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1-Alkyl-2-(*Z*-1,2-diferrocenylvinyl)oxazolinium tetrafluoroborates: synthesis, characterization and nucleophilic ring opening

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Determination of the Crystal Structure

Data were obtained on an Oxford Difraction Gemini A diffractometer with a CCD area detector, and the CrystAlisPro and CrysAlis RED software packages were used for data collection and data integration [1]. The structures were solved using SHELXS-97[2] and refined by full-matrix least-squares on F^2 with SHELXL-97 [3,4]. Weighted Rfactors, R_w, and all goodness-of-fit indicators, S, were based F^2. The observed criterion of ($F^2 > 2\sigma F^2$) was used only for calculating the R factors. All non-hydrogen atoms were refined with anisotropic thermal parameters in the final cycles on refinement. Hydrogen atoms were placed in idealized positions, with C-H distances of 0.93and 0.98Å for aromatic and satured carbon atoms, respectively. The isotropic thermal parameters of the hydrogen atoms were assigned the values of U_{iso}=1.2 times the thermal parameters of the parent non-hydrogen atom. The unit cell parameters and the X-ray diffraction intensities were recorded on a Gemini (detector AtlasCCD, Cryojet N_2) diffractometer.

Crystallographic References

1.- CrysAlis, C. C. D. and CrysAlis, R; Oxford Difraction: Abingdon, U.K. 2009.

2.- G. M. Sheldrick. SHELXS-97, Crystal Structure Solution; University of Göttingen, Germany, 1990.

3.- G. M.Sheldrick. SHELXS-97, Crystal Structure Refinament; University of Göttingen, Germany, 1997.

The crystallographic data for **4a** and **5b** have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication under the CCDC numbers **1474953 and 1474954**, respectively These data can be obtained free of charge via www.ccdc.cam.ac.uk/const/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44)1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

Single crystal X-ray structure determination of 4a

Crystals of 4a were obtained by crystallization from dichloromethane

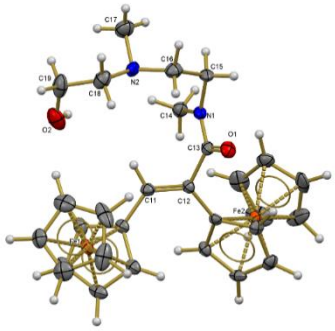
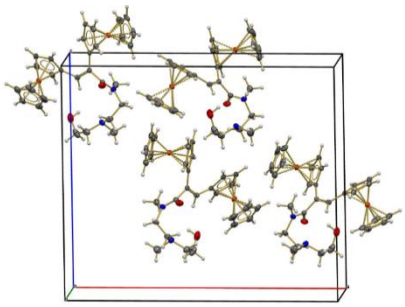


Fig. S-1: Crystal structure of 4a



Cristal Packing of 4a

Table S-1: Selected bond lengths and bond angles of compound 4a

Bond lengths (A)		Bond angles °	
C(11)-C(12)	1.346(5)	C(11)-C(12)-C(13)	116.1(3)
C(12)-C(13)	1.500(5)	O(1)-C(13)-N(1)	121.5(4)
C(13)-O(1)	1.246(5)	O(1)-C(13)-C(12)	118.8(4)
C(13)-N(1)	1.340(5)	N(1)-C(13)-C(12)	119.6(3)
C(14)-N(1)	1.460(5)	N(1)-C(15)-C(16)	110.5(3)
C(15)-N(1)	1.468(5)	N(2)-C(16)-C(15)	112.0(4)
C(16)-N(2)	1.462(5)	N(2)-C(18)-C(19)	114.4(4)
C(17)-N(2)	1.462(6)	O(2)-C(19)-C(18)	114.7(4)
C(18)-N(2)	1.461(6)	C(13)-N(1)-C(14)	124.3(3)
C(18)-C(19)	1.499(7)	C(14)-N(1)-C(15)	117.4(3)
C(19)-O(2)	1.409(6)	C(13)-N(1)-C(15)	117.6(3)
		C(18)-N(2)-C(16)	110.6(3)
		C(18)-N(2)-C(17)	110.6(3)
		C(16)-N(2)-C(17)	110.1(4)

Table S-2: Crystallographic data and structure refinement detail for compound 4a

Identification code	shelx	
Empirical formula	C29 H34 Fe2 N2 02	
Formula weight	554.28	
Temperature	130(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P -1	
Unit cell dimensions	a = 21.8387(11) Å	α= 90 °.
	b = 6.0311(3) Å	β= 90°.
	c = 19.0490(8) Å	ϒ = 90°.
Volume	4136.1(3) Å3	
Z	4	
Density (calculated)	1.467 Mg/m3	
Absorption coefficient	1.186 mm-1	
F(000)	1160	
Theta range for data collection	3.505 to 29.551°.	
Index ranges	-30<=h<=27, -8<=k<=8, -24<=l<=26	
Reflections collected	26278	
Independent reflections	6287 [R(int) = 0.0594]	
Completeness to theta	99.8 %	
Refinement method	Full-matrix least-squares on F2	
Goodness-of-fit on F2	6287 / 2 / 321	

Single crystal X-ray structure determination of 5b

Crystals of **5b** were obtained by crystallization from dichloromethane,

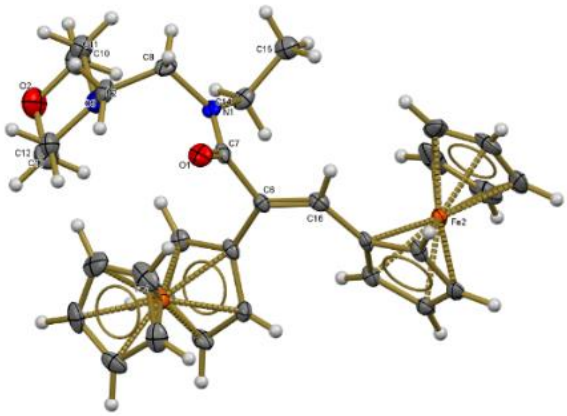
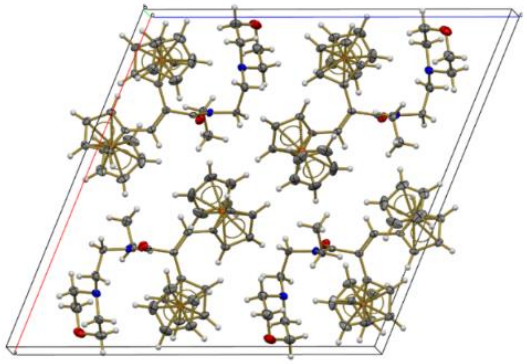


Fig. S-2: Crystal structure of 5b



Cristal Packing of 5b

Table S-3: Selected bond lengths and bond angles of compound 5b

Bond lengths (A)		Bond angles °	
C(6)-C(16)	1.339(4)	C(16)-C(6)-C(7)	118.3(2),
C(6)-C(7)	1.513(4)	O(1)-C(7)-N(1)	121.7(3)
C(7)-O(1)	1.229(3)	O(1)-C(7)-C(6)	120.9(3)
C(7)-N(1)	1.340(5)	N(1)-C(7)-C(6)	117.3(2)
C(14)-N(1)	1.348(4)	N(1)-C(8)-C(9)	111.1(2)
C(8)-N(1)	1.465(4)	N(2)-C(10)-C(11)	109.8(3)
C(10)-N(2)	1.460(4)	C(7)-N(1)-C(8)	118.0(2)
		C(14)-N(1)-C(7)	124.7(2)
		C(10)-N(2)-C(9)	112.3(2)

Table S-4: Crystallographic data and structure refinement detail for compound 5b

Identification code	shelx	
Empirical formula	C31 H36 Fe2 N2 O2	
Formula weight	580.32	
Temperature	130(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P -21/c	
Unit cell dimensions	a = 19.5186(16) Å	α= 90°.
	b = 7.3492(5) Å	β= 112.066 (9)°.
	c = 19.7752(18) Å	ϒ = 90°.
Volume	2628.9(4) $Å^3$	
Z	4	
Density (calculated)	1.466 Mg/m3	
Absorption coefficient	1.136 mm^{-1}	
F(000)	1216	
Theta range for data collection	3.466 to 29.579°.	
Index ranges	-24<=h<=26, -10<=k<=7, -24<=l<=23	
Reflections collected	15151	
Independent reflections	6245 [R(int) = 0.0498]	
Completeness to theta	99.7 %	
Refinement method	Full-matrix least-squares on F2	
Goodness-of-fit on F2	6245 / 0 / 335	

ADITIONAL SPECTROSCOPY DATA

Compound 3a: *2-(Z-1,2-Diferrocenylvinyl)-3-methyl-4,5-dihydrooxazol-3-ium tetrafluoroborate*

