Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Symmetric and unsymmetric 3,3'-linked bispyrroles via ring-enlargement reactions of furan-derived donor-acceptor cyclopropanes

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Supplementary information

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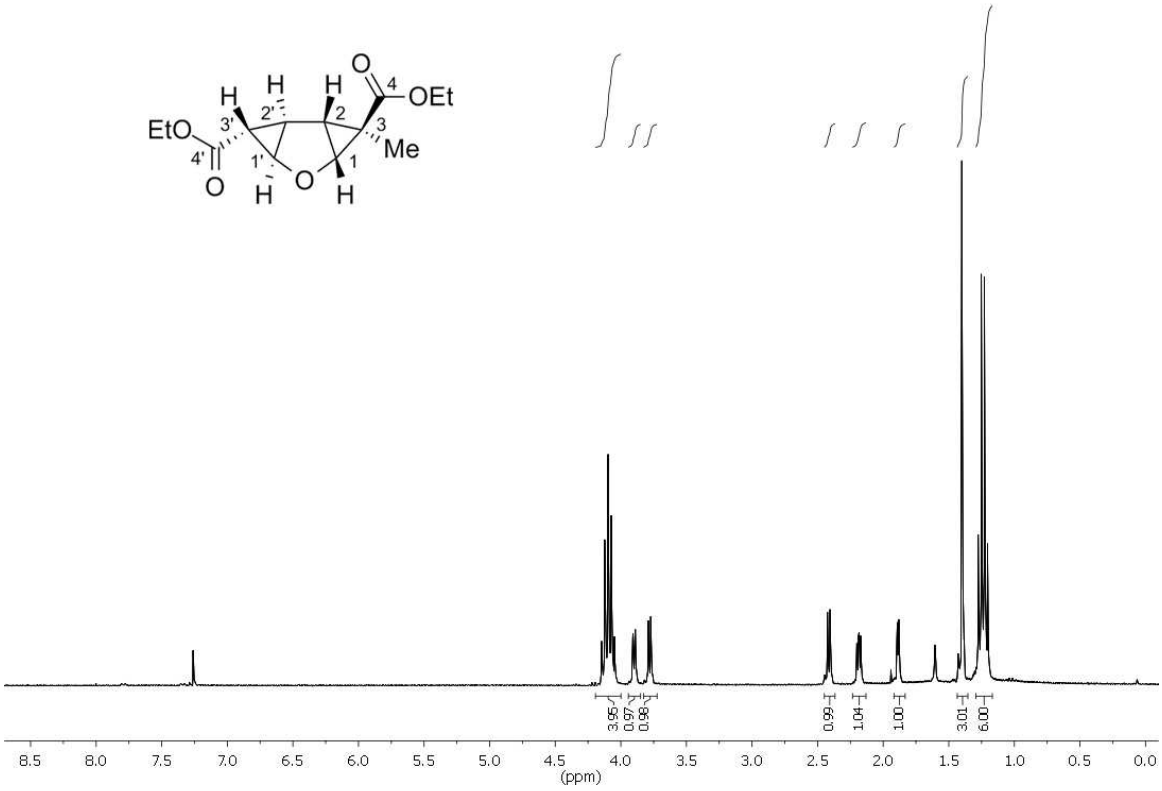

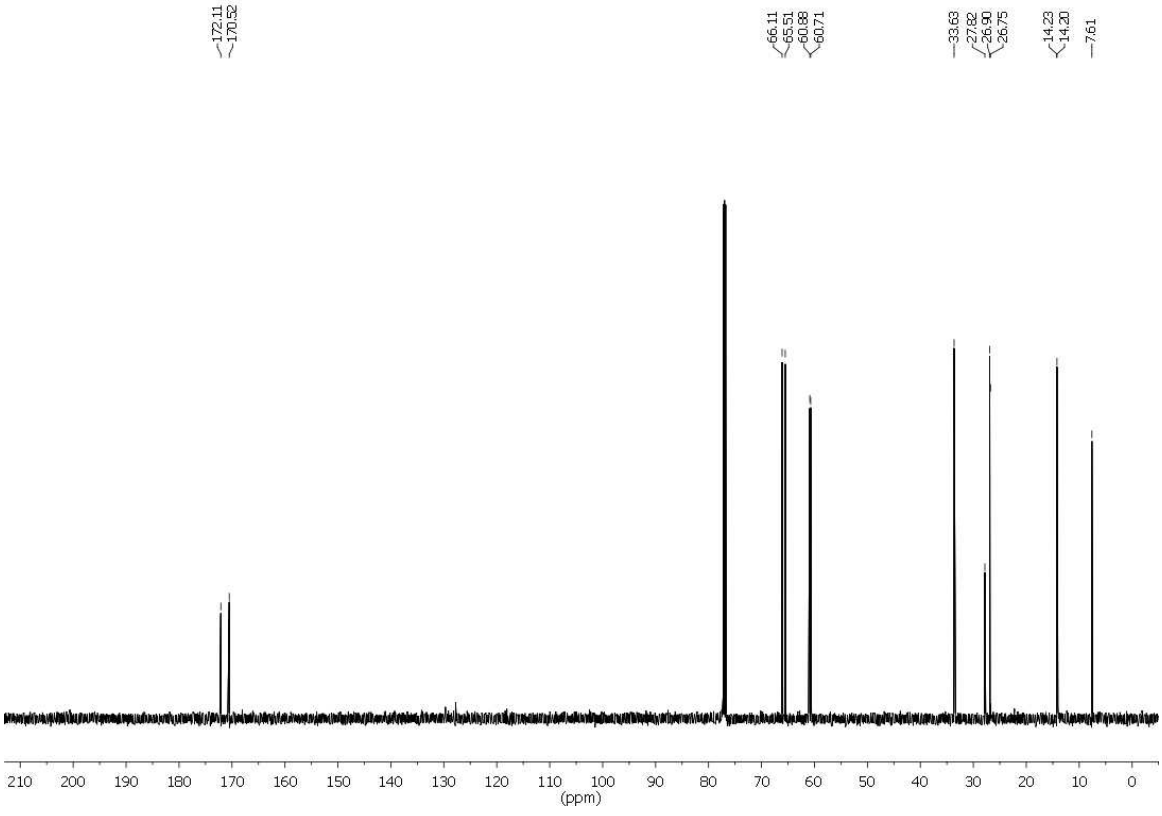

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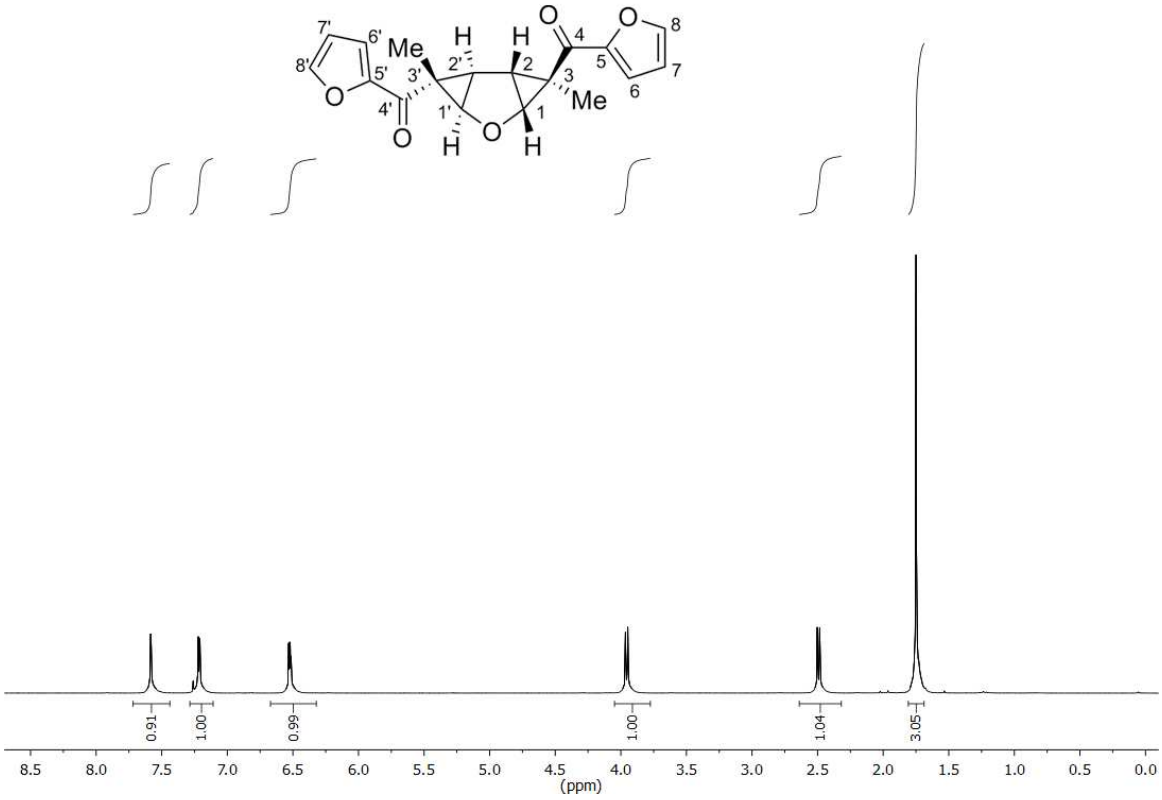

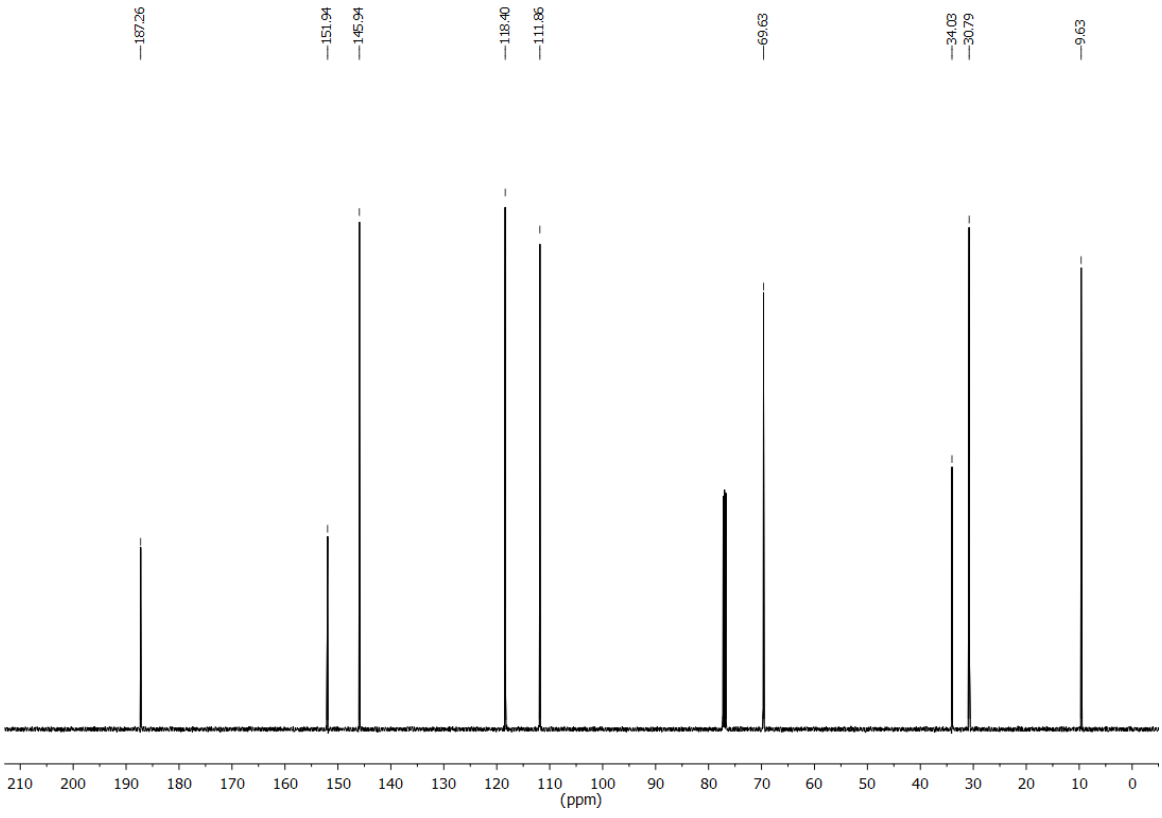

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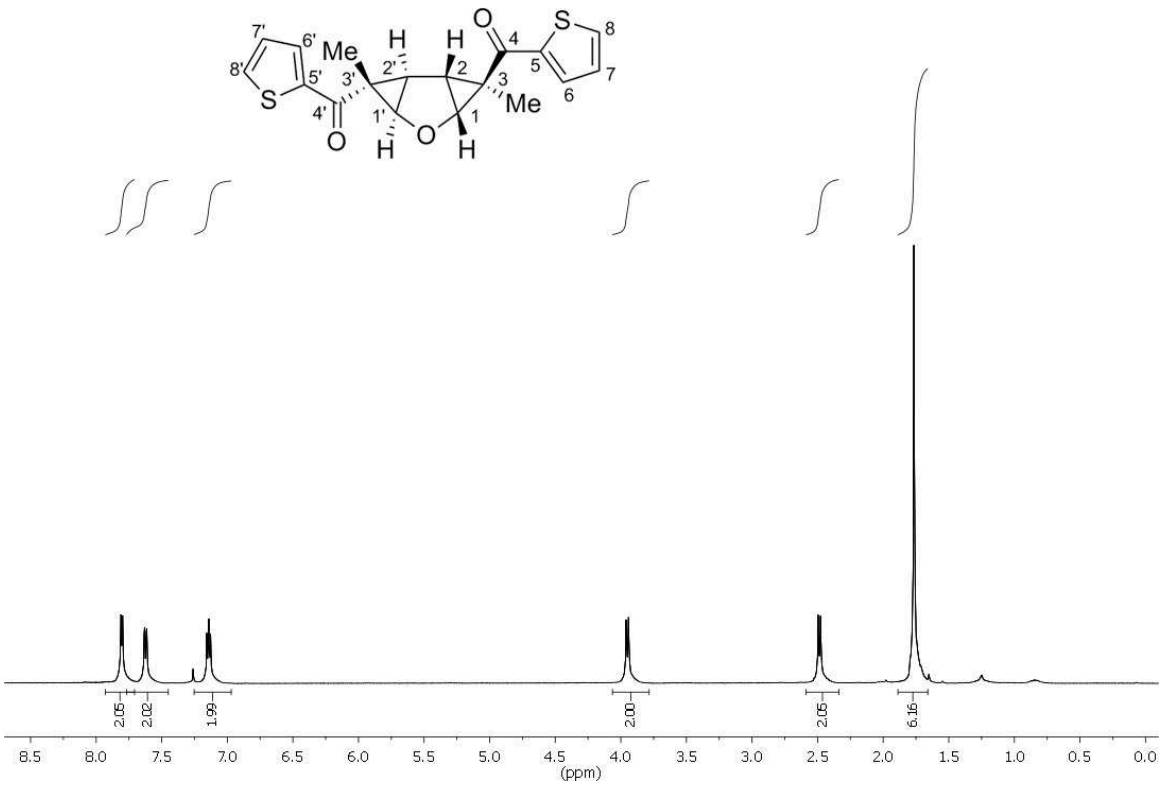

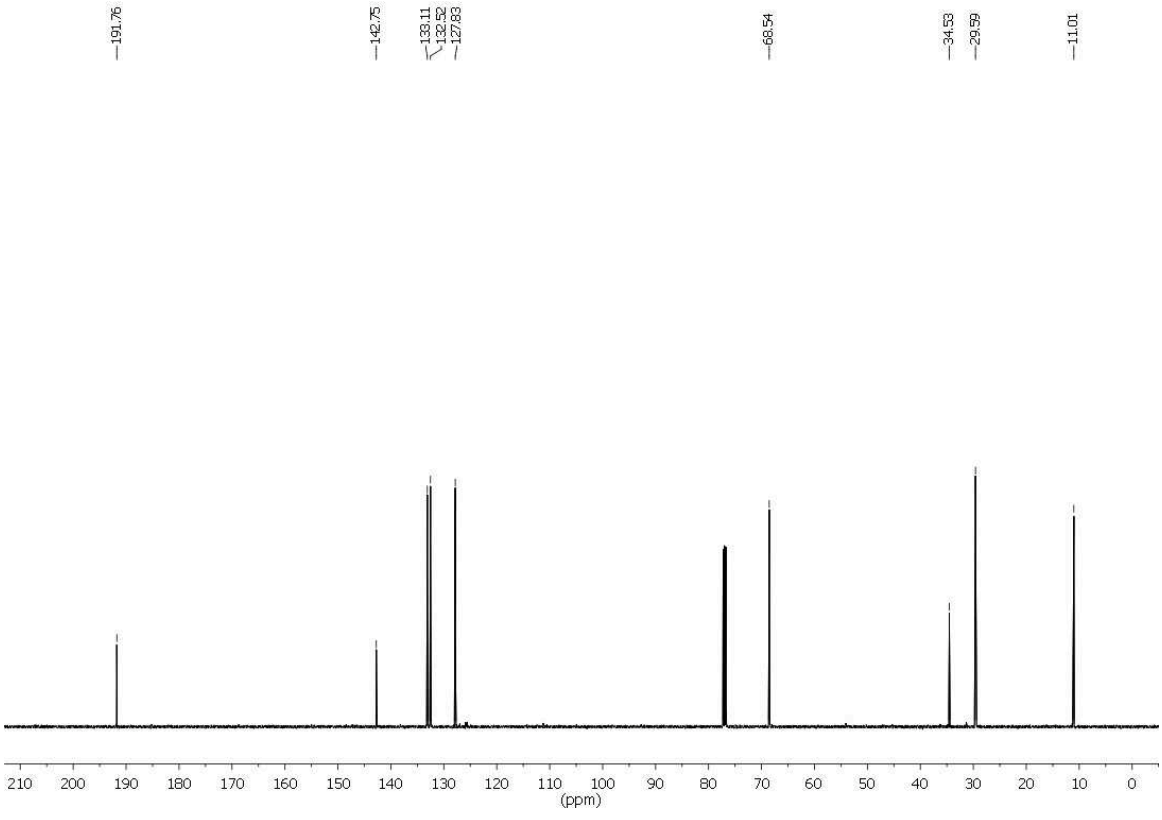

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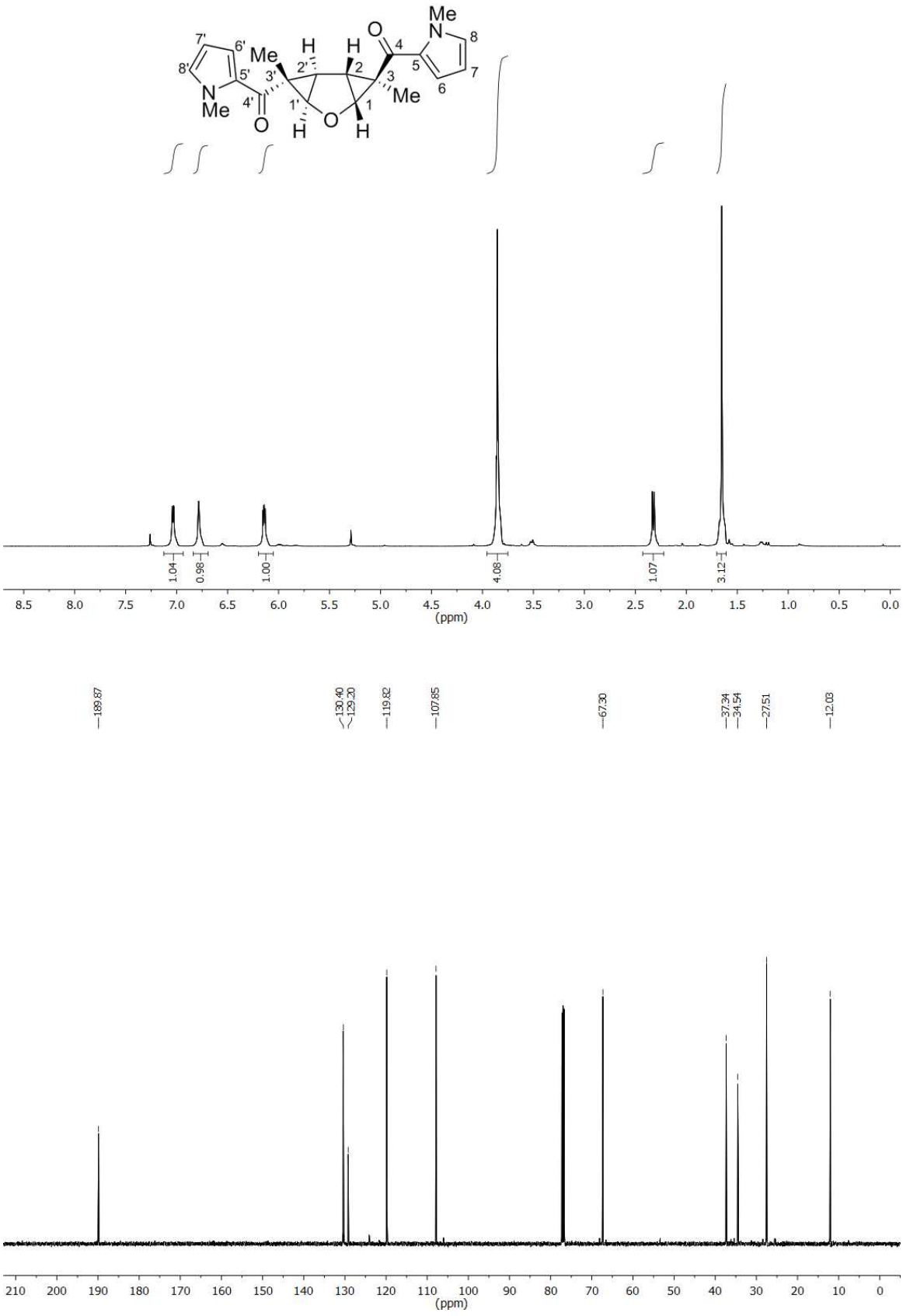

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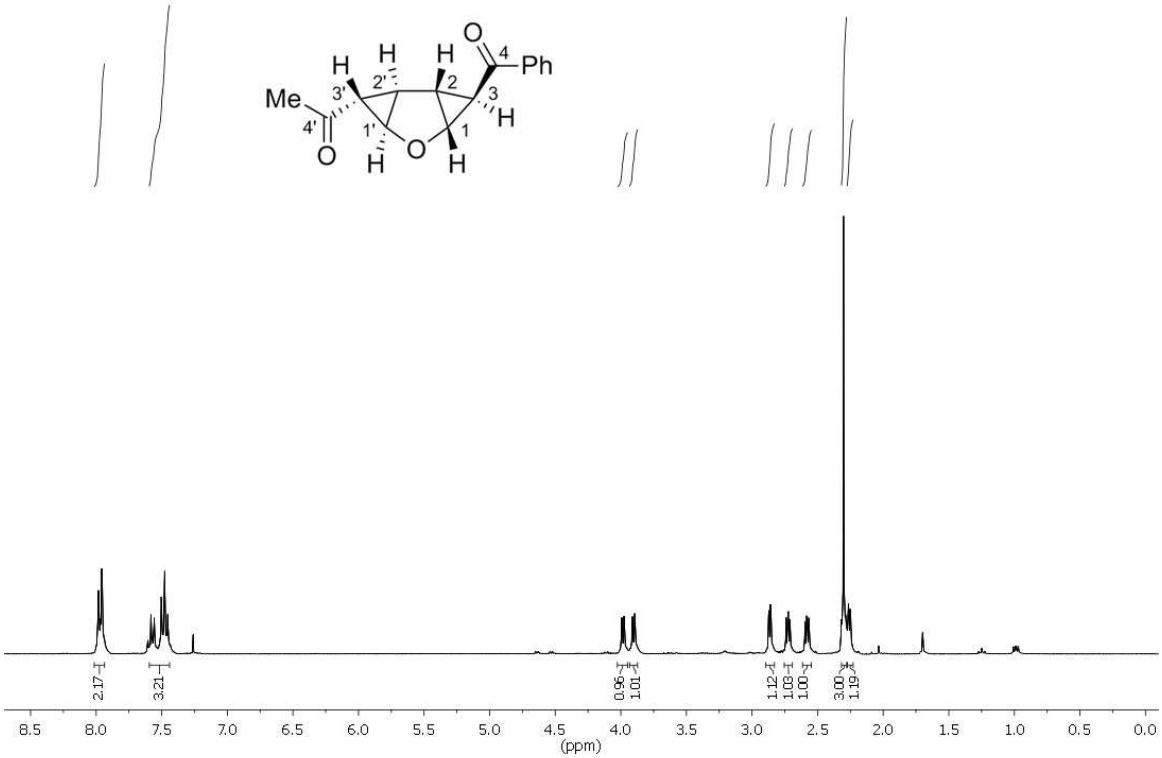

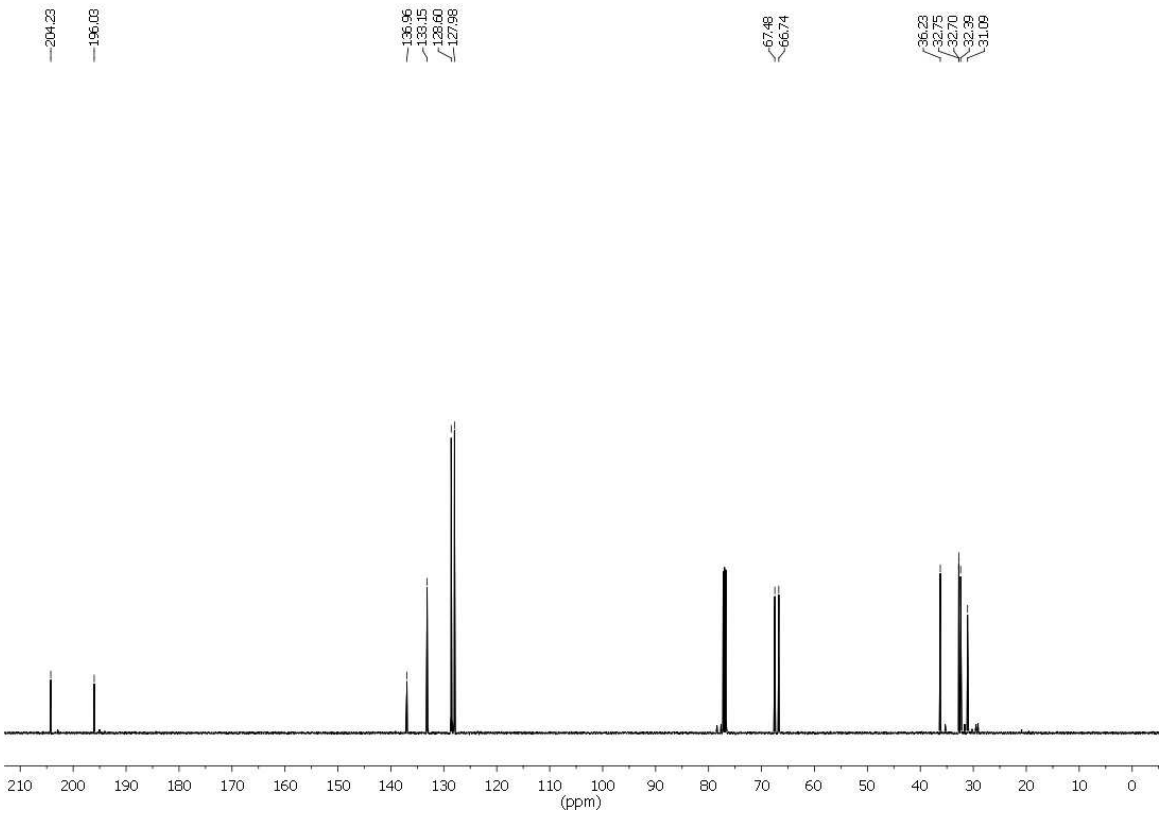

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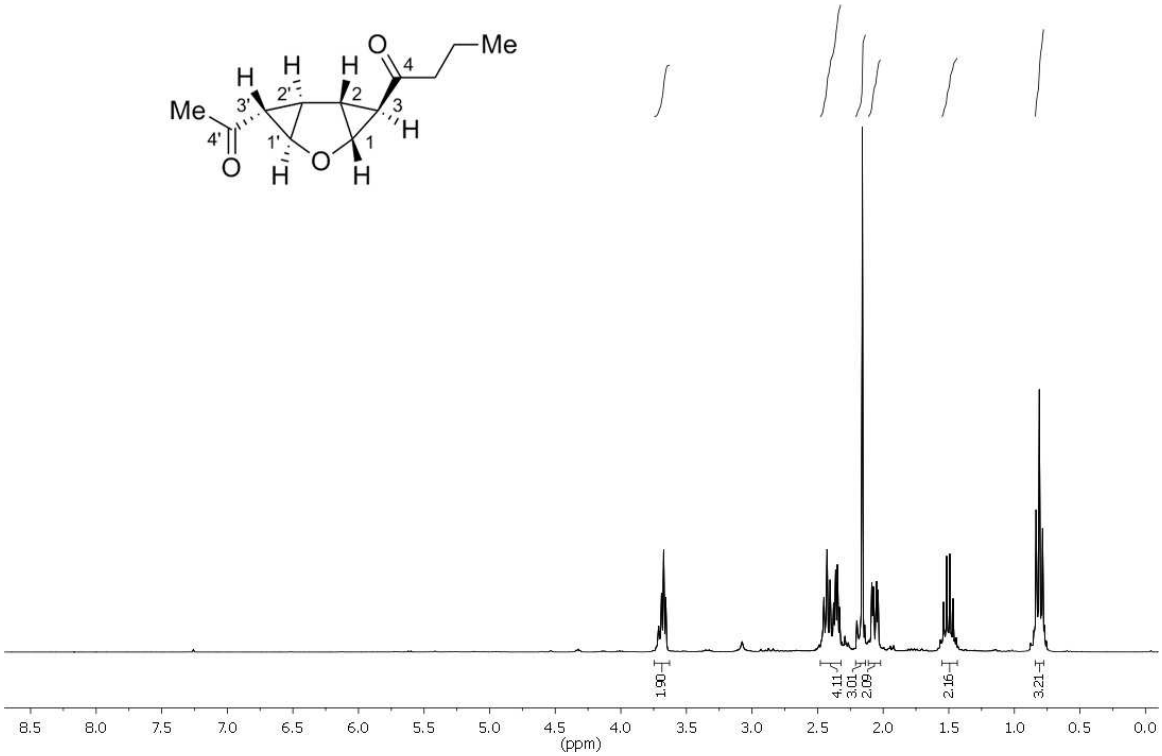

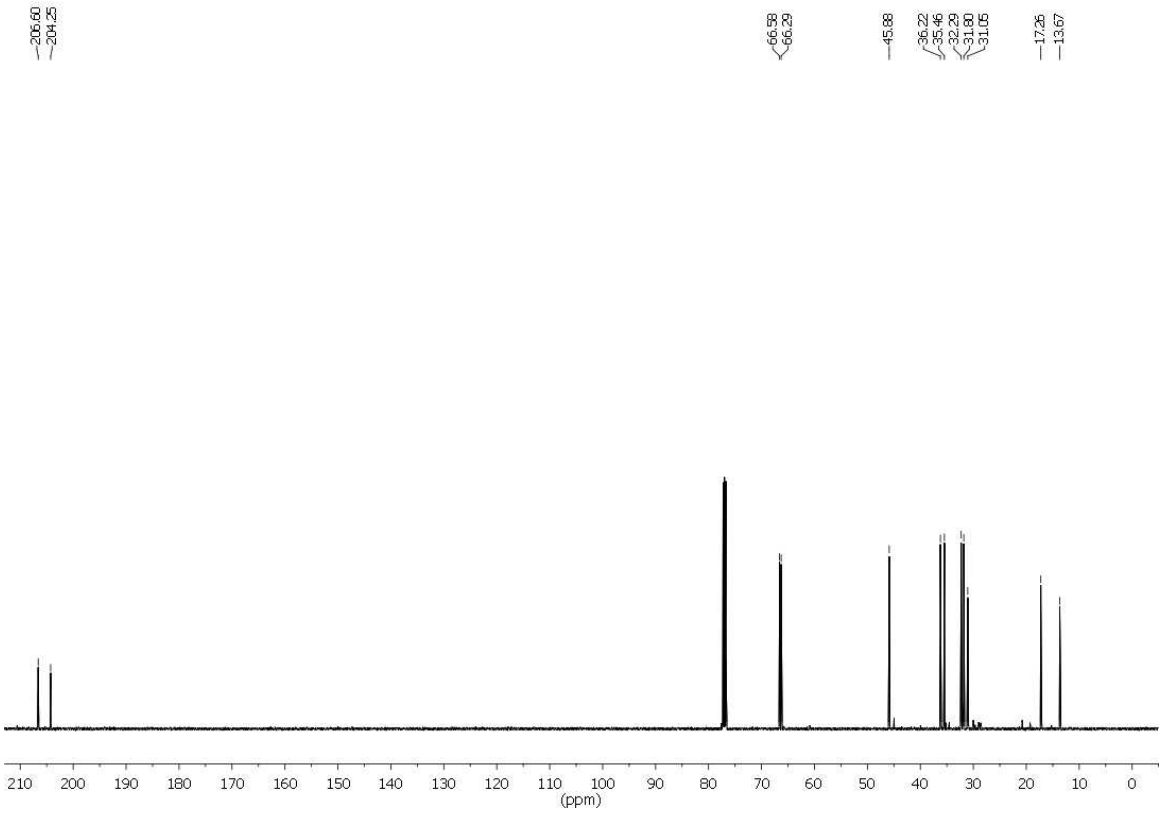

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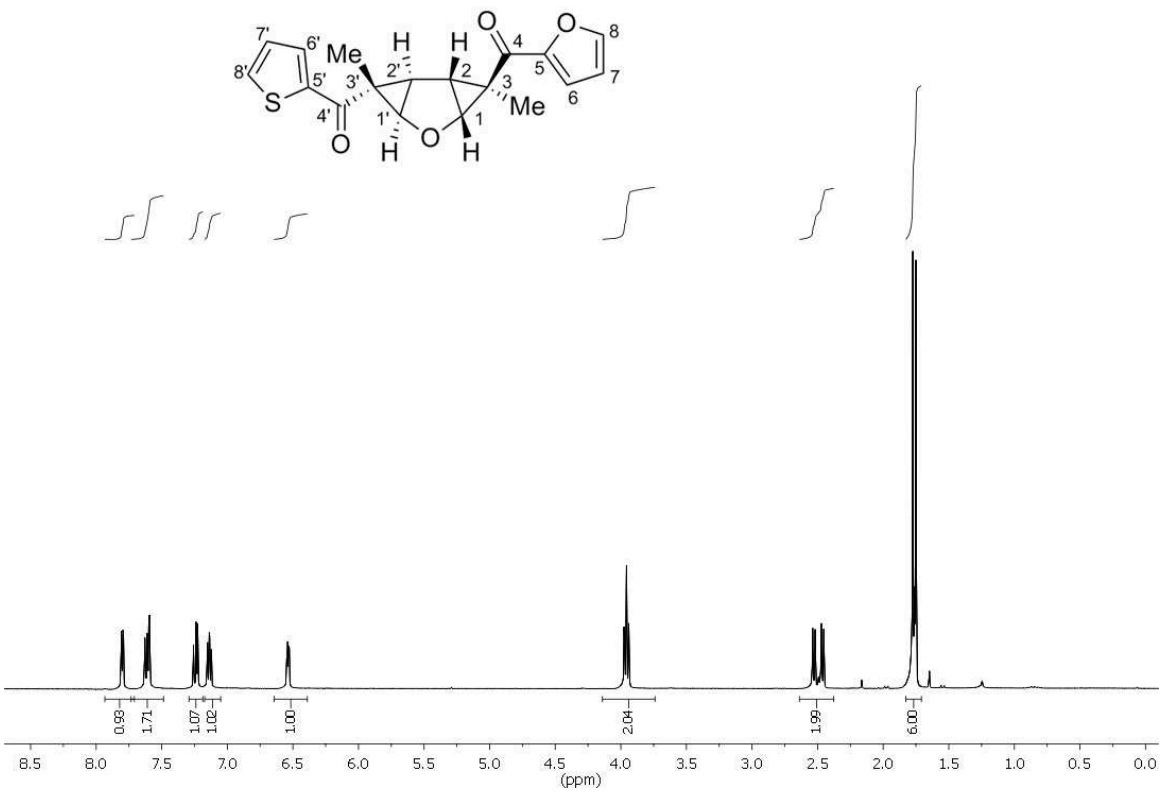

