

Supporting Information

Photoactive bimetallic framework for direct aminoformylation of nitroarenes

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1. Synthesis of g-C₃N₄ and AgPd@g-C₃N₄ catalyst

a) Synthesis of g-C₃N₄

b) Synthesis of AgPd@g-C₃N₄ catalyst

3. TEM image of g-C₃N₄ support (S1)

4. Recycling of AgPd@g-C₃N₄ catalyst (S2)

5. XPS spectra of recycled AgPd@g-C₃N₄ catalyst (S3)

6. XRD spectra of recycled AgPd@g-C₃N₄ catalyst (S4)

7. TEM spectra of recycled AgPd@g-C₃N₄ catalyst (S5)

8. GC-MASS data of the product

9. ¹H and ¹³C NMR of the product

1. Synthesis of g-C₃N₄ and AgPd@g-C₃N₄ catalyst

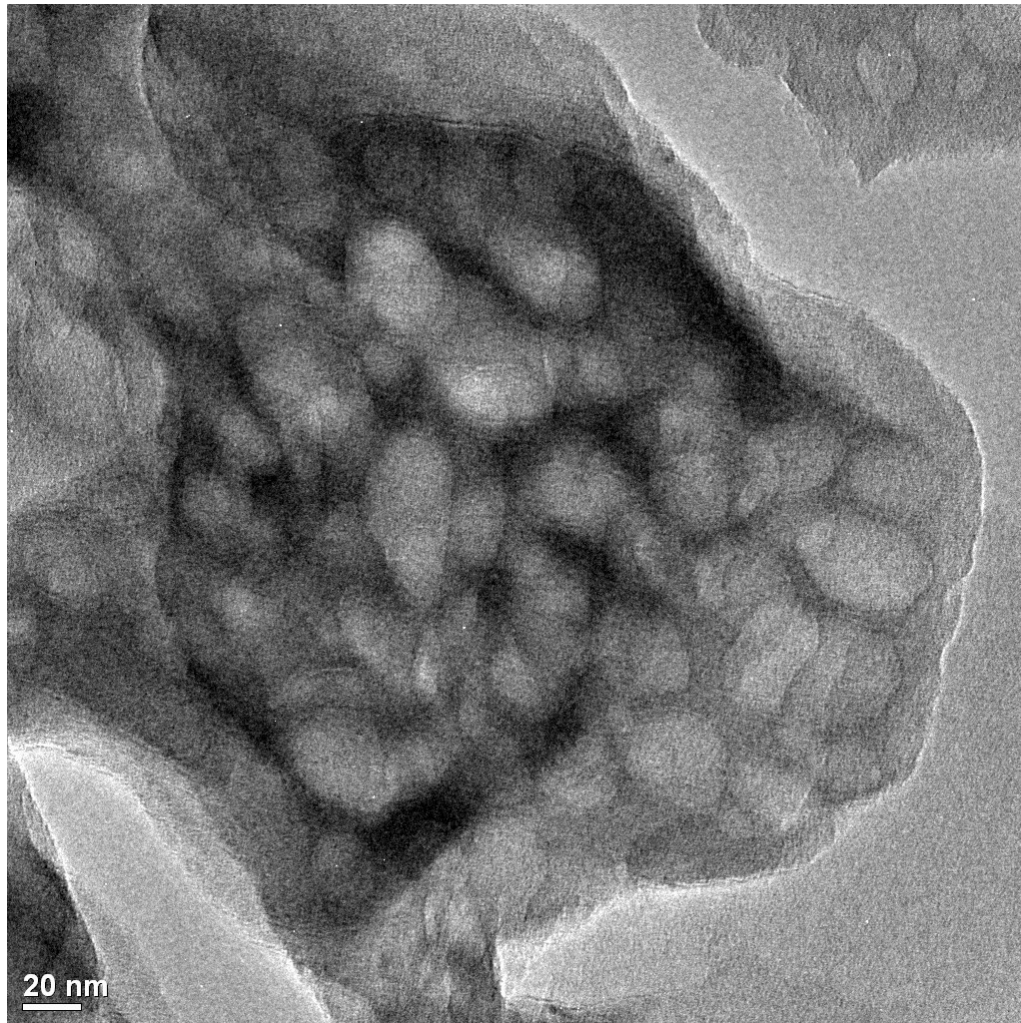
a) Synthesis of g-C₃N₄:

The pure urea obtained from Aldrich was calcinated at 500 °C in a closed furnace. A pale yellow solid of pure graphitic carbon nitride (g-C₃N₄) was obtained and used directly as a heterogeneous support in the synthesis of AgPd@g-C₃N₄ catalyst.

b) Synthesis of AgPd@g-C₃N₄ catalyst:

Graphitic carbon nitride, g-C₃N₄ (1000 mg) was dispersed in 500 mL of water using sonication. The aqueous solution of AgNO₃ was added in the aqueous suspension of g-C₃N₄. After 10 min, the aqueous solution of Pd(NO₃)₂ was added and the reaction mixture was stirred for 12 h. The reaction temperature was raised to 50 °C and excess of sodium borohydride (NaBH₄) was added in portions to reduce the Ag and Pd salts. The reaction mixture turned black after addition of NaBH₄. The stirring of reaction mixture was continued for another 12 hours. The catalyst was filtered off, washed with methanol and dried under vacuum at 50 °C. The isolated AgPd@g-C₃N₄ catalyst was then characterized using TEM, SEM, XRD, XPS and ICP-AES analysis.

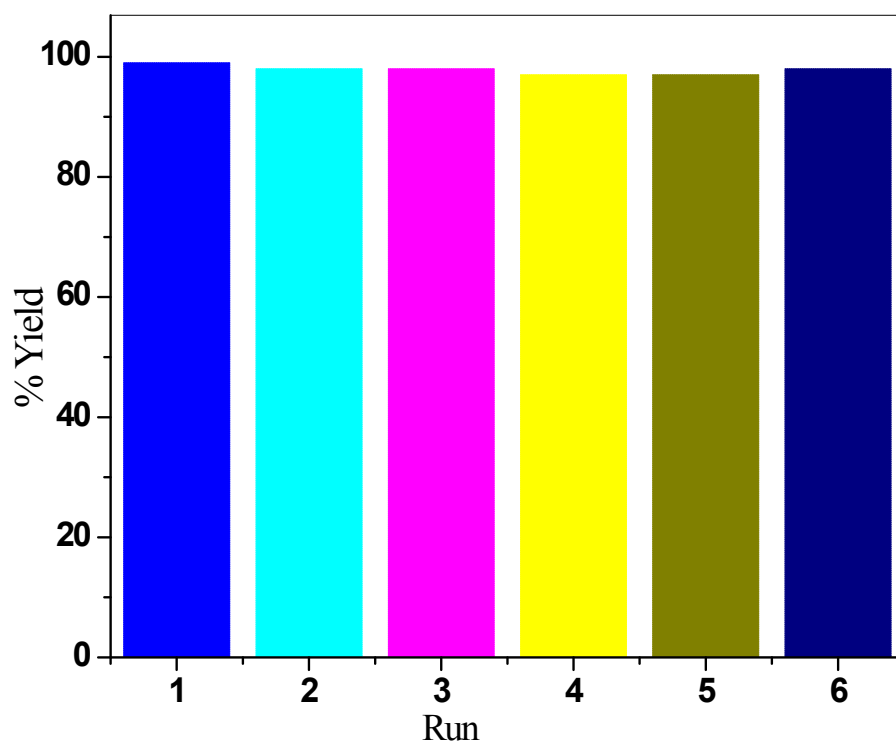
3. TEM image of g-C₃N₄ support



S1 TEM image of g-C₃N₄ support

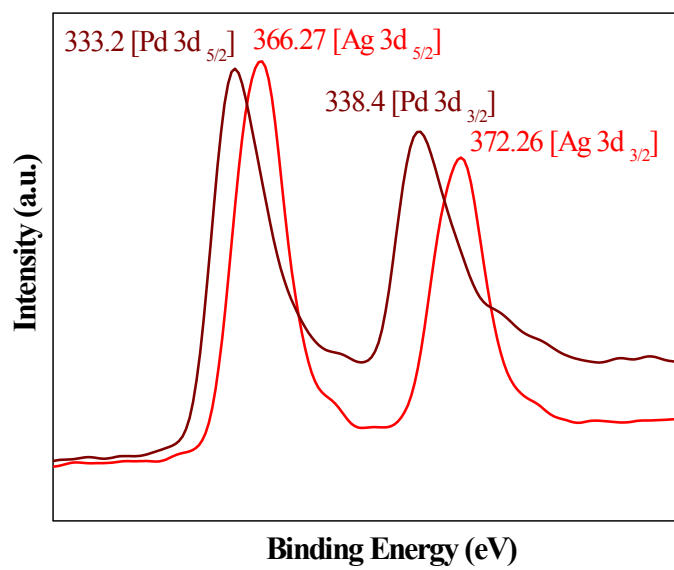
3. Recycling of catalyst:

After the completion of each reaction, the AgPd@g-C₃N₄ catalyst was recovered using centrifuge, washed with water followed by acetone: methanol (1:1) mixture and dried under vacuum and used for the fresh set of reactants. It was observed that the catalyst remains active even after sixth cycle and could be reused several times without losing activity.