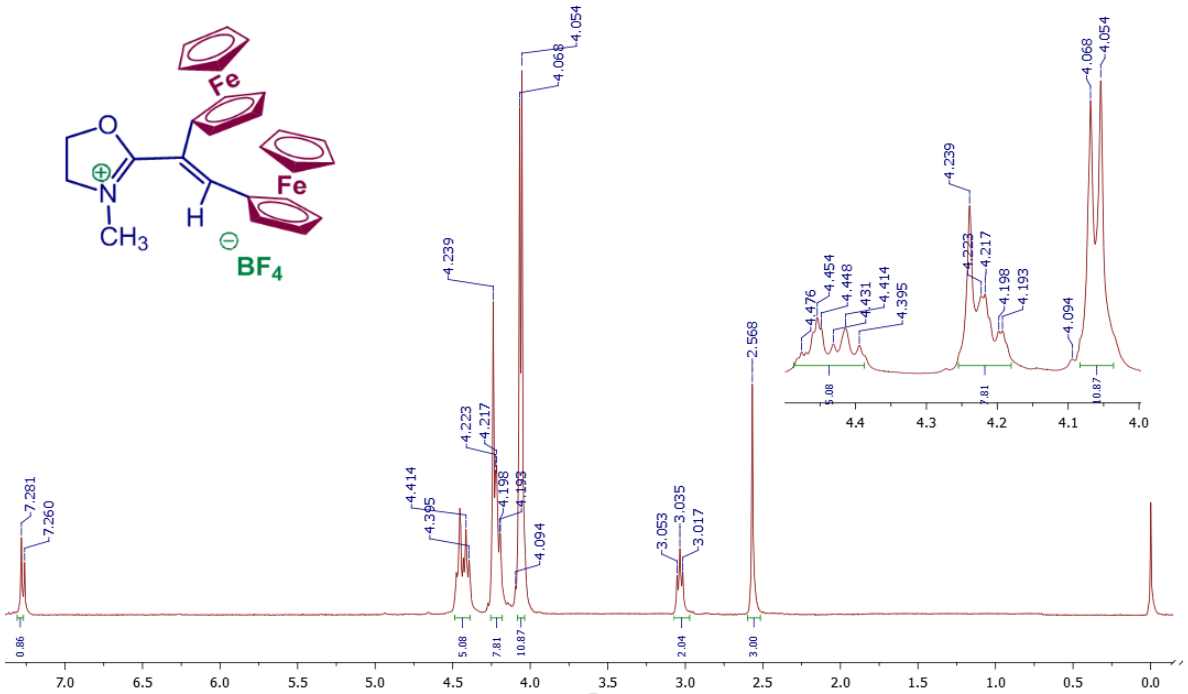


Fig. S-3: ^{1}H-NMR (300 MHz, $CDCl_3$, TMS) spectrum of compound **3a**

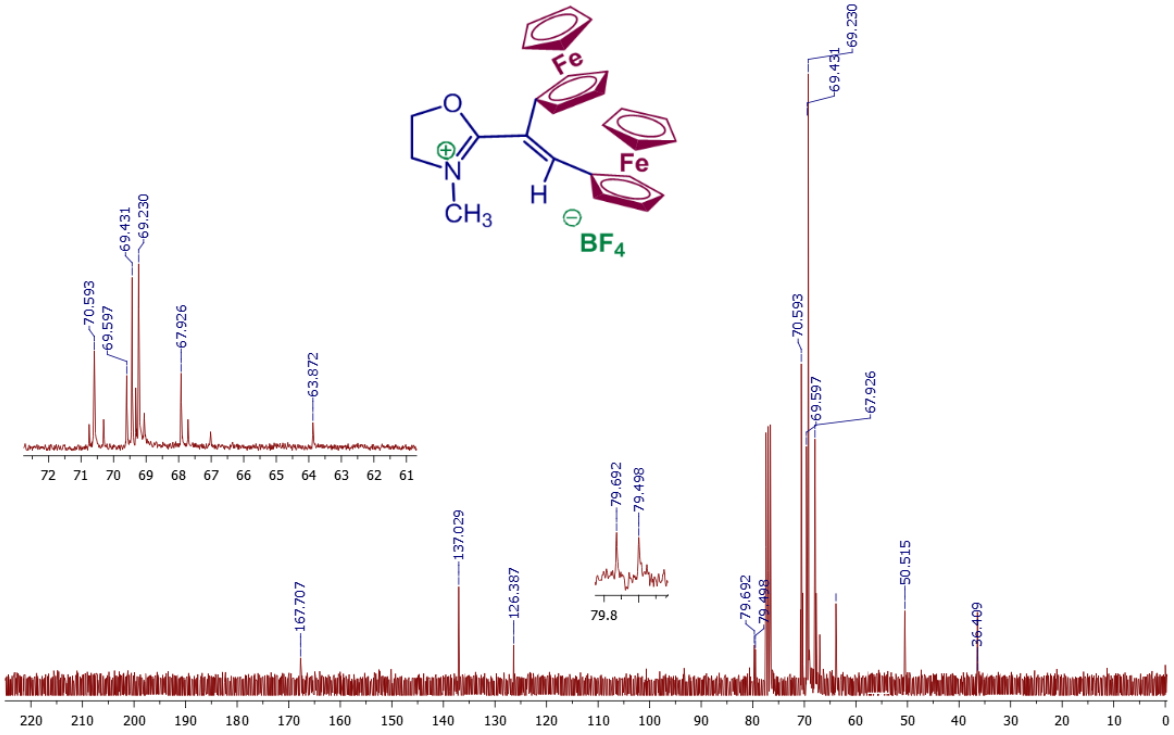


Fig. S-4: ^{13}C-NMR (75 MHz, $CDCl_3$, TMS) spectrum of compound **3a**

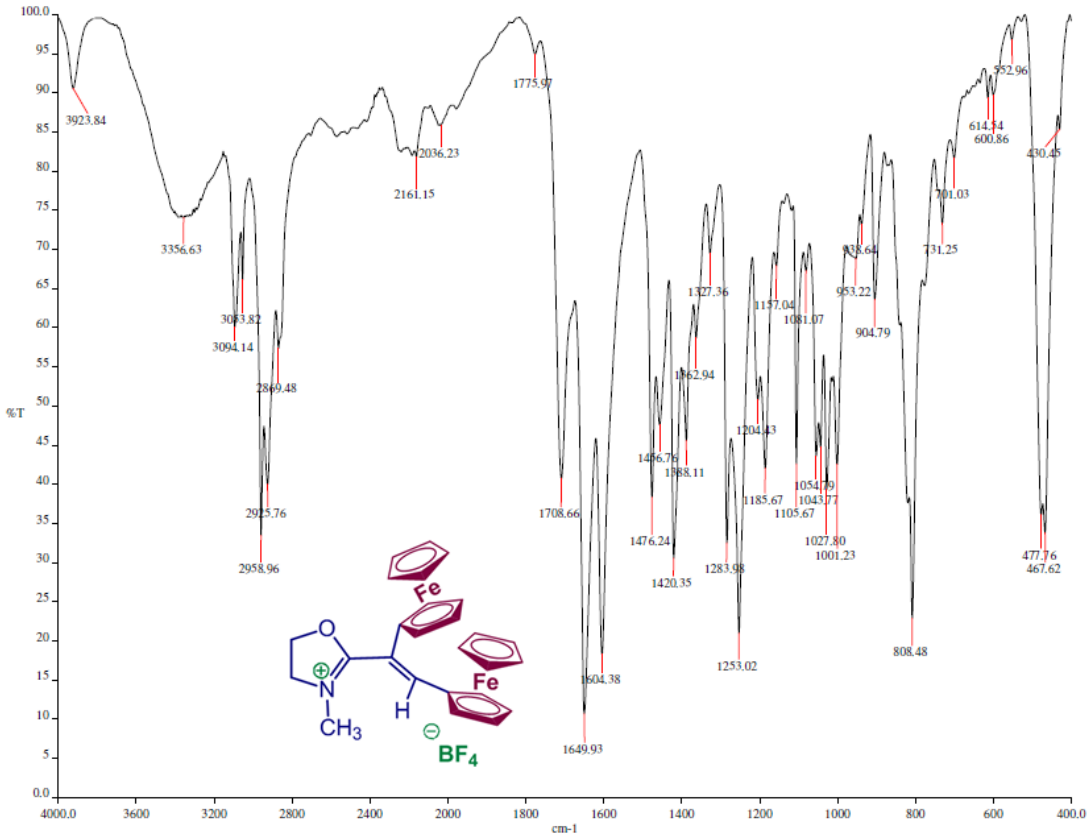
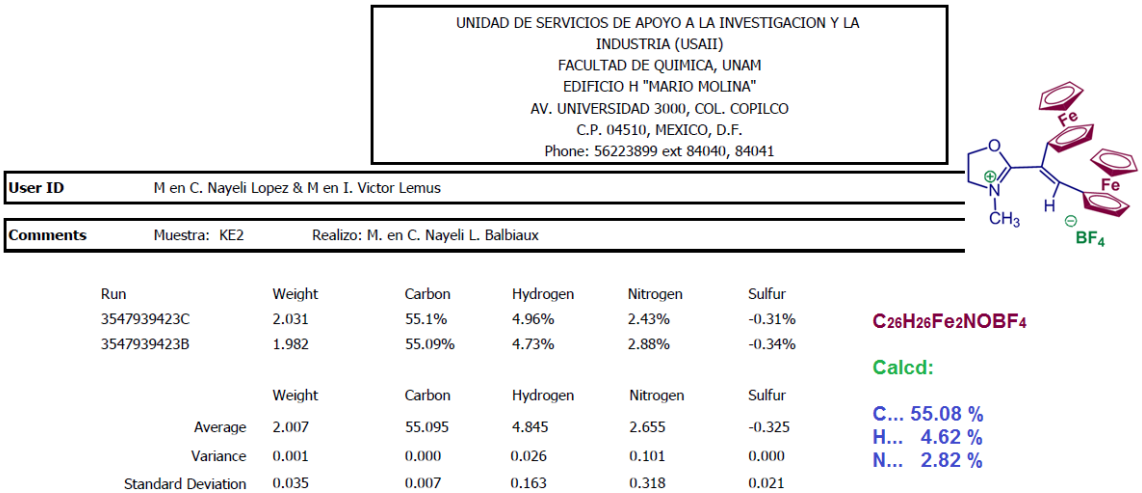


Fig. S-5: IR (KBr) spectrum of compound **3a**



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User ID M en C. Nayeli Lopez & M en I. Victor Lemus

Comments Muestra: KE2 Realizo: M. en C. Nayeli L. Balbiaux

Run	Weight	Carbon	Hydrogen	Nitrogen	Sulfur
3547939423C	2.031	55.1%	4.96%	2.43%	-0.31%
3547939423B	1.982	55.09%	4.73%	2.88%	-0.34%

	Weight	Carbon	Hydrogen	Nitrogen	Sulfur
Average	2.007	55.095	4.845	2.655	-0.325
Variance	0.001	0.000	0.026	0.101	0.000
Standard Deviation	0.035	0.007	0.163	0.318	0.021

$C_{26}H_{26}Fe_2NOBF_4$

Calcd:

C... 55.08 %
H... 4.62 %
N... 2.82 %

Fig. S-6: Elemental Analysis spectrum of compound **3a**

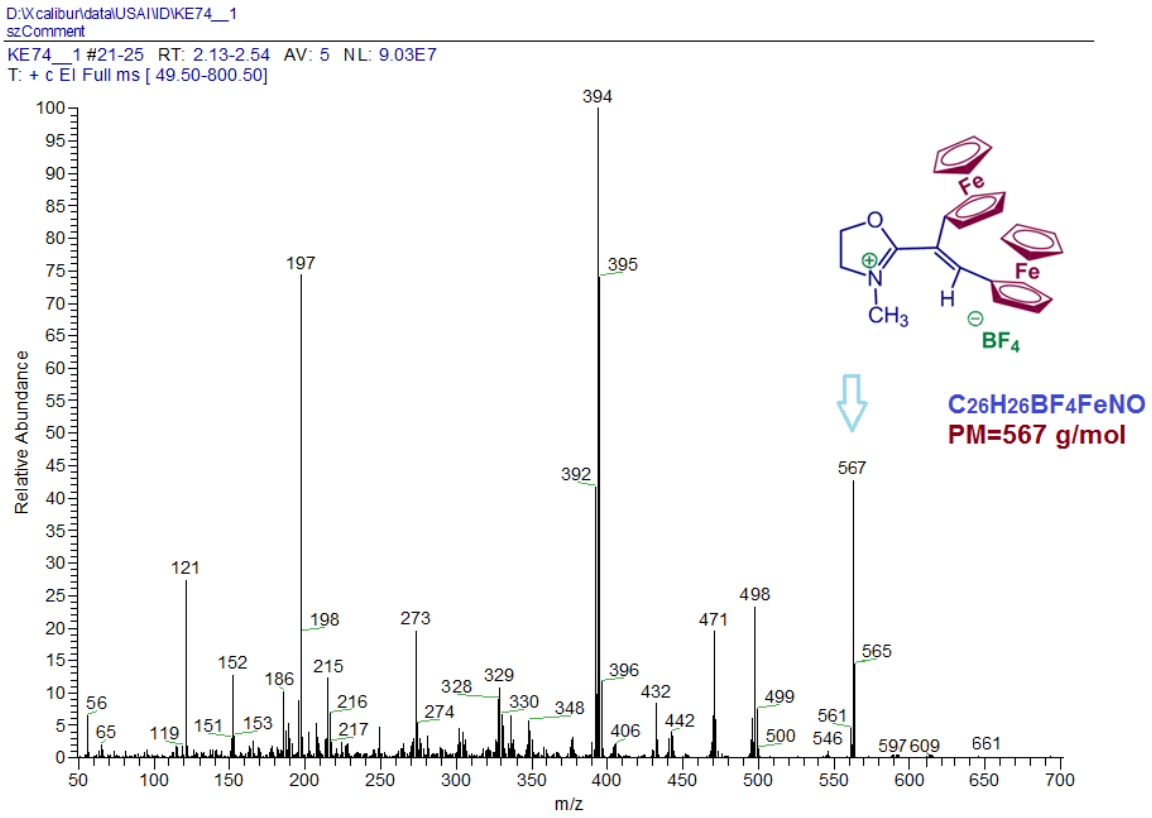

Fig. S-7: Mass spectrometry (EI, 70 Ev): m/z [M+] spectrum of compound **3a**

Compound 3b : 2-(Z-1,2-Diferrocenylvinyl)-3-ethyl-4,5-dihydrooxazol-3-ium tetrafluoroborate

