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Supporting Information

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for

Structure and Mössbauer Spectrum of a (μ-1,2-Peroxo)bis(μ-carboxylato)diiron(III) Model for the Peroxo Intermediate in the MMO Hydroxylase Reaction Cycle‡

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Experimental

Materials, Methods, and Instrumentation. Chemicals were obtained from commercial suppliers and used as received unless stated otherwise. Air-sensitive materials were handled either in a Vacuum Atmospheres glove box under nitrogen or by using standard Schlenk techniques under argon. Solvents used in the glove box were purified by standard methods. The compound K[HB(pz')$_3$],[1] pz' = 3,5-bis(isopropyl)pyrazolyl, was synthesized according to the literature. Elemental analyses were performed by either Microlytics or Desert Analytics. ^{1}H-NMR spectra were obtained on a Bruker AC 250 spectrometer and the chemical shifts were referenced to an internal standard of tetramethylsilane. Electronic spectra were recorded on a Hewlett-Packard 8451 diode array spectrometer by using an immersion dewar equipped with a quartz window or on a Varian Cary spectrometer. Infrared spectra were obtained on a BioRad SPC 3200 FTIR instrument. Resonance Raman spectra were obtained by using a Coherent Innova 90 argon ion laser with an

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excitation wavelength of 647.1 nm and 50 mW of power. A 0.6-m single monochromator (1200 grooves/mm grating), with an entrance slit of 400 μm, and a liquid nitrogen cooled-CCD detector (Princeton Instruments, Inc., #LNCCD-512TK) were used in a standard backscattering configuration. A holographic notch filter (Kaiser Optical Systems) was used to attenuate Rayleigh scattering. The spectra of **2b** in toluene solution were obtained at -78°C by using an immersion dewar equipped with quartz window. A total of 100 scans each with a 3.2 s exposure time were collected for each sample. Raman shifts were calibrated with DMF as a standard. The data were processed by using CSMA software (Princeton Instruments, Inc. Version 2.4A) on a Gateway 2000 computer. Mössbauer spectra were obtained with a conventional constant acceleration transmission Mössbauer spectrometer. The source was ^{57}Co in Rh matrix maintained at room temperature. A thin iron foil was used for velocity scale calibration. Toluene solutions of **2b** were frozen in liquid nitrogen and quickly introduced into the cryostat, where they were immediately further cooled to liquid helium temperatures. Data were collected at 4.2 K.

Fe{HB(pz')$_3$}Br (6). In the dry box a mixture of K[HB(pz')$_3$] (2.01 g, 4.0 mmol) and $FeBr_2$ (880 mg, 4.0 mmol) in THF (8 mL) was stirred for 3 h and filtered. The filter cake was dissolved in dichloromethane (5 mL) and undissolved KBr was filtered off. The filtrate was allowed to evaporate slowly to yield a light pink crystalline solid (1.70 g; 71 %). The identity of the product was confirmed by CCA (data not provided). IR (KBr, cm^{-1}) 2965, 2929, 2867, 2552 (B-H), 1584, 1425, 1397, 1381, 1363, 1301, 1167, 1071, 902, 816, 792, 752, 718, 655, 518. Anal. Calcd for $C_{27}H_{46}N_6BFeBr$: C, 53.94; H, 7.71; N, 13.98. Found: C, 54.01; H, 7.56; N, 14.07.

[Fe{HB(pz')$_3$}(O$_2$CPh)] (1a). To a solution of $FeBr_2$ (216 mg, 1.00 mmol) in THF (20 mL) was added K[HB(pz')$_3$] (516 mg, 1.02 mmol). The mixture was stirred for 1 h before NaOBz (145 mg, 1.01 mmol) was added. The mixture was stirred for an additional 4 h and filtered. The filtrate was evaporated to dryness and the residue

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was taken up with pentane. The pentane solution was filtered and the filtrate was allowed to evaporate slowly to a produce light yellow crystalline solid (554 mg, 86%). IR (KBr, cm^{-1}) 2967, 2928, 2868, 2540 (B-H), 1603 (Ph), 1536 (COO) 1418 (COO) 1381, 1366 1264, 1174, 1136, 1054, 1006, 897, 791, 761, 710, 689, 657, 517, 458, 432.

[Fe{HB(pz')$_3$}(O$_2$CCH$_2$Ph)] (2a). A mixture of **6** (301 mg, 0.50 mmol) and NaO_2CCH_2Ph (84 mg; 0.50 mmol) in THF (4 mL) was stirred for 3 h. The resulting milky mixture was filtered and the filtrate was evaporated to dryness. The residue was taken up with pentane (3 mL) and filtered. The filtrate was allowed to evaporate slowly to produce colorless crystalline solid (280 mg, 85%). UV-vis (toluene; nm) 372 (ε = 120 cm^{-1} M^{-1}). IR (KBr, cm^{-1}) 2966, 2930, 2868, 2542 (B-H), 1562, 1535, 1472, 1381, 1366, 1301, 1175, 1031, 791, 758, 731, 696, 658. Anal. Calcd for $C_{35}H_{53}N_6O_2BFe$: C, 64.03; H, 8.14; N, 12.80. Found: C, 64.00; H, 8.12; N, 12.85.

[Fe{HB(pz')$_3$}(O$_2$C(*p*-tol)] (3a). The compound was prepared as a light yellow crystalline solid (yield 80 - 85%) by the methods reported for **1a** or **2a** IR (KBr, cm^{-1}) 2961, 2929, 2868, 2532 (B-H), 1612, 1592, 1536, 1473, 1425, 1364, 1299, 1171, 1134, 1110, 1048, 864, 818, 790, 765, 755, 715, 654, 630, 430. Anal. Calcd for $C_{35}H_{53}N_6O_2BFe$: C, 64.03; H, 8.14; N, 12.80. Found: C, 63.97; H, 8.20; N, 12.81.

[Fe{HB(pz')$_3$}(O$_2$CBiph)], Biph = 4-biphenyl (4a). Compound **4a** was prepared by the method described for **2a**. Light yellow/green crystals were obtained (yield 80%). IR (KBr, cm^{-1}) 2966, 2930, 2868, 2542 (B-H), 1562, 1535, 1472, 1381, 1366, 1301, 1175, 1031, 791, 758, 731, 696, 658. Anal. Calcd for $C_{40}H_{55}N_6O_2BFe$: C, 66.86; H, 7.71; N, 11.70. Found: C, 66.44; H, 7.65; N, 11.63.

[Fe{HB(pz')$_3$}(O$_2$CNaph)], Naph = 2-naphthyl (5a). The compound **5a** was prepared as a light yellow/green crystalline solid (yield 84%) by the method reported for **2a**. Anal. Calcd for $C_{38}H_{53}N_6O_2BFe$: C, 65.91; H, 7.71; N, 12.14. Found: C, 64.35; H, 7.72; N, 11.93.

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X-ray Crystallography. Many attempts were made to crystallize the peroxo complexes $[Fe_2(\mu\text{-}\bar{O}_2)(\mu\text{-}O_2CR)_2\{HB(pz')_3\}_2]$ (R = phenyl (**1b**); -CH_2Ph (**2b**); *p*-tol (**3b**); 4-biphenyl (**4b**); 2-naphthyl (**5b**). In a typical experiment the precursor **1a** - **5a** was dissolved in pentane (~1.5 mL) in the glove box. The solution was divided into six small tubes (0.6 x 3.5 cm), each of which was then placed in a vial (3 x 5 cm) containing 2 mL of either diisopropyl ether or *tert*-butyl methyl ether (or 1:1 mixture of the ethers/pentane). These vapor diffusion set-ups were sealed with septa, taken out of the box, and allowed to cool in a dry ice/acetone bath. Dry dioxygen was introduced to the crystallization set-ups through the septa for ~5 min. During this time the solutions of the Fe(II) complexes turned deep blue-green, indicating formation of the peroxo complexes $[Fe_2(\mu\text{-}O_2)(\mu\text{-}O_2CR)_2\{HB(pz')_3\}_2]$.[2] The vapor diffusion set-ups were then placed in a -80°C freezer. Crystal formation was observed in 3 - 4 days in most cases. Stacked-thin-plate crystals were typically obtained for **1b** and **3b**, whereas clusters of small needle-shape crystals were obtained for **4b** and **5b**. Preliminary X-ray photographs of these crystals confirmed their crystallinity, but they diffracted too weakly or they were not single crystals suitable for X-ray work. The crystallization set-ups for **2b**, however, provided large block-shaped crystals with a typical dimension of 0.3 x 0.3 x 0.3 mm, which turned out to be acceptable for X-ray work. The thermal instability of the peroxo crystals requires careful handling either under the cold N_2 stream or on a dry ice block.

