Fe Mössbauer spectra were collected at 80 K.

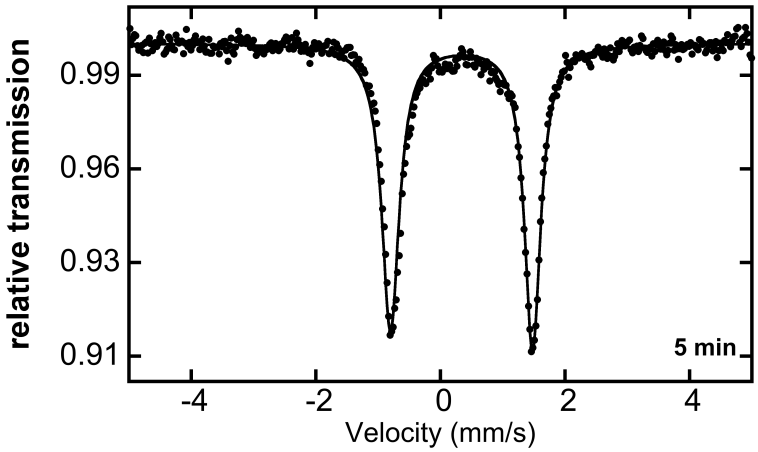


Figure S4. ^{57}Fe Mössbauer spectrum of **1b** reacting with 88.5 equiv* of PhMgBr in THF. After 5 minutes, **1b** remains intact.
*Equivalents determined by AAS.

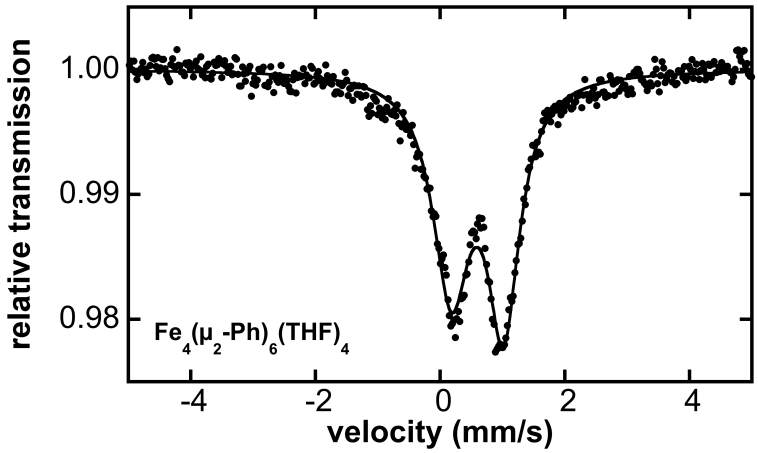


Figure S5. ^{57}Fe Mössbauer spectrum of $Fe_4(\mu\text{-}Ph)_6(THF)_4 \cdot 2$ THF (**2a**) taken at 80 K. The spectrum was fit to one species with $\delta = 0.60$ mm/s and $\Delta E_Q = 0.84$ mm/s.

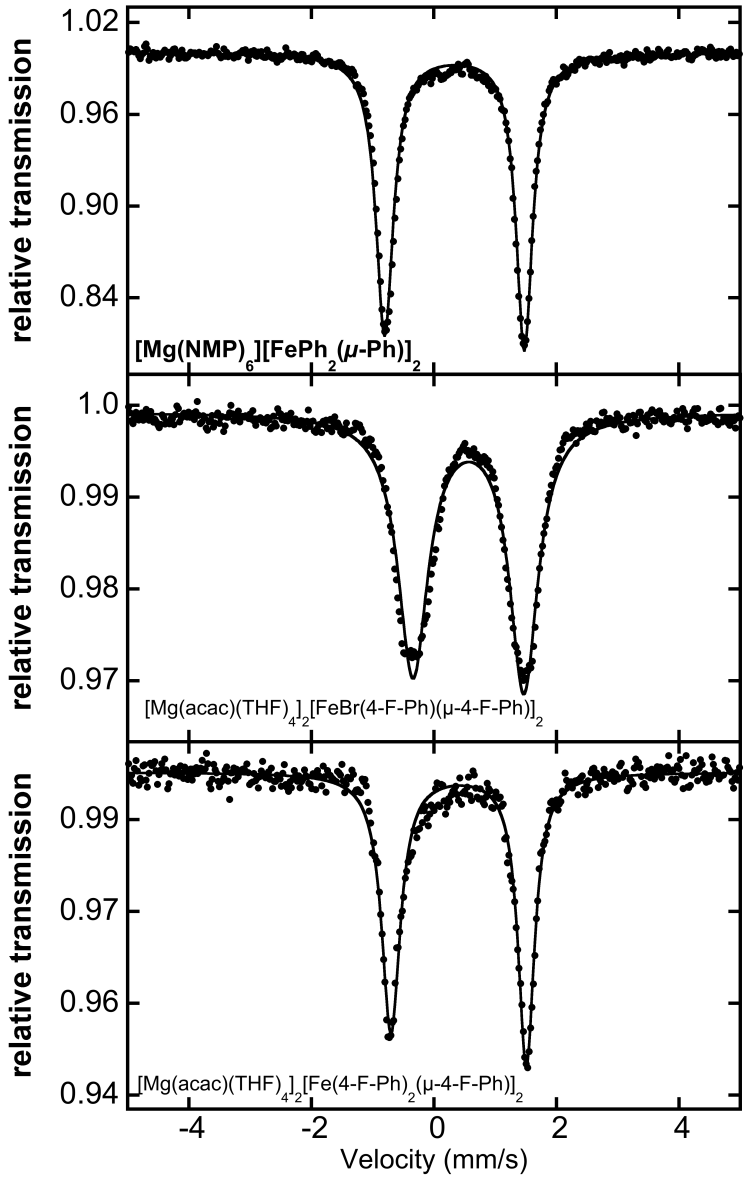


Figure S6. ^{57}Fe Mössbauer spectra comparison of the dimers taken at 80 K. Each dimer was fit to one species, and parameters can be observed in Table S2.

Table S2. Mössbauer parameters (isomer shift, δ, and quadrupole splitting ΔE_Q) of the dimers.

Complex	δ (mm/s)	ΔE_Q (mm/s)
1a / 1b ($[Fe(Ph)_2(\mu\text{-}Ph)]_2)^{2-}$	0.34	2.28
4 $[Mg(acac)(THF)_4]_2[FeBr(4\text{-}F\text{-}Ph)(\mu\text{-}4\text{-}F\text{-}Ph)]_2$	0.56	1.81
1c $[Mg(acac)(THF)_4]_2[Fe(4\text{-}F\text{-}Ph)_2(\mu\text{-}4\text{-}F\text{-}Ph)]_2$	0.40	2.21

2.2 MCD spectra

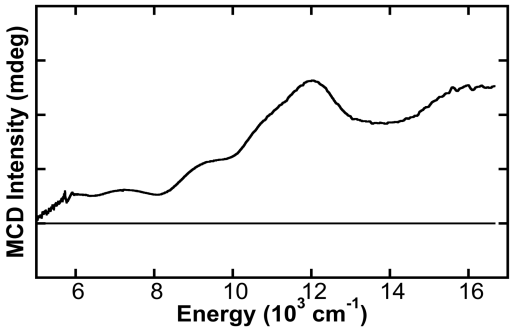


Figure S7. NIR MCD spectra of solution sample of $Fe_4(\mu\text{-}Ph)_6(THF)_4 \cdot 2$ THF (**2a**) taken at 5 K, 7 T.

2.3 EPR spectra

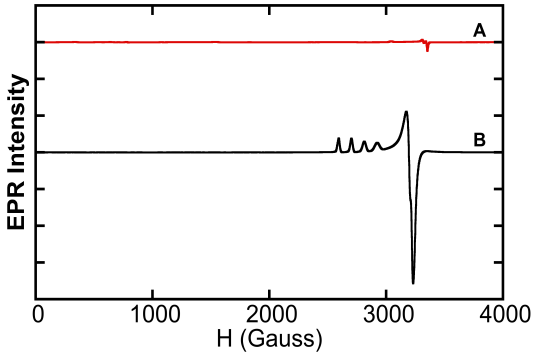


Figure S8. (A) Example 10 K EPR spectrum of $[Mg(NMP)_6][FePh_2(\mu\text{-}Ph)]_2 \cdot 3.5$ THF (**1b**) taken after dissolving **1b** in THF at -80 °C. Less than 1% of total iron in solution in EPR active. Concentration of **1b** was determined by AAS. (B) 10 K EPR spectrum of 3 mM $CuSO_4 \bullet 5H_2O$ in 9:1 methanol:ethanol. All EPR spectra were recorded at 0.10 mW (low power is necessary for the Cu sample signal to not saturate). (A) and (B) are on the same y-axis scale for direct comparison of EPR signal intensity.

Table S3. Quantification of EPR signal using a 3 mM $CuSO_4 \bullet 5H_2O$ standard. Total Fe in solution was determined by AAS. At -20 °C, 70% **1b** remains in solution after 45 seconds. Quantification of the EPR signal should correlate with data from ^{57}Fe Mössbauer spectroscopy. Quantification of the EPR signal at 10 K indicates neither **1b** or the products of decay are EPR active.

Time (s)	% of EPR active Fe in solution
30	< 1%
60	< 1%
150	< 1%
300	< 1%
1200	< 1%

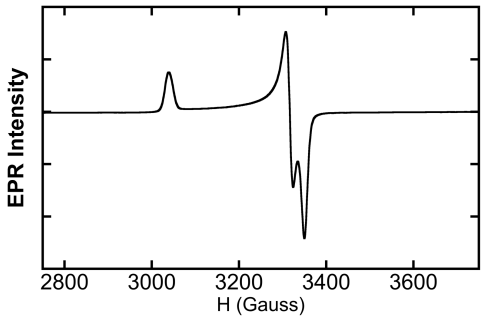


Figure S9. Example 10 K EPR spectrum of the reaction between $FeCl_2$ and PhMgBr in THF at -30 °C. Less than 5% of all iron in solution was determined to be EPR active.

2.4 GC Reaction Studies

Scheme S1. General reaction with $[Mg(NMP)_6][FePh_2(\mu\text{-}Ph)]_2 \cdot 3.5$ THF (**1b**) and $Fe_4(\mu\text{-}Ph)_6(THF)_4 \cdot 2$ THF (**2a**).

Table S4. Stoichiometric reactivity of **1b** and **2a** with bromocyclohexane at various temperatures in THF. All % yields are with respect to bromocyclohexane. Bromocyclohexane equiv are with respect to **1b** or **2a**.

Reaction Temperature (°C)	Catalyst (isolated)	Bromocyclohexane equiv	Time (s)	Bromocyclohexane	Phenylcyclohexane	TON
-80	**1b**	9.4	6	>99	0	--
-80	**1b**	9.4	10800	>99	0	--
-20	**1b**	3.84	5	>99	0	--
-20	**1b**	3.84	1200	>99	0	--
RT	**2a**	15	5	86	5	0.96
RT	**2a**	15	10	77	12	1.81
RT	**2a**	15	30	65	24	3.65
RT	**2a**	15	60	62	29	4.39

Table S5. Reactivity studies of **2a** with bromocyclohexane in the presence of excess PhMgBr in THF at RT. All % yields are with respect to bromocyclohexane. Bromocyclohexane equiv are with respect to **2a**.

Catalyst	Bromocyclohexane (equiv)	PhMgBr (equiv)	Celite filtrations	Time (s)	Bromocyclohexane (% yield)	Phenylcyclohexane (% yield)
2a (isolated)	13	15	N/A	5	68	7
2a (isolated)	13	15	N/A	10	52	28
2a (isolated)	13	15	N/A	60	12	78
2a (isolated)	13	15	N/A	600	0	>95
2a (in-situ)	20	20	0	1200	0	32
2a (in-situ)	20	20	1	1200	0	74
2a (in-situ)	20	20	2	600	0	>95
2a (in-situ)	20	20	2	1200	0	>95

2.5 ^{1}H NMR of $Fe_4(p\text{-tolyl})_6(THF)_3$

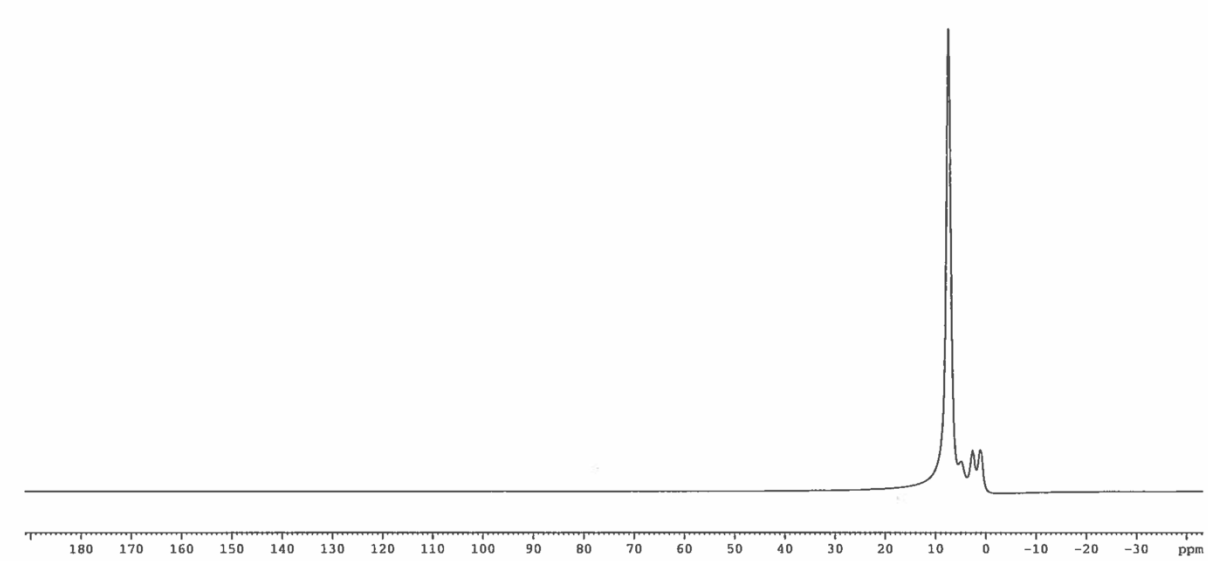


Figure S10. ^{1}H NMR spectrum of crystalline material of $Fe_4(p\text{-tolyl})_6(THF)_4$ dissolved in trifluorotoluene at -30 °C. ^{1}H NMR was recorded on a Bruker Avance 400 MHz. No other resonances were observed downfield from 10 ppm for $Fe_4(p\text{-tolyl})_6(THF)_4$.

3. Single Crystal X-ray Diffraction Data

3.1 CCDC Deposition. All crystal structures reported have been deposited with the Cambridge Crystallographic Data Center (CCDC). The crystal structures have been assigned the following CCDC deposition numbers:

$Mg(acac)(THF)_4]_2[FePh_2(\mu\text{-}Ph)]_2 \cdot 4$ THF (**1a**): 1851552

$[Mg(NMP)_6][FePh_2(\mu\text{-}Ph)]_2 \cdot 3.5$ THF (**1b**): 1851555

$[Mg(acac)(THF)_4]_2[Fe(4\text{-}F\text{-}Ph)_2(\mu\text{-}4\text{-}F\text{-}Ph)]_2 \cdot 2$ THF (**1c**): 1851554

$Fe_4(\mu\text{-}Ph)_6(THF)_4 \cdot 2$ THF (**2a**): 1851553

$Fe_4(\mu\text{-}p\text{-tolyl})_6(THF)_4 \cdot 2$ THF $\cdot C_5H_{12}$ (**2b**): 1851551

$Fe_4(\mu\text{-}p\text{-tolyl})_6(THF)_3 \cdot$ THF (**2c**): 1851559

$Fe_4(\mu\text{-}4\text{-}F\text{-}Ph)_6(THF)_4$ (**2d**): 1851556

$[trans\text{-}Fe(acac)_2(THF)_2]_{0.58} \cdot [trans\text{-}Mg(acac)_2(THF)_2]_{0.42}$ (**3**): 1851558

$[Mg(acac)(THF)_4]_2[FeBr(4\text{-}F\text{-}Ph)(\mu\text{-}4\text{-}F\text{-}Ph)]_2 \cdot 2.5$ THF (**4**): 1851557

3.2 $[Mg(acac)(THF)_4]_2[FePh_2(\mu\text{-}Ph)]_2 \cdot 4\ THF$ (1a)

REFERENCE NUMBER: neisc21

CRYSTAL STRUCTURE REPORT

$C_{94}\ H_{140}\ Fe_2\ Mg_2\ O_{16}$

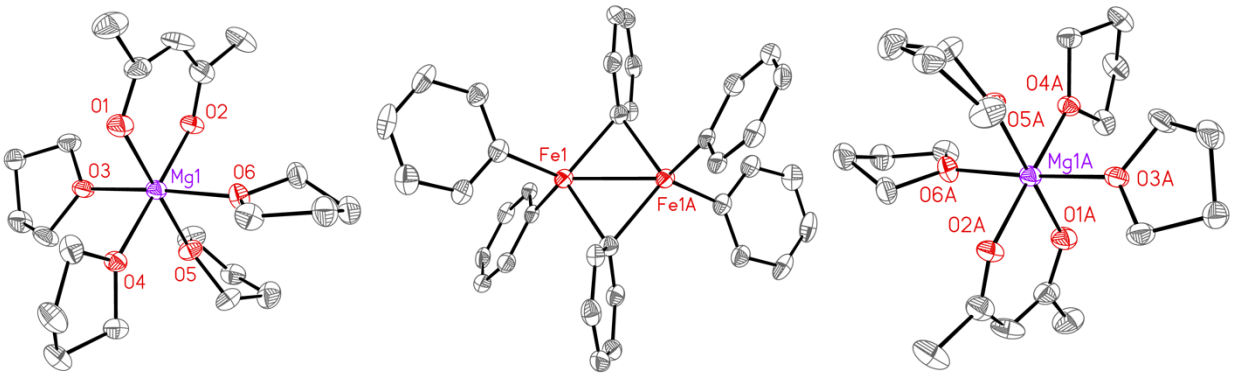
or

$[Mg(acac)(THF)_4]_2[FePh_2(\mu\text{-}Ph)]_2 \cdot 4THF$

Report prepared for:

S. Carpenter, Prof. M. Neidig

September 13, 2016



William W. Brennessel

X-ray Crystallographic Facility

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120 Trustee Road

Rochester, NY 14627

Data collection

A crystal (0.20 x 0.20 x 0.08 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 60 seconds and a detector distance of 3.99 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 4074 strong reflections from the actual data collection after integration.[3] See Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SHELXT-2014/5[4] and refined using SHELXL-2016/4.[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 $R1 = 0.0553$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1301$ (F^2, all data).

Structure description

The structure is the one suggested. The asymmetric unit contains one half of a dianionic iron complex located at a crystallographic inversion center that generates the other half, one cationic magnesium complex in a general position, and two cocrystallized THF solvent molecules in general positions. One Mg-coordinating THF ligand and one cocrystallized THF solvent molecule are modeled as disordered over two positions each (0.88:0.12 and 0.53:0.47, respectively).

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] 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-2016/4*; *Acta. Cryst.* **2015**, C*71*, 3-8.

Some equations of interest:

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

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

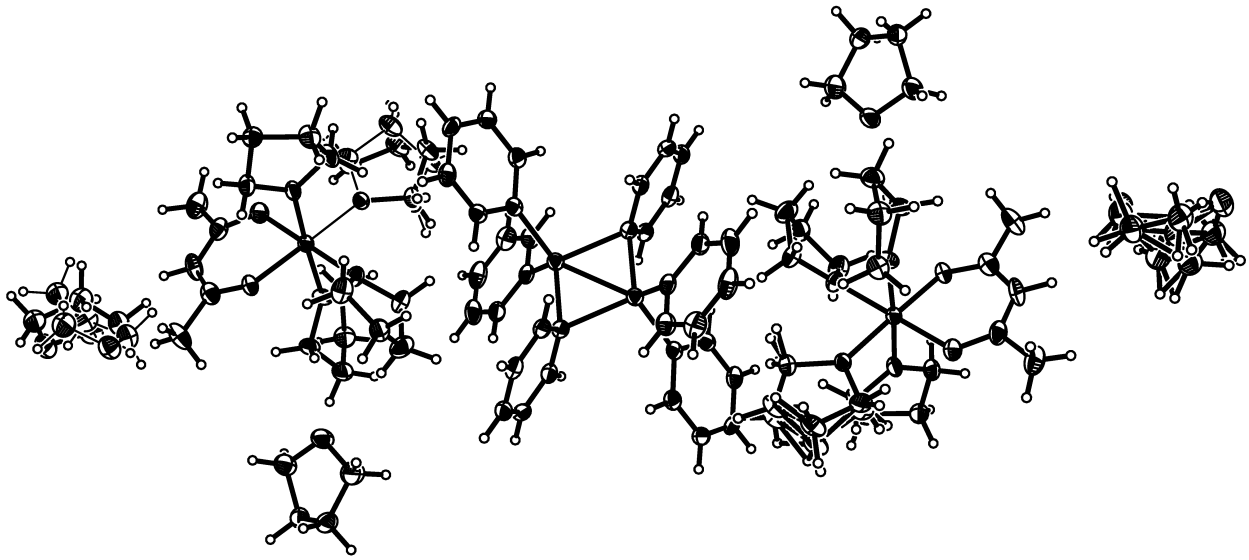
$$wR2 = [\Sigma[w(F_o^2-F_c^2)^2] \ / \ \Sigma\ [w(F_o^2)^2]]^{1/2}$$

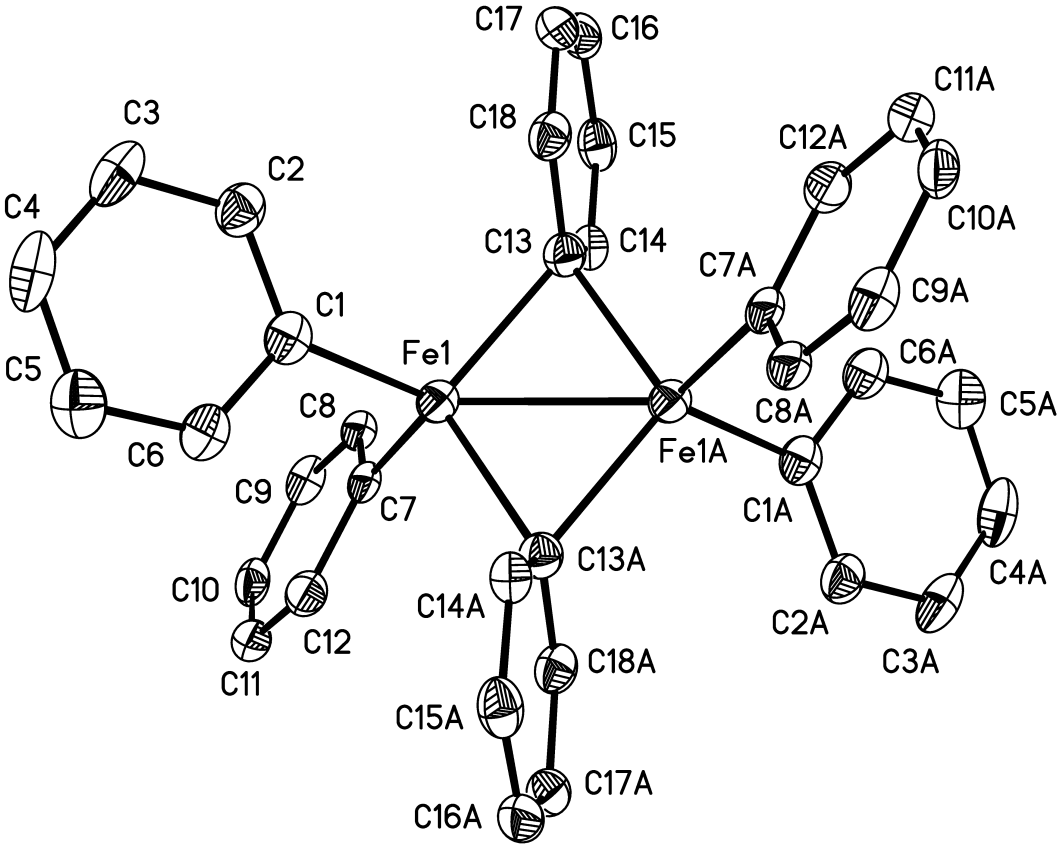
where $w = 1 \ / \ [\sigma^2\ (F_o^2) + (aP)^2 + bP]$ and

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

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

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

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C16
C11A
C3
C18
C15
C12A
C2
C10A
C4
C13
C14
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Fe1
C6A
C5
C8A
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Fe1A
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C7
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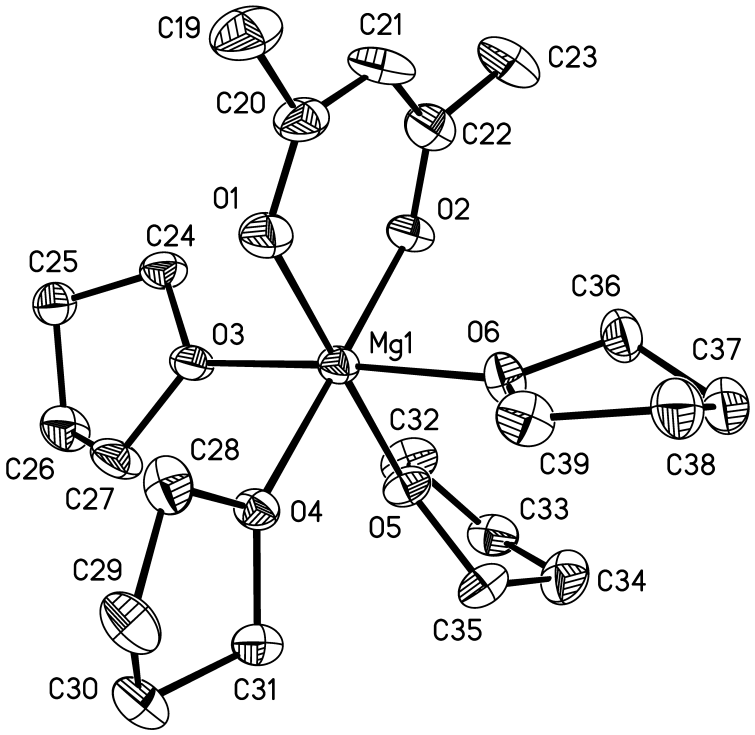
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C21
C23
C20
C22
O1
O2
C24
C25
C36
O3
Mg1
O6
C37
C32
C26
C28
C39
C38
C27
O4
O5
C33
C29
C34
C35
C30
C31

Table S6. Crystal data and structure refinement for neisc21.

Identification code	neisc21	
Empirical formula	C94 H140 Fe2 Mg2 O16	
Formula weight	1686.37	
Temperature	100.0(5) K	
Wavelength	0.71073 Å	
Crystal system	triclinic	
Space group	*P*-1	
Unit cell dimensions	a = 12.4649(12) Å	α = 69.5966(19)°
	b = 12.9212(12) Å	β = 81.444(2)°
	c = 15.2879(15) Å	γ = 88.910(2)°
Volume	2280.7(4) Å^3	
Z	1	
Density (calculated)	1.228 Mg/m^3	
Absorption coefficient	0.395 mm^{-1}	
F(000)	908	
Crystal color, morphology	orange, plate	
Crystal size	0.20 x 0.20 x 0.08 mm^3	
Theta range for data collection	1.653 to 27.481°	
Index ranges	$-16 \leq h \leq 16$, $-16 \leq k \leq 16$, $-19 \leq l \leq 19$	
Reflections collected	32921	
Independent reflections	10437 [R(int) = 0.1039]	
Observed reflections	5850	
Completeness to theta = 27.481°	99.8%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.7456 and 0.6751	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	10437 / 52 / 551	
Goodness-of-fit on F^2	0.985	
Final R indices [I>2sigma(I)]	R1 = 0.0553, wR2 = 0.1050	
R indices (all data)	R1 = 0.1217, wR2 = 0.1301	
Largest diff. peak and hole	0.427 and -0.327 e.Å^{-3}	

Table S7. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for neisc21. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
FE1	9681(1)	4280(1)	4705(1)	22(1)
C1	8405(2)	3123(2)	5364(2)	24(1)
C2	7303(3)	3276(3)	5274(2)	29(1)
C3	6526(3)	2406(3)	5596(2)	37(1)
C4	6835(3)	1331(3)	6020(2)	39(1)
C5	7908(3)	1142(3)	6131(2)	36(1)
C6	8660(3)	2020(2)	5816(2)	29(1)
C7	10500(2)	3927(2)	3558(2)	21(1)
C8	10411(2)	4497(2)	2608(2)	23(1)
C9	10904(2)	4168(3)	1875(2)	28(1)
C10	11513(2)	3228(3)	2053(2)	28(1)
C11	11623(2)	2636(3)	2979(2)	29(1)
C12	11136(2)	2980(2)	3706(2)	27(1)
C13	9144(2)	5987(2)	4293(2)	24(1)
C14	9511(3)	6711(2)	3362(2)	24(1)
C15	8856(3)	7465(2)	2818(2)	27(1)
C16	7779(3)	7551(2)	3167(2)	30(1)
C17	7377(3)	6873(2)	4077(2)	29(1)
C18	8041(2)	6128(2)	4620(2)	25(1)
MG1	6938(1)	2246(1)	1958(1)	21(1)
O1	6442(2)	827(2)	1843(1)	31(1)
O2	6025(2)	3084(2)	967(1)	26(1)
C19	5297(3)	-479(3)	1645(2)	44(1)
C20	5632(3)	706(3)	1470(2)	31(1)
C21	5037(3)	1567(3)	931(2)	38(1)
C22	5248(3)	2693(3)	717(2)	31(1)
C23	4501(3)	3510(3)	168(2)	44(1)
O3	8295(2)	2437(2)	936(1)	25(1)
C24	8268(2)	2727(3)	-65(2)	28(1)
C25	9428(2)	2649(3)	-502(2)	29(1)
C26	10065(3)	2984(3)	138(2)	33(1)

C27	9408(2)	2413(3)	1099(2)	33(1)
O4	7855(2)	1325(2)	2997(1)	22(1)
C28	7931(3)	136(3)	3239(3)	28(1)
C29	8555(3)	-255(3)	4066(3)	33(1)
C30	9310(3)	743(3)	3866(3)	32(1)
C31	8572(4)	1698(3)	3516(3)	27(1)
O4'	7855(2)	1325(2)	2997(1)	22(1)
C28'	8160(30)	218(19)	3020(30)	28(1)
C29'	9140(20)	-47(19)	3523(18)	33(1)
C30'	8880(20)	520(20)	4244(15)	32(1)
C31'	8390(40)	1590(20)	3690(30)	27(1)
O5	7452(2)	3723(2)	2072(1)	25(1)
C32	7690(3)	4731(2)	1260(2)	36(1)
C33	7612(3)	5677(2)	1635(2)	32(1)
C34	6989(3)	5169(3)	2622(2)	40(1)
C35	7343(3)	4016(3)	2917(2)	31(1)
O6	5613(2)	2156(2)	3012(1)	26(1)
C36	4735(2)	2925(3)	2875(2)	31(1)
C37	4229(3)	2814(3)	3865(2)	36(1)
C38	4295(3)	1578(3)	4392(2)	34(1)
C39	5350(3)	1274(2)	3910(2)	29(1)
O7	4348(2)	5599(2)	2228(2)	40(1)
C40	4456(3)	6658(3)	2329(3)	50(1)
C41	3425(3)	7251(3)	2101(2)	37(1)
C42	2597(3)	6302(3)	2373(2)	31(1)
C43	3250(3)	5425(3)	2113(3)	39(1)
O8	643(4)	-14(4)	590(3)	46(1)
C44	1544(5)	359(6)	-165(5)	42(1)
C45	2532(6)	454(7)	276(5)	46(1)
C46	2063(13)	318(11)	1299(8)	42(3)
C47	1073(6)	-424(7)	1464(5)	40(2)
O8'	2264(4)	1576(4)	561(3)	40(1)
C44'	1990(16)	633(11)	1390(9)	42(3)
C45'	1516(8)	-235(8)	1082(6)	40(2)
C46'	2202(7)	8(6)	105(5)	42(1)
C47'	2321(7)	1265(6)	-254(5)	46(1)

Table S8. Bond lengths [Å] and angles [°] for neisc21.

FE1-C(7)	2.082(3)	C(16)-C(17)	1.384(4)
FE1-C(1)	2.084(3)	C(16)-H(16)	0.9500
FE1-C(13)	2.193(3)	C(17)-C(18)	1.385(4)
FE1-C(13)#1	2.212(3)	C(17)-H(17)	0.9500
FE1-FE1#1	2.5175(9)	C(18)-H(18)	0.9500
C(1)-C(6)	1.404(4)	MG1-O(1)	2.018(2)
C(1)-C(2)	1.405(4)	MG1-O(2)	2.020(2)
C(2)-C(3)	1.398(4)	MG1-O(3)	2.079(2)
C(2)-H(2)	0.9500	MG1-O(5)	2.096(2)
C(3)-C(4)	1.387(5)	MG1-O(4)	2.101(2)
C(3)-H(3)	0.9500	MG1-O(4')	2.101(2)
C(4)-C(5)	1.379(5)	MG1-O(6)	2.102(2)
C(4)-H(4)	0.9500	O(1)-C(20)	1.268(4)
C(5)-C(6)	1.386(4)	O(2)-C(22)	1.266(4)
C(5)-H(5)	0.9500	C(19)-C(20)	1.515(4)
C(6)-H(6)	0.9500	C(19)-H(19A)	0.9800
C(7)-C(8)	1.402(4)	C(19)-H(19B)	0.9800
C(7)-C(12)	1.414(4)	C(19)-H(19C)	0.9800
C(8)-C(9)	1.390(4)	C(20)-C(21)	1.407(5)
C(8)-H(8)	0.9500	C(21)-C(22)	1.396(4)
C(9)-C(10)	1.385(4)	C(21)-H(21)	0.9500
C(9)-H(9)	0.9500	C(22)-C(23)	1.504(4)
C(10)-C(11)	1.381(4)	C(23)-H(23A)	0.9800
C(10)-H(10)	0.9500	C(23)-H(23B)	0.9800
C(11)-C(12)	1.391(4)	C(23)-H(23C)	0.9800
C(11)-H(11)	0.9500	O(3)-C(27)	1.443(3)
C(12)-H(12)	0.9500	O(3)-C(24)	1.449(3)
C(13)-C(14)	1.415(4)	C(24)-C(25)	1.516(4)
C(13)-C(18)	1.422(4)	C(24)-H(24A)	0.9900
C(13)-FE1#1	2.212(3)	C(24)-H(24B)	0.9900
C(14)-C(15)	1.383(4)	C(25)-C(26)	1.521(4)
C(14)-H(14)	0.9500	C(25)-H(25A)	0.9900
C(15)-C(16)	1.386(4)	C(25)-H(25B)	0.9900
C(15)-H(15)	0.9500	C(26)-C(27)	1.510(4)

C(26)-H(26A)	0.9900	C(33)-H(33A)	0.9900
C(26)-H(26B)	0.9900	C(33)-H(33B)	0.9900
C(27)-H(27A)	0.9900	C(34)-C(35)	1.477(4)
C(27)-H(27B)	0.9900	C(34)-H(34A)	0.9900
O(4)-C(28)	1.455(4)	C(34)-H(34B)	0.9900
O(4)-C(31)	1.469(4)	C(35)-H(35A)	0.9900
C(28)-C(29)	1.512(5)	C(35)-H(35B)	0.9900
C(28)-H(28A)	0.9900	O(6)-C(39)	1.442(3)
C(28)-H(28B)	0.9900	O(6)-C(36)	1.454(3)
C(29)-C(30)	1.526(5)	C(36)-C(37)	1.509(4)
C(29)-H(29A)	0.9900	C(36)-H(36A)	0.9900
C(29)-H(29B)	0.9900	C(36)-H(36B)	0.9900
C(30)-C(31)	1.516(5)	C(37)-C(38)	1.524(4)
C(30)-H(30A)	0.9900	C(37)-H(37A)	0.9900
C(30)-H(30B)	0.9900	C(37)-H(37B)	0.9900
C(31)-H(31A)	0.9900	C(38)-C(39)	1.523(4)
C(31)-H(31B)	0.9900	C(38)-H(38A)	0.9900
O(4')-C(28')	1.466(15)	C(38)-H(38B)	0.9900
O(4')-C(31')	1.468(15)	C(39)-H(39A)	0.9900
C(28')-C(29')	1.516(16)	C(39)-H(39B)	0.9900
C(28')-H(28C)	0.9900	O(7)-C(43)	1.436(4)
C(28')-H(28D)	0.9900	O(7)-C(40)	1.440(4)
C(29')-C(30')	1.516(18)	C(40)-C(41)	1.506(5)
C(29')-H(29C)	0.9900	C(40)-H(40A)	0.9900
C(29')-H(29D)	0.9900	C(40)-H(40B)	0.9900
C(30')-C(31')	1.517(16)	C(41)-C(42)	1.518(4)
C(30')-H(30C)	0.9900	C(41)-H(41A)	0.9900
C(30')-H(30D)	0.9900	C(41)-H(41B)	0.9900
C(31')-H(31C)	0.9900	C(42)-C(43)	1.513(4)
C(31')-H(31D)	0.9900	C(42)-H(42A)	0.9900
O(5)-C(32)	1.450(3)	C(42)-H(42B)	0.9900
O(5)-C(35)	1.456(3)	C(43)-H(43A)	0.9900
C(32)-C(33)	1.516(4)	C(43)-H(43B)	0.9900
C(32)-H(32A)	0.9900	O(8)-C(47)	1.437(9)
C(32)-H(32B)	0.9900	O(8)-C(44)	1.439(7)
C(33)-C(34)	1.514(4)	C(44)-C(45)	1.516(8)

C(44)-H(44A)	0.9900	C(3)-C(2)-C(1)	123.1(3)
C(44)-H(44B)	0.9900	C(3)-C(2)-H(2)	118.4
C(45)-C(46)	1.536(12)	C(1)-C(2)-H(2)	118.4
C(45)-H(45A)	0.9900	C(4)-C(3)-C(2)	119.9(3)
C(45)-H(45B)	0.9900	C(4)-C(3)-H(3)	120.0
C(46)-C(47)	1.513(11)	C(2)-C(3)-H(3)	120.0
C(46)-H(46A)	0.9900	C(5)-C(4)-C(3)	119.0(3)
C(46)-H(46B)	0.9900	C(5)-C(4)-H(4)	120.5
C(47)-H(47A)	0.9900	C(3)-C(4)-H(4)	120.5
C(47)-H(47B)	0.9900	C(4)-C(5)-C(6)	120.0(3)
O(8')-C(44')	1.421(13)	C(4)-C(5)-H(5)	120.0
O(8')-C(47')	1.430(8)	C(6)-C(5)-H(5)	120.0
C(44')-C(45')	1.519(11)	C(5)-C(6)-C(1)	123.9(3)
C(44')-H(44C)	0.9900	C(5)-C(6)-H(6)	118.1
C(44')-H(44D)	0.9900	C(1)-C(6)-H(6)	118.1
C(45')-C(46')	1.537(10)	C(8)-C(7)-C(12)	113.8(3)
C(45')-H(45C)	0.9900	C(8)-C(7)-FE1	125.7(2)
C(45')-H(45D)	0.9900	C(12)-C(7)-FE1	120.2(2)
C(46')-C(47')	1.524(9)	C(9)-C(8)-C(7)	123.3(3)
C(46')-H(46C)	0.9900	C(9)-C(8)-H(8)	118.4
C(46')-H(46D)	0.9900	C(7)-C(8)-H(8)	118.4
C(47')-H(47C)	0.9900	C(10)-C(9)-C(8)	120.9(3)
C(47')-H(47D)	0.9900	C(10)-C(9)-H(9)	119.5
C(7)-FE1-C(1)	108.82(11)	C(8)-C(9)-H(9)	119.5
C(7)-FE1-C(13)	111.93(11)	C(11)-C(10)-C(9)	118.0(3)
C(1)-FE1-C(13)	112.69(12)	C(11)-C(10)-H(10)	121.0
C(7)-FE1-C(13)#1	105.53(11)	C(9)-C(10)-H(10)	121.0
C(1)-FE1-C(13)#1	107.24(11)	C(10)-C(11)-C(12)	120.6(3)
C(13)-FE1-C(13)#1	110.28(9)	C(10)-C(11)-H(11)	119.7
C(7)-FE1-FE1#1	124.09(8)	C(12)-C(11)-H(11)	119.7
C(1)-FE1-FE1#1	126.61(8)	C(11)-C(12)-C(7)	123.4(3)
C(13)-FE1-FE1#1	55.49(8)	C(11)-C(12)-H(12)	118.3
C(13)#1-FE1-FE1#1	54.79(8)	C(7)-C(12)-H(12)	118.3
C(6)-C(1)-C(2)	114.1(3)	C(14)-C(13)-C(18)	113.3(3)
C(6)-C(1)-FE1	118.1(2)	C(14)-C(13)-FE1	118.1(2)
C(2)-C(1)-FE1	126.8(2)	C(18)-C(13)-FE1	116.1(2)

C(14)-C(13)-FE1#1	115.4(2)	O(4)-MG1-O(6)	90.04(8)
C(18)-C(13)-FE1#1	117.7(2)	O(4')-MG1-O(6)	90.04(8)
FE1-C(13)-FE1#1	69.71(9)	C(20)-O(1)-MG1	125.9(2)
C(15)-C(14)-C(13)	123.4(3)	C(22)-O(2)-MG1	126.8(2)
C(15)-C(14)-H(14)	118.3	C(20)-C(19)-H(19A)	109.5
C(13)-C(14)-H(14)	118.3	C(20)-C(19)-H(19B)	109.5
C(14)-C(15)-C(16)	120.9(3)	H(19A)-C(19)-H(19B)	109.5
C(14)-C(15)-H(15)	119.6	C(20)-C(19)-H(19C)	109.5
C(16)-C(15)-H(15)	119.6	H(19A)-C(19)-H(19C)	109.5
C(17)-C(16)-C(15)	118.4(3)	H(19B)-C(19)-H(19C)	109.5
C(17)-C(16)-H(16)	120.8	O(1)-C(20)-C(21)	125.5(3)
C(15)-C(16)-H(16)	120.8	O(1)-C(20)-C(19)	115.4(3)
C(16)-C(17)-C(18)	120.3(3)	C(21)-C(20)-C(19)	119.1(3)
C(16)-C(17)-H(17)	119.9	C(22)-C(21)-C(20)	125.4(3)
C(18)-C(17)-H(17)	119.9	C(22)-C(21)-H(21)	117.3
C(17)-C(18)-C(13)	123.8(3)	C(20)-C(21)-H(21)	117.3
C(17)-C(18)-H(18)	118.1	O(2)-C(22)-C(21)	124.4(3)
C(13)-C(18)-H(18)	118.1	O(2)-C(22)-C(23)	116.9(3)
O(1)-MG1-O(2)	88.87(9)	C(21)-C(22)-C(23)	118.8(3)
O(1)-MG1-O(3)	92.77(9)	C(22)-C(23)-H(23A)	109.5
O(2)-MG1-O(3)	91.39(9)	C(22)-C(23)-H(23B)	109.5
O(1)-MG1-O(5)	179.81(10)	H(23A)-C(23)-H(23B)	109.5
O(2)-MG1-O(5)	90.96(8)	C(22)-C(23)-H(23C)	109.5
O(3)-MG1-O(5)	87.15(8)	H(23A)-C(23)-H(23C)	109.5
O(1)-MG1-O(4)	89.28(8)	H(23B)-C(23)-H(23C)	109.5
O(2)-MG1-O(4)	178.02(9)	C(27)-O(3)-C(24)	109.5(2)
O(3)-MG1-O(4)	89.38(8)	C(27)-O(3)-MG1	125.33(16)
O(5)-MG1-O(4)	90.89(8)	C(24)-O(3)-MG1	124.91(17)
O(1)-MG1-O(4')	89.28(8)	O(3)-C(24)-C(25)	105.5(2)
O(2)-MG1-O(4')	178.02(9)	O(3)-C(24)-H(24A)	110.6
O(3)-MG1-O(4')	89.38(8)	C(25)-C(24)-H(24A)	110.6
O(5)-MG1-O(4')	90.89(8)	O(3)-C(24)-H(24B)	110.6
O(1)-MG1-O(6)	91.58(9)	C(25)-C(24)-H(24B)	110.6
O(2)-MG1-O(6)	89.33(9)	H(24A)-C(24)-H(24B)	108.8
O(3)-MG1-O(6)	175.60(9)	C(24)-C(25)-C(26)	101.5(2)
O(5)-MG1-O(6)	88.50(8)	C(24)-C(25)-H(25A)	111.5

C(26)-C(25)-H(25A)	111.5	H(30A)-C(30)-H(30B)	109.2
C(24)-C(25)-H(25B)	111.5	O(4)-C(31)-C(30)	104.8(3)
C(26)-C(25)-H(25B)	111.5	O(4)-C(31)-H(31A)	110.8
H(25A)-C(25)-H(25B)	109.3	C(30)-C(31)-H(31A)	110.8
C(27)-C(26)-C(25)	101.9(2)	O(4)-C(31)-H(31B)	110.8
C(27)-C(26)-H(26A)	111.4	C(30)-C(31)-H(31B)	110.8
C(25)-C(26)-H(26A)	111.4	H(31A)-C(31)-H(31B)	108.9
C(27)-C(26)-H(26B)	111.4	C(28')-O(4')-C(31')	108.3(13)
C(25)-C(26)-H(26B)	111.4	C(28')-O(4')-MG1	117.8(10)
H(26A)-C(26)-H(26B)	109.3	C(31')-O(4')-MG1	133.6(9)
O(3)-C(27)-C(26)	105.3(2)	O(4')-C(28')-C(29')	105.7(13)
O(3)-C(27)-H(27A)	110.7	O(4')-C(28')-H(28C)	110.6
C(26)-C(27)-H(27A)	110.7	C(29')-C(28')-H(28C)	110.6
O(3)-C(27)-H(27B)	110.7	O(4')-C(28')-H(28D)	110.6
C(26)-C(27)-H(27B)	110.7	C(29')-C(28')-H(28D)	110.6
H(27A)-C(27)-H(27B)	108.8	H(28C)-C(28')-H(28D)	108.7
C(28)-O(4)-C(31)	108.8(3)	C(30')-C(29')-C(28')	101.5(14)
C(28)-O(4)-MG1	120.95(19)	C(30')-C(29')-H(29C)	111.5
C(31)-O(4)-MG1	130.0(2)	C(28')-C(29')-H(29C)	111.5
O(4)-C(28)-C(29)	106.2(3)	C(30')-C(29')-H(29D)	111.5
O(4)-C(28)-H(28A)	110.5	C(28')-C(29')-H(29D)	111.5
C(29)-C(28)-H(28A)	110.5	H(29C)-C(29')-H(29D)	109.3
O(4)-C(28)-H(28B)	110.5	C(29')-C(30')-C(31')	102.8(15)
C(29)-C(28)-H(28B)	110.5	C(29')-C(30')-H(30C)	111.2
H(28A)-C(28)-H(28B)	108.7	C(31')-C(30')-H(30C)	111.2
C(28)-C(29)-C(30)	101.7(3)	C(29')-C(30')-H(30D)	111.2
C(28)-C(29)-H(29A)	111.4	C(31')-C(30')-H(30D)	111.2
C(30)-C(29)-H(29A)	111.4	H(30C)-C(30')-H(30D)	109.1
C(28)-C(29)-H(29B)	111.4	O(4')-C(31')-C(30')	105.6(13)
C(30)-C(29)-H(29B)	111.4	O(4')-C(31')-H(31C)	110.6
H(29A)-C(29)-H(29B)	109.3	C(30')-C(31')-H(31C)	110.6
C(31)-C(30)-C(29)	102.1(3)	O(4')-C(31')-H(31D)	110.6
C(31)-C(30)-H(30A)	111.4	C(30')-C(31')-H(31D)	110.6
C(29)-C(30)-H(30A)	111.4	H(31C)-C(31')-H(31D)	108.8
C(31)-C(30)-H(30B)	111.4	C(32)-O(5)-C(35)	107.8(2)
C(29)-C(30)-H(30B)	111.4	C(32)-O(5)-MG1	122.41(17)

C(35)-O(5)-MG1	127.66(17)	C(38)-C(37)-H(37A)	111.4
O(5)-C(32)-C(33)	106.7(2)	C(36)-C(37)-H(37B)	111.4
O(5)-C(32)-H(32A)	110.4	C(38)-C(37)-H(37B)	111.4
C(33)-C(32)-H(32A)	110.4	H(37A)-C(37)-H(37B)	109.2
O(5)-C(32)-H(32B)	110.4	C(39)-C(38)-C(37)	103.7(2)
C(33)-C(32)-H(32B)	110.4	C(39)-C(38)-H(38A)	111.0
H(32A)-C(32)-H(32B)	108.6	C(37)-C(38)-H(38A)	111.0
C(34)-C(33)-C(32)	103.8(2)	C(39)-C(38)-H(38B)	111.0
C(34)-C(33)-H(33A)	111.0	C(37)-C(38)-H(38B)	111.0
C(32)-C(33)-H(33A)	111.0	H(38A)-C(38)-H(38B)	109.0
C(34)-C(33)-H(33B)	111.0	O(6)-C(39)-C(38)	106.2(2)
C(32)-C(33)-H(33B)	111.0	O(6)-C(39)-H(39A)	110.5
H(33A)-C(33)-H(33B)	109.0	C(38)-C(39)-H(39A)	110.5
C(35)-C(34)-C(33)	102.7(3)	O(6)-C(39)-H(39B)	110.5
C(35)-C(34)-H(34A)	111.2	C(38)-C(39)-H(39B)	110.5
C(33)-C(34)-H(34A)	111.2	H(39A)-C(39)-H(39B)	108.7
C(35)-C(34)-H(34B)	111.2	C(43)-O(7)-C(40)	109.3(2)
C(33)-C(34)-H(34B)	111.2	O(7)-C(40)-C(41)	106.8(3)
H(34A)-C(34)-H(34B)	109.1	O(7)-C(40)-H(40A)	110.4
O(5)-C(35)-C(34)	105.4(2)	C(41)-C(40)-H(40A)	110.4
O(5)-C(35)-H(35A)	110.7	O(7)-C(40)-H(40B)	110.4
C(34)-C(35)-H(35A)	110.7	C(41)-C(40)-H(40B)	110.4
O(5)-C(35)-H(35B)	110.7	H(40A)-C(40)-H(40B)	108.6
C(34)-C(35)-H(35B)	110.7	C(40)-C(41)-C(42)	102.4(3)
H(35A)-C(35)-H(35B)	108.8	C(40)-C(41)-H(41A)	111.3
C(39)-O(6)-C(36)	109.4(2)	C(42)-C(41)-H(41A)	111.3
C(39)-O(6)-MG1	126.69(17)	C(40)-C(41)-H(41B)	111.3
C(36)-O(6)-MG1	123.39(17)	C(42)-C(41)-H(41B)	111.3
O(6)-C(36)-C(37)	104.0(2)	H(41A)-C(41)-H(41B)	109.2
O(6)-C(36)-H(36A)	111.0	C(43)-C(42)-C(41)	102.7(3)
C(37)-C(36)-H(36A)	111.0	C(43)-C(42)-H(42A)	111.2
O(6)-C(36)-H(36B)	111.0	C(41)-C(42)-H(42A)	111.2
C(37)-C(36)-H(36B)	111.0	C(43)-C(42)-H(42B)	111.2
H(36A)-C(36)-H(36B)	109.0	C(41)-C(42)-H(42B)	111.2
C(36)-C(37)-C(38)	102.0(3)	H(42A)-C(42)-H(42B)	109.1
C(36)-C(37)-H(37A)	111.4	O(7)-C(43)-C(42)	106.1(3)

O(7)-C(43)-H(43A)	110.5	C(46)-C(47)-H(47B)	110.9
C(42)-C(43)-H(43A)	110.5	H(47A)-C(47)-H(47B)	108.9
O(7)-C(43)-H(43B)	110.5	C(44')-O(8')-C(47')	109.7(6)
C(42)-C(43)-H(43B)	110.5	O(8')-C(44')-C(45')	106.4(9)
H(43A)-C(43)-H(43B)	108.7	O(8')-C(44')-H(44C)	110.4
C(47)-O(8)-C(44)	107.9(5)	C(45')-C(44')-H(44C)	110.4
O(8)-C(44)-C(45)	107.1(5)	O(8')-C(44')-H(44D)	110.4
O(8)-C(44)-H(44A)	110.3	C(45')-C(44')-H(44D)	110.4
C(45)-C(44)-H(44A)	110.3	H(44C)-C(44')-H(44D)	108.6
O(8)-C(44)-H(44B)	110.3	C(44')-C(45')-C(46')	100.5(8)
C(45)-C(44)-H(44B)	110.3	C(44')-C(45')-H(45C)	111.7
H(44A)-C(44)-H(44B)	108.6	C(46')-C(45')-H(45C)	111.7
C(44)-C(45)-C(46)	104.0(7)	C(44')-C(45')-H(45D)	111.7
C(44)-C(45)-H(45A)	111.0	C(46')-C(45')-H(45D)	111.7
C(46)-C(45)-H(45A)	111.0	H(45C)-C(45')-H(45D)	109.4
C(44)-C(45)-H(45B)	111.0	C(47')-C(46')-C(45')	101.8(6)
C(46)-C(45)-H(45B)	111.0	C(47')-C(46')-H(46C)	111.4
H(45A)-C(45)-H(45B)	109.0	C(45')-C(46')-H(46C)	111.4
C(47)-C(46)-C(45)	101.2(7)	C(47')-C(46')-H(46D)	111.4
C(47)-C(46)-H(46A)	111.5	C(45')-C(46')-H(46D)	111.4
C(45)-C(46)-H(46A)	111.5	H(46C)-C(46')-H(46D)	109.3
C(47)-C(46)-H(46B)	111.5	O(8')-C(47')-C(46')	106.5(6)
C(45)-C(46)-H(46B)	111.5	O(8')-C(47')-H(47C)	110.4
H(46A)-C(46)-H(46B)	109.4	C(46')-C(47')-H(47C)	110.4
O(8)-C(47)-C(46)	104.2(7)	O(8')-C(47')-H(47D)	110.4
O(8)-C(47)-H(47A)	110.9	C(46')-C(47')-H(47D)	110.4
C(46)-C(47)-H(47A)	110.9	H(47C)-C(47')-H(47D)	108.6
O(8)-C(47)-H(47B)	110.9		

Symmetry transformations used to generate equivalent atoms:

#1 -x+2,-y+1,-z+1

3.3 $[Mg(NMP)_6][FePh_2(\mu\text{-}Ph)]_2 \cdot 3.5\ THF$ (1b)

REFERENCE NUMBER: neisc24

CRYSTAL STRUCTURE REPORT

$C_{80}\ H_{112}\ Fe_2\ Mg\ N_6\ O_{9.5}$

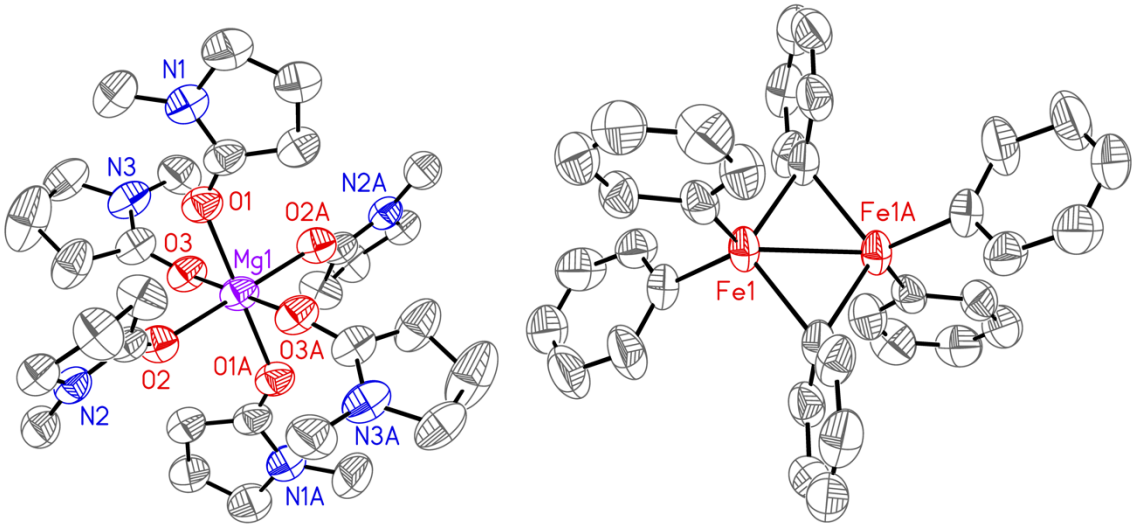
or

$[Mg(NMP)_6][FePh_2(\mu\text{-}Ph)]_2 \cdot 3.5THF$

Report prepared for:

S. Carpenter, Prof. M. Neidig

February 21, 2017



William W. Brennessel

X-ray Crystallographic Facility

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120 Trustee Road

Rochester, NY 14627

Data collection

A crystal (0.48 x 0.24 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 100.0(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 60 seconds and a detector distance of 3.99 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 4028 strong reflections from the actual data collection after integration.[3] See Table 1 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 $P4_2/n$ was determined based on systematic absences. 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 $R1 = 0.0534$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1561$ (F^2, all data).