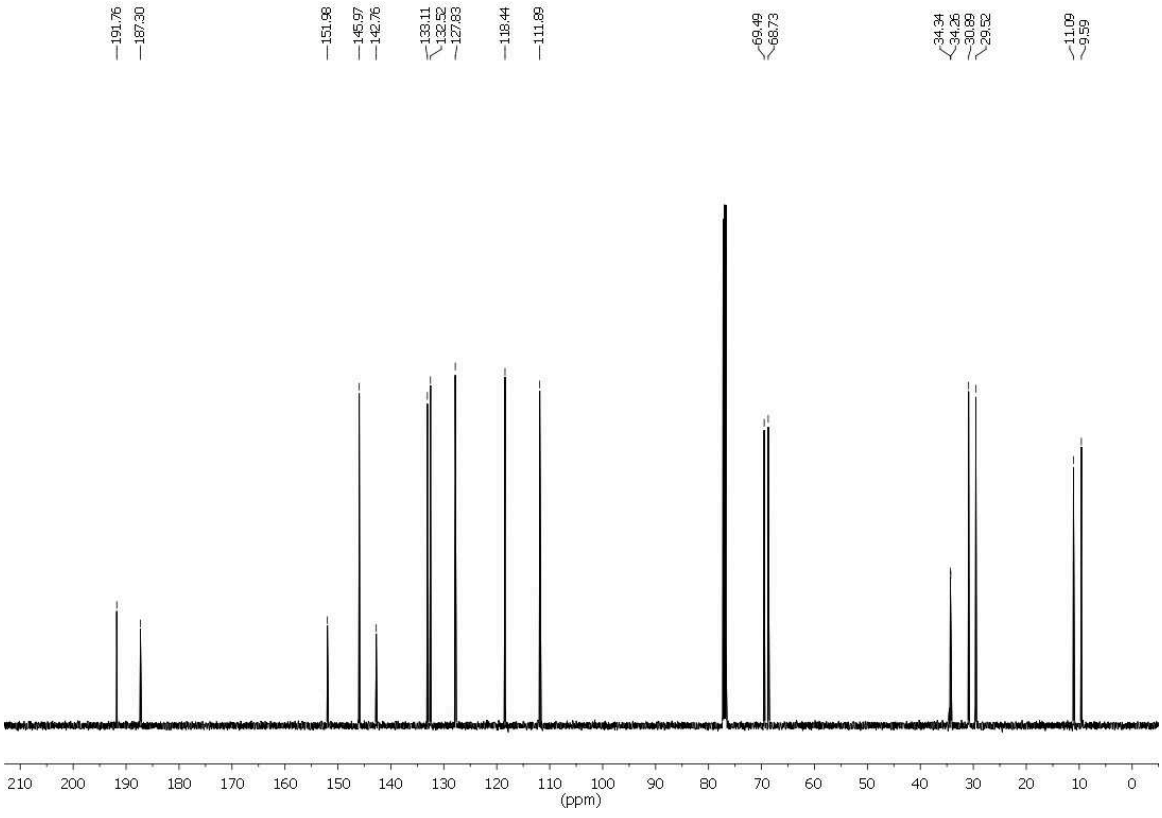

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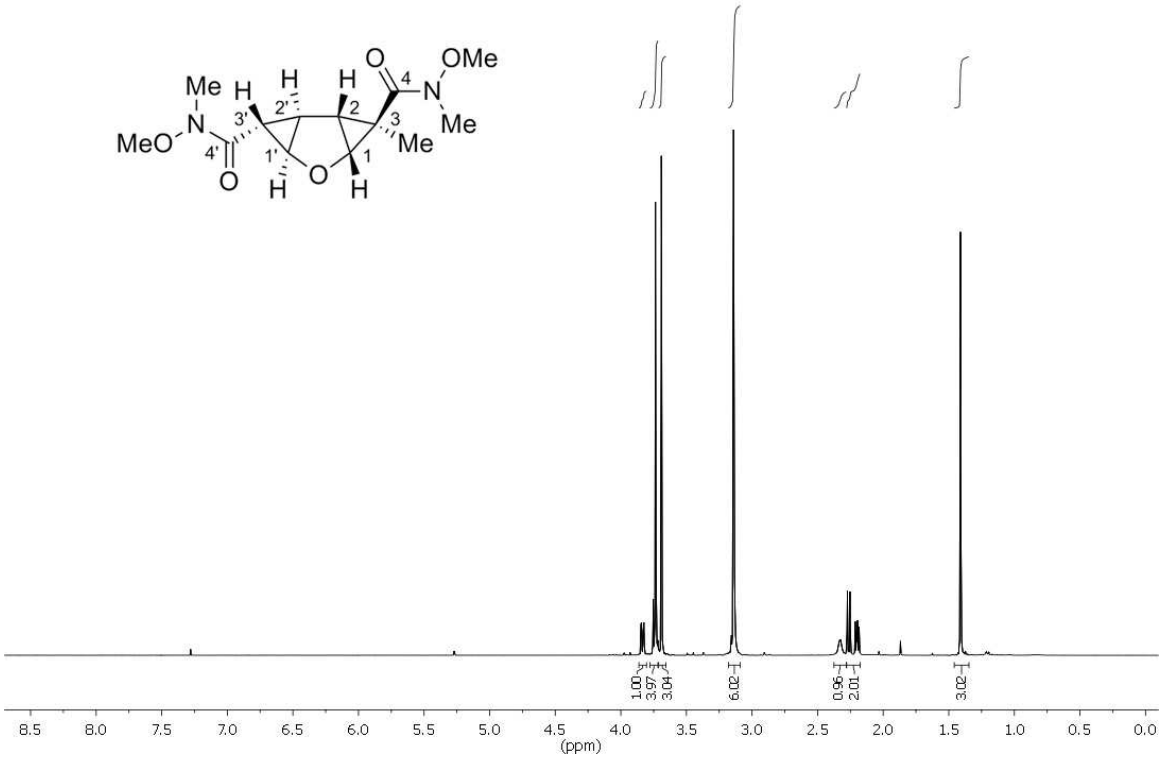

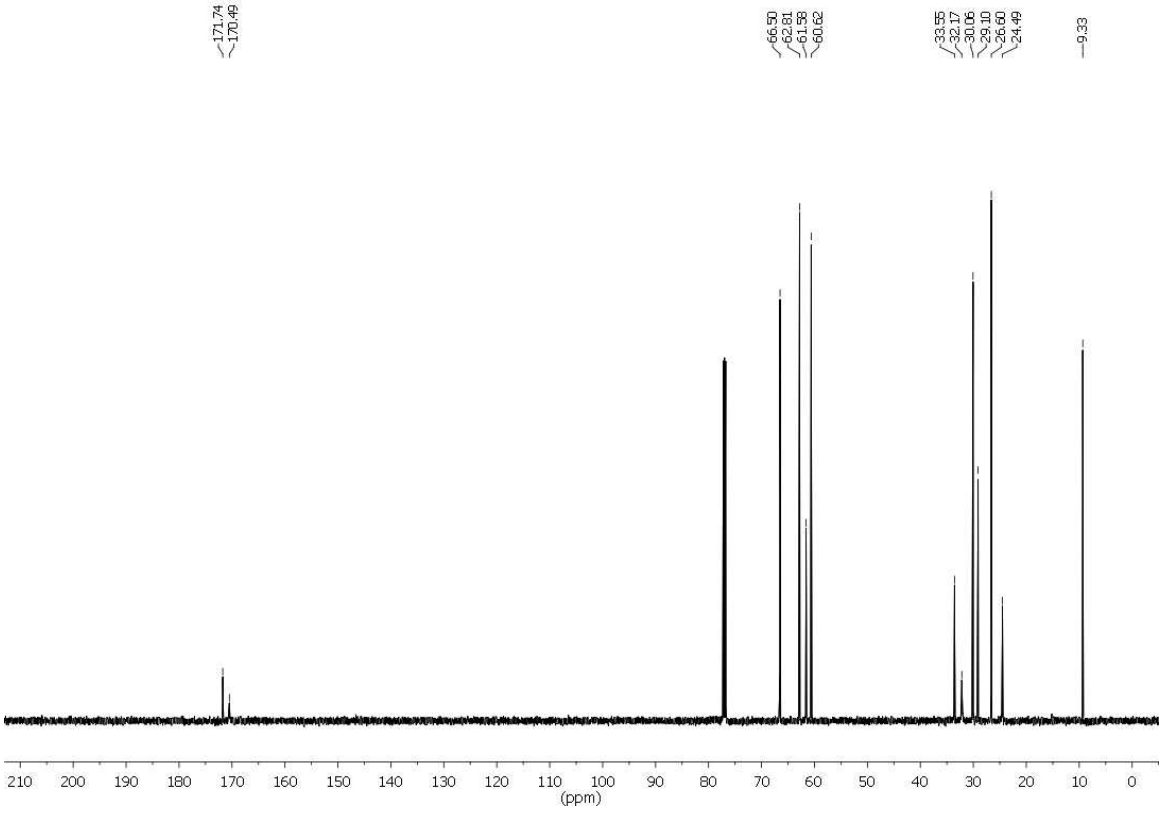

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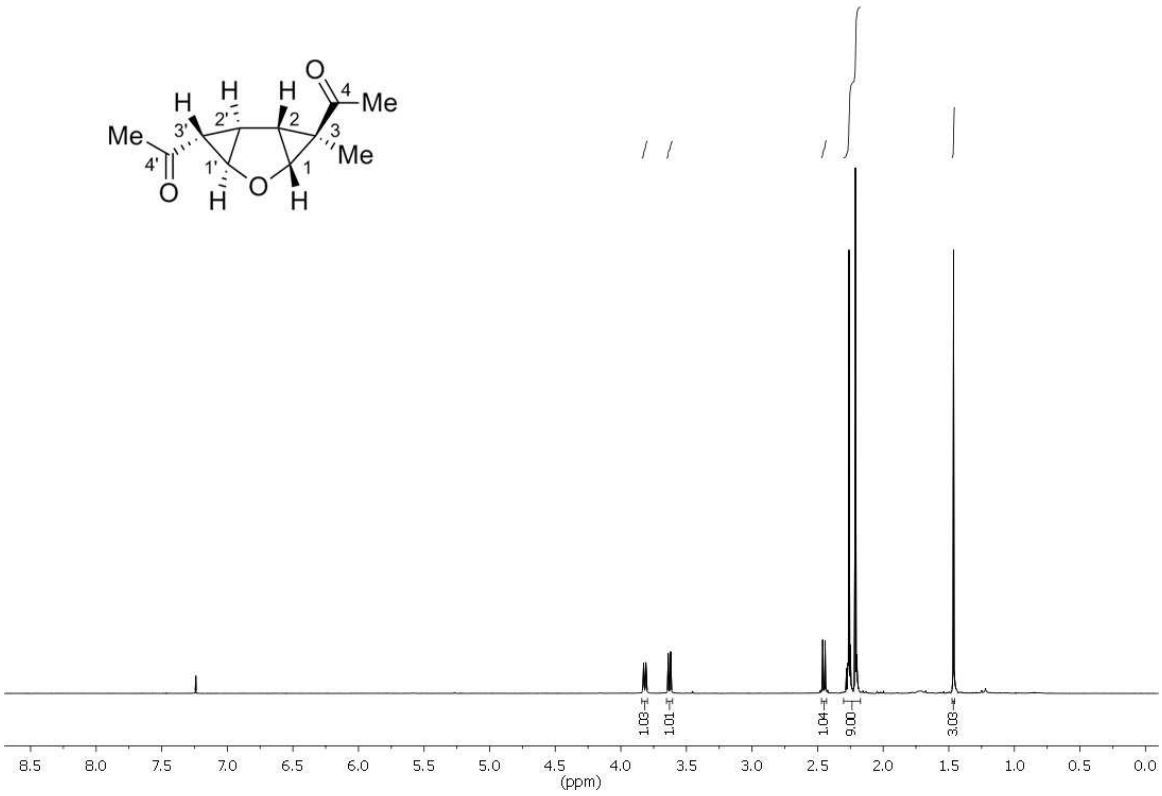

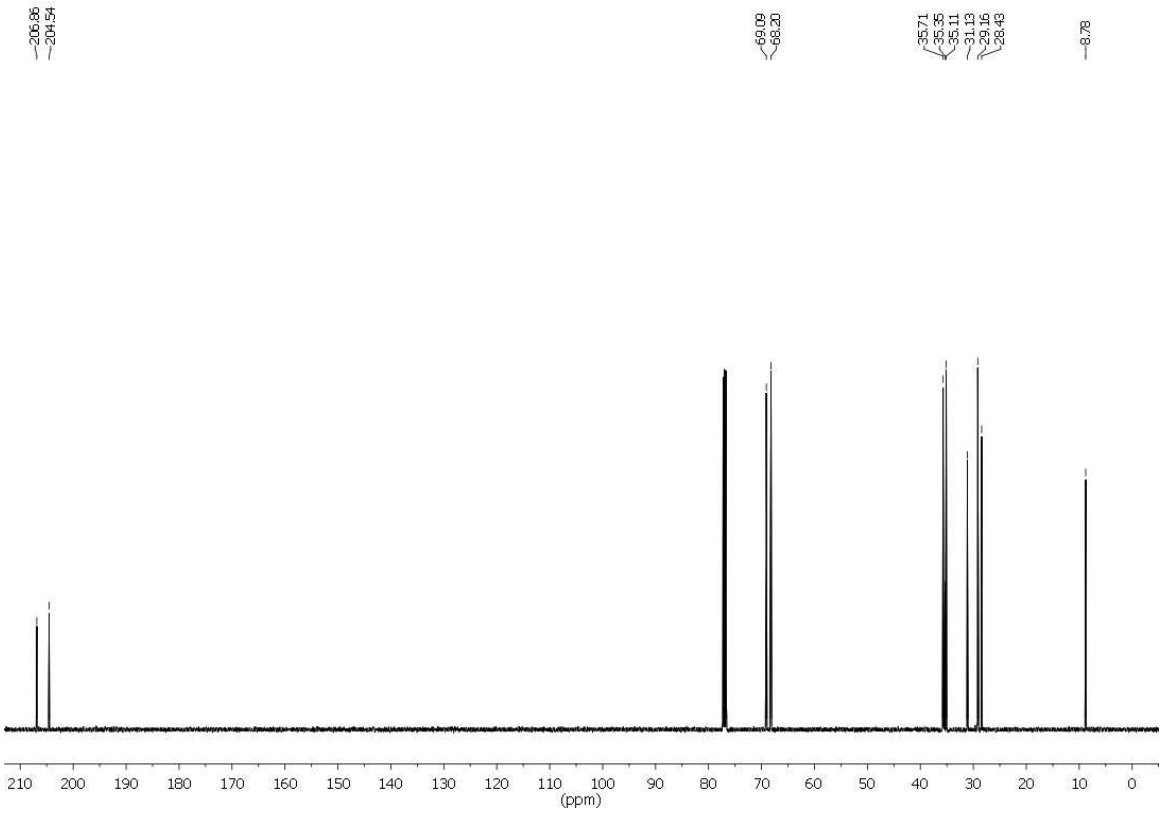

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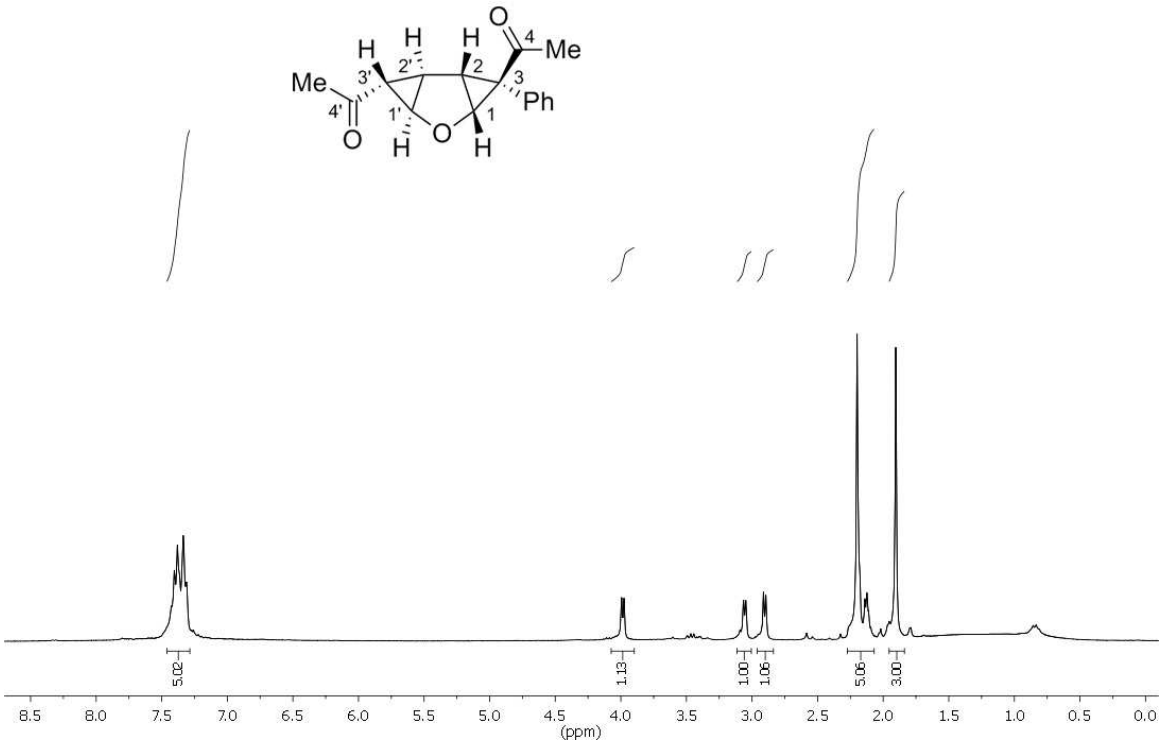

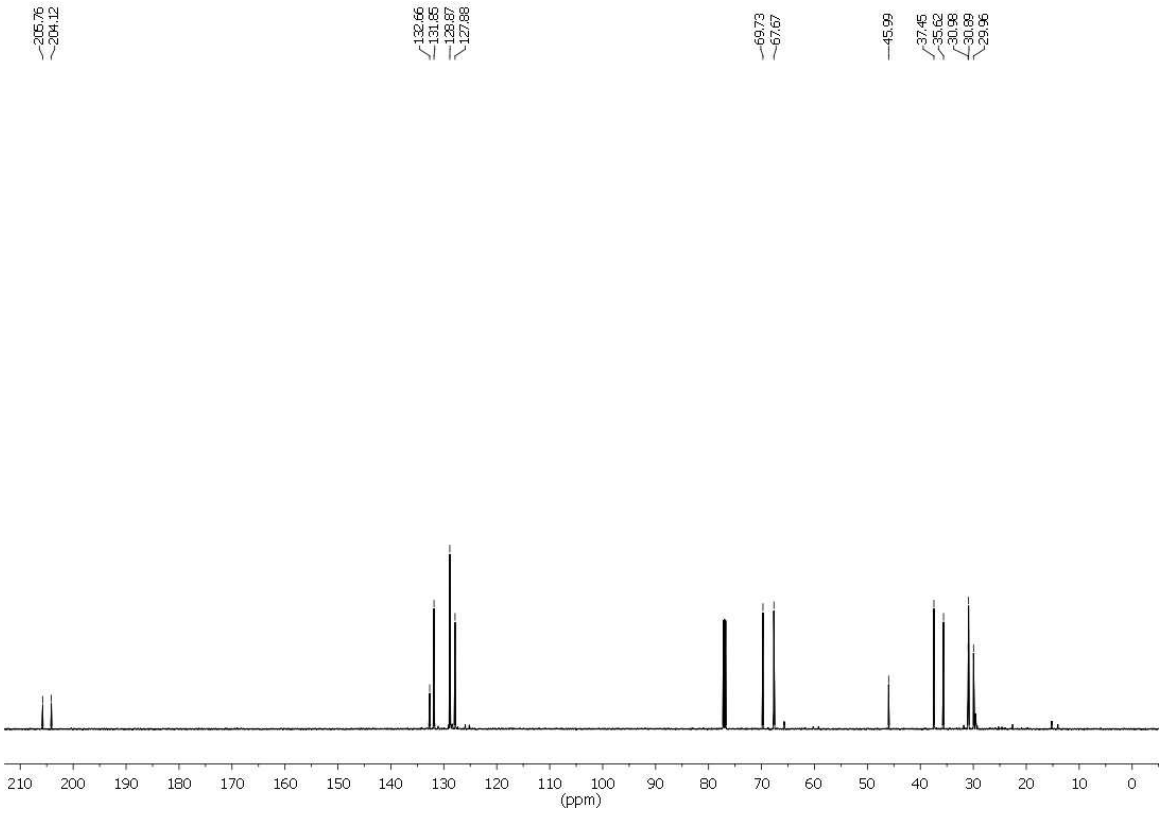

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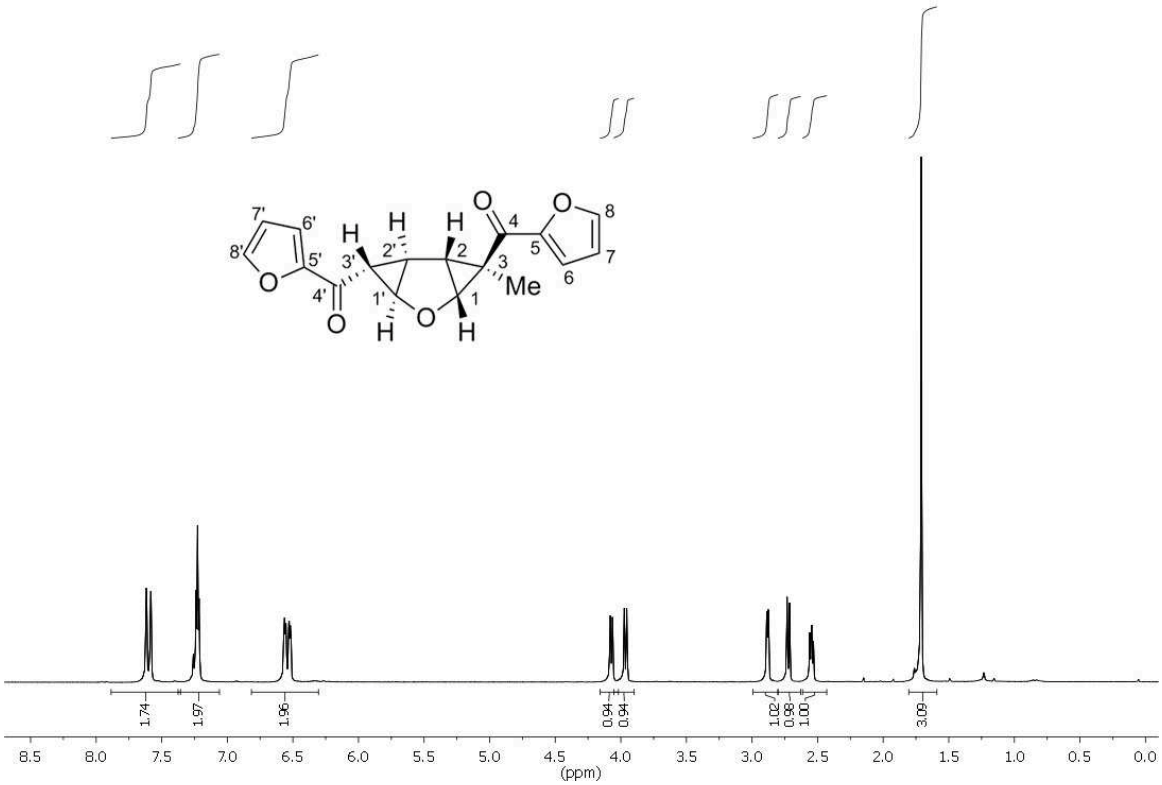

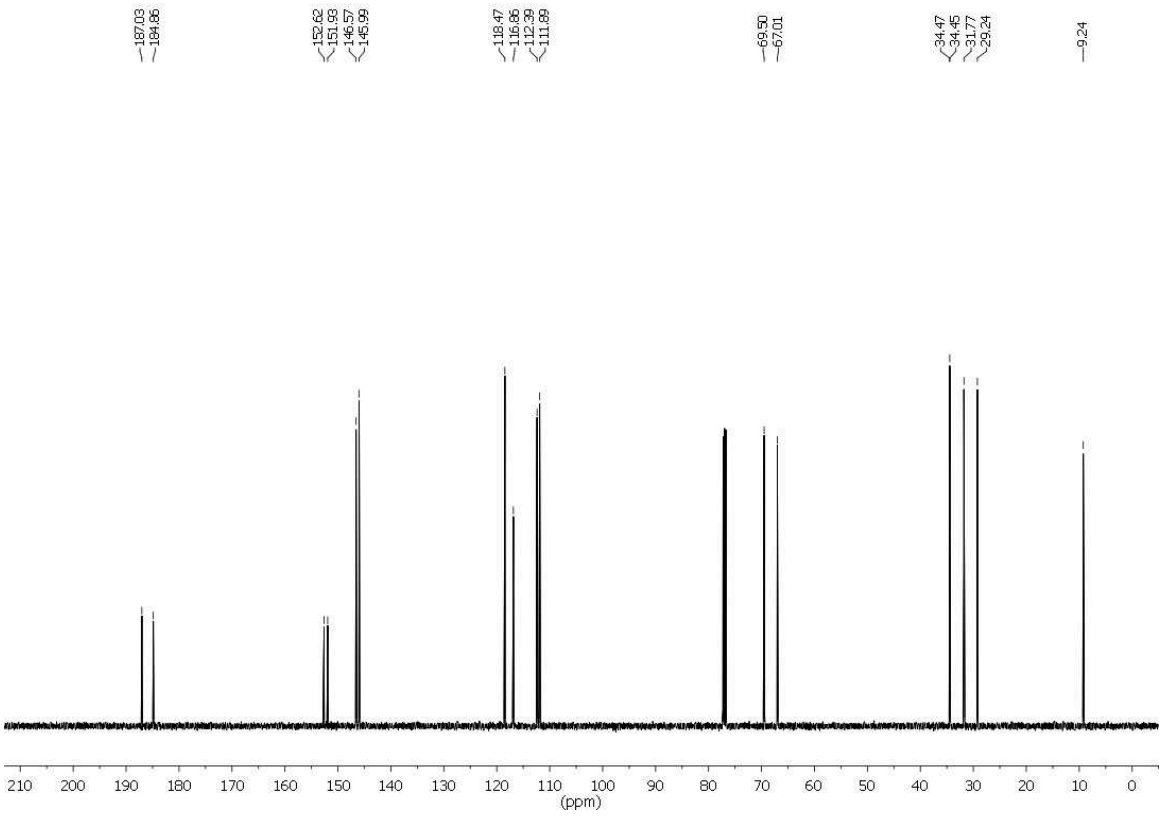

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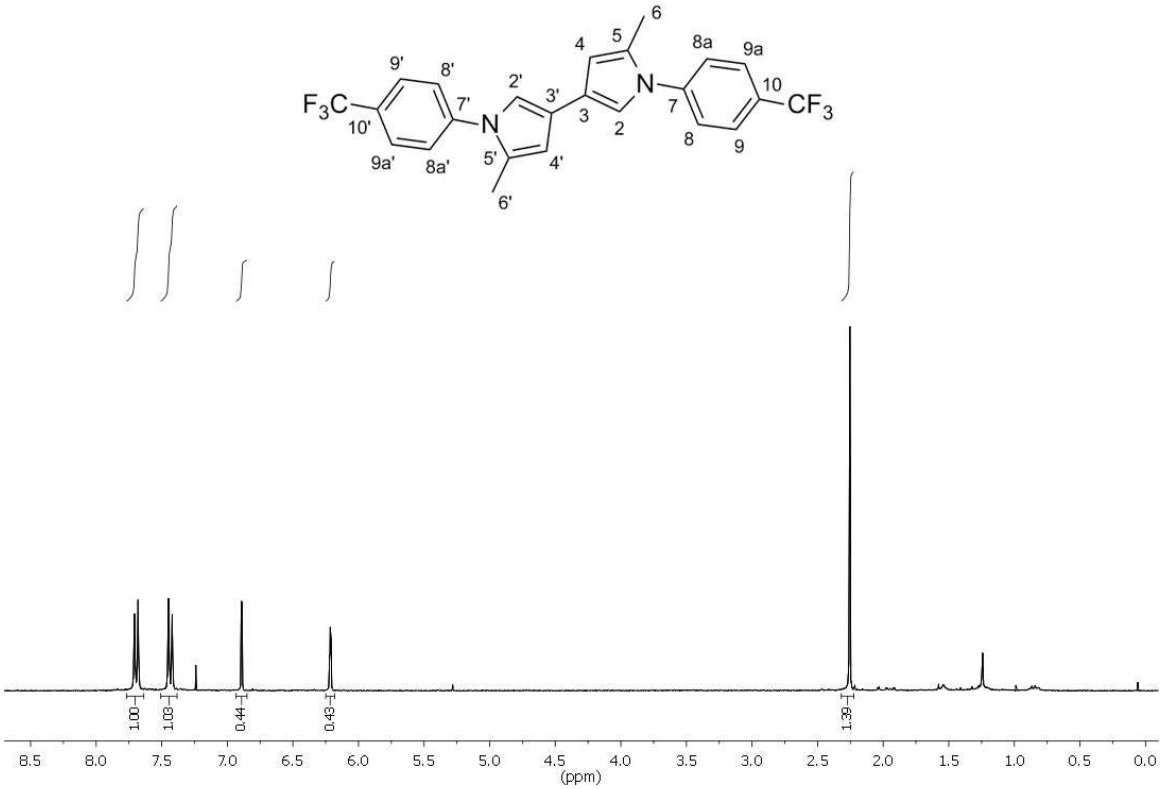

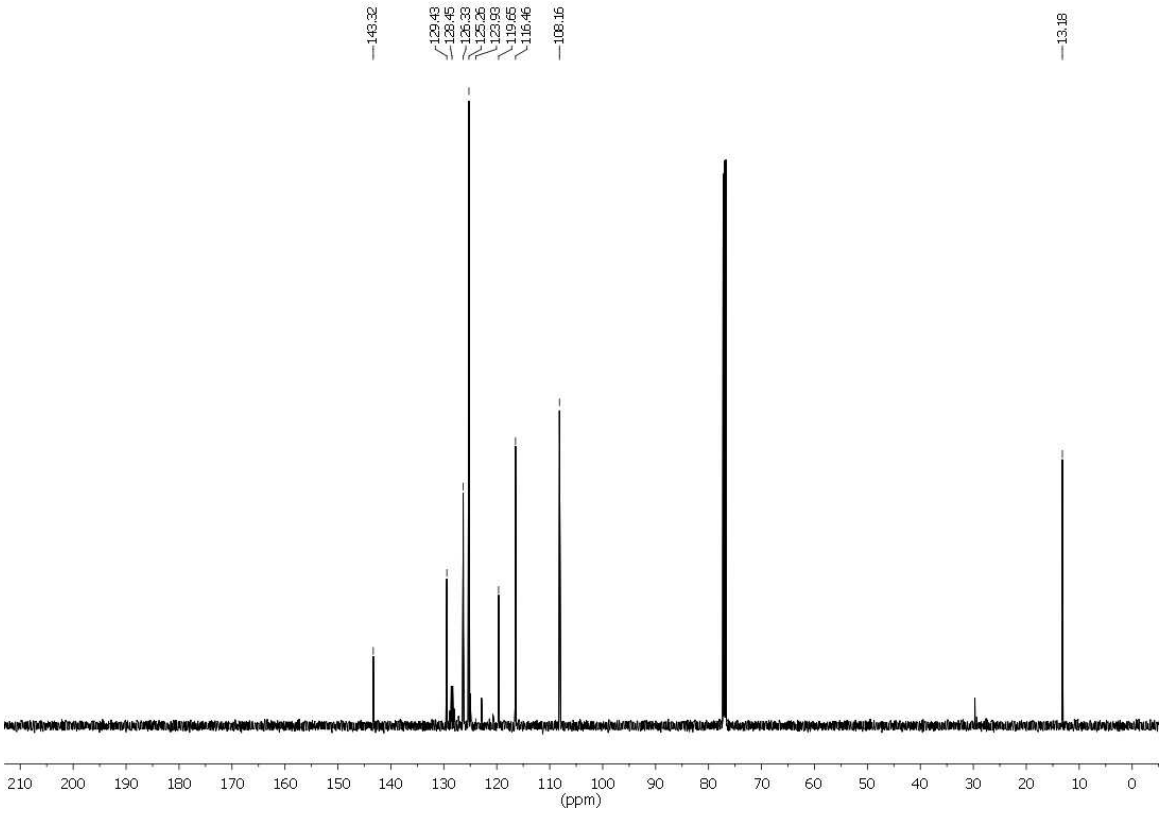

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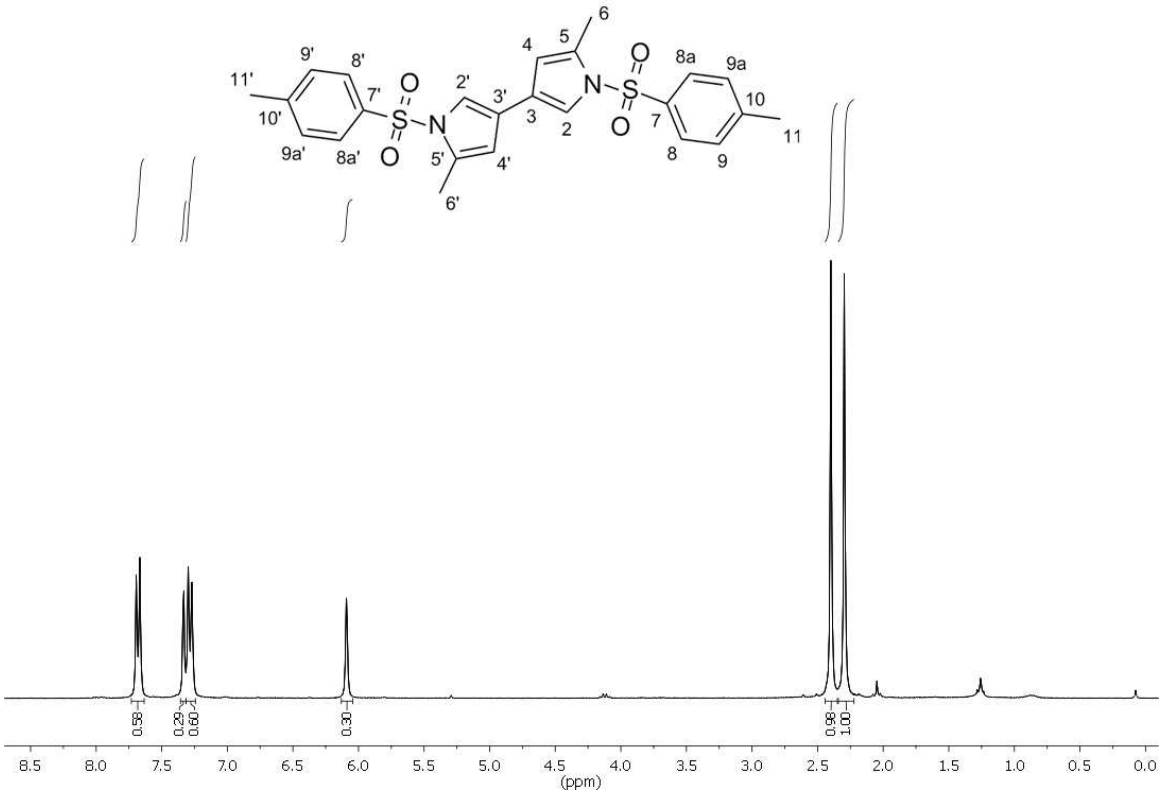

