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# Synthesis, X-ray structure and characterization of a novel $[\text{fc}(\text{IMH})_2\text{H}]^+[\text{Co}(\text{hfac})_3]^-$ salt with hydrogen bonded ferrocenyl bis(imino hydroxylamino) building blocks

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Dedicated to Professor Ernest Otto Fischer, Pioneer of Organometallic Chemistry on the occasion of his 85th birthday

## Abstract

The reaction of ferrocenylene bis( $\alpha$ -nitronyl nitroxide) diradical (**1**;  $[\text{fc}(\text{NIT})_2]$ ) with  $\text{Co}(\text{hfac})_2$  produces the reduction of the radical character of **1** with the formation of the diamagnetic ferrocenyl bis( $\alpha$ -imino nitroxide) cation,  $[\text{fc}(\text{IMH})_2\text{H}]^+$ , which forms with the  $[\text{Co}(\text{II})\text{hfac}_3]^-$ , as the counterion, the complex salt **4**. This salt crystallizes in the  $P\bar{1}$  spatial group exhibiting an exotic supramolecular architecture in which the molecular ions are bonded through hydrogen bonds forming single chains with a zipper-like arrangement. Remarkable is the presence in complex salt **4** of two proton tautomeric forms in the  $[\text{fc}(\text{IMH})_2\text{H}]^+$  units.

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**Keywords:** Metallocene; Nitronyl nitroxides; Reduction; Complex salt; Hexafluoroacetylacetonate

## 1. Introduction

The first example of an  $\alpha$ -nitronyl nitroxide radical was synthesized in 1968 by Ullman and co-workers [1]. Since then, their use as building blocks in the preparation of organic magnetic molecular materials [2] has attracted a great deal of interest because of their exceptional stability and their rich chemistry. Such stability has allowed the obtaining of several  $\alpha$ -nitronyl nitroxides bearing different groups at the  $\alpha$ -position, some of them even exhibiting a bulk ferromagnet behavior at low temperatures, like the  $\beta$ -phase of the *p*-nitrophenyl nitronyl nitroxide radical [3]. However, even though several families of radicals have been up to now synthesized, the critical temperature ( $T_C$ ) above which such radicals order magnetically is very low,

generally below 2 K. To overcome such inconvenient the so-called metal-radical approach, which was first reported by Gatteschi and Rey [4], can be used. This approach benefits from the ability of nitroxides to act as ligands in metal complexes, on condition that a strong magnetic exchange coupling between is established. Although it usually has an antiferromagnetic nature, transition metal ions with  $S > 1/2$  can be used to avoid a total compensation between the spins of the radical and the metal.

Choosing the metal-radical approach, several structural motifs have been so far reported, most of them exhibiting low dimensionality. Certainly, up to now, different metal nitronyl nitroxides complexes ranging from monomers, dimeric clusters and hexameric ring structures [5] up to polymeric one-dimensional chains [6–8] have been obtained. However, to obtain transition metal complexes having two- and three-dimensional architectures, the use of poly-nitronyl nitroxides, bearing two or more radical groups within the same unit, has been implemented [9]. In this order, recently we have

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synthesized a series of  $\alpha$ -nitronyl nitroxide diradicals **1** (M: Fe, Ru) connected by different 1,1'-metallo-cenylene bridges [10]. The advantages of using such bridges are twofold. First, they have been shown to be effective magnetic couplers transmitting the magnetic interactions through their skeletons via a spin polarization mechanism [11]. And second, the conformational flexibility of metallocenylene  $\alpha$ -nitronyl nitroxides makes them interesting candidates for transition metal complexes because this degree of freedom will permit to obtain novel supramolecular architectures.