Structure description

The structure is the one suggested. The asymmetric unit contains one-half of a $Mg(NMP)_6$ dication located at a crystallographic inversion center which is coincident with the Mg atom, one-half of a dianionic dinuclear iron complex located at a crystallographic inversion center, one cocrystallized THF solvent molecule in a general position, one-half of a THF molecule located along a crystallographic two-fold axis, and one-quarter of a THF molecule located at a crystallographic -4 position. One unique NMP ligand is modeled as disordered over two positions (0.83:0.17) and all three THF molecules are modeled as disordered: molecule O4/C38-C41 is modeled over two general positions (0.90:0.10); molecule O5/C42-C45 is modeled as disordered over the two-fold axis (0.50:0.50) and additionally over two general positions (refined to 0.50:0.50); and molecule O6/C46-C49 is modeled as disordered over the -4 position (0.25:0.25:0.25:0.25).

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] 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:

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

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

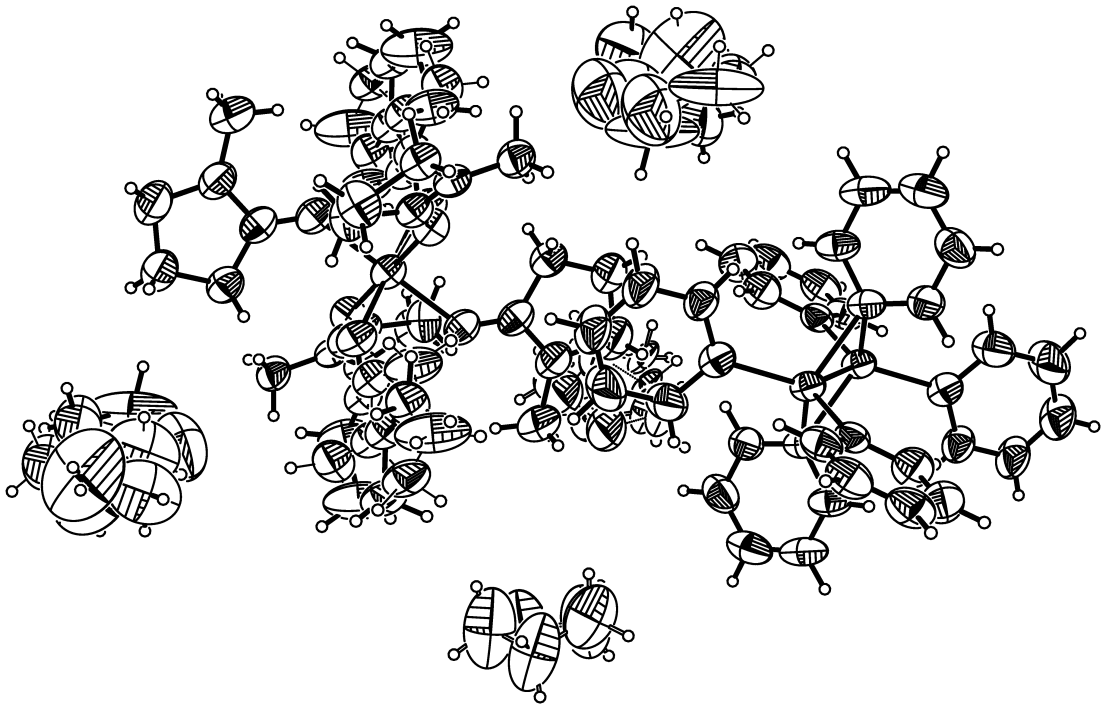
$$wR2 = [\Sigma[w(F_o^2 - F_c^2)^2] \,/\, \Sigma\, [w(F_o^2)^2]]^{1/2}$$

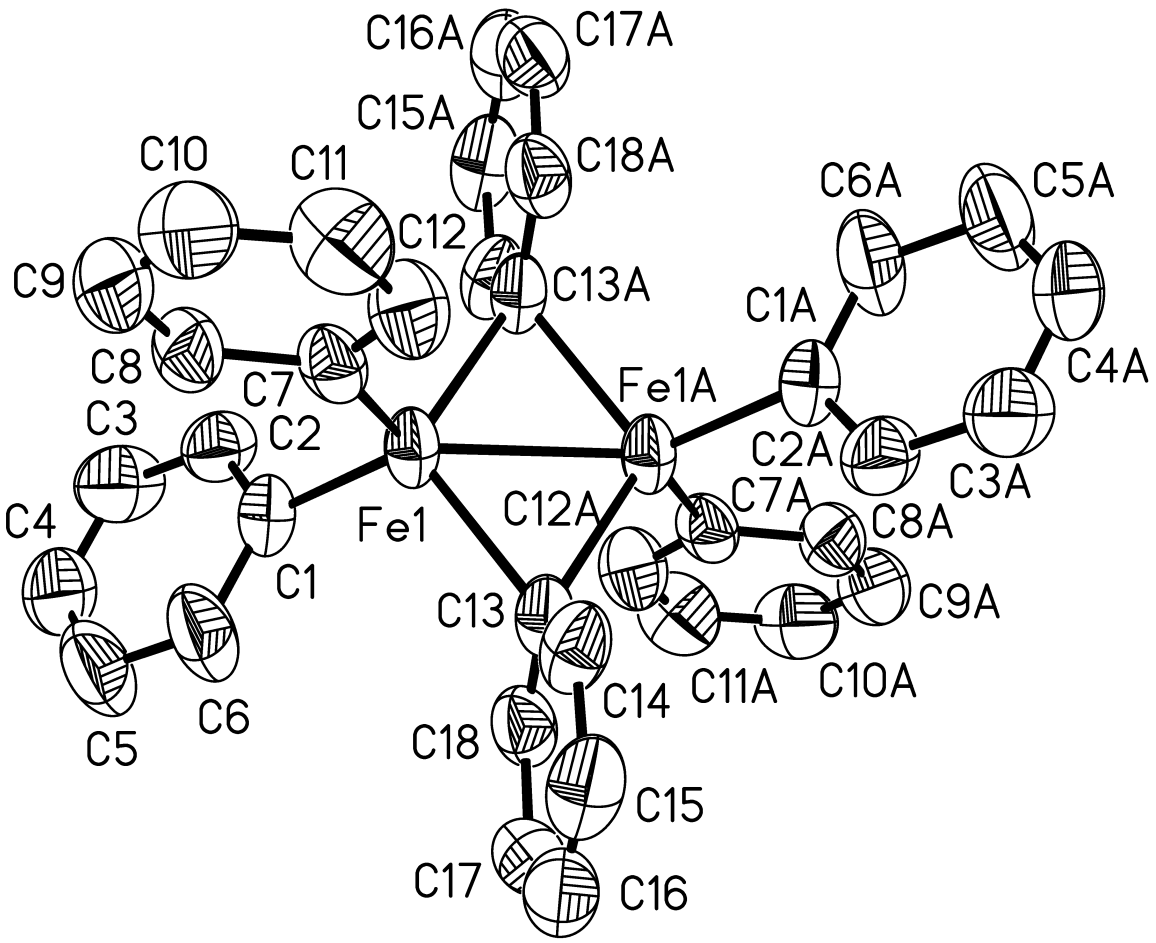
where $w = 1 \,/\, [\sigma^2 (F_o^2) + (aP)^2 + bP]$ and

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

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

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

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C15A
C18A
C10
C11
C12
C13A
C6A
C5A
C9
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C7
Fe1A
C3
C2
C2A
C3A
C4
Fe1
C12A
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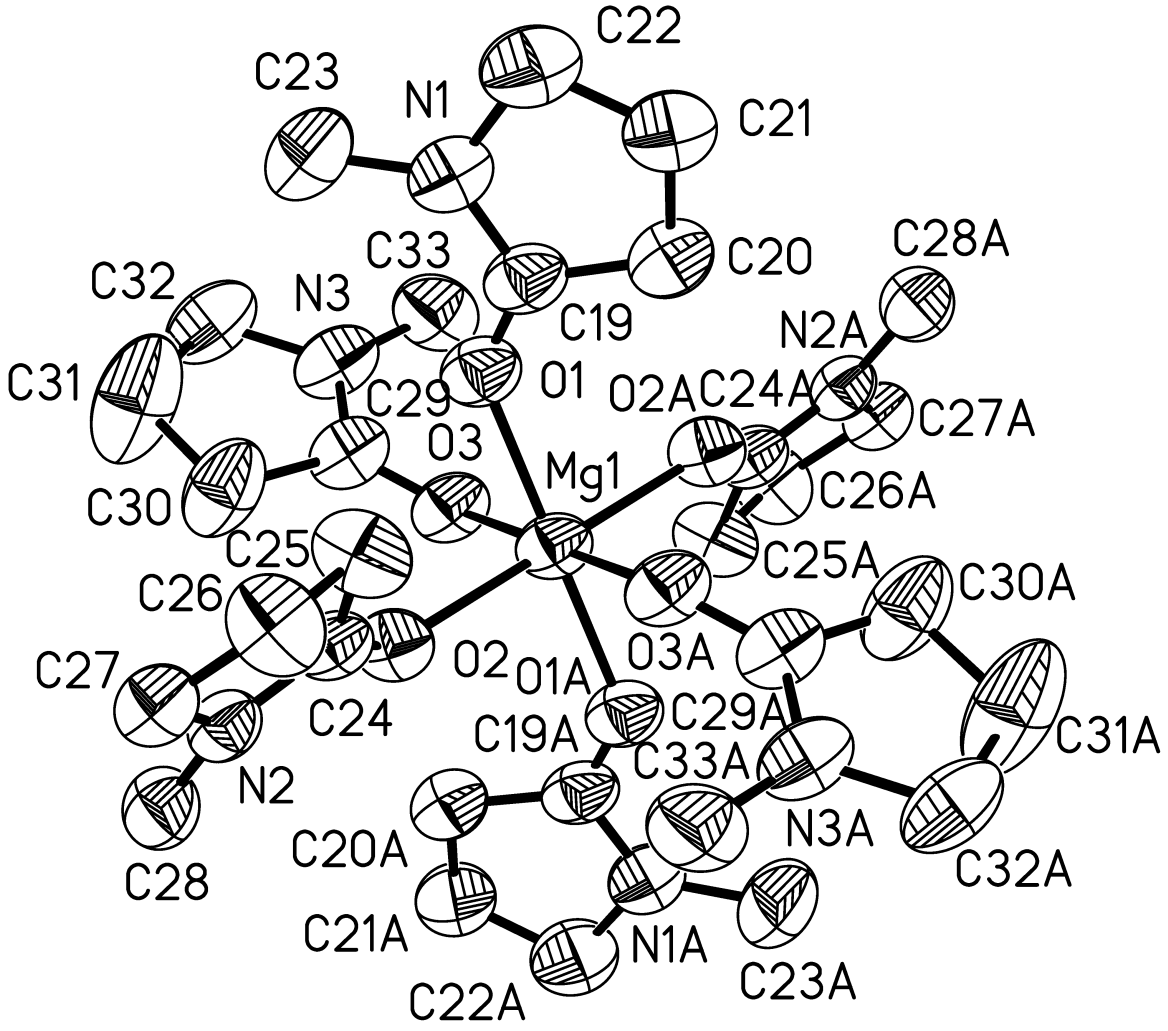
C14
C6
C18
C5
C15
C17
C16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C23
N1
C21
C33
C20
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N3
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C31
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C24A
C27A
O3
Mg1
C30
C26A
C25
C25A
C30A
C26
O2
O1A
O3A
C27
C24
C29A
C31A
C19A
C33A
N2
N3A
C20A
C32A
C28
C21A
N1A
C23A
C22A

Table S9. Crystal data and structure refinement for neisc24.

Identification code	neisc24	
Empirical formula	C80 H112 Fe2 Mg N6 O9.5	
Formula weight	1445.76	
Temperature	100.0(5) K	
Wavelength	0.71073 Å	
Crystal system	tetragonal	
Space group	$P4_2/n$	
Unit cell dimensions	a = 20.048(2) Å	α = 90°
	b = 20.048(2) Å	β = 90°
	c = 19.320(2) Å	γ = 90°
Volume	7764.9(18) Å^3	
Z	4	
Density (calculated)	1.237 Mg/m^3	
Absorption coefficient	0.441 mm^{-1}	
F(000)	3096	
Crystal color, morphology	dark red, block	
Crystal size	0.48 x 0.24 x 0.18 mm^3	
Theta range for data collection	1.436 to 25.272°	
Index ranges	$-24 \leq h \leq 24$, $-24 \leq k \leq 24$, $-23 \leq l \leq 23$	
Reflections collected	78802	
Independent reflections	7037 [R(int) = 0.0647]	
Observed reflections	4657	
Completeness to theta = 25.028°	100.0%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.7452 and 0.6677	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7037 / 118 / 554	
Goodness-of-fit on F^2	1.033	
Final R indices [I>2sigma(I)]	$R1$ = 0.0534, $wR2$ = 0.1299	
R indices (all data)	$R1$ = 0.0908, $wR2$ = 0.1561	
Largest diff. peak and hole	0.309 and -0.220 e.Å^{-3}	

Table S10. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for neisc24. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Fe1	5296(1)	266(1)	5497(1)	50(1)
C1	5516(2)	1273(2)	5608(2)	56(1)
C2	6072(2)	1576(2)	5323(2)	65(1)
C3	6261(2)	2227(2)	5473(3)	88(1)
C4	5900(2)	2613(2)	5914(3)	87(1)
C5	5340(3)	2349(2)	6204(3)	103(2)
C6	5147(2)	1693(2)	6045(2)	91(2)
C7	5635(2)	-275(2)	6341(2)	54(1)
C8	6021(2)	46(2)	6840(2)	63(1)
C9	6289(2)	-274(3)	7412(2)	77(1)
C10	6169(2)	-936(3)	7514(2)	83(1)
C11	5791(2)	-1285(2)	7047(2)	85(1)
C12	5530(2)	-955(2)	6467(2)	73(1)
C13	4205(2)	154(2)	5422(2)	53(1)
C14	3895(2)	-288(2)	5891(2)	63(1)
C15	3283(2)	-145(3)	6201(2)	78(1)
C16	2960(2)	440(3)	6080(2)	83(1)
C17	3245(2)	892(2)	5633(2)	79(1)
C18	3844(2)	744(2)	5310(2)	65(1)
Mg1	5000	5000	5000	53(1)
O1	4563(1)	4094(1)	5180(1)	60(1)
C19	4585(2)	3549(2)	4885(2)	60(1)
C20	5056(2)	3338(2)	4324(2)	64(1)
C21	4811(2)	2665(2)	4107(3)	89(1)
C22	4325(2)	2448(2)	4628(3)	94(1)
N1	4179(2)	3045(2)	5027(2)	79(1)
C23	3697(3)	3048(3)	5591(3)	112(2)
O2	4051(1)	5429(1)	4953(1)	56(1)
C24	3520(2)	5284(2)	4658(2)	53(1)
C25	3380(2)	4678(2)	4228(2)	72(1)
C26	2730(2)	4852(2)	3854(2)	76(1)

C27	2398(2)	5358(2)	4326(2)	59(1)
N2	2969(1)	5648(1)	4691(1)	52(1)
C28	2890(2)	6222(2)	5135(2)	67(1)
O3	5017(6)	5094(3)	6059(2)	60(1)
C29	4619(2)	4884(2)	6513(2)	59(1)
C30	3893(3)	5042(4)	6567(3)	92(2)
C31	3670(3)	4706(5)	7215(3)	131(3)
C32	4263(3)	4383(3)	7533(3)	82(2)
N3	4802(2)	4516(2)	7041(2)	66(1)
C33	5486(3)	4290(3)	7147(3)	82(2)
O3'	4940(30)	5193(16)	6044(8)	60(1)
C29'	5005(9)	4799(10)	6548(10)	59(1)
C30'	5580(10)	4343(16)	6710(14)	92(2)
C31'	5341(13)	3970(20)	7342(16)	131(3)
C32'	4654(12)	4172(14)	7495(12)	82(2)
N3'	4499(9)	4676(11)	6971(11)	66(1)
C33'	3895(13)	5079(16)	6933(16)	82(2)
O4	3369(5)	6842(4)	6846(6)	211(3)
C34	3380(6)	7030(7)	7575(6)	196(5)
C35	3570(9)	7680(9)	7671(9)	302(11)
C36	3612(6)	7943(4)	7023(9)	172(5)
C37	3408(4)	7483(10)	6512(5)	204(8)
O4'	3090(50)	7430(40)	6490(40)	211(3)
C34'	3470(60)	7190(40)	7090(50)	196(5)
C35'	3330(60)	7590(50)	7660(40)	302(11)
C36'	3220(40)	8210(30)	7370(40)	172(5)
C37'	2820(30)	8060(40)	6790(30)	204(8)
O5	2510(40)	2570(20)	4413(14)	127(8)
C38	2490(40)	3110(20)	3930(20)	127(8)
C39	2296(18)	2820(30)	3259(18)	111(10)
C40	2540(20)	2140(30)	3306(15)	96(8)
C41	2440(40)	1953(17)	4040(20)	124(12)
O5'	2720(20)	2343(13)	4662(14)	127(8)
C38'	2670(30)	2936(12)	4244(18)	127(8)
C39'	2670(20)	2687(12)	3521(17)	111(10)
C40'	2370(20)	2020(11)	3586(14)	96(8)

C41'	2690(30)	1765(12)	4223(15)	124(12)
O6	1887(18)	2705(18)	7180(16)	198(10)
C42	2120(20)	2045(14)	7030(20)	174(11)
C43	2780(20)	1990(20)	7410(20)	154(10)
C44	2700(20)	2490(20)	7993(14)	177(12)
C45	2370(30)	3057(18)	7590(30)	197(13)

Table S11. Bond lengths [Å] and angles [°] for neisc24.

Fe(1)-C(7)	2.071(3)	C(16)-H(16)	0.9500
Fe(1)-C(1)	2.078(3)	C(17)-C(18)	1.385(5)
Fe(1)-C(13)	2.204(3)	C(17)-H(17)	0.9500
Fe(1)-C(13)#1	2.205(3)	C(18)-H(18)	0.9500
Fe(1)-Fe(1)#1	2.4969(8)	Mg(1)-O(1)#2	2.046(2)
C(1)-C(2)	1.384(5)	Mg(1)-O(1)	2.047(2)
C(1)-C(6)	1.405(5)	Mg(1)-O(3)#2	2.054(4)
C(2)-C(3)	1.390(5)	Mg(1)-O(3)	2.054(4)
C(2)-H(2)	0.9500	Mg(1)-O(3')	2.057(17)
C(3)-C(4)	1.361(6)	Mg(1)-O(3')#2	2.057(17)
C(3)-H(3)	0.9500	Mg(1)-O(2)#2	2.090(2)
C(4)-C(5)	1.363(6)	Mg(1)-O(2)	2.090(2)
C(4)-H(4)	0.9500	O(1)-C(19)	1.233(4)
C(5)-C(6)	1.405(6)	C(19)-N(1)	1.326(5)
C(5)-H(5)	0.9500	C(19)-C(20)	1.497(5)
C(6)-H(6)	0.9500	C(20)-C(21)	1.496(5)
C(7)-C(8)	1.395(5)	C(20)-H(20A)	0.9900
C(7)-C(12)	1.402(5)	C(20)-H(20B)	0.9900
C(8)-C(9)	1.386(5)	C(21)-C(22)	1.468(6)
C(8)-H(8)	0.9500	C(21)-H(21A)	0.9900
C(9)-C(10)	1.364(6)	C(21)-H(21B)	0.9900
C(9)-H(9)	0.9500	C(22)-N(1)	1.455(5)
C(10)-C(11)	1.371(6)	C(22)-H(22A)	0.9900
C(10)-H(10)	0.9500	C(22)-H(22B)	0.9900
C(11)-C(12)	1.401(5)	N(1)-C(23)	1.456(6)
C(11)-H(11)	0.9500	C(23)-H(23A)	0.9800
C(12)-H(12)	0.9500	C(23)-H(23B)	0.9800
C(13)-C(18)	1.404(5)	C(23)-H(23C)	0.9800
C(13)-C(14)	1.413(5)	O(2)-C(24)	1.243(4)
C(14)-C(15)	1.395(5)	C(24)-N(2)	1.326(4)
C(14)-H(14)	0.9500	C(24)-C(25)	1.500(5)
C(15)-C(16)	1.359(6)	C(25)-C(26)	1.530(5)
C(15)-H(15)	0.9500	C(25)-H(25A)	0.9900
C(16)-C(17)	1.376(6)	C(25)-H(25B)	0.9900

C(26)-C(27)	1.517(5)	C(32')-H(32C)	0.9900
C(26)-H(26A)	0.9900	C(32')-H(32D)	0.9900
C(26)-H(26B)	0.9900	N(3')-C(33')	1.457(17)
C(27)-N(2)	1.464(4)	C(33')-H(33D)	0.9800
C(27)-H(27A)	0.9900	C(33')-H(33E)	0.9800
C(27)-H(27B)	0.9900	C(33')-H(33F)	0.9800
N(2)-C(28)	1.443(4)	O(4)-C(37)	1.440(12)
C(28)-H(28A)	0.9800	O(4)-C(34)	1.458(12)
C(28)-H(28B)	0.9800	C(34)-C(35)	1.371(13)
C(28)-H(28C)	0.9800	C(34)-H(34A)	0.9900
O(3)-C(29)	1.259(9)	C(34)-H(34B)	0.9900
C(29)-N(3)	1.310(5)	C(35)-C(36)	1.361(17)
C(29)-C(30)	1.493(7)	C(35)-H(35A)	0.9900
C(30)-C(31)	1.490(8)	C(35)-H(35B)	0.9900
C(30)-H(30A)	0.9900	C(36)-C(37)	1.412(12)
C(30)-H(30B)	0.9900	C(36)-H(36A)	0.9900
C(31)-C(32)	1.487(8)	C(36)-H(36B)	0.9900
C(31)-H(31A)	0.9900	C(37)-H(37A)	0.9900
C(31)-H(31B)	0.9900	C(37)-H(37B)	0.9900
C(32)-N(3)	1.464(6)	O(4')-C(34')	1.460(19)
C(32)-H(32A)	0.9900	O(4')-C(37')	1.48(2)
C(32)-H(32B)	0.9900	C(34')-C(35')	1.40(2)
N(3)-C(33)	1.459(6)	C(34')-H(34C)	0.9900
C(33)-H(33A)	0.9800	C(34')-H(34D)	0.9900
C(33)-H(33B)	0.9800	C(35')-C(36')	1.38(3)
C(33)-H(33C)	0.9800	C(35')-H(35C)	0.9900
O(3')-C(29')	1.262(18)	C(35')-H(35D)	0.9900
C(29')-N(3')	1.326(16)	C(36')-C(37')	1.41(2)
C(29')-C(30')	1.504(17)	C(36')-H(36C)	0.9900
C(30')-C(31')	1.511(19)	C(36')-H(36D)	0.9900
C(30')-H(30C)	0.9900	C(37')-H(37C)	0.9900
C(30')-H(30D)	0.9900	C(37')-H(37D)	0.9900
C(31')-C(32')	1.468(19)	O(5)-C(41)	1.437(17)
C(31')-H(31C)	0.9900	O(5)-C(38)	1.437(17)
C(31')-H(31D)	0.9900	C(38)-C(39)	1.473(19)
C(32')-N(3')	1.464(16)	C(38)-H(38A)	0.9900

C(38)-H(38B)	0.9900	C(7)-Fe(1)-C(13)	108.97(13)
C(39)-C(40)	1.47(2)	C(1)-Fe(1)-C(13)	108.43(14)
C(39)-H(39A)	0.9900	C(7)-Fe(1)-C(13)#1	106.54(13)
C(39)-H(39B)	0.9900	C(1)-Fe(1)-C(13)#1	110.89(13)
C(40)-C(41)	1.472(18)	C(13)-Fe(1)-C(13)#1	111.01(10)
C(40)-H(40A)	0.9900	C(7)-Fe(1)-Fe(1)#1	122.57(10)
C(40)-H(40B)	0.9900	C(1)-Fe(1)-Fe(1)#1	126.44(9)
C(41)-H(41A)	0.9900	C(13)-Fe(1)-Fe(1)#1	55.53(8)
C(41)-H(41B)	0.9900	C(13)#1-Fe(1)-Fe(1)#1	55.48(8)
O(5')-C(41')	1.437(17)	C(2)-C(1)-C(6)	113.6(3)
O(5')-C(38')	1.441(18)	C(2)-C(1)-Fe(1)	123.9(3)
C(38')-C(39')	1.484(18)	C(6)-C(1)-Fe(1)	122.2(3)
C(38')-H(38C)	0.9900	C(1)-C(2)-C(3)	123.4(4)
C(38')-H(38D)	0.9900	C(1)-C(2)-H(2)	118.3
C(39')-C(40')	1.47(2)	C(3)-C(2)-H(2)	118.3
C(39')-H(39C)	0.9900	C(4)-C(3)-C(2)	121.3(4)
C(39')-H(39D)	0.9900	C(4)-C(3)-H(3)	119.4
C(40')-C(41')	1.480(19)	C(2)-C(3)-H(3)	119.4
C(40')-H(40C)	0.9900	C(3)-C(4)-C(5)	118.4(4)
C(40')-H(40D)	0.9900	C(3)-C(4)-H(4)	120.8
C(41')-H(41C)	0.9900	C(5)-C(4)-H(4)	120.8
C(41')-H(41D)	0.9900	C(4)-C(5)-C(6)	120.1(4)
O(6)-C(42)	1.434(9)	C(4)-C(5)-H(5)	119.9
O(6)-C(45)	1.435(9)	C(6)-C(5)-H(5)	119.9
C(42)-C(43)	1.518(9)	C(1)-C(6)-C(5)	123.2(4)
C(42)-H(42A)	0.9900	C(1)-C(6)-H(6)	118.4
C(42)-H(42B)	0.9900	C(5)-C(6)-H(6)	118.4
C(43)-C(44)	1.523(10)	C(8)-C(7)-C(12)	114.3(3)
C(43)-H(43A)	0.9900	C(8)-C(7)-Fe(1)	119.0(3)
C(43)-H(43B)	0.9900	C(12)-C(7)-Fe(1)	126.7(3)
C(44)-C(45)	1.524(9)	C(9)-C(8)-C(7)	123.6(4)
C(44)-H(44A)	0.9900	C(9)-C(8)-H(8)	118.2
C(44)-H(44B)	0.9900	C(7)-C(8)-H(8)	118.2
C(45)-H(45A)	0.9900	C(10)-C(9)-C(8)	119.8(4)
C(45)-H(45B)	0.9900	C(10)-C(9)-H(9)	120.1
C(7)-Fe(1)-C(1)	110.99(13)	C(8)-C(9)-H(9)	120.1

C(9)-C(10)-C(11)	119.9(4)	O(1)#2-Mg(1)-O(3')	91.5(12)
C(9)-C(10)-H(10)	120.0	O(1)-Mg(1)-O(3')	88.5(12)
C(11)-C(10)-H(10)	120.0	O(1)#2-Mg(1)-O(3')#2	88.5(12)
C(10)-C(11)-C(12)	119.4(4)	O(1)-Mg(1)-O(3')#2	91.5(12)
C(10)-C(11)-H(11)	120.3	O(3)#2-Mg(1)-O(3')#2	7.2(13)
C(12)-C(11)-H(11)	120.3	O(3)-Mg(1)-O(3')#2	172.8(13)
C(11)-C(12)-C(7)	122.9(4)	O(3')-Mg(1)-O(3')#2	180.0
C(11)-C(12)-H(12)	118.5	O(1)#2-Mg(1)-O(2)#2	89.04(9)
C(7)-C(12)-H(12)	118.5	O(1)-Mg(1)-O(2)#2	90.97(9)
C(18)-C(13)-C(14)	113.7(3)	O(3)#2-Mg(1)-O(2)#2	91.2(3)
C(18)-C(13)-Fe(1)	115.9(2)	O(3)-Mg(1)-O(2)#2	88.8(3)
C(14)-C(13)-Fe(1)	117.2(3)	O(1)#2-Mg(1)-O(2)	90.96(9)
C(18)-C(13)-Fe(1)#1	115.6(2)	O(1)-Mg(1)-O(2)	89.03(9)
C(14)-C(13)-Fe(1)#1	118.5(3)	O(3)#2-Mg(1)-O(2)	88.8(3)
Fe(1)-C(13)-Fe(1)#1	68.99(10)	O(3)-Mg(1)-O(2)	91.2(3)
C(15)-C(14)-C(13)	122.2(4)	O(3')-Mg(1)-O(2)	84.7(15)
C(15)-C(14)-H(14)	118.9	O(3')#2-Mg(1)-O(2)	95.3(15)
C(13)-C(14)-H(14)	118.9	O(2)#2-Mg(1)-O(2)	180.0
C(16)-C(15)-C(14)	121.6(4)	C(19)-O(1)-Mg(1)	133.8(2)
C(16)-C(15)-H(15)	119.2	O(1)-C(19)-N(1)	123.8(4)
C(14)-C(15)-H(15)	119.2	O(1)-C(19)-C(20)	127.4(3)
C(15)-C(16)-C(17)	118.5(4)	N(1)-C(19)-C(20)	108.8(3)
C(15)-C(16)-H(16)	120.7	C(21)-C(20)-C(19)	104.6(3)
C(17)-C(16)-H(16)	120.7	C(21)-C(20)-H(20A)	110.8
C(16)-C(17)-C(18)	120.2(4)	C(19)-C(20)-H(20A)	110.8
C(16)-C(17)-H(17)	119.9	C(21)-C(20)-H(20B)	110.8
C(18)-C(17)-H(17)	119.9	C(19)-C(20)-H(20B)	110.8
C(17)-C(18)-C(13)	123.9(4)	H(20A)-C(20)-H(20B)	108.9
C(17)-C(18)-H(18)	118.1	C(22)-C(21)-C(20)	107.0(4)
C(13)-C(18)-H(18)	118.1	C(22)-C(21)-H(21A)	110.3
O(1)#2-Mg(1)-O(1)	180.0	C(20)-C(21)-H(21A)	110.3
O(1)#2-Mg(1)-O(3)#2	85.4(3)	C(22)-C(21)-H(21B)	110.3
O(1)-Mg(1)-O(3)#2	94.6(3)	C(20)-C(21)-H(21B)	110.3
O(1)#2-Mg(1)-O(3)	94.6(3)	H(21A)-C(21)-H(21B)	108.6
O(1)-Mg(1)-O(3)	85.4(3)	N(1)-C(22)-C(21)	104.6(3)
O(3)#2-Mg(1)-O(3)	180.0	N(1)-C(22)-H(22A)	110.8

C(21)-C(22)-H(22A)	110.8	C(24)-N(2)-C(27)	114.2(3)
N(1)-C(22)-H(22B)	110.8	C(28)-N(2)-C(27)	121.2(3)
C(21)-C(22)-H(22B)	110.8	N(2)-C(28)-H(28A)	109.5
H(22A)-C(22)-H(22B)	108.9	N(2)-C(28)-H(28B)	109.5
C(19)-N(1)-C(22)	113.2(4)	H(28A)-C(28)-H(28B)	109.5
C(19)-N(1)-C(23)	124.1(4)	N(2)-C(28)-H(28C)	109.5
C(22)-N(1)-C(23)	122.2(3)	H(28A)-C(28)-H(28C)	109.5
N(1)-C(23)-H(23A)	109.5	H(28B)-C(28)-H(28C)	109.5
N(1)-C(23)-H(23B)	109.5	C(29)-O(3)-Mg(1)	130.8(7)
H(23A)-C(23)-H(23B)	109.5	O(3)-C(29)-N(3)	123.6(6)
N(1)-C(23)-H(23C)	109.5	O(3)-C(29)-C(30)	126.5(5)
H(23A)-C(23)-H(23C)	109.5	N(3)-C(29)-C(30)	109.7(4)
H(23B)-C(23)-H(23C)	109.5	C(31)-C(30)-C(29)	104.8(5)
C(24)-O(2)-Mg(1)	134.7(2)	C(31)-C(30)-H(30A)	110.8
O(2)-C(24)-N(2)	124.3(3)	C(29)-C(30)-H(30A)	110.8
O(2)-C(24)-C(25)	127.2(3)	C(31)-C(30)-H(30B)	110.8
N(2)-C(24)-C(25)	108.5(3)	C(29)-C(30)-H(30B)	110.8
C(24)-C(25)-C(26)	103.6(3)	H(30A)-C(30)-H(30B)	108.9
C(24)-C(25)-H(25A)	111.0	C(32)-C(31)-C(30)	107.7(5)
C(26)-C(25)-H(25A)	111.0	C(32)-C(31)-H(31A)	110.2
C(24)-C(25)-H(25B)	111.0	C(30)-C(31)-H(31A)	110.2
C(26)-C(25)-H(25B)	111.0	C(32)-C(31)-H(31B)	110.2
H(25A)-C(25)-H(25B)	109.0	C(30)-C(31)-H(31B)	110.2
C(27)-C(26)-C(25)	104.1(3)	H(31A)-C(31)-H(31B)	108.5
C(27)-C(26)-H(26A)	110.9	N(3)-C(32)-C(31)	104.0(4)
C(25)-C(26)-H(26A)	110.9	N(3)-C(32)-H(32A)	111.0
C(27)-C(26)-H(26B)	110.9	C(31)-C(32)-H(32A)	111.0
C(25)-C(26)-H(26B)	110.9	N(3)-C(32)-H(32B)	111.0
H(26A)-C(26)-H(26B)	109.0	C(31)-C(32)-H(32B)	111.0
N(2)-C(27)-C(26)	102.3(3)	H(32A)-C(32)-H(32B)	109.0
N(2)-C(27)-H(27A)	111.3	C(29)-N(3)-C(33)	123.1(4)
C(26)-C(27)-H(27A)	111.3	C(29)-N(3)-C(32)	113.7(5)
N(2)-C(27)-H(27B)	111.3	C(33)-N(3)-C(32)	123.1(4)
C(26)-C(27)-H(27B)	111.3	N(3)-C(33)-H(33A)	109.5
H(27A)-C(27)-H(27B)	109.2	N(3)-C(33)-H(33B)	109.5
C(24)-N(2)-C(28)	123.9(3)	H(33A)-C(33)-H(33B)	109.5

N(3)-C(33)-H(33C)	109.5	C(35)-C(34)-H(34A)	109.1
H(33A)-C(33)-H(33C)	109.5	O(4)-C(34)-H(34A)	109.1
H(33B)-C(33)-H(33C)	109.5	C(35)-C(34)-H(34B)	109.1
C(29')-O(3')-Mg(1)	129(2)	O(4)-C(34)-H(34B)	109.1
O(3')-C(29')-N(3')	121(3)	H(34A)-C(34)-H(34B)	107.9
O(3')-C(29')-C(30')	129(3)	C(36)-C(35)-C(34)	105.1(11)
N(3')-C(29')-C(30')	110.2(14)	C(36)-C(35)-H(35A)	110.7
C(29')-C(30')-C(31')	103.2(15)	C(34)-C(35)-H(35A)	110.7
C(29')-C(30')-H(30C)	111.1	C(36)-C(35)-H(35B)	110.7
C(31')-C(30')-H(30C)	111.1	C(34)-C(35)-H(35B)	110.7
C(29')-C(30')-H(30D)	111.1	H(35A)-C(35)-H(35B)	108.8
C(31')-C(30')-H(30D)	111.1	C(35)-C(36)-C(37)	111.9(9)
H(30C)-C(30')-H(30D)	109.1	C(35)-C(36)-H(36A)	109.2
C(32')-C(31')-C(30')	108.7(16)	C(37)-C(36)-H(36A)	109.2
C(32')-C(31')-H(31C)	109.9	C(35)-C(36)-H(36B)	109.2
C(30')-C(31')-H(31C)	109.9	C(37)-C(36)-H(36B)	109.2
C(32')-C(31')-H(31D)	109.9	H(36A)-C(36)-H(36B)	107.9
C(30')-C(31')-H(31D)	109.9	C(36)-C(37)-O(4)	106.6(8)
H(31C)-C(31')-H(31D)	108.3	C(36)-C(37)-H(37A)	110.4
N(3')-C(32')-C(31')	104.6(15)	O(4)-C(37)-H(37A)	110.4
N(3')-C(32')-H(32C)	110.8	C(36)-C(37)-H(37B)	110.4
C(31')-C(32')-H(32C)	110.8	O(4)-C(37)-H(37B)	110.4
N(3')-C(32')-H(32D)	110.8	H(37A)-C(37)-H(37B)	108.6
C(31')-C(32')-H(32D)	110.8	C(34')-O(4')-C(37')	99(2)
H(32C)-C(32')-H(32D)	108.9	C(35')-C(34')-O(4')	109(2)
C(29')-N(3')-C(33')	120.1(18)	C(35')-C(34')-H(34C)	109.8
C(29')-N(3')-C(32')	113.1(14)	O(4')-C(34')-H(34C)	109.8
C(33')-N(3')-C(32')	126.5(18)	C(35')-C(34')-H(34D)	109.8
N(3')-C(33')-H(33D)	109.5	O(4')-C(34')-H(34D)	109.8
N(3')-C(33')-H(33E)	109.5	H(34C)-C(34')-H(34D)	108.2
H(33D)-C(33')-H(33E)	109.5	C(36')-C(35')-C(34')	103(3)
N(3')-C(33')-H(33F)	109.5	C(36')-C(35')-H(35C)	111.1
H(33D)-C(33')-H(33F)	109.5	C(34')-C(35')-H(35C)	111.1
H(33E)-C(33')-H(33F)	109.5	C(36')-C(35')-H(35D)	111.1
C(37)-O(4)-C(34)	101.6(9)	C(34')-C(35')-H(35D)	111.1
C(35)-C(34)-O(4)	112.4(12)	H(35C)-C(35')-H(35D)	109.1

C(35')-C(36')-C(37')	103(2)	H(41A)-C(41)-H(41B)	108.8
C(35')-C(36')-H(36C)	111.2	C(41')-O(5')-C(38')	109.3(16)
C(37')-C(36')-H(36C)	111.2	O(5')-C(38')-C(39')	104.5(15)
C(35')-C(36')-H(36D)	111.2	O(5')-C(38')-H(38C)	110.9
C(37')-C(36')-H(36D)	111.2	C(39')-C(38')-H(38C)	110.9
H(36C)-C(36')-H(36D)	109.1	O(5')-C(38')-H(38D)	110.9
C(36')-C(37')-O(4')	107(2)	C(39')-C(38')-H(38D)	110.9
C(36')-C(37')-H(37C)	110.3	H(38C)-C(38')-H(38D)	108.9
O(4')-C(37')-H(37C)	110.3	C(40')-C(39')-C(38')	103.0(16)
C(36')-C(37')-H(37D)	110.3	C(40')-C(39')-H(39C)	111.2
O(4')-C(37')-H(37D)	110.3	C(38')-C(39')-H(39C)	111.2
H(37C)-C(37')-H(37D)	108.5	C(40')-C(39')-H(39D)	111.2
C(41)-O(5)-C(38)	108.8(16)	C(38')-C(39')-H(39D)	111.2
O(5)-C(38)-C(39)	106.0(16)	H(39C)-C(39')-H(39D)	109.1
O(5)-C(38)-H(38A)	110.5	C(39')-C(40')-C(41')	101.9(16)
C(39)-C(38)-H(38A)	110.5	C(39')-C(40')-H(40C)	111.4
O(5)-C(38)-H(38B)	110.5	C(41')-C(40')-H(40C)	111.4
C(39)-C(38)-H(38B)	110.5	C(39')-C(40')-H(40D)	111.4
H(38A)-C(38)-H(38B)	108.7	C(41')-C(40')-H(40D)	111.4
C(40)-C(39)-C(38)	103.4(16)	H(40C)-C(40')-H(40D)	109.3
C(40)-C(39)-H(39A)	111.1	O(5')-C(41')-C(40')	103.3(17)
C(38)-C(39)-H(39A)	111.1	O(5')-C(41')-H(41C)	111.1
C(40)-C(39)-H(39B)	111.1	C(40')-C(41')-H(41C)	111.1
C(38)-C(39)-H(39B)	111.1	O(5')-C(41')-H(41D)	111.1
H(39A)-C(39)-H(39B)	109.1	C(40')-C(41')-H(41D)	111.1
C(39)-C(40)-C(41)	104.2(18)	H(41C)-C(41')-H(41D)	109.1
C(39)-C(40)-H(40A)	110.9	C(42)-O(6)-C(45)	110(2)
C(41)-C(40)-H(40A)	110.9	O(6)-C(42)-C(43)	104.8(18)
C(39)-C(40)-H(40B)	110.9	O(6)-C(42)-H(42A)	110.8
C(41)-C(40)-H(40B)	110.9	C(43)-C(42)-H(42A)	110.8
H(40A)-C(40)-H(40B)	108.9	O(6)-C(42)-H(42B)	110.8
O(5)-C(41)-C(40)	105.1(17)	C(43)-C(42)-H(42B)	110.8
O(5)-C(41)-H(41A)	110.7	H(42A)-C(42)-H(42B)	108.9
C(40)-C(41)-H(41A)	110.7	C(42)-C(43)-C(44)	102.4(16)
O(5)-C(41)-H(41B)	110.7	C(42)-C(43)-H(43A)	111.3
C(40)-C(41)-H(41B)	110.7	C(44)-C(43)-H(43A)	111.3

C(42)-C(43)-H(43B)	111.3	H(44A)-C(44)-H(44B)	109.6
C(44)-C(43)-H(43B)	111.3	O(6)-C(45)-C(44)	102.3(18)
H(43A)-C(43)-H(43B)	109.2	O(6)-C(45)-H(45A)	111.3
C(43)-C(44)-C(45)	99(2)	C(44)-C(45)-H(45A)	111.3
C(43)-C(44)-H(44A)	111.9	O(6)-C(45)-H(45B)	111.3
C(45)-C(44)-H(44A)	111.9	C(44)-C(45)-H(45B)	111.3
C(43)-C(44)-H(44B)	111.9	H(45A)-C(45)-H(45B)	109.2
C(45)-C(44)-H(44B)	111.9		

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y,-z+1 #2 -x+1,-y+1,-z+1

3.4 $Fe_4(\mu\text{-}Ph)_6(THF)_4 \cdot 2\ THF$ (2a)

REFERENCE NUMBER: neisc36

CRYSTAL STRUCTURE REPORT

$C_{60}\ H_{78}\ Fe_4\ O_6$

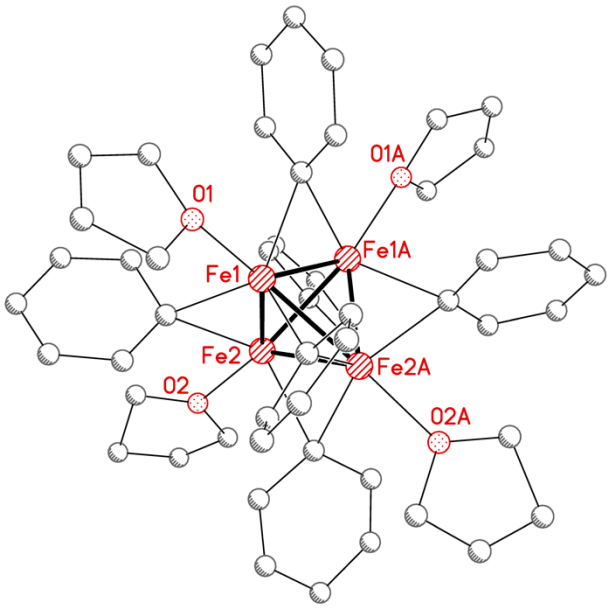
or

$Fe(\mu_2\text{-}Ph)_6(THF)_4 \cdot 2THF$

Report prepared for:

S. Carpenter, Prof. M. Neidig

November 09, 2017



William W. Brennessel

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Rochester, NY 14627

Data collection

A crystal (0.14 x 0.10 x 0.04 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 60 seconds and a detector distance of 4.04 cm. A randomly oriented region of reciprocal space was surveyed: three major sections of frames were collected with 0.50º steps in ω at three 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 1373 strong reflections from the actual data collection after integration.[3] See Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SHELXT-2014/5[4] and refined using SHELXL-2017/1.[5] The space group *Fddd* was determined based on systematic absences. 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 atoms were refined with isotropic 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 $R1 = 0.2483$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.4911$ (F^2, all data).

