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Synthesis, characterization and crystal structure of a dinuclear iron nitrosyl complex with 2-mercapto-1-[2-(4-pyridyl)-ethyl]-benzimidazolyl

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Abstract

A new dinuclear iron nitrosyl complex $[\text{Fe}_2(\text{C}_{14}\text{H}_{12}\text{N}_3\text{S})_2(\text{NO})_4]$ (**1**) ($\text{C}_{14}\text{H}_{12}\text{N}_3\text{S}$ = 2-mercapto-1-[2-(4-pyridyl)-ethyl]-benzimidazolyl) has been obtained by the reaction of $\text{Fe}(\text{NO})_2(\text{CO})_2$ with 2-mercapto-1-[2-(4-pyridyl)-ethyl]-benzimidazole in CH_3OH under moderate condition. Complex **1** was characterized by IR, UV-vis, electrochemistry and single crystal X-ray diffraction. IR spectrum displays two strong characteristic NO stretching frequencies (ν_{NO}) in solution and in solid state. Cyclic voltammetry shows one irreversible, two quasi-reversible and two reversible one-electron reductions and irreversible oxidizations. This result is consistent with the fact that complex **1** is very unstable and ready to lose NO in the air. As showing in the single crystal X-ray diffraction, complex **1** forms a “chair-shape” structure by the connections of two iron centers and S-C-N frames of benzimidazole. The dihedral angle of benzimidazole ring and 2Fe-2S plane is 73.6° . The crystal data are the following: **1**, monoclinic, space group $\text{P}2(1)/c$, $a = 10.43940(10) \text{ \AA}$, $b = 16.0900(2) \text{ \AA}$, $c = 10.13240(10) \text{ \AA}$, $\alpha = 90^\circ$, $\beta = 111.0940(10)^\circ$, $\gamma = 90^\circ$, $V = 1587.89(3) \text{ \AA}^3$, $Z = 4$.

Keywords

2-mercapto-1-[2-(4-pyridyl)-ethyl]-benzimidazole; dinuclear iron nitrosyl complex; infrared spectrum; electrochemistry; crystal structure

1. Introduction

Nitric oxide (NO) plays significant roles in the diverse physiological processes: it reacts with proteins that act as biological messengers and regulate gene transcription, controls blood pressure, inhibits tumor growth, and modulates vasodilation [1–3]. NO is also a cytotoxic weapon generated by macrophages that injure cells by attacking the iron centers in varieties of proteins such as ferredoxins and ribonucleotide reductase [4]. In general, NO affects proteins by reaction with their metal active centers and thiols, however, its products

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Supporting Information Available

X-ray crystallographic file in CIF format for the structure determinations of complex $[\text{Fe}_2(\text{C}_{14}\text{H}_{12}\text{N}_3\text{S})_2(\text{NO})_4]$ ($\text{C}_{14}\text{H}_{12}\text{N}_3\text{S}$ = 2-mercapto-1-[2-(4-pyridyl)-ethyl]-benzimidazolyl). This data has been deposited to the Cambridge Crystallographic Data Center (CCDC 717919), it is available free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html.

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(NO_x, ONOO- and M-NO) are also observed to react with amino acid side chains, nucleic acids and a variety of low molecular weight amines [5].

At present, a number of investigations have shown that dinitrosyl iron complexes (DNICs) and S-nitrosothiols (RSNOs) are two possible forms for storage and transport of NO in biological systems. People have identified that DNICs serve as products of the biosynthetic evolution of NO *in vitro* and intermediates of the iron-catalyzed degradation and formation of RSNOs [3a–b,6]. For example, the anaerobic reaction of *C. Vinosum* high-potential iron protein (HiPIP) with nitric oxide produces protein-bound dinitrosyl iron complexes with a typical $g_{av} = 2.03$ EPR signal [5]. Therefore, the synthesis and investigation of biologically correlative dinitrosyl iron complexes with different ligands, which are able to produce diverse biological and chemical properties, bears significant meanings in order to understand mechanisms of NO storage and release in the organism. Over the past years, plenty of molecular models of DNICs containing a wide range of O/S/N/P ligation have been obtained [7], but only several papers concerning DNICs with bidentate [N,S] ligands have been reported [7e,8]. In this contribution, a new dinuclear iron nitrosyl complex [Fe₂(C₁₄H₁₂N₃S)₂(NO)₄] (C₁₄H₁₂N₃S = 2-mercapto-1-[2-(4-pyridyl)-ethyl]-benzimidazolyl) were isolated and characterized by IR, UV-vis, electrochemistry and single crystal X-ray diffraction.