Crystals of **2b**, suitable for X-ray diffraction, were grown from pentane/isopropyl ether as described above. On a block of dry ice, a crystal **2b** was immersed in Paratone N and mounted rapidly on a glass fiber. The crystal was transferred immediately to a Siemens SMART/CCD three circle (χ fixed at 54.65°) diffractometer equipped with a cold stream of N_2 gas and a graphite-monochromated Mo K_α radiation source. Twenty five frames of two-dimensional diffraction images were collected and processed to deduce an initial unit cell and

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orientation matrix. Profiles of some reflections showed fine structure and broad peaks. Data collection was performed at -150°C with an X-ray power of 2.2 Kw. A total of 1321 frames of two-dimensional diffraction images were collected, each of which was measured for 30 sec. Crystal data, collection parameters, and structure solution and refinement parameters for **2b** are provided in Table S1.

The raw data frames were processed to produce conventional intensity data by the program SAINT.[3] An initial background was determined from the first 12° of data. Integration was performed with constant spot sizes of 1.75° in the detector plane and 0.9° in omega. The intensity data were corrected for Lorentz and polarization effects but not for absorption. The data were filtered to eliminate severe outliers from the final reflection file, excluding 10.29% of the total reflections collected.

All aspects of the solution and refinement were handled by SHELXTL version 5.0.[4] The structure was solved by direct methods and standard difference Fourier techniques. Two independent (μ-peroxo)diiron(III) molecules were found in the asymmetric unit, the structures of which are almost mirror images of one another. There are also five pentane molecules, an isopropyl ether and a methylene chloride in the asymmetric unit. The origin of the methylene chloride is unclear, but the pentane solvent used in this experiment may have been inadvertently contaminated with a small amount of this compound. One pentane molecule is well-ordered but the other lattice solvents are severely disordered and partially occupied. Refinement was carried out on F^2 for all reflections except for those with either very negative F^2 or reflections flagged for potential systematic errors. Final non-hydrogen atom atomic positional and equivalent isotropic thermal parameters, final anisotropic temperature factors, hydrogen positional parameters, and bond distances and angles for **2b** are provided in Tables S2-S5, respectively.

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Table S1. Summary of Crystal Data, Data Collection Parameters, and Structure Refinement for $2[Fe_2(\mu\text{-}O_2)(\mu\text{-}O_2CCH_2Ph)_2\{HB(pz')_3\}_2]\cdot 2.5\ C_5H_{12}\cdot 0.5C_6H_{14}O\text{-}\cdot 0.5CH_2Cl_2$ (**2b**).

Crystal Data	
Empirical Formula	$C_{160}H_{230}B_4ClFe_4N_{24}O_{12.5}$
Color; Habit	Dark green; block
Crystal Size (mm)	0.30 x 0.30 x 0.30
Crystal System	Monoclinic
Space Group	$P2_1/n$
a =	17.222 (4) Å
b =	24.206 (4) Å
c =	45.449 (10) Å
β =	96.198 (11)°
Volume	18836 (7) $Å^3$
Z	4
Formula Weight	1507.89
Density(calc.)	1.065 mg/m^3
Absorption Coefficient	0.372 mm^{-1}
F(000)	6444
Data Collection	
Diffractometer Used	Siemens SMART/CCD
Radiation	Mo K_α (λ = 0.71073 Å)
Temperature, °C	-150
2θ Range	$2° \le 2\theta \le 45°$
Scan Speed	1.2 deg/min in ω
Index Ranges	$-19 \le h \le 18,\ -23 \le k \le 26,\ -39 \le l \le 50$

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Table S1 (continued). Summary of Crystal Data, Data Collection Parameters, and Structure Refinement for **2b**.

Total Reflections	58263
Total Independent Reflections	23921 (R_{int} = 8.48 %)
Reflections (I > 2σ(I))	17972
Solution and Refinement	
System used	Siemens SHELXTL, version 5
Solution	Direct Methods
Refinement method	Full-Matrix Least-Squares on F^2
Weighting scheme	$w^{-1} = \sigma^2(F_o^2)+(0.0599(P))^2 + 277.5855(P)$, where $P = [\max(F_o^2, 0) + 2(F_c^2)]/3$
Final R indices (I>2σ(I))	R_1 = 14.18 %, where $R1 = \Sigma\|\|F_o\| - \|F_c\|\|/\Sigma\|F_o\|$; wR_2 = 30.23%, where $wR2 = \left[\Sigma\left[w\left(F_o^2 - F_c^2\right)^2\right]/\Sigma\left[w\left(F_o^2\right)^2\right]\right]^{1/2}$
R Indices (all data)	R_1 = 17.66 %, wR_2 = 32.98 %
Goodness-of-fit	1.204, where GOF = $[\Sigma(w((F_o^2)-(F_c^2))^2)/(n-p)]^{1/2}$, and n and p denote the number of data and parameters.
Data/restraints/parameters	23205/1138/1801; (I>2σ(I)), 17972/1138/1801
maximum and minimum difference peaks	2.586 , -0.808 e/Å^{-3} near the disordered solvent molecules
Largest and mean Δ/σ	0.230, 0.039

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Table S2. Atom coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for $2[Fe_2(\mu\text{-}O_2)(\mu\text{-}O_2CCH_2Ph)_2\{HB(pz')_3\}_2]\cdot 2.5C_5H_{12}\cdot 0.5C_6H_{14}O\cdot 0.5CH_2Cl_2$ (**2b**). U(eq) is defined as one third of the trace of the orthoganlized Uij tensor.

atom	x	y	z	U(eq)
Fe(1)	1028(1)	3356(1)	1314(1)	19(1)
Fe(2)	-337(1)	4694(1)	1208(1)	19(1)
B(11)	2120(7)	2268(5)	1253(3)	24(3)
B(12)	-1415(7)	5794(5)	1241(3)	24(3)
O(11)	-56(4)	3497(3)	1329(1)	20(2)
O(12)	-512(4)	3929(3)	1193(1)	22(2)
O(171)	1092(4)	3913(3)	982(2)	25(2)
O(172)	310(4)	4638(3)	854(1)	21(2)
O(181)	1298(4)	3863(3)	1671(1)	24(2)
O(182)	558(4)	4614(3)	1541(1)	25(2)
N(111)	888(4)	2690(3)	985(2)	20(2)
N(112)	1493(5)	2311(3)	984(2)	22(2)
N(121)	2313(5)	3304(3)	1306(2)	24(2)
N(122)	2633(5)	2791(3)	1271(2)	25(2)
N(131)	1174(5)	2663(3)	1608(2)	22(2)
N(132)	1669(5)	2239(3)	1539(2)	21(2)
N(141)	-1325(4)	4951(3)	901(2)	23(2)
N(142)	-1679(4)	5465(3)	956(2)	24(2)
N(151)	-1143(4)	4889(3)	1525(2)	18(2)
N(152)	-1501(4)	5408(3)	1509(2)	24(2)
N(161)	7(4)	5572(3)	1215(2)	19(2)

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Table S2 (continued). Atom coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for **2b**.

atom	x	y	z	U(eq)
N(162)	-577(5)	5974(3)	1238(2)	27(2)
C(111)	-709(6)	2795(5)	366(2)	33(3)
C(112)	-989(6)	2651(5)	895(2)	32(3)
C(113)	-410(5)	2875(4)	691(2)	23(2)
C(114)	369(6)	2570(4)	752(2)	26(2)
C(115)	639(6)	2123(4)	594(2)	27(2)
C(116)	1343(6)	1967(4)	748(2)	23(2)
C(117)	1891(6)	1520(5)	667(2)	30(2)
C(118)	1452(7)	997(5)	556(3)	47(3)
C(119)	2413(7)	1738(5)	432(3)	47(3)
C(121)	3283(7)	4600(5)	1153(3)	41(3)
C(122)	3080(7)	4436(5)	1689(3)	43(3)
C(123)	2801(6)	4277(4)	1364(2)	28(2)
C(124)	2905(6)	3667(4)	1317(2)	26(2)
C(125)	3605(6)	3387(5)	1283(3)	37(3)
C(126)	3412(6)	2822(5)	1253(3)	33(3)
C(127)	3946(6)	2342(5)	1198(3)	40(3)
C(128)	4262(8)	2075(6)	1480(3)	64(4)
C(129)	4601(7)	2510(6)	1024(3)	51(3)
C(131)	768(7)	3066(5)	2308(2)	39(3)
C(132)	-368(7)	2495(5)	2085(3)	41(3)
C(133)	329(6)	2852(4)	2019(2)	29(2)
C(134)	885(6)	2506(4)	1860(2)	25(2)
C(135)	1175(6)	1979(4)	1943(2)	30(2)

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Table S2 (continued). Atom coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for **2b**.

