Supplementary Materials

Undecametallic and Hexadecametallic Ferric Oxo-Hydroxo/Ethoxo Pivalate Clusters

Svetlana G. Baca,*[,a,b] Manfred Speldrich,[a] Jan van Leusen,[a] Arkady Ellern,[c] and Paul Kögerler*[,a,d]

[a]*Institute of Inorganic Chemistry, RWTH Aachen University, D-52074 Aachen, Germany*

[b]*Institute of Applied Physics, Academy of Sciences of Moldova, 2028 Chisinau, Moldova*

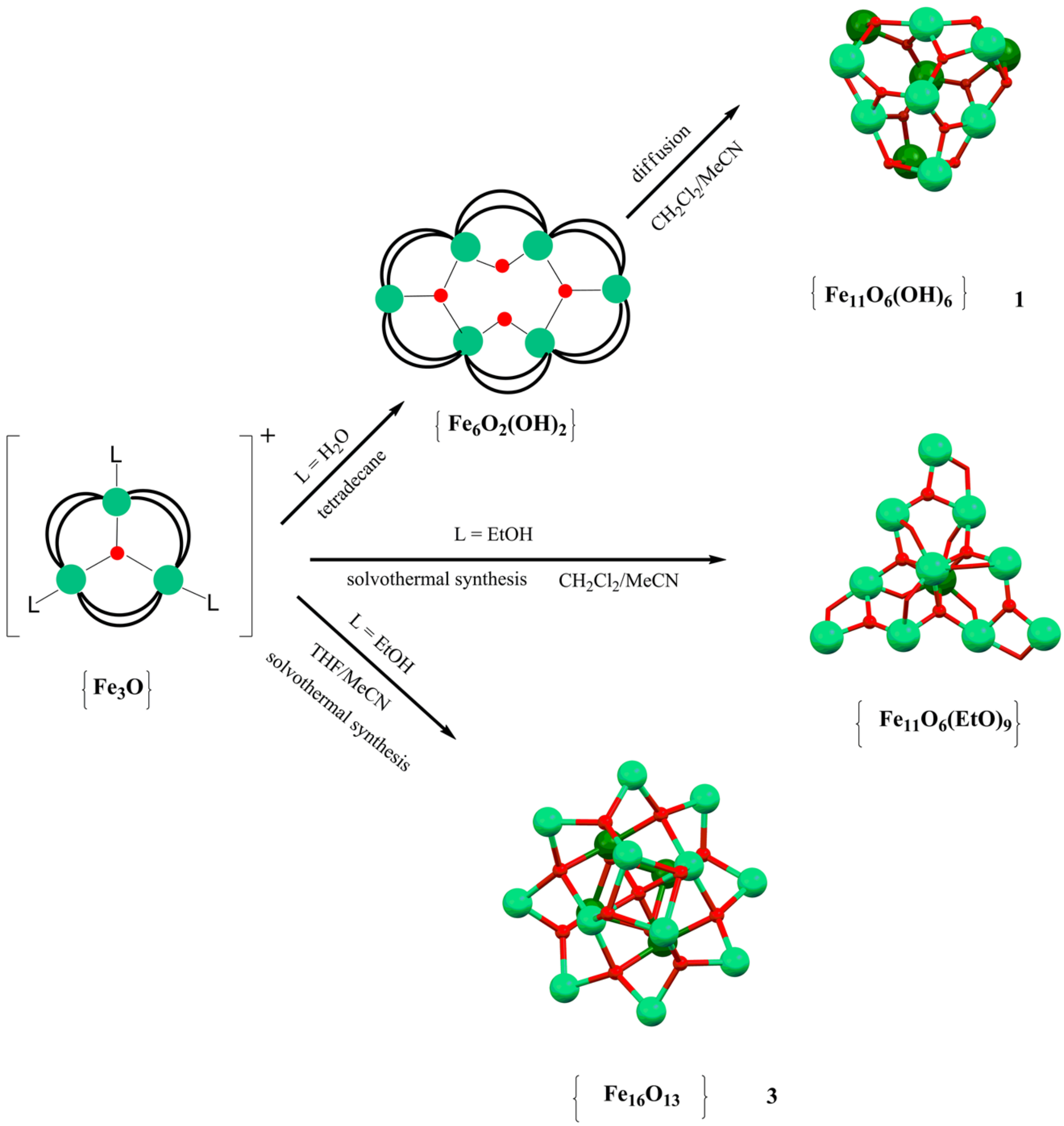
[c]*Ames Laboratory, Iowa state University, Ames, Iowa 50011, United States*

[d] *Peter Grünberg Institute-6, Research Centre Jülich, D-52425 Jülich, Germany*

Corresponding authors:

*(S.G.B.) E-mail: sbaca_md@yahoo.com; Fax: +373-22-725887; Tel: +373-22-738154

(P.K.) E-mail: paul.koegerler@ac.rwth-aachen.de; Fax: +49-241-80-92642; Tel: +49-241-80-93642



Scheme S1. Synthesis of Fe_{11} and Fe_{16} clusters.

Experimental Section

Syntheses

All reactions were carried out under aerobic conditions using commercial grade solvents. $[Fe_3O(O_2CCMe_3)_6(H_2O)_3]O_2CCMe_3\cdot 2Me_3CCO_2H$[1] and $[Fe_6O_2(OH)_2(O_2CCMe_3)_{12}]$[1-2] were synthesized as described elsewhere. $[Fe_3O(O_2CCMe_3)_6(EtOH)_3]NO_3\cdot EtOH$ was prepared by a slightly modified procedure reported in [1] as following: iron(III) nitrate nonahydrate and pivalic acid were heated until the volume of the mixture was reduced up to 1/3 and then ethanol was added. The resulting yellow precipitate was filtered off in 2 days, washed with hexane and dried in air. Its identity has been confirmed by a single-crystal X-ray diffraction analysis (the brown crystals of $[Fe_3O(O_2CCMe_3)_6(EtOH)_3]NO_3\cdot EtOH$ (**4**) were prepared by slow evaporation of its solution in dichloromethane: monoclinic, space group $P2_1/n$, a = 13.661(2), b = 17.953(3), c = 21.562(4) Å, β=100.919(2)°, V = 5192.5(15) Å^3, T = 173(2) K, Z = 4, 60093 reflections collected, R_1 = 0.0389, wR_2 = 0.0845 using 10746 reflections with $I > 2\sigma(I)$). Elemental analysis (%) calcd. for $C_{38}H_{78}Fe_3NO_{20}$ (FW = 1036.56): C 44.03, H 7.58, N 1.35; found: C 43.60, H 6.99, N 1.31. IR (KBr): ν = 3439 (br, m), 2962 (m), 2929 (m), 2871 (sh), 1581 (m), 1525 (vs), 1485 (s), 1424 (vs), 1379 (m), 1363 (m), 1229 (s), 1159 (w), 1097 (m), 1048 (m), 891 (m), 789 (w), 608 (m), 441 (m). ESI-MS: m/z 934 $[Fe_3O(Piv)_6(thf)_2]^+$ (72%); m/z 862 $[Fe_3O(Piv)_6(thf)]^+$ (100%).

Synthesis of $[Fe_{11}O_6(OH)_6(O_2CCMe_3)_{15}]$ (1): $[Fe_6O_2(OH)_2(O_2CCMe_3)_{12}]$ (0.240 g) was refluxed in CH_2Cl_2 (5 mL) for 10 min and the resulting mixture was filtered. The light brown needle-shaped single crystals of **1** suitable for X-ray measurements were obtained by slow diffusion of acetonitrile (5 mL) into the filtrate at room temperature. The crystals were filtered off, washed with MeCN and dried in air. (Yield: 0.152 g; 80% based on Fe). Elemental analysis (%) calcd. for $C_{75}H_{141}Fe_{11}O_{42}$ (FW = 2329.23): C 38.67, H 6.10; found: C 38.53, H 6.12. IR (KBr): ν = 3606 (m), 2962 (m), 2931 (m), 2873 (sh), 1583 (vs), 1563 (sh), 1524 (sh), 1485 (vs), 1459 (sh), 1422 (vs), 1379 (m), 1358 (m), 1229 (s), 1032 (w), 938 (w), 898 (w), 822 (m), 783 (m), 699 (sh), 650 (m), 608 (m), 515 (w), 442 (m).

Synthesis of $[Fe_{11}O_6(EtO)_9(O_2CCMe_3)_{12}]\cdot 1.5CH_2Cl_2\cdot MeCN$ ($2\cdot 1.5CH_2Cl_2\cdot MeCN$): $[Fe_3O(O_2CCMe_3)_6(EtOH)_3]NO_3\cdot EtOH$ (0.208 g), CH_2Cl_2 (2 mL) and MeCN (3 mL) were placed in a sealed PTFE-lined steel autoclave and heated for 4 hours at 120°C and then allowed to slowly cool to room temperature over 48 hours. Light brown needle-shaped crystals of **2·1.5CH$_2$Cl$_2$·MeCN,** which are crystallized with a small amount of hexagonal dark brown crystals of **3**, were removed by filtration. Additional amount of **2** without any crystals **3** was received after standing the filtrate at r.t. for several days. The crystals were filtered off, washed with MeCN and dried in air. To remove the co-precipitated dark brown crystals of **3**, the first precipitate was dissolve in cold CH$_2$Cl$_2$, filtered and the resulting solution was carefully layered with MeCN. (Yield: 0.080 g; 59% based on Fe). Elemental analysis (%) calcd. for $C_{81.5}H_{159}Cl_3Fe_{11}NO_{39}$ (FW = 2497.77): C 39.19, H 6.42, N 0.56; found: C 38.47, H 6.35, N 0.00. IR (KBr): ν = 3430 (br, w), 2963 (m), 2930 (m), 2871 (sh), 1553 (s), 1485 (vs), 1428 (vs), 1379 (m), 1363 (m), 1231 (s), 1102 (w), 1056 (w), 1033 (w), 937 (w), 899 (w), 787 (w), 726 (sh), 604 (m), 529 (m), 441 (m).

Synthesis of [Fe$_{16}$O$_{13}$(EtO)$_6$(O$_2$CCMe$_3$)$_{16}$] (3):

3·0.25thf. [Fe$_3$O(O$_2$CCMe$_3$)$_6$(EtOH)$_3$]NO$_3$·EtOH (0.208 g), thf (2.5 mL) and MeCN (2.5 mL) were placed in a sealed PTFE-lined steel autoclave and heated for 4 hours at 120°C and then allowed to slowly cool to room temperature over 48 hours. Dark brown crystals of **3·0.25thf** suitable for X-ray analysis were removed by filtration, washed with MeCN and dried in air. The additional amount of **3·0.25thf** were crystalized by slow evaporation of the filtrate. (Yield: 0.051 g; 45% based on Fe). Elemental analysis (%) calcd. for $C_{93}H_{176}Fe_{16}O_{51.25}$ (FW = 3007.94): C 37.19, H 5.91; found: C 37.51, H 5.72. IR (KBr): ν = 3425 (br, w), 2963 (m), 2930 (m), 2870 (sh), 1553 (s), 1527 (s), 1485 (vs), 1429 (vs), 1414 (sh), 1379 (m), 1360 (m), 1230 (s), 1105 (m), 1065 (m), 1040 (m), 938 (w), 882 (m), 791 (m), 725 (m), 694 (m), 642 (m), 599 (m), 579 (sh), 509 (m), 442 (m).

3·3thf. [Fe$_3$O(O$_2$CCMe$_3$)$_6$(EtOH)$_3$]NO$_3$·EtOH (0.500 g) and thf (5 mL) were placed in a sealed PTFE-lined steel autoclave and heated for 4 hours at 120°C and then allowed to slowly cool to room temperature over 48 hours. Dark brown plate-shaped single crystal of **3·3thf** was used for single crystal X-ray diffraction analysis and the remaining crystalline product was collected by filtration, washed with small amounts of hexane and dried in air. The additional amount of **3·3thf** were crystalized by slow evaporation of the filtrate. (Yield: 0.075 g; 25% based on Fe). Elemental analysis (%) calc. for $C_{104}H_{198}Fe_{16}O_{54}$ (FW = 3206.22): C 38.96, H 6.22; found: C 37.39, H 6.42. IR (KBr): ν = 3443 (br, w), 2962 (m), 2930 (m), 2871 (sh), 1553 (s), 1526 (s), 1485 (vs), 1429 (vs), 1415 (sh), 1379 (m), 1360 (m), 1230 (s), 1105 (w), 1066 (w), 1039 (m), 882 (m), 791 (w), 725 (m), 693 (w), 644 (w), 599 (m), 579 (sh), 509 (m), 437(m).

Physical measurements

IR spectra were recorded on a Perkin-Elmer Spectrum One spectrometer using KBr pellets in the region 4000 – 400 cm^{-1}. TGA/DTA measurements were carried out with a Mettler Toledo TGA/SDTA 851 in dry N_2 (60 ml min^{-1}) at a heating rate of 10 K min^{-1} (Figs. S4-S8). Electrospray ionization mass spectrometry was carried out with a LTQ Orbitrap XL mass spectrometer in tetrahydrofuran. Magnetic susceptibility data were recorded using a Quantum Design MPMS-5XL SQUID magnetometer as a function of field (0.1 to 5.0 Tesla) and temperature (2.0 to 290.0 K). Experimental data were corrected for sample holder (PTFE capsules) and diamagnetic contributions calculated from tabulated values.

X-ray crystallography

Single-crystal X-ray diffraction experiments were carried out on a Bruker diffractometer with APEX II CCD detector using graphite monochromated MoKa radiation with a detector distance of 50.6 mm. Full-sphere data collection with exposures of 30 s per frame were made with ω scans in the range 0–180° at φ = 0, 120, and 240°. A semi-empirical absorption correction was based on a fit of a spherical harmonic function to the empirical transmission surface as sampled by multiple equivalent measurements[3] using SADABS software.[4] The edge of a detector corresponds to a resolution of 0.71 Å, however, the datasets have been truncated to obtain the statistically relevant resolution. The positions of metal atoms were found by the direct methods. The remaining atoms were located in an alternating series of least-squares cycles and difference Fourier maps. All non-hydrogen atoms were refined in full-matrix anisotropic approximation using the SHELX suite of programs.[5] All hydrogen atoms were placed in the structure factor calculation at idealized positions and were allowed to ride on the neighboring atoms with relative isotropic displacement coefficients. The carboxylate ligands are highly disordered; therefore various restrains were applied to obtain reasonable geometrical parameters and thermal displacement coefficients. Crystal structures have huge voids accessible for solvents but not all of them were objectively located on a difference Fourier map and refined. The attempt to apply SQUEEZE[6] routine disigned to treat electron density of disordered and diffused solvents did not result in any improvement;

therefore the original datasets were used for the final refinements. All calculations were performed using BRUKER APEX II software suite[6] and DIAMOND[7] software suite was used for graphical representation. The summary of the data collection and the crystallographic parameters of compounds **1–3** are listed in Table S1 and selected bond distances and angles in Table S2. CCDC - 875371 (**1**), 875369 (**2·1.5CH_2Cl_2·MeCN**), 875370 (**3·0.25thf**), 875373 (**3·3thf**) and 875372 (**4**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB21EZ, UK; fax: (+44)1223-336-033; or deposit@ccdc.cam.ac.uk).

