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POLYHEDRON

Polyhedron 26 (2007) 791-796

Synthesis and characterization of a monomeric and a dihydroxo-bridged dimeric complexes of iron(III) with α -benzoinoxime

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Received 14 August 2006; accepted 6 September 2006 Available online 27 September 2006

Abstract

The reaction of α -benzoinoxime, H₂BNO with FeCl₃ in the presence of Et₃N as a base gives the mononuclear Fe(III) complex, Fe(HBNO)₃ (1). Treatment of 1 with a methanolic solution of KOH at room temperature leads to a dinuclear Fe(III)–Fe(III) complex, [Fe(HBNO)₂OH]₂ (2). The complexes were initially characterized on the basis of their elemental, mass and thermal analyses. The IR studies were useful in assigning the coordination mode of the benzoinoxime ligand to the iron metal. In addition, the presence of a hydroxo-bridge in the dimeric complex 2 is inferred from the IR spectral studies. Room-temperature Mössbauer studies indicated octahedral, high-spin iron(III). Variable-temperature magnetic susceptibility measurements supported the existence of the μ -dihydroxobridging structure core, Fe^{III}(μ -OH)₂Fe^{III} in the dinuclear complex 2. Theoretical modelling of the magnetic data indicated a weak antiferromagnetic spin exchange between the iron(III) centers ($J = -8.35 \text{ cm}^{-1}$, g = 2.01, $\rho = 0.02$ and TIP = $1.7 \times 10^{-4} \text{ cm}^3 \text{ mol}^{-1}$ for $H = -2JS_1 \cdot S_2$). The electronic spectra of the complexes revealed two bands due to d–d transitions and one band assignable to an oxygen (p_{π}) \rightarrow Fe($d_{\pi*}$) LMCT transition observed in each complex. An additional charge-transfer transition, assignable to μ -hydroxo(p_{π}) \rightarrow Fe($d_{\pi*}$), was observed for the dimeric complex 2. The structural and vibrational behaviors of these complexes have been elucidated with quantum mechanical methods.

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Keywords: Iron complexes; Benzoinoxime; Dimer; Dihydroxo bridge; Magnetic studies; Exchange coupling; Semi-empirical calculations

1. Introduction

The importance of iron as an essential element can be estimated by the wide range of iron proteins and enzymes playing different roles in biological systems. Proteins containing non-heme iron centers are widely spread in nature. They perform a broad range of functions, especially activating dioxygen for the oxidation of various substrates [1]. Dinuclear iron complexes in particular have biological relevance since they give valuable information about the binding mode of dioxygen to the iron sites of proteins involved in its transport and activation. They provide structural models for dinuclear sites in several proteins

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involved in oxygen storage of hemerythrin and oxygen activation of methanemonooxygenase [2–4]. Efforts to model these proteins have simulated the study of complexes containing Fe(μ -O)₂Fe and Fe(μ -OH)₂Fe cores [5–7]. In addition, these systems were treated as models for understanding the magnitude of exchange coupling interactions between the two iron centers and its relation to geometrical parameters [8]. Studies of some diiron complexes have shown that the oxo-bridge is responsible for strong antiferromagnetic coupling ($-50 > J > -200 \text{ cm}^{-1}$) and those complexes with hydroxo, alkoxo, phenoxo-bridges are weakly coupled ($0 > J > -30 \text{ cm}^{-1}$) [6,8–10].

We reported previously the synthesis and characterization of dinuclear chromium and molybdenum complexes containing oxo and hydroxo-bridged ligands [11,12]. In the present work, we describe the preparation and characterization of two new iron(III) complexes of the

^{0277-5387/\$ -} see front matter @ 2006 Elsevier Ltd. All rights reserved. doi:10.1016/j.poly.2006.09.029

 α -benzoinoxime ligand. The coordination properties of the complexes were studied using FTIR, electronic and Mössbauer techniques. A magneto-structural correlation has been studied by variable-temperature magnetic susceptibility and theoretical modelling of the magnetic data. Semi-empirical theoretical calculations (MM+ and parameterized PM3) have been used to predict the most favored geometry for each complex.



α-benzoinoxime ligand (H2BNO)

2. Experimental

Ferric chloride, $FeCl_3 \cdot 6H_2O$, and α -benzoinoxime, H_2BNO , were used as purchased from Aldrich chemical Co. Inc. Solvents were dried according to standard procedures.

