

Supporting Information

In(OTf)₃-Catalysed One-pot Versatile Pyrrole Synthesis through Domino Annulation of α -Oxoketene-*N,S*-acetals with Nitroolefins

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General Information

All the commercially available reagents were purchased from Merck, Aldrich and Fluka, and were used as received. All ^1H and ^{13}C NMR spectra were recorded on JEOL AL 300 and BRUKER 400 MHz FT-NMR spectrometer. Chemical shifts are given as δ value (in ppm) with reference to tetramethylsilane (TMS) as the internal standard. The IR spectra were recorded on PerkinElmer Spectrum Version 10.03.05 FT-IR spectrophotometer. Mass spectra were recorded on a Agilent Q-TOF B.05.00 (B5042.0), BRUKERmicroTOF-Q III and maXis ESI mass spectrometer instrument. X-ray diffraction was measured on Xcalibur Oxford CCD Diffractometer. All the reactions were monitored by TLC using precoated sheets of silica gel G/UV-254 of 0.25 mm thickness (Merck 60F₂₅₄) using UV light for visualization. Melting points were determined with Büchi B-540 melting point apparatus and are uncorrected. Solvents were dried and purified by standard procedures.

Typical experimental procedures

General procedure for synthesis of 1,2,3,4-tetra and 1,2,3,4,5-pentasubstituted pyrroles

α -Oxoketene-*N,S*-acetal,¹ β -nitrostyrenes/ β -methyl- β -nitrostyrenes² were prepared from reported procedures.

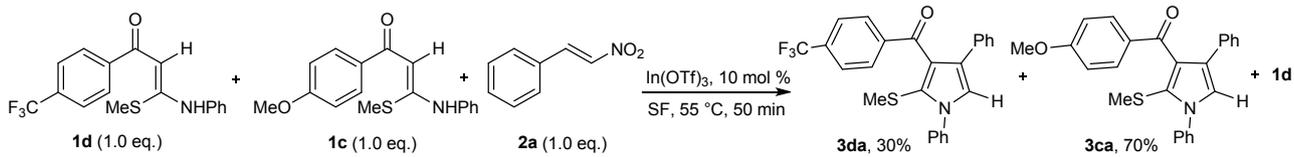
Experimental procedure for Synthesis of 3:

The α -oxoketene-*N,S*-acetal **1** (1.0 mmol), β -nitrostyrene/ β -methyl- β -nitrostyrene **2** (1.0 mmol) were taken with $\text{In}(\text{OTf})_3$ (10 mol%) in a 25 ml round bottom flask and the whole reaction mixture was heated in oil bath at 55 °C for the stipulated period of time till the completion of the reaction (monitored by TLC). After completion of reaction, organic phase was extracted with EtOAc, washed with water (2×20 mL) and brine (1×20 mL). Dried over sodium sulphate and the crude compounds were purified with silica gel column chromatography.

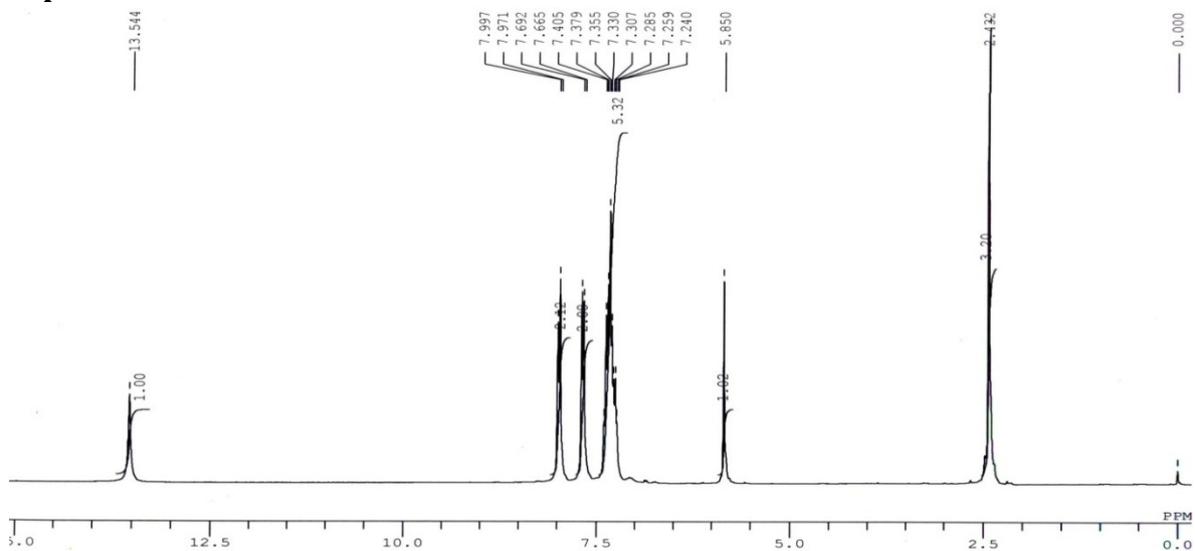
Competition Reactions

a) Competition reaction between α -oxoketene-*N,S*-acetals (**1c** vs. **1d**):

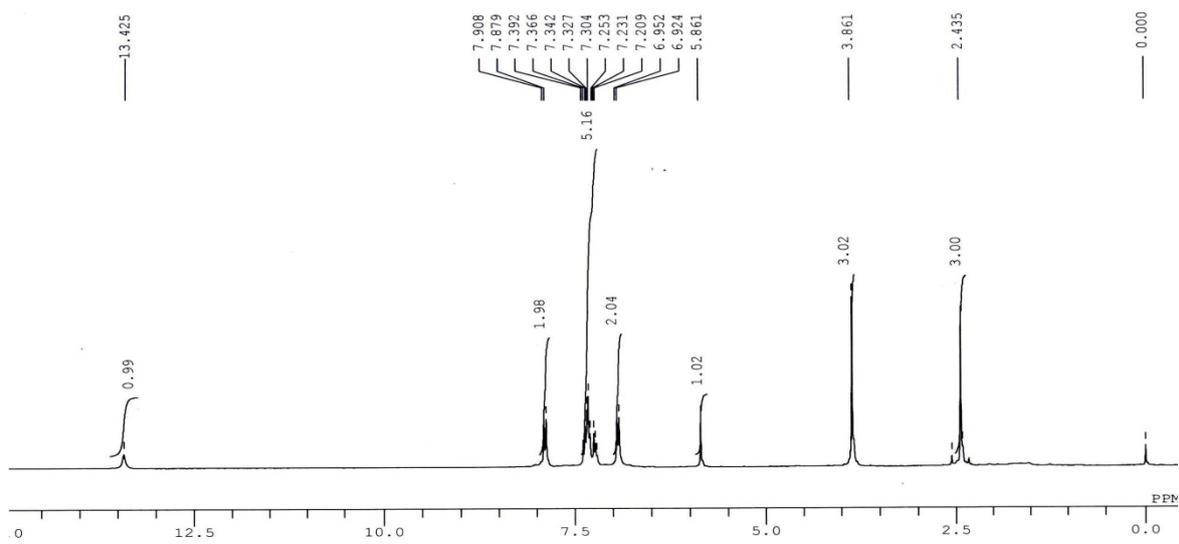
To an oven-dried 25 mL round bottom flask, equipped with a magnetic stirrer bar was charged with **1d** (1.0 eq.), **1c** (1.0 eq.) and **2a** (1.0 eq.). To this reaction mixture 10 mol% of $\text{In}(\text{OTf})_3$ was added and the whole reaction mixture was put on a pre-heated oil bath at 55 °C. After completion of the reaction (monitored through TLC), 5 mL of distilled water was added and extracted with EtOAc, washed with brine and distilled water (1×20 mL). Dried over anhydrous sodium sulphate and solvent was evaporated under reduced pressure to obtain the crude product, which was analyzed by ^1H NMR using TMS as internal standard.



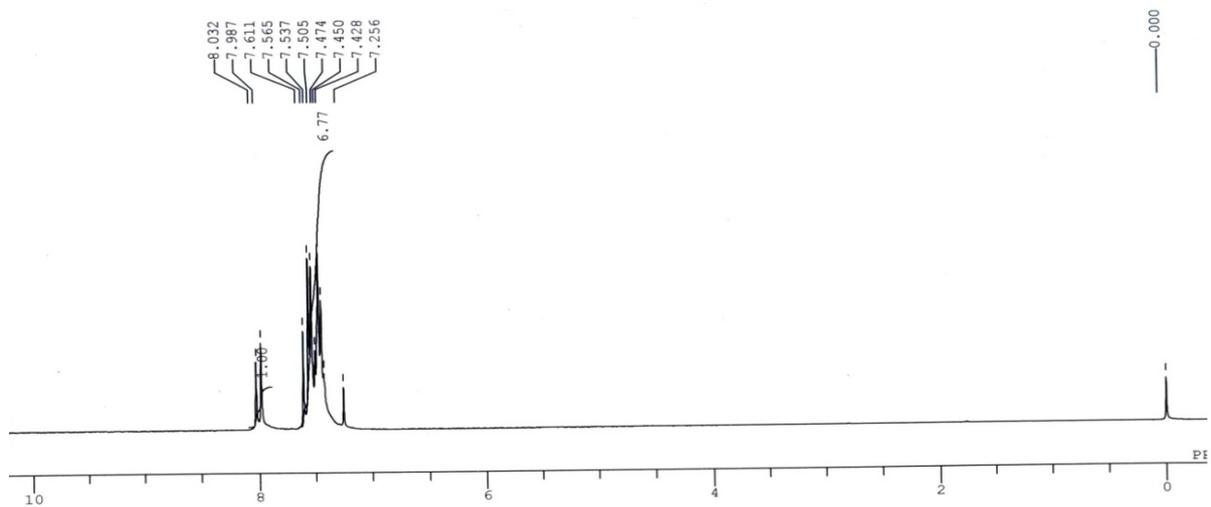
¹H-NMR Spectrum of 1d:



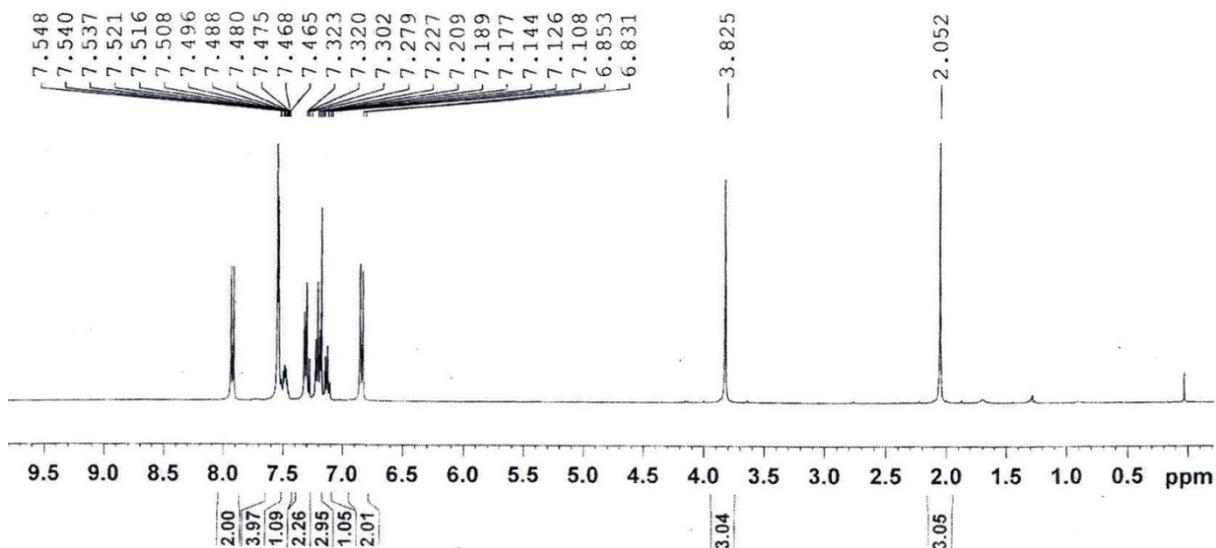
¹H-NMR Spectrum of 1c:



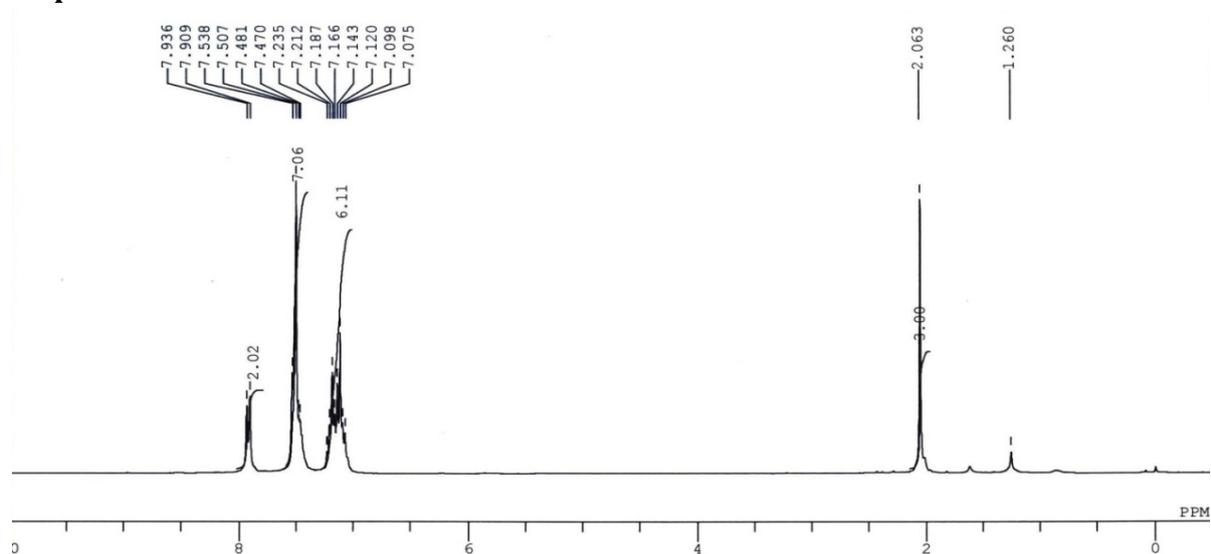
¹H-NMR Spectrum of 2a:



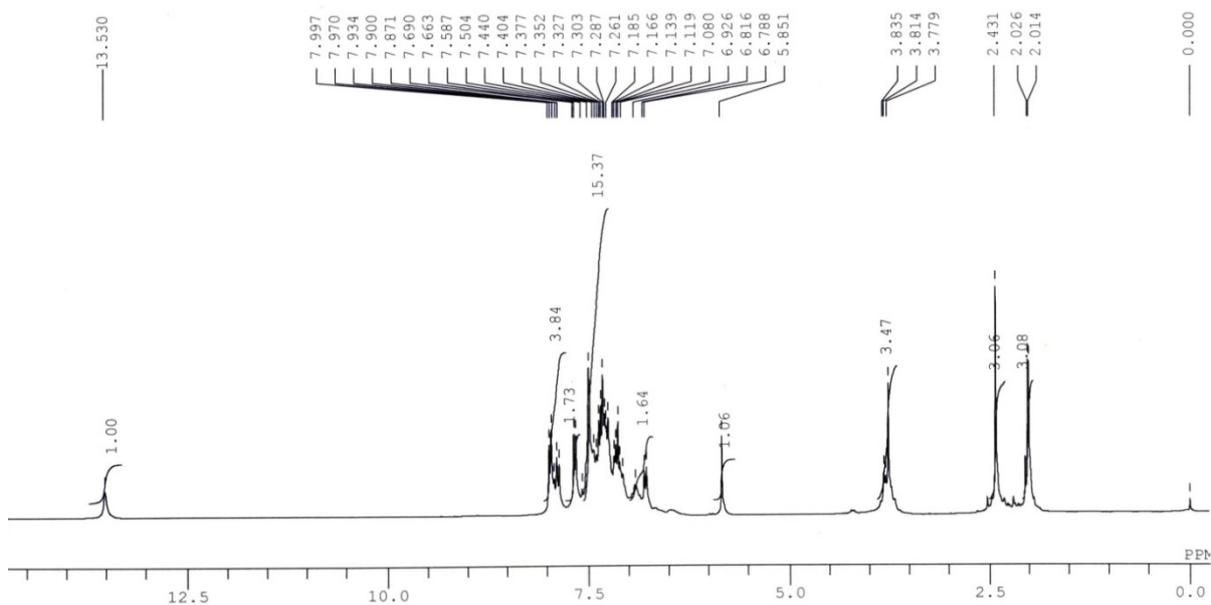
¹H-NMR Spectrum of 3ca:



¹H-NMR Spectrum of 3da:

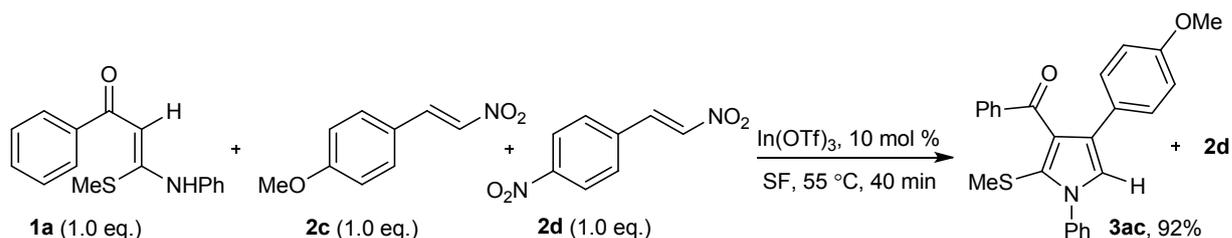


¹H-NMR Spectrum of crude reaction mixture:

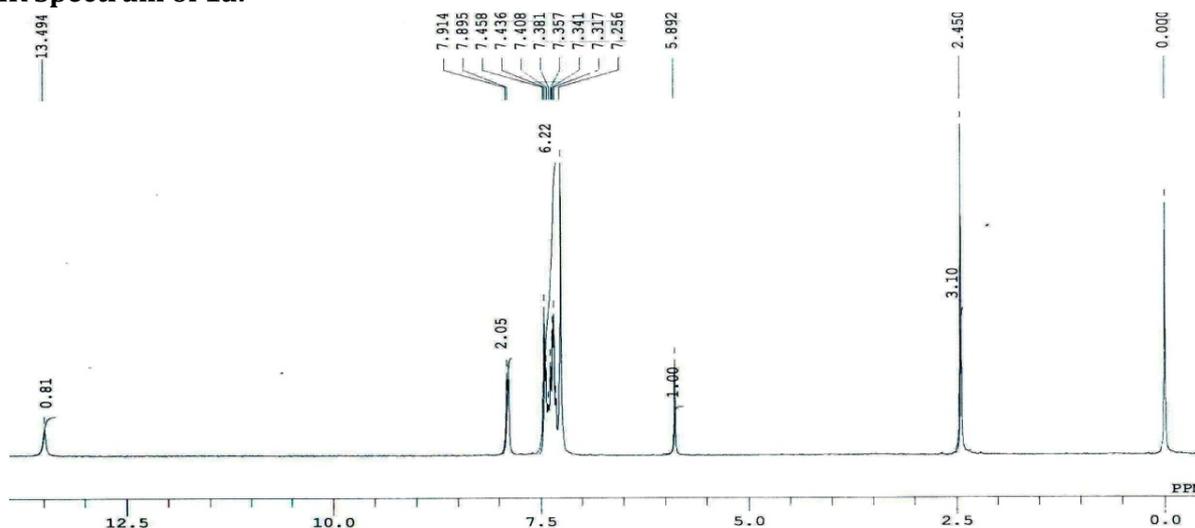


Since, presence of characteristic peaks of α -oxoketene-N,S-acetal at 5.8 ppm (of α -H), 13.53 ppm (of N-H) with integration value of ~ 1 and at 2.43 ppm with integration value of ~ 3 confirms the presence of only one α -oxoketene-N,S-acetal, but it can't be predicted from NMR because both α -oxoketene-N,S-acetals have almost similar characteristic peaks. TLC pattern clearly shows that **1c** was fully consumed and **1d** was left.

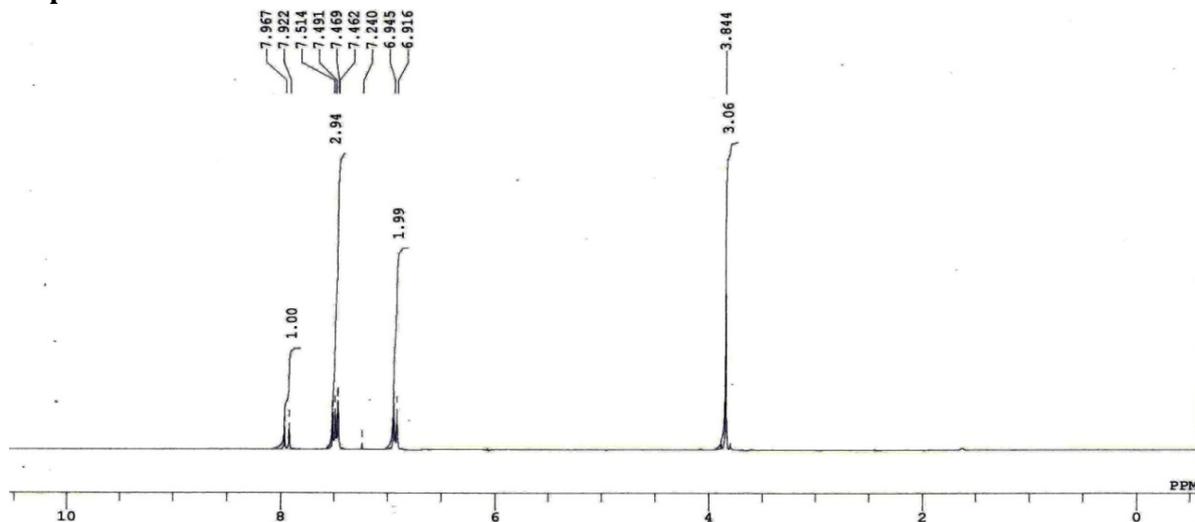
b) **Competition reaction between β -nitrostyrenes (2cvs. 2d):** To an oven-dried 25 mL round bottom flask, equipped with a magnetic stirrer bar was charged with **1a** (1.0 eq.), **2c** (1.0 eq.) and **2d** (1.0 eq.). To this reaction mixture 10 mol % of $\text{In}(\text{OTf})_3$ was added and the whole reaction mixture was put on a pre-heated oil bath at 55 °C. After completion of the reaction (monitored through TLC), 5 mL of distilled water was added and extracted with EtOAc, washed with brine and distilled water (1 \times 20 mL). Dried over anhydrous sodium sulphate and solvent was evaporated under reduced pressure to obtain the crude product, which was analyzed by ^1H NMR using TMS as internal standard.



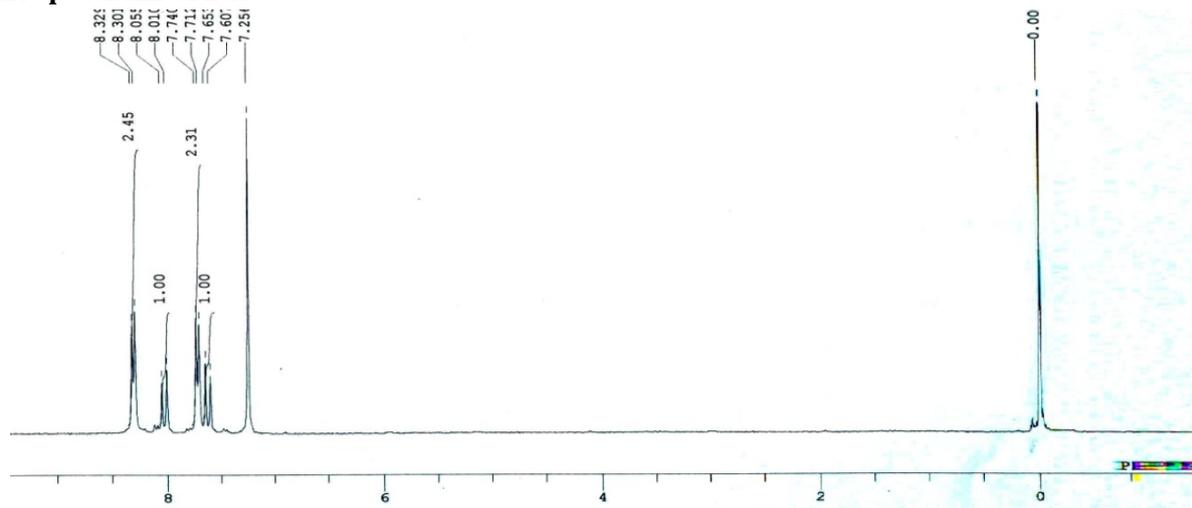
^1H -NMR Spectrum of **1a**:



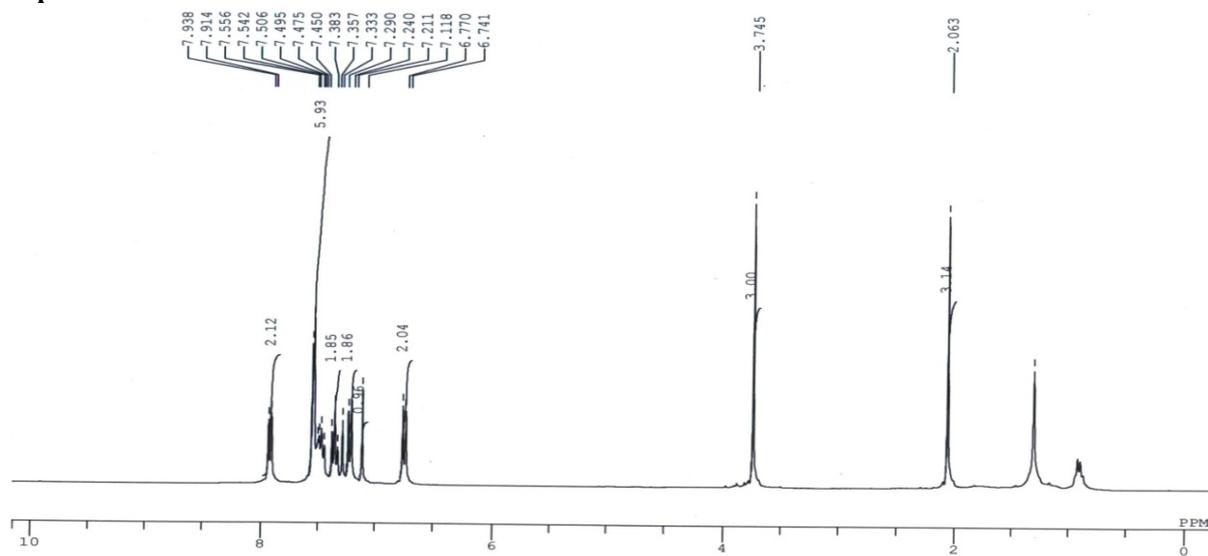
^1H -NMR Spectrum of **2c**:



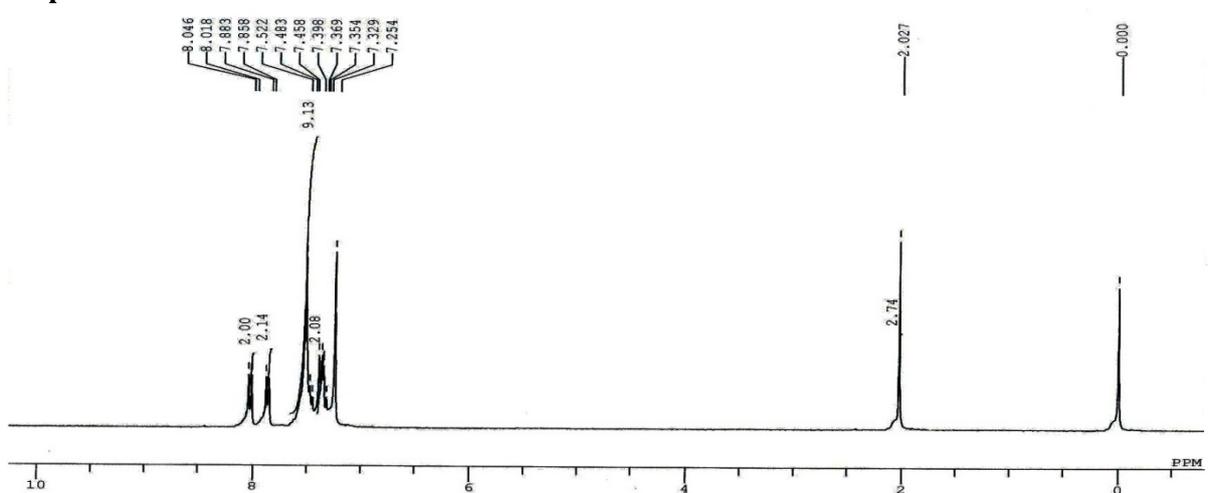
¹H-NMR Spectrum of 2d:



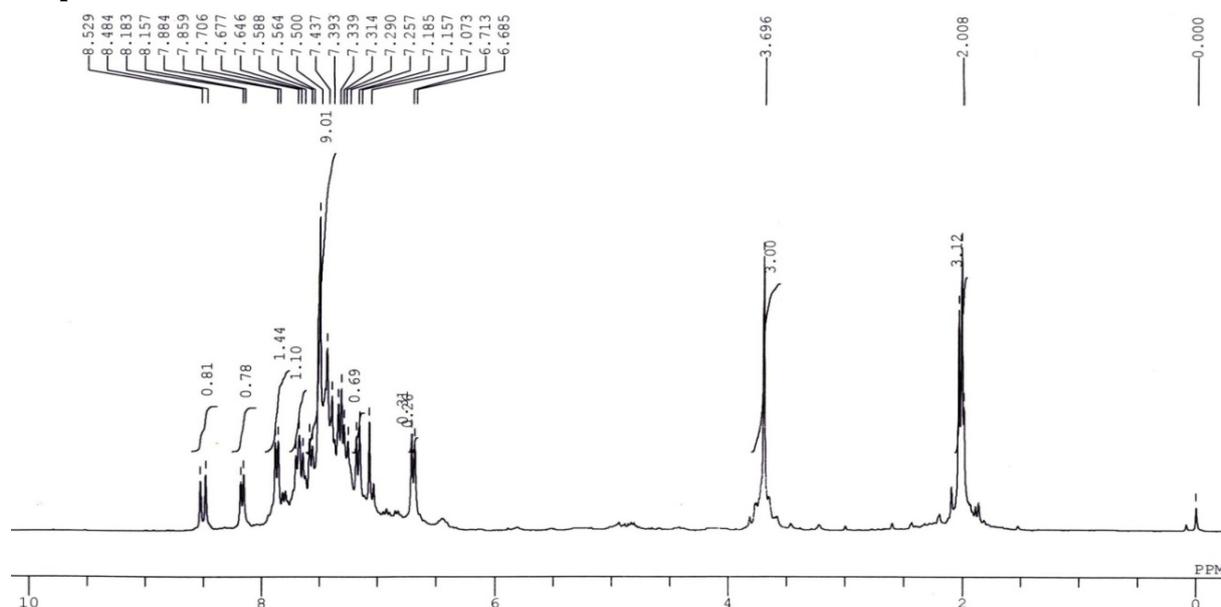
¹H-NMR Spectrum of 3ac:



¹H-NMR Spectrum of 3ad:



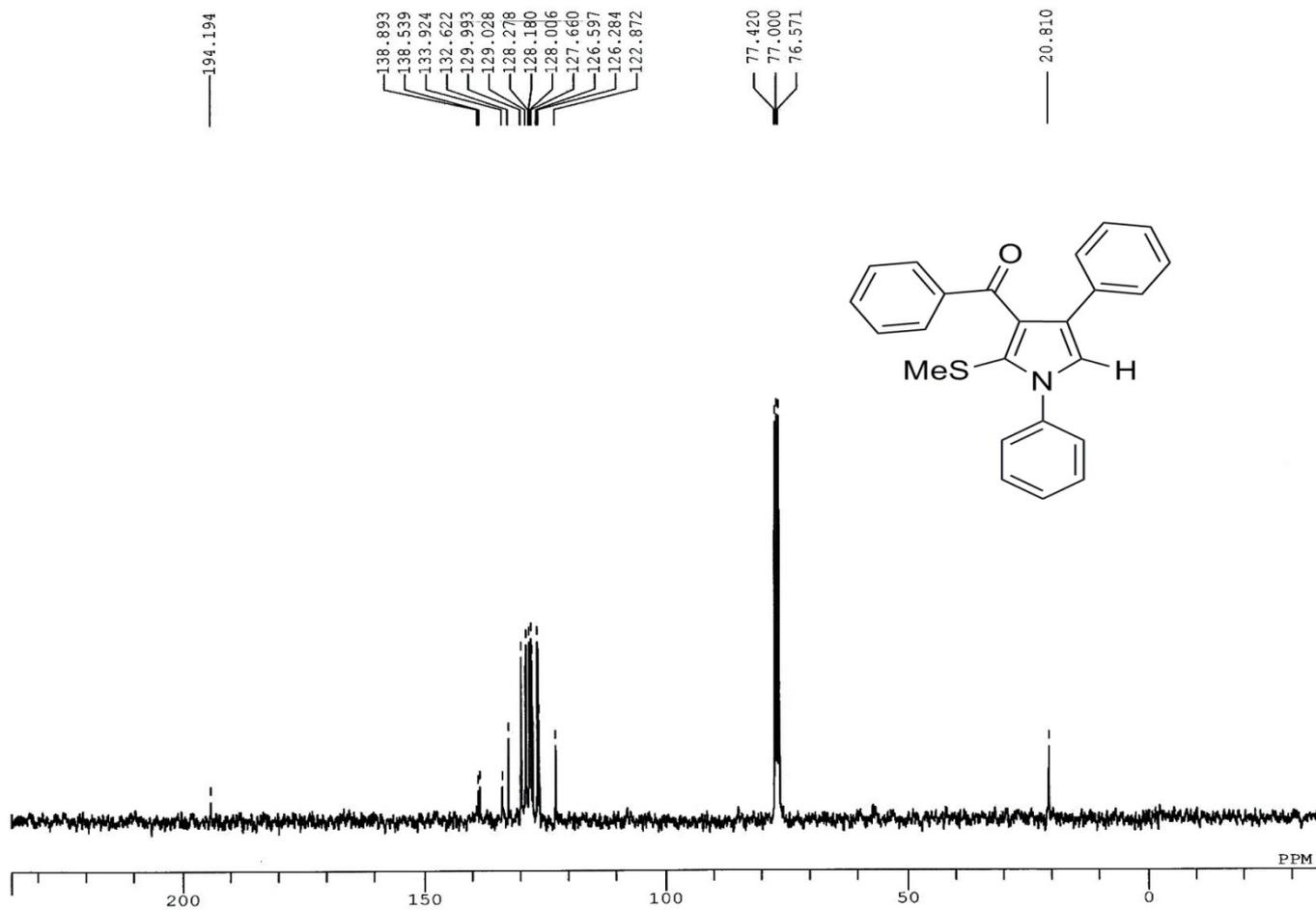
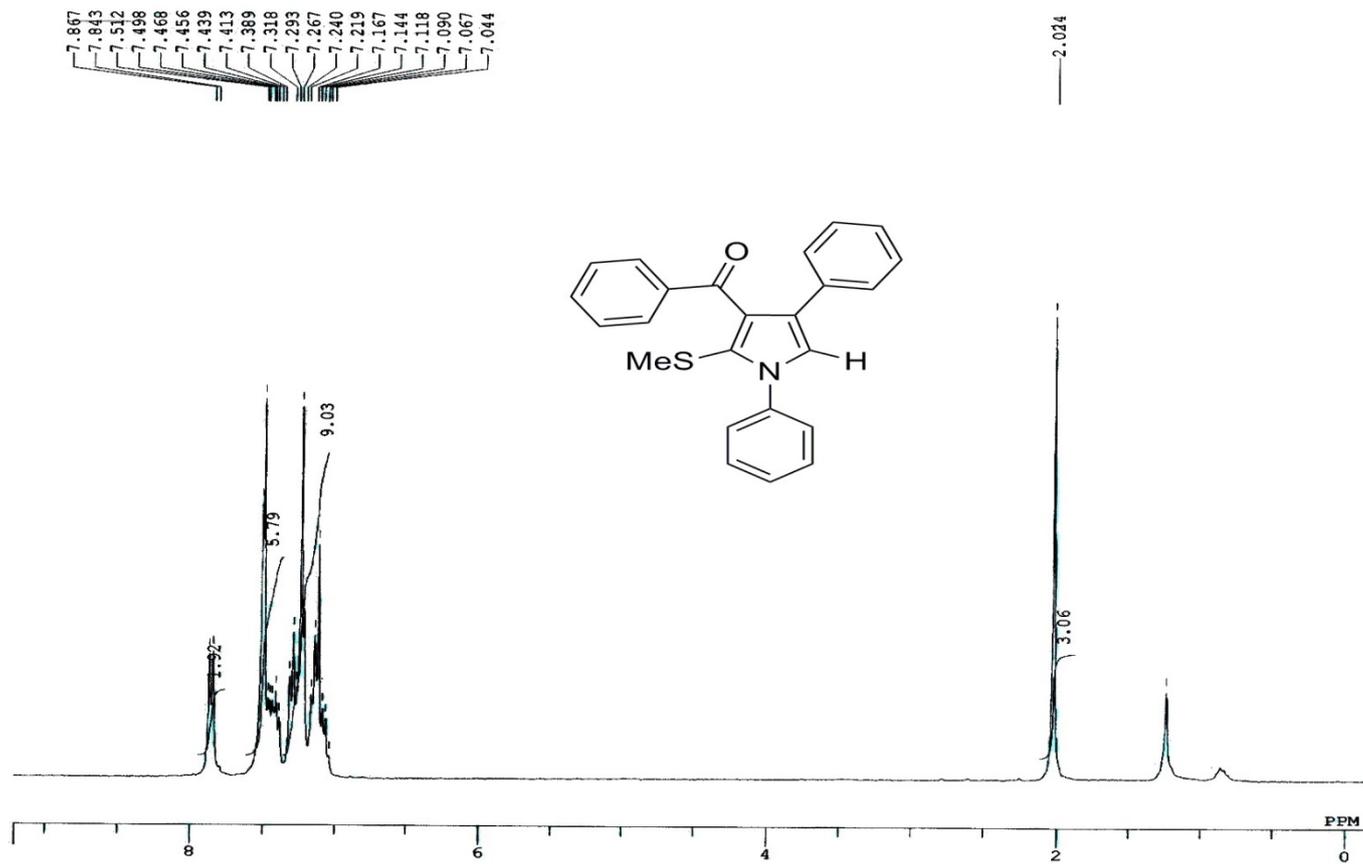
¹H-NMR Spectrum of crude reaction mixture:



¹H-NMR spectrum of α -oxoketene-N,S-acetal shows characteristic peaks of α -H at 5-6 ppm and that of N-H at 12-15 ppm, which are absent in ¹H-NMR spectrum of crude mixture. It clearly shows depletion of α -oxoketene-N,S-acetal **1a** from reaction mixture. And if, there is reminiscent of **2c** in reaction mixture, it would be clearly shown in spectra as $-\text{OCH}_3$ peak different with $-\text{OCH}_3$ of product pyrrole. But there is only one peak of $-\text{OCH}_3$ with integration value of ~ 3 . Now, on the basis of these observations we can clearly say that, there are only product pyrrole **3ac** and β -nitrostyrene **2d** in the crude mixture.

