Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting information

Contents

Synthesis of 2-Anilinopyridyl-Triazole Conjugates as Antimitotic Agents

Ahmed Kamal[*],[a,b,c,d] A.V. Subba Rao,[a,b] M.V.P.S Vishnuvardhan,[a] T. Srinivas Reddy,[c] Konderu Swapna,[d] Chandrakant Bagul,[d] N.V. Subba Reddy[a] and Vunnam srinivasulu[a]

[a]*Medicinal Chemistry and Pharmacology,* [b]*Academy of Scientific and Innovative Research,*

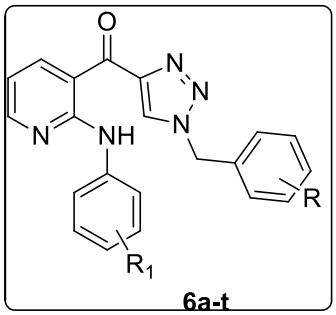
[c]*IICT-RMIT Research Centre, CSIR-Indian Institute of Chemical Technology, Hyderabad, 500007, India.*

[d]*Department of Medicinal Chemistry, National Institute of Pharmaceutical Education & Research (NIPER), Hyderabad 500 037, India.*

*****Correspondence: ahmedkamal@iict.res.in; Phone: (+) 91-40-27193157; Fax: (+) 91-40-27193189

MATERIAL AND METHODS

All chemicals and reagents were obtained from Aldrich (Sigma– Aldrich), St. Louis, MO, USA), Lancaster (Alfa Aesar, Johnson Matthey Company, Ward Hill, MA, USA), or Spectrochem Pvt. Ltd (Mumbai, India) and were used without further purification. Reactions were monitored by TLC performed on silica gel glass plate containing 60 GF-254, and visualization was achieved by UV light or iodine indicator. Column chromatography was performed with Merck 60–120 mesh silica gel. ^{1}H and ^{13}C NMR spectra were determined in $CDCl_3$ by using Varian and Avance instruments. Chemical shifts are expressed in parts per million (δ in ppm) downfield from internal TMS and coupling constants are expressed in Hz. ^{1}H NMR spectroscopic data are reported in the following order: multiplicity (s, singlet; br s, broad singlet; d, doublet; dd, doublet of doublets; t, triplet; m, multiplet), coupling constants in Hz, number of protons. ESI mass spectra were recorded on a Micro mass Quattro LC using ESI+ software with capillary voltage 3.98 kV and an ESI mode positive ion trap detector. Melting points were determined with an Electro thermal melting point apparatus, and are uncorrected.

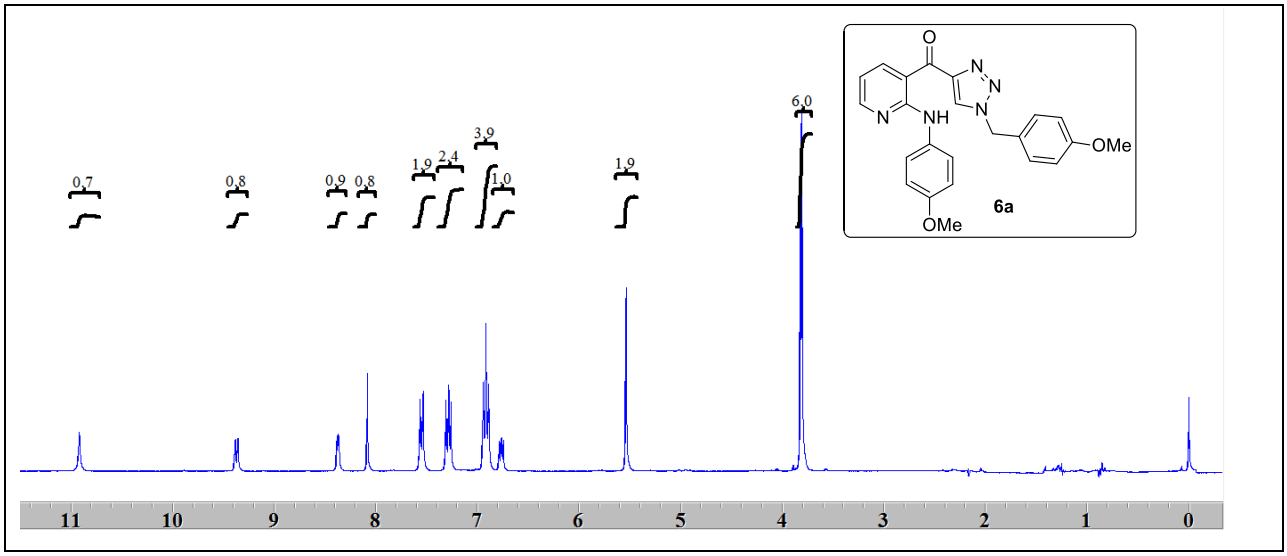


6a-t

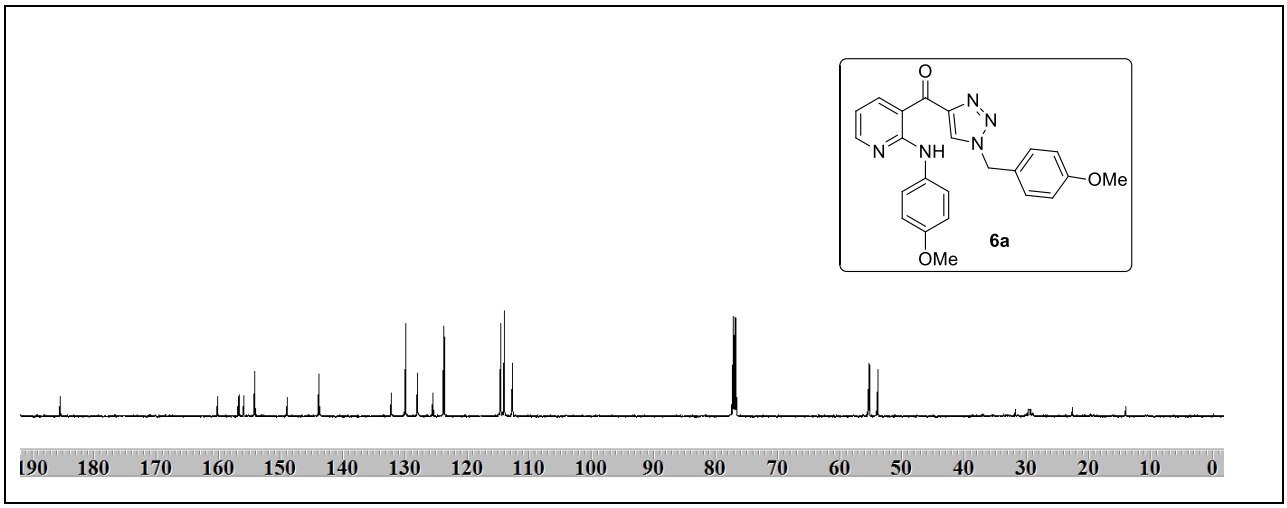
S. No	Compound	R_1	R
1	**6a**	4-OMe	4-OMe
2	**6b**	4-OMe	3,4-diOMe
3	**6c**	4-OMe	3,5-diOMe
4	**6d**	4-OMe	3-OPh
5	**6e**	4-OMe	4-F
6	**6f**	3,4-diOMe	4-OMe
7	**6g**	3,4-diOMe	3,4-diOMe
8	**6h**	3,4-diOMe	3,5-diOMe
9	**6i**	3,4-diOMe	3-OPh
10	**6j**	3,4-diOMe	4-F
11	**6k**	3,4,5-triOMe	4-OMe
12	**6l**	3,4,5-triOMe	3,4-diOMe
13	**6m**	3,4,5-triOMe	3,5-diOMe
14	**6n**	3,4,5-triOMe	3-OPh
15	**6o**	3,4,5-triOMe	4-F
16	**6p**	4-F	4-OMe
17	**6q**	4-F	3,4-diOMe
18	**6r**	4-F	3,5-diOMe
19	**6s**	4-F	3-OPh
20	**6t**	4-F	4-F

General method for synthesis of (1-benzyl-1H-1,2,3-triazol-4-yl)(2-(phenylamino)pyridin-3-yl)methanone (6a-t)

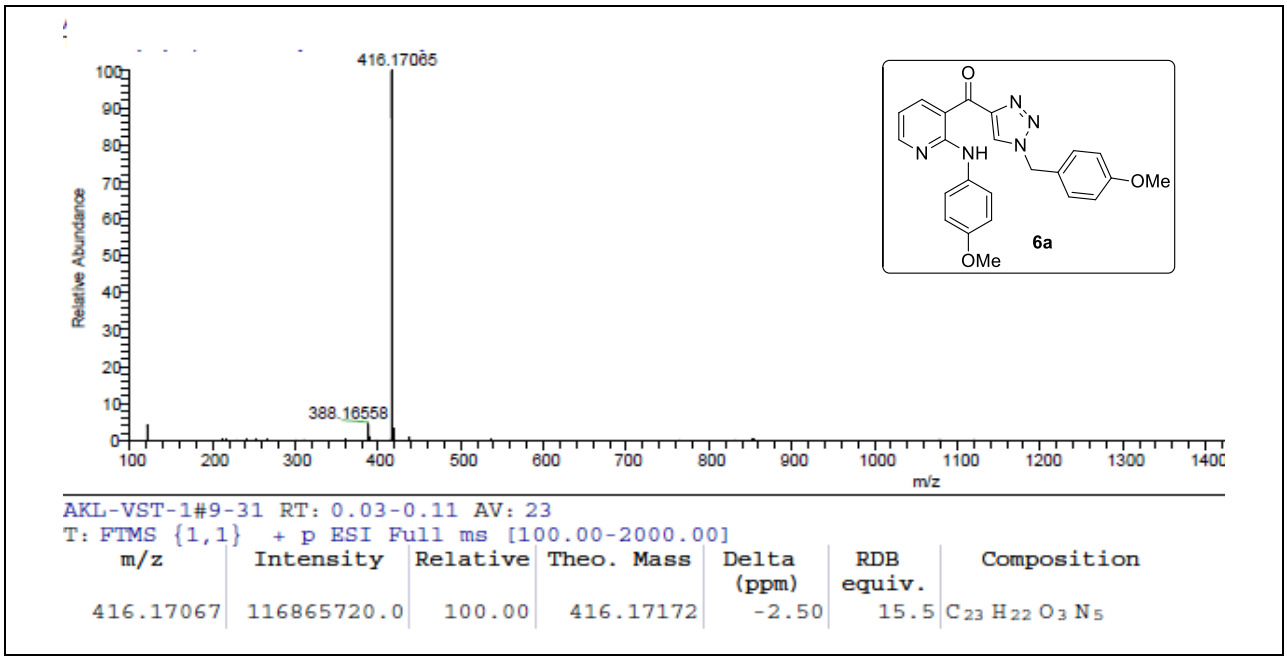
To a solution of substituted ethynyl ketones (**14a-d**) (0.59 mmol) and substituted benzyl azides (**17a-e**) (0.65 mmol) in 2:1 mixture of water and tert-butyl alcohol, sodium ascorbate (0.06 mmol) and copper (II) sulfate (0.03 mmol) were added sequentially. The reaction was stirred at room temperature for overnight, TLC analysis indicated completion of reaction. The solvent was concentrated under vacuum and extracted with EtOAc to give crude product. The crude products were purified by column chromatography to afford pure products **(6a-t)** as yellow solids.



^{1}H NMR SPECTRUM OF COMPOUND 6a

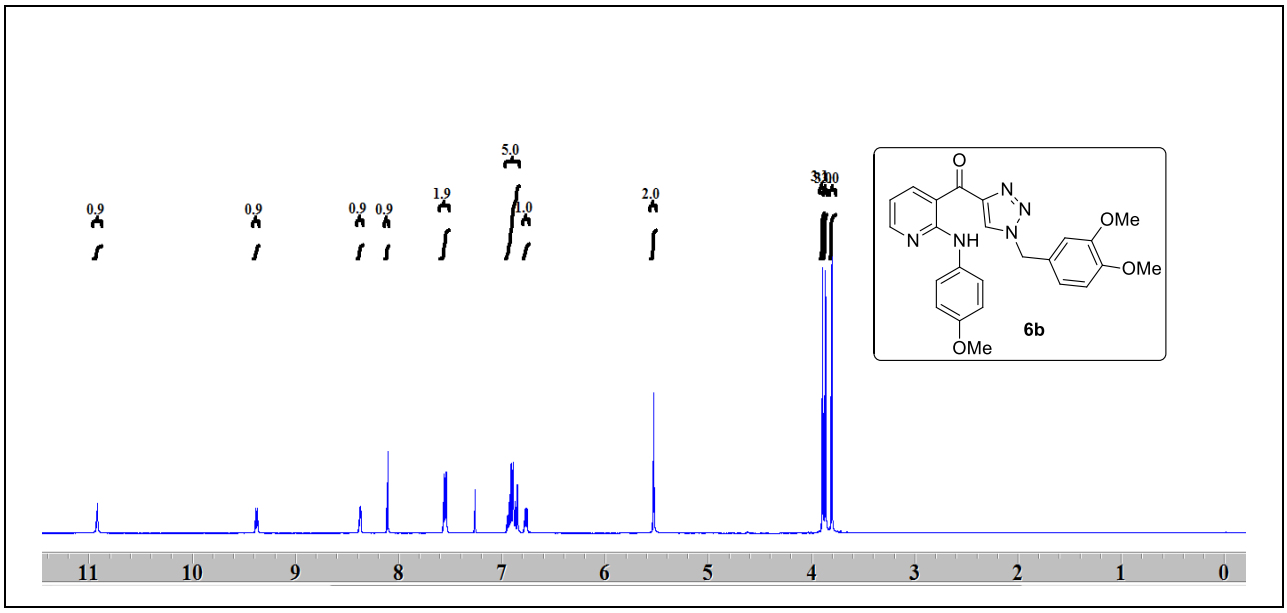


^{13}C NMR SPECTRUM OF COMPOUND 6a

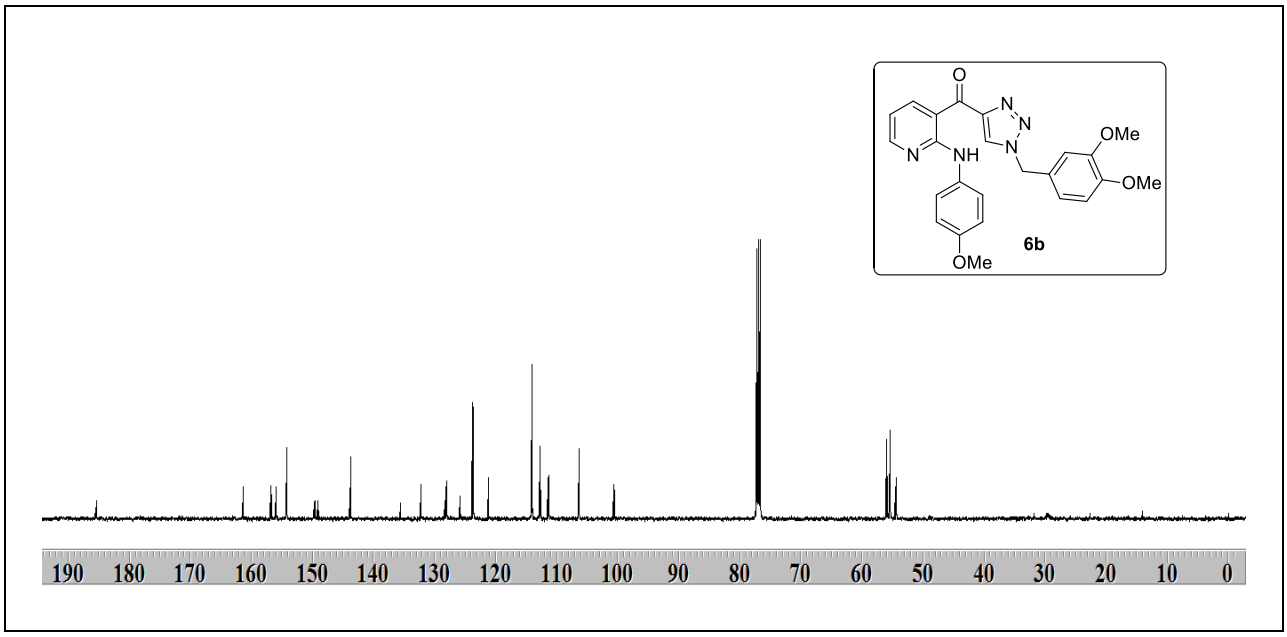


m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
416.17067	116865720.0	100.00	416.17172	-2.50	15.5	$C_{23}H_{22}O_3N_5$

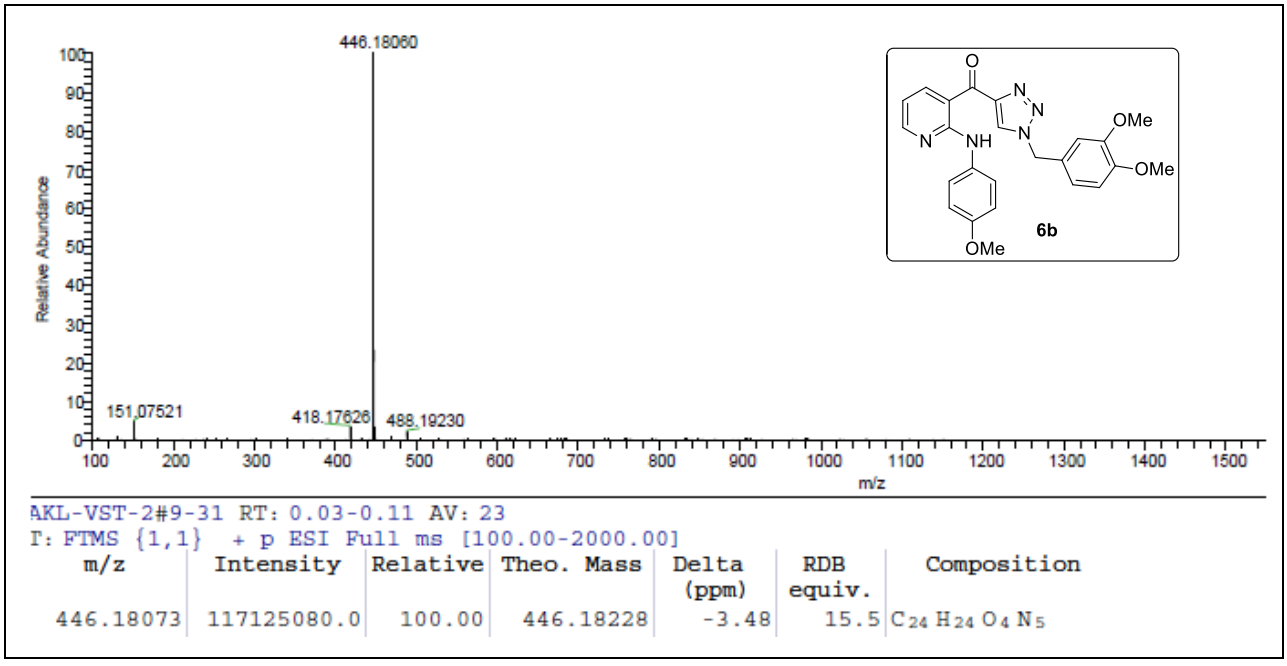
HIGH RESOLUTION MASS SPECTRUM OF COMPOUND 6a