atom	x	y	z	U(eq)
C(136)	1668(6)	1823(4)	1734(2)	24(2)
C(137)	2096(6)	1281(4)	1709(2)	31(2)
C(138)	1622(6)	908(5)	1487(3)	43(3)
C(139)	2266(8)	1008(5)	2010(3)	50(3)
C(141)	-1246(8)	4283(5)	203(3)	46(3)
C(142)	-2247(7)	3858(5)	497(4)	61(4)
C(143)	-1512(6)	4208(4)	511(2)	28(2)
C(144)	-1679(6)	4766(4)	641(2)	24(2)
C(145)	-2261(6)	5154(5)	529(2)	32(3)
C(146)	-2240(6)	5581(4)	731(2)	29(2)
C(147)	-2755(6)	6083(5)	721(3)	40(3)
C(148)	-3433(7)	5984(6)	913(3)	59(4)
C(149)	-3068(9)	6229(5)	406(3)	61(4)
C(151)	-1682(6)	3644(4)	1683(2)	30(2)
C(152)	-1079(7)	3979(5)	2189(2)	37(3)
C(153)	-1114(6)	4063(4)	1848(2)	27(2)
C(154)	-1340(6)	4649(4)	1769(2)	25(2)
C(155)	-1843(6)	4988(5)	1912(3)	34(3)
C(156)	-1926(6)	5472(5)	1747(2)	31(2)
C(157)	-2374(6)	5989(5)	1809(2)	35(3)
C(158)	-2043(8)	6232(5)	2114(3)	56(4)
C(159)	-3242(7)	5847(6)	1811(3)	57(4)
C(161)	1680(6)	5659(5)	869(2)	33(3)
C(162)	2081(6)	5843(5)	1410(3)	39(3)

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Table S2 (continued). Atom coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for **2b.**

atom	x	y	z	U(eq)
C(163)	1470(5)	5587(4)	1183(2)	22(2)
C(164)	690(6)	5860(4)	1214(2)	25(2)
C(165)	554(5)	6432(4)	1227(2)	24(2)
C(166)	-256(6)	6494(4)	1240(2)	32(2)
C(167)	-719(6)	7016(5)	1274(3)	36(3)
C(168)	-298(7)	7501(5)	1141(3)	50(3)
C(169)	-830(7)	7122(5)	1597(3)	46(3)
C(171)	788(6)	4254(4)	801(2)	21(2)
C(172)	965(7)	4203(5)	478(2)	38(3)
C(173)	1788(7)	4065(5)	446(2)	34(2)
C(174)	2305(8)	4466(6)	351(3)	47(3)
C(175)	3089(8)	4346(7)	325(3)	61(4)
C(176)	3374(8)	3821(7)	384(3)	61(4)
C(177)	2887(8)	3413(6)	480(3)	53(3)
C(178)	2093(7)	3538(5)	503(2)	37(3)
C(181)	968(6)	4317(4)	1726(2)	24(2)
C(182)	1090(7)	4523(5)	2047(2)	33(2)
C(183)	1116(6)	5136(4)	2091(2)	25(2)
C(184)	1791(7)	5388(5)	2204(2)	34(2)
C(185)	1847(8)	5948(6)	2254(2)	47(3)
C(186)	1172(9)	6271(5)	2184(3)	51(3)
C(187)	497(7)	6035(5)	2071(2)	38(3)
C(188)	464(7)	5462(5)	2021(2)	32(2)
Fe(3)	5994(1)	10117(1)	1296(1)	16(1)

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Table S2 (continued). Atom coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for **2b**.

atom	x	y	z	U(eq)
Fe(4)	4626(1)	8782(1)	1163(1)	16(1)
B(21)	7097(6)	11197(5)	1264(2)	20(3)
B(22)	3504(6)	7700(5)	1175(2)	18(2)
O(21)	4922(4)	9973(3)	1297(1)	20(2)
O(22)	4470(3)	9549(3)	1153(1)	17(1)
O(271)	6080(4)	9576(3)	960(1)	21(2)
O(272)	5322(4)	8838(3)	821(1)	22(2)
O(281)	6249(3)	9589(3)	1646(1)	20(2)
O(282)	5480(4)	8864(3)	1510(1)	19(2)
N(211)	7280(4)	10167(3)	1296(2)	18(2)
N(212)	7613(4)	10684(3)	1267(2)	18(2)
N(221)	5894(4)	10799(3)	980(2)	20(2)
N(222)	6476(4)	11191(3)	993(2)	19(2)
N(231)	6154(4)	10794(3)	1607(2)	21(2)
N(232)	6654(4)	11216(3)	1543(2)	22(2)
N(241)	3776(5)	8579(3)	1468(2)	22(2)
N(242)	3386(4)	8078(3)	1434(2)	23(2)
N(251)	4955(5)	7896(3)	1186(2)	22(2)
N(252)	4378(4)	7508(3)	1208(2)	18(2)
N(261)	3668(4)	8527(3)	840(2)	18(2)
N(262)	3334(4)	8019(3)	883(2)	18(2)
C(211)	8222(7)	8890(5)	1104(2)	39(3)
C(212)	8025(7)	9003(5)	1645(2)	38(3)
C(213)	7764(6)	9182(4)	1328(2)	27(2)

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Table S2 (continued). Atom coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for **2b**.

atom	x	y	z	U(eq)
C(214)	7859(6)	9803(4)	1294(2)	24(2)
C(215)	8580(6)	10078(4)	1268(2)	29(2)
C(216)	8397(5)	10625(4)	1245(2)	25(2)
C(217)	8918(6)	11112(5)	1204(3)	35(3)
C(218)	9561(7)	10946(5)	1000(3)	44(3)
C(219)	9268(7)	11367(6)	1491(3)	58(4)
C(221)	4356(6)	10667(4)	334(2)	28(2)
C(222)	3982(6)	10837(4)	851(2)	29(2)
C(223)	4625(5)	10613(4)	664(2)	23(2)
C(224)	5388(5)	10921(4)	742(2)	17(2)
C(225)	5652(6)	11382(4)	594(2)	24(2)
C(226)	6343(5)	11541(4)	757(2)	20(2)
C(227)	6886(6)	12003(4)	698(2)	29(2)
C(228)	6420(8)	12543(5)	636(4)	68(4)
C(229)	7337(8)	11866(6)	441(3)	61(4)
C(231)	4544(6)	10948(4)	2068(3)	34(3)
C(232)	5665(6)	10346(5)	2292(2)	29(2)
C(233)	5256(5)	10590(4)	2004(2)	20(2)
C(234)	5850(5)	10936(4)	1857(2)	20(2)
C(235)	6165(6)	11443(4)	1956(2)	26(2)
C(236)	7134(6)	12139(4)	1747(2)	29(2)
C(237)	6660(6)	11607(4)	1755(2)	24(2)
C(238)	6672(8)	12555(5)	1537(3)	48(3)
C(239)	7312(8)	12369(6)	2056(3)	58(4)

J4915-15

Table S2 (continued). Atom coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for **2b.**

atom	x	y	z	U(eq)
C(241)	3796(7)	9478(5)	2135(2)	35(3)
C(242)	3257(6)	9829(4)	1623(2)	30(2)
C(243)	3788(6)	9402(4)	1794(2)	24(2)
C(244)	3528(5)	8843(4)	1703(2)	22(2)
C(245)	2988(6)	8493(5)	1826(2)	34(3)
C(246)	2917(7)	8029(5)	1655(2)	34(3)
C(247)	2435(7)	7524(5)	1691(3)	41(3)
C(248)	1641(7)	7674(5)	1798(3)	54(4)
C(249)	2868(9)	7127(6)	1891(4)	68(4)
C(251)	7081(6)	7599(5)	1385(2)	31(3)
C(252)	6606(6)	7822(5)	856(2)	35(3)
C(253)	6424(6)	7869(5)	1182(2)	27(2)
C(254)	5643(5)	7607(4)	1217(2)	21(2)
C(255)	5495(6)	7053(4)	1266(2)	32(2)
C(256)	4695(6)	7006(4)	1256(2)	22(2)
C(257)	4211(7)	6489(4)	1285(3)	36(3)
C(258)	4020(8)	6217(5)	978(3)	59(4)
C(259)	4628(9)	6081(6)	1502(4)	85(6)
C(261)	2780(6)	9615(5)	419(3)	38(3)
C(262)	3858(7)	9199(5)	151(2)	32(3)
C(263)	3538(6)	9266(4)	452(2)	20(2)
C(264)	3345(6)	8711(4)	576(2)	21(2)
C(265)	2828(6)	8329(4)	447(2)	21(2)
C(266)	2814(5)	7890(4)	640(2)	22(2)

J4915-16

Table S2 (continued). Atom coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for **2b**.

