Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Electronic Supplementary Information (ESI)

Synthesis of New Terpyridine-like ligands based on Triazolopyridines and Benzotriazoles

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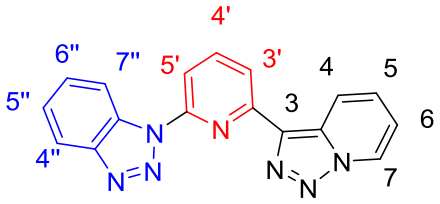
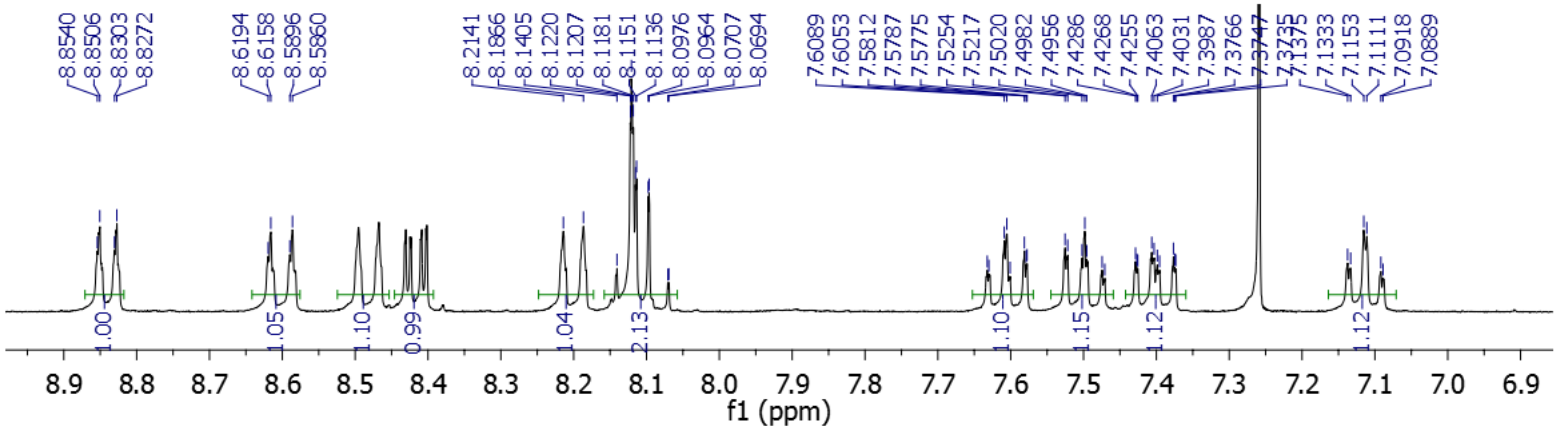
d) Centro de Innovación en Química Avanzada (ORFEO−CINQA), E-46100, Valencia, Spain

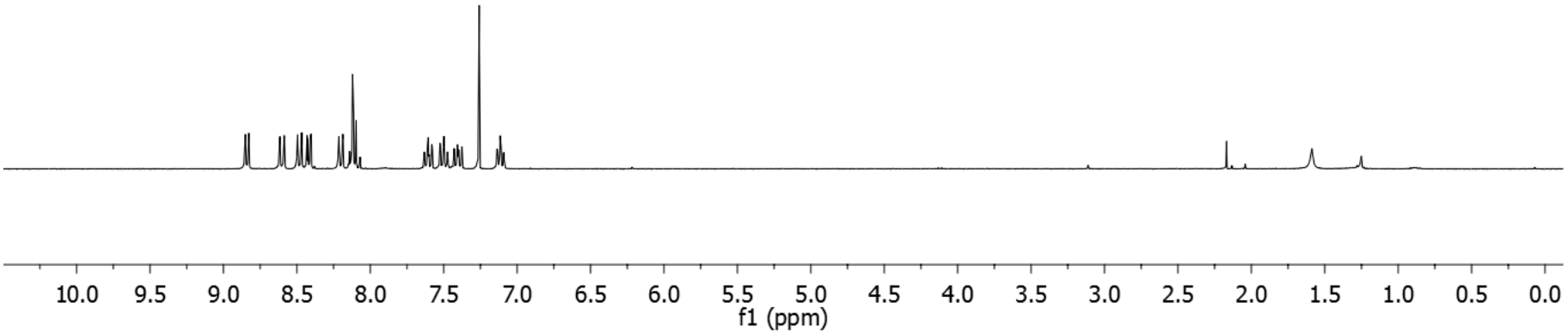
S1: Materials and Methods

Starting materials, if commercially available, were purchased and used as such. The solvents used were of spectroscopic or equivalent grade. When known compounds had to be prepared by literature procedures, pertinent references are given. Melting points or ranges (m.p.) given were determined on a Büchi B-545 heated stage. ^{1}H and (^{1}H decoupled) ^{13}C nuclear magnetic resonance (NMR) spectra were recorded at 300 and 75 MHz. Chemical shifts are reported in δ units, parts per million (ppm), and were measured relative to the signals for residual deuterated water or deuterated methanol. Coupling constants (J) are given in Hz. Coupling patterns are abbreviated as, for example, s (singlet), d (doublet), t (triplet), q (quartet), td (triplet of doublets), m (multiplet), app. s (apparent singlet) and br. (broad). COSY and DEPT/ed-HSQC experiments were performed for all compounds. IR spectra were recorded using FT-IR ATR. HRMS were recorded using TOF electro-spray ionization (ESI-positive). UV-Visible spectra were measured on an Agilent 8453 spectrometer equipped with a Peltier temperature controller system (±0.1 °C). The emission spectra were recorded with a PTI MO- 5020 spectrofluorimeter in the 300–700 nm range. Quantum yield was determined with a Hamamatsu-PHA equipment. The absorbance of the excitation wavelength was maintained lower than 0.15. 10^{-5} M solutions of ligands were prepared using 98/2 ethanol/water v/v as solvent. M^{2+} solutions were prepared solving the corresponding perchlorate in 98/2 ethanol/water v/v 10^{-3} mol dm^{-3} concentration. Working solutions were obtained mixing 2 mL of the solution of ligands with the corresponding amounts of the solutions of the metals.

S2: NMR Spectra







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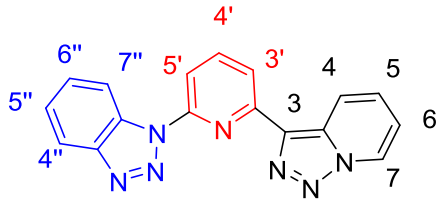