^{1}H NMR SPECTRUM OF COMPOUND 6b



^{13}C NMR SPECTRUM OF COMPOUND 6b



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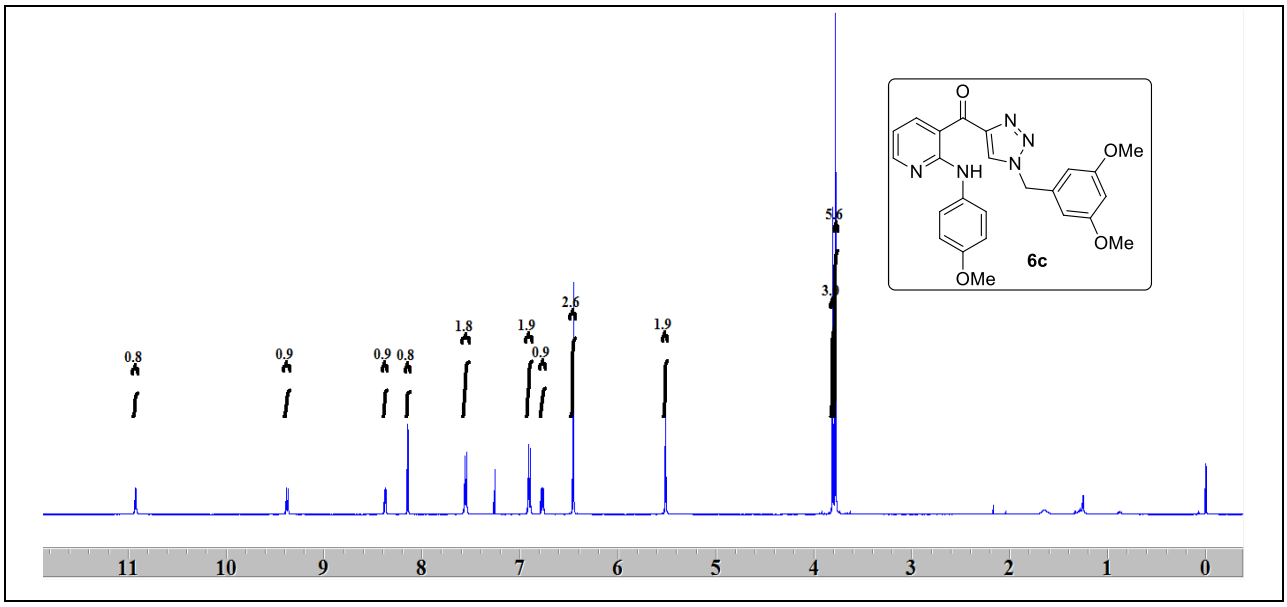
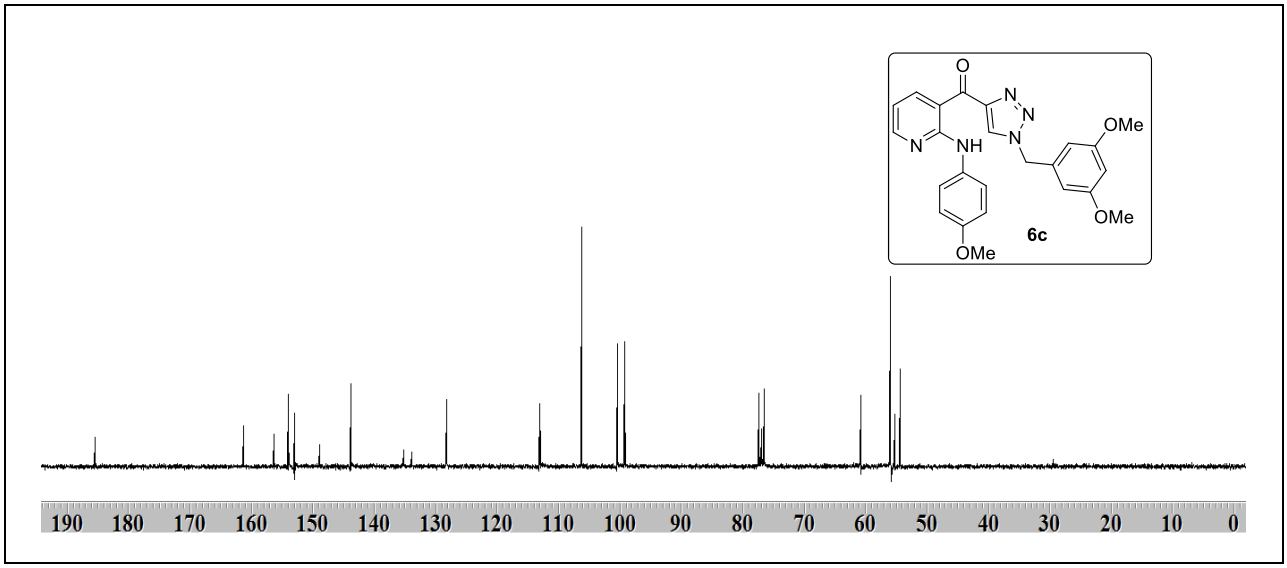
m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
446.18073	117125080.0	100.00	446.18228	-3.48	15.5	$C_{24}H_{24}O_4N_5$

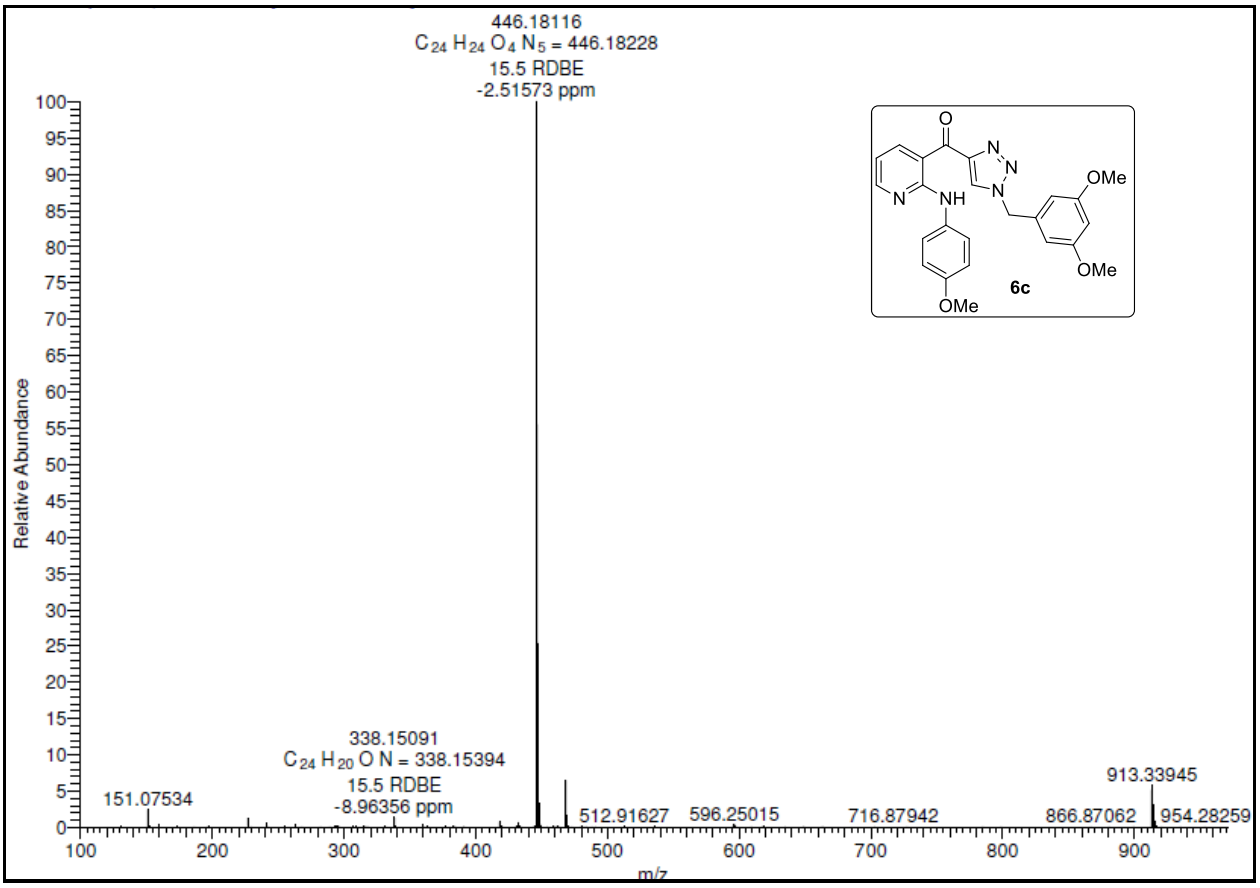
HIGH RESOLUTION MASS SPECTRUM OF COMPOUND 6b



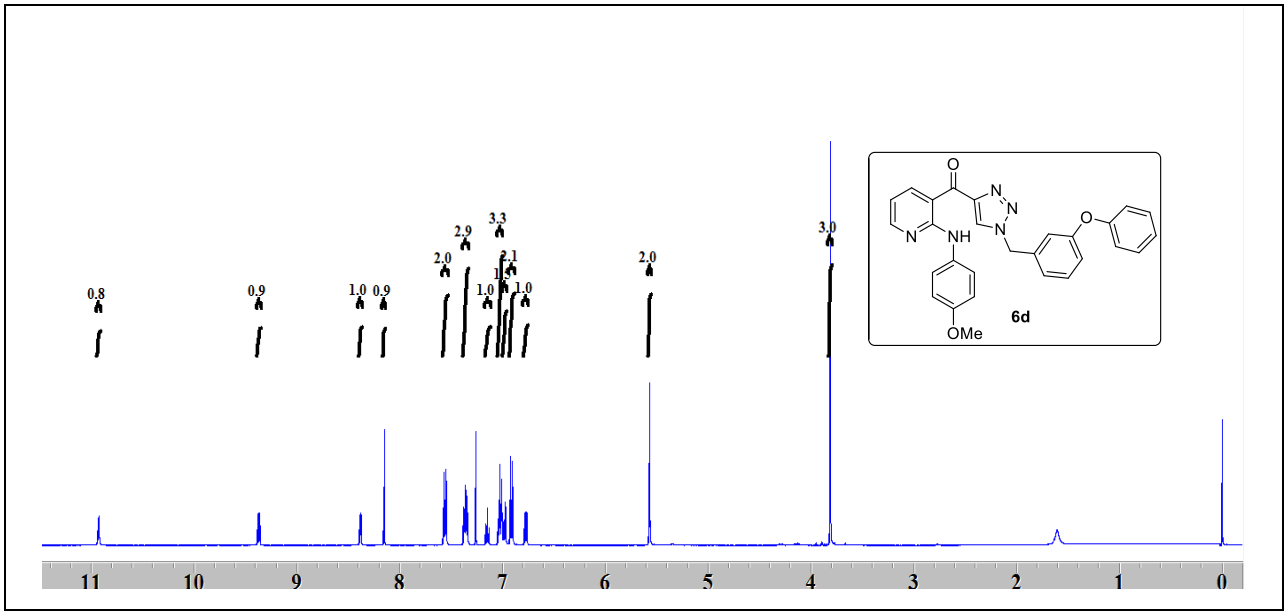
^{1}H NMR SPECTRUM OF COMPOUND 6c



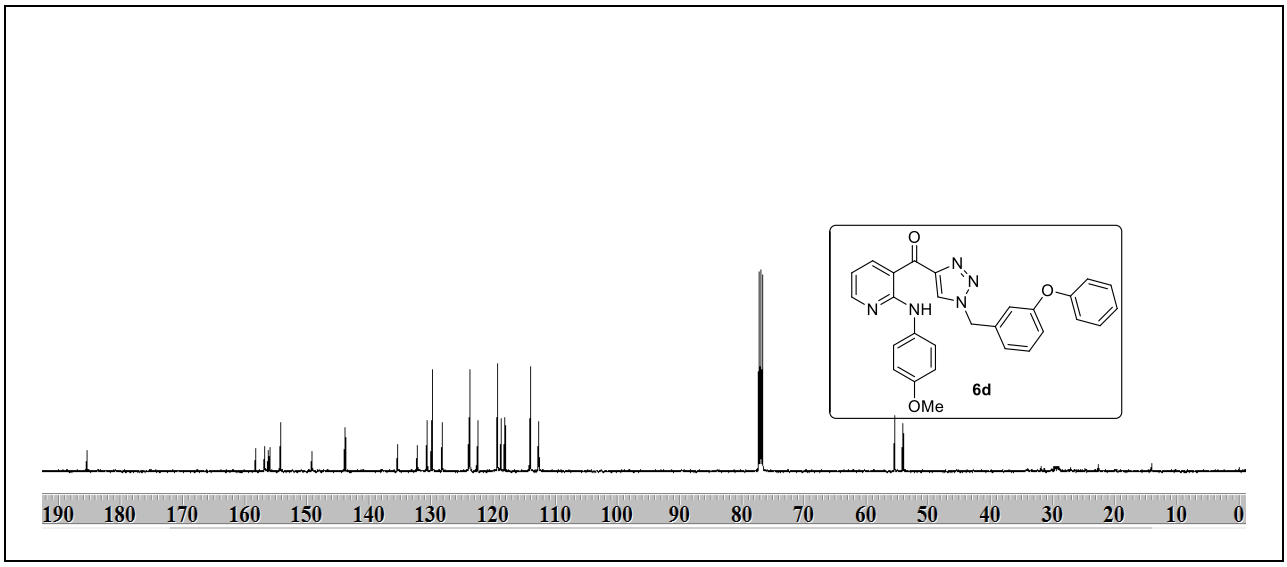
^{13}C NMR SPECTRUM OF COMPOUND 6c



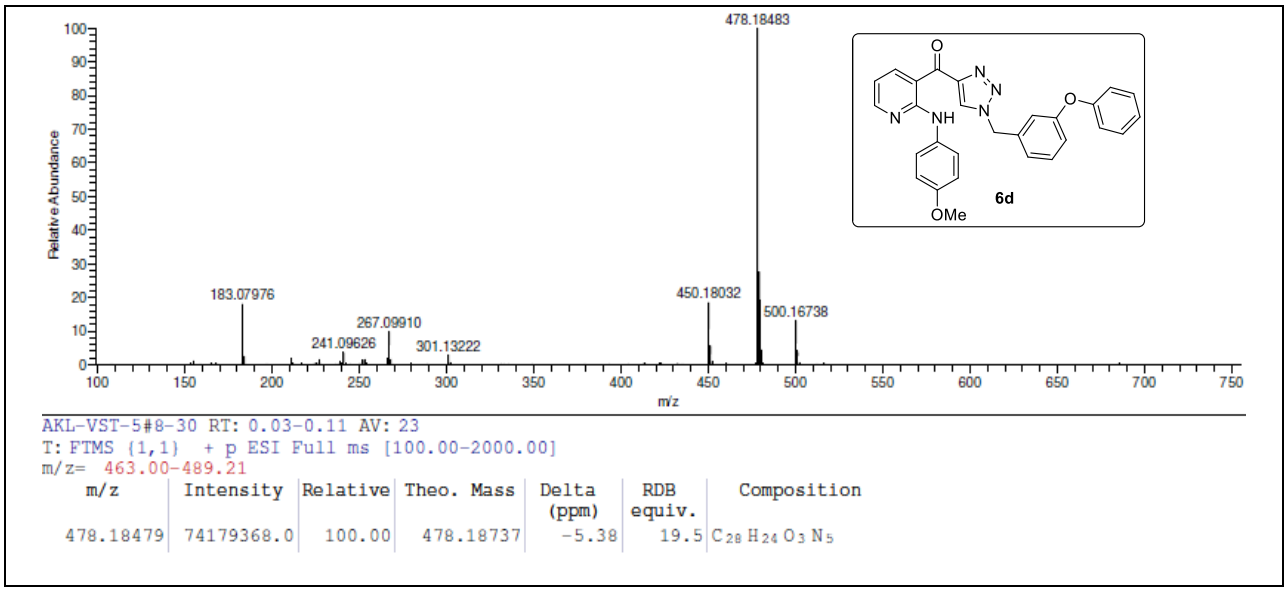
HIGH RESOLUTION MASS SPECTRUM OF COMPOUND 6c



^{1}H NMR SPECTRUM OF COMPOUND 6d



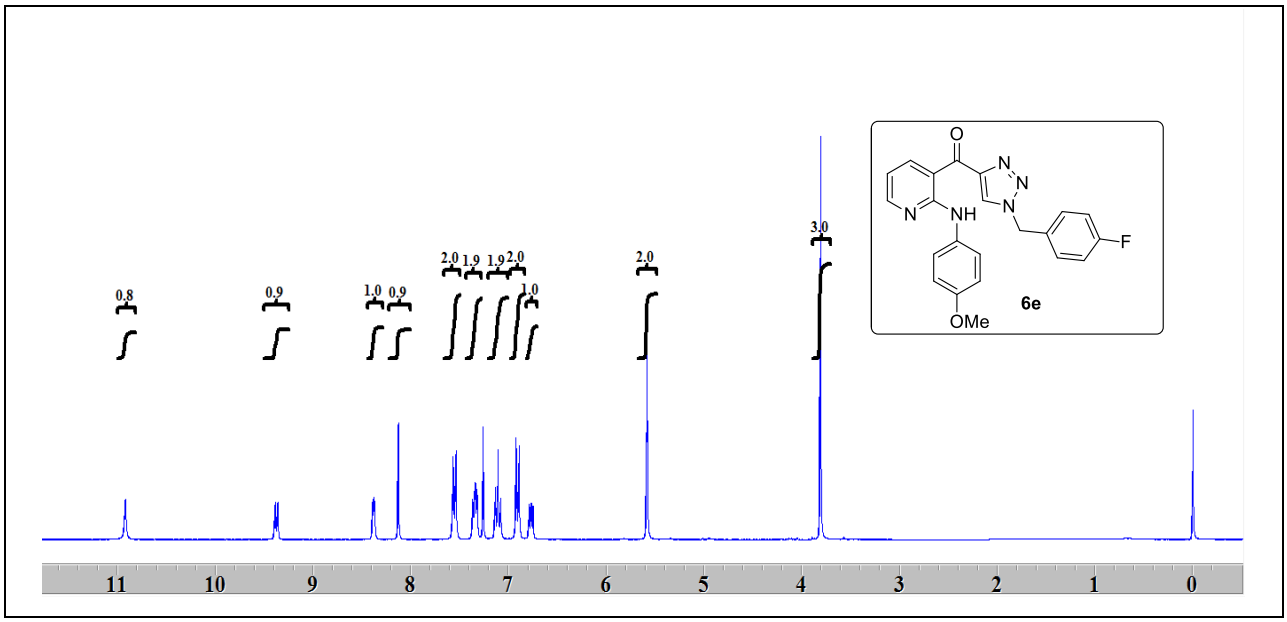
^{13}C NMR SPECTRUM OF COMPOUND 6d



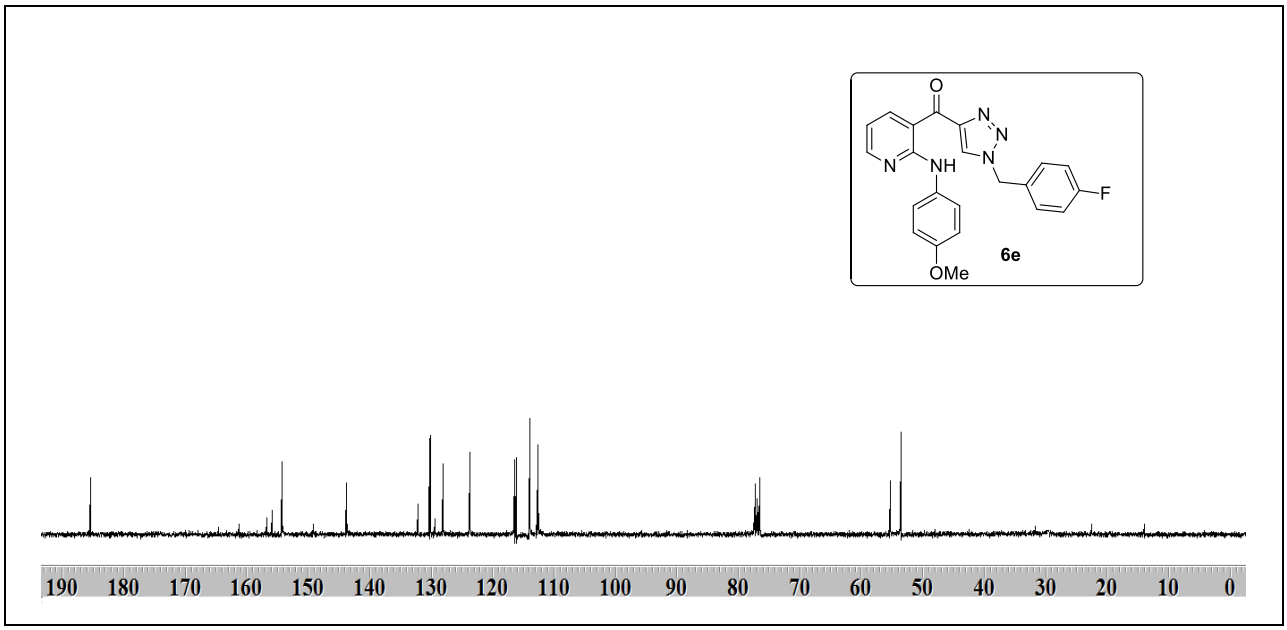
AKL-VST-5#8-30 RT: 0.03-0.11 AV: 23
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]
m/z= 463.00-489.21

m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
478.18479	74179368.0	100.00	478.18737	-5.38	19.5	$C_{28}H_{24}O_3N_5$