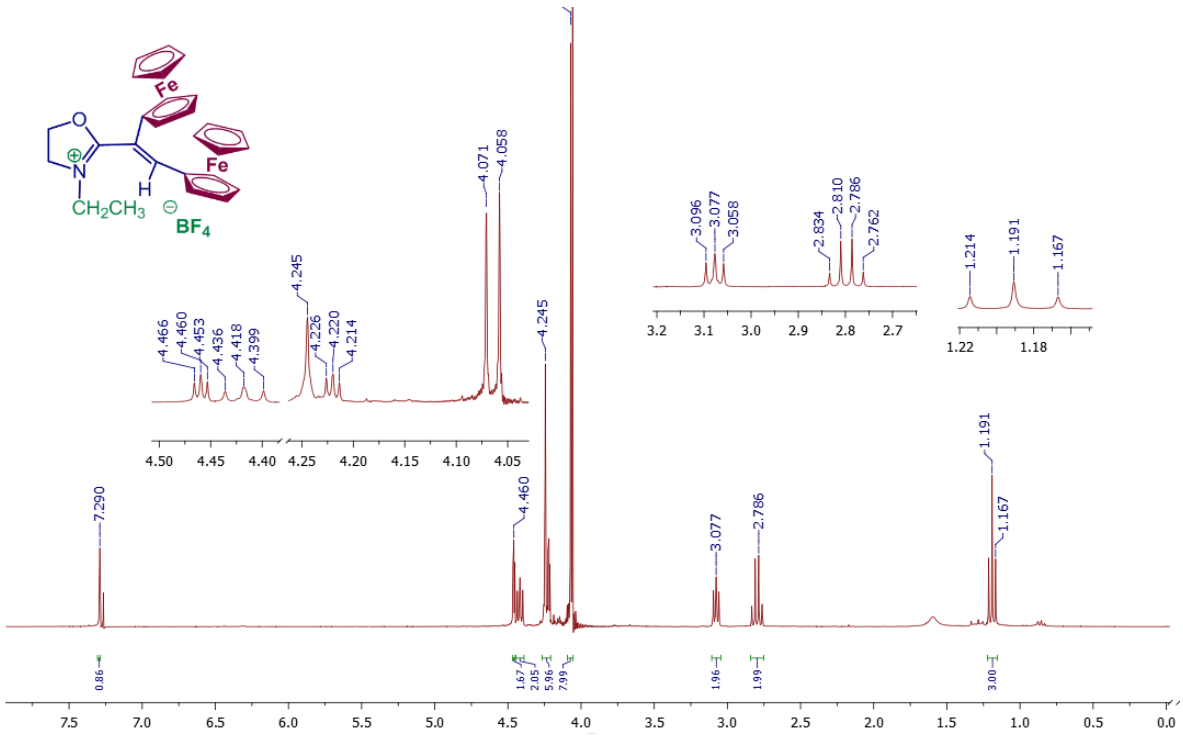

Fig. S-8: ^{1}H-NMR (300 MHz, $CDCl_3$, TMS) spectrum of compound **3b**

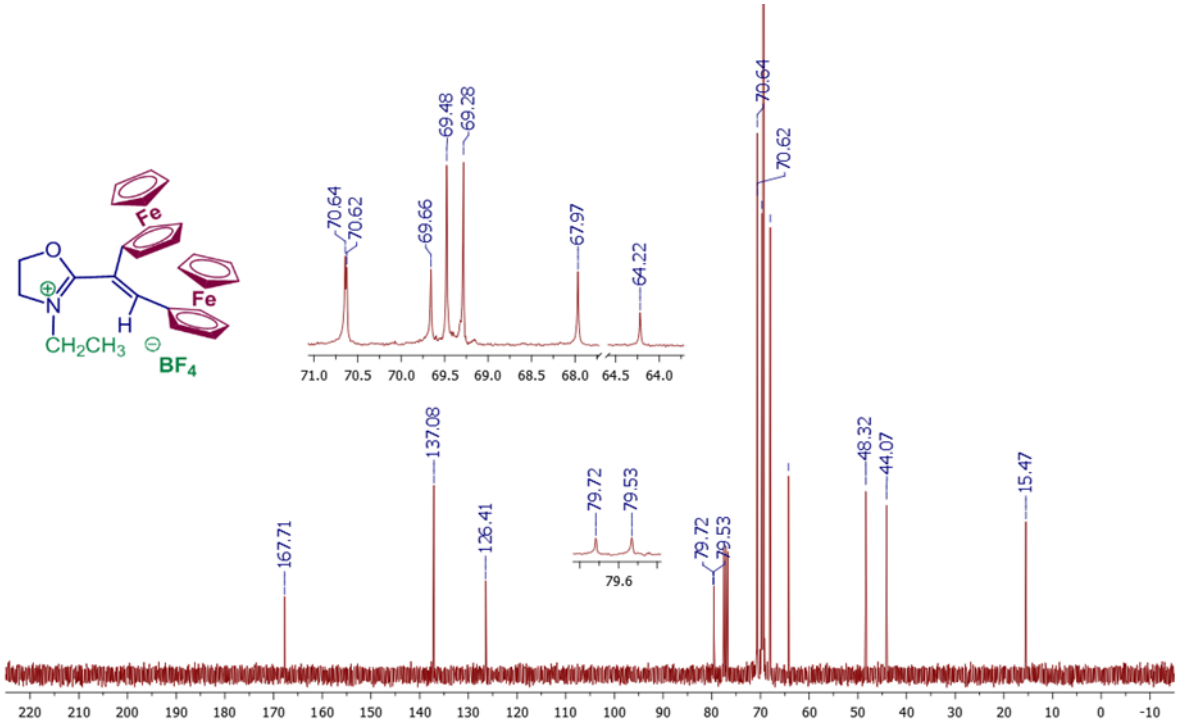

Fig. S-9: ^{13}C-NMR (75 MHz, $CDCl_3$, TMS) spectrum of compound **3b**

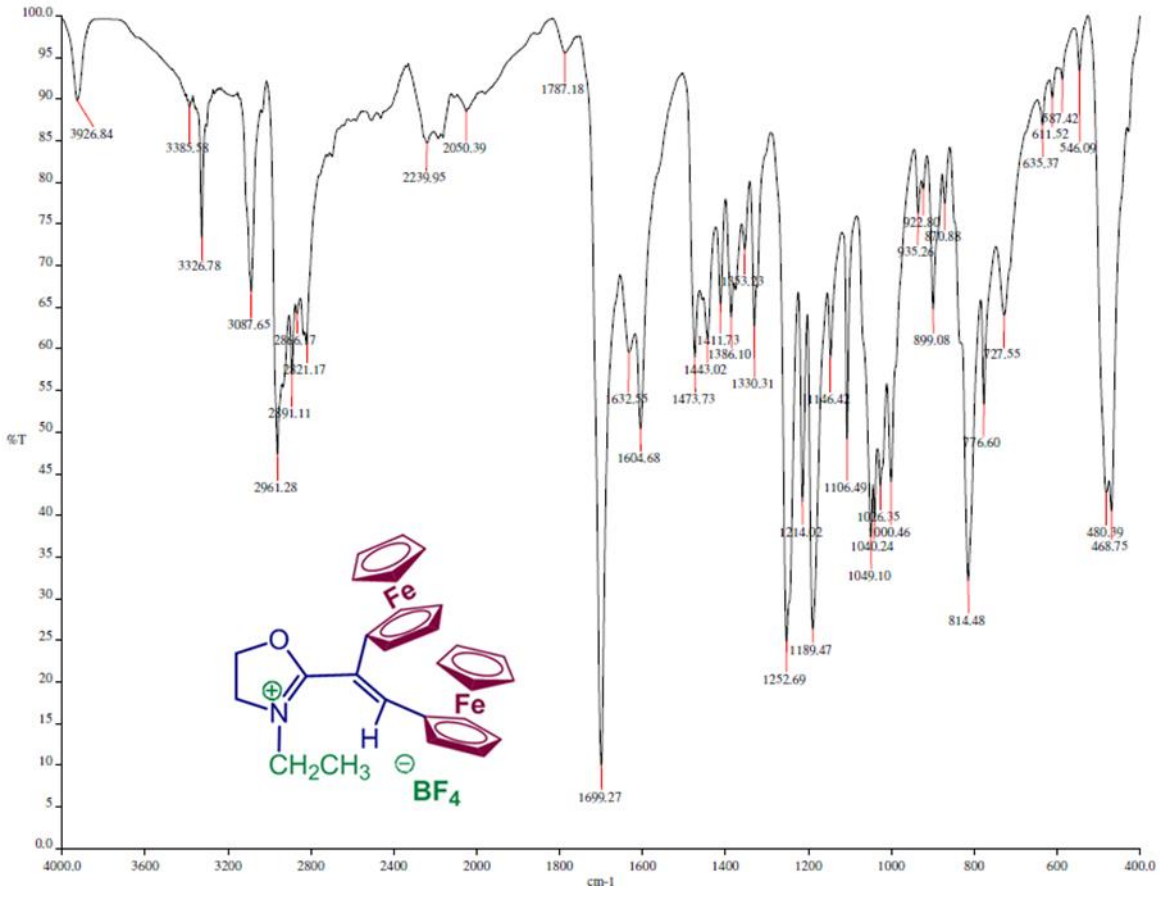

Fig. S-10: IR (KBr) spectrum of compound **3b**

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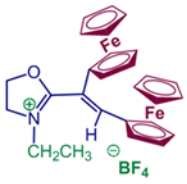

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Comments	Muestra: DR4Ae Realizo: Victor Lemus

