

## **A One-Pot Ultrasound-Assisted Regio and Stereoselective Synthesis of Indenoquinoxaline Engrafted Spiropyrrolidines**

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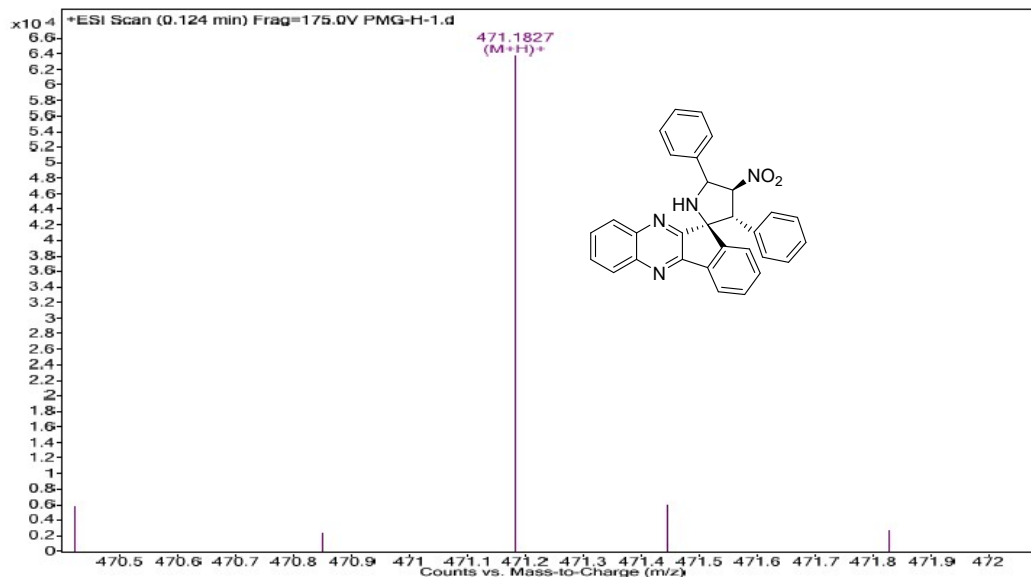
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**X-Ray Crystallographic analysis of compound 5k:** Upon repeated crystallisation from chloroform using the vapor diffusion method, needles of **5i** were obtained. The single crystal X-ray diffraction study of a single crystal of **5k** (Crystal size/mm<sup>3</sup> 0.28 × 0.22 × 0.2) was carried out by mounting a single crystal on top of thin glass fibre glued with epoxy glue. The single crystal X-ray diffraction data of the compound **5k** of was collected on Bruker D8 Quest diffractometer equipped with a microfocus anode (MoK $\alpha$ ) (Radiation  $\lambda$  = 0.71073) and a PHOTON 100 CMOS detector. The crystal was kept at 294.0 K during data collection. The data were integrated and scaled using Bruker suite program and the structures were solved by the direct method and then refined by full-matrix least-squares minimization using SHELXL. All the non-hydrogen atoms were refined anisotropically and the hydrogen atoms were placed using calculated position on riding model.

# HR-ESIMS Spectrum of compound 5a

<b>INSTRUMENT</b>	<b>UNAVAILABLE</b>	<b>POSITION</b>	<b>UNAVAILABLE</b>	<b>SAMPLETYPE</b>	<b>UNAVAILABLE</b>	<b>STATUS</b>	<b>Success</b>
<b>Inj Vol</b>	Unavailable	<b>InjPosition</b>	Unavailable	<b>SampleType</b>	Unavailable	<b>IRM Calibration Status</b>	Success
<b>Data Filename</b>	PMG-H-1.d	<b>ACQ Method</b>		<b>Comment</b>	Sample information is unavailable	<b>Acquired Time</b>	Unavailable



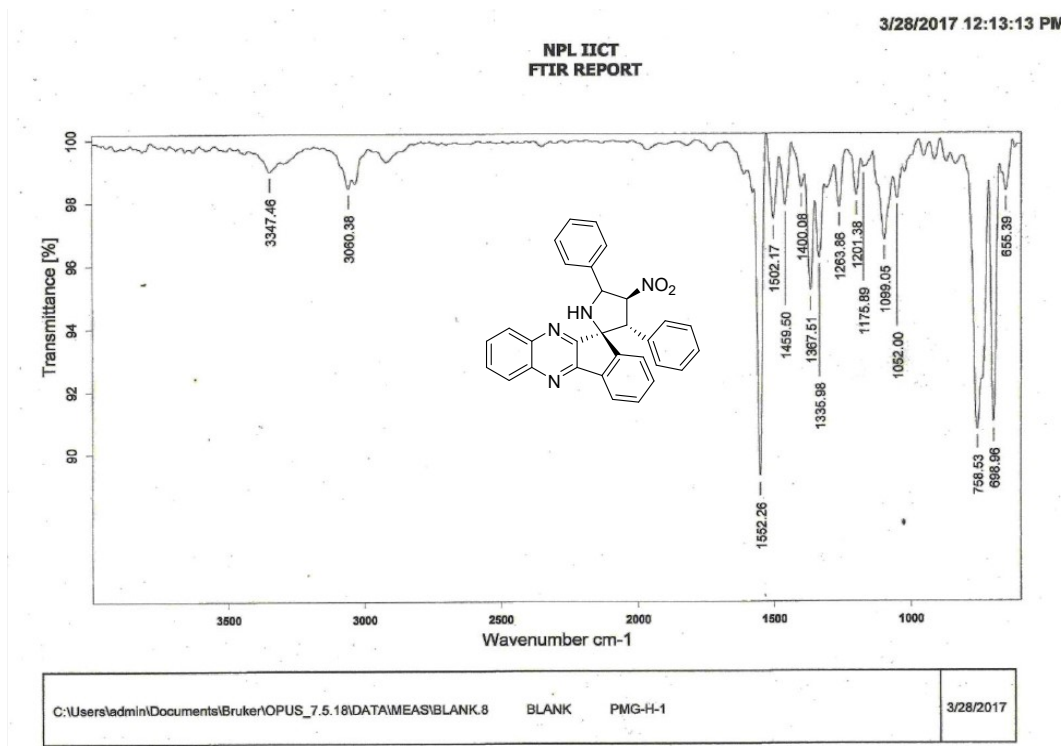
MS Formula Results: + Scan (0.124 min) - PMG-H-1.d (PMG-H-1.d)

m/z	Ion	Formula	Abundance
471.1827	(M+H) <sup>+</sup>	C <sub>30</sub> H <sub>23</sub> N <sub>2</sub> O <sub>2</sub>	63888

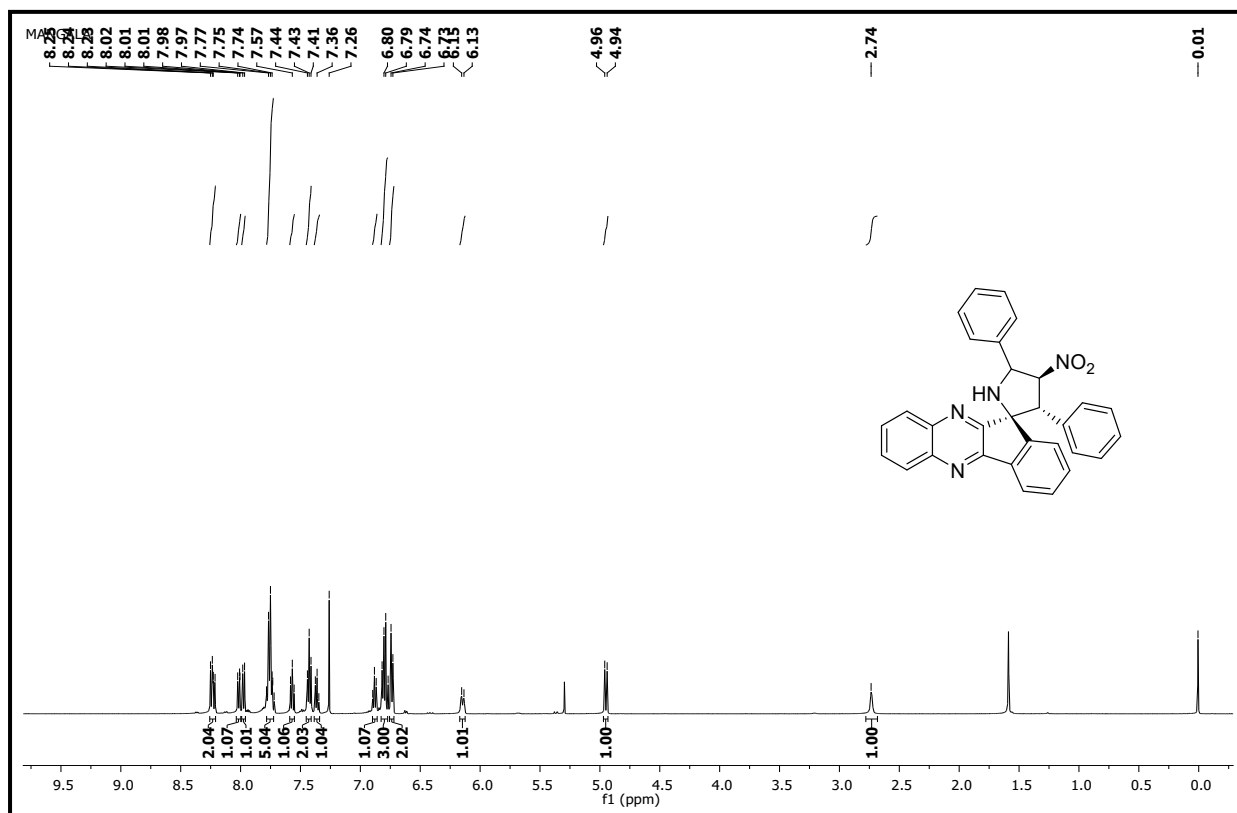
Best	Formula (M)	Ion Formula	Calc. m/z	Score	Cross Score	Mass	Calc. Mass	Diff (ppm)	Abn. Diff (ppm)	Abund. Match	Spacing Match	Mass Match	m/z	DBE
7	C <sub>30</sub> H <sub>23</sub> N <sub>2</sub> O <sub>2</sub>	C <sub>30</sub> H <sub>23</sub> N <sub>2</sub> O <sub>2</sub>	471.1816	44.7		470.1794	470.1740	-2.48	2.48	0	0	80.87	471.1827	22

# IR Spectrum of compound 5a

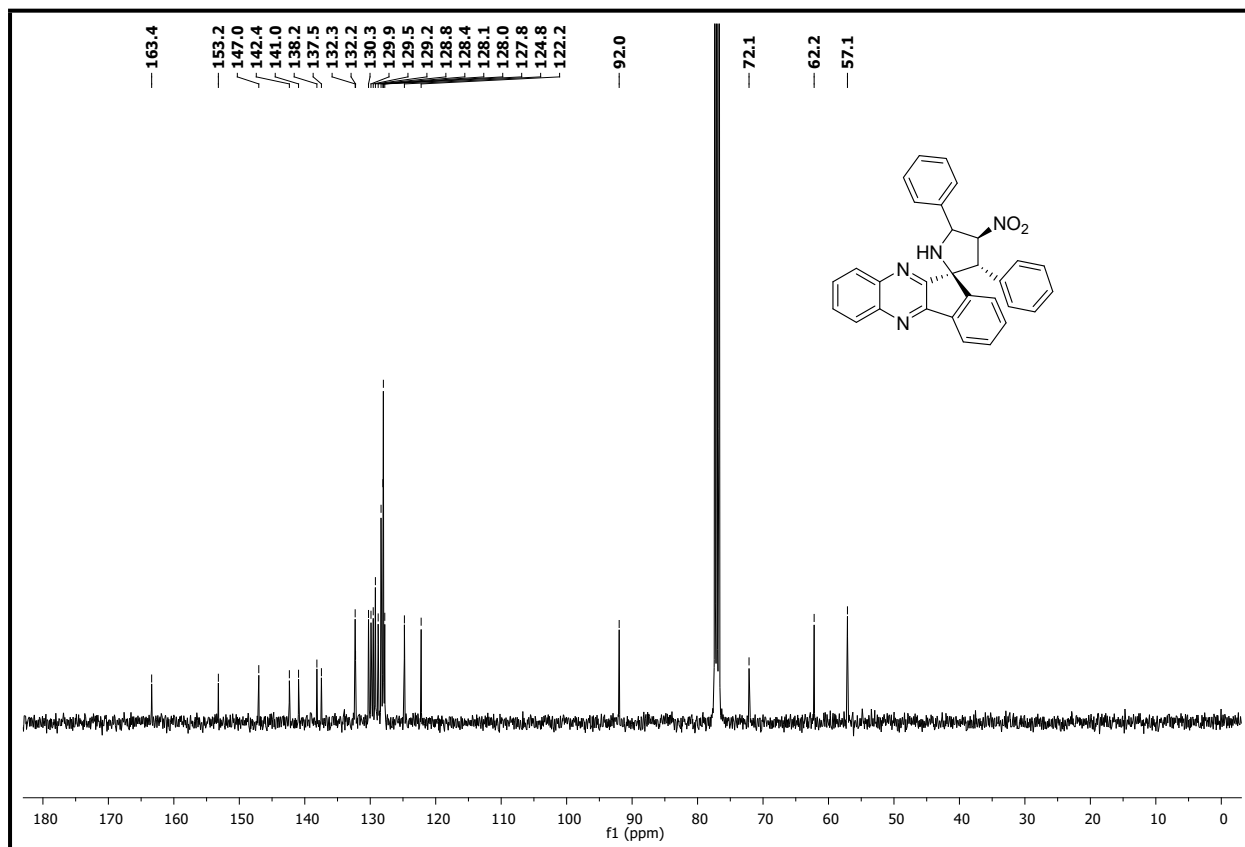


C:\Users\admin\Documents\Bruker\OPUS_7.5.18\DATA\MEAS\BLANK.8	BLANK	PMG-H-1	3/28/2017
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### <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>, 500 MHz) of compound 5a

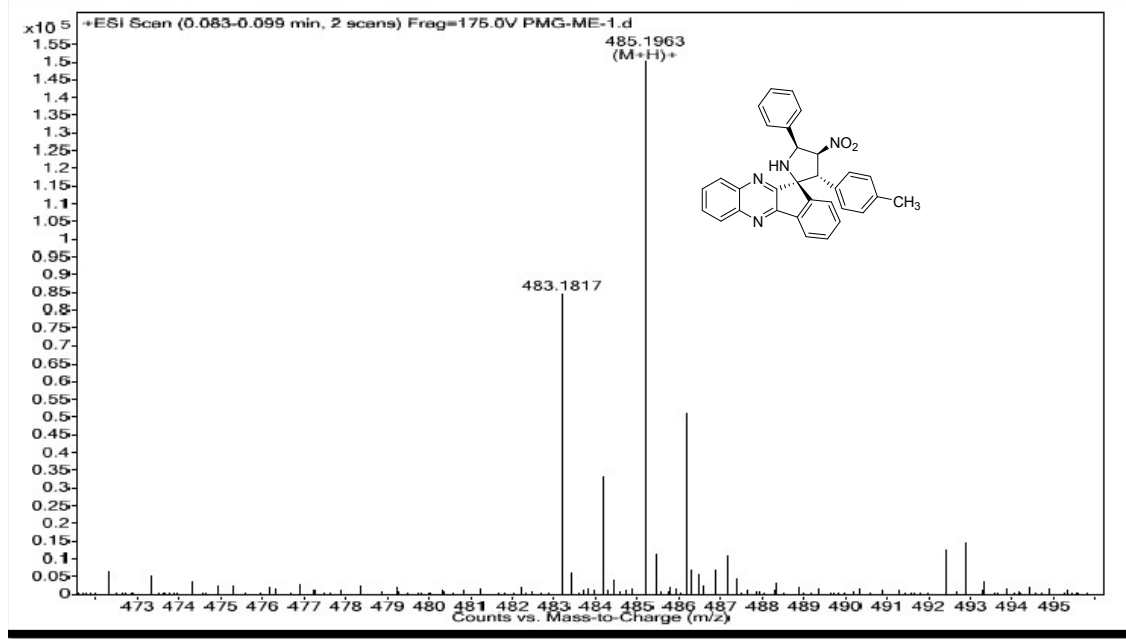


### <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>, 125 MHz) of compound 5a



# HR-ESIM Spectrum of compound 5b

**Sample Name:** PMG-ME-1  
**Inj Vol:** -1  
**Data Filename:** PMG-ME-1.d  
**Injection:** InjPosition  
**ACQ Method:** SCS...m  
**Sample Type:** Sample  
**IRM Calibration Status:** Success  
**Acquired Time:** 12/5/2016 2:05:36 PM



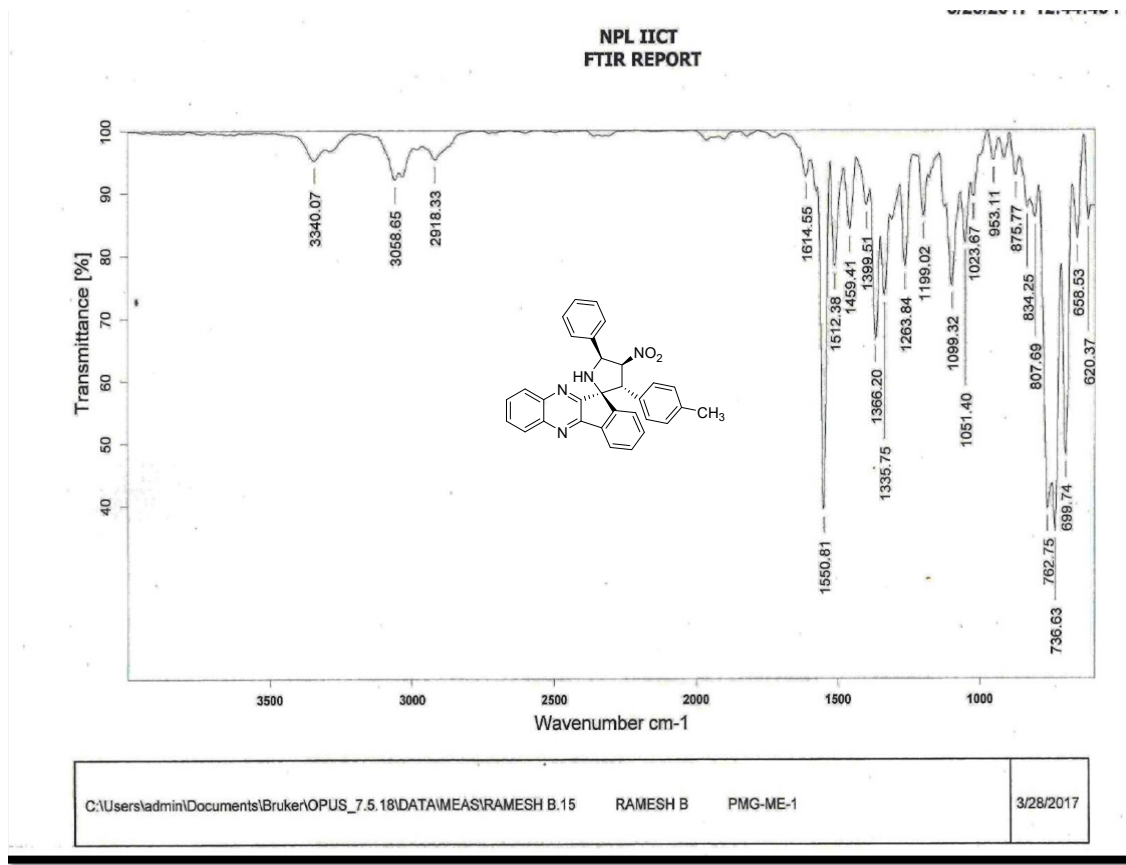
MS Formula Results: + Scan (0.083-0.099 min) - PMG-ME-1.d (PMG-ME-1.d)

m/z	Ion	Formula	Abundance
485.1963	(M+H)+	C31H25N4O2	149969.9

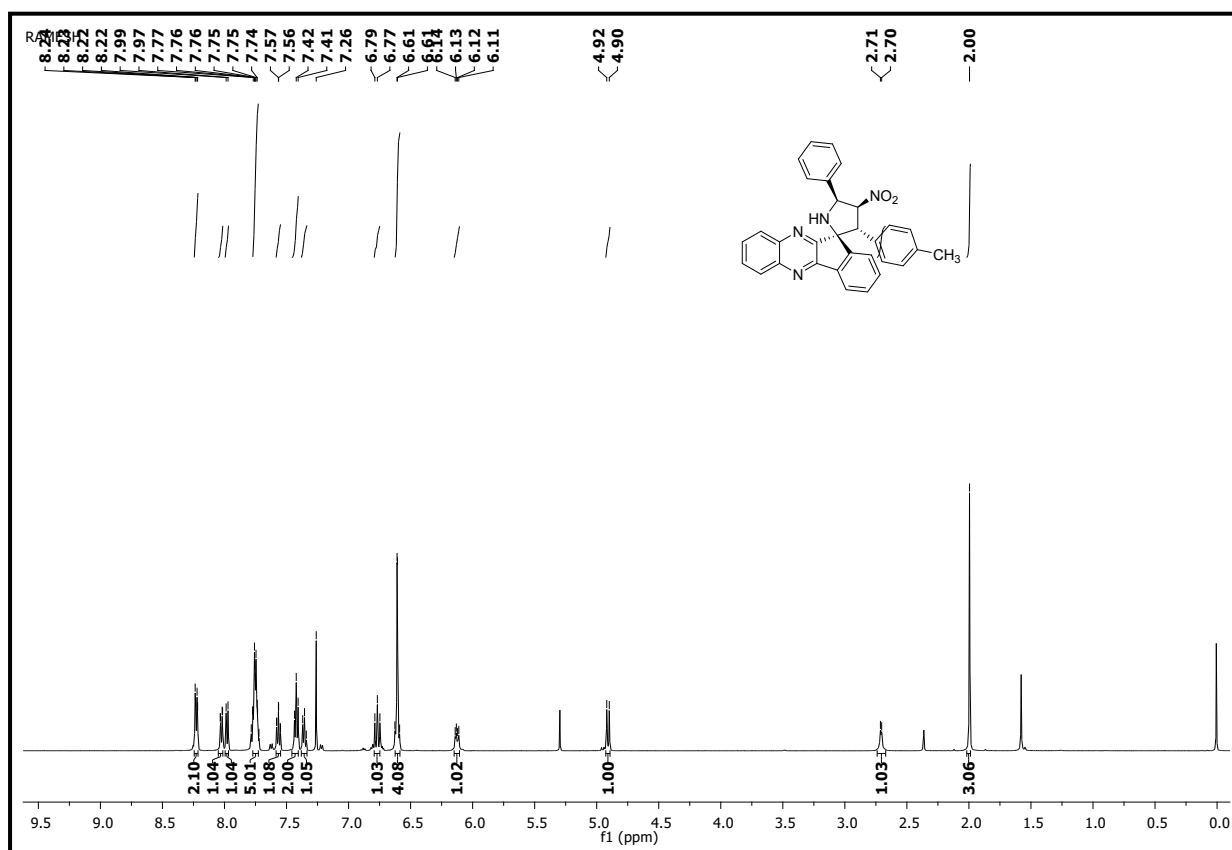
Best	Formula (M)	Ion Formula	Calc. m/z	Score	Cross Score	Mass	Calc. Mass	Diff (ppm)	Abs Diff (ppm)	Abund. Match	Spacing Match	Mass Match	m/z	DBE
✓	C31H24N4O2	C31H25N4O2	485.1972	96.56		484.1891	484.1899	1.71	1.71	98.13	93.85	96.97	485.1963	22

# IR Spectrum of compound 5b

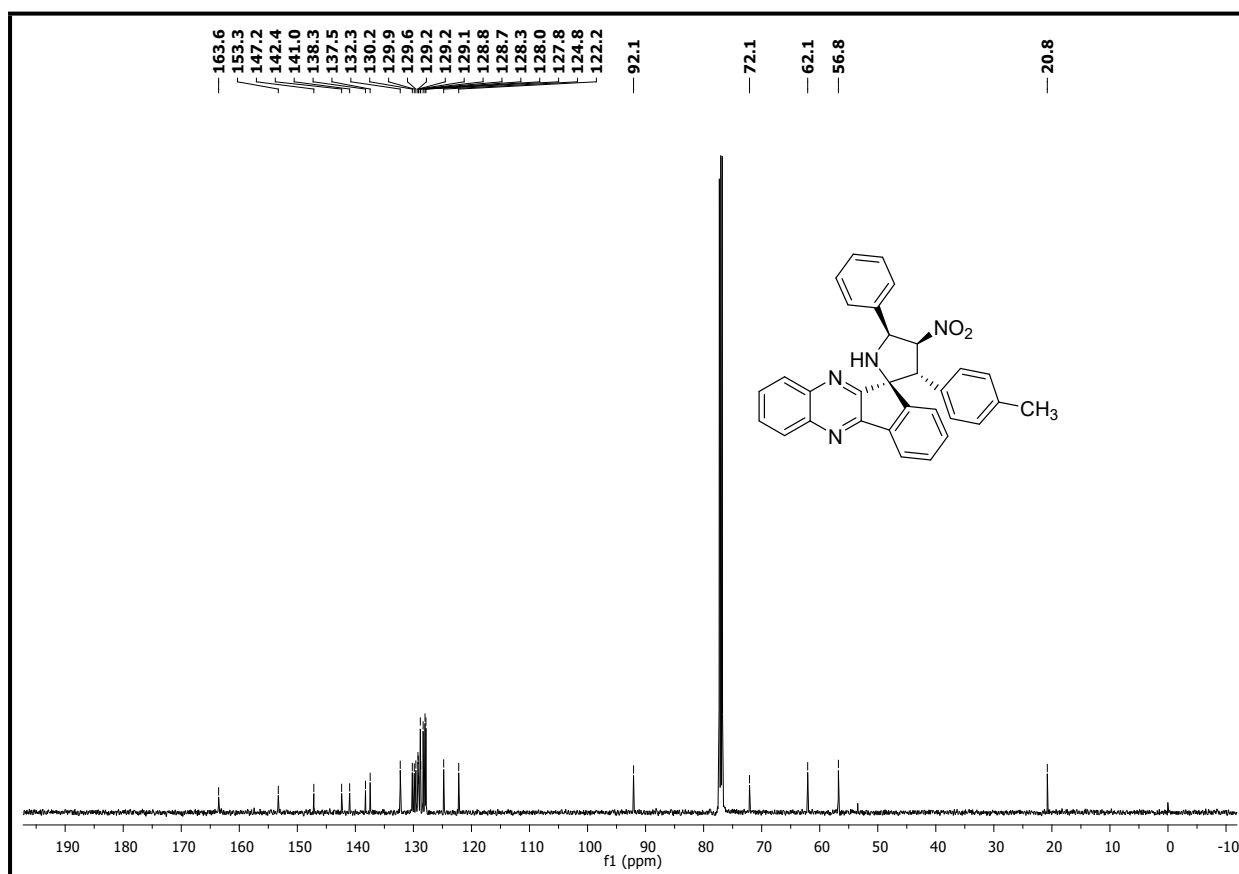


C:\Users\admin\Documents\Bruker\OPUS\_7.5.18\DATA\MEAS\RAMESH B.15 RAMESH B PMG-ME-1 3/28/2017

### <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>, 500 MHz) of compound 5b



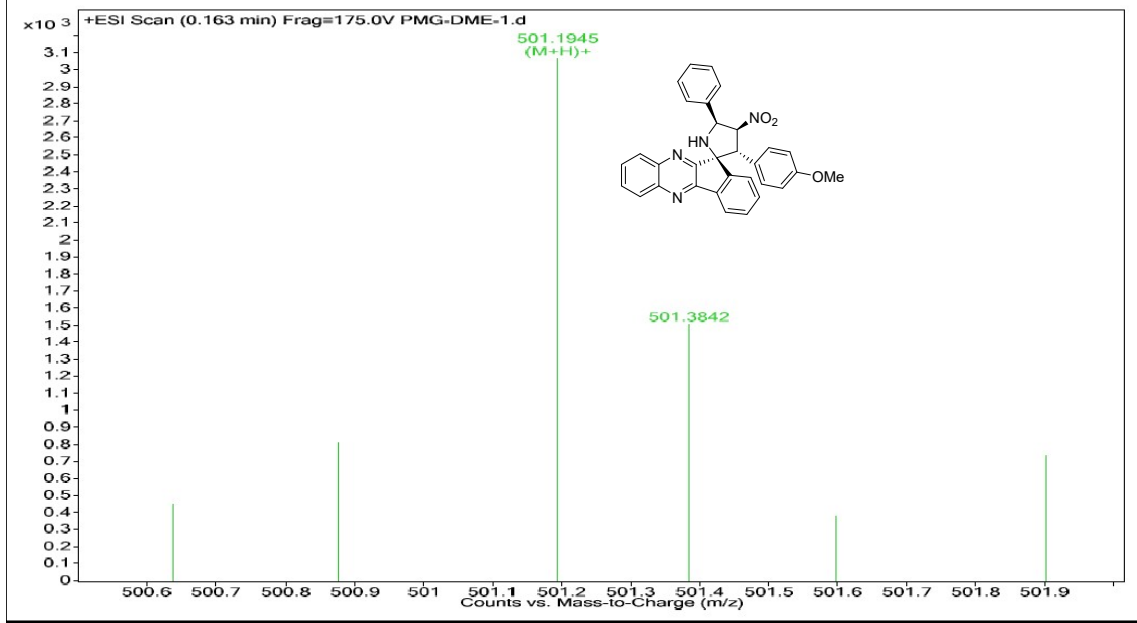
### <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>, 125 MHz) of compound 5b





## HR-ESIM Spectrum of compound 5c

Sample Name	PMG-DME-1	Position	Vial 50	Instrument Name	Instrument 1	User Name	
Inj Vol	-1	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	PMG-DME-1.d	ACQ Method	SCS....m	Comment		Acquired Time	12/5/2016 3:33:49 P



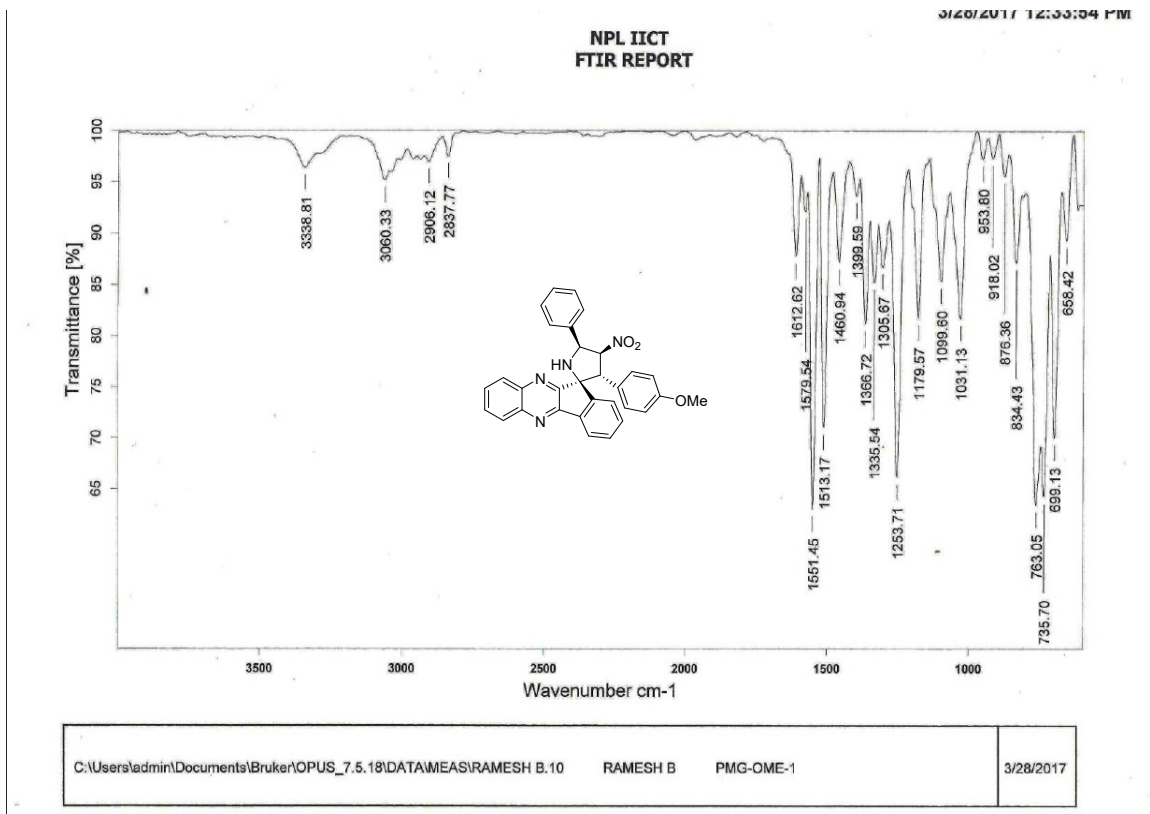
MS Formula Results: + Scan (0.163 min) (PMG-DME-1.d)

m/z	Ion	Formula	Abundance
501.1945	(M+H) <sup>+</sup>	C <sub>31</sub> H <sub>25</sub> N <sub>4</sub> O <sub>3</sub>	3065.7

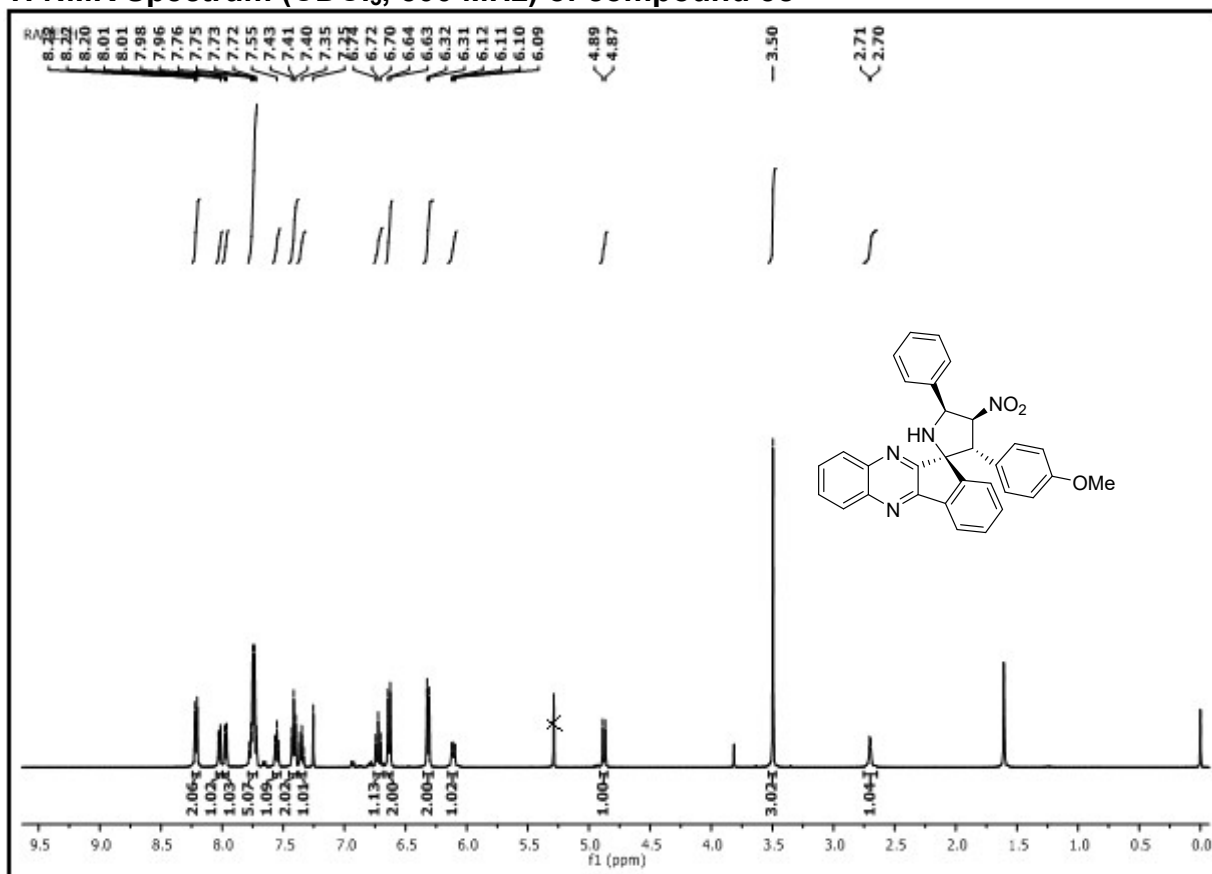
  

Best	Formula (M)	Ion Formula	Calc m/z	Score	Cross Score	Mass	Calc Mass	Diff (ppm)	Abs Diff (ppm)	Abund Match	Spacing Match	Mass Match	m/z	DBE
✓	C <sub>31</sub> H <sub>24</sub> N <sub>4</sub> O <sub>3</sub>	C <sub>31</sub> H <sub>25</sub> N <sub>4</sub> O <sub>3</sub>	501.1921	82.06		500.1873	500.1848	-5.01	5.01	65.75	29.41	76.18	501.1945	22

