Electronic Supplementary Material (ESI) for Dalton Transactions

Supporting Information

Conversion of $\{Fe(NO)_2\}^{10}$ Dinitrosyl Iron to Nitrato Iron(III) Species by Molecular Oxygen

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Contents

Experimental

General All syntheses and manipulations prior to the addition of dioxygen were performed under an inert atmosphere using an MBraun glovebox (< 0.1 ppm O_2, <0.1 ppm H_2O) or standard Schlenk line techniques unless stated otherwise. Solvents were purified by passing through alumina columns under an Ar atmosphere (MBraun solvent purification system) and stored over 4 Å molecular sieves prior to use. 2,4-Di-*tert*-butylphenol (DBP), diphenyldisulfide (PhSSPh), and 2,9-dimethyl-1,10-phenanthroline were purchased from Aldrich and used as received. $^{18}O_2$ gas (98% ^{18}O enriched) and $H_2{}^{18}O$ (97.6% ^{18}O enriched) were purchased from ICON. 2,4-di-*t*-butyl-6-nitrophenol (NO_2DBP)[1] and 2,2' dihydroxy-3,3',5,5'-tetra-*t*-butyl-1,1'biphenyl ($(DBP)_2$)[2] were synthesized according to literature procedures. Elemental analysis was carried out in duplicate by Columbia Analytical Services (Tucson, AZ) and Midwest Microlab (Indianapolis, IN). GC-MS data were recorded using a Hewlett-Packard (Agilent) GCD 1800C GC-MS spectrometer and ESI data were recorded on a Thermo LCQ Deca XP Max ion trap mass spectrometer with a Shimadzu HPLC system.

Synthesis of [Fe(dmp)$(NO)_2$] (1)

One equivalent of 2,9-dimethyl-1,10-phenanthroline (0.484 g, 0.0023 mol) was added to 0.400 g (0.0023 mol) $Fe(CO)_2(NO)_2$ in 3 mL acetonitrile. The reaction mixture was stirred for 1 hr., at which time brown solid had precipitated from the solution and was collected, rinsed with ether, and dried. This provided us with a 73% yield of the DNIC (0.555 g, 0.0017 mol). IR (KBr): ν_{NO} 1628, 1692 cm^{-1}; UV-Vis (CH_2Cl_2): 278 (31134 M^{-1} cm^{-1}), 294 (20511 M^{-1} cm^{-1}), 334 (3752 M^{-1} cm^{-1}), 381 (3172 M^{-1} cm^{-1}), and 451 (2221 M^{-1} cm^{-1}) nm; ^{1}H NMR (DMSO-d_6) showed 8.69 (d, 2H), 8.15 (s, 2H), 7.89 (d, 2H), and 2.47 (s, 6H) ppm. Anal. Calcd for $C_{14}H_{12}Fe_2N_4O_2$: C, 51.88; H, 3.73; N, 17.29. Found: C, 51.15; H, 3.73; N, 17.07.

Synthesis of [$Fe_2O(NO_3)_4$(dmp)$_2$] (2)

3 equivalents of O_2 were added via gas tight syringe to a solution of 0.042 g (0.13 mmol) [Fe(dmp)$(NO)_2$] in acetonitrile. Upon bubbling oxygen, the dark brown solution immediately became lighter in color and opaque due to some precipitation. The reaction

mixture was stirred for a few moments and then allowed to sit in its Schlenk flask for an hour. At this time, a quantity of black crystalline solid had come out of solution and was isolated by removing solvent and rinsing with additional equivalents of acetonitrile (0.022 g, 0.028 mmol, 43% yield). IR(KBr): 812, 1286, 1510 cm^{-1}. Anal. Calcd for $C_{28}H_{24}Fe_2N_8O_{13}•H_2O$: C, 41.45; H, 3.23; N, 13.83. Found: C, 41.55; H, 3.78; N, 13.70. From this reaction, black crystals of X-ray quality were obtained. The remaining material had concomitantly decomposed to a reddish insoluble precipitate with IR (in KBr) features typical of free nitrate (1384 cm^{-1}).[3] It was possible to separate this from **2** because it was a lighter weight powder and was easily rinsed away from the crystals with additional solvent.