Variable-temperature (10–300 K) magnetic susceptibility measurements were obtained with an Oxford Faraday magnetometer. Mössbauer measurements were carried out on a multichannel analyzer in constant acceleration mode. ⁵⁷Co in a Rh matrix (20 mc) was used as a radioactive source and was held at room temperature. The isomer shift was expressed relative to an iron metal absorber. Infrared spectra $(4000-400 \text{ cm}^{-1})$ were recorded as KBr pellets on a Thermo Nicolet Nexus 670 FTIR spectrometer. The thermal analyses (TGA-DTA) were carried out under nitrogen with a heating rate of 10 °C/min using a Perkin-Elmer thermal analyzer. The electronic absorption spectra (190-1100 nm) were measured on a Cary 50 spectrophotometer. Samples were run on 10^{-4} M solutions in DMSO. Elemental analyses were performed using a Perkin-Elmer 2400 CHN elemental analyzer. The semi-empirical calculations in this study have been carried out using the Hyperchem 6.01 program package [13]. The Polak-Ribiere version of the conjugate gradient method was used in all energy minimization calculations with a coverage criterion less than 0.001 kcal mol⁻¹ Å⁻¹. First and separately the benzoinoxime ligand was optimized and the results obtained have been applied for the oxime ligand in each calculation thereafter. The values of Fe-O and Fe-N bond lengths and Fe-O-Fe bond angle in the literatures were used as the starting inputs [6,14–17]. Initial optimization of different geometries of the complexes was performed with molecular mechanics (MM+). The geometry was further refined using

parameterized PM3 method. Available parameters for Fe metal were implemented in the PM3 semi-empirical method [18]. The parameterized PM3 was used for the geometry optimization of the complexes under study. Theoretical harmonic vibrational frequencies are related to the experimental fundamentals by the optimum scaling factor λ , determined through a least-squares procedure, given by $\lambda = \sum_{i}^{\text{all}} \omega_{i}^{\text{theor}} v_{i}^{\text{expt}} / \sum_{i}^{\text{all}} (\omega_{i}^{\text{theor}})^{2}$, where $\omega_{i}^{\text{theor}}$ and v_{i}^{expt} are the *i*th theoretical harmonic and *i*th experimental fundamental frequencies (in cm⁻¹) [19]. The molecular root mean square error (rms) was calculated by the square root of the sum over all the modes of Δ_{\min} given in Ref. [19], where Δ_{\min} is the minimized residual for each mode.

2.1. Synthesis of tris(benzoinoximato)iron(III), Fe(HBNO)₃ (1)

A solution of triethylamine (0.2 mL) in ethanol (5 mL) was added dropwise to an ethanolic solution (30 mL) of α -benzoinoxime, H₂BNO (0.38 g, 1.67 mmol) with stirring at room temperature. To the resulting solution, was added an ethanolic solution (10 mL) of FeCl₃ · 6H₂O (0.15 g, 0.55 mmol) and the mixture was heated at 70 °C for 18 h. The purple residue was separated from the reaction medium and then washed with cold absolute ethanol and dried. The residue was dissolved in dichloromethane. Chromatographic separation on a neutral Al₂O₃ column eluted with CH₂Cl₂–ether (90:10% v/v) gave Fe(HBNO)₃ as a purple band. Purple microcrystals of the complex were obtained by slow evaporation of the solvent with a flow of argon. The crystals were dried in a vacuum overnight. The product was obtained in 0.35 g (87%) yield.

Anal. Calc. for $C_{42}H_{36}FeN_3O_6$ (MW = 734.62): C, 68.67; H, 4.94; N, 5.72. Found: C, 68.50; H, 5.10; N, 5.77%. TGA, % weight loss (temp. °C): 61.22 (75–210), 23.26 (213–436).

2.2. Synthesis of μ-dihydroxobis{bis(benzoinoximato)iron(III)}, [Fe(HBNO)₂(μ-OH)]₂ (**2**)

To a solution of Fe(HBNO)₃ (0.5 g, 0.68 mmol) in 10 mL CH₃CN was added 5 mL CH₃OH solution of KOH (0.04 g, 0.71 mmol), and the mixture was stirred for 36 h. The color of the solution turned red and was dried in vacuum. The residue was washed with hot ethanol (95%) to remove KHBNO. Then the residue was redissolved in CH₂Cl₂ and subjected to chromatography on silica gel column with CH₂Cl₂-ether (80:20% v/v). The red band was eluted and collected. A dark red solid was obtained from the eluent by slow evaporation of the solvent with a flow of argon. The solid product was dried in a vacuum overnight. The product as a red solid was obtained in 64% (0.23 g) yield.

