

6 Síntesi i caracterització de productes que contenen la unitat oxazolidin-2-ona

▪ N,N-bis(5-(N-oxazolidin-2-ona)-3-tiapentil)anilina (8)

En un baló de tres boques de 1000 ml realitzem una suspensió de Cs_2CO_3 (5.38 g, 16.5 mmol) en 600 ml de DMF seca i desoxigenem mitjançant bombolleig de nitrogen. Preparem una dissolució de N,N-bis(2-mercaptoetil)anilina (7) (2.37 g, 11 mmol) i una solució de bis(2-cloroetil)amina clorhidrat (1) (1.98 g, 11 mmol) en 50 ml de DMF seca i desoxigenada, respectivament. Sobre la suspensió de Cs_2CO_3 en agitació intensa s'addicionen a temperatura ambient les dues solucions de manera simultània a una velocitat de 2 ml/h mitjançant un perfussor. Un cop finalitzada l'addició mantenim la solució resultant en agitació durant 24 h. Filtrem i evaporem el dissolvent, restant un oli groguenc molt dens. Realitzem extraccions amb èter etílic en primer lloc (2 x 200 ml) i finalment amb clorur de metilè (2 x 200 ml). Evaporem el clorur de metilè i obtenim un sòlid que purifiquem dissolent-lo en la mínima quantitat de clorur de metilè i fent-lo precipitar en forma de sòlid blanc microcristal·lí afegint 2-3 ml de ciclohexà i èter de petroli (1.23 g, 51%). Anàlisi trobat (calculat per $\text{C}_{20}\text{H}_{29}\text{N}_3\text{O}_4\text{S}_2$): C, 53.16 (54.64); H, 6.29(6.65); N, 9.27(9.56); S, 14.25 (14.56). P.m.: 439.60 g/mol. Ió $[\text{M}]^+$ trobat per espectroscòpia de masses d'impacte electrònic.

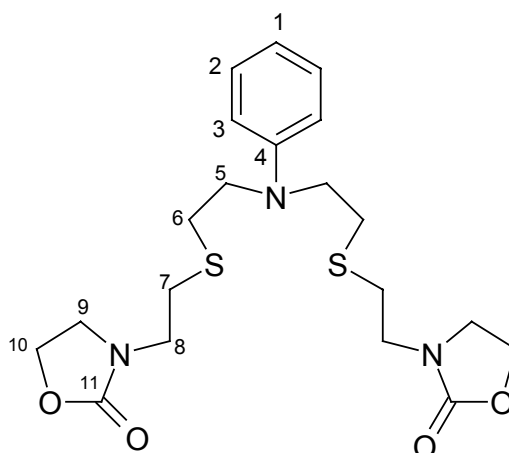
IR (KBr)

ν (cm^{-1})	3081(pI), 3025(pI), 2957(Im), 2918(Im), 2280(Im), 1900(pI), 1733(mI), 1595(I), 1500(I), 1261(I), 742(I), 685(Im)
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RMN (CDCl_3)

^1H δ (ppm)	Multiplicitat	Integral	Assignació
7.22	t	2H	H ₂
6.70	t	3H	H ₁
6.68	d		H ₃
4.31	pt	4H	H ₁₀
3.59	pt	4H	H ₉
3.56	pt	4H	H ₅
3.45	pt	4H	H ₈
2.76	pt	8H	H ₆ , H ₇

^{13}C δ (ppm)	Assignació	^{13}C δ (ppm)	Assignació
158.32	C ₁₁	51.33	C ₅
146.53	C ₄	44.76	C ₉
129.45	C ₂	43.56	C ₈
116.78	C ₁	29.73	C ₆ , C ₇
111.99	C ₃	28.90	C ₆ , C ₇
61.75	C ₁₀		



▪ **3-(2-cloroetil)-oxazolidin-2-ona (9)**

Refluïm durant 5 h una suspensió del bis(2-cloroetil)amina clorhidrat (**1**) (3.10 g, 17 mmol) i Cs_2CO_3 (6.42 g, 18 mmol) en 100 ml de DMF. Evaporem el dissolvent i obtenim un oli de color ocre molt dens. Realitzem diverses extraccions amb èter etílic (3 x 100 ml) i finalment, després d'evaporar el dissolvent, obtenim un oli groc que cristal·litza en forma de petites agulles (1.32 g, 51%). Anàlisi trobat (calculat per $\text{C}_5\text{H}_8\text{NO}_2\text{Cl}$): C, 40.15 (40.12); H, 5.68(5.39); N, 9.63 (9.36). P.m.: 149.58 g/mol. Ió $[\text{M}]^+$ trobat per espectroscòpia de masses d'impacte electrònic.

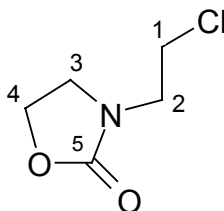
IR (KBr)

ν (cm^{-1})	2967(lm), 2918(lm), 1751(ml), 1272(ml), 1515(l), 1042(l), 763(lm)
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RMN (CDCl_3)

^1H δ (ppm)	Multiplicitat	Integral	Assignació
4.37	pt	2H	H_4
3.7	m	6H	$\text{H}_1, \text{H}_2, \text{H}_3$

^{13}C δ (ppm)	Assignació
158.12	C_5
61.77	C_4
45.71	C_3
45.03	C_2
41.41	C_1



▪ **6-oxa-1,11-bis(N-oxazolidin-2-ona)-2,10-ditiaundecà (10)**

En un baló de 250 ml preparam una dissolució de 2-mercaptoetil (0.50 g, 3.6 mmol) en 100 ml de metanol desoxigenat, afegim NaOH (0.288 g, 7.2 mmol) i escalfem lleugerament. Addicionem, gota a gota, sobre aquesta dissolució de ditiolat una dissolució de 3-(2-cloroetil)-oxazolidin-2-ona (1.076 g, 7.2 mmol) en 50 ml de metanol desoxigenat. Agitem durant 24 h, evaporem el dissolvent i obtenim un oli ocre força fosc i que dissolem en clorur de metilè per tal de precipitar el NaCl format. Realitzem una columna en sílice utilitzant com eluent una solució de cloroform-metanol (10:1), descartem les primeres fraccions eluïdes i finalment obtenim el producte en forma de oli groc fosc molt dens (0.52 g, 40%). Anàlisi trobat (calculat per $C_{14}H_{24}N_2O_5S_2$): C, 46.39 (46.14); H, 7.18 (6.64); N, 6.39 (7.69); S, 17.60 (20.09). P.m.: 365.48 g/mol. Ió $[M+H]^+$ amb distribució isotòpica 365.05(100), 366.05(18), 367.05(11), 368.05(2) i ió $[M+NH_4]^+$ amb distribució isotòpica 382.05(64), 383.05(11), 384.05(7), 385.05(1) trobat per espectroscòpia de masses d'ionització química. P. m. isòtop majoritari: 364.11 g/mol.

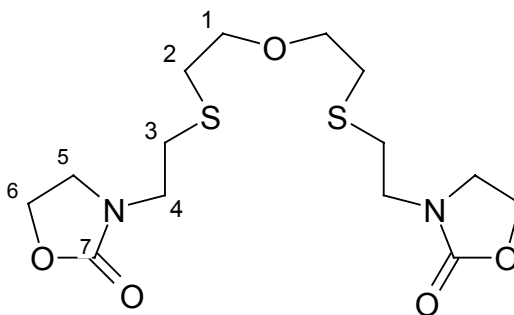
IR (NaCl)

ν (cm ⁻¹)	2918(I), 2860(I), 1746(mI), 1430(I), 1268(I), 1110(I), 762(lm)
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RMN (CDCl₃)

¹ H δ(ppm)	Multiplicitat	Integral	Assignació
4.35	pt	4H	H ₆
3.66	m	8H	H ₁ , H ₅
3.48	pt	4H	H ₄
2.78	m	8H	H ₂ , H ₃

¹³ C δ(ppm)	Assignació
158.36	C ₇
70.74	C ₁
61.80	C ₆
44.86	C ₅
43.58	C ₄
31.35, 30.20	C ₂ , C ₃



7 Complexos. Síntesi i caracterització

7.1 Complexos de Pd(II)

▪ Síntesi general i caracterització

Dissolem la sal de $[\text{Pd}(\text{CH}_3\text{CN})_4](\text{BF}_4)_2$ en acetonitril (5 ml) i sobre la solució groga resultant addicionem a poc a poc una solució del lligand corresponent en clorur de metilè (5ml). Portem a reflux la solució durant 2 h i agitem durant 12 h a temperatura ambient. Evaporem els dissolvents, afegim èter etílic (25 ml), agitem durant 30 min i filtrem el sòlid obtingut. Realitzem rentats amb clorur de metilè, per tal d'eliminar l'excés de lligand, i èter etílic.

En el cas del complex $[\text{Pd}(\text{L17})](\text{BF}_4)_2$ es fa reaccionar un excés de metall perquè la purificació del complex obtingut es realitza dissolent aquest en clorur de metilè per separar-lo de l'excés de metall que no ha reaccionat.

Els complexos dels lligands fosfamacrocíclics s'han sintetitzat emprant tècniques Schlenk i dissolvents perfectament desoxigenats.

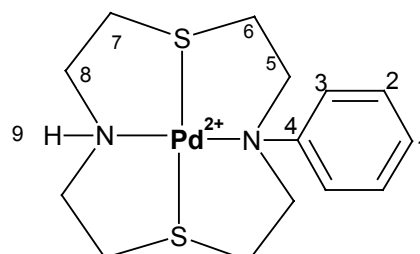
COMPLEX	Formula molecular proposada i pes molecular (g/mol)	Reactius		R(%)	ANÀLISI ELEMENTAL			
		L	Pd(II)		%C	%H	%N	%S
$[\text{Pd}(\text{L1})](\text{BF}_4)_2$	$\text{C}_{14}\text{H}_{22}\text{B}_2\text{F}_8\text{N}_2\text{PdS}_2$ 562.49	100 mg 0.34 mmol	142 mg, 0.32 mmol	100 mg 55%	29.81 (29.90)	4.22 (3.94)	4.88 (4.98)	10.37 (11.40)
$[\text{Pd}(\text{L2})](\text{BF}_4)_2$	$\text{C}_{16}\text{H}_{26}\text{B}_2\text{F}_8\text{N}_2\text{PdS}_2$ 590.55	50 mg 0.16 mmol	67 mg 0.15 mmol	45 mg 52%	32.58 (32.54)	4.17 (4.44)	4.58 (4.74)	10.80 (10.86)
$[\text{Pd}(\text{L4})](\text{BF}_4)_2$	$\text{C}_{16}\text{H}_{26}\text{B}_2\text{F}_8\text{NPPdS}_2$ 607.51	120mg 0.36 mmol	151 mg 0.34 mmol	77 mg 39%	30.35 (31.63)	4.30 (4.31)	2.30 (2.30)	10.33 (10.55)
$[\text{Pd}(\text{L5})](\text{BF}_4)_2$	$\text{C}_{20}\text{H}_{26}\text{B}_2\text{F}_8\text{NPPdS}_2$ 655.56	50 mg 0.13 mmol	53 mg 0.12 mmol	30 mg 39%	39.48 (36.64)	4.22 (3.99)	2.45 (2.13)	10.42 (9.78)
$[\text{Pd}(\text{L15})](\text{BF}_4)_2$	$\text{C}_{27}\text{H}_{36}\text{B}_2\text{F}_8\text{FeN}_2\text{PdS}_2$ 788.59	100 mg 0.20 mmol	80 mg 0.19 mmol	128 mg 85%	41.30 (41.12)	4.51 (4.60)	3.57 (3.55)	8.09 (8.13)
$[\text{Pd}(\text{L17})](\text{BF}_4)_2$	$\text{C}_{27}\text{H}_{36}\text{B}_2\text{F}_8\text{FeNPPdS}_2$ $\frac{1}{2}\text{CH}_2\text{Cl}_2$ 848.03	100 mg 0.19 mmol	84 mg 0.20 mmol	85 mg 55%	38.94 (38.79)	4.38 (4.40)	1.80 (1.65)	7.11 (7.56)
$[\text{Pd}_2(\text{Bi-L15})](\text{BF}_4)_4$	$\text{C}_{44}\text{H}_{62}\text{B}_4\text{F}_{16}\text{FeN}_4\text{Pd}_2\text{S}_4$ 2 CH_2Cl_2 1556.02	100 g 0.12 mmol	102 mg 0.23 mmol	136 mg 85%	34.16 (35.39)	4.33 (4.26)	4.26 (3.60)	7.80 (8.21)

COMPLEX	DESCRIPCIÓ	SOLUBILITAT (color de la solució)	Λ (μS) (dissolvent)	IR (cm^{-1})
[Pd(L1)](BF₄)₂	sòlid groc	acetoneitril (grog) acetona (grog) metanol calent (grog)	255 (acetoneitril)	3270(pI), 3225(pI), 2958(pI), 2920(pI), 1600(pI), 1494(pI), 1461(pI), 1417(pI), 1215(lm), 1125(lm), 1039(ml), 1029(ml), 966(ml), 780(lm), 725(lm), 697(l)
[Pd(L2)](BF₄)₂	sòlid taronja	acetoneitril (taronja) acetona (taronja) metanol calent (taronja)	260 (acetoneitril)	3377(b), 3234(pI), 3007(p), 2941(pI), 1160(pI), 1592(pI), 1487(pI), 1470(pI), 1055(ml), 1020(ml), 996(ml), 775(l), 741(l), 702(ml)
[Pd(L4)](BF₄)₂	sòlid groc ataronjat	acetoneitril (grog) acetona (grog) metanol calent (grog)	200 (acetoneitril)	3606(b), 3251(pI), 2965(pI), 2934(pI), 1626(pI), 1436(lm), 1410(lm), 1370(pI), 1285(pI), 1045(ml), 1030(ml), 834(l), 751(lm), 726(lm), 694(lm), 700(lm)
[Pd(L5)](BF₄)₂	sòlid groc ataronjat	acetoneitril (grog) acetona (grog) metanol calent (grog)	220 (acetoneitril)	3630(b), 3010(pI), 2945(pI), 2925(pI), 1715(pI), 1605(lm), 1505(lm), 1435(lm), 1235(pI), 1048(ml), 1025(ml), 996(l), 743(pI), 692(lm), 651(lm)
[Pd(L15)](BF₄)₂	sòlid taronja fosc	acetoneitril (taronja) acetona (taronja) nitrometà (taronja)	291 (acetoneitril)	3600(b), 3087(pI), 2994(pI), 1738(lm), 1467(lm), 1454(lm), 1425(lm), 1353(lm), 1216(lm), 1043(ml), 1025(ml), 725(lm), 928(l), 832(lm), 697(lm), 669(lm)
[Pd(L17)](BF₄)₂	sòlid taronja fosc	acetoneitril (taronja) acetona (taronja) clorur de metilè (taronja) nitrometà (taronja)	300 (acetoneitril)	3631(pI), 3554(pI), 3082(pI), 2917(pI), 1637(pI), 1593(pI), 1488-1408(pI), 1025(b), 921(lm), 811(lm), 767(lm), 730(lm), 699(lm)
[Pd₂(Bi-L15)](BF₄)₄	sòlid taronja fosc	acetoneitril (taronja) acetona (taronja) nitrometà (taronja)	522 (acetoneitril)	3614(b), 3559(b), 3073(pI), 2995(pI), 2951(pI), 1700-1600(lm), 1490(lm), 1460(lm), 1452(lm), 1410(lm), 1315(lm), 1050(ml), 11019(ml), 929(ml), 845(l), 764(l), 737(l), 697(l)

[Pd(L1)](BF₄)₂RMN (CD₃CN)

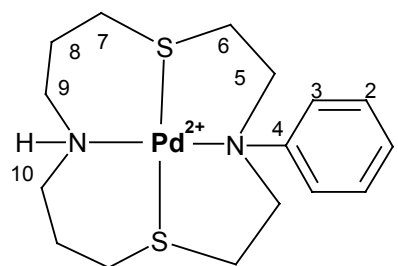
¹ H δ(ppm)	Multiplicitat	Integral	Assignació
7.75-7.72	m	2H	H ₂
7.59-7.55	m	3H	H ₁ , H ₃
4.53	m	2H	H ₅
3.83	m	6H	H ₇ , H ₆ , H ₈
3.63	m	2H	H ₅
3.34	m	4H	H ₈ , H ₇
3.11	m	2H	H ₆

¹³ C δ(ppm)	Assignació
146.58	C ₄
130.29	C ₂
129.84	C ₁
123.76	C ₃
66.75	C ₅
57.14	C ₈
35.23	C ₇
33.92	C ₆

**[Pd(L2)](BF₄)₂**RMN (CD₃CN)

¹ H δ(ppm)	Multiplicitat	Integral	Assignació
7.67-7.60	m	5H	H _{aromàtics}
4.5-2.5	m	20H	H _{alifàtics}

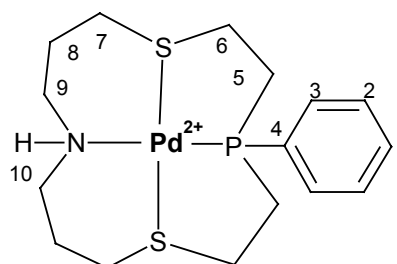
¹³ C δ(ppm)	Assignació
145.00	C ₄
131.29, 131.10, 130.97	C ₂ , C ₁
125.28 (banda ampla)	C ₃
70-21	C _{alifàtics}

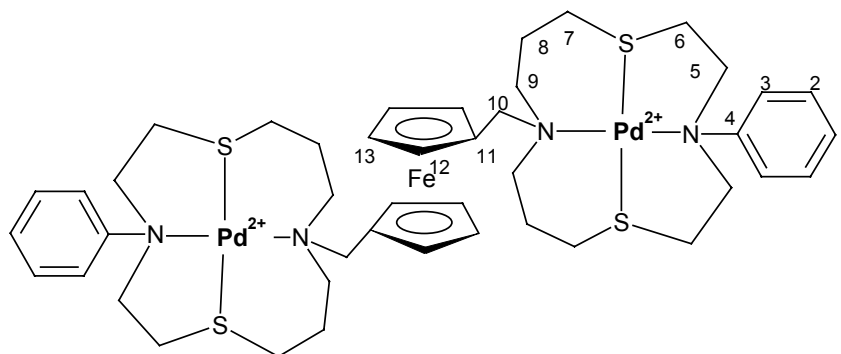
**[Pd(L4)](BF₄)₂**RMN (CD₃CN)

¹ H δ(ppm)	Multiplicitat	Integral	Assignació
8.00-7.05	m	5H	H _{aromàtics}
4.0-2.4	m	20H	H _{alifàtics}

¹³ C δ(ppm)	Multiplicitat	Assignació
134.65, 133.88, 133.69	m	C ₄ , C ₃
130.51, 130.31	m	C ₂ , C ₁
49.90	s	C ₉
38.04	s	C ₇
33.85	d (J _{C5-P} =34.26 Hz)	C ₅
32.79	s	C ₆
24.17	s	C ₈

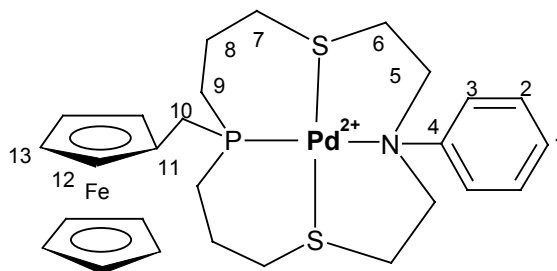
³¹ P δ(ppm)	Multiplicitat
107.97	s



$[\text{Pd}_2(\text{Bi-L15})](\text{BF}_4)_4$ RMN (CD_3CN)

^1H δ (ppm)	Multiplicitat	Integral	Assignació
7.78	m	4H	H ₁
7.60	m	6H	H ₂ , H ₃
4.79	s	12H	H ₁₃
4.59	s		H ₁₂
4.59	m		H ₅
3.8-3.5	m	20H	H ₁₀ , H ₉ , H ₇ , H ₅
3.30	m	4H	H ₇
3.1-2.3	m	16H	H ₆ , H ₈

^{13}C δ (ppm)	Multiplicitat	Assignació
144.13	s	C ₄
131.07	s	C ₂
-	-	C ₁
125.15	s	C ₃
80.34	s	C ₁₁
73.19	s	C ₁₂
71.82	s	C ₁₃
68.17	s	C ₅
60.46	s	C ₉
60.28	b	C ₁₀
35.92	s	C ₆
32.04	s	C ₇
23.66	s	C ₈

[Pd(L17)](BF₄)₂RMN (CD₃CN)

¹ H δ(ppm)	Multiplicitat	Integral	Assignació
7.8-7.5	m	5H	H ₁ , H ₂ , H ₃
4.4-4.2	m	11H	H ₁₁ , H ₁₂ , H ₁₃ , H _(Cp no substituït) , H ₅
3.76	m	2H	H ₅
3.33	m	8H	H ₁₀ , H ₇ , H ₆
3.09	m	2H	H ₆
2.7-2.0	m	8H	H ₈ , H ₉

¹³ C δ(ppm)	Multiplicitat	Assignació
144.96	s	C ₄
131.17	s	C ₂
130.72	s	C ₁
125.16	s	C ₃
70.17	b	C _(Cp no substituït)
63.99	s	C ₅
40.78	s	C ₆
34.68	s	C ₇
25.05	d	C ₁₀
22.76	s	C ₈
21.57	d (J _{C9-P} = 30.25 Hz)	C ₉

³¹ P δ(ppm)	Multiplicitat
24.13	s

7.2 Complexos de Cu(II)

▪ Síntesi general i caracterització

Com a sals de partida s'ha emprat $\text{Cu}(\text{ClO}_4)_2 \cdot 6 \text{H}_2\text{O}$ i $\text{Cu}(\text{BF}_4)_2 \cdot n \text{H}_2\text{O}$ (20-22% en Cu).

Dissolem el macrocicle en clorur de metilè (5ml) i l'afegim sobre una solució de la sal metàl·lica en etanol absolut (25 ml). La solució esdevé de color verd fosc. Agitem durant 12 h, evaporem el dissolvent i afegim èter etílic. Agitem durant 30 min i finalment obtenim una precipitat que rentem diverses vegades amb etanol i èter etílic.

El complex $[\text{Cu}(\text{ClO}_4)(\text{L1})]\text{ClO}_4$ s'extreu del cru de reacció amb clorur de metilè. El complex $[\text{Cu}(\text{L2})](\text{BF}_4)_2$ precipita completament en el si de la reacció.

COMPLEX	Formula molecular proposada pes molecular (g/mol)	Reactius		R (%)	ANÀLISI ELEMENTAL			
		L	Cu(II)		%C	%H	%N	%S
$[\text{Cu}(\text{ClO}_4)(\text{L1})]\text{ClO}_4$	$\text{C}_{14}\text{H}_{22}\text{Cl}_2\text{CuN}_2\text{O}_8\text{S}_2$ 544.91	100 mg 0.35 mmol	122 mg 0.34 mmol	70 mg 40%	30.20 (30.86)	3.98 (4.07)	5.11 (5.14)	9.97 (11.77)
$[\text{Cu}(\text{L2})](\text{BF}_4)_2$	$\text{C}_{16}\text{H}_{26}\text{B}_2\text{CuF}_8\text{N}_2\text{S}_2$ 1 H_2O 565.69	100 mg 0.32 mmol	95 mg 0.30 mmol	122 mg 74%	33.94 (33.97)	4.72 (4.98)	5.02 (4.95)	10.81 (11.33)
$[\text{Cu}(\text{L15})](\text{BF}_4)_2$	$\text{C}_{27}\text{H}_{36}\text{B}_2\text{CuF}_8\text{FeN}_2\text{S}_2$ 745.72	100 mg 0.20 mmol	60 mg 0.19 mmol	108 mg 76%	43.67 (43.47)	5.09 (4.87)	4.18 (3.76)	9.14 (8.60)

COMPLEX	DESCRIPCIÓ	SOLUBILITAT (color de la solució)	Λ (μS) (dissolvent)	IR (cm^{-1})
$[\text{Cu}(\text{ClO}_4)(\text{L1})]\text{ClO}_4$	sòlid verd fosc	nitrometà (verda) acetona (verda) etanol (verda) clorur de metilè (verda)	107 (acetona) 140 (metanol)	3510(b), 3104(pI), 2970(pI), 2965(pI), 2900(pI), 1737(lm), 1596(lm), 1574(lm), 1494(lm), 1454(lm), 1416(lm), 1367(lm), 1284(lm), 1228(lm), 1179(lm), 1078(bl), 929(lm)
$[\text{Cu}(\text{L2})](\text{BF}_4)_2$	sòlid violeta	cloroform (violeta) clorur de metilè (violeta) acetona (violeta) metanol (verda)	98 (acetona) 160 (metanol)	3429(b), 3151(ml), 2923(im), 1705(lm), 1597(l), 1504(l), 1417(lm), 1360(lm), 1300(lm), 1105(ml), 1063(ml), 1057(ml), 752(lm), 700(lm), 533(lm), 522(lm)
$[\text{Cu}(\text{L15})](\text{BF}_4)_2$	sòlid verd	parcialment insoluble en metanol, etanol i acetoniitril (solucions verdes)	-	3552(b), 3113(lm), 2935(lm), 2928(lm), 1598(i), 1505(l), 1470(lm), 1283(lm), 1099(ml), 1070(ml), 826(lm), 756(lm), 697(lm), 521(lm)

Espectroscòpia electrònica (UV-VIS)

Complex	Concentració	Dissolvent	λ (nm)	ϵ (l cm ⁻¹ mol ⁻¹)
[Cu(ClO ₄)(L1)]ClO ₄	1 mM	acetona	640	222
		metanol	637	212
[Cu(L2)](BF ₄) ₂	1 mM	acetona	556	556
		metanol	580	290

7.3 Complexos de Ni(II)

▪ Síntesi general i caracterització

Dissolem el macrocicle en clorur de metilè (5ml) i l'afegim sobre una solució de Ni(ClO₄)₂·6 H₂O en acetona (5 ml) observant immediatament canvis de color en la solució. Agitem durant 12 h, evaporem el dissolvent i afegim èter etílic (25 ml). Agitem durant 30 min i finalment obtenim un precipitat que rentem diverses vegades amb clorur de metilè i èter etílic. La síntesi del complex [Ni(L17)](ClO₄)₂ també es va realitzar emprant acetonitril en lloc d'acetona, obtenint el mateix resultat.

Els complexos de Ni(II) pentacoordinats es sintetitzen per addició de solucions dels corresponents halurs (2 ml) en forma de sal de tetrabutilamoni (n-Bu₄NX) sobre solucions del complex [Ni(L17)](ClO₄)₂ en acetonitril (2 ml). Un cop realitzada la mescla, s'agita durant tota la nit, s'evaporen els dissolvents de síntesi i es dissol el sòlid obtingut en la mínima quantitat d'una dissolució calenta de clorur de metilè-toluè 1:1. Els cristalls porpres formats després de l'evaporació parcial del clorur de metilè, es filtren, es renten diverses vegades amb èter etílic i s'assequen al buit.

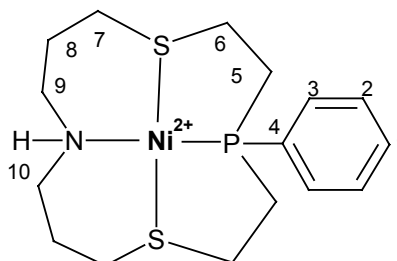
COMPLEX	Formula molecular proposada pes molecular (g/mol)	Reactius		R (%)	ANÀLISI ELEMENTAL			
		L	Ni(II)		%C	%H	%N	%S
[Ni(L4)](ClO ₄) ₂	C ₁₆ H ₂₆ Cl ₂ NNiO ₈ PS ₂ 585.08	50 mg 0.15 mmol	52 mg 0.14 mmol	50 mg 61%	33.27 (32.84)	4.46 (4.47)	2.51 (2.39)	11.07 (10.96)
[Ni(L17)](ClO ₄) ₂	C ₂₇ H ₃₆ Cl ₂ FeNNiO ₈ PS ₂ 783.12	60 mg 0.11 mmol	42 mg 0.12 mmol	82 mg 85%	41.39 (41.41)	4.86 (4.63)	1.95 (1.79)	8.29 (8.19)

COMPLEX	Formula molecular proposada pes molecular (g/mol)	Reactius		R (%)	ANÀLISI ELEMENTAL			
		[Ni(L17)](ClO ₄) ₂	n-Bu ₄ NX		%C	%H	%N	%S
[NiCl(L17)]ClO ₄	C ₂₇ H ₃₆ Cl ₂ FeNNiO ₄ PS ₂ 2 CH ₂ Cl ₂ 888.92	25 mg 0.032 mmol	9 mg 0.035 mmol	10 mg 45%	39.22 (39.18)	4.47 (4.53)	1.34 (1.57)	7.81 (7.21)
[NiBr(L17)]ClO ₄	C ₂₇ H ₃₆ BrClFeNNiO ₄ PS ₂ 1 CH ₂ Cl ₂ 848.511	50 mg 0.064 mmol	21 mg 0.070 mmol	30 mg 55%	39.62 (39.63)	4.51 (4.51)	1.66 (1.65)	7.55 (7.55)
[NiI(L17)]ClO ₄	C ₂₇ H ₃₆ IClFeNNiO ₄ PS ₂ 2 CH ₂ Cl ₂ 980.44	25 mg 0.032 mmol	13 mg 0.035 mmol	12 mg 44%	34.24 (35.52)	3.64 (4.11)	1.39 (1.42)	6.59 (6.54)

COMPLEX	DESCRIPCIÓ	SOLUBILITAT (color de la solució)	Δ (μS) (dissolvent)	IR (cm ⁻¹)
[Ni(L4)](ClO ₄) ₂	sòlid taronja	acetona (taronja) nitrometà (taronja) acetonitril (taronja)	242 (acetonitril)	3484(b), 3200(b), 2972(pI), 2934(pI), 1622(banda), 1466(lm), 1451(lm), 1435(lm), 1411(lm), 1290(pl), 1245(pl), 1056(banda intensa), 998(I), 927(I), 840(I), 722(I), 693(I), 602(ml)
[Ni(L17)](ClO ₄) ₂	sòlid vermell fosc	acetonitril (vermella) acetona (vermella) nitrometà (vermella)	233 (acetonitril)	3484(b), 3200(b), 3050(pI), 2972(pI), 2934(pI), 1622(lm), 1466(pI), 1451(lm), 1435(I), 1313(pI), 1290(pI), 1245(pI), 1056(banda intensa), 998(I), 927(I), 840(I), 722(I), 693(I), 699(I)
[NiCl(L17)]ClO ₄	sòlid porpra	acetonitril (porpra) nitrometà (porpra) clorur de metilè (porpra)	153 (acetonitril)	3626(pI), 3540(pI), 3009(pI), 2974(pI), 2909(pI), 1622(lm), 1608(lm), 1496(lm), 1460(lm), 1410(lm), 1386(lm), 1250-1100(lm), 1095(I), 1083(I), 1000(I), 790(lm), 735 (Im), 620(I)
[NiBr(L17)]ClO ₄	sòlid porpra fosc	acetonitril (porpra) nitrometà (porpra) clorur de metilè (porpra) cloroform (porpra)	135 (acetonitril)	3317(b), 3100-3000(pI), 2953(pI), 2917(lm), 2822(lm), 1620(lm), 1610(lm), 1488(lm), 1461(lm), 1414(lm), 1392(lm), 1250-1100(lm), 1095(I), 1088(I), 996(I), 900(I), 822(I), 804(ml), 769(lm), 735(lm), 703(lm)
[NiI(L17)]ClO ₄	sòlid porpra fosc	acetonitril (porpra) nitrometà (porpra) clorur de metilè (porpra)	162 (acetonitril)	3096(pI), 2937(pI), 2920(pI), 1612(B), 1503(lm), 1488(lm), 1450(lm), 1404(lm), 1392(lm), 1084(ml), 1080(ml), 922(I), 836(lm), 774(lm), 705 (im)

Espectroscòpia electrònica (UV-VIS)

Complex	Concentració	Dissolvent	λ (nm)	ϵ (1 cm ⁻¹ mol ⁻¹)
[Ni(L4)](ClO ₄) ₂	1 mM	acetonitril	409	572
		acetona	449	759
[Ni(L17)](ClO ₄) ₂	1 mM	acetonitril	477	326
[Ni(L17)](ClO ₄) ₂	1 mM	acetonitril	477	312
[NiCl(L17)]ClO ₄	1 mM	acetonitril	490	558
[NiBr(L17)]ClO ₄	1 mM	acetonitril	494	598
[Ni(L17)]ClO ₄	1 mM	acetonitril	413	511
			507	525

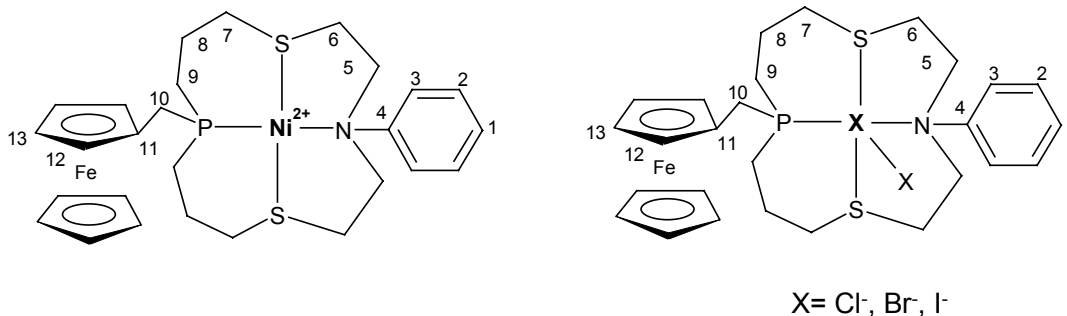
[Ni(L4)](ClO₄)₂RMN (CD₃COCD₃)

¹ H δ (ppm)	Multiplicitat	Integral	Assignació
8.1-7.4	m	5H	H _{aromàtics}
4.1-2.5	m	16H	H _{alifàtics}

¹³ C δ (ppm)	Multiplicitat	Assignació
135-125	-	C _{aromàtics}
50-20	-	C _{alifàtics}

³¹ P δ (ppm)	Multiplicitat
47.29	s

[Ni(L17)](ClO₄)₂, [NiCl(L17)]ClO₄, [NiBr(L17)]ClO₄ i [NiI(L17)]ClO₄ RMN (CD₃CN)



¹H RMN

Complex	H _{aromatics}	H ₅	H ₆	H ₇	H ₈	H ₉	H ₁₀	H ₁₂	H ₁₃	H _{Cp}
[Ni(L17)](ClO ₄) ₂	8.00-7.50	4.55 (m) 3.60 (m)	2.98 (dd) 2.66 (m)	3.43 (m) 3.10 (m)	2.70-1.50		3.27 (d)	4.47(m)	4.28(m)	4.32 (s)
[NiCl(L17)]ClO ₄	8.30-7.40	5.11 (m) 3.27 (m)	2.89 (m) 2.57 (m)	3.94 (m) 2.91 (m)	2.70-1.50		3.21 (d)	4.34(m)	4.36(m)	4.27(s)
[NiBr(L17)]ClO ₄	8.30-7.40	5.30 (m) 3.37 (m)	2.93 (m) 2.60 (m)	4.03 (m) 2.95 (m)	2.70-1.50		3.24 (d)	4.36(m)	4.38(m)	4.29(s)
[NiI(L17)]ClO ₄	8.30-7.40	5.53 (m) 3.51 (m)	2.99 (m) 2.70 (m)	4.11 (m) 3.01 (m)	2.70-1.50		3.32 (d)	4.35 (m)		4.30(s)

¹³C{¹H} RMN

Complex	C ₁	C ₂	C ₃	C ₄	C ₅	C ₆	C ₇	C ₈	C ₉	C ₁₀
[Ni(L17)](ClO ₄) ₂	130.73	131.04	129.77	144.85	62.80	38.64	34.57	21.79	21.09 d J _{C-P} = 30.18 Hz	27.24 d J _{C-P} = 22.15 Hz
[NiCl(L17)]ClO ₄	129.47	130.01	124.87	-	60.29	38.01	34.29	22.02	20.32 d J _{C-P} = 30.51 Hz	-
[NiBr(L17)]ClO ₄	129.53	130.08	124.89	145.03	60.75	37.87	34.99	22.07	20.69 d J _{C-P} = 30.51 Hz	-
[NiI(L17)]ClO ₄	129.72	130.24	124.85	-	61.61	37.87	36.16	22.09	20.34 d J _{C-P} = 30.52 Hz	-

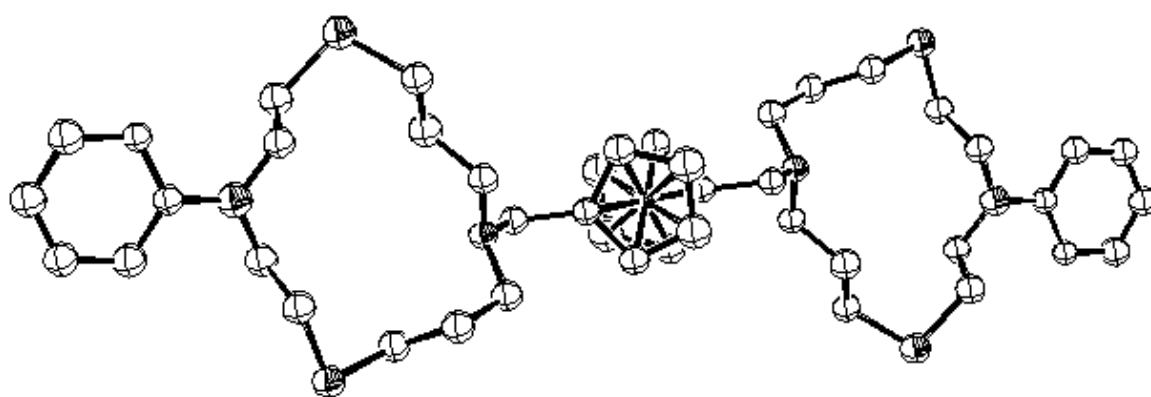
Complex	C ₁₁	C ₁₂	C ₁₃	C _{Cp}
[Ni(L17)](ClO ₄) ₂	70.19	69.31	80.05	69.79
[NiCl(L17)]ClO ₄	69.53	68.28	-	69.04
[NiBr(L17)]ClO ₄	69.42	68.30	-	69.02
[NiI(L17)]ClO ₄	69.41	68.41	-	69.07

³¹P{¹H} RMN

Complex	δ (ppm)
[Ni(L17)](ClO ₄) ₂	9.72
[NiCl(L17)]ClO ₄	11.57
[NiBr(L17)]ClO ₄	11.87
[NiI(L17)]ClO ₄	12.24

SÍNTESI I CARACTERITZACIÓ DE MACROCICLES AMB FUNCIONALITZACIONS ALIFÀTIQUES I ELECTROQUÍMICAMENT ACTIVES

ANNEX



Departament de Química
Facultat de Ciències



Bellaterra, març del 2004

Autor: Mateu Pla Roca

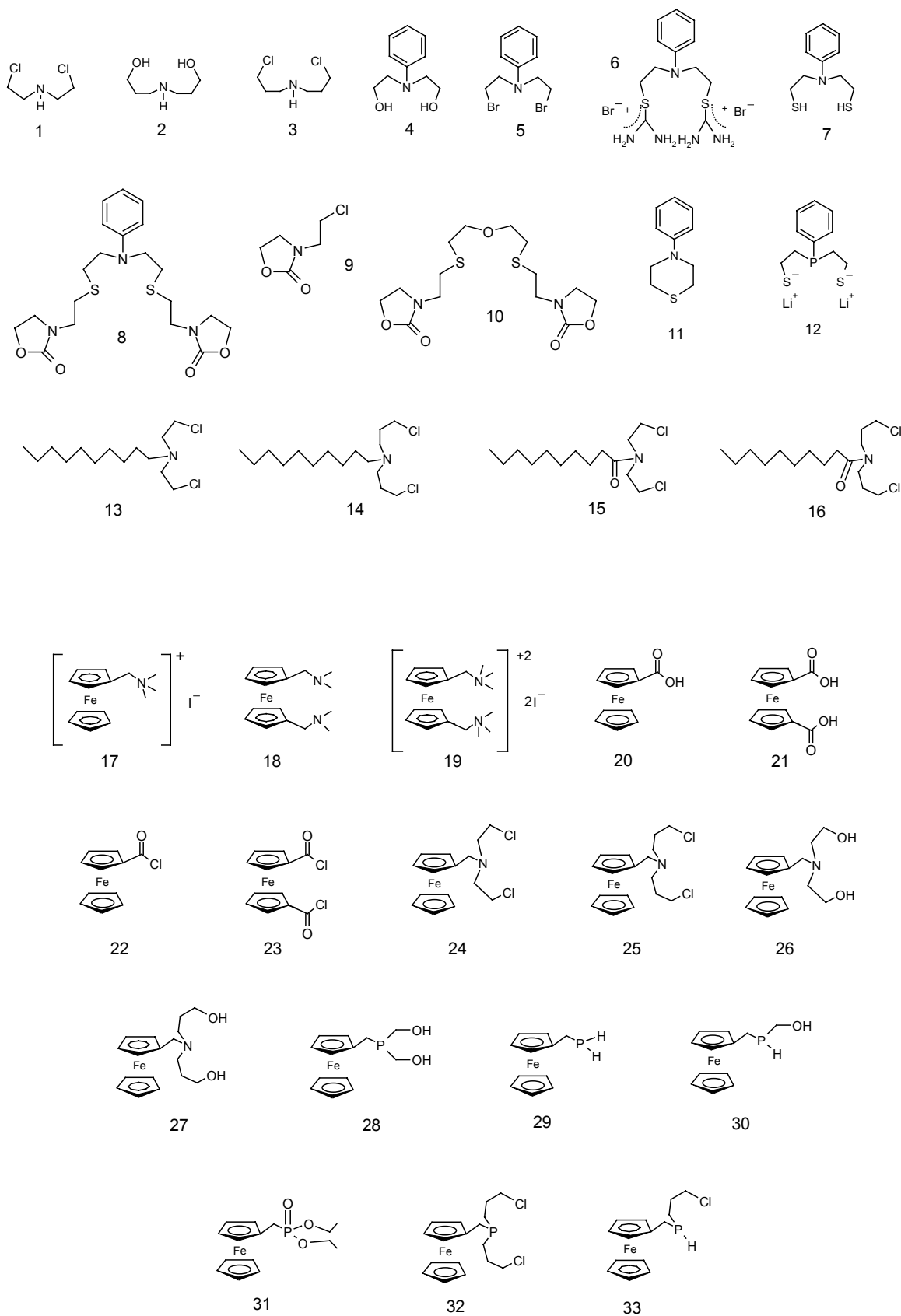
Direcció: Lluís Escriche Martínez

ÍNDIX

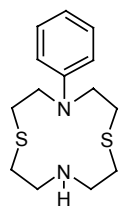
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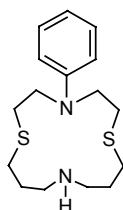
PRECURSORS DE SÍNTESI



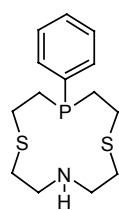
MACROCICLES



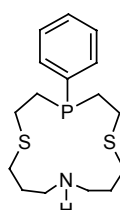
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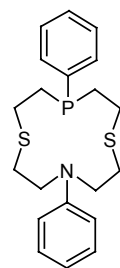
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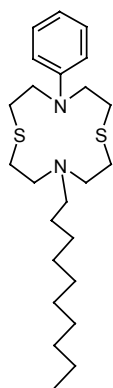
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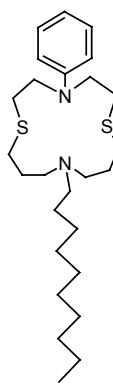
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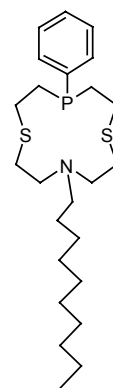
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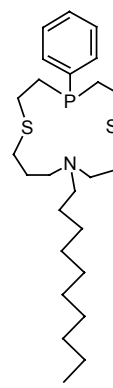
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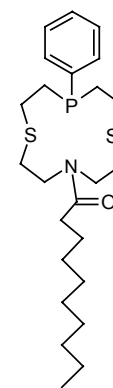
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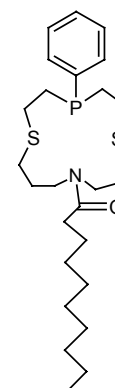
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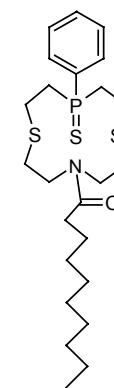
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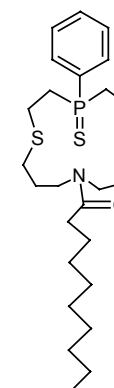
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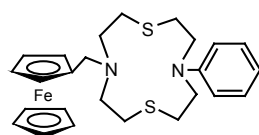
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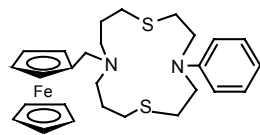
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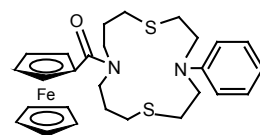
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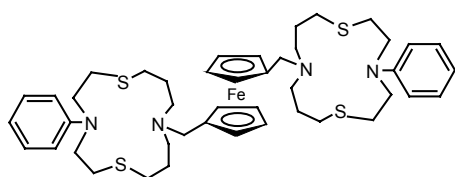
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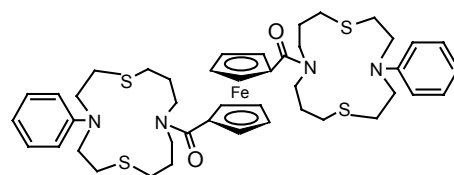
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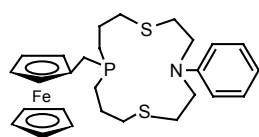
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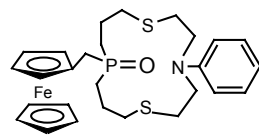
Bi-L15



Bi-L16



L17



L18

APÈNDIX I:

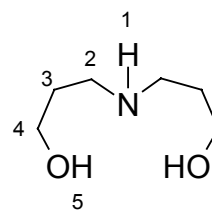
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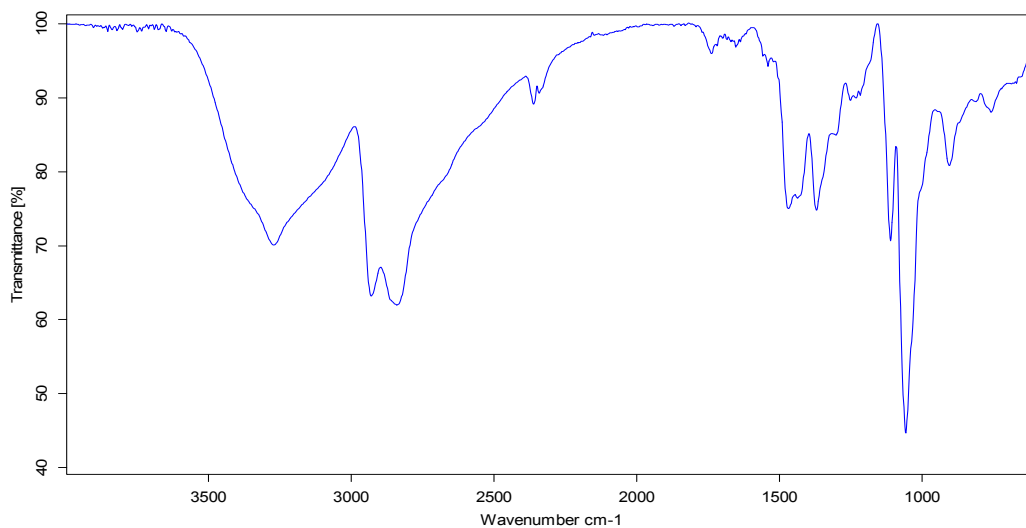
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1 Precursors de síntesi

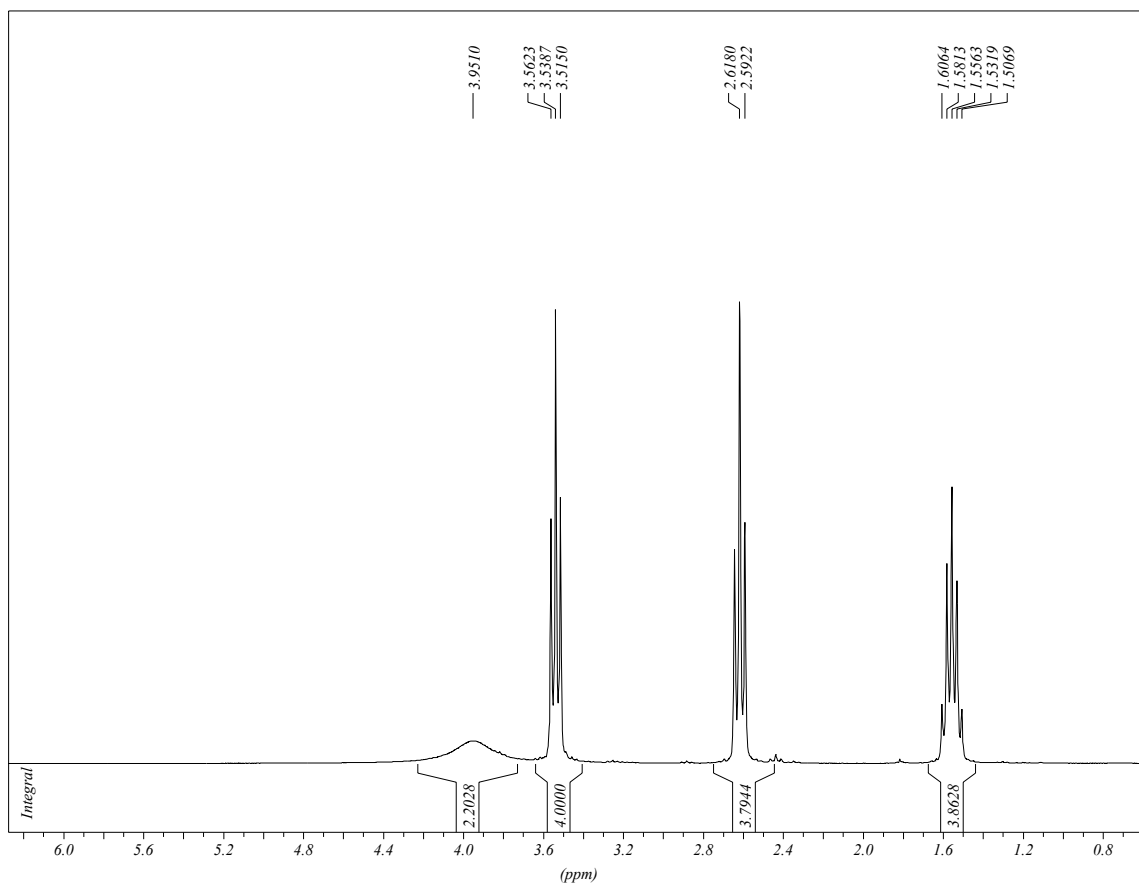
- bis(3-hidroxipropil)amina (2)



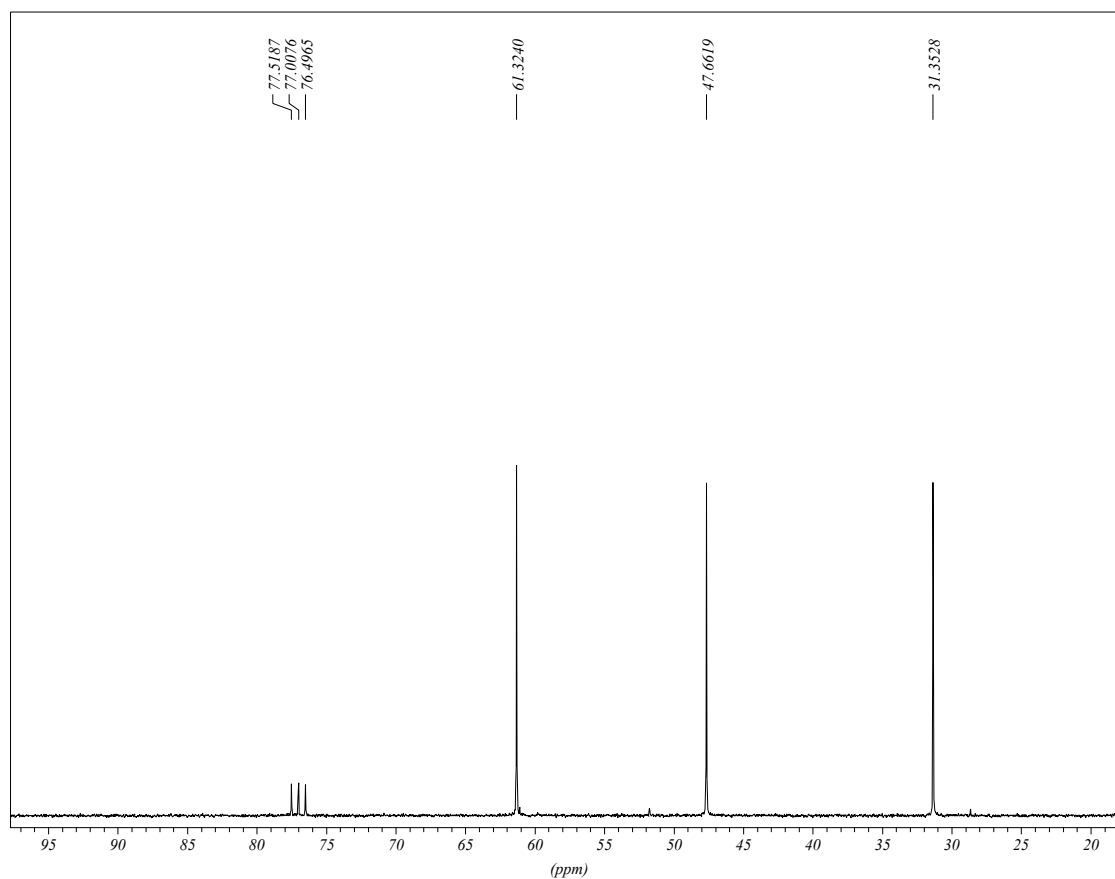
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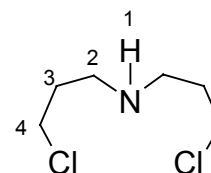
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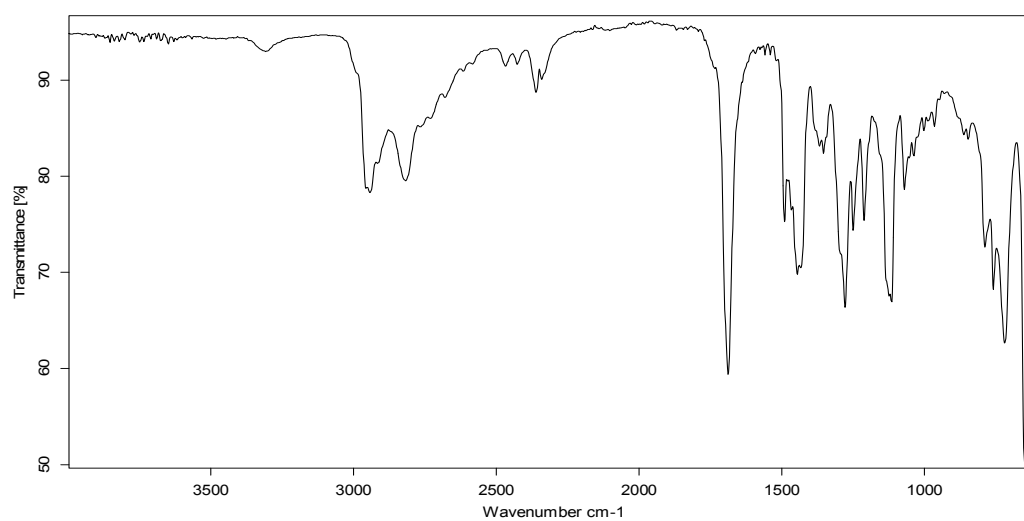
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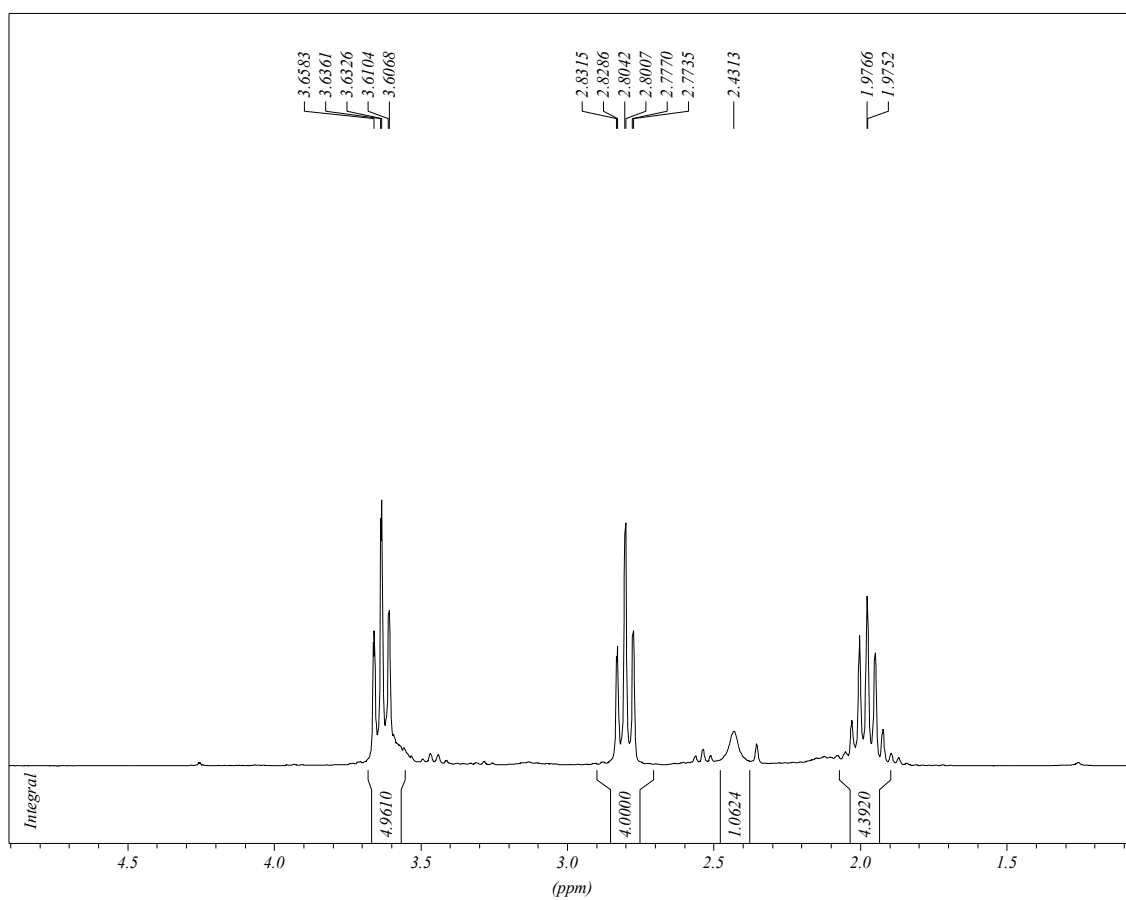
▪ bis(3-cloropropil)amina (3)



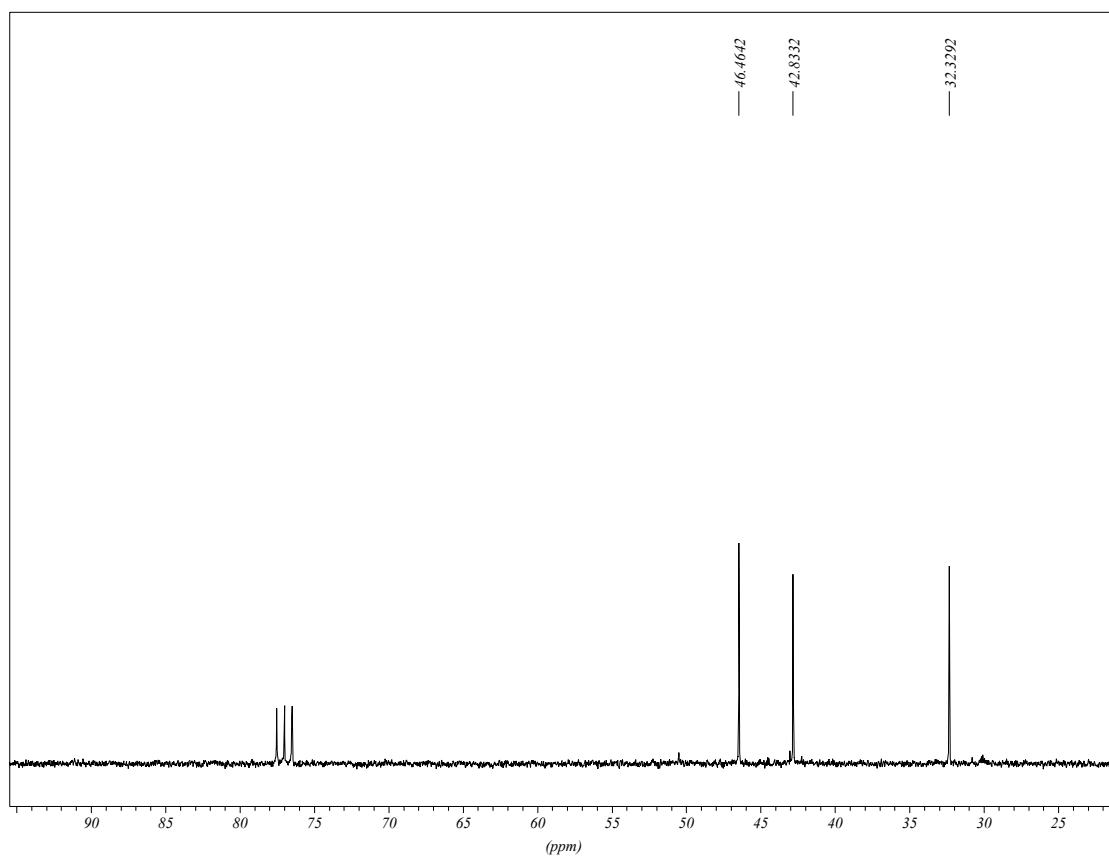
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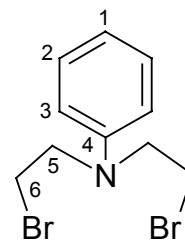
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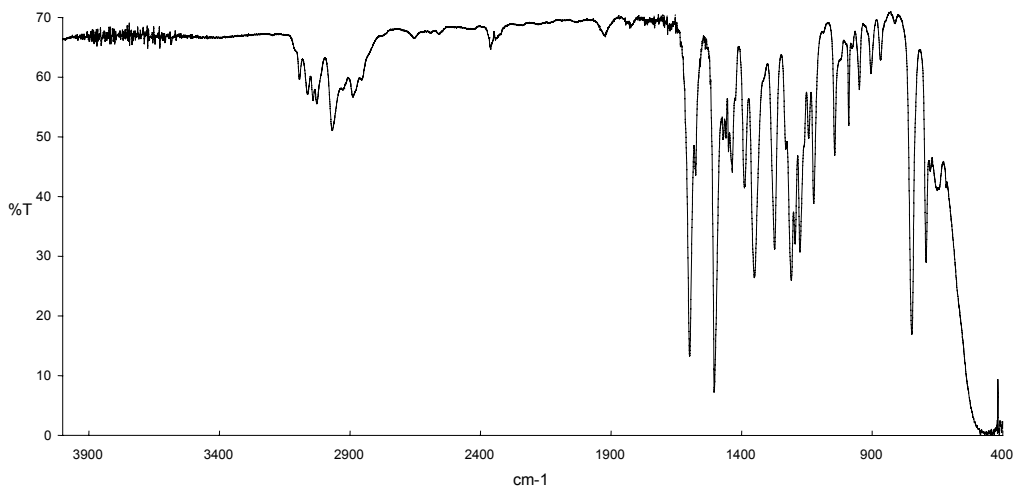
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



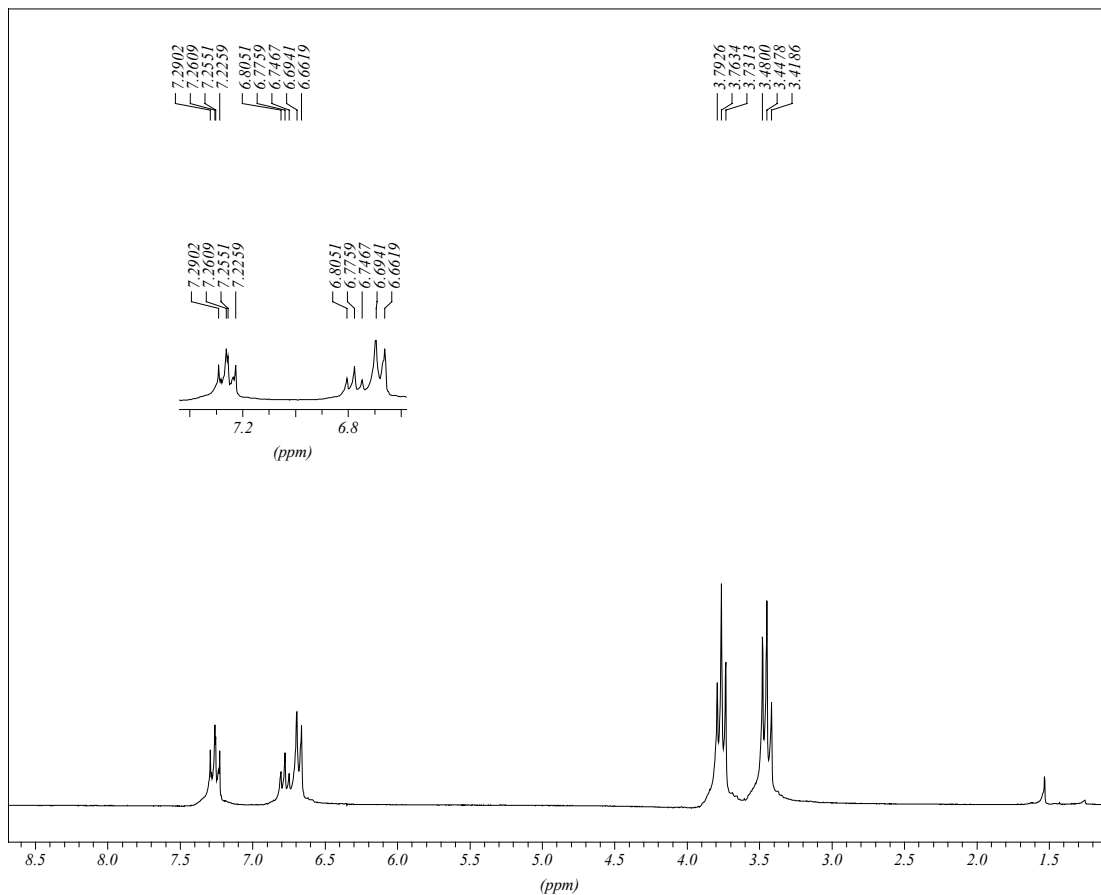
▪ N,N-bis(2-bromoetil)anilina (5)



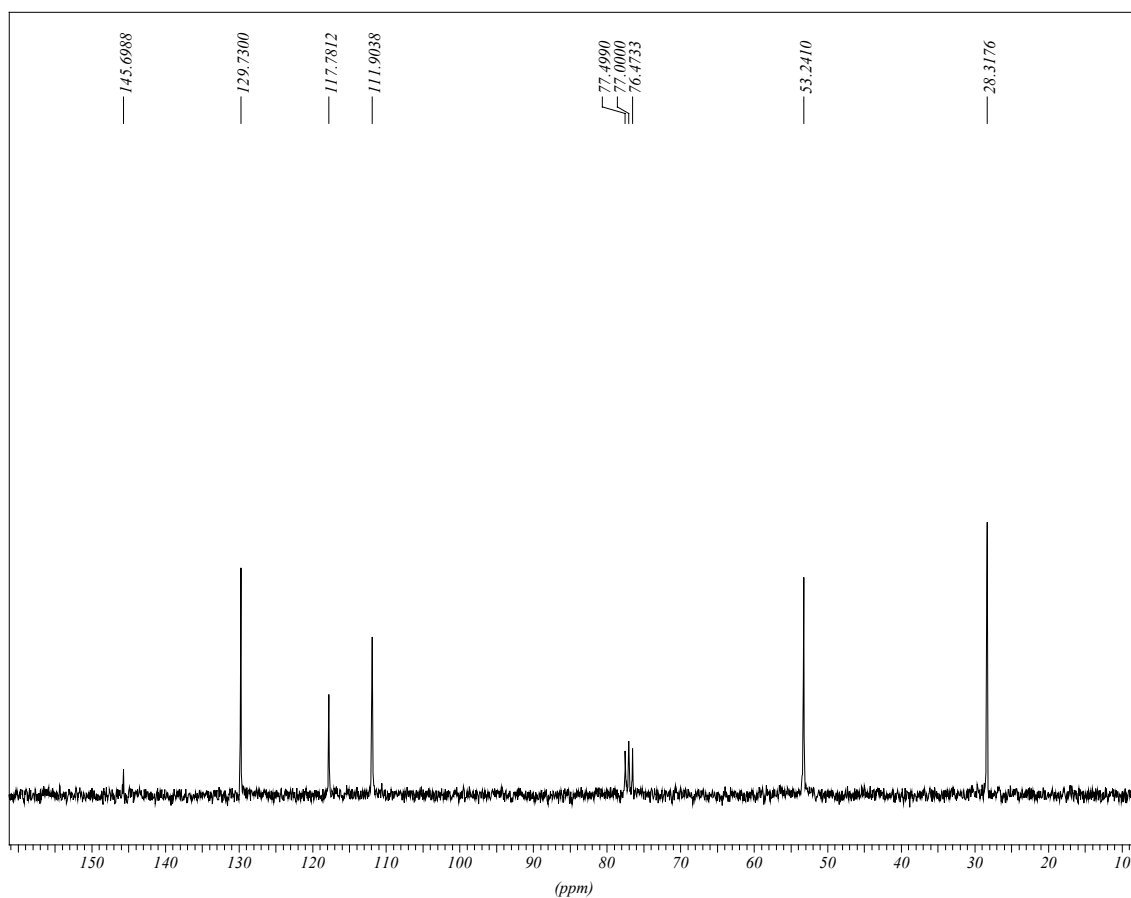
IR (NaCl)



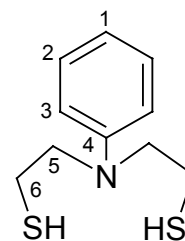
¹H RMN (CDCl₃)



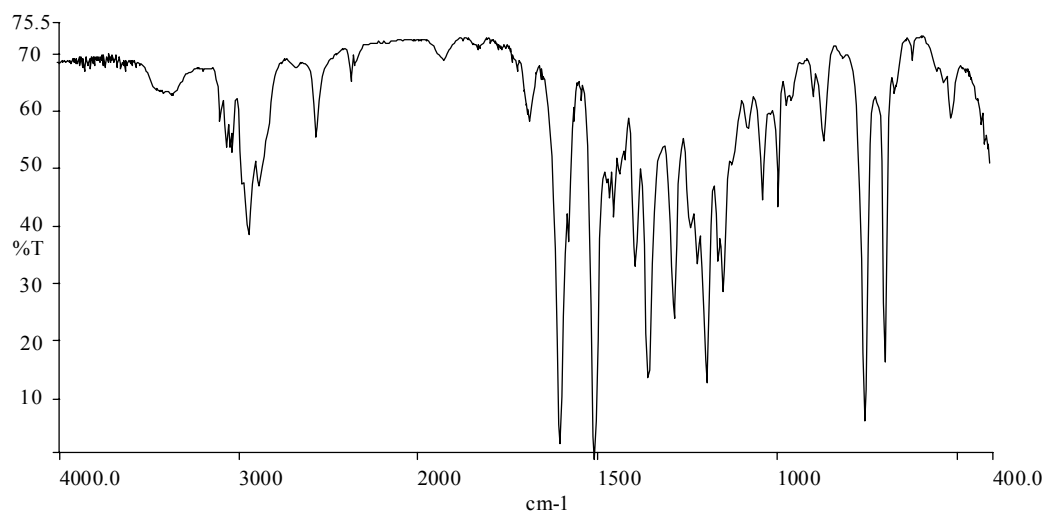
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