2. Experimental Section

2.1 Materials and methods

Fe(NO)₂(CO)₂ was synthesized according to the published procedure [9]. Other chemicals were purchased from Aldrich Chemical Co. and were used without further purification. All solvents were purified and/or dried by standard techniques and degassed under vacuum prior to use. IR spectra were recorded on a Nicolet AVATAR 370 FTIR infrared spectrophotometer. UV-Visible spectra were measured on a Varian Cary 300 Bio UV-visible spectrophotometer.

2.2 Electrochemistry

Cyclic voltammetry (CV) was carried out with a CH Instruments electrochemical analyzer 730 A. A three-electrode system consisted of platinum working electrode, a platinum wire counter electrode and an Ag/Ag⁺ reference electrode was used. The reference electrode was separated from the bulk of the solution by a fritted-glass bridge filled with the solvent/supporting electrolyte mixture. The CV data were recorded with the scan rate of 100mV/s. The potential values are reported vs ferrocene/ferrocenium ion; the $E_{1/2}[\text{Fe}(\text{Cp})_2/\text{Fe}(\text{Cp})_2^+]$ under our experimental conditions is 0.21V for complex **1**.

2.3 X-ray analysis

Crystallographic data and structure refinements parameters of compound **1** are summarized in Table 1. The diffraction data of compound **1** were collected on a SMART CCD diffractometer with a graphite-monochromated Mo K α sealed tube ($\lambda = 0.71073\text{\AA}$) at 273 K, using a ω scan mode with an increment of 0.3°. Preliminary unit cell parameters were obtained from 45 frames. Final unit cell parameters were obtained by global refinements of reflections obtained from integration of all the frame data. The collected frames were integrated using the preliminary cell-orientation matrix. The empirical absorption correction was based on equivalent reflections and other possible effects such as absorption by the glass fiber were simultaneously corrected. Each structure was solved by direct methods followed by successive difference Fourier methods. All non-hydrogen atoms were refined anisotropically. Computations were performed using SHELXTL and final full-matrix refinements were against F^2 . The SMART software was used for collecting frames of data,

indexing reflections, and determination of lattice constants; SAINT-PLUS for integration of intensity of reflections and scaling; SADABS for absorption correction; and SHELXTL for space group and structure determination, refinements, graphics, and structure reporting [10–12].

2.4 Synthesis of complex 1

A mixture of $\text{Fe}(\text{NO})_2(\text{CO})_2$ (0.2 mL, 1.8 mmol) and 2-mercapto-1-[2-(4-pyridyl)-ethyl]-benzimidazole (460 mg, 1.8 mmol) in 30 mL methanol was stirred 96 hours at ambient temperature under nitrogen atmosphere. The reaction was monitored by FT-IR and the IR ν_{NO} stretching frequencies shifted from 1806 and 1759 cm^{-1} to 1788 and 1731 cm^{-1} . The reaction solution was filtered to remove dissolved impurity after putting at -35°C overnight. The residue was redissolved in CH_2Cl_2 and methanol was slowly added to the solution. The mixed solution was then left at -35°C in a glove box overnight to crystallize. The black crystal, suitable for X-ray crystallography, was collected by filtration and washed with methanol. The solid was dried under vacuum for several hours. Yield: 266 mg (40%). The complex is soluble in common organic solvents such as CH_2Cl_2 , THF and acetone. FT-IR ν_{NO} : 1789, 1732 cm^{-1} (CH_2Cl_2); 1788, 1731 cm^{-1} (CH_3OH); 1781, 1715 cm^{-1} (KBr). UV-vis spectrum (λ_{max}): 229, 287, 376(sh), 437nm(sh) (CH_2Cl_2).