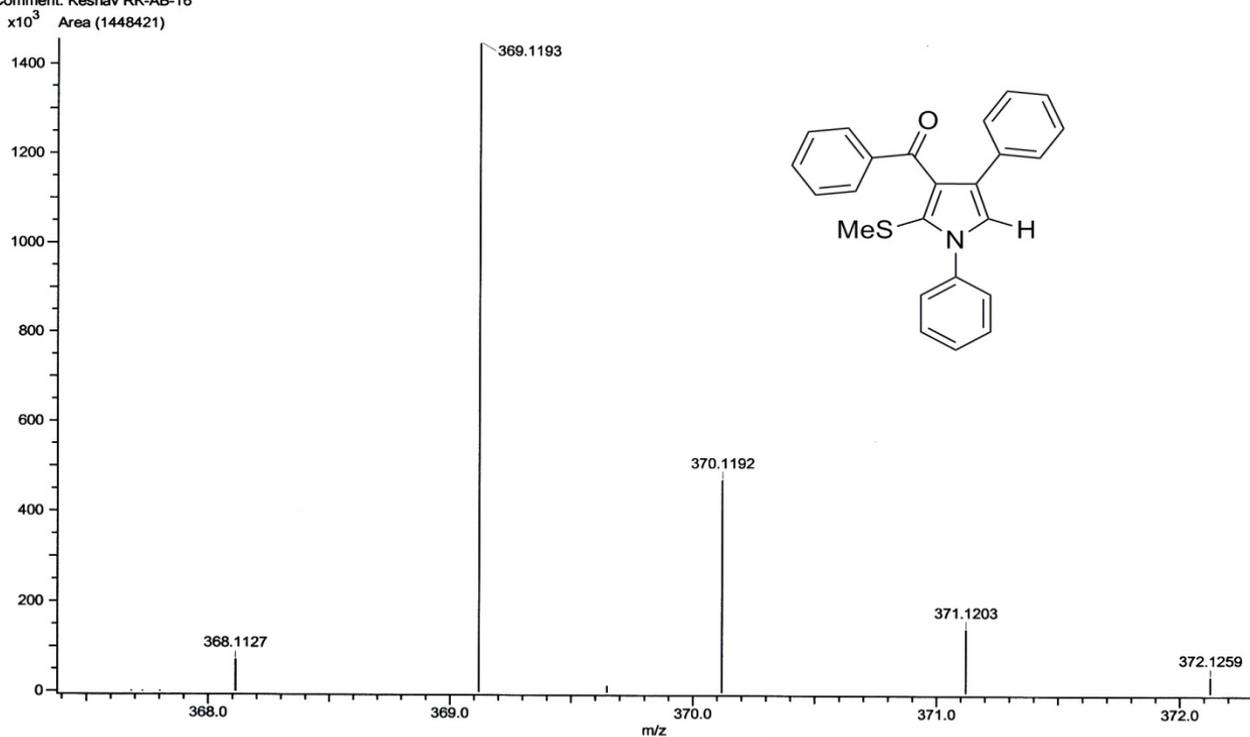
Characterization data for isolated compounds

① **3aa**. 3-Benzoyl-2-(methylthio)-1,4-diphenylpyrrole

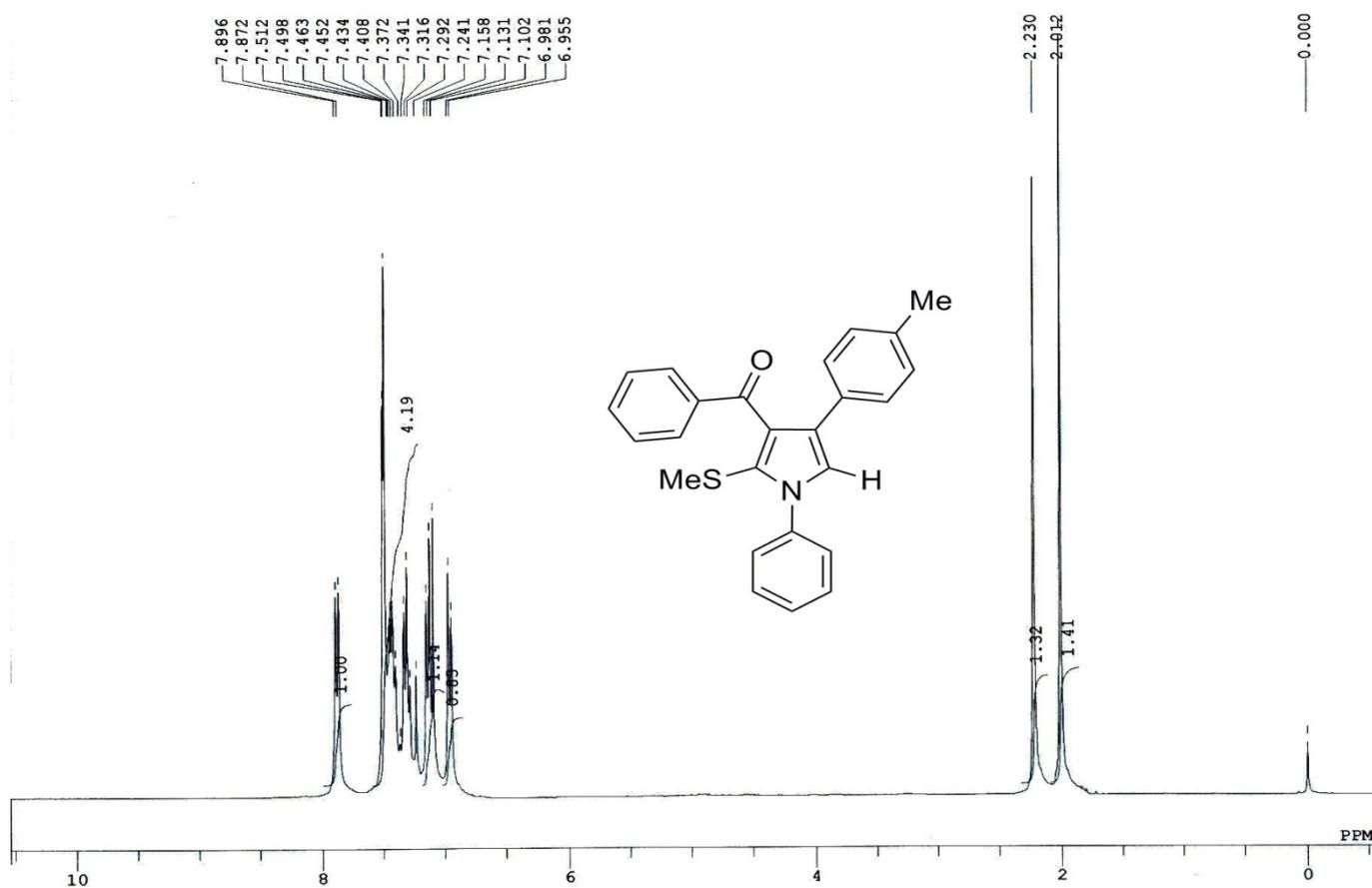


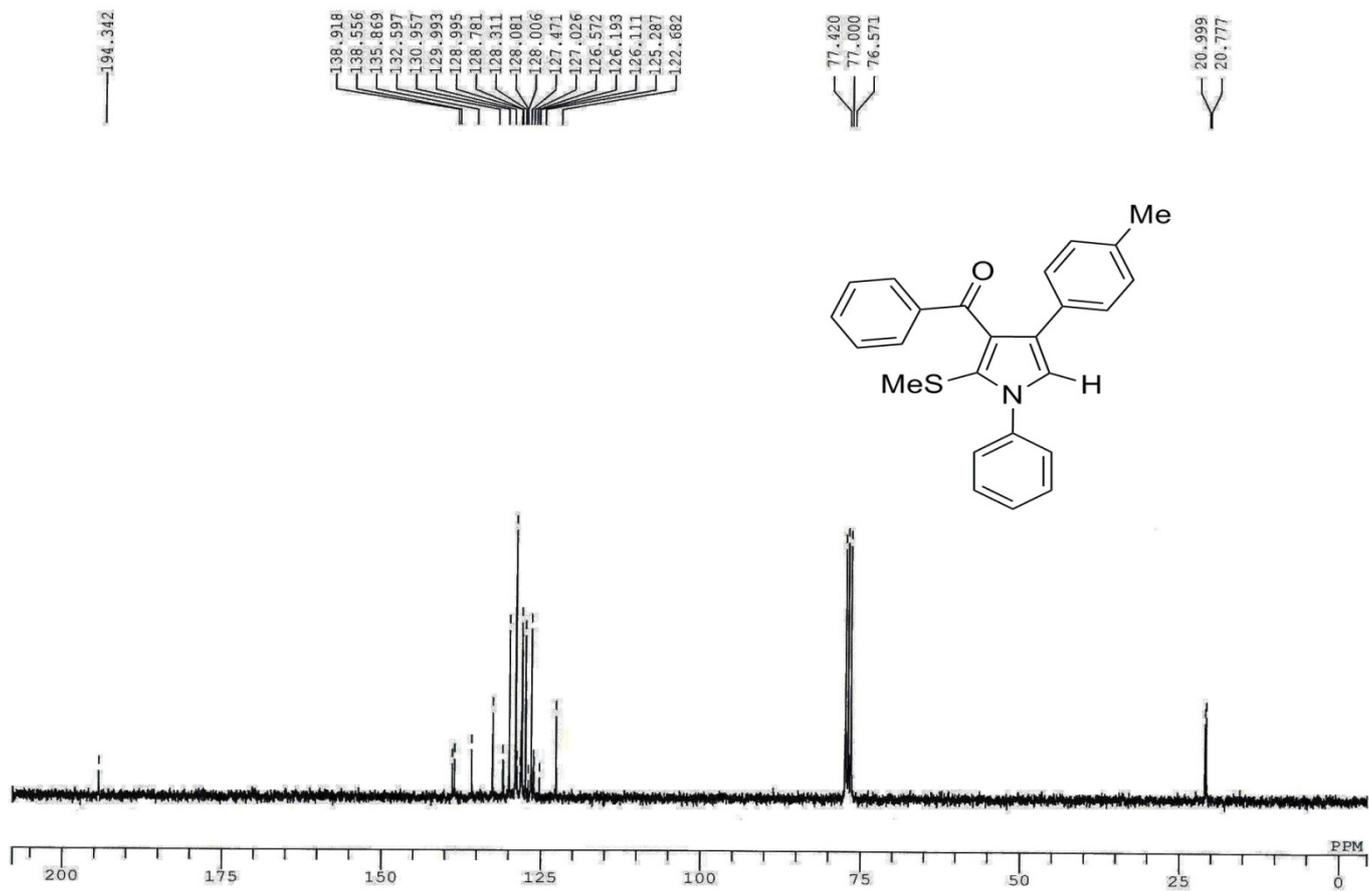
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Comment: Keshav RK-AB-16

Experiment Date/Time: 5/17/2013 2:20:31 PM
Ionization Mode: EI+



② **3ab.** 3-Benzoyl-2-(methylthio)-1-phenyl-4-(*p*-tolyl)pyrrole



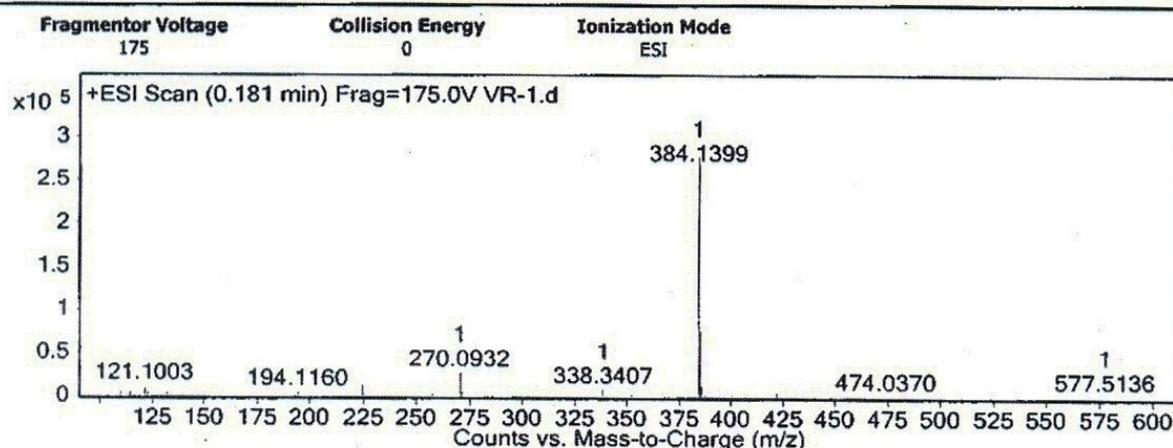


Qualitative Analysis Report

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Comment			

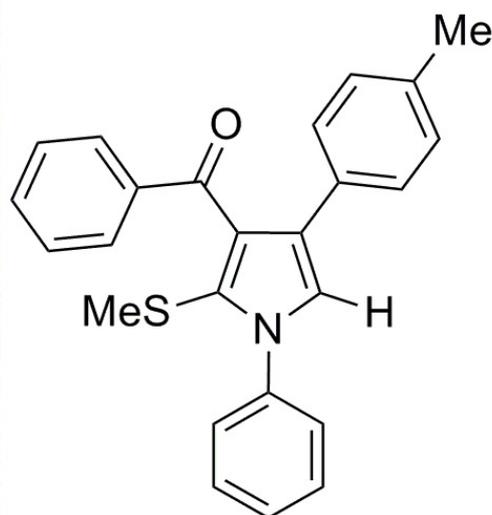
Acquisition SW 6200 series TOF/6500 series
 Version Q-TOF B.05.00 (B5042.0)

User Spectra

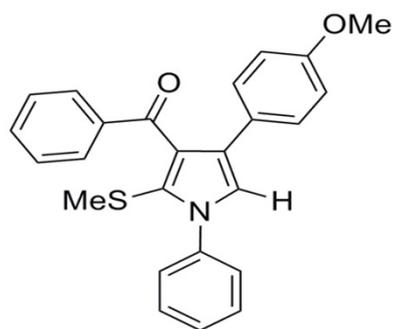
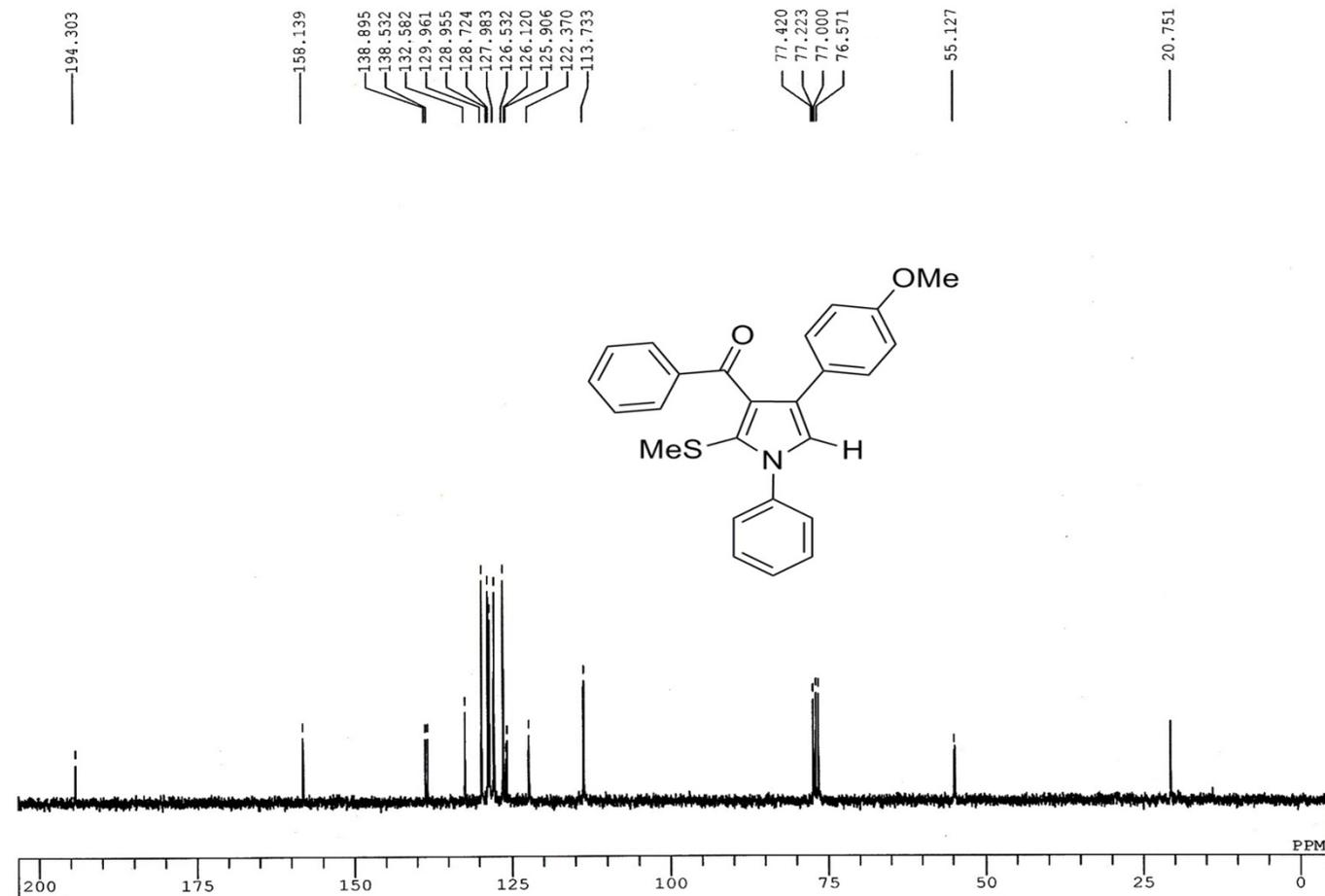
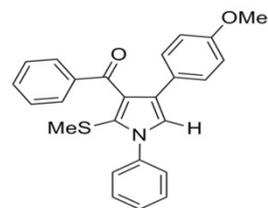
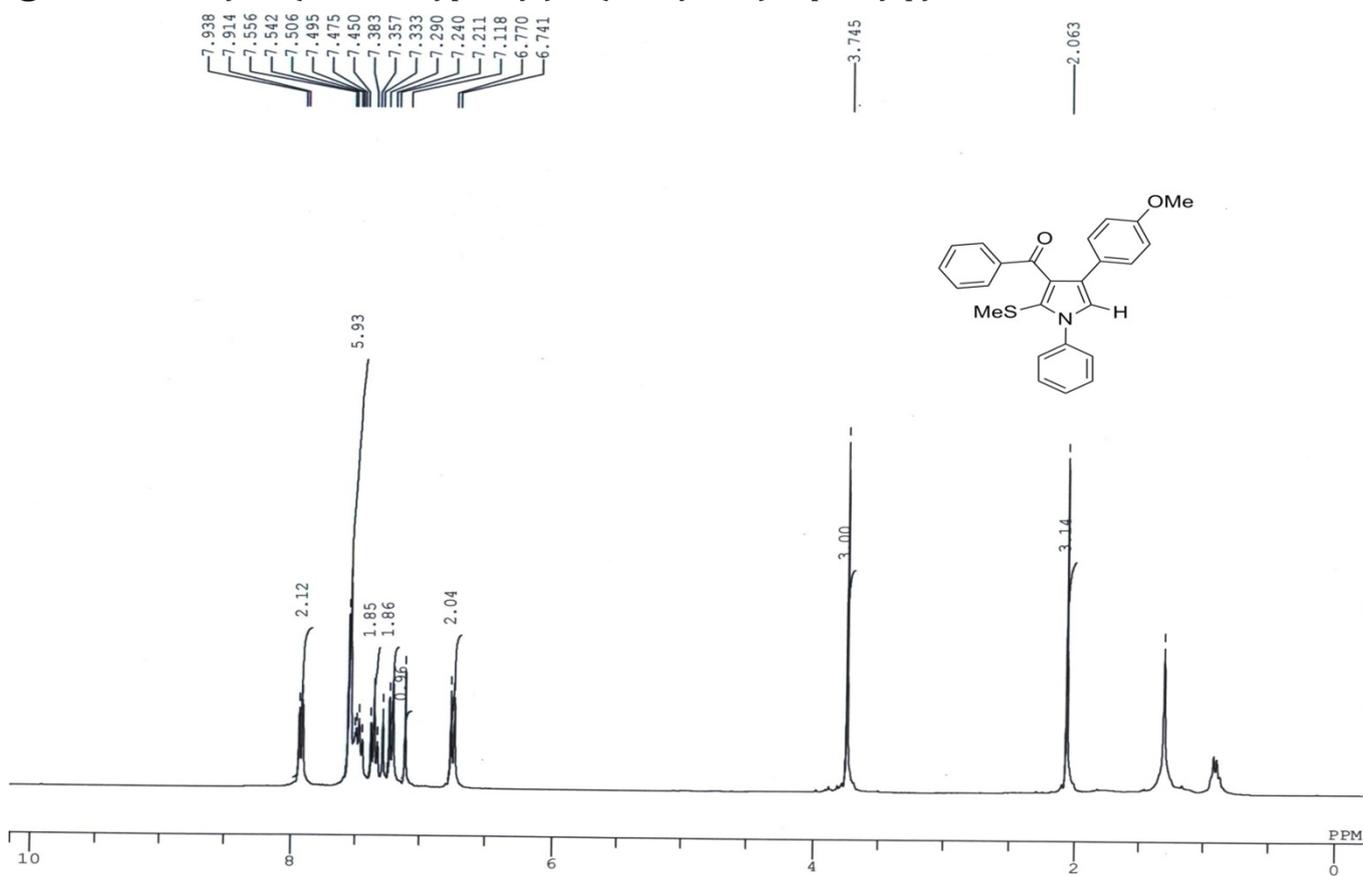


Peak List

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114.0909		4570.33		
118.0853		3601.55		
121.1003		10676.85		
122.9634		5639.33		
131.9292		4179.25		
149.0222		5644.65		
194.116		4177.08		
222.0902		3513.6		
224.1269	1	12671.9		
270.0932	1	26843.81		
271.0961	1	5109.94		
338.3407	1	7783.98		
384.1399	1	278294.84	C ₂₅ H ₂₂ N O S	(M+H) ⁺
385.1427	1	76666.23	C ₂₅ H ₂₂ N O S	(M+H) ⁺
386.1405	1	13946.29	C ₂₅ H ₂₂ N O S	(M+H) ⁺
387.1384	1	3938.15	C ₂₅ H ₂₂ N O S	(M+H) ⁺
406.1208	1	15571.78		
407.1245	1	4354.93		
422.0949	1	4794.14		



③3ac. 3-Benzoyl-4-(4-methoxyphenyl)-2-(methylthio)-1-phenylpyrrole

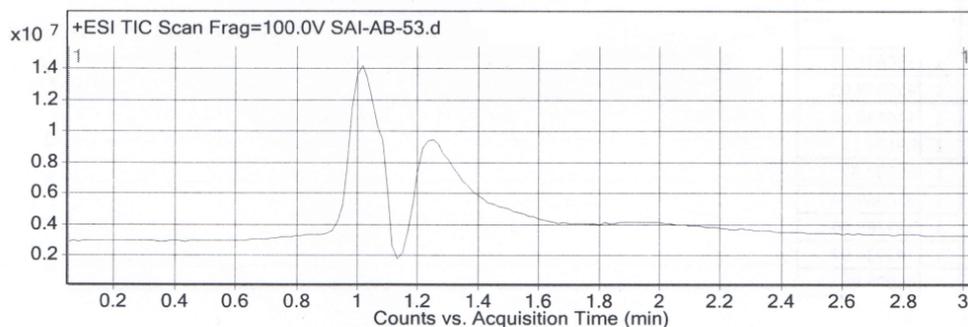


Qualitative Analysis Report

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IRM Calibration Status	Success	DA Method	Regular.m
Comment			
Sample Group		Info.	
Acquisition SW	J200 series TOF/6500 series		
Version	Q-TOF B.05.00 (B5042.0)		

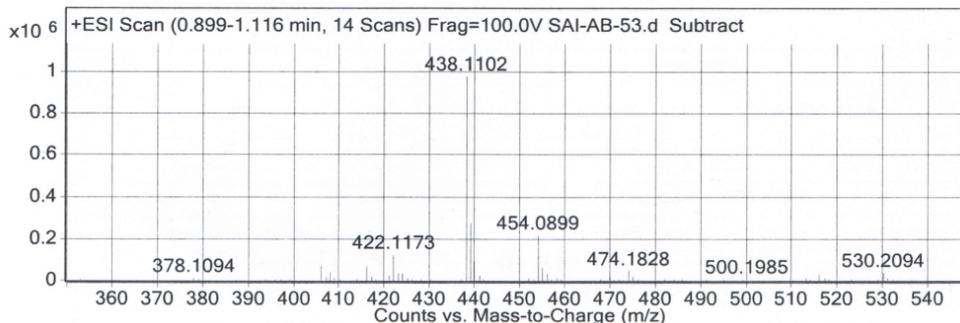
User Chromatograms

Fragmentor Voltage 100 Collision Energy 0 Ionization Mode ESI



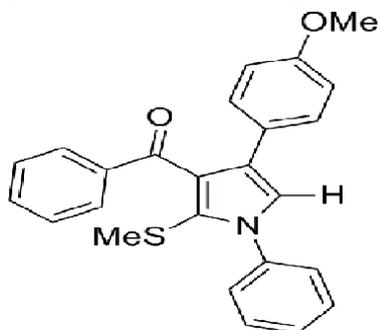
User Spectra

Fragmentor Voltage 100 Collision Energy 0 Ionization Mode ESI



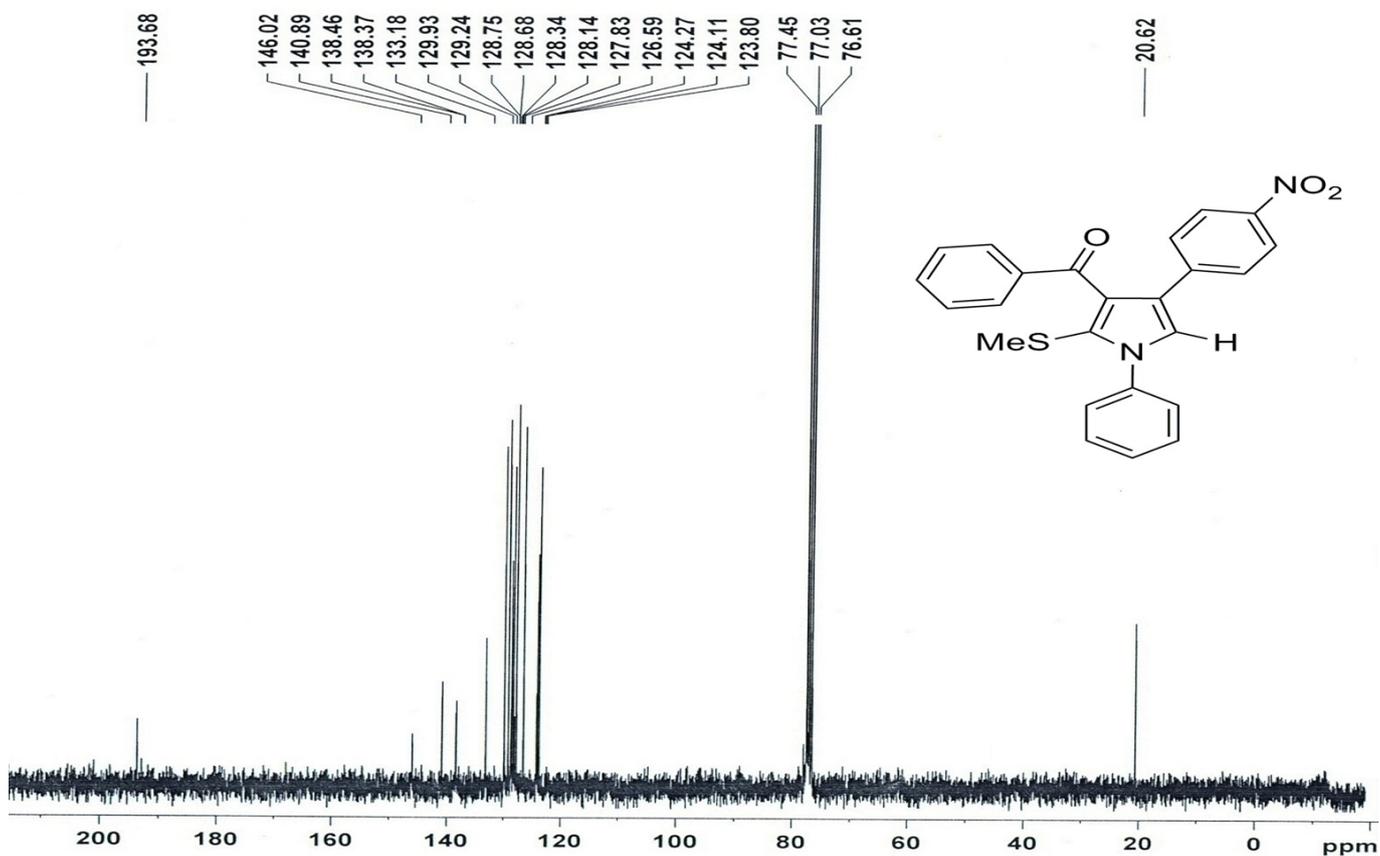
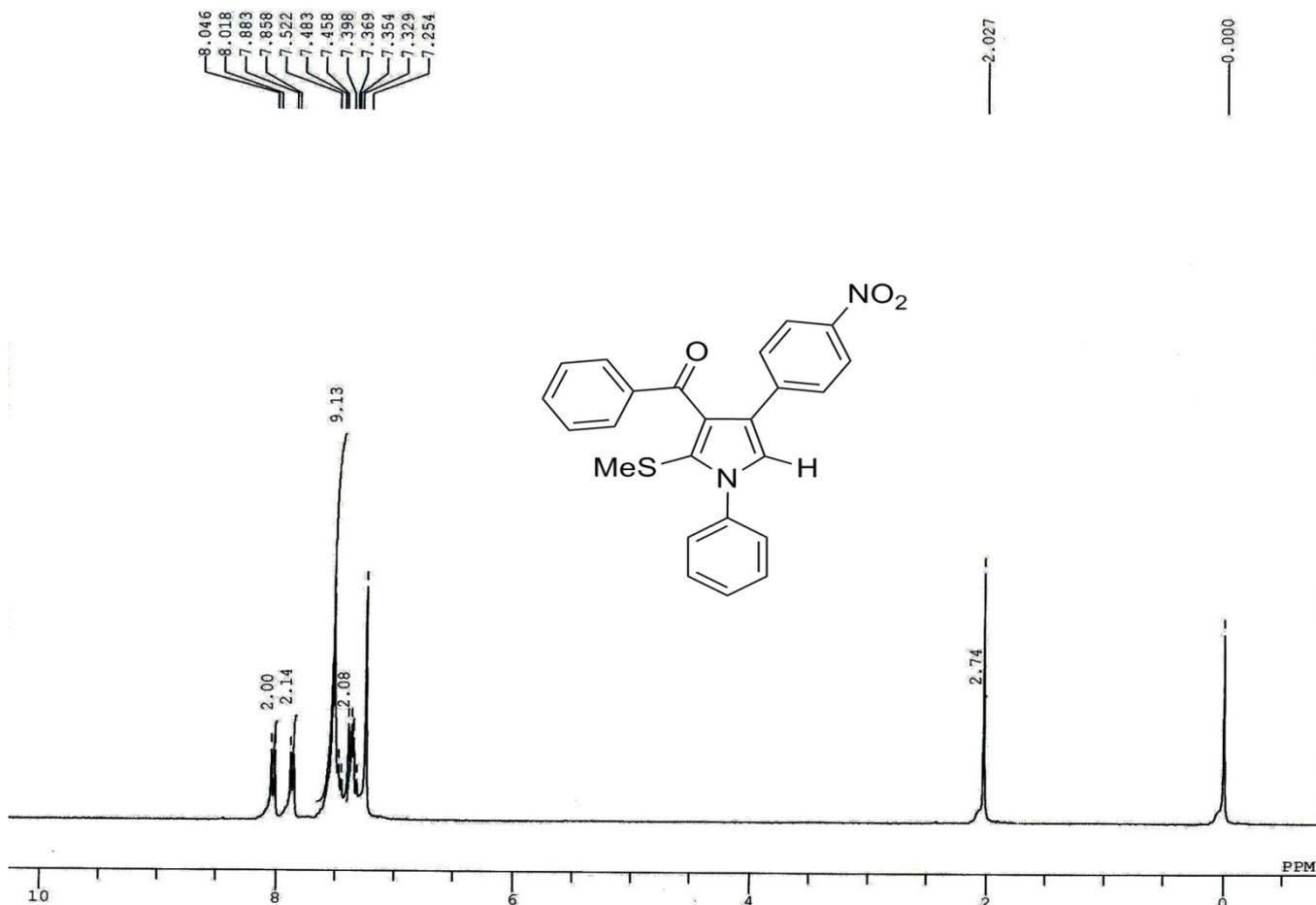
Peak List

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439.1136	1	276707.78
454.0899	1	217077.16
685.4334	1	114115.21
821.2428	1	290473.69
822.2465	1	165990.78
853.2355	1	1156950.25
854.2389	1	672438.63
855.2388	1	316002.81



Fragmentor Voltage Collision Energy Ionization Mode

④ 3ad. 3-Benzoyl-2-(methylthio)-4-(4-nitrophenyl)-1-phenylpyrrole

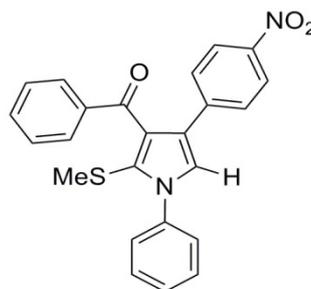
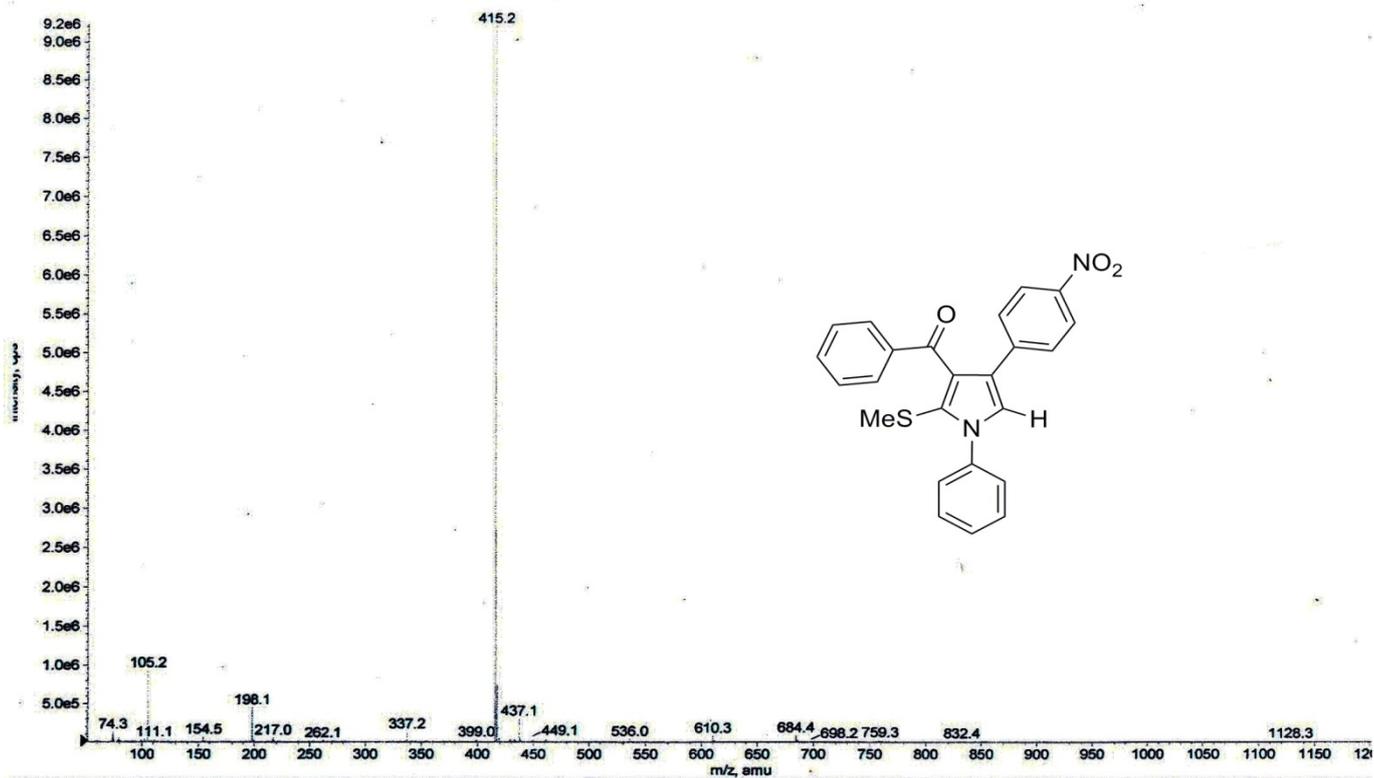


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Acq. Time: 13:19

*Ranbaxy Research Labs, Gurgaon, India.
Printing Time: 1:36:37 PM
Printing Date: Tuesday, September 04, 2012

