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Synthesis, structure and reactivity study of 1,1′-bis(*N*-*ter*-butyl-*N*-hydroxyamino)ferrocene

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Abstract

The synthesis of 1,1′-bis(*N*-*ter*-butyl-*N*-hydroxyamino)ferrocene **1**, is described, and the X-ray analysis showed eclipsed positions of the two *ter*-butylhydroxyamino groups; this structure can be explained by the interactions of the hydroxyl hydrogens with the nitrogen atoms of the other *ter*-butylhydroxyamino group. This interaction is maintained in solution and may explain that the oxidation of **1** using Ag_2O gave a decomposition reaction instead of the expected 1,1′-bis(*N*-*ter*-butylaminoxy)ferrocene. © 2002 Published by Elsevier Science B.V.

Keywords: Ferrocene; Radical; Hydroxylamine; X-ray

1. Introduction

In the last few years, the development and study of new coupling units that promote ferromagnetic interactions between pure organic radicals has been a subject of interest [1].

During our search in the synthesis, structure, and magnetic properties of molecules bearing two or more groups with unpaired electrons, as for instance *ter*-butyl nitroxide [2], we envisioned the case of metallocene, and more precisely ferrocene, as magnetic coupler. These complexes have been successfully used by Miller and Epstein as building blocks of molecular solids promoting intermolecular magnetic exchange interaction [3]. Their use as intramolecular magnetic coupler was studied by Veciana et al. in the case of two polychlorinated triphenylmethyl radical units connected by a 1,1′-metallocenylendivinylene bridge [4]; the same author studied the case of 1,1′-metallocenes (ferrocene and ruthenocene) as coupler of two nitronylaminoxide radicals, and found that the metallocene units were able to transmit magnetic interactions by a spin polarisation mechanism [5].

Interestingly, the disubstituted 1,1′-ferrocene derivatives may chose different conformations which favour intra- or intermolecular interactions. Indeed, if the trans form of 1,1′-ferrocene is frequent, due to specific interactions, the conformation may be different. For instance, Togni et al. find a completely eclipsed conformation in the case of 1,1′-bis(5,6-dihydro-1,3-dithiolo[4,5-*b*][1,4]dithiin-2-ylidene)methyl]ferrocene which they explained by intramolecular stacking [6]. Veciana and coworkers observed a cisoid conformation in the case of 2,2′-di-(1,1′-ferrocenediyl)-4,4,5,5-tetramethyl-4,5-dihydro-1H-imidazol-3-oxide-1-yl oxyl diradical due to an intramolecular hydrogen bond between one methyl group of one radical subunit and one of the two N–O groups of the other radical subunit [5].

2. Results and discussion

In our case, we will report on the synthesis, structure and reactivity of 1,1′-bis(*N*-*ter*-butyl-*N*-hydroxyamino)ferrocene **1**.

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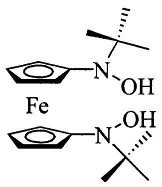
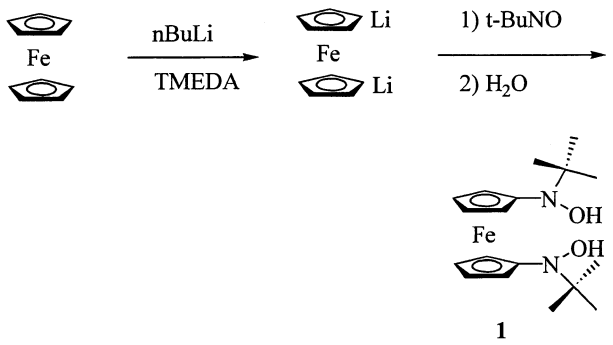