3. Results and Discussion

3.1 Spectroscopic characterization

The complex **1** was obtained by the reaction of $\text{Fe}(\text{NO})_2(\text{CO})_2$ with 2-mercapto-1-[2-(4-pyridyl)-ethyl]-benzimidazole in CH_3OH under ambient condition. The reaction was performed under nitrogen atmosphere in a glove box and monitored by IR spectroscopy. Upon substitution, the red reaction solution turned to dark brown and red brown solid gradually generated. Meanwhile, the carbonyl stretching frequencies (ν_{CO} 2087 and 2037 cm^{-1}) disappeared, indicating that both carbonyl groups were replaced by the sulfur and nitrogen of the ligand; while the typical IR absorptions of nitrosyl groups (ν_{NO}) shift from 1806, 1759 cm^{-1} to 1788 and 1731 cm^{-1} , as shown in Figure 1, suggesting that the ligand act as an electron donor. The observations are consistent with the formation of dinuclear iron complexes as characterized by single crystal X-ray diffraction. The characteristic nitrosyl stretching frequencies (ν_{NO} , CH_2Cl_2 1789 and 1732 cm^{-1}) of complex **1** is similar to the reported values (ν_{NO} , (THF) 1789 and 1736 cm^{-1}) of DNICs with bidentate [N,S] ligands [7e]. When comparing with the dinuclear iron complexes $[\text{Fe}_2(\mu\text{-RS})_2(\text{NO})_4]$ (R = n-Pr, tBu, 6-methyl-2-pyridyl and 4,6-dimethyl-2-pyrimidyl) with [S,S] ligation [13] and cyclic tetranuclear iron complex $[\text{Fe}(\text{NO})_2(\text{Im-H})_4]$ (Im = imidazole) with [N,N] ligation [7c] (Scheme 1), their IR spectra bear the same pattern but differ in the position (ν_{NO} , (CH_2Cl_2) 1789 and 1732 cm^{-1} for **1** vs 1797–1775 and 1763–1748 cm^{-1} for $[\text{Fe}_2(\mu\text{-RS})_2(\text{NO})_4]$ and 1801 and 1735 cm^{-1} for $[\text{Fe}(\text{NO})_2(\text{Im-H})_4]$) and the separation of NO stretching frequencies $\Delta\nu_{\text{NO}}$ ($\sim 57 \text{ cm}^{-1}$ for **1** vs $\sim 29 \text{ cm}^{-1}$ for $[\text{Fe}_2(\mu\text{-RS})_2(\text{NO})_4]$ and $\sim 66 \text{ cm}^{-1}$ for $[\text{Fe}(\text{NO})_2(\text{Im-H})_4]$). These results suggest that the position and $\Delta\nu_{\text{NO}}$ of IR spectrum may serve as an efficient tool for the discrimination of the existence of the neutral dinuclear iron nitrosyl complexes containing the variety of [N,N]/[N,S]/[S,S] ligations.

The electronic absorption spectrum of complex **1** was measured in CH_2Cl_2 . As shown in Figure 2, it shows one strong band at 229 nm, one medium band at 286 nm, and two weak shoulder bands at 378 and 437 nm. The high-energy absorption bands at 229 and 286 nm were thought to be the ligand-localized $\pi \rightarrow \pi^*$ transition of the coordinated 2-mercapto-1-[2-(4-pyridyl)-ethyl]-benzimidazolyl. While two lower-energy absorption bands at 378 and 437 nm were assigned as an electron transition from metal to the ligand [15]. The observations are differed from dinuclear iron complexes $[\text{Fe}_2\text{S}_2(\text{NO})_4]$ [14] and $[\text{Fe}_2(\mu\text{-RS})_2(\text{NO})_4]$ (R =

n-Pr, tBu), which show one strong at 242, 239, 241 nm and two medium bands at 314, 312, 313 and 374, 363, 358 nm, respectively; but the electronic absorption spectrum of complexes **1** is in a slightly similar shape to complex $[\text{Fe}(\text{NO})_2(\text{Im-H})_4]$ [16], which shows one strong band at 248 nm, one medium band at 282 nm, and two weak shoulder bands at 351 and 513 nm. These results show that the absorption spectra of these dinuclear complexes are related to their ligands but not ligation mode to some extent.