Electrochemical Measurements

A cyclic voltammogram of a 0.01 M solution of $[Fe(dmp)(NO)_2]$ (**1**) in dichloromethane with 0.1 M Bu_4NPF_6 as the supporting electrolyte was recorded at room temperature. A 3-electrode system comprised of a glassy carbon working electrode, platinum wire auxiliary electrode, and a Ag/Ag^+ reference electrode was employed and data were referenced to the ferrocene/ferrocenium couple at 0.00 V. Electrochemical measurements were performed in a glovebox. A reversible redox wave at $E_{1/2}$ = -402 mV was observed and attributed to $\{Fe(NO)_2\}^{9/10}$, an assignment supported by an analogous system in the literature.[4]

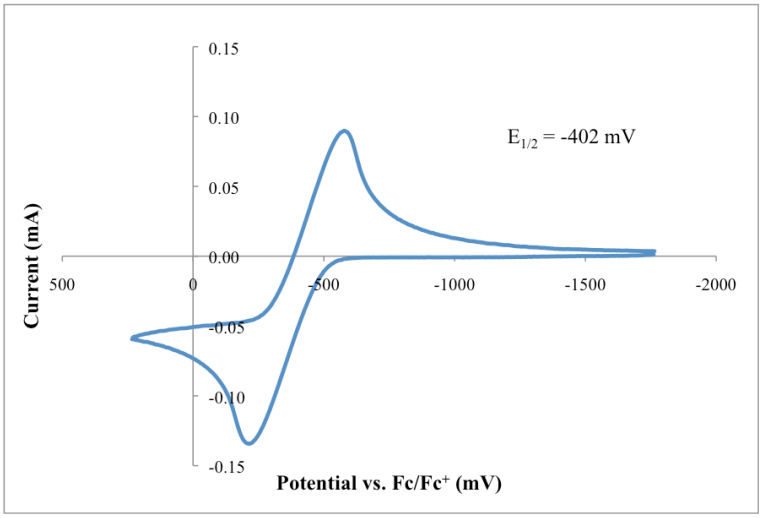

Figure S1. Cyclic voltammogram of $[Fe(dmp)(NO)_2]$ (**1**) in dichloromethane showing a reversible $\{Fe(NO)_2\}^{9/10}$ redox couple at $E_{1/2}$ = – 402 mV.

Reactivity with 2,4 Di-*t*-butylphenol (GC-MS analysis)

GC-MS calibration curves were made for DBP, NO_2DBP and $(DBP)_2$ in the 0.1 mM to 1 mM range and 0.4 mM of PhSSPh was used as an internal standard to provide reproducible data from day to day measurements. Calibration curves were based on peak area of the compound.

For the reaction of [Fe(dmp)$(NO)_2$] (**1**) with O_2 in the presence of DBP, O_2 was bubbled for 30 sec. through a 1.0 mL solution of 2.6 mM [Fe(TMEDA)$(NO)_2$] (**1**) and 1 equiv. DBP in CH_2Cl_2 until a color change from dark brown to pale yellow was observed. The mixture was allowed to stand overnight, during which time a small amount of precipitation occurred. The precipitate was removed by filtration through silica and washed with an additional 1.0 mL of CH_2Cl_2 to ensure collection of all DBP derivatives. To account for volume changes due to rinsing, as well as solvent evaporation during the course of the experiment, the total volume of the filtrate was then measured and used to correct the initial concentration of DBP. 0.5 mL of the solution was diluted to 1.0 mL with CH_2Cl_2 in order to ensure that its concentration was within range of the calibration curve and to this was added 0.2 mL of 2.3 mM PhSSPh in CH_2Cl_2. This solution (0.4 mM PhSSPh, 0.8 mM initial DBP) was used as is for GC-MS data collection. The area of each peak (DBP, NO_2DBP and $(DBP)_2$) was corrected based on the ratio of the average area of PhSSPh in the respective calibration curves and the area of PhSSPh in each sample. The experiment was conducted in triplicate and the percent yield of NO_2DBP was 65±3% and for $(DBP)_2$, 0.3±0.4%.

^{18}O Labeling Experiments

Synthesis of **2** with $^{18}O_2$

The reaction and subsequent workup of the experiment was performed in a glovebox (< 0.1 ppm O_2, <0.1 ppm H_2O) to prevent any contamination from atmospheric O_2 and H_2O. The labeling experiment was conducted in the same manner as the reaction of [Fe(dmp)$(NO)_2$] (**1**) with O_2 to produce **2** discussed on page S2 except that, instead of bubbling $^{16}O_2$, 25 mL (3 equiv.) of $^{18}O_2$ (ICON, 98% ^{18}O enriched) were added to the headspace of the stirring solution of **1** in MeCN.