While this appears to be a favorable crystallization, the crystals were simply too small for the available instrumentation to provide adequate intensity data. This structure serves solely to support the material's formulation in the solid state.

Structure description

The structure is the one suggested. The asymmetric unit contains one-half of the iron cluster, located along a crystallographic two-fold axis that includes atoms C13, C16, C17, and C20, and one cocrystallized THF solvent molecule in a general position.

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 2017.3-0; Bruker AXS: Madison, WI, 2017.

[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 2017/1; *Acta. Cryst.* **2015**, C*71*, 3-8.

Some equations of interest:

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

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

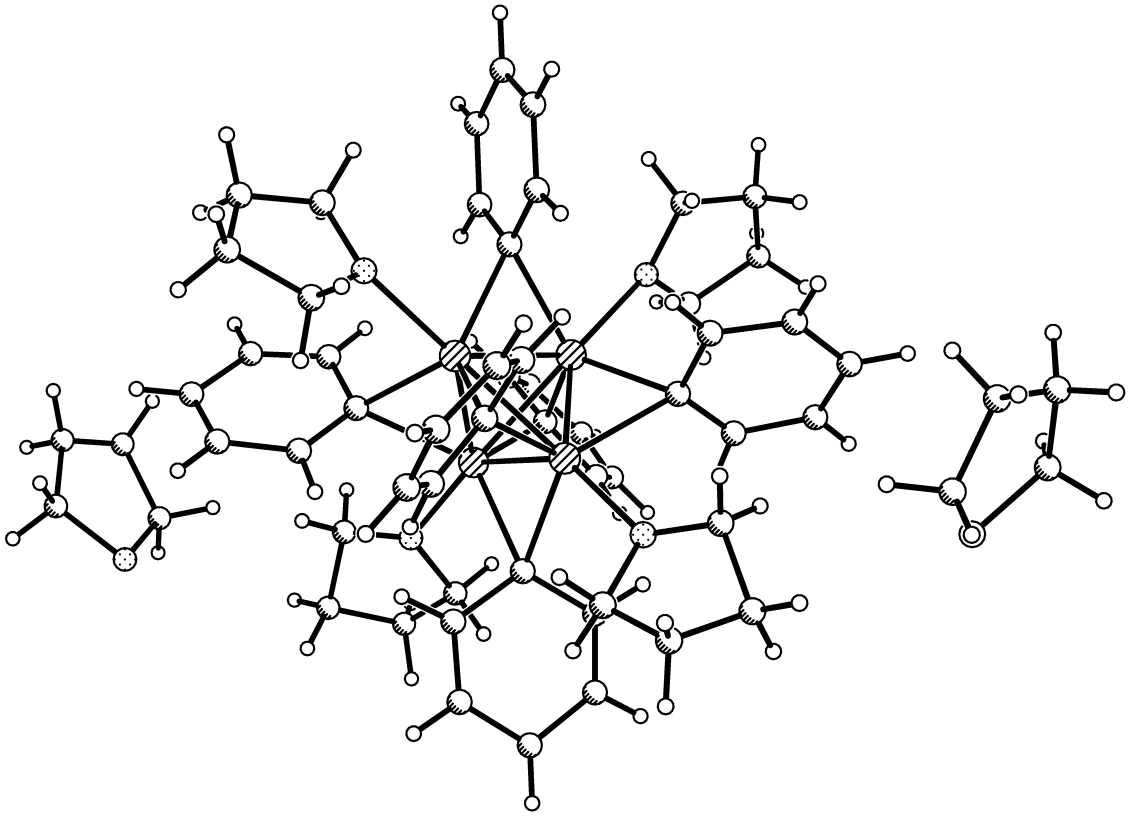
$$wR2 = [\Sigma[w(F_o^2-F_c^2)^2] \ / \ \Sigma\ [w(F_o^2)^2]]^{1/2}$$

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



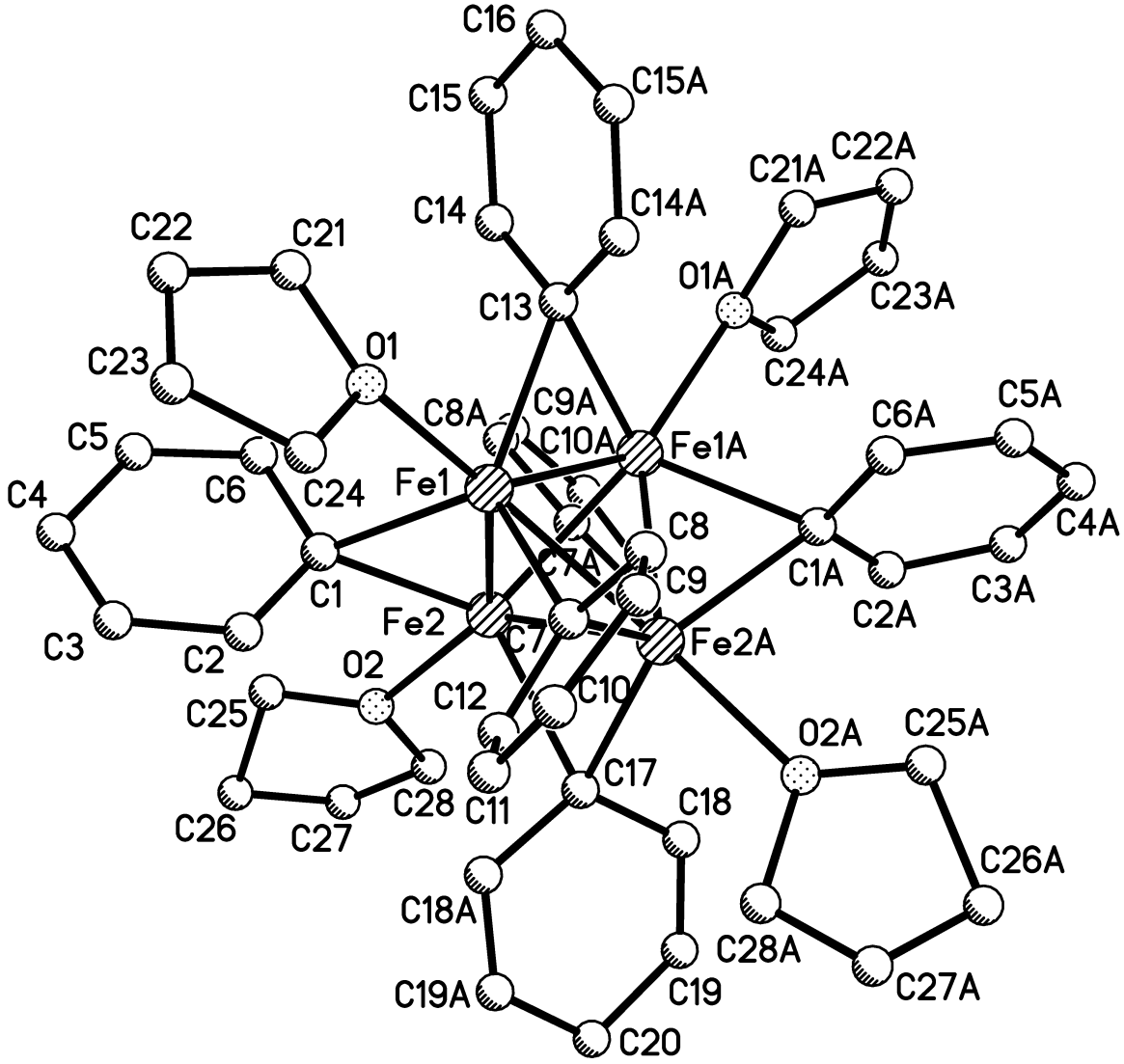

C16
C15
C15A
C14
C14A
C22
C21
C21A
C22A
C13
O1A
C23A
C23
O1
C24A
C8A
C9A
C10A
Fe1A
C6A
C5A
C5
C6
C24
Fe1
C4
C8
C4A
C7A
C9
C1A
C1
C3A
C3
C2
Fe2
C7
C2A
Fe2A
O2
C12
C10
C25
O2A
C25A
C11
C17
C18
C28
C26
C27
C26A
C18A
C28A
C19
C19A
C27A
C20

Table S12. Crystal data and structure refinement for neisc36.

Identification code	neisc36	
Empirical formula	C60 H78 Fe4 O6	
Formula weight	1118.62	
Temperature	100.0(5) K	
Wavelength	0.71073 Å	
Crystal system	orthorhombic	
Space group	*Fddd*	
Unit cell dimensions	a = 20.134(8) Å	α = 90°
	b = 24.672(9) Å	β = 90°
	c = 43.274(18) Å	γ = 90°
Volume	21497(15) Å^3	
Z	16	
Density (calculated)	1.383 Mg/m^3	
Absorption coefficient	1.109 mm^{-1}	
F(000)	9440	
Crystal color, morphology	brown, plate	
Crystal size	0.14 x 0.10 x 0.04 mm^3	
Theta range for data collection	1.388 to 25.446°	
Index ranges	$-24 \le h \le 22$, $-29 \le k \le 29$, $-51 \le l \le 52$	
Reflections collected	39382	
Independent reflections	4959 [R(int) = 0.5026]	
Observed reflections	2067	
Completeness to theta = 25.242°	100.0%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.7452 and 0.4171	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4959 / 4 / 141	
Goodness-of-fit on F^2	1.949	
Final R indices [I>2sigma(I)]	R1 = 0.2483, wR2 = 0.4569	
R indices (all data)	R1 = 0.3772, wR2 = 0.4911	
Largest diff. peak and hole	3.365 and -2.841 e.Å^{-3}	

Table S13. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for neisc36. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Fe1	5858(2)	5884(1)	4102(1)	28(1)
Fe2	6725(2)	5916(1)	4503(1)	41(1)
C1	6346(11)	5163(8)	4277(4)	26(5)
C2	5997(12)	4805(9)	4432(5)	41(6)
C3	6019(14)	4254(10)	4384(6)	56(8)
C4	6460(14)	4026(11)	4194(5)	53(7)
C5	6905(12)	4382(8)	4037(5)	34(6)
C6	6864(11)	4931(8)	4064(5)	31(6)
C7	4957(13)	6125(9)	4349(5)	42(6)
C8	4451(13)	6402(9)	4154(5)	39(6)
C9	3740(15)	6295(10)	4182(6)	59(8)
C10	3489(17)	5934(11)	4392(6)	75(9)
C11	3966(17)	5703(12)	4585(7)	80(10)
C12	4654(13)	5794(9)	4577(5)	46(7)
C13	6250	6250	3678(7)	30(8)
C14	6763(11)	6025(8)	3499(4)	29(5)
C15	6785(11)	6026(7)	3181(4)	27(5)
C16	6250	6250	3010(8)	41(9)
C17	6250	6250	4924(8)	50(10)
C18	6457(15)	6713(11)	5101(6)	64(8)
C19	6497(15)	6690(11)	5403(6)	62(8)
C20	6250	6250	5586(10)	70(12)
O1	5264(7)	5362(5)	3787(3)	33(4)
C21	5478(13)	5085(9)	3520(5)	42(6)
C22	5141(16)	4587(11)	3492(7)	76(9)
C23	4586(18)	4573(13)	3712(8)	93(11)
C24	4680(14)	5082(10)	3894(6)	62(8)
O2	7340(12)	5408(9)	4792(5)	99(8)
C25	7579(15)	4912(11)	4759(6)	66(8)
C26	7910(20)	4739(16)	5070(8)	122(14)
C27	8200(20)	5255(16)	5153(9)	133(16)

C28	7870(30)	5660(20)	5030(13)	210(30)
O3	7493(17)	3344(12)	4835(7)	165(12)
C29	7502(17)	2859(13)	4608(8)	100(12)
C30	8020(30)	2830(20)	4337(9)	190(20)
C31	8330(20)	3215(17)	4363(9)	154(18)
C32	8154(18)	3496(15)	4677(8)	122(14)

Table S14. Bond lengths [Å] and angles [°] for neisc36.

Fe(1)-C(1)	2.17(2)	C(13)-C(14)#1	1.41(2)
Fe(1)-C(7)	2.19(3)	C(13)-C(14)	1.41(2)
Fe(1)-C(13)	2.19(2)	C(14)-C(15)	1.38(2)
Fe(1)-O(1)	2.225(14)	C(14)-H(14)	0.9500
Fe(1)-Fe(1)#1	2.400(6)	C(15)-C(16)	1.42(2)
Fe(1)-Fe(2)#1	2.453(4)	C(15)-H(15)	0.9500
Fe(1)-Fe(2)	2.462(5)	C(16)-H(16)	0.9500
Fe(2)-C(7)#1	2.11(3)	C(17)-C(18)#1	1.44(3)
Fe(2)-O(2)	2.16(2)	C(17)-C(18)	1.44(3)
Fe(2)-C(17)	2.22(3)	C(18)-C(19)	1.31(3)
Fe(2)-C(1)	2.23(2)	C(18)-H(18)	0.9500
Fe(2)-Fe(2)#1	2.527(7)	C(19)-C(20)	1.43(3)
C(1)-C(2)	1.32(3)	C(19)-H(19)	0.9500
C(1)-C(6)	1.50(3)	C(20)-H(20)	0.9500
C(2)-C(3)	1.37(3)	O(1)-C(21)	1.41(2)
C(2)-H(2)	0.9500	O(1)-C(24)	1.44(3)
C(3)-C(4)	1.34(3)	C(21)-C(22)	1.41(3)
C(3)-H(3)	0.9500	C(21)-H(21A)	0.9900
C(4)-C(5)	1.43(3)	C(21)-H(21B)	0.9900
C(4)-H(4)	0.9500	C(22)-C(23)	1.47(4)
C(5)-C(6)	1.36(3)	C(22)-H(22A)	0.9900
C(5)-H(5)	0.9500	C(22)-H(22B)	0.9900
C(6)-H(6)	0.9500	C(23)-C(24)	1.50(3)
C(7)-C(12)	1.42(3)	C(23)-H(23A)	0.9900
C(7)-C(8)	1.49(3)	C(23)-H(23B)	0.9900
C(8)-C(9)	1.46(4)	C(24)-H(24A)	0.9900
C(8)-H(8)	0.9500	C(24)-H(24B)	0.9900
C(9)-C(10)	1.37(3)	O(2)-C(25)	1.32(3)
C(9)-H(9)	0.9500	O(2)-C(28)	1.61(6)
C(10)-C(11)	1.39(4)	C(25)-C(26)	1.56(4)
C(10)-H(10)	0.9500	C(25)-H(25A)	0.9900
C(11)-C(12)	1.40(4)	C(25)-H(25B)	0.9900
C(11)-H(11)	0.9500	C(26)-C(27)	1.45(5)
C(12)-H(12)	0.9500	C(26)-H(26A)	0.9900

C(26)-H(26B)	0.9900	C(13)-Fe(1)-Fe(2)	108.7(4)
C(27)-C(28)	1.33(6)	O(1)-Fe(1)-Fe(2)	146.3(4)
C(27)-H(27A)	0.9900	Fe(1)#1-Fe(1)-Fe(2)	60.57(13)
C(27)-H(27B)	0.9900	Fe(2)#1-Fe(1)-Fe(2)	61.88(18)
C(28)-H(28A)	0.9900	C(7)#1-Fe(2)-O(2)	92.7(9)
C(28)-H(28B)	0.9900	C(7)#1-Fe(2)-C(17)	113.4(6)
O(3)-C(32)	1.54(3)	O(2)-Fe(2)-C(17)	89.2(8)
O(3)-C(29)	1.55(3)	C(7)#1-Fe(2)-C(1)	125.1(8)
C(29)-C(30)	1.57(3)	O(2)-Fe(2)-C(1)	88.2(8)
C(29)-H(29A)	0.9900	C(17)-Fe(2)-C(1)	121.5(6)
C(29)-H(29B)	0.9900	C(7)#1-Fe(2)-Fe(1)#1	56.7(7)
C(30)-C(31)	1.15(6)	O(2)-Fe(2)-Fe(1)#1	149.0(7)
C(30)-H(30A)	0.9900	C(17)-Fe(2)-Fe(1)#1	106.8(5)
C(30)-H(30B)	0.9900	C(1)-Fe(2)-Fe(1)#1	104.6(5)
C(31)-C(32)	1.56(3)	C(7)#1-Fe(2)-Fe(1)	110.5(7)
C(31)-H(31A)	0.9900	O(2)-Fe(2)-Fe(1)	142.7(7)
C(31)-H(31B)	0.9900	C(17)-Fe(2)-Fe(1)	106.5(5)
C(32)-H(32A)	0.9900	C(1)-Fe(2)-Fe(1)	54.7(5)
C(32)-H(32B)	0.9900	Fe(1)#1-Fe(2)-Fe(1)	58.47(16)
C(1)-Fe(1)-C(7)	115.5(8)	C(7)#1-Fe(2)-Fe(2)#1	104.0(7)
C(1)-Fe(1)-C(13)	117.7(6)	O(2)-Fe(2)-Fe(2)#1	144.3(6)
C(7)-Fe(1)-C(13)	126.5(6)	C(17)-Fe(2)-Fe(2)#1	55.3(5)
C(1)-Fe(1)-O(1)	89.0(7)	C(1)-Fe(2)-Fe(2)#1	106.5(6)
C(7)-Fe(1)-O(1)	90.7(7)	Fe(1)#1-Fe(2)-Fe(2)#1	59.24(12)
C(13)-Fe(1)-O(1)	85.3(6)	Fe(1)-Fe(2)-Fe(2)#1	58.88(13)
C(1)-Fe(1)-Fe(1)#1	108.6(6)	C(2)-C(1)-C(6)	115.3(19)
C(7)-Fe(1)-Fe(1)#1	109.9(6)	C(2)-C(1)-Fe(1)	119.2(18)
C(13)-Fe(1)-Fe(1)#1	56.8(4)	C(6)-C(1)-Fe(1)	114.5(13)
O(1)-Fe(1)-Fe(1)#1	142.1(4)	C(2)-C(1)-Fe(2)	121.1(16)
C(1)-Fe(1)-Fe(2)#1	111.3(5)	C(6)-C(1)-Fe(2)	110.3(15)
C(7)-Fe(1)-Fe(2)#1	53.6(6)	Fe(1)-C(1)-Fe(2)	68.0(6)
C(13)-Fe(1)-Fe(2)#1	109.0(4)	C(1)-C(2)-C(3)	125(2)
O(1)-Fe(1)-Fe(2)#1	143.6(4)	C(1)-C(2)-H(2)	117.6
Fe(1)#1-Fe(1)-Fe(2)#1	60.96(12)	C(3)-C(2)-H(2)	117.7
C(1)-Fe(1)-Fe(2)	57.3(6)	C(4)-C(3)-C(2)	122(3)
C(7)-Fe(1)-Fe(2)	103.7(6)	C(4)-C(3)-H(3)	118.9

C(2)-C(3)-H(3)	118.9	Fe(1)-C(13)-Fe(1)#1	66.3(8)
C(3)-C(4)-C(5)	117(2)	C(15)-C(14)-C(13)	125(2)
C(3)-C(4)-H(4)	121.6	C(15)-C(14)-H(14)	117.4
C(5)-C(4)-H(4)	121.6	C(13)-C(14)-H(14)	117.4
C(6)-C(5)-C(4)	122(2)	C(14)-C(15)-C(16)	120(2)
C(6)-C(5)-H(5)	118.8	C(14)-C(15)-H(15)	120.2
C(4)-C(5)-H(5)	118.8	C(16)-C(15)-H(15)	120.2
C(5)-C(6)-C(1)	118(2)	C(15)#1-C(16)-C(15)	117(3)
C(5)-C(6)-H(6)	120.9	C(15)#1-C(16)-H(16)	121.3
C(1)-C(6)-H(6)	120.9	C(15)-C(16)-H(16)	121.3
C(12)-C(7)-C(8)	111(2)	C(18)#1-C(17)-C(18)	116(3)
C(12)-C(7)-Fe(2)#1	115.3(17)	C(18)#1-C(17)-Fe(2)#1	127.5(15)
C(8)-C(7)-Fe(2)#1	117.9(16)	C(18)-C(17)-Fe(2)#1	105.5(13)
C(12)-C(7)-Fe(1)	122.5(17)	C(18)#1-C(17)-Fe(2)	105.5(13)
C(8)-C(7)-Fe(1)	114.6(16)	C(18)-C(17)-Fe(2)	127.5(15)
Fe(2)#1-C(7)-Fe(1)	69.6(8)	Fe(2)#1-C(17)-Fe(2)	69.4(10)
C(9)-C(8)-C(7)	123(2)	C(19)-C(18)-C(17)	121(3)
C(9)-C(8)-H(8)	118.7	C(19)-C(18)-H(18)	119.5
C(7)-C(8)-H(8)	118.7	C(17)-C(18)-H(18)	119.5
C(10)-C(9)-C(8)	122(3)	C(18)-C(19)-C(20)	124(3)
C(10)-C(9)-H(9)	118.8	C(18)-C(19)-H(19)	117.9
C(8)-C(9)-H(9)	118.8	C(20)-C(19)-H(19)	117.9
C(11)-C(10)-C(9)	114(3)	C(19)-C(20)-C(19)#1	113(4)
C(11)-C(10)-H(10)	122.9	C(19)-C(20)-H(20)	123.5
C(9)-C(10)-H(10)	122.9	C(19)#1-C(20)-H(20)	123.5
C(10)-C(11)-C(12)	127(3)	C(21)-O(1)-C(24)	106.4(17)
C(10)-C(11)-H(11)	116.6	C(21)-O(1)-Fe(1)	128.2(14)
C(12)-C(11)-H(11)	116.6	C(24)-O(1)-Fe(1)	121.2(13)
C(11)-C(12)-C(7)	122(3)	O(1)-C(21)-C(22)	110(2)
C(11)-C(12)-H(12)	118.9	O(1)-C(21)-H(21A)	109.6
C(7)-C(12)-H(12)	118.9	C(22)-C(21)-H(21A)	109.6
C(14)#1-C(13)-C(14)	113(3)	O(1)-C(21)-H(21B)	109.6
C(14)#1-C(13)-Fe(1)	111.1(11)	C(22)-C(21)-H(21B)	109.6
C(14)-C(13)-Fe(1)	124.4(11)	H(21A)-C(21)-H(21B)	108.1
C(14)#1-C(13)-Fe(1)#1	124.4(11)	C(21)-C(22)-C(23)	109(3)
C(14)-C(13)-Fe(1)#1	111.1(11)	C(21)-C(22)-H(22A)	109.8

C(23)-C(22)-H(22A)	109.8	C(26)-C(27)-H(27B)	109.5
C(21)-C(22)-H(22B)	109.8	H(27A)-C(27)-H(27B)	108.0
C(23)-C(22)-H(22B)	109.8	C(27)-C(28)-O(2)	107(4)
H(22A)-C(22)-H(22B)	108.2	C(27)-C(28)-H(28A)	110.4
C(22)-C(23)-C(24)	103(3)	O(2)-C(28)-H(28A)	110.4
C(22)-C(23)-H(23A)	111.1	C(27)-C(28)-H(28B)	110.1
C(24)-C(23)-H(23A)	111.1	O(2)-C(28)-H(28B)	110.1
C(22)-C(23)-H(23B)	111.2	H(28A)-C(28)-H(28B)	108.5
C(24)-C(23)-H(23B)	111.2	C(32)-O(3)-C(29)	84(3)
H(23A)-C(23)-H(23B)	109.1	O(3)-C(29)-C(30)	121(3)
O(1)-C(24)-C(23)	110(2)	O(3)-C(29)-H(29A)	107.0
O(1)-C(24)-H(24A)	109.8	C(30)-C(29)-H(29A)	107.0
C(23)-C(24)-H(24A)	109.7	O(3)-C(29)-H(29B)	107.0
O(1)-C(24)-H(24B)	109.7	C(30)-C(29)-H(29B)	107.0
C(23)-C(24)-H(24B)	109.7	H(29A)-C(29)-H(29B)	106.7
H(24A)-C(24)-H(24B)	108.2	C(31)-C(30)-C(29)	104.1(19)
C(25)-O(2)-C(28)	101(3)	C(31)-C(30)-H(30A)	110.9
C(25)-O(2)-Fe(2)	133(2)	C(29)-C(30)-H(30A)	110.9
C(28)-O(2)-Fe(2)	121(3)	C(31)-C(30)-H(30B)	111.0
O(2)-C(25)-C(26)	108(3)	C(29)-C(30)-H(30B)	110.9
O(2)-C(25)-H(25A)	110.0	H(30A)-C(30)-H(30B)	108.9
C(26)-C(25)-H(25A)	110.1	C(30)-C(31)-C(32)	109(2)
O(2)-C(25)-H(25B)	110.0	C(30)-C(31)-H(31A)	109.7
C(26)-C(25)-H(25B)	110.0	C(32)-C(31)-H(31A)	109.8
H(25A)-C(25)-H(25B)	108.4	C(30)-C(31)-H(31B)	109.8
C(27)-C(26)-C(25)	99(3)	C(32)-C(31)-H(31B)	109.8
C(27)-C(26)-H(26A)	112.1	H(31A)-C(31)-H(31B)	108.2
C(25)-C(26)-H(26A)	112.1	O(3)-C(32)-C(31)	118(2)
C(27)-C(26)-H(26B)	112.0	O(3)-C(32)-H(32A)	107.8
C(25)-C(26)-H(26B)	112.0	C(31)-C(32)-H(32A)	107.8
H(26A)-C(26)-H(26B)	109.7	O(3)-C(32)-H(32B)	107.8
C(28)-C(27)-C(26)	111(4)	C(31)-C(32)-H(32B)	107.8
C(28)-C(27)-H(27A)	109.6	H(32A)-C(32)-H(32B)	107.1
C(26)-C(27)-H(27A)	109.4		
C(28)-C(27)-H(27B)	109.3		

Symmetry transformations used to generate equivalent atoms: #1 -x+5/4,-y+5/4,z

3.5 $Fe_4(\mu\text{-}p\text{-tolyl})_6(THF)_4 \cdot 2\ THF \cdot C_5H_{12}$ (2b)

REFERENCE NUMBER: neisc35

CRYSTAL STRUCTURE REPORT

$C_{71}\ H_{102}\ Fe_4\ O_6$

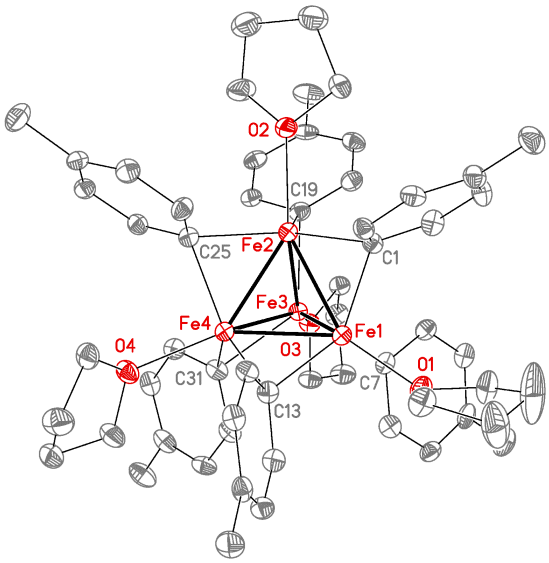
or

$Fe_4(\mu\text{-}p\text{-tolyl})_6(THF)_4 \cdot 2THF \cdot C_5H_{12}$

Report prepared for:

S. Carpenter, Prof. M. Neidig

September 04, 2017



William W. Brennessel

X-ray Crystallographic Facility

Department of Chemistry, University of Rochester

120 Trustee Road

Rochester, NY 14627

Data collection

A crystal (0.28 x 0.26 x 0.24 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 60 seconds and a detector distance of 4.01 cm. A randomly oriented region of reciprocal space was surveyed: eleven major sections of frames were collected with 0.50º steps in ω at eleven 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 3912 strong reflections from the actual data collection after integration.[3] See Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SHELXT-2014/5[4] and refined using SHELXL-2017/1.[5] The space group $P2_1/n$ was determined based on systematic absences. 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 refinement stalled at $R1 = 0.116$, at which point twin modeling was required. After the non-merohedral twin law, [0.66 0 -0.34 / 0 -1 0 / -1.66 0 -0.66], a 180 degree rotation about reciprocal lattice [-1 0 1], was determined,[6] the data were re-integrated,[3] and a new absorption correction was applied.[7] There were 12232 unique reflections solely in the first component, 12121 unique reflections solely in the second component, and 7304 unique overlapping reflections. The mass ratio of the two components refined to 0.61:0.39.

The final full matrix least squares refinement converged to $R1 = 0.0492$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1115$ (F^2, all data).

Structure description

The structure is the one suggested. The asymmetric unit contains one tetranuclear iron complex, one *n*-pentane and two THF cocrystallized solvent molecules, all 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, 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 2017.3-0; Bruker AXS: Madison, WI, 2017.

[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 2017/1; *Acta. Cryst.* **2015**, C*71*, 3-8.

[6] a) Parsons, S.; Gould, B.; Cooper, R.; Farrugia, L. *ROTAX*; University of Edinburgh: Edinburgh, Scotland, 2003; b) Sheldrick, G. M. *CELL_NOW: A program that analyzes a list of reflections to find a cell and orientation matrix despite the presence of several twin domains or other junk*, version 2008/2; University of Göttingen: Göttingen, Germany, 2008.

[7] Sheldrick, G. M. *TWINABS*, version 2012/1; University of Göttingen: Göttingen, Germany, 2012.

Some equations of interest:

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

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

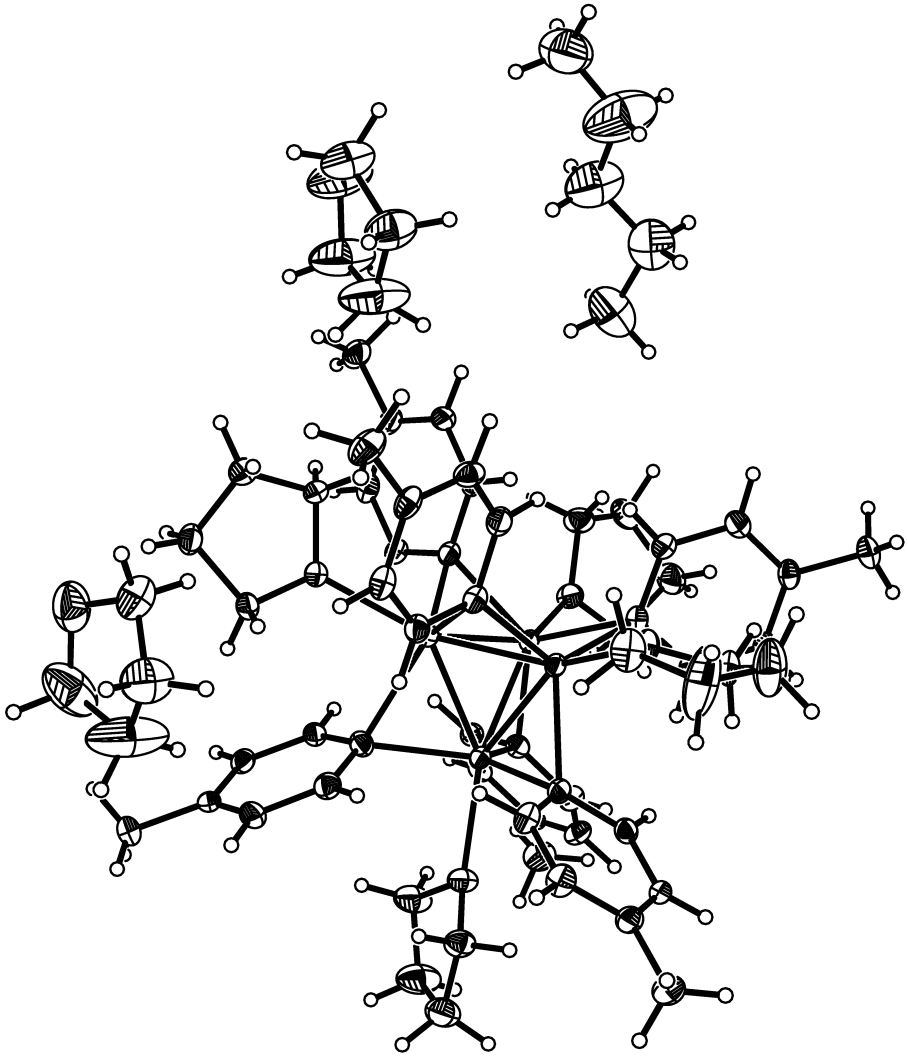
$$wR2 = [\Sigma [w(F_o^2 - F_c^2)^2] \,/\, \Sigma\, [w(F_o^2)^2]]^{1/2}$$

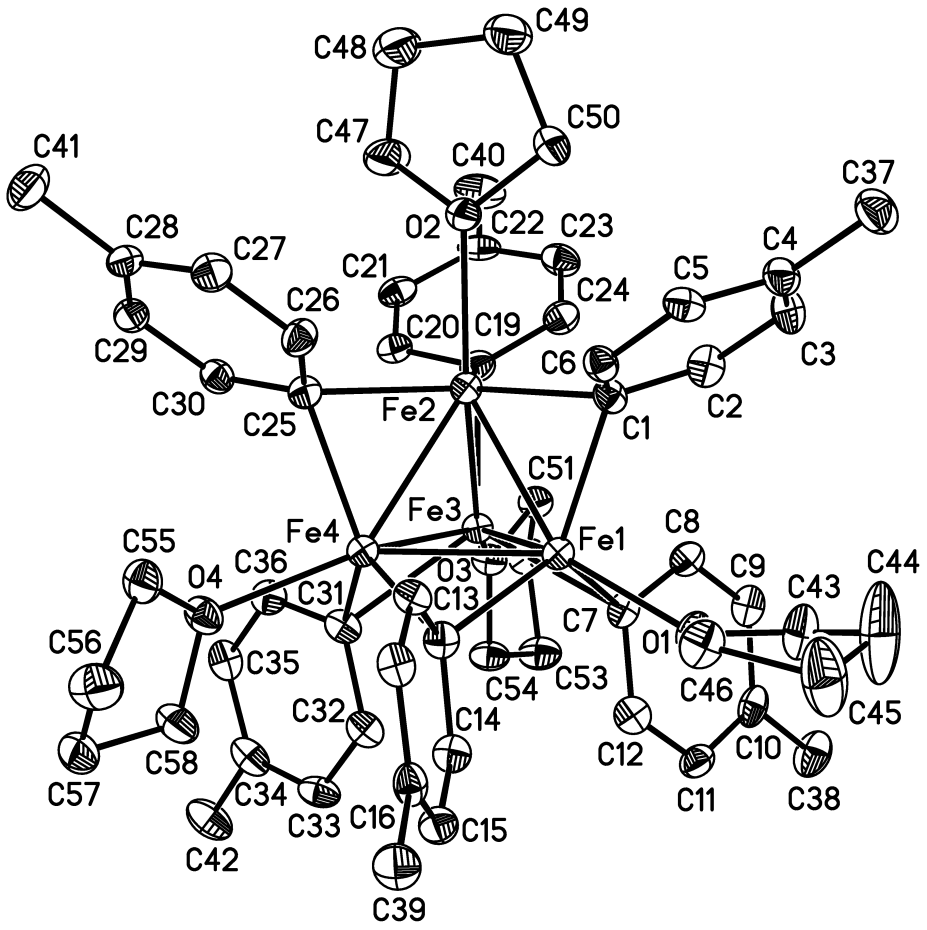
where $w = 1 \,/\, [\sigma^2 (F_o^2) + (aP)^2 + bP]$ and

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

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

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




C49
C48
C50
C47
C41
C40
C37
O2
C22
C23
C28
C27
C4
C21
C5
C24
C26
C20
C19
C29
C6
C3
C30
C25
Fe2
C1
C2
C51
Fe3
Fe4
Fe1
C8
C55
C36
O4
O3
C9
C44
C31
C13
C43
C7
C56
C35
O1
C54
C53
C46
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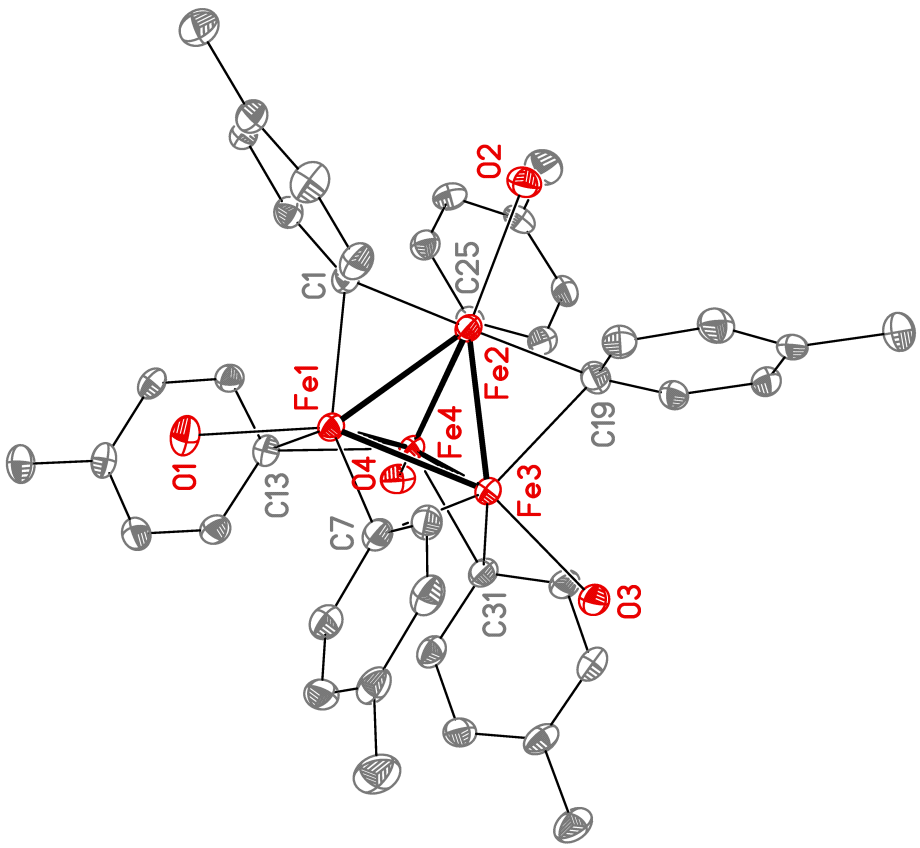
C57
C34
C33
C16
C15
C11
C38
C42
C39


O2
C1
C25
Fe1
Fe2
C19
O1
C13
O4
Fe4
Fe3
C7
C31
O3

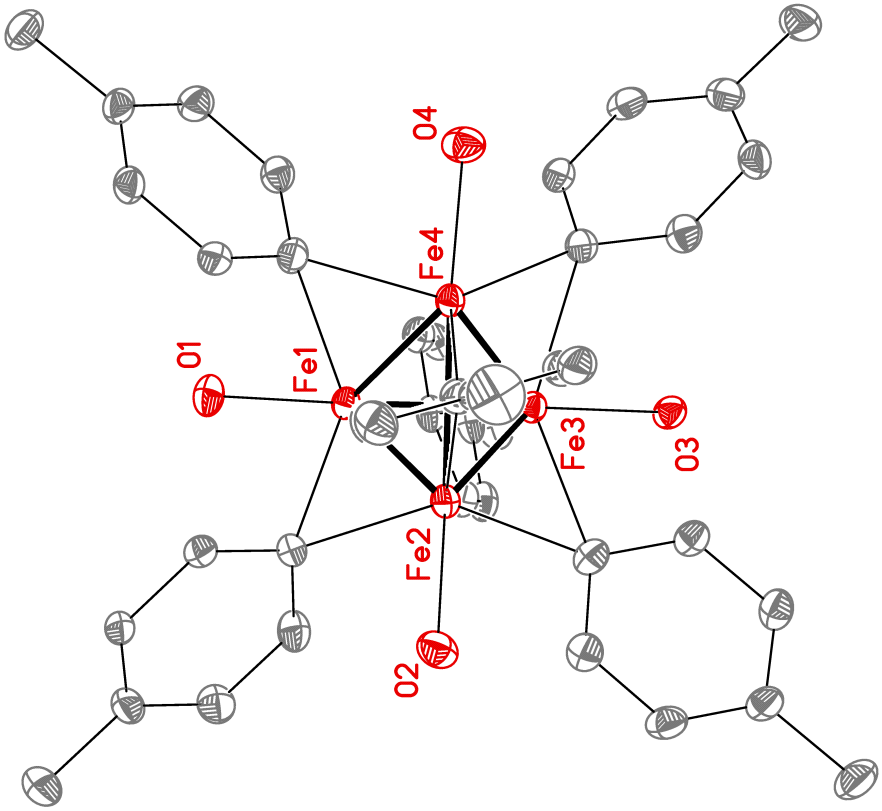

O4
Fe4
O1
Fe1
Fe3
O3
Fe2
O2

Table S15. Crystal data and structure refinement for neisc35.

Identification code	neisc35	
Empirical formula	C71 H102 Fe4 O6	
Formula weight	1274.92	
Temperature	100.0(5) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 12.2994(11) Å	α = 90°
	b = 22.698(2) Å	β = 99.1880(19)°
	c = 23.587(2) Å	γ = 90°
Volume	6500.3(10) Å^3	
Z	4	
Density (calculated)	1.303 Mg/m^3	
Absorption coefficient	0.925 mm^{-1}	
F(000)	2720	
Crystal color, morphology	brown, block	
Crystal size	0.28 x 0.26 x 0.24 mm^3	
Theta range for data collection	1.749 to 27.946°	
Index ranges	$-16 \le h \le 15$, $0 \le k \le 29$, $0 \le l \le 31$	
Reflections collected	403100	
Independent reflections	15554 [R(int) = 0.1592]	
Observed reflections	11514	
Completeness to theta = 27.878°	100.0%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.7456 and 0.6645	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	15554 / 0 / 739	
Goodness-of-fit on F^2	1.025	
Final R indices [I>2sigma(I)]	$R1$ = 0.0492, $wR2$ = 0.0987	
R indices (all data)	$R1$ = 0.0824, $wR2$ = 0.1115	
Largest diff. peak and hole	0.971 and -0.714 e.Å^{-3}	

Table S16. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for neisc35. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Fe1	2923(1)	3275(1)	6318(1)	16(1)
Fe2	934(1)	2984(1)	6050(1)	16(1)
Fe3	2326(1)	2259(1)	6379(1)	16(1)
Fe4	1840(1)	2942(1)	7083(1)	15(1)
C1	1779(2)	3685(1)	5622(1)	18(1)
C2	2046(3)	3568(1)	5070(2)	24(1)
C3	1978(3)	3991(1)	4639(2)	25(1)
C4	1619(3)	4560(1)	4724(1)	22(1)
C5	1345(2)	4690(1)	5259(1)	20(1)
C6	1427(2)	4266(1)	5691(1)	19(1)
C7	3909(2)	2561(1)	6095(1)	20(1)
C8	3853(3)	2290(1)	5554(1)	21(1)
C9	4725(3)	1980(1)	5383(2)	24(1)
C10	5732(3)	1935(1)	5745(2)	23(1)
C11	5834(3)	2216(1)	6272(2)	23(1)
C12	4948(3)	2509(1)	6443(2)	23(1)
C13	2994(2)	3683(1)	7187(1)	19(1)
C14	4034(3)	3659(1)	7535(1)	21(1)
C15	4475(3)	4119(1)	7892(1)	23(1)
C16	3890(3)	4640(1)	7926(1)	21(1)
C17	2846(3)	4677(1)	7596(1)	20(1)
C18	2420(3)	4216(1)	7239(1)	19(1)
C19	793(3)	2102(1)	5725(1)	21(1)
C20	122(2)	1679(1)	5948(1)	20(1)
C21	-443(2)	1240(1)	5616(2)	22(1)
C22	-401(3)	1191(1)	5033(2)	23(1)
C23	236(3)	1607(1)	4796(2)	25(1)
C24	813(3)	2042(1)	5134(1)	23(1)
C25	91(2)	3161(1)	6791(1)	19(1)
C26	-384(2)	3725(1)	6721(1)	22(1)
C27	-1388(3)	3868(1)	6880(2)	24(1)

C28	-1996(2)	3452(2)	7126(1)	22(1)
C29	-1559(2)	2886(1)	7205(1)	21(1)
C30	-553(2)	2746(1)	7040(1)	20(1)
C31	2480(3)	2079(1)	7322(1)	20(1)
C32	3580(3)	2046(1)	7609(1)	22(1)
C33	3962(3)	1609(1)	8002(1)	24(1)
C34	3266(3)	1167(1)	8136(1)	25(1)
C35	2172(3)	1186(1)	7874(1)	24(1)
C36	1796(3)	1630(1)	7484(1)	22(1)
C37	1542(3)	5019(2)	4259(2)	30(1)
C38	6666(3)	1575(2)	5575(2)	34(1)
C39	4371(3)	5151(1)	8296(2)	28(1)
C40	-1014(3)	710(2)	4674(2)	35(1)
C41	-3104(3)	3611(2)	7289(2)	34(1)
C42	3696(3)	668(2)	8531(2)	34(1)
O1	4287(2)	3884(1)	6160(1)	24(1)
C43	4880(3)	3793(2)	5684(2)	35(1)
C44	5186(5)	4373(2)	5504(3)	75(2)
C45	5146(4)	4779(2)	5983(2)	56(1)
C46	4292(3)	4504(1)	6298(2)	27(1)
O2	-653(2)	3226(1)	5479(1)	24(1)
C47	-1632(3)	2891(2)	5521(2)	38(1)
C48	-2535(3)	3184(2)	5144(2)	46(1)
C49	-2014(3)	3496(2)	4698(2)	37(1)
C50	-780(3)	3424(2)	4894(2)	27(1)
O3	2783(2)	1294(1)	6269(1)	21(1)
C51	2526(3)	953(1)	5748(1)	21(1)
C52	3085(3)	362(1)	5894(2)	26(1)
C53	4140(3)	548(2)	6273(2)	29(1)
C54	3769(3)	1057(1)	6613(2)	23(1)
O4	1536(2)	3047(1)	7994(1)	24(1)
C55	903(3)	3532(2)	8174(2)	27(1)
C56	1557(3)	3740(2)	8733(2)	37(1)
C57	2064(3)	3165(2)	8993(2)	31(1)
C58	2364(3)	2865(2)	8471(2)	30(1)
O5	3434(3)	-1(1)	4500(1)	57(1)

C59	2382(5)	190(3)	4219(2)	77(2)
C60	2521(4)	764(3)	3945(3)	103(3)
C61	3739(4)	820(2)	3956(2)	59(1)
C62	4178(3)	468(2)	4473(2)	44(1)
O6	7619(3)	343(2)	7934(2)	80(1)
C63	6616(4)	568(3)	7671(3)	86(2)
C64	6816(4)	989(3)	7215(3)	88(2)
C65	8045(4)	986(2)	7224(2)	58(1)
C66	8412(4)	459(2)	7592(2)	61(1)
C67	10076(5)	1582(3)	8869(3)	78(2)
C68	9454(6)	2129(3)	8810(4)	113(3)
C69	8275(4)	2107(3)	8721(3)	76(2)
C70	7647(5)	2673(2)	8727(2)	68(2)
C71	6411(4)	2567(3)	8563(2)	72(2)

Table S17. Bond lengths [Å] and angles [°] for neisc35.