Run	Weight	Carbon	Hydrogen	Nitrogen	Sulfur
3366835513B	3.067	56.86%	4.56%	2.12%	0.15%
3366835513A	2.983	56.9%	4.54%	2.11%	-0.1%

	Weight	Carbon	Hydrogen	Nitrogen	Sulfur
Average	3.025	56.880	4.550	2.115	0.025
Variance	0.004	0.001	0.000	0.000	0.031
Standard Deviation	0.059	0.028	0.014	0.007	0.177

$C_{27}H_{28}BF_4Fe_2NO$

Calcd:

C... 57.20 %
H... 4.98 %
N... 2.47 %

Fig. S-11: Elemental Analysis spectrum of compound **3b**

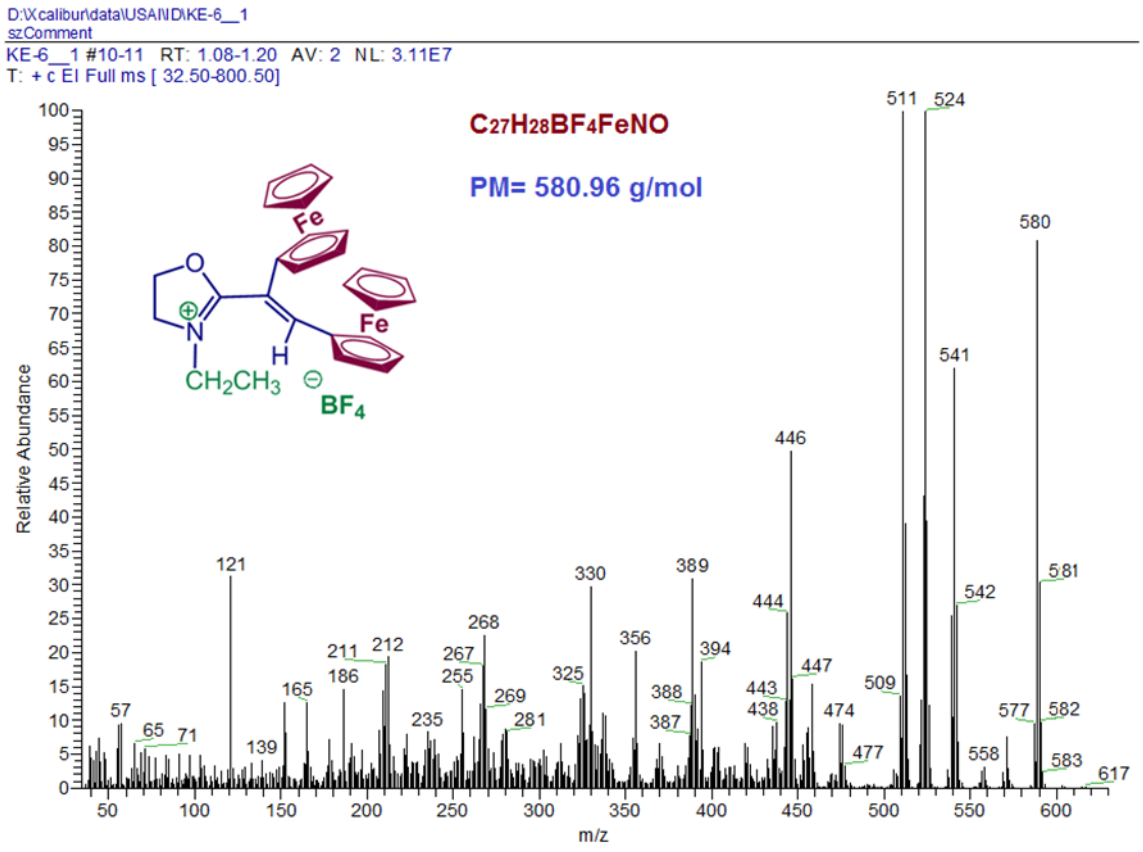

Fig. S-12: Mass spectrometry (EI, 70 Ev): m/z [M+] spectrum of compound **3b**

Compound 4a: 3,6-Diaza-(E-2,3-diferrocenyl)acryloyl-3,6-dimethylheptanol

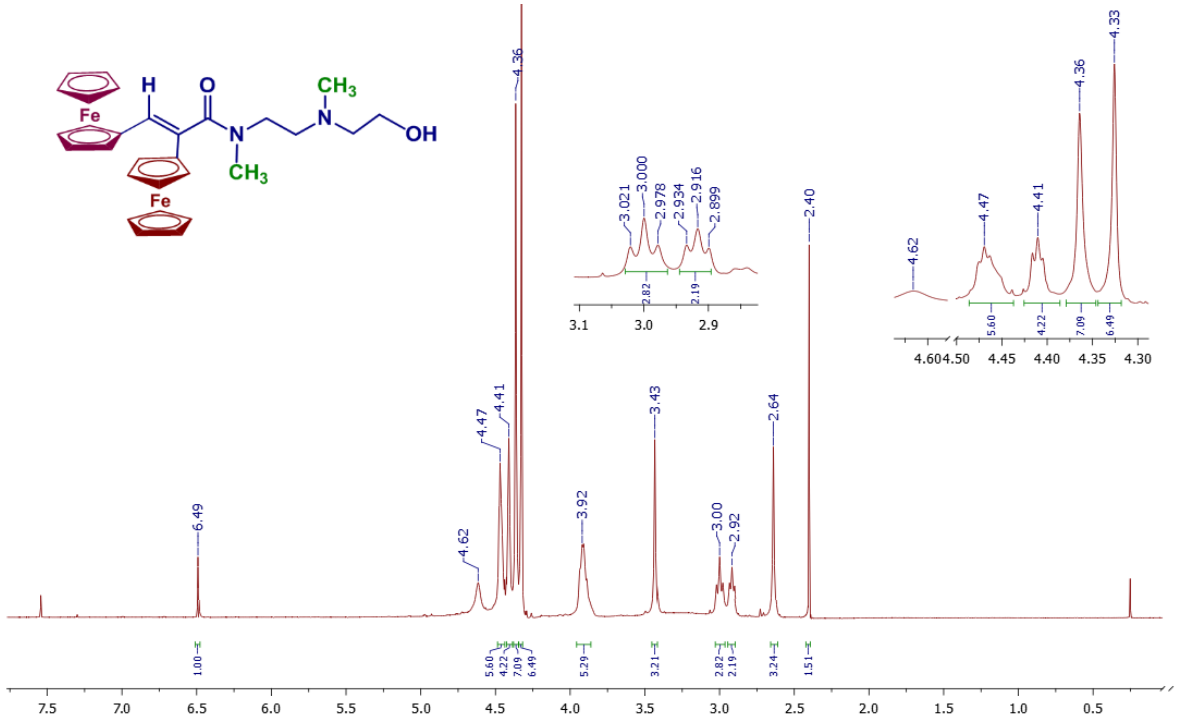

Fig. S-13: ^{1}H-NMR (300 MHz, $CDCl_3$, TMS) spectrum of compound **4a**

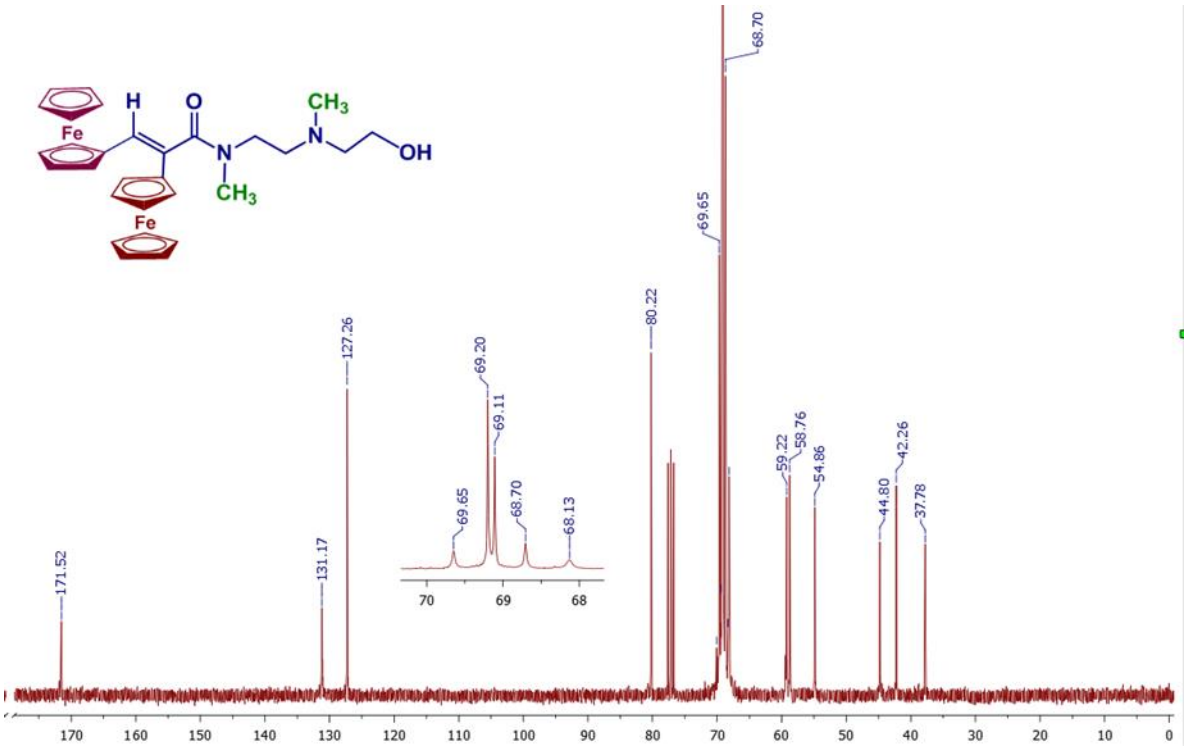

Fig. S-14: ^{13}C-NMR (75 MHz, $CDCl_3$, TMS) spectrum of compound **4a**

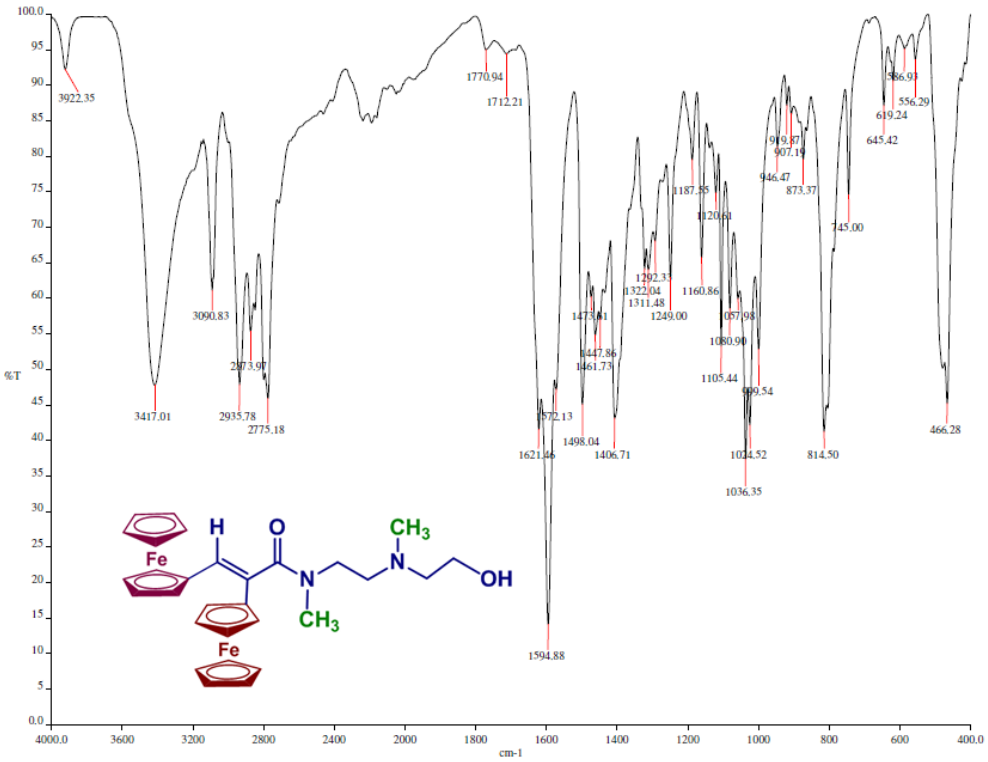

Fig. S-15: IR (KBr) spectrum of compound **4a**

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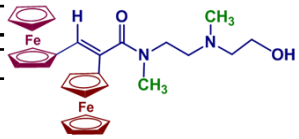
Comments	Muestra: ke67 Realizo: Victor Lemus



