

Ultrasound mediated, green innovation for the synthesis of polysubstituted 1,4-dihydropyridines

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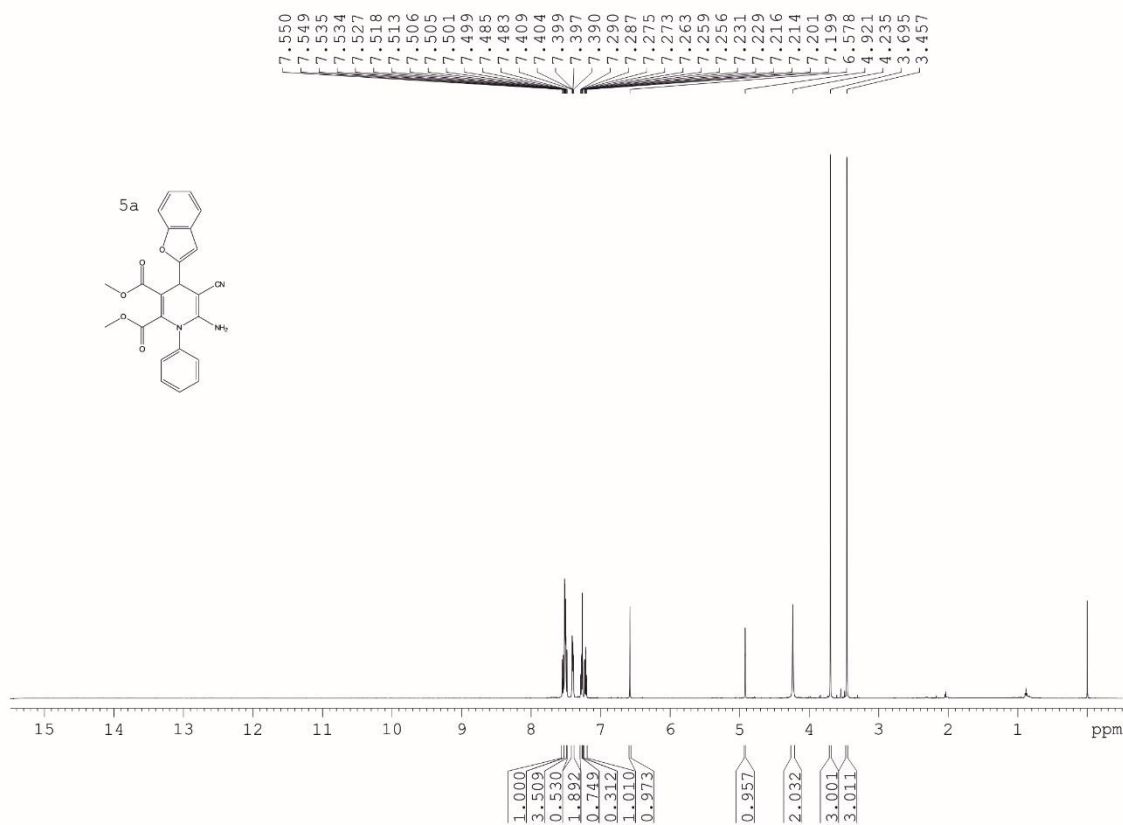
Palace road, Bangalore- 560 001, India

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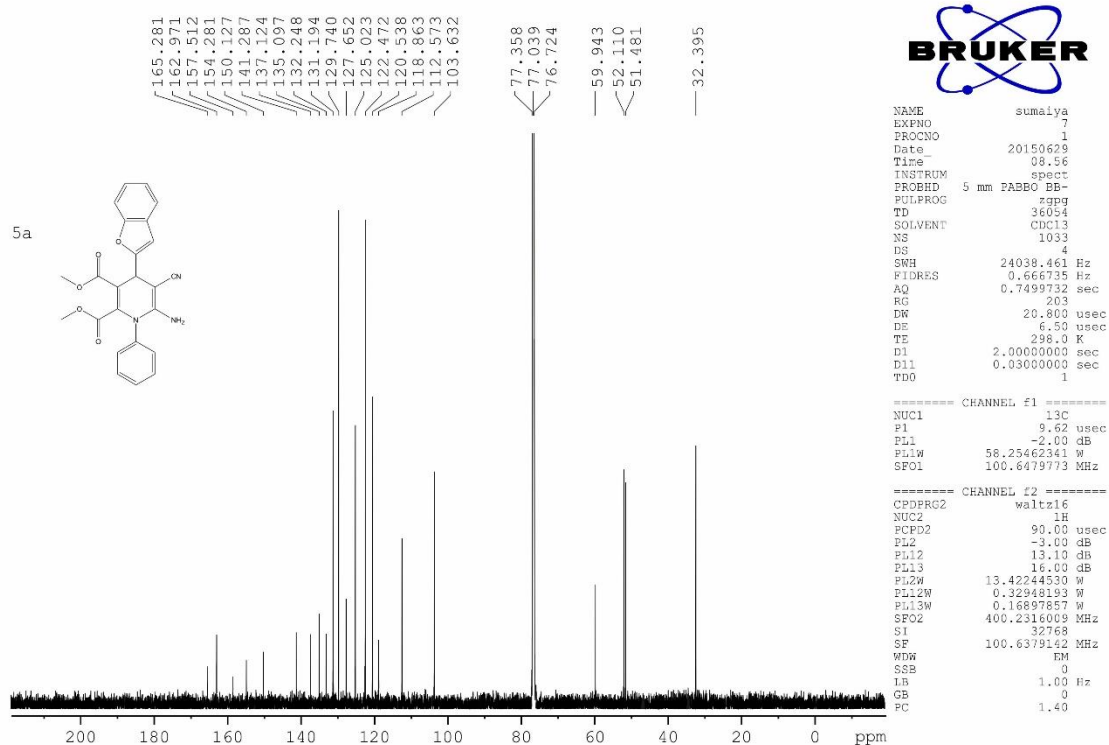
Supplementary data

Product	Analysis			
5a	¹ H NMR	¹³ C NMR	ESI-MS	CHN
5b	¹ H NMR	¹³ C NMR	ESI-MS	CHN
5c	¹ H NMR	¹³ C NMR	ESI-MS	CHN
5d	¹ H NMR	¹³ C NMR	ESI-MS	CHN
5e	¹ H NMR	¹³ C NMR	ESI-MS	CHN
5f	¹ H NMR	¹³ C NMR	ESI-MS	CHN
5g	¹ H NMR	¹³ C NMR	ESI-MS	CHN
5h	¹ H NMR	¹³ C NMR	ESI-MS	CHN
5i	¹ H NMR	¹³ C NMR	ESI-MS	CHN
5j	¹ H NMR	¹³ C NMR	ESI-MS	CHN
5k	¹ H NMR	¹³ C NMR	ESI-MS	CHN
5l	¹ H NMR	¹³ C NMR	ESI-MS	CHN
5m	¹ H NMR	¹³ C NMR	ESI-MS	CHN
5n	¹ H NMR	¹³ C NMR	ESI-MS	CHN
5o	¹ H NMR	¹³ C NMR	ESI-MS	CHN
5p	¹ H NMR	¹³ C NMR	-	-
5q	¹ H NMR	¹³ C NMR	-	-
5r	¹ H NMR	¹³ C NMR	-	-

¹H NMR spectrum of 5a



¹³C NMR spectrum of 5a



ESI-MS of 5a

Display Report

Analysis Info

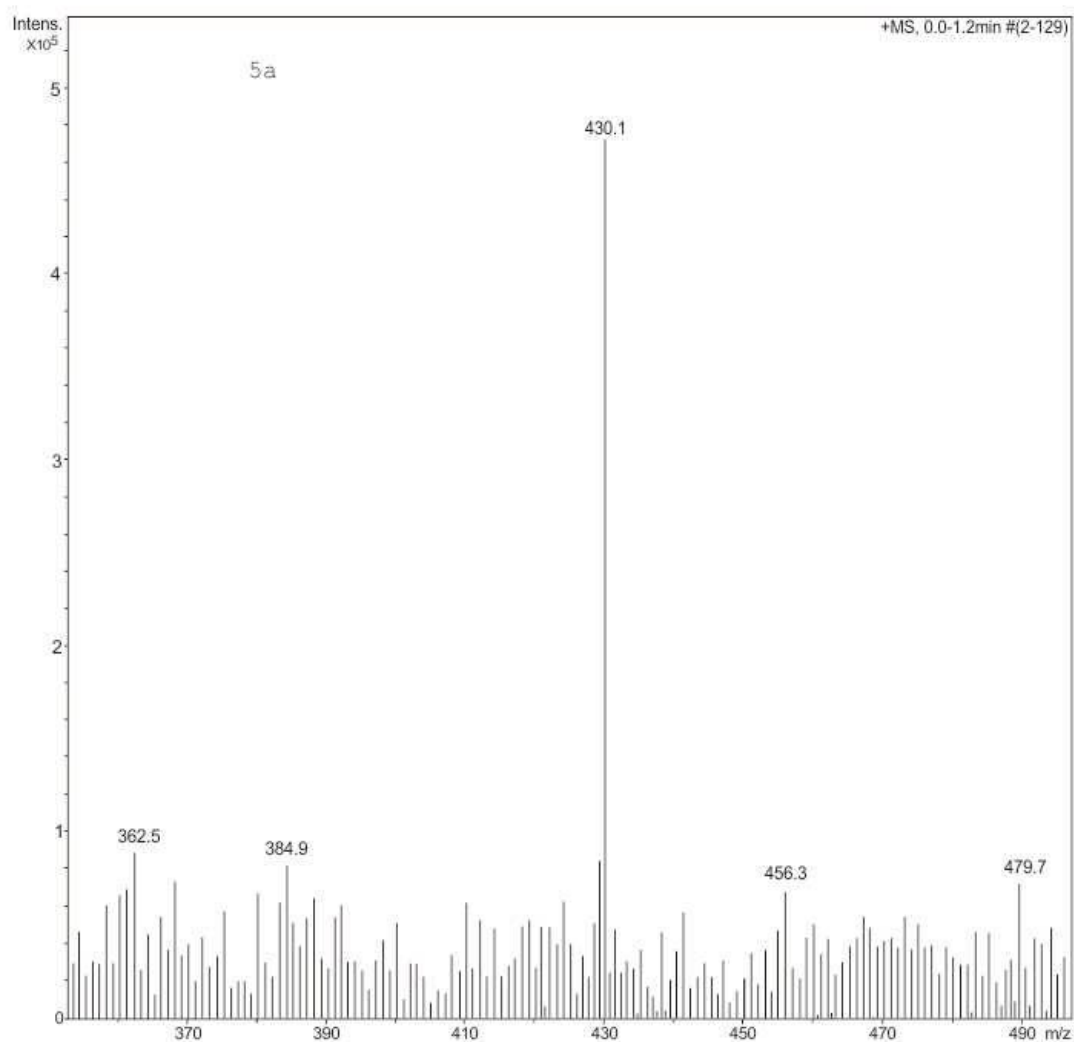
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Sample Name Default

Acquisition Parameter

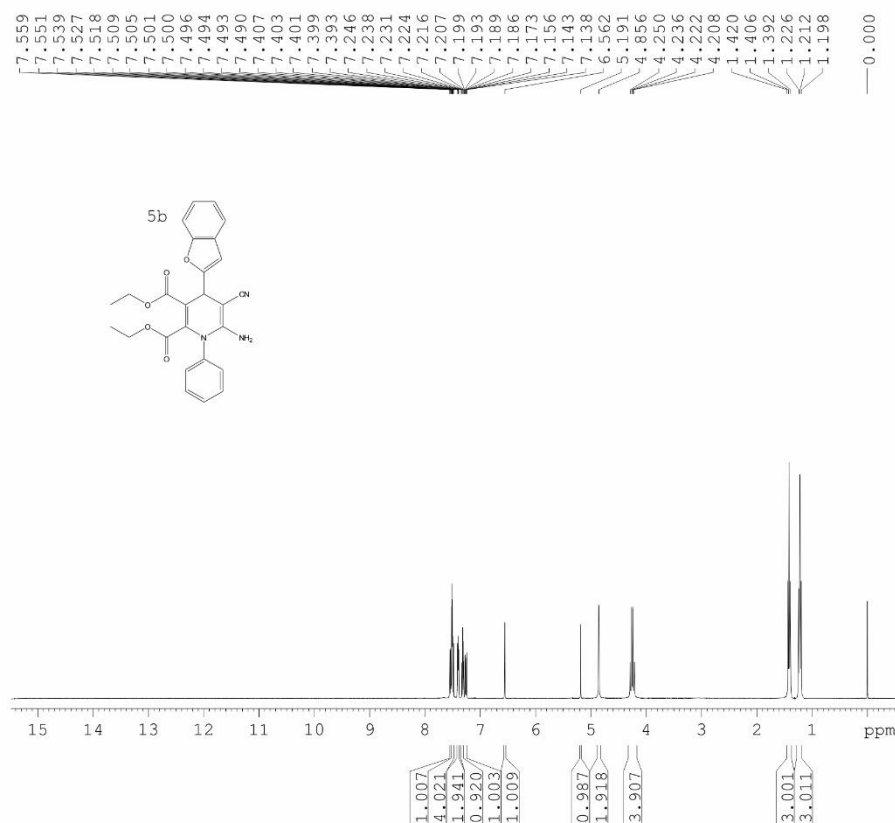
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Capillary Exit	104.5 Volt	Skimmer	40.0 Volt
Accumulation Time	Ultra Scan	Averages	5 Spectra

Acquisition Date 16/3/2015 2:44:15 PM

Operator MBU
Instrument HCT ultra ETD II



¹H NMR spectrum of 5b



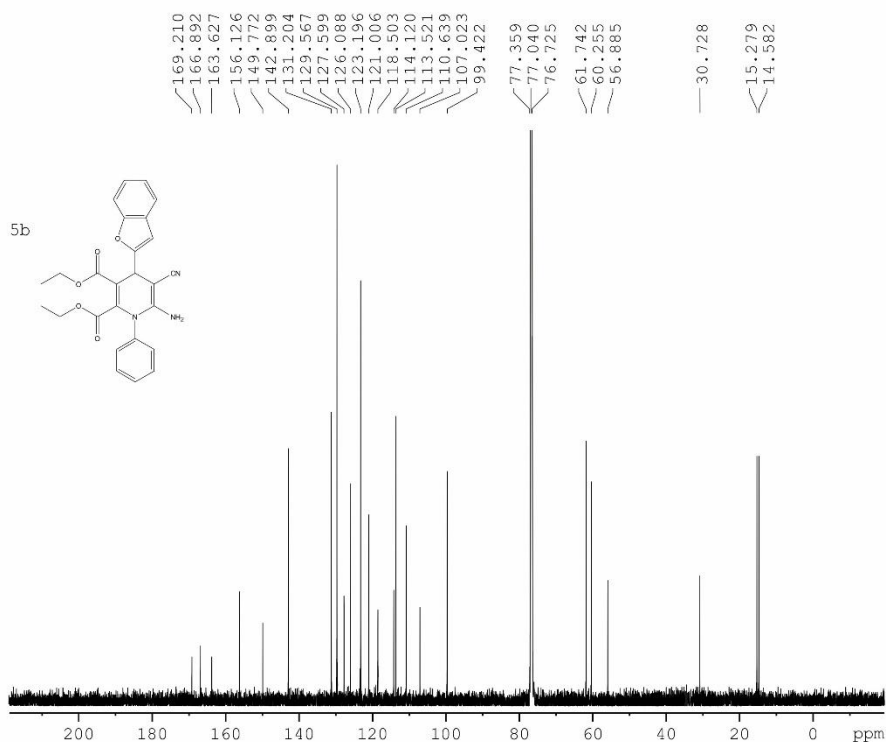
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PROCNO 1

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FIDRES 0.250026 Hz
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DE 6.50 usec
TE 298.0 K
D1 2.0000000 sec
TD0 1

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F2 - Processing parameters
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SF 500.1300120 MHz
WDW EM
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PC 1.00

¹³C NMR spectrum of 5b



NAME sumaiya
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Time 07.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
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FIDRES 0.666735 Hz
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PLW 58.25462341 W
SFO1 100.6479773 MHz

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PCPD2 90.00 usec
PL2 -3.00 dB
PL12 13.10 dB
PL13 16.00 dB
PL2W 13.42244530 W
PL12W 0.32948193 W
PL13W 0.16897857 W
SFO2 400.2316009 MHz
SI 32768
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WDW EM
SSB 0
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ESI-MS of 5b

Display Report

Analysis Info

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Sample Name Default

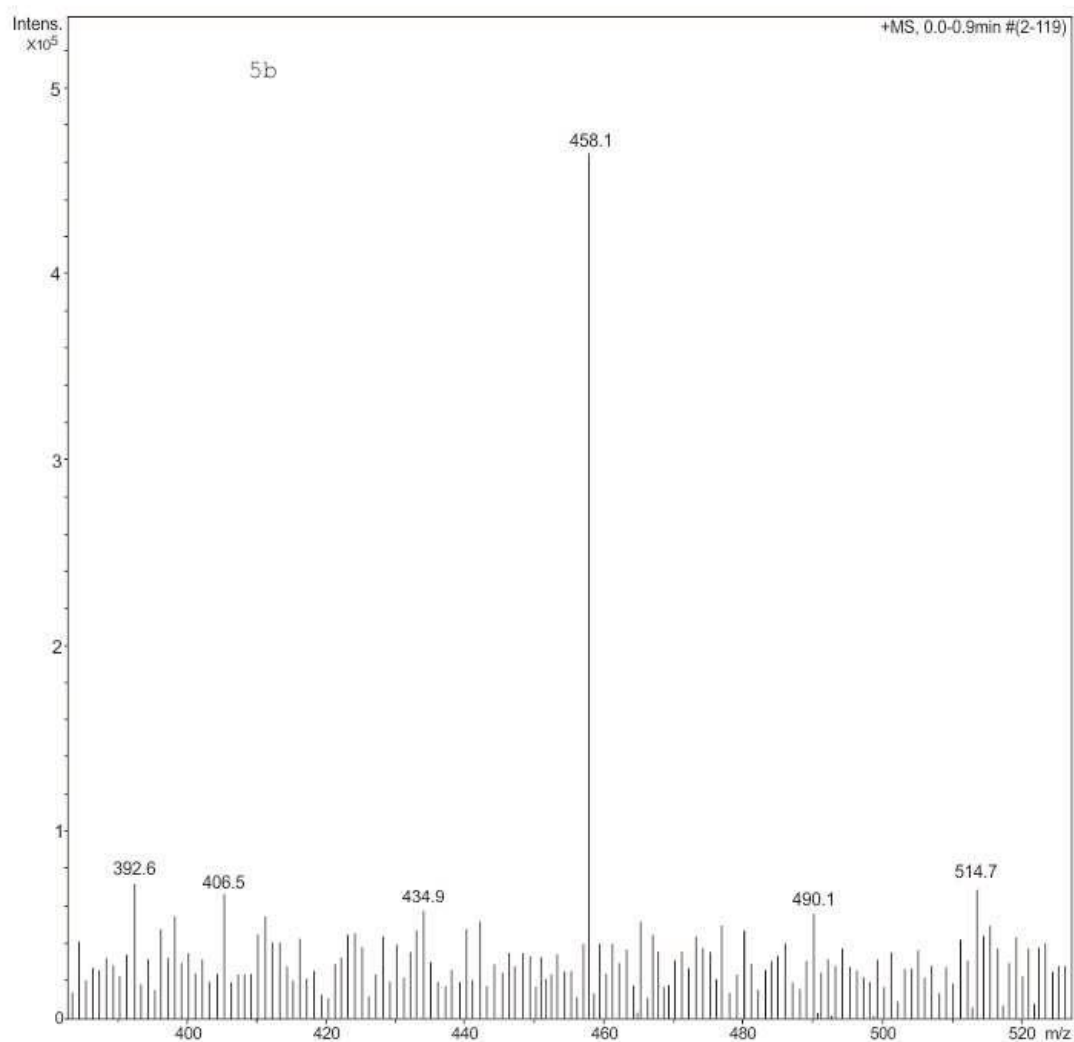
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Operator MBU
Instrument HCT ultra ETD II

Acquisition Parameter

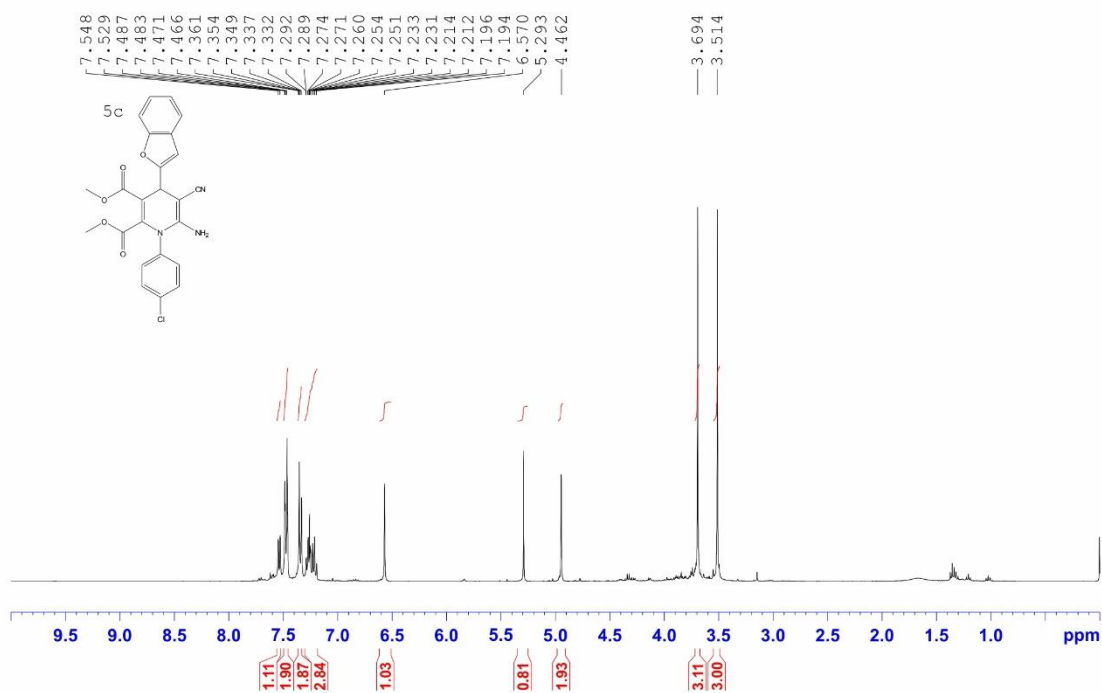
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Accumulation Time	Ultra Scan	Averages	5 Spectra