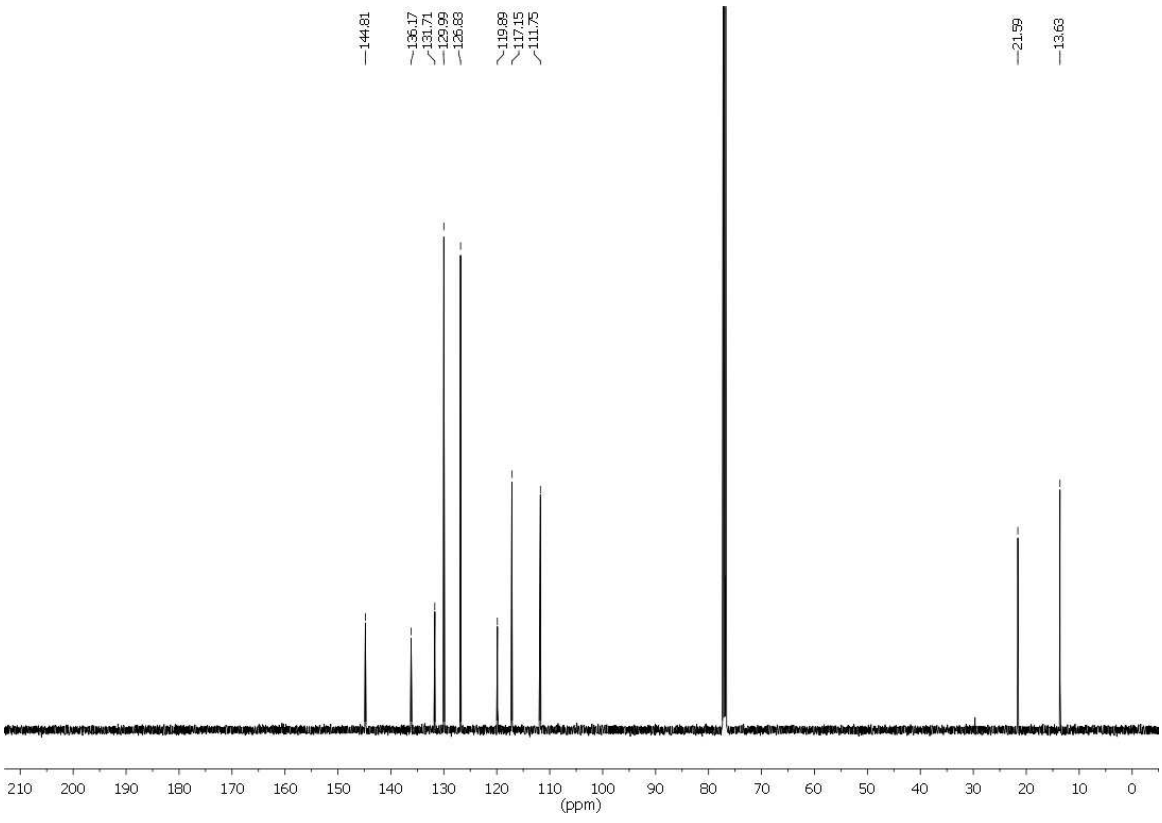

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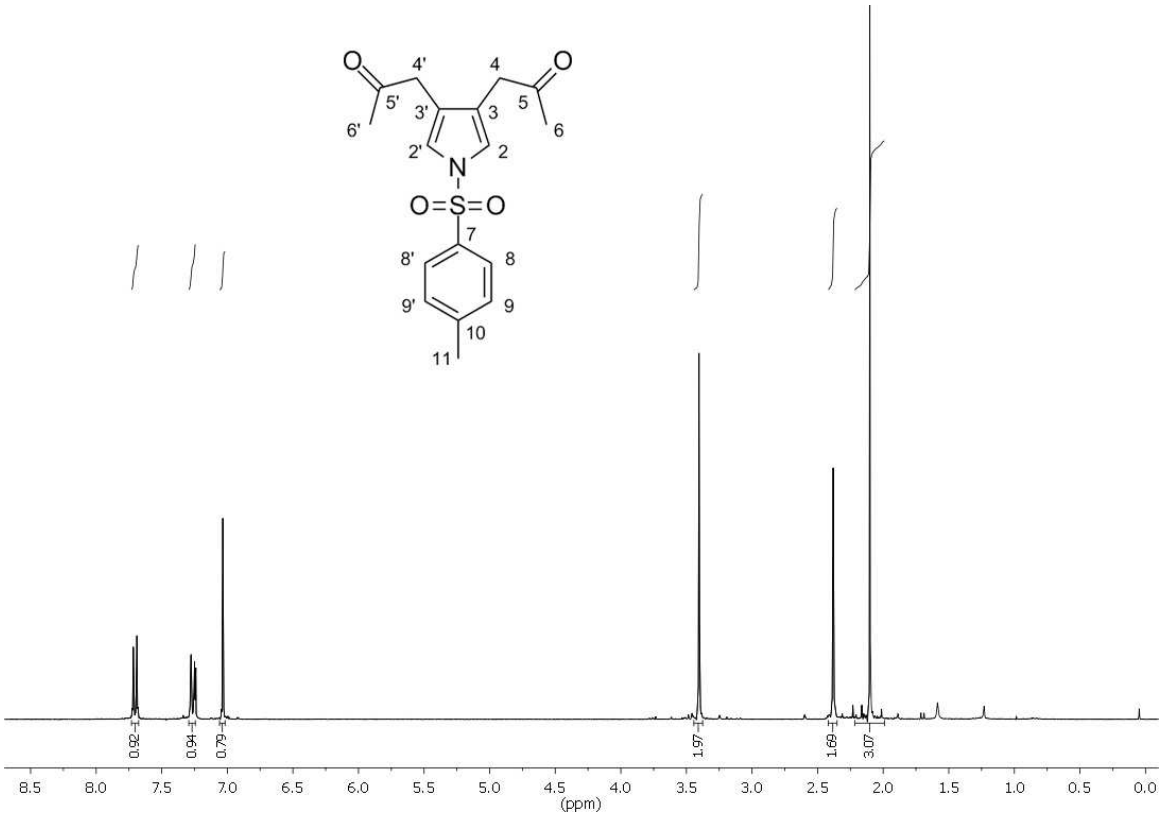
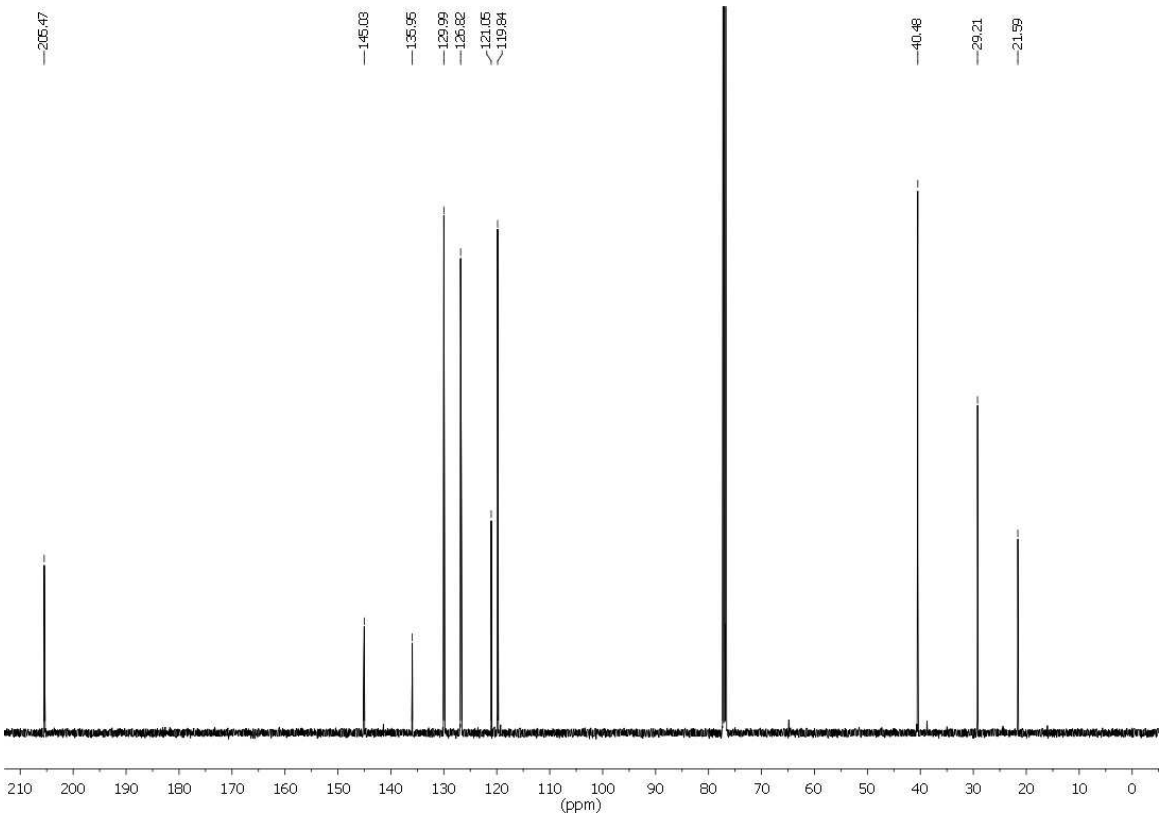
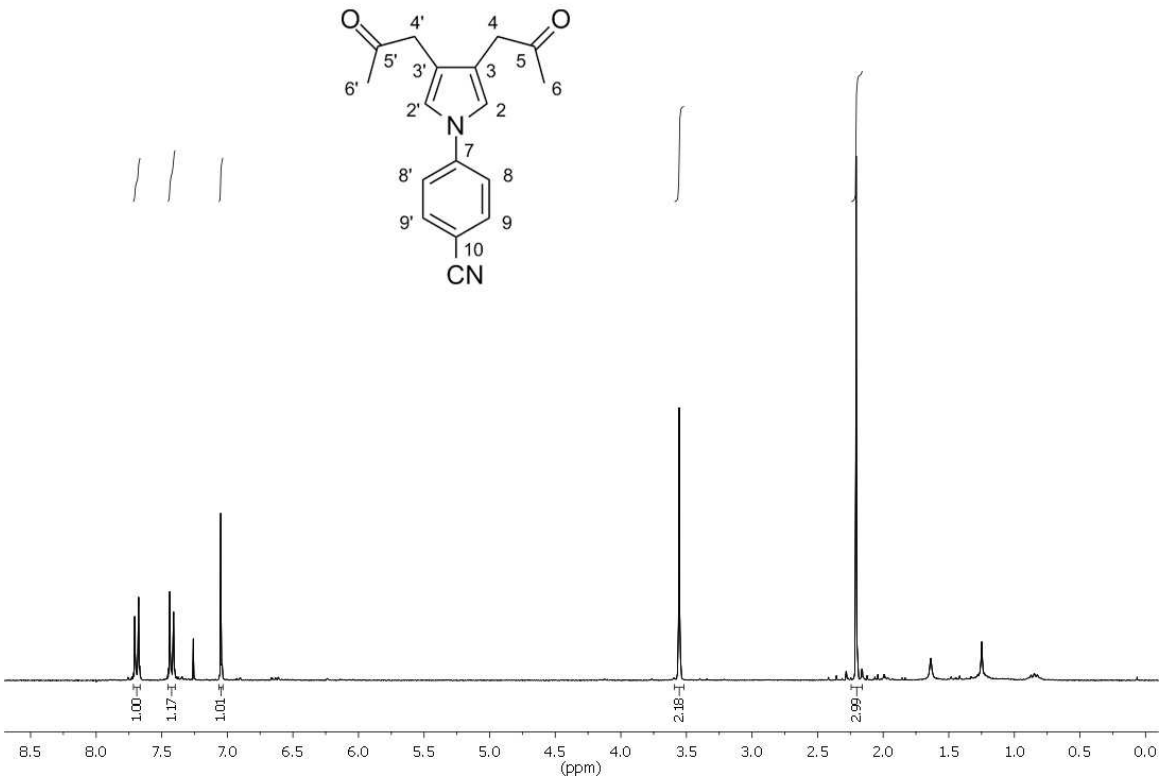
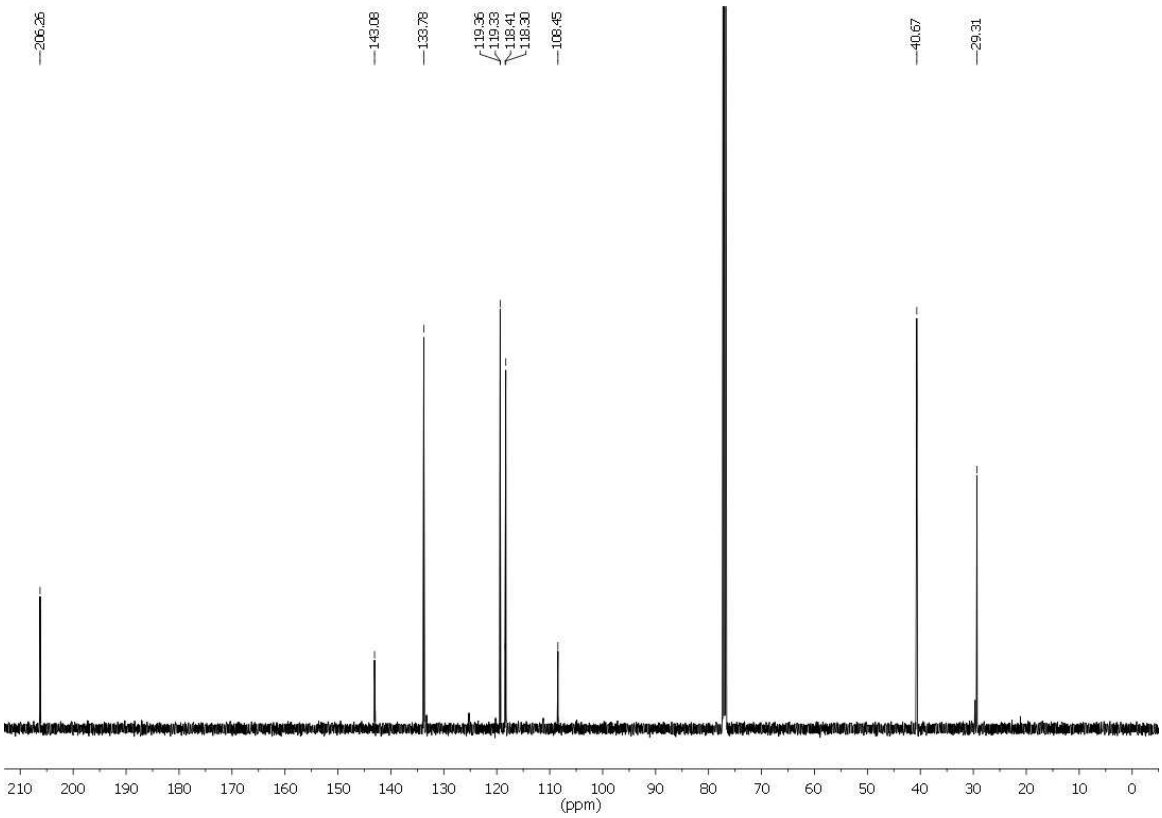


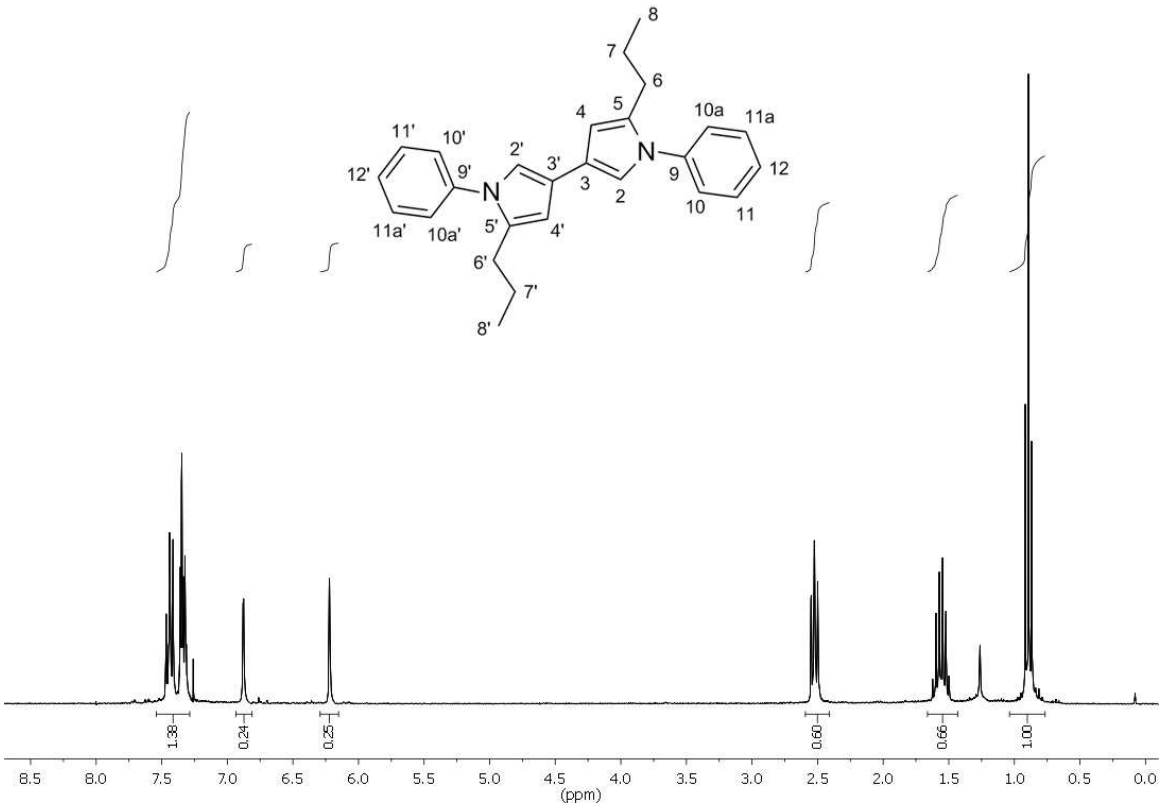


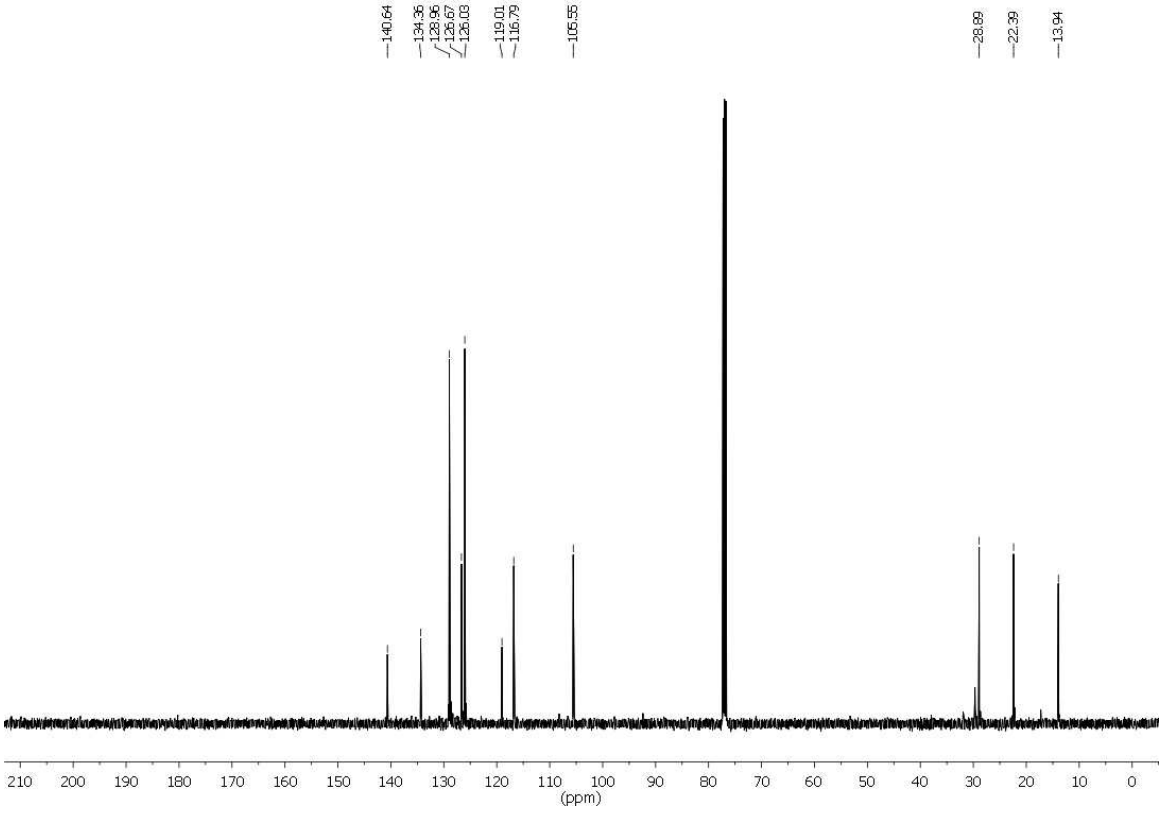
Compound 26ad (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))



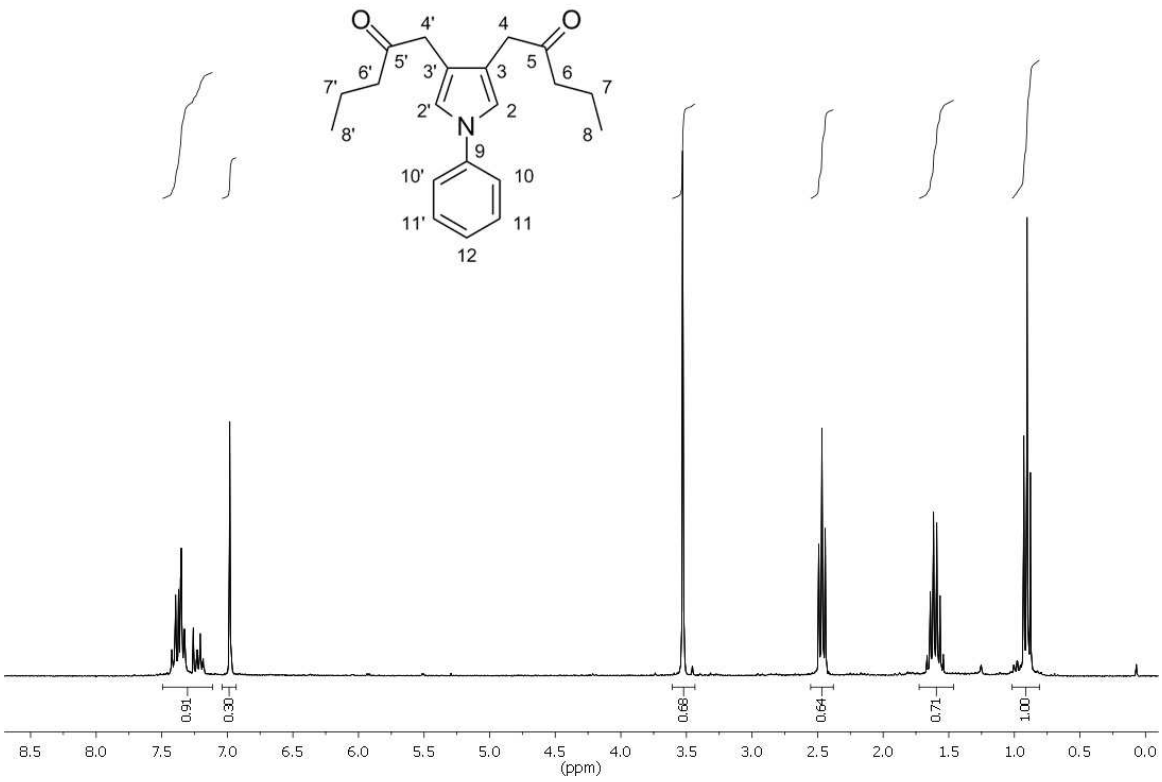


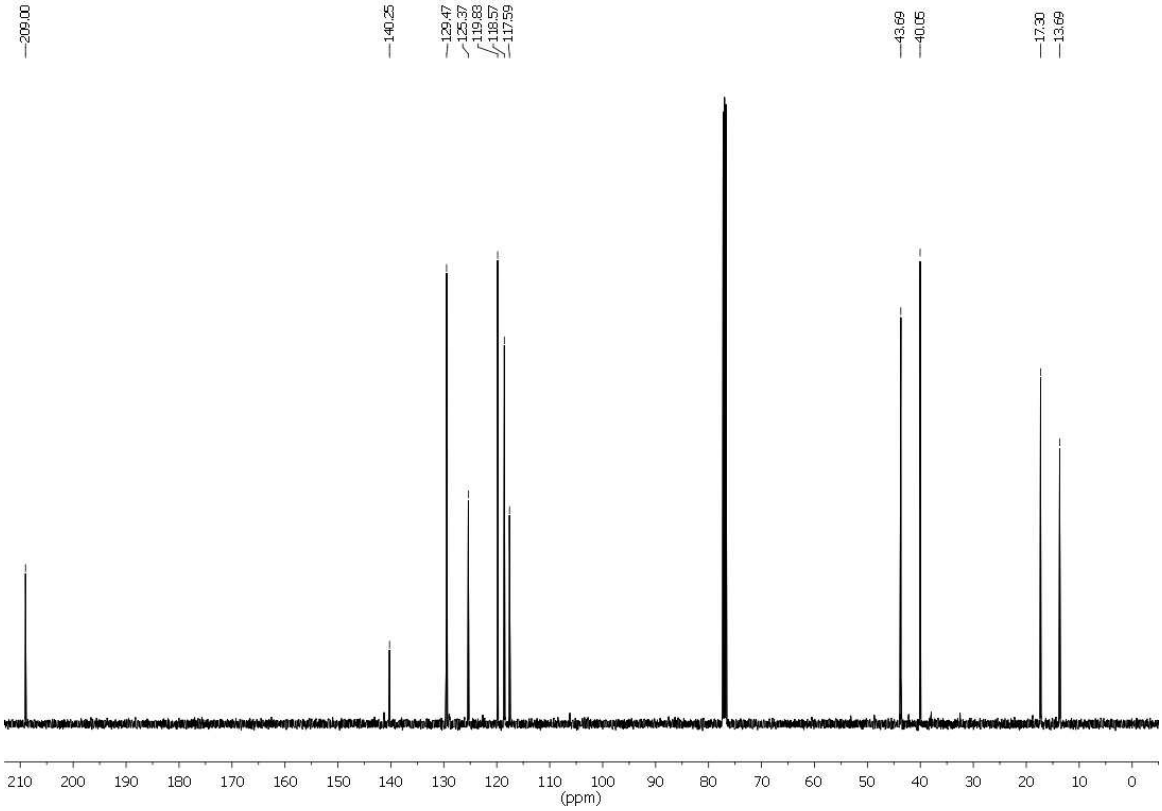
Compound 25ba (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))





Compound 26ba (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))





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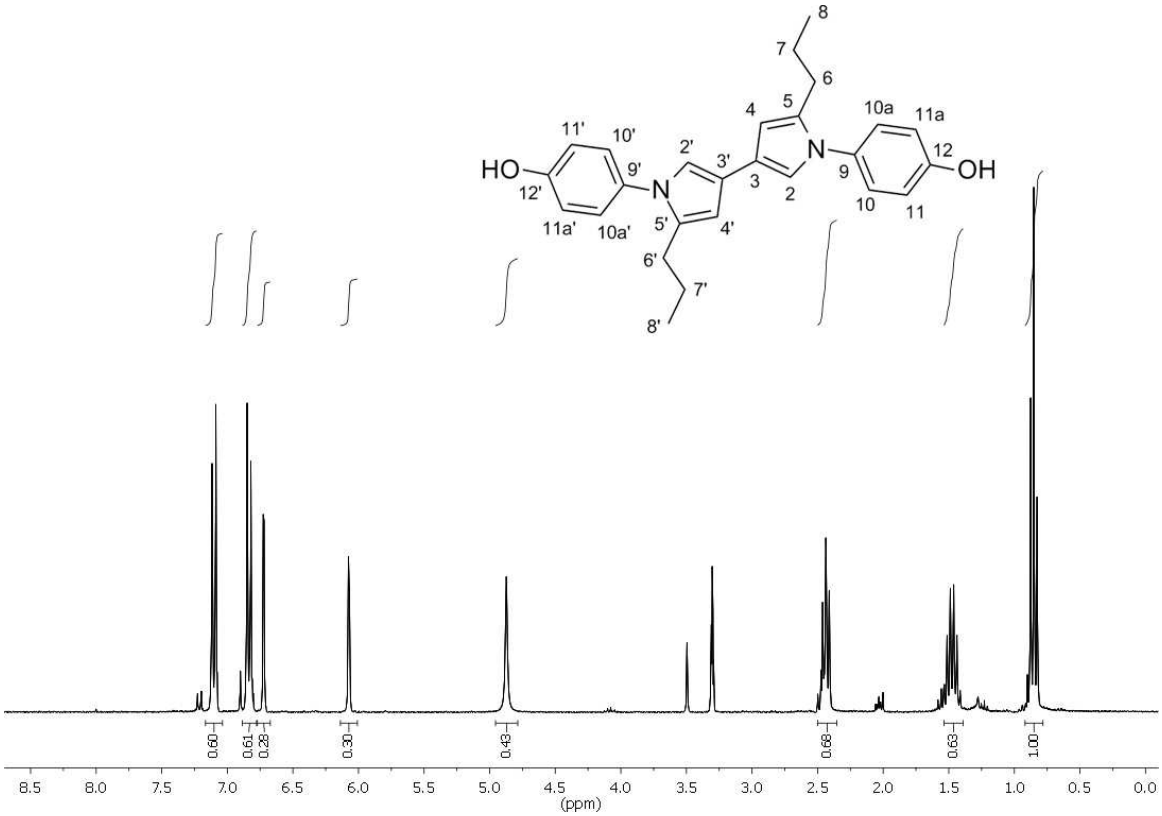

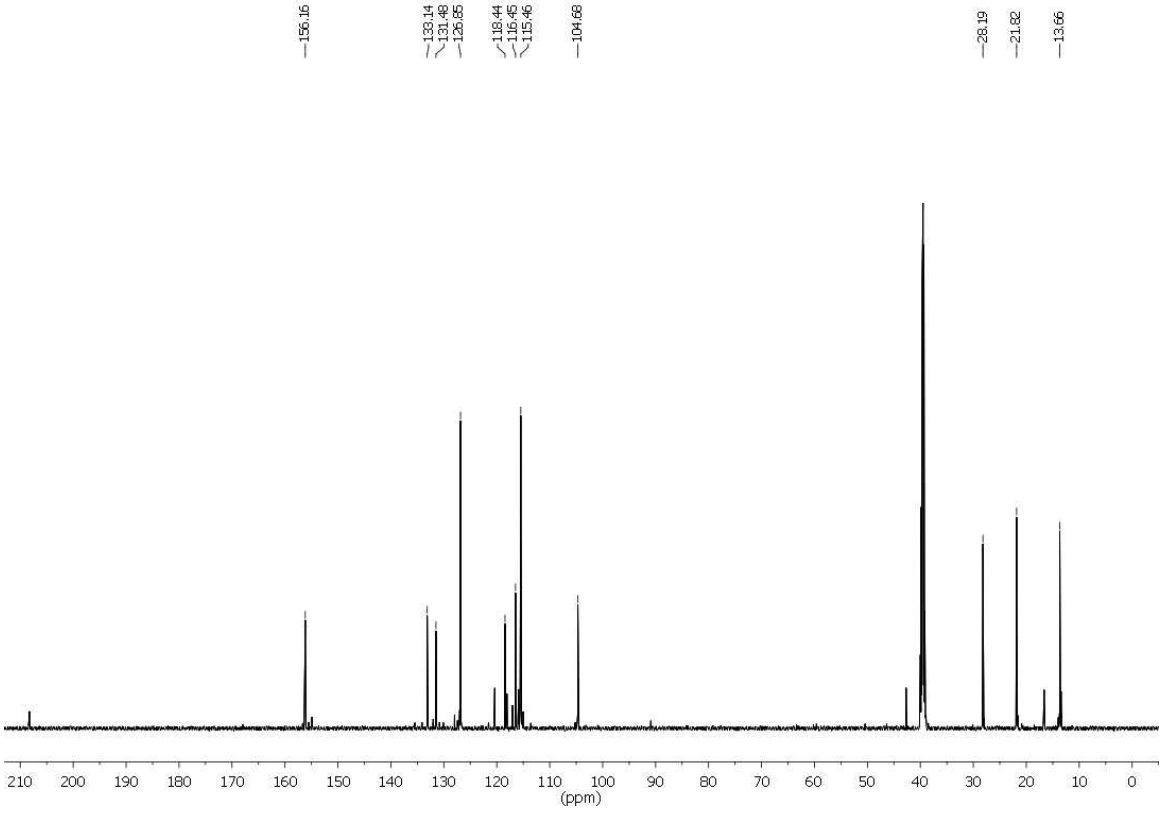

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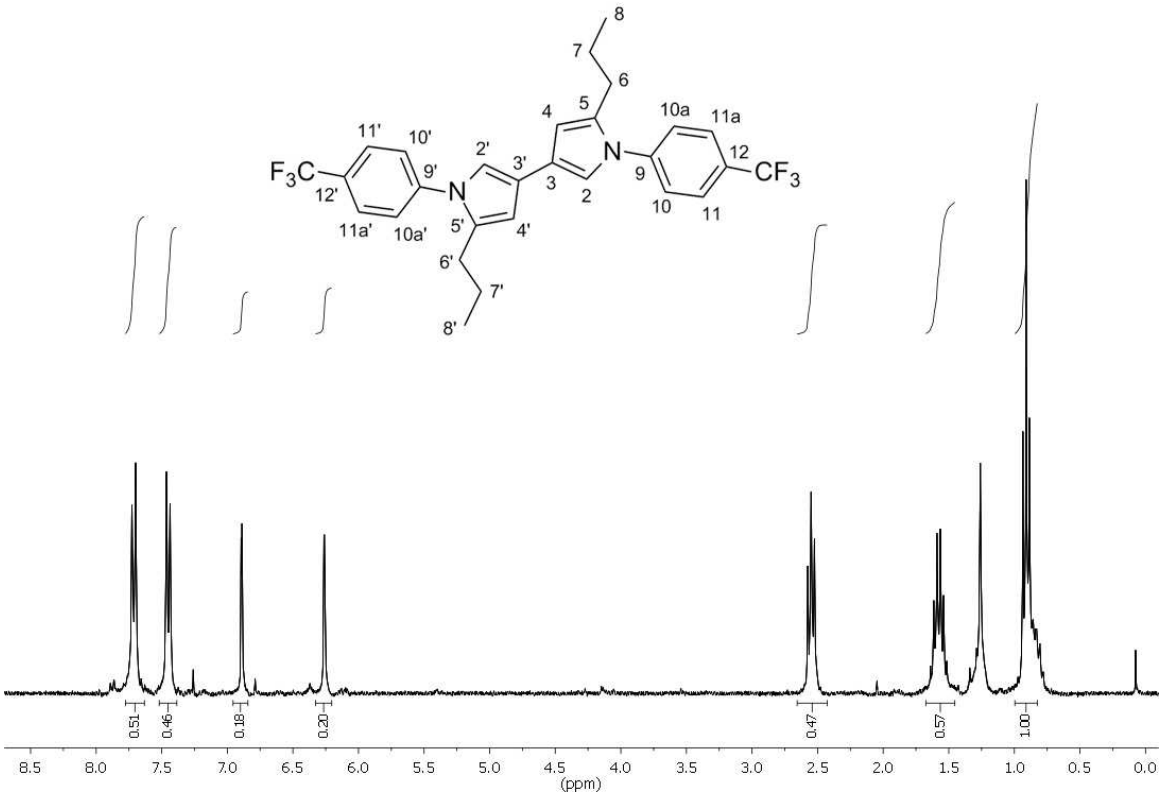

