

Electronic Supplementary Material (ESI)

Electrochemical Rhodium Catalyzed Alkyne Annulation with Pyrazoles through Anodic Oxidation – a Metal Oxidant/Additive Free Methodology

Subban Kathiravan* and Prasad Anaspure

Bioorganic & Biophysical Chemistry Laboratory, Linnaeus University Centre for
Biomaterials Chemistry, Department of Chemistry & Biomedical Sciences,
Linnaeus University, Kalmar SE-39182, Sweden
suppan.kathiravan@lnu.se

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1. General considerations:

All the catalytic reactions were conducted under nitrogen atmosphere by using standard Schlenk technique. The solvents and chemicals were purchased from Aldrich and Chemtronica in Sweden. All glassware's dried overnight at 120°C and if needed flame dried further. Column chromatography was performed on silica gel (Carlo Erba, 60Å). Thin layer chromatography was performed on a silica gel precoated on aluminum foils with fluorescence indicator (254 nm). Preparative thin layer chromatography was performed on plates available at Aldrich (Analtech, UV₂₅₄ 20×20 cm, 500 micron). Yields refer to isolated compounds and ¹H NMR determined their purity.

Nuclear magnetic resonance (NMR) spectroscopy was performed at 400 MHz (¹H NMR), 101 MHz (¹³C NMR), and 376 MHz (¹⁹F NMR) on Bruker Ascend 400 instrument. Chemical shifts (δ) are provided in ppm and spectra referred to non-deuterated solvent signal.

Mass spectra (HRMS) were obtained from Lund University Kemi Centrum Mass Spectrometry facility. Instrument: Waters XEVO-G2 QTOF. ESI+: Capillary voltage 3 kV, Cone voltage 35V, Ext 4, Source Temp 120, Des Temp 300, Cone gas 50, Des gas 400. Continuum resolution mode, m/z 100-1200, manual lock mass correction by Leucine Enkephalin (m/z 556.2771).

Electrochemical reactions were carried out in undivided electrochemical cells (50 mL) using pre-dried glassware. RVC electrodes were obtained from SGL carbon, Wiesbaden, Germany and used with the following dimensions: (30 mm × 5 mm). Platinum electrodes (99.9 %) were

obtained from ChemPur Karlsruhe, Germany and used with the following dimensions: (10 mm × 6 mm). Ni foam electrodes were obtained from RECEMAT BV, The Netherlands and used with the following dimensions (30 mm × 6 mm). Electrocatalysis was conducted using GAMRY instruments Reference 600 & 600 plus potentiostat in constant current mode.

Substituted pyrazoles **1** were prepared according to the reported methods.¹ Alkyne **2a**, **4g-4k** was commercially available and used as received. Alkynes **4a-f** were synthesized according to the reported methods.²

2. Description of electrochemical setup



Figure S1: Glassware setup for electrochemical reactions

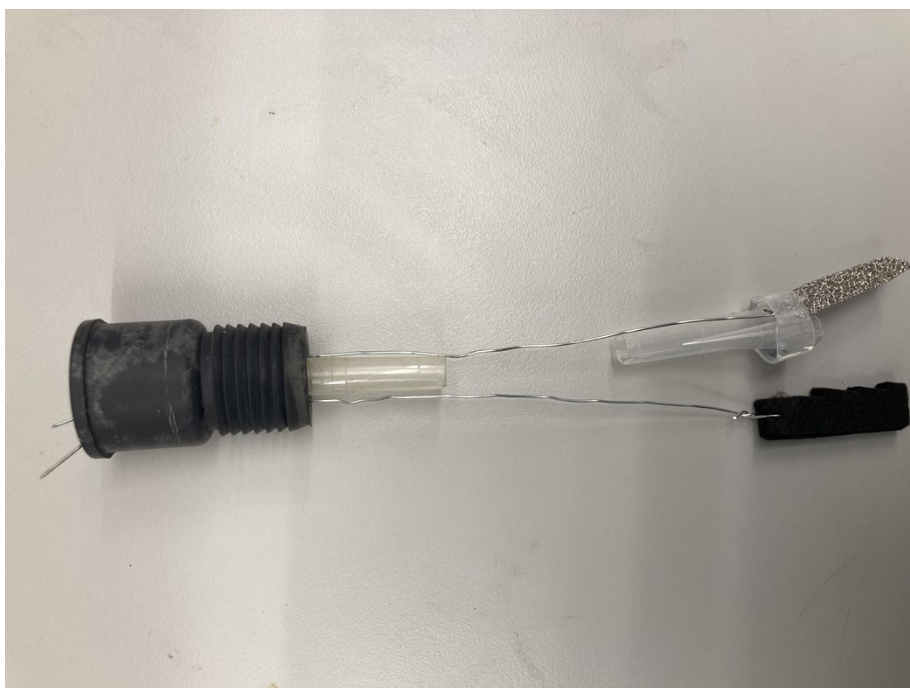


Figure S2: Arrangement of RVC and Ni electrodes

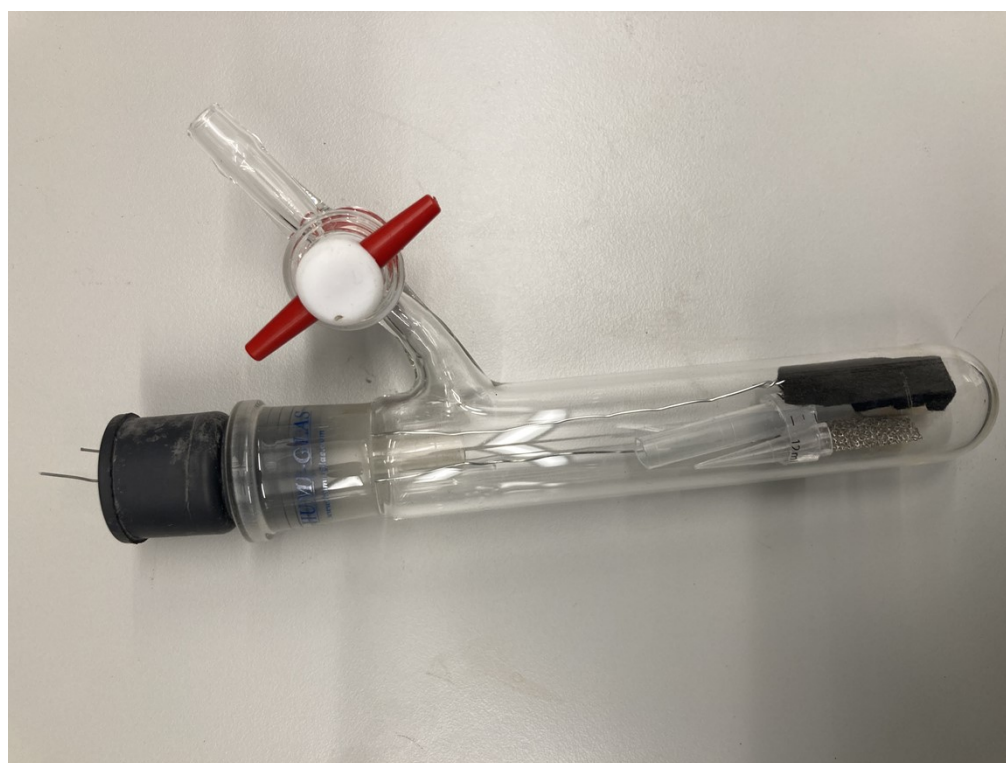


Figure S3: Final setup for reactions



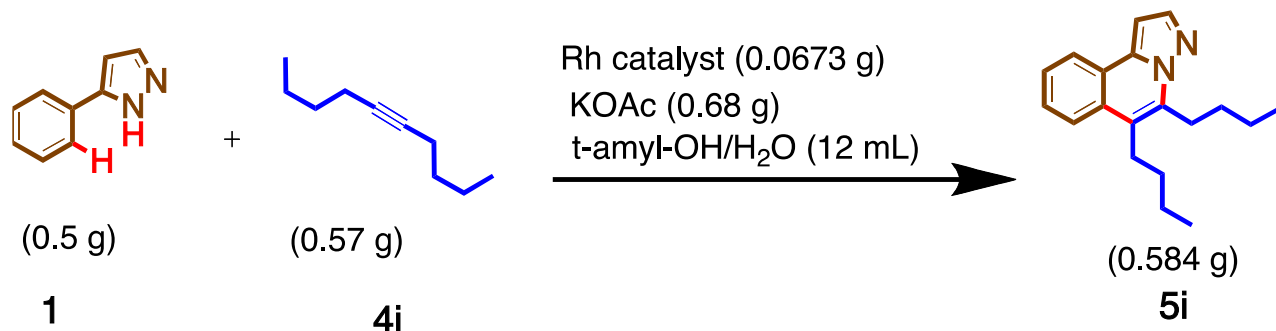
Figure S4: Electrochemical station with GAMRY 600 Plus potentiostat



Figure S5: Electrochemical station with GAMRY 600 potentiostat

4. E-factor calculation

To evaluate the efficacy of this green methodology for the alkyne annulation with pyrazole, we undertook a study to calculate the E-factor associated with this reaction and found the total amount of 18.97, which is quite good, factor in comparison to the conventional methods.



Total amount of reactants: 0.5 g + 0.57 g + 0.0673 g + 0.68 g + 9.849 g = 11.663 g

Amount of final product: 0.584 g

Amount of waste: (11.663-0.584) g = 11.079 g

E-factor = Amount of waste/Amount of product = 11.079/0.584 = 18.97

5. Cyclic voltammetry studies

Cyclic voltammetry were recorded with a Gamry potentiostat at room temperature. A Ni foam plate was used as counter electrode, a RVC electrode was used as the working electrode, and Ag wire electrode was used as the reference. The measurements were carried out at a scan rate of 100 mV/s.

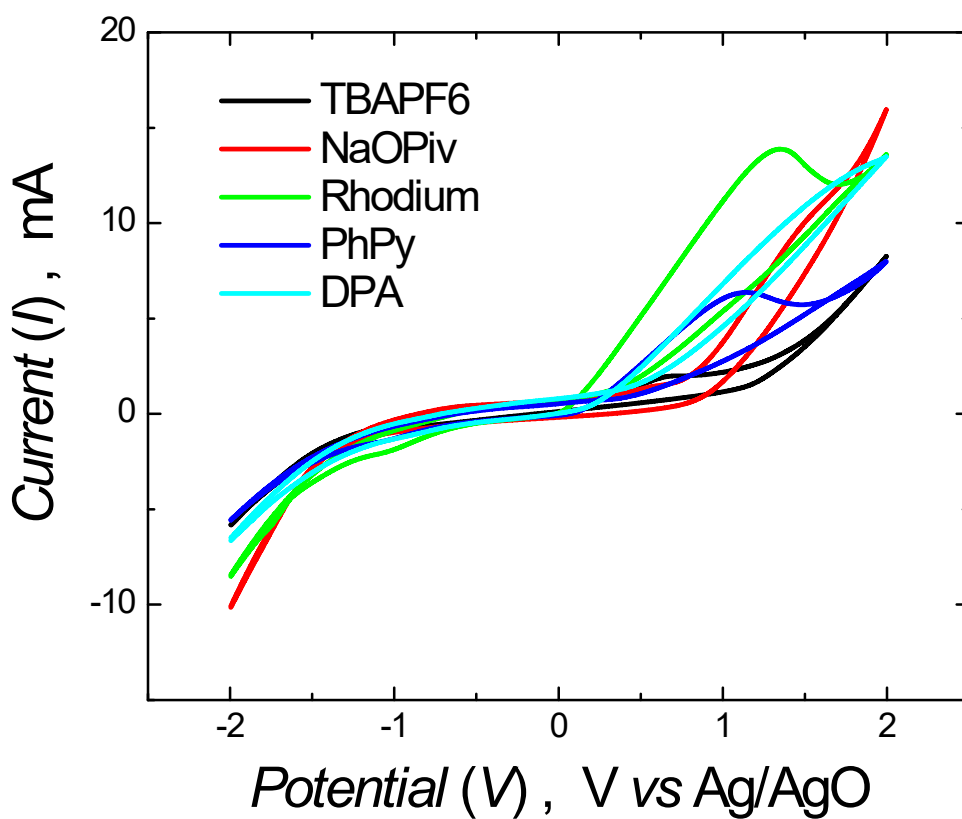
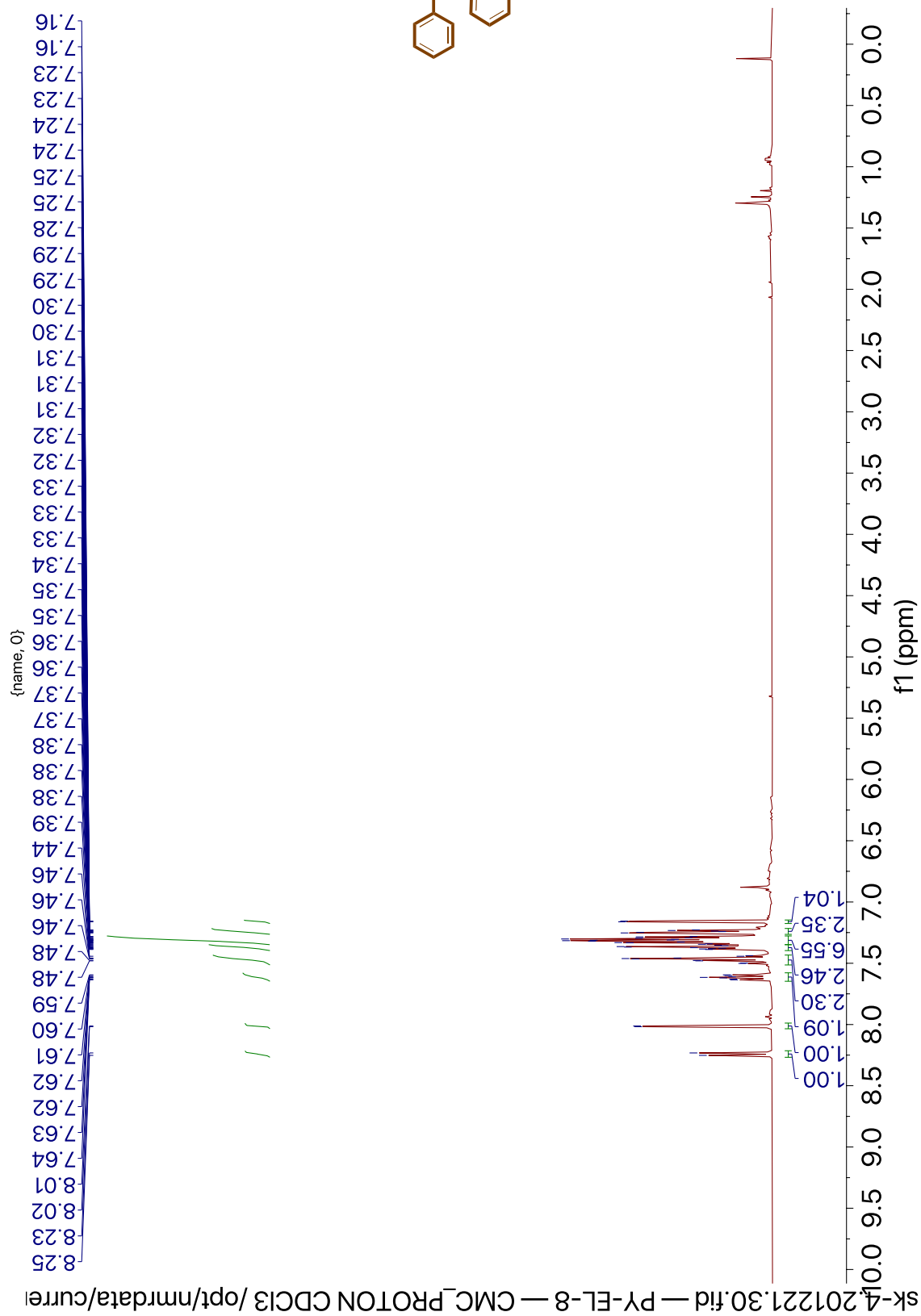
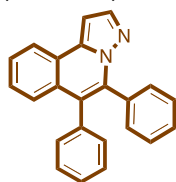


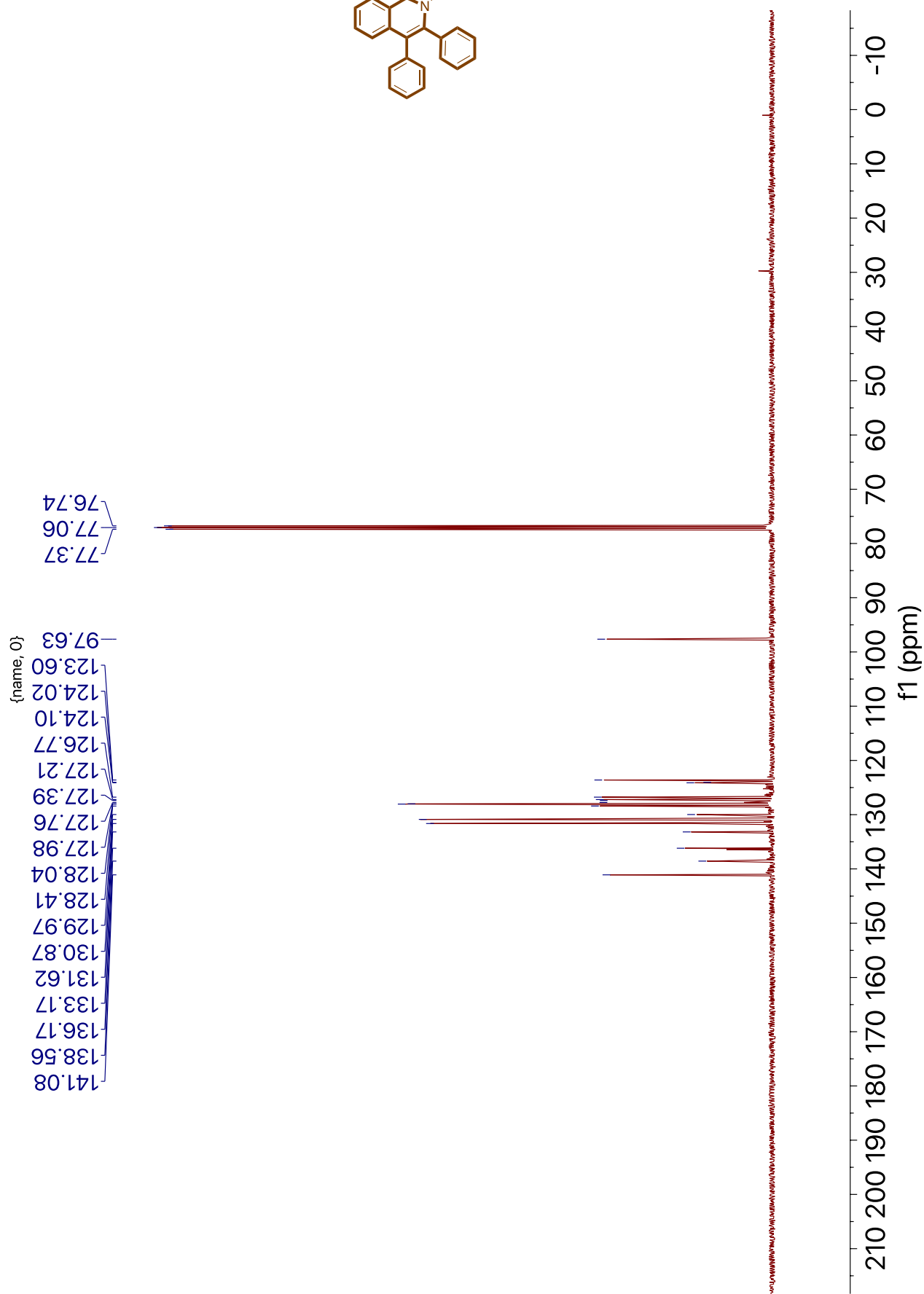
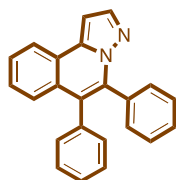
Figure S7: Cyclic voltammetry at 100 mV/s: $n\text{Bu}_4\text{NPF}_6$ (100 mM in MeCN), concentration of substrates (KOAc 100 mM), $[\text{Cp}^*\text{RhCl}_2]_2$ catalyst - 5 mM, 2-phenyl pyrazole - 10 mM, diphenylacetylene - 10 mM.

6. Copies of Spectra

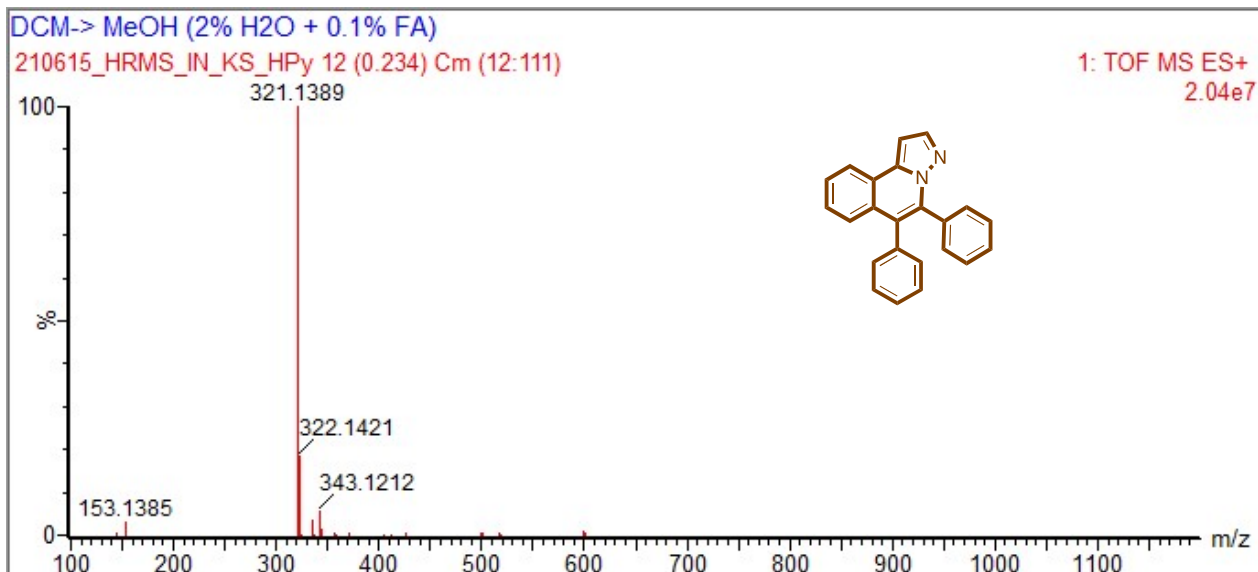
Compound 3a, ¹H NMR, CDCl₃, 400 MHz



Compound 3a, ^{13}C NMR, CDCl_3 , 101 MHz



sk.201221.31.fid - PY-EL-8 - C13CPD CDCl3 /opt/nmrdata/current_data.sk



Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

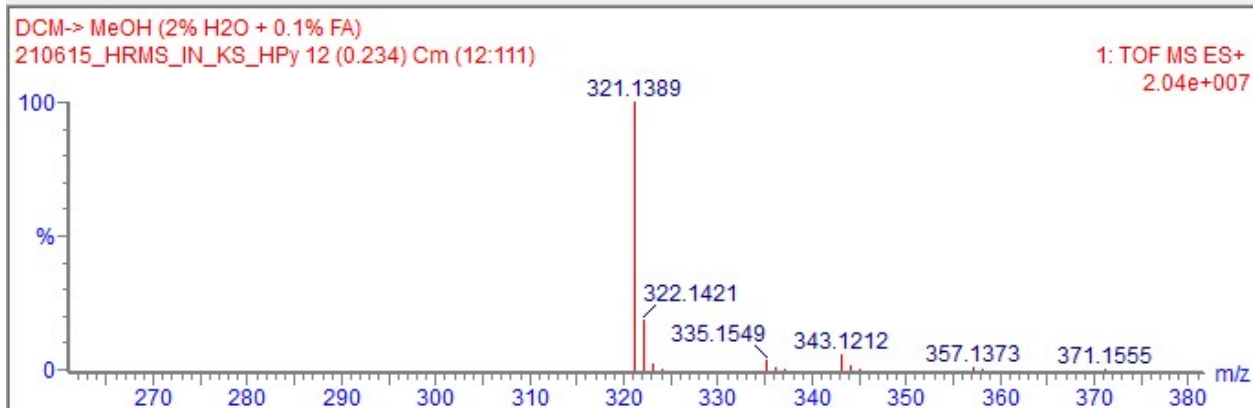
Monoisotopic Mass, Even Electron Ions

200 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

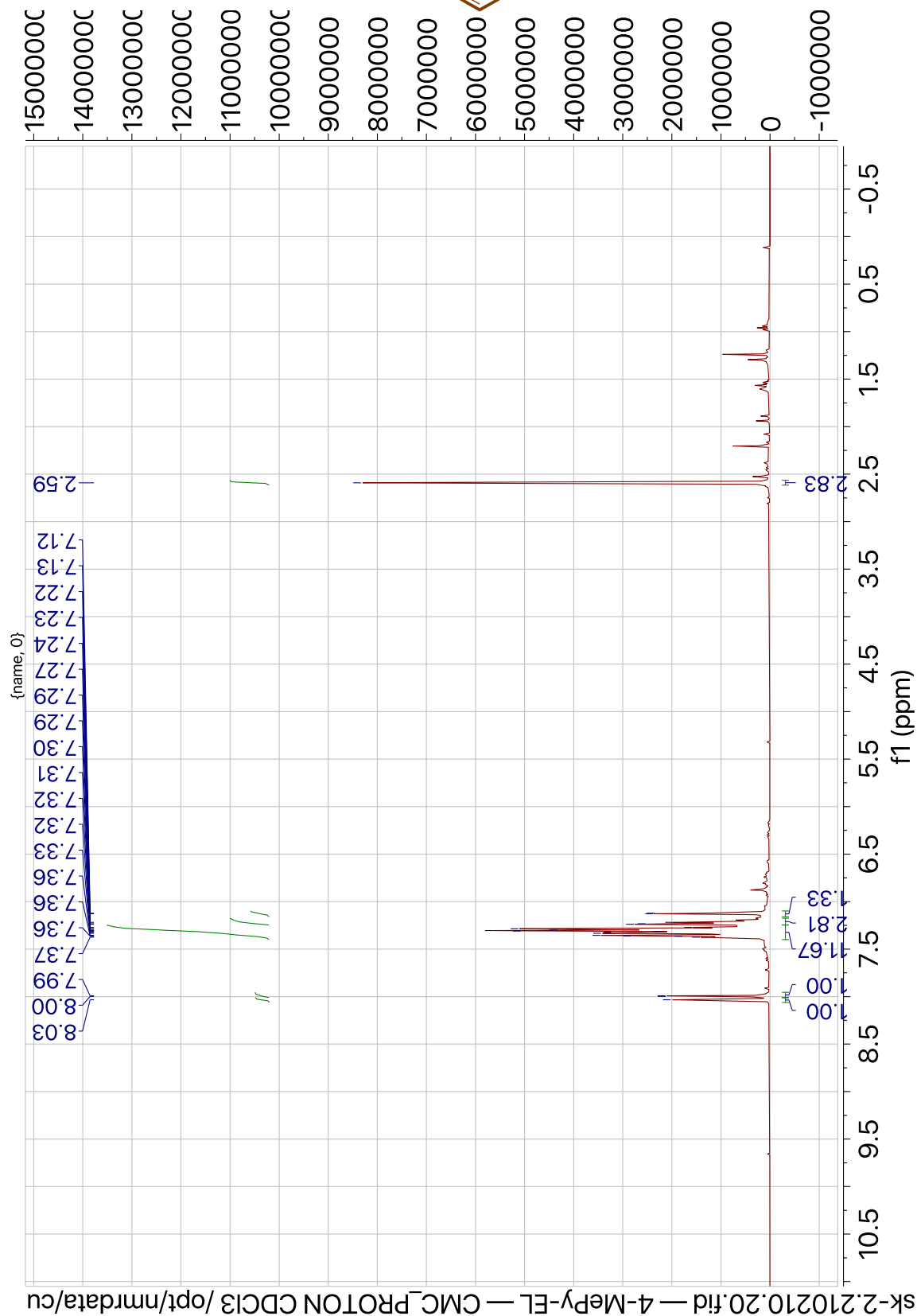
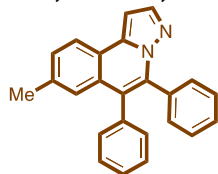
C: 0-50 H: 0-100 N: 0-5 O: 0-3 Na: 0-1

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	N	O	Na
321.1389	321.1392	-0.3	-0.9	16.5	C23 H17 N2	39.1	n/a	n/a	23	17	2		

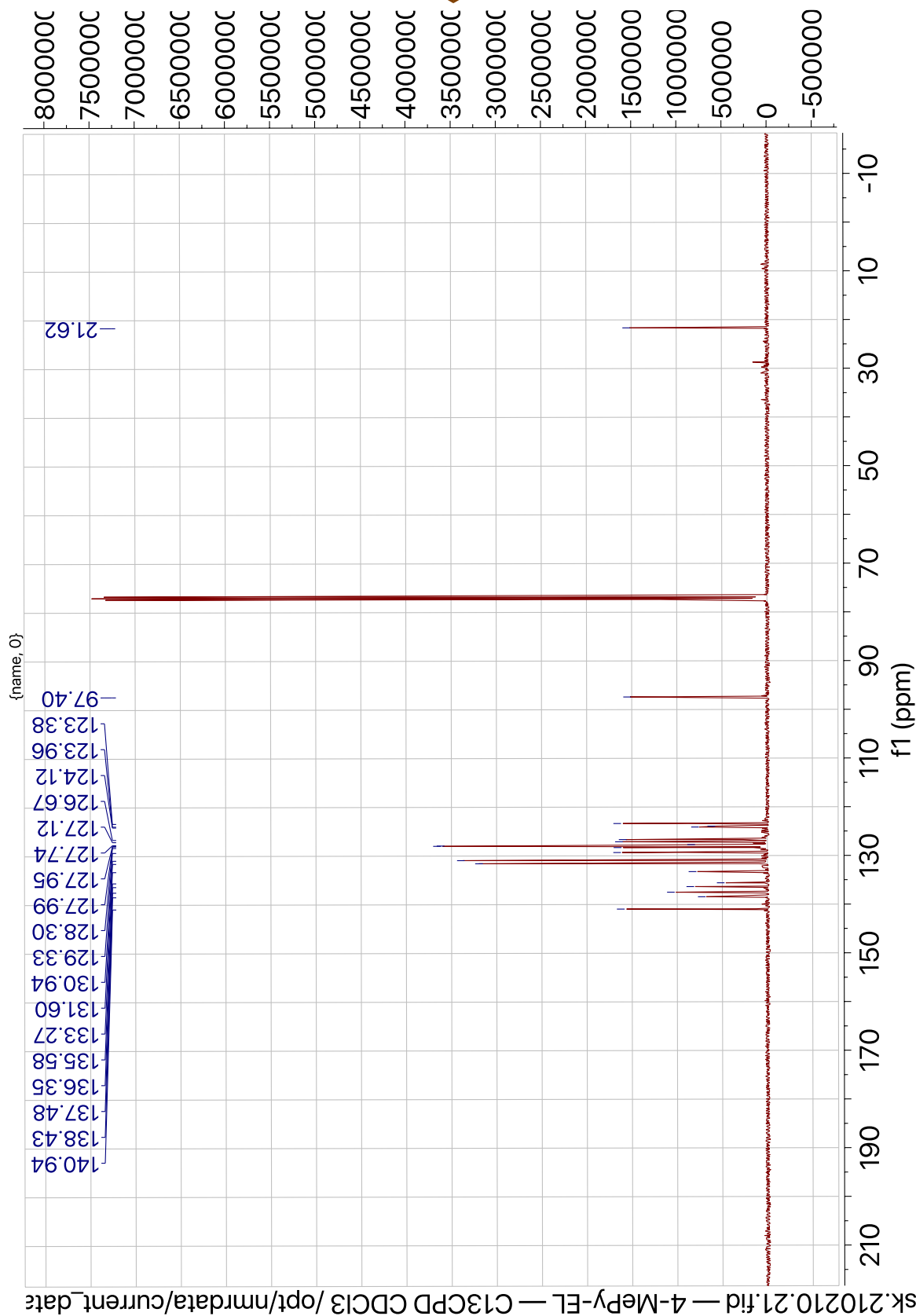
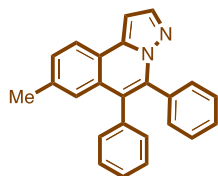


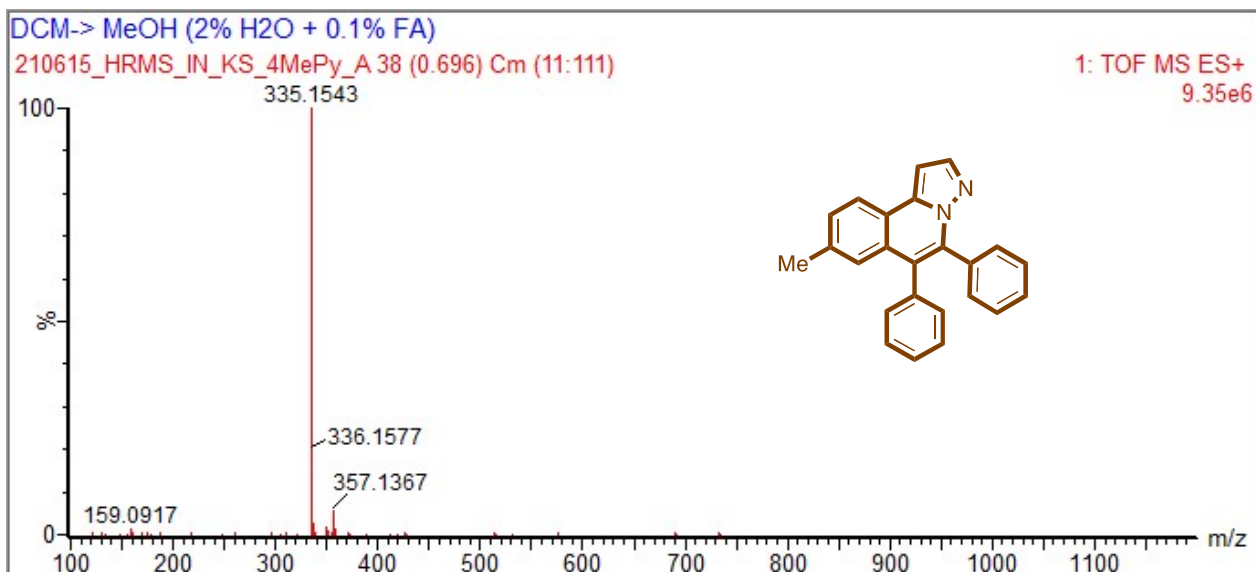
HRMS spectra of **3a**

Compound 3b, ¹H NMR, CDCl₃, 400 MHz



Compound 3b, ¹³C NMR, CDCl₃, 101 MHz





Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

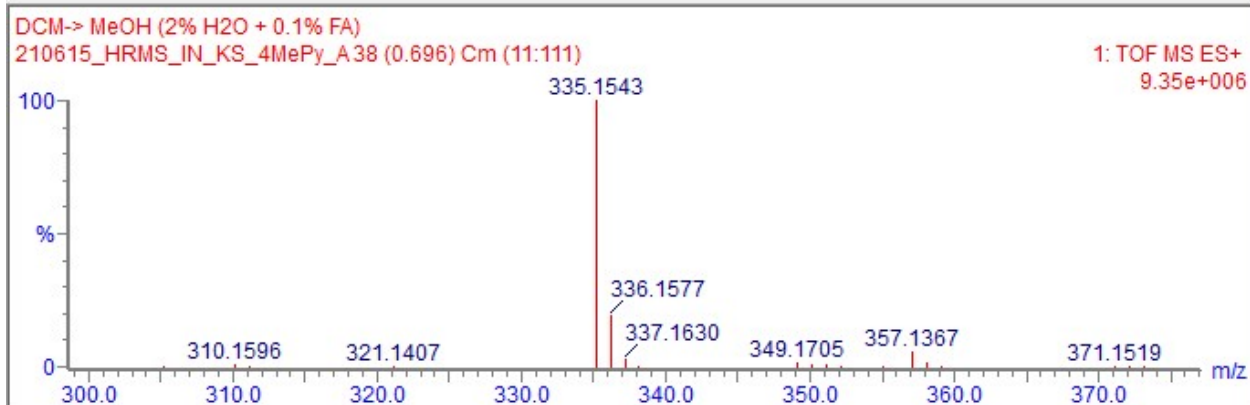
Monoisotopic Mass, Even Electron Ions

208 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

Elements Used:

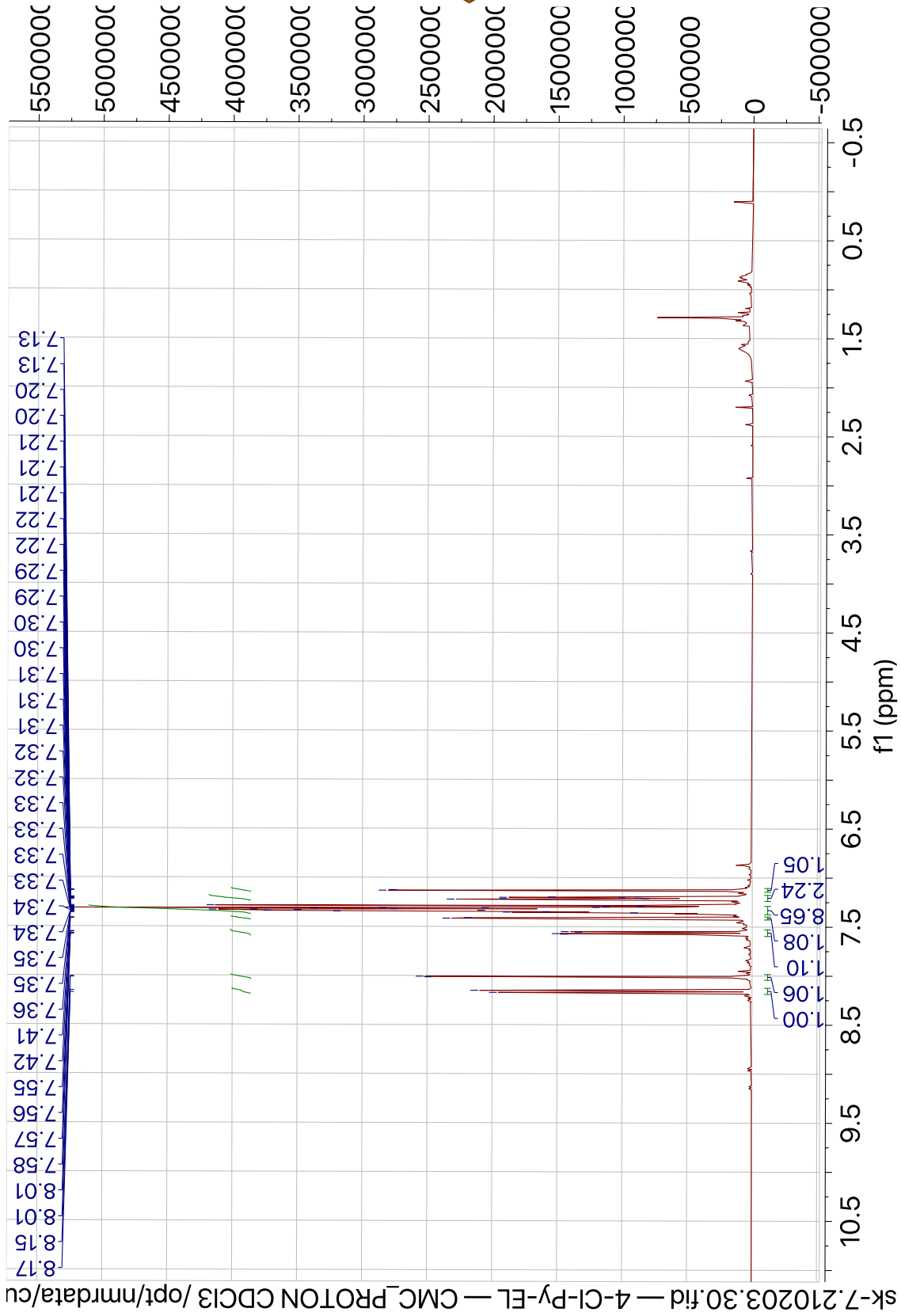
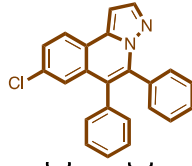
C: 0-50 H: 0-100 N: 0-5 O: 0-3 Na: 0-1

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i...	Fit Conf %	C	H	N	O	Na
335.1543	335.1548	-0.5	-1.5	16.5	C ₂₄ H ₁₉ N ₂	3...	32.42	24	19	2		
	335.1524	1.9	5.7	13.5	C ₂₂ H ₂₀ N ₂ Na	B...	67.58	22	20	2		1