atom	x	y	z	U(eq)
C(267)	2314(6)	7378(4)	623(2)	31(2)
C(268)	1549(7)	7476(6)	763(4)	61(4)
C(269)	2155(10)	7184(6)	301(3)	74(5)
C(271)	5775(5)	9239(4)	773(2)	19(2)
C(272)	5976(5)	9284(4)	458(2)	20(2)
C(273)	6805(5)	9480(4)	416(2)	21(2)
C(274)	7310(6)	9133(5)	293(2)	36(3)
C(275)	8072(7)	9309(5)	260(3)	44(3)
C(276)	8326(7)	9829(5)	345(3)	42(3)
C(277)	7803(6)	10187(5)	463(3)	36(3)
C(278)	7055(6)	10013(4)	503(2)	28(2)
C(281)	5914(5)	9135(4)	1699(2)	21(2)
C(282)	5989(6)	8955(4)	2024(2)	27(2)
C(283)	5998(6)	8328(4)	2064(2)	27(2)
C(284)	5327(7)	8023(4)	1999(2)	31(2)
C(285)	5334(9)	7450(5)	2043(3)	47(3)
C(286)	6025(11)	7196(6)	2163(3)	63(4)
C(287)	6671(10)	7512(6)	2225(3)	63(4)
C(288)	6674(8)	8072(6)	2180(3)	47(3)
C(301)	9671(8)	5784(5)	363(3)	52(3)
C(302)	9734(10)	6354(7)	310(4)	77(5)
C(303)	9064(10)	6700(7)	368(4)	82(5)
C(304)	9084(10)	7330(7)	290(4)	85(5)
C(305)	8396(11)	7645(8)	398(4)	98(6)

J4915-17

Table S2 (continued). Atom coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for **2b**.

atom	x	y	z	U(eq)
C(401)	4207(20)	7160(16)	-2656(8)	95(12)
C(402)	4812(28)	7045(22)	-2374(11)	171(22)
C(403)	5741(16)	7242(12)	-2367(6)	64(8)
C(404)	6743(11)	7612(8)	-2215(4)	24(5)
C(405)	6389(27)	7027(19)	-2132(10)	125(16)
C(406)	7069(14)	6994(10)	-2122(5)	124(8)
C(407)	4578(20)	7518(15)	-2614(8)	86(11)
C(501)	10441(19)	11022(13)	2606(7)	74(9)
C(502)	10166(30)	10554(22)	2379(12)	172(23)
C(503)	10678(27)	9978(16)	2338(9)	133(17)
C(504)	11266(27)	9324(19)	2235(11)	156(20)
C(505)	10628(40)	9671(26)	2036(12)	249(38)
C(506)	10613(32)	9216(20)	1960(10)	164(22)
C(507)	10758(24)	10667(16)	2677(8)	107(13)
C(601)	1004(26)	9144(20)	1106(10)	125(16)
C(602)	716(29)	9331(21)	860(11)	132(17)
C(603)	735(14)	9033(10)	612(5)	42(6)
C(604)	503(23)	9246(16)	322(8)	94(12)
C(605)	455(22)	8903(16)	99(8)	96(12)
C(701)	5776(31)	4024(22)	-196(11)	120(16)
C(707)	6240(51)	3823(37)	-218(19)	224(37)
C(702)	5524(25)	3287(18)	-114(9)	106(13)
O(703)	5275(14)	2862(10)	-326(5)	89(7)
C(704)	5197(12)	2285(9)	-261(5)	28(5)

J4915-18

Table S2 (continued). Atom coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2 x 10^3$) for **2b.**

atom	x	y	z	U(eq)
C(705)	4319(14)	2386(10)	-10(5)	38(6)
C(706)	4707(19)	2084(14)	-91(7)	72(9)
C(800)	4056(36)	5786(26)	-1467(15)	174(23)
C(801)	4163(59)	5645(44)	-1846(25)	262(46)
C(802)	4261(65)	5920(48)	-2021(27)	265(49)
C(803)	3665(58)	5939(41)	-2286(22)	275(43)
C(804)	4041(36)	5787(25)	-1058(14)	157(21)
C(805)	3415(33)	5575(23)	-1220(12)	144(19)
C(806)	4309(26)	5731(19)	-796(11)	115(14)
C(1)	7944(11)	10440(8)	2090(4)	22(5)
Cl(1)	7957(4)	9918(3)	2338(2)	62(2)
Cl(2)	8367(6)	11034(4)	2205(2)	97(3)

J4915-19

Table S3. Anisotropic displacement parameters ($Å^2$ x10^3) for 2[$Fe_2(\mu\text{-}O_2)(\mu\text{-}O_2CCH_2Ph)_2\{HB(\bar{p}z')_3\}_2$]·2.5$C_5H_{12}$·0.5$C_6H_{14}$O·0.5$CH_2Cl_2$ (**2b**). The anisotropic displacement factor exponent takes the form: $-2\pi^2(h^2a^{*2}U_{11} + \ldots + 2hka^*b^*U_{12})$.

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Fe(1)	19(1)	19(1)	19(1)	-2(1)	2(1)	4(1)
Fe(2)	19(1)	16(1)	22(1)	-2(1)	2(1)	3(1)
O(11)	22(4)	20(4)	20(4)	0(3)	6(3)	6(3)
O(12)	21(4)	15(4)	30(4)	5(3)	4(3)	7(3)
O(171)	20(4)	27(4)	27(4)	13(3)	4(3)	8(3)
O(172)	25(4)	20(4)	20(4)	1(3)	8(3)	6(3)
O(181)	20(4)	24(4)	26(4)	-4(3)	-6(3)	7(3)
O(182)	23(4)	32(4)	20(4)	0(3)	7(3)	1(3)
N(111)	13(4)	27(5)	21(5)	1(4)	5(4)	11(3)
N(112)	21(4)	29(5)	18(4)	2(4)	5(4)	16(4)
N(121)	28(5)	19(4)	25(5)	-1(4)	-1(4)	4(4)
N(122)	19(5)	29(5)	27(5)	-6(4)	4(4)	6(4)
N(131)	32(5)	12(4)	22(5)	-1(4)	6(4)	8(4)
N(132)	26(5)	17(5)	20(5)	-1(4)	3(4)	9(4)
N(141)	20(4)	15(4)	32(5)	0(4)	0(4)	11(4)
N(142)	17(4)	17(4)	37(5)	1(4)	2(4)	12(4)
N(151)	14(4)	15(4)	24(5)	-3(4)	5(4)	8(3)
N(152)	18(4)	22(5)	34(5)	-9(4)	9(4)	9(4)
N(161)	18(4)	19(4)	23(5)	-3(4)	10(4)	4(3)
N(162)	24(5)	20(5)	36(5)	7(4)	2(4)	9(4)
C(111)	30(6)	29(6)	38(5)	1(5)	-3(5)	1(5)
C(112)	12(5)	44(7)	41(6)	2(5)	7(5)	-3(5)

J4915-20

Table S3 (continued). Anisotropic displacement parameters ($Å^2$ x10^3) for **2b.**

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(113)	11(5)	23(6)	33(5)	-3(5)	0(4)	-2(4)
C(114)	17(5)	27(6)	35(6)	2(5)	12(4)	1(4)
C(115)	21(5)	27(6)	31(6)	-4(5)	-4(4)	4(4)
C(116)	24(5)	22(5)	24(6)	2(4)	2(4)	1(4)
C(117)	26(6)	41(6)	22(6)	-8(5)	-1(4)	14(4)
C(118)	40(7)	30(6)	72(9)	-18(6)	6(6)	12(5)
C(119)	48(8)	43(8)	51(8)	1(6)	19(6)	14(6)
C(121)	46(7)	23(6)	57(7)	6(5)	15(6)	1(5)
C(122)	40(7)	42(7)	46(6)	-9(5)	-1(6)	-9(6)
C(123)	25(6)	20(5)	39(6)	0(5)	4(5)	1(4)
C(124)	19(5)	28(5)	28(6)	0(4)	-2(4)	4(4)
C(125)	21(6)	36(5)	55(8)	0(6)	6(5)	4(4)
C(126)	26(6)	29(5)	45(7)	4(5)	2(5)	7(4)
C(127)	14(5)	36(6)	68(8)	-6(6)	1(5)	16(5)
C(128)	50(9)	64(10)	77(9)	20(7)	3(7)	24(7)
C(129)	30(6)	64(9)	60(8)	-10(7)	15(6)	1(6)
C(131)	66(8)	30(7)	21(6)	1(5)	6(5)	4(6)
C(132)	42(6)	40(7)	44(7)	-4(6)	19(5)	5(5)
C(133)	30(6)	28(6)	29(6)	-1(5)	4(4)	10(4)
C(134)	23(5)	23(5)	31(6)	-5(4)	7(4)	1(4)
C(135)	33(6)	30(6)	29(6)	4(5)	12(5)	13(5)
C(136)	21(5)	23(5)	28(6)	0(4)	1(4)	7(4)
C(137)	33(6)	34(6)	24(5)	-1(4)	1(5)	20(5)
C(138)	29(6)	36(7)	63(8)	-11(6)	1(5)	13(5)

JK915-21

Table S3 (continued). Anisotropic displacement parameters ($Å^2$ x10^3) for **2b.**