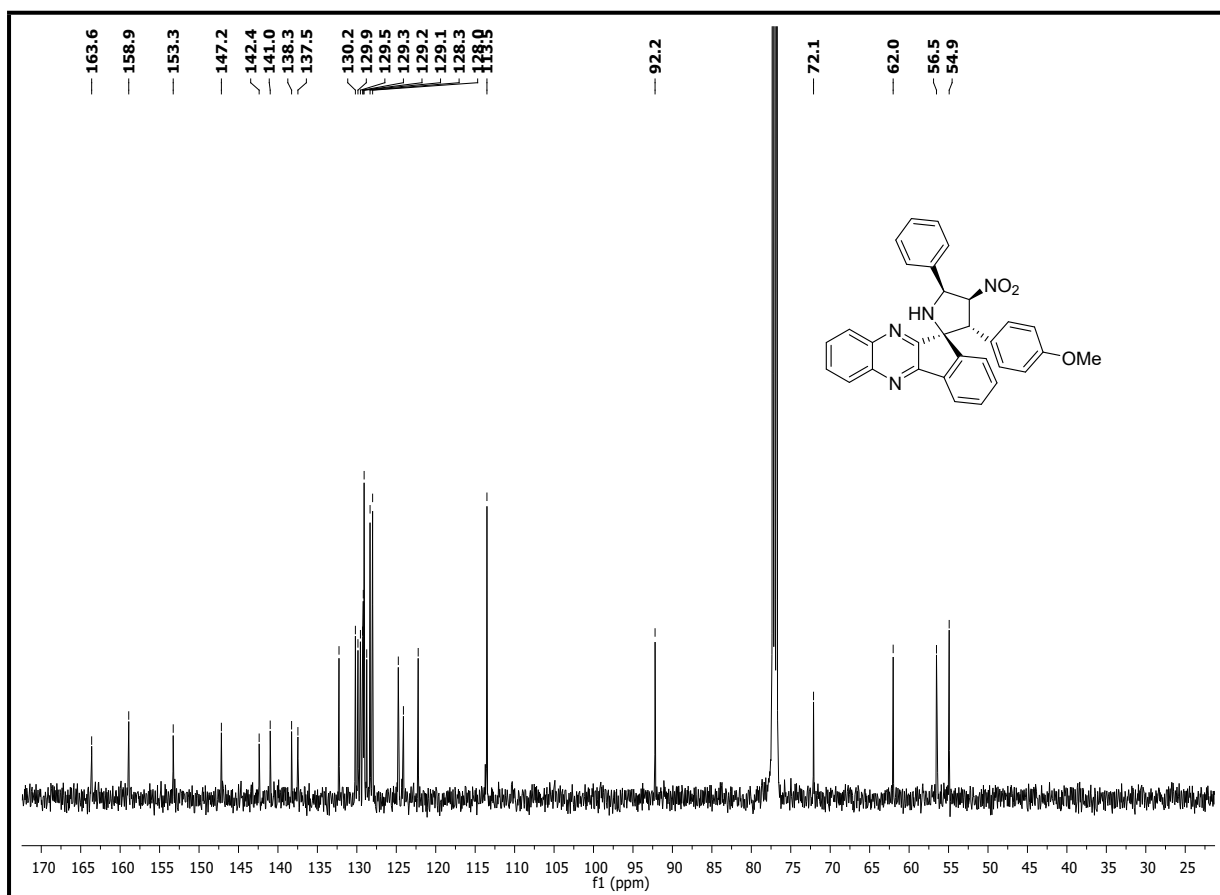
## IR Spectrum of compound 5c



**<sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>, 500 MHz) of compound 5c**

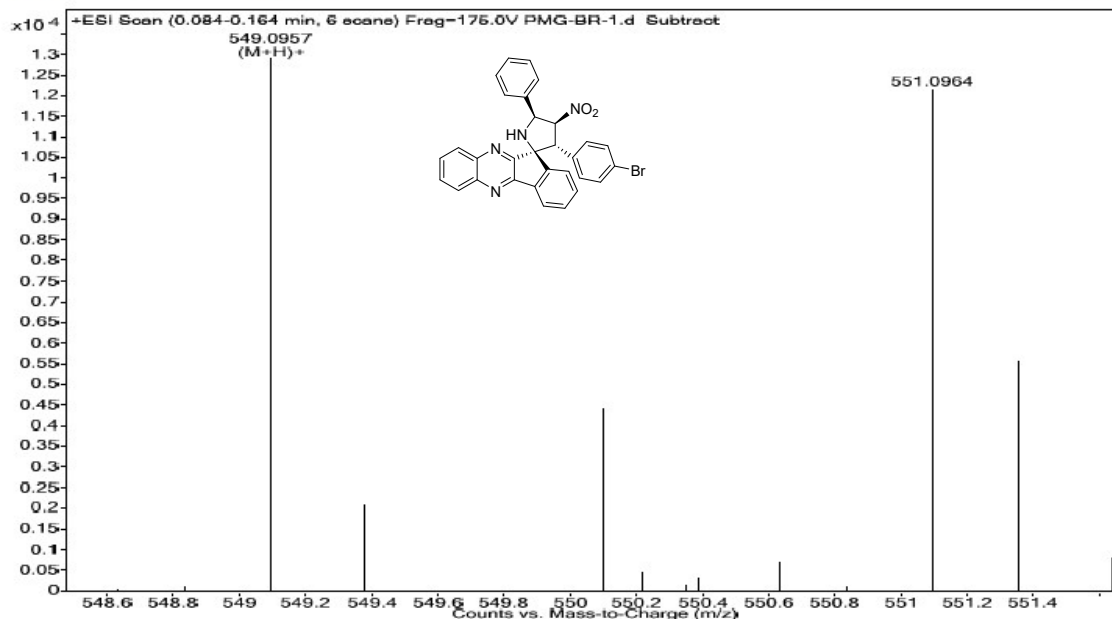


**<sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>, 125 MHz) of compound 5c**



## HR-ESIM Spectrum of compound 5d

**Sample Name** PMG-BR-1      **Position** Vial 11      **Instrument Name** Instrument 1      **User Name**  
**Inj Vol** 0.5      **InjPosition**      **SampleType** Sample      **IRM Calibration Status** Success  
**Data Filename** PMG-BR-1.d      **ACQ Method**      **Comment**      **Acquired Time** 12/2/2016 4:22:33 PM



MS Formula Results: + Scan (0.084-0.164 min) Sub - PMG-BR-1.d (PMG-BR-1.d)

m/z	Ion	Formula	Abundance
549.0957	(M+H) <sup>+</sup>	C <sub>30</sub> H <sub>22</sub> BrN <sub>4</sub> O <sub>2</sub>	12879.7

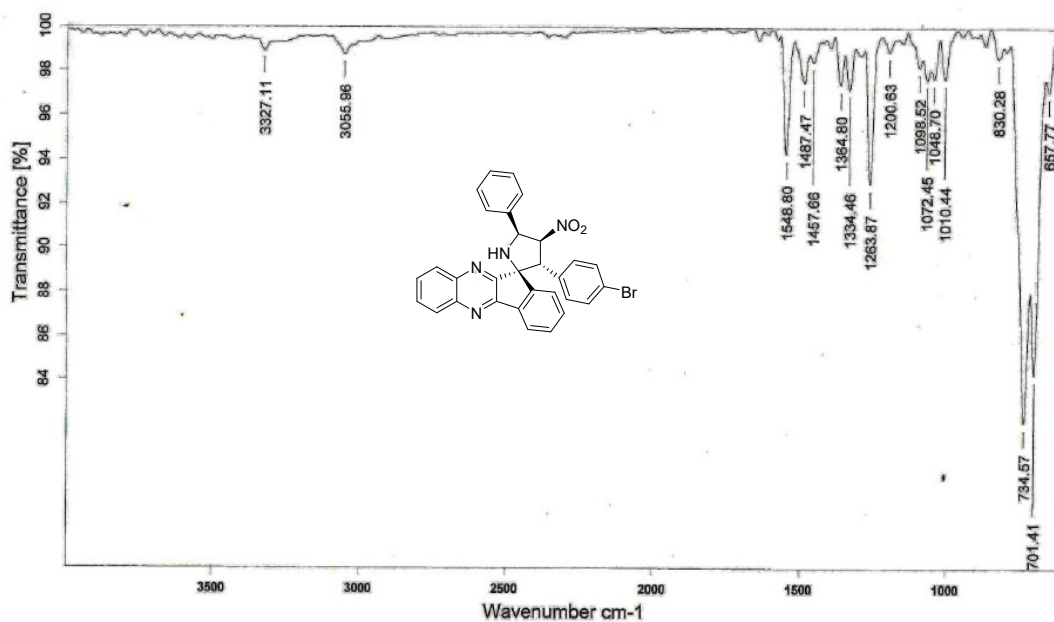
  

Best	Formula (M)	Ion Formula	Calc m/z	Score	Cross Score	Mass	Calc Mass	Diff (ppm)	Abn Diff (ppm)	Abund Match	Spacing Match	Mass Match	m/z	DBE
1	C <sub>30</sub> H <sub>21</sub> BrN <sub>4</sub> O <sub>2</sub>	C <sub>30</sub> H <sub>22</sub> BrN <sub>4</sub> O <sub>2</sub>	549.0921	99.97		549.0929	549.0940	-9.91	9.91	99.03	81.41	42.11	549.0957	22

## IR Spectrum of compound 5d

3/28/2017 12:22:26 PM

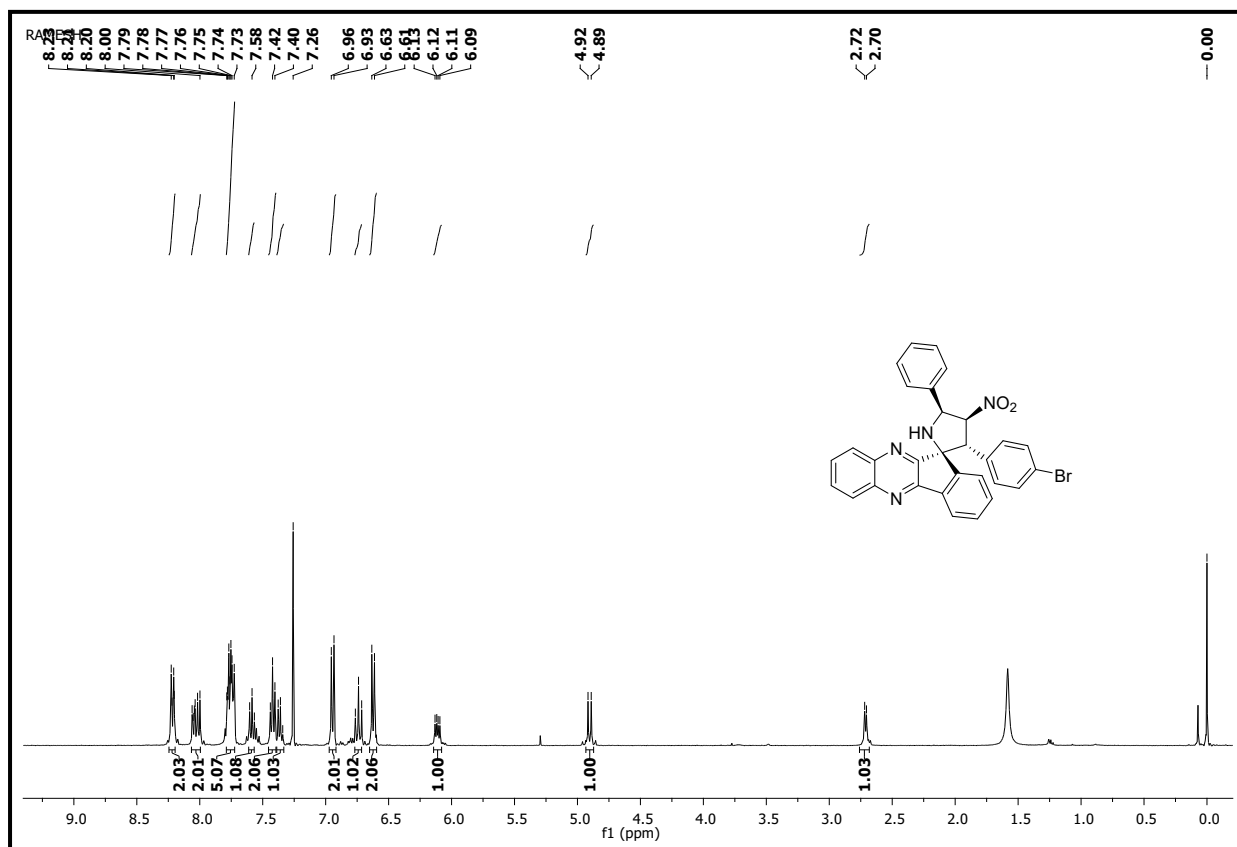
**NPL IICT  
FTIR REPORT**



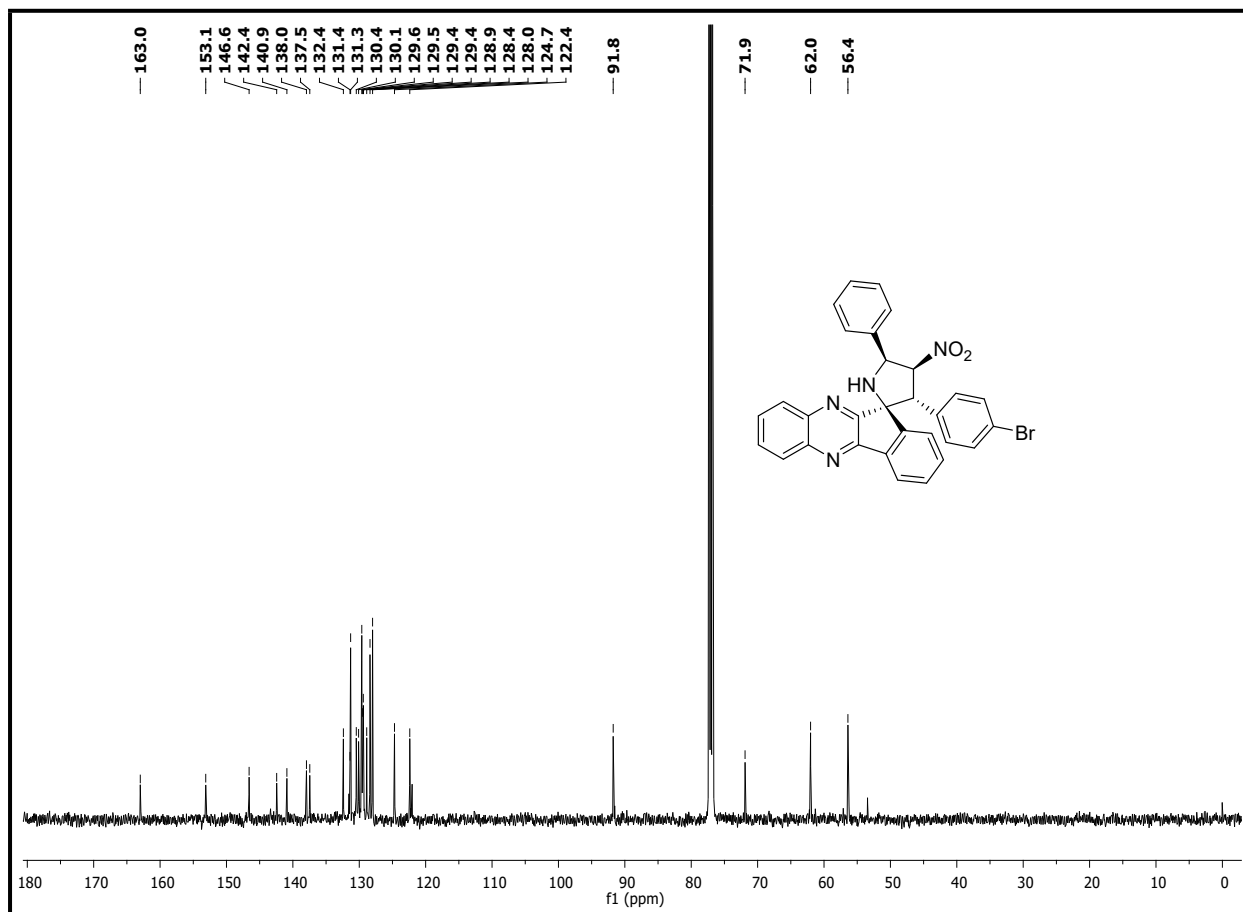
C:\Users\admin\Documents\Bruker\OPUS\_7.5.18\DATA\MEAS\RAMESH B.4      RAMESH B      PMG-BR1

3/28/2017

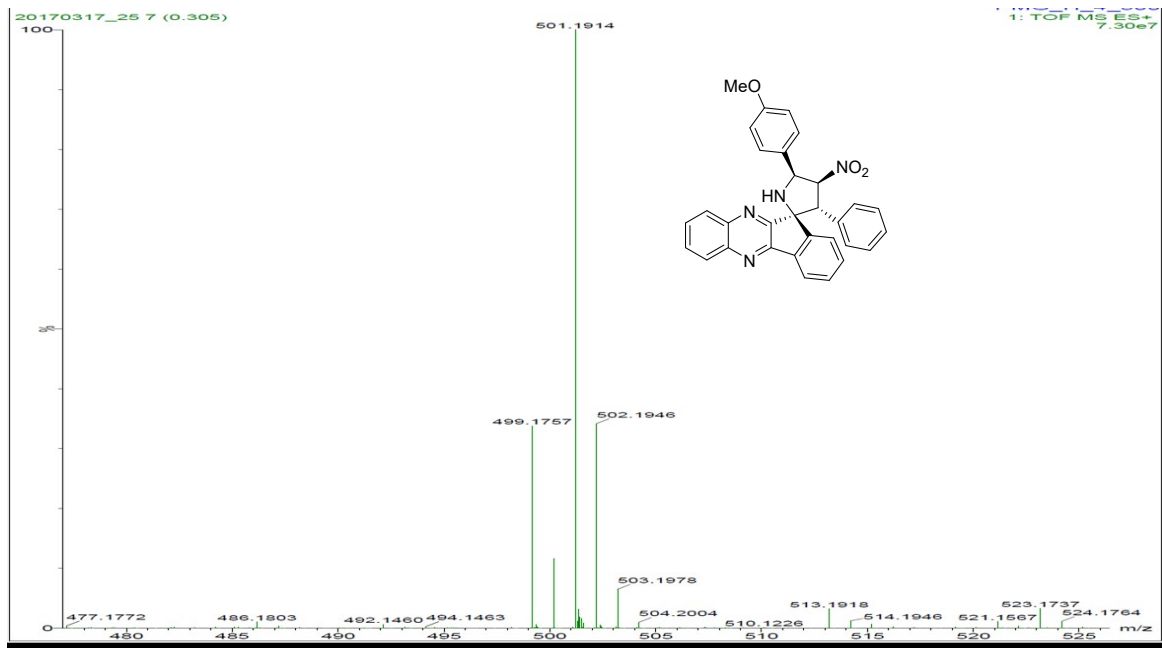
### <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>, 400 MHz) of compound 5d



### <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>, 100 MHz) of compound 5d



## HR-ESIM Spectrum of compound 5e



21 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

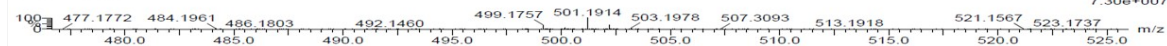
C: 0-31 H: 0-25 N: 0-4 O: 0-3 Na: 0-1

20170317\_25 7 (0.305)

PMG\_H\_4\_500

1: TOF MS ES+

7.30e+07



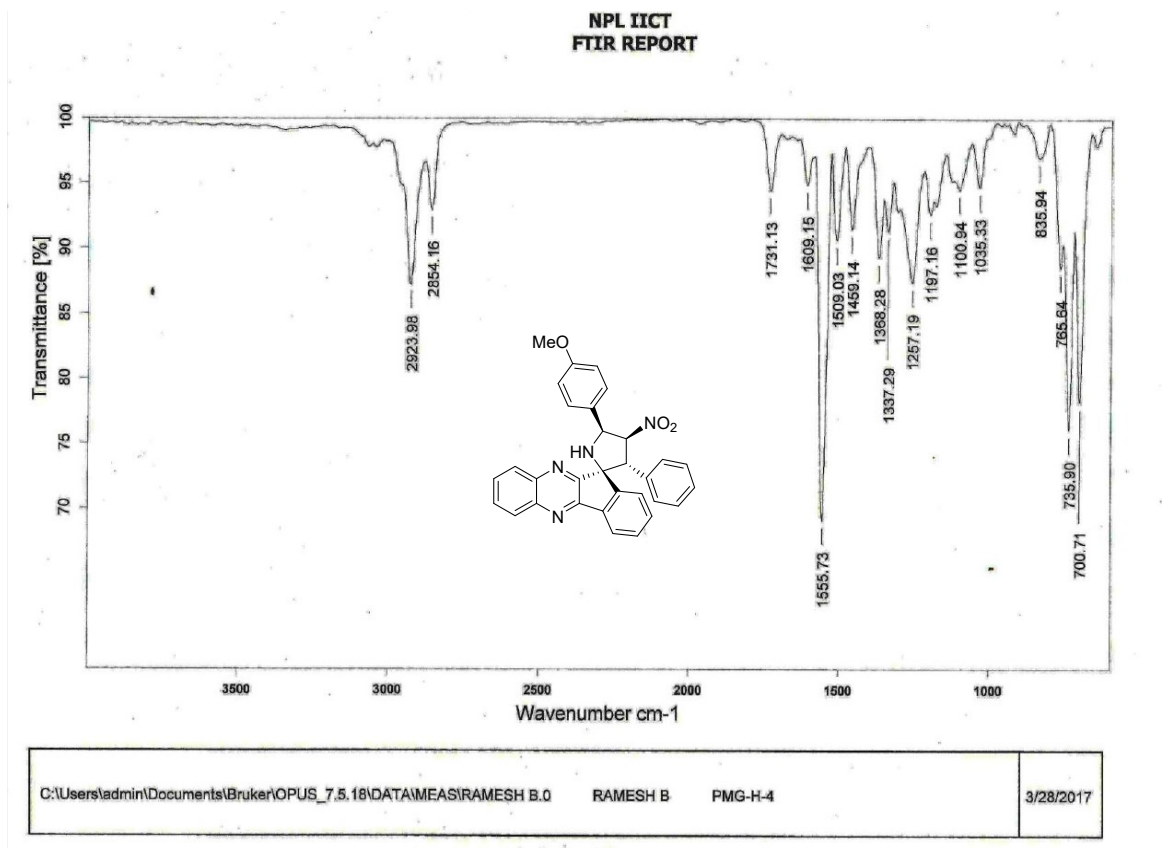
Minimum:

Maximum:

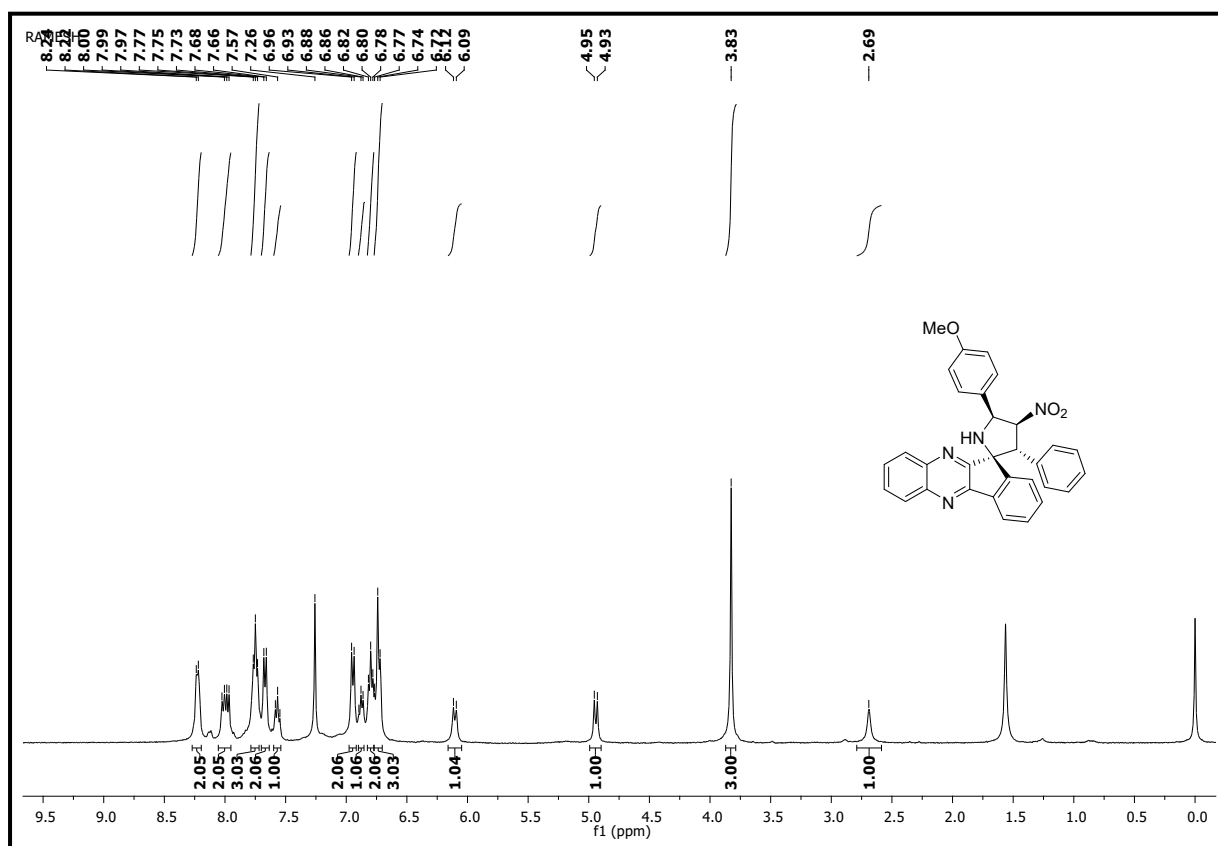
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
501.1914	501.1927	-1.3	-2.6	21.5	491.9	n/a	n/a	C31 H25 N4 O3

501.1914 501.1927 -1.3 -2.6 21.5 491.9 n/a n/a C31 H25 N4 O3

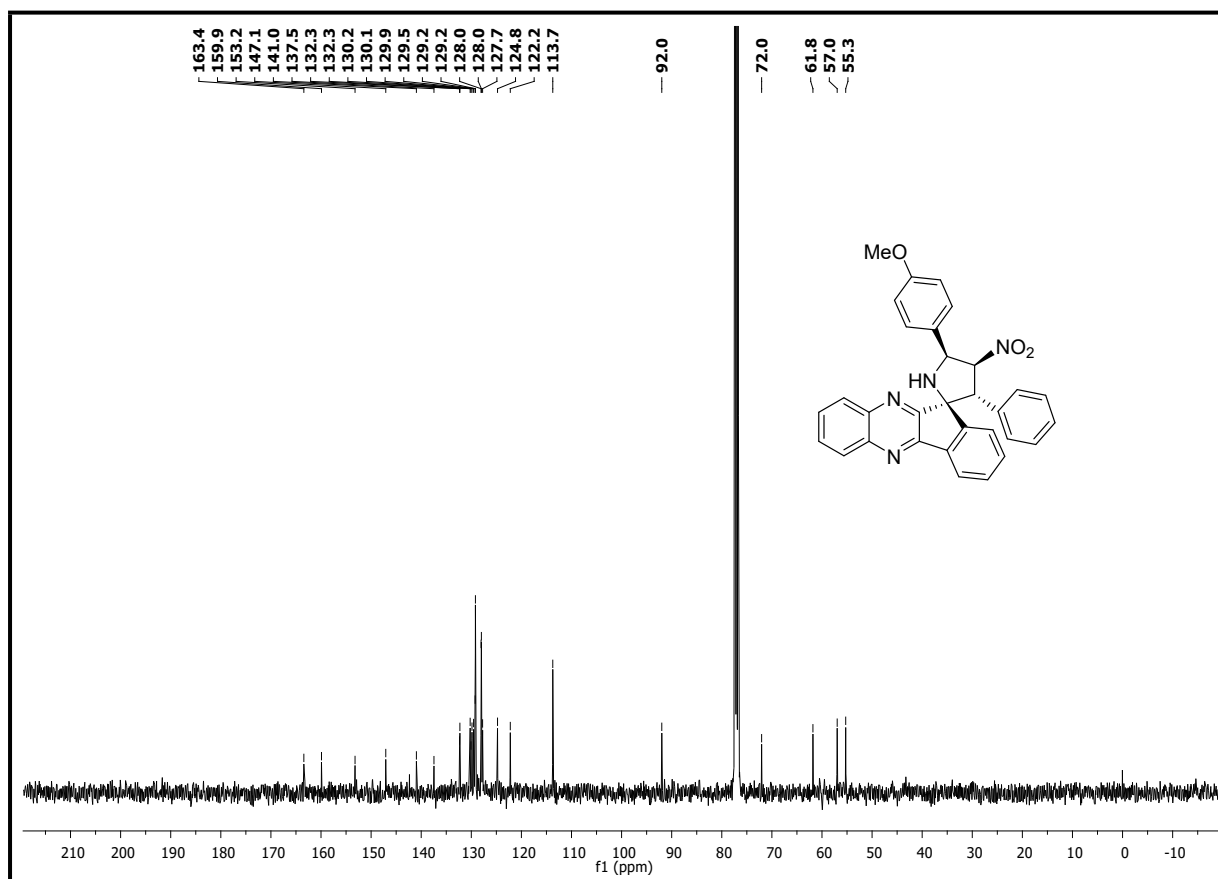
## IR Spectrum of compound 5e



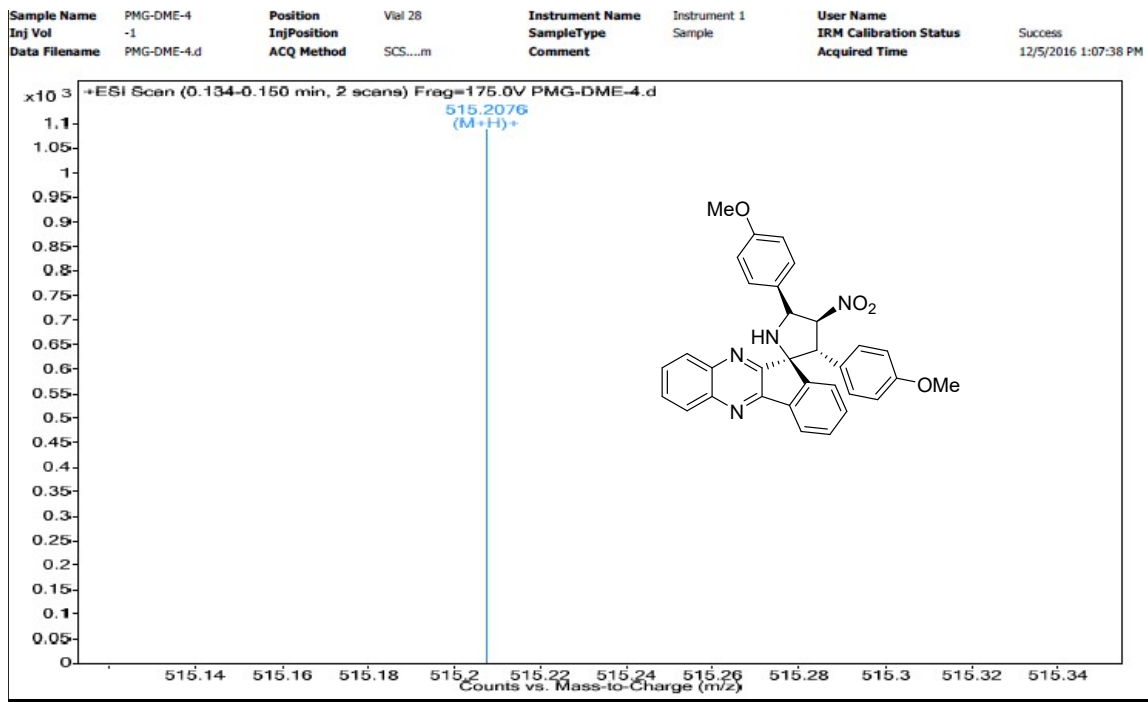
### <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>, 400 MHz) of compound 5e



### <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>, 100 MHz) of compound 5e



## HR-ESIM Spectrum of compound 5f



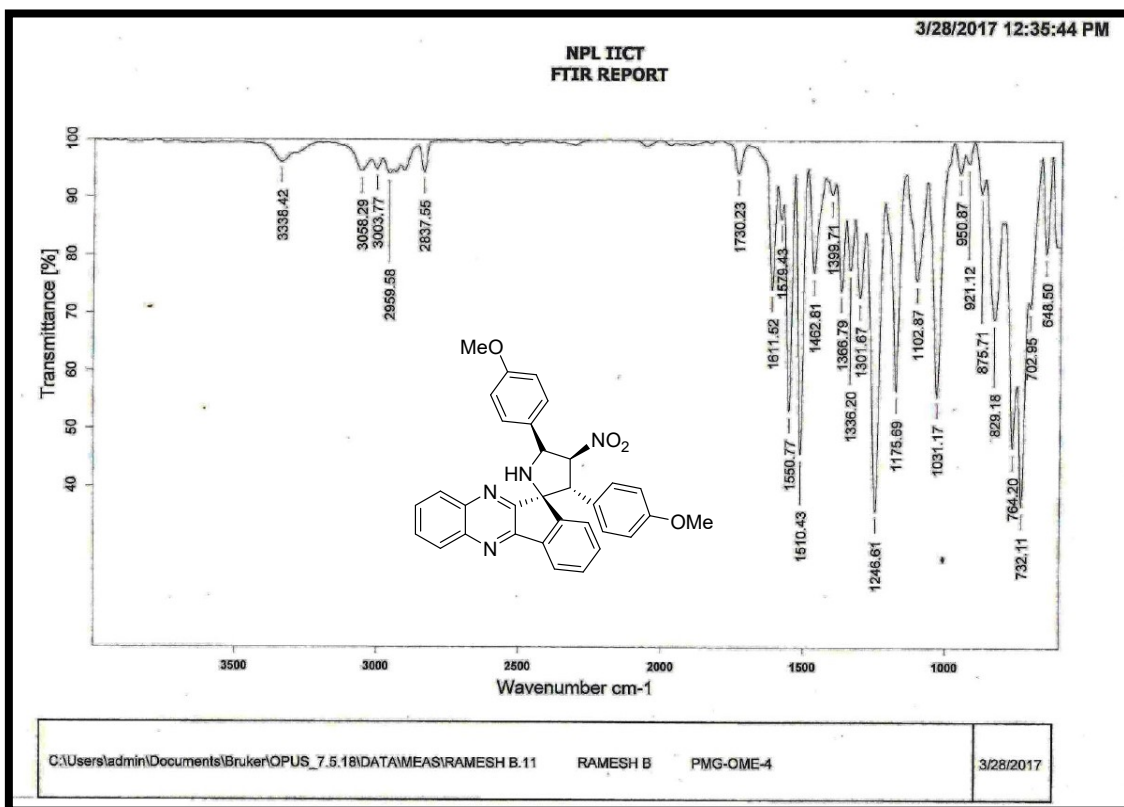
MS Formula Results: + Scan (0.134-0.150 min) (PMG-DME-4.d)

m/z	Ion	Formula	Abundance
515.2076	(M+H) <sup>+</sup>	C <sub>32</sub> H <sub>27</sub> N <sub>4</sub> O <sub>3</sub>	1086.9

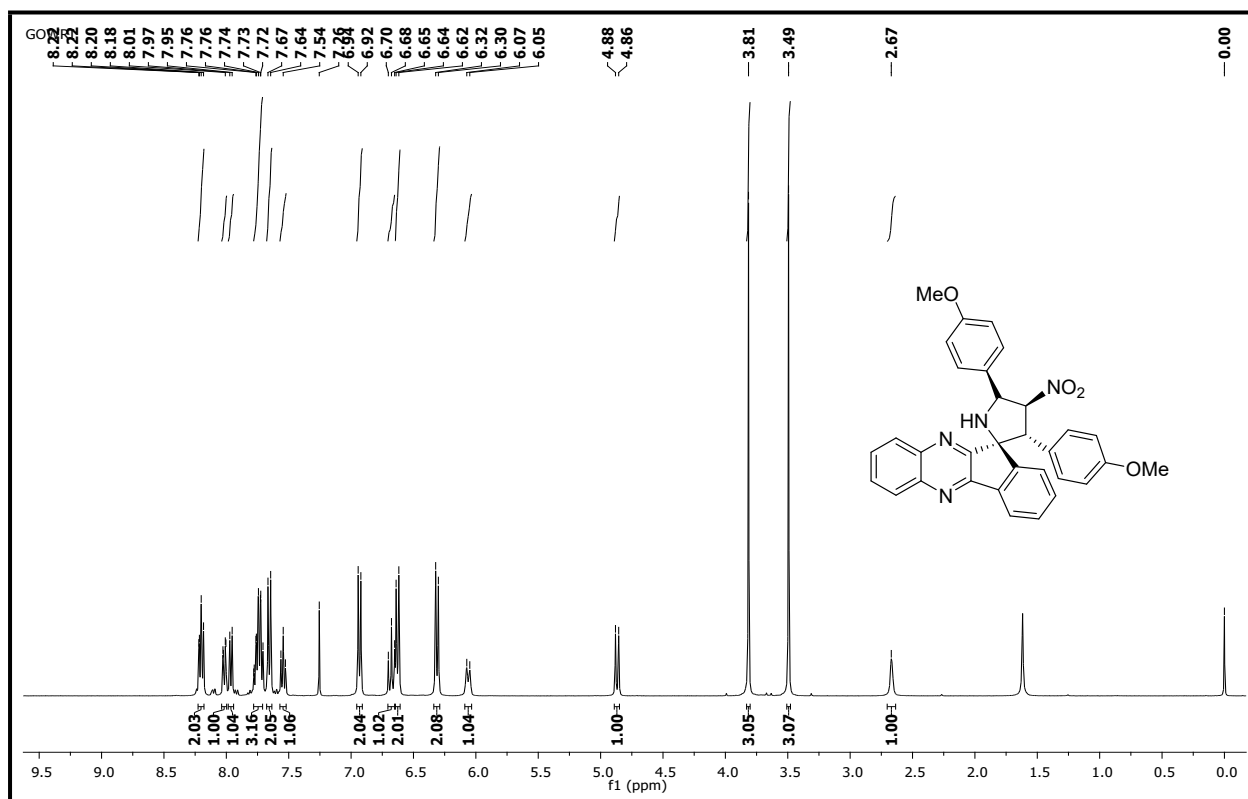
  

Best	Formula (M)	Ion Formula	Calc. m/z	Score	Cross Score	Mass	Calc. Mass	Diff (ppm)	Abs Diff (ppm)	Abund Match	Spacing Match	Mass Match	m/z	DBE
✓	C <sub>32</sub> H <sub>26</sub> N <sub>4</sub> O <sub>3</sub>	C <sub>32</sub> H <sub>27</sub> N <sub>4</sub> O <sub>3</sub>	515.2078	47.53		514.2003	514.2005	0.4	0.4	0	0	99.82	515.2076	22