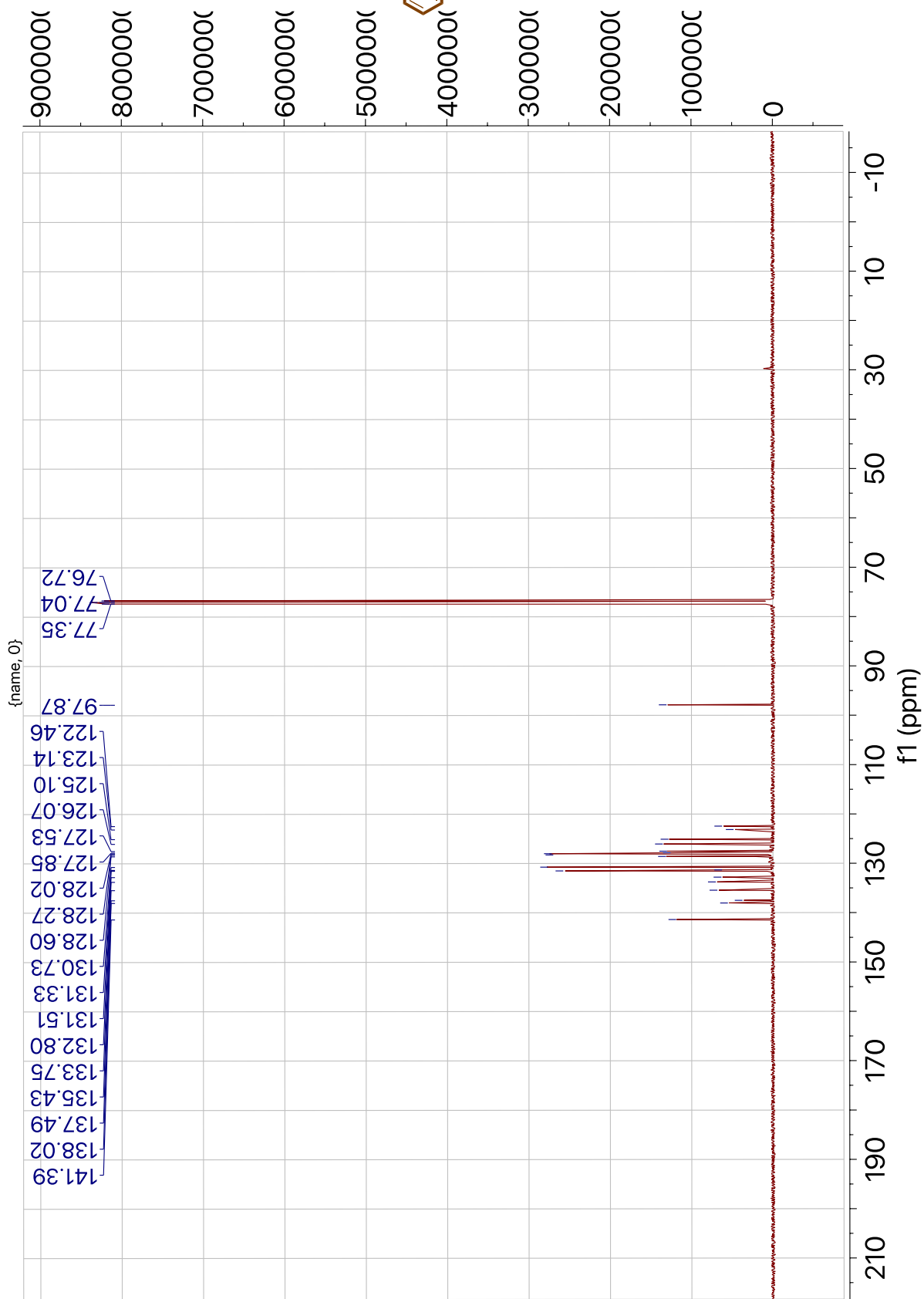
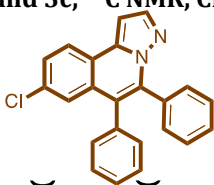


HRMS spectra of **3b**

Compound 3c, ¹H NMR, CDCl₃, 400 MHz



Compound 3c, ¹³C NMR, CDCl₃, 101 MHz

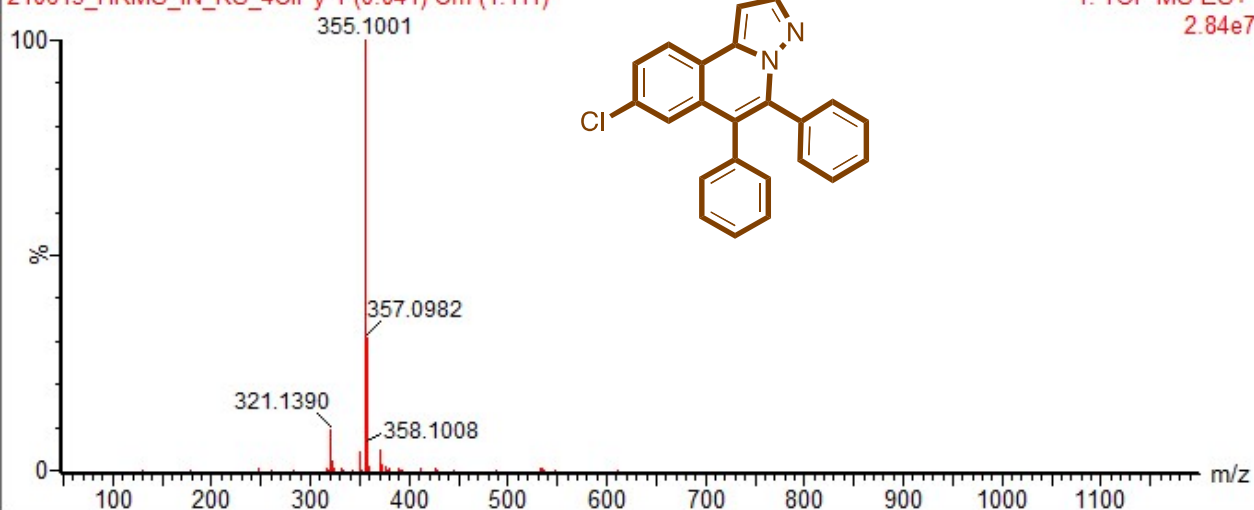


sk.210203.31.fid - 4-Cl-Py-EL - C13CPD CDCl3 /opt/nmrdata/current_data

DCM-> MeOH (2% H2O + 0.1% FA)

210615_HRMS_IN_KS_4CIPy 1 (0.041) Cm (1:111)

1: TOF MS ES+
2.84e7



Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

413 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

Elements Used:

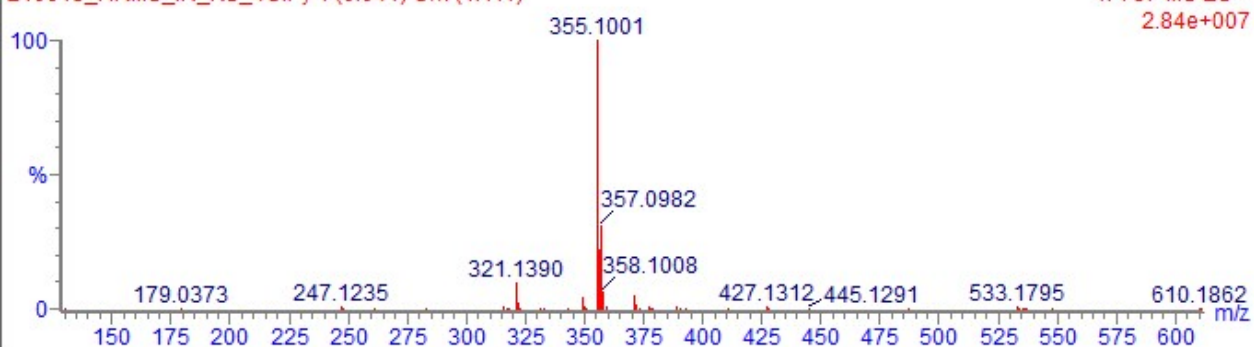
C: 0-50 H: 0-100 N: 0-5 O: 0-3 Na: 0-1 Cl: 0-1

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-...	Fit Conf %	C	H	N	O	Na	Cl
355.1001	355.1002	-0.1	-0.3	16.5	C23 H16 N2 Cl	6.0...	100.00	23	16	2			1
	355.0984	1.7	4.8	21.5	C24 H11 N4	7.1...	0.00	24	11	4			

DCM-> MeOH (2% H2O + 0.1% FA)

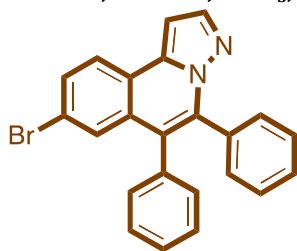
210615_HRMS_IN_KS_4CIPy 1 (0.041) Cm (1:111)

1: TOF MS ES+
2.84e+007

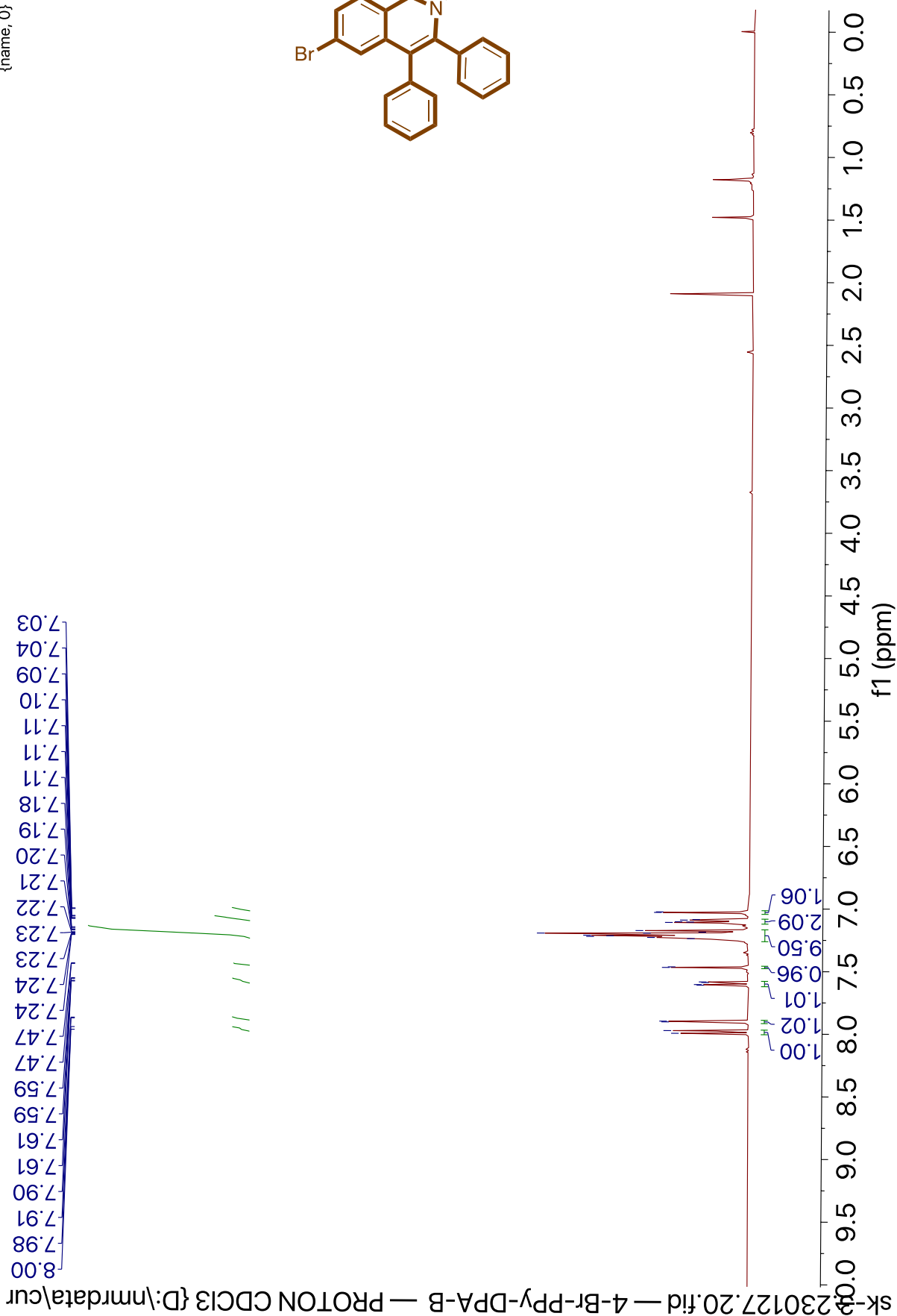


HRMS spectra of **3c**

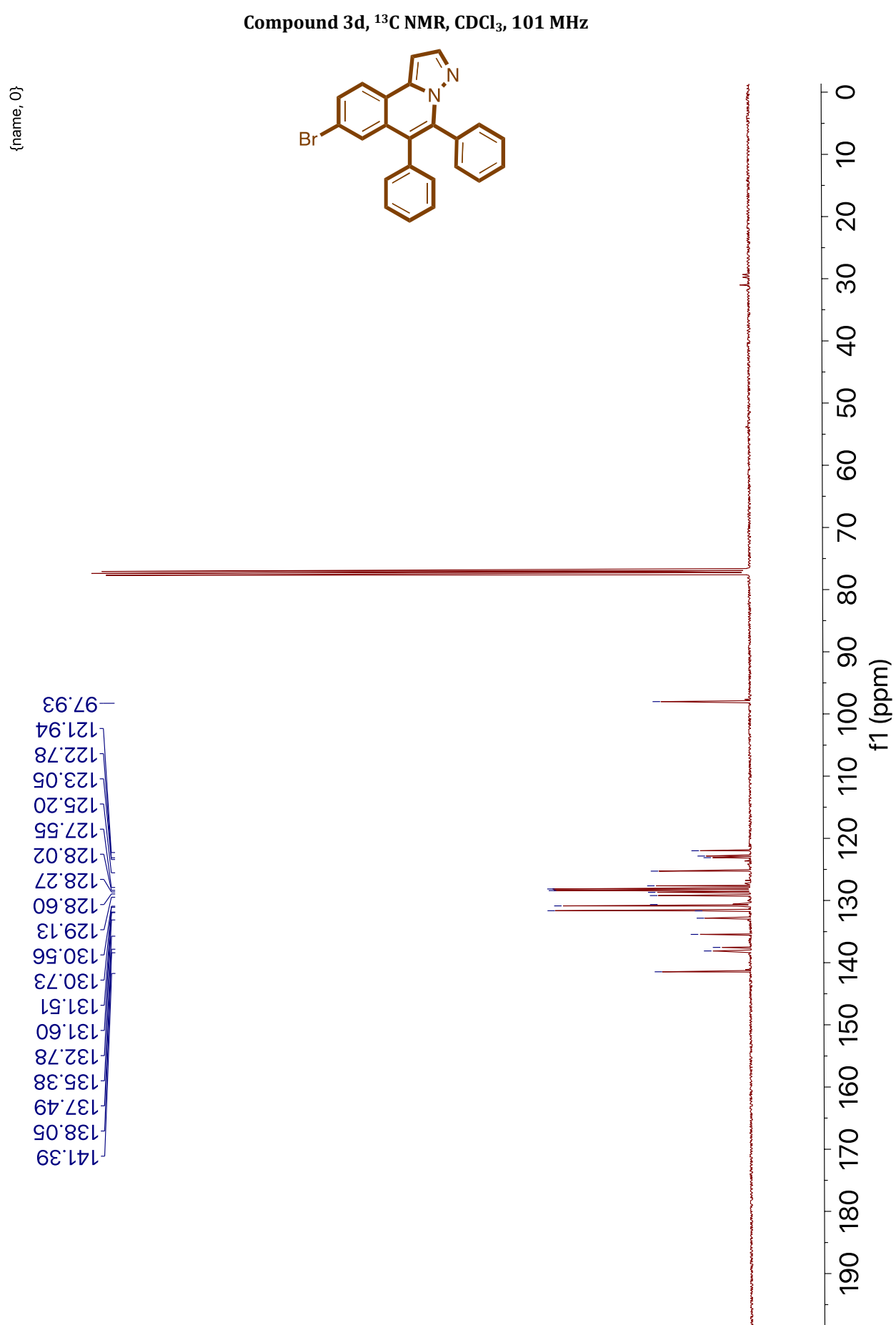
Compound 3d, ¹H NMR, CDCl₃, 400 MHz



{name, 0}



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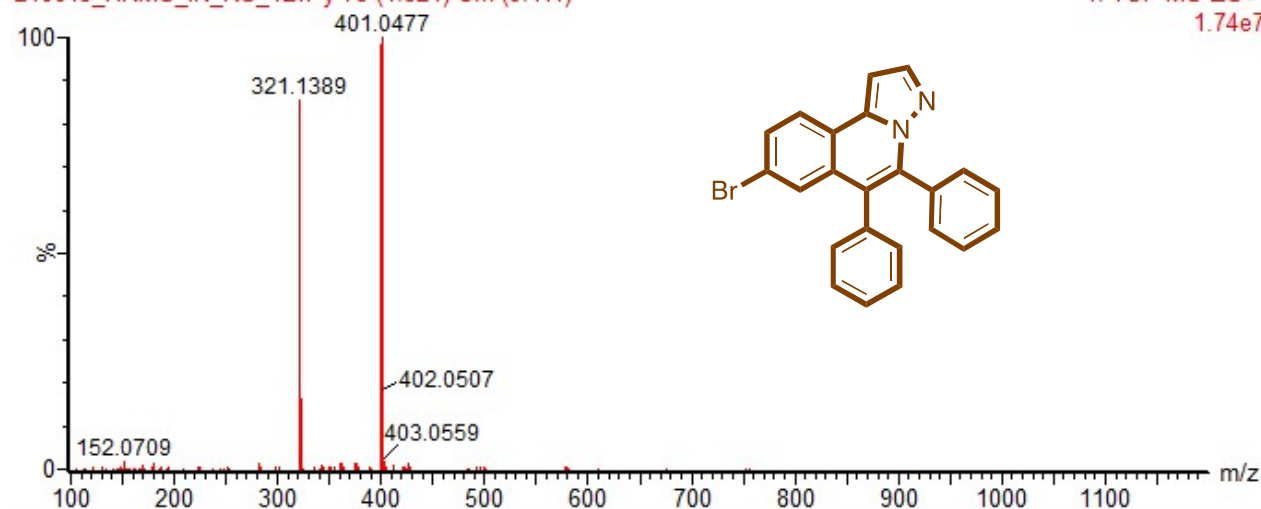


{name, 0}

DCM-> MeOH (2% H2O + 0.1% FA)

210615_HRMS_IN_KS_4BrPy 73 (1.324) Cm (9:111)

1: TOF MS ES+
1.74e7



Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

439 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-50

H: 0-100

N: 0-5

O: 0-3

Na: 0-1

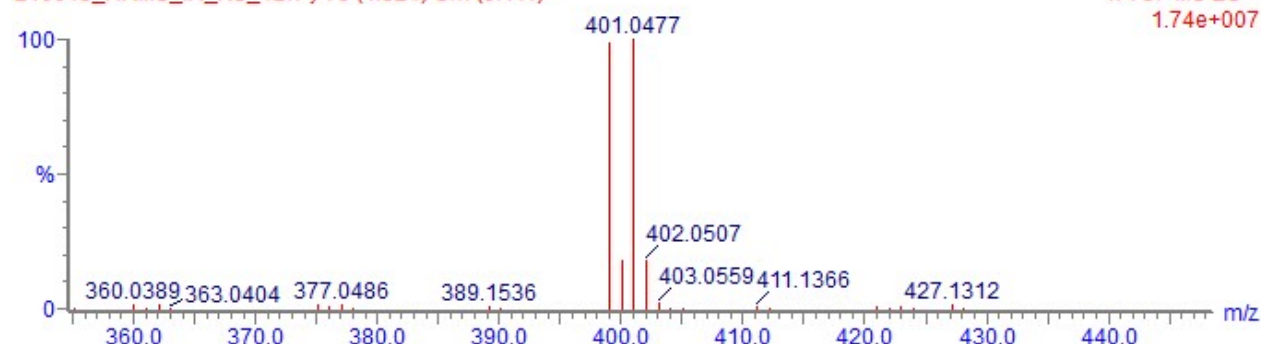
Br: 0-1

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-Fl...	Fit Conf %	C	H	N	O	Na	Br
399.0493	399.0494	-0.1	-0.3	20.5	C22 H8 N4 O3 Na	51...	0.00	22	8	4	3	1	
	399.0497	-0.4	-1.0	16.5	C23 H16 N2 Br		100.00	23	16	2			1

DCM-> MeOH (2% H2O + 0.1% FA)

210615_HRMS_IN_KS_4BrPy 73 (1.324) Cm (9:111)

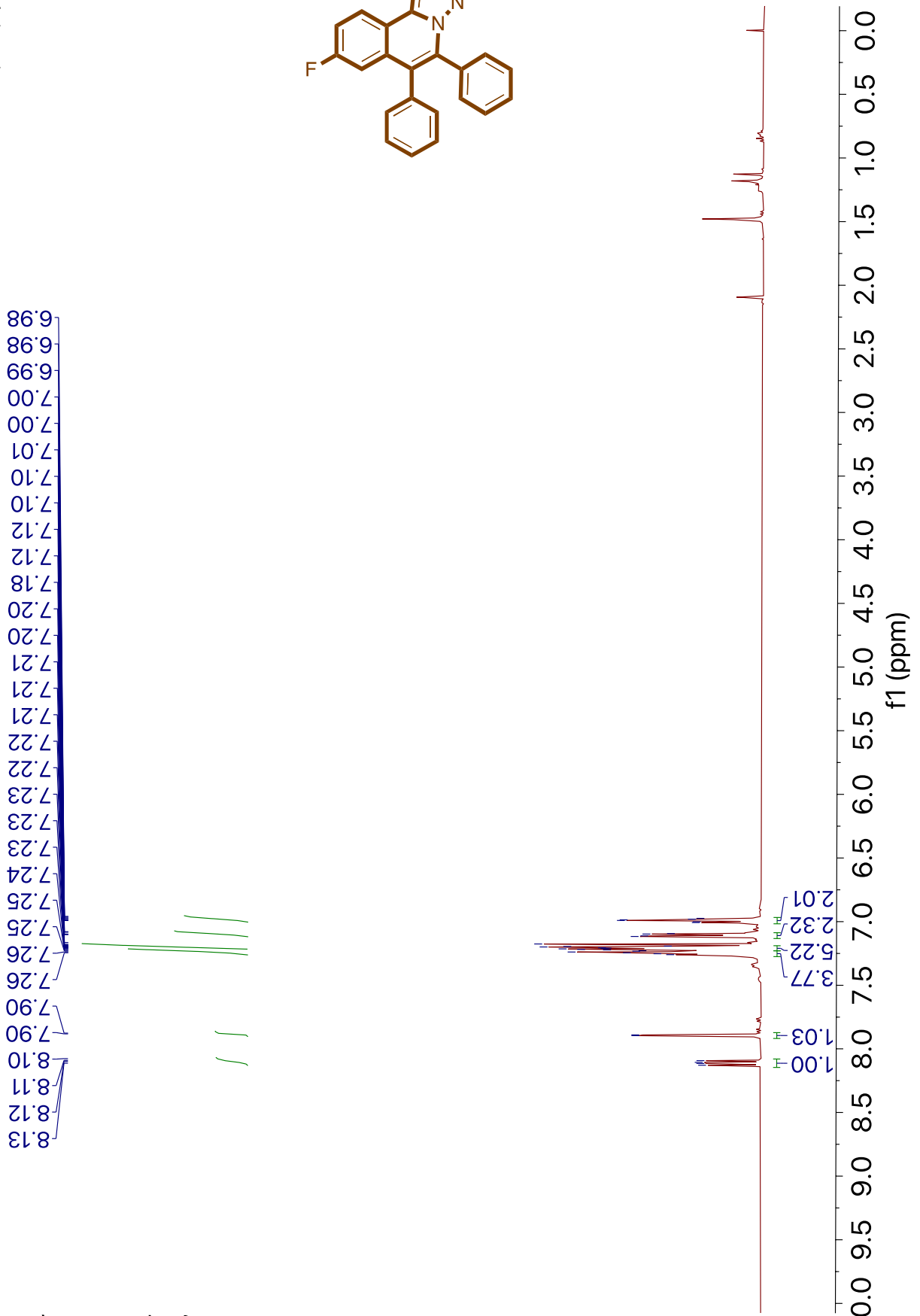
1: TOF MS ES+
1.74e+007



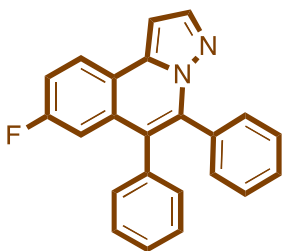
HRMS spectra of **3d**

sk-2230126_30.fid — 4-F-PY-DPA-B — PROTON CDCl3 {D:\nmrdata\curr

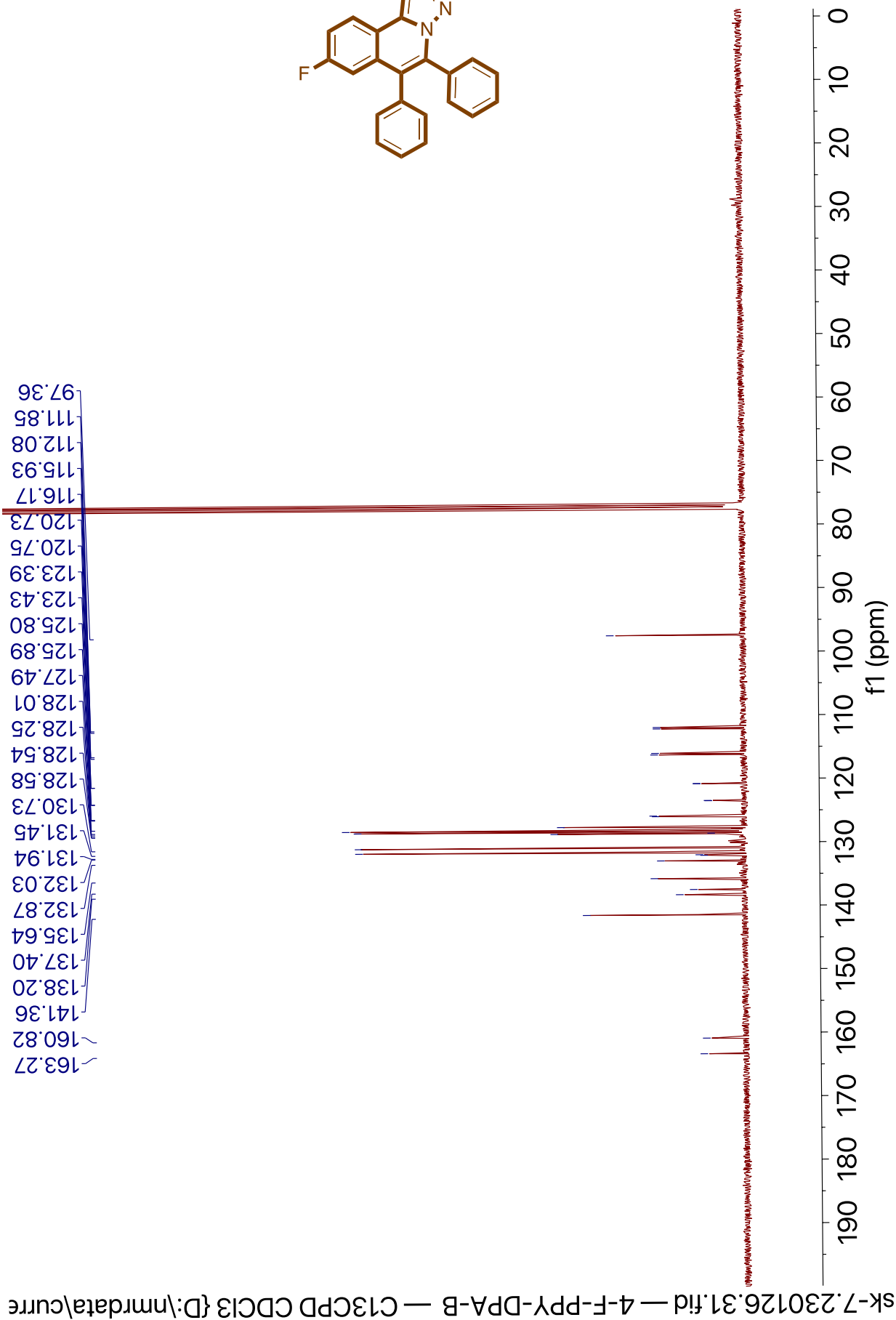
{name, 0}



Compound 3e, ¹³C NMR, CDCl₃, 101 MHz

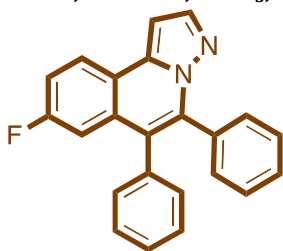


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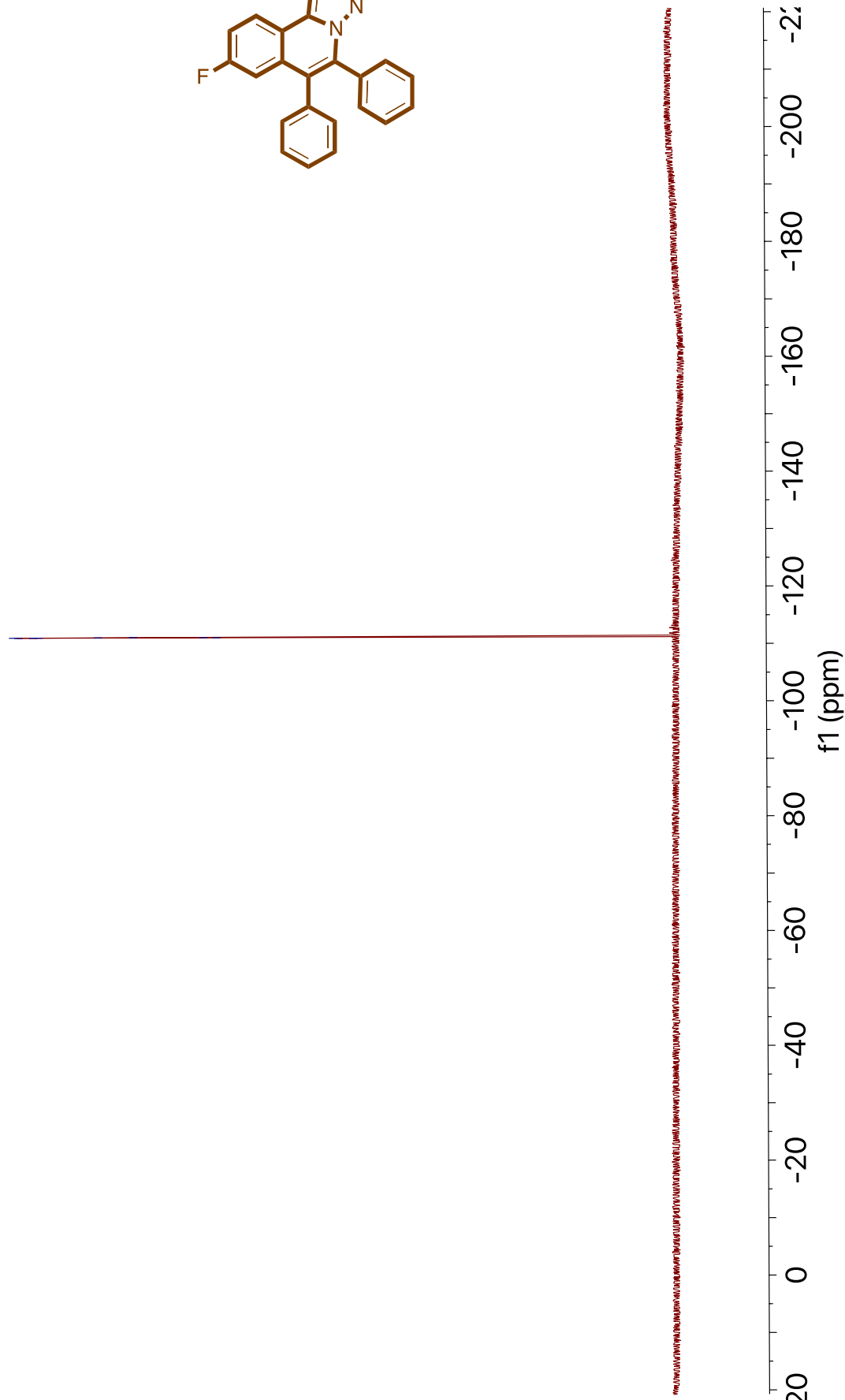


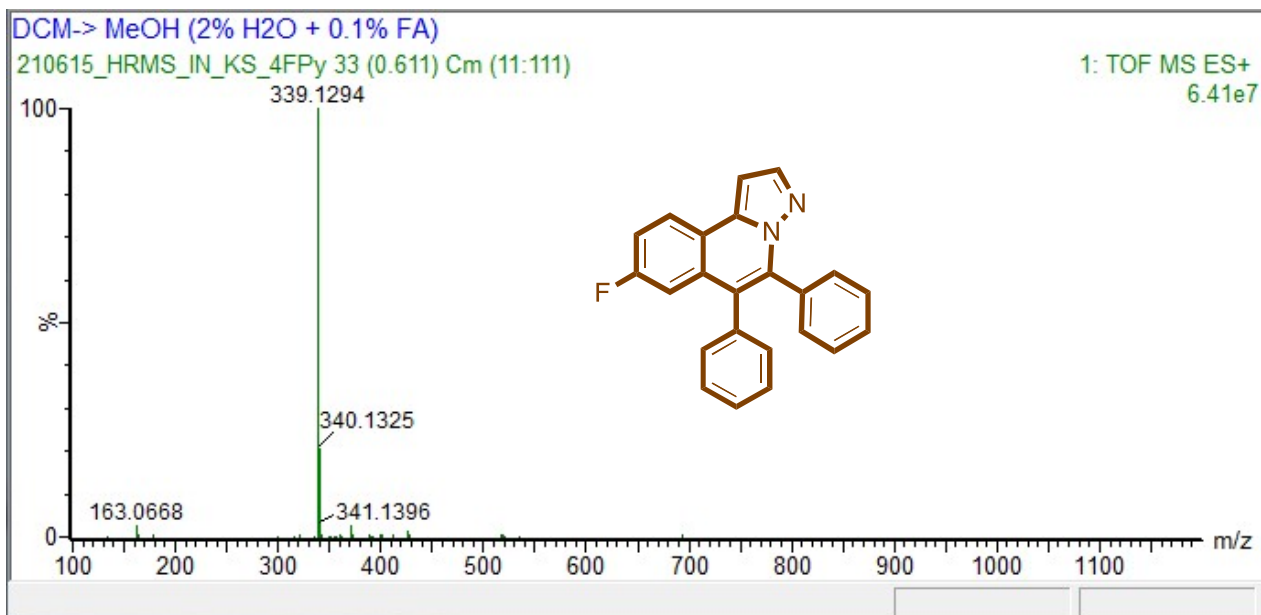
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Compound 3e, ¹⁹F NMR, CDCl₃, 375 MHz



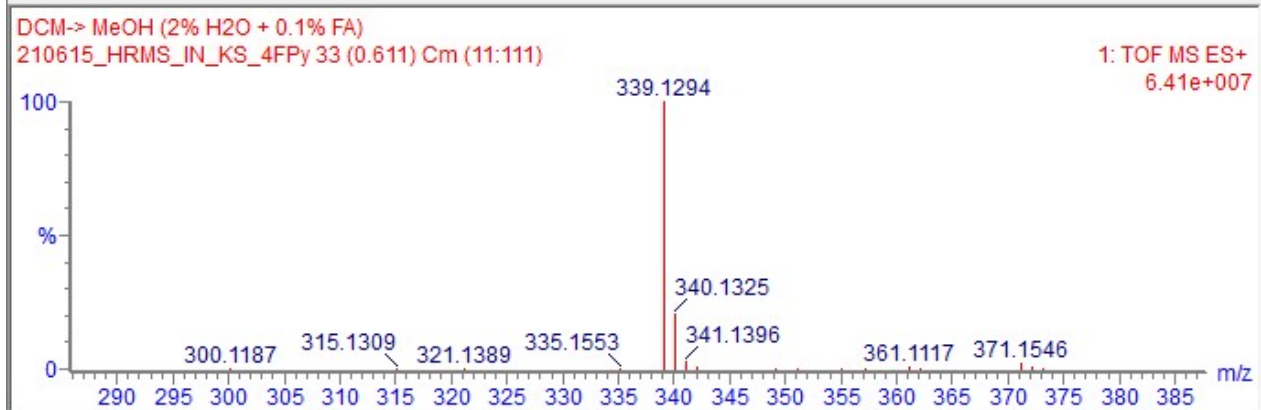
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-111.49





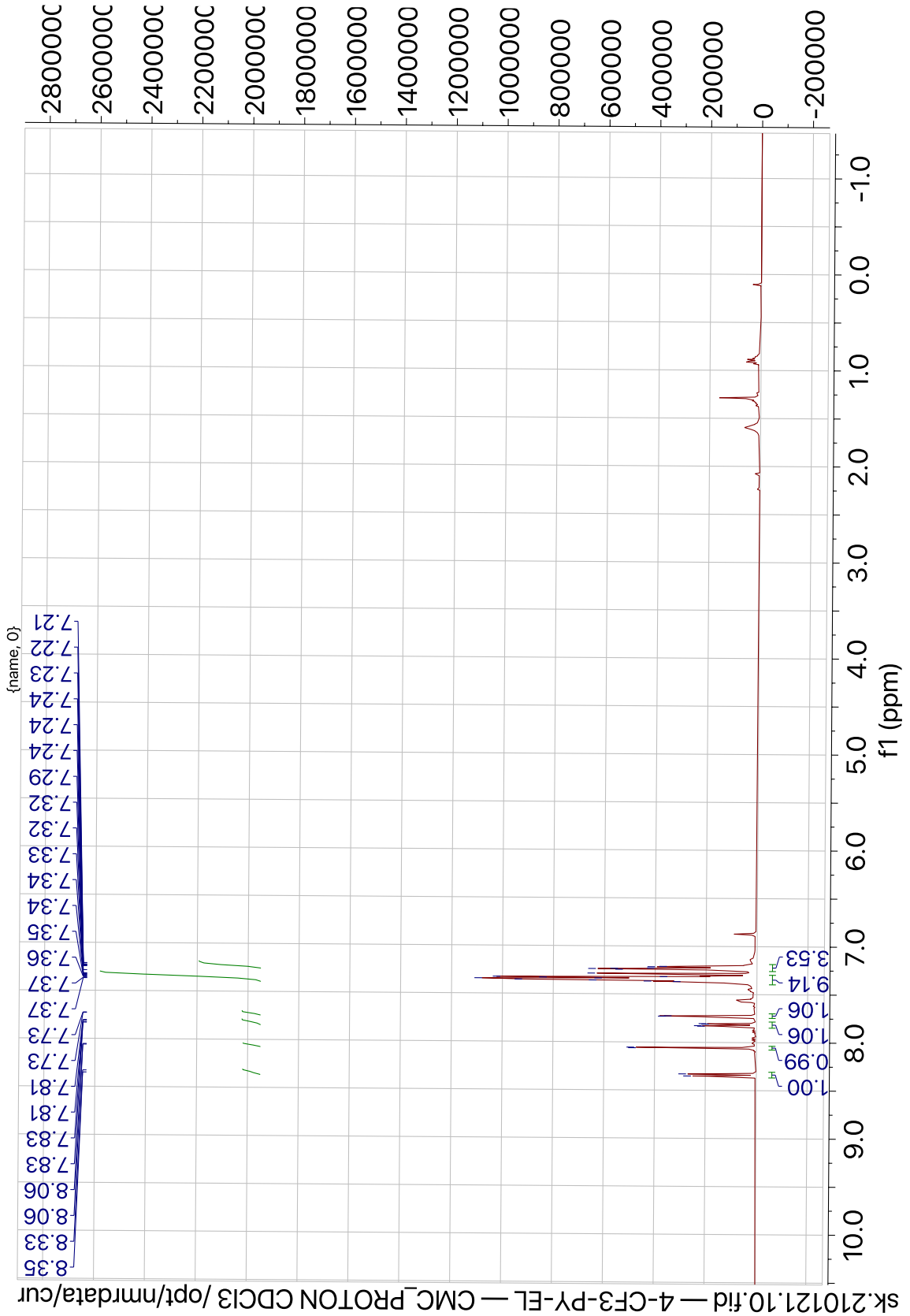
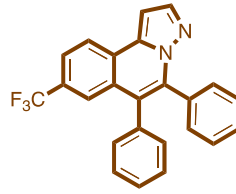
Single Mass Analysis
 Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0
 Element prediction: Off
 Number of isotope peaks used for i-FIT = 3
 Monoisotopic Mass, Even Electron Ions
 404 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
 Elements Used:
 C: 0-50 H: 0-100 N: 0-5 O: 0-3 F: 0-1 Na: 0-1

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-FI...	Fit Conf %	C	H	N	O	F	Na
339.1294	339.1298	-0.4	-1.2	16.5	C23 H16 N2 F	n/a	n/a	23	16	2		1	