Run	Weight	Carbon	Hydrogen	Nitrogen	Sulfur
1701821368B	2.36	62.31%	6.33%	5.08%	0.16%
1701821368A	1.927	62.24%	6.03%	5.11%	-0.08%

	Weight	Carbon	Hydrogen	Nitrogen	Sulfur
Average	2.144	62.275	6.180	5.095	0.040
Variance	0.094	0.002	0.045	0.000	0.029
Standard Deviation	0.306	0.049	0.212	0.021	0.170

$C_{29}H_{34}FE_2N_2O_2$

Calcd:

C... 61.43 %
H... 6.05 %
N... 4.94 %

Fig. S-16: Elemental Analysis spectrum of compound 4a

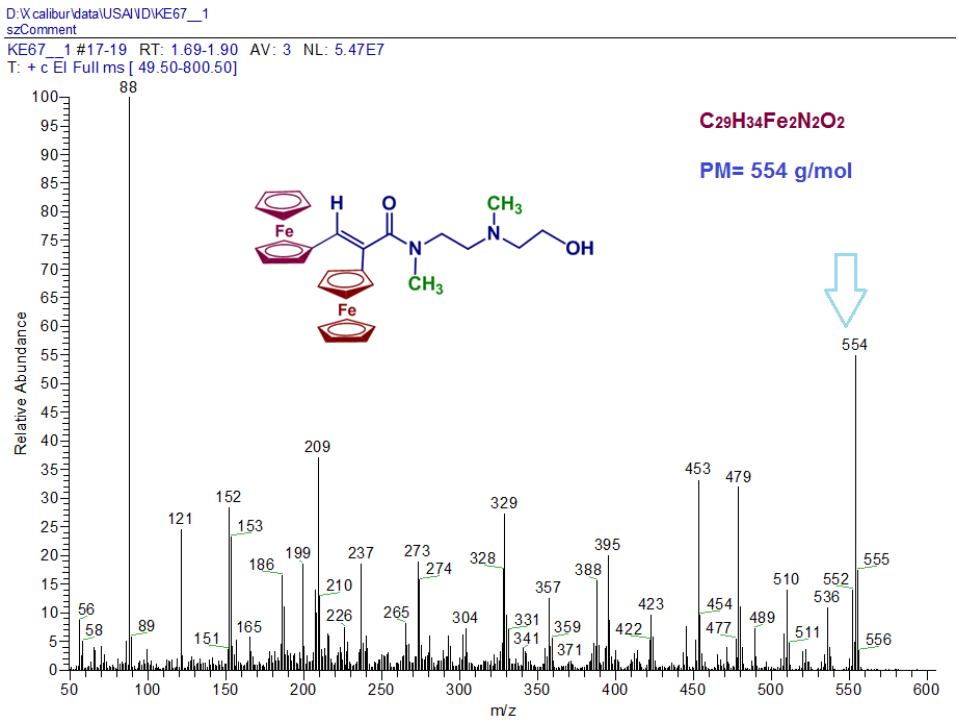


Fig. S-17: Mass spectrometry (EI, 70 Ev): m/z [M+] spectrum of compound 4a

Compound 4b : 3,6-Diaza-(E-2,3-diferrocenyl)acryloyl-3,6-diethylheptanol

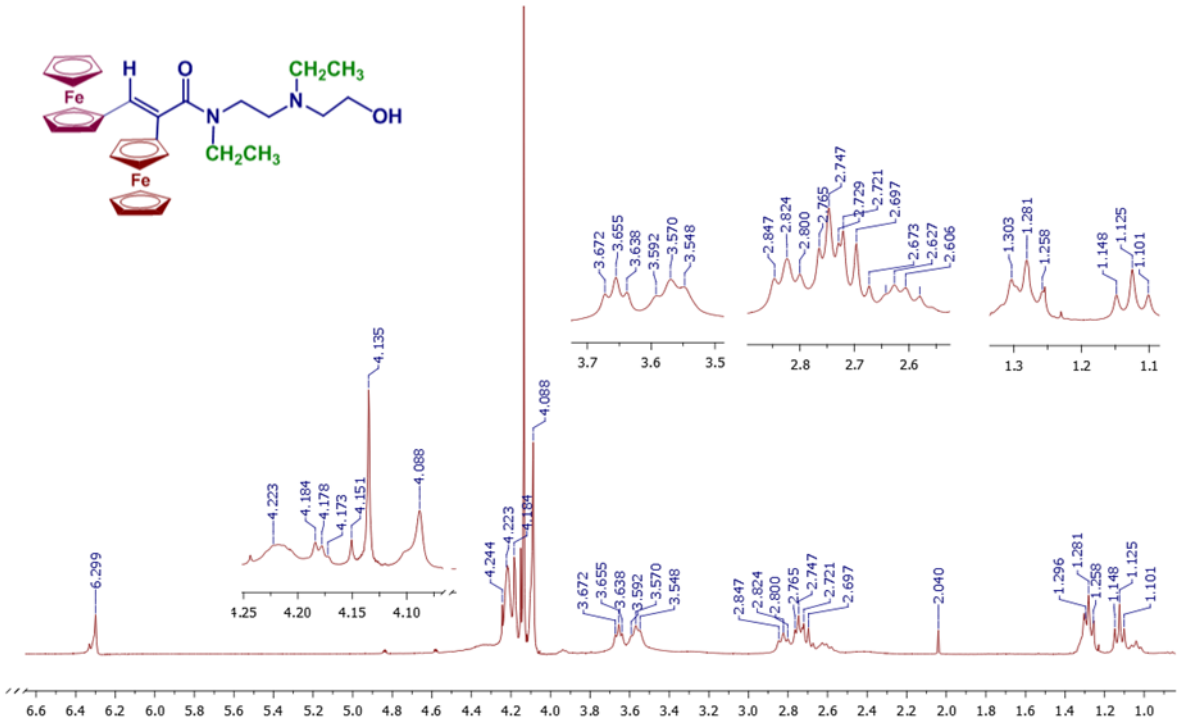