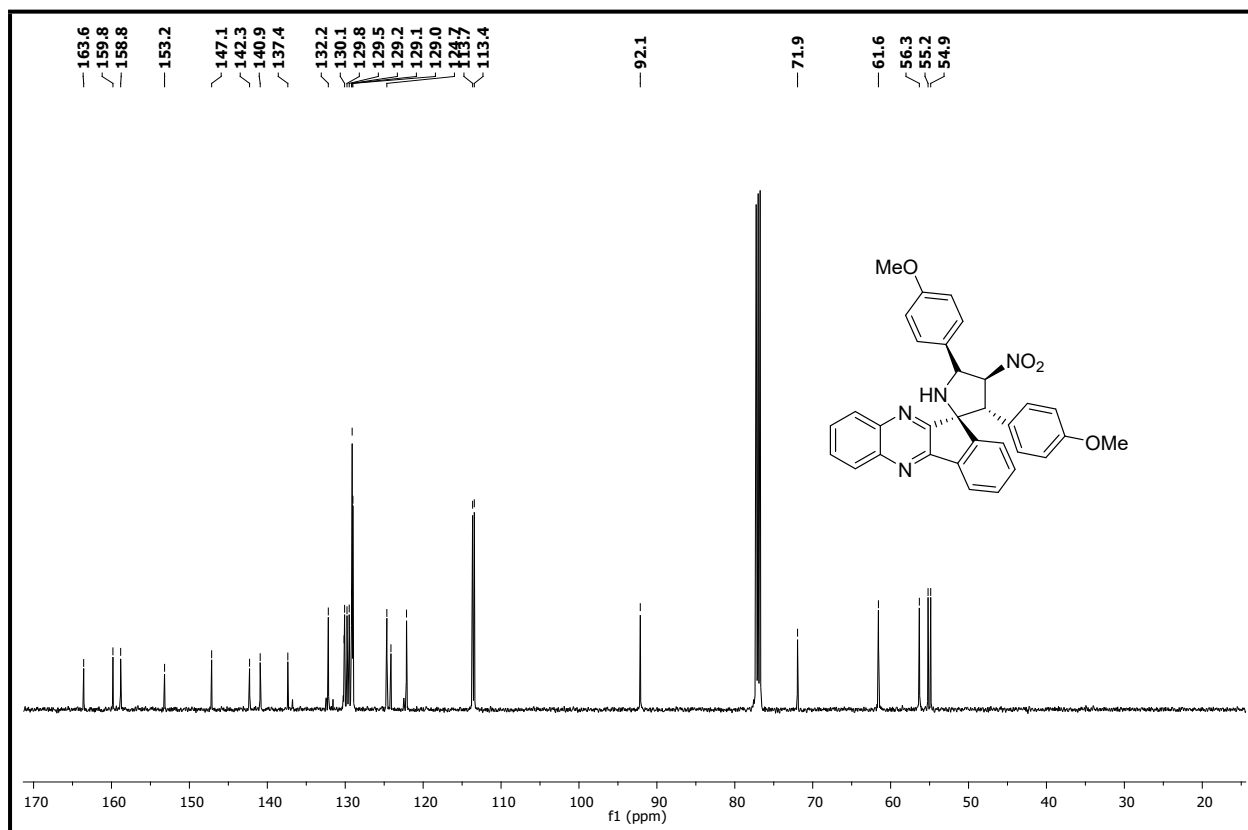
## IR Spectrum of compound 5f



### <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>, 400 MHz) of compound 5f

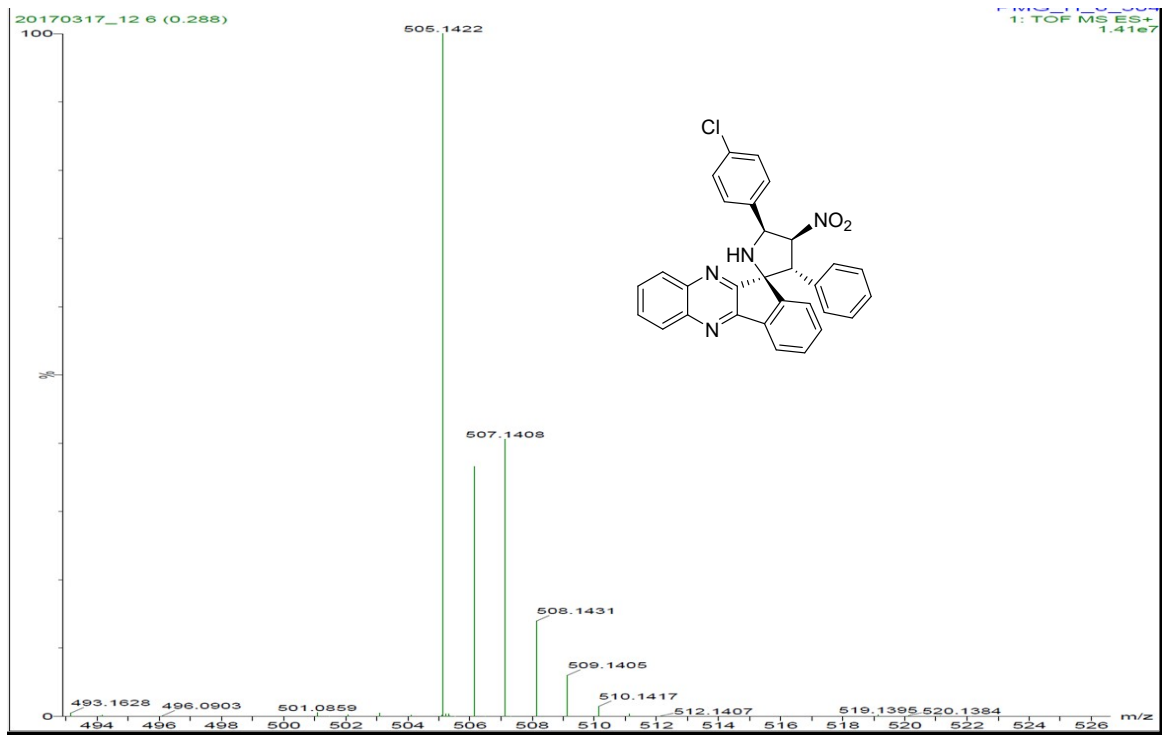


### <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>, 100 MHz) of compound 5f





## HR-ESIM Spectrum of compound 5g



29 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-30 H: 0-22 N: 0-4 O: 0-2 Cl: 0-2

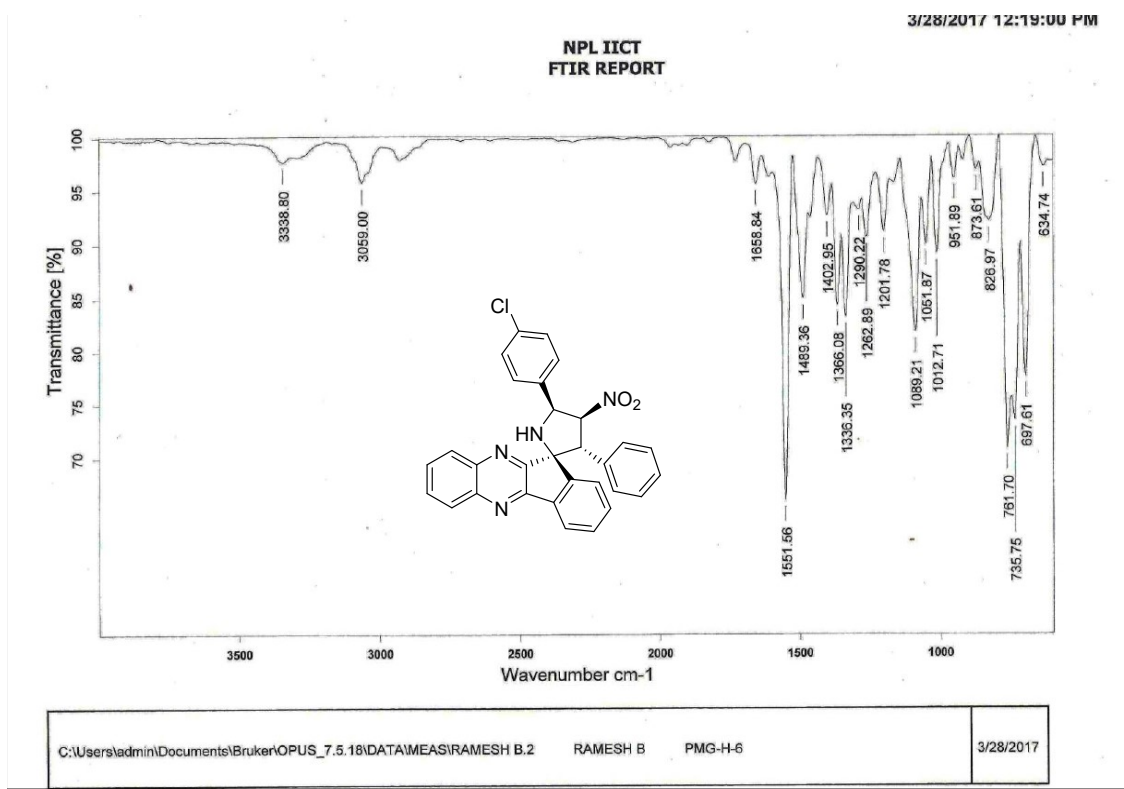
20170317\_12 6 (0.288)

1: TOF MS ES+

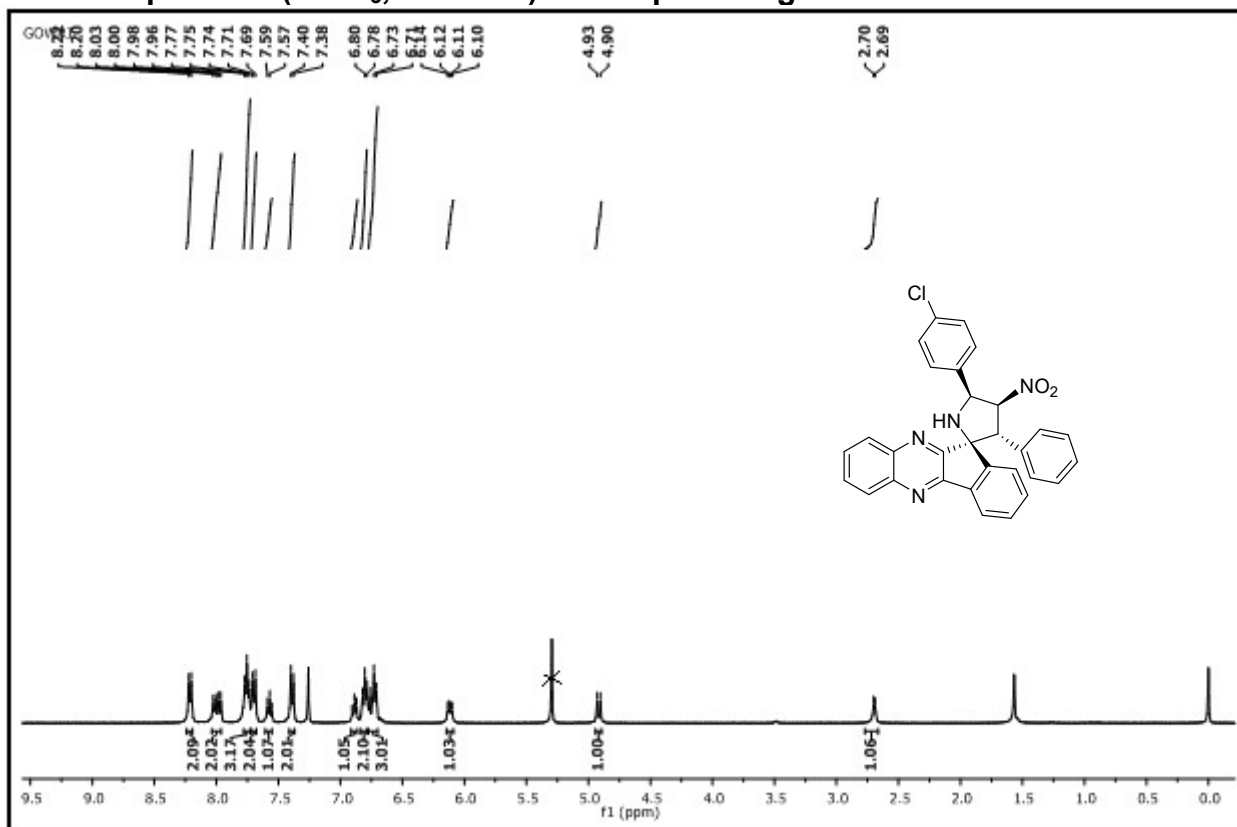
PMG\_H\_6\_504

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
505.1422	505.1431	-0.9	-1.8	31.5	122.2	n/a	n/a	C30 H22 N4 O2 Cl

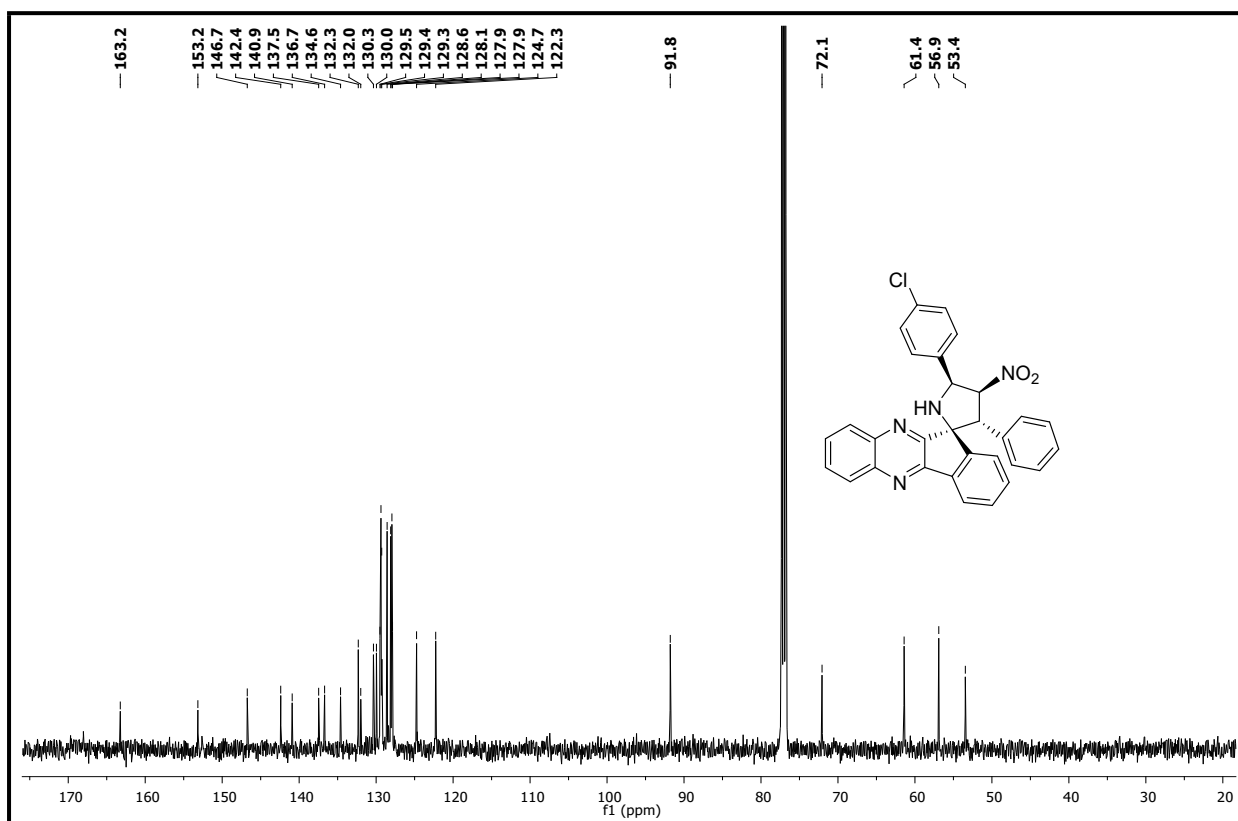
## IR Spectrum of compound 5g



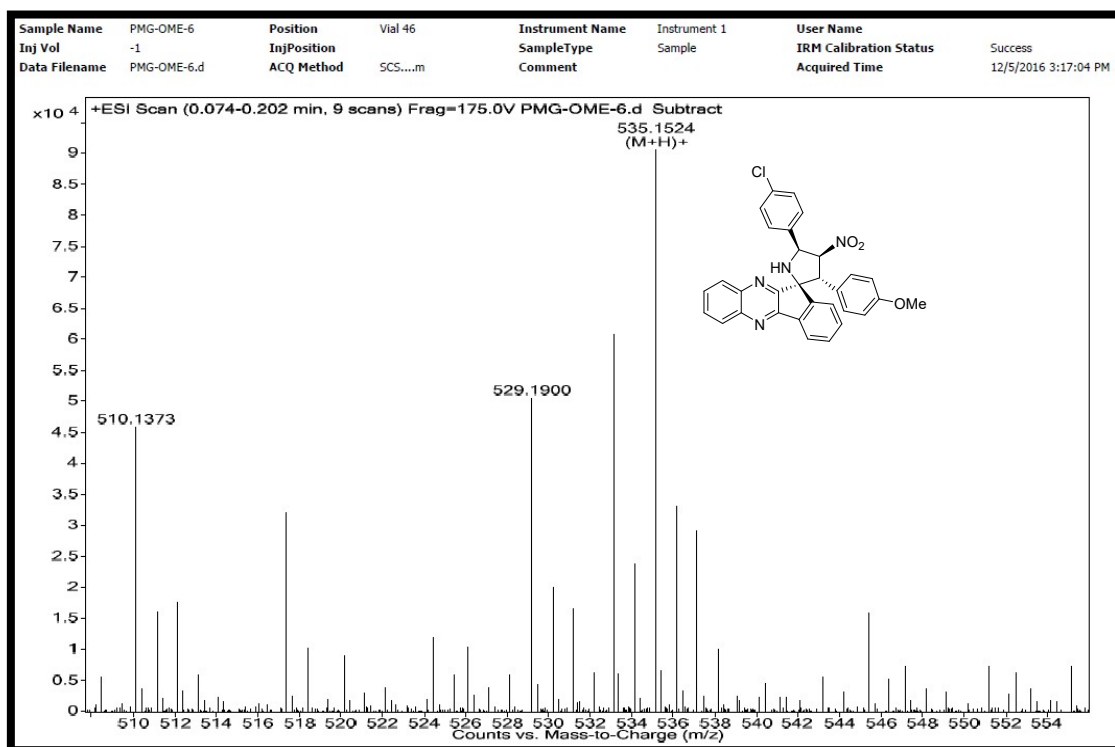
**<sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>, 500 MHz) of compound 5g**



**<sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>, 100 MHz) of compound 5g**



## HR-ESIM Spectrum of compound 5h



MS Formula Results: + Scan (0.074-0.202 min) SUD - PMG-OME-6.d (PMG-OME-6.d)

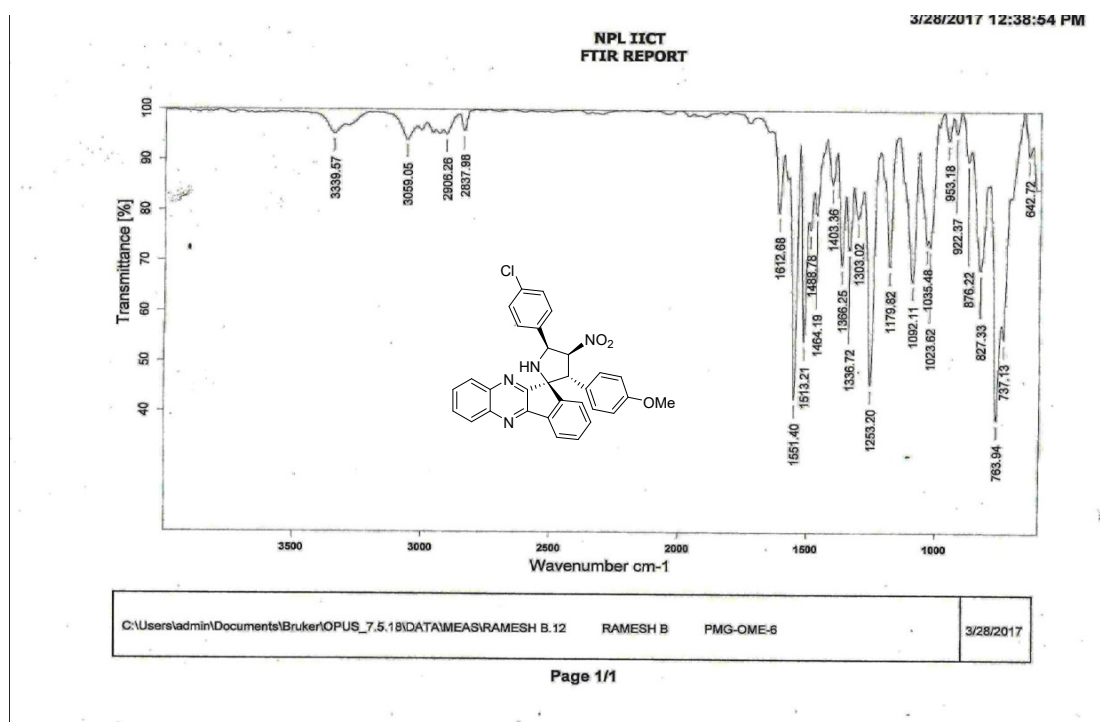
m/z	Ion	Formula	Abundance
535.1524	(M+H)+	C31 H24 Cl N4 O3	90561.3

Best	Formula (M)	Ion Formula	Calc. m/z	Score	Cross Score	Mass	Calc. Mass	Diff (ppm)	Abs Diff (ppm)	Abund Match	Spacing Match	Mass Match	m/z	DBE
<input checked="" type="checkbox"/>	C31 H23 Cl N4 O3	C31 H24 Cl N4 O3	535.1631	76.9		534.1453	534.1459	1.06	1.06	39.16	78.56	98.72	535.1524	22

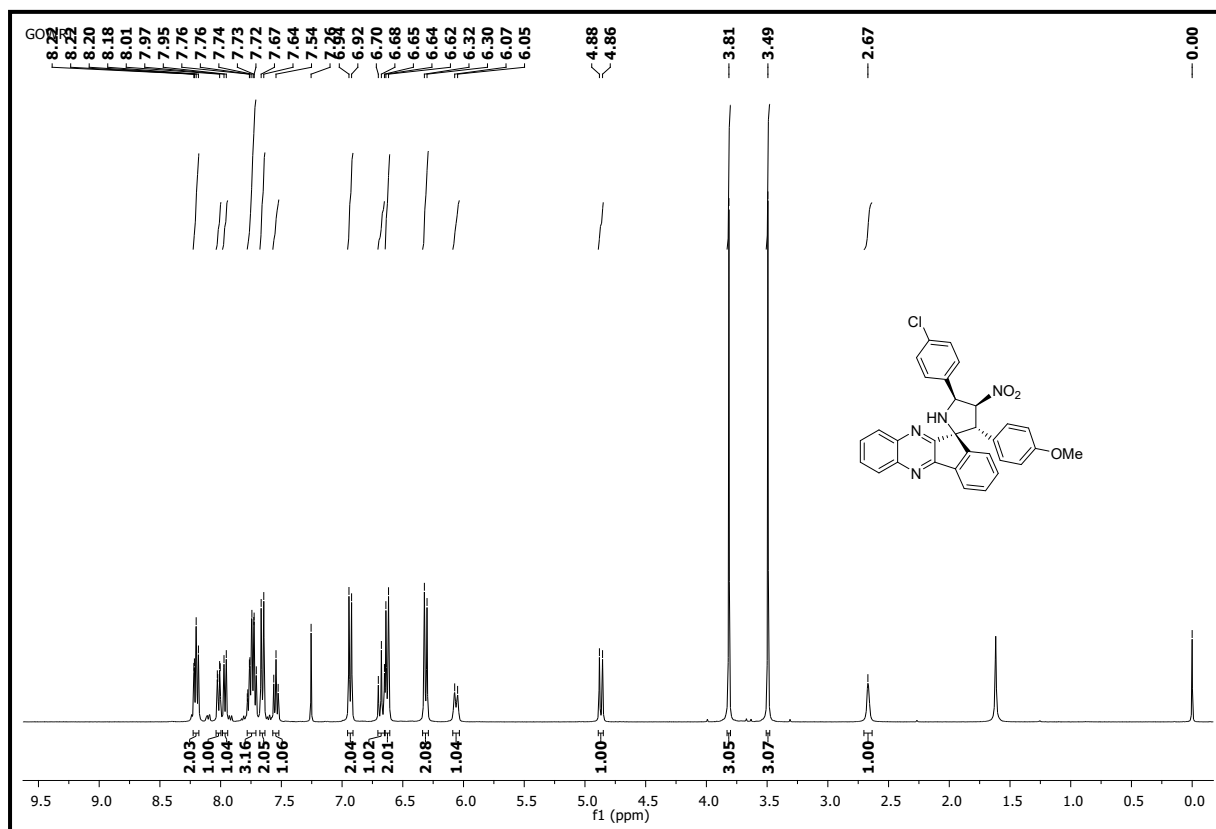
IR

## Spectrum of compound 5h

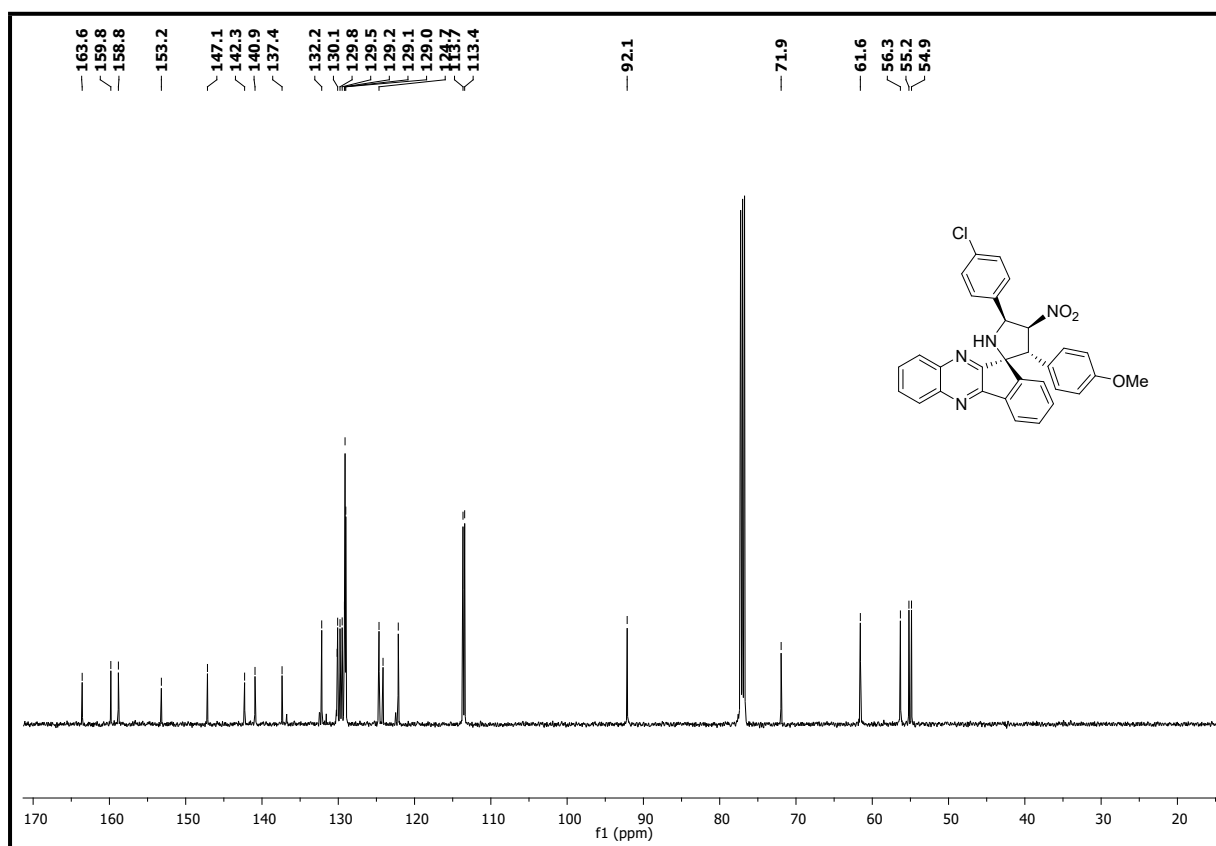


S19

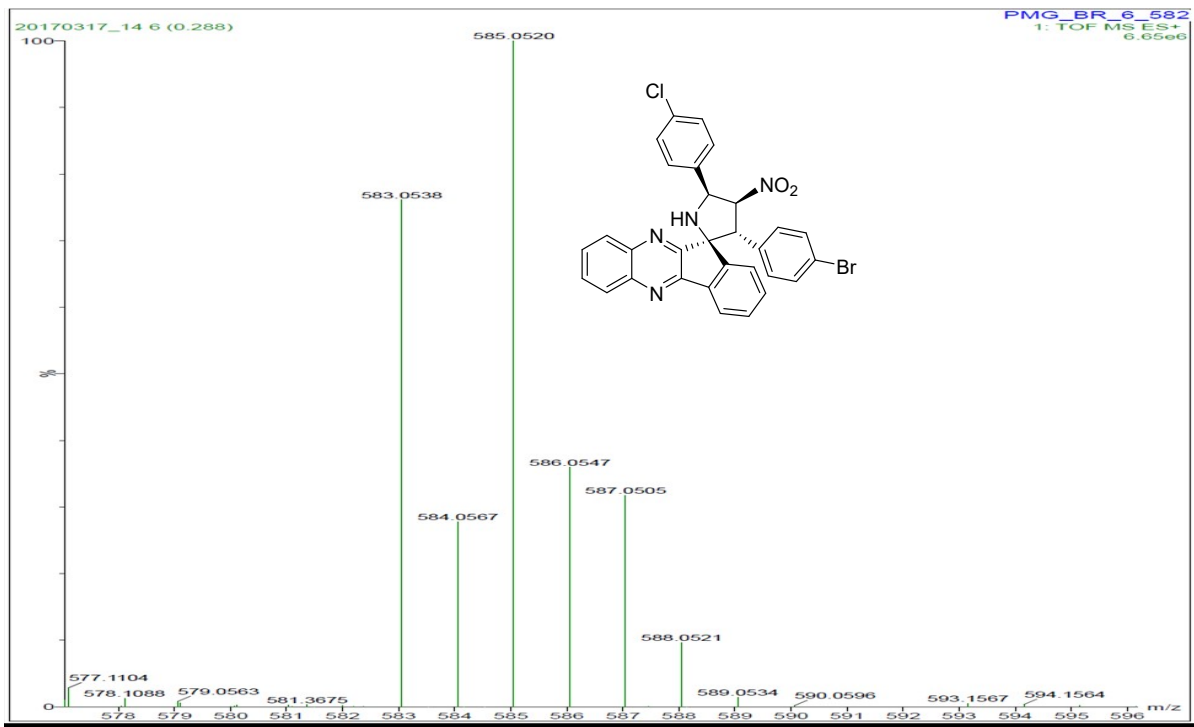
### <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>, 400 MHz) of compound 5h



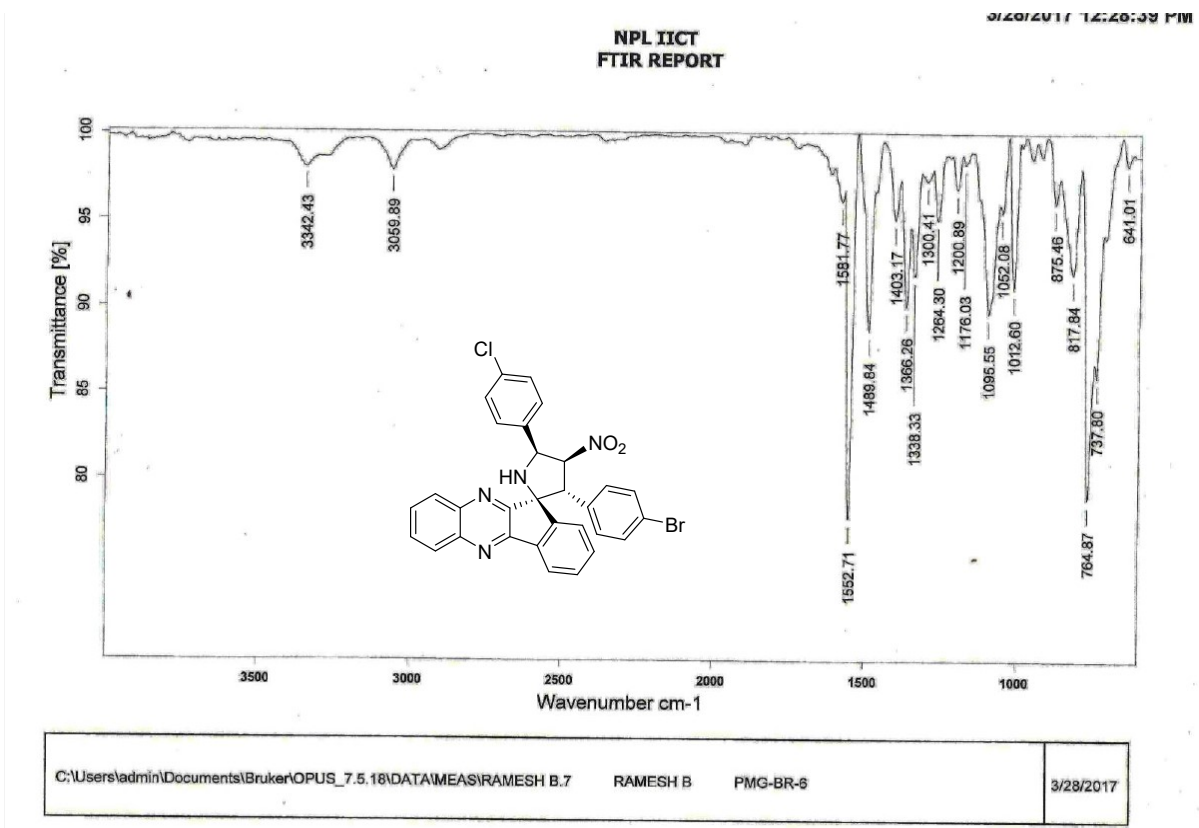
### <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>, 100 MHz) of compound 5h



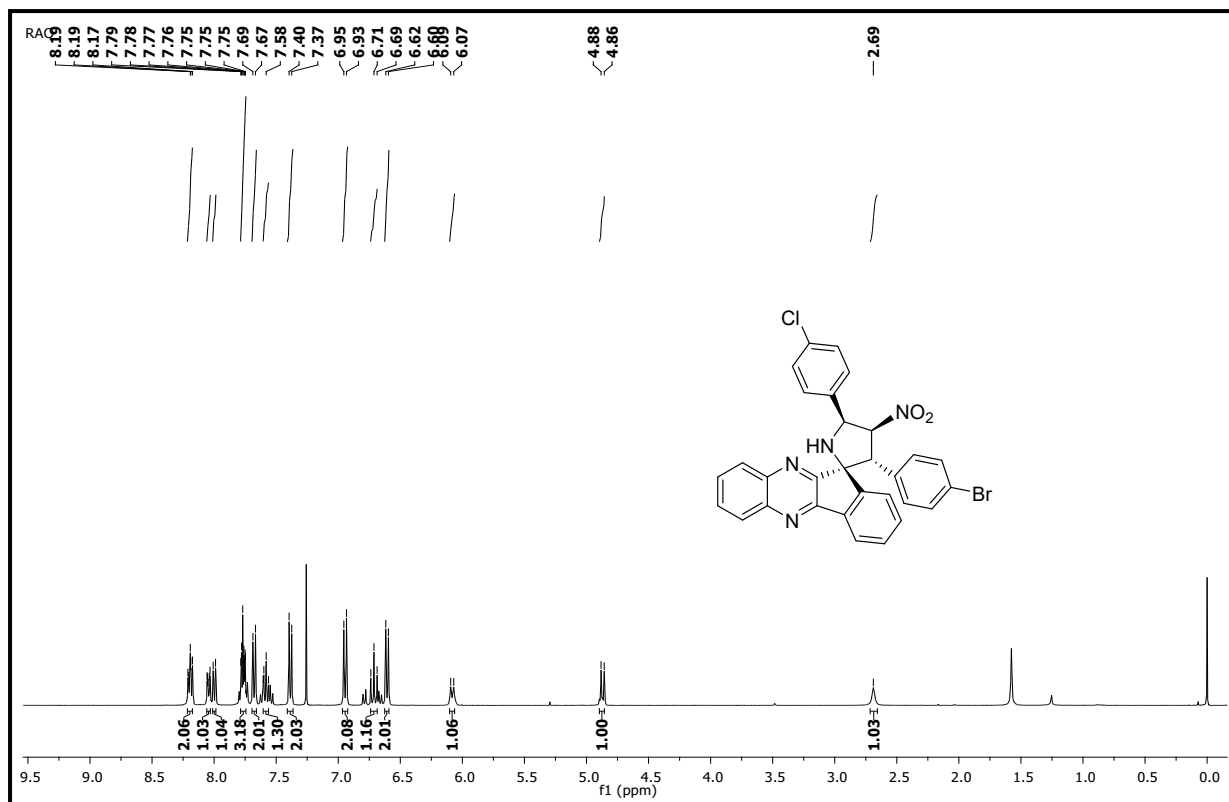
## HR-ESIM Spectrum of compound 5i



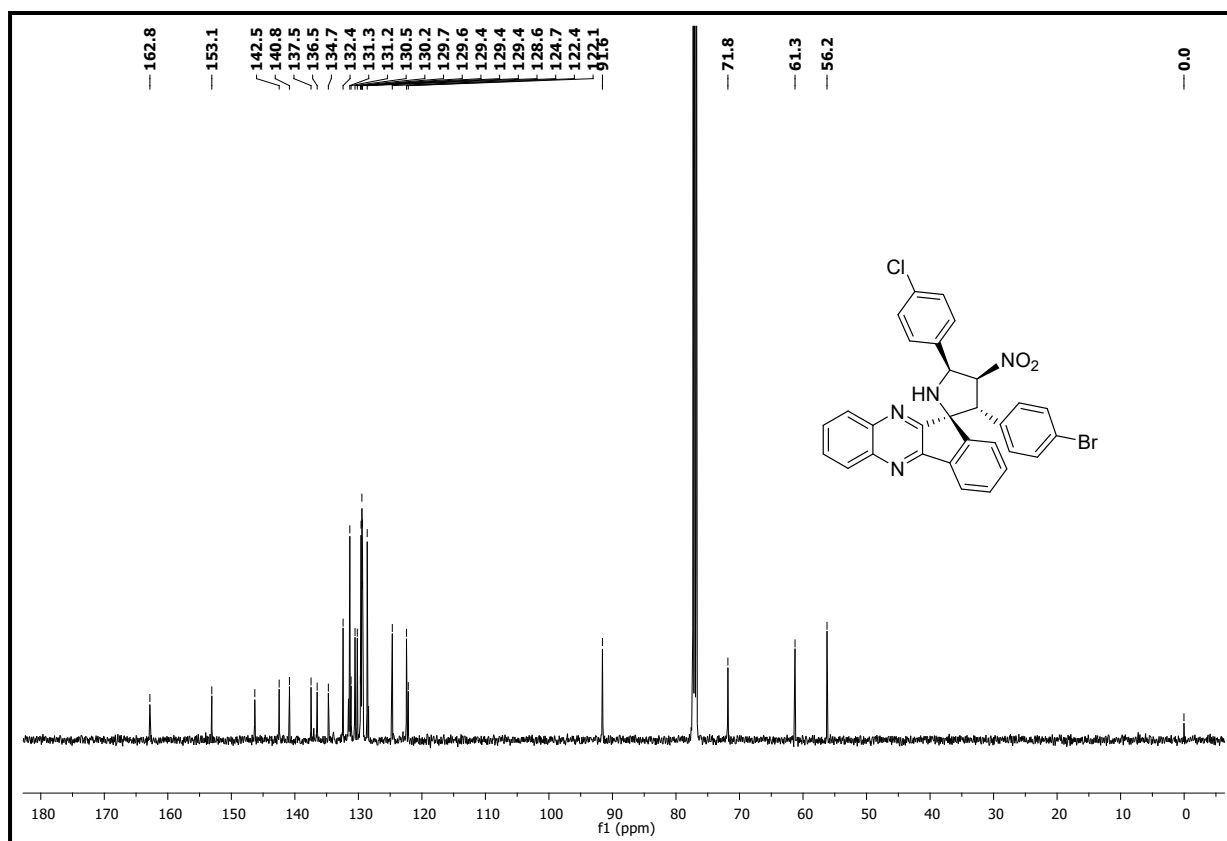
## IR Spectrum of compound 5i



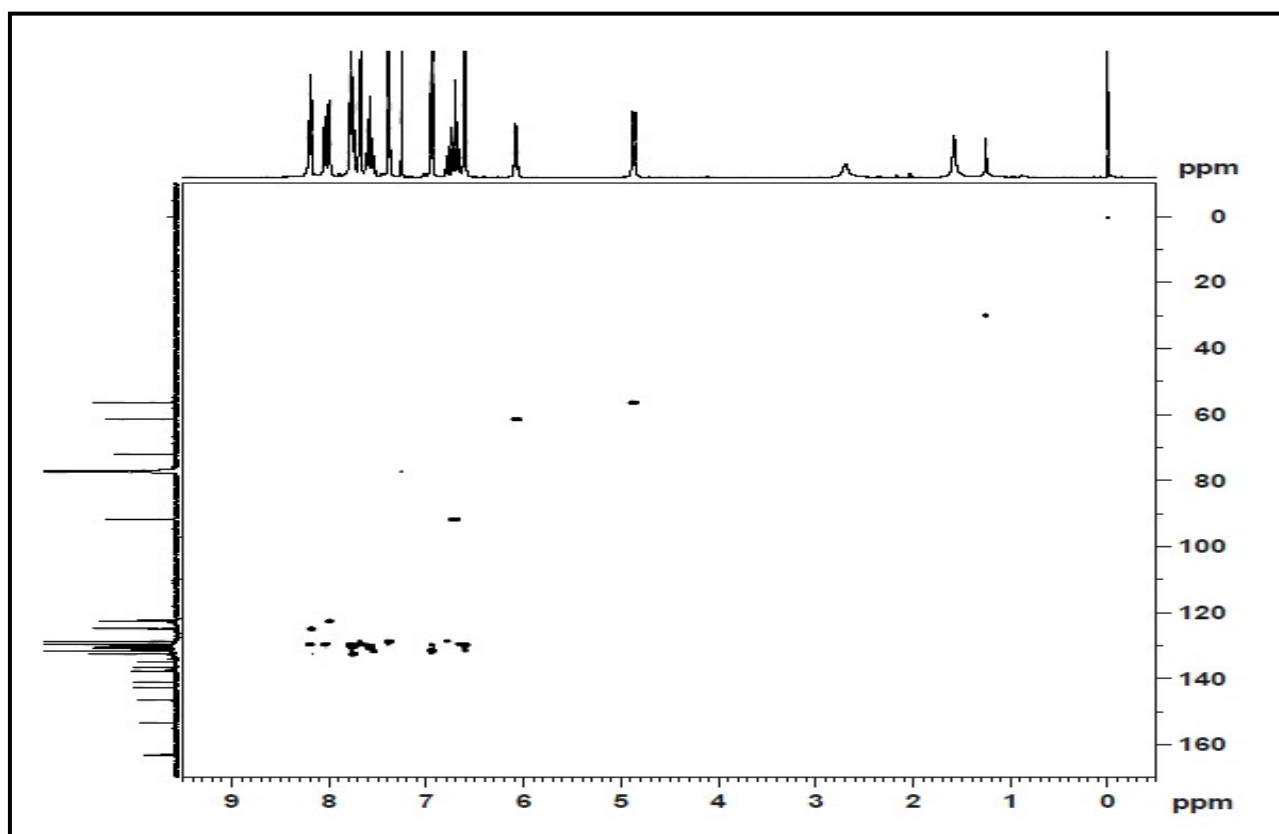
**<sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>, 400 MHz) of compound 5i**