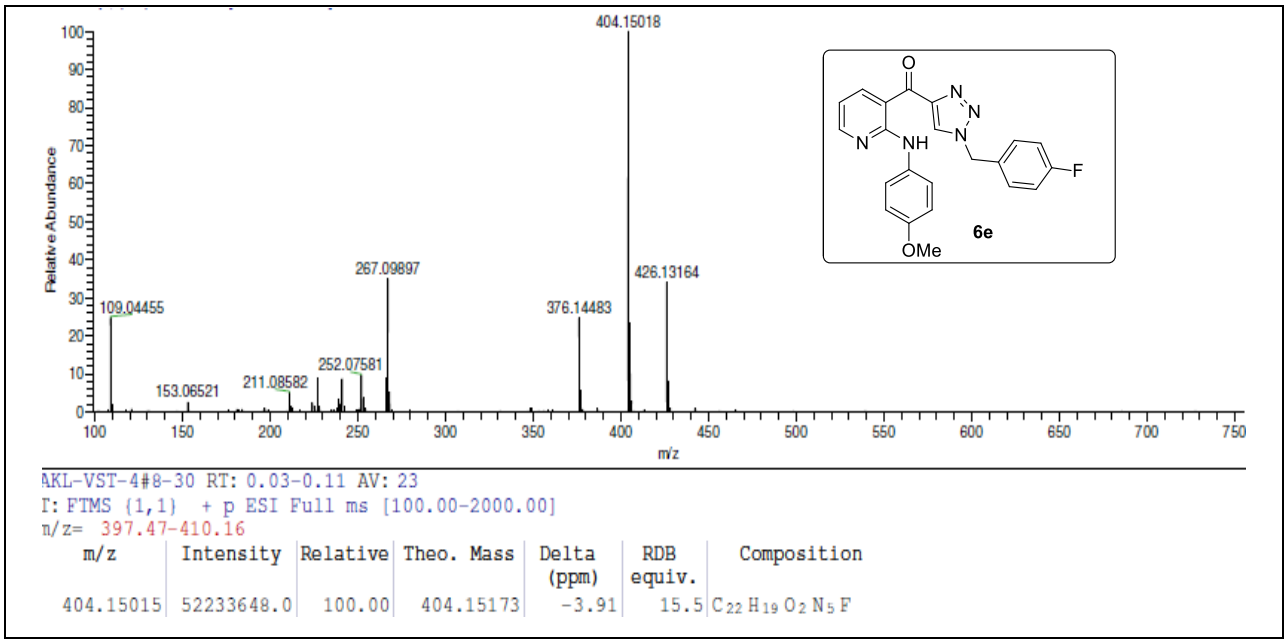
HIGH RESOLUTION MASS SPECTRUM OF COMPOUND 6d



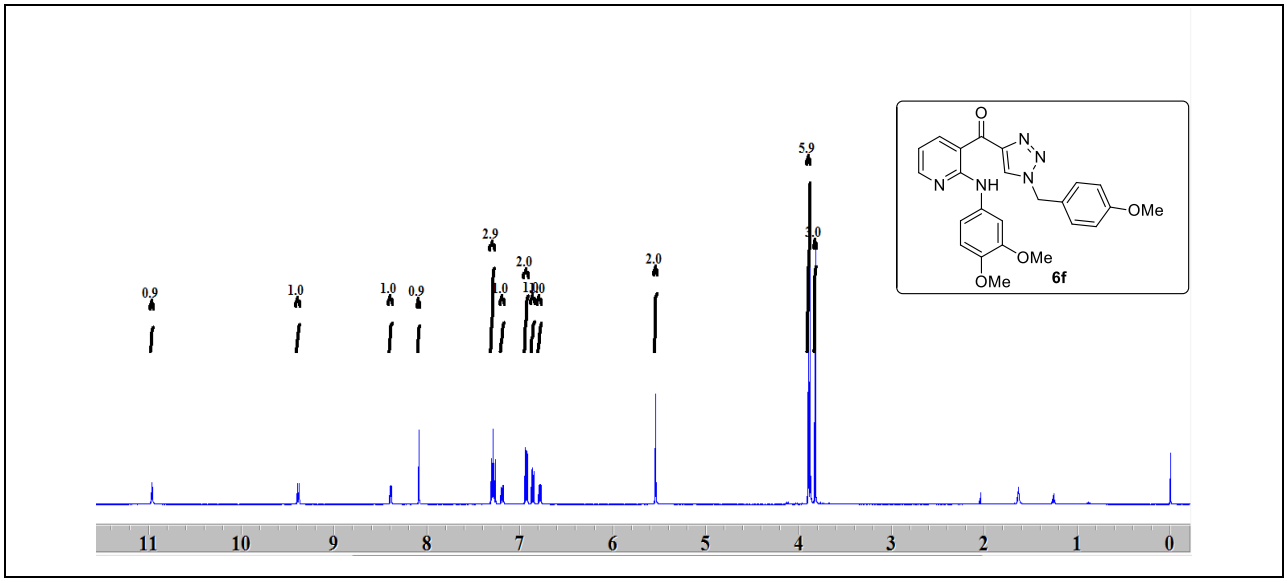
^{1}H NMR SPECTRUM OF COMPOUND 6e



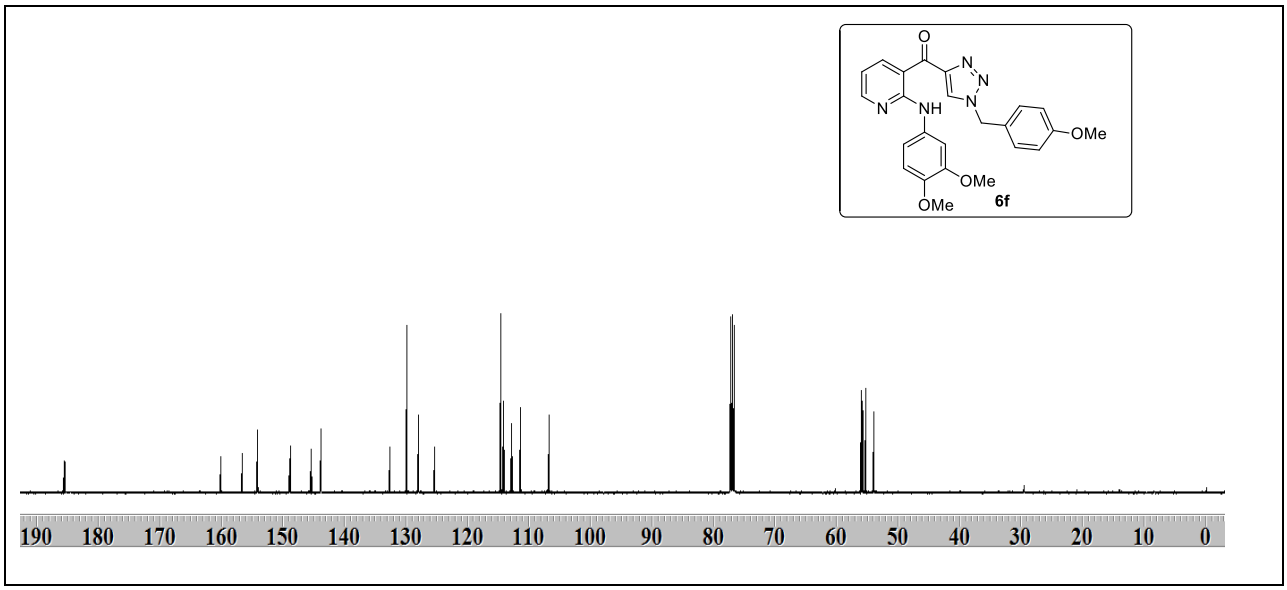
^{13}C NMR SPECTRUM OF COMPOUND 6e



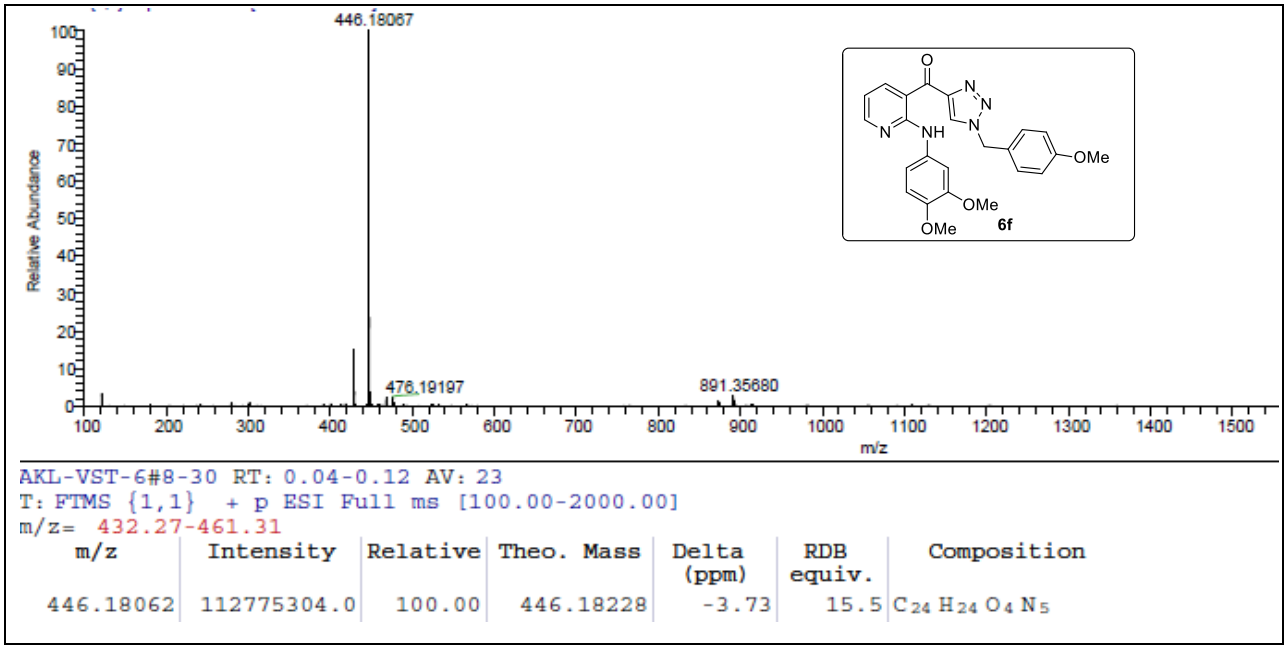
HIGH RESOLUTION MASS SPECTRUM OF COMPOUND 6e



^{1}H NMR SPECTRUM OF COMPOUND 6f



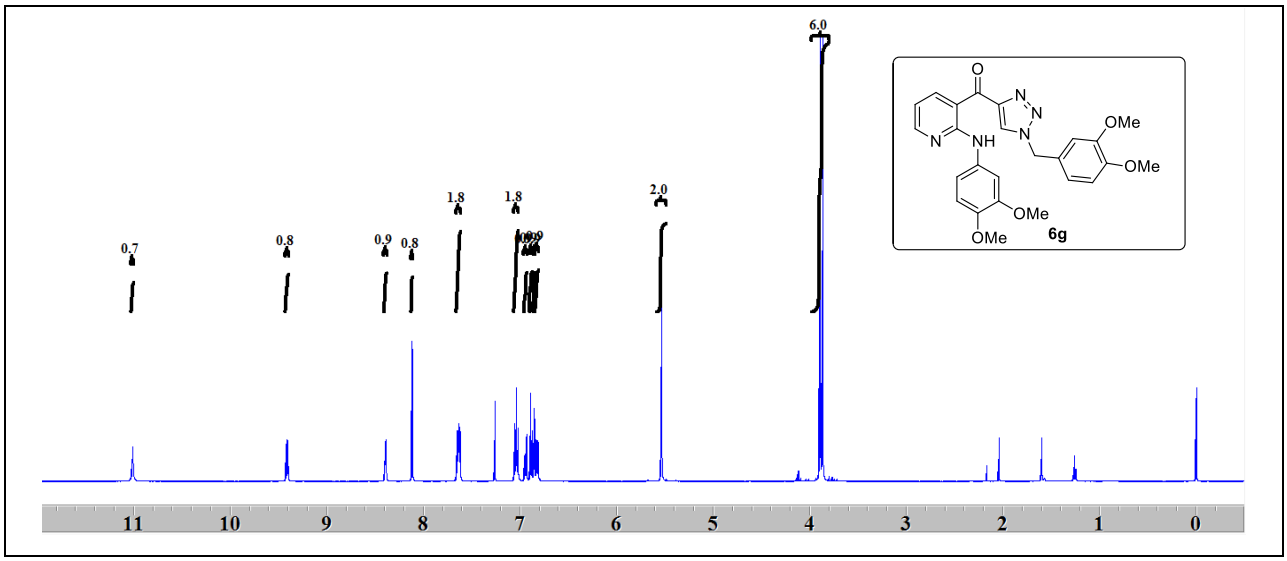
^{13}C NMR SPECTRUM OF COMPOUND 6f



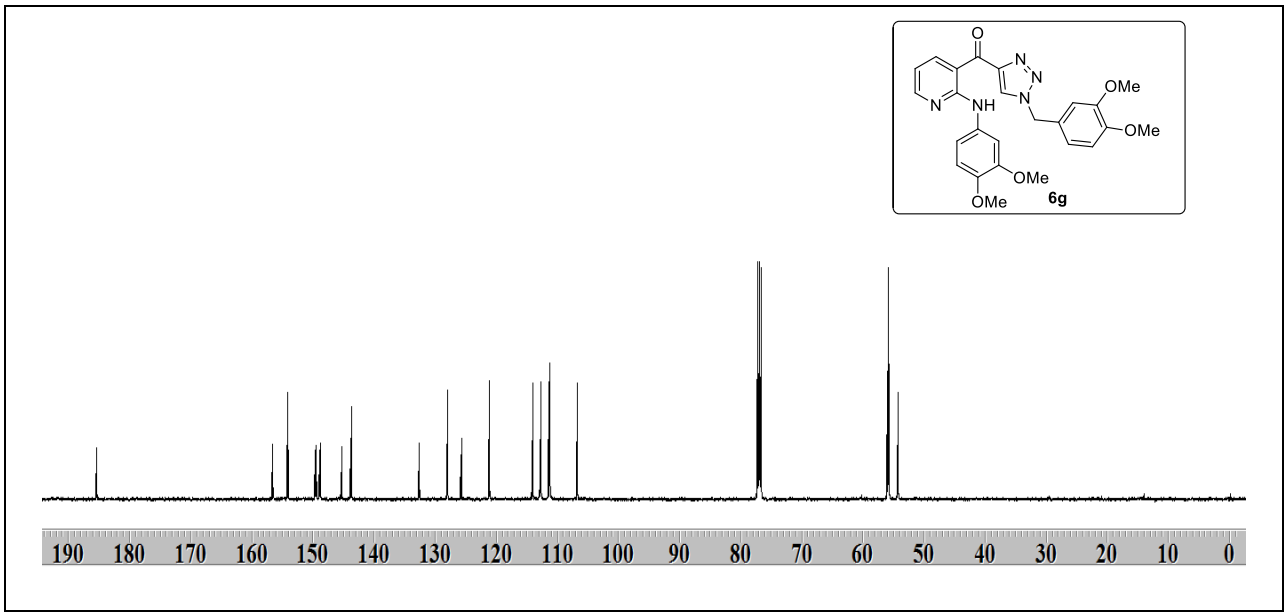
AKL-VST-6#8-30 RT: 0.04-0.12 AV: 23
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]
m/z= 432.27-461.31

m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
446.18062	112775304.0	100.00	446.18228	-3.73	15.5	$C_{24}H_{24}O_4N_5$