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(139)	60(8)	54(8)	36(6)	16(6)	9(6)	32(7)
C(141)	67(9)	32(7)	39(6)	1(5)	9(6)	11(6)
C(142)	48(8)	24(7)	115(12)	-23(7)	26(8)	-8(5)
C(143)	22(5)	24(5)	36(6)	-1(5)	-3(5)	8(4)
C(144)	21(5)	19(5)	33(6)	5(4)	2(5)	3(4)
C(145)	26(6)	38(6)	31(6)	-4(5)	-4(5)	14(5)
C(146)	32(6)	25(5)	28(6)	-7(4)	-10(5)	7(4)
C(147)	30(6)	32(6)	53(7)	-4(5)	-12(5)	12(5)
C(148)	35(7)	59(9)	82(9)	-8(8)	4(6)	19(6)
C(149)	79(10)	36(8)	64(7)	-5(6)	-17(7)	43(7)
C(151)	34(6)	18(5)	40(6)	-5(5)	14(5)	-7(5)
C(152)	54(8)	25(6)	35(5)	3(5)	16(5)	9(6)
C(153)	30(6)	21(5)	31(5)	-2(4)	8(5)	-4(4)
C(154)	20(5)	26(5)	30(6)	-1(4)	-1(4)	-5(4)
C(155)	28(6)	33(6)	43(7)	6(5)	17(5)	0(5)
C(156)	27(6)	31(5)	38(6)	-2(4)	15(5)	3(4)
C(157)	35(6)	45(7)	29(6)	1(5)	15(5)	23(5)
C(158)	61(8)	41(8)	65(8)	-23(6)	-1(7)	30(7)
C(159)	38(6)	74(10)	57(9)	3(7)	7(6)	20(6)
C(161)	29(6)	28(6)	42(6)	-9(5)	3(5)	8(5)
C(162)	33(6)	37(7)	44(7)	-9(6)	-12(5)	-1(5)
C(163)	19(5)	11(5)	35(5)	6(4)	2(4)	0(4)
C(164)	23(5)	19(5)	32(6)	12(5)	4(4)	0(4)
C(165)	13(4)	16(5)	37(6)	7(5)	-16(4)	4(4)

JL4915-22

Table S3 (continued). Anisotropic displacement parameters ($Å^2$ x10^3) for **2b.**

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(166)	27(5)	25(5)	42(7)	-11(5)	-4(5)	1(4)
C(167)	24(6)	35(6)	53(7)	-5(5)	17(5)	8(5)
C(168)	43(7)	16(5)	91(10)	4(6)	6(7)	21(5)
C(169)	34(7)	46(8)	56(7)	-25(6)	-6(6)	14(6)
C(171)	25(5)	19(6)	19(5)	1(5)	1(4)	-5(5)
C(172)	45(6)	52(8)	19(5)	2(5)	2(5)	16(5)
C(173)	43(5)	41(6)	18(6)	1(5)	4(5)	7(4)
C(174)	60(6)	52(7)	34(7)	11(6)	22(6)	11(5)
C(175)	54(7)	74(8)	57(9)	14(8)	17(7)	-3(7)
C(176)	39(7)	92(9)	56(9)	6(8)	27(7)	15(6)
C(177)	53(7)	55(7)	53(8)	-4(6)	19(7)	18(6)
C(178)	35(5)	47(6)	29(6)	8(5)	4(5)	2(5)
C(181)	29(6)	28(6)	14(5)	2(4)	5(5)	1(5)
C(182)	34(6)	34(5)	31(6)	-10(5)	5(5)	3(5)
C(183)	35(5)	35(5)	6(5)	-8(4)	4(4)	-5(4)
C(184)	39(6)	47(6)	16(5)	3(5)	5(5)	-15(5)
C(185)	62(7)	57(7)	22(6)	-10(6)	1(6)	-28(6)
C(186)	87(8)	28(6)	41(7)	-8(6)	16(7)	-20(5)
C(187)	59(6)	34(5)	23(6)	-10(5)	11(5)	0(5)
C(188)	41(6)	34(5)	22(6)	-4(5)	6(5)	-2(4)
Fe(3)	13(1)	18(1)	16(1)	-4(1)	1(1)	-1(1)
Fe(4)	14(1)	17(1)	16(1)	-4(1)	1(1)	-2(1)
O(21)	18(3)	21(4)	23(4)	-13(3)	4(3)	4(3)
O(22)	17(3)	17(4)	18(3)	-2(3)	1(3)	-2(3)

J4915-23

Table S3 (continued). Anisotropic displacement parameters ($Å^2$ x10^3) for **2b.**

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O(271)	24(4)	17(4)	22(4)	-6(3)	0(3)	-4(3)
O(272)	21(4)	28(4)	17(4)	-6(3)	4(3)	-3(3)
O(281)	13(3)	18(4)	27(4)	0(3)	1(3)	-3(3)
O(282)	24(4)	19(4)	13(3)	-1(3)	-6(3)	0(3)
N(211)	15(4)	16(4)	22(4)	7(3)	2(3)	-5(4)
N(212)	16(4)	16(4)	21(4)	-3(3)	3(3)	-3(3)
N(221)	17(4)	16(4)	27(5)	-13(4)	-1(4)	-3(3)
N(222)	16(4)	10(4)	29(5)	-1(4)	-3(4)	2(3)
N(231)	18(4)	28(5)	17(5)	1(4)	0(4)	-4(4)
N(232)	16(4)	30(5)	20(4)	-6(4)	1(3)	-8(4)
N(241)	21(4)	21(4)	23(5)	1(4)	-5(4)	-1(4)
N(242)	19(4)	15(4)	35(5)	-3(4)	3(4)	-3(3)
N(251)	25(5)	18(5)	19(4)	-7(4)	-9(4)	-6(4)
N(252)	17(4)	19(5)	19(4)	-2(3)	0(3)	4(3)
N(261)	16(4)	17(4)	23(5)	0(4)	3(4)	1(3)
N(262)	14(4)	21(4)	19(4)	-1(4)	0(3)	5(3)
C(211)	41(7)	42(7)	35(6)	-5(5)	4(5)	13(6)
C(212)	40(7)	38(7)	36(6)	3(5)	2(5)	0(6)
C(213)	24(6)	24(5)	33(6)	-4(5)	2(4)	3(4)
C(214)	25(5)	26(5)	21(5)	-4(4)	1(4)	-1(4)
C(215)	18(5)	28(5)	41(6)	4(5)	3(5)	6(4)
C(216)	12(5)	28(5)	34(6)	6(5)	0(4)	5(4)
C(217)	11(5)	32(6)	62(7)	-4(5)	6(5)	1(4)
C(218)	42(7)	33(7)	59(8)	2(6)	22(6)	-15(6)

J4915-24

Table S3 (continued). Anisotropic displacement parameters ($Å^2$ x10^3) for **2b.**

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(219)	31(7)	79(10)	67(8)	-26(7)	14(6)	-18(7)
C(221)	29(6)	32(6)	22(5)	-2(5)	-6(4)	-6(5)
C(222)	29(5)	20(6)	39(6)	5(5)	9(5)	7(5)
C(223)	21(5)	17(5)	30(5)	-3(4)	-3(4)	-3(4)
C(224)	21(5)	15(5)	14(5)	-4(4)	-1(4)	1(4)
C(225)	25(5)	32(6)	14(5)	9(4)	2(4)	-6(4)
C(226)	21(5)	16(5)	22(5)	-1(4)	2(4)	3(4)
C(227)	28(6)	29(6)	29(6)	-1(5)	-1(4)	-10(4)
C(228)	63(9)	34(7)	107(13)	32(8)	10(8)	0(6)
C(229)	67(9)	71(10)	51(8)	-12(7)	32(7)	-38(7)
C(231)	25(5)	25(6)	55(8)	3(5)	10(5)	3(4)
C(232)	26(6)	34(6)	26(6)	-4(4)	0(4)	4(5)
C(233)	22(5)	21(5)	19(5)	-2(4)	2(4)	5(4)
C(234)	17(5)	23(5)	17(5)	-5(4)	-6(4)	0(4)
C(235)	37(6)	30(6)	11(5)	-5(4)	6(4)	-6(4)
C(236)	22(6)	30(6)	36(6)	-11(4)	9(5)	-6(4)
C(237)	21(5)	27(5)	25(6)	-8(4)	0(4)	-8(4)
C(238)	53(8)	24(6)	65(8)	-5(5)	-1(6)	-6(6)
C(239)	80(10)	53(9)	43(6)	-19(6)	14(6)	-47(8)
C(241)	42(7)	33(7)	29(5)	-7(5)	5(5)	-14(5)
C(242)	42(7)	12(5)	37(6)	-6(4)	12(5)	-5(4)
C(243)	22(5)	20(5)	31(5)	-5(4)	11(4)	-6(4)
C(244)	20(5)	21(5)	25(5)	0(4)	6(4)	-2(4)
C(245)	38(7)	33(6)	34(6)	-9(5)	19(5)	-19(5)

JA915-25

Table S3 (continued). Anisotropic displacement parameters ($Å^2$ x10^3) for **2b.**