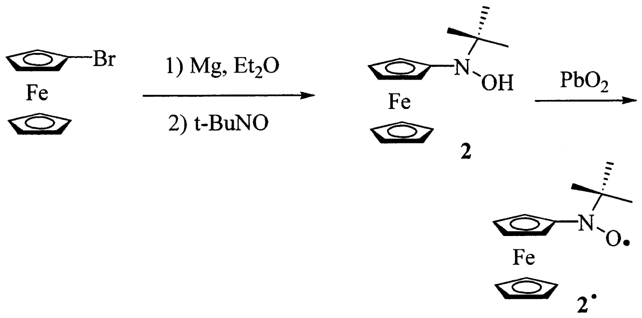
1

The *ter*-butylhydroxyamine group was chosen because it is easily grafted to organic moieties and its oxidation is easy and gives usually stable radicals. We have synthesised **1** according to Eq. (1).



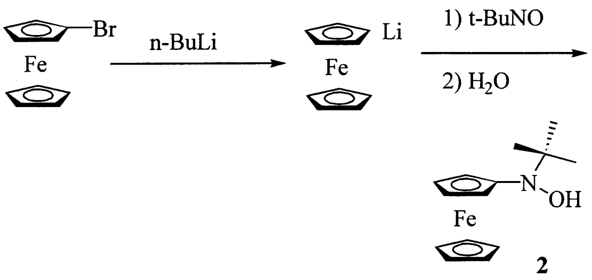
(1)

Compound **1** was fully characterised by IR, NMR and elemental analysis. The ^{1}H-NMR spectrum ($CDCl_3$) was unusual. Indeed, the hydroxyl protons appeared as a sharp signal, at very low field, $\delta = 9.6$ ppm, independently of the concentration. This position was confirmed by D_2O exchange. As a comparison, ferrocenyl-*N*-*ter*-butyl-*N*-hydroxylamine **2** was prepared; the latter compound was synthesised several years ago according to Eq. (2), but all physical properties were not given [7].



(2)

We were unable to reproduce this Grignard procedure reaction and prepared **2** using the condensation of the organolithium derivative with 2-methyl-2-nitrosopropane (Eq. (3)).



(3)

The ^{1}H-NMR spectrum ($CDCl_3$) gave the hydroxyl proton at $\delta = 4.8$ ppm, i.e. a more usual chemical shift for this group. The latter value confirms the special structure of compound **1**.

In order to solve this structure, we did a single crystal X-ray analysis. Table 1 gives the crystal data, Fig. 1 shows the ORTEP view and atomic numbering scheme of the hydroxylamine **1**, and Table 2 gives selected interatomic distances and bond angle for **1**.

The total number of collected reflections was 3664. After semi-empirical absorption corrections and elimination of the systematic absences, 2664 data were used in the calculation. The structure readily solved (SHELXS-86 program [8]) in either centro *A*2/*a* or non-centrosymmetric *Aa* space group, another setting of the standard groups *C*2/*c* (No 15) and *Cc* (No 9), respectively. The probability tests given by the program seemed to suggest a slight preference for a non-centrosymmetrical arrangement of the atoms in the

Table 1
Summary of crystal data

Empirical formula	$C_{18}H_{28}FeN_2O_2$	
Formula mass	360.28	
Crystal colour	Colourless	
Recryst. Solv.	Et_2O	
M.p. (°C)	142–144	
Crystal size (mm)	0.65 × 0.30 × 0.05	
Temperature (K)	173	
Radiation	Mo–K_α	
a (Å)	11.024(1)	
b (Å)	9.396(1)	
c (Å)	17.956(5)	
β (°)	106.94(2)	
V (Å^3)	1779.1(6)	
Z	4	
$D_{calc.}$ (g.cm^{-3}) at 175 K	1.3456	
Measured reflections	3664	
Unique reflections	2603	
Observed reflections	2500	
Crystal system	Monoclinic	
Space group	*A*2/*a* (No 15)	*Aa* (No 9)
Number of variables	145	286
R_1, 1612 $F_O > 4\ \sigma(F_O)$	0.0357	0.0339
wR_2 (on F_2)	0.0733	0.0681
Goodness-of-fit	0.937	0.899
Res. electron density	0.43(−0.49)	0.27(−0.52)