[1] N. V. Gerbeleu, A. S. Batsanov, G. A. Timko, Iu. T. Struchkov, K. M. Indrichan, G. A. Popovich, *Dokl. Akad. Nauk* SSSR (Russ.) **1987**, *293*, 364-367.

[2] O. S. Manole, A.S. Batsanov, Iu.T. Struchkov, G.A. Timko, L.D. Synzheryan, N.V. Gerbeleu, *Koord. Khim. (Russ.)* **1994**, *20*, 231-237.

[3] R. H. Blessing, *Acta Cryst*. **1995**, *A51*, 33-38.

[4] G. M. Sheldrick, *Acta Cryst.* **2008**, *A64*, 112-122.

[5] A. L. Spek, *J. Appl. Cryst*. **2003**, *36*, 7-13

[6] Bruker AXS Inc., Madison, Wisconsin, USA. **2008.**

[7] Diamond Version 3.2h 1997-2012 Crystal Impact GbR, Bonn, Germany

Table S1. Crystal Data and Details of Structural Determinations for **1–3**

	1	**2·1.5CH$_2$Cl$_2$·MeCN**	**3·0.25thf**	**3·3thf**
Empirical formula	$C_{75}H_{141}Fe_{11}O_{42}$	$C_{81.5}H_{159}Cl_3Fe_{11}NO_{39}$	$C_{93}H_{176}Fe_{16}O_{51.25}$	$C_{104}H_{198}Fe_{16}O_{54}$
M_r (g mol^{-1})	2329.23	2497.80	3007.94	3206.22
T (K)	173(2)	173(2)	173(2)	100(2)
Crystal system	monoclinic	triclinic	monoclinic	monoclinic
Space group	$P2_1/c$	P-1	$P2_1/c$	$C2/c$
a (Å)	31.488(2)	13.2026(18)	32.725(4)	35.891(7)
b (Å)	26.215(2)	16.084(2)	28.973(4)	30.379(6)
c (Å)	31.169(2)	27.889(4)	29.299(4)	31.253(6)
α (°)	90	95.898(2)	90	90
β (°)	117.430(2)	90.145(2)	90.576(1)	124.42(3)
γ (°)	90	96.779(2)	90	90
V (Å^3)	22837(3)	5849.1(14)	27778.2(6)	28110.1(10)
Z, ρ (Mg m^{-3})	8, 1.355	2, 1.418	8, 1.438	8, 1.515
μ (mm^{-1})	1.426	1.462	1.690	1.677
F(000)	9704	2608	12480	13360
Crystal size (mm)	0.31 x 0.13 x 0.07	0.16 x 0.11 x 0.08	0.33 x 0.19 x 0.10	0.20 x 0.19 x 0.09
Theta range for data collection (°)	0.73–17.27	1.41–23.26	1.16–23.26	0.96–23.26
Index ranges	$-26 \leq h \leq 26$, $-21 \leq k \leq 21$, $-26 \leq l \leq 25$	$-14 \leq h \leq 14$, $-17 \leq k \leq 17$, $-30 \leq l \leq 30$	$-36 \leq h \leq 36$, $-32 \leq k \leq 32$, $-32 \leq l \leq 32$	$-39 \leq h \leq 39$, $-33 \leq k \leq 33$, $-34 \leq l \leq 34$
Reflections collected	111627 [R_{int} = 0.1677]	40076 [R_{int} = 0.0686]	169522 [R_{int} = 0.0883]	130183 [R_{int} = 0.1245]
Completeness to θ_{max}	99.7%	99.9%	99.9%	99.8%
Data /restraints/ parameters	13873/8582/2296	16793/1169/1293	39883/9232/2909	20167/2098/1621
Final R indices [$I>2\sigma(I)$]	R_1 = 0.0932, wR_2 = 0.2135	R_1 = 0.0701, wR_2 = 0.1752	R_1 = 0.0760, wR_2 = 0.2002	R_1 = 0.0481, wR_2 = 0.0958
R indices (all data)	R_1 = 0.1298, wR_2 = 0.2403	R_1 = 0.1237, wR_2 = 0.2052	R_1 = 0.1238, wR_2 = 0.2402	R_1 = 0.0862, wR_2 = 0.1182
Goodness-of-fit on F^2	1.004	1.033	1.035	1.026
Largest diff. peak and hole (e Å^{-3})	2.013 and −1.170	1.199 and −1.261	2.03 and −1.657	0.934 and −0.671

Table S2. Selected Bond Distances (Å) and Angles (°) in **1–3**

1			
Fe1–O4	1.935(11)	Fe12–O44	1.902(12)
Fe1–O2	1.918(11)	Fe12–O43	1.911(13)
Fe1–O3	2.043(11)	Fe12–O45	2.090(13)
Fe1–O1	2.054(11)	Fe12–O54	2.093(12)
Fe1–O15	2.113(13)	Fe12–O57	2.101(17)
Fe1–O13	2.107(12)	Fe12–O55	2.109(17)
Fe2–O2	1.907(11)	Fe13–O44	1.876(11)
Fe2–O19	1.922(13)	Fe13–O67	1.941(14)
Fe2–O14	1.972(12)	Fe13–O58	1.976(15)
Fe2–O17	2.040(13)	Fe13–O64	2.029(14)
Fe2–O5	2.073(11)	Fe13–O46	2.097(12)
Fe2–O1	2.138(11)	Fe13–O45	2.142(13)
Fe3–O6	1.902(11)	Fe14–O47	1.911(13)
Fe3–O21	1.941(13)	Fe14–O48	1.923(11)
Fe3–O23	1.980(13)	Fe14–O46	2.038(11)
Fe3–O18	2.033(12)	Fe14–O66	2.068(14)
Fe3–O1	2.078(11)	Fe14–O49	2.067(12)
Fe3–O5	2.122(12)	Fe14–O69	2.099(14)
Fe4–O6	1.892(11)	Fe15–O48	1.905(11)
Fe4–O7	1.937(11)	Fe15–O73	1.928(14)
Fe4–O8	2.045(11)	Fe15–O70	1.949(15)
Fe4–O5	2.089(11)	Fe15–O71	2.014(16)
Fe4–O25	2.098(12)	Fe15–O50	2.083(13)
Fe4–O24	2.108(13)	Fe15–O49	2.139(14)
Fe5–O9	1.878(11)	Fe16–O51	1.905(14)
Fe5–O31	1.942(13)	Fe16–O83	1.929(16)
Fe5–O33	2.003(13)	Fe16–O76	1.980(15)
Fe5–O28	2.028(13)	Fe16–O72	2.043(18)
Fe5–O8	2.102(11)	Fe16–O49	2.043(13)
Fe5–O10	2.158(11)	Fe16–O50	2.043(13)
Fe6–O7	1.888(11)	Fe17–O53	1.906(12)
Fe6–O26	1.970(12)	Fe17–O51	1.901(12)
Fe6–O29	1.945(13)	Fe17–O50	2.043(13)
Fe6–O27	2.029(12)	Fe17–O52	2.063(14)
Fe6–O10	2.096(10)	Fe17–O75	2.075(15)
Fe6–O8	2.136(11)	Fe17–O77	2.126(12)
Fe7–O9	1.927(11)	Fe18–O53	1.905(12)
Fe7–O11	1.921(11)	Fe18–O79	1.961(15)
Fe7–O12	2.042(12)	Fe18–O78	1.959(13)
Fe7–O10	2.017(11)	Fe18–O54	2.062(11)
Fe7–O34	2.096(12)	Fe18–O82	2.051(16)

Fe7–O35	2.097(13)	Fe18–O52	2.163(14)
Fe8–O11	1.900(11)	Fe19–O43	1.902(12)
Fe8–O39	1.936(12)	Fe19–O62	1.94(2)
Fe8–O36	1.978(13)	Fe19–O56	2.00(2)
Fe8–O37	2.016(13)	Fe19–O81	2.034(16)
Fe8–O3	2.097(12)	Fe19–O52	2.080(15)
Fe8–O12	2.119(11)	Fe19–O54	2.140(14)
Fe9–O4	1.878(11)	Fe20–O48	1.890(11)
Fe9–O41	1.945(13)	Fe20–O44	1.944(12)
Fe9–O16	1.969(12)	Fe20–O53	1.909(12)
Fe9–O38	2.015(13)	Fe20–O80	2.072(14)
Fe9–O12	2.088(12)	Fe20–O74	2.085(13)
Fe9–O3	2.128(11)	Fe20–O68	2.070(13)
Fe10–O9	1.929(11)	Fe21–O43	1.910(14)
Fe10–O4	1.919(11)	Fe21–O51	1.914(13)
Fe10–O6	1.929(11)	Fe21–O47	1.907(11)
Fe10–O42	2.073(13)	Fe21–O84	2.095(17)
Fe10–O22	2.106(13)	Fe21–O59	2.105(16)
Fe10–O32	2.112(13)	Fe21–O61	2.098(18)
Fe11–O11	1.911(11)	Fe22–O47	1.913(12)
Fe11–O7	1.927(11)	Fe22–O60	1.923(15)
Fe11–O2	1.918(11)	Fe22–O65	1.984(14)
Fe11–O30	2.081(13)	Fe22–O63	2.024(15)
Fe11–O40	2.101(12)	Fe22–O45	2.082(13)
Fe11–O20	2.106(12)	Fe22–O46	2.168(11)
O2–Fe1–O1	79.4(5)	O43–Fe12–O45	102.9(5)
O4–Fe1–O1	104.0(5)	O44–Fe12–O54	104.3(5)
O3–Fe1–O1	174.3(5)	O43–Fe12–O54	80.0(5)
O2–Fe1–O13	89.5(5)	O45–Fe12–O54	175.3(5)
O4–Fe1–O13	156.0(5)	O44–Fe12–O57	89.1(7)
O3–Fe1–O13	83.8(4)	O43–Fe12–O57	155.2(7)
O1–Fe1–O13	92.5(5)	O45–Fe12–O57	94.9(6)
O2–Fe1–O15	155.9(5)	O54–Fe12–O57	81.3(6)
O4–Fe1–O15	88.4(5)	O44–Fe12–O55	154.3(8)
O3–Fe1–O15	93.3(5)	O43–Fe12–O55	89.3(9)
O1–Fe1–O15	81.6(5)	O45–Fe12–O55	82.4(6)
O13–Fe1–O15	76.5(5)	O54–Fe12–O55	94.1(6)
O2–Fe2–O19	102.5(5)	O57–Fe12–O55	75.9(9)
O2–Fe2–O14	96.6(5)	O44–Fe13–O67	103.9(5)
O19–Fe2–O14	93.6(5)	O44–Fe13–O58	96.7(6)
O2–Fe2–O17	164.5(5)	O67–Fe13–O58	93.3(8)
O19–Fe2–O17	92.7(5)	O44–Fe13–O64	164.2(6)
O14–Fe2–O17	84.8(5)	O67–Fe13–O64	91.5(6)

O2–Fe2–O5	92.6(4)	O58–Fe13–O64	85.6(6)
O19–Fe2–O5	93.6(5)	O44–Fe13–O46	92.5(5)
O14–Fe2–O5	166.8(5)	O67–Fe13–O46	91.4(5)
O17–Fe2–O5	83.8(5)	O58–Fe13–O46	168.4(5)
O2–Fe2–O1	77.6(4)	O64–Fe13–O46	83.6(5)
O19–Fe2–O1	174.8(5)	O44–Fe13–O45	77.5(5)
O14–Fe2–O1	91.6(5)	O67–Fe13–O45	173.7(6)
O17–Fe2–O1	87.0(5)	O58–Fe13–O45	92.5(7)
O5–Fe2–O1	81.2(4)	O64–Fe13–O45	86.8(6)
O6–Fe3–O21	103.9(5)	O46–Fe13–O45	82.5(5)
O6–Fe3–O23	98.0(5)	O47–Fe14–O48	109.5(5)
O21–Fe3–O23	94.9(6)	O47–Fe14–O46	80.1(5)
O6–Fe3–O18	165.4(5)	O48–Fe14–O46	104.8(5)
O21–Fe3–O18	89.9(5)	O47 –Fe14–O49	103.0(5)
O23–Fe3–O18	85.1(5)	O48–Fe14–O49	78.8(5)
O6–Fe3–O1	91.6(5)	O46–Fe14–O49	174.3(5)
O21–Fe3–O1	91.9(5)	O47–Fe14–O66	89.6(6)
O23–Fe3–O1	166.6(5)	O48–Fe14–O66	155.5(6)
O18–Fe3–O1	83.4(5)	O46–Fe14–O66	93.2(5)
O6–Fe3–O5	77.0(4)	O49–Fe14–O66	82.1(5)
O21–Fe3–O5	173.3(5)	O47–Fe14–O69	156.5(5)
O23–Fe3–O5	91.5(5)	O48–Fe14–O69	89.7(5)
O18–Fe3–O5	88.7(5)	O46–Fe14–O69	81.9(5)
O1–Fe3–O5	81.5(4)	O49–Fe14–O69	93.8(6)
O6–Fe4–O7	110.1(5)	O66–Fe14–O69	76.4(6)
O6–Fe4–O8	104.3(4)	O48–Fe15–O73	101.9(6)
O7–Fe4–O8	79.3(4)	O48–Fe15–O70	97.2(6)
O6–Fe4–O5	78.1(4)	O73–Fe15–O70	94.9(6)
O7–Fe4–O5	104.0(4)	O48–Fe15–O71	165.8(7)
O8–Fe4–O5	175.1(5)	O73–Fe15–O71	91.3(7)
O6–Fe4–O25	155.2(5)	O70–Fe15–O71	86.9(7)
O7–Fe4–O25	88.8(5)	O48–Fe15–O50	91.8(5)
O8–Fe4–O25	94.7(5)	O73–Fe15–O50	92.6(5)
O5–Fe4–O25	81.9(5)	O70–Fe15–O50	166.8(6)
O6–Fe4–O24	90.3(5)	O71–Fe15–O50	82.1(7)
O7–Fe4–O24	156.0(5)	O48–Fe15–O49	77.4(5)
O8–Fe4–O24	83.5(5)	O73–Fe15–O49	173.1(6)
O5–Fe4–O24	92.2(5)	O70–Fe15–O49	91.9(6)
O25–Fe4–O24	75.9(5)	O71–Fe15–O49	88.9(6)
O9–Fe5–O31	103.3(5)	O50–Fe15–O49	80.6(5)
O9–Fe5–O33	96.9(5)	O51–Fe16–O83	103.0(7)
O31–Fe5–O33	92.8(5)	O51–Fe16–O76	97.1(7)
O9–Fe5–O28	163.4(5)	O83–Fe16–O76	93.6(7)
O31–Fe5–O28	92.9(5)	O51–Fe16–O72	161.7(6)