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Scan End	900 m/z
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Auto MS/MS	off

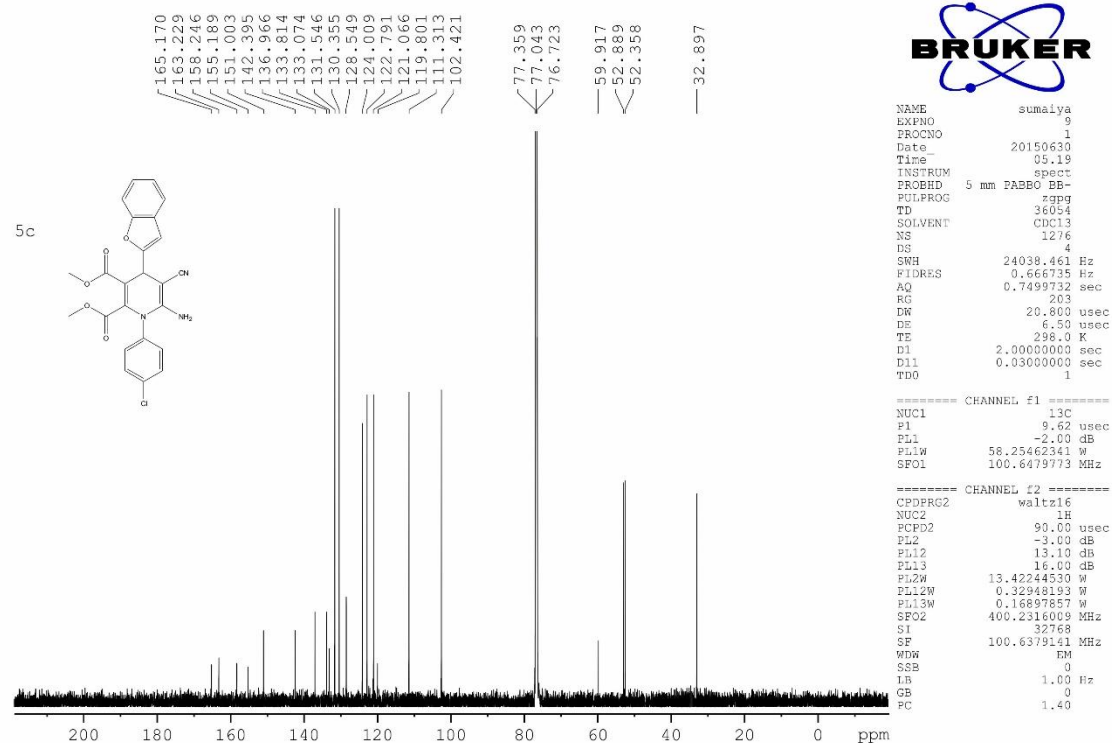


¹H NMR spectrum of 5c

Feb09-2015
PROTON CDCl₃



¹³C NMR spectrum of 5c



ESI-MS of 5c

Display Report

Analysis Info

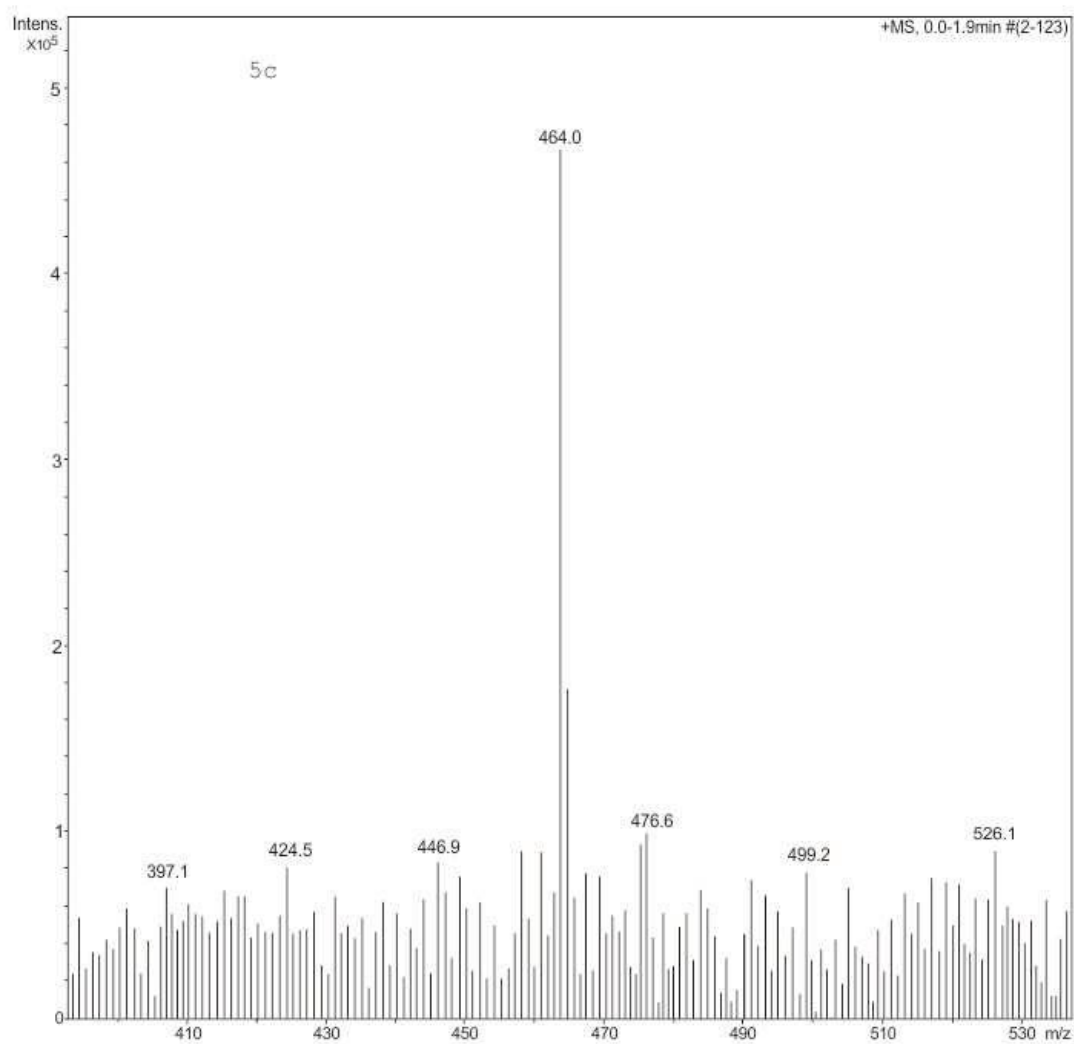
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Method DEF_MS.M
Sample Name Default

Acquisition Parameter

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Capillary Exit	104.5 Volt	Skimmer	40.0 Volt
Accumulation Time	Ultra Scan	Averages	5 Spectra

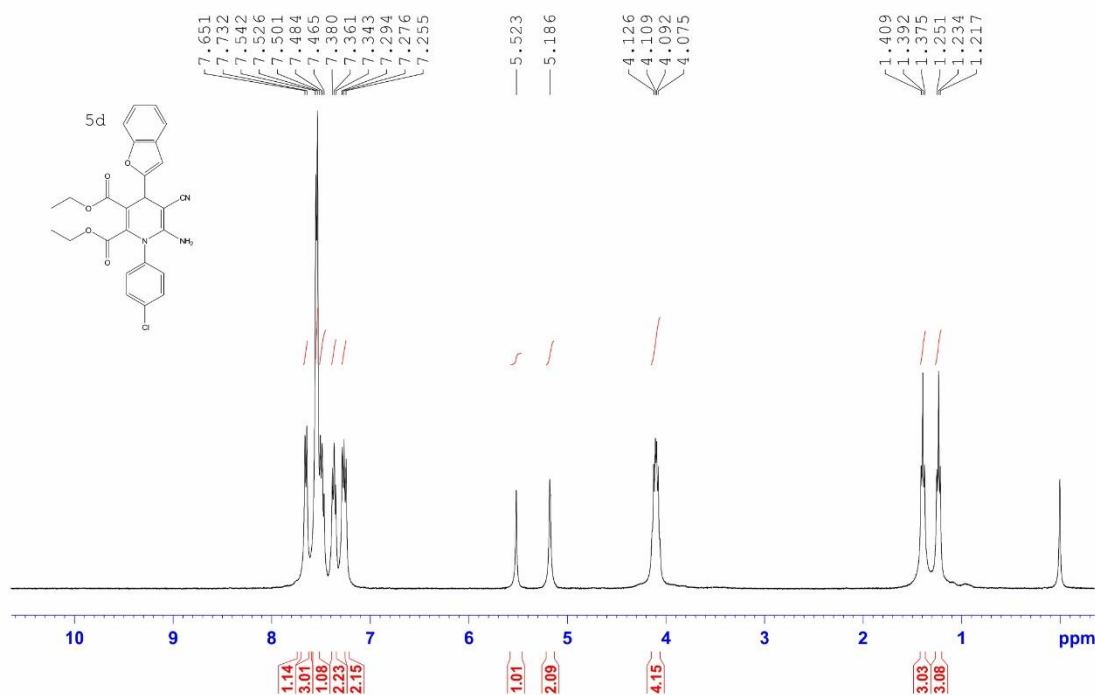
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Operator MBU
Instrument HCT ultra ETD II

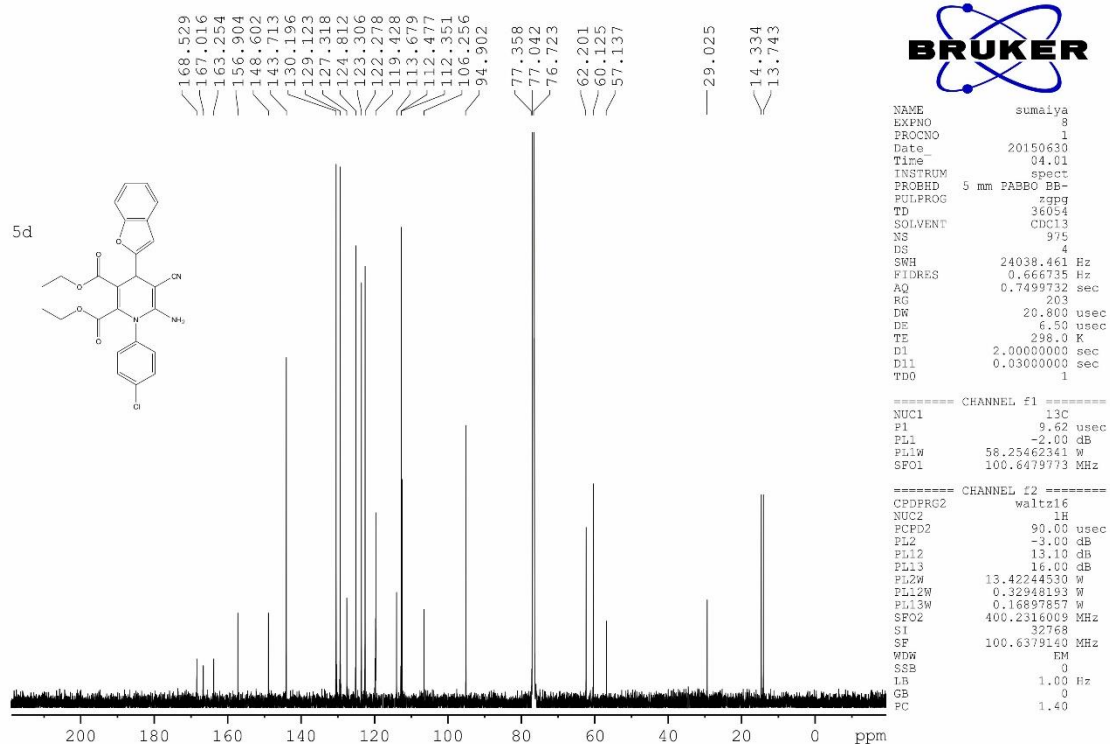


¹H NMR spectrum of 5d

Feb09-2015
PROTON CDC13



¹³C NMR spectrum of 5d



ESI-MS of 5d

Display Report

Analysis Info

Analysis Name F:\Reports\GS\16MAR15-----492.d
Method DEF_MS.M
Sample Name Default

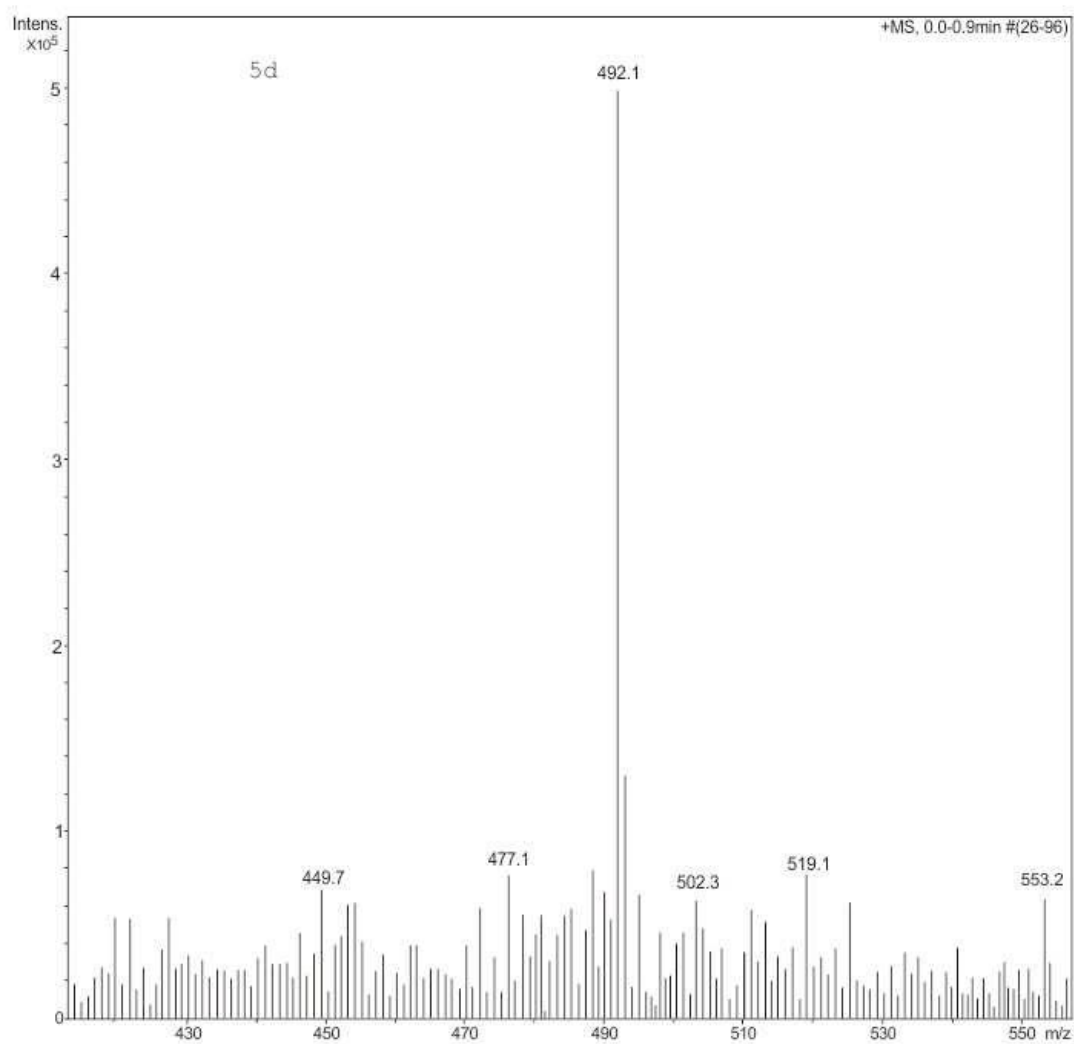
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Operator MBU
Instrument HCT ultra ETD II

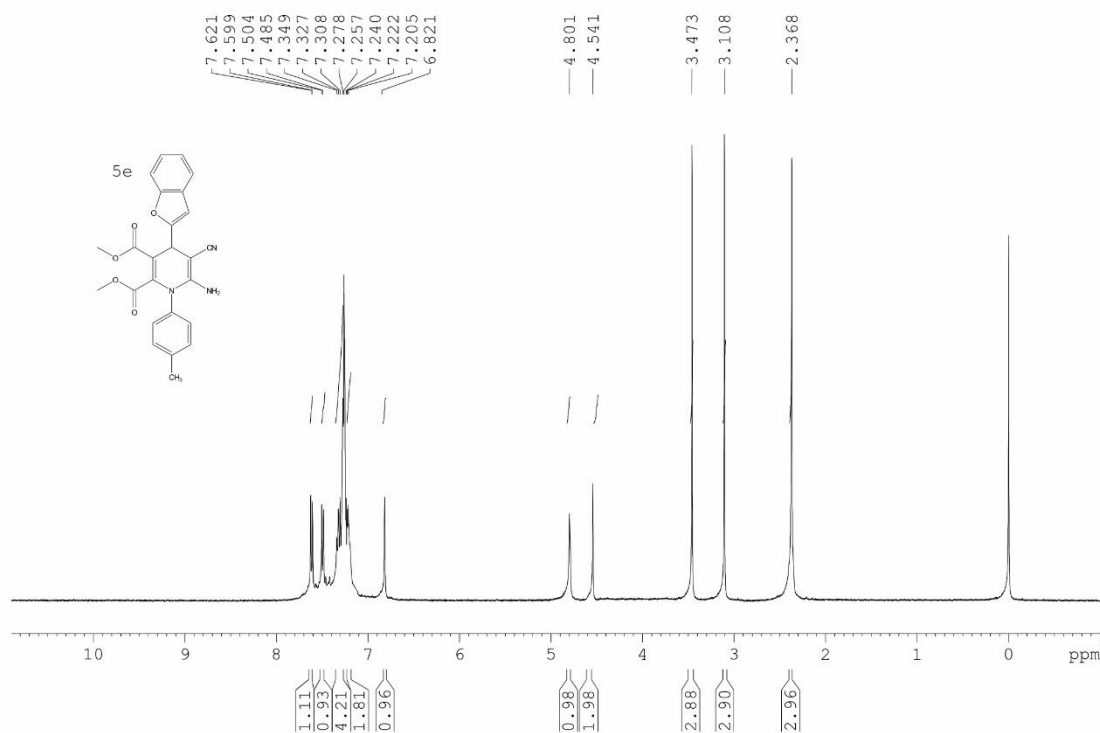
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Accumulation Time	Ultra Scan	Averages	5 Spectra

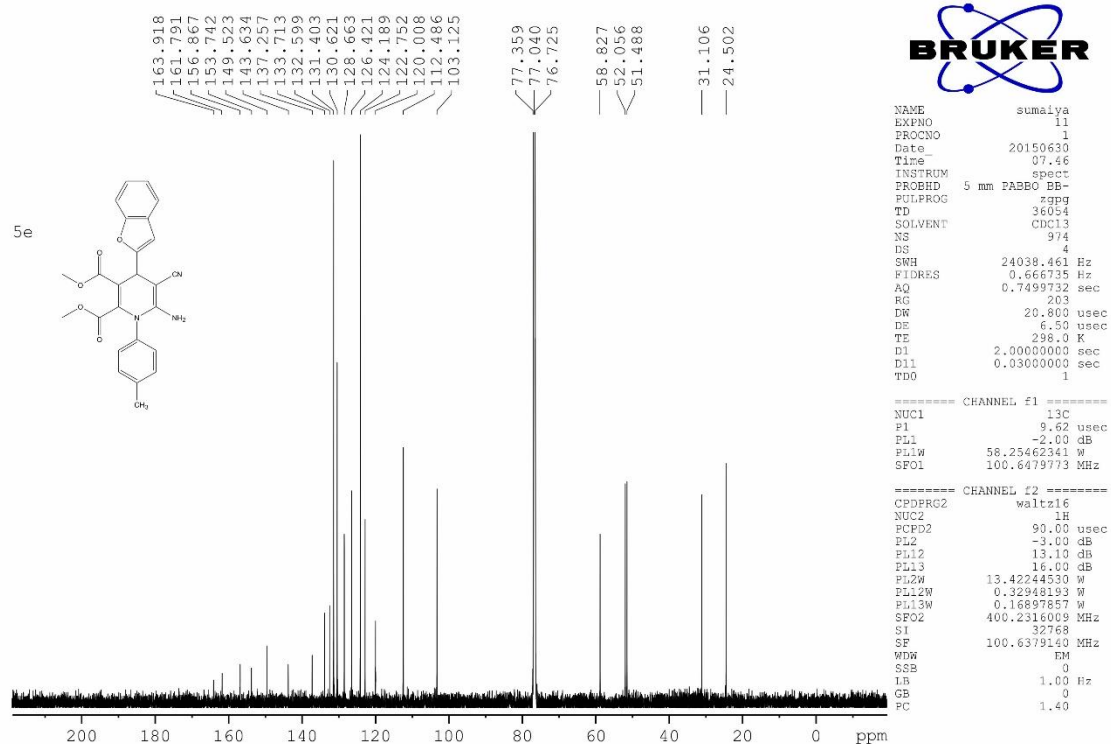
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Trap Drive	43.3
Auto MS/MS	off