HIGH RESOLUTION MASS SPECTRUM OF COMPOUND 6f



^{1}H NMR SPECTRUM OF COMPOUND 6g



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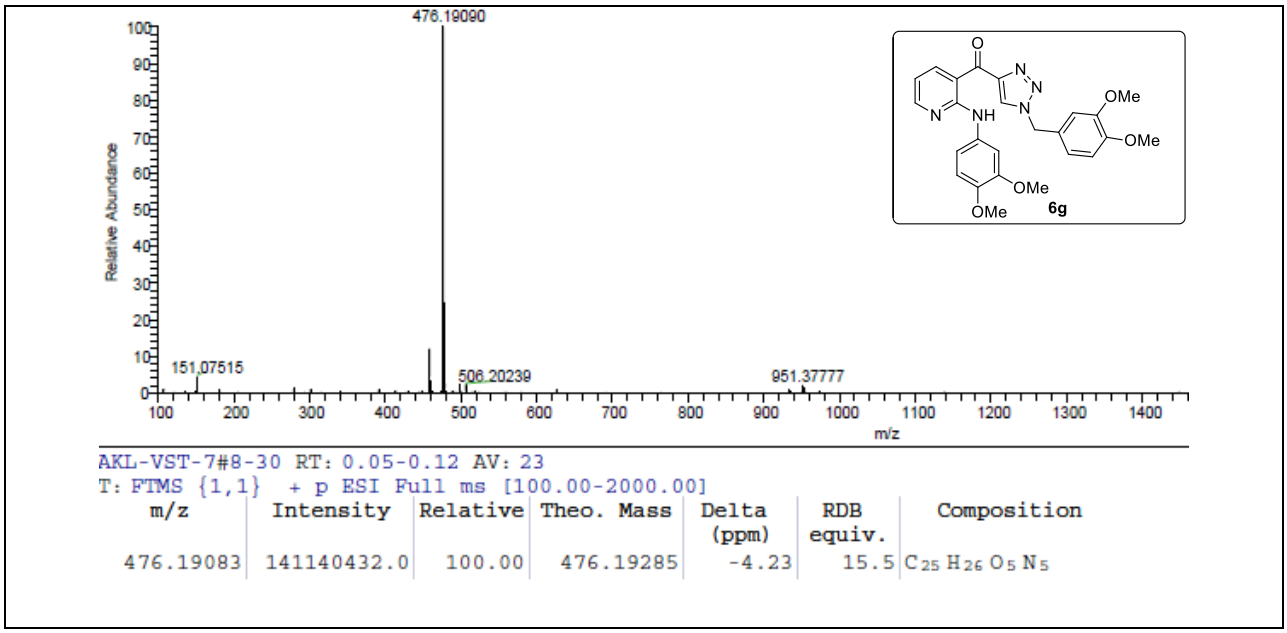
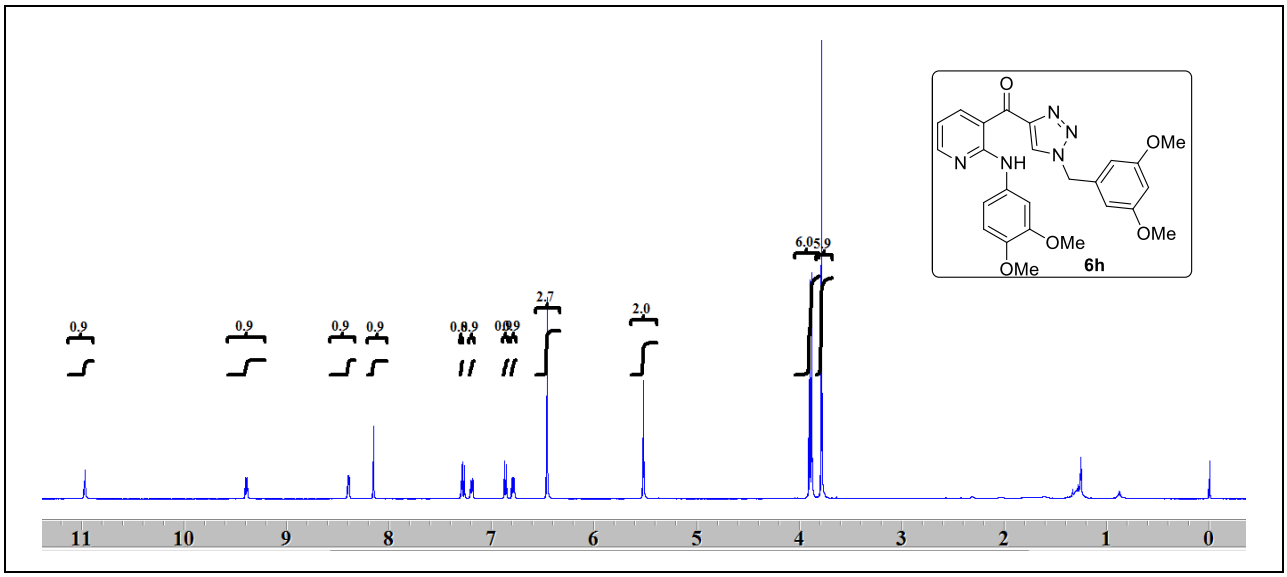
^{13}C NMR SPECTRUM OF COMPOUND 6g



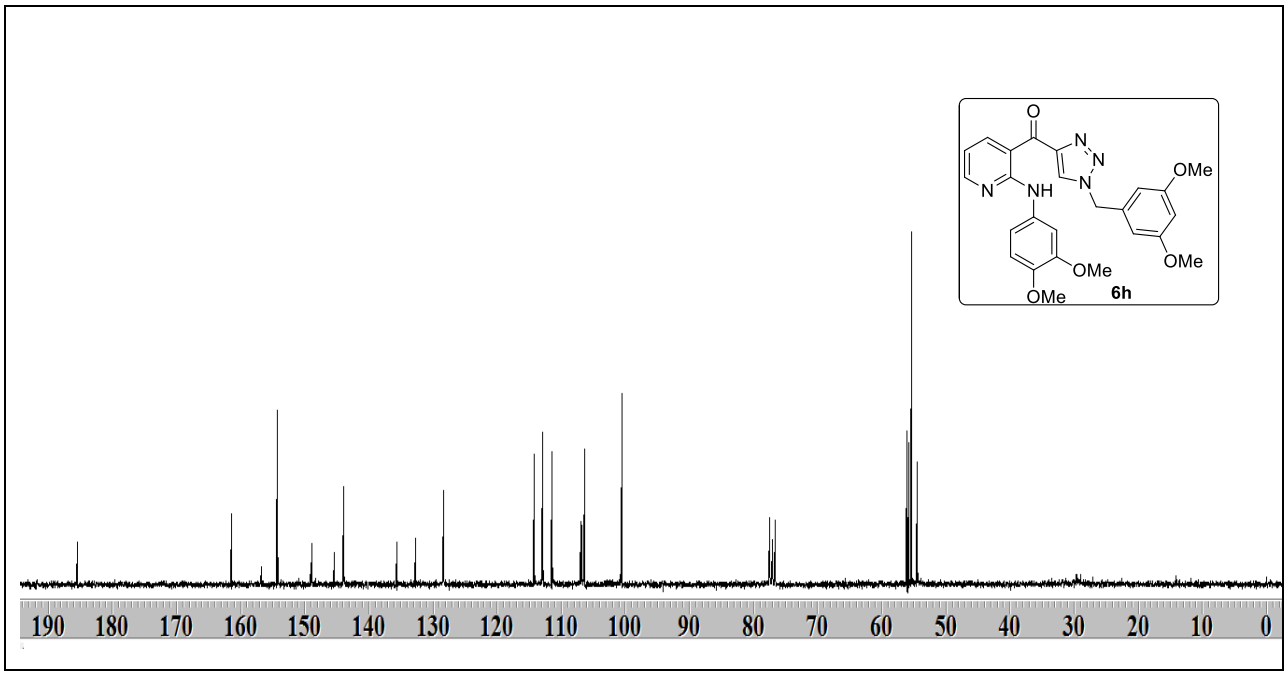
AKL-VST-7#8-30 RT: 0.05-0.12 AV: 23
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]

m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
476.19083	141140432.0	100.00	476.19285	-4.23	15.5	$C_{25}H_{26}O_5N_5$

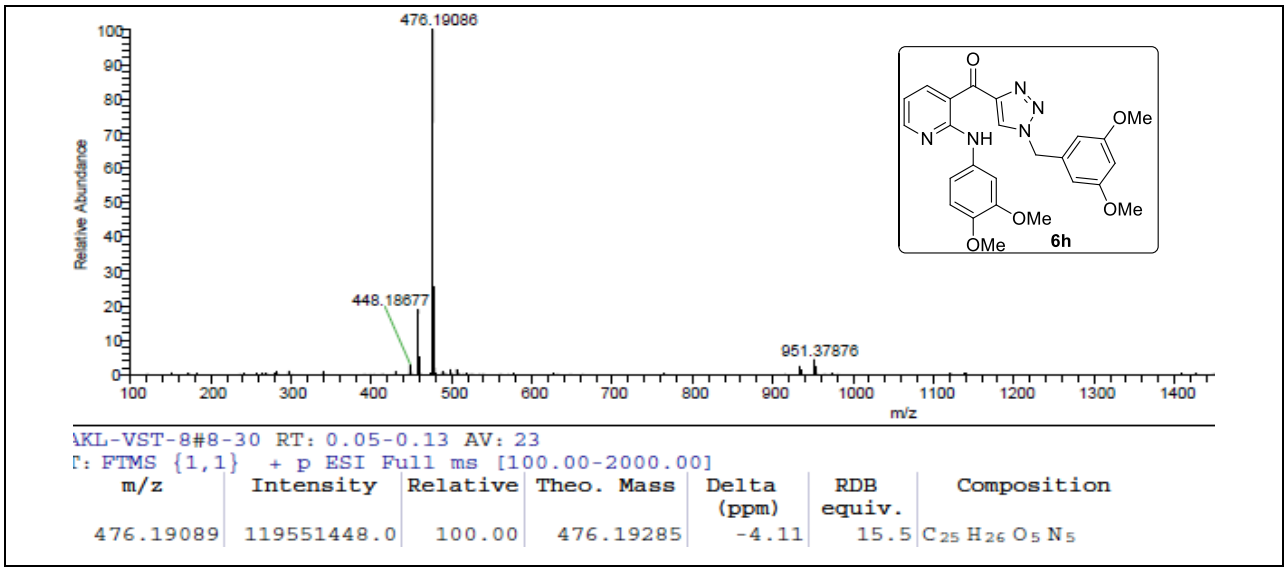
HIGH RESOLUTION MASS SPECTRUM OF COMPOUND 6g



^{1}H NMR SPECTRUM OF COMPOUND 6h

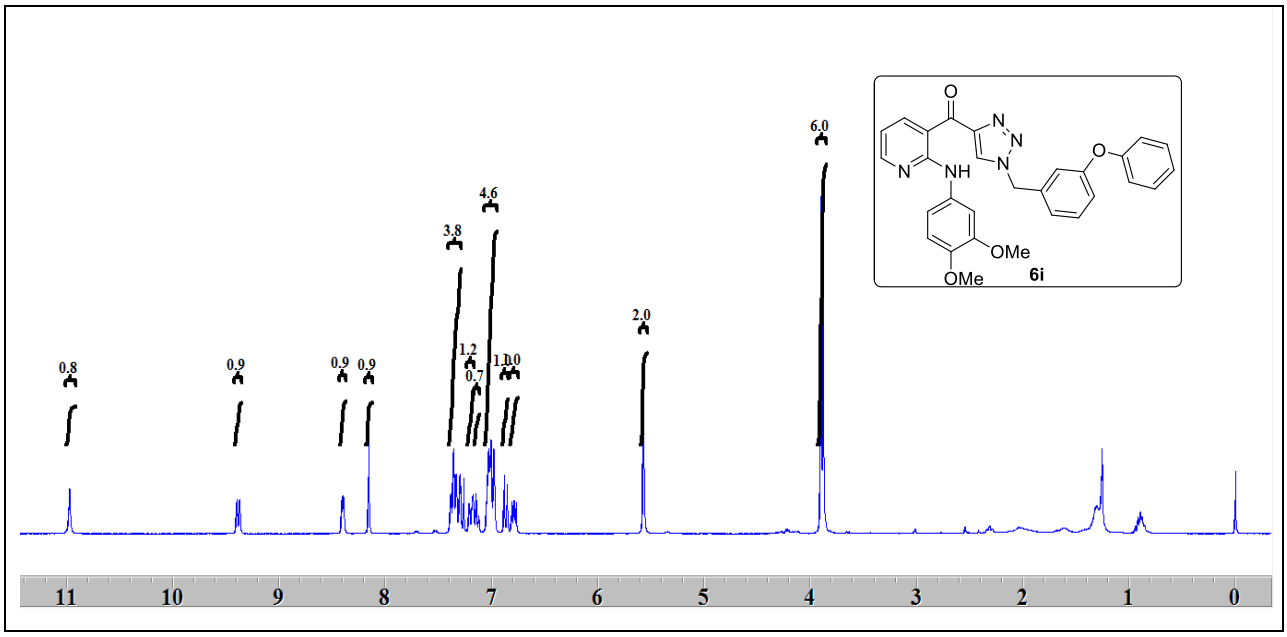


^{13}C NMR SPECTRUM OF COMPOUND 6h

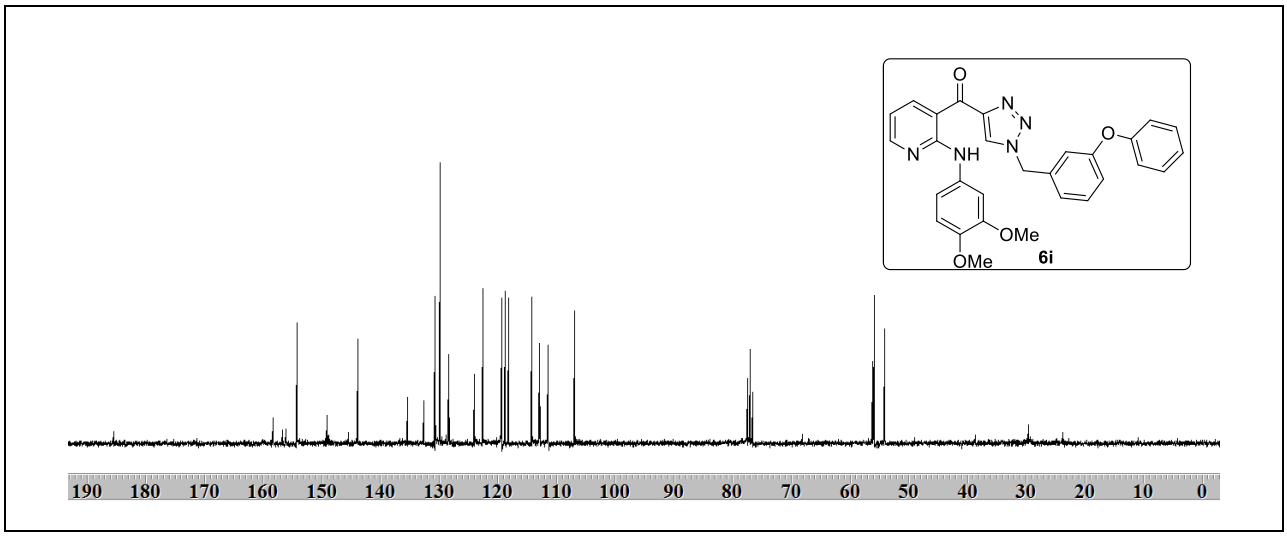


m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
476.19089	119551448.0	100.00	476.19285	-4.11	15.5	$C_{25}H_{26}O_5N_5$

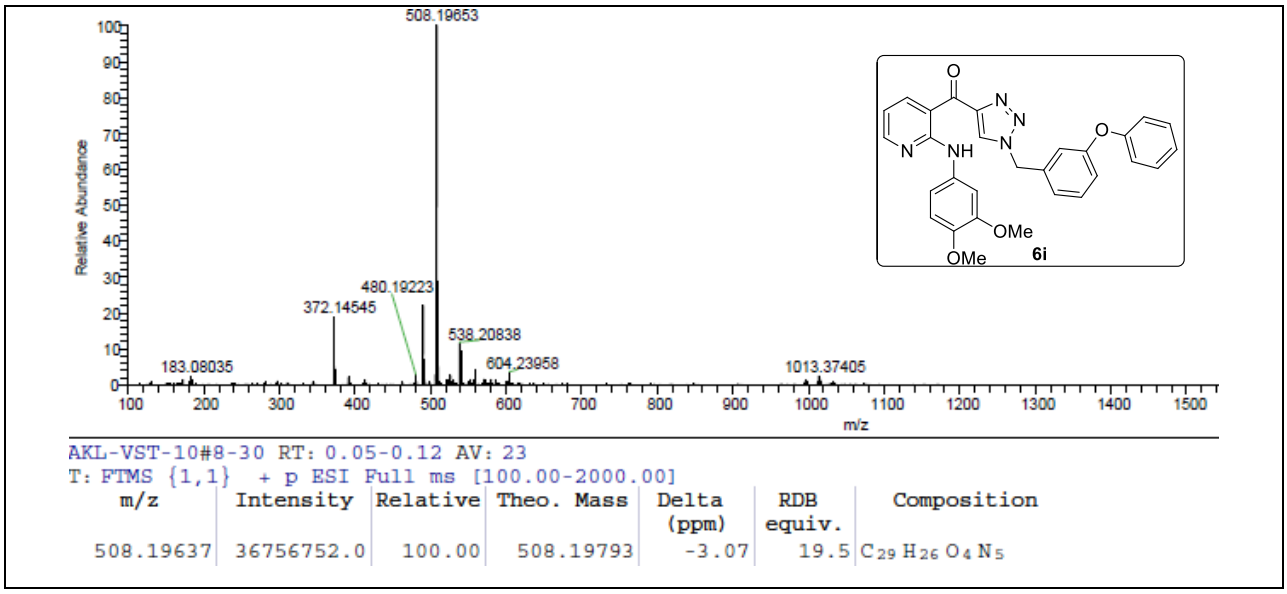
HIGH RESOLUTION MASS SPECTRUM OF COMPOUND 6h



^{1}H NMR SPECTRUM OF COMPOUND 6i



^{13}C NMR SPECTRUM OF COMPOUND 6i



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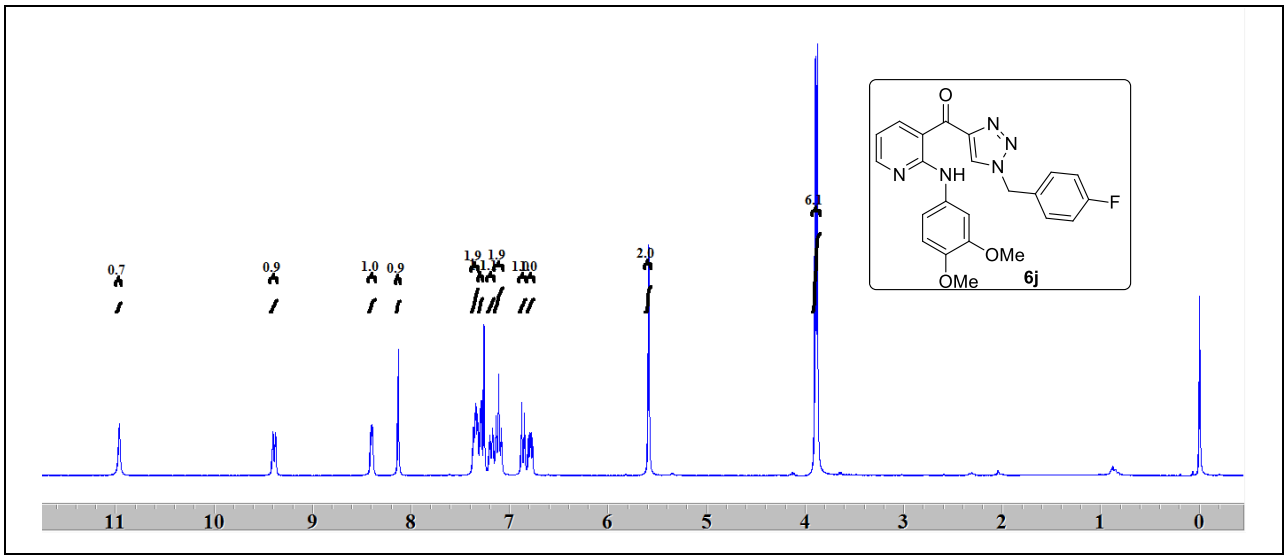
m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
508.19637	36756752.0	100.00	508.19793	-3.07	19.5	$C_{29}H_{26}O_4N_5$