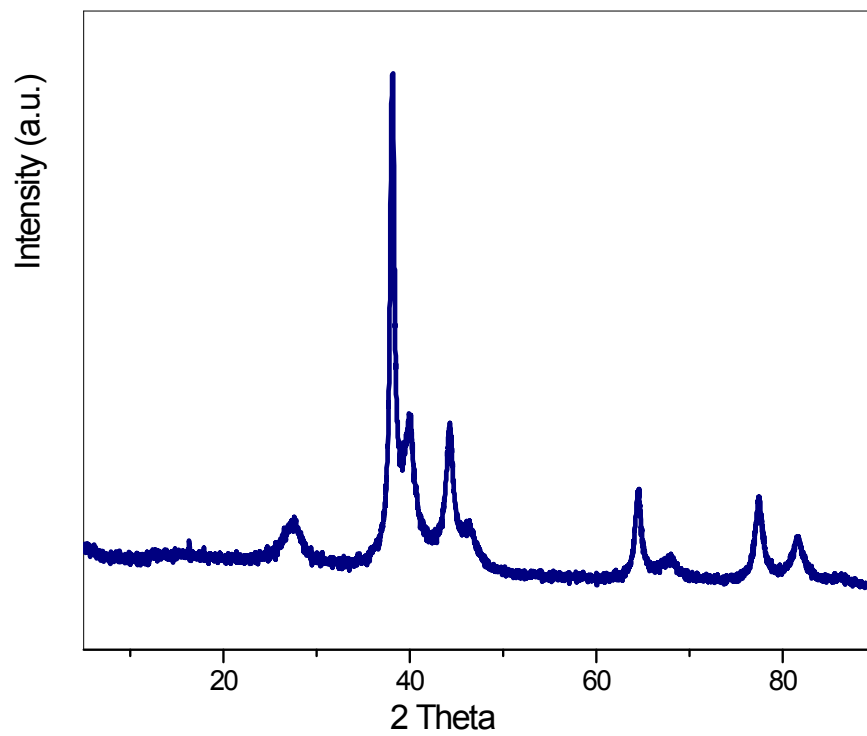
S2 Recycling of AgPd@g-C₃N₄ catalyst

5. XPS spectra of recycled AgPd@g-C₃N₄ catalyst



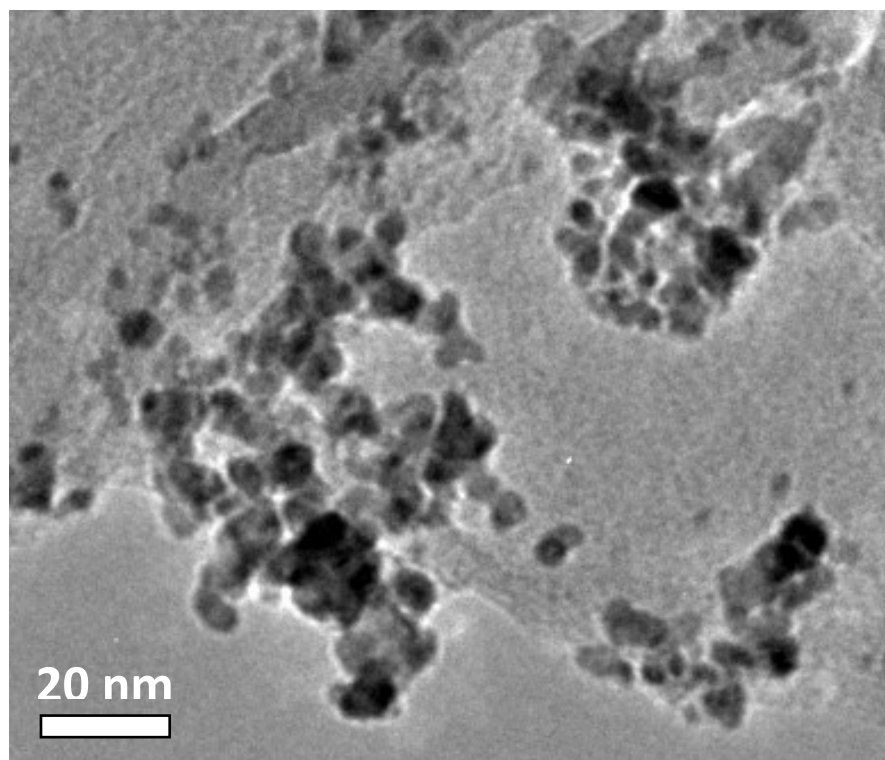
S3 XPS spectra of recycled AgPd@g-C₃N₄ catalyst

6. XRD spectra of recycled AgPd@g-C₃N₄ catalyst



S4 XRD spectra of recycled AgPd@g-C₃N₄ catalyst

7. TEM spectra of recycled AgPd@g-C₃N₄ catalyst

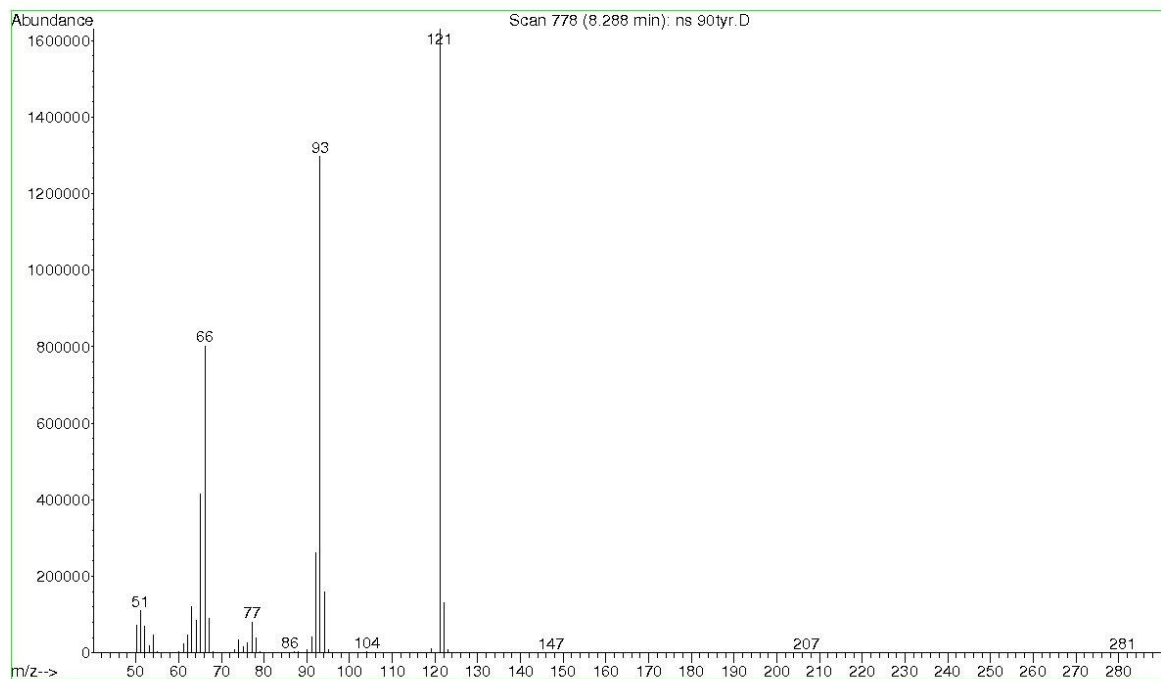
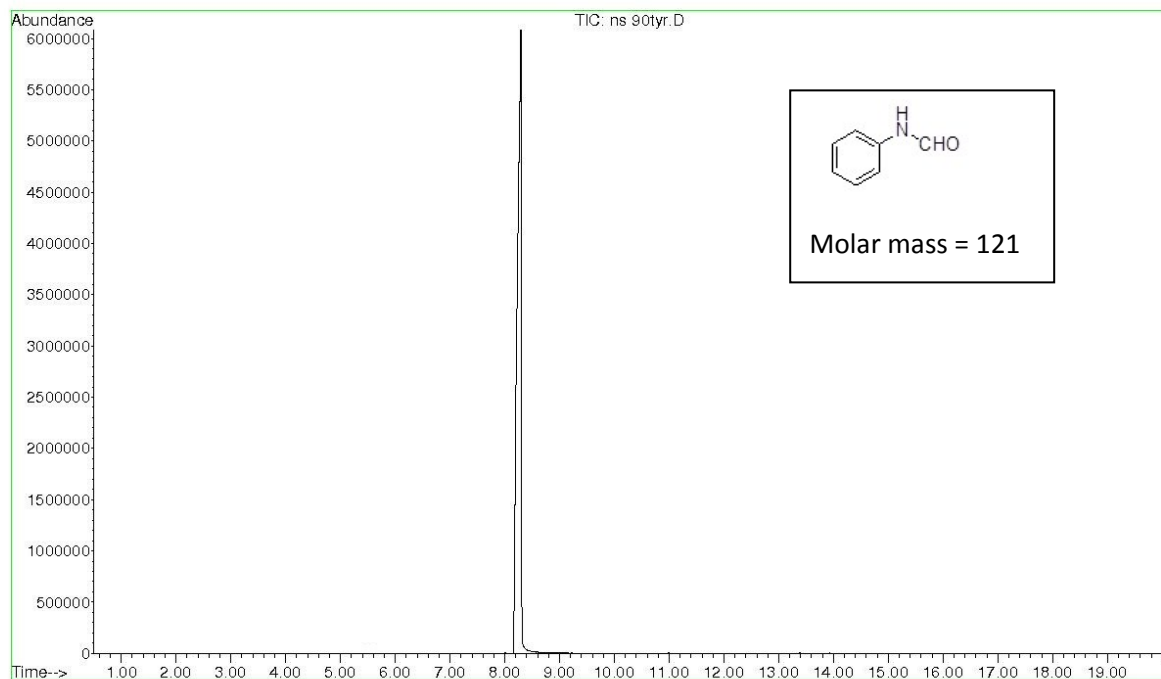


S5 TEM spectra of recycled AgPd@g-C₃N₄ catalyst

GC-MASS Spectra of the product

File :C:\Sanny\Data\ns 90tyr.D
Operator : kt
Acquired : 10 Nov 2015 12:25 pm using AcqMethod SAMPLES- 20 MIN_20151015_A.M
Instrument : Instrument #1
Sample Name: ns 90tyr
Misc Info :
Vial Number: 15

Table 2, Entry 1



File :C:\Sanny\Data\NS 139 RT.D
Operator : kt
Acquired : 12 Nov 2015 12:10 pm using AcqMethod SAMPLES- 20 MIN_20151015_A.M
Instrument : Instrument #1
Sample Name: NS 139 RT
Misc Info :
Vial Number: 16