Fe(1)-C(7)	2.139(3)	C(7)-C(8)	1.409(4)
Fe(1)-C(1)	2.193(3)	C(8)-C(9)	1.395(4)
Fe(1)-C(13)	2.238(3)	C(8)-H(8)	0.9500
Fe(1)-O(1)	2.250(2)	C(9)-C(10)	1.391(5)
Fe(1)-Fe(3)	2.4311(6)	C(9)-H(9)	0.9500
Fe(1)-Fe(2)	2.5156(6)	C(10)-C(11)	1.387(5)
Fe(1)-Fe(4)	2.5251(6)	C(10)-C(38)	1.516(4)
Fe(2)-C(19)	2.141(3)	C(11)-C(12)	1.389(4)
Fe(2)-C(25)	2.206(3)	C(11)-H(11)	0.9500
Fe(2)-C(1)	2.230(3)	C(12)-H(12)	0.9500
Fe(2)-O(2)	2.256(2)	C(13)-C(14)	1.406(4)
Fe(2)-Fe(3)	2.4118(6)	C(13)-C(18)	1.414(4)
Fe(2)-Fe(4)	2.5138(6)	C(14)-C(15)	1.395(4)
Fe(3)-C(31)	2.240(3)	C(14)-H(14)	0.9500
Fe(3)-C(7)	2.264(3)	C(15)-C(16)	1.394(4)
Fe(3)-C(19)	2.266(3)	C(15)-H(15)	0.9500
Fe(3)-O(3)	2.285(2)	C(16)-C(17)	1.394(5)
Fe(3)-Fe(4)	2.4152(6)	C(16)-C(39)	1.513(4)
Fe(4)-C(31)	2.151(3)	C(17)-C(18)	1.393(4)
Fe(4)-C(13)	2.191(3)	C(17)-H(17)	0.9500
Fe(4)-C(25)	2.207(3)	C(18)-H(18)	0.9500
Fe(4)-O(4)	2.252(2)	C(19)-C(24)	1.405(5)
C(1)-C(6)	1.405(4)	C(19)-C(20)	1.420(4)
C(1)-C(2)	1.417(4)	C(20)-C(21)	1.383(4)
C(2)-C(3)	1.392(5)	C(20)-H(20)	0.9500
C(2)-H(2)	0.9500	C(21)-C(22)	1.390(5)
C(3)-C(4)	1.389(4)	C(21)-H(21)	0.9500
C(3)-H(3)	0.9500	C(22)-C(23)	1.398(5)
C(4)-C(5)	1.388(4)	C(22)-C(40)	1.508(5)
C(4)-C(37)	1.506(4)	C(23)-C(24)	1.390(5)
C(5)-C(6)	1.392(4)	C(23)-H(23)	0.9500
C(5)-H(5)	0.9500	C(24)-H(24)	0.9500
C(6)-H(6)	0.9500	C(25)-C(26)	1.407(4)
C(7)-C(12)	1.409(5)	C(25)-C(30)	1.417(4)

C(26)-C(27)	1.385(4)	C(42)-H(42B)	0.9800
C(26)-H(26)	0.9500	C(42)-H(42C)	0.9800
C(27)-C(28)	1.388(4)	O(1)-C(46)	1.444(4)
C(27)-H(27)	0.9500	O(1)-C(43)	1.447(4)
C(28)-C(29)	1.393(4)	C(43)-C(44)	1.452(5)
C(28)-C(41)	1.517(4)	C(43)-H(43A)	0.9900
C(29)-C(30)	1.392(4)	C(43)-H(43B)	0.9900
C(29)-H(29)	0.9500	C(44)-C(45)	1.465(6)
C(30)-H(30)	0.9500	C(44)-H(44A)	0.9900
C(31)-C(36)	1.412(4)	C(44)-H(44B)	0.9900
C(31)-C(32)	1.414(4)	C(45)-C(46)	1.515(5)
C(32)-C(33)	1.389(4)	C(45)-H(45A)	0.9900
C(32)-H(32)	0.9500	C(45)-H(45B)	0.9900
C(33)-C(34)	1.388(5)	C(46)-H(46A)	0.9900
C(33)-H(33)	0.9500	C(46)-H(46B)	0.9900
C(34)-C(35)	1.389(5)	O(2)-C(50)	1.435(4)
C(34)-C(42)	1.507(5)	O(2)-C(47)	1.442(4)
C(35)-C(36)	1.393(5)	C(47)-C(48)	1.466(5)
C(35)-H(35)	0.9500	C(47)-H(47A)	0.9900
C(36)-H(36)	0.9500	C(47)-H(47B)	0.9900
C(37)-H(37A)	0.9800	C(48)-C(49)	1.495(5)
C(37)-H(37B)	0.9800	C(48)-H(48A)	0.9900
C(37)-H(37C)	0.9800	C(48)-H(48B)	0.9900
C(38)-H(38A)	0.9800	C(49)-C(50)	1.524(5)
C(38)-H(38B)	0.9800	C(49)-H(49A)	0.9900
C(38)-H(38C)	0.9800	C(49)-H(49B)	0.9900
C(39)-H(39A)	0.9800	C(50)-H(50A)	0.9900
C(39)-H(39B)	0.9800	C(50)-H(50B)	0.9900
C(39)-H(39C)	0.9800	O(3)-C(51)	1.444(4)
C(40)-H(40A)	0.9800	O(3)-C(54)	1.451(4)
C(40)-H(40B)	0.9800	C(51)-C(52)	1.522(4)
C(40)-H(40C)	0.9800	C(51)-H(51A)	0.9900
C(41)-H(41A)	0.9800	C(51)-H(51B)	0.9900
C(41)-H(41B)	0.9800	C(52)-C(53)	1.514(5)
C(41)-H(41C)	0.9800	C(52)-H(52A)	0.9900
C(42)-H(42A)	0.9800	C(52)-H(52B)	0.9900

C(53)-C(54)	1.518(4)	C(64)-C(65)	1.508(6)
C(53)-H(53A)	0.9900	C(64)-H(64A)	0.9900
C(53)-H(53B)	0.9900	C(64)-H(64B)	0.9900
C(54)-H(54A)	0.9900	C(65)-C(66)	1.506(6)
C(54)-H(54B)	0.9900	C(65)-H(65A)	0.9900
O(4)-C(55)	1.450(4)	C(65)-H(65B)	0.9900
O(4)-C(58)	1.451(4)	C(66)-H(66A)	0.9900
C(55)-C(56)	1.506(5)	C(66)-H(66B)	0.9900
C(55)-H(55A)	0.9900	C(67)-C(68)	1.454(8)
C(55)-H(55B)	0.9900	C(67)-H(67A)	0.9800
C(56)-C(57)	1.532(5)	C(67)-H(67B)	0.9800
C(56)-H(56A)	0.9900	C(67)-H(67C)	0.9800
C(56)-H(56B)	0.9900	C(68)-C(69)	1.432(8)
C(57)-C(58)	1.504(5)	C(68)-H(68A)	0.9900
C(57)-H(57A)	0.9900	C(68)-H(68B)	0.9900
C(57)-H(57B)	0.9900	C(69)-C(70)	1.499(7)
C(58)-H(58A)	0.9900	C(69)-H(69A)	0.9900
C(58)-H(58B)	0.9900	C(69)-H(69B)	0.9900
O(5)-C(62)	1.412(5)	C(70)-C(71)	1.527(7)
O(5)-C(59)	1.423(6)	C(70)-H(70A)	0.9900
C(59)-C(60)	1.477(8)	C(70)-H(70B)	0.9900
C(59)-H(59A)	0.9900	C(71)-H(71A)	0.9800
C(59)-H(59B)	0.9900	C(71)-H(71B)	0.9800
C(60)-C(61)	1.500(7)	C(71)-H(71C)	0.9800
C(60)-H(60A)	0.9900	C(7)-Fe(1)-C(1)	117.32(12)
C(60)-H(60B)	0.9900	C(7)-Fe(1)-C(13)	126.73(12)
C(61)-C(62)	1.487(6)	C(1)-Fe(1)-C(13)	115.87(11)
C(61)-H(61A)	0.9900	C(7)-Fe(1)-O(1)	87.62(10)
C(61)-H(61B)	0.9900	C(1)-Fe(1)-O(1)	91.20(10)
C(62)-H(62A)	0.9900	C(13)-Fe(1)-O(1)	88.60(10)
C(62)-H(62B)	0.9900	C(7)-Fe(1)-Fe(3)	58.97(8)
O(6)-C(63)	1.387(6)	C(1)-Fe(1)-Fe(3)	106.48(8)
O(6)-C(66)	1.388(6)	C(13)-Fe(1)-Fe(3)	107.85(8)
C(63)-C(64)	1.487(7)	O(1)-Fe(1)-Fe(3)	146.41(6)
C(63)-H(63A)	0.9900	C(7)-Fe(1)-Fe(2)	107.83(9)
C(63)-H(63B)	0.9900	C(1)-Fe(1)-Fe(2)	56.05(8)

C(13)-Fe(1)-Fe(2)	103.45(8)	C(31)-Fe(3)-Fe(2)	112.75(8)
O(1)-Fe(1)-Fe(2)	147.20(6)	C(7)-Fe(3)-Fe(2)	107.37(8)
Fe(3)-Fe(1)-Fe(2)	58.330(17)	C(19)-Fe(3)-Fe(2)	54.37(8)
C(7)-Fe(1)-Fe(4)	109.46(8)	O(3)-Fe(3)-Fe(2)	142.35(6)
C(1)-Fe(1)-Fe(4)	108.30(8)	C(31)-Fe(3)-Fe(4)	54.89(8)
C(13)-Fe(1)-Fe(4)	54.37(8)	C(7)-Fe(3)-Fe(4)	109.19(8)
O(1)-Fe(1)-Fe(4)	142.64(6)	C(19)-Fe(3)-Fe(4)	107.36(8)
Fe(3)-Fe(1)-Fe(4)	58.290(16)	O(3)-Fe(3)-Fe(4)	141.71(6)
Fe(2)-Fe(1)-Fe(4)	59.828(17)	Fe(2)-Fe(3)-Fe(4)	62.769(18)
C(19)-Fe(2)-C(25)	115.64(12)	C(31)-Fe(3)-Fe(1)	104.69(8)
C(19)-Fe(2)-C(1)	121.47(12)	C(7)-Fe(3)-Fe(1)	54.08(7)
C(25)-Fe(2)-C(1)	122.75(11)	C(19)-Fe(3)-Fe(1)	109.59(8)
C(19)-Fe(2)-O(2)	90.07(10)	O(3)-Fe(3)-Fe(1)	145.07(5)
C(25)-Fe(2)-O(2)	87.59(10)	Fe(2)-Fe(3)-Fe(1)	62.587(18)
C(1)-Fe(2)-O(2)	88.64(10)	Fe(4)-Fe(3)-Fe(1)	62.803(18)
C(19)-Fe(2)-Fe(3)	59.34(9)	C(31)-Fe(4)-C(13)	117.81(12)
C(25)-Fe(2)-Fe(3)	105.59(8)	C(31)-Fe(4)-C(25)	125.78(12)
C(1)-Fe(2)-Fe(3)	105.93(8)	C(13)-Fe(4)-C(25)	116.29(11)
O(2)-Fe(2)-Fe(3)	149.40(6)	C(31)-Fe(4)-O(4)	87.47(10)
C(19)-Fe(2)-Fe(4)	108.03(9)	C(13)-Fe(4)-O(4)	90.89(10)
C(25)-Fe(2)-Fe(4)	55.30(8)	C(25)-Fe(4)-O(4)	88.41(10)
C(1)-Fe(2)-Fe(4)	107.48(8)	C(31)-Fe(4)-Fe(3)	58.42(9)
O(2)-Fe(2)-Fe(4)	142.72(6)	C(13)-Fe(4)-Fe(3)	110.00(8)
Fe(3)-Fe(2)-Fe(4)	58.682(17)	C(25)-Fe(4)-Fe(3)	105.43(8)
C(19)-Fe(2)-Fe(1)	110.82(9)	O(4)-Fe(4)-Fe(3)	145.22(6)
C(25)-Fe(2)-Fe(1)	108.33(8)	C(31)-Fe(4)-Fe(2)	112.09(9)
C(1)-Fe(2)-Fe(1)	54.63(8)	C(13)-Fe(4)-Fe(2)	104.92(9)
O(2)-Fe(2)-Fe(1)	143.12(6)	C(25)-Fe(4)-Fe(2)	55.25(8)
Fe(3)-Fe(2)-Fe(1)	59.082(17)	O(4)-Fe(4)-Fe(2)	143.65(6)
Fe(4)-Fe(2)-Fe(1)	60.275(17)	Fe(3)-Fe(4)-Fe(2)	58.549(18)
C(31)-Fe(3)-C(7)	114.14(12)	C(31)-Fe(4)-Fe(1)	104.32(9)
C(31)-Fe(3)-C(19)	125.20(11)	C(13)-Fe(4)-Fe(1)	56.12(8)
C(7)-Fe(3)-C(19)	120.52(12)	C(25)-Fe(4)-Fe(1)	107.95(8)
C(31)-Fe(3)-O(3)	87.41(10)	O(4)-Fe(4)-Fe(1)	146.86(6)
C(7)-Fe(3)-O(3)	91.01(9)	Fe(3)-Fe(4)-Fe(1)	58.907(17)
C(19)-Fe(3)-O(3)	87.98(10)	Fe(2)-Fe(4)-Fe(1)	59.897(18)

C(6)-C(1)-C(2)	113.9(3)	C(10)-C(11)-C(12)	121.1(3)
C(6)-C(1)-Fe(1)	119.0(2)	C(10)-C(11)-H(11)	119.4
C(2)-C(1)-Fe(1)	113.1(2)	C(12)-C(11)-H(11)	119.4
C(6)-C(1)-Fe(2)	116.3(2)	C(11)-C(12)-C(7)	123.5(3)
C(2)-C(1)-Fe(2)	118.3(2)	C(11)-C(12)-H(12)	118.2
Fe(1)-C(1)-Fe(2)	69.32(9)	C(7)-C(12)-H(12)	118.2
C(3)-C(2)-C(1)	123.0(3)	C(14)-C(13)-C(18)	113.7(3)
C(3)-C(2)-H(2)	118.5	C(14)-C(13)-Fe(4)	123.2(2)
C(1)-C(2)-H(2)	118.5	C(18)-C(13)-Fe(4)	110.0(2)
C(4)-C(3)-C(2)	121.3(3)	C(14)-C(13)-Fe(1)	114.7(2)
C(4)-C(3)-H(3)	119.4	C(18)-C(13)-Fe(1)	119.2(2)
C(2)-C(3)-H(3)	119.4	Fe(4)-C(13)-Fe(1)	69.51(9)
C(5)-C(4)-C(3)	117.3(3)	C(15)-C(14)-C(13)	123.6(3)
C(5)-C(4)-C(37)	121.3(3)	C(15)-C(14)-H(14)	118.2
C(3)-C(4)-C(37)	121.5(3)	C(13)-C(14)-H(14)	118.2
C(4)-C(5)-C(6)	121.2(3)	C(16)-C(15)-C(14)	121.1(3)
C(4)-C(5)-H(5)	119.4	C(16)-C(15)-H(15)	119.5
C(6)-C(5)-H(5)	119.4	C(14)-C(15)-H(15)	119.5
C(5)-C(6)-C(1)	123.4(3)	C(17)-C(16)-C(15)	117.1(3)
C(5)-C(6)-H(6)	118.3	C(17)-C(16)-C(39)	121.1(3)
C(1)-C(6)-H(6)	118.3	C(15)-C(16)-C(39)	121.8(3)
C(12)-C(7)-C(8)	113.5(3)	C(18)-C(17)-C(16)	121.1(3)
C(12)-C(7)-Fe(1)	114.7(2)	C(18)-C(17)-H(17)	119.4
C(8)-C(7)-Fe(1)	127.3(2)	C(16)-C(17)-H(17)	119.4
C(12)-C(7)-Fe(3)	123.1(2)	C(17)-C(18)-C(13)	123.4(3)
C(8)-C(7)-Fe(3)	102.4(2)	C(17)-C(18)-H(18)	118.3
Fe(1)-C(7)-Fe(3)	66.95(8)	C(13)-C(18)-H(18)	118.3
C(9)-C(8)-C(7)	123.6(3)	C(24)-C(19)-C(20)	113.9(3)
C(9)-C(8)-H(8)	118.2	C(24)-C(19)-Fe(2)	115.6(2)
C(7)-C(8)-H(8)	118.2	C(20)-C(19)-Fe(2)	121.2(2)
C(10)-C(9)-C(8)	120.6(3)	C(24)-C(19)-Fe(3)	123.1(2)
C(10)-C(9)-H(9)	119.7	C(20)-C(19)-Fe(3)	108.9(2)
C(8)-C(9)-H(9)	119.7	Fe(2)-C(19)-Fe(3)	66.29(9)
C(11)-C(10)-C(9)	117.5(3)	C(21)-C(20)-C(19)	123.1(3)
C(11)-C(10)-C(38)	121.4(3)	C(21)-C(20)-H(20)	118.4
C(9)-C(10)-C(38)	121.1(3)	C(19)-C(20)-H(20)	118.4

C(20)-C(21)-C(22)	121.5(3)	C(36)-C(31)-Fe(3)	116.1(2)
C(20)-C(21)-H(21)	119.3	C(32)-C(31)-Fe(3)	114.0(2)
C(22)-C(21)-H(21)	119.3	Fe(4)-C(31)-Fe(3)	66.69(9)
C(21)-C(22)-C(23)	117.0(3)	C(33)-C(32)-C(31)	123.6(3)
C(21)-C(22)-C(40)	121.2(3)	C(33)-C(32)-H(32)	118.2
C(23)-C(22)-C(40)	121.8(3)	C(31)-C(32)-H(32)	118.2
C(24)-C(23)-C(22)	121.2(3)	C(34)-C(33)-C(32)	120.9(3)
C(24)-C(23)-H(23)	119.4	C(34)-C(33)-H(33)	119.5
C(22)-C(23)-H(23)	119.4	C(32)-C(33)-H(33)	119.5
C(23)-C(24)-C(19)	123.3(3)	C(33)-C(34)-C(35)	117.7(3)
C(23)-C(24)-H(24)	118.3	C(33)-C(34)-C(42)	121.1(3)
C(19)-C(24)-H(24)	118.3	C(35)-C(34)-C(42)	121.2(3)
C(26)-C(25)-C(30)	113.8(3)	C(34)-C(35)-C(36)	120.9(3)
C(26)-C(25)-Fe(2)	108.2(2)	C(34)-C(35)-H(35)	119.6
C(30)-C(25)-Fe(2)	124.9(2)	C(36)-C(35)-H(35)	119.6
C(26)-C(25)-Fe(4)	127.4(2)	C(35)-C(36)-C(31)	123.4(3)
C(30)-C(25)-Fe(4)	107.6(2)	C(35)-C(36)-H(36)	118.3
Fe(2)-C(25)-Fe(4)	69.44(9)	C(31)-C(36)-H(36)	118.3
C(27)-C(26)-C(25)	123.6(3)	C(4)-C(37)-H(37A)	109.5
C(27)-C(26)-H(26)	118.2	C(4)-C(37)-H(37B)	109.5
C(25)-C(26)-H(26)	118.2	H(37A)-C(37)-H(37B)	109.5
C(26)-C(27)-C(28)	121.1(3)	C(4)-C(37)-H(37C)	109.5
C(26)-C(27)-H(27)	119.5	H(37A)-C(37)-H(37C)	109.5
C(28)-C(27)-H(27)	119.5	H(37B)-C(37)-H(37C)	109.5
C(27)-C(28)-C(29)	117.5(3)	C(10)-C(38)-H(38A)	109.5
C(27)-C(28)-C(41)	120.6(3)	C(10)-C(38)-H(38B)	109.5
C(29)-C(28)-C(41)	121.9(3)	H(38A)-C(38)-H(38B)	109.5
C(30)-C(29)-C(28)	120.9(3)	C(10)-C(38)-H(38C)	109.5
C(30)-C(29)-H(29)	119.5	H(38A)-C(38)-H(38C)	109.5
C(28)-C(29)-H(29)	119.5	H(38B)-C(38)-H(38C)	109.5
C(29)-C(30)-C(25)	123.1(3)	C(16)-C(39)-H(39A)	109.5
C(29)-C(30)-H(30)	118.5	C(16)-C(39)-H(39B)	109.5
C(25)-C(30)-H(30)	118.5	H(39A)-C(39)-H(39B)	109.5
C(36)-C(31)-C(32)	113.5(3)	C(16)-C(39)-H(39C)	109.5
C(36)-C(31)-Fe(4)	121.2(2)	H(39A)-C(39)-H(39C)	109.5
C(32)-C(31)-Fe(4)	117.1(2)	H(39B)-C(39)-H(39C)	109.5

C(22)-C(40)-H(40A)	109.5	C(44)-C(45)-H(45B)	111.1
C(22)-C(40)-H(40B)	109.5	C(46)-C(45)-H(45B)	111.1
H(40A)-C(40)-H(40B)	109.5	H(45A)-C(45)-H(45B)	109.0
C(22)-C(40)-H(40C)	109.5	O(1)-C(46)-C(45)	105.5(3)
H(40A)-C(40)-H(40C)	109.5	O(1)-C(46)-H(46A)	110.6
H(40B)-C(40)-H(40C)	109.5	C(45)-C(46)-H(46A)	110.6
C(28)-C(41)-H(41A)	109.5	O(1)-C(46)-H(46B)	110.6
C(28)-C(41)-H(41B)	109.5	C(45)-C(46)-H(46B)	110.6
H(41A)-C(41)-H(41B)	109.5	H(46A)-C(46)-H(46B)	108.8
C(28)-C(41)-H(41C)	109.5	C(50)-O(2)-C(47)	105.5(3)
H(41A)-C(41)-H(41C)	109.5	C(50)-O(2)-Fe(2)	126.96(18)
H(41B)-C(41)-H(41C)	109.5	C(47)-O(2)-Fe(2)	118.8(2)
C(34)-C(42)-H(42A)	109.5	O(2)-C(47)-C(48)	106.2(3)
C(34)-C(42)-H(42B)	109.5	O(2)-C(47)-H(47A)	110.5
H(42A)-C(42)-H(42B)	109.5	C(48)-C(47)-H(47A)	110.5
C(34)-C(42)-H(42C)	109.5	O(2)-C(47)-H(47B)	110.5
H(42A)-C(42)-H(42C)	109.5	C(48)-C(47)-H(47B)	110.5
H(42B)-C(42)-H(42C)	109.5	H(47A)-C(47)-H(47B)	108.7
C(46)-O(1)-C(43)	109.3(2)	C(47)-C(48)-C(49)	105.9(3)
C(46)-O(1)-Fe(1)	122.56(18)	C(47)-C(48)-H(48A)	110.6
C(43)-O(1)-Fe(1)	121.39(19)	C(49)-C(48)-H(48A)	110.6
O(1)-C(43)-C(44)	106.5(3)	C(47)-C(48)-H(48B)	110.6
O(1)-C(43)-H(43A)	110.4	C(49)-C(48)-H(48B)	110.6
C(44)-C(43)-H(43A)	110.4	H(48A)-C(48)-H(48B)	108.7
O(1)-C(43)-H(43B)	110.4	C(48)-C(49)-C(50)	104.7(3)
C(44)-C(43)-H(43B)	110.4	C(48)-C(49)-H(49A)	110.8
H(43A)-C(43)-H(43B)	108.6	C(50)-C(49)-H(49A)	110.8
C(43)-C(44)-C(45)	107.5(4)	C(48)-C(49)-H(49B)	110.8
C(43)-C(44)-H(44A)	110.2	C(50)-C(49)-H(49B)	110.8
C(45)-C(44)-H(44A)	110.2	H(49A)-C(49)-H(49B)	108.9
C(43)-C(44)-H(44B)	110.2	O(2)-C(50)-C(49)	106.0(3)
C(45)-C(44)-H(44B)	110.2	O(2)-C(50)-H(50A)	110.5
H(44A)-C(44)-H(44B)	108.5	C(49)-C(50)-H(50A)	110.5
C(44)-C(45)-C(46)	103.4(3)	O(2)-C(50)-H(50B)	110.5
C(44)-C(45)-H(45A)	111.1	C(49)-C(50)-H(50B)	110.5
C(46)-C(45)-H(45A)	111.1	H(50A)-C(50)-H(50B)	108.7

C(51)-O(3)-C(54)	109.1(2)	C(55)-C(56)-C(57)	102.1(3)
C(51)-O(3)-Fe(3)	125.90(18)	C(55)-C(56)-H(56A)	111.3
C(54)-O(3)-Fe(3)	119.37(17)	C(57)-C(56)-H(56A)	111.3
O(3)-C(51)-C(52)	104.6(3)	C(55)-C(56)-H(56B)	111.3
O(3)-C(51)-H(51A)	110.8	C(57)-C(56)-H(56B)	111.3
C(52)-C(51)-H(51A)	110.8	H(56A)-C(56)-H(56B)	109.2
O(3)-C(51)-H(51B)	110.8	C(58)-C(57)-C(56)	101.2(3)
C(52)-C(51)-H(51B)	110.8	C(58)-C(57)-H(57A)	111.5
H(51A)-C(51)-H(51B)	108.9	C(56)-C(57)-H(57A)	111.5
C(53)-C(52)-C(51)	101.6(3)	C(58)-C(57)-H(57B)	111.5
C(53)-C(52)-H(52A)	111.5	C(56)-C(57)-H(57B)	111.5
C(51)-C(52)-H(52A)	111.5	H(57A)-C(57)-H(57B)	109.4
C(53)-C(52)-H(52B)	111.5	O(4)-C(58)-C(57)	105.8(3)
C(51)-C(52)-H(52B)	111.5	O(4)-C(58)-H(58A)	110.6
H(52A)-C(52)-H(52B)	109.3	C(57)-C(58)-H(58A)	110.6
C(52)-C(53)-C(54)	102.9(3)	O(4)-C(58)-H(58B)	110.6
C(52)-C(53)-H(53A)	111.2	C(57)-C(58)-H(58B)	110.6
C(54)-C(53)-H(53A)	111.2	H(58A)-C(58)-H(58B)	108.7
C(52)-C(53)-H(53B)	111.2	C(62)-O(5)-C(59)	107.3(3)
C(54)-C(53)-H(53B)	111.2	O(5)-C(59)-C(60)	108.2(4)
H(53A)-C(53)-H(53B)	109.1	O(5)-C(59)-H(59A)	110.1
O(3)-C(54)-C(53)	106.4(3)	C(60)-C(59)-H(59A)	110.1
O(3)-C(54)-H(54A)	110.5	O(5)-C(59)-H(59B)	110.1
C(53)-C(54)-H(54A)	110.5	C(60)-C(59)-H(59B)	110.1
O(3)-C(54)-H(54B)	110.5	H(59A)-C(59)-H(59B)	108.4
C(53)-C(54)-H(54B)	110.5	C(59)-C(60)-C(61)	104.6(5)
H(54A)-C(54)-H(54B)	108.6	C(59)-C(60)-H(60A)	110.8
C(55)-O(4)-C(58)	109.4(2)	C(61)-C(60)-H(60A)	110.8
C(55)-O(4)-Fe(4)	122.57(19)	C(59)-C(60)-H(60B)	110.8
C(58)-O(4)-Fe(4)	120.37(18)	C(61)-C(60)-H(60B)	110.8
O(4)-C(55)-C(56)	104.9(3)	H(60A)-C(60)-H(60B)	108.9
O(4)-C(55)-H(55A)	110.8	C(62)-C(61)-C(60)	101.5(4)
C(56)-C(55)-H(55A)	110.8	C(62)-C(61)-H(61A)	111.5
O(4)-C(55)-H(55B)	110.8	C(60)-C(61)-H(61A)	111.5
C(56)-C(55)-H(55B)	110.8	C(62)-C(61)-H(61B)	111.5
H(55A)-C(55)-H(55B)	108.8	C(60)-C(61)-H(61B)	111.5

H(61A)-C(61)-H(61B)	109.3	H(67A)-C(67)-H(67C)	109.5
O(5)-C(62)-C(61)	106.8(4)	H(67B)-C(67)-H(67C)	109.5
O(5)-C(62)-H(62A)	110.4	C(69)-C(68)-C(67)	119.4(6)
C(61)-C(62)-H(62A)	110.4	C(69)-C(68)-H(68A)	107.5
O(5)-C(62)-H(62B)	110.4	C(67)-C(68)-H(68A)	107.5
C(61)-C(62)-H(62B)	110.4	C(69)-C(68)-H(68B)	107.5
H(62A)-C(62)-H(62B)	108.6	C(67)-C(68)-H(68B)	107.5
C(63)-O(6)-C(66)	109.1(4)	H(68A)-C(68)-H(68B)	107.0
O(6)-C(63)-C(64)	108.8(4)	C(68)-C(69)-C(70)	118.7(6)
O(6)-C(63)-H(63A)	109.9	C(68)-C(69)-H(69A)	107.6
C(64)-C(63)-H(63A)	109.9	C(70)-C(69)-H(69A)	107.6
O(6)-C(63)-H(63B)	109.9	C(68)-C(69)-H(69B)	107.6
C(64)-C(63)-H(63B)	109.9	C(70)-C(69)-H(69B)	107.6
H(63A)-C(63)-H(63B)	108.3	H(69A)-C(69)-H(69B)	107.1
C(63)-C(64)-C(65)	105.6(4)	C(69)-C(70)-C(71)	110.9(5)
C(63)-C(64)-H(64A)	110.6	C(69)-C(70)-H(70A)	109.5
C(65)-C(64)-H(64A)	110.6	C(71)-C(70)-H(70A)	109.5
C(63)-C(64)-H(64B)	110.6	C(69)-C(70)-H(70B)	109.5
C(65)-C(64)-H(64B)	110.6	C(71)-C(70)-H(70B)	109.5
H(64A)-C(64)-H(64B)	108.8	H(70A)-C(70)-H(70B)	108.1
C(66)-C(65)-C(64)	102.6(4)	C(70)-C(71)-H(71A)	109.5
C(66)-C(65)-H(65A)	111.2	C(70)-C(71)-H(71B)	109.5
C(64)-C(65)-H(65A)	111.2	H(71A)-C(71)-H(71B)	109.5
C(66)-C(65)-H(65B)	111.2	C(70)-C(71)-H(71C)	109.5
C(64)-C(65)-H(65B)	111.2	H(71A)-C(71)-H(71C)	109.5
H(65A)-C(65)-H(65B)	109.2	H(71B)-C(71)-H(71C)	109.5
O(6)-C(66)-C(65)	108.2(4)		
O(6)-C(66)-H(66A)	110.1		
C(65)-C(66)-H(66A)	110.1		
O(6)-C(66)-H(66B)	110.1		
C(65)-C(66)-H(66B)	110.1		
H(66A)-C(66)-H(66B)	108.4		
C(68)-C(67)-H(67A)	109.5		
C(68)-C(67)-H(67B)	109.5		
H(67A)-C(67)-H(67B)	109.5		
C(68)-C(67)-H(67C)	109.5		

3.6 $Fe_4(\mu\text{-}p\text{-tolyl})_6(THF)_3 \cdot THF$ (2c)

REFERENCE NUMBER: neisc32

CRYSTAL STRUCTURE REPORT

$C_{58}\ H_{74}\ Fe_4\ O_4$

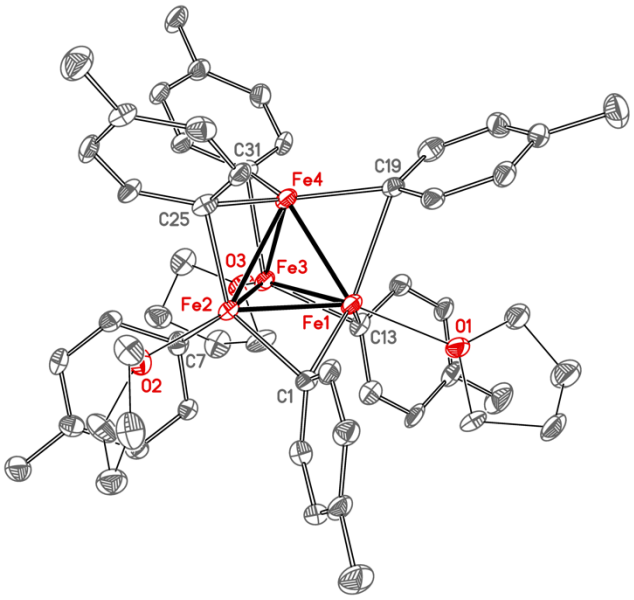
or

$Fe_4(\mu_2\text{-}p\text{-tolyl})_6(THF)_3 \cdot THF$

Report prepared for:

S. Carpenter, Prof. M. Neidig

July 08, 2017



William W. Brennessel

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120 Trustee Road

Rochester, NY 14627

Data collection

A crystal (0.40 x 0.14 x 0.06 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 90 seconds and a detector distance of 4.04 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 2940 strong reflections from the actual data collection after integration.[3] See Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SHELXT-2014/5[4] and refined using SHELXL-2017/1.[5] The space group $P2_1/c$ was determined based on systematic absences. 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 $R1 = 0.0525$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1150$ (F^2, all data).

Structure description

The structure is the one suggested. The asymmetric unit contains one tetranuclear iron complex and one cocrystallized well-separated THF solvent molecule, both in general positions. One THF ligand is modeled as disordered over two positions (0.69:0.31).

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 2017/1; *Acta. Cryst.* **2015**, C*71*, 3-8.

Some equations of interest:

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

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

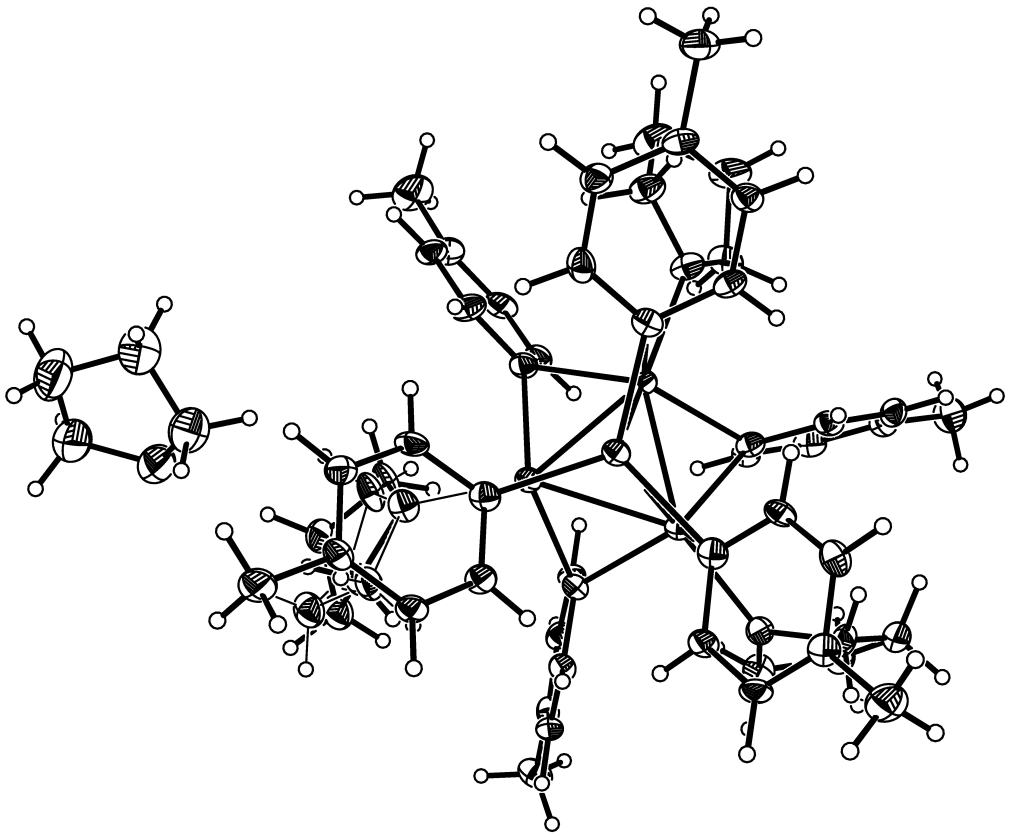
$$wR2 = [\Sigma[w(F_o^2-F_c^2)^2] \ / \ \Sigma\ [w(F_o^2)^2]]^{1/2}$$

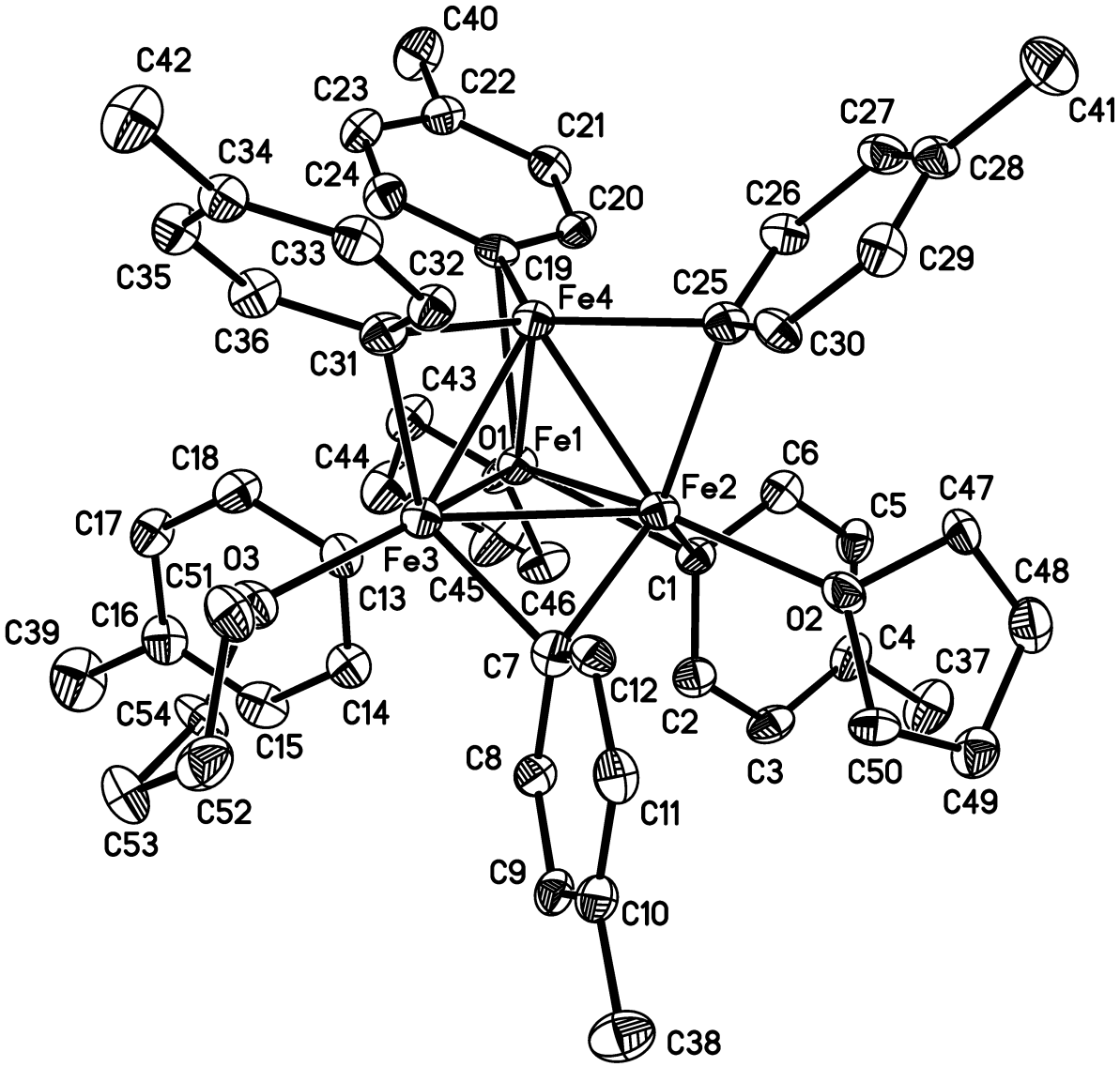
where $w = 1 \ / \ [\sigma^2 (F_o^2) + (aP)^2 + bP]$ and

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

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

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

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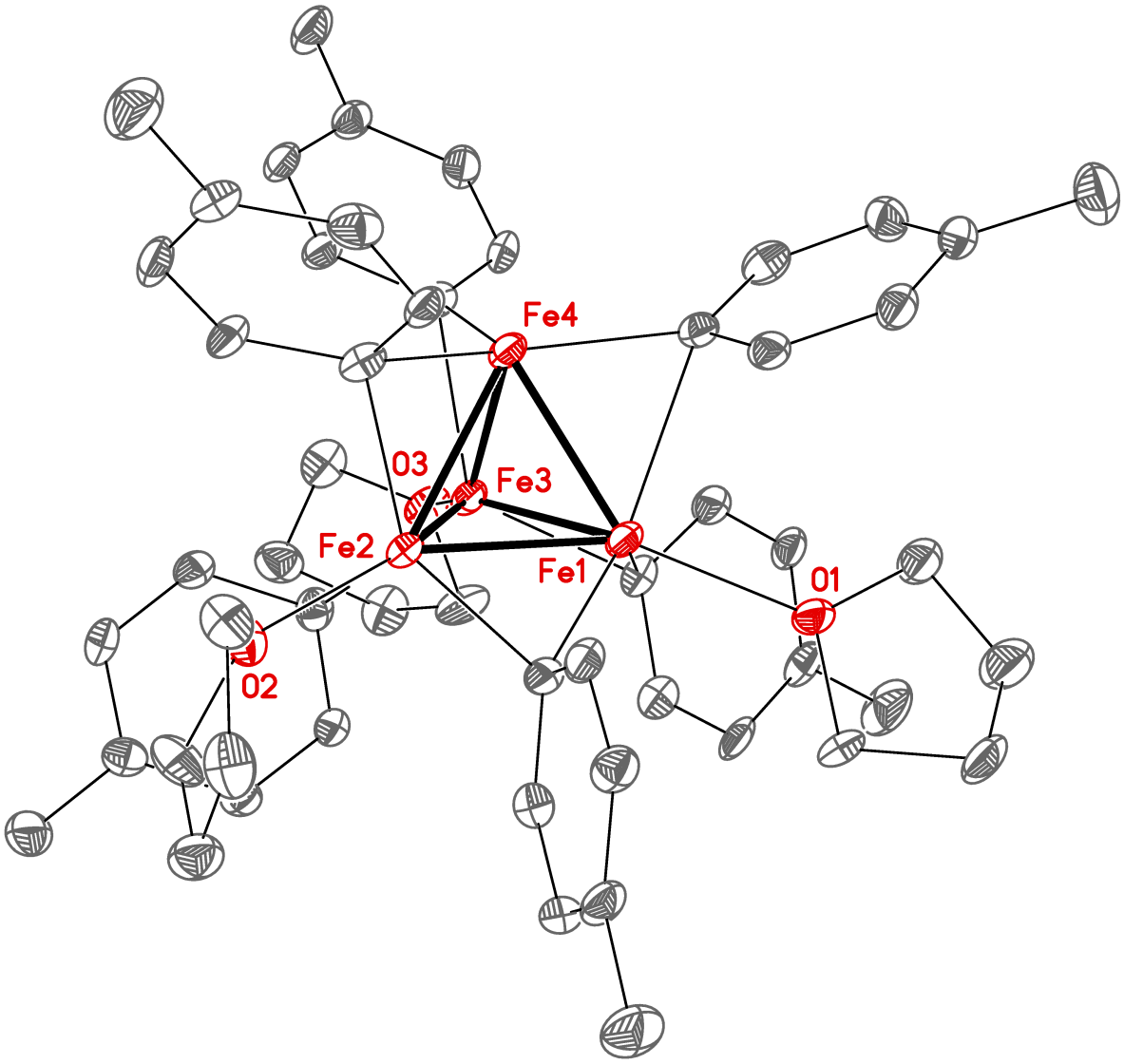
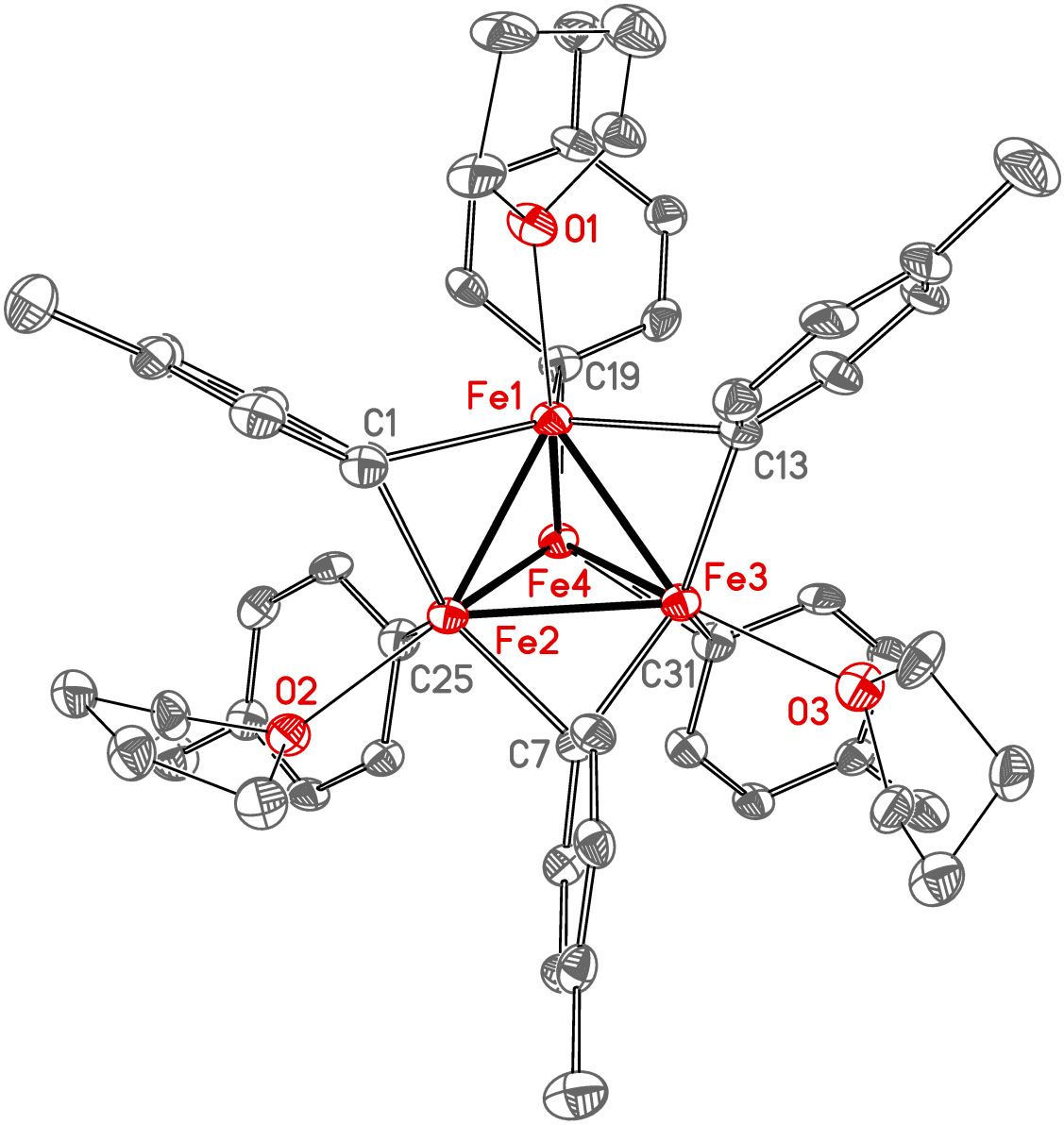
C42
C22
C23
C21
C41
C27
C34
C24
C28
C20
C26
C33
C32
C19
C29
C35
Fe4
C25
C36
C31
C30
C43
O1
Fe1
C44
C18
C6
Fe2
C47
C17
C5
C51
O3
Fe3
C48
C13
C45
C46
C1
C16
O2
C4
C39
C7
C12
C37
C54
C14
C2
C15
C8
C3
C50
C52
C11
C49
C53
C9
C10
C38


Fe4
O3
Fe3
Fe2
Fe1
O1
O2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C19
Fe1
C1
C13
Fe4
Fe3
Fe2
C25
C31
O2
O3
C7

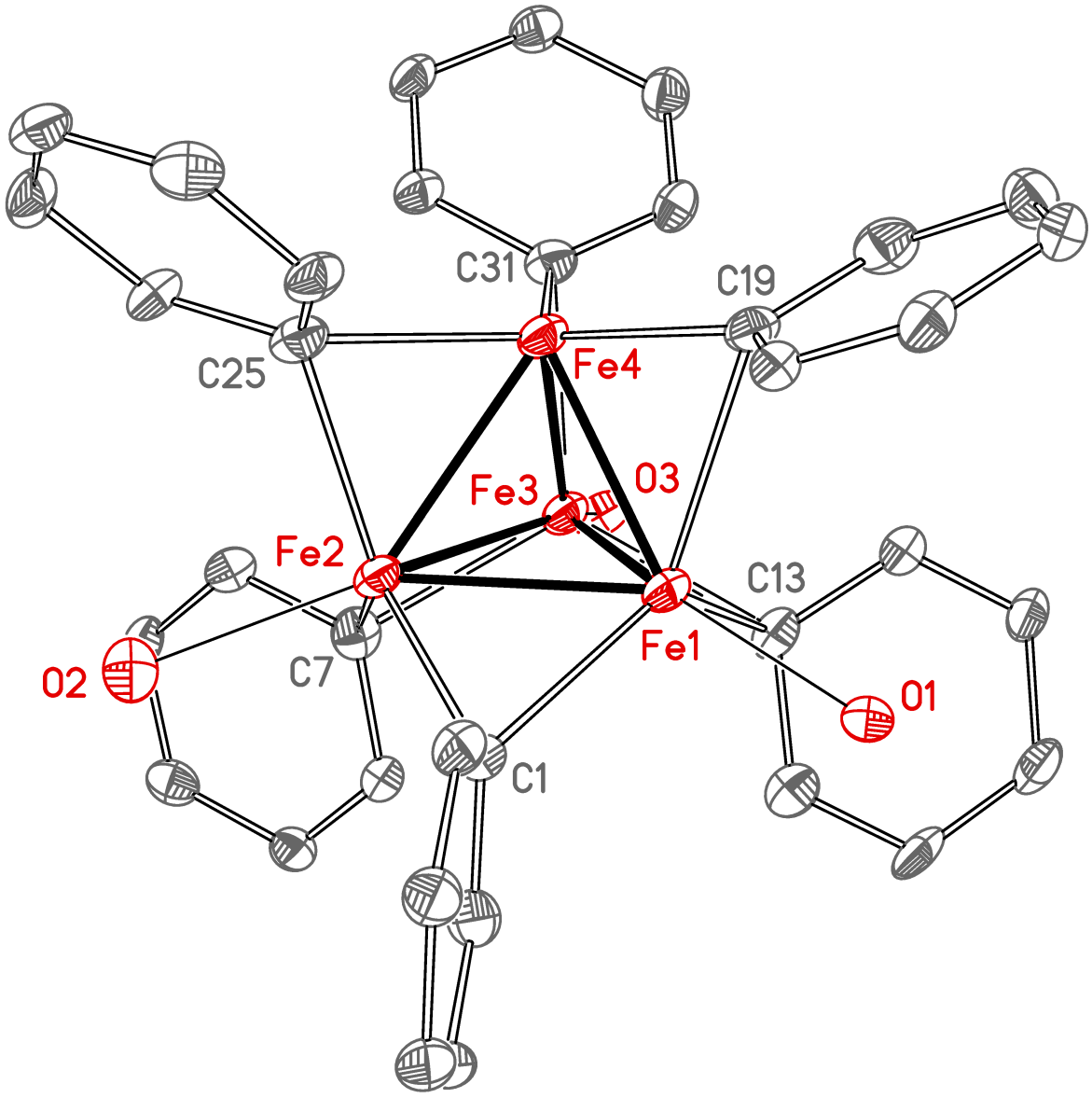

C31
C19
C25
Fe4
Fe3
O3
Fe2
C13
Fe1
O2
C7
O1
C1

Table S18. Crystal data and structure refinement for neisc32.

