

Supporting Information

Formation Pathway of Roussin's Red Ester (RRE) via Reaction of $\{\text{Fe}(\text{NO})_2\}^{10}$

DNIC and Thiol: The Facile Synthetic Route for Synthesizing

Cysteine-Containing Dinitrosyl Iron Complex (DNIC)

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Table S1. Crystallographic data for [Fe(NO)₂(μ-CO)(μ-S(CH₂)₂NH₃)Fe(NO)₂] (**1**), [(μ-SC₂H₄NH₂)(μ-SC₂H₄NH₃)Fe₂(NO)₄] (**2**), [(S(CH₂)₂NH₂)(S(CH₂)₂NH₃)Fe(NO)₂] (**4**) and [(S(C₆H₅))(μ-S(CH₂)₂NH₃)Fe(NO)₂] (**5**).

Complexes	1	2	4	5
Chemical formula	C ₃ H ₇ Fe ₂ N ₅ O ₅ S	C ₄ H ₁₃ Fe ₂ N ₆ O ₄ S ₂	C ₄ H ₁₃ FeN ₄ O ₂ S ₂	C ₈ H ₁₂ FeN ₃ O ₂ S ₂
	·(C ₁₂ H ₂₄ O ₆)	·(C ₁₂ H ₂₄ O ₆)	·(C ₁₂ H ₂₄ O ₆)	·1.5(C ₁₂ H ₂₄ O ₆)
Formula Mass	601.21	648.33	533.47	689.65
Crystal system	Monoclinic	Triclinic	Monoclinic	Triclinic
<i>a</i> /Å	10.8834(6)	9.002(2)	10.6804(6)	8.203(3)
<i>b</i> /Å	10.7139(6)	11.907(3)	14.6441(11)	13.821(5)
<i>c</i> /Å	22.0706(11)	15.175(4)	16.5578(9)	15.719(4)
<i>α</i> /°	90	69.492(4)	90	107.632(7)
<i>β</i> /°	98.478(2)	73.004(4)	103.811(4)	95.624(9)
<i>γ</i> /°	90	69.942(4)	90	97.094(10)
Unit cell volume/Å ³	2545.4(2)	1403.5(6)	2514.8(3)	1668.0(9)
Temperature/K	200(2)	200(2)	200(2)	200(2)
Space group	P 2 ₁ /c	P -1	P 2 ₁ /n	P -1
No. of formula units per unit cell, Z	4	2	4	1
Radiation type Synchrotron	Mo K _α	Mo K _α	Mo K _α	Mo K _α
Absorption coefficient, μ/mm ⁻¹	1.282	1.239	0.811	0.635
No. of reflections measured	17967	11294	14202	12922
No. of independent reflections	4519	4836	4417	5769
<i>R</i> _{int}	0.0432	0.0296	0.0537	0.0880
Final <i>R</i> _{<i>I</i>} values (<i>I</i> > 2σ(<i>I</i>))	0.0362	0.0608	0.0436	0.0759
Final <i>wR</i> (<i>F</i> ²) values (<i>I</i> > 2σ(<i>I</i>))	0.0859	0.2165	0.0997	0.1621
Final <i>R</i> _{<i>I</i>} values (all data)	0.0570	0.0730	0.0751	0.1751
Final <i>wR</i> (<i>F</i> ²) values (all data)	0.1031	0.2297	0.1147	0.2021
Goodness of fit on <i>F</i> ²	1.065	0.926	1.012	1.014

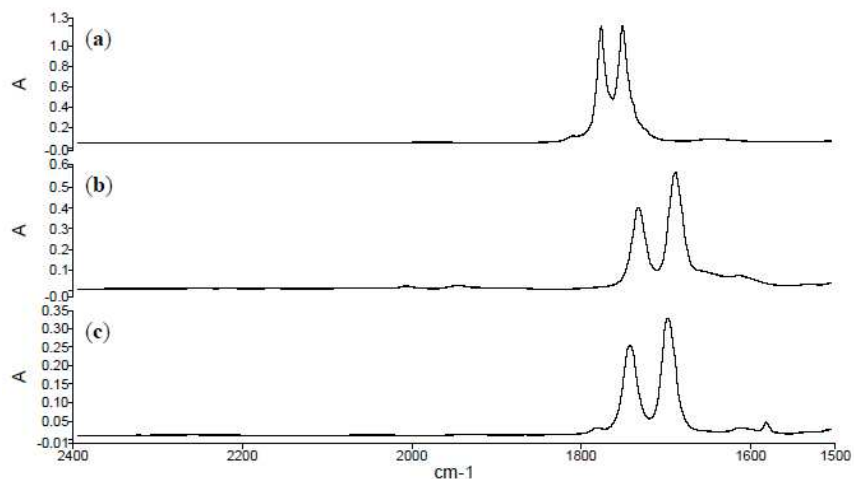


Figure S5. FT-IR spectra of (a) neutral RRE $[(SC_2H_4NH_2)Fe(NO)_2]_2$ (**3**) with ν_{NO} stretching frequencies at 1749 s, 1774 s, 1808 vw cm^{-1} (THF), (b) neutral DNIC $[(SC_2H_4NH_2)(SC_2H_4NH_3)Fe(NO)_2]$ (**4**) with ν_{NO} stretching frequencies at 1685 s, 1731 s cm^{-1} (THF), and (c) $[(SC_6H_5)(S(CH_2)_2NH_3)Fe(NO)_2]$ (**5**) with ν_{NO} stretching frequencies at 1693 s, 1739 s cm^{-1} (THF).

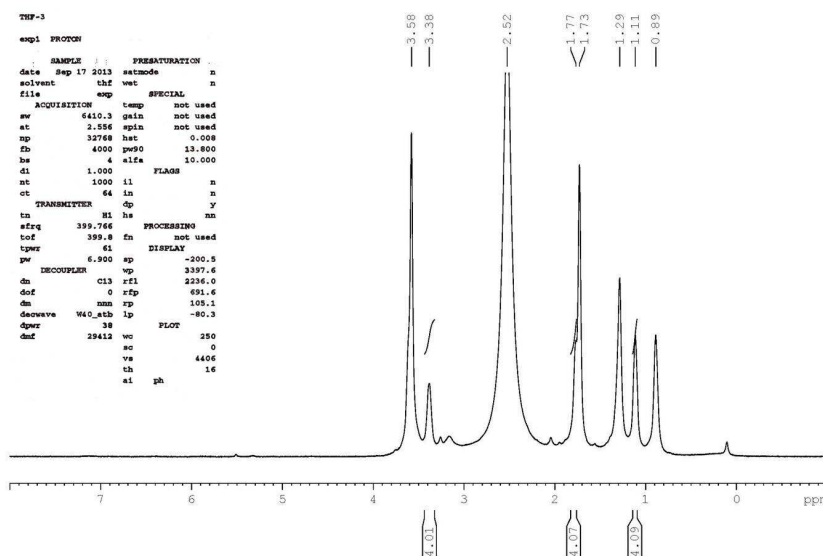


Figure S6. 1H NMR spectrum (400 MHz; d_8 -THF) of complex **3**: δ 1.12 (br), 1.77 (br), 3.38 (br) ppm ($-CH_2CH_2NH_2$).