HIGH RESOLUTION MASS SPECTRUM OF COMPOUND 6i



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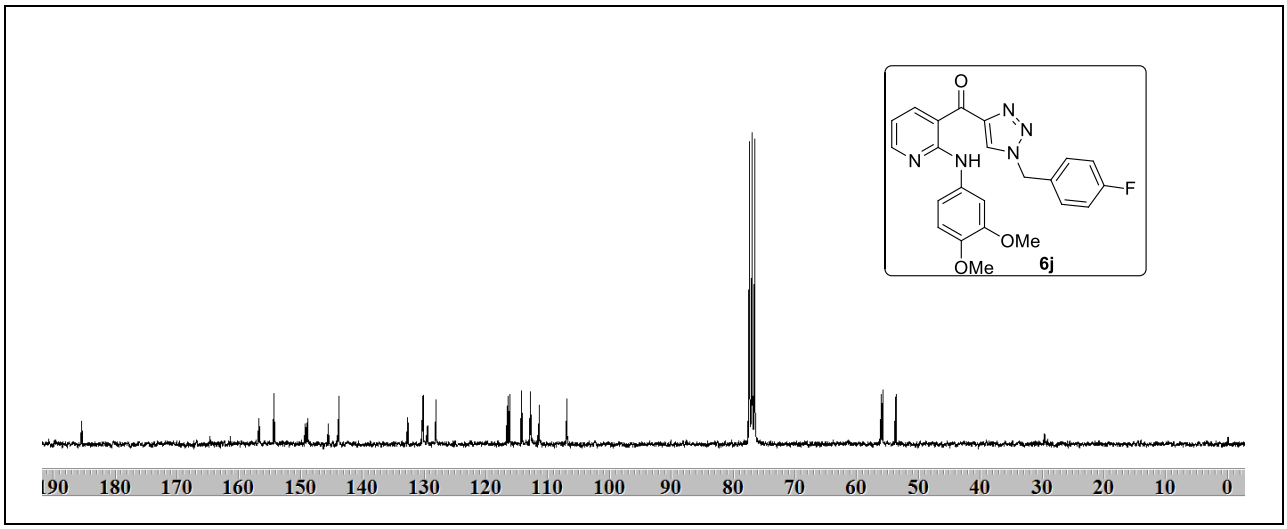
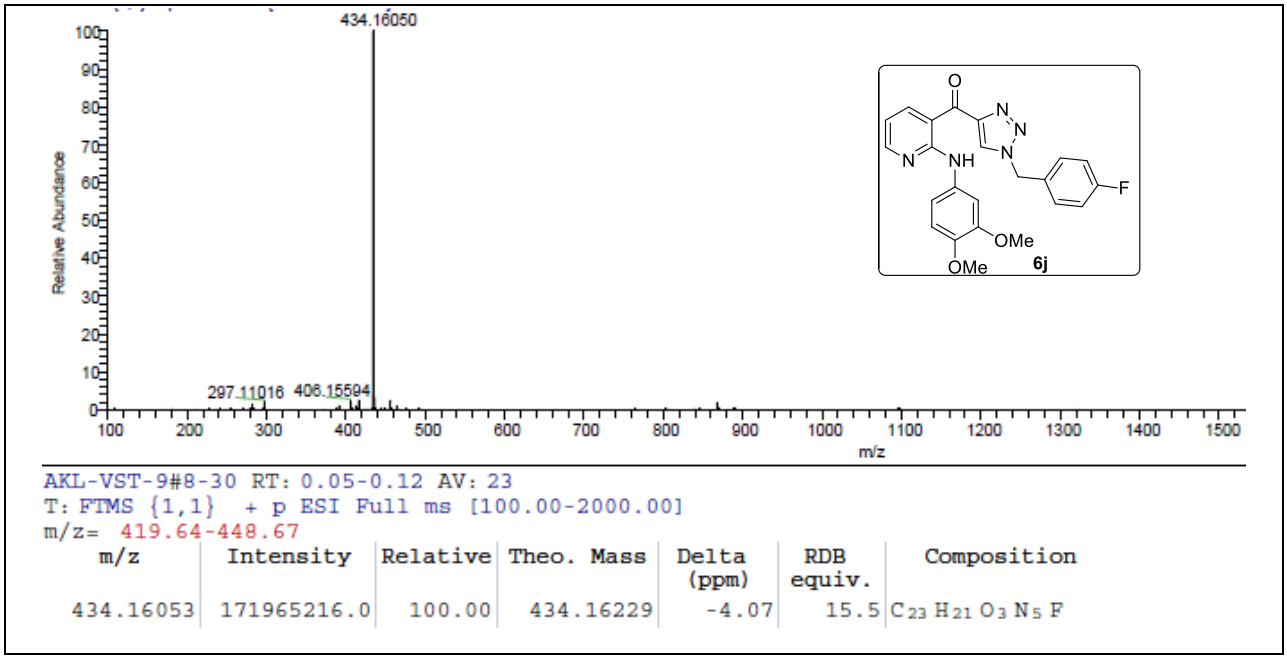


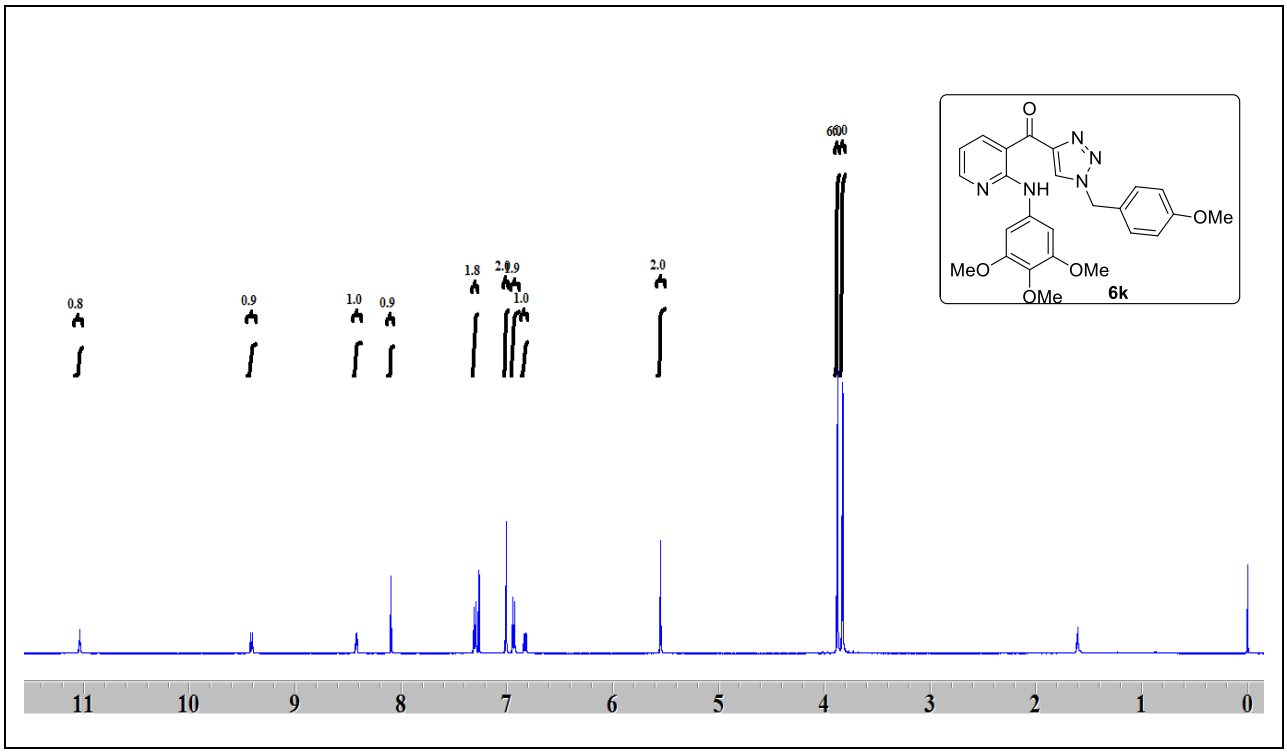
^{13}C NMR SPECTRUM OF COMPOUND 6j



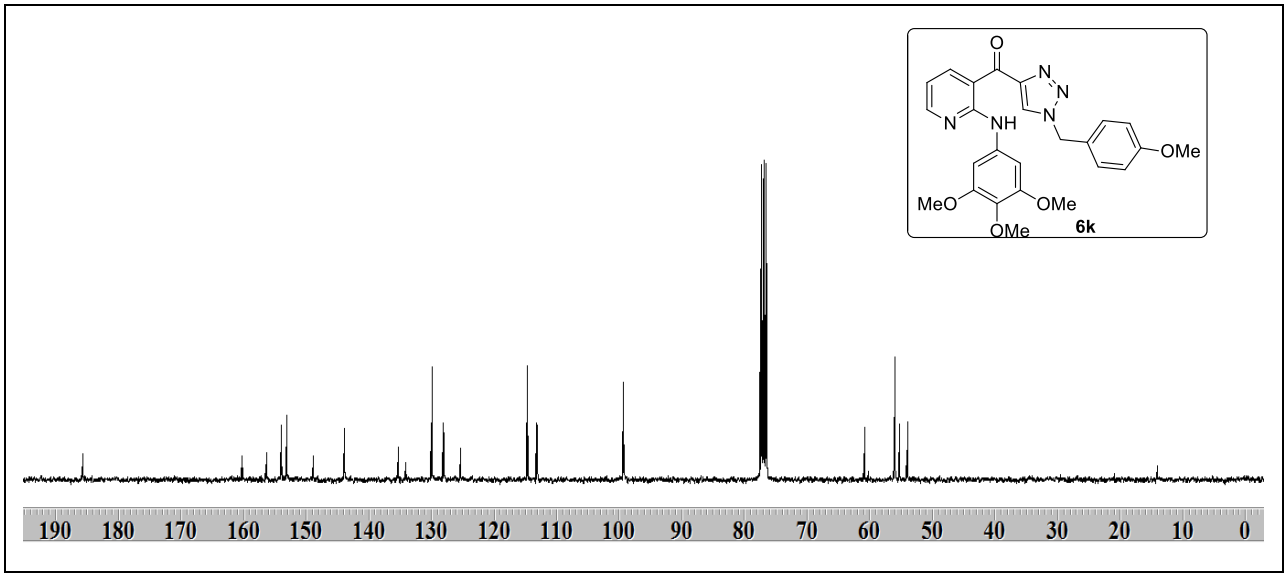
AKL-VST-9#8-30 RT: 0.05-0.12 AV: 23
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]
m/z= 419.64-448.67

m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
434.16053	171965216.0	100.00	434.16229	-4.07	15.5	$C_{23}H_{21}O_3N_5F$

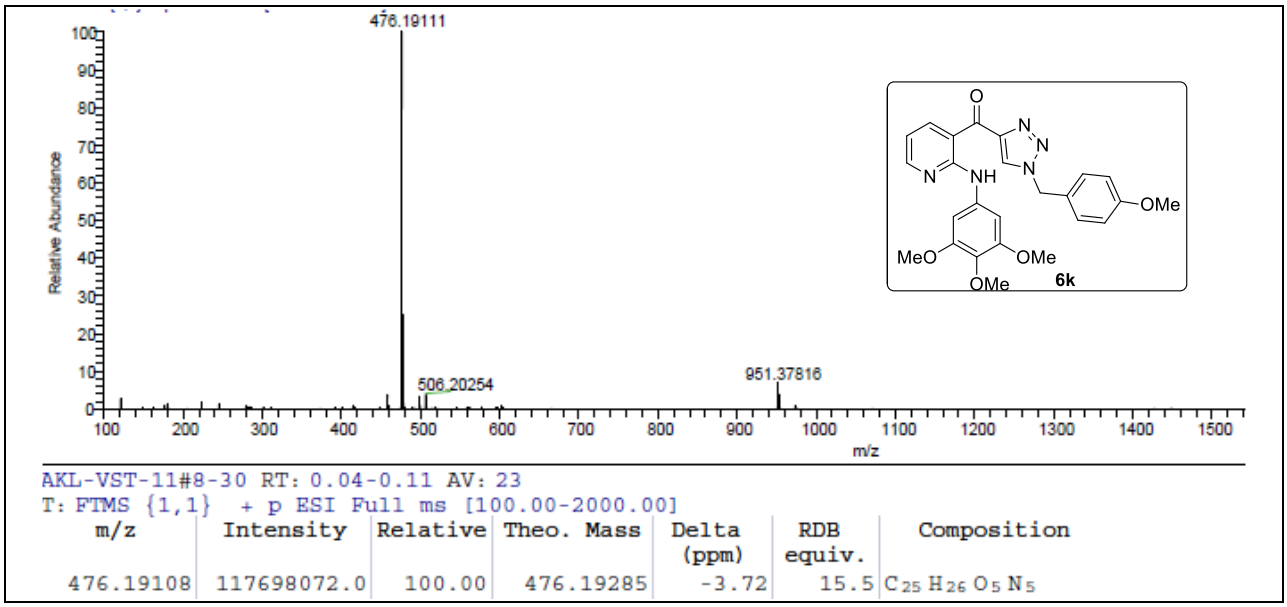
HIGH RESOLUTION MASS SPECTRUM OF COMPOUND 6j



^{1}H NMR SPECTRUM OF COMPOUND 6k



^{13}C NMR SPECTRUM OF COMPOUND 6k



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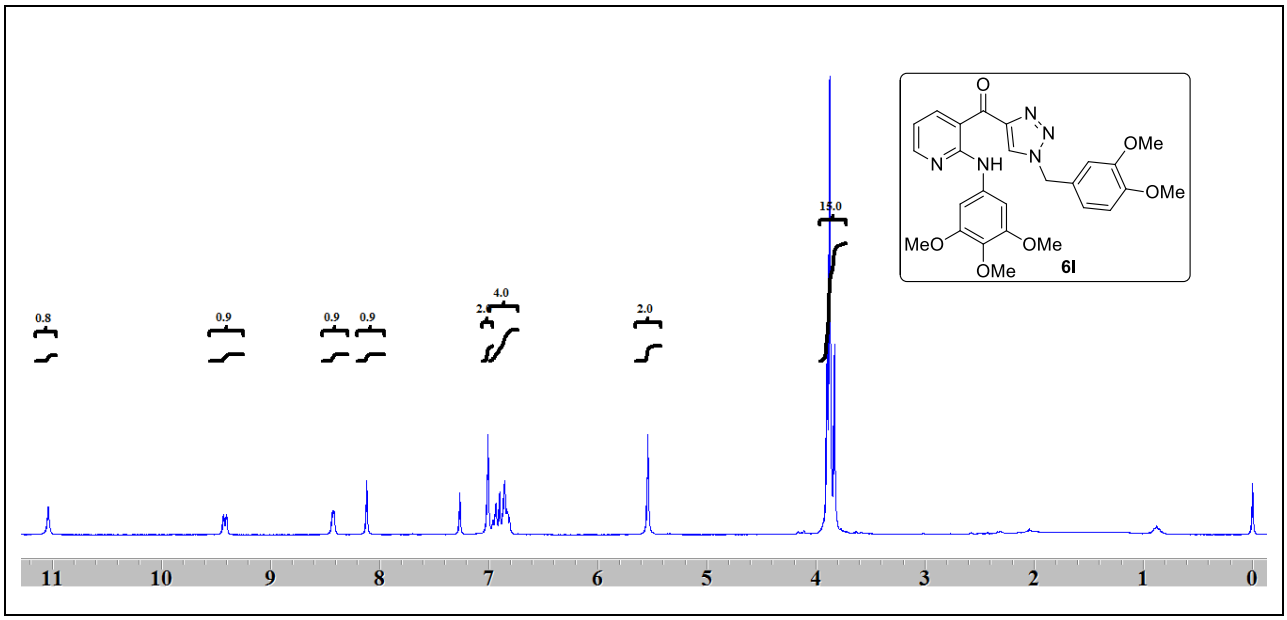
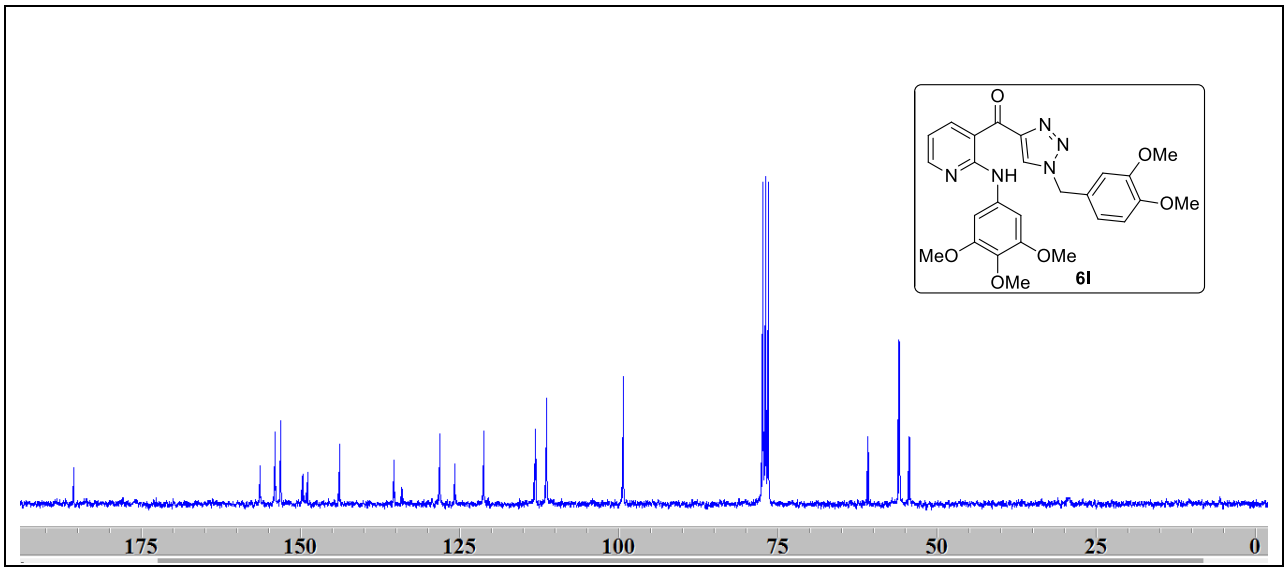
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]

m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
476.19108	117698072.0	100.00	476.19285	-3.72	15.5	$C_{25}H_{26}O_5N_5$

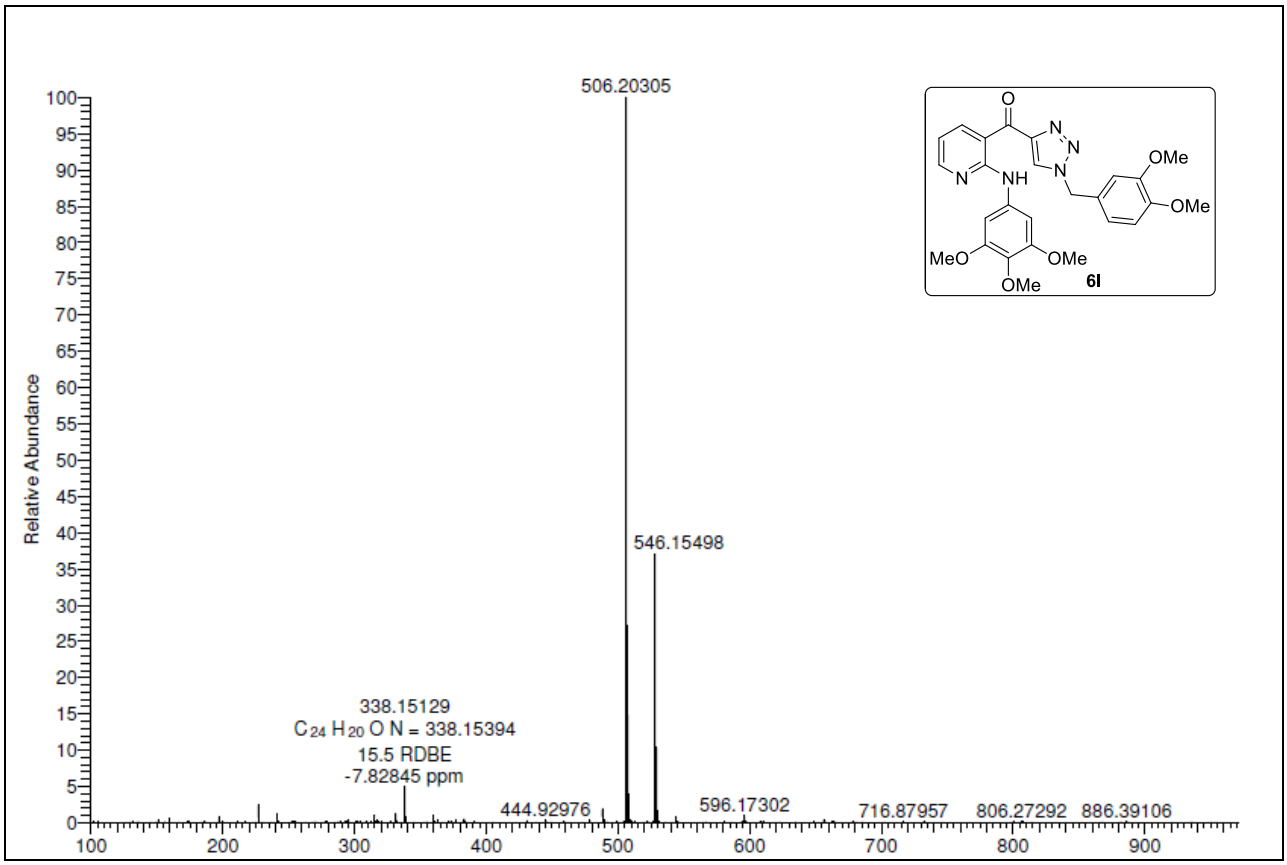
HIGH RESOLUTION MASS SPECTRUM OF COMPOUND 6k