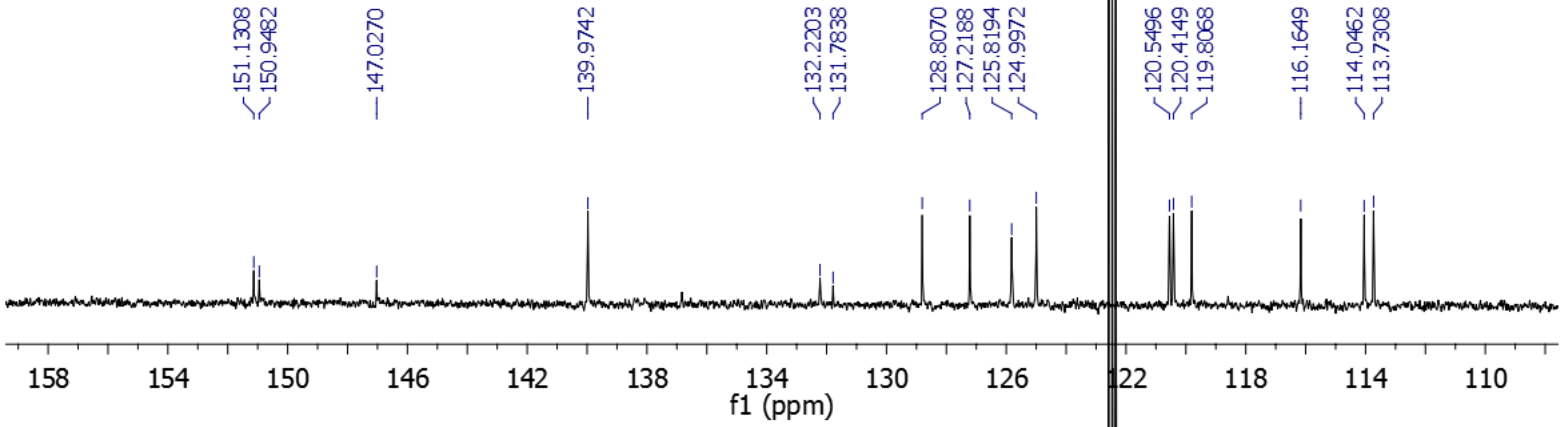

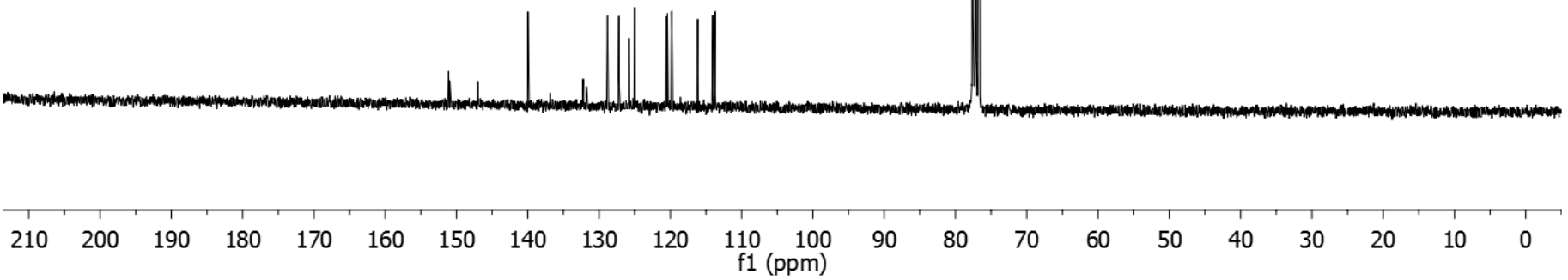

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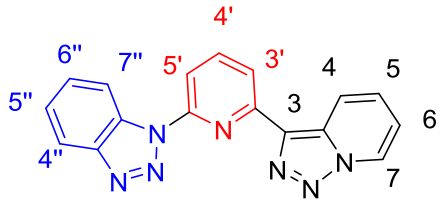

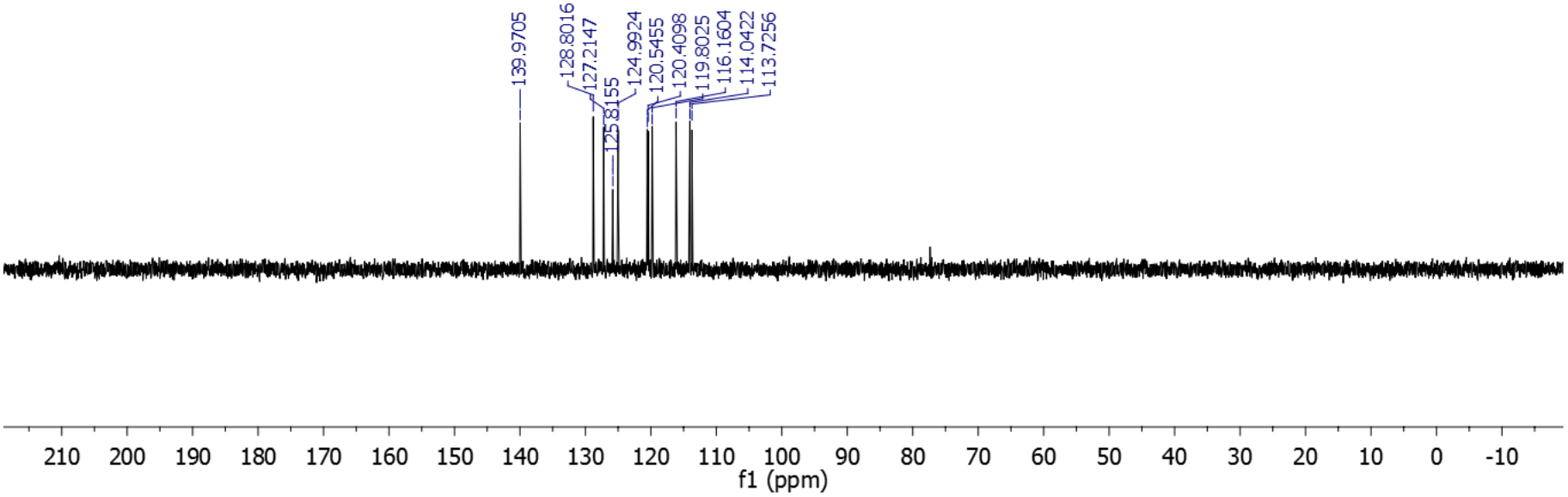

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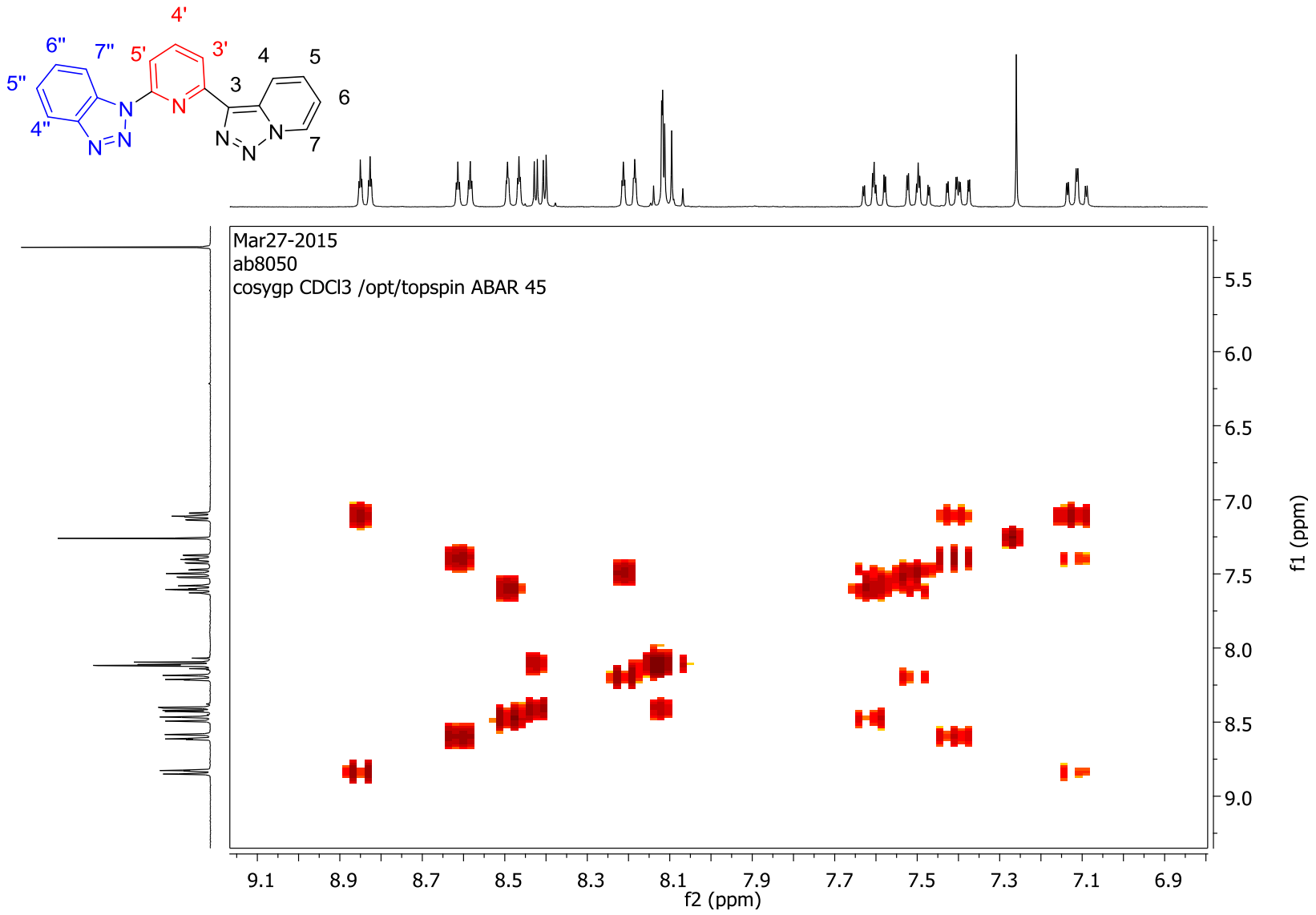