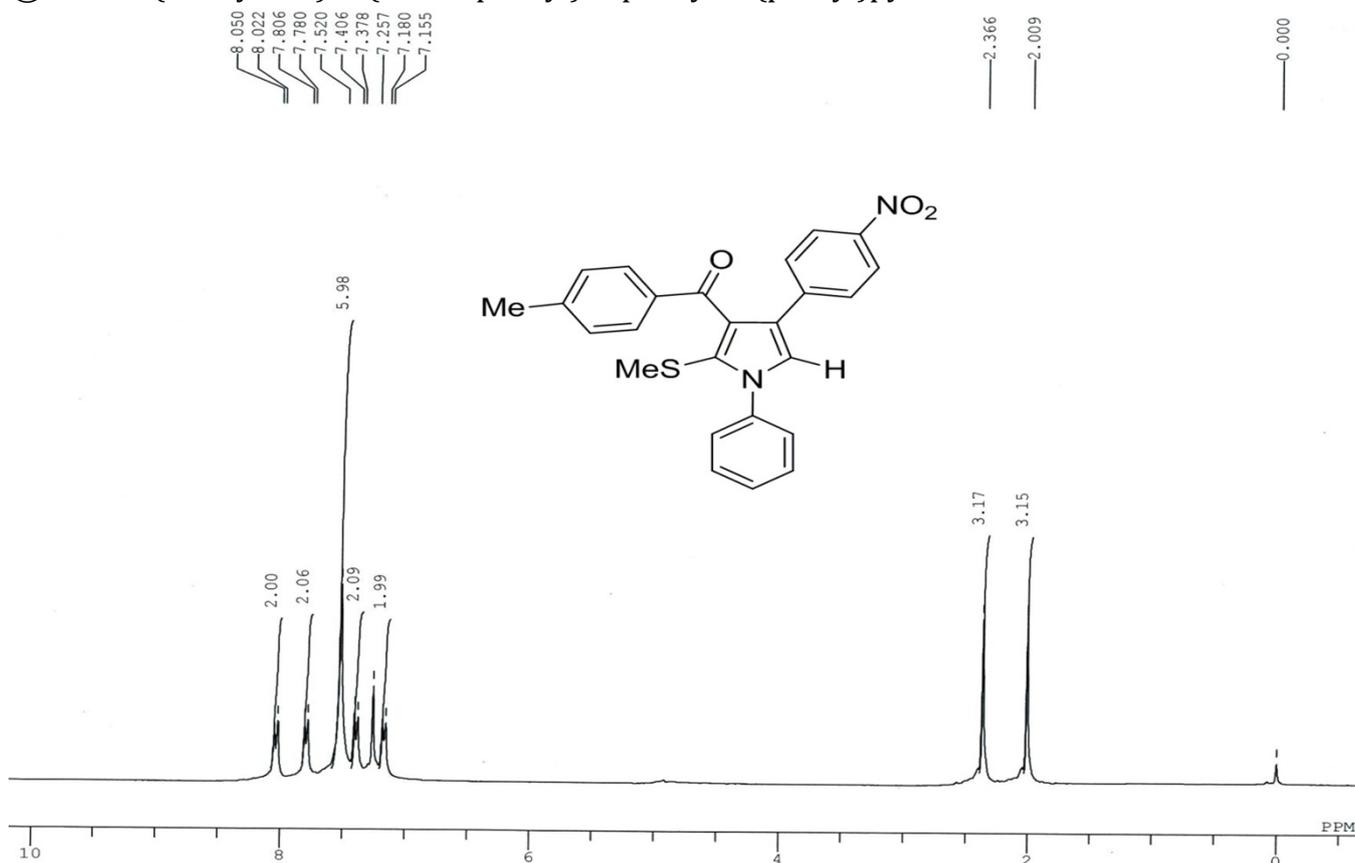
Sample Comment: RGA(B5422)112
Acq. File: 04 Sep 2012SET1.wiff
Instrument: ERL/AR/43-003

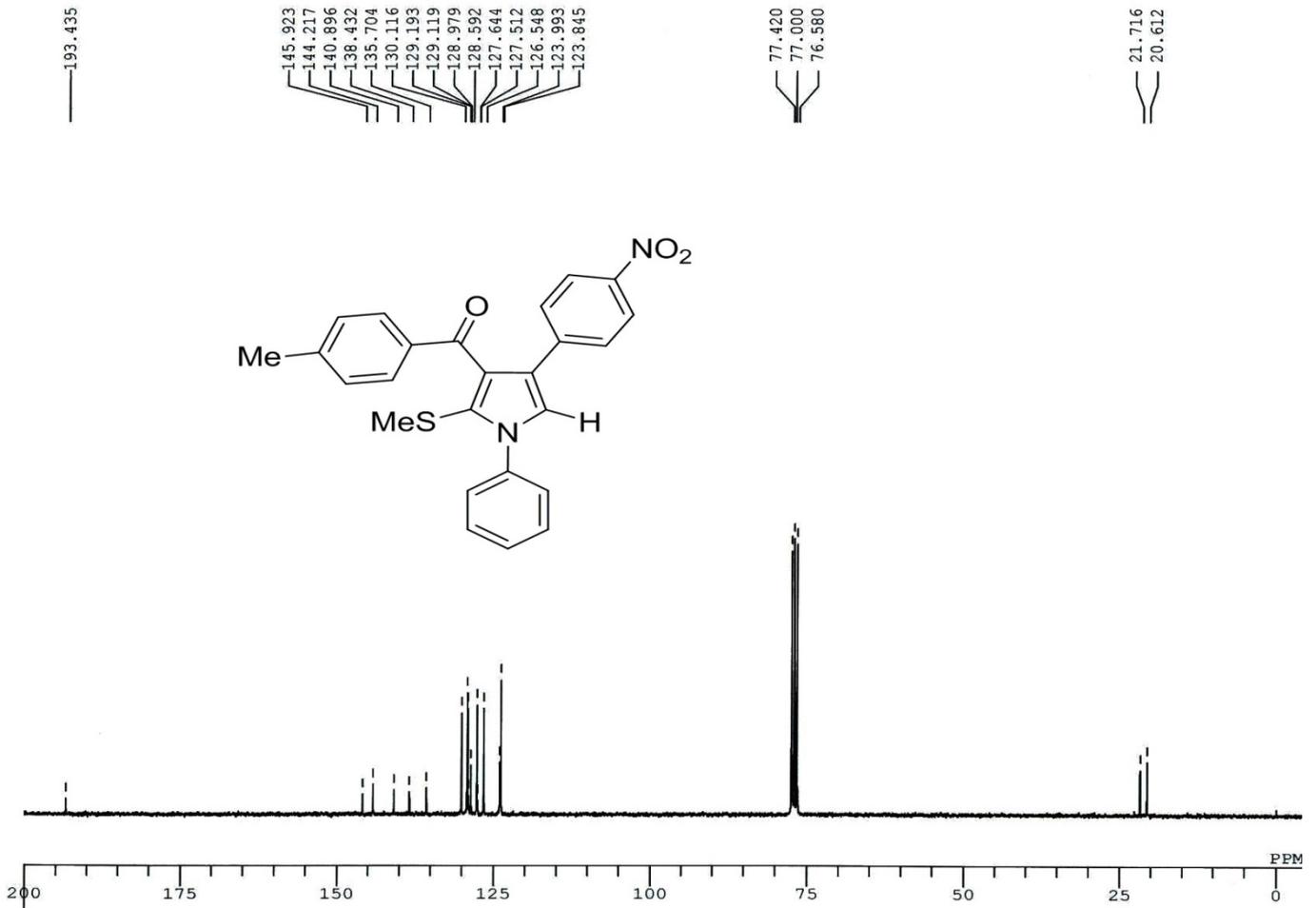
+Q1: 0.250 to 0.334 min from Sample 36 (#ASH(E-365)008\PROJ:ASENAPINE MALEATE) of 04 Sep 2012SET1.wiff (Turbo Spray), subtracted (0.66... Max 9.2e



Operator: Rajendra Kumar Gupta
Workstation: GURIARD095
Project: MASS SAMPLES

⑤ 3bd. 2-(methylthio)-4-(4-nitrophenyl)-1-phenyl-3-(p-tolyl)pyrrole



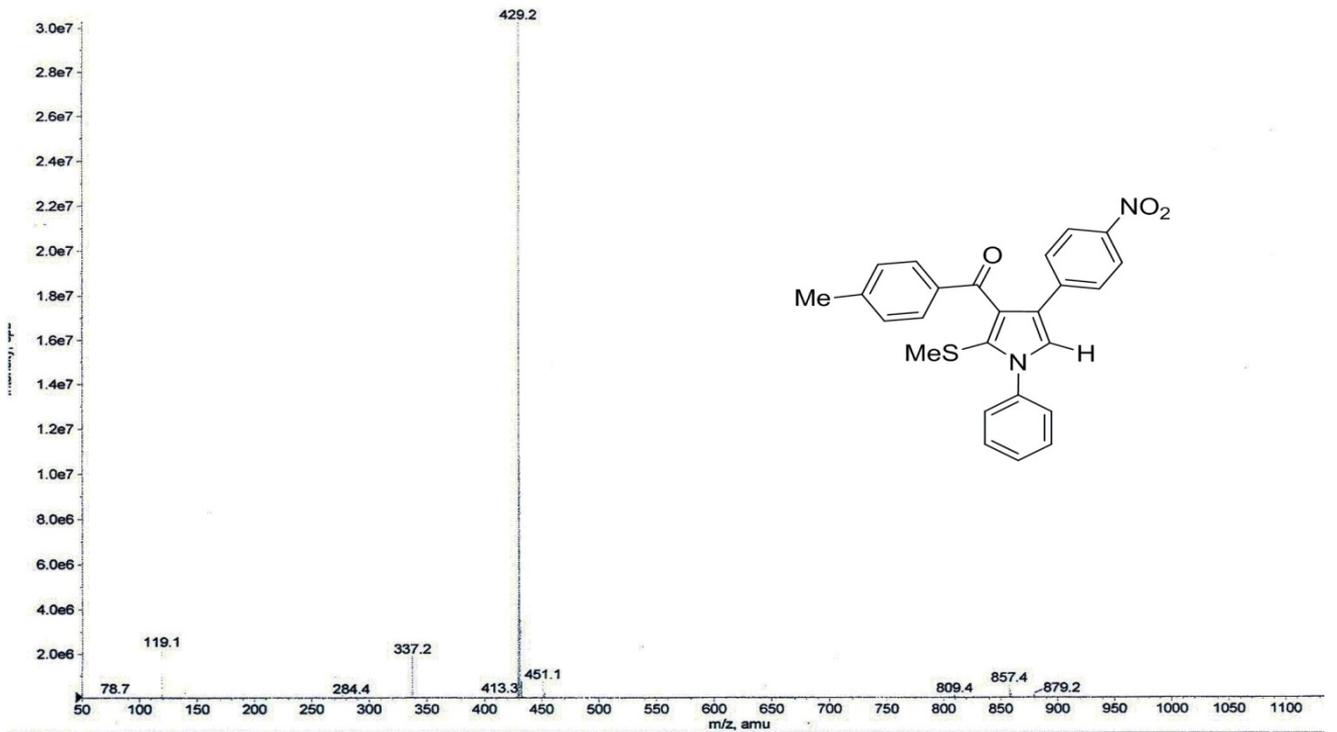


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 Acq. Time: 13:17

*Ranbaxy Research Labs, Gurgaon, India.
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 Printing Date: Tuesday, September 04, 2012

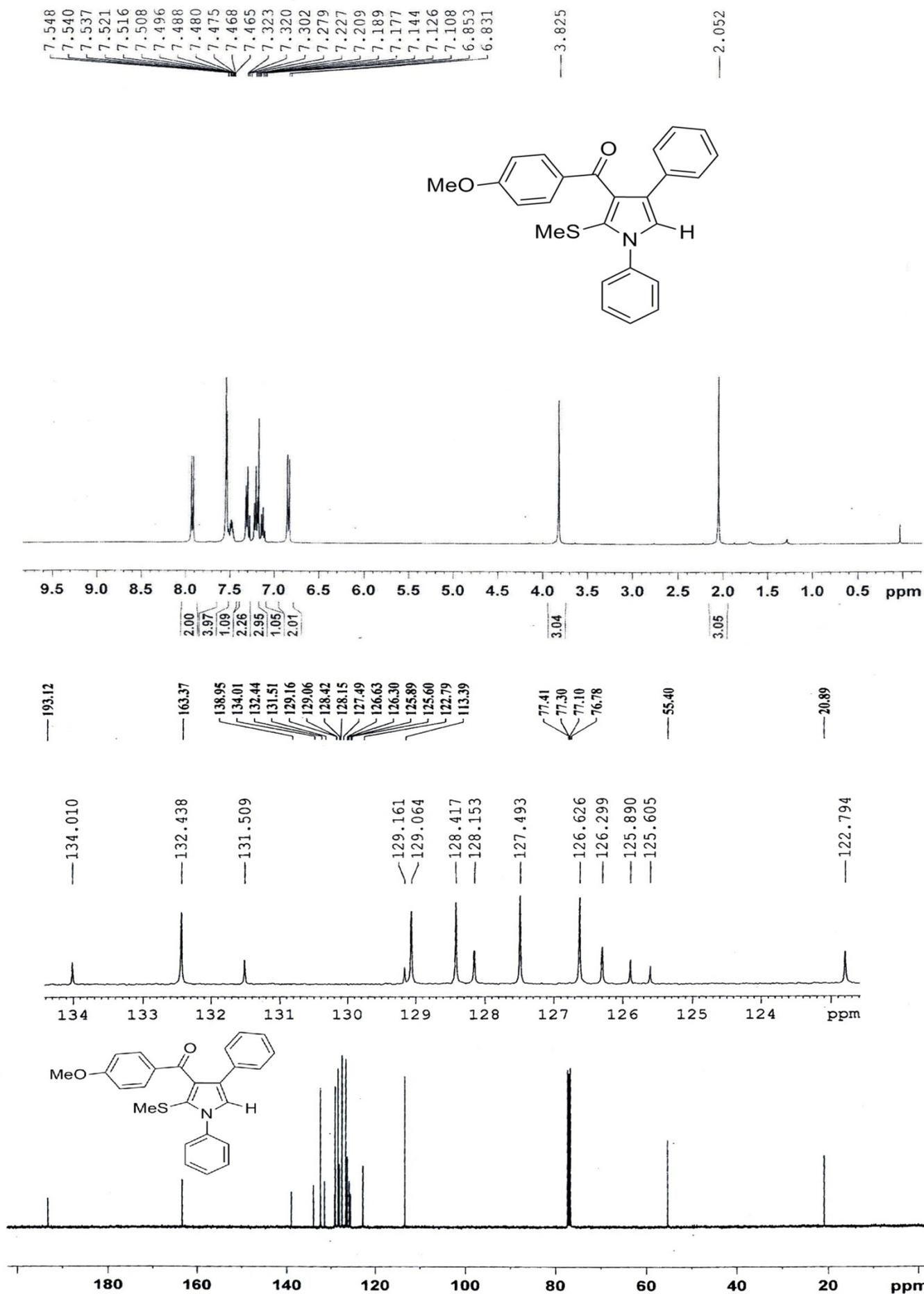
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 Acq. File: 04 Sep 2012SET1.wiff
 Instrument: RRL/AR/43-003

+Q1: 0.167 to 0.334 min from Sample 35 (#ASH(E-365)006\PROJ\ASENAPINE MALEATE) of 04 Sep 2012SET1.wiff (Turbo Spray), subtracted (0.66...)



Operator: Rajendra Kumar Gupta
 Workstation: GURIARD095
 Project: MASS SAMPLES

⑥3ca. 3-(4-Methoxybenzoyl)-2-(methylthio)-1,4-diphenylpyrrole

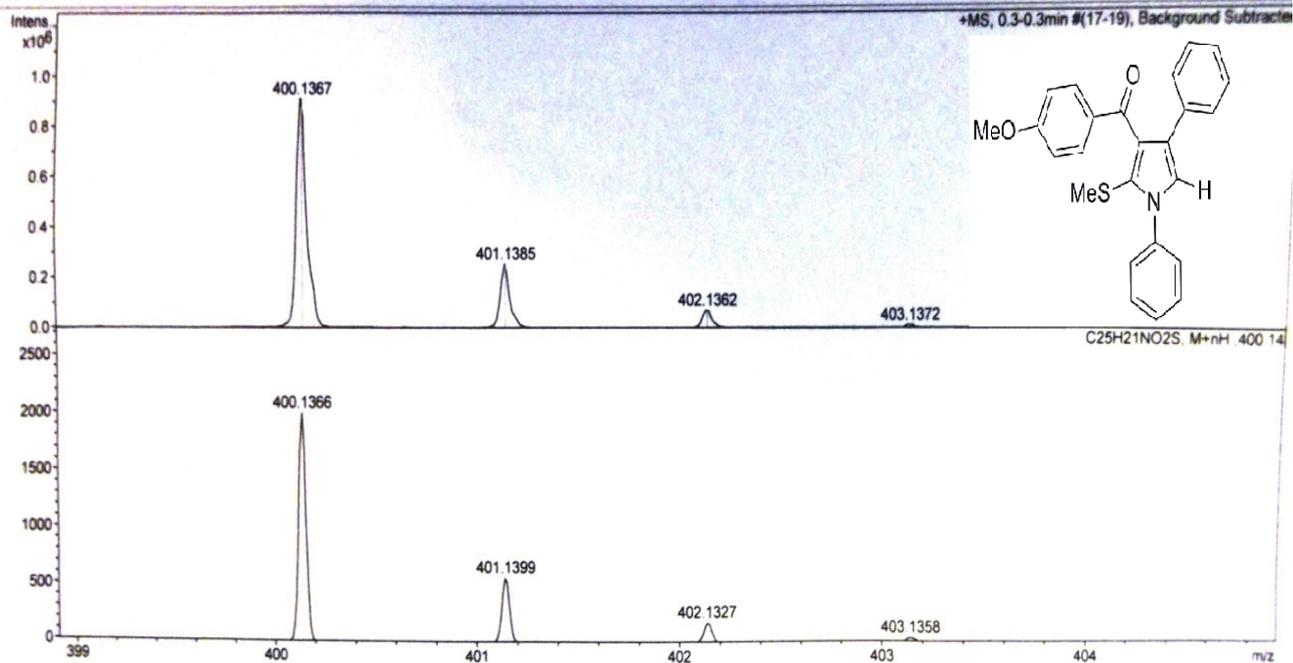


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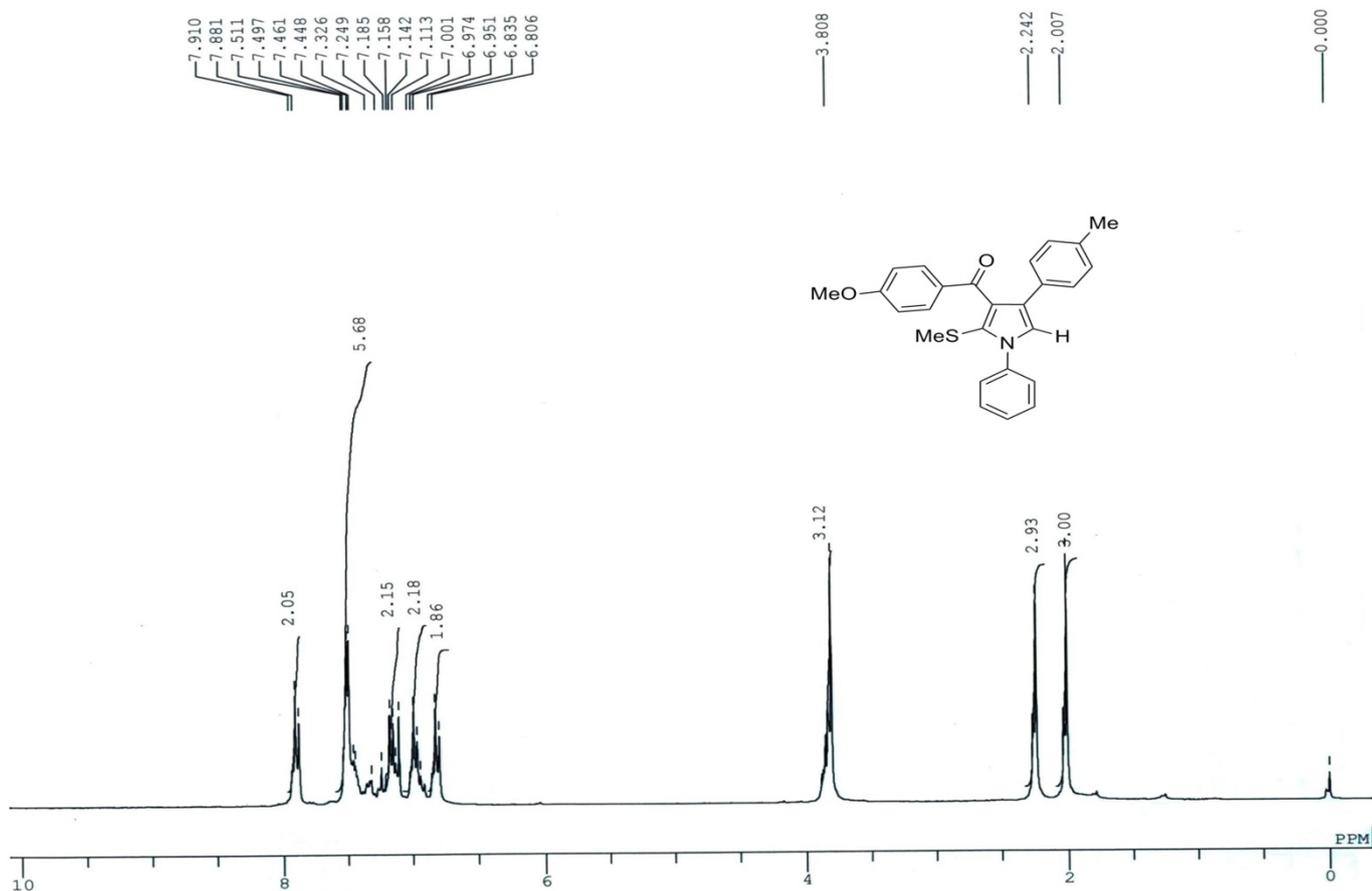
Operator BDAL@DE
 Instrument / Ser# microTOF 10237

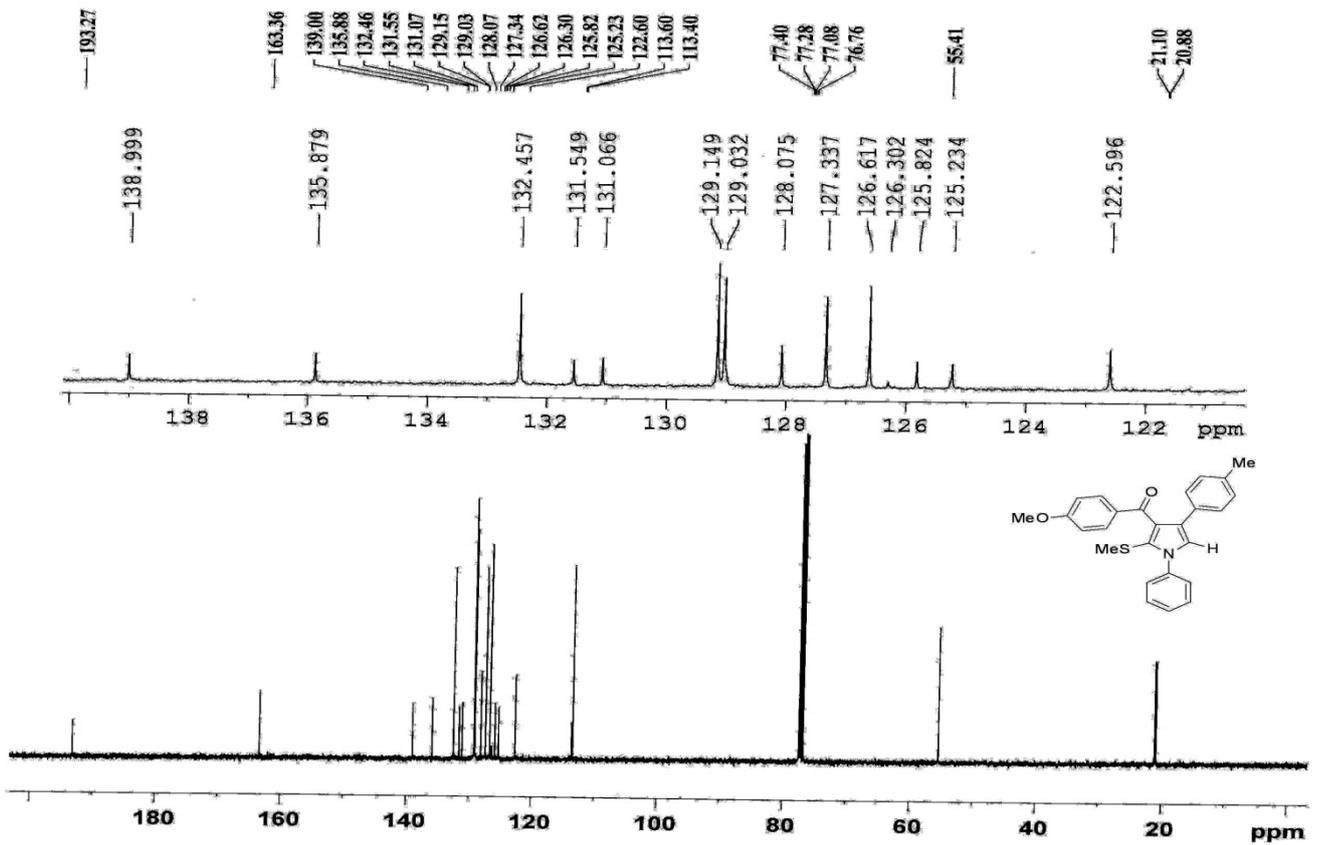
Acquisition Parameter

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Scan End	1600 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Source



⑦ 3cb.3-(4-Methoxybenzoyl)-2-(methylthio)-1-phenyl-4-(*p*-tolyl)pyrrole





Display Report

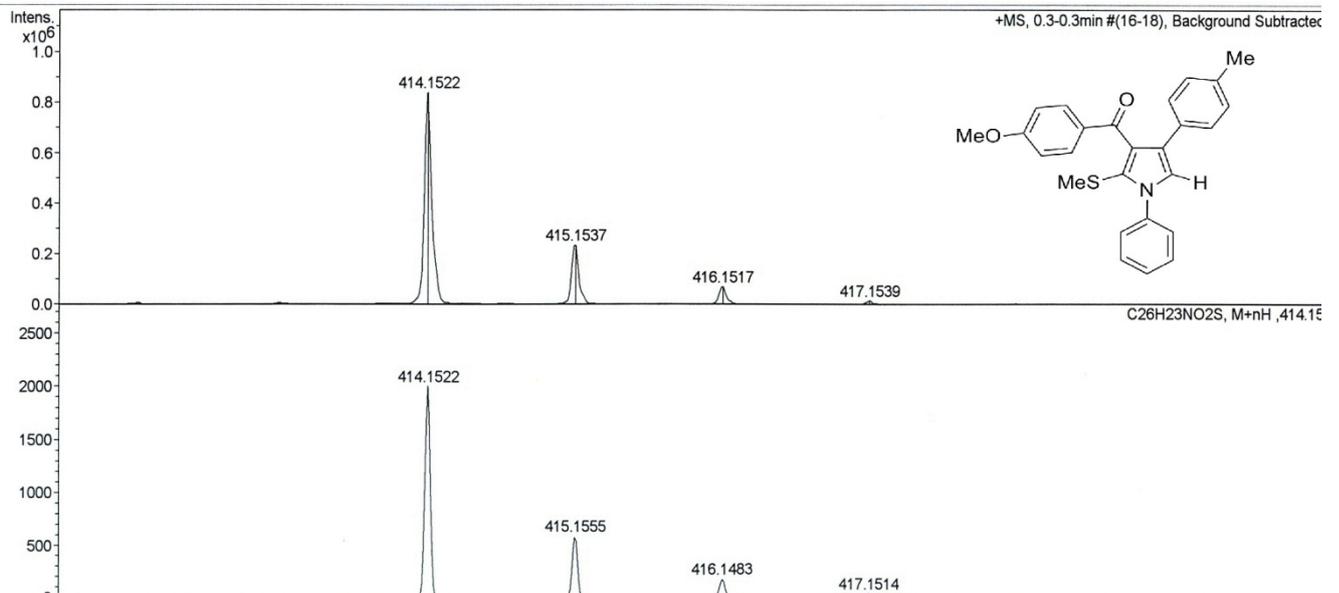
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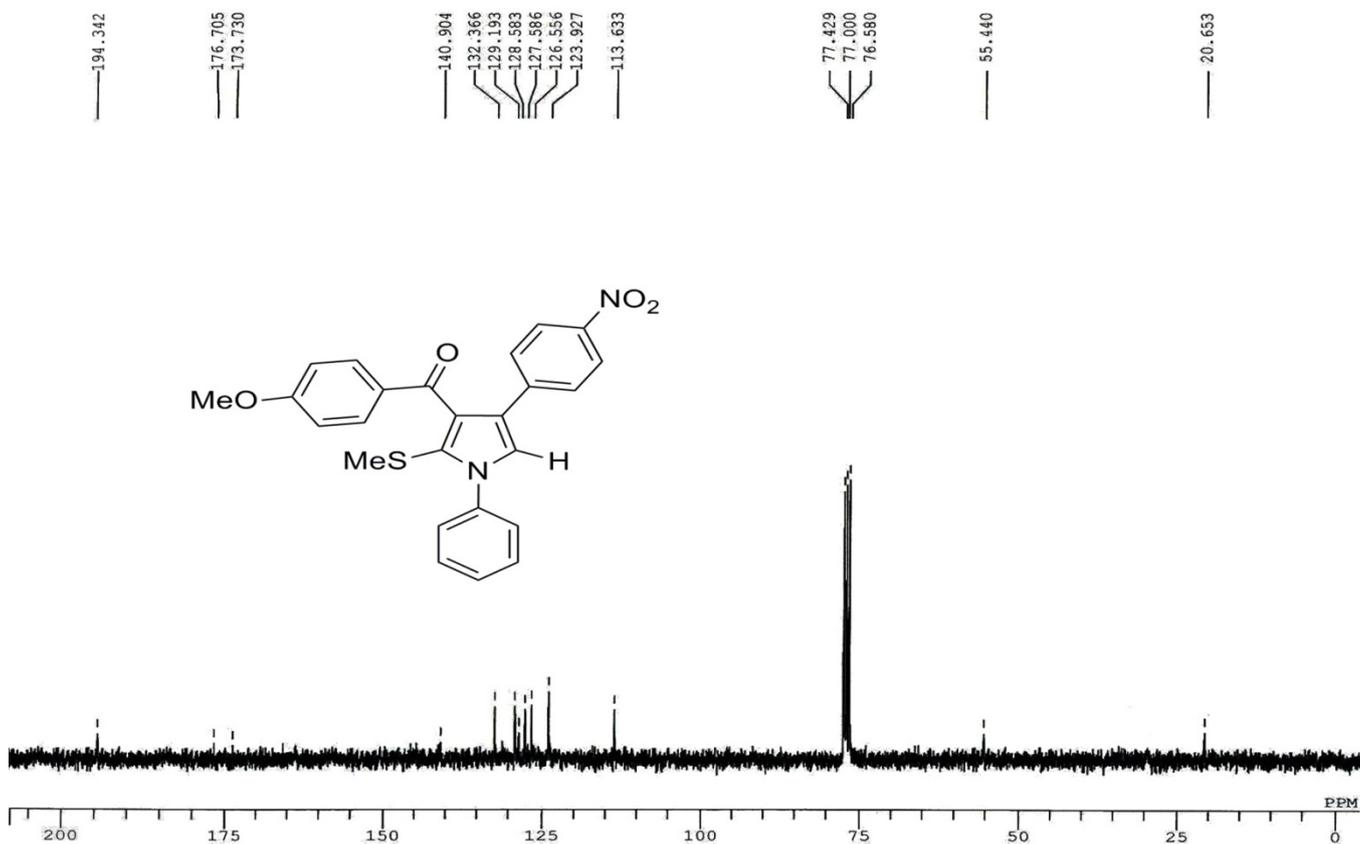
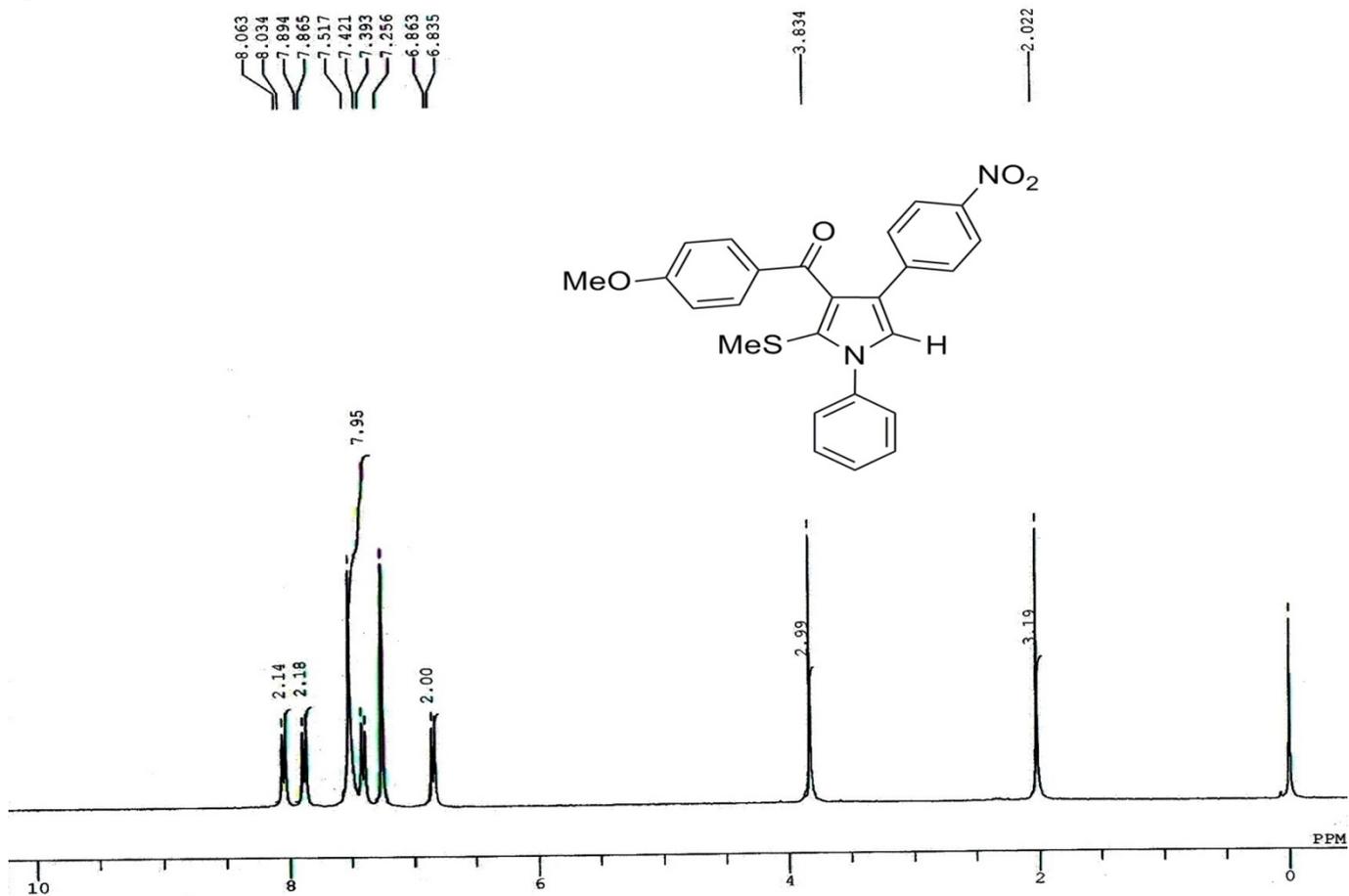
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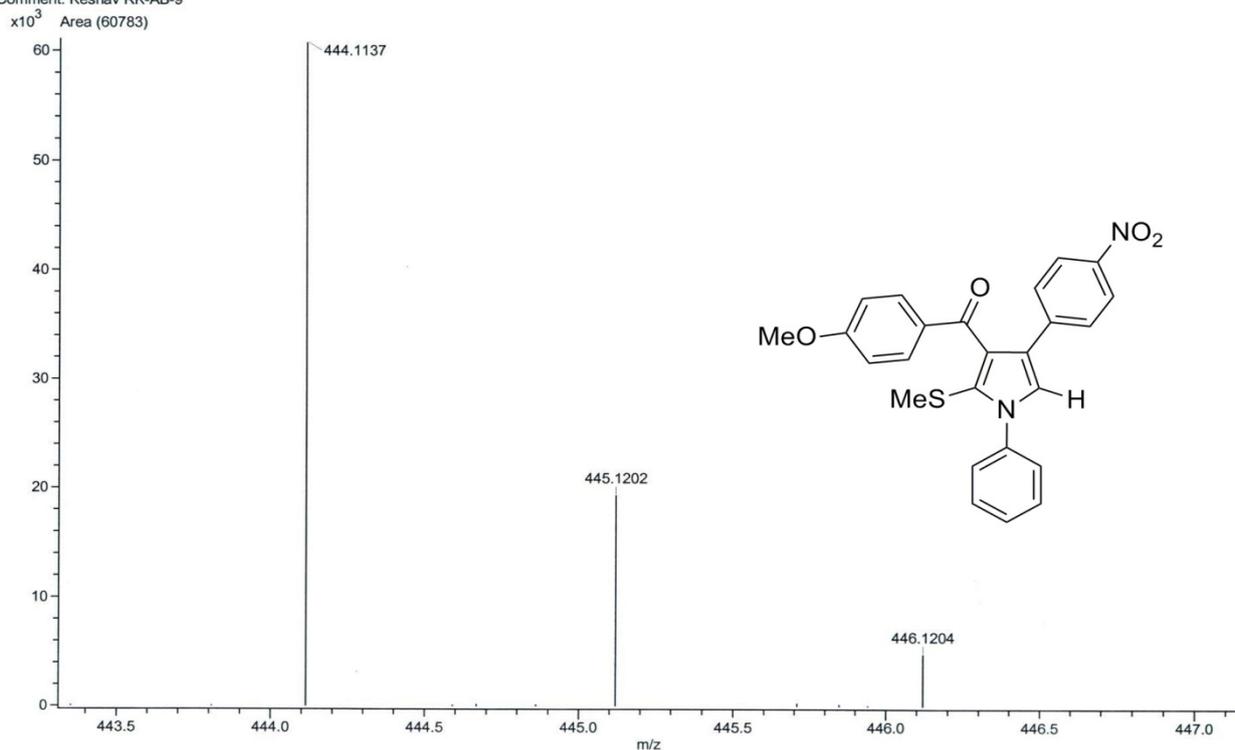