Table 2, Entry 2

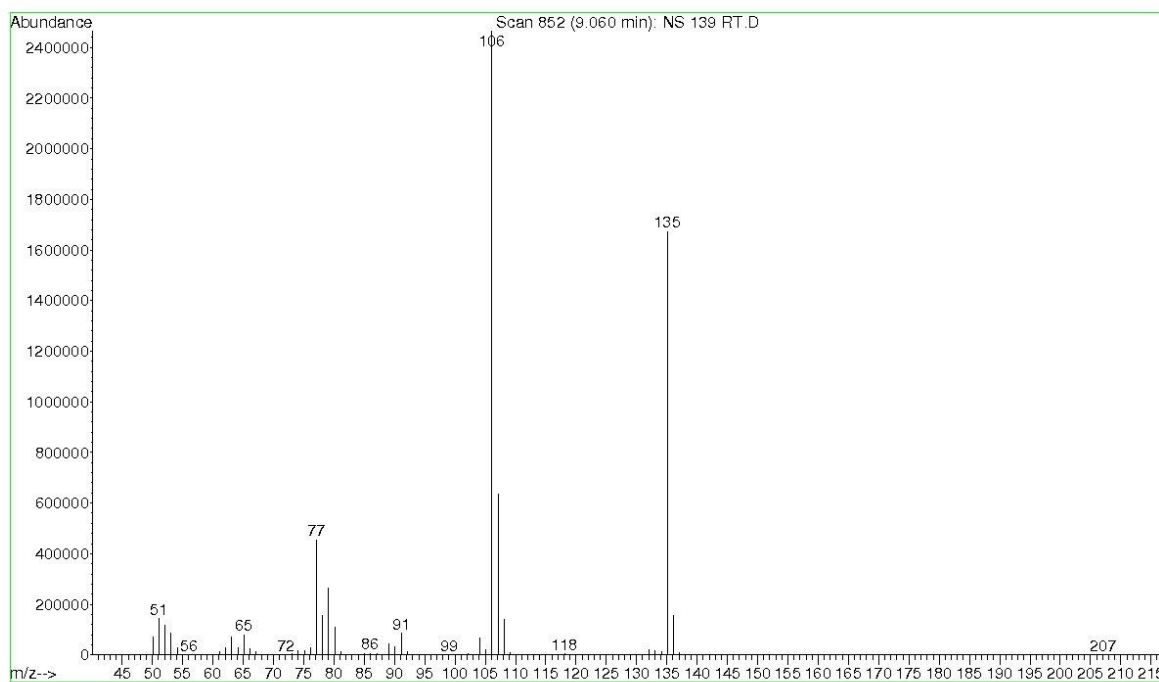
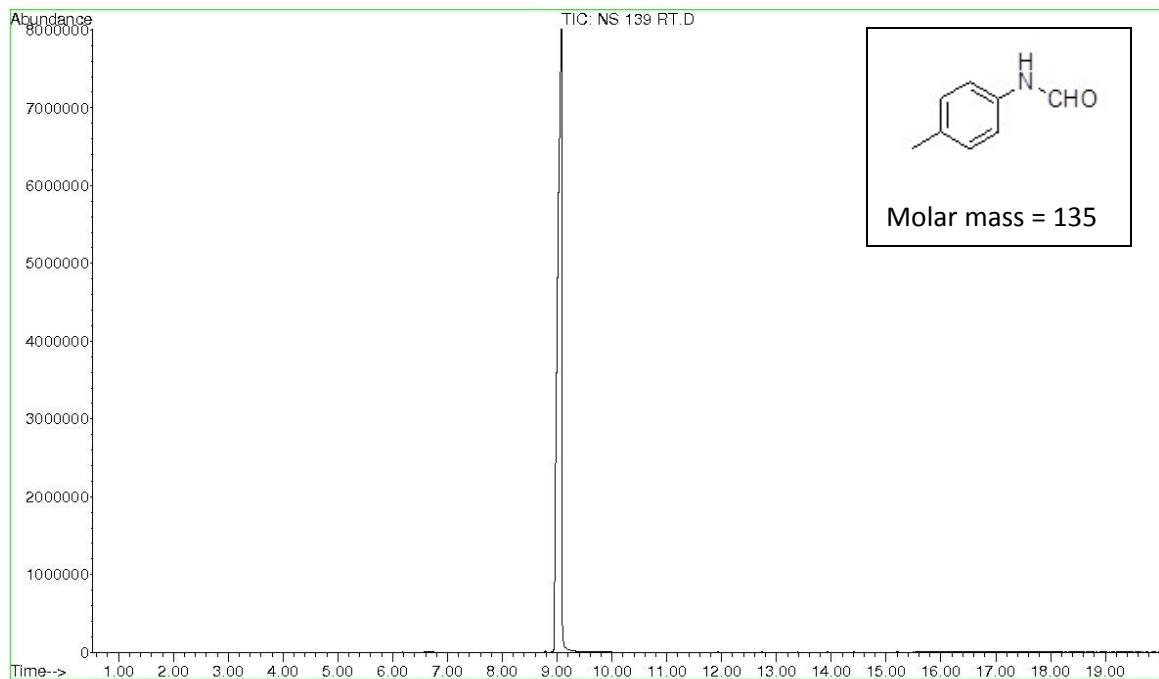


Table 2, Entry 4

File :C:\Sanny\Data\NS93.D
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 Acquired : 3 Nov 2015 4:05 pm using AcqMethod SAMPLES- 20 MIN_20151015_A.M
 Instrument : Instrument #1
 Sample Name: NS93
 Misc Info :
 Vial Number: 7

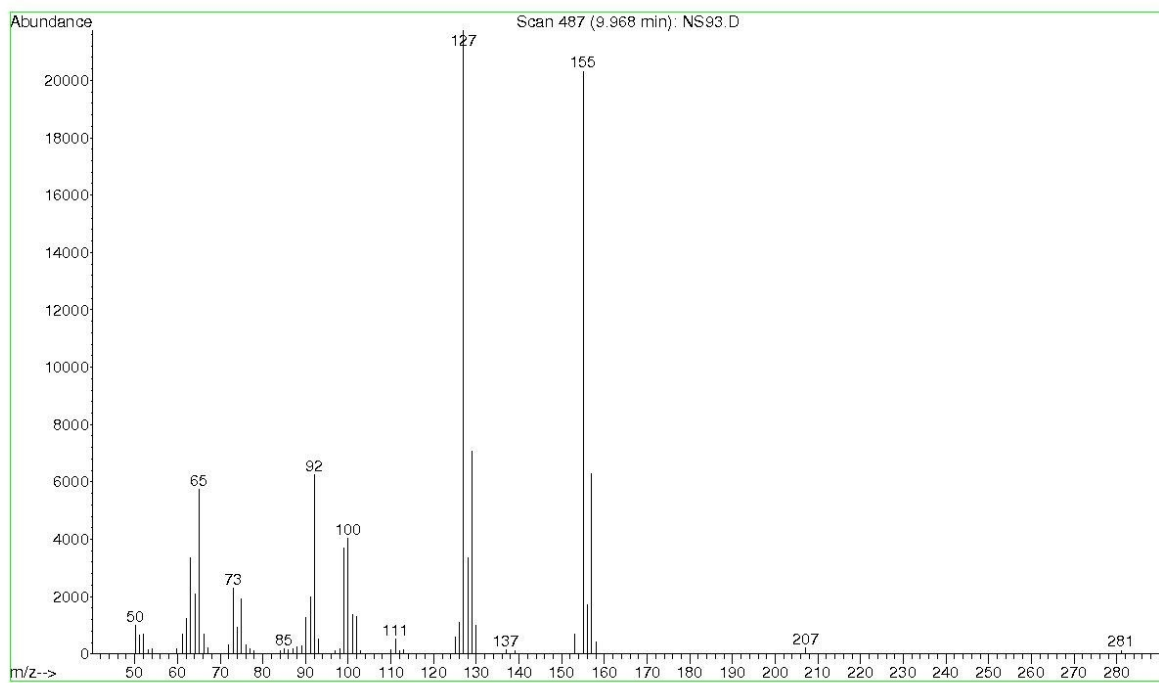
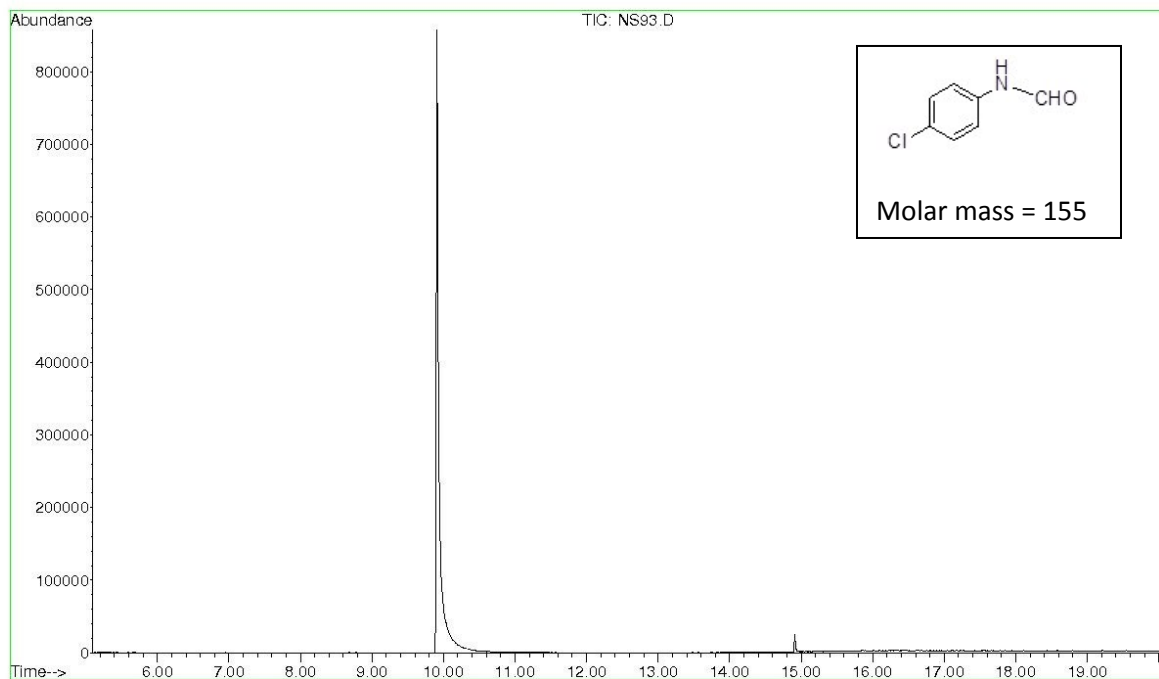


Table 2, Entry 5

File :C:\Sanny\Data\NS 97 HJT.D
Operator : kt
Acquired : 19 Oct 2015 1:12 pm using AcqMethod SAMPLES- 20 MIN_20151015_A.M
Instrument : Instrument #1
Sample Name: NS 97 HJT
Misc Info :
Vial Number: 8