Synthesis of **2** in the presence of $H_2{}^{18}O$

2 was prepared in the same way as discussed on page S2 except that 10 equiv. of $H_2{}^{18}O$ were added to the reaction mixture before bringing out of the glovebox to add O_2.

Crystal Structure Report for $[Fe_2O(NO_3)_4(dmp)_2]$ (2)

A metallic dark black diamond-like specimen of $C_{15}H_{13.50}FeN_{4.50}O_{6.50}$, approximate dimensions 0.300 mm x 0.400 mm x 2.000 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured.

The total exposure time was 3.19 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 13820 reflections to a maximum θ angle of 26.12° (0.81 Å resolution), of which 3314 were independent (average redundancy 4.170, completeness = 98.7%, R_{int} = 6.87%, R_{sig} = 5.92%) and 2427 (73.23%) were greater than $2\sigma(F^2)$. The final cell constants of a = 17.72(3) Å, b = 12.90(2) Å, c = 14.74(3) Å, β = 94.40(3)°, volume = 3359.(11) $Å^3$, are based upon the refinement of the XYZ-centroids of 2593 reflections above 20 σ(I) with 4.688° < 2θ < 45.88°. Data were corrected for absorption effects using the multi-scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.733. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.2537 and 0.7647.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group C 1 2/c 1, with Z = 8 for the formula unit, $C_{15}H_{13.50}FeN_{4.50}O_{6.50}$. The final anisotropic full-matrix least-squares refinement on F^2 with 249 variables converged at R1 = 4.31%, for the observed data and wR2 = 11.72% for all data. The goodness-of-fit was 1.044. The largest peak in the final difference electron density synthesis was 0.349 $e^-/Å^3$ and the largest hole was -0.501 $e^-/Å^3$ with an RMS deviation of 0.073 $e^-/Å^3$. On the basis of the final model, the calculated density was 1.647 g/cm^3 and F(000), 1704 e^-.

Table 1. Sample and crystal data for $[Fe_2O(NO_3)_4(dmp)_2]$ (2).

Identification code	$[Fe_2O(NO_3)_4(dmp)_2]$ (2)	
Chemical formula	$C_{15}H_{13.50}FeN_{4.50}O_{6.50}$	
Formula weight	416.65	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal size	0.300 x 0.400 x 2.000 mm	
Crystal habit	metallic dark black diamond	
Crystal system	monoclinic	
Space group	C 1 2/c 1	
Unit cell dimensions	a = 17.72(3) Å	α = 90°
	b = 12.90(2) Å	β = 94.40(3)°
	c = 14.74(3) Å	γ = 90°
Volume	3359.(11) $Å^3$	
Z	8	
Density (calculated)	1.647 Mg/cm^3	
Absorption coefficient	0.945 mm^{-1}	
F(000)	1704	

Table 2. Data collection and structure refinement for $[Fe_2O(NO_3)_4(dmp)_2]$ (2).

Theta range for data collection	2.34 to 26.12°
Index ranges	-21<=h<=21, -15<=k<=15, -18<=l<=18
Reflections collected	13820
Independent reflections	3314 [R(int) = 0.0687]
Coverage of independent reflections	98.7%
Absorption correction	multi-scan
Max. and min. transmission	0.7647 and 0.2537
Structure solution technique	direct methods
Structure solution program	SHELXS-97 (Sheldrick, 2008)
Refinement method	Full-matrix least-squares on F^2

Refinement program	SHELXL-97 (Sheldrick, 2008)	
Function minimized	$\Sigma w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	3314 / 0 / 249	
Goodness-of-fit on F^2	1.044	
Δ/σ_{max}	0.113	
Final R indices	2427 data; $I>2\sigma(I)$	R1 = 0.0431, wR2 = 0.1021
	all data	R1 = 0.0680, wR2 = 0.1172
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0526P)^2+1.7554P]$ where $P=(F_o^2+2F_c^2)/3$	
Largest diff. peak and hole	0.349 and -0.501 eÅ^{-3}	
R.M.S. deviation from mean	0.073 eÅ^{-3}	

Table 3. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å^2) for $[Fe_2O(NO_3)_4(dmp)_2]$ (2).