O33–Fe5–O28	85.2(5)	O83–Fe16–O72	95.0(8)
O9–Fe5–O8	90.6(4)	O76–Fe16–O72	84.9(8)
O31–Fe5–O8	93.4(5)	O51–Fe16–O49	91.0(5)
O33–Fe5–O8	168.9(5)	O83–Fe16–O49	92.1(7)
O28–Fe5–O8	85.2(5)	O76–Fe16–O49	168.7(7)
O9–Fe5–O10	76.5(4)	O72–Fe16–O49	84.8(7)
O31–Fe5–O10	175.6(5)	O51–Fe16–O50	76.9(5)
O33–Fe5–O10	91.6(5)	O83–Fe16–O50	174.0(7)
O28–Fe5–O10	87.0(5)	O76–Fe16–O50	92.3(5)
O8–Fe5–O10	82.2(4)	O72–Fe16–O50	84.8(6)
O7–Fe6–O29	102.3(5)	O49–Fe16–O50	81.9(5)
O7–Fe6–O26	96.8(5)	O51–Fe17–O53	109.4(5)
O29–Fe6–O26	93.6(5)	O51–Fe17–O50	79.0(5)
O7–Fe6–O27	166.1(5)	O53–Fe17–O50	104.1(5)
O29–Fe6–O27	91.0(5)	O51–Fe17–O52	104.7(6)
O26–Fe6–O27	86.4(5)	O53–Fe17–O52	79.5(5)
O7–Fe6–O10	91.1(4)	O50–Fe17–O52	173.9(5)
O29–Fe6–O10	92.1(5)	O51–Fe17–O75	90.0(5)
O26–Fe6–O10	169.1(5)	O53–Fe17–O75	155.5(6)
O27–Fe6–O10	84.2(5)	O50–Fe17–O75	94.0(7)
O7–Fe6–O8	78.1(4)	O52–Fe17–O75	81.2(7)
O29–Fe6–O8	175.0(5)	O51–Fe17–O77	155.8(5)
O26–Fe6–O8	91.3(5)	O53–Fe17–O77	90.1(5)
O27–Fe6–O8	88.3(5)	O50–Fe17–O77	82.5(5)
O10–Fe6–O8	82.9(4)	O52–Fe17–O77	92.6(5)
O11–Fe7–O9	111.0(4)	O75–Fe17–O77	75.7(5)
O11–Fe7–O10	105.3(4)	O53–Fe18–O78	97.8(5)
O9–Fe7–O10	79.0(4)	O53–Fe18–O79	104.0(6)
O11–Fe7–O12	78.6(4)	O78–Fe18–O79	93.2(6)
O9–Fe7–O12	104.3(5)	O53–Fe18–O82	163.1(7)
O10–Fe7–O12	173.8(4)	O78–Fe18–O82	83.6(6)
O11–Fe7–O34	153.3(5)	O79–Fe18–O82	92.7(7)
O9–Fe7–O34	89.9(5)	O53–Fe18–O54	92.0(5)
O10–Fe7–O34	94.5(5)	O78–Fe18–O54	166.2(5)
O12–Fe7–O34	80.3(5)	O79–Fe18–O54	93.7(6)
O11–Fe7–O35	88.9(5)	O82–Fe18–O54	84.2(6)
O9–Fe7–O35	154.9(5)	O53–Fe18–O52	77.0(5)
O10–Fe7–O35	81.1(5)	O78–Fe18–O52	91.0(5)
O12–Fe7–O35	94.3(5)	O79–Fe18–O52	175.5(6)
O34–Fe7–O35	76.6(5)	O82–Fe18–O52	86.1(7)
O11–Fe8–O39	103.1(5)	O54–Fe18–O52	81.8(6)
O11–Fe8–O36	97.0(5)	O43–Fe19–O62	101.1(7)
O39–Fe8–O36	94.3(5)	O43–Fe19–O56	96.5(7)
O11–Fe8–O37	163.5(5)	O62–Fe19–O56	93.4(11)

O39–Fe8–O37	92.8(5)	O43–Fe19–O81	165.7(7)
O36–Fe8–O37	86.2(5)	O62–Fe19–O81	92.6(8)
O11–Fe8–O3	90.6(5)	O56–Fe19–O81	86.6(7)
O39–Fe8–O3	92.3(5)	O43–Fe19–O52	93.0(6)
O36–Fe8–O3	168.5(5)	O62–Fe19–O52	94.3(8)
O37–Fe8–O3	84.1(5)	O56–Fe19–O52	166.4(7)
O11–Fe8–O12	77.1(4)	O81–Fe19–O52	81.9(6)
O39–Fe8–O12	173.6(5)	O43–Fe19–O54	79.0(5)
O36–Fe8–O12	92.0(5)	O62–Fe19–O54	176.2(8)
O37–Fe8–O12	86.6(5)	O56–Fe19–O54	90.3(9)
O3–Fe8–O12	81.3(4)	O81–Fe19–O54	87.1(7)
O4–Fe9–O41	101.8(5)	O52–Fe19–O54	81.9(5)
O4–Fe9–O16	98.3(5)	O48–Fe20–O53	95.3(5)
O41–Fe9–O16	94.3(5)	O48–Fe20–O44	95.5(5)
O4–Fe9–O38	165.3(5)	O53–Fe20–O44	96.2(5)
O41–Fe9–O38	92.0(5)	O48–Fe20–O68	88.6(5)
O16–Fe9–O38	85.4(5)	O53–Fe20–O68	169.5(5)
O4–Fe9–O12	91.2(5)	O44–Fe20–O68	93.1(5)
O41–Fe9–O12	92.9(5)	O48–Fe20–O80	170.3(5)
O16–Fe9–O12	166.6(5)	O53–Fe20–O80	93.3(5)
O38–Fe9–O12	83.1(5)	O44–Fe20–O80	88.0(5)
O4–Fe9–O3	77.6(4)	O68–Fe20–O80	82.2(5)
O41–Fe9–O3	174.2(5)	O48–Fe20–O74	94.9(5)
O16–Fe9–O3	91.4(5)	O53–Fe20–O74	88.4(5)
O38–Fe9–O3	88.2(5)	O44–Fe20–O74	168.1(5)
O12–Fe9–O3	81.3(4)	O68–Fe20–O74	81.5(5)
O4–Fe10–O6	95.5(5)	O80–Fe20–O74	80.8(5)
O4–Fe10–O9	95.0(5)	O47–Fe21–O43	96.4(5)
O6–Fe10–O9	95.4(4)	O47–Fe21–O51	94.6(5)
O4–Fe10–O42	94.7(5)	O43–Fe21–O51	96.5(5)
O6–Fe10–O42	168.5(5)	O47–Fe21–O84	89.3(7)
O9–Fe10–O42	88.8(5)	O43–Fe21–O84	166.9(7)
O4–Fe10–O22	90.6(5)	O51–Fe21–O84	94.8(6)
O6–Fe10–O22	94.3(5)	O47–Fe21–O61	168.8(7)
O9–Fe10–O22	168.3(5)	O43–Fe21–O61	93.2(8)
O42–Fe10–O22	80.5(5)	O51–Fe21–O61	90.1(7)
O4–Fe10–O32	171.3(5)	O84–Fe21–O61	80.2(8)
O6–Fe10–O32	87.8(5)	O47–Fe21–O59	94.6(6)
O9–Fe10–O32	92.7(5)	O43–Fe21–O59	86.5(6)
O42–Fe10–O32	81.3(5)	O51–Fe21–O59	169.9(6)
O22–Fe10–O32	81.1(5)	O84–Fe21–O59	81.2(7)
O11–Fe11–O2	94.6(5)	O61–Fe21–O59	80.2(7)
O11–Fe11–O7	95.7(5)	O47–Fe22–O60	104.1(6)
O2–Fe11–O7	95.9(5)	O47–Fe22–O65	97.0(6)

O11–Fe11–O30	89.9(5)	O60–Fe22–O65	94.9(7)
O2–Fe11–O30	169.7(5)	O47–Fe22–O63	164.1(6)
O7–Fe11–O30	92.9(5)	O60–Fe22–O63	91.0(7)
O11–Fe11–O40	93.2(5)	O65–Fe22–O63	86.2(6)
O2–Fe11–O40	89.1(5)	O47–Fe22–O45	90.7(5)
O7–Fe11–O40	169.4(5)	O60–Fe22–O45	92.4(6)
O30–Fe11–O40	81.4(5)	O65–Fe22–O45	167.7(6)
O11–Fe11–O20	168.8(5)	O63–Fe22–O45	83.8(5)
O2–Fe11–O20	93.9(5)	O47–Fe22–O46	76.8(4)
O7–Fe11–O20	90.7(4)	O60–Fe22–O46	174.5(7)
O30–Fe11–O20	80.5(5)	O65–Fe22–O46	90.3(6)
O40–Fe11–O20	79.7(4)	O63–Fe22–O46	87.6(5)
O44–Fe12–O43	111.3(5)	O45–Fe22–O46	82.2(5)
O44–Fe12–O45	78.2(5)		
2·1.5CH_2Cl_2·MeCN			
Fe1–O8	1.945(6)	Fe6–O30	2.003(7)
Fe1–O9	1.954(6)	Fe6–O9	2.008(6)
Fe1–O7	1.966(6)	Fe7–O6	1.962(6)
Fe1–O1	2.083(6)	Fe7–O1	1.991(5)
Fe1–O3	2.086(6)	Fe7–O12	2.001(6)
Fe1–O2	2.100(6)	Fe7–O15	2.008(6)
Fe2–O4	1.813(6)	Fe7–O19	2.020(6)
Fe2–O1	1.947(6)	Fe7–O33	2.069(6)
Fe2–O18	1.983(6)	Fe8–O11	1.944(6)
Fe2–O7	2.002(6)	Fe8–O12	1.952(6)
Fe2–O16	2.008(6)	Fe8–O10	1.959(6)
Fe3–O4	1.967(6)	Fe8–O1	2.089(6)
Fe3–O2	1.982(5)	Fe8–O2	2.091(5)
Fe3–O10	2.002(6)	Fe8–O3	2.107(6)
Fe3–O13	2.006(6)	Fe9–O4	1.881(6)
Fe3–O22	2.021(6)	Fe9–O13	1.966(6)
Fe3–O20	2.069(6)	Fe9–O21	2.014(6)
Fe4–O5	1.821(6)	Fe9–O17	2.024(6)
Fe4–O2	1.944(5)	Fe9–O35	2.090(7)
Fe4–O23	1.984(6)	Fe9–O34	2.104(7)
Fe4–O8	1.996(6)	Fe10–O5	1.884(6)
Fe4–O24	2.000(6)	Fe10–O14	1.995(6)
Fe5–O5	1.960(6)	Fe10–O27	1.997(7)
Fe5–O3	1.981(6)	Fe10–O25	2.019(7)
Fe5–O14	2.005(6)	Fe10–O37	2.093(6)
Fe5–O11	2.011(6)	Fe10–O36	2.104(7)
Fe5–O28	2.017(6)	Fe11–O6	1.898(6)
Fe5–O26	2.071(7)	Fe11–O15	1.980(6)
Fe6–O6	1.812(6)	Fe11–O31	1.986(7)

Fe6–O3	1.956(6)	Fe11–O32	2.014(6)
Fe6–O29	1.963(7)	Fe11–O39	2.093(6)
		Fe11–O38	2.106(7)
O8–Fe1–O9	101.5(3)	O3–Fe6–O29	98.3(3)
O8–Fe1–O7	98.8(3)	O6–Fe6–O30	100.6(3)
O9–Fe1–O7	98.9(2)	O3–Fe6–O30	156.1(3)
O8–Fe1–O1	156.7(2)	O6–Fe6–O9	101.9(3)
O9–Fe1–O1	101.8(2)	O3–Fe6–O9	80.6(2)
O7–Fe1–O1	78.9(2)	O29–Fe6–O9	158.2(3)
O8–Fe1–O3	103.1(2)	O30–Fe6–O9	88.0(3)
O9–Fe1–O3	78.7(2)	O6–Fe7–O1	96.4(2)
O7–Fe1–O3	158.1(2)	O6–Fe7–O12	93.5(3)
O1–Fe1–O3	80.3(2)	O1–Fe7–O12	80.4(2)
O8–Fe1–O2	77.8(2)	O6–Fe7–O15	77.3(2)
O9–Fe1–O2	158.3(2)	O1–Fe7–O15	171.8(3)
O7–Fe1–O2	102.7(2)	O12–Fe7–O15	104.9(3)
O1–Fe1–O2	80.2(2)	O6–Fe7–O19	165.0(2)
O3–Fe1–O2	80.4(2)	O1–Fe7–O19	98.4(2)
O4–Fe2–O1	101.0(2)	O12–Fe7–O19	91.5(3)
O4–Fe2–O18	98.7(3)	O15–Fe7–O19	87.8(2)
O1–Fe2–O18	99.3(2)	O6–Fe7–O33	90.3(2)
O4–Fe2–O7	99.3(2)	O1–Fe7–O33	84.7(2)
O1–Fe2–O7	81.4(2)	O12–Fe7–O33	164.9(2)
O18–Fe2–O7	161.5(3)	O15–Fe7–O33	90.2(3)
O4–Fe2–O16	101.6(3)	O19–Fe7–O33	88.5(3)
O1–Fe2–O16	156.3(2)	O11–Fe8–O12	103.1(2)
O18–Fe2–O16	84.2(3)	O11–Fe8–O10	102.8(2)
O7–Fe2–O16	88.0(2)	O12–Fe8–O10	101.8(3)
O4–Fe3–O2	96.2(2)	O11–Fe8–O1	158.6(2)
O4–Fe3–O10	94.5(2)	O12–Fe8–O1	79.1(2)
O2–Fe3–O10	80.7(2)	O10–Fe8–O1	97.4(2)
O4–Fe3–O13	76.3(2)	O11–Fe8–O2	96.8(2)
O2–Fe3–O13	171.8(2)	O12–Fe8–O2	159.2(2)
O10–Fe3–O13	103.0(2)	O10–Fe8–O2	79.1(2)
O4–Fe3–O22	165.1(2)	O1–Fe8–O2	80.2(2)
O2–Fe3–O22	98.2(2)	O11–Fe8–O3	79.0(2)
O10–Fe3–O22	91.6(2)	O12–Fe8–O3	97.8(2)
O13–Fe3–O22	89.1(3)	O10–Fe8–O3	159.2(2)
O4–Fe3–O20	90.0(2)	O1–Fe8–O3	79.7(2)
O2–Fe3–O20	85.6(2)	O2–Fe8–O3	80.1(2)
O10–Fe3–O20	166.0(2)	O4–Fe9–O13	79.2(2)
O13–Fe3–O20	91.0(2)	O4–Fe9–O21	99.0(3)
O22–Fe3–O20	87.3(2)	O13–Fe9–O21	91.4(3)