Identification code	neisc32	
Empirical formula	C58 H74 Fe4 O4	
Formula weight	1058.57	
Temperature	100.0(5) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 16.159(3) Å	α = 90°
	b = 17.191(3) Å	β = 98.545(3)°
	c = 18.694(3) Å	γ = 90°
Volume	5135.6(14) Å^3	
Z	4	
Density (calculated)	1.369 Mg/m^3	
Absorption coefficient	1.153 mm^{-1}	
F(000)	2232	
Crystal color, morphology	brown, needle	
Crystal size	0.40 x 0.14 x 0.06 mm^3	
Theta range for data collection	1.955 to 25.427°	
Index ranges	$-19 \le h \le 19$, $-19 \le k \le 20$, $-22 \le l \le 22$	
Reflections collected	46614	
Independent reflections	9390 [R(int) = 0.1604]	
Observed reflections	5638	
Completeness to theta = 25.242°	100.0%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.7452 and 0.5366	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9390 / 26 / 614	
Goodness-of-fit on F^2	1.006	
Final R indices [I>2sigma(I)]	$R1$ = 0.0525, $wR2$ = 0.0951	
R indices (all data)	$R1$ = 0.1176, $wR2$ = 0.1150	
Largest diff. peak and hole	0.529 and -0.609 e.Å^{-3}	

Table S19. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for neisc32. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Fe1	2394(1)	5760(1)	2188(1)	18(1)
Fe2	1394(1)	6574(1)	2696(1)	16(1)
Fe3	2961(1)	6664(1)	3151(1)	17(1)
Fe4	2144(1)	5515(1)	3388(1)	17(1)
C1	1199(3)	6256(3)	1585(2)	18(1)
C2	1271(3)	6861(3)	1091(2)	22(1)
C3	760(3)	6917(3)	421(2)	23(1)
C4	142(3)	6368(3)	202(2)	24(1)
C5	57(3)	5768(3)	682(2)	22(1)
C6	562(3)	5718(3)	1344(2)	21(1)
C7	2120(3)	7655(3)	3017(2)	20(1)
C8	2313(3)	8156(3)	2470(2)	19(1)
C9	2214(3)	8956(3)	2487(2)	21(1)
C10	1899(3)	9310(3)	3055(2)	21(1)
C11	1709(3)	8837(3)	3610(2)	22(1)
C12	1823(3)	8039(3)	3589(2)	19(1)
C13	3647(3)	6311(3)	2297(2)	21(1)
C14	3698(3)	6751(3)	1669(2)	24(1)
C15	4394(3)	6754(3)	1319(2)	26(1)
C16	5085(3)	6296(3)	1560(2)	26(1)
C17	5054(3)	5848(3)	2173(2)	24(1)
C18	4361(3)	5857(3)	2522(2)	25(1)
C19	2433(3)	4535(3)	2823(2)	19(1)
C20	1900(3)	4090(3)	2316(2)	20(1)
C21	2095(3)	3356(3)	2085(2)	22(1)
C22	2855(3)	2998(3)	2358(2)	22(1)
C23	3397(3)	3416(3)	2861(2)	24(1)
C24	3198(3)	4153(3)	3081(2)	24(1)
C25	856(3)	5815(3)	3471(2)	21(1)
C26	427(3)	5118(3)	3311(2)	19(1)
C27	-255(3)	4889(3)	3641(2)	24(1)

C28	-556(3)	5356(3)	4144(2)	22(1)
C29	-157(3)	6061(3)	4303(2)	22(1)
C30	524(3)	6279(3)	3982(2)	20(1)
C31	3053(3)	6031(3)	4210(2)	21(1)
C32	2665(3)	6310(3)	4778(2)	21(1)
C33	3024(3)	6298(3)	5495(2)	21(1)
C34	3830(3)	6018(3)	5699(2)	21(1)
C35	4250(3)	5748(3)	5148(2)	24(1)
C36	3874(3)	5754(3)	4433(2)	23(1)
C37	-420(3)	6443(3)	-509(2)	36(1)
C38	1778(3)	10177(3)	3066(3)	31(1)
C39	5833(3)	6270(4)	1162(3)	44(2)
C40	3067(3)	2212(3)	2090(3)	35(1)
C41	-1287(3)	5107(3)	4493(3)	35(1)
C42	4235(3)	5997(3)	6472(2)	32(1)
O1	2600(2)	5152(2)	1194(2)	24(1)
C43	3296(3)	4606(3)	1202(2)	29(1)
C44	3577(3)	4663(3)	482(3)	40(2)
C45	2775(3)	4902(3)	-3(2)	39(2)
C46	2379(3)	5458(3)	475(2)	27(1)
O2	211(2)	7251(2)	2558(2)	22(1)
C47	-596(3)	6872(3)	2441(3)	24(1)
C48	-1039(3)	7176(3)	1726(3)	30(1)
C49	-596(3)	7943(3)	1609(2)	28(1)
C50	74(3)	8023(3)	2263(3)	29(1)
O3	4028(2)	7388(2)	3618(2)	25(1)
C51	4045(17)	7829(9)	4277(8)	27(2)
C52	4236(5)	8652(5)	4062(5)	32(2)
C53	4864(5)	8500(5)	3543(4)	31(2)
C54	4463(11)	7814(7)	3115(6)	28(2)
O3'	4028(2)	7388(2)	3618(2)	25(1)
C51'	3920(40)	7790(20)	4285(18)	27(2)
C52'	4454(15)	8515(11)	4348(9)	32(2)
C53'	4441(13)	8711(9)	3556(10)	31(2)
C54'	4550(30)	7901(14)	3249(17)	28(2)
O4	6407(2)	7568(2)	4734(2)	46(1)

C55	6085(4)	6809(3)	4829(3)	46(2)
C56	6819(4)	6270(3)	4850(3)	47(2)
C57	7533(4)	6771(4)	5225(4)	61(2)
C58	7260(4)	7582(4)	5062(4)	60(2)

Table S20. Bond lengths [Å] and angles [°] for neisc32.

Fe(1)-O(1)	2.200(3)	C(7)-C(8)	1.406(6)
Fe(1)-C(13)	2.216(5)	C(8)-C(9)	1.386(6)
Fe(1)-C(1)	2.255(4)	C(8)-H(8)	0.9500
Fe(1)-Fe(4)	2.3751(9)	C(9)-C(10)	1.384(6)
Fe(1)-C(19)	2.412(5)	C(9)-H(9)	0.9500
Fe(1)-Fe(2)	2.4344(9)	C(10)-C(11)	1.387(6)
Fe(1)-Fe(3)	2.4504(9)	C(10)-C(38)	1.503(6)
Fe(2)-C(1)	2.124(4)	C(11)-C(12)	1.387(6)
Fe(2)-O(2)	2.220(3)	C(11)-H(11)	0.9500
Fe(2)-C(25)	2.222(4)	C(12)-H(12)	0.9500
Fe(2)-C(7)	2.232(5)	C(13)-C(18)	1.406(6)
Fe(2)-Fe(4)	2.4479(9)	C(13)-C(14)	1.410(6)
Fe(2)-Fe(3)	2.5532(10)	C(14)-C(15)	1.381(6)
Fe(3)-C(13)	2.161(4)	C(14)-H(14)	0.9500
Fe(3)-C(7)	2.171(5)	C(15)-C(16)	1.385(7)
Fe(3)-O(3')	2.199(3)	C(15)-H(15)	0.9500
Fe(3)-O(3)	2.199(3)	C(16)-C(17)	1.386(6)
Fe(3)-C(31)	2.244(4)	C(16)-C(39)	1.512(6)
Fe(3)-Fe(4)	2.4527(10)	C(17)-C(18)	1.378(6)
Fe(4)-C(19)	2.078(5)	C(17)-H(17)	0.9500
Fe(4)-C(31)	2.153(5)	C(18)-H(18)	0.9500
Fe(4)-C(25)	2.172(5)	C(19)-C(20)	1.409(6)
C(1)-C(6)	1.406(6)	C(19)-C(24)	1.419(6)
C(1)-C(2)	1.408(6)	C(20)-C(21)	1.385(6)
C(2)-C(3)	1.397(6)	C(20)-H(20)	0.9500
C(2)-H(2)	0.9500	C(21)-C(22)	1.401(6)
C(3)-C(4)	1.390(6)	C(21)-H(21)	0.9500
C(3)-H(3)	0.9500	C(22)-C(23)	1.386(6)
C(4)-C(5)	1.387(6)	C(22)-C(40)	1.498(6)
C(4)-C(37)	1.500(6)	C(23)-C(24)	1.384(6)
C(5)-C(6)	1.381(6)	C(23)-H(23)	0.9500
C(5)-H(5)	0.9500	C(24)-H(24)	0.9500
C(6)-H(6)	0.9500	C(25)-C(26)	1.395(6)
C(7)-C(12)	1.401(6)	C(25)-C(30)	1.410(6)

C(26)-C(27)	1.397(6)	C(42)-H(42B)	0.9800
C(26)-H(26)	0.9500	C(42)-H(42C)	0.9800
C(27)-C(28)	1.378(6)	O(1)-C(46)	1.439(5)
C(27)-H(27)	0.9500	O(1)-C(43)	1.462(5)
C(28)-C(29)	1.385(6)	C(43)-C(44)	1.486(6)
C(28)-C(41)	1.496(6)	C(43)-H(43A)	0.9900
C(29)-C(30)	1.382(6)	C(43)-H(43B)	0.9900
C(29)-H(29)	0.9500	C(44)-C(45)	1.524(7)
C(30)-H(30)	0.9500	C(44)-H(44A)	0.9900
C(31)-C(32)	1.397(6)	C(44)-H(44B)	0.9900
C(31)-C(36)	1.413(6)	C(45)-C(46)	1.513(6)
C(32)-C(33)	1.380(6)	C(45)-H(45A)	0.9900
C(32)-H(32)	0.9500	C(45)-H(45B)	0.9900
C(33)-C(34)	1.388(6)	C(46)-H(46A)	0.9900
C(33)-H(33)	0.9500	C(46)-H(46B)	0.9900
C(34)-C(35)	1.394(6)	O(2)-C(50)	1.442(5)
C(34)-C(42)	1.496(6)	O(2)-C(47)	1.446(5)
C(35)-C(36)	1.384(6)	C(47)-C(48)	1.512(6)
C(35)-H(35)	0.9500	C(47)-H(47A)	0.9900
C(36)-H(36)	0.9500	C(47)-H(47B)	0.9900
C(37)-H(37A)	0.9800	C(48)-C(49)	1.531(7)
C(37)-H(37B)	0.9800	C(48)-H(48A)	0.9900
C(37)-H(37C)	0.9800	C(48)-H(48B)	0.9900
C(38)-H(38A)	0.9800	C(49)-C(50)	1.514(6)
C(38)-H(38B)	0.9800	C(49)-H(49A)	0.9900
C(38)-H(38C)	0.9800	C(49)-H(49B)	0.9900
C(39)-H(39A)	0.9800	C(50)-H(50A)	0.9900
C(39)-H(39B)	0.9800	C(50)-H(50B)	0.9900
C(39)-H(39C)	0.9800	O(3)-C(51)	1.443(8)
C(40)-H(40A)	0.9800	O(3)-C(54)	1.453(8)
C(40)-H(40B)	0.9800	C(51)-C(52)	1.514(12)
C(40)-H(40C)	0.9800	C(51)-H(51A)	0.9900
C(41)-H(41A)	0.9800	C(51)-H(51B)	0.9900
C(41)-H(41B)	0.9800	C(52)-C(53)	1.526(10)
C(41)-H(41C)	0.9800	C(52)-H(52A)	0.9900
C(42)-H(42A)	0.9800	C(52)-H(52B)	0.9900

C(53)-C(54)	1.516(10)	C(1)-Fe(1)-Fe(4)	106.57(11)
C(53)-H(53A)	0.9900	O(1)-Fe(1)-C(19)	90.29(13)
C(53)-H(53B)	0.9900	C(13)-Fe(1)-C(19)	111.84(16)
C(54)-H(54A)	0.9900	C(1)-Fe(1)-C(19)	122.24(16)
C(54)-H(54B)	0.9900	Fe(4)-Fe(1)-C(19)	51.45(10)
O(3')-C(51')	1.457(14)	O(1)-Fe(1)-Fe(2)	142.61(8)
O(3')-C(54')	1.466(14)	C(13)-Fe(1)-Fe(2)	111.58(12)
C(51')-C(52')	1.521(16)	C(1)-Fe(1)-Fe(2)	53.69(11)
C(51')-H(51C)	0.9900	Fe(4)-Fe(1)-Fe(2)	61.17(3)
C(51')-H(51D)	0.9900	C(19)-Fe(1)-Fe(2)	106.29(11)
C(52')-C(53')	1.516(16)	O(1)-Fe(1)-Fe(3)	146.59(9)
C(52')-H(52C)	0.9900	C(13)-Fe(1)-Fe(3)	54.89(11)
C(52')-H(52D)	0.9900	C(1)-Fe(1)-Fe(3)	109.03(12)
C(53')-C(54')	1.527(16)	Fe(4)-Fe(1)-Fe(3)	61.07(3)
C(53')-H(53C)	0.9900	C(19)-Fe(1)-Fe(3)	102.28(11)
C(53')-H(53D)	0.9900	Fe(2)-Fe(1)-Fe(3)	63.02(3)
C(54')-H(54C)	0.9900	C(1)-Fe(2)-O(2)	91.20(15)
C(54')-H(54D)	0.9900	C(1)-Fe(2)-C(25)	118.07(17)
O(4)-C(58)	1.424(6)	O(2)-Fe(2)-C(25)	88.22(15)
O(4)-C(55)	1.425(6)	C(1)-Fe(2)-C(7)	118.26(17)
C(55)-C(56)	1.501(8)	O(2)-Fe(2)-C(7)	90.23(14)
C(55)-H(55A)	0.9900	C(25)-Fe(2)-C(7)	123.68(17)
C(55)-H(55B)	0.9900	C(1)-Fe(2)-Fe(1)	58.84(12)
C(56)-C(57)	1.524(8)	O(2)-Fe(2)-Fe(1)	150.04(8)
C(56)-H(56A)	0.9900	C(25)-Fe(2)-Fe(1)	105.28(13)
C(56)-H(56B)	0.9900	C(7)-Fe(2)-Fe(1)	103.37(12)
C(57)-C(58)	1.481(8)	C(1)-Fe(2)-Fe(4)	108.38(13)
C(57)-H(57A)	0.9900	O(2)-Fe(2)-Fe(4)	143.20(8)
C(57)-H(57B)	0.9900	C(25)-Fe(2)-Fe(4)	55.18(12)
C(58)-H(58A)	0.9900	C(7)-Fe(2)-Fe(4)	106.16(12)
C(58)-H(58B)	0.9900	Fe(1)-Fe(2)-Fe(4)	58.22(3)
O(1)-Fe(1)-C(13)	91.72(14)	C(1)-Fe(2)-Fe(3)	109.75(12)
O(1)-Fe(1)-C(1)	89.04(14)	O(2)-Fe(2)-Fe(3)	143.26(8)
C(13)-Fe(1)-C(1)	125.91(17)	C(25)-Fe(2)-Fe(3)	106.43(12)
O(1)-Fe(1)-Fe(4)	141.46(9)	C(7)-Fe(2)-Fe(3)	53.46(12)
C(13)-Fe(1)-Fe(4)	105.72(12)	Fe(1)-Fe(2)-Fe(3)	58.79(3)

Fe(4)-Fe(2)-Fe(3)	58.69(3)	C(25)-Fe(4)-Fe(2)	57.13(12)
C(13)-Fe(3)-C(7)	121.01(17)	Fe(1)-Fe(4)-Fe(2)	60.61(3)
C(13)-Fe(3)-O(3')	89.68(15)	C(19)-Fe(4)-Fe(3)	113.14(12)
C(7)-Fe(3)-O(3')	92.40(15)	C(31)-Fe(4)-Fe(3)	57.88(12)
C(13)-Fe(3)-O(3)	89.68(15)	C(25)-Fe(4)-Fe(3)	111.66(13)
C(7)-Fe(3)-O(3)	92.40(15)	Fe(1)-Fe(4)-Fe(3)	60.98(3)
C(13)-Fe(3)-C(31)	122.81(17)	Fe(2)-Fe(4)-Fe(3)	62.80(3)
C(7)-Fe(3)-C(31)	116.17(17)	C(6)-C(1)-C(2)	113.8(4)
O(3')-Fe(3)-C(31)	88.80(14)	C(6)-C(1)-Fe(2)	118.6(3)
O(3)-Fe(3)-C(31)	88.80(14)	C(2)-C(1)-Fe(2)	115.9(3)
C(13)-Fe(3)-Fe(1)	57.04(12)	C(6)-C(1)-Fe(1)	116.3(3)
C(7)-Fe(3)-Fe(1)	104.69(12)	C(2)-C(1)-Fe(1)	117.3(3)
O(3')-Fe(3)-Fe(1)	146.72(8)	Fe(2)-C(1)-Fe(1)	67.47(13)
O(3)-Fe(3)-Fe(1)	146.72(8)	C(3)-C(2)-C(1)	122.9(4)
C(31)-Fe(3)-Fe(1)	107.99(12)	C(3)-C(2)-H(2)	118.5
C(13)-Fe(3)-Fe(4)	104.88(13)	C(1)-C(2)-H(2)	118.5
C(7)-Fe(3)-Fe(4)	107.95(13)	C(4)-C(3)-C(2)	121.4(4)
O(3')-Fe(3)-Fe(4)	142.72(8)	C(4)-C(3)-H(3)	119.3
O(3)-Fe(3)-Fe(4)	142.72(8)	C(2)-C(3)-H(3)	119.3
C(31)-Fe(3)-Fe(4)	54.36(12)	C(5)-C(4)-C(3)	116.7(4)
Fe(1)-Fe(3)-Fe(4)	57.95(3)	C(5)-C(4)-C(37)	122.2(5)
C(13)-Fe(3)-Fe(2)	109.16(12)	C(3)-C(4)-C(37)	121.1(4)
C(7)-Fe(3)-Fe(2)	55.67(12)	C(6)-C(5)-C(4)	121.6(5)
O(3')-Fe(3)-Fe(2)	147.84(9)	C(6)-C(5)-H(5)	119.2
O(3)-Fe(3)-Fe(2)	147.84(9)	C(4)-C(5)-H(5)	119.2
C(31)-Fe(3)-Fe(2)	101.39(12)	C(5)-C(6)-C(1)	123.6(5)
Fe(1)-Fe(3)-Fe(2)	58.18(3)	C(5)-C(6)-H(6)	118.2
Fe(4)-Fe(3)-Fe(2)	58.51(3)	C(1)-C(6)-H(6)	118.2
C(19)-Fe(4)-C(31)	121.01(18)	C(12)-C(7)-C(8)	113.9(4)
C(19)-Fe(4)-C(25)	121.29(18)	C(12)-C(7)-Fe(3)	124.1(3)
C(31)-Fe(4)-C(25)	114.25(17)	C(8)-C(7)-Fe(3)	111.2(3)
C(19)-Fe(4)-Fe(1)	65.20(12)	C(12)-C(7)-Fe(2)	111.8(3)
C(31)-Fe(4)-Fe(1)	113.99(12)	C(8)-C(7)-Fe(2)	118.6(3)
C(25)-Fe(4)-Fe(1)	108.98(12)	Fe(3)-C(7)-Fe(2)	70.87(14)
C(19)-Fe(4)-Fe(2)	117.71(12)	C(9)-C(8)-C(7)	123.5(4)
C(31)-Fe(4)-Fe(2)	107.61(13)	C(9)-C(8)-H(8)	118.2

C(7)-C(8)-H(8)	118.2	C(24)-C(19)-Fe(4)	117.0(3)
C(10)-C(9)-C(8)	120.7(4)	C(20)-C(19)-Fe(1)	100.1(3)
C(10)-C(9)-H(9)	119.6	C(24)-C(19)-Fe(1)	121.9(3)
C(8)-C(9)-H(9)	119.6	Fe(4)-C(19)-Fe(1)	63.35(13)
C(9)-C(10)-C(11)	117.6(4)	C(21)-C(20)-C(19)	124.1(4)
C(9)-C(10)-C(38)	120.6(4)	C(21)-C(20)-H(20)	118.0
C(11)-C(10)-C(38)	121.8(4)	C(19)-C(20)-H(20)	118.0
C(12)-C(11)-C(10)	120.9(4)	C(20)-C(21)-C(22)	121.0(4)
C(12)-C(11)-H(11)	119.6	C(20)-C(21)-H(21)	119.5
C(10)-C(11)-H(11)	119.6	C(22)-C(21)-H(21)	119.5
C(11)-C(12)-C(7)	123.3(4)	C(23)-C(22)-C(21)	116.7(4)
C(11)-C(12)-H(12)	118.3	C(23)-C(22)-C(40)	122.8(4)
C(7)-C(12)-H(12)	118.3	C(21)-C(22)-C(40)	120.5(4)
C(18)-C(13)-C(14)	113.5(4)	C(24)-C(23)-C(22)	121.7(4)
C(18)-C(13)-Fe(3)	115.0(3)	C(24)-C(23)-H(23)	119.2
C(14)-C(13)-Fe(3)	124.4(3)	C(22)-C(23)-H(23)	119.2
C(18)-C(13)-Fe(1)	119.1(3)	C(23)-C(24)-C(19)	123.5(4)
C(14)-C(13)-Fe(1)	108.7(3)	C(23)-C(24)-H(24)	118.3
Fe(3)-C(13)-Fe(1)	68.07(13)	C(19)-C(24)-H(24)	118.3
C(15)-C(14)-C(13)	123.3(5)	C(26)-C(25)-C(30)	113.9(4)
C(15)-C(14)-H(14)	118.4	C(26)-C(25)-Fe(4)	103.3(3)
C(13)-C(14)-H(14)	118.4	C(30)-C(25)-Fe(4)	130.3(3)
C(14)-C(15)-C(16)	121.2(4)	C(26)-C(25)-Fe(2)	126.5(3)
C(14)-C(15)-H(15)	119.4	C(30)-C(25)-Fe(2)	109.6(3)
C(16)-C(15)-H(15)	119.4	Fe(4)-C(25)-Fe(2)	67.69(13)
C(15)-C(16)-C(17)	117.3(4)	C(25)-C(26)-C(27)	123.1(4)
C(15)-C(16)-C(39)	121.4(4)	C(25)-C(26)-H(26)	118.5
C(17)-C(16)-C(39)	121.3(5)	C(27)-C(26)-H(26)	118.5
C(18)-C(17)-C(16)	121.1(5)	C(28)-C(27)-C(26)	121.2(4)
C(18)-C(17)-H(17)	119.5	C(28)-C(27)-H(27)	119.4
C(16)-C(17)-H(17)	119.5	C(26)-C(27)-H(27)	119.4
C(17)-C(18)-C(13)	123.6(4)	C(27)-C(28)-C(29)	117.2(4)
C(17)-C(18)-H(18)	118.2	C(27)-C(28)-C(41)	120.8(5)
C(13)-C(18)-H(18)	118.2	C(29)-C(28)-C(41)	122.0(4)
C(20)-C(19)-C(24)	113.0(4)	C(30)-C(29)-C(28)	121.3(4)
C(20)-C(19)-Fe(4)	128.4(3)	C(30)-C(29)-H(29)	119.4

C(28)-C(29)-H(29)	119.4	H(38B)-C(38)-H(38C)	109.5
C(29)-C(30)-C(25)	123.2(4)	C(16)-C(39)-H(39A)	109.5
C(29)-C(30)-H(30)	118.4	C(16)-C(39)-H(39B)	109.5
C(25)-C(30)-H(30)	118.4	H(39A)-C(39)-H(39B)	109.5
C(32)-C(31)-C(36)	113.6(4)	C(16)-C(39)-H(39C)	109.5
C(32)-C(31)-Fe(4)	110.3(3)	H(39A)-C(39)-H(39C)	109.5
C(36)-C(31)-Fe(4)	125.6(3)	H(39B)-C(39)-H(39C)	109.5
C(32)-C(31)-Fe(3)	121.7(3)	C(22)-C(40)-H(40A)	109.5
C(36)-C(31)-Fe(3)	111.1(3)	C(22)-C(40)-H(40B)	109.5
Fe(4)-C(31)-Fe(3)	67.76(13)	H(40A)-C(40)-H(40B)	109.5
C(33)-C(32)-C(31)	124.2(4)	C(22)-C(40)-H(40C)	109.5
C(33)-C(32)-H(32)	117.9	H(40A)-C(40)-H(40C)	109.5
C(31)-C(32)-H(32)	117.9	H(40B)-C(40)-H(40C)	109.5
C(32)-C(33)-C(34)	120.9(4)	C(28)-C(41)-H(41A)	109.5
C(32)-C(33)-H(33)	119.5	C(28)-C(41)-H(41B)	109.5
C(34)-C(33)-H(33)	119.5	H(41A)-C(41)-H(41B)	109.5
C(33)-C(34)-C(35)	116.9(4)	C(28)-C(41)-H(41C)	109.5
C(33)-C(34)-C(42)	122.1(4)	H(41A)-C(41)-H(41C)	109.5
C(35)-C(34)-C(42)	121.0(4)	H(41B)-C(41)-H(41C)	109.5
C(36)-C(35)-C(34)	121.3(4)	C(34)-C(42)-H(42A)	109.5
C(36)-C(35)-H(35)	119.3	C(34)-C(42)-H(42B)	109.5
C(34)-C(35)-H(35)	119.3	H(42A)-C(42)-H(42B)	109.5
C(35)-C(36)-C(31)	123.0(4)	C(34)-C(42)-H(42C)	109.5
C(35)-C(36)-H(36)	118.5	H(42A)-C(42)-H(42C)	109.5
C(31)-C(36)-H(36)	118.5	H(42B)-C(42)-H(42C)	109.5
C(4)-C(37)-H(37A)	109.5	C(46)-O(1)-C(43)	109.1(3)
C(4)-C(37)-H(37B)	109.5	C(46)-O(1)-Fe(1)	124.6(3)
H(37A)-C(37)-H(37B)	109.5	C(43)-O(1)-Fe(1)	120.6(2)
C(4)-C(37)-H(37C)	109.5	O(1)-C(43)-C(44)	106.7(4)
H(37A)-C(37)-H(37C)	109.5	O(1)-C(43)-H(43A)	110.4
H(37B)-C(37)-H(37C)	109.5	C(44)-C(43)-H(43A)	110.4
C(10)-C(38)-H(38A)	109.5	O(1)-C(43)-H(43B)	110.4
C(10)-C(38)-H(38B)	109.5	C(44)-C(43)-H(43B)	110.4
H(38A)-C(38)-H(38B)	109.5	H(43A)-C(43)-H(43B)	108.6
C(10)-C(38)-H(38C)	109.5	C(43)-C(44)-C(45)	102.0(4)
H(38A)-C(38)-H(38C)	109.5	C(43)-C(44)-H(44A)	111.4

C(45)-C(44)-H(44A)	111.4	H(49A)-C(49)-H(49B)	108.8
C(43)-C(44)-H(44B)	111.4	O(2)-C(50)-C(49)	106.0(4)
C(45)-C(44)-H(44B)	111.4	O(2)-C(50)-H(50A)	110.5
H(44A)-C(44)-H(44B)	109.2	C(49)-C(50)-H(50A)	110.5
C(46)-C(45)-C(44)	102.5(4)	O(2)-C(50)-H(50B)	110.5
C(46)-C(45)-H(45A)	111.3	C(49)-C(50)-H(50B)	110.5
C(44)-C(45)-H(45A)	111.3	H(50A)-C(50)-H(50B)	108.7
C(46)-C(45)-H(45B)	111.3	C(51)-O(3)-C(54)	109.7(5)
C(44)-C(45)-H(45B)	111.3	C(51)-O(3)-Fe(3)	123.5(8)
H(45A)-C(45)-H(45B)	109.2	C(54)-O(3)-Fe(3)	117.0(6)
O(1)-C(46)-C(45)	104.6(4)	O(3)-C(51)-C(52)	104.1(8)
O(1)-C(46)-H(46A)	110.8	O(3)-C(51)-H(51A)	110.9
C(45)-C(46)-H(46A)	110.8	C(52)-C(51)-H(51A)	110.9
O(1)-C(46)-H(46B)	110.8	O(3)-C(51)-H(51B)	110.9
C(45)-C(46)-H(46B)	110.8	C(52)-C(51)-H(51B)	110.9
H(46A)-C(46)-H(46B)	108.9	H(51A)-C(51)-H(51B)	109.0
C(50)-O(2)-C(47)	105.8(3)	C(51)-C(52)-C(53)	100.9(8)
C(50)-O(2)-Fe(2)	127.4(3)	C(51)-C(52)-H(52A)	111.6
C(47)-O(2)-Fe(2)	121.6(3)	C(53)-C(52)-H(52A)	111.6
O(2)-C(47)-C(48)	106.0(4)	C(51)-C(52)-H(52B)	111.6
O(2)-C(47)-H(47A)	110.5	C(53)-C(52)-H(52B)	111.6
C(48)-C(47)-H(47A)	110.5	H(52A)-C(52)-H(52B)	109.4
O(2)-C(47)-H(47B)	110.5	C(54)-C(53)-C(52)	101.3(7)
C(48)-C(47)-H(47B)	110.5	C(54)-C(53)-H(53A)	111.5
H(47A)-C(47)-H(47B)	108.7	C(52)-C(53)-H(53A)	111.5
C(47)-C(48)-C(49)	104.7(4)	C(54)-C(53)-H(53B)	111.5
C(47)-C(48)-H(48A)	110.8	C(52)-C(53)-H(53B)	111.5
C(49)-C(48)-H(48A)	110.8	H(53A)-C(53)-H(53B)	109.3
C(47)-C(48)-H(48B)	110.8	O(3)-C(54)-C(53)	105.1(6)
C(49)-C(48)-H(48B)	110.8	O(3)-C(54)-H(54A)	110.7
H(48A)-C(48)-H(48B)	108.9	C(53)-C(54)-H(54A)	110.7
C(50)-C(49)-C(48)	104.9(4)	O(3)-C(54)-H(54B)	110.7
C(50)-C(49)-H(49A)	110.8	C(53)-C(54)-H(54B)	110.7
C(48)-C(49)-H(49A)	110.8	H(54A)-C(54)-H(54B)	108.8
C(50)-C(49)-H(49B)	110.8	C(51')-O(3')-C(54')	105.9(11)
C(48)-C(49)-H(49B)	110.8	C(51')-O(3')-Fe(3)	114.6(18)

C(54')-O(3')-Fe(3)	129.0(16)	C(57)-C(56)-H(56B)	111.4
O(3')-C(51')-C(52')	108.1(13)	H(56A)-C(56)-H(56B)	109.3
O(3')-C(51')-H(51C)	110.1	C(58)-C(57)-C(56)	104.7(5)
C(52')-C(51')-H(51C)	110.1	C(58)-C(57)-H(57A)	110.8
O(3')-C(51')-H(51D)	110.1	C(56)-C(57)-H(57A)	110.8
C(52')-C(51')-H(51D)	110.1	C(58)-C(57)-H(57B)	110.8
H(51C)-C(51')-H(51D)	108.4	C(56)-C(57)-H(57B)	110.8
C(53')-C(52')-C(51')	100.5(15)	H(57A)-C(57)-H(57B)	108.9
C(53')-C(52')-H(52C)	111.7	O(4)-C(58)-C(57)	108.2(5)
C(51')-C(52')-H(52C)	111.7	O(4)-C(58)-H(58A)	110.1
C(53')-C(52')-H(52D)	111.7	C(57)-C(58)-H(58A)	110.1
C(51')-C(52')-H(52D)	111.7	O(4)-C(58)-H(58B)	110.1
H(52C)-C(52')-H(52D)	109.4	C(57)-C(58)-H(58B)	110.1
C(52')-C(53')-C(54')	100.3(15)	H(58A)-C(58)-H(58B)	108.4
C(52')-C(53')-H(53C)	111.7		
C(54')-C(53')-H(53C)	111.7		
C(52')-C(53')-H(53D)	111.7		
C(54')-C(53')-H(53D)	111.7		
H(53C)-C(53')-H(53D)	109.5		
O(3')-C(54')-C(53')	105.2(13)		
O(3')-C(54')-H(54C)	110.7		
C(53')-C(54')-H(54C)	110.7		
O(3')-C(54')-H(54D)	110.7		
C(53')-C(54')-H(54D)	110.7		
H(54C)-C(54')-H(54D)	108.8		
C(58)-O(4)-C(55)	108.1(4)		
O(4)-C(55)-C(56)	105.4(4)		
O(4)-C(55)-H(55A)	110.7		
C(56)-C(55)-H(55A)	110.7		
O(4)-C(55)-H(55B)	110.7		
C(56)-C(55)-H(55B)	110.7		
H(55A)-C(55)-H(55B)	108.8		
C(55)-C(56)-C(57)	101.9(5)		
C(55)-C(56)-H(56A)	111.4		
C(57)-C(56)-H(56A)	111.4		
C(55)-C(56)-H(56B)	111.4		

3.7 $Fe_4(\mu\text{-}4\text{-}F\text{-}Ph)_6(THF)_4$ (2d)

REFERENCE NUMBER: neisc52

CRYSTAL STRUCTURE REPORT

$C_{52}\ H_{56}\ F_6\ Fe_4\ O_4$

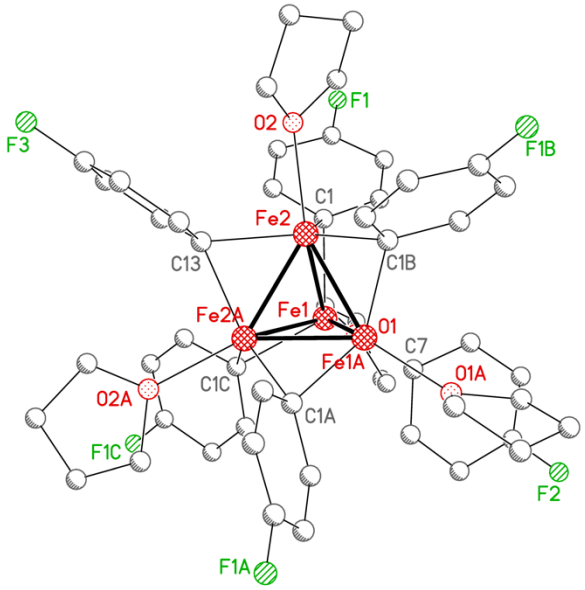
or

$Fe_4(\mu\text{-}4\text{-}F\text{-}Ph)_6(THF)_4 \cdot x\text{Solvent}$

Report prepared for:

S. Carpenter, Prof. M. Neidig

April 04, 2018



William W. Brennessel

X-ray Crystallographic Facility

Department of Chemistry, University of Rochester

120 Trustee Road

Rochester, NY 14627

Data collection

A crystal (0.164 x 0.152 x 0.124 mm^3) was placed onto a thin glass optical fiber or a nylon loop and mounted on a XtaLab Synergy-S Dualflex diffractometer equipped with a HyPix-6000HE HPC area detector for data collection at 100.0(1) K. A preliminary set of cell constants and an orientation matrix were calculated from a small sampling of reflections.[1] A short pre-experiment was run, from which an optimal data collection strategy was determined. The full data collection was carried out using a PhotonJet (Mo) X-ray Source with a frame time of 71.89 seconds and a detector distance of 40.0 mm. Series of frames were collected in 0.50º steps in ω at different 2θ, κ, and ϕ settings. After the intensity data were corrected for absorption, the final cell constants were calculated from the xyz centroids of 12384 strong reflections from the actual data collection after integration.[1] See Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SHELXT-2018/2[2] and refined using SHELXL-2018/3.[3] The space group $I4_1/amd$ was determined based on systematic absences. Most or all non-hydrogen atoms were assigned from the solution. Full-matrix least squares / difference Fourier cycles were performed which located any 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 in channels along [010] were fixed and added to the calculated structure factors using the SQUEEZE routine of program Platon,[4] which determined there to be 660 electrons in 1850 $Å^3$ fixed per unit cell. Because the exact identity and amount of solvent was unknown, no solvent was included in the atom list or molecular formula. Thus all calculated quantities that derive from the molecular formula (e.g., F(000), density, molecular weight, etc.) are known to be incorrect.

The final full matrix least squares refinement converged to $R1 = 0.0626$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.2041$ (F^2, all data).

Structure description

The structure is the one suggested. The asymmetric unit contains one-half of a tetranuclear iron complex located at a crystallographic *2mm.* position and cocrystallized solvent that was not modeled (see above). Phenyl ligand C1-C6/F1 is modeled as disordered over two general positions (fixed at 0.50:0.50 due to a nearby symmetry element). Phenyl ligands C7-C12/F2 and C13-C18/F3 are modeled as disordered over *2mm.* positions (0.25:0.25:0.25:0.25). THF ligand O1/C19-C22 is modeled as disordered over a crystallographic mirror plane (0.50:0.50) and additionally over two general positions (0.502(8):0.498(8)). THF ligand O2/C23-C26 is modeled as disordered over a crystallographic mirror plane (0.50:0.50).

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] *CrysAlisPro*, version 171.39.43c; Rigaku Corporation: Oxford, UK, 2018.

[2] Sheldrick, G. M. *SHELXT*, version 2018/2; *Acta. Crystallogr.* **2015**, *A71*, 3-8.

[3] Sheldrick, G. M. *SHELXL*, version 2018/3; *Acta. Crystallogr.* **2015**, *C71*, 3-8.

[4] Spek, A. L. *PLATON*, version 150216; *Acta. Crystallogr.* **2015**, *C71*, 9-18.

Some equations of interest:

$$R_{int} = \Sigma\, |F_o^2 - \langle F_o^2 \rangle| \,/\, \Sigma |F_o^2|$$

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

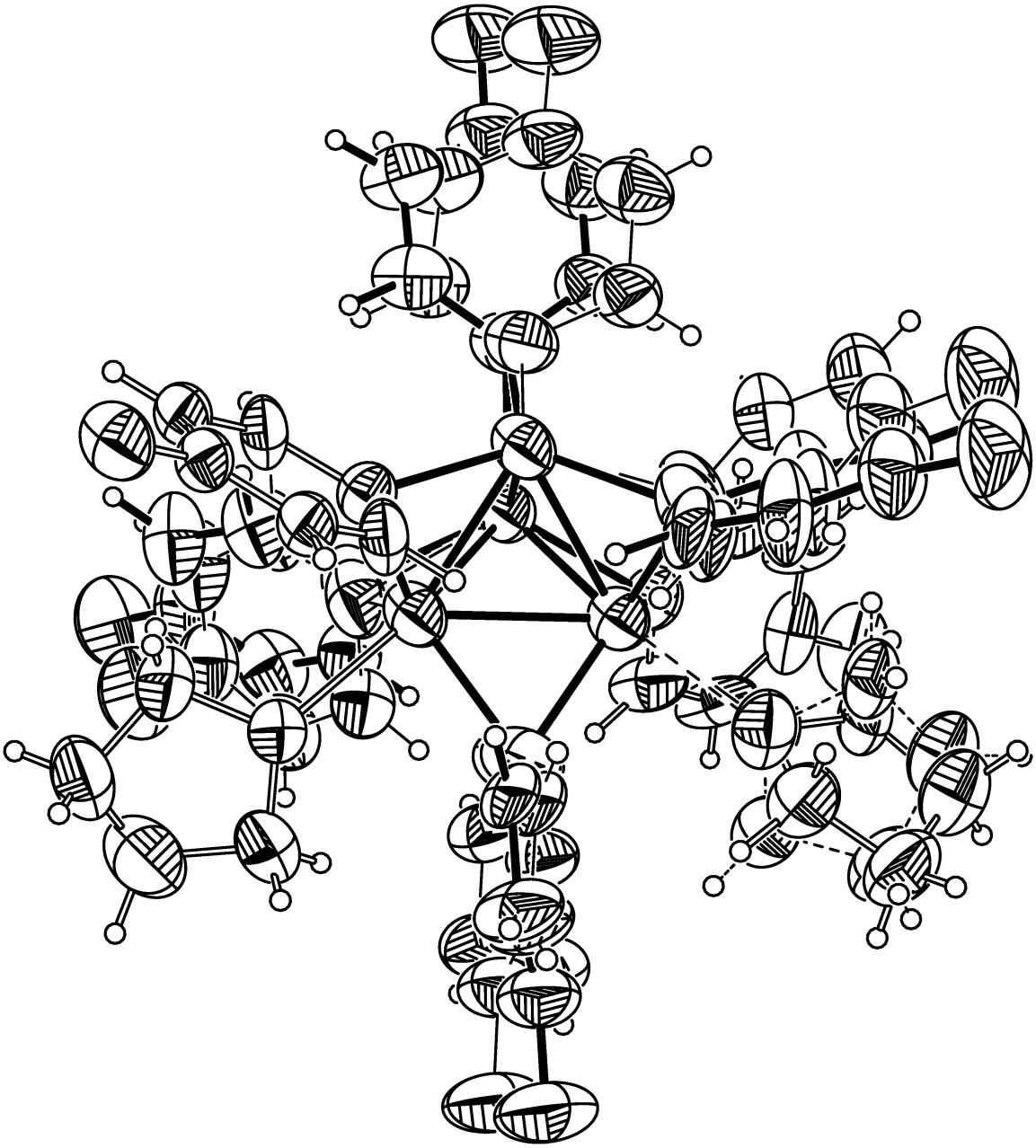
$$wR2 = [\Sigma[w(F_o^2 - F_c^2)^2] \,/\, \Sigma\,[w(F_o^2)^2]]^{1/2}$$

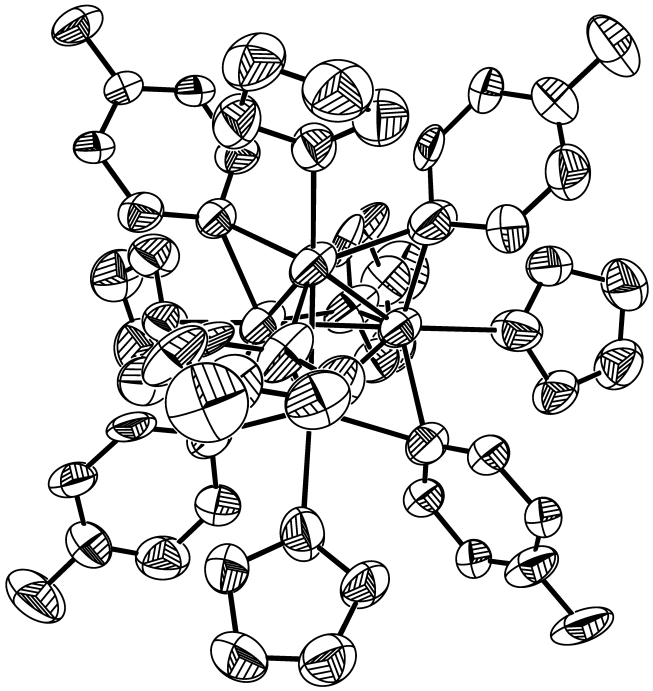
where $w = 1 \,/\, [\sigma^2 (F_o^2) + (aP)^2 + bP]$ and

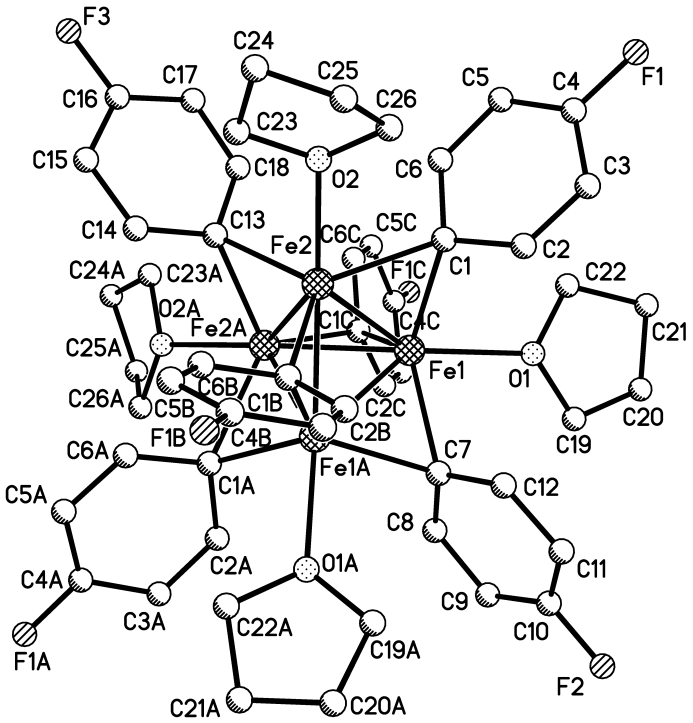
$$P = 1/3 \max (0, F_o^2) + 2/3\, F_c^2$$

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

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



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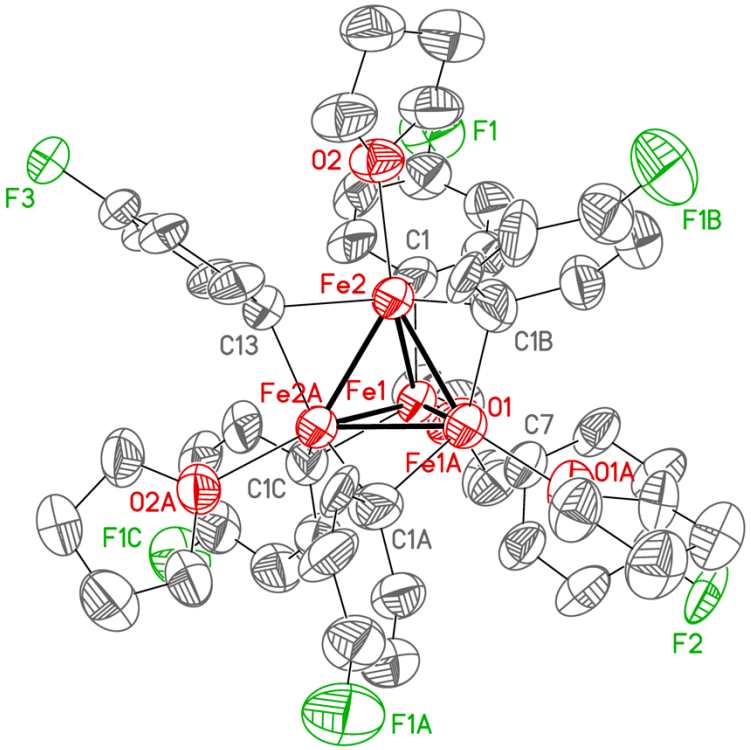
C24
C17
C25
C5
C4
F1
C16
C26
C23
C15
C18
O2
C6
C3
C14
C13
Fe2
C6C
C5C
C2
C24A
C23A
F1C
C1
C22
O2A
Fe2A
C1C
C4C
C21
C25A
Fe1
O1
C6B
C26A
C5B
C1B
C2C
C20
F1B
C4B
C2B
C19
C6A
Fe1A
C7
C1A
C12
C5A
C8
C2A
O1A
C11
C4A
C9
C3A
C10
C22A
C19A
F1A
F2
C21A
C20A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O2
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F1B
C1
Fe2
C13
C1B
Fe2A
Fe1
O1
C7
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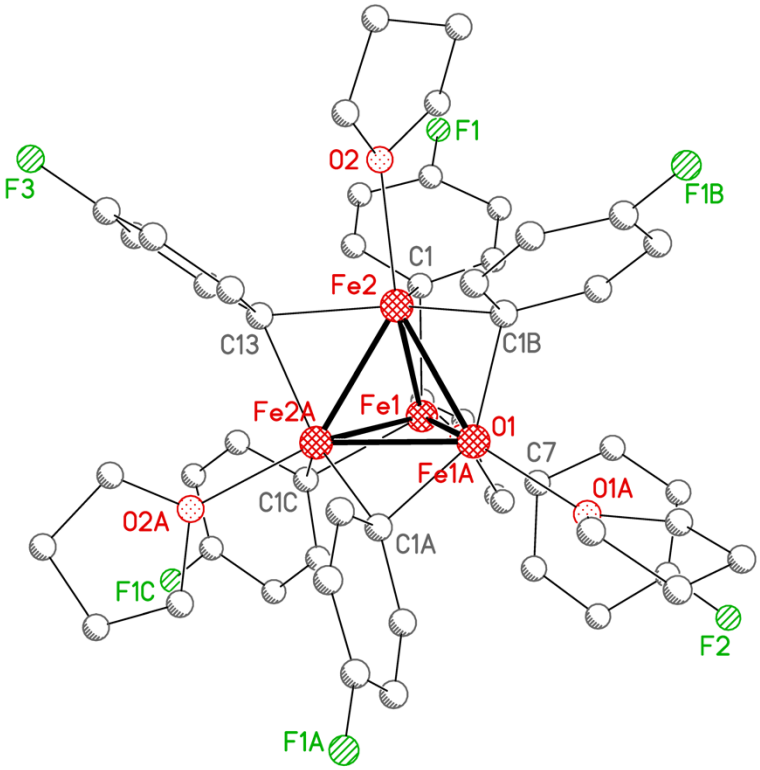
O2A
C1C
F1C
C1A
F2
F1A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C13
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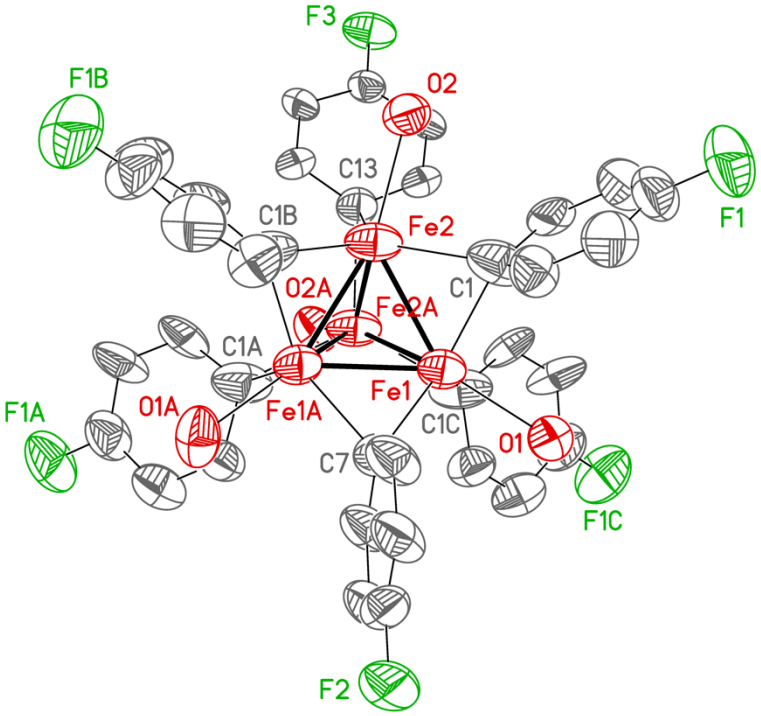
F2
F1A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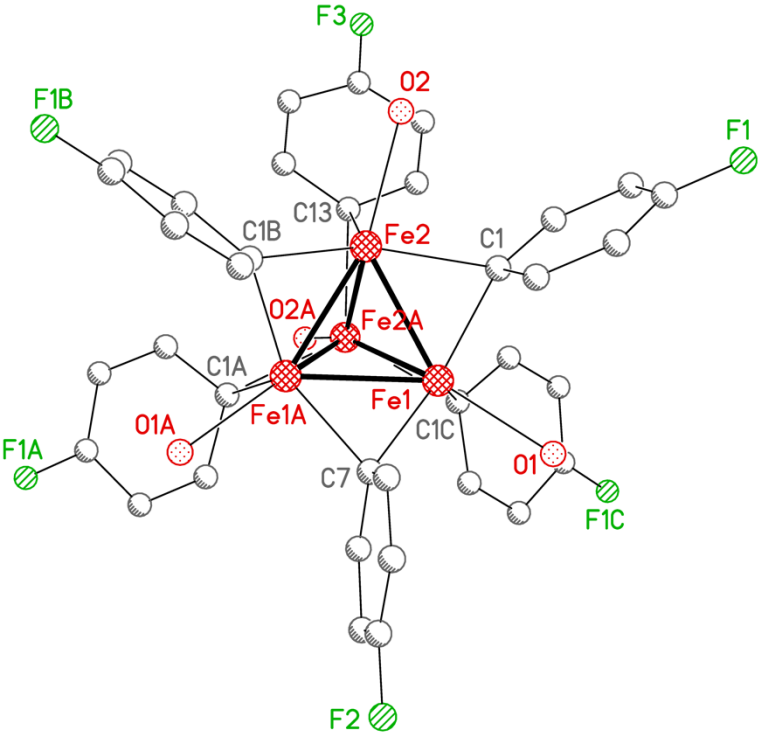
Fe1A
C1C
O1
C7
F1C
F2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C13
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C1
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Fe2A
C1A
Fe1
O1A
Fe1A
C1C
F1A
C7
O1
F1C
F2

Table S21. Crystal data and structure refinement for neisc52.