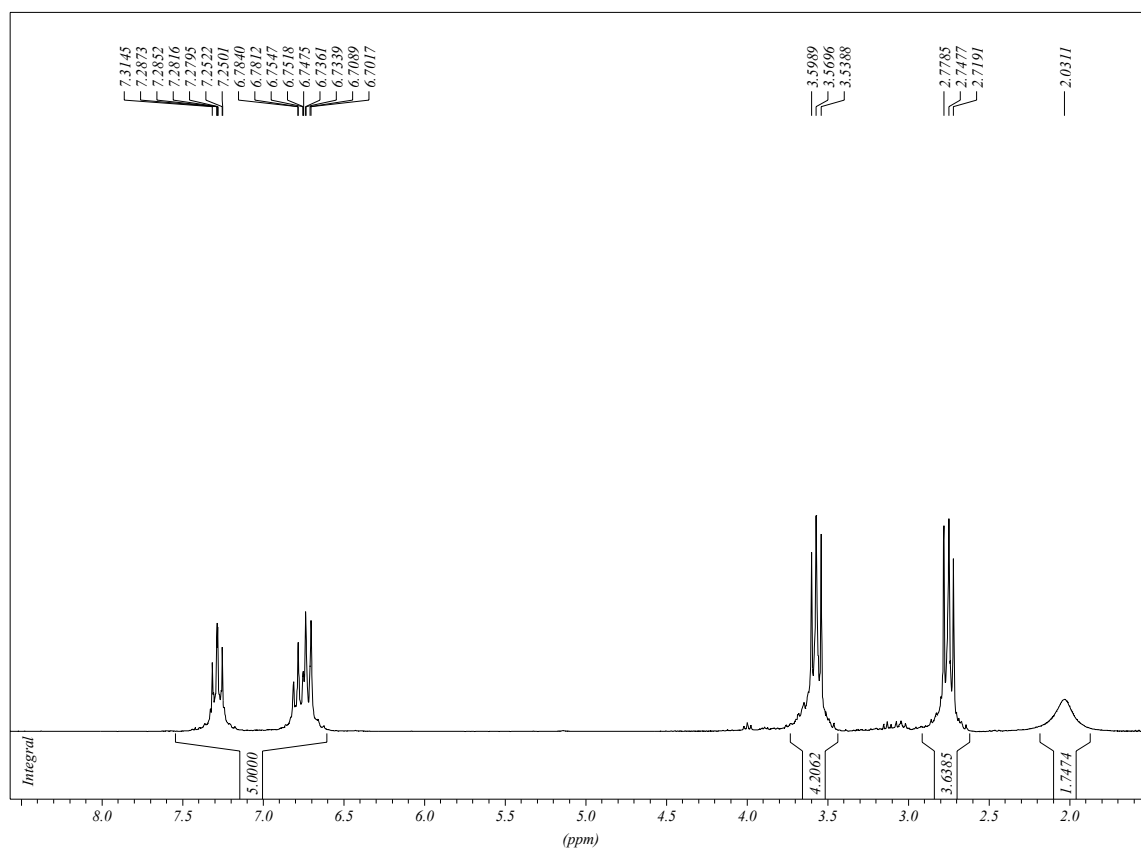
▪ **N,N-bis(2-mercaptoetil)anilina (7)**



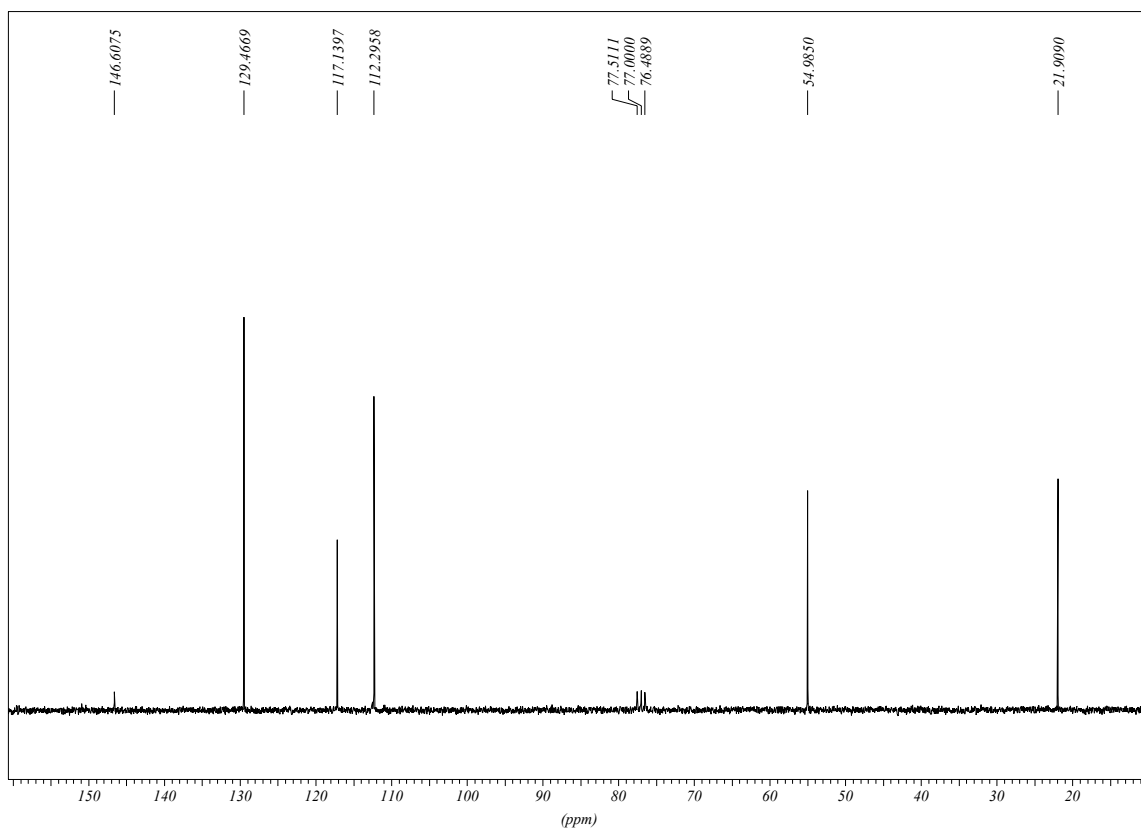
IR (NaCl)



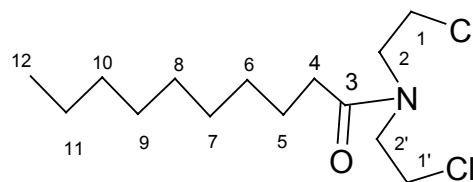
^1H RMN (CDCl_3)



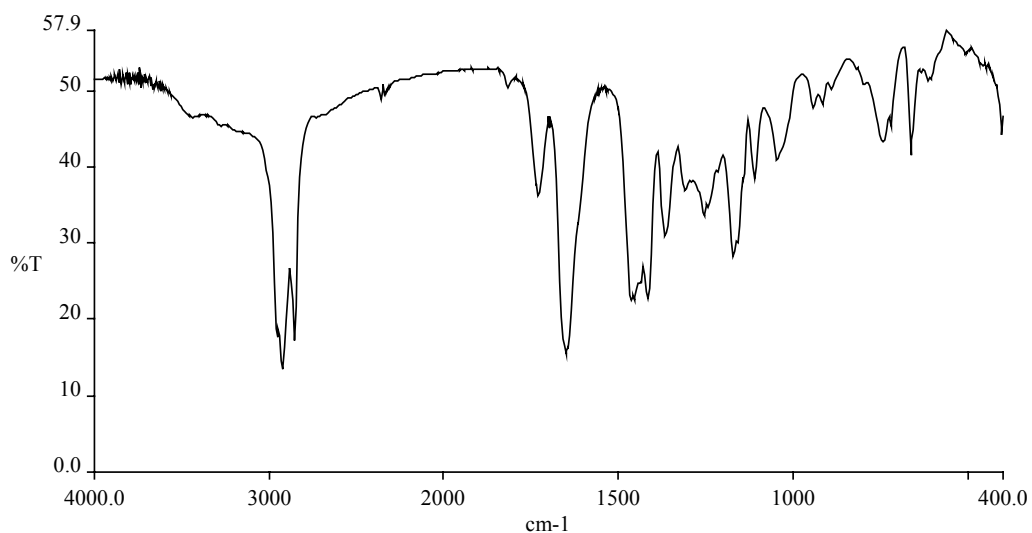
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



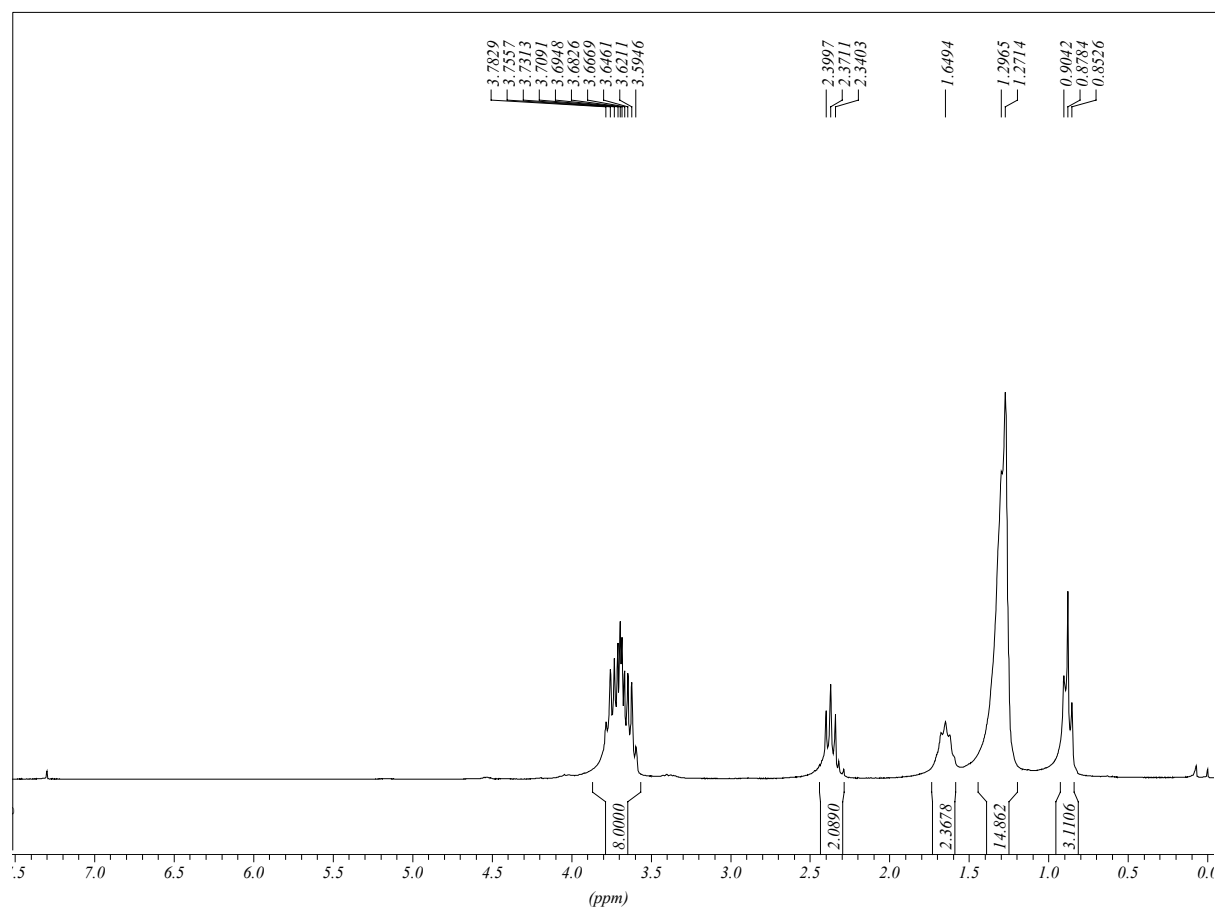
▪ N,N-bis(2-cloroetil)decanamida (15)



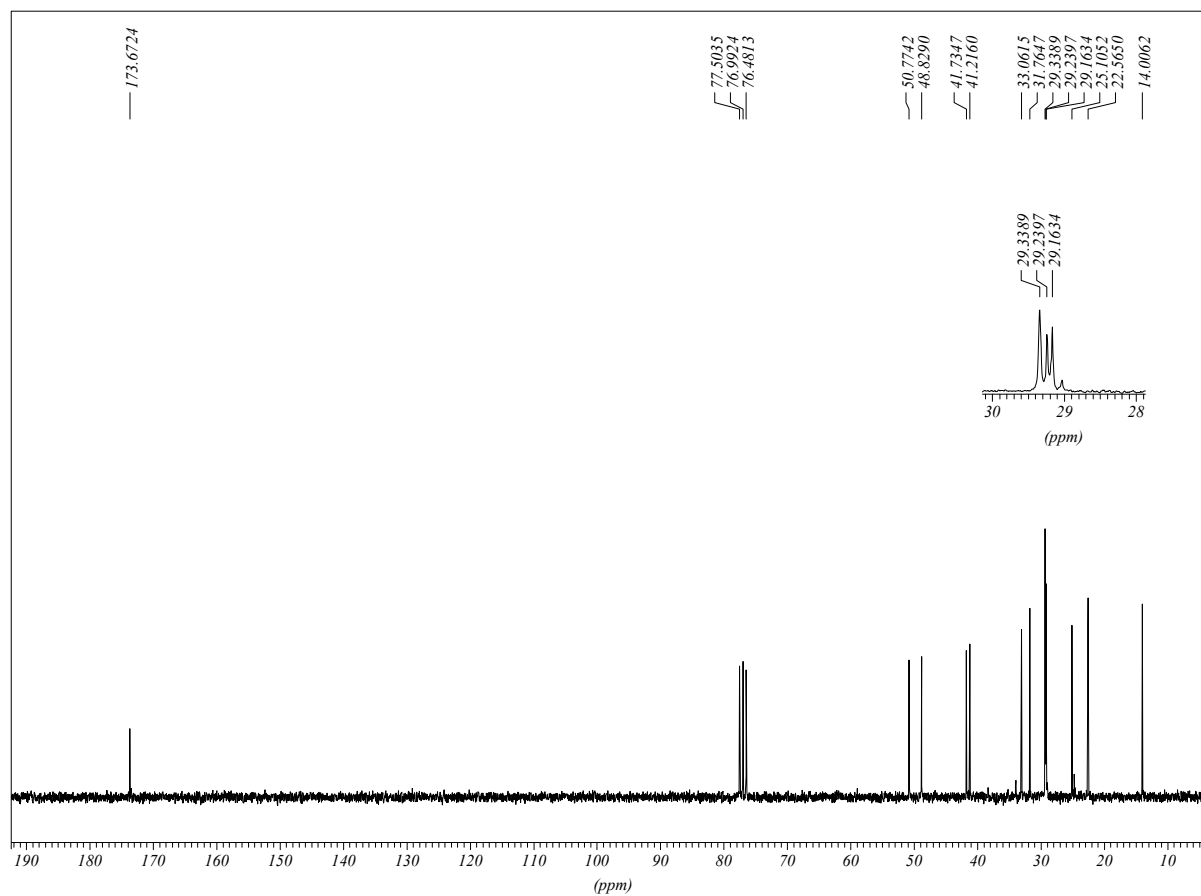
IR (KBr)



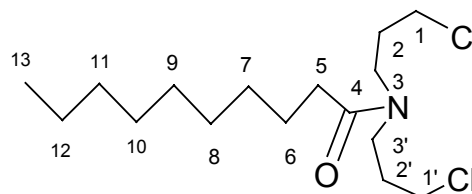
¹H RMN (CDCl₃)



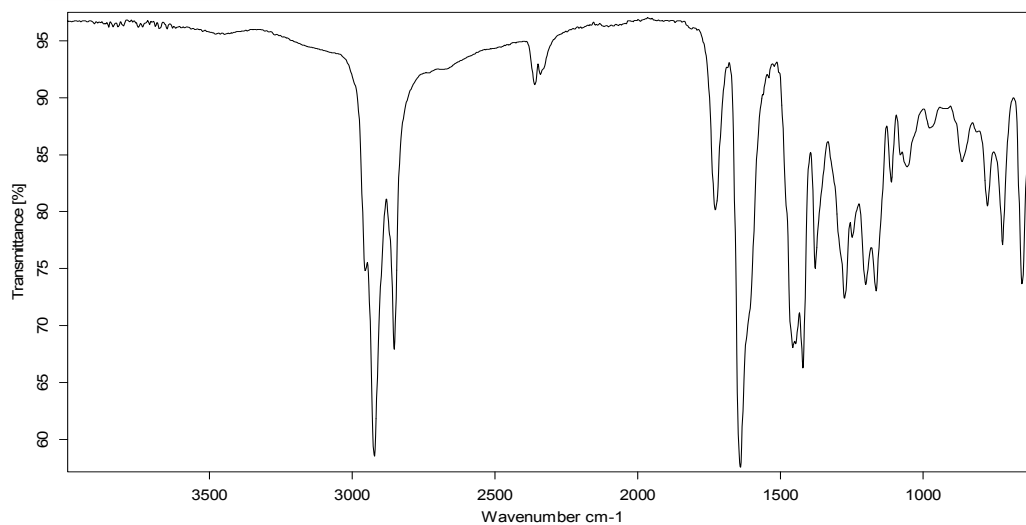
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



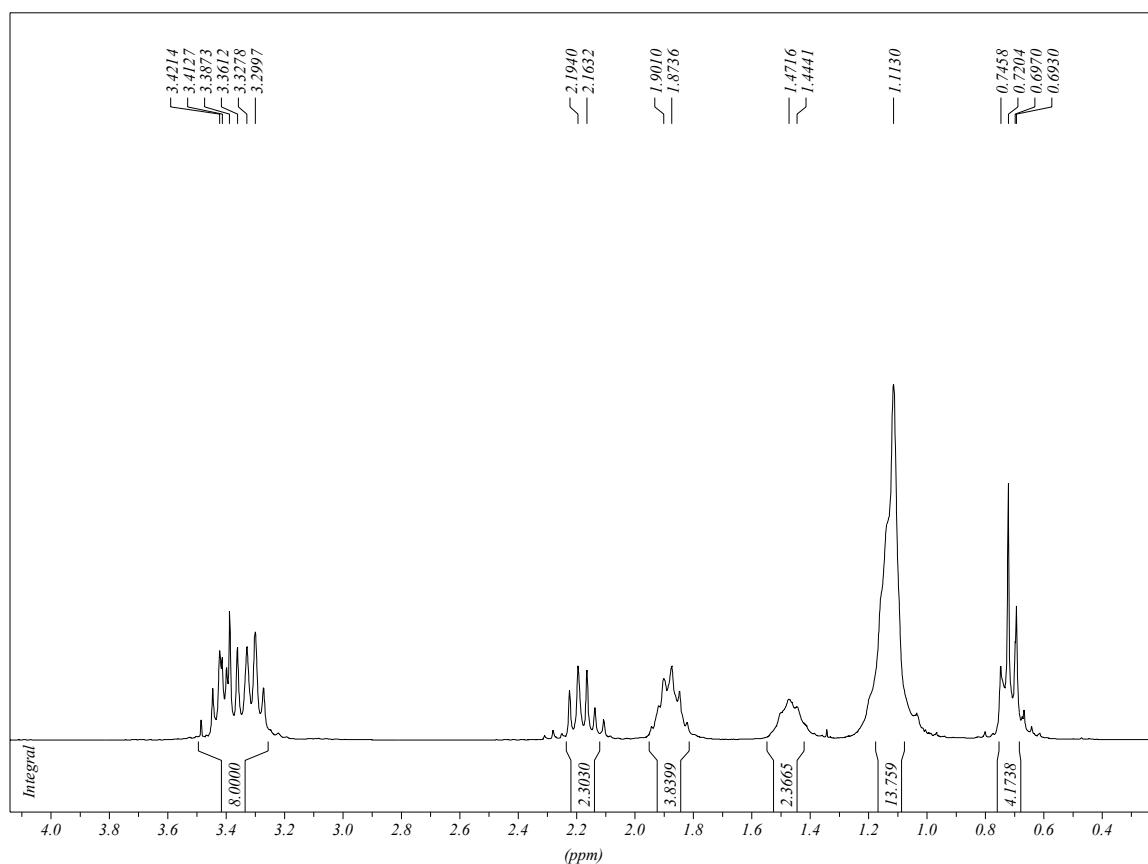
▪ **N,N-bis(2-cloropropil)decanamida (16)**



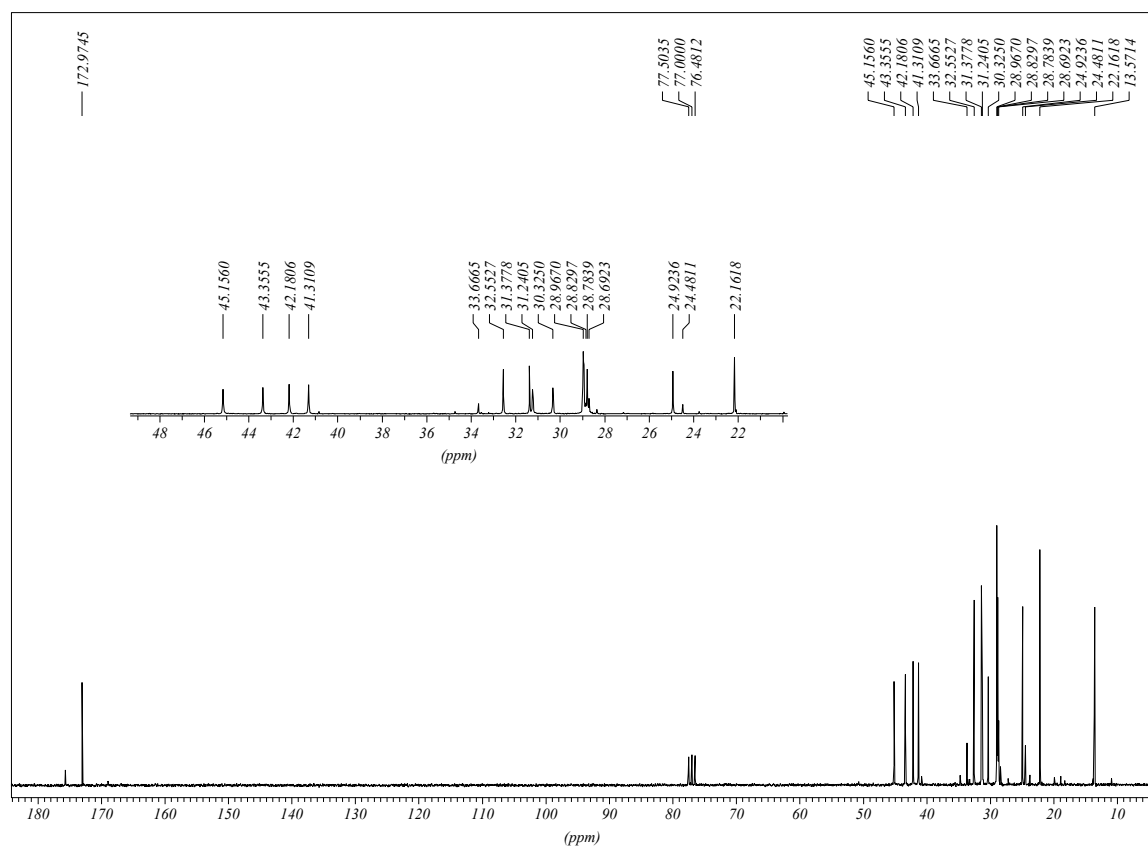
IR (ATR)



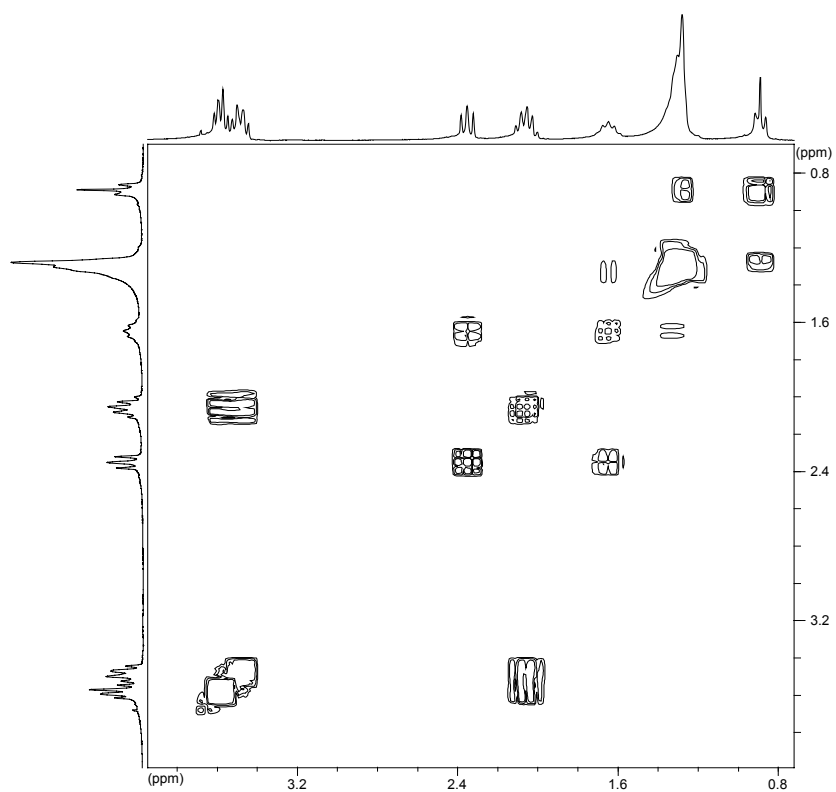
^1H RMN (CDCl_3)



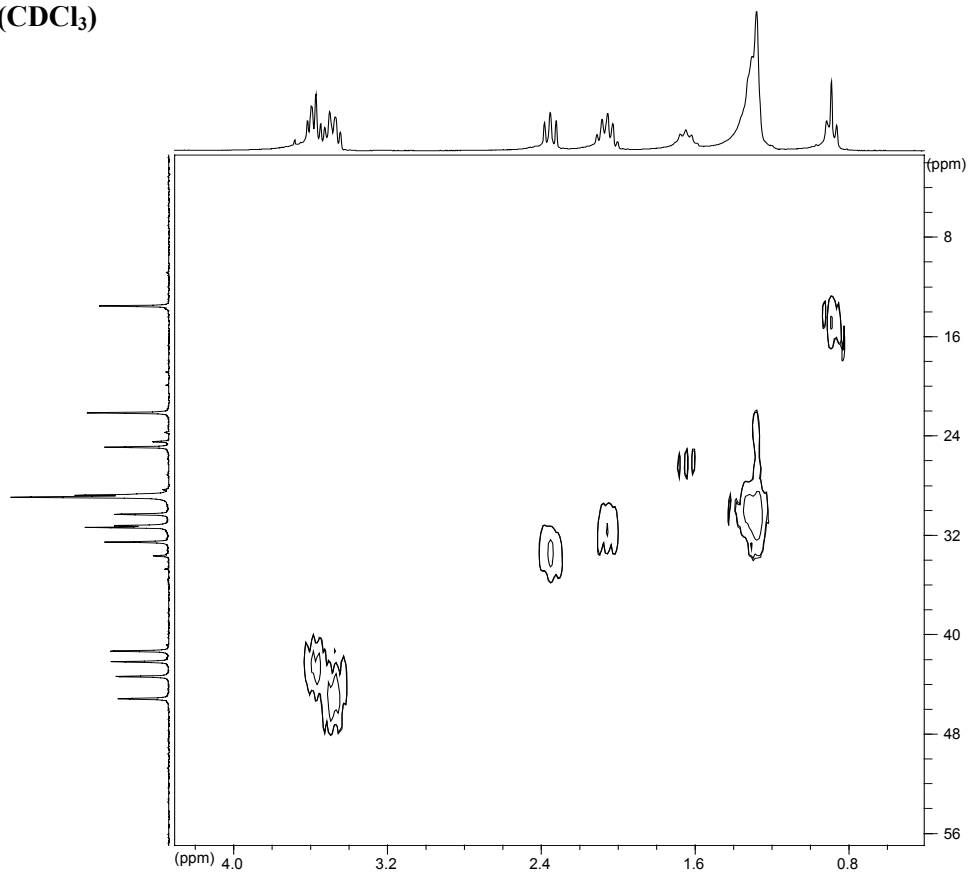
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



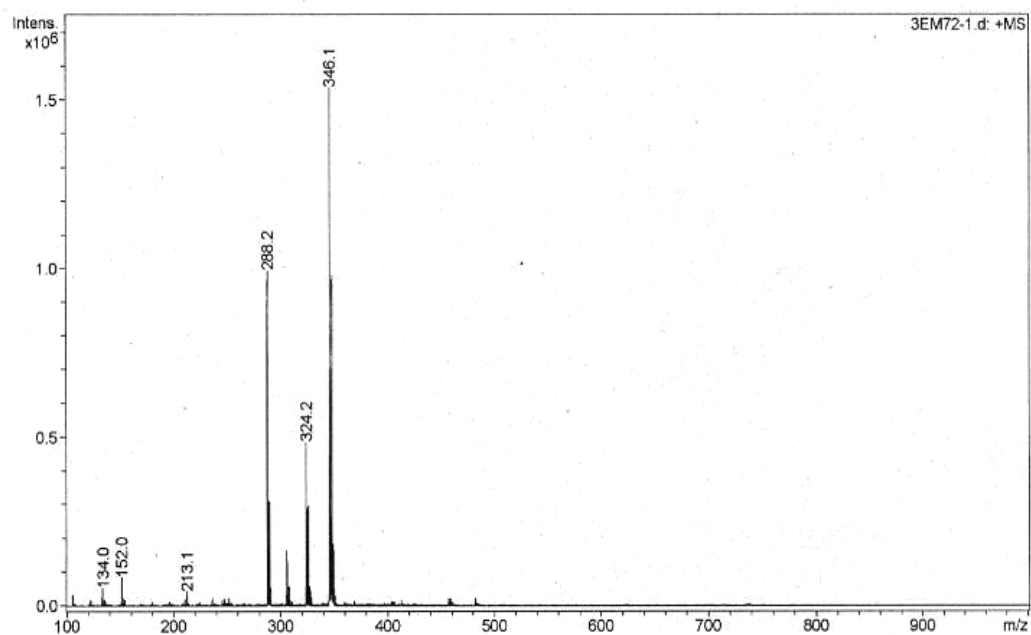
COSY (CDCl₃)



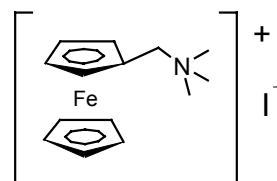
HMQC (CDCl₃)



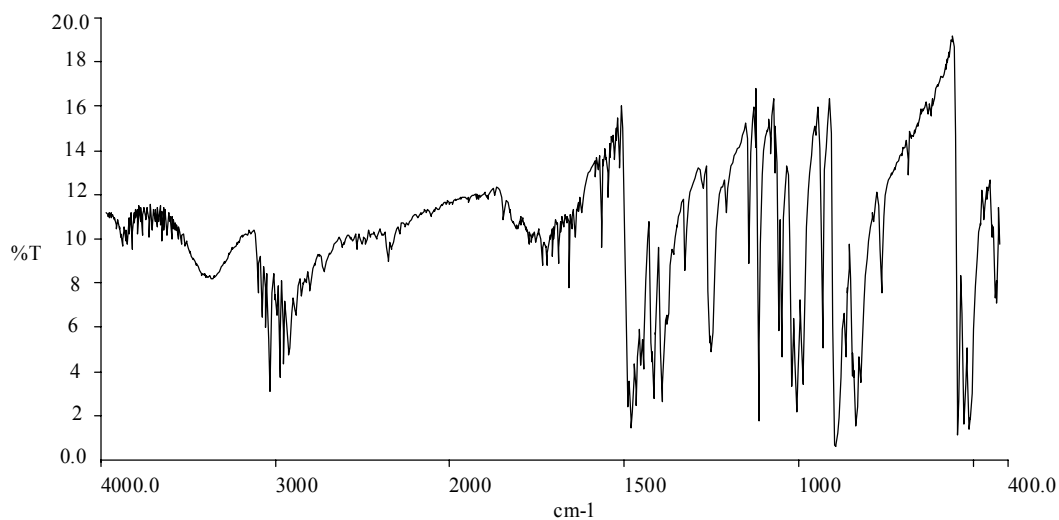
ESPECTROSCÒPIA DE MASSES (ESI(+)-IT) (10 ppm en MeOH)



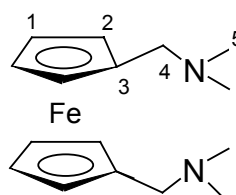
▪ **iodur de (ferrocenilmetil)trimetilamoni (17)**



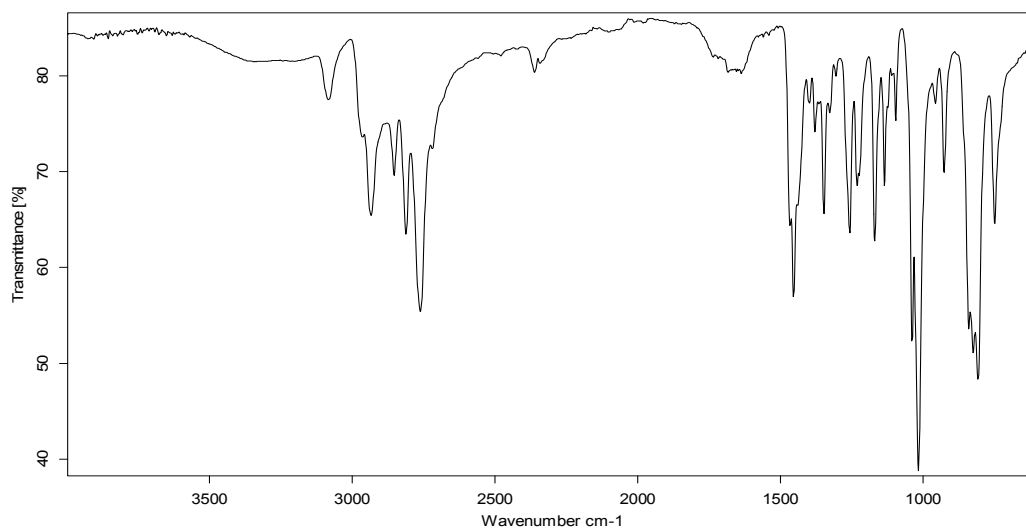
IR (KBr)



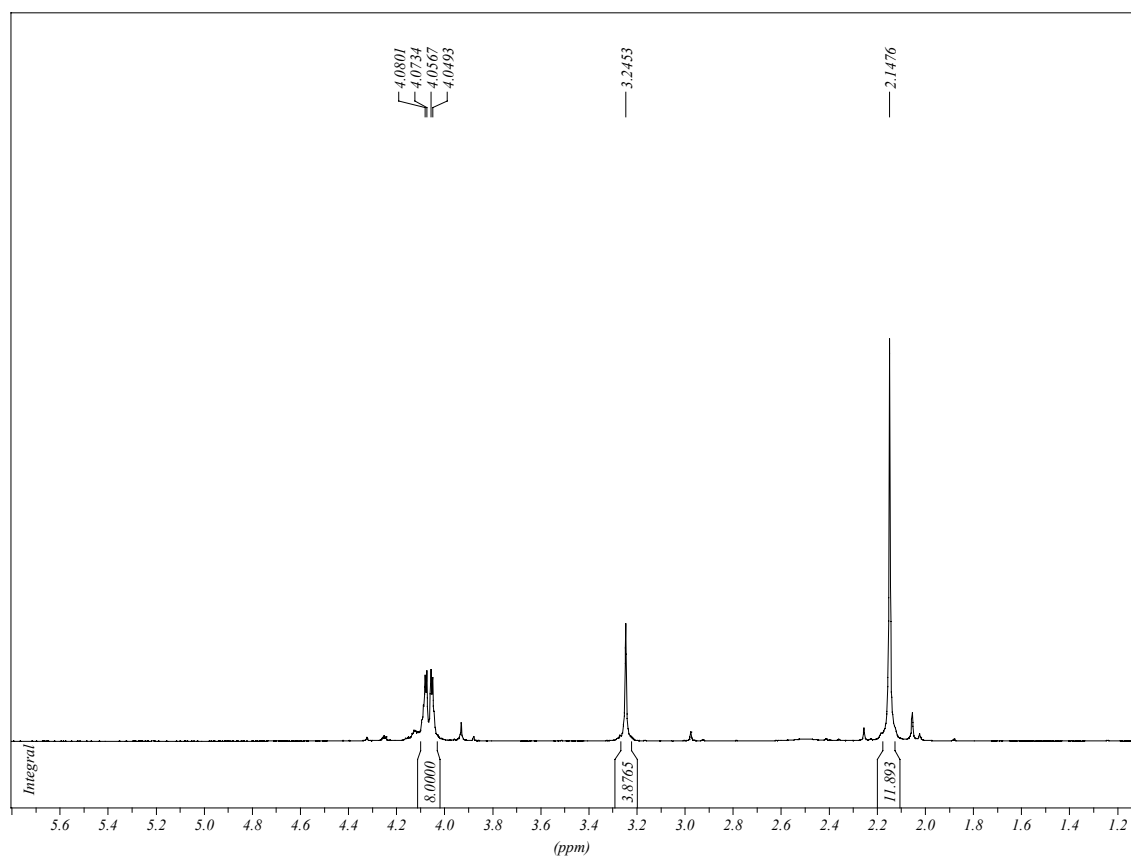
▪ 1,1'-bis(N,N-dimetilaminometil)ferrocè (18)



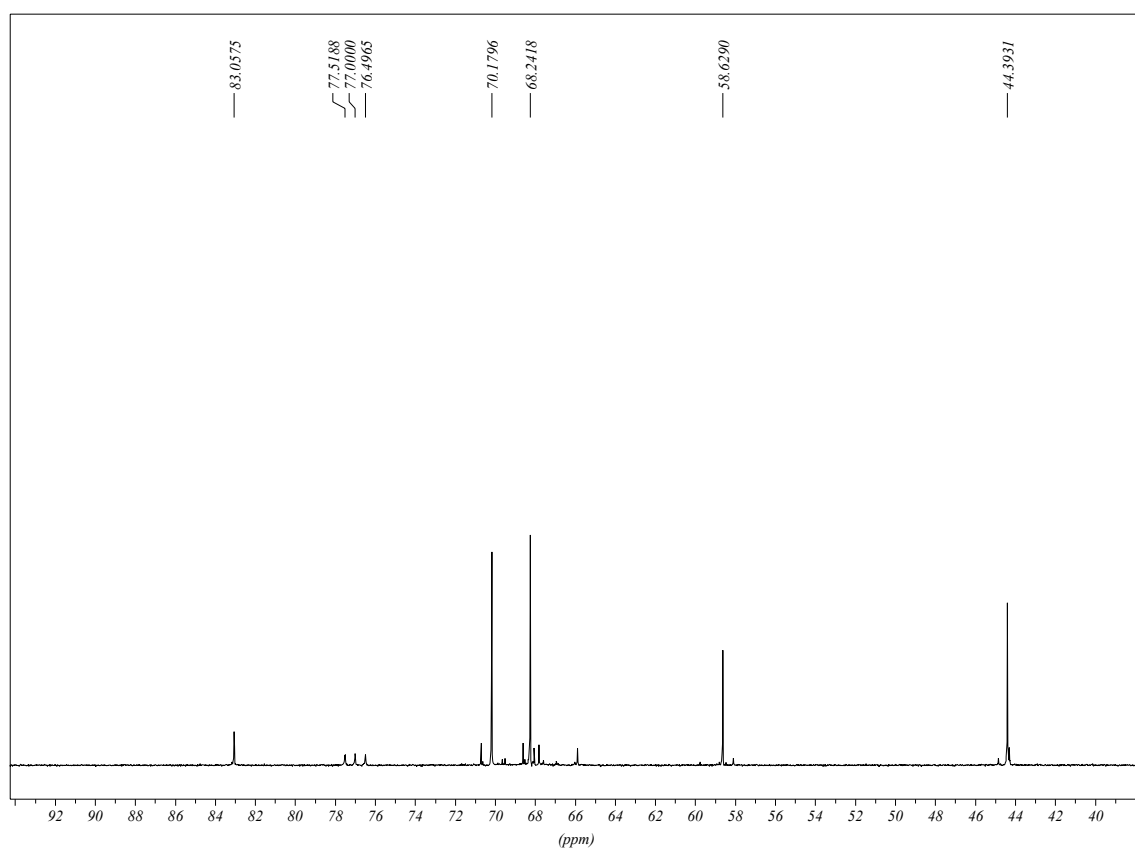
IR (ATR)



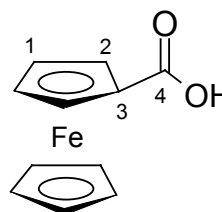
¹H RMN (CDCl₃)



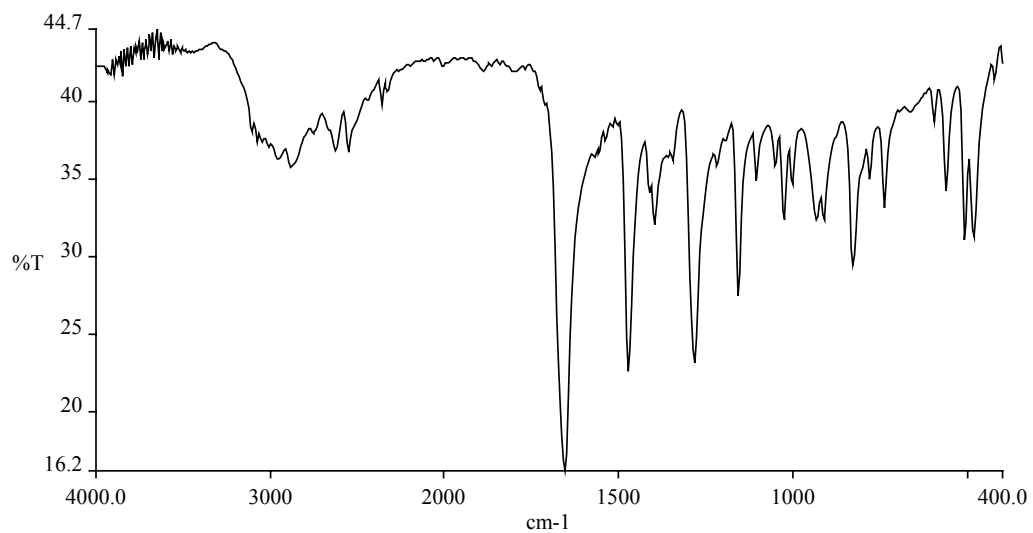
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



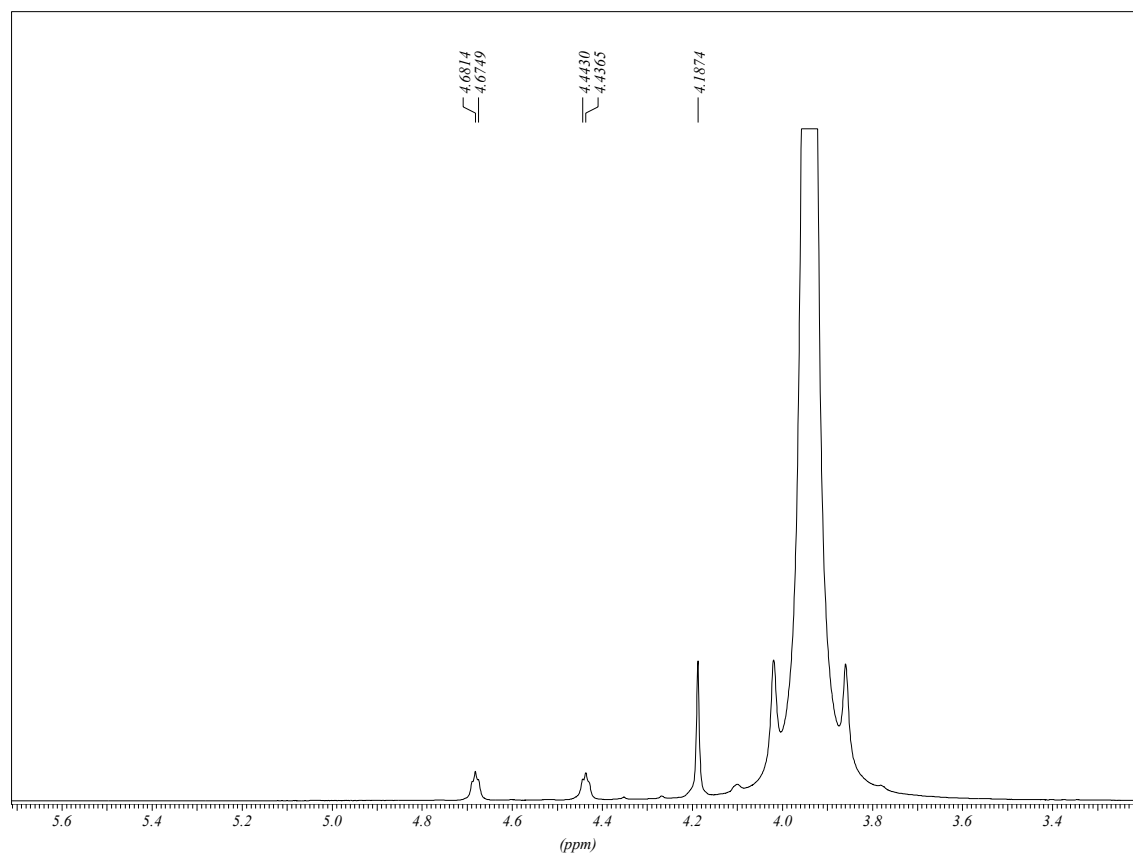
▪ àcid ferrocenoic (20)



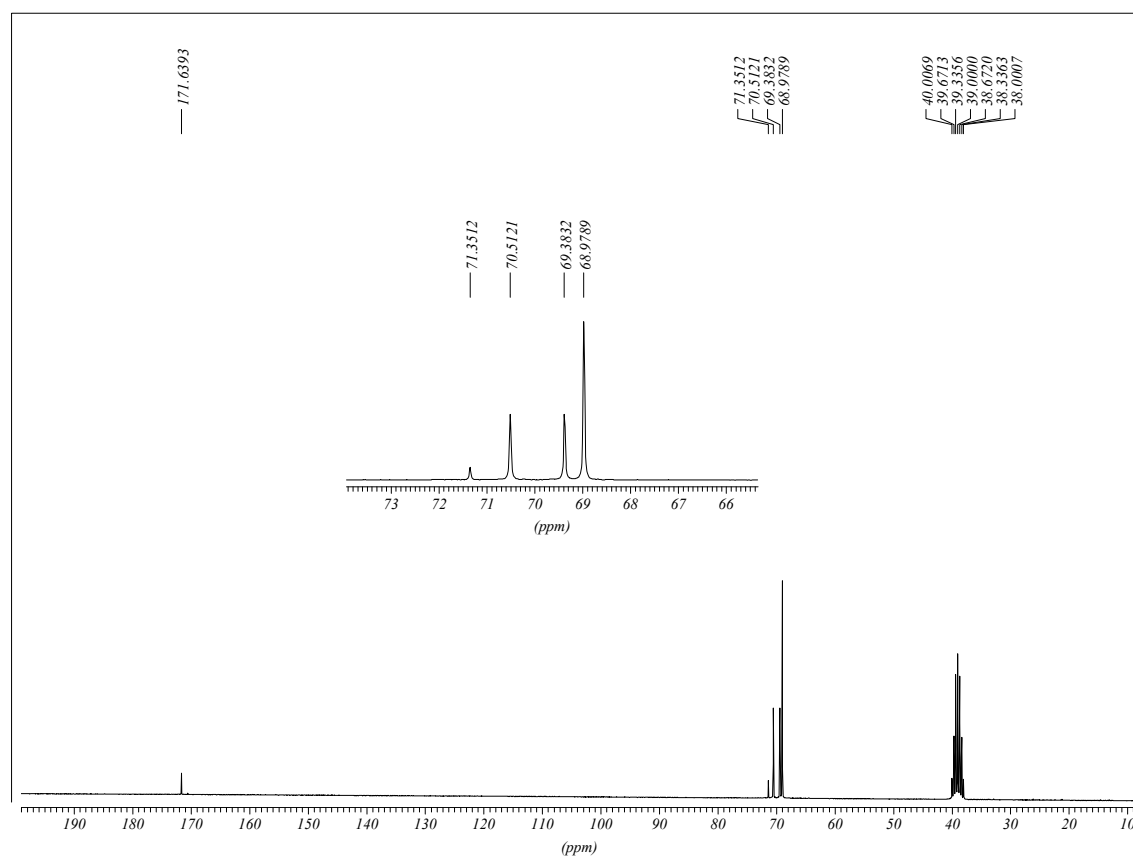
IR (KBr)



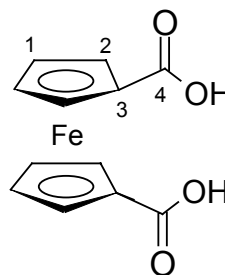
^1H RMN (DMSO- d_6)



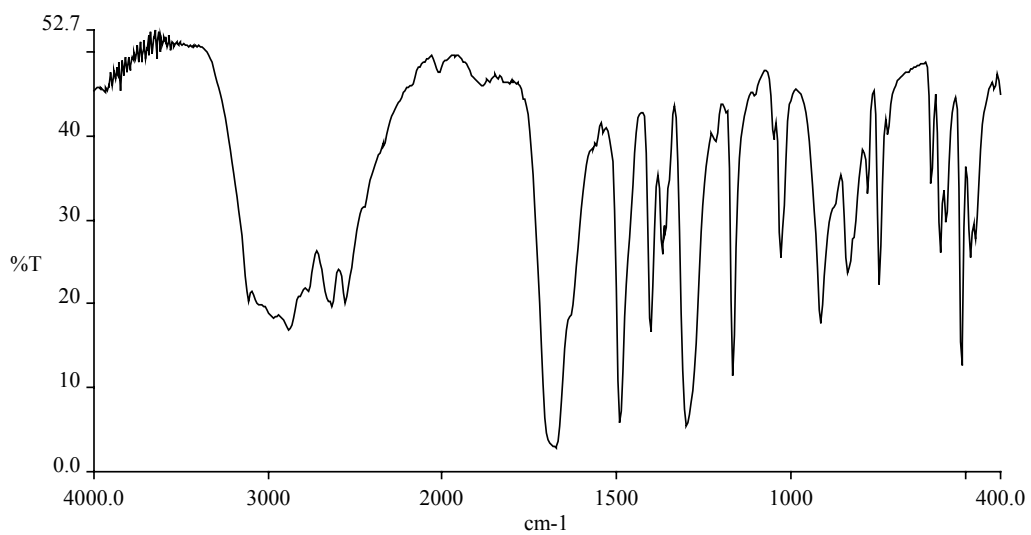
$^{13}\text{C}\{^1\text{H}\}$ RMN (DMSO- d_6)



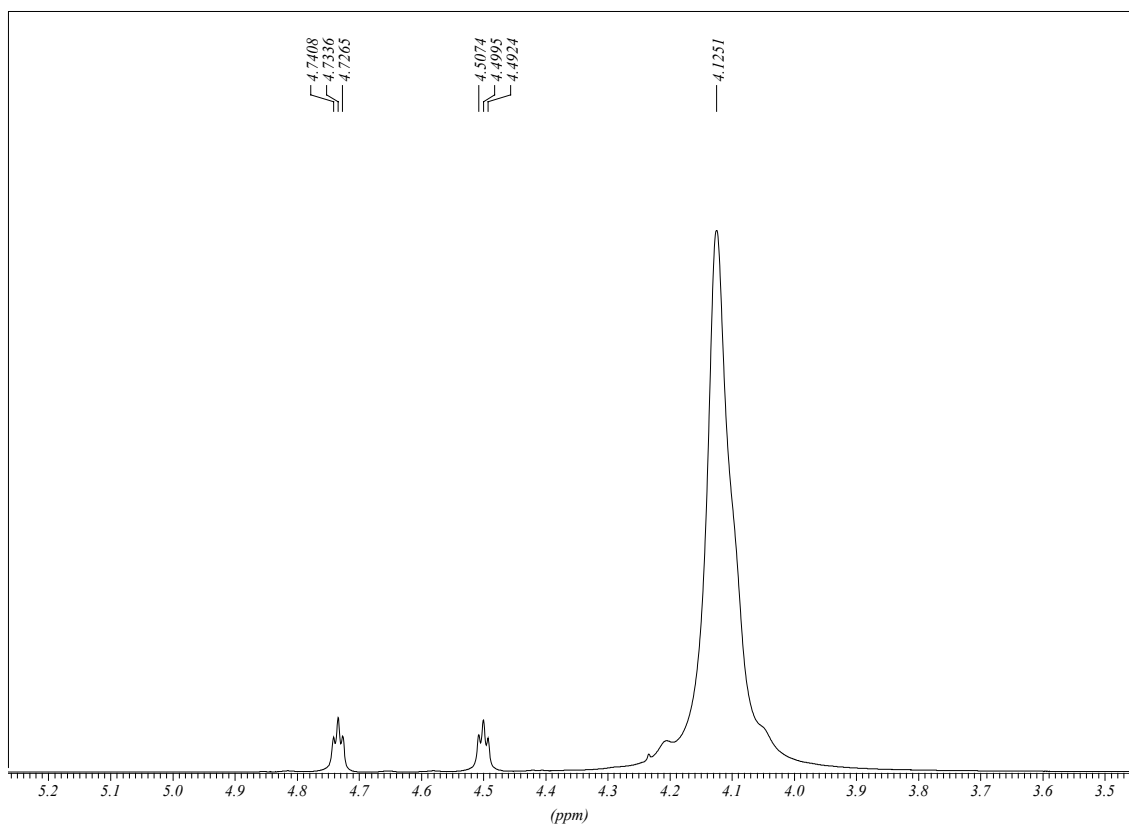
▪ àcid 1,1'-ferrocedicarboxílic (21)



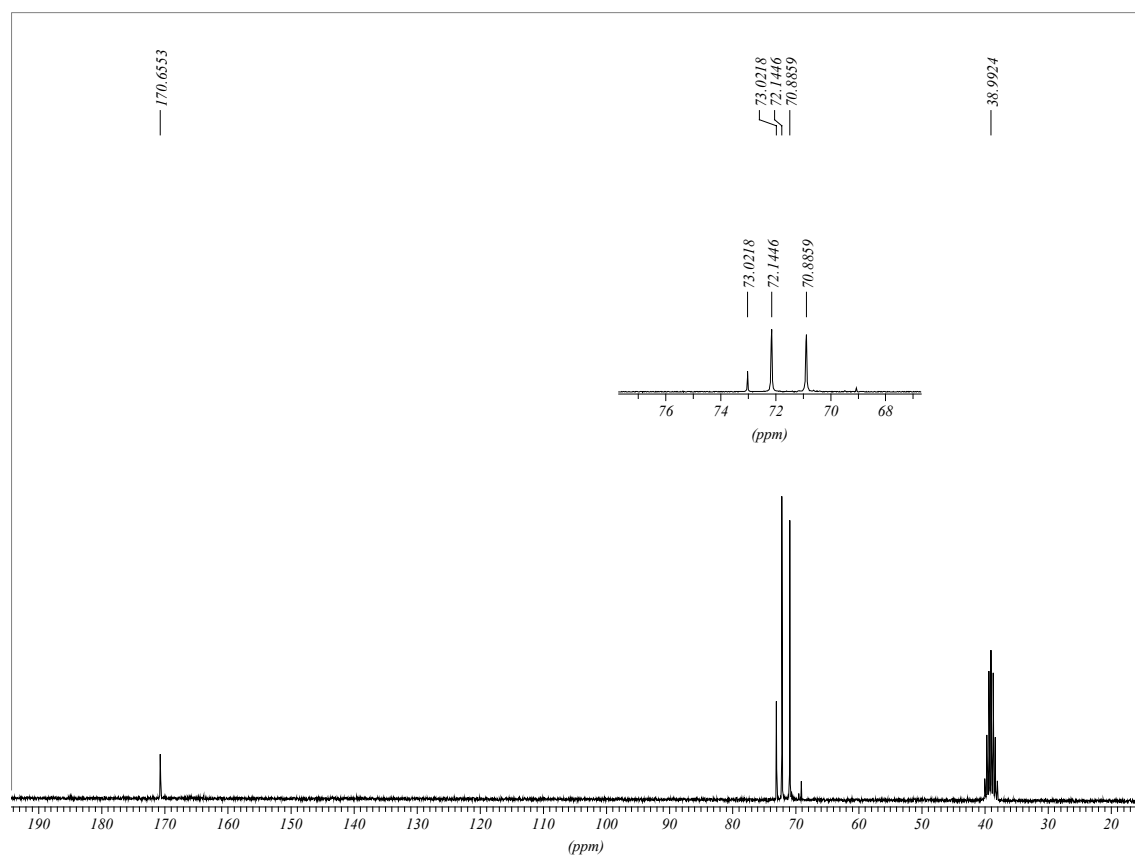
IR (KBr)



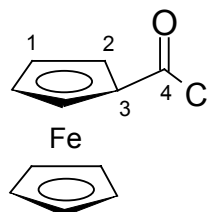
¹H RMN (DMSO-d₆)



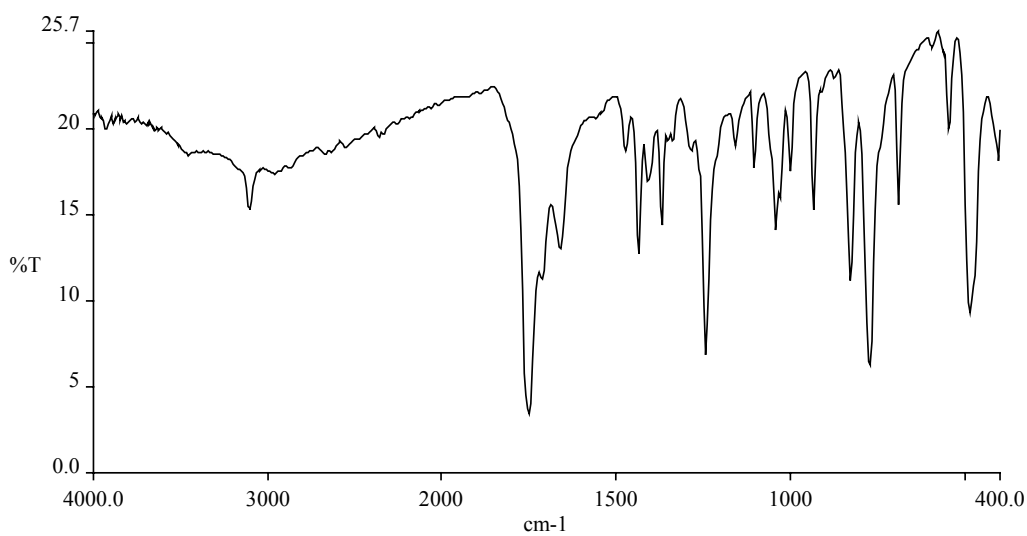
$^{13}\text{C}\{^1\text{H}\}$ RMN (DMSO- d_6)



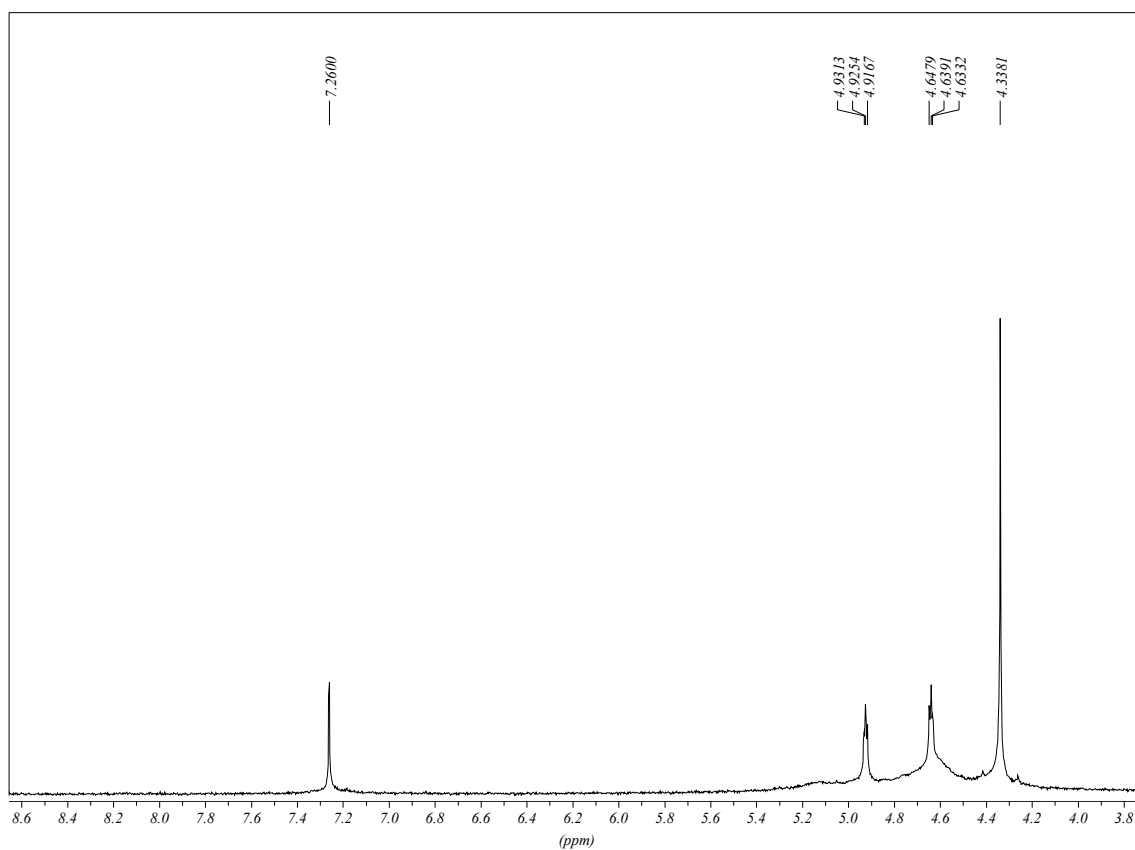
▪ (clorocarbonil)ferrocè (22)



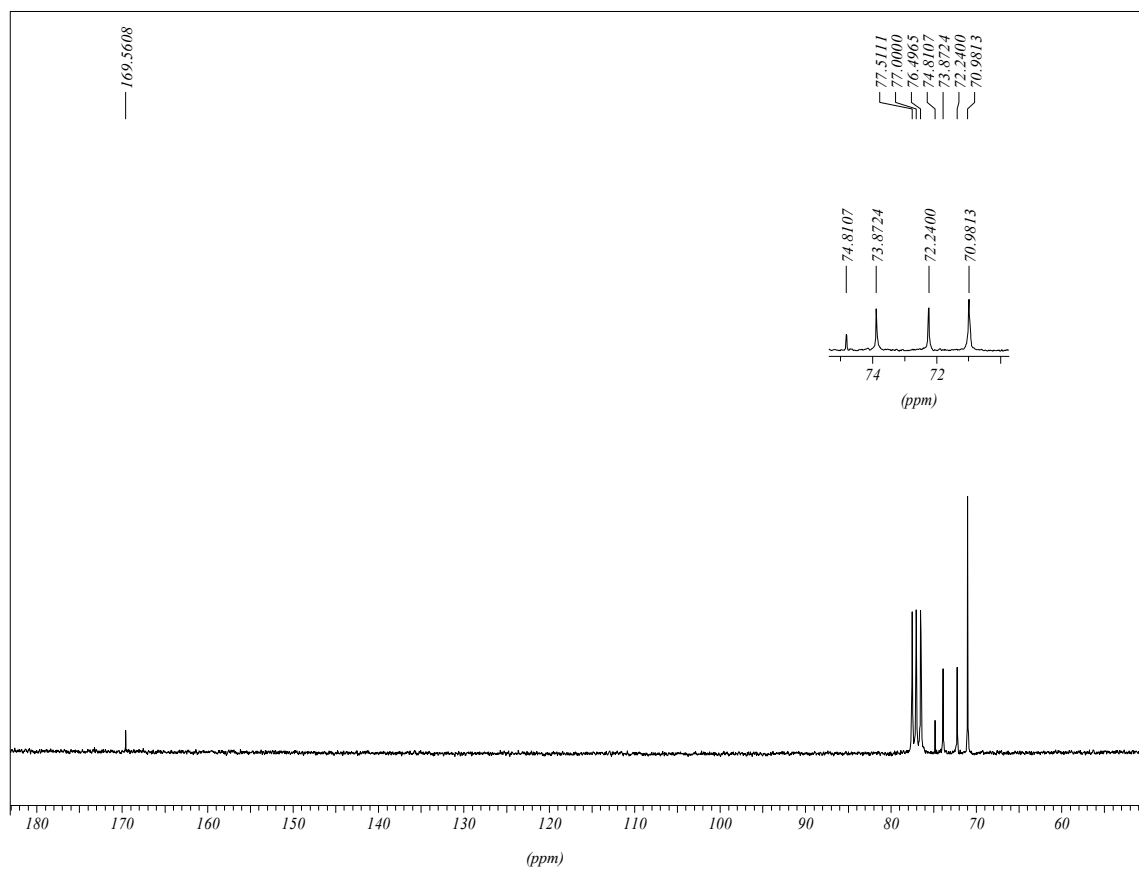
IR (KBr)



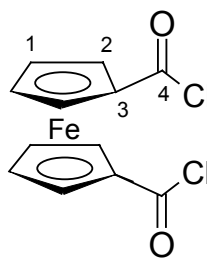
^1H RMN (CDCl_3)



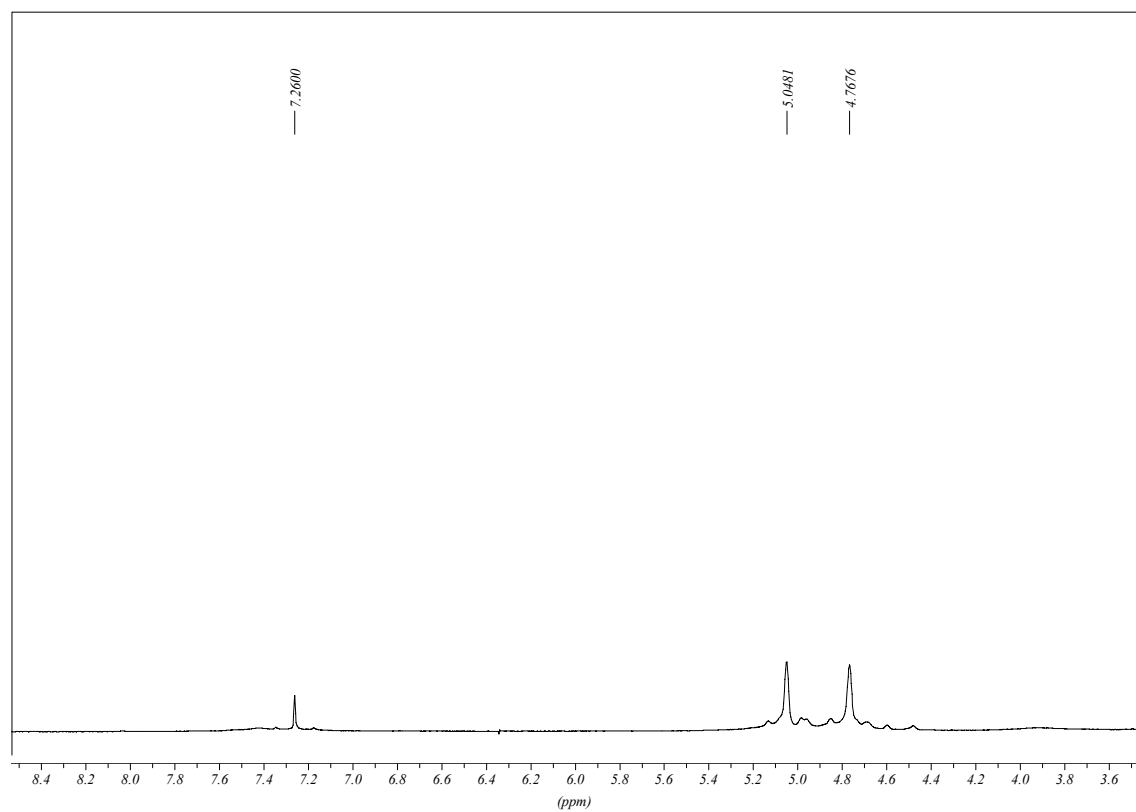
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



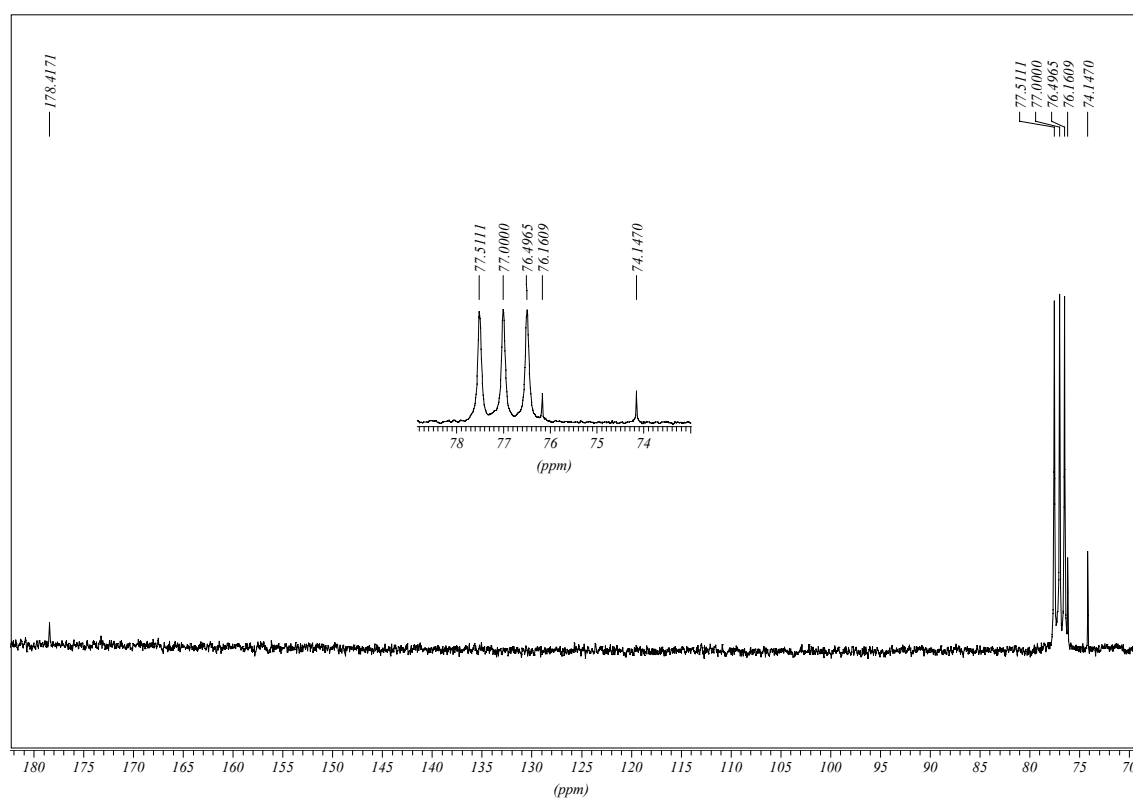
▪ 1,1'-bis(clorocarbonil)ferrocè (23)



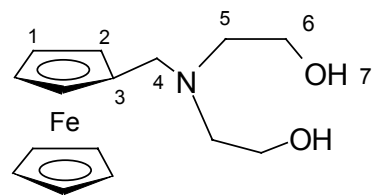
^1H RMN (CDCl_3)



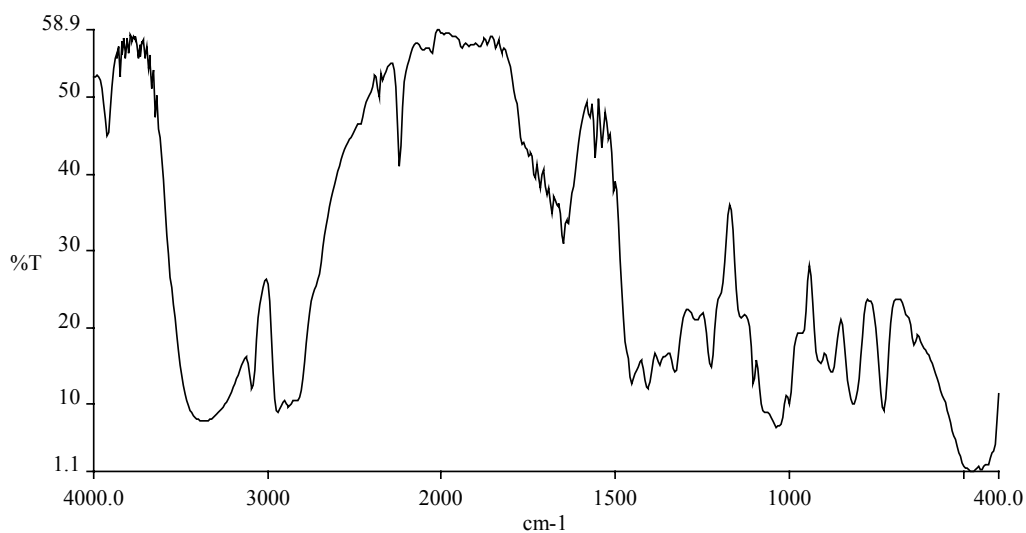
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