O5–Fe4–O2	101.3(2)	O4–Fe9–O17	94.1(3)
O5–Fe4–O23	100.1(3)	O13–Fe9–O17	173.3(3)
O2–Fe4–O23	98.2(2)	O21–Fe9–O17	89.5(3)
O5–Fe4–O8	100.4(3)	O4–Fe9–O35	167.4(3)
O2–Fe4–O8	80.3(2)	O13–Fe9–O35	98.2(3)
O23–Fe4–O8	159.3(3)	O21–Fe9–O35	93.4(3)
O5–Fe4–O24	101.0(3)	O17–Fe9–O35	88.4(3)
O2–Fe4–O24	156.7(2)	O4–Fe9–O34	105.1(3)
O23–Fe4–O24	84.6(3)	O13–Fe9–O34	95.1(3)
O8–Fe4–O24	88.9(2)	O21–Fe9–O34	155.8(3)
O5–Fe5–O3	96.1(2)	O17–Fe9–O34	86.7(3)
O5–Fe5–O14	77.1(2)	O35–Fe9–O34	62.7(3)
O3–Fe5–O14	172.7(3)	O5–Fe10–O14	79.1(2)
O5–Fe5–O11	93.3(2)	O5–Fe10–O27	100.0(3)
O3–Fe5–O11	80.5(2)	O14–Fe10–O27	90.6(3)
O14–Fe5–O11	102.5(3)	O5–Fe10–O25	94.3(3)
O14–Fe5–O26	89.9(3)	O14–Fe10–O25	173.3(3)
O11–Fe5–O26	167.6(2)	O27–Fe10–O25	89.5(3)
O28–Fe5–O26	87.6(3)	O5–Fe10–O37	165.6(3)
O5–Fe5–O28	165.9(3)	O14–Fe10–O37	96.7(3)
O3–Fe5–O28	97.8(3)	O27–Fe10–O37	93.7(3)
O14–Fe5–O28	88.9(3)	O25–Fe10–O37	90.0(3)
O11–Fe5–O28	91.5(3)	O5–Fe10–O36	103.8(3)
O5–Fe5–O26	90.6(2)	O14–Fe10–O36	95.6(3)
O3–Fe5–O26	87.3(2)	O27–Fe10–O36	156.1(3)
O6–Fe6–O3	102.3(3)	O25–Fe10–O36	86.9(3)
O6–Fe6–O29	99.5(3)	O37–Fe10–O36	62.7(3)
3 0.25thf			
Fe1–O1	1.930(6)	Fe16–O49	1.997(7)
Fe1–O22	1.980(7)	Fe16–O11	2.000(6)
Fe1–O2	1.983(6)	Fe16–O37	2.032(7)
Fe1–O20	2.007(7)	Fe16–O17	2.161(7)
Fe1–O24	2.012(7)	Fe17–O52	1.930(7)
Fe1–O14	2.162(6)	Fe17–O75	1.989(8)
Fe2–O1	1.905(7)	Fe17–O53	2.001(6)
Fe2–O15	1.992(7)	Fe17–O71	2.008(7)
Fe2–O5	2.034(6)	Fe17–O73	2.020(7)
Fe2–O23	2.050(7)	Fe17–O65	2.160(7)
Fe2–O26	2.050(7)	Fe18–O52	1.903(7)
Fe2–O3	2.079(6)	Fe18–O66	1.999(8)
Fe3–O1	1.916(6)	Fe18–O76	2.036(8)
Fe3–O27	1.988(7)	Fe18–O56	2.045(7)
Fe3–O4	2.001(6)	Fe18–O77	2.060(8)
Fe3–O25	2.018(7)	Fe18–O55	2.074(7)

Fe3–O28	2.021(7)	Fe19–O52	1.918(7)
Fe3–O14	2.144(7)	Fe19–O78	1.983(7)
Fe4–O6	1.917(6)	Fe19–O54	1.994(6)
Fe4–O2	1.995(6)	Fe19–O79	2.006(7)
Fe4–O32	1.998(7)	Fe19–O74	2.031(7)
Fe4–O21	2.006(7)	Fe19–O65	2.156(7)
Fe4–O30	2.012(7)	Fe20–O62	1.919(7)
Fe4–O16	2.180(7)	Fe20–O81	1.985(8)
Fe5–O3	1.808(6)	Fe20–O53	1.986(7)
Fe5–O9	1.971(6)	Fe20–O83	1.995(7)
Fe5–O2	2.028(6)	Fe20–O72	2.004(7)
Fe5–O8	2.030(6)	Fe20–O68	2.181(8)
Fe5–O16	2.044(6)	Fe21–O57	1.793(7)
Fe6–O7	1.805(6)	Fe21–O63	1.979(6)
Fe6–O9	1.967(6)	Fe21–O53	2.029(7)
Fe6–O2	2.035(6)	Fe21–O54	2.034(6)
Fe6–O4	2.035(6)	Fe21–O65	2.076(7)
Fe6–O14	2.088(6)	Fe23–O80	2.003(7)
Fe7–O10	1.902(6)	Fe23–O87	2.006(7)
Fe7–O15	1.991(7)	Fe23–O67	2.168(7)
Fe7–O3	2.026(7)	Fe24–O56	1.807(7)
Fe7–O38	2.029(7)	Fe24–O63	1.958(6)
Fe7–O40	2.030(6)	Fe24–O54	2.035(7)
Fe7–O5	2.084(7)	Fe24–O59	2.039(6)
Fe8–O5	1.795(6)	Fe24–O67	2.061(7)
Fe8–O9	1.968(6)	Fe25–O60	1.907(7)
Fe8–O11	2.030(6)	Fe25–O66	1.983(7)
Fe8–O4	2.049(6)	Fe25–O91	2.032(8)
Fe8–O17	2.057(6)	Fe25–O55	2.036(7)
Fe9–O13	1.924(6)	Fe25–O89	2.045(8)
Fe9–O4	1.970(6)	Fe25–O56	2.066(7)
Fe9–O34	1.987(7)	Fe26–O62	1.913(7)
Fe9–O29	1.998(7)	Fe26–O69	1.972(7)
Fe9–O36	2.004(7)	Fe26–O64	2.008(7)
Fe9–O17	2.195(6)	Fe26–O82	2.027(7)
Fe10–O6	1.927(6)	Fe26–O93	2.039(7)
Fe10–O44	1.978(7)	Fe26–O57	2.111(7)
Fe10–O8	1.982(6)	Fe27–O58	1.906(7)
Fe10–O42	2.004(7)	Fe27–O69	1.989(7)
Fe10–O31	2.027(7)	Fe27–O57	2.024(7)
Fe10–O16	2.151(7)	Fe27–O86	2.033(7)
Fe11–O6	1.905(6)	Fe27–O95	2.040(7)
Fe11–O18	1.996(7)	Fe27–O64	2.099(7)
Fe11–O7	2.006(6)	Fe28–O62	1.918(7)

Fe11–O45	2.034(7)	Fe28–O94	1.986(7)
Fe11–O33	2.036(7)	Fe28–O61	1.993(7)
Fe11–O12	2.107(6)	Fe28–O84	2.012(7)
Fe12–O10	1.926(6)	Fe28–O102	2.016(8)
Fe12–O41	1.986(7)	Fe28–O68	2.155(7)
Fe12–O43	1.993(7)	Fe29–O64	1.800(7)
Fe12–O8	2.003(7)	Fe29–O63	1.977(6)
Fe12–O46	2.022(7)	Fe29–O59	2.025(7)
Fe12–O19	2.148(6)	Fe29–O61	2.038(6)
Fe13–O12	1.795(6)	Fe29–O70	2.067(7)
Fe13–O9	1.981(6)	Fe30–O58	1.927(7)
Fe13–O8	2.028(6)	Fe30–O96	1.978(7)
Fe13–O11	2.051(6)	Fe30–O59	2.000(7)
Fe13–O19	2.052(6)	Fe30–O97	2.010(7)
Fe14–O13	1.912(7)	Fe30–O88	2.035(7)
Fe14–O18	1.980(6)	Fe30–O67	2.147(7)
Fe14–O12	2.005(6)	Fe31–O60	1.924(7)
Fe14–O35	2.026(7)	Fe31–O92	1.984(7)
Fe14–O50	2.042(7)	Fe31–O61	1.985(7)
Fe14–O7	2.129(6)	Fe31–O101	2.012(7)
Fe15–O10	1.926(6)	Fe31–O100	2.021(8)
Fe15–O11	1.983(6)	Fe31–O70	2.169(7)
Fe15–O39	2.000(7)	Fe32–O60	1.921(7)
Fe15–O47	2.000(7)	Fe32–O59	1.986(7)
Fe15–O48	2.005(7)	Fe32–O90	1.995(7)
Fe15–O19	2.198(7)	Fe32–O98	2.003(7)
Fe16–O13	1.920(6)	Fe32–O99	2.015(8)
Fe16–O51	1.987(7)	Fe32–O70	2.182(7)
O1–Fe1–O22	96.1(3)	O52–Fe17–O75	96.0(3)
O1–Fe1–O2	89.5(3)	O52–Fe17–O53	89.5(3)
O22–Fe1–O2	96.6(3)	O75–Fe17–O53	97.5(3)
O1–Fe1–O20	171.3(3)	O52–Fe17–O71	171.4(3)
O22–Fe1–O20	91.7(3)	O75–Fe17–O71	91.6(3)
O2–Fe1–O20	93.5(3)	O53–Fe17–O71	93.5(3)
O1–Fe1–O24	91.9(3)	O52–Fe17–O73	91.5(3)
O22–Fe1–O24	90.8(3)	O75–Fe17–O73	90.4(3)
O2–Fe1–O24	172.3(3)	O53–Fe17–O73	171.9(3)
O20–Fe1–O24	84.0(3)	O71–Fe17–O73	84.4(3)
O1–Fe1–O14	76.0(3)	O52–Fe17–O65	75.9(3)
O22–Fe1–O14	171.5(3)	O75–Fe17–O65	171.6(3)
O2–Fe1–O14	80.6(2)	O53–Fe17–O65	80.5(3)
O20–Fe1–O14	96.4(3)	O71–Fe17–O65	96.6(3)
O24–Fe1–O14	92.3(3)	O73–Fe17–O65	91.9(3)

O1–Fe2–O15	176.0(3)	O52–Fe18–O66	176.1(3)
O1–Fe2–O5	97.5(3)	O52–Fe18–O76	92.3(3)
O15–Fe2–O5	78.9(3)	O66–Fe18–O76	90.7(3)
O1–Fe2–O23	92.6(3)	O52–Fe18–O56	98.0(3)
O15–Fe2–O23	90.7(3)	O66–Fe18–O56	78.8(3)
O5–Fe2–O23	167.1(3)	O76–Fe18–O56	167.8(3)
O1–Fe2–O26	91.7(3)	O52–Fe18–O77	92.3(3)
O15–Fe2–O26	90.4(3)	O66–Fe18–O77	90.0(3)
O5–Fe2–O26	95.1(3)	O76–Fe18–O77	92.9(3)
O23–Fe2–O26	92.6(3)	O56–Fe18–O77	93.3(3)
O1–Fe2–O3	96.7(3)	O52–Fe18–O55	97.0(3)
O15–Fe2–O3	81.0(3)	O66–Fe18–O55	80.3(3)
O5–Fe2–O3	80.1(3)	O76–Fe18–O55	92.3(3)
O23–Fe2–O3	90.9(3)	O56–Fe18–O55	80.0(3)
O26–Fe2–O3	170.8(3)	O77–Fe18–O55	169.1(3)
O1–Fe3–O27	96.4(3)	O52–Fe19–O78	96.6(3)
O1–Fe3–O4	89.0(3)	O52–Fe19–O54	89.2(3)
O27–Fe3–O4	98.0(3)	O78–Fe19–O54	96.6(3)
O1–Fe3–O25	92.3(3)	O52–Fe19–O79	172.2(3)
O27–Fe3–O25	90.0(3)	O78–Fe19–O79	90.6(3)
O4–Fe3–O25	171.7(3)	O54–Fe19–O79	92.7(3)
O1–Fe3–O28	172.3(3)	O52–Fe19–O74	91.4(3)
O27–Fe3–O28	91.1(3)	O78–Fe19–O74	90.4(3)
O4–Fe3–O28	92.0(3)	O54–Fe19–O74	172.9(3)
O25–Fe3–O28	85.6(3)	O79–Fe19–O74	85.8(3)
O1–Fe3–O14	76.7(3)	O52–Fe19–O65	76.2(3)
O27–Fe3–O14	172.9(3)	O78–Fe19–O65	172.3(3)
O4–Fe3–O14	81.0(2)	O54–Fe19–O65	80.7(3)
O25–Fe3–O14	91.4(3)	O79–Fe19–O65	96.6(3)
O28–Fe3–O14	95.9(3)	O74–Fe19–O65	92.5(3)
O6–Fe4–O2	88.9(3)	O62–Fe20–O81	97.3(3)
O6–Fe4–O32	96.9(3)	O62–Fe20–O53	89.8(3)
O2–Fe4–O32	98.3(3)	O81–Fe20–O53	98.0(3)
O6–Fe4–O21	170.2(3)	O62–Fe20–O83	93.6(3)
O2–Fe4–O21	93.2(3)	O81–Fe20–O83	91.2(3)
O32–Fe4–O21	92.3(3)	O53–Fe20–O83	169.7(3)
O6–Fe4–O30	92.9(3)	O62–Fe20–O72	168.2(3)
O2–Fe4–O30	170.8(3)	O81–Fe20–O72	94.0(3)
O32–Fe4–O30	90.4(3)	O53–Fe20–O72	91.8(3)
O21–Fe4–O30	83.6(3)	O83–Fe20–O72	82.9(3)
O6–Fe4–O16	75.5(3)	O62–Fe20–O68	74.8(3)
O2–Fe4–O16	79.9(2)	O81–Fe20–O68	171.8(3)
O32–Fe4–O16	172.2(3)	O53–Fe20–O68	80.1(3)
O21–Fe4–O16	95.4(3)	O83–Fe20–O68	91.4(3)