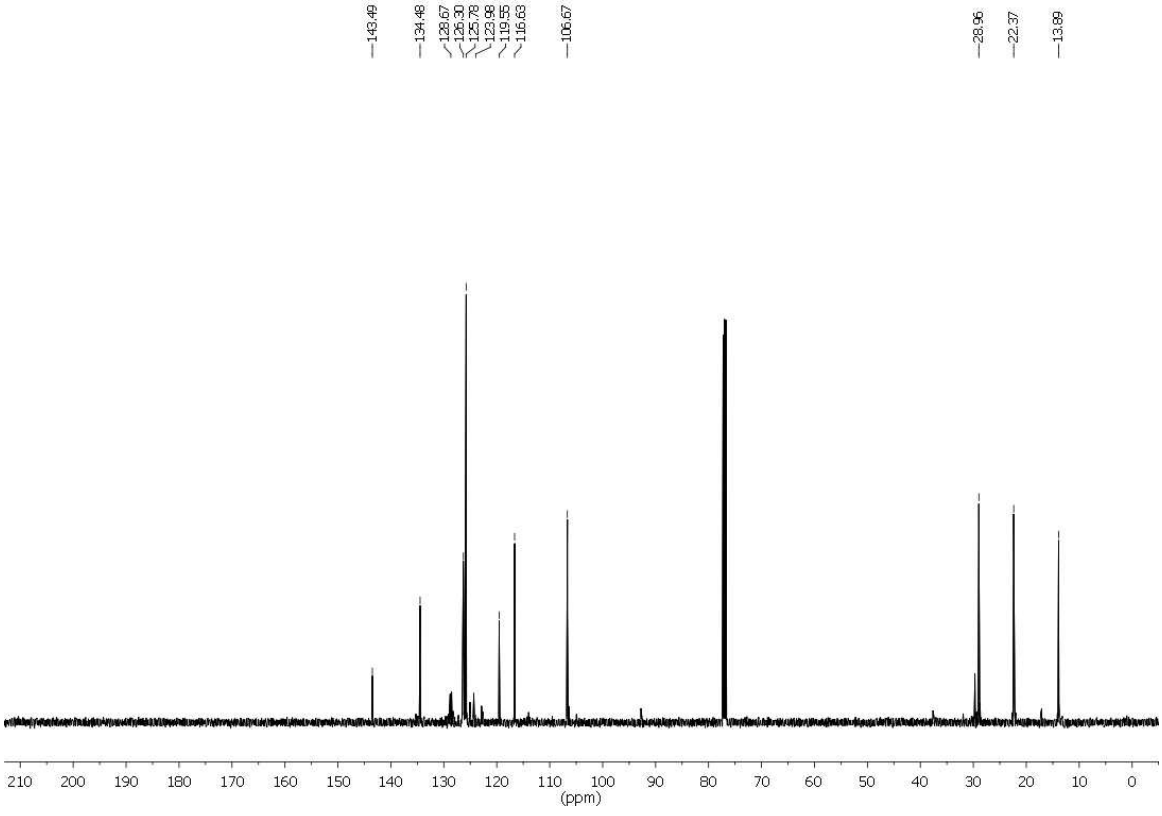

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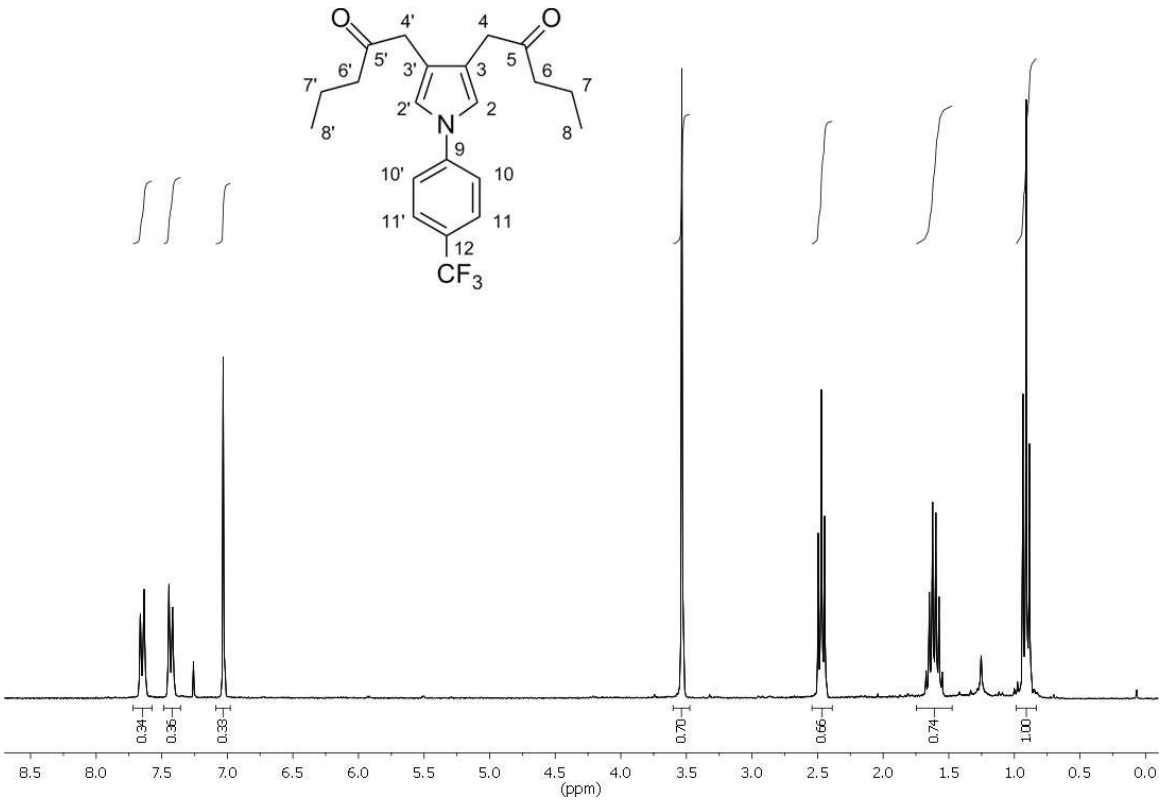

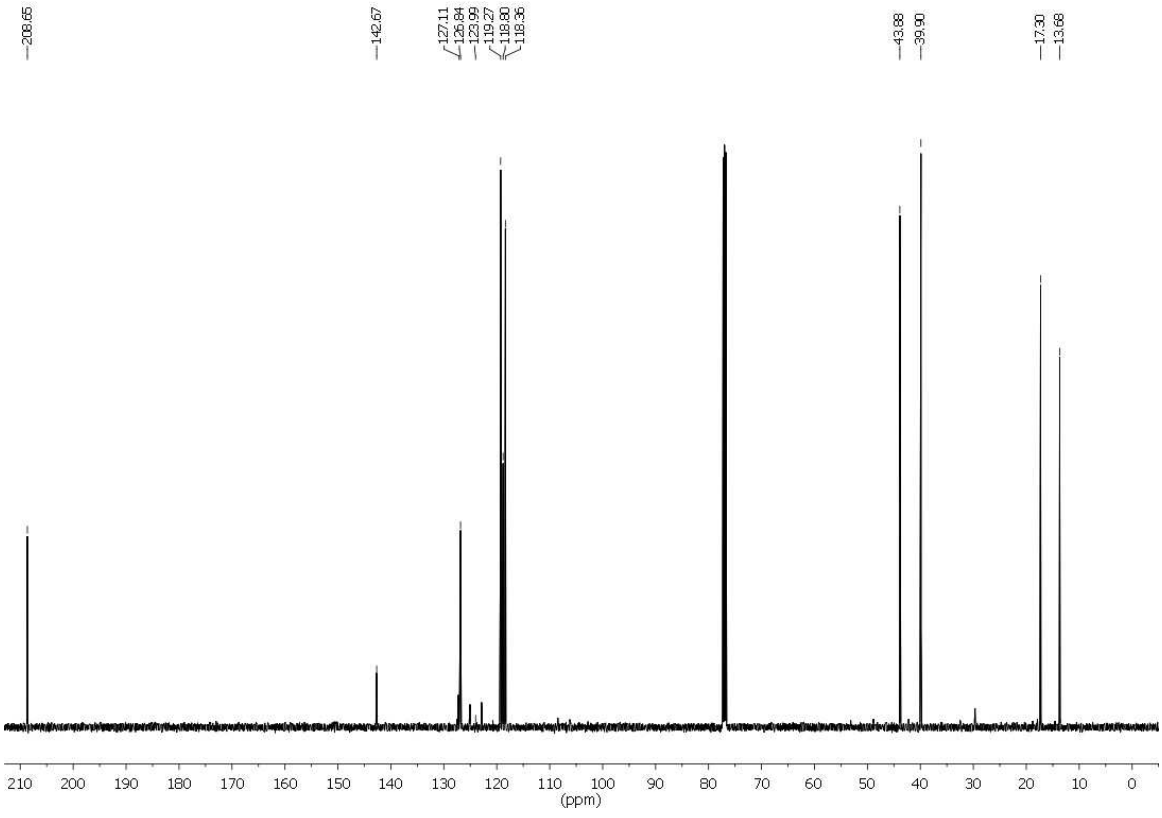

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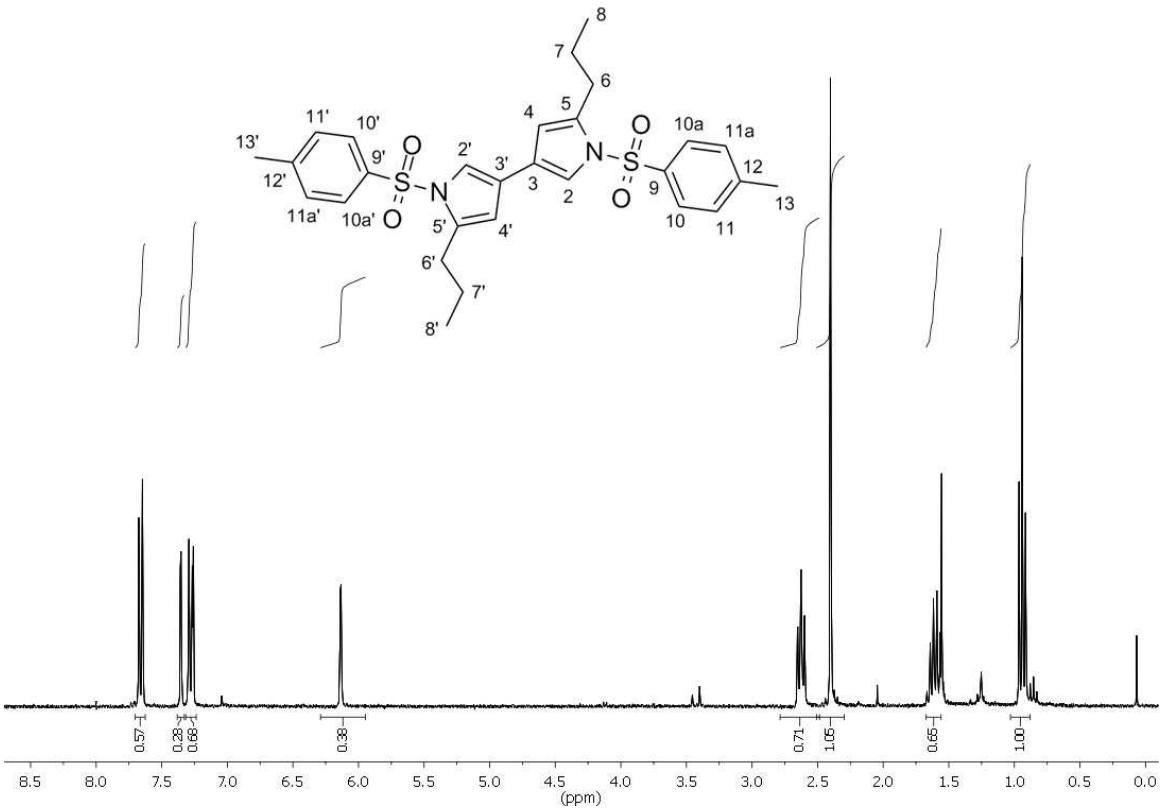

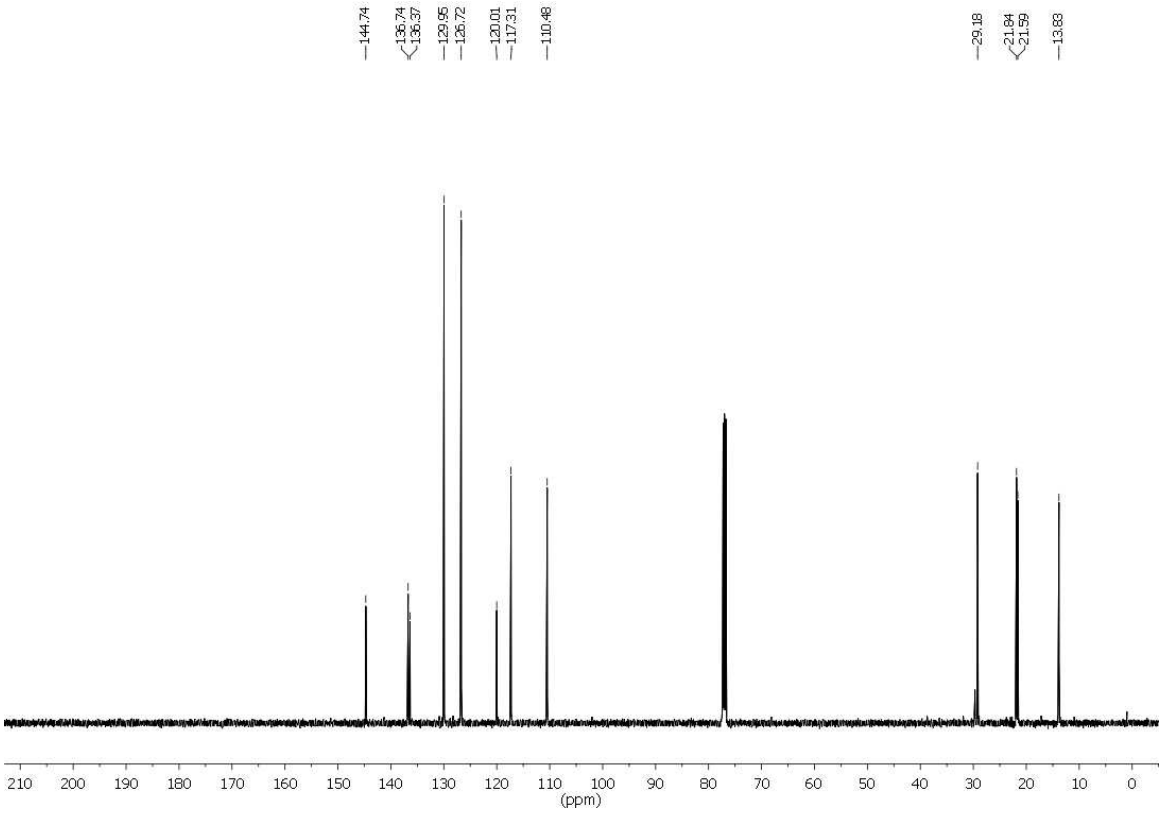

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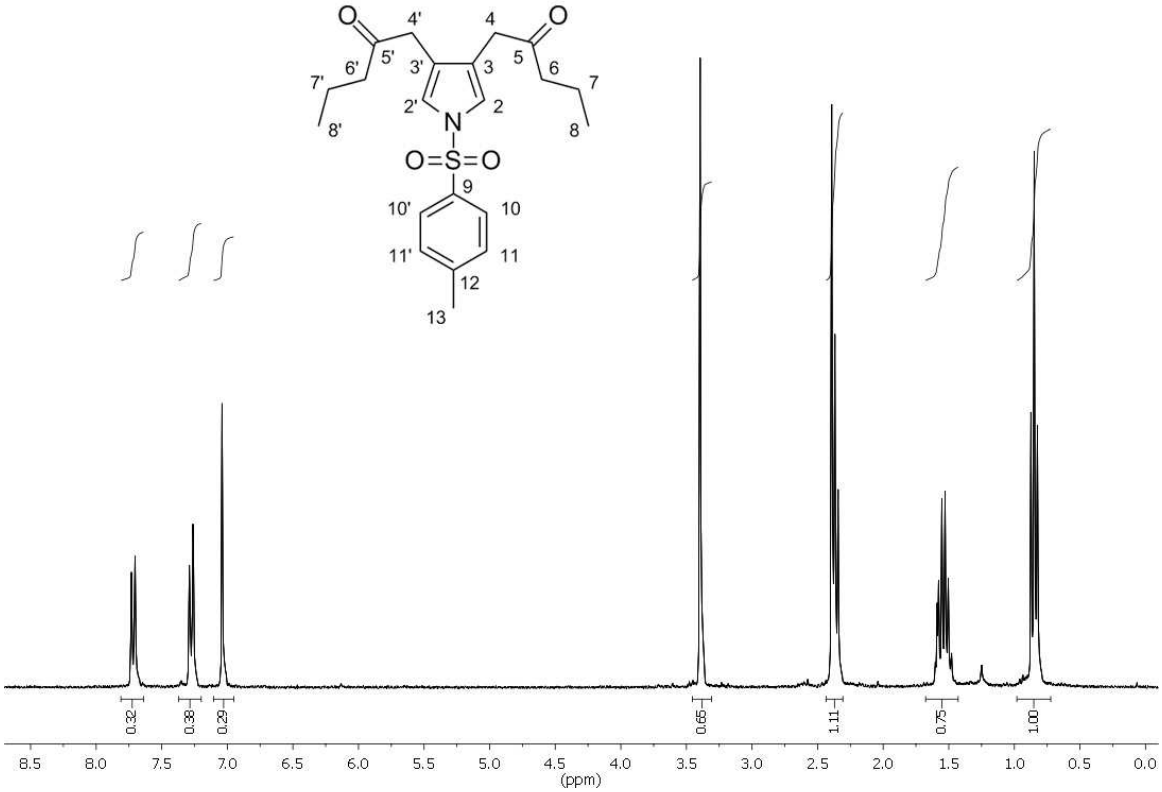

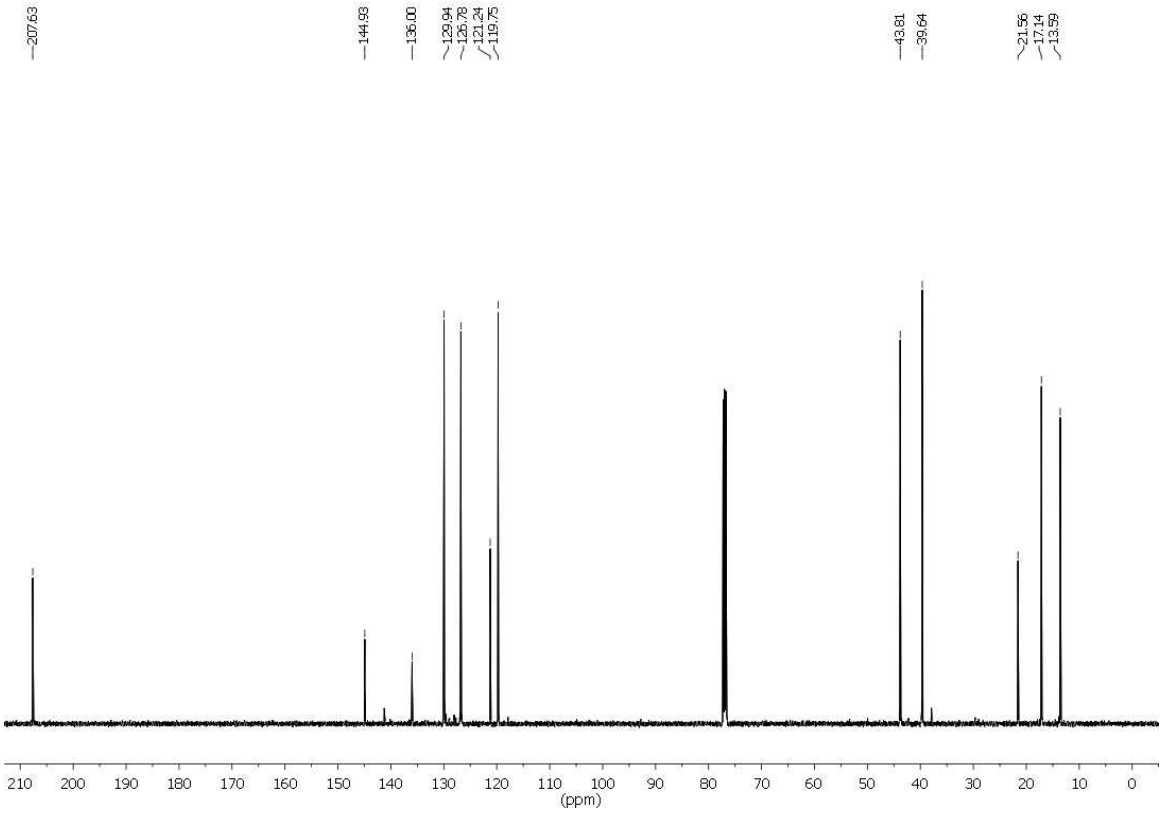

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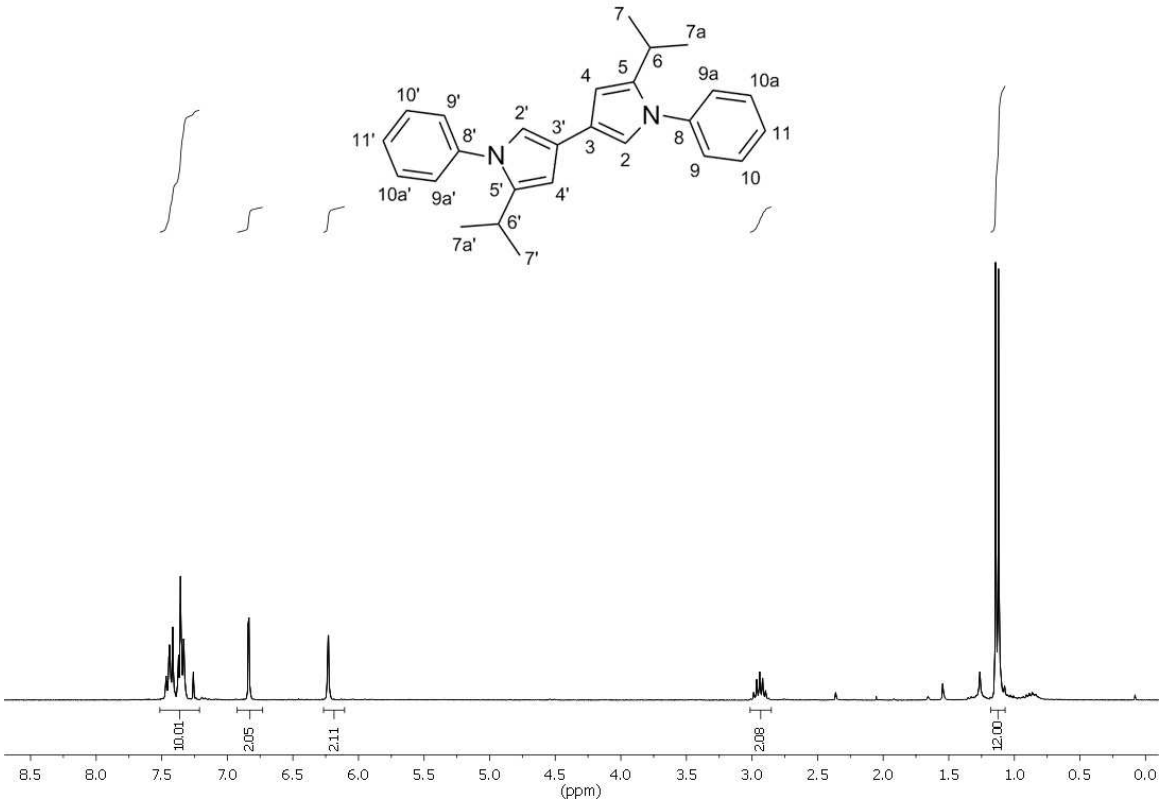

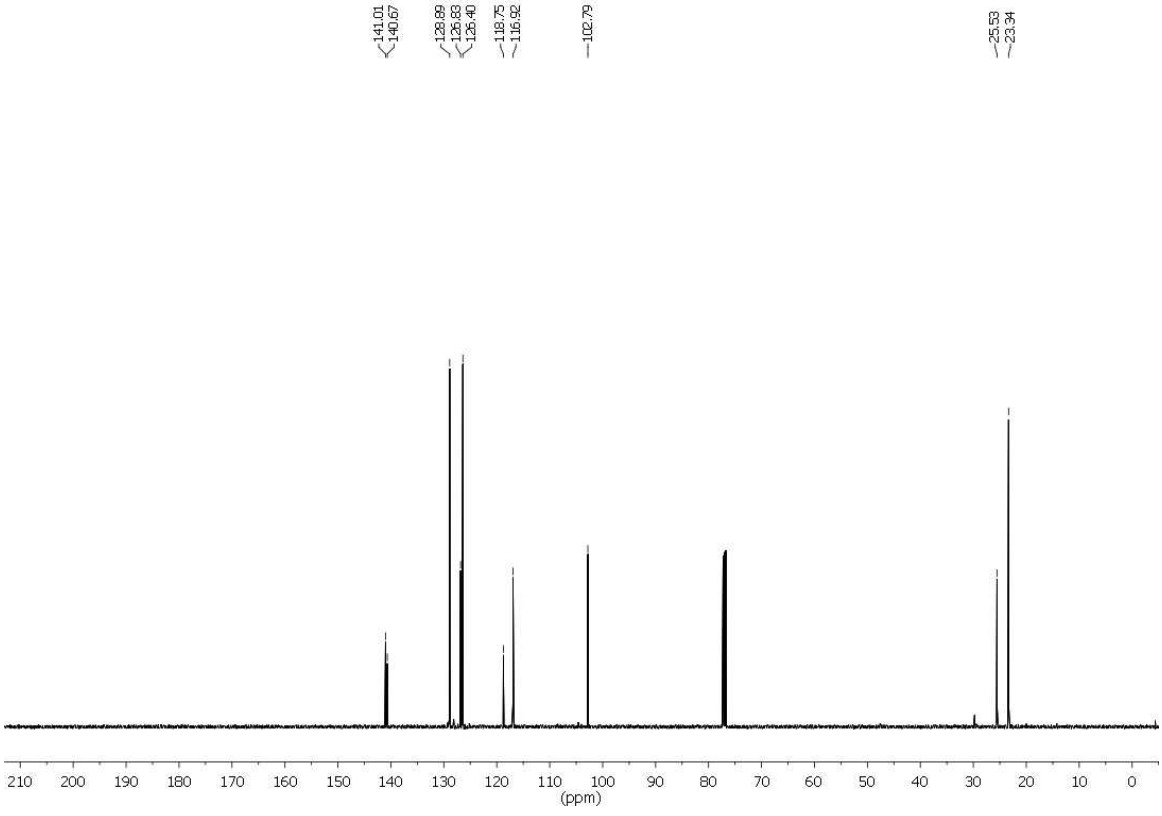

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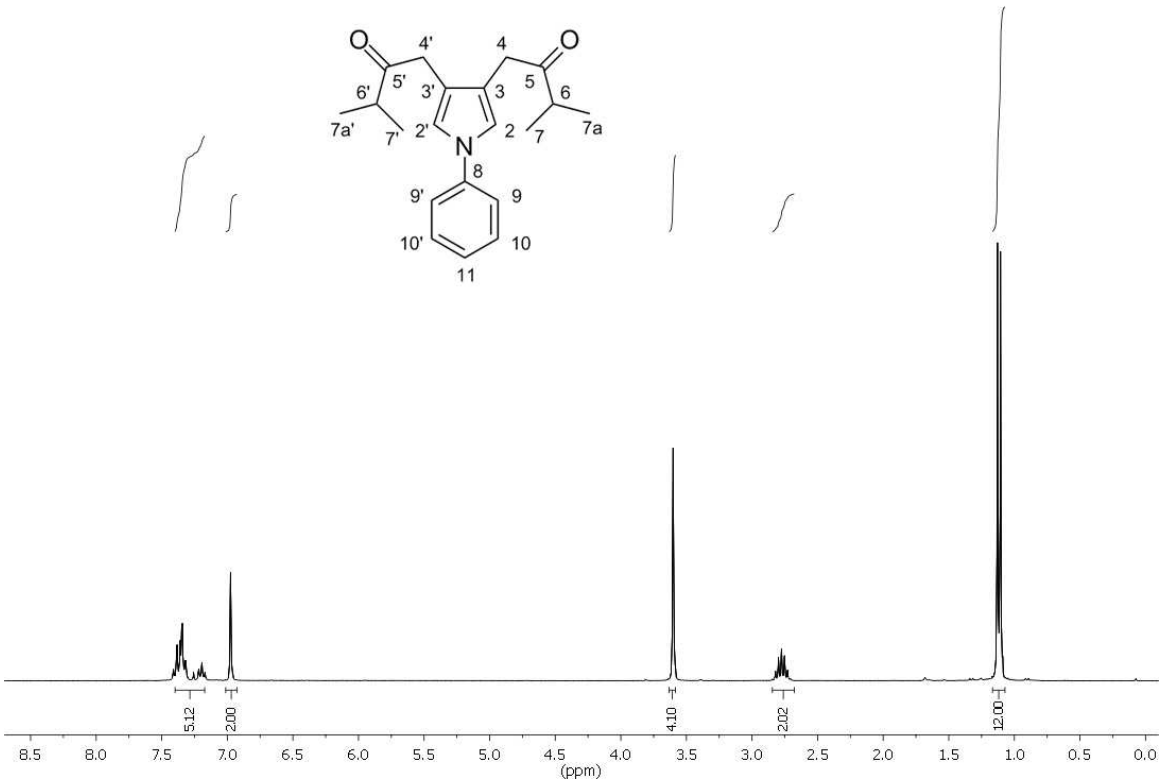
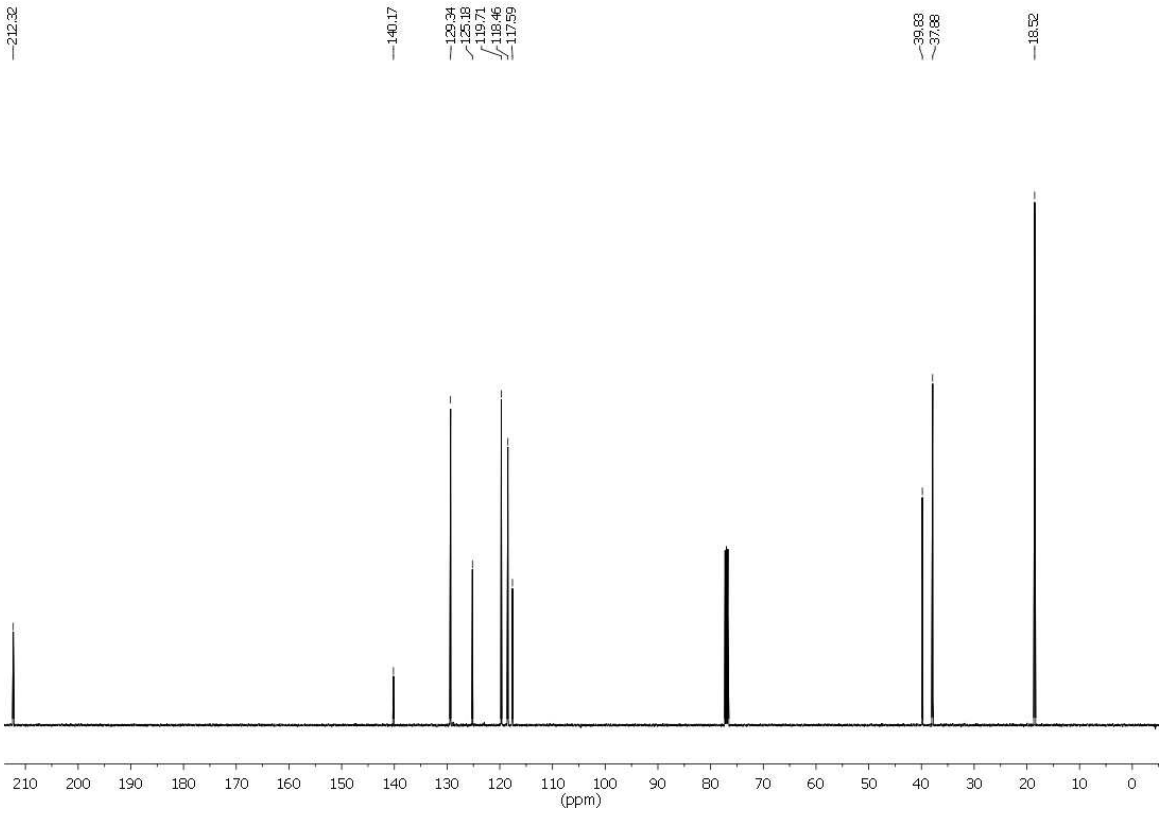
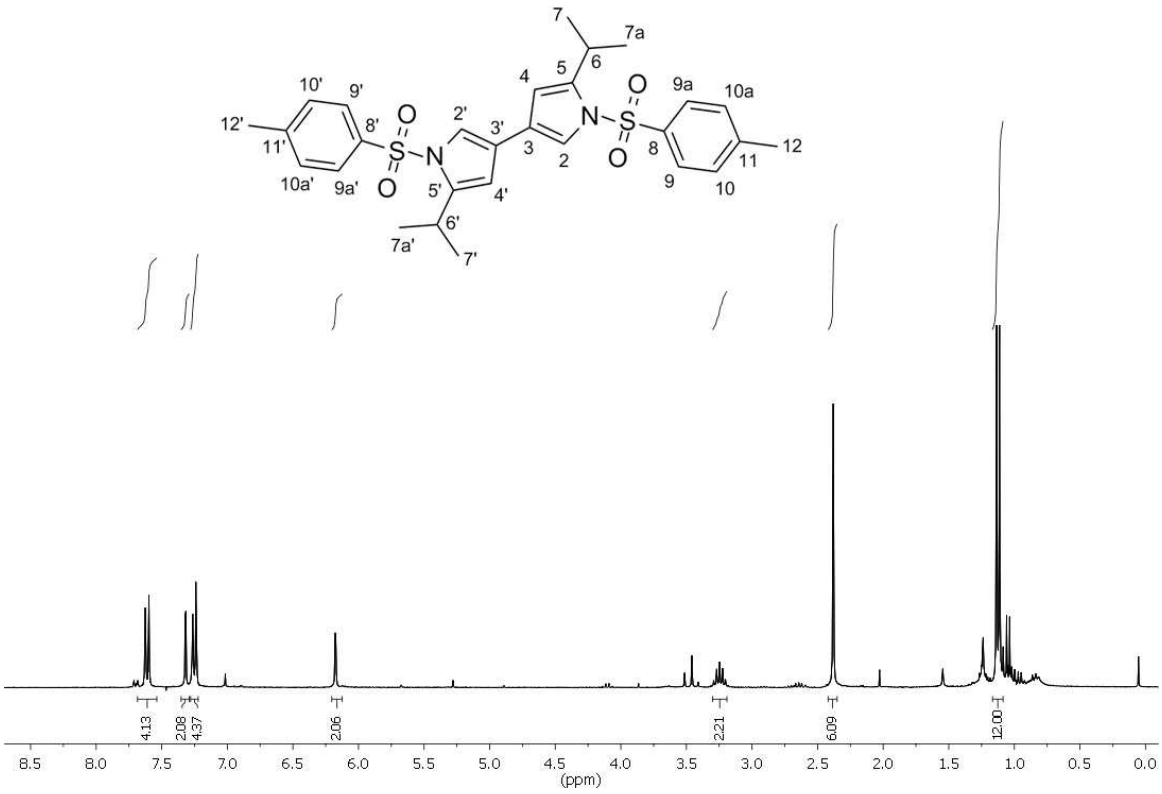
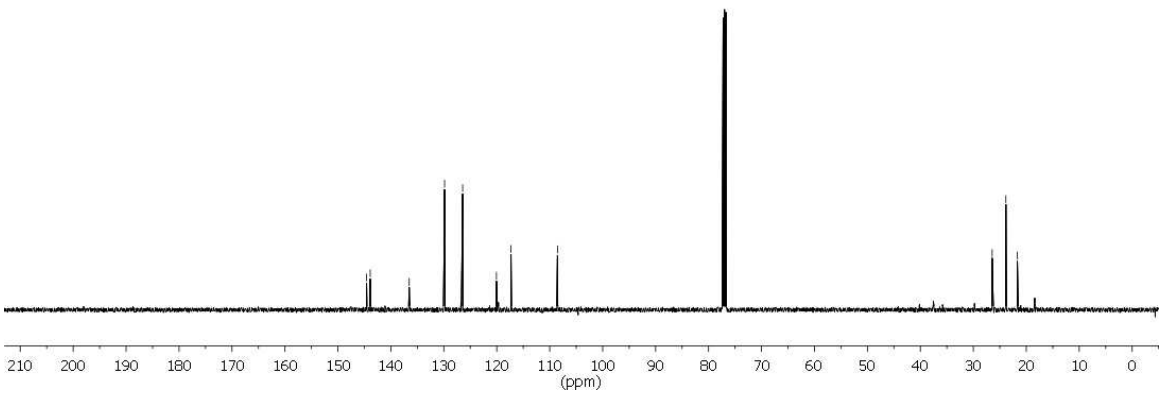




Compound 25cb (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))