Fig. S-18: ^{1}H-NMR (300 MHz, $CDCl_3$, TMS) spectrum of compound **4b**

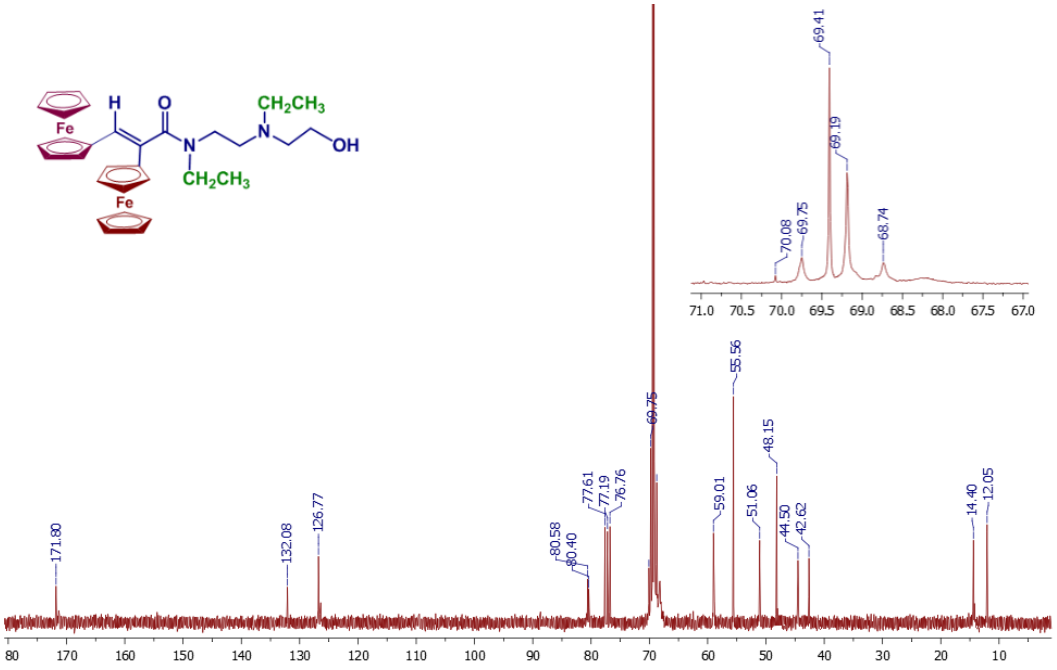


Fig. S-19: ^{13}C-NMR (75 MHz, $CDCl_3$, TMS) spectrum of compound **4b**

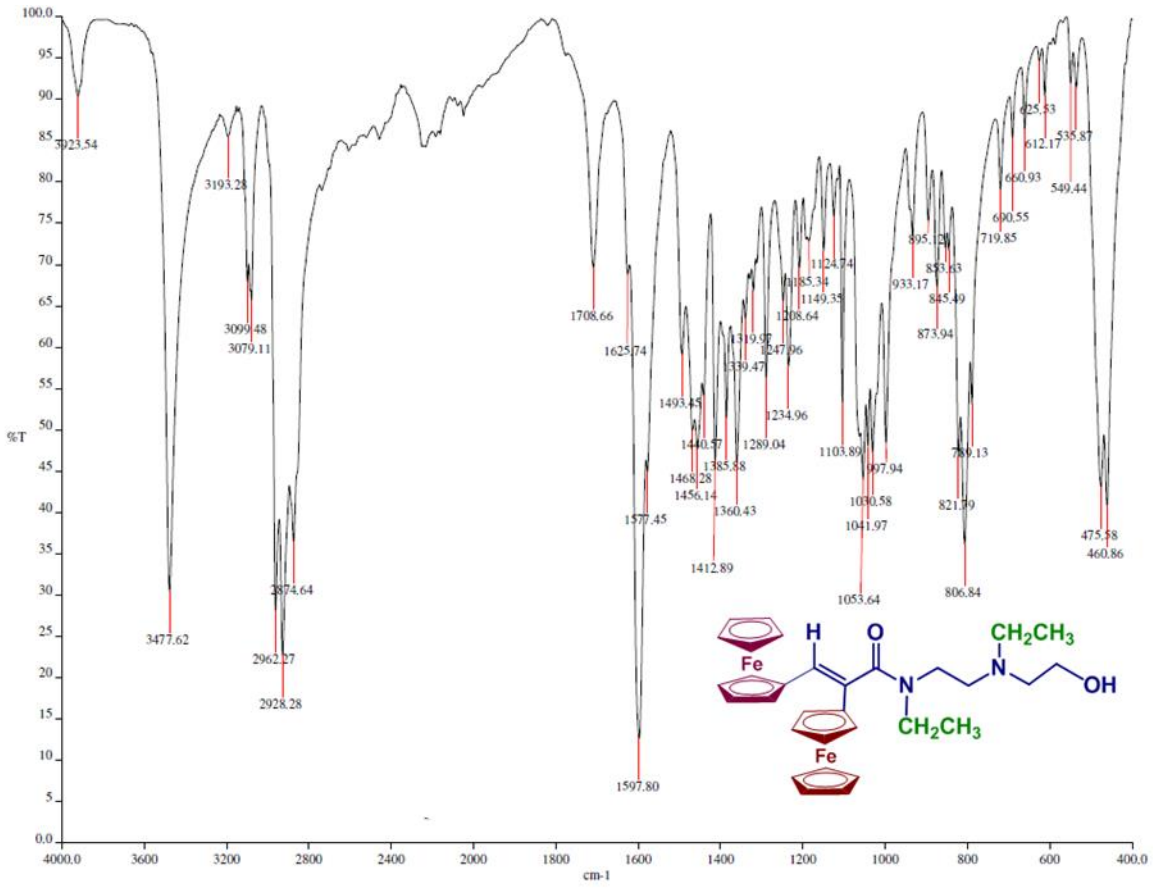


Fig. S-20: IR (KBr) spectrum of compound **4b**

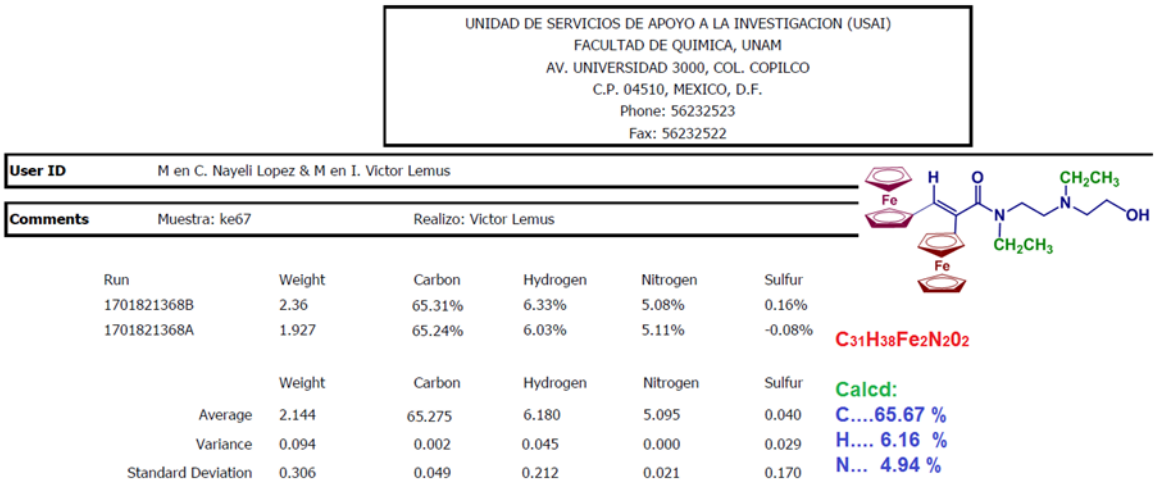


Fig. S-21: Elemental Analysis spectrum of compound **4b**

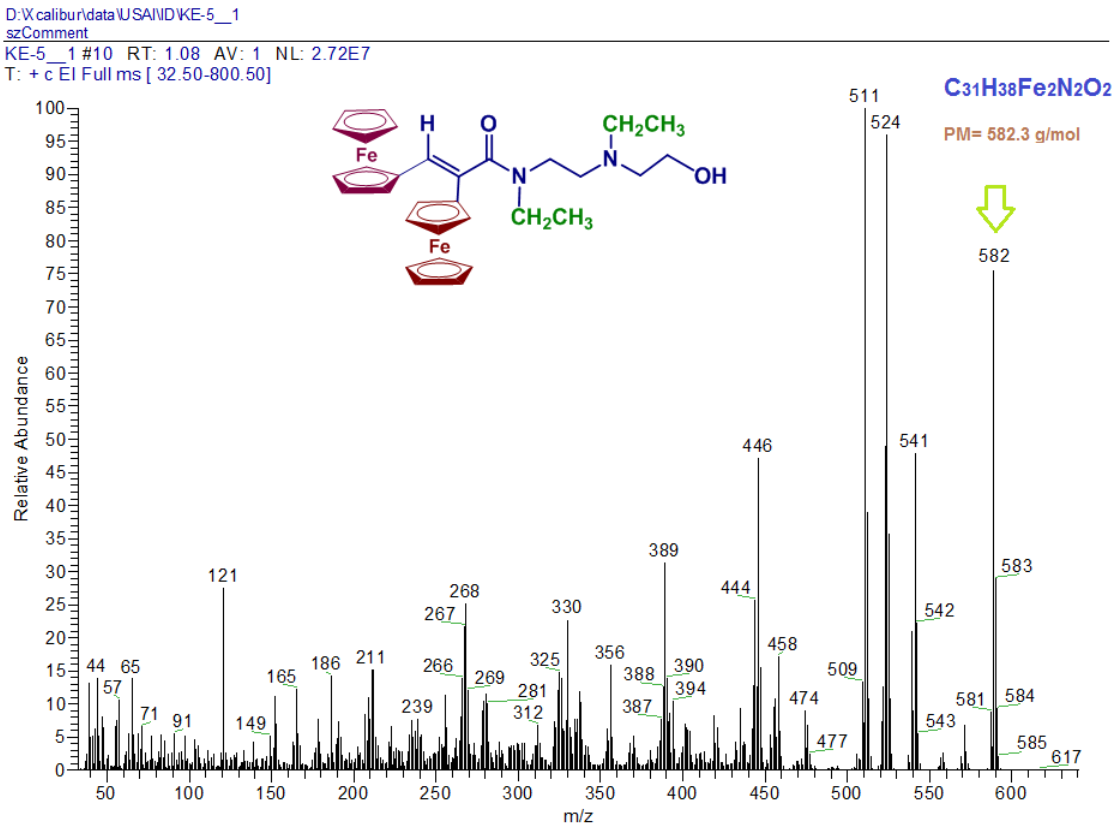


Fig. S-22: Mass spectrometry (EI, 70 Ev): m/z [M+] spectrum of compound **4b**

Compound 5a: (E) N-Methyl-N-(2-morpholinoethyl)-2,3-diferrocenyl-acrylamide

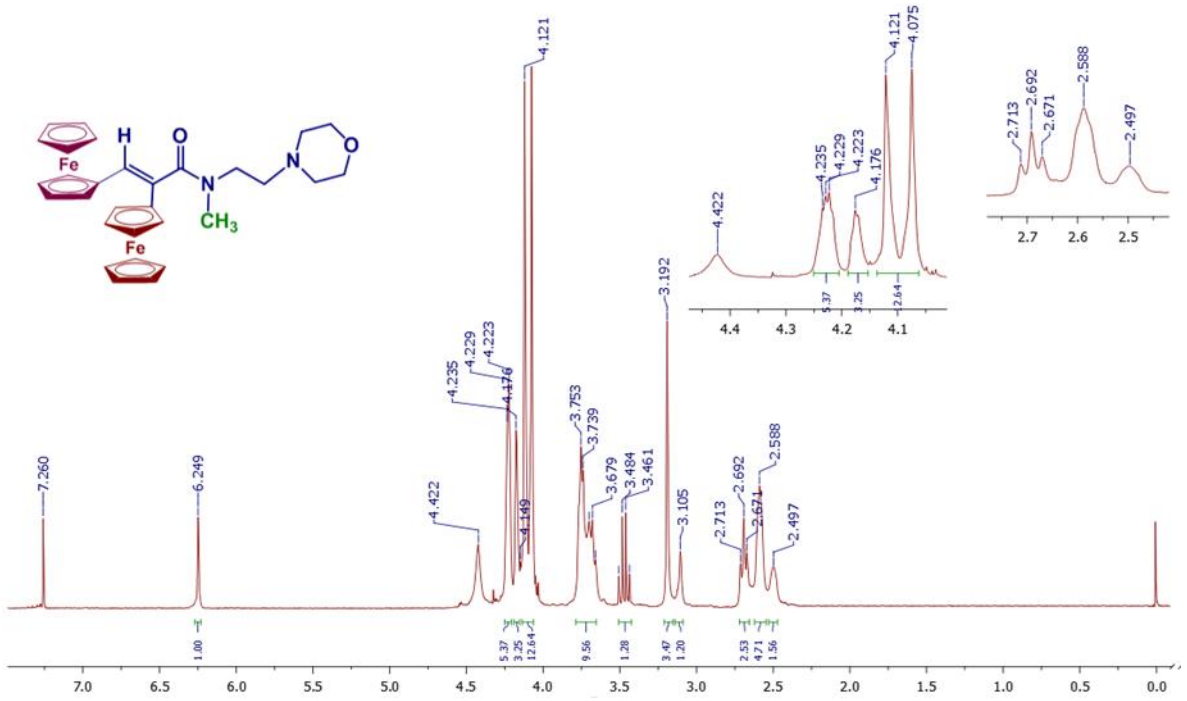


Fig. S-23: ^{1}H-NMR (300 MHz, $CDCl_3$, TMS) spectrum of compound **5a**