Identification code	neisc52	
Empirical formula	C52 H56 F6 Fe4 O4	
Formula weight	1082.36	
Temperature	100.0(1) K	
Wavelength	0.71073 Å	
Crystal system	tetragonal	
Space group	$I4_1/amd$	
Unit cell dimensions	a = 15.5897(4) Å	α = 90°
	b = 15.5897(4) Å	β = 90°
	c = 45.3118(12) Å	γ = 90°
Volume	11012.5(6) Å^3	
Z	8	
Density (calculated)	1.306 Mg/m^3	
Absorption coefficient	1.092 mm^{-1}	
F(000)	4464	
Crystal color, morphology	brown, block	
Crystal size	0.164 x 0.152 x 0.124 mm^3	
Theta range for data collection	3.218 to 26.372°	
Index ranges	$-19 \le h \le 18$, $-17 \le k \le 19$, $-56 \le l \le 56$	
Reflections collected	39711	
Independent reflections	3041 [R(int) = 0.0360]	
Observed reflections	2358	
Completeness to theta = 26.372°	99.7%	
Absorption correction	Multi-scan	
Max. and min. transmission	1.0000 and 0.8300	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3041 / 204 / 295	
Goodness-of-fit on F^2	1.049	
Final R indices [I>2sigma(I)]	$R1$ = 0.0626, $wR2$ = 0.1906	
R indices (all data)	$R1$ = 0.0755, $wR2$ = 0.2041	
Largest diff. peak and hole	0.632 and -0.464 e.Å^{-3}	

Table S22. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for neisc52. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Fe1	4199(1)	7500	1671(1)	58(1)
Fe2	5000	8289(1)	2056(1)	65(1)
C1	3724(6)	8680(5)	1871(2)	84(2)
C2	3535(5)	9343(5)	1654(2)	69(2)
C3	2856(5)	9924(5)	1653(2)	86(2)
C4	2265(5)	9881(5)	1885(2)	83(2)
C5	2377(8)	9281(7)	2102(2)	88(2)
C6	3073(8)	8716(8)	2095(2)	69(2)
F1	1612(4)	10455(5)	1883(2)	119(2)
C1'	3816(6)	8756(5)	1856(2)	84(2)
C2'	4074(5)	9510(5)	1695(2)	69(2)
C3'	3496(6)	10196(5)	1693(2)	86(2)
C4'	2737(5)	10117(5)	1842(2)	83(2)
C5'	2473(8)	9406(7)	1996(2)	88(2)
C6'	3031(8)	8700(8)	2007(2)	69(2)
F1'	2182(4)	10788(4)	1834(2)	119(2)
C7	5000	7500	1272(2)	62(2)
C8	5036(12)	6725(9)	1123(3)	76(3)
C9	5049(14)	6635(14)	815(3)	85(4)
C10	5024(11)	7375(13)	655(2)	72(6)
C11	4989(16)	8174(14)	777(3)	85(4)
C12	4977(13)	8210(9)	1086(3)	76(3)
F2	5036(15)	7316(14)	357(1)	95(6)
C13	5000	7500	2457(1)	52(1)
C14	5693(7)	7547(11)	2655(2)	59(2)
C15	5595(8)	7603(12)	2958(2)	51(2)
C16	4781(5)	7613(7)	3072(2)	47(3)
C17	4073(7)	7570(14)	2895(2)	51(2)
C18	4209(7)	7516(12)	2597(3)	59(2)
F3	4697(5)	7668(8)	3372(1)	71(4)
O1	3092(7)	7570(20)	1349(3)	79(4)

C19	3012(9)	7185(11)	1061(3)	73(3)
C20	2094(12)	7220(19)	963(5)	92(5)
C21	1650(9)	7778(14)	1184(3)	95(5)
C22	2216(8)	7712(13)	1447(3)	78(4)
O1'	2978(8)	7544(17)	1416(3)	79(4)
C19'	2643(15)	6754(15)	1305(4)	73(3)
C20'	2300(20)	7027(18)	1006(4)	92(5)
C21'	2006(13)	7930(16)	1078(5)	95(5)
C22'	2791(13)	8279(15)	1238(5)	78(4)
O2	4748(3)	9457(3)	2347(1)	66(2)
C23	5332(6)	9588(5)	2591(2)	85(3)
C24	5318(7)	10517(5)	2666(2)	96(3)
C25	5113(16)	10936(5)	2384(2)	111(3)
C26	4462(7)	10284(5)	2243(2)	93(3)

Table S23. Bond lengths [Å] and angles [°] for neisc52.

Fe(1)-C(1)	2.179(6)	C(4')-F(1')	1.358(8)
Fe(1)-C(1)#1	2.180(6)	C(4')-C(5')	1.373(11)
Fe(1)-C(7)	2.197(6)	C(5')-C(6')	1.403(11)
Fe(1)-C(1')#1	2.213(6)	C(5')-H(5')	0.9500
Fe(1)-C(1')	2.213(6)	C(6')-H(6')	0.9500
Fe(1)-O(1')	2.228(8)	C(7)-C(8)	1.385(13)
Fe(1)-O(1)	2.262(8)	C(7)-C(12)	1.390(12)
Fe(1)-Fe(2)	2.4740(9)	C(8)-C(9)	1.404(14)
Fe(1)-Fe(2)#2	2.4740(9)	C(8)-H(8)	0.9500
Fe(1)-Fe(1)#2	2.4965(15)	C(9)-C(10)	1.363(14)
Fe(2)-C(1')	2.181(6)	C(9)-H(9)	0.9500
Fe(2)-C(1')#3	2.181(6)	C(10)-F(2)	1.355(8)
Fe(2)-C(13)	2.194(5)	C(10)-C(11)	1.362(15)
Fe(2)-C(1)#3	2.244(7)	C(11)-C(12)	1.405(13)
Fe(2)-C(1)	2.244(7)	C(11)-H(11)	0.9500
Fe(2)-O(2)	2.282(5)	C(12)-H(12)	0.9500
Fe(2)-Fe(2)#2	2.4602(14)	C(13)-C(18)	1.387(11)
C(1)-C(6)	1.438(11)	C(13)-C(14)	1.406(10)
C(1)-C(2)	1.456(10)	C(14)-C(15)	1.384(12)
C(2)-C(3)	1.392(10)	C(14)-H(14)	0.9500
C(2)-H(2)	0.9500	C(15)-C(16)	1.370(12)
C(3)-C(4)	1.401(10)	C(15)-H(15)	0.9500
C(3)-H(3)	0.9500	C(16)-C(17)	1.364(11)
C(4)-F(1)	1.355(8)	C(16)-F(3)	1.367(8)
C(4)-C(5)	1.370(10)	C(17)-C(18)	1.370(12)
C(5)-C(6)	1.397(11)	C(17)-H(17)	0.9500
C(5)-H(5)	0.9500	C(18)-H(18)	0.9500
C(6)-H(6)	0.9500	O(1)-C(19)	1.443(8)
C(1')-C(6')	1.406(11)	O(1)-C(22)	1.453(8)
C(1')-C(2')	1.442(11)	C(19)-C(20)	1.500(12)
C(2')-C(3')	1.398(10)	C(19)-H(19A)	0.9900
C(2')-H(2')	0.9500	C(19)-H(19B)	0.9900
C(3')-C(4')	1.369(11)	C(20)-C(21)	1.497(14)
C(3')-H(3')	0.9500	C(20)-H(20A)	0.9900

C(20)-H(20B)	0.9900	C(1)#1-Fe(1)-C(1')#1	5.1(6)
C(21)-C(22)	1.485(12)	C(7)-Fe(1)-C(1')#1	117.8(3)
C(21)-H(21A)	0.9900	C(7)-Fe(1)-C(1')	117.8(3)
C(21)-H(21B)	0.9900	C(1')#1-Fe(1)-C(1')	124.5(6)
C(22)-H(22A)	0.9900	C(7)-Fe(1)-O(1')	93.3(5)
C(22)-H(22B)	0.9900	C(1')#1-Fe(1)-O(1')	89.6(7)
O(1')-C(22')	1.428(8)	C(1')-Fe(1)-O(1')	86.5(6)
O(1')-C(19')	1.430(8)	C(1)-Fe(1)-O(1)	88.2(7)
C(19')-C(20')	1.517(13)	C(1)#1-Fe(1)-O(1)	92.8(10)
C(19')-H(19C)	0.9900	C(7)-Fe(1)-O(1)	84.5(5)
C(19')-H(19D)	0.9900	C(1)-Fe(1)-Fe(2)	57.23(18)
C(20')-C(21')	1.517(14)	C(1)#1-Fe(1)-Fe(2)	107.3(2)
C(20')-H(20C)	0.9900	C(7)-Fe(1)-Fe(2)	107.11(9)
C(20')-H(20D)	0.9900	C(1')#1-Fe(1)-Fe(2)	107.96(19)
C(21')-C(22')	1.523(13)	C(1')-Fe(1)-Fe(2)	55.14(16)
C(21')-H(21C)	0.9900	O(1')-Fe(1)-Fe(2)	141.4(6)
C(21')-H(21D)	0.9900	O(1)-Fe(1)-Fe(2)	144.7(8)
C(22')-H(22C)	0.9900	C(1)-Fe(1)-Fe(2)#2	107.3(2)
C(22')-H(22D)	0.9900	C(1)#1-Fe(1)-Fe(2)#2	57.23(18)
O(2)-C(26)	1.445(9)	C(7)-Fe(1)-Fe(2)#2	107.11(9)
O(2)-C(23)	1.446(7)	O(1)-Fe(1)-Fe(2)#2	149.7(10)
C(23)-C(24)	1.488(11)	Fe(2)-Fe(1)-Fe(2)#2	59.63(3)
C(23)-H(23A)	0.9900	C(1)-Fe(1)-Fe(1)#2	109.9(2)
C(23)-H(23B)	0.9900	C(1)#1-Fe(1)-Fe(1)#2	109.9(2)
C(24)-C(25)	1.472(12)	C(7)-Fe(1)-Fe(1)#2	55.39(10)
C(24)-H(24A)	0.9900	O(1)-Fe(1)-Fe(1)#2	139.8(5)
C(24)-H(24B)	0.9900	Fe(2)-Fe(1)-Fe(1)#2	59.695(18)
C(25)-C(26)	1.572(16)	Fe(2)#2-Fe(1)-Fe(1)#2	59.696(18)
C(25)-H(25A)	0.9900	C(1')-Fe(2)-C(1')#3	115.6(6)
C(25)-H(25B)	0.9900	C(1')-Fe(2)-C(13)	122.1(3)
C(26)-H(26A)	0.9900	C(1')#3-Fe(2)-C(13)	122.1(3)
C(26)-H(26B)	0.9900	C(13)-Fe(2)-C(1)#3	117.5(3)
C(1)-Fe(1)-C(1)#1	115.1(6)	C(13)-Fe(2)-C(1)	117.5(3)
C(1)-Fe(1)-C(7)	122.3(3)	C(1)#3-Fe(2)-C(1)	124.9(6)
C(1)#1-Fe(1)-C(7)	122.3(3)	C(13)-Fe(2)-O(2)	88.19(15)
C(1)-Fe(1)-C(1')#1	119.8(2)	C(1)#3-Fe(2)-O(2)	98.8(2)

C(1)-Fe(2)-O(2)	81.23(19)	C(5)-C(6)-C(1)	126.2(9)
C(13)-Fe(2)-Fe(2)#2	55.89(9)	C(5)-C(6)-H(6)	116.9
C(1)#3-Fe(2)-Fe(2)#2	105.7(2)	C(1)-C(6)-H(6)	116.9
C(1)-Fe(2)-Fe(2)#2	105.7(2)	C(6')-C(1')-C(2')	122.7(7)
O(2)-Fe(2)-Fe(2)#2	142.89(12)	C(6')-C(1')-Fe(2)	120.9(7)
C(13)-Fe(2)-Fe(1)#2	107.79(8)	C(2')-C(1')-Fe(2)	104.3(6)
C(1)#3-Fe(2)-Fe(1)#2	54.77(15)	C(6')-C(1')-Fe(1)	111.4(7)
C(1)-Fe(2)-Fe(1)#2	108.5(2)	C(2')-C(1')-Fe(1)	117.0(6)
O(2)-Fe(2)-Fe(1)#2	153.07(13)	Fe(2)-C(1')-Fe(1)	68.53(19)
Fe(2)#2-Fe(2)-Fe(1)#2	60.184(17)	C(3')-C(2')-C(1')	116.6(7)
C(1')-Fe(2)-Fe(1)	56.34(16)	C(3')-C(2')-H(2')	121.7
C(1')#3-Fe(2)-Fe(1)	107.4(2)	C(1')-C(2')-H(2')	121.7
C(13)-Fe(2)-Fe(1)	107.79(8)	C(4')-C(3')-C(2')	119.0(8)
C(1)#3-Fe(2)-Fe(1)	108.5(2)	C(4')-C(3')-H(3')	120.5
C(1)-Fe(2)-Fe(1)	54.77(15)	C(2')-C(3')-H(3')	120.5
O(2)-Fe(2)-Fe(1)	135.86(11)	F(1')-C(4')-C(3')	117.9(8)
Fe(2)#2-Fe(2)-Fe(1)	60.185(17)	F(1')-C(4')-C(5')	116.4(9)
Fe(1)#2-Fe(2)-Fe(1)	60.61(3)	C(3')-C(4')-C(5')	125.7(8)
C(6)-C(1)-C(2)	107.7(7)	C(4')-C(5')-C(6')	117.7(10)
C(6)-C(1)-Fe(1)	124.5(7)	C(4')-C(5')-H(5')	121.1
C(2)-C(1)-Fe(1)	112.9(5)	C(6')-C(5')-H(5')	121.1
C(6)-C(1)-Fe(2)	111.9(6)	C(5')-C(6')-C(1')	118.3(10)
C(2)-C(1)-Fe(2)	128.7(7)	C(5')-C(6')-H(6')	120.9
Fe(1)-C(1)-Fe(2)	68.00(19)	C(1')-C(6')-H(6')	120.9
C(3)-C(2)-C(1)	128.3(8)	C(8)-C(7)-C(12)	113.7(8)
C(3)-C(2)-H(2)	115.9	C(8)-C(7)-Fe(1)	115.1(7)
C(1)-C(2)-H(2)	115.9	C(12)-C(7)-Fe(1)	118.8(7)
C(2)-C(3)-C(4)	117.7(7)	C(8)-C(7)-Fe(1)#2	112.2(7)
C(2)-C(3)-H(3)	121.1	C(12)-C(7)-Fe(1)#2	120.8(8)
C(4)-C(3)-H(3)	121.1	Fe(1)-C(7)-Fe(1)#2	69.2(2)
F(1)-C(4)-C(5)	123.5(9)	C(7)-C(8)-C(9)	124.9(15)
F(1)-C(4)-C(3)	117.2(8)	C(7)-C(8)-H(8)	117.5
C(5)-C(4)-C(3)	119.3(8)	C(9)-C(8)-H(8)	117.5
C(4)-C(5)-C(6)	120.8(9)	C(10)-C(9)-C(8)	116.3(15)
C(4)-C(5)-H(5)	119.6	C(10)-C(9)-H(9)	121.9
C(6)-C(5)-H(5)	119.6	C(8)-C(9)-H(9)	121.9

F(2)-C(10)-C(11)	117.9(9)	O(1)-C(19)-H(19B)	109.8
F(2)-C(10)-C(9)	118.1(10)	C(20)-C(19)-H(19B)	109.8
C(11)-C(10)-C(9)	124.1(9)	H(19A)-C(19)-H(19B)	108.2
C(10)-C(11)-C(12)	116.2(14)	C(21)-C(20)-C(19)	105.3(10)
C(10)-C(11)-H(11)	121.9	C(21)-C(20)-H(20A)	110.7
C(12)-C(11)-H(11)	121.9	C(19)-C(20)-H(20A)	110.7
C(7)-C(12)-C(11)	124.8(14)	C(21)-C(20)-H(20B)	110.7
C(7)-C(12)-H(12)	117.6	C(19)-C(20)-H(20B)	110.7
C(11)-C(12)-H(12)	117.6	H(20A)-C(20)-H(20B)	108.8
C(18)-C(13)-C(14)	112.9(7)	C(22)-C(21)-C(20)	102.8(11)
C(18)-C(13)-Fe(2)#2	112.9(6)	C(22)-C(21)-H(21A)	111.2
C(14)-C(13)-Fe(2)#2	123.9(6)	C(20)-C(21)-H(21A)	111.2
C(18)-C(13)-Fe(2)	111.7(7)	C(22)-C(21)-H(21B)	111.2
C(14)-C(13)-Fe(2)	119.9(6)	C(20)-C(21)-H(21B)	111.2
Fe(2)#2-C(13)-Fe(2)	68.21(18)	H(21A)-C(21)-H(21B)	109.1
C(15)-C(14)-C(13)	123.5(10)	O(1)-C(22)-C(21)	109.0(9)
C(15)-C(14)-H(14)	118.2	O(1)-C(22)-H(22A)	109.9
C(13)-C(14)-H(14)	118.2	C(21)-C(22)-H(22A)	109.9
C(16)-C(15)-C(14)	118.4(10)	O(1)-C(22)-H(22B)	109.9
C(16)-C(15)-H(15)	120.8	C(21)-C(22)-H(22B)	109.9
C(14)-C(15)-H(15)	120.8	H(22A)-C(22)-H(22B)	108.3
C(17)-C(16)-F(3)	120.5(8)	C(22')-O(1')-C(19')	114.8(9)
C(17)-C(16)-C(15)	121.9(8)	C(22')-O(1')-Fe(1)	119.4(15)
F(3)-C(16)-C(15)	117.6(7)	C(19')-O(1')-Fe(1)	117.9(14)
C(16)-C(17)-C(18)	117.2(10)	O(1')-C(19')-C(20')	101.5(11)
C(16)-C(17)-H(17)	121.4	O(1')-C(19')-H(19C)	111.5
C(18)-C(17)-H(17)	121.4	C(20')-C(19')-H(19C)	111.5
C(17)-C(18)-C(13)	126.1(10)	O(1')-C(19')-H(19D)	111.5
C(17)-C(18)-H(18)	116.9	C(20')-C(19')-H(19D)	111.5
C(13)-C(18)-H(18)	116.9	H(19C)-C(19')-H(19D)	109.3
C(19)-O(1)-C(22)	105.0(10)	C(21')-C(20')-C(19')	100.1(13)
C(19)-O(1)-Fe(1)	129.0(11)	C(21')-C(20')-H(20C)	111.8
C(22)-O(1)-Fe(1)	121.9(9)	C(19')-C(20')-H(20C)	111.8
O(1)-C(19)-C(20)	109.5(10)	C(21')-C(20')-H(20D)	111.7
O(1)-C(19)-H(19A)	109.8	C(19')-C(20')-H(20D)	111.7
C(20)-C(19)-H(19A)	109.8	H(20C)-C(20')-H(20D)	109.5

C(20')-C(21')-C(22')	100.9(13)	H(23A)-C(23)-H(23B)	108.5
C(20')-C(21')-H(21C)	111.6	C(25)-C(24)-C(23)	103.6(6)
C(22')-C(21')-H(21C)	111.6	C(25)-C(24)-H(24A)	111.0
C(20')-C(21')-H(21D)	111.6	C(23)-C(24)-H(24A)	111.0
C(22')-C(21')-H(21D)	111.6	C(25)-C(24)-H(24B)	111.0
H(21C)-C(21')-H(21D)	109.4	C(23)-C(24)-H(24B)	111.0
O(1')-C(22')-C(21')	98.4(10)	H(24A)-C(24)-H(24B)	109.0
O(1')-C(22')-H(22C)	112.1	C(24)-C(25)-C(26)	102.0(8)
C(21')-C(22')-H(22C)	112.1	C(24)-C(25)-H(25A)	111.4
O(1')-C(22')-H(22D)	112.1	C(26)-C(25)-H(25A)	111.4
C(21')-C(22')-H(22D)	112.1	C(24)-C(25)-H(25B)	111.4
H(22C)-C(22')-H(22D)	109.7	C(26)-C(25)-H(25B)	111.4
C(26)-O(2)-C(23)	108.5(6)	H(25A)-C(25)-H(25B)	109.2
C(26)-O(2)-Fe(2)	125.1(4)	O(2)-C(26)-C(25)	104.2(7)
C(23)-O(2)-Fe(2)	116.5(4)	O(2)-C(26)-H(26A)	110.9
O(2)-C(23)-C(24)	107.7(7)	C(25)-C(26)-H(26A)	110.9
O(2)-C(23)-H(23A)	110.2	O(2)-C(26)-H(26B)	110.9
C(24)-C(23)-H(23A)	110.2	C(25)-C(26)-H(26B)	110.9
O(2)-C(23)-H(23B)	110.2	H(26A)-C(26)-H(26B)	108.9
C(24)-C(23)-H(23B)	110.2		

Symmetry transformations used to generate equivalent atoms:

#1 x,-y+3/2,z #2 -x+1,-y+3/2,z+0 #3 -x+1,y,z

3.8 [*trans*-Fe(acac)$_2$(THF)$_2$]$_{0.58}$ · [*trans*-Mg(acac)$_2$(THF)$_2$]$_{0.42}$ (3)

REFERENCE NUMBER: neisc31

CRYSTAL STRUCTURE REPORT

C_{18} H_{30} $Fe_{0.58}$ $Mg_{0.42}$ O_6

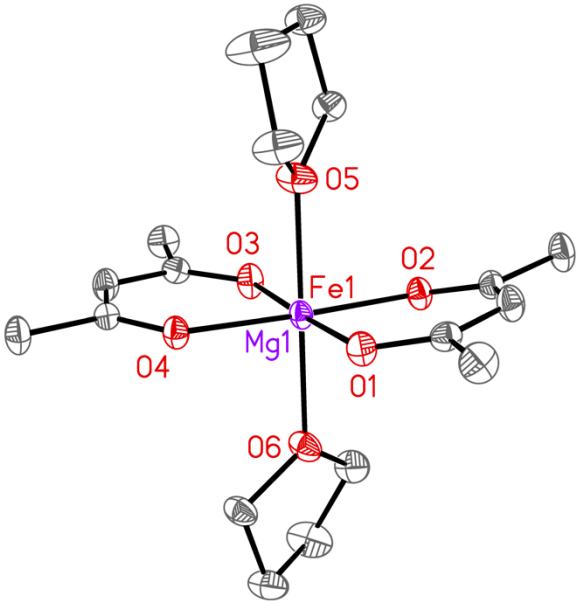
or

[*trans*-Fe(acac)$_2$(THF)$_2$]$_{0.58}$ · [*trans*-Mg(acac)$_2$(THF)$_2$]$_{0.42}$

Report prepared for:

S. Carpenter, Prof. M. Neidig

July 05, 2017



William W. Brennessel

X-ray Crystallographic Facility

Department of Chemistry, University of Rochester

120 Trustee Road

Rochester, NY 14627

Data collection

A crystal (0.40 x 0.40 x 0.34 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: six major sections of frames were collected with 0.50º steps in ω at six 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 4093 strong reflections from the actual data collection after integration.[3] See Table 1 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 $P2_1/c$ was determined based on systematic absences. 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 $R1 = 0.0405$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1096$ (F^2, all data).

Structure description

The structure is the one suggested. The asymmetric unit contains one complex in a general position. The metal site is modeled as a disorder of Fe and Mg (0.58:0.42). Both THF ligands are modeled as disordered over two positions (0.74:0.26 and 0.72:0.28 for THF molecules O5/C11-C14 and O6/C15-C18, respectively).

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 2017/1; *Acta. Cryst.* **2015**, C*71*, 3-8.

Some equations of interest:

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

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

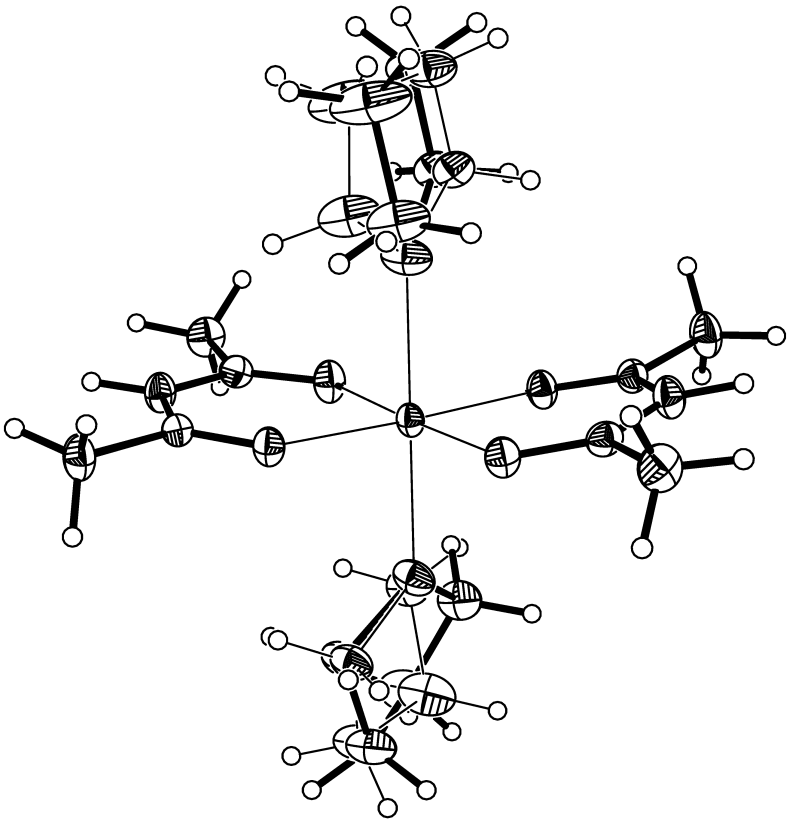
$$wR2 = [\Sigma [w(F_o^2 - F_c^2)^2] \,/\, \Sigma\, [w(F_o^2)^2]]^{1/2}$$

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



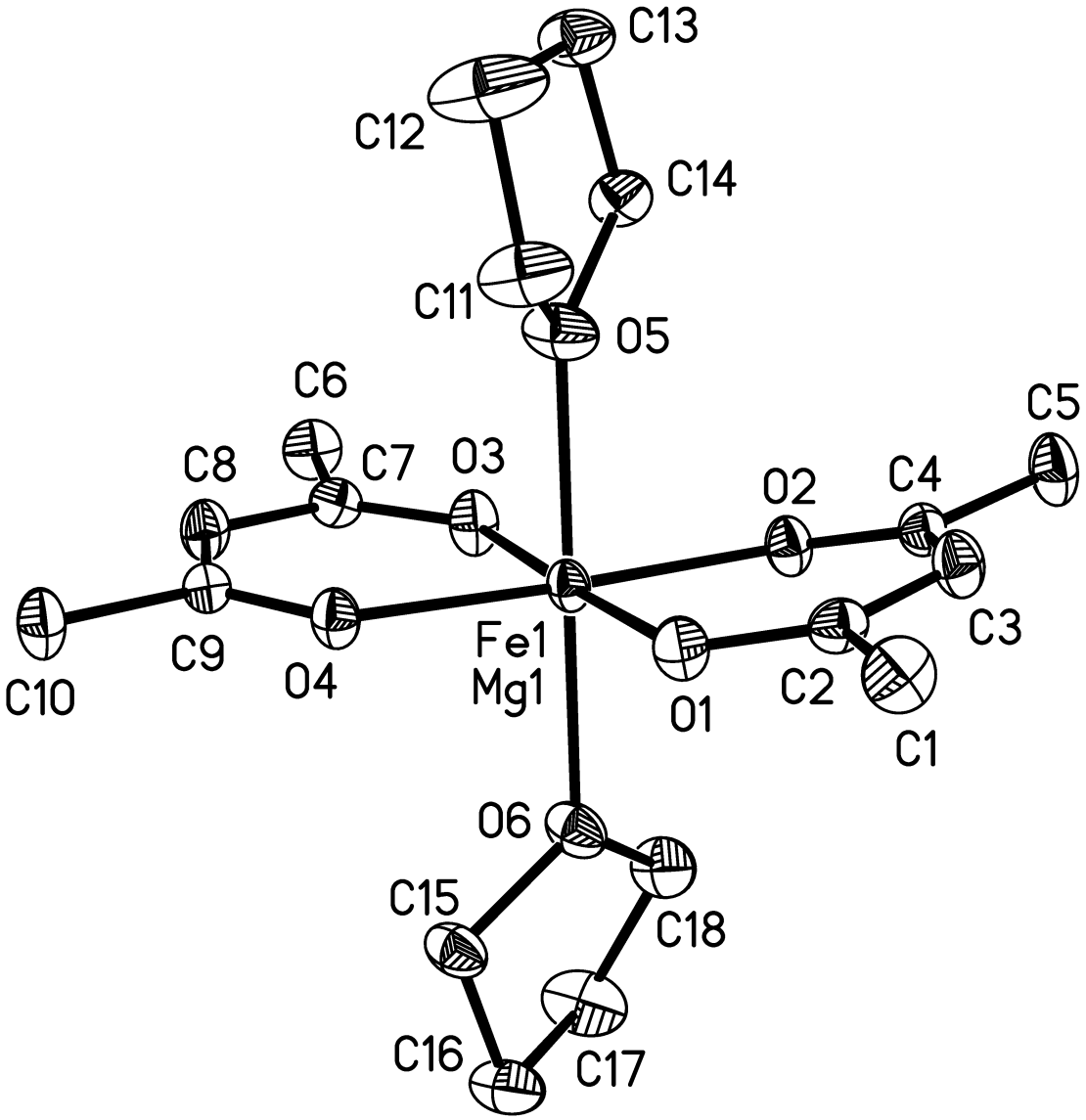

C13
C12
C14
C11
O5
C6
C5
C8
C7
O3
O2
C4
C3
C9
O4
Fe1
Mg1
O1
C2
C10
C1
O6
C15
C18
C16
C17

Table S24. Crystal data and structure refinement for neisc31.

Identification code	neisc31	
Empirical formula	C18 H30 Fe0.58 Mg0.42 O6	
Formula weight	384.94	
Temperature	100.0(5) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 15.2106(8) Å	α = 90°
	b = 11.1429(6) Å	β = 114.0312(10)°
	c = 12.9636(7) Å	γ = 90°
Volume	2006.76(19) Å^3	
Z	4	
Density (calculated)	1.274 Mg/m^3	
Absorption coefficient	0.499 mm^{-1}	
F(000)	824	
Crystal color, morphology	yellow-brown, block	
Crystal size	0.40 x 0.40 x 0.34 mm^3	
Theta range for data collection	2.343 to 38.677°	
Index ranges	$-26 \leq h \leq 26$, $-19 \leq k \leq 19$, $-22 \leq l \leq 22$	
Reflections collected	66978	
Independent reflections	11048 [R(int) = 0.0429]	
Observed reflections	8035	
Completeness to theta = 37.785°	99.1%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.7476 and 0.6462	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	11048 / 52 / 257	
Goodness-of-fit on F^2	1.015	
Final R indices [I>2sigma(I)]	$R1$ = 0.0405, $wR2$ = 0.0981	
R indices (all data)	$R1$ = 0.0667, $wR2$ = 0.1096	
Largest diff. peak and hole	0.528 and -0.470 e.Å^{-3}	

Table S25. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for neisc31. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Fe1	2614(1)	2632(1)	2680(1)	16(1)
Mg1	2614(1)	2632(1)	2680(1)	16(1)
O1	1954(1)	3756(1)	3378(1)	22(1)
O2	3202(1)	4106(1)	2271(1)	21(1)
C1	1266(1)	5481(1)	3802(1)	28(1)
C2	1879(1)	4885(1)	3280(1)	20(1)
C3	2322(1)	5608(1)	2738(1)	24(1)
C4	2948(1)	5185(1)	2269(1)	19(1)
C5	3363(1)	6071(1)	1702(1)	29(1)
O3	3263(1)	1553(1)	1933(1)	24(1)
O4	2012(1)	1136(1)	3024(1)	20(1)
C6	3674(1)	-96(1)	1082(1)	26(1)
C7	3146(1)	440(1)	1740(1)	19(1)
C8	2582(1)	-311(1)	2099(1)	22(1)
C9	2067(1)	68(1)	2727(1)	18(1)
C10	1533(1)	-865(1)	3099(1)	26(1)
O5	1361(1)	2806(1)	1089(1)	28(1)
C11	398(1)	2879(3)	1021(2)	34(1)
C12	-251(3)	2691(6)	-169(3)	54(1)
C13	334(3)	3041(3)	-852(3)	29(1)
C14	1373(2)	3131(2)	13(2)	23(1)
O6	3854(1)	2462(1)	4279(1)	25(1)
C15	3939(4)	1608(5)	5151(5)	25(1)
C16	4943(5)	1762(7)	6054(5)	32(1)
C17	5519(3)	2095(4)	5361(3)	39(1)
C18	4794(1)	2864(2)	4419(1)	29(1)
O5'	1361(1)	2806(1)	1089(1)	28(1)
C11'	427(4)	2335(7)	894(5)	34(1)
C12'	-164(9)	2436(19)	-251(10)	54(1)
C13'	295(9)	3272(11)	-788(10)	29(1)
C14'	1301(7)	3368(8)	70(7)	23(1)

O6'	3854(1)	2462(1)	4279(1)	25(1)
C15'	3874(11)	1725(15)	5208(13)	25(1)
C16'	4928(12)	1610(20)	5996(13)	32(1)
C17'	5450(8)	2353(13)	5425(9)	39(1)
C18'	4805(3)	2236(4)	4201(3)	29(1)

Table S26. Bond lengths [Å] and angles [°] for neisc31.

Fe(1)-O(1)	2.0332(7)	C(9)-C(10)	1.5140(12)
Fe(1)-O(3)	2.0338(7)	C(10)-H(10A)	0.9800
Fe(1)-O(4)	2.0377(6)	C(10)-H(10B)	0.9800
Fe(1)-O(2)	2.0408(7)	C(10)-H(10C)	0.9800
Fe(1)-O(6)	2.1696(8)	O(5)-C(11)	1.4327(17)
Fe(1)-O(5)	2.1725(8)	O(5)-C(14)	1.448(3)
Mg(1)-O(1)	2.0332(7)	C(11)-C(12)	1.469(4)
Mg(1)-O(3)	2.0338(7)	C(11)-H(11A)	0.9900
Mg(1)-O(4)	2.0377(6)	C(11)-H(11B)	0.9900
Mg(1)-O(2)	2.0408(7)	C(12)-C(13)	1.540(4)
Mg(1)-O(6')	2.1696(8)	C(12)-H(12A)	0.9900
Mg(1)-O(5')	2.1725(8)	C(12)-H(12B)	0.9900
O(1)-C(2)	1.2651(11)	C(13)-C(14)	1.523(4)
O(2)-C(4)	1.2622(10)	C(13)-H(13A)	0.9900
C(1)-C(2)	1.5113(12)	C(13)-H(13B)	0.9900
C(1)-H(1A)	0.9800	C(14)-H(14A)	0.9900
C(1)-H(1B)	0.9800	C(14)-H(14B)	0.9900
C(1)-H(1C)	0.9800	O(6)-C(18)	1.4374(15)
C(2)-C(3)	1.4071(13)	O(6)-C(15)	1.443(4)
C(3)-C(4)	1.4045(12)	C(15)-C(16)	1.508(5)
C(3)-H(3)	0.9500	C(15)-H(15A)	0.9900
C(4)-C(5)	1.5132(13)	C(15)-H(15B)	0.9900
C(5)-H(5A)	0.9800	C(16)-C(17)	1.534(5)
C(5)-H(5B)	0.9800	C(16)-H(16A)	0.9900
C(5)-H(5C)	0.9800	C(16)-H(16B)	0.9900
O(3)-C(7)	1.2639(11)	C(17)-C(18)	1.532(4)
O(4)-C(9)	1.2642(10)	C(17)-H(17A)	0.9900
C(6)-C(7)	1.5119(12)	C(17)-H(17B)	0.9900
C(6)-H(6A)	0.9800	C(18)-H(18A)	0.9900
C(6)-H(6B)	0.9800	C(18)-H(18B)	0.9900
C(6)-H(6C)	0.9800	O(5')-C(14')	1.431(9)
C(7)-C(8)	1.4074(13)	O(5')-C(11')	1.437(5)
C(8)-C(9)	1.4042(12)	C(11')-C(12')	1.391(12)
C(8)-H(8)	0.9500	C(11')-H(11C)	0.9900

C(11')-H(11D)	0.9900	O(6)-Fe(1)-O(5)	179.31(3)
C(12')-C(13')	1.496(14)	O(1)-Mg(1)-O(3)	177.83(3)
C(12')-H(12C)	0.9900	O(1)-Mg(1)-O(4)	93.36(3)
C(12')-H(12D)	0.9900	O(3)-Mg(1)-O(4)	88.27(3)
C(13')-C(14')	1.484(12)	O(1)-Mg(1)-O(2)	88.16(3)
C(13')-H(13C)	0.9900	O(3)-Mg(1)-O(2)	90.18(3)
C(13')-H(13D)	0.9900	O(4)-Mg(1)-O(2)	177.75(3)
C(14')-H(14C)	0.9900	O(1)-Mg(1)-O(6')	91.33(3)
C(14')-H(14D)	0.9900	O(3)-Mg(1)-O(6')	90.09(3)
O(6')-C(15')	1.447(12)	O(4)-Mg(1)-O(6')	90.71(3)
O(6')-C(18')	1.513(4)	O(2)-Mg(1)-O(6')	90.93(3)
C(15')-C(16')	1.515(13)	O(1)-Mg(1)-O(5')	88.07(3)
C(15')-H(15C)	0.9900	O(3)-Mg(1)-O(5')	90.51(3)
C(15')-H(15D)	0.9900	O(4)-Mg(1)-O(5')	88.98(3)
C(16')-C(17')	1.529(13)	O(2)-Mg(1)-O(5')	89.40(3)
C(16')-H(16C)	0.9900	O(6')-Mg(1)-O(5')	179.31(3)
C(16')-H(16D)	0.9900	C(2)-O(1)-Fe(1)	127.48(6)
C(17')-C(18')	1.493(11)	C(2)-O(1)-Mg(1)	127.48(6)
C(17')-H(17C)	0.9900	C(4)-O(2)-Fe(1)	126.98(6)
C(17')-H(17D)	0.9900	C(4)-O(2)-Mg(1)	126.98(6)
C(18')-H(18C)	0.9900	C(2)-C(1)-H(1A)	109.5
C(18')-H(18D)	0.9900	C(2)-C(1)-H(1B)	109.5
O(1)-Fe(1)-O(3)	177.83(3)	H(1A)-C(1)-H(1B)	109.5
O(1)-Fe(1)-O(4)	93.36(3)	C(2)-C(1)-H(1C)	109.5
O(3)-Fe(1)-O(4)	88.27(3)	H(1A)-C(1)-H(1C)	109.5
O(1)-Fe(1)-O(2)	88.16(3)	H(1B)-C(1)-H(1C)	109.5
O(3)-Fe(1)-O(2)	90.18(3)	O(1)-C(2)-C(3)	125.27(8)
O(4)-Fe(1)-O(2)	177.75(3)	O(1)-C(2)-C(1)	116.03(8)
O(1)-Fe(1)-O(6)	91.33(3)	C(3)-C(2)-C(1)	118.70(8)
O(3)-Fe(1)-O(6)	90.09(3)	C(4)-C(3)-C(2)	124.85(8)
O(4)-Fe(1)-O(6)	90.71(3)	C(4)-C(3)-H(3)	117.6
O(2)-Fe(1)-O(6)	90.93(3)	C(2)-C(3)-H(3)	117.6
O(1)-Fe(1)-O(5)	88.07(3)	O(2)-C(4)-C(3)	125.36(8)
O(3)-Fe(1)-O(5)	90.51(3)	O(2)-C(4)-C(5)	115.77(8)
O(4)-Fe(1)-O(5)	88.98(3)	C(3)-C(4)-C(5)	118.87(8)
O(2)-Fe(1)-O(5)	89.40(3)	C(4)-C(5)-H(5A)	109.5

C(4)-C(5)-H(5B)	109.5	O(5)-C(11)-H(11B)	110.3
H(5A)-C(5)-H(5B)	109.5	C(12)-C(11)-H(11B)	110.3
C(4)-C(5)-H(5C)	109.5	H(11A)-C(11)-H(11B)	108.6
H(5A)-C(5)-H(5C)	109.5	C(11)-C(12)-C(13)	105.6(3)
H(5B)-C(5)-H(5C)	109.5	C(11)-C(12)-H(12A)	110.6
C(7)-O(3)-Fe(1)	128.17(6)	C(13)-C(12)-H(12A)	110.6
C(7)-O(3)-Mg(1)	128.17(6)	C(11)-C(12)-H(12B)	110.6
C(9)-O(4)-Fe(1)	127.69(6)	C(13)-C(12)-H(12B)	110.6
C(9)-O(4)-Mg(1)	127.69(6)	H(12A)-C(12)-H(12B)	108.8
C(7)-C(6)-H(6A)	109.5	C(14)-C(13)-C(12)	105.2(3)
C(7)-C(6)-H(6B)	109.5	C(14)-C(13)-H(13A)	110.7
H(6A)-C(6)-H(6B)	109.5	C(12)-C(13)-H(13A)	110.7
C(7)-C(6)-H(6C)	109.5	C(14)-C(13)-H(13B)	110.7
H(6A)-C(6)-H(6C)	109.5	C(12)-C(13)-H(13B)	110.7
H(6B)-C(6)-H(6C)	109.5	H(13A)-C(13)-H(13B)	108.8
O(3)-C(7)-C(8)	124.99(8)	O(5)-C(14)-C(13)	106.1(2)
O(3)-C(7)-C(6)	115.80(8)	O(5)-C(14)-H(14A)	110.5
C(8)-C(7)-C(6)	119.21(8)	C(13)-C(14)-H(14A)	110.5
C(9)-C(8)-C(7)	124.95(8)	O(5)-C(14)-H(14B)	110.5
C(9)-C(8)-H(8)	117.5	C(13)-C(14)-H(14B)	110.5
C(7)-C(8)-H(8)	117.5	H(14A)-C(14)-H(14B)	108.7
O(4)-C(9)-C(8)	125.58(8)	C(18)-O(6)-C(15)	109.8(2)
O(4)-C(9)-C(10)	116.00(8)	C(18)-O(6)-Fe(1)	121.74(7)
C(8)-C(9)-C(10)	118.42(8)	C(15)-O(6)-Fe(1)	124.9(2)
C(9)-C(10)-H(10A)	109.5	O(6)-C(15)-C(16)	105.8(4)
C(9)-C(10)-H(10B)	109.5	O(6)-C(15)-H(15A)	110.6
H(10A)-C(10)-H(10B)	109.5	C(16)-C(15)-H(15A)	110.6
C(9)-C(10)-H(10C)	109.5	O(6)-C(15)-H(15B)	110.6
H(10A)-C(10)-H(10C)	109.5	C(16)-C(15)-H(15B)	110.6
H(10B)-C(10)-H(10C)	109.5	H(15A)-C(15)-H(15B)	108.7
C(11)-O(5)-C(14)	109.83(14)	C(15)-C(16)-C(17)	102.3(4)
C(11)-O(5)-Fe(1)	122.88(8)	C(15)-C(16)-H(16A)	111.3
C(14)-O(5)-Fe(1)	125.90(12)	C(17)-C(16)-H(16A)	111.3
O(5)-C(11)-C(12)	106.87(17)	C(15)-C(16)-H(16B)	111.3
O(5)-C(11)-H(11A)	110.3	C(17)-C(16)-H(16B)	111.3
C(12)-C(11)-H(11A)	110.3	H(16A)-C(16)-H(16B)	109.2

C(18)-C(17)-C(16)	101.8(3)	O(5')-C(14')-H(14C)	110.0
C(18)-C(17)-H(17A)	111.4	C(13')-C(14')-H(14C)	110.0
C(16)-C(17)-H(17A)	111.4	O(5')-C(14')-H(14D)	110.0
C(18)-C(17)-H(17B)	111.4	C(13')-C(14')-H(14D)	110.0
C(16)-C(17)-H(17B)	111.4	H(14C)-C(14')-H(14D)	108.3
H(17A)-C(17)-H(17B)	109.3	C(15')-O(6')-C(18')	105.3(6)
O(6)-C(18)-C(17)	106.4(2)	C(15')-O(6')-Mg(1)	124.1(7)
O(6)-C(18)-H(18A)	110.4	C(18')-O(6')-Mg(1)	115.72(16)
C(17)-C(18)-H(18A)	110.4	O(6')-C(15')-C(16')	105.4(9)
O(6)-C(18)-H(18B)	110.4	O(6')-C(15')-H(15C)	110.7
C(17)-C(18)-H(18B)	110.4	C(16')-C(15')-H(15C)	110.7
H(18A)-C(18)-H(18B)	108.6	O(6')-C(15')-H(15D)	110.7
C(14')-O(5')-C(11')	108.1(5)	C(16')-C(15')-H(15D)	110.7
C(14')-O(5')-Mg(1)	128.1(4)	H(15C)-C(15')-H(15D)	108.8
C(11')-O(5')-Mg(1)	123.8(2)	C(15')-C(16')-C(17')	104.1(10)
C(12')-C(11')-O(5')	108.8(6)	C(15')-C(16')-H(16C)	110.9
C(12')-C(11')-H(11C)	109.9	C(17')-C(16')-H(16C)	110.9
O(5')-C(11')-H(11C)	109.9	C(15')-C(16')-H(16D)	110.9
C(12')-C(11')-H(11D)	109.9	C(17')-C(16')-H(16D)	110.9
O(5')-C(11')-H(11D)	109.9	H(16C)-C(16')-H(16D)	108.9
H(11C)-C(11')-H(11D)	108.3	C(18')-C(17')-C(16')	102.8(9)
C(11')-C(12')-C(13')	109.0(10)	C(18')-C(17')-H(17C)	111.2
C(11')-C(12')-H(12C)	109.9	C(16')-C(17')-H(17C)	111.2
C(13')-C(12')-H(12C)	109.9	C(18')-C(17')-H(17D)	111.2
C(11')-C(12')-H(12D)	109.9	C(16')-C(17')-H(17D)	111.2
C(13')-C(12')-H(12D)	109.9	H(17C)-C(17')-H(17D)	109.1
H(12C)-C(12')-H(12D)	108.3	C(17')-C(18')-O(6')	98.4(5)
C(14')-C(13')-C(12')	103.3(9)	C(17')-C(18')-H(18C)	112.1
C(14')-C(13')-H(13C)	111.1	O(6')-C(18')-H(18C)	112.1
C(12')-C(13')-H(13C)	111.1	C(17')-C(18')-H(18D)	112.1
C(14')-C(13')-H(13D)	111.1	O(6')-C(18')-H(18D)	112.1
C(12')-C(13')-H(13D)	111.1	H(18C)-C(18')-H(18D)	109.7
H(13C)-C(13')-H(13D)	109.1		
O(5')-C(14')-C(13')	108.7(7)		

3.9 $[Mg(acac)(THF)_4]_2[FeBr(4\text{-}F\text{-}Ph)(\mu\text{-}4\text{-}F\text{-}Ph)]_2 \cdot 2.5\ THF$ (4)

REFERENCE NUMBER: neisc29

CRYSTAL STRUCTURE REPORT

$C_{76}\ H_{114}\ Br_2\ F_4\ Fe_2\ Mg_2\ O_{14.5}$

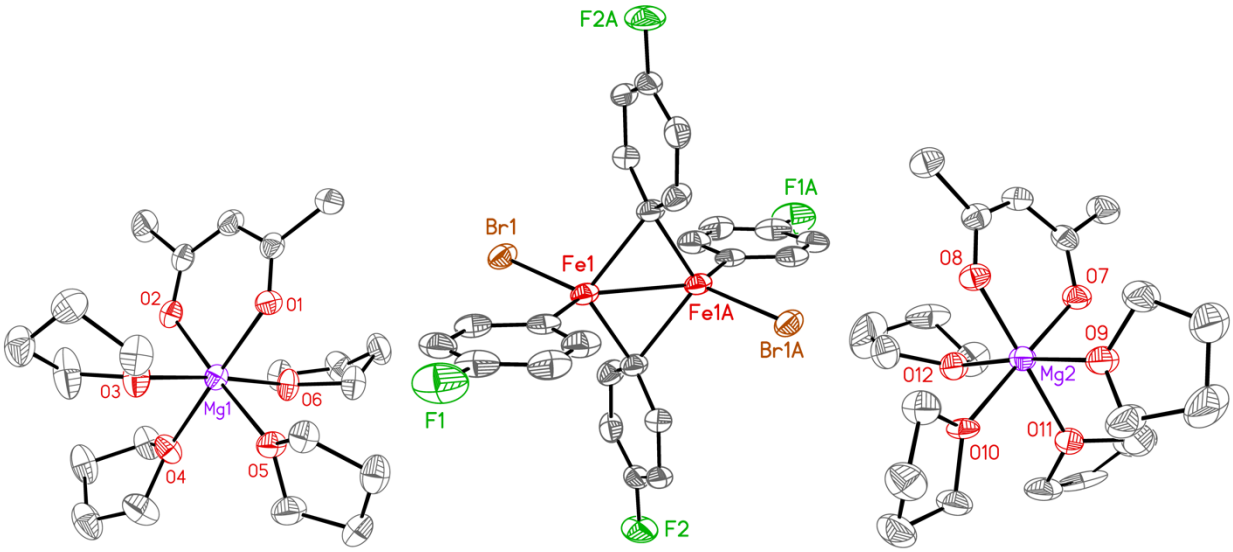
or

$[Mg(acac)(THF)_4]_2[FeBr(4\text{-}F\text{-}Ph)(\mu\text{-}4\text{-}F\text{-}Ph)]_2 \cdot 2.5THF$

Report prepared for:

S. Carpenter, Prof. M. Neidig

June 13, 2017



William W. Brennessel

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Data collection

A crystal (0.50 x 0.20 x 0.12 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 50 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 3982 strong reflections from the actual data collection after integration.[3] See Table 1 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 $R1 = 0.0554$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1514$ (F^2, all data).