Following this former idea, metallocenylene  $\alpha$ -nitronyl nitroxide diradicals have been shown to react with transition metal hexafluoroacetylacetonates complexes in nonpolar solvents to form different coordination compounds. For instance, a polymeric complex **2** showing spontaneous resolution of the two possible enantiomeric forms, and consequently optical activity, has been obtained [12]. However, if the same reaction is carried out at higher temperatures, a partial reduction of the radicals to diamagnetic compounds occurs, yielding the dimeric complex **3**. Its structure consists of two metallocenylene nitronyl nitroxide moieties coordinating two manganese ions through the reduced radical in a butterfly arrangement, in which competitive magnetic interactions lead to a rare spin frustration phenomenon [13].

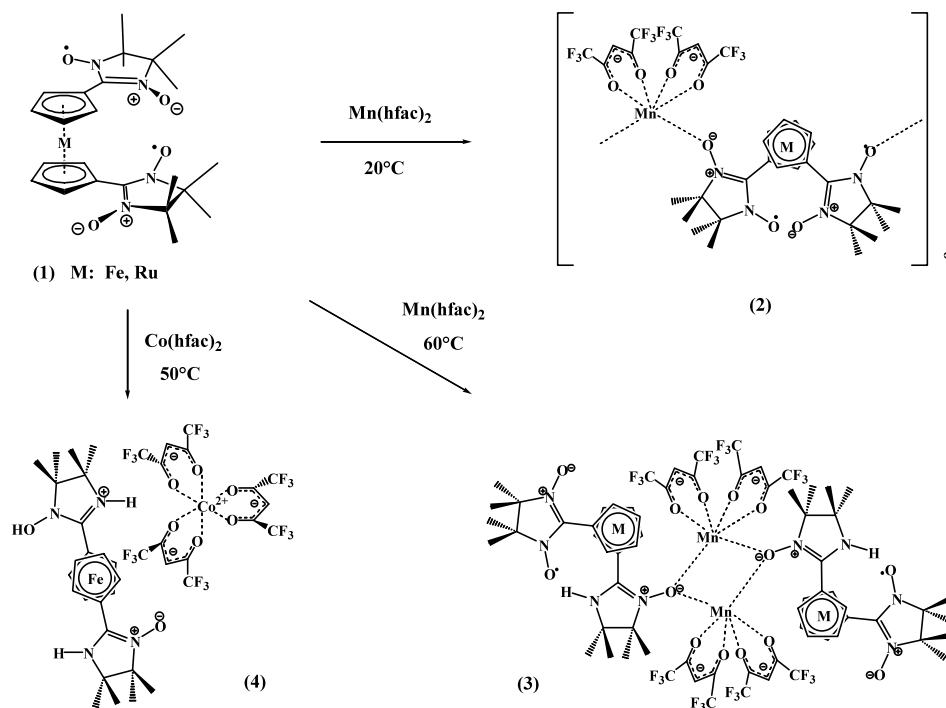
Here we report for the first time the reaction of a 1,1'-ferrocenylene diradical with  $\text{Co}(\text{hfac})_2$  to yield a salt consisting of a cationic ferrocene part  $[\text{fc}(\text{IMH})_2\text{H}]^+$ , where both radical moieties have been fully reduced to

the diamagnetic iminonitroxide (IMH) form, and an anionic  $[\text{Co}(\text{hfac})_3]^-$  unit.