O30–Fe4–O16	91.8(3)	O72–Fe20–O68	94.0(3)
O3–Fe5–O9	100.1(3)	O57–Fe21–O63	99.7(3)
O3–Fe5–O2	113.7(3)	O57–Fe21–O53	114.1(3)
O9–Fe5–O2	81.6(2)	O63–Fe21–O53	81.7(3)
O3–Fe5–O8	113.8(3)	O57–Fe21–O54	114.3(3)
O9–Fe5–O8	82.1(3)	O63–Fe21–O54	81.4(3)
O2–Fe5–O8	131.6(3)	O53–Fe21–O54	130.7(3)
O3–Fe5–O16	119.1(3)	O57–Fe21–O65	120.9(3)
O9–Fe5–O16	140.8(3)	O63–Fe21–O65	139.4(3)
O2–Fe5–O16	82.5(3)	O53–Fe21–O65	81.9(3)
O8–Fe5–O16	82.2(3)	O54–Fe21–O65	81.8(3)
O7–Fe6–O9	99.6(3)	O55–Fe22–O63	100.1(3)
O7–Fe6–O2	114.3(3)	O55–Fe22–O53	113.9(3)
O9–Fe6–O2	81.6(3)	O63–Fe22–O53	81.7(3)
O7–Fe6–O4	114.7(3)	O55–Fe22–O61	113.6(3)
O9–Fe6–O4	81.6(2)	O63–Fe22–O61	82.3(3)
O2–Fe6–O4	130.1(3)	O53–Fe22–O61	131.8(3)
O7–Fe6–O14	121.4(3)	O55–Fe22–O68	119.1(3)
O9–Fe6–O14	139.1(3)	O63–Fe22–O68	140.8(3)
O2–Fe6–O14	81.3(2)	O53–Fe22–O68	82.1(3)
O4–Fe6–O14	81.6(2)	O61–Fe22–O68	82.3(3)
O10–Fe7–O15	174.6(3)	O58–Fe23–O54	88.8(3)
O10–Fe7–O3	98.1(3)	O58–Fe23–O85	96.9(3)
O15–Fe7–O3	82.3(3)	O54–Fe23–O85	97.9(3)
O10–Fe7–O38	91.2(3)	O58–Fe23–O80	169.7(3)
O15–Fe7–O38	88.0(3)	O54–Fe23–O80	92.7(3)
O3–Fe7–O38	169.7(3)	O85–Fe23–O80	93.0(3)
O10–Fe7–O40	93.3(3)	O58–Fe23–O87	92.7(3)
O15–Fe7–O40	92.0(3)	O54–Fe23–O87	171.7(3)
O3–Fe7–O40	92.5(3)	O85–Fe23–O87	90.0(3)
O38–Fe7–O40	91.3(3)	O80–Fe23–O87	84.5(3)
O10–Fe7–O5	97.0(3)	O58–Fe23–O67	75.5(3)
O15–Fe7–O5	77.8(3)	O54–Fe23–O67	80.9(3)
O3–Fe7–O5	80.1(3)	O85–Fe23–O67	172.2(3)
O38–Fe7–O5	94.5(3)	O80–Fe23–O67	94.7(3)
O40–Fe7–O5	168.1(3)	O87–Fe23–O67	91.4(3)
O5–Fe8–O9	100.4(3)	O56–Fe24–O63	100.5(3)
O5–Fe8–O11	113.4(3)	O56–Fe24–O54	114.0(3)
O9–Fe8–O11	82.9(3)	O63–Fe24–O54	81.9(3)
O5–Fe8–O4	113.5(3)	O56–Fe24–O59	113.2(3)
O9–Fe8–O4	81.3(2)	O63–Fe24–O59	81.7(3)
O11–Fe8–O4	132.4(2)	O54–Fe24–O59	132.0(3)
O5–Fe8–O17	119.0(3)	O56–Fe24–O67	119.3(3)
O9–Fe8–O17	140.5(3)	O63–Fe24–O67	140.2(3)

O11−Fe8−O17	82.5(3)	O54−Fe24−O67	82.4(3)
O4−Fe8−O17	82.0(2)	O59−Fe24−O67	82.2(3)
O13−Fe9−O4	90.3(3)	O60−Fe25−O66	175.7(3)
O13−Fe9−O34	96.7(3)	O60−Fe25−O91	93.2(3)
O4−Fe9−O34	98.4(3)	O66−Fe25−O91	91.0(3)
O13−Fe9−O29	168.5(3)	O60−Fe25−O55	98.0(3)
O4−Fe9−O29	92.1(3)	O66−Fe25−O55	81.7(3)
O34−Fe9−O29	94.1(3)	O91−Fe25−O55	92.1(3)
O13−Fe9−O36	92.5(3)	O60−Fe25−O89	91.0(3)
O4−Fe9−O36	170.9(3)	O66−Fe25−O89	88.9(3)
O34−Fe9−O36	89.9(3)	O91−Fe25−O89	93.0(3)
O29−Fe9−O36	83.6(3)	O55−Fe25−O89	169.3(3)
O13−Fe9−O17	74.9(3)	O60−Fe25−O56	97.1(3)
O4−Fe9−O17	80.4(2)	O66−Fe25−O56	78.6(3)
O34−Fe9−O17	171.5(3)	O91−Fe25−O56	168.0(3)
O29−Fe9−O17	94.3(3)	O55−Fe25−O56	80.4(3)
O36−Fe9−O17	91.9(3)	O89−Fe25−O56	92.9(3)
O6−Fe10−O44	97.0(3)	O62−Fe26−O69	174.5(3)
O6−Fe10−O8	89.1(3)	O62−Fe26−O64	98.1(3)
O44−Fe10−O8	97.2(3)	O69−Fe26−O64	81.6(3)
O6−Fe10−O42	170.5(3)	O62−Fe26−O82	90.9(3)
O44−Fe10−O42	92.1(3)	O69−Fe26−O82	88.9(3)
O8−Fe10−O42	92.3(3)	O64−Fe26−O82	169.0(3)
O6−Fe10−O31	92.8(3)	O62−Fe26−O93	93.6(3)
O44−Fe10−O31	90.6(3)	O69−Fe26−O93	91.9(3)
O8−Fe10−O31	171.6(3)	O64−Fe26−O93	93.6(3)
O42−Fe10−O31	84.4(3)	O82−Fe26−O93	92.0(3)
O6−Fe10−O16	76.0(3)	O62−Fe26−O57	95.8(3)
O44−Fe10−O16	172.7(3)	O69−Fe26−O57	78.7(3)
O8−Fe10−O16	80.6(2)	O64−Fe26−O57	80.3(3)
O42−Fe10−O16	94.9(3)	O82−Fe26−O57	92.6(3)
O31−Fe10−O16	91.9(3)	O93−Fe26−O57	169.4(3)
O6−Fe11−O18	174.8(3)	O58−Fe27−O69	175.0(3)
O6−Fe11−O7	97.4(3)	O58−Fe27−O57	96.8(3)
O18−Fe11−O7	81.5(3)	O69−Fe27−O57	80.4(3)
O6−Fe11−O45	91.2(3)	O58−Fe27−O86	92.6(3)
O18−Fe11−O45	89.4(3)	O69−Fe27−O86	91.8(3)
O7−Fe11−O45	169.3(3)	O57−Fe27−O86	94.6(3)
O6−Fe11−O33	93.3(3)	O58−Fe27−O95	91.7(3)
O18−Fe11−O33	91.8(3)	O69−Fe27−O95	90.7(3)
O7−Fe11−O33	93.3(3)	O57−Fe27−O95	169.2(3)
O45−Fe11−O33	92.5(3)	O86−Fe27−O95	91.7(3)
O6−Fe11−O12	96.4(3)	O58−Fe27−O64	96.5(3)
O18−Fe11−O12	78.4(3)	O69−Fe27−O64	78.9(3)

O7–Fe11–O12	80.6(3)	O57–Fe27–O64	80.2(3)
O45–Fe11–O12	92.2(3)	O86–Fe27–O64	170.0(3)
O33–Fe11–O12	169.1(3)	O95–Fe27–O64	92.2(3)
O10–Fe12–O41	96.4(3)	O62–Fe28–O94	97.5(3)
O10–Fe12–O43	172.3(3)	O62–Fe28–O61	88.2(3)
O41–Fe12–O43	91.0(3)	O94–Fe28–O61	97.4(3)
O10–Fe12–O8	88.5(3)	O62–Fe28–O84	93.5(3)
O41–Fe12–O8	95.6(3)	O94–Fe28–O84	91.2(3)
O43–Fe12–O8	92.9(3)	O61–Fe28–O84	171.0(3)
O10–Fe12–O46	92.1(3)	O62–Fe28–O102	171.2(3)
O41–Fe12–O46	91.1(3)	O94–Fe28–O102	91.1(3)
O43–Fe12–O46	85.6(3)	O61–Fe28–O102	92.8(3)
O8–Fe12–O46	173.2(3)	O84–Fe28–O102	84.2(3)
O10–Fe12–O19	75.8(3)	O62–Fe28–O68	75.4(3)
O41–Fe12–O19	171.6(3)	O94–Fe28–O68	172.7(3)
O43–Fe12–O19	96.9(3)	O61–Fe28–O68	81.0(3)
O8–Fe12–O19	81.3(2)	O84–Fe28–O68	90.9(3)
O46–Fe12–O19	92.3(3)	O102–Fe28–O68	96.1(3)
O12–Fe13–O9	100.2(3)	O64–Fe29–O63	100.2(3)
O12–Fe13–O8	112.5(3)	O64–Fe29–O59	114.3(3)
O9–Fe13–O8	81.9(2)	O63–Fe29–O59	81.6(3)
O12–Fe13–O11	114.3(3)	O64–Fe29–O61	113.3(3)
O9–Fe13–O11	82.0(2)	O63–Fe29–O61	82.4(3)
O8–Fe13–O11	132.4(3)	O59–Fe29–O61	131.8(3)
O12–Fe13–O19	117.7(3)	O64–Fe29–O70	118.3(3)
O9–Fe13–O19	142.1(3)	O63–Fe29–O70	141.5(3)
O8–Fe13–O19	83.1(3)	O59–Fe29–O70	82.7(3)
O11–Fe13–O19	82.8(3)	O61–Fe29–O70	82.3(3)
O13–Fe14–O18	173.4(3)	O58–Fe30–O96	97.0(3)
O13–Fe14–O12	98.2(3)	O58–Fe30–O59	89.3(3)
O18–Fe14–O12	81.3(3)	O96–Fe30–O59	96.3(3)
O13–Fe14–O35	90.5(3)	O58–Fe30–O97	171.2(3)
O18–Fe14–O35	89.3(3)	O96–Fe30–O97	91.4(3)
O12–Fe14–O35	169.0(3)	O59–Fe30–O97	92.2(3)
O13–Fe14–O50	94.8(3)	O58–Fe30–O88	92.1(3)
O18–Fe14–O50	91.8(3)	O96–Fe30–O88	90.4(3)
O12–Fe14–O50	93.3(3)	O59–Fe30–O88	172.9(3)
O35–Fe14–O50	92.7(3)	O97–Fe30–O88	85.3(3)
O13–Fe14–O7	94.6(3)	O58–Fe30–O67	75.8(3)
O18–Fe14–O7	78.8(3)	O96–Fe30–O67	172.3(3)
O12–Fe14–O7	80.1(2)	O59–Fe30–O67	81.0(3)
O35–Fe14–O7	92.5(3)	O97–Fe30–O67	96.0(3)
O50–Fe14–O7	169.2(3)	O88–Fe30–O67	92.6(3)
O10–Fe15–O11	88.7(3)	O60–Fe31–O92	97.0(3)

O10–Fe15–O39	97.5(3)	O60–Fe31–O61	88.7(3)
O11–Fe15–O39	97.0(3)	O92–Fe31–O61	96.1(3)
O10–Fe15—O47	92.6(3)	O60–Fe31–O101	172.4(3)
O11–Fe15–O47	171.0(3)	O92–Fe31–O101	90.3(3)
O39–Fe15–O47	91.6(3)	O61–Fe31–O101	92.4(3)
O10–Fe15–O48	171.8(3)	O60–Fe31–O100	92.0(3)
O11–Fe15–O48	92.2(3)	O92–Fe31–O100	91.0(3)
O39–Fe15–O48	90.5(3)	O61–Fe31–O100	172.7(3)
O47–Fe15–O48	85.2(3)	O101–Fe31–O100	86.0(3)
O10–Fe15–O19	74.7(3)	O60–Fe31–O70	75.9(3)
O11–Fe15–O19	80.8(2)	O92–Fe31–O70	172.3(3)
O39–Fe15–O19	171.9(3)	O61–Fe31–O70	81.0(3)
O47–Fe15–O19	91.0(3)	O101–Fe31–O70	96.9(3)
O48–Fe15–O19	97.4(3)	O100–Fe31–O70	92.2(3)
O13–Fe16–O51	98.3(3)	O60–Fe32–O59	88.8(3)
O13–Fe16–O49	170.9(3)	O60–Fe32–O90	97.8(3)
O51–Fe16–O49	90.7(3)	O59–Fe32–O90	97.3(3)
O13–Fe16–O11	87.6(3)	O60–Fe32–O98	170.2(3)
O51–Fe16–O11	95.3(3)	O59–Fe32–O98	92.8(3)
O49–Fe16–O11	92.8(3)	O90–Fe32–O98	91.6(3)
O13–Fe16–O37	92.0(3)	O60–Fe32–O99	92.0(3)
O51–Fe16–O37	91.5(3)	O59–Fe32–O99	171.6(3)
O49–Fe16–O37	86.5(3)	O90–Fe32–O99	90.9(3)
O11–Fe16–O37	173.2(3)	O98–Fe32–O99	85.0(3)
O13–Fe16–O17	75.8(3)	O60–Fe32–O70	75.6(3)
O51–Fe16–O17	172.9(3)	O59–Fe32–O70	80.7(3)
O49–Fe16–O17	95.3(3)	O90–Fe32–O70	173.1(3)
O11–Fe16–O17	80.6(2)	O98–Fe32–O70	95.1(3)
O37–Fe16–O17	92.7(3)	O99–Fe32–O70	91.4(3)
3 3thf			
Fe1–O3	1.897(4)	Fe9–O9	1.913(4)
Fe1–O14	1.982(4)	Fe9–O40	1.987(4)
Fe1–O2	2.015(4)	Fe9–O5	1.989(3)
Fe1–O22	2.044(4)	Fe9–O31	2.002(4)
Fe1–O20	2.051(4)	Fe9–O49	2.022(4)
Fe1–O1	2.086(4)	Fe9–O15	2.142(4)
Fe2–O4	1.901(4)	Fe10–O12	1.796(4)
Fe2–O14	1.987(4)	Fe10–O13	1.968(3)
Fe2–O1	2.029(4)	Fe10–O6	2.028(4)
Fe2–O26	2.035(4)	Fe10–O5	2.035(3)
Fe2–O24	2.040(4)	Fe10–O17	2.054(4)
Fe2–O2	2.075(4)	Fe11–O10	1.928(4)
Fe3–O3	1.919(4)	Fe11–O6	1.987(4)
Fe3–O5	1.982(4)	Fe11–O42	1.972(4)

