Regioselectivity-tunable Self-1,3-dipolar [3+3] Cyclizations of Azomethine Ylides to Assemble Dispirooxindole-piperazines

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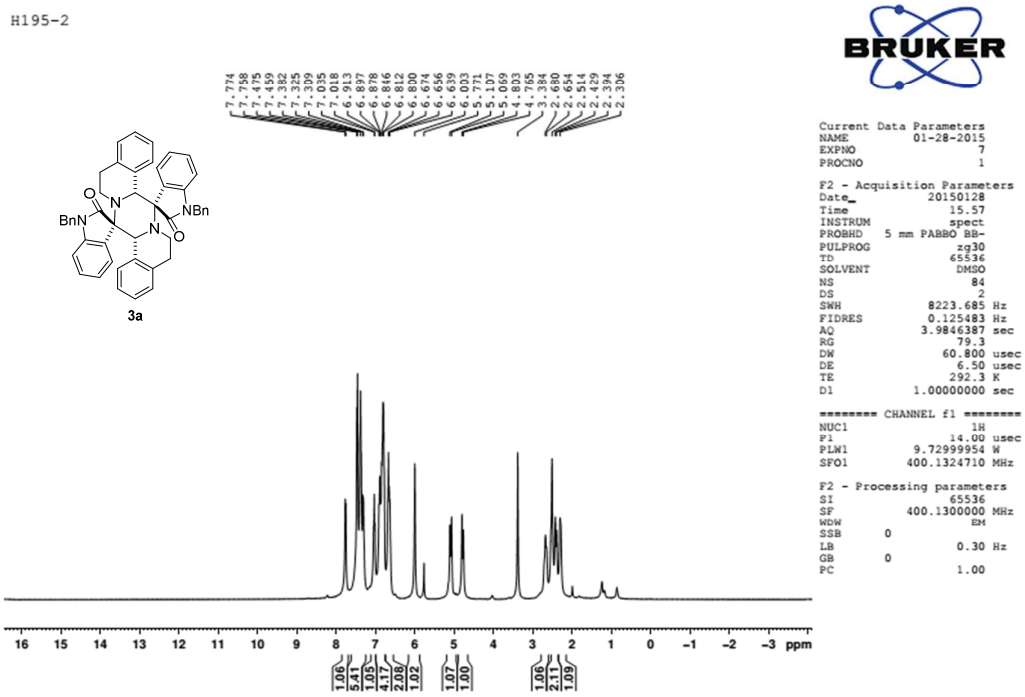
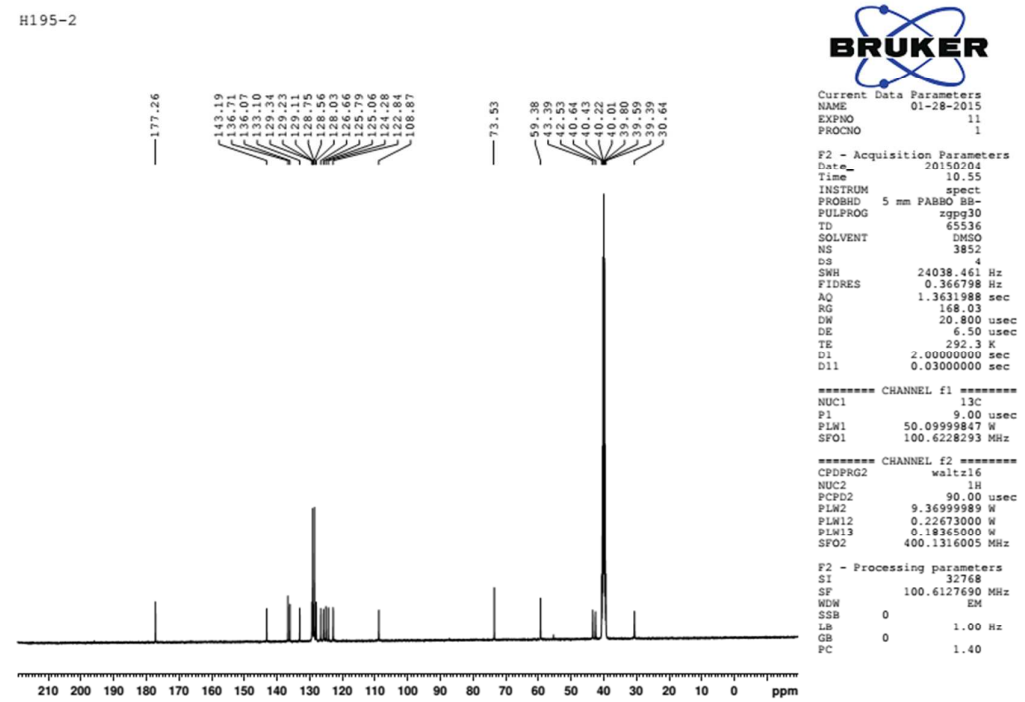
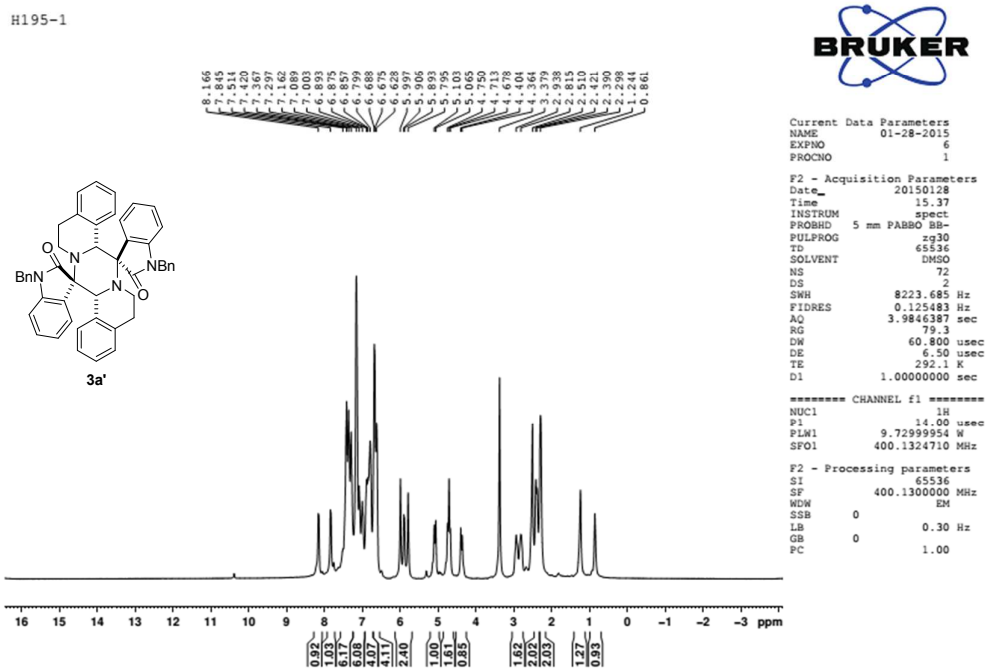
^{1}H NMR and ^{13}C NMR spectra for compounds **3a, 3a', 3b, 3b', 3c, 3c', 3d, 3d', 3e, 3e', 3f, 3f', 3g, 3h, 3i, 3i', 5a, 5b, 5c, 5d, 5e, 5f, 7a, 7b, 7c, 7d**; X-ray structure of **dispirooxindole—piperazine 3a, 3a'**, **7a**.

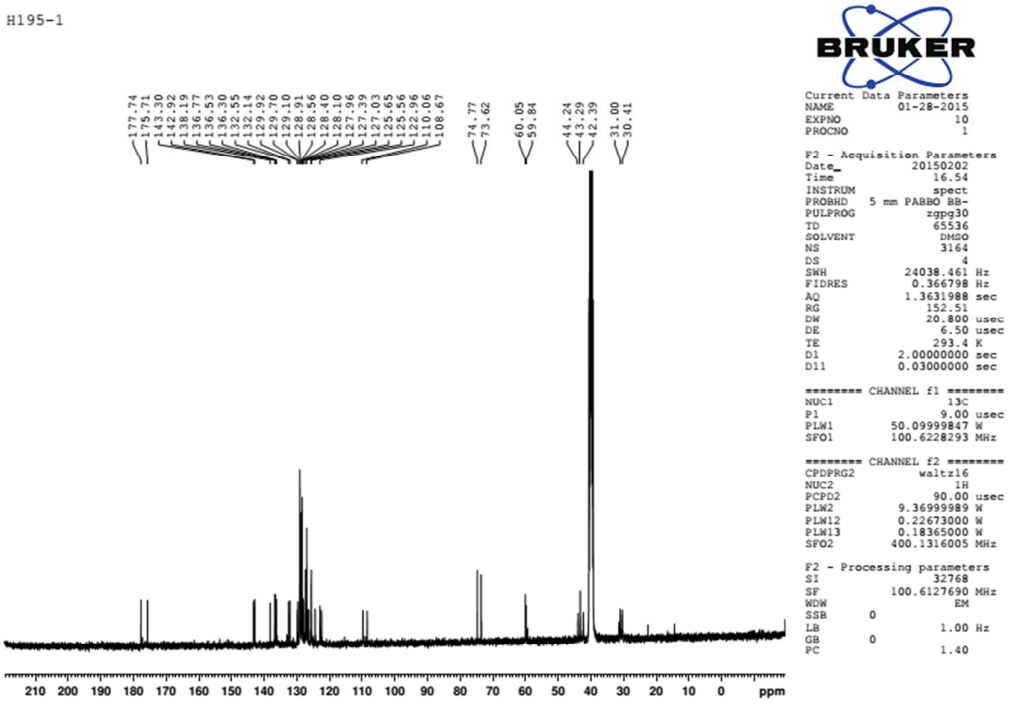
^{1}H NMR (400 MHz; DMSO-d_6), ^{13}C NMR (100 MHz; DMSO-d_6)





^{1}H NMR (400 MHz; DMSO-d_6), ^{13}C NMR (100 MHz; DMSO-d_6)





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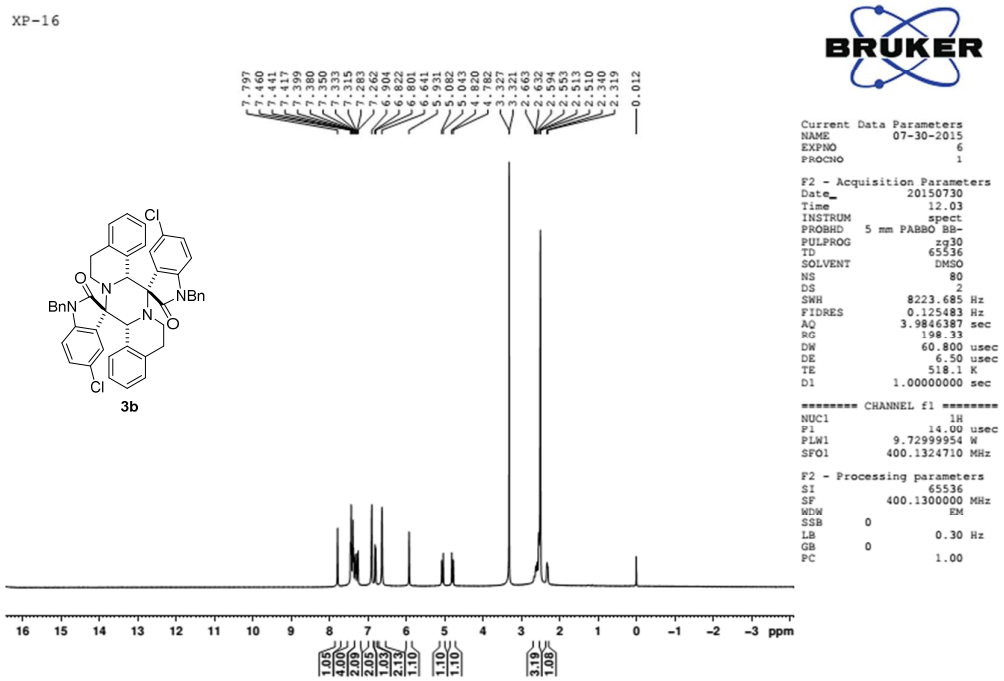
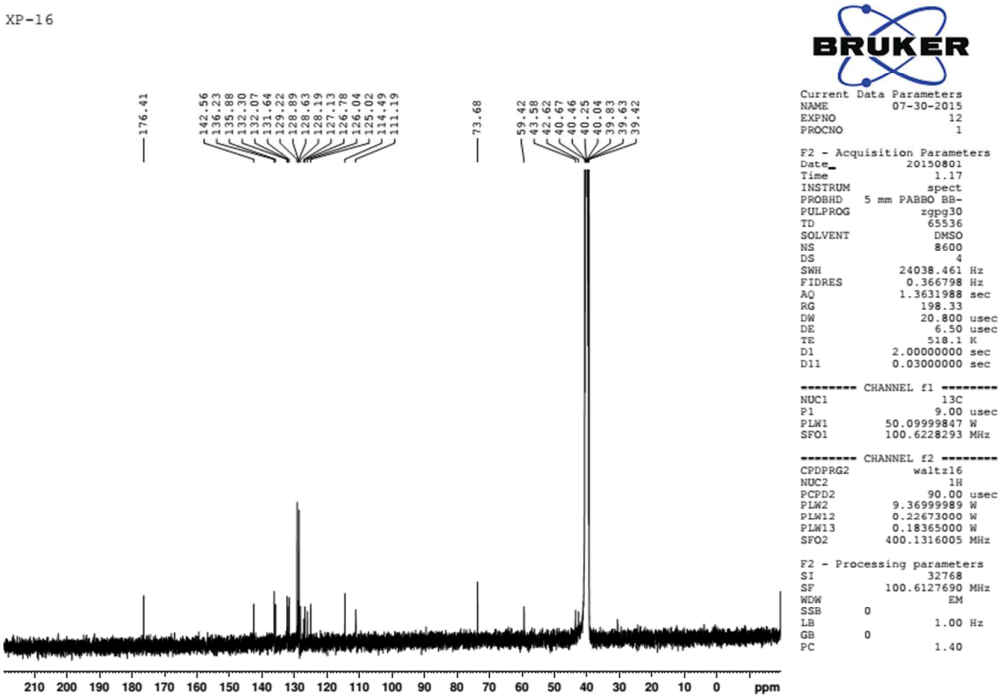
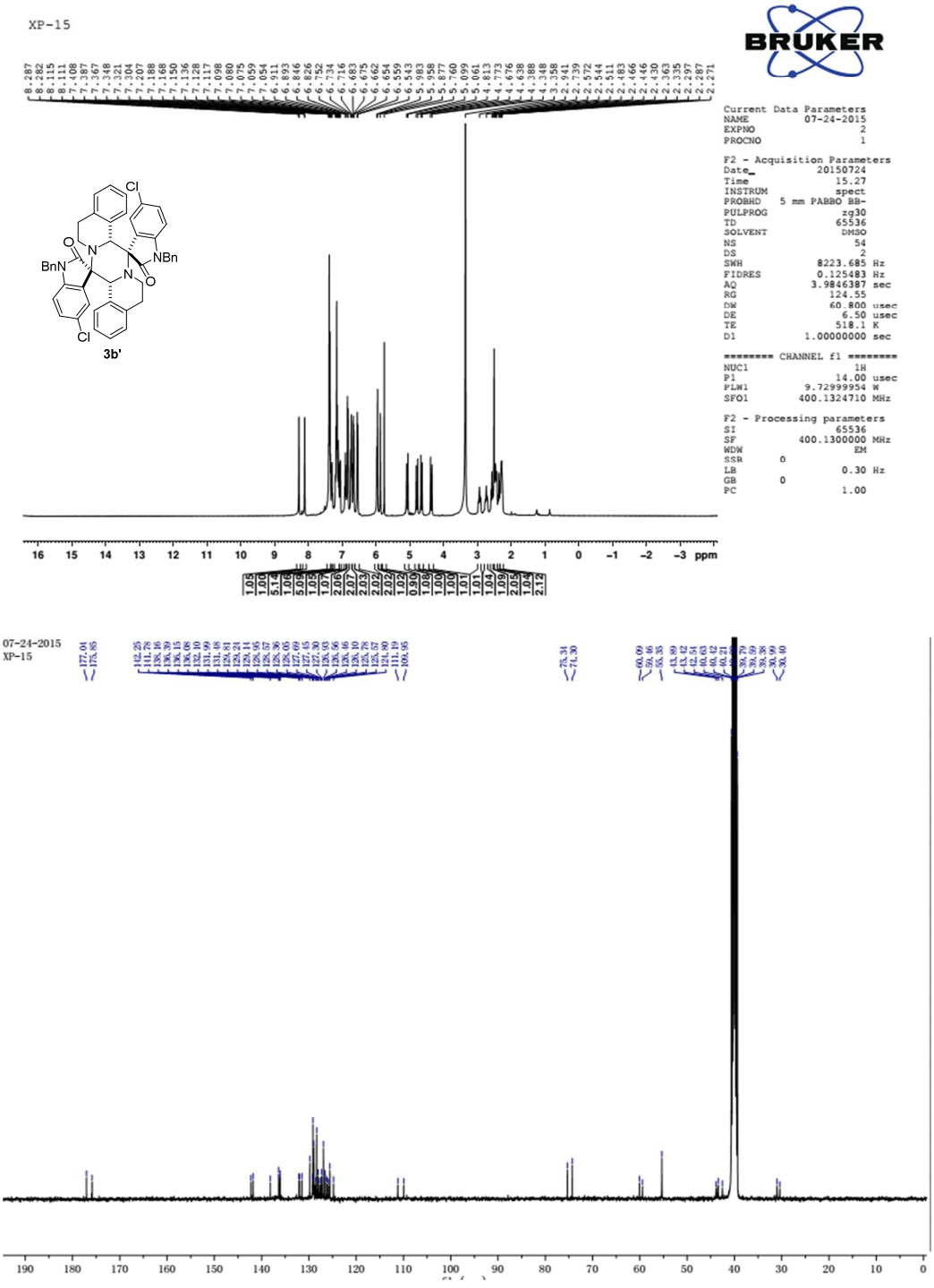
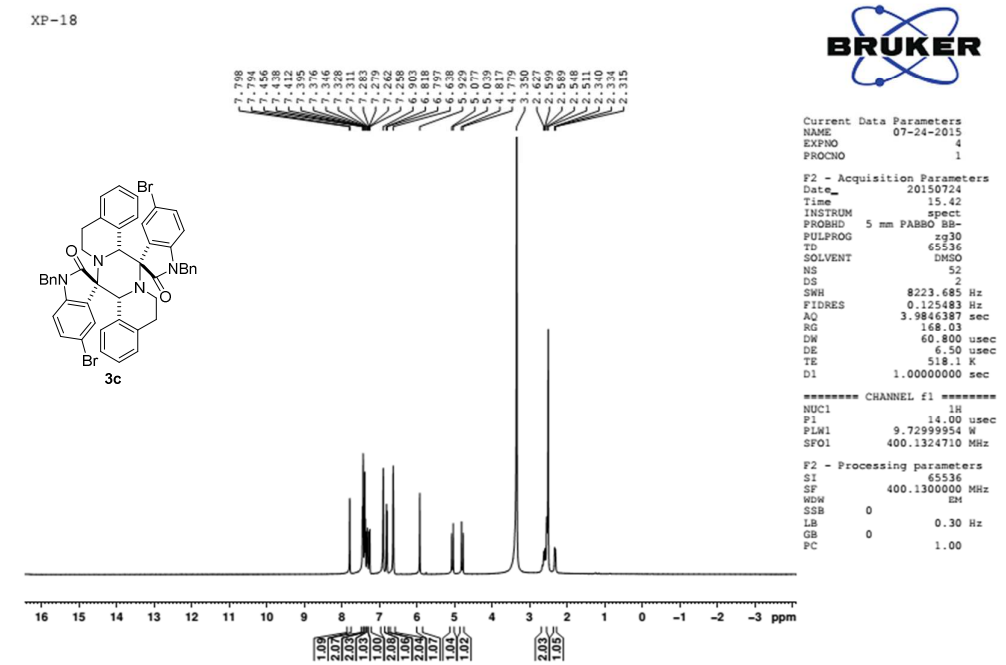