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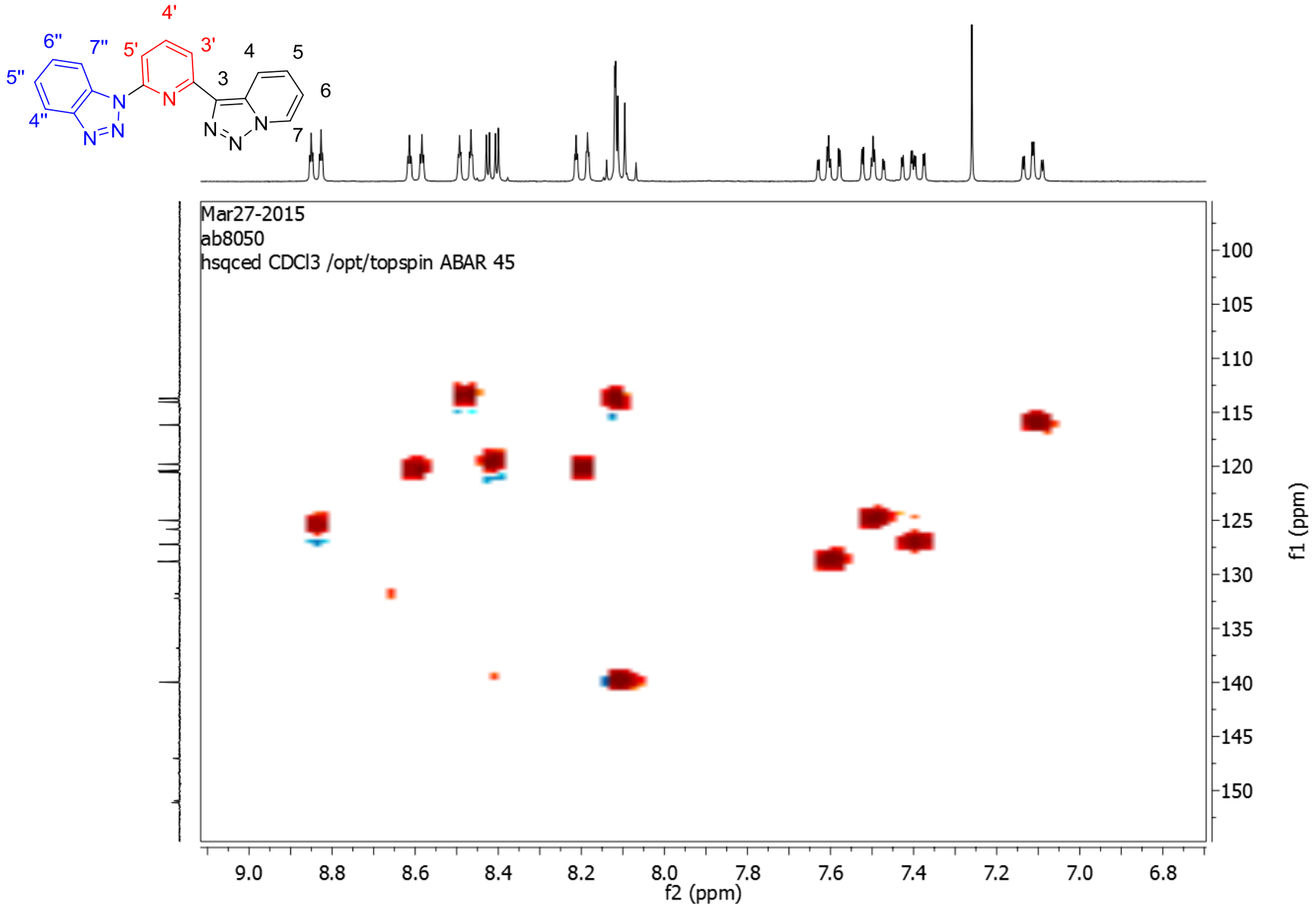

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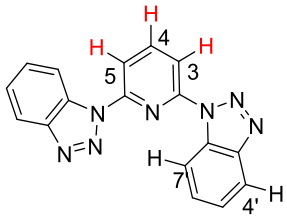