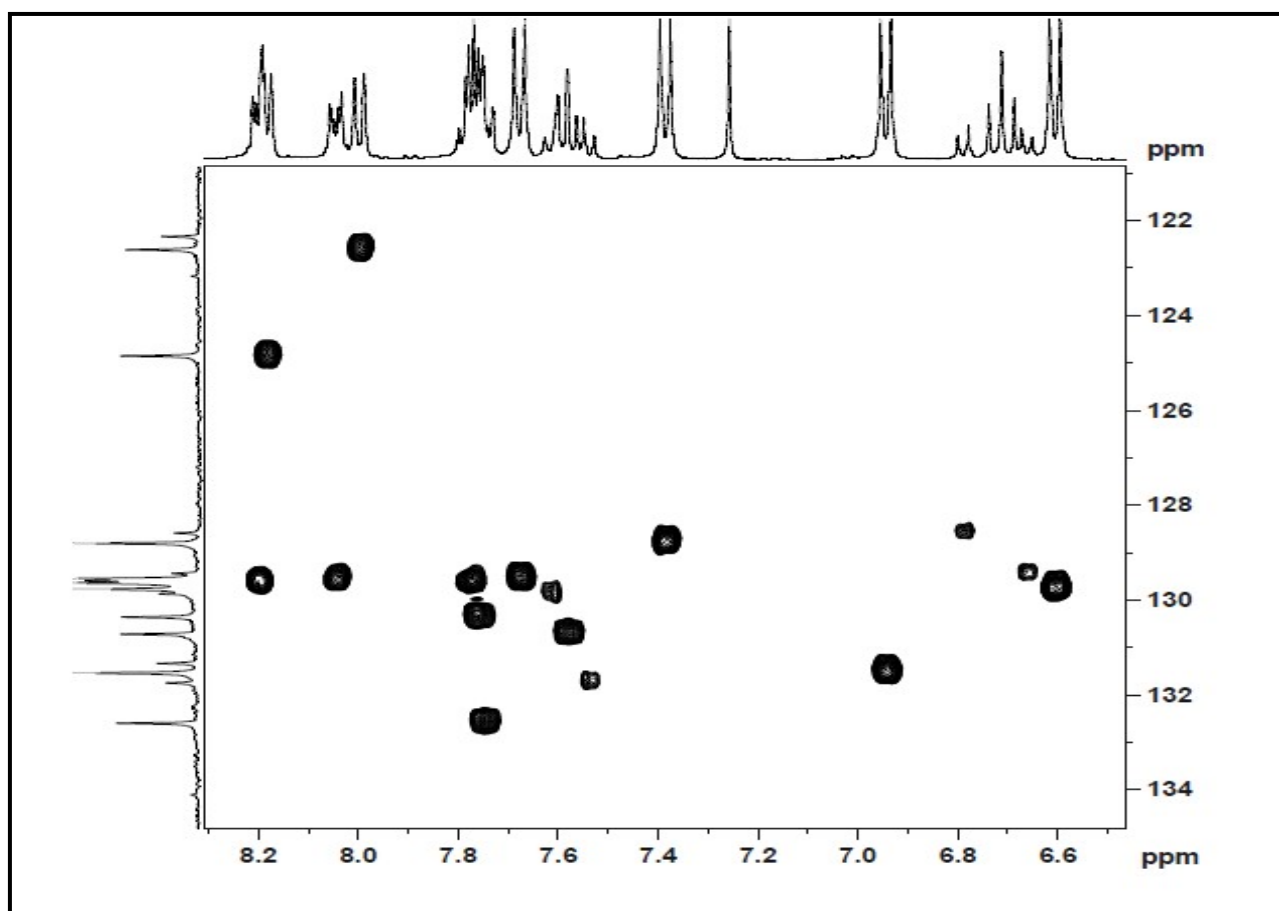
**<sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>, 100 MHz) of compound 5i**



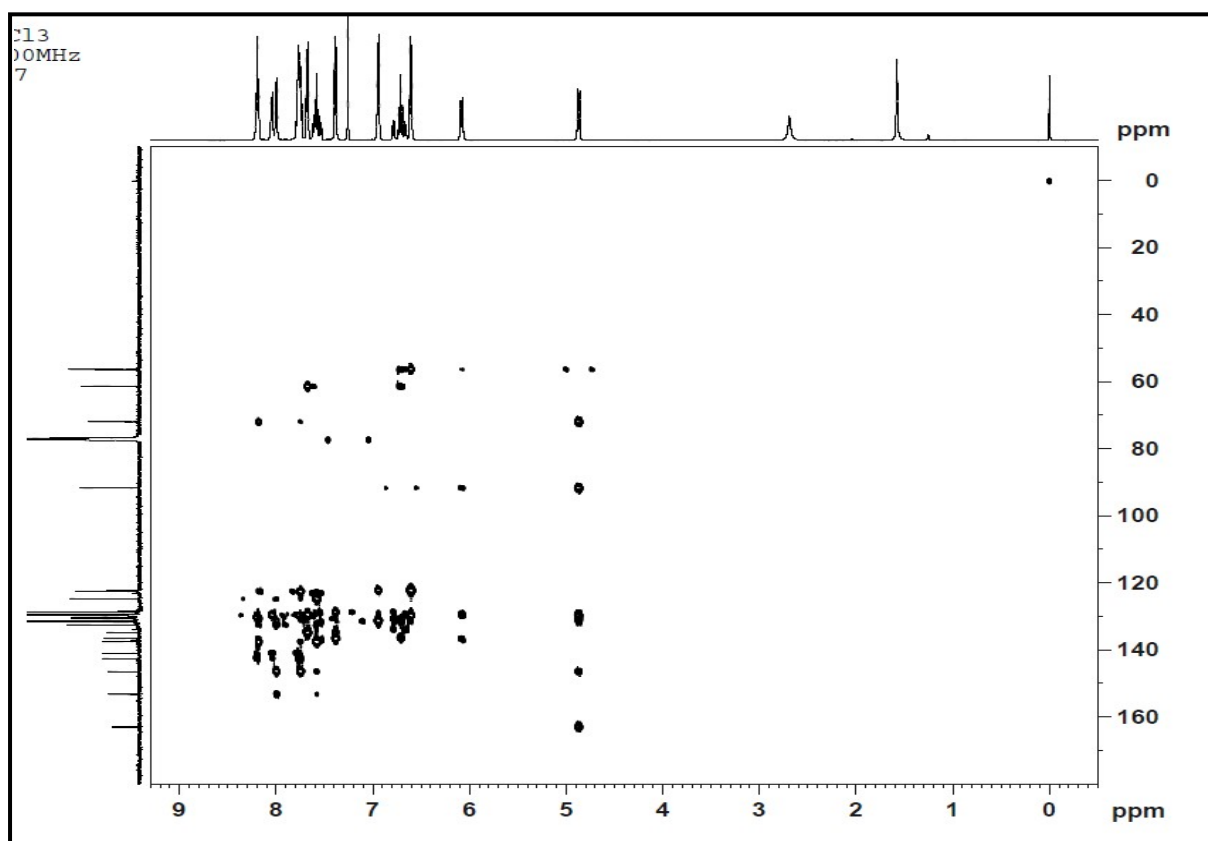
HSQC NMR Spectrum (CDCl<sub>3</sub>, 400 MHz) of compound 5i



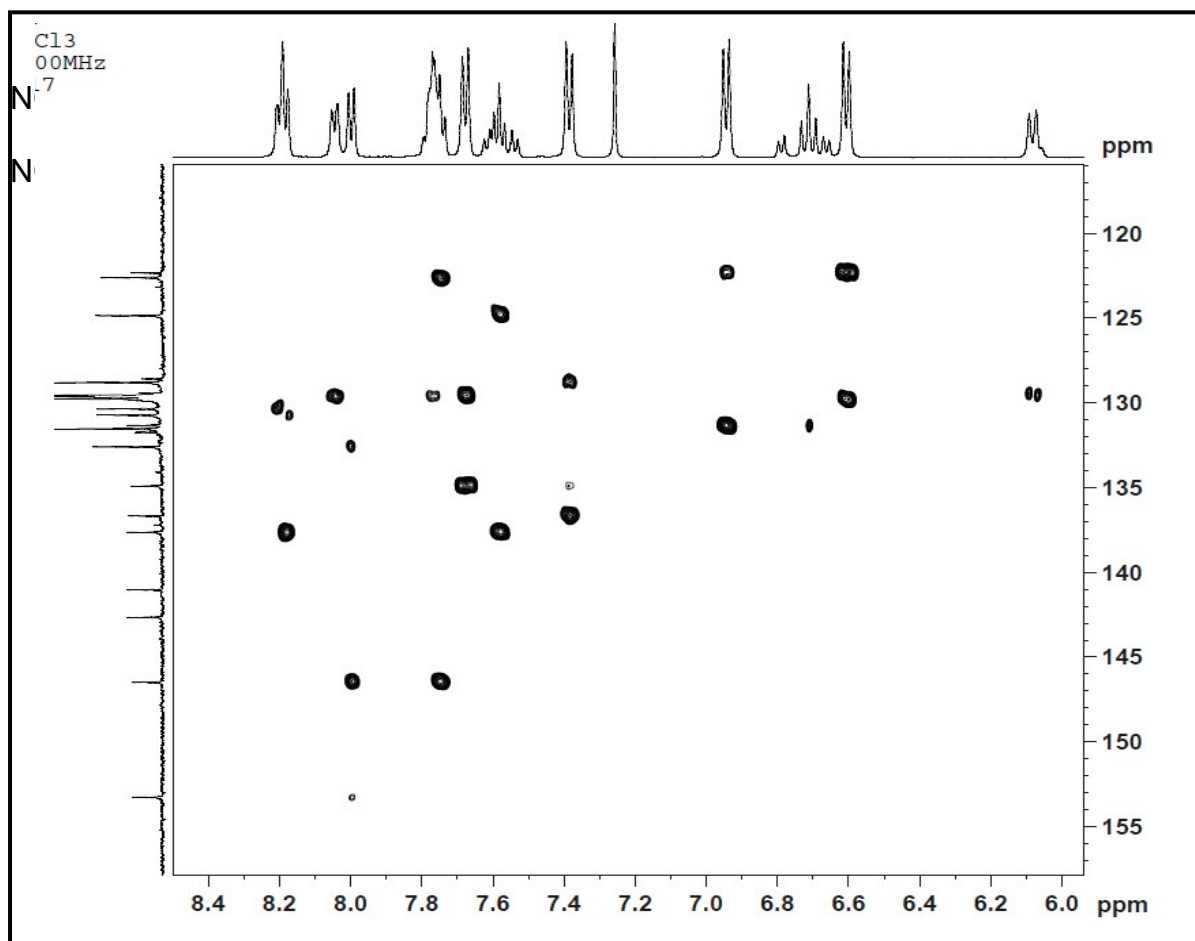
HSQC NMR Spectrum(expansion) (CDCl<sub>3</sub>, 400 MHz) of compound 5i



HMBC NMR Spectrum (CDCl<sub>3</sub>, 400 MHz) of compound 5i

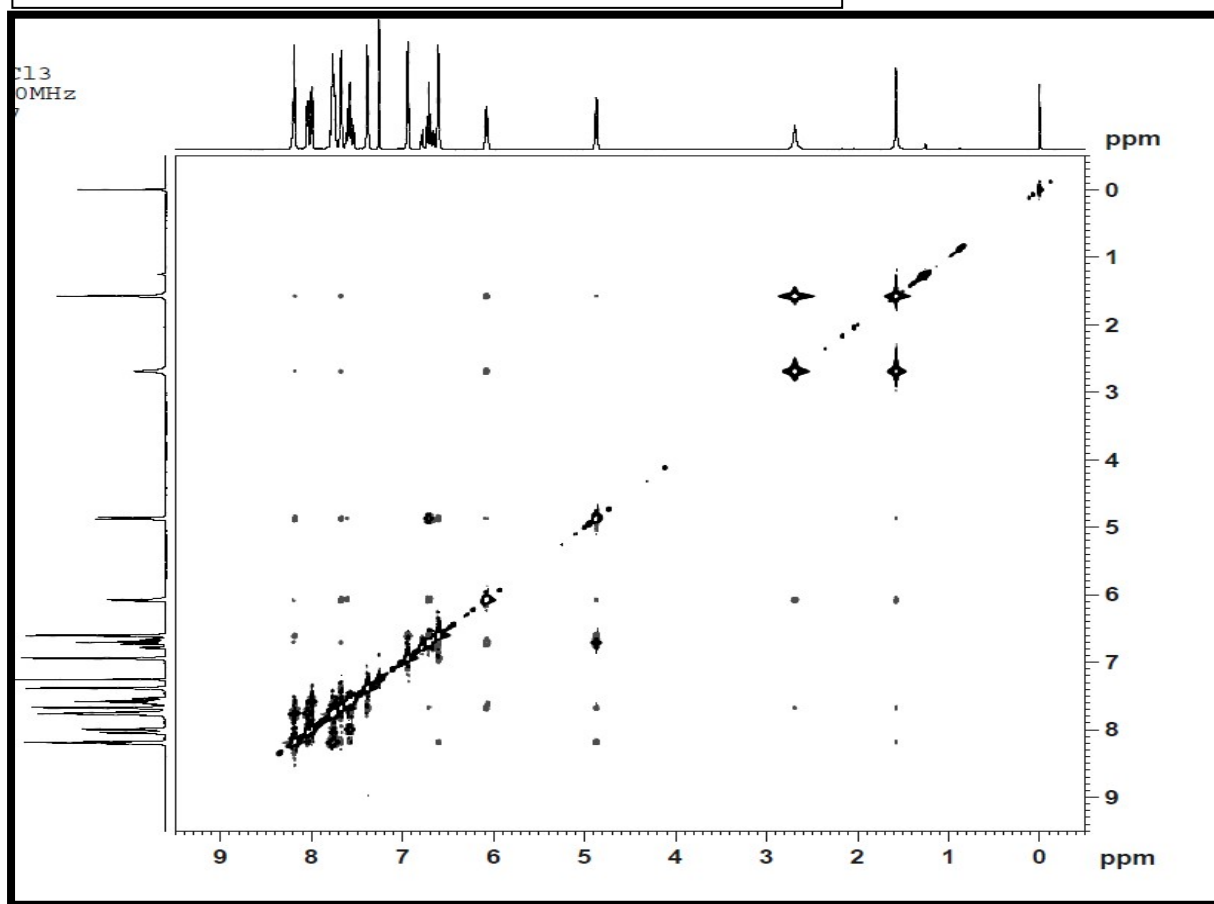


HMBC NMR Spectrum(expansion) (CDCl<sub>3</sub>, 400 MHz) of compound 5i

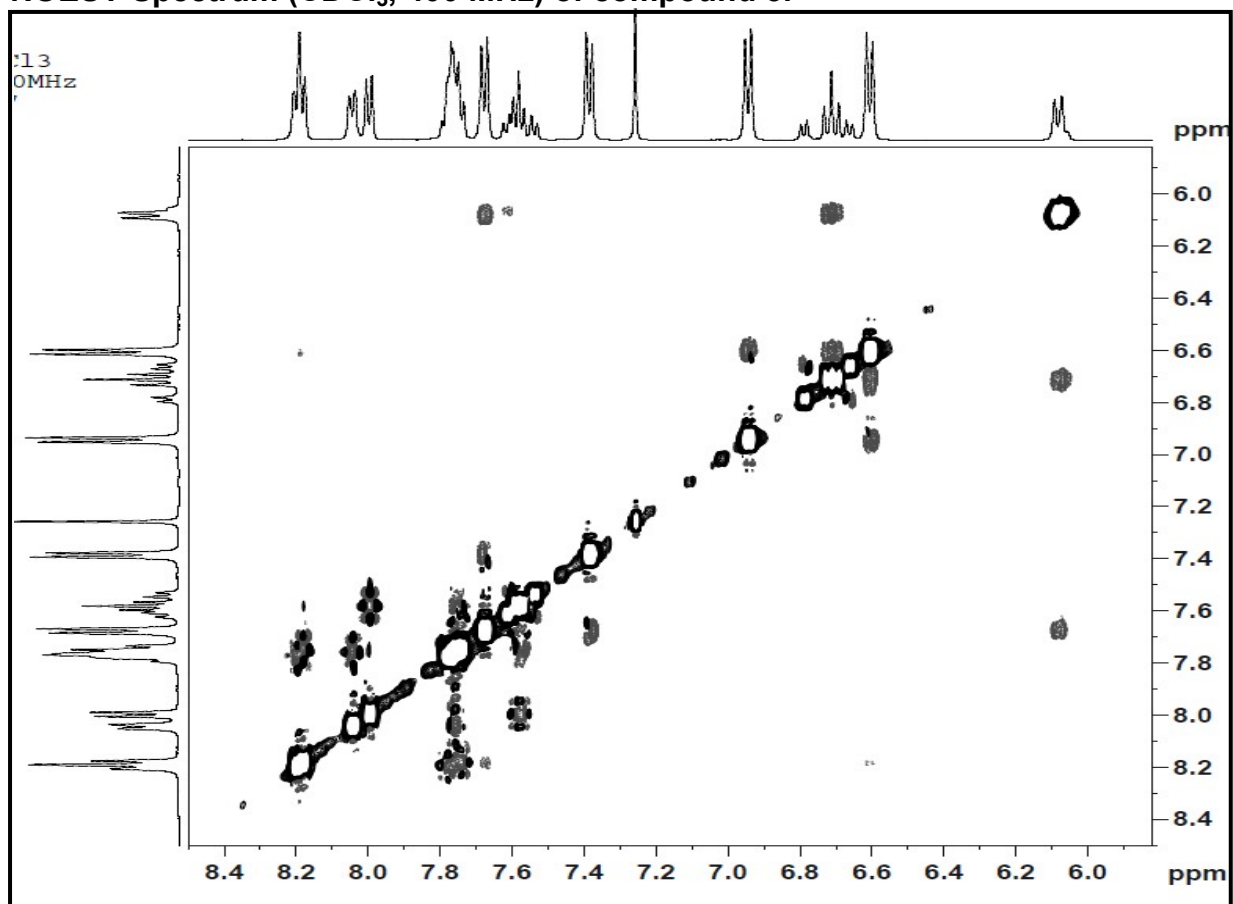




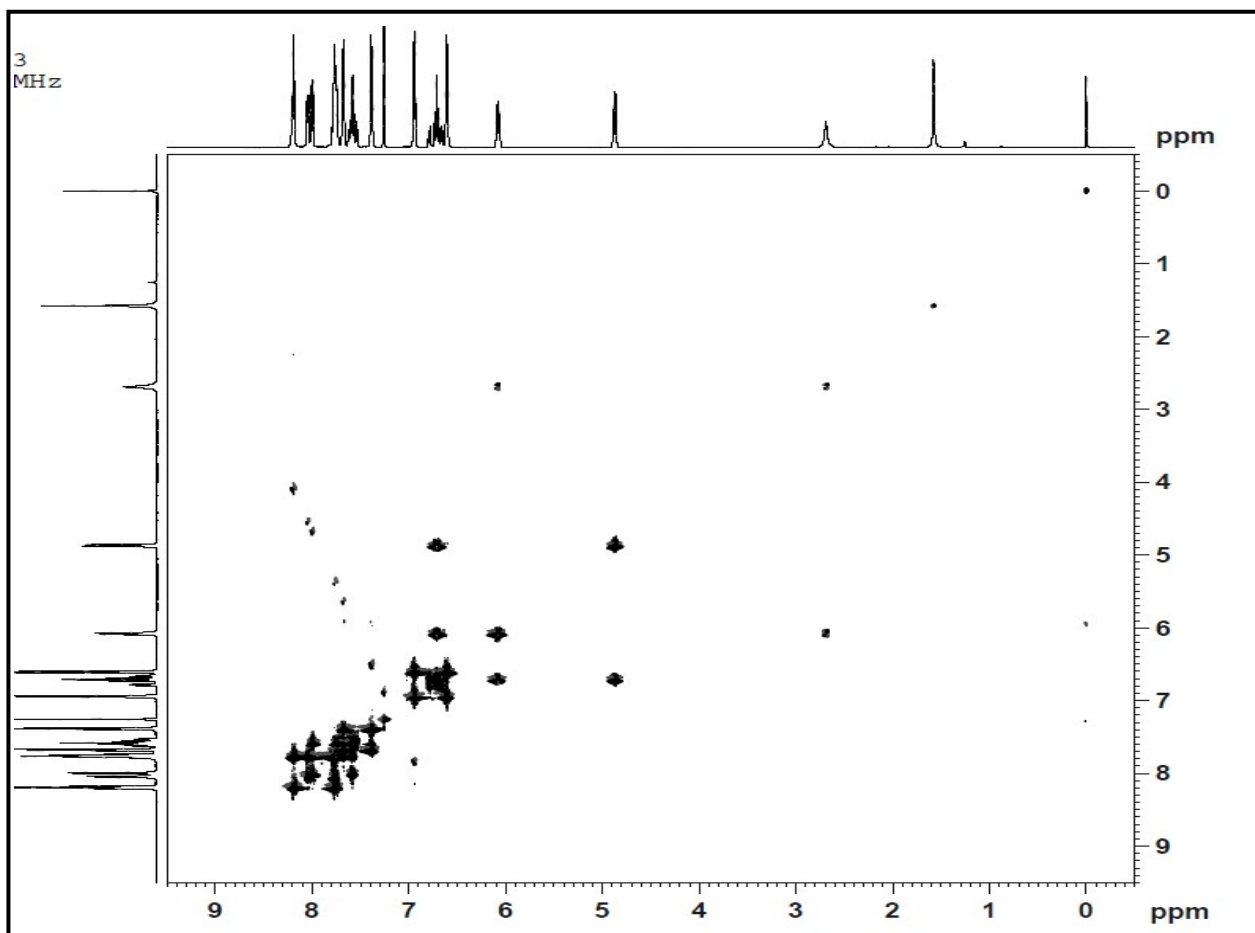
NOESY Spectrum (CDCl<sub>3</sub>, 400 MHz) of compound 5i



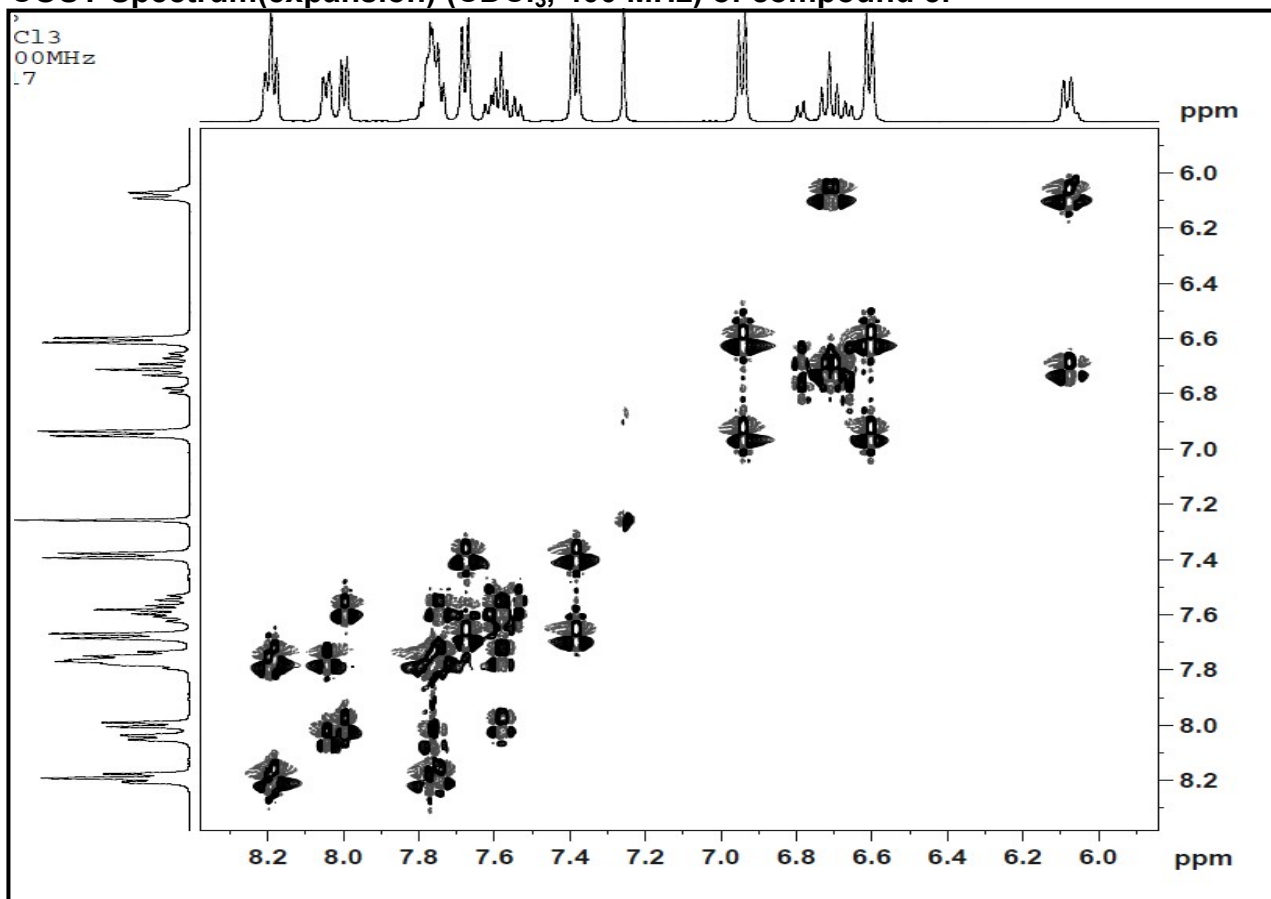
NOESY Spectrum (CDCl<sub>3</sub>, 400 MHz) of compound 5i



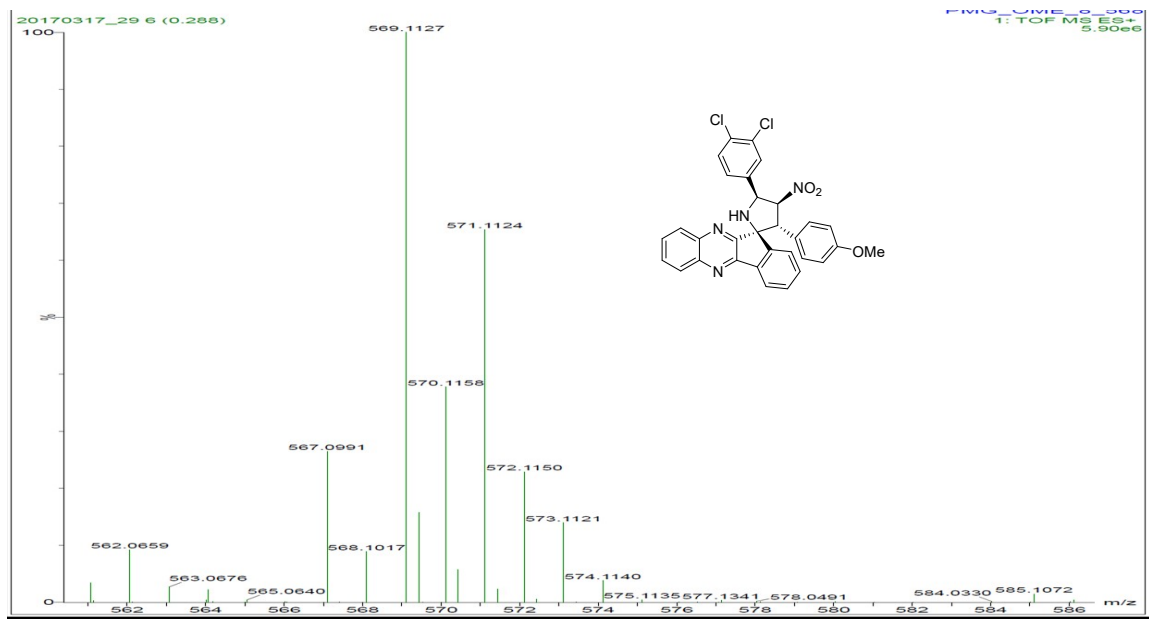
COSY Spectrum (CDCl<sub>3</sub>, 400 MHz) of compound 5i



COSY Spectrum(expansion) (CDCl<sub>3</sub>, 400 MHz) of compound 5i



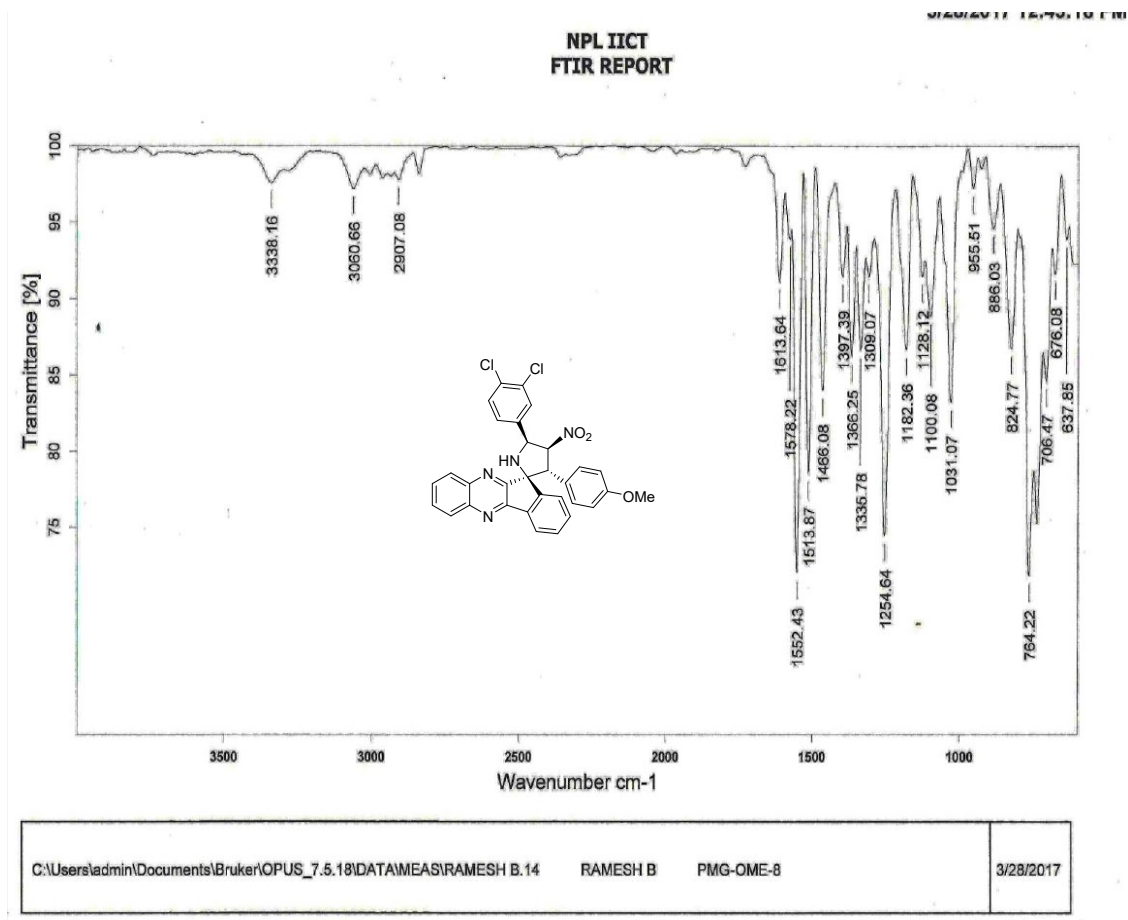
## HR-ESIM Spectrum of compound 5j



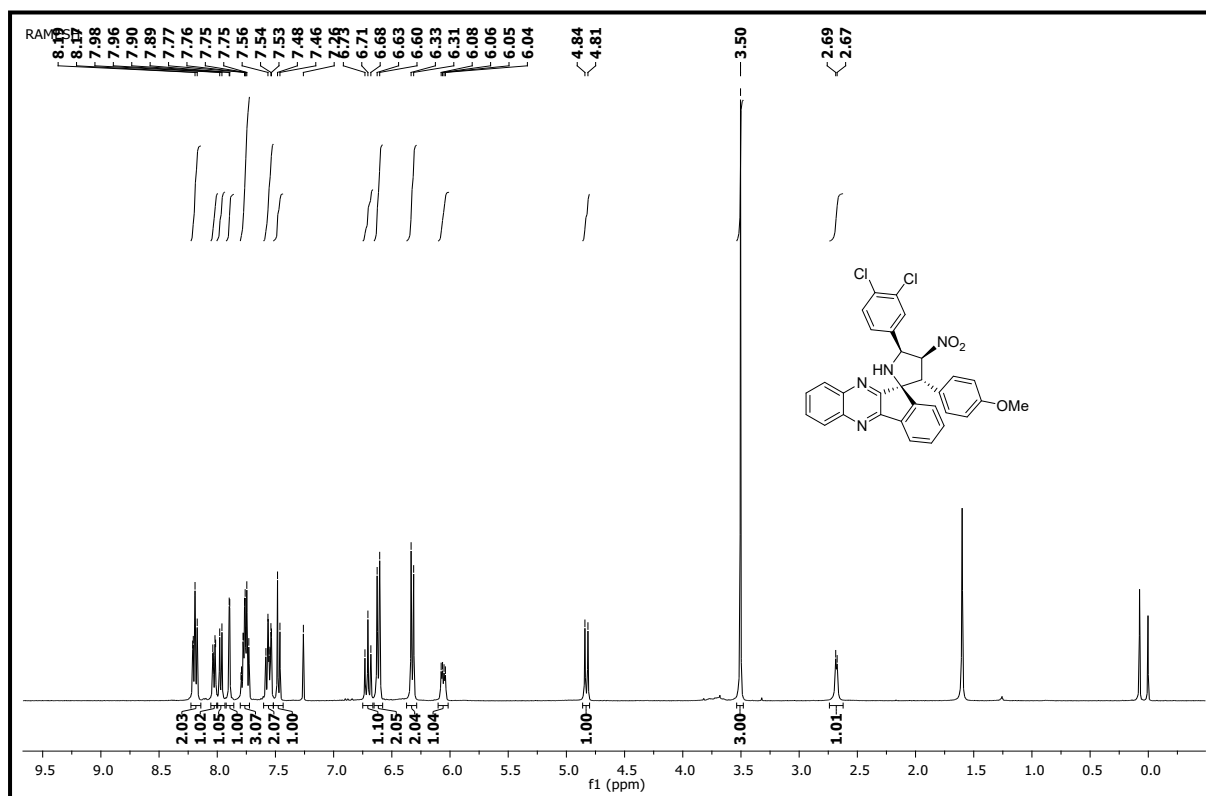
91 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)  
 Elements Used:  
 C: 0-31 H: 0-23 N: 0-4 O: 0-3 Na: 0-1 Cl: 0-2  
 20170317\_29.6 (0.288) PMG\_OME\_8\_568  
1: TOF MS ES+  
5.90e+006

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
569.1127	569.1147	-2.0	-3.5	21.5	131.6	n/a	n/a	C31 H23 N4 O3 Cl2