⑧ 3cd. 3-(4-methoxybenzoyl)-2-(methylthio)-4-(4-nitrophenyl)-1-phenylpyrrole

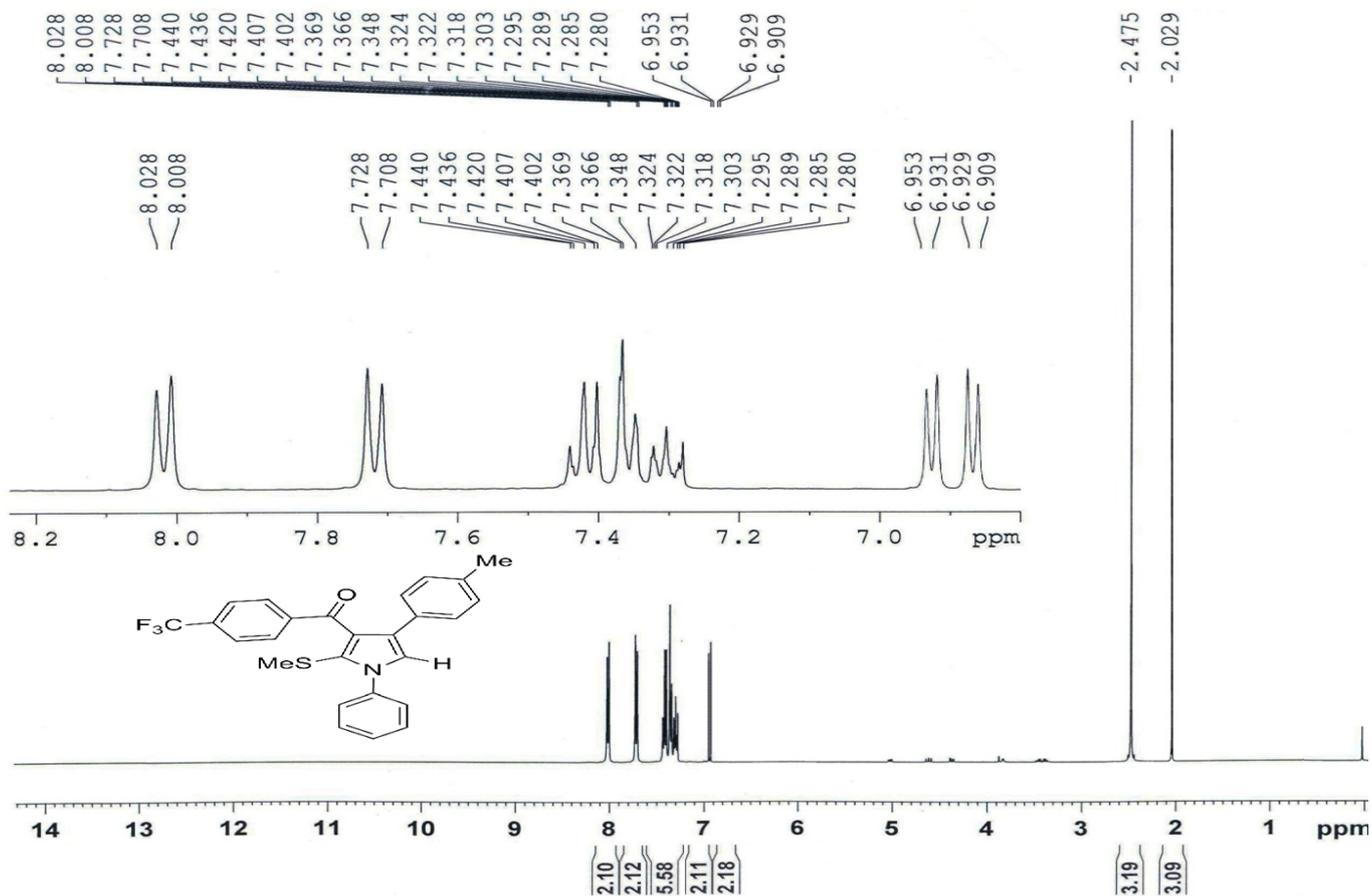


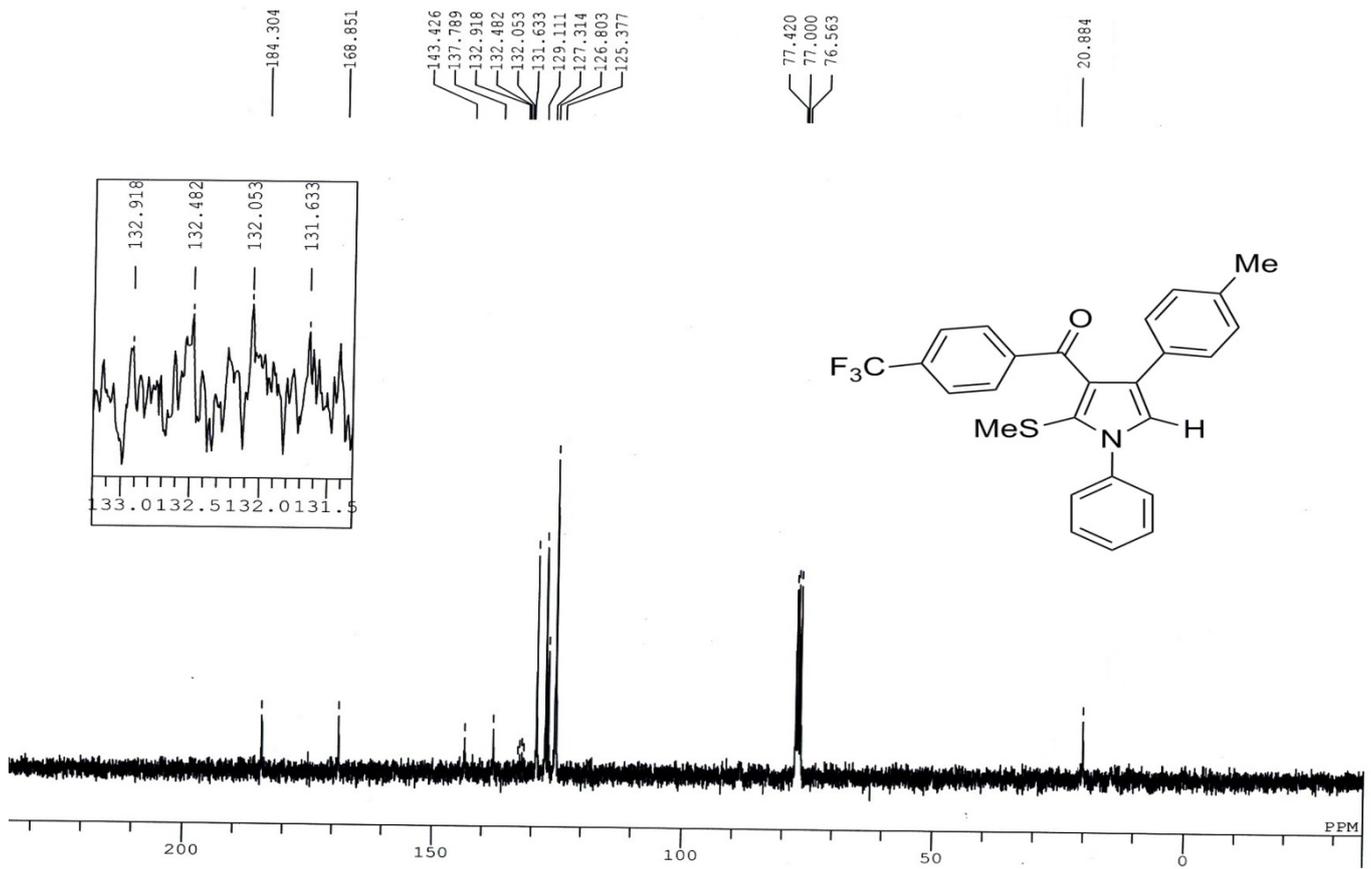
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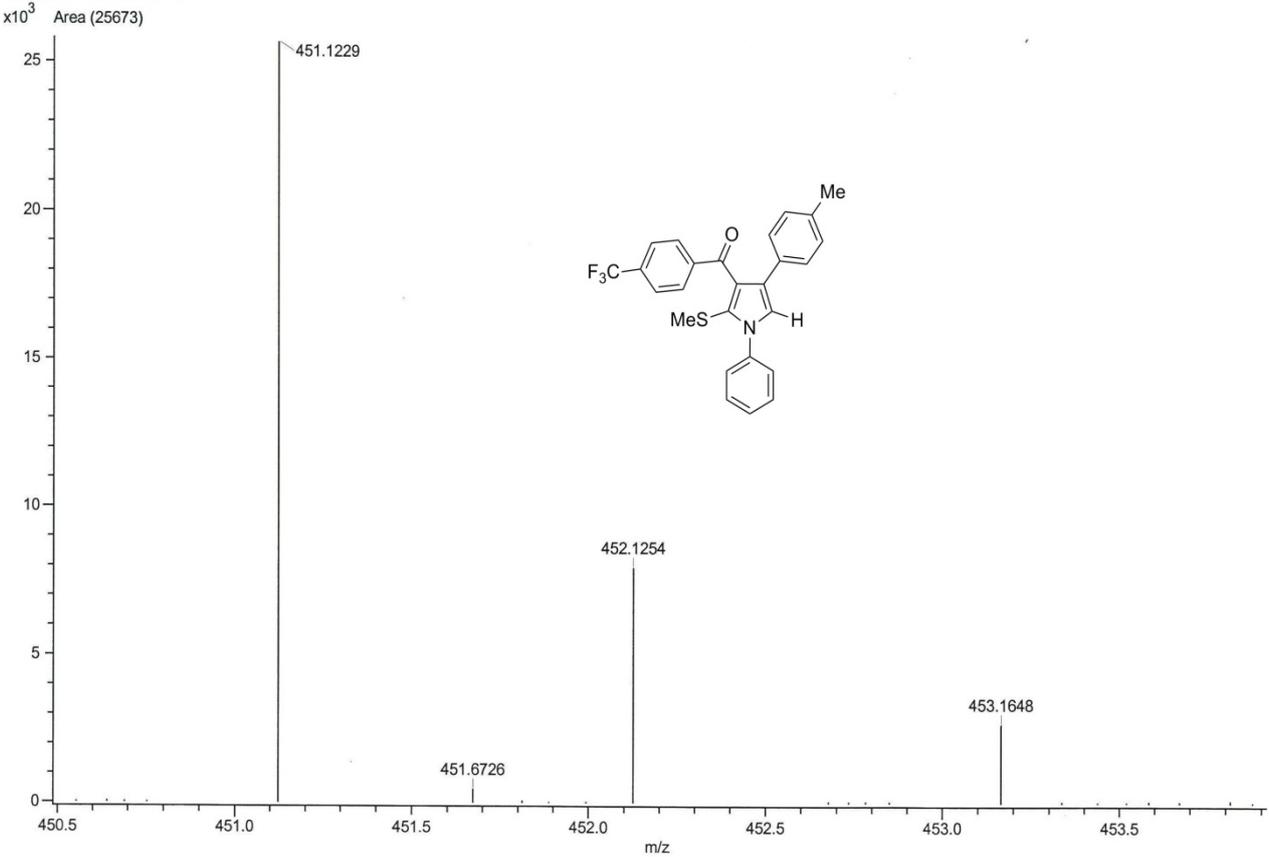
⑨3db.2-(Methylthio)-1-phenyl-4-(*p*-tolyl)-3-(4-trifluoromethyl)pyrrole



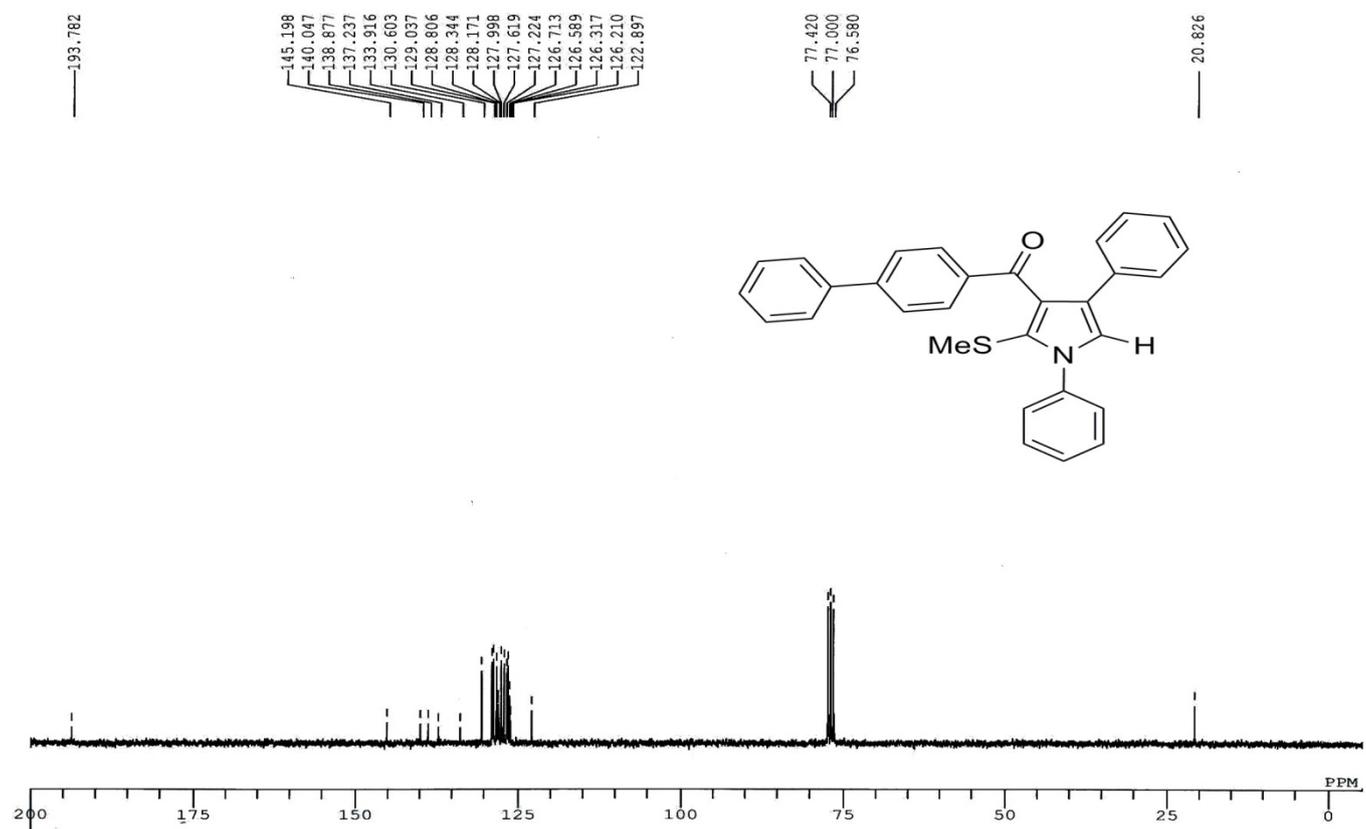
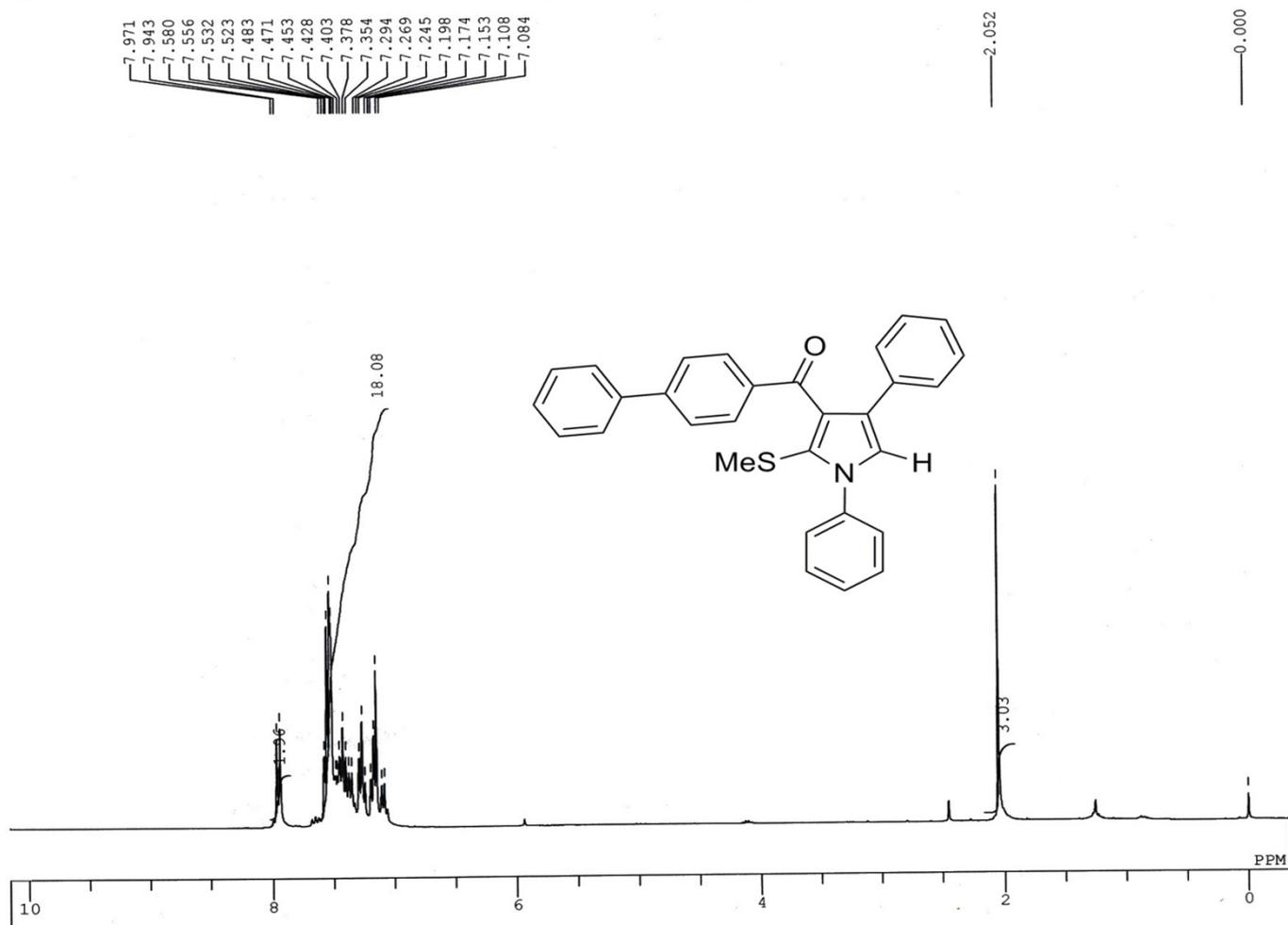


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⑩ 3ea. 1,4-Diphenyl-2-(methylthio)-3-(4-phenylbenzoyl)pyrrole

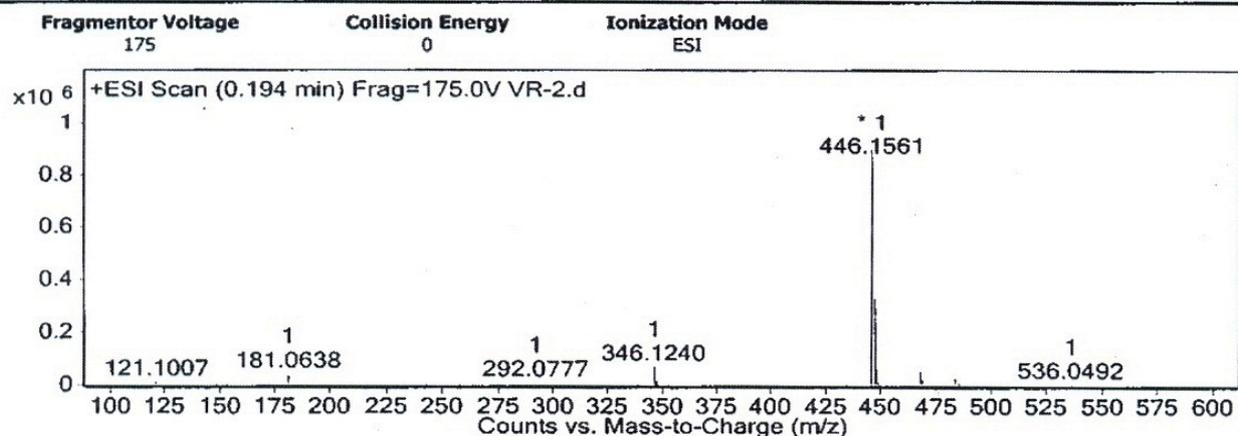


Qualitative Analysis Report

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Comment			

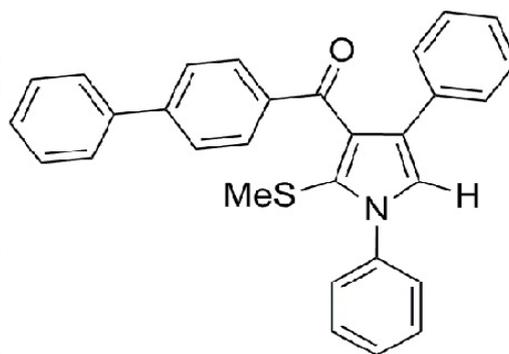
Acquisition SW Version 6200 series TOF/6500 series
 Q-TOF B.05.00 (B5042.0)

User Spectra

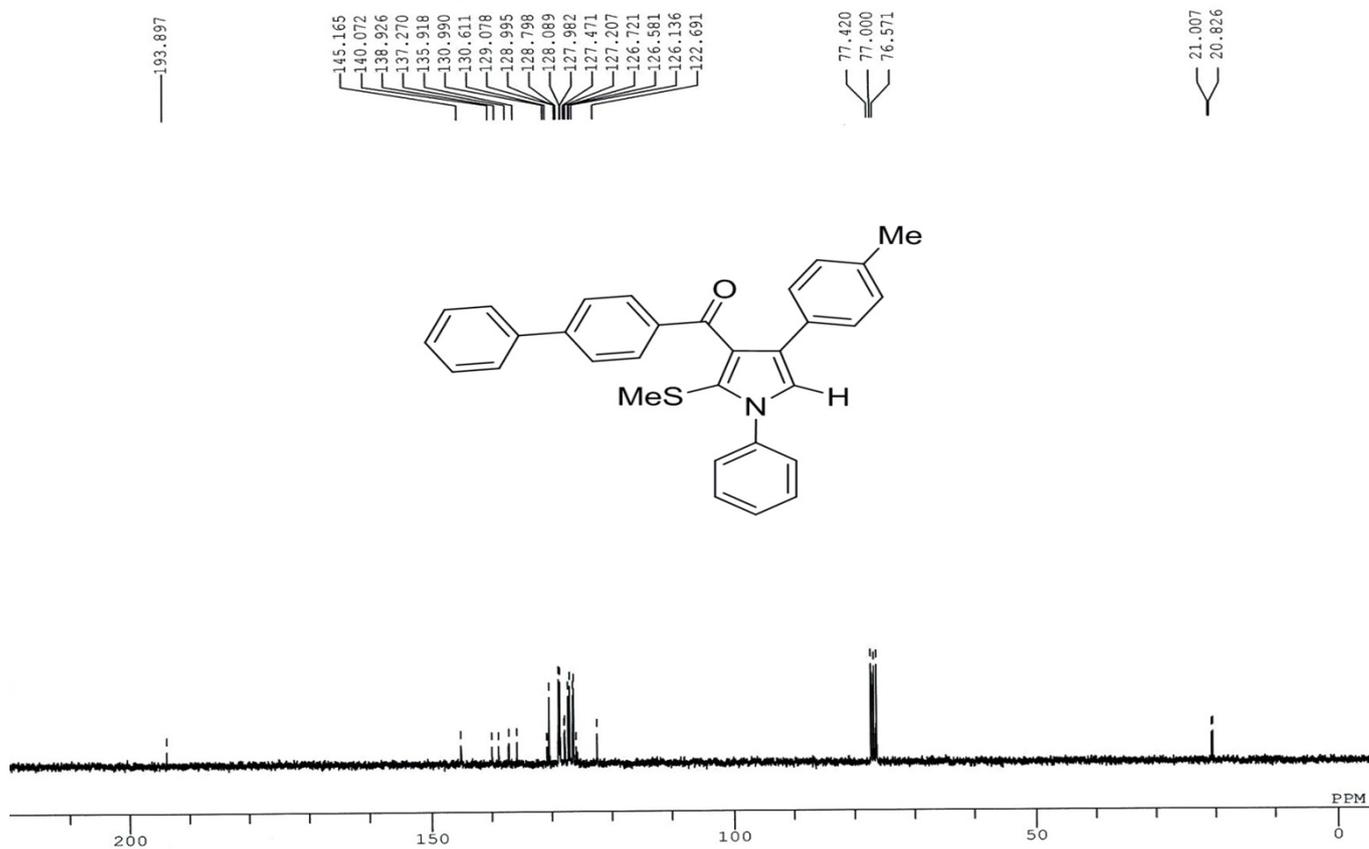
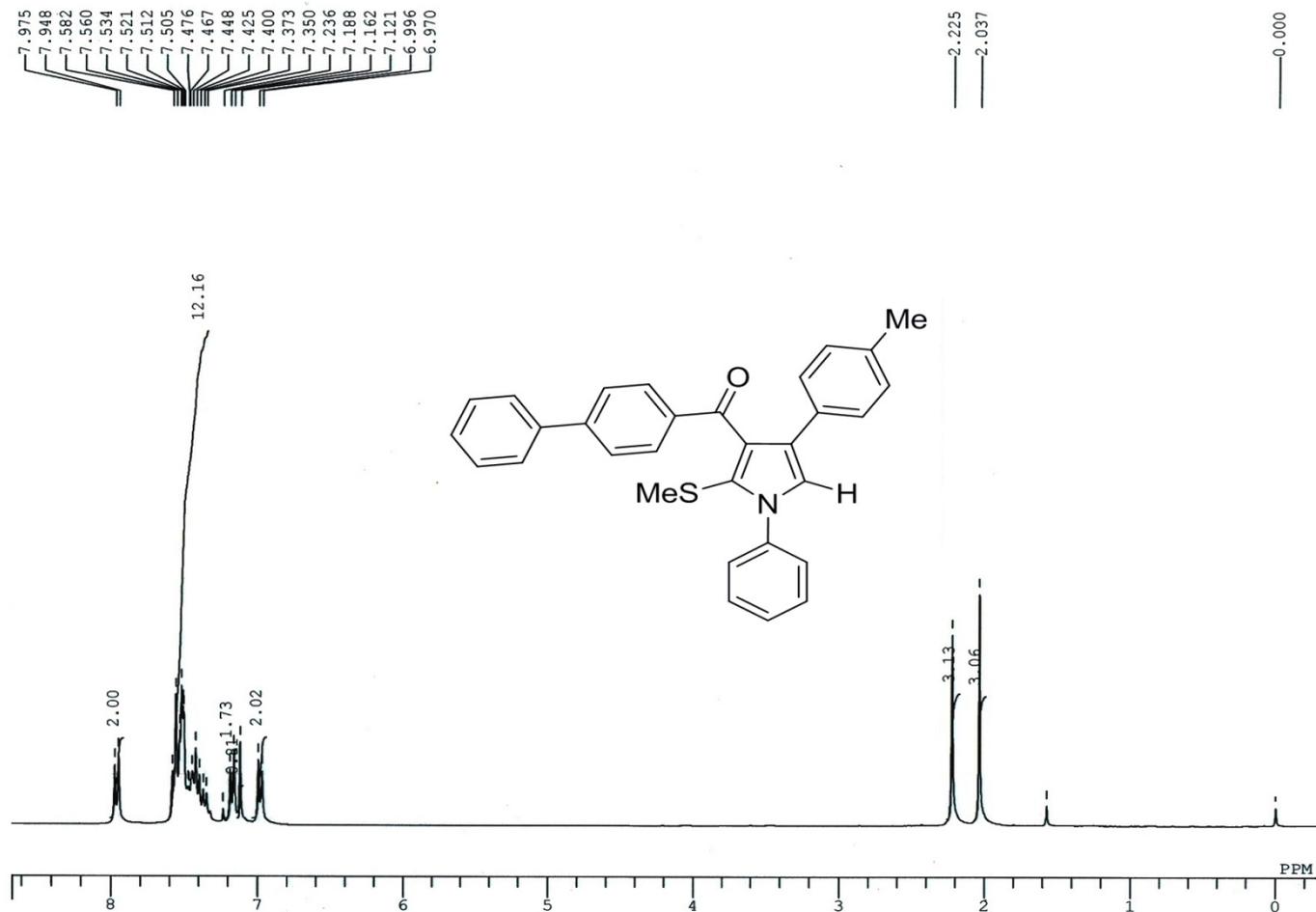


Peak List

m/z	z	Abund	Formula	Ion
109.9432		5759.35		
121.1007		10097.73		
122.9633		8213.98		
152.0615		7541.29		
153.0686	1	7778.12		
181.0638	1	40027.32		
182.0677	1	5079.79		
292.0777	1	7707.75		
338.3404	1	7243.15		
346.124	1	68521.2		
347.1273	1	16675.82		
446.1561	1	903105.81	C30 H24 N O S	(M+H)+
447.1587	1	331320.91	C30 H24 N O S	(M+H)+
448.1575	1	66786.55	C30 H24 N O S	(M+H)+
449.1571	1	13269.06	C30 H24 N O S	(M+H)+
462.1498	1	5441.58		
468.1365	1	56181.04		
469.1393	1	19012.19		
484.1103	1	30586.4		
485.1139	1	9621.76		



⑪ 3eb. 2-(Methylthio)-1-phenyl-3-(4-phenylbenzoyl)-4-(*p*-tolyl)pyrrole



Display Report

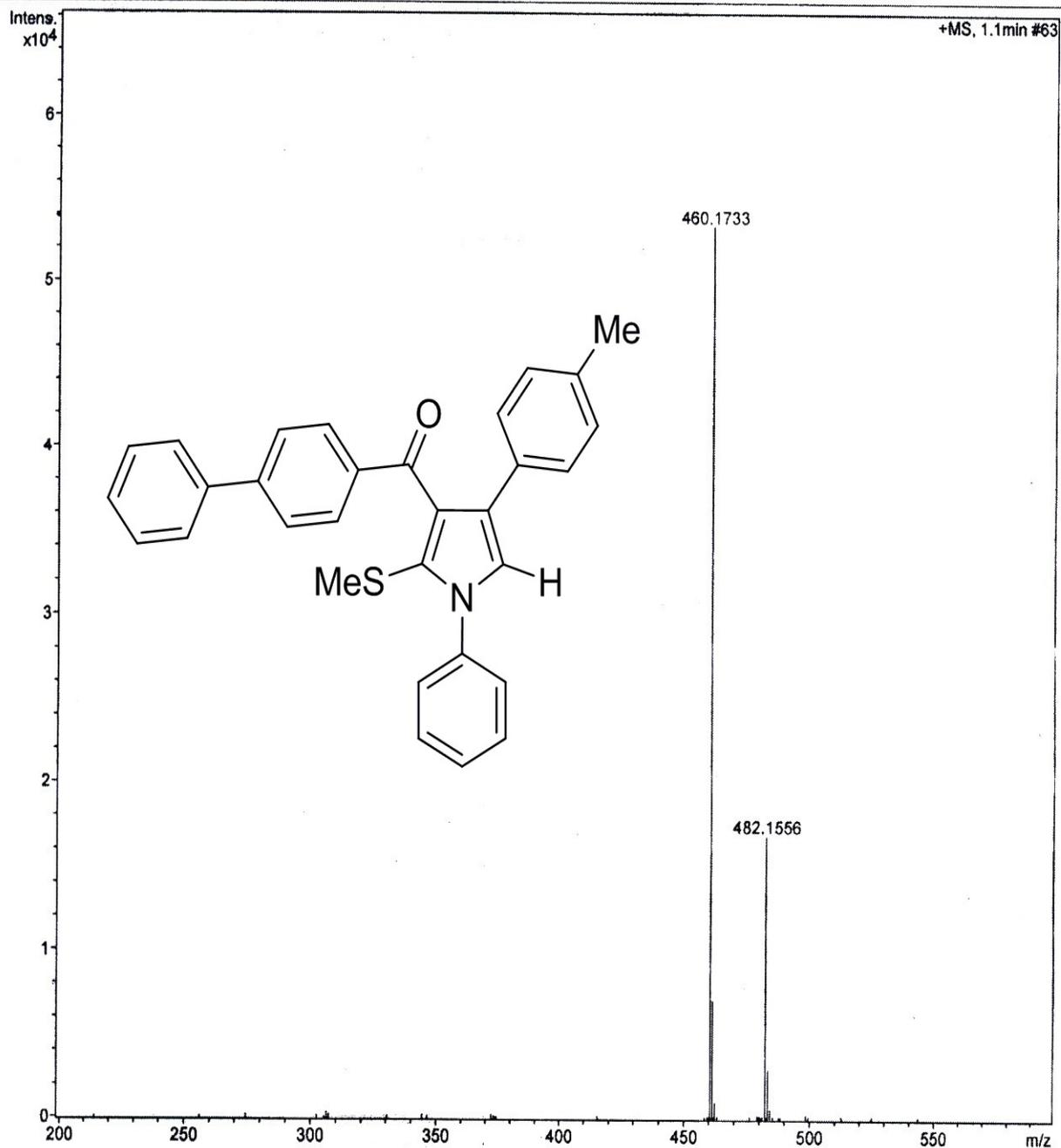
Analysis Info

Analysis Name D:\Data\2013\PROF.DBJ\ANGCSR-1.d
Method TL-P.m
Sample Name GCSR-1-CHCL3
Comment

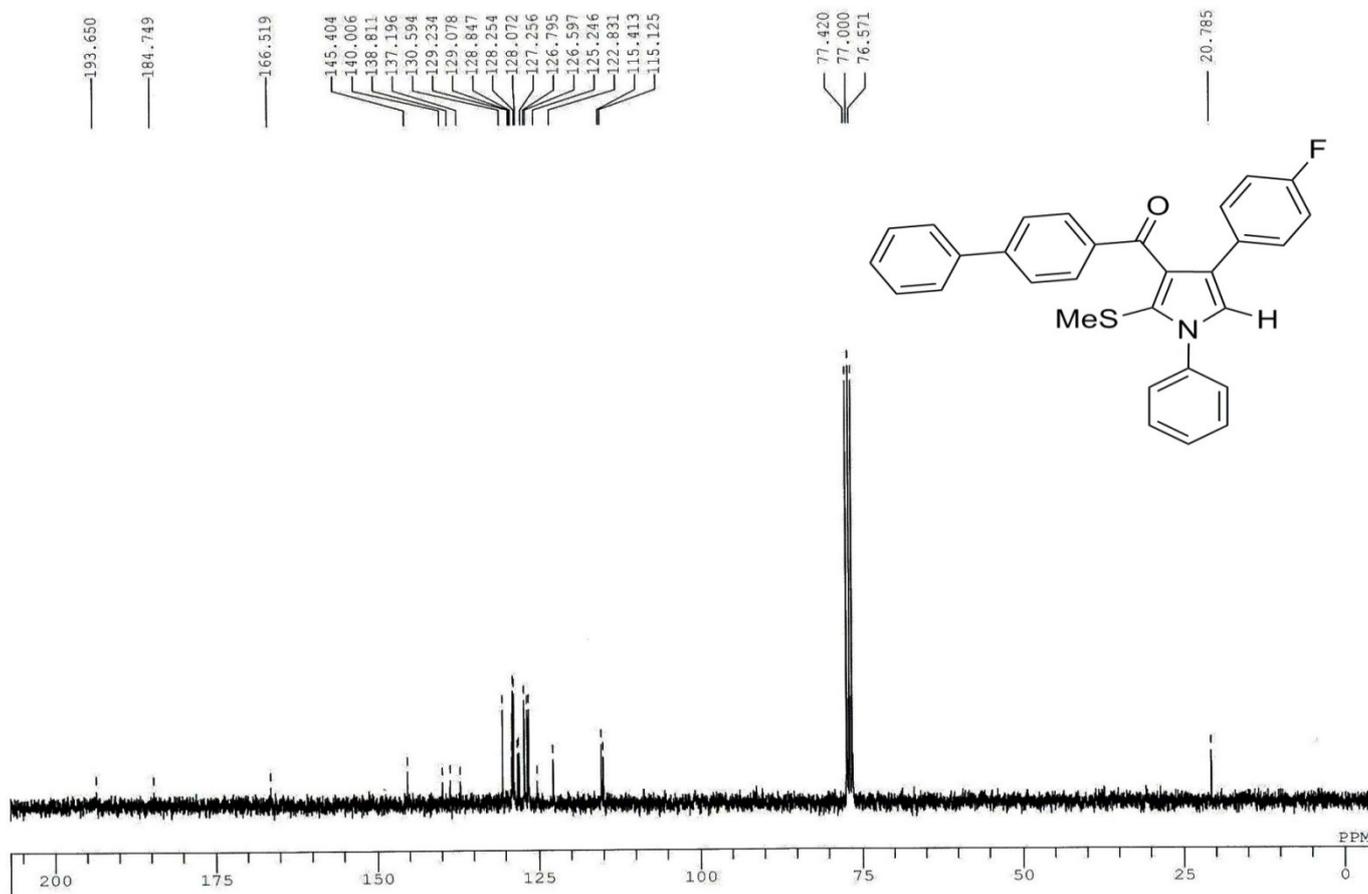
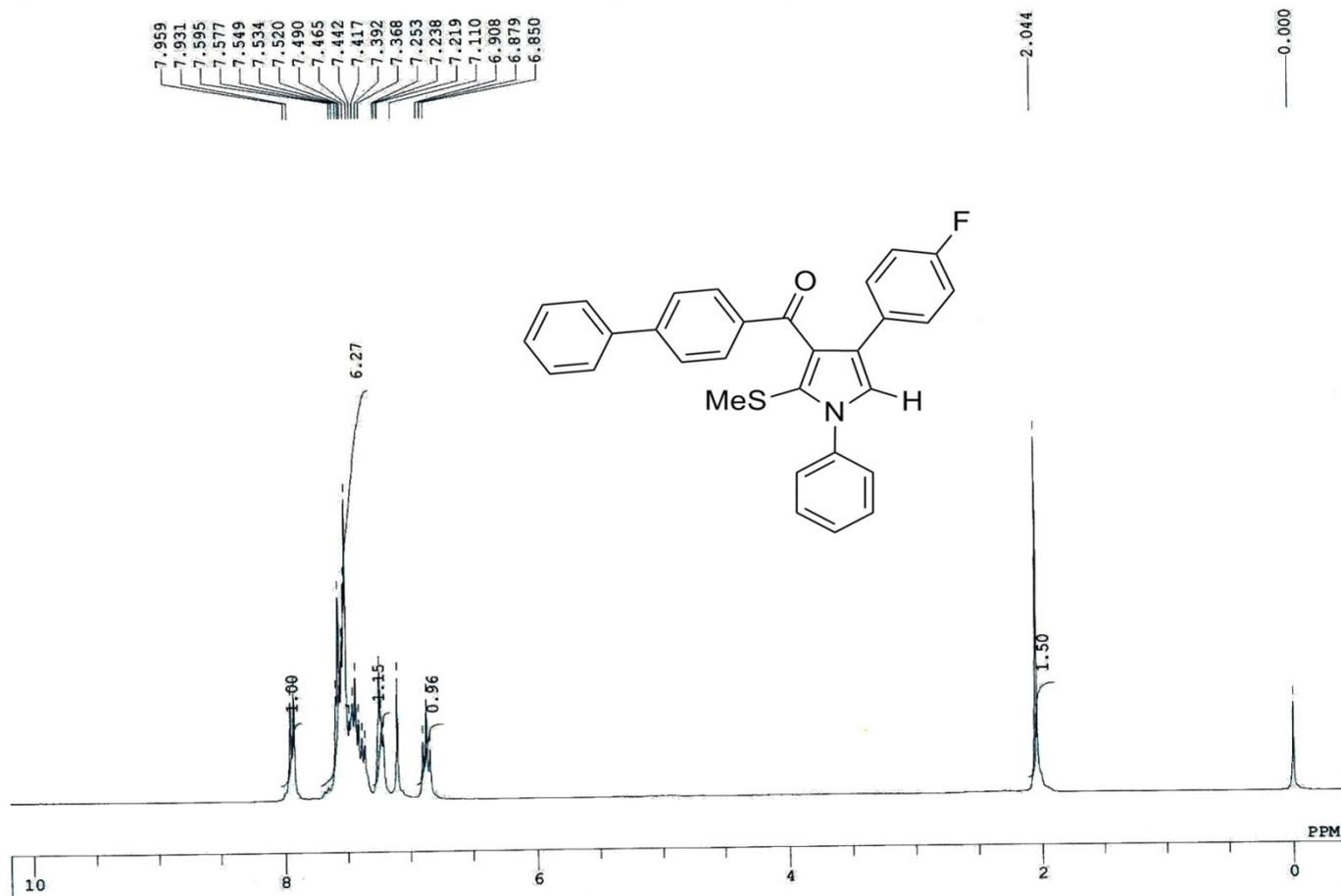
Acquisition Date 1/5/2013 3:21:45 PM
Operator Rajesh Vashisth
Instrument maXis 10138