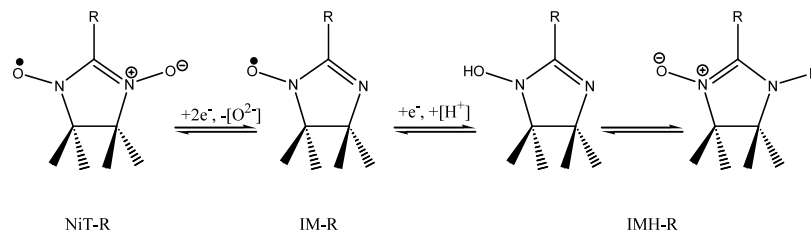
## 2. Results and discussion

Reaction of ferrocenylene bis( $\alpha$ -nitronyl nitroxide) (**1**; M = Fe) with  $\text{Co}(\text{hfac})_2$  in *n*-heptane at 50 °C (see Scheme 1) leads to a color change within a few minutes, from the initial dark-green to the final red-brown color. This fact is a qualitative indication that the reduction of the radical is taking place. Indeed, it is already known that transition metals can cause reduction of  $\alpha$ -nitronyl nitroxides (NIT) to their corresponding diamagnetic  $\alpha$ -imino nitroxide (IMH) form, in a two-step reduction process (see Scheme 2) [14a–e]. In all the examples so far reported, the resulting reduced radicals bind to the metal ion through the oxygen atom of the amidino-oxide group although in the present case, the resulting brownish crystalline material was characterized to be the salt  $[\text{fc}(\text{IMH})_2\text{H}][\text{Co}(\text{hfac})_3]$  (**4**) by X-ray crystallography. From here, two results deserve to be emphasized. First, both radical units of the ferrocene-based diradical have been reduced and second, the lack of coordination to the metal ion through the oxygen atom of the amidino-oxide group.

As shown in Fig. 1, the molecular structure is unique in several other terms. Compound **4** consists of a cationic ferrocene-based unit  $[\text{fc}(\text{IMH})_2\text{H}]^+$ , in which both radical moieties are fully reduced to the diamagnetic  $\alpha$ -imino nitroxide (IMH) form, and an anionic



Scheme 1.



Scheme 2.

[Co(hfac)<sub>3</sub>]<sup>−</sup> unit. The cationic ferrocene part [fc(IMH)<sub>2</sub>H]<sup>+</sup> holds an inversion centre located at the iron atom. In this way, both IMH groups on each molecule are equivalent and arranged in a *transoid* manner, making the two oxygen atoms to face away from each other. On the other side, in the [Co(hfac)<sub>3</sub>]<sup>−</sup> unit the Co(II) ion is surrounded by three hfac ligands in a nearly undistorted octahedral coordination sphere with Co–O distances similar to those described in the literature, ranging from 2.050(5) to 2.071(5) Å [15] (Table 1).

As far as intermolecular interactions are concerned, strong hydrogen bonds between two oxygen atoms of neighboring reduced ferrocene-based molecules are present, with short intermolecular distances of O1···O2A (2.072 Å) and O1A···O2 (1.783 Å). Such short O···O values should not be overestimated because of correlation effects in the two disordered models. The existence of such hydrogen bonds may be explained if a proton resides alternatively on O1A or O2A, i.e. if the amidinoxide form of one molecule interacts with the protonated IMH group of the immonium-hydroxylamino form of a neighboring molecule. For this to be achieved, the two tautomeric forms shown in Scheme 2 must coexist within the same crystal structure.

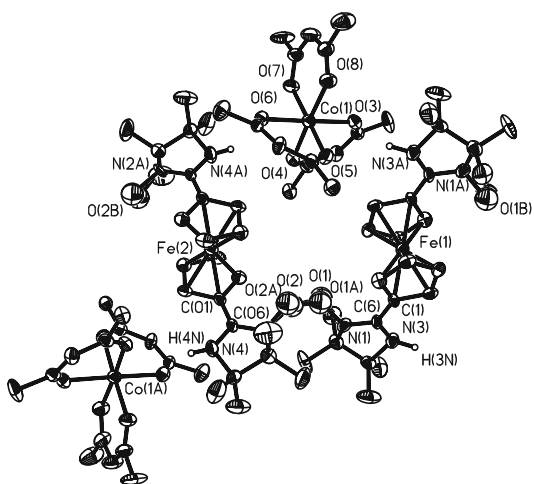


Fig. 1. View of the symmetric unit of complex salt **4**. Thermal ellipsoids are presented at the 30% probability level and fluorine and hydrogen atoms (except those on amino groups) are omitted for clarity.