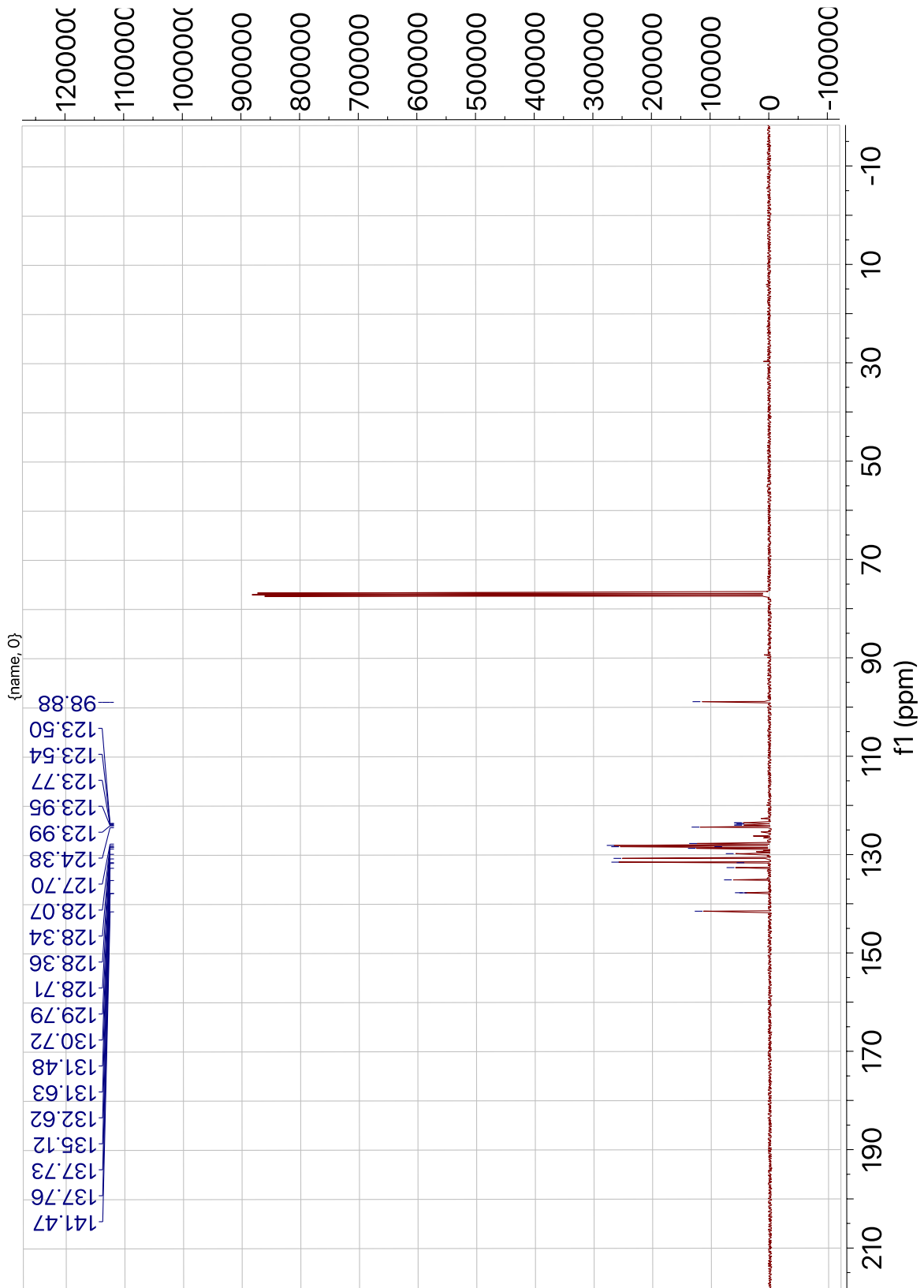
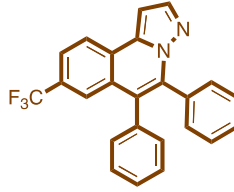


HRMS spectra of **3e**

Compound 3f, ¹H NMR, CDCl₃, 400 MHz

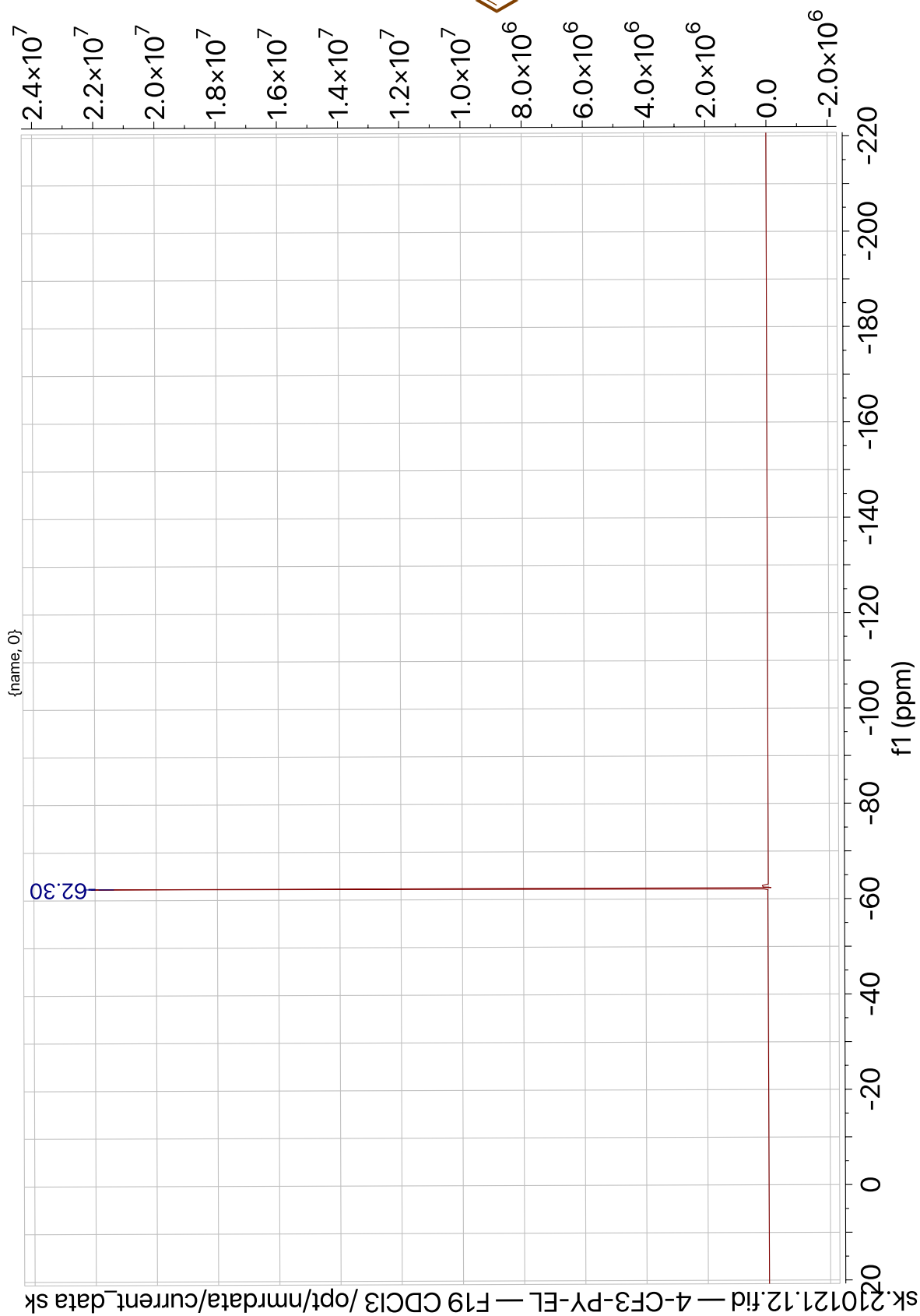
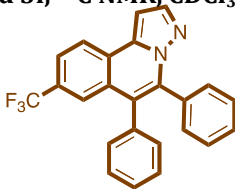


Compound 3f, ¹³C NMR, CDCl₃, 101 MHz



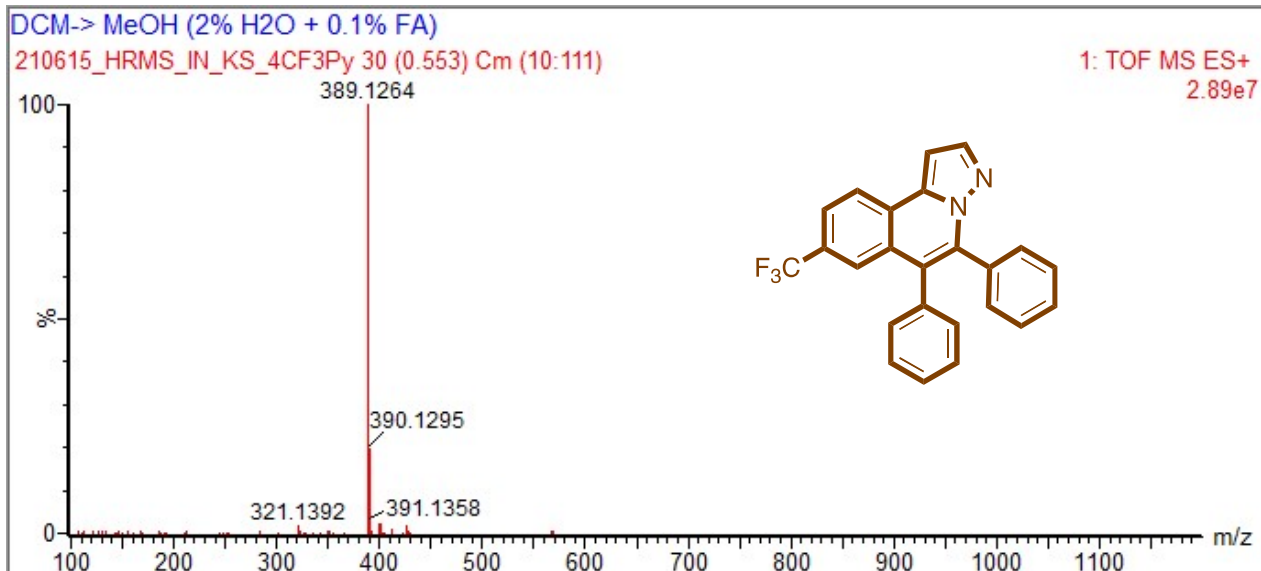
sk.210121.11.fid - 4-CF3-PY-EL - C13CPD CDCl3 /opt/nmrdata/current_da

Compound 3f, ^{19}C NMR, CDCl_3 , 101 MHz



{name, 0}

sk.210121.12.fid - 4-CF3-PY-EL - F19 CDCl3 /opt/nmrdata/current_data sk



Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

404 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-50

H: 0-100

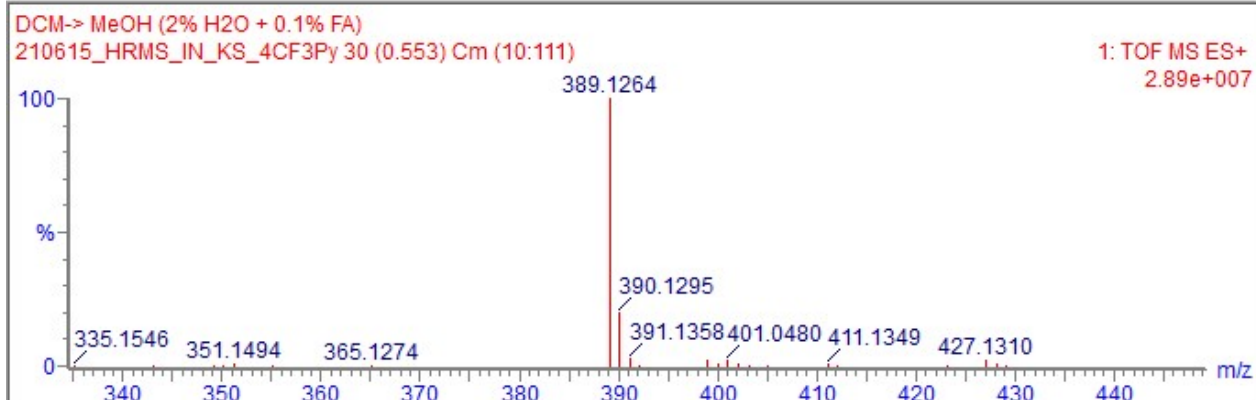
N: 0-5

O: 0-3

F: 2-3

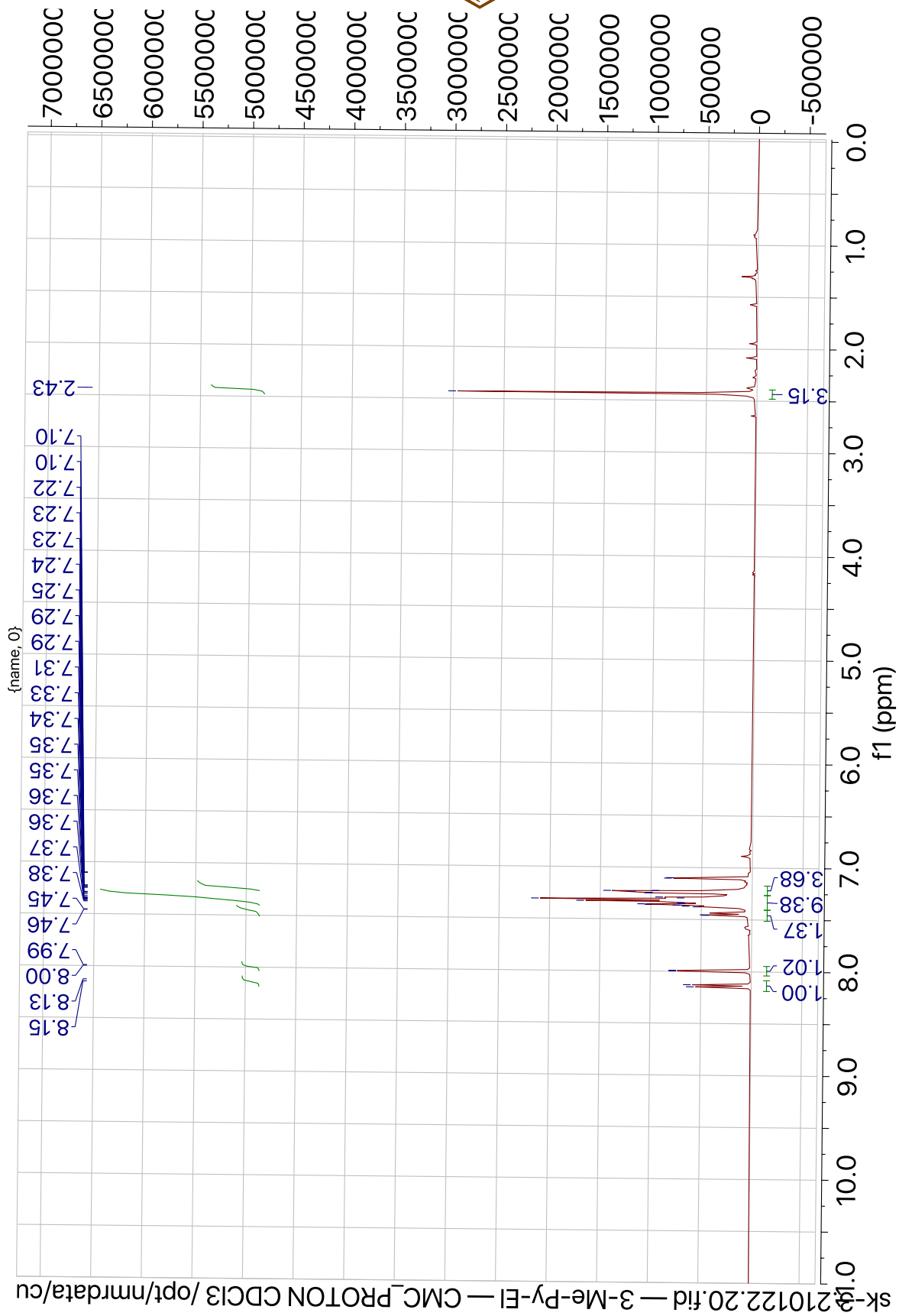
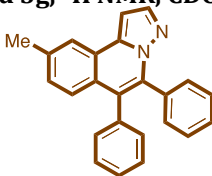
Na: 0-1

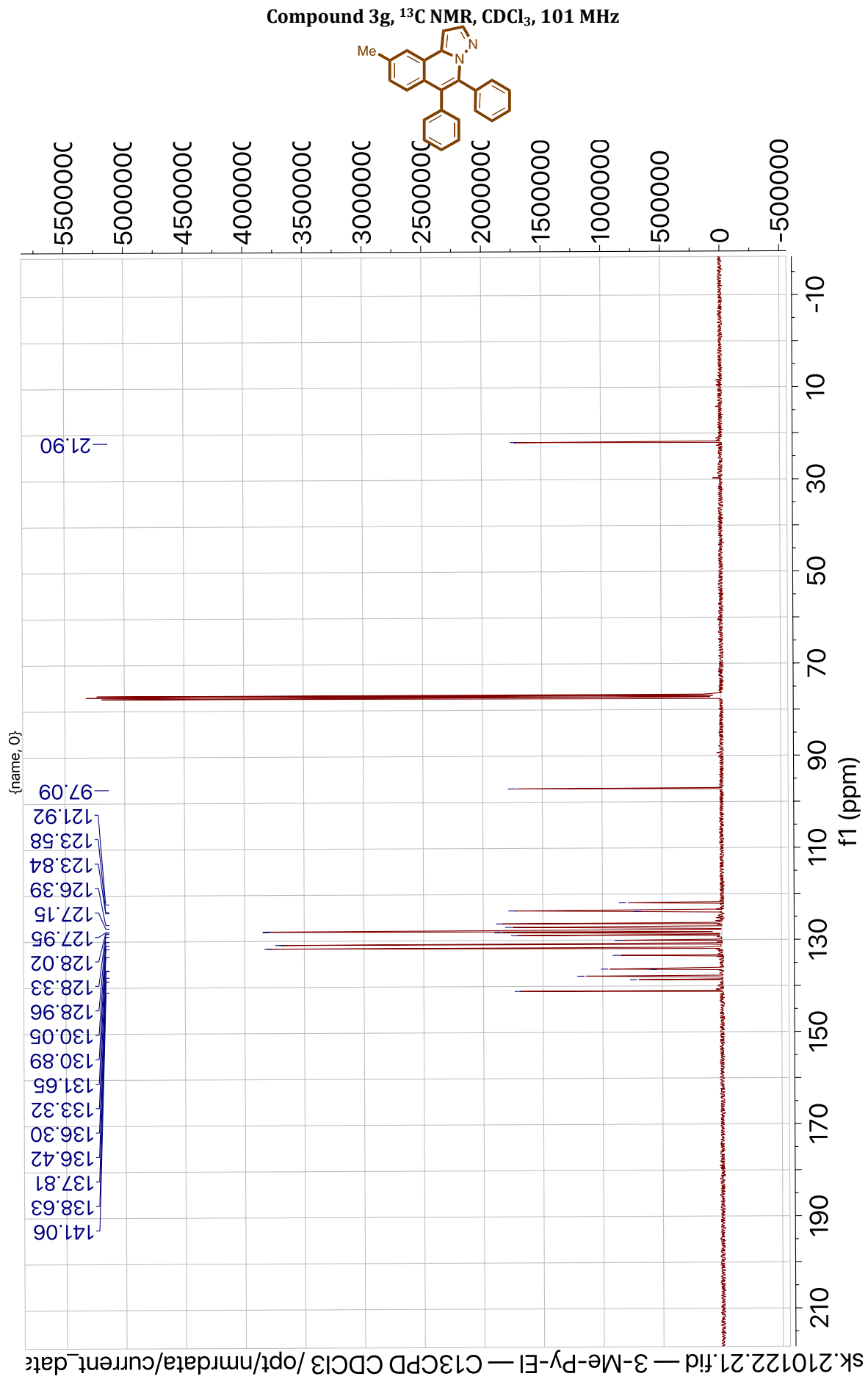
Mass	Calc. Mass	mDa	PPM	DBE	Formula	Fit Conf %	C	H	N	O	F	Na
389.1264	389.1266	-0.2	-0.5	16.5	C ₂₄ H ₁₆ N ₂ F ₃	41.1/a	24	16	2		3	



HRMS spectra of **3f**

Compound 3g, ¹H NMR, CDCl₃, 400 MHz

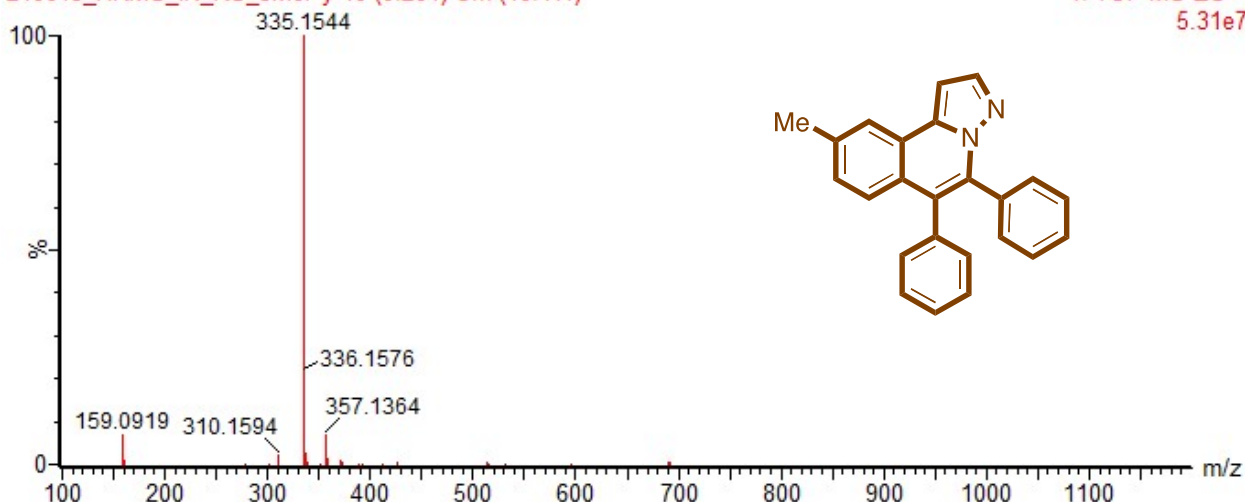




DCM-> MeOH (2% H2O + 0.1% FA)

210615_HRMS_IN_KS_3MePy 10 (0.201) Cm (10:111)

1: TOF MS ES+
5.31e7



Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

208 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-50

H: 0-100

N: 0-5

O: 0-3

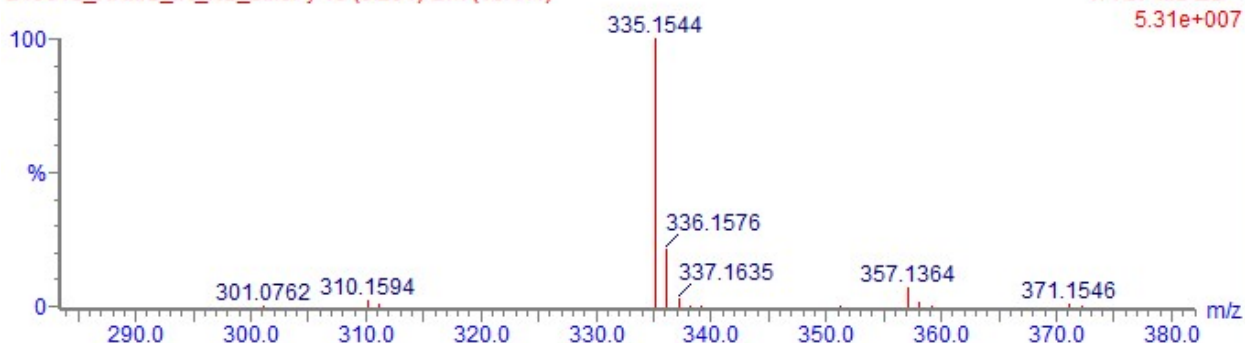
Na: 0-1

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i...	Fit Conf %	C	H	N	O	Na
335.1544	335.1548	-0.4	-1.2	16.5	C24 H19 N2	4...	17.55	24	19	2		
	335.1524	2.0	6.0	13.5	C22 H20 N2 Na	4...	82.45	22	20	2		1

DCM-> MeOH (2% H2O + 0.1% FA)

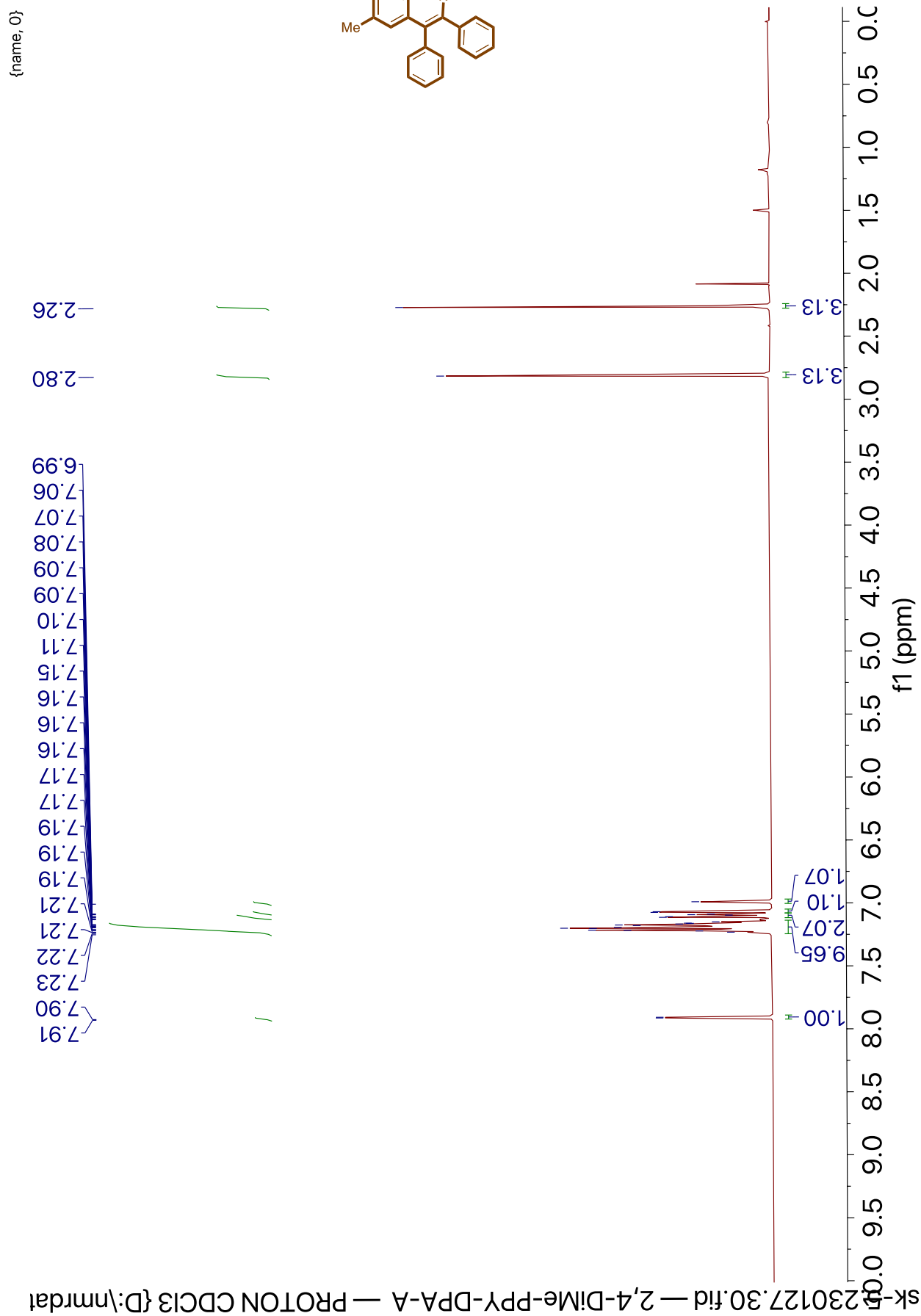
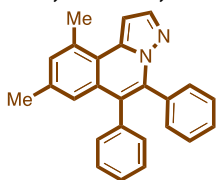
210615_HRMS_IN_KS_3MePy 10 (0.201) Cm (10:111)

1: TOF MS ES+
5.31e+007



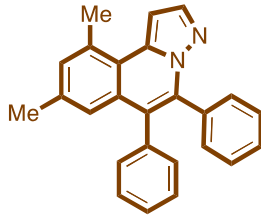
HRMS spectra of **3g**

Compound 3h, ¹H NMR, CDCl₃, 400 MHz



sk-1230127.30.ftd - 2,4-DiMe-PY-DPA-A - PROTON CDCl3 {D:\nmrdat

Compound 3h, ¹³C NMR, CDCl₃, 101 MHz



{name, 0}

~23.84
~21.59

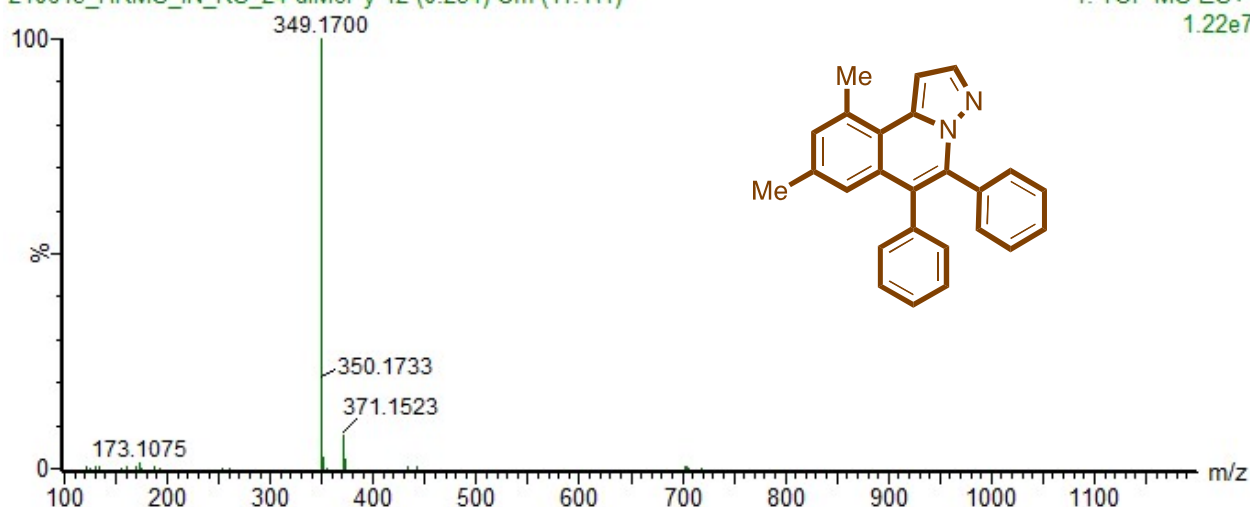
140.71
137.76
136.97
136.89
136.41
134.42
133.75
131.66
131.36
130.80
128.21
128.00
127.95
127.07
124.67
124.28
121.65
101.70

sk-6.230127.31.fid - 2,4-DiMe-PY-DPA-A - C13CPD CDCl3 {D:\nmrdate

DCM-> MeOH (2% H2O + 0.1% FA)

210615_HRMS_IN_KS_24-diMePy 12 (0.234) Cm (11:111)

1: TOF MS ES+
1.22e7



Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

216 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

Elements Used:

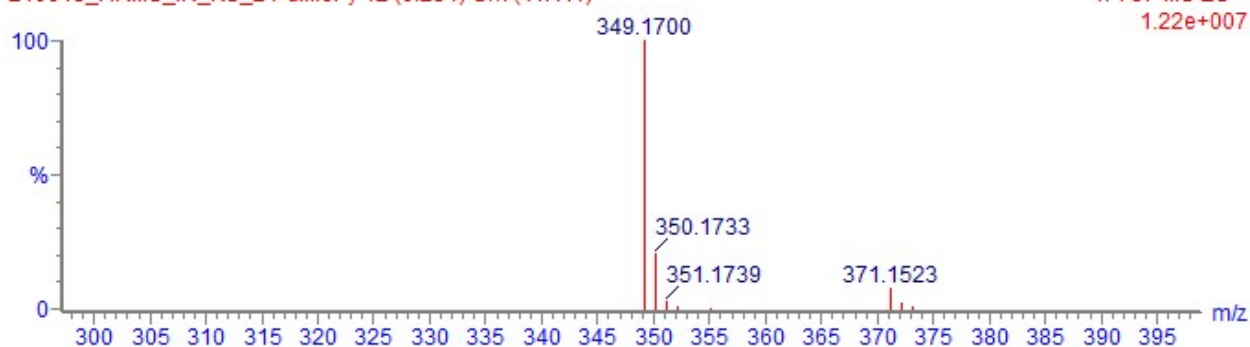
C: 0-50 H: 0-100 N: 0-5 O: 0-3 Na: 0-1

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-Fl...	Fit Conf %	C	H	N	O	Na
349.1700	349.1705	-0.5	-1.4	16.5	C ₂₅ H ₂₁ N ₂	6.683	18.58	25	21	2		
	349.1681	1.9	5.4	13.5	C ₂₃ H ₂₂ N ₂ Na	5.206	81.42	23	22	2		1

DCM-> MeOH (2% H2O + 0.1% FA)

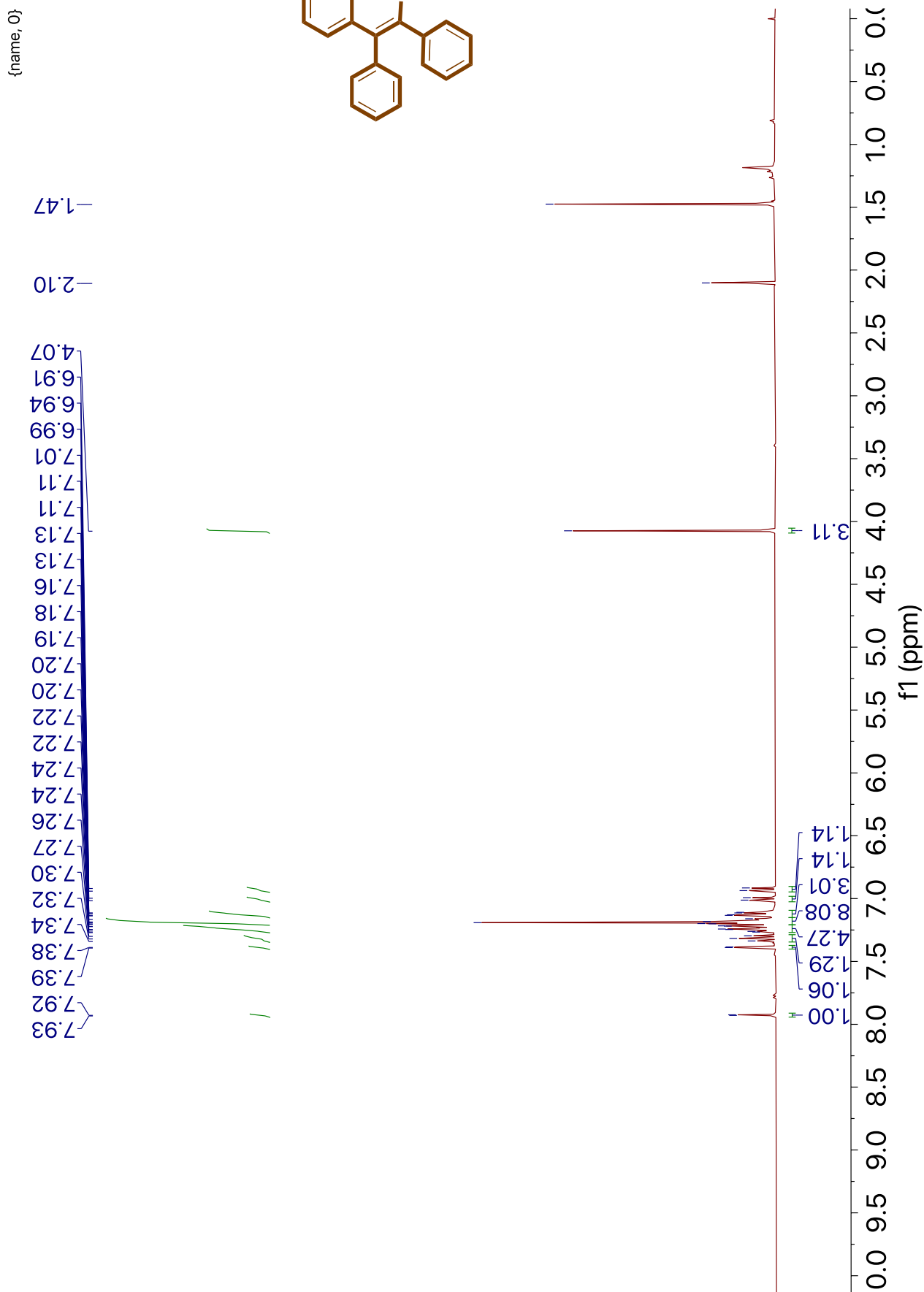
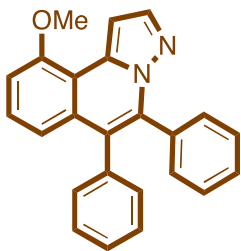
210615_HRMS_IN_KS_24-diMePy 12 (0.234) Cm (11:111)