Fe3−O21	1.995(4)	Fe11−O33	2.012(4)
Fe3−O30	2.012(4)	Fe11−O44	2.019(4)
Fe3−O28	2.013(4)	Fe11−O16	2.151(4)
Fe3−O17	2.164(4)	Fe12−O9	1.919(4)
Fe4−O3	1.919(4)	Fe12−O7	1.982(3)
Fe4−O23	1.982(4)	Fe12−O50	1.992(4)
Fe4−O6	1.990(4)	Fe12−O37	1.997(4)
Fe4−O32	1.996(4)	Fe12−O48	2.007(4)
Fe4−O29	2.016(4)	Fe12−O15	2.180(4)
Fe4−O17	2.149(4)	Fe13−O11	1.802(4)
Fe5−O1	1.798(4)	Fe13−O13	1.976(4)
Fe5−O13	1.971(3)	Fe13−O7	2.021(3)
Fe5−O5	2.028(4)	Fe13−O8	2.028(4)
Fe5−O7	2.039(4)	Fe13−O18	2.067(3)
Fe5−O15	2.058(4)	Fe14−O10	1.916(4)
Fe6−O2	1.803(4)	Fe14-O8	1.990(4)
Fe6−O13	1.962(3)	Fe14-O46	1.995(4)
Fe6−O6	2.029(4)	Fe14−O39	2.006(4)
Fe6−O8	2.032(4)	Fe14−O45	2.009(4)
Fe6−O16	2.053(4)	Fe14−O16	2.168(4)
Fe7−O4	1.911(4)	Fe15−O9	1.904(4)
Fe7−O25	1.983(4)	Fe15−O19	1.993(4)
Fe7−O7	1.991(4)	Fe15−O41	2.023(4)
Fe7−O36	1.996(4)	Fe15−O51	2.027(4)
Fe7−O34	2.029(4)	Fe15−O12	2.035(4)
Fe7−O18	2.136(4)	Fe15−O11	2.070(4)
Fe8−O4	1.927(4)	Fe16−O10	1.903(4)
Fe8−O27	1.982(4)	Fe16−O19	1.987(4)
Fe8−O8	1.987(4)	Fe16−O11	2.029(3)
Fe8−O35	2.009(4)	Fe16−O43	2.032(4)
Fe8−O38	2.011(4)	Fe16−O47	2.042(4)
Fe8−O18	2.167(4)	Fe16−O12	2.075(4)
O3−Fe1−O14	176.69(16)	O9−Fe9−O40	97.33(16)
O3−Fe1−O2	98.96(15)	O9−Fe9−O5	88.25(15)
O14−Fe1−O2	79.33(15)	O40−Fe9−O5	96.33(15)
O3−Fe1−O22	92.27(15)	O9−Fe9−O31	170.54(16)
O14−Fe1−O22	90.69(15)	O40−Fe9−O31	91.86(16)
O2−Fe1−O22	94.41(15)	O5−Fe9−O31	92.99(15)
O3−Fe1−O20	91.25(16)	O9−Fe9−O49	92.33(16)
O14−Fe1−O20	90.14(16)	O40−Fe9−O49	89.79(16)
O2−Fe1−O20	167.87(15)	O5−Fe9−O49	173.72(15)
O22−Fe1−O20	91.70(16)	O31−Fe9−O49	85.43(16)
O3−Fe1−O1	96.43(15)	O9−Fe9−O15	75.82(14)

O14–Fe1–O1	80.51(15)	O40–Fe9–O15	172.74(15)
O2–Fe1–O1	80.21(14)	O5–Fe9–O15	81.23(14)
O22–Fe1–O1	170.37(15)	O31–Fe9–O15	95.09(15)
O20–Fe1–O1	92.21(15)	O49–Fe9–O15	92.85(15)
O4–Fe2–O14	173.84(16)	O12–Fe10–O13	100.36(15)
O4–Fe2–O1	97.52(15)	O12–Fe10–O6	113.62(15)
O14–Fe2–O1	81.79(15)	O13–Fe10–O6	81.77(14)
O4–Fe2–O26	91.30(16)	O12–Fe10–O5	114.49(15)
O14–Fe2–O26	88.83(16)	O13–Fe10–O5	81.69(14)
O1–Fe2–O26	169.55(16)	O6–Fe10–O5	131.10(14)
O4–Fe2–O24	93.82(15)	O12–Fe10–O17	119.01(15)
O14–Fe2–O24	92.33(16)	O13–Fe10–O17	140.62(15)
O1–Fe2–O24	92.36(15)	O6–Fe10–O17	82.69(14)
O26–Fe2–O24	92.64(16)	O5–Fe10–O17	81.79(14)
O4–Fe2–O2	96.05(15)	O10–Fe11–O42	95.76(16)
O14–Fe2–O2	77.79(15)	O10–Fe11–O6	89.67(15)
O1–Fe2–O2	80.17(14)	O42–Fe11–O6	97.46(15)
O26–Fe2–O2	93.38(15)	O10–Fe11–O33	172.41(16)
O24–Fe2–O2	168.31(16)	O42–Fe11–O33	91.12(16)
O3–Fe3–O5	89.54(15)	O6–Fe11–O33	92.64(16)
O3–Fe3–O21	96.63(16)	O10–Fe11–O44	92.40(16)
O5–Fe3–O21	98.48(15)	O42–Fe11–O44	90.77(16)
O3–Fe3–O30	171.80(16)	O6–Fe11–O44	171.27(16)
O5–Fe3–O30	92.65(15)	O33–Fe11–O44	84.27(16)
O21–Fe3–O30	90.87(16)	O10–Fe11–O16	76.24(14)
O3–Fe3–O28	92.06(15)	O42–Fe11–O16	171.77(15)
O5–Fe3–O28	171.55(16)	O6–Fe11–O16	80.77(14)
O21–Fe3–O28	89.58(16)	O33–Fe11–O16	96.99(15)
O30–Fe3–O28	84.65(16)	O44–Fe11–O16	91.49(15)
O3–Fe3–O17	75.76(15)	O9–Fe12–O7	88.29(15)
O5–Fe3–O17	80.33(14)	O9–Fe12–O50	97.80(16)
O21–Fe3–O17	172.27(15)	O7–Fe12–O50	97.25(15)
O30–Fe3–O17	96.81(15)	O9–Fe12–O37	169.18(16)
O28–Fe3–O17	92.02(15)	O7–Fe12–O37	92.12(15)
O3–Fe4–O23	97.28(16)	O50–Fe12–O37	92.87(16)
O3–Fe4–O6	88.99(15)	O9–Fe12–O48	92.85(15)
O23–Fe4–O6	96.98(16)	O7–Fe12–O48	171.67(16)
O3–Fe4–O32	170.87(16)	O50–Fe12–O48	90.78(16)
O23–Fe4–O32	91.54(16)	O37–Fe12–O48	85.22(15)
O6–Fe4–O32	92.30(15)	O9–Fe12–O15	74.78(14)
O3–Fe4–O29	91.56(16)	O7–Fe12–O15	80.77(14)
O23–Fe4–O29	89.51(16)	O50–Fe12–O15	172.33(15)
O6–Fe4–O29	173.37(16)	O37–Fe12–O15	94.61(15)
O32–Fe4–O29	86.13(16)	O48–Fe12–O15	91.56(15)

O3–Fe4–O17	76.11(15)	O11–Fe13–O13	100.16(15)
O23–Fe4–O17	173.12(15)	O11–Fe13–O7	113.65(15)
O6–Fe4–O17	81.20(14)	O13–Fe13–O7	81.78(14)
O32–Fe4–O17	95.15(15)	O11–Fe13–O8	113.87(15)
O29–Fe4–O17	92.52(15)	O13–Fe13–O8	81.63(14)
O1–Fe5–O13	99.84(15)	O7–Fe13–O8	131.62(14)
O1–Fe5–O5	112.73(16)	O11–Fe13–O18	120.01(15)
O13–Fe5–O5	81.79(14)	O13–Fe13–O18	139.83(14)
O1–Fe5–O7	114.76(16)	O7–Fe13–O18	82.05(14)
O13–Fe5–O7	81.45(14)	O8–Fe13–O18	82.19(14)
O5–Fe5–O7	131.57(15)	O10–Fe14–O8	89.22(15)
O1–Fe5–O15	119.71(15)	O10–Fe14–O46	96.92(16)
O13–Fe5–O15	140.45(15)	O8–Fe14–O46	97.25(15)
O5–Fe5–O15	82.37(14)	O10–Fe14–O39	169.87(16)
O7–Fe5–O15	82.48(14)	O8–Fe14–O39	92.02(15)
O2–Fe6–O13	100.20(15)	O46–Fe14–O39	92.90(16)
O2–Fe6–O6	113.71(16)	O10–Fe14–O45	92.71(15)
O13–Fe6–O6	81.85(14)	O8–Fe14–O45	171.25(16)
O2–Fe6–O8	114.00(16)	O46–Fe14–O45	90.99(16)
O13–Fe6–O8	81.87(14)	O39–Fe14–O45	84.61(15)
O6–Fe6–O8	131.51(15)	O10–Fe14–O16	76.07(14)
O2–Fe6–O16	119.02(15)	O8–Fe14–O16	80.48(14)
O13–Fe6–O16	140.78(15)	O46–Fe14–O16	172.60(15)
O6–Fe6–O16	82.22(14)	O39–Fe14–O16	94.21(15)
O8–Fe6–O16	82.37(14)	O45–Fe14–O16	91.70(15)
O4–Fe7–O25	97.05(16)	O9–Fe15–O19	174.15(16)
O4–Fe7–O7	88.48(15)	O9–Fe15–O41	93.49(16)
O25–Fe7–O7	96.22(15)	O19–Fe15–O41	92.35(15)
O4–Fe7–O36	171.83(16)	O9–Fe15–O51	90.73(16)
O25–Fe7–O36	91.03(16)	O19–Fe15–O51	89.51(15)
O7–Fe7–O36	91.84(15)	O41–Fe15–O51	91.17(16)
O4–Fe7–O34	92.59(15)	O9–Fe15–O12	97.16(15)
O25–Fe7–O34	90.11(16)	O19–Fe15–O12	82.23(15)
O7–Fe7–O34	173.41(16)	O41–Fe15–O12	92.37(15)
O36–Fe7–O34	86.18(15)	O51–Fe15–O12	171.14(16)
O4–Fe7–O18	76.40(14)	O9–Fe15–O11	96.35(15)
O25–Fe7–O18	172.89(15)	O19–Fe15–O11	77.81(14)
O7–Fe7–O18	81.00(14)	O41–Fe15–O11	168.49(15)
O36–Fe7–O18	95.59(15)	O51–Fe15–O11	94.67(15)
O34–Fe7–O18	92.92(14)	O12–Fe15–O11	80.50(14)
O4–Fe8–O27	96.67(16)	O10–Fe16–O19	176.57(15)
O4–Fe8–O8	88.78(15)	O10–Fe16–O11	98.00(15)
O27–Fe8–O8	97.51(16)	O19–Fe16–O11	78.92(15)
O4–Fe8–O35	92.49(15)	O10–Fe16–O43	92.05(15)

O27–Fe8–O35	90.31(16)	O19–Fe16–O43	90.84(15)
O8–Fe8–O35	171.88(16)	O11–Fe16–O43	167.84(15)
O4–Fe8–O38	171.31(16)	O10–Fe16–O47	91.75(16)
O27–Fe8–O38	91.66(16)	O19–Fe16–O47	89.98(15)
O8–Fe8–O38	92.58(15)	O11–Fe16–O47	93.83(15)
O35–Fe8–O38	84.97(16)	O43–Fe16–O47	92.69(15)
O4–Fe8–O18	75.33(14)	O10–Fe16–O12	96.67(15)
O27–Fe8–O18	171.77(15)	O19–Fe16–O12	81.38(15)
O8–Fe8–O18	80.64(14)	O11–Fe16–O12	80.53(14)
O35–Fe8–O18	91. 92(14)	O43–Fe16–O12	91.56(15)
O38–Fe8–O18	96.42(15)	O47–Fe16–O12	170.43(15)

Table S3. BVS values

$[Fe_{11}O_6(OH)_6(O_2CCMe_3)_{15}]$ (**1**)				$[Fe_{11}O_6(EtO)_9(O_2CCMe_3)_{12}]$ (**2·1.5CH$_2$Cl$_2$·MeCN**)	
Fe1	3.024	Fe12	2.887	Fe1	2.994
Fe2	3.150	Fe13	3.101	Fe2	3.042
Fe3	3.203	Fe14	3.041	Fe3	3.075
Fe4	2.936	Fe15	3.224	Fe4	3.044
Fe5	3.056	Fe16	3.137	Fe5	3.082
Fe6	3.162	Fe17	2.960	Fe6	3.057
Fe7	2.973	Fe18	2.958	Fe7	3.071
Fe8	3.082	Fe19	3.071	Fe8	2.992
Fe9	3.098	Fe20	3.219	Fe9	3.221
Fe10	3.081	Fe21	3.239	Fe10	3.193
Fe11	3.190	Fe22	3.108	Fe11	3.222
$[Fe_{16}O_{13}(EtO)_6(O_2CCMe_3)_{16}]$ (**3**)					
3·0.25thf				**3·3thf**	
Fe1	3.077	Fe17	3.024	Fe1	3.075
Fe2	3.014	Fe18	3.004	Fe2	3.082
Fe3	3.057	Fe19	3.059	Fe3	3.070
Fe4	3.043	Fe20	3.100	Fe4	3.106
Fe5	2.864	Fe21	2.846	Fe5	2.870
Fe6	2.816	Fe22	2.834	Fe6	2.886
Fe7	3.074	Fe23	3.100	Fe7	3.118
Fe8	2.862	Fe24	2.847	Fe8	3.072
Fe9	3.091	Fe25	3.058	Fe9	3.105
Fe10	3.079	Fe26	3.070	Fe10	2.885
Fe11	3.055	Fe27	3.042	Fe11	3.085
Fe12	3.072	Fe28	3.066	Fe12	3.083
Fe13	2.844	Fe29	2.845	Fe13	2.867
Fe14	3.047	Fe30	3.042	Fe14	3.070
Fe15	3.045	Fe31	3.054	Fe15	3.090
Fe16	3.049	Fe32	3.056	Fe16	3.069

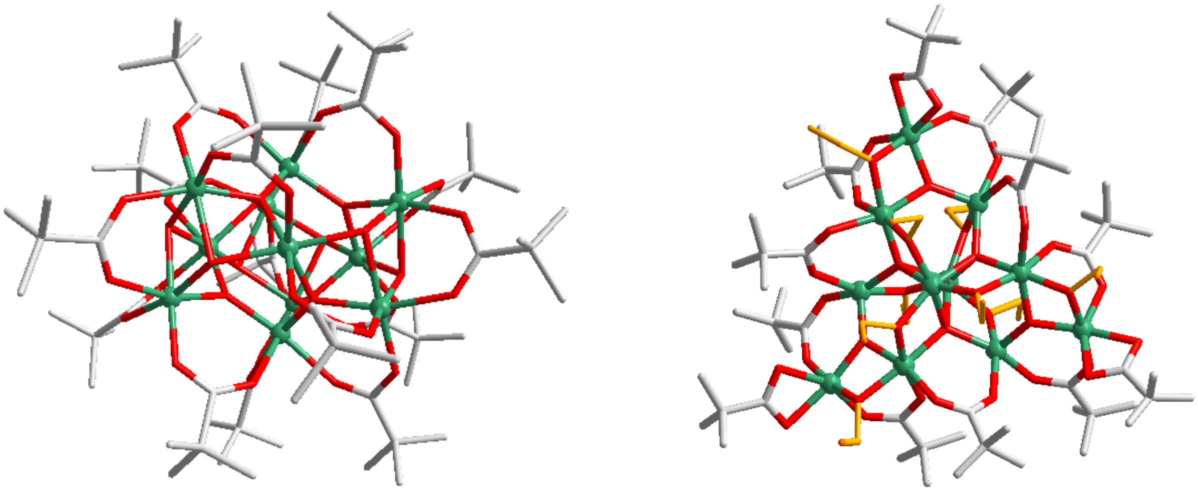


Figure S1. Structures of the Fe_{11} metallic core in **1** (left) and **$2 \cdot 1.5CH_2Cl_2 \cdot MeCN$** (right). Hydrogen atoms and solvent molecules are omitted for clarity. Color scheme: Fe, dark green spheres; C, grey sticks; O, red sticks; C atoms of the coordinated ethanol molecules are shown as yellow sticks.