Such an assignment is complicated by the fact that in the crystal structure both IMH-groups are superimposed because of a 1:1 statistically disorder, which makes it impossible to distinguish between the two forms exactly. Moreover, in the hydroxylamino form the heterocycle is not planar; in fact two slightly displaced oxygen for each ring can be found in the crystal structure determination, both with the same intensity. Previous related examples reported in the literature indicate that typical N–O bond lengths amidinoxides range from 1.36 to 1.40 Å [13,14]. For hydroxylamines, the bond lengths range from 1.407(6) Å [16] to 1.46 Å, the latter value being observed for the 1,3-bis hydroxyimidazolidine precursor of the *p*-nitrophenyl nitronyl nitroxide [17]. Finally, in the crystal structure of complex **4** the bond lengths found are N1–O1: 1.42(3) Å; N1–O1A: 1.43(3) Å; N2–O2: 1.470(19) Å and N2–O2A: 1.386(19) Å; thus located in the average range between the amidinoxide and hydroxylamine tautomeric forms.

For all other atoms of the imidazole heterocycles the disordering is too small to be found, apart from higher displacement factors present for the methyl groups. This fact is not surprising at all if we consider the small differences in bond lengths found in the literature. Indeed, typical C–N double bonds in amidinoxides range from 1.30 to 1.32 Å [13,14] whereas in the only example so far reported of a hydroxylamino form, the C–N bond length is 1.29 Å [16]. For single C–N bonds between the alpha carbon and the nitrogen atom, the values range between 1.34 and 1.36 Å [13,14,16].

Finally, additional hydrogen bonds between the amino- and immonium H-atoms and the oxygen atoms of the hfac groups were also observed (see Scheme 3). The bond lengths are H4N···O4: 2.529 Å; H4N···O6: 2.575 Å; H3N···O3: 2.574 Å and H3N···O5: 2.481 Å. This hydrogen bonds are leading to a repetitive arrangement of the cationic [fc(IMH)<sub>2</sub>H]<sup>+</sup> and anionic [Co(II)(hfac)<sub>3</sub>]<sup>−</sup> units, where single chains with a zipper-like structure are shielded from each other by means of their bulky CF<sub>3</sub> groups (see Fig. 2), which are statistically disordered (1:1).

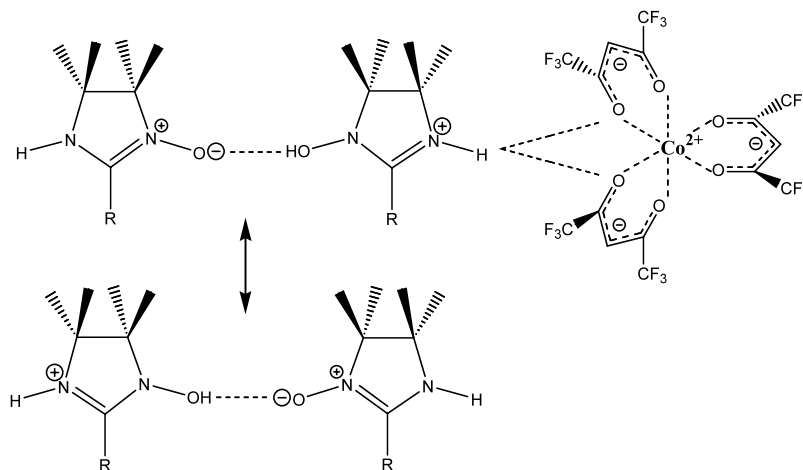
To fully address of two proton tautomeric forms, the infrared spectra of a microcrystalline sample of **4** was collected. The resulting FT-IR spectrum exhibits a sharp band at 3361 cm<sup>−1</sup>, characteristic of a N–H vibration