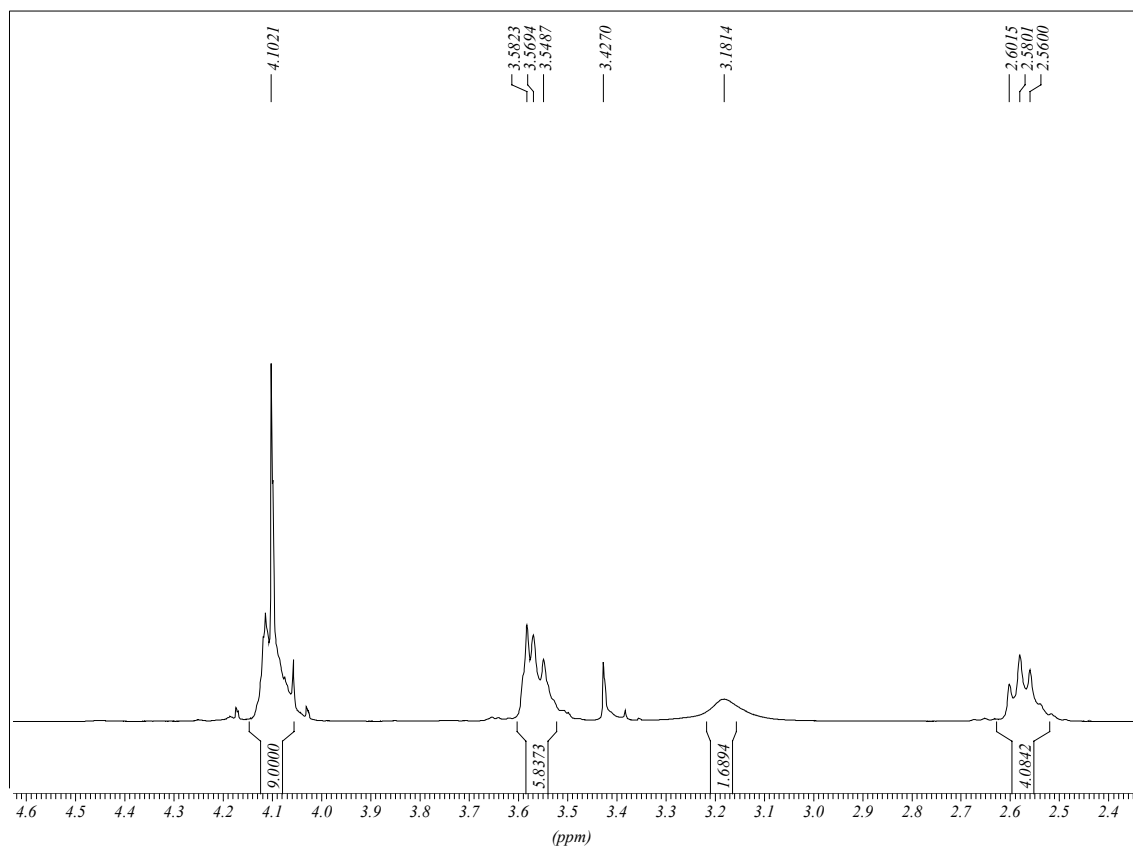
▪ N,N-bis(2-hidroxietil)(ferrocenilmetil)amina (26)



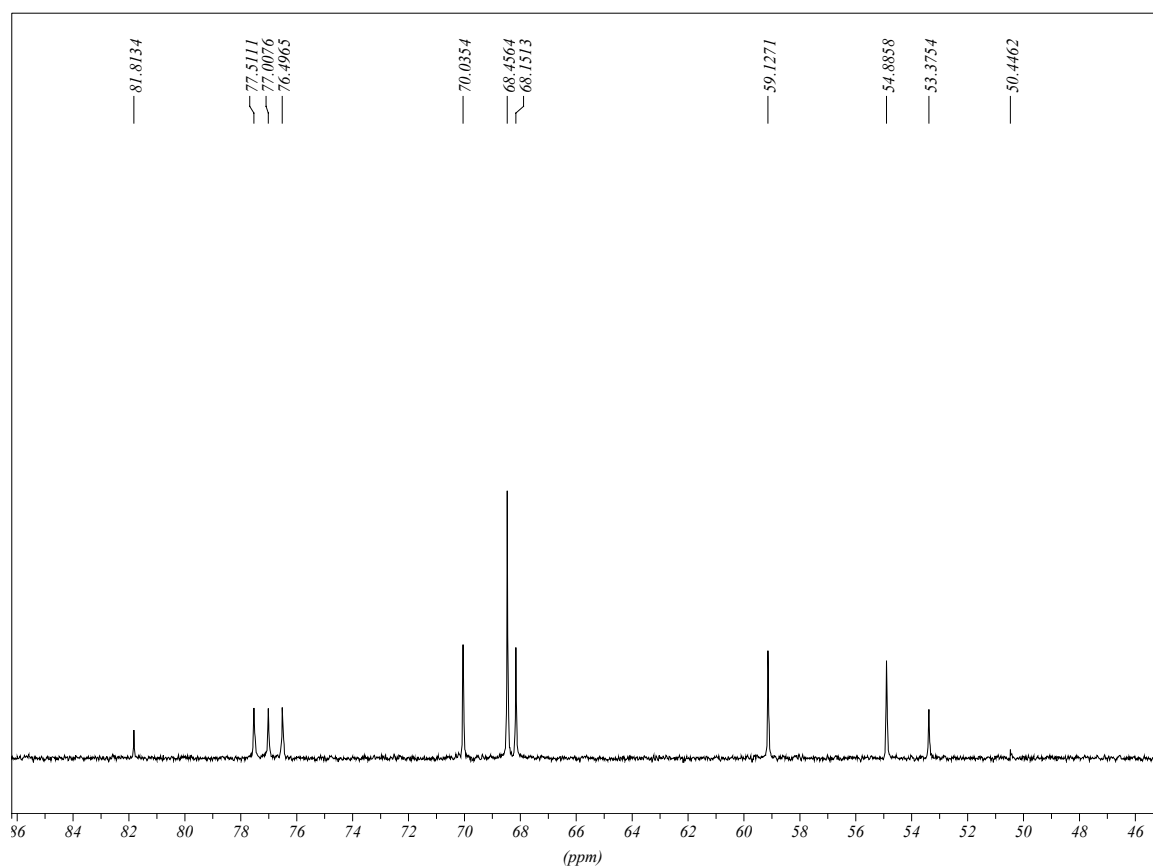
IR (KBr)



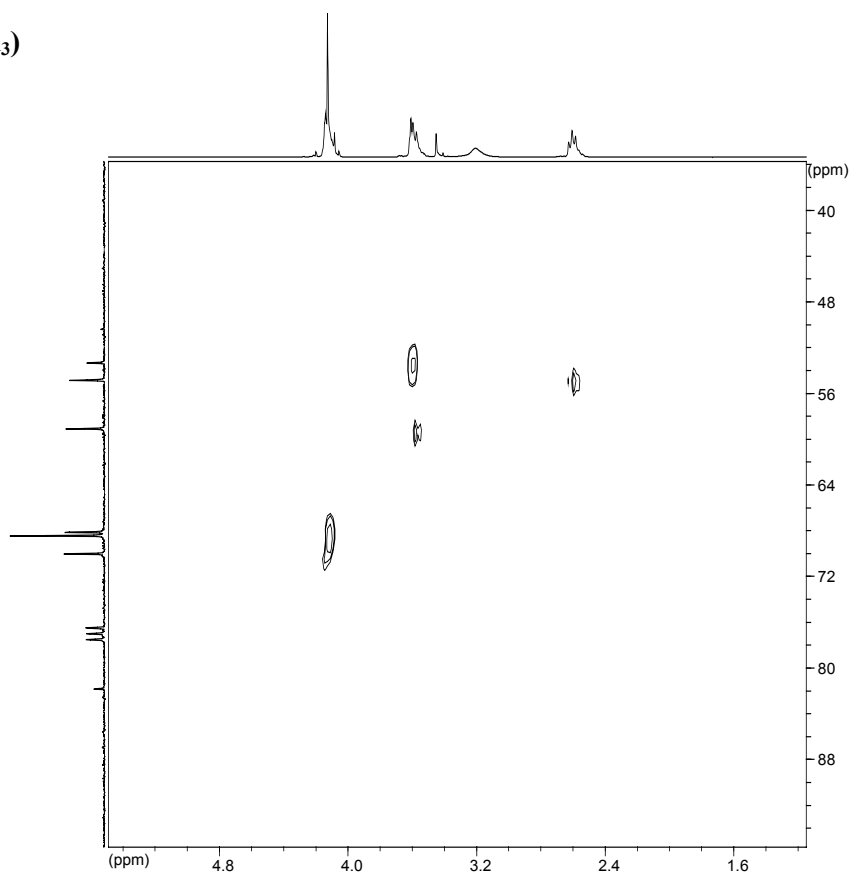
^1H RMN (CDCl_3)



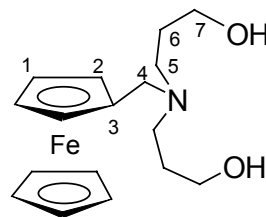
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



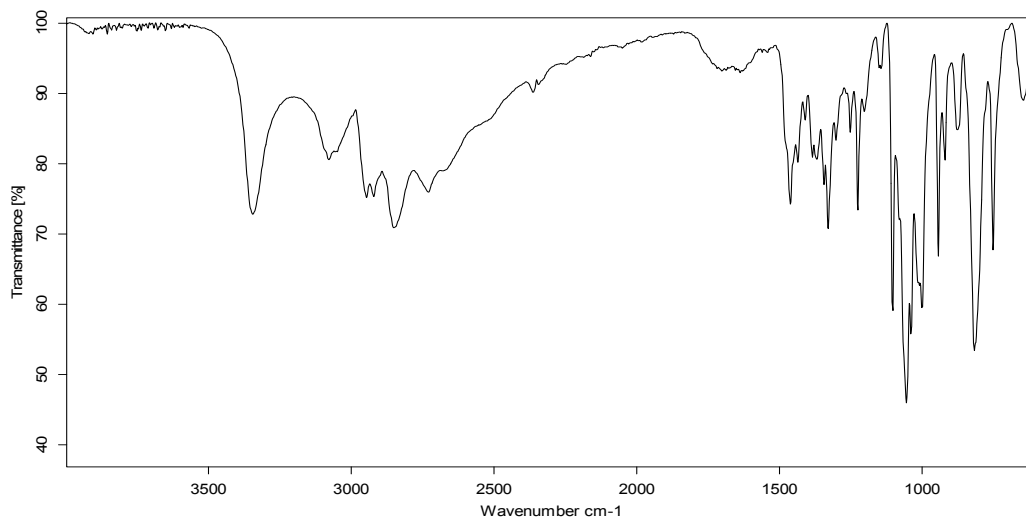
HMQC (CDCl_3)



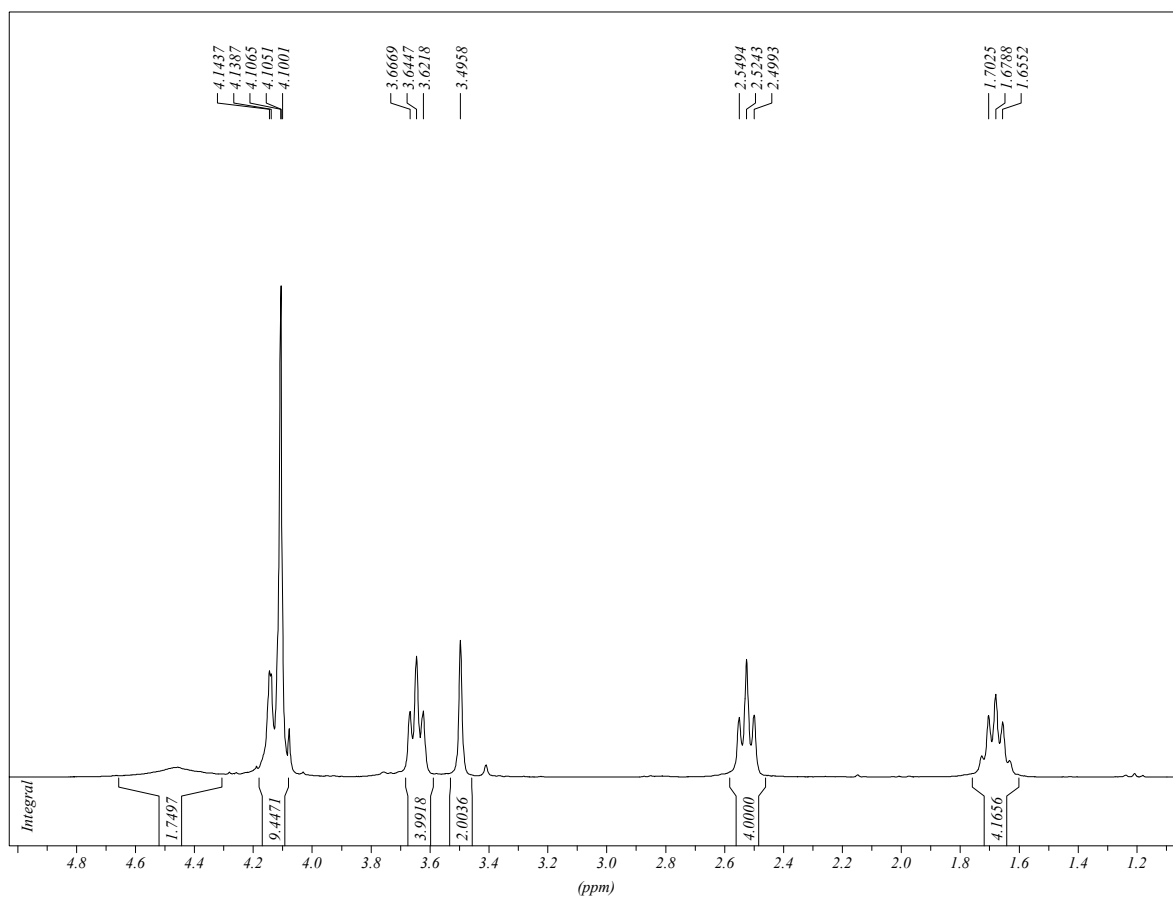
▪ N,N-bis(3-hidroxipropil)(ferrocenilmetil)amina (27)



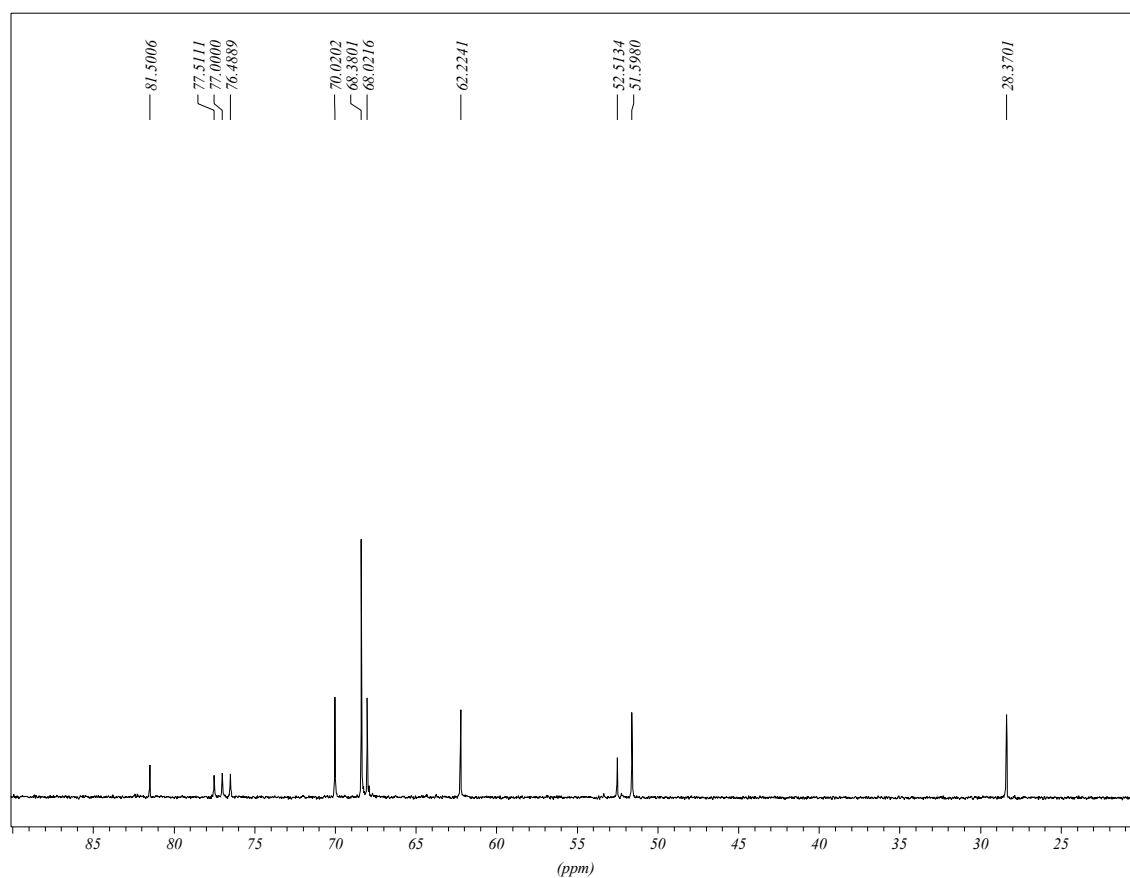
IR (ATR)



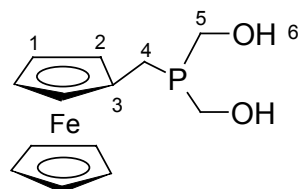
¹H RMN (CDCl₃)



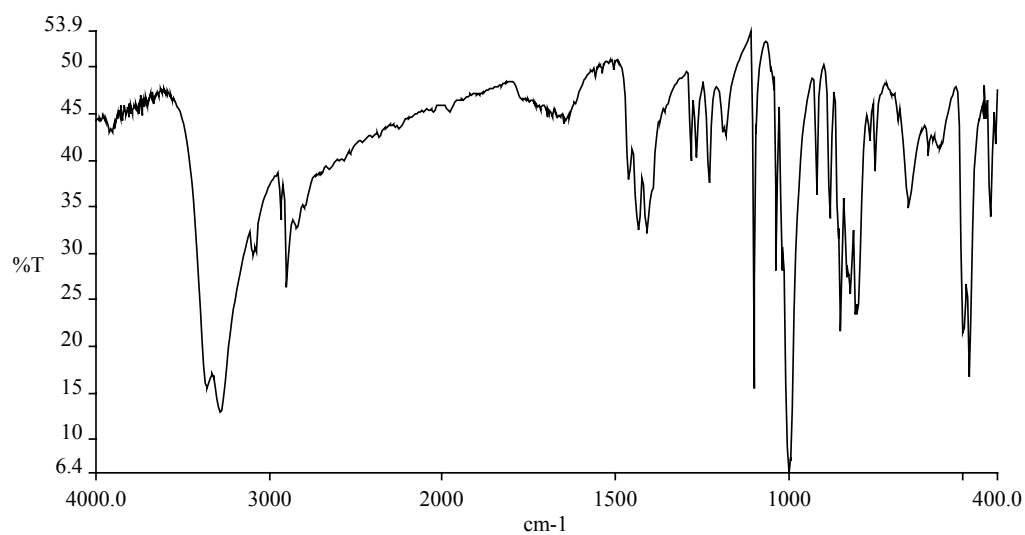
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



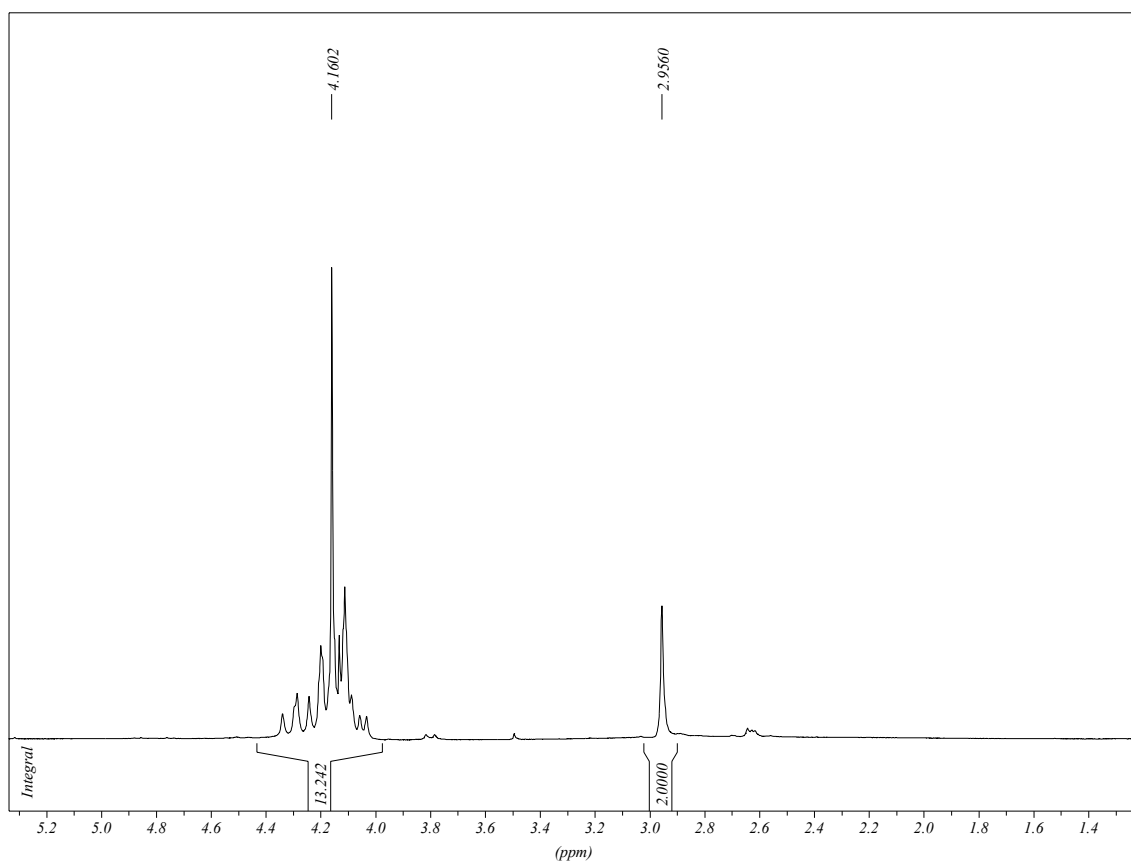
▪ (ferrocenilmetil)bis(hidroximetil)fosfà (28)



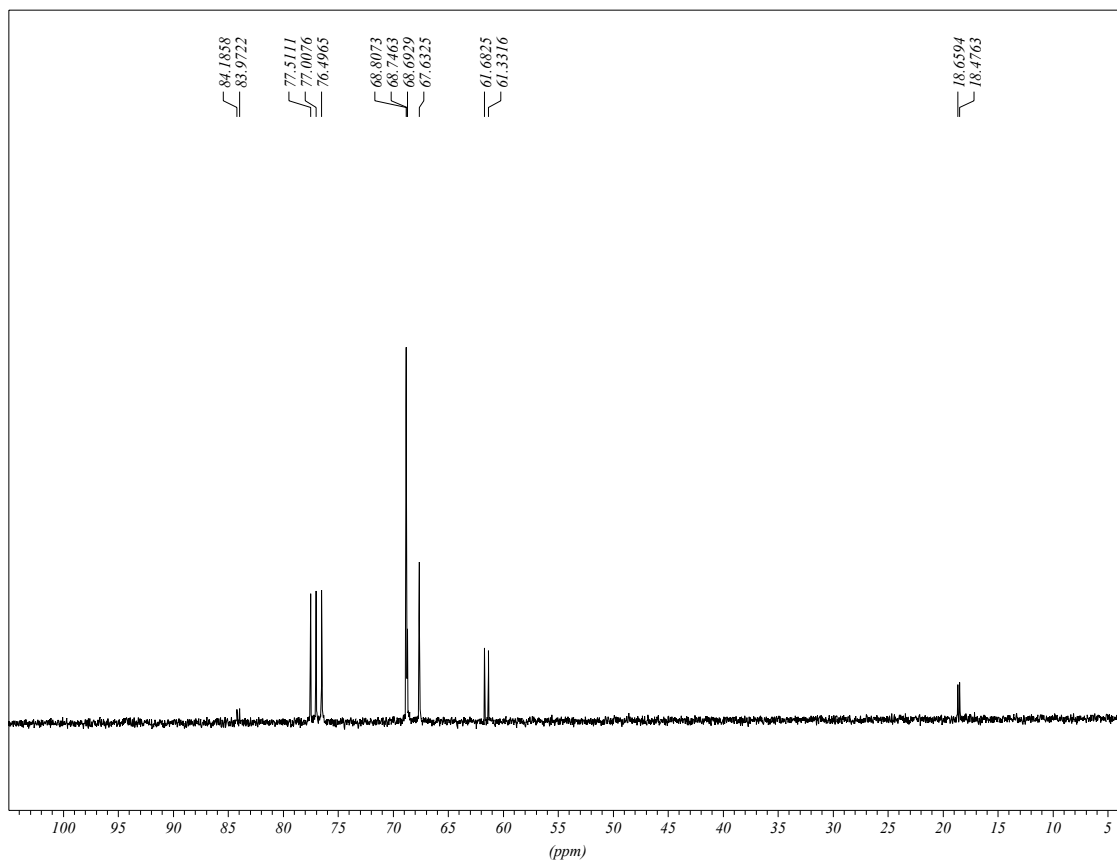
IR (ATR)



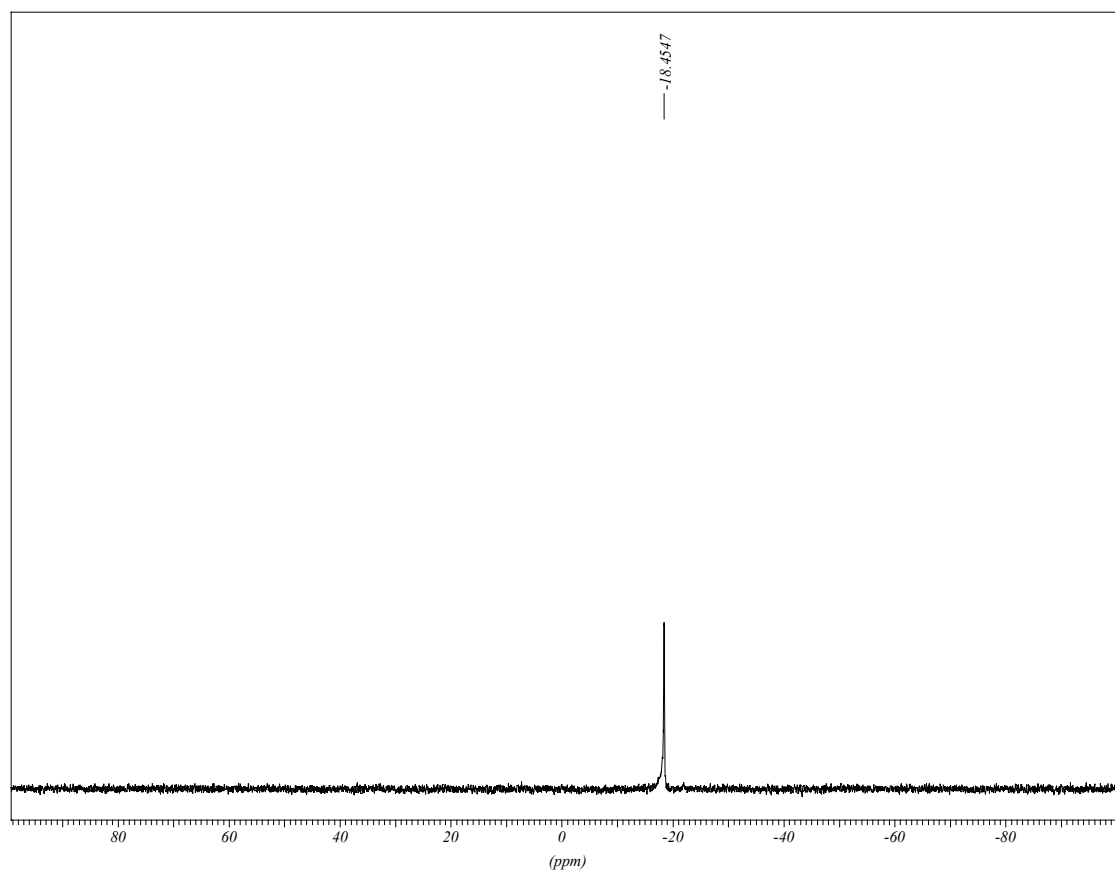
^1H RMN (CDCl_3)



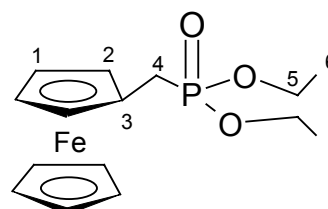
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



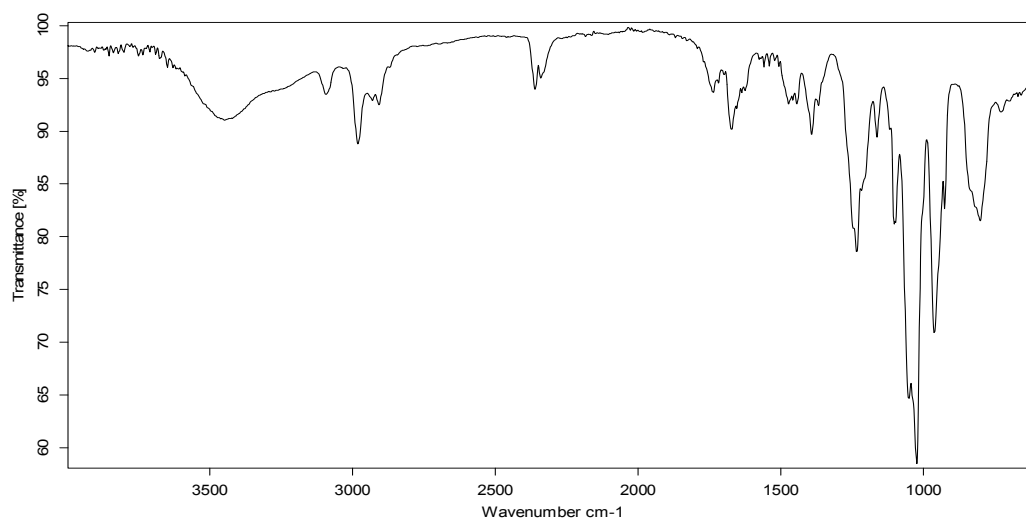
$^{31}\text{P}\{^1\text{H}\}$ RMN (CDCl_3)



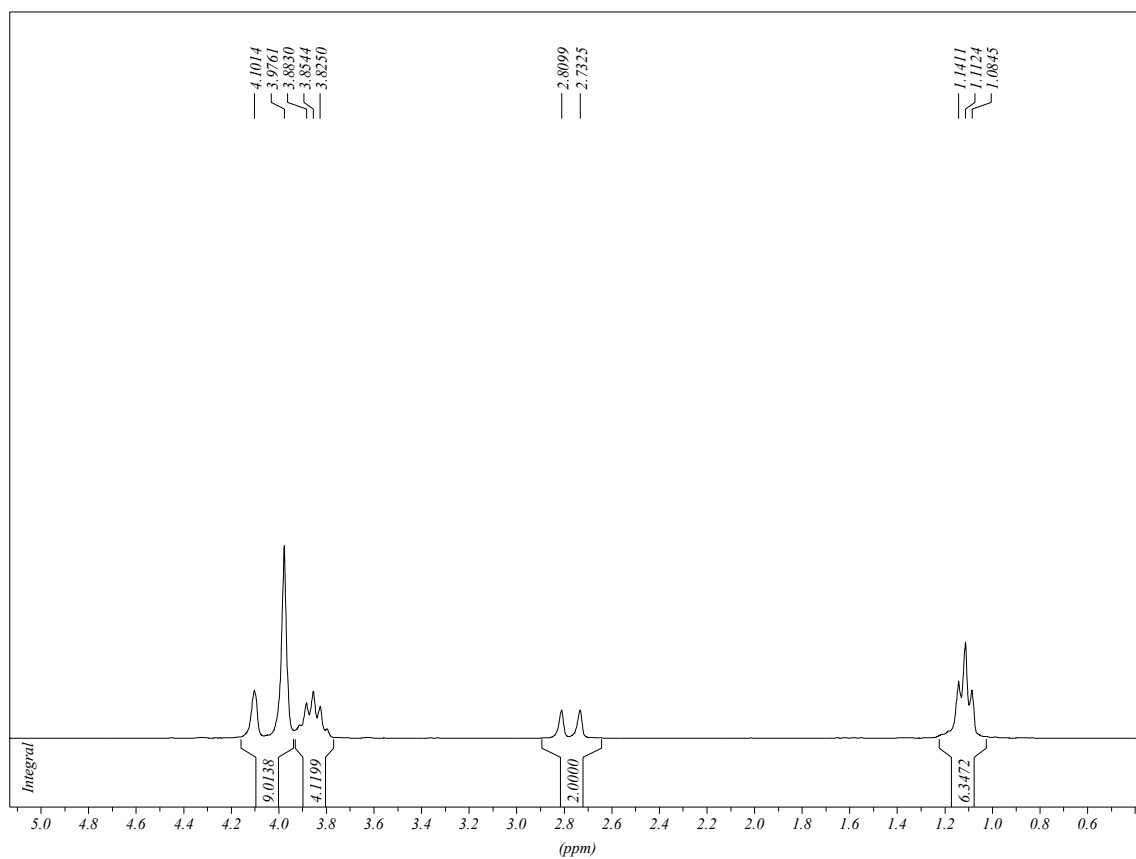
▪ dietil (ferrocenilmetil)fosfonat (31)



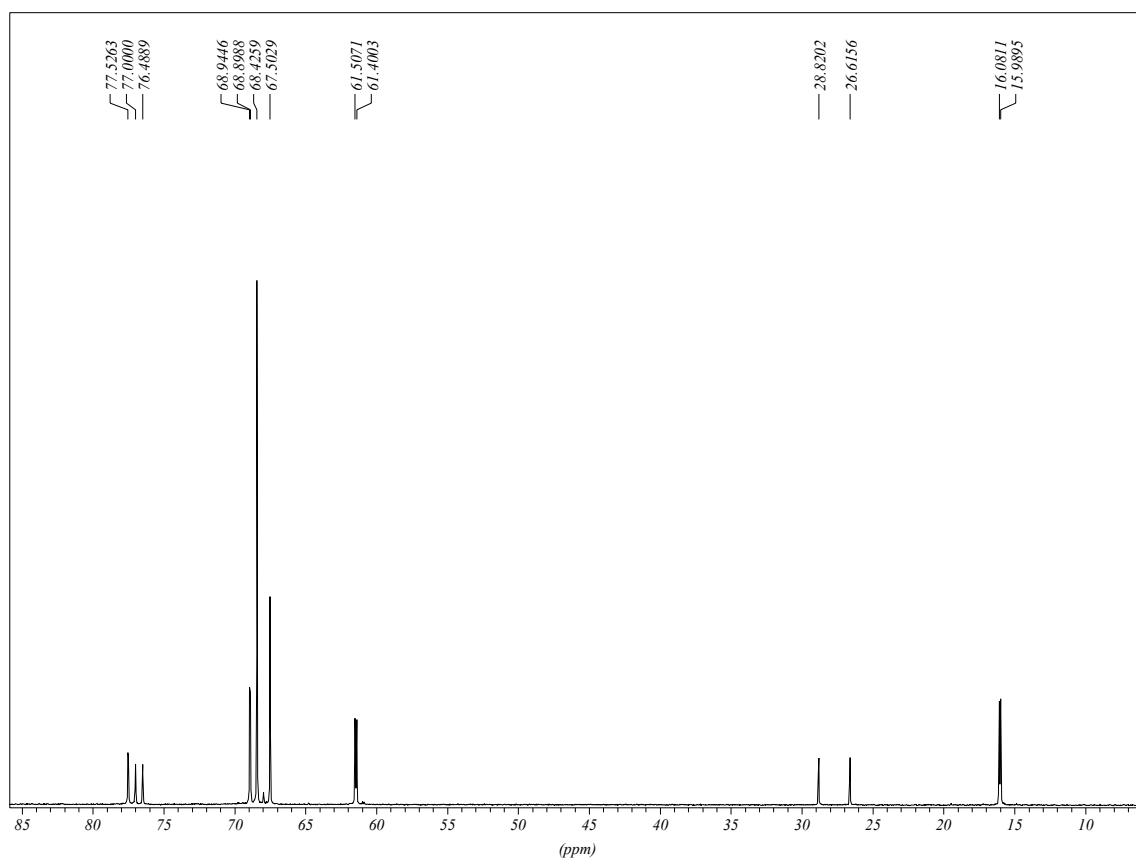
IR (ATR)



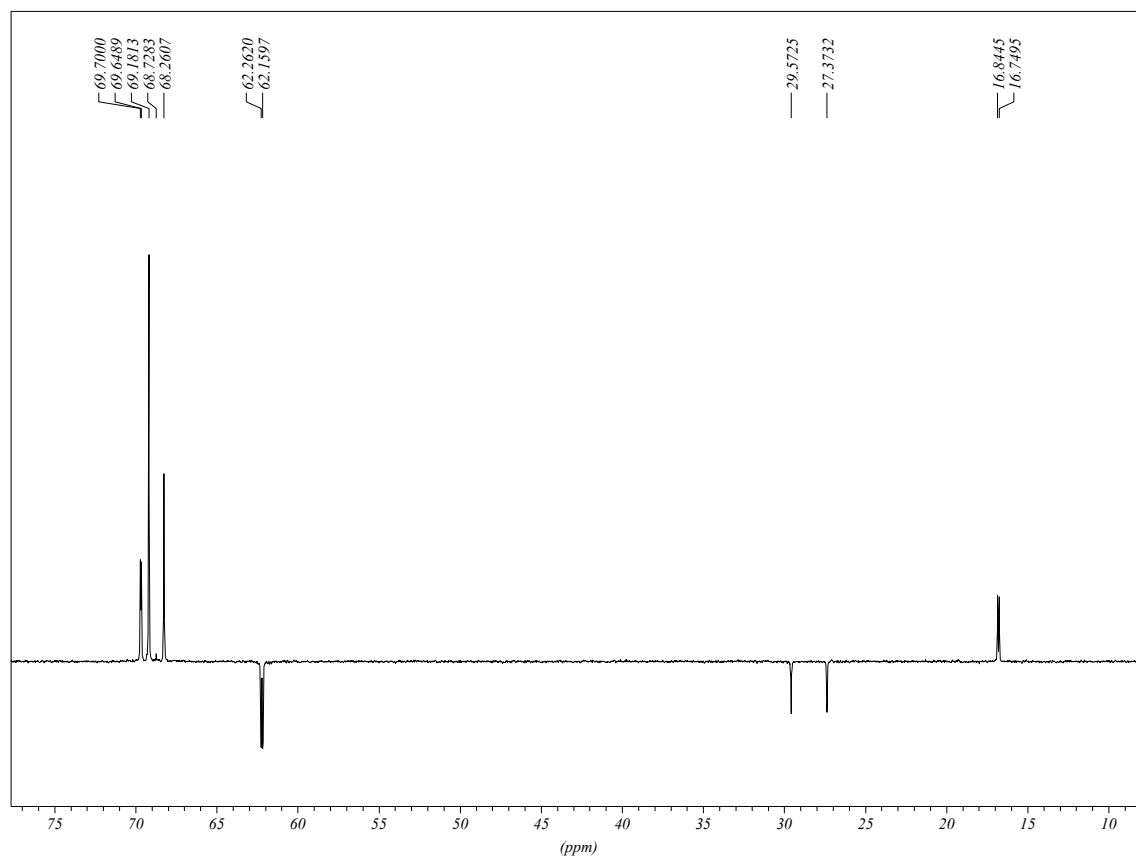
^1H RMN (CDCl_3)



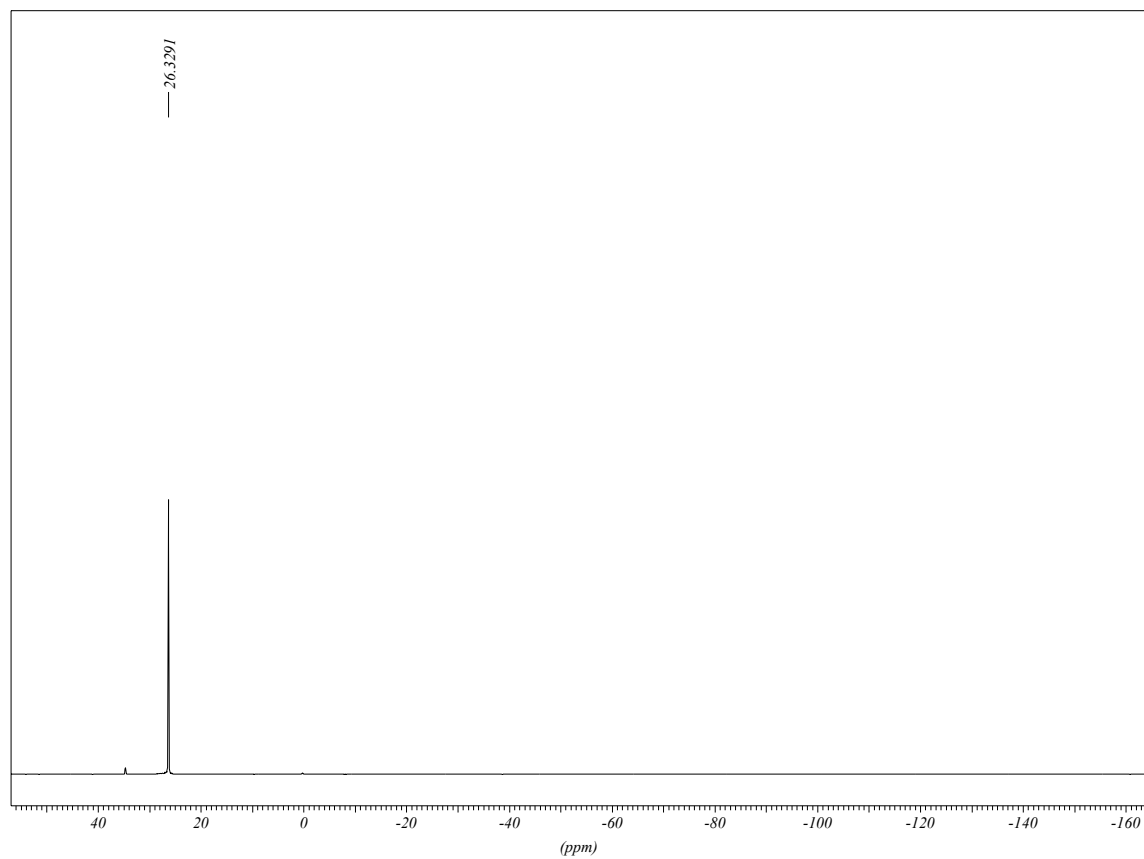
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



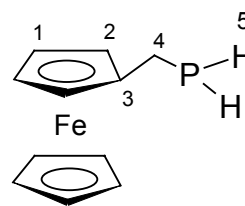
DEPT 135° (CDCl₃)



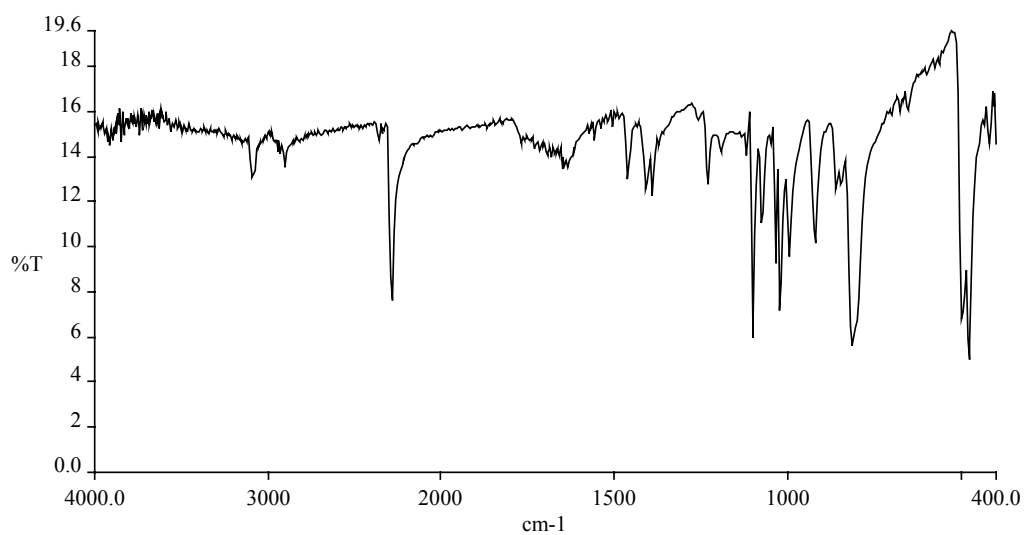
³¹P{¹H} RMN (CDCl₃)



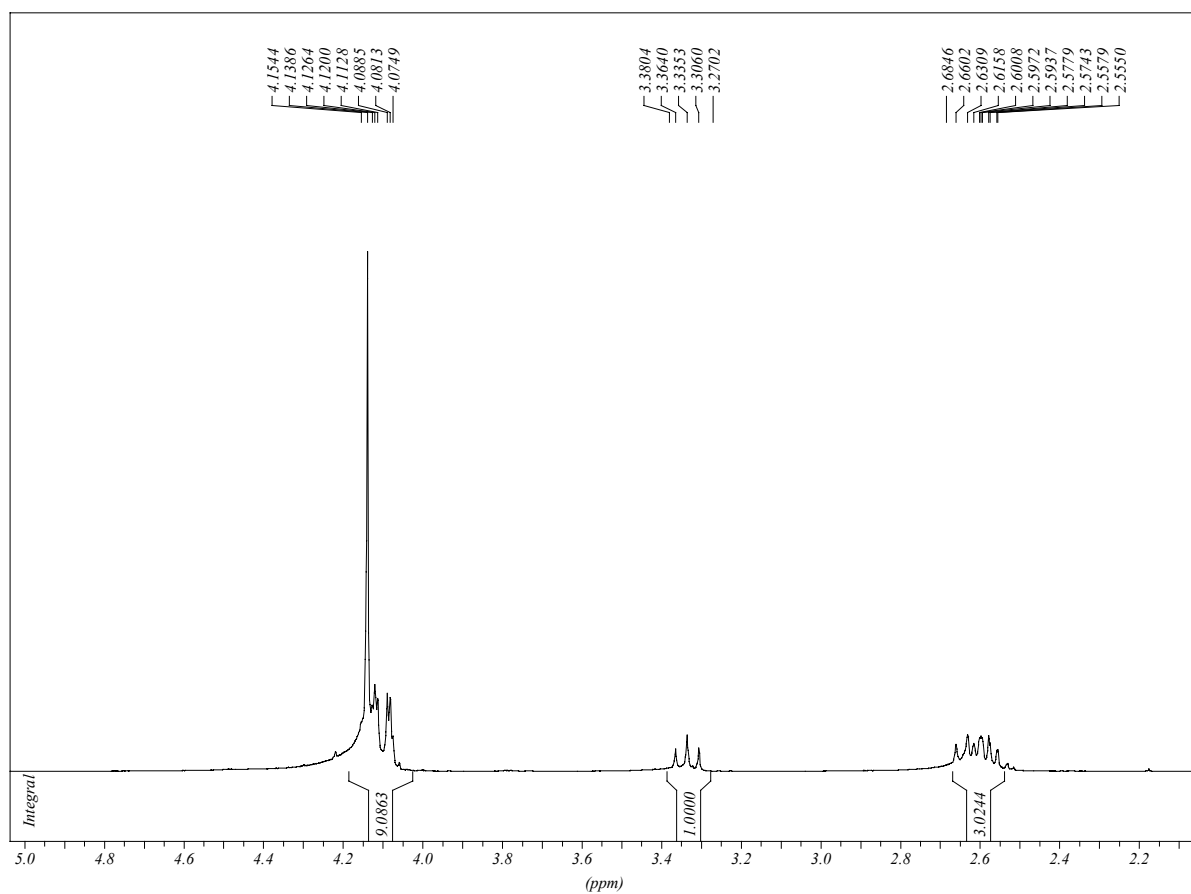
▪ (ferrocenilmetil)fosfà (29)



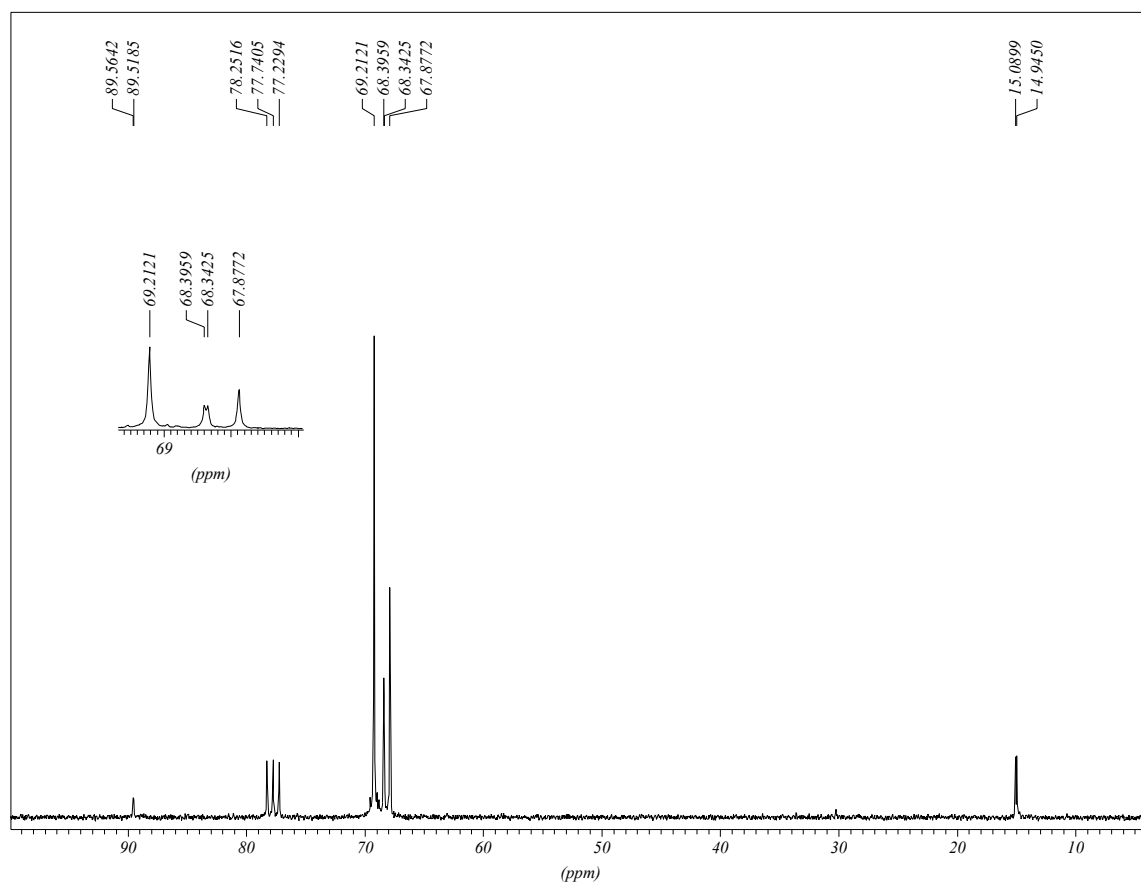
IR (KBr)



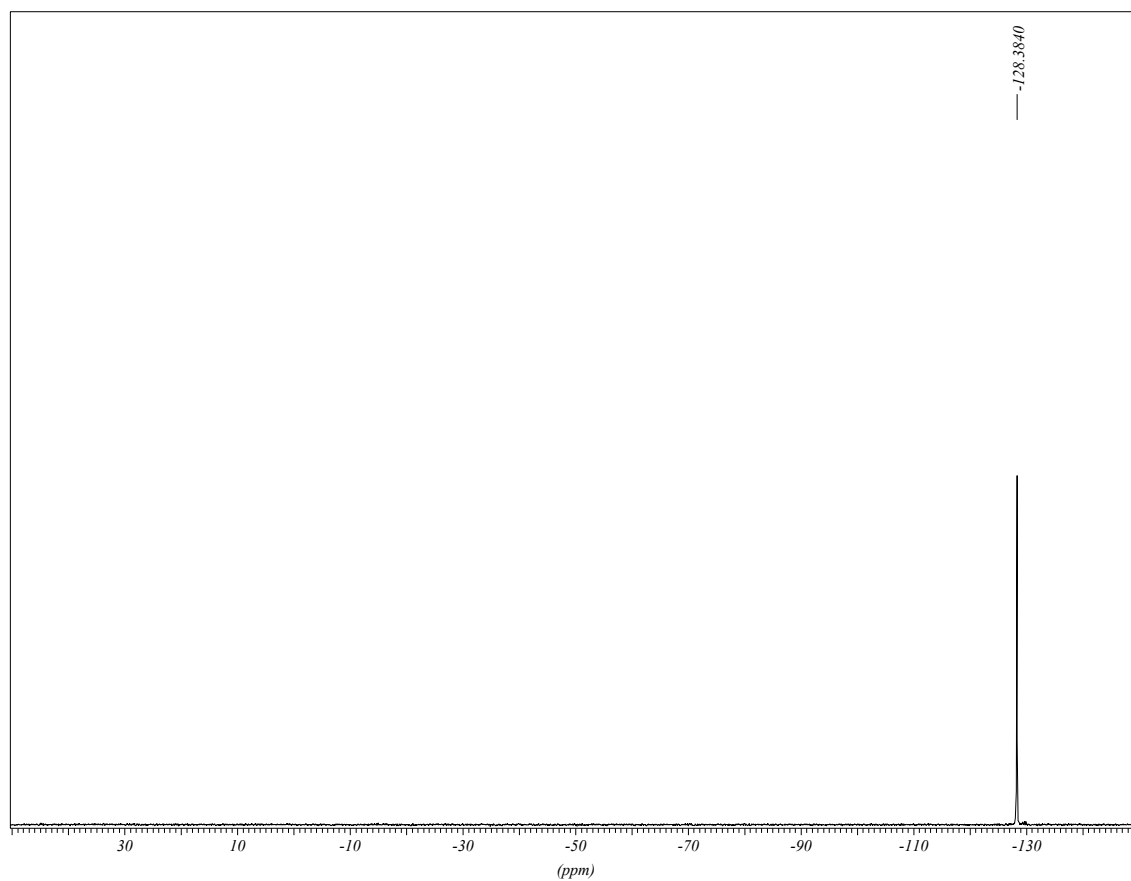
^1H RMN (CDCl_3)



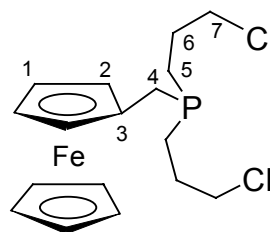
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



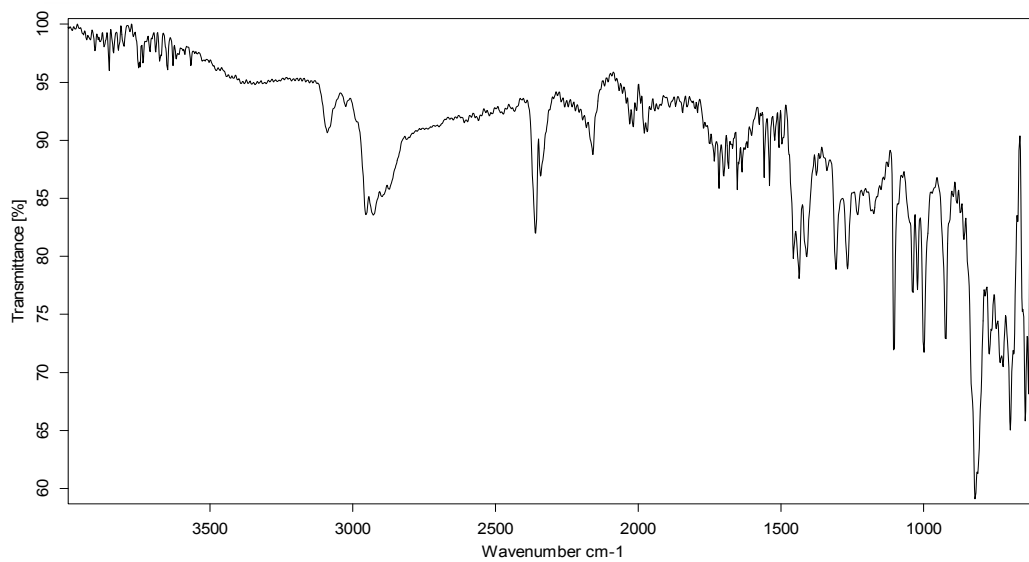
$^{31}\text{P}\{^1\text{H}\}$ RMN (CDCl_3)



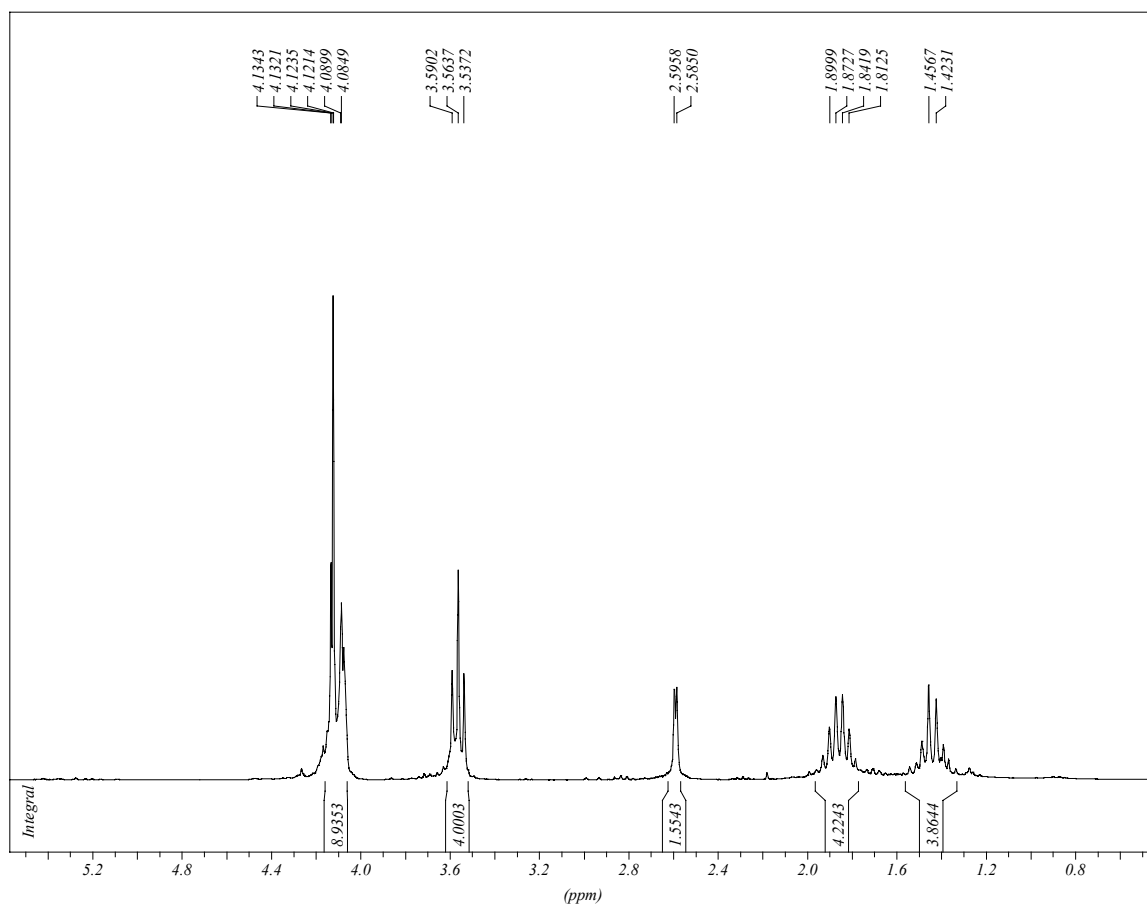
▪ bis(3-cloropropil)(ferrocenilmetil)fosfà (32)



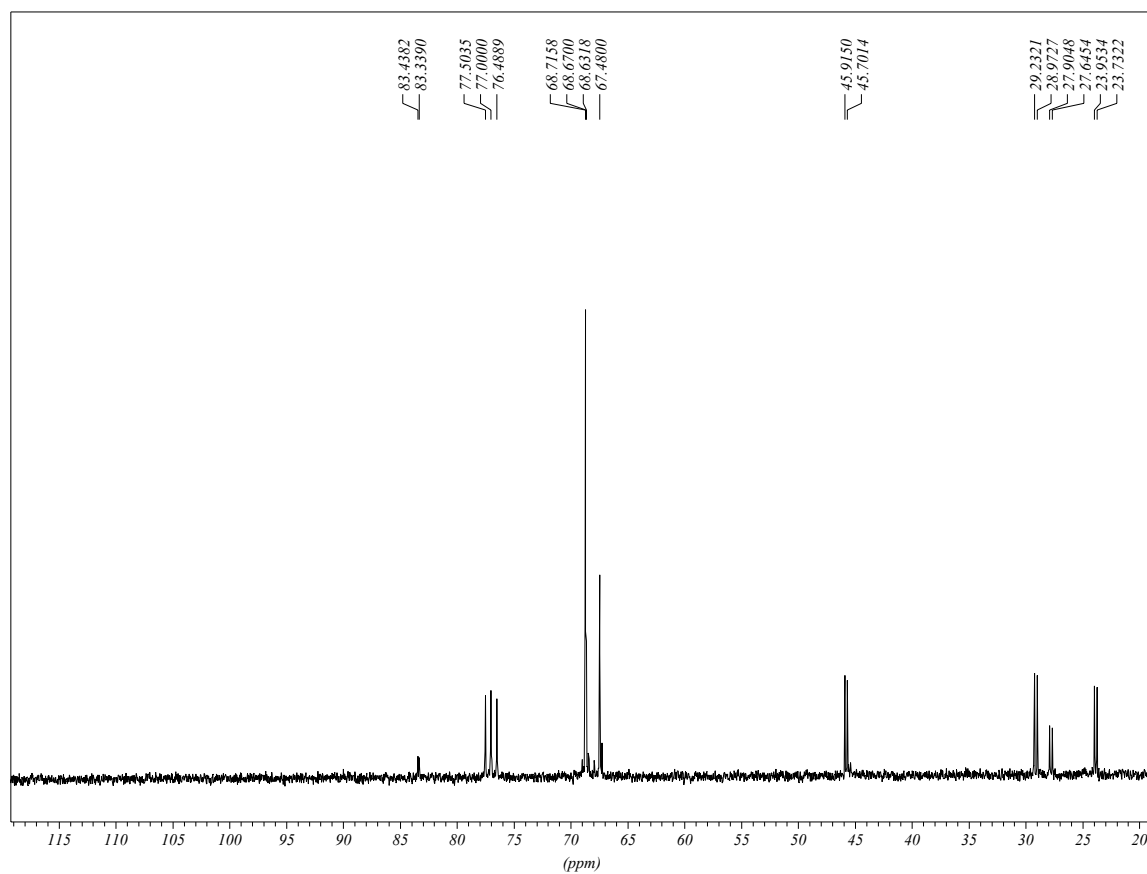
IR (ATR)



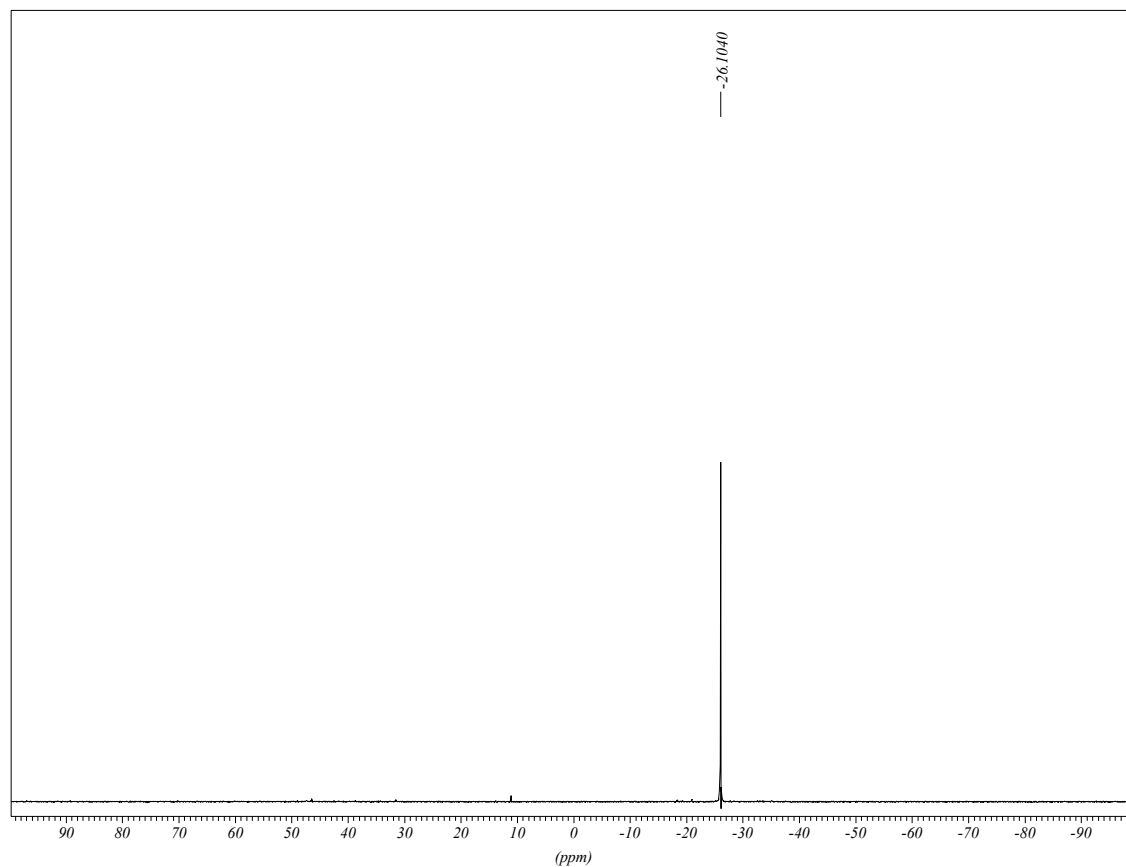
¹H RMN (CDCl₃)



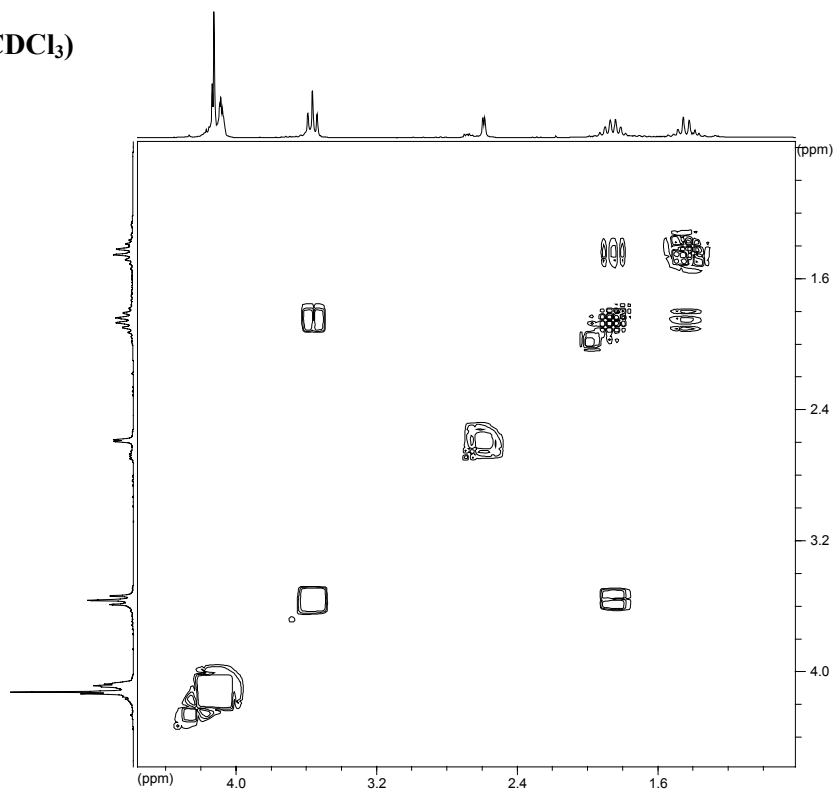
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



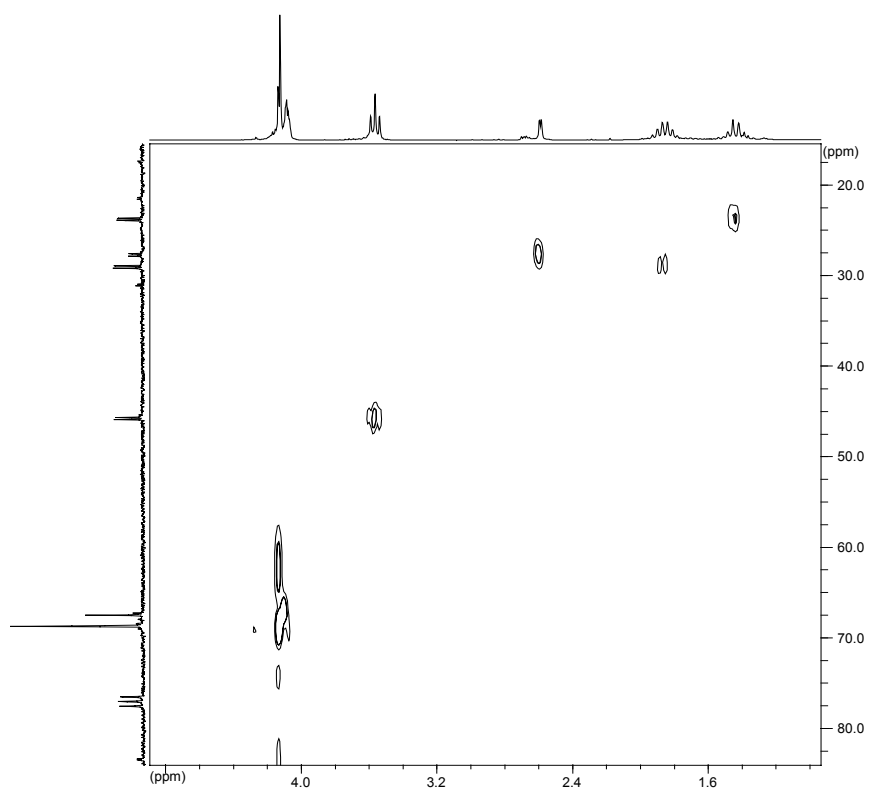
$^{31}\text{P}\{^1\text{H}\}$ RMN (CDCl_3)



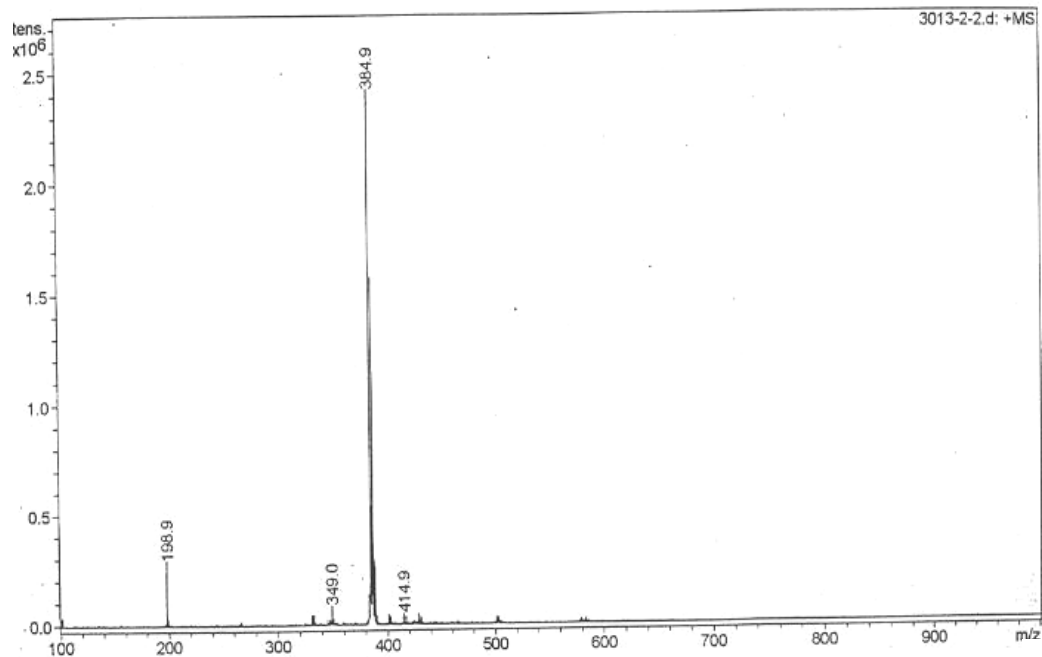
COSY (CDCl₃)