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(246)	43(6)	32(5)	29(6)	-8(4)	14(5)	-14(5)
C(247)	63(7)	26(6)	37(7)	-9(5)	18(6)	-19(5)
C(248)	40(6)	42(8)	82(10)	13(7)	11(7)	-25(5)
C(249)	66(9)	41(8)	97(12)	16(7)	11(8)	-10(6)
C(251)	16(5)	34(7)	41(6)	-3(5)	-3(5)	-6(5)
C(252)	31(6)	41(7)	35(5)	-12(5)	8(5)	3(5)
C(253)	18(5)	32(6)	31(5)	-1(5)	5(4)	5(4)
C(254)	18(5)	24(5)	22(5)	-2(4)	-1(4)	-2(4)
C(255)	37(5)	16(5)	43(7)	-1(5)	9(5)	4(4)
C(256)	23(5)	11(5)	32(6)	-7(4)	-2(4)	0(4)
C(257)	35(6)	7(5)	62(7)	3(5)	-7(6)	1(4)
C(258)	71(10)	26(7)	80(8)	-23(6)	3(8)	-12(7)
C(259)	82(11)	43(9)	119(12)	45(8)	-31(10)	-31(8)
C(261)	31(6)	23(6)	61(8)	10(6)	5(5)	3(5)
C(262)	44(7)	31(7)	22(5)	4(5)	5(5)	0(5)
C(263)	20(5)	21(5)	20(5)	2(4)	2(4)	-1(4)
C(264)	23(5)	20(5)	21(5)	-2(4)	1(4)	0(4)
C(265)	25(5)	20(5)	17(5)	-1(4)	-5(4)	0(4)
C(266)	9(5)	24(5)	32(6)	-1(4)	3(4)	0(4)
C(267)	25(5)	27(6)	38(6)	3(5)	-15(5)	-15(5)
C(268)	23(6)	42(8)	117(12)	2(8)	10(7)	-19(6)
C(269)	109(12)	63(10)	42(7)	-3(6)	-26(7)	-45(9)
C(271)	16(5)	22(6)	19(5)	6(5)	1(4)	4(5)
C(272)	20(5)	30(6)	10(5)	-11(4)	-2(4)	-5(4)

J4915-26

Table S3 (continued). Anisotropic displacement parameters ($Å^2$ x10^3) for **2b.**

atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(273)	18(5)	34(5)	10(5)	2(4)	0(4)	-3(4)
C(274)	32(5)	43(6)	33(6)	-3(5)	8(5)	9(5)
C(275)	35(6)	57(7)	43(7)	-2(6)	19(6)	15(5)
C(276)	28(6)	62(7)	36(7)	5(6)	6(5)	2(5)
C(277)	30(6)	34(6)	45(7)	5(5)	6(5)	-11(4)
C(278)	26(5)	33(5)	25(6)	2(5)	7(4)	-5(4)
C(281)	8(5)	31(6)	24(5)	-2(4)	2(4)	11(5)
C(282)	32(6)	31(5)	19(5)	-9(4)	4(5)	-6(4)
C(283)	43(6)	27(5)	12(5)	-2(4)	1(4)	6(4)
C(284)	41(6)	26(5)	26(6)	3(5)	6(5)	8(4)
C(285)	90(8)	24(5)	29(7)	-5(5)	11(6)	-7(6)
C(286)	136(11)	33(7)	20(7)	8(6)	9(8)	33(6)
C(287)	96(9)	61(7)	29(7)	-7(7)	-3(7)	55(7)
C(288)	46(6)	64(7)	31(7)	-1(6)	-2(5)	20(6)

J4915-27

Table S4. Hydrogen atom coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for $2[Fe_2(\mu\text{-}O_2)(\mu\text{-}O_2CCH_2Ph)_2\{HB(pz')_3\}_2]\cdot 2.5C_5H_{12}\cdot 0.5C_6H_{14}O\cdot 0.5CH_2Cl_2$ (**2b**).

atom	x	y	z	U_{11}
HB1	2448(7)	1931(5)	1237(3)	29
HB2	-1756(7)	6126(5)	1252(3)	29
H(11A)	-325(6)	2942(5)	242(2)	39
H(11B)	-1205(6)	2991(5)	322(2)	39
H(11C)	-788(6)	2401(5)	325(2)	39
H(11D)	-780(6)	2708(5)	1102(2)	39
H(11E)	-1070(6)	2255(5)	858(2)	39
H(11F)	-1488(6)	2846(5)	855(2)	39
H(11G)	-324(5)	3277(4)	732(2)	27
H(11H)	390(6)	1961(4)	419(2)	32
H(11I)	2242(6)	1420(5)	849(2)	36
H(11J)	1827(7)	717(5)	506(3)	57
H(11K)	1094(7)	1086(5)	380(3)	57
H(11L)	1154(7)	854(5)	711(3)	57
H(11M)	2766(7)	1444(5)	380(3)	56
H(11N)	2720(7)	2054(5)	513(3)	56
H(11O)	2080(7)	1854(5)	254(3)	56
H(12A)	3218(7)	4998(5)	1182(3)	50
H(12B)	3100(7)	4505(5)	947(3)	50
H(12C)	3836(7)	4503(5)	1194(3)	50
H(12D)	3008(7)	4834(5)	1716(3)	51
H(12E)	3634(7)	4343(5)	1733(3)	51

J4915-28

Table S4 (continued). Hydrogen atom coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for **2b**.

atom	x	y	z	U_{11}
H(12F)	2775(7)	4232(5)	1823(3)	51
H(12G)	2236(6)	4374(4)	1320(2)	34
H(12H)	4108(6)	3547(5)	1280(3)	45
H(12I)	3625(6)	2060(5)	1079(3)	47
H(12J)	3830(8)	1967(6)	1592(3)	77
H(12K)	4602(8)	2336(6)	1598(3)	77
H(12L)	4564(8)	1747(6)	1438(3)	77
H(12M)	4384(7)	2685(6)	838(3)	61
H(12N)	4902(7)	2183(6)	980(3)	61
H(12O)	4942(7)	2773(6)	1140(3)	61
H(13A)	415(7)	3292(5)	2413(2)	47
H(13B)	1213(7)	3291(5)	2263(2)	47
H(13C)	955(7)	2753(5)	2432(2)	47
H(13D)	-730(7)	2718(5)	2187(3)	49
H(13E)	-181(7)	2183(5)	2210(3)	49
H(13F)	-639(7)	2356(5)	1899(3)	49
H(13G)	139(6)	3171(4)	1891(2)	35
H(13H)	1057(6)	1770(4)	2109(2)	36
H(13I)	2606(6)	1362(4)	1632(2)	37
H(13J)	1899(6)	557(5)	1471(3)	52
H(13K)	1552(6)	1089(5)	1294(3)	52
H(13L)	1110(6)	837(5)	1555(3)	52
H(13M)	2543(8)	659(5)	1988(3)	59

J4915-29

Table S4 (continued). Hydrogen atom coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for **2b**.

atom	x	y	z	U_{11}
H(13N)	1774(8)	934(5)	2092(3)	59
H(13O)	2592(8)	1253(5)	2143(3)	59
H(14A)	-1138(8)	3921(5)	120(3)	55
H(14B)	-770(8)	4509(5)	218(3)	55
H(14C)	-1658(8)	4469(5)	74(3)	55
H(14D)	-2146(7)	3496(5)	413(4)	73
H(14E)	-2669(7)	4042(5)	372(4)	73
H(14F)	-2401(7)	3811(5)	697(4)	73
H(14G)	-1087(6)	4022(4)	642(2)	33
H(14H)	-2597(6)	5124(5)	350(2)	38
H(14I)	-2436(6)	6401(5)	808(3)	48
H(14J)	-3767(7)	6312(6)	906(3)	71
H(14K)	-3219(7)	5911(6)	1118(3)	71
H(14L)	-3741(7)	5665(6)	835(3)	71
H(14M)	-3401(9)	6557(5)	406(3)	74
H(14N)	-3374(9)	5919(5)	317(3)	74
H(14O)	-2630(9)	6305(5)	291(3)	74
H(15A)	-1519(6)	3268(4)	1740(2)	36
H(15B)	-1677(6)	3691(4)	1469(2)	36
H(15C)	-2212(6)	3709(4)	1736(2)	36
H(15D)	-932(7)	3596(5)	2239(2)	45
H(15E)	-1593(7)	4057(5)	2254(2)	45
H(15F)	-691(7)	4231(5)	2290(2)	45

J4915-30

Table S4 (continued). Hydrogen atom coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for **2b**.