Structure description

The structure is the one suggested. The asymmetric unit contains one iron atom and two fluorophenyl ligands of two independent dianionic iron complexes located near crystallographic inversion centers that generate the full dianions, two monoanionic magnesium complexes in general positions, two cocrystallized THF solvent molecules in general positions, and one half of a cocrystallized THF solvent molecule located on a crystallographic inversion center. Two Mg-coordinating THF molecules are modeled as disordered over two positions each (0.53:0.47 and 0.65:0.35 for molecules O10/C55-C58 and O11/C59-C62, respectively). Cocrystallized THF solvent molecule O15/C75-C78 is modeled as disordered over a crystallographic inversion center (0.50:0.50).

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:

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

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

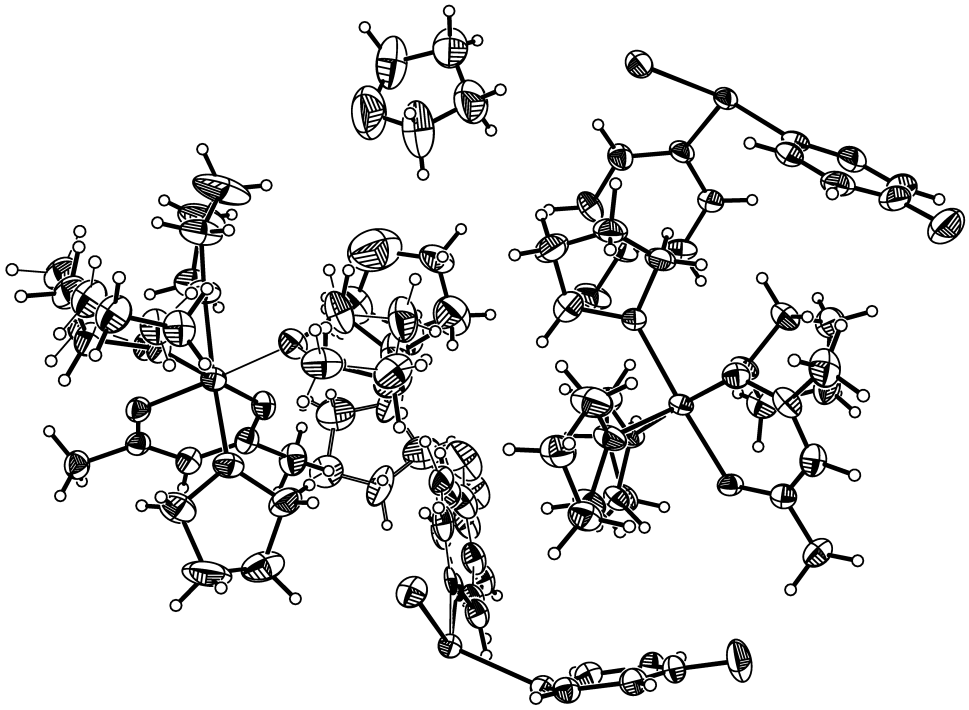
$$wR2 = [\Sigma[w(F_o^2-F_c^2)^2] \ / \ \Sigma\ [w(F_o^2)^2]]^{1/2}$$

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

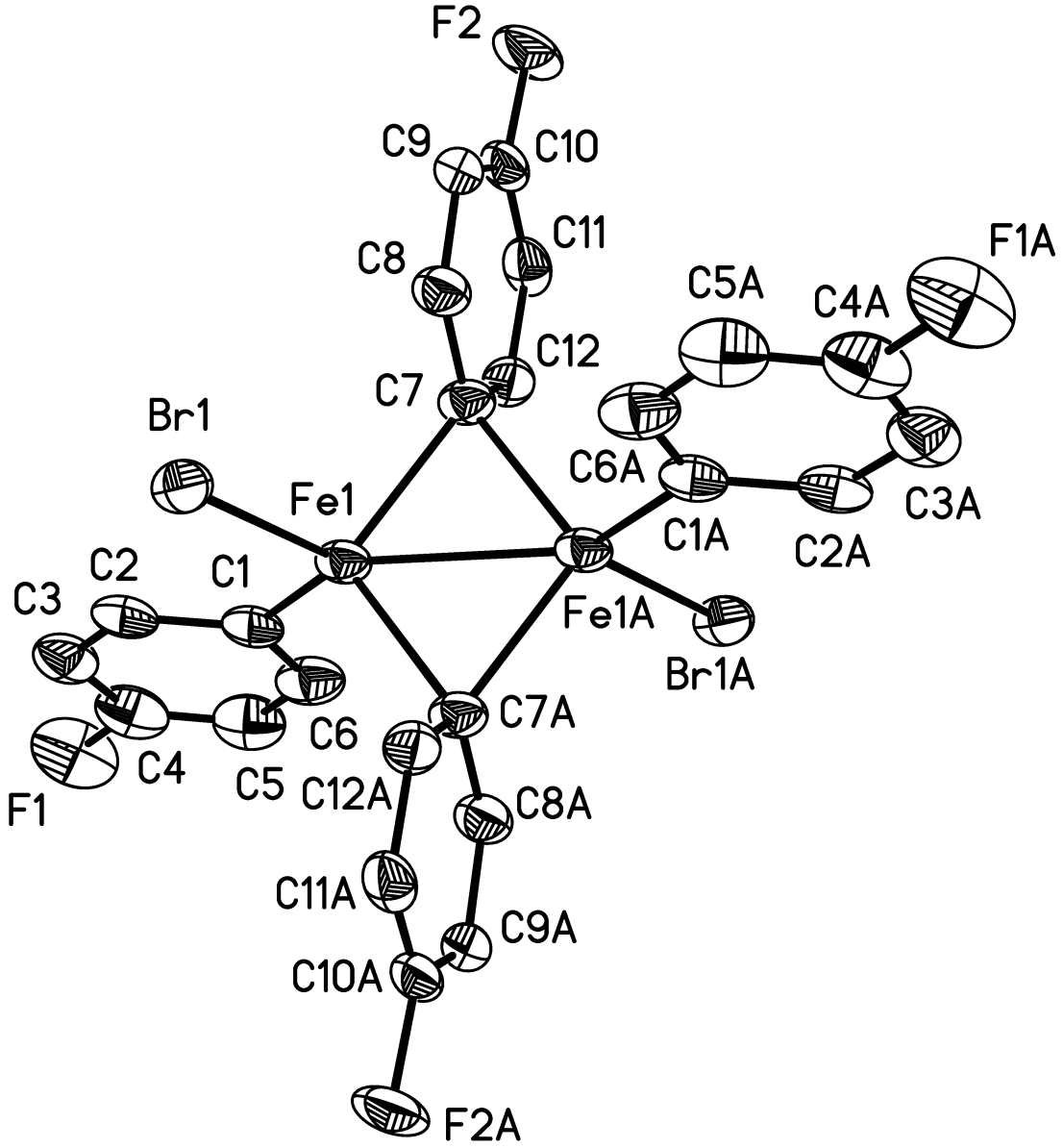
$$P = 1/3 \max\ (0, F_o^2) + 2/3\ F_c^2$$

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

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



The asymmetric unit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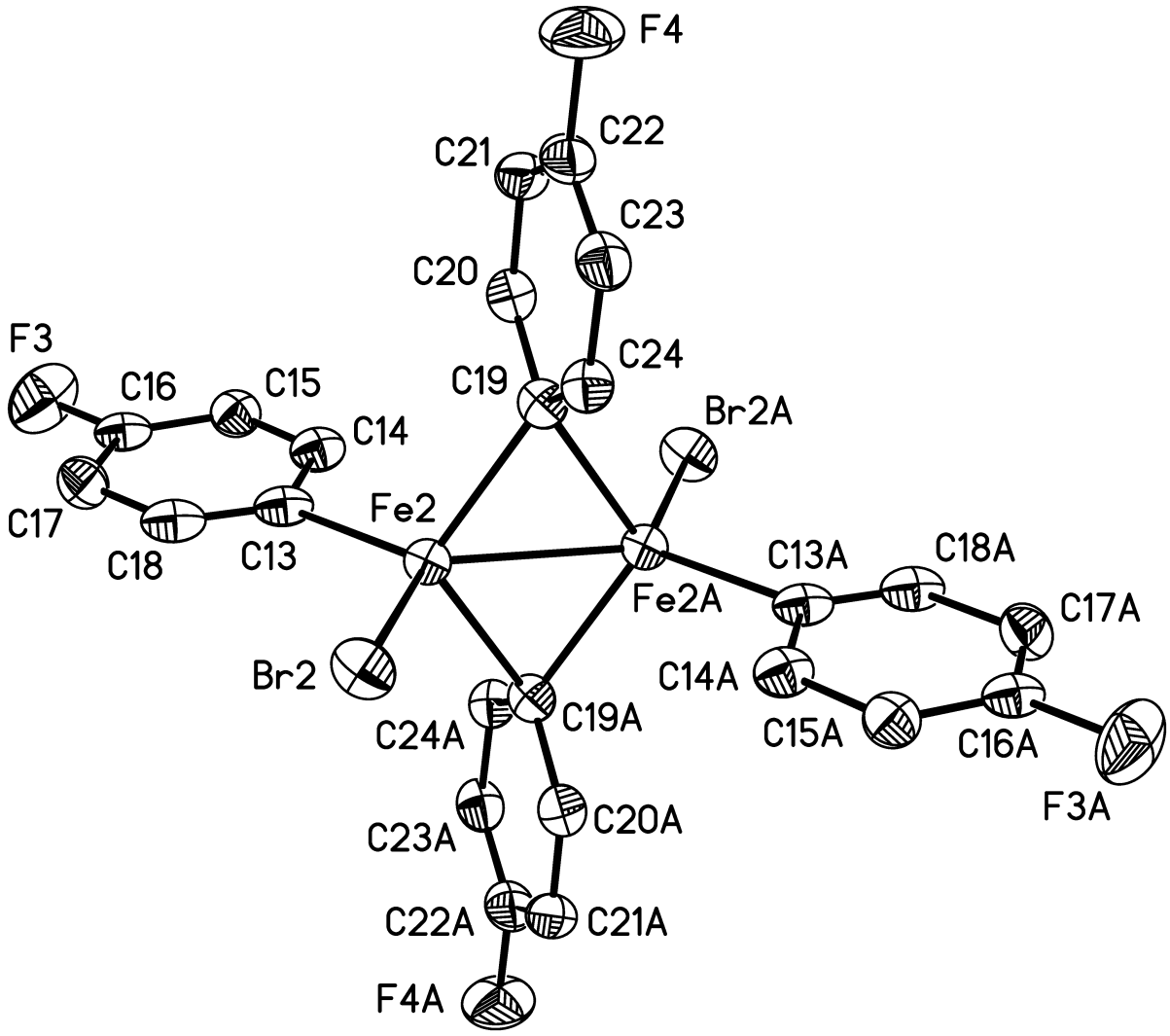
C9
C10
C11
C8
C12
C7
Br1
Fe1
C1
C2
C3
C4
C5
C6
F1
C7A
C12A
C8A
C11A
C9A
C10A
F2A
Fe1A
Br1A
C1A
C6A
C5A
C4A
F1A
C2A
C3A


F4
C22
C21
C23
C20
C24
F3
C16
C15
C19
Br2A
C14
Fe2
C17
C18
C13
C13A
C18A
Fe2A
C17A
Br2
C14A
C19A
C24A
C15A
C16A
F3A
C23A
C20A
C22A
C21A
F4A

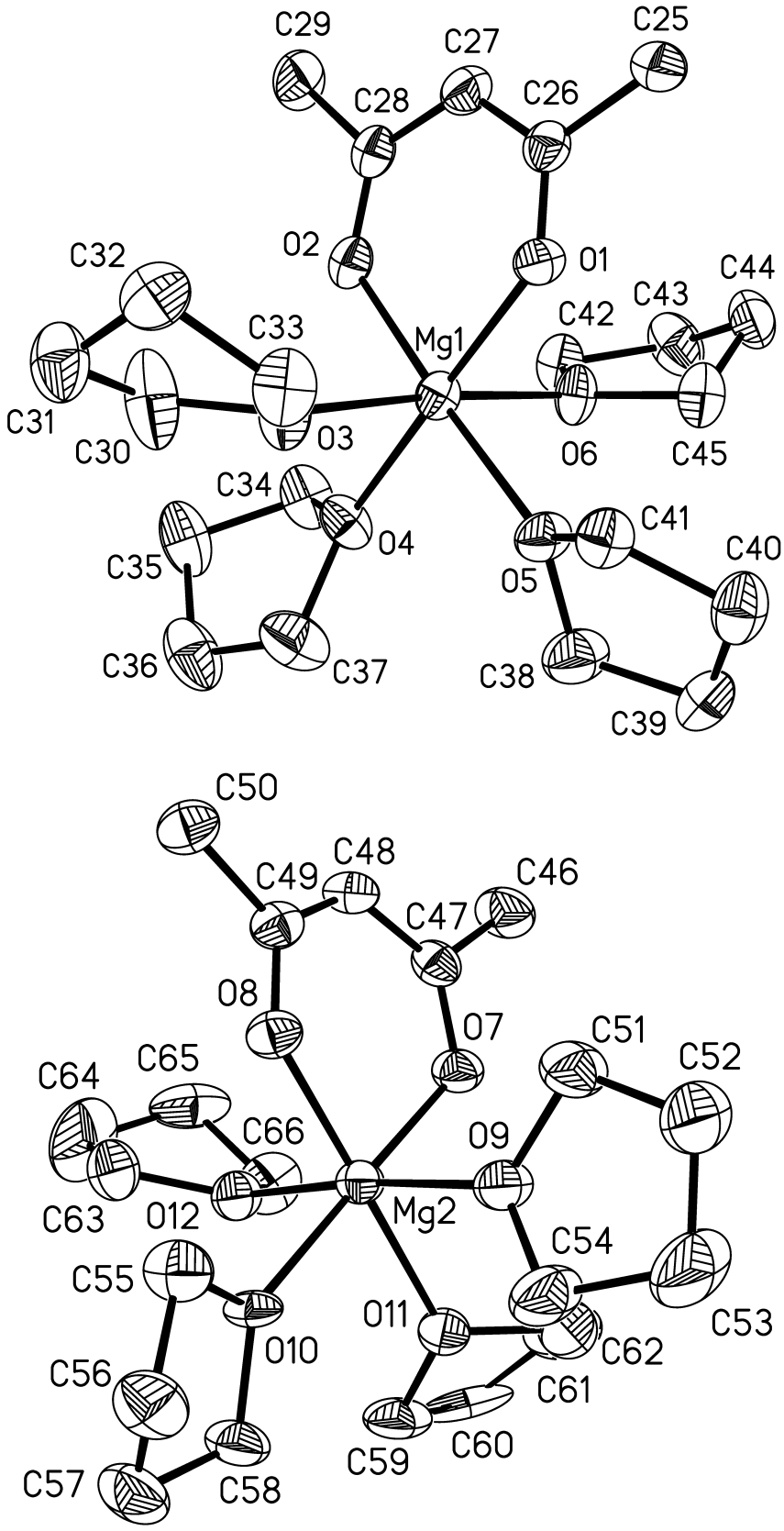

C29
C27
C25
C28
C26
O2
O1
C32
C44
C43
C33
C42
Mg1
C31
C30
O3
O6
C45
C34
C41
O4
C40
C35
O5
C36
C37
C38
C39
C50
C48
C46
C49
C47
O8
O7
C51
C52
C64
C65
C66
O9
C63
O12
Mg2
C54
C55
O11
C53
O10
C62
C56
C61
C59
C60
C57
C58

Table S27. Crystal data and structure refinement for neisc29.

Identification code	neisc29	
Empirical formula	C76 H114 Br2 F4 Fe2 Mg2 O14.5	
Formula weight	1655.81	
Temperature	100.0(5) K	
Wavelength	0.71073 Å	
Crystal system	triclinic	
Space group	*P*-1	
Unit cell dimensions	a = 12.3417(9) Å	α = 100.0619(15)°
	b = 15.4069(11) Å	β = 99.2790(16)°
	c = 21.5668(15) Å	γ = 94.4973(16)°
Volume	3960.9(5) Å^3	
Z	2	
Density (calculated)	1.388 Mg/m^3	
Absorption coefficient	1.461 mm^{-1}	
F(000)	1736	
Crystal color, morphology	orange, needle	
Crystal size	0.50 x 0.20 x 0.12 mm^3	
Theta range for data collection	1.682 to 28.306°	
Index ranges	$-16 \le h \le 16$, $-20 \le k \le 20$, $-28 \le l \le 28$	
Reflections collected	59640	
Independent reflections	19681 [R(int) = 0.0644]	
Observed reflections	12120	
Completeness to theta = 28.283°	99.8%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.7457 and 0.5836	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	19681 / 99 / 959	
Goodness-of-fit on F^2	1.058	
Final R indices [I>2sigma(I)]	R1 = 0.0554, wR2 = 0.1323	
R indices (all data)	R1 = 0.1056, wR2 = 0.1514	
Largest diff. peak and hole	0.978 and -0.594 e.Å^{-3}	

Table S28. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for neisc29. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Fe1	745(1)	4473(1)	-108(1)	29(1)
Br1	2264(1)	5088(1)	-591(1)	38(1)
F1	1337(3)	520(2)	47(1)	72(1)
F2	2296(2)	6651(2)	2617(1)	55(1)
C1	923(3)	3172(3)	-22(2)	34(1)
C2	1868(4)	2783(3)	-170(2)	39(1)
C3	2023(4)	1909(3)	-137(2)	44(1)
C4	1197(4)	1397(3)	37(2)	48(1)
C5	279(4)	1721(3)	201(2)	50(1)
C6	145(4)	2612(3)	168(2)	45(1)
C7	782(3)	5411(2)	771(2)	32(1)
C8	1591(3)	6141(2)	890(2)	36(1)
C9	2127(3)	6542(3)	1502(2)	37(1)
C10	1814(3)	6221(3)	2008(2)	36(1)
C11	1055(3)	5505(3)	1937(2)	37(1)
C12	544(3)	5101(2)	1318(2)	32(1)
Fe2	9562(1)	5350(1)	4564(1)	24(1)
Br2	10365(1)	5204(1)	3579(1)	37(1)
F4	7029(2)	1614(2)	3962(1)	60(1)
C13	8209(7)	6132(6)	4573(5)	28(1)
C14	7697(7)	6207(6)	5128(4)	32(1)
C15	6799(6)	6695(5)	5165(4)	33(2)
C16	6438(4)	7099(4)	4663(4)	28(1)
F3	5552(4)	7589(3)	4675(4)	64(2)
C17	6903(6)	7023(5)	4124(4)	39(2)
C18	7825(6)	6517(5)	4129(4)	32(1)
C13'	8207(12)	6145(10)	4513(9)	28(1)
C14'	7630(14)	6336(11)	5019(9)	32(1)
C15'	6738(13)	6840(10)	4966(8)	33(2)
C16'	6498(9)	7118(6)	4390(8)	28(1)
F3'	5608(9)	7622(7)	4327(7)	64(2)

C17'	7026(13)	6954(10)	3893(8)	39(2)
C18'	7937(13)	6427(11)	3967(8)	32(1)
C19	9054(3)	4003(2)	4671(2)	27(1)
C20	7982(3)	3861(2)	4809(2)	30(1)
C21	7293(3)	3073(2)	4576(2)	34(1)
C22	7688(3)	2401(2)	4194(2)	38(1)
C23	8714(3)	2479(2)	4035(2)	34(1)
C24	9383(3)	3285(2)	4268(2)	29(1)
Mg1	4507(1)	3688(1)	2408(1)	24(1)
O1	4636(2)	2871(2)	3046(1)	27(1)
O2	4466(2)	2634(2)	1705(1)	30(1)
C25	4311(3)	1604(2)	3483(2)	38(1)
C26	4444(3)	2040(2)	2922(2)	28(1)
C27	4344(3)	1505(2)	2314(2)	35(1)
C28	4393(3)	1829(2)	1749(2)	30(1)
C29	4345(4)	1164(3)	1134(2)	48(1)
O3	6227(2)	3979(2)	2460(1)	39(1)
C30	6720(4)	3876(4)	1878(2)	62(1)
C31	7921(3)	3783(3)	2088(2)	49(1)
C32	7949(3)	3454(3)	2705(2)	42(1)
C33	7080(3)	3965(3)	2992(2)	42(1)
O4	4291(2)	4512(2)	1730(1)	33(1)
C34	3716(3)	4231(3)	1072(2)	41(1)
C35	4502(4)	4583(3)	687(2)	49(1)
C36	4892(4)	5498(3)	1086(2)	54(1)
C37	4804(4)	5411(3)	1762(2)	54(1)
O5	4542(2)	4735(2)	3175(1)	31(1)
C38	3988(4)	5516(3)	3150(2)	45(1)
C39	3685(3)	5814(3)	3792(2)	40(1)
C40	4289(3)	5244(3)	4219(2)	40(1)
C41	5100(3)	4827(2)	3833(2)	32(1)
O6	2748(2)	3498(2)	2344(1)	32(1)
C42	1931(3)	3105(3)	1781(2)	38(1)
C43	872(3)	2871(3)	2026(2)	50(1)
C44	1281(4)	2821(3)	2714(2)	48(1)
C45	2264(3)	3516(3)	2910(2)	43(1)

Mg2	9710(1)	9204(1)	2735(1)	28(1)
O7	8644(2)	9261(2)	3346(1)	32(1)
O8	10572(2)	10313(2)	3277(1)	31(1)
C46	7967(4)	9538(3)	4320(2)	45(1)
C47	8808(3)	9717(2)	3916(2)	31(1)
C48	9698(3)	10366(2)	4175(2)	33(1)
C49	10497(3)	10654(2)	3847(2)	30(1)
C50	11343(3)	11429(2)	4176(2)	39(1)
O9	8723(2)	9948(2)	2175(1)	38(1)
C51	8234(4)	10732(3)	2435(2)	47(1)
C52	7434(5)	10927(4)	1889(3)	74(2)
C53	7464(6)	10224(4)	1321(3)	97(2)
C54	8538(4)	9866(3)	1489(2)	57(1)
O10	10791(17)	9146(7)	2086(9)	29(2)
C55	11538(10)	9931(6)	2065(5)	43(2)
C56	11828(10)	9731(7)	1406(5)	50(2)
C57	11880(9)	8739(7)	1304(5)	51(2)
C58	10907(14)	8414(7)	1586(8)	41(2)
O10'	10840(19)	9398(8)	2137(10)	29(2)
C55'	11304(13)	10275(7)	2078(6)	43(2)
C56'	11533(11)	10161(8)	1400(5)	50(2)
C57'	11868(11)	9234(9)	1300(6)	51(2)
C58'	11085(17)	8732(8)	1625(9)	41(2)
O11	8820(2)	8056(2)	2138(1)	36(1)
C59	9113(15)	7155(6)	1990(20)	50(1)
C60	8237(12)	6576(8)	2200(10)	68(3)
C61	7367(7)	7180(5)	2324(5)	71(2)
C62	7640(7)	7967(8)	2019(15)	58(1)
O11'	8820(2)	8056(2)	2138(1)	36(1)
C59'	9170(30)	7182(11)	1970(40)	50(1)
C60'	8240(20)	6533(14)	2070(20)	68(3)
C61'	7225(13)	6980(10)	1872(10)	71(2)
C62'	7641(11)	7961(13)	2030(30)	58(1)
O12	10644(2)	8363(2)	3220(1)	36(1)
C63	11811(4)	8558(3)	3481(2)	50(1)
C64	12012(5)	8041(4)	4017(3)	86(2)

C65	10897(5)	7884(3)	4204(2)	62(1)
C66	10169(4)	7740(3)	3565(2)	51(1)
O13	4608(4)	8172(3)	2115(2)	110(2)
C67	4681(5)	8021(4)	2779(3)	78(2)
C68	4918(5)	8916(4)	3212(3)	84(2)
C69	5648(5)	9398(4)	2857(3)	70(2)
C70	5176(5)	9074(3)	2182(3)	72(2)
O14	5049(5)	8692(3)	448(3)	136(2)
C71	3944(5)	8909(3)	128(3)	81(2)
C72	3335(5)	8061(3)	-164(3)	72(2)
C73	3867(5)	7415(4)	185(3)	93(2)
C74	4995(5)	7738(4)	378(4)	101(2)
O15	6253(7)	10446(5)	5295(4)	77(2)
C75	5410(40)	10610(40)	4780(20)	78(5)
C76	4280(40)	10210(20)	4760(30)	82(6)
C77	4570(40)	9380(40)	5090(20)	78(5)
C78	5780(40)	9540(20)	5260(30)	82(6)

Table S29. Bond lengths [Å] and angles [°] for neisc29.

Fe(1)-C(1)	2.072(4)	C(13)-C(18)	1.262(8)
Fe(1)-C(7)	2.166(4)	C(13)-C(14)	1.432(8)
Fe(1)-C(7)#1	2.212(4)	C(14)-C(15)	1.392(8)
Fe(1)-Br(1)	2.4886(6)	C(14)-H(14)	0.9500
Fe(1)-Fe(1)#1	2.5903(11)	C(15)-C(16)	1.372(8)
F(1)-C(4)	1.378(5)	C(15)-H(15)	0.9500
F(2)-C(10)	1.379(4)	C(16)-C(17)	1.367(8)
C(1)-C(6)	1.399(5)	C(16)-F(3)	1.378(6)
C(1)-C(2)	1.408(6)	C(17)-C(18)	1.429(8)
C(2)-C(3)	1.387(6)	C(17)-H(17)	0.9500
C(2)-H(2)	0.9500	C(18)-H(18)	0.9500
C(3)-C(4)	1.379(6)	C(13')-C(18')	1.327(13)
C(3)-H(3)	0.9500	C(13')-C(14')	1.397(13)
C(4)-C(5)	1.348(6)	C(14')-C(15')	1.398(14)
C(5)-C(6)	1.409(6)	C(14')-H(14')	0.9500
C(5)-H(5)	0.9500	C(15')-C(16')	1.378(14)
C(6)-H(6)	0.9500	C(15')-H(15')	0.9500
C(7)-C(8)	1.403(5)	C(16')-C(17')	1.338(14)
C(7)-C(12)	1.413(5)	C(16')-F(3')	1.396(12)
C(8)-C(9)	1.389(5)	C(17')-C(18')	1.443(14)
C(8)-H(8)	0.9500	C(17')-H(17')	0.9500
C(9)-C(10)	1.370(6)	C(18')-H(18')	0.9500
C(9)-H(9)	0.9500	C(19)-C(20)	1.411(5)
C(10)-C(11)	1.361(6)	C(19)-C(24)	1.412(5)
C(11)-C(12)	1.394(5)	C(20)-C(21)	1.392(5)
C(11)-H(11)	0.9500	C(20)-H(20)	0.9500
C(12)-H(12)	0.9500	C(21)-C(22)	1.378(5)
Fe(2)-C(13)	2.134(7)	C(21)-H(21)	0.9500
Fe(2)-C(13')	2.148(12)	C(22)-C(23)	1.366(6)
Fe(2)-C(19)	2.182(3)	C(23)-C(24)	1.401(5)
Fe(2)-C(19)#2	2.210(3)	C(23)-H(23)	0.9500
Fe(2)-Br(2)	2.4680(6)	C(24)-H(24)	0.9500
Fe(2)-Fe(2)#2	2.4710(10)	Mg(1)-O(2)	2.010(3)
F(4)-C(22)	1.372(4)	Mg(1)-O(1)	2.017(3)

Mg(1)-O(5)	2.091(3)	C(35)-H(35A)	0.9900
Mg(1)-O(4)	2.097(3)	C(35)-H(35B)	0.9900
Mg(1)-O(3)	2.115(3)	C(36)-C(37)	1.506(6)
Mg(1)-O(6)	2.147(3)	C(36)-H(36A)	0.9900
O(1)-C(26)	1.256(4)	C(36)-H(36B)	0.9900
O(2)-C(28)	1.258(4)	C(37)-H(37A)	0.9900
C(25)-C(26)	1.509(5)	C(37)-H(37B)	0.9900
C(25)-H(25A)	0.9800	O(5)-C(38)	1.435(5)
C(25)-H(25B)	0.9800	O(5)-C(41)	1.451(4)
C(25)-H(25C)	0.9800	C(38)-C(39)	1.497(6)
C(26)-C(27)	1.402(5)	C(38)-H(38A)	0.9900
C(27)-C(28)	1.403(5)	C(38)-H(38B)	0.9900
C(27)-H(27)	0.9500	C(39)-C(40)	1.529(6)
C(28)-C(29)	1.519(5)	C(39)-H(39A)	0.9900
C(29)-H(29A)	0.9800	C(39)-H(39B)	0.9900
C(29)-H(29B)	0.9800	C(40)-C(41)	1.515(5)
C(29)-H(29C)	0.9800	C(40)-H(40A)	0.9900
O(3)-C(33)	1.432(4)	C(40)-H(40B)	0.9900
O(3)-C(30)	1.470(5)	C(41)-H(41A)	0.9900
C(30)-C(31)	1.505(6)	C(41)-H(41B)	0.9900
C(30)-H(30A)	0.9900	O(6)-C(45)	1.440(4)
C(30)-H(30B)	0.9900	O(6)-C(42)	1.453(4)
C(31)-C(32)	1.501(6)	C(42)-C(43)	1.527(5)
C(31)-H(31A)	0.9900	C(42)-H(42A)	0.9900
C(31)-H(31B)	0.9900	C(42)-H(42B)	0.9900
C(32)-C(33)	1.526(6)	C(43)-C(44)	1.505(6)
C(32)-H(32A)	0.9900	C(43)-H(43A)	0.9900
C(32)-H(32B)	0.9900	C(43)-H(43B)	0.9900
C(33)-H(33A)	0.9900	C(44)-C(45)	1.507(6)
C(33)-H(33B)	0.9900	C(44)-H(44A)	0.9900
O(4)-C(34)	1.454(4)	C(44)-H(44B)	0.9900
O(4)-C(37)	1.462(5)	C(45)-H(45A)	0.9900
C(34)-C(35)	1.504(5)	C(45)-H(45B)	0.9900
C(34)-H(34A)	0.9900	Mg(2)-O(7)	2.003(3)
C(34)-H(34B)	0.9900	Mg(2)-O(8)	2.008(3)
C(35)-C(36)	1.516(7)	Mg(2)-O(10)	2.080(8)

Mg(2)-O(10')	2.084(9)	C(56)-C(57)	1.513(12)
Mg(2)-O(12)	2.102(3)	C(56)-H(56A)	0.9900
Mg(2)-O(11')	2.108(3)	C(56)-H(56B)	0.9900
Mg(2)-O(11)	2.108(3)	C(57)-C(58)	1.520(11)
Mg(2)-O(9)	2.118(3)	C(57)-H(57A)	0.9900
O(7)-C(47)	1.281(4)	C(57)-H(57B)	0.9900
O(8)-C(49)	1.270(4)	C(58)-H(58A)	0.9900
C(46)-C(47)	1.497(5)	C(58)-H(58B)	0.9900
C(46)-H(46A)	0.9800	O(10')-C(58')	1.457(10)
C(46)-H(46B)	0.9800	O(10')-C(55')	1.462(11)
C(46)-H(46C)	0.9800	C(55')-C(56')	1.515(11)
C(47)-C(48)	1.398(5)	C(55')-H(55C)	0.9900
C(48)-C(49)	1.391(5)	C(55')-H(55D)	0.9900
C(48)-H(48)	0.9500	C(56')-C(57')	1.508(13)
C(49)-C(50)	1.512(5)	C(56')-H(56C)	0.9900
C(50)-H(50A)	0.9800	C(56')-H(56D)	0.9900
C(50)-H(50B)	0.9800	C(57')-C(58')	1.522(12)
C(50)-H(50C)	0.9800	C(57')-H(57C)	0.9900
O(9)-C(54)	1.442(5)	C(57')-H(57D)	0.9900
O(9)-C(51)	1.458(4)	C(58')-H(58C)	0.9900
C(51)-C(52)	1.500(6)	C(58')-H(58D)	0.9900
C(51)-H(51A)	0.9900	O(11)-C(62)	1.429(8)
C(51)-H(51B)	0.9900	O(11)-C(59)	1.455(8)
C(52)-C(53)	1.493(7)	C(59)-C(60)	1.525(11)
C(52)-H(52A)	0.9900	C(59)-H(59A)	0.9900
C(52)-H(52B)	0.9900	C(59)-H(59B)	0.9900
C(53)-C(54)	1.490(7)	C(60)-C(61)	1.501(12)
C(53)-H(53A)	0.9900	C(60)-H(60A)	0.9900
C(53)-H(53B)	0.9900	C(60)-H(60B)	0.9900
C(54)-H(54A)	0.9900	C(61)-C(62)	1.518(15)
C(54)-H(54B)	0.9900	C(61)-H(61A)	0.9900
O(10)-C(58)	1.452(9)	C(61)-H(61B)	0.9900
O(10)-C(55)	1.474(10)	C(62)-H(62A)	0.9900
C(55)-C(56)	1.507(10)	C(62)-H(62B)	0.9900
C(55)-H(55A)	0.9900	O(11')-C(62')	1.429(13)
C(55)-H(55B)	0.9900	O(11')-C(59')	1.445(13)

C(59')-C(60')	1.523(14)	C(70)-H(70B)	0.9900
C(59')-H(59C)	0.9900	O(14)-C(74)	1.445(7)
C(59')-H(59D)	0.9900	O(14)-C(71)	1.515(8)
C(60')-C(61')	1.507(17)	C(71)-C(72)	1.440(7)
C(60')-H(60C)	0.9900	C(71)-H(71A)	0.9900
C(60')-H(60D)	0.9900	C(71)-H(71B)	0.9900
C(61')-C(62')	1.519(16)	C(72)-C(73)	1.481(8)
C(61')-H(61C)	0.9900	C(72)-H(72A)	0.9900
C(61')-H(61D)	0.9900	C(72)-H(72B)	0.9900
C(62')-H(62C)	0.9900	C(73)-C(74)	1.411(8)
C(62')-H(62D)	0.9900	C(73)-H(73A)	0.9900
O(12)-C(63)	1.448(5)	C(73)-H(73B)	0.9900
O(12)-C(66)	1.457(5)	C(74)-H(74A)	0.9900
C(63)-C(64)	1.515(7)	C(74)-H(74B)	0.9900
C(63)-H(63A)	0.9900	O(15)-C(78)	1.46(3)
C(63)-H(63B)	0.9900	O(15)-C(75)	1.47(4)
C(64)-C(65)	1.508(8)	C(75)-C(76)	1.466(19)
C(64)-H(64A)	0.9900	C(75)-H(75A)	0.9900
C(64)-H(64B)	0.9900	C(75)-H(75B)	0.9900
C(65)-C(66)	1.489(6)	C(76)-C(77)	1.60(7)
C(65)-H(65A)	0.9900	C(76)-H(76A)	0.9900
C(65)-H(65B)	0.9900	C(76)-H(76B)	0.9900
C(66)-H(66A)	0.9900	C(77)-C(78)	1.469(19)
C(66)-H(66B)	0.9900	C(77)-H(77A)	0.9900
O(13)-C(70)	1.479(7)	C(77)-H(77B)	0.9900
O(13)-C(67)	1.482(7)	C(78)-H(78A)	0.9900
C(67)-C(68)	1.502(8)	C(78)-H(78B)	0.9900
C(67)-H(67A)	0.9900	C(1)-Fe(1)-C(7)	116.53(14)
C(67)-H(67B)	0.9900	C(1)-Fe(1)-C(7)#1	112.26(14)
C(68)-C(69)	1.504(8)	C(7)-Fe(1)-C(7)#1	107.46(11)
C(68)-H(68A)	0.9900	C(1)-Fe(1)-Br(1)	110.62(11)
C(68)-H(68B)	0.9900	C(7)-Fe(1)-Br(1)	104.52(10)
C(69)-C(70)	1.460(7)	C(7)#1-Fe(1)-Br(1)	104.51(9)
C(69)-H(69A)	0.9900	C(1)-Fe(1)-Fe(1)#1	134.19(12)
C(69)-H(69B)	0.9900	C(7)-Fe(1)-Fe(1)#1	54.56(10)
C(70)-H(70A)	0.9900	C(7)#1-Fe(1)-Fe(1)#1	52.90(10)

Br(1)-Fe(1)-Fe(1)#1	115.06(3)	C(12)-C(11)-H(11)	121.0
C(6)-C(1)-C(2)	115.0(4)	C(11)-C(12)-C(7)	122.5(4)
C(6)-C(1)-Fe(1)	124.6(3)	C(11)-C(12)-H(12)	118.7
C(2)-C(1)-Fe(1)	120.4(3)	C(7)-C(12)-H(12)	118.7
C(3)-C(2)-C(1)	123.1(4)	C(13)-Fe(2)-C(19)	110.6(3)
C(3)-C(2)-H(2)	118.4	C(13')-Fe(2)-C(19)	112.2(5)
C(1)-C(2)-H(2)	118.4	C(13)-Fe(2)-C(19)#2	109.3(3)
C(4)-C(3)-C(2)	118.0(4)	C(13')-Fe(2)-C(19)#2	111.1(5)
C(4)-C(3)-H(3)	121.0	C(19)-Fe(2)-C(19)#2	111.52(10)
C(2)-C(3)-H(3)	121.0	C(13)-Fe(2)-Br(2)	114.9(3)
C(5)-C(4)-F(1)	119.4(4)	C(13')-Fe(2)-Br(2)	111.3(5)
C(5)-C(4)-C(3)	122.8(4)	C(19)-Fe(2)-Br(2)	106.28(9)
F(1)-C(4)-C(3)	117.8(4)	C(19)#2-Fe(2)-Br(2)	104.07(9)
C(4)-C(5)-C(6)	118.1(4)	C(13)-Fe(2)-Fe(2)#2	127.4(3)
C(4)-C(5)-H(5)	121.0	C(19)-Fe(2)-Fe(2)#2	56.30(9)
C(6)-C(5)-H(5)	121.0	C(19)#2-Fe(2)-Fe(2)#2	55.22(9)
C(1)-C(6)-C(5)	123.0(4)	Br(2)-Fe(2)-Fe(2)#2	117.70(3)
C(1)-C(6)-H(6)	118.5	C(18)-C(13)-C(14)	118.3(6)
C(5)-C(6)-H(6)	118.5	C(18)-C(13)-Fe(2)	125.2(7)
C(8)-C(7)-C(12)	115.5(3)	C(14)-C(13)-Fe(2)	116.5(6)
C(8)-C(7)-Fe(1)	115.3(3)	C(15)-C(14)-C(13)	119.9(6)
C(12)-C(7)-Fe(1)	119.6(3)	C(15)-C(14)-H(14)	120.1
C(8)-C(7)-Fe(1)#1	115.1(3)	C(13)-C(14)-H(14)	120.1
C(12)-C(7)-Fe(1)#1	111.6(3)	C(16)-C(15)-C(14)	118.2(6)
Fe(1)-C(7)-Fe(1)#1	72.54(11)	C(16)-C(15)-H(15)	120.9
C(9)-C(8)-C(7)	123.0(4)	C(14)-C(15)-H(15)	120.9
C(9)-C(8)-H(8)	118.5	C(17)-C(16)-C(15)	122.8(6)
C(7)-C(8)-H(8)	118.5	C(17)-C(16)-F(3)	116.6(6)
C(10)-C(9)-C(8)	117.7(4)	C(15)-C(16)-F(3)	120.6(6)
C(10)-C(9)-H(9)	121.1	C(16)-C(17)-C(18)	115.3(6)
C(8)-C(9)-H(9)	121.1	C(16)-C(17)-H(17)	122.4
C(11)-C(10)-C(9)	123.3(3)	C(18)-C(17)-H(17)	122.4
C(11)-C(10)-F(2)	118.8(4)	C(13)-C(18)-C(17)	125.5(6)
C(9)-C(10)-F(2)	117.9(4)	C(13)-C(18)-H(18)	117.2
C(10)-C(11)-C(12)	117.9(4)	C(17)-C(18)-H(18)	117.2
C(10)-C(11)-H(11)	121.0	C(18')-C(13')-C(14')	122.1(11)

C(18')-C(13')-Fe(2)	115.8(12)	C(23)-C(24)-H(24)	118.5
C(14')-C(13')-Fe(2)	122.1(12)	C(19)-C(24)-H(24)	118.5
C(13')-C(14')-C(15')	120.7(12)	O(2)-Mg(1)-O(1)	89.01(11)
C(13')-C(14')-H(14')	119.7	O(2)-Mg(1)-O(5)	176.78(11)
C(15')-C(14')-H(14')	119.7	O(1)-Mg(1)-O(5)	87.81(10)
C(16')-C(15')-C(14')	114.7(13)	O(2)-Mg(1)-O(4)	90.20(11)
C(16')-C(15')-H(15')	122.6	O(1)-Mg(1)-O(4)	177.14(11)
C(14')-C(15')-H(15')	122.6	O(5)-Mg(1)-O(4)	92.94(10)
C(17')-C(16')-C(15')	126.9(12)	O(2)-Mg(1)-O(3)	90.85(11)
C(17')-C(16')-F(3')	117.5(13)	O(1)-Mg(1)-O(3)	95.77(11)
C(15')-C(16')-F(3')	115.7(13)	O(5)-Mg(1)-O(3)	90.00(11)
C(16')-C(17')-C(18')	116.6(13)	O(4)-Mg(1)-O(3)	86.99(11)
C(16')-C(17')-H(17')	121.7	O(2)-Mg(1)-O(6)	91.74(10)
C(18')-C(17')-H(17')	121.7	O(1)-Mg(1)-O(6)	88.02(10)
C(13')-C(18')-C(17')	119.1(12)	O(5)-Mg(1)-O(6)	87.62(10)
C(13')-C(18')-H(18')	120.4	O(4)-Mg(1)-O(6)	89.26(10)
C(17')-C(18')-H(18')	120.4	O(3)-Mg(1)-O(6)	175.45(12)
C(20)-C(19)-C(24)	115.0(3)	C(26)-O(1)-Mg(1)	126.4(2)
C(20)-C(19)-Fe(2)	115.4(2)	C(28)-O(2)-Mg(1)	127.3(2)
C(24)-C(19)-Fe(2)	118.8(2)	C(26)-C(25)-H(25A)	109.5
C(20)-C(19)-Fe(2)#2	120.3(2)	C(26)-C(25)-H(25B)	109.5
C(24)-C(19)-Fe(2)#2	111.1(2)	H(25A)-C(25)-H(25B)	109.5
Fe(2)-C(19)-Fe(2)#2	68.48(10)	C(26)-C(25)-H(25C)	109.5
C(21)-C(20)-C(19)	123.3(3)	H(25A)-C(25)-H(25C)	109.5
C(21)-C(20)-H(20)	118.4	H(25B)-C(25)-H(25C)	109.5
C(19)-C(20)-H(20)	118.4	O(1)-C(26)-C(27)	125.4(3)
C(22)-C(21)-C(20)	117.9(3)	O(1)-C(26)-C(25)	115.8(3)
C(22)-C(21)-H(21)	121.1	C(27)-C(26)-C(25)	118.8(3)
C(20)-C(21)-H(21)	121.1	C(26)-C(27)-C(28)	124.5(3)
C(23)-C(22)-F(4)	118.1(4)	C(26)-C(27)-H(27)	117.8
C(23)-C(22)-C(21)	122.9(3)	C(28)-C(27)-H(27)	117.8
F(4)-C(22)-C(21)	119.0(4)	O(2)-C(28)-C(27)	125.4(3)
C(22)-C(23)-C(24)	118.0(3)	O(2)-C(28)-C(29)	116.3(3)
C(22)-C(23)-H(23)	121.0	C(27)-C(28)-C(29)	118.2(3)
C(24)-C(23)-H(23)	121.0	C(28)-C(29)-H(29A)	109.5
C(23)-C(24)-C(19)	123.0(3)	C(28)-C(29)-H(29B)	109.5

H(29A)-C(29)-H(29B)	109.5	C(35)-C(34)-H(34A)	111.0
C(28)-C(29)-H(29C)	109.5	O(4)-C(34)-H(34B)	111.0
H(29A)-C(29)-H(29C)	109.5	C(35)-C(34)-H(34B)	111.0
H(29B)-C(29)-H(29C)	109.5	H(34A)-C(34)-H(34B)	109.0
C(33)-O(3)-C(30)	108.1(3)	C(34)-C(35)-C(36)	101.1(4)
C(33)-O(3)-Mg(1)	126.9(2)	C(34)-C(35)-H(35A)	111.5
C(30)-O(3)-Mg(1)	121.1(2)	C(36)-C(35)-H(35A)	111.5
O(3)-C(30)-C(31)	106.2(4)	C(34)-C(35)-H(35B)	111.5
O(3)-C(30)-H(30A)	110.5	C(36)-C(35)-H(35B)	111.5
C(31)-C(30)-H(30A)	110.5	H(35A)-C(35)-H(35B)	109.4
O(3)-C(30)-H(30B)	110.5	C(37)-C(36)-C(35)	105.1(3)
C(31)-C(30)-H(30B)	110.5	C(37)-C(36)-H(36A)	110.7
H(30A)-C(30)-H(30B)	108.7	C(35)-C(36)-H(36A)	110.7
C(32)-C(31)-C(30)	103.8(3)	C(37)-C(36)-H(36B)	110.7
C(32)-C(31)-H(31A)	111.0	C(35)-C(36)-H(36B)	110.7
C(30)-C(31)-H(31A)	111.0	H(36A)-C(36)-H(36B)	108.8
C(32)-C(31)-H(31B)	111.0	O(4)-C(37)-C(36)	106.8(4)
C(30)-C(31)-H(31B)	111.0	O(4)-C(37)-H(37A)	110.4
H(31A)-C(31)-H(31B)	109.0	C(36)-C(37)-H(37A)	110.4
C(31)-C(32)-C(33)	101.1(3)	O(4)-C(37)-H(37B)	110.4
C(31)-C(32)-H(32A)	111.6	C(36)-C(37)-H(37B)	110.4
C(33)-C(32)-H(32A)	111.6	H(37A)-C(37)-H(37B)	108.6
C(31)-C(32)-H(32B)	111.6	C(38)-O(5)-C(41)	105.9(3)
C(33)-C(32)-H(32B)	111.6	C(38)-O(5)-Mg(1)	125.8(2)
H(32A)-C(32)-H(32B)	109.4	C(41)-O(5)-Mg(1)	128.3(2)
O(3)-C(33)-C(32)	105.0(3)	O(5)-C(38)-C(39)	107.7(3)
O(3)-C(33)-H(33A)	110.8	O(5)-C(38)-H(38A)	110.2
C(32)-C(33)-H(33A)	110.8	C(39)-C(38)-H(38A)	110.2
O(3)-C(33)-H(33B)	110.8	O(5)-C(38)-H(38B)	110.2
C(32)-C(33)-H(33B)	110.8	C(39)-C(38)-H(38B)	110.2
H(33A)-C(33)-H(33B)	108.8	H(38A)-C(38)-H(38B)	108.5
C(34)-O(4)-C(37)	104.9(3)	C(38)-C(39)-C(40)	104.4(3)
C(34)-O(4)-Mg(1)	125.0(2)	C(38)-C(39)-H(39A)	110.9
C(37)-O(4)-Mg(1)	129.4(2)	C(40)-C(39)-H(39A)	110.9
O(4)-C(34)-C(35)	103.7(3)	C(38)-C(39)-H(39B)	110.9
O(4)-C(34)-H(34A)	111.0	C(40)-C(39)-H(39B)	110.9