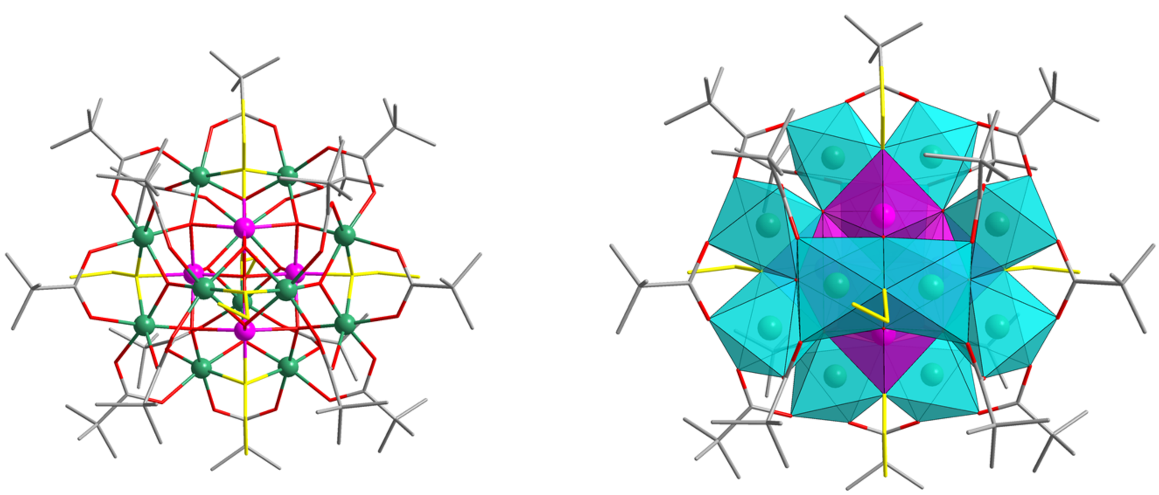
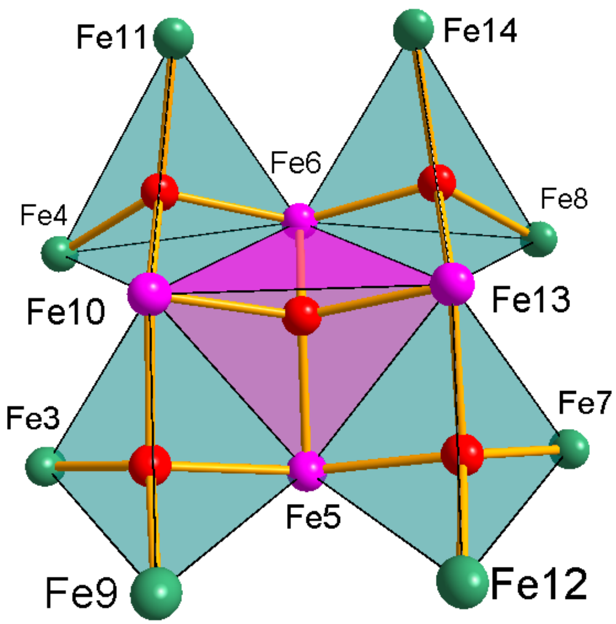
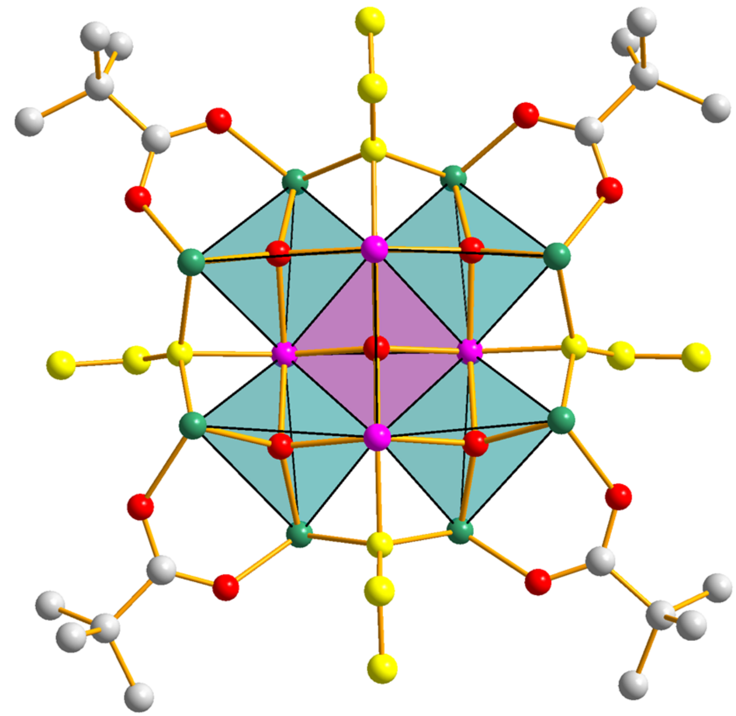


Figure S2. A ball-and-stick (left) and polyhedral (right) representation of the Fe_{16} metallic core in **3**. Hydrogen atoms and solvent molecules are omitted for clarity. Color scheme: hexa-coordinated Fe, dark green spheres or light blue polyhedra; penta-coordinated Fe, pink spheres or pink polyhedra; C, grey sticks; O, red sticks; ethanol molecules are shown as yellow sticks.

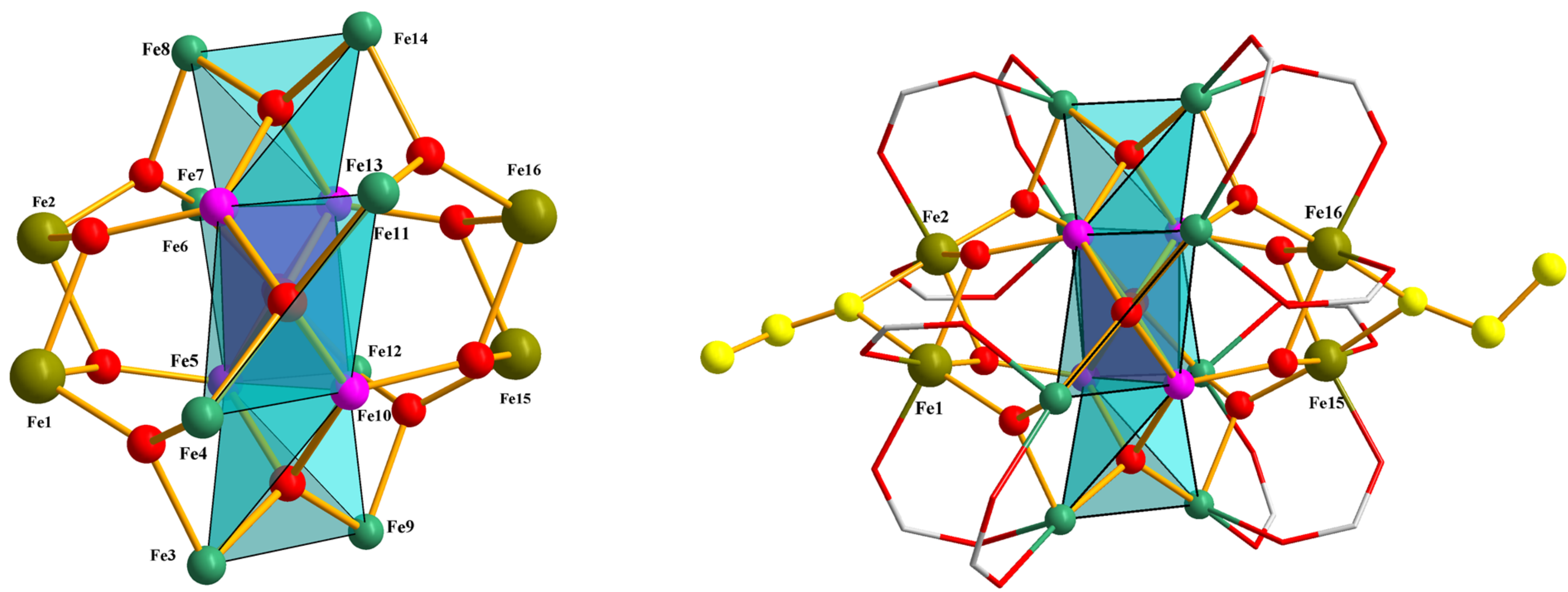
The metallic Fe_{16} core in **3** can be also described as an array of five [Fe_4–(μ_4-O)] tetrahedra, with the central tetrahedron [defined by Fe5, Fe6, F10, and Fe13 atoms as shown in Fig. S3a] surrounding by other four edge-sharing tetrahedral units. The peripheral Fe atoms of the tetrahedra are further connected to each other through pivalate or μ_3-ethoxo groups (Fig. S3b). Two iron atoms [Fe1 and Fe2] from one side and other two Fe atoms [Fe15 and Fe16] from other side attached to this conglomerate by four μ_3-O atoms with the formation of eight [Fe_3–(μ_3-O)] triangular units (Fig. S3c). Four of these units (two on each side) are essentially scalene with two longer Fe…Fe separations of 3.388(1) – 3.423(1) Å and one short being 3.081(1) – 3.093(1) Å, and their central μ_3-O atoms [O3, O4, O9 and O10] lying almost in their Fe_3 plane [0.112, 0.150, 0.162 and 0.113 Å, respectively]. Each pair of Fe atoms within triangle units is additionally bridged by a carboxylate (Fig. S3d). In other four [Fe_3–(μ_3-O)] triangular units Fe atoms are also form a scalene triangle with one short edge [Fe1…Fe2, 2.719(1) and Fe15…Fe16, 2.713(1) Å, the short separation corresponds to the one additionally linked by a μ_2-ethoxo ligand] and two longer ones being in the range of 3.345(1) – 3.412(1) Å, but their μ_3-O atoms [O1, O2, O11 and O12] distinctly out of their Fe_3 plane [0.627, 0.626, 0.629 and 0.630 Å, respectively].



a



b



c **d**

Figure S3. Arrangement of the 16 Fe atoms in **3**: (a) an array of five [Fe_4–(μ_4-O)] tetrahedral units in **3**; (b) view showing 12 Fe atoms forming the tetrahedral units with coordinated pivalate and μ_3-ethoxo ligands colored in yellow; (c) view showing all 16 Fe atoms in the metallic core of **3** emphasizing the attachment of four Fe atoms (in light green) to the array of tetrahedral units with (d) 12 bridging pivalates (sticks) in four [Fe_3–(μ_3-O)] triangular units and μ_2-ethoxo groups colored in yellow.

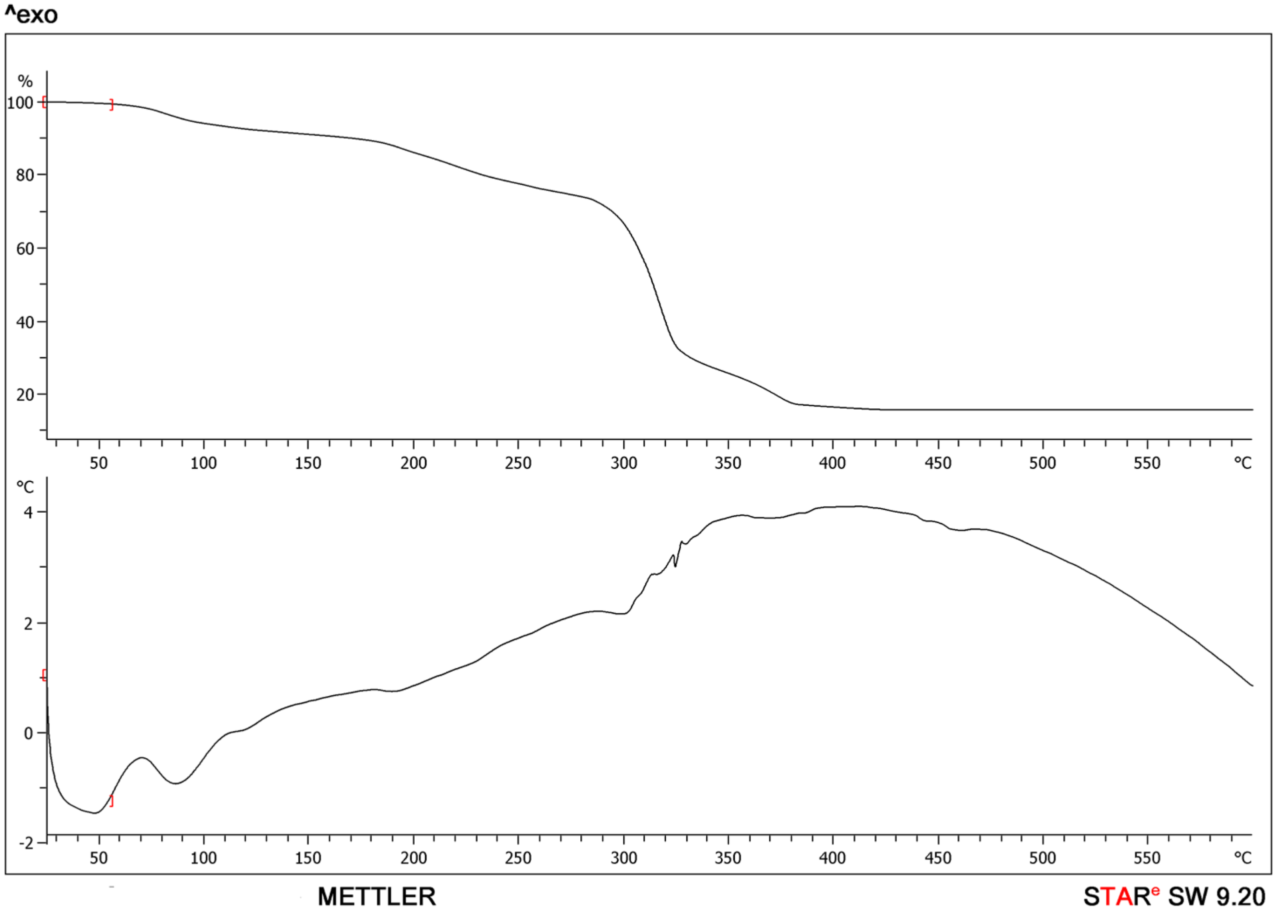


Figure S4. TGA/DTA graphs for $[Fe_3O(O_2CCMe_3)_6(EtOH)_3]NO_3{\cdot}EtOH$.

