

Inorganic Chemistry

including bioinorganic chemistry

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Supporting Information:

The ligands are synthesized as previously described from an activated form of 2,3-dimethoxybenzoic acid and one-half equivalent of diamine followed by deprotection with BBr_3 .³³

Crystal data for $(\text{Et}_4\text{N})_6[\text{Fe}_2(\text{1})_3] \cdot 10 \text{ H}_2\text{O}$: the complex crystallized by slow diffusion of ether into an aqueous nitromethane solution in space group P3_221 (#154) with the following cell constants based on 5474 reflections with $I > 10\sigma(I)$ and 2θ between 3° and 45° : $a = 16.4329(6) \text{ \AA}$, $c = 36.071(12) \text{ \AA}$, $V = 8443(6) \text{ \AA}^3$, $Z = 3$. A total of 33999 reflections were collected at 153 K using ω scans on a Siemens SMART diffractometer with graphite monochromated Mo- $\text{K}\alpha$ radiation ($\lambda = 0.71069 \text{ \AA}$), $3^\circ < 2\theta < 45^\circ$, of which 8104 were unique. The structure was solved by direct methods (SHELXS-86). As the helical complex is positioned on a crystallographic two-fold axis, only one half is unique. All iron, oxygen, nitrogen, and carbon atoms present in the anionic complex were refined anisotropically as were full-occupancy solvent water atoms and two well-defined tetraethylammonium cations. The remaining solvent molecules and cation were disordered and modeled at reduced occupancies with the aid of rigid groups for the cation. For greater detail, see supplemental information. Hydrogen atoms were included at idealized positions, 0.95 \AA from their parent atoms on all the atoms except those of two rigid bodies modeling cation disorder. Full matrix refinement of 569 variables based on 4156 reflections ($|F_O| > 3\sigma(F_O)$, $8.24^\circ < 2\theta < 45^\circ$) converged with final residuals of $R(R_W) = 8.0(8.1)$. The absolute configuration of the space group was assigned by inversion and subsequent refinement of the structure. The difference in residuals between the two enantiomers was statistically significant, but small. At this level of refinement, this difference can not distinguish between an inversion-twin and a spontaneous resolution of two enantiomers.

Crystal data for $(\text{Me}_4\text{N})_4[\text{Fe}_2(\text{1})_2(\text{OH})_2] \cdot 8.5 \text{ H}_2\text{O}$: the complex crystallized by slow diffusion of acetone into an aqueous acetonitrile solution in space group C2/c (#15)

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with the following cell constants based on 25 reflections ($18^\circ < 2\theta < 36^\circ$): $a = 18.462(2)$ Å, $b = 13.990(2)$ Å, $c = 23.252(2)$ Å, $\beta = 94.47(1)^\circ$, $V = 5987(1)$ Å³. A total of 4889 reflections were collected at 203 K using ω scans on an Enraf-Nonius CAD4 diffractometer with graphite monochromated Mo-K α radiation ($\lambda = 0.71069$ Å), $5^\circ < 2\theta < 46^\circ$, of which 3898 were unique. The structure was solved by direct methods (SAPI-91). All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were placed at idealized positions (0.95 Å bond distance). Four and one-quarter independent solvent waters were present in a well-defined hydrogen-bonding network. All water oxygens are located ~ 2.8 Å from other water oxygens or oxygens in the anionic complex. Full matrix refinement of 386 variables based on 2382 reflections ($|F_o| > 3\sigma(F_o)$, $8.24^\circ < 2\theta < 47^\circ$) converged with final residuals of $R(R_w) = 5.7(7.3)$.

All calculations were performed using the teXsan crystallographic software package of Molecular Structure Corporation.