Table 1  
Selected bond lengths (Å) and bonds angles (°) for 4

Bond lengths			
N(1)–O(1)	1.42(3)	N(4)–C(06)	1.334(8)
N(1)–O(1A)	1.43(3)	C(01)–C(06)	1.451(9)
N(1)–C(6)	1.304(8)	Co(1)–O(3)	2.071(5)
N(3)–C(6)	1.325(9)	Co(1)–O(4)	2.077(5)
C(1)–C(6)	1.448(10)	Co(1)–O(5)	2.068(5)
N(2)–O(2A)	1.386(19)	Co(1)–O(6)	2.060(5)
N(2)–O(2)	1.470(19)	Co(1)–O(7)	2.051(5)
N(2)–C(06)	1.294(8)	Co(1)–O(8)	2.050(5)
Bond angles			
C(6)–N(1)–O(1)	122.1(14)	O(3)–Co(1)–O(6)	178.8(2)
C(6)–N(1)–O(1A)	121.9(13)	O(4)–Co(1)–O(8)	176.38(19)
N(1)–C(6)–N(3)	109.0(7)	O(5)–Co(1)–O(7)	176.4(2)
N(1)–C(6)–C(1)	126.2(8)	O(3)–Co(1)–O(5)	92.88(18)
N(3)–C(6)–C(1)	124.8(6)	O(3)–Co(1)–O(7)	89.93(19)
C(06)–N(2)–O(2)	121.3(9)	O(3)–Co(1)–O(8)	88.9(2)
C(06)–N(2)–O(2A)	122.7(10)	O(4)–Co(1)–O(5)	92.80(19)
N(2)–C(06)–N(4)	108.6(7)	O(4)–Co(1)–O(6)	93.47(19)
N(2)–C(06)–C(01)	127.1(7)	O(4)–Co(1)–O(7)	89.6(2)
N(4)–C(06)–C(01)	124.3(6)	O(7)–Co(1)–O(6)	89.6(2)
O(3)–Co(1)–O(4)	87.57(19)	O(8)–Co(1)–O(5)	88.0(2)
O(5)–Co(1)–O(6)	87.58(19)	O(8)–Co(1)–O(6)	90.10(19)
O(7)–Co(1)–O(8)	89.8(2)		

and a broad band between 3100 and 3600  $\text{cm}^{-1}$  characteristic of O–H vibrations. An additional absorption at 1601  $\text{cm}^{-1}$  of medium intensity, characteristic of C–N double bonds in reduced iminonitroxides, is also observed.

Variable-temperature magnetic susceptibility data between 2 and 300 K were in agreement with the theoretical expectations. At 300 K, the  $\mu_{\text{eff}}$  moment was 3.7  $\mu_{\text{B}}$ , a value that is close to that expected for a high-spin Co(II) ( $S = 3/2$ , 3.5  $\mu_{\text{B}}$ ) complex. As the temperature is decreased,  $\mu_{\text{eff}}$  remains constant down to very low temperatures where a decrease of  $\mu_{\text{eff}}$  is observed due to the presence of zero-field splitting and the presence of weak antiferromagnetic interactions.



Scheme 3.

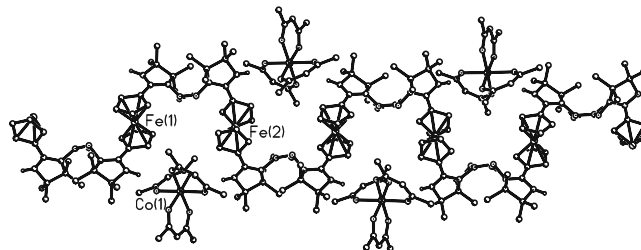


Fig. 2. Zipper-like arrangement of the complex salt 4. Fluorine and hydrogen atoms (except those on amino groups) are omitted for clarity.