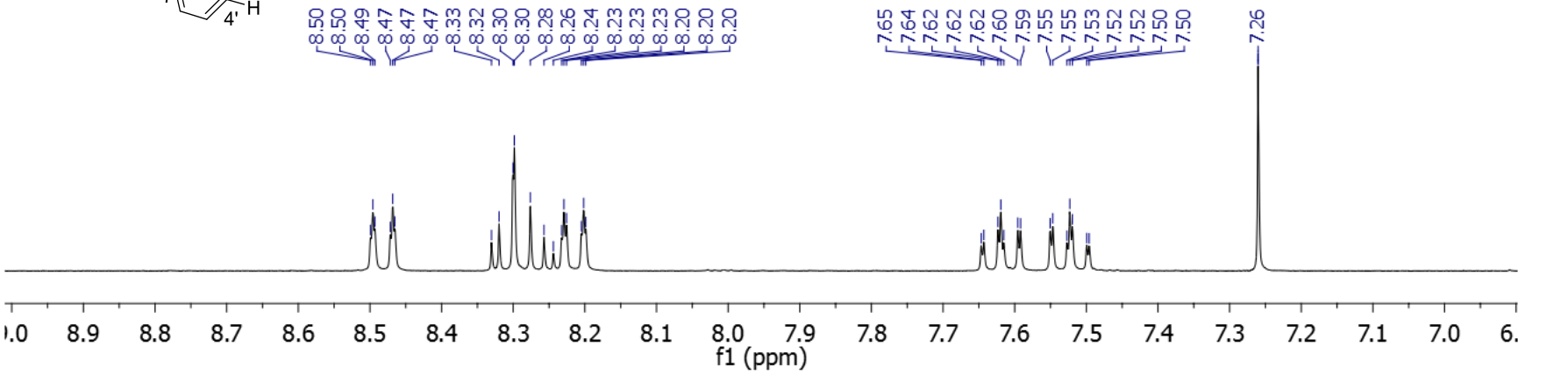

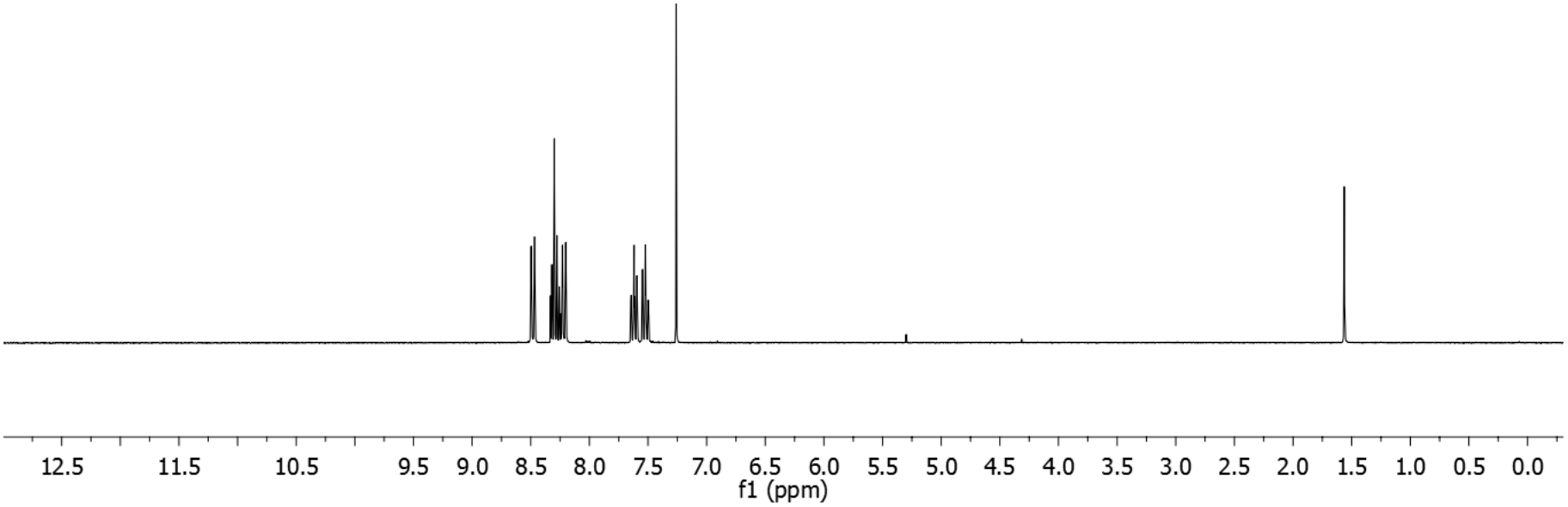

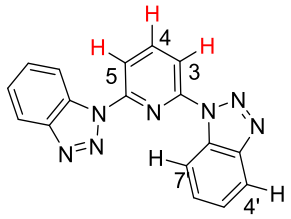
H
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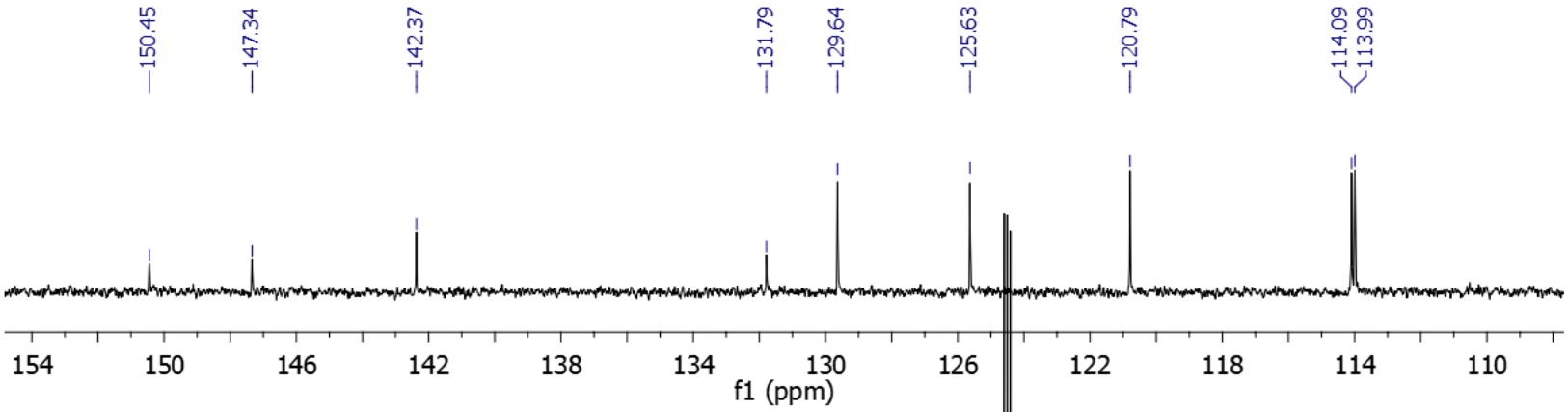
H
5
3
N
N
N
N
N
H
7
4'
H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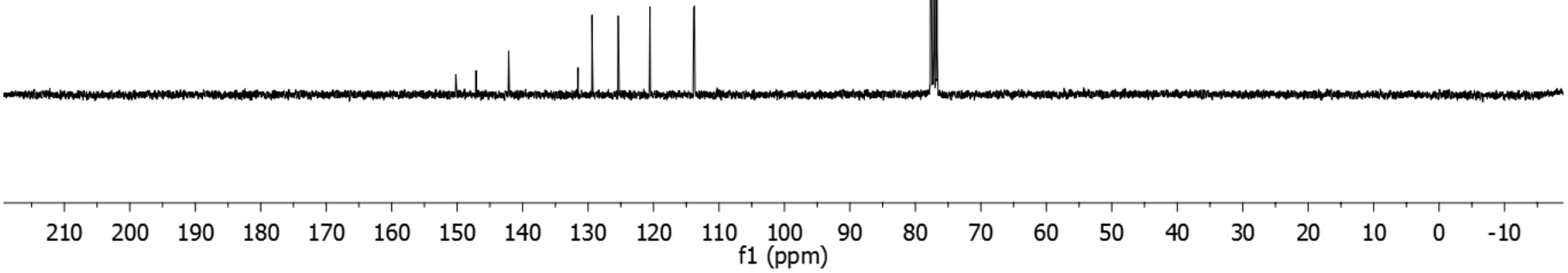
134
130
126
122
118
114
110
f1 (ppm)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200
190
180
170
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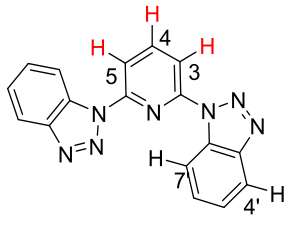
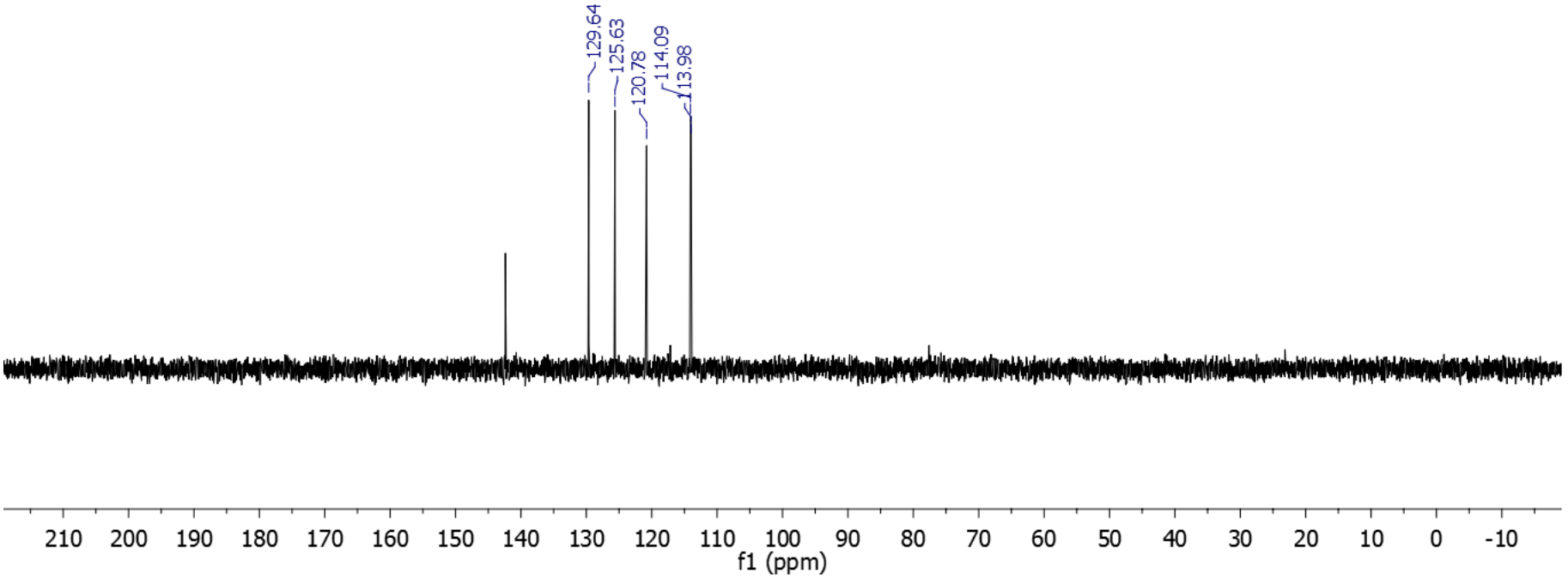
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

S2: NMR Spectra





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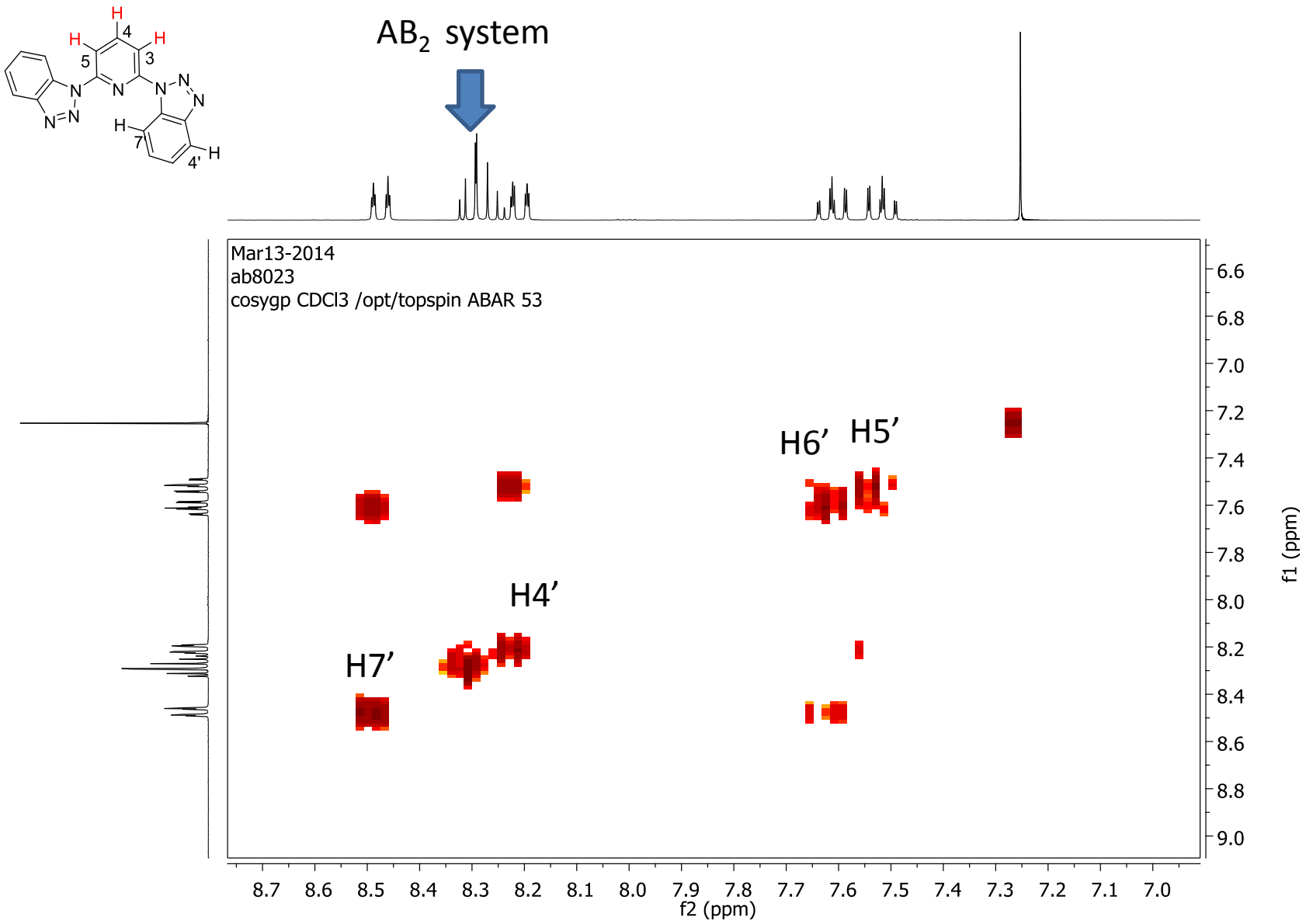