HMQC (CDCl₃)



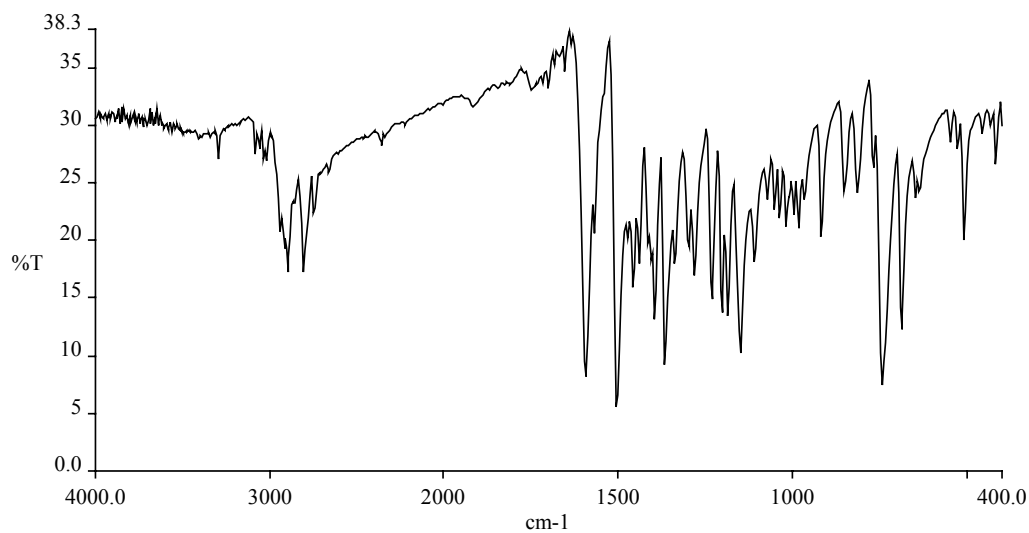
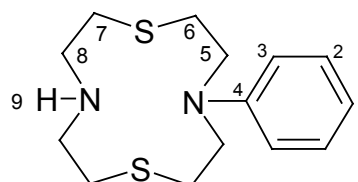
ESPECTROSCÒPIA DE MASSES (ESI(+)-IT) (10 ppm en MeOH, 0.1% HCOOH)



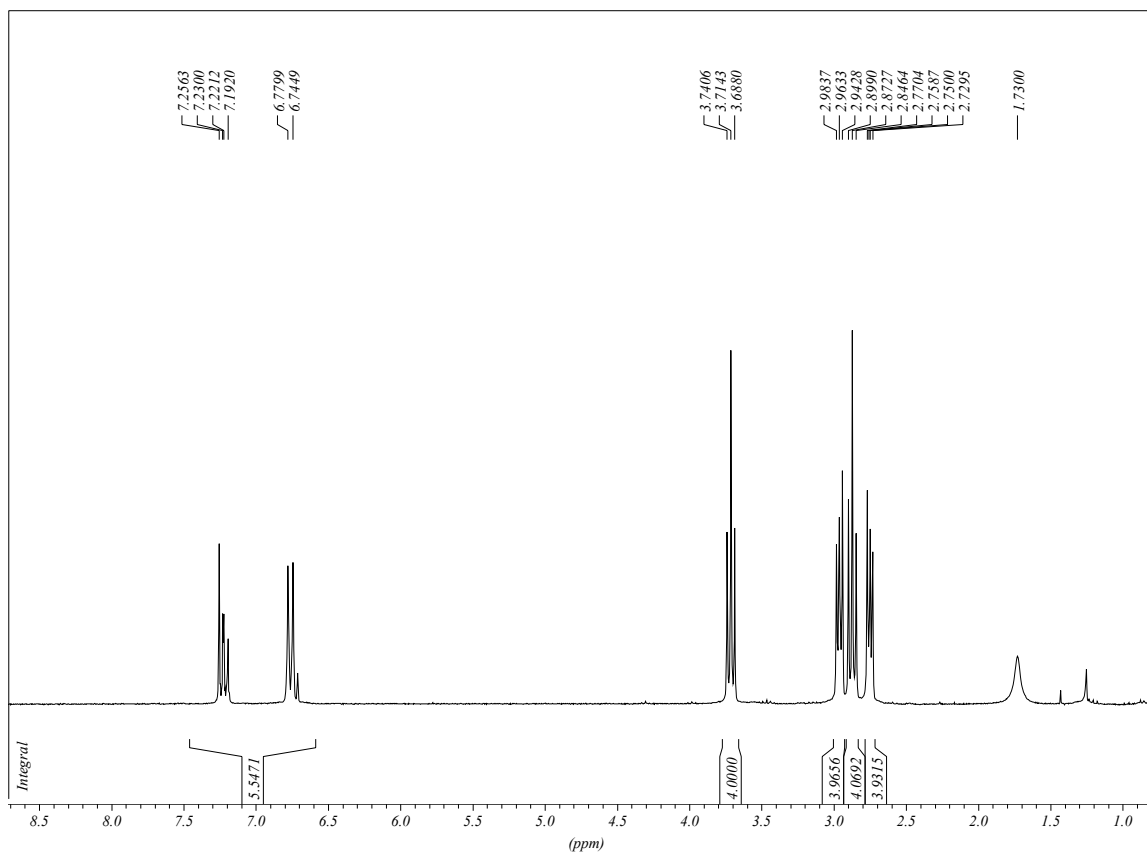
2 Lligands macrocíclics

- 4-fenil-1,7-ditia-4,10-diazaciclododecà (L1)

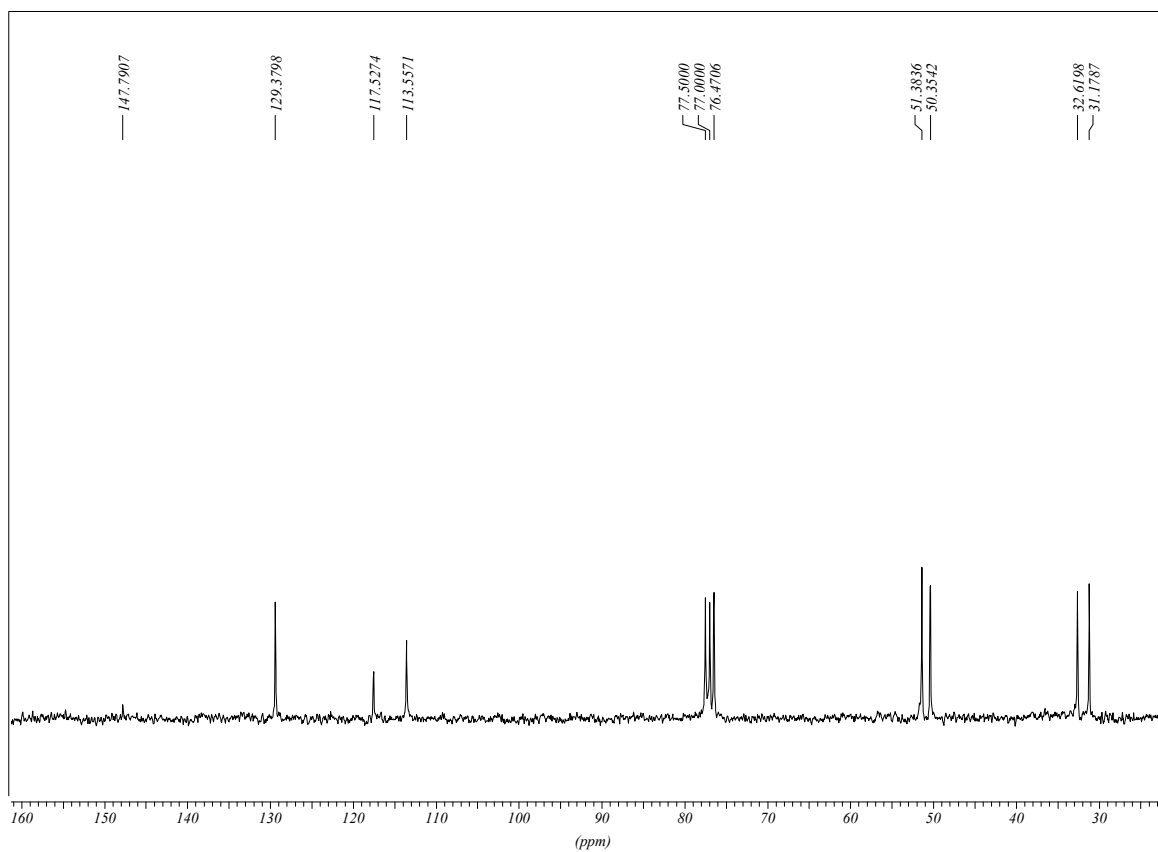
IR (KBr)



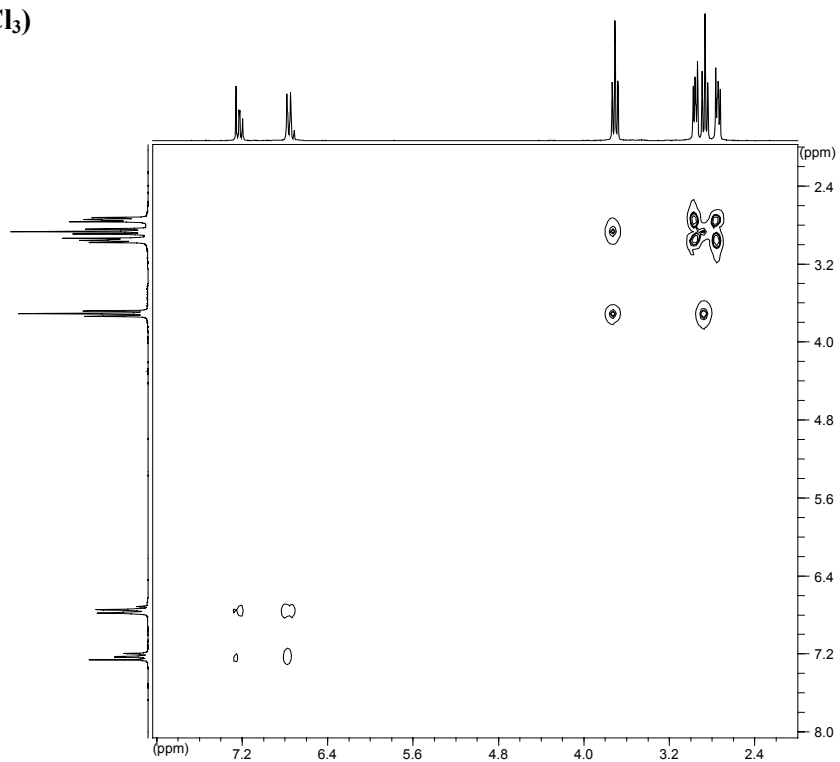
^1H RMN (CDCl_3)



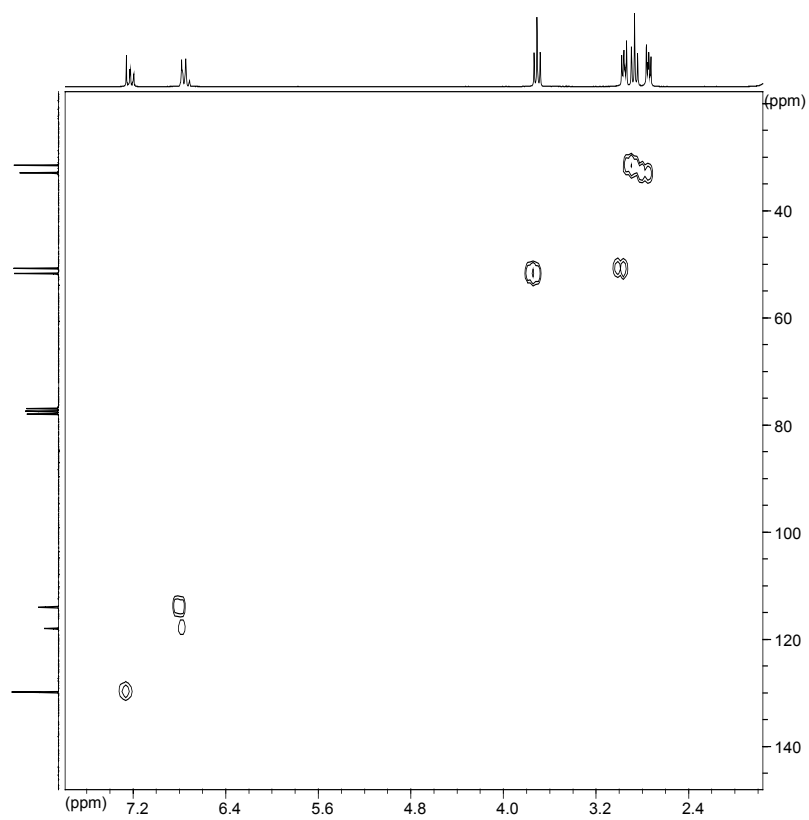
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



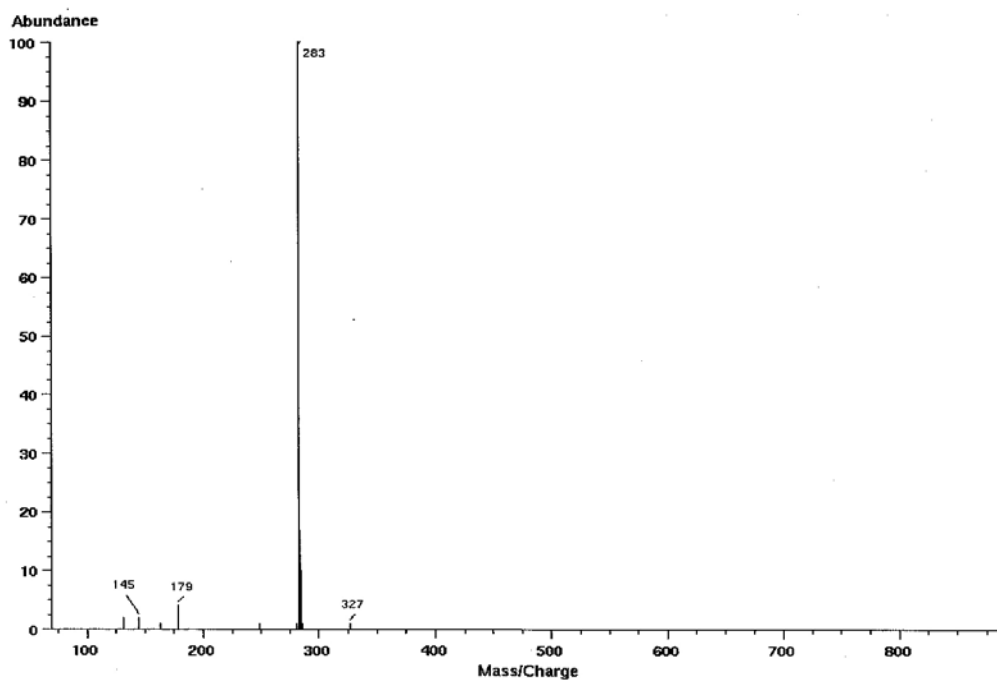
COSY (CDCl₃)



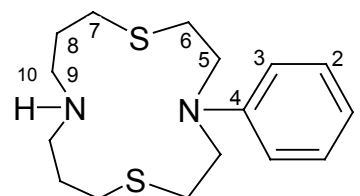
HMQC (CDCl₃)



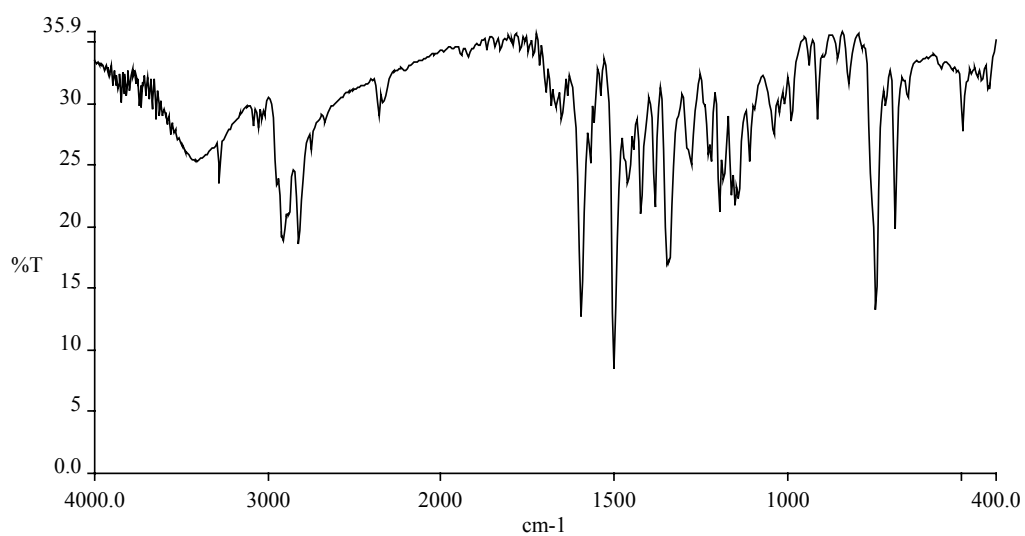
ESPECTROSCÒPIA DE MASSES (IQ)



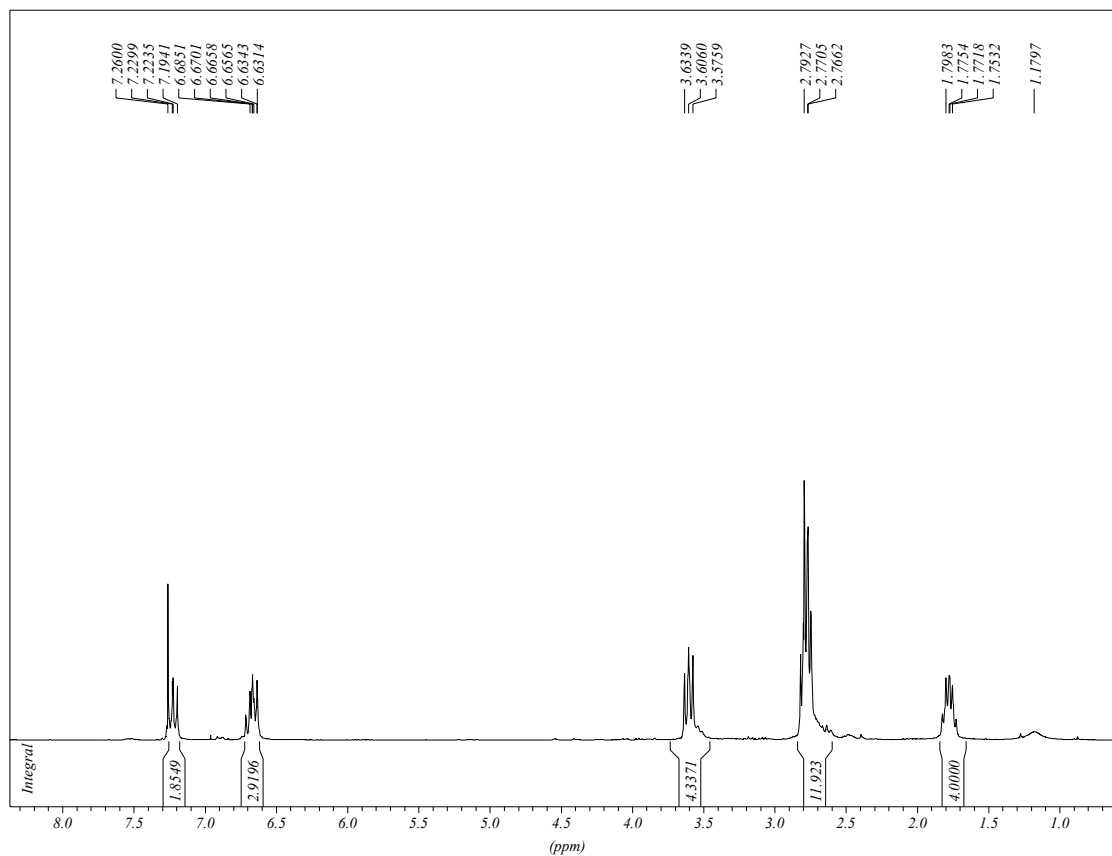
- 4-fenil-1,7-ditia-4,11-diazaciclodecà (L2)



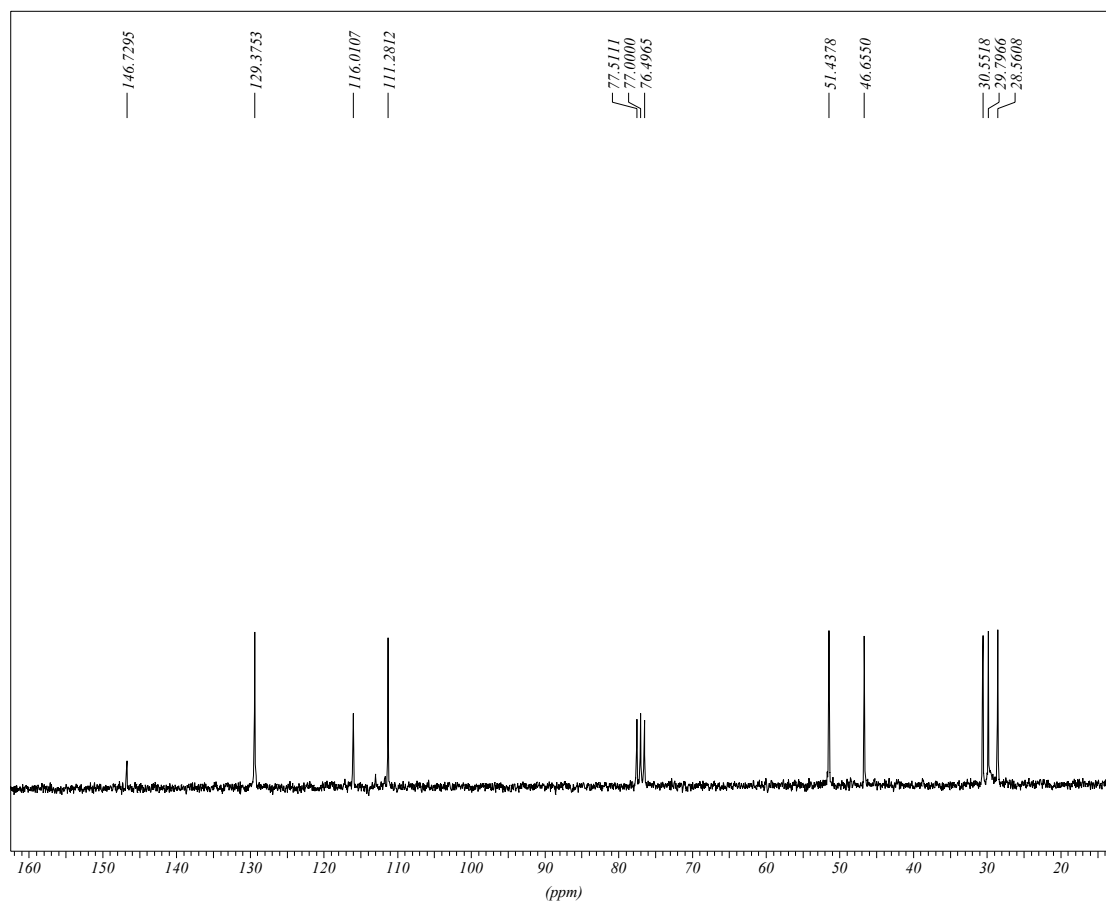
IR (KBr)



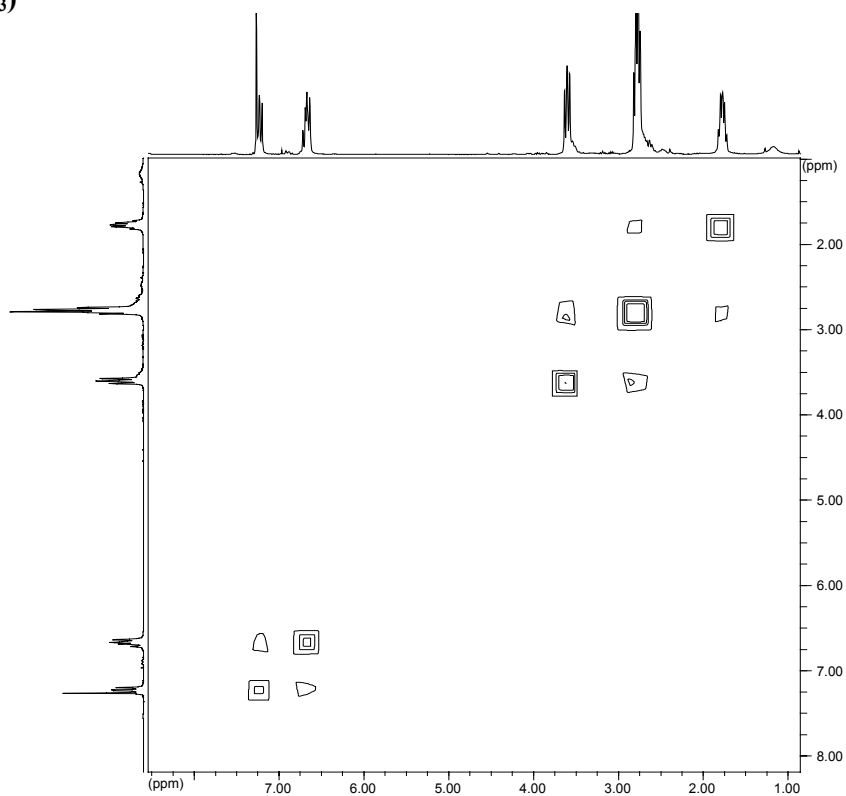
^1H RMN (CDCl_3)



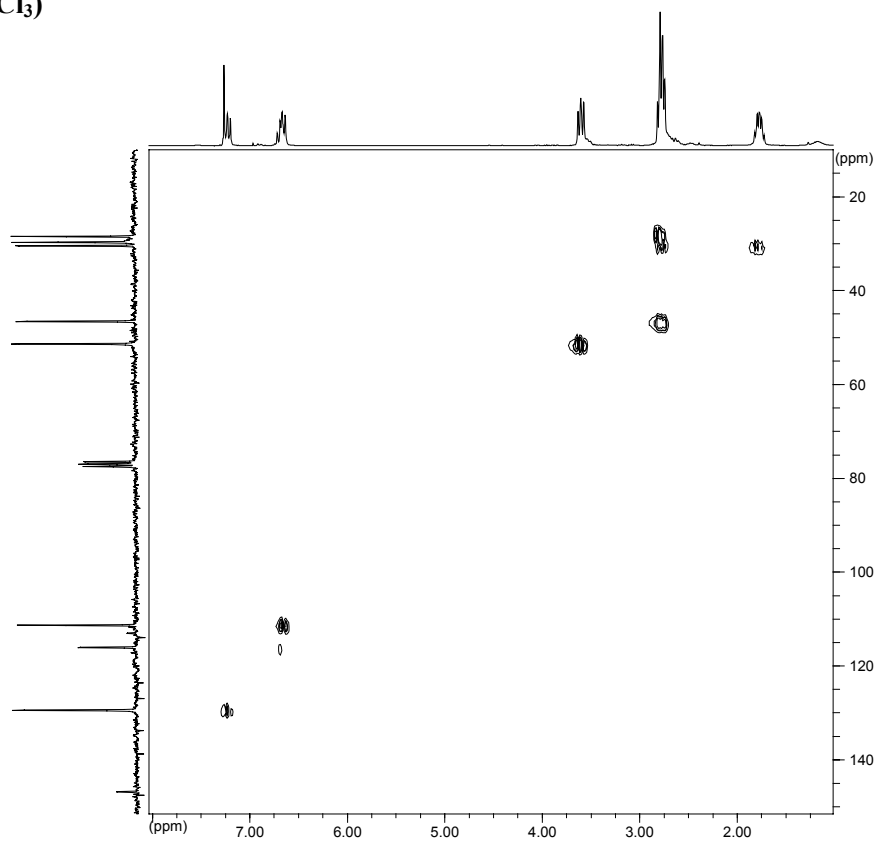
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



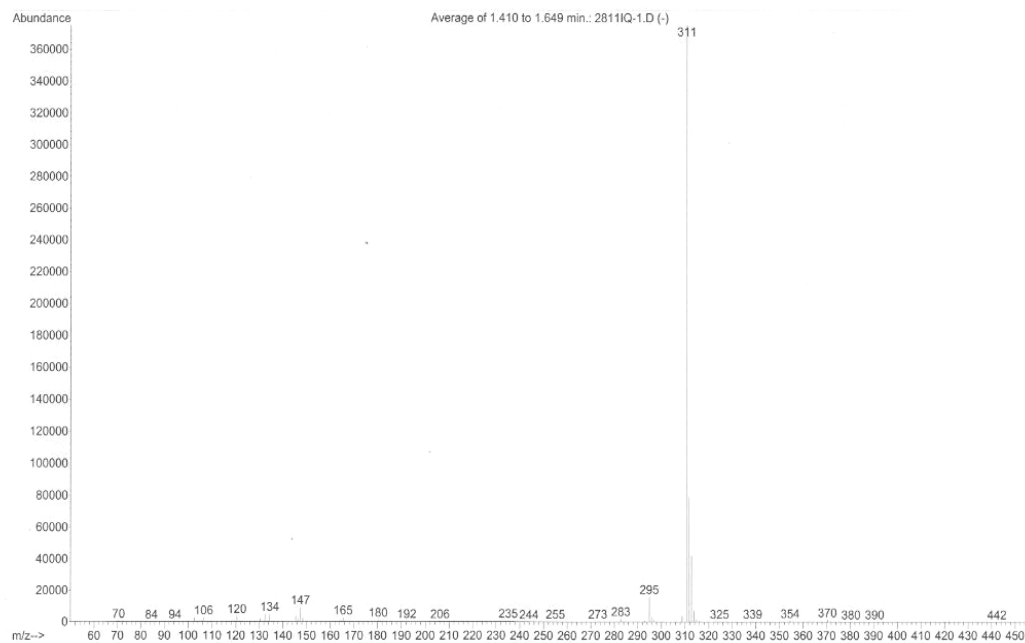
COSY (CDCl₃)



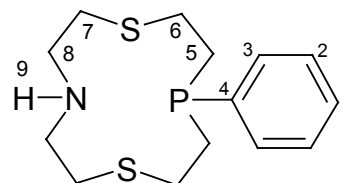
HMQC (CDCl₃)



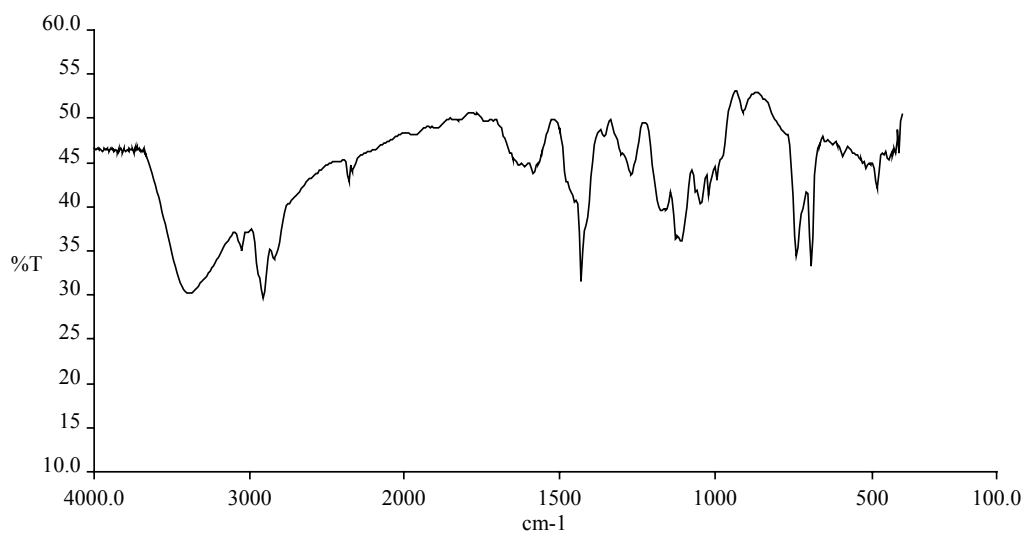
ESPECTROSCÒPIA DE MASSES (IQ)



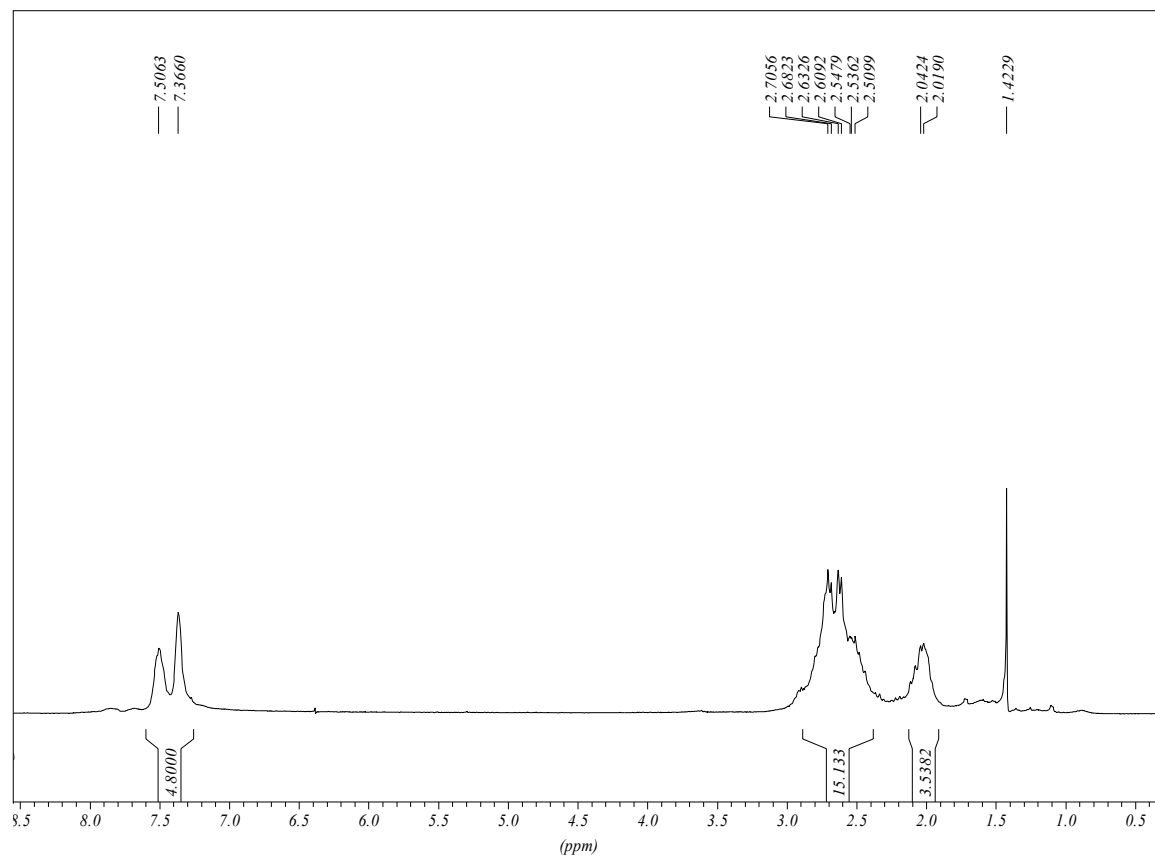
- 4-fenil-1,7-ditia-4-fosfa-10-azaciclododecà (L3)



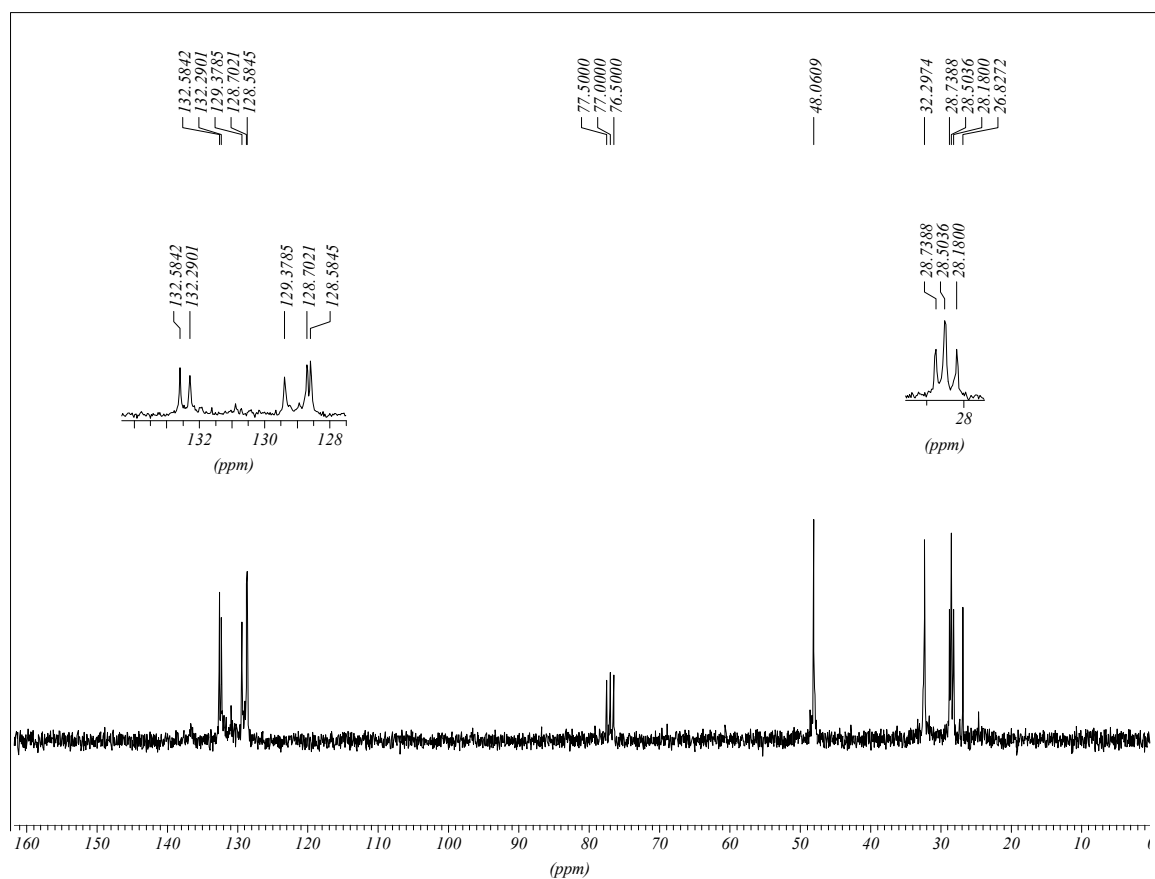
IR (KBr)



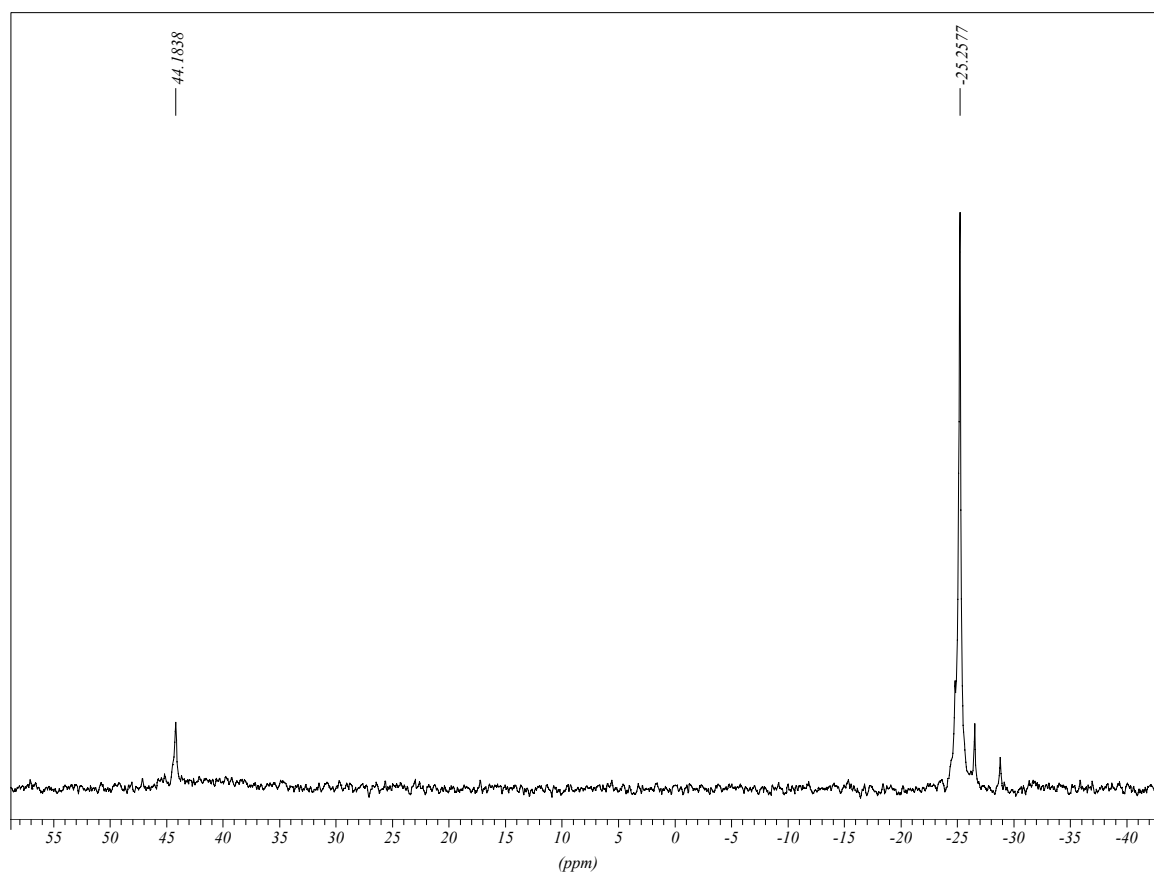
^1H RMN (CDCl_3)



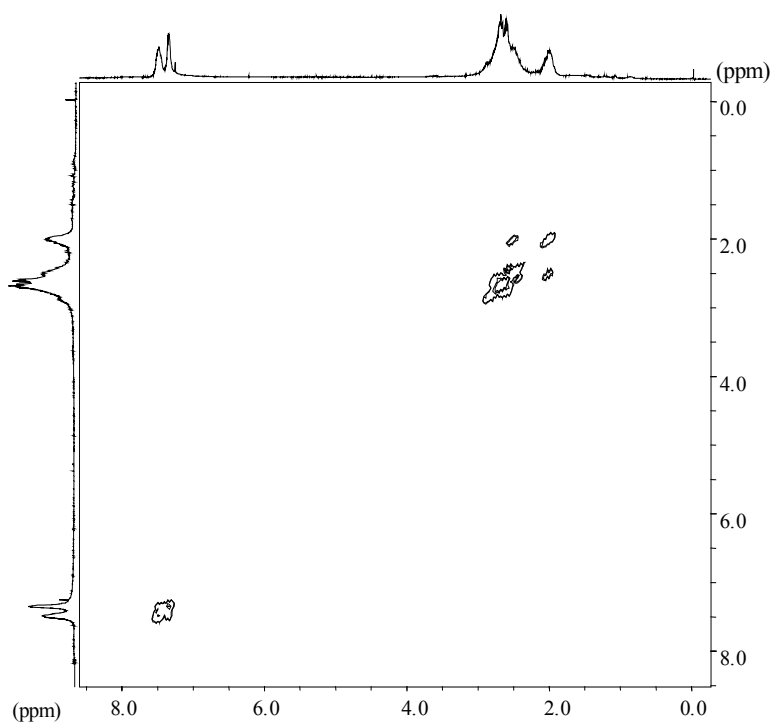
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



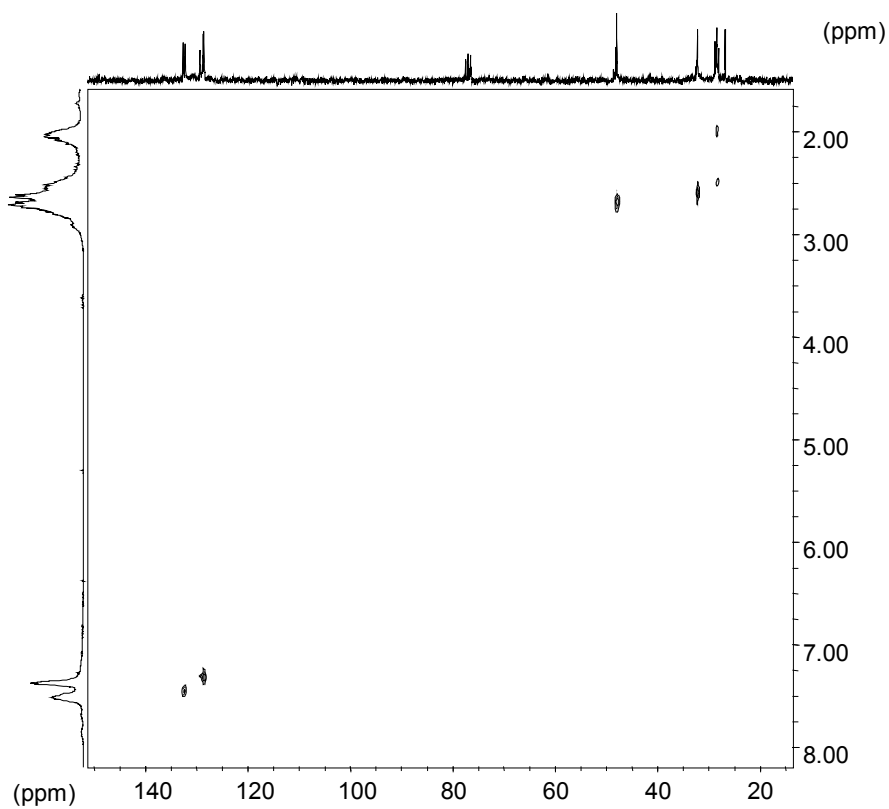
$^{31}\text{P}\{^1\text{H}\}$ RMN (CDCl_3)



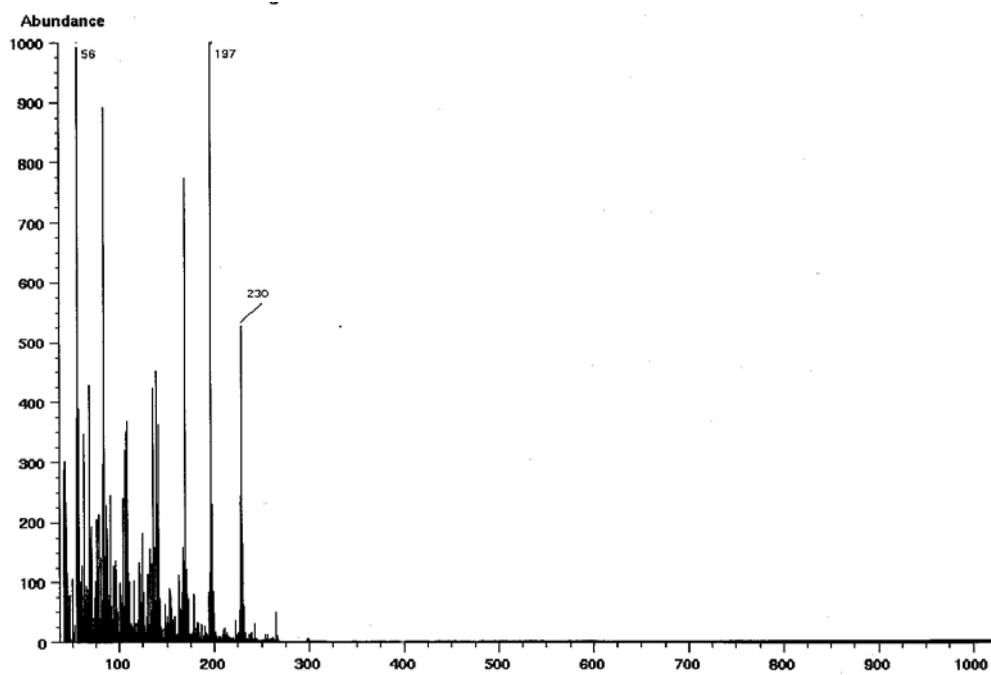
COSY (CDCl_3)



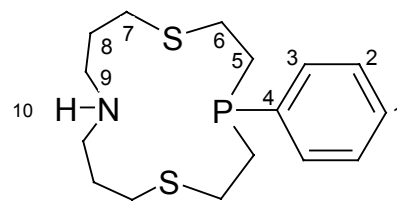
HMQC (CDCl₃)



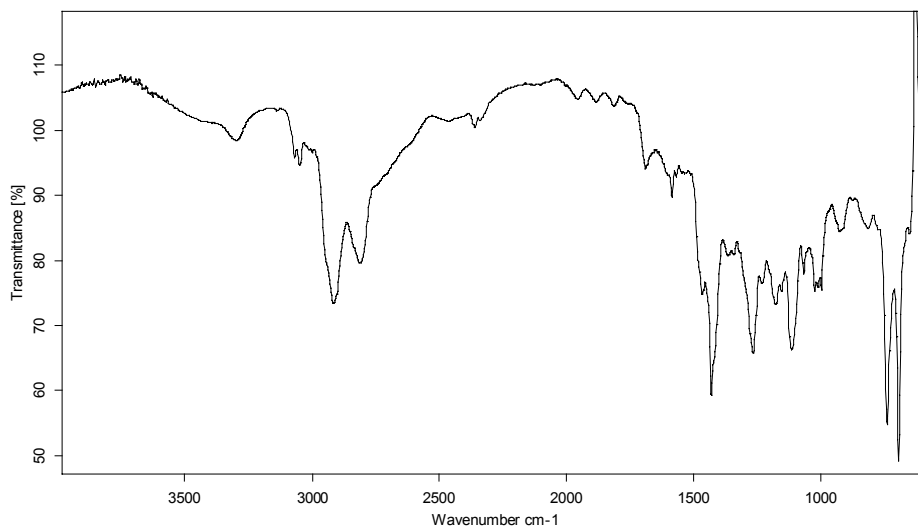
ESPECTROSCÒPIA DE MASSES (IE)



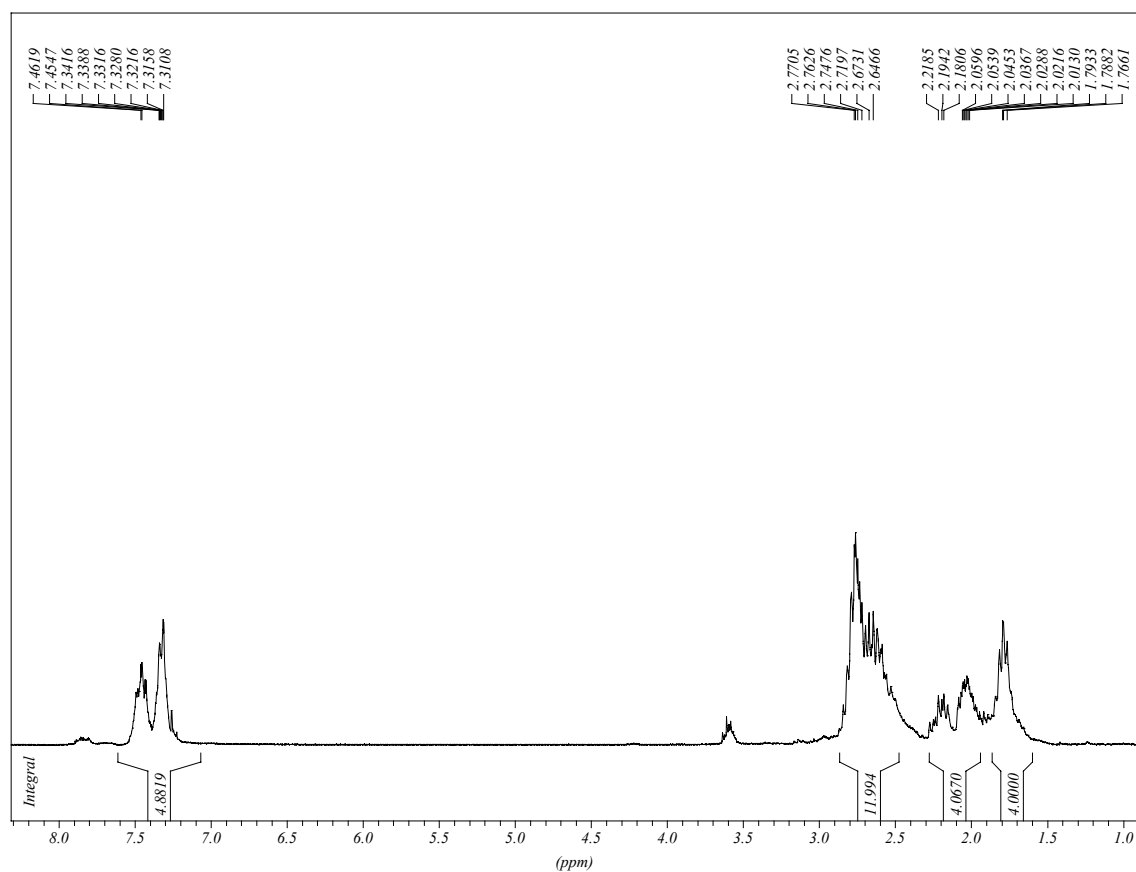
▪ 4-fenil-1,7-ditia-4-fosfa-11-azacicloctetradecà (L4)



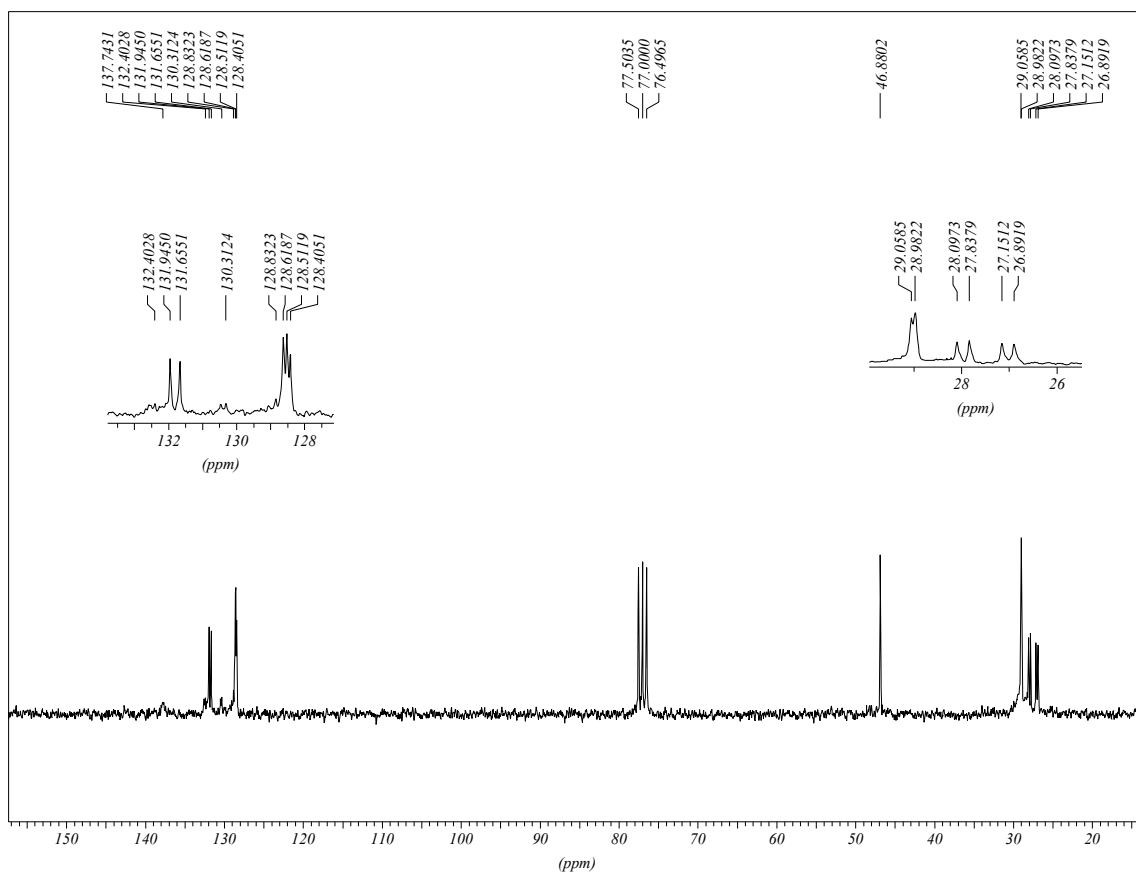
IR (NaCl)



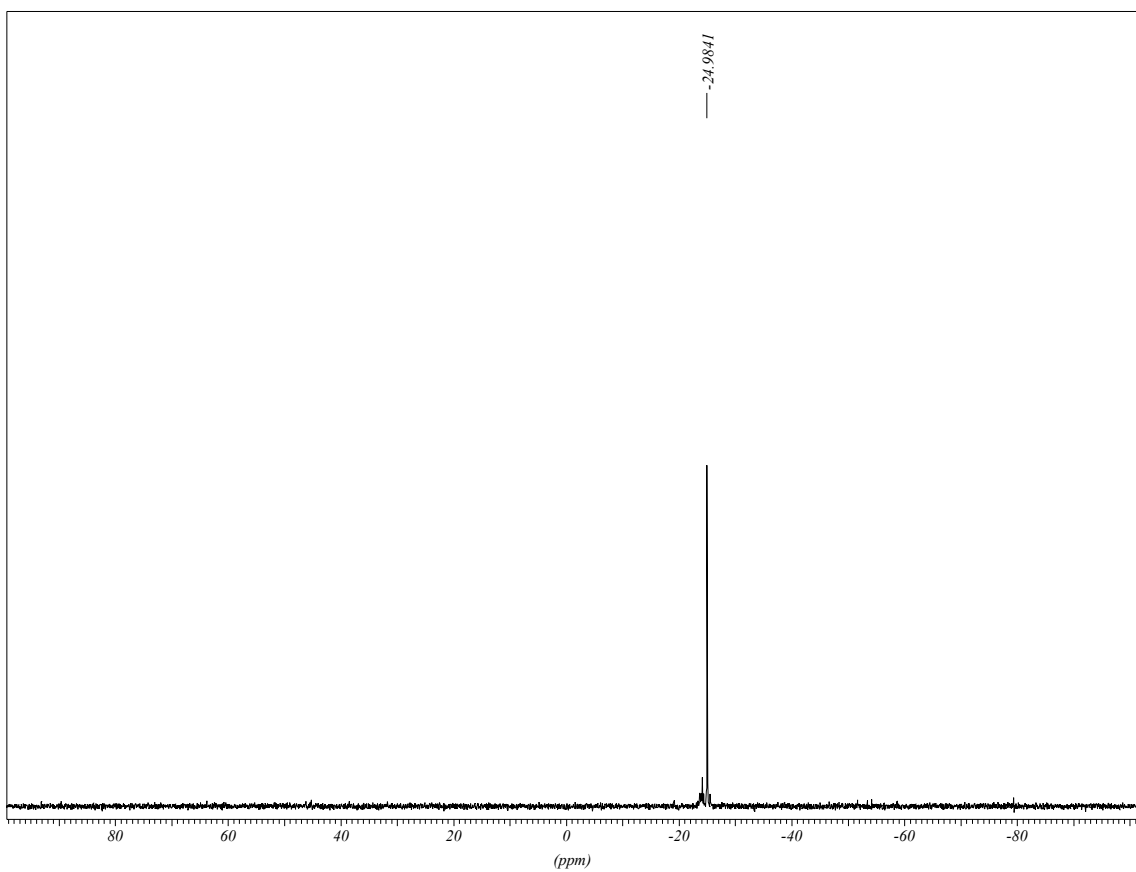
¹H RMN (CDCl₃)



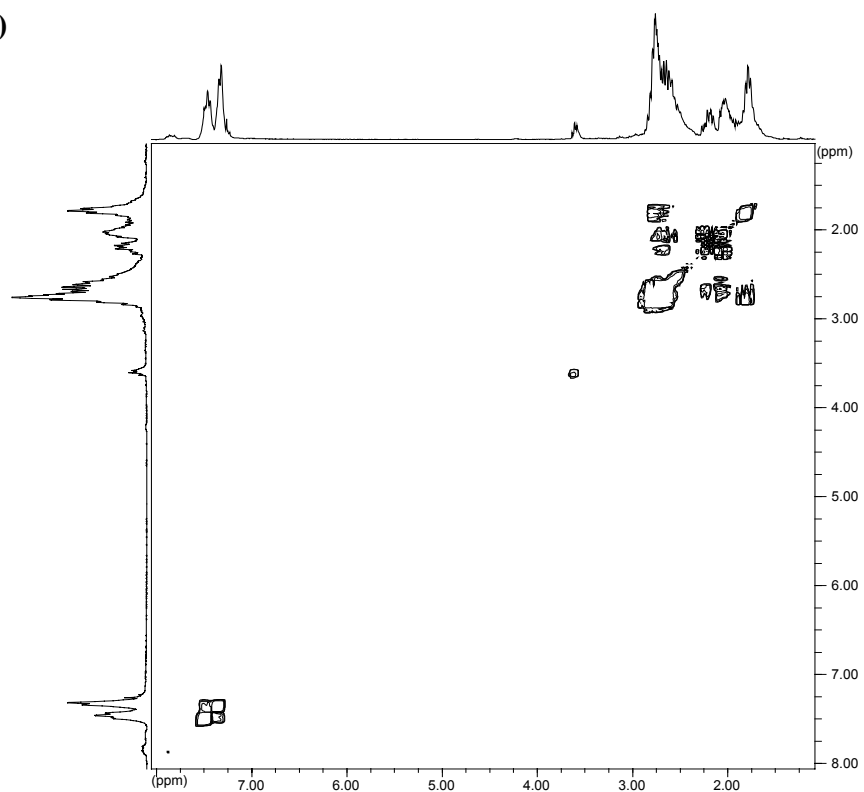
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



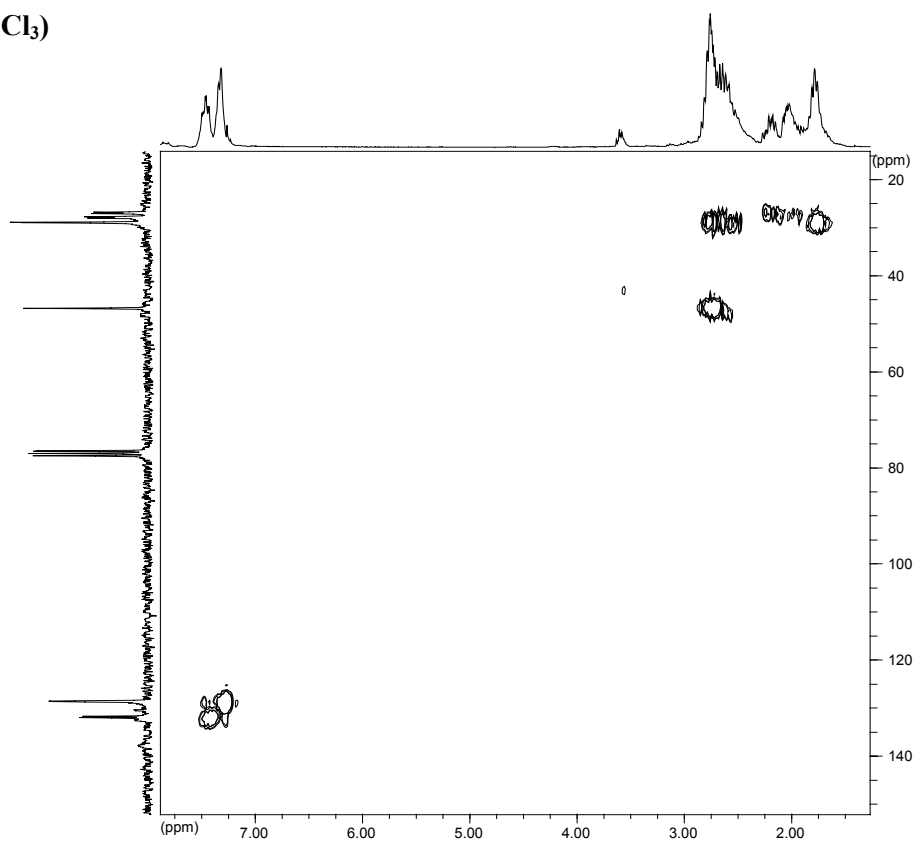
$^{31}\text{P}\{^1\text{H}\}$ RMN (CDCl_3)



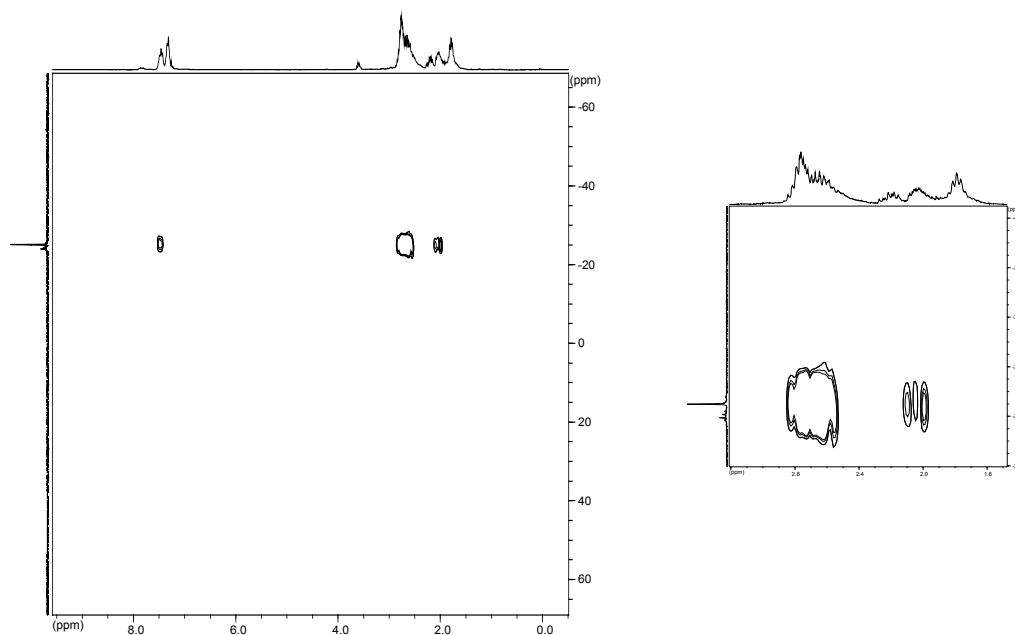
COSY (CDCl₃)



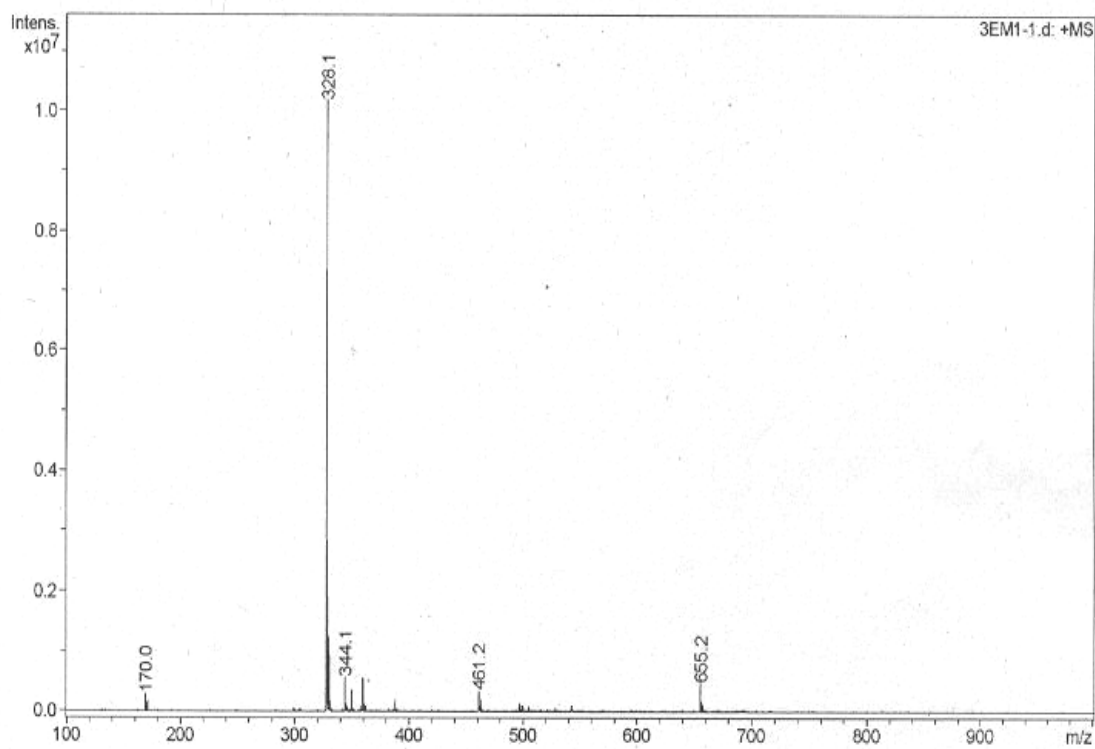
HMQC (CDCl₃)



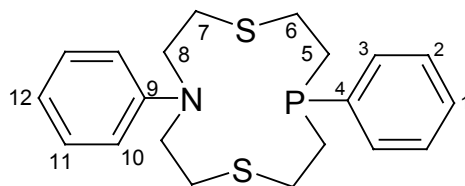
HMBC (CDCl₃)



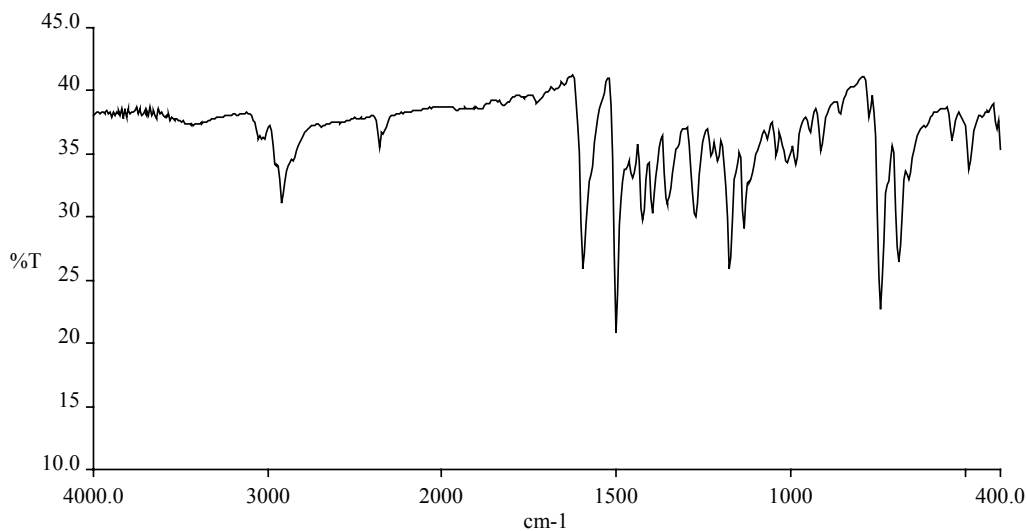
ESPECTROSCÒPIA DE MASSES (ESI(+)-IT) (10 ppm en MeOH)