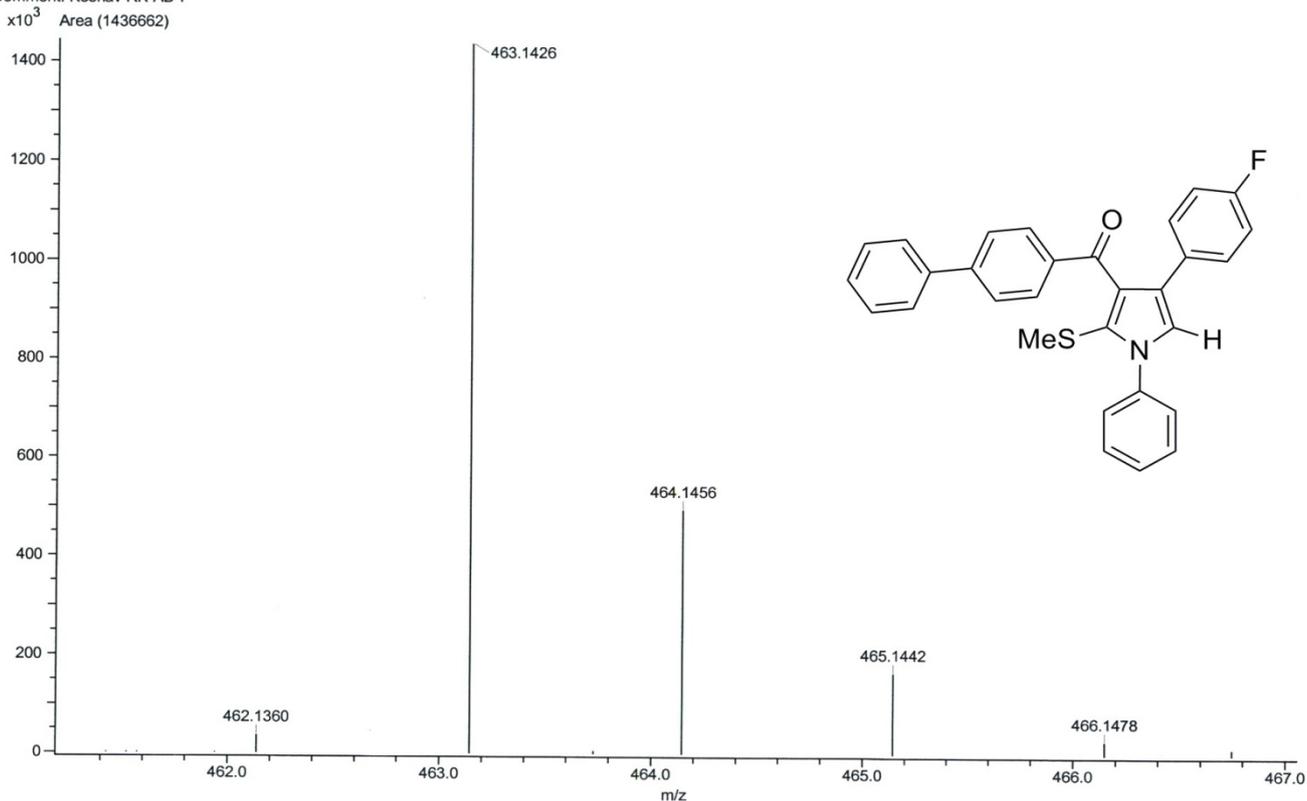
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	4.4 psi
Focus	Not active	Set Capillary	3800 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	600 m/z	Set Collision Cell RF	350.0 Vpp	Set Divert Valve	Waste

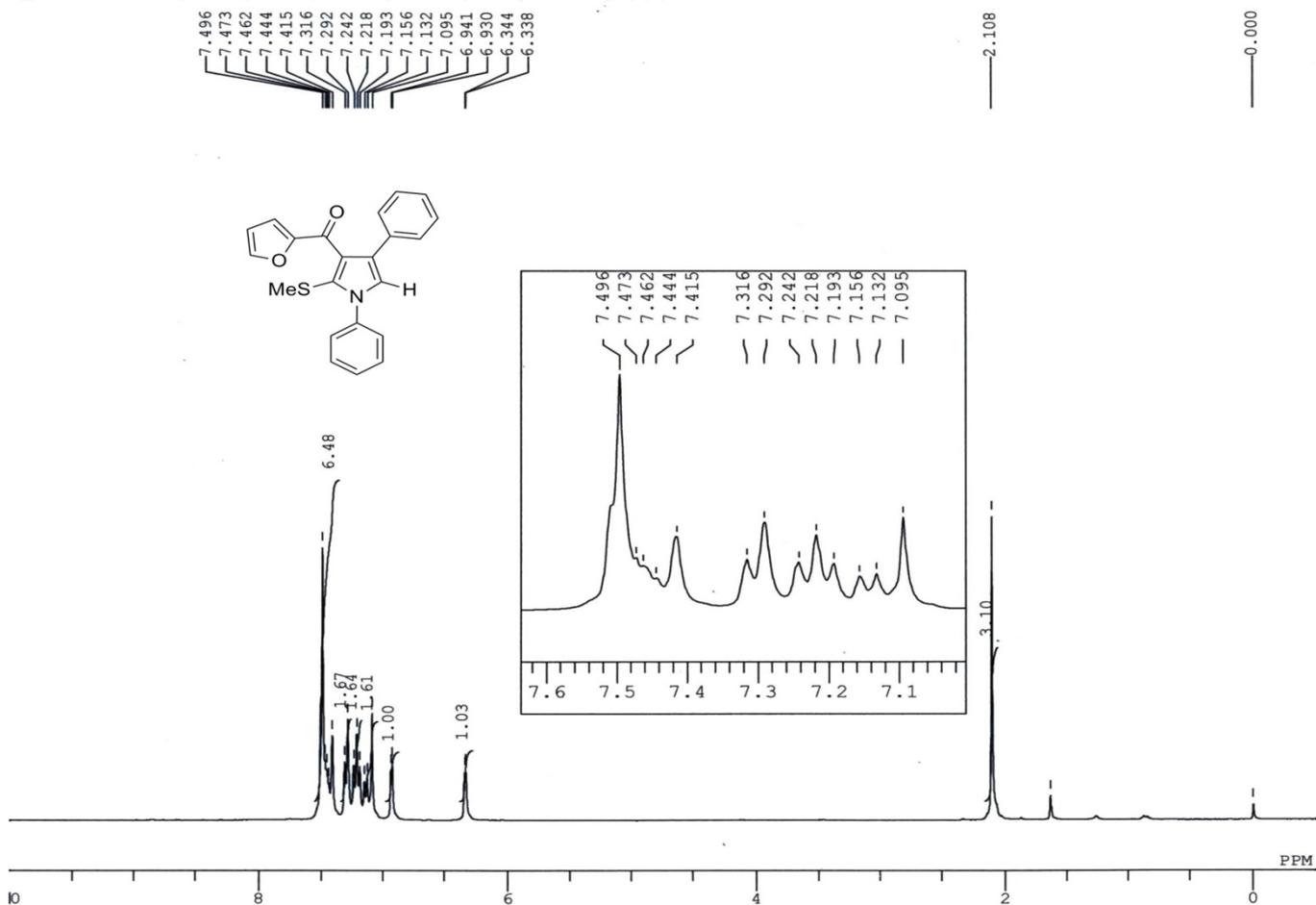


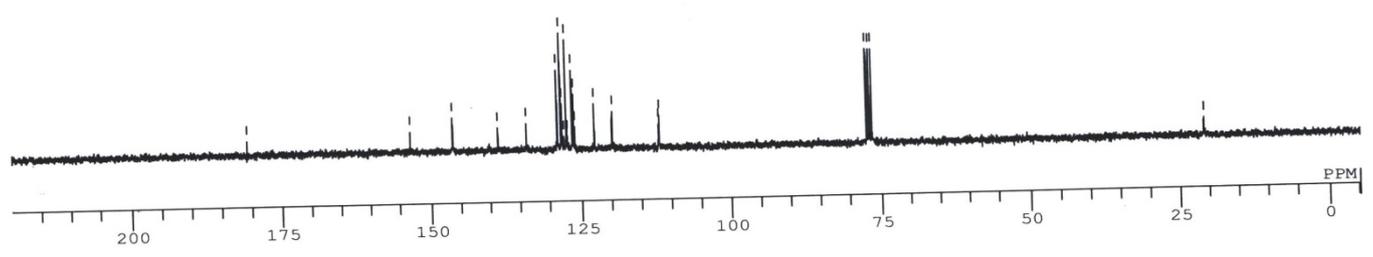
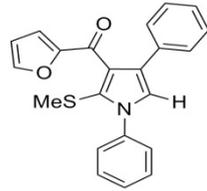
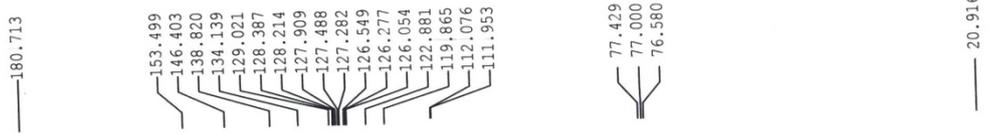
⑫ 3ee. 4-(4-Fluorophenyl)-2-(Methylthio)-1-phenyl-3-(4-phenylbenzoyl)pyrrole





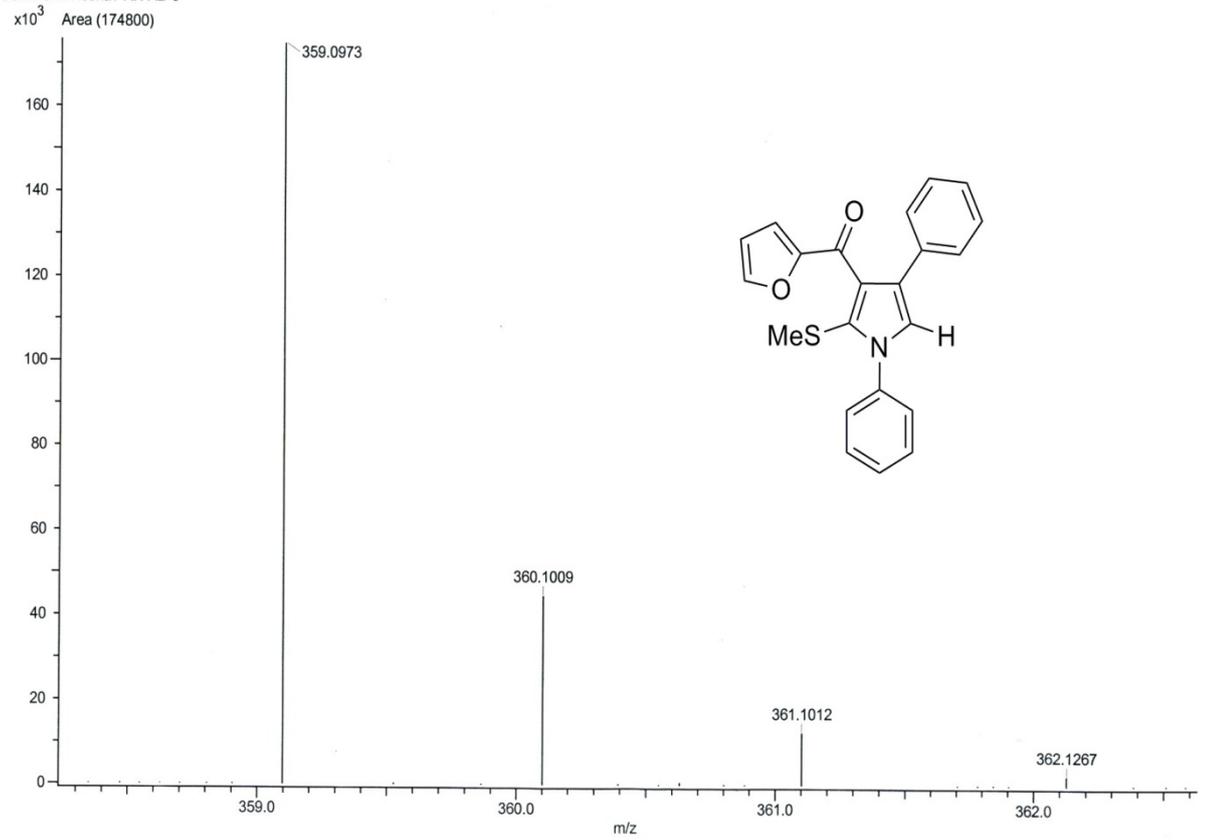
⑬ 3fa. 1,4-Diphenyl-2-(methylthio)-3-(2-furoyl)pyrrole



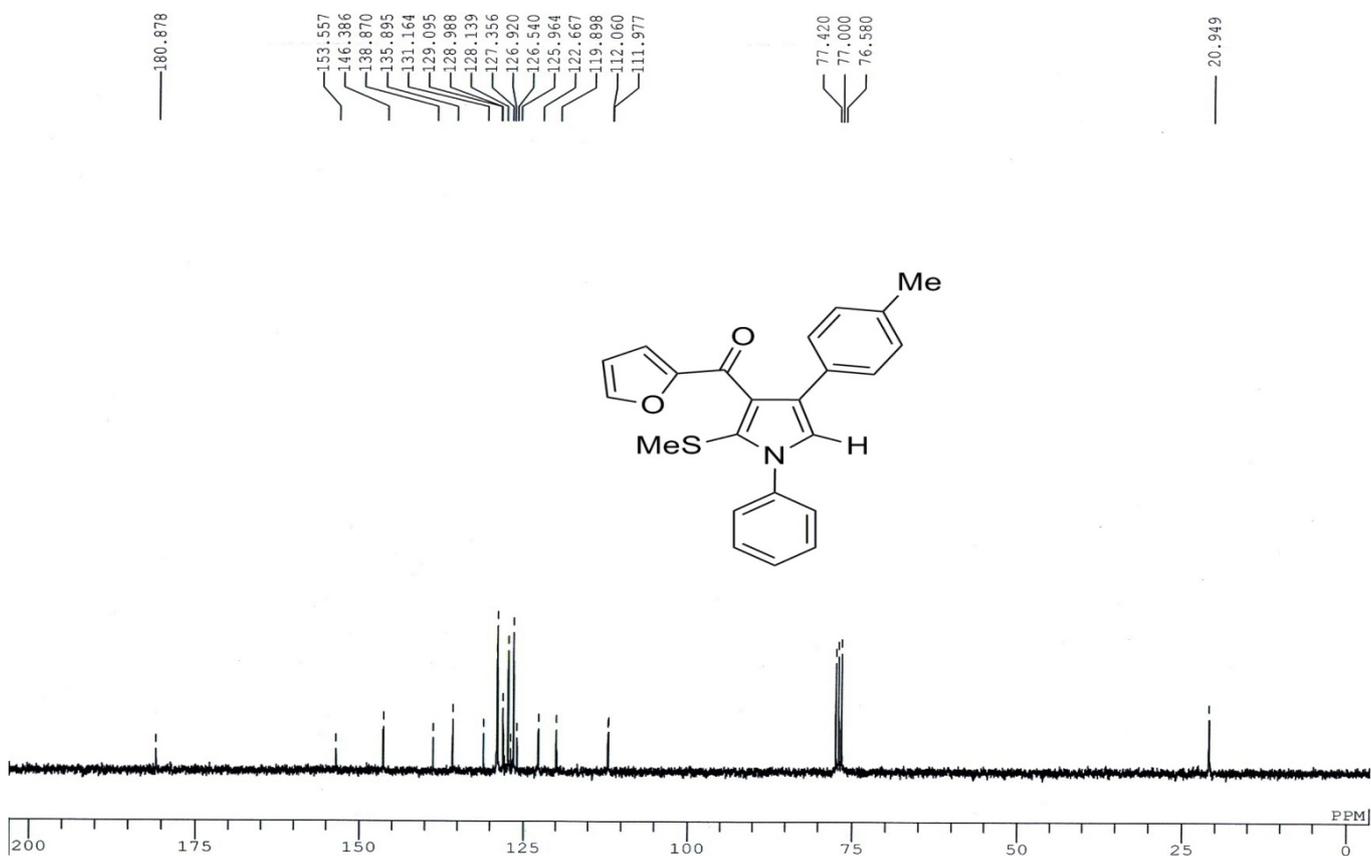
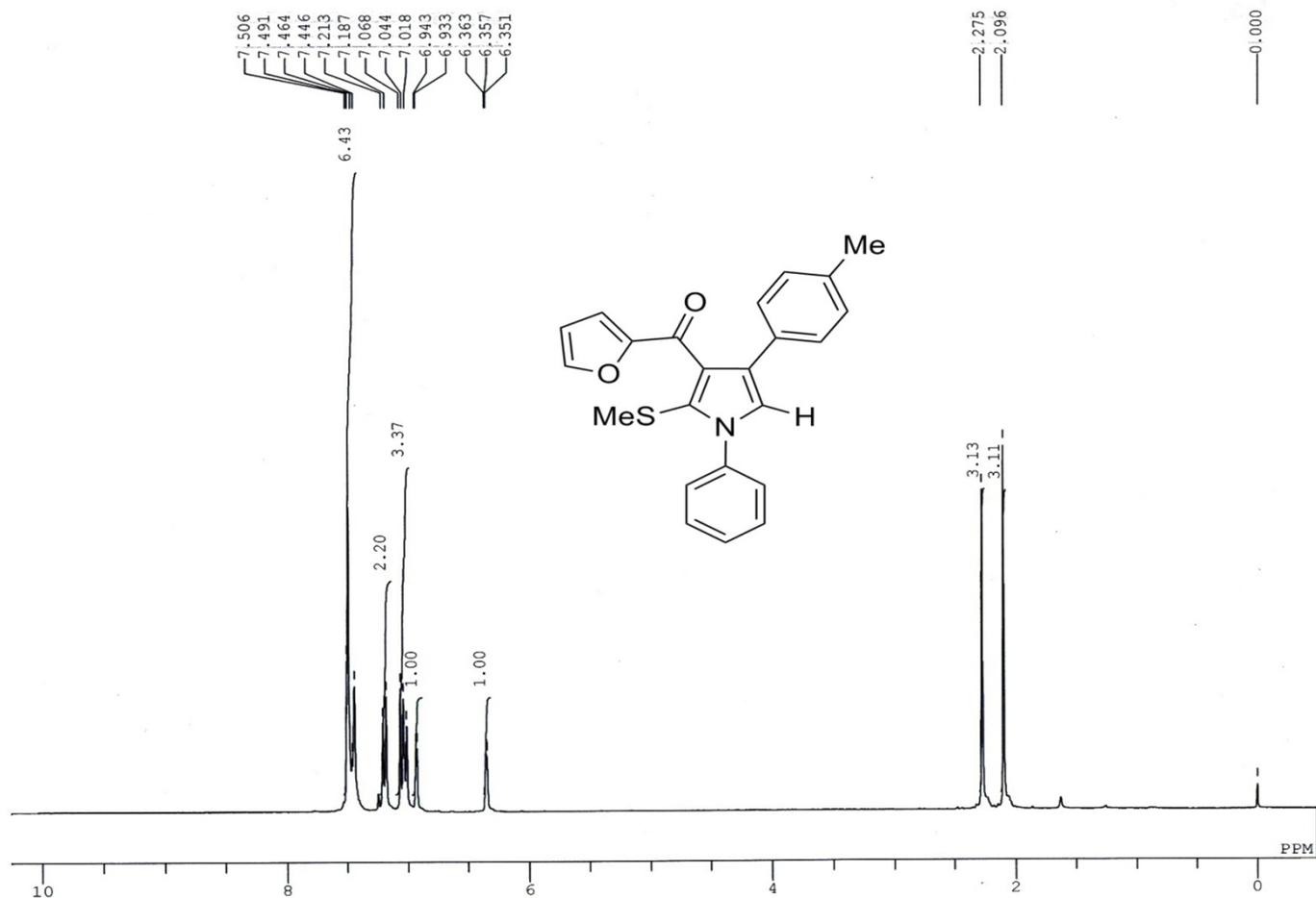


Acq. Data Name: keshav130513-2
 Creation Parameters: Average(MS[1] Time:0.32..0.60)
 Comment: Keshav RK-AB-8

Experiment Date/Time: 5/13/2013 3:28:30 PM
 Ionization Mode: EI+



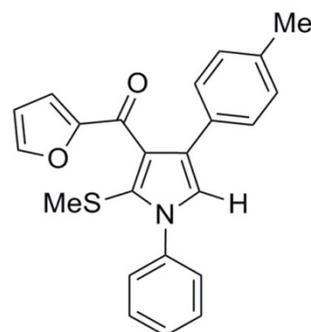
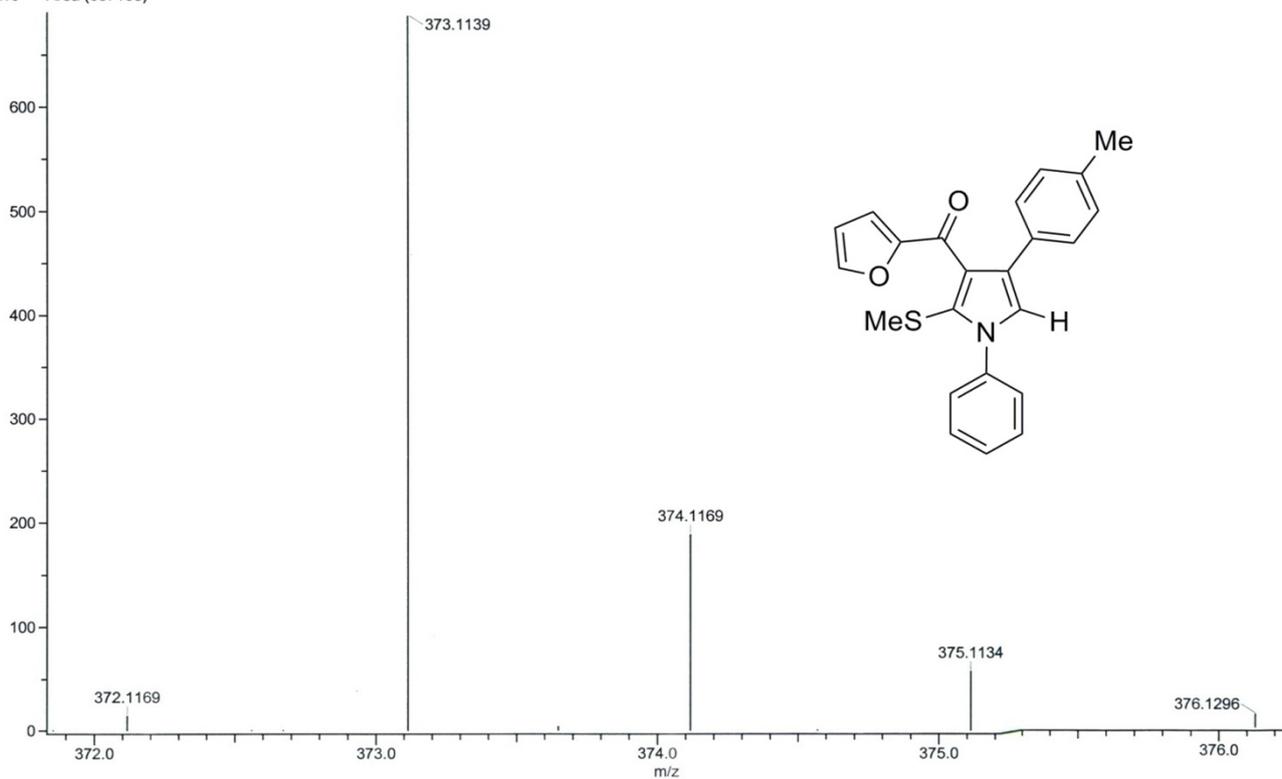
⑭ 3fb.3-(2-Furoyl)-2-(methylthio)-1-phenyl-4-(*p*-tolyl)pyrrole



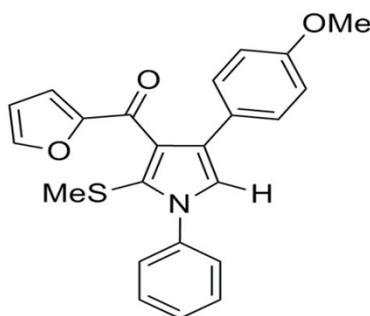
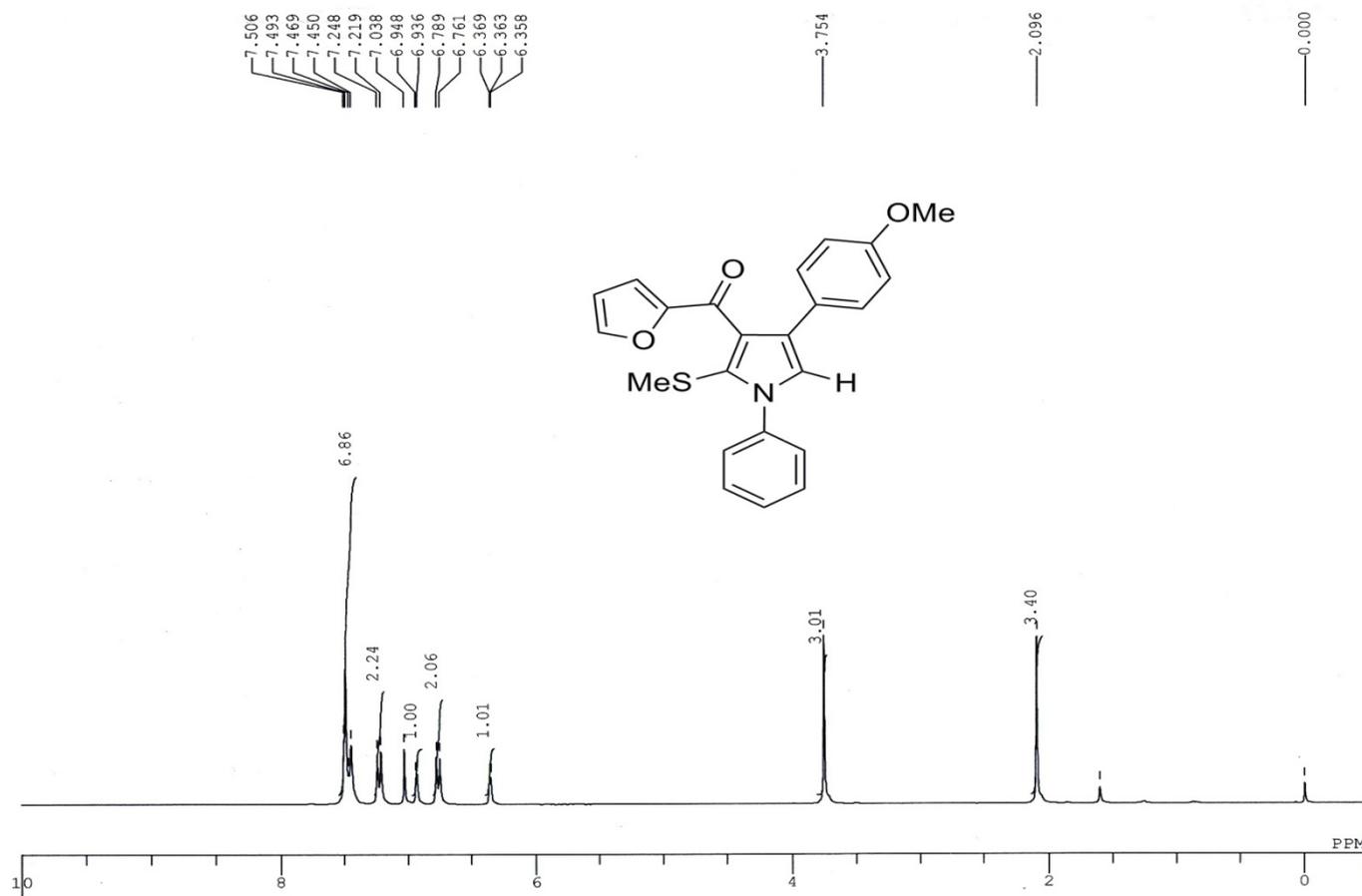
Acq. Data Name: keshav130510-4
Creation Parameters: Average(MS[1] Time:0.72..0.76)
Comment: Keshav RK-AB-1

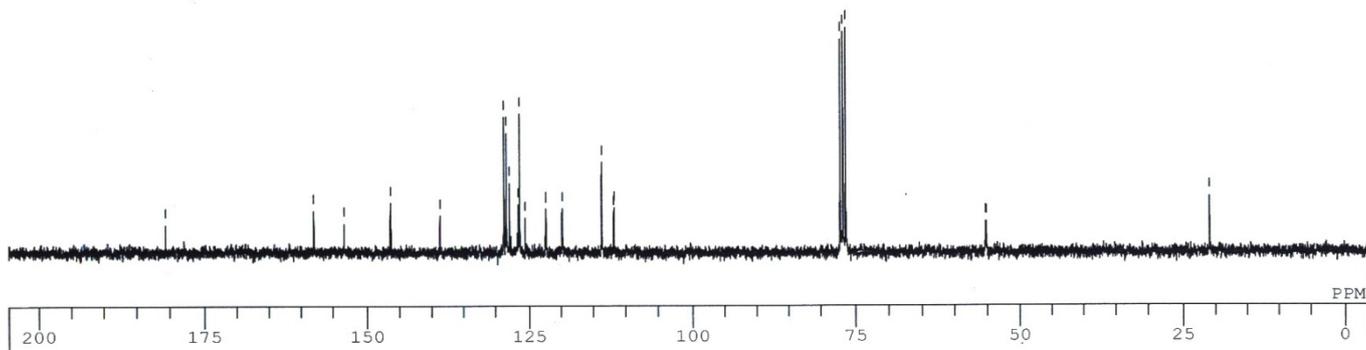
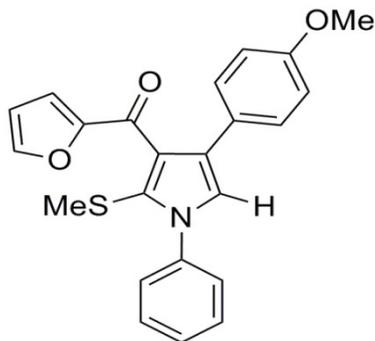
Experiment Date/Time: 5/10/2013 11:59:34 AM
Ionization Mode: EI+

$\times 10^3$ Area (687108)