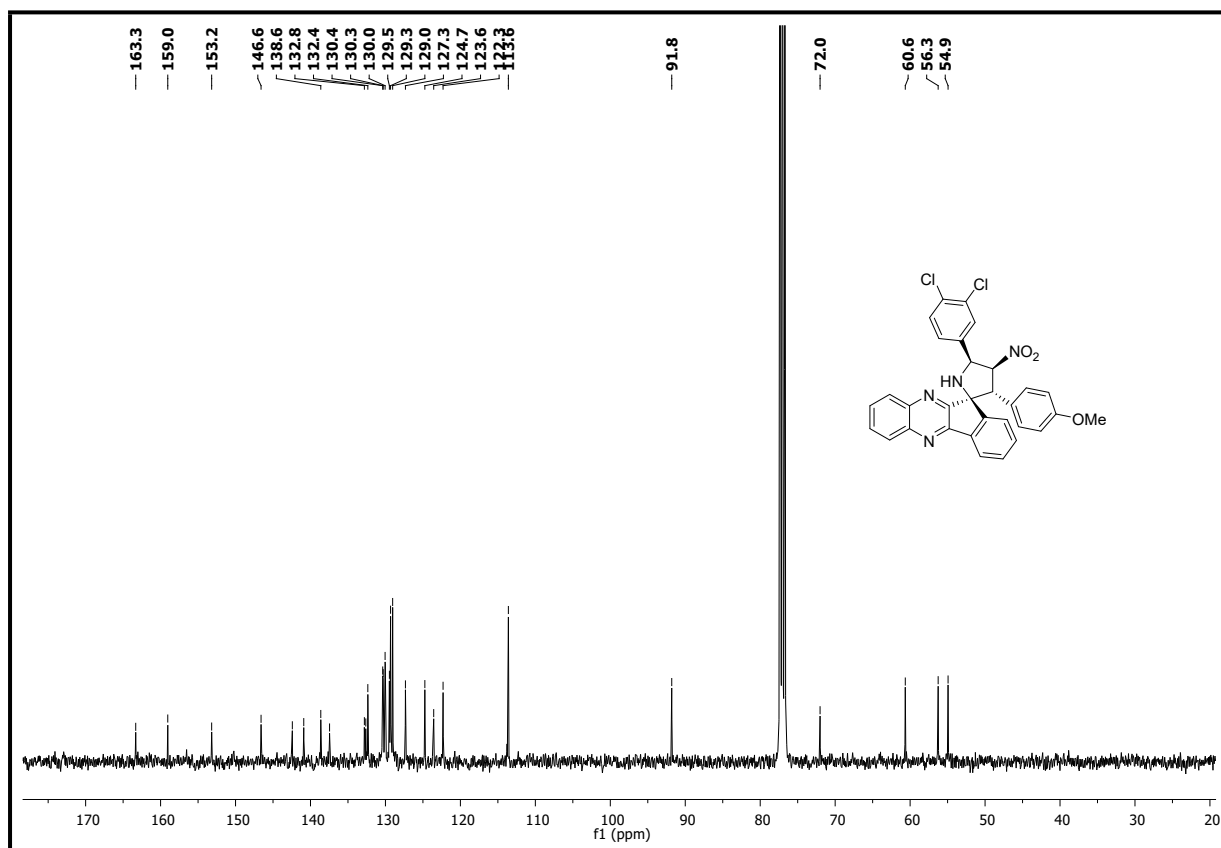
## IR Spectrum of compound 5j



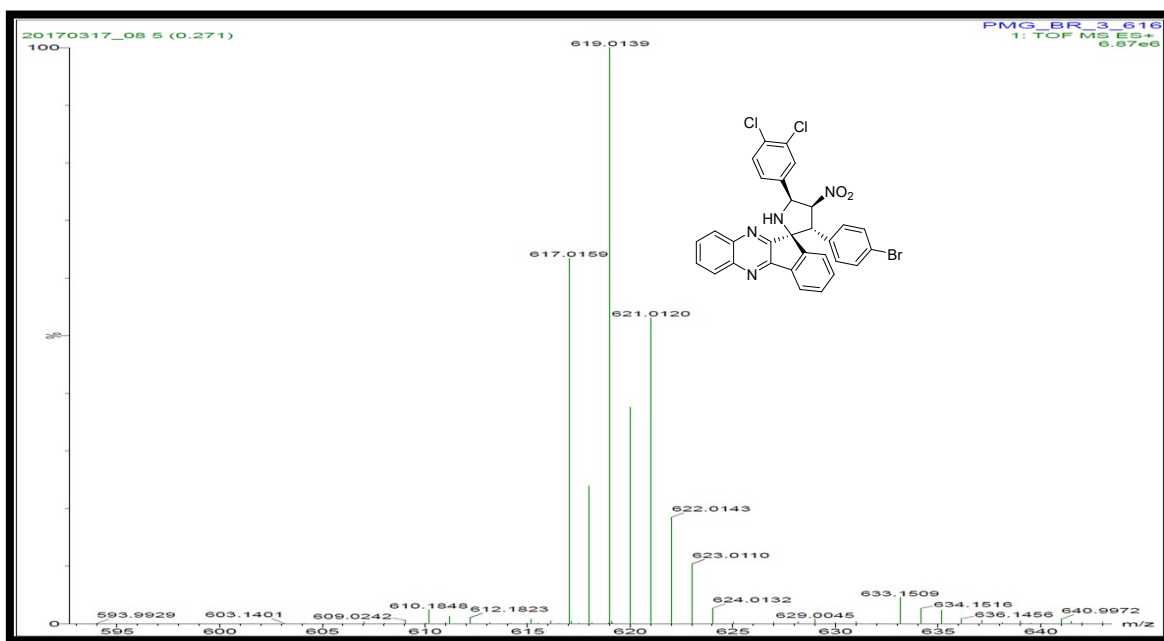
### <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>, 400 MHz) of compound 5j



### <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>, 100 MHz) of compound 5j



# HR-ESIM Spectrum of compound 5k



120 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

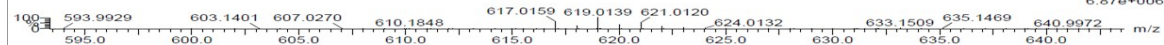
C: 0-30 H: 0-20 N: 0-5 O: 0-3 Cl: 0-2 Br: 0-1

20170317\_08 5 (0.271)

PMG\_BR\_3\_616

1: TOF MS ES+

6.87e+006



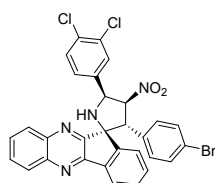
Minimum:

Maximum: 5.0 10.0 -1.5

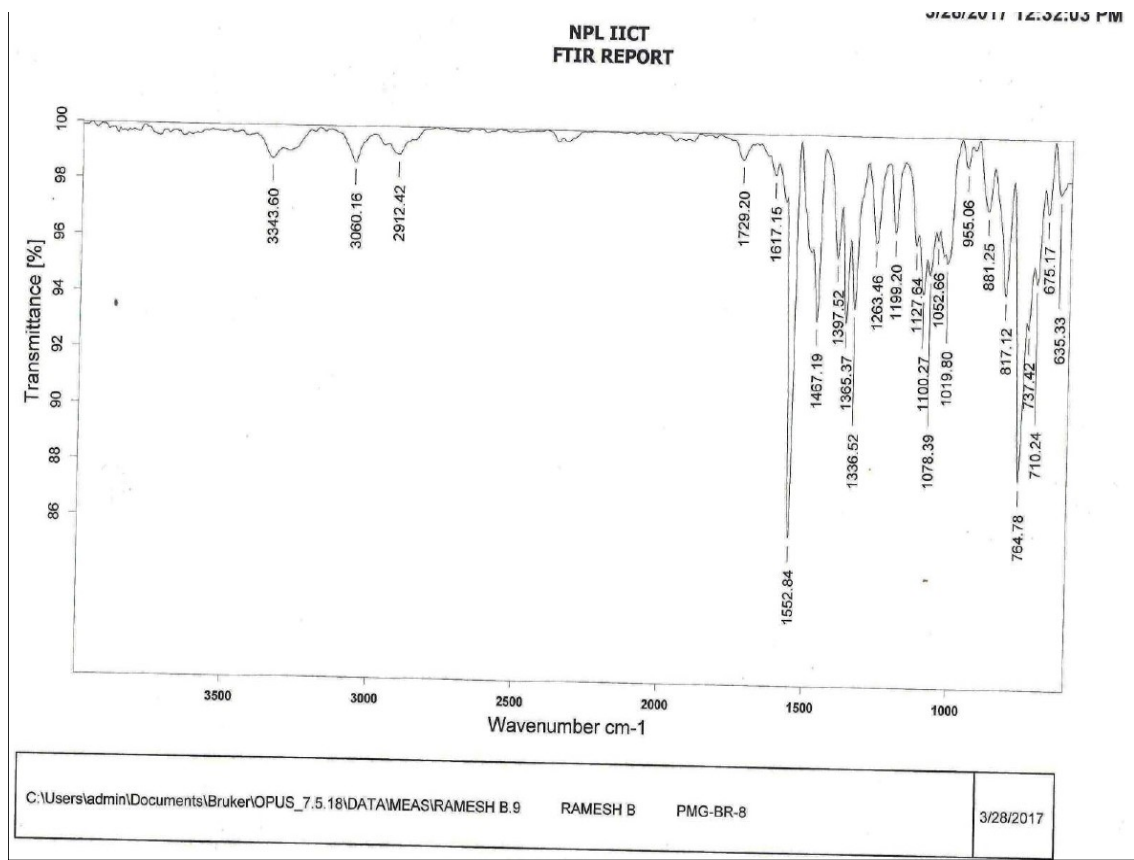
50.0

Mass Calc. Mass mDa PPM DBE i-FIT Norm Conf(%) Formula

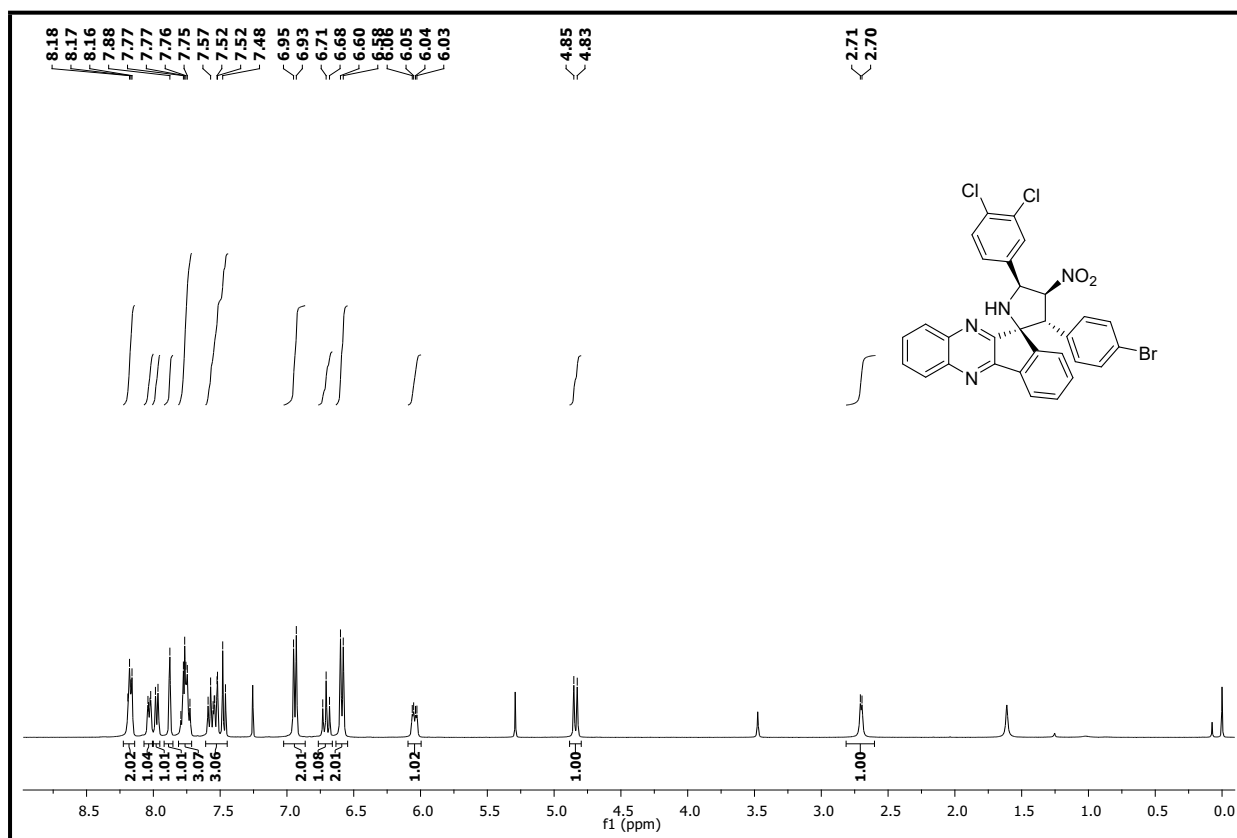
617.0159 617.0147 1.2 1.9 21.5 143.5 n/a n/a C30 H20 N4 O2 Cl2 Br



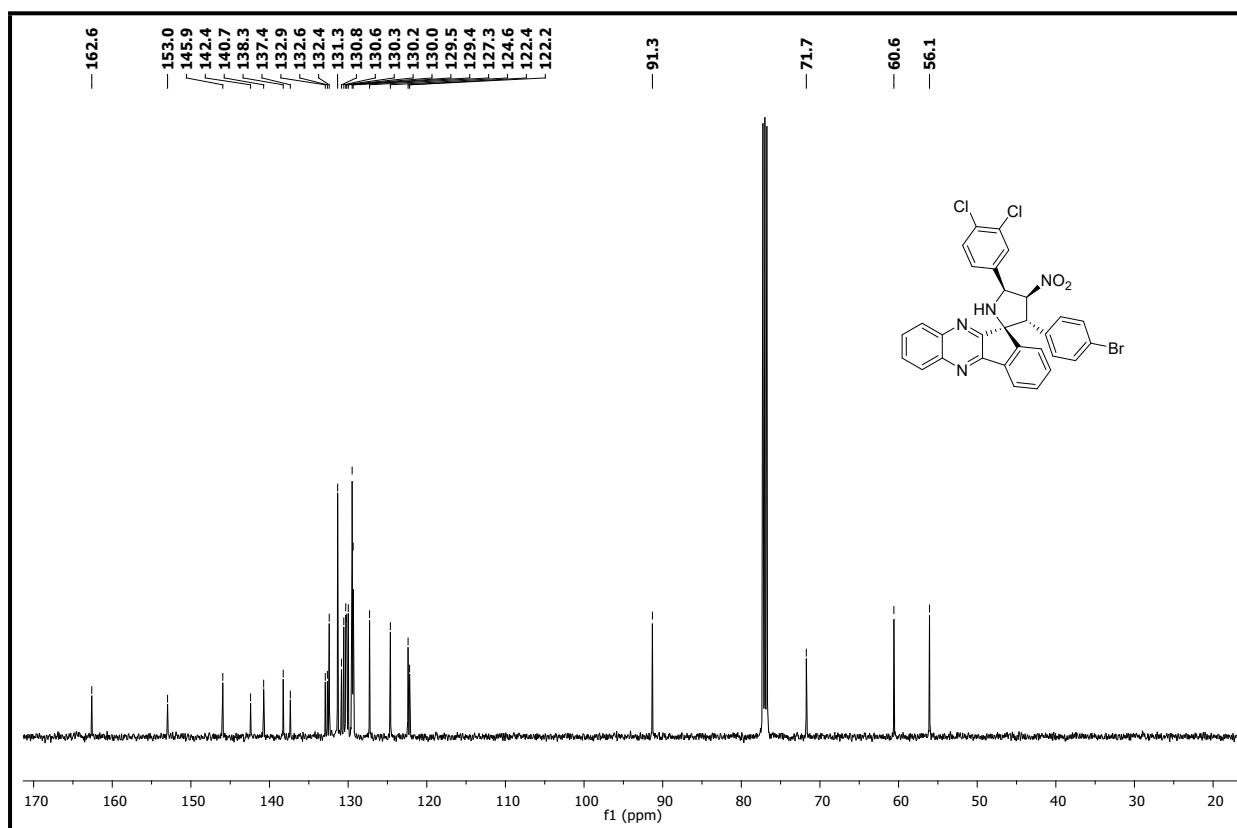
## R Spectrum of compound 5k



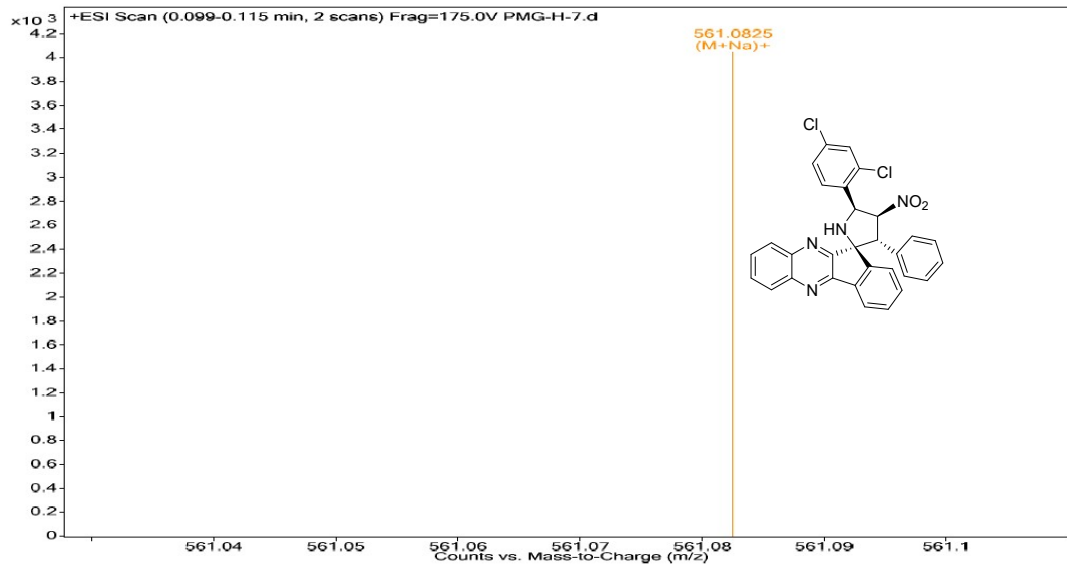
## <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>, 500 MHz) of compound 5k



**<sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>, 125 MHz) of compound 5k**



**HR-ESIM Spectrum of compound 5l**



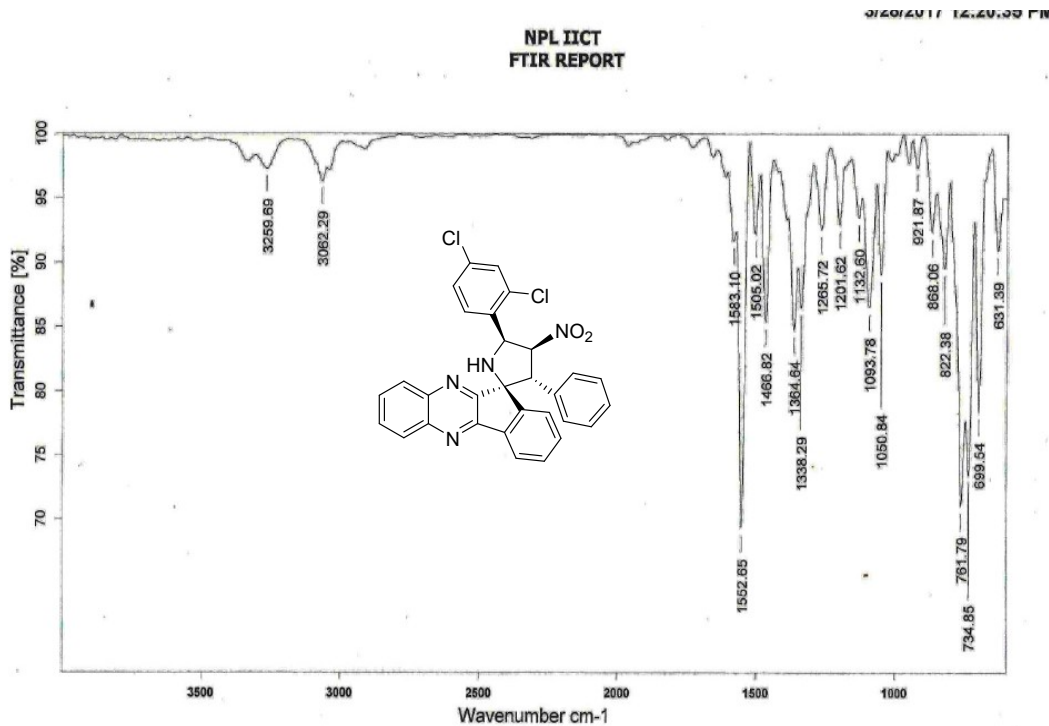
MS Formula Results: + Scan (0.099-0.115 min) - PMG-H-7.d (PMG-H-7.d)

m/z	Ion	Formula	Abundance
561.0825	(M+Na) <sup>+</sup>	C <sub>30</sub> H <sub>20</sub> Cl <sub>2</sub> N <sub>4</sub> O <sub>2</sub>	4645.4

Best	Formula (M)	Ion Formula	Calc. m/z	Score	Cross Score	Mass	Calc. Mass	Diff (ppm)	Abs. Diff (ppm)	Abund. Match	Spacing Match	Mass Match	m/z	DBE
<input checked="" type="checkbox"/>	C <sub>30</sub> H <sub>20</sub> Cl <sub>2</sub> N <sub>4</sub> O <sub>2</sub>	C <sub>30</sub> H <sub>20</sub> Cl <sub>2</sub> N <sub>4</sub> O <sub>2</sub>	561.0856	43.95		538.0925	538.0963	7.07	7.07	9.55	57.51	57.82	561.0825	22

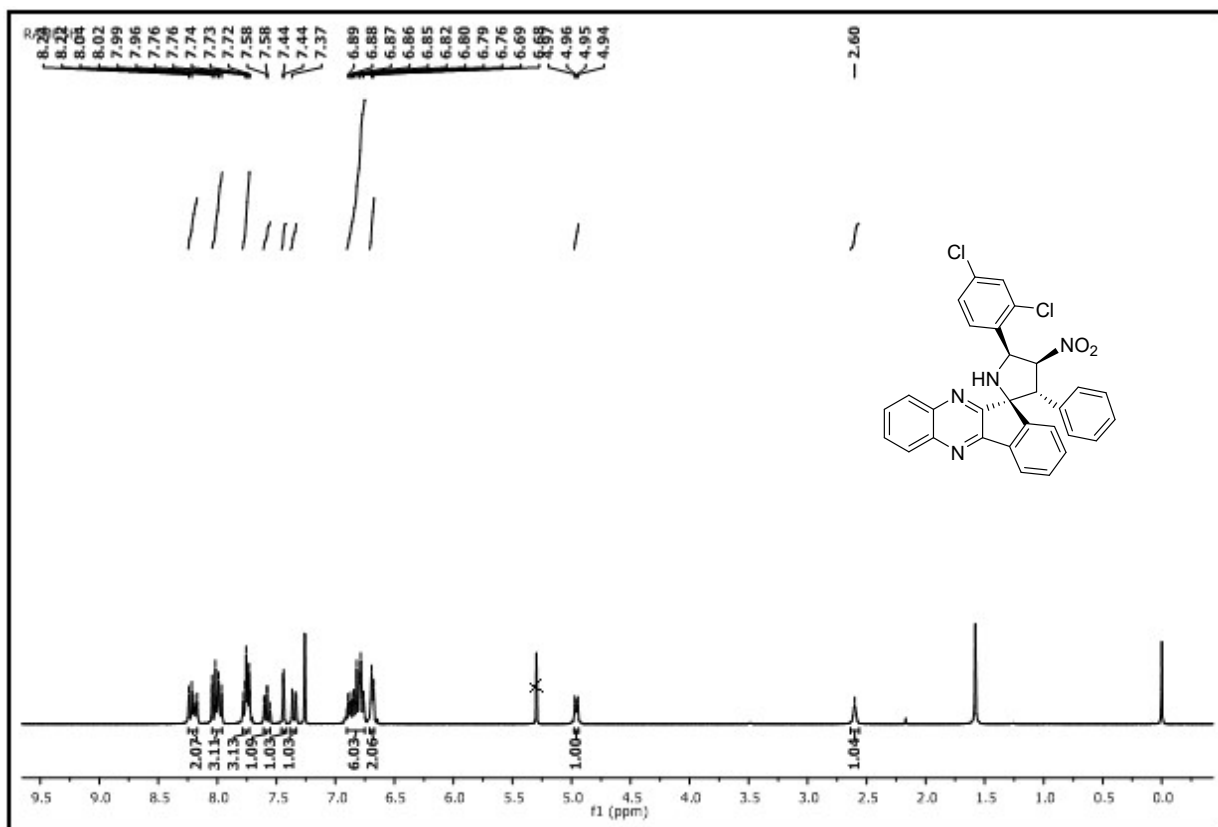
### IR Spectrum of compound 5I



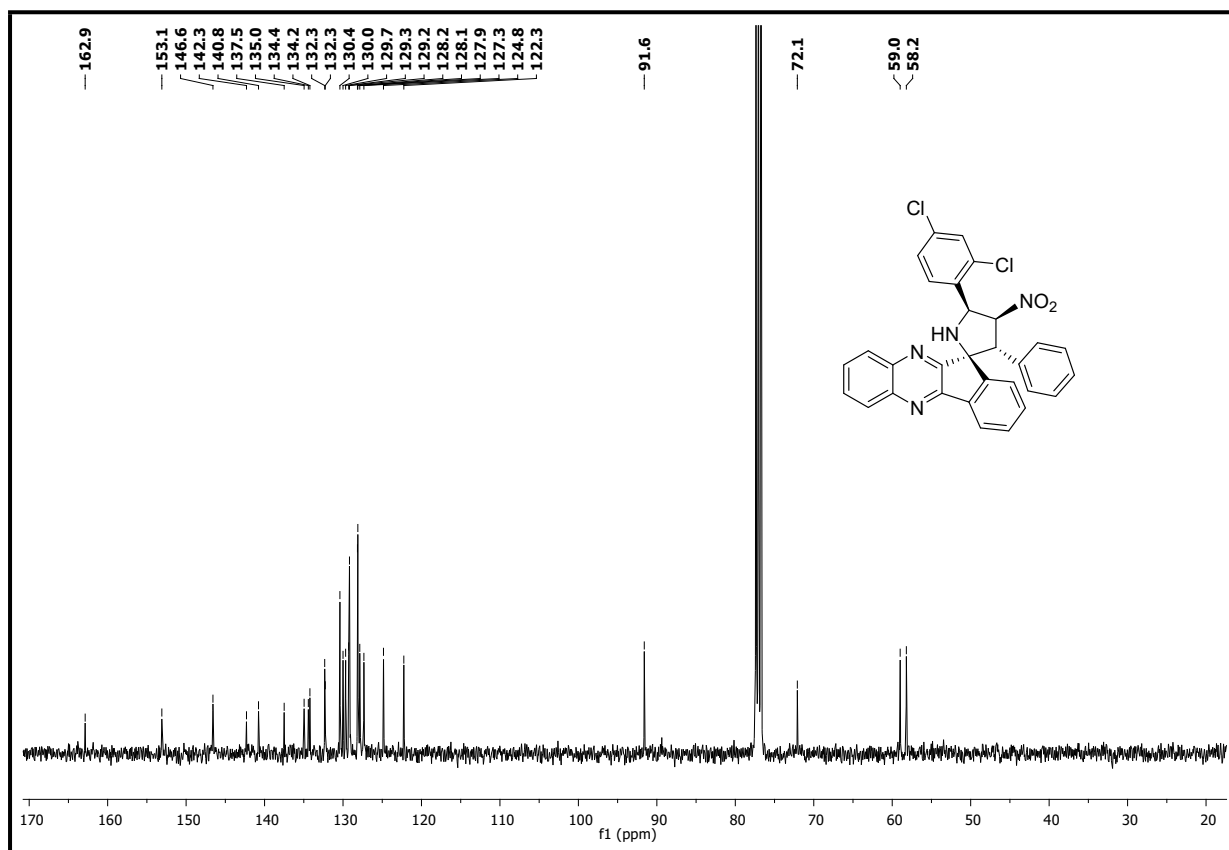
C:\Users\admin\Documents\Bruker\OPUS\_7.5.18\DATA\MEAS\RAMESH B.3 RAMESH B PMG-H-7 3/28/2017

### <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>, 500 MHz) of compound 5I

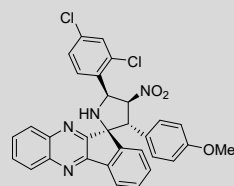




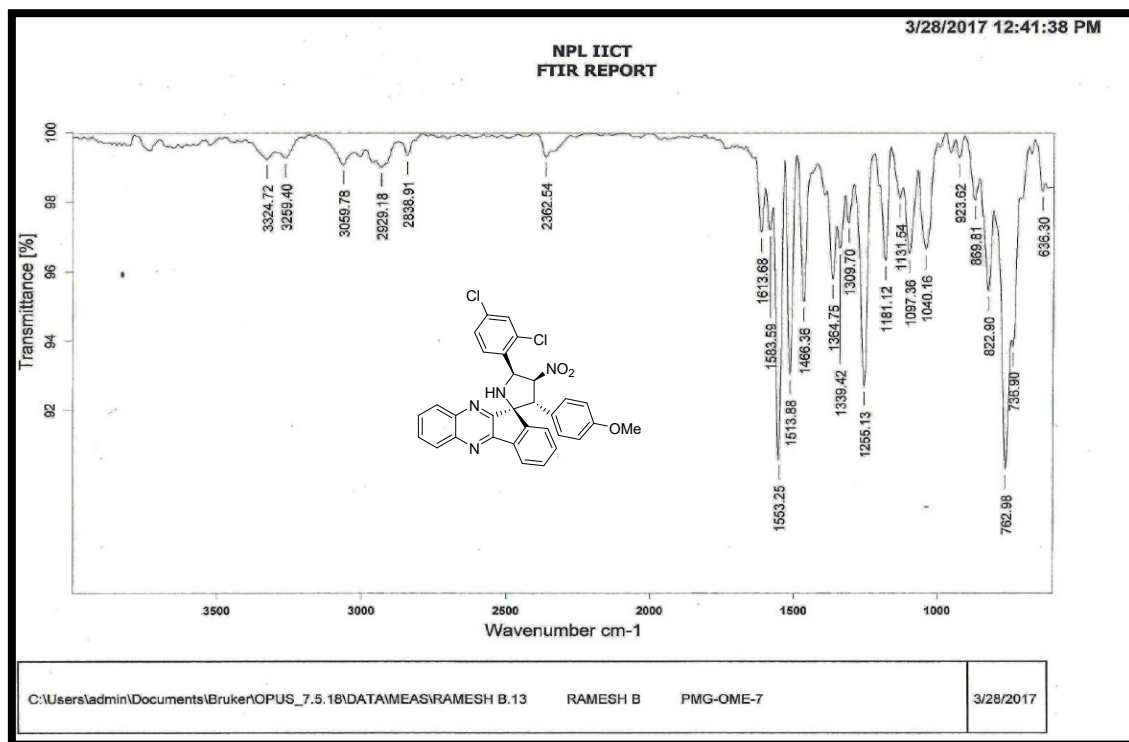
<sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>, 100 MHz) of compound 5l



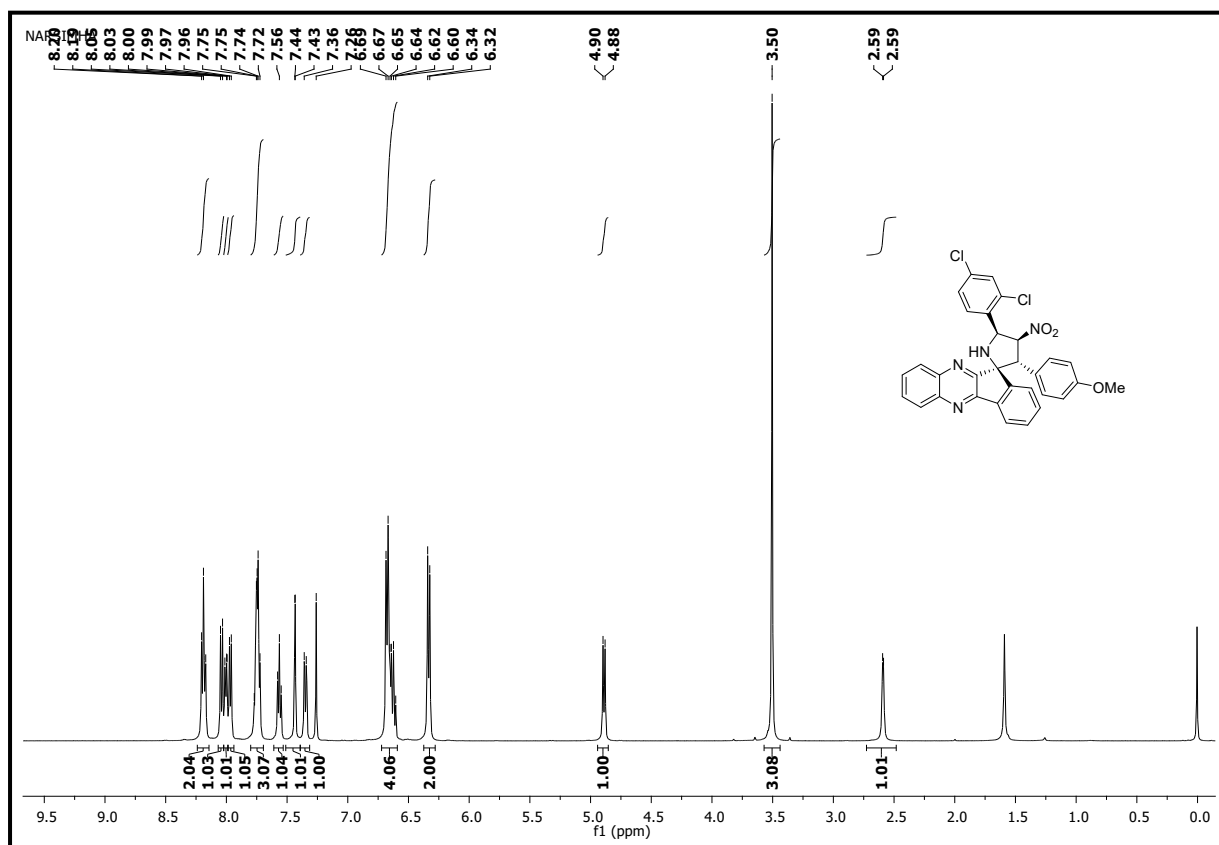
HR-ESIM Spectrum of compound 5m



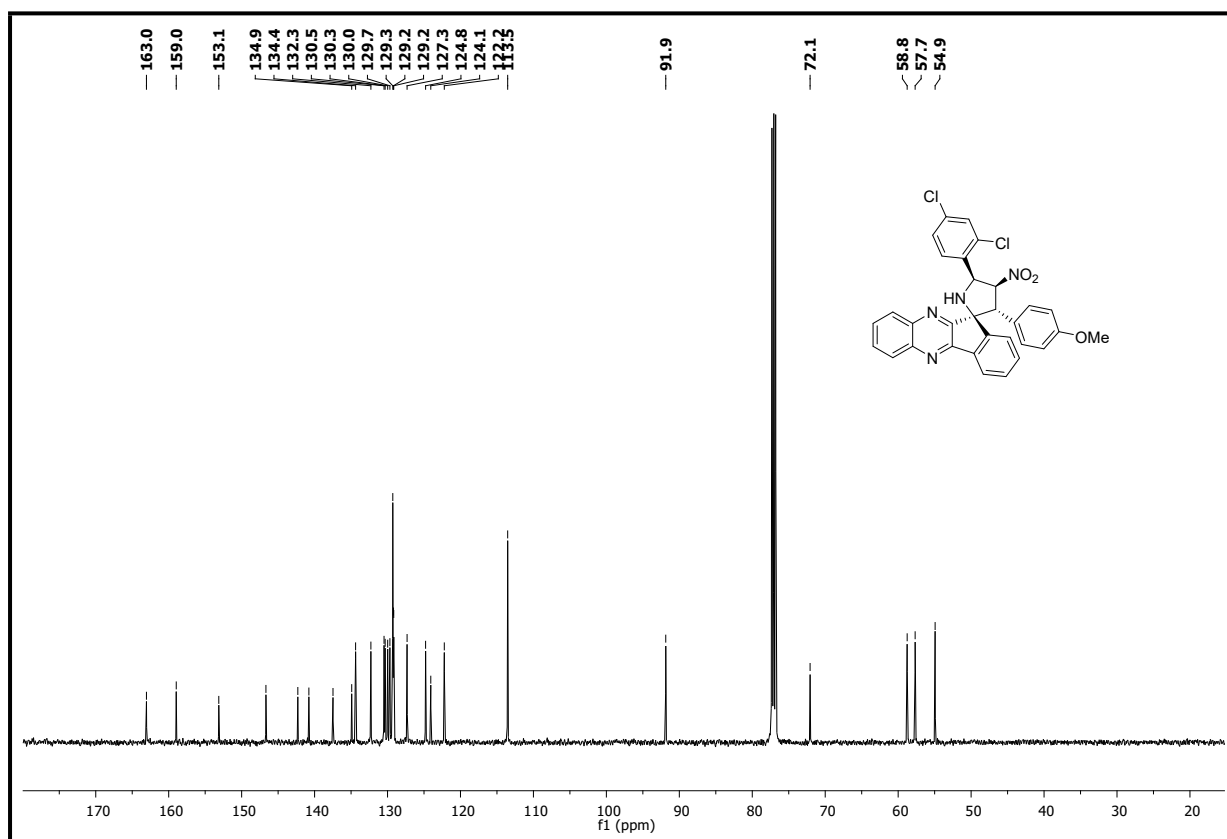
IR Spectrum of compound 5m



<sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>, 400 MHz) of compound 5m

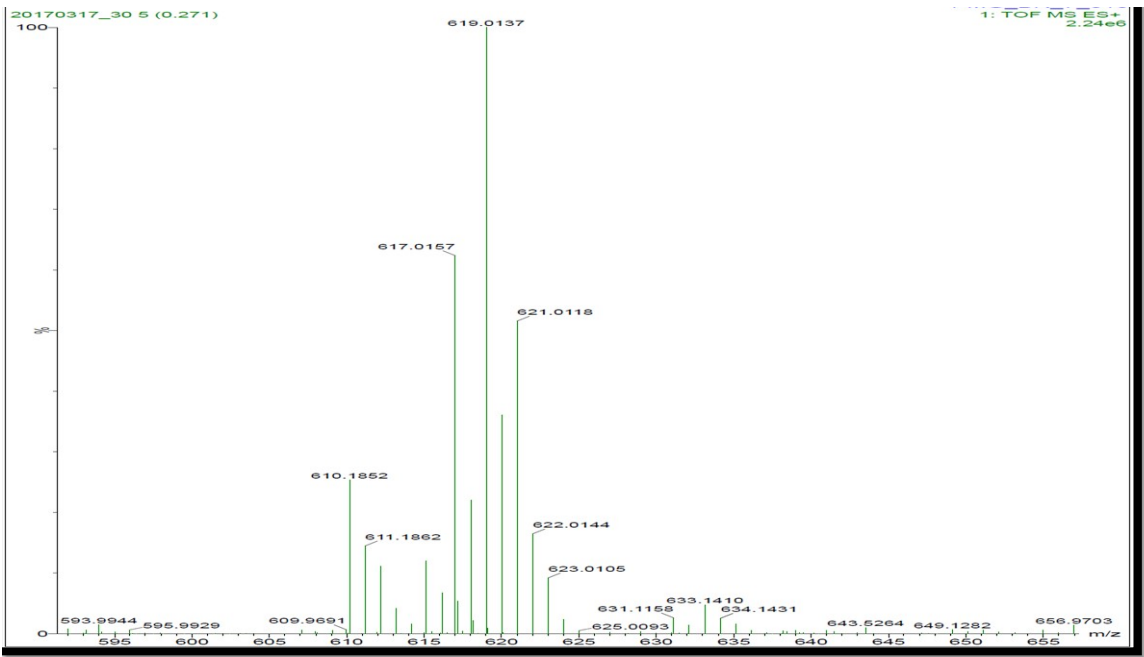


<sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>, 100 MHz) of compound 5m