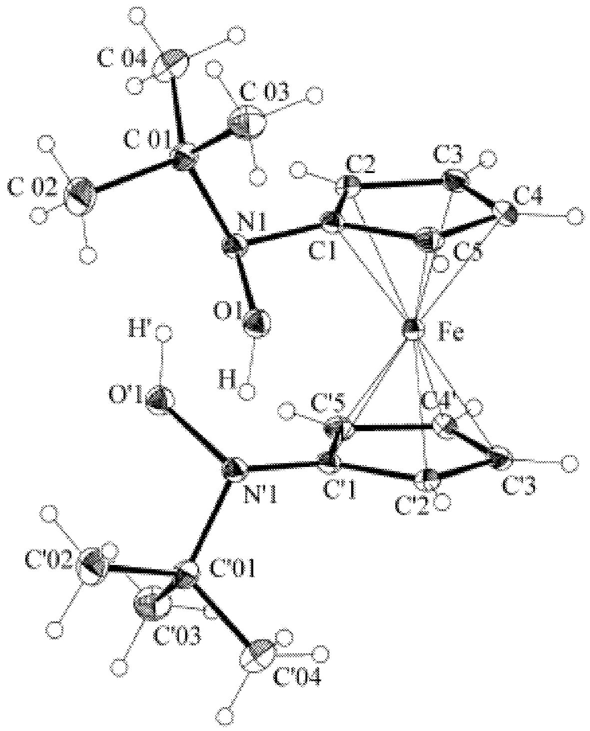


Fig. 1. ORTEP plot of **1** corresponding to *A*2/*a* space group. For the *Aa* space group, atoms C′(1–5) correspond to C(6–10), N′1 to N2, O′1 to O2 and C′(01–04) to C(05–08).

Table 2
Selected bond distances (Å) and angles (°) for **1**

Fe–C1	2.038(2)	C1–N1	1.438(2)
Fe–C2	2.038(2)	N1–C01	1.497(2)
Fe–C3	2.046(2)	C01–C02	1.520(3)
Fe–C4	2.044(2)	C01–C03	1.521(3)
Fe–C5	2.041(2)	C01–C04	1.523(3)
C1–C2	1.420(2)	N1–O1	1.526(2)
C2–C3	1.419(2)	N1–O1a	1.544(3)
C3–C4	1.414(2)	O1–H	0.972(4)
C4–C5	1.417(3)	O1a–H	0.827(4)
C5–C1	1.419(2)	O1···O1′	1.739(3)
C5–C1–C2	108.1(2)	C01–N1–O1	104.2(1)
C1–C2–C3	107.8(2)	C01–N1–O1a	103.2(2)
C2–C3–C4	108.0(2)	N1–C01–C02	106.4(2)
C3–C4–C5	108.3(2)	N1–C01–C03	109.8(2)
C4–C5–C1	107.7(2)	N1–C01–C04	110.3(2)
C5–C1–N1	126.1(2)	C02–C01–C03	110.5(2)
C2–C1–N1	125.7(2)	C03–C01–C04	109.9(2)
C1–N1–C01	115.5(1)	C04–C01–C02	110.0(2)
C1–N1–O1	105.4(1)	N1–O1a···O′1	112.5(2)
C1–N1–O1a	105.7(2)	N1–O1···O′1a	115.8(2)
O1–N1–O1a	123.5(2)		

crystal (*0kl*, *h0l*, *hk0* and *Rest* values were: 0.921, 0.719, 0.881 and 0.736).