1: TOF MS ES+
1.22e+007



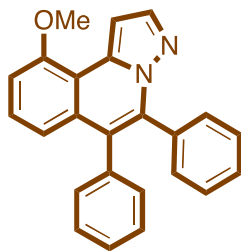
HRMS spectra of **3h**

Compound 3i, ¹H NMR, CDCl₃, 400 MHz

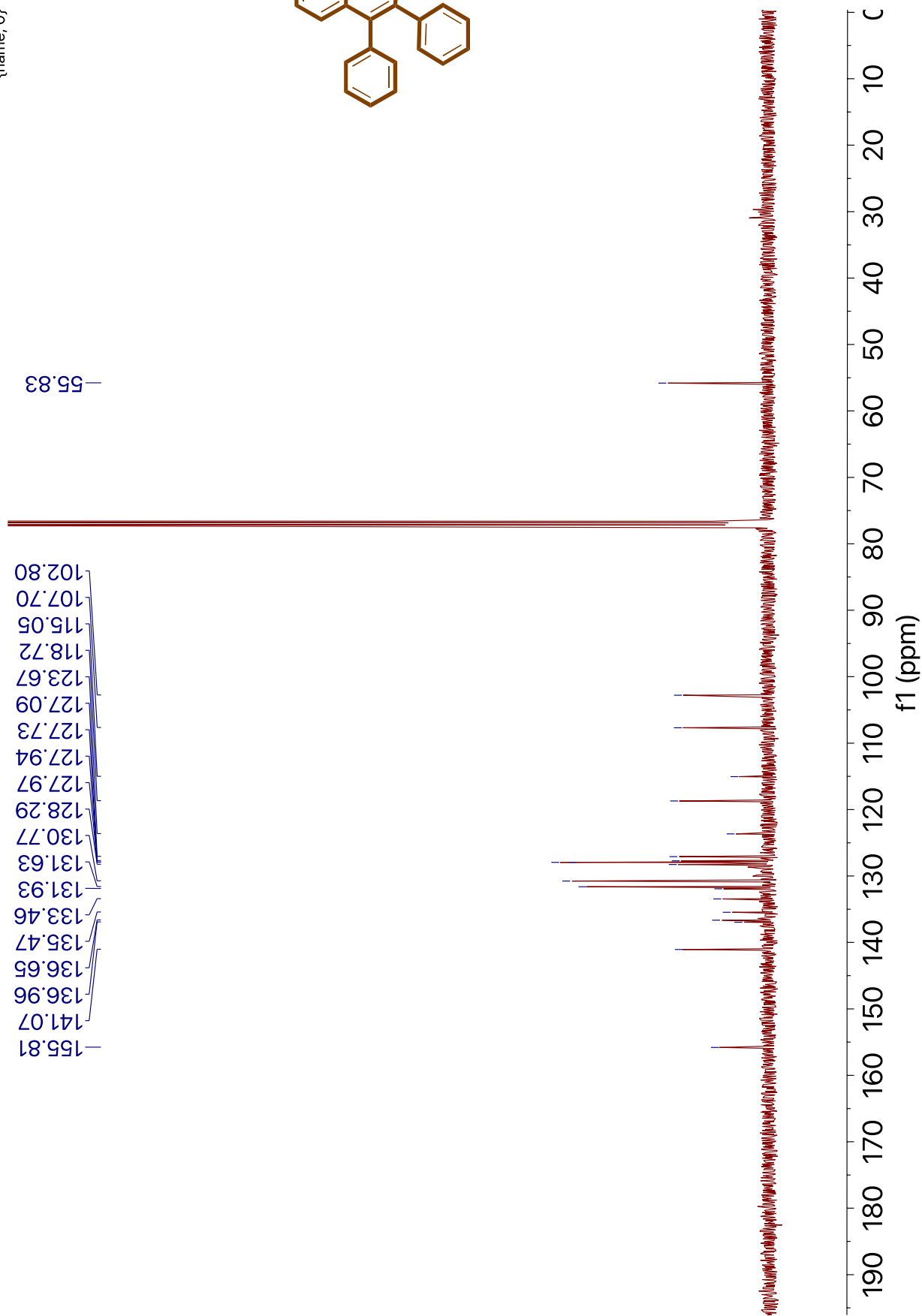


sk-3 230126.40.fid - 2-OMe-PY-DPA-A - PROTON CDCl3 {D:\nmrdata\

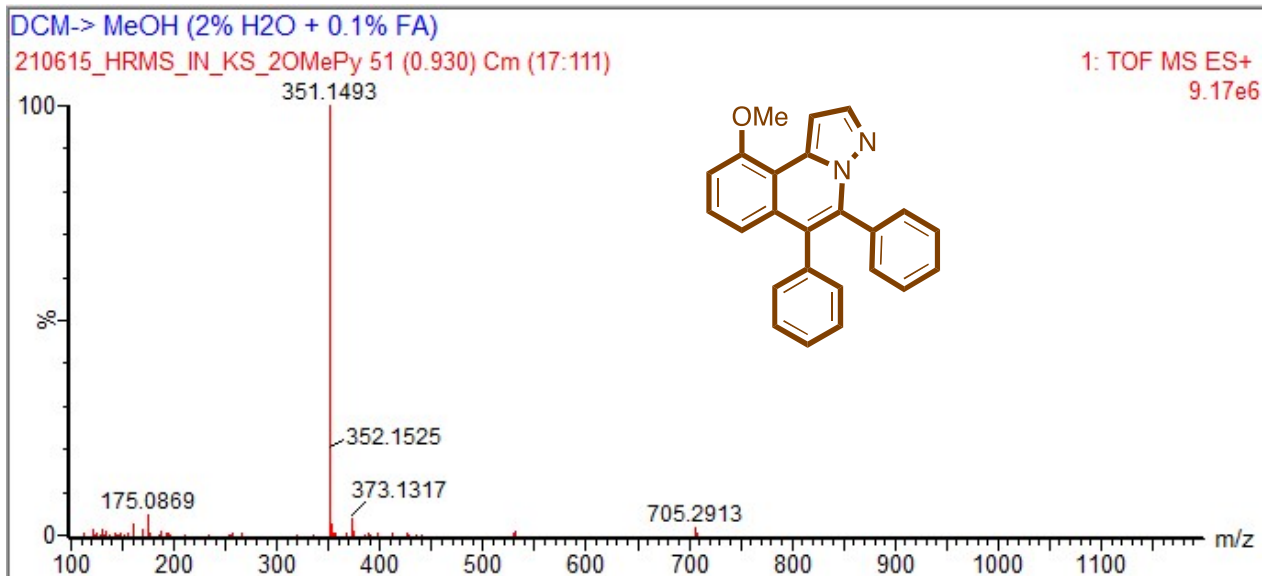
Compound 3i, ¹³C NMR, CDCl₃, 101 MHz



{name, 0}



sk-9.230126.41.fid - 2-OMe-PY-DPA-A - C13CPD CDCl3 {D:\nmrdata\c



Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

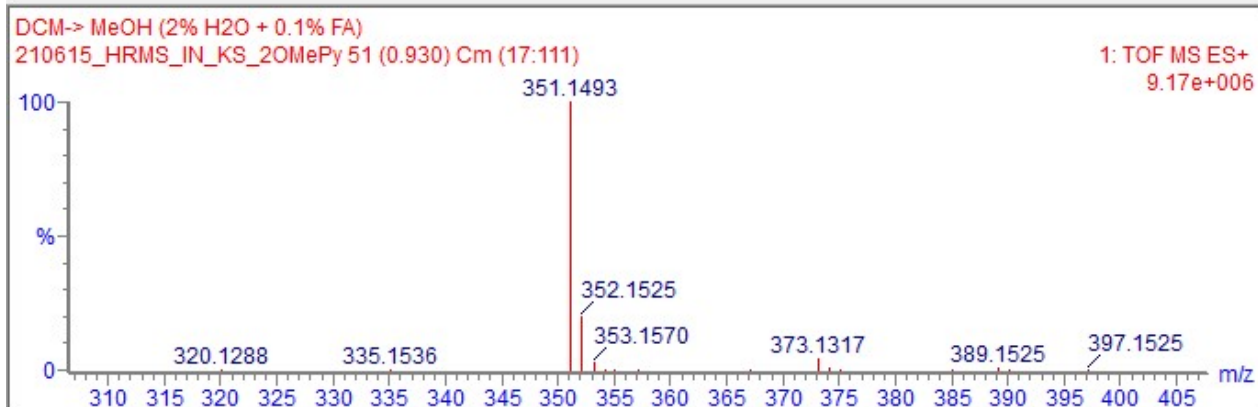
Monoisotopic Mass, Even Electron Ions

216 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

Elements Used:

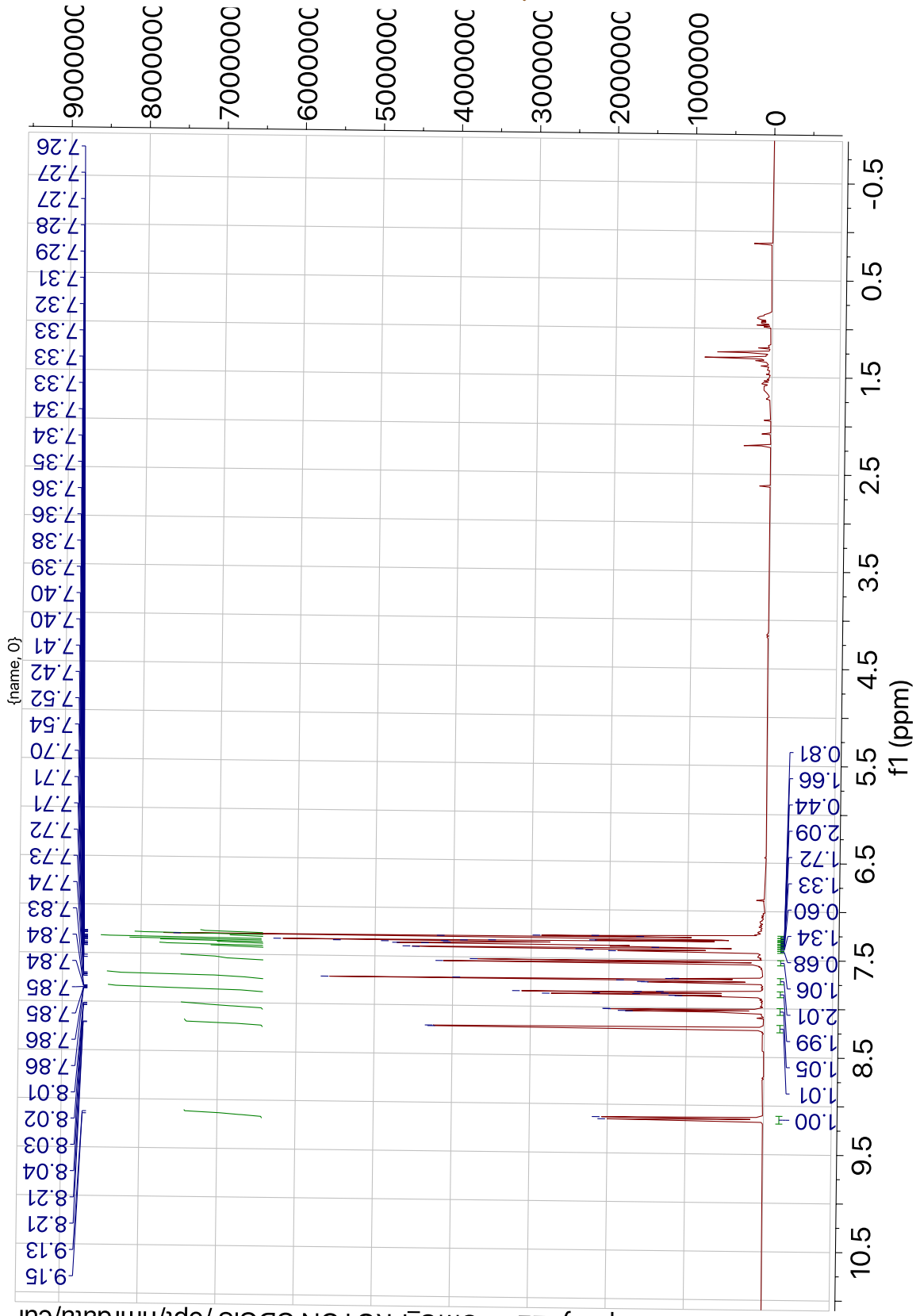
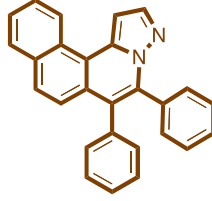
C: 0-50 H: 0-100 N: 0-5 O: 0-3 Na: 0-1

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i...	Fit Conf %	C	H	N	O	Na
351.1493	351.1497	-0.4	-1.1	16.5	C ₂₄ H ₁₉ N ₂ O	30...	45.56	24	19	2	1	
	351.1473	2.0	5.7	13.5	C ₂₂ H ₂₀ N ₂ O Na	30...	54.44	22	20	2	1	1



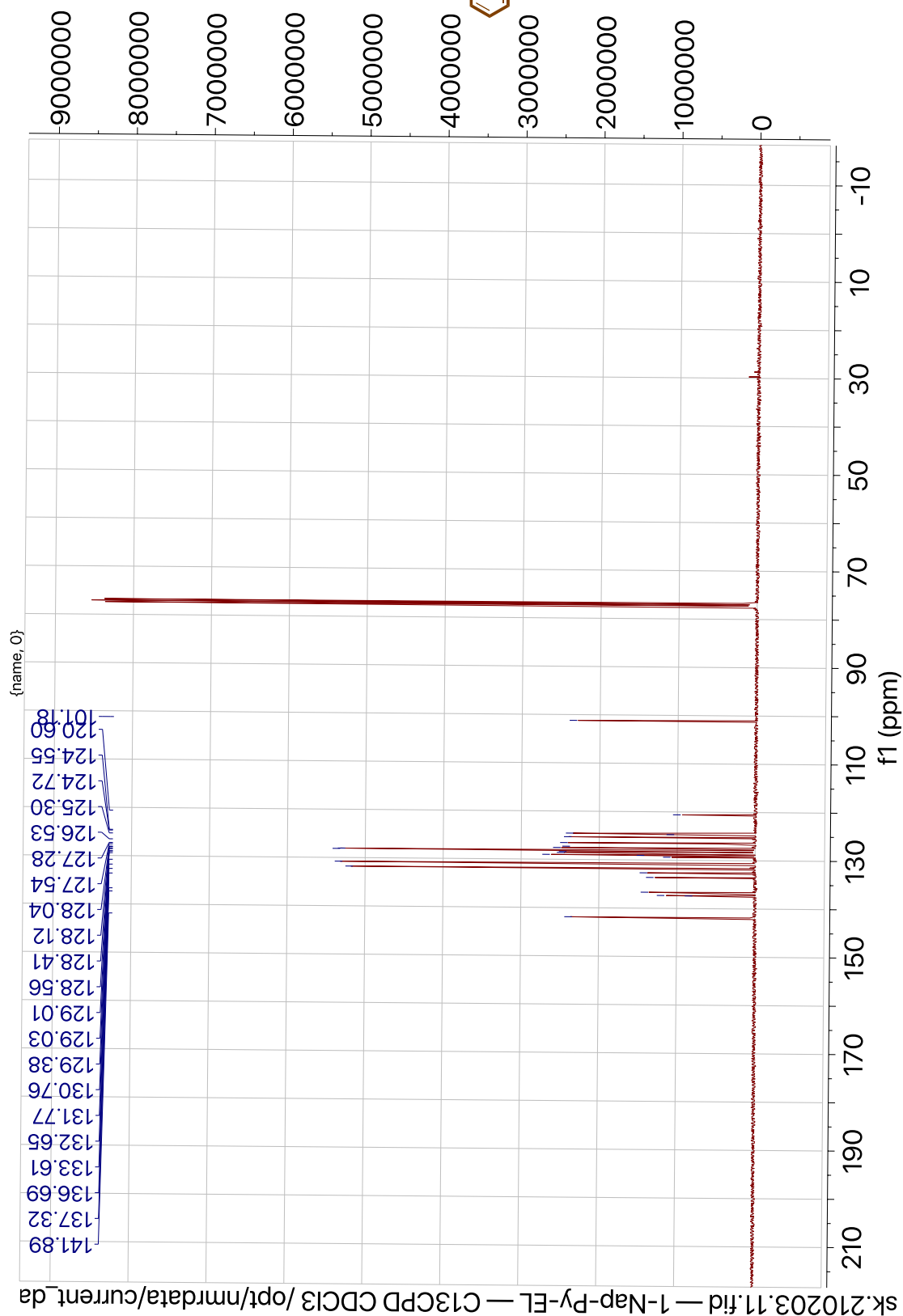
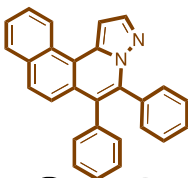
HRMS spectra of **3i**

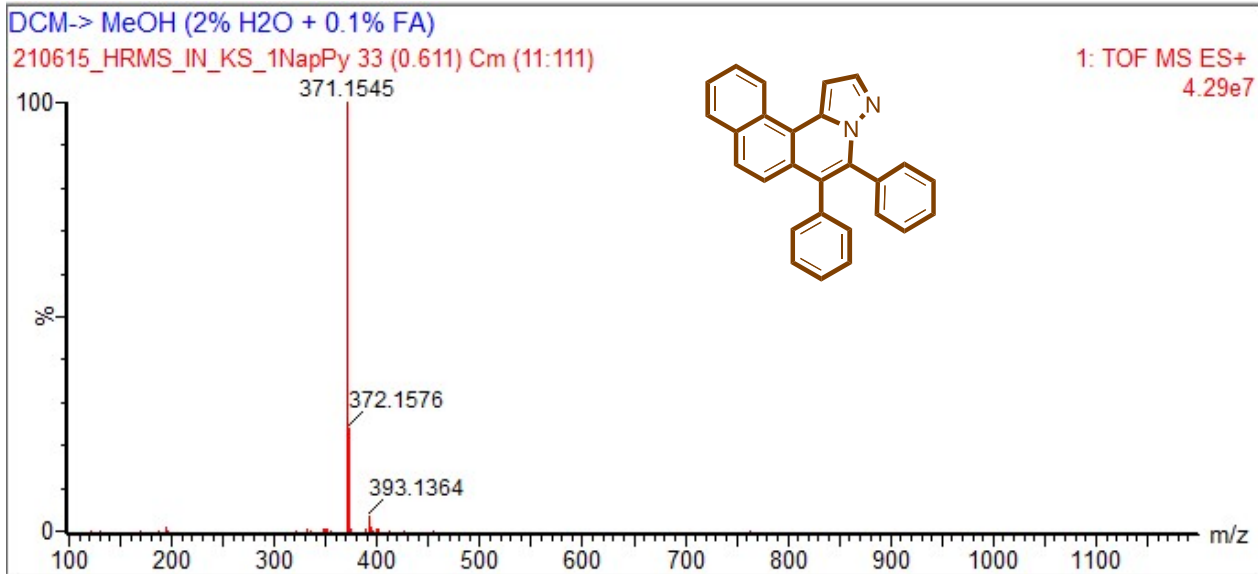
Compound 3J, ¹H NMR, CDCl₃, 400 MHz



sk.210203.10.fid - 1-Nap-Py-EL - CMC_PROTON CDCl3 /opt/nmrdata/cur

Compound 3J, ¹³C NMR, CDCl₃, 101 MHz





Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

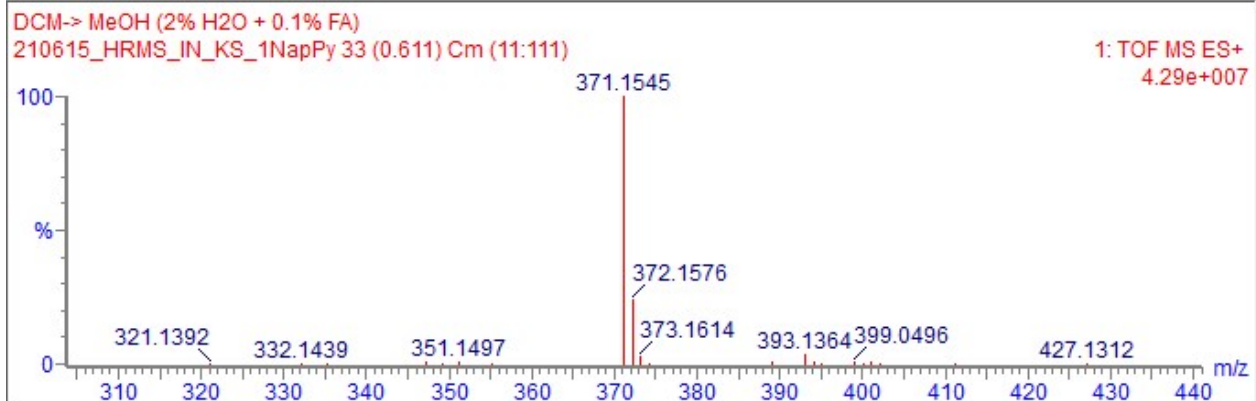
Monoisotopic Mass, Even Electron Ions

227 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

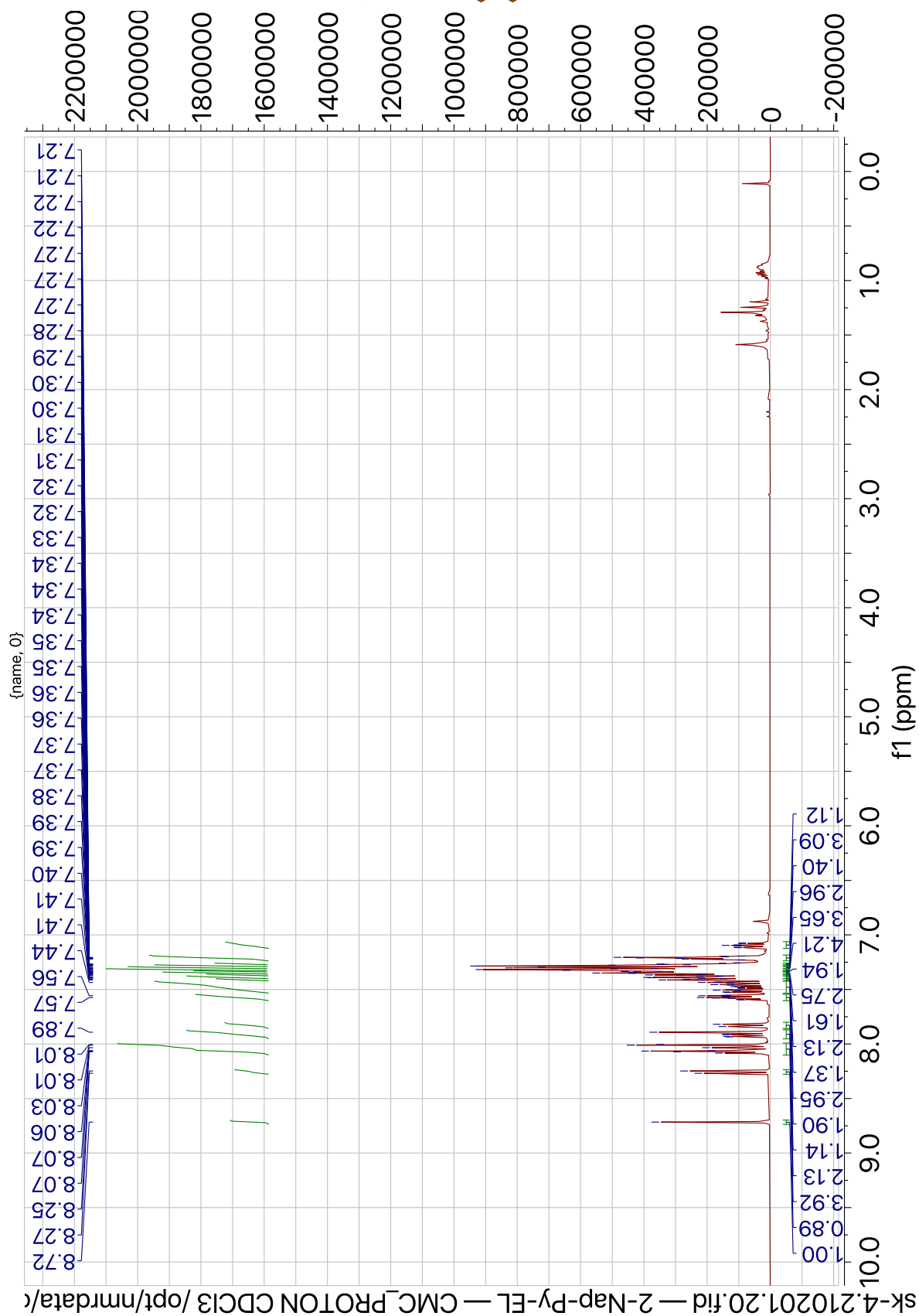
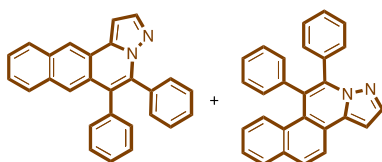
C: 0-50 H: 0-100 N: 0-5 O: 0-3 Na: 0-1

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT Norm	Fit Conf %	C	H	N	O	Na
371.1545	371.1548	-0.3	-0.8	19.5	C ₂₇ H ₁₉ N ₂	n/a	n/a	27	19	2		

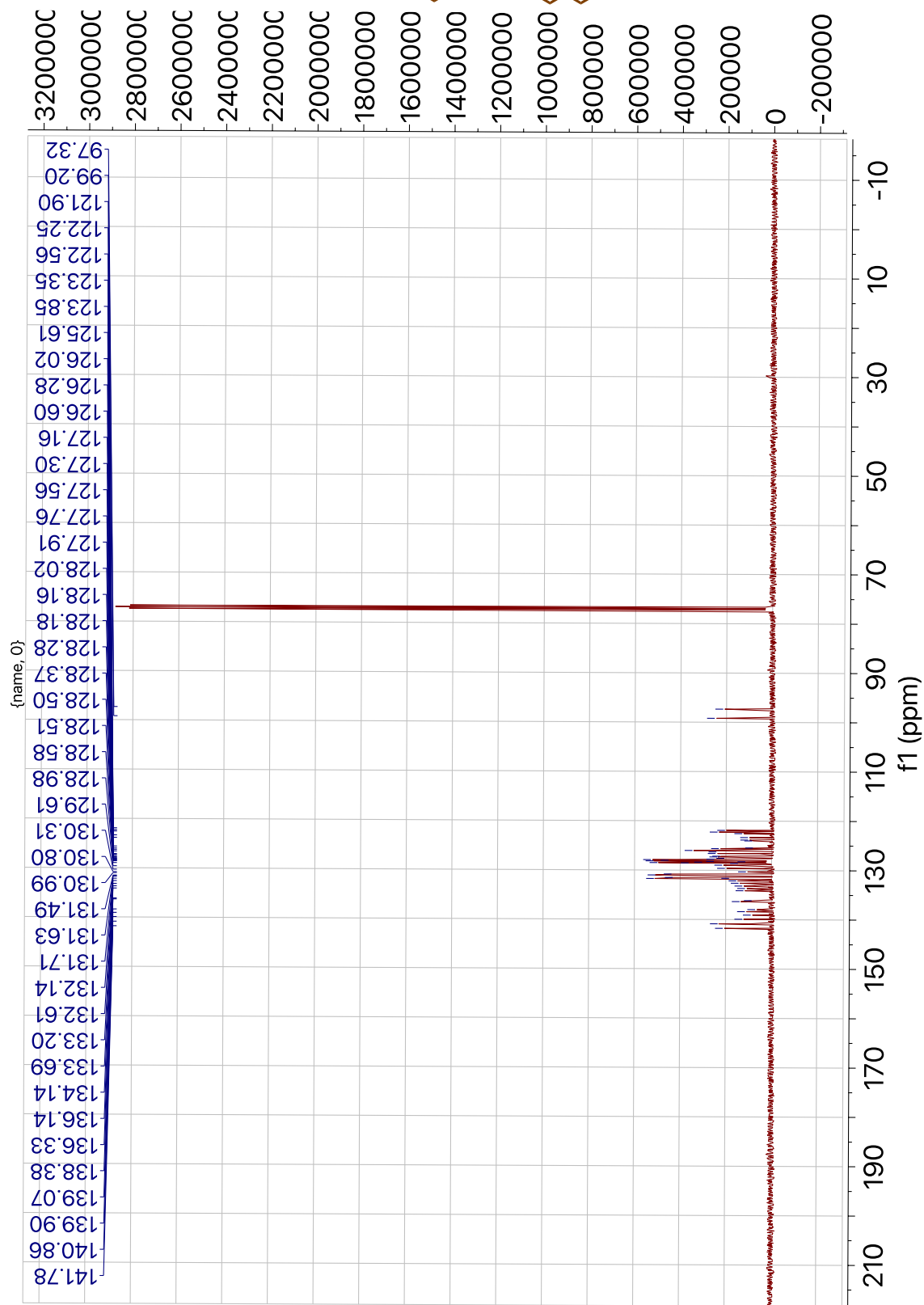
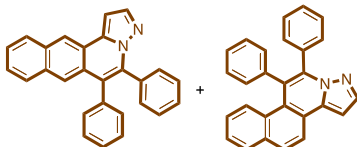


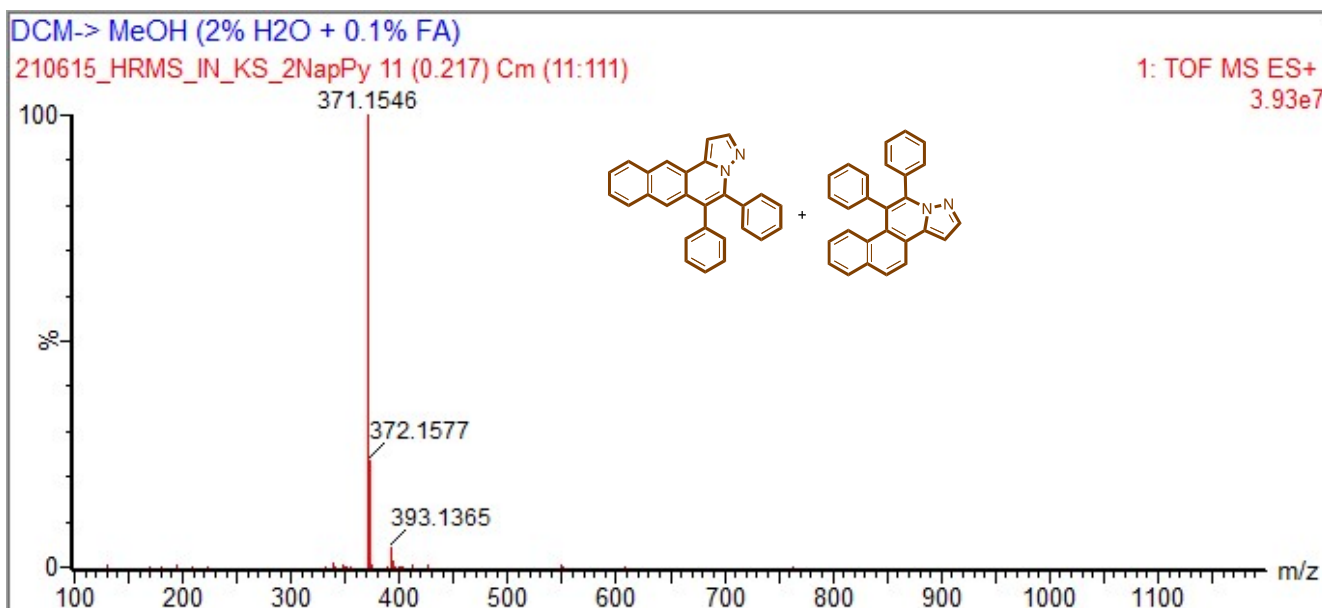
HRMS spectra of **3j**

Compound 3K+3K', ¹H NMR, CDCl₃, 400 MHz



Compound 3K+3K', ¹³C NMR, CDCl₃, 101 MHZ





Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

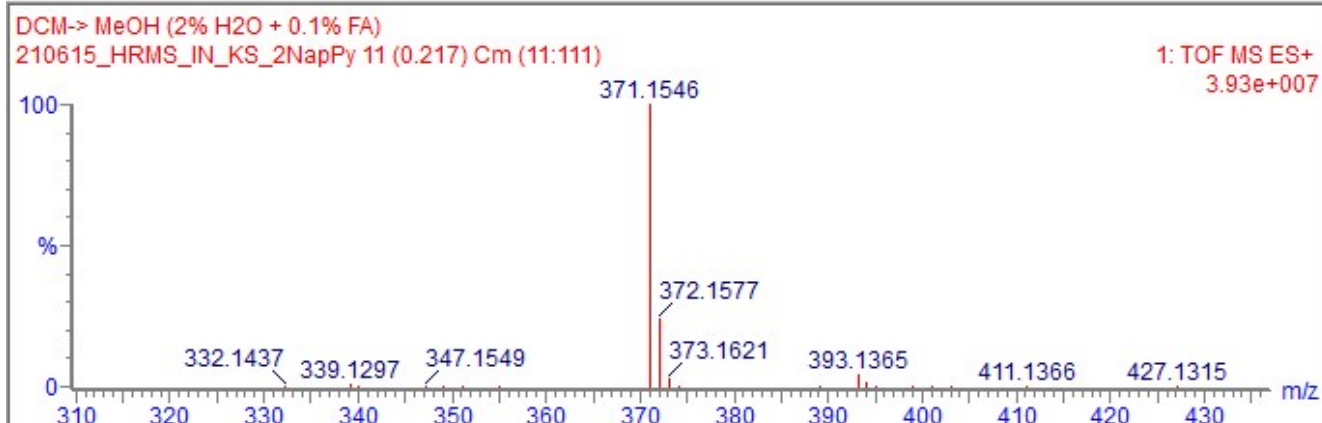
Monoisotopic Mass, Even Electron Ions

227 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

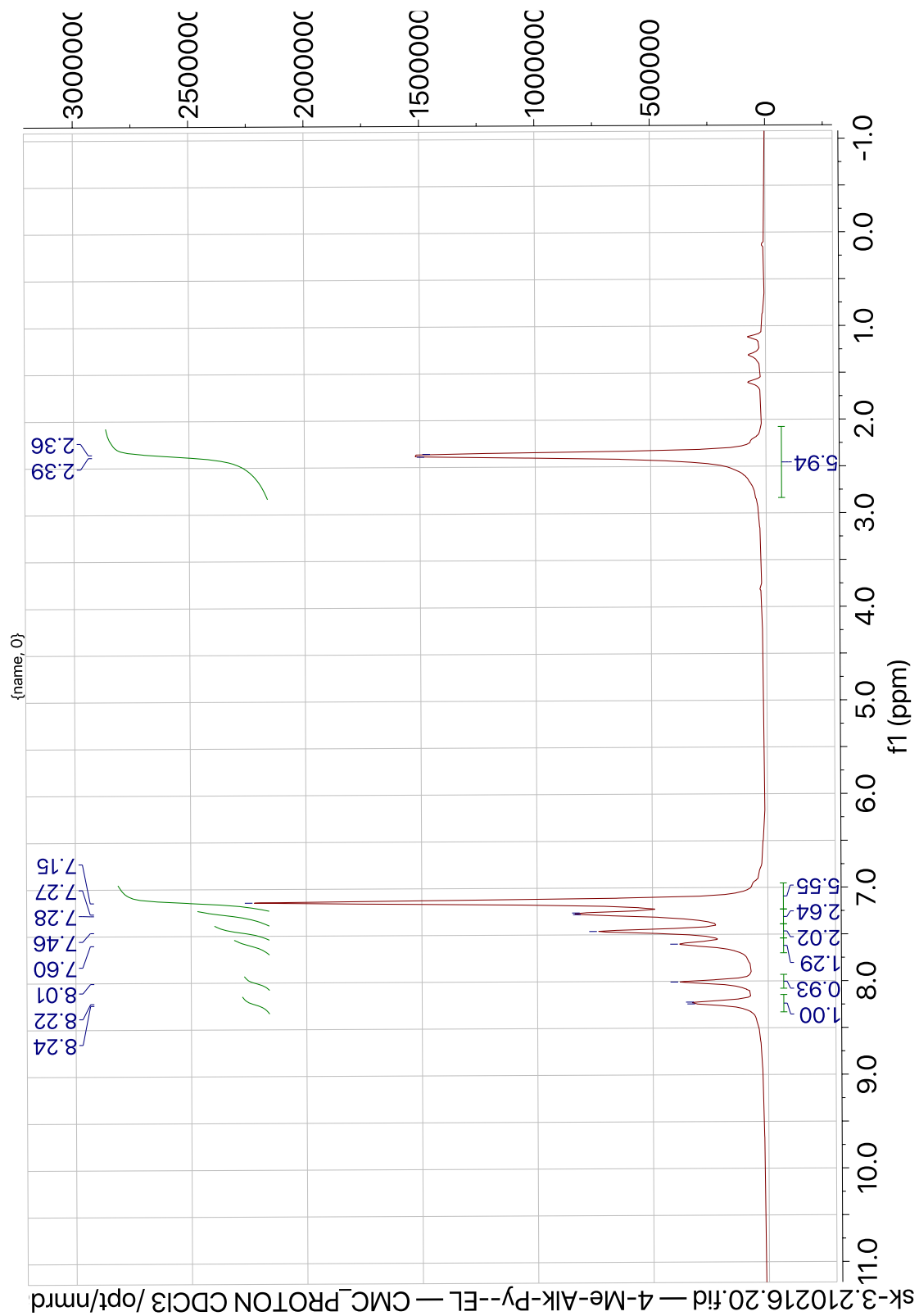
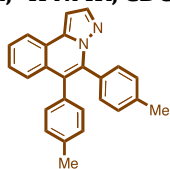
C: 0-50 H: 0-100 N: 0-5 O: 0-3 Na: 0-1

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	N	O	Na
371.1546	371.1548	-0.2	-0.5	19.5	C27 H19 N2	40.1	n/a	n/a	27	19	2		

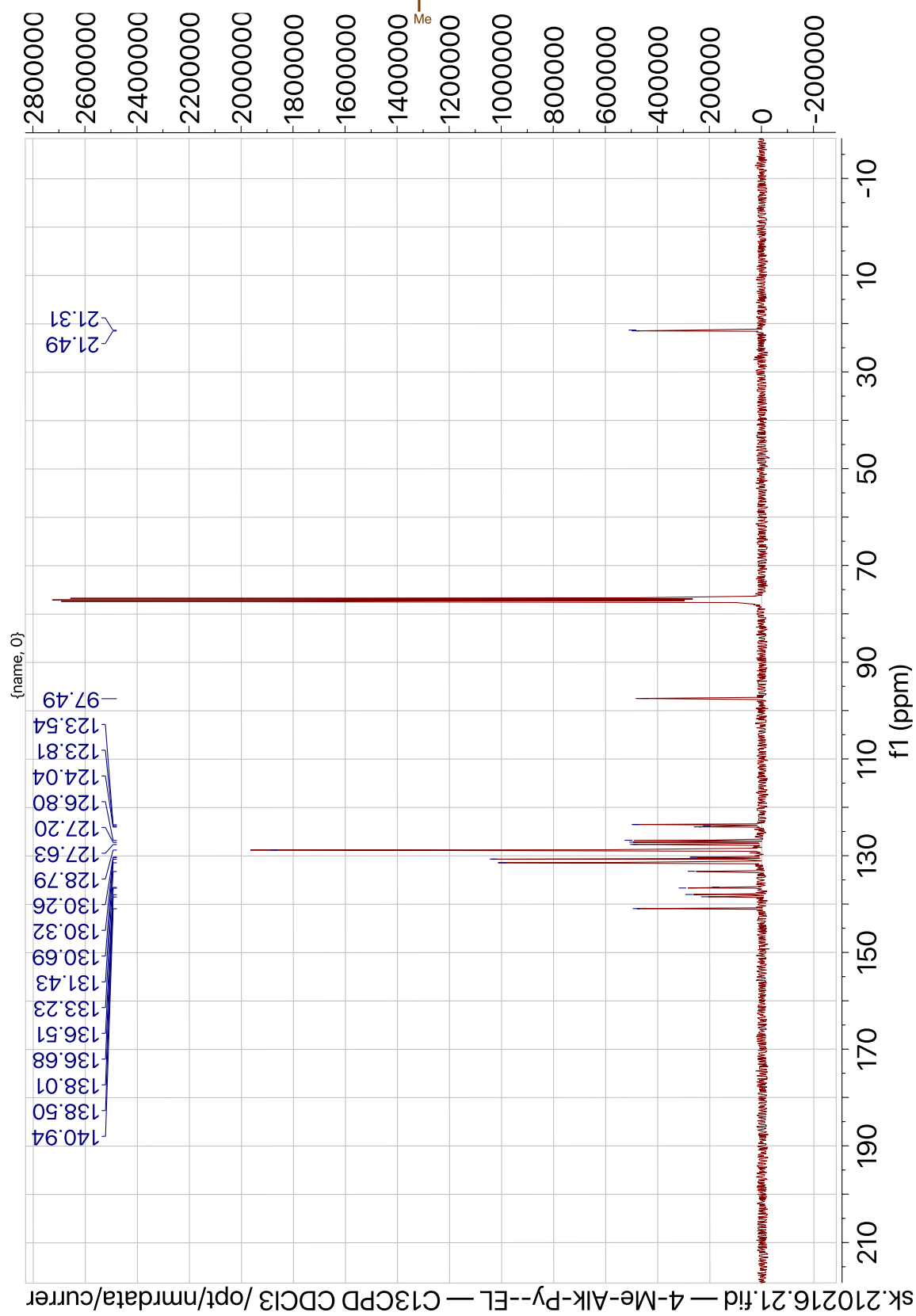
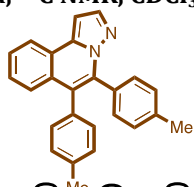


HRMS spectra of 3K + 3K'

Compound 5a, ¹H NMR, CDCl₃, 400 MHz



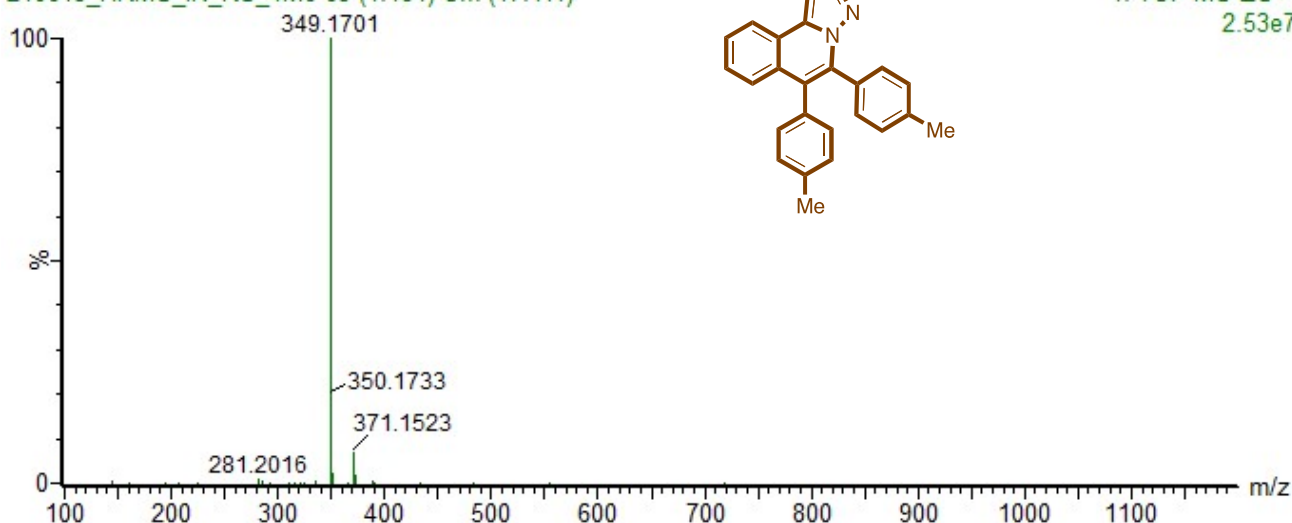
Compound 5a, ¹³C NMR, CDCl₃, 101 MHz



DCM-> MeOH (2% H2O + 0.1% FA)

210615_HRMS_IN_KS_4Me 65 (1.181) Cm (17:111)

1: TOF MS ES+
2.53e7



Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

216 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-50

H: 0-100

N: 0-5

O: 0-3

Na: 0-1

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i...	Fit Conf %	C	H	N	O	Na
349.1701	349.1705	-0.4	-1.1	16.5	C25 H21 N2	41.5	n...	n/a	25	21	2		

DCM-> MeOH (2% H2O + 0.1% FA)

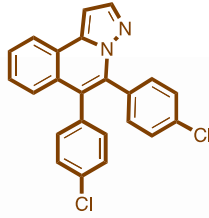
210615_HRMS_IN_KS_4Me 65 (1.181) Cm (17:111)