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x/a	y/b	z/c	U(eq)
C1	0.48997(17)	0.9951(3)	0.1210(2)	0.0344(8)
C2	0.57370(18)	0.9943(2)	0.1145(2)	0.0314(7)
C3	0.6091(2)	0.9024(3)	0.0922(2)	0.0417(8)
C4	0.6844(2)	0.9015(3)	0.0804(3)	0.0456(9)
C5	0.72562(18)	0.9935(3)	0.0862(2)	0.0358(8)
C6	0.80331(19)	0.9992(3)	0.0680(2)	0.0434(9)
C7	0.84022(18)	0.0895(3)	0.0722(2)	0.0443(9)
C8	0.80392(17)	0.1816(3)	0.0973(2)	0.0367(8)
C9	0.83985(19)	0.2781(3)	0.1022(2)	0.0449(10)
C10	0.80172(19)	0.3625(3)	0.1274(2)	0.0433(9)
C11	0.72624(18)	0.3555(3)	0.1489(2)	0.0352(8)
C12	0.6867(2)	0.4504(3)	0.1763(2)	0.0433(9)
C13	0.68824(16)	0.0818(2)	0.1115(2)	0.0290(7)
C14	0.72772(17)	0.1787(3)	0.1183(2)	0.0306(7)

	x/a	y/b	z/c	U(eq)
C15	0.0	0.1875(5)	0.25	0.0509(14)
C16	0.0	0.0782(5)	0.25	0.147(5)
Fe1	0.57953(2)	0.21828(3)	0.19392(3)	0.02784(16)
N1	0.61319(13)	0.08153(19)	0.12718(17)	0.0281(6)
N2	0.68988(14)	0.2640(2)	0.14412(17)	0.0292(6)
N3	0.66001(17)	0.2199(3)	0.34762(19)	0.0446(8)
N4	0.50578(15)	0.3335(2)	0.0758(2)	0.0376(7)
N5	0.0	0.2723(4)	0.25	0.0828(19)
O1	0.5	0.1765(2)	0.25	0.0299(7)
O2	0.65730(12)	0.13991(19)	0.29937(15)	0.0387(6)
O3	0.62424(14)	0.29707(18)	0.31491(15)	0.0405(6)
O4	0.6934(2)	0.2214(3)	0.4197(2)	0.1062(14)
O5	0.52200(12)	0.24092(17)	0.05827(15)	0.0341(5)
O6	0.53140(14)	0.36385(18)	0.15380(17)	0.0425(6)
O7	0.47019(14)	0.3897(2)	0.02332(18)	0.0538(7)

Table 4. Bond lengths (Å) for $[Fe_2O(NO_3)_4(dmp)_2]$ (2).

C1-C2	1.494(5)	C1-H1A	0.98
C1-H1B	0.98	C1-H1C	0.98
C2-N1	1.331(4)	C2-C3	1.392(5)
C3-C4	1.360(5)	C3-H3	0.95
C4-C5	1.392(5)	C4-H4	0.95
C5-C13	1.383(5)	C5-C6	1.425(5)
C6-C7	1.335(5)	C6-H6	0.95
C7-C8	1.414(5)	C7-H7	0.95
C8-C9	1.398(5)	C8-C14	1.409(5)
C9-C10	1.348(5)	C9-H9	0.95
C10-C11	1.401(5)	C10-H10	0.95
C11-N2	1.343(4)	C11-C12	1.482(5)
C12-H12A	0.98	C12-H12B	0.98
C12-H12C	0.98	C13-N1	1.367(4)
C13-C14	1.433(5)	C14-N2	1.358(4)
C15-N5	1.093(7)	C15-C16	1.410(9)

C16-H16A	0.9805	C16-H16B	0.9805
C16-H16C	0.9805	Fe1-O1	1.772(3)
Fe1-N1	2.127(4)	Fe1-O6	2.127(4)
Fe1-O3	2.151(4)	Fe1-O5	2.194(4)
Fe1-N2	2.221(4)	Fe1-O2	2.238(3)
N3-O4	1.176(5)	N3-O2	1.252(4)
N3-O3	1.257(4)	N4-O7	1.204(4)
N4-O5	1.259(4)	N4-O6	1.266(4)
O1-Fe1#1	1.772(3)		

Symmetry transformations used to generate equivalent atoms:
#1 -x+1, y, -z+1/2

Table 5. Bond angles (°) for $[Fe_2O(NO_3)_4(dmp)_2]$ (2).