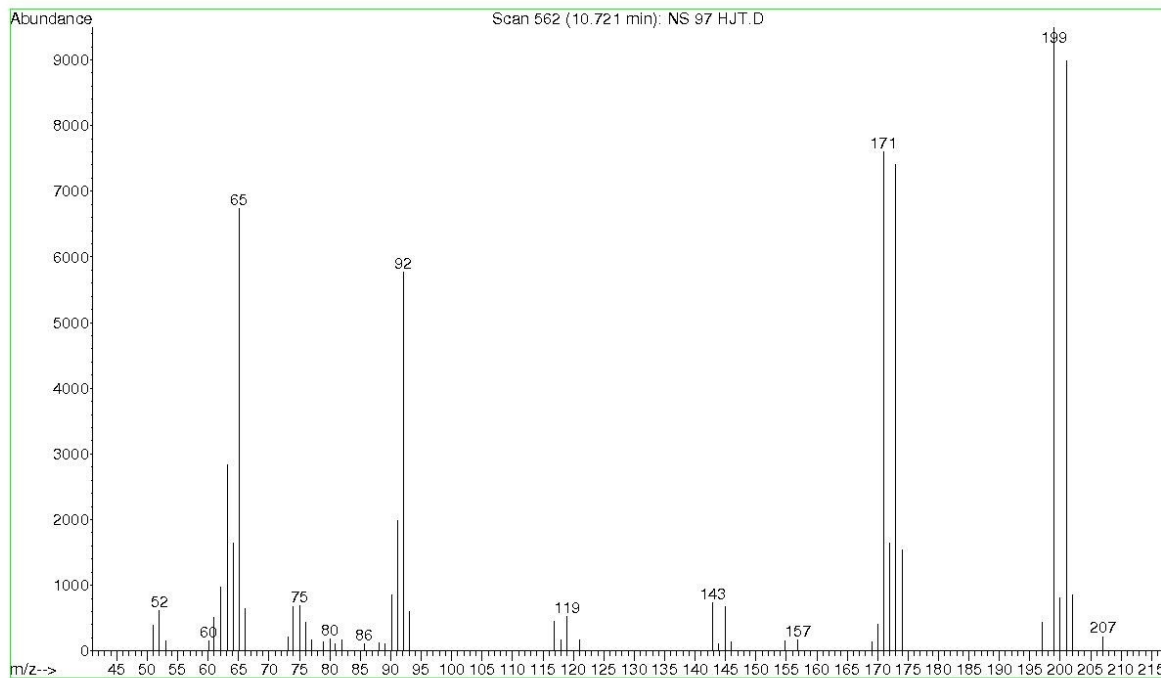
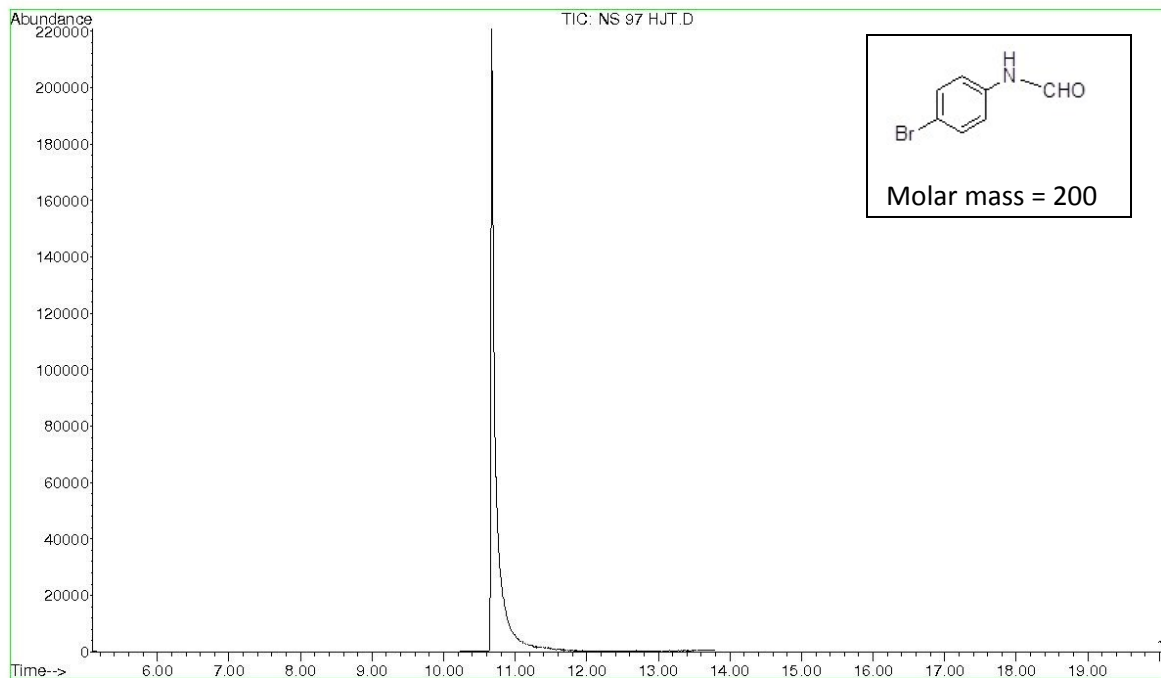
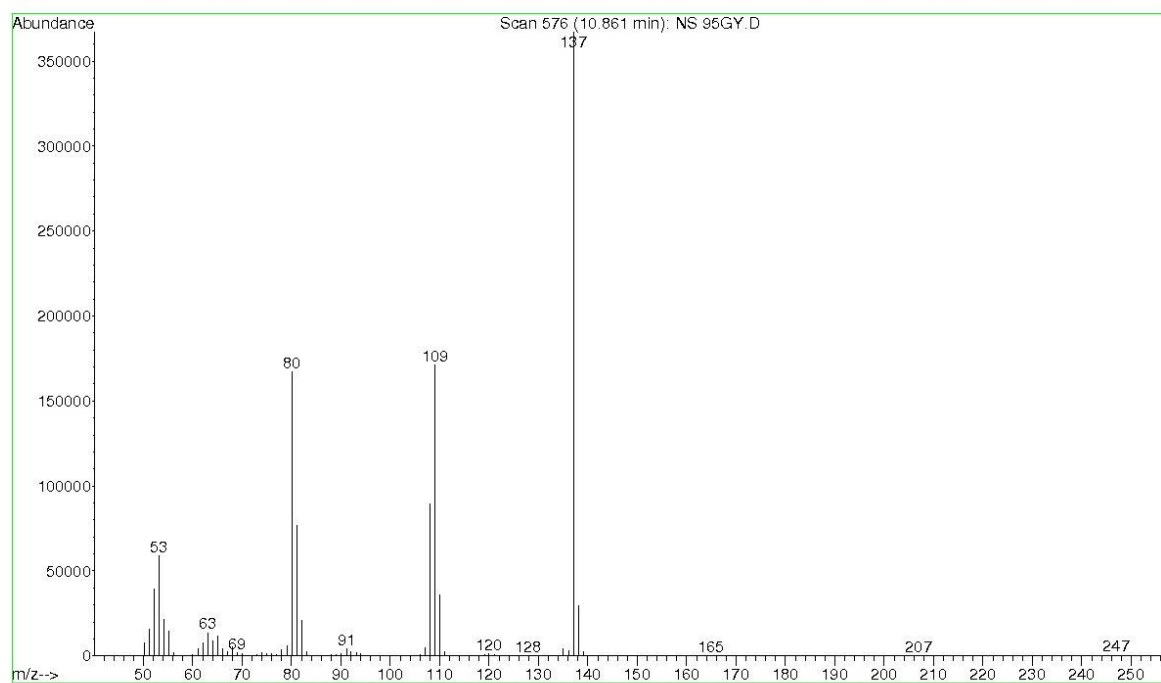
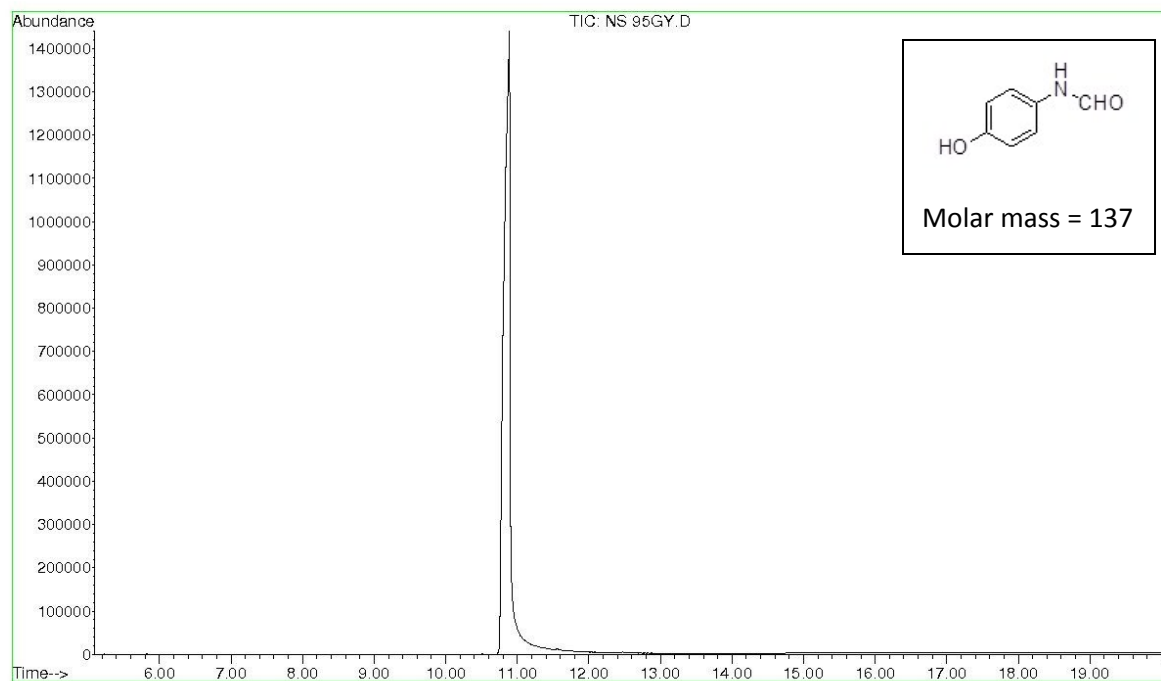


Table 2, Entry 6

File : C:\Sanny\Data\NS 95GY.D
Operator : kt
Acquired : 10 Nov 2015 4:29 pm using AcqMethod SAMPLES- 20 MIN_20151015_A.M
Instrument : Instrument #1
Sample Name: NS 95GY
Misc Info :
Vial Number: 17



File :C:\Sanny\Data\NS 94GY.D
Operator : kt
Acquired : 10 Nov 2015 5:36 pm using AcqMethod SAMPLES- 20 MIN_20151015_A.M
Instrument : Instrument #1
Sample Name: NS 94GY
Misc Info :
Vial Number: 18