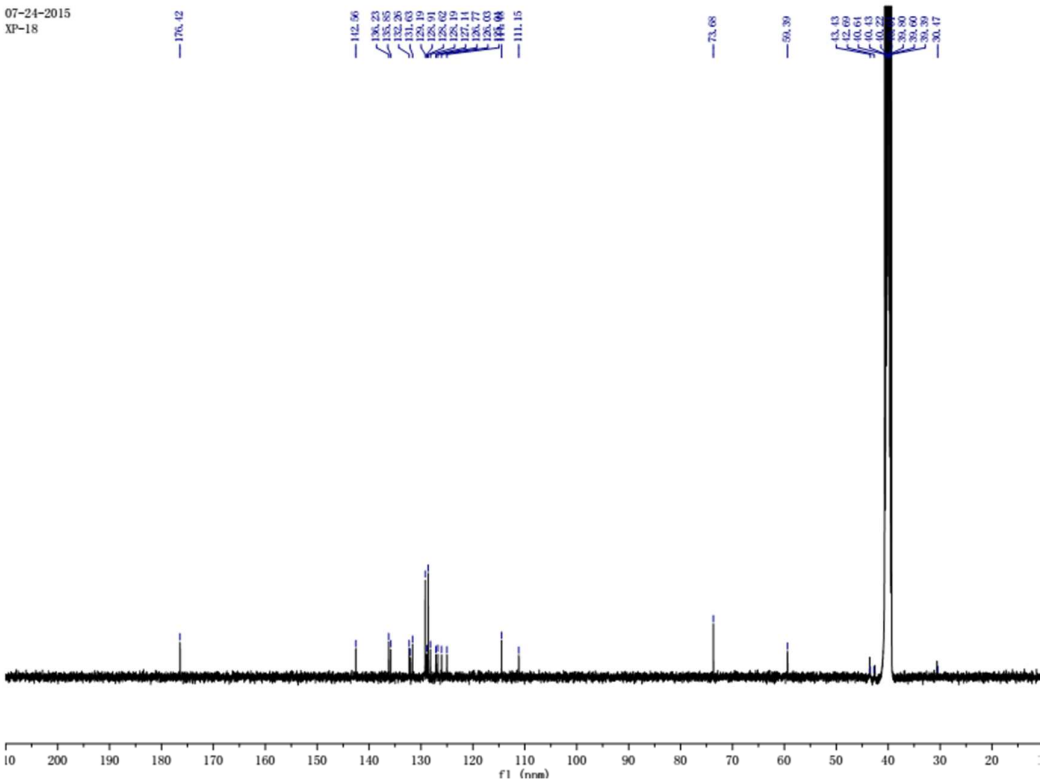


^{1}H NMR (400 MHz; DMSO-d_6), ^{13}C NMR (100 MHz; DMSO-d_6)

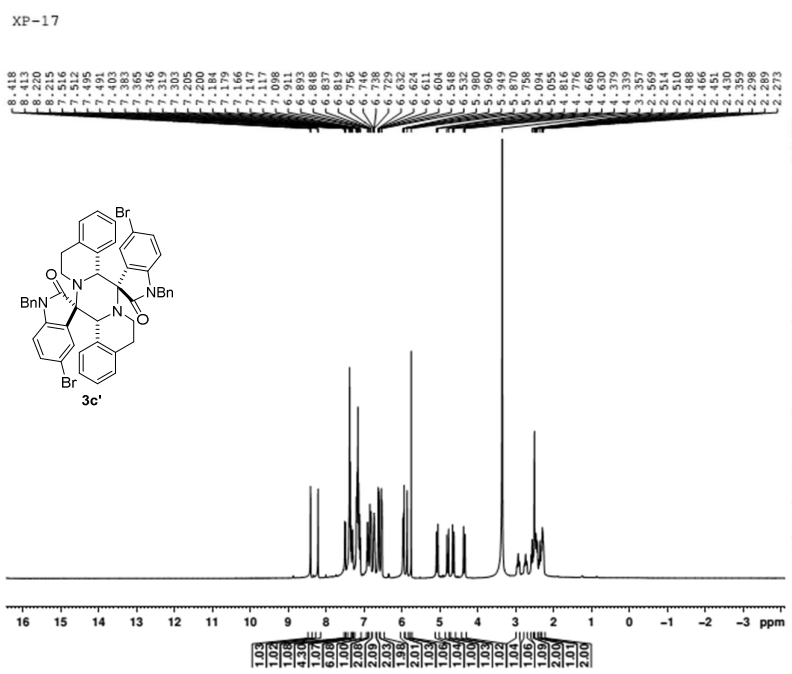


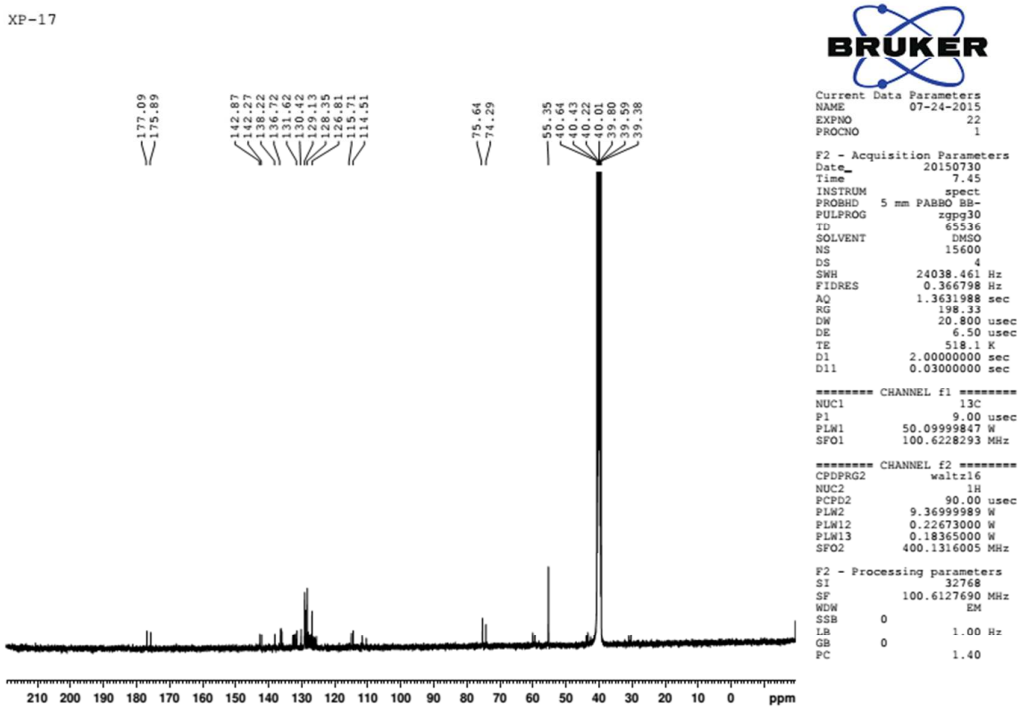
^{1}H NMR (400 MHz; DMSO-d_6), ^{13}C NMR (100 MHz; DMSO-d_6)



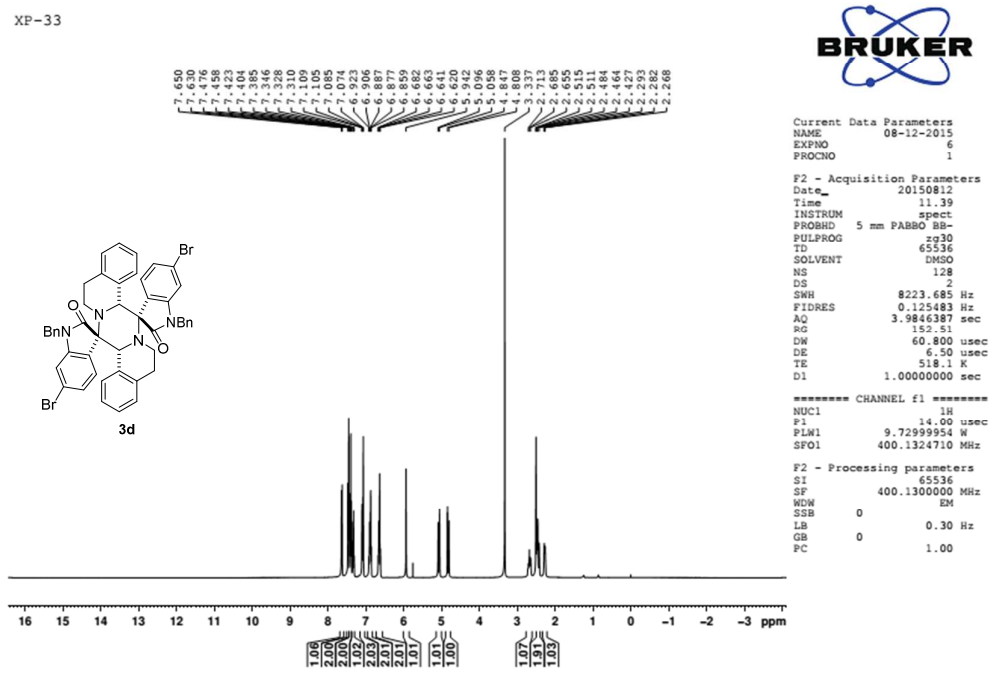


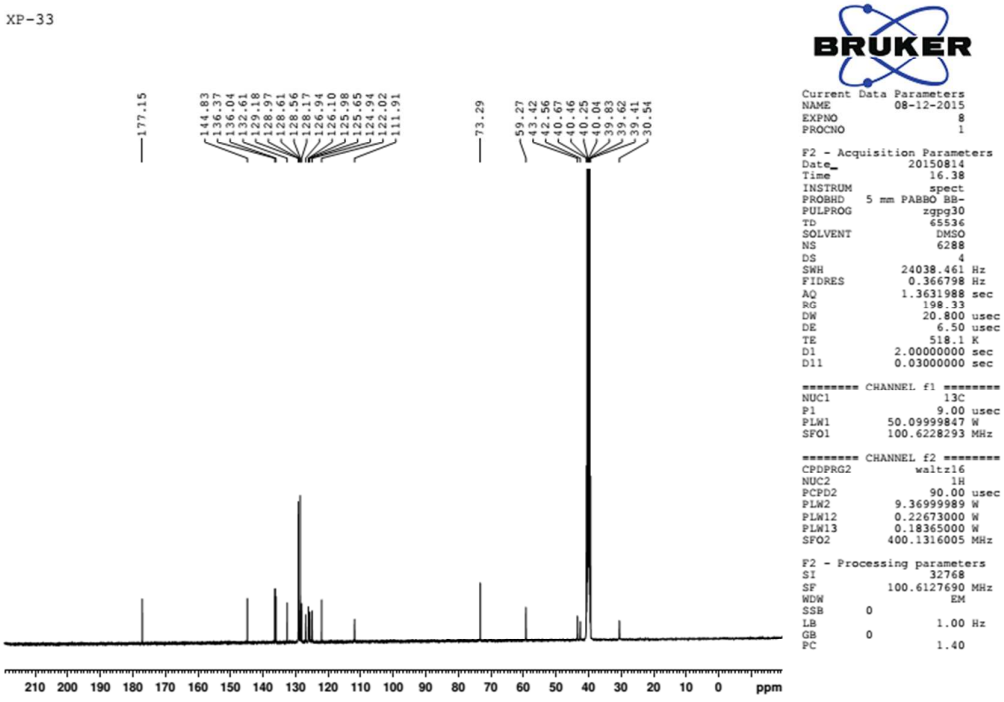
^{1}H NMR (400 MHz; DMSO-d_6), ^{13}C NMR (100 MHz; DMSO-d_6)