Anal. Calc. for $C_{56}H_{50}Fe_2N_4O_{10}$ (MW = 1050.74): C, 64.01; H, 4.80; N, 5.33. Found: C, 63.92; H, 4.77; N, 5.29%. TGA, % weight loss (temp. °C): 46.41 (61–152), 29.27 (154–326), 10.50 (328–480).

3. Results and discussion

The stoichiometric reaction between α -benzoinoxime, H₂BNO and FeCl₃ in the presence of triethylamine as a base has yielded the monomeric complex Fe(HBNO)₃ (1). Addition of OH⁻ to complex 1 at room temperature afforded the dimeric product 2. Chromatographic analyses of both complexes have indicated one isomeric form for each complex.

The complexes were initially characterized on the basis of their elemental analyses, mass spectra and thermal analvses (TGA, DTA). The parent ion peak (m/z) was observed in the mass spectra at 734 and 1051 for complexes 1 and 2, respectively. The thermal analyses (TGA, DTA) of complex 1 showed a weight loss of 61.22% in the temperature range 75-210 °C, corresponding to the loss of two molecules of benzoinoxime ligand. The mass loss of 23.26% in the temperature range 213-436 °C, corresponds to two phenyl groups and one OH of the third benzoinoxime ligand. For the dimeric complex 2, three endothermic peaks were observed. The mass loss of 46.41% in the temperature range 61-152 °C corresponds to two benzoinoxime ligands and two OH groups. The other weight losses of 29.27% and 10.50% in the temperature ranges 154-326 and 328-480°C, respectively, are due to gradual decomposition of the organic fragments of the other benzoinoxime ligands.

The primary ligand, α -benzoinoxime, showed characteristic IR bands at 3326, 1670 and 950 cm⁻¹ due to the OH, C=N and N-O stretching vibrations of the oxime group. These bands are observed for complex **1** at 3421 (OH), 1625 (C=N), 1089 and 1024 cm⁻¹ (N-O). The C=N stretching vibration has shifted by 45 cm^{-1} to lower frequency. In contrast, the bands corresponding to OH and N–O stretches have shifted to higher frequencies with respect to the parent ligand. Such behavior is evidently indicates that the α -benzoinoxime ligands coordinate to the iron metal through the nitrogen atom of the oxime group [11,20]. The presence of two bands for the N–O stretching vibration indicates different types of N–O groups [21,22]. Consequently, the peaks at 1089 and 1024 cm⁻¹ are assigned to free N–O and hydrogen-bonded N–O, respectively.

The infrared spectrum of complex 2 revealed two strong absorptions at 968 and 913 cm⁻¹ that are not present in the spectrum of the monomeric complex Fe(HBNO)₃. In addition, no band between 800 and 850 cm^{-1} attributable to an oxo bridged structural unit is observed in the IR spectrum of the dimeric complex [Fe(HBNO)₂OH]₂ [5,10,23]. Furthermore, the similarity in the absorption peaks due to coordinated benzoinoxime ligand in both the monomeric and the dimeric complexes rules against any type of benzoinoximate bridging in the dimer. Therefore, the bands at 968 and 913 cm⁻¹ in the [Fe(HBNO)₂OH]₂ dimer are assigned to a vibrational mode associated with the $Fe_2(OH)_2^{4+}$ structural unit. Consistent with this assignment, the infrared spectra of [Fe(pic)₂OH]₂ and $[Cr(HBNO)(BNIM)OH]_2$ showed bands due to $Fe_2(OH)_2^{4+}$ and $Cr_2(OH)_2^{4+}$ at 950 and 970 cm⁻¹, respectively [10,12]. Similarly to the monomeric complex 1, a peak due to stretching vibration of oxime OH is observed at 3422 cm^{-1} for the dimeric complex. An additional peak was observed at 3599 cm^{-1} which is assigned to the



Fig. 1. Chemical structures for complexes 1 and 2 used for semi-empirical calculations.

bridging OH stretch. Finally, bands indicative of two types of N–O groups were observed at 1117 and 1080 cm⁻¹ (free N–O) and at 1052 and 1024 cm⁻¹ (hydrogen-bonded N–O). Usually, oxime complexes show two N–O bands in the range 1190–1260 and 1071–1110 cm⁻¹ for strongly hydrogen-bonded species, whilst in free and weakly hydrogen-bonded species the N–O absorption occurs at ca. 1150 and 1020 cm⁻¹ [21,22,24].