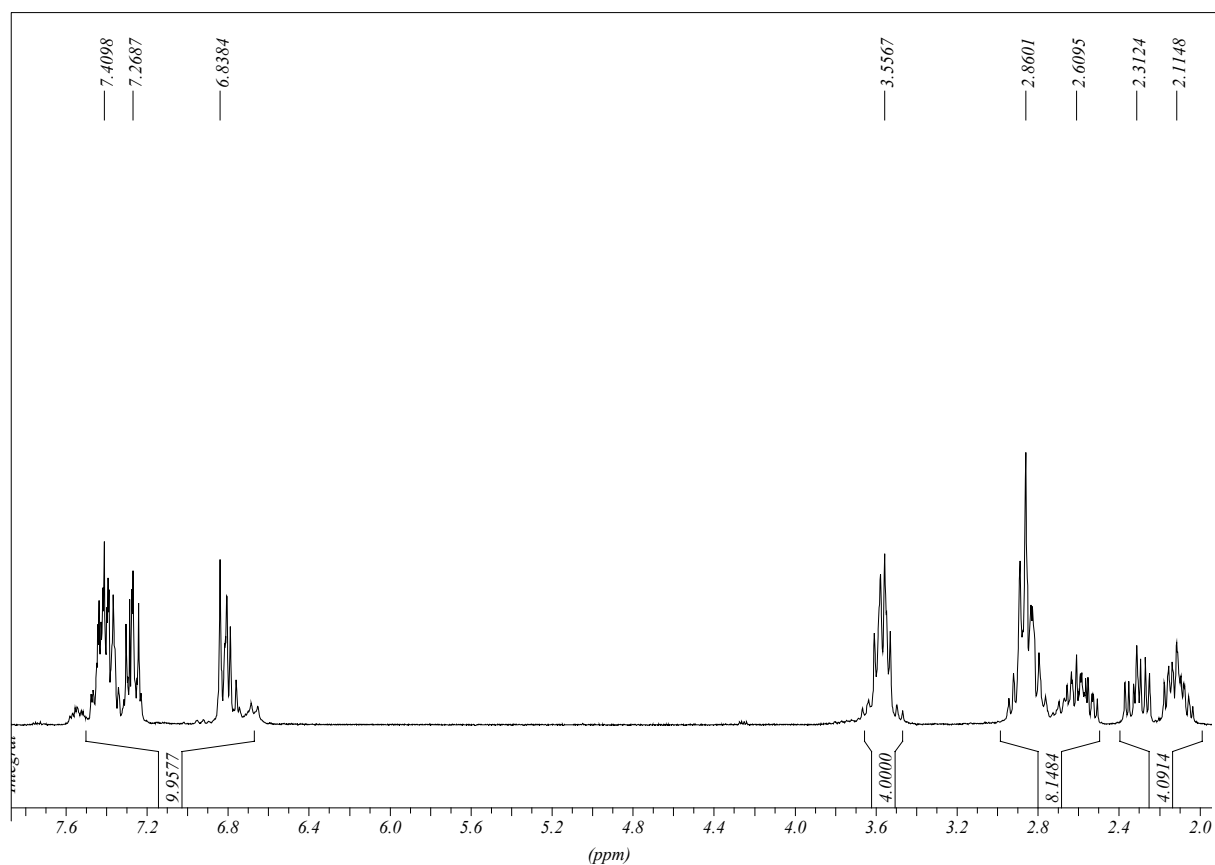
▪ 4,10-difenil-1,7-ditia-4-fosfa-10-azaciclododecà (L5)



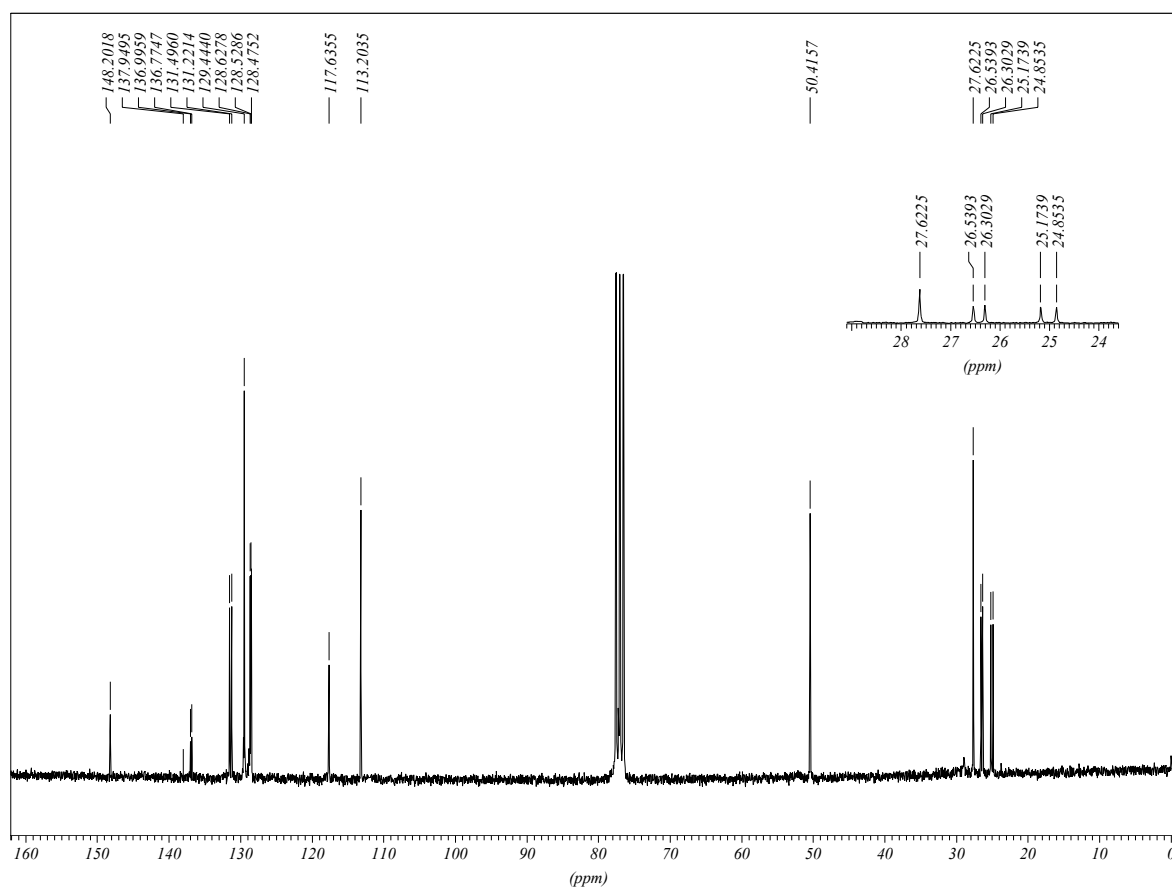
IR (KBr)



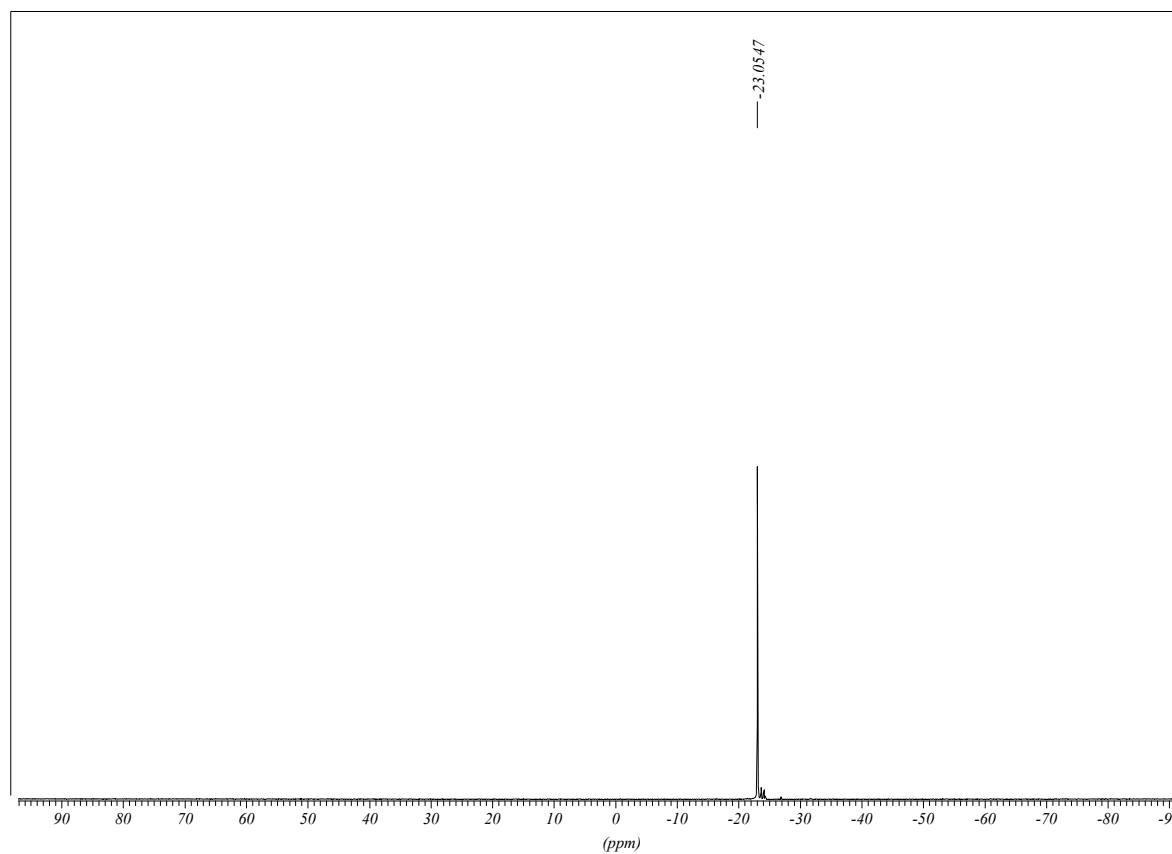
¹H RMN (CDCl₃)



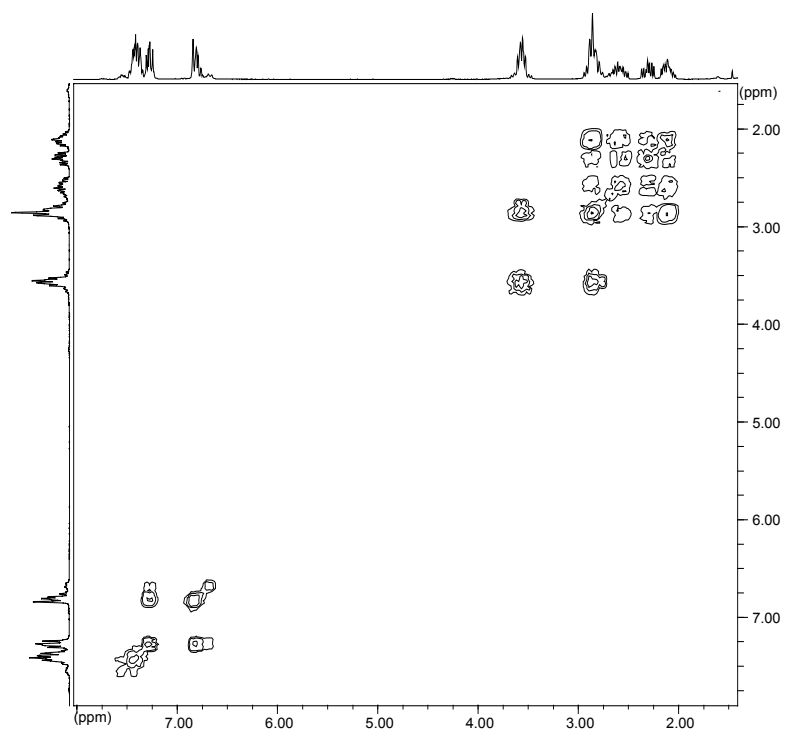
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



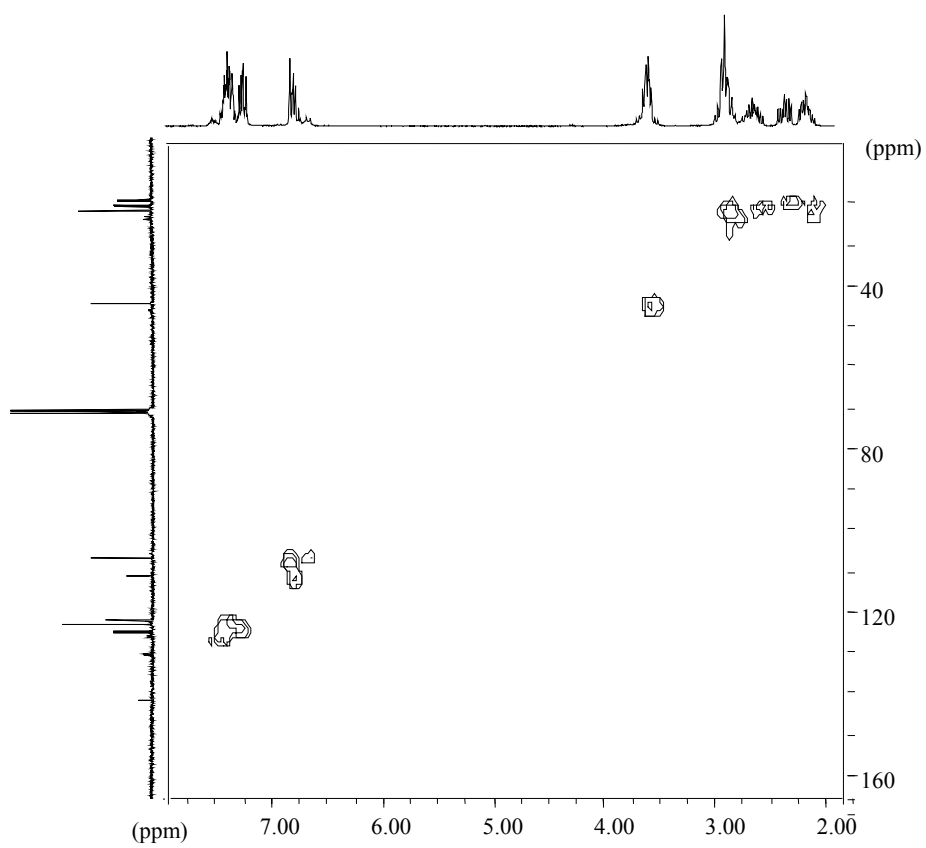
$^{31}\text{P}\{^1\text{H}\}$ RMN (CDCl_3)



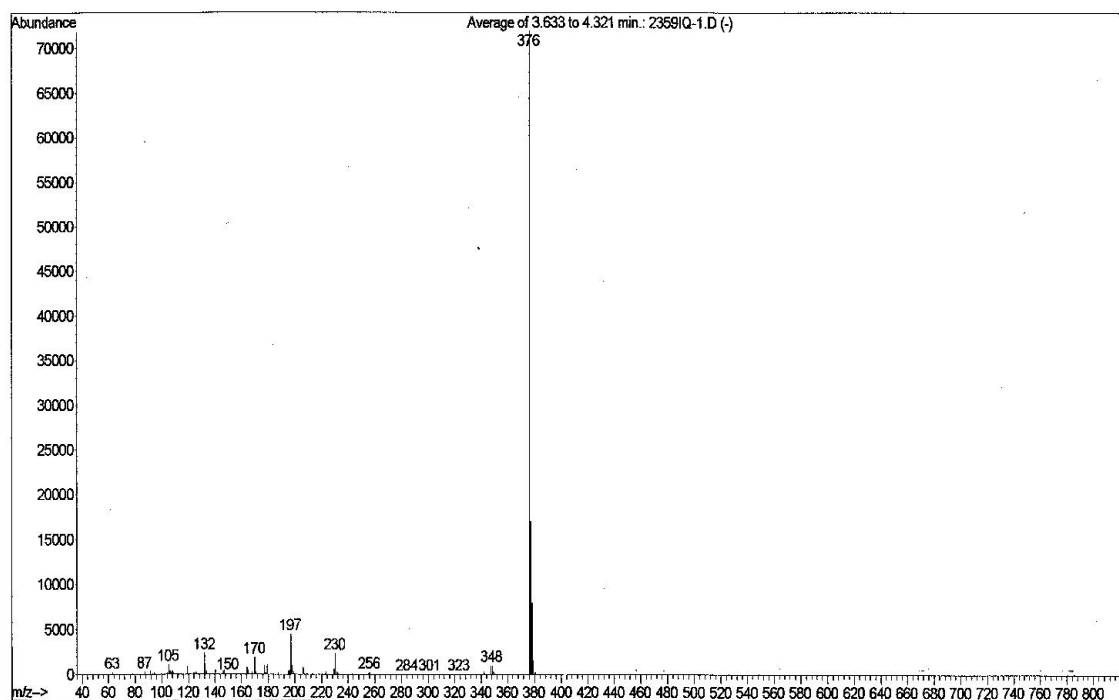
COSY (CDCl₃)



HMQC (CDCl₃)

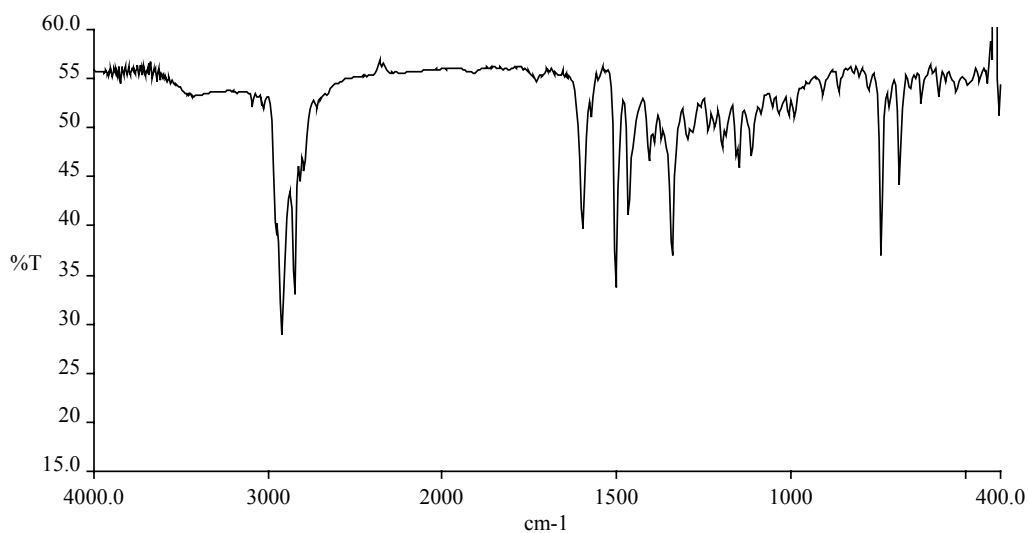
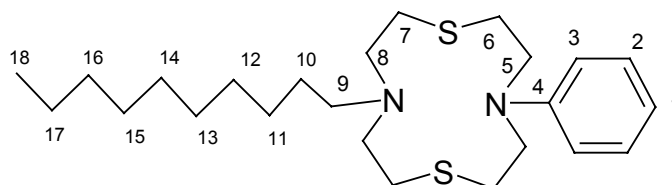


ESPECTROSCÒPIA DE MASSES (IQ)

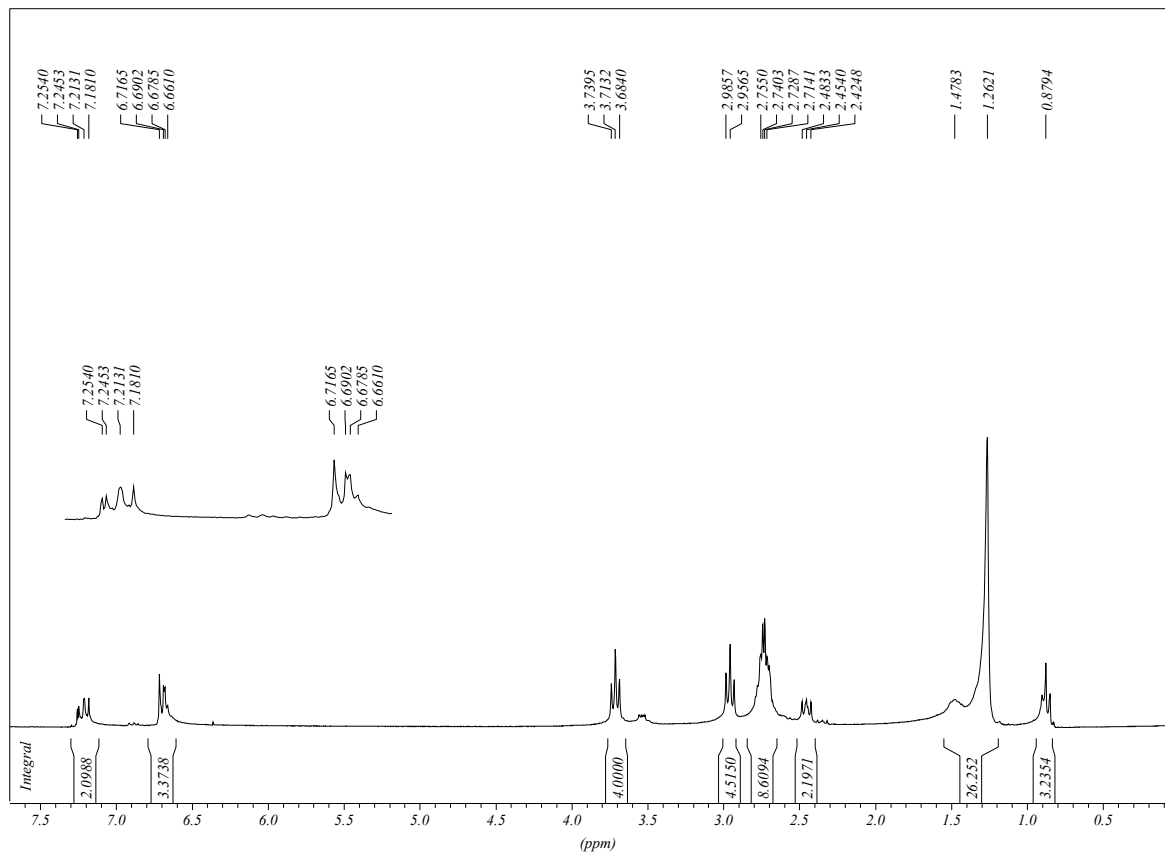


▪ 4-fenil-10-decil-1,7-ditia-4,10-diazaciclododecà (L6)

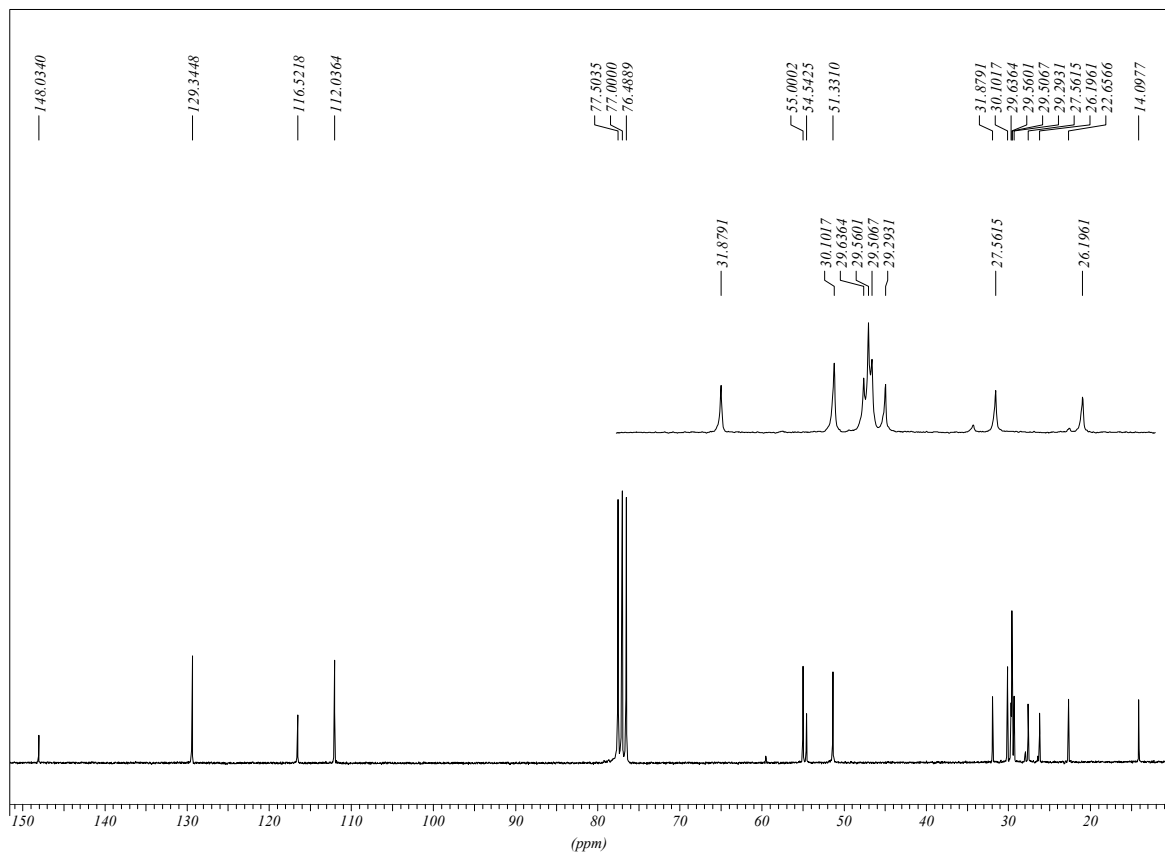
IR (KBr)



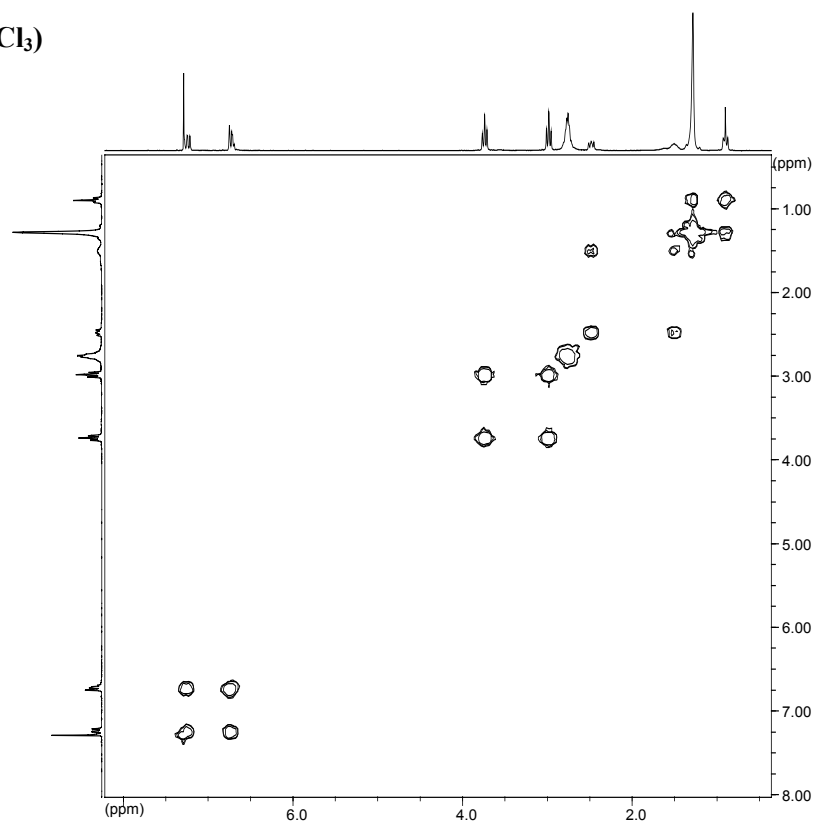
^1H RMN (CDCl_3)



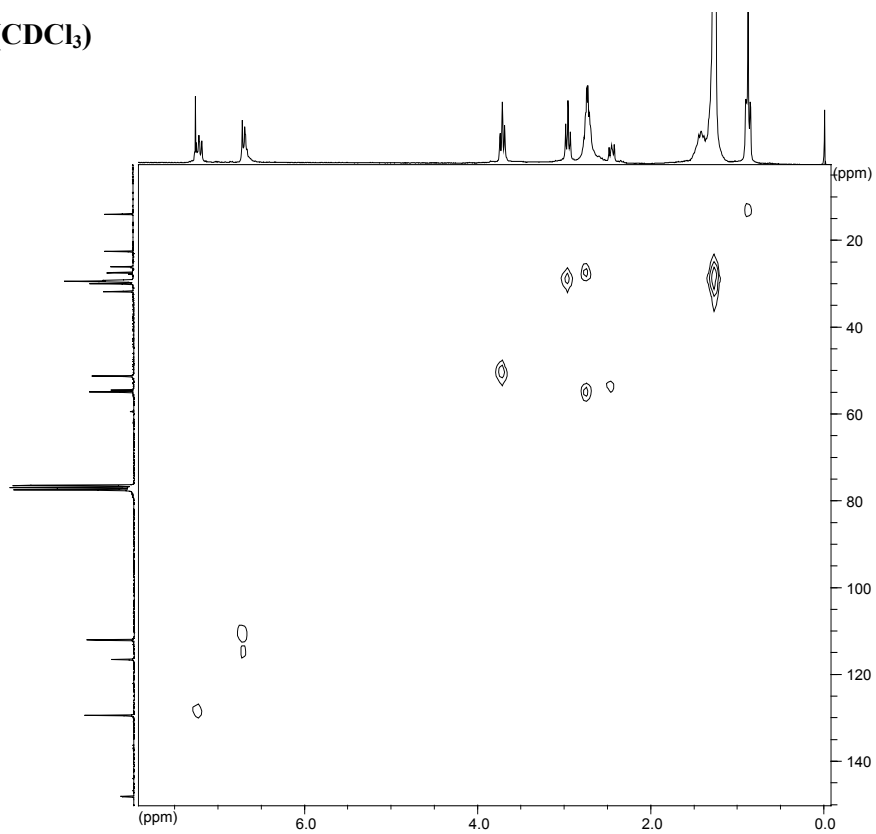
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



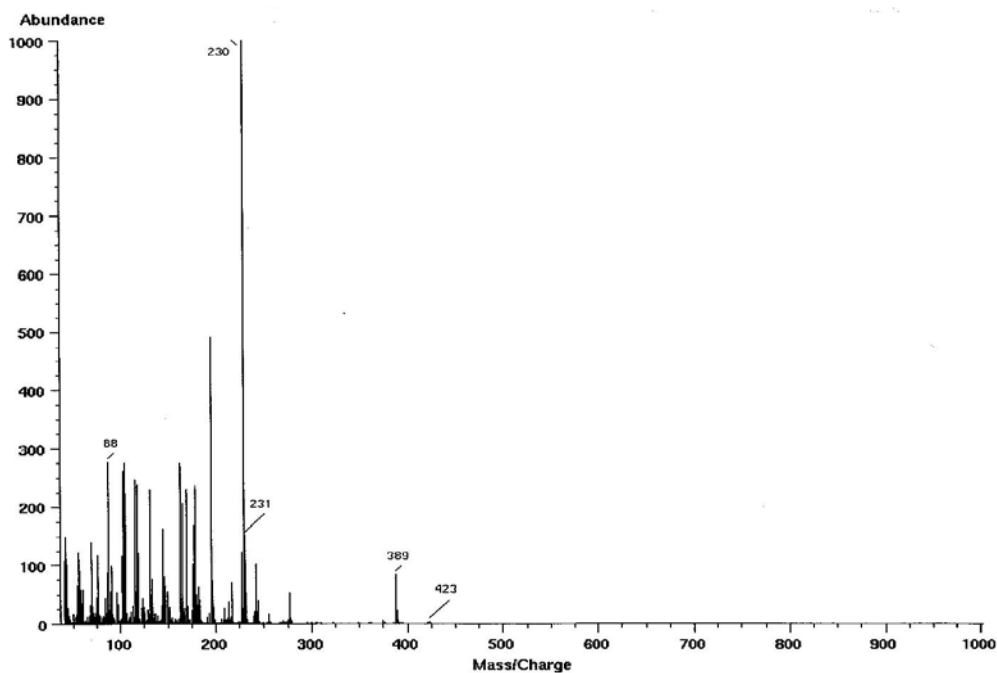
COSY (CDCl₃)



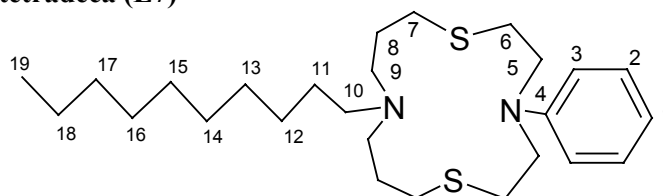
HMQC (CDCl₃)



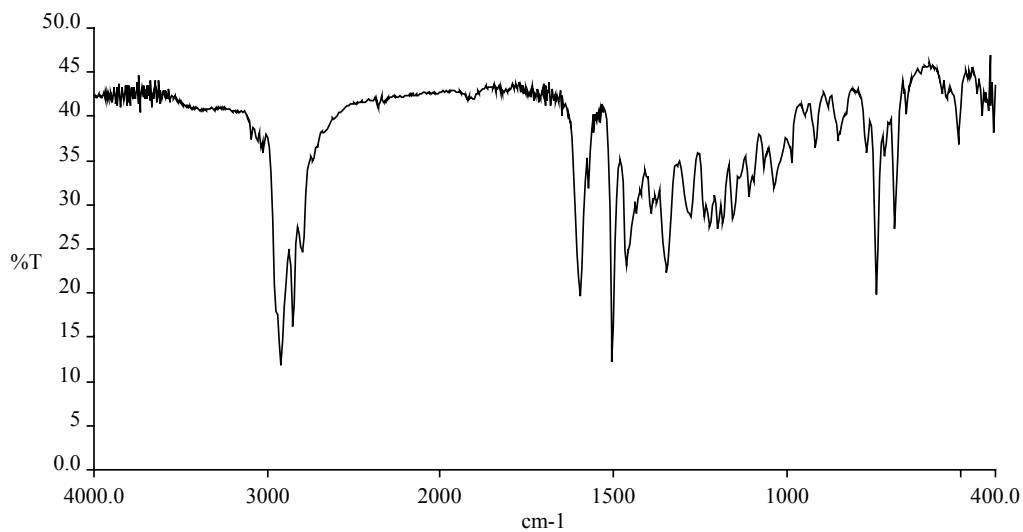
ESPECTROSCÒPIA DE MASSES (IQ)



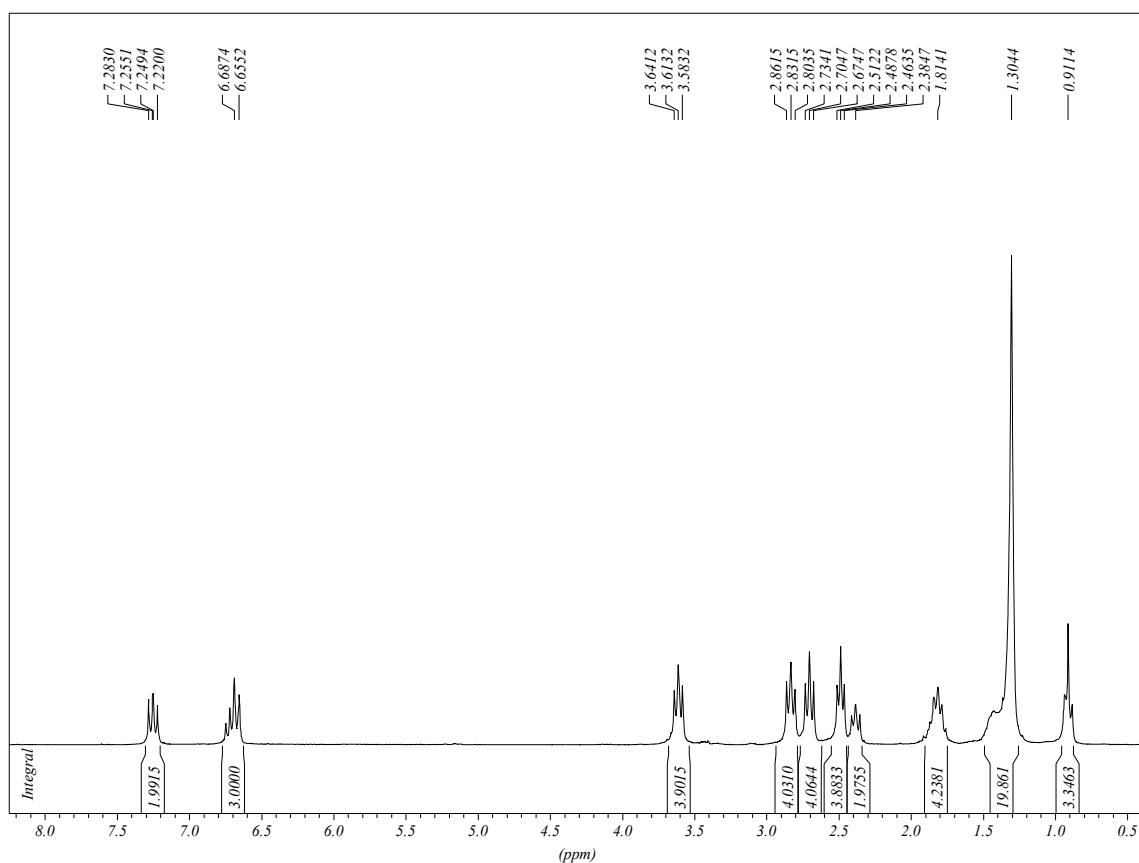
- 4-fenil-11-decil-1,7-ditia-4,11-diazaciclottedradecà (L7)



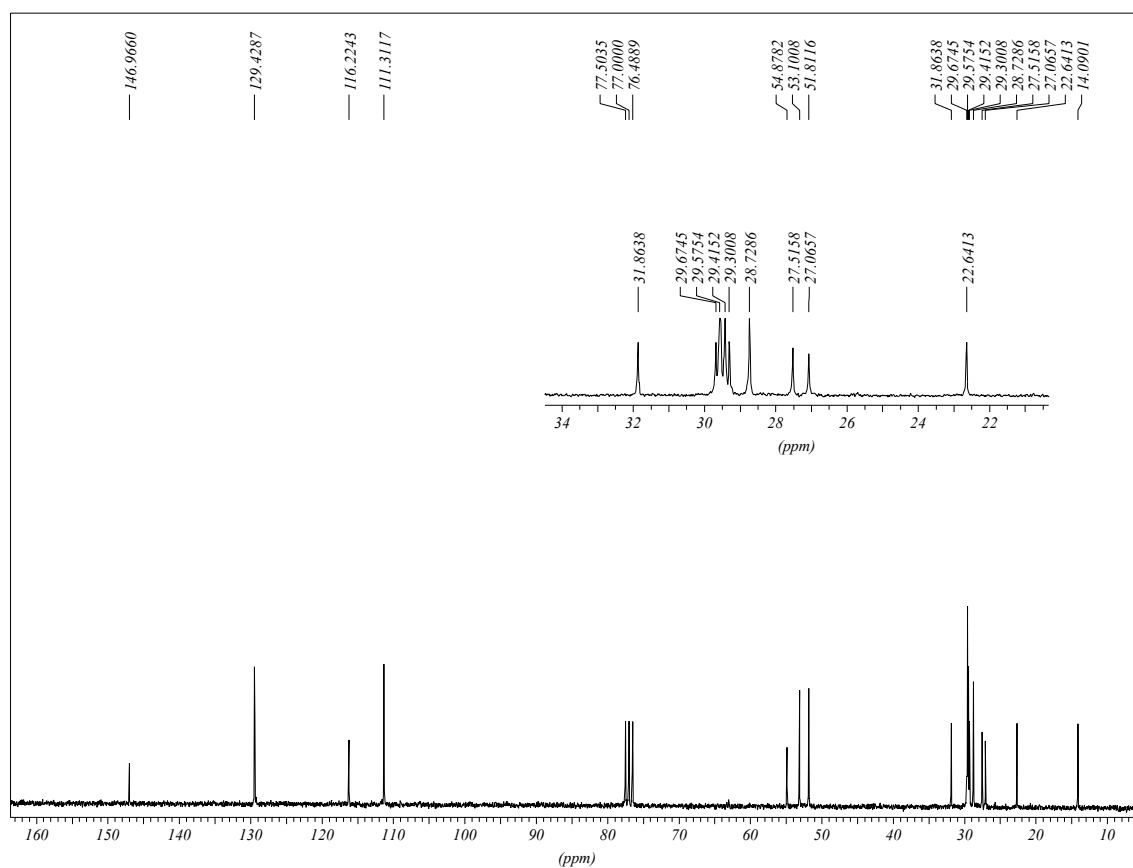
IR (KBr)



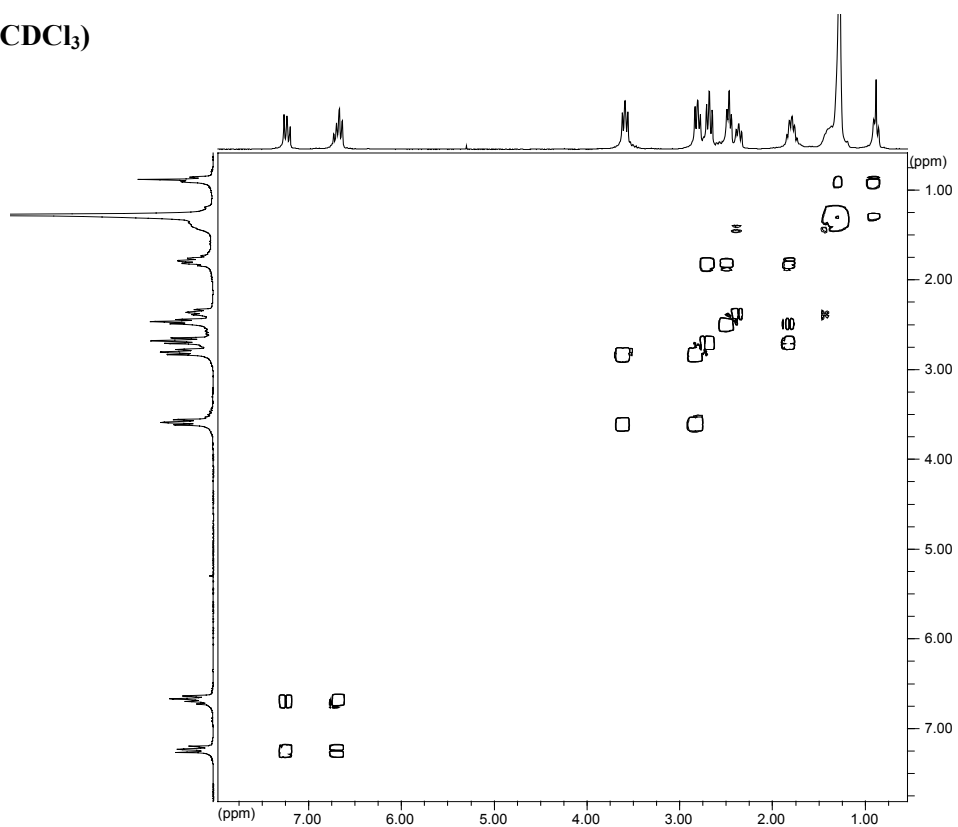
^1H RMN (CDCl_3)



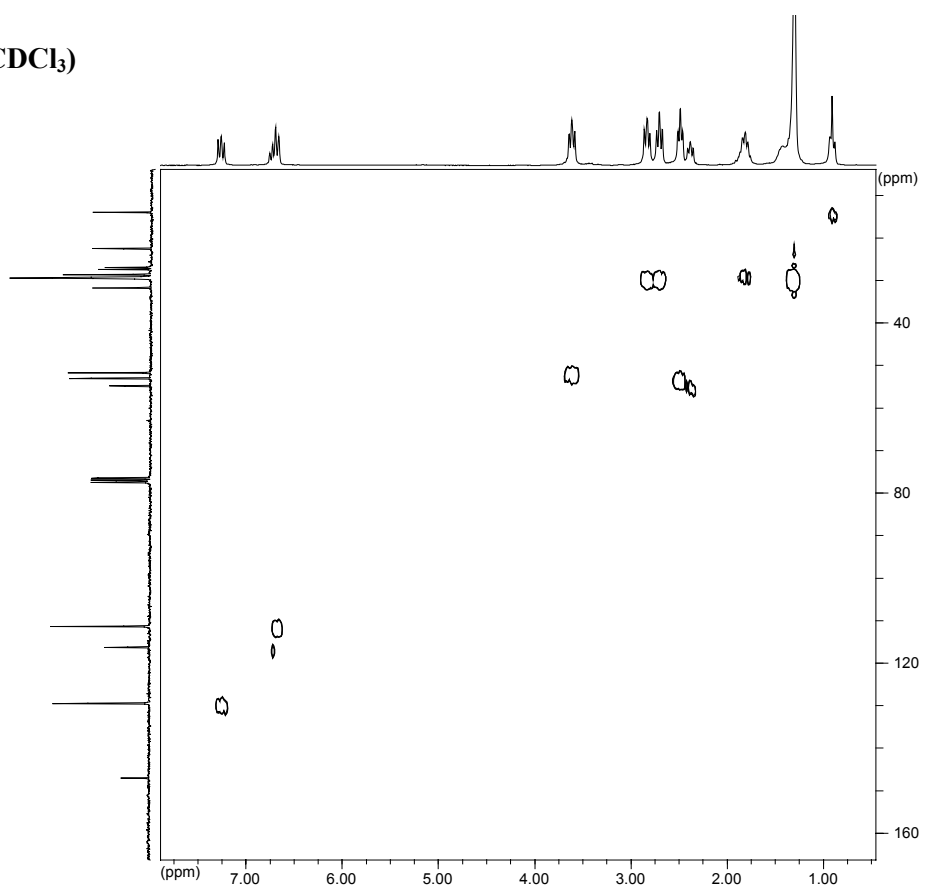
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



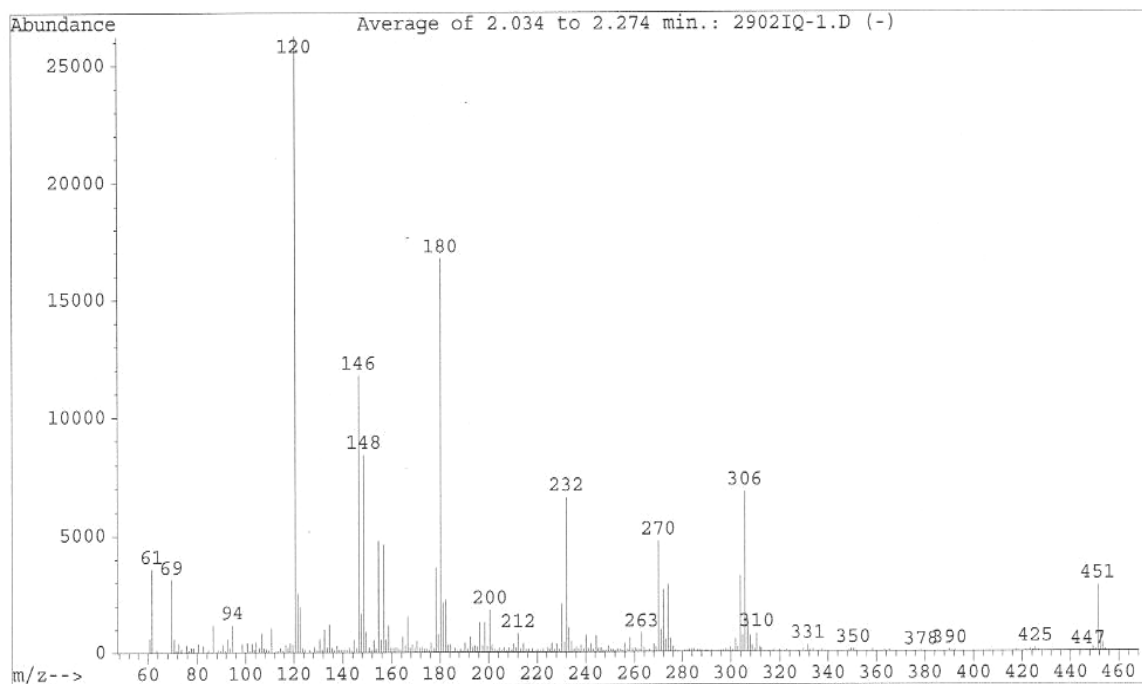
COSY (CDCl₃)



HMQC (CDCl₃)

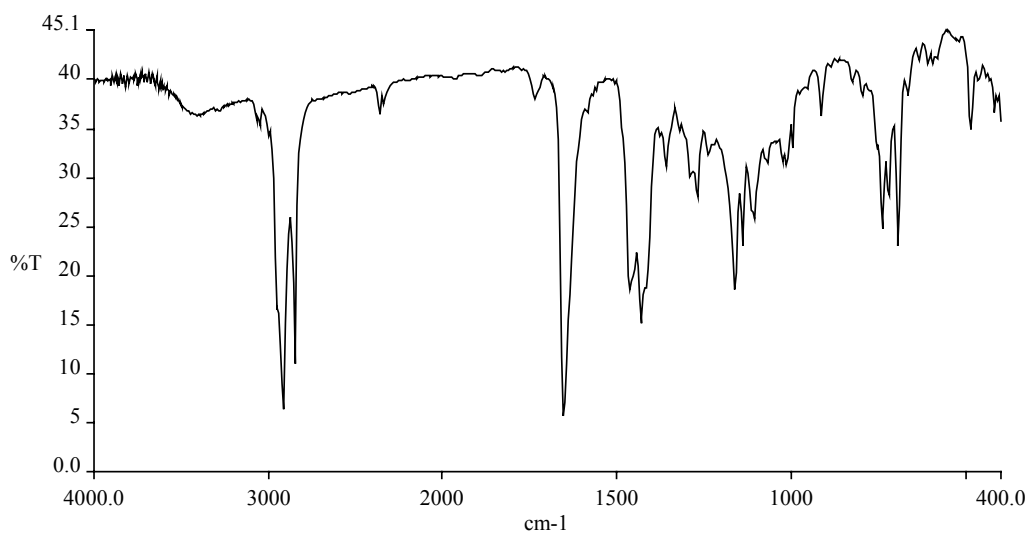
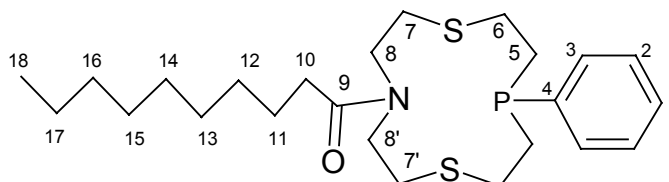


ESPECTROSCÒPIA DE MASSES (IQ)

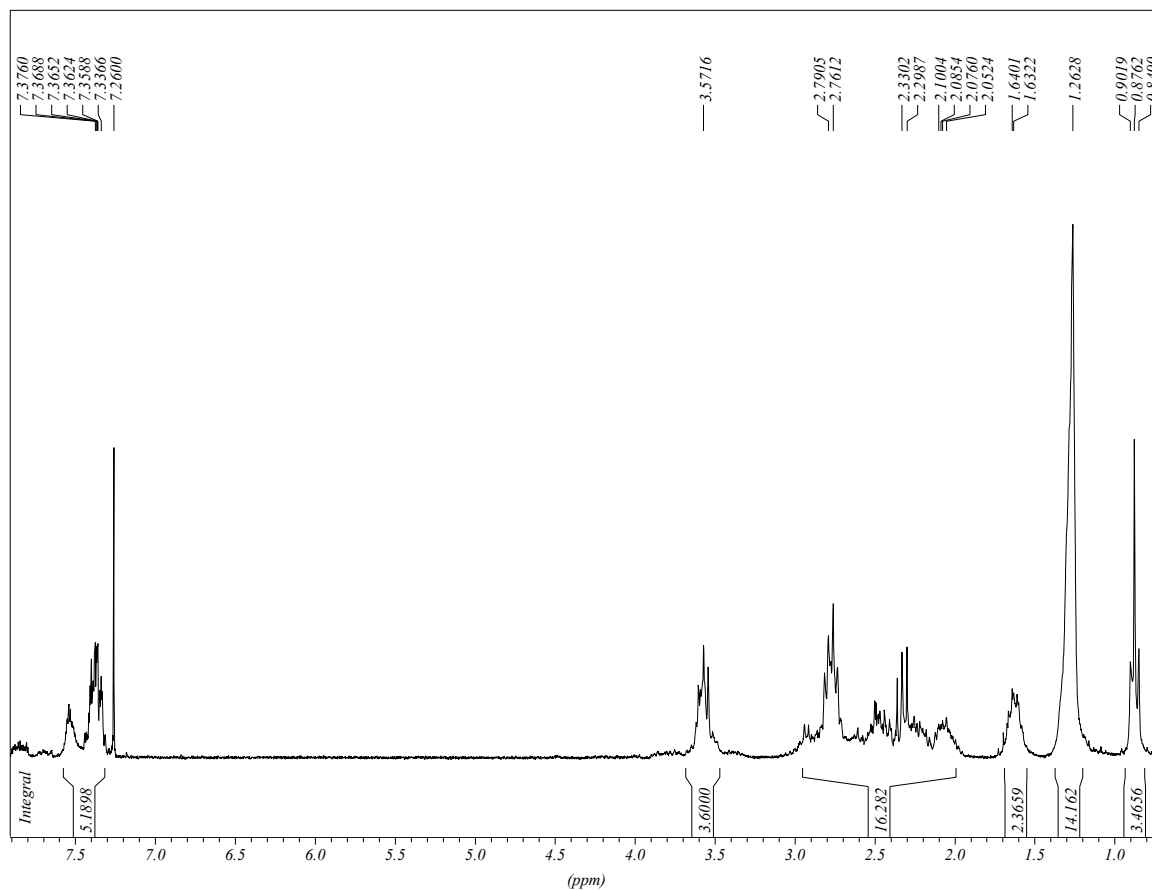


▪ **4-fenil-10-decanoil-1,7-ditia-4-fosfa-10-azaciclododecà (L10)**

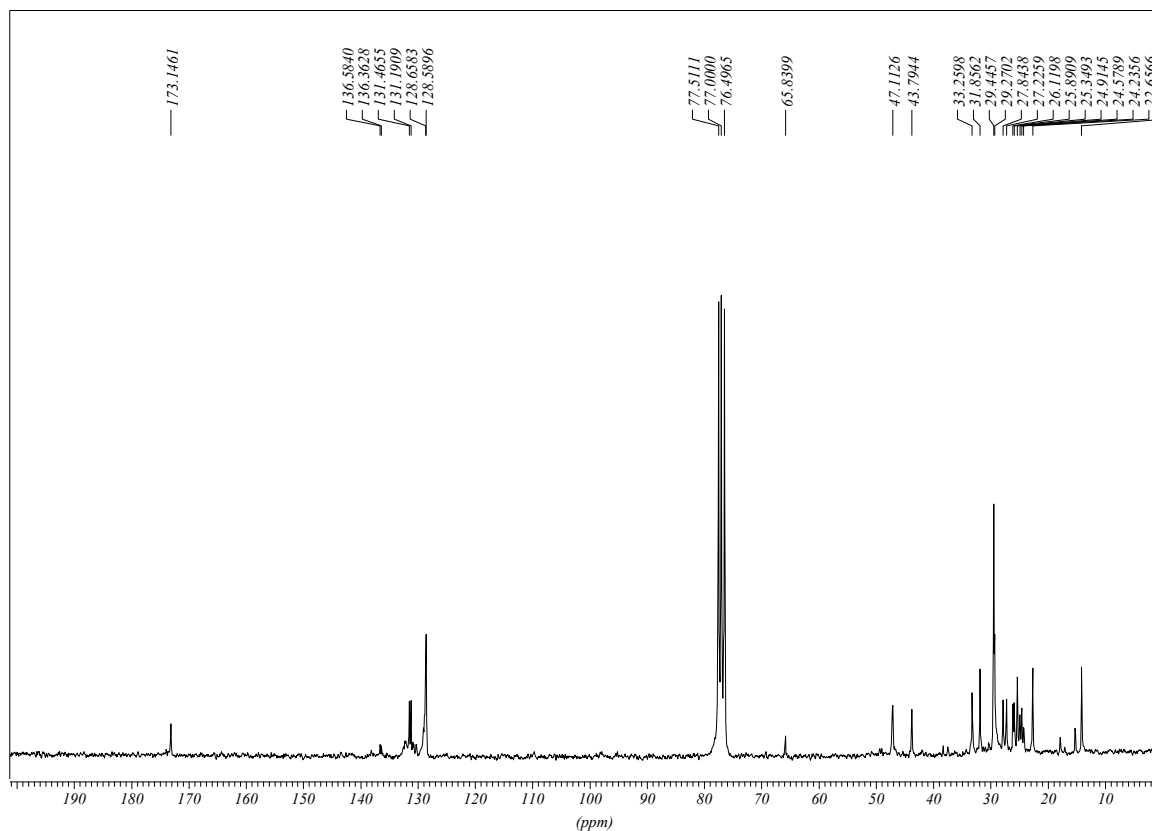
IR (NaCl)



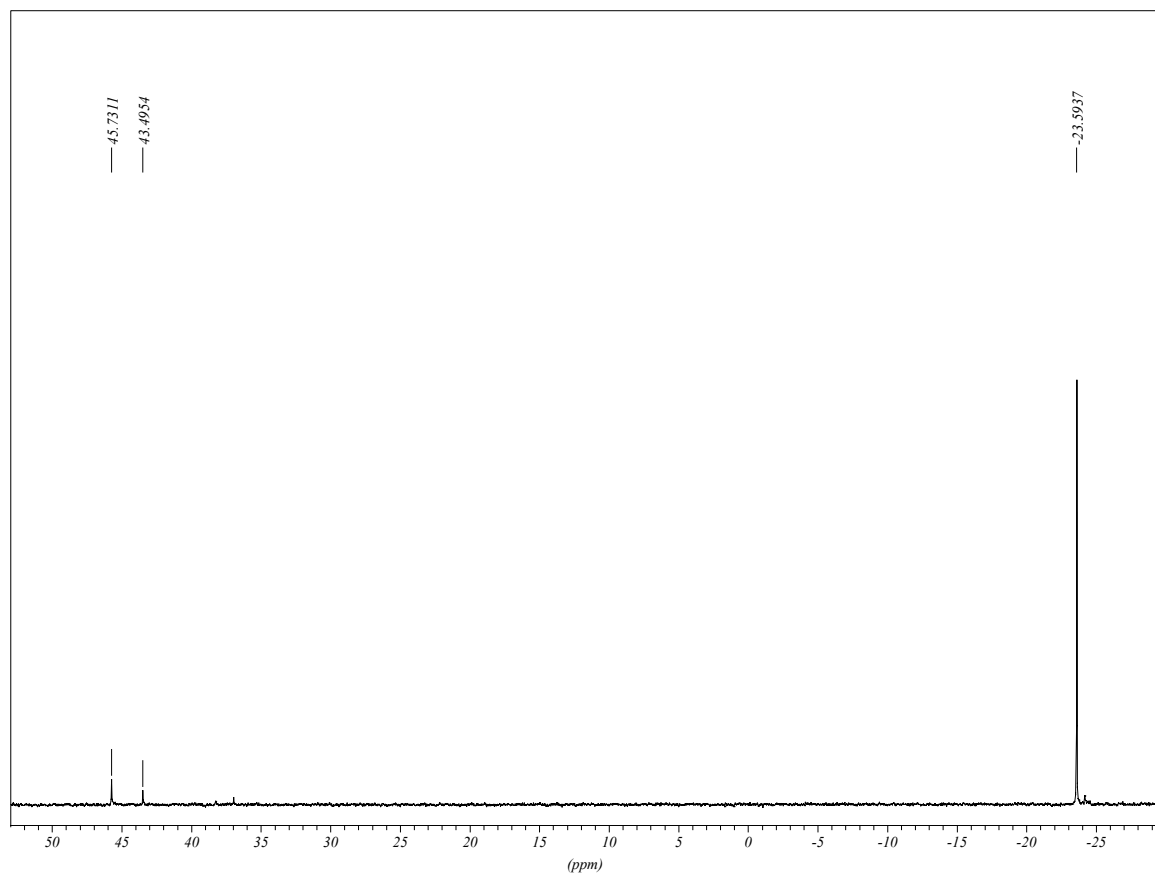
^1H RMN (CDCl_3)



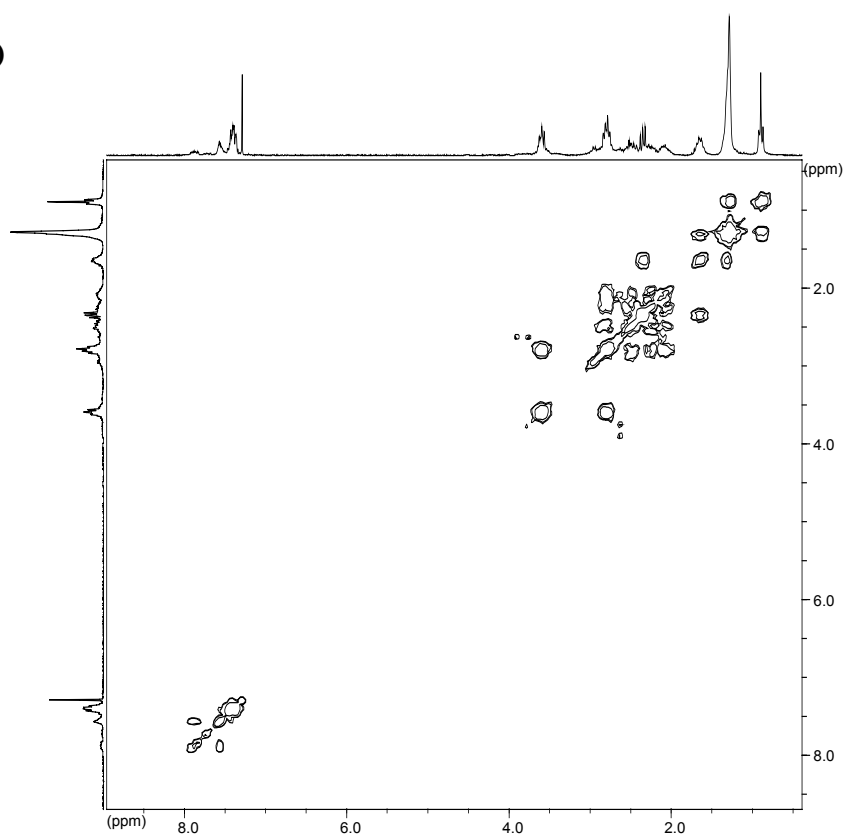
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



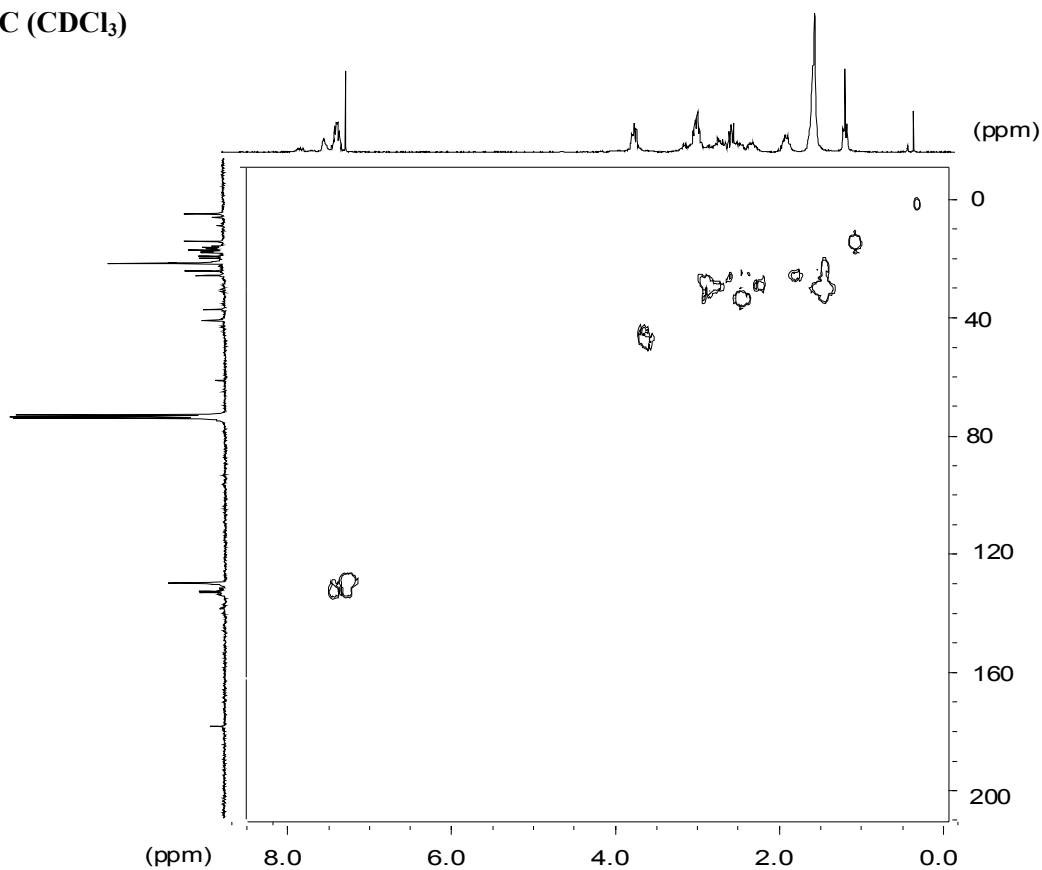
$^{31}\text{P}\{^1\text{H}\}$ RMN (CDCl_3)



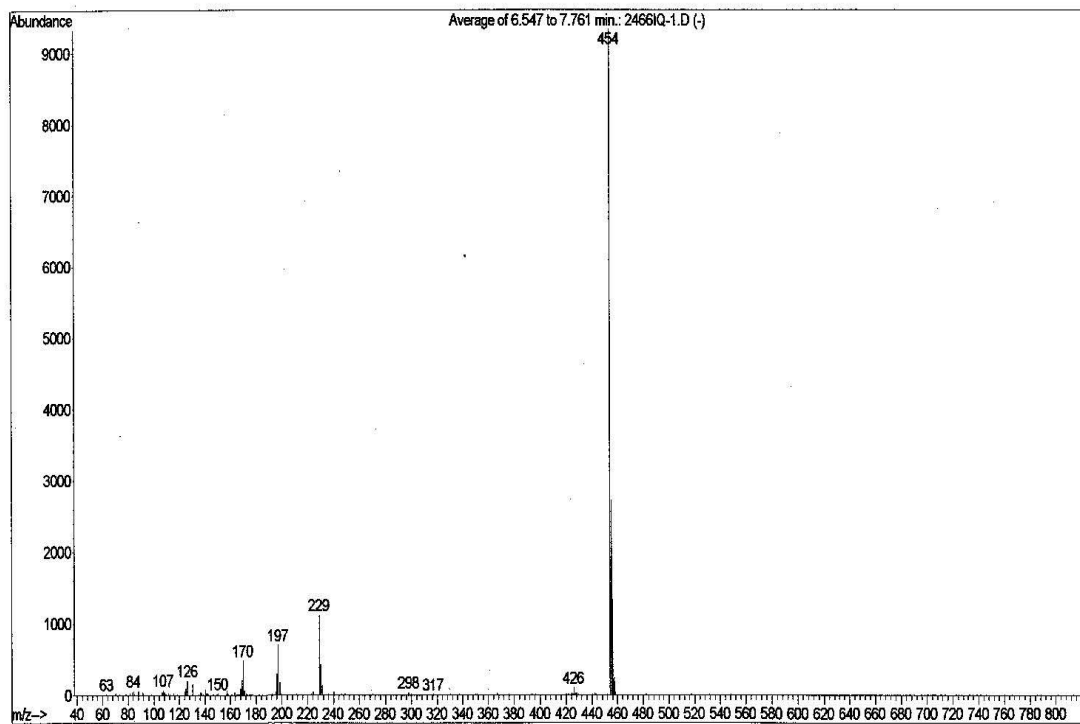
COSY (CDCl_3)



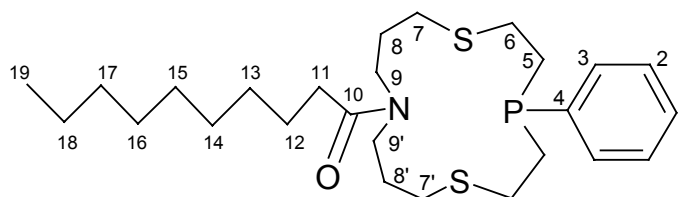
HMQC (CDCl₃)



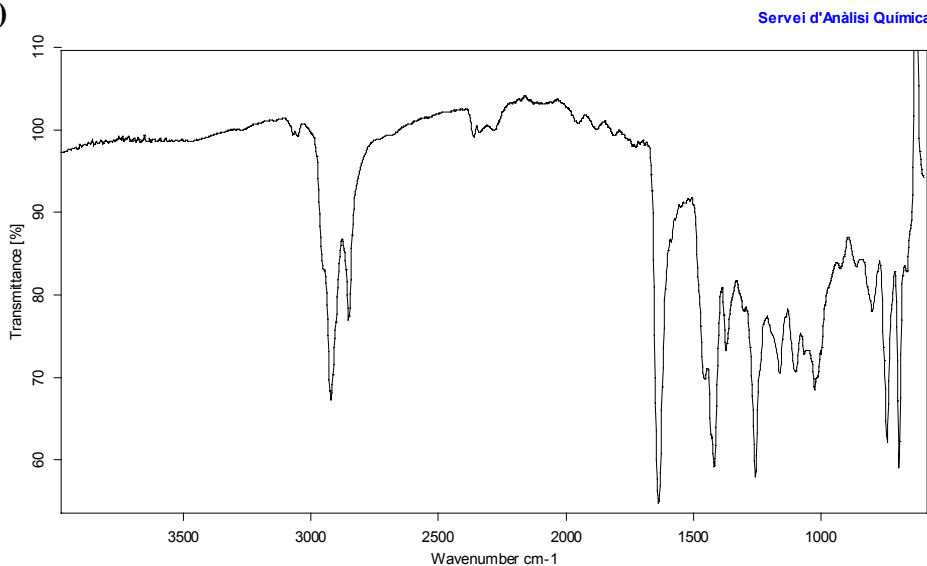
ESPECTROSCÒPIA DE MASSES (IQ)



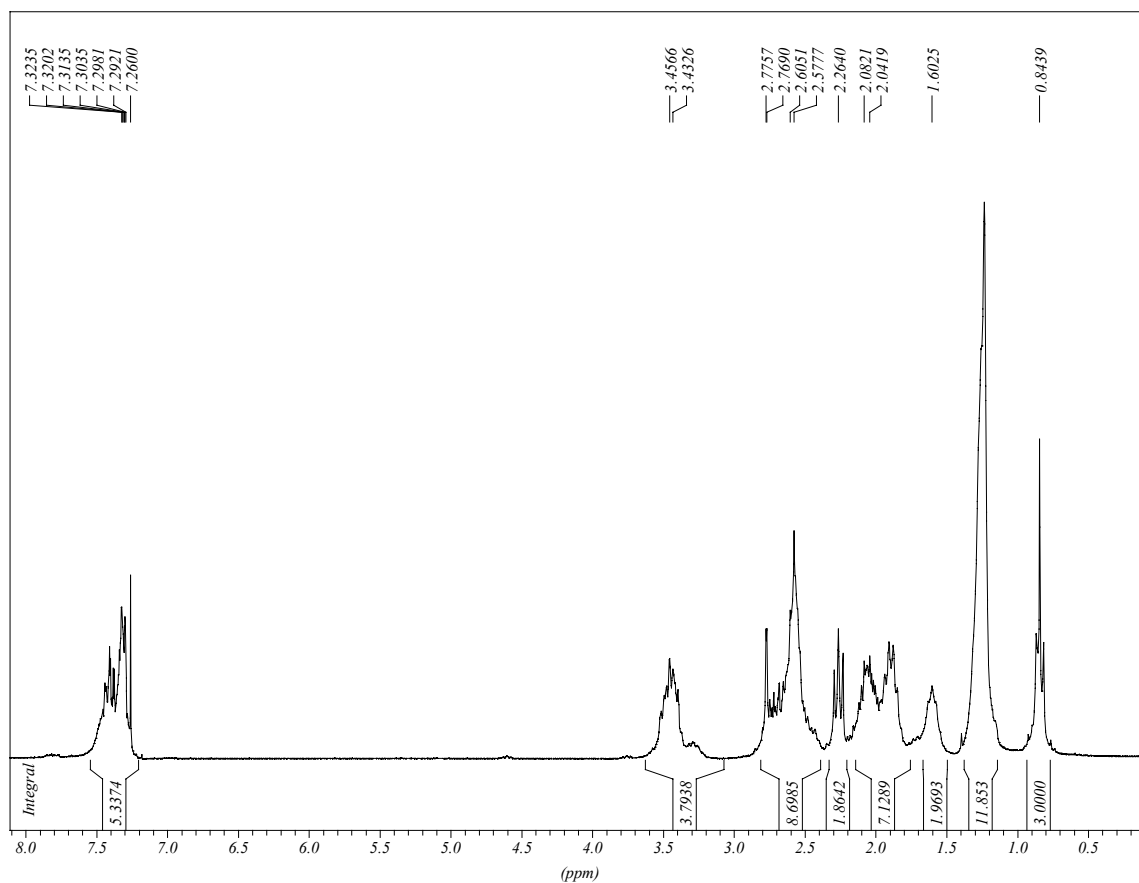
▪ 4-fenil-11-decanoil-1,7-ditia-4-fosfa-11-azaciclo-tetradecà (L11)