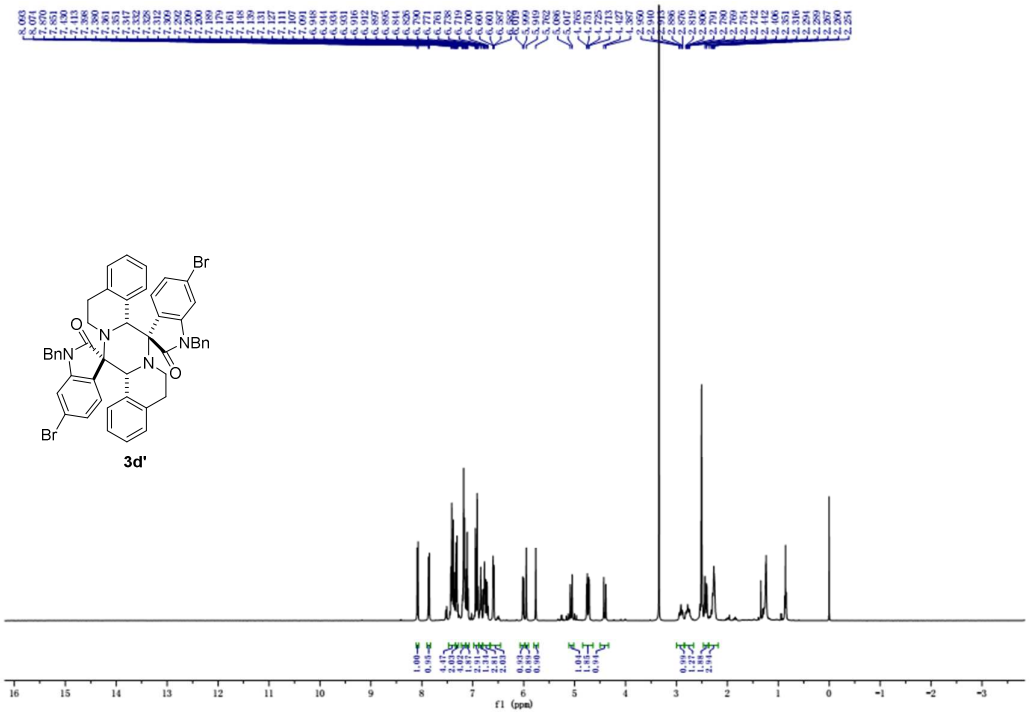


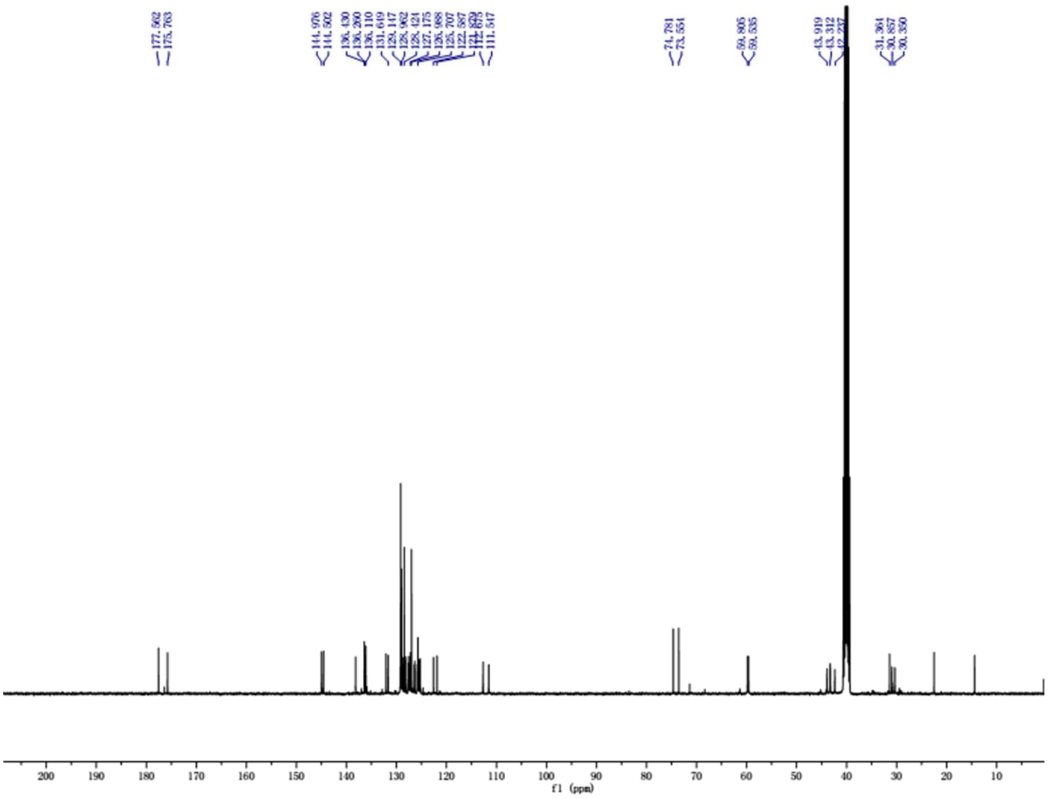
^{1}H NMR (400 MHz; DMSO-d_6), ^{13}C NMR (100 MHz; DMSO-d_6)



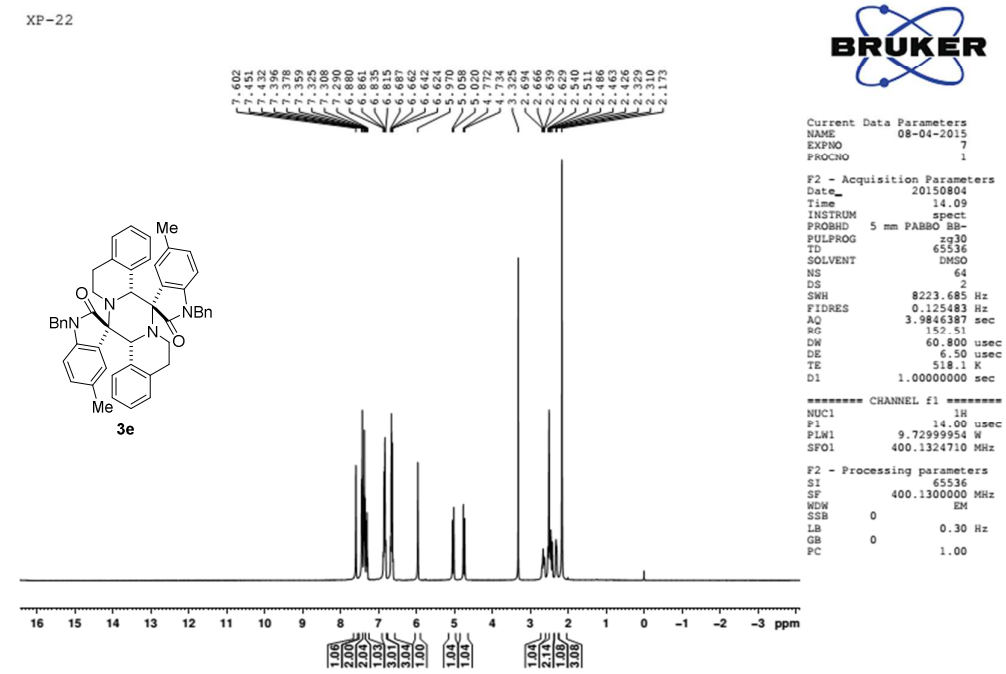


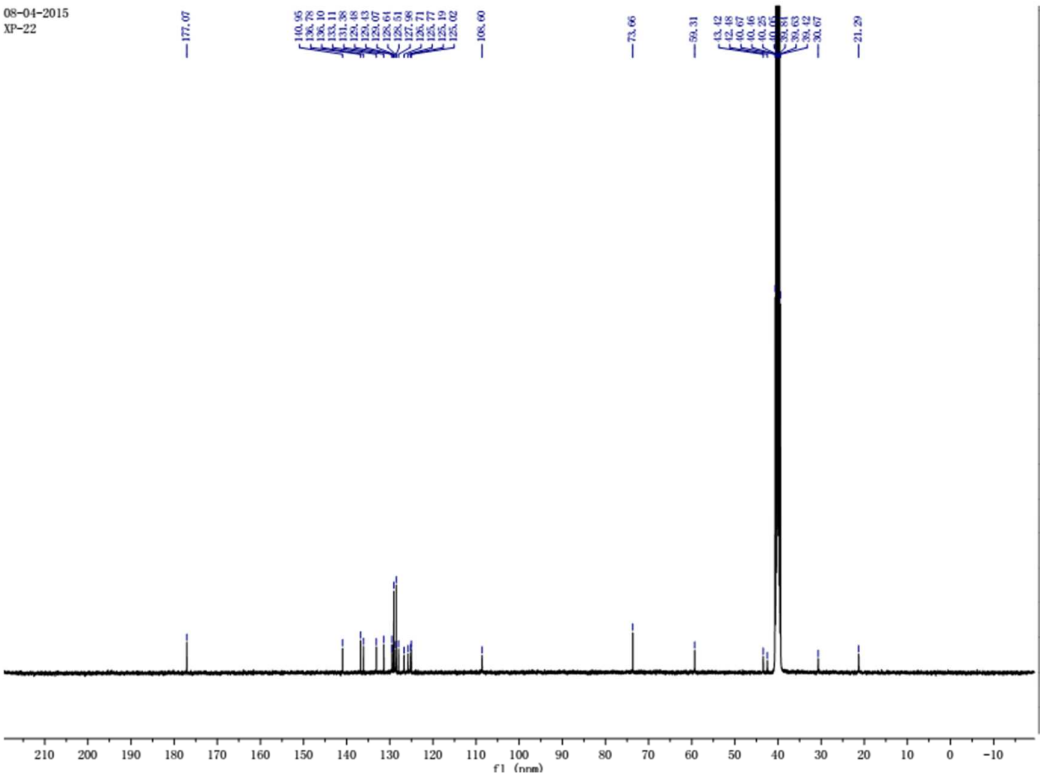
^{1}H NMR (400 MHz; DMSO-d_6), ^{13}C NMR (100 MHz; DMSO-d_6)





^{1}H NMR (400 MHz; DMSO-d_6), ^{13}C NMR (100 MHz; DMSO-d_6)





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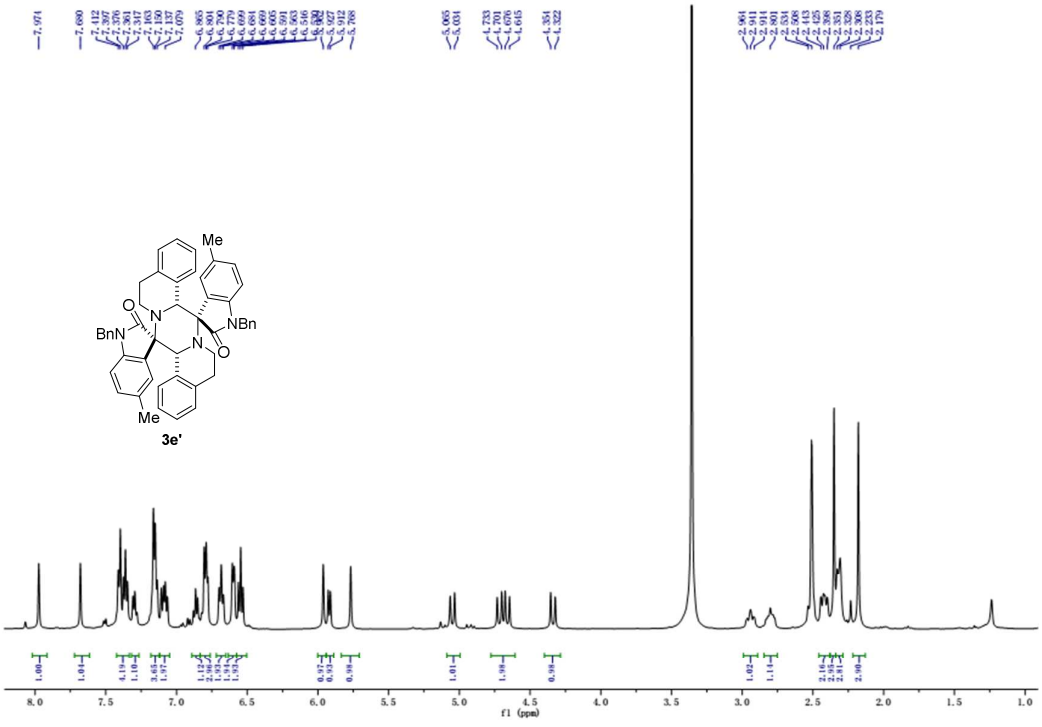
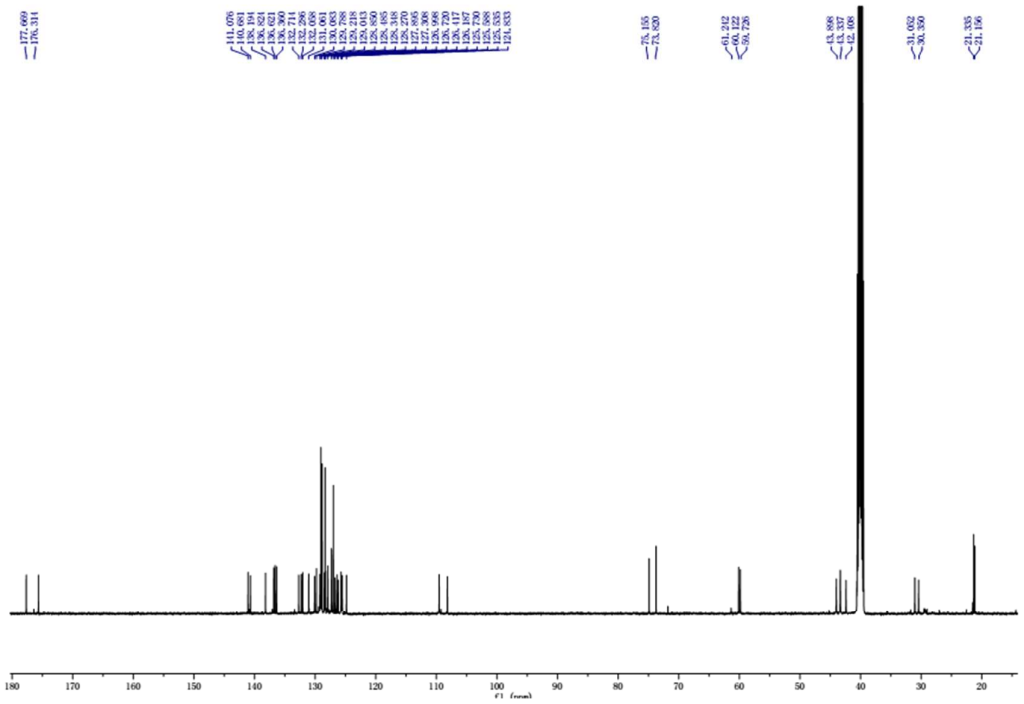
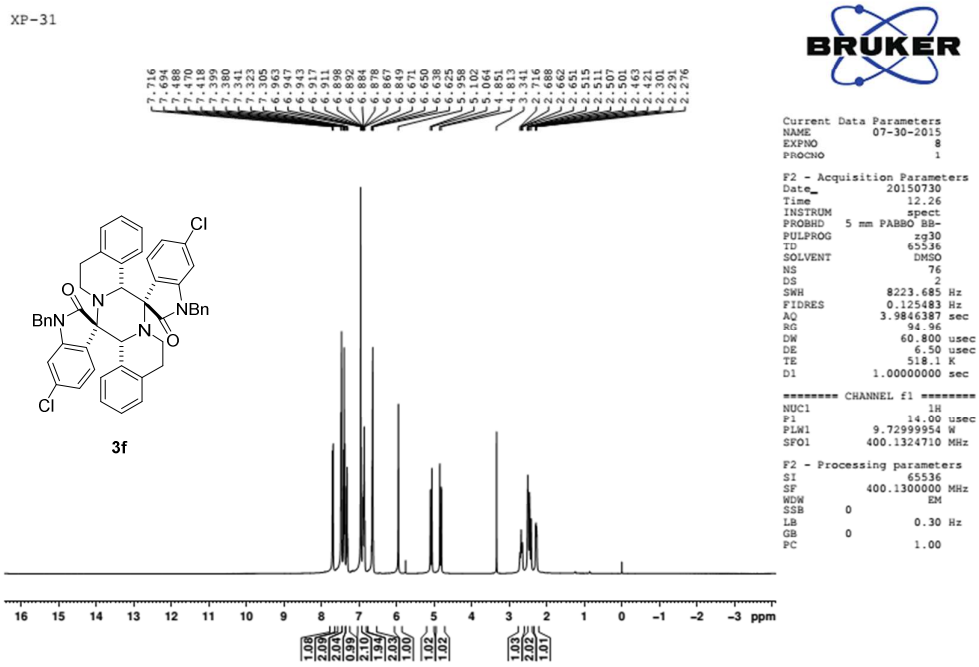
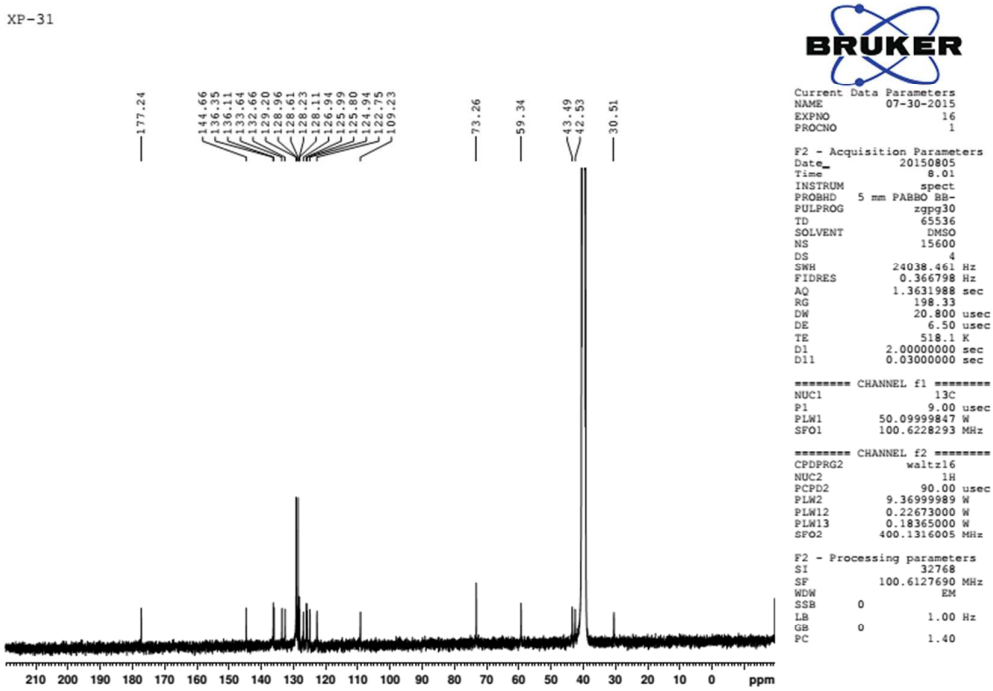