3.2 Electrochemical properties

The redox behavior of complex **1** was studied by cyclic voltammetry (CV) in CH_2Cl_2 . The experiment was performed with the supporting electrolyte tetrabutylammonium hexafluorophosphate. The complex exhibited irreversible oxidations. It is consistent with the fact that this complex is very unstable in air. As shown in Figure 3, complex **1** exhibits two reversible one-electron reductions at -1.44 , -2.06V , two quasi-reversible one-electron reduction at -1.02 , -1.16V and one irreversible reduction at -0.72V . All of these reductions can be attributed to iron-sulfur-nitrogen-based and ligand-based redox processes. When comparing with dinuclear iron complexes $[\text{Fe}_2(\mu\text{-RS})_2(\text{NO})_4]$ with [S,S] ligation (half-wave potentials $E_{1/2} = -1.16$, -1.84V and -1.20 , -1.81V for $\text{R} = \text{n-Pr}$ and tBu ; $E_{1/2} = -0.99$ and -0.91V for $\text{R} = 6\text{-methyl-2-pyridyl}$ and $4,6\text{-dimethyl-2-pyrimidyl}$), complex **1** possesses more reduction process and more positive value for the first reduction peak, showing that this complex is easier to be reduced as its ligand bearing less electron donor effect and the redox properties of different kinds of dinuclear iron nitrosyl complexes are significantly influenced by the electron properties of the ligand.

3.3 Structural studies

The molecular structure of complex **1** was determined by X-ray diffraction analysis. Single crystal of the complex was obtained by slow diffusion of MeOH into the corresponding CH_2Cl_2 solutions at -35°C under nitrogen atmosphere. As shown in Figure 4, the complex shows a centrosymmetric dinuclear “chair-shape” structure by the linkage of iron, sulfur and nitrogen atoms. The selected bond lengths and bond angles are listed in Table 2. Two benzimidazolyl planes are almost parallel to each other along opposite direction and form an angle of $\sim 113^\circ$ with the 2Fe-2S plane. The $\text{Fe}(1)\text{-Fe}(1a)$ distance of ca $4.14\text{ \AA}$ shows that there is almost no interaction between the two iron centers, which is clearly differed from dinuclear iron complexes $[\text{Fe}_2(\mu\text{-RS})_2(\text{NO})_4]$ (ca $2.70\text{ \AA}$) [13]. The Fe-S and $\text{Fe-N}(\text{imidazole})$ bond distance for complex **1** is $2.3032(7)$ and $2.0222(16)\text{ \AA}$, respectively, which is longer than the relevant Fe-S distance (ca $2.267\text{ \AA}$) in the $[\text{Fe}_2(\mu\text{-RS})_2(\text{NO})_4]$ [13] and $\text{Fe-N}(\text{imidazole})$ (ca $2.009\text{ \AA}$) in the $[\text{Fe}(\text{NO})_2(\text{Im-H})_4]$ [7c]. In fact, complex **1** is more stable than the latter two kinds of complexes in air. The average $\text{Fe-N}(\text{NO})$ bond distance for complex **1** is $1.683\text{ \AA}$, which is similar to the reported value in the $[\text{Fe}_2(\text{SC}_7\text{H}_4\text{SN})_2(\text{NO})_4]$ ($\text{SC}_7\text{H}_4\text{SN} = 2\text{-benzothiazolyl thiolate}$) with bidentate [N,S] ligands (average value: $1.687\text{ \AA}$) [7e], but slightly longer than the reported value in the dinuclear complex $[\text{Fe}_2(\mu\text{-RS})_2(\text{NO})_4]$ ($\text{R} = \text{n-Pr}$, tBu , $6\text{-methyl-2-pyridyl}$ and $4,6\text{-dimethyl-2-pyrimidyl}$) with [S,S] ligation (average value: $\sim 1.670\text{ \AA}$) [13], and slightly shorter than the reported value in the tetramer $[\text{Fe}(\text{NO})_2(\text{Im-H})_4]$ with [N,N] ligation (average value: $1.694\text{ \AA}$) [7c]. These results suggest that the NO bondage with iron is closely interconnected to the ligands and ligation atoms for these neutrally dinuclear iron nitrosyl complexes, therefore, further enhance the understanding on the properties and possible NO release mechanism of different variety of nitrosyl iron complexes. Interestingly, the differences between the two Fe-N-O bond angles on each iron are small for the complexes $[\text{Fe}_2(\mu\text{-RS})_2(\text{NO})_4]$ and $[\text{Fe}(\text{NO})_2(\text{Im-H})_4]$, ($\sim 1.3^\circ$ and $\sim 1.1^\circ$ apart, respectively), while a bigger value (4.8°) was observed for the complex **1**. This is attributed to the different circumjacent environment of the two NOs on each iron, leading to dissimilar bending of the two NOs.