Table 2, Entry 7

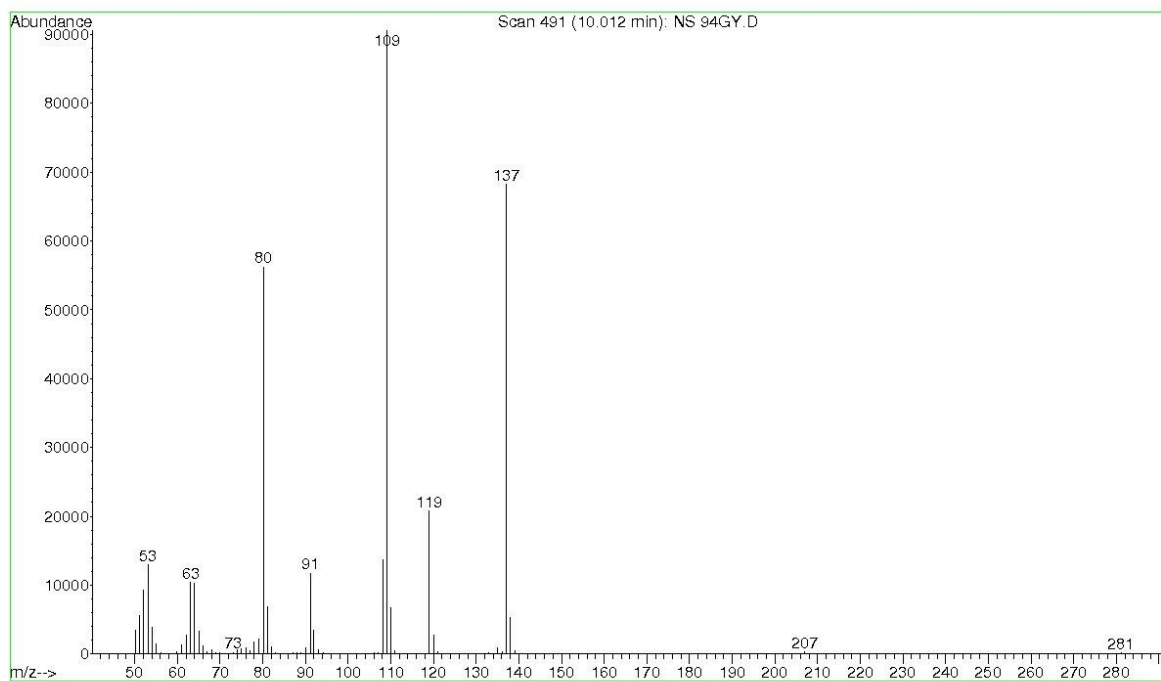
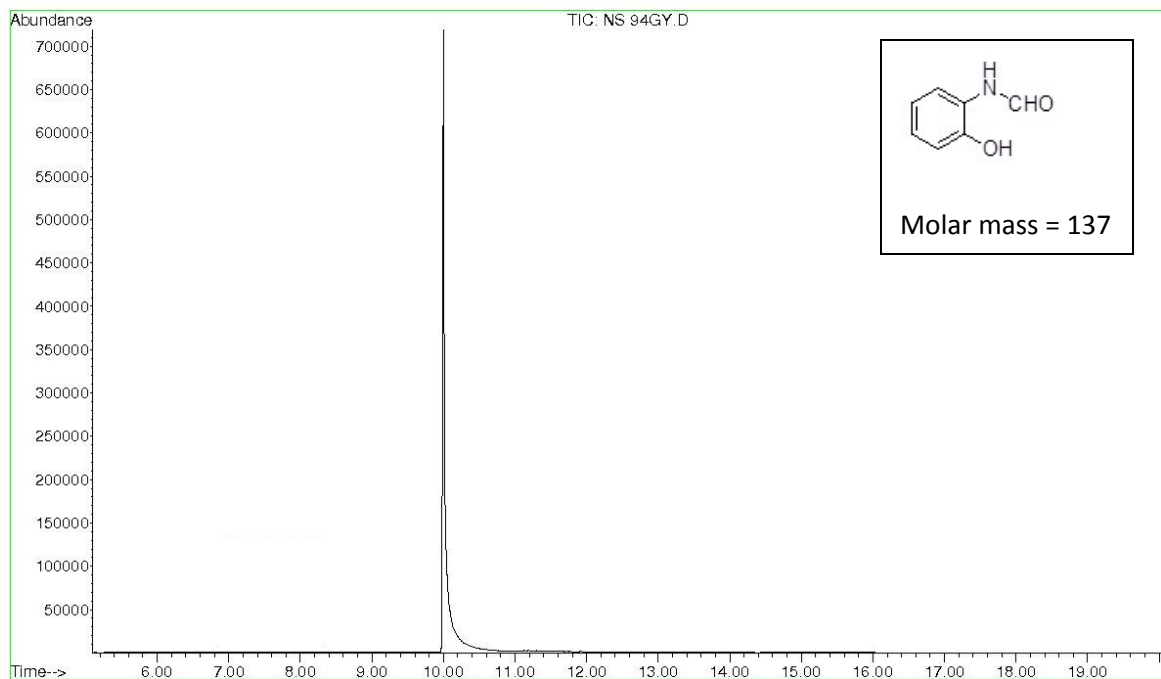
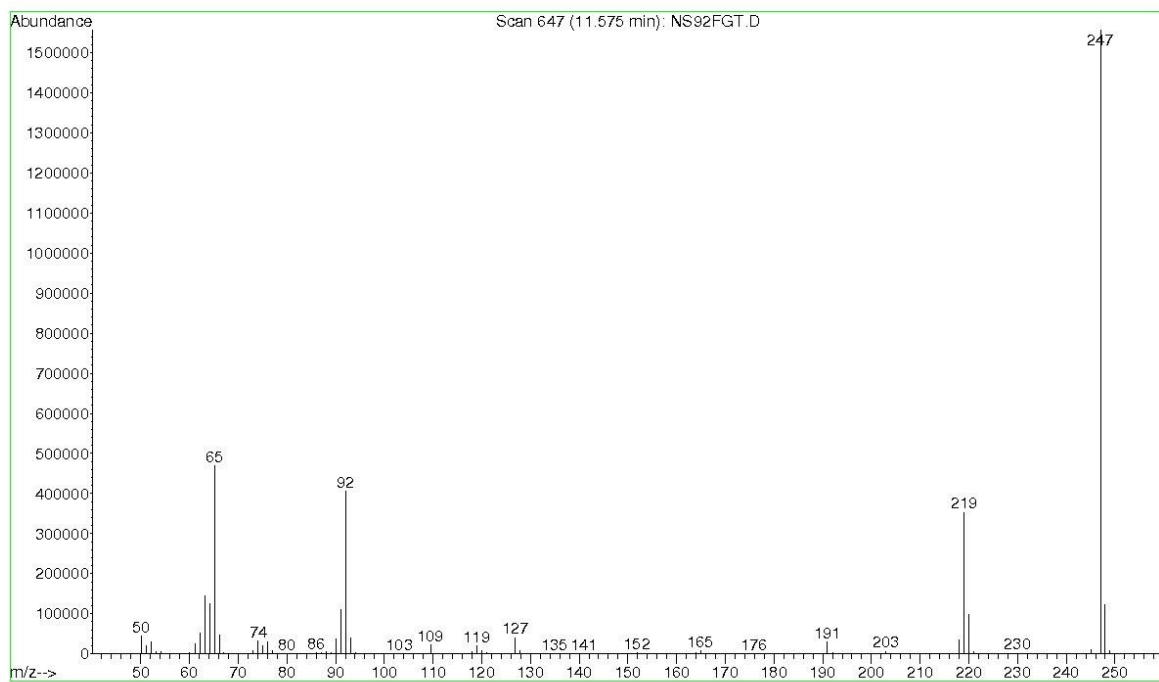
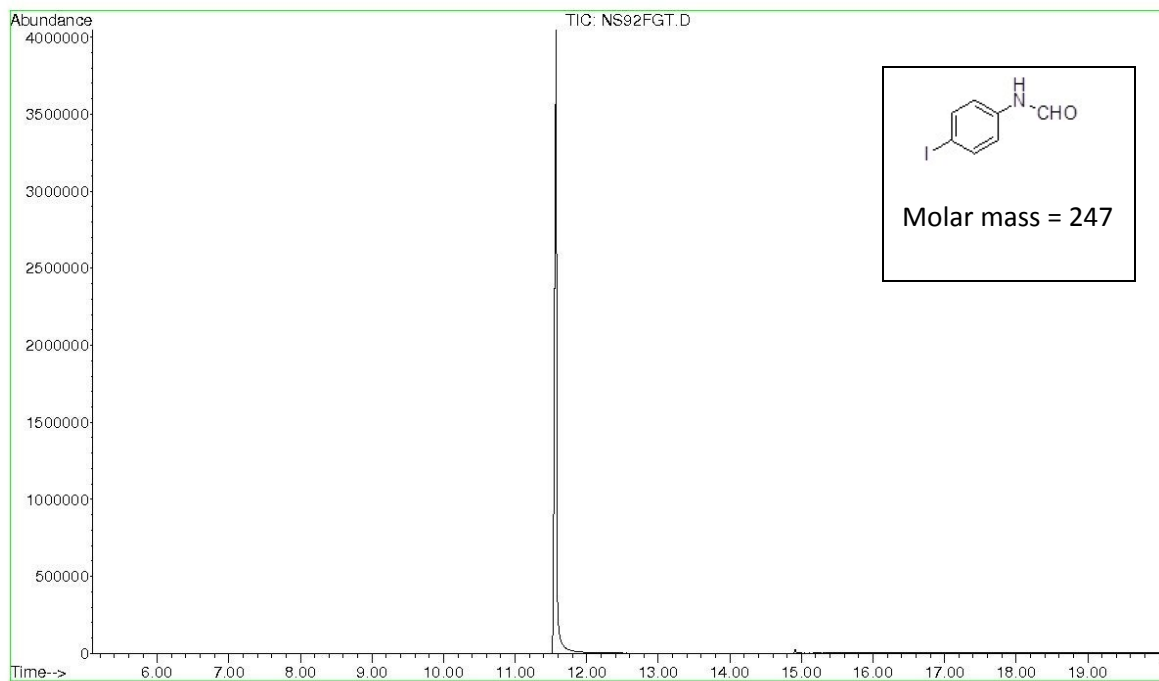


Table 2, Entry8

File :C:\Sanny\Data\NS92FGT.D
Operator : kt
Acquired : 12 Nov 2015 5:18 pm using AcqMethod SAMPLES- 20 MIN_20151015_A.M
Instrument : Instrument #1
Sample Name: NS92FGT
Misc Info :
Vial Number: 10



File :C:\Sanny\Data\NS 151.D
Operator : kt
Acquired : 9 Dec 2015 3:19 pm using AcqMethod SAMPLES- 20 MIN_20151015_A.M
Instrument : Instrument #1
Sample Name: NS 151
Misc Info :
Vial Number: 18