^{1}H NMR (400 MHz; DMSO-d_6), ^{13}C NMR (100 MHz; DMSO-d_6)





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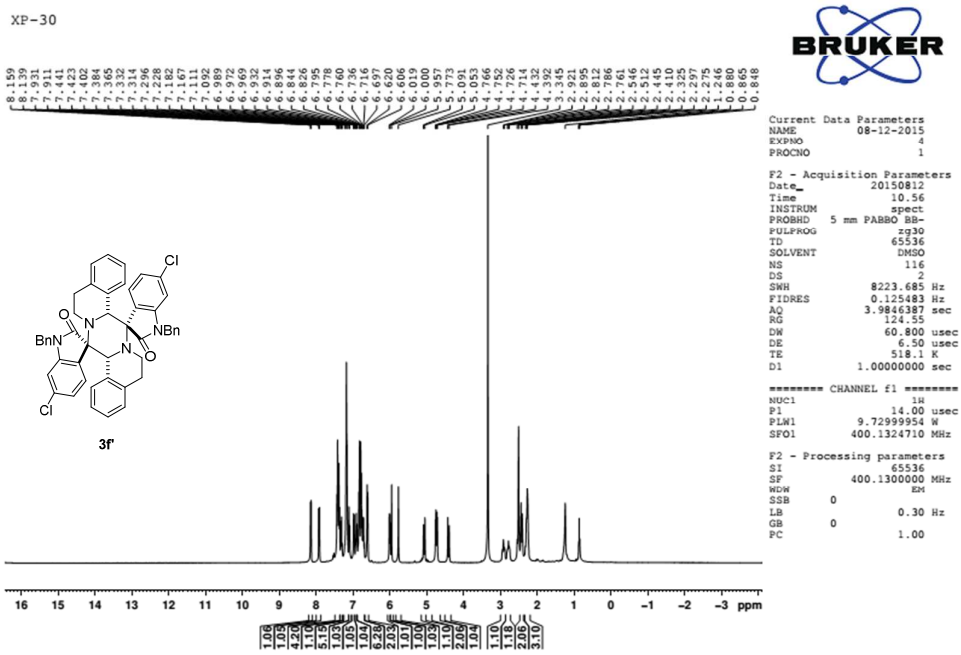
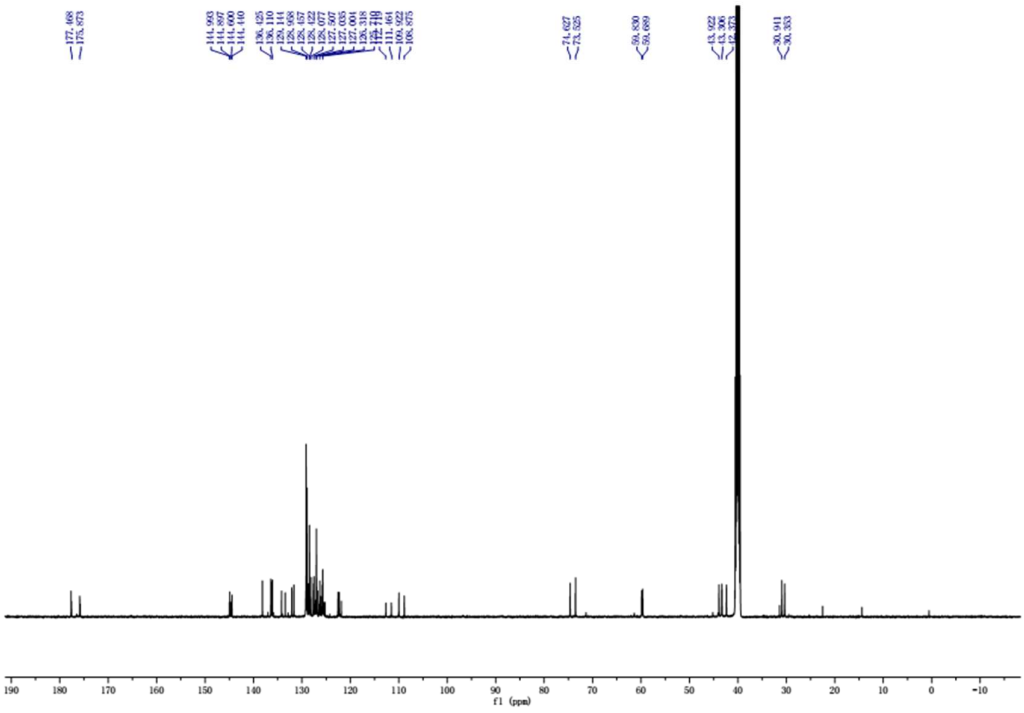
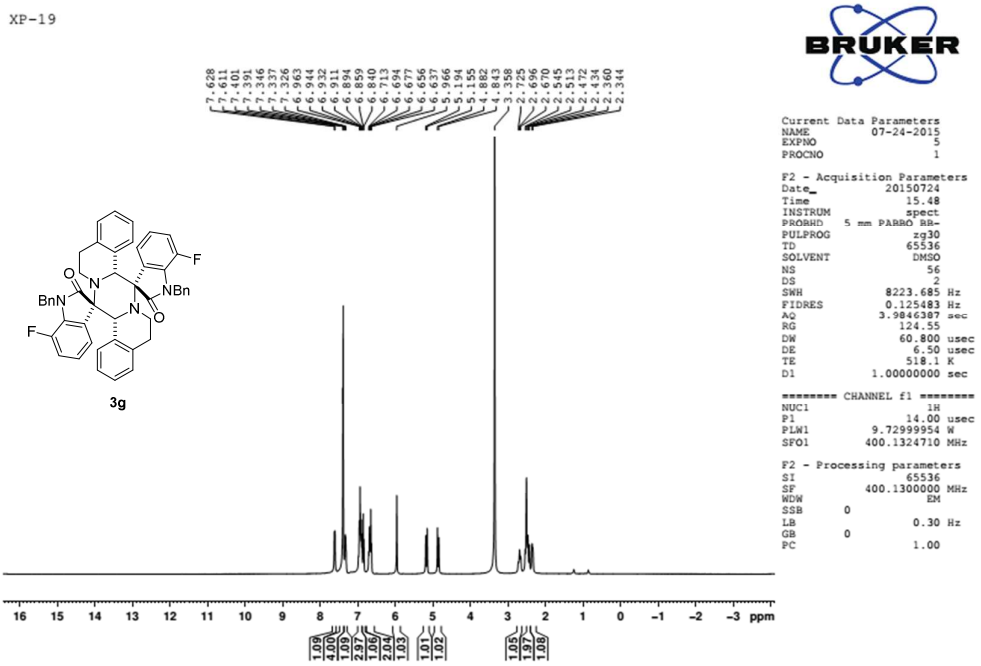
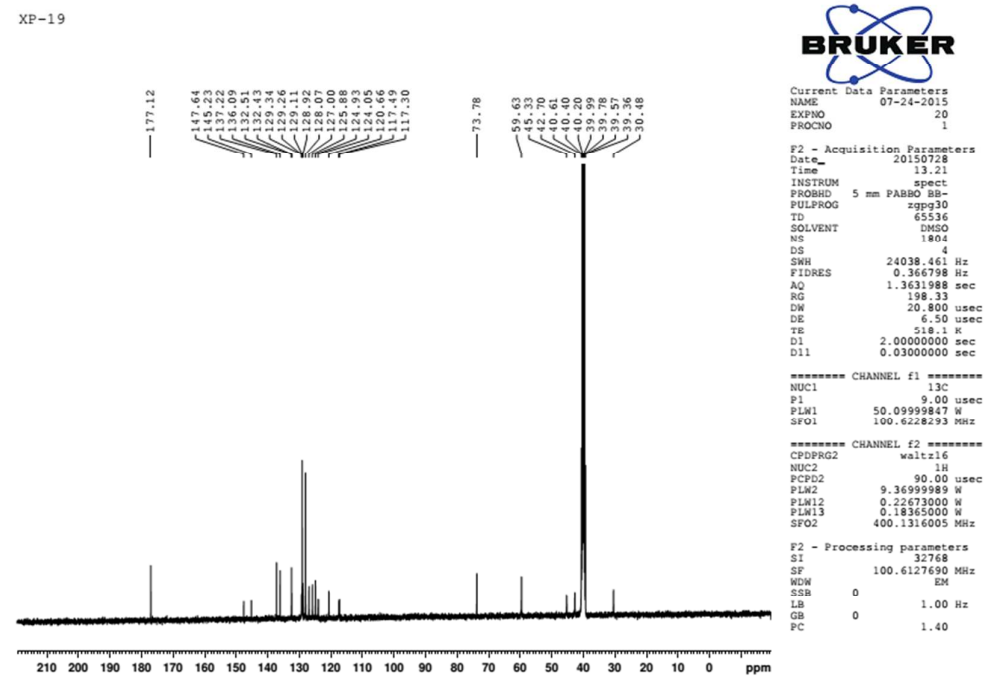


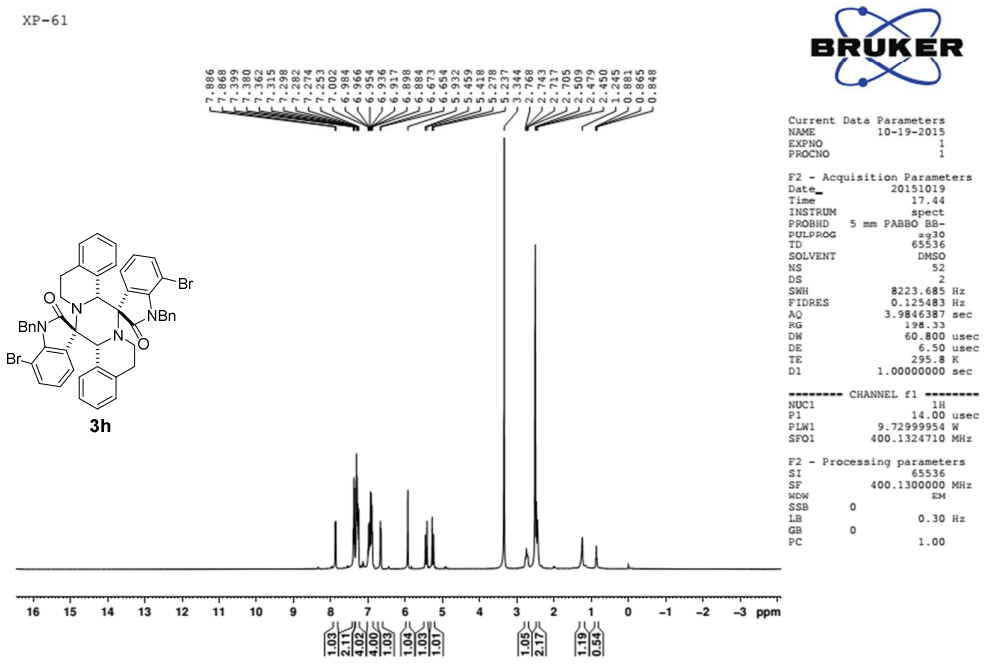


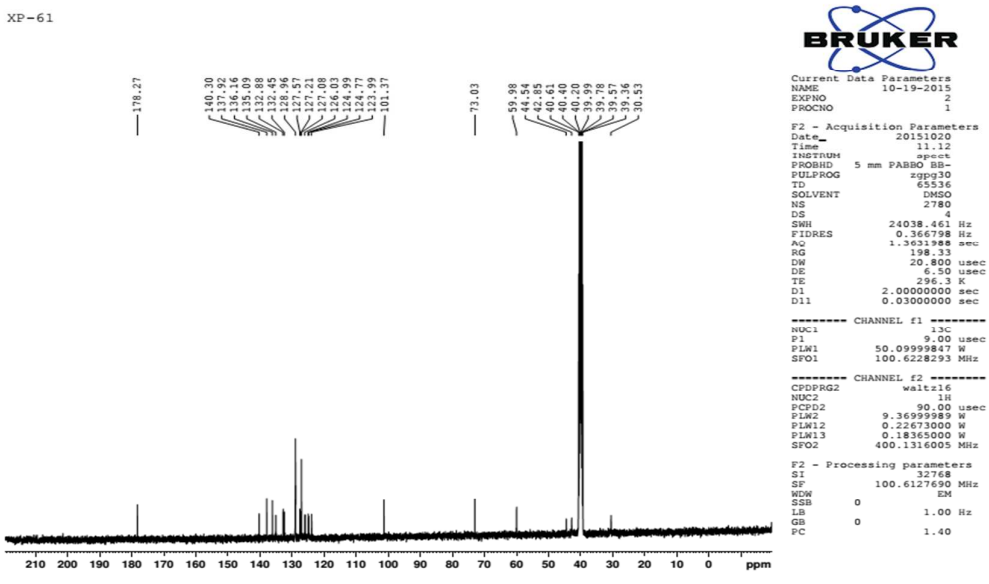
^{1}H NMR (400 MHz; DMSO-d_6), ^{13}C NMR (100 MHz; DMSO-d_6)