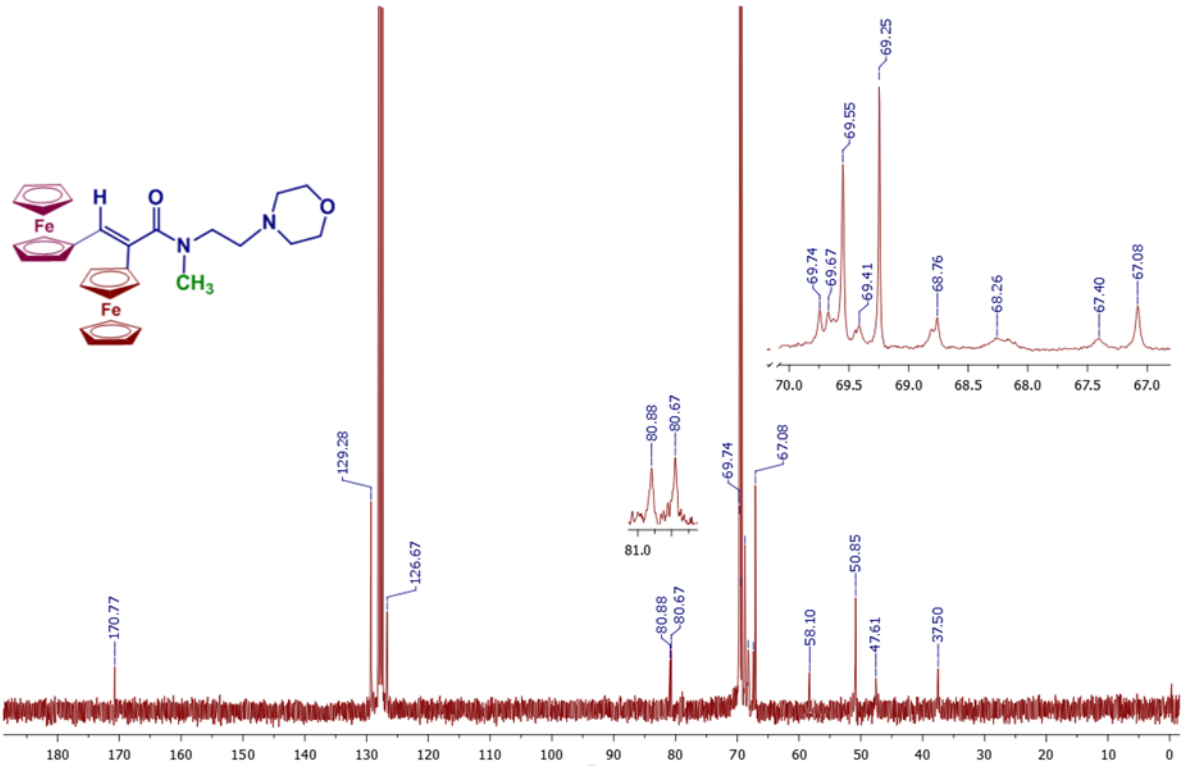


Fig. S-24: ^{13}C-NMR (75 MHz, C6D6, TMS) spectrum of compound **5a**

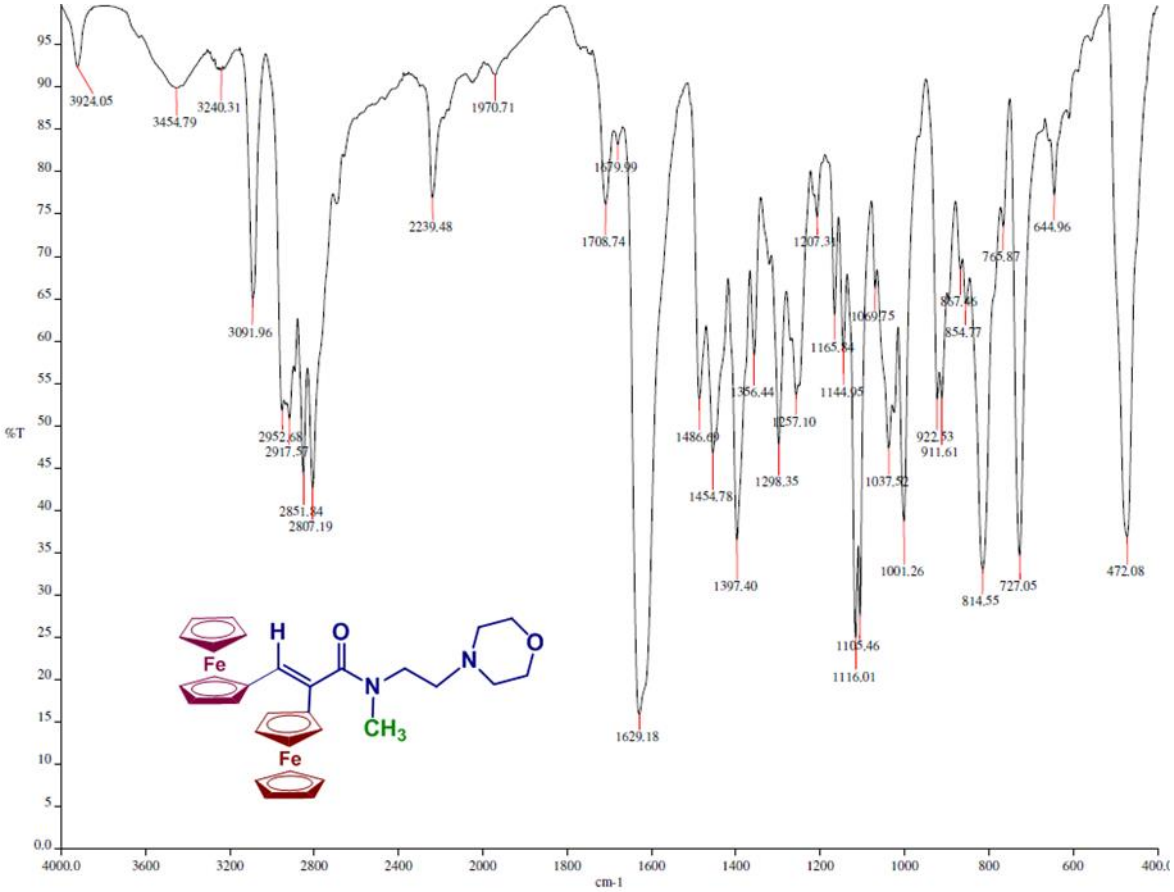


Fig. S-25: IR (KBr) spectrum of compound **5a**

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User ID	M en C. Nayeli Lopez & M en I. Victor Lemus				
Comments	Muestra: ke67	Realizo: Victor Lemus			

Run	Weight	Carbon	Hydrogen	Nitrogen	Sulfur
1701821368B	2.766	62.31%	6.13%	5.08%	0.000
1701821368A	1.927	62.24%	6.03%	5.11%	-0.08%

	Weight	Carbon	Hydrogen	Nitrogen	Sulfur
Average	2.346	62.275	6.080	5.095	0.040
Variance	0.094	0.002	0.045	0.000	0.029
Standard Deviation	0.306	0.049	0.212	0.021	0.170