¹H NMR spectrum of 5e



^{13}C NMR spectrum of 5e



ESI-MS of 5e

Display Report

Analysis Info

Analysis Name F:\Reports\GS\16MAR15-----444.d
Method DEF_MS.M
Sample Name Default

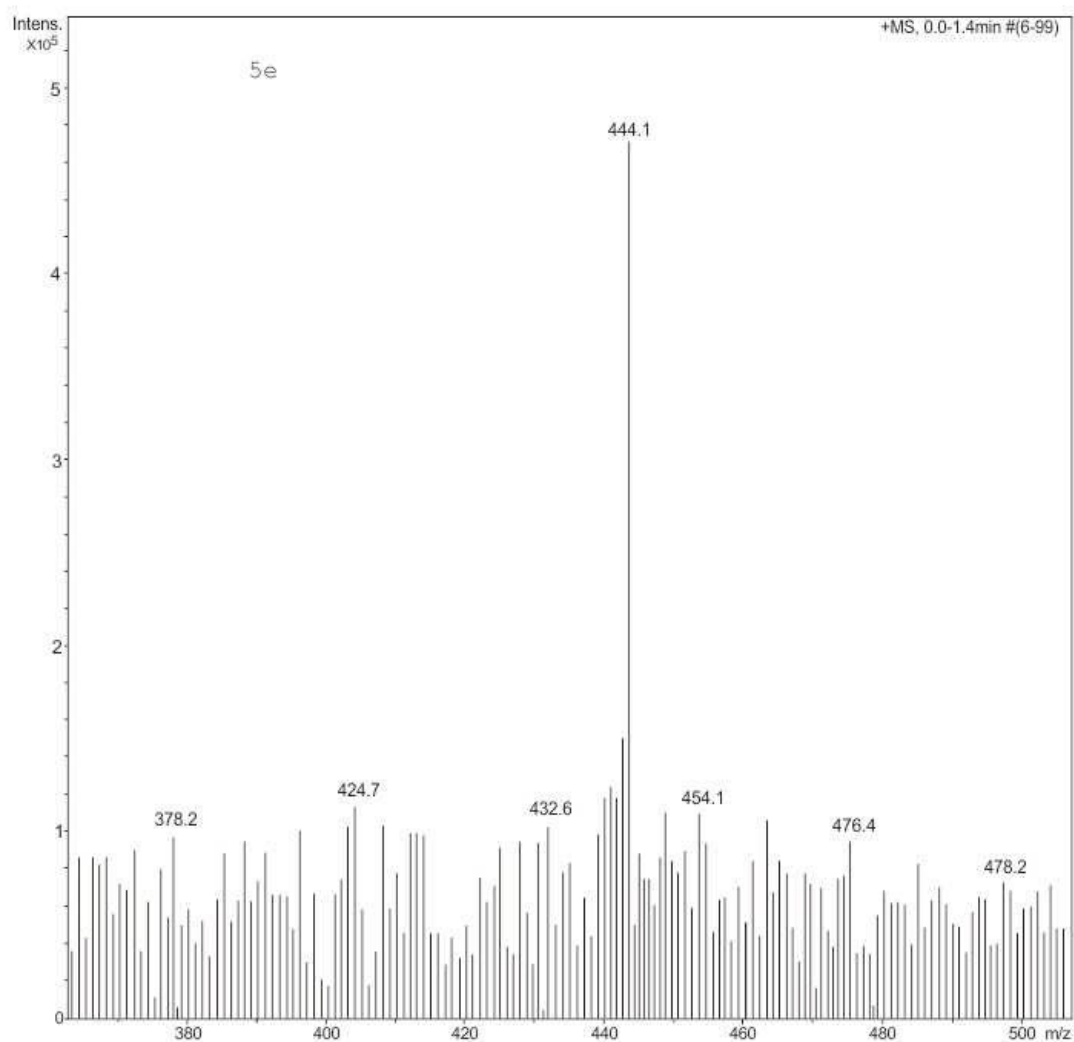
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Operator MBU
Instrument HCT ultra ETD II

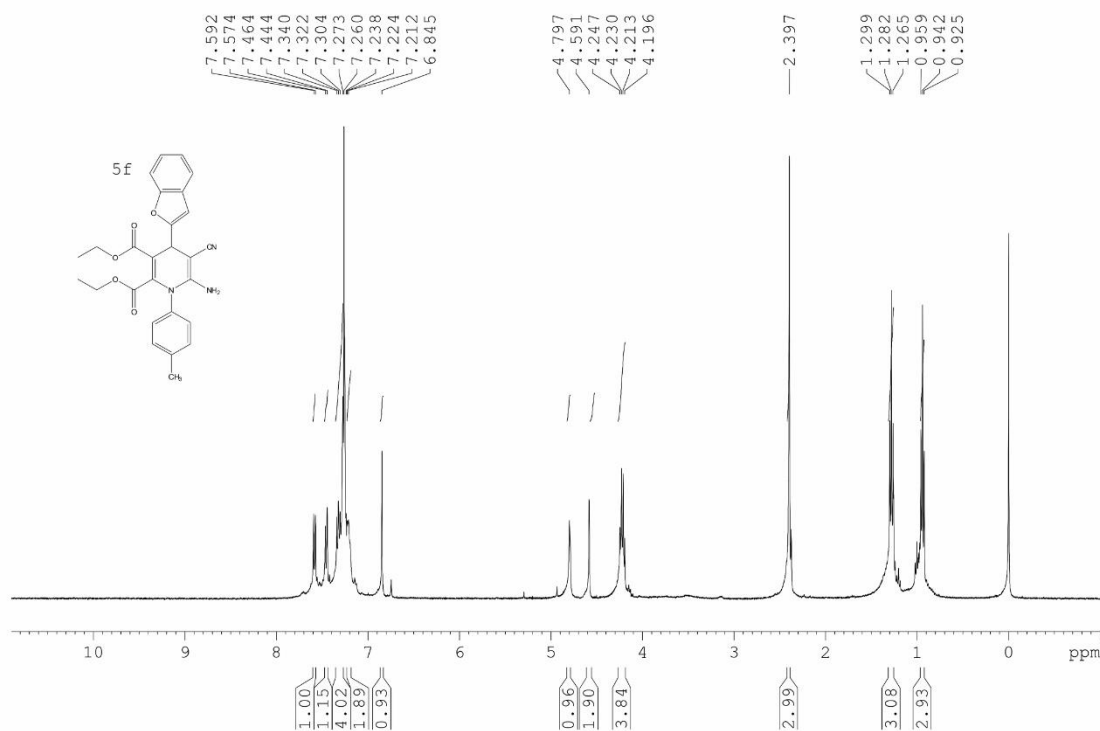
Acquisition Parameter

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Accumulation Time	Ultra Scan	Averages	5 Spectra

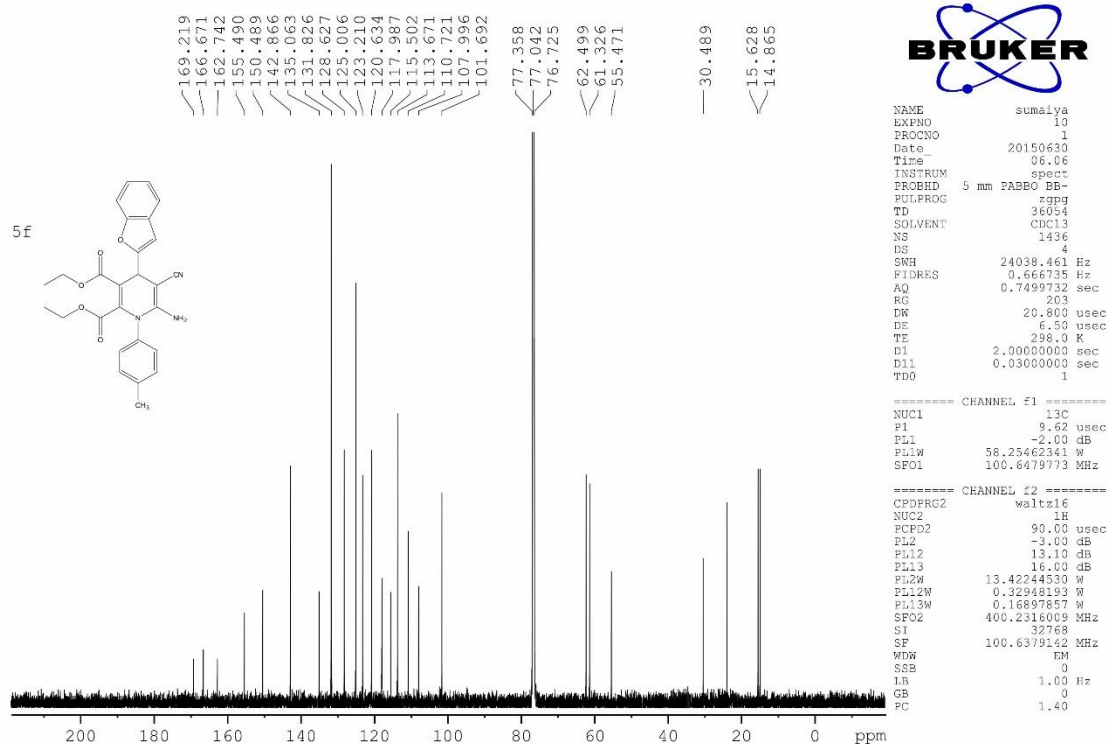
Alternating Ion Polarity	off
Scan End	900 m/z
Trap Drive	43.3
Auto MS/MS	off



¹H NMR spectrum of 5f



¹³C NMR spectrum of 5f



ESI-MS of 5f

Display Report

Analysis Info

Analysis Name F:\Reports\GS\16MAR15-----472.d
Method DEF_MS.M
Sample Name Default

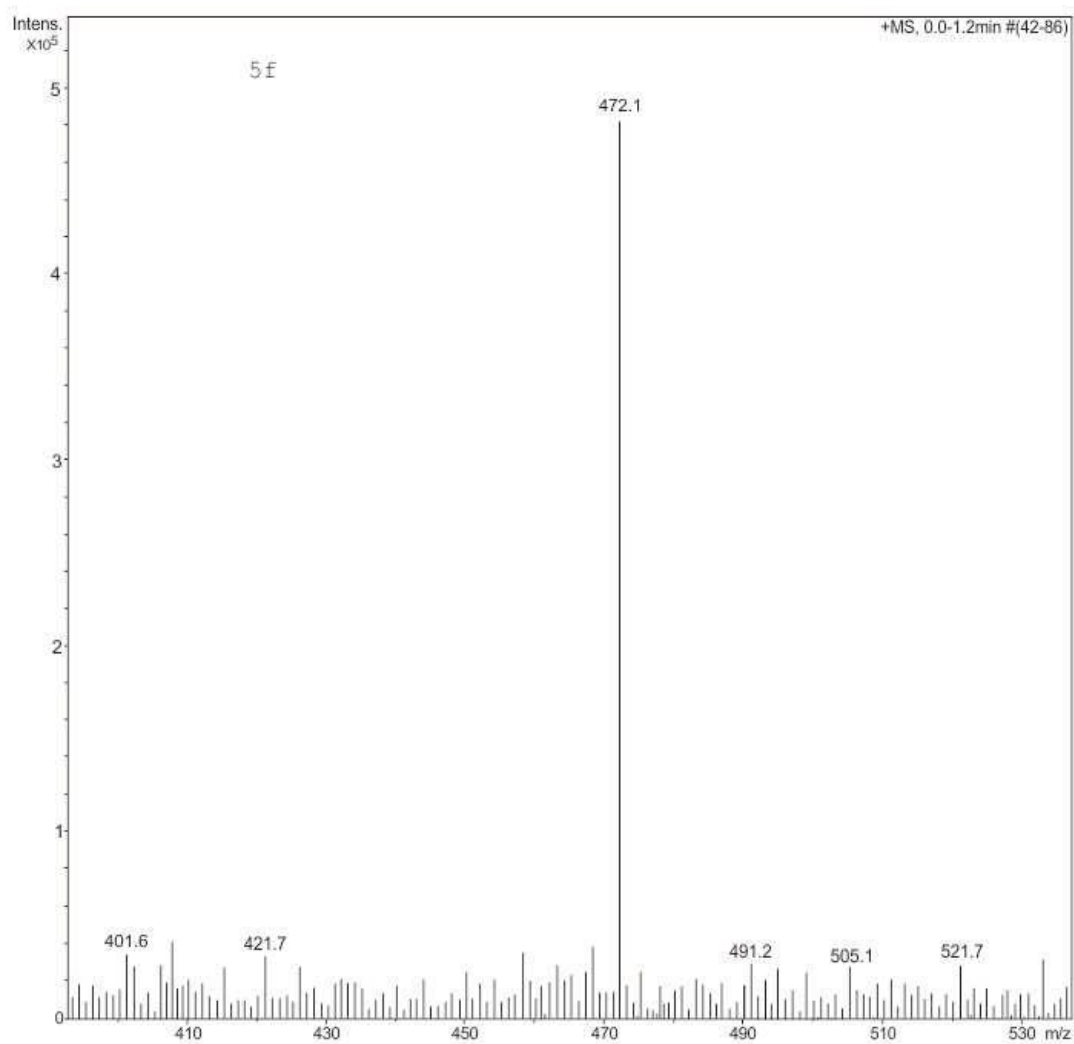
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Operator MBU
Instrument HCT ultra ETD II

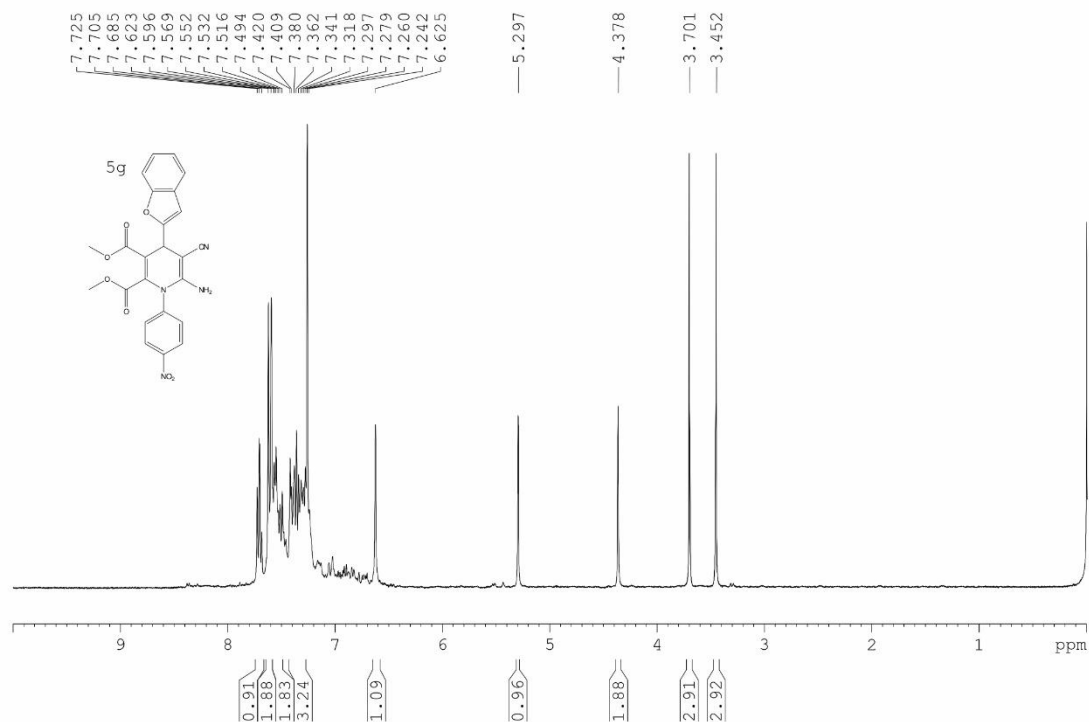
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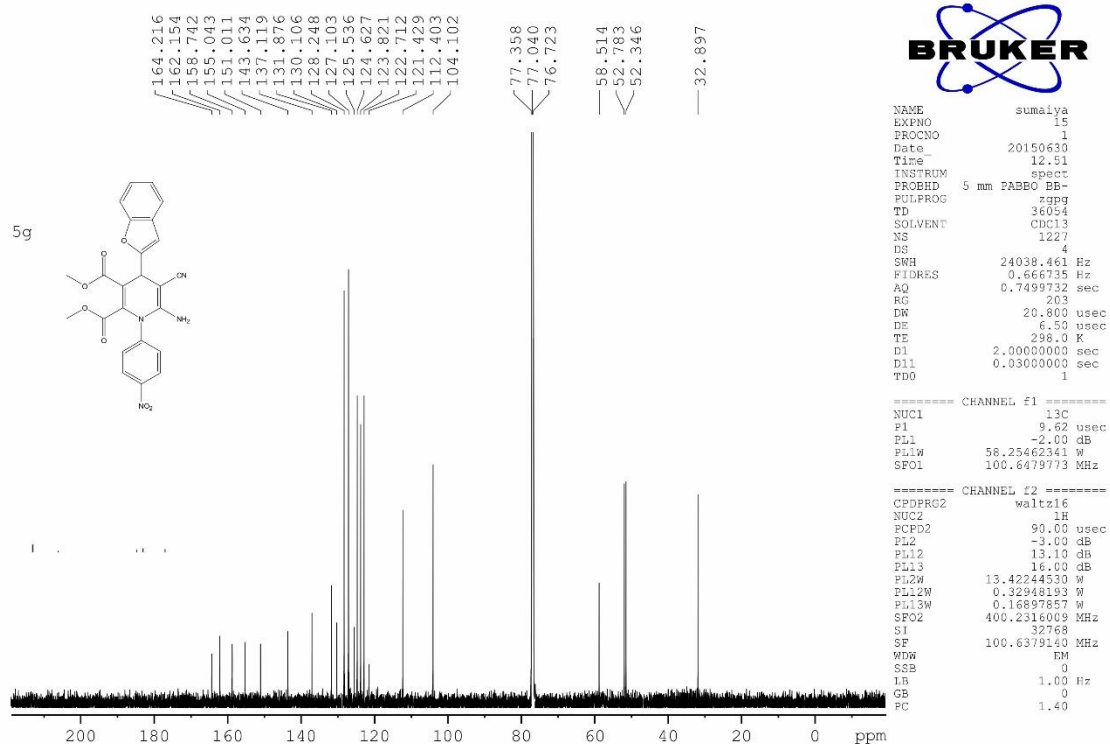
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Scan End	900 m/z
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Auto MS/MS	off



¹H NMR spectrum of 5g



¹³C NMR spectrum of 5g



ESI-MS of 5g

Display Report

Analysis Info

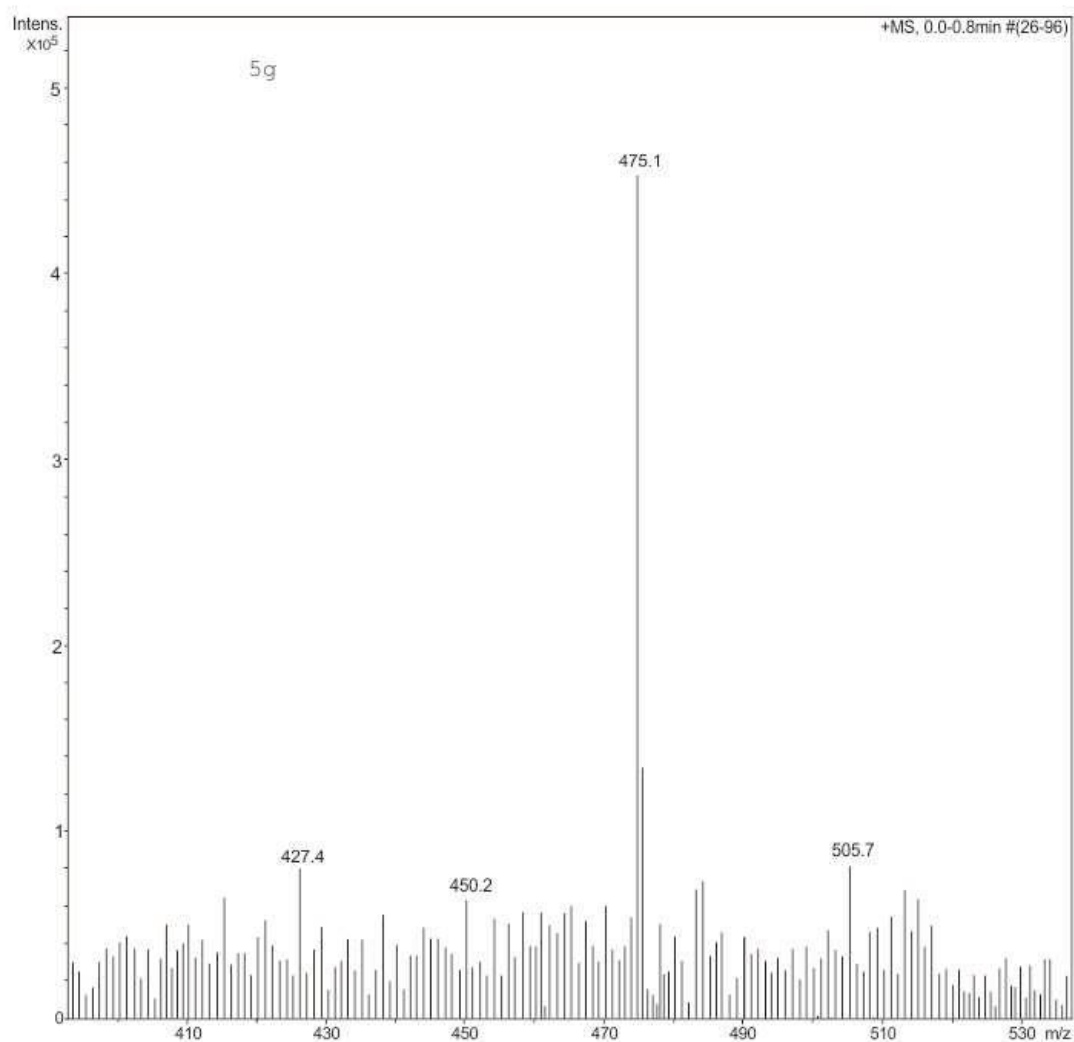
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Method DEF_MS.M
Sample Name Default