Table 2, Entry 10

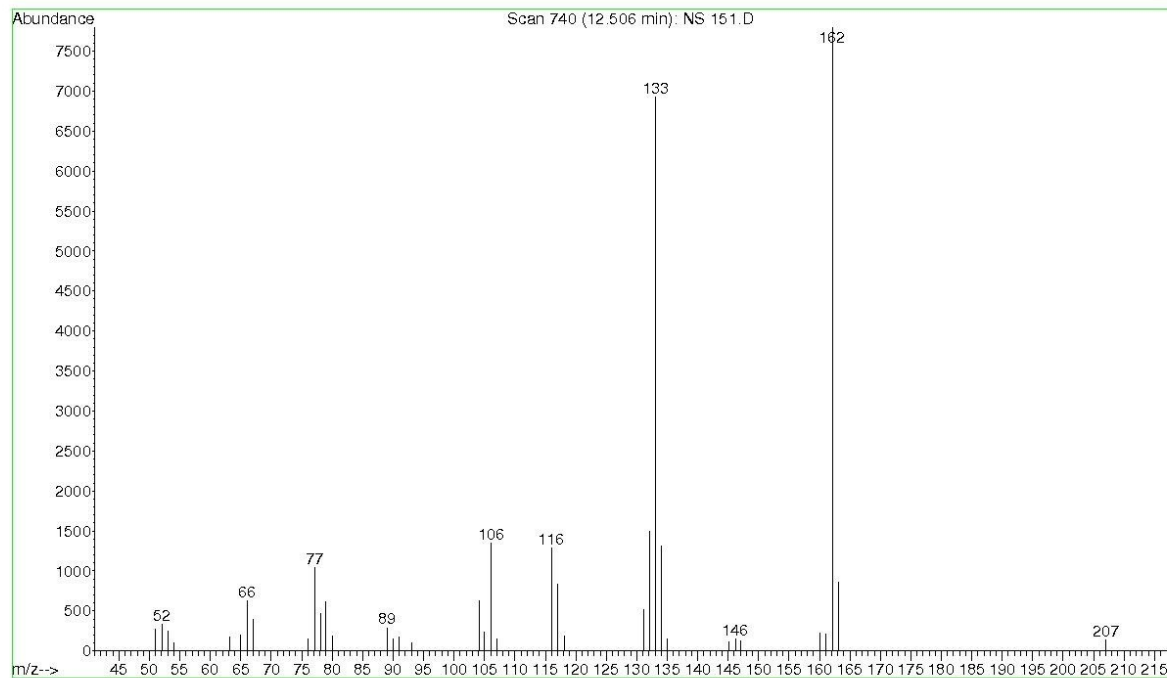
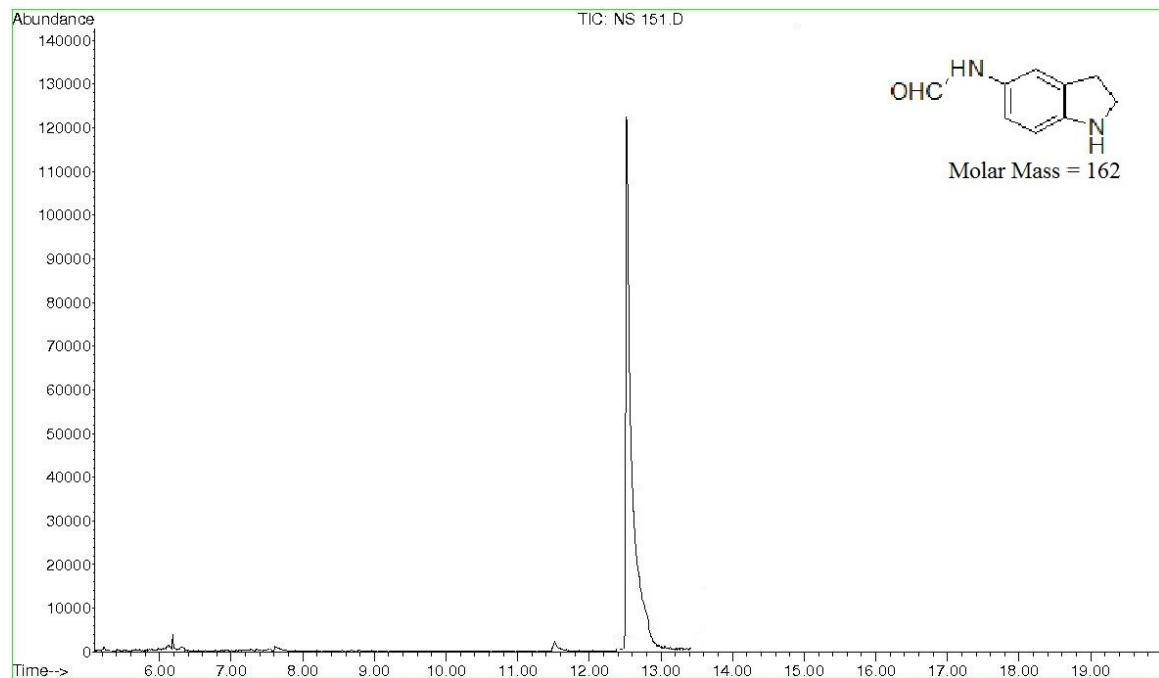


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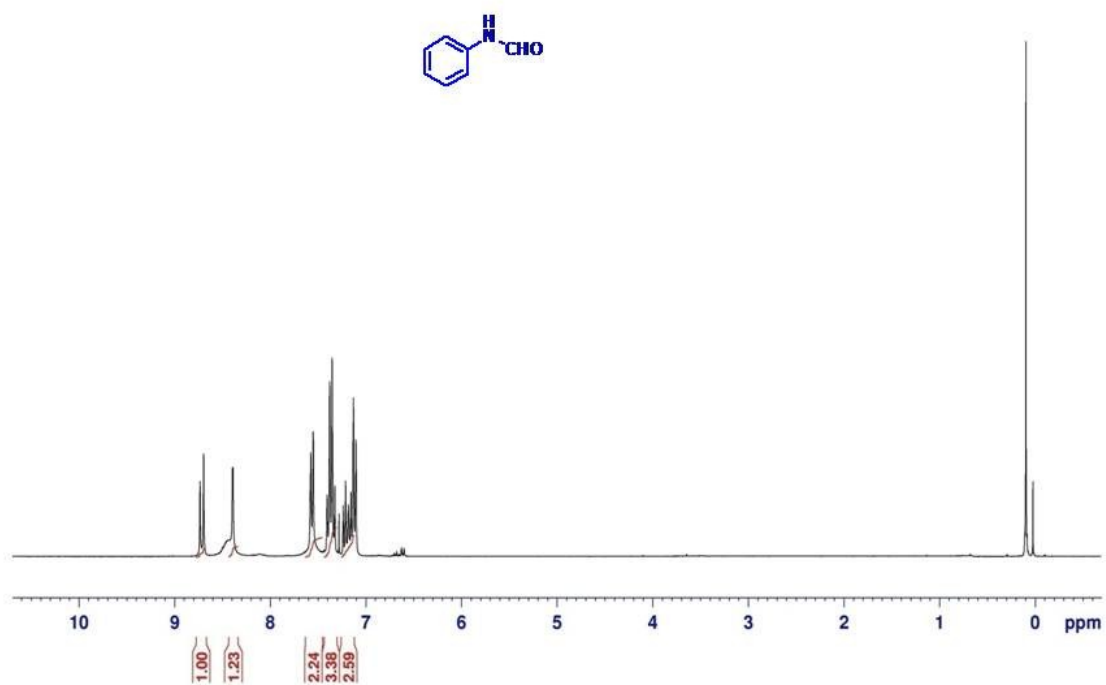


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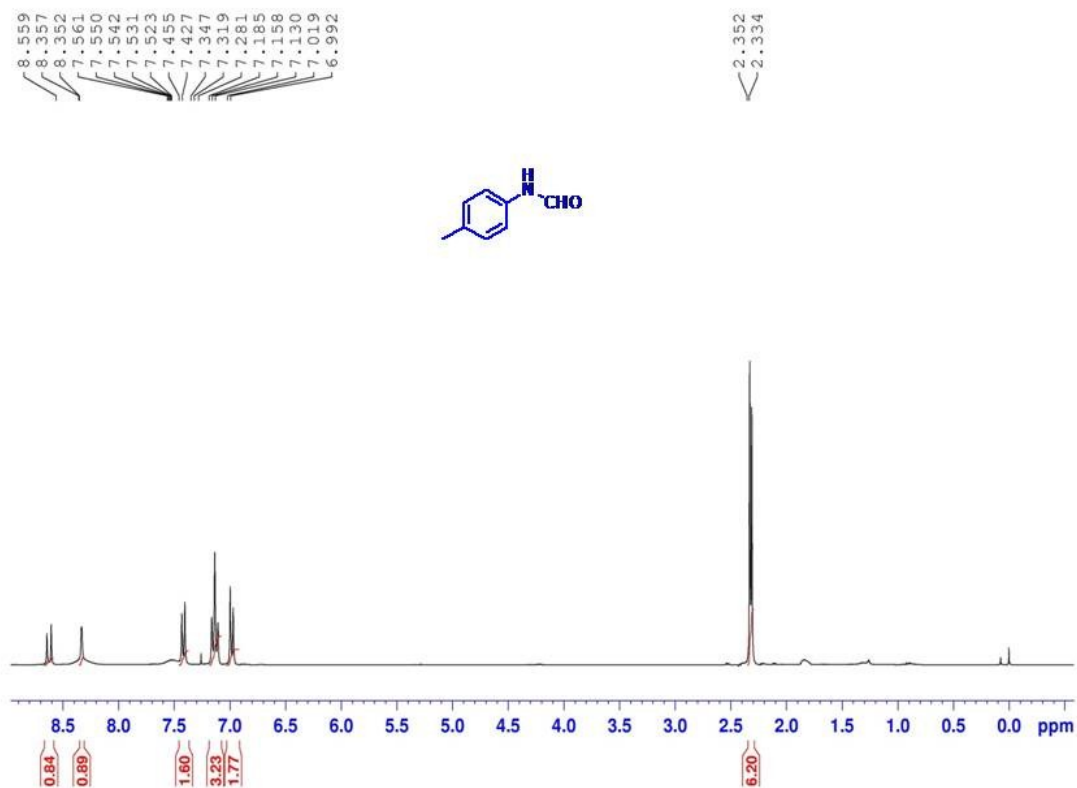


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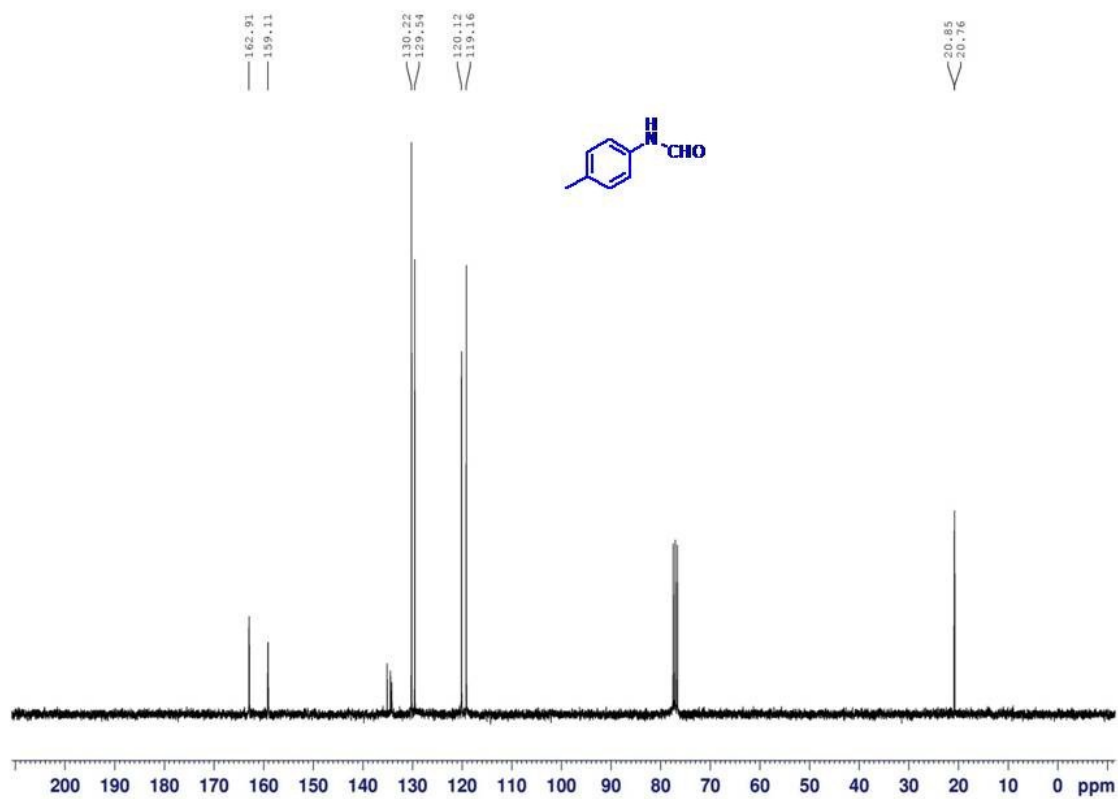