$C_{30}H_{34}Fe_2N_2O_2$

Calcd:
C... 63.55 %
H... 6.05 %
N... 4.94 %

Fig. S-26: Elemental Analysis spectrum of compound **5a**

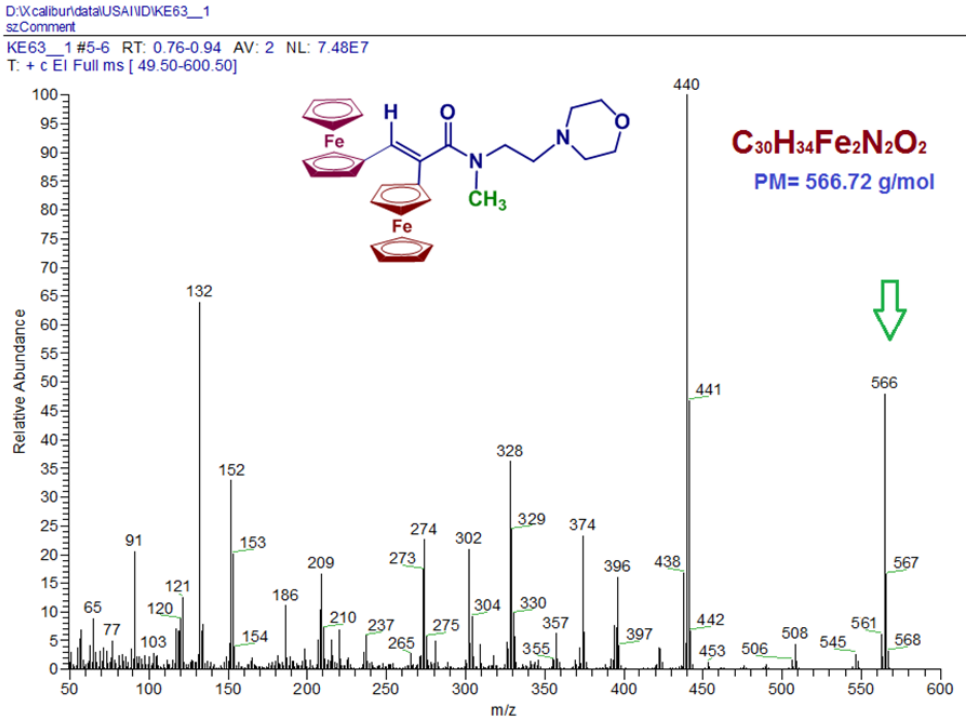

Fig. S-27: Mass spectrometry (EI, 70 Ev): m/z [M+] spectrum of compound **5a**

Compound 5b: (E) N-Ethyl-N-(2-morpholinoethyl)-2,3-diferrocenyl-acrylamide

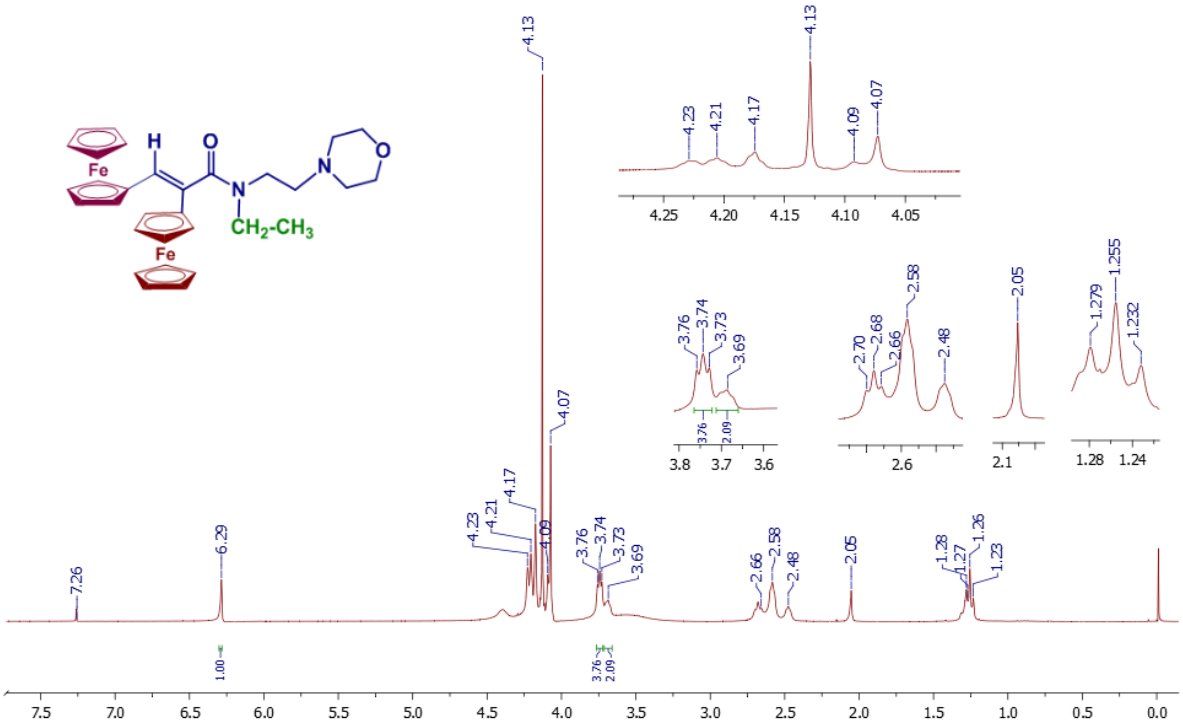

Fig. S-28: ^{1}H-NMR (300 MHz, $CDCl_3$, TMS) spectrum of compound **5b**

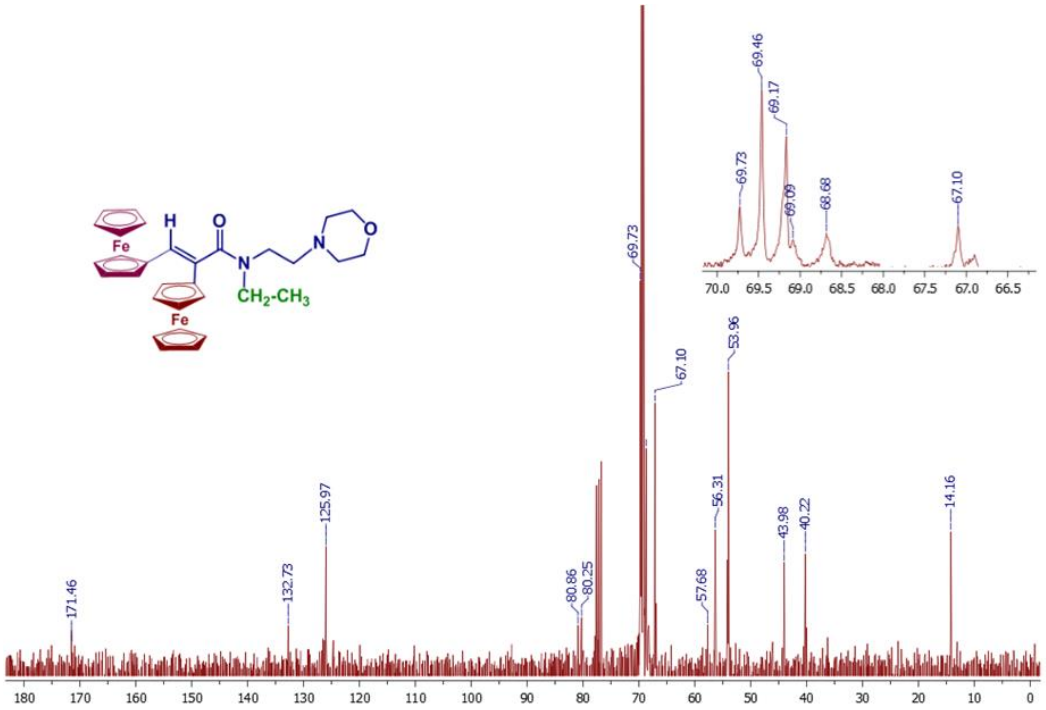

Fig. S-29: ^{13}C-NMR (75 MHz, $CDCl_3$, TMS) spectrum of compound **5b**

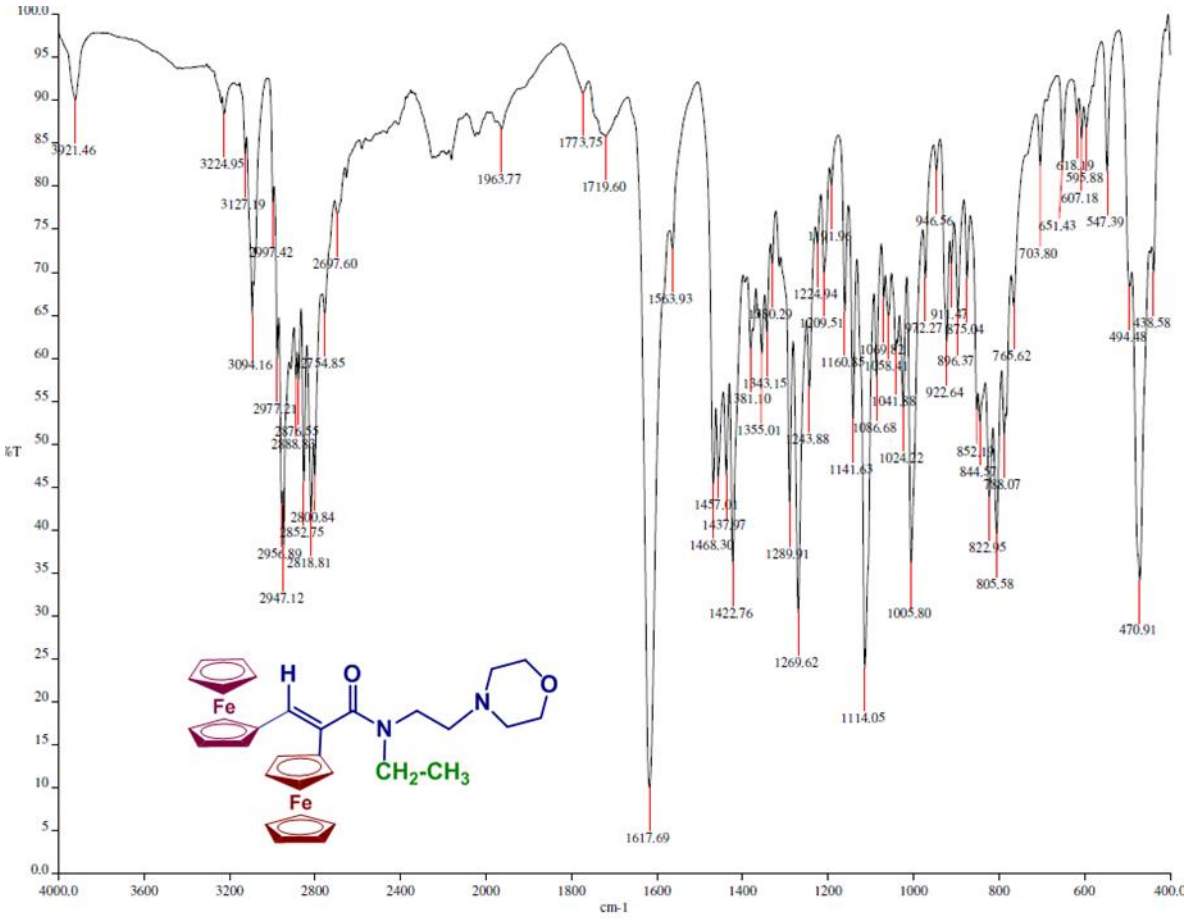

Fig. S-30: IR (KBr) spectrum of compound **5b**

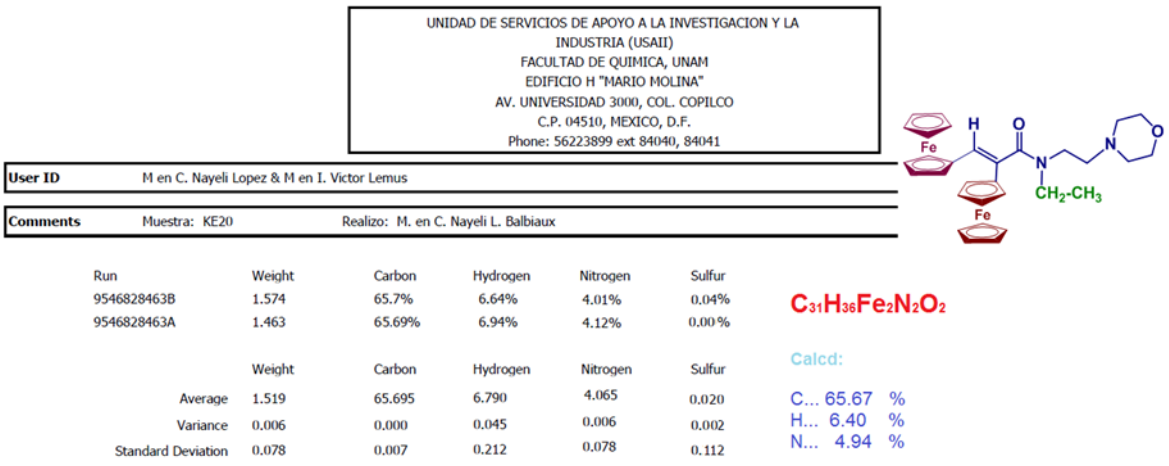

Fig. S-31: Elemental Analysis spectrum of compound **5b**

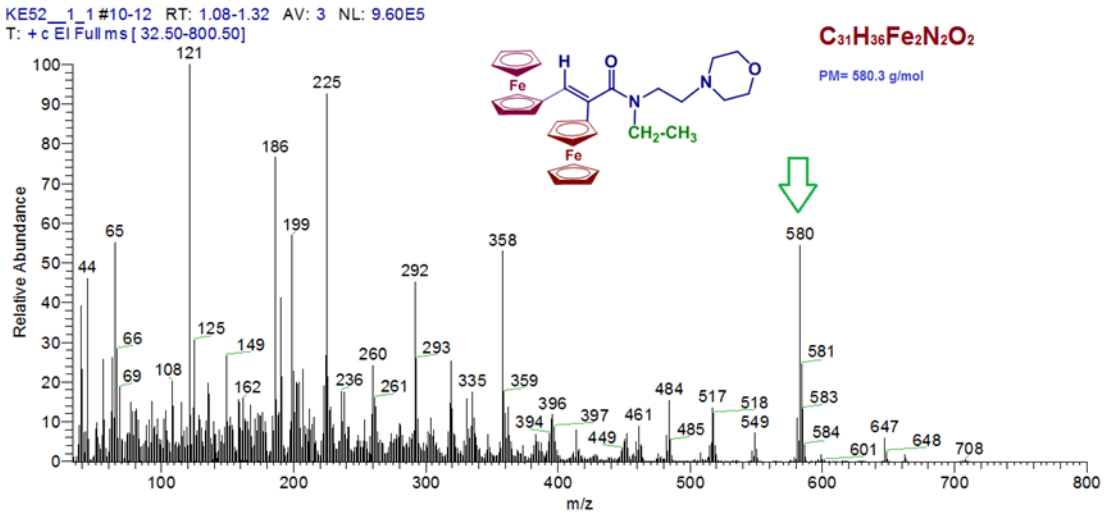

Fig. S-32: Mass spectrometry (EI, 70 Ev): m/z [M+] spectrum of compound **5b**