H(39A)-C(39)-H(39B)	108.9	C(44)-C(45)-H(45A)	110.8
C(41)-C(40)-C(39)	104.4(3)	O(6)-C(45)-H(45B)	110.8
C(41)-C(40)-H(40A)	110.9	C(44)-C(45)-H(45B)	110.8
C(39)-C(40)-H(40A)	110.9	H(45A)-C(45)-H(45B)	108.8
C(41)-C(40)-H(40B)	110.9	O(7)-Mg(2)-O(8)	89.01(11)
C(39)-C(40)-H(40B)	110.9	O(7)-Mg(2)-O(10)	178.9(7)
H(40A)-C(40)-H(40B)	108.9	O(8)-Mg(2)-O(10)	91.6(4)
O(5)-C(41)-C(40)	103.6(3)	O(7)-Mg(2)-O(10')	169.5(4)
O(5)-C(41)-H(41A)	111.0	O(8)-Mg(2)-O(10')	82.4(5)
C(40)-C(41)-H(41A)	111.0	O(7)-Mg(2)-O(12)	91.83(11)
O(5)-C(41)-H(41B)	111.0	O(8)-Mg(2)-O(12)	93.53(11)
C(40)-C(41)-H(41B)	111.0	O(10)-Mg(2)-O(12)	89.1(6)
H(41A)-C(41)-H(41B)	109.0	O(10')-Mg(2)-O(12)	94.6(7)
C(45)-O(6)-C(42)	109.7(3)	O(7)-Mg(2)-O(11')	92.66(11)
C(45)-O(6)-Mg(1)	121.0(2)	O(8)-Mg(2)-O(11')	177.97(12)
C(42)-O(6)-Mg(1)	127.1(2)	O(10')-Mg(2)-O(11')	95.8(5)
O(6)-C(42)-C(43)	106.0(3)	O(12)-Mg(2)-O(11')	87.57(11)
O(6)-C(42)-H(42A)	110.5	O(7)-Mg(2)-O(11)	92.66(11)
C(43)-C(42)-H(42A)	110.5	O(8)-Mg(2)-O(11)	177.97(12)
O(6)-C(42)-H(42B)	110.5	O(10)-Mg(2)-O(11)	86.7(4)
C(43)-C(42)-H(42B)	110.5	O(12)-Mg(2)-O(11)	87.57(11)
H(42A)-C(42)-H(42B)	108.7	O(7)-Mg(2)-O(9)	90.09(12)
C(44)-C(43)-C(42)	103.3(3)	O(8)-Mg(2)-O(9)	91.62(11)
C(44)-C(43)-H(43A)	111.1	O(10)-Mg(2)-O(9)	88.9(7)
C(42)-C(43)-H(43A)	111.1	O(10')-Mg(2)-O(9)	84.2(8)
C(44)-C(43)-H(43B)	111.1	O(12)-Mg(2)-O(9)	174.54(11)
C(42)-C(43)-H(43B)	111.1	O(11')-Mg(2)-O(9)	87.23(11)
H(43A)-C(43)-H(43B)	109.1	O(11)-Mg(2)-O(9)	87.23(11)
C(43)-C(44)-C(45)	103.4(3)	C(47)-O(7)-Mg(2)	126.4(2)
C(43)-C(44)-H(44A)	111.1	C(49)-O(8)-Mg(2)	127.1(2)
C(45)-C(44)-H(44A)	111.1	C(47)-C(46)-H(46A)	109.5
C(43)-C(44)-H(44B)	111.1	C(47)-C(46)-H(46B)	109.5
C(45)-C(44)-H(44B)	111.1	H(46A)-C(46)-H(46B)	109.5
H(44A)-C(44)-H(44B)	109.0	C(47)-C(46)-H(46C)	109.5
O(6)-C(45)-C(44)	104.9(3)	H(46A)-C(46)-H(46C)	109.5
O(6)-C(45)-H(45A)	110.8	H(46B)-C(46)-H(46C)	109.5

O(7)-C(47)-C(48)	124.5(3)	O(9)-C(54)-C(53)	105.4(4)
O(7)-C(47)-C(46)	116.5(3)	O(9)-C(54)-H(54A)	110.7
C(48)-C(47)-C(46)	118.9(3)	C(53)-C(54)-H(54A)	110.7
C(49)-C(48)-C(47)	125.7(3)	O(9)-C(54)-H(54B)	110.7
C(49)-C(48)-H(48)	117.2	C(53)-C(54)-H(54B)	110.7
C(47)-C(48)-H(48)	117.2	H(54A)-C(54)-H(54B)	108.8
O(8)-C(49)-C(48)	124.5(3)	C(58)-O(10)-C(55)	110.1(7)
O(8)-C(49)-C(50)	116.3(3)	C(58)-O(10)-Mg(2)	128.8(7)
C(48)-C(49)-C(50)	119.2(3)	C(55)-O(10)-Mg(2)	121.0(6)
C(49)-C(50)-H(50A)	109.5	O(10)-C(55)-C(56)	103.6(8)
C(49)-C(50)-H(50B)	109.5	O(10)-C(55)-H(55A)	111.0
H(50A)-C(50)-H(50B)	109.5	C(56)-C(55)-H(55A)	111.0
C(49)-C(50)-H(50C)	109.5	O(10)-C(55)-H(55B)	111.0
H(50A)-C(50)-H(50C)	109.5	C(56)-C(55)-H(55B)	111.0
H(50B)-C(50)-H(50C)	109.5	H(55A)-C(55)-H(55B)	109.0
C(54)-O(9)-C(51)	107.4(3)	C(55)-C(56)-C(57)	102.4(7)
C(54)-O(9)-Mg(2)	127.6(2)	C(55)-C(56)-H(56A)	111.3
C(51)-O(9)-Mg(2)	124.4(2)	C(57)-C(56)-H(56A)	111.3
O(9)-C(51)-C(52)	105.9(3)	C(55)-C(56)-H(56B)	111.3
O(9)-C(51)-H(51A)	110.6	C(57)-C(56)-H(56B)	111.3
C(52)-C(51)-H(51A)	110.6	H(56A)-C(56)-H(56B)	109.2
O(9)-C(51)-H(51B)	110.6	C(56)-C(57)-C(58)	102.7(8)
C(52)-C(51)-H(51B)	110.6	C(56)-C(57)-H(57A)	111.2
H(51A)-C(51)-H(51B)	108.7	C(58)-C(57)-H(57A)	111.2
C(53)-C(52)-C(51)	106.9(4)	C(56)-C(57)-H(57B)	111.2
C(53)-C(52)-H(52A)	110.3	C(58)-C(57)-H(57B)	111.2
C(51)-C(52)-H(52A)	110.3	H(57A)-C(57)-H(57B)	109.1
C(53)-C(52)-H(52B)	110.3	O(10)-C(58)-C(57)	104.7(7)
C(51)-C(52)-H(52B)	110.3	O(10)-C(58)-H(58A)	110.8
H(52A)-C(52)-H(52B)	108.6	C(57)-C(58)-H(58A)	110.8
C(54)-C(53)-C(52)	103.7(4)	O(10)-C(58)-H(58B)	110.8
C(54)-C(53)-H(53A)	111.0	C(57)-C(58)-H(58B)	110.8
C(52)-C(53)-H(53A)	111.0	H(58A)-C(58)-H(58B)	108.9
C(54)-C(53)-H(53B)	111.0	C(58')-O(10')-C(55')	109.5(8)
C(52)-C(53)-H(53B)	111.0	C(58')-O(10')-Mg(2)	126.0(8)
H(53A)-C(53)-H(53B)	109.0	C(55')-O(10')-Mg(2)	123.2(8)

O(10')-C(55')-C(56')	105.0(9)	C(61)-C(60)-H(60B)	111.0
O(10')-C(55')-H(55C)	110.8	C(59)-C(60)-H(60B)	111.0
C(56')-C(55')-H(55C)	110.8	H(60A)-C(60)-H(60B)	109.0
O(10')-C(55')-H(55D)	110.8	C(60)-C(61)-C(62)	105.0(9)
C(56')-C(55')-H(55D)	110.8	C(60)-C(61)-H(61A)	110.7
H(55C)-C(55')-H(55D)	108.8	C(62)-C(61)-H(61A)	110.7
C(57')-C(56')-C(55')	101.5(8)	C(60)-C(61)-H(61B)	110.7
C(57')-C(56')-H(56C)	111.5	C(62)-C(61)-H(61B)	110.7
C(55')-C(56')-H(56C)	111.5	H(61A)-C(61)-H(61B)	108.8
C(57')-C(56')-H(56D)	111.5	O(11)-C(62)-C(61)	101.9(9)
C(55')-C(56')-H(56D)	111.5	O(11)-C(62)-H(62A)	111.4
H(56C)-C(56')-H(56D)	109.3	C(61)-C(62)-H(62A)	111.4
C(56')-C(57')-C(58')	103.5(9)	O(11)-C(62)-H(62B)	111.4
C(56')-C(57')-H(57C)	111.1	C(61)-C(62)-H(62B)	111.4
C(58')-C(57')-H(57C)	111.1	H(62A)-C(62)-H(62B)	109.2
C(56')-C(57')-H(57D)	111.1	C(62')-O(11')-C(59')	107.5(15)
C(58')-C(57')-H(57D)	111.1	C(62')-O(11')-Mg(2)	119.7(15)
H(57C)-C(57')-H(57D)	109.0	C(59')-O(11')-Mg(2)	129(2)
O(10')-C(58')-C(57')	104.8(8)	O(11')-C(59')-C(60')	106.0(15)
O(10')-C(58')-H(58C)	110.8	O(11')-C(59')-H(59C)	110.5
C(57')-C(58')-H(58C)	110.8	C(60')-C(59')-H(59C)	110.5
O(10')-C(58')-H(58D)	110.8	O(11')-C(59')-H(59D)	110.5
C(57')-C(58')-H(58D)	110.8	C(60')-C(59')-H(59D)	110.5
H(58C)-C(58')-H(58D)	108.9	H(59C)-C(59')-H(59D)	108.7
C(62)-O(11)-C(59)	105.0(9)	C(61')-C(60')-C(59')	102.3(16)
C(62)-O(11)-Mg(2)	119.9(9)	C(61')-C(60')-H(60C)	111.3
C(59)-O(11)-Mg(2)	131.0(14)	C(59')-C(60')-H(60C)	111.3
O(11)-C(59)-C(60)	105.3(10)	C(61')-C(60')-H(60D)	111.3
O(11)-C(59)-H(59A)	110.7	C(59')-C(60')-H(60D)	111.3
C(60)-C(59)-H(59A)	110.7	H(60C)-C(60')-H(60D)	109.2
O(11)-C(59)-H(59B)	110.7	C(60')-C(61')-C(62')	103.9(12)
C(60)-C(59)-H(59B)	110.7	C(60')-C(61')-H(61C)	111.0
H(59A)-C(59)-H(59B)	108.8	C(62')-C(61')-H(61C)	111.0
C(61)-C(60)-C(59)	103.8(7)	C(60')-C(61')-H(61D)	111.0
C(61)-C(60)-H(60A)	111.0	C(62')-C(61')-H(61D)	111.0
C(59)-C(60)-H(60A)	111.0	H(61C)-C(61')-H(61D)	109.0

O(11')-C(62')-C(61')	108.8(11)	C(68)-C(67)-H(67A)	110.3
O(11')-C(62')-H(62C)	109.9	O(13)-C(67)-H(67B)	110.3
C(61')-C(62')-H(62C)	109.9	C(68)-C(67)-H(67B)	110.3
O(11')-C(62')-H(62D)	109.9	H(67A)-C(67)-H(67B)	108.6
C(61')-C(62')-H(62D)	109.9	C(67)-C(68)-C(69)	100.8(5)
H(62C)-C(62')-H(62D)	108.3	C(67)-C(68)-H(68A)	111.6
C(63)-O(12)-C(66)	108.1(3)	C(69)-C(68)-H(68A)	111.6
C(63)-O(12)-Mg(2)	124.0(2)	C(67)-C(68)-H(68B)	111.6
C(66)-O(12)-Mg(2)	123.2(3)	C(69)-C(68)-H(68B)	111.6
O(12)-C(63)-C(64)	105.3(4)	H(68A)-C(68)-H(68B)	109.4
O(12)-C(63)-H(63A)	110.7	C(70)-C(69)-C(68)	104.5(5)
C(64)-C(63)-H(63A)	110.7	C(70)-C(69)-H(69A)	110.9
O(12)-C(63)-H(63B)	110.7	C(68)-C(69)-H(69A)	110.9
C(64)-C(63)-H(63B)	110.7	C(70)-C(69)-H(69B)	110.9
H(63A)-C(63)-H(63B)	108.8	C(68)-C(69)-H(69B)	110.9
C(65)-C(64)-C(63)	104.8(4)	H(69A)-C(69)-H(69B)	108.9
C(65)-C(64)-H(64A)	110.8	C(69)-C(70)-O(13)	108.3(5)
C(63)-C(64)-H(64A)	110.8	C(69)-C(70)-H(70A)	110.0
C(65)-C(64)-H(64B)	110.8	O(13)-C(70)-H(70A)	110.0
C(63)-C(64)-H(64B)	110.8	C(69)-C(70)-H(70B)	110.0
H(64A)-C(64)-H(64B)	108.9	O(13)-C(70)-H(70B)	110.0
C(66)-C(65)-C(64)	100.4(4)	H(70A)-C(70)-H(70B)	108.4
C(66)-C(65)-H(65A)	111.7	C(74)-O(14)-C(71)	107.6(5)
C(64)-C(65)-H(65A)	111.7	C(72)-C(71)-O(14)	104.9(5)
C(66)-C(65)-H(65B)	111.7	C(72)-C(71)-H(71A)	110.8
C(64)-C(65)-H(65B)	111.7	O(14)-C(71)-H(71A)	110.8
H(65A)-C(65)-H(65B)	109.5	C(72)-C(71)-H(71B)	110.8
O(12)-C(66)-C(65)	105.7(4)	O(14)-C(71)-H(71B)	110.8
O(12)-C(66)-H(66A)	110.6	H(71A)-C(71)-H(71B)	108.8
C(65)-C(66)-H(66A)	110.6	C(71)-C(72)-C(73)	105.3(5)
O(12)-C(66)-H(66B)	110.6	C(71)-C(72)-H(72A)	110.7
C(65)-C(66)-H(66B)	110.6	C(73)-C(72)-H(72A)	110.7
H(66A)-C(66)-H(66B)	108.7	C(71)-C(72)-H(72B)	110.7
C(70)-O(13)-C(67)	104.7(4)	C(73)-C(72)-H(72B)	110.7
O(13)-C(67)-C(68)	107.0(5)	H(72A)-C(72)-H(72B)	108.8
O(13)-C(67)-H(67A)	110.3	C(74)-C(73)-C(72)	106.9(5)

C(74)-C(73)-H(73A)	110.3	C(75)-C(76)-H(76A)	112.0
C(72)-C(73)-H(73A)	110.3	C(77)-C(76)-H(76A)	112.0
C(74)-C(73)-H(73B)	110.3	C(75)-C(76)-H(76B)	112.0
C(72)-C(73)-H(73B)	110.3	C(77)-C(76)-H(76B)	112.0
H(73A)-C(73)-H(73B)	108.6	H(76A)-C(76)-H(76B)	109.7
C(73)-C(74)-O(14)	106.1(6)	C(78)-C(77)-C(76)	102(3)
C(73)-C(74)-H(74A)	110.5	C(78)-C(77)-H(77A)	111.3
O(14)-C(74)-H(74A)	110.5	C(76)-C(77)-H(77A)	111.3
C(73)-C(74)-H(74B)	110.5	C(78)-C(77)-H(77B)	111.3
O(14)-C(74)-H(74B)	110.5	C(76)-C(77)-H(77B)	111.3
H(74A)-C(74)-H(74B)	108.7	H(77A)-C(77)-H(77B)	109.2
C(78)-O(15)-C(75)	94(4)	O(15)-C(78)-C(77)	115(4)
O(15)-C(75)-C(76)	116(4)	O(15)-C(78)-H(78A)	108.6
O(15)-C(75)-H(75A)	108.3	C(77)-C(78)-H(78A)	108.6
C(76)-C(75)-H(75A)	108.3	O(15)-C(78)-H(78B)	108.6
O(15)-C(75)-H(75B)	108.3	C(77)-C(78)-H(78B)	108.6
C(76)-C(75)-H(75B)	108.3	H(78A)-C(78)-H(78B)	107.5
H(75A)-C(75)-H(75B)	107.4		
C(75)-C(76)-C(77)	99(3)		

Symmetry transformations used to generate equivalent atoms:

#1 -x,-y+1,-z #2 -x+2,-y+1,-z+1

3.10 [Mg(acac)(THF)$_4$]$_2$[Fe(4-F-Ph)$_2$(μ-4-F-Ph)]$_2$ · 2 THF (1c)

REFERENCE NUMBER: neism38

CRYSTAL STRUCTURE REPORT

C_{86} H_{118} F_6 Fe_2 Mg_2 O_{14}

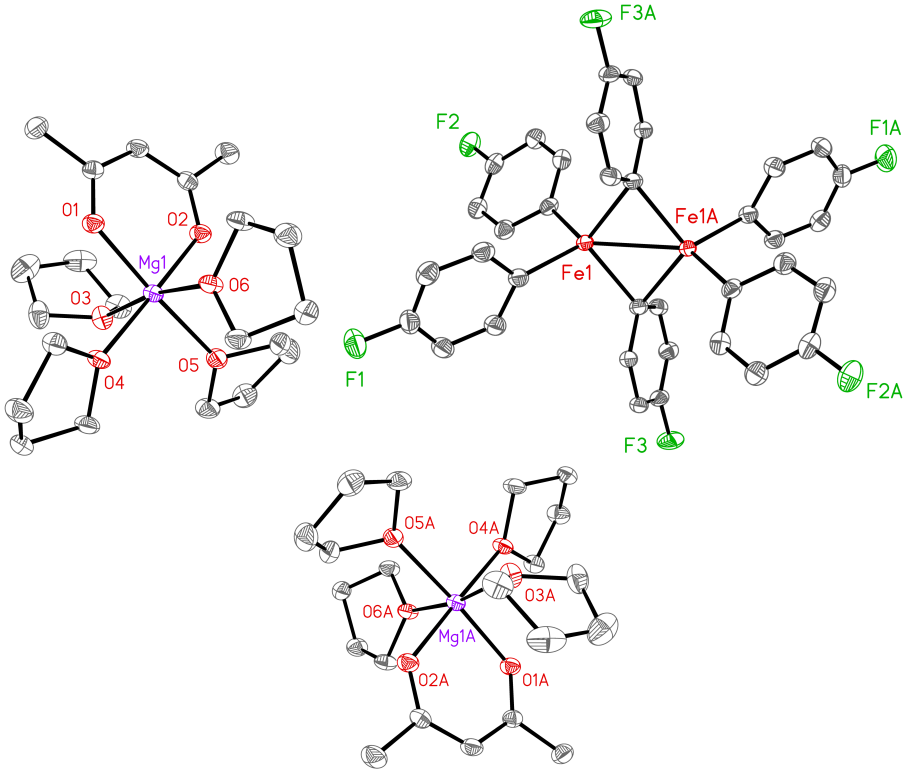
or

[Mg(acac)(THF)$_4$]$_2$[Fe(4-F-Ph)$_2$(μ-4-F-Ph)]$_2$ · 2THF

Report prepared for:

Dr. S. Muñoz, Prof. M. Neidig

January 09, 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 0.40 x 0.16 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 45 seconds and a detector distance of 4.00 cm. A randomly oriented region of reciprocal space was surveyed: six major sections of frames were collected with 0.50º steps in ω at six 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 4055 strong reflections from the actual data collection after integration.[3] See Table 1 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 $R1 = 0.0476$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1326$ (F^2, all data).

Structure description

The structure is one of the ones suggested. The asymmetric unit contains one half of a dianionic iron complex located at a crystallographic inversion center that generates the other half, one cationic magnesium complex in a general position, and one cocrystallized THF solvent molecule in a general position. The cocrystallized THF solvent molecule is modeled as disordered over two positions (0.78:0.22).

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] 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.

Some equations of interest:

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

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

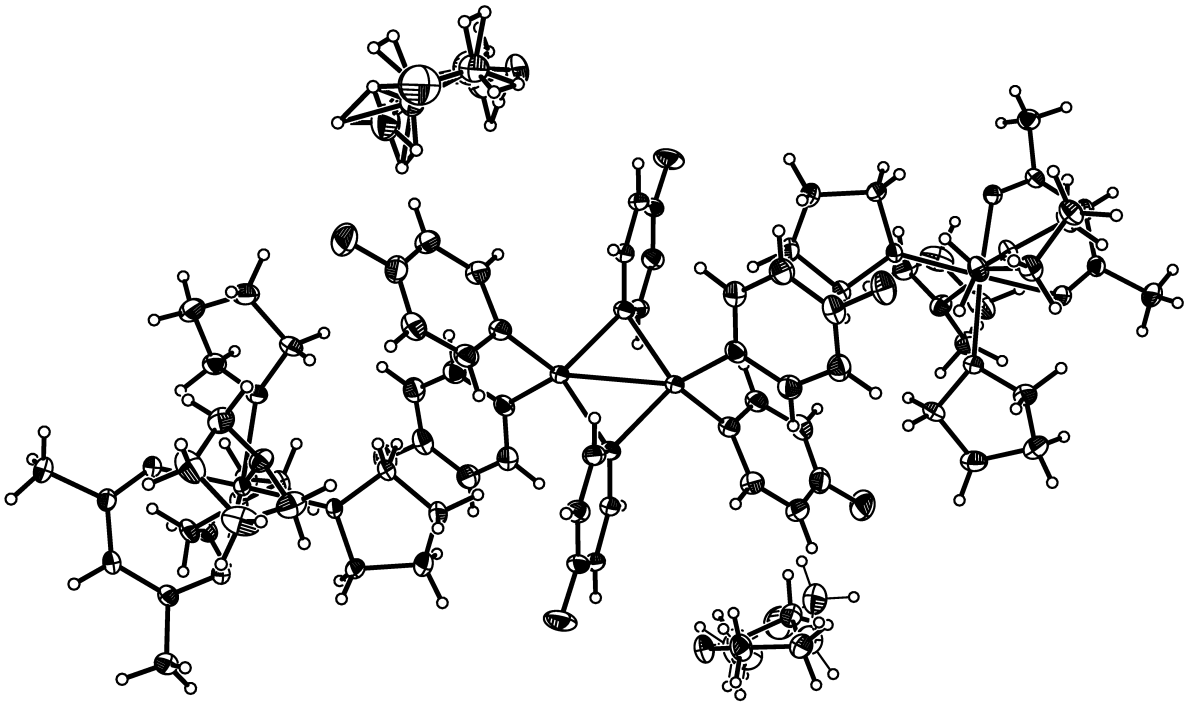
$$wR2 = [\Sigma [w(F_o^2 - F_c^2)^2] \,/\, \Sigma\, [w(F_o^2)^2]]^{1/2}$$

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



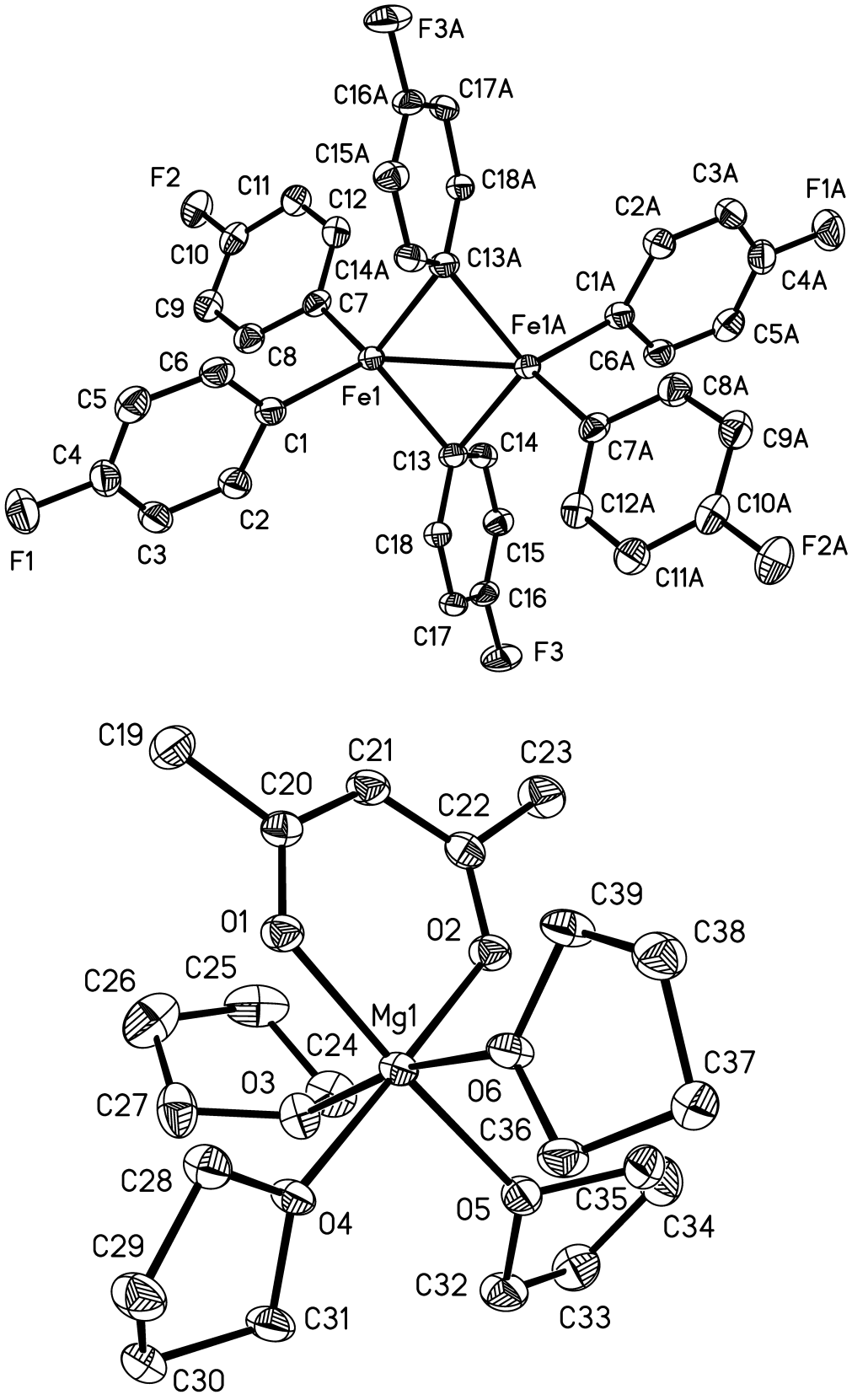

F3A
C16A
C17A
C15A
C18A
F2
C11
C12
C3A
F1A
C2A
C10
C14A
C13A
C1A
C4A
C9
C7
Fe1A
C5A
C8
C6A
C6
C8A
C5
Fe1
C1
C7A
C9A
C4
C13
C14
C12A
C10A
C2
C18
C15
F2A
F1
C3
C11A
C16
C17
F3
C19
C21
C23
C20
C22
C39
O1
O2
C38
C26
C25
Mg1
C24
C37
C27
O3
O6
C36
C28
C35
O4
O5
C34
C29
C32
C33
C31
C30

Table S30. Crystal data and structure refinement for neism38.

Identification code	neism38	
Empirical formula	C86 H118 F6 Fe2 Mg2 O14	
Formula weight	1650.12	
Temperature	100.0(5) K	
Wavelength	0.71073 Å	
Crystal system	triclinic	
Space group	*P*-1	
Unit cell dimensions	a = 12.7545(19) Å	α = 96.604(3)°
	b = 12.8200(19) Å	β = 108.178(3)°
	c = 14.971(2) Å	γ = 113.494(3)°
Volume	2050.9(5) Å^3	
Z	1	
Density (calculated)	1.336 Mg/m^3	
Absorption coefficient	0.445 mm^{-1}	
F(000)	876	
Crystal color, morphology	red-orange, plate	
Crystal size	0.45 x 0.40 x 0.16 mm^3	
Theta range for data collection	1.801 to 35.013°	
Index ranges	$-20 \le h \le 20$, $-20 \le k \le 20$, $-24 \le l \le 24$	
Reflections collected	67503	
Independent reflections	17928 [R(int) = 0.0549]	
Observed reflections	11956	
Completeness to theta = 34.972°	99.3%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.7469 and 0.6872	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	17928 / 52 / 544	
Goodness-of-fit on F^2	1.063	
Final R indices [I>2sigma(I)]	R1 = 0.0476, wR2 = 0.1177	
R indices (all data)	R1 = 0.0831, wR2 = 0.1326	
Largest diff. peak and hole	0.698 and -0.493 e.Å^{-3}	

Table S31. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for neism38. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Fe1	5389(1)	4220(1)	5142(1)	19(1)
F1	8246(1)	2178(1)	3374(1)	48(1)
F2	4994(1)	799(1)	7848(1)	37(1)
F3	246(1)	1699(1)	1631(1)	40(1)
C1	6415(1)	3771(1)	4462(1)	24(1)
C2	5781(2)	2691(1)	3720(1)	29(1)
C3	6366(2)	2143(2)	3344(1)	32(1)
C4	7645(2)	2694(2)	3724(1)	33(1)
C5	8342(2)	3731(2)	4454(1)	32(1)
C6	7718(2)	4256(1)	4812(1)	27(1)
C7	5164(1)	3113(1)	6066(1)	23(1)
C8	5065(1)	1977(1)	5758(1)	28(1)
C9	5009(1)	1194(1)	6342(1)	30(1)
C10	5050(1)	1553(1)	7261(1)	28(1)
C11	5156(1)	2643(1)	7622(1)	29(1)
C12	5228(1)	3402(1)	7018(1)	27(1)
C13	3633(1)	3884(1)	4005(1)	22(1)
C14	2495(1)	3318(1)	4113(1)	23(1)
C15	1360(1)	2584(1)	3335(1)	25(1)
C16	1359(1)	2394(1)	2412(1)	26(1)
C17	2425(1)	2876(1)	2235(1)	25(1)
C18	3541(1)	3611(1)	3028(1)	22(1)
Mg1	9983(1)	2292(1)	7810(1)	17(1)
O1	11437(1)	3349(1)	9086(1)	21(1)
O2	10802(1)	1258(1)	7753(1)	22(1)
C19	13463(1)	4306(1)	10353(1)	26(1)
C20	12453(1)	3298(1)	9465(1)	20(1)
C21	12710(1)	2405(1)	9123(1)	23(1)
C22	11881(1)	1435(1)	8294(1)	20(1)
C23	12274(2)	532(1)	8013(1)	30(1)
O3	10795(1)	3324(1)	6986(1)	24(1)

C24	11335(2)	2982(2)	6362(1)	30(1)
C25	12726(2)	3755(2)	6924(1)	41(1)
C26	12840(2)	4880(2)	7529(1)	43(1)
C27	11518(2)	4600(1)	7370(1)	32(1)
O4	9158(1)	3392(1)	7902(1)	20(1)
C28	9378(1)	4126(1)	8823(1)	22(1)
C29	8620(2)	4784(2)	8550(1)	29(1)
C30	8581(1)	4878(1)	7533(1)	25(1)
C31	8448(1)	3692(1)	7092(1)	25(1)
O5	8546(1)	1114(1)	6456(1)	23(1)
C32	8121(2)	1306(1)	5504(1)	28(1)
C33	8258(2)	441(2)	4810(1)	32(1)
C34	8360(2)	-480(1)	5362(1)	33(1)
C35	8127(1)	-154(1)	6262(1)	27(1)
O6	8894(1)	1292(1)	8483(1)	21(1)
C36	7585(1)	932(1)	8226(1)	23(1)
C37	7108(1)	-193(1)	8532(1)	27(1)
C38	8213(1)	65(1)	9465(1)	27(1)
C39	9318(1)	729(1)	9210(1)	25(1)
O7	5937(1)	2939(1)	212(1)	32(1)
C40	7148(3)	3155(4)	237(3)	33(1)
C41	7616(2)	2550(2)	956(2)	35(1)
C42	6414(5)	1461(4)	768(5)	36(1)
C43	5505(2)	1974(2)	621(2)	33(1)
O7'	8044(8)	2987(7)	619(6)	68(2)
C40'	6865(14)	2988(15)	114(14)	58(4)
C41'	5936(11)	2112(11)	374(9)	62(3)
C42'	6590(20)	1457(17)	880(20)	54(4)
C43'	7932(11)	2386(9)	1351(8)	54(2)

Table S32. Bond lengths [Å] and angles [°] for neism38.

Fe(1)-C(1)	2.0918(15)	C(16)-C(17)	1.376(2)
Fe(1)-C(7)	2.0945(14)	C(17)-C(18)	1.394(2)
Fe(1)-C(13)	2.1866(14)	C(17)-H(17)	0.9500
Fe(1)-C(13)#1	2.2302(14)	C(18)-H(18)	0.9500
Fe(1)-Fe(1)#1	2.5847(5)	Mg(1)-O(2)	1.9972(10)
F(1)-C(4)	1.3701(18)	Mg(1)-O(1)	2.0282(11)
F(2)-C(10)	1.3755(17)	Mg(1)-O(6)	2.0827(10)
F(3)-C(16)	1.3755(17)	Mg(1)-O(4)	2.0842(10)
C(1)-C(6)	1.407(2)	Mg(1)-O(3)	2.1031(11)
C(1)-C(2)	1.414(2)	Mg(1)-O(5)	2.1264(11)
C(2)-C(3)	1.397(2)	O(1)-C(20)	1.2729(16)
C(2)-H(2)	0.9500	O(2)-C(22)	1.2708(16)
C(3)-C(4)	1.376(2)	C(19)-C(20)	1.519(2)
C(3)-H(3)	0.9500	C(19)-H(19A)	0.9800
C(4)-C(5)	1.367(2)	C(19)-H(19B)	0.9800
C(5)-C(6)	1.408(2)	C(19)-H(19C)	0.9800
C(5)-H(5)	0.9500	C(20)-C(21)	1.3958(19)
C(6)-H(6)	0.9500	C(21)-C(22)	1.408(2)
C(7)-C(12)	1.398(2)	C(21)-H(21)	0.9500
C(7)-C(8)	1.419(2)	C(22)-C(23)	1.5004(19)
C(8)-C(9)	1.400(2)	C(23)-H(23A)	0.9800
C(8)-H(8)	0.9500	C(23)-H(23B)	0.9800
C(9)-C(10)	1.378(2)	C(23)-H(23C)	0.9800
C(9)-H(9)	0.9500	O(3)-C(24)	1.4458(17)
C(10)-C(11)	1.376(2)	O(3)-C(27)	1.4517(19)
C(11)-C(12)	1.398(2)	C(24)-C(25)	1.521(2)
C(11)-H(11)	0.9500	C(24)-H(24A)	0.9900
C(12)-H(12)	0.9500	C(24)-H(24B)	0.9900
C(13)-C(14)	1.412(2)	C(25)-C(26)	1.540(3)
C(13)-C(18)	1.4199(19)	C(25)-H(25A)	0.9900
C(14)-C(15)	1.396(2)	C(25)-H(25B)	0.9900
C(14)-H(14)	0.9500	C(26)-C(27)	1.509(3)
C(15)-C(16)	1.374(2)	C(26)-H(26A)	0.9900
C(15)-H(15)	0.9500	C(26)-H(26B)	0.9900

C(27)-H(27A)	0.9900	C(38)-C(39)	1.520(2)
C(27)-H(27B)	0.9900	C(38)-H(38A)	0.9900
O(4)-C(28)	1.4550(16)	C(38)-H(38B)	0.9900
O(4)-C(31)	1.4620(16)	C(39)-H(39A)	0.9900
C(28)-C(29)	1.516(2)	C(39)-H(39B)	0.9900
C(28)-H(28A)	0.9900	O(7)-C(43)	1.432(3)
C(28)-H(28B)	0.9900	O(7)-C(40)	1.445(4)
C(29)-C(30)	1.528(2)	C(40)-C(41)	1.509(5)
C(29)-H(29A)	0.9900	C(40)-H(40A)	0.9900
C(29)-H(29B)	0.9900	C(40)-H(40B)	0.9900
C(30)-C(31)	1.507(2)	C(41)-C(42)	1.520(5)
C(30)-H(30A)	0.9900	C(41)-H(41A)	0.9900
C(30)-H(30B)	0.9900	C(41)-H(41B)	0.9900
C(31)-H(31A)	0.9900	C(42)-C(43)	1.519(7)
C(31)-H(31B)	0.9900	C(42)-H(42A)	0.9900
O(5)-C(32)	1.4465(17)	C(42)-H(42B)	0.9900
O(5)-C(35)	1.4541(17)	C(43)-H(43A)	0.9900
C(32)-C(33)	1.530(2)	C(43)-H(43B)	0.9900
C(32)-H(32A)	0.9900	O(7')-C(43')	1.421(11)
C(32)-H(32B)	0.9900	O(7')-C(40')	1.458(14)
C(33)-C(34)	1.541(2)	C(40')-C(41')	1.464(14)
C(33)-H(33A)	0.9900	C(40')-H(40C)	0.9900
C(33)-H(33B)	0.9900	C(40')-H(40D)	0.9900
C(34)-C(35)	1.515(2)	C(41')-C(42')	1.515(17)
C(34)-H(34A)	0.9900	C(41')-H(41C)	0.9900
C(34)-H(34B)	0.9900	C(41')-H(41D)	0.9900
C(35)-H(35A)	0.9900	C(42')-C(43')	1.507(18)
C(35)-H(35B)	0.9900	C(42')-H(42C)	0.9900
O(6)-C(36)	1.4462(16)	C(42')-H(42D)	0.9900
O(6)-C(39)	1.4576(16)	C(43')-H(43C)	0.9900
C(36)-C(37)	1.512(2)	C(43')-H(43D)	0.9900
C(36)-H(36A)	0.9900	C(1)-Fe(1)-C(7)	103.49(6)
C(36)-H(36B)	0.9900	C(1)-Fe(1)-C(13)	108.10(6)
C(37)-C(38)	1.529(2)	C(7)-Fe(1)-C(13)	113.32(5)
C(37)-H(37A)	0.9900	C(1)-Fe(1)-C(13)#1	112.77(6)
C(37)-H(37B)	0.9900	C(7)-Fe(1)-C(13)#1	110.78(5)

C(13)-Fe(1)-C(13)#1	108.38(4)	C(10)-C(11)-H(11)	121.3
C(1)-Fe(1)-Fe(1)#1	126.64(4)	C(12)-C(11)-H(11)	121.3
C(7)-Fe(1)-Fe(1)#1	129.87(4)	C(11)-C(12)-C(7)	124.39(14)
C(13)-Fe(1)-Fe(1)#1	54.97(4)	C(11)-C(12)-H(12)	117.8
C(13)#1-Fe(1)-Fe(1)#1	53.40(4)	C(7)-C(12)-H(12)	117.8
C(6)-C(1)-C(2)	113.61(14)	C(14)-C(13)-C(18)	114.16(12)
C(6)-C(1)-Fe(1)	126.80(11)	C(14)-C(13)-Fe(1)	121.19(10)
C(2)-C(1)-Fe(1)	117.56(11)	C(18)-C(13)-Fe(1)	115.65(10)
C(3)-C(2)-C(1)	124.45(15)	C(14)-C(13)-Fe(1)#1	108.97(10)
C(3)-C(2)-H(2)	117.8	C(18)-C(13)-Fe(1)#1	118.32(10)
C(1)-C(2)-H(2)	117.8	Fe(1)-C(13)-Fe(1)#1	71.63(4)
C(4)-C(3)-C(2)	117.65(15)	C(15)-C(14)-C(13)	123.87(13)
C(4)-C(3)-H(3)	121.2	C(15)-C(14)-H(14)	118.1
C(2)-C(3)-H(3)	121.2	C(13)-C(14)-H(14)	118.1
C(5)-C(4)-F(1)	118.55(16)	C(16)-C(15)-C(14)	117.74(14)
C(5)-C(4)-C(3)	122.34(15)	C(16)-C(15)-H(15)	121.1
F(1)-C(4)-C(3)	119.10(16)	C(14)-C(15)-H(15)	121.1
C(4)-C(5)-C(6)	118.24(15)	C(15)-C(16)-F(3)	118.73(14)
C(4)-C(5)-H(5)	120.9	C(15)-C(16)-C(17)	122.71(13)
C(6)-C(5)-H(5)	120.9	F(3)-C(16)-C(17)	118.56(13)
C(1)-C(6)-C(5)	123.70(15)	C(16)-C(17)-C(18)	118.02(13)
C(1)-C(6)-H(6)	118.1	C(16)-C(17)-H(17)	121.0
C(5)-C(6)-H(6)	118.1	C(18)-C(17)-H(17)	121.0
C(12)-C(7)-C(8)	114.28(13)	C(17)-C(18)-C(13)	123.44(13)
C(12)-C(7)-Fe(1)	127.37(11)	C(17)-C(18)-H(18)	118.3
C(8)-C(7)-Fe(1)	117.99(11)	C(13)-C(18)-H(18)	118.3
C(9)-C(8)-C(7)	123.35(15)	O(2)-Mg(1)-O(1)	88.22(4)
C(9)-C(8)-H(8)	118.3	O(2)-Mg(1)-O(6)	93.62(4)
C(7)-C(8)-H(8)	118.3	O(1)-Mg(1)-O(6)	94.40(4)
C(10)-C(9)-C(8)	117.82(15)	O(2)-Mg(1)-O(4)	178.72(5)
C(10)-C(9)-H(9)	121.1	O(1)-Mg(1)-O(4)	90.52(4)
C(8)-C(9)-H(9)	121.1	O(6)-Mg(1)-O(4)	86.26(4)
F(2)-C(10)-C(11)	118.44(15)	O(2)-Mg(1)-O(3)	94.33(4)
F(2)-C(10)-C(9)	118.94(14)	O(1)-Mg(1)-O(3)	92.45(5)
C(11)-C(10)-C(9)	122.63(14)	O(6)-Mg(1)-O(3)	169.66(4)
C(10)-C(11)-C(12)	117.49(15)	O(4)-Mg(1)-O(3)	85.94(4)

O(2)-Mg(1)-O(5)	87.18(4)	H(24A)-C(24)-H(24B)	109.0
O(1)-Mg(1)-O(5)	175.30(4)	C(24)-C(25)-C(26)	104.61(14)
O(6)-Mg(1)-O(5)	86.86(4)	C(24)-C(25)-H(25A)	110.8
O(4)-Mg(1)-O(5)	94.08(4)	C(26)-C(25)-H(25A)	110.8
O(3)-Mg(1)-O(5)	86.93(4)	C(24)-C(25)-H(25B)	110.8
C(20)-O(1)-Mg(1)	127.49(9)	C(26)-C(25)-H(25B)	110.8
C(22)-O(2)-Mg(1)	129.07(9)	H(25A)-C(25)-H(25B)	108.9
C(20)-C(19)-H(19A)	109.5	C(27)-C(26)-C(25)	104.35(14)
C(20)-C(19)-H(19B)	109.5	C(27)-C(26)-H(26A)	110.9
H(19A)-C(19)-H(19B)	109.5	C(25)-C(26)-H(26A)	110.9
C(20)-C(19)-H(19C)	109.5	C(27)-C(26)-H(26B)	110.9
H(19A)-C(19)-H(19C)	109.5	C(25)-C(26)-H(26B)	110.9
H(19B)-C(19)-H(19C)	109.5	H(26A)-C(26)-H(26B)	108.9
O(1)-C(20)-C(21)	125.36(13)	O(3)-C(27)-C(26)	106.12(14)
O(1)-C(20)-C(19)	116.60(12)	O(3)-C(27)-H(27A)	110.5
C(21)-C(20)-C(19)	118.03(12)	C(26)-C(27)-H(27A)	110.5
C(20)-C(21)-C(22)	124.53(13)	O(3)-C(27)-H(27B)	110.5
C(20)-C(21)-H(21)	117.7	C(26)-C(27)-H(27B)	110.5
C(22)-C(21)-H(21)	117.7	H(27A)-C(27)-H(27B)	108.7
O(2)-C(22)-C(21)	124.56(12)	C(28)-O(4)-C(31)	109.28(10)
O(2)-C(22)-C(23)	116.46(13)	C(28)-O(4)-Mg(1)	123.16(8)
C(21)-C(22)-C(23)	118.98(13)	C(31)-O(4)-Mg(1)	126.95(8)
C(22)-C(23)-H(23A)	109.5	O(4)-C(28)-C(29)	105.48(11)
C(22)-C(23)-H(23B)	109.5	O(4)-C(28)-H(28A)	110.6
H(23A)-C(23)-H(23B)	109.5	C(29)-C(28)-H(28A)	110.6
C(22)-C(23)-H(23C)	109.5	O(4)-C(28)-H(28B)	110.6
H(23A)-C(23)-H(23C)	109.5	C(29)-C(28)-H(28B)	110.6
H(23B)-C(23)-H(23C)	109.5	H(28A)-C(28)-H(28B)	108.8
C(24)-O(3)-C(27)	105.38(11)	C(28)-C(29)-C(30)	103.06(11)
C(24)-O(3)-Mg(1)	125.17(9)	C(28)-C(29)-H(29A)	111.2
C(27)-O(3)-Mg(1)	120.79(9)	C(30)-C(29)-H(29A)	111.2
O(3)-C(24)-C(25)	103.79(13)	C(28)-C(29)-H(29B)	111.2
O(3)-C(24)-H(24A)	111.0	C(30)-C(29)-H(29B)	111.2
C(25)-C(24)-H(24A)	111.0	H(29A)-C(29)-H(29B)	109.1
O(3)-C(24)-H(24B)	111.0	C(31)-C(30)-C(29)	101.53(11)
C(25)-C(24)-H(24B)	111.0	C(31)-C(30)-H(30A)	111.5

C(29)-C(30)-H(30A)	111.5	H(35A)-C(35)-H(35B)	108.9
C(31)-C(30)-H(30B)	111.5	C(36)-O(6)-C(39)	109.74(10)
C(29)-C(30)-H(30B)	111.5	C(36)-O(6)-Mg(1)	126.00(8)
H(30A)-C(30)-H(30B)	109.3	C(39)-O(6)-Mg(1)	123.88(8)
O(4)-C(31)-C(30)	105.46(11)	O(6)-C(36)-C(37)	104.82(11)
O(4)-C(31)-H(31A)	110.6	O(6)-C(36)-H(36A)	110.8
C(30)-C(31)-H(31A)	110.6	C(37)-C(36)-H(36A)	110.8
O(4)-C(31)-H(31B)	110.6	O(6)-C(36)-H(36B)	110.8
C(30)-C(31)-H(31B)	110.6	C(37)-C(36)-H(36B)	110.8
H(31A)-C(31)-H(31B)	108.8	H(36A)-C(36)-H(36B)	108.9
C(32)-O(5)-C(35)	104.88(11)	C(36)-C(37)-C(38)	101.79(11)
C(32)-O(5)-Mg(1)	130.98(9)	C(36)-C(37)-H(37A)	111.4
C(35)-O(5)-Mg(1)	120.77(8)	C(38)-C(37)-H(37A)	111.4
O(5)-C(32)-C(33)	105.89(12)	C(36)-C(37)-H(37B)	111.4
O(5)-C(32)-H(32A)	110.6	C(38)-C(37)-H(37B)	111.4
C(33)-C(32)-H(32A)	110.6	H(37A)-C(37)-H(37B)	109.3
O(5)-C(32)-H(32B)	110.6	C(39)-C(38)-C(37)	102.51(11)
C(33)-C(32)-H(32B)	110.6	C(39)-C(38)-H(38A)	111.3
H(32A)-C(32)-H(32B)	108.7	C(37)-C(38)-H(38A)	111.3
C(32)-C(33)-C(34)	104.09(12)	C(39)-C(38)-H(38B)	111.3
C(32)-C(33)-H(33A)	110.9	C(37)-C(38)-H(38B)	111.3
C(34)-C(33)-H(33A)	110.9	H(38A)-C(38)-H(38B)	109.2
C(32)-C(33)-H(33B)	110.9	O(6)-C(39)-C(38)	105.61(11)
C(34)-C(33)-H(33B)	110.9	O(6)-C(39)-H(39A)	110.6
H(33A)-C(33)-H(33B)	109.0	C(38)-C(39)-H(39A)	110.6
C(35)-C(34)-C(33)	104.52(13)	O(6)-C(39)-H(39B)	110.6
C(35)-C(34)-H(34A)	110.8	C(38)-C(39)-H(39B)	110.6
C(33)-C(34)-H(34A)	110.8	H(39A)-C(39)-H(39B)	108.8
C(35)-C(34)-H(34B)	110.8	C(43)-O(7)-C(40)	108.3(2)
C(33)-C(34)-H(34B)	110.8	O(7)-C(40)-C(41)	106.1(2)
H(34A)-C(34)-H(34B)	108.9	O(7)-C(40)-H(40A)	110.5
O(5)-C(35)-C(34)	104.50(12)	C(41)-C(40)-H(40A)	110.5
O(5)-C(35)-H(35A)	110.9	O(7)-C(40)-H(40B)	110.5
C(34)-C(35)-H(35A)	110.9	C(41)-C(40)-H(40B)	110.5
O(5)-C(35)-H(35B)	110.9	H(40A)-C(40)-H(40B)	108.7
C(34)-C(35)-H(35B)	110.9	C(40)-C(41)-C(42)	101.0(3)

C(40)-C(41)-H(41A)	111.6	O(7')-C(40')-H(40D)	110.3
C(42)-C(41)-H(41A)	111.6	C(41')-C(40')-H(40D)	110.3
C(40)-C(41)-H(41B)	111.6	H(40C)-C(40')-H(40D)	108.6
C(42)-C(41)-H(41B)	111.6	C(40')-C(41')-C(42')	104.2(12)
H(41A)-C(41)-H(41B)	109.4	C(40')-C(41')-H(41C)	110.9
C(43)-C(42)-C(41)	101.4(3)	C(42')-C(41')-H(41C)	110.9
C(43)-C(42)-H(42A)	111.5	C(40')-C(41')-H(41D)	110.9
C(41)-C(42)-H(42A)	111.5	C(42')-C(41')-H(41D)	110.9
C(43)-C(42)-H(42B)	111.5	H(41C)-C(41')-H(41D)	108.9
C(41)-C(42)-H(42B)	111.5	C(43')-C(42')-C(41')	102.9(11)
H(42A)-C(42)-H(42B)	109.3	C(43')-C(42')-H(42C)	111.2
O(7)-C(43)-C(42)	106.9(3)	C(41')-C(42')-H(42C)	111.2
O(7)-C(43)-H(43A)	110.4	C(43')-C(42')-H(42D)	111.2
C(42)-C(43)-H(43A)	110.4	C(41')-C(42')-H(42D)	111.2
O(7)-C(43)-H(43B)	110.4	H(42C)-C(42')-H(42D)	109.1
C(42)-C(43)-H(43B)	110.4	O(7')-C(43')-C(42')	102.0(11)
H(43A)-C(43)-H(43B)	108.6	O(7')-C(43')-H(43C)	111.4
C(43')-O(7')-C(40')	108.6(9)	C(42')-C(43')-H(43C)	111.4
O(7')-C(40')-C(41')	106.9(10)	O(7')-C(43')-H(43D)	111.4
O(7')-C(40')-H(40C)	110.3	C(42')-C(43')-H(43D)	111.4
C(41')-C(40')-H(40C)	110.3	H(43C)-C(43')-H(43D)	109.2

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y+1,-z+