Acquisition Parameter

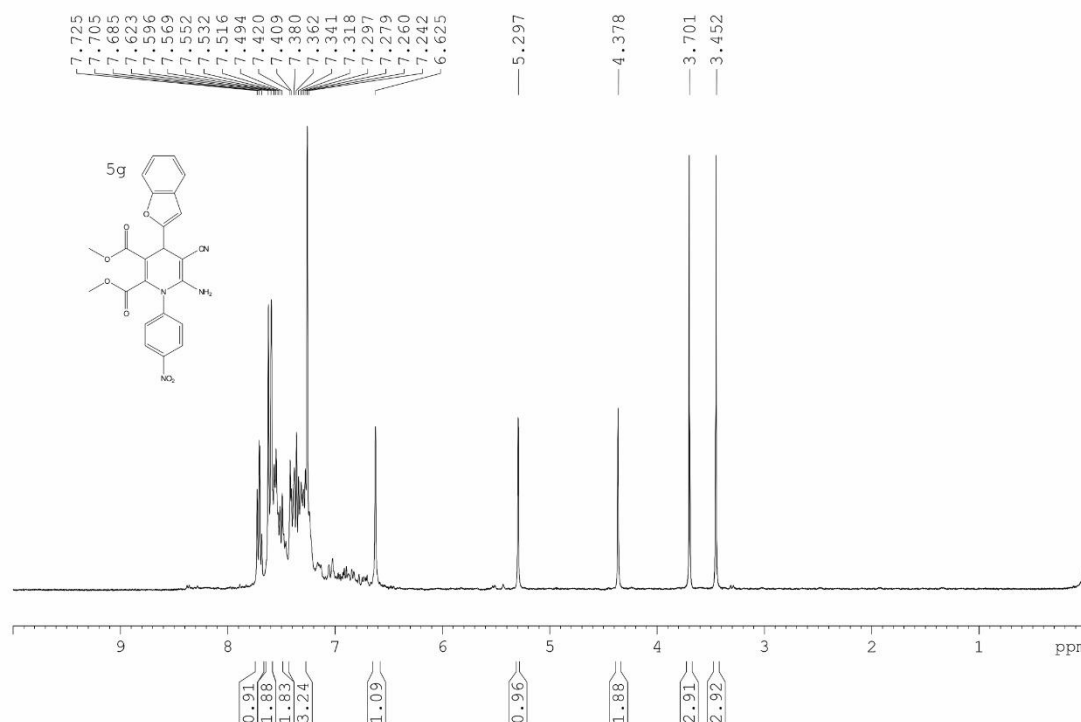
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Accumulation Time	Ultra Scan	Averages	5 Spectra

Acquisition Date 16/3/2015 3:53:53 PM

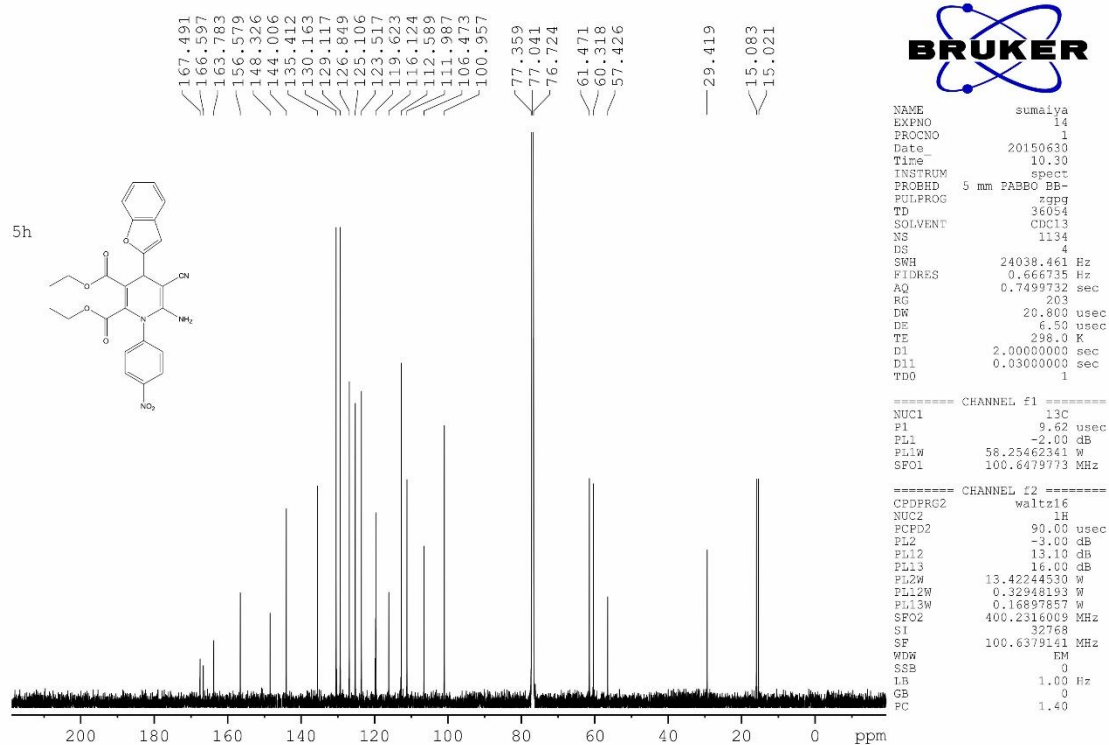
Operator MBU
Instrument HCT ultra ETD II



¹H NMR spectrum of 5h



¹³C NMR spectrum of 5h



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PROCNO 1
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Time 10.30
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PULPROG zgpg
TD 36054
SOLVENT CDC13
NS 1134
DS 4
SWH 24038.461 Hz
FIDRES 0.666735 Hz
AQ 0.7499732 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TDO 1

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P1 9.62 usec
PL1 -2.00 dB
PL1W 58.25462341 W
SF01 100.6479773 MHz

===== CHANNEL f2 =====
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PCPD2 90.00 usec
PL2 -3.00 dB
PL12 13.10 dB
PL13 16.00 dB
PL2W 13.42244530 W
PL12W 0.32948193 W
PL13W 0.16897857 W
SF02 400.2316009 MHz
SI 32768
SF 100.6379141 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
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ESI-MS of 5h

Display Report

Analysis Info

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Method DEF_MS.M
Sample Name Default

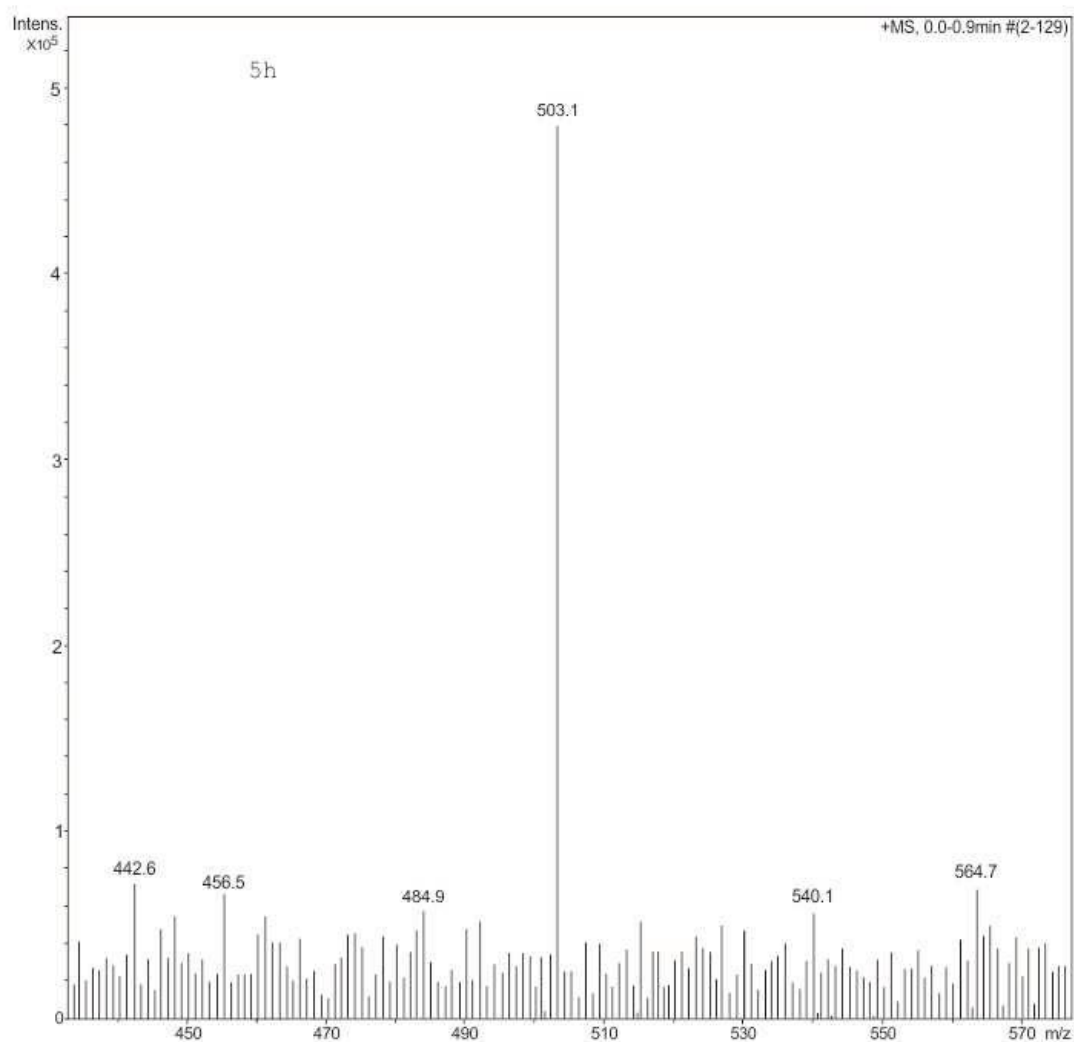
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Accumulation Time	Ultra Scan	Averages	5 Spectra

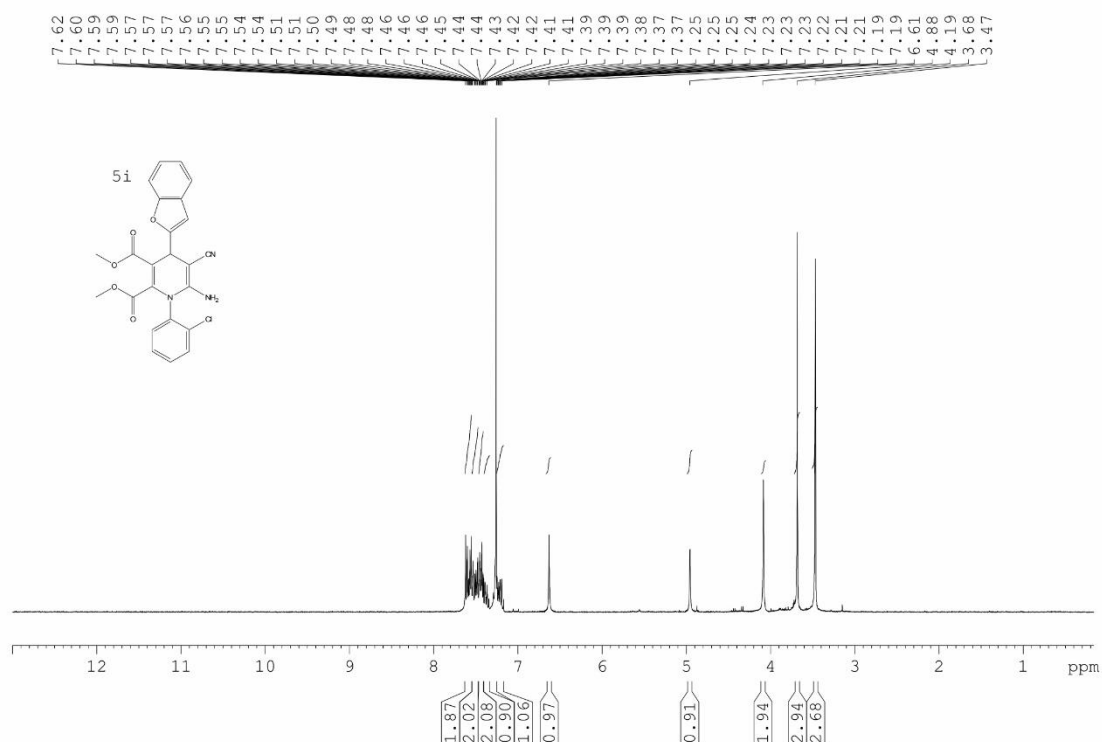
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Operator MBU
Instrument HCT ultra ETD II

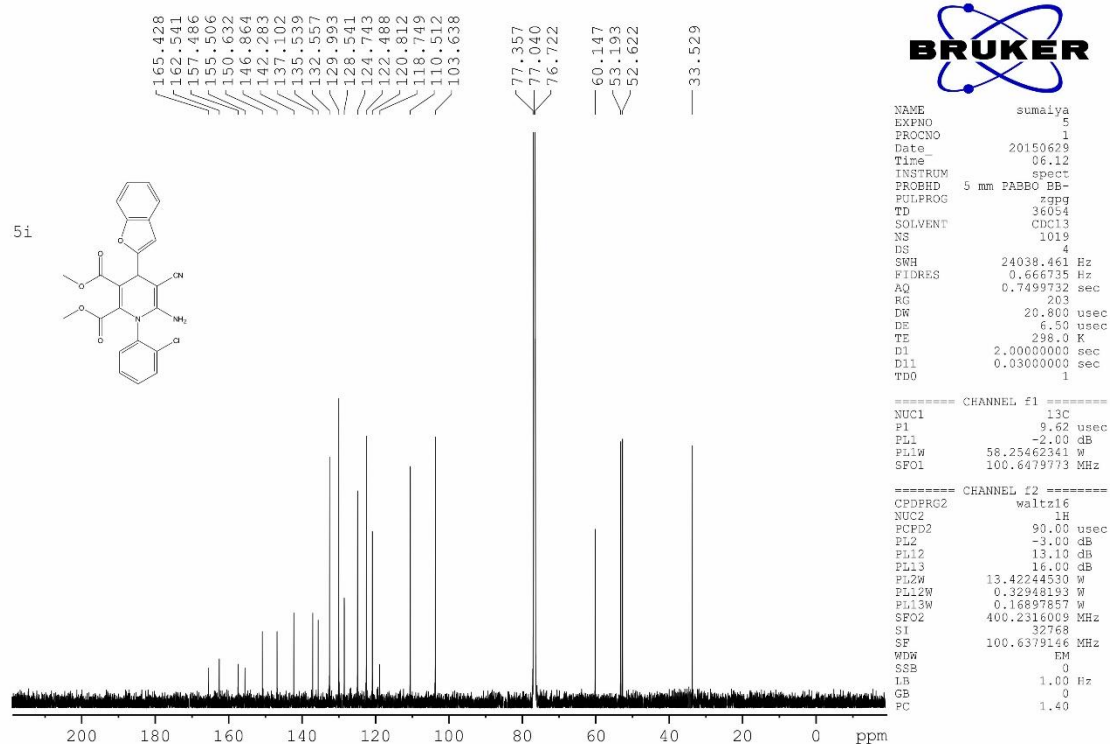
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Scan End	900 m/z
Trap Drive	43.3
Auto MS/MS	off



¹H NMR spectrum of 5i



¹³C NMR spectrum of 5i



ESI-MS of 5i

Display Report

Analysis Info

Analysis Name F:\Reports\GS\17MAR15-----464.d
Method DEF_MS.M
Sample Name Default

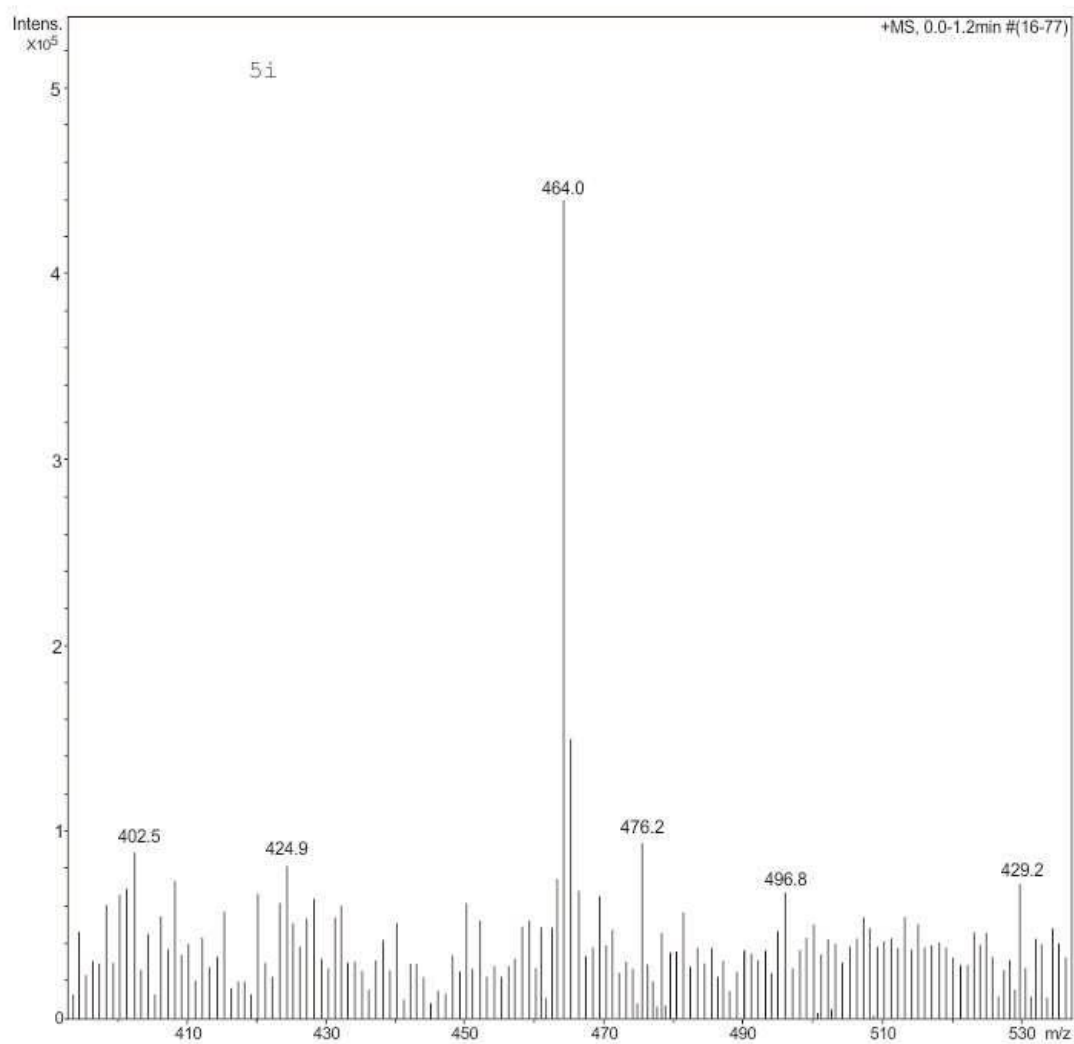
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Operator MBU
Instrument HCT ultra ETD II