### 3. Conclusion

Co(hfac)<sub>2</sub> complex is able to reduce the ferrocenyl bis( $\alpha$ -nitronyl nitroxide) diradical 1 with producing the diamagnetic ferrocenyl bis( $\alpha$ -imino nitroxide) cation, [fc(IMH)<sub>2</sub>H]<sup>+</sup>, which forms with the [Co(II)hfac<sub>3</sub>]<sup>-</sup>, as the counterion, the complex salt 4. This salt exhibits an exotic supramolecular architecture in which the molecular ions are bonded through hydrogen bonds forming single chains with a zipper-like arrangement. Remarkable is the presence in complex salt 4 of two proton tautomeric forms in the [fc(IMH)<sub>2</sub>H]<sup>+</sup> units.

### 4. Experimental

#### 4.1. General considerations

All procedures were carried out under Argon atmosphere using standard Schlenk techniques. CH<sub>2</sub>Cl<sub>2</sub> was distilled over P<sub>2</sub>O<sub>5</sub>; *n*-heptane (99.5%) and Co(hfac)<sub>2</sub>·2H<sub>2</sub>O were purchased from Aldrich Chemical Company and used as received. Synthesis of the metallocenylene bis( $\alpha$ -nitronyl nitroxides) was described earlier [10].

#### 4.2. Synthesis of salt 4

One hundred milligram  $\text{Co}(\text{hfac})_2 \cdot 2\text{H}_2\text{O}$  (0.19 mmol) were refluxed in 10 ml of dry *n*-heptane and the water was removed by azeotropic distillation. A solution of 50 mg (0.10 mmol) of the ferrocenylene diradical 1 ( $M = \text{Fe}$ ) in 2 ml of dry  $\text{CHCl}_3$  was added under stirring; the color of the solution changes from dark green to red-brown during 5 min. The solution was filtered and the solid that precipitated was recrystallized in dry  $\text{CHCl}_3$ . After 1 month orange-brown crystals, suitable for X-ray structure determination could be collected. IR data ( $\text{KBr}$ ,  $\text{cm}^{-1}$ ): 3600–3100 (w, OH); 3361 (m, NH); 3118 (w); 1643 (s-vs); 1601 (m, C=N–); 1559 (s); 1530 (s); 1487 (s); 1399 (m); 1377 (m); 1260 (vs); 1202 (vs); 1150 (vs); 1095 (s); 1024 (s); 887 (w-m); 799 (s); 735 (m); 669 (s); 582 (s); 480 (s). The melting point could not be determined since decomposition in a broad range occurs. Satisfactory elemental analysis (C, H, N) were obtained.

#### 4.3. Crystal data and refinement details for 4

*Color, Habit*: orange-brown plate, *Crystal dimension*:  $0.1 \times 0.1 \times 0.04 \text{ mm}^3$ .  $\text{C}_{39}\text{H}_{38}\text{CoF}_{18}\text{FeN}_4\text{O}_8$ ,  $M = 1147.51$ , triclinic  $P\bar{1}$ ,  $a = 10.0422(9) \text{ \AA}$ ,  $b = 14.898(1) \text{ \AA}$ ,  $c = 16.585(1) \text{ \AA}$ ,  $\alpha = 85.110(5)^\circ$ ,  $\beta = 77.943(4)^\circ$ ,  $\gamma = 83.906(5)^\circ$ ,  $V = 2407.7(3) \text{ \AA}^3$ ,  $T = 223(2) \text{ K}$ ,  $Z = 2$ ,  $\text{Mo-K}\alpha$  ( $\lambda = 0.71073 \text{ \AA}$ ), 8769 reflections collected, 4493 independent reflections with  $I > 2s(I)$ ,  $R_1 [I > 2s(I)] = 0.0522$ ,  $wR_2$  (all data) = 0.1299, GooF: 0.979. Data were measured with a Nonius Kappa CCD and the structure was solved by direct methods (SHELXS-97) and refined by full-matrix-least-squares methods on  $F^2$  (SHELXL-97) [18]. Hydrogens on N(3) and N(4) were found and refined separate.

### 5. Supplementary material

Crystallographic data for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre, CCDC 214907 for 4. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; email: deposit@ccdc.cam.ac.uk or www: <http://www.ccdc.cam.ac.uk>).

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