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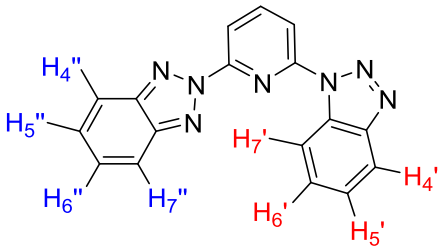

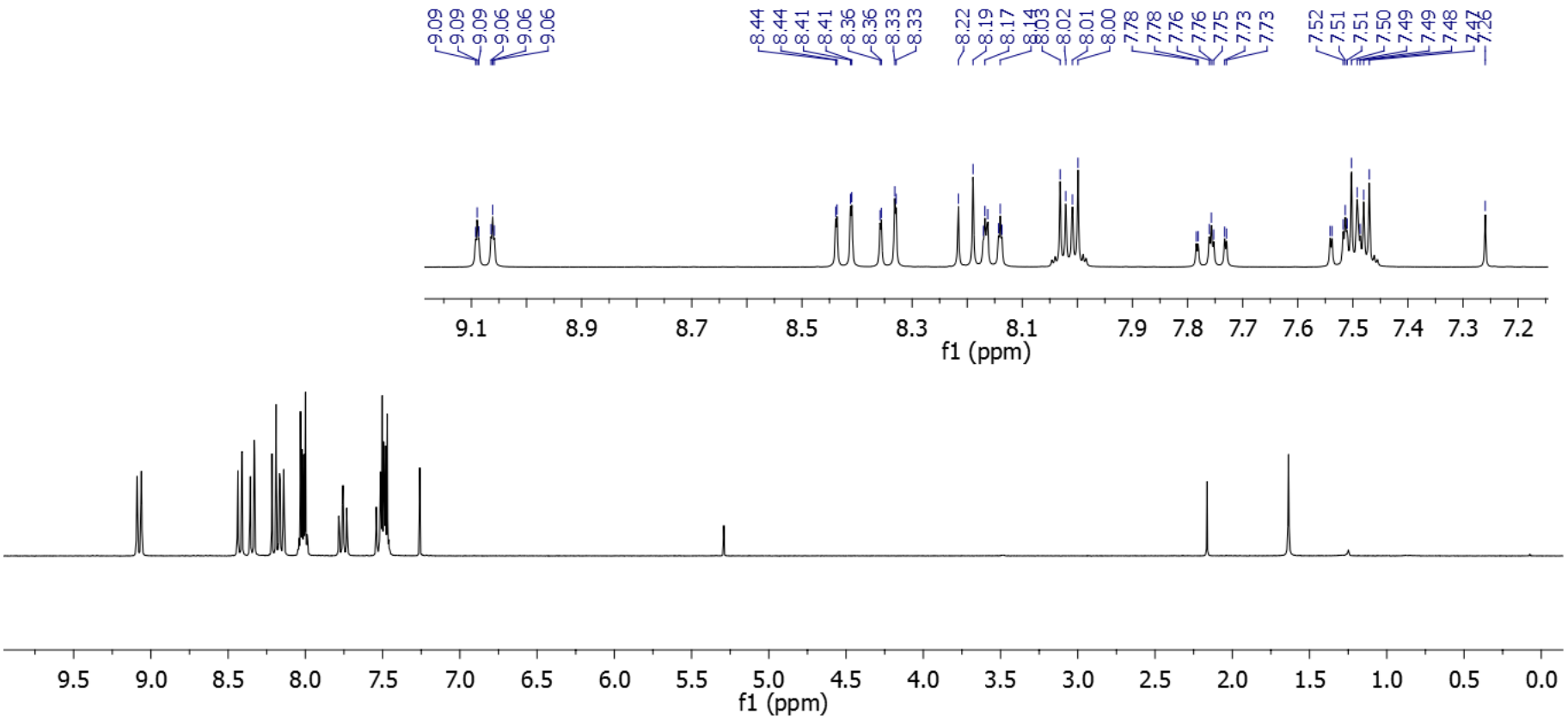

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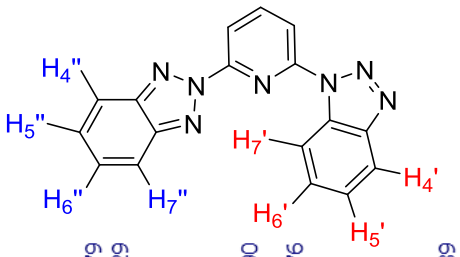