Table 2, Entry 4

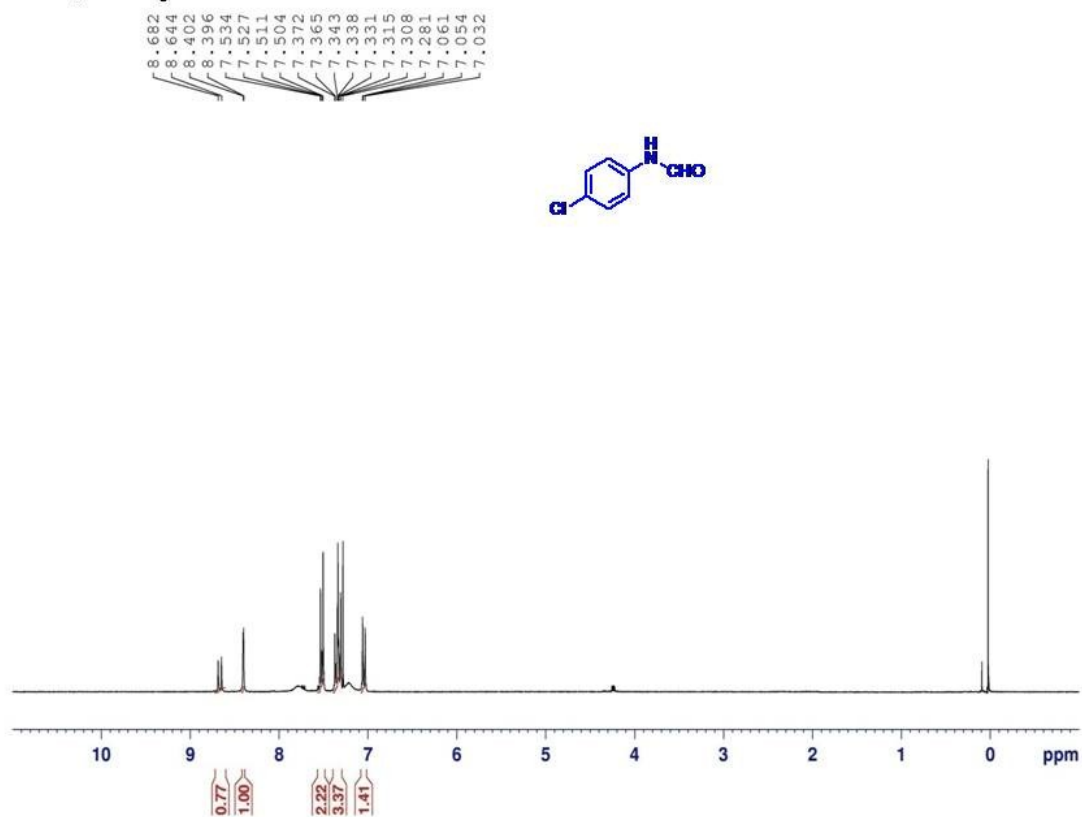


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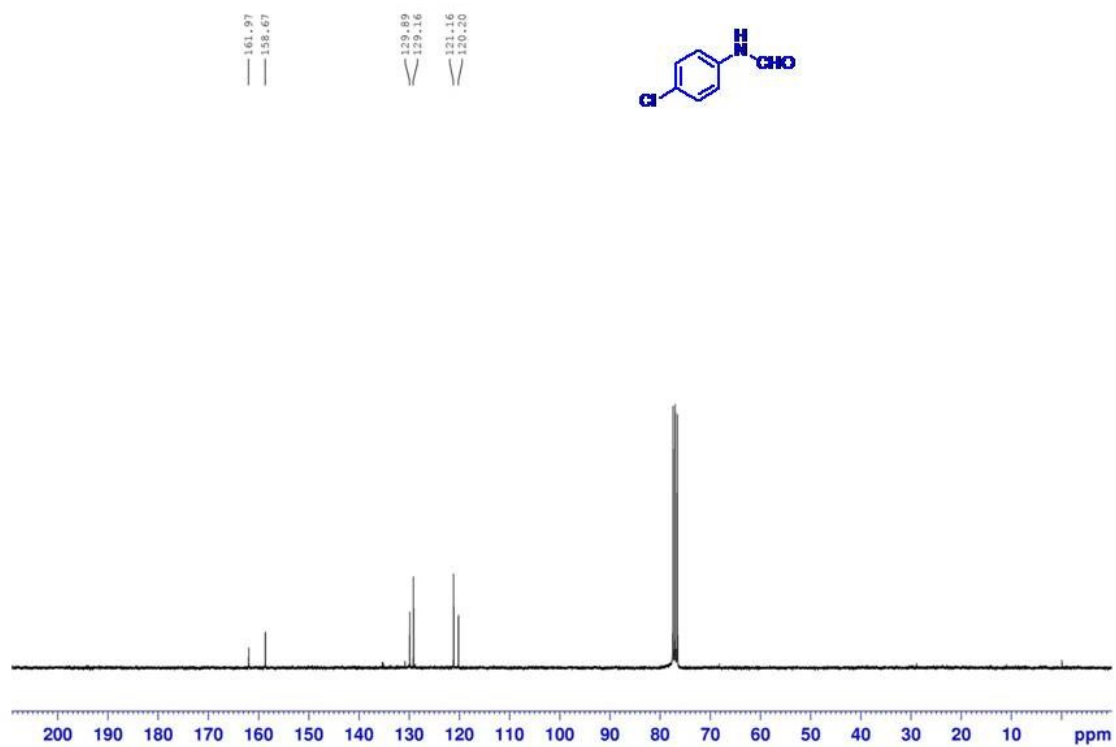


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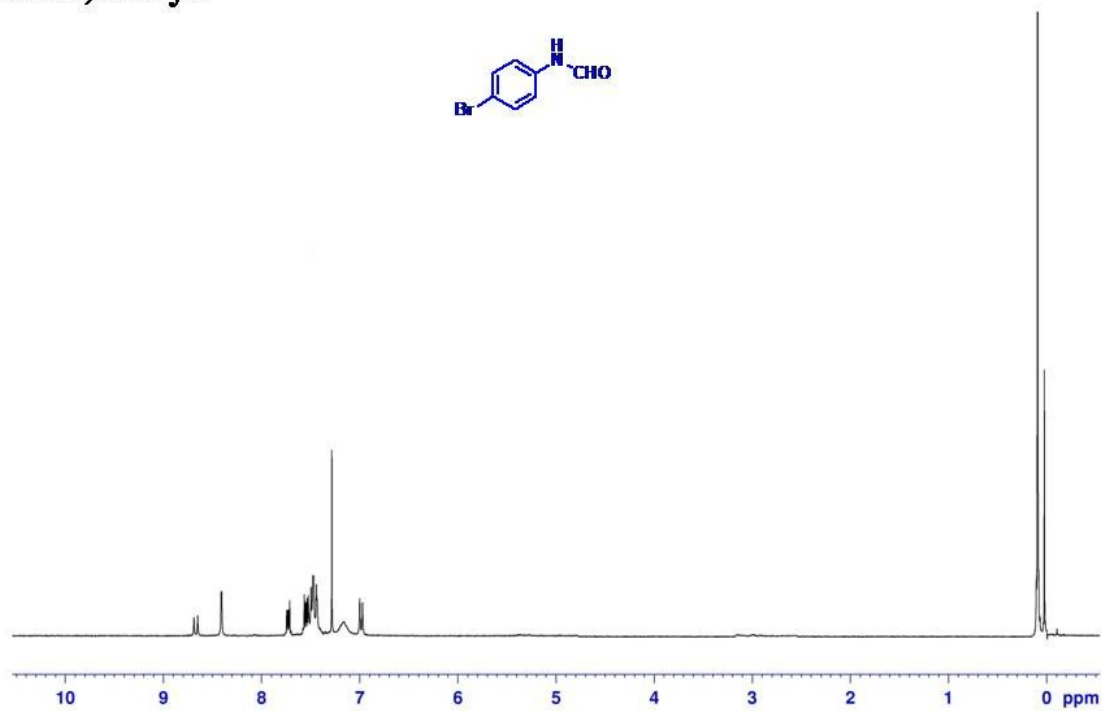
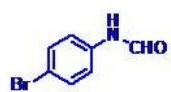


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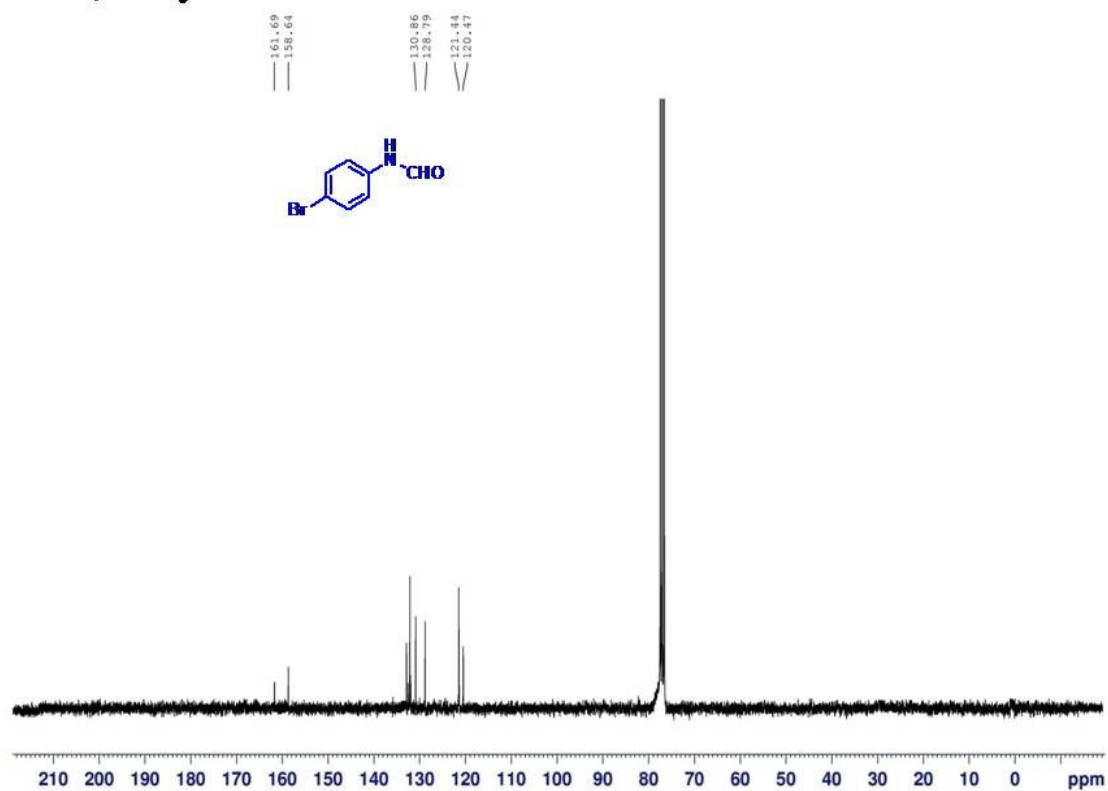