HR-ESIM Spectrum of compound 5n





98 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)  
 Elements Used:  
 C: 0-31 H: 0-23 N: 0-4 O: 0-3 Cl: 0-2 Br: 0-1

20170317\_30 5 (0.271) PMG\_BR\_7\_616  
2.24e+006

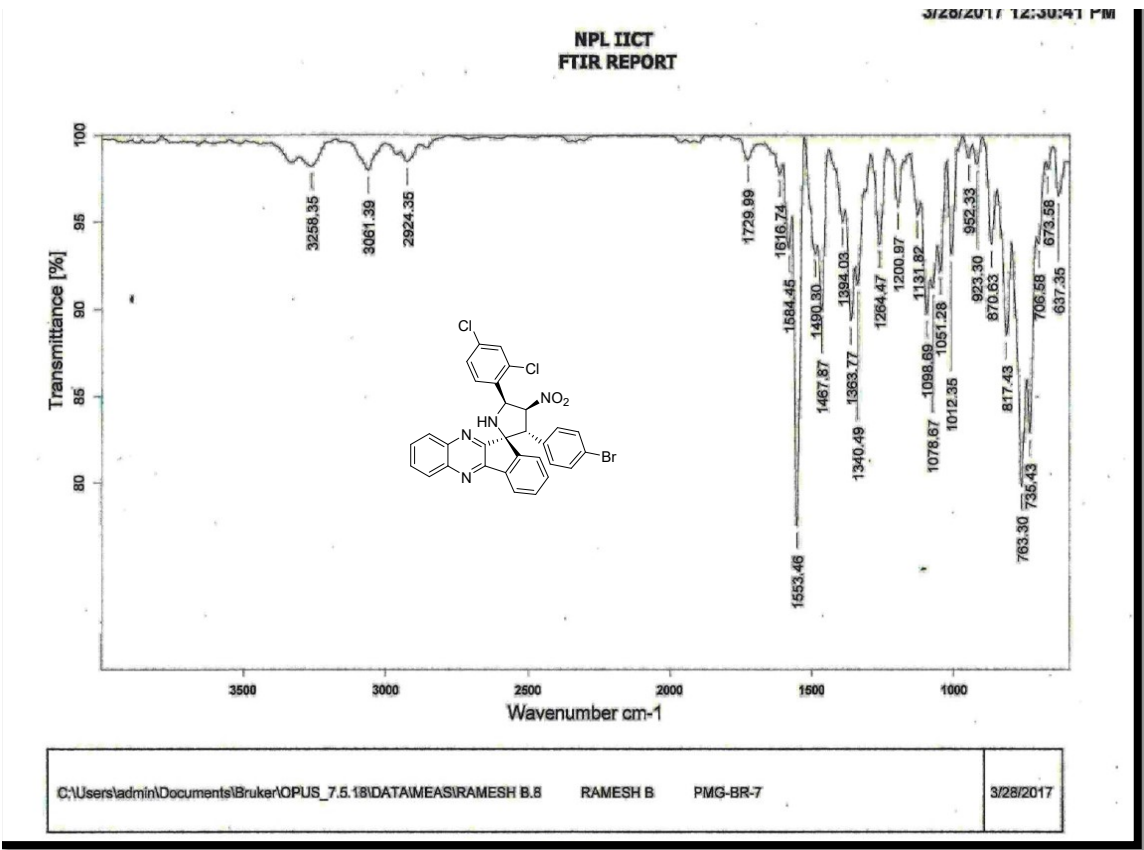
1: TOF MS ES+

m/z	Relative Intensity
593.9944	~1
595.9929	~1
609.9691	~1
610.1852	~10
611.1862	~5
617.0157	~30
619.0137	100
621.0118	~25
622.0144	~5
623.0105	~3
625.0093	~1
631.1158	~1
633.1410	~1
634.1431	~1
643.5264	~1
649.1282	~1
656.9703	~1

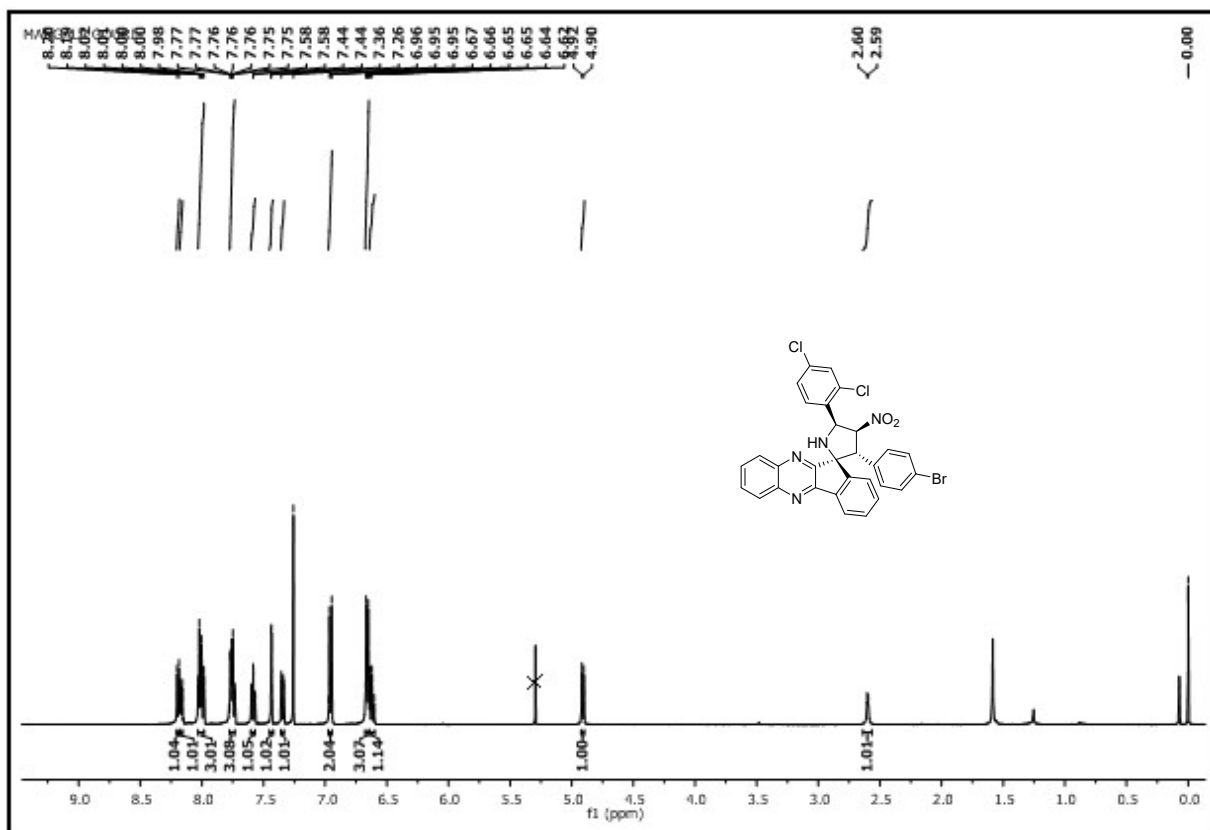
Minimum: 5.0 10.0 -1.5  
 Maximum: 50.0 50.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
617.0157	617.0147	1.0	1.6	21.5	119.3	n/a	n/a	C39 H20 N4 O3 Cl2 Br

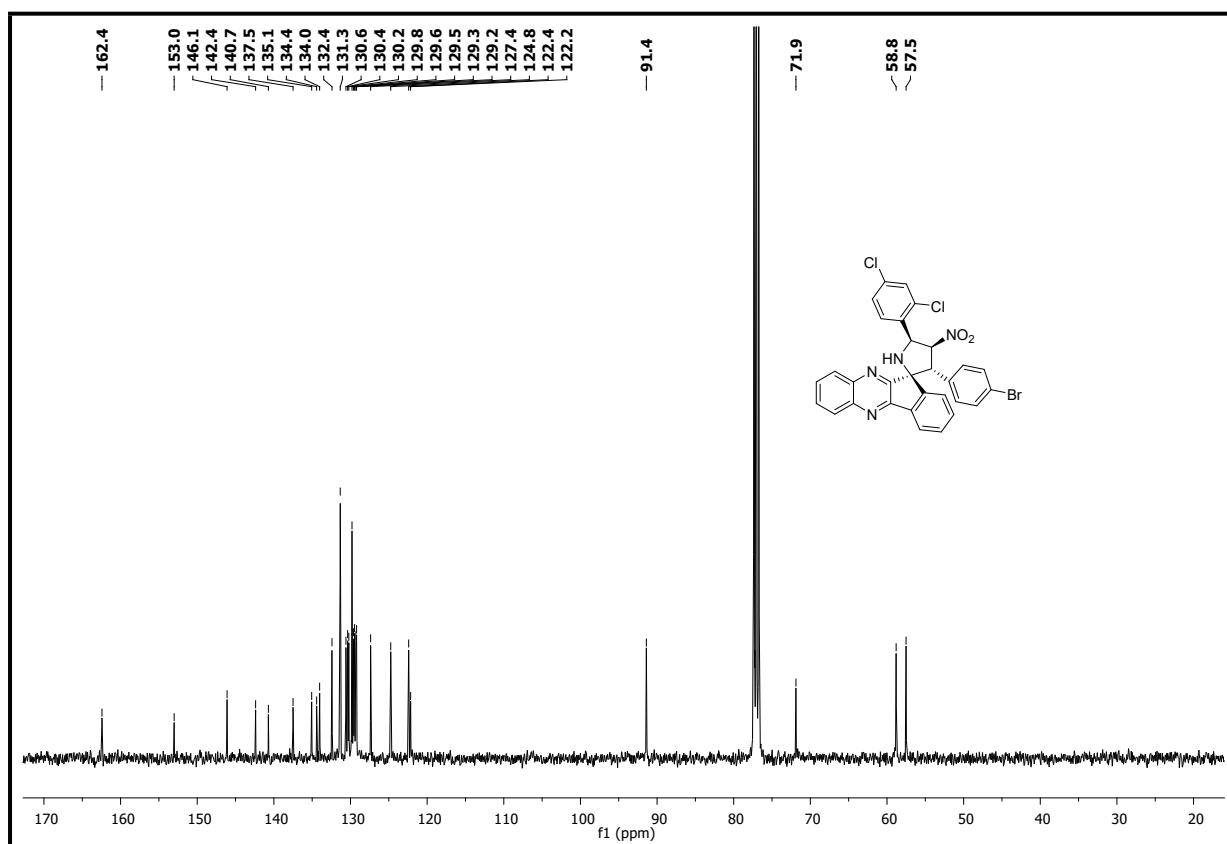
### IR Spectrum of compound 5n



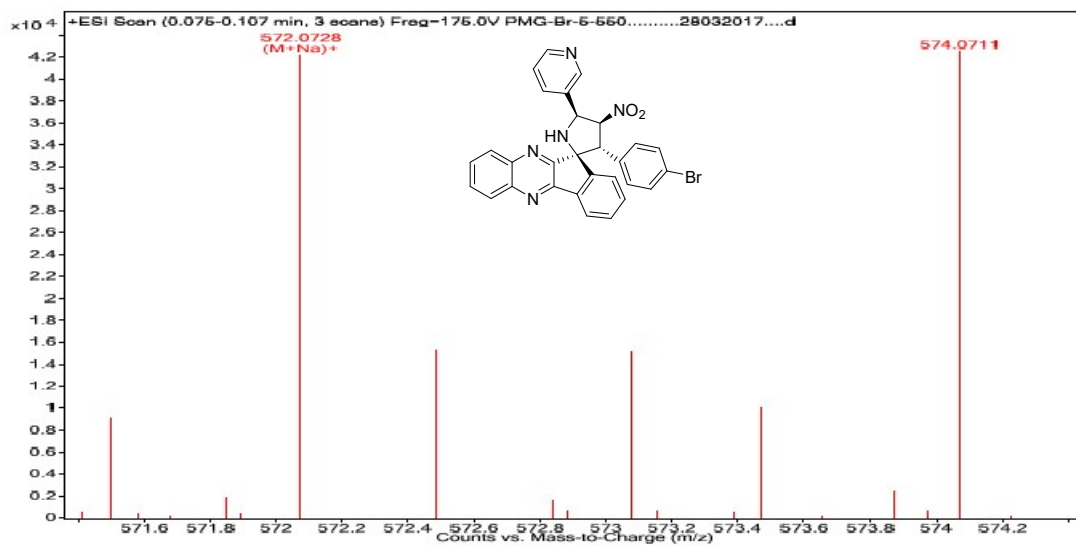
### <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>, 500 MHz) of compound 5n



**<sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>, 125 MHz) of compound 5n**



**HR-ESIM Spectrum of compound 5o**



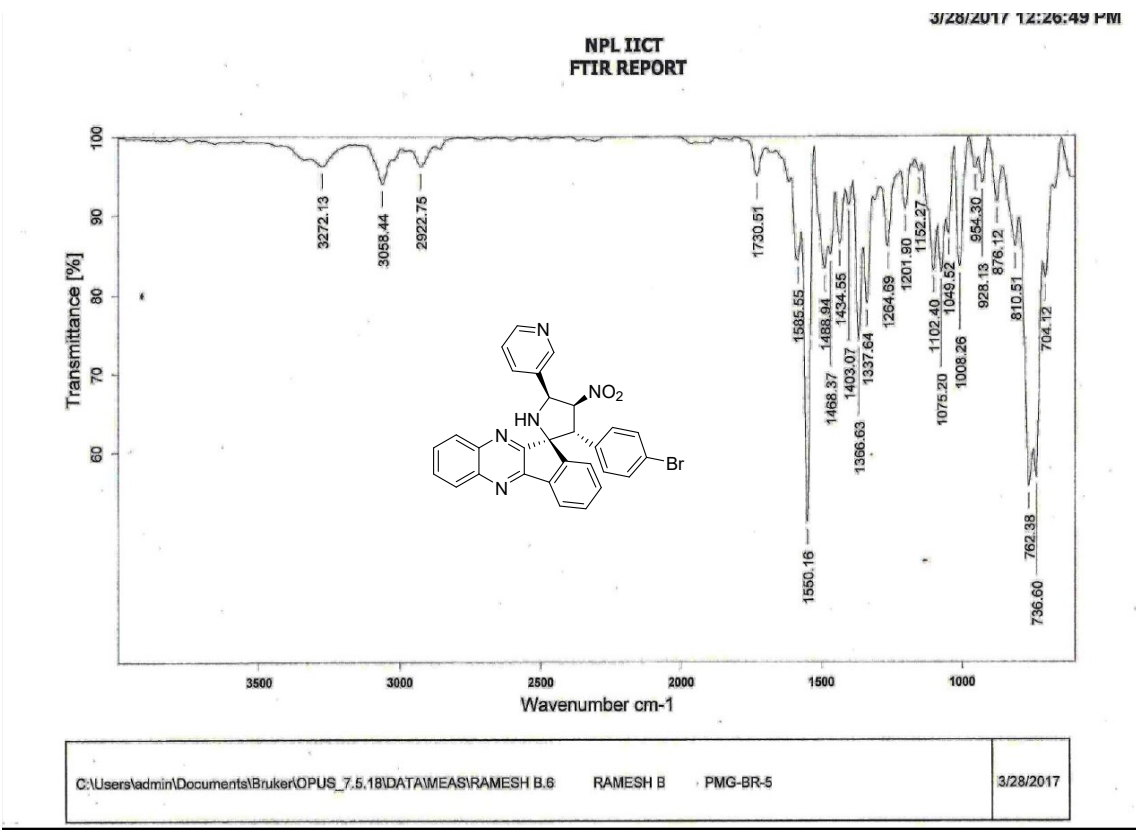
MS Formula Results: + Scan (0.075-0.107 min) (PMG-Br-5-550.....28032017.....d)

m/z	Ion	Formula	Abundance
572.0728	(M+Na)+	C29H20BrN5O2	42108

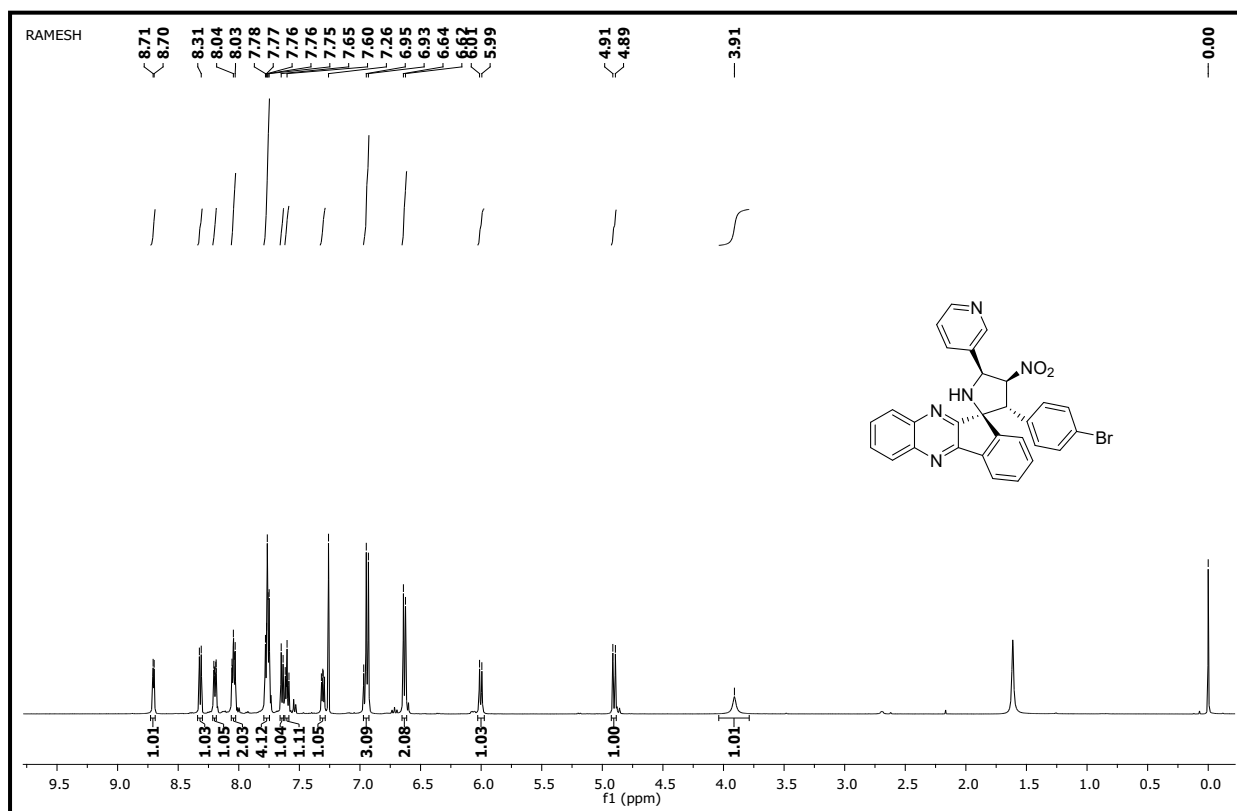
  

Det	Formula (M)	Ion Formula	Calc m/z	Score	Crab Score	Mass	Calc Mass	Diff (ppm)	Ala Diff (ppm)	Abund Match	Spacing Match	Mass Match	m/z	DBE
✓	C29H20BrN5O2	C29H20BrN5NaO2	572.0693	80.61		549.0636	549.06	-6.4	6.4	95.82	96.84	63.37	572.0728	22

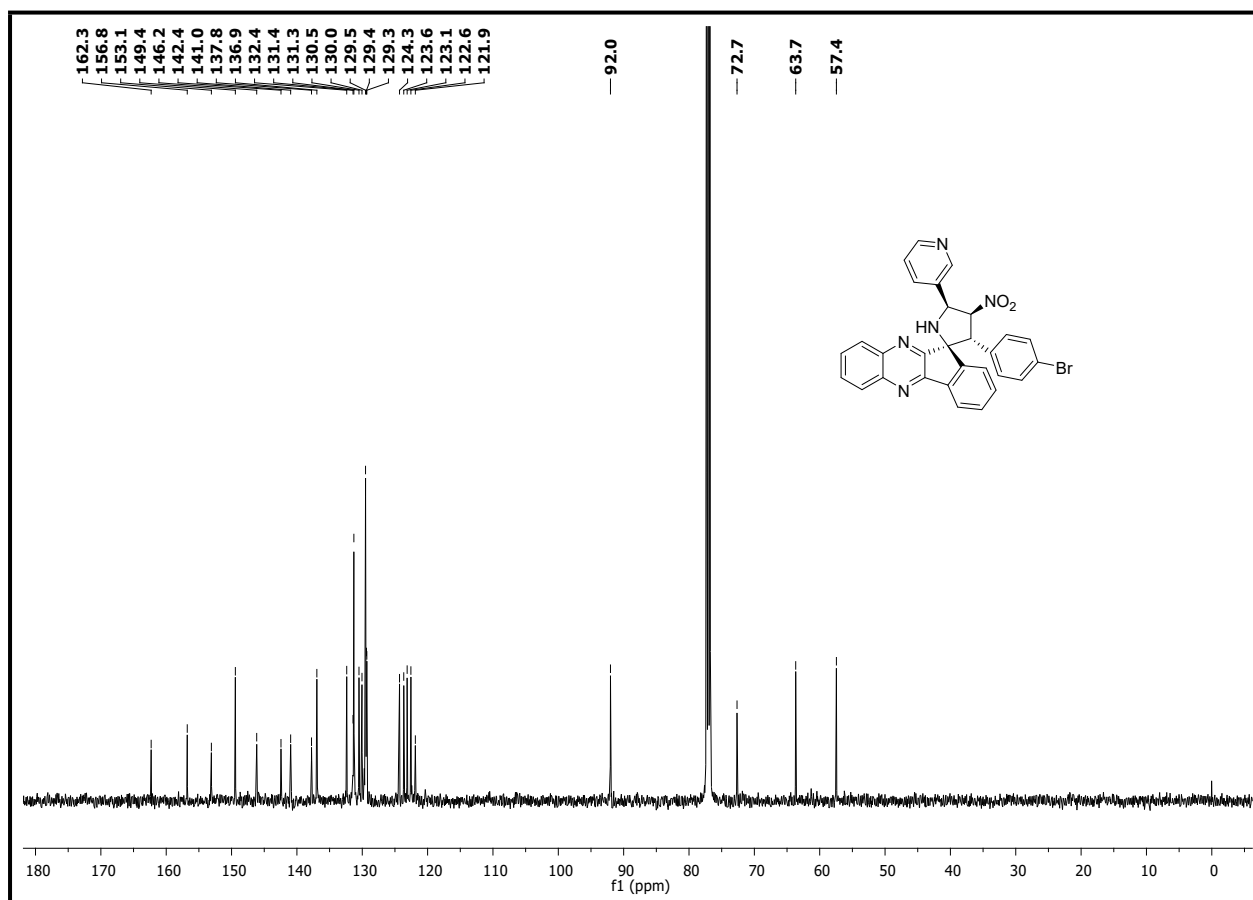
### IR Spectrum of compound 5o



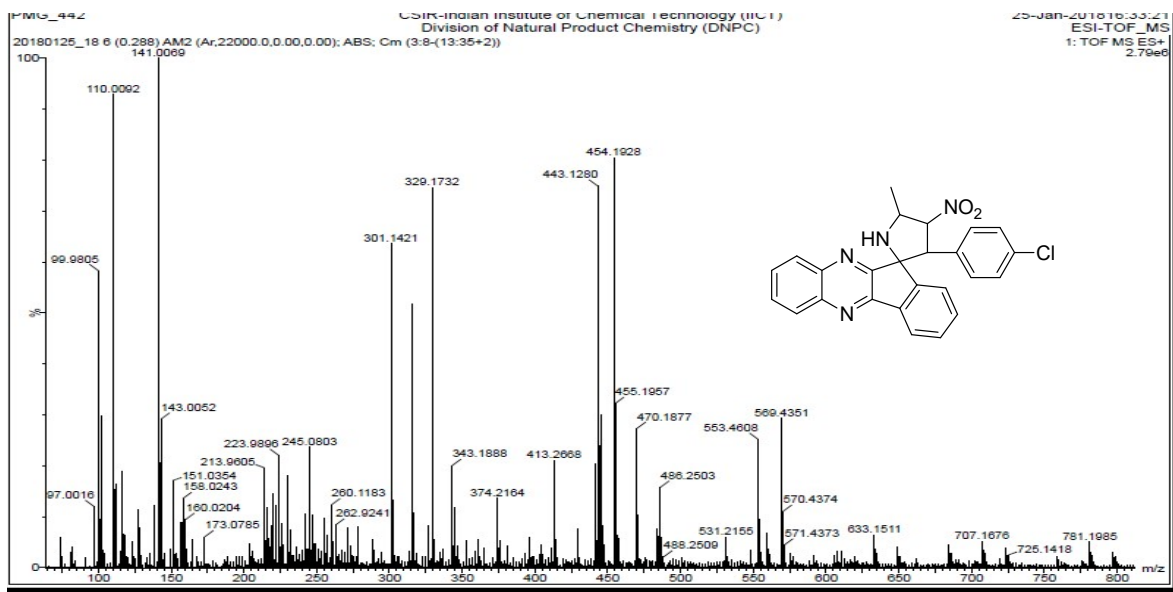
### <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>, 500 MHz) of compound 5o



**<sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>, 125 MHz) of compound 5o**



**HR-ESIM Spectrum of compound 7a**



### Elemental Composition Report

#### Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

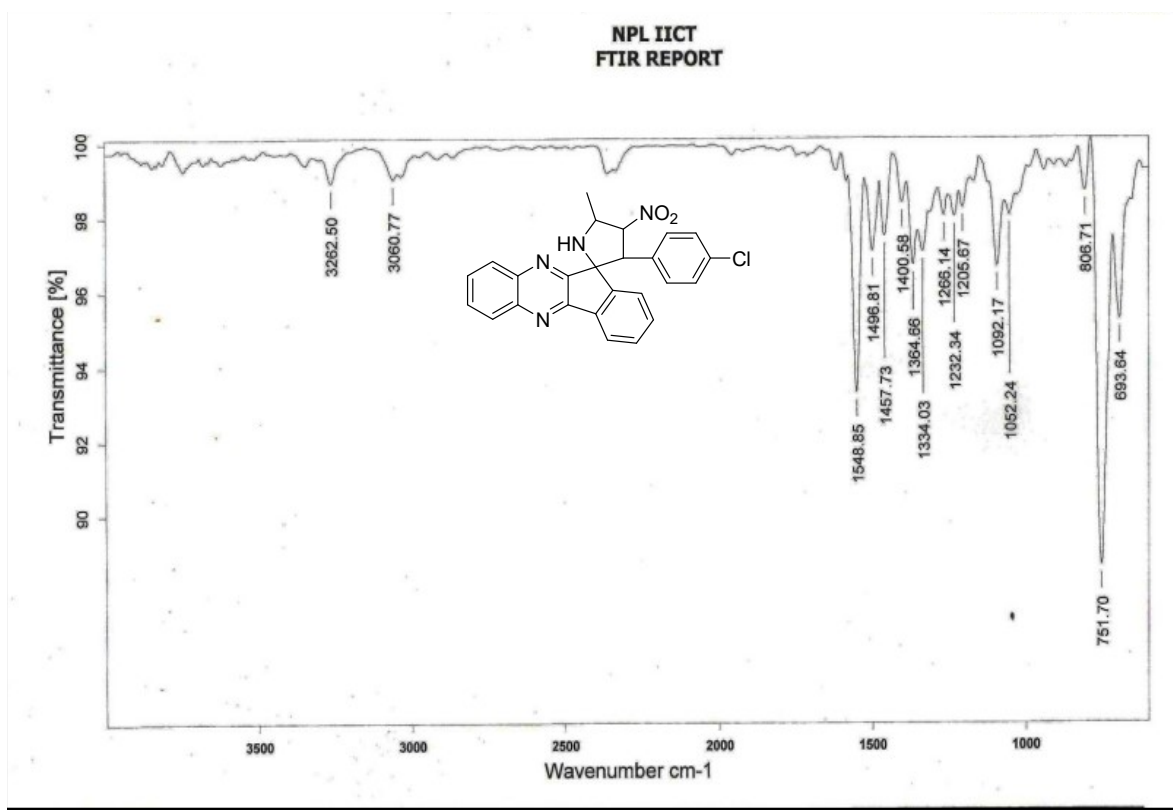
16 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)

Elements Used:

C: 0-27 H: 0-20 N: 0-4 O: 0-2 Cl: 0-1

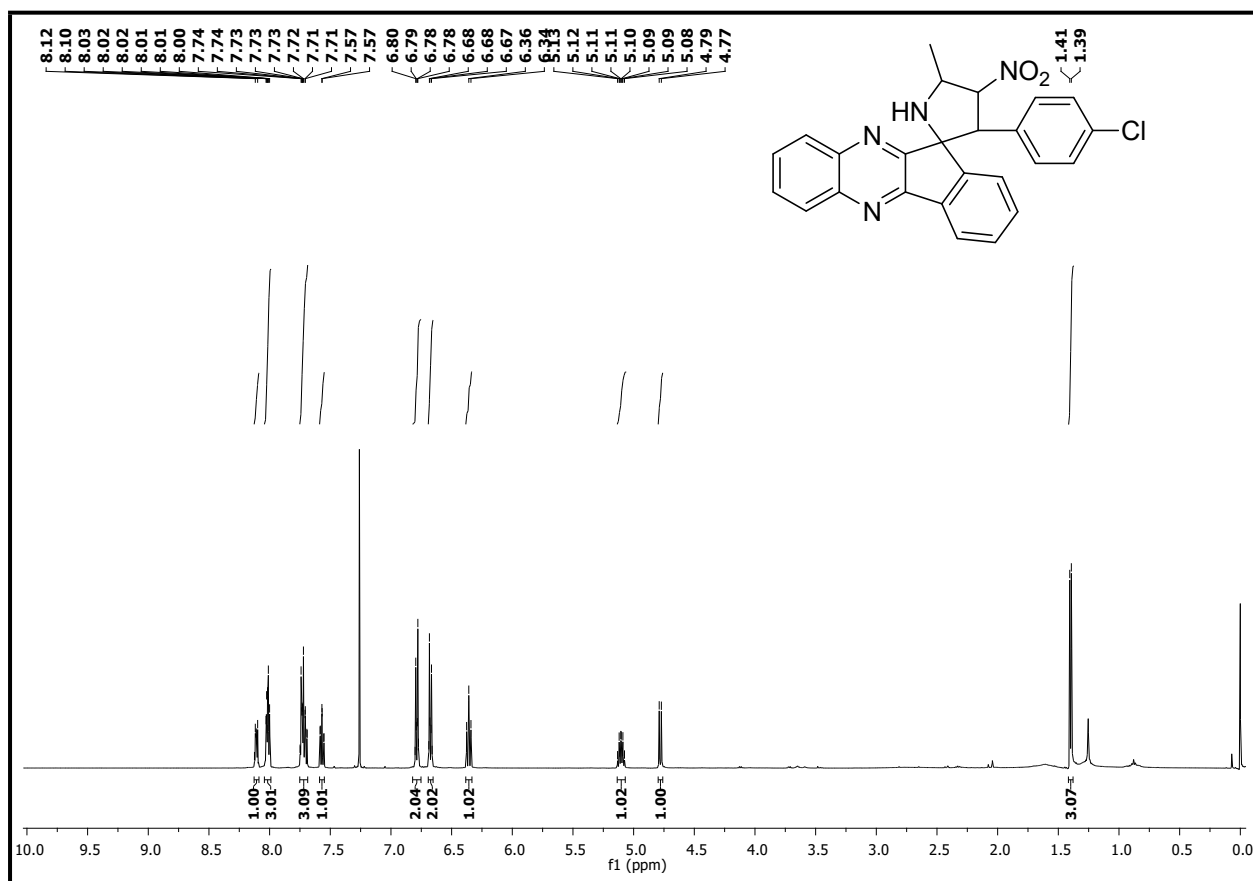
Minimum:	8.0	8.0	-1.5						
Maximum:	8.0	8.0	50.0						
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula	
443.1280	443.1275	0.5	1.1	17.5	276.8	n/a	n/a	C25 H20 N4 O2 Cl	

### IR Spectrum of compound 7a

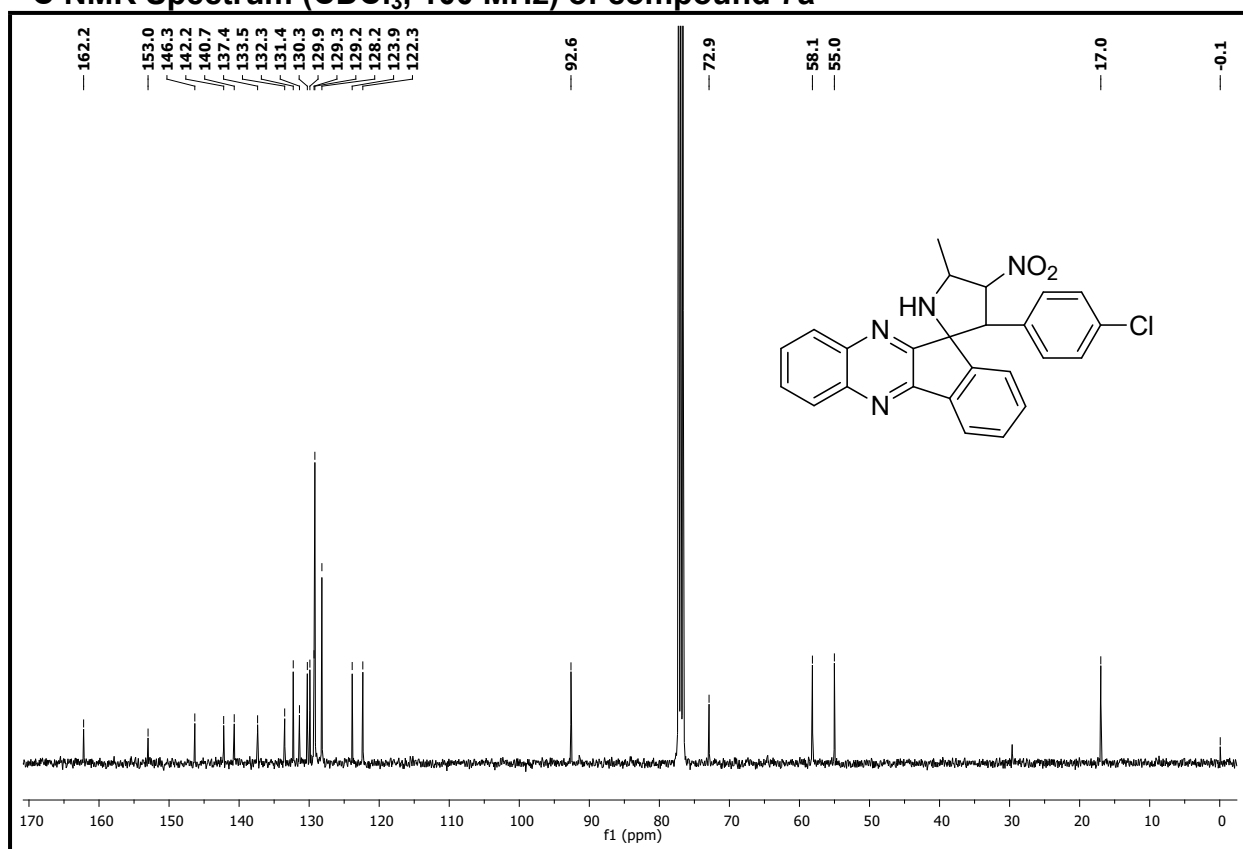


### <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>, 500 MHz) of compound 7a





**<sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>, 100 MHz) of compound 7a**

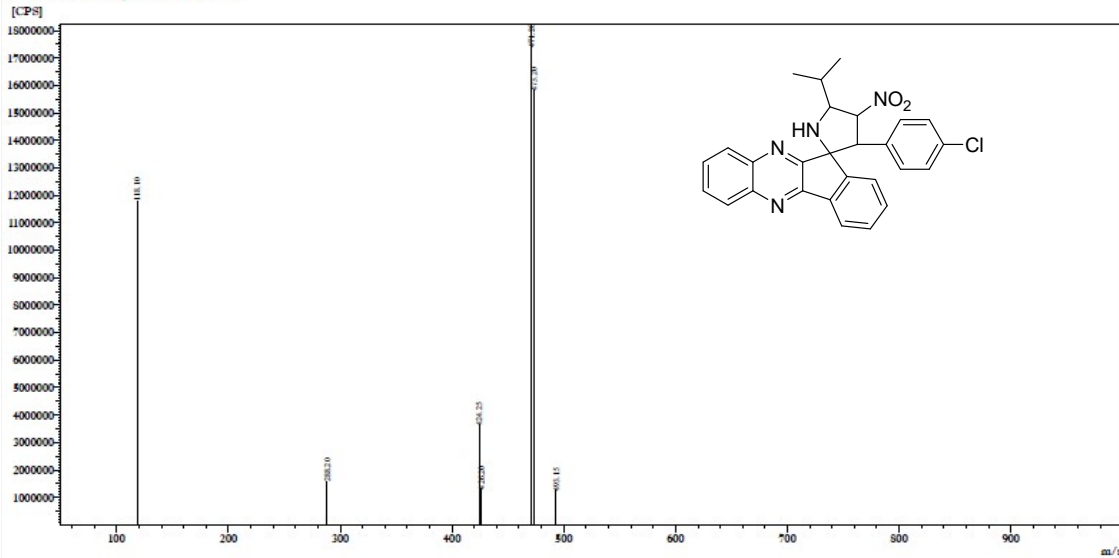


**ESIM Spectrum of compound 7b**

Sample Name : PMG-VCL-470  
Data File : 110418.50.icd  
Date Acquired : 4/11/2018 1:00:38 PM  
Batch File : 11-04-2018.icb