1: TOF MS ES+
2.53e+007



HRMS spectra of 5a

Compound 5b, ¹³C NMR, CDCl₃, 101 MHz

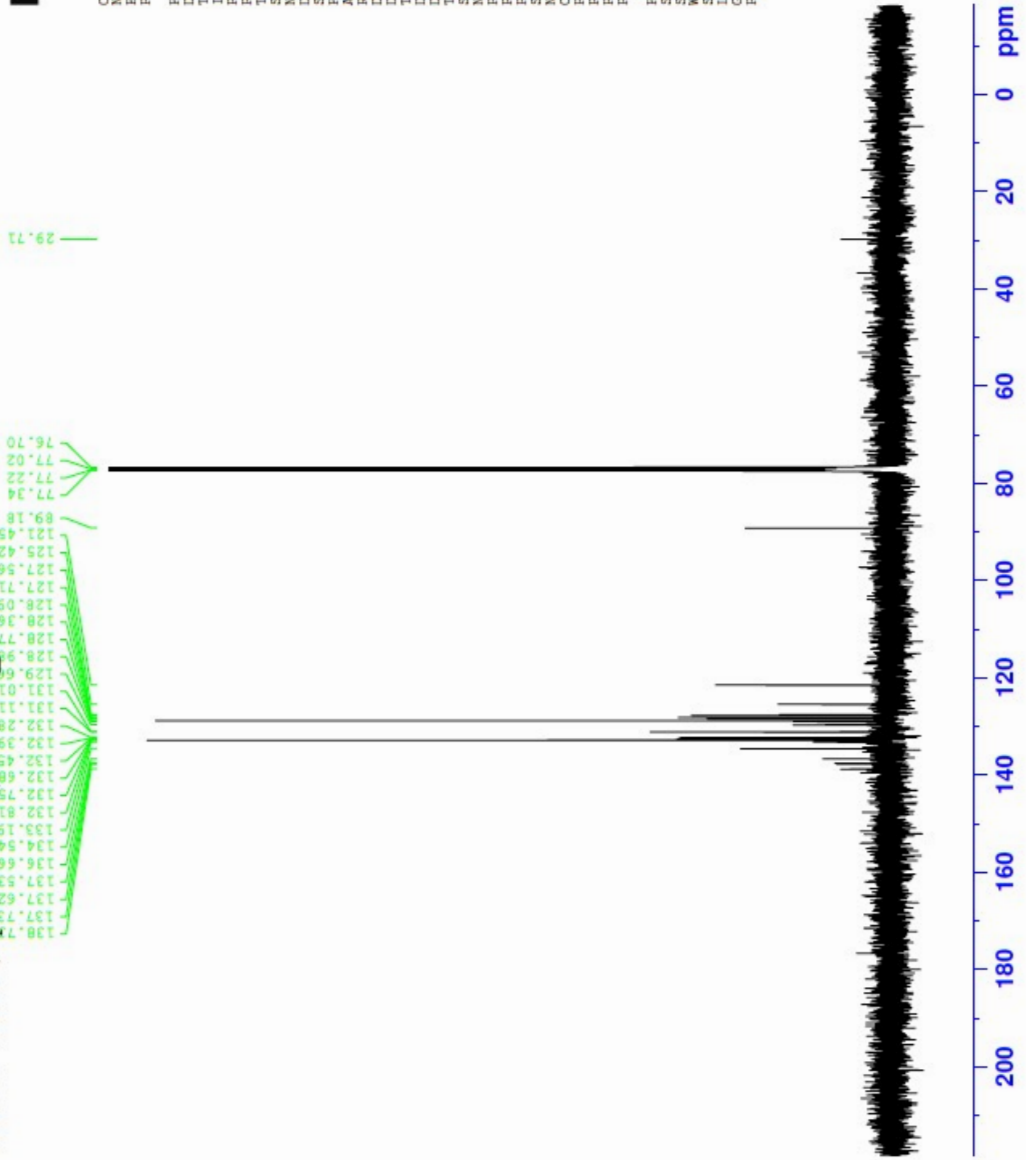


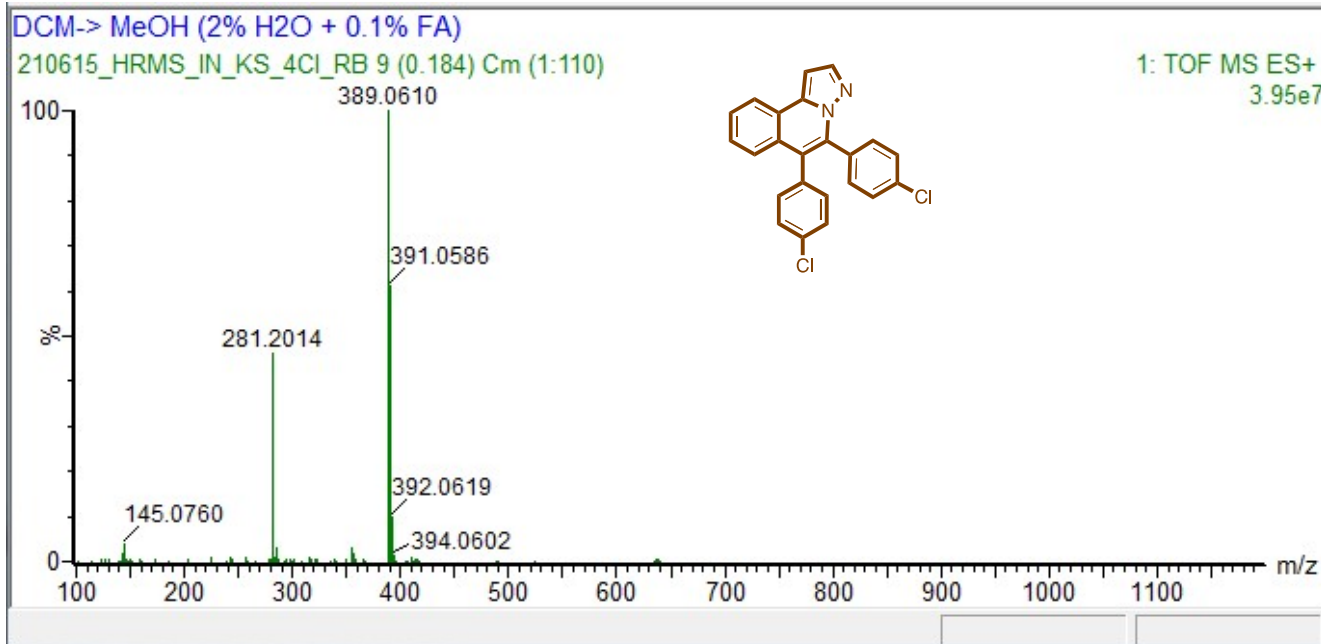
Current Data Parameters
 NAME sk.210219
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20210219
 Time 15.43 h
 INSTRUM Avance Neo Nanobay 400 MHz
 PROBHD Z163739_0067
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 23809.573 Hz
 FIDRES 0.16666 Hz
 AQ 1.3762560 sec
 RG 101
 DW 21.000 usec
 DE 6.50 usec
 TE 298.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1
 SF01 100.6228298 MHz
 NUC1 13C
 P1 12.00 usec
 PL1 0.00 usec
 P1M1 61.27700043 W
 SF02 400.1316005 MHz
 NUC2 1H
 CPOPRG12 maltz65
 PCPD2 90.00 usec
 P1M2 14.63000011 W
 P1M12 0.16232000 W
 P1M13 0.08164700 W

F2 - Processing parameters
 SI 32768
 SF 100.6127685 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

PY-4-ClRA-Alk-EL
 C13CPD CDCl3 /opt/nmrdata/current_data_sk_6





Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

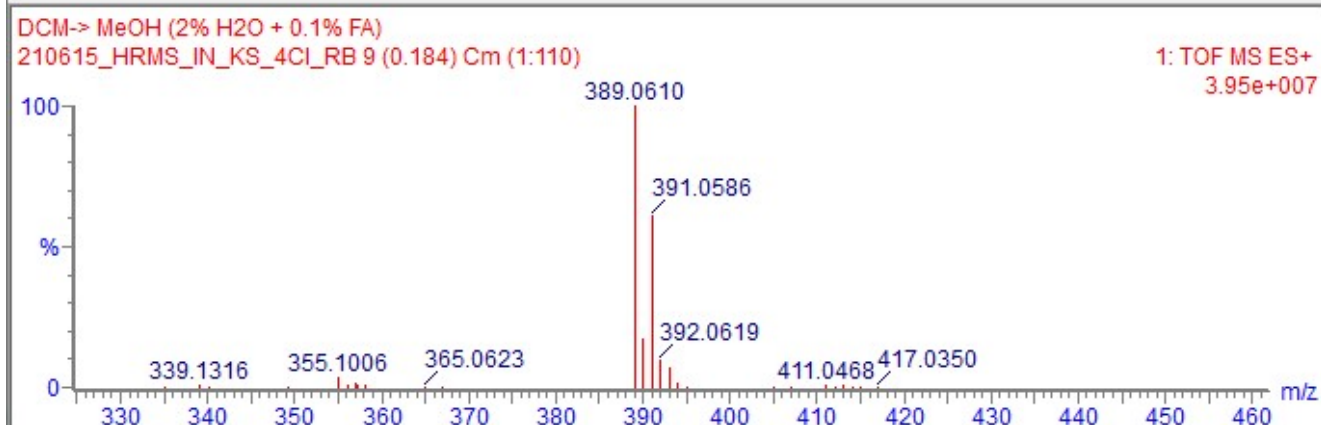
Monoisotopic Mass, Even Electron Ions

644 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)

Elements Used:

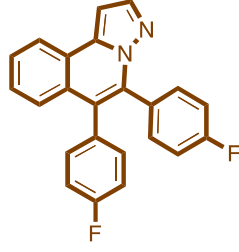
C: 0-50 H: 0-100 N: 0-5 O: 0-3 Na: 0-1 Cl: 0-2

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-...	Fit Conf %	C	H	N	O	Na	Cl
389.0610	389.0612	-0.2	-0.5	16.5	C23 H15 N2 Cl2	0...	99.81	23	15	2			2
	389.0603	0.7	1.8	25.5	C29 H9 O2	1...	0.00	29	9		2		
	389.0594	1.6	4.1	21.5	C24 H10 N4 Cl	6...	0.19	24	10	4			1

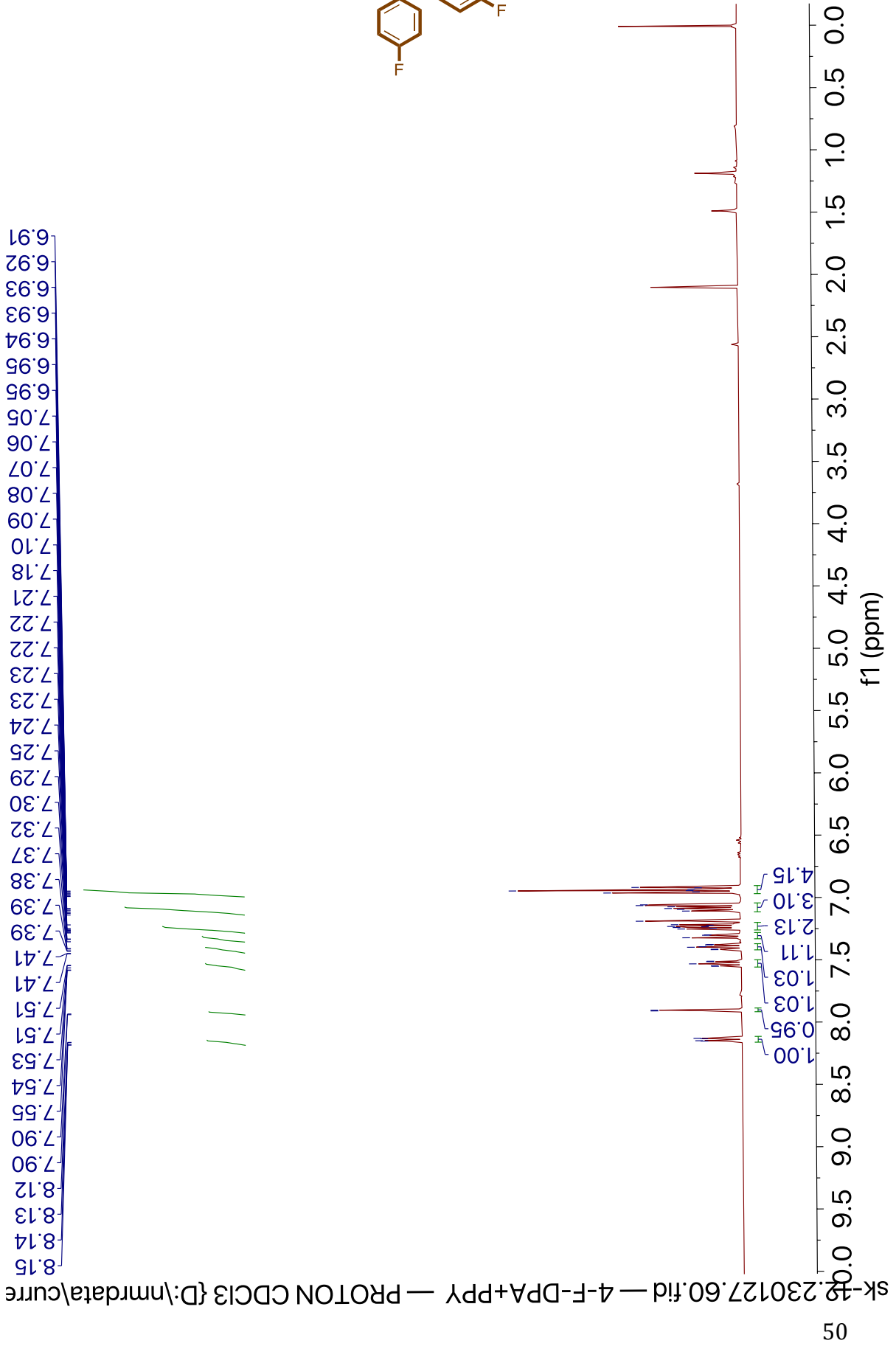


HRMS spectra of **5b**

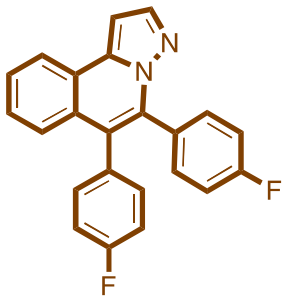
Compound 5c, ¹H NMR, CDCl₃, 400 MHz



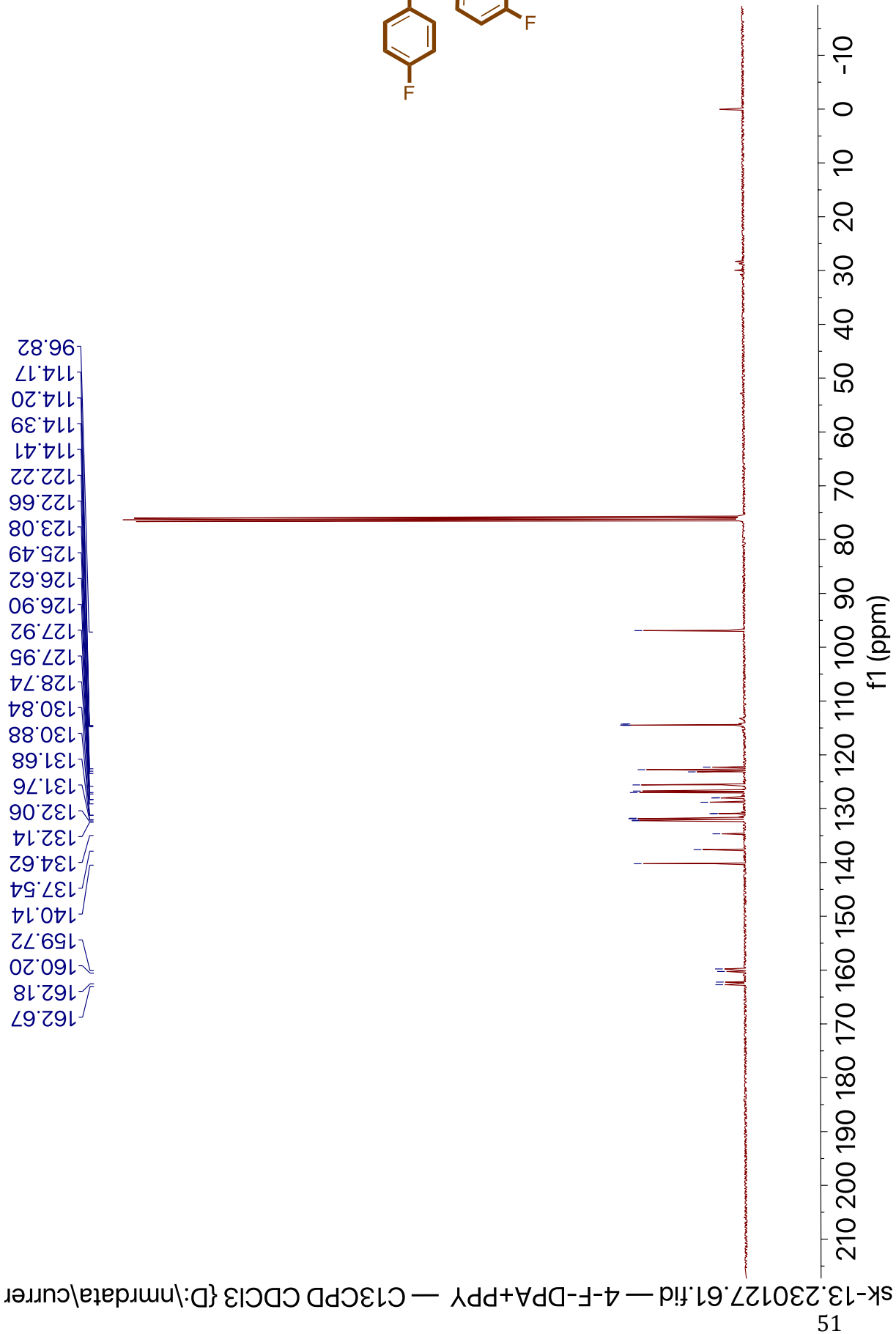
{name, 0}



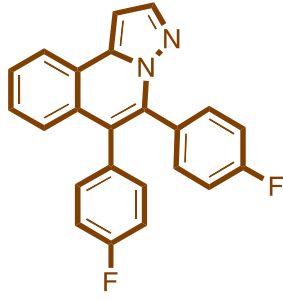
Compound 5c, ¹³C NMR, CDCl₃, 101 MHz



{name, 0}

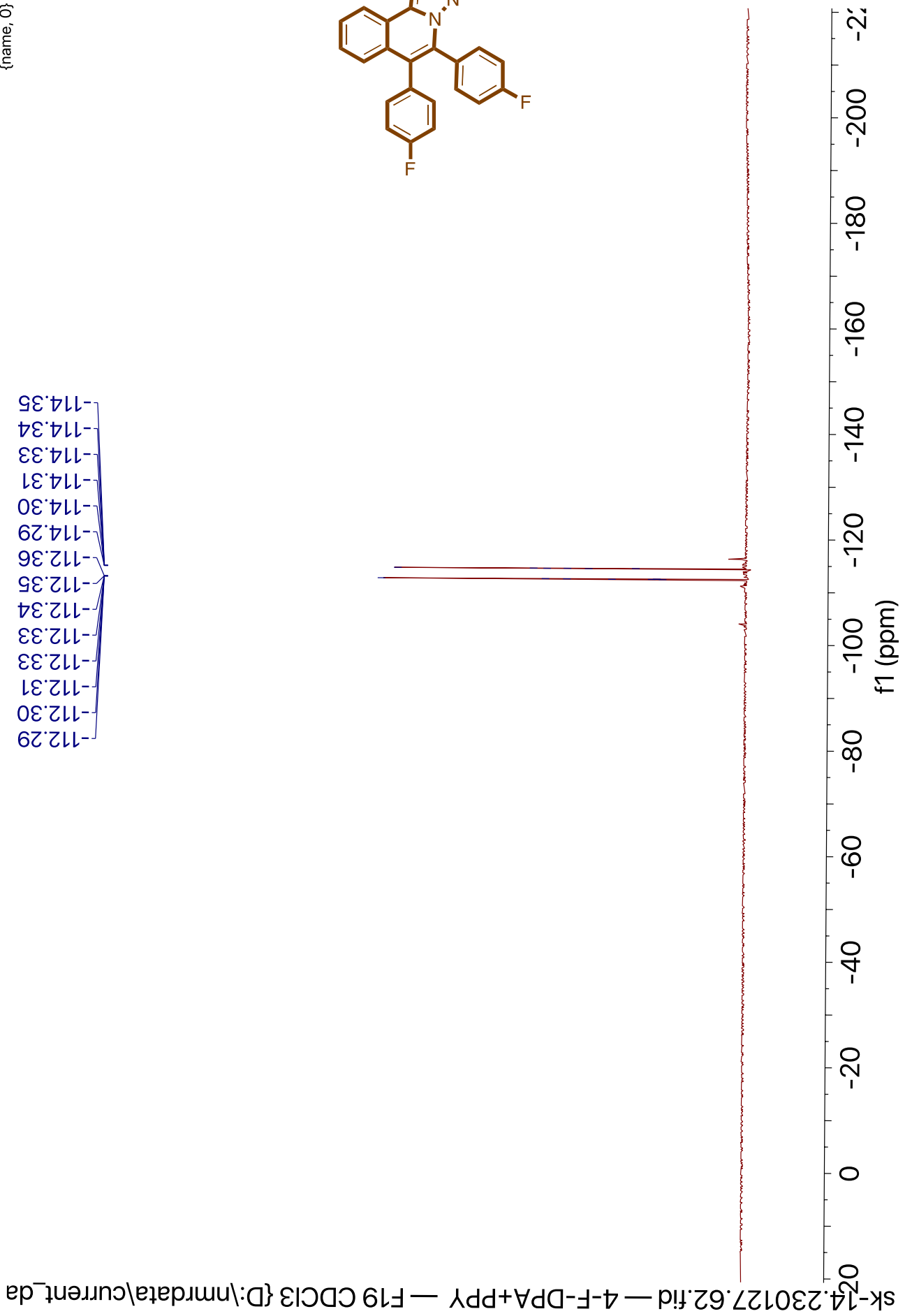


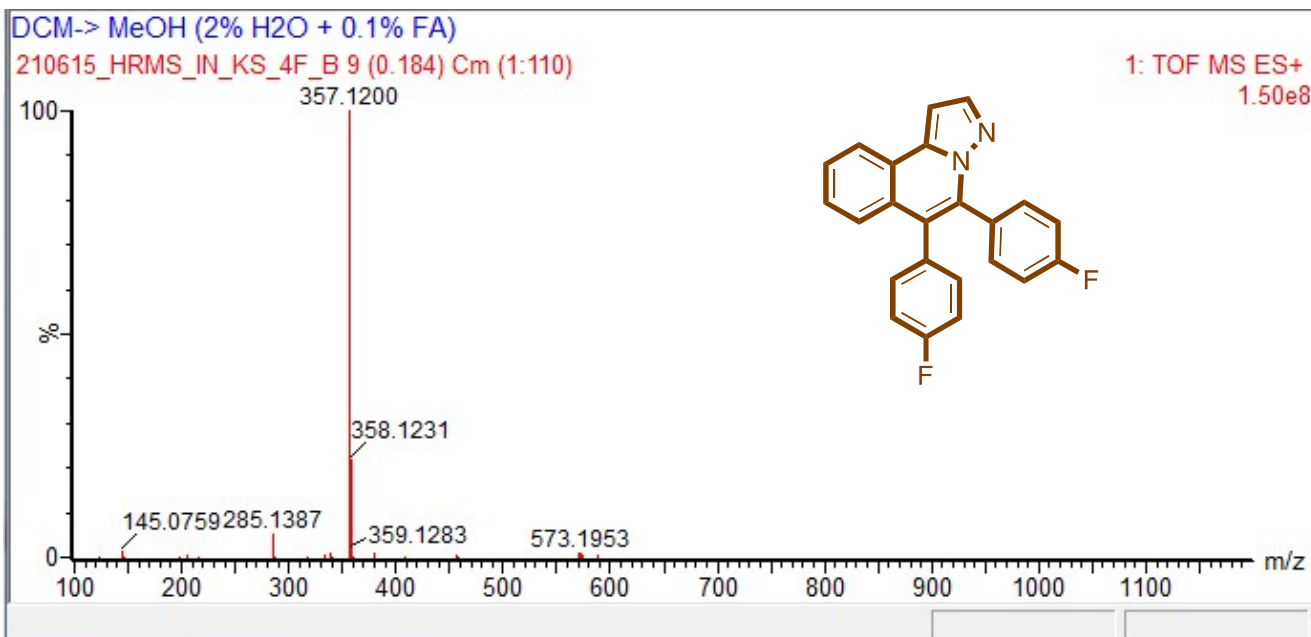
Compound 5c, ¹³C NMR, CDCl₃, 101 MHz



{name, 0}

114.35
114.34
114.33
114.31
114.30
114.29
112.36
112.35
112.34
112.33
112.33
112.31
112.30
112.29





Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

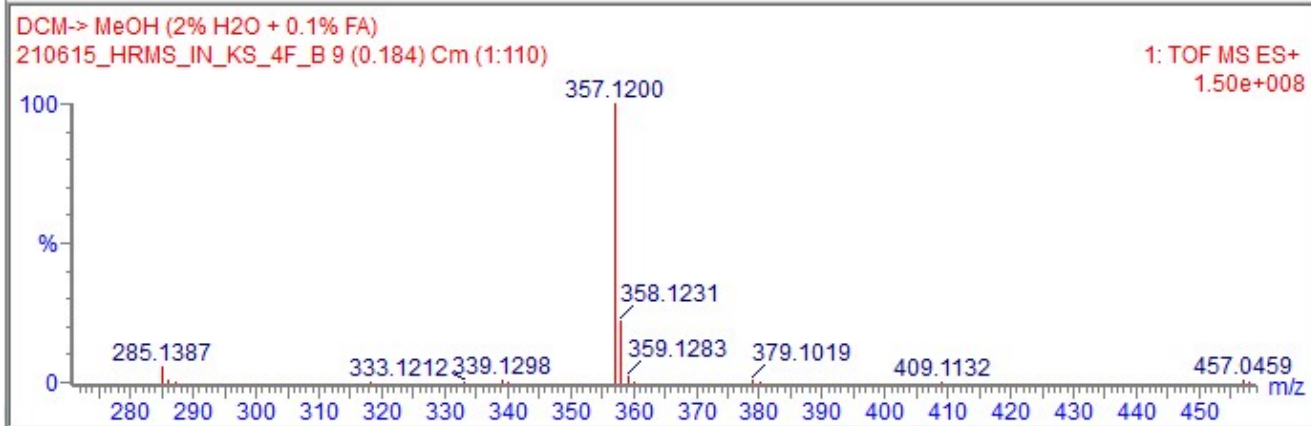
Monoisotopic Mass, Even Electron Ions

615 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

Elements Used:

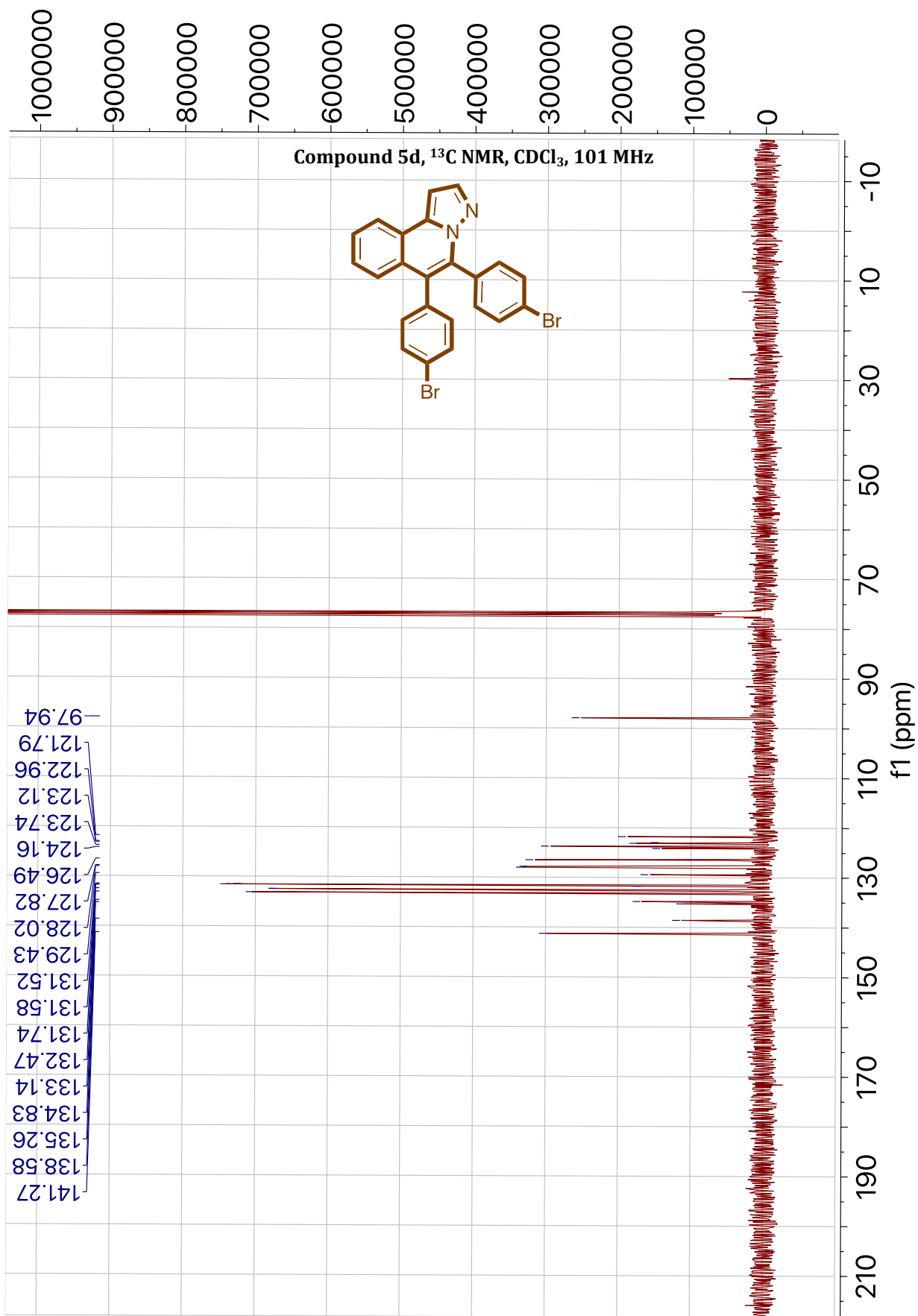
C: 0-50 H: 0-100 N: 0-5 O: 0-3 F: 0-2 Na: 0-1

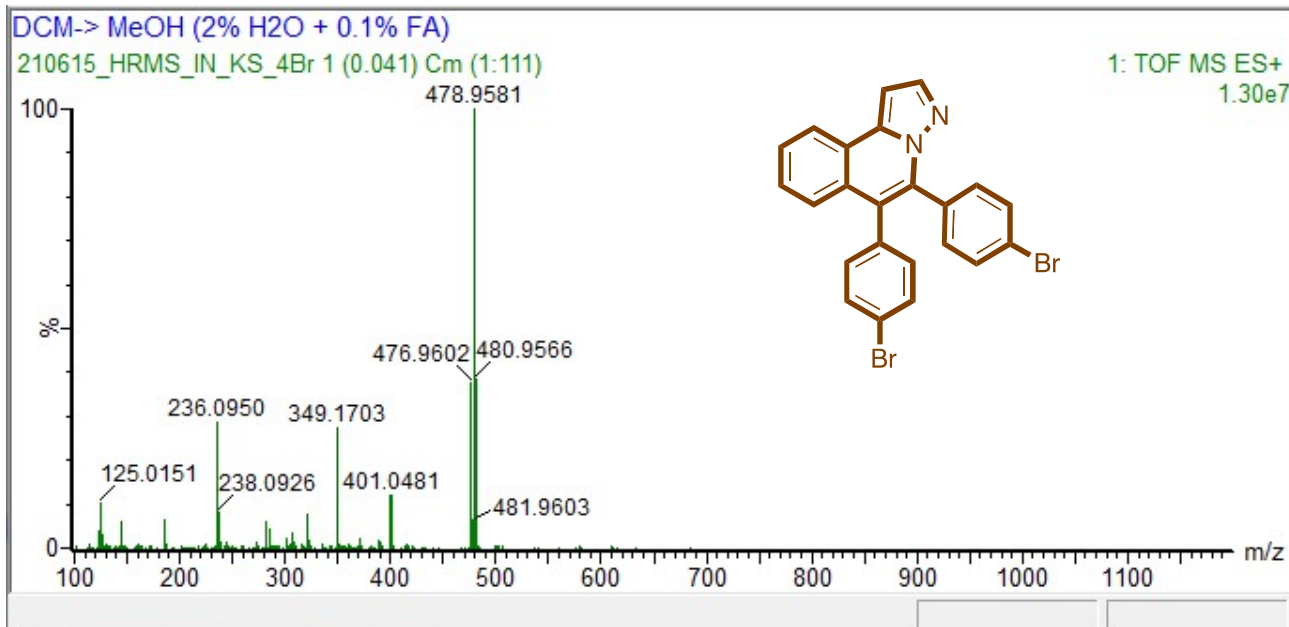
Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-Fl...	Fit Conf %	C	H	N	O	F	Na
357.1200	357.1203	-0.3	-0.8	16.5	C23 H15 N2 F2	40....	43.00	23	15	2		2	
	357.1215	-1.5	-4.2	12.5	C20 H18 N2 O3 Na	40....	57.00	20	18	2	3		1



HRMS spectra of 5c

sk.210216.11.fid — 4-Br-AIK-Py-EL — C13CPD CDCl3 /opt/nmrdata/current/





Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

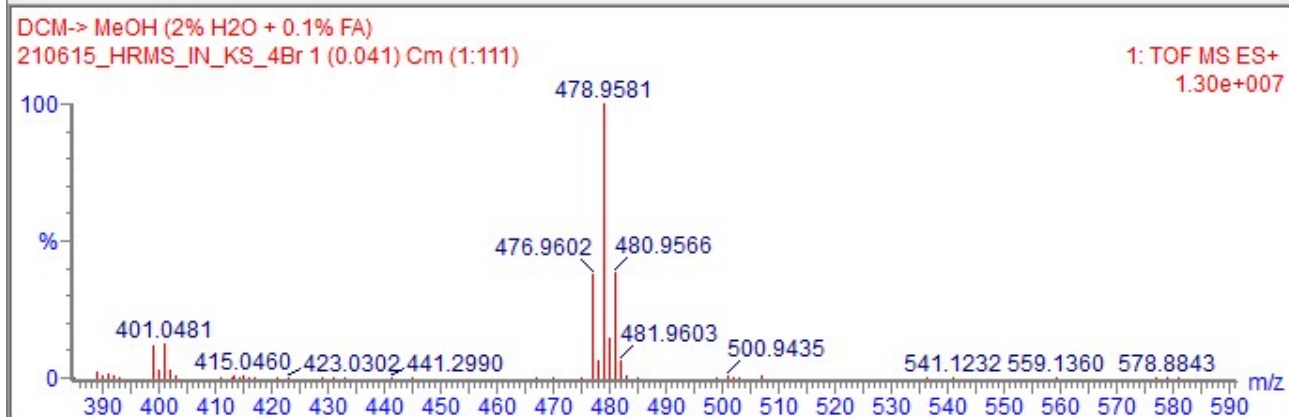
Monoisotopic Mass, Even Electron Ions

719 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

Elements Used:

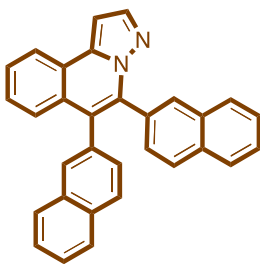
C: 0-50 H: 0-100 N: 0-5 O: 0-3 Na: 0-1 Br: 0-2

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i...	Fit Conf %	C	H	N	O	Na	Br
476.9602	476.9602	0.0	0.0	16.5	C23 H15 N2 Br2	50...	98.95	23	15	2			2
	476.9599	0.3	0.6	20.5	C22 H7 N4 O3 Na Br	64...	1.05	22	7	4	3	1	1