atom	x	y	z	U_{11}
H(15G)	-580(6)	3995(4)	1788(2)	32
H(15H)	-2079(6)	4904(5)	2086(3)	41
H(15I)	-2315(6)	6268(5)	1651(2)	43
H(15J)	-2333(8)	6567(5)	2154(3)	68
H(15K)	-1490(8)	6323(5)	2110(3)	68
H(15L)	-2097(8)	5959(5)	2269(3)	68
H(15M)	-3533(7)	6182(6)	1851(3)	68
H(15N)	-3302(7)	5572(6)	1965(3)	68
H(15O)	-3446(7)	5697(6)	1618(3)	68
H(16A)	2185(6)	5482(5)	850(2)	39
H(16B)	1715(6)	6054(5)	824(2)	39
H(16C)	1277(6)	5488(5)	730(2)	39
H(16D)	2590(6)	5672(5)	1394(3)	47
H(16E)	1930(6)	5780(5)	1610(3)	47
H(16F)	2114(6)	6241(5)	1374(3)	47
H(16G)	1428(5)	5183(4)	1225(2)	26
H(16H)	932(5)	6718(4)	1228(2)	28
H(16I)	-1246(6)	6972(5)	1160(3)	44
H(16J)	-593(7)	7842(5)	1162(3)	60
H(16K)	-259(7)	7430(5)	931(3)	60
H(16L)	228(7)	7540(5)	1246(3)	60
H(16M)	-1131(7)	7462(5)	1613(3)	55
H(16N)	-318(7)	7161(5)	1712(3)	55

J4915-31

Table S4 (continued). Hydrogen atom coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **2b**.

atom	x	y	z	U_{11}
H(16O)	-1111(7)	6811(5)	1674(3)	55
H(17A)	624(7)	3914(5)	379(2)	46
H(17B)	835(7)	4557(5)	375(2)	46
H(17C)	2113(8)	4827(6)	303(3)	57
H(17D)	3426(8)	4627(7)	266(3)	73
H(17E)	3902(8)	3736(7)	360(3)	73
H(17F)	3086(8)	3055(6)	529(3)	63
H(17G)	1758(7)	3253(5)	558(2)	44
H(18A)	663(7)	4373(5)	2153(2)	40
H(18B)	1585(7)	4365(5)	2142(2)	40
H(18C)	2244(7)	5167(5)	2250(2)	41
H(18D)	2326(8)	6112(6)	2333(2)	57
H(18E)	1192(9)	6659(5)	2217(3)	61
H(18F)	44(7)	6255(5)	2024(2)	46
H(18G)	-11(7)	5298(5)	1939(2)	38
HB3	7429(6)	11535(5)	1258(2)	24
HB4	3152(6)	7372(5)	1176(2)	21
H(21A)	8165(7)	8489(5)	1124(2)	47
H(21B)	8775(7)	8990(5)	1141(2)	47
H(21C)	8020(7)	9003(5)	903(2)	47
H(21D)	7960(7)	8602(5)	1663(2)	46
H(21E)	7708(7)	9192(5)	1781(2)	46
H(21F)	8576(7)	9099(5)	1696(2)	46

J4915-32

Table S4 (continued). Hydrogen atom coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **2b**.

atom	x	y	z	U_{11}
H(21G)	7198(6)	9088(4)	1281(2)	33
H(21H)	9082(6)	9915(4)	1268(2)	34
H(21I)	8586(6)	11402(5)	1096(3)	42
H(21J)	9898(7)	11265(5)	975(3)	52
H(21K)	9310(7)	10828(5)	807(3)	52
H(21L)	9875(7)	10642(5)	1092(3)	52
H(21M)	9599(7)	11680(6)	1449(3)	70
H(21N)	9584(7)	11091(6)	1607(3)	70
H(21O)	8849(7)	11496(6)	1604(3)	70
H(22A)	3864(6)	10466(4)	287(2)	34
H(22B)	4755(6)	10512(4)	219(2)	34
H(22C)	4278(6)	11058(4)	283(2)	34
H(22D)	3493(6)	10635(4)	799(2)	35
H(22E)	3897(6)	11232(4)	810(2)	35
H(22F)	4153(6)	10785(4)	1062(2)	35
H(22G)	4713(5)	10213(4)	711(2)	27
H(22H)	5407(6)	11550(4)	419(2)	28
H(22I)	7265(6)	12058(4)	878(2)	35
H(22J)	6779(8)	12843(5)	598(4)	82
H(22K)	6149(8)	12637(5)	808(4)	82
H(22L)	6036(8)	12493(5)	462(4)	82
H(22M)	7686(8)	12173(6)	406(3)	73
H(22N)	6971(8)	11805(6)	263(3)	73

J4915-33

Table S4 (continued). Hydrogen atom coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for **2b**.

atom	x	y	z	U_{11}
H(22O)	7645(8)	11530(6)	486(3)	73
H(23A)	4171(6)	10719(4)	2162(3)	41
H(23B)	4290(6)	11099(4)	1882(3)	41
H(23C)	4722(6)	11252(4)	2201(3)	41
H(23D)	5291(6)	10123(5)	2389(2)	35
H(23E)	5862(6)	10646(5)	2424(2)	35
H(23F)	6101(6)	10112(5)	2246(2)	35
H(23G)	5069(5)	10282(4)	1868(2)	24
H(23H)	6059(6)	11636(4)	2129(2)	31
H(23I)	7638(6)	12053(4)	1666(2)	35
H(23J)	6570(8)	12392(5)	1339(3)	58
H(23K)	6979(8)	12894(5)	1526(3)	58
H(23L)	6176(8)	12642(5)	1613(3)	58
H(23M)	7616(8)	12710(6)	2049(3)	70
H(23N)	7613(8)	12097(6)	2181(3)	70
H(23O)	6821(8)	12448(6)	2139(3)	70
H(24A)	3972(7)	9852(5)	2190(2)	42
H(24B)	4154(7)	9208(5)	2238(2)	42
H(24C)	3269(7)	9420(5)	2191(2)	42
H(24D)	3428(6)	10202(4)	1684(2)	36
H(24E)	2717(6)	9774(4)	1666(2)	36
H(24F)	3286(6)	9785(4)	1411(2)	36
H(24G)	4332(6)	9455(4)	1742(2)	28

J4915-34

Table S4 (continued). Hydrogen atom coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **2b**.

atom	x	y	z	U_{11}
H(24H)	2725(6)	8568(5)	1995(2)	41
H(24I)	2329(7)	7345(5)	1493(3)	49
H(24J)	1368(7)	7939(5)	1661(3)	65
H(24K)	1726(7)	7838(5)	1996(3)	65
H(24L)	1323(7)	7339(5)	1806(3)	65
H(24M)	2544(9)	6799(6)	1912(4)	82
H(24N)	3000(9)	7298(6)	2086(4)	82
H(24O)	3348(9)	7018(6)	1809(4)	82
H(25A)	7578(6)	7778(5)	1356(2)	37
H(25B)	7110(6)	7206(5)	1336(2)	37
H(25C)	6976(6)	7641(5)	1591(2)	37
H(25D)	7113(6)	7993(5)	835(2)	42
H(25E)	6198(6)	8012(5)	726(2)	42
H(25F)	6622(6)	7432(5)	799(2)	42
H(25G)	6398(6)	8269(5)	1234(2)	32
H(25H)	5869(6)	6765(4)	1300(2)	38
H(25I)	3709(7)	6599(4)	1361(3)	43
H(25J)	3707(8)	5884(5)	998(3)	71
H(25K)	4507(8)	6117(5)	898(3)	71
H(25L)	3726(8)	6477(5)	844(3)	71
H(25M)	4300(9)	5753(6)	1516(4)	102
H(25N)	4727(9)	6255(6)	1697(4)	102
H(25O)	5125(9)	5973(6)	1432(4)	102

J4915-35

Table S4 (continued). Hydrogen atom coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **2b**.

atom	x	y	z	U_{11}
H(26A)	2889(6)	9979(5)	338(3)	46
H(26B)	2589(6)	9659(5)	613(3)	46
H(26C)	2382(6)	9427(5)	284(3)	46
H(26D)	3980(7)	9564(5)	74(2)	38
H(26E)	3465(7)	9017(5)	12(2)	38
H(26F)	4334(7)	8974(5)	176(2)	38
H(26G)	3939(6)	9454(4)	593(2)	24
H(26H)	2531(6)	8362(4)	259(2)	26
H(26I)	2613(6)	7081(4)	738(2)	38
H(26J)	1236(7)	7137(6)	749(4)	73
H(26K)	1253(7)	7775(6)	658(4)	73
H(26L)	1672(7)	7578(6)	972(4)	73
H(26M)	1829(10)	6851(6)	292(3)	88
H(26N)	2651(10)	7101(6)	223(3)	88
H(26O)	1882(10)	7476(6)	181(3)	88
H(27A)	5608(5)	9194(4)	294(2)	24
H(27B)	7143(6)	8774(5)	230(2)	43
H(27C)	8420(7)	9063(5)	177(3)	53
H(27D)	8845(7)	9943(5)	324(3)	50
H(27E)	7962(6)	10553(5)	517(3)	44
H(27F)	6711(6)	10255(4)	590(2)	33
H(28A)	6477(6)	9110(4)	2126(2)	32
H(28B)	5547(6)	9111(4)	2119(2)	32

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Table S4 (continued). Hydrogen atom coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **2b**.