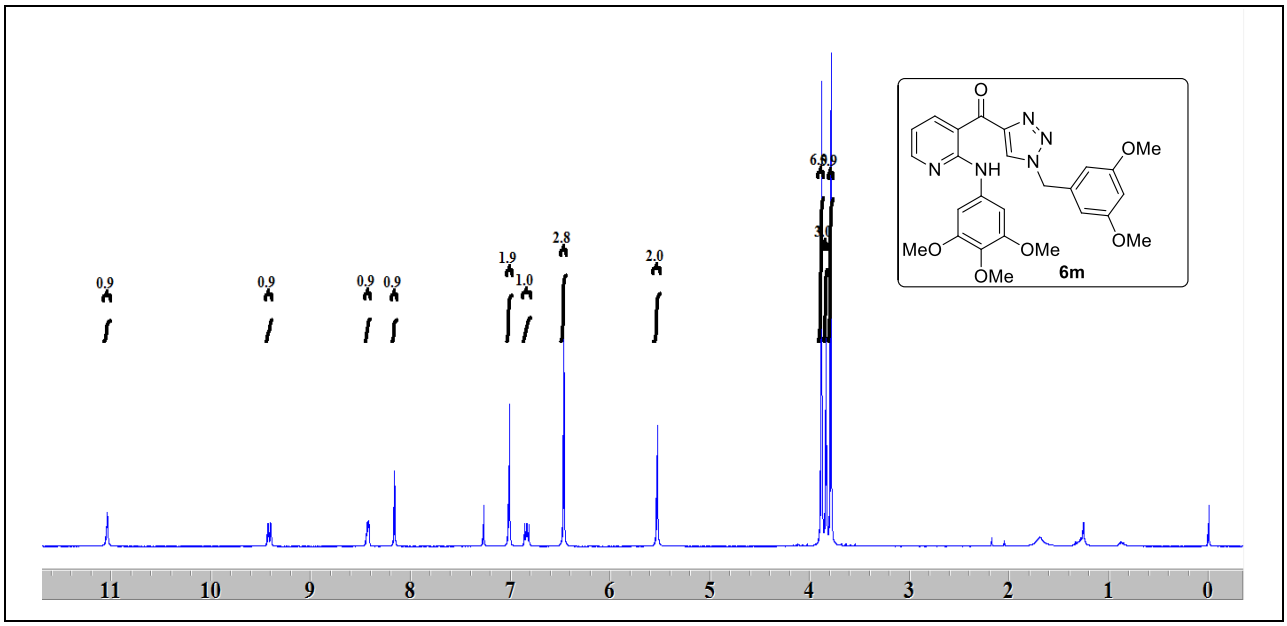
^{1}H NMR SPECTRUM OF COMPOUND 6l



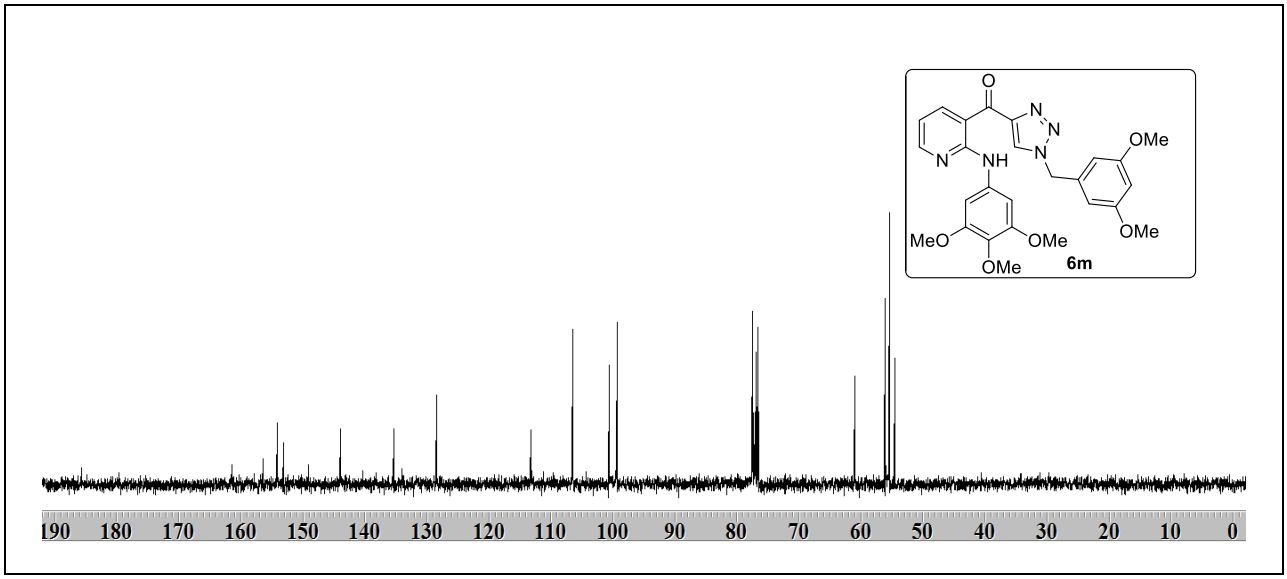
^{13}C NMR SPECTRUM OF COMPOUND 6l



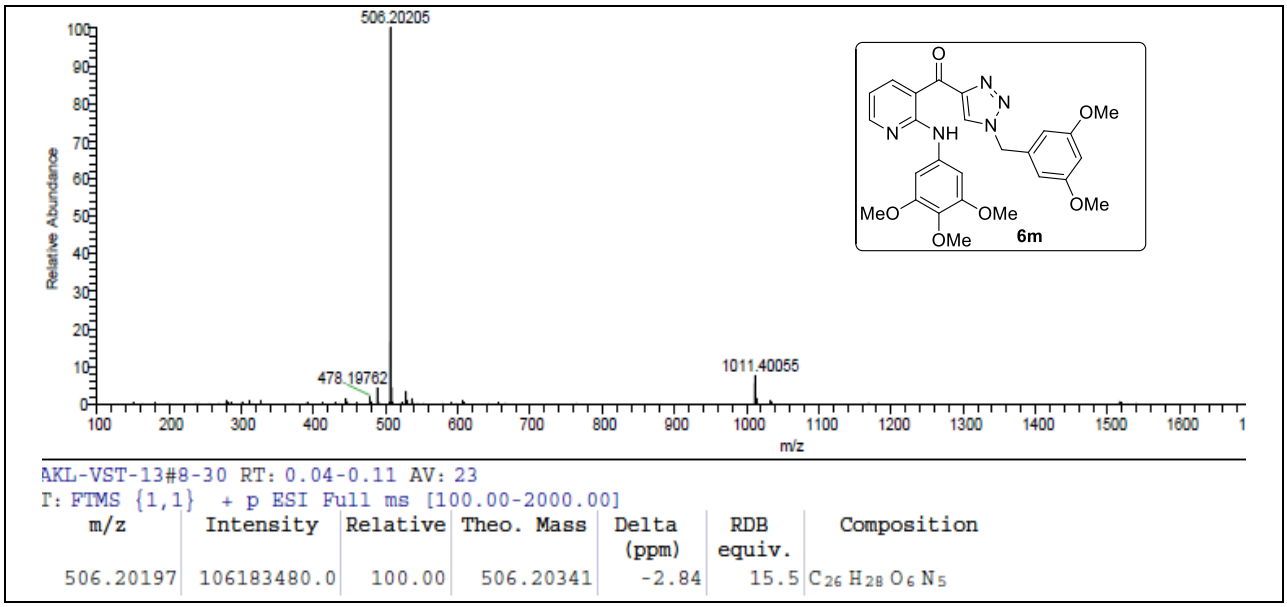
HIGH RESOLUTION MASS SPECTRUM OF COMPOUND 6l



^{1}H NMR SPECTRUM OF COMPOUND 6m



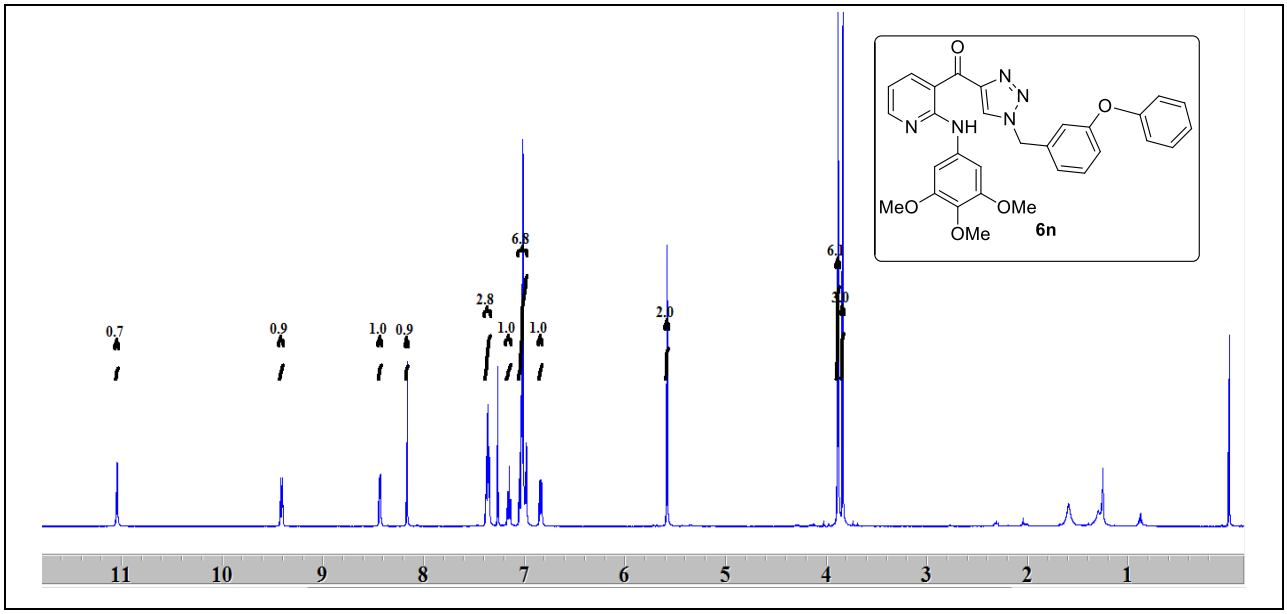
^{13}C NMR SPECTRUM OF COMPOUND 6m



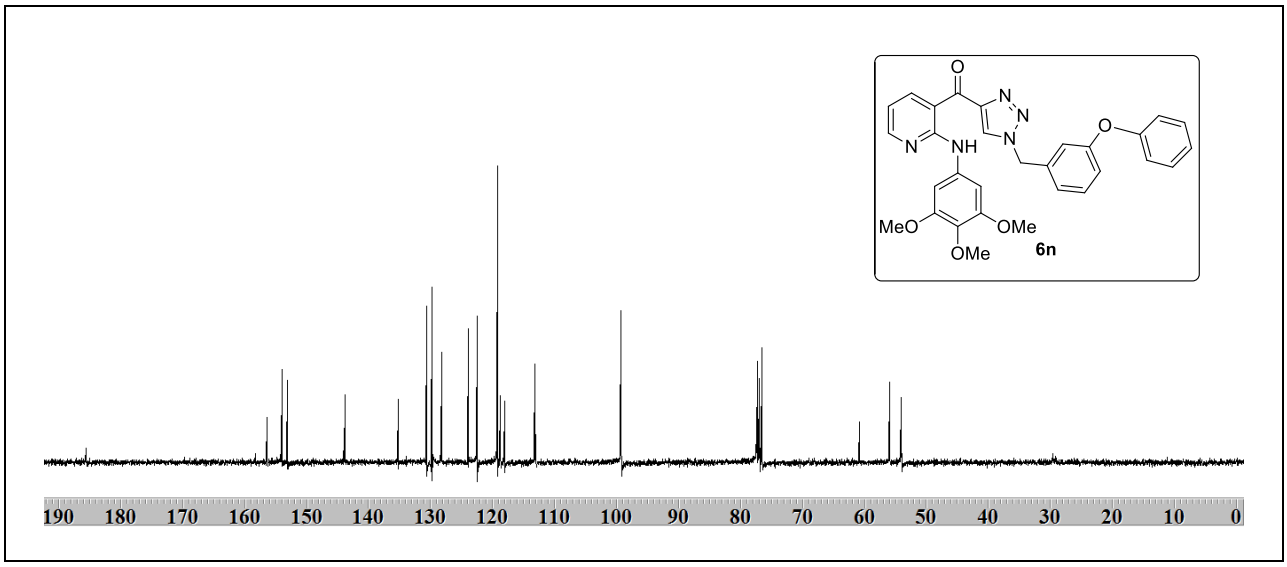
AKL-VST-13#8-30 RT: 0.04-0.11 AV: 23
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]

m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
506.20197	106183480.0	100.00	506.20341	-2.84	15.5	$C_{26}H_{28}O_6N_5$

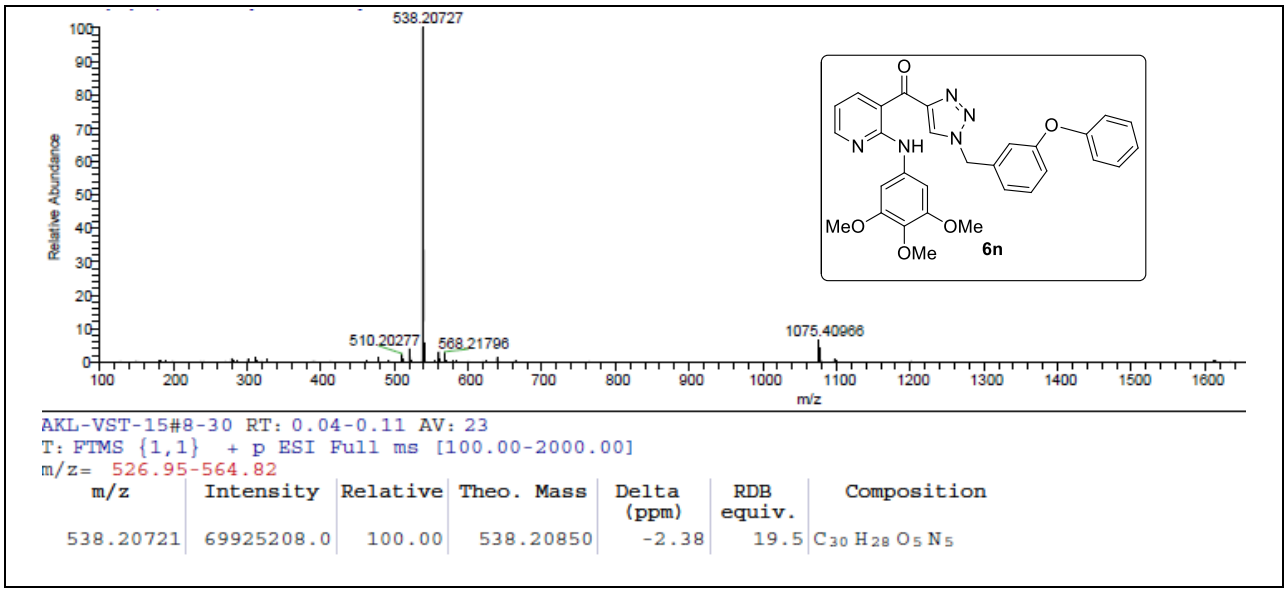
HIGH RESOLUTION MASS SPECTRUM OF COMPOUND 6m



^{1}H NMR SPECTRUM OF COMPOUND 6n

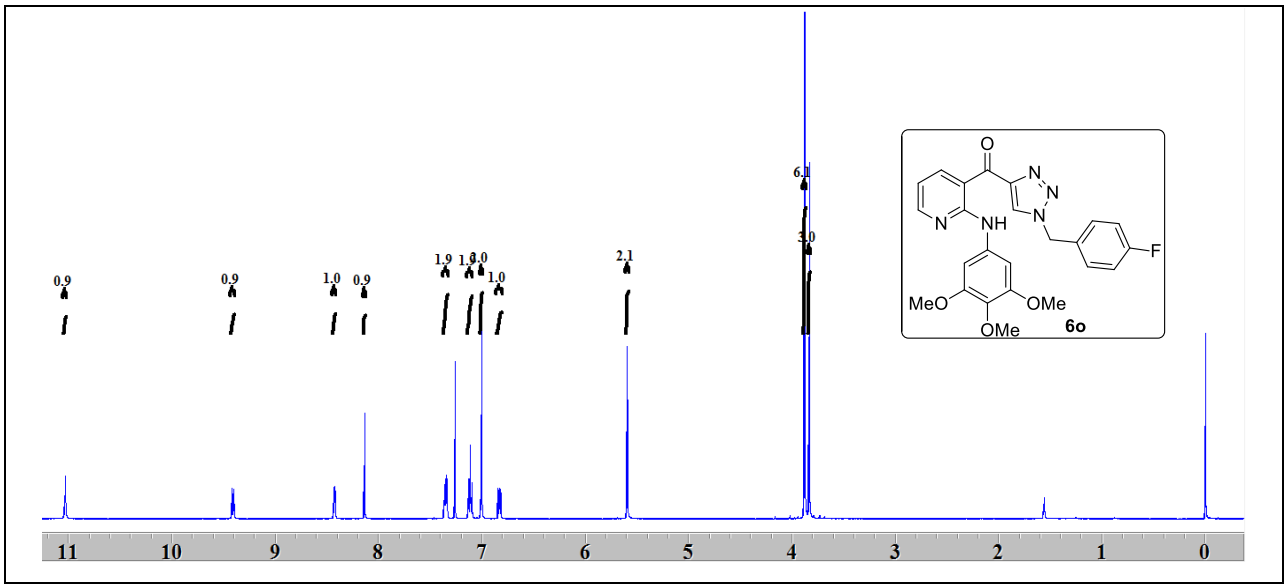


^{13}C NMR SPECTRUM OF COMPOUND 6n

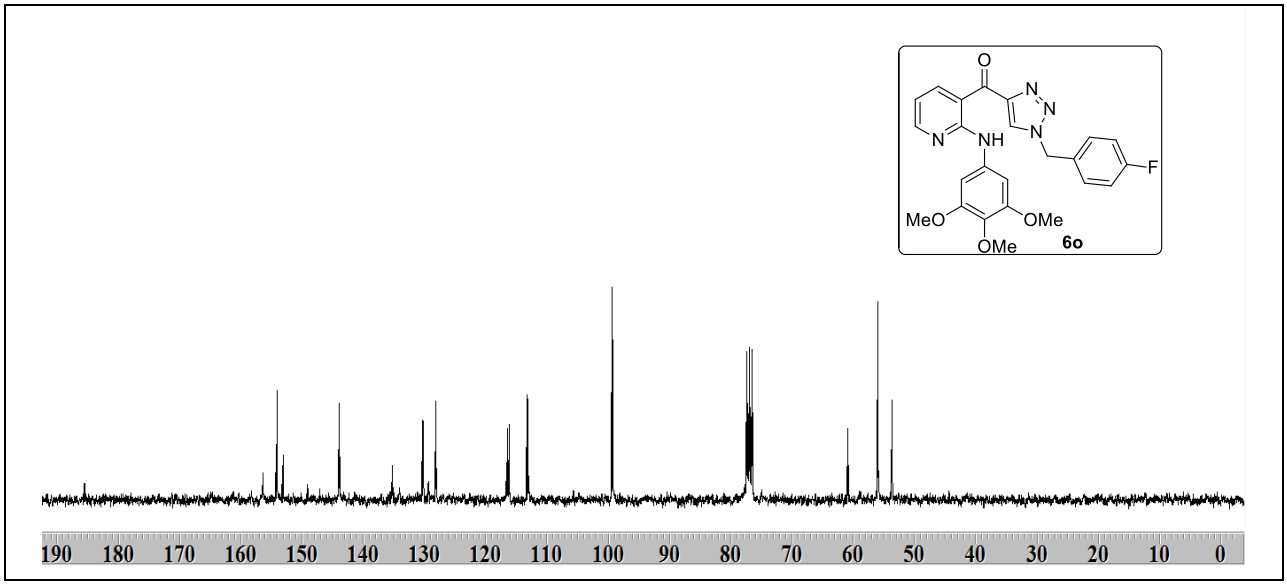


m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
538.20721	69925208.0	100.00	538.20850	-2.38	19.5	$C_{30}H_{28}O_5N_5$