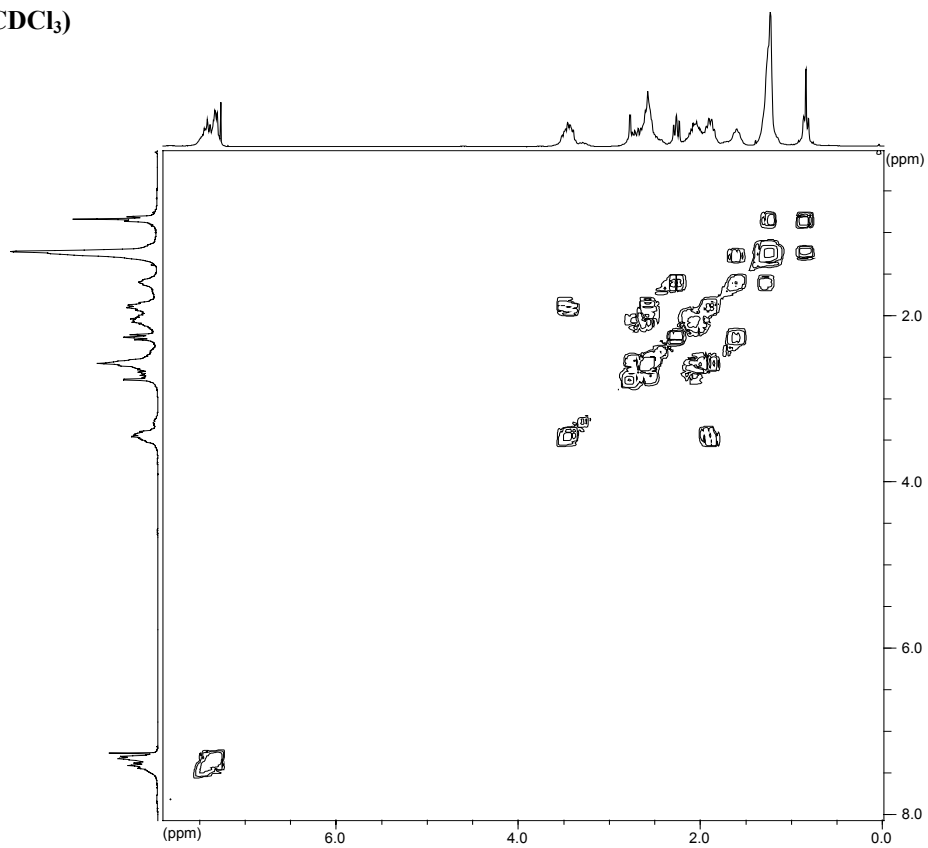
IR (ATR)



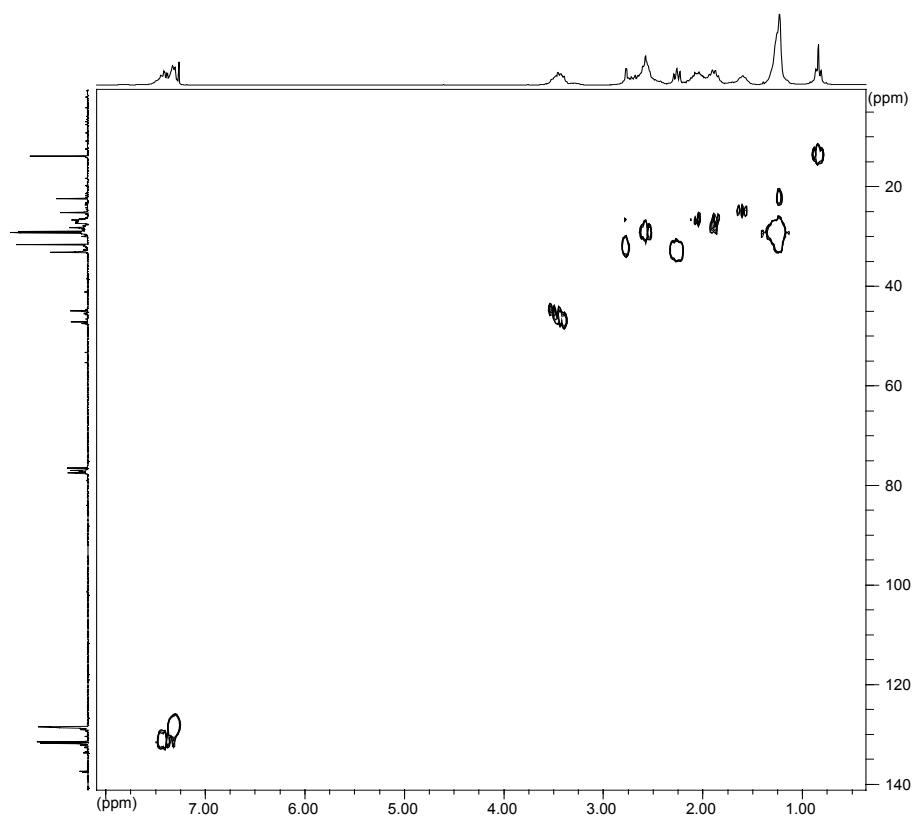
¹H RMN (CDCl₃)



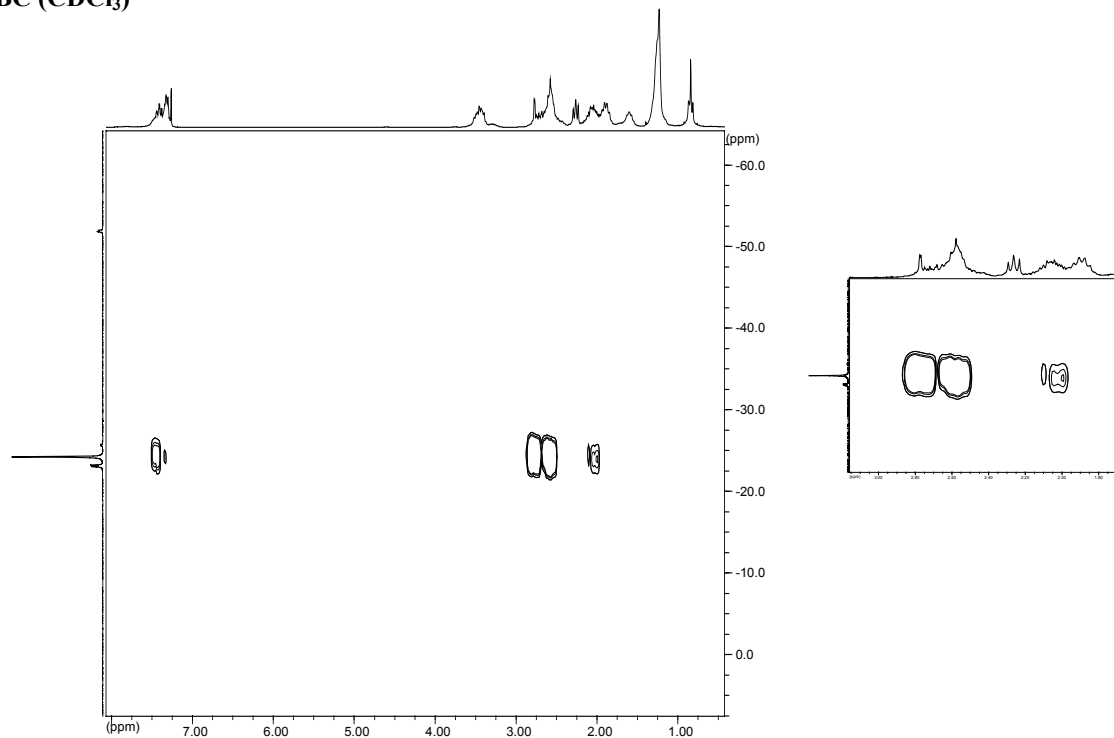
COSY (CDCl₃)



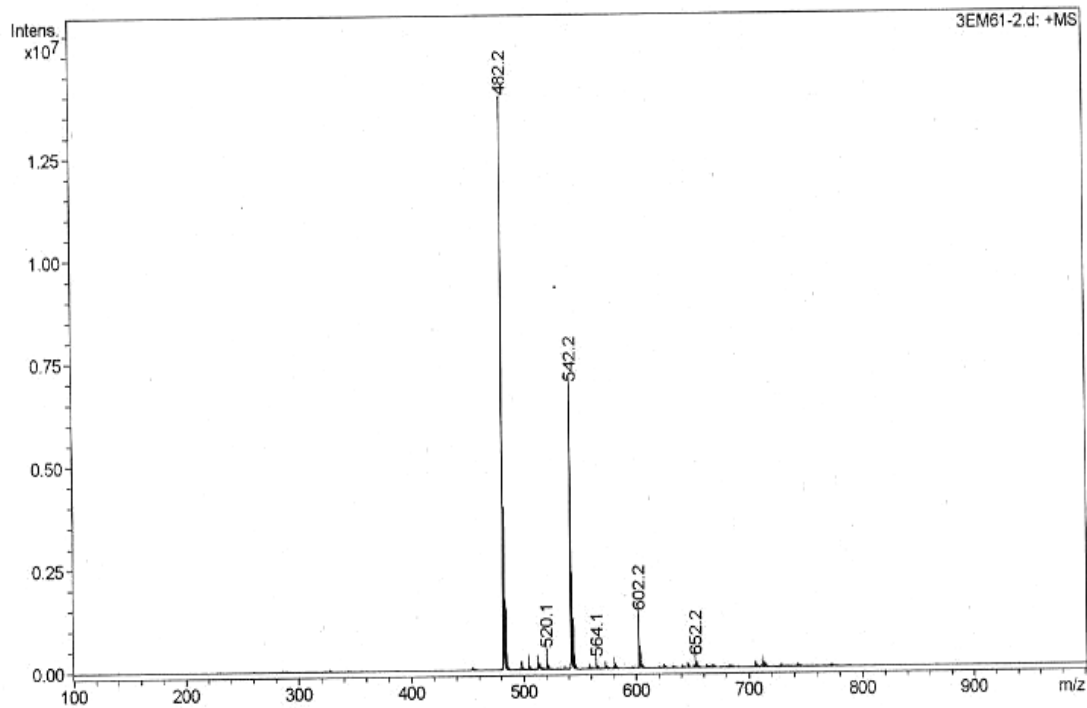
HMQC (CDCl₃)



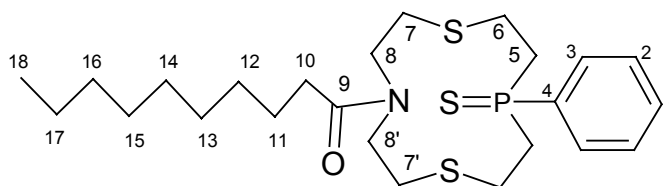
HMBC (CDCl₃)



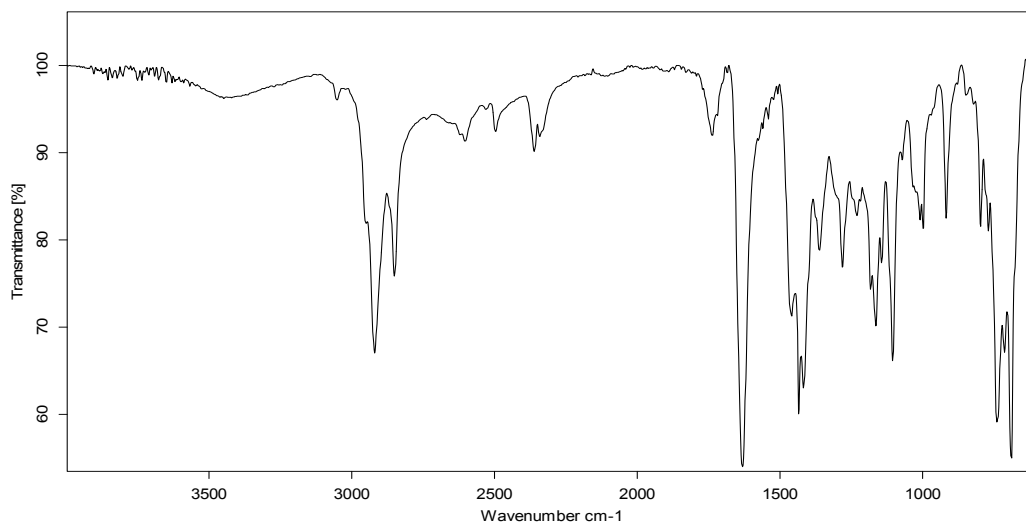
ESPECTROSCÒPIA DE MASSES (ESI(+)-IT) (10 ppm CHCl₃-MeOH 1:1, 0.1% HCOOH)



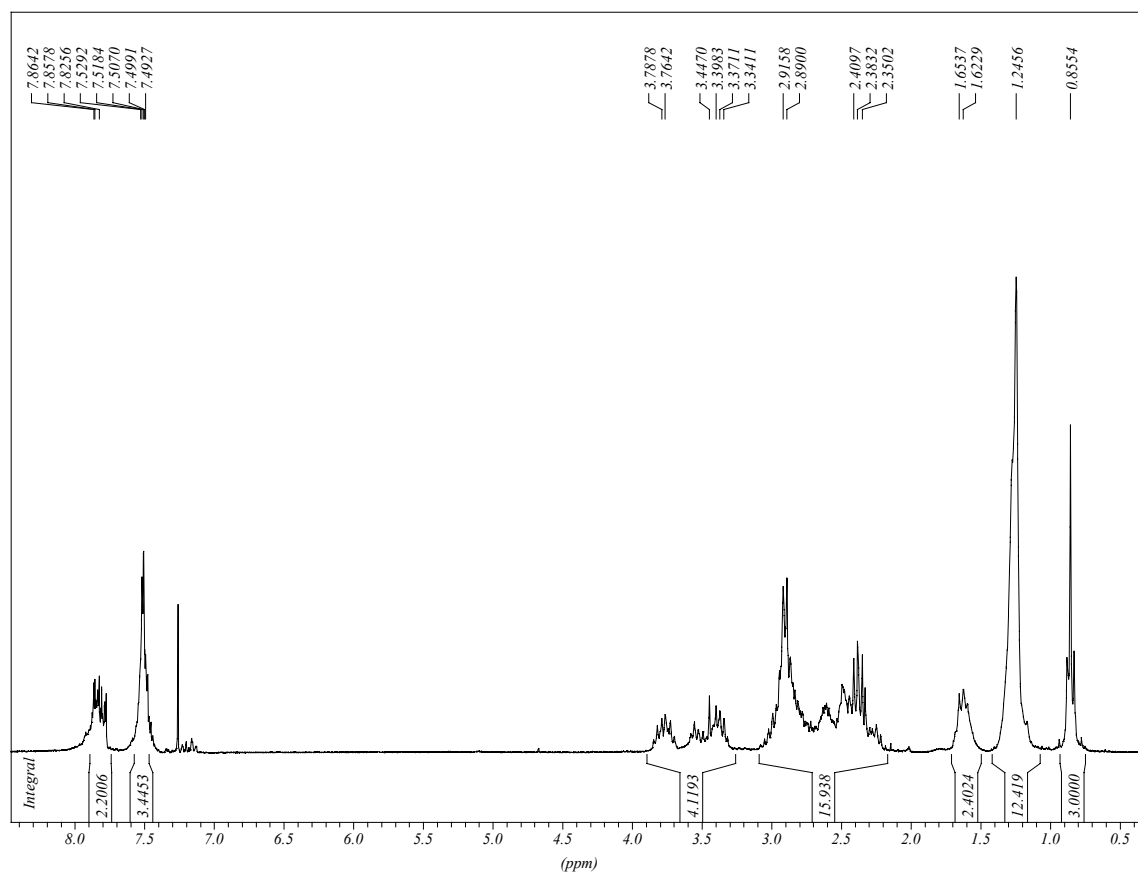
▪ 4-fenil-10-decanoil-1,7-ditia-4-fosfa-10-azaciclododecà-4-sulfur (L12)



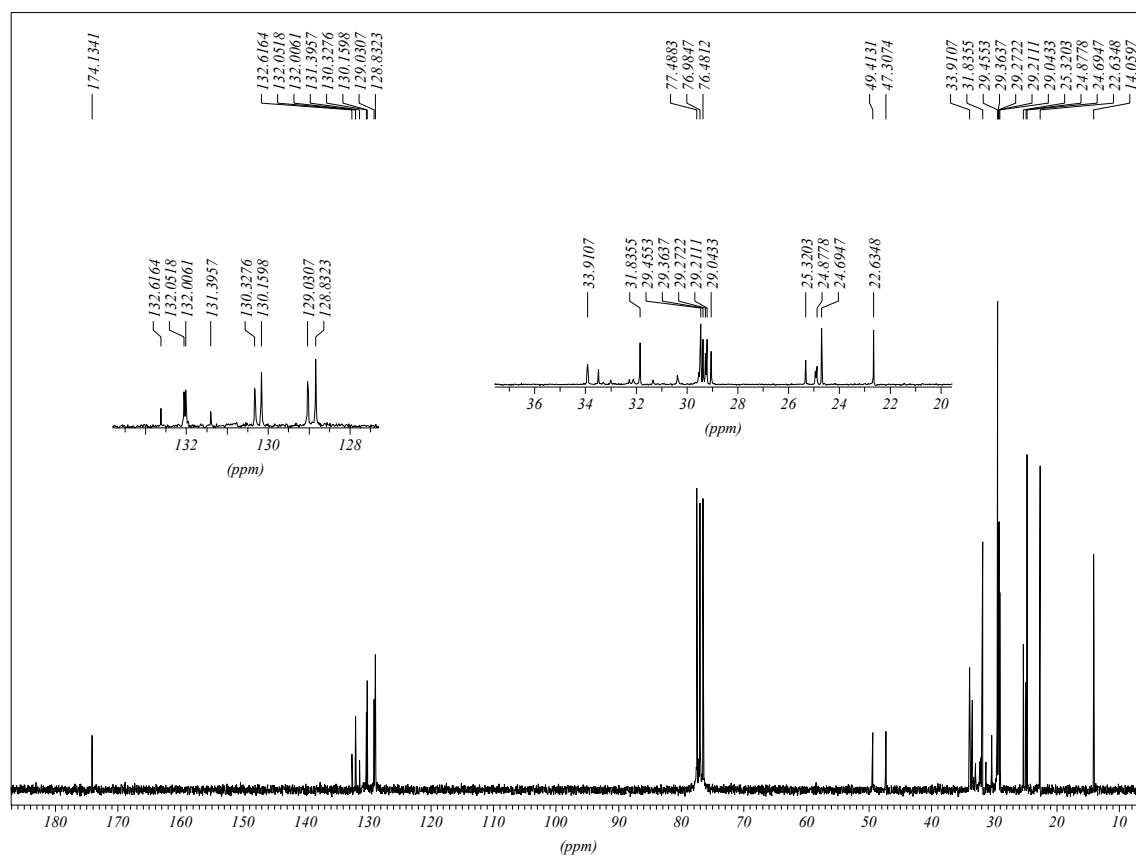
IR (ATR)



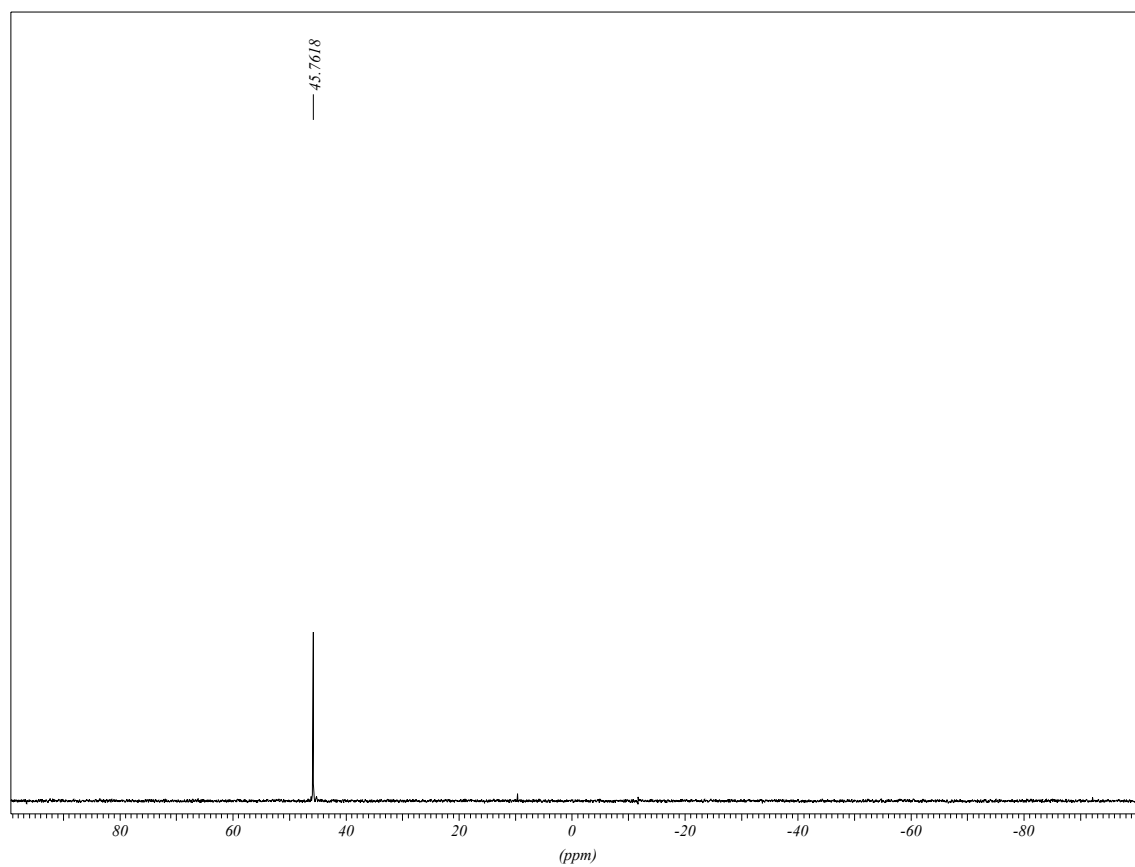
¹H RMN (CDCl₃)



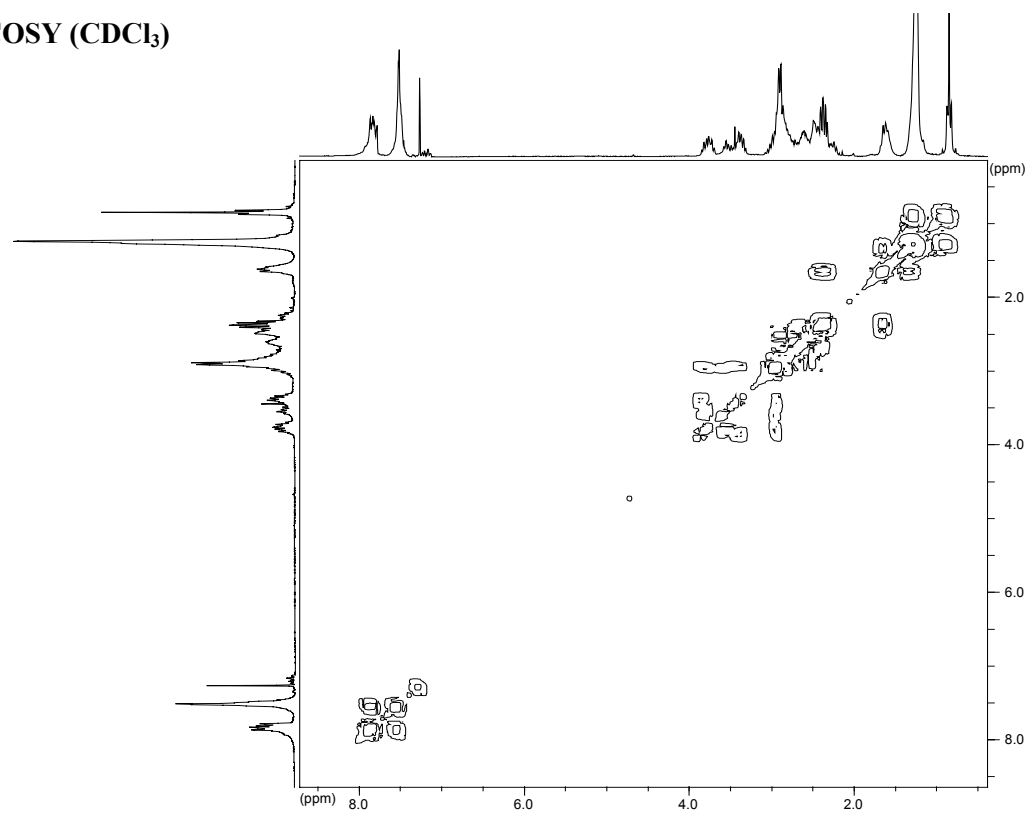
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



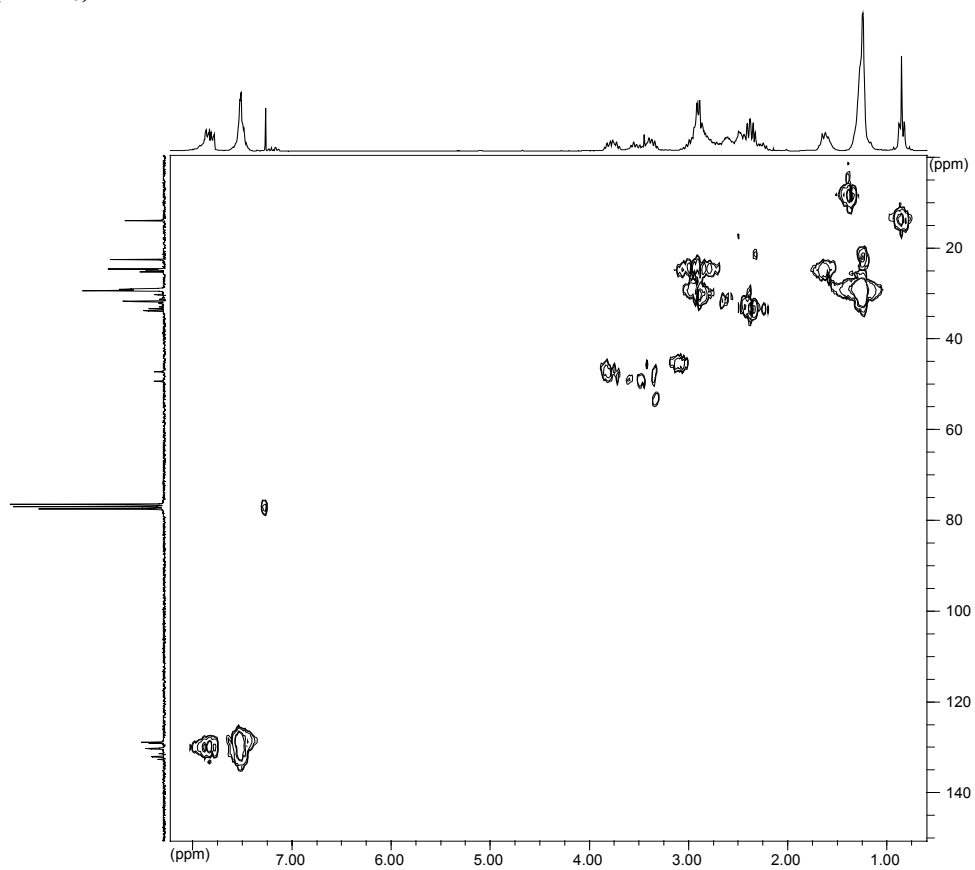
$^{31}\text{P}\{^1\text{H}\}$ RMN (CDCl_3)



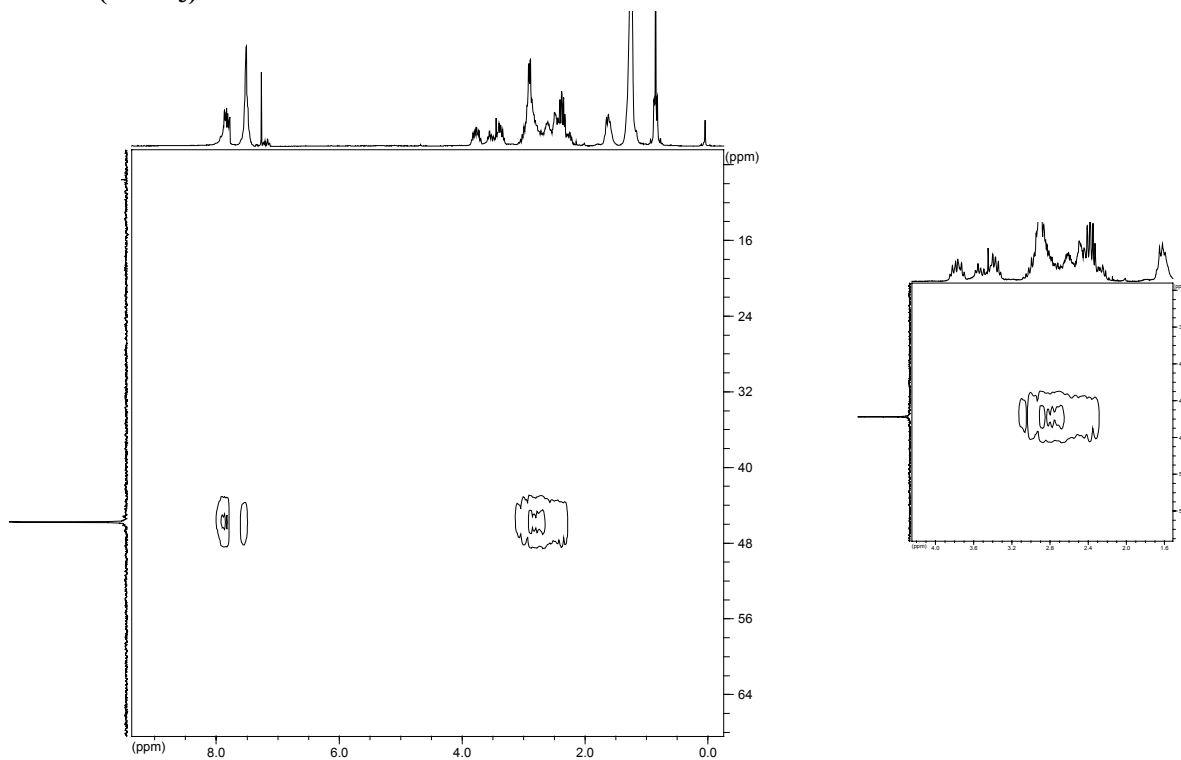
COSY (CDCl₃)



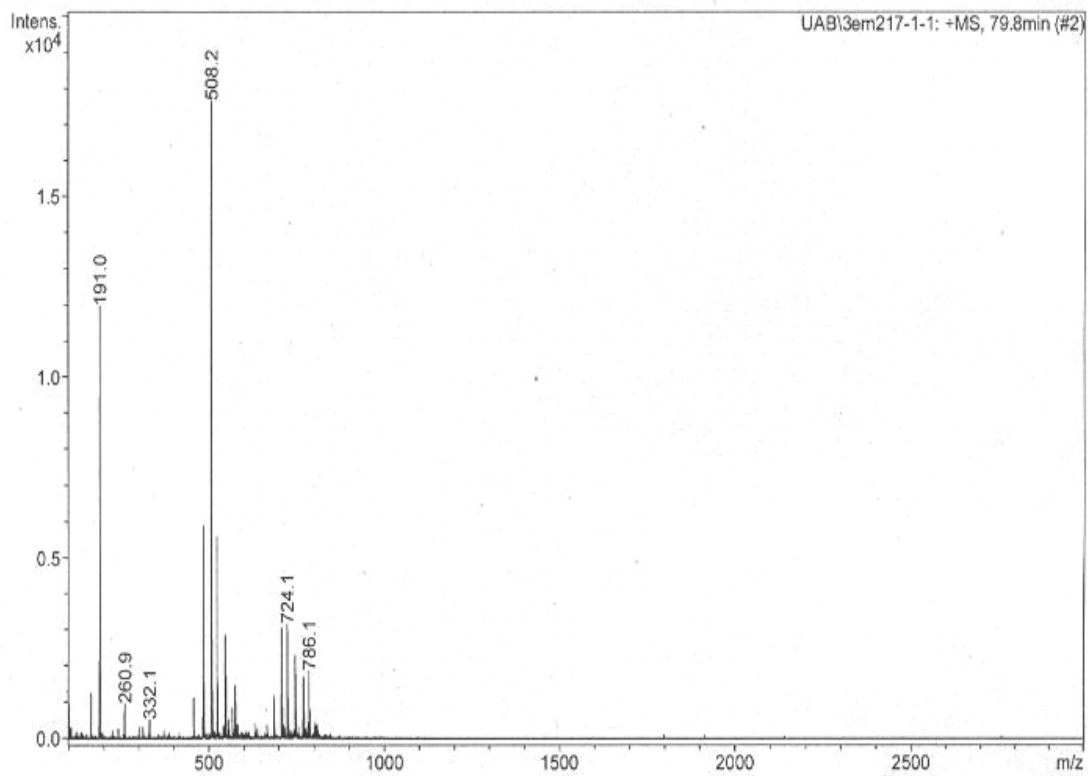
HMQC (CDCl₃)



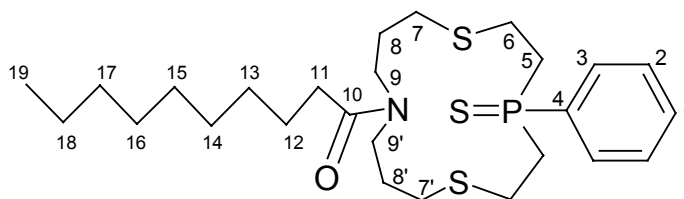
HMBC (CDCl₃)



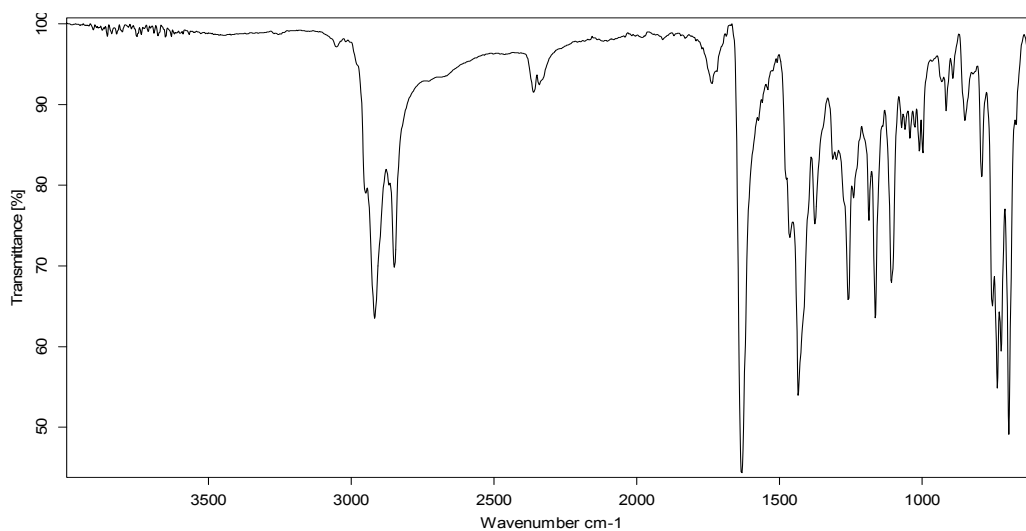
ESPECTROSCÒPIA DE MASSES (ESI(+)-IT) (10 ppm en MeOH)



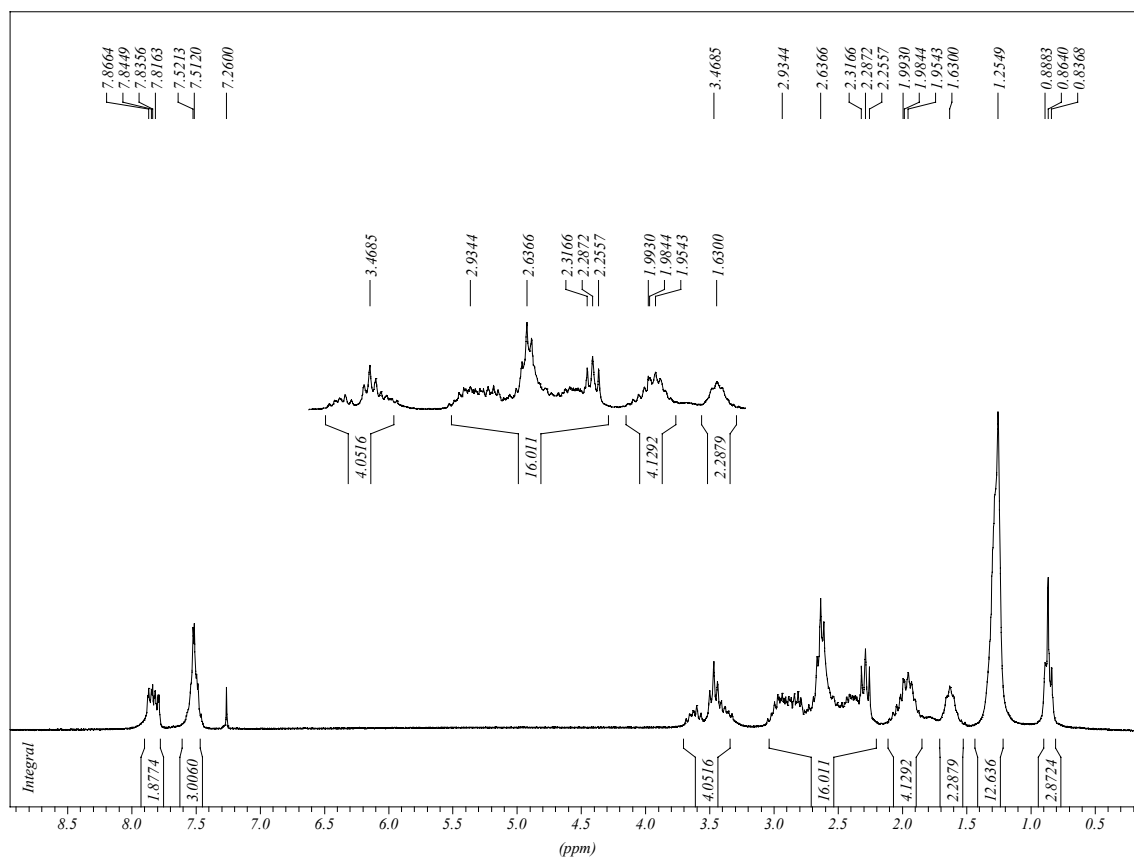
▪ 4-fenil-11-decanoil-1,7-ditia-4-fosfa-11-azaciclo-tetradecà-4-sulfur (L13)



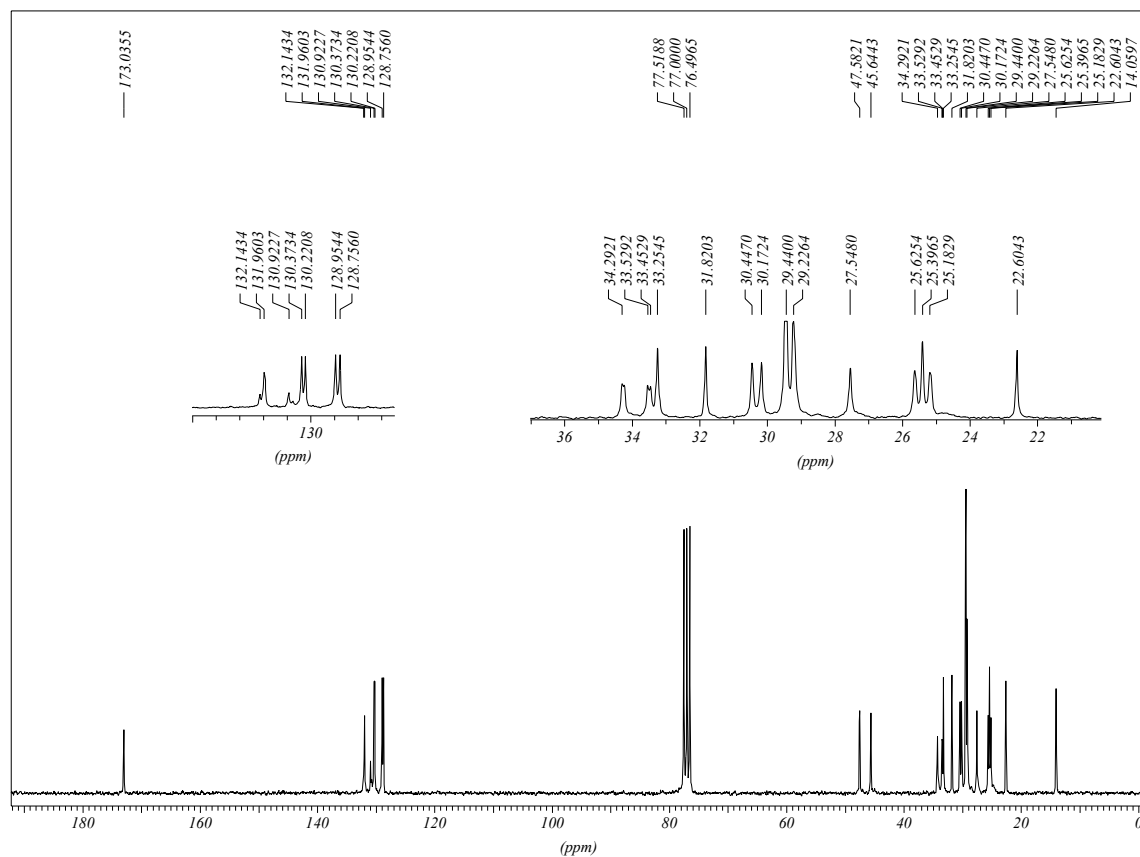
IR (ATR)



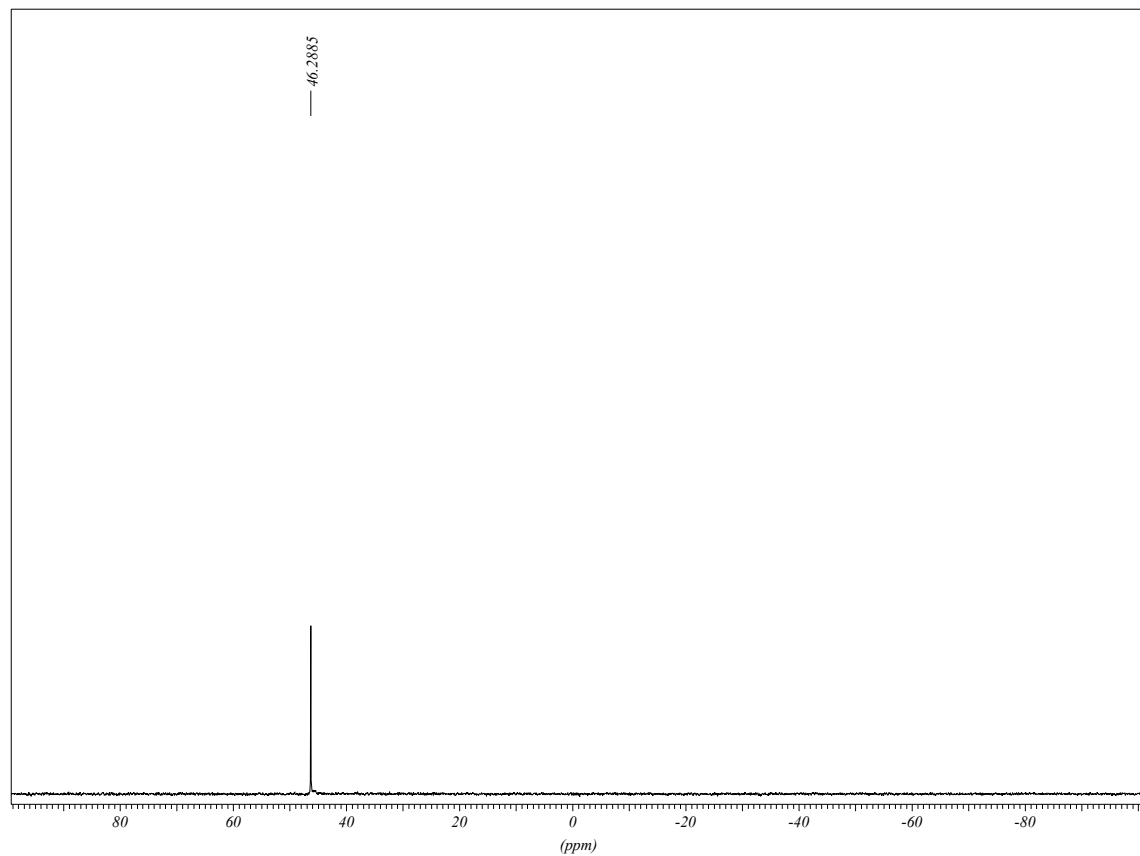
^1H RMN (CDCl_3)



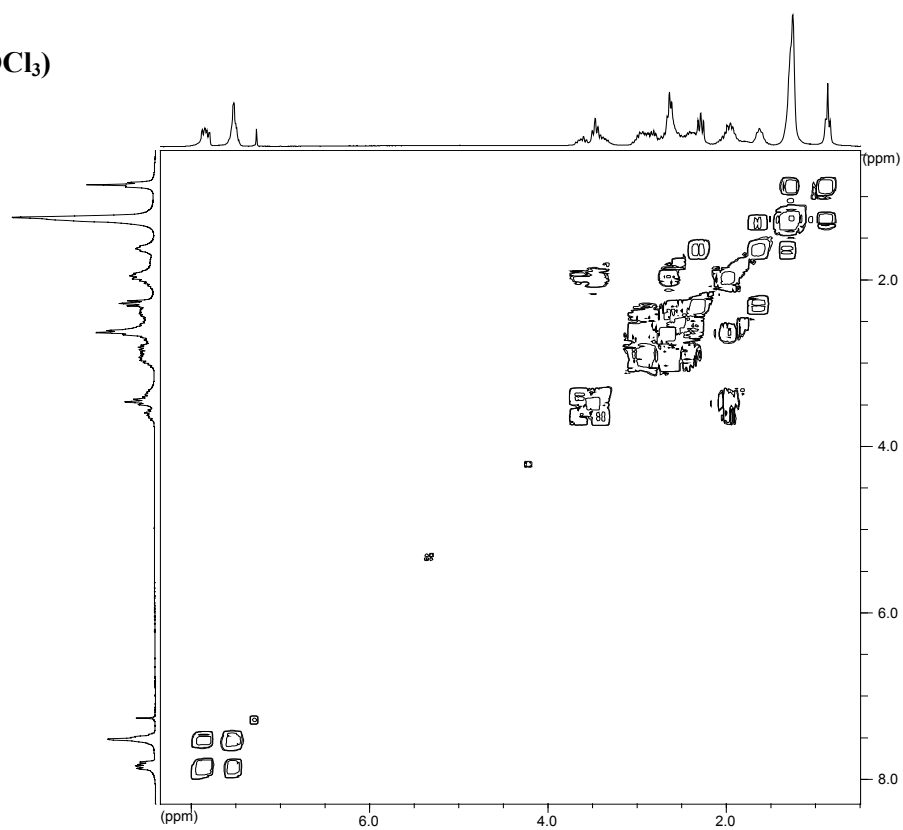
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



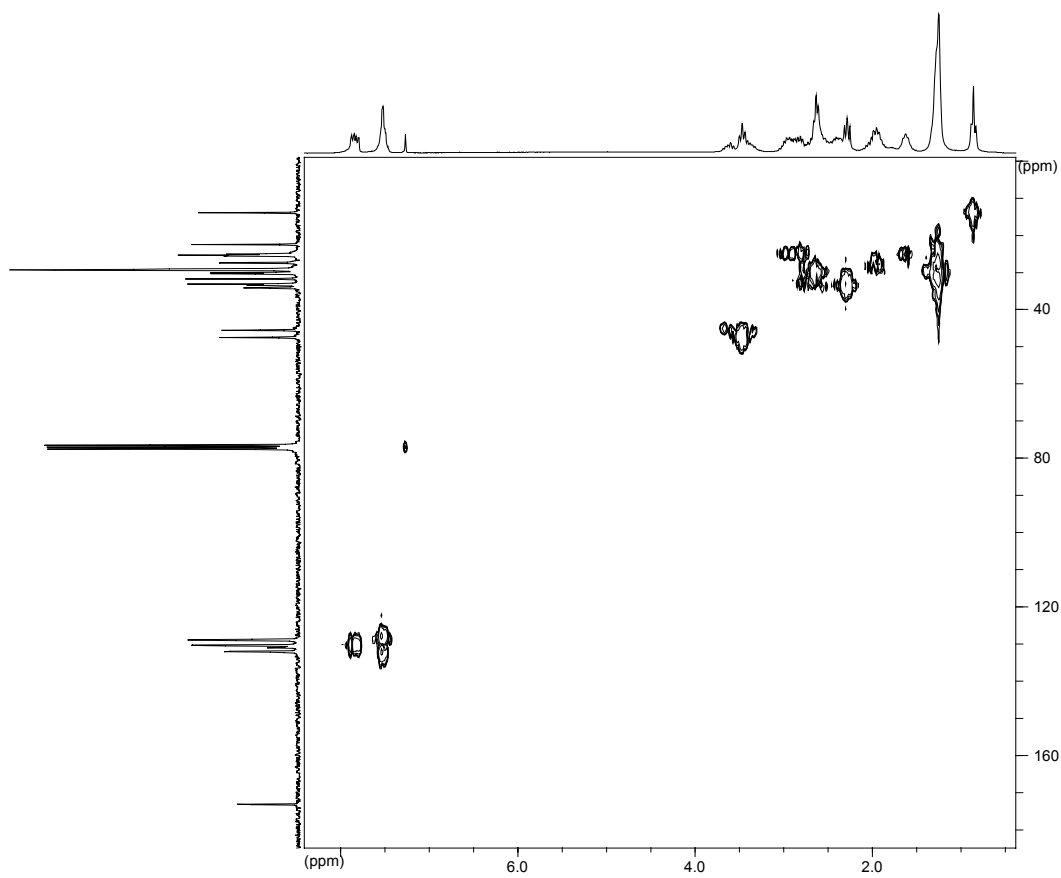
$^{31}\text{P}\{^1\text{H}\}$ RMN (CDCl_3)



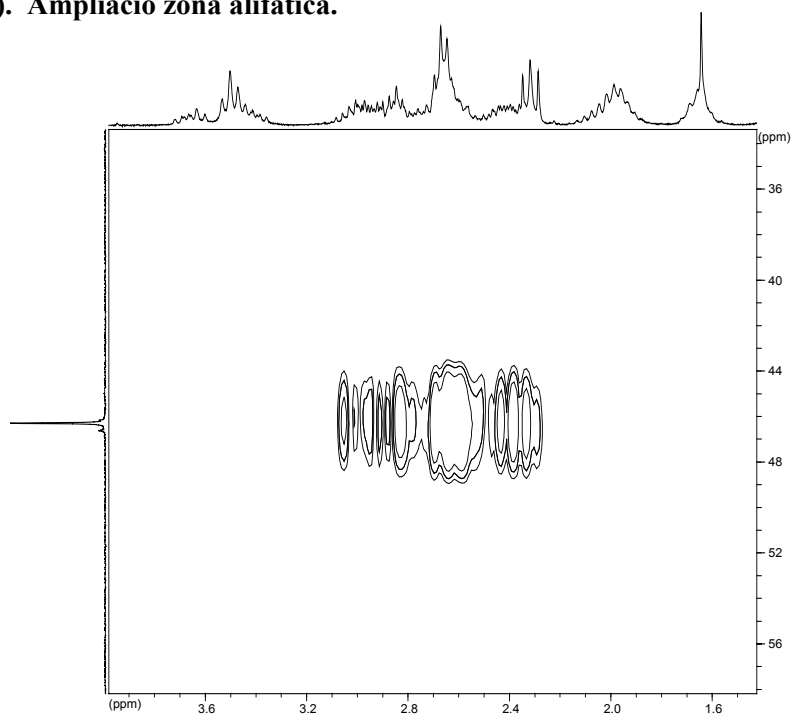
COSY (CDCl₃)



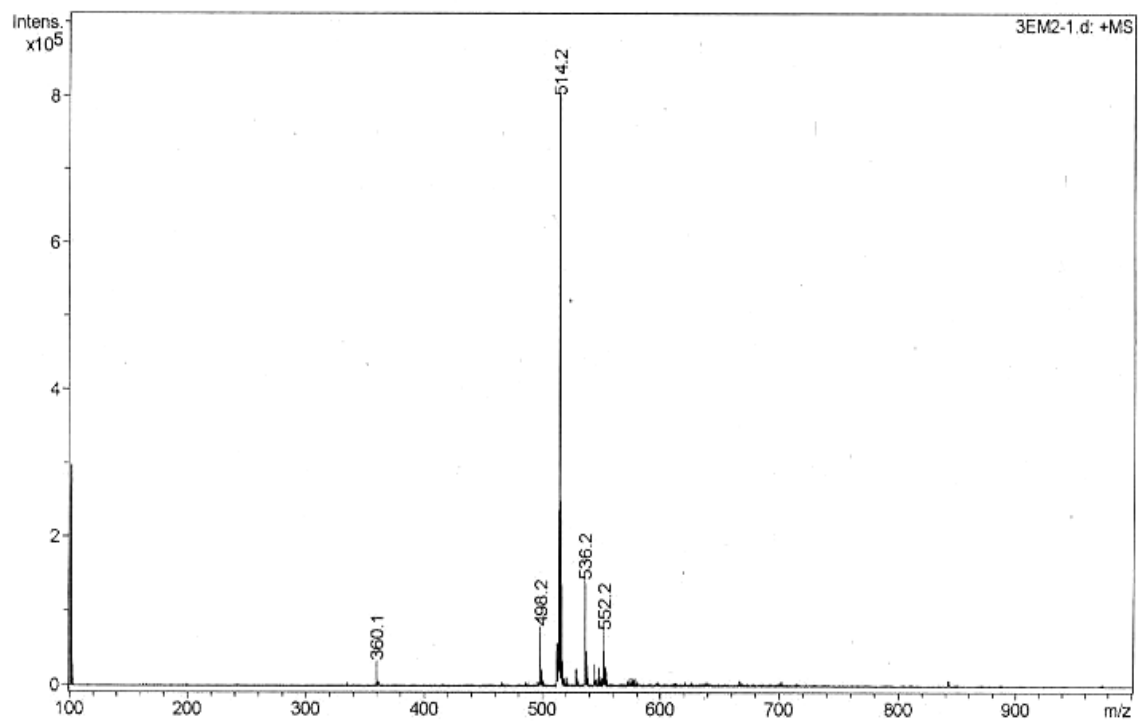
HMQC (CDCl₃)



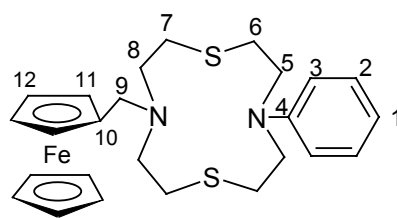
HMBC (CDCl₃). Ampliació zona alifàtica.



ESPECTROSCÒPIA DE MASSES (ESI(+)-IT) (10 ppm en CH₃CN)

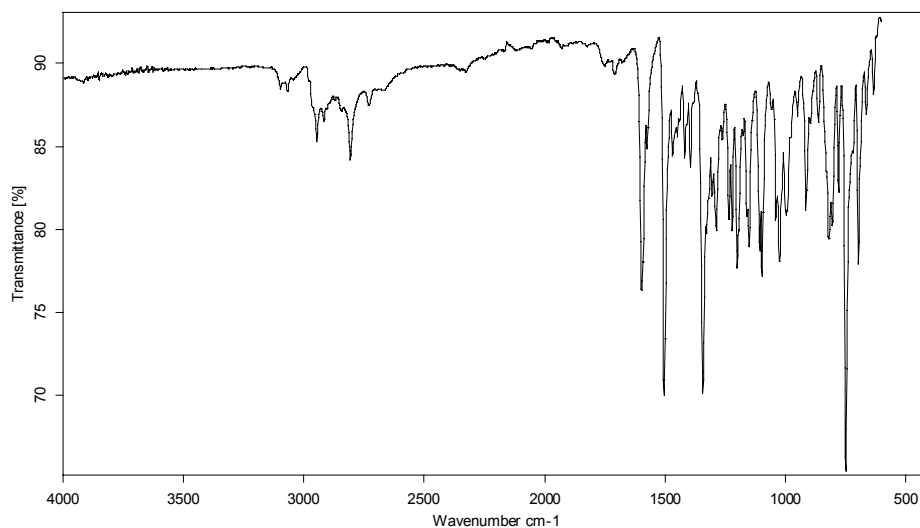


▪ (4-fenil-10-ilmetil-1,7-ditia-4,10-diazaciclododecà)ferrocè (L14)

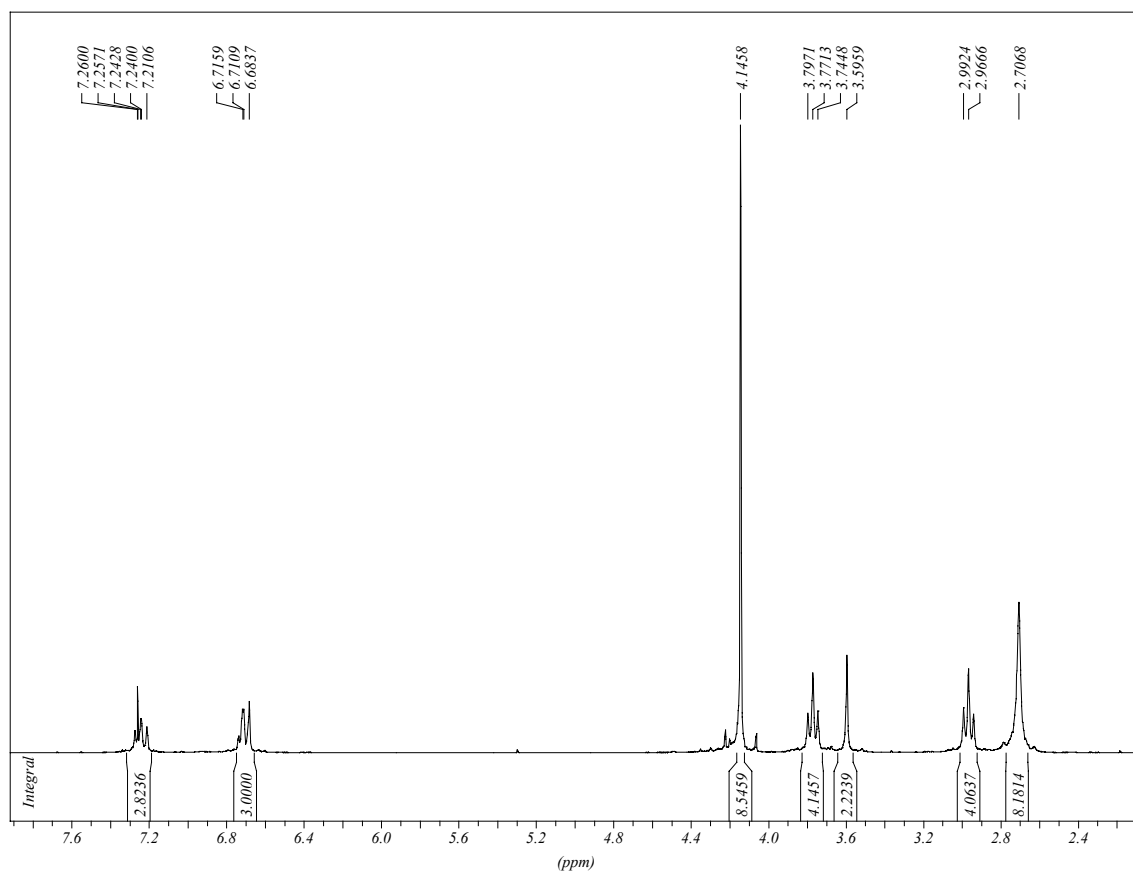


IR (ATR)

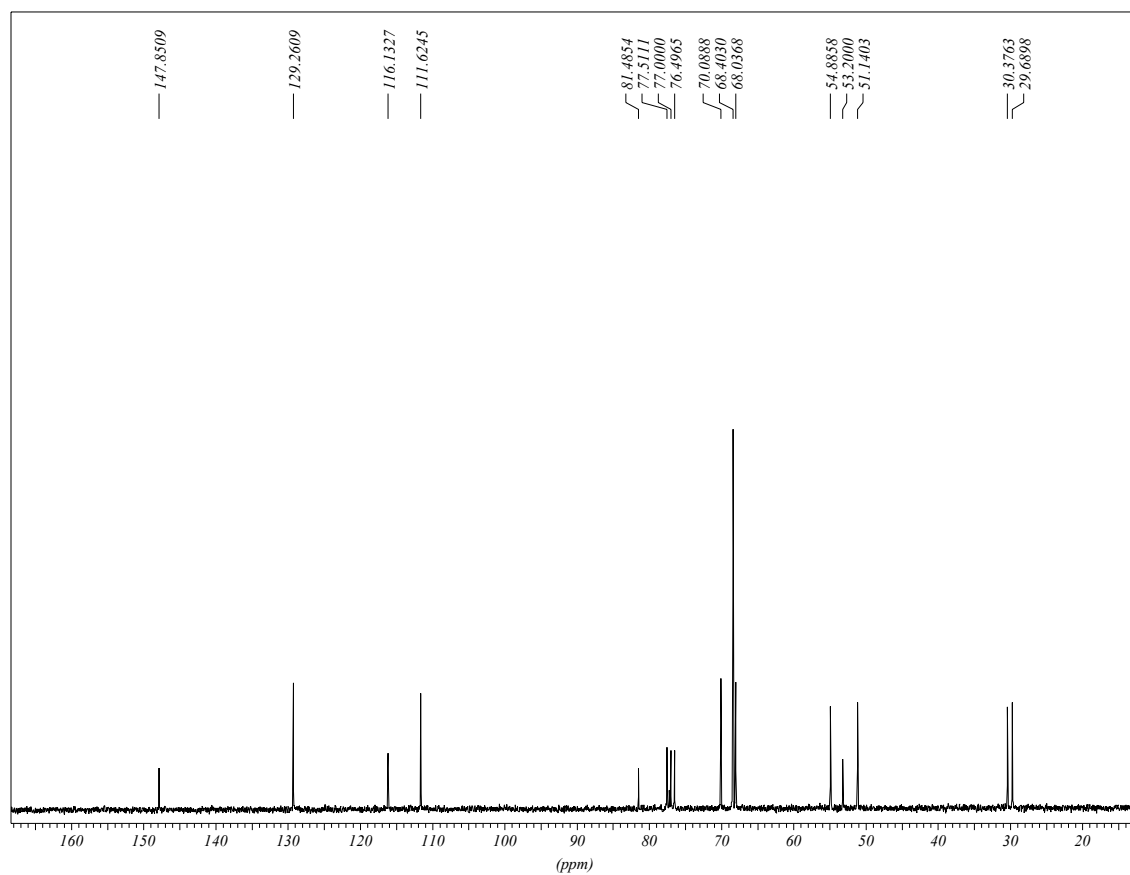
Servei d'Anàlisi Química



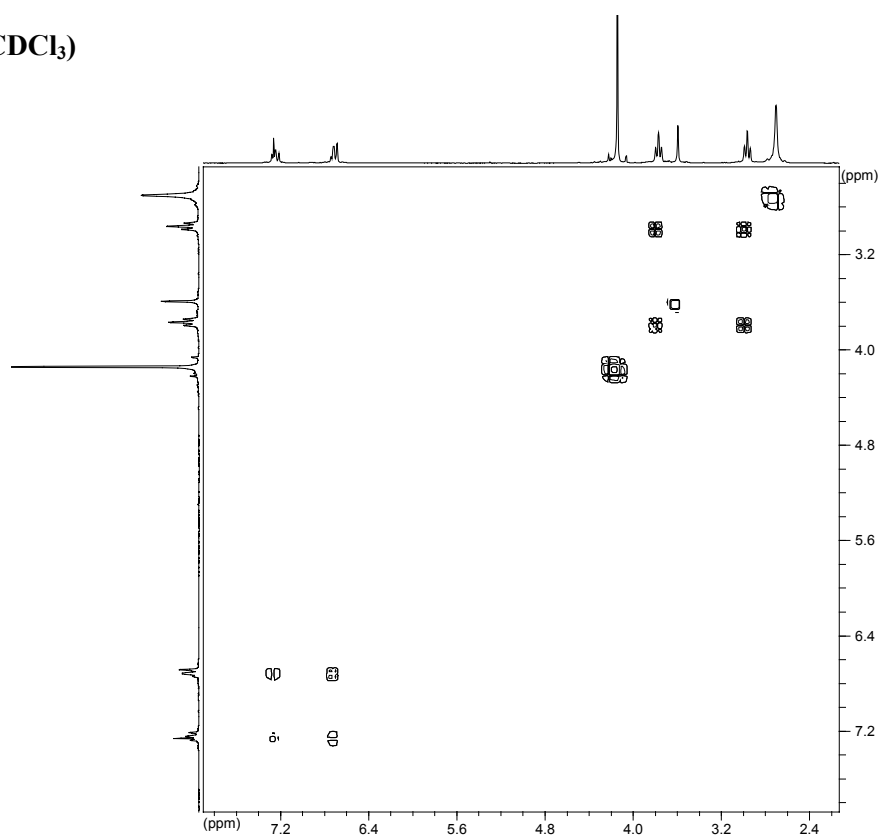
¹H RMN (CDCl₃)



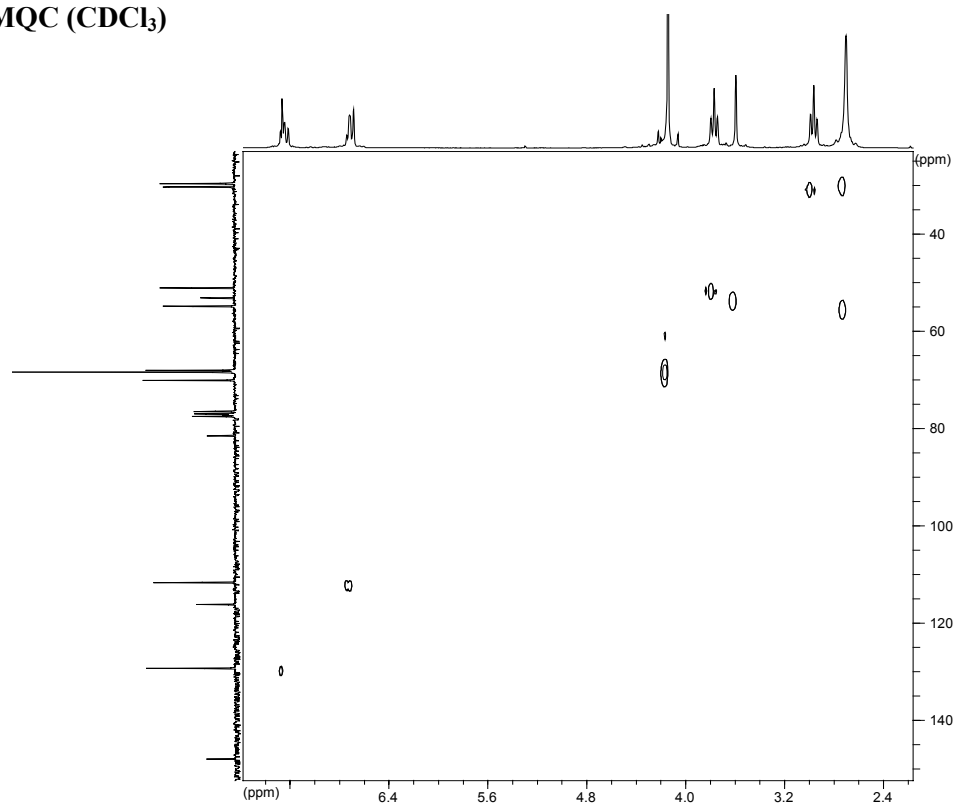
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



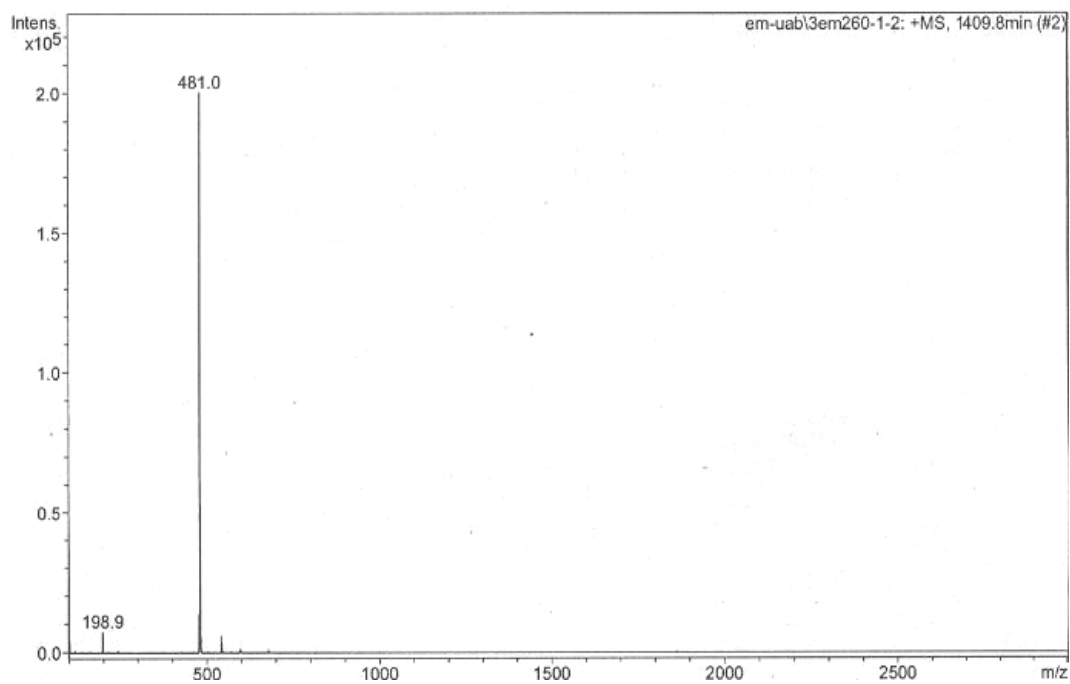
COSY (CDCl_3)



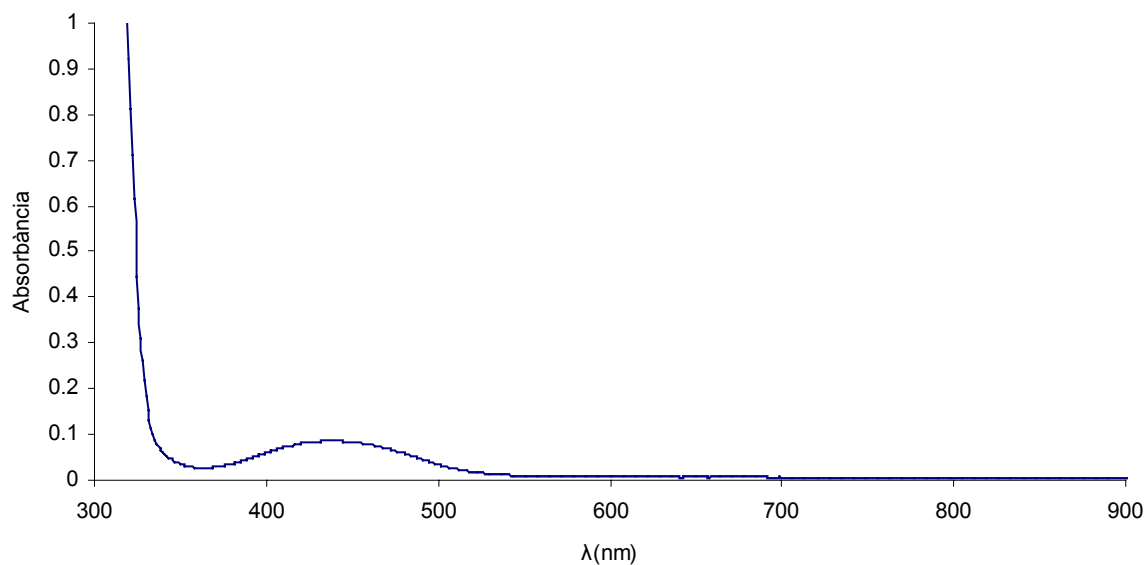
HMQC (CDCl₃)



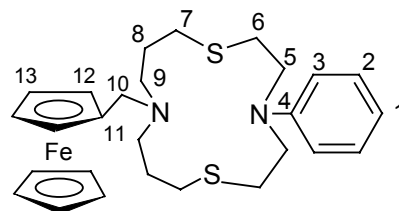
ESPECTROSCÒPIA DE MASSES (ESI(+)-IT) (10 ppm en CHCl₃-CH₃CN 1:2)



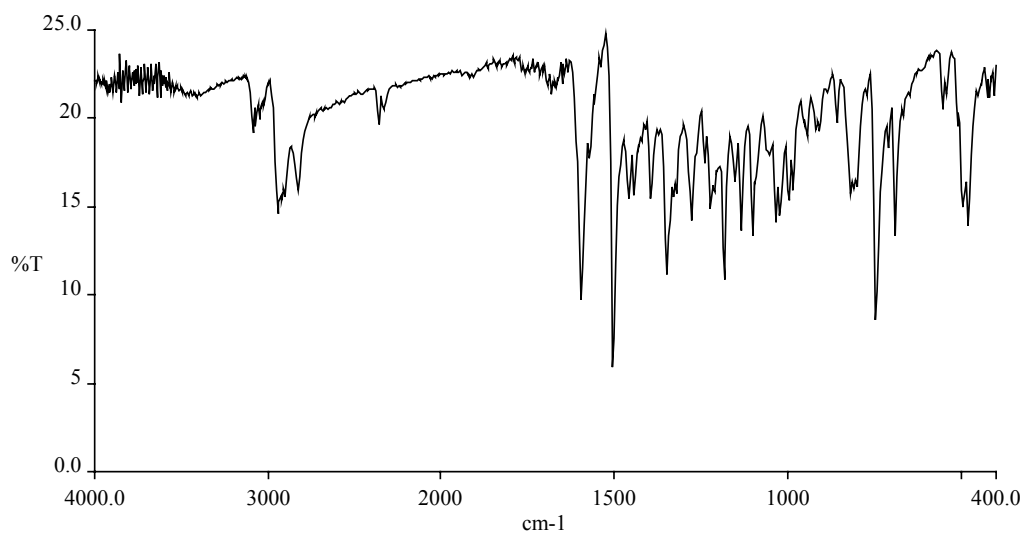
UV-VIS (CH₃CN, 1 mM)