⑮ 3fc.3-(2-Furoyl)-4-(4-methoxyphenyl)-2-(methylthio)-1-phenylpyrrole

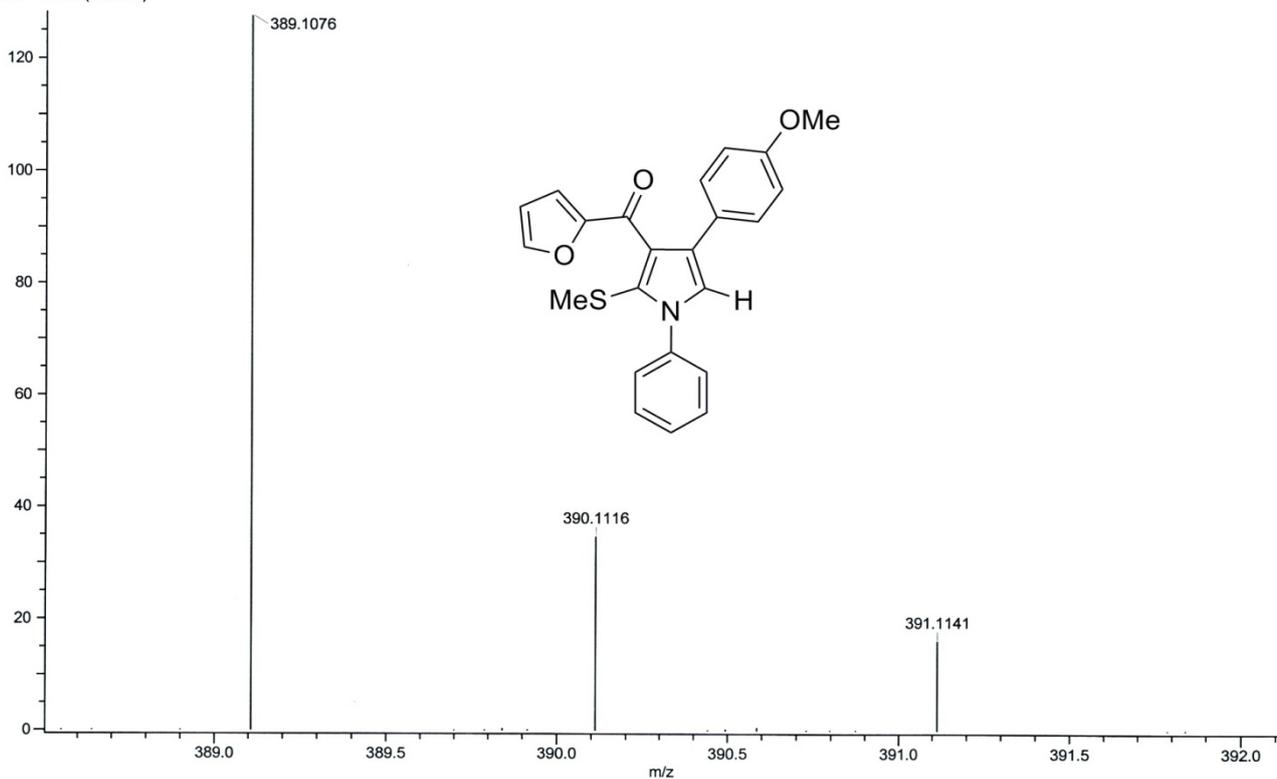




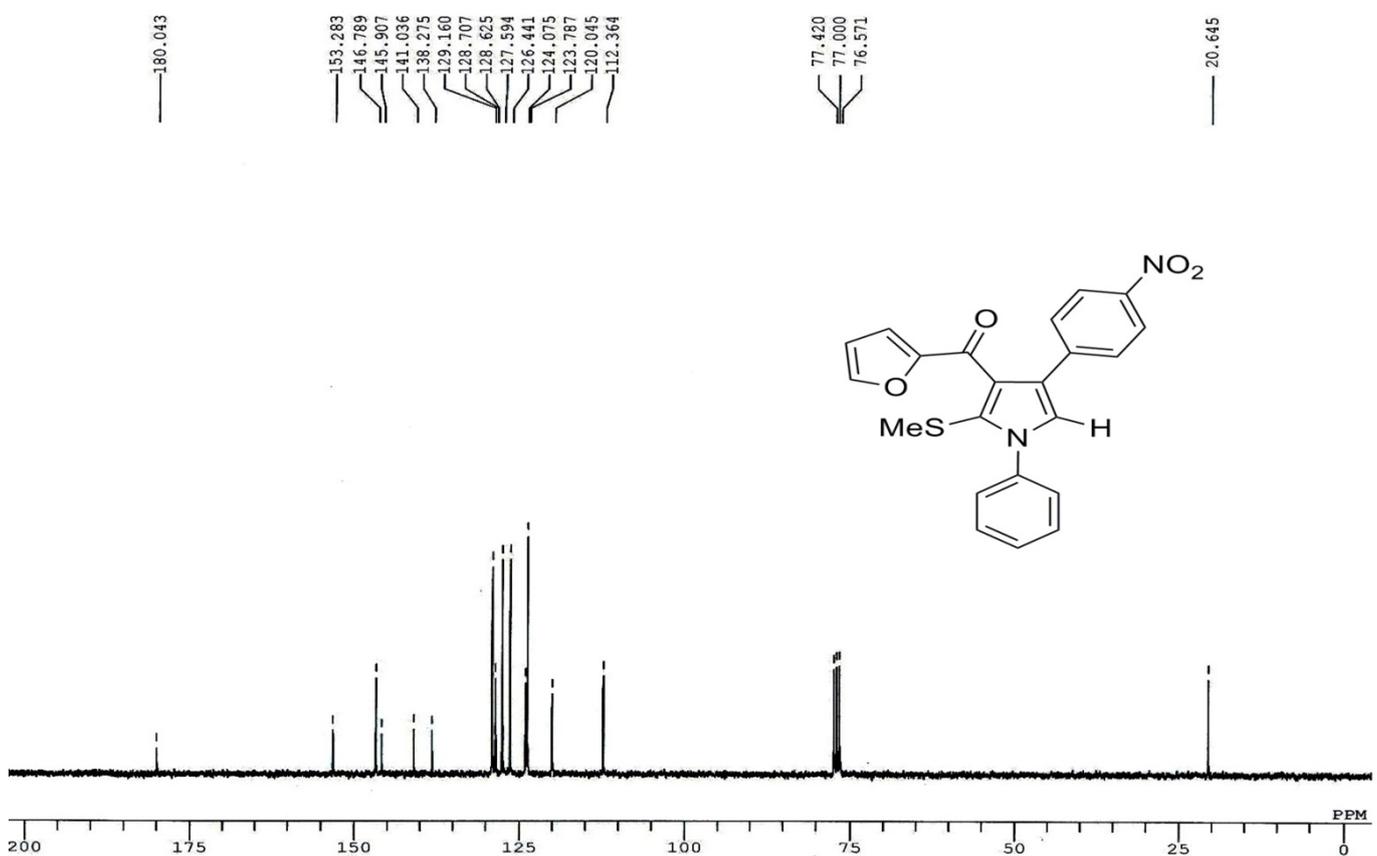
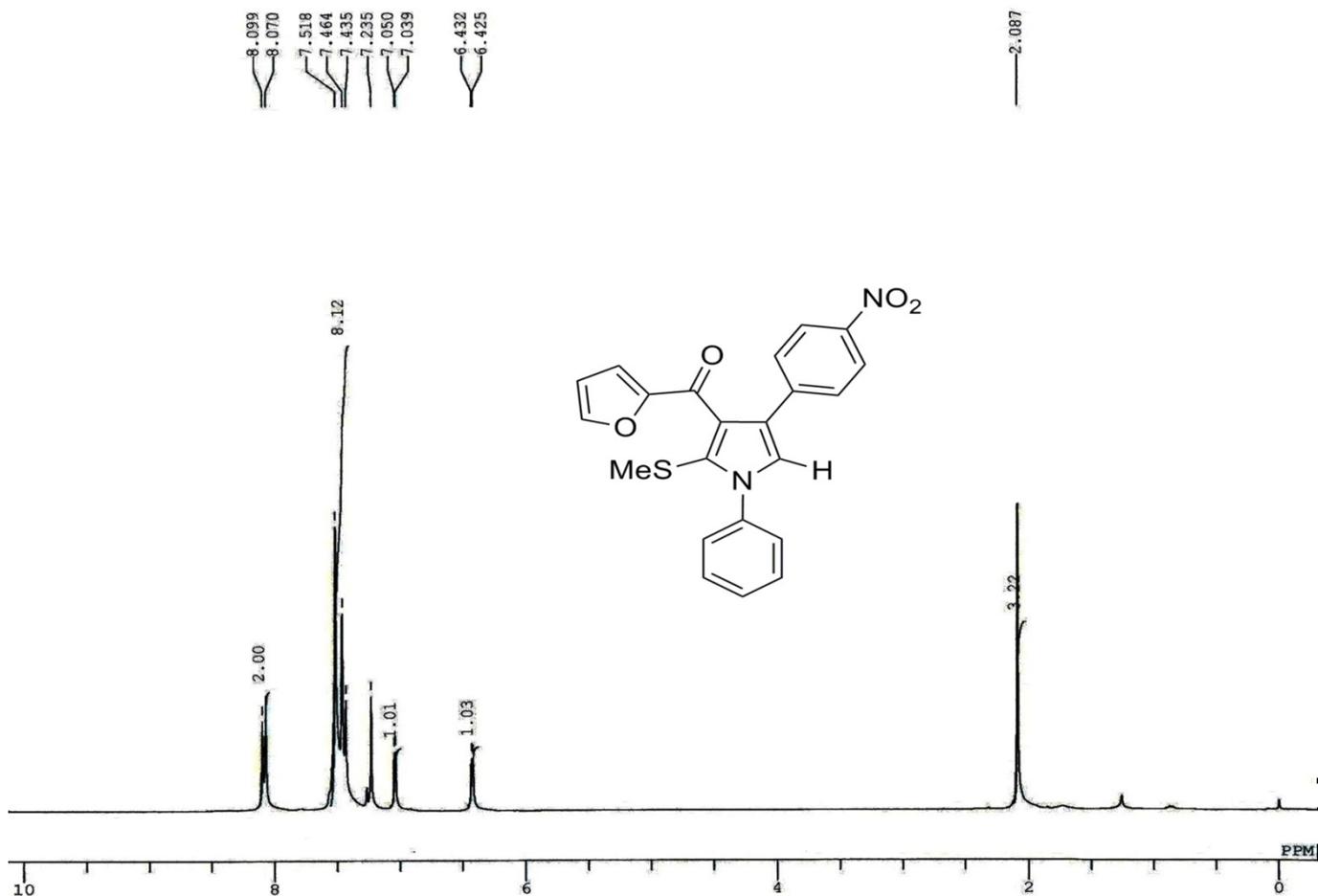
Acq. Data Name: keshav130517-5
Creation Parameters: Average(MS[1] Time:0.25..0.37)
Comment: Keshav RK-AB-11

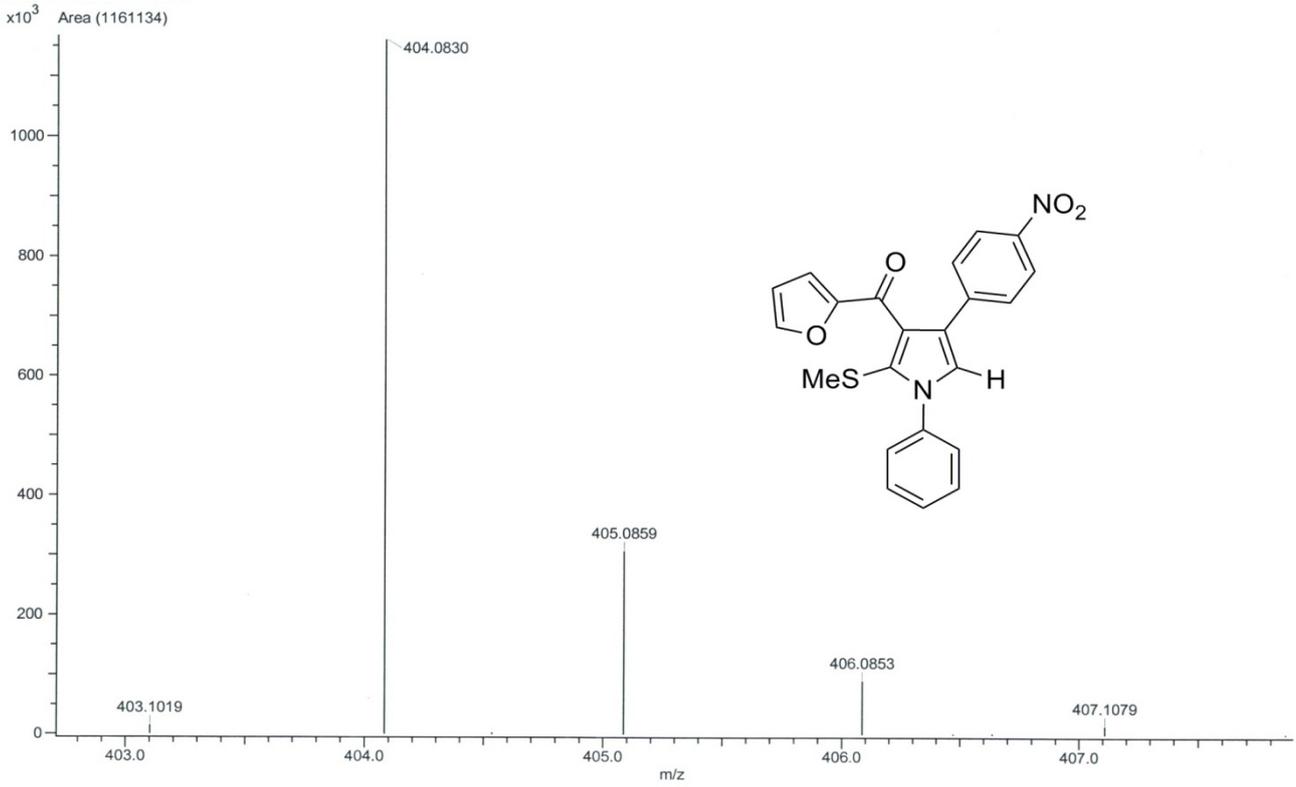
Experiment Date/Time: 5/17/2013 2:45:40 PM
Ionization Mode: EI+

$\times 10^3$ Area (127559)

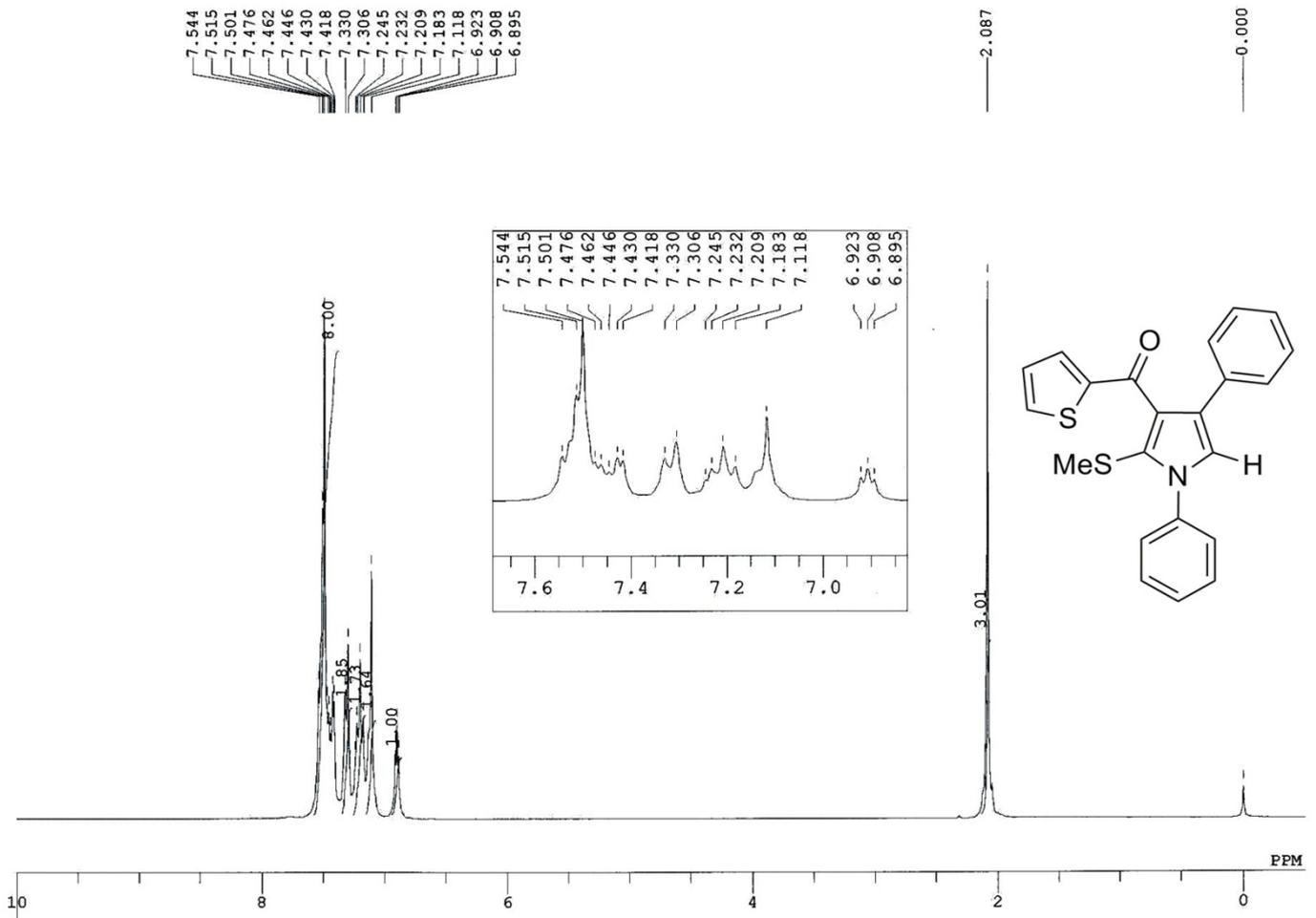


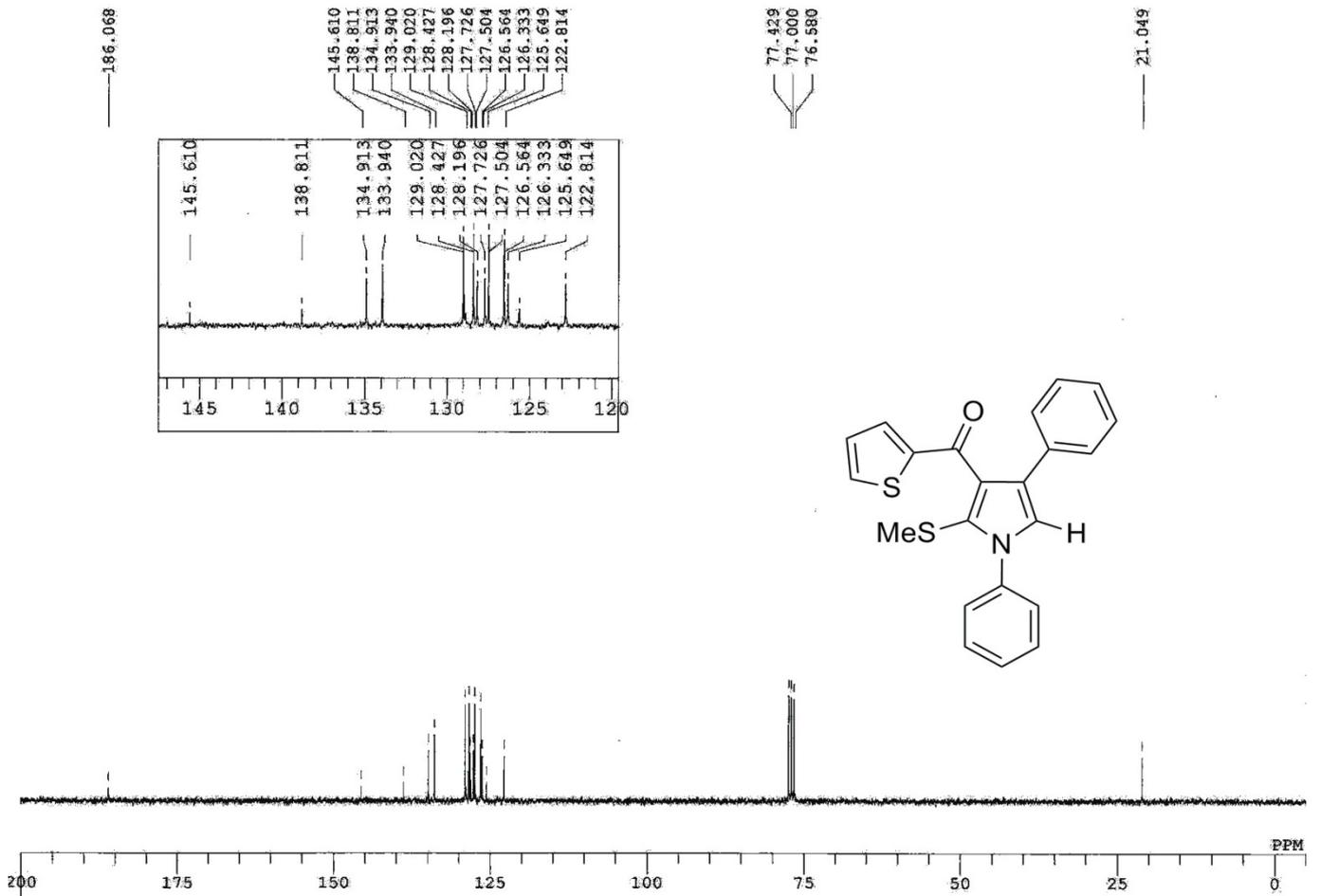
⑩ 3fd.3-(2-Furoyl)-2-(methylthio)-4-(4-nitrophenyl)-1-phenylpyrrole





⑰ 3ga. 1,4-Diphenyl-2-(methylthio)-3-(2-thienoyl)pyrrole

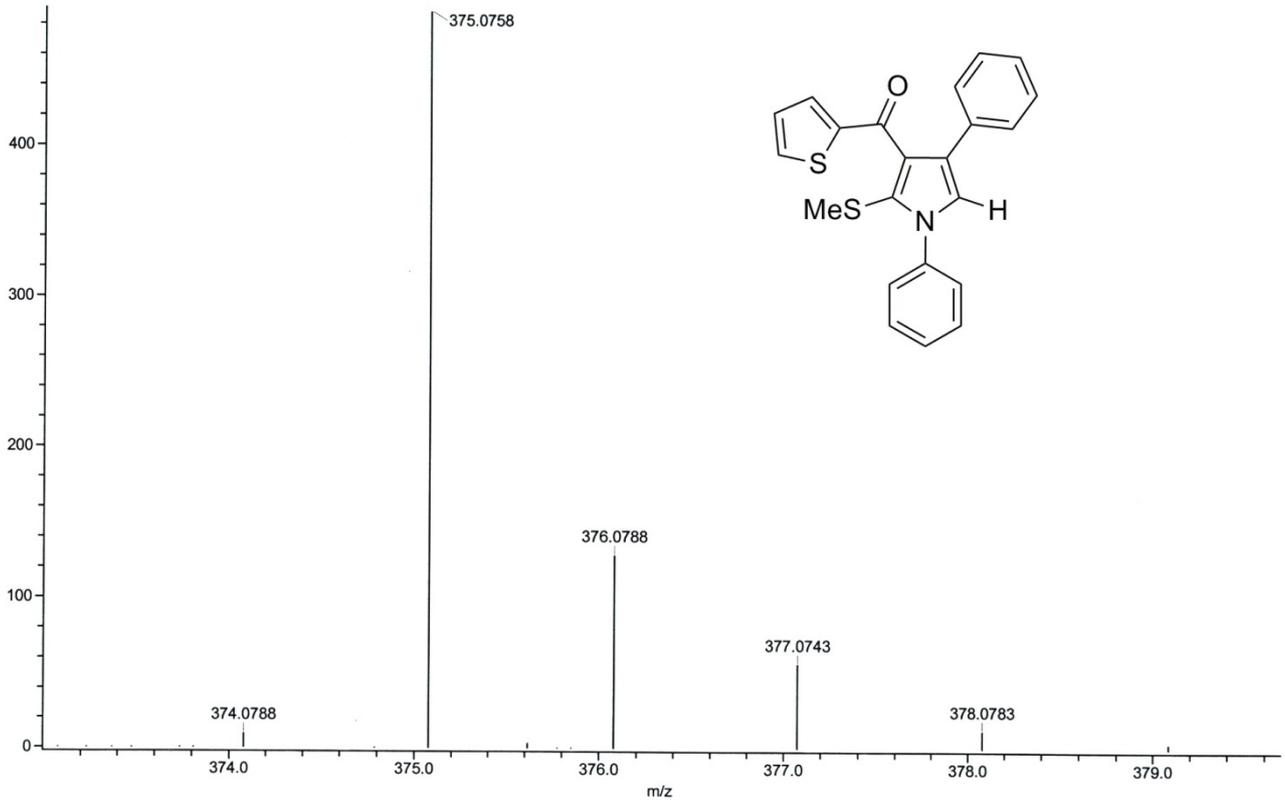




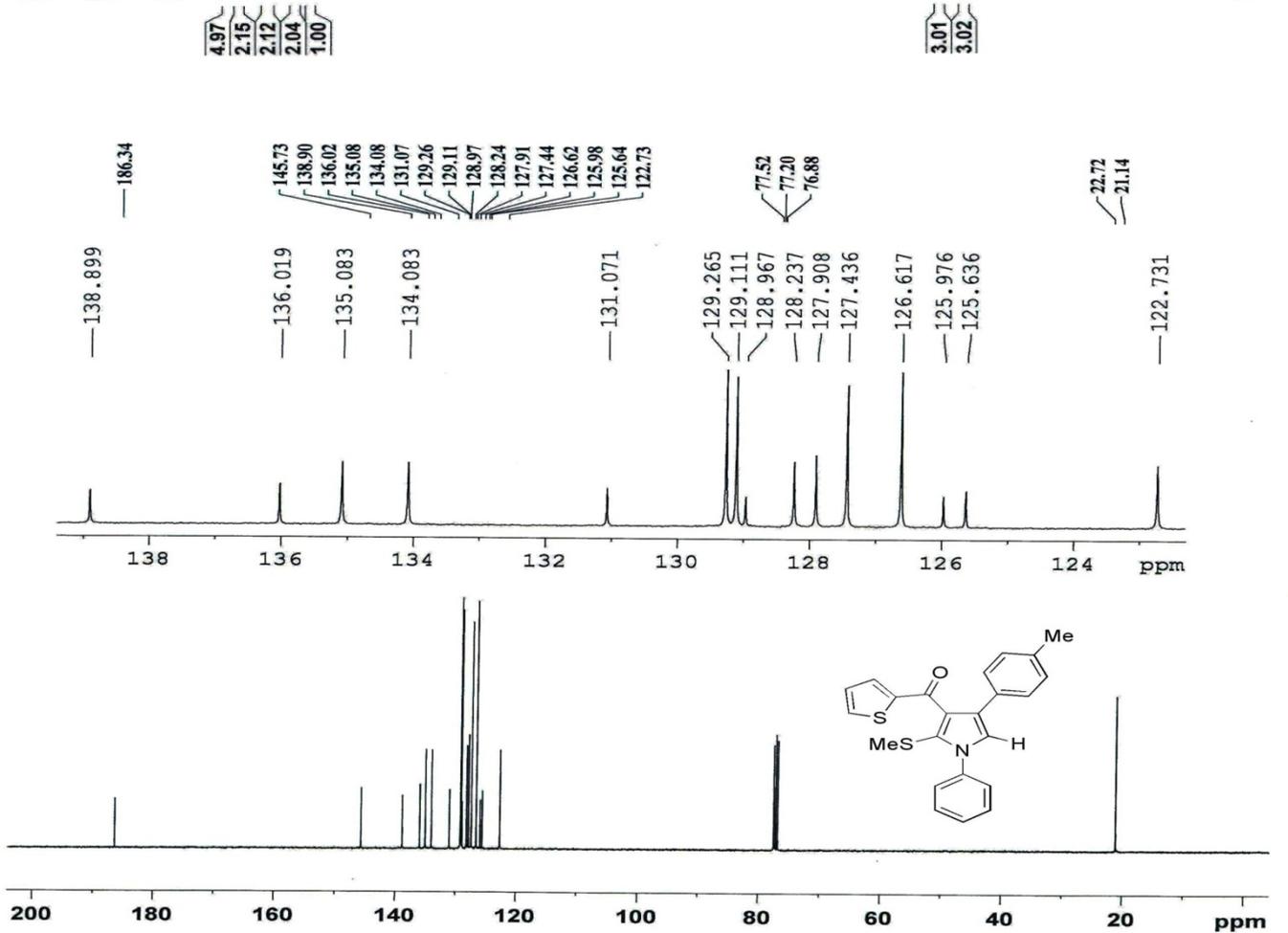
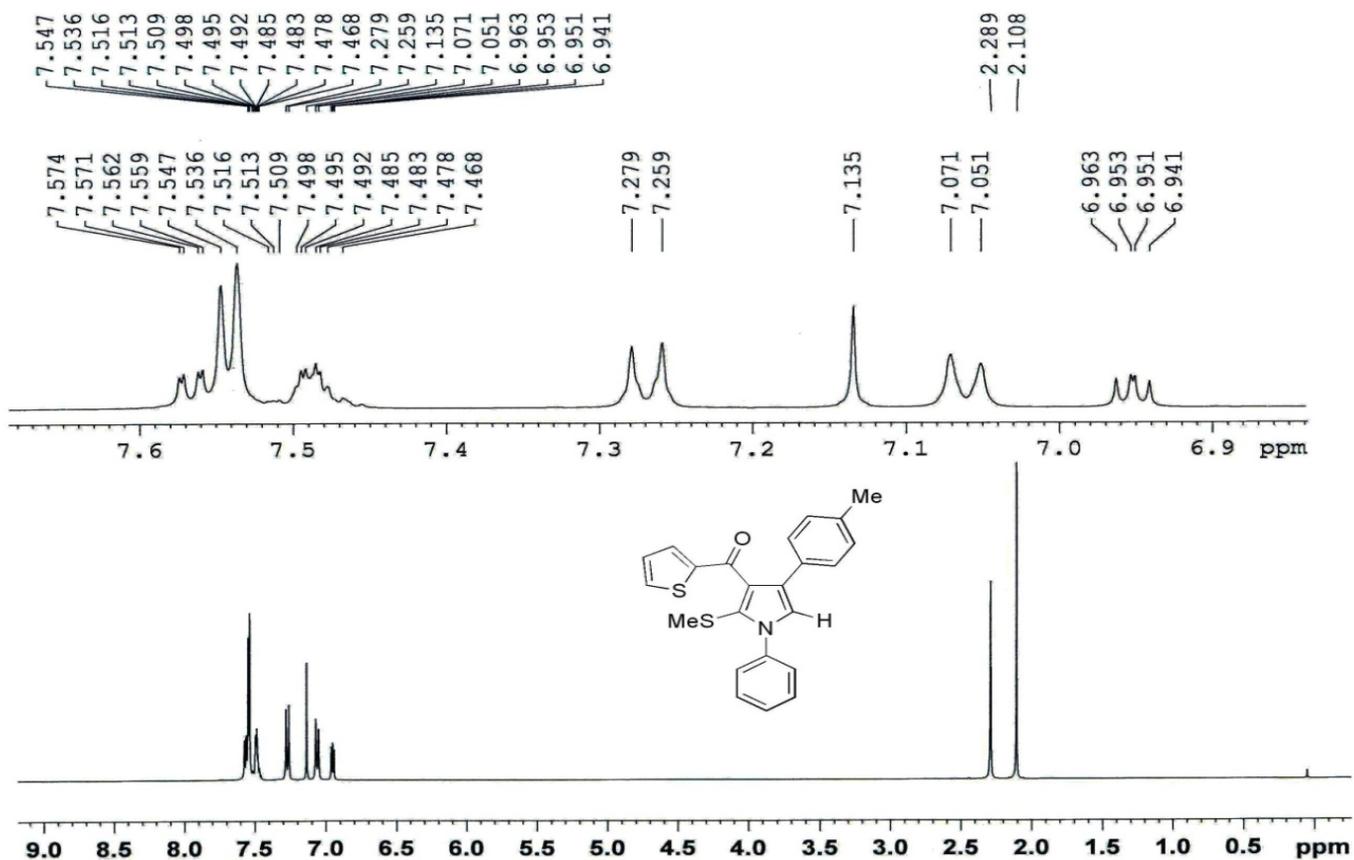
Acq. Data Name: keshav130510-3
 Creation Parameters: Average(MS[1] Time:1.18..1.28)
 Comment: Keshav RK-AB-4

Experiment Date/Time: 5/10/2013 11:51:26 AM
 Ionization Mode: EI+

$\times 10^3$ Area (488220)

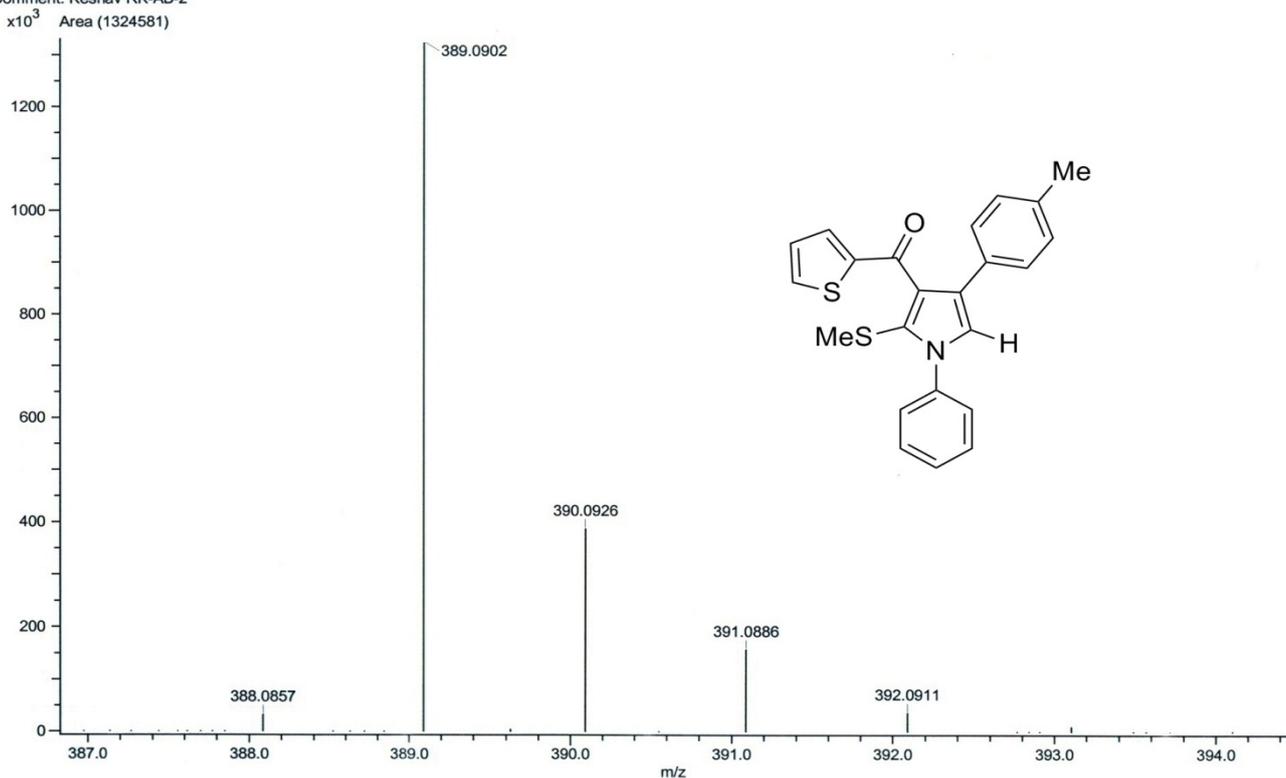


⑱ **3gb.** 2-(Methylthio)-1-phenyl-3-(2-thienoyl)-4-(*p*-tolyl)pyrrole



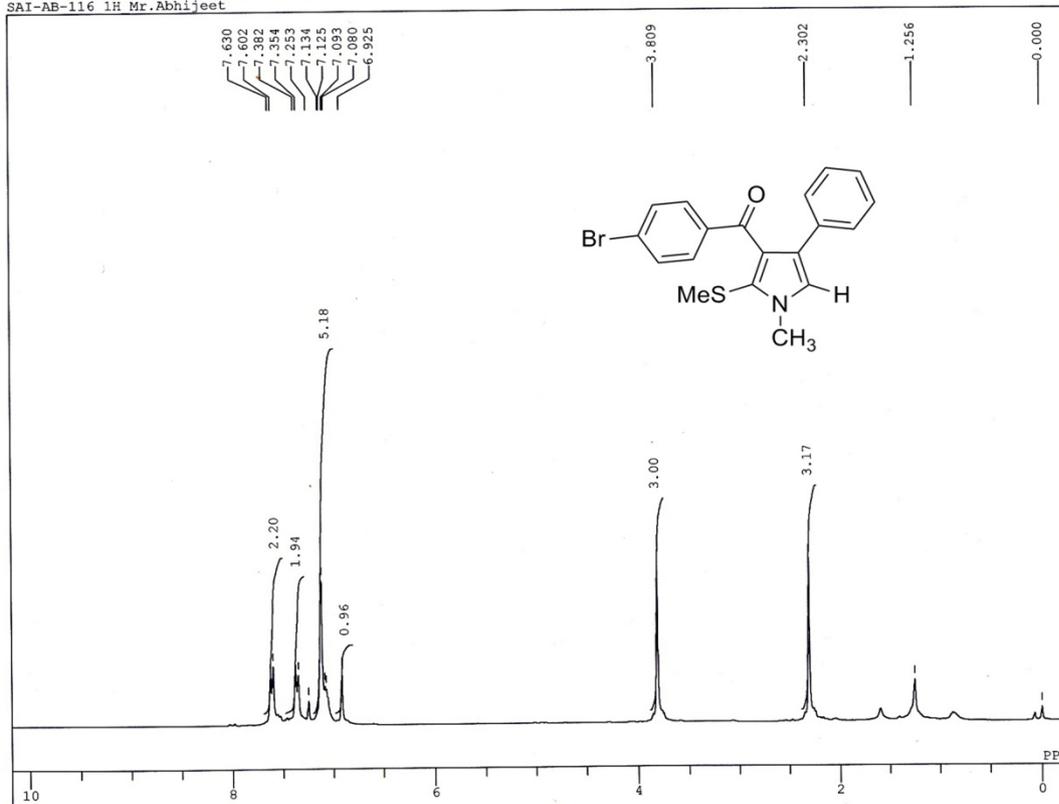
Acq. Data Name: keshav130510-1
 Creation Parameters: Average(MS[1] Time:0.13..0.25)
 Comment: Keshav RK-AB-2

Experiment Date/Time: 5/10/2013 11:33:22 AM
 Ionization Mode: EI+



① 3ha.3-(4-Bromobenzoyl)-4-phenyl-2-(methylthio)-1-methylpyrrole

C:\WINNMR98\Data\SAI-AB-116_1H1NON_E1.als
 SAI-AB-116 1H Mr.Abhijeet

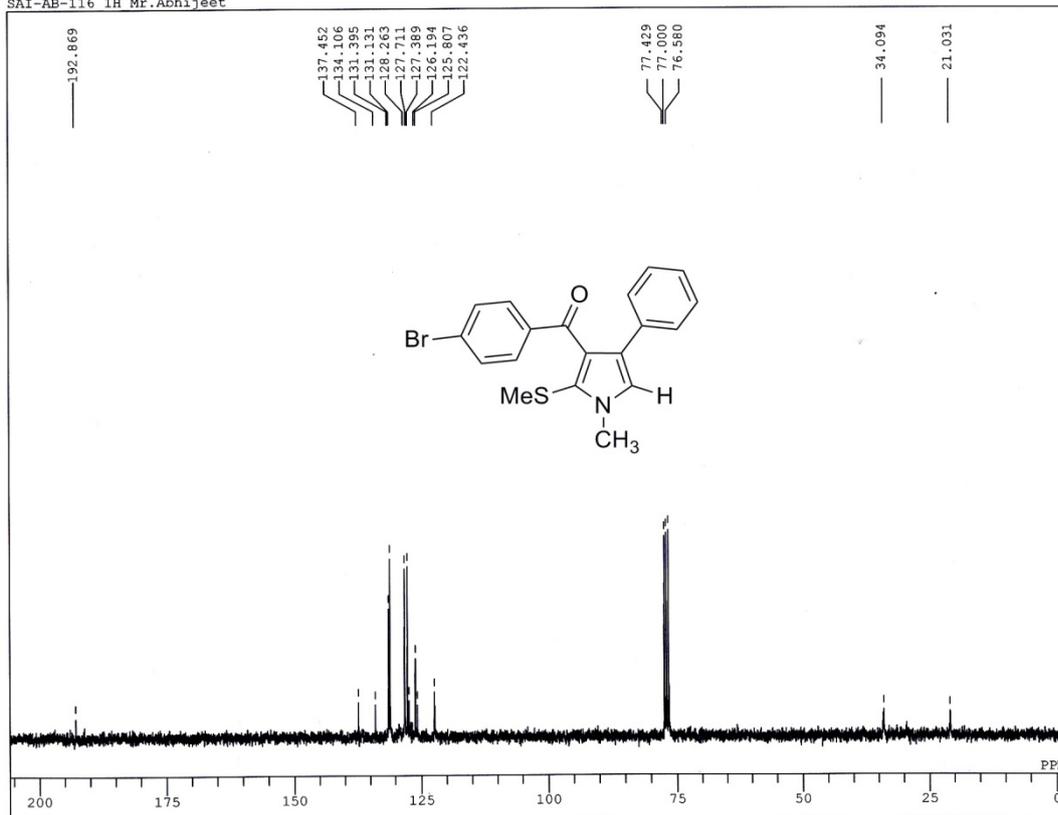


JEOL AL300 FTNMR
 CHEMISTRY DEPARTMENT
 Banaras Hindu University
 VARANASI-221005

Operator : Nagendra Kuma
 Shishir Singh

DFILE C:\WINNMR98\Data\
 COMNT SAI-AB-116_1H_Mr..
 DATIM Tue Feb 11 12:37:
 OBNUC 1H
 EXMOD NON
 OBFREQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.0 Hz
 POINT 32768
 FREQU 6016.8 Hz
 SCANS 16
 ACQTM 5.446 sec
 PD 1.547 sec
 PW1 5.6 us
 IRNUC 1H
 CTEMP 21.2 c
 SLVNT CDCL₃
 EXREF 0.00 ppm
 BF 1.20 Hz
 RGAIN 17

C:\WINNMR98\Data\SAI-AB-116_1H2BCM_E1.als
SAI-AB-116 1H Mr.Abhijeet



JEOL AL300 FTNMR
CHEMISTRY DEPARTMENT
Banaras Hindu University
VARANASI-221005

Operator : Nagendra Kuma
Shishir Singh

DFILE C:\WINNMR98\Data\
COMNT SAI-AB-116 1H Mr..
DATIM Tue Feb 11 12:53:
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 300
ACQTM 1.606 sec
PD 1.394 sec
FW1 5.9 us
IRNUC 1H
CTEMP 22.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 22