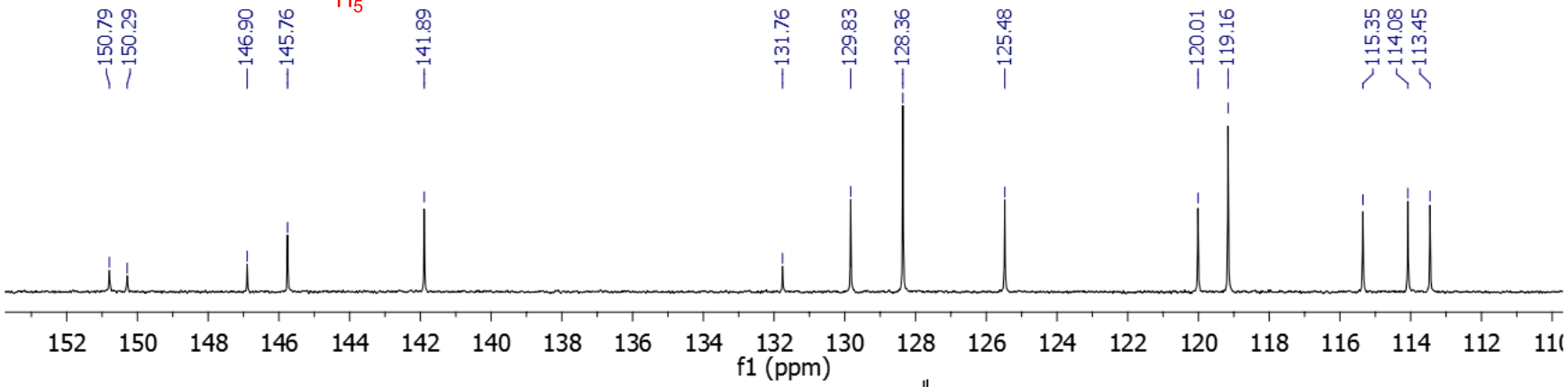

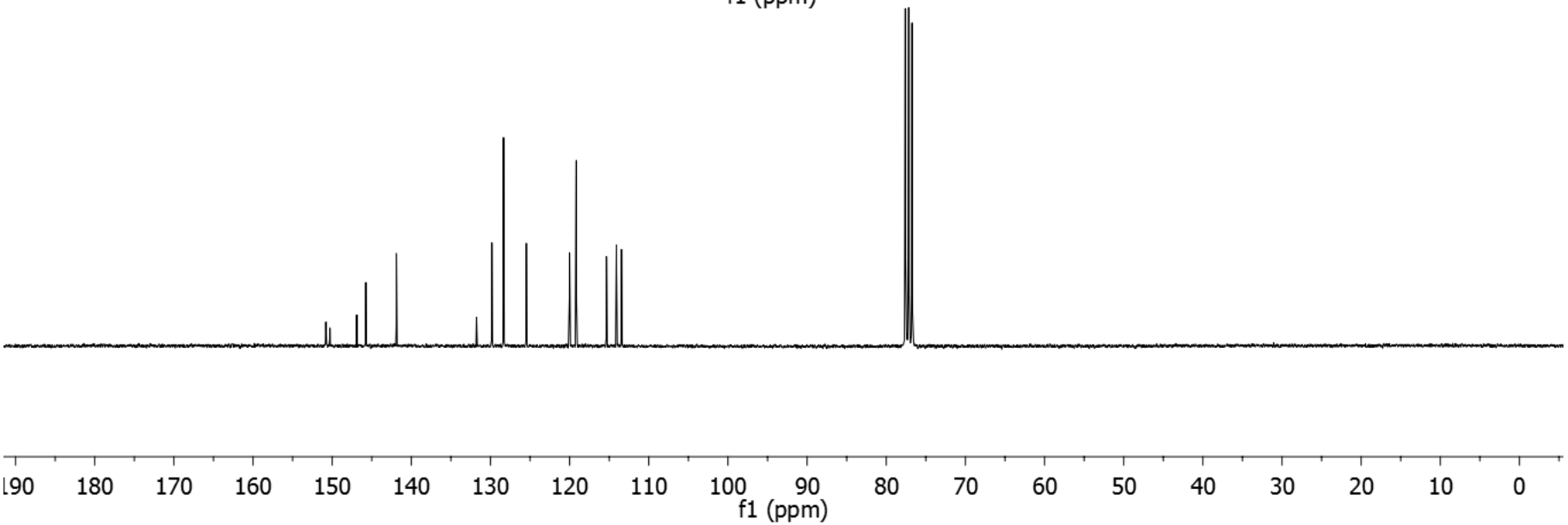

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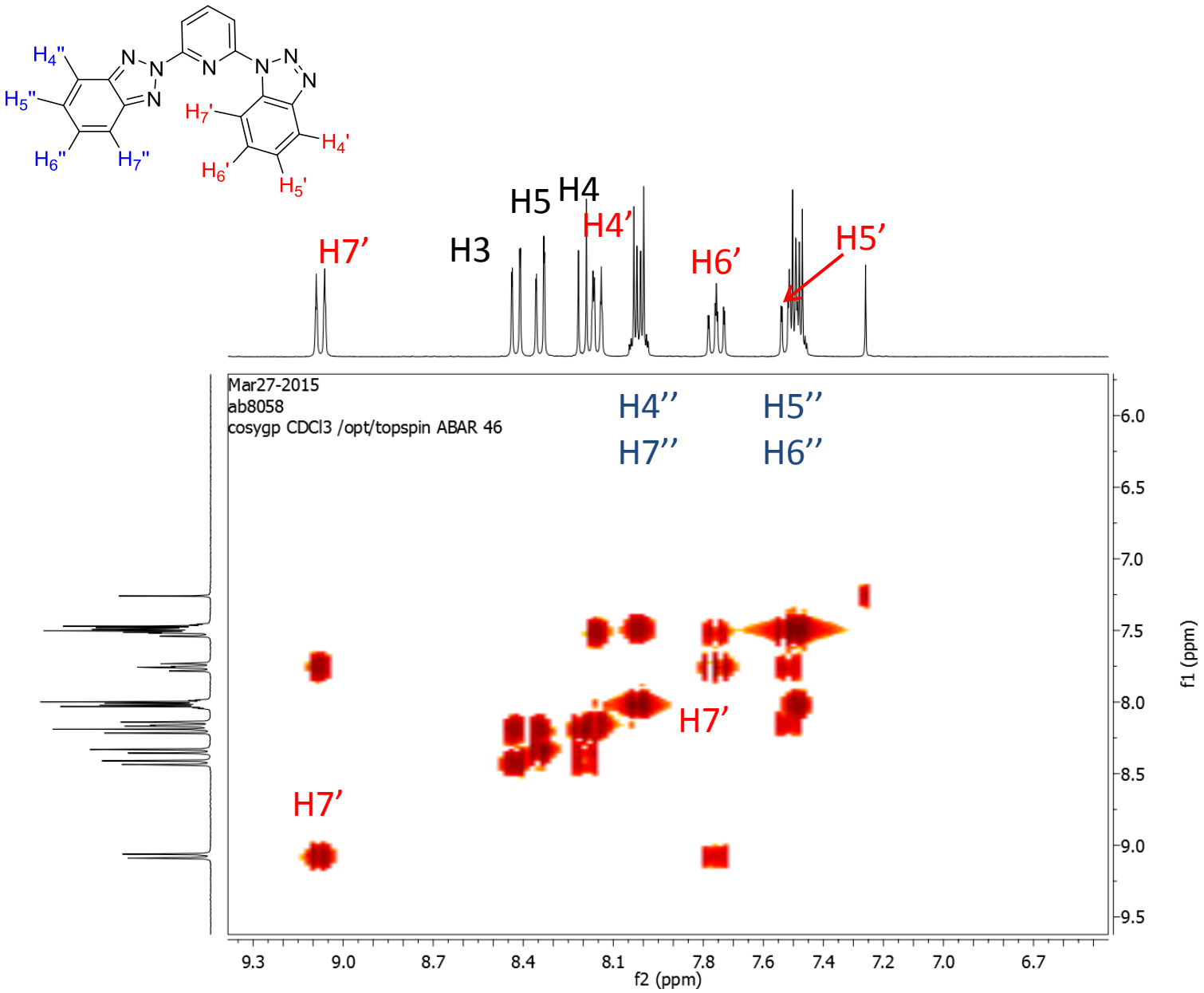