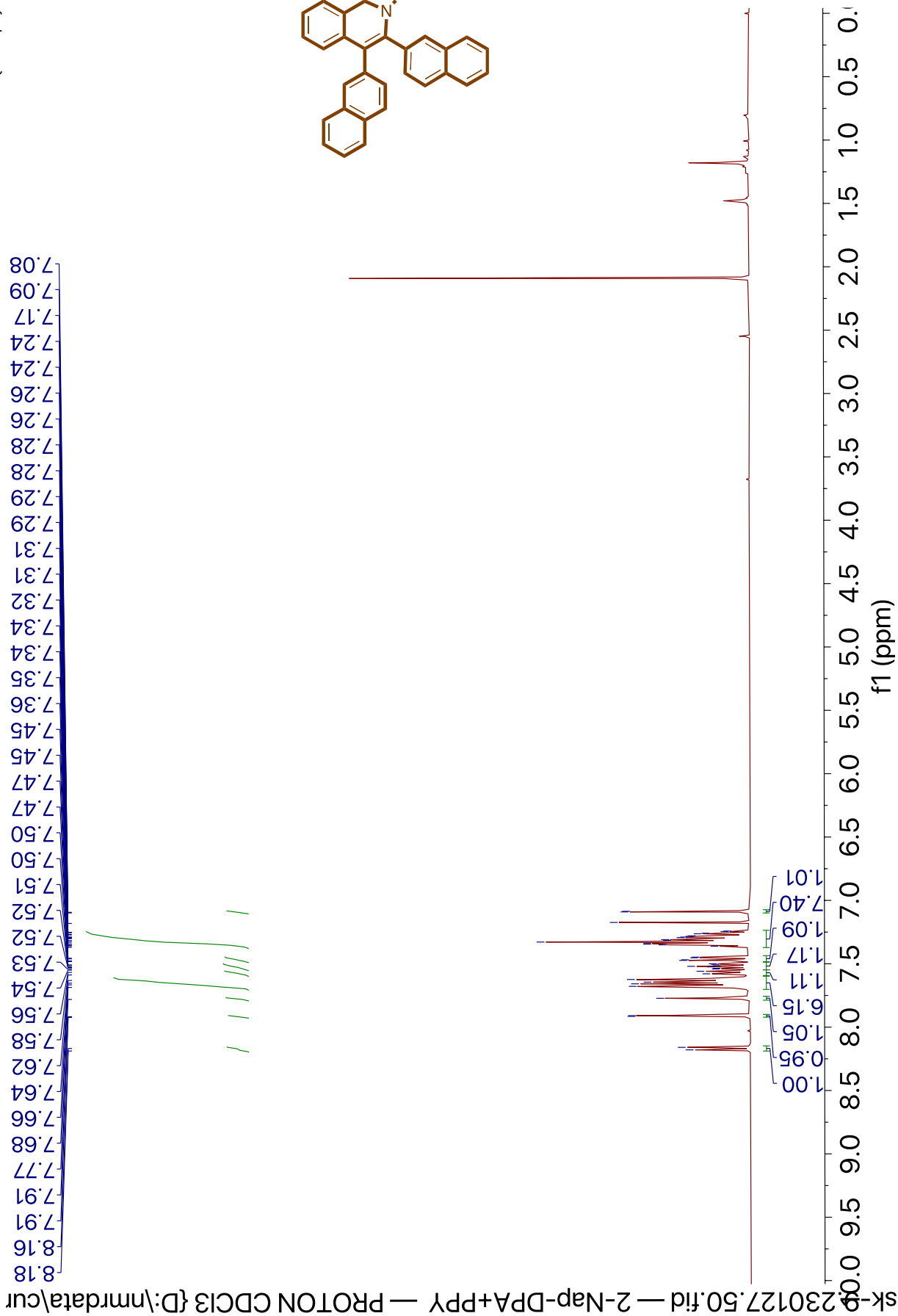


HRMS spectra of **5d**

Compound 5e, ¹H NMR, CDCl₃, 400 MHz

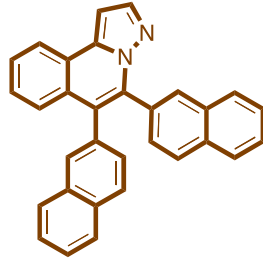


{name, 0}

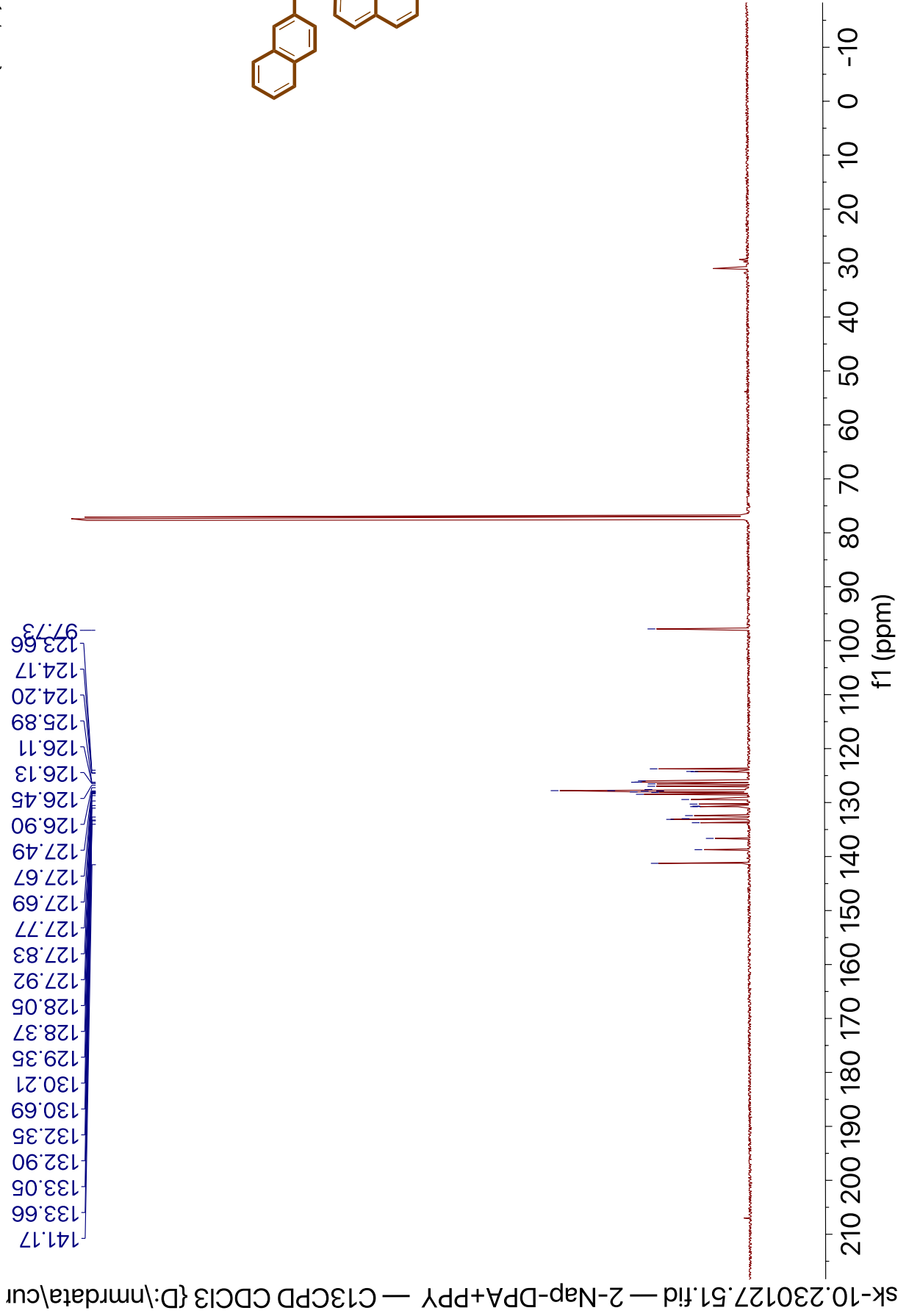


sk-9-230127.50.fid - 2-Nap-DPA+PPY - PROTON CDCl3 {D:\nmrdata\cur

Compound 5e, ¹³C NMR, CDCl₃, 101 MHz



{name, 0}

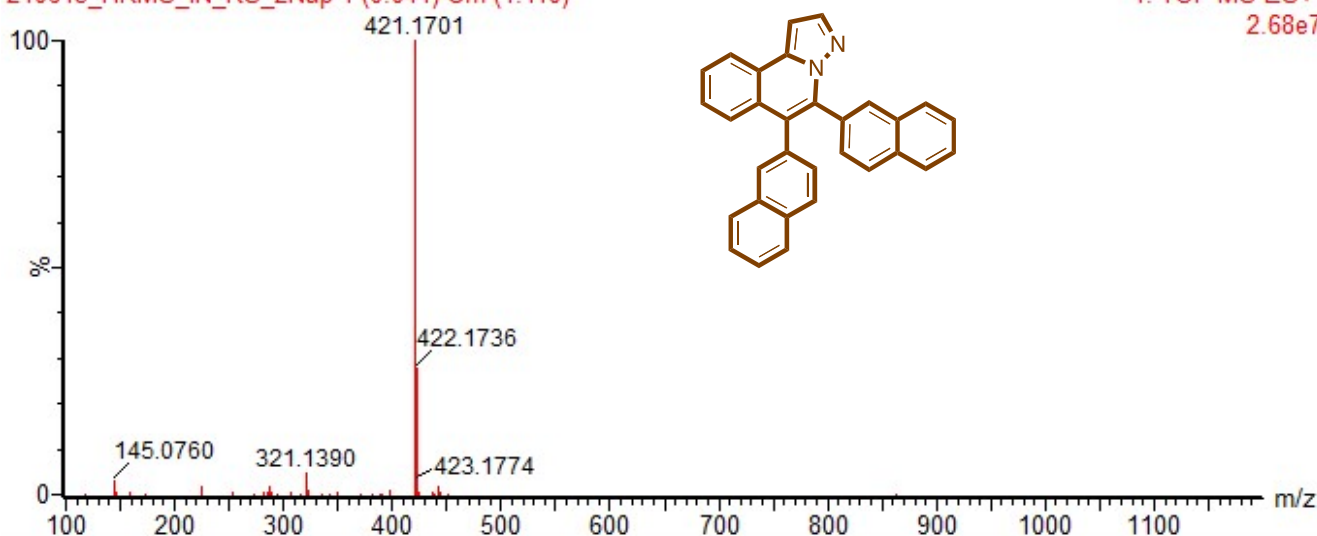


sk-10.230127.51.fid - 2-Nap-DPA+PY - C13CPD CDCl3 {D:\nmrdata\cur

DCM-> MeOH (2% H2O + 0.1% FA)

210615_HRMS_IN_KS_2Nap 1 (0.041) Cm (1:110)

1: TOF MS ES+
2.68e7



Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

256 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-50

H: 0-100

N: 0-5

O: 0-3

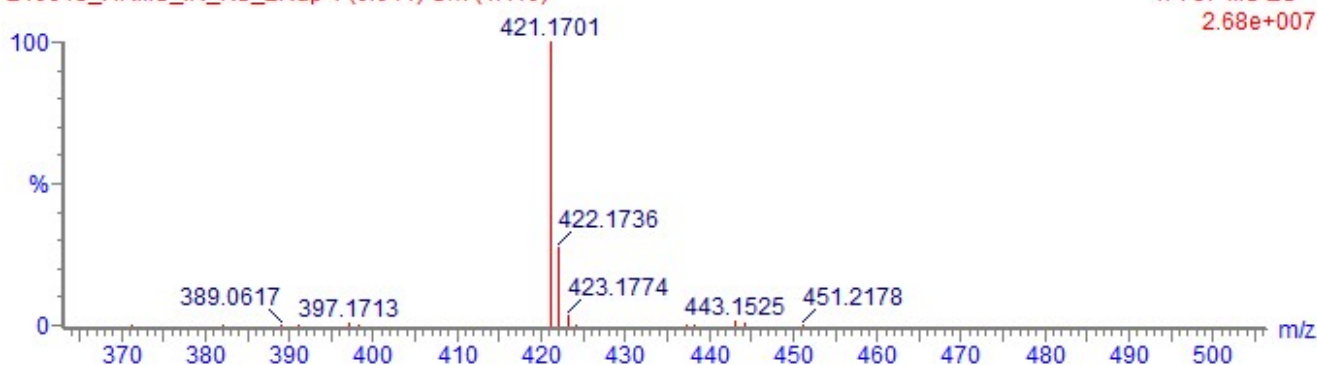
Na: 0-1

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	N	O	Na
421.1701	421.1705	-0.4	-0.9	22.5	C31 H21 N2	38.1	n/a	n/a	31	21	2		

DCM-> MeOH (2% H2O + 0.1% FA)

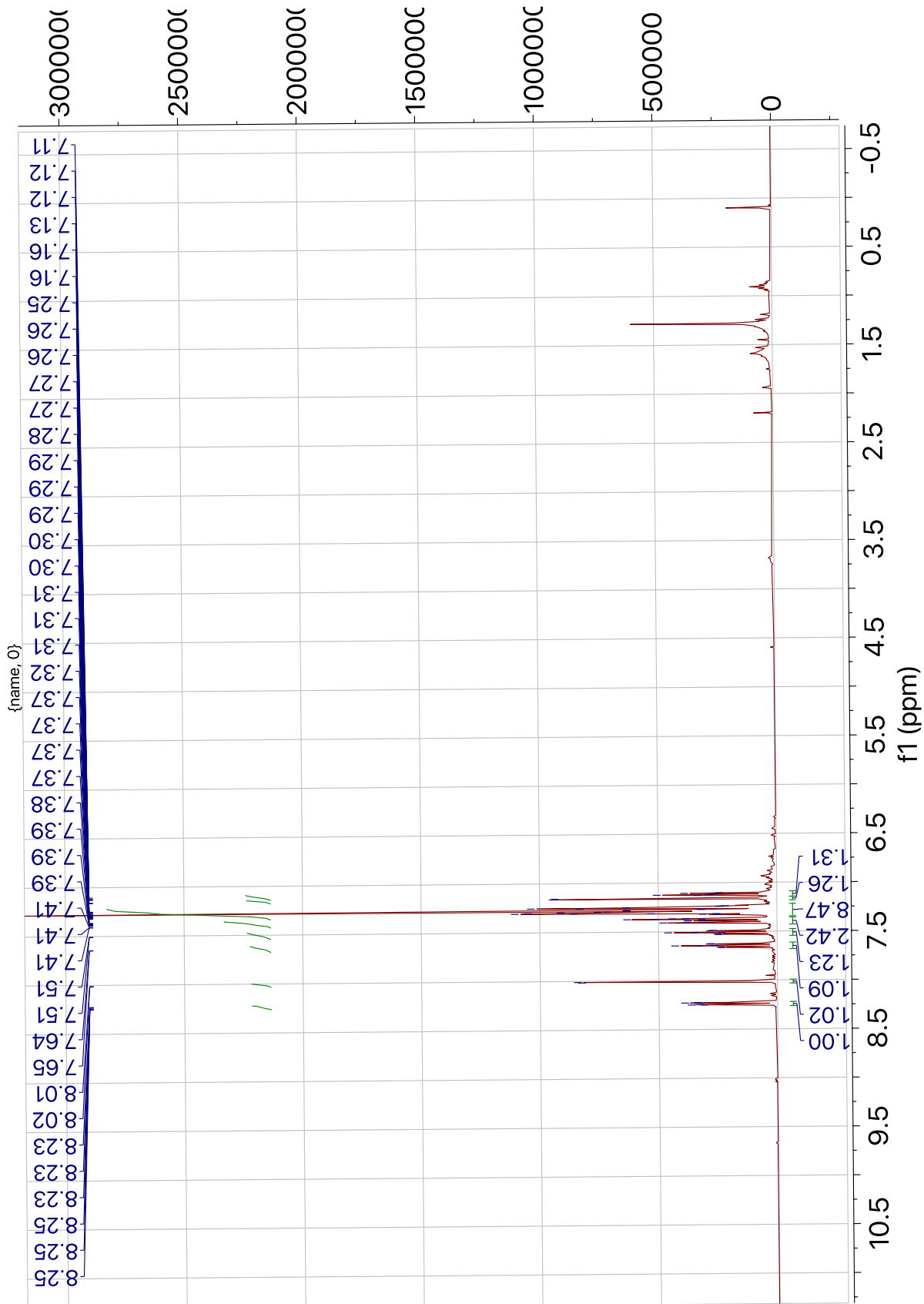
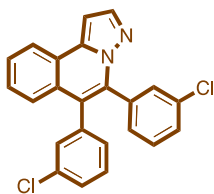
210615_HRMS_IN_KS_2Nap 1 (0.041) Cm (1:110)

1: TOF MS ES+
2.68e+007

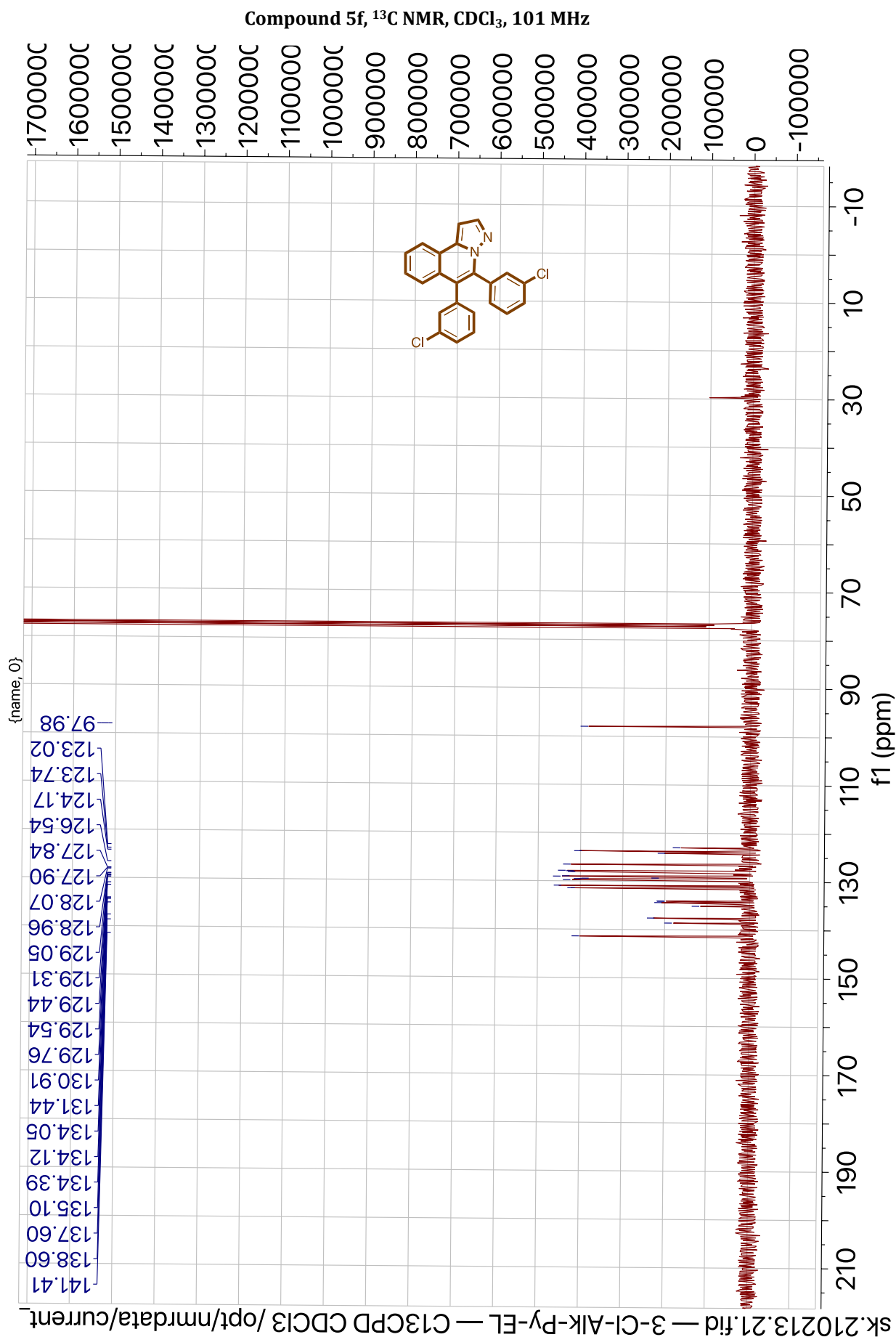


HRMS spectra of 5e

Compound 5f, ¹H NMR, CDCl₃, 400 MHz



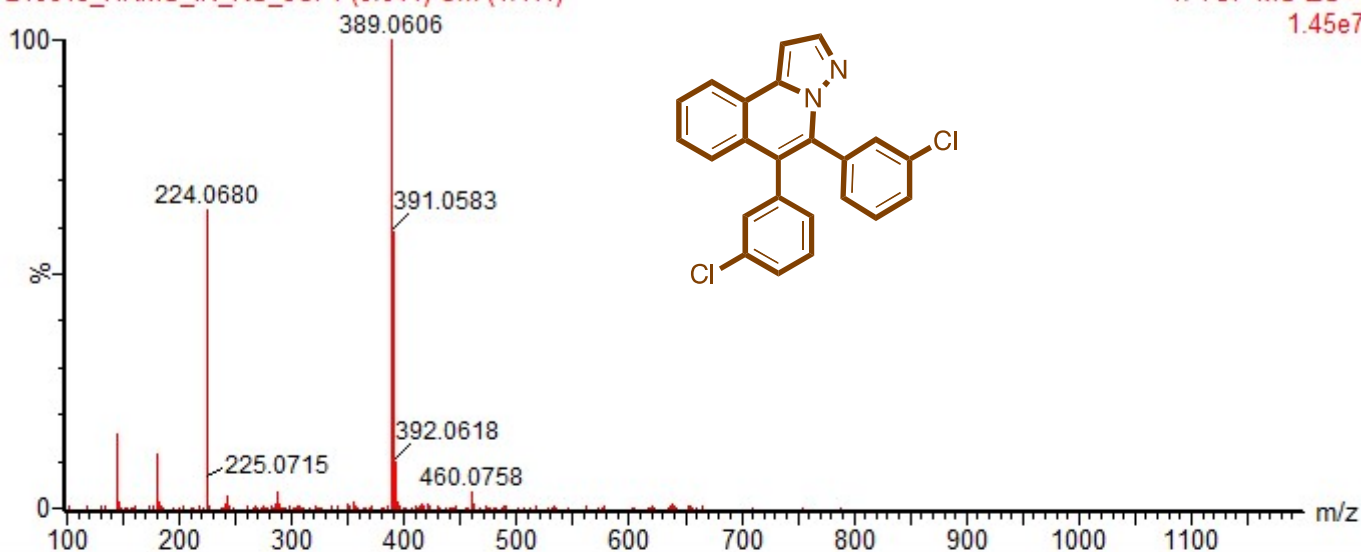
sk-3.210213.20.fid - 3-Cl-AIK-Py-EL - CMC_PROTON CDCl3 /opt/nmrdat



DCM-> MeOH (2% H2O + 0.1% FA)

210615_HRMS_IN_KS_3Cl 1 (0.041) Cm (1:111)

1: TOF MS ES+
1.45e7



Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

644 formula(e) evaluated with 4 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-50

H: 0-100

N: 0-5

O: 0-3

Na: 0-1

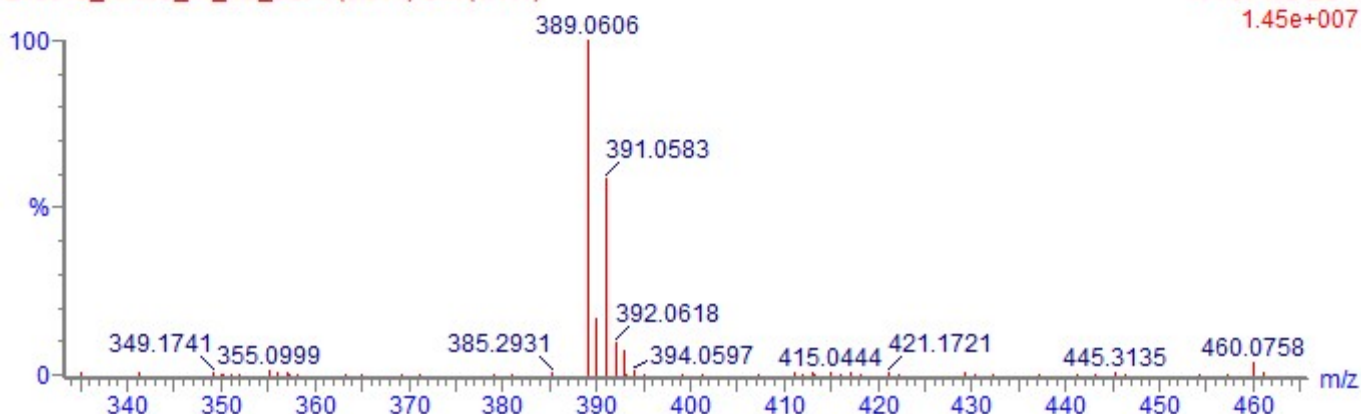
Cl: 0-2

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-FI...	Fit Conf %	C	H	N	O	Na	Cl
389.0606	389.0603	0.3	0.8	25.5	C29 H9 O2	516....	0.00	29	9		2		
	389.0612	-0.6	-1.5	16.5	C23 H15 N2 Cl2	30.134	87.48	23	15	2			2
	389.0594	1.2	3.1	21.5	C24 H10 N4 Cl	45.452	0.43	24	10	4			1
	389.0588	1.8	4.6	13.5	C21 H16 N2 Na Cl2	32.113	12.09	21	16	2		1	2

DCM-> MeOH (2% H2O + 0.1% FA)

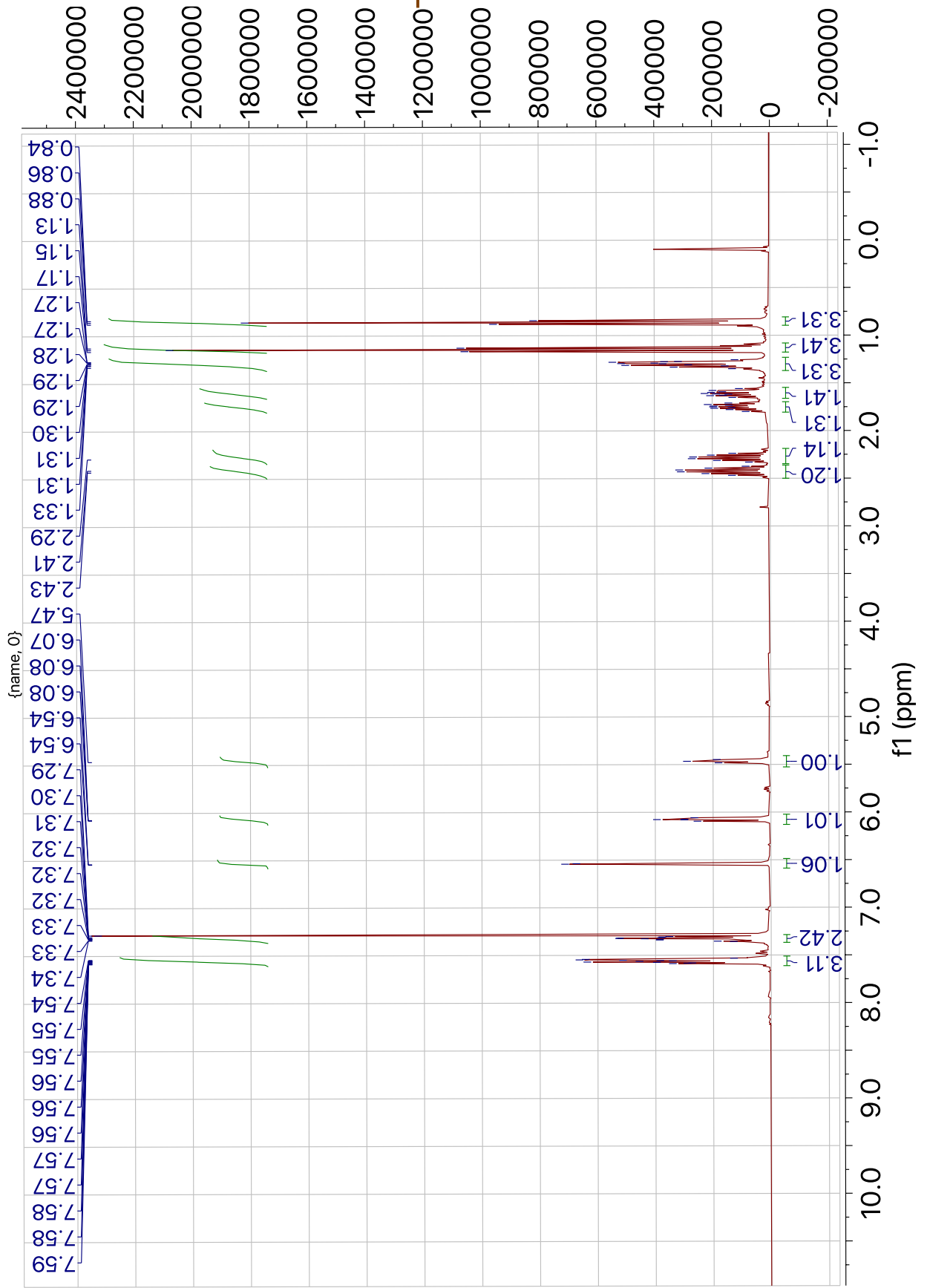
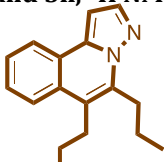
210615_HRMS_IN_KS_3Cl 1 (0.041) Cm (1:111)

1: TOF MS ES+
1.45e+007

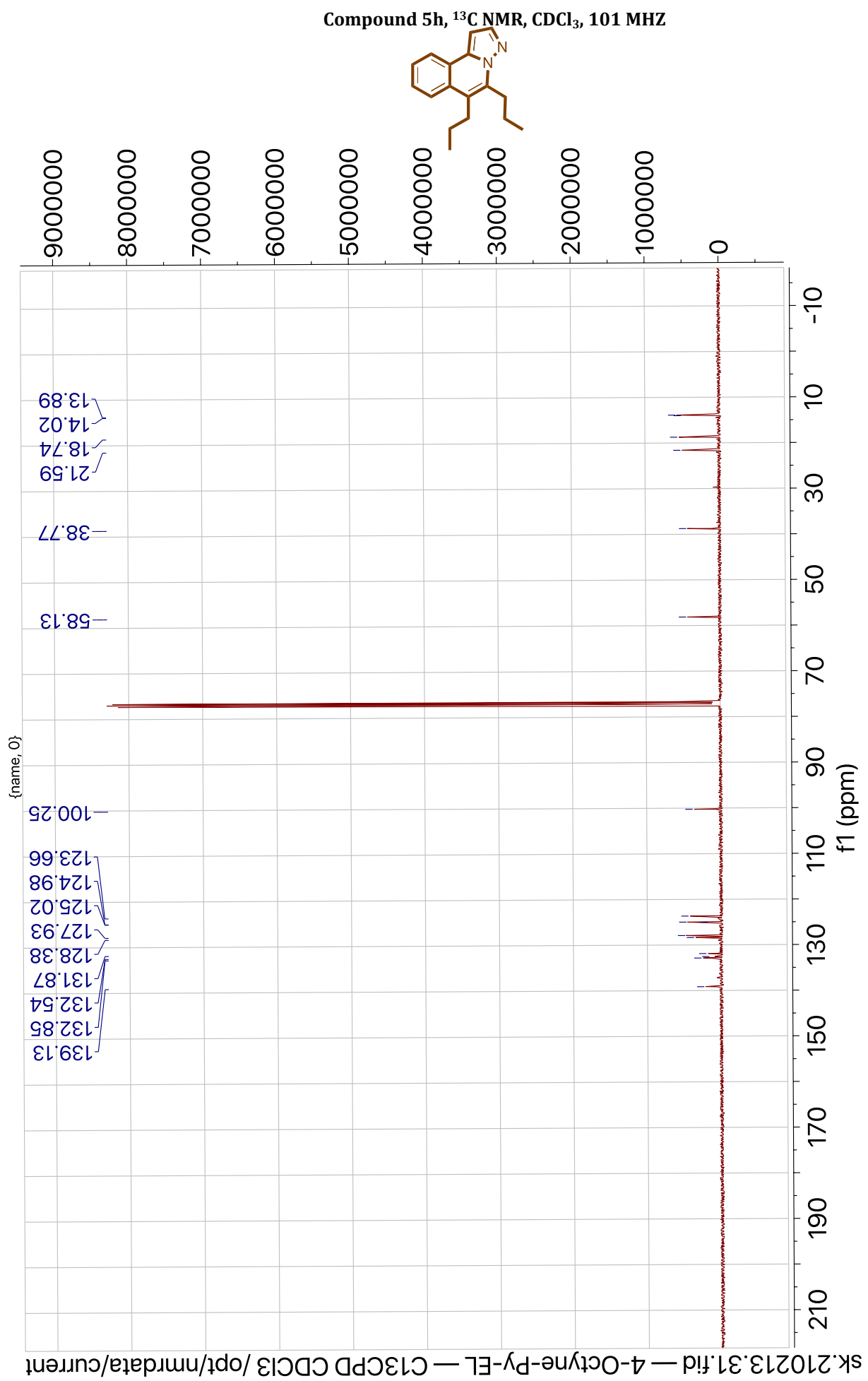


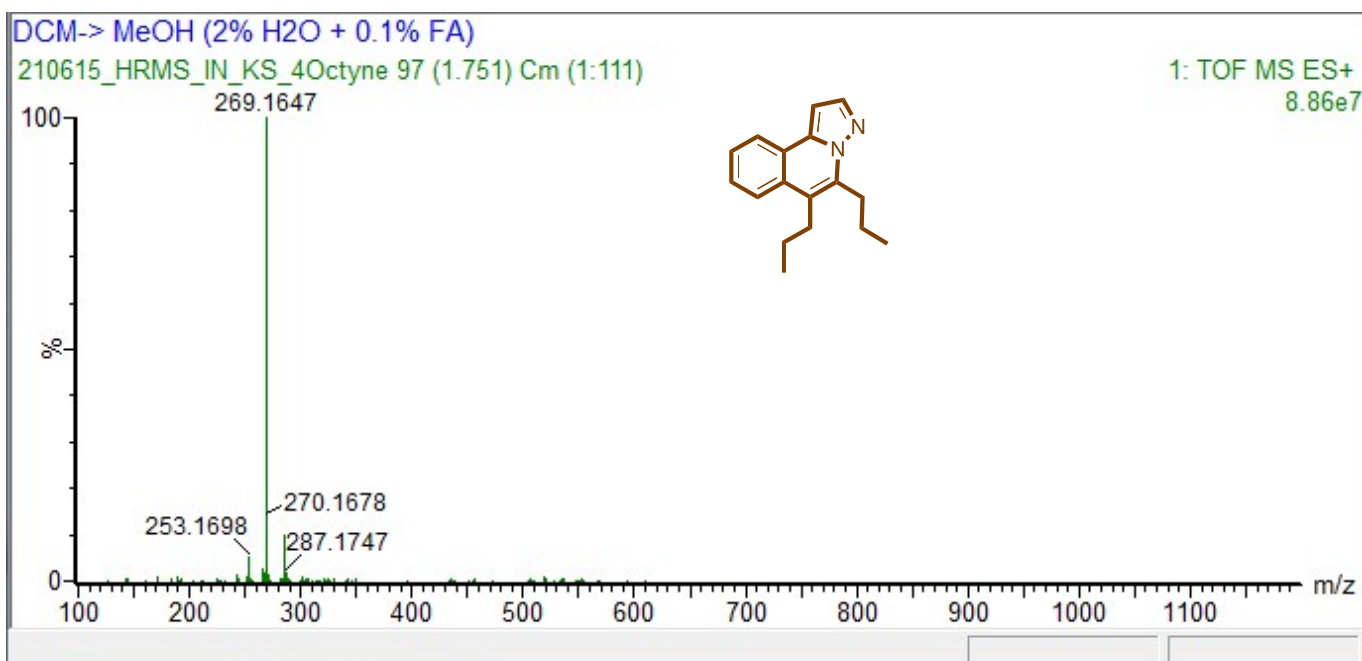
HRMS spectra of 5f

Compound 5h, ¹H NMR, CDCl₃, 400 MHz



sk-5.210213.30.fid — 4-Octyne-Py-EL — CMC_PROTON CDCl₃ /opt/nmrda





Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

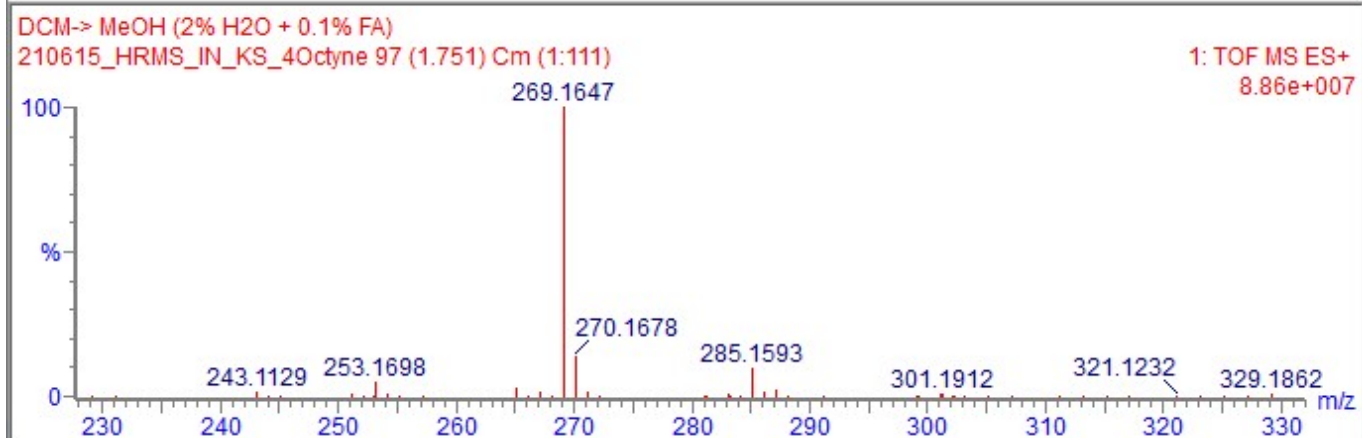
Monoisotopic Mass, Even Electron Ions

160 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

Elements Used:

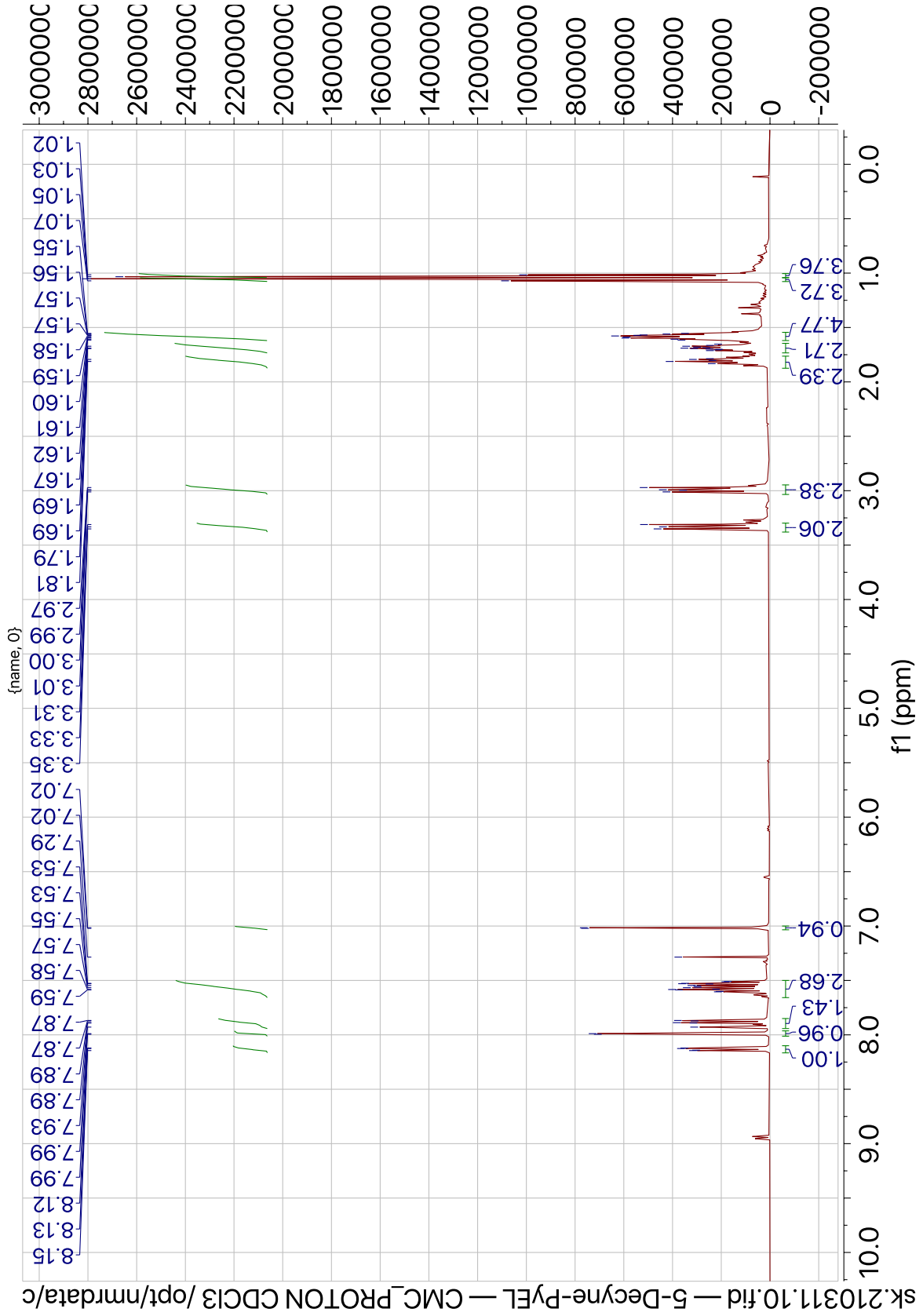
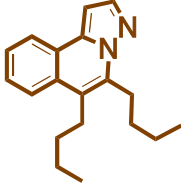
C: 0-50 H: 0-100 N: 0-5 O: 0-3 Na: 0-1

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i.	Fit Conf %	C	H	N	O	Na
253.1698	253.1705	-0.7	-2.8	8.5	C17 H21 N2	6	37.92	17	21	2		
	253.1681	1.7	6.7	5.5	C15 H22 N2 Na	6	62.08	15	22	2		1

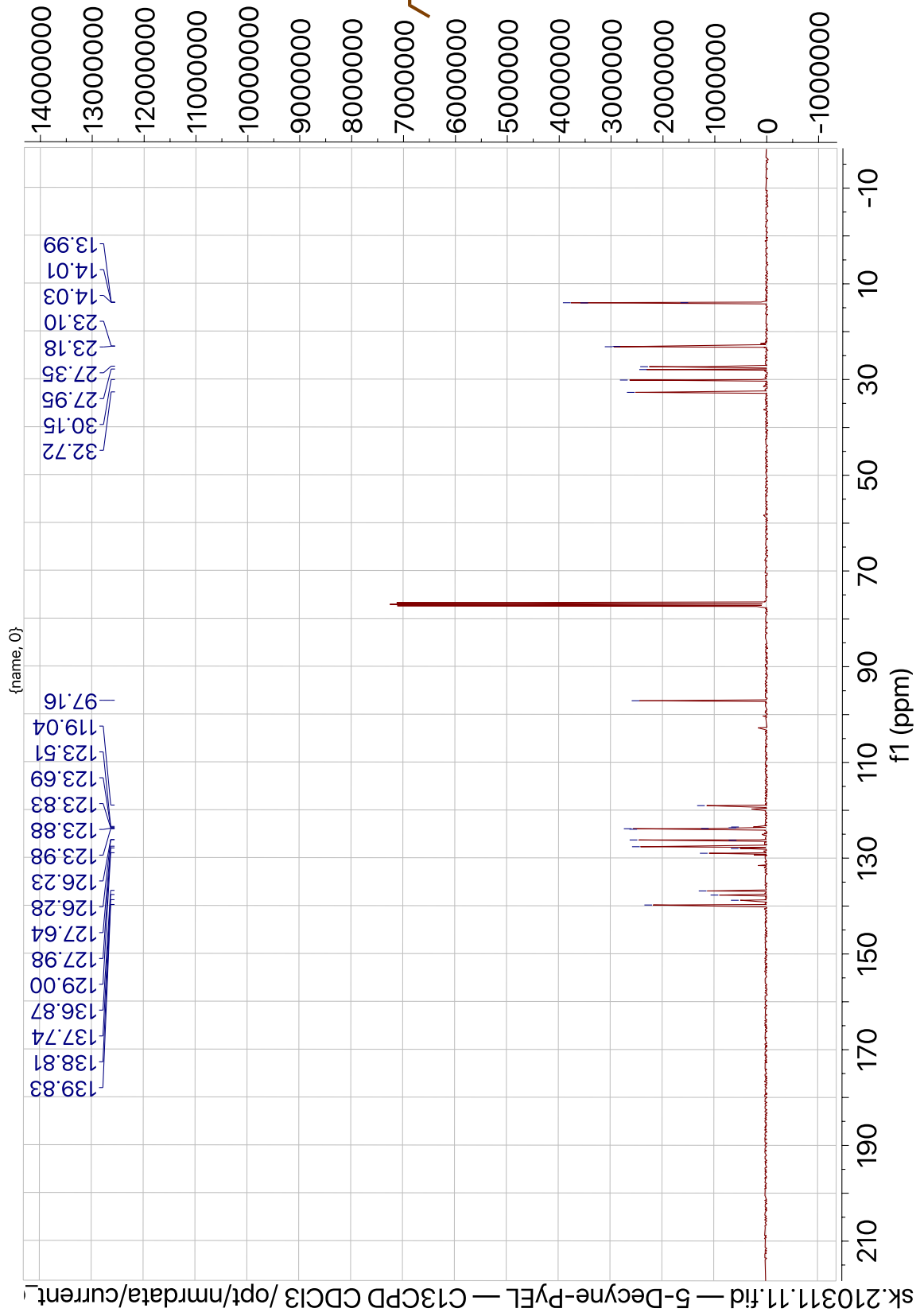
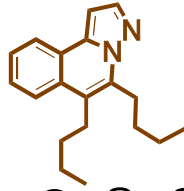