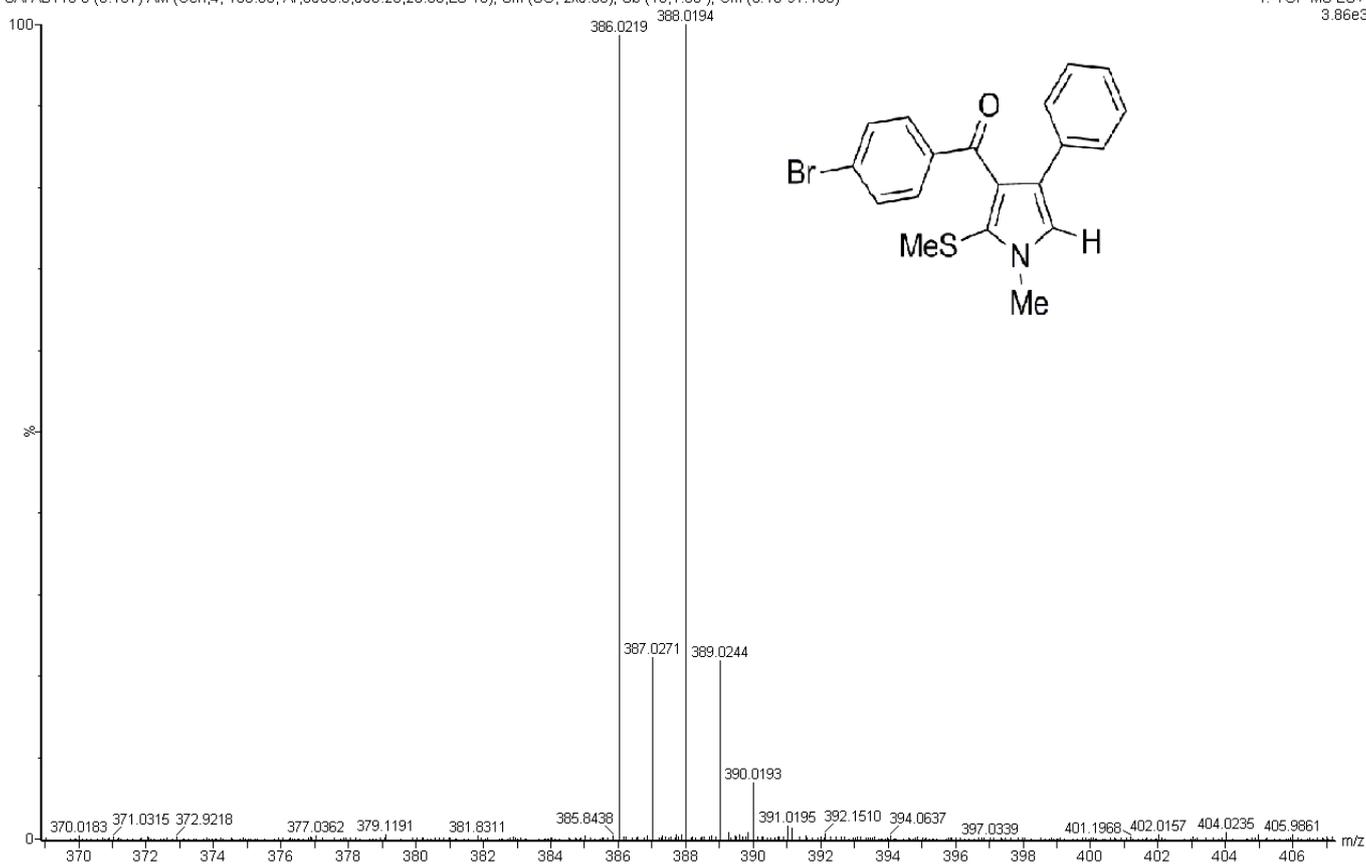
Electrospray ionisation-MS

WATERS-Q-ToF Premier-HAB213

14:41:5321-Feb-2014

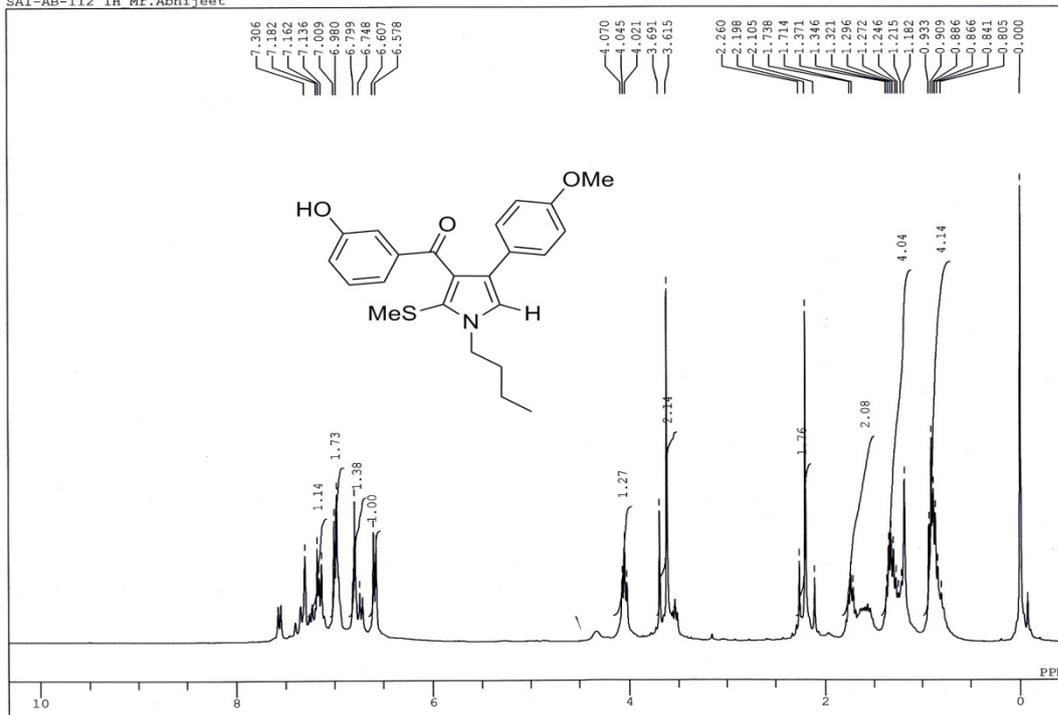
SAI AB116 8 (0.167) AM (Cen,4, 100.00, Ar,8500.0,556.28,20.00,LS 10); Sm (SG, 2x5.00); Sb (10,1.00); Cm (8:10-97:108)

1: TOF MS ES+
3.86e3



20 3ic.3-(3-Hydroxybenzoyl)-4-(4-methoxyphenyl)-2-(methylthio)-1-(n-butyl)pyrrole

C:\WINNMR98\COMMON_DEFAULT.ALS
SAI-AB-112 1H Mr.Abhijeet

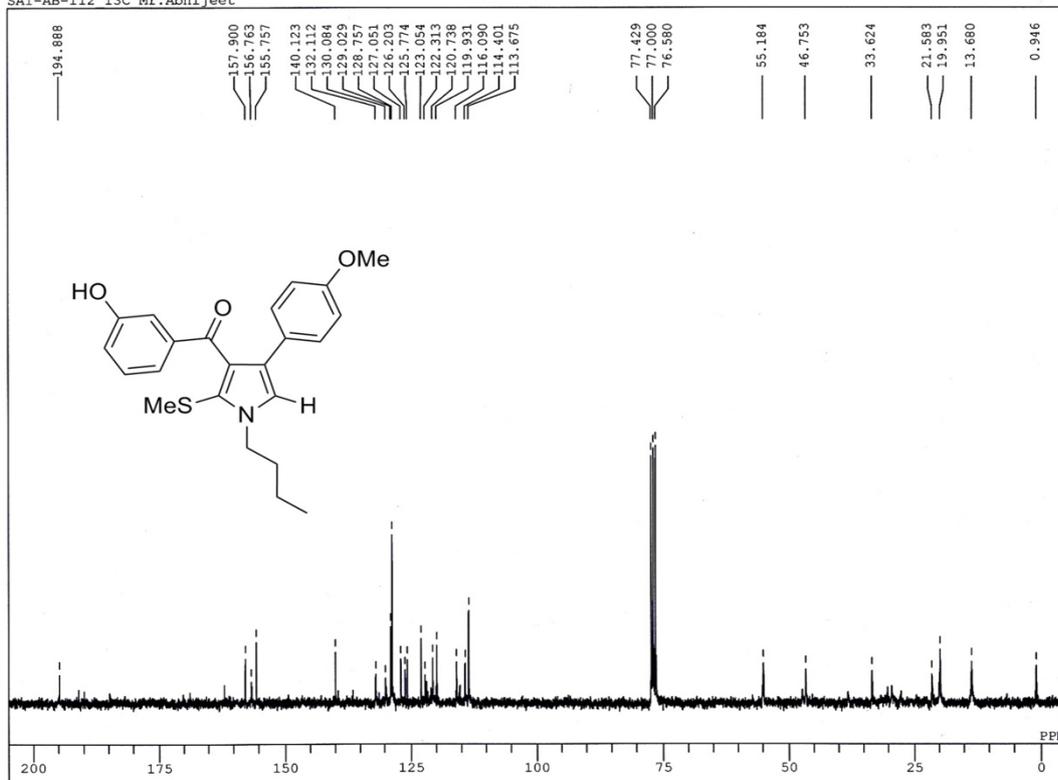


JEOL AL300 FTNMR
CHEMISTRY DEPARTMENT
Banaras Hindu University
VARANASI-221005

Operator : Nagendra Kuma
Shishir Singh

DFILE C:\WINNMR98\COMMON\
COMNT SAI-AB-112 1H Mr..
DATIM Mon Jan 27 13:03:
OBNUC 1H
EXMOD NON
OBFREQ 300.40 MHz
OBSETE 130.00 KHz
OBFIN 1150.0 Hz
POINT 32768
FREQU 9505.7 Hz
SCANS 34
ACQTM 3.447 sec
PD 1.547 sec
EW1 5.2 us
IRNUC 1H
CTEMP CDCL3 21.0 c
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 14

C:\WINNMR98\COMMON_DEFAULT.ALS
SAI-AB-112 13C Mr.Abhijeet



JEOL AL300 FTNMR
CHEMISTRY DEPARTMENT
Banaras Hindu University
VARANASI-221005

Operator : Nagendra Kuma
Shishir Singh

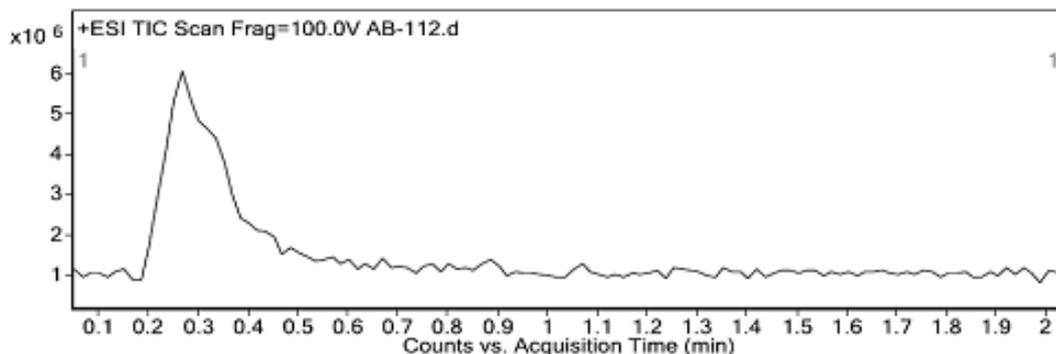
DFILE C:\WINNMR98\COMMON\
COMNT SAI-AB-112 13C Mr..
DATIM Mon Jan 27 12:58:
OBNUC 13C
EXMOD BCM
OBFREQ 75.45 MHz
OBSETE 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 604
ACQTM 1.606 sec
PD 1.394 sec
EW1 5.9 us
IRNUC 1H
CTEMP CDCL3 21.2 c
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 23

Qualitative Analysis Report

Data Filename	AB-112.d	Sample Name	AB-112
Sample Type	Sample	Position	Vial 52
Instrument Name	Instrument 1	User Name	
Acq Method	Direct Mass.m	Acquired Time	2/10/2014 7:32:56 PM
IRM Calibration Status	Success	DA Method	Regular.m
Comment			
Sample Group		Info.	
Acquisition SW	6200 series TOF/6500 series		
Version	Q-TOF B.05.00 (B5042.0)		

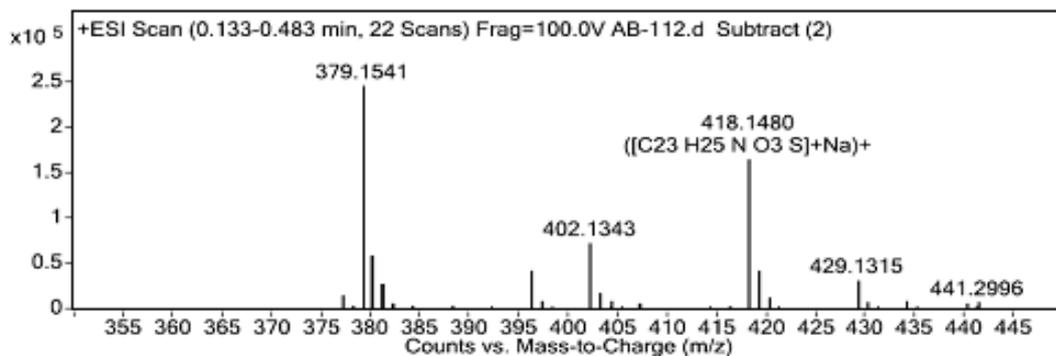
User Chromatograms

Fragmentor Voltage 100 Collision Energy 0 Ionization Mode ESI



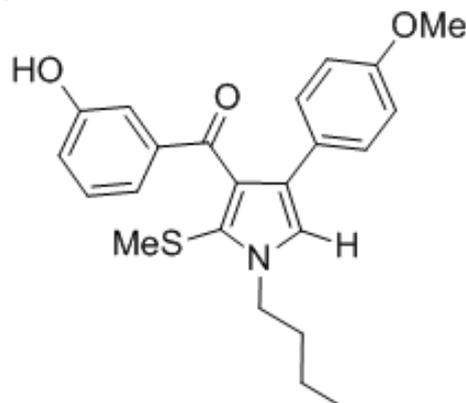
User Spectra

Fragmentor Voltage 100 Collision Energy 0 Ionization Mode ESI



Peak List

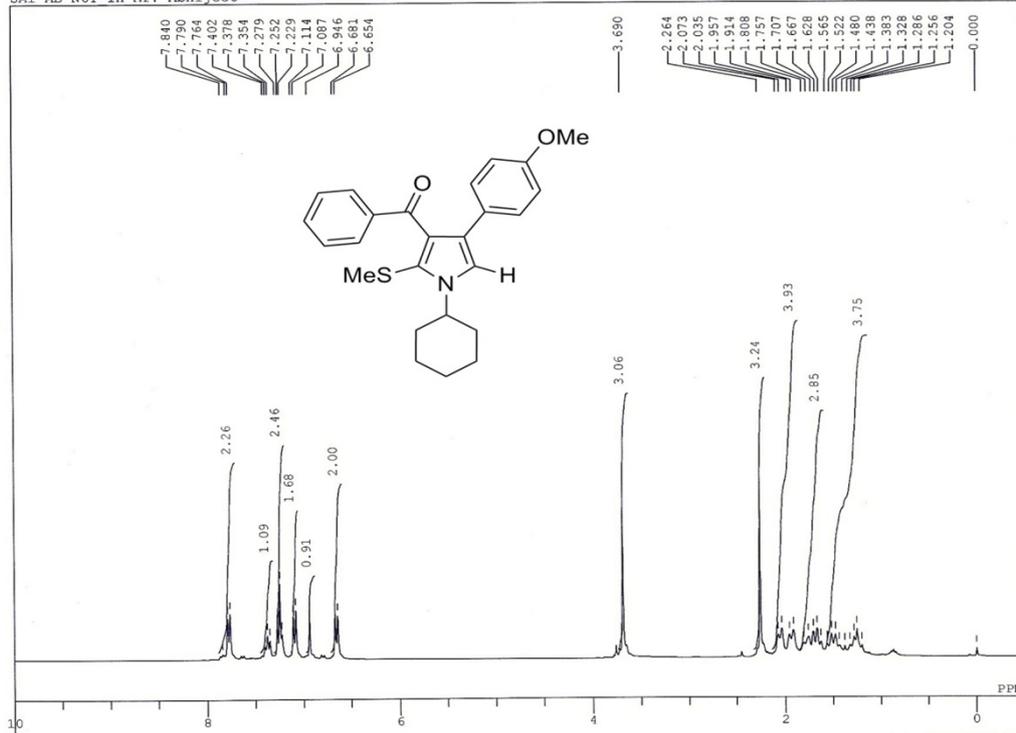
m/z	z	Abund	Formula	Ion
379.1541	1	246315.36		
380.1566	1	59263.1		
396.1658	1	42546.48	C23 H25 N O3 S	(M+H)+
402.1343	1	72458.56		
418.148	1	165348.09	C23 H25 N O3 S	(M+Na)+
419.1511	1	42244.34	C23 H25 N O3 S	(M+Na)+
797.2931	1	114281.48		
798.296	1	56799.66		
813.3069	1	150981		
814.3099	1	78406.17		



--- End Of Report ---

213jc. 3-Benzoyl-4-(4-methoxyphenyl)-2-(methylthio)-1-cyclohexylpyrrole

C:\WINNMR98\Data\Sai-AB-N6Y_1H1NON_E4.als
Sai-AB-N6Y 1H Mr. Abhijeet

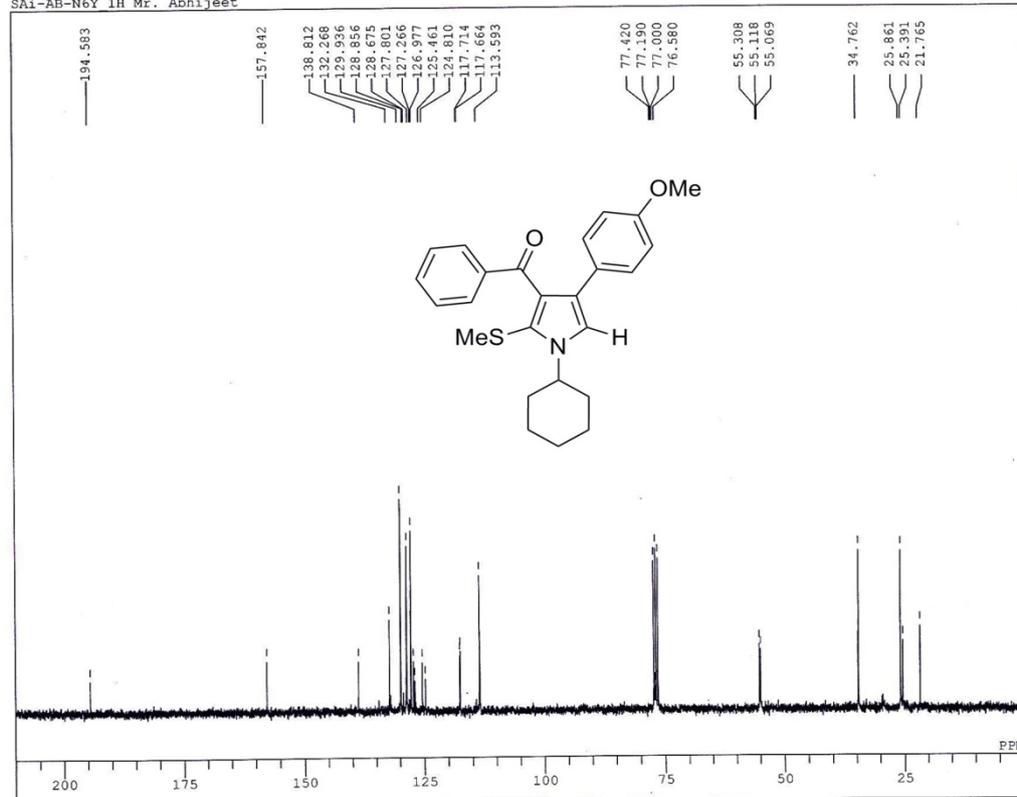


JEOL AL300 FTNMR
CHEMISTRY DEPARTMENT
Banaras Hindu University,
VARANASI-221005

Operator : Nagendra Kumar
Shishir Singh

DFILE C:\WINNMR98\Data\Sai-AB-N6Y_1H Mr. Abhijeet
COMNT Sai-AB-N6Y_1H Mr. Abhijeet
DATIM Wed Nov 27 14:11:01 2005
OBNUC 1H
EXMOD NON
OBFREQ 300.40 MHz
OBSETE 130.00 KHz
OBFIN 1150.0 Hz
POINT 32768
FREQU 6016.8 Hz
SCANS 16
AQTM 5.446 sec
PD 1.547 sec
PWI 5.6 us
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 13

C:\WINNMR98\Data\Sai-AB-N6Y_1H2BCM_E4.als
Sai-AB-N6Y 1H Mr. Abhijeet



JEOL AL300 FTNMR
CHEMISTRY DEPARTMENT
Banaras Hindu University,
VARANASI-221005

Operator : Nagendra Kumar
Shishir Singh

DFILE C:\WINNMR98\Data\Sai-AB-N6Y_1H Mr. Abhijeet
COMNT Sai-AB-N6Y_1H Mr. Abhijeet
DATIM Wed Nov 27 14:21:24 2005
OBNUC 13C
EXMOD BCM
OBFREQ 75.45 MHz
OBSETE 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 200
AQTM 1.606 sec
PD 1.394 sec
PWI 5.9 us
IRNUC 1H
CTEMP 23.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 24

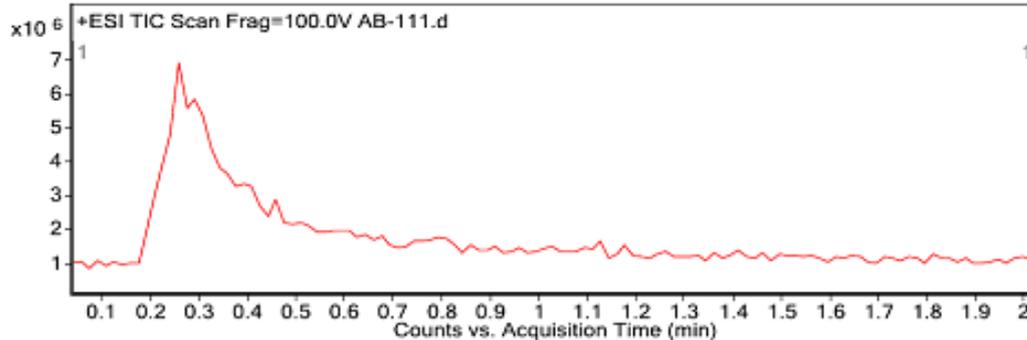
Qualitative Analysis Report

Data Filename	AB-111.d	Sample Name	AB-111
Sample Type	Sample	Position	Vial 51
Instrument Name	Instrument 1	User Name	
Acq Method	Direct Mass.m	Acquired Time	2/10/2014 7:29:29 PM
IRM Calibration Status	Success	DA Method	Regular.m
Comment			

Sample Group		Info.
Acquisition SW	6200 series TOF/6500 series	
Version	Q-TOF B.05.00 (B5042.0)	

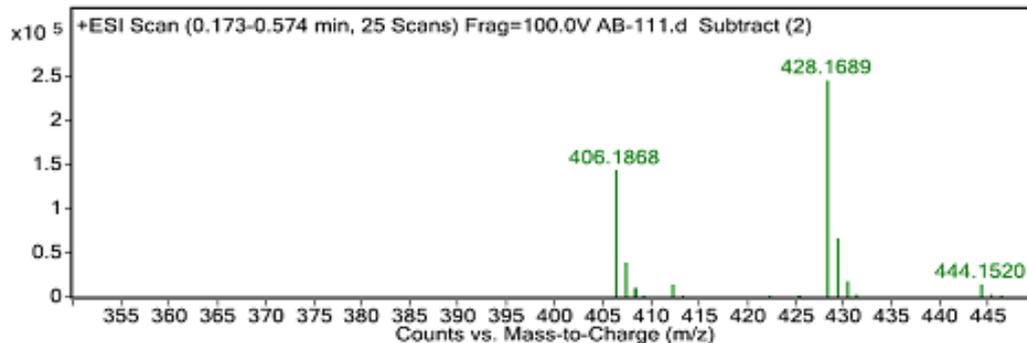
User Chromatograms

Fragmentor Voltage 100 Collision Energy 0 Ionization Mode ESI



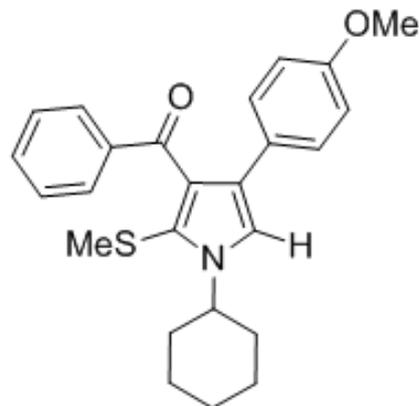
User Spectra

Fragmentor Voltage 100 Collision Energy 0 Ionization Mode ESI



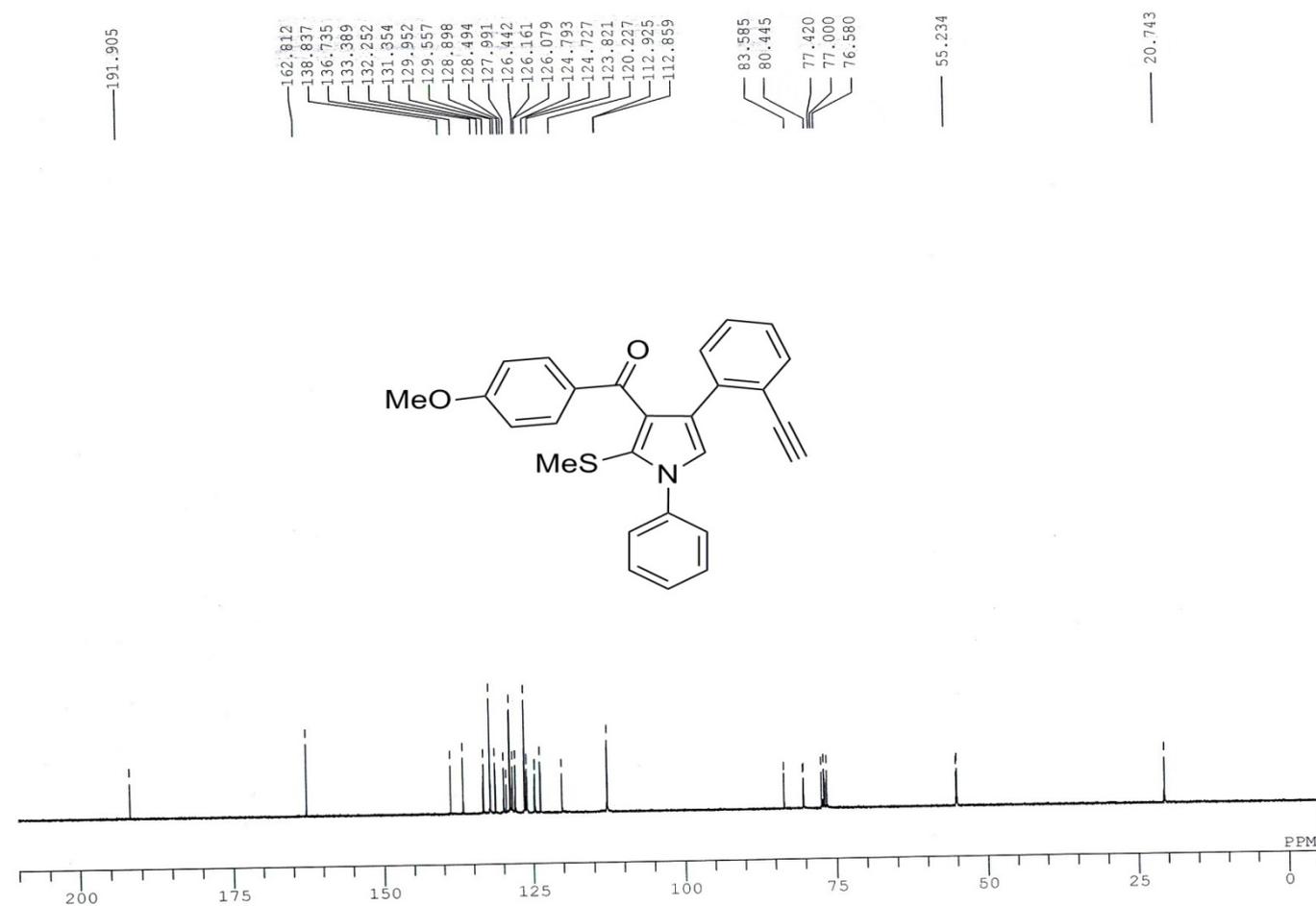
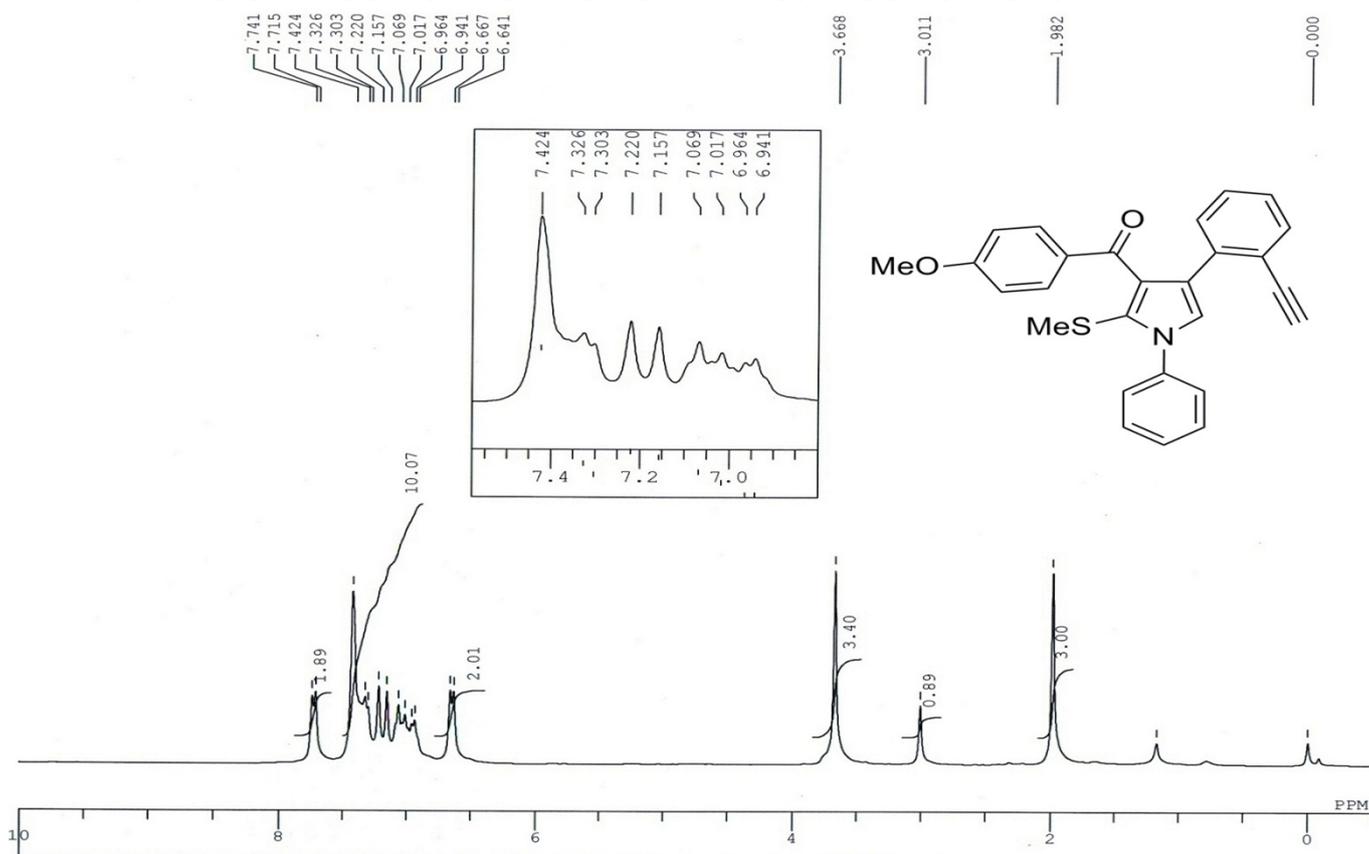
Peak List

m/z	z	Abund
406.1868	1	143456.75
407.1898	1	39430.51
428.1689	1	245924.06
429.172	1	67131.23
619.3039	1	34442.8
817.335	1	45181.71
833.3492	1	468918.88
834.352	1	264181.75
835.3513	1	115320.43
836.3519	1	37198.17



--- End Of Report ---

②3cf. 4-(2-Ethynylphenyl)-2-(methylthio)-3-(4-methoxyphenyl)-1-phenylpyrrole



Display Report

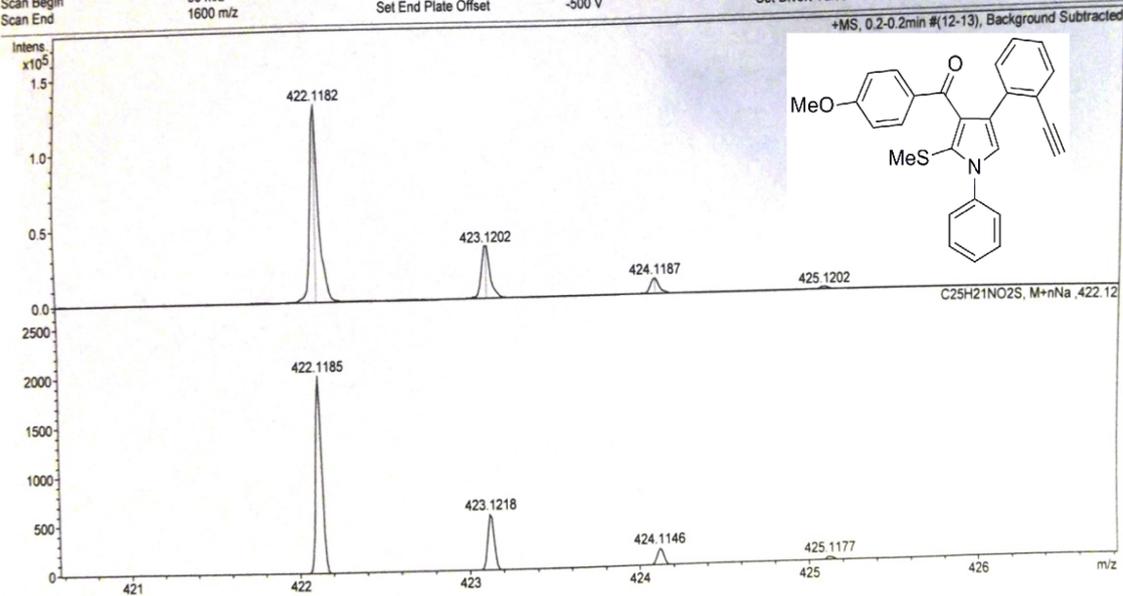
37.37 107.6 °C

Analysis Name: Data\091313\Aug\sam090813\RKABC2_low_5_01_61733.d
 Method: hystar.pl.m
 Sample Name: RKABC2_low
 Comment:

Acquisition Date: 09.08.2013 09:38:01
 Operator: BDAL@DE
 Instrument / Ser#: microTOF 10237

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.6 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	8.0 l/min
Scan End	1600 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Source

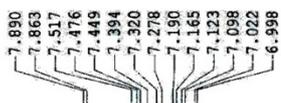


Bruker Compass DataAnalysis 4.0

printed: 09.08.2013 11:26:17

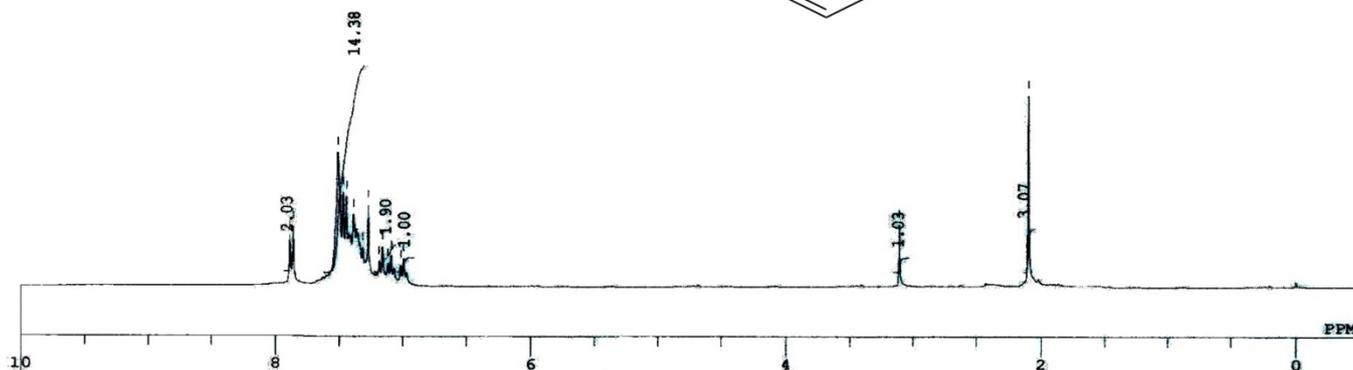
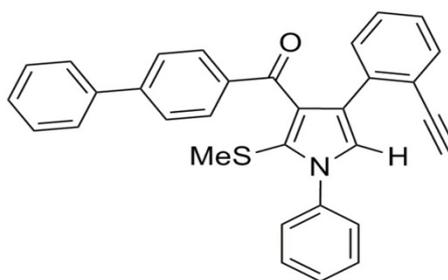
Page 1 of 1