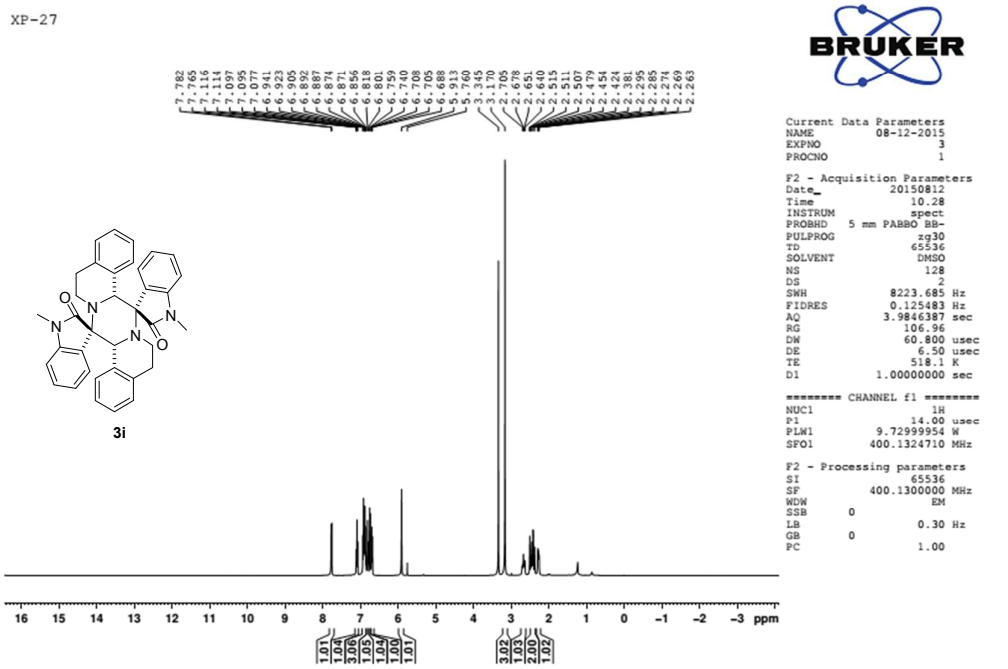


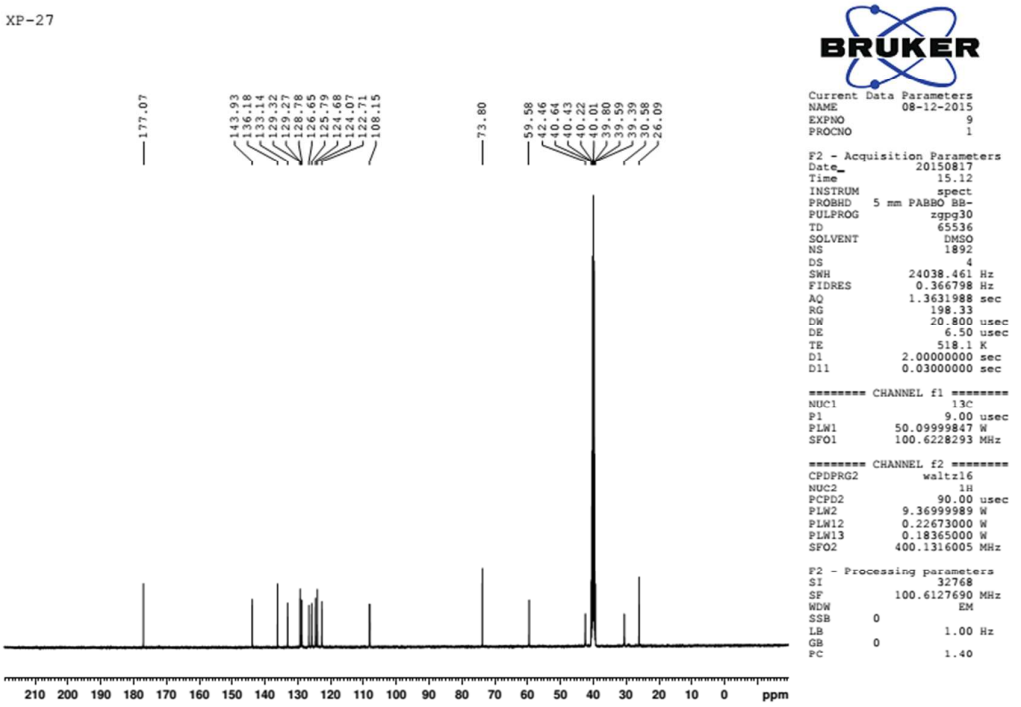
^{1}H NMR (400 MHz; DMSO-d_6), ^{13}C NMR (100 MHz; DMSO-d_6)





^{1}H NMR (400 MHz; DMSO-d_6), ^{13}C NMR (100 MHz; DMSO-d_6)





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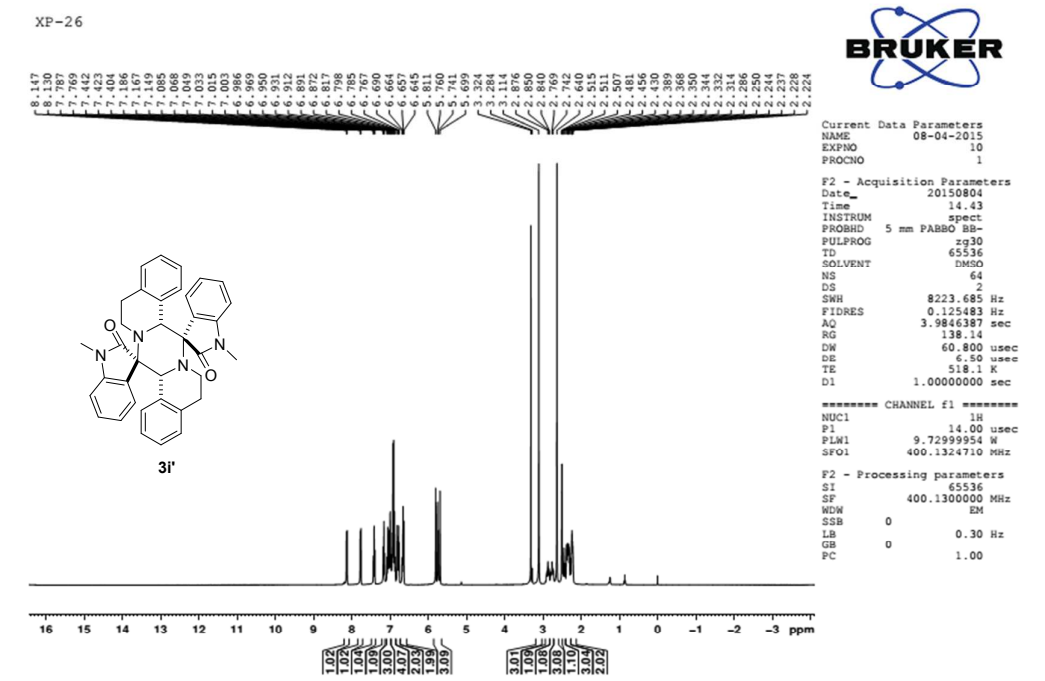

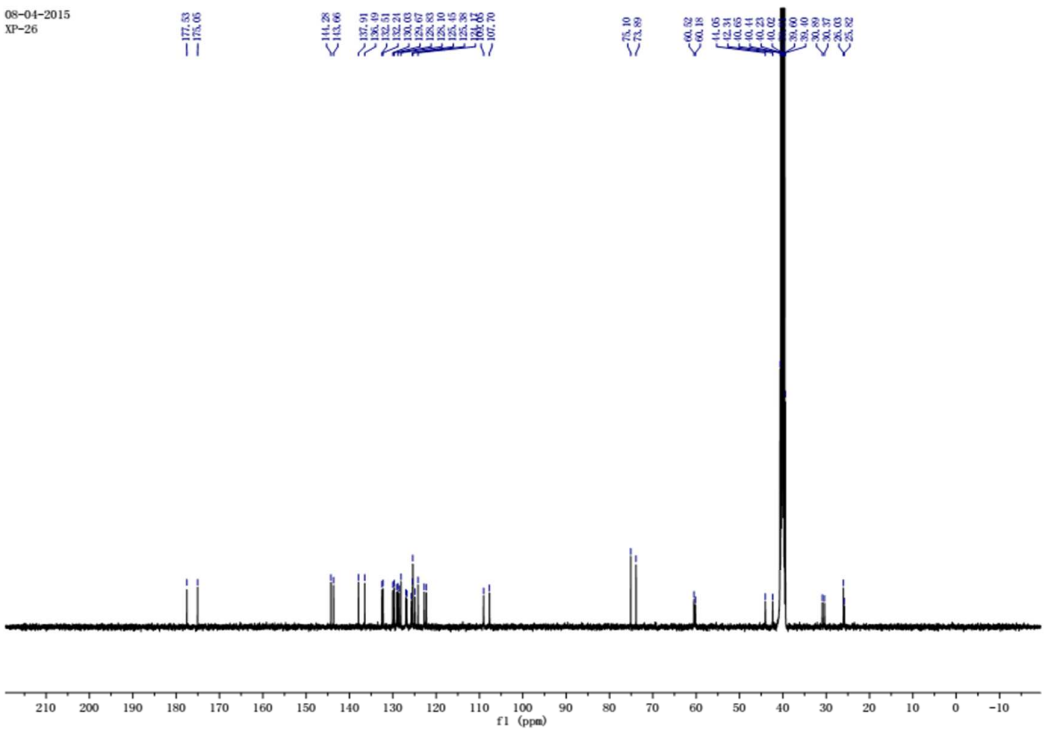

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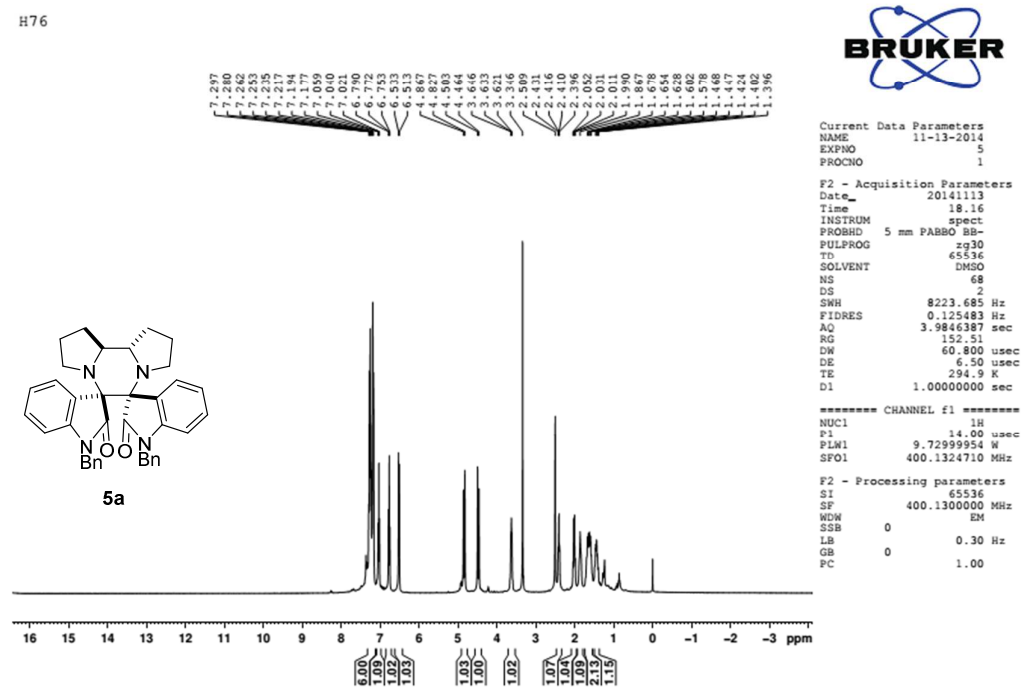

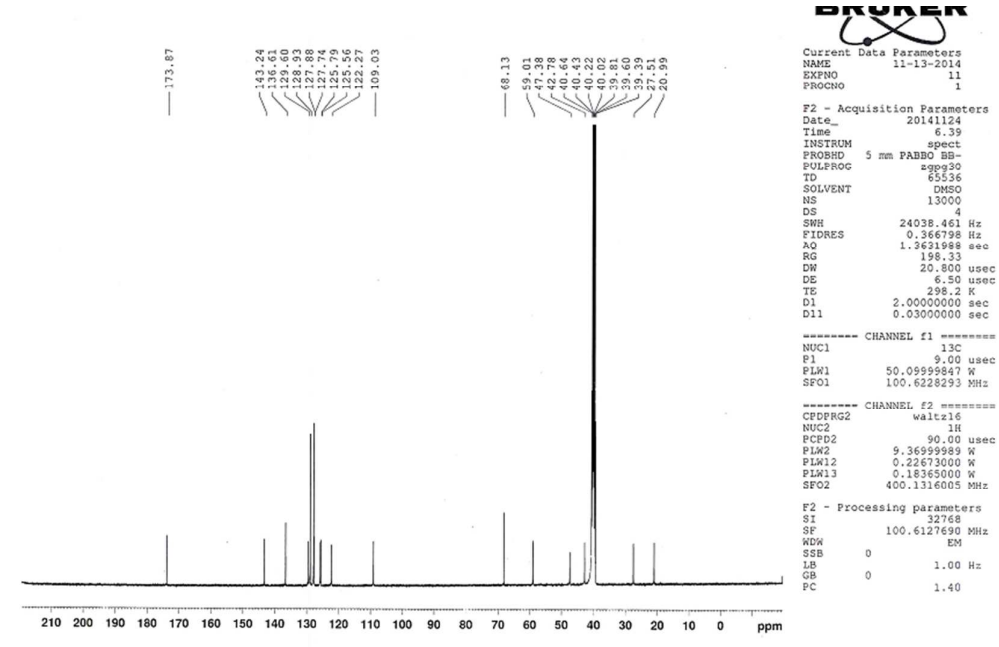

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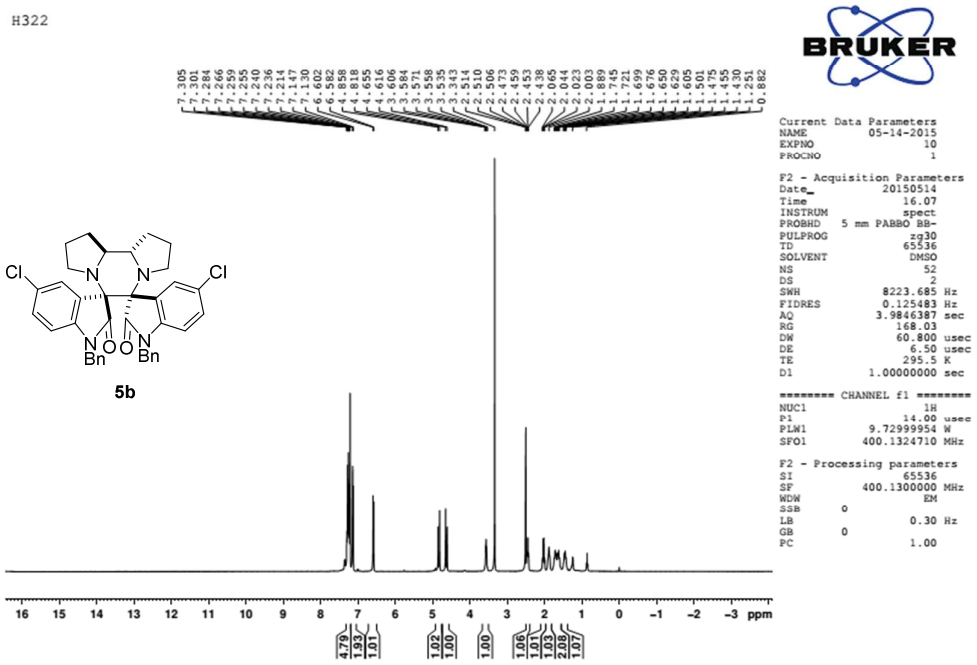