The monomeric complex 1 is expected to have two geometrical isomers; facial 1A and meridional 1B (Fig. 1), but only one isomer is obtained as indicated by the chromatographic analysis. Now the question is whether the Fe(HBNO)₃ has a facial or meridional configuration. Also, the dimeric complex 2, is expected to adopt one of four geometrical isomers; 2A, 2B, 2C or 2D (Fig. 1). The spectroscopic techniques gave no information about the geometry of the complex. Hence, it was considered worthwhile to model the complexes using molecular mechanics MM+ and PM3 calculations, especially with the lack of structural analysis due to the poor quality of the crystals obtained.

The PM3 calculated results for each structure are summarized in Table 1. It can be seen that for the Fe(HBNO)₃ complex, the facial geometry **1A** is more stable than the meridional one **1B** by 3.8 kcal/mol. These results are in agreement with the structure of the Co(BMO)₃ complex, whose structure is characterized with a facial configuration by X-ray diffraction analysis [25]. For the dimeric complex **2**, the energy calculated for **2A** is more stable than those for the other configurations.

The most favored structures for the monomeric and dimeric iron(III) complexes based on semi-empirical PM3 geometry optimization are shown in Fig. 2. In both structures, the benzoinoxime ligand is coordinated to Fe(III) with the oxime nitrogen atom and the deprotonated alcoholic oxygen atom.

The observed and calculated vibrational frequencies for configurations **1A** and **2A** are compared in Table 2. The optimized scale factors determined for **1A** and **2A** are 0.998407 and 0.997745, respectively. The calculated harmonic vibrational frequencies compare well with fundamental experimental frequencies. The calculated root mean square error (rms) per molecule is 70.8 and 64.4 cm^{-1} for **1A** and **2A**, respectively. The rms values compare very well with those reported for vibrational frequency calculations using MP2-fc/631G(d) (rms = 63) and MP2-fc/631G(d,p) (rms = 61) [15].

The magnetic susceptibility data measured for $Fe(HBNO)_3$ revealed a magnetic moment of 5.86 B.M. at



Fig. 2. The most stable geometries for the $Fe(HBNO)_3$ complex (1A) and the $[Fe(HBNO)_2OH]_2$ complex (2A).

300 K, which is close to the spin-only value (5.92) of high-spin Fe(III). The magnetic moment showed no significant temperature-dependence over the temperature range 300–10 K. Such behavior is indicative of a monomeric, octahedral high-spin Fe(III) complex [23].

Table 1

Calculated results for each structure of the monomeric complex 1 (1A and 1B) and of the dimeric complex 2 (2A, 2B, 2C and 2D)

		-				
Calculation results ^{a,b} (kcal/mol)	1A	1B	2A	2B	2C	2D
Total energy	-191659.1	-191655.3	-278205.7	-278149.9	-278201.5	-278201.1
Heat of formation	-284.63	-280.83	-802.07	-746.25	-797.84	-797.45

^a Total charge = 0 for all complexes.

^b The metal in all complexes is high-spin Fe(III).

Table 2 Characteristic calculated and observed vibrational frequencies (cm^{-1}) for conformations 1A and 2A

1A		2A		Assignment	
Calculated	Observed	Calculated	Observed		
		3596	3599	v(O–H) bridge	
3437	3421	3433	3422	v(O–H) oxime	
3064	3063	3083	3061	v(C-H) aromatic	
3029	3019	3029	3024		
2952	2932	2926	2922	v(C–H) aliphatic	
2815	2859	2877	2870	· / •	
1645	1625	1619	1629	v(C=N)	
1598	1607	1596	1607	v(C=C) + ring	
				deformation	
		1108	1117	v(N–O)	
1086	1089	1076	1080		
		1048	1050		
1020	1024	1011	1024		
		941	968	v(Fe–OH–Fe)	
		910	913		
481	496	480	496	v(M–O)	
441	448	443		v(M–N)	

Magnetic susceptibility data for $[Fe(HBNO)_2OH]_2$ indicate a weak intramolecular antiferromagnetic spin exchange interaction. Both the magnitude and temperature dependence of the magnetic susceptibility typify the behavior of an $S_1 = S_2 = 5/2$ dimer [26]. The effective magnetic moment drops from 8.55 B.M. per molecule at ~300 K to 4.01 B.M. at ~11 K. The data were analyzed with the spin-spin interaction model based on the exchange Hamiltonian($\hat{H} = -2J\hat{S}_1 \cdot \hat{S}_2$). The molar paramagnetic susceptibility of an $S_1 = S_2 = 5/2$ exchange coupled Fe(III)– Fe(III) dimer is given by the following equation [27,28]:

$$\begin{split} \chi_{\rm M} &= \frac{2N\beta^2 g^2}{kT} \left[\frac{\chi'}{\chi''} \right] (1-\rho) + \frac{2.19g^2}{T} \rho + \text{TIP} \\ \chi' &= \left[(55+30 \exp(-10J/kT) + 14 \exp(-18J/kT) \right. \\ &+ 5 \exp(-24J/kT) + \exp(-28J/kT) \right] \\ \chi'' &= \left[11+9 \exp(-10J/kT) + 7 \exp(-18J/kT) \right. \\ &+ 5 \exp(-24J/kT) + 3 \exp(-28J/kT) + \exp(-30J/kT) \right] \end{split}$$

where, ρ gauges the amount of magnetically dilute ferric impurities and the other terms have their usual meanings. A non-linear least-squares fit to 51 experimental data points yielded $J = -8.35 \pm 0.2 \text{ cm}^{-1}$, $g = 2.01 \pm 0.02$, $\rho = 0.02 \pm 0.01$ and TIP = $1.7 \times 10^{-4} \pm 0.02 \text{ cm}^3 \text{ mol}^{-1}$ with $R = 8.29 \times 10^{-4}$. The fit is illustrated in Fig. 3. The value of the coupling constant J is in good agreement with the values reported for other dihydroxo-bridged Fe(III) dimers [6,10,29]. They exhibited coupling constants in the range of -7 to -11 cm^{-1} . In contrast, oxo-bridged Fe(III) dimers are invariably characterized by a coupling constant near -100 cm^{-1} [30].

The Mössbauer spectrum of the dimeric complex 2 at 298 K is shown in Fig. 4. The spectrum consists of a single symmetric quadrupole doublet with $\Delta E_{\rm O} = 0.87$ mm/s and



Fig. 3. Temperature dependence of the molar paramagnetic susceptibility, χ_m , curve for [Fe(HMNO)₂OH]₂. The solid line represent the least-squares fit of the data to the theoretical equation given in the text.



Fig. 4. Room-temperature Mössbauer spectrum of [Fe(HMNO)₂OH]₂.

 $\delta = 0.43$ mm/s. The values of the quadrupole splitting, ΔE_Q , and the isomer shift, δ , are consistent with the formulation of a complex containing high-spin Fe(III) and are similar to the Mössbauer parameters reported for number of dihydroxo-bridged diiron(III) dimers [30–32].

The electronic absorption spectra recorded for the two complexes and the parent ligand as well in DMSO solution over the range 190–1100 nm are summarized in Table 3. The parent ligand displayed two absorptions at 284 and 405 nm corresponding, respectively, to the intraligand π - π^* and n- π^* transitions. The electronic spectra of the Fe(III) complexes are characterized by a band that can

796

Table 3

The electronic absorption data for α -benzoinoxime and its iron(III) complexes recorded in DMSO

Compound	λ , nm (log ε)
H ₂ BNO	284 (2.7), 405 ^a (1.5)
Fe(HBNO) ₃	305 (4.1), 480 ^a (3.3), 609 (1.2), 794 (1.0)
[Fe(HBNO) ₂ OH] ₂	304 (3.9), 496 ^a (3.4), 563 (3.2) 825 (1.1), 935 (1.2)
3 61 11	

^a Shoulder.

be assigned to a ligand-based $\pi - \pi^*$ transition, which is normally the most intense band in the higher energy region of the spectra. Both complexes displayed a band that can be assigned to a charge-transfer transition from p_{π} orbitals of the oxime alcoholic oxygen atom to the half-filled $d_{\pi*}$ orbitals of the Fe(III) metal [32,33]. This band is observed at 480 nm for monomeric complex 1, and at 496 nm for dimeric complex 2. The red shift of this charge-transfer band for the dimeric complex with respect to the monomeric complex may explain the colors of the DMSO solutions of the two complexes (solutions of complexes 1 and 2 have purple and red colors, respectively). Two low-energy transitions (log $\varepsilon \leq 1.2$) were observed in the spectra of both complexes and most probably are due to spin-forbidden d-d transitions. They have been assigned to the $^6A_1 \rightarrow {}^4T_1$ and ${}^6A_1 \rightarrow {}^4T_2$ transitions of high-spin Fe(III) [34]. The dominant feature of the spectrum of the dimeric complex 2 is an intense transition observed at 563 nm $(\log \varepsilon = 3.2)$, which is absent in the spectra of both the monomeric complex and the parent ligand itself. This transition can be assigned to a charge-transfer transition from p_{π} orbitals of the hydroxo-bridged oxygen atom to $d_{\pi*}$ orbitals of the Fe(III) metal. Consistence with this assignment, a similar transition was observed for number of dihydroxo-bridged Fe(III) complexes [32,35].

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