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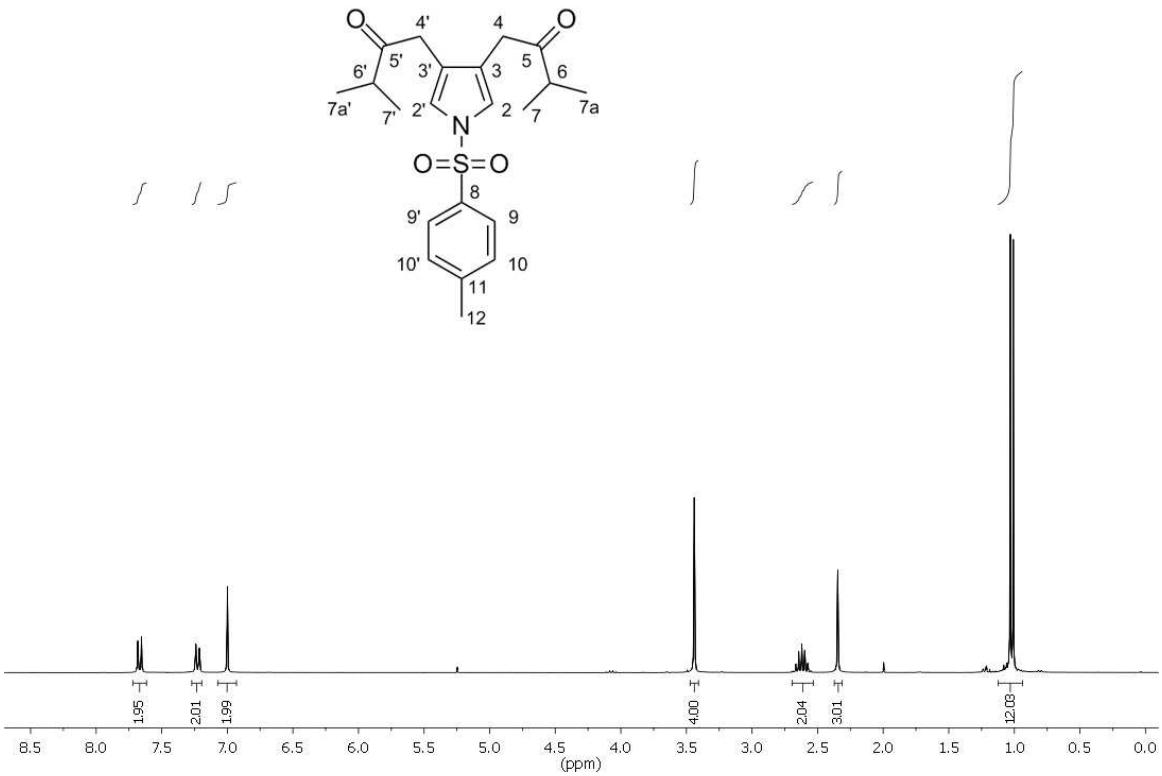
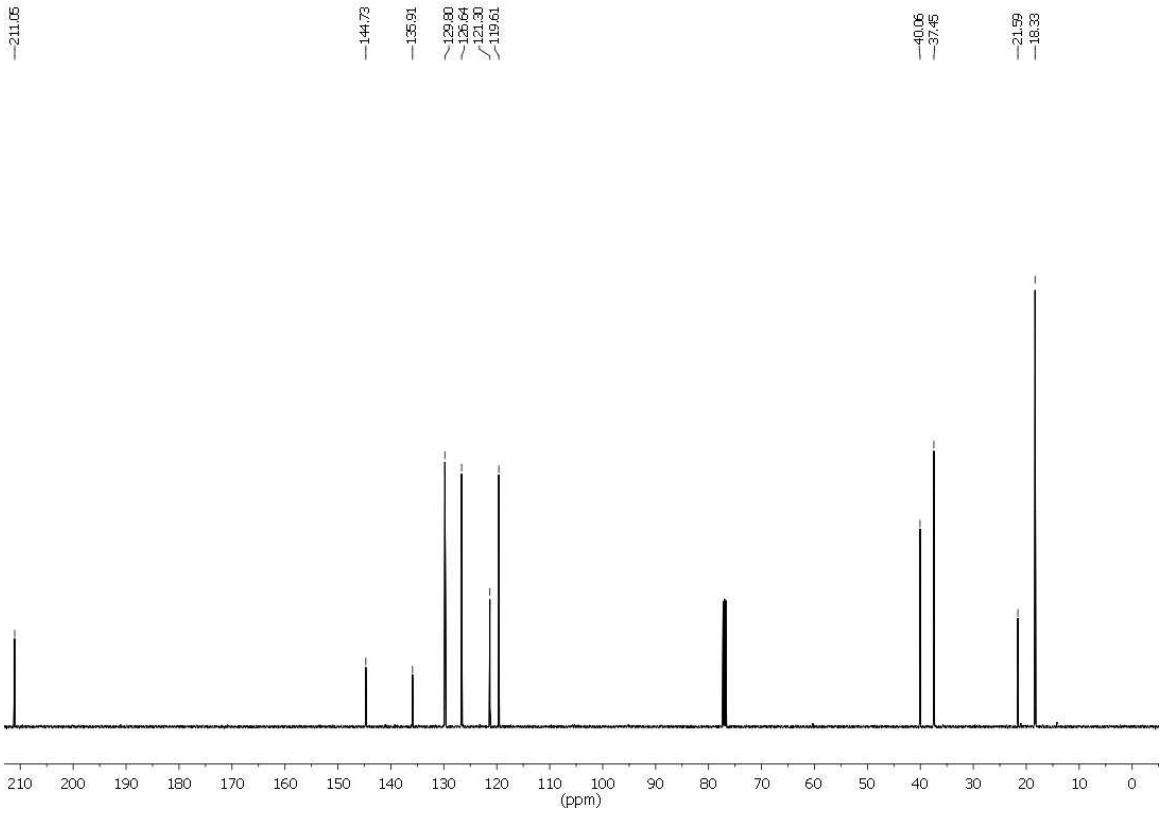
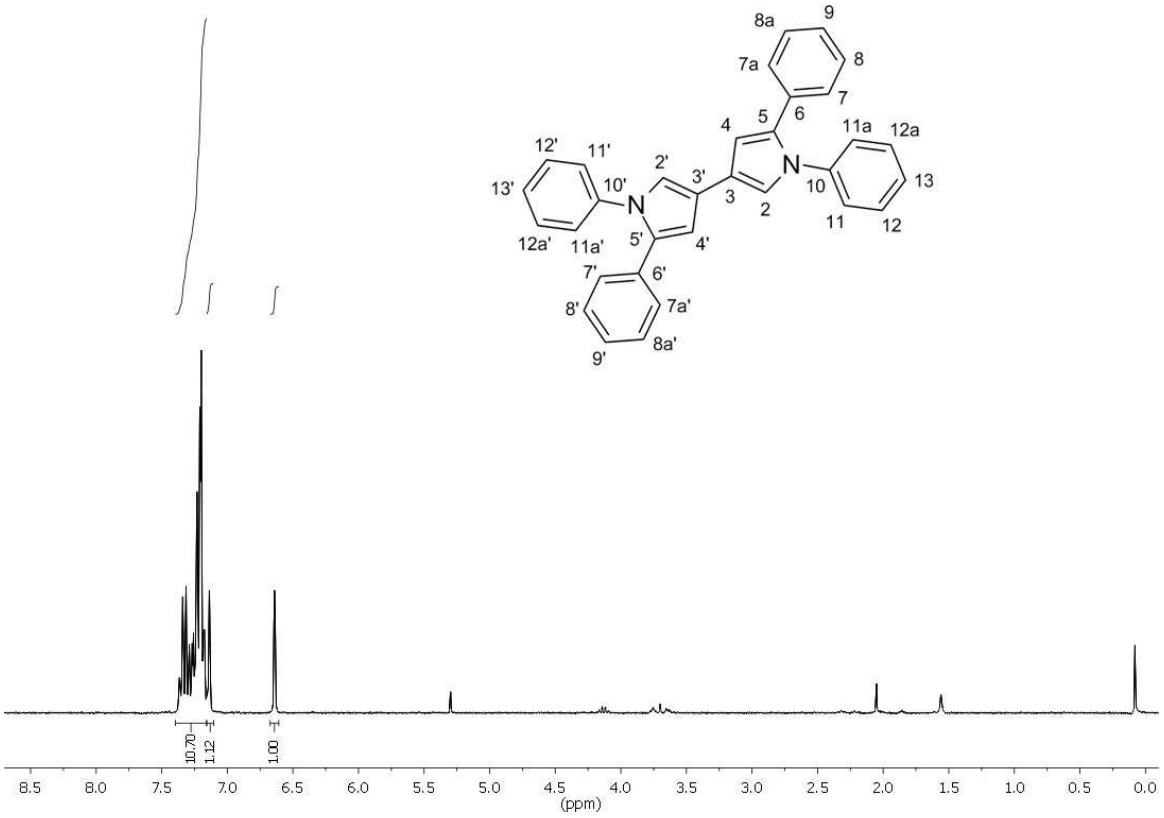
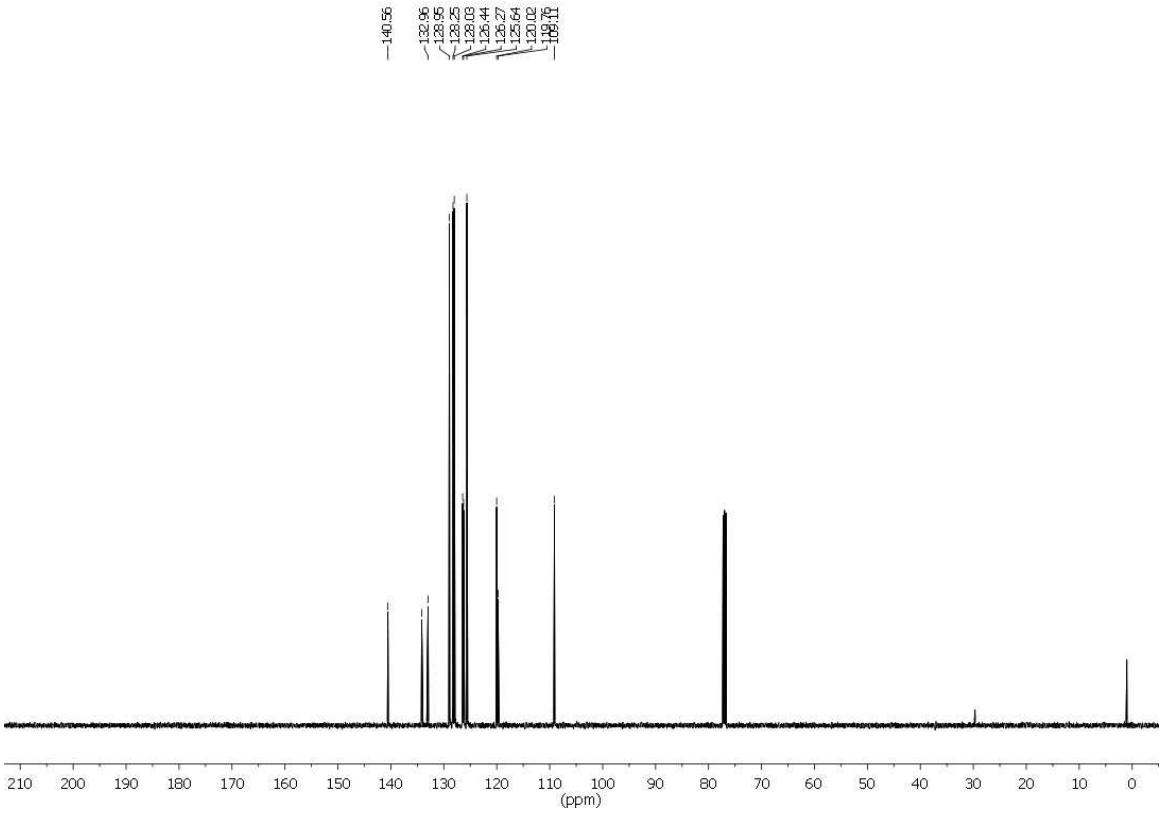


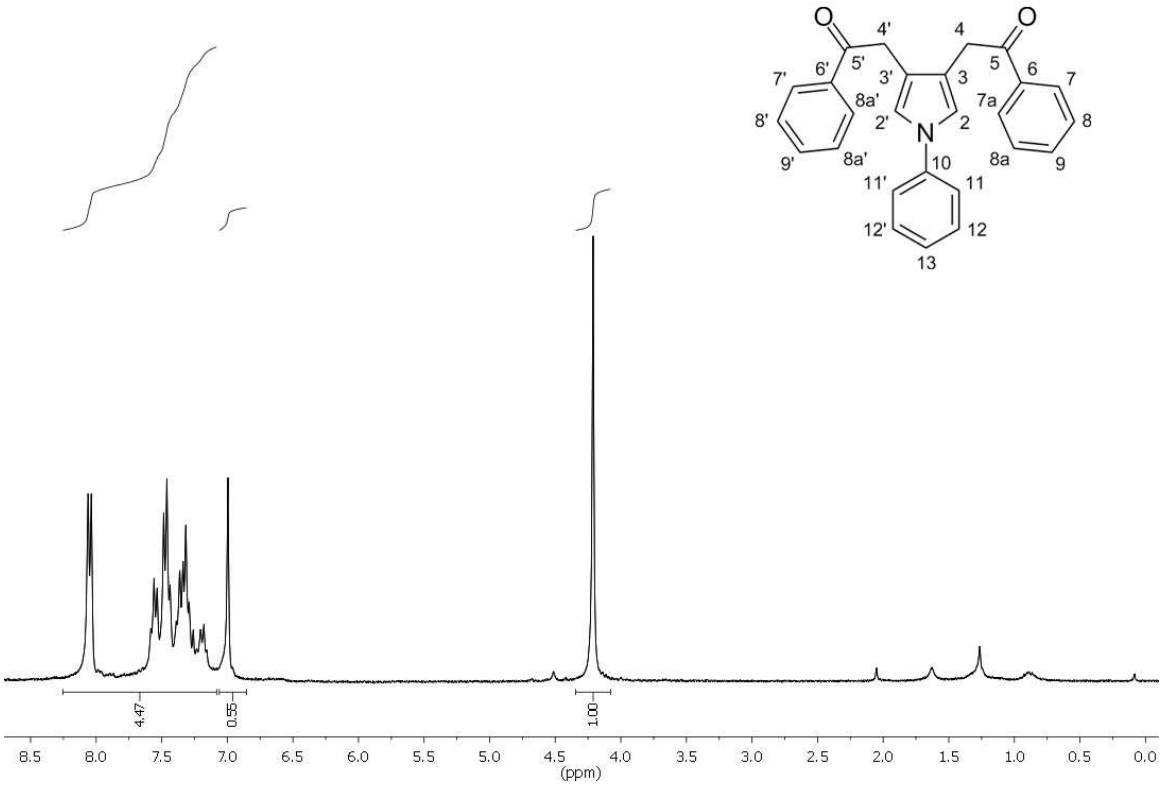


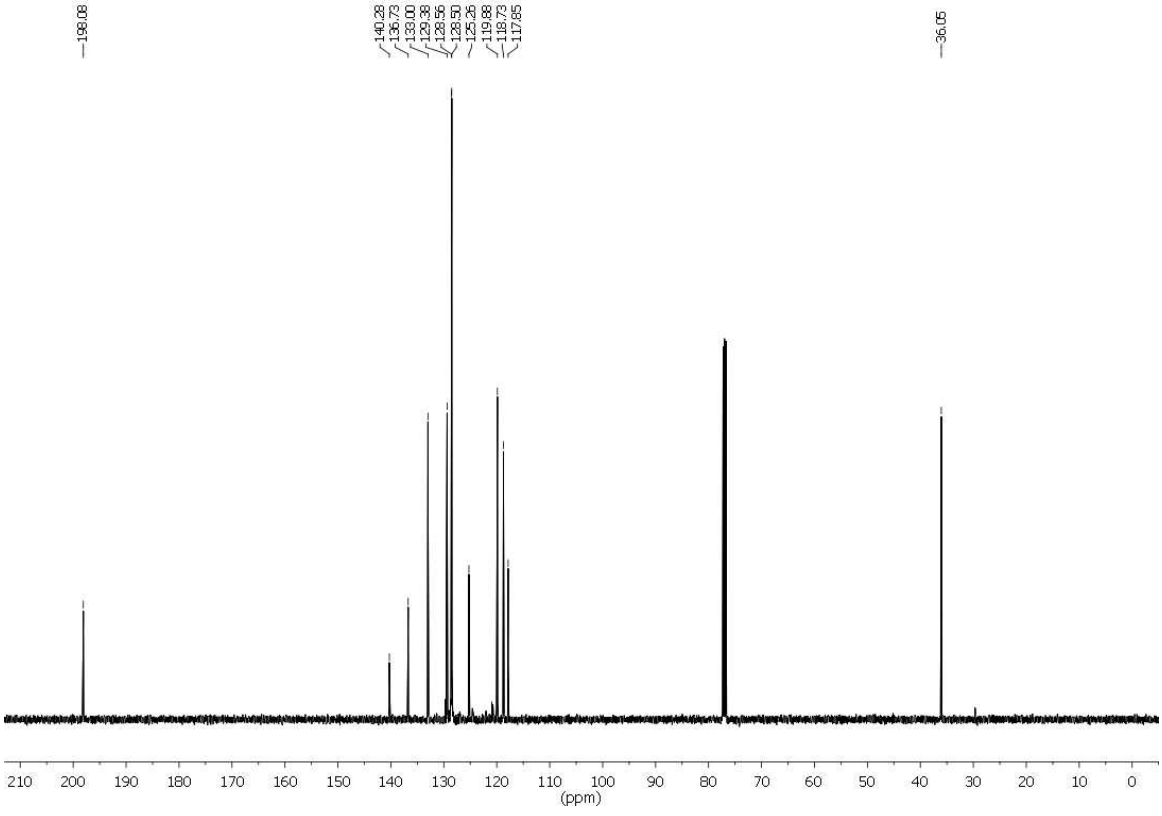
Compound 25da (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))



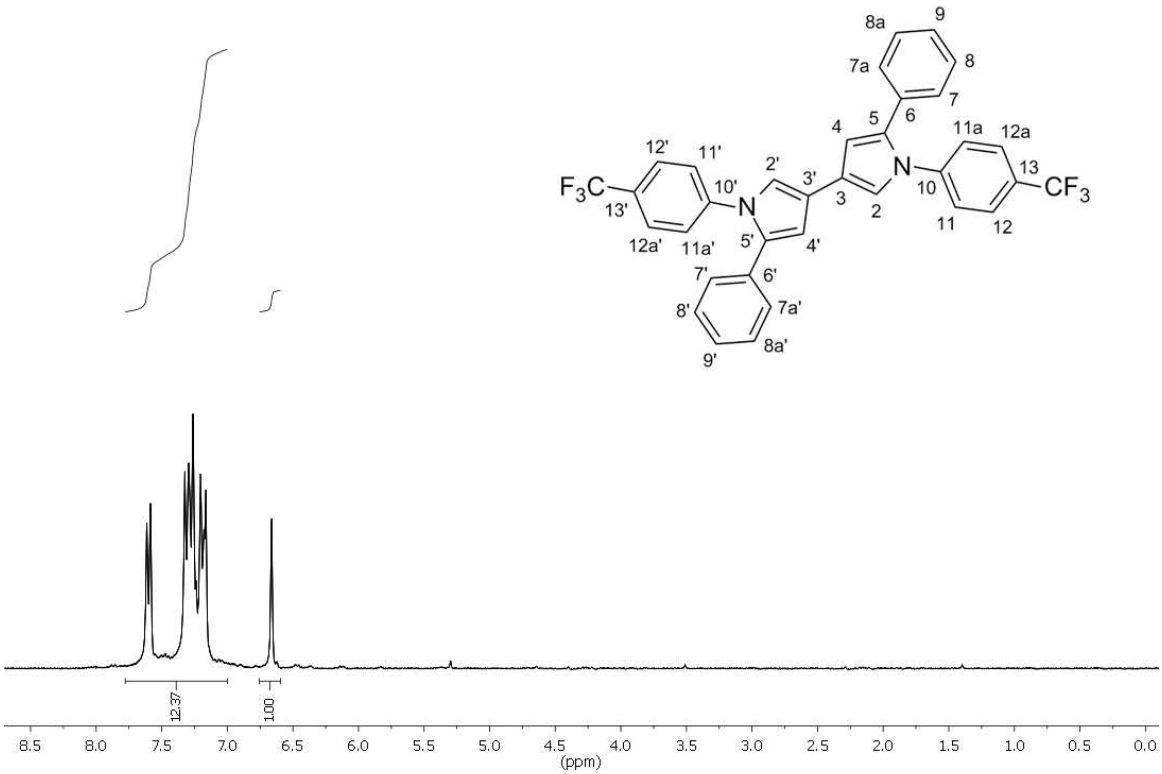


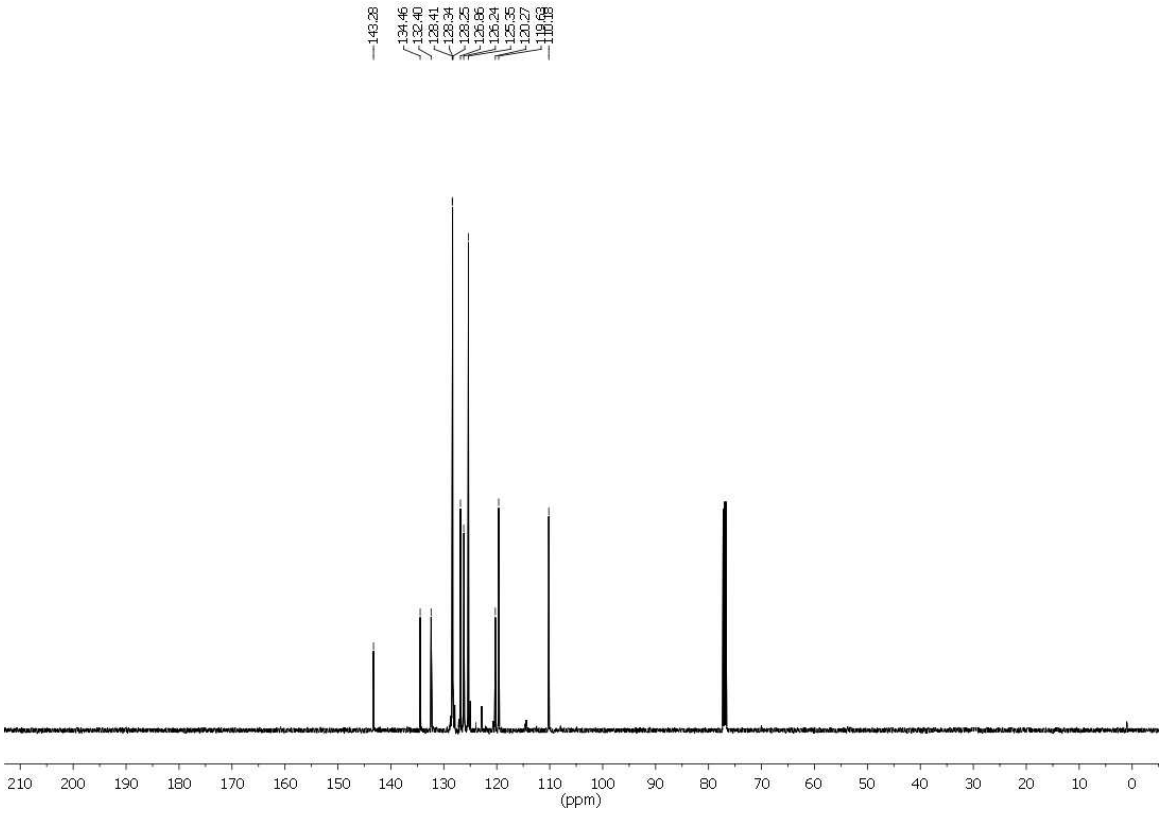
Compound 26da (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))



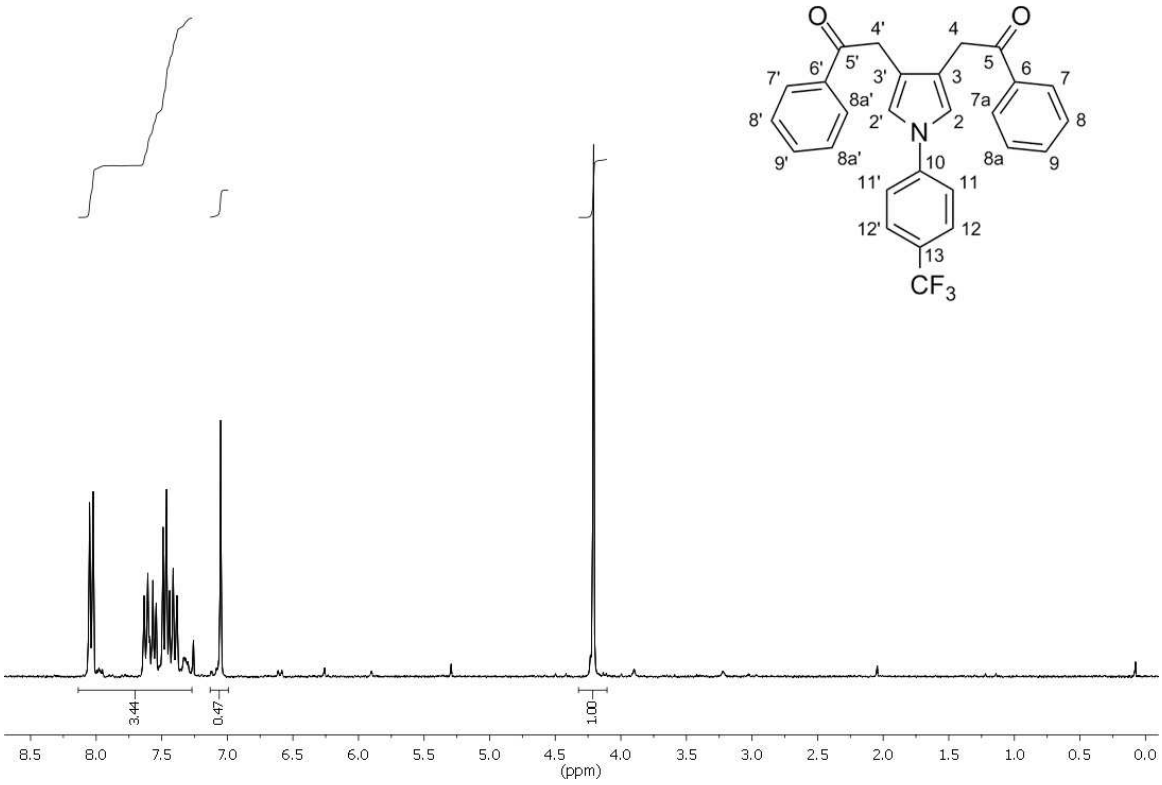


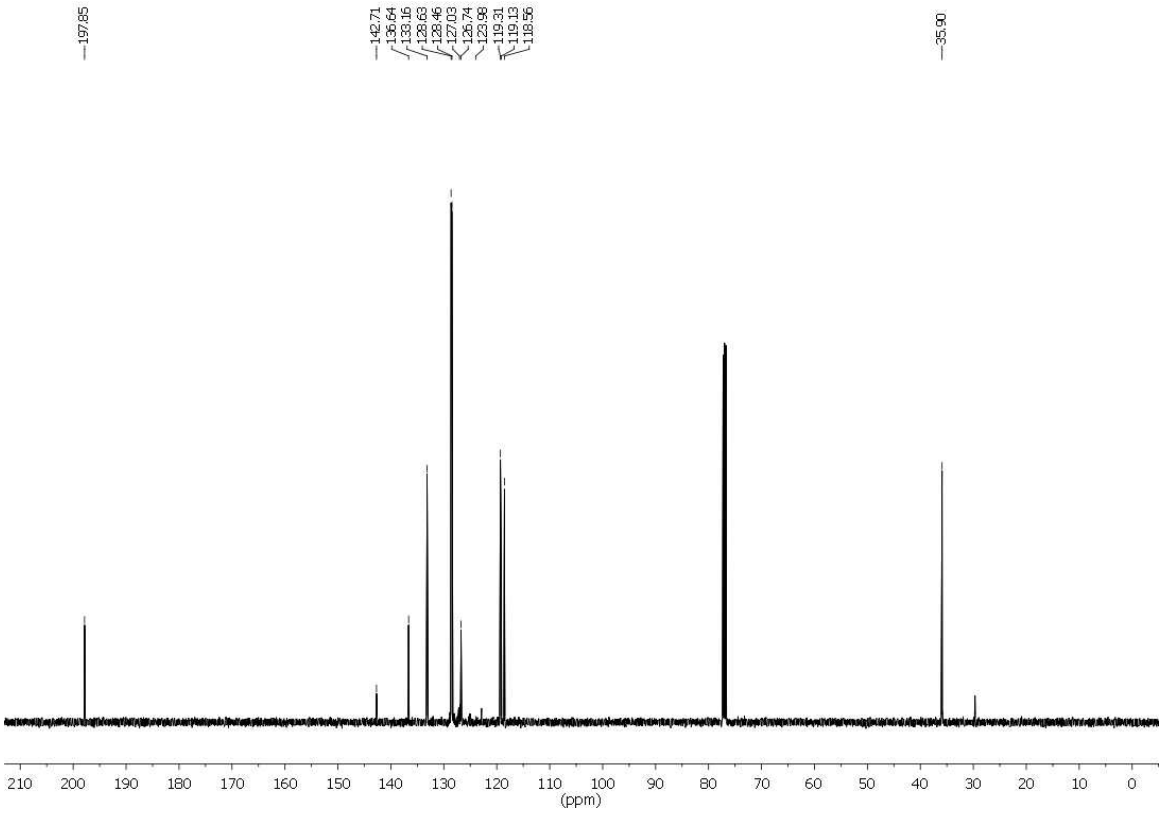
Compound 25db (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))



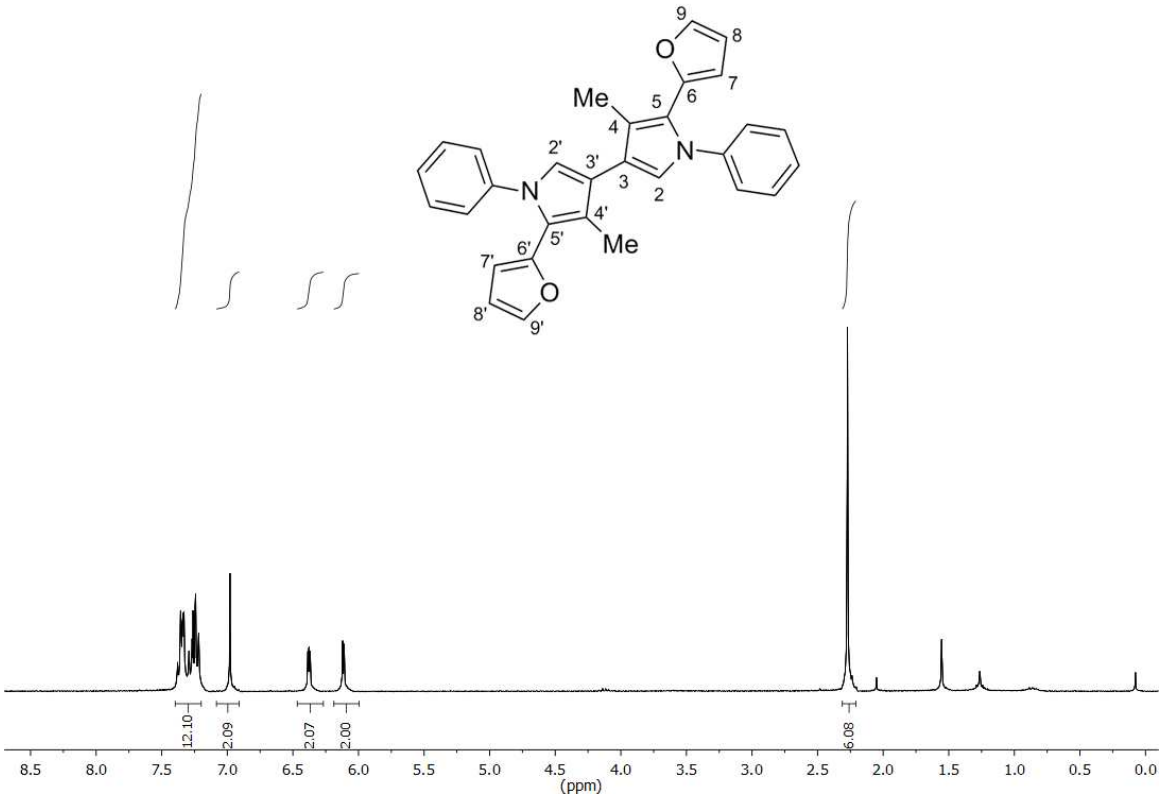


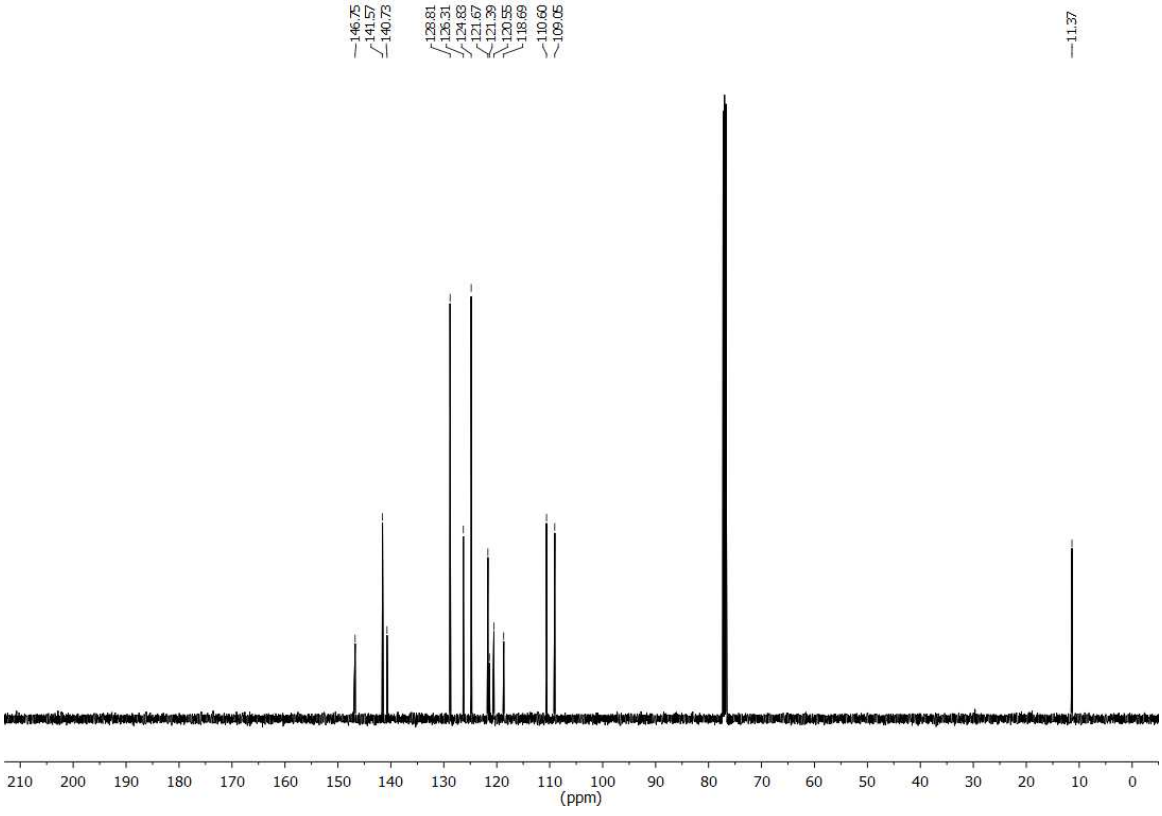
Compound 26db (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))