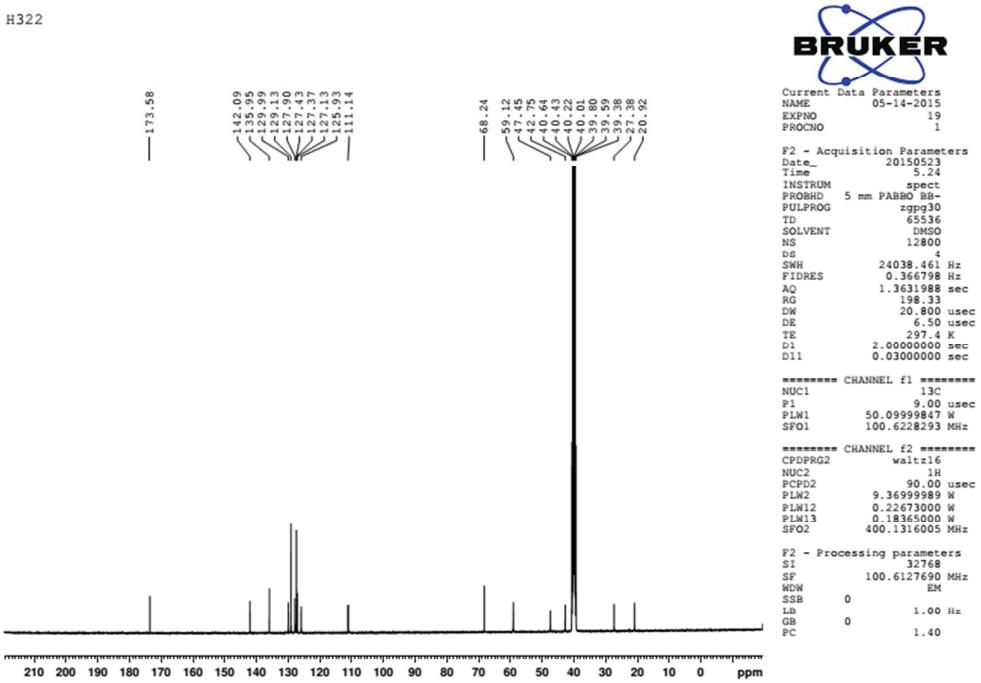

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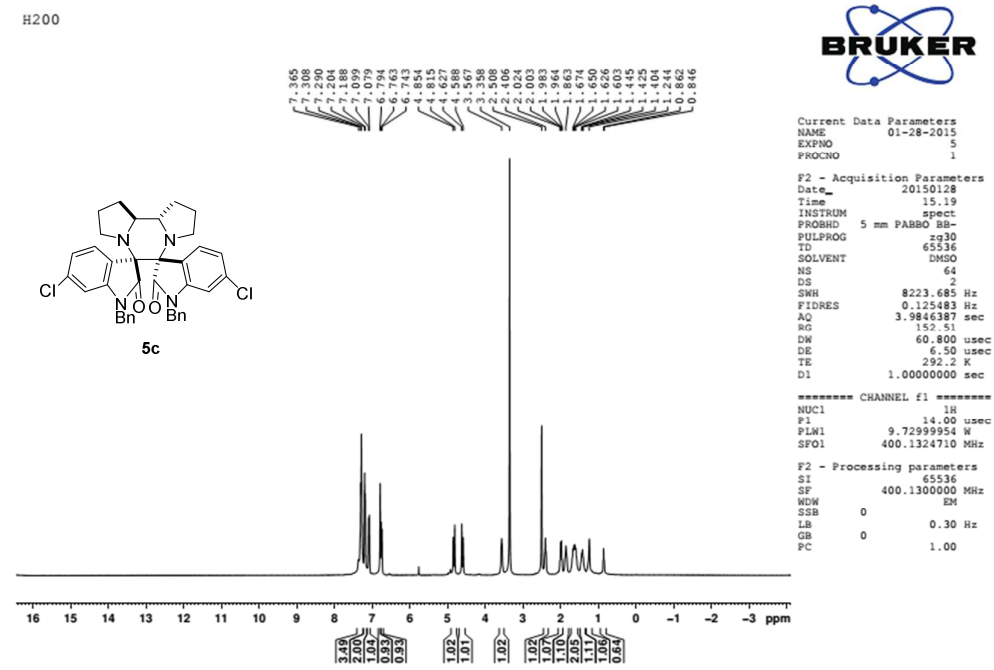

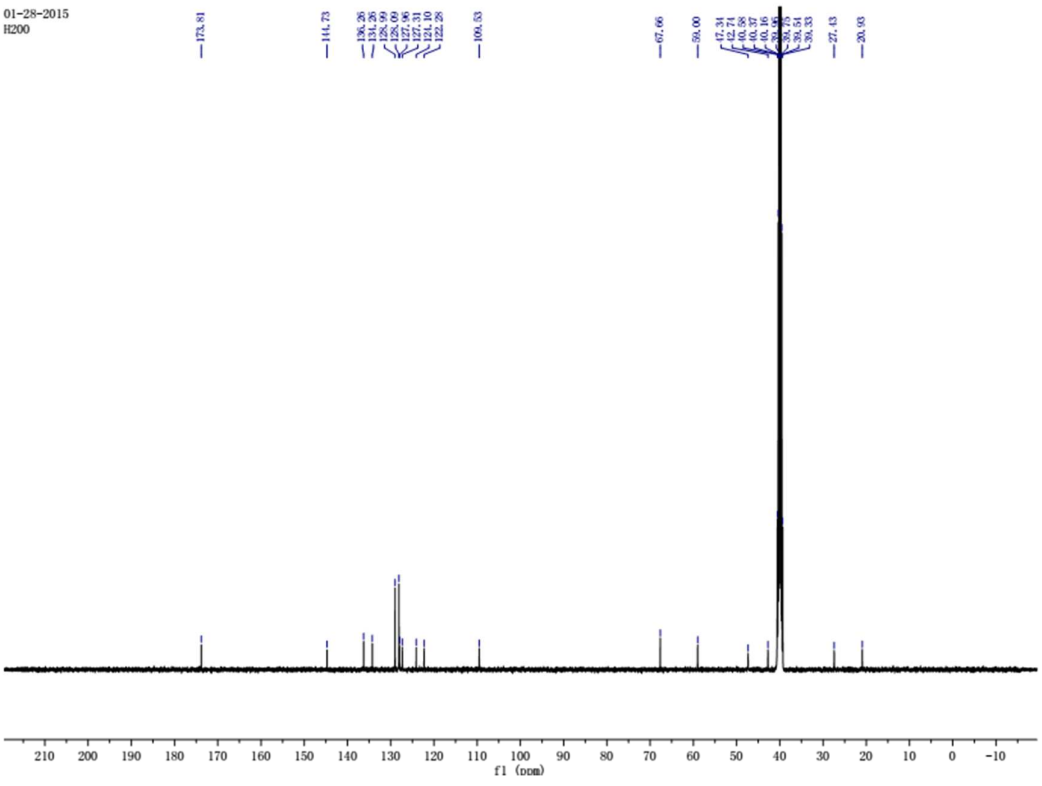

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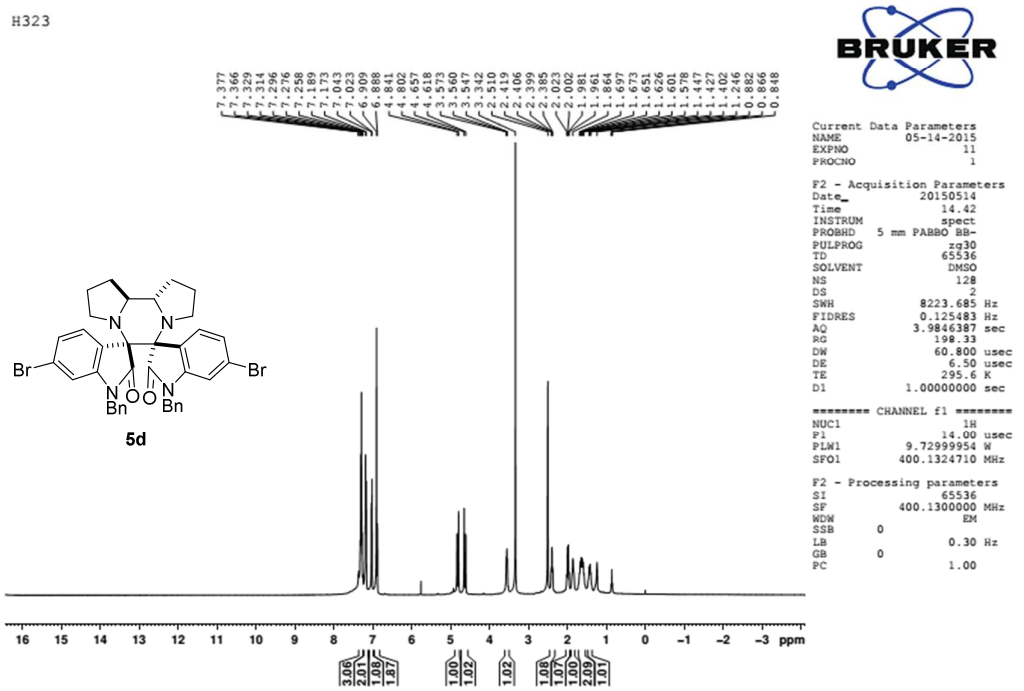

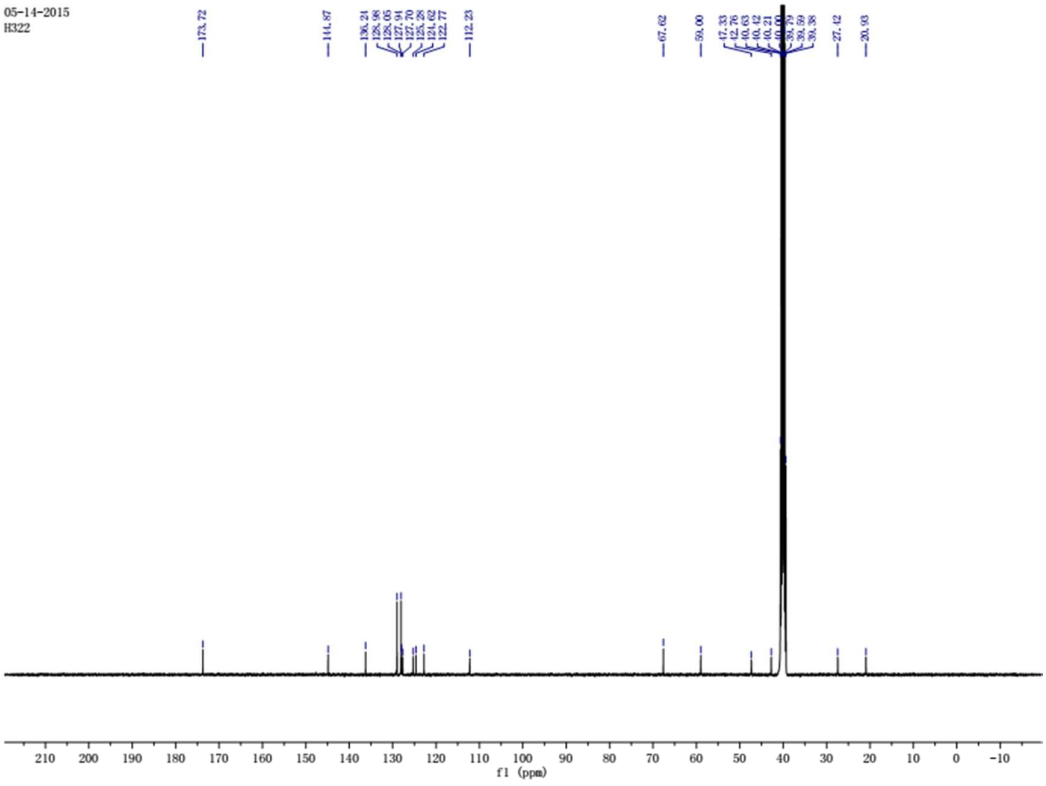

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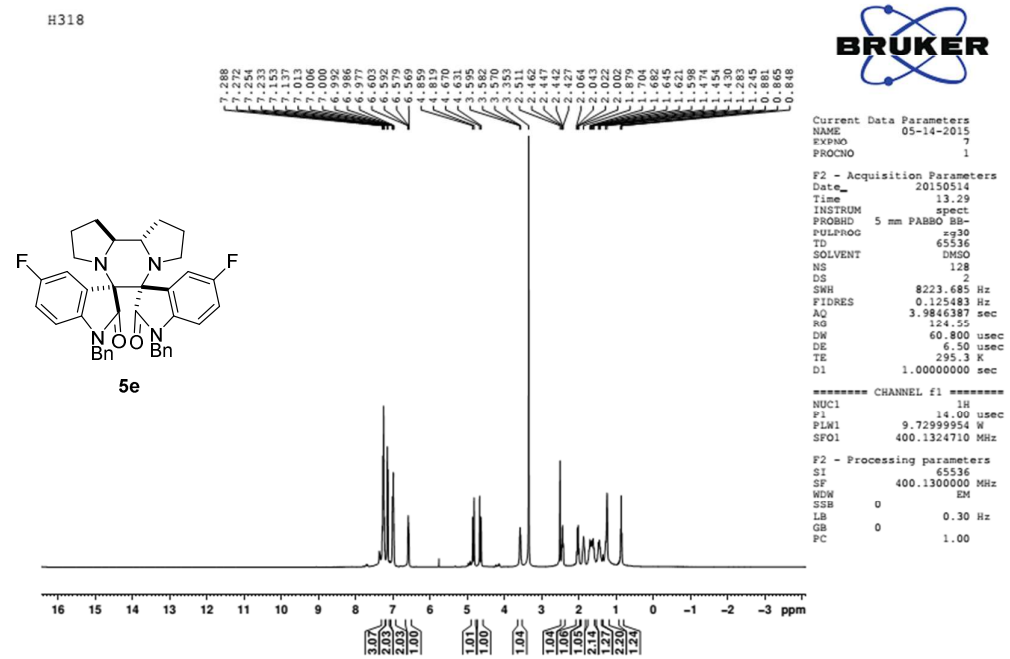

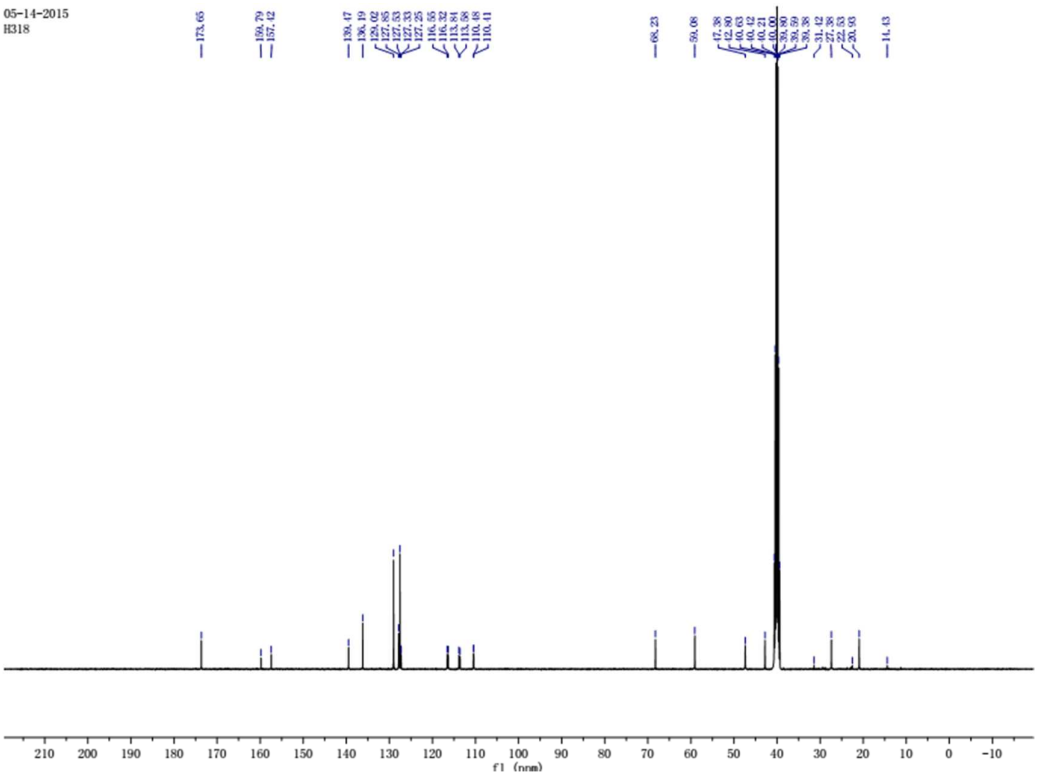

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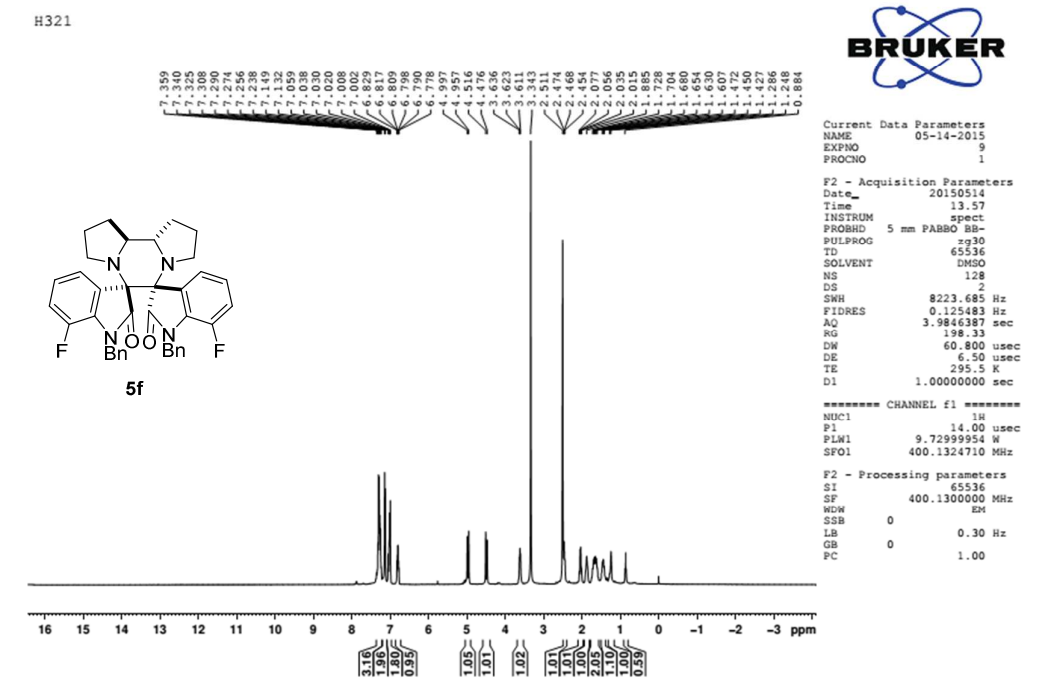