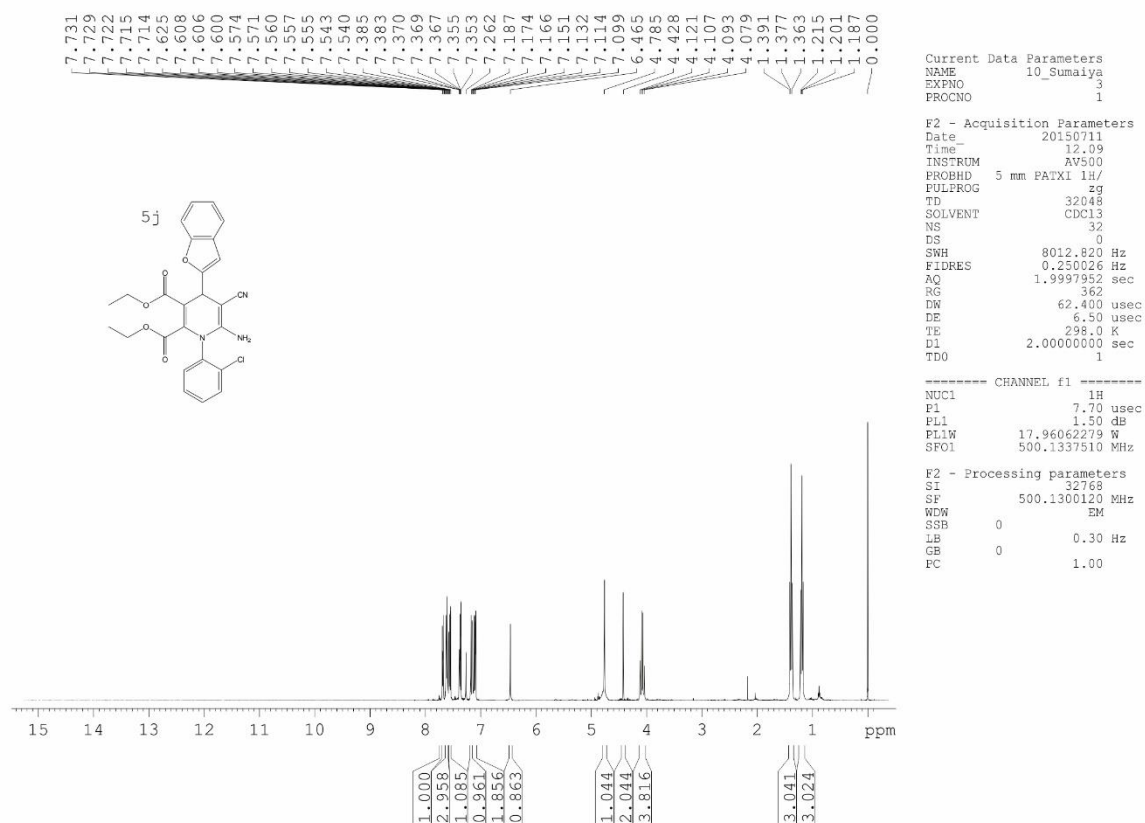
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Accumulation Time	Ultra Scan	Averages	5 Spectra

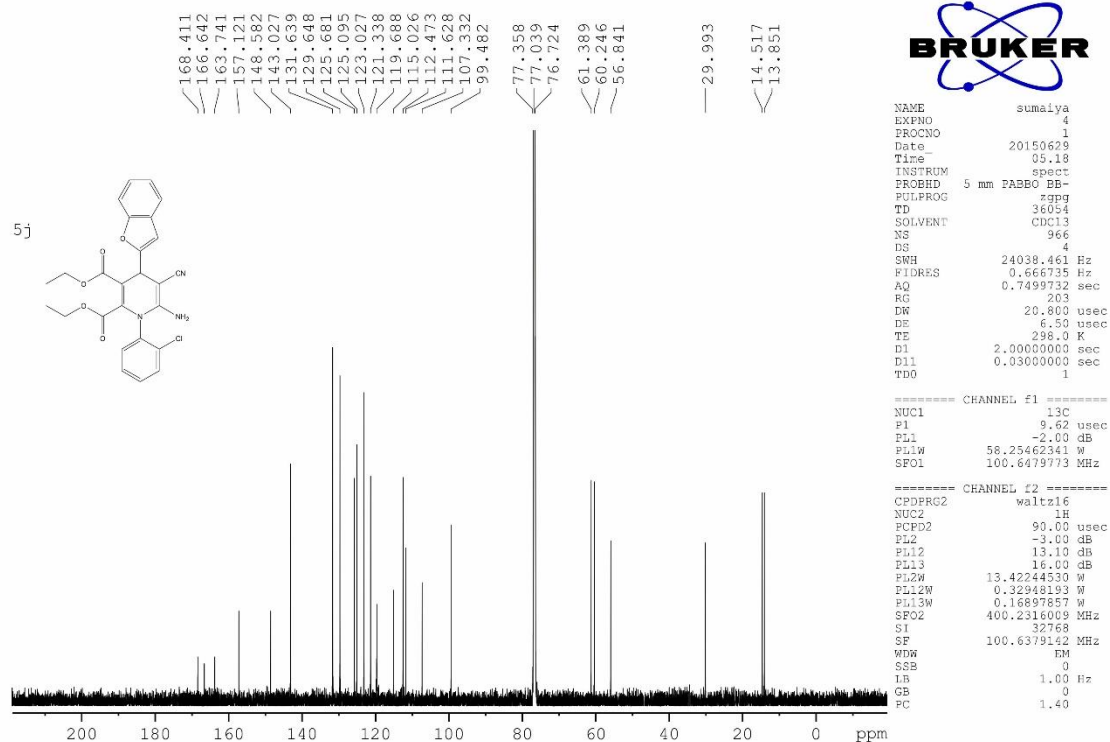
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Auto MS/MS	off



¹H NMR spectrum of 5j



¹³C NMR spectrum of 5j



ESI-MS of 5j

Display Report

Analysis Info

Analysis Name F:\Reports\GS\17MAR15-----492.d
Method DEF_MS.M
Sample Name Default

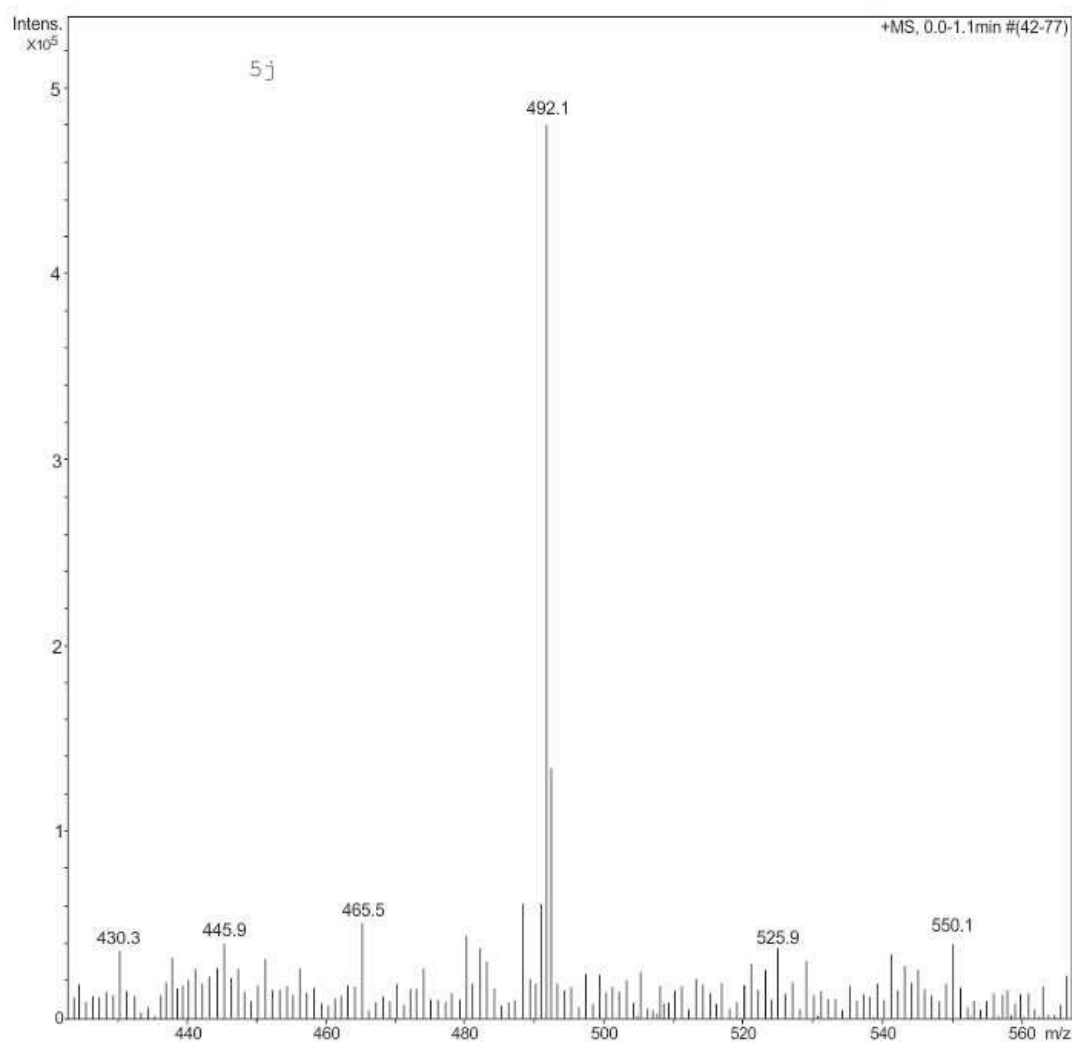
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Operator MBU
Instrument HCT ultra ETD II

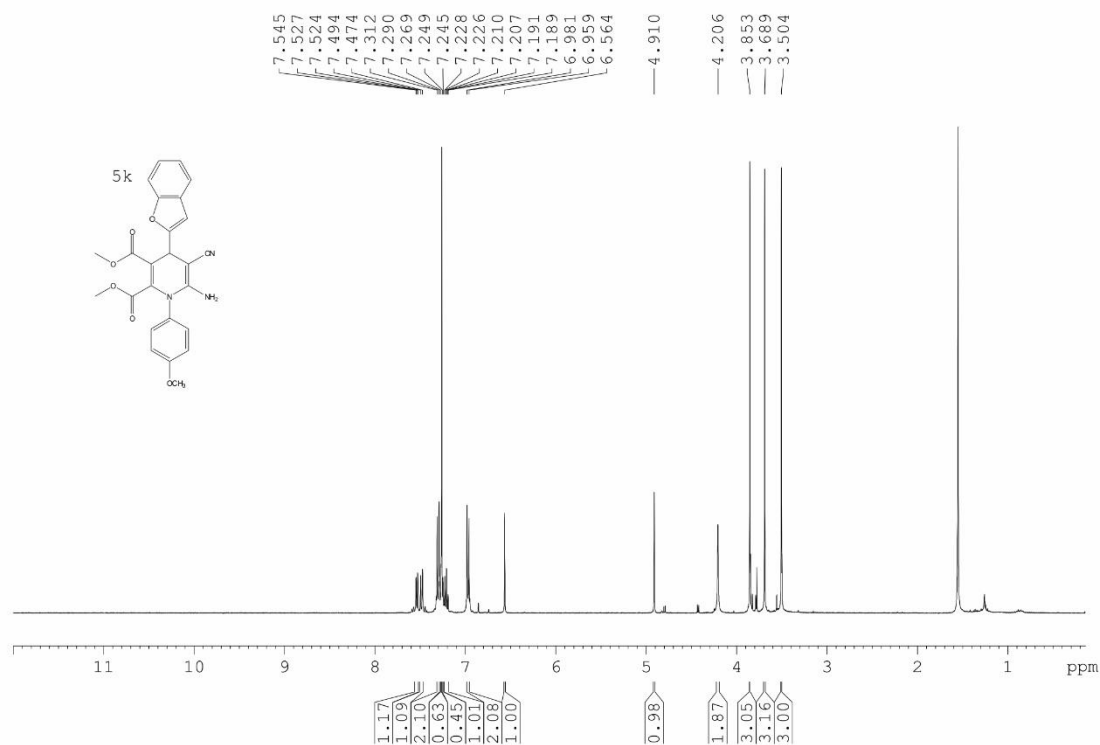
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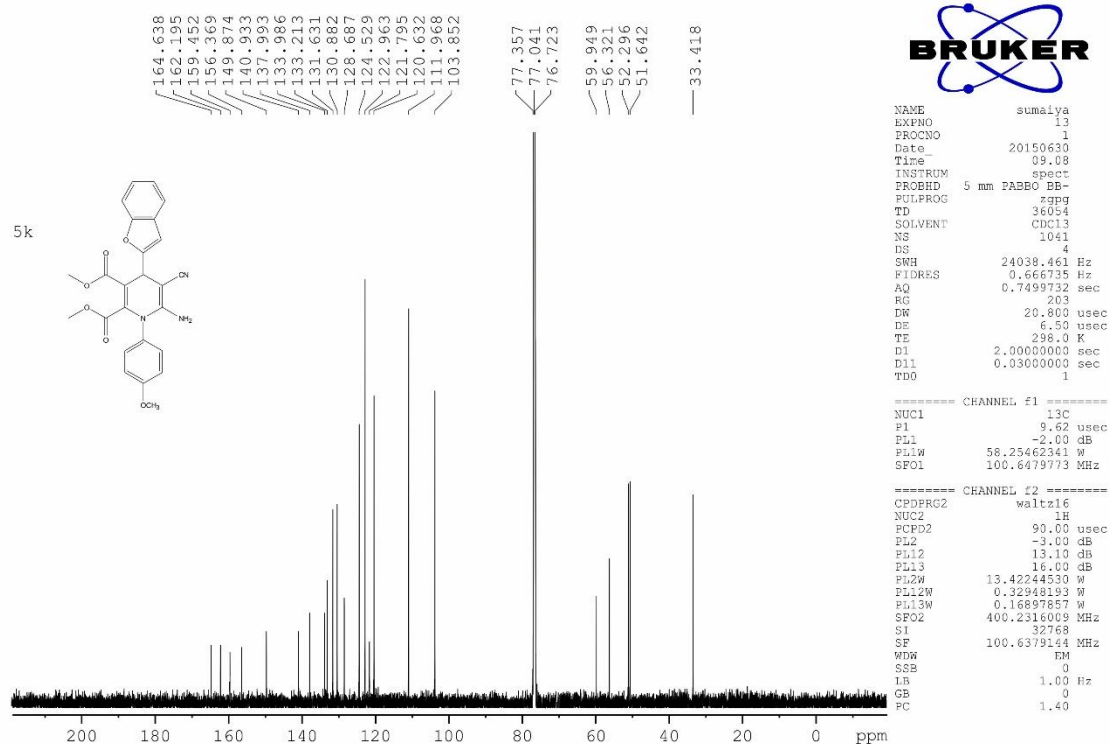
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Auto MS/MS	off



¹H NMR spectrum of 5k



¹³C NMR spectrum of 5k



ESI-MS of 5k

Display Report

Analysis Info

Analysis Name F:\Reports\GS\17MAR15-----460.d
Method DEF_MS.M
Sample Name Default

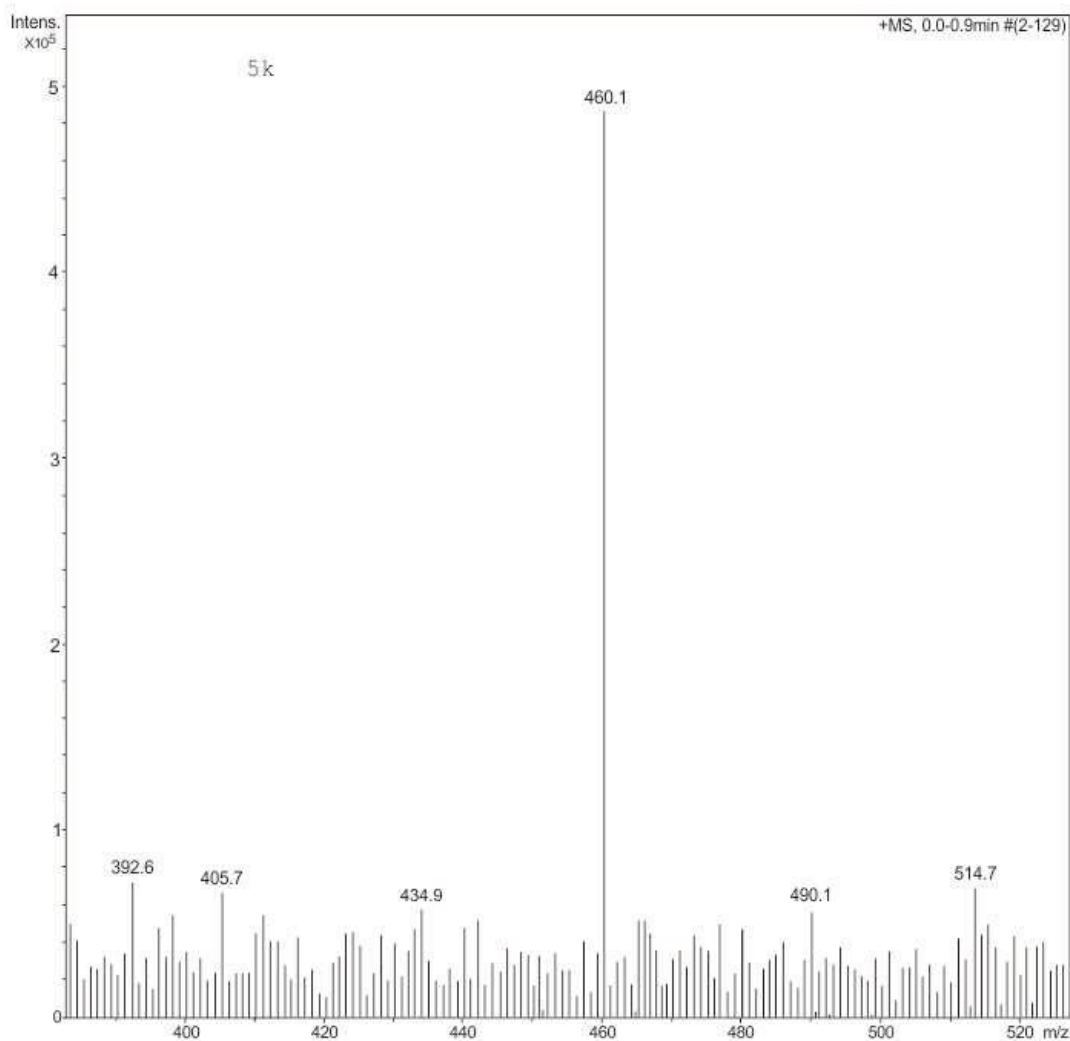
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Averages 5 Spectra

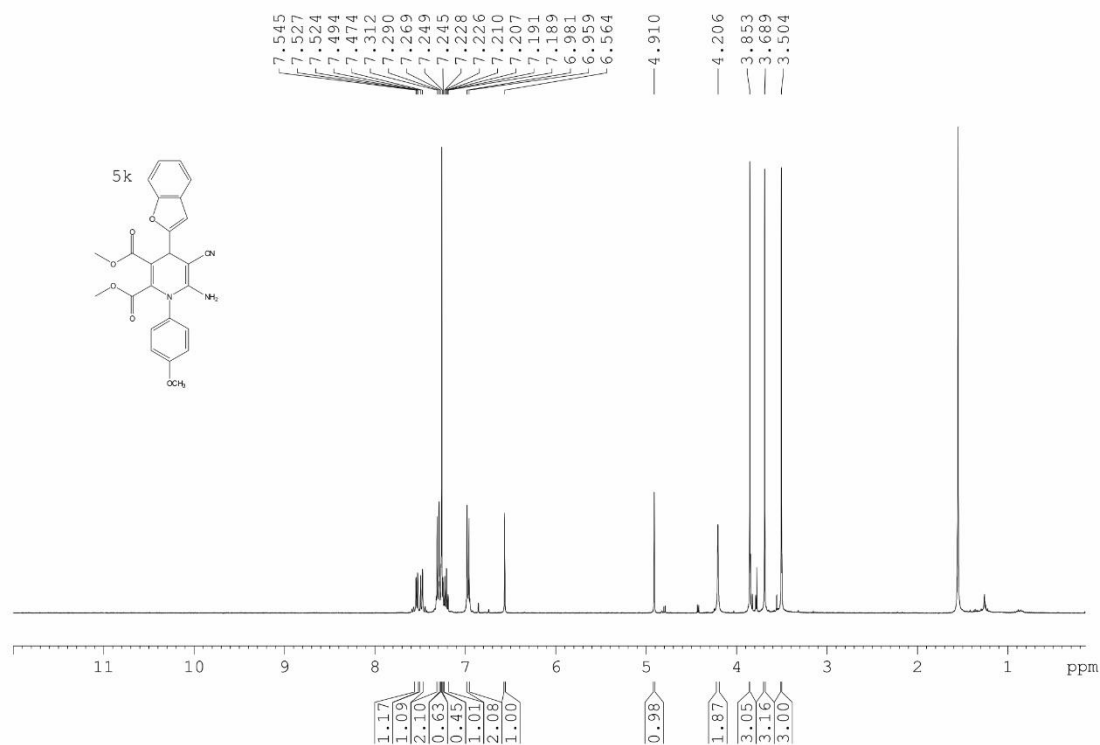
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Operator MBU
Instrument HCT ultra ETD II