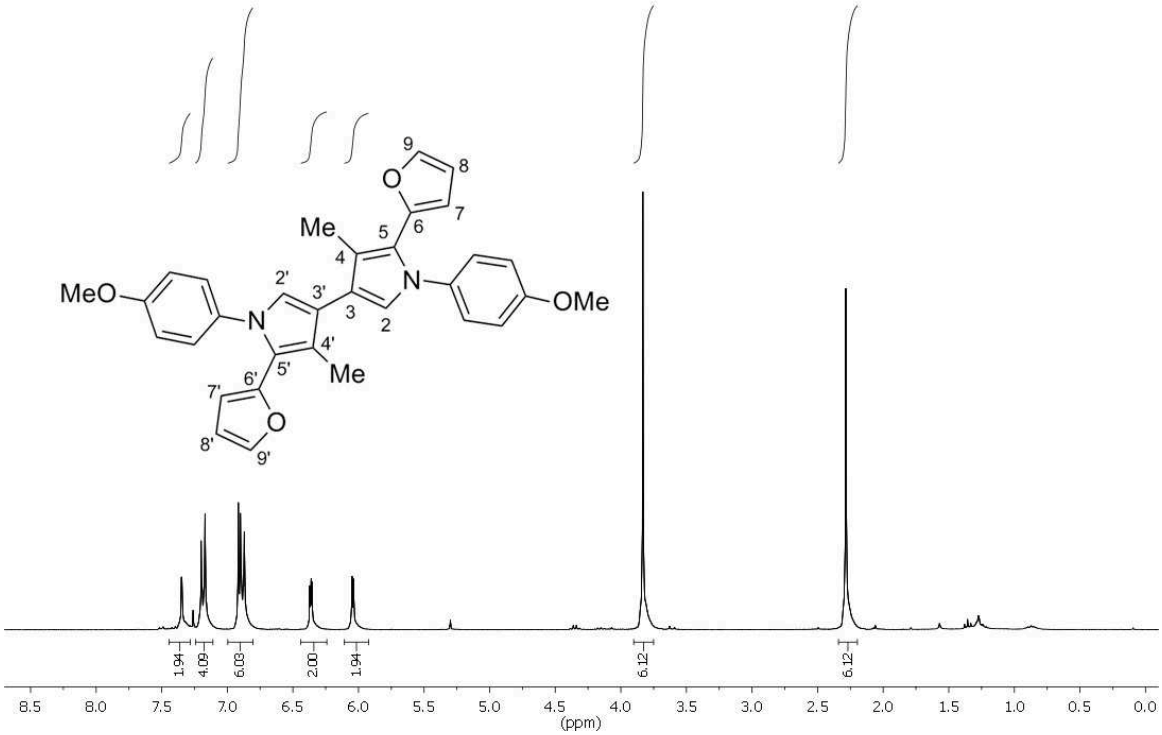


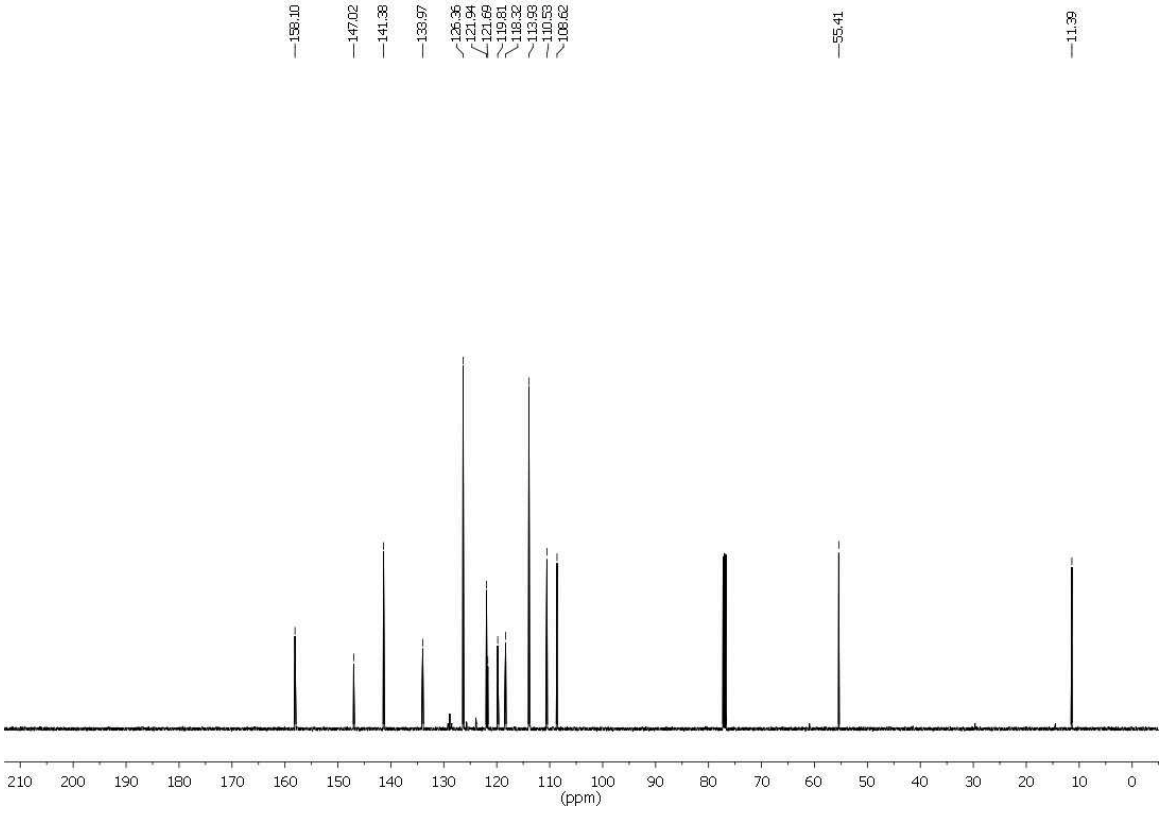
Compound 25ea (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))



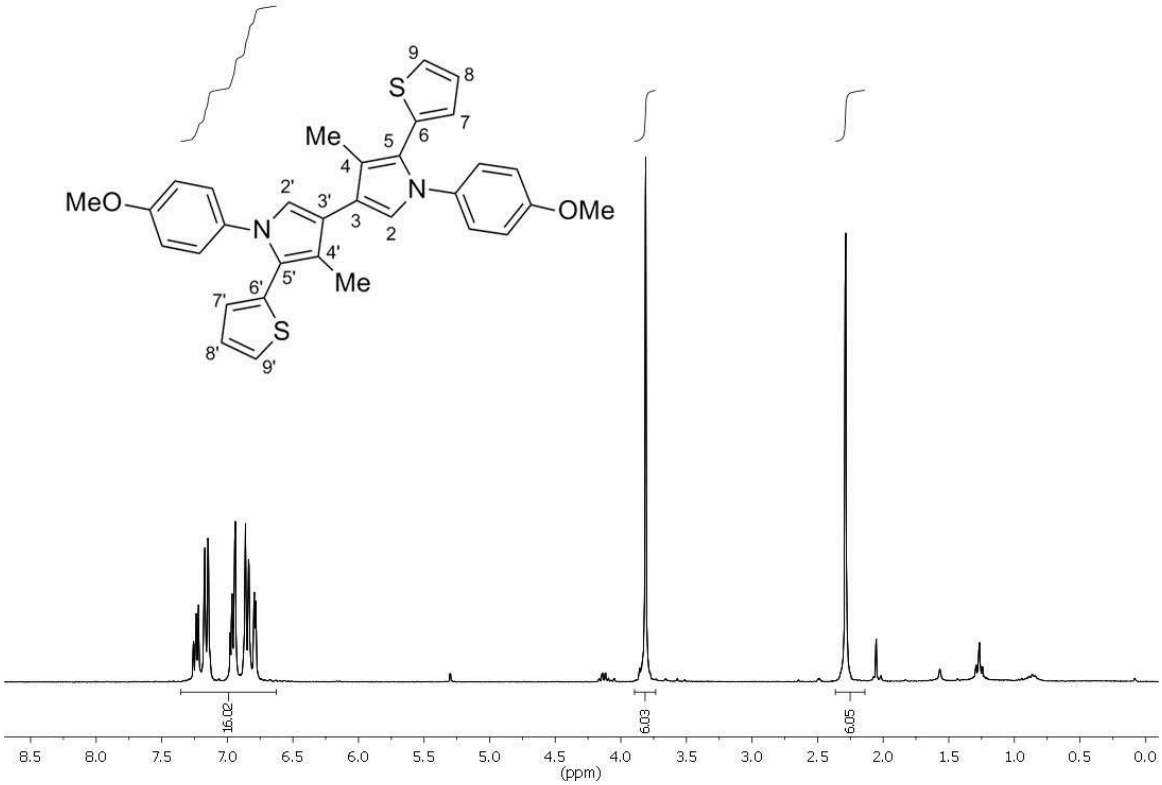


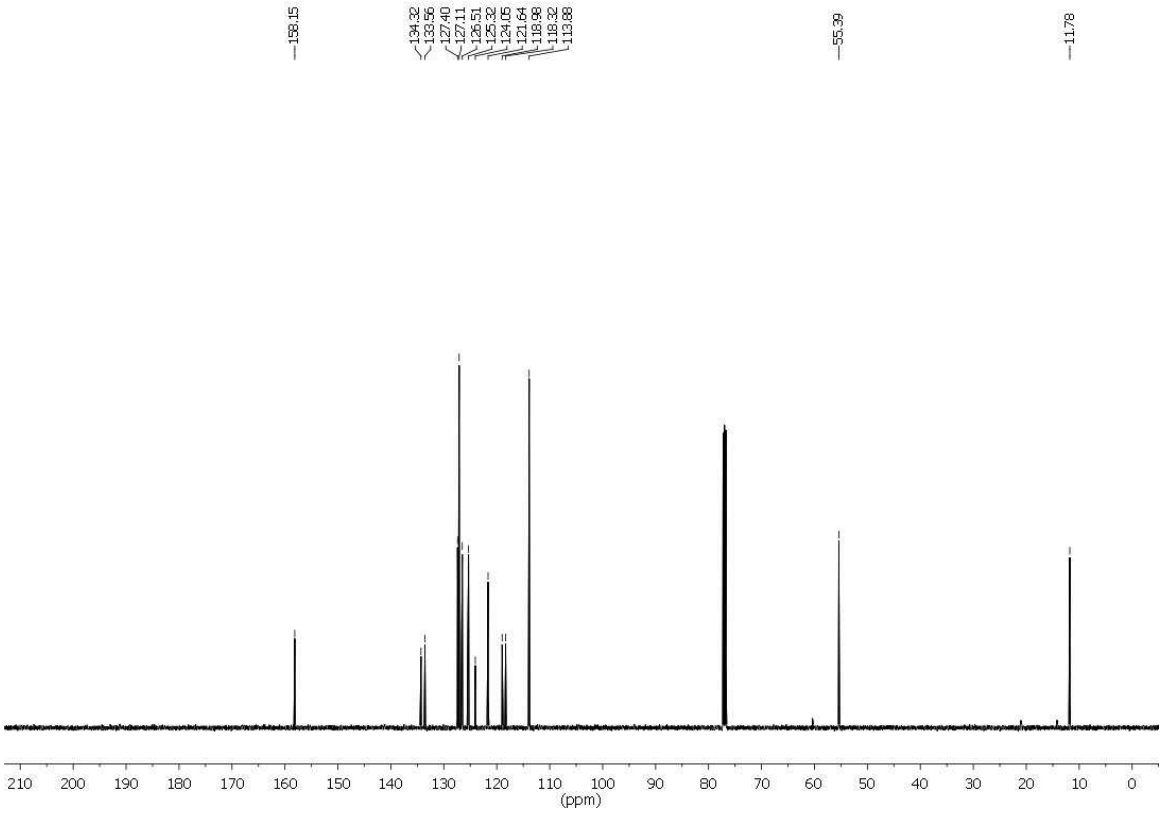
Compound 25eb (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))



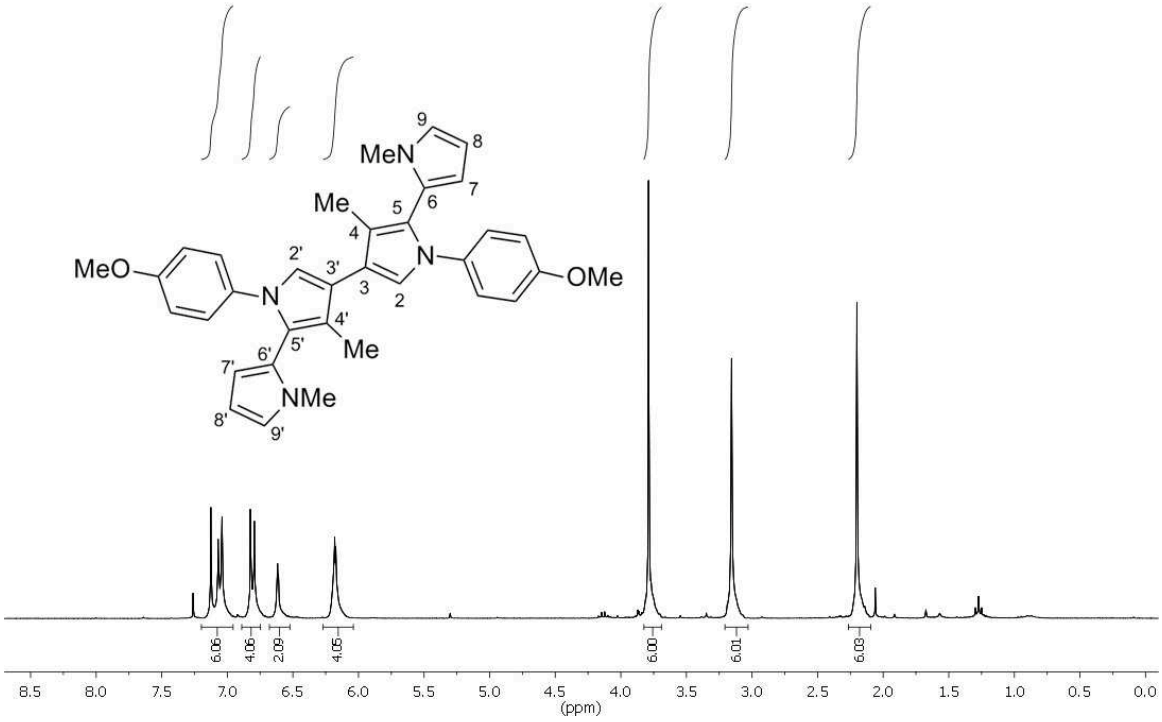


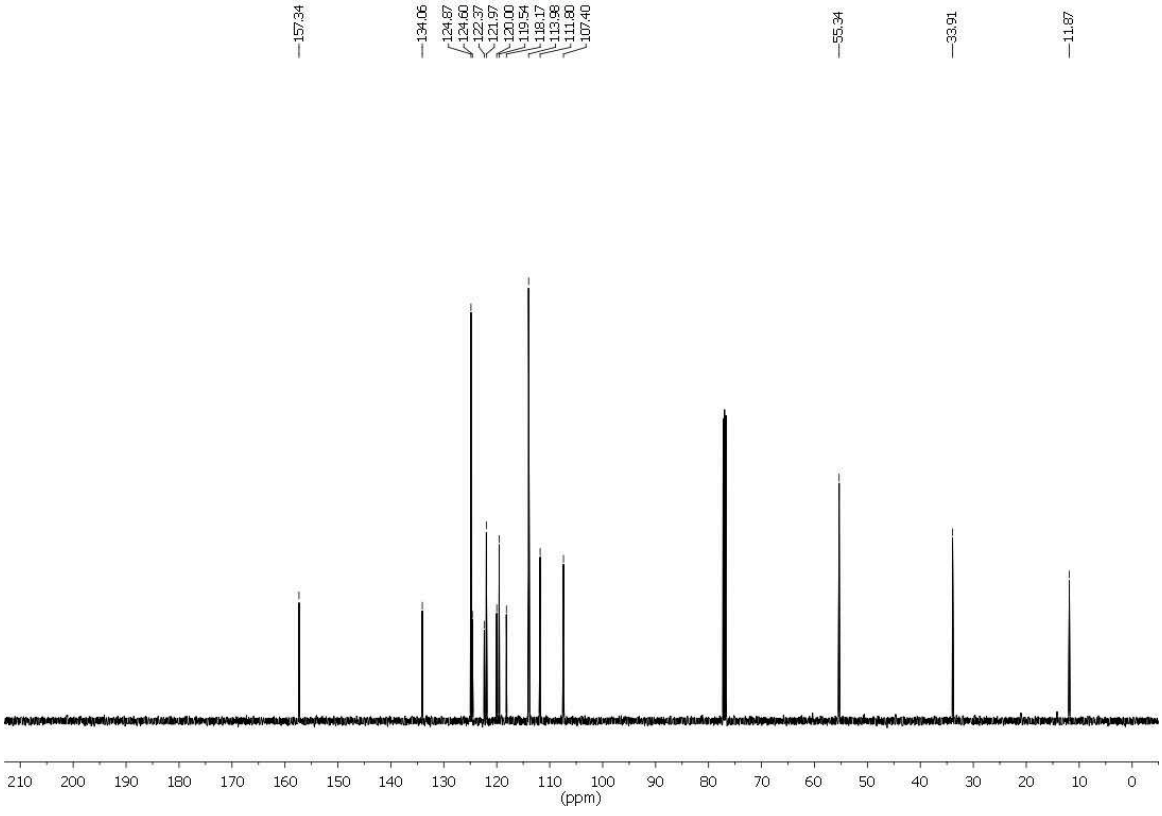
Compound 25fa (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))



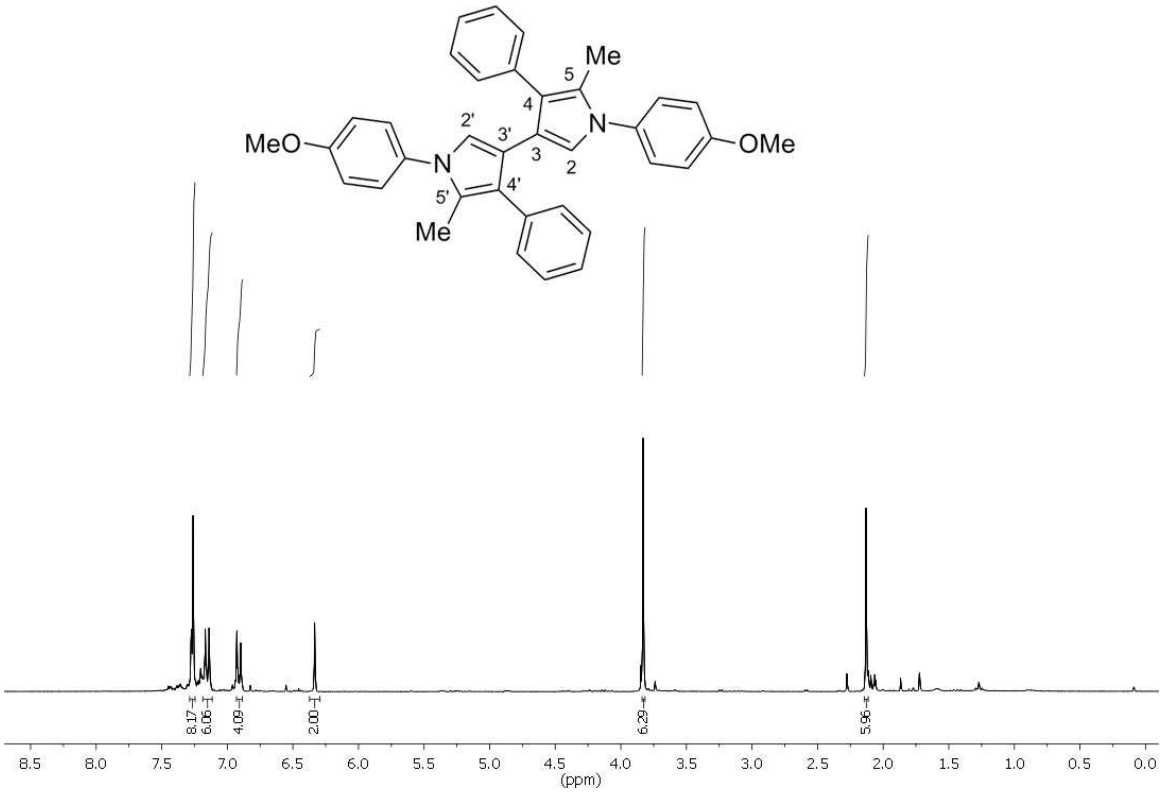


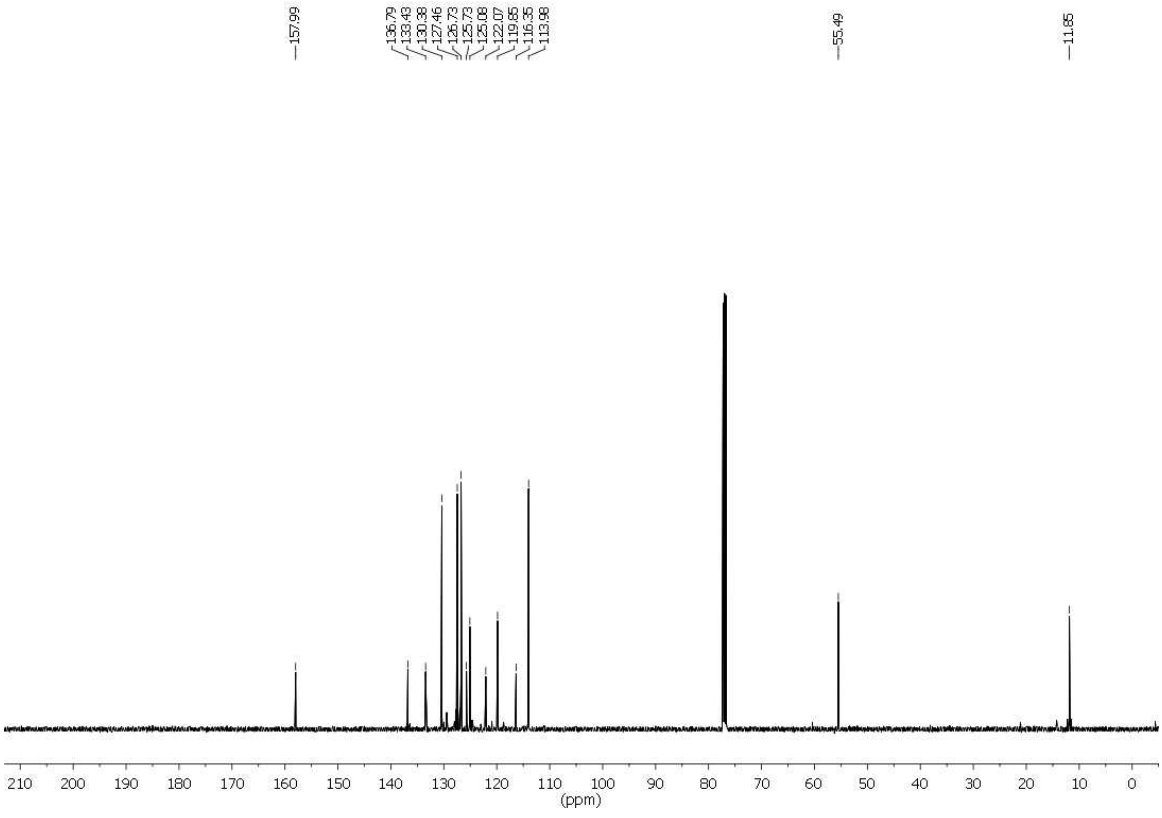
Compound 25ga (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))



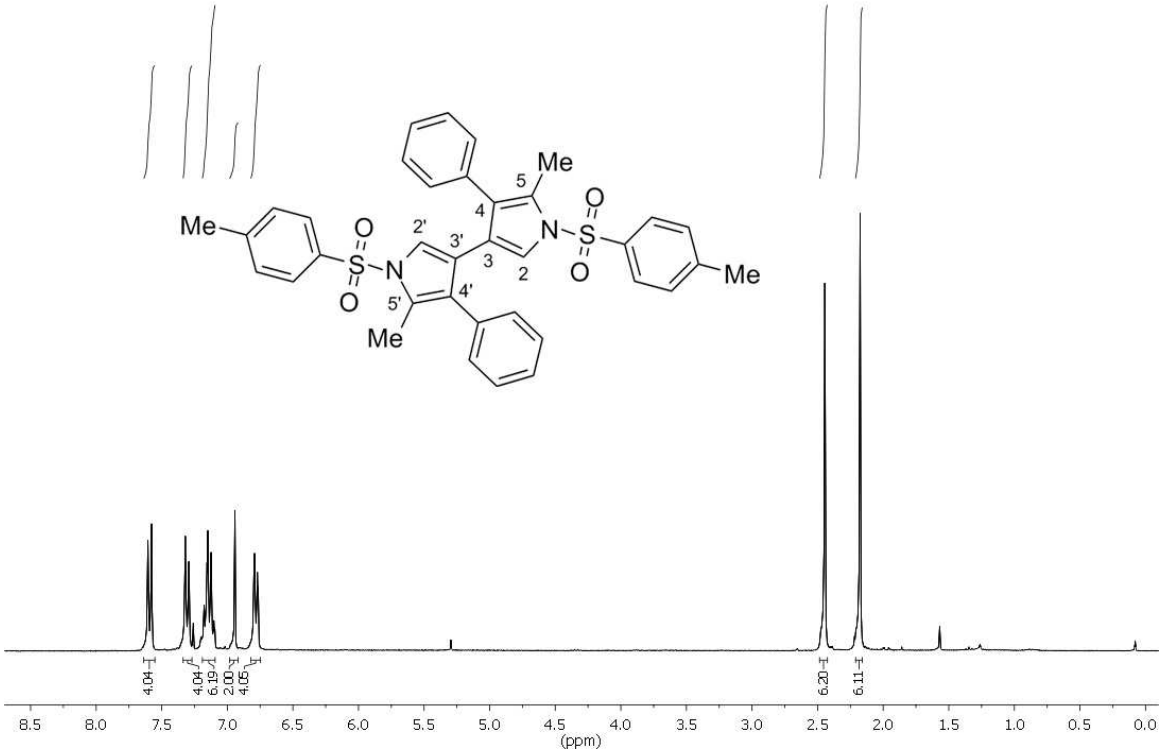


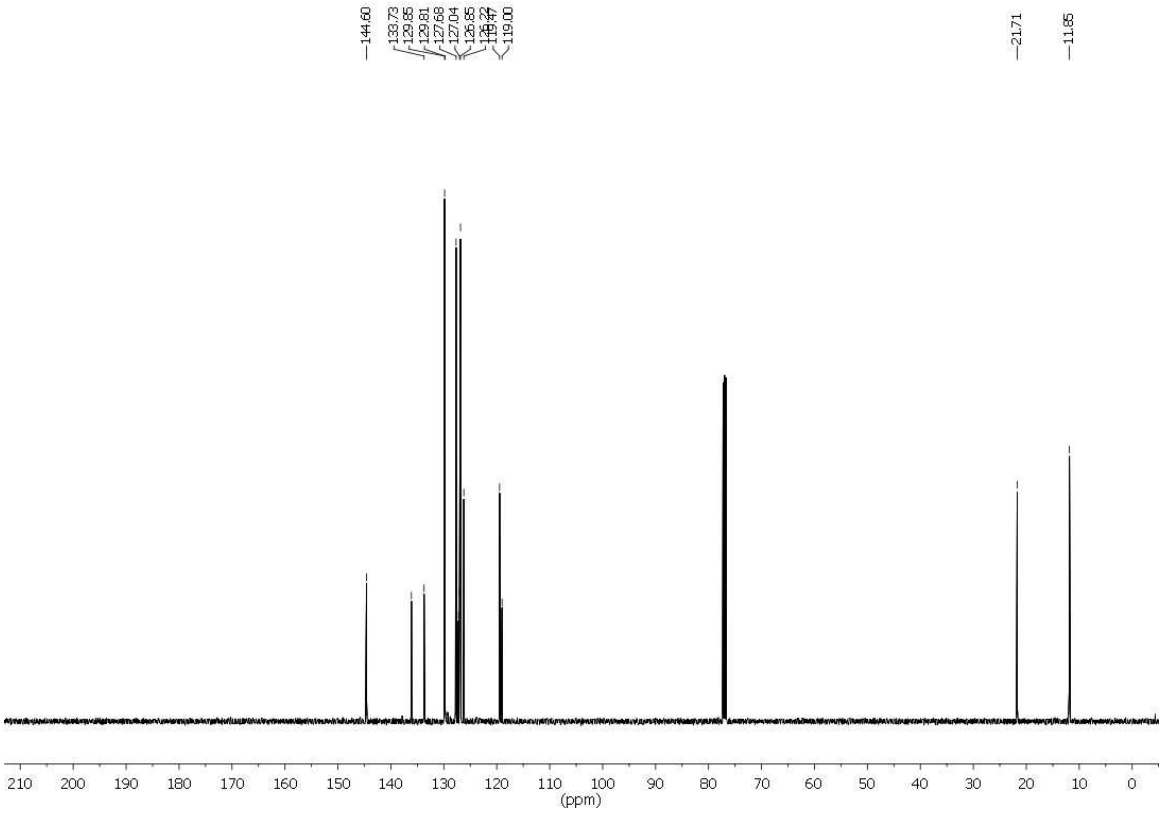
Compound 25ha (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))



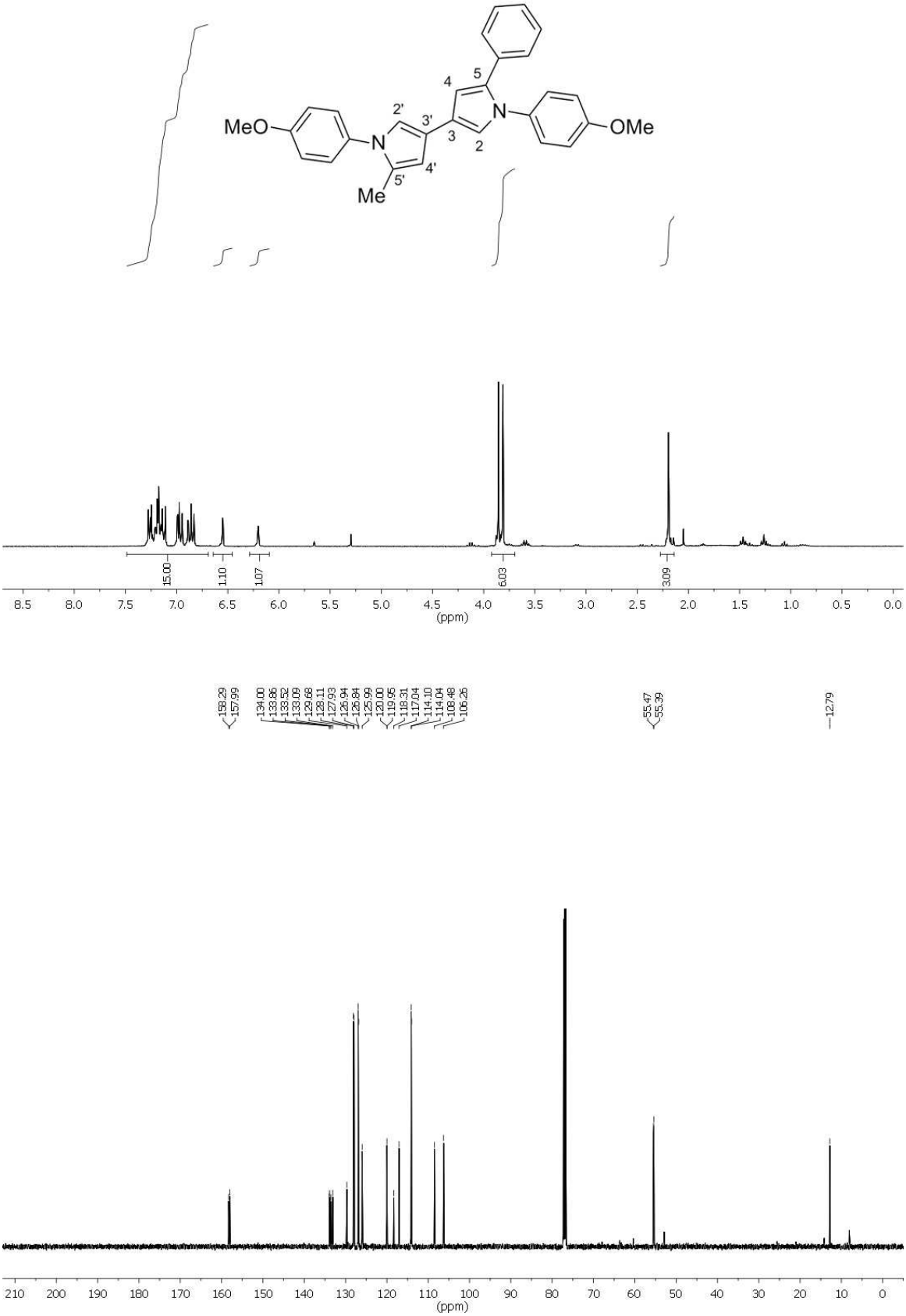


Compound 25hb (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))

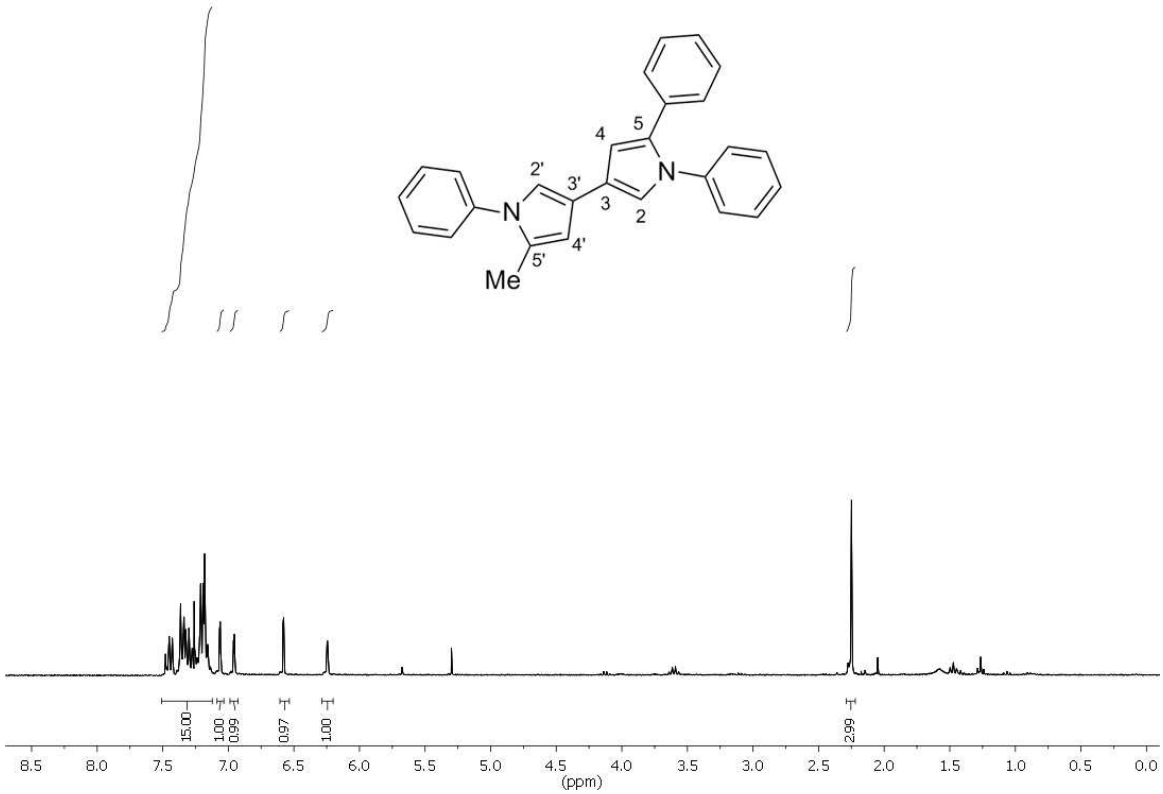


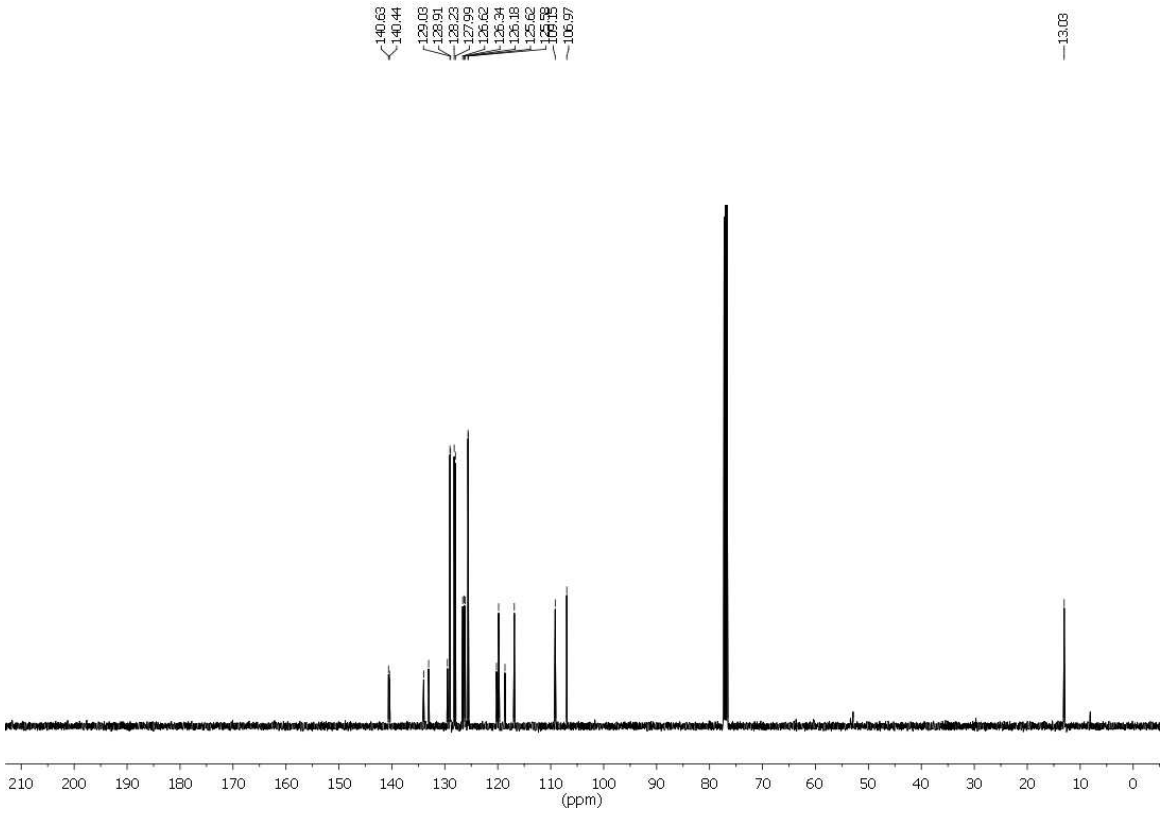


Compound 34a (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))