MS Spectrum  
D:\DATA\APRIL-2018\110418.50.icd

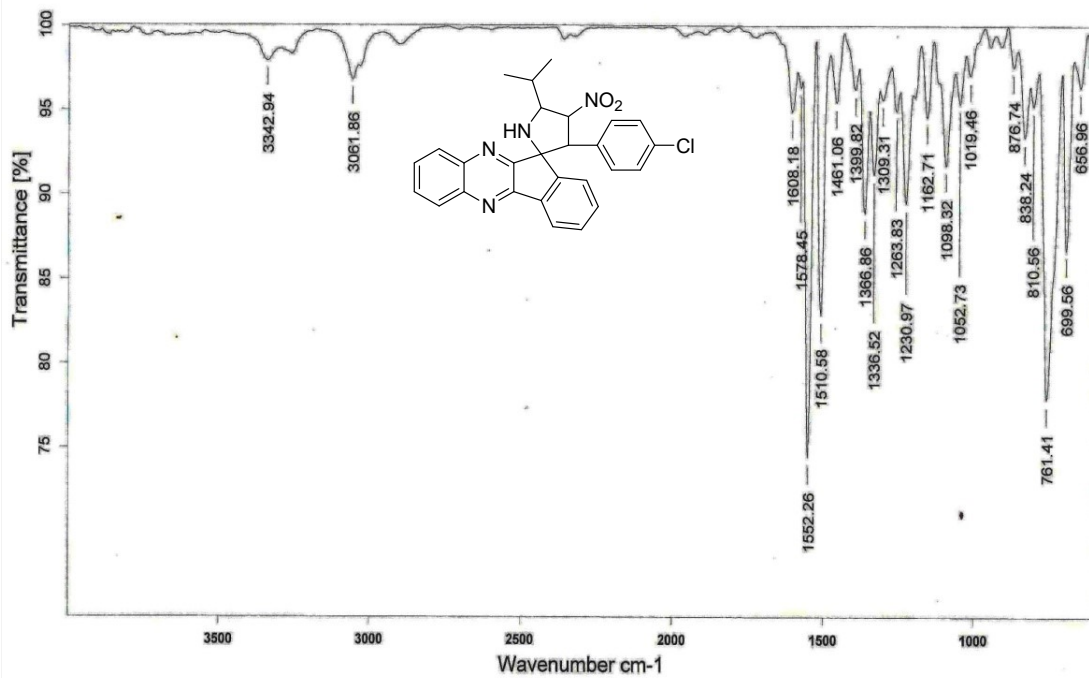
Averaged ESI Positive  
Spectrum Mode: Averaged 0.125-0.144(61-166)



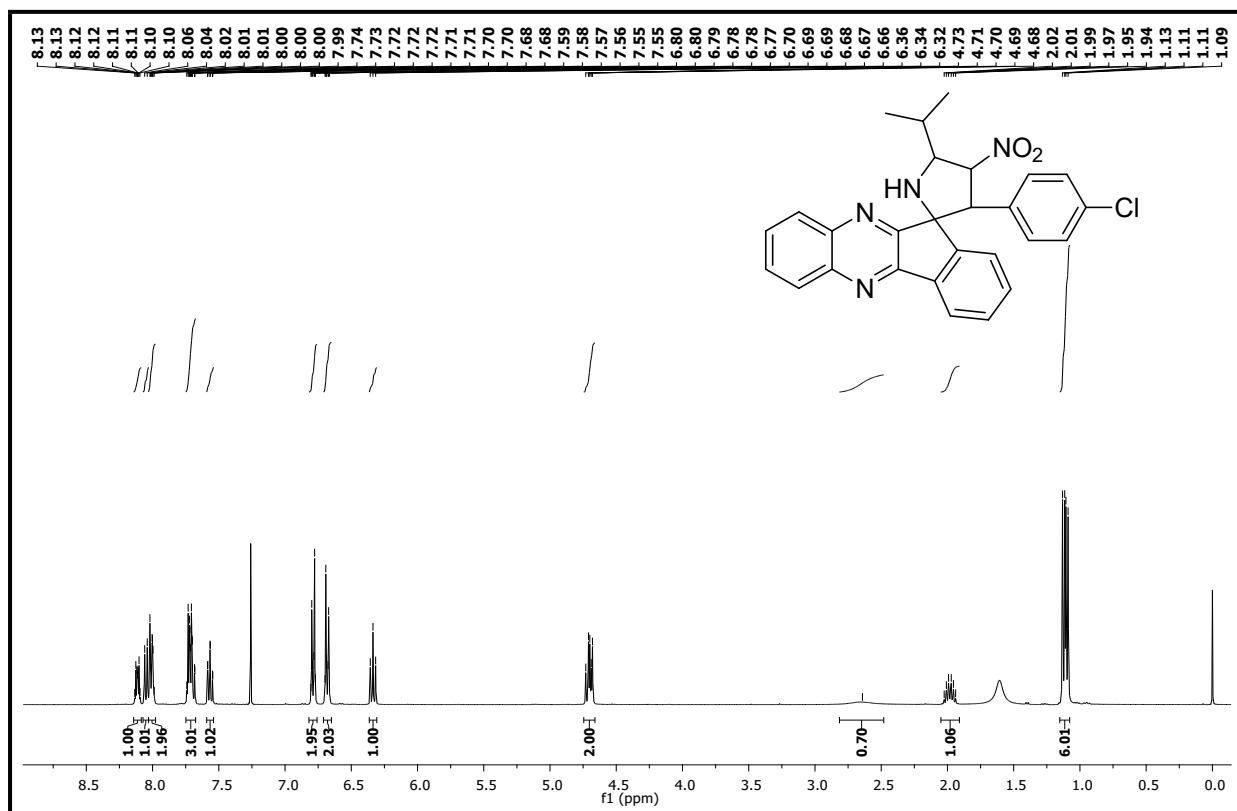
IR Spectrum of compound 7b

3/28/2017 12:46:22 PM

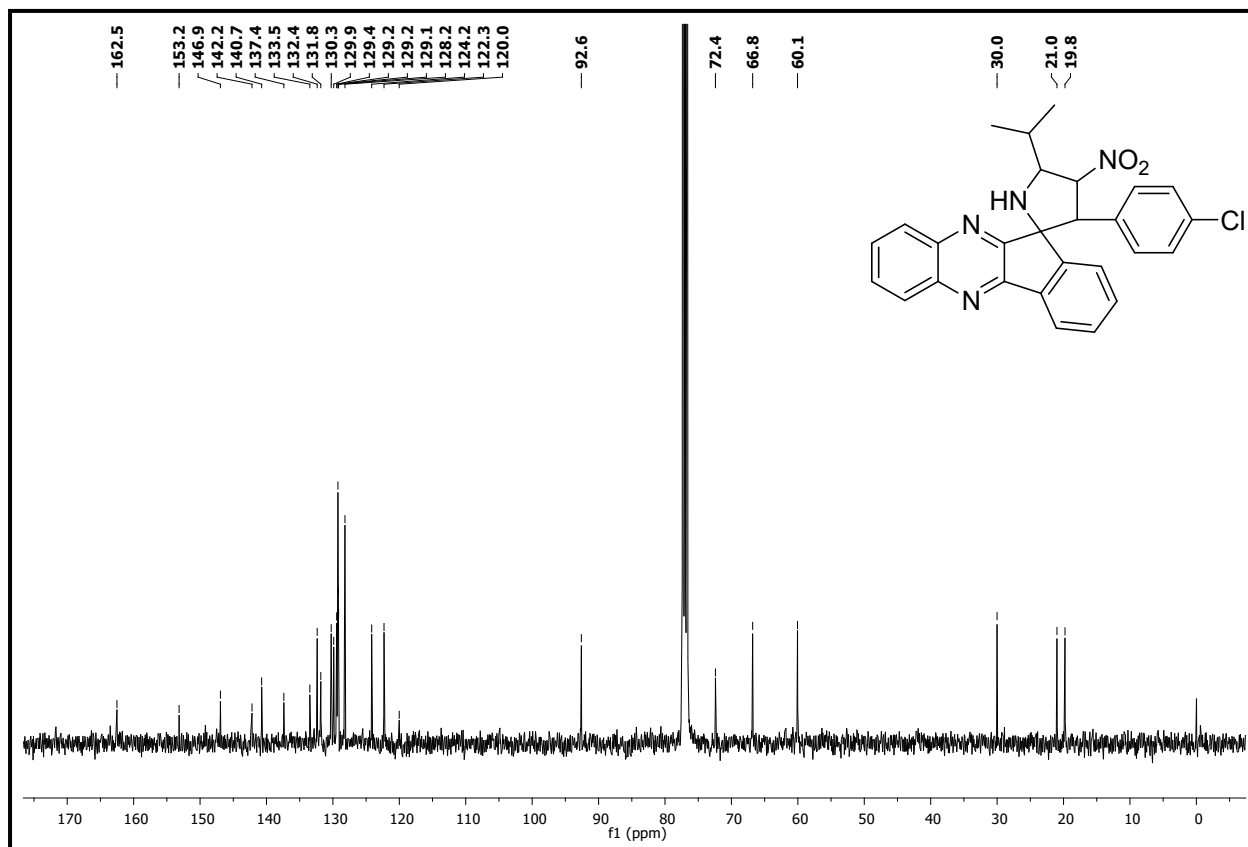
NPL IICT  
FTIR REPORT



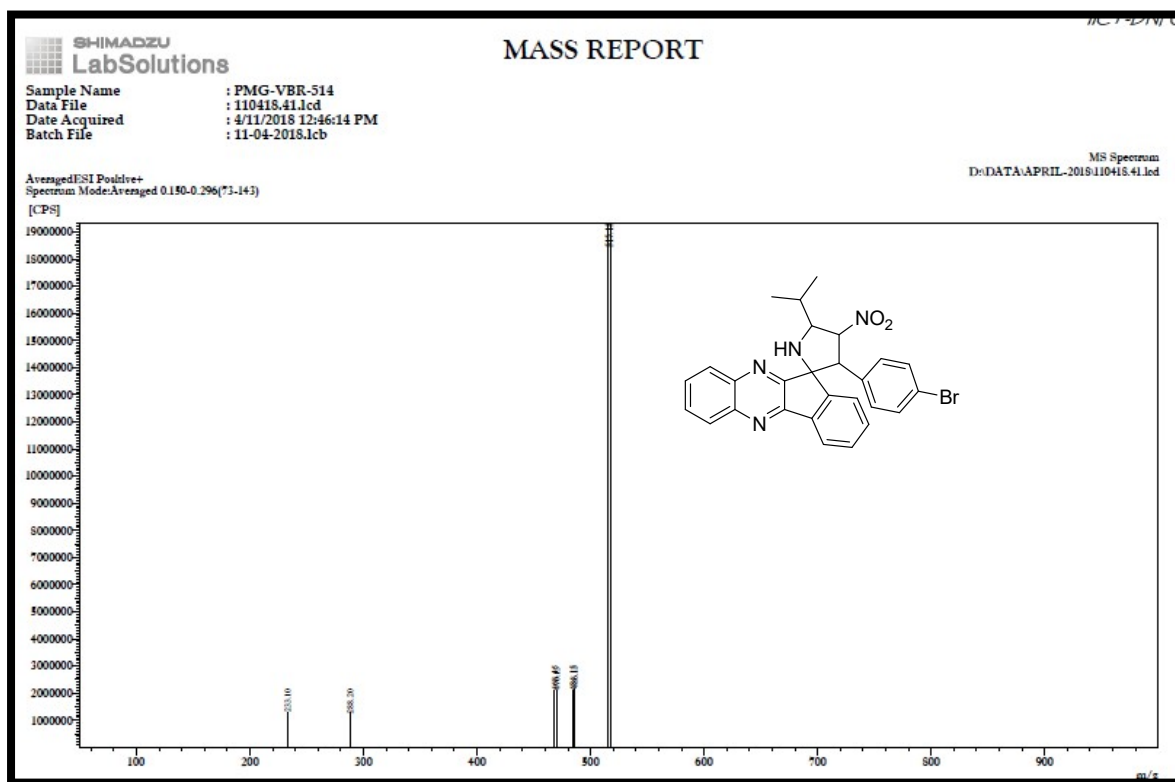
### <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>, 400 MHz) of compound 7b



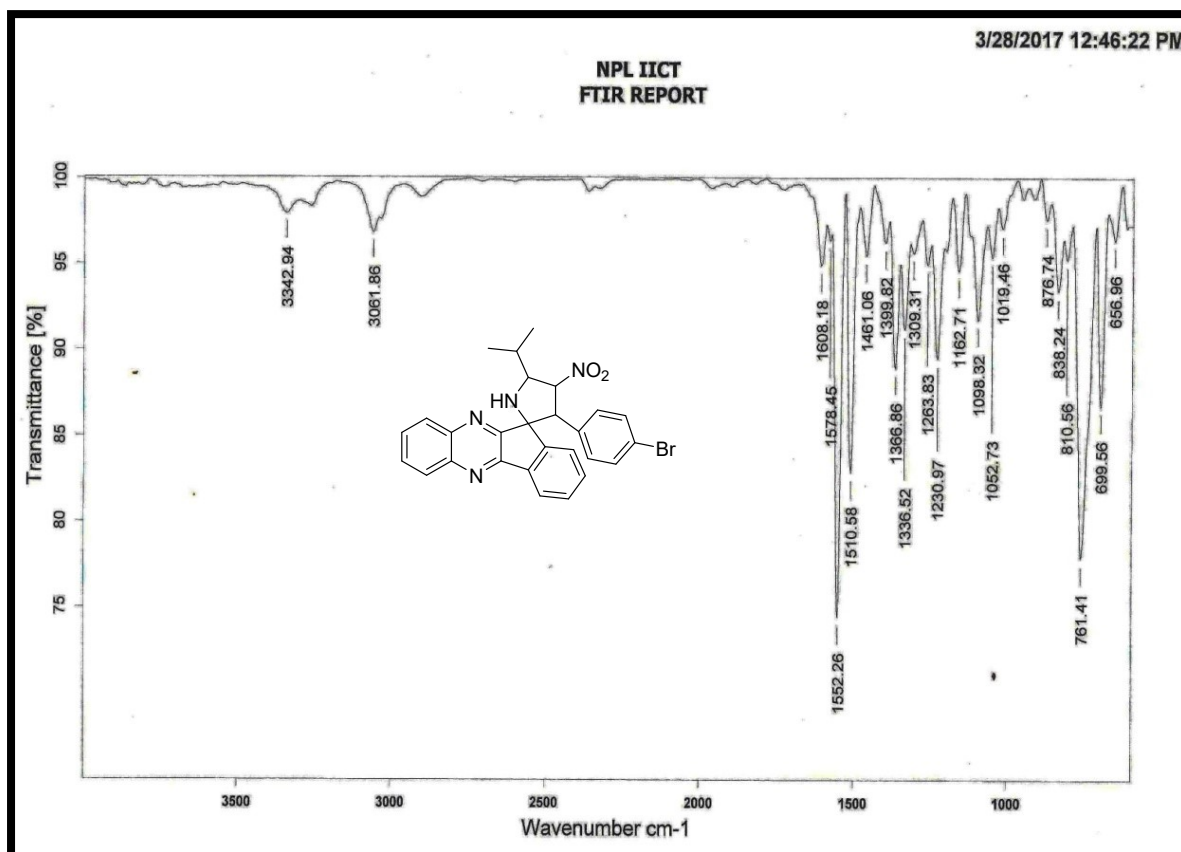
### <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>, 100 MHz) of compound 7b



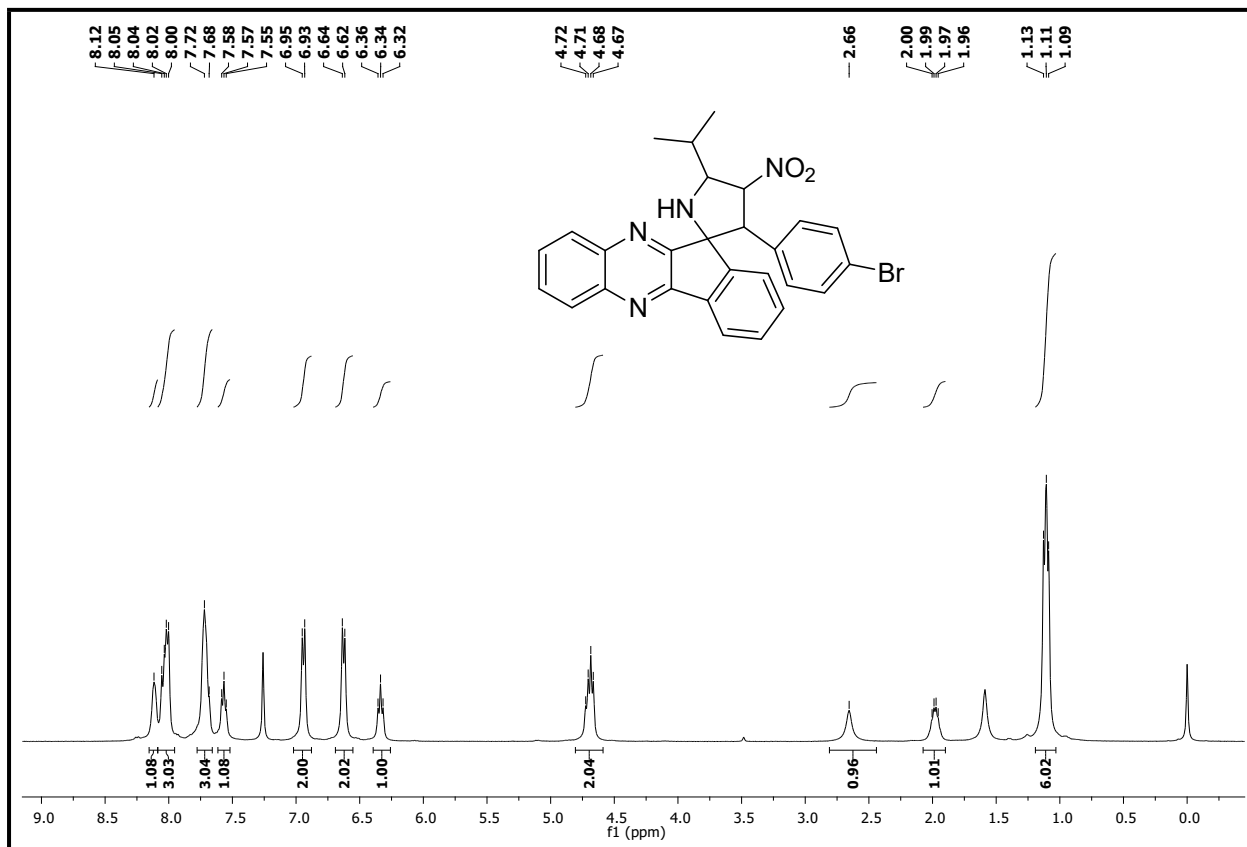
# ESIM Spectrum of compound 7c



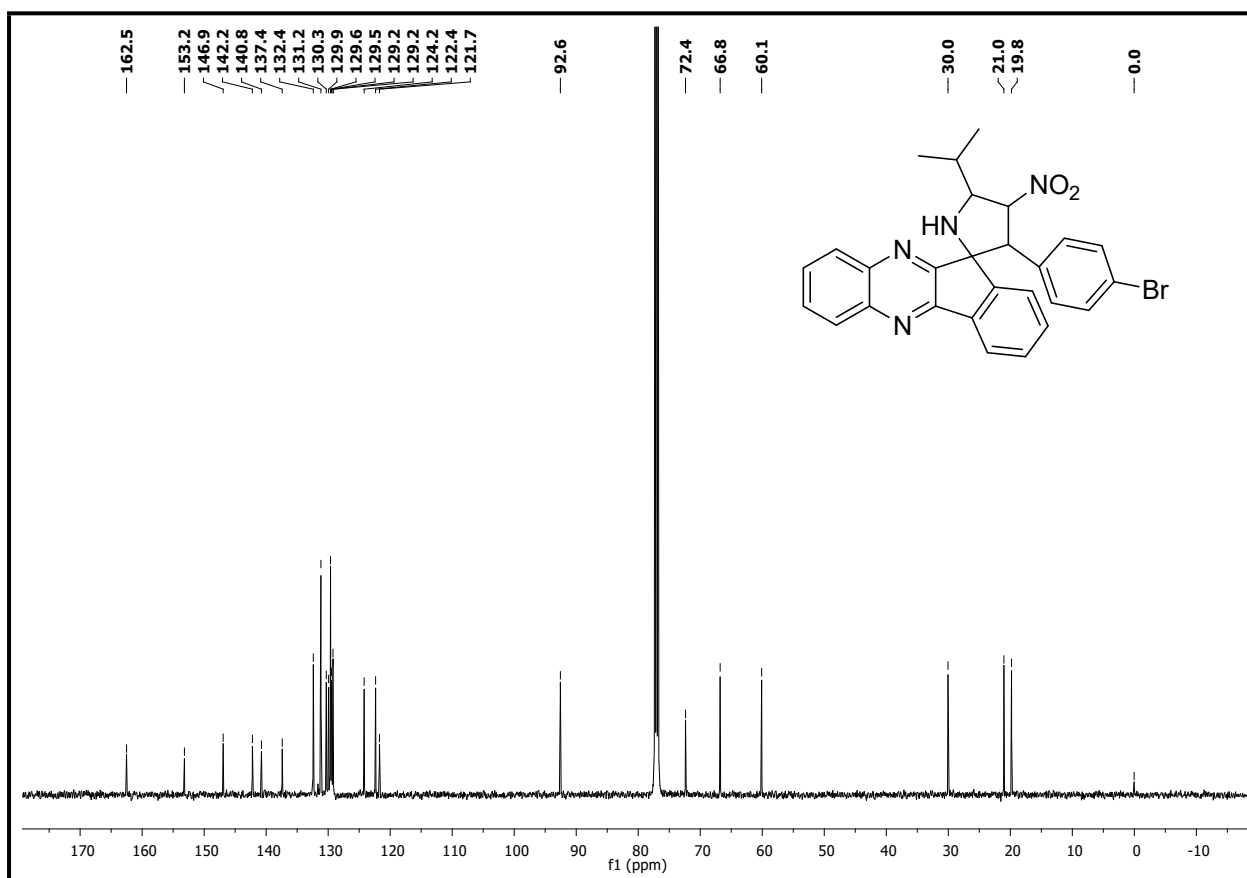
# IR Spectrum of compound 7c



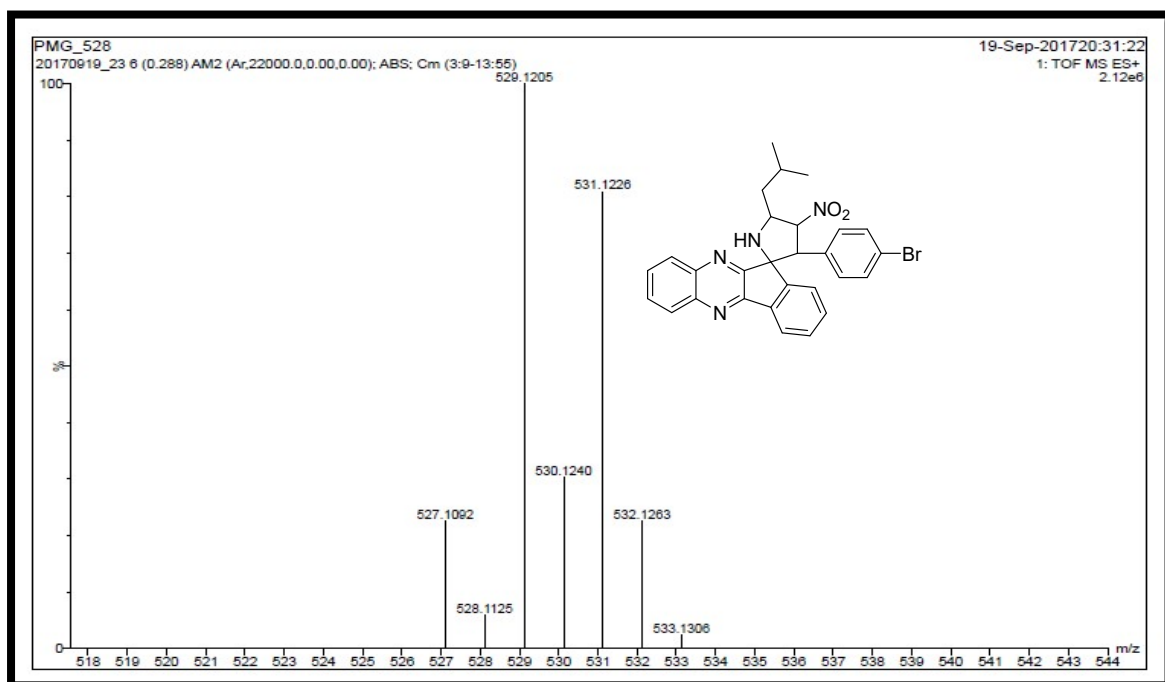
### <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>, 400 MHz) of compound 7c



### <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>, 100 MHz) of compound 7c



## HR-ESIM Spectrum of compound 7d



### Elemental Composition Report

#### Single Mass Analysis

Tolerance = 20.0 PPM / DBE: min = 1.0, max = 50.0  
Element prediction: Off  
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

99 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)

Elements Used:

C: 0-28 H: 0-27 N: 0-5 O: 0-5 Br: 0-1

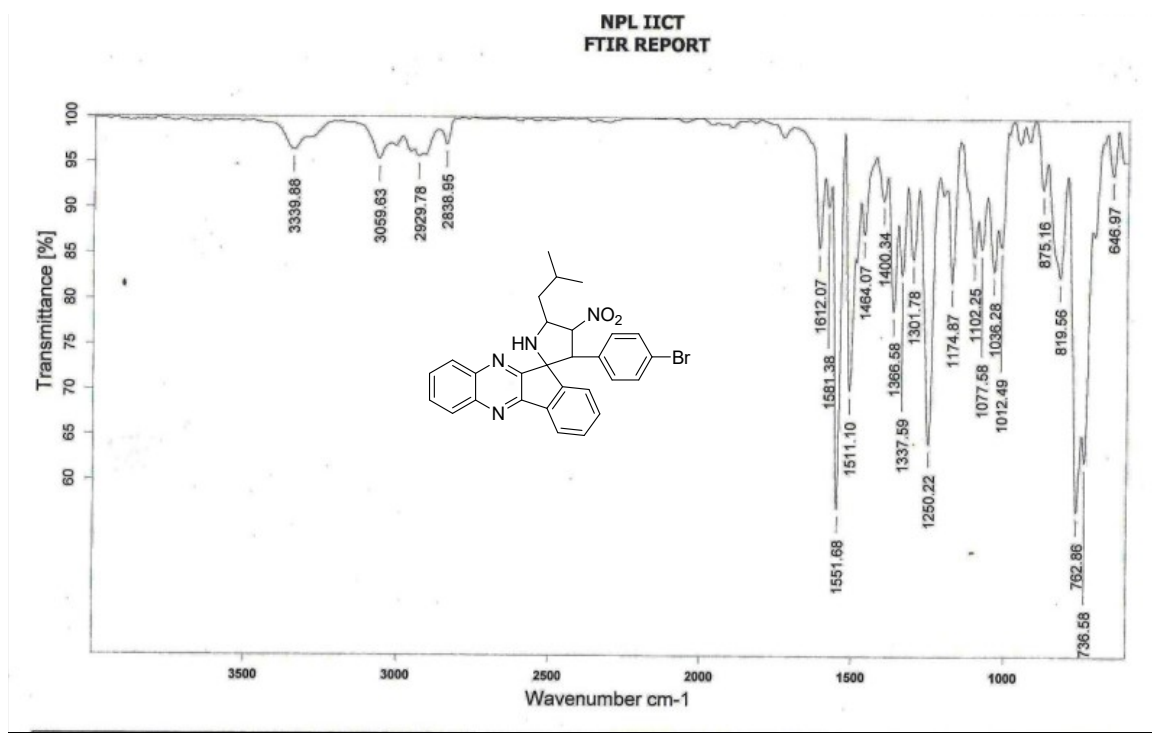
PMG\_528  
20170919\_23 6 (0.288) AM2 (Ar,22000.0,0.00,0.00); ABS: Cm (3:9-13:55)

19-Sep-201720:31:22

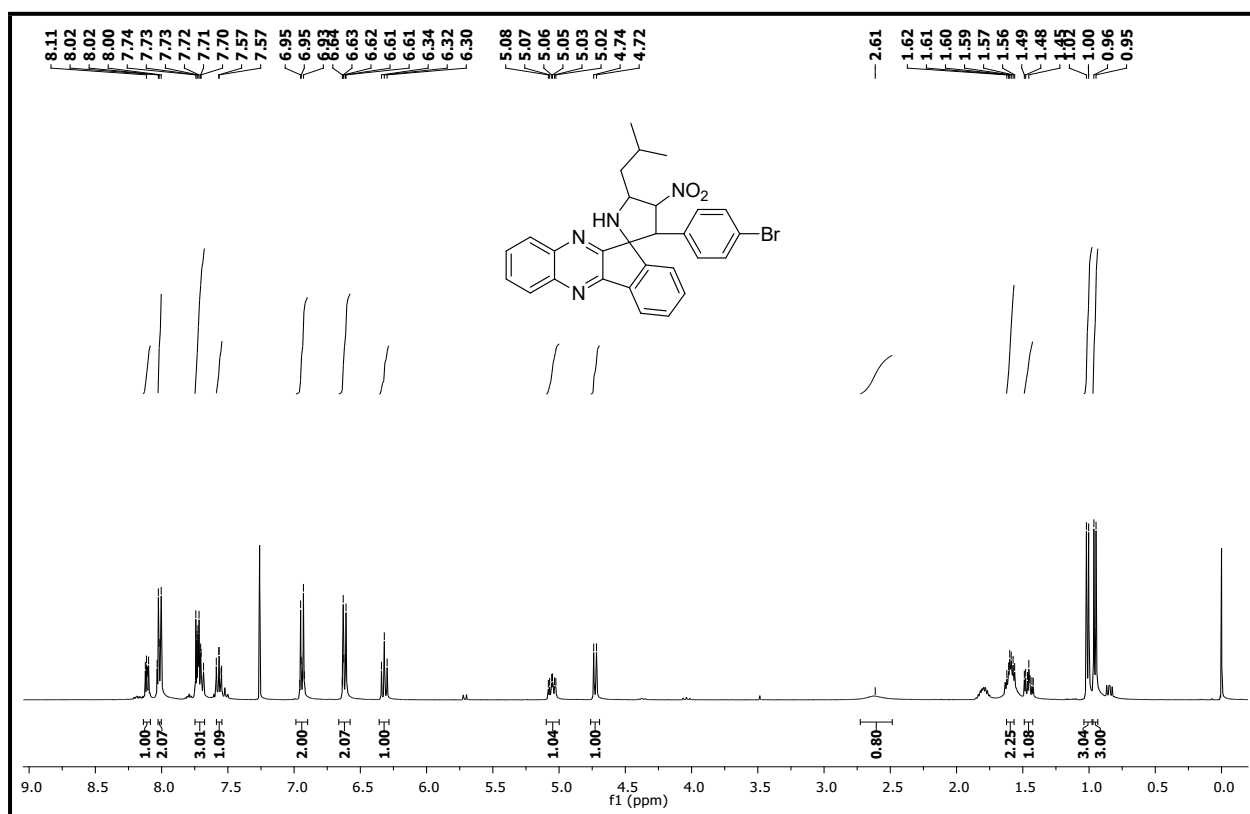
1: TOF MS ES+  
2.12e+006

m/z	527.0	527.1092	528.0	528.1125	529.0	529.1205	530.0	530.1240	531.0	531.1226	532.0	532.1263	533.0	533.1306	538.0	538.2173
Minimum:			5.0		20.0		10.0		50.0							
Maximum:																
Mass		Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula							
529.1205		529.1239	-3.4	-6.4	17.5	37.5	n/a	n/a	C28 H26 N4 O2 Br							

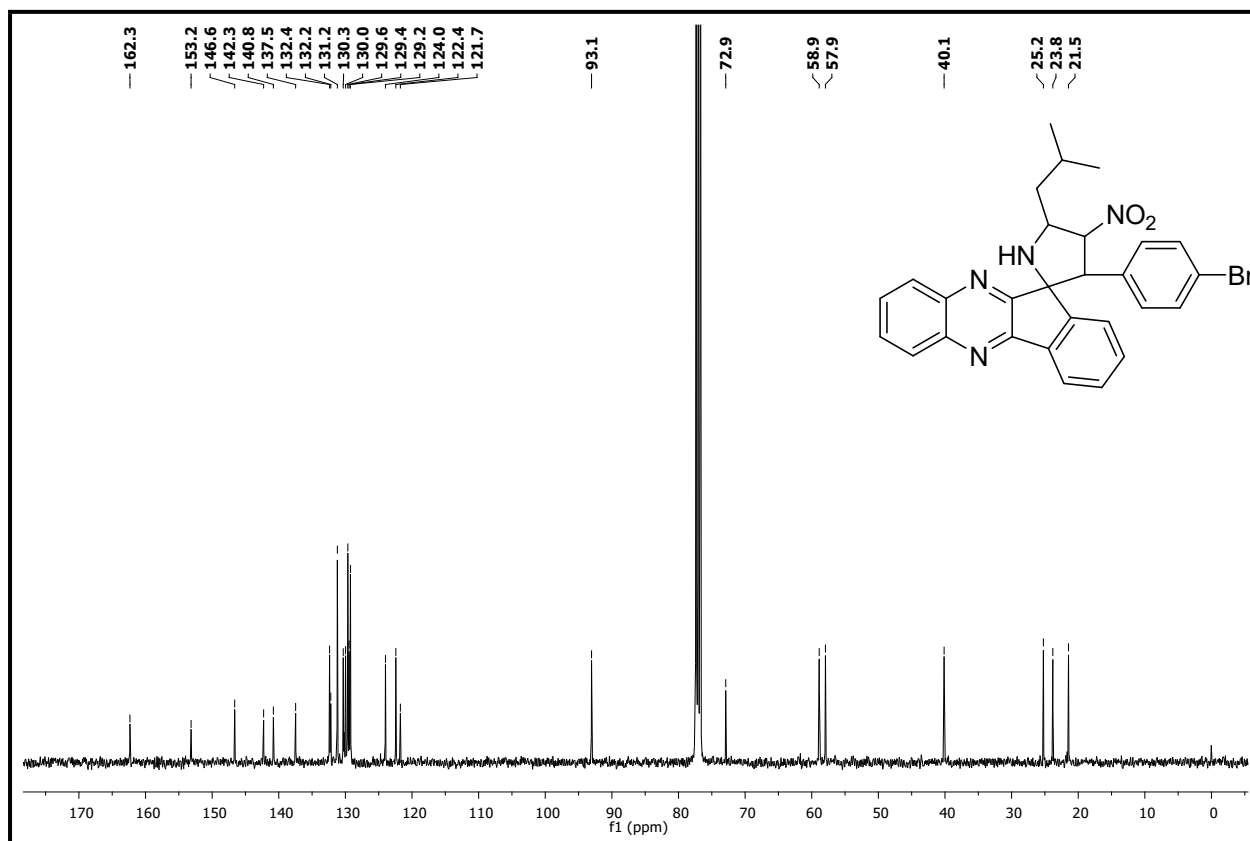
## R Spectrum of compound 7d



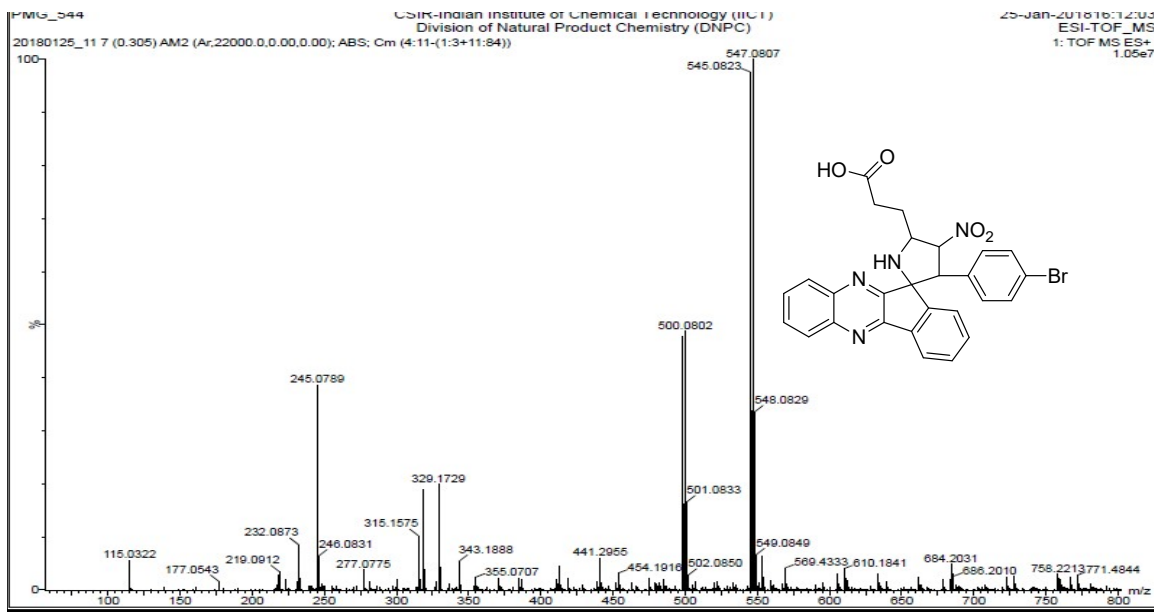
### <sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>, 400 MHz) of compound 7d



### <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>, 100 MHz) of compound 7d



## HR-ESIM Spectrum of compound 7e



### Single Mass Analysis

Tolerance = 5.0 ppm / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

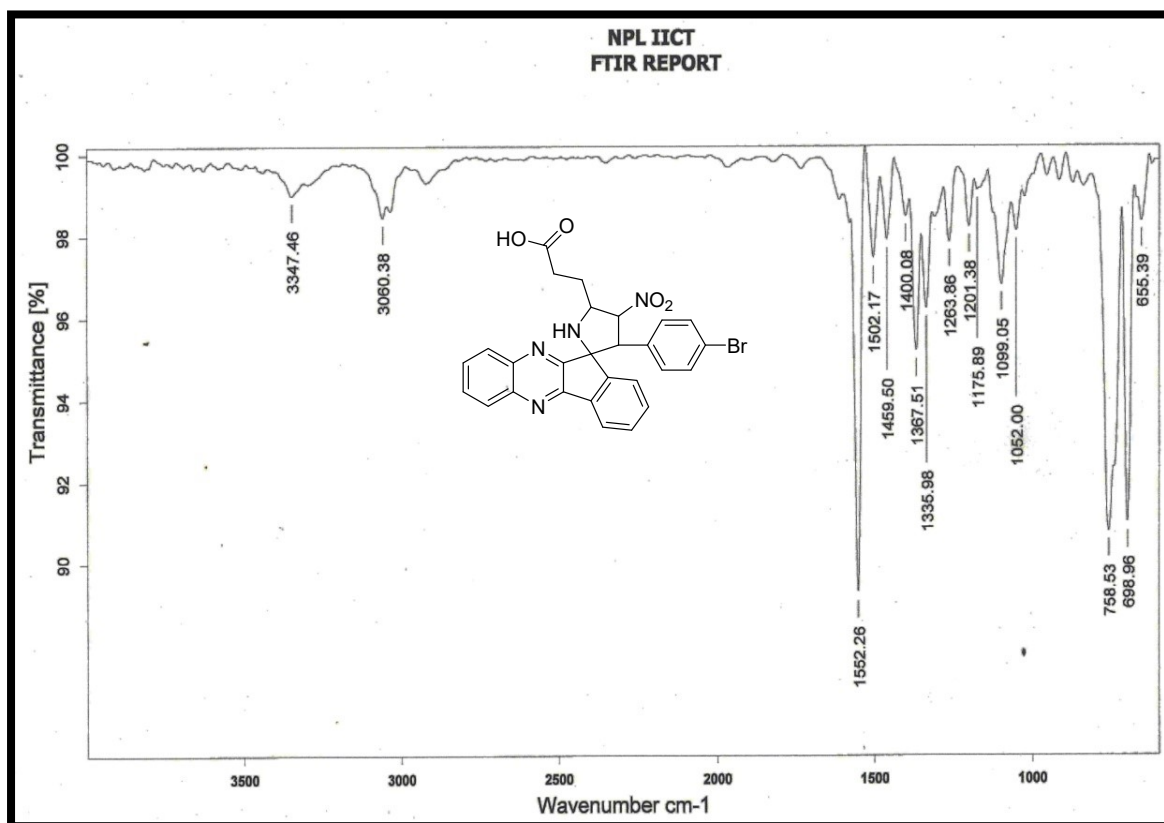
50 formulae evaluated with 1 results within limits (up to 10 closest results for each mass)