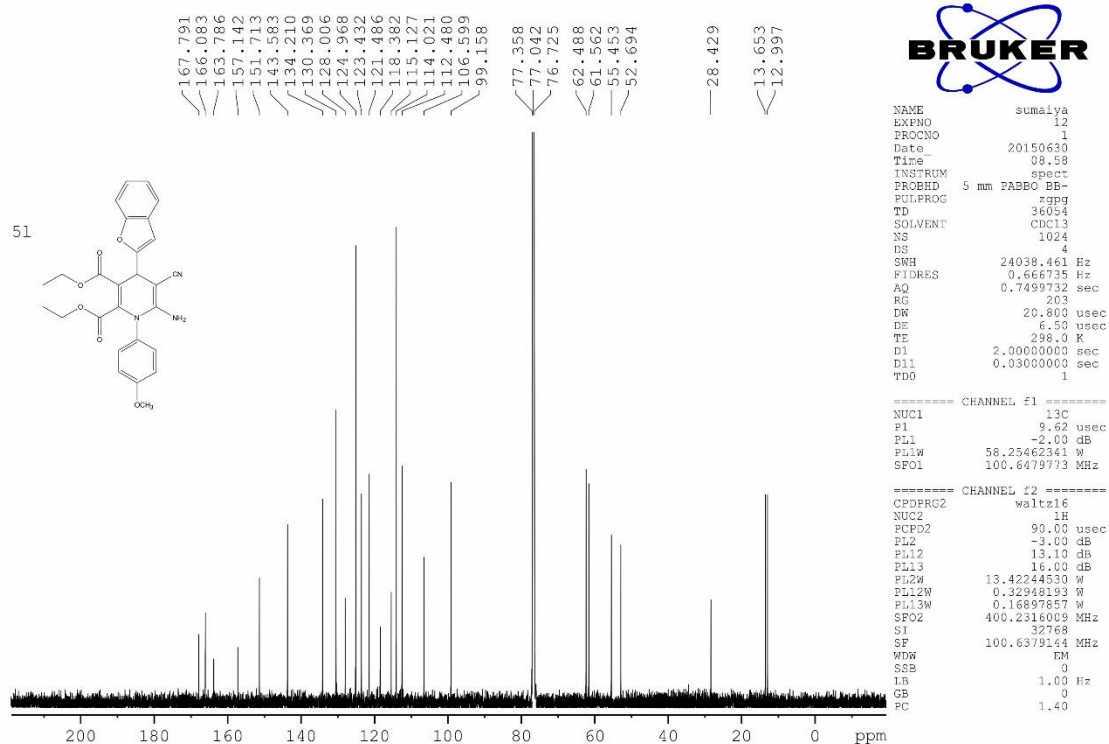
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Auto MS/MS off



¹H NMR spectrum of 5l



¹³C NMR spectrum of 5l



ESI-MS of 5l

Display Report

Analysis Info

Analysis Name F:\Reports\GS\17MAR15-----488.d
Method DEF_MS.M
Sample Name Default

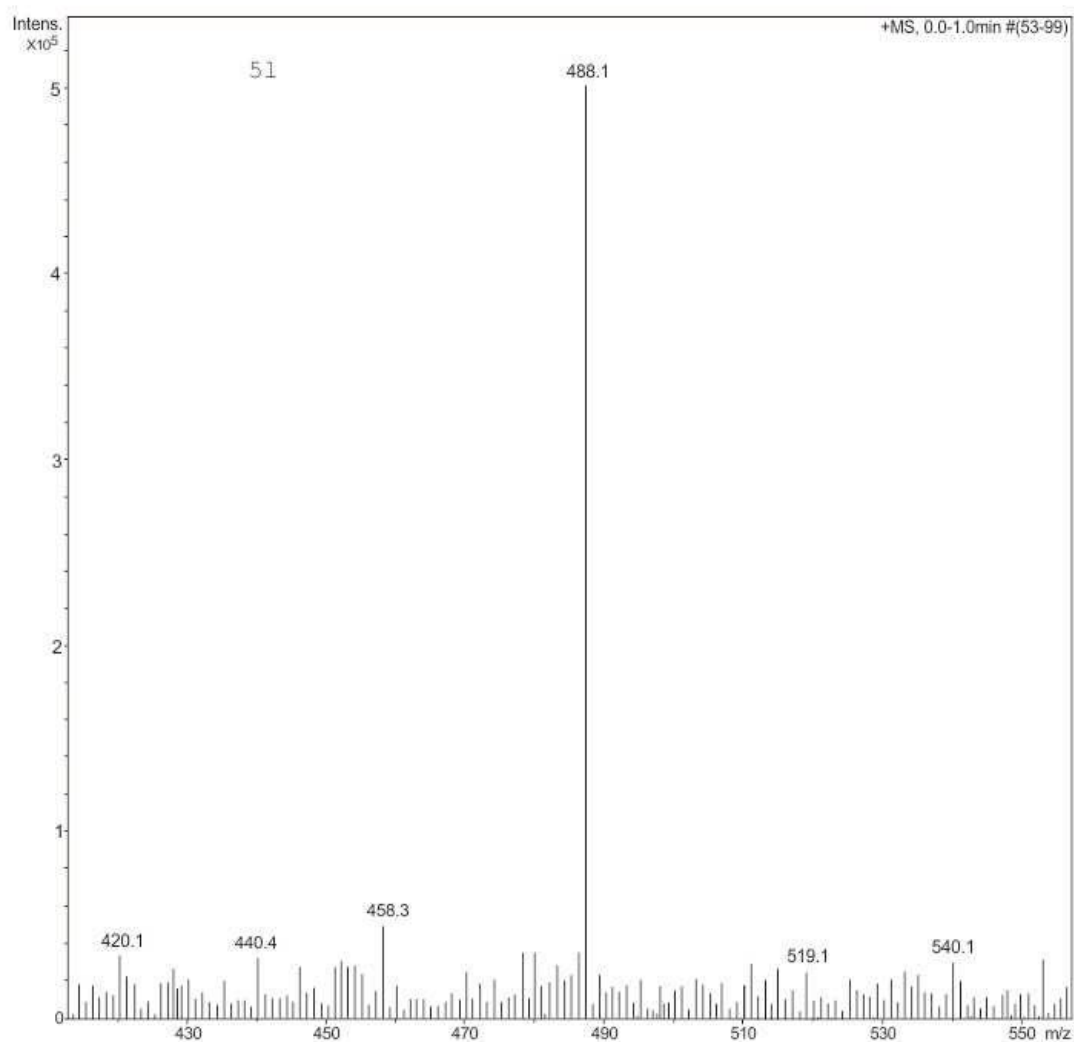
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Operator MBU
Instrument HCT ultra ETD II

Acquisition Parameter

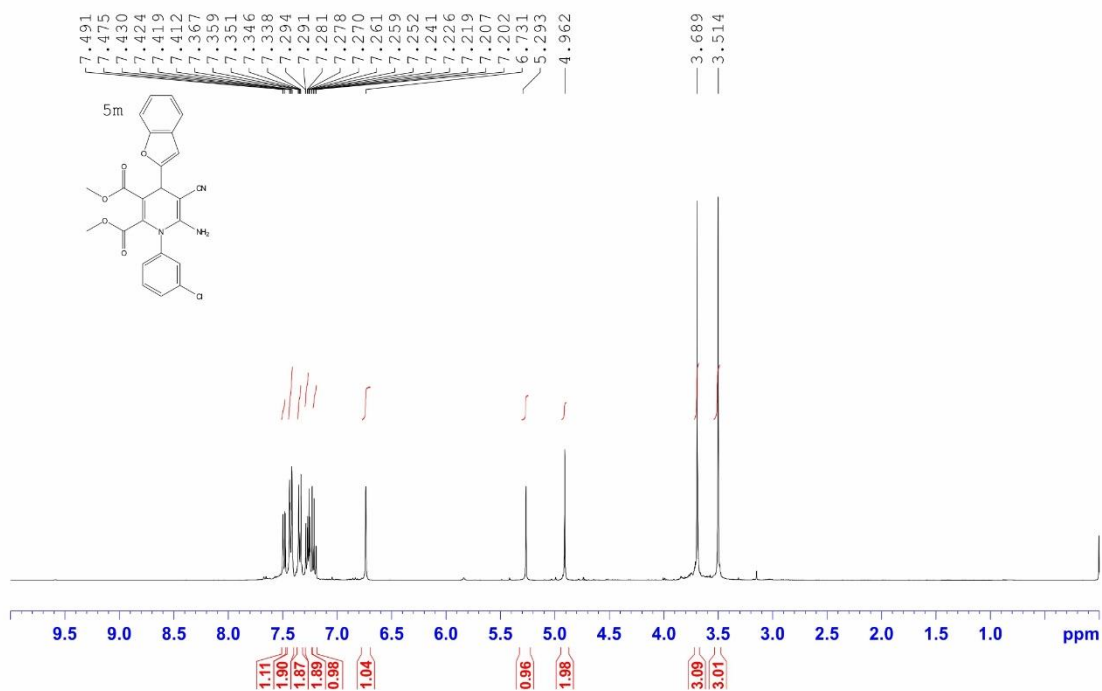
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Capillary Exit	104.5 Volt	Skimmer	40.0 Volt
Accumulation Time	Ultra Scan	Averages	5 Spectra

Alternating Ion Polarity	off
Scan End	800 m/z
Trap Drive	43.3
Auto MS/MS	off

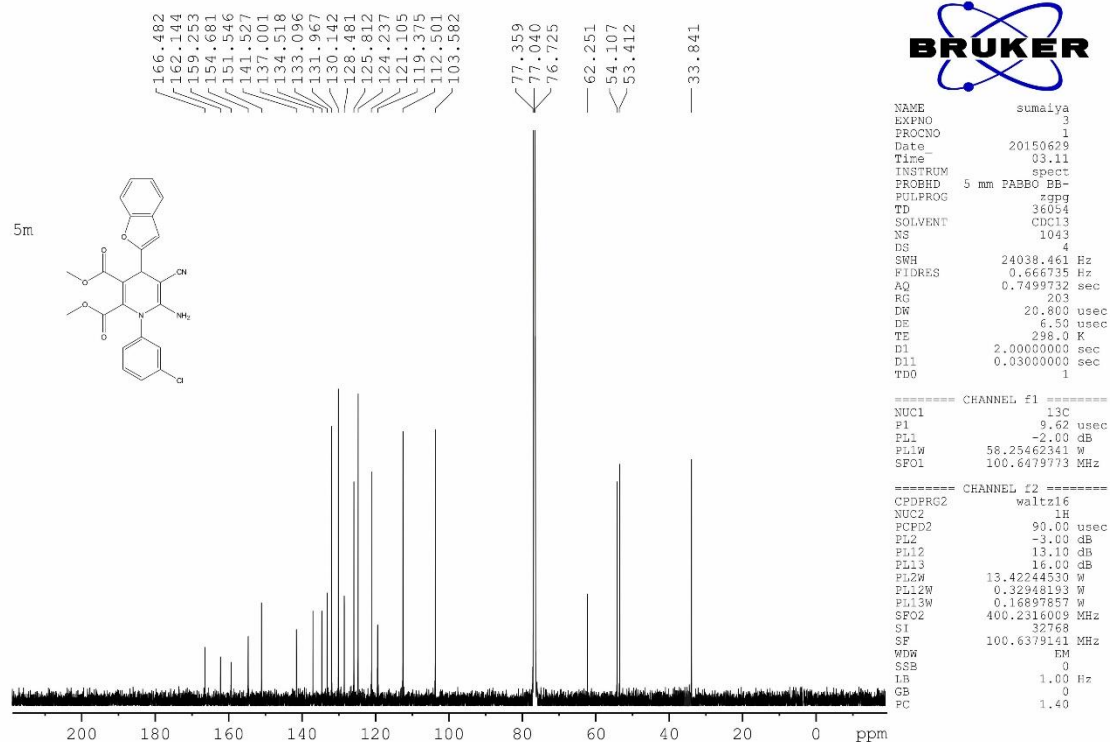


¹H NMR spectrum of 5m

Feb09-2015
PROTON CDCl₃



¹³C NMR spectrum of 5m



ESI-MS of 5m

Display Report

Analysis Info

Analysis Name F:\Reports\GS\17MAR15-----464.d

Method DEF_MS.M

Sample Name Default

Acquisition Parameter

Ion Source Type ESI

Mass Range Mode 3960 μ s

Capillary Exit 104.5 Volt

Accumulation Time Ultra Scan

Ion Polarity

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Skimmer

40.0 Volt

Averages

5 Spectra

Acquisition Date 17/3/2015 9:28:21 PM

Operator MBU

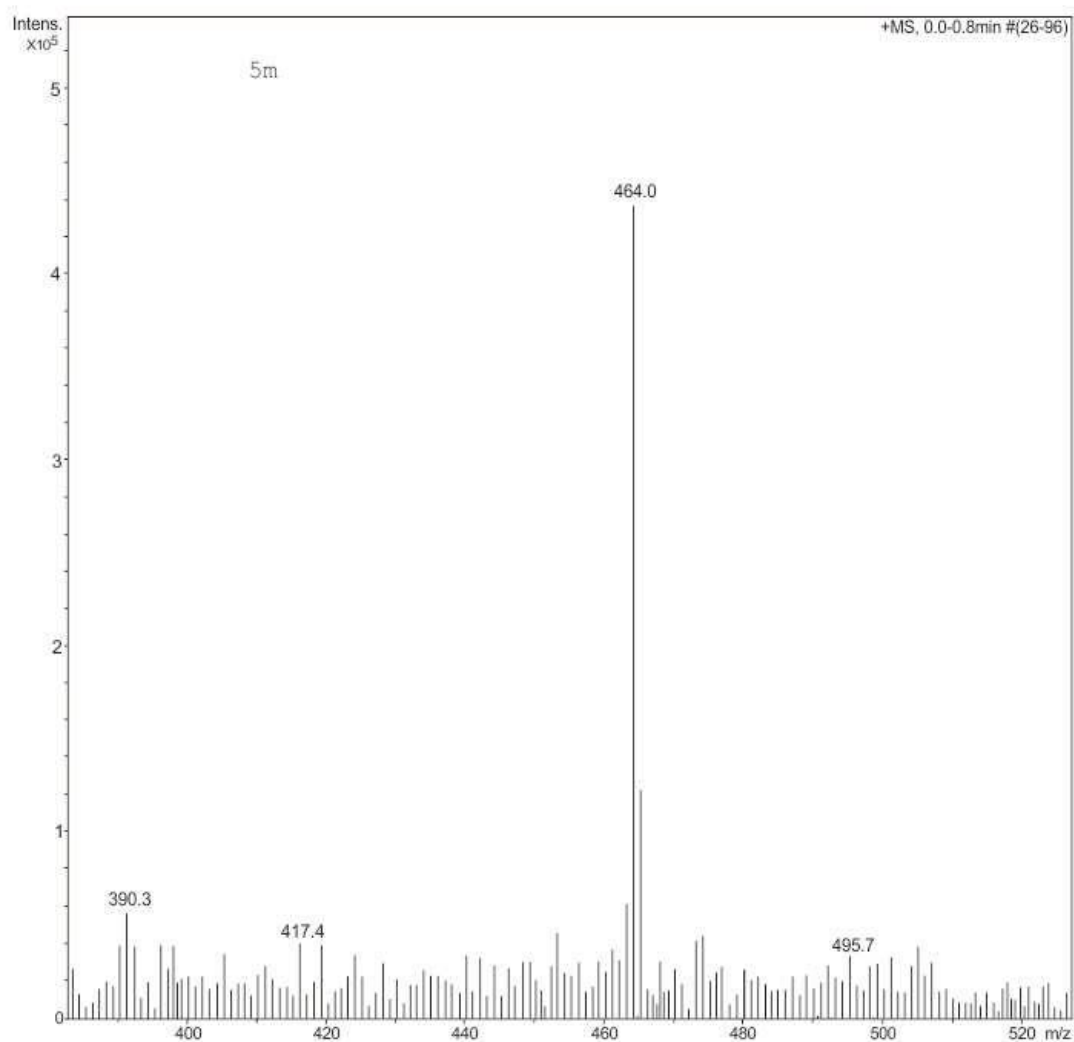
Instrument HCT ultra ETD II

Alternating Ion Polarity off

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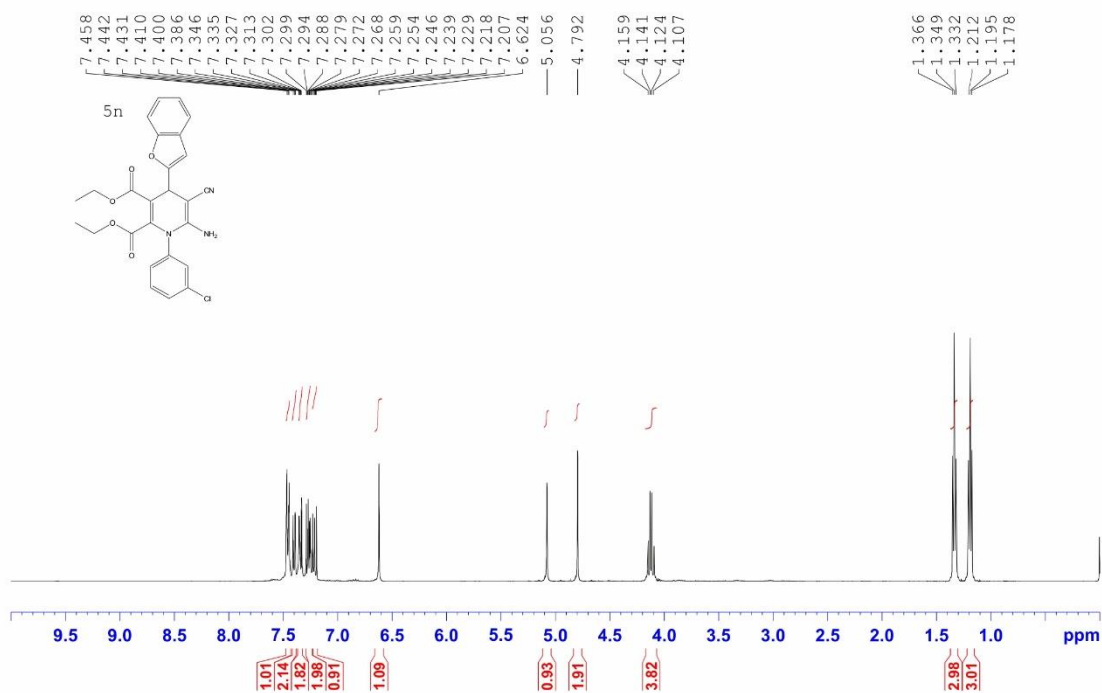
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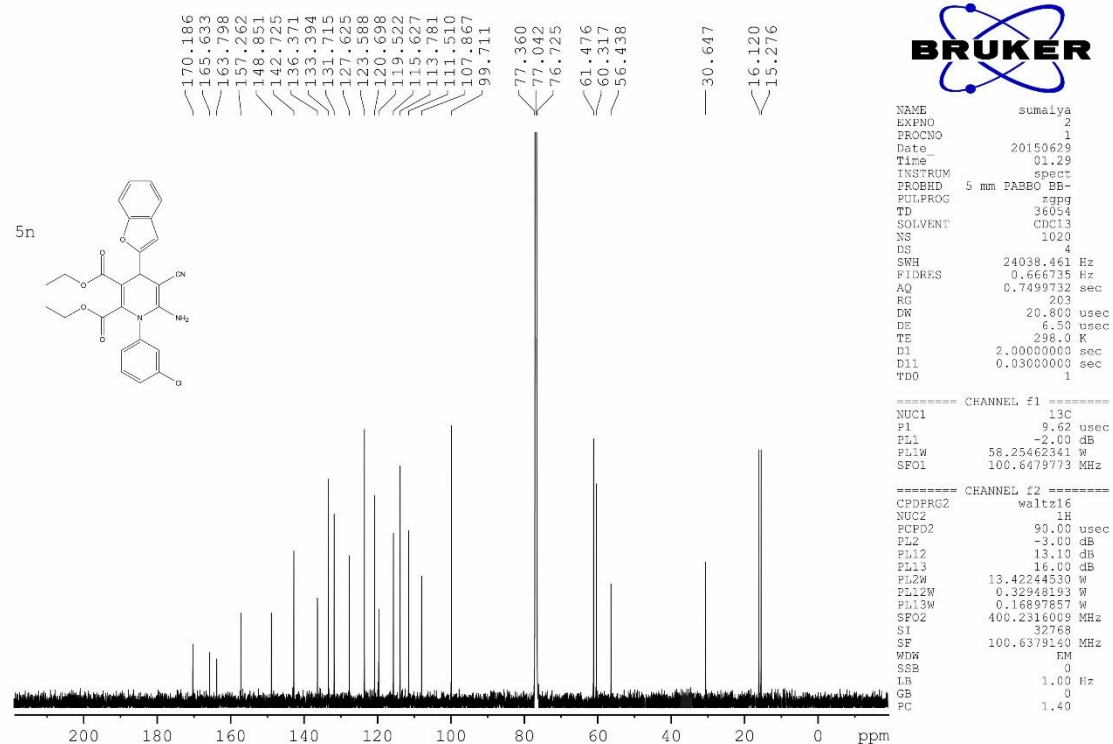


¹H NMR spectrum of 5n

Feb09-2015
PROTON CDC13



¹³C NMR spectrum of 5n



ESI-MS of 5n

Display Report

Analysis Info

Analysis Name F:\Reports\GS\17MAR15-----492.d
Method DEF_MS.M
Sample Name Default