C2-C1-H1A	109.5	C2-C1-H1B	109.5
H1A-C1-H1B	109.5	C2-C1-H1C	109.5
H1A-C1-H1C	109.5	H1B-C1-H1C	109.5
N1-C2-C3	120.8(3)	N1-C2-C1	120.0(3)
C3-C2-C1	119.2(3)	C4-C3-C2	120.1(3)
C4-C3-H3	119.9	C2-C3-H3	119.9
C3-C4-C5	120.0(3)	C3-C4-H4	120.0
C5-C4-H4	120.0	C13-C5-C4	117.3(3)
C13-C5-C6	119.9(3)	C4-C5-C6	122.8(3)
C7-C6-C5	120.9(3)	C7-C6-H6	119.6
C5-C6-H6	119.6	C6-C7-C8	120.9(3)
C6-C7-H7	119.5	C8-C7-H7	119.5
C9-C8-C14	116.9(3)	C9-C8-C7	123.2(3)
C14-C8-C7	119.9(3)	C10-C9-C8	119.8(3)
C10-C9-H9	120.1	C8-C9-H9	120.1
C9-C10-C11	121.2(3)	C9-C10-H10	119.4
C11-C10-H10	119.4	N2-C11-C10	120.5(3)
N2-C11-C12	120.3(3)	C10-C11-C12	119.2(3)
C11-C12-H12A	109.5	C11-C12-H12B	109.5
H12A-C12-H12B	109.5	C11-C12-H12C	109.5
H12A-C12-H12C	109.5	H12B-C12-H12C	109.5
N1-C13-C5	122.4(3)	N1-C13-C14	117.8(3)

C5-C13-C14	119.7(3)	N2-C14-C8	122.9(3)
N2-C14-C13	118.5(3)	C8-C14-C13	118.6(3)
N5-C15-C16	180.000(10)	C15-C16-H16A	109.5
C15-C16-H16B	109.5	H16A-C16-H16B	109.4
C15-C16-H16C	109.5	H16A-C16-H16C	109.4
H16B-C16-H16C	109.4	O1-Fe1-N1	103.01(14)
O1-Fe1-O6	94.69(14)	N1-Fe1-O6	136.38(13)
O1-Fe1-O3	90.71(13)	N1-Fe1-O3	132.59(11)
O6-Fe1-O3	85.87(14)	O1-Fe1-O5	97.77(13)
N1-Fe1-O5	79.39(12)	O6-Fe1-O5	58.71(9)
O3-Fe1-O5	144.02(12)	O1-Fe1-N2	170.85(7)
N1-Fe1-N2	77.41(13)	O6-Fe1-N2	91.03(13)
O3-Fe1-N2	82.58(13)	O5-Fe1-N2	91.30(14)
O1-Fe1-O2	90.46(15)	N1-Fe1-O2	76.54(14)
O6-Fe1-O2	143.52(10)	O3-Fe1-O2	57.94(14)
O5-Fe1-O2	155.75(9)	N2-Fe1-O2	80.71(14)
C2-N1-C13	119.1(3)	C2-N1-Fe1	127.1(2)
C13-N1-Fe1	112.8(2)	C11-N2-C14	118.7(3)
C11-N2-Fe1	130.3(2)	C14-N2-Fe1	110.1(2)
O4-N3-O2	121.5(4)	O4-N3-O3	122.5(4)
O2-N3-O3	116.0(3)	O7-N4-O5	123.8(3)
O7-N4-O6	122.1(3)	O5-N4-O6	114.1(3)
Fe1-O1-Fe1#1	144.6(2)	N3-O2-Fe1	91.0(2)
N3-O3-Fe1	95.0(2)	N4-O5-Fe1	92.09(18)
N4-O6-Fe1	95.0(2)		

Symmetry transformations used to generate equivalent atoms:
#1 -x+1, y, -z+1/2

Table 6. Anisotropic atomic displacement parameters (Å^2) for [$Fe_2O(NO_3)_4(dmp)_2$] (2).