Elements Used:

C: 0-27 H: 0-22 N: 0-4 O: 0-5 Br: 0-1

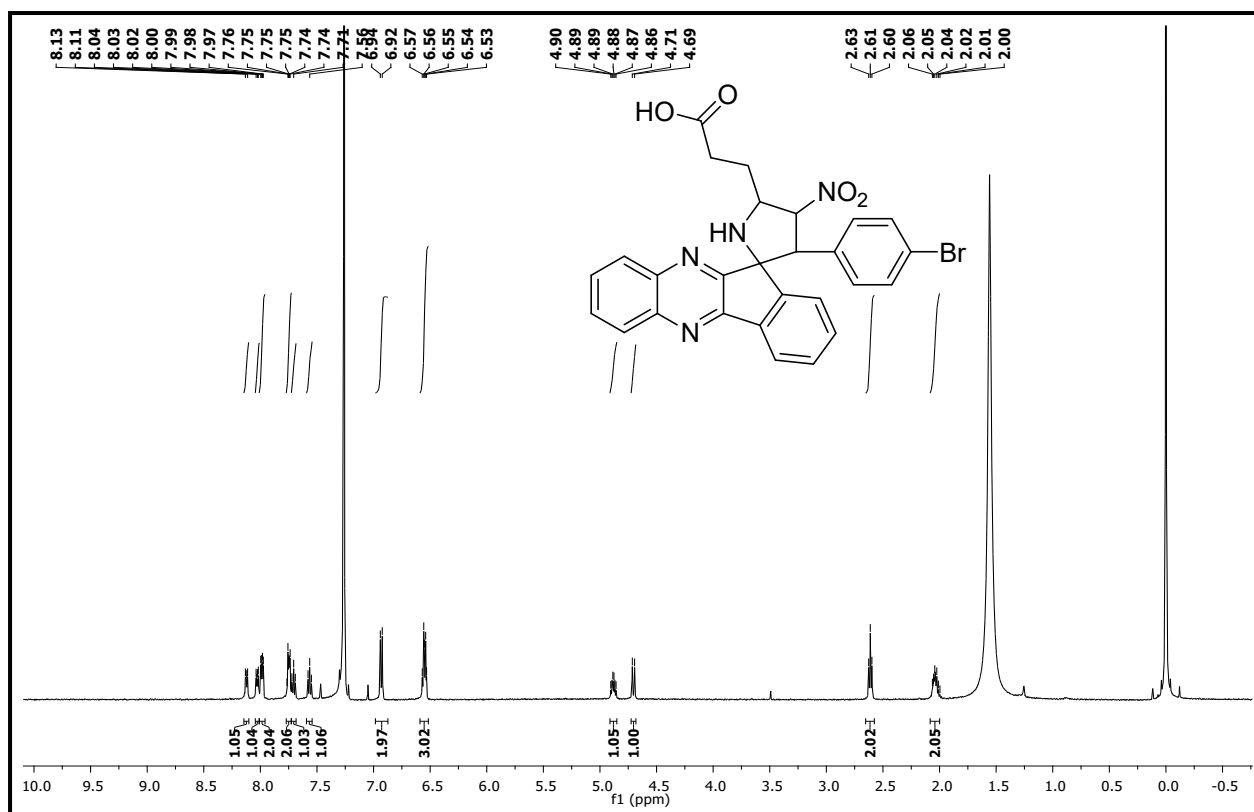
Mass	Calc. Mass	mDa	ppm	DBE	i-FIT	Norm	Conf (%)	Formula
545.0823	545.0824	-0.1	-0.2	18.5	102.7	n/a	n/a	C27 H22 N4 O4 Br

## IR Spectrum of compound 7e

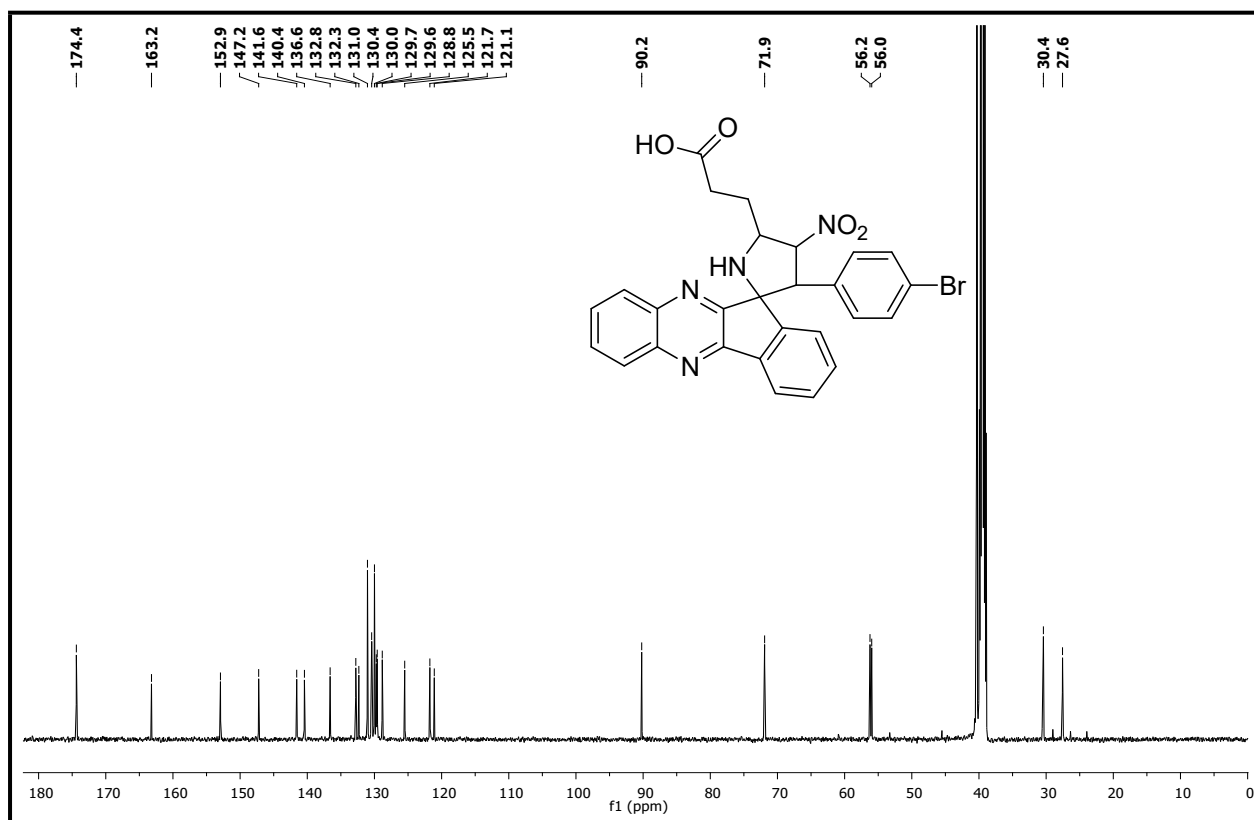




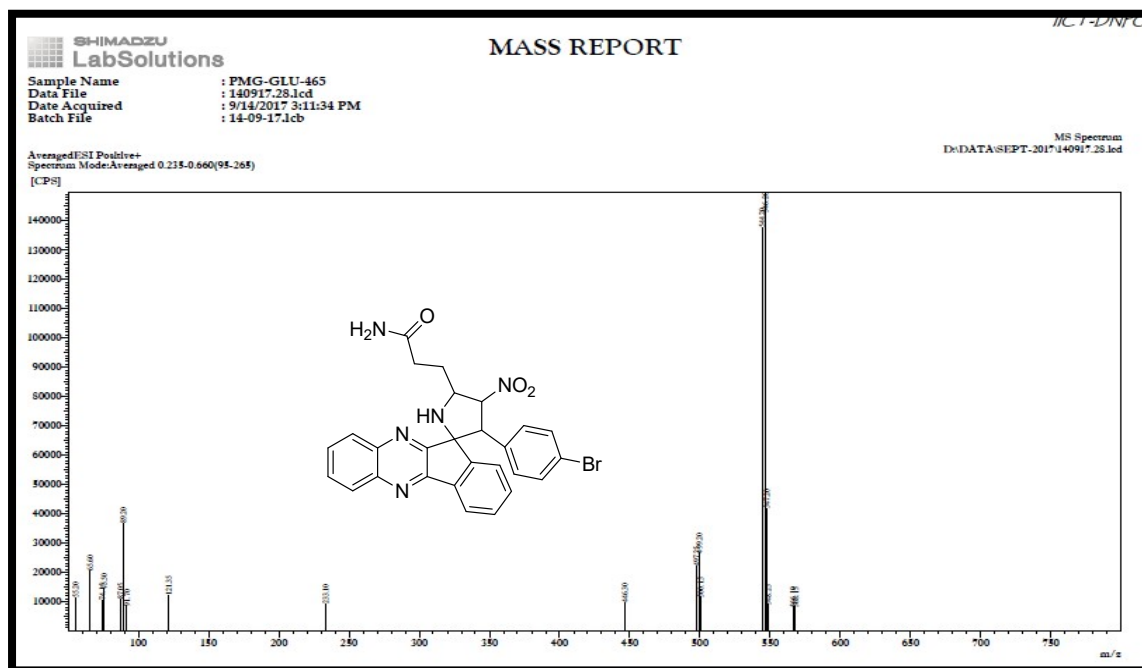
**<sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>, 400 MHz) of compound 7e**



**<sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>, 100 MHz) of compound 7e**



## ESIM Spectrum of compound 7f



**Elemental Composition Report**

Page 1

**Single Mass Analysis**

Tolerance = 20.0 PPM / DBE: min = 1.0, max = 50.0  
 Element prediction: Off  
 Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions  
 65 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)  
 Elements Used: C: 0-27 H: 0-25 N: 0-5 O: 0-5 Br: 0-1

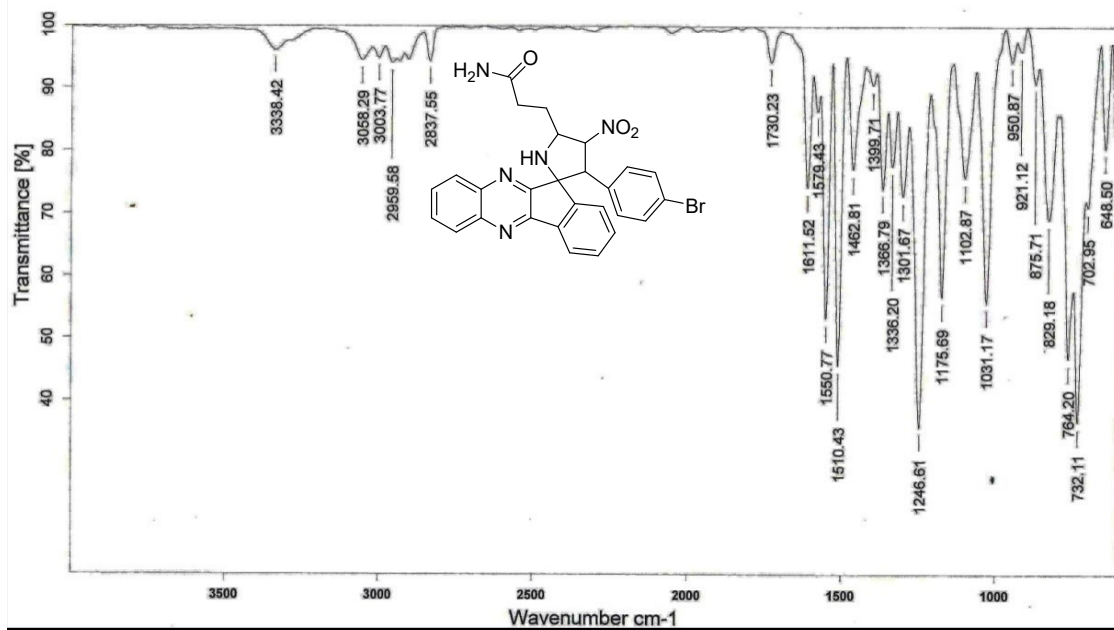
PMG GLU 543  
 20170919\_10 6 (0.288) AM2 (Ar,22000.0,0.00,0.00); ABS; Cm (2:10-18:36)

19-Sep-2017 19:52:11  
 1: TOF MS ES+  
 9.40e+005

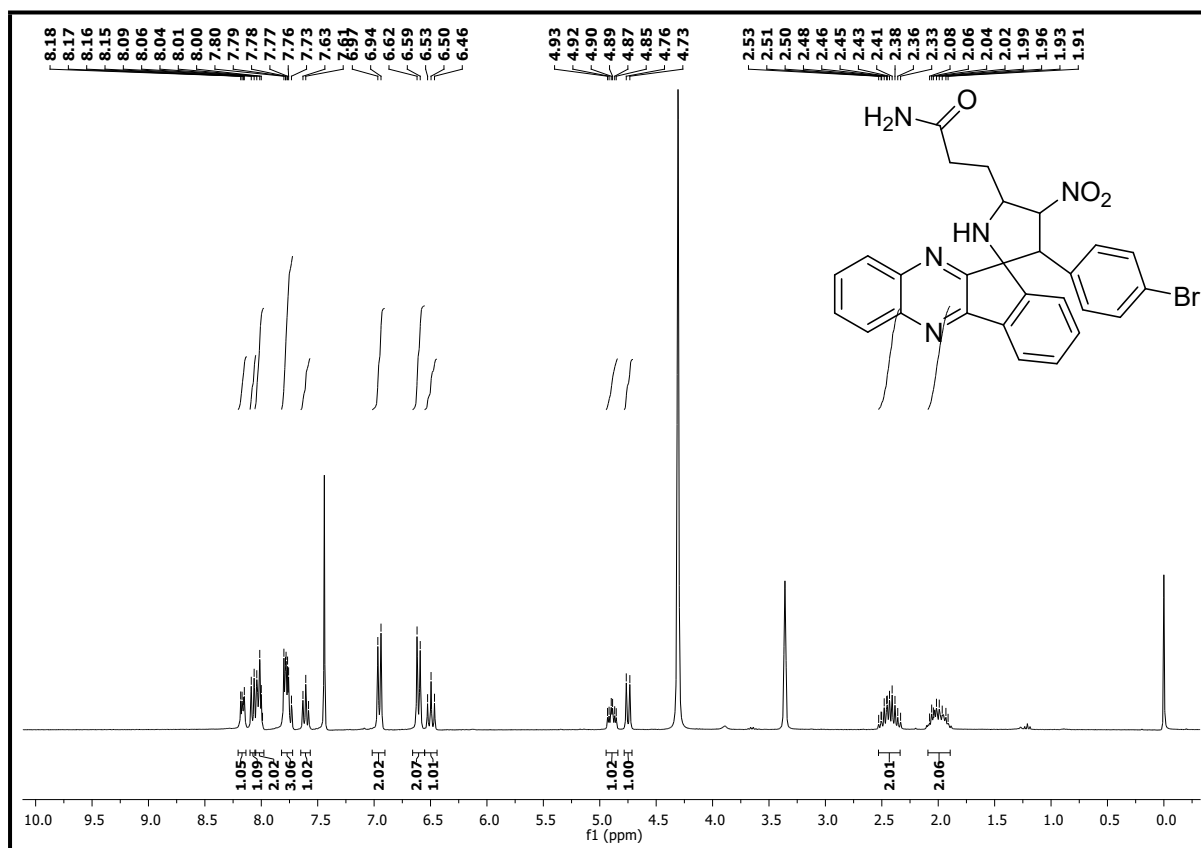
m/z	544.0988	544.2347	545.1025	545.7654	546.0974	546.2590	547.1008	547.2247	547.7676	547.9371
Minimum	5.0	20.0	1.0							
Maximum			50.0							
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula		
544.0988	544.0984	0.4	0.7	18.5	68.9	n/a	n/a	C27 H23 N5 O3 Br		

## IR Spectrum of compound 7f

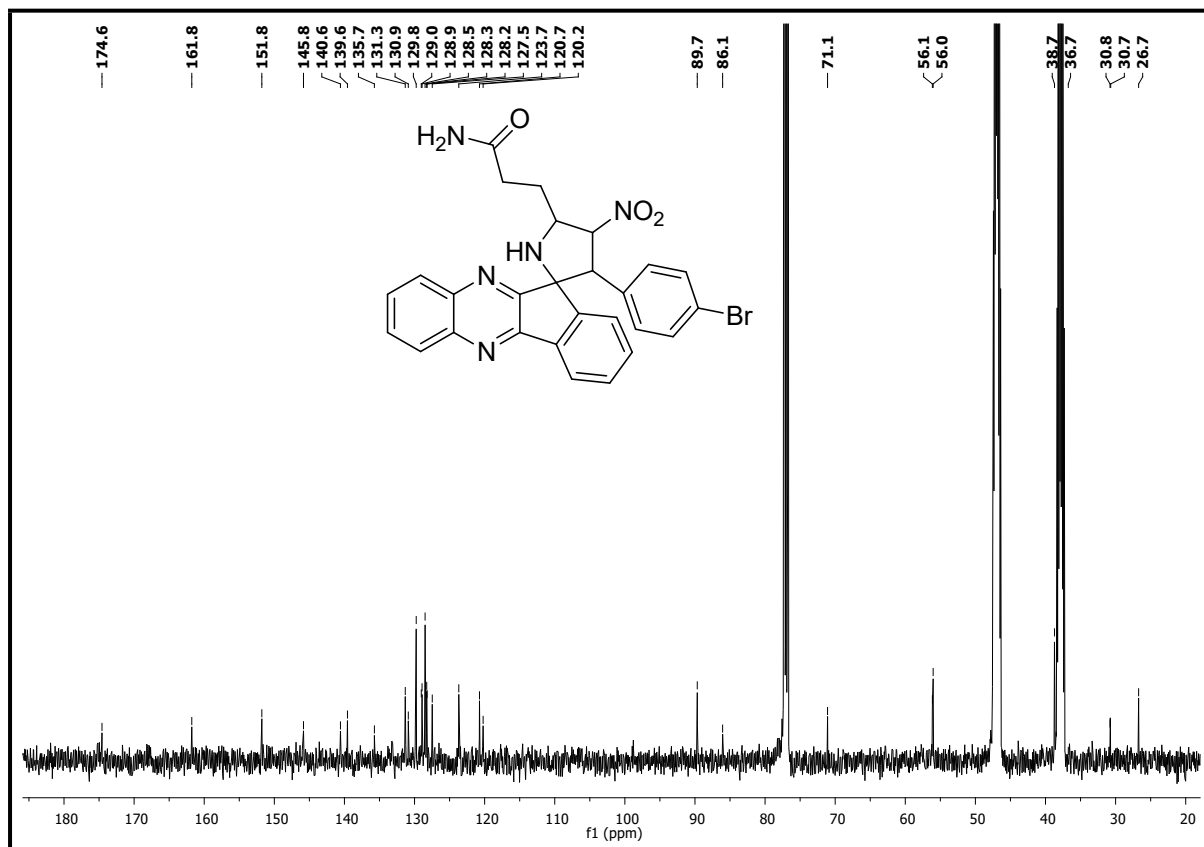
NPL ICT  
FTIR REPORT



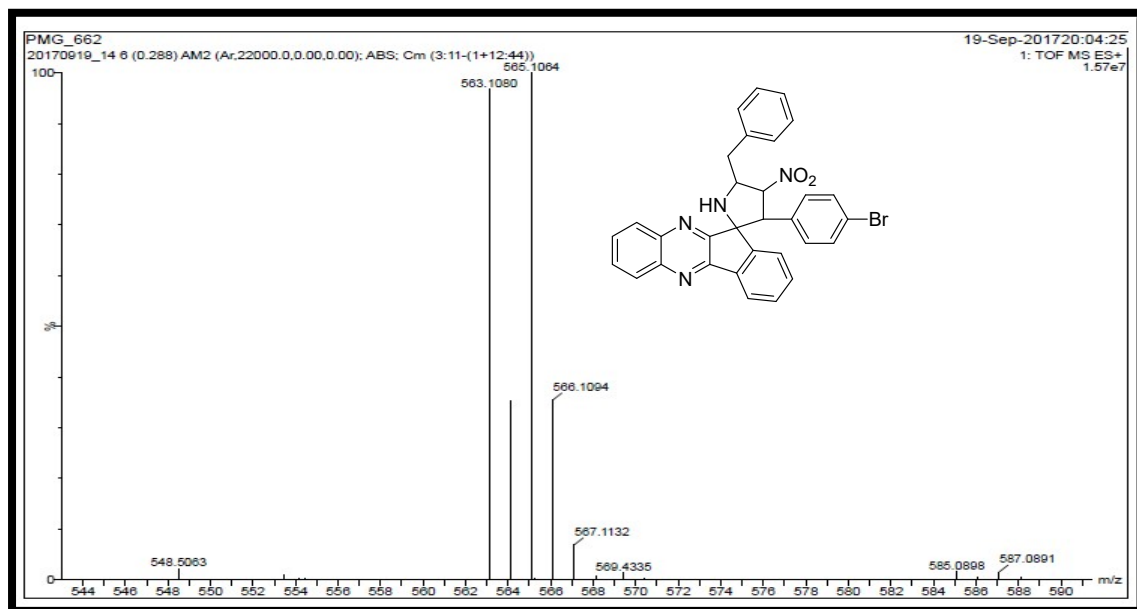
<sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>+CD<sub>3</sub>OD, 400 MHz) of compound 7f



<sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>+CD<sub>3</sub>OD+DMSO-d<sub>6</sub>, 100 MHz) of compound 7f



HR-ESIM Spectrum of compound 7g



Elemental Composition Report

Page 1

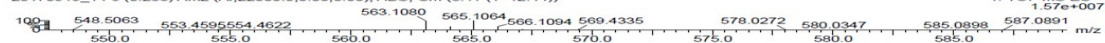
Single Mass Analysis

Tolerance = 20.0 PPM / DBE: min = 1.0, max = 50.0  
 Element prediction: Off  
 Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions  
 64 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)  
 Elements Used:  
 C: 0-31 H: 0-27 N: 0-5 O: 0-5 Br: 0-1

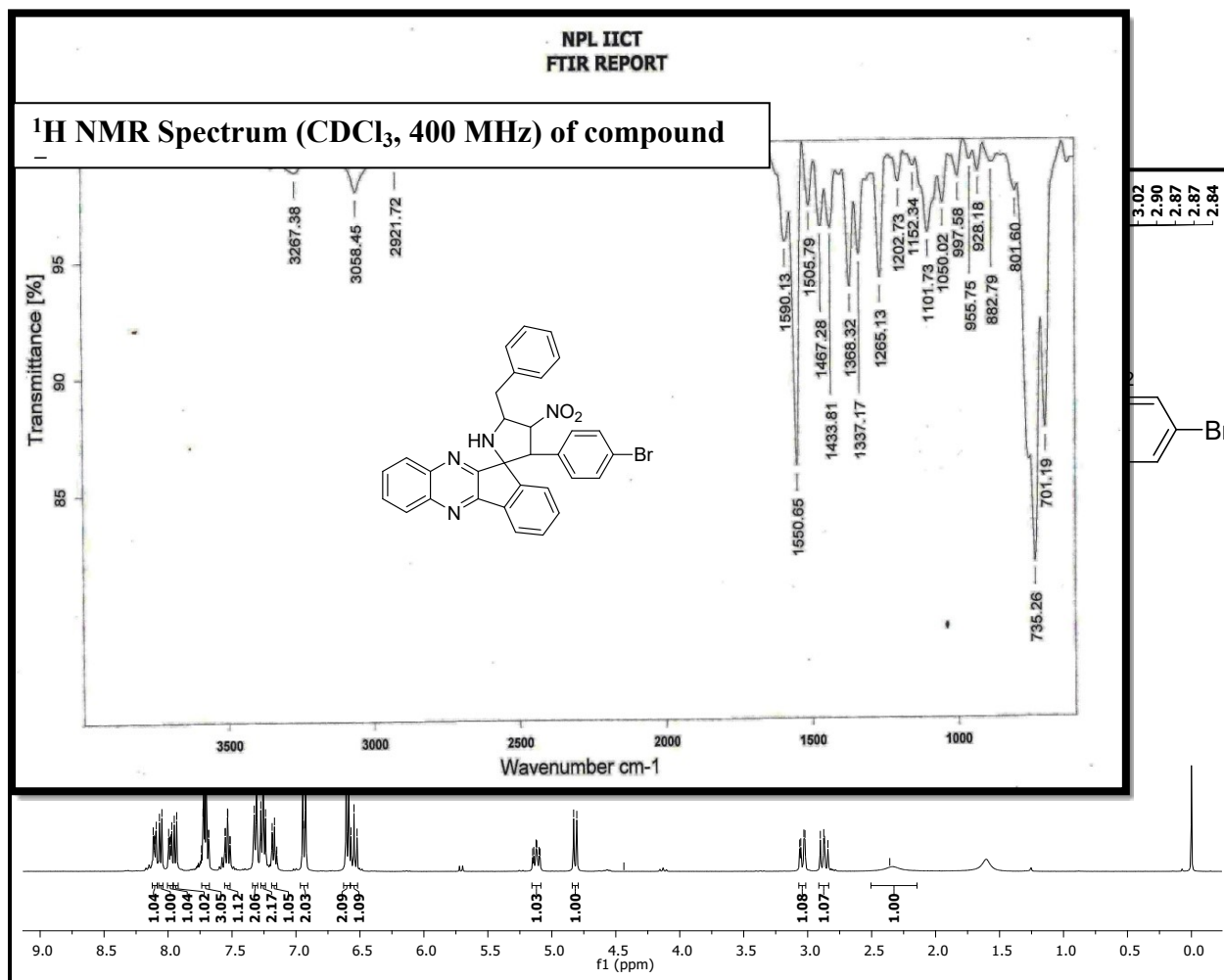
PMG\_662  
 20170919\_14 6 (0.288) AM2 (Ar,22000.0,0.00,0.00); ABS; Cm (3:11-(1+12:44))

19-Sep-201720:04:25  
 1: TOF MS ES+  
 1.57e+007

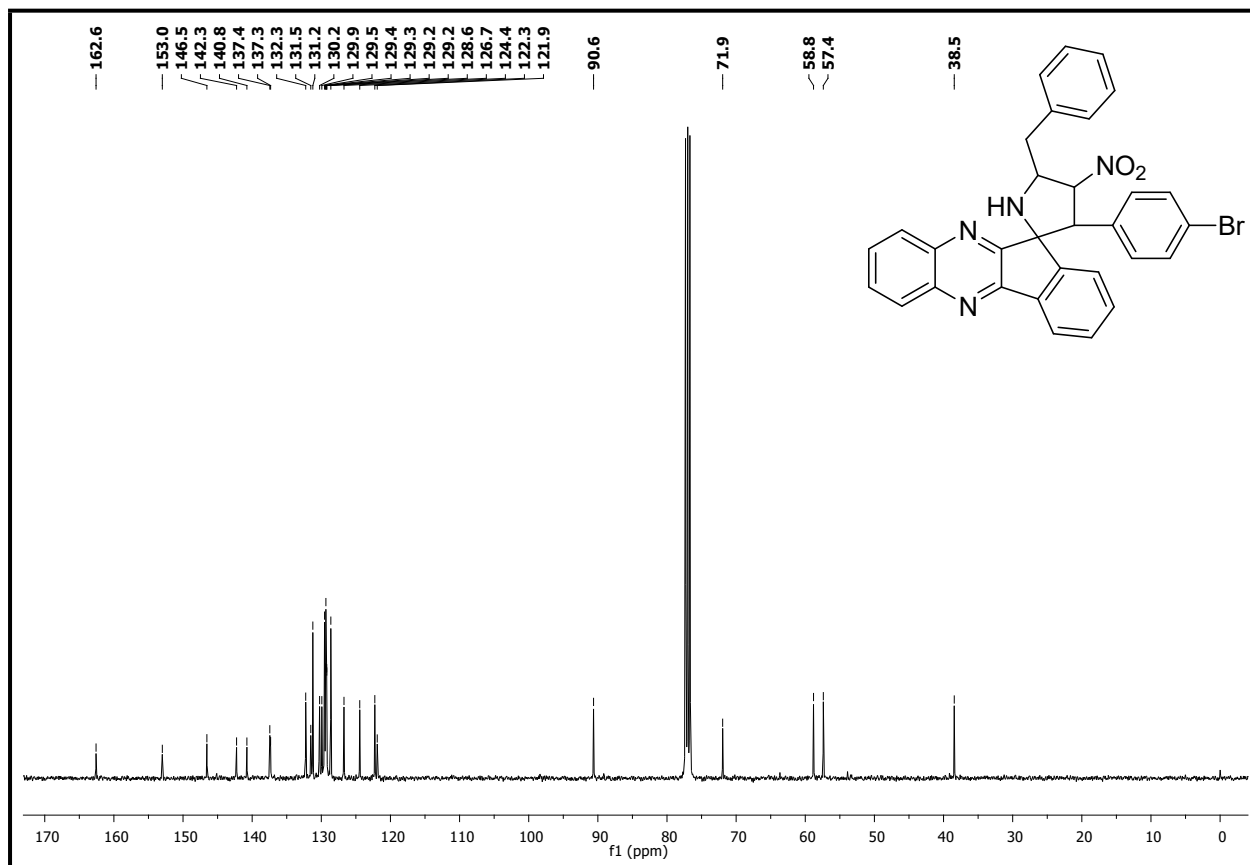


Mass	Calc. Mass	Mass mDa	PFM	DBE	i-FIT	Norm	Conf (%)	Formula
563.1080	563.1083	-0.3	-0.5	21.5	67.9	n/a	n/a	C31 H24 N4 O2 Br

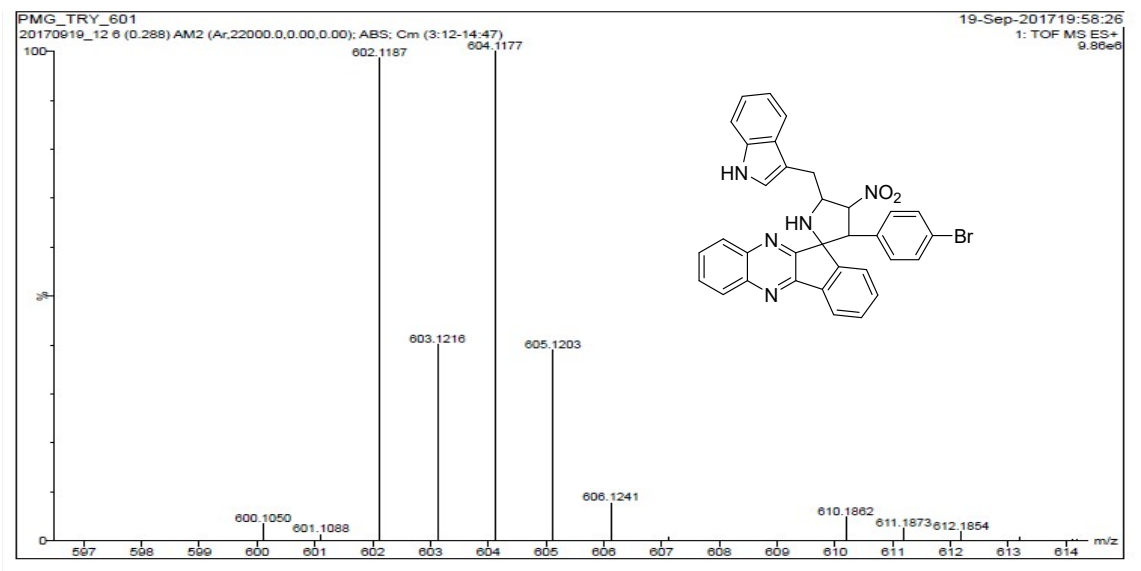
## IR Spectrum of compound 7g



## <sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>, 100 MHz) of compound 7g



### HR-ESIM Spectrum of compound 7h



Elemental Composition Report

Single Mass Analysis

Tolerance = 20.0 PPM / DBE: min = 1.0, max = 50.0

Element prediction: Off

Number of isotope peaks used for I-FIT = 3

Monoisotopic Mass, Even Electron Ions

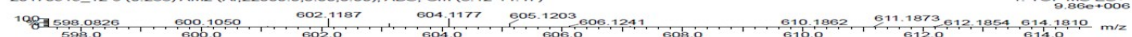
60 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)

Elements Used:

C: 0-33 H: 0-25 N: 0-5 O: 0-5 Br: 0-1

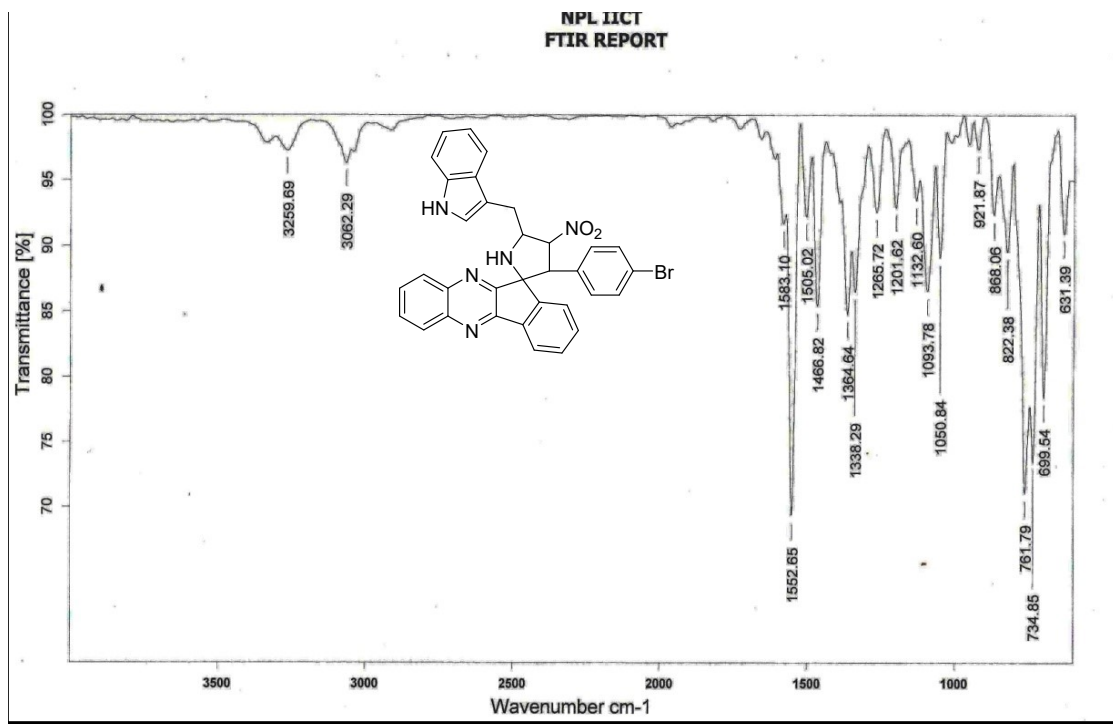
PMG\_TRY\_601  
20170919\_12.6 (0.288) AM2 (Ar,22000.0,0.00,0.00); ABS; Cm (3:12-14:47)

19-Sep-2017 19:58:26

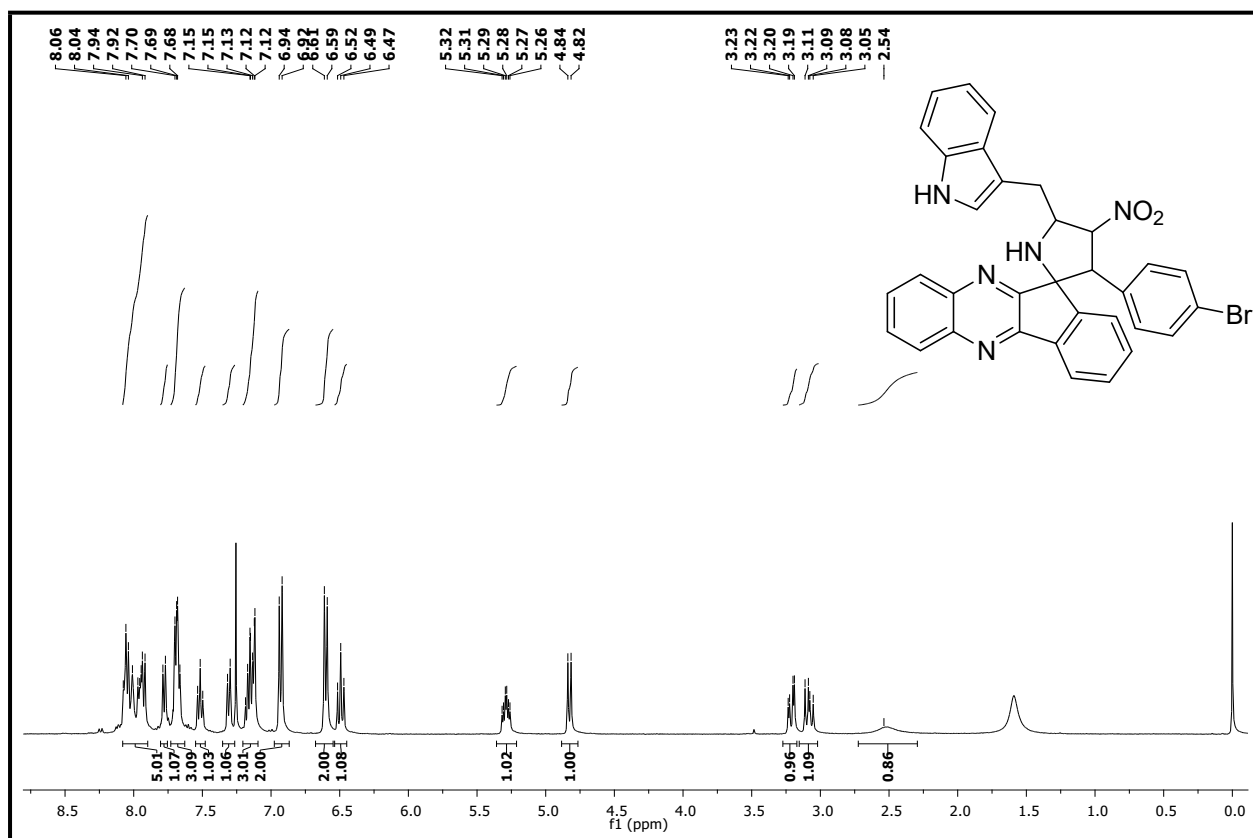


Mass	Calc. Mass	mDa	PFM	DBE	i-FIT	Norm	Conf (%)	Formula
602.1187	602.1192	-0.5	-0.8	23.5	32.2	n/a	n/a	C33 H25 N5 O2 Br

IR Spectrum of compound 7h



<sup>1</sup>H NMR Spectrum (CDCl<sub>3</sub>, 400 MHz) of compound 7h



**<sup>13</sup>C NMR Spectrum (CDCl<sub>3</sub>, 100 MHz) of compound 7h**