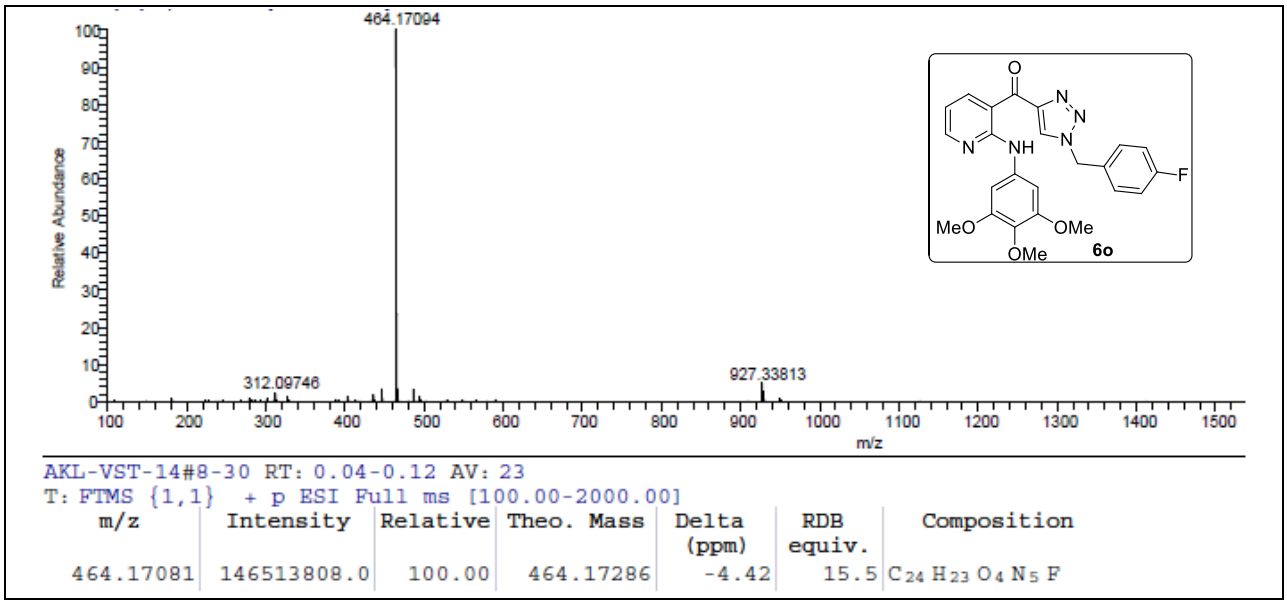
HIGH RESOLUTION MASS SPECTRUM OF COMPOUND 6n



^{1}H NMR SPECTRUM OF COMPOUND 6o



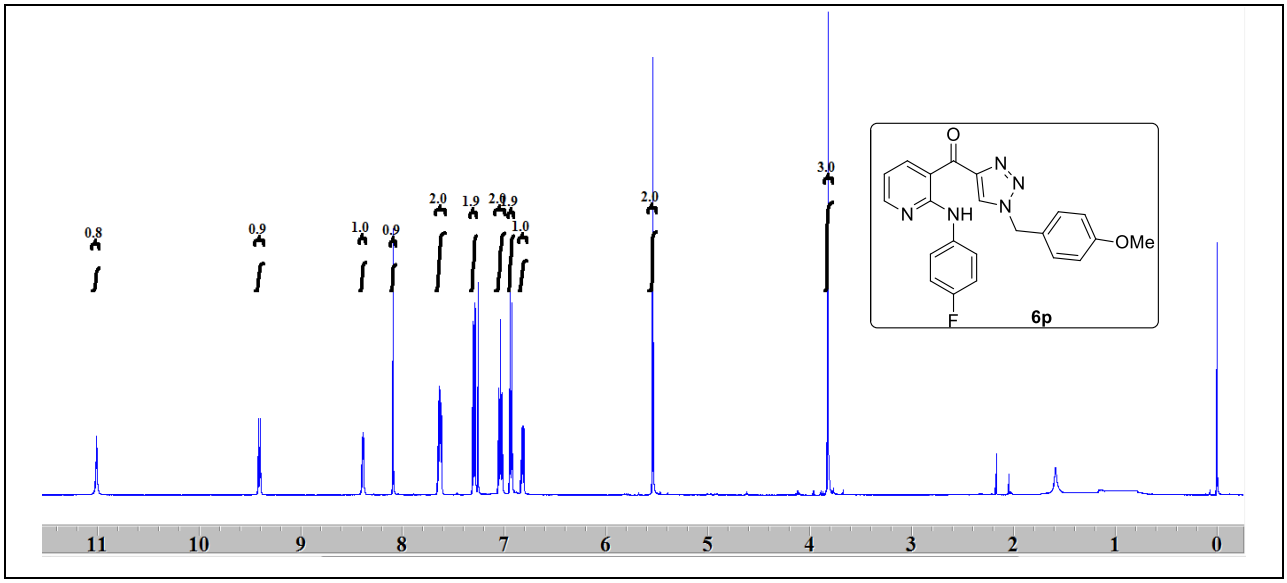
^{13}C NMR SPECTRUM OF COMPOUND 6o



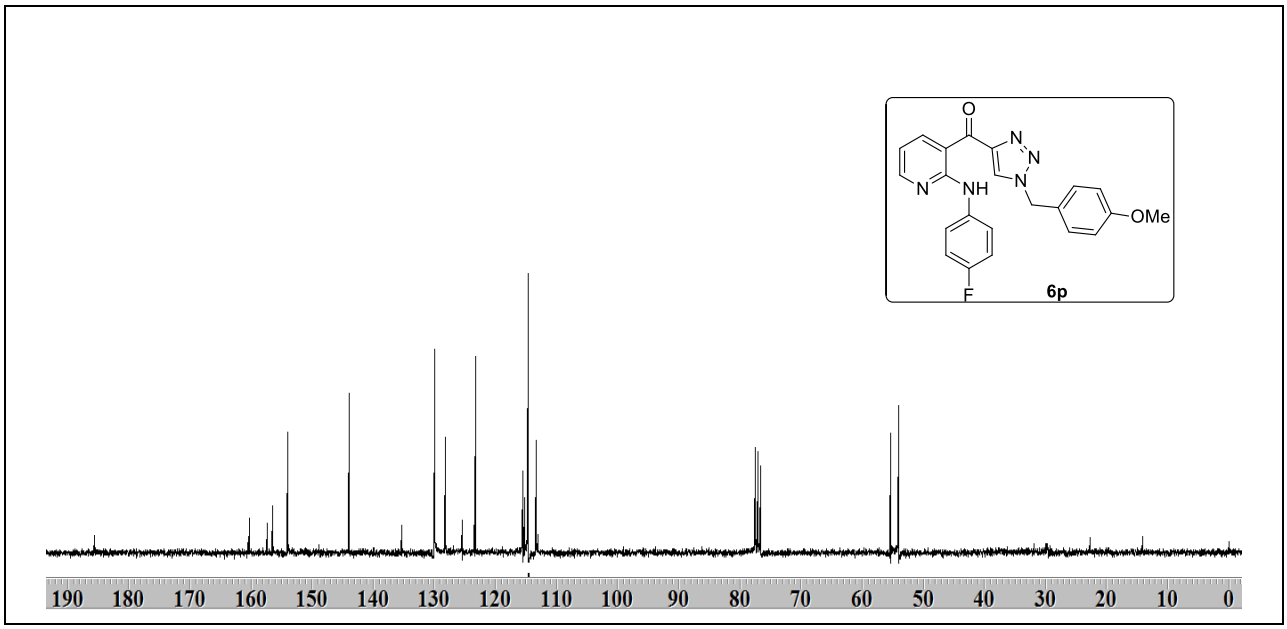
AKL-VST-14#8-30 RT: 0.04-0.12 AV: 23
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]

m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
464.17081	146513808.0	100.00	464.17286	-4.42	15.5	$C_{24}H_{23}O_4N_5F$

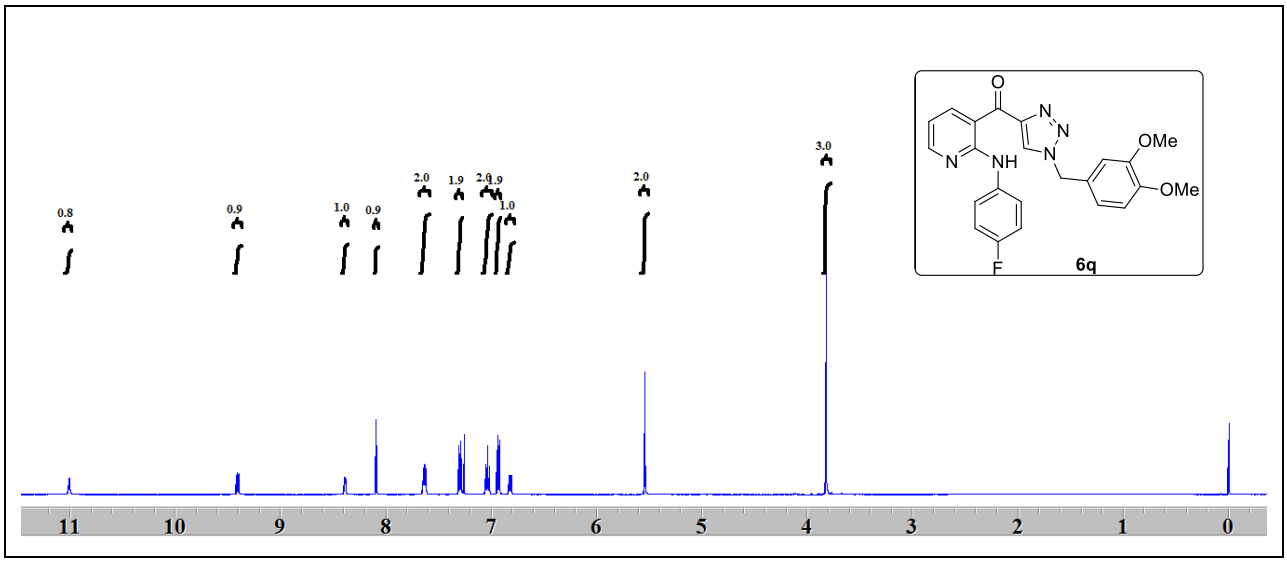
HIGH RESOLUTION MASS SPECTRUM OF COMPOUND 6o



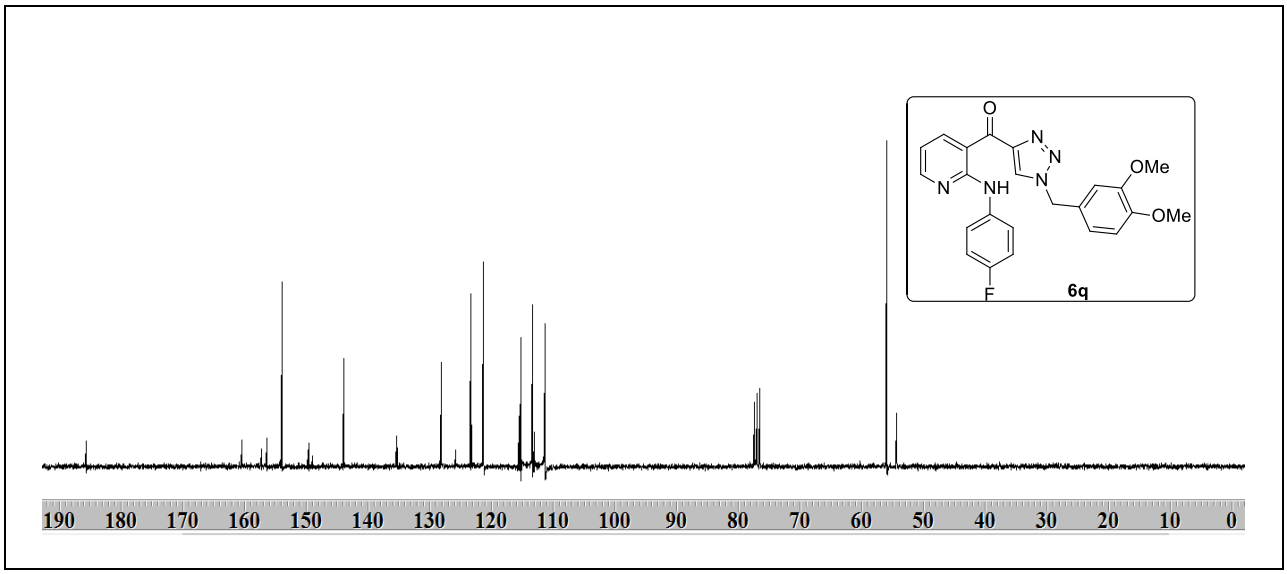
^{1}H NMR SPECTRUM OF COMPOUND 6p



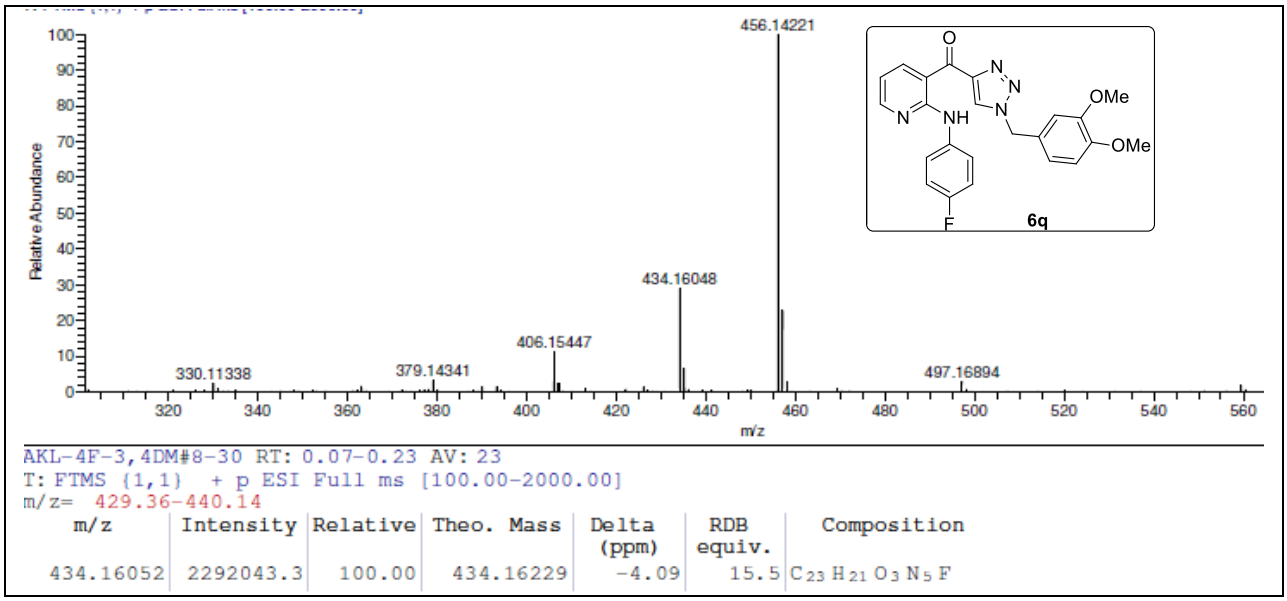
^{13}C NMR SPECTRUM OF COMPOUND 6p



^{1}H NMR SPECTRUM OF COMPOUND 6q

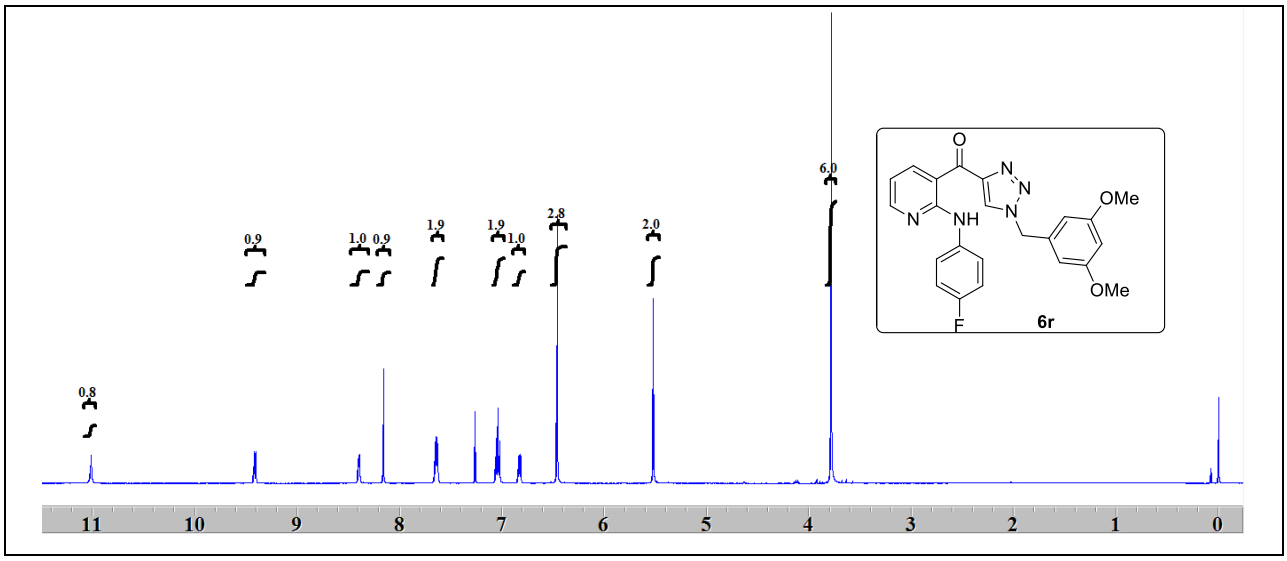


^{13}C NMR SPECTRUM OF COMPOUND 6q

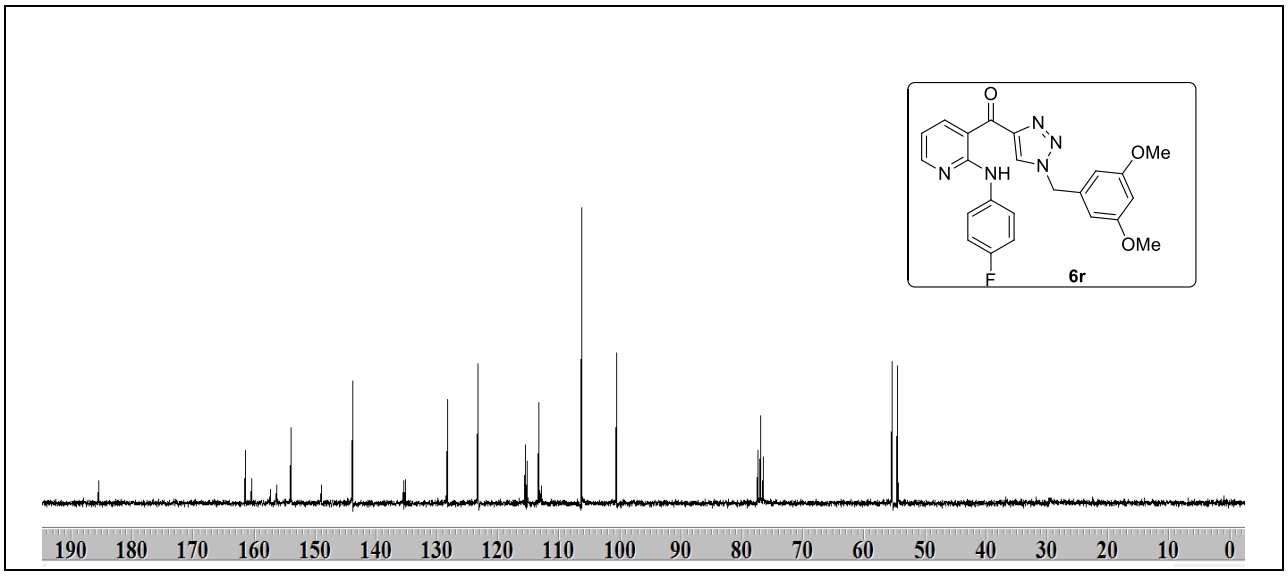


m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
434.16052	2292043.3	100.00	434.16229	-4.09	15.5	$C_{23}H_{21}O_3N_5F$