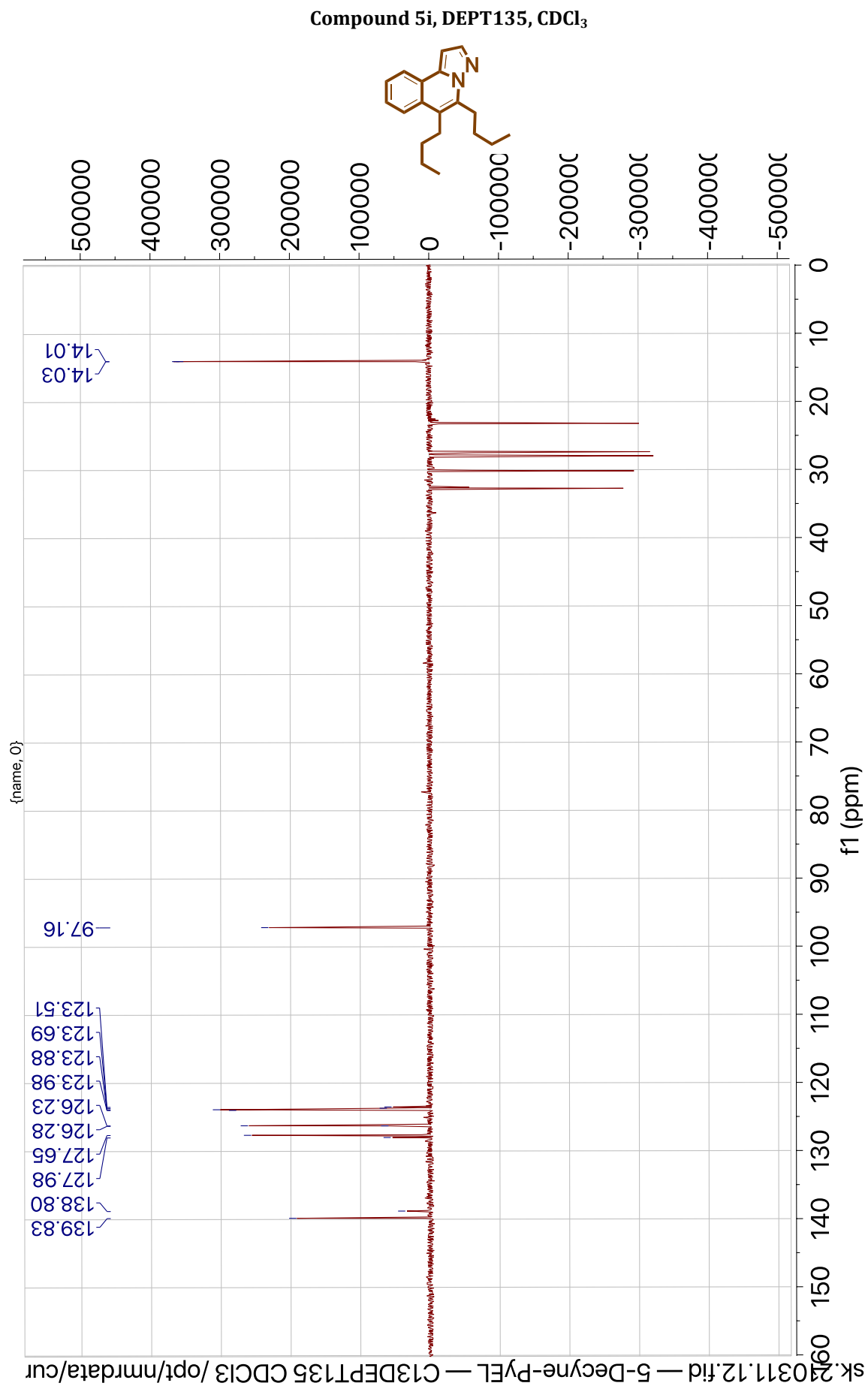
HRMS spectra of **5h**

Compound 5i, ¹H NMR, CDCl₃, 400 MHz



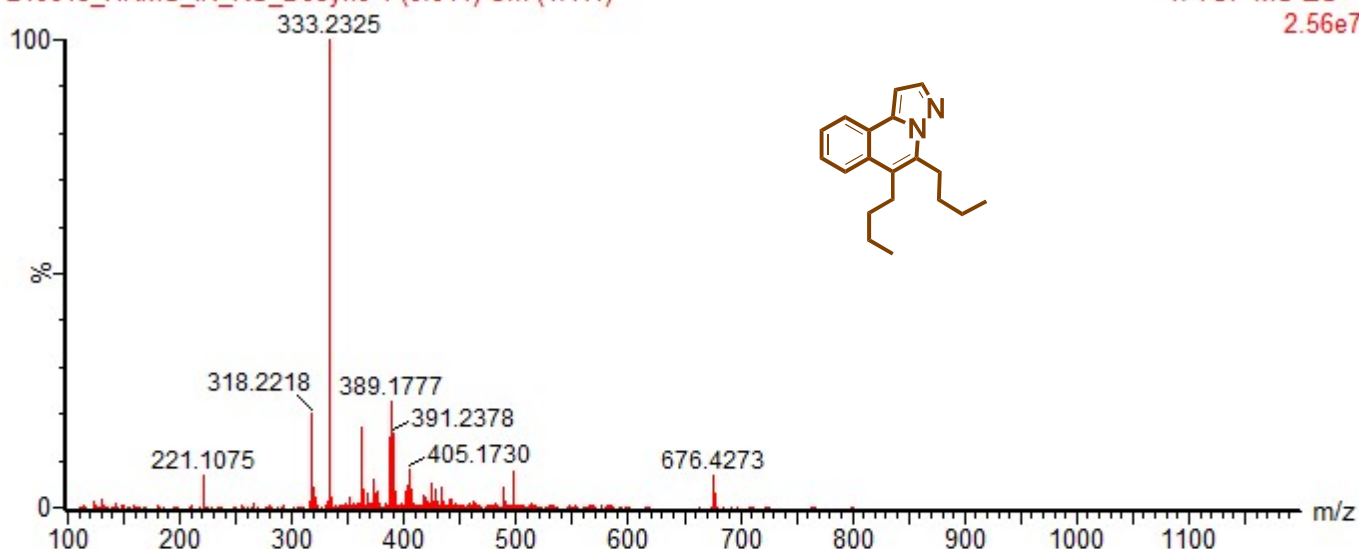
Compound 5i, ¹³C NMR, CDCl₃, 101 MHz





DCM-> MeOH (2% H2O + 0.1% FA)
 210615_HRMS_IN_KS_Decyne 1 (0.041) Cm (1:111)

1: TOF MS ES+
 2.56e7



Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

207 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

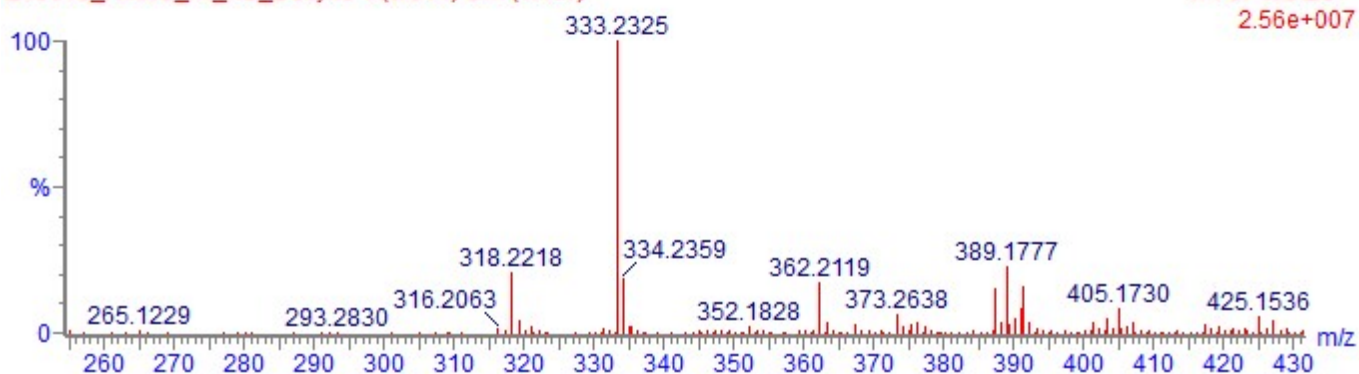
Elements Used:

C: 0-50 H: 0-100 N: 0-5 O: 0-3 Na: 0-1

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i, Fit Conf ...	C	H	N	O	Na
333.2325	333.2331	-0.6	-1.8	10.5	C23 H29 N2	106.8	0..45.27	23	29	2		
	333.2307	1.8	5.4	7.5	C21 H30 N2 Na	106.6	0..54.73	21	30	2		1

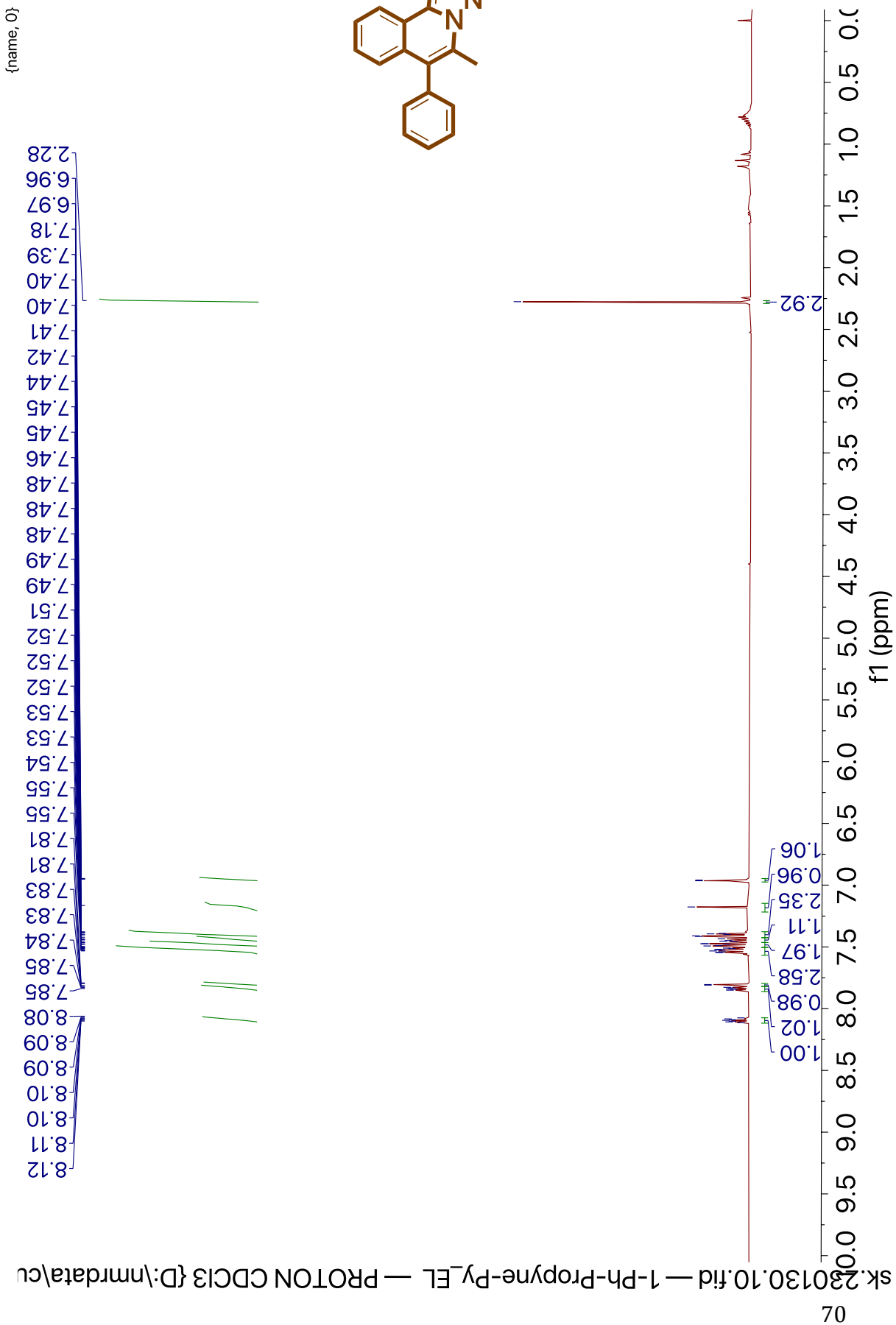
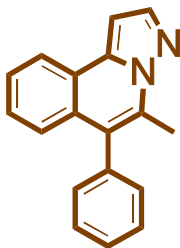
DCM-> MeOH (2% H2O + 0.1% FA)
 210615_HRMS_IN_KS_Decyne 1 (0.041) Cm (1:111)

1: TOF MS ES+
 2.56e+007

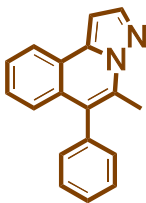


HRMS spectra of **5i**

Compound 5j, ¹H NMR, CDCl₃, 400 MHz



Compound 5j, ¹³C NMR, CDCl₃, 101 MHz



{name, 0}

-15.02

140.47
138.01
135.94
133.88
130.40
129.86
128.91
128.73
127.86
127.25
124.46
124.18
123.88
116.25
97.32

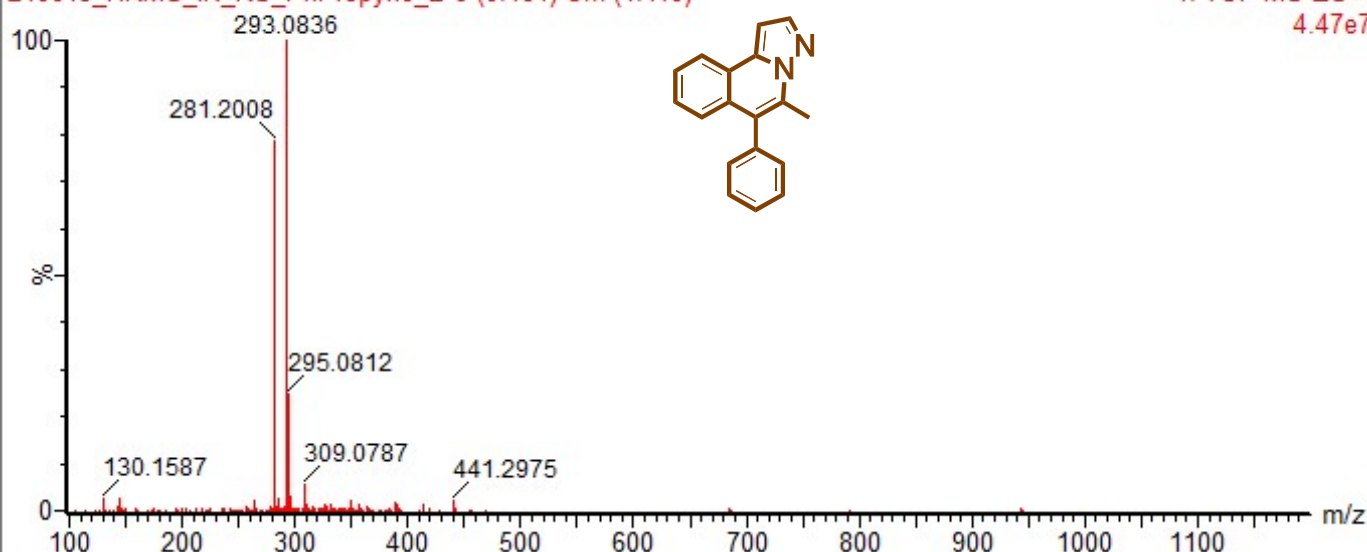
f1 (ppm)

sk-4.230130.11.fid - 1-Ph-Propyne-Py_EL - C13CPD CDCl3 {D:\nmrdata\}

DCM-> MeOH (2% H2O + 0.1% FA)

210615_HRMS_IN_KS_PhPropyne_B 9 (0.184) Cm (1:110)

1: TOF MS ES+
4.47e7



Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

176 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-50

H: 0-100

N: 0-5

O: 0-3

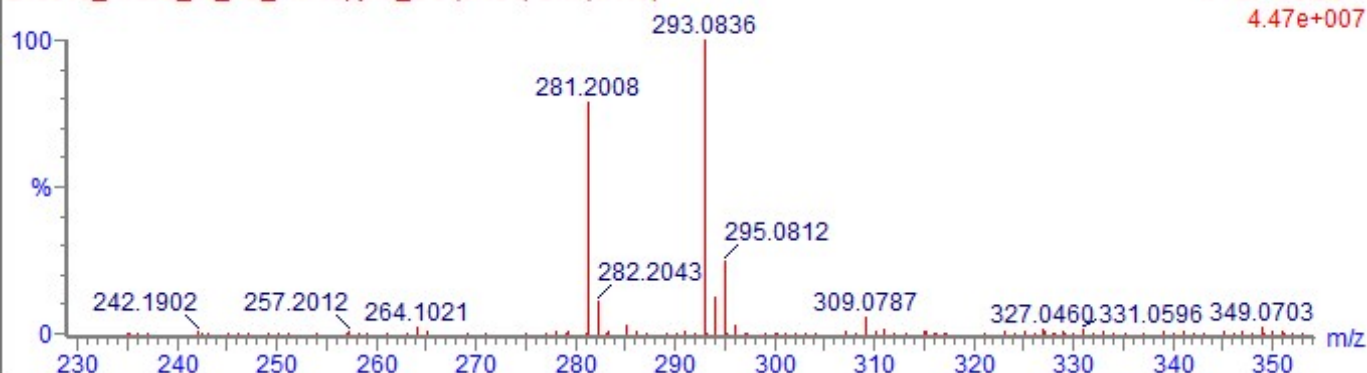
Na: 0-1

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-...	Fit Conf %	C	H	N	O	Na
281.2008	281.2018	-1.0	-3.6	8.5	C19 H25 N2	6...	35.66	19	25	2		
	281.1994	1.4	5.0	5.5	C17 H26 N2 Na	6...	64.34	17	26	2		1

DCM-> MeOH (2% H2O + 0.1% FA)

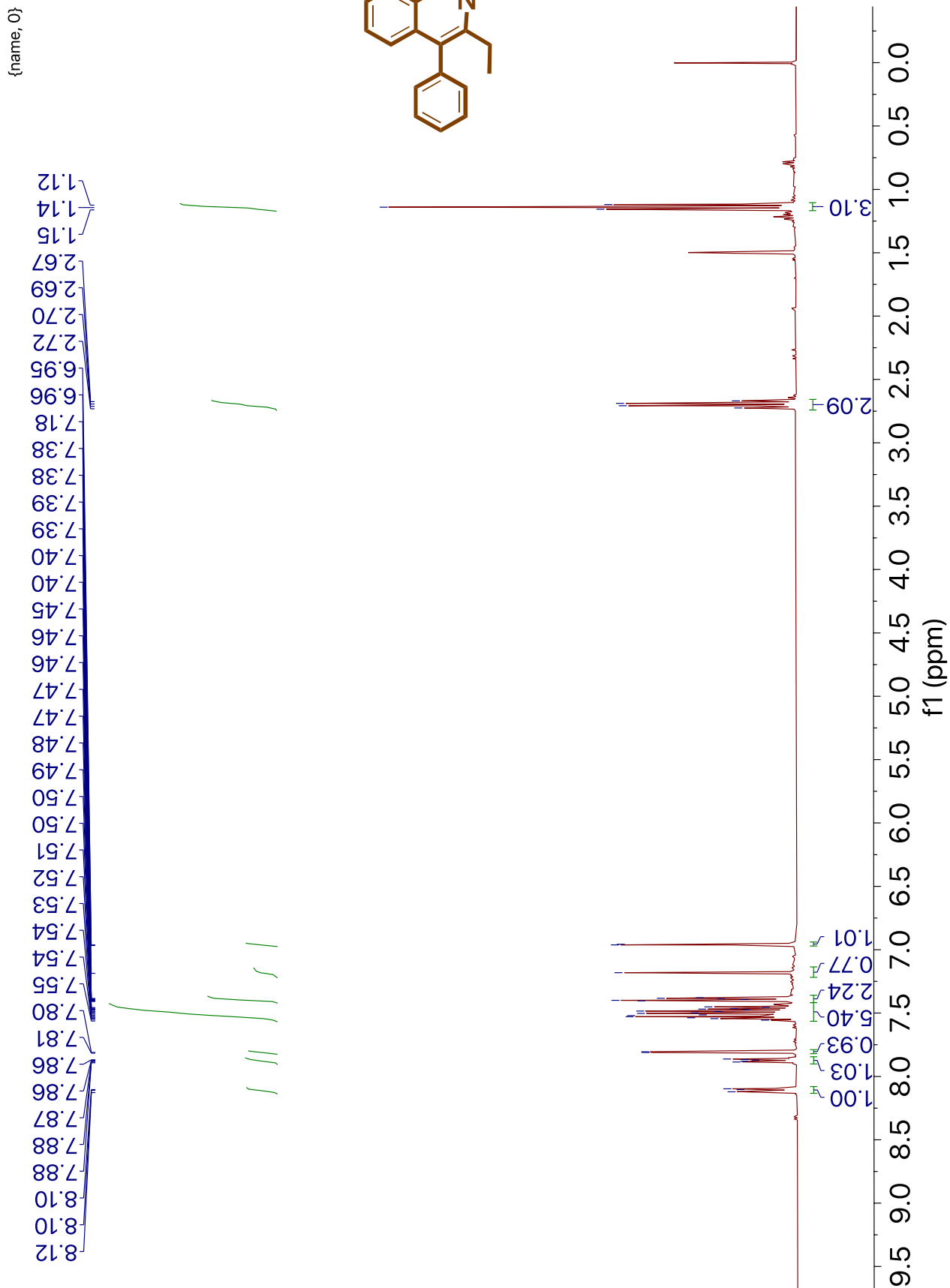
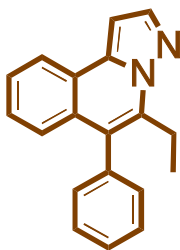
210615_HRMS_IN_KS_PhPropyne_B 9 (0.184) Cm (1:110)

1: TOF MS ES+
4.47e+007



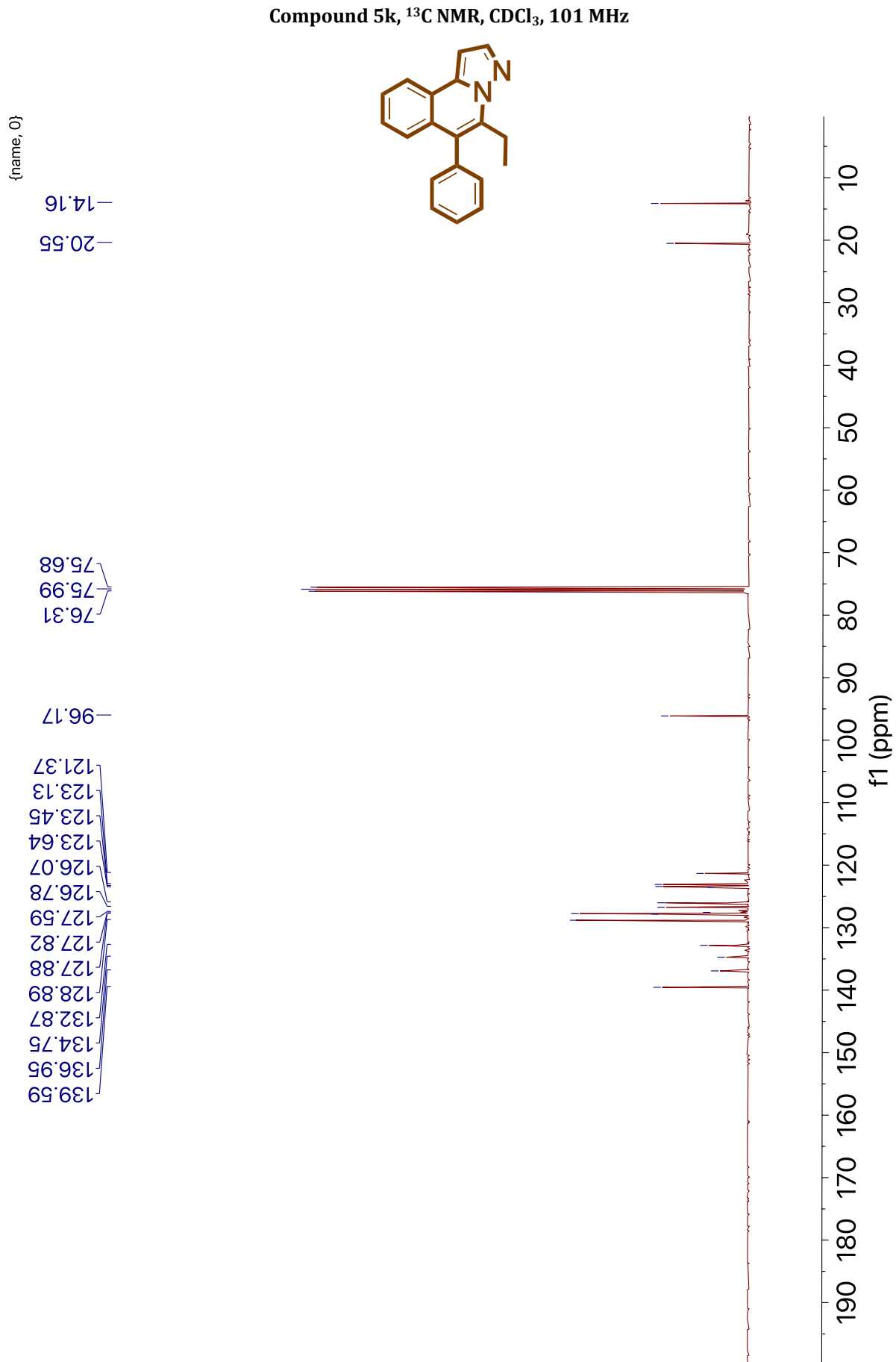
HRMS spectra of 5j

Compound 5k, ¹H NMR, CDCl₃, 400 MHz



sk-2.230130.20.fid - 1-Ph-Butyne-Py-EL - PROTON CDCl₃ {D:\nmrdata\}

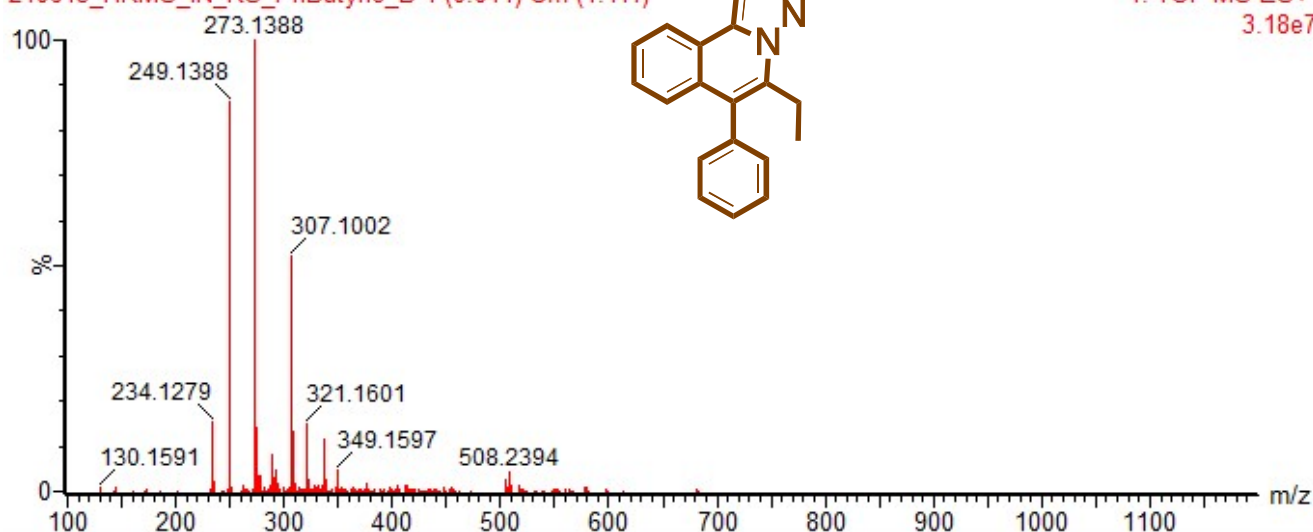
sk-3.230130.21.fid — 1-Ph-Butyne-Py-EL — C13CPD CDCl3 {D:\nmrdata\c



DCM-> MeOH (2% H2O + 0.1% FA)

210615_HRMS_IN_KS_PhButyne_B 1 (0.041) Cm (1:111)

1: TOF MS ES+
3.18e7



Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

171 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

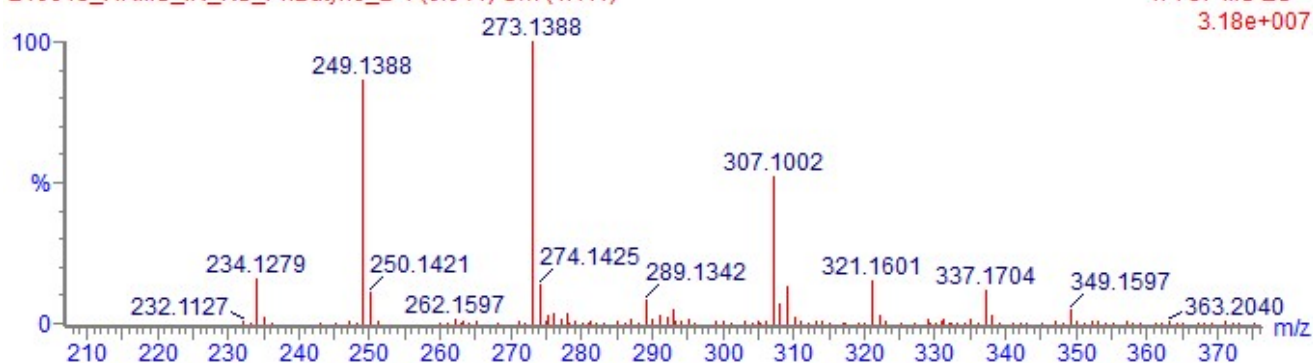
C: 0-50 H: 0-100 N: 0-5 O: 0-3 Na: 0-1

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	N	O	Na
273.1388	273.1392	-0.4	-1.5	12.5	C19 H17 N2	70.5	n/a	n/a	19	17	2		

DCM-> MeOH (2% H2O + 0.1% FA)

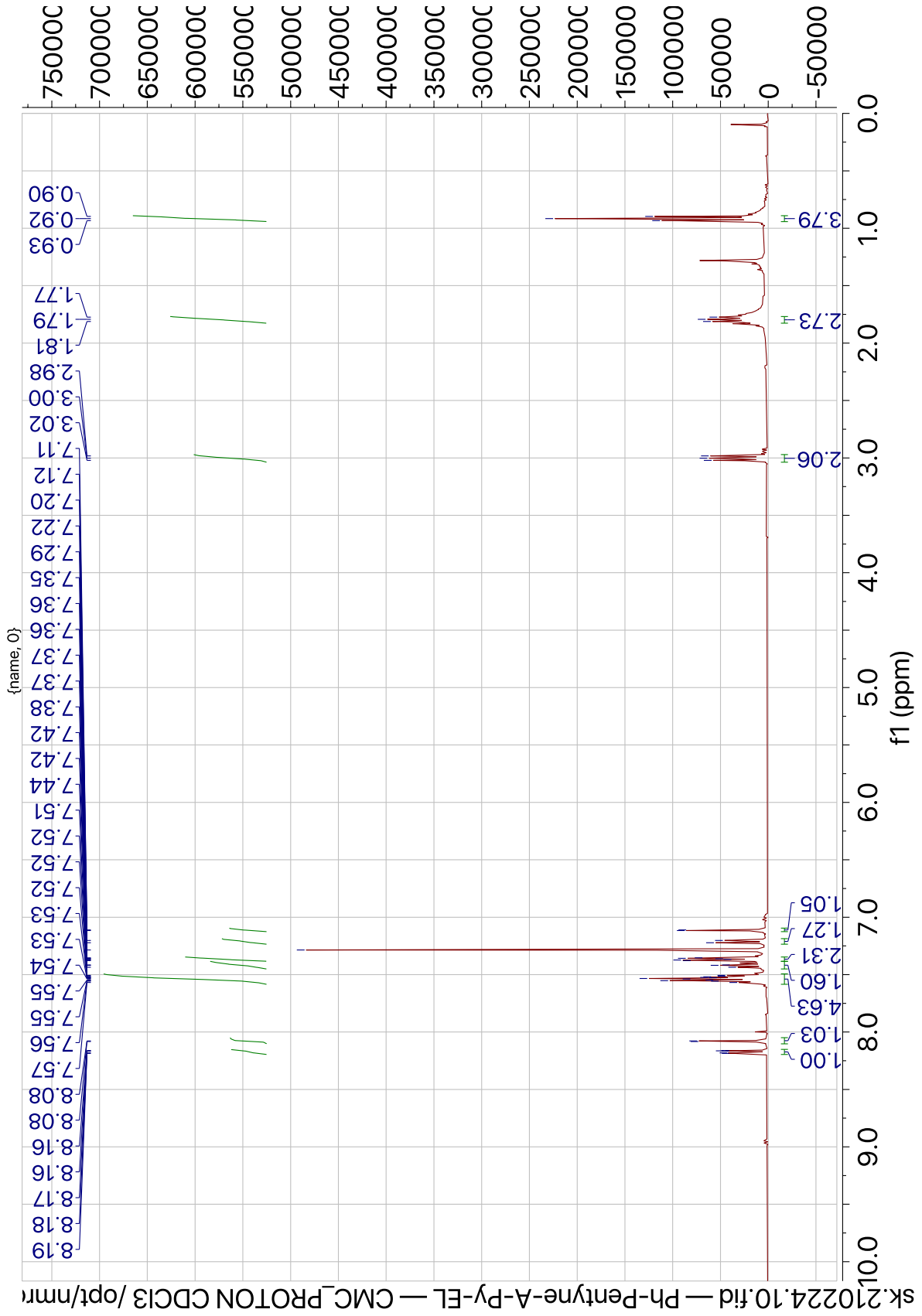
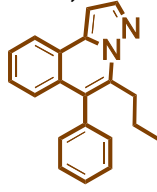
210615_HRMS_IN_KS_PhButyne_B 1 (0.041) Cm (1:111)

1: TOF MS ES+
3.18e+007

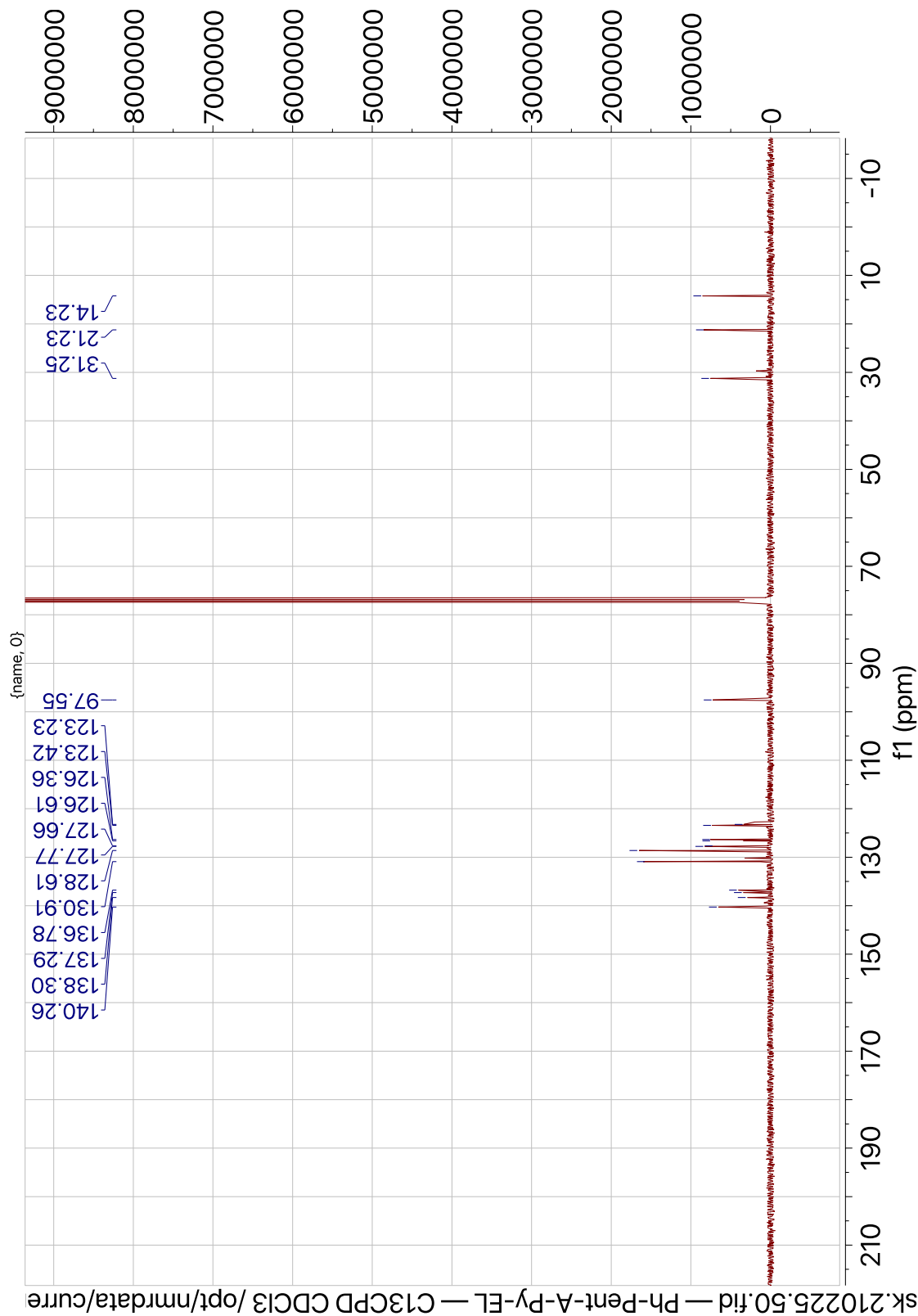
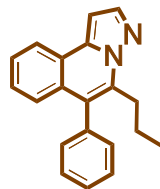