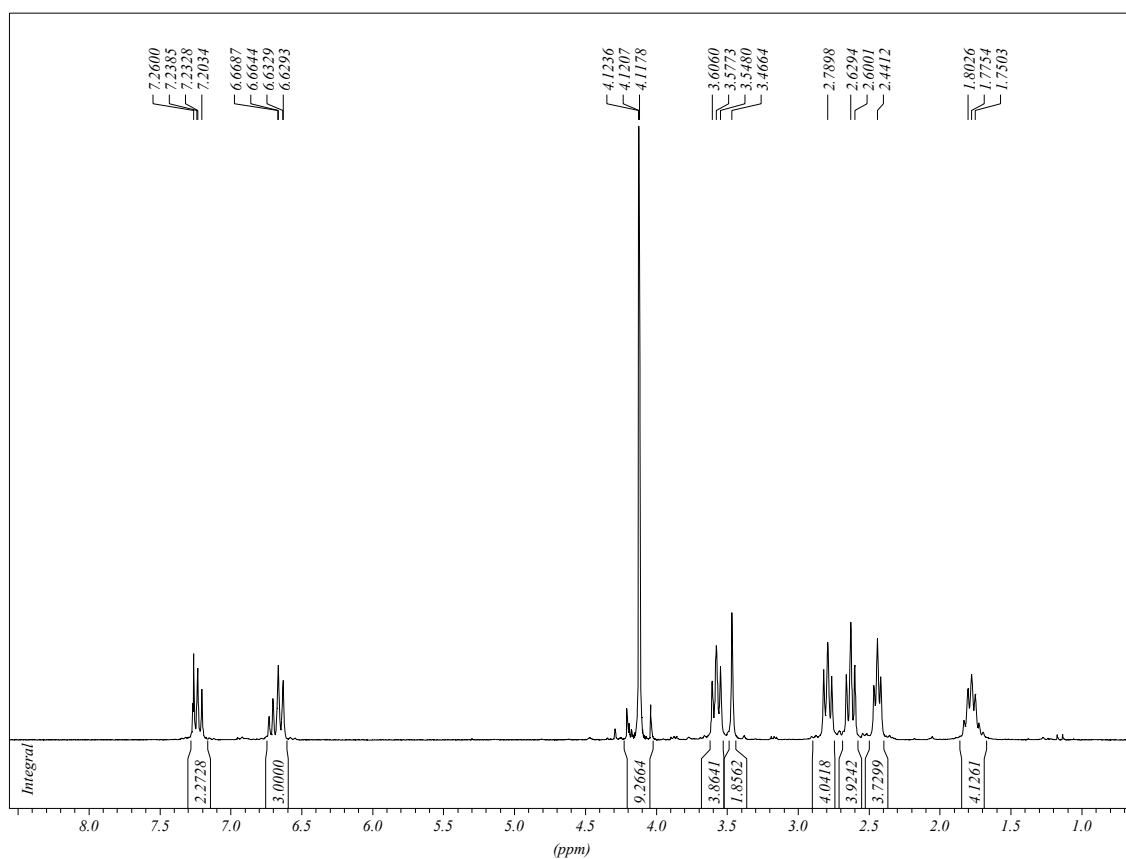
▪ (4-fenil-11-ilmetil-1,7-ditia-4,11-diazaciclottedradecà)ferrocè (L15)



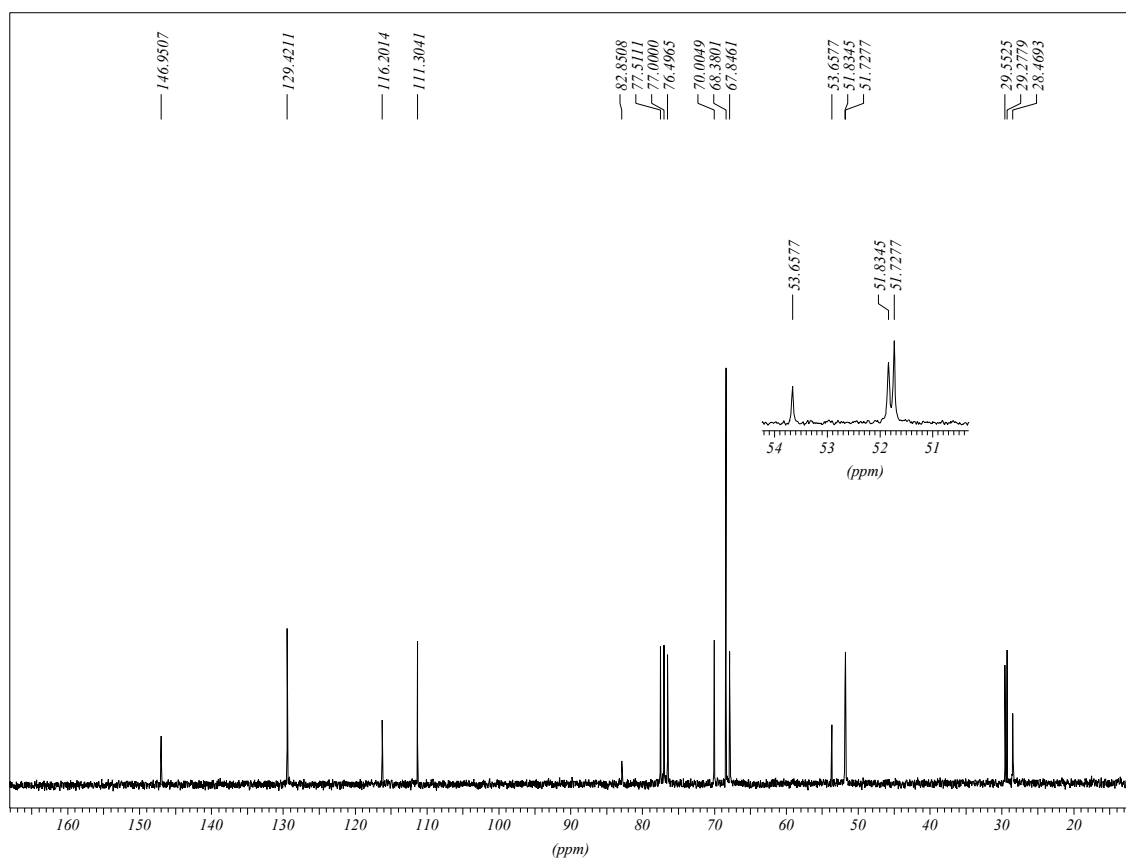
IR (KBr)



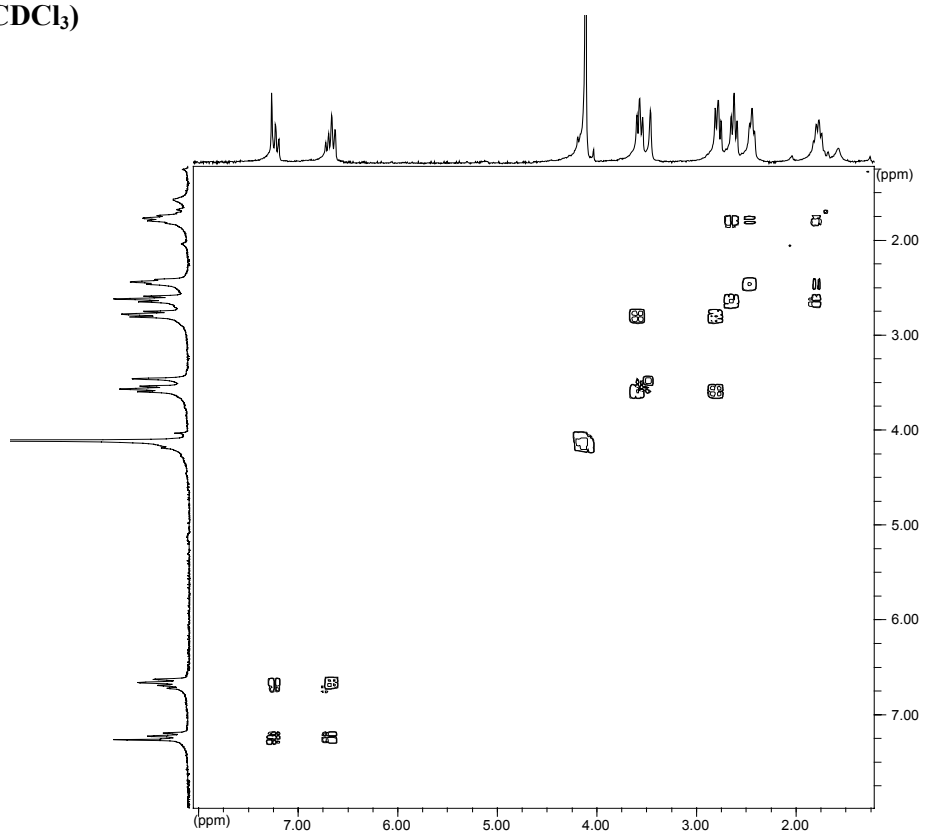
^1H RMN (CDCl_3)



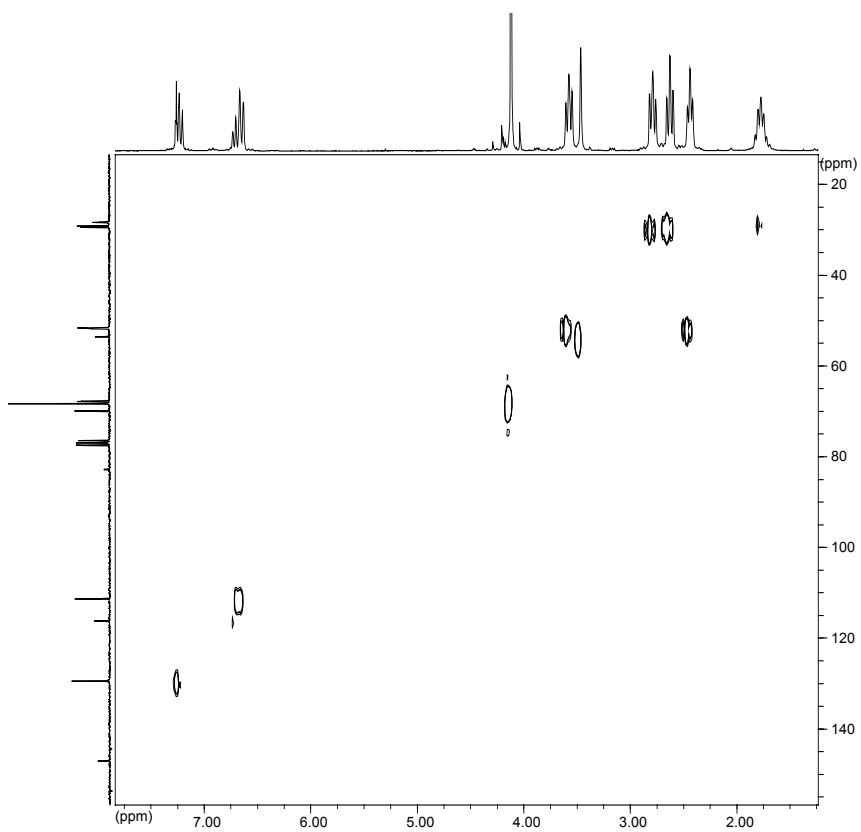
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



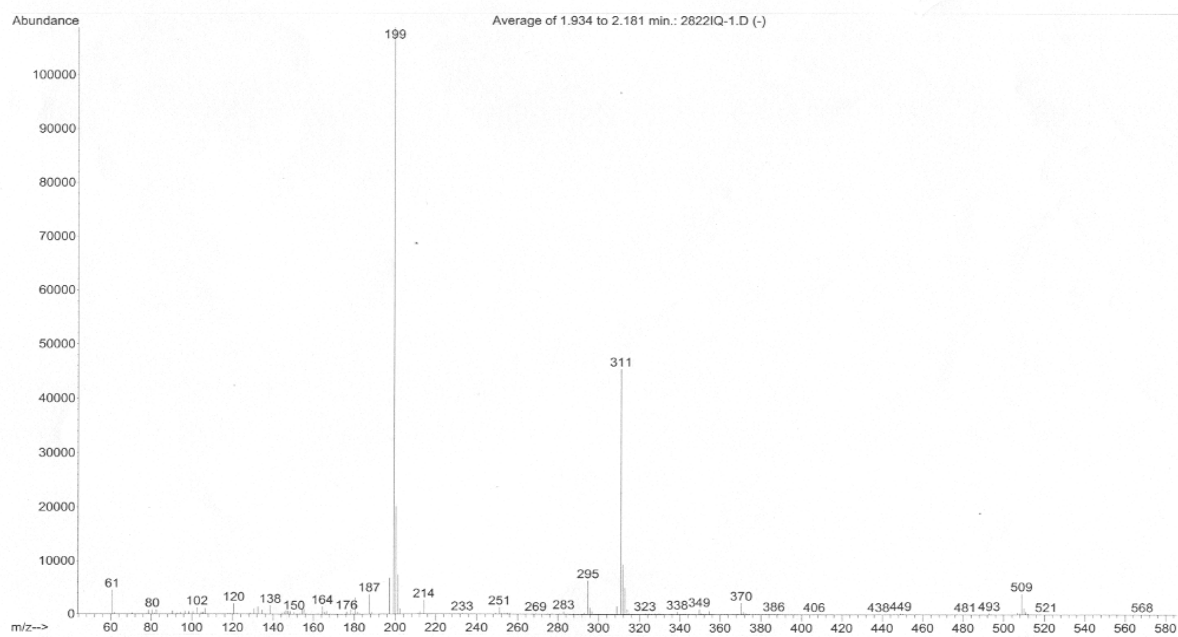
COSY (CDCl₃)



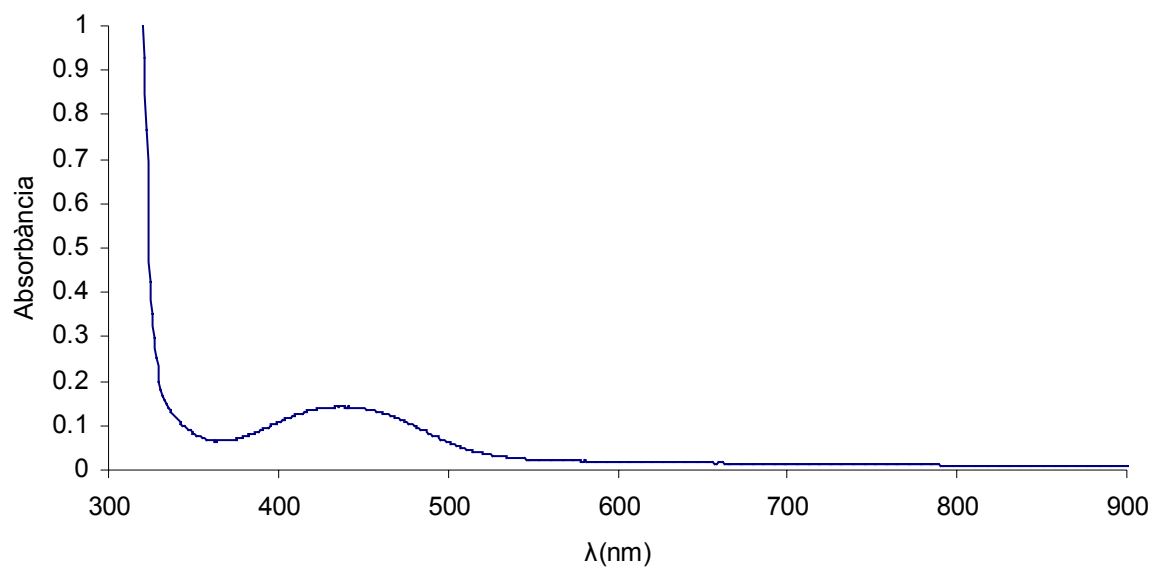
HMQC (CDCl₃)



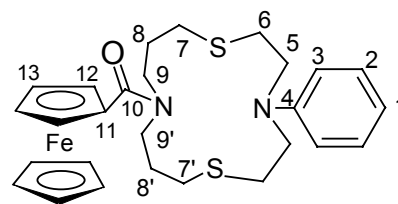
ESPECTROSCÒPIA DE MASSES (IQ)



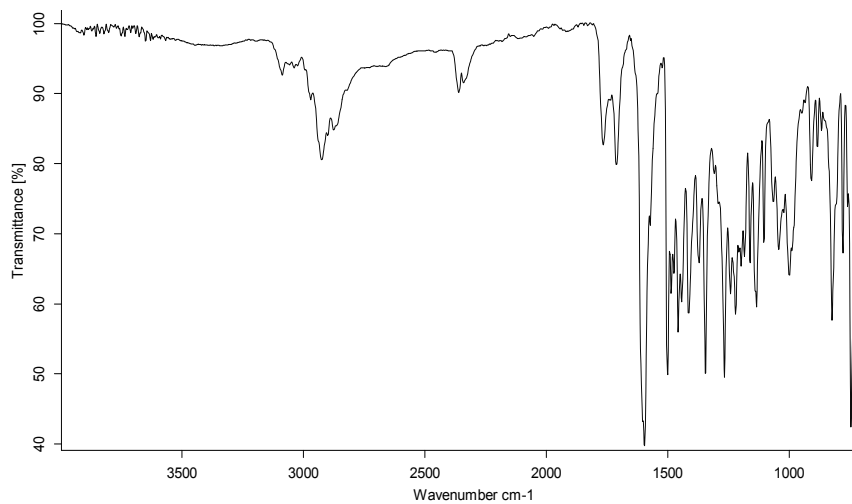
UV-VIS (CH₃CN, 1 mM)



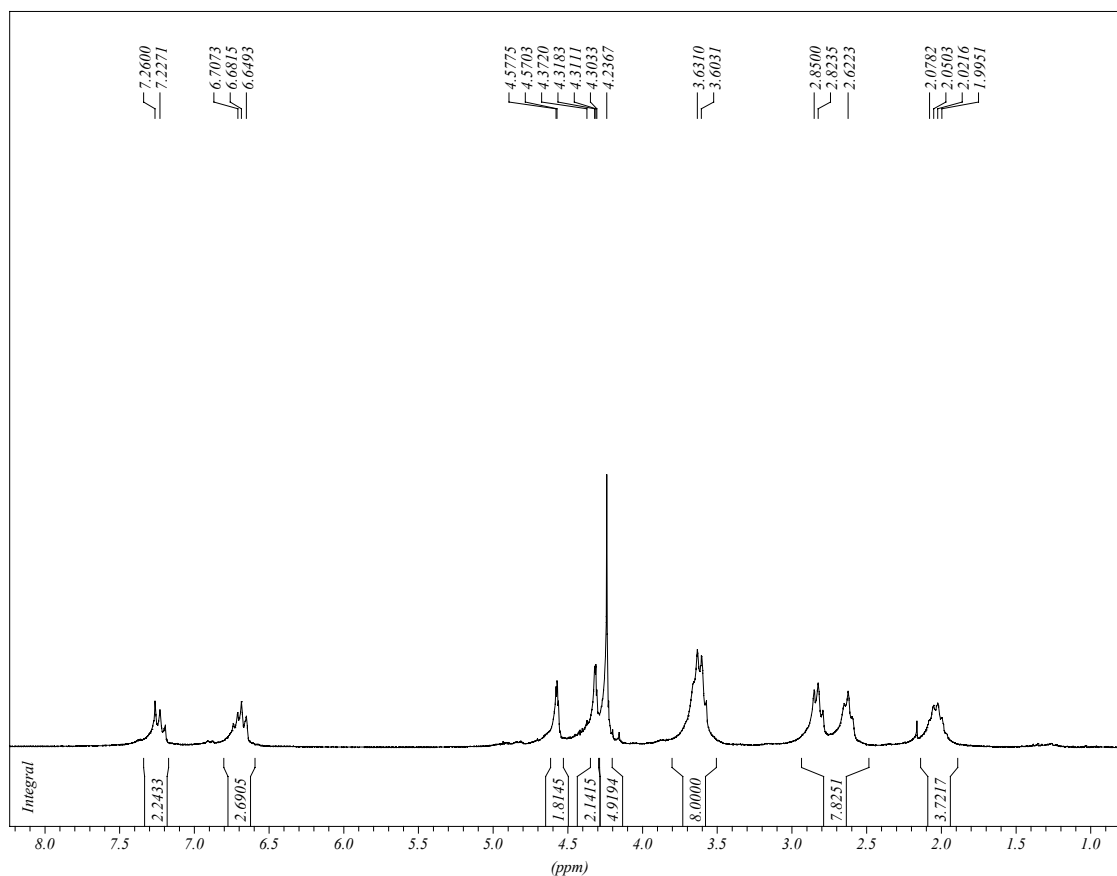
▪ (4-fenil-11-ilcarbonil-1,7-ditia-4,11-diazacictotradecà)ferrocè (L16)



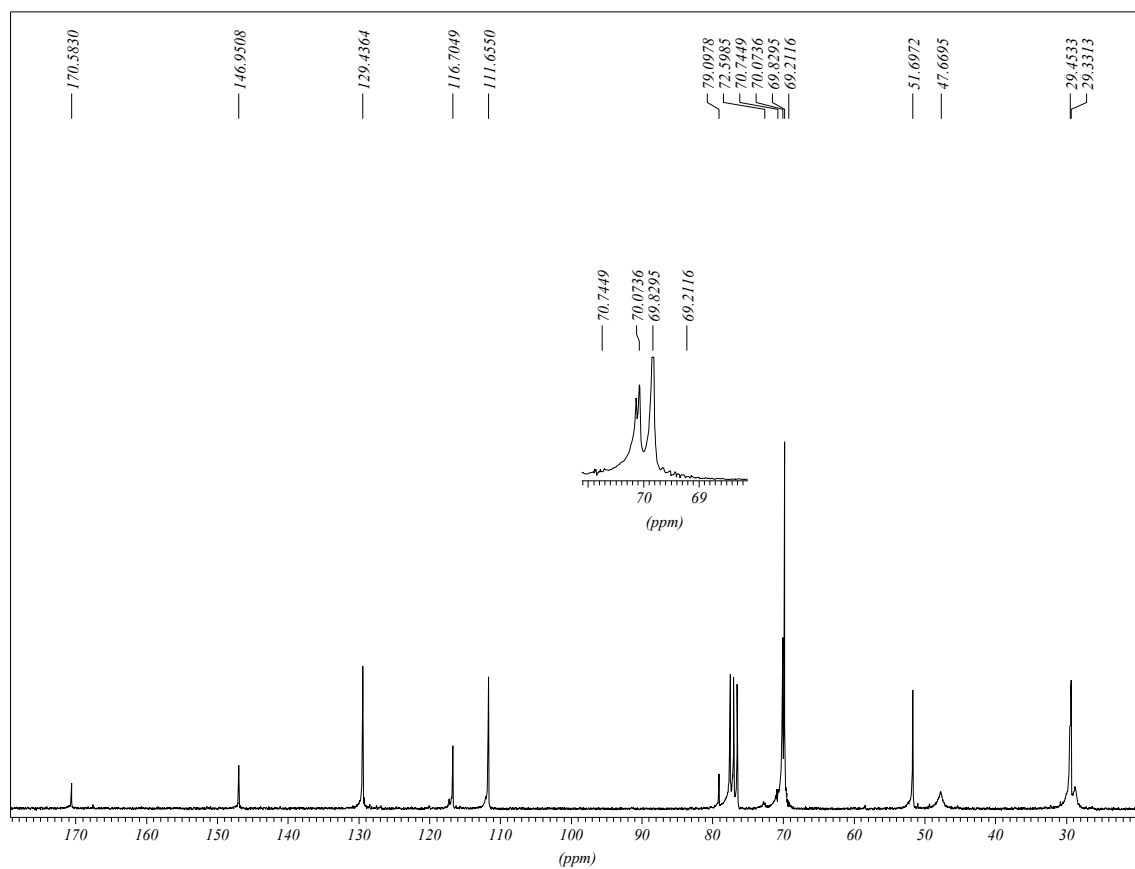
IR (ATR)



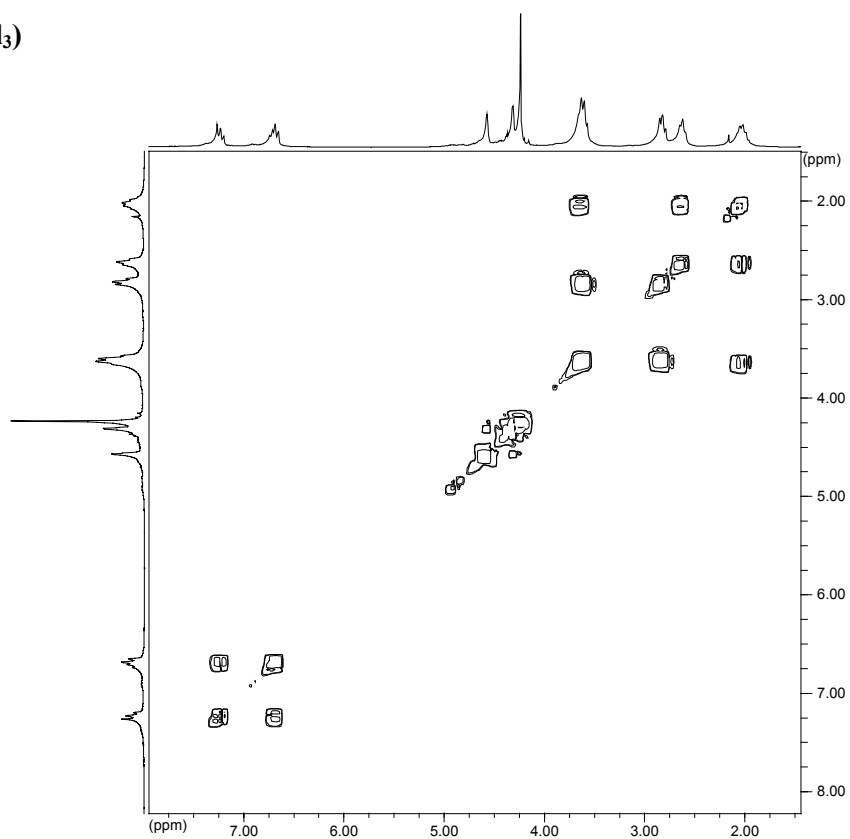
¹H RMN (CDCl₃)



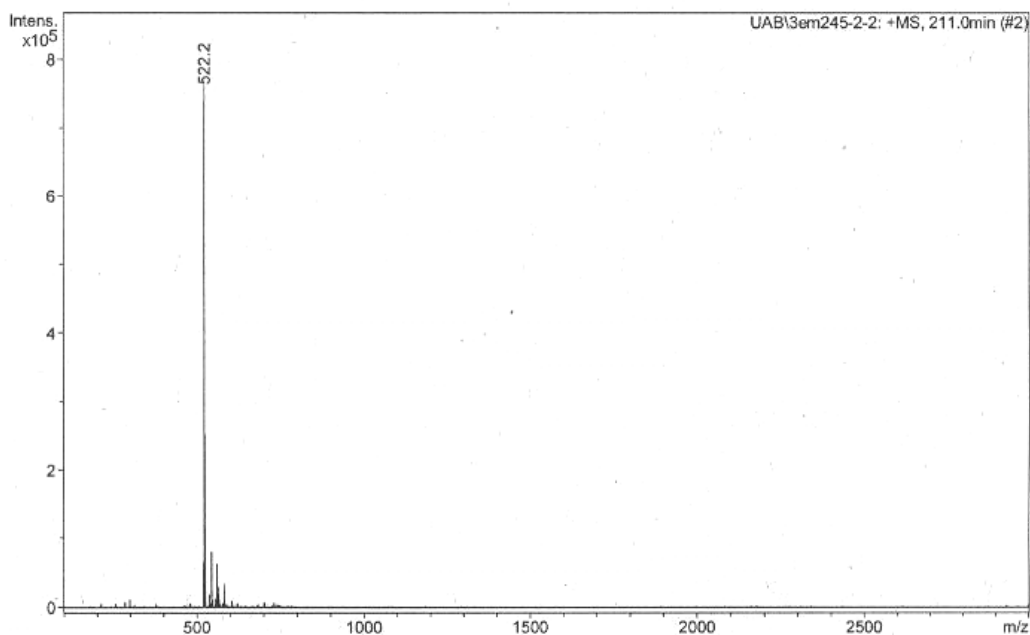
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



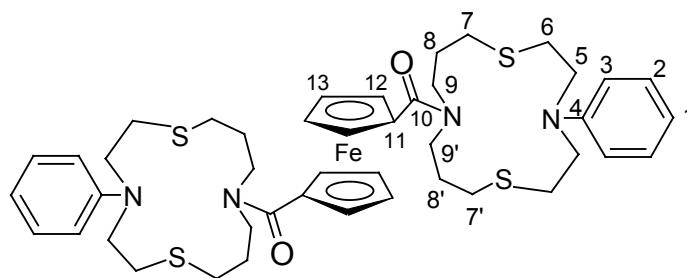
COSY (CDCl_3)



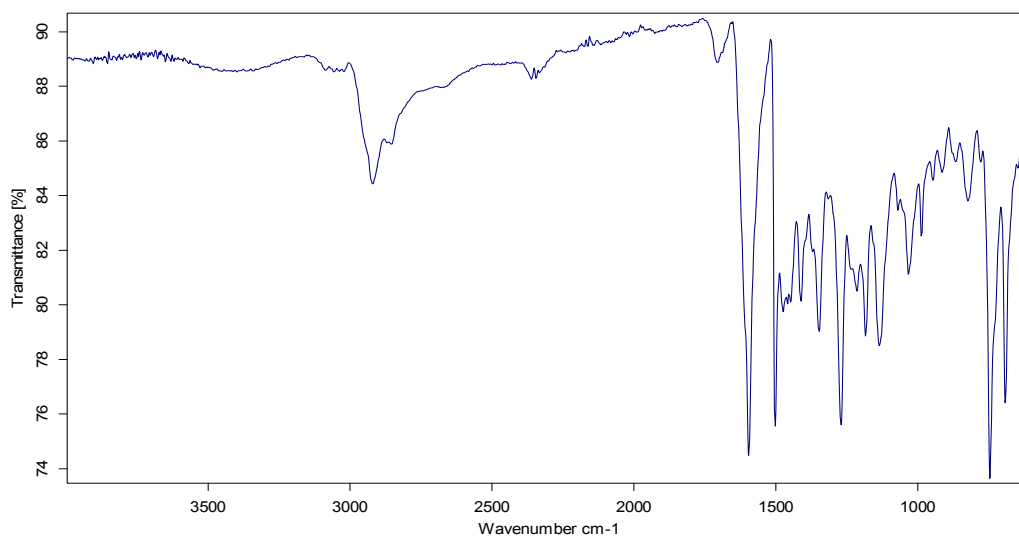
ESPECTROSCÒPIA DE MASSES (ESI(+)-IT) (10 ppm en CH₃CN)



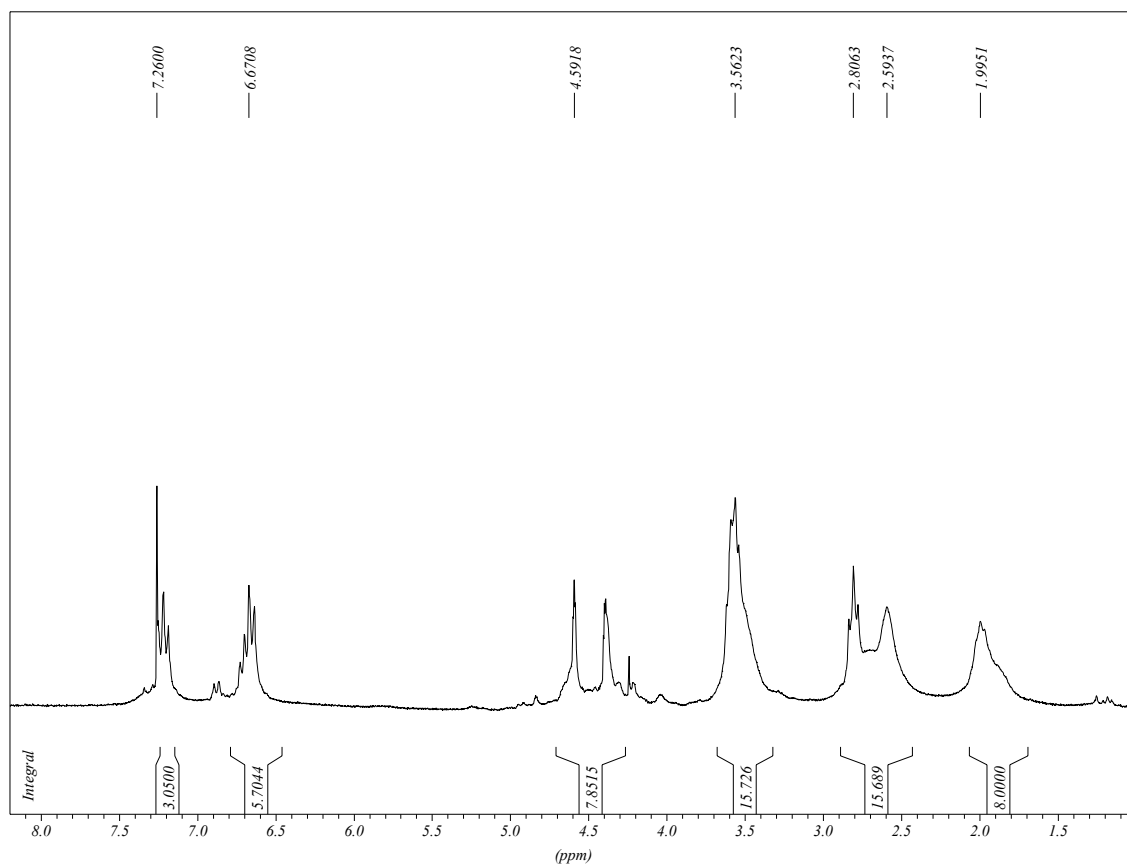
- 1,1'-bis(4-fenil-11-ilcarbonil-7-ditia-4,11-diazacicotetradecà)ferrocè (Bi-L16)



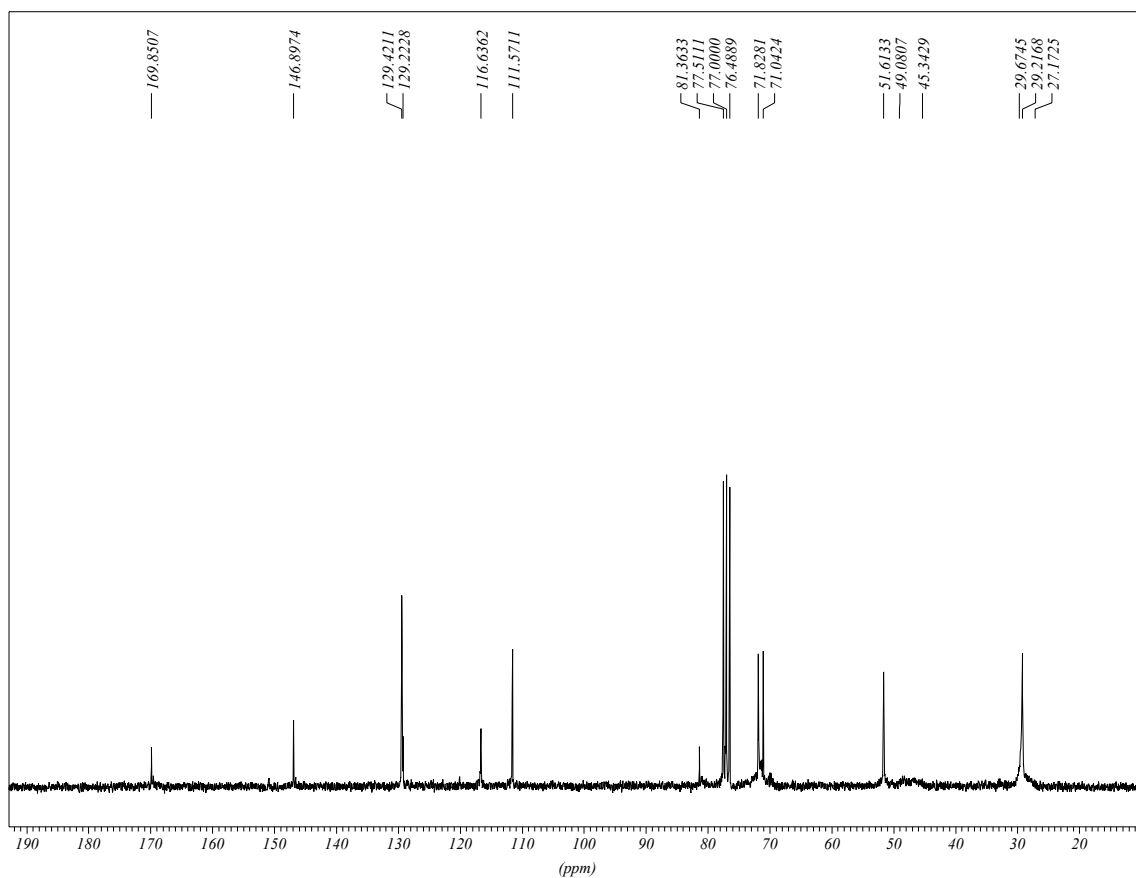
IR (ATR)



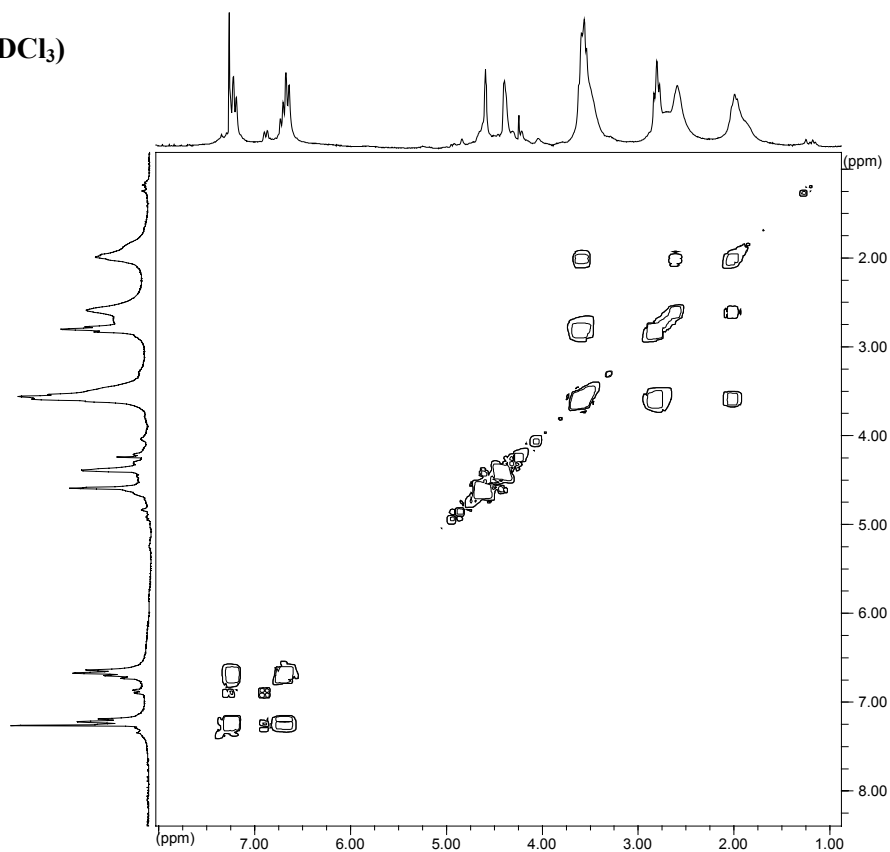
^1H RMN (CDCl_3)



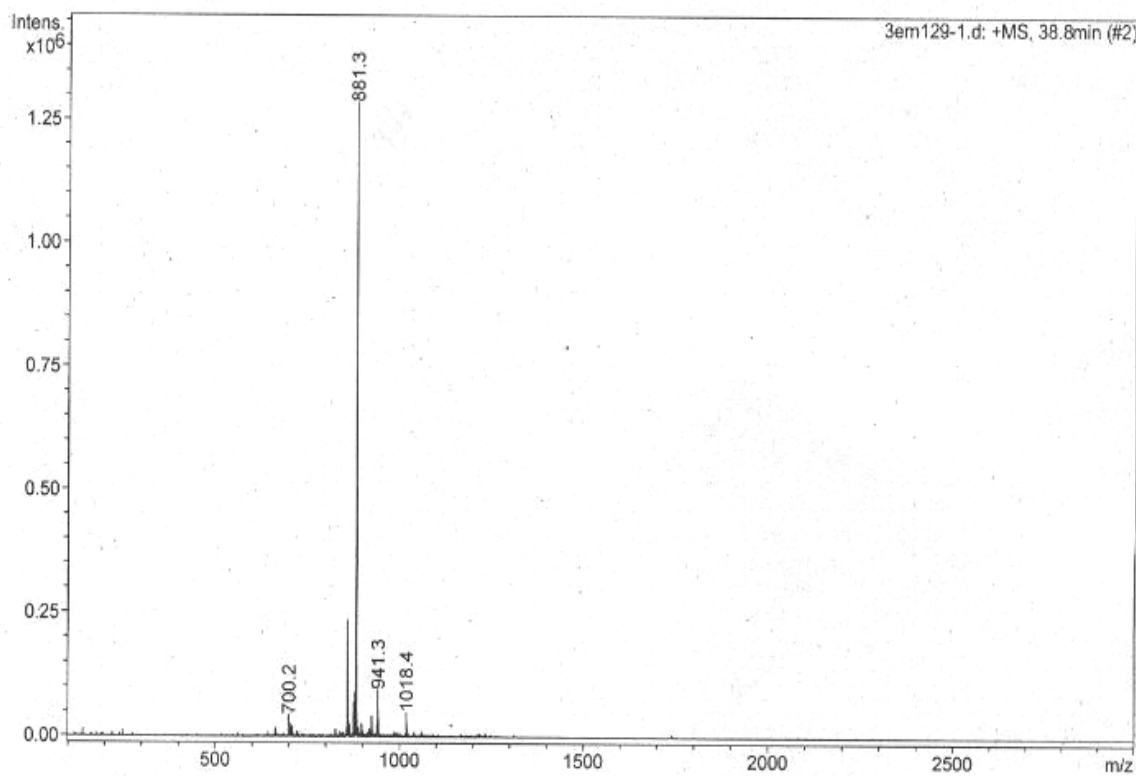
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



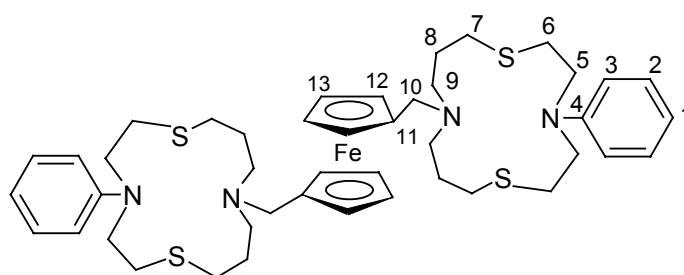
COSY (CDCl₃)



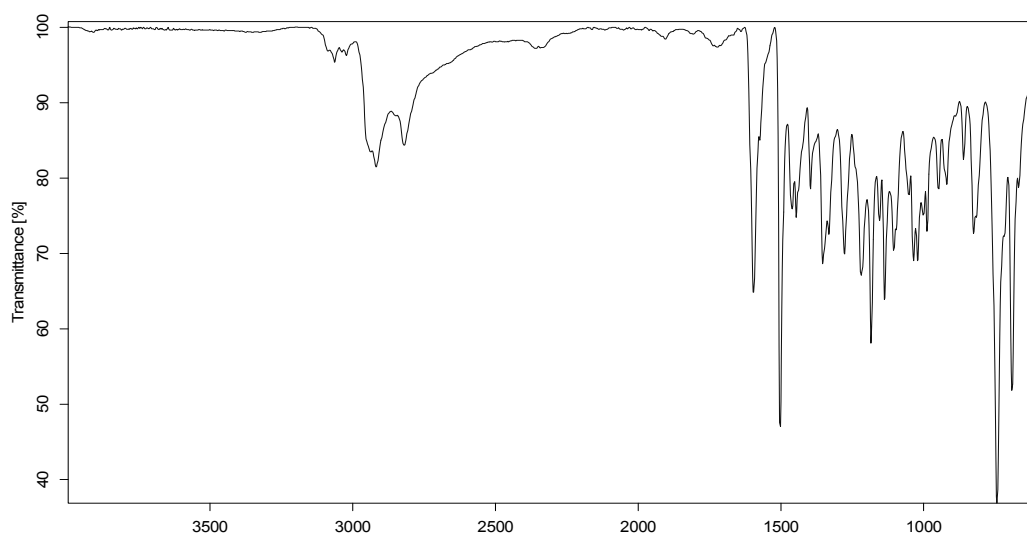
ESPECTROSCÒPIA DE MASSES (ESI(+)-IT) (10 ppm en CH₃CN)



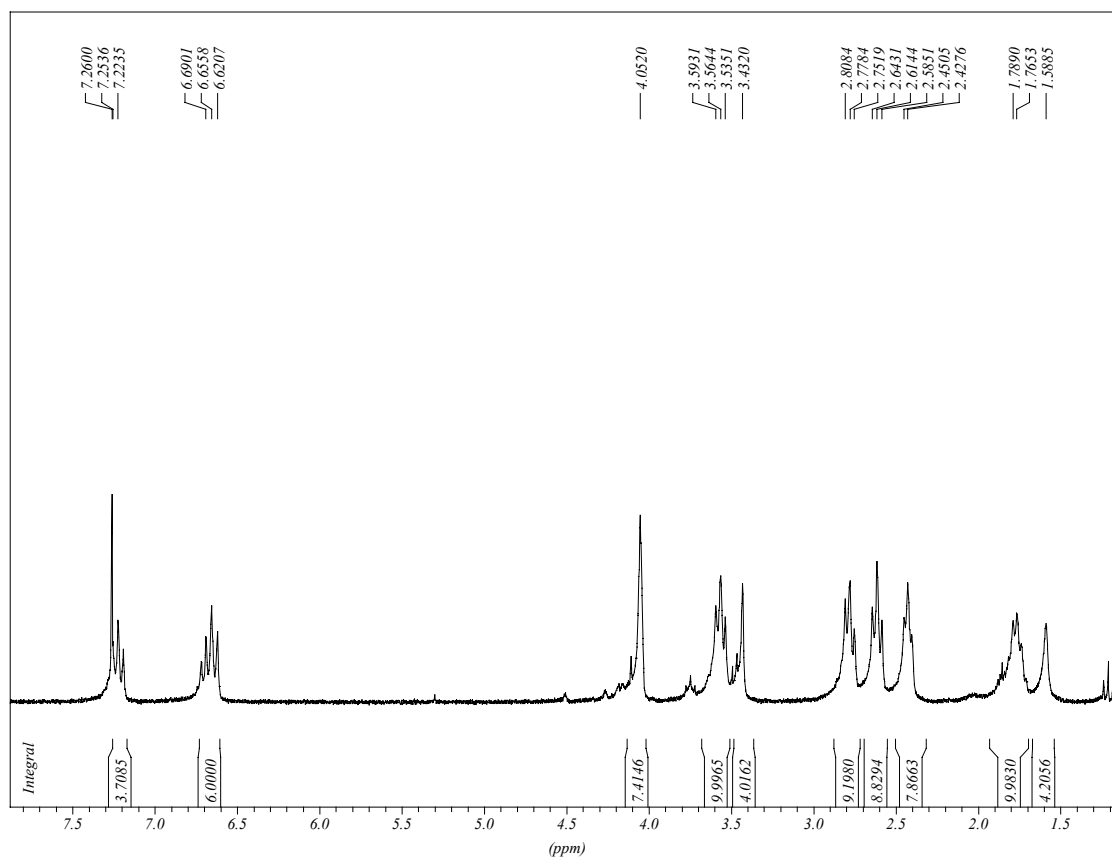
▪ 1,1'-bis(4-fenil-11-ilmetil-1,7-ditia-4,11-diazacictotradecà)ferrocè (Bi-L15)



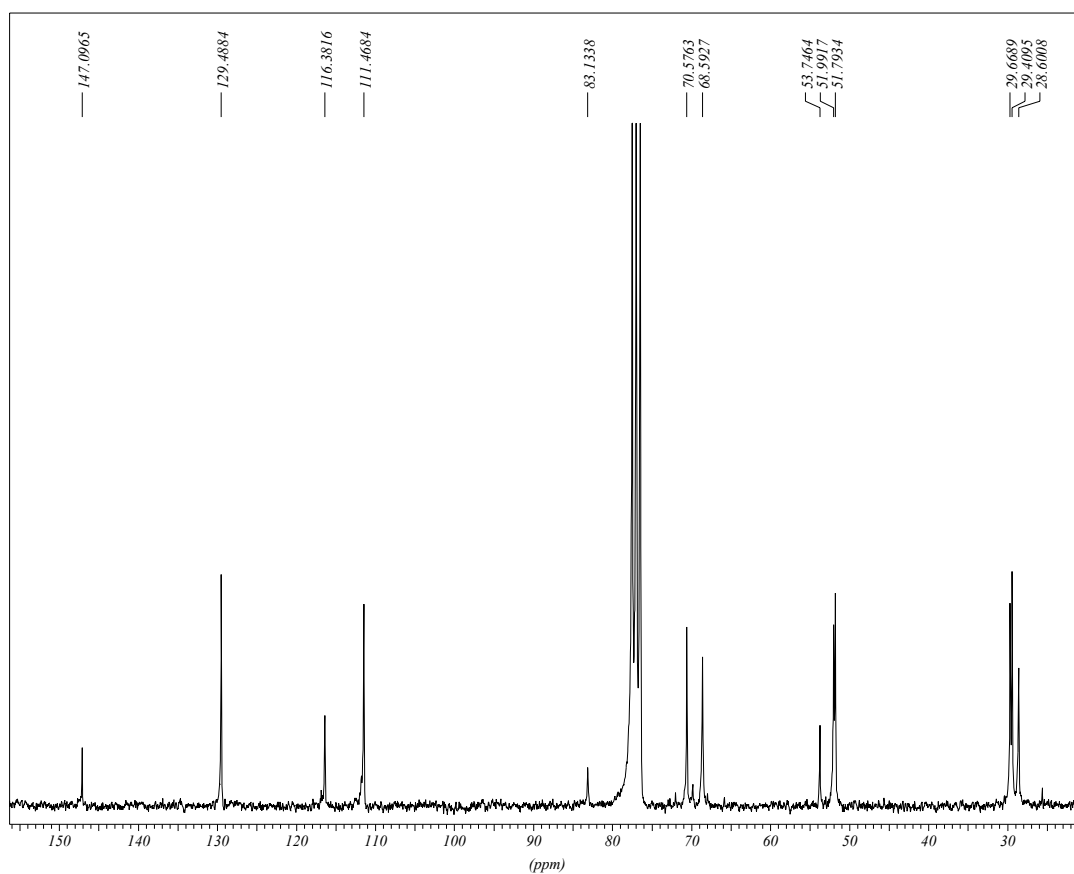
IR (ATR)



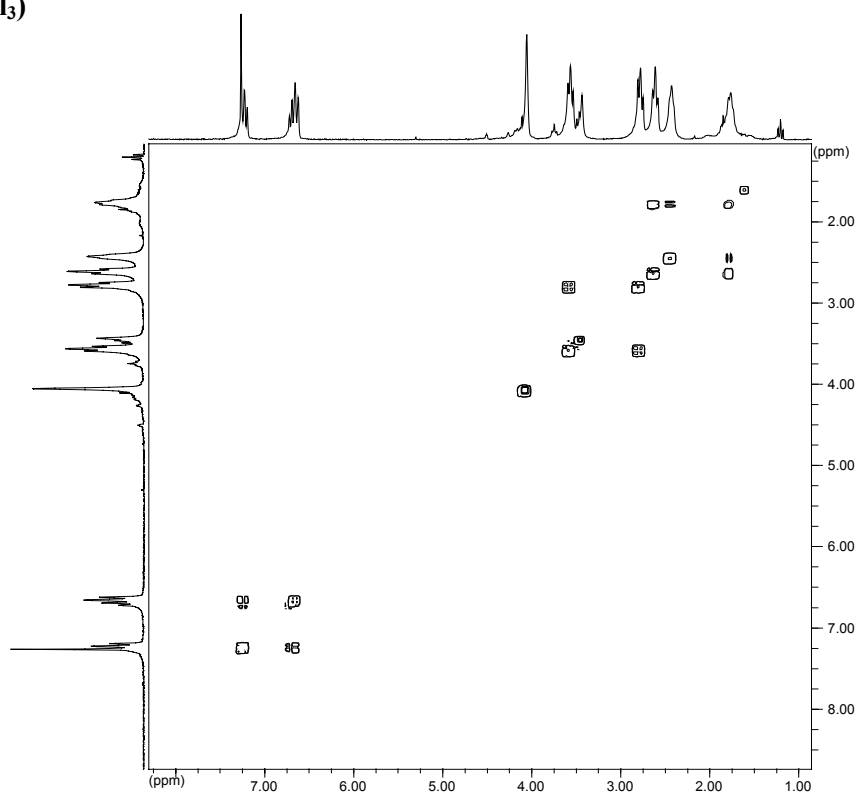
¹H RMN (CDCl₃)



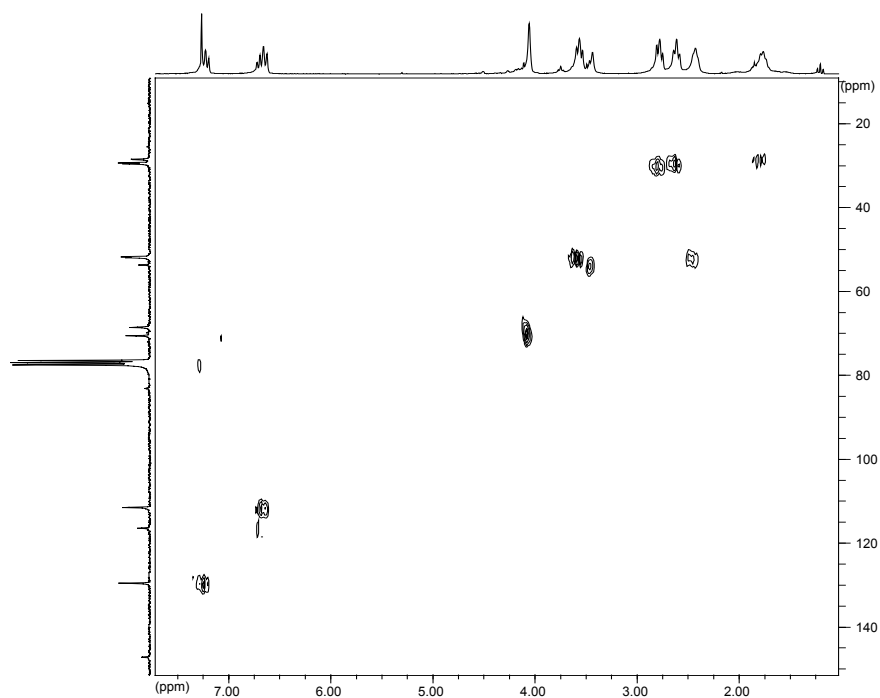
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



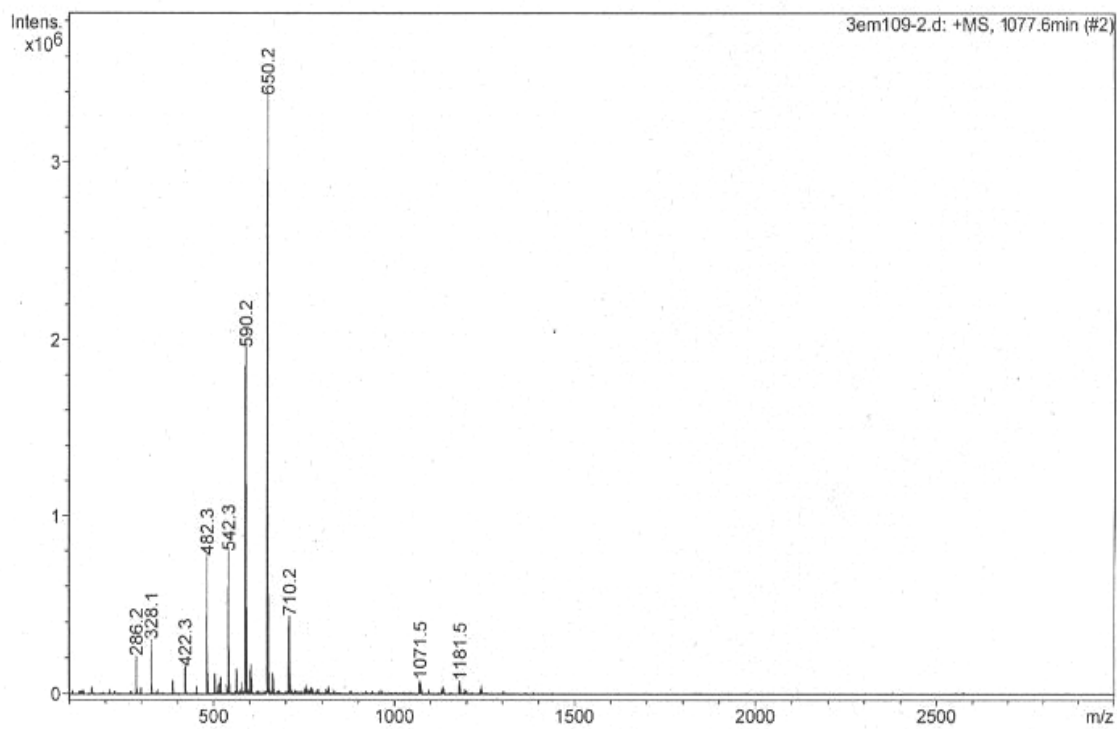
COSY (CDCl_3)



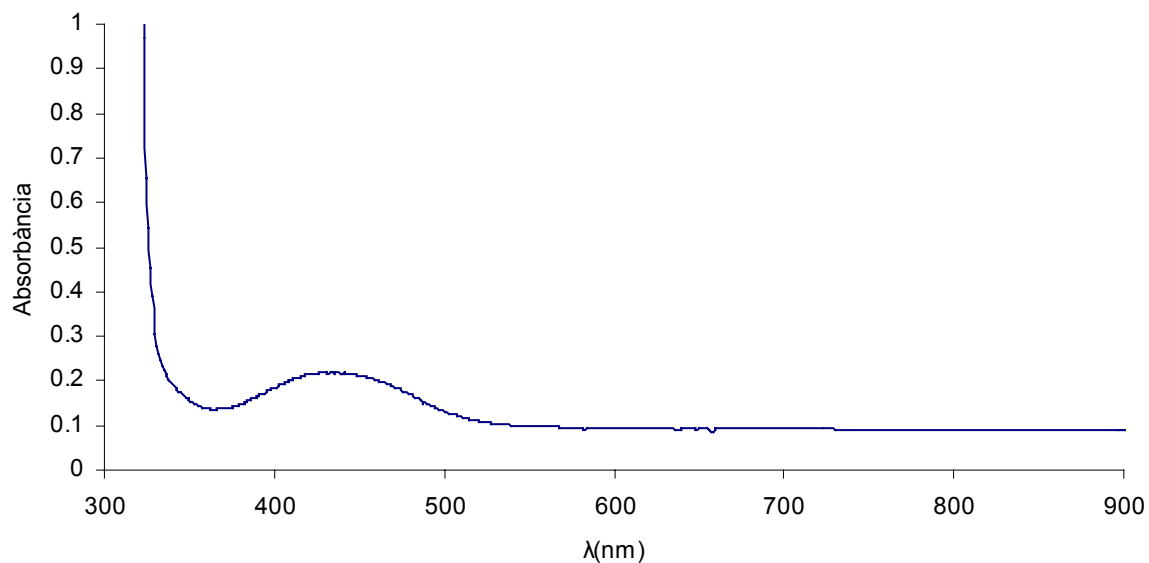
HMQC (CDCl₃)



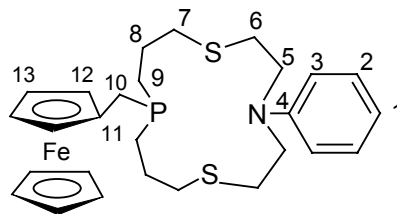
ESPECTROSCÒPIA DE MASSES (ESI(+)-IT) (10 ppm en MeOH-CHCl₃ 1:1, 0.1% HCOOH)



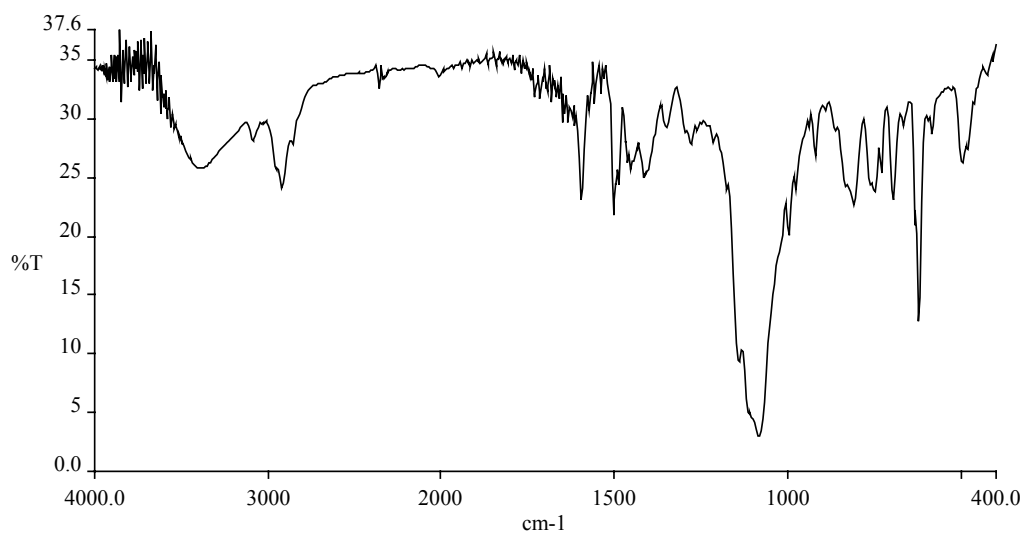
UV-VIS (CH₂Cl₂, 1 mM)



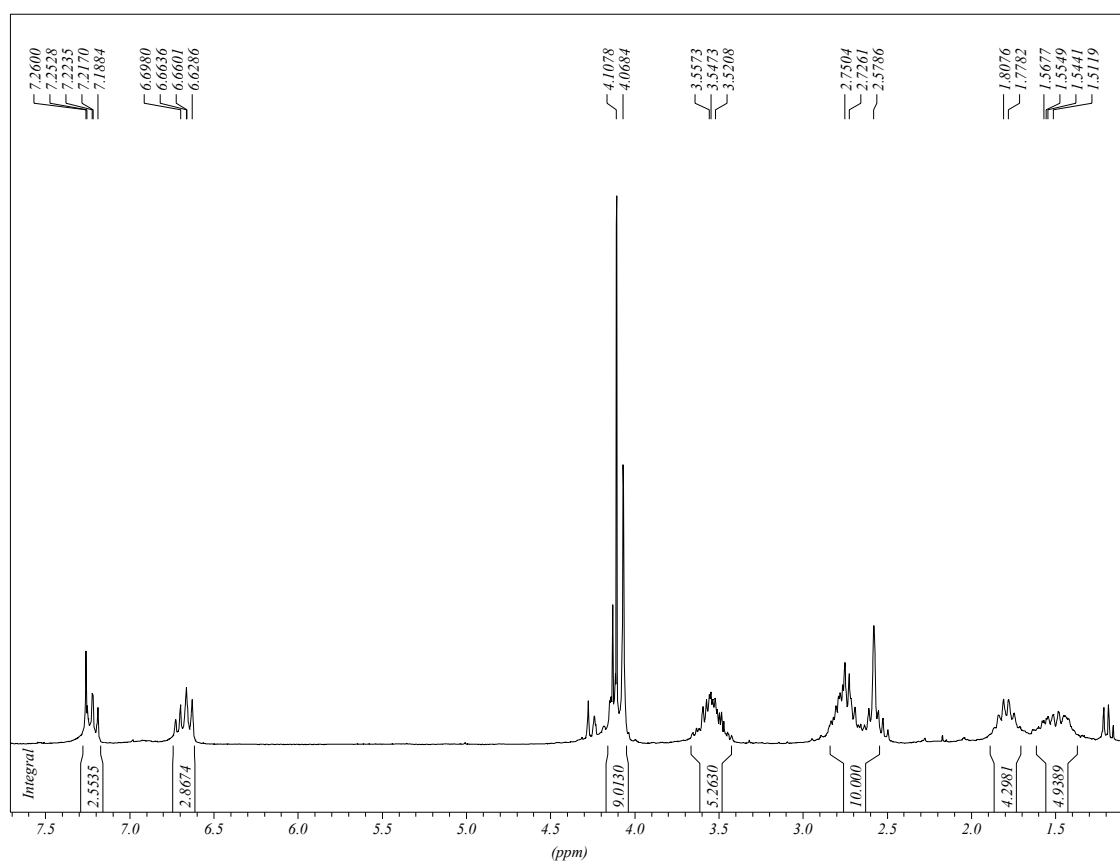
- 4-phenil-11-ferrocenilmetil-1,7-ditia-4-aza-11-fosfaciclotetradecà (L17)



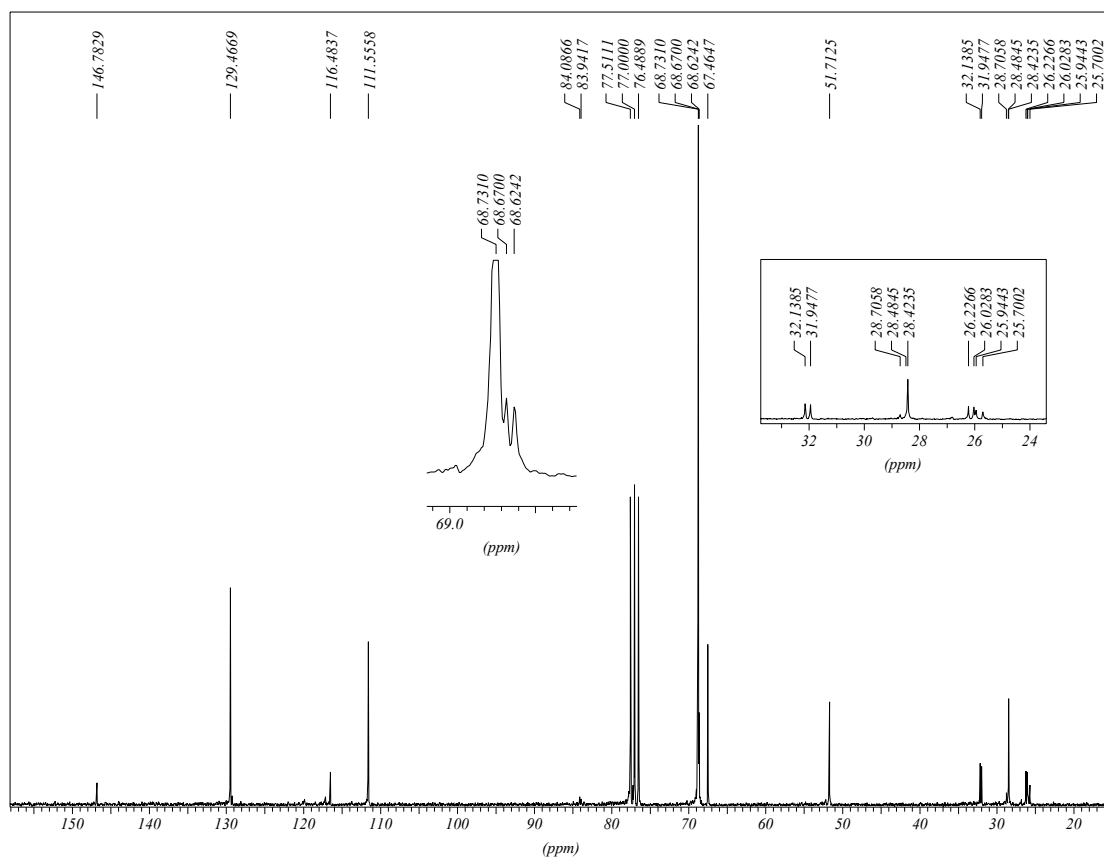
IR (KBr)



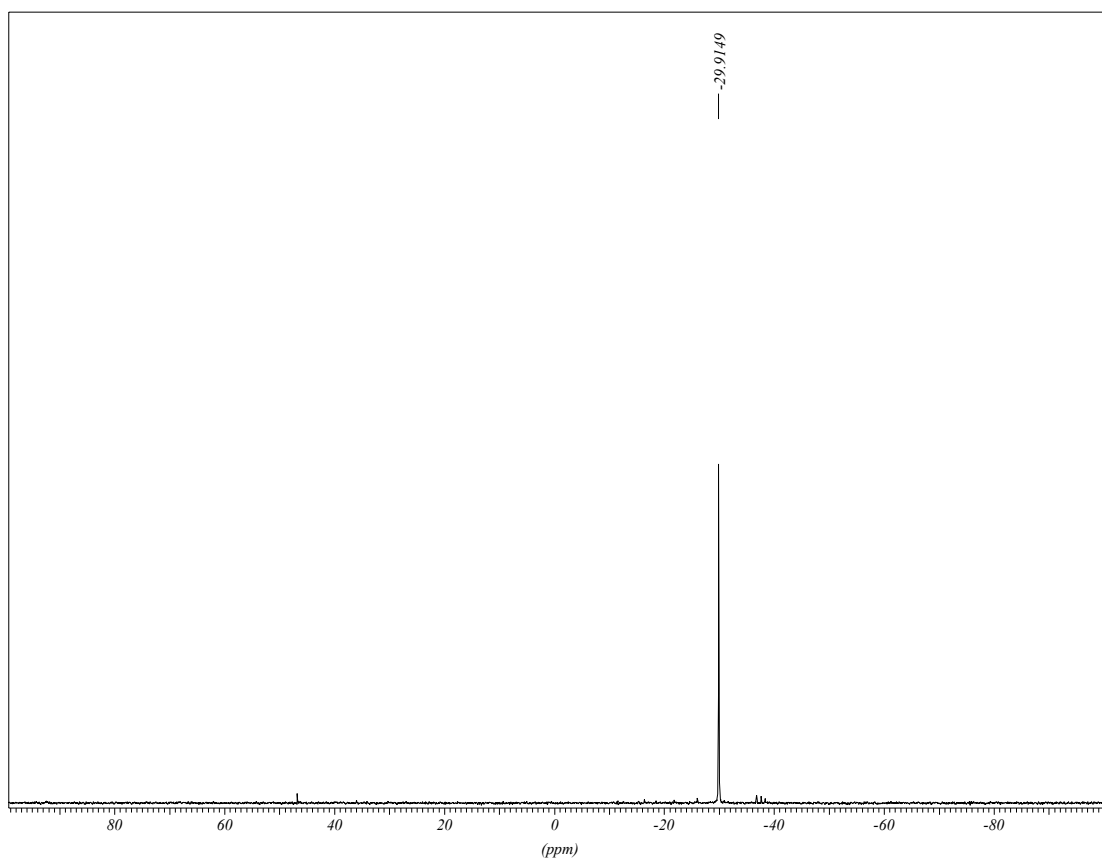
^1H RMN (CDCl_3)