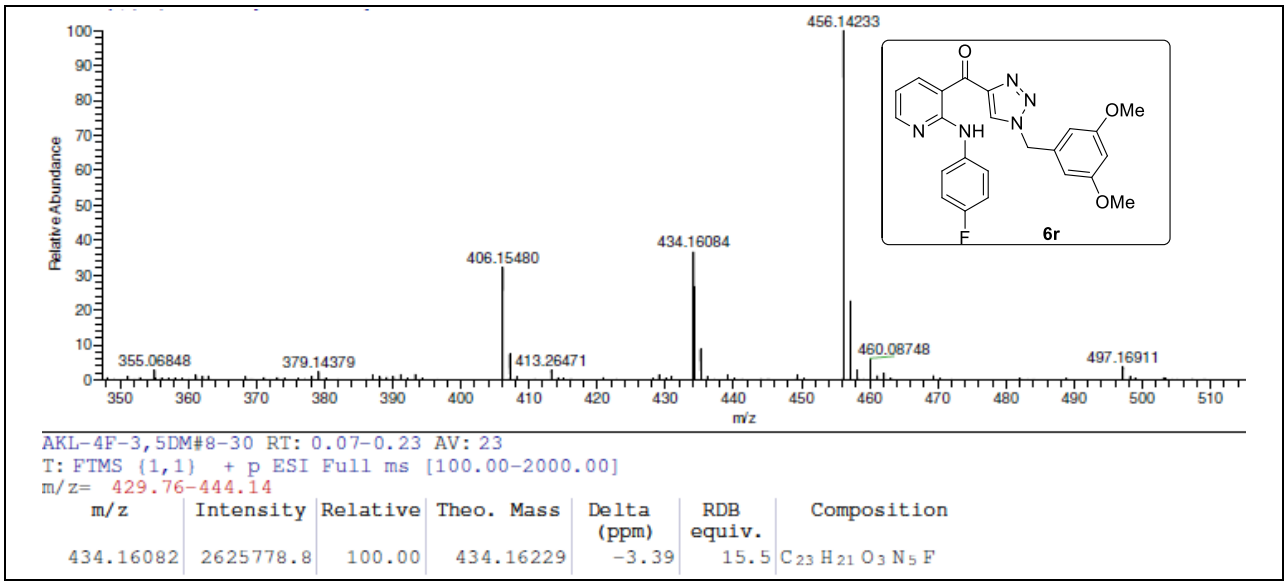
HIGH RESOLUTION MASS SPECTRUM OF COMPOUND 6q



^{1}H NMR SPECTRUM OF COMPOUND 6r

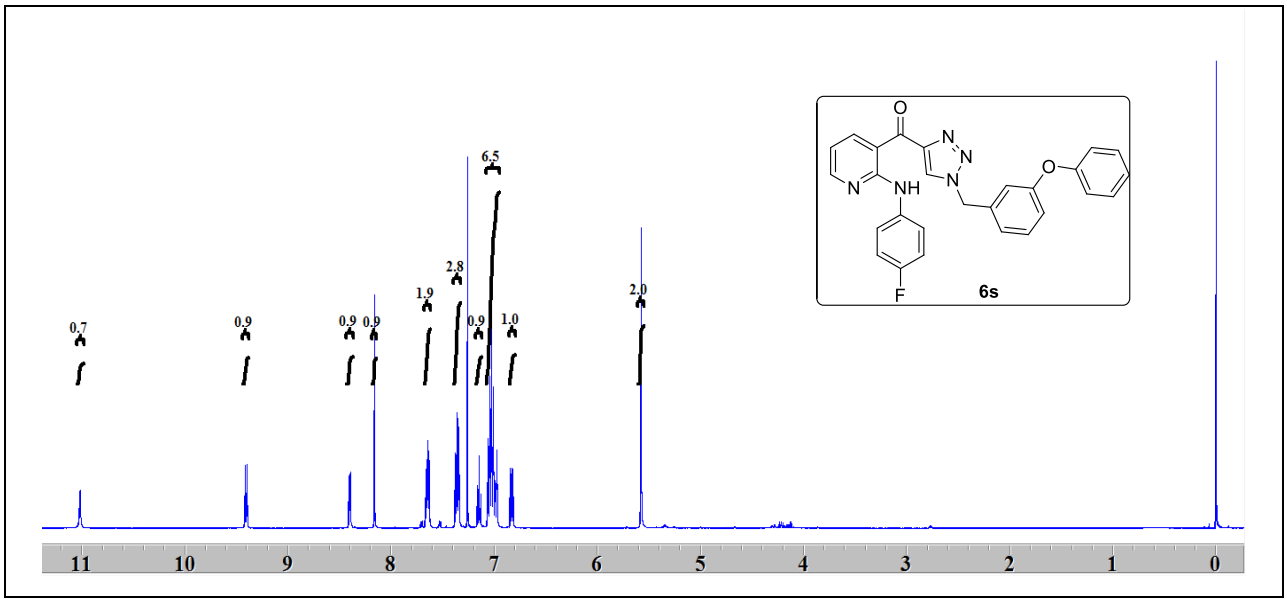


^{13}C NMR SPECTRUM OF COMPOUND 6r

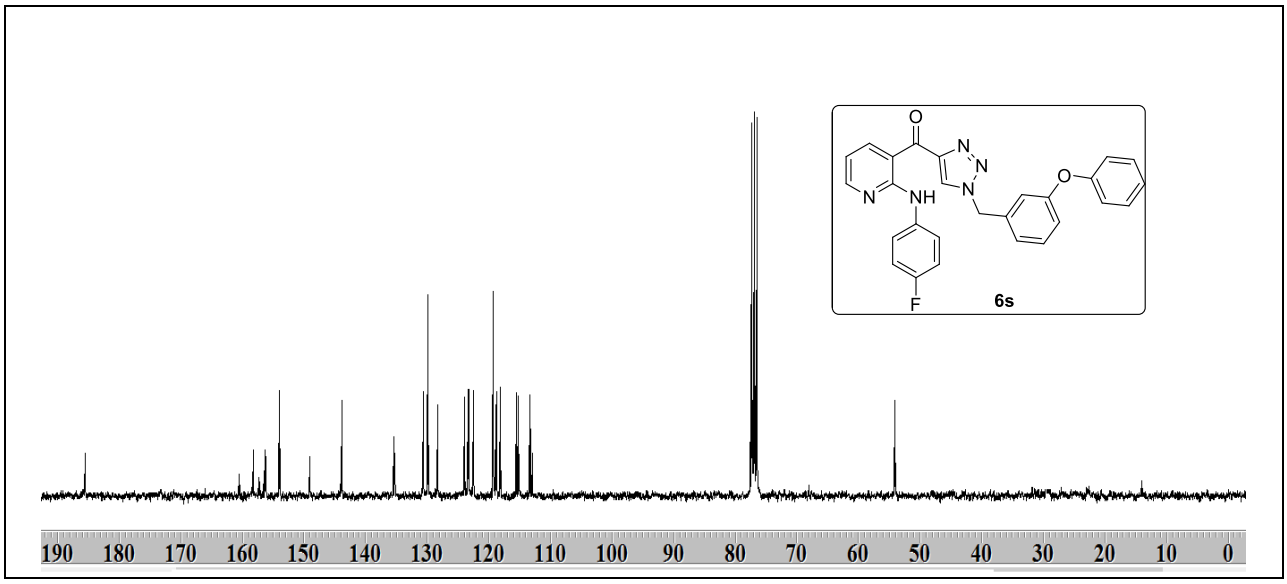


m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
434.16082	2625778.8	100.00	434.16229	-3.39	15.5	$C_{23}H_{21}O_3N_5F$

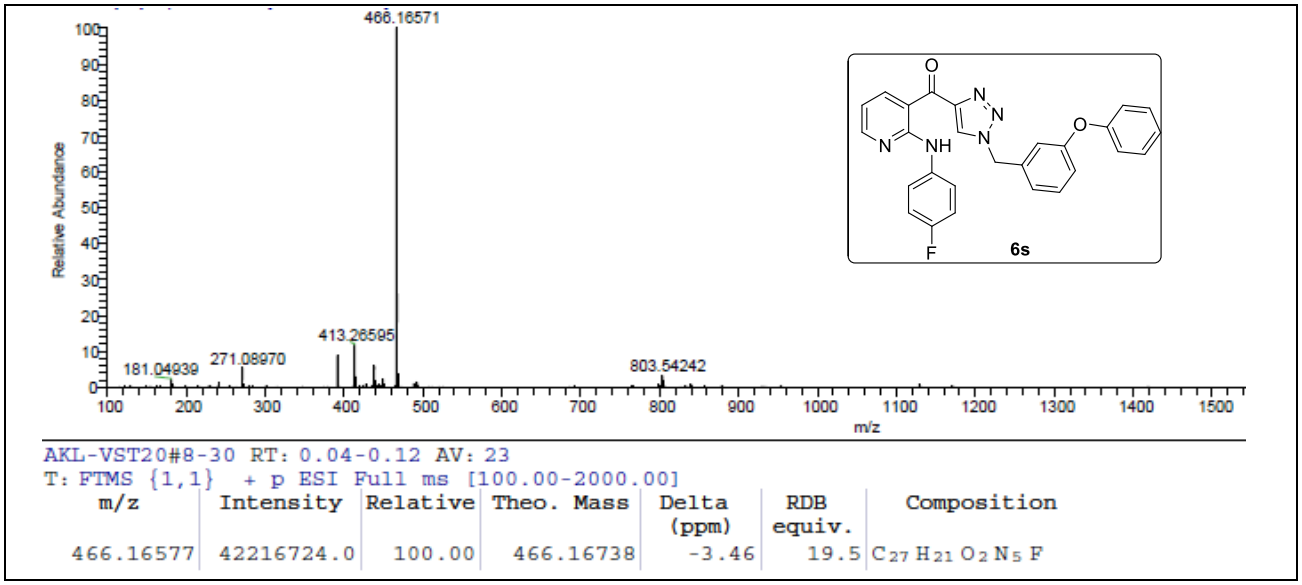
HIGH RESOLUTION MASS SPECTRUM OF COMPOUND 6r



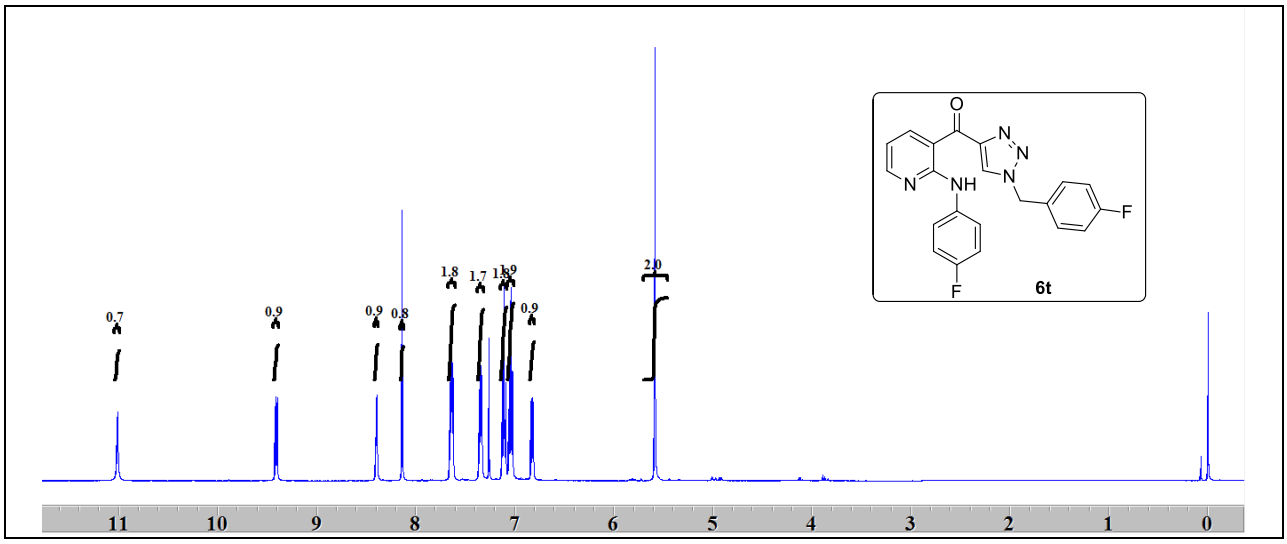
^{1}H NMR SPECTRUM OF COMPOUND 6s



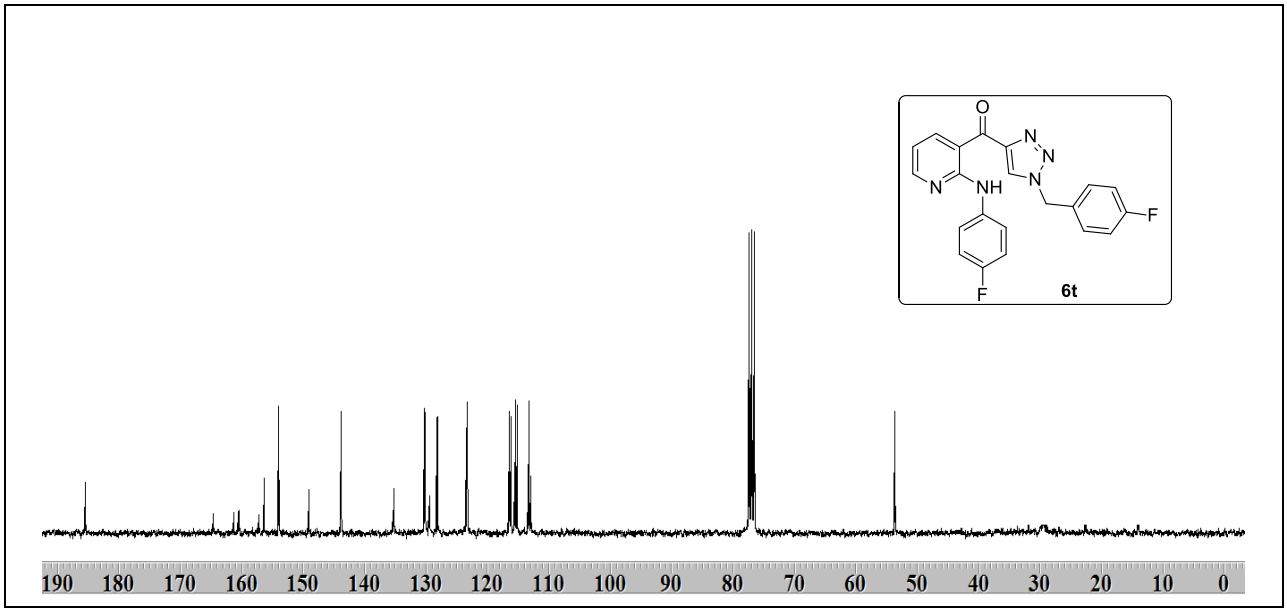
^{13}C NMR SPECTRUM OF COMPOUND 6s



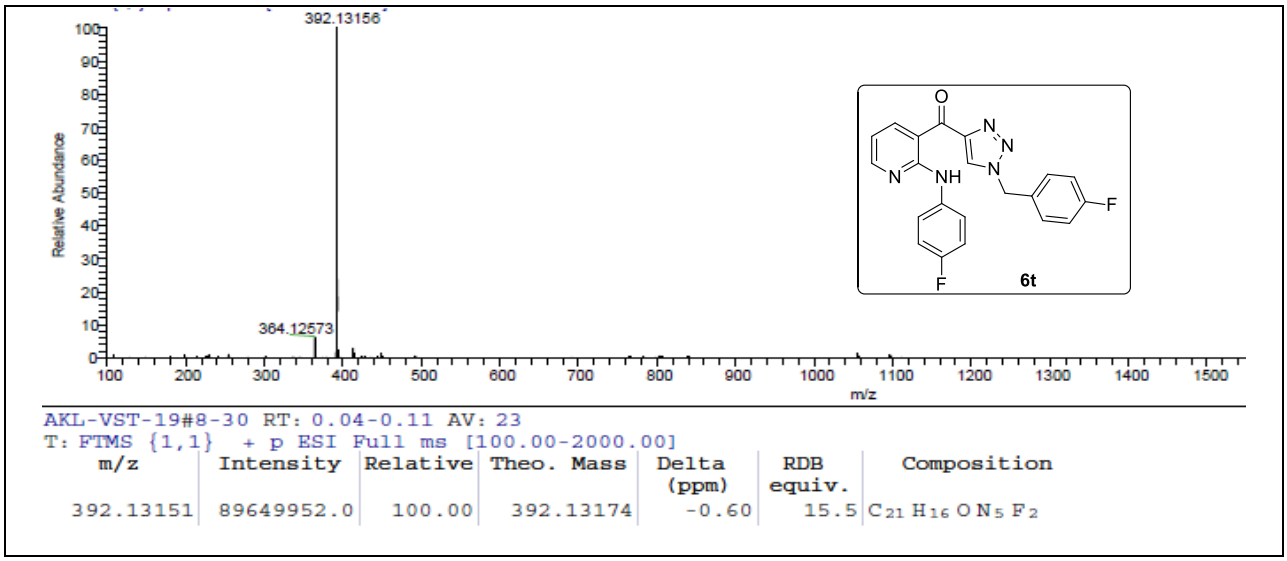
HIGH RESOLUTION MASS SPECTRUM OF COMPOUND 6s



^{1}H NMR SPECTRUM OF COMPOUND 6t



^{13}C NMR SPECTRUM OF COMPOUND 6t



AKL-VST-19#8-30 RT: 0.04-0.11 AV: 23
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]

m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
392.13151	89649952.0	100.00	392.13174	-0.60	15.5	$C_{21}H_{16}ON_5F_2$

HIGH RESOLUTION MASS SPECTRUM OF COMPOUND 6t