A full determination of the structure was achieved with the *Aa* space group. Refinement of the whole molecule, with 286 parameters for 2664 data (equivalent reflections not merged; 2559 $F_o > 4\ \sigma(F_o)$) gave $R_1 =$ 0.0339 and $wR_2 = 0.0681$ with the Flack *x* parameter = 0.037(47) in accordance with an acentric group. The goodness of fit was $S = 0.899$. Moreover, another refinement with 61 merged Friedel pairs gave fairly similar results: 286 parameters for 2609 unique data (2500 $F_o > 4\ \sigma(F_o)$) gave $R_1 = 0.0336$ and $wR_2 = 0.0666$ with the Flack *x* parameter = 0.036(47) and $S = 0.902$. It is worth to note that very large correlation matrix elements appeared in the refinement listing: the 24 largest had values in the range 0.959–0.908. Most of these strong correlations occur between atoms C7 and C2, N2 and N1, C10 and C5 or C9 and C4 (Fig. 1), suggesting these atoms correspond each other by a rotation around a twofold axis going through the Fe atom and roughly parallel to the cyclopentadienyl rings. Unfortunately, the determination failed to give accurate molecular informations. For instance, a sampling of bond lengths (Å) gives:

(a)	N1–C1	1.472(11)	N1–C01	1.490(13)
	N1–O1	1.533(13)	N1–O2	1.525(12)
(b)	N2–C6	1.396(12)	N2–C05	1.509(13)
	N2–O3	1.57(2)	N2–O4	1.539(11)

In view of the relative high number of parameters to handle and the possibility of having the *C*2 molecular symmetry, another structure determination was conducted with the centred space group *A*2/*a*. Only a few correlation matrix elements appeared in the refinement: five were listed in the range 0.642–0.541. Only half of the molecule (145 parameters) was refined using 2603 unique data (2500 $F_o > 4\ \sigma\ (F_o)$). Final R_1 (wR_2) values are 0.0357 (0.0733), with $S = 0.937$. The sample of bond lengths (Å), chosen as above, is giving:

(c)	N1–C1	1.440(2)	N1–C01	1.498(2)
	N1–O1	1.528(2)	N1–O1a	1.546(3)

The new bond lengths (c) are not mean values of the preceding lengths (a, b) but most of them generally lay inside each bond distance range (a–b). It is noteworthy that all standard deviations in line (c) are dramatically reduced compared with the unacceptable values reported on lines (a) et (b). In conclusion, the centrosymmetric space group *A2*/*a* leads to slightly larger R_1 and wR_2 values but produces more accurate bond and angle calculations which are retained for Table 2.

The X-ray analysis of **1** indicated an eclipsed structure for the N(OH)*t*-Bu substituants. The molecule exhibits a rigorous twofold axis going through the iron atom, parallel to the *b* axis and perpendicular to the N1, O1, N′, O′1 mean plane. A hydrogen bonding occurs between the hydroxyl group O1–H and the nitrogen atom N_1' of the opposite cyclopentadienyl ring (N′1···H: 1.859(4) Å). In addition, the hydroxyl group is disordered over two positions noted O1–H and O1a–H in the ratio 0.54: 0.46 (Fig. 2). However, the H atom remains in the same position in this disorder.

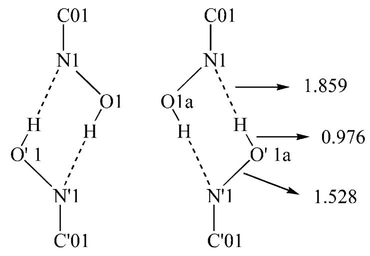


Fig. 2. Schematic view of the oxygen positions (in the ratio 0.54:0.46) in the structure of **1** (distances in Å).