atom	x	y	z	U_{11}
H(28C)	4857(7)	8202(4)	1923(2)	37
H(28D)	4875(9)	7238(5)	1993(3)	57
H(28E)	6042(11)	6809(6)	2199(3)	75
H(28F)	7143(10)	7338(6)	2304(3)	76
H(28G)	7138(8)	8280(6)	2229(3)	57
H(30A)	10147(8)	5597(5)	317(3)	63
H(30B)	9221(8)	5634(5)	238(3)	63
H(30C)	9599(8)	5723(5)	572(3)	63
H(30D)	9825(10)	6405(7)	100(4)	93
H(30E)	10202(10)	6494(7)	433(4)	93
H(30F)	9000(10)	6669(7)	582(4)	99
H(30G)	8590(10)	6539(7)	258(4)	99
H(30K)	9580(10)	7492(7)	381(4)	102
H(30L)	9068(10)	7372(7)	72(4)	102
H(30H)	8430(11)	8036(8)	345(4)	118
H(30I)	8416(11)	7610(8)	614(4)	118
H(30J)	7904(11)	7491(8)	305(4)	118
H(60A)	936(26)	9414(20)	1261(10)	150
H(60B)	1562(26)	9071(20)	1099(10)	150
H(60C)	738(26)	8799(20)	1148(10)	150
H(60D)	980(29)	9686(21)	829(11)	159
H(60E)	161(29)	9417(21)	878(11)	159
H(60F)	411(14)	8701(10)	634(5)	51

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Table S4 (continued). Hydrogen atom coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for **2b**.

atom	x	y	z	U_{11}
H(60J)	1279(14)	8903(10)	611(5)	51
H(60K)	-15(23)	9422(16)	326(8)	113
H(60L)	876(23)	9541(16)	283(8)	113
H(60G)	293(22)	9105(16)	-85(8)	115
H(60H)	71(22)	8615(16)	127(8)	115
H(60I)	967(22)	8733(16)	85(8)	115
H(1A)	8201(11)	10308(8)	1918(4)	27
H(1B)	7392(11)	10519(8)	2018(4)	27

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Table S5. Bond lengths (Å) and angles (deg) for 2[$Fe_2(\mu\text{-}O_2)(\mu\text{-}O_2CCH_2Ph)_2$-{$HB(pz')_3$}$_2$]·2.5$C_5H_{12}$·0.5$C_6H_{14}O$·0.5$CH_2Cl_2$ (**2b**).

Fe(1)-O(11)	1.905(6)	N(111)-N(112)	1.389(10)
Fe(1)-O(171)	2.037(7)	N(112)-C(116)	1.359(13)
Fe(1)-O(181)	2.050(7)	N(121)-C(124)	1.344(13)
Fe(1)-N(131)	2.143(8)	N(121)-N(122)	1.374(11)
Fe(1)-N(111)	2.192(8)	N(122)-C(126)	1.357(13)
Fe(1)-N(121)	2.222(8)	N(131)-C(134)	1.349(13)
Fe(2)-O(12)	1.876(6)	N(131)-N(132)	1.392(11)
Fe(2)-O(182)	2.050(7)	N(132)-C(136)	1.342(13)
Fe(2)-O(172)	2.058(6)	N(141)-C(144)	1.346(13)
Fe(2)-N(151)	2.159(7)	N(141)-N(142)	1.417(11)
Fe(2)-N(141)	2.171(8)	N(142)-C(146)	1.359(13)
Fe(2)-N(161)	2.205(8)	N(151)-C(154)	1.328(13)
B(11)-N(122)	1.540(14)	N(151)-N(152)	1.398(11)
B(11)-N(112)	1.544(14)	N(152)-C(156)	1.382(13)
B(11)-N(132)	1.587(14)	N(161)-C(164)	1.369(12)
B(12)-N(162)	1.509(14)	N(161)-N(162)	1.411(11)
B(12)-N(142)	1.546(14)	N(162)-C(166)	1.373(13)
B(12)-N(152)	1.555(14)	C(111)-C(113)	1.525(14)
O(11)-O(12)	1.409(9)	C(112)-C(113)	1.530(14)
O(171)-C(171)	1.239(11)	C(113)-C(114)	1.529(13)
O(172)-C(171)	1.281(11)	C(114)-C(115)	1.405(14)
O(181)-C(181)	1.274(12)	C(115)-C(116)	1.384(14)
O(182)-C(181)	1.261(12)	C(116)-C(117)	1.510(14)
N(111)-C(114)	1.342(13)	C(117)-C(118)	1.53(2)

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Table S5 (continued). Bond lengths (Å) and angles (deg) for **2b.**

C(117)-C(119)	1.56(2)	C(151)-C(153)	1.546(14)
C(121)-C(123)	1.55(2)	C(152)-C(153)	1.557(14)
C(122)-C(123)	1.55(2)	C(153)-C(154)	1.506(14)
C(123)-C(124)	1.505(14)	C(154)-C(155)	1.40(2)
C(124)-C(125)	1.405(14)	C(155)-C(156)	1.39(2)
C(125)-C(126)	1.41(2)	C(156)-C(157)	1.51(2)
C(126)-C(127)	1.52(2)	C(157)-C(159)	1.53(2)
C(127)-C(128)	1.49(2)	C(157)-C(158)	1.55(2)
C(127)-C(129)	1.50(2)	C(161)-C(163)	1.519(14)
C(131)-C(133)	1.53(2)	C(162)-C(163)	1.525(14)
C(132)-C(133)	1.54(2)	C(163)-C(164)	1.517(13)
C(133)-C(134)	1.514(14)	C(164)-C(165)	1.407(14)
C(134)-C(135)	1.404(14)	C(165)-C(166)	1.410(14)
C(135)-C(136)	1.392(14)	C(166)-C(167)	1.51(2)
C(136)-C(137)	1.516(14)	C(167)-C(169)	1.52(2)
C(137)-C(139)	1.52(2)	C(167)-C(168)	1.54(2)
C(137)-C(138)	1.52(2)	C(171)-C(172)	1.538(14)
C(141)-C(143)	1.53(2)	C(172)-C(173)	1.48(2)
C(142)-C(143)	1.52(2)	C(173)-C(178)	1.39(2)
C(143)-C(144)	1.513(14)	C(173)-C(174)	1.42(2)
C(144)-C(145)	1.427(14)	C(174)-C(175)	1.40(2)
C(145)-C(146)	1.38(2)	C(175)-C(176)	1.38(2)
C(146)-C(147)	1.50(2)	C(176)-C(177)	1.39(2)
C(147)-C(149)	1.52(2)	C(177)-C(178)	1.41(2)
C(147)-C(148)	1.55(2)	C(181)-C(182)	1.536(14)

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Table S5 (continued). Bond lengths (Å) and angles (deg) for **2b.**

C(182)-C(183)	1.50(2)	O(21)-O(22)	1.406(8)
C(183)-C(184)	1.36(2)	O(271)-C(271)	1.251(11)
C(183)-C(188)	1.38(2)	O(272)-C(271)	1.279(11)
C(184)-C(185)	1.38(2)	O(281)-C(281)	1.277(12)
C(185)-C(186)	1.41(2)	O(282)-C(281)	1.260(11)
C(186)-C(187)	1.35(2)	N(211)-C(214)	1.331(12)
C(187)-C(188)	1.40(2)	N(211)-N(212)	1.388(10)
Fe(3)-O(21)	1.881(6)	N(212)-C(216)	1.373(12)
Fe(3)-O(271)	2.028(6)	N(221)-C(224)	1.346(12)
Fe(3)-O(281)	2.052(6)	N(221)-N(222)	1.377(11)
Fe(3)-N(231)	2.163(8)	N(222)-C(226)	1.367(12)
Fe(3)-N(221)	2.182(8)	N(231)-C(234)	1.346(12)
Fe(3)-N(211)	2.218(8)	N(231)-N(232)	1.386(11)
Fe(4)-O(22)	1.877(6)	N(232)-C(237)	1.352(12)
Fe(4)-O(282)	2.046(6)	N(241)-C(244)	1.352(12)
Fe(4)-O(272)	2.067(6)	N(241)-N(242)	1.388(11)
Fe(4)-N(241)	2.176(8)	N(242)-C(246)	1.361(13)
Fe(4)-N(261)	2.177(8)	N(251)-C(254)	1.370(12)
Fe(4)-N(251)	2.218(8)	N(251)-N(252)	1.379(11)
B(21)-N(212)	1.525(13)	N(252)-C(256)	1.341(12)
B(21)-N(222)	1.543(13)	N(261)-C(264)	1.345(12)
B(21)-N(232)	1.548(13)	N(261)-N(262)	1.380(11)
B(22)-N(242)	1.523(13)	N(262)-C(266)	1.380(12)
B(22)-N(262)	1.538(13)	C(211)-C(213)	1.527(14)
B(22)-N(252)	1.566(13)	C(212)-C(213)	1.53(2)