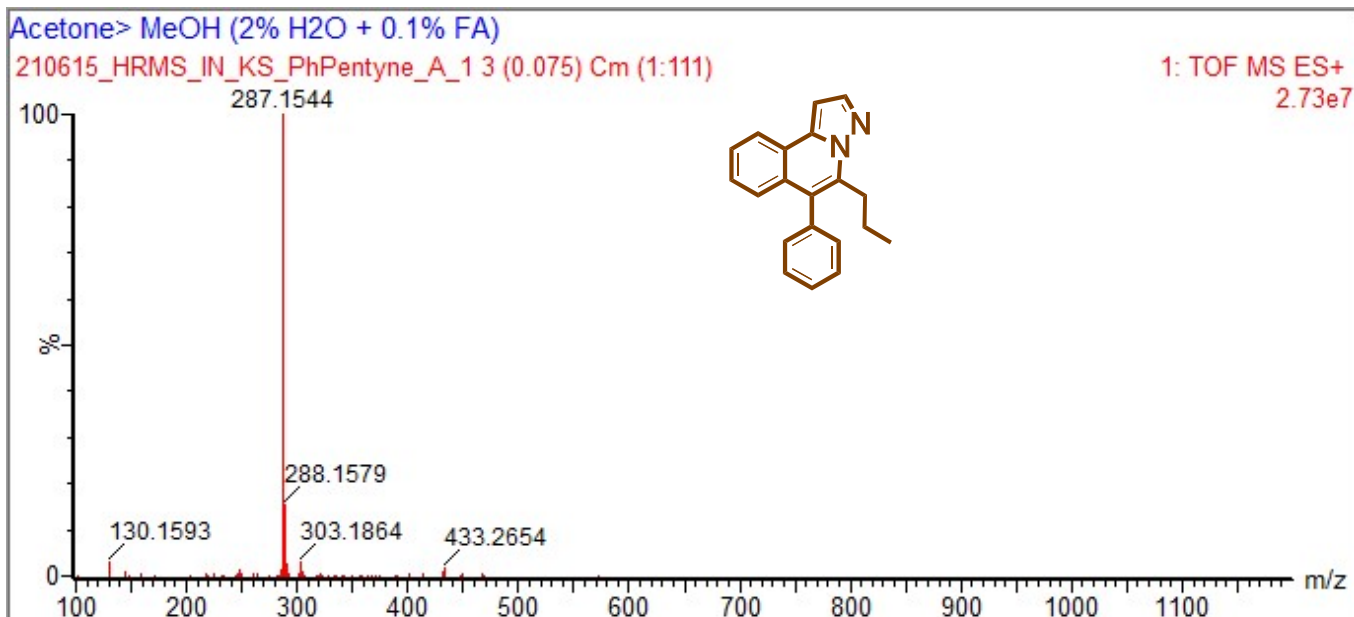
HRMS spectra of **5k**

Compound 5l, ¹H NMR, CDCl₃, 400 MHz



Compound 5l, ¹³C NMR, CDCl₃, 101 MHz





Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

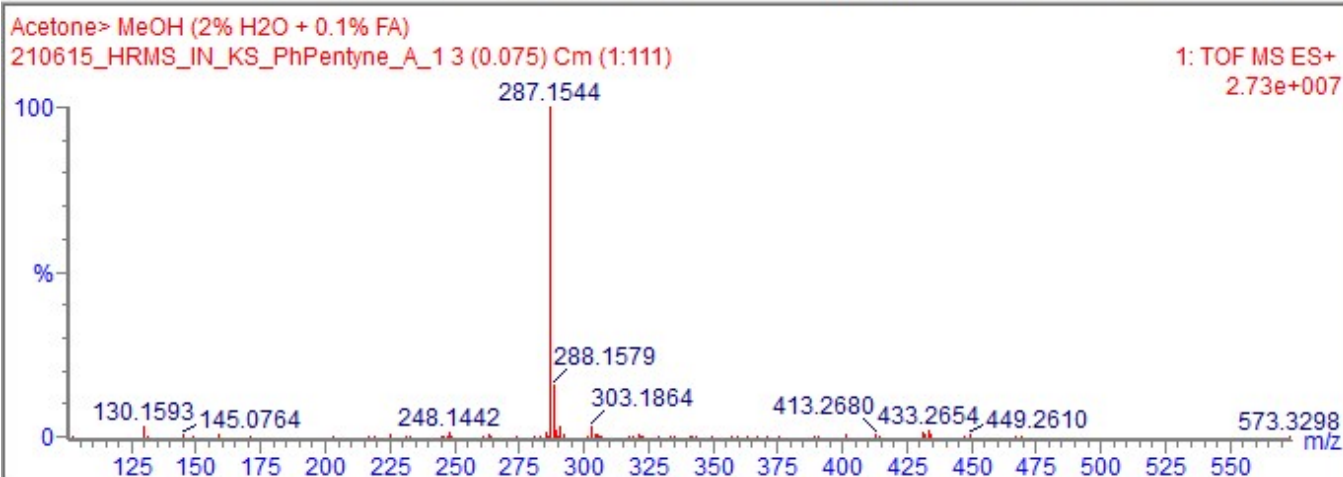
Monoisotopic Mass, Even Electron Ions

464 formula(e) evaluated with 5 results within limits (all results (up to 1000) for each mass)

Elements Used:

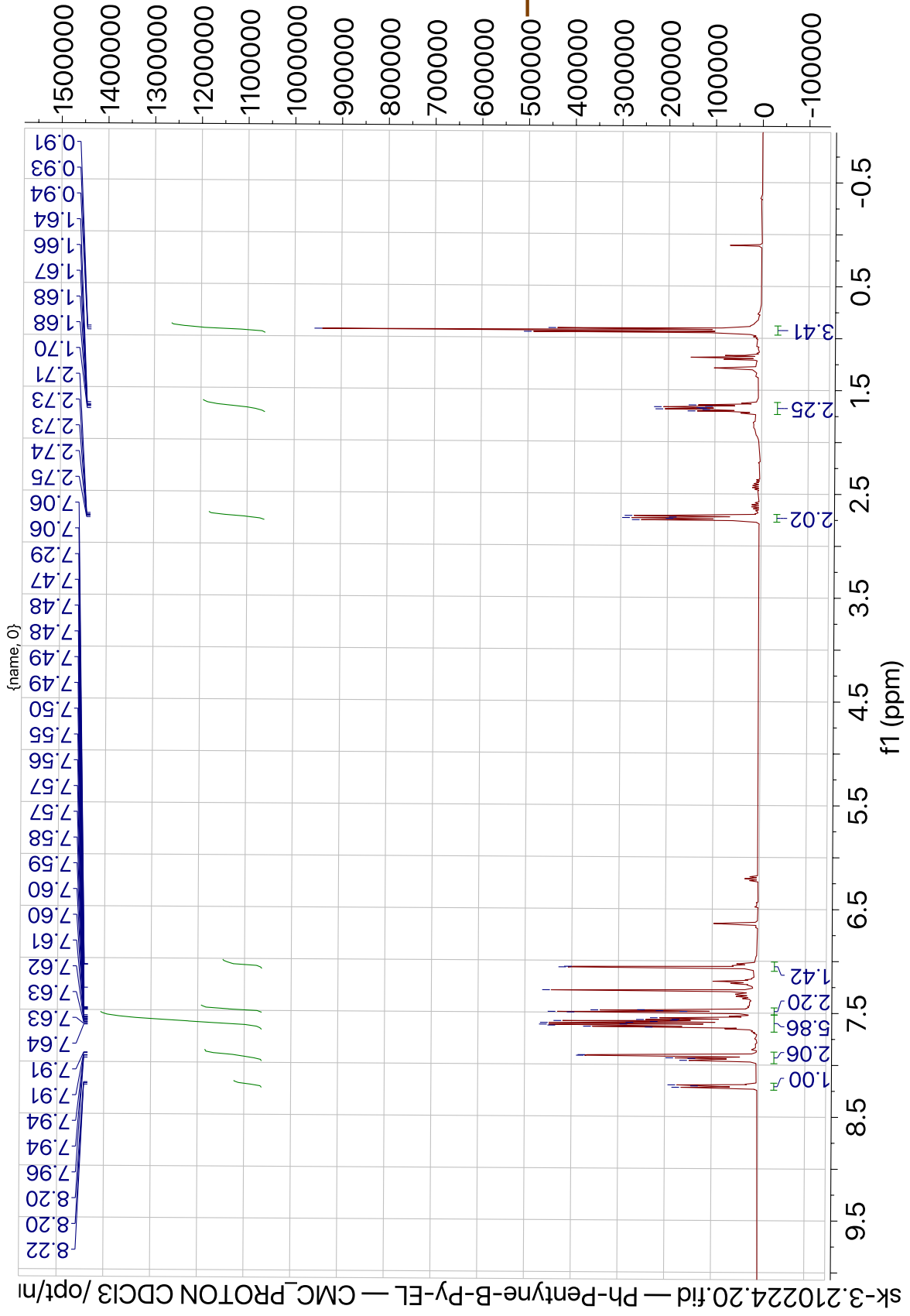
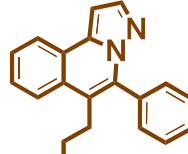
C: 0-50 H: 0-100 N: 0-5 O: 0-3 Na: 0-1 Cl: 0-2

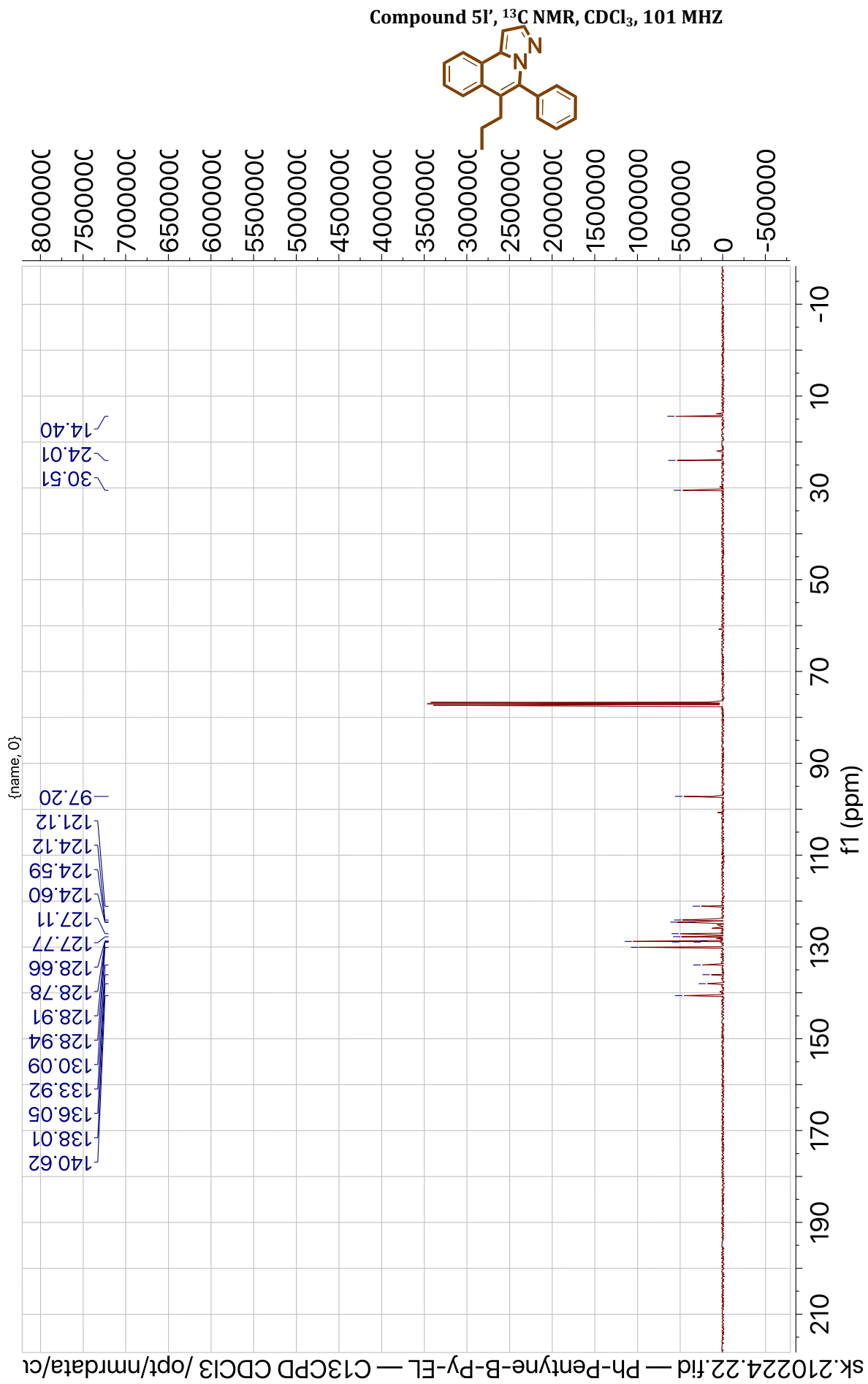
Mass	Calc. Mass	mDa	PPM	DBE	Formula	i...	Fit Conf %	C	H	N	O	Na	Cl
287.1544	287.1545	-0.1	-0.3	-1.5	C13 H29 O2 Cl2	71...	0.00	13	29		2		2
287.1542	287.1542	0.2	0.7	4.5	C17 H25 Na Cl	71...	0.00	17	25			1	1
287.1548	287.1548	-0.4	-1.4	12.5	C20 H19 N2	61...	28.96	20	19	2			
287.1526	287.1526	1.8	6.3	3.5	C14 H24 N2 O2 Cl	71...	0.00	14	24	2	2		1



HRMS spectra of **5I**

Compound 5I', ¹H NMR, CDCl₃, 400 MHz

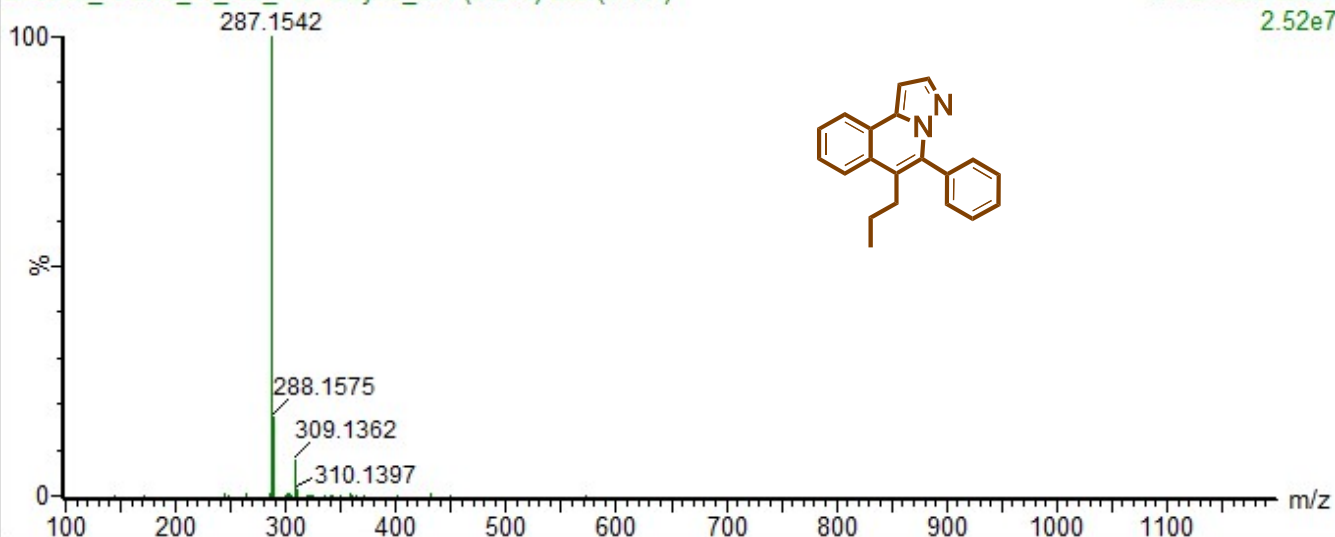




DCM-> MeOH (2% H2O + 0.1% FA)

210615_HRMS_IN_KS_PhPentyne_B 1 (0.041) Cm (1:111)

1: TOF MS ES+
2.52e7



Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

179 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

Elements Used:

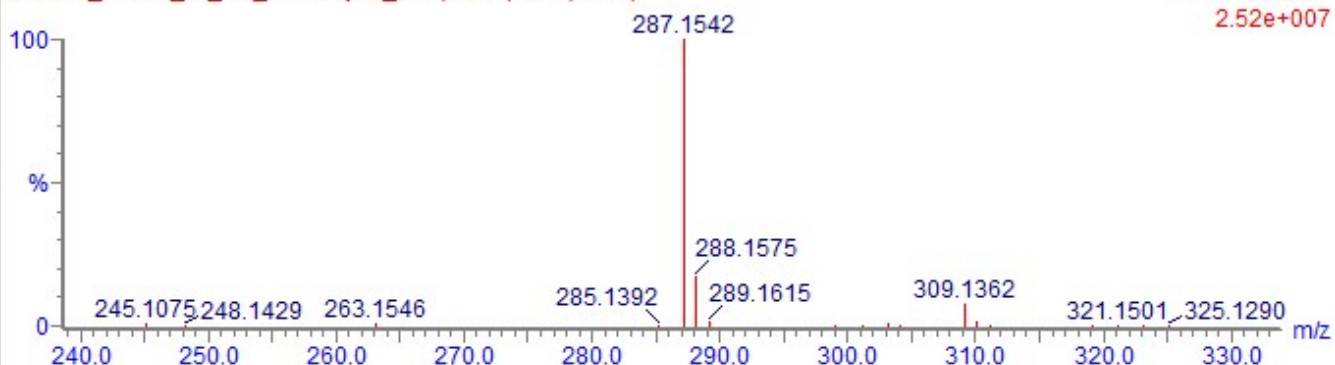
C: 0-50 H: 0-100 N: 0-5 O: 0-3 Na: 0-1

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i...	Fit Conf %	C	H	N	O	Na
287.1542	287.1548	-0.6	-2.1	12.5	C20 H19 N2	3.0...	42.44	20	19	2		
	287.1524	1.8	6.3	9.5	C18 H20 N2 Na	3.0...	57.56	18	20	2		1

DCM-> MeOH (2% H2O + 0.1% FA)

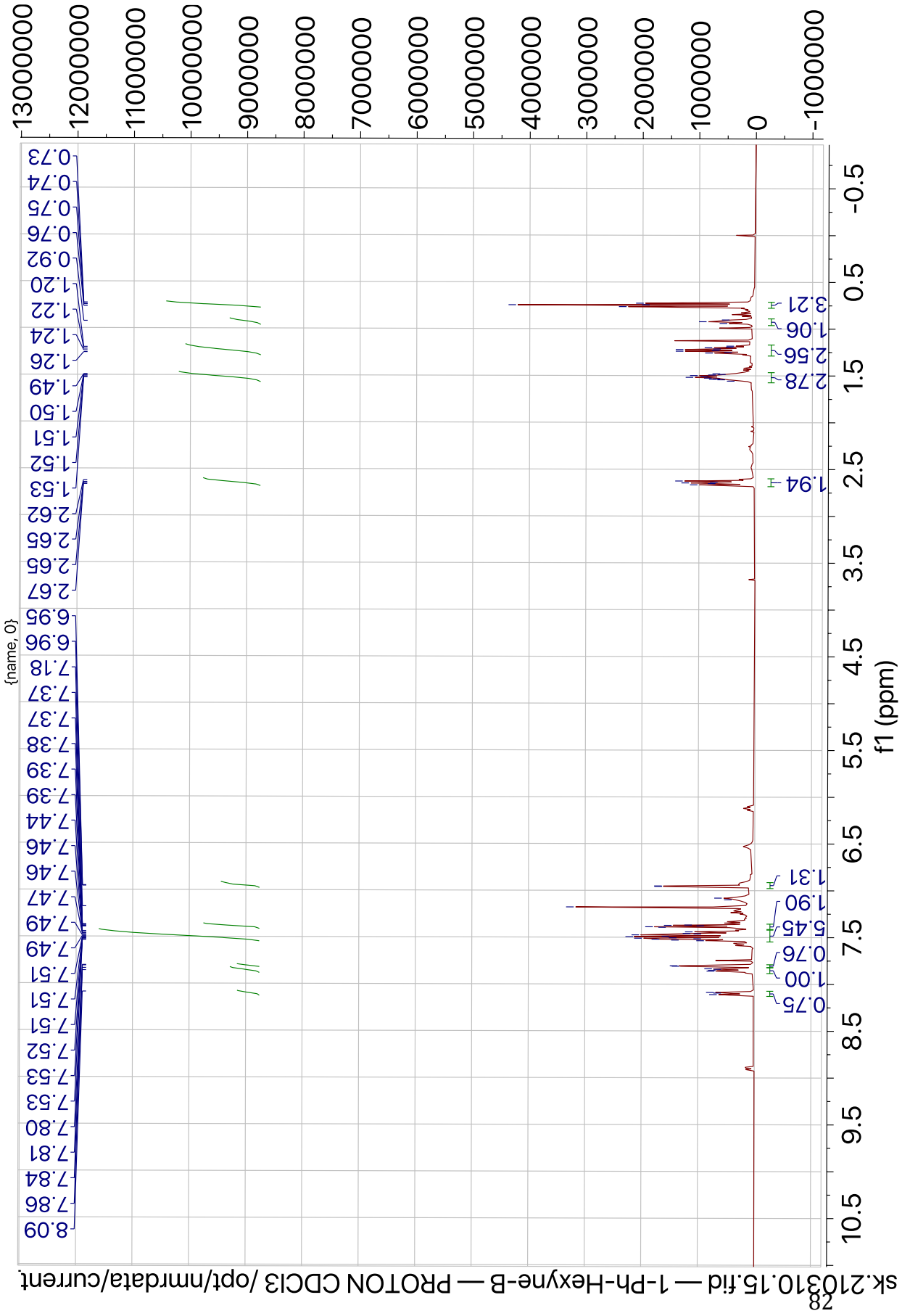
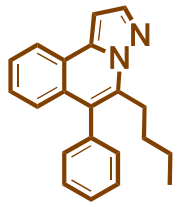
210615_HRMS_IN_KS_PhPentyne_B 1 (0.041) Cm (1:111)

1: TOF MS ES+
2.52e+007



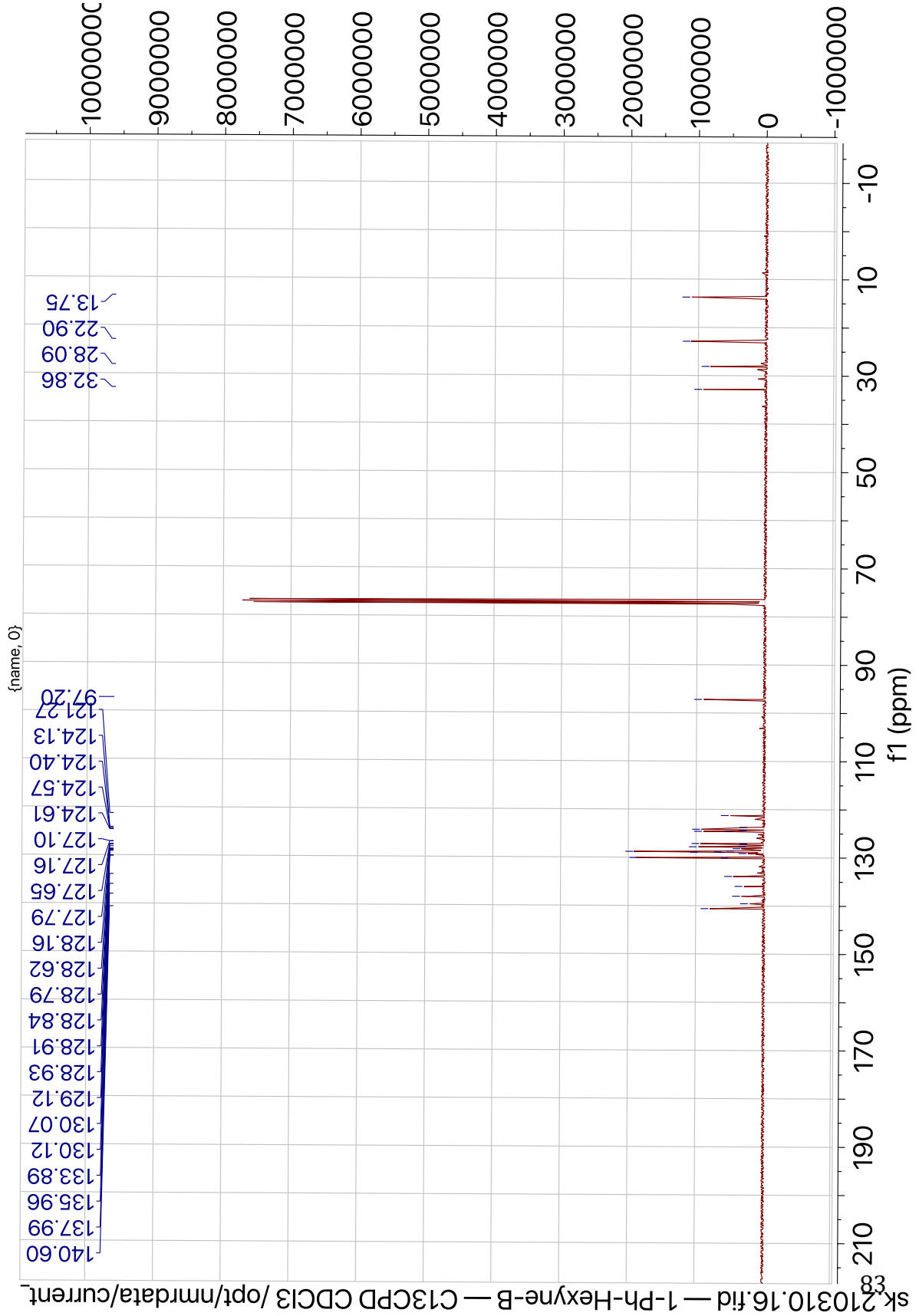
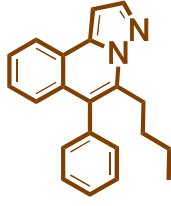
HRMS spectra of 5I'

Compound 5m, ¹H NMR, CDCl₃, 400 MHz



sk.210310.15.fid — 1-Ph-Hexyne-B — PROTON CDCl₃ /opt/nmrdata/current

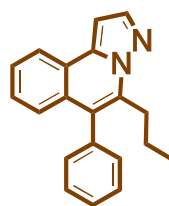
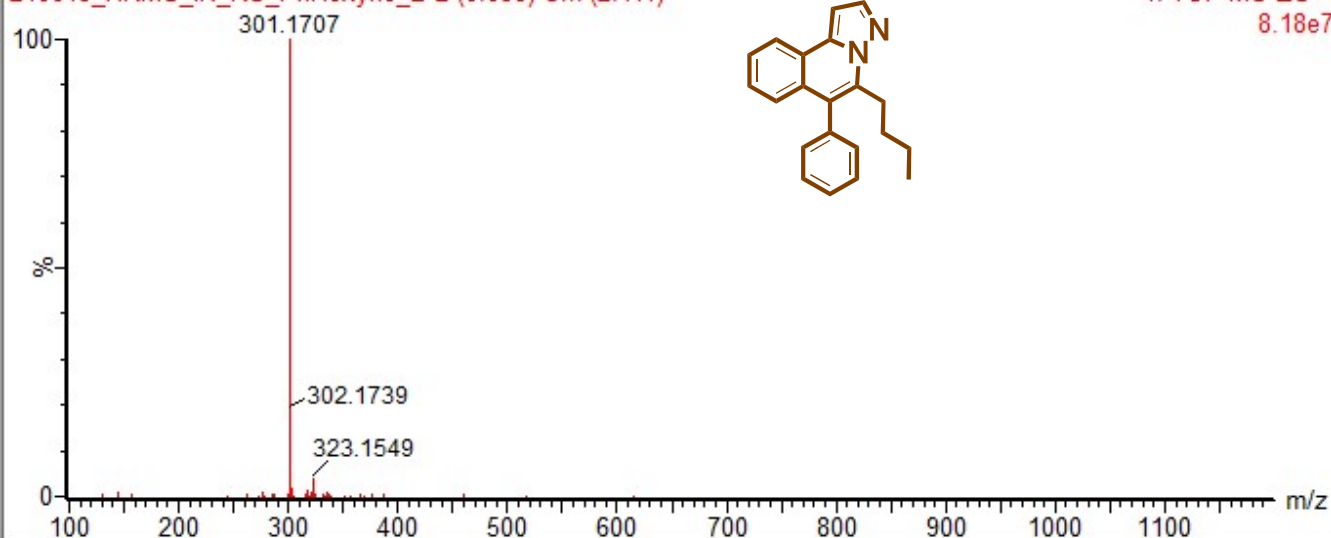
Compound 5m, ^{13}C NMR, CDCl_3 , 101 MHz



DCM-> MeOH (2% H2O + 0.1% FA)

210615_HRMS_IN_KS_PhHexyne_B 2 (0.058) Cm (2:111)

1: TOF MS ES+
8.18e7



Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

187 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-50

H: 0-100

N: 0-5

O: 0-3

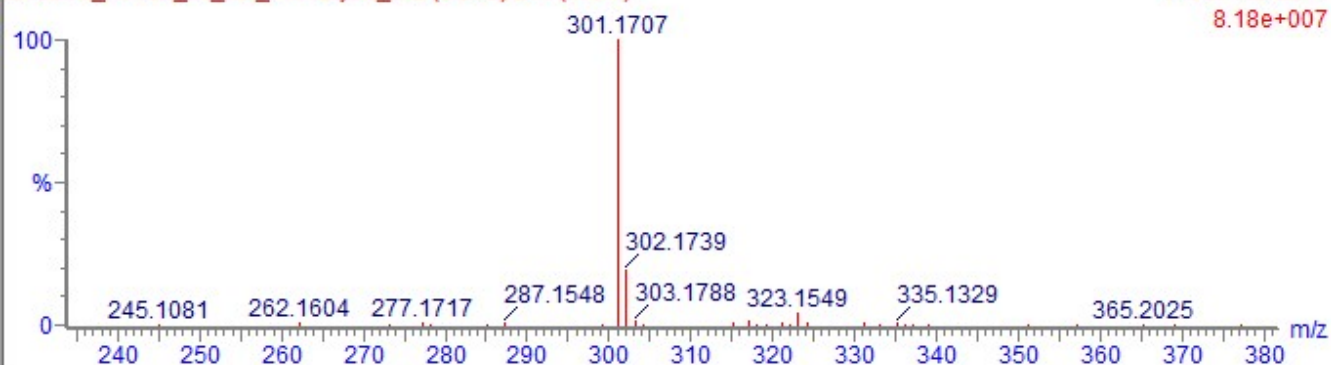
Na: 0-1

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	N	O	Na
301.1707	301.1705	0.2	0.7	12.5	C21 H21 N2	43.1	n/a	n/a	21	21	2		

DCM-> MeOH (2% H2O + 0.1% FA)

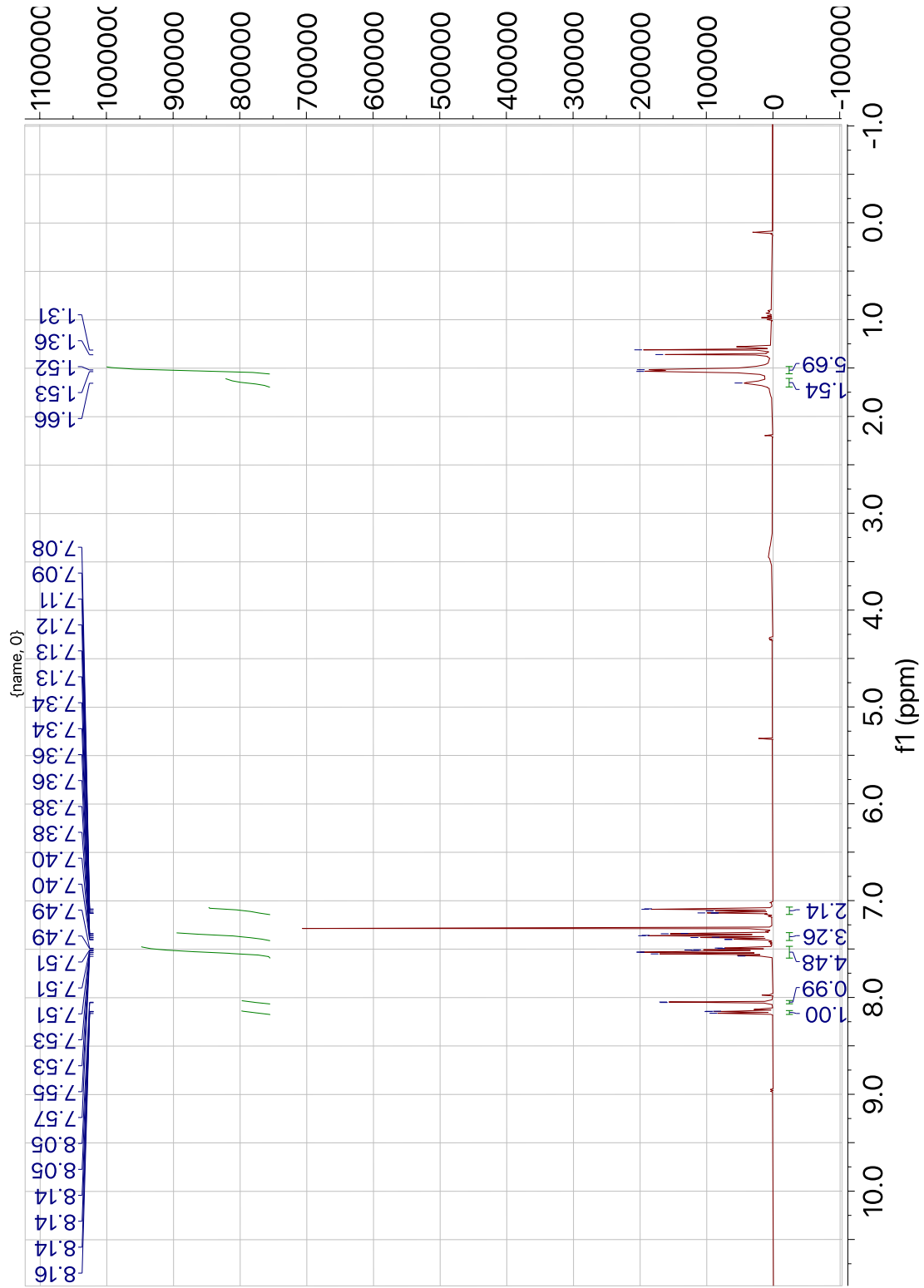
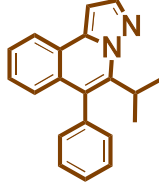
210615_HRMS_IN_KS_PhHexyne_B 2 (0.058) Cm (2:111)

1: TOF MS ES+
8.18e+007



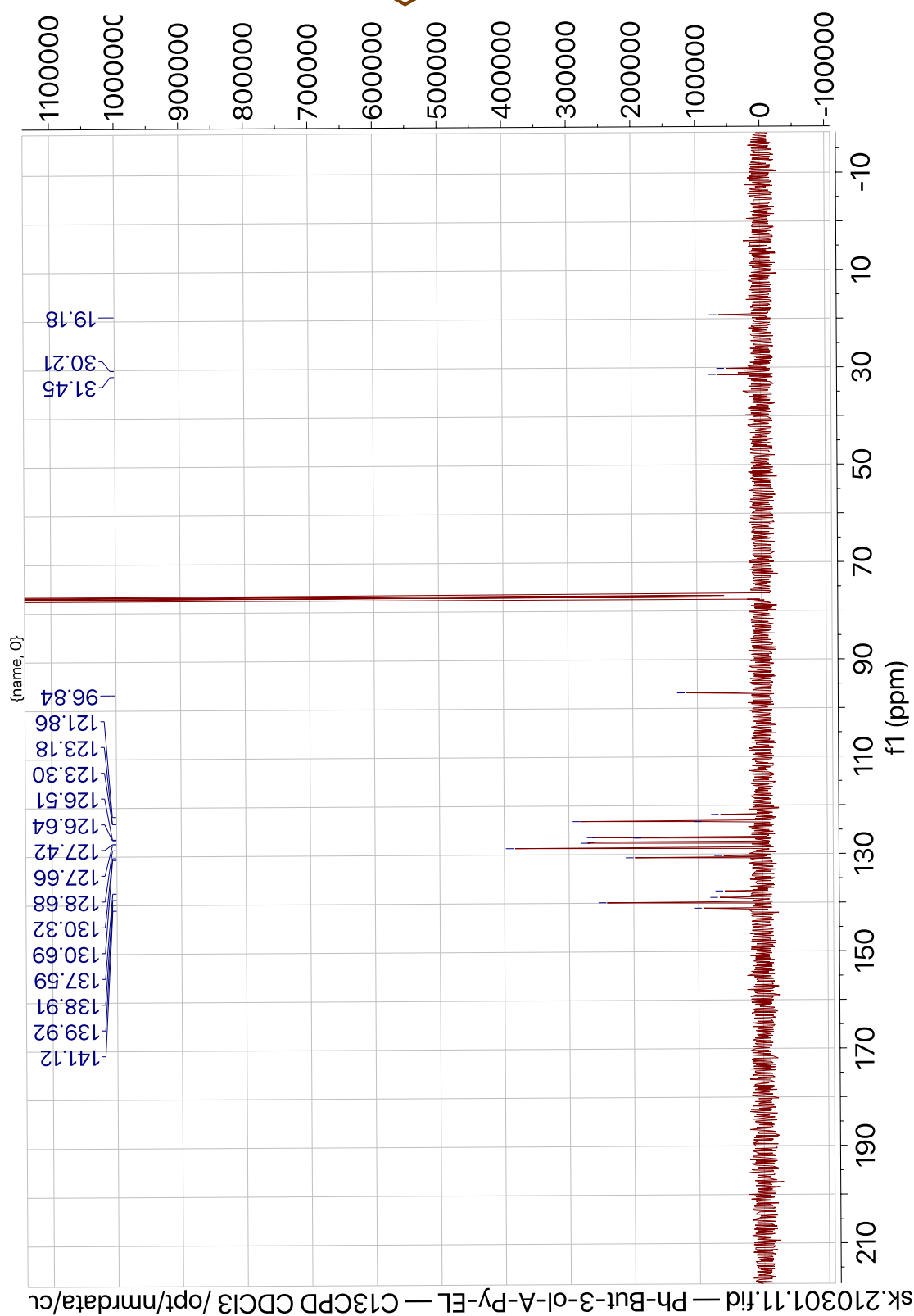
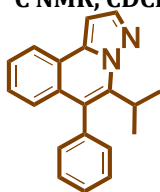
HRMS spectra of **5m**

Compound 5n, ¹H NMR, CDCl₃, 400 MHz



sk.210301.10.fid - Ph-But-3-ol-A-Py-EL - CMC_PROTON CDCl3 /opt/nmr

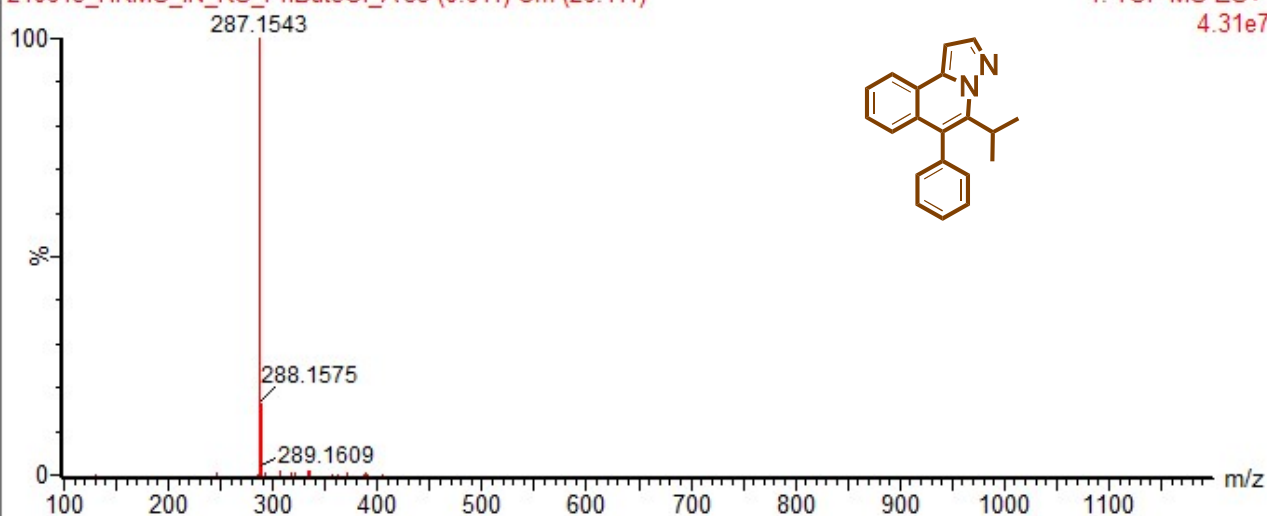
Compound 5n, ¹³C NMR, CDCl₃, 101 MHz



DCM-> MeOH (2% H2O + 0.1% FA)

210615_HRMS_IN_KS_PhBut3OI_A 33 (0.611) Cm (26:111)

1: TOF MS ES+
4.31e7



Single Mass Analysis

Tolerance = 2.0 mDa / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

179 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-50

H: 0-100

N: 0-5

O: 0-3

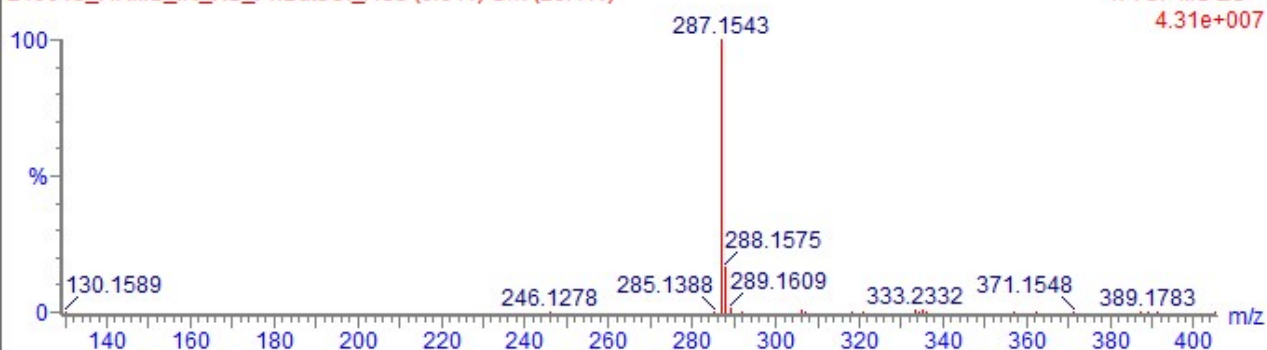
Na: 0-1

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i..	Fit Conf %	C	H	N	O	Na
287.1543	287.1548	-0.5	-1.7	12.5	C20 H19 N2	30...	79.55	20	19	2		
	287.1524	1.9	6.6	9.5	C18 H20 N2 Na	31...	20.45	18	20	2		1

DCM-> MeOH (2% H2O + 0.1% FA)

210615_HRMS_IN_KS_PhBut3OI_A 33 (0.611) Cm (26:111)

1: TOF MS ES+
4.31e+007



HRMS spectra of 5n

References:

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