The ^{1}H-NMR spectrum (vide supra) confirmed this structure in solution at room temperature in $CDCl_3$. A variable temperature ^{1}H-NMR study was also performed in order to try to break the hydrogen bonding. In C_6D_6 as solvent, we observed first, at room temperature, two different shapes of the two sets of signals corresponding to the Cp ring: a signal at low field (4.5 ppm) and a sharp signal at high field (3.85 ppm) (in $CDCl_3$, a singlet at 4.5 and a triplet at 4.0 ppm were observed). When heated in 10 °C steps from 20 to 70 °C, the peak sharpened and finally, at 75 °C, two triplets (1.9 Hz) are observed. This is the only difference observed in the variable temperature ^{1}H-NMR spectrum. In DMSO as solvent, the room temperature ^{1}H-NMR spectrum gave a sharp singlet at 9.1 ppm (OH), and two triplets (1.9 Hz) at 4.4 and 4.0 ppm. The compound was heated stepwise (20 °C) from 20 to 120 °C and finally 130 °C. At 100 °C we observed the enlargement of all the peaks of the OH, Cp and Me groups. At 120 °C the OH peak disappeared and was replaced by two signals at 8.5 and 8.8 ppm and many new broad signals appeared in the spectrum. When heated at 130 °C, all the peaks enlarged dramatically. The latter spectrum was also observed when the solution was cooled again to 60 °C and these data clearly indicate a decomposition reaction above 100 °C. At room temperature, the differences in the shapes of the signals observed in the spectra of **1** in $CDCl_3$, C_6D_6 and DMSO may be attributed to the solvation of the OH bonds.

The chemical oxidation was found to be more difficult than, for instance phenyl-substituted *ter*-butylhydroxylamines.[9] Indeed, no reaction was observed in the oxidation of **1** using PbO_2 in toluene as solvent. The oxidation occurs with freshly prepared Ag_2O in $CHCl_3$ and, after filtration the solution was studied using UV, ESR and IR spectroscopy, and compared with the spectra obtained with **1**, **2**, and **2**$^\bullet$. The ESR spectrum of the oxidised solution of **1** shows a 1:1:1 triplet but it corresponded only to traces of a N–O$^\bullet$ radical. The UV spectra of **1**, **2**$^\bullet$ and the oxidation solution of **1** are shown in Fig. 3 and are quite informative.

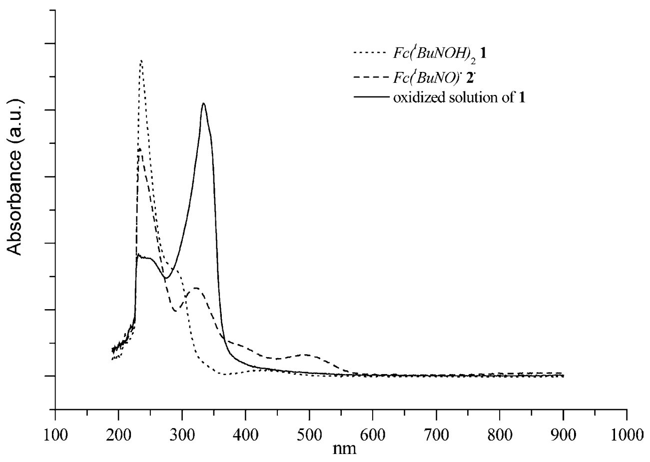


Fig. 3. UV spectra of **1**, **2** and oxidised solution of **1**.

Indeed, radical **2**$^\bullet$ gives the expected spectrum with a main absorbance at 233 nm due to the ferrocene unit (π–π*) and a broad absorbance at 320 nm (n–π*, N–O$^\bullet$) plus two signals of low intensity at 396 and 486 nm. On the contrary, if **1** has the main absorbance at 233 nm and a shoulder due to n–π* (N–OH) at 295 nm, the oxidated solution showed a broad absorbance at 231 nm and the major one at 333 nm; no absorption was observed at 400 and 490 nm. This spectrum is very different from the spectrum expected for the diradical **1**$^\bullet$. Finally, the IR spectrum of the solution showed the disappearance of the vibration due to the ferrocenyl C–H (3100 cm^{-1}) and the appearance of an intense band in carbonyl region at 1785 cm^{-1}.