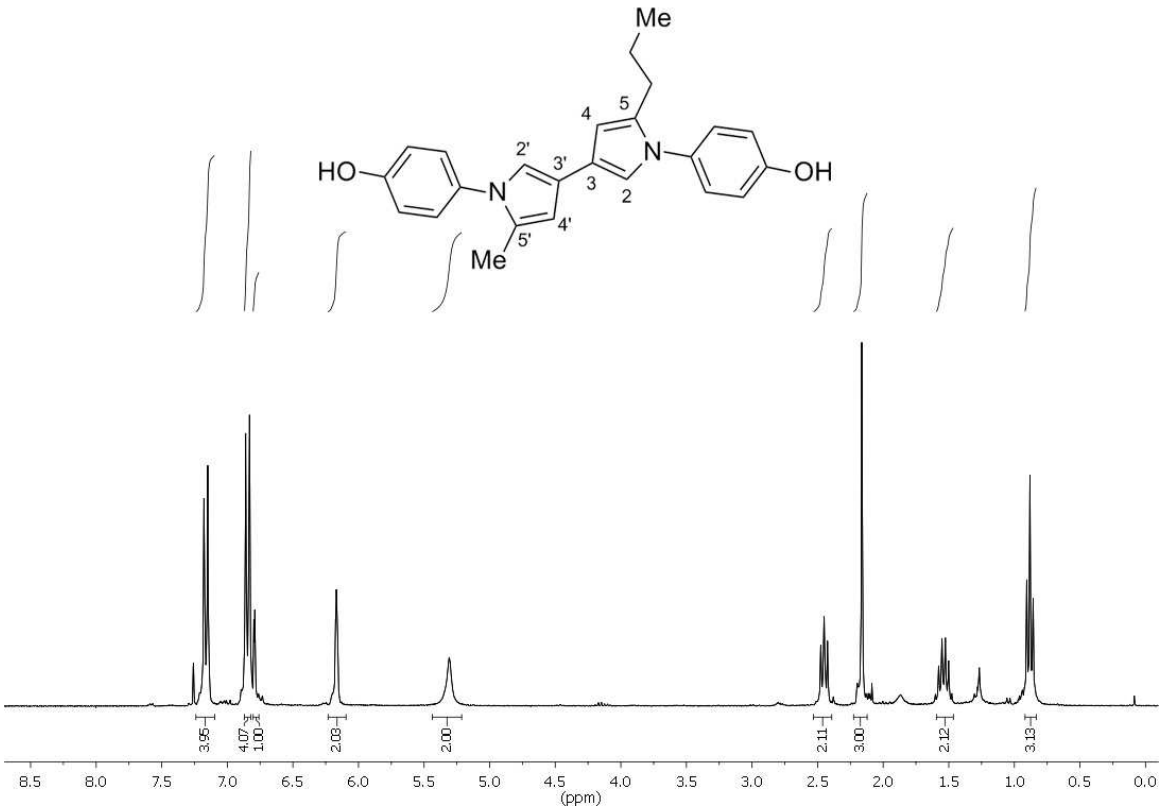


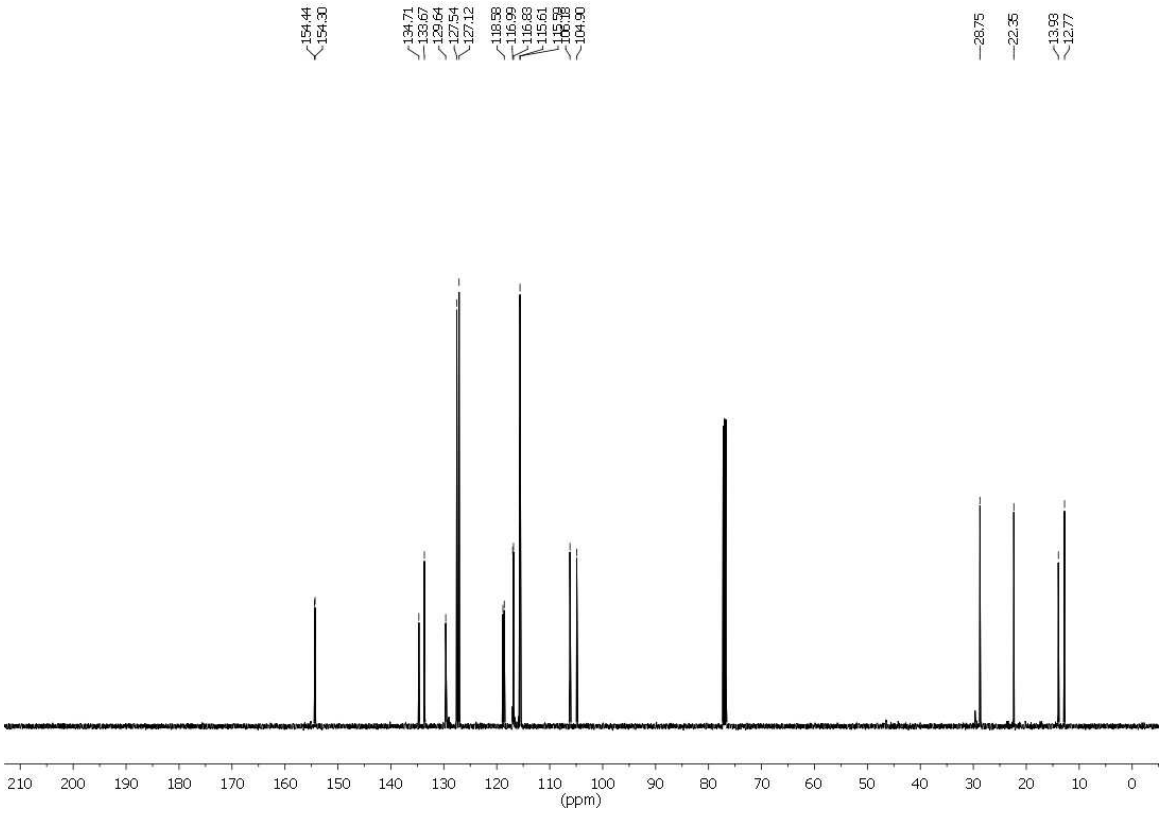
Compound 34b (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))



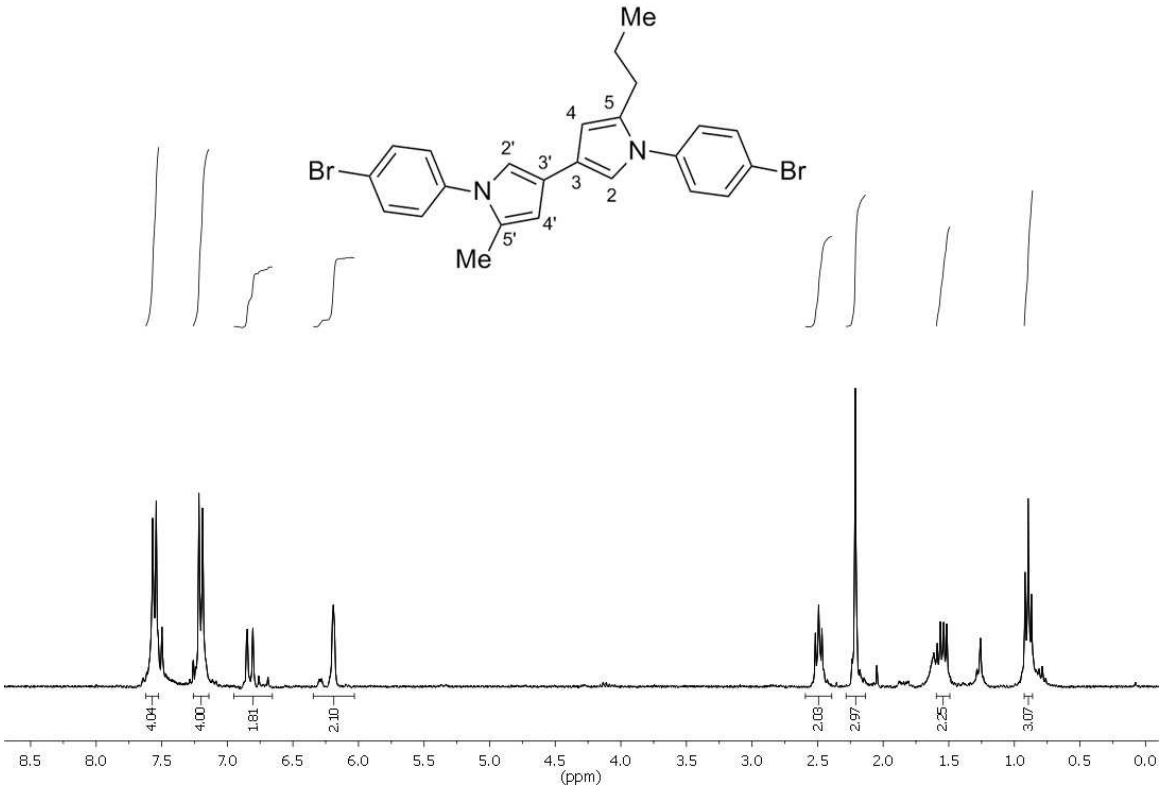


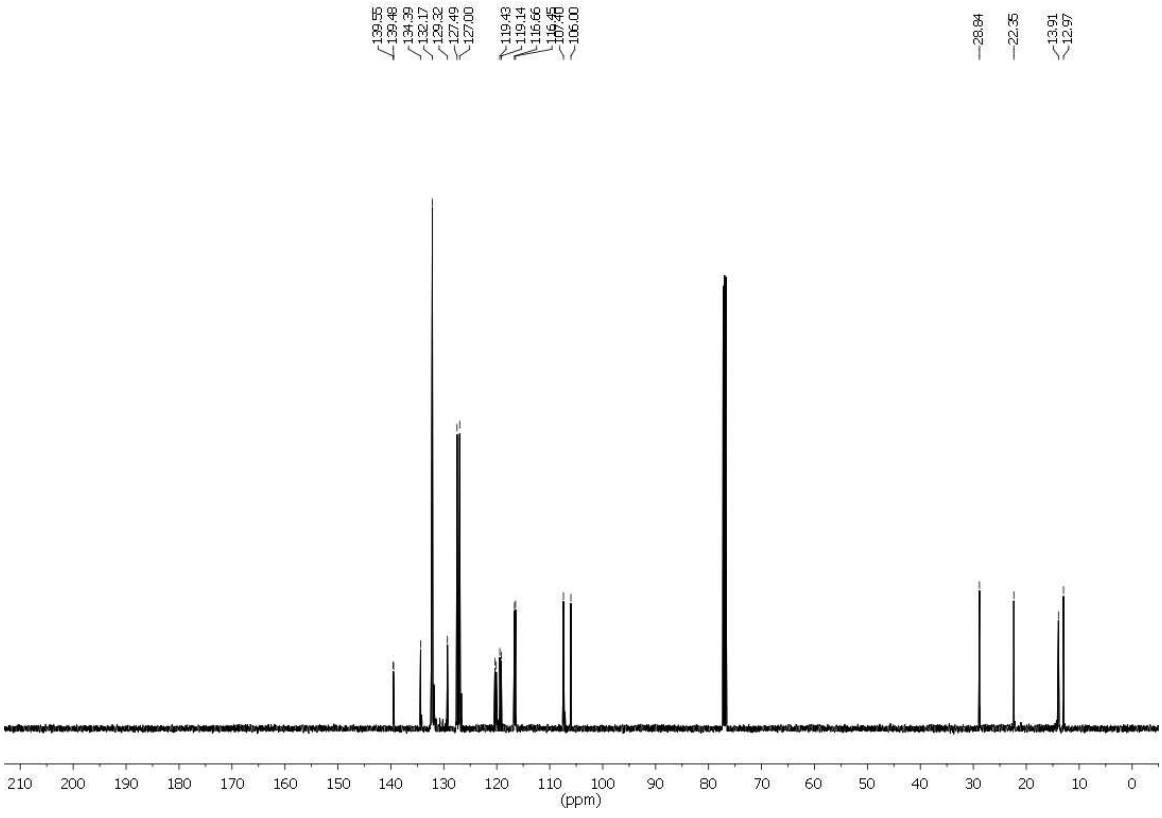
Compound 34c (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))



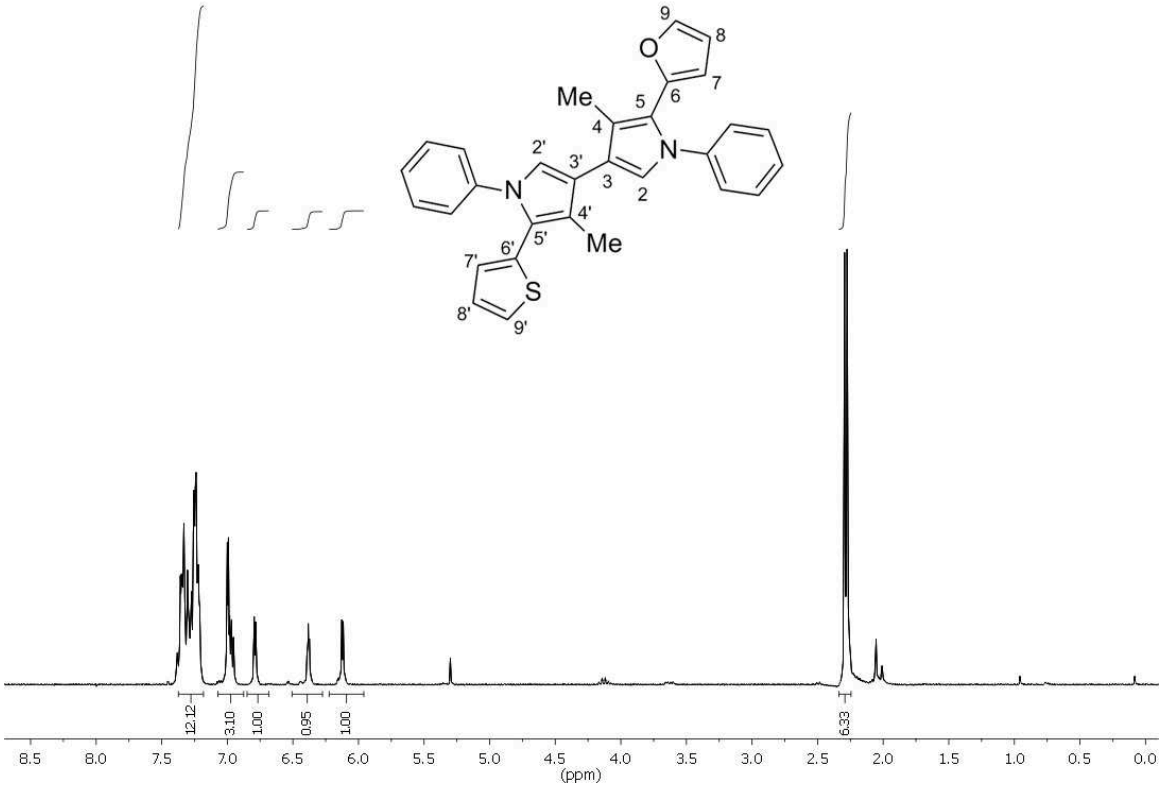


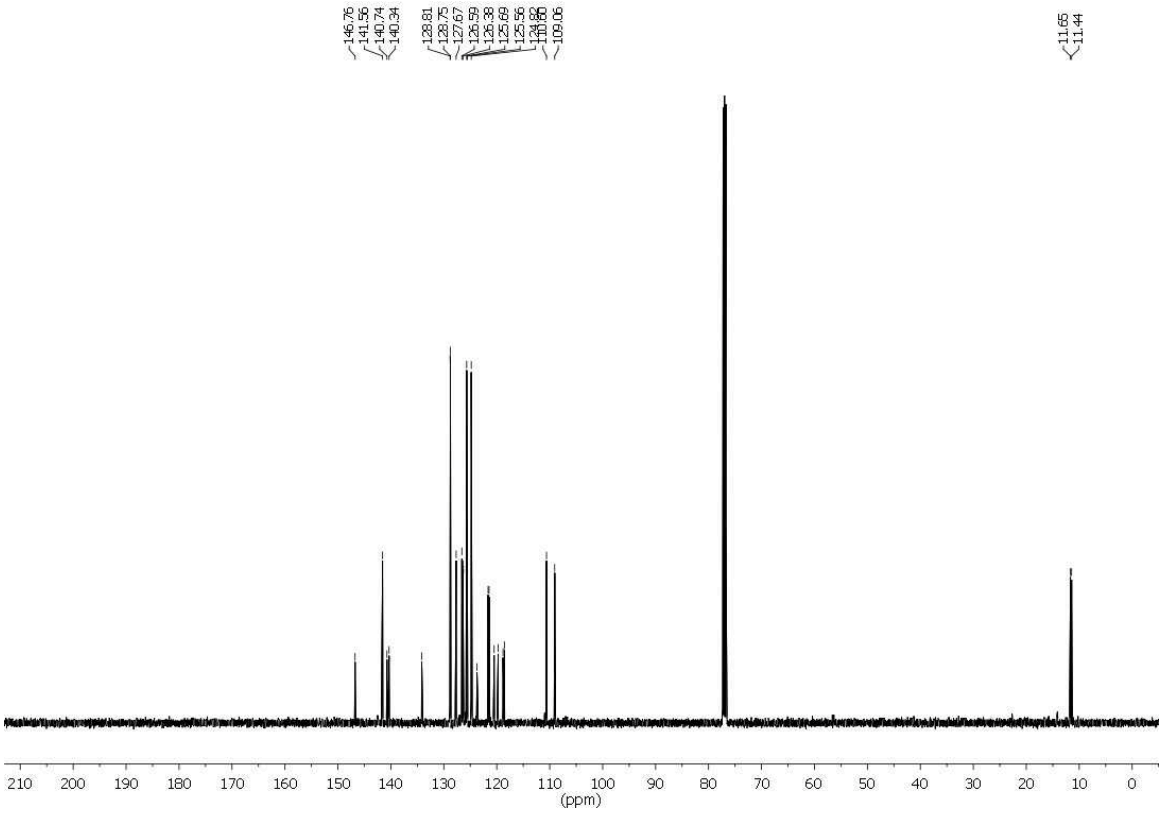
Compound 34d (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))



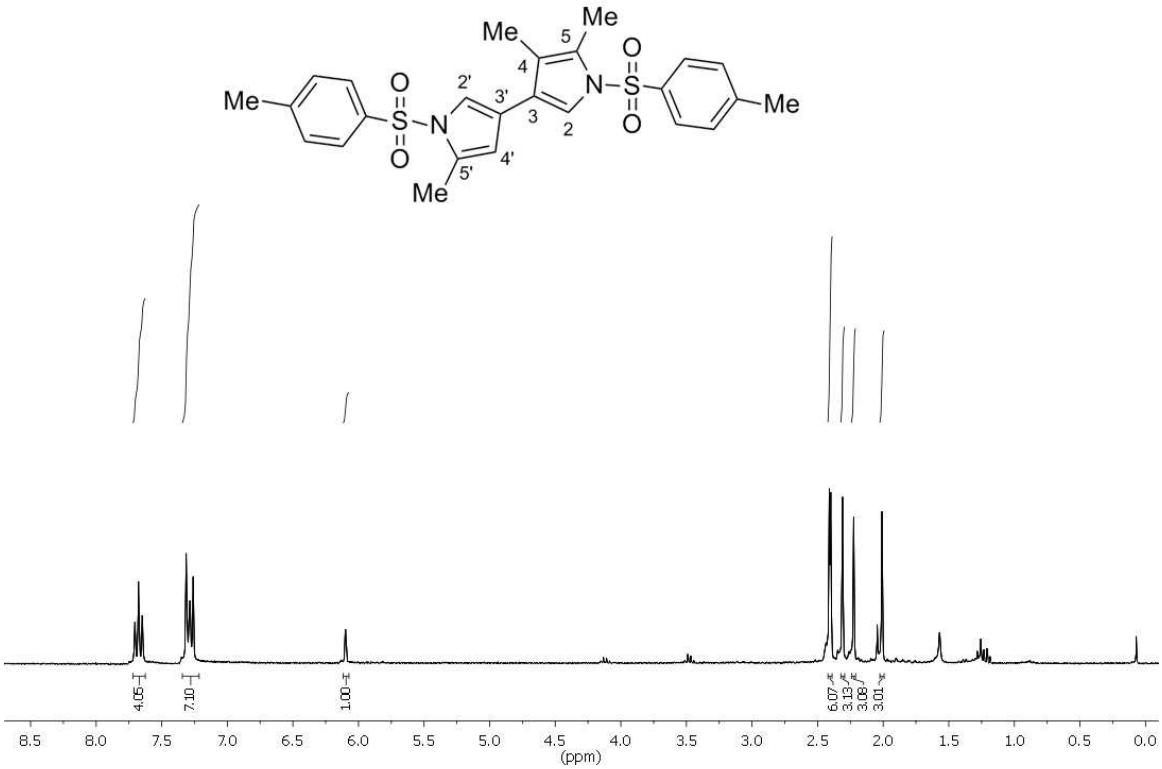


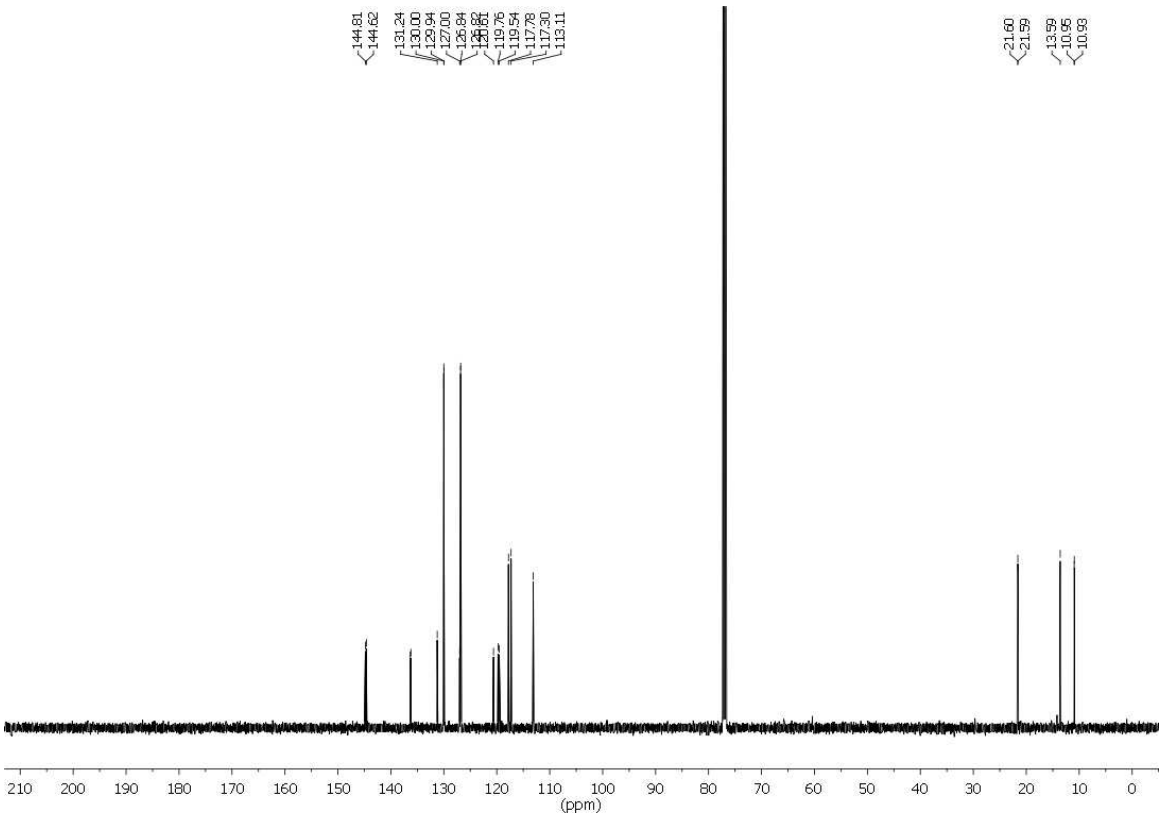
Compound 34e (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))



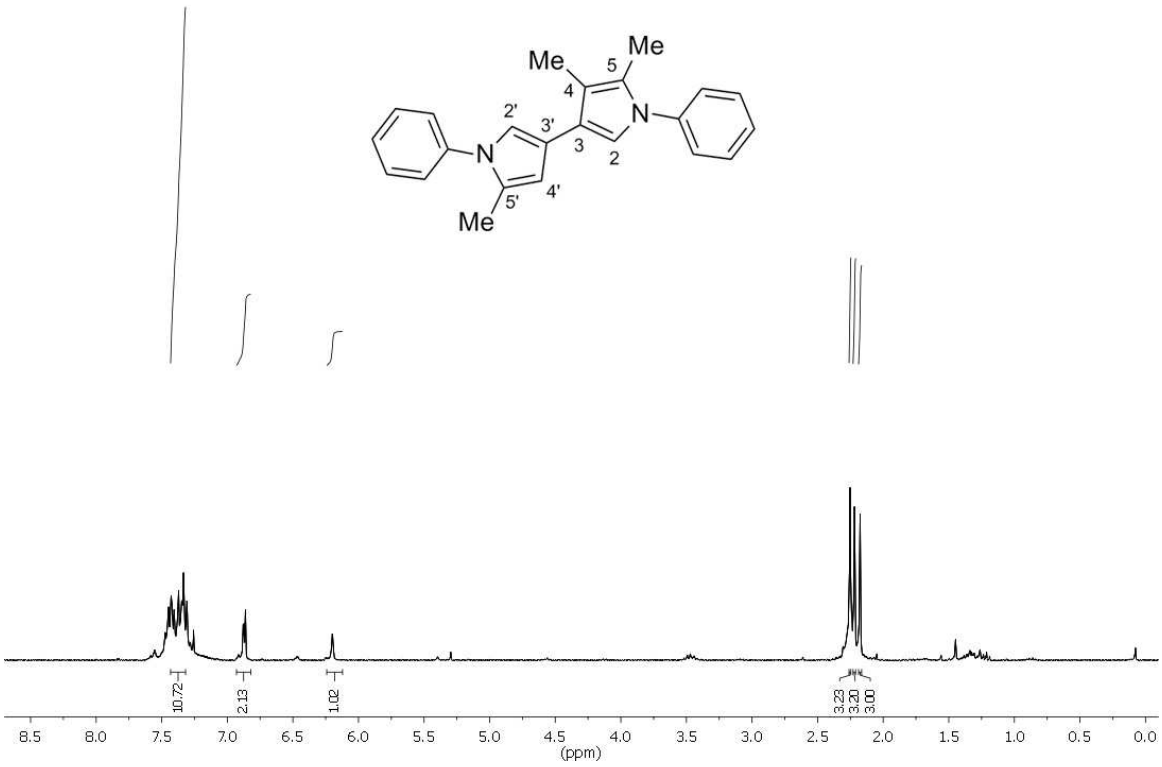


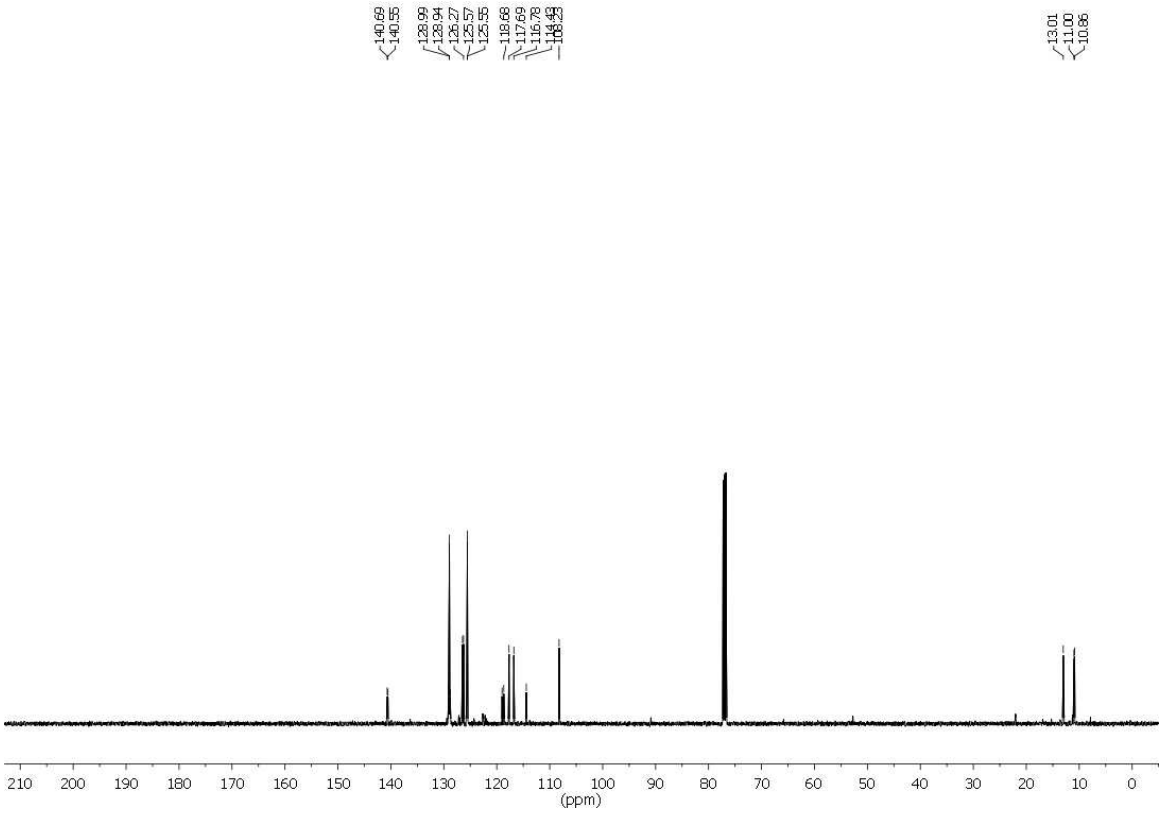
Compound 35a (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))



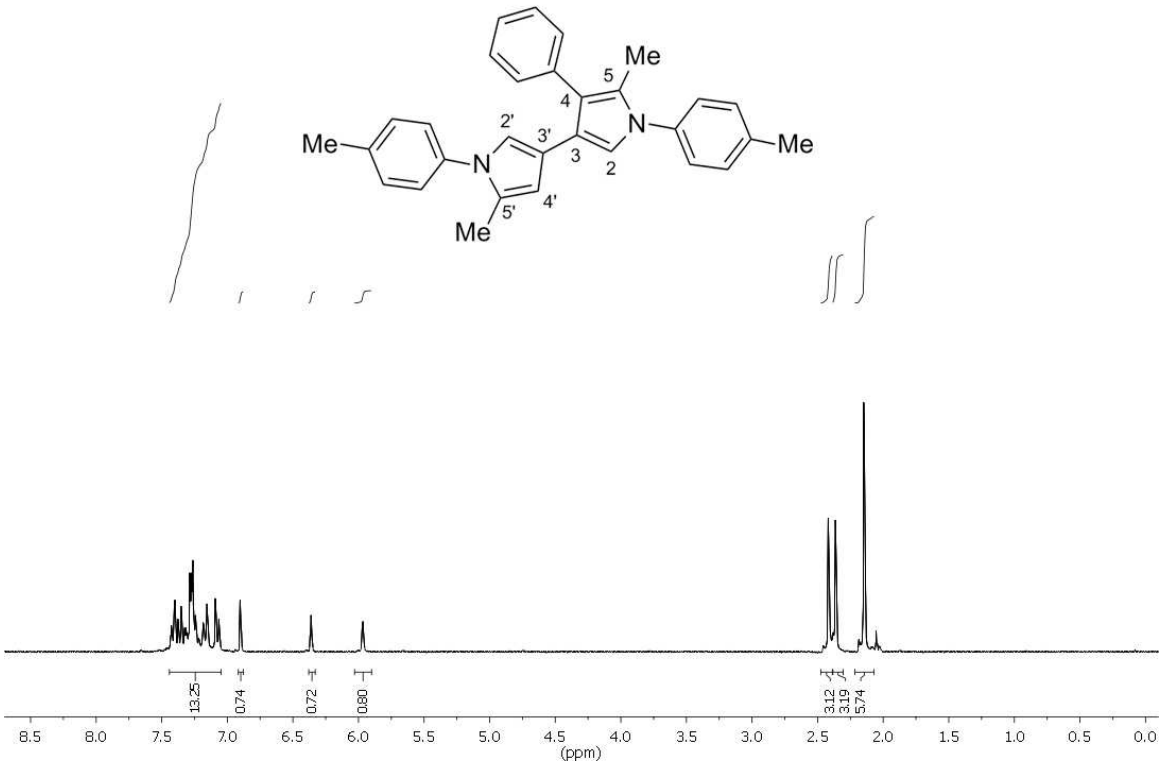


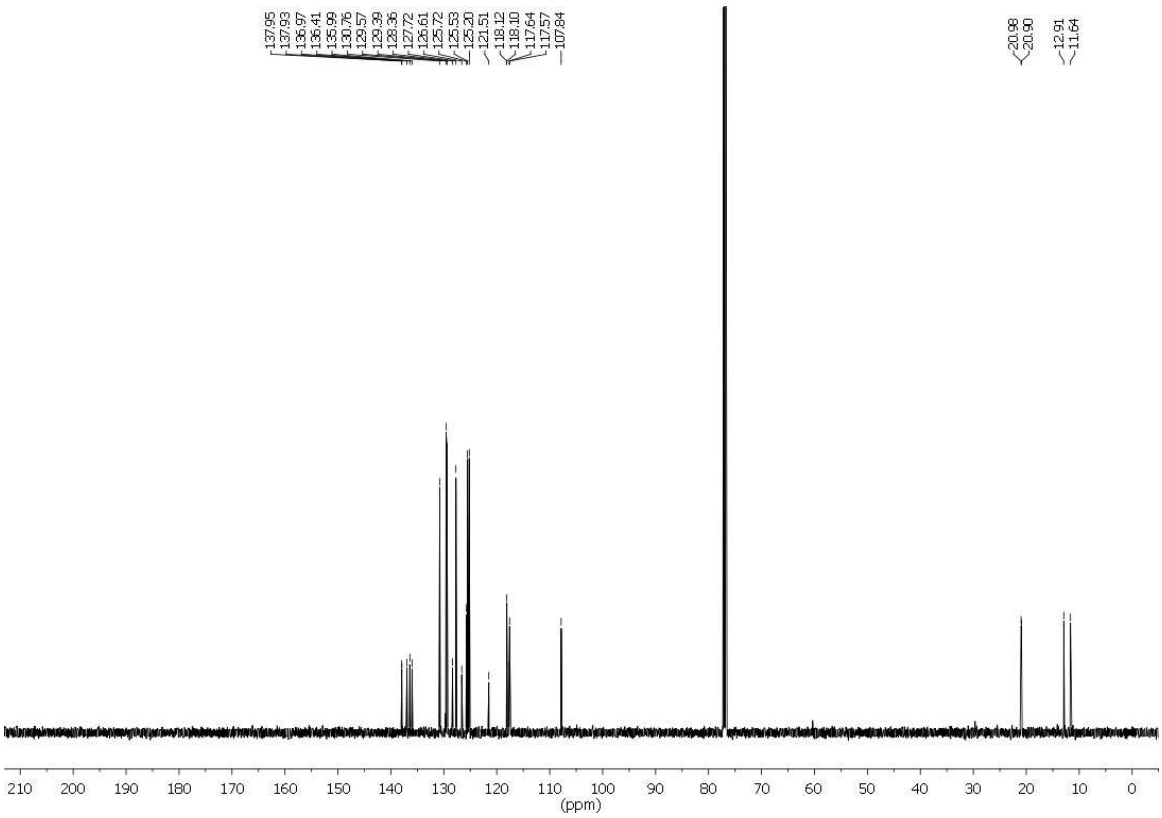
Compound 35b (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))