4. Conclusions

In summary, a new dinuclear iron nitrosyl complex $[\text{Fe}_2(\text{C}_{14}\text{H}_{12}\text{N}_3\text{S})_2(\text{NO})_4]$ ($\text{C}_{14}\text{H}_{12}\text{N}_3\text{S} = 2\text{-mercapto-1-[2-(4-pyridyl)-ethyl]-benzimidazolyl}$) was prepared. The complex has been fully characterized by IR, UV-vis, electrochemistry and single crystal X-ray diffraction method. The results were compared with experimental parameters of relevant complexes with [N,N] and [S,S] ligation. Infrared spectrum shows that the position and $\Delta\nu_{\text{NO}}$ of IR spectrum may serve as an efficient tool for the discrimination of the existence of the neutral dinuclear iron nitrosyl complexes containing the variety of [N,N]/[N,S]/[S,S] ligations; electrochemical data suggest that redox property of different kinds of dinuclear iron nitrosyl complexes is significantly influenced by the electron properties of the ligand; and structural studies help understanding the relationship between structures and the stability and possible NO release mechanism of different variety of dinuclear iron nitrosyl complexes.

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

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Figure 1.
Infrared spectrum of complex **1** in KBr.

Figure 2.
Electronic absorption spectrum of complex **1** in CH₂Cl₂.

Figure 3.

Cyclic voltammograms of a 2mM solution of complex **1** in 0.1 M [NBu₄][PF₆]/CH₂Cl₂, scan rate: 100 mV/S.

Figure 4.
Molecular structure of complex **1** with thermal ellipsoids drawn at the 30% probability.

Scheme 1.

Schematic structure of complex **1** (a), $[\text{Fe}(\text{NO})_2(\text{Im-H})]_4$ (b) [7c], $[\text{Fe}_2(\mu\text{-RS})_2(\text{NO})_4]$ (c) [13] and $[\text{Fe}_2(\text{SC}_7\text{H}_4\text{SN})_2(\text{NO})_4]$ (d) [7e].

Table 1Crystallographic data collection and refinement parameters for the complex **1**.

formula	$\text{C}_{28}\text{H}_{24}\text{N}_{10}\text{O}_4\text{S}_2\text{Fe}_2$
M_r	740.41
size (mm^3)	$0.32 \times 0.21 \times 0.12$
T (K)	273(2)
$\lambda(\text{\AA})$	0.71073
crystal system	Monoclinic
space group	P2(1)/c
a ($\text{\AA}$)	10.43940(10)
b ($\text{\AA}$)	16.0900(2)
c ($\text{\AA}$)	10.13240(10)
$\alpha(^{\circ})$	90
$\beta(^{\circ})$	111.0940(10)
$\gamma(^{\circ})$	90
$V(\text{\AA}^3)$	1587.89(3)
Z	4
$F(000)$	756
d (g cm^{-3})	1.549
$\mu(\text{mm}^{-1})$	1.097
range $\theta(^{\circ})$	2.09 to 25.12
Reflec. collected	9830
Indep. reflec.	2817 ($R_{\text{int}}=0.0420$)
$R1$ [$I > 2\sigma(I)$]	0.0299
$wR2$ [$I > 2\sigma(I)$]	0.0799
$R1$ (all data)	0.0394
$wR2$ (all data)	0.0846
goodness of fit	0.971

Table 2Selected Bond Lengths (Å) and Bond Angles (°) for complex **1**.

Fe1-Fe1a	4.136	Fe1-S1a	2.3032(7)
Fe1-N1	1.679(2)	N1-O1	1.153(3)
Fe1-N2	1.687(2)	N2-O2	1.161(2)
Fe1-N4	2.0222(16)	S1-C7	1.7336(19)
O1-N1-Fe1	169.1(2)	N1-Fe1-S1a	108.00(8)
O2-N2-Fe1	164.3(2)	N2-Fe1-S1a	102.98(7)
N1-Fe1-N2	115.28(11)	N4-Fe1-S1a	108.08(5)
N1-Fe1-N4	111.60(9)	C7-S1-Fe1a	106.44(7)
N2-Fe1-N4	110.29(8)	N4-C7-S1	126.26(14)