Cyclic voltamograms of **1** shows that it is more easily oxidised than ferrocene ($\Delta = -100$ mV). If compared with 1,1′-diaminoferrocene which has a large cathodic shift due to the high degree of electron donation from the amino group ($E_{1/2} = -0.602$ V vs. $F_c^{0/+}$)[10] **1** is situated between ferrocene and 1,1′-diaminoferrocene.

All these data may be interpreted by the oxidation of the ferrocenyl part of **1**, followed by rapid decomposition which led to an intractable mixture.

In conclusion, 1,1′-bis(*N*-*ter*-butyl-*N*-hydroxyamino)ferrocene was synthesised and it eclipsed structure demonstrated by single crystal X-ray diffraction analysis and NMR experiments. The oxidation of **1** using Ag_2O led to the decomposition of the reaction mixture. As the structure of radical **2**$^\bullet$ was determined by X-ray analysis [7], and is stable, it may be conclude that the eclipsed structure of **1** favour the decomposition after oxidation.

3. Experimental

3.1. General

The solvents were dried as follows: THF, CaH_2 followed by Na–benzophenone; Et_2O, Na–benzophenone; toluene, P_2O_5; CH_2Cl_2, P_2O_5.

Argon ($O_2 < 5$ ppm; $H_2O < 5$ ppm) was used as the inert gas.

NMR: in solution, ^{1}H, and ^{13}C-NMR spectra were obtained in solution using a Bruker DPX-200 spectrometer. Chemical shifts, δ, in ppm, are referenced to TMS.

IR spectra were obtained on a Perkin–Elmer 1600 FT instrument working at 4 cm^{-1} resolution, values are given in cm^{-1}.

UV spectra were taken with a Secomam Anthélie Advanced instrument, using quartz cells and $CHCl_3$ or CH_2Cl_2 as a solvent. Concentrations were in the range of 10^{-5} mol l^{-1} and absorbances are given in nm.

Mass spectroscopy was performed on a JEOL JMS-DX300 spectrometer using electron impact or fast atom bombardment (FAB) modes. The matrixes used are *m*-nitrobenzylic alcohol (NBA) or thioglycerol (GT).

ESR spectra were recorded on a Bruker ELE-X-SYS instrument operating in the X-band (9,5 GHz). $CHCl_3$ was degassed before use. Concentrations were in the 10^{-5}–10^{-6} mol l^{-1} range.

The cyclic voltammetries were run in CH_3CN (0.01 M) using n-Bu_4PF_6 (0.1M) as supporting electrolyte, with a platinum wire electrode and a 300 mV s^{-1} scan speed.

Elemental analyses were performed by the Service Central de Microanalyse du CNRS, Vernaison, France.

3.2. Crystal structure determination

The intensity data for the compound was collected on a CAD-4 automated diffractometer, with graphite-monochromated Mo–K_α radiation.

The following compounds were synthesised according to published methods: 2-methyl-2-nitrosopropane [11], 1-(chloromercuri)ferrocene [12], 1-bromoferrocene [12], 1-lithioferrocene [13], 1,1′-dilithioferrocene [14].

3.3. 1-(*N*-*ter*-butyl-*N*-hydroxyamino)ferrocene

A 2.1 M solution of n-butyllithium (1.6 ml, 3.3 mmol) was added dropwise to a solution of 1-bromoferrocene (0.80 g, 3 mmol) in diethylether (70 ml) at −20 °C. After stirring at that temperature for 30 min, the mixture was allowed to reach 0 °C and a solution of 1-methyl-2-nitrosopropane (0.3 g, 3.5 mmol) in diethylether (20 ml) was added dropwise. After stirring at room temperature (r.t.) for 1 h and usual work-up, ferrocene was recovered by sublimation and 1-(*N*-*ter*-butyl-*N*-hydroxyamino)ferrocene (106 mg, 0.39 mmol, 13% yield) was obtained as an orange crystalline solid. M.p. 55–57 °C. ^{1}H-NMR ($CDCl_3$): $\delta = 1.09$ (s, 9 H, Me), 4.06 (t, $J = 1.8$ Hz, 2H, $H_{3,4}$ Cp), 4.28 (s, 5H, Cp), 4.3 (t, $J = 1.8$ Hz, $H_{2,5}$ Cp), 4.78 (broad s, 1 H, OH). IR (CCl_4) $\nu = 3591$, 3253 (OH) 3102 (CH, Fc), 1363 (N–OH, N–O).