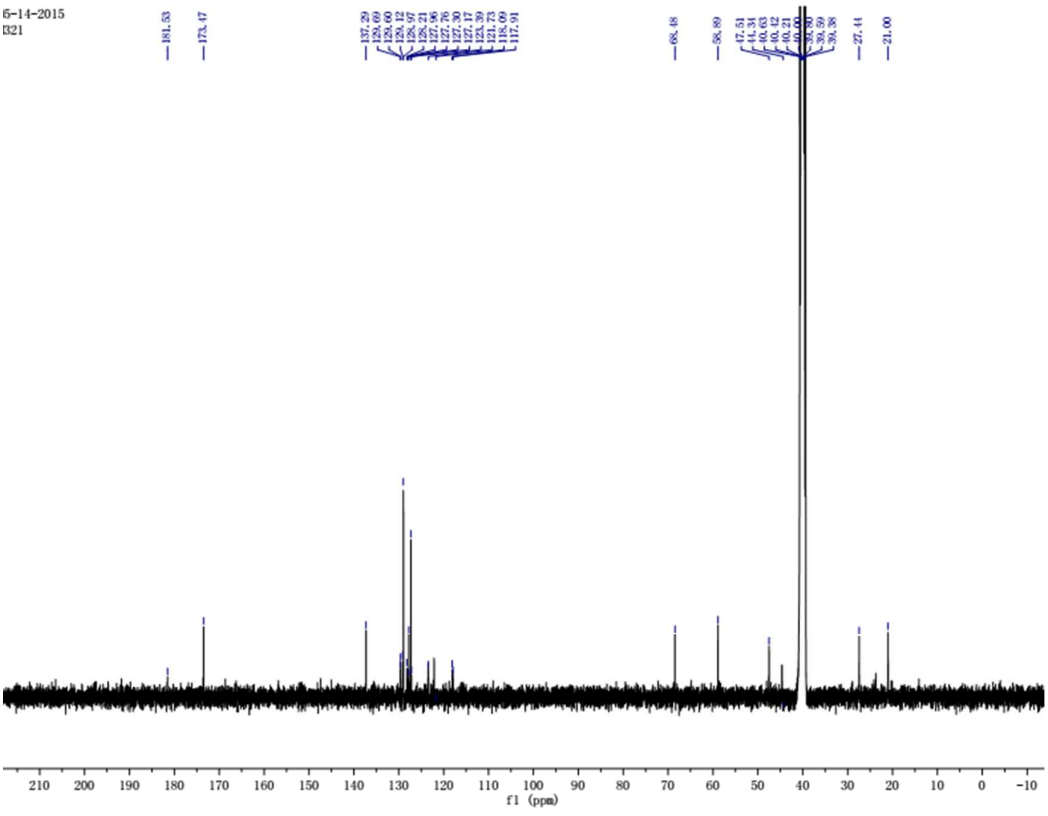

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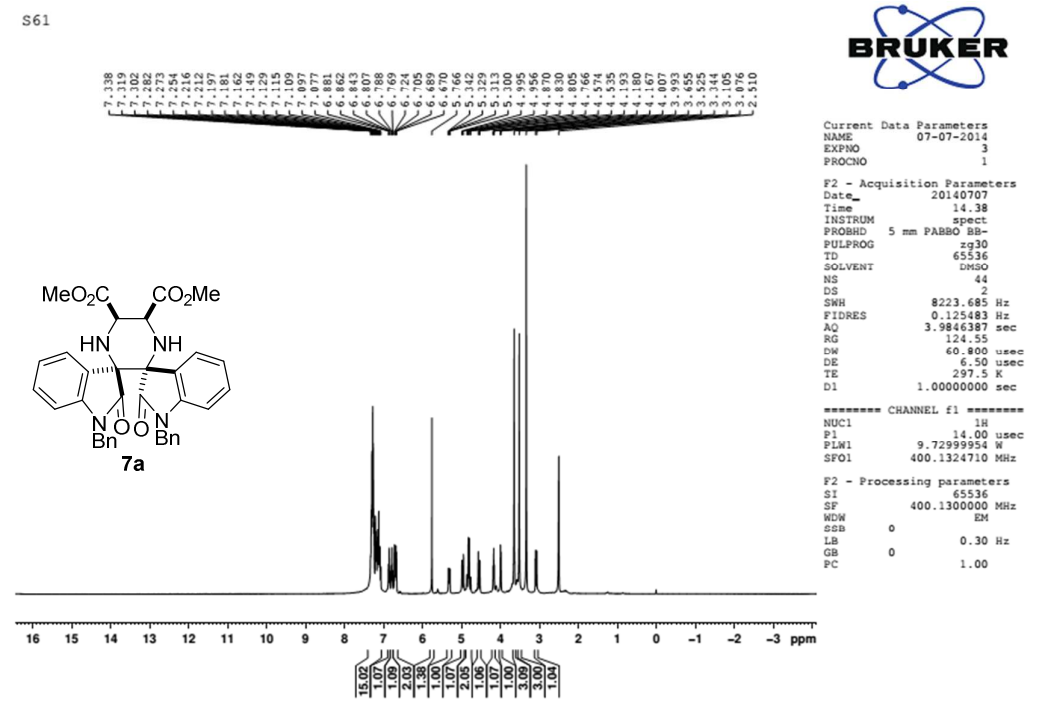

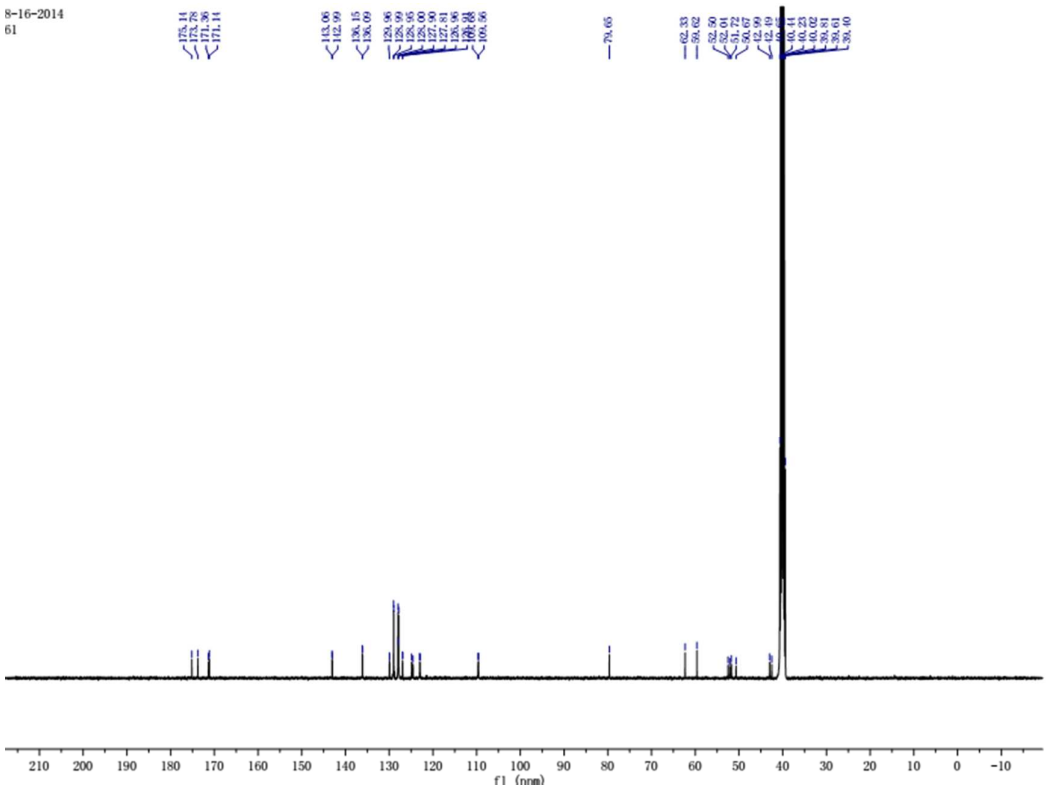

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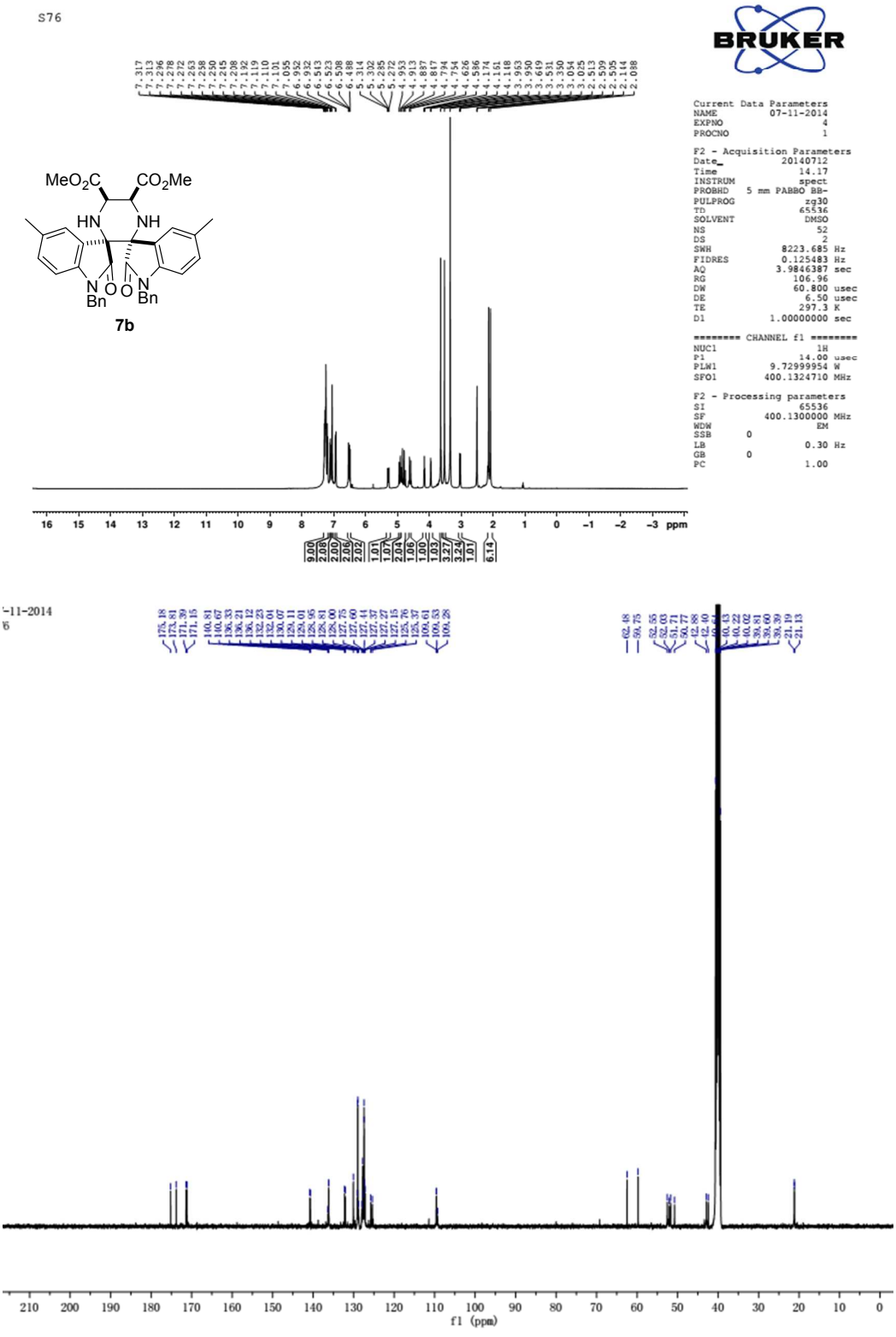

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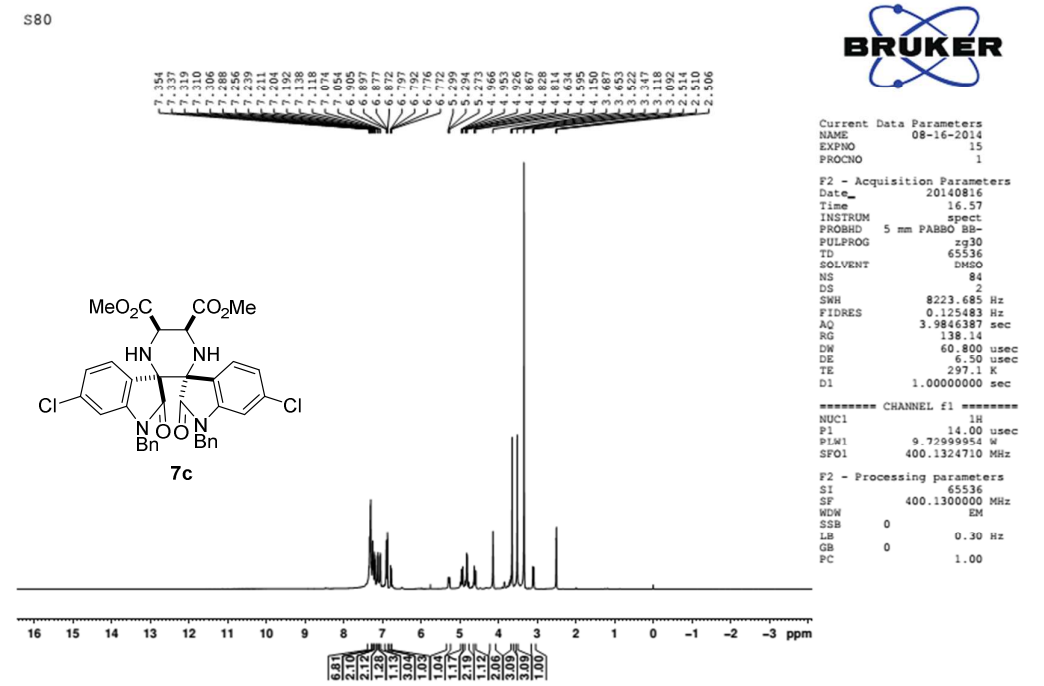