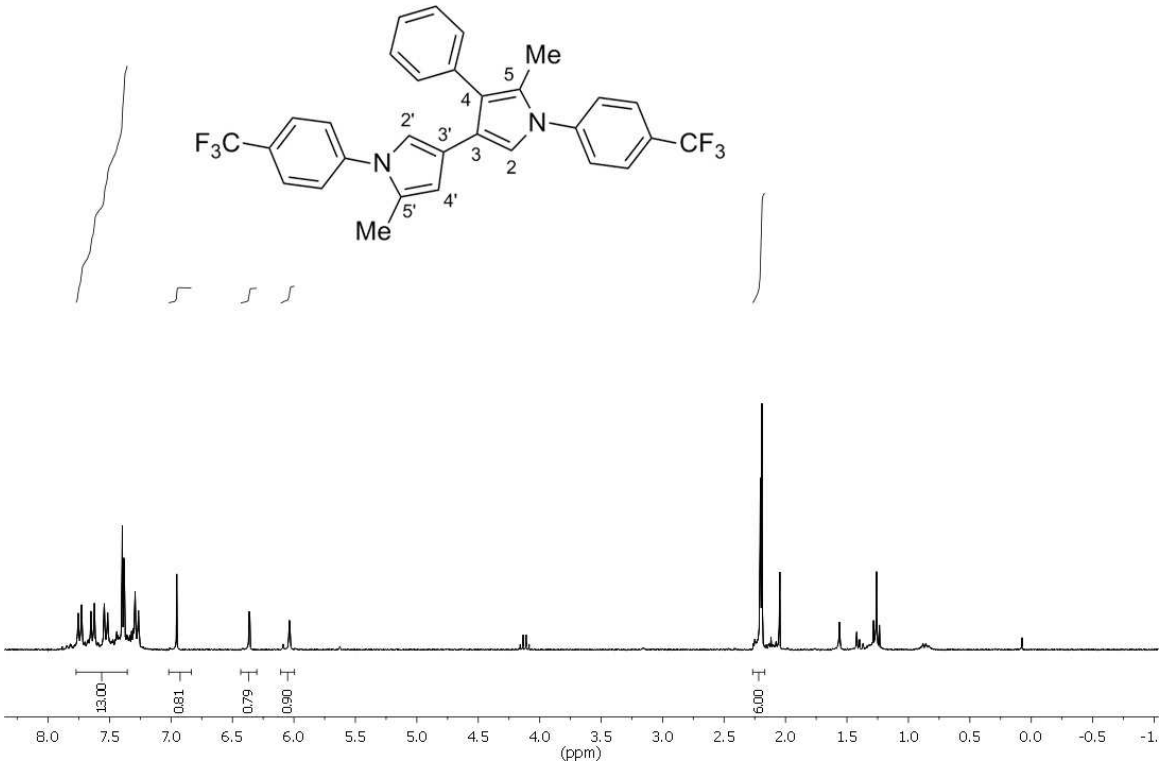


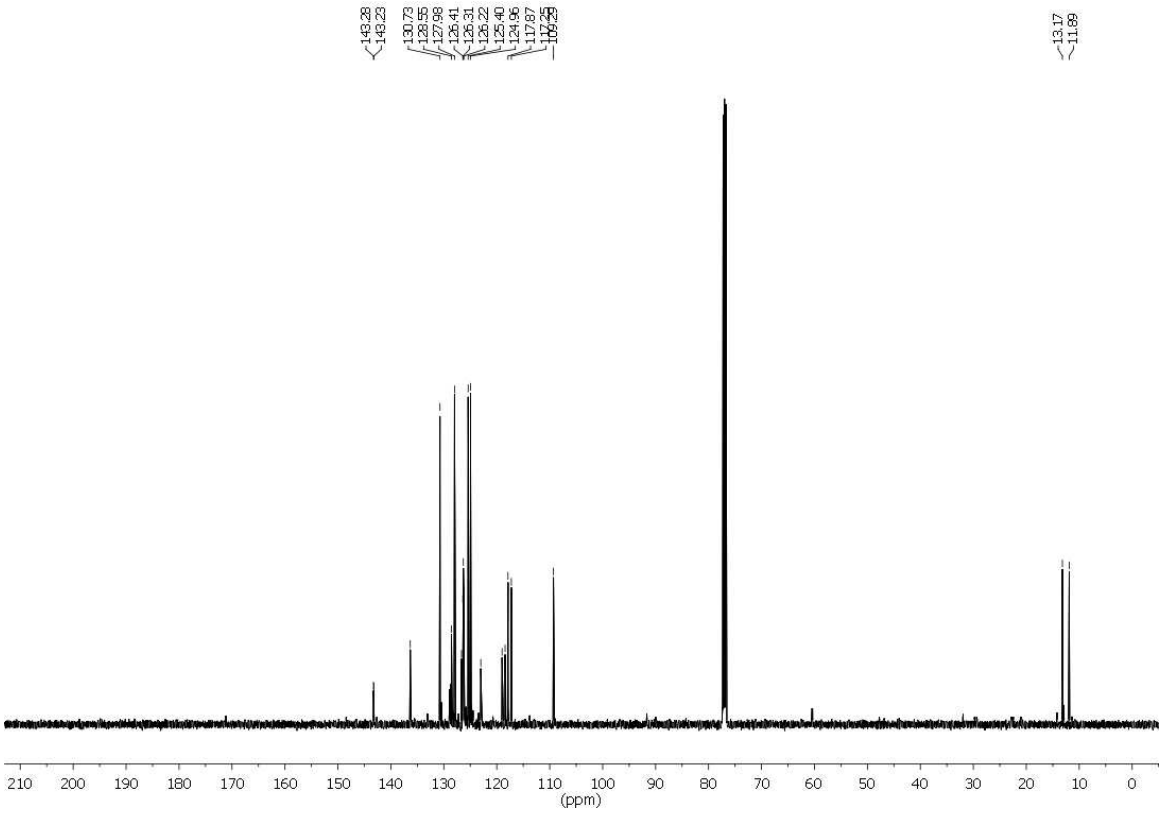
Compound 35c (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))



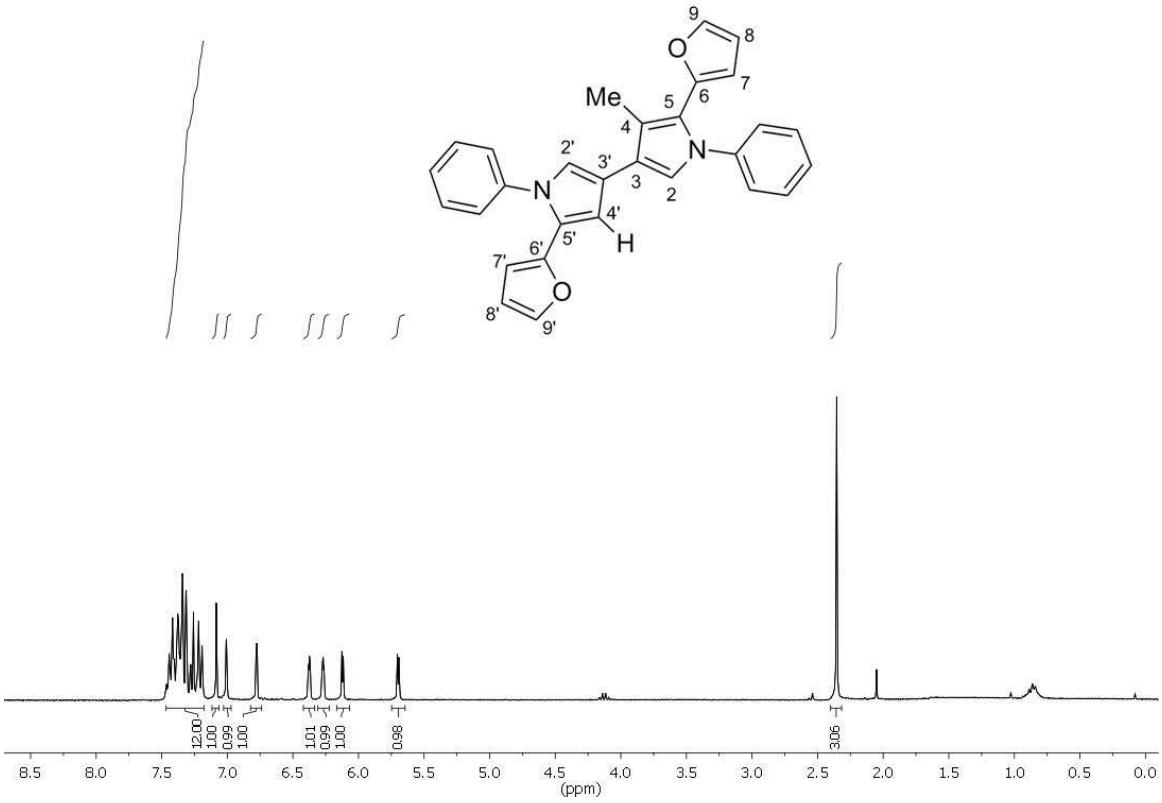


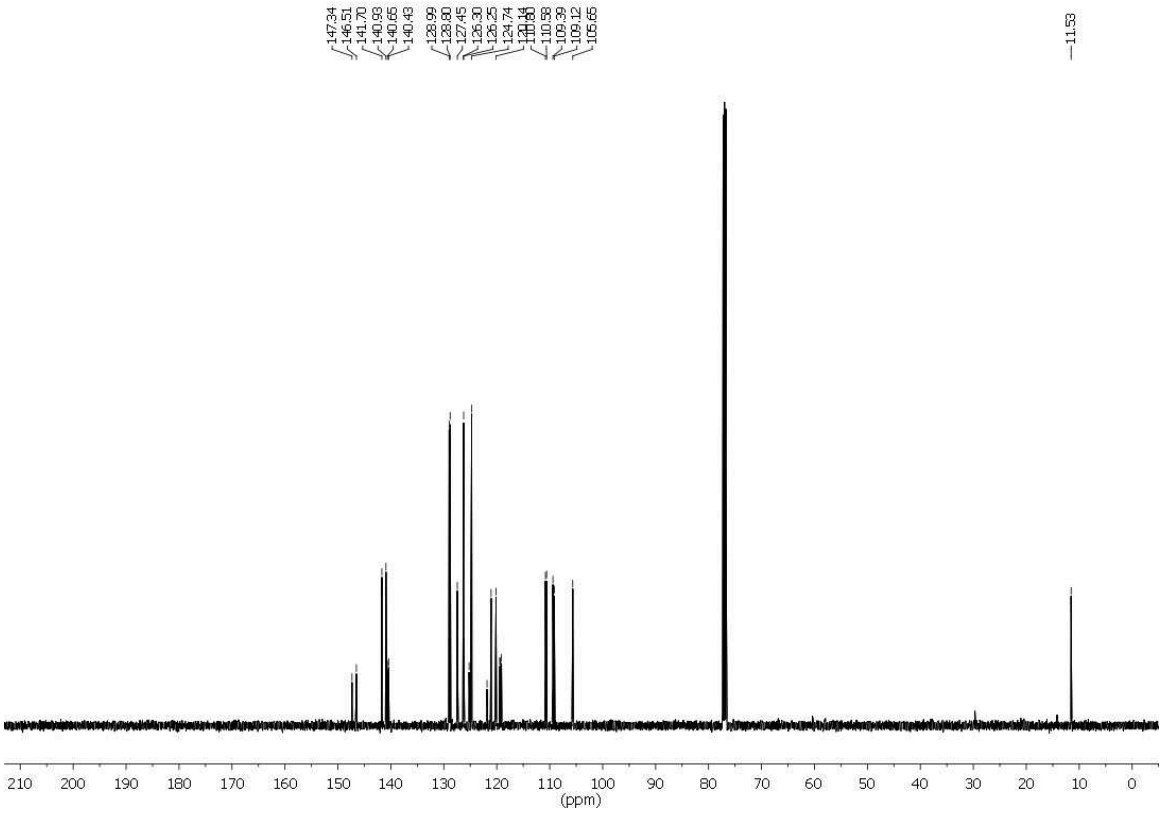
Compound 35d (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))





Compound 35e (^{1}H-NMR (300 MHz, $CDCl_3$), ^{13}C-NMR (125 MHz, $CDCl_3$))





X-ray diffraction

For the X-ray crystal structures of **25aa** and **26da** a single crystal was mounted with inert oil on a MiTeGen-Loop. The data was collected from the shock-cooled crystals at 100 K. The data for **25aa** was collected on a Bruker TXS-Mo rotating anode source with mirror optics and MoK_α radiation, $\lambda = 0.71073$ Å. The data for **26da** was collected on a Bruker Smart-6000 with Bruker SMART-Cu rotating anode and mirror optics. Data reduction was done with SAINT,[1] and an empirical absorption correction with SADABS[2] was applied. The structures were solved by direct methods (SHELXS-97)[3] and refined by full-matrix least-squares methods against F^2 (SHELXL-97 and ShelXle).[3,4] All non-hydrogen atoms were refined with anisotropic displacement parameters. The hydrogen atoms were refined isotropically on calculated positions using a riding model with their U_{iso} values constrained to 1.5 times the U_{eq} of their pivot atoms for terminal sp^3 carbon atoms and 1.2 times for all other carbon atoms. Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre. The CCDC numbers, crystal data and experimental details for the X-ray measurements are listed in the supporting information. CCDC 849623, 849627 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via `www.ccdc.cam.ac.uk/data_request/cif`.

Compound 25aa

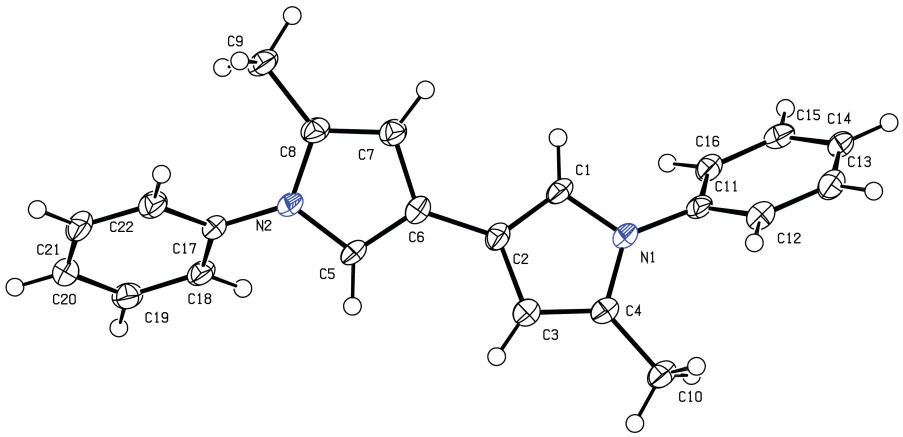


Table 0.1: Crystal data and structure refinement of **25aa**

CCDC number	849623
Empirical formula	$C_{22}H_{20}N_2$

Formula weight	312.40	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 9.9199(12) Å	α = 90°.
	b = 9.5808(11) Å	β = 105.506(3)°.
	c = 18.108(2) Å	γ = 90°.
Volume	1658.3(3) Å^3	
Z	4	
Density (calculated)	1.251 Mg/m^3	
Absorption coefficient	0.073 mm^{-1}	
F(000)	664	
Crystal size	0.22 x 0.20 x 0.01 mm^3	
Theta range for data collection	2.14 to 25.35°.	
Index ranges	-11<=h<=11, -11<=k<=11, -21<=l<=21	
Reflections collected	15338	
Independent reflections	3033 [R_{int} = 0.0545]	
Completeness to theta = 25.35°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.0000 and 0.5583	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3033 / 0 / 219	
Goodness-of-fit on F^2	1.094	
Final R indices [I>2sigma(I)]	R_1 = 0.0517, wR_2 = 0.1384	
R indices (all data)	R_1 = 0.0643, wR_2 = 0.1470	
Largest diff. peak and hole	0.267 and -0.301 e·Å^{-3}	

Table 0.2: Bond lengths [Å] of **25aa**

Atoms	Bond length [Å]	Atoms	Bond length [Å]
N(1)-C(4)	1.382(2)	C(10)-H(10C)	0.9800
N(1)-C(1)	1.385(2)	C(11)-C(12)	1.387(3)
N(1)-C(11)	1.421(2)	C(11)-C(16)	1.390(3)
N(2)-C(8)	1.386(2)	C(12)-C(13)	1.384(3)

N(2)-C(5)	1.388(2)	C(12)-H(12)	0.9500
N(2)-C(17)	1.416(2)	C(13)-C(14)	1.386(3)
C(1)-C(2)	1.363(3)	C(13)-H(13)	0.9500
C(1)-H(1)	0.9500	C(14)-C(15)	1.383(3)
C(2)-C(3)	1.431(3)	C(14)-H(14)	0.9500
C(2)-C(6)	1.466(3)	C(15)-C(16)	1.377(3)
C(3)-C(4)	1.369(3)	C(15)-H(15)	0.9500
C(3)-H(3)	0.9500	C(16)-H(16)	0.9500
C(4)-C(10)	1.493(3)	C(17)-C(22)	1.392(3)
C(5)-C(6)	1.364(3)	C(17)-C(18)	1.393(3)
C(5)-H(5)	0.9500	C(18)-C(19)	1.381(3)
C(6)-C(7)	1.425(3)	C(18)-H(18)	0.9500
C(7)-C(8)	1.359(3)	C(19)-C(20)	1.391(3)
C(7)-H(7)	0.9500	C(19)-H(19)	0.9500
C(8)-C(9)	1.490(3)	C(20)-C(21)	1.387(3)
C(9)-H(9A)	0.9800	C(20)-H(20)	0.9500
C(9)-H(9B)	0.9800	C(21)-C(22)	1.380(3)
C(9)-H(9C)	0.9800	C(21)-H(21)	0.9500
C(10)-H(10A)	0.9800	C(22)-H(22)	0.9500
C(10)-H(10B)	0.9800		

Table 0.3: Bond angles [°] of **25aa**

Atoms	Bond angle [°]	Atoms	Bond angle [°]
C(4)-N(1)-C(1)	108.73(15)	C(4)-C(10)-H(10C)	109.5
C(4)-N(1)-C(11)	128.24(15)	H(10A)-C(10)-H(10C)	109.5
C(1)-N(1)-C(11)	122.81(16)	H(10B)-C(10)-H(10C)	109.5
C(8)-N(2)-C(5)	108.45(16)	C(12)-C(11)-C(16)	120.00(18)
C(8)-N(2)-C(17)	126.95(16)	C(12)-C(11)-N(1)	121.11(17)
C(5)-N(2)-C(17)	124.25(15)	C(16)-C(11)-N(1)	118.87(17)
C(2)-C(1)-N(1)	108.80(17)	C(13)-C(12)-C(11)	119.55(18)
C(2)-C(1)-H(1)	125.6	C(13)-C(12)-H(12)	120.2
N(1)-C(1)-H(1)	125.6	C(11)-C(12)-H(12)	120.2
C(1)-C(2)-C(3)	106.64(16)	C(12)-C(13)-C(14)	120.42(18)
C(1)-C(2)-C(6)	124.59(17)	C(12)-C(13)-H(13)	119.8

C(3)-C(2)-C(6)	128.74(18)	C(14)-C(13)-H(13)	119.8
C(4)-C(3)-C(2)	108.17(17)	C(15)-C(14)-C(13)	119.72(19)
C(4)-C(3)-H(3)	125.9	C(15)-C(14)-H(14)	120.1
C(2)-C(3)-H(3)	125.9	C(13)-C(14)-H(14)	120.1
C(3)-C(4)-N(1)	107.66(16)	C(16)-C(15)-C(14)	120.30(19)
C(3)-C(4)-C(10)	129.87(18)	C(16)-C(15)-H(15)	119.9
N(1)-C(4)-C(10)	122.41(17)	C(14)-C(15)-H(15)	119.9
C(6)-C(5)-N(2)	108.63(16)	C(15)-C(16)-C(11)	119.98(18)
C(6)-C(5)-H(5)	125.7	C(15)-C(16)-H(16)	120.0
N(2)-C(5)-H(5)	125.7	C(11)-C(16)-H(16)	120.0
C(5)-C(6)-C(7)	106.60(16)	C(22)-C(17)-C(18)	119.59(18)
C(5)-C(6)-C(2)	127.32(17)	C(22)-C(17)-N(2)	120.63(17)
C(7)-C(6)-C(2)	126.00(18)	C(18)-C(17)-N(2)	119.78(17)
C(8)-C(7)-C(6)	108.70(17)	C(19)-C(18)-C(17)	120.26(18)
C(8)-C(7)-H(7)	125.7	C(19)-C(18)-H(18)	119.9
C(6)-C(7)-H(7)	125.7	C(17)-C(18)-H(18)	119.9
C(7)-C(8)-N(2)	107.61(17)	C(18)-C(19)-C(20)	120.23(19)
C(7)-C(8)-C(9)	128.97(19)	C(18)-C(19)-H(19)	119.9
N(2)-C(8)-C(9)	123.41(18)	C(20)-C(19)-H(19)	119.9
C(8)-C(9)-H(9A)	109.5	C(21)-C(20)-C(19)	119.21(19)
C(8)-C(9)-H(9B)	109.5	C(21)-C(20)-H(20)	120.4
H(9A)-C(9)-H(9B)	109.5	C(19)-C(20)-H(20)	120.4
C(8)-C(9)-H(9C)	109.5	C(22)-C(21)-C(20)	121.02(19)
H(9A)-C(9)-H(9C)	109.5	C(22)-C(21)-H(21)	119.5
H(9B)-C(9)-H(9C)	109.5	C(20)-C(21)-H(21)	119.5
C(4)-C(10)-H(10A)	109.5	C(21)-C(22)-C(17)	119.66(18)
C(4)-C(10)-H(10B)	109.5	C(21)-C(22)-H(22)	120.2
H(10A)-C(10)-H(10B)	109.5	C(17)-C(22)-H(22)	120.2

Table 0.4: Torsion angles [°] of **25aa**

Atoms	Torsion angle [°]	Atoms	Torsion angle [°]
C(4)-N(1)-C(1)-C(2)	0.2(2)	C(5)-N(2)-C(8)-C(9)	178.68(18)
C(11)-N(1)-C(1)-C(2)	175.16(16)	C(17)-N(2)-C(8)-C(9)	5.3(3)
N(1)-C(1)-C(2)-C(3)	0.0(2)	C(4)-N(1)-C(11)-C(12)	-58.3(3)

N(1)-C(1)-C(2)-C(6)	-178.31(16)	C(1)-N(1)-C(11)-C(12)	127.8(2)
C(1)-C(2)-C(3)-C(4)	-0.1(2)	C(4)-N(1)-C(11)-C(16)	122.9(2)
C(6)-C(2)-C(3)-C(4)	178.04(18)	C(1)-N(1)-C(11)-C(16)	-51.0(2)
C(2)-C(3)-C(4)-N(1)	0.3(2)	C(16)-C(11)-C(12)-C(13)	0.7(3)
C(2)-C(3)-C(4)-C(10)	-176.81(19)	N(1)-C(11)-C(12)-C(13)	-178.07(17)
C(1)-N(1)-C(4)-C(3)	-0.3(2)	C(11)-C(12)-C(13)-C(14)	0.7(3)
C(11)-N(1)-C(4)-C(3)	-174.89(17)	C(12)-C(13)-C(14)-C(15)	-1.0(3)
C(1)-N(1)-C(4)-C(10)	177.06(17)	C(13)-C(14)-C(15)-C(16)	-0.2(3)
C(11)-N(1)-C(4)-C(10)	2.5(3)	C(14)-C(15)-C(16)-C(11)	1.6(3)
C(8)-N(2)-C(5)-C(6)	0.7(2)	C(12)-C(11)-C(16)-C(15)	-1.8(3)
C(17)-N(2)-C(5)-C(6)	174.22(16)	N(1)-C(11)-C(16)-C(15)	176.98(17)
N(2)-C(5)-C(6)-C(7)	-0.7(2)	C(8)-N(2)-C(17)-C(22)	-51.0(3)
N(2)-C(5)-C(6)-C(2)	-177.43(17)	C(5)-N(2)-C(17)-C(22)	136.66(19)
C(1)-C(2)-C(6)-C(5)	154.1(2)	C(8)-N(2)-C(17)-C(18)	128.4(2)
C(3)-C(2)-C(6)-C(5)	-23.8(3)	C(5)-N(2)-C(17)-C(18)	-44.0(3)
C(1)-C(2)-C(6)-C(7)	-22.0(3)	C(22)-C(17)-C(18)-C(19)	-0.6(3)
C(3)-C(2)-C(6)-C(7)	160.08(19)	N(2)-C(17)-C(18)-C(19)	-179.94(17)
C(5)-C(6)-C(7)-C(8)	0.5(2)	C(17)-C(18)-C(19)-C(20)	1.5(3)
C(2)-C(6)-C(7)-C(8)	177.30(18)	C(18)-C(19)-C(20)-C(21)	-0.9(3)
C(6)-C(7)-C(8)-N(2)	-0.1(2)	C(19)-C(20)-C(21)-C(22)	-0.6(3)
C(6)-C(7)-C(8)-C(9)	-179.05(19)	C(20)-C(21)-C(22)-C(17)	1.6(3)
C(5)-N(2)-C(8)-C(7)	-0.3(2)	C(18)-C(17)-C(22)-C(21)	-0.9(3)
C(17)-N(2)-C(8)-C(7)	-173.67(17)	N(2)-C(17)-C(22)-C(21)	178.41(17)

Compound 26da

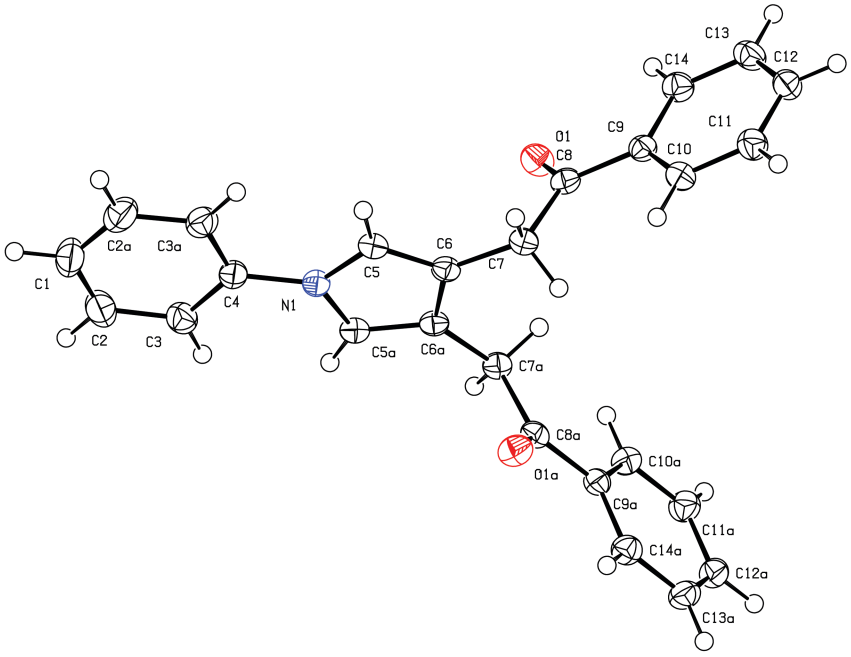


Table 0.5: Crystal data and structure refinement of **26da**

CCDC number	849627	
Empirical formula	$C_{26}H_{21}NO_2$	
Formula weight	379.44	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	$C2/c$	
Unit cell dimensions	a = 17.313(3) Å	$\alpha = 90°$.
	b = 12.727(2) Å	$\beta = 93.106(8)°$.
	c = 8.9581(15) Å	$\gamma = 90°$.
Volume	1970.9(6) $Å^3$	
Z	4	
Density (calculated)	1.279 Mg/m^3	
Absorption coefficient	0.636 mm^{-1}	
$F(000)$	800	
Crystal size	0.050 x 0.040 x 0.020 mm^3	
Theta range for data collection	4.31 to 68.37°.	

Index ranges	-20<=h<=19, -14<=k<=14, -10<=l<=10
Reflections collected	16522
Independent reflections	1777 [R_{int} = 0.0289]
Completeness to theta = 68.37°	97.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9871 and 0.8777
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1777 / 0 / 133
Goodness-of-fit on F^2	1.074
Final R indices [I>2sigma(I)]	R_1 = 0.0297, wR_2 = 0.0761
R indices (all data)	$R1$ = 0.0326, wR_2 = 0.0790
Largest diff. peak and hole	0.180 and -0.167 e·Å^{-3}

Table 0.6: Bond lengths [Å] of **26da**

Atoms	Bond length [Å]	Atoms	Bond length [Å]
N(1)-C(5)#1	1.3853(13)	C(7)-C(8)	1.5258(15)
N(1)-C(5)	1.3854(13)	C(7)-H(7A)	0.9900
N(1)-C(4)	1.4184(19)	C(7)-H(7B)	0.9900
O(1)-C(8)	1.2205(13)	C(8)-C(9)	1.4937(15)
C(1)-C(2)	1.3805(16)	C(9)-C(10)	1.3944(15)
C(1)-C(2)#1	1.3805(16)	C(9)-C(14)	1.3999(15)
C(1)-H(1)	0.9500	C(10)-C(11)	1.3856(16)
C(2)-C(3)	1.3866(17)	C(10)-H(10)	0.9500
C(2)-H(2)	0.9500	C(11)-C(12)	1.3853(16)
C(3)-C(4)	1.3938(14)	C(11)-H(11)	0.9500
C(3)-H(3)	0.9500	C(12)-C(13)	1.3848(16)
C(4)-C(3)#1	1.3939(14)	C(12)-H(12)	0.9500
C(5)-C(6)	1.3613(15)	C(13)-C(14)	1.3818(16)
C(5)-H(5)	0.9500	C(13)-H(13)	0.9500
C(6)-C(6)#1	1.426(2)	C(14)-H(14)	0.9500
C(6)-C(7)	1.4939(14)		

Symmetry transformations used to generate equivalent atoms:
#1 -x,y,-z+1/2

Table 0.7: Bond angles [°] of **26da**

Atoms	Bond angle [°]	Atoms	Bond angle [°]
C(5)#1-N(1)-C(5)	107.83(12)	C(6)-C(7)-H(7B)	108.7
C(5)#1-N(1)-C(4)	126.08(6)	C(8)-C(7)-H(7B)	108.7
C(5)-N(1)-C(4)	126.08(6)	H(7A)-C(7)-H(7B)	107.6
C(2)-C(1)-C(2)#1	119.27(16)	O(1)-C(8)-C(9)	120.50(9)
C(2)-C(1)-H(1)	120.4	O(1)-C(8)-C(7)	121.41(10)
C(2)#1-C(1)-H(1)	120.4	C(9)-C(8)-C(7)	118.09(9)
C(1)-C(2)-C(3)	120.84(12)	C(10)-C(9)-C(14)	118.83(10)
C(1)-C(2)-H(2)	119.6	C(10)-C(9)-C(8)	122.27(9)
C(3)-C(2)-H(2)	119.6	C(14)-C(9)-C(8)	118.90(9)
C(2)-C(3)-C(4)	119.88(12)	C(11)-C(10)-C(9)	120.53(10)
C(2)-C(3)-H(3)	120.1	C(11)-C(10)-H(10)	119.7
C(4)-C(3)-H(3)	120.1	C(9)-C(10)-H(10)	119.7
C(3)-C(4)-C(3)#1	119.28(15)	C(12)-C(11)-C(10)	119.97(10)
C(3)-C(4)-N(1)	120.36(7)	C(12)-C(11)-H(11)	120.0
C(3)#1-C(4)-N(1)	120.36(7)	C(10)-C(11)-H(11)	120.0
C(6)-C(5)-N(1)	108.71(9)	C(13)-C(12)-C(11)	120.10(10)
C(6)-C(5)-H(5)	125.6	C(13)-C(12)-H(12)	120.0
N(1)-C(5)-H(5)	125.6	C(11)-C(12)-H(12)	120.0
C(5)-C(6)-C(6)#1	107.37(6)	C(14)-C(13)-C(12)	120.14(10)
C(5)-C(6)-C(7)	126.33(9)	C(14)-C(13)-H(13)	119.9
C(6)#1-C(6)-C(7)	126.29(6)	C(12)-C(13)-H(13)	119.9
C(6)-C(7)-C(8)	114.15(9)	C(13)-C(14)-C(9)	120.42(10)
C(6)-C(7)-H(7A)	108.7	C(13)-C(14)-H(14)	119.8
C(8)-C(7)-H(7A)	108.7	C(9)-C(14)-H(14)	119.8

Symmetry transformations used to generate equivalent atoms:
#1 -x,y,-z+1/2

Table 0.8: Torsion angles [°] of **26da**

Atoms	Torsion angle [°]	Atoms	Torsion angle [°]
C(2)#1-C(1)-C(2)-C(3)	0.49(8)	C(6)-C(7)-C(8)-O(1)	-2.04(14)
C(1)-C(2)-C(3)-C(4)	-0.98(16)	C(6)-C(7)-C(8)-C(9)	178.36(9)

C(2)-C(3)-C(4)-C(3)#1	0.48(8)	O(1)-C(8)-C(9)-C(10)	-173.80(10)
C(2)-C(3)-C(4)-N(1)	-179.52(8)	C(7)-C(8)-C(9)-C(10)	5.81(15)
C(5)#1-N(1)-C(4)-C(3)	2.68(7)	O(1)-C(8)-C(9)-C(14)	6.71(15)
C(5)-N(1)-C(4)-C(3)	-177.32(7)	C(7)-C(8)-C(9)-C(14)	-173.68(9)
C(5)#1-N(1)-C(4)-C(3)#1	-177.31(7)	C(14)-C(9)-C(10)-C(11)	-0.87(16)
C(5)-N(1)-C(4)-C(3)#1	2.68(7)	C(8)-C(9)-C(10)-C(11)	179.64(10)
C(5)#1-N(1)-C(5)-C(6)	0.24(5)	C(9)-C(10)-C(11)-C(12)	0.08(17)
C(4)-N(1)-C(5)-C(6)	-179.76(5)	C(10)-C(11)-C(12)-C(13)	0.90(17)
N(1)-C(5)-C(6)-C(6)#1	-0.60(13)	C(11)-C(12)-C(13)-C(14)	-1.07(17)
N(1)-C(5)-C(6)-C(7)	178.41(8)	C(12)-C(13)-C(14)-C(9)	0.26(17)
C(5)-C(6)-C(7)-C(8)	-103.41(12)	C(10)-C(9)-C(14)-C(13)	0.70(16)
C(6)#1-C(6)-C(7)-C(8)	75.42(15)	C(8)-C(9)-C(14)-C(13)	-179.79(9)

Symmetry transformations used to generate equivalent atoms:
#1 -x,y,-z+1/2

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