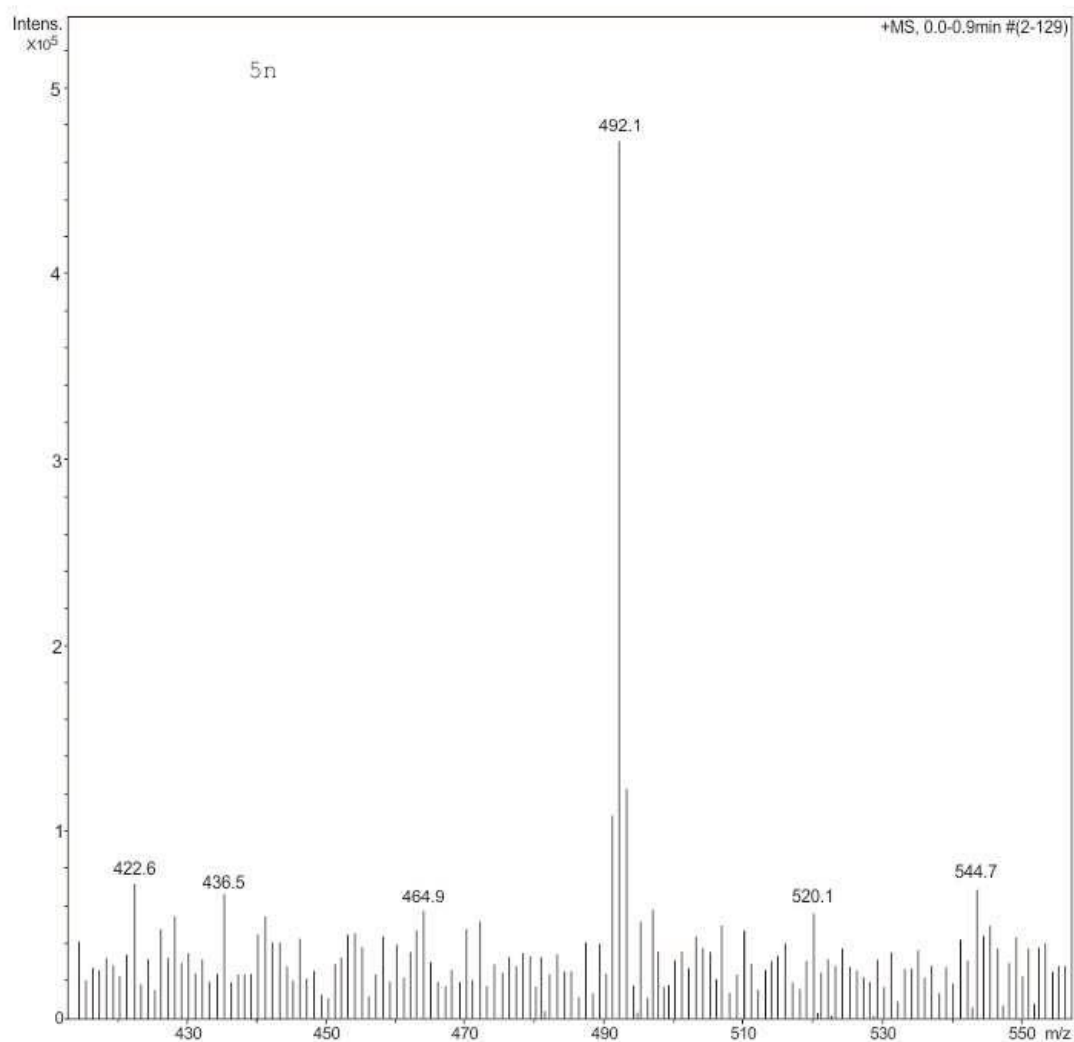
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Accumulation Time	Ultra Scan	Averages	5 Spectra

Acquisition Date 17/3/2015 8:04:42 PM

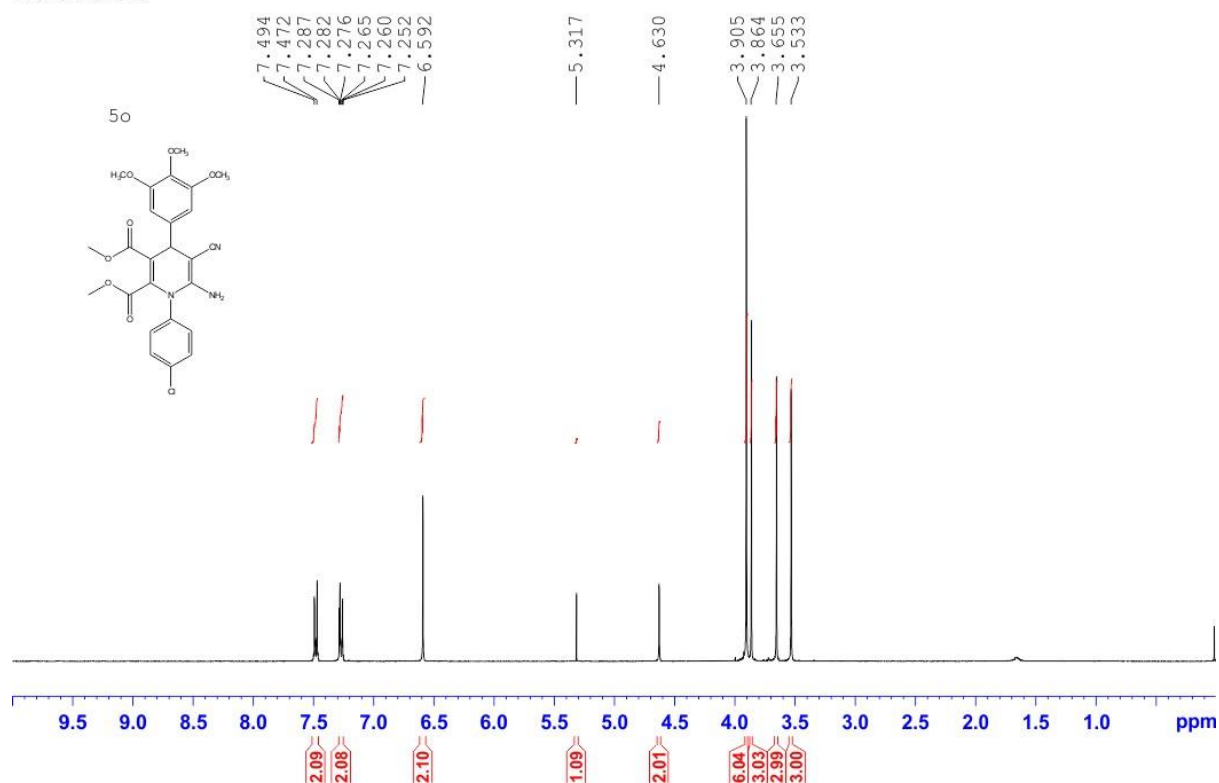
Operator MBU
Instrument HCT ultra ETD II

Alternating Ion Polarity	off
Scan End	900 m/z
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Auto MS/MS	off