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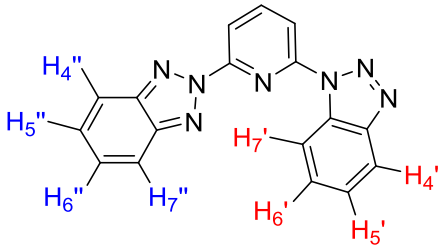
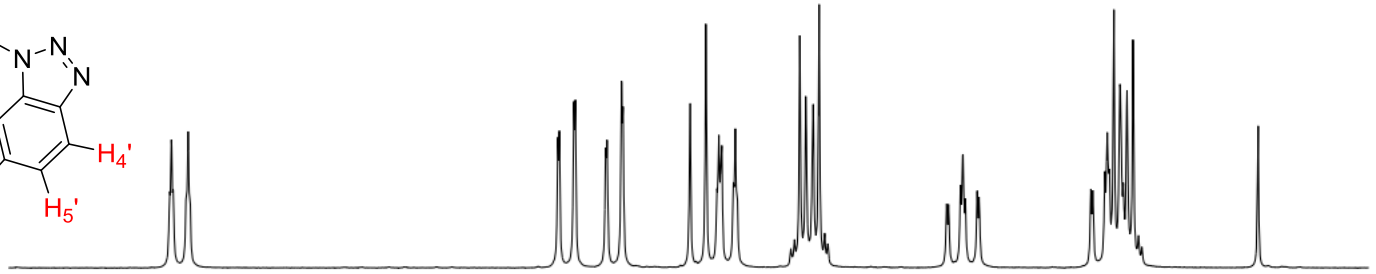
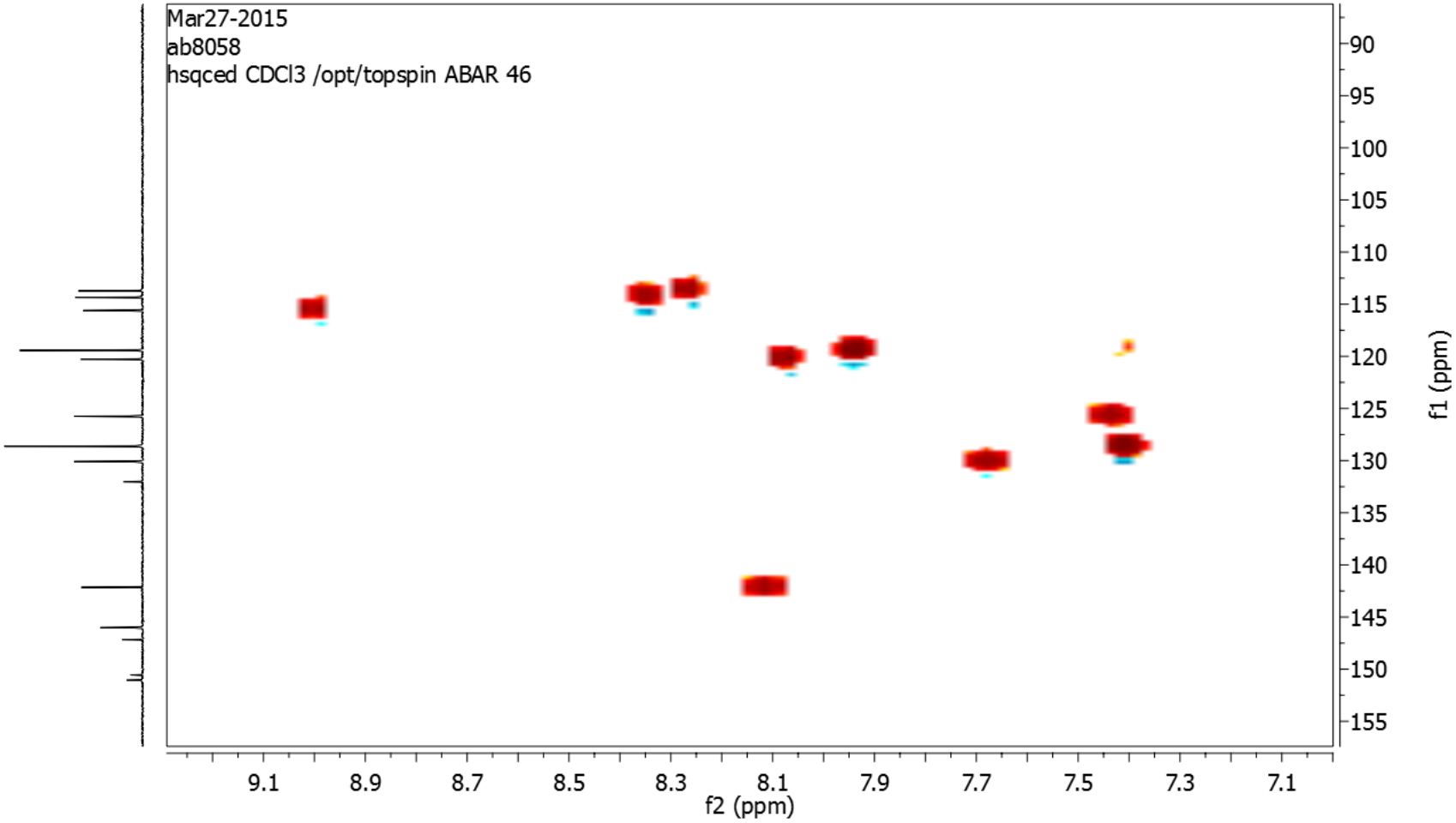
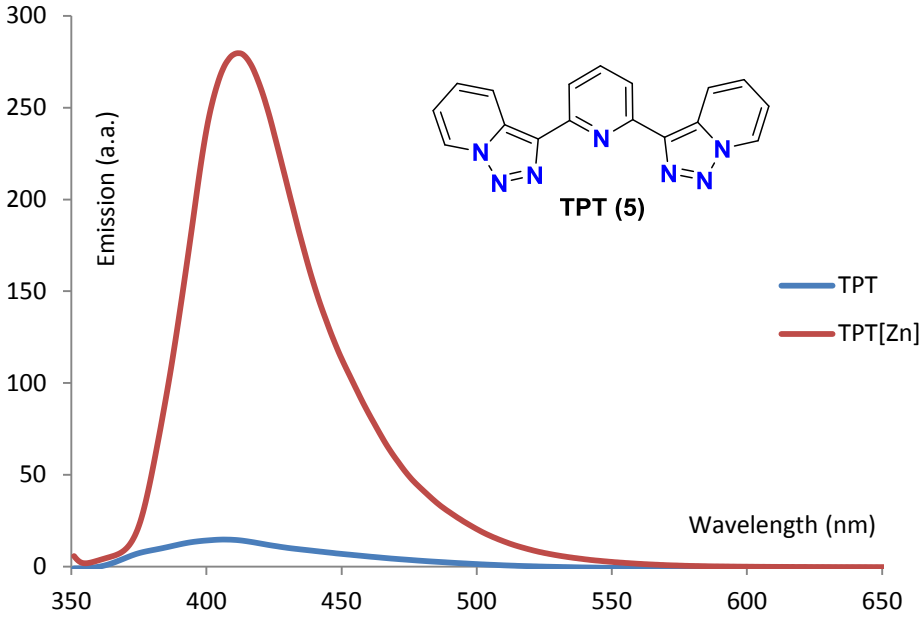
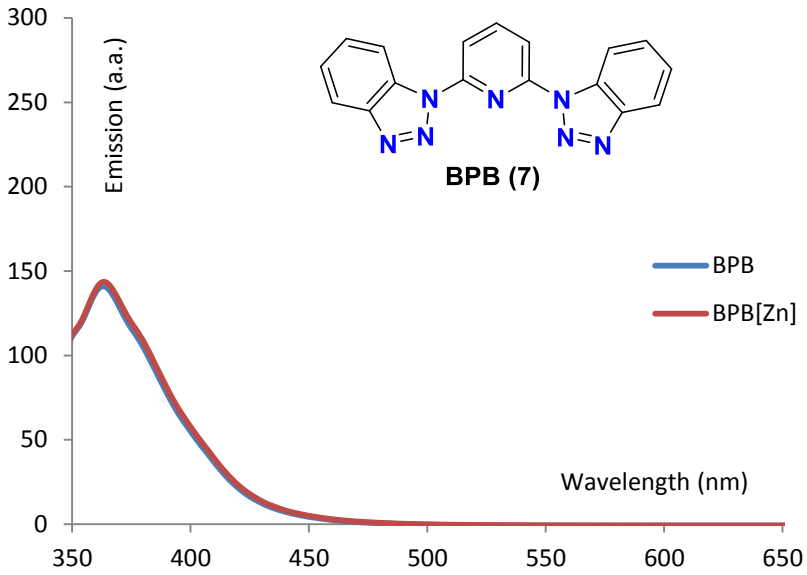