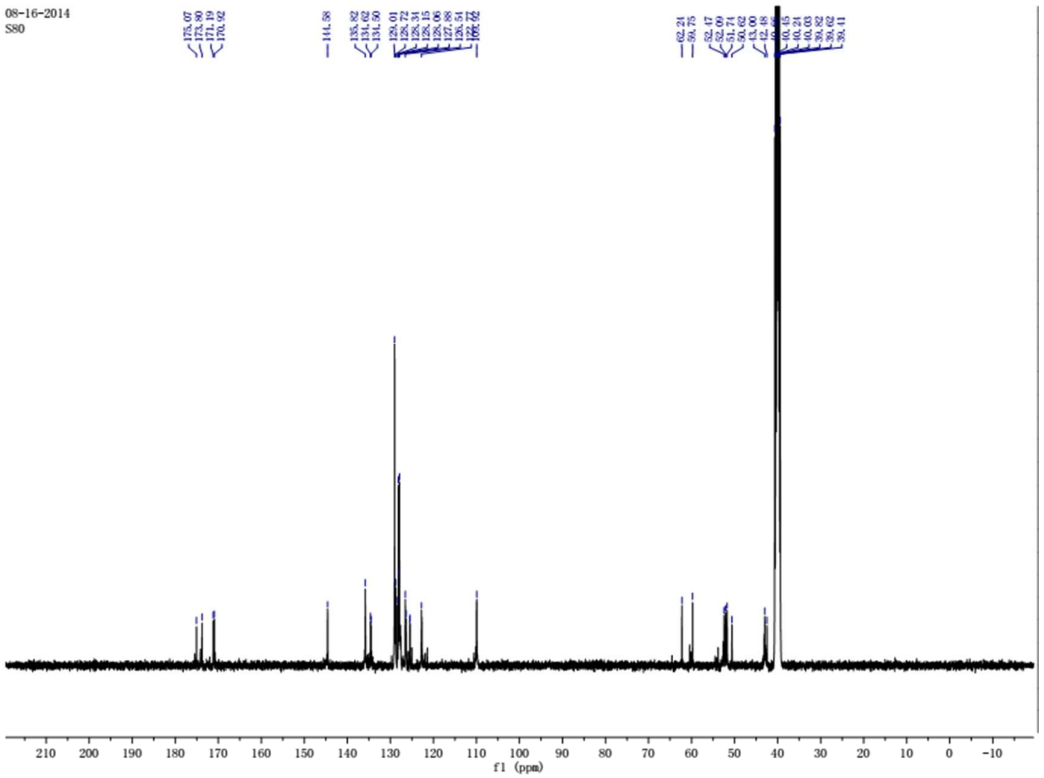

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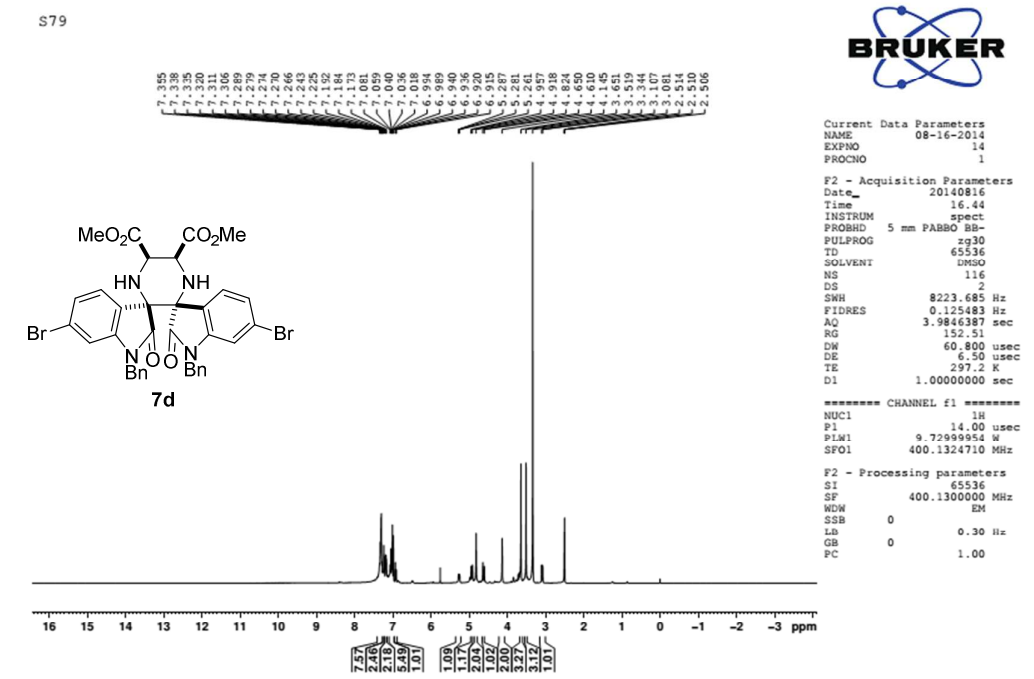
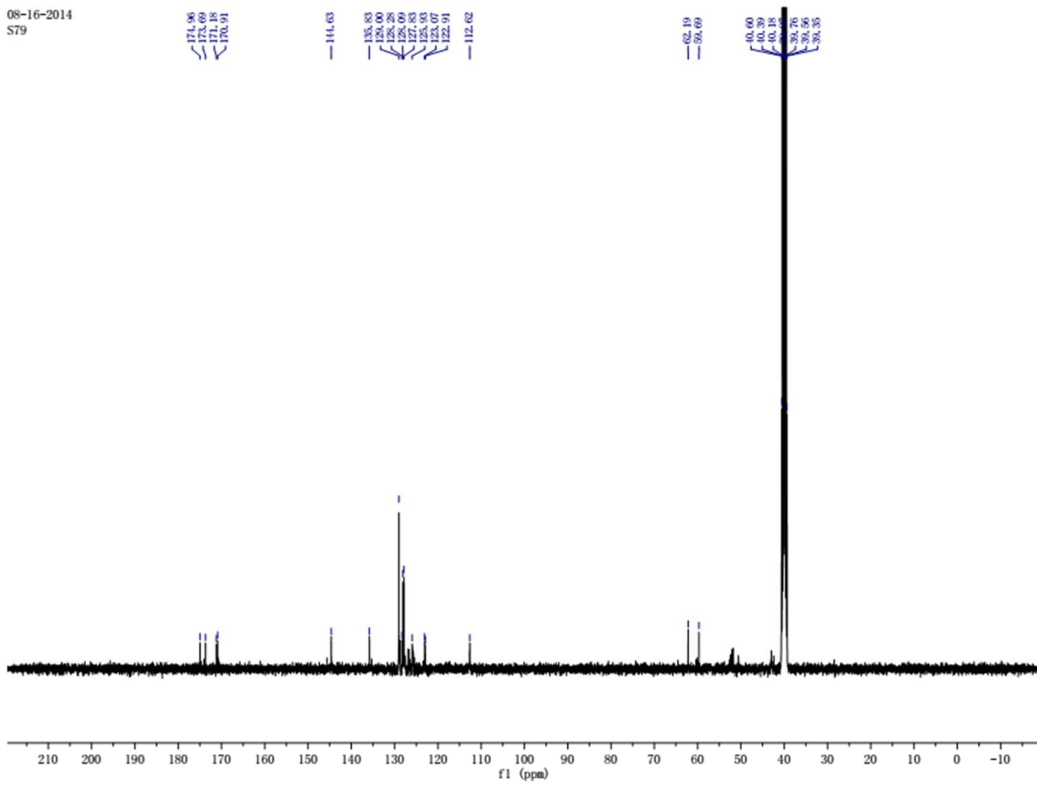
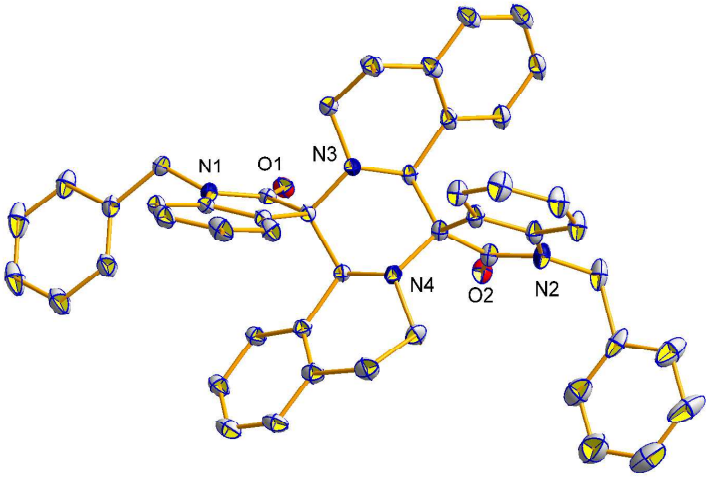
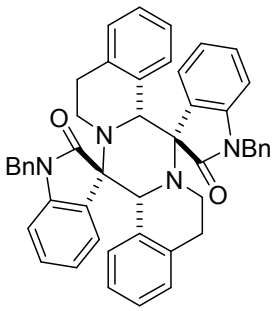




Crystal structure of dispirooxindole-piperazine 3a

X-Ray crystallographic analysis of dispirooxindole-piperazine 3a (CCDC 1421682) showing the thermal ellipsoids at 30% probability level.





3a

Bond precision: C-C = 0.0035 A Wavelength=0.71073

Cell: a=9.2740(4) b=11.7097(4) c=20.2631(7)
alpha=84.558(2) beta=85.736(2) gamma=69.051(2)
Temperature: 295 K

	Calculated	Reported
Volume	2043.83(14)	2043.83(13)
Space group	P -1	P-1
Hall group	-P 1	?
Moiety formula	C48 H40 N4 O2	?
Sum formula	C48 H40 N4 O2	C48 H40 N4 O2
Mr	704.84	704.84
Dx,g cm-3	1.145	1.145
Z	2	2
Mu (mm-1)	0.070	0.070
F000	744.0	744.0
F000'	744.28	
h,k,lmax	11,13,24	11,13,24
Nref	7189	7171
Tmin,Tmax	0.986,0.986	0.986,0.986
Tmin'	0.986	

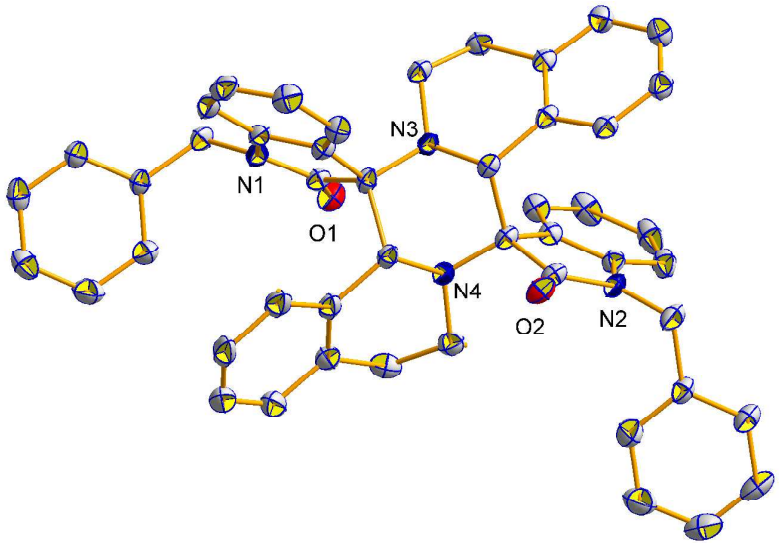
Correction method= # Reported T Limits: Tmin=0.986 Tmax=0.986
AbsCorr = ?

Data completeness= 0.997 Theta(max)= 25.010

R(reflections)= 0.0491(4820) wR2(reflections)= 0.1595(7171)

S = 1.064 Npar= 518

X-Ray crystallographic analysis of dispirooxindole-piperazine 3a' (CCDC 1421681) showing the thermal ellipsoids at 30% probability level.



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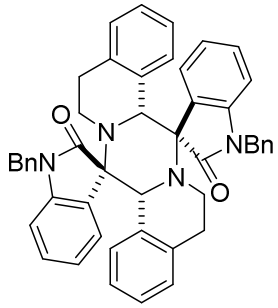


3a'

Bond precision: C-C = 0.0040 A Wavelength=0.71073

Cell: a=12.4855(14) b=12.3203(14) c=29.040(3)
alpha=90 beta=100.910(6) gamma=90
Temperature: 296 K

	Calculated	Reported
Volume	4386.3(8)	4386.4(9)
Space group	P 21/c	P2(1)/c
Hall group	-P 2ybc	?
Moiety formula	C48 H40 N4 O2	?
Sum formula	C48 H40 N4 O2	C48 H40 Cl0 N4 O2
Mr	704.84	704.84
Dx,g cm-3	1.067	1.067
Z	4	4
Mu (mm-1)	0.066	0.066
F000	1488.0	1488.0
F000'	1488.56	
h,k,lmax	14,14,34	14,14,34
Nref	7752	7679
Tmin,Tmax	0.987,0.987	
Tmin'	0.987	

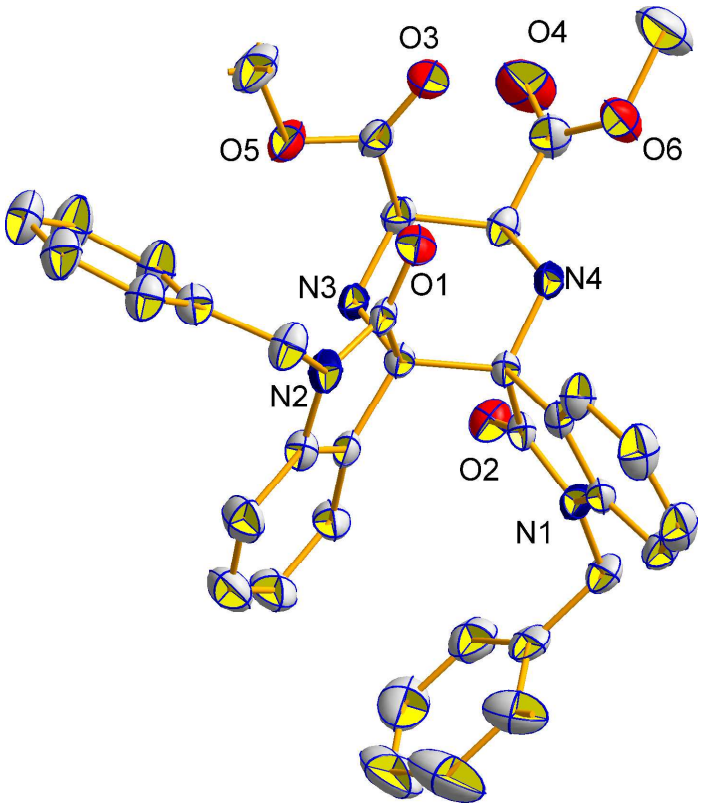
Correction method= Not given

Data completeness= 0.991 Theta(max)= 25.030

R(reflections)= 0.0593(4329) wR2(reflections)= 0.1809(7679)

S = 0.956 Npar= 487

X-Ray crystallographic analysis of dispirooxindole-piperazine 7a (CCDC 1021637) showing the thermal ellipsoids at 30% probability level.



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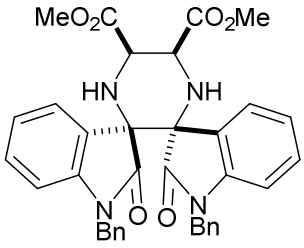

7a

Bond precision: C-C = 0.0056 A Wavelength=0.71073

Cell: a=13.369(18) b=21.18(3) c=10.884(15)
alpha=90 beta=106.287(15) gamma=90
Temperature: 296 K

	Calculated	Reported
Volume	2958(7)	2958(7)
Space group	P 21/c	P2(1)/c
Hall group	-P 2ybc	?
Moiety formula	C36 H32 N4 O6	?
Sum formula	C36 H32 N4 O6	C2.44 H2.17 N0.27 O0.41
Mr	616.66	41.81
Dx,g cm-3	1.385	1.385
Z	4	59
Mu (mm-1)	0.096	0.096
F000	1296.0	1296.0
F000'	1296.60	
h,k,lmax	17,27,14	17,27,14
Nref	7039	6912
Tmin,Tmax	0.977,0.990	
Tmin'	0.972	

Correction method= Not given

Data completeness= 0.982 Theta(max)= 27.840

R(reflections)= 0.0844(4042) wR2(reflections)= 0.2199(6912)

S = 1.046 Npar= 421