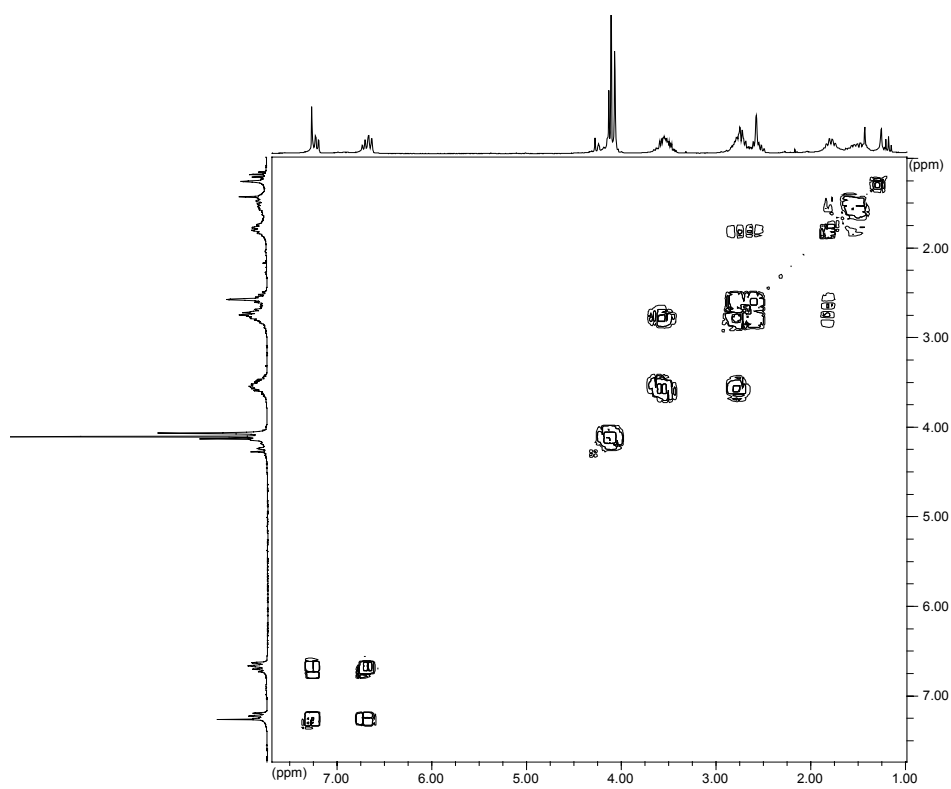
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



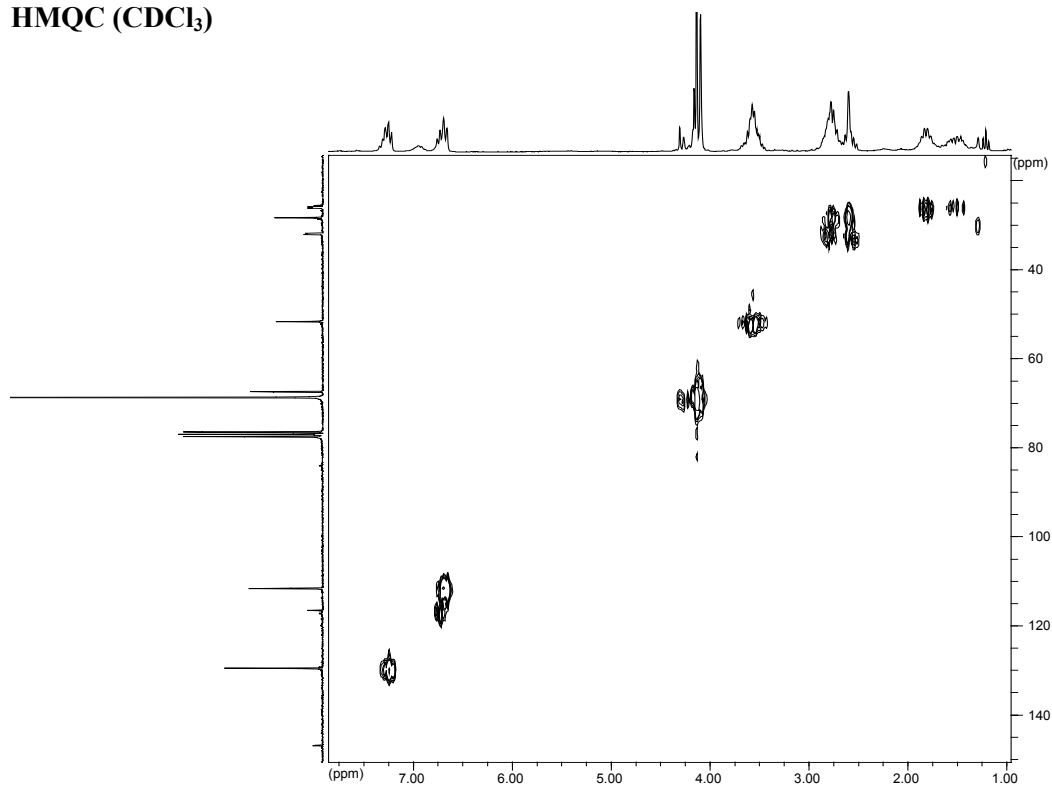
$^{31}\text{P}\{^1\text{H}\}$ RMN (CDCl_3)



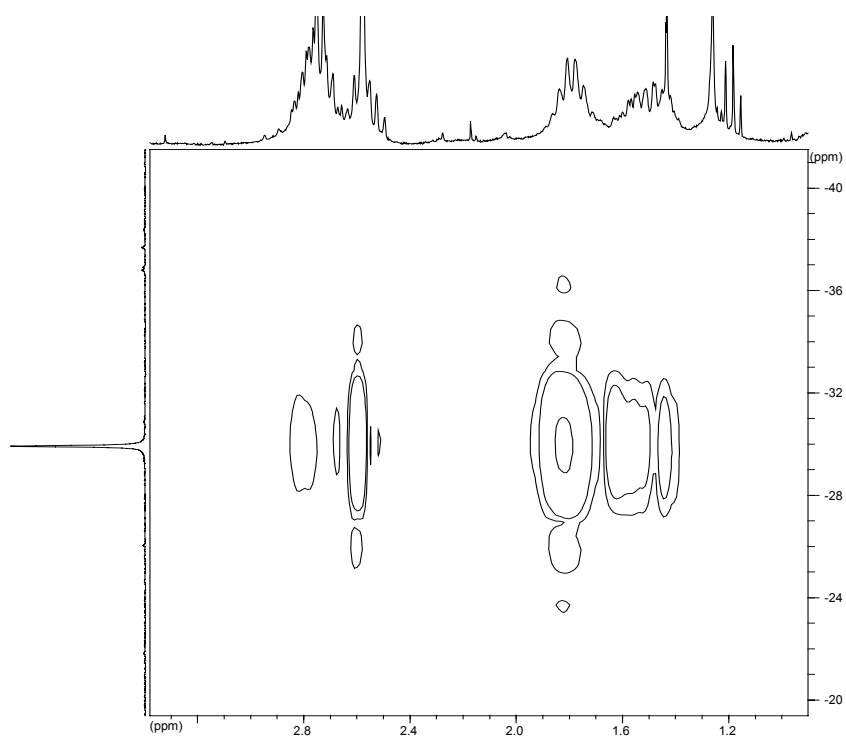
COSY (CDCl_3)



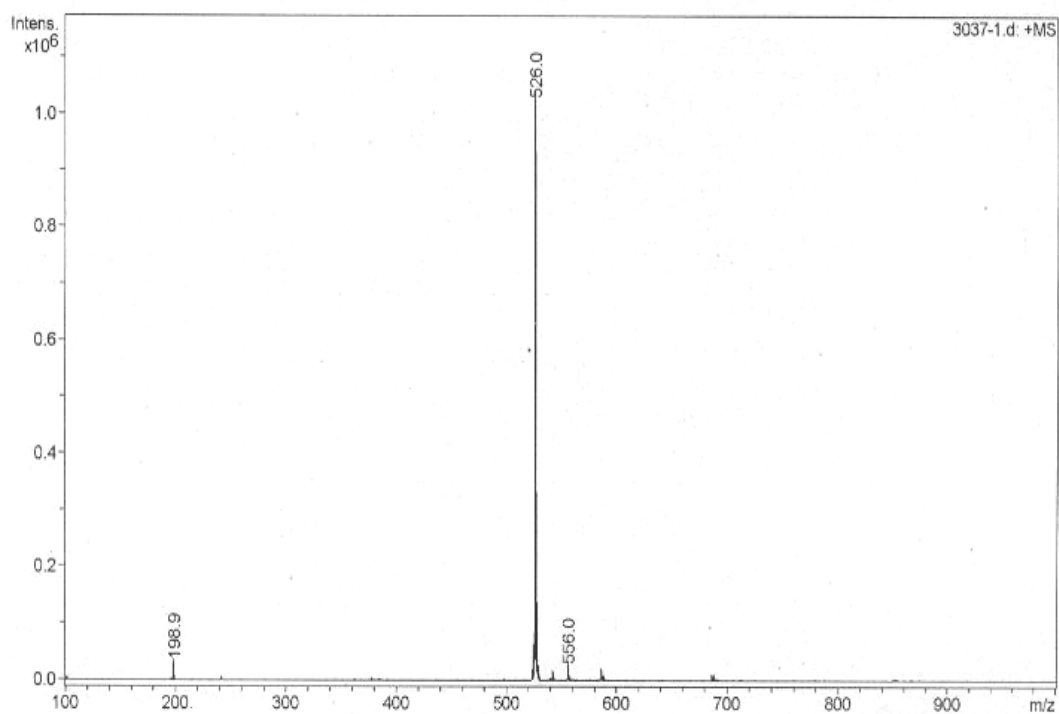
HMQC (CDCl₃)



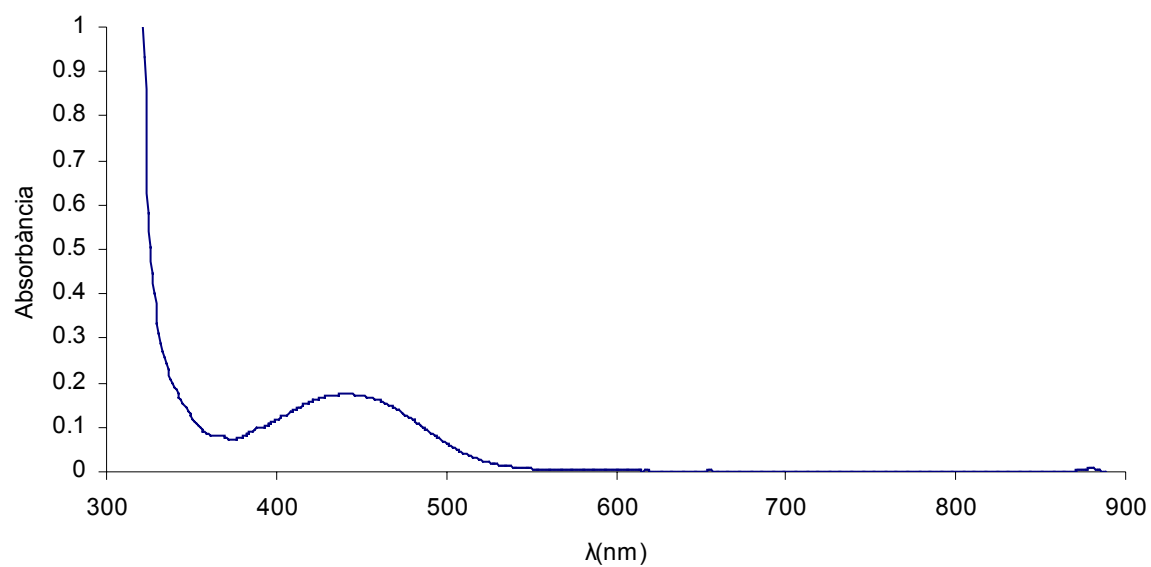
HMBC (CDCl₃)



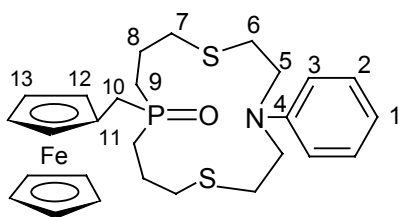
ESPECTROSCÒPIA DE MASSES (ESI(+)-IT) (10 ppm en CH₃CN)



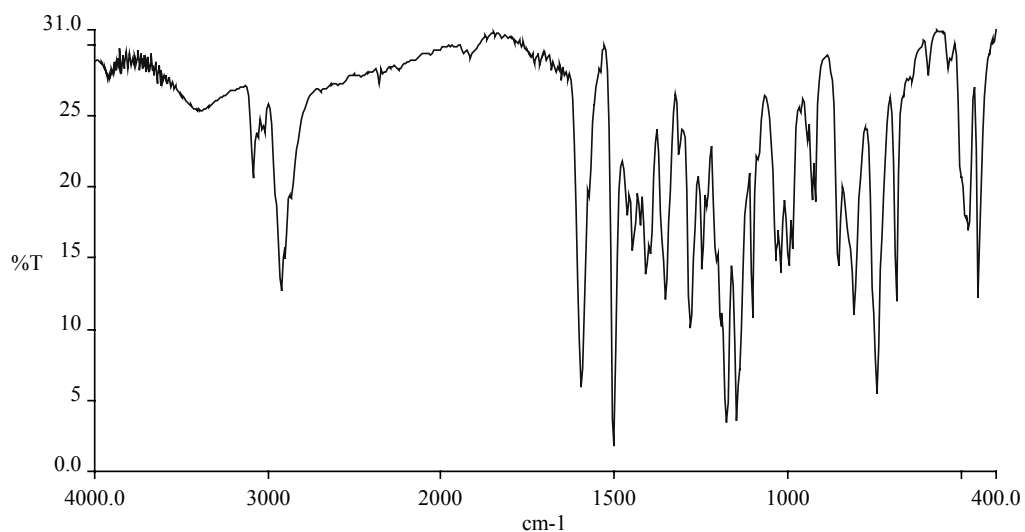
UV-VIS (CH₂Cl₂-CH₃CN, 1 mM)



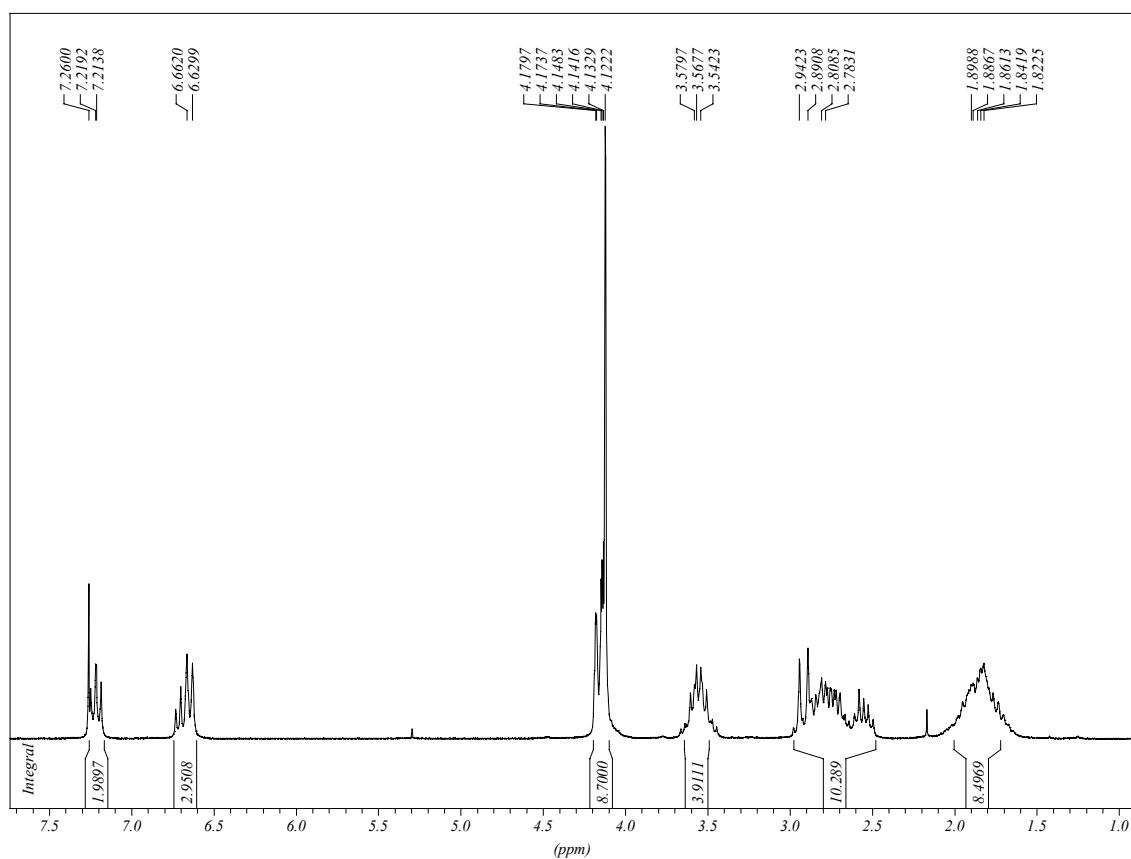
▪ 4-fenil-11-ferrocenilmetil-1,7-ditia-4-aza-11-fosfaciclotetradecà-11-òxid (L18)



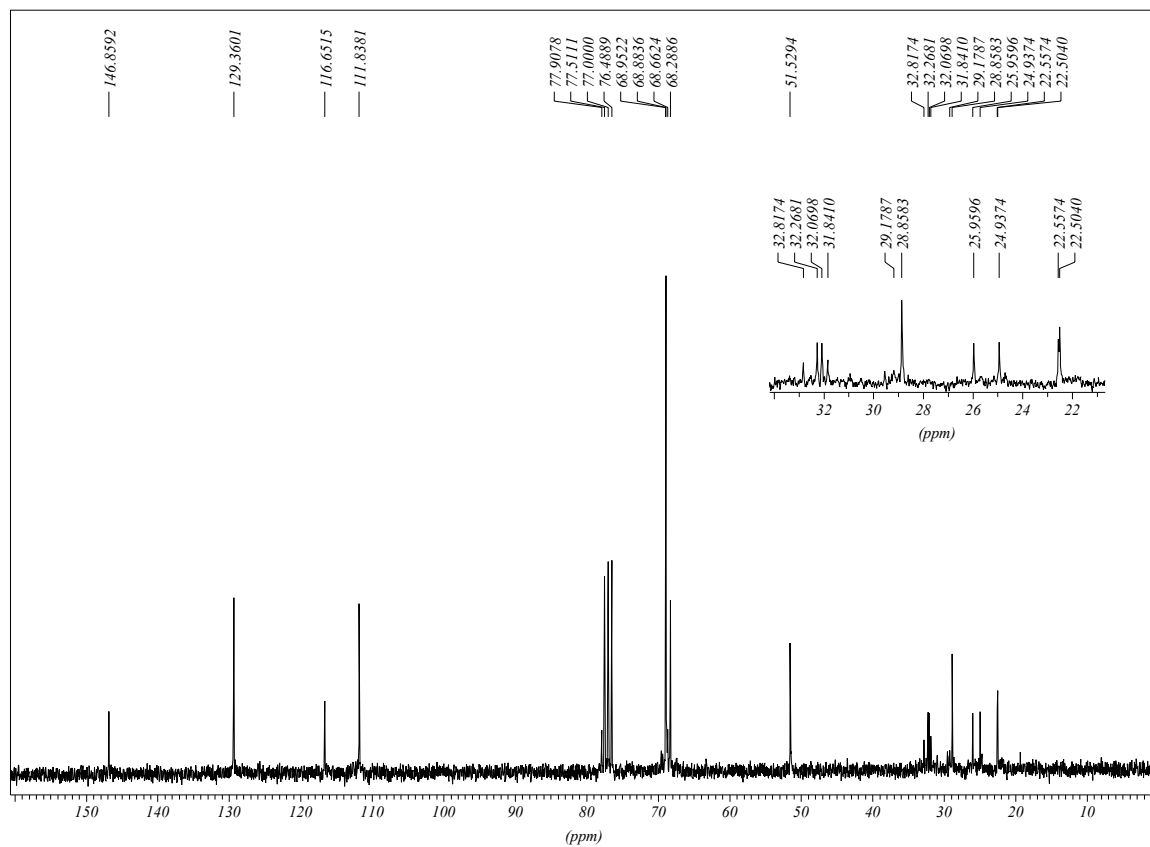
IR (KBr)



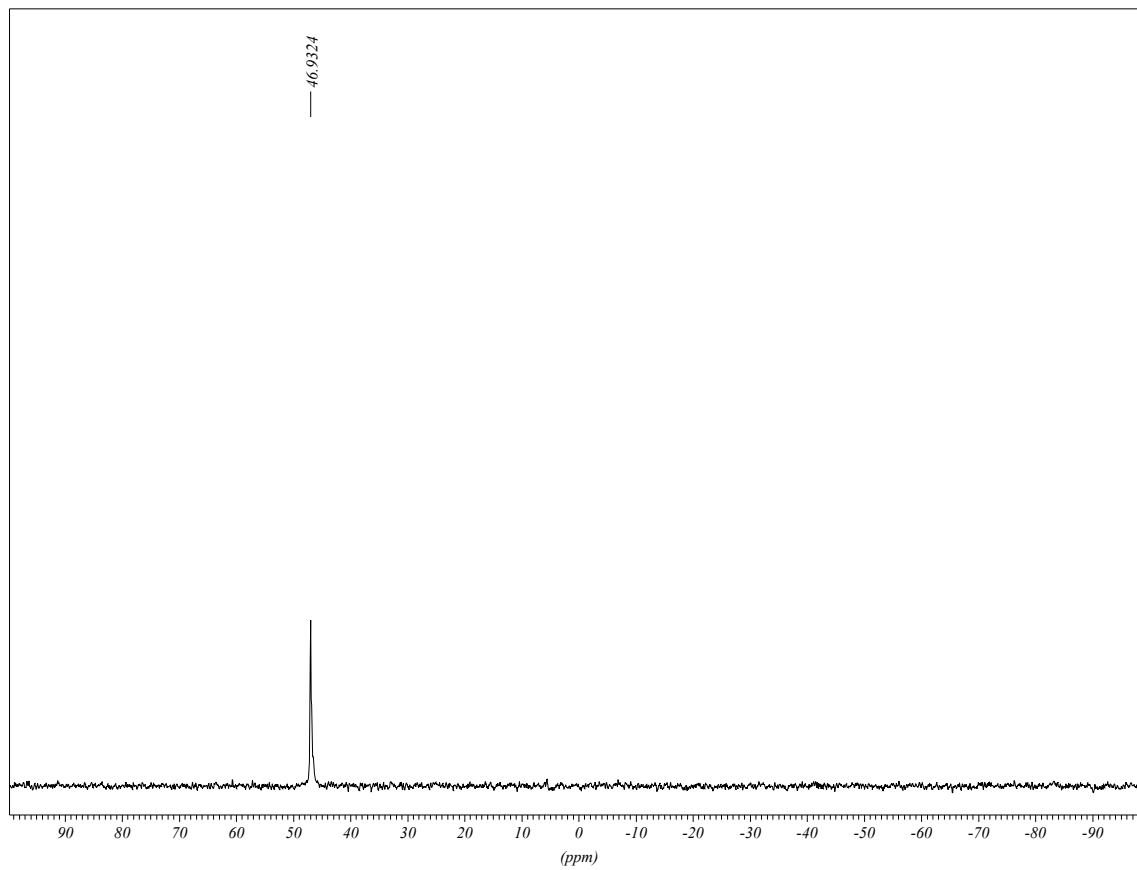
¹H RMN (CDCl₃)



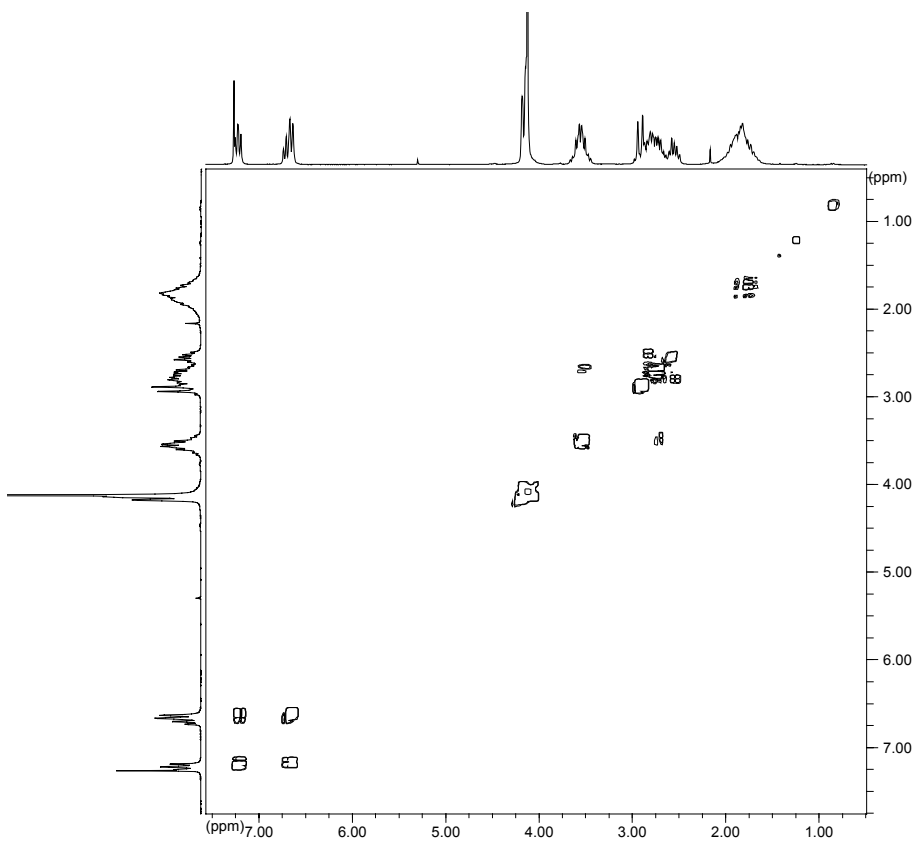
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



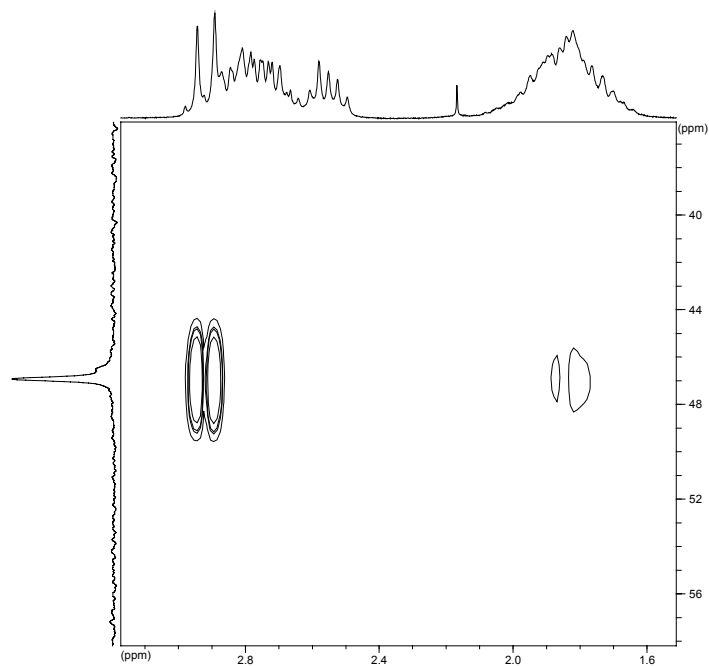
$^{31}\text{P}\{^1\text{H}\}$ RMN (CDCl_3)



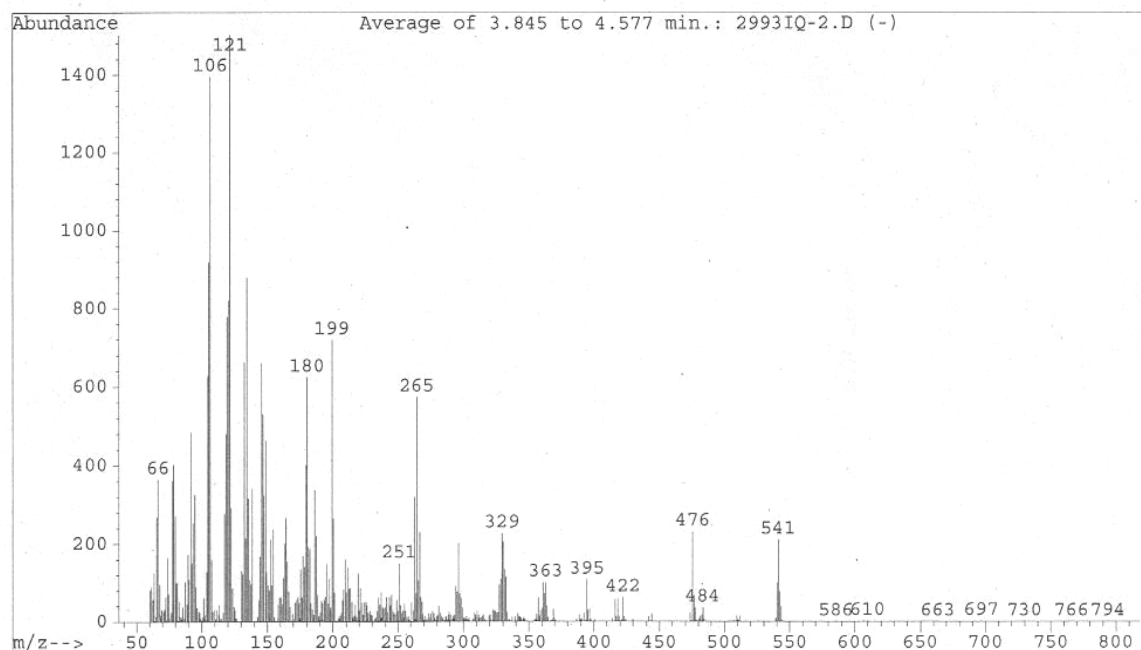
COSY (CDCl₃)



HMBC (CDCl₃)

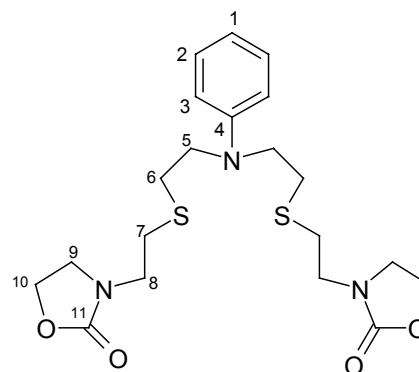


ESPECTROSCÒPIA DE MASSES (IQ)

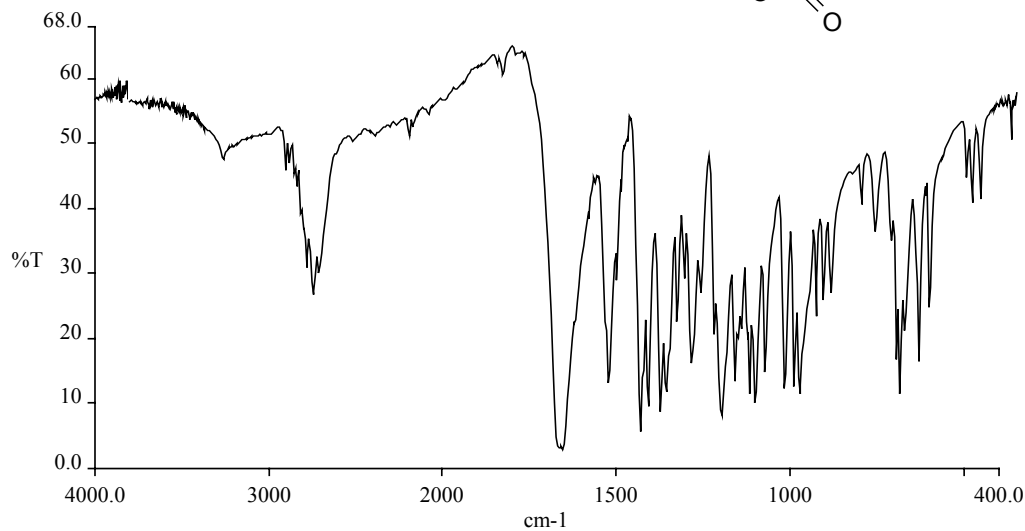


3 Derivats de l'oxazolidin-2-ona

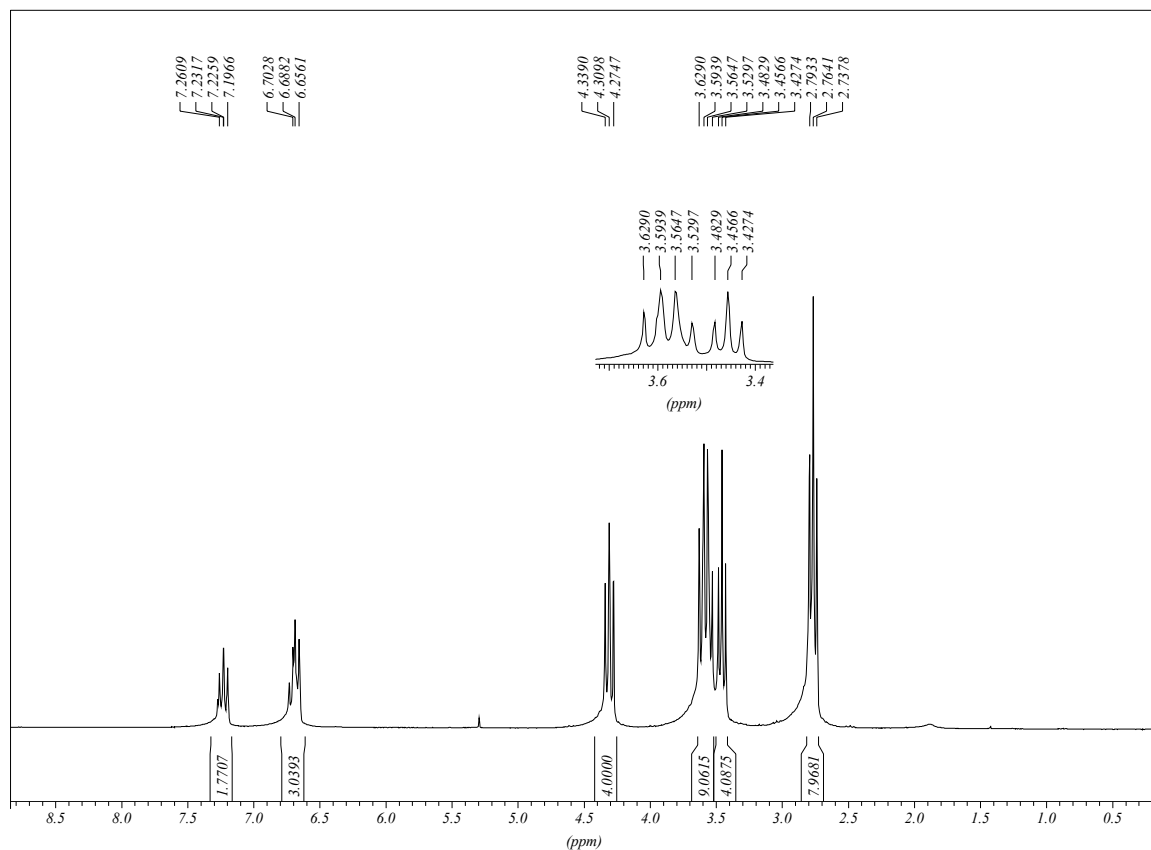
- N,N-bis(5-(N-oxazolidin-2-ona)-3-tiapentil)anilina (8)



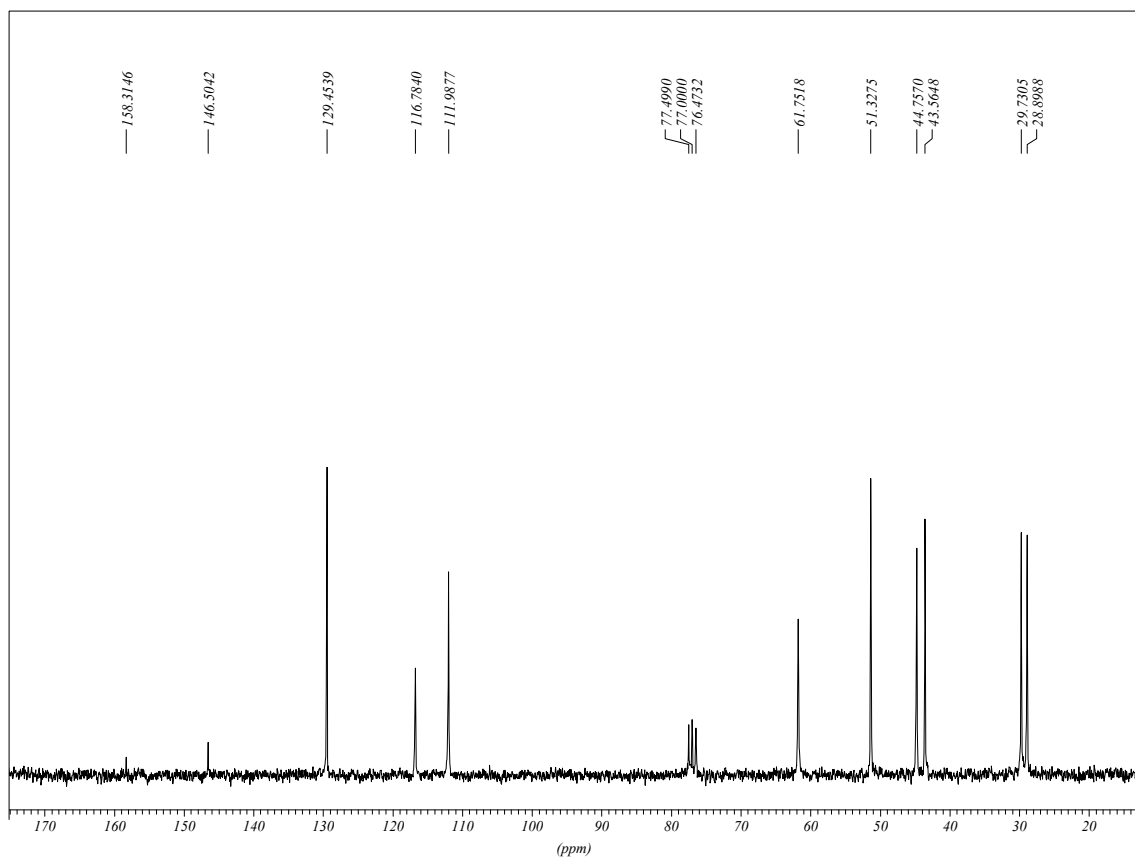
IR (KBr)



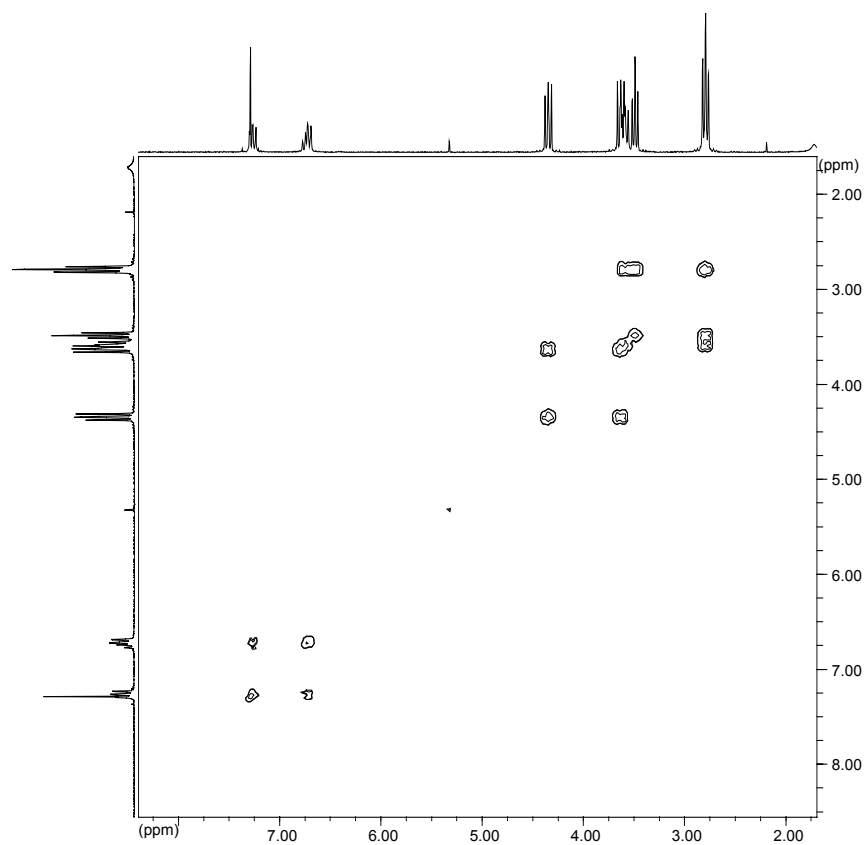
^1H RMN (CDCl_3)



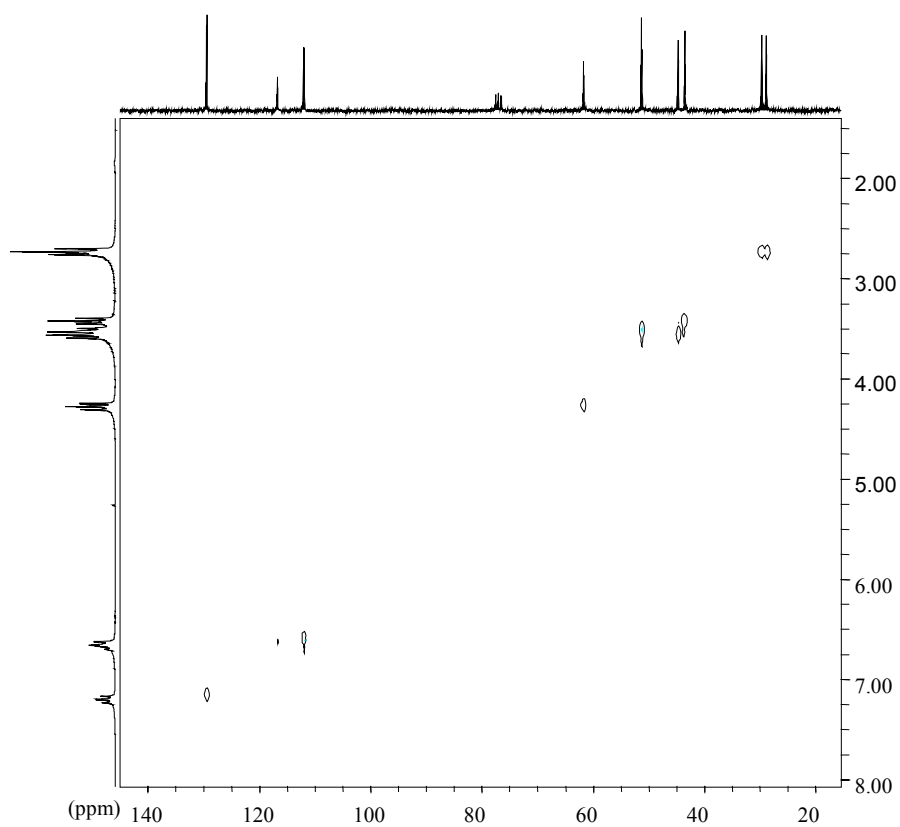
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



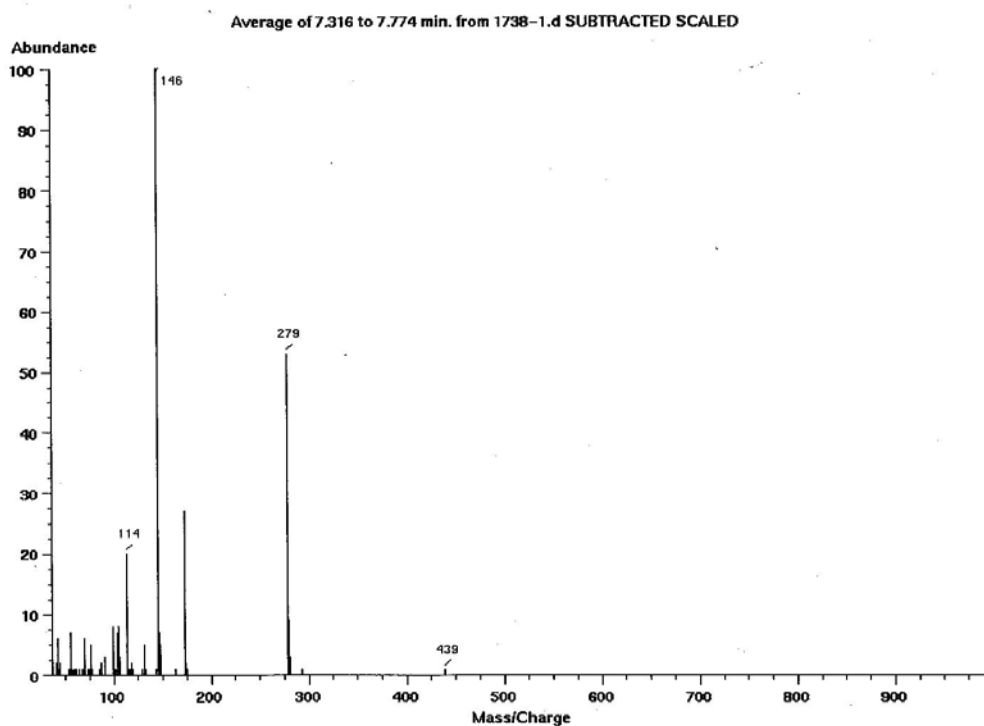
COSY (CDCl₃)



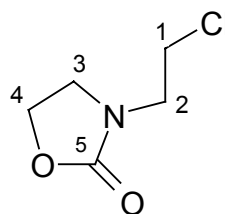
HMQC (CDCl₃)



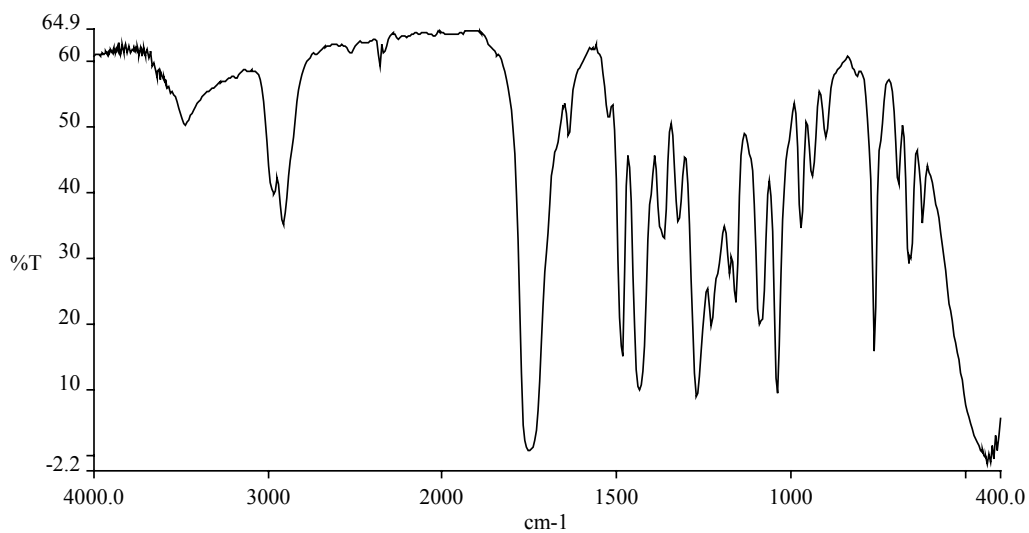
ESPECTROSCÒPIA DE MASSES (IQ)



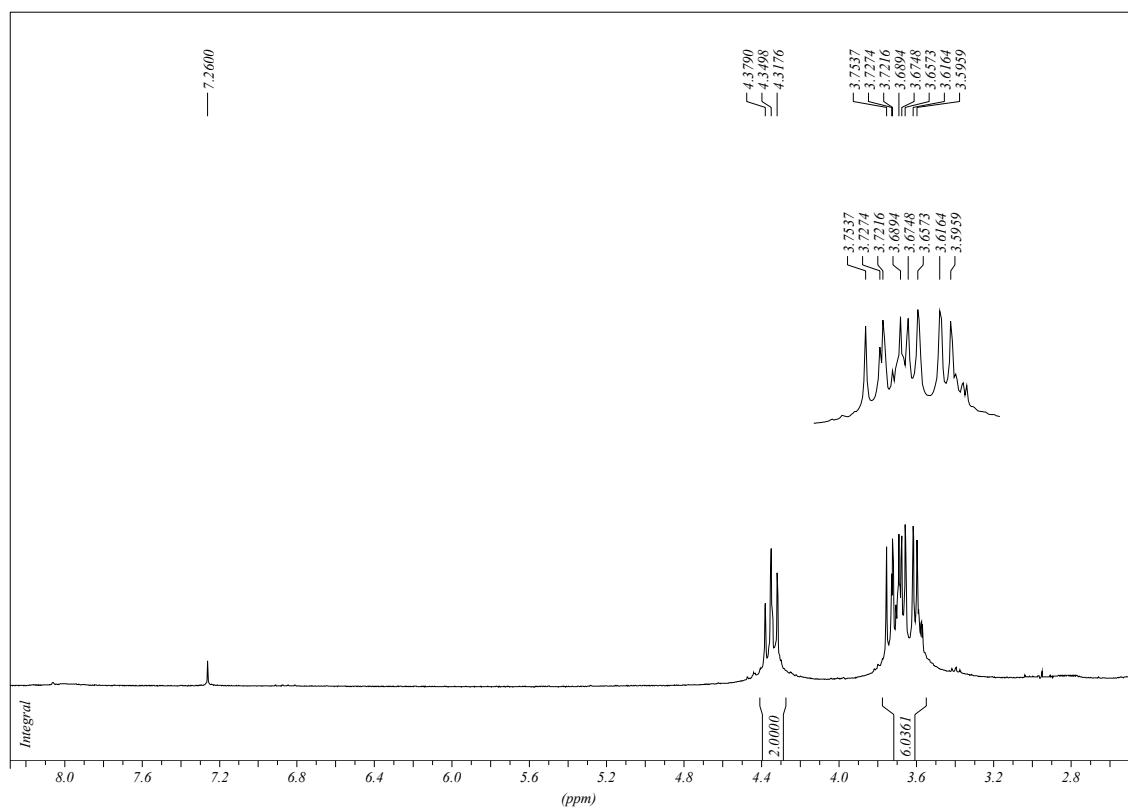
- 3-(2-cloroetil)oxazolidin-2-ona (9)



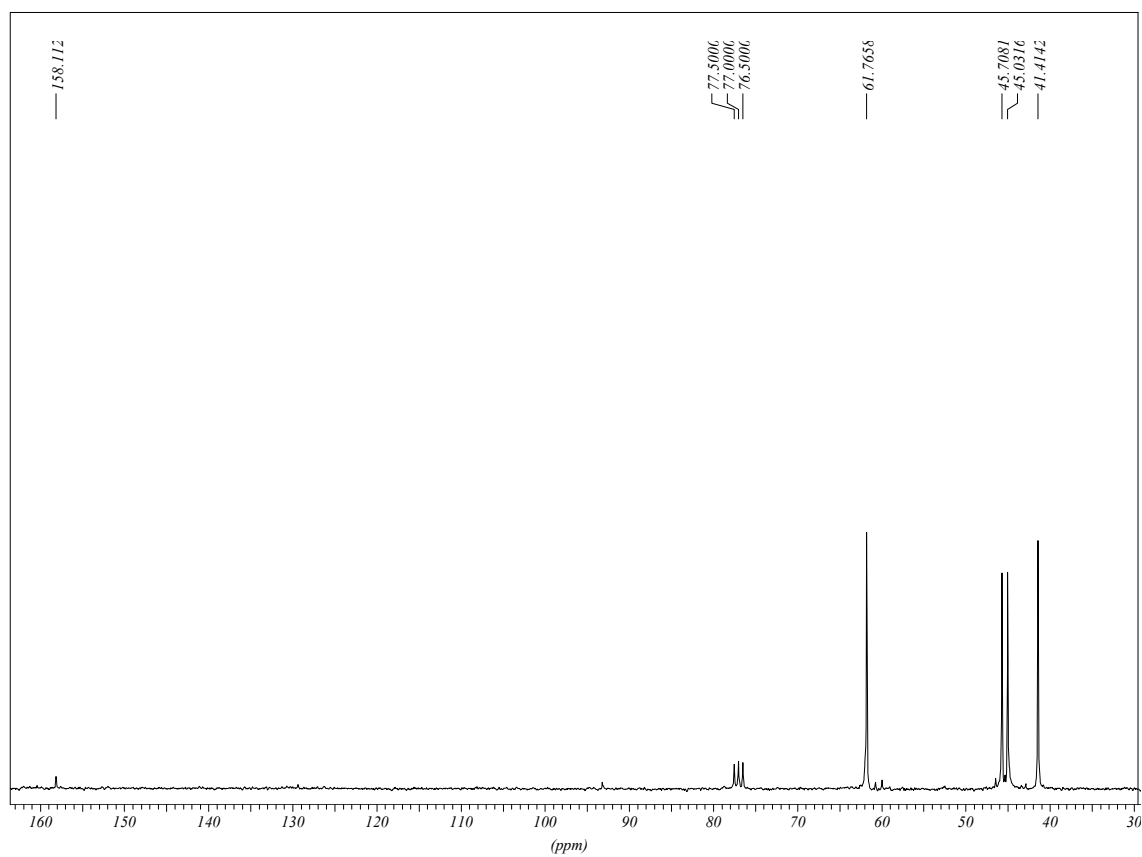
IR (NaCl)



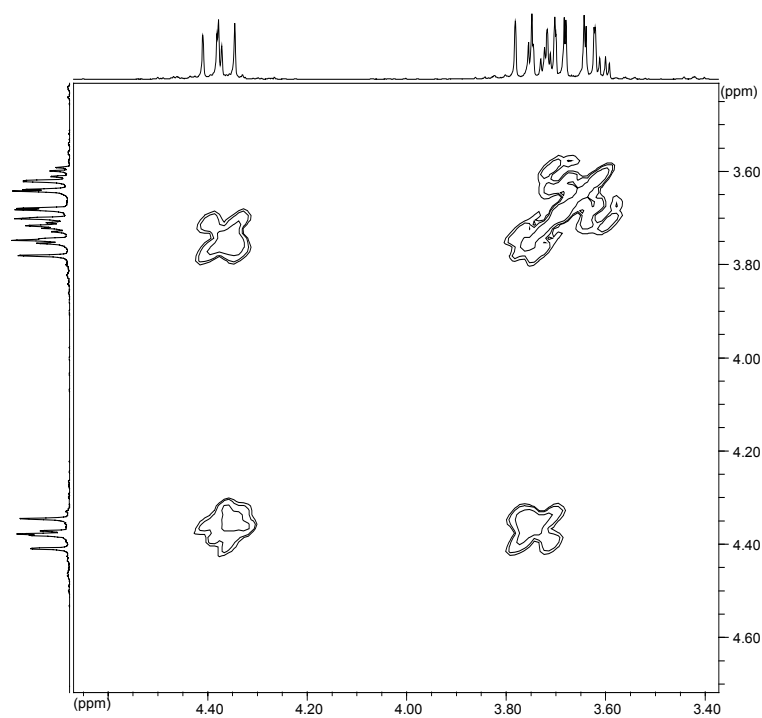
^1H RMN (CDCl_3)



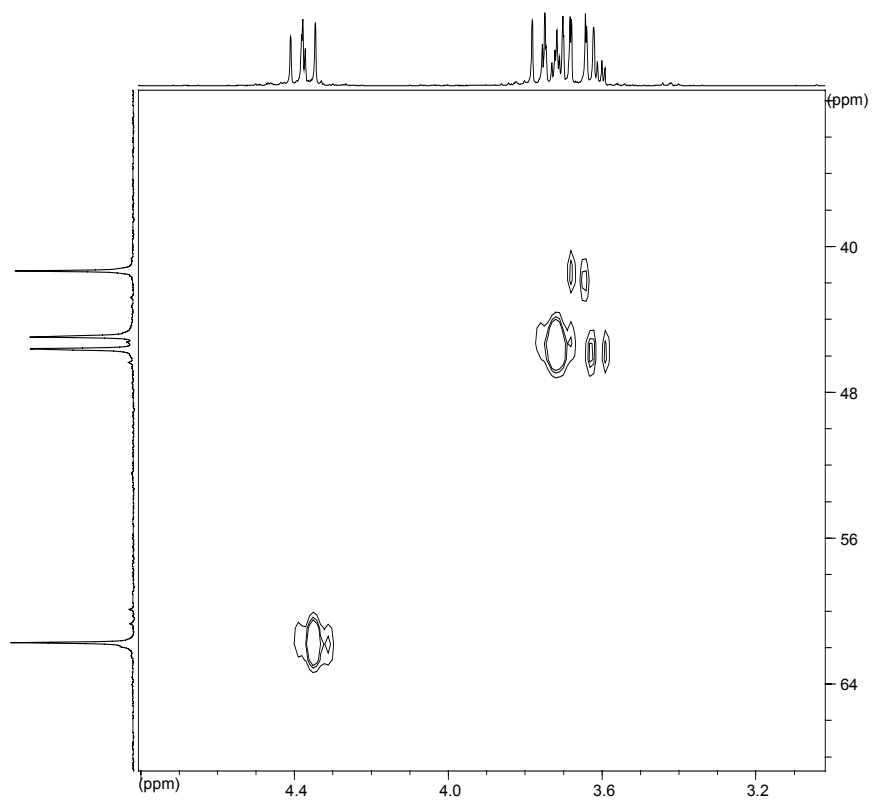
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



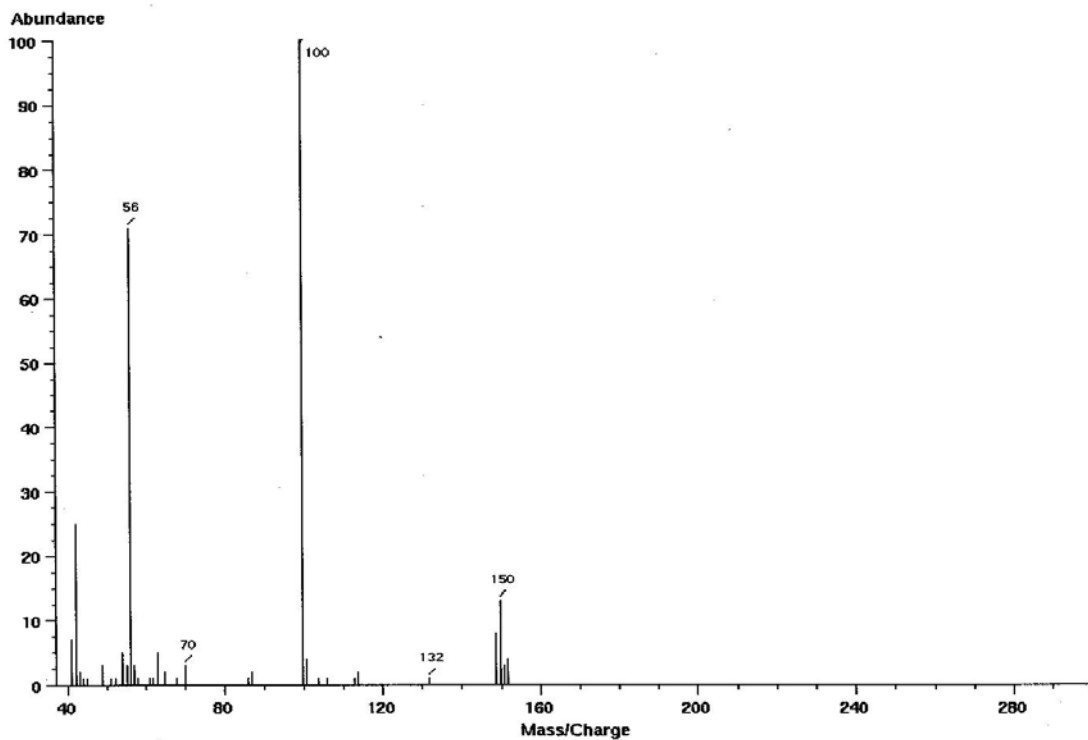
COSY (CDCl₃)



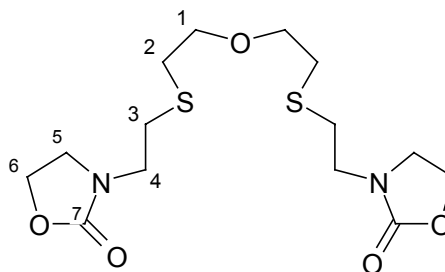
HMQC (CDCl₃)



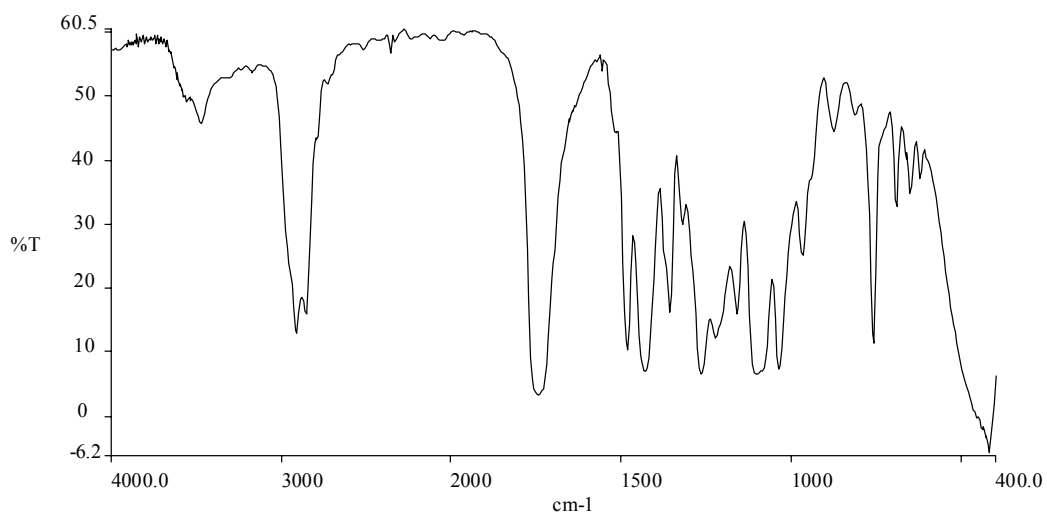
ESPECTROSCÒPIA DE MASSES (IQ)



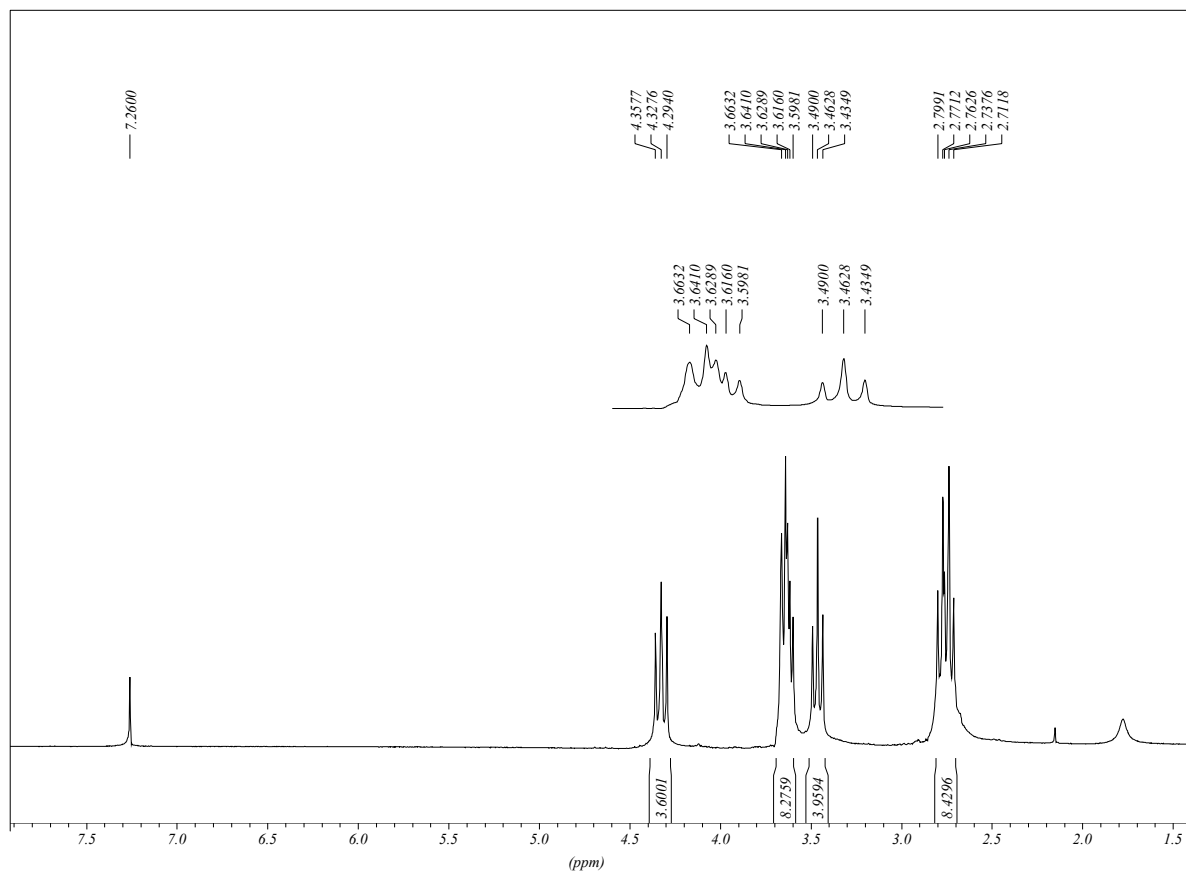
- 6-oxa-1,11-bis(N-oxazolidin-2-ona)-2,10-ditiaundecà (10)



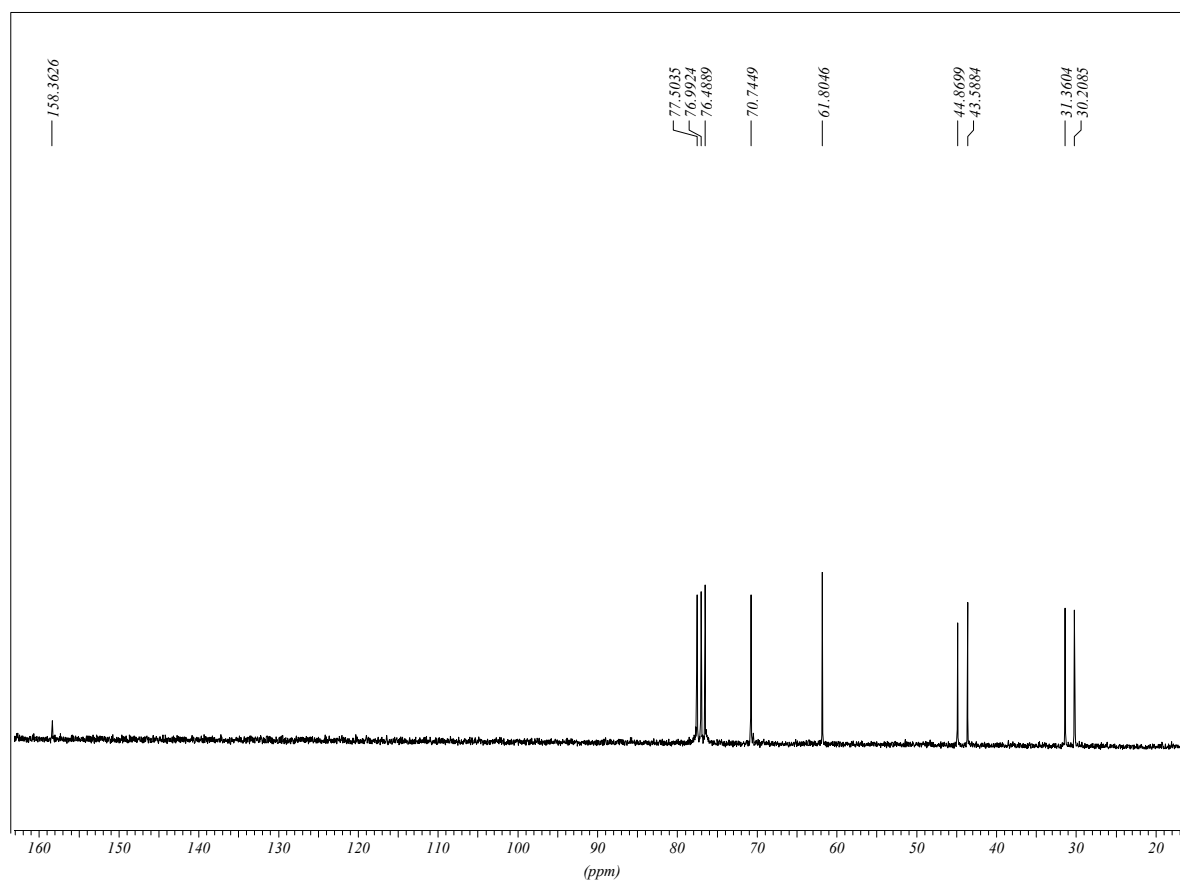
IR (NaCl)



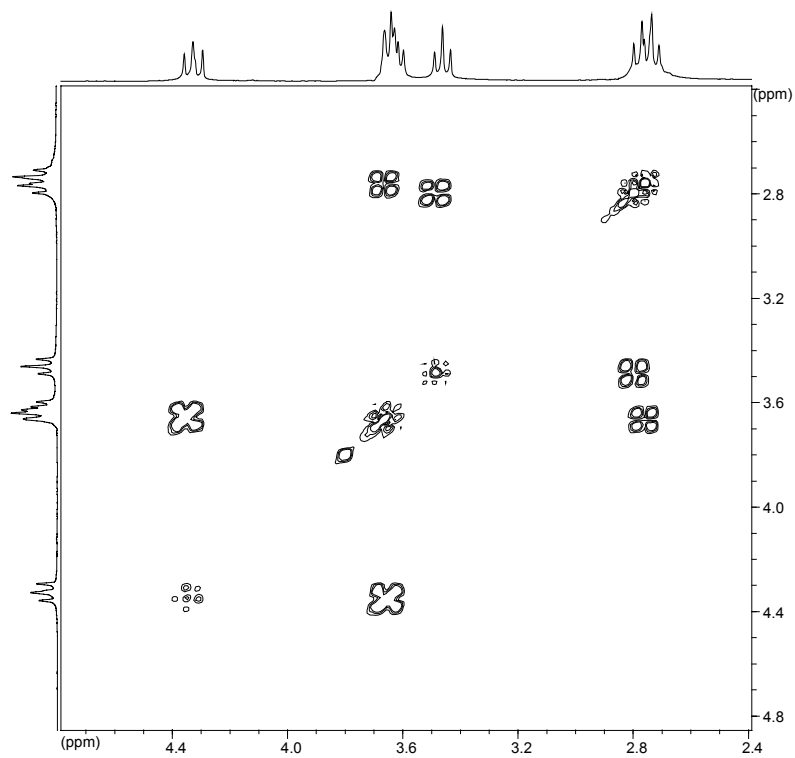
^1H RMN (CDCl_3)



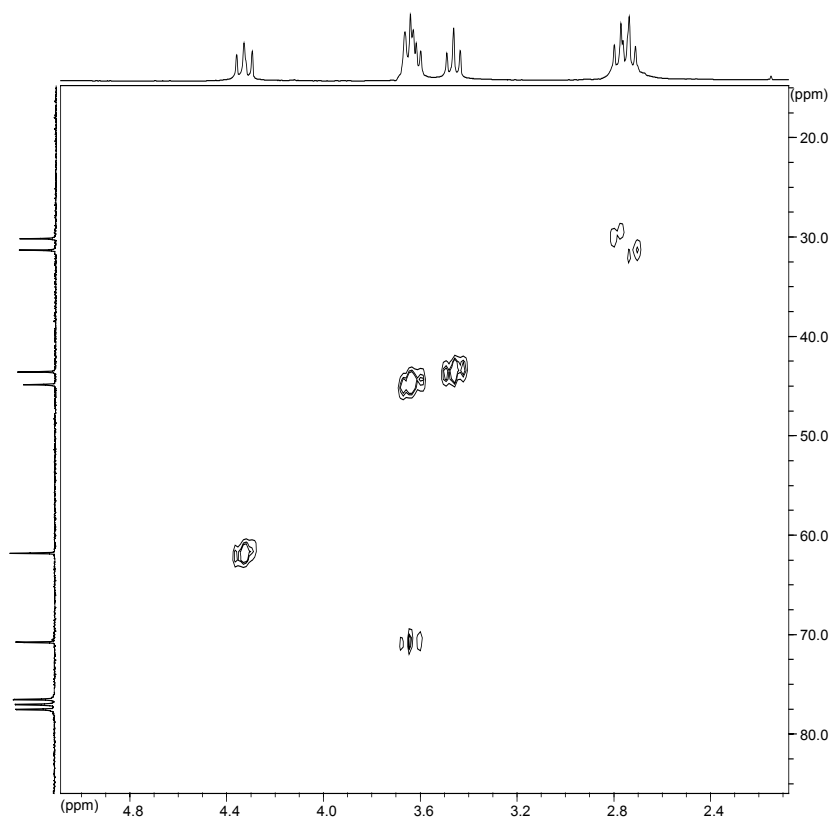
$^{13}\text{C}\{^1\text{H}\}$ RMN (CDCl_3)



COSY (CDCl₃)



HMQC (CDCl₃)



ESPECTROSCÒPIA DE MASSES (IQ)