The anisotropic atomic displacement factor exponent takes the form:
$-2\pi^2[h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0289(17)	0.0324(19)	0.0418(18)	-0.0057(14)	0.0032(15)	-0.0065(13)
C2	0.0294(16)	0.0301(18)	0.0343(17)	-0.0007(14)	0.0007(14)	-0.0005(13)
C3	0.0416(19)	0.032(2)	0.051(2)	-0.0033(16)	0.0021(17)	0.0033(16)
C4	0.048(2)	0.037(2)	0.052(2)	-0.0016(17)	0.0079(18)	0.0132(17)
C5	0.0315(17)	0.043(2)	0.0333(17)	0.0080(15)	0.0041(14)	0.0117(15)
C6	0.0324(18)	0.056(2)	0.043(2)	0.0141(17)	0.0081(16)	0.0196(18)
C7	0.0209(16)	0.070(3)	0.043(2)	0.0199(18)	0.0084(15)	0.0119(17)
C8	0.0204(15)	0.060(2)	0.0289(16)	0.0129(16)	-0.0003(13)	-0.0018(15)
C9	0.0229(16)	0.074(3)	0.0371(19)	0.0156(19)	0.0006(14)	-0.0142(18)
C10	0.0342(18)	0.057(2)	0.0374(18)	0.0071(17)	-0.0037(16)	-0.0200(18)
C11	0.0354(18)	0.043(2)	0.0266(16)	0.0040(15)	-0.0011(14)	-0.0134(16)
C12	0.050(2)	0.041(2)	0.0402(19)	-0.0041(16)	0.0073(17)	-0.0207(17)
C13	0.0236(15)	0.0370(19)	0.0265(15)	0.0031(13)	0.0024(13)	0.0006(13)
C14	0.0234(15)	0.0440(19)	0.0243(15)	0.0057(14)	0.0003(13)	-0.0008(14)
C15	0.050(3)	0.042(3)	0.058(3)	0	-0.018(3)	0
C16	0.151(9)	0.041(4)	0.229(12)	0	-0.110(9)	0
Fe1	0.0232(2)	0.0291(3)	0.0318(3)	-0.00185(19)	0.00608(18)	-0.00336(19)
N1	0.0225(12)	0.0316(15)	0.0300(14)	-0.0003(11)	0.0016(11)	0.0009(11)
N2	0.0262(13)	0.0365(16)	0.0251(13)	0.0021(11)	0.0030(11)	-0.0063(11)
N3	0.0415(17)	0.064(2)	0.0278(15)	0.0048(16)	0.0012(14)	-0.0251(17)
N4	0.0312(15)	0.0397(18)	0.0433(17)	0.0091(14)	0.0112(13)	-0.0041(13)
N5	0.107(5)	0.045(3)	0.090(4)	0	-0.035(4)	0
O1	0.0253(15)	0.0280(16)	0.0375(16)	0	0.0097(13)	0
O2	0.0319(12)	0.0441(15)	0.0401(13)	0.0036(12)	0.0034(10)	-0.0056(11)
O3	0.0478(14)	0.0398(15)	0.0350(12)	-0.0040(11)	0.0102(11)	-0.0133(12)
O4	0.133(3)	0.127(3)	0.055(2)	0.013(2)	-0.020(2)	-0.062(3)
O5	0.0328(12)	0.0315(13)	0.0385(13)	0.0005(10)	0.0048(10)	0.0055(10)
O6	0.0475(14)	0.0313(13)	0.0497(15)	0.0002(11)	0.0115(12)	-0.0053(11)
O7	0.0499(15)	0.0528(17)	0.0600(16)	0.0292(14)	0.0127(13)	0.0158(13)

Table 7. Hydrogen atomic coordinates and isotropic atomic displacement parameters ($Å^2$) for $[Fe_2O(NO_3)_4(dmp)_2]$ (2).

	x/a	y/b	z/c	U(eq)
H1A	0.4785	-0.0271	0.1820	0.052
H1B	0.4660	-0.0525	0.0757	0.052
H1C	0.4705	0.0654	0.1095	0.052
H3	0.5805	-0.1598	0.0852	0.05
H4	0.7090	-0.1619	0.0683	0.055
H6	0.8292	-0.0621	0.0526	0.052
H7	0.8916	0.0919	0.0582	0.053
H9	0.8910	0.2842	0.0879	0.054
H10	0.8265	0.4279	0.1306	0.052
H12A	0.6465	0.4681	0.1294	0.065
H12B	0.7229	0.5078	0.1834	0.065
H12C	0.6644	0.4380	0.2342	0.065
H16A	1.0521	0.0529	0.2498	0.22
H16B	0.9703	0.0529	0.1957	0.22
H16C	0.9776	0.0529	0.3046	0.22

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