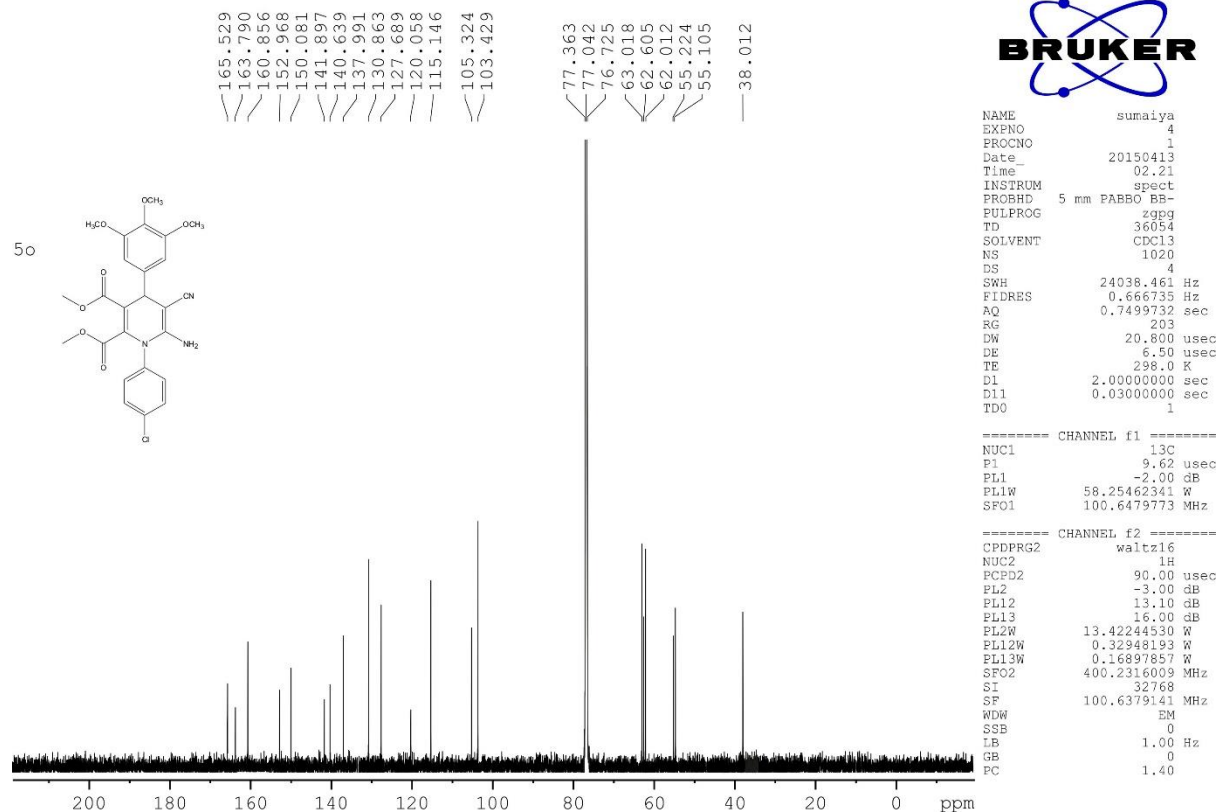


¹H NMR spectrum of 5n

Feb04-2015-SB-456
PROTON CDCl₃



¹³C NMR spectrum of 5n



```

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PROCNO    1
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RG         203
DW         20.800 usec
DE         6.50 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         9.62 usec
PL1        -2.00 dB
PL1W       58.25462341 W
SFO1       100.6479773 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      90.00 usec
PL2        -3.00 dB
PL12       13.10 dB
PL13       16.00 dB
PL2W       13.42244530 W
PL12W      0.32948193 W
PL13W      0.16897857 W
SFO2       400.2316009 MHz
SI         32768
SF         100.6379141 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
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ESI-MS of 5n

Display Report

Analysis Info

Analysis Name F:\Reports\GS\12FEB15-----513.d
Method DEF_MS.M
Sample Name Default

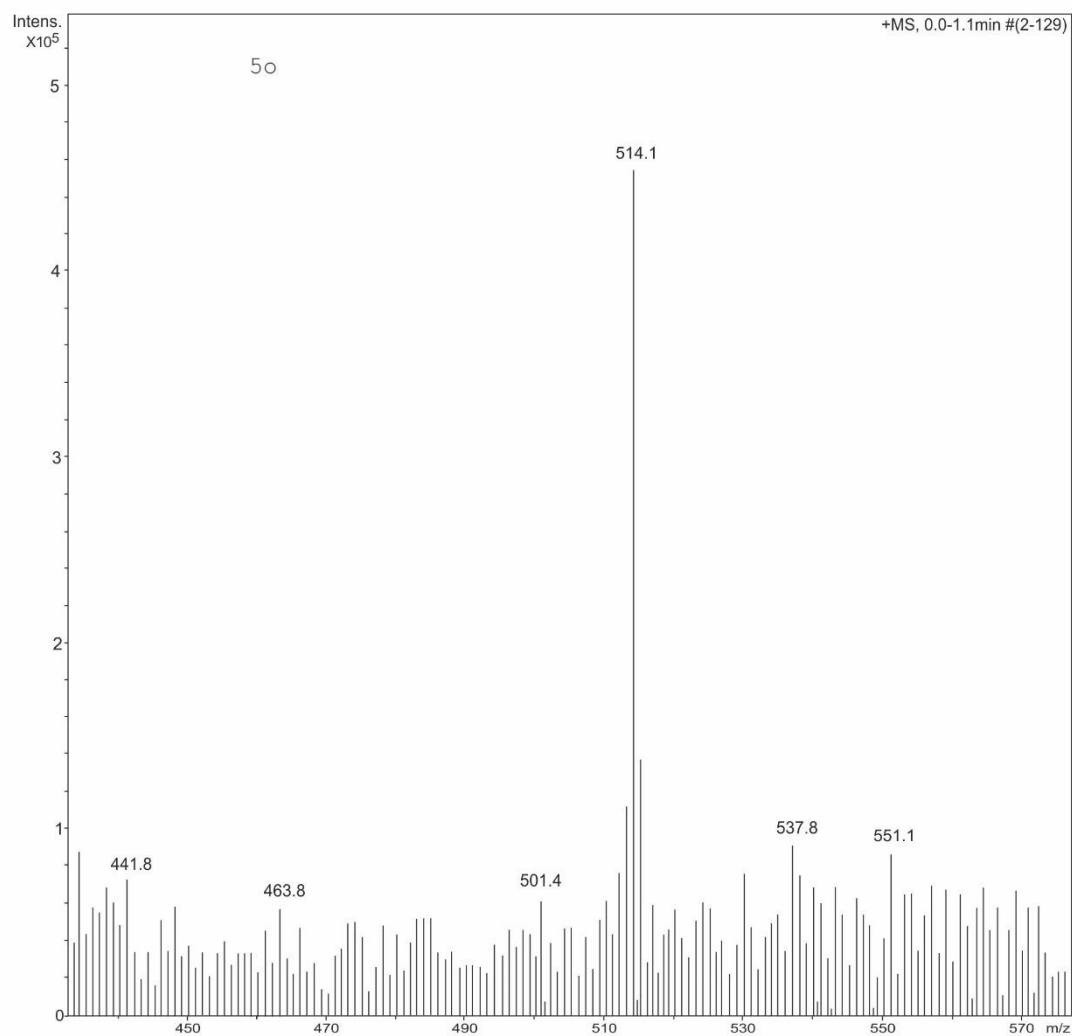
Acquisition Date 12/2/2015 5:30:41 PM

Operator MBU
Instrument HCT ultra ETD II

Acquisition Parameter

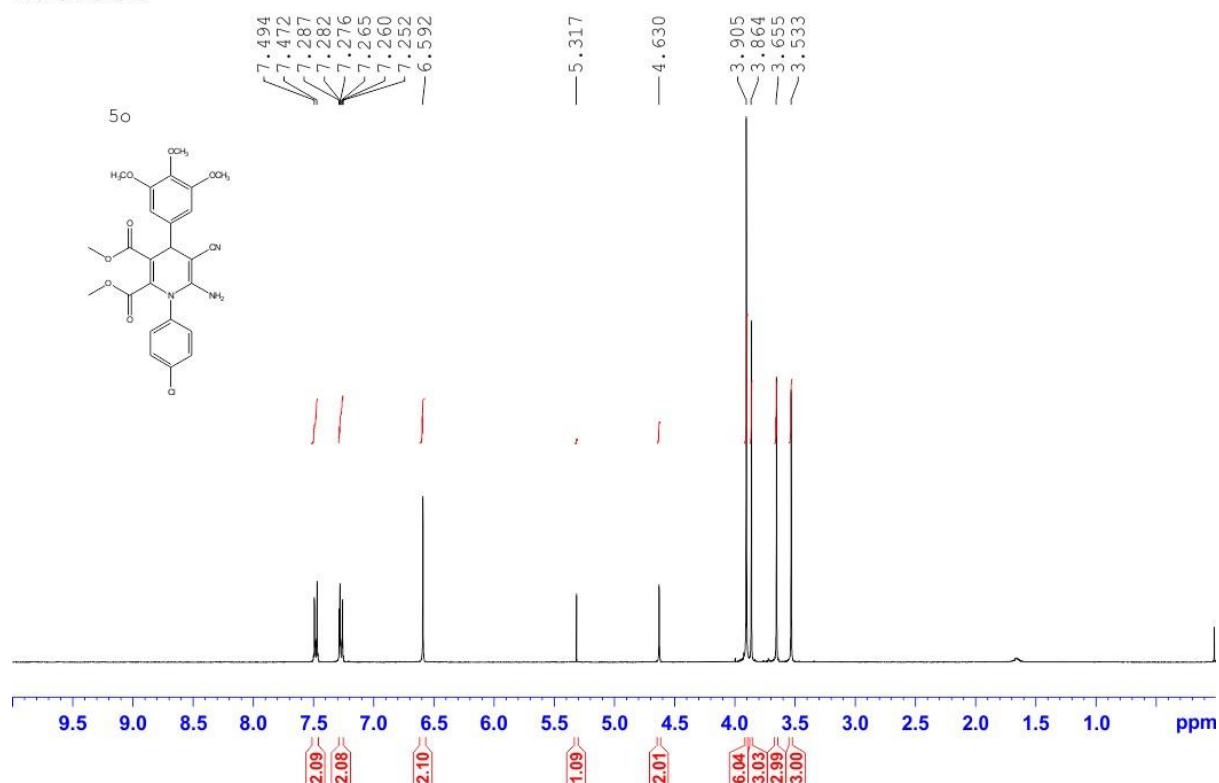
Ion Source Type	ESI	Ion Polarity	Positive
Mass Range Mode	3960 μ s	Scan Begin	100 m/z
Capillary Exit	104.5 Volt	Skimmer	40.0 Volt
Accumulation Time	Ultra Scan	Averages	5 Spectra

Alternating Ion Polarity	off
Scan End	900 m/z
Trap Drive	43.3
Auto MS/MS	off

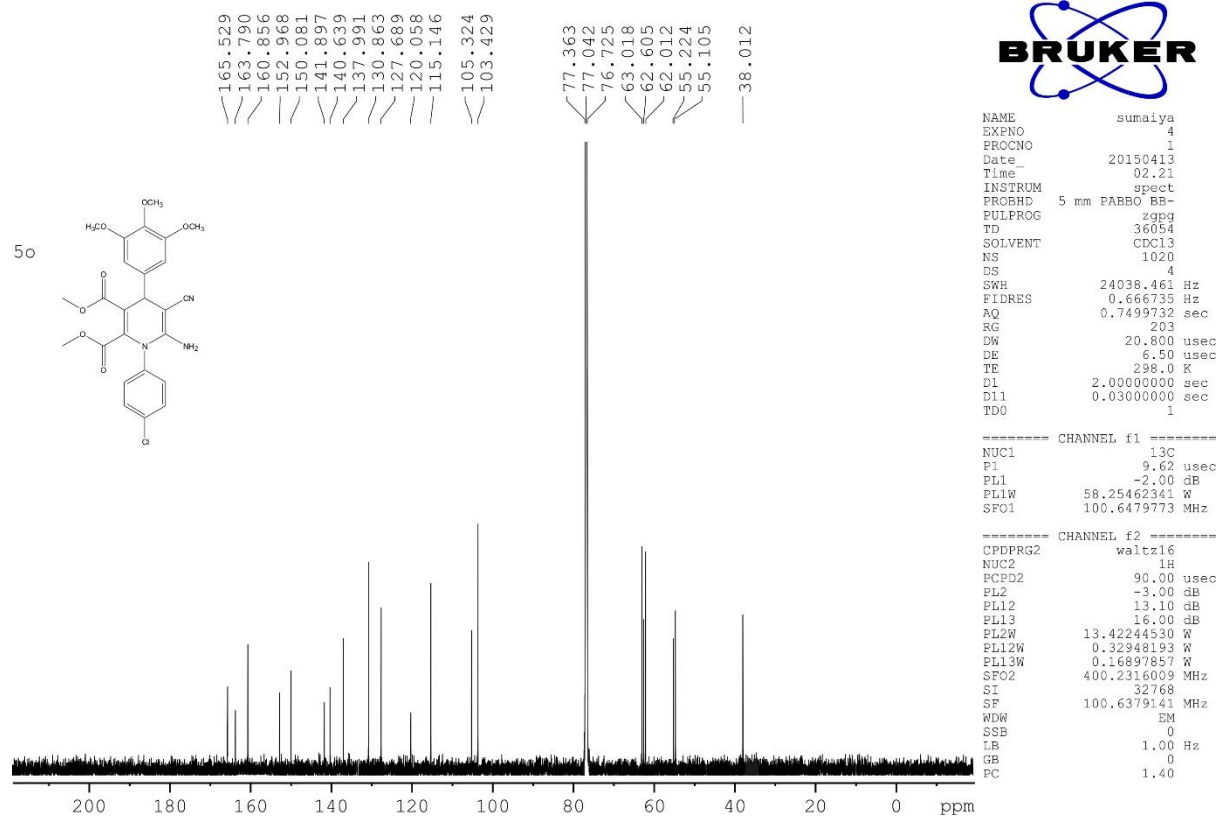


¹H NMR spectrum of 5o

Feb04-2015-SB-456
PROTON CDCl3



¹³C NMR spectrum of 5o



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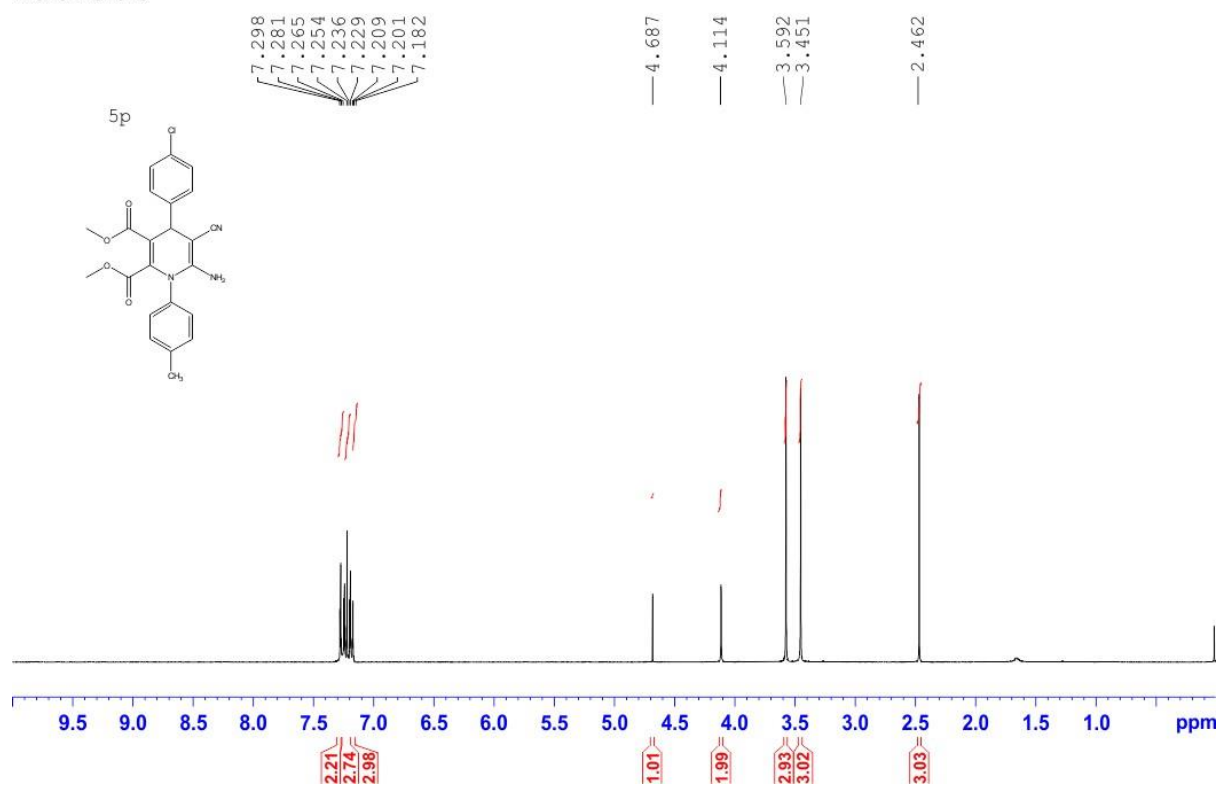
NAME      sumaiya
EXPNO     4
PROCNO    1
Date_     20150413
Time      02.21
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg
TD         36054
SOLVENT   CDCl3
NS         1020
DS         4
SWH        24038.461 Hz
FIDRES     0.666735 Hz
AQ         0.7499732 sec
RG         203
DW         20.800 usec
DE         6.50 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         9.62 usec
PL1        -2.00 dB
PL1W       58.25462341 W
SFO1       100.6479773 MHz

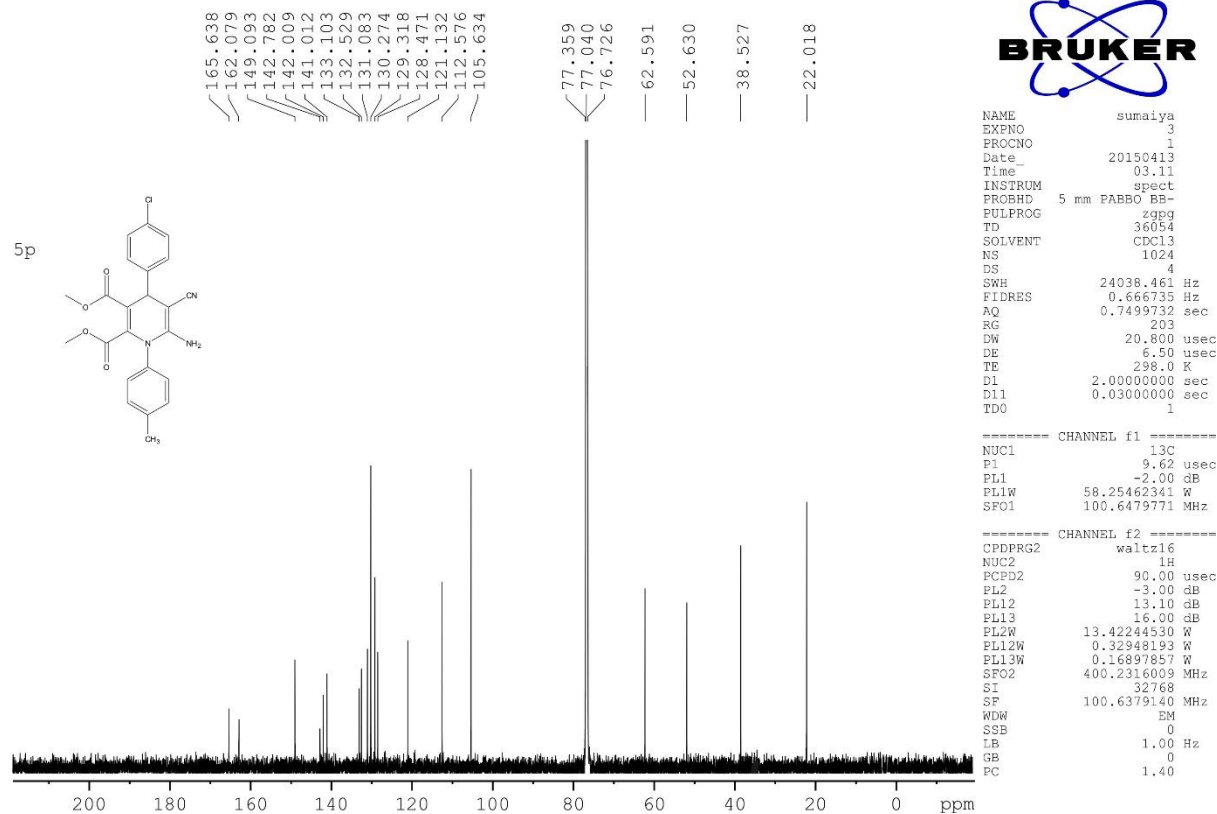
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      90.00 usec
PL2        -3.00 dB
PL12       13.10 dB
PL13       16.00 dB
PL2W       13.42244530 W
PL12W      0.32948193 W
PL13W      0.16897857 W
SFO2       400.2316009 MHz
SI         32768
SF         100.6379141 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
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¹H NMR spectrum of 5p

Feb09-2015-SB-457
PROTON CDC13

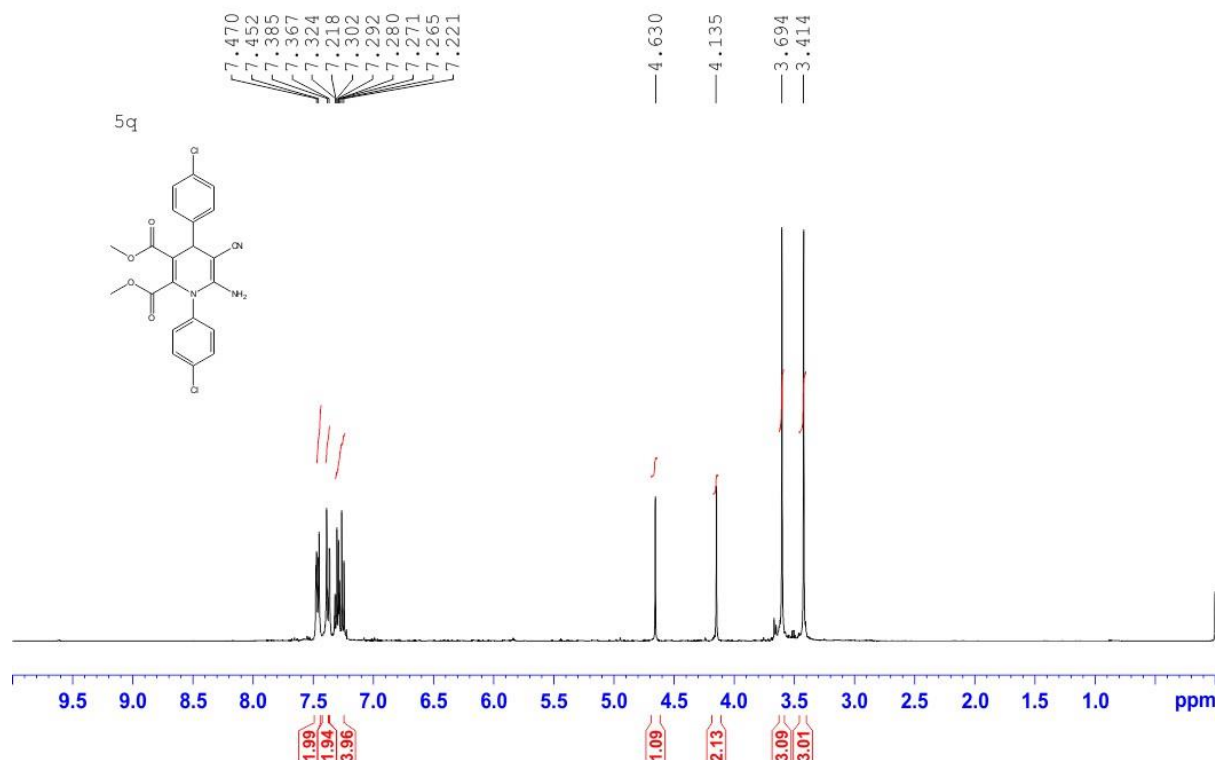


¹³C NMR spectrum of 5p

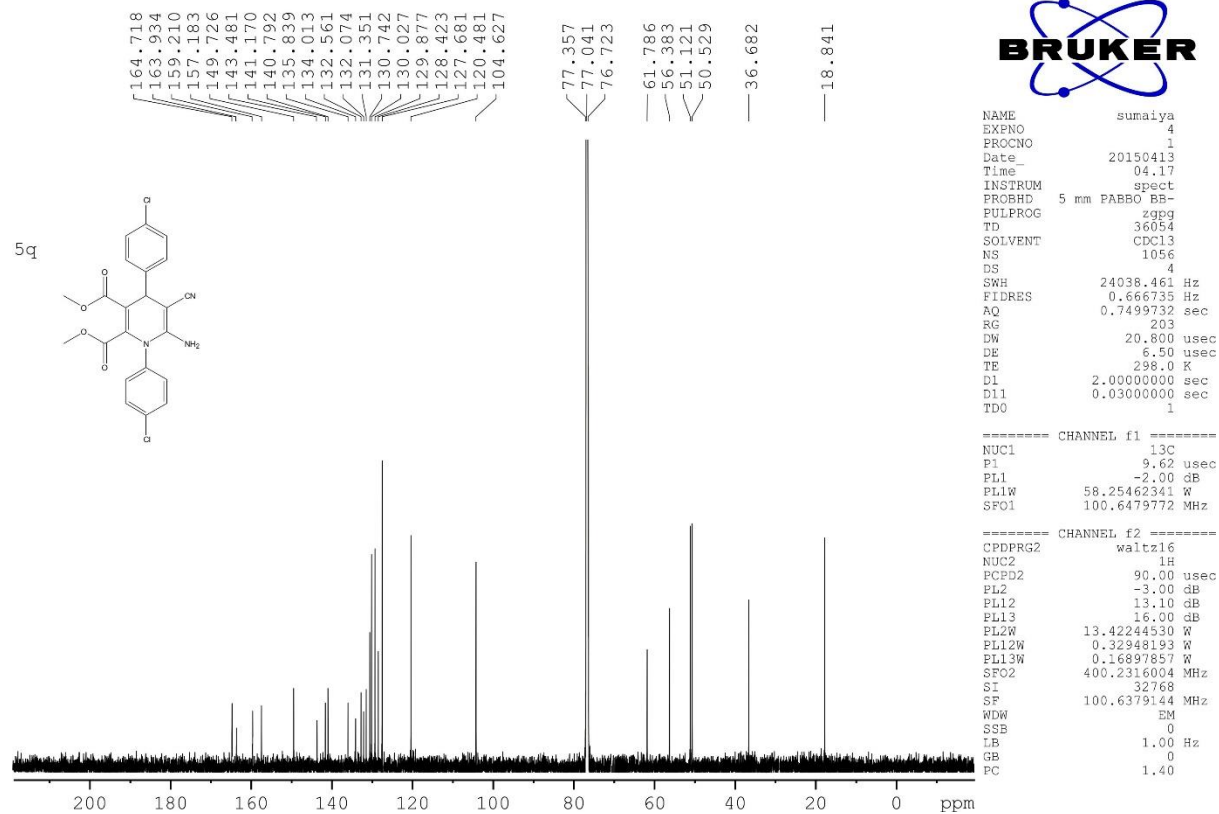


¹H NMR spectrum of 5q

Feb09-2015
PROTON CDCl3

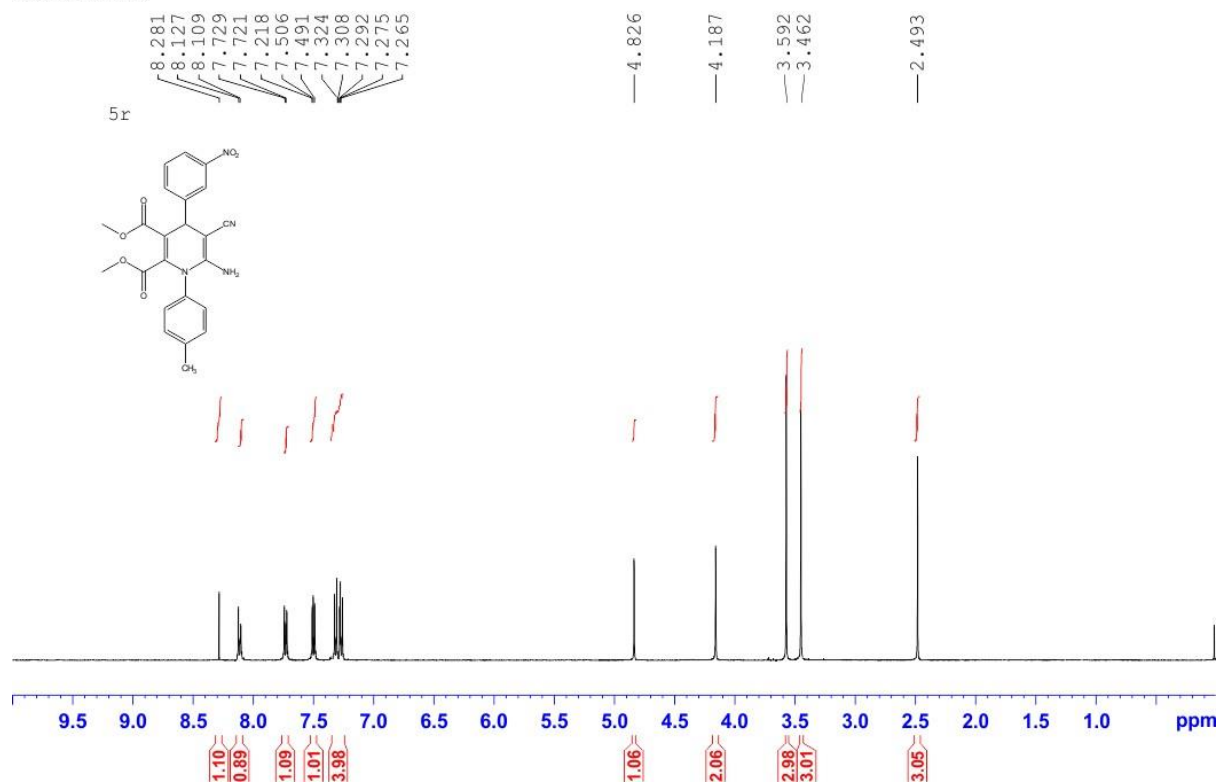


¹³C NMR spectrum of 5q

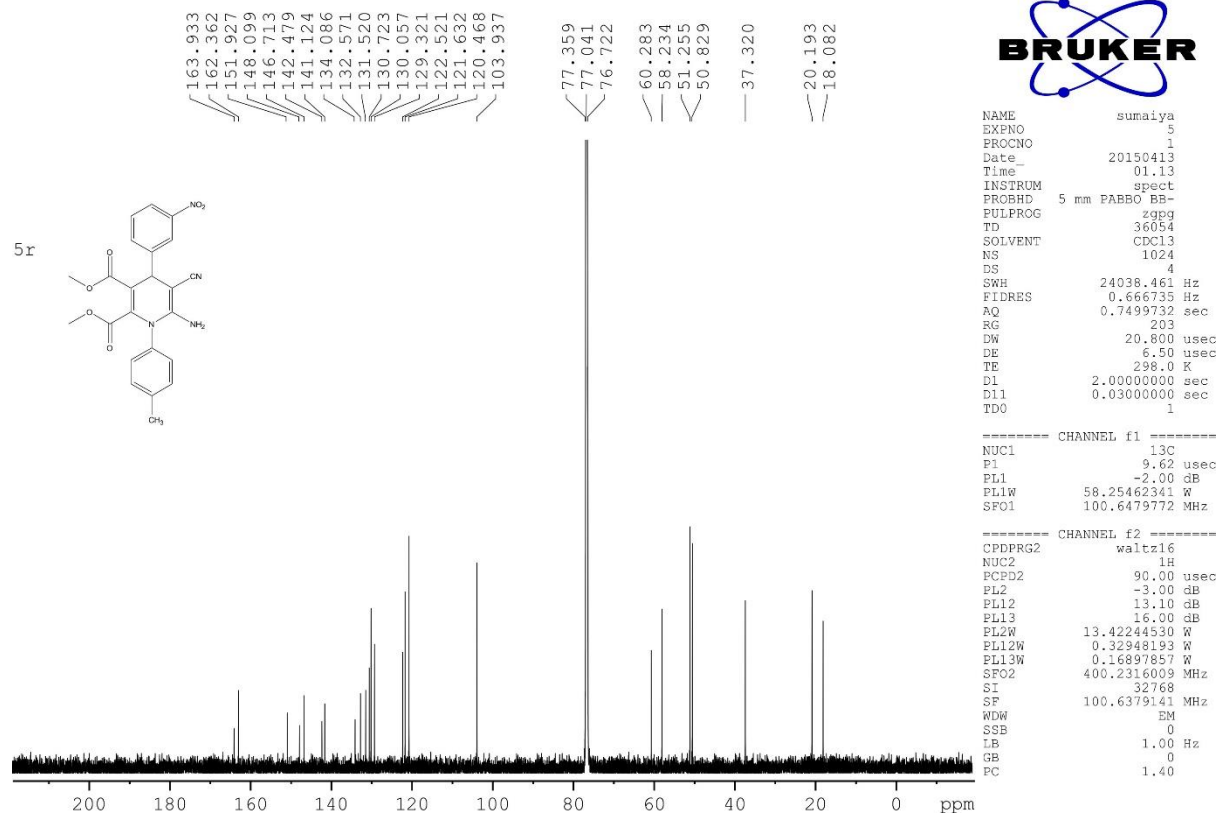


¹H NMR spectrum of 5r

Feb09-2015-SB-550
PROTON CDCl₃



¹³C NMR spectrum of 5r



CHN Analysis

Document: Untitled (VarioMICRO) from 28/08/14 (new)

Apotex labs

Vario MICRO CHNS

Serial Number: 15106090

Test Report

	Weight [mg]	Name	N [%]	C [%]	H [%]	C/H ratio	C/N ratio	Date	Time
1	1.0000	Blank	0.00	0.00	0.000	0.0000	0.0000	21.06.2015	08.36
2	1.0000	Blank	0.00	0.00	0.000	0.0000	0.0000	21.06.2015	08.59
3	1.0000	Blank	0.00	0.00	0.000	0.0000	0.0000	21.06.2015	09.12
4	1.0000	Blank	0.00	0.00	0.000	0.0000	0.0000	21.06.2015	09.34
5	3.1024	sulfanilamide	16.81	43.53	4.636	9.4015	2.5891	21.06.2015	10.01
6	3.5624	sulfanilamide	16.85	43.46	4.622	9.4015	2.5785	21.06.2015	10.37
7	2.2214	5a	9.74	67.18	4.41	15.2335	6.8973	21.06.2015	10.59
8	2.2578	5b	9.11	68.34	5.02	13.6135	7.5016	21.06.2015	11.17
9	3.1690	5c	9.03	62.19	3.98	15.6256	6.8870	21.06.2015	11.52
10	2.3688	5d	8.59	63.55	4.59	13.8453	7.3981	21.06.2015	12.37
11	3.1553	5e	9.42	67.79	4.85	13.9773	7.1963	21.06.2015	12.57
12	3.2142	5f	8.98	68.69	5.38	12.7676	7.6492	21.06.2015	13.15
13	2.1489	5g	11.88	60.84	3.89	15.6401	5.1212	21.06.2015	13.41
14	3.1689	5h	11.17	62.21	4.47	13.9172	5.5693	21.06.2015	14.11
15	3.2557	5i	9.02	62.19	3.98	15.6256	6.8946	21.06.2015	14.50
16	2.8943	5j	8.59	63.42	4.50	14.0933	7.3830	21.06.2015	15.15
17	3.0516	5k	9.17	65.21	4.66	13.9935	7.1112	21.06.2015	15.43
18	3.0325	5l	8.60	66.59	5.12	13.0058	7.7430	21.06.2015	16.12
19	3.1468	5m	9.01	62.11	3.97	15.6448	6.8934	21.06.2015	16.35
20	2.6791	5n	8.59	63.43	4.48	14.1584	7.3841	21.06.2015	17.24
44	2.4578	5o	8.29	58.31	4.80	12.1479	7.0337	21.06.2015	17.24

Name: Praveen.

Access: VarioMICRO Common

21/06/2015 18:25:19 PM

VarioMICRO V1.9.7.9 CHNS Mode. Ser. No. 15106090

Elementar Analysensysteme GmbH