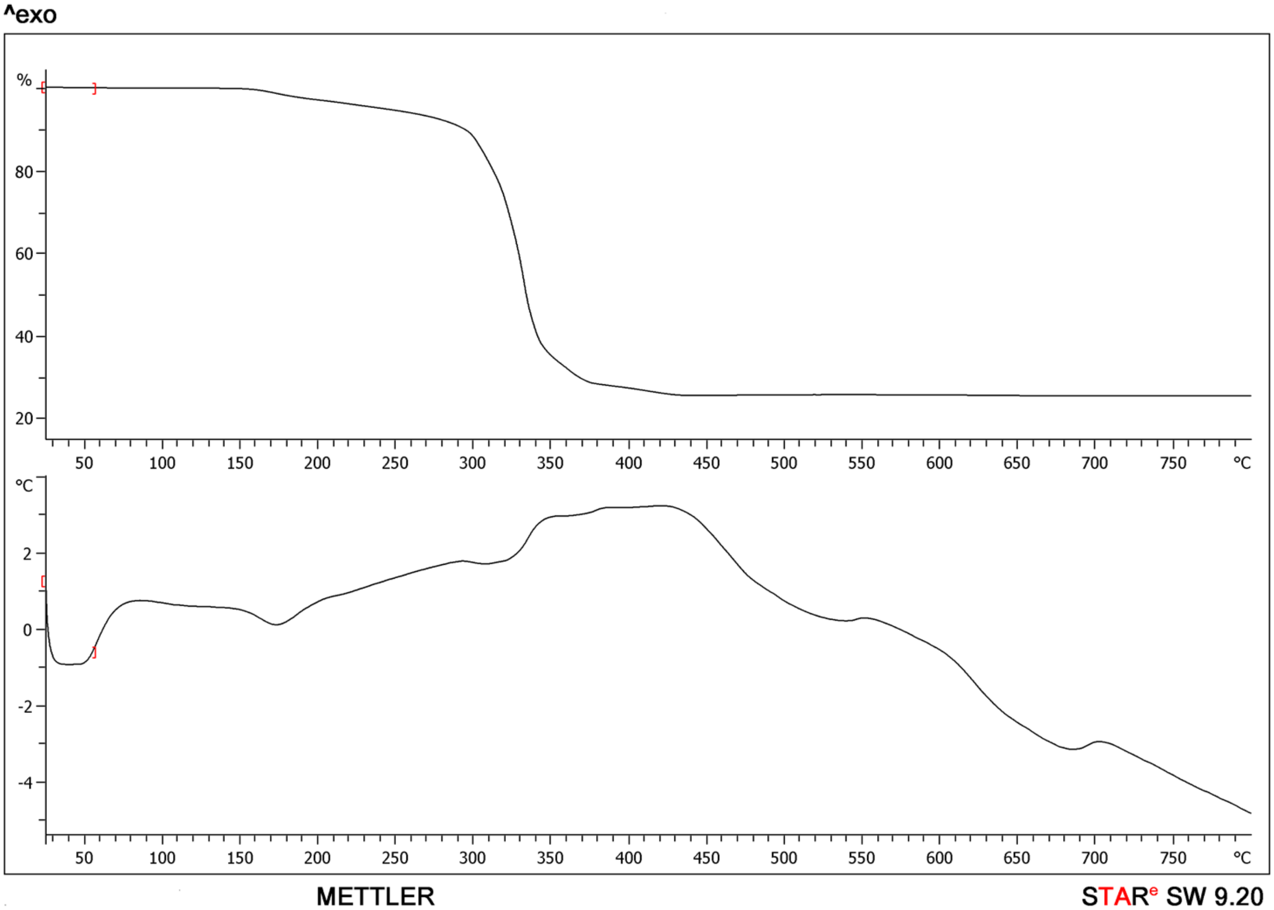


Figure S5. TGA/DTA graphs for $[Fe_{11}O_6(OH)_6(O_2CCMe_3)_{15}]$ (**1**).

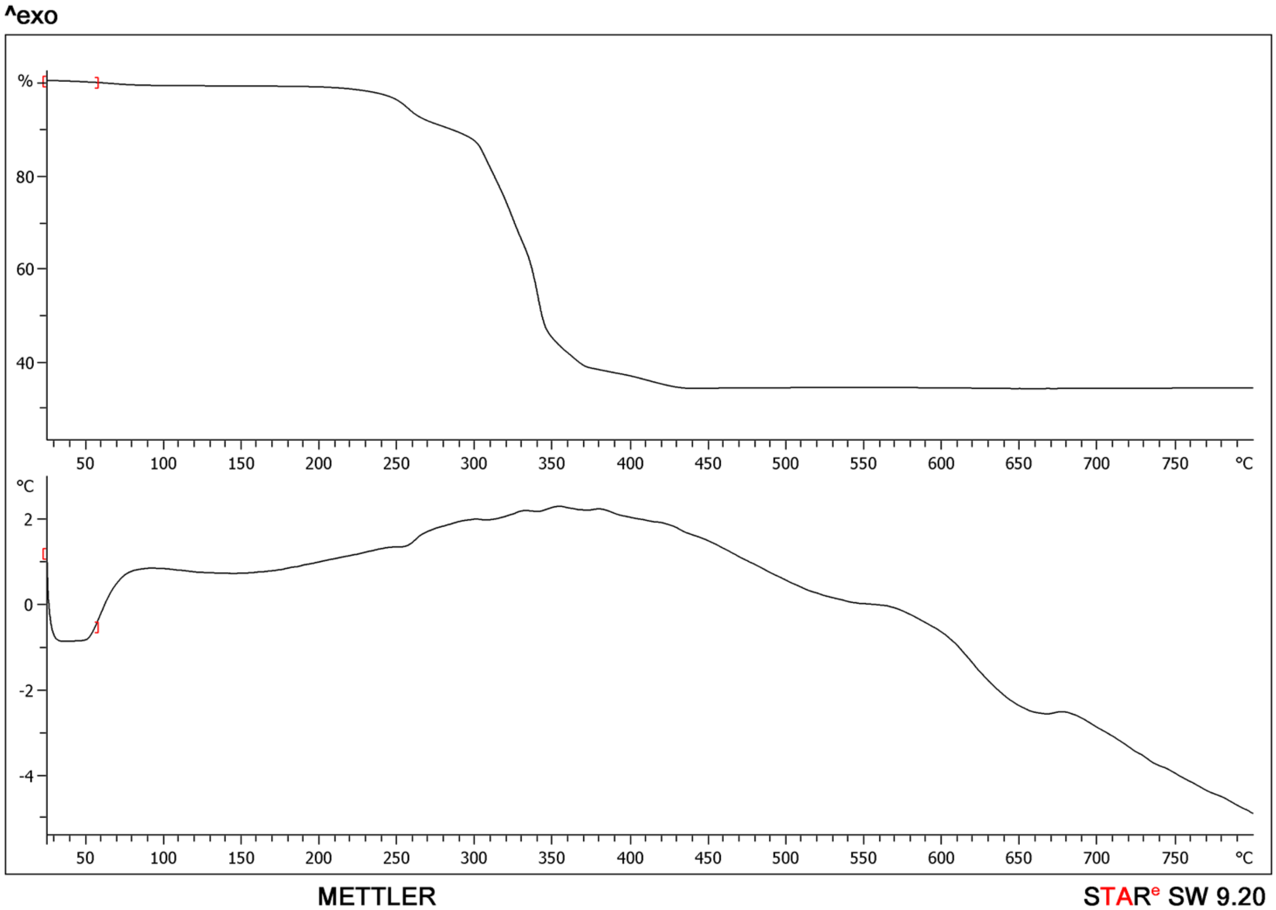


Figure S6. TGA/DTA graphs for $[Fe_{11}O_6(EtO)_9(O_2CCMe_3)_{12}]$ (**2·1.5CH$_2$Cl$_2$·MeCN**).

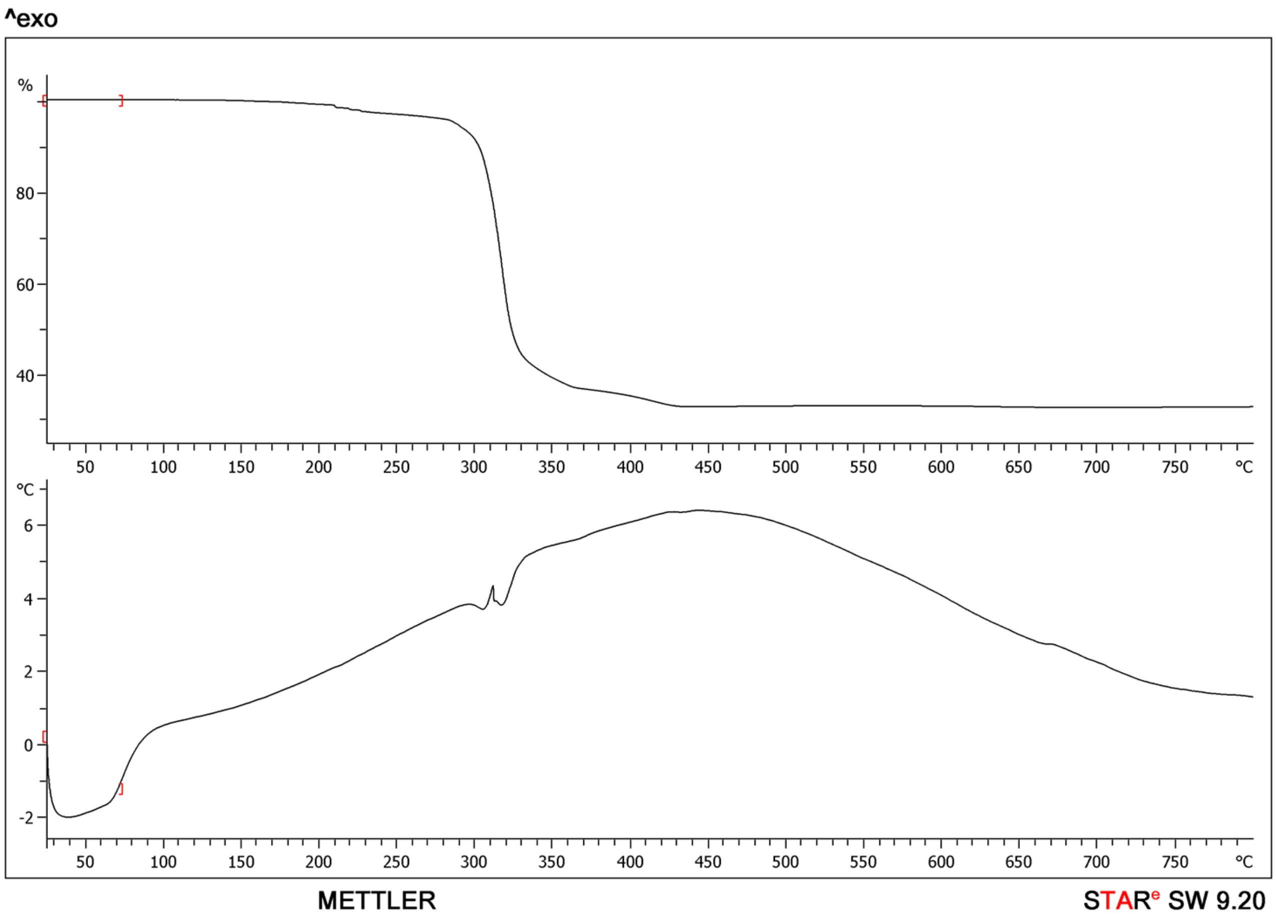


Figure S7. TGA/DTA graphs for $[Fe_{16}O_{13}(EtO)_6(O_2CCMe_3)_{16}]$ (**3**) [**3·0.25thf**].

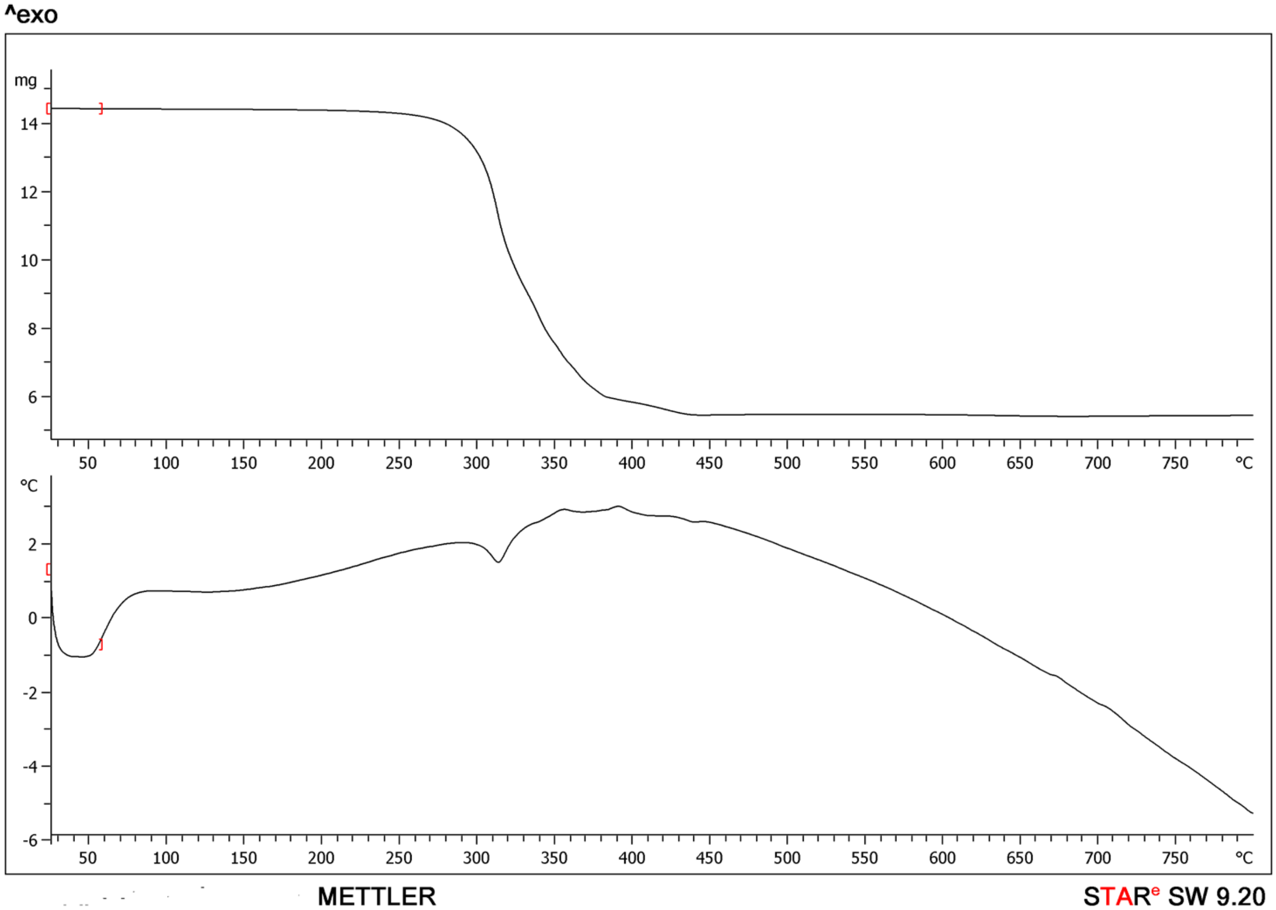


Figure S8. TGA/DTA graphs for $[Fe_{16}O_{13}(EtO)_6(O_2CCMe_3)_{16}]$·(**3**) [**3·3thf**].