②3ef. 4-(2-Ethynylphenyl)-2-(methylthio)-1-phenyl-3-(4-phenylbenzoyl)pyrrole



3.111

2.099

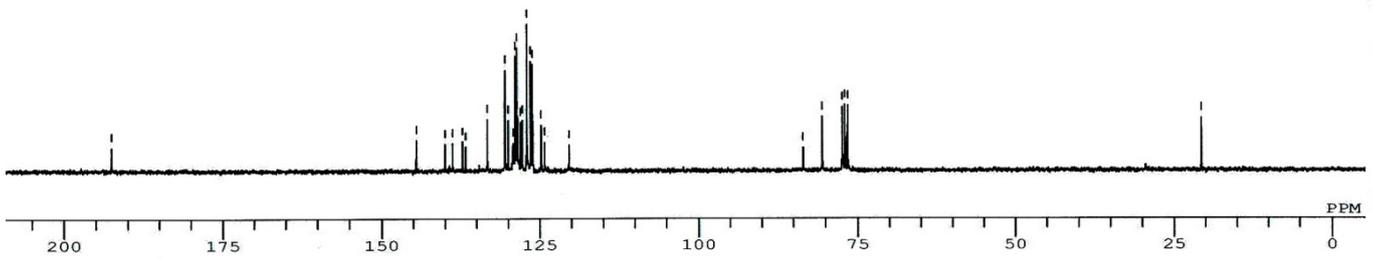
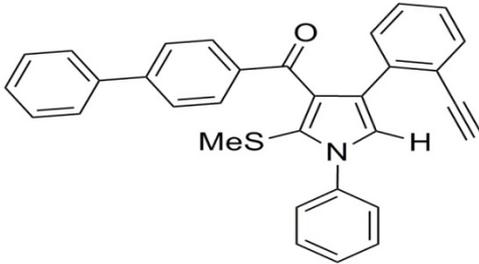


192.620

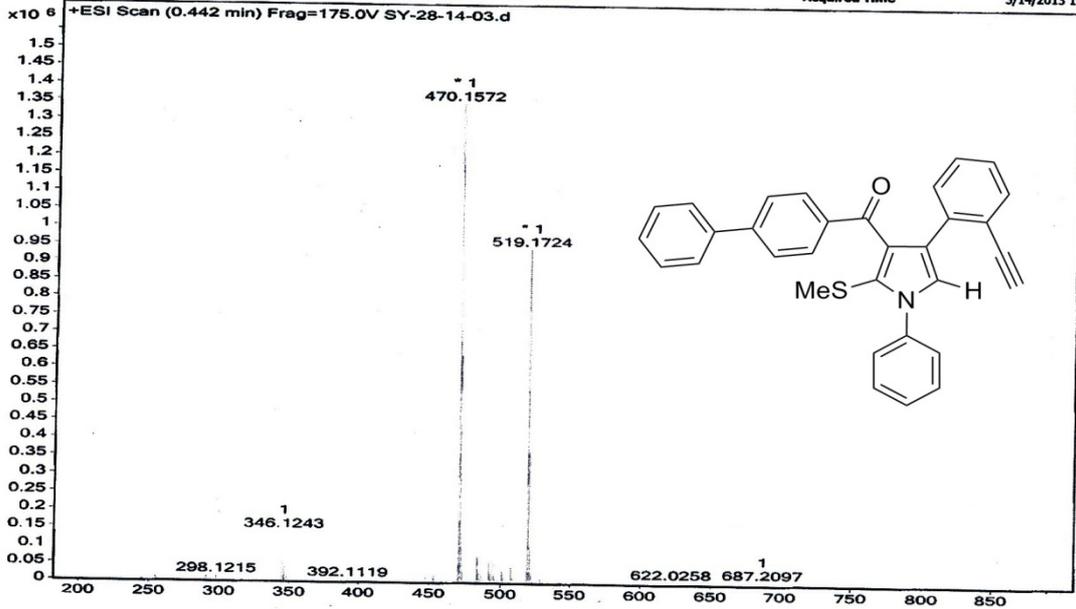
144.522
140.031
138.828
137.262
136.817
133.347
130.545
130.034
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128.954
128.880
128.690
128.509
128.097
127.817
127.083
126.482
126.218
126.169
124.792
124.240
120.466

83.535
80.552
77.429
77.000
76.580

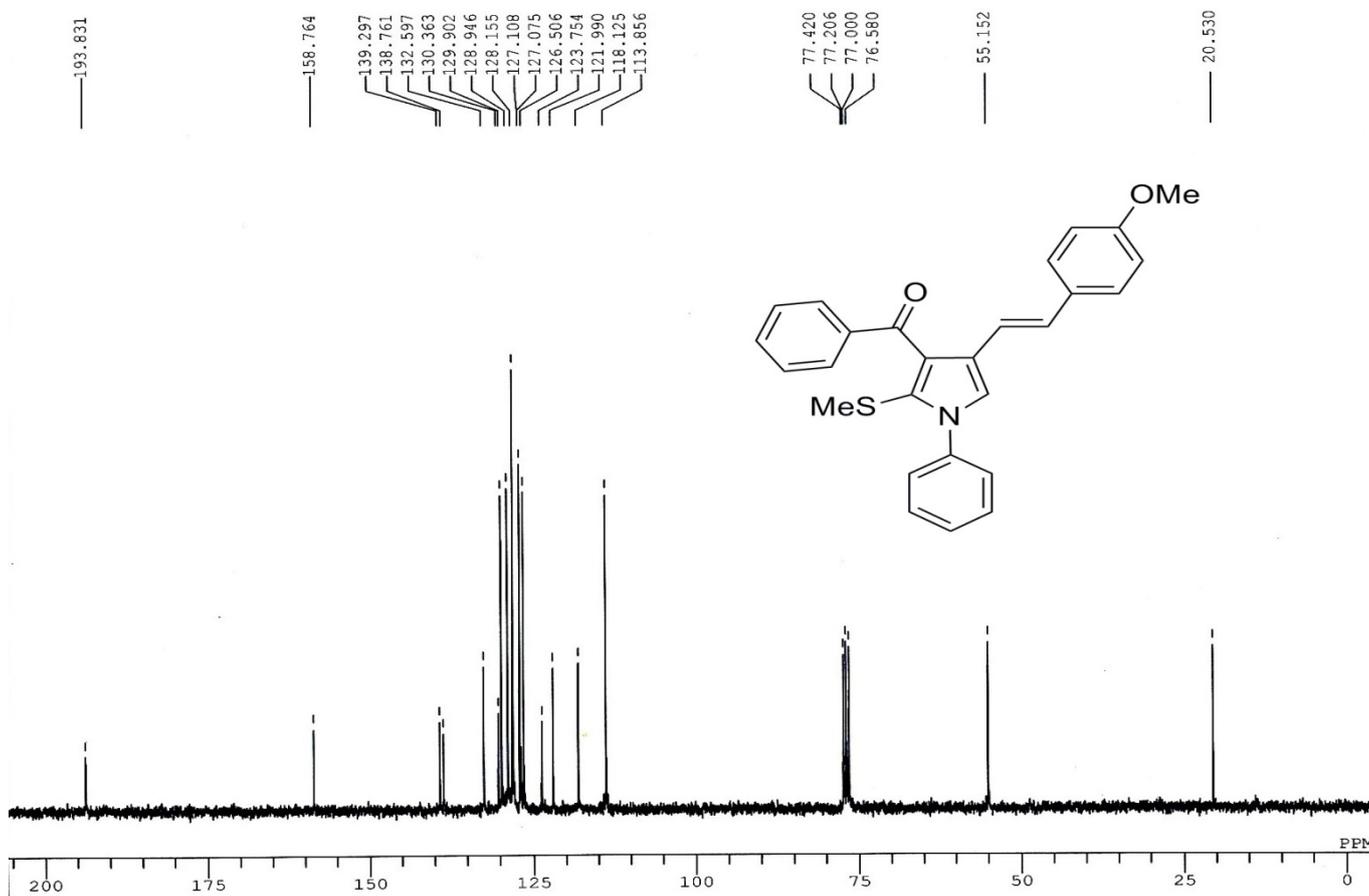
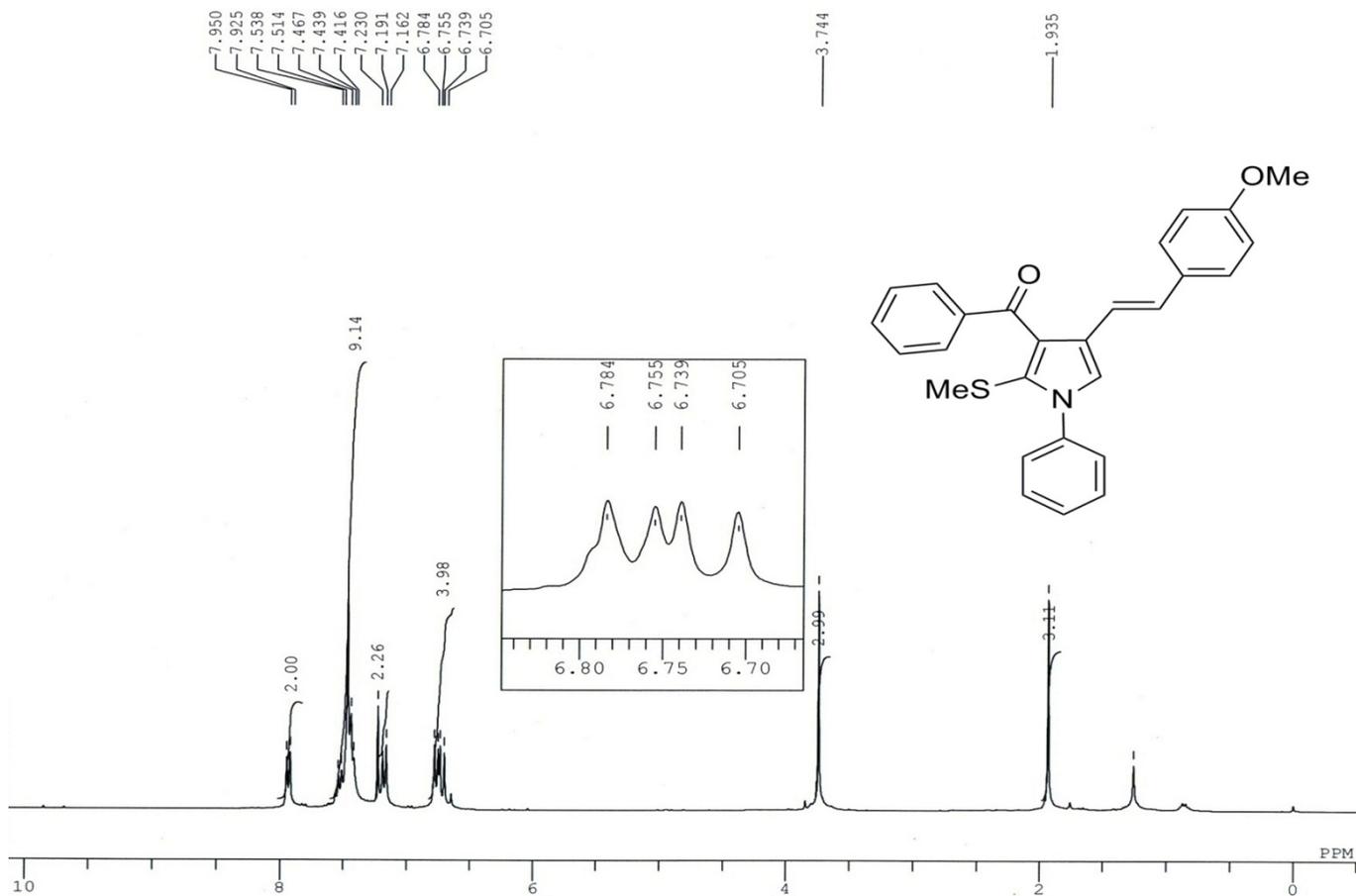
20.735

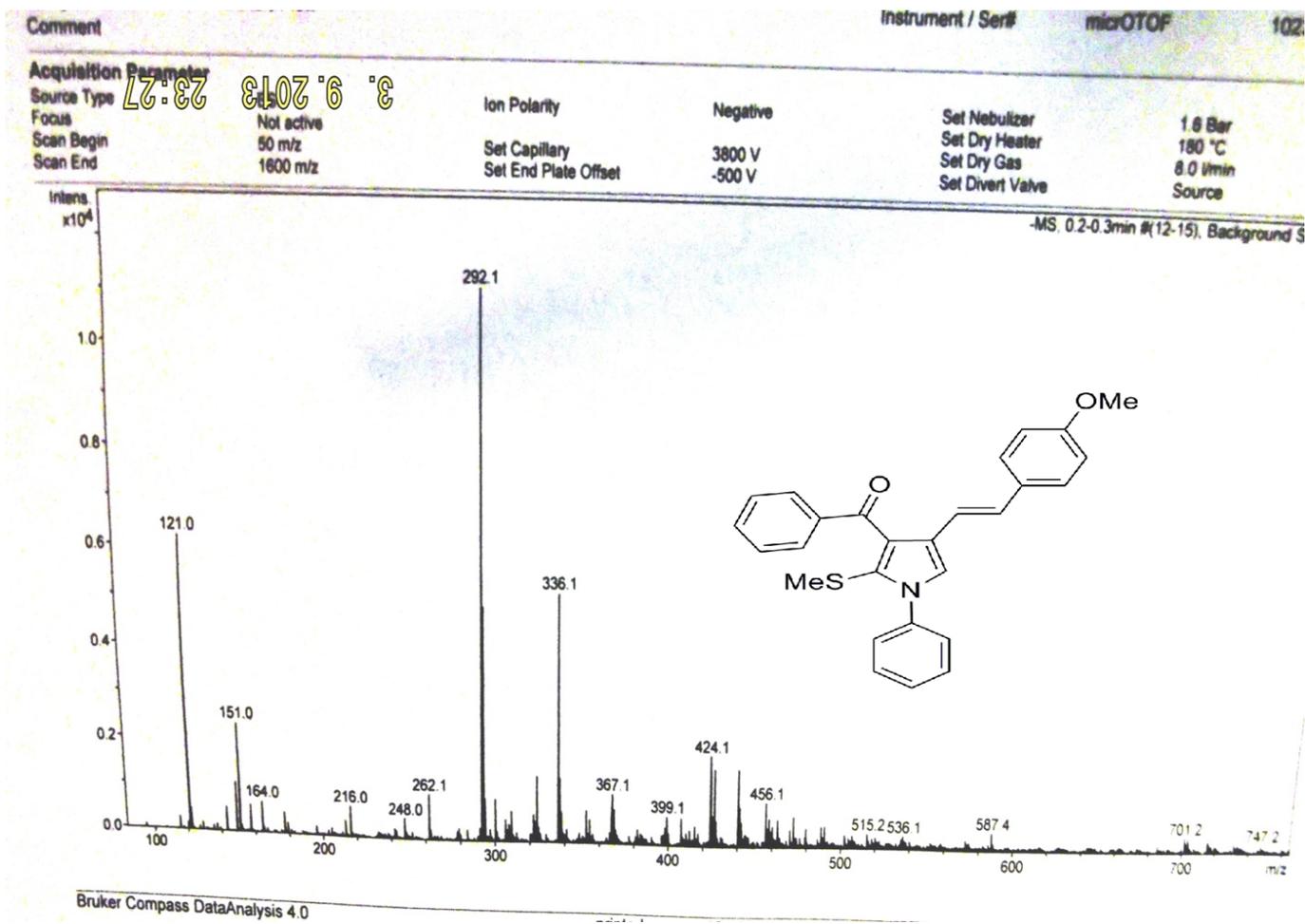


Sample Name	SY-28	Position	pid1	Instrument Name	Instrument 1	User Name	JNCASR-PC/admin
Inj Vol	0.1	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	SY-28-14-03.d	ACQ Method	demo.m	Comment		Acquired Time	3/14/2013 12:23:39 PM

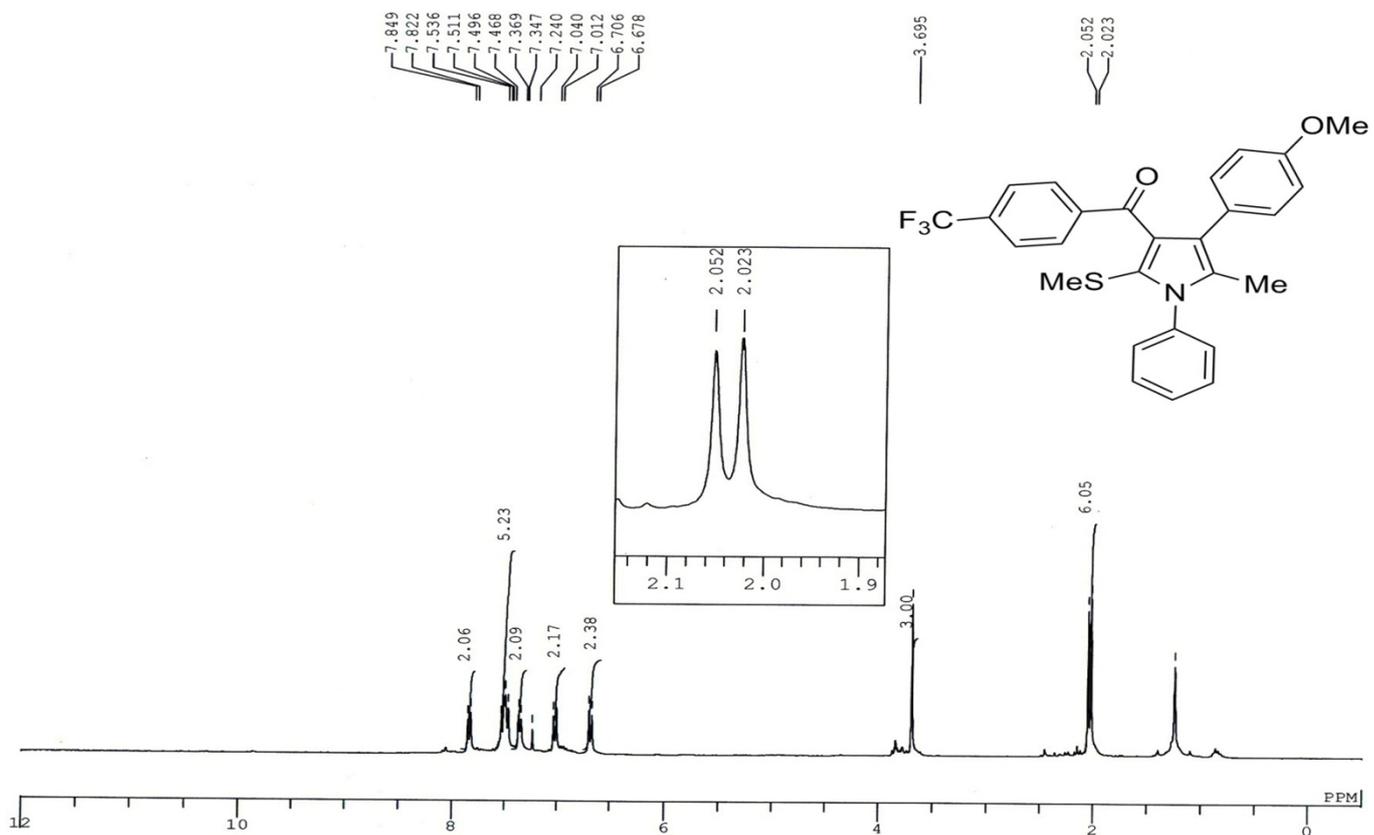


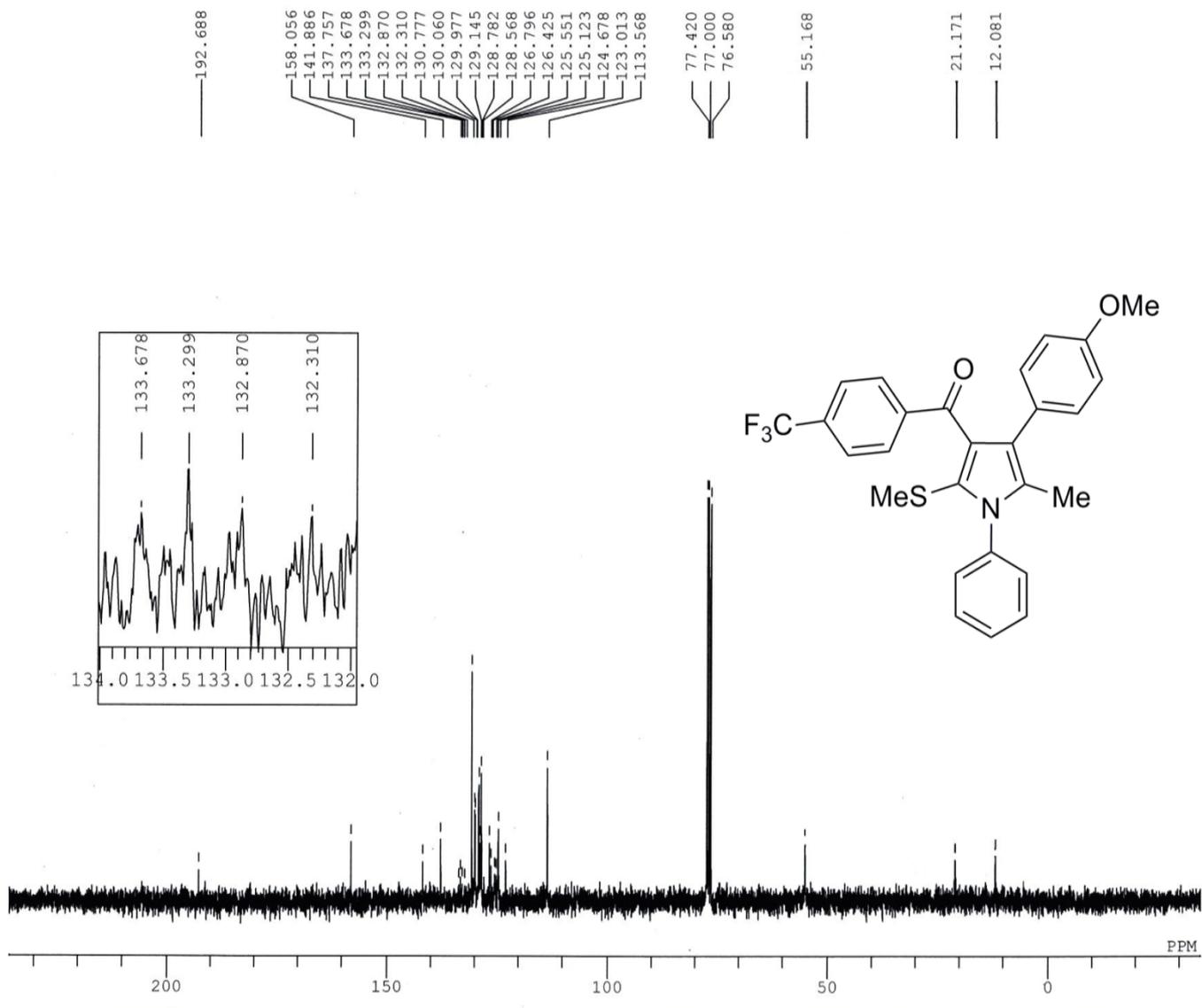
④3ag. 3-Benzoyl-4-((E)-4-methoxystyryl)-2-(methylthio)-1-phenylpyrrole





253dh. 4-(4-Methoxyphenyl)-5-methyl-2-(methylthio)-1-phenyl-3-(4-trifluoromethyl)pyrrole

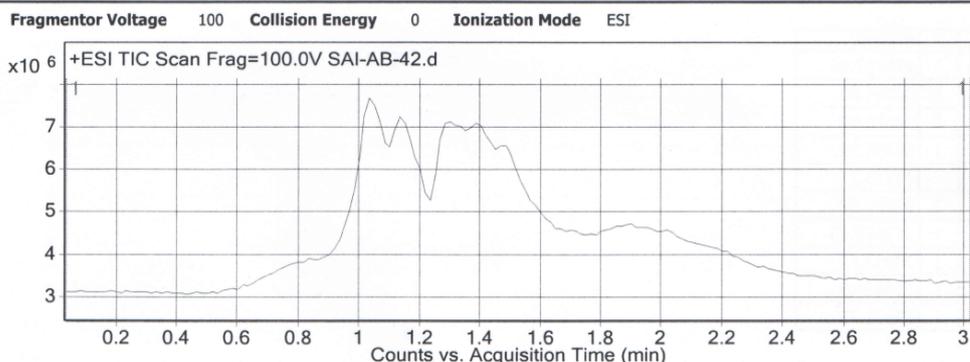




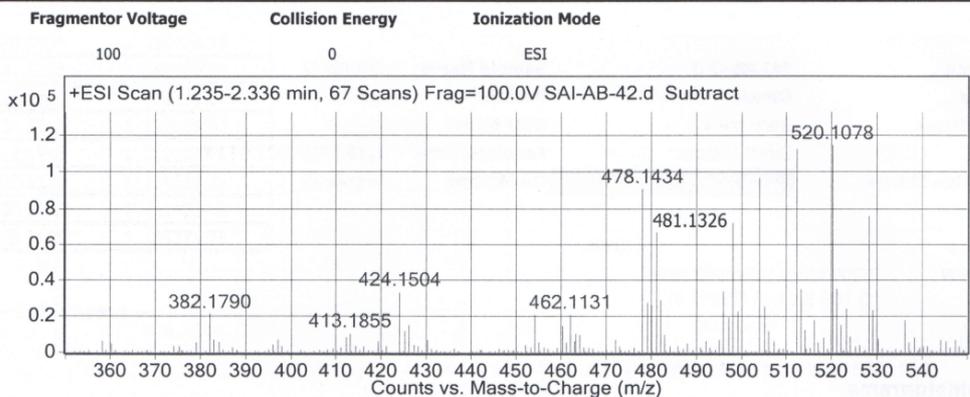
Qualitative Analysis Report

Data Filename	SAI-AB-42.d	Sample Name	SAI-AB-42
Sample Type	Sample	Position	Vial 19
Instrument Name	Instrument 1	User Name	
Acq Method	Direct Mass.m	Acquired Time	7/16/2013 1:21:44 PM
IRM Calibration Status	Success	DA Method	Regular.m
Comment			
Sample Group		Info.	
Acquisition SW	6200 series TOF/6500 series		
Version	Q-TOF B.05.00 (B5042.0)		

User Chromatograms

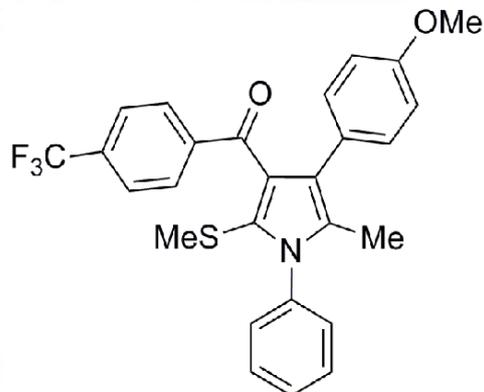


User Spectra



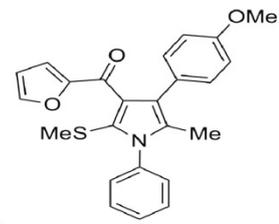
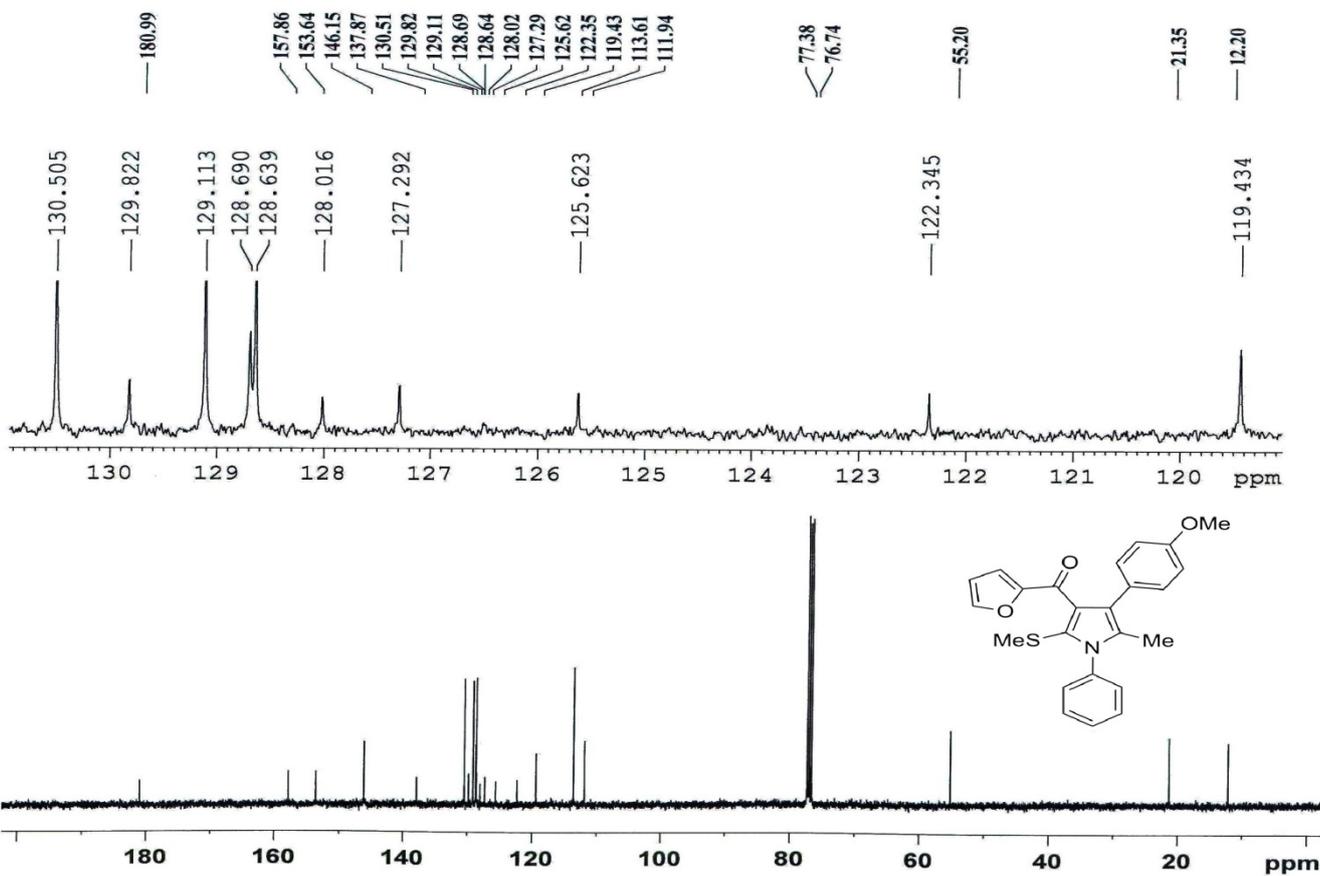
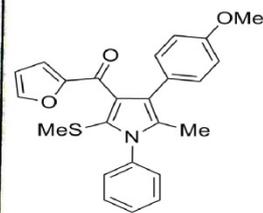
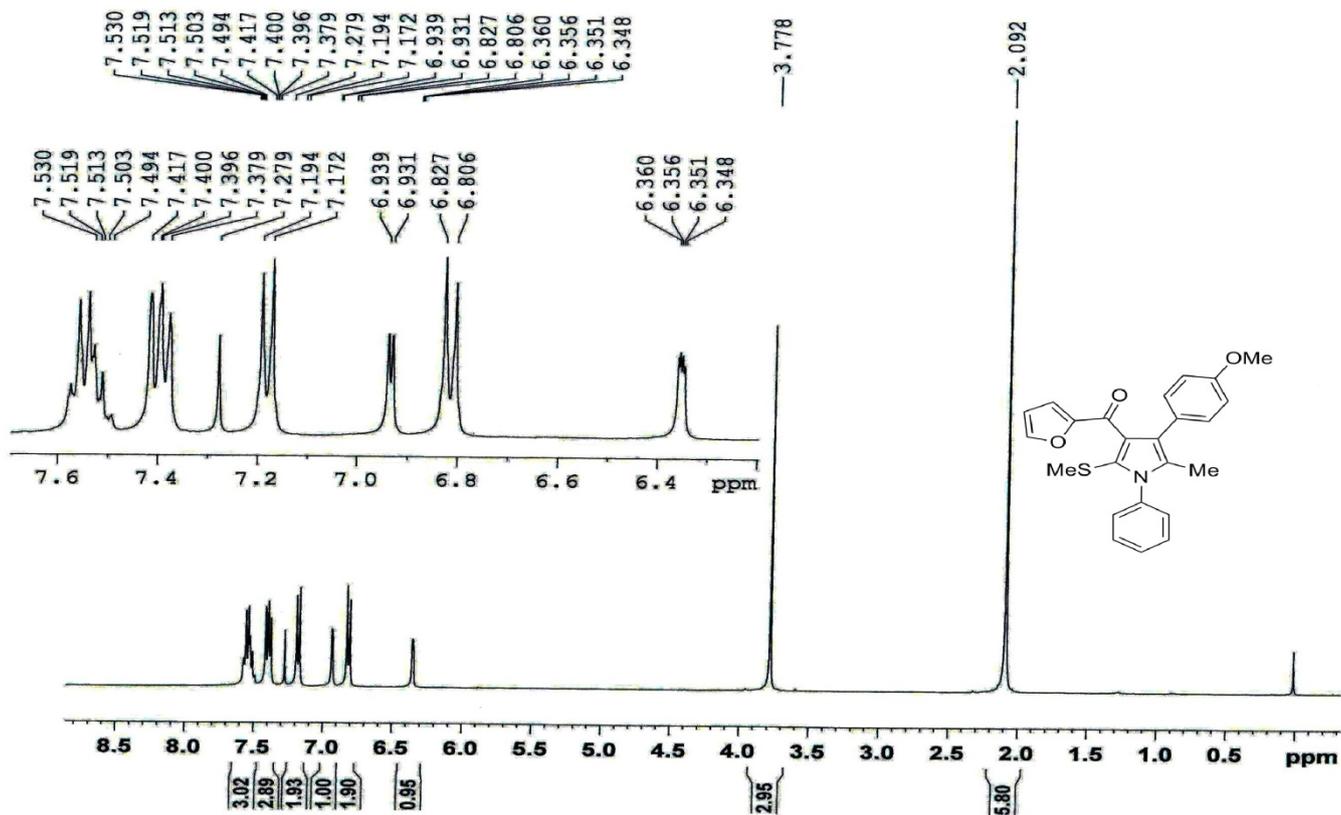
Peak List

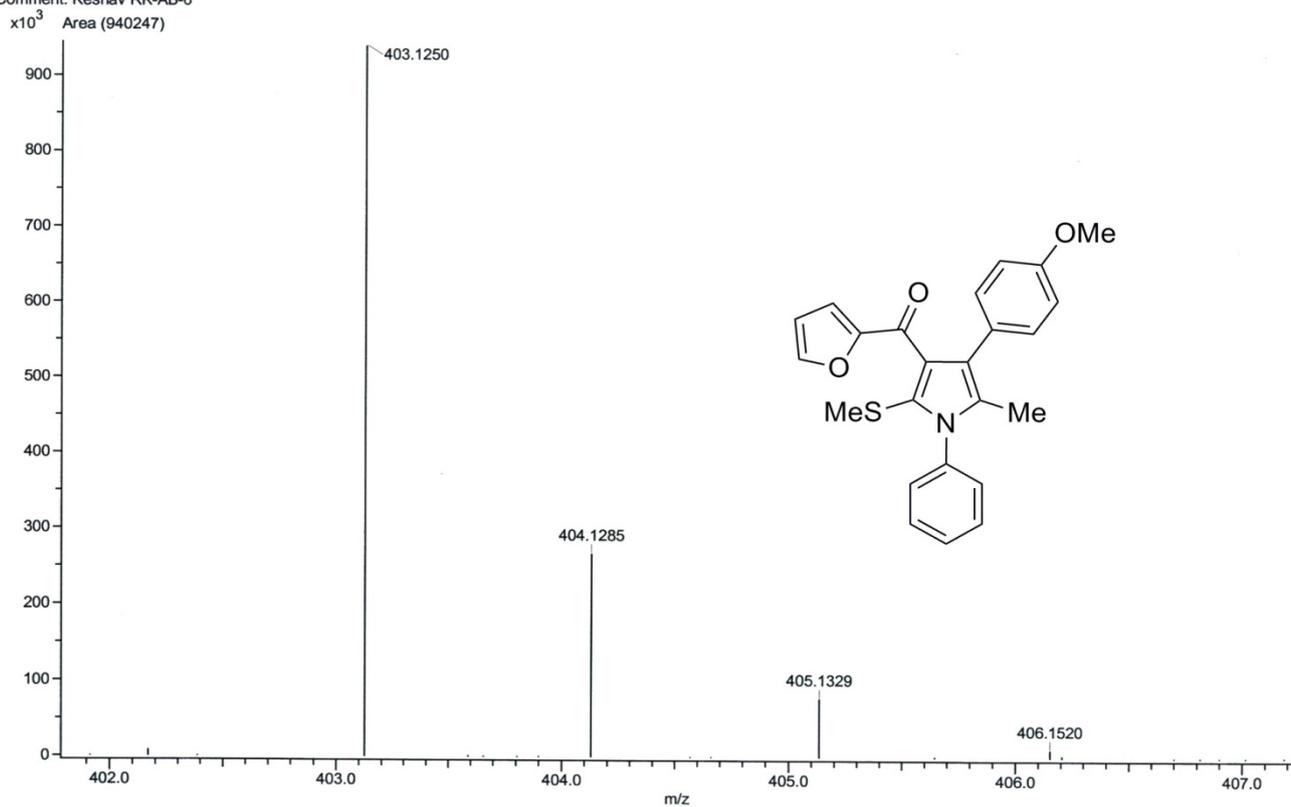
m/z	z	Abund
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342.1671	1	46344.09
478.1434	1	90620.97
481.1326	1	66885.29
498.134		72223.27
504.1215	1	97004.67
512.1496	1	112677.13
513.1527	1	34953.79
520.1078	1	114834.8
528.1445	1	75371.75



Fragmentor Voltage Collision Energy Ionization Mode

②63fh. 3-(2-Furoyl)-4-(4-methoxyphenyl)-5-methyl-2-(methylthio)-1-phenylpyrrole





References:

- [1] G. H. Elgemeie, H. A. Ali, A. H. Elghandour, A. M. Hussein, *Synth. Commun.* **2003**,*33*, 555-562.
- [2] D. E. Worrall, *Org. Synth.* **1941**,*Coll. Vol. 1*, 413.