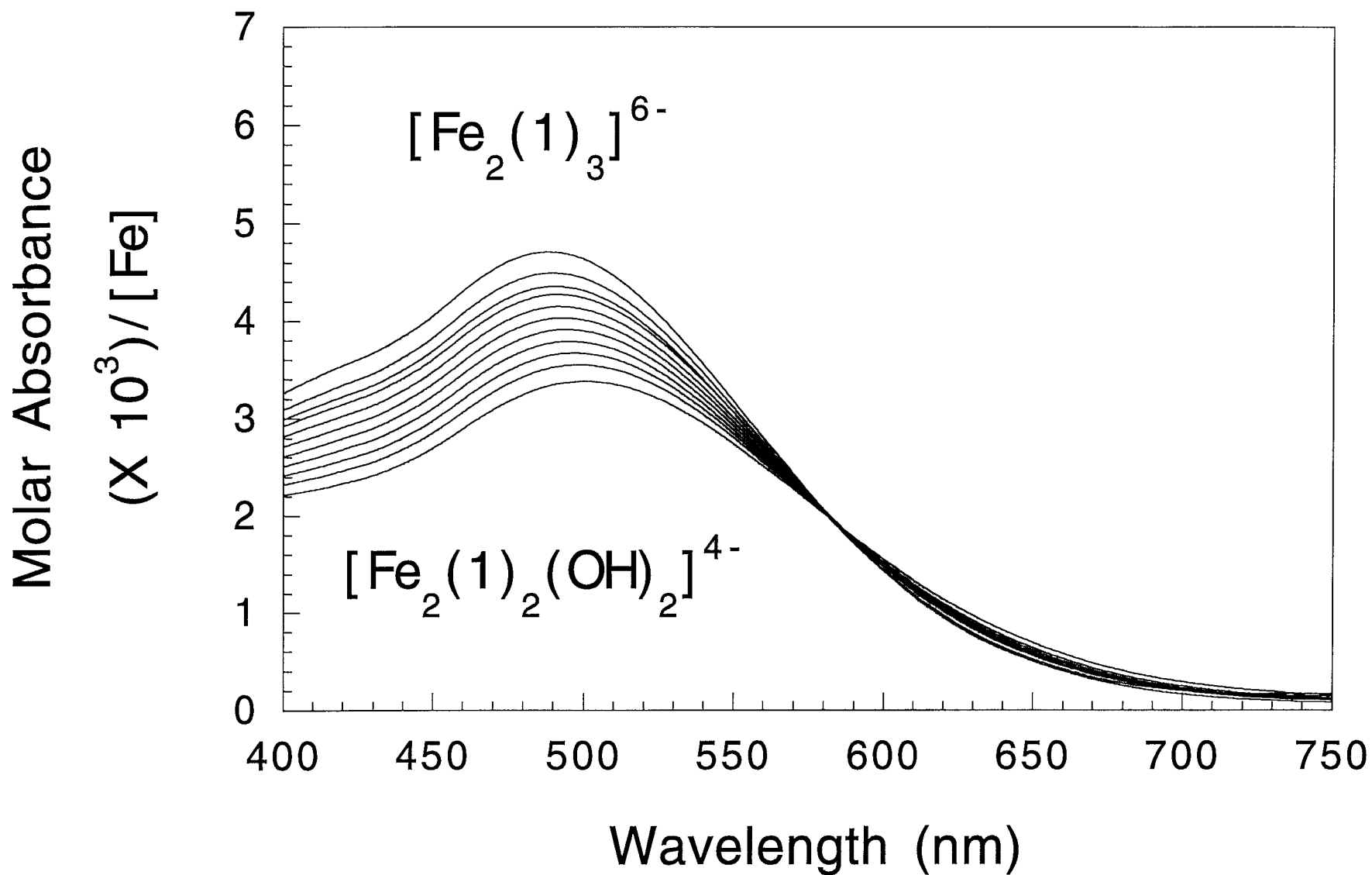


Fig. S-1. Visible titration of $[\text{Fe}_2(1)_2(\text{OH})_2]^{4-}$ to $[\text{Fe}_2(1)_3]^{6-}$ by the incremental addition of **1** (pH = 9 buffered solution).

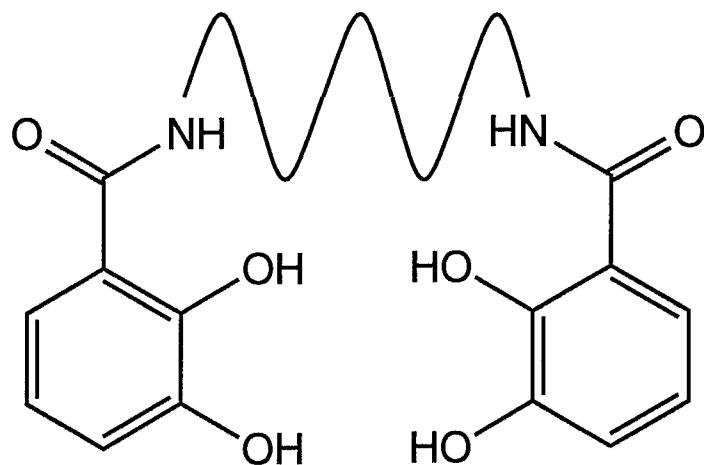
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Table S-1. Spectroscopic Data for **1H₄**

1H₄: ¹H NMR (400 MHz, d₆-DMSO): δ = 8.94 (t, ³J(H,H) = 5.4 Hz, 2H; **NH**), 7.26 (d, ³J(H,H) = 8.2 Hz, 2H; **H_a**), 6.90 (d, ³J(H,H) = 7.77 Hz; **H_c**), 6.68 (t, ³J(H,H) = 7.93 Hz, 2H; **H_b**), 3.47 (s, 4H, HN-CH₂-CH₂-NH). FABH⁺ Calcd: 333.11. Found: 333.

Bis-Catecholamide



1H₄