3.4. 1-(*N*-*ter*-butyl-*N*-aminoxy)ferrocene

1-(*N*-*ter*-butyl-*N*-hydroxyamino)ferrocene was dissolved in dichloromethane and the solution was stirred under air for 30 min. The solvent was removed under vacuum to leave 1-(*N*-*ter*-butyl-*N*-aminoxy)ferrocene as a red–black solid. IR (CCl_4) $\nu = 3102$ (CH, Fc), 1363 (N–O). (litt. [7] 1320, N–O). ESR (CH_2Cl_2) $g = 2.0059$, $a_N = 12$G. We have not observed the hyperfine coupling as obtained by Forrester et al.[7] UV (CH_2Cl_2) 233 (π*–π*, Fc), 320 (π*–π*, N–O), 386 (n–π*, N–O), 486 (d–π*, Fc). MS (FAB^{+}, NBA), m/z (%) = 273 (80) [M^{+} + 1], 257 (75) [M^{+} −O].

3.5. 1,1′-di(*N*-*ter*-butyl-*N*-hydroxyamino)ferrocene

A solution of 2-methyl-2-nitrosopropane (2.6 g, 30 mmol) in diethylether (50 ml) was added dropwise to a solution of 1,1′-dilithioferrocene (2.8 g, 14 mmol) in diethylether (50 ml) cooled to −100 °C. After stirring at −30 °C for 16 h, the mixture was allowed to reach 0 °C and hydrolysed. After standard work-up the residue was dissolved in pentane and treated five times with charcoal to leave, after evaporation of the solvent, an orange crystalline solid (1.8 g, 5 mmol, 17% yield). Single crystal was obtained by slow evaporation of a diethylether solution. M.p. 142–144 °C (dec.). ^{1}H-NMR ($CDCl_3$) $\delta = 1.11$ (s, 18H, Me), 4.01 (t, $J = 1.65$ Hz, 4H, $H_{3,4}$ Cp), 4.5 (s, 4H, $H_{2,5}$, Cp), 9.57 (s, 2H, OH). ^{13}C-NMR ($CDCl_3$) $\delta = 26$ ($C(Me)_3$), 65 (CH_3), 117, 125, 131, 136 (Cp). IR (CCl_4) $\nu = 3219$ (OH), 3095 (Cp), 1361 (N–OH). UV (CH_2Cl_2) 236 (π*–π*, Fc), 290 (π*–π*, N–OH), 386 (n–π*, N–O), 427 (d–π*, Fc). MS (FAB^{+}, NBA), m/z (%) = 360 (100) [M^{+}]. Anal. Calc. For $C_{18}H_{28}FeN_2O_2$: C, 60.17; H, 7.80; Fe, 15.54; N, 7.80. Found: C, 60.08; H, 7.75; Fe, 15.33; N, 7.87%.

3.6. Oxidation of 1,1′-bis(*N*-*ter*-butyl-*N*-hydroxyamino)ferrocene

A solution of 1,1′-di(*N*-*ter*-butyl-*N*-hydroxyamino)ferrocene in $CHCl_3$ was treated with freshly prepared Ag_2O during 10 min to 10 h. After filtration the solution showed a 1:1:1 triplet in ESR but the concentration of the radical was low. See text for UV values.

4. Supporting information available

All tables concerning the determination with *Aa* along with the reminding tables for the determination with *A*2/*a* group are available at the Cambridge Crystallographic Data Centre: CCDC 173319 and 173320. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1233-

336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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