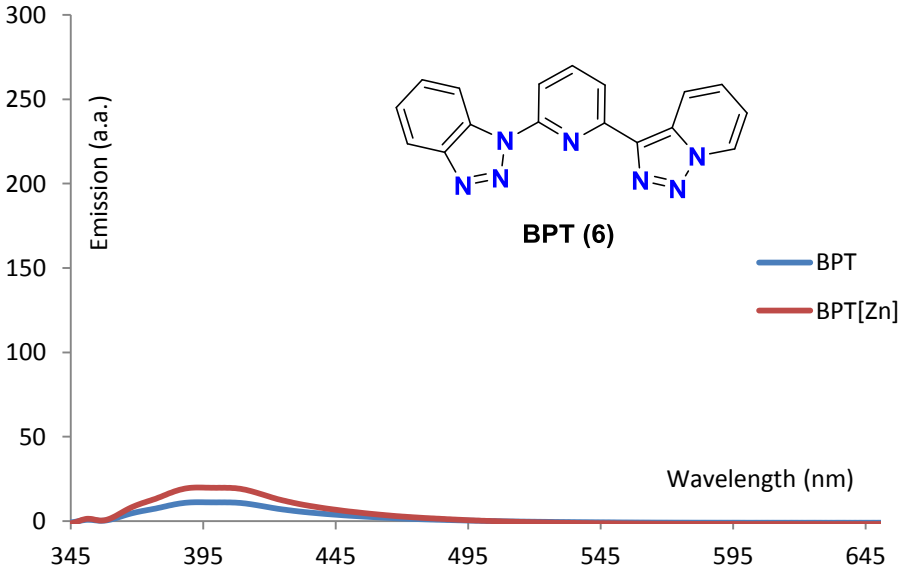




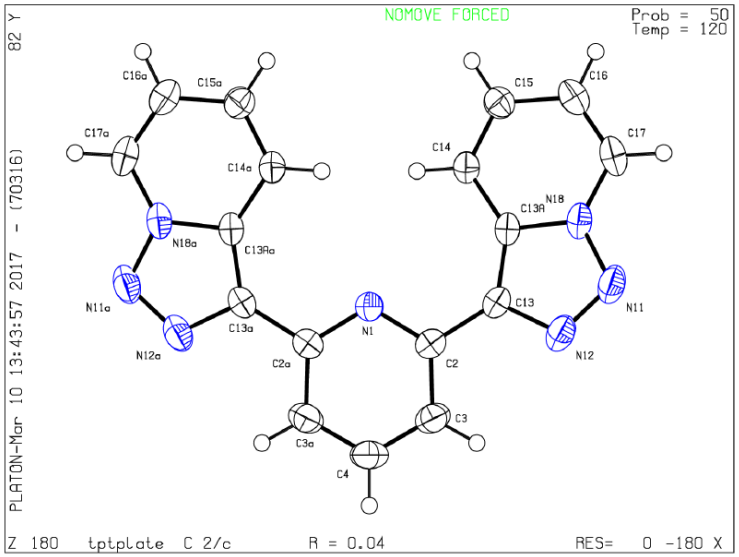
S3: Fluorescence Spectra



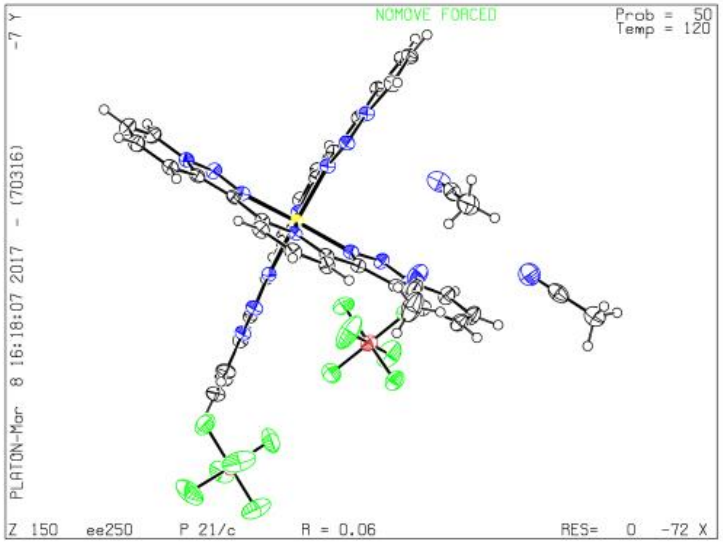




S4 Ortep for TPT and Ru(TPT)$_2$

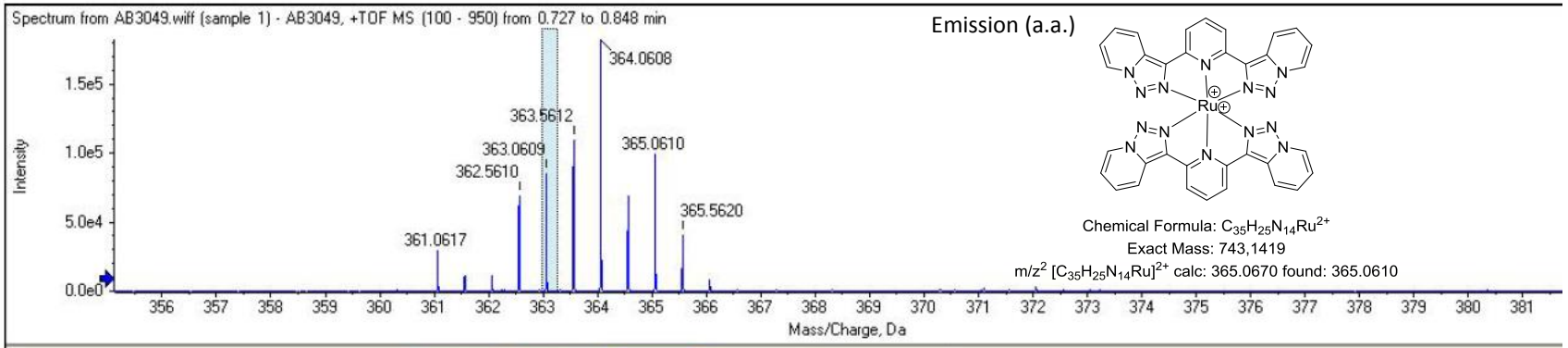


TPT CDCC-1537651



Ru(**TPT**)$_2$ **CDCC**-1537650

S5: HRMS and NMR Spectra of $Ru(TPT)_2$



Emission (a.a.)

Chemical Formula: $C_{35}H_{25}N_{14}Ru^{2+}$
Exact Mass: 743,1419
m/z^2 $[C_{35}H_{25}N_{14}Ru]^{2+}$ calc: 365.0670 found: 365.0610

Found elemental compositions

Find Any | Find

Hit	Formula	m/z	RDB	ppm	MS Rank	MSMS ppm	M R
1	RuC21H22N14F8	363.0607	13.0	-0.1	1		
2	RuC14H27N12F12P	363.0608	1.0	-0.3	2		
3	RuC23H25N8F11	363.0608	9.0	-0.5	3		
4	RuC29H21N14F3	363.0607	24.0	-0.6	4		
5	C32H22N2F12P2	363.0606	18.0	0.8	5		
6	RuC22H26N12F7P	363.0609	12.0	-0.8	6		
7	RuC31H24N8F6	363.0609	20.0	-1.0	7		
8	RuC15H31N10F11P2	363.0611	0.0	-1.1	8		
9	C30H19N8F9P2	363.0605	22.0	1.1	9		
10	RuC24H29N6F10P	363.0610	8.0	-1.3	10		
11	RuC30H25N12F2P	363.0610	23.0	-1.3	11		
12	C28H16N14F6P2	363.0604	26.0	1.5	12		
13	RuC33H27N2F9	363.0610	16.0	-1.5	13		
14	RuC23H30N10F6P2	363.0612	11.0	-1.6	14		
15	C20H17N14F11P2	363.0603	15.0	1.7	15		
16	RuC32H28N6F5P	363.0611	19.0	-1.8	16		
17	C29H15N10F10P	363.0602	23.0	1.8	17		
18	RuC25H33N4F9P2	363.0613	7.0	-2.0	18		
19	RuC31H29N10FP2	363.0612	22.0	-2.1	19		
20	RuC34H31F8P	363.0612	15.0	-2.2	20		
21	RuC33H32N4F4P2	363.0614	18.0	-2.5	21		

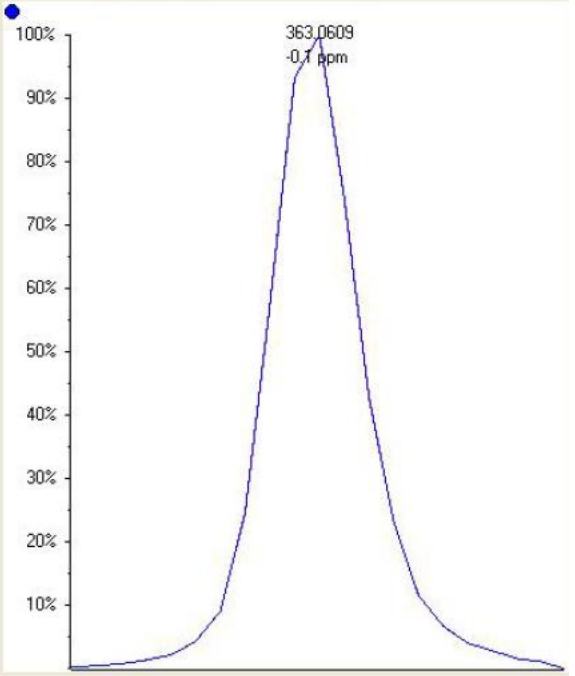
MS Details | MSMS Details | Compound Details

Isotope cluster details — Charge +2

Peak	Use	m/z	% Intensity	Width
0	☑	363.0609	100.0	0.011

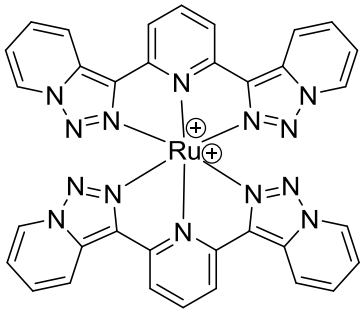
Elements from
Elements to C34H35F12P2N14Ru
Mass tolerance (ppm) 4
Intensity tolerance (%) 50
#C/#heteroatoms greater than 0.01

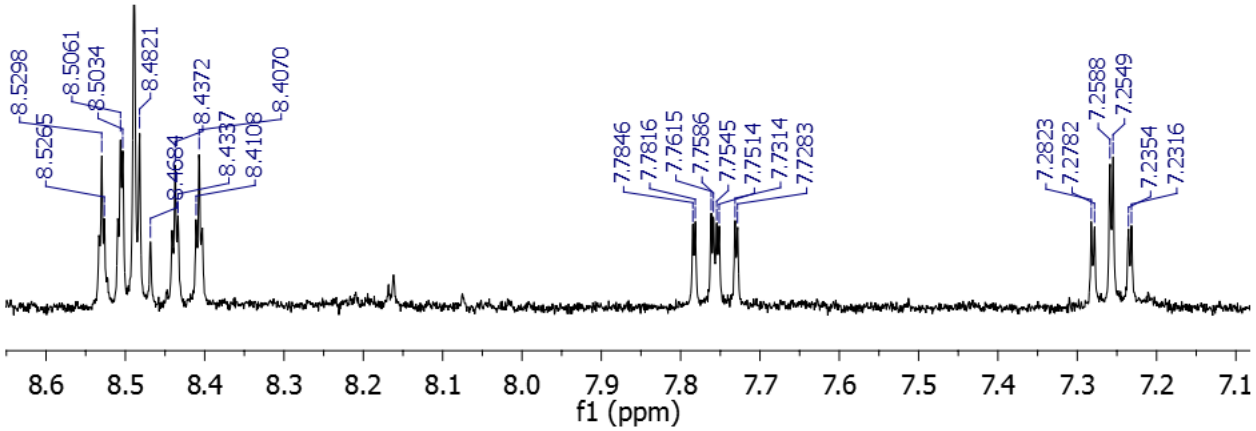
MS result summary for RuC21H24N14F8, [M+2H]++



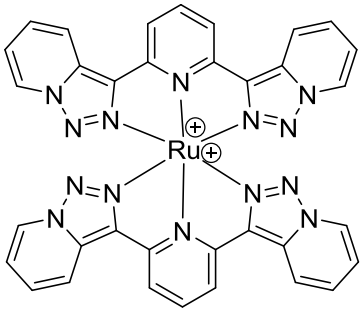
Ion type: [M+2H]++ 2 additional ions...

S5: HRMS and NMR Spectra of $Ru(TPT)_2$





S5: HRMS and NMR Spectra of $Ru(TPT)_2$



CD_3CN

152.5047
140.5921
138.8458
133.0381
131.6198
127.3967
119.3899
119.2279
117.6174

205 200 195 190 185 180 175 170 165 160 155 150 145 140 135 130 125 120 115
f1 (ppm)

152.5047
140.5921
138.8458
133.0381
131.6198
127.3967
119.3899
119.2279
117.6174

Ethylen glycol

220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)