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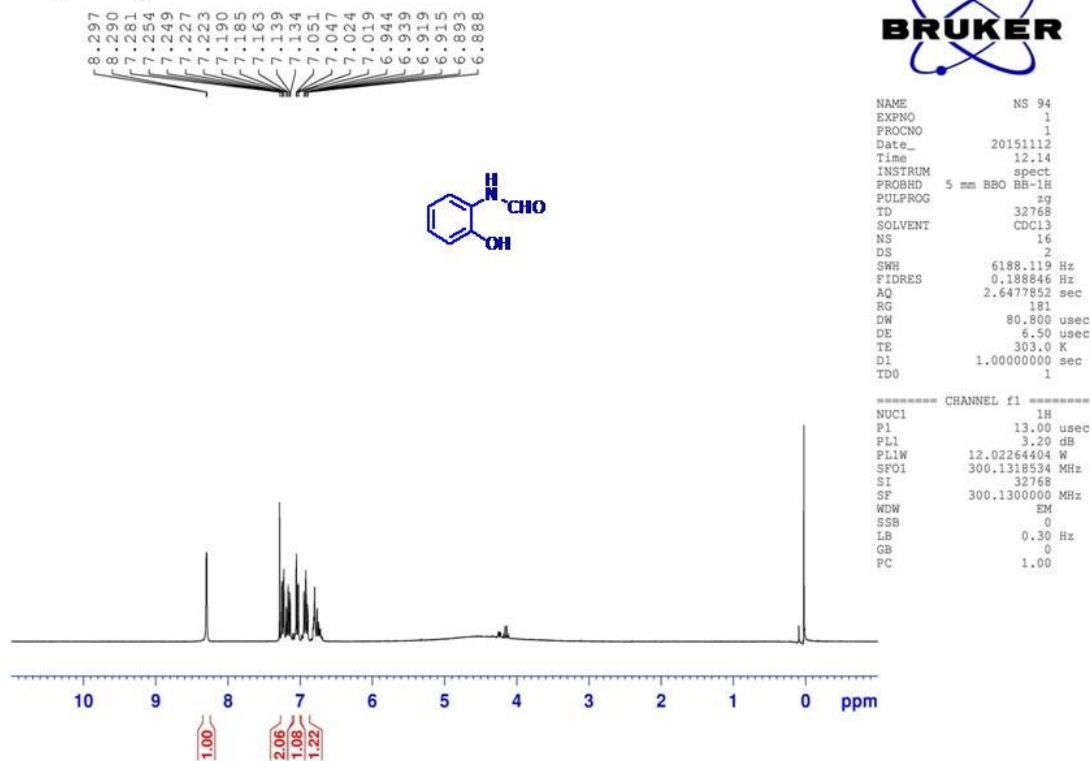


Table 2, Entry 7

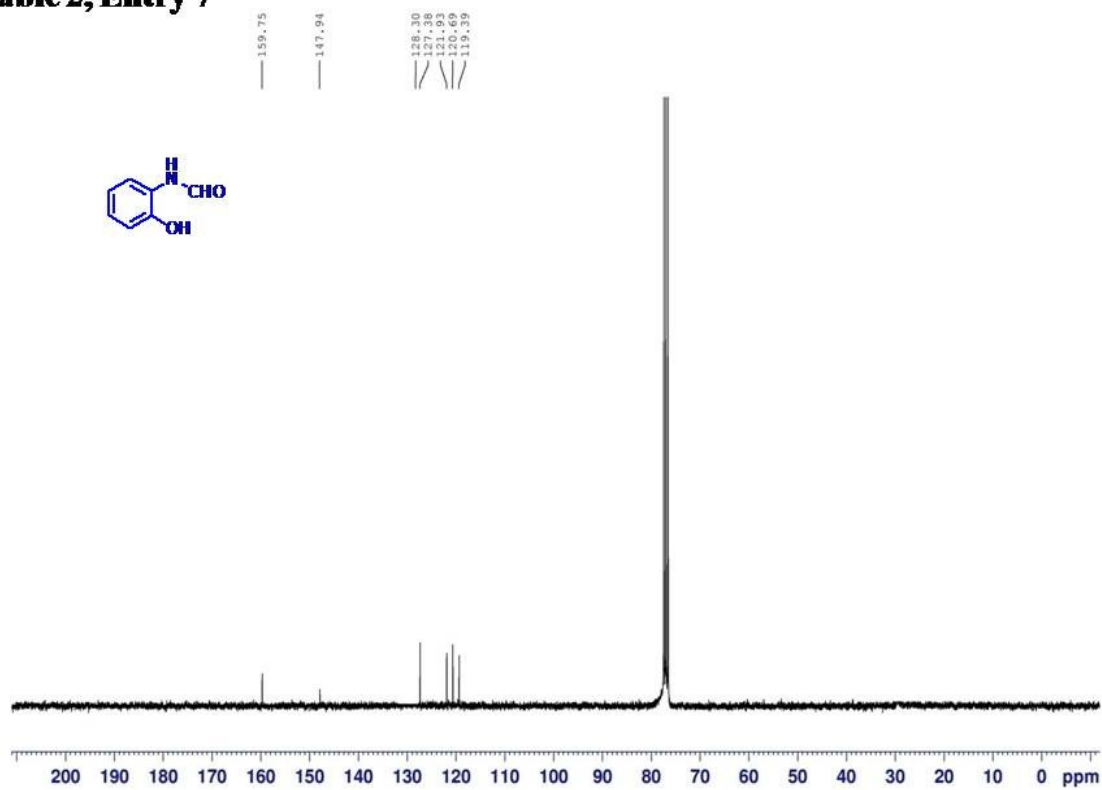
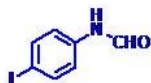


Table 2, Entry 8

8.704
8.666
8.409
8.404
7.701
7.673
7.644
7.361
7.332
6.888
6.859



```
NAME          NS 92
EXPNO         1
PROCNO        1
Date_         20151113
Time          14.19
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg
TD            32768
SOLVENT       CDCl3
NS            16
DS            2
SWH           6188.119 Hz
FIDRES        0.188846 Hz
AQ            2.6477852 sec
RG            181
DW            80.800 usec
DE            6.50 usec
TE            303.0 K
D1            1.00000000 sec
TD0           1
```

```
===== CHANNEL f1 =====
NUC1          1H
P1            13.00 usec
PL1           3.20 dB
PL1W          12.02264404 W
SFO1          300.1318534 MHz
SI            32768
SF            300.1300000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
```

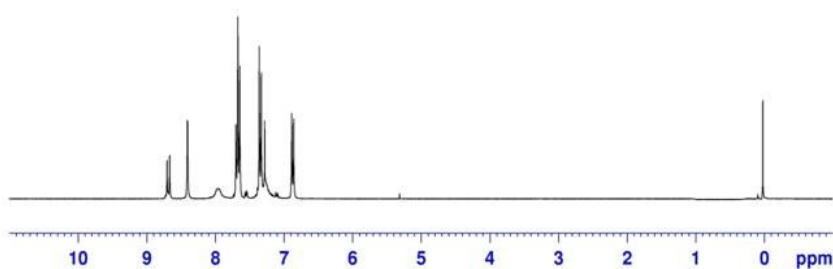
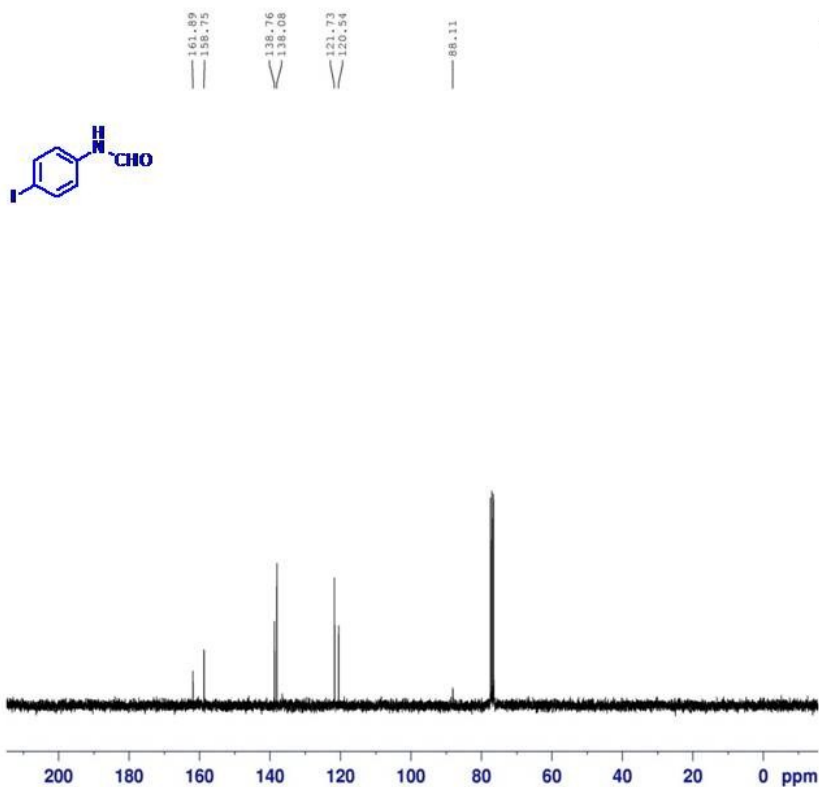


Table 2, Entry 8



```

=====
NAME      NS 92
EXPNO     2
PROCNO    1
Date_     20151113
Time      15.19
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgig
TD         32768
SOLVENT   CDC13
NS         1024
DS         4
SWH        17985.611 Hz
FIDRES     0.548877 Hz
AQ         0.9110282 sec
RG         8192
DW         27.800 usec
DE         6.50 usec
TE         303.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1        10.00 usec
PL1       1.80 dB
PL1W      49.78760910 W
SFO1      75.4752953 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     80.00 usec
PL2        3.20 dB
PL12      18.98 dB
PL2W      12.02264404 W
PL12W     0.31768745 W
SFO2      300.1312005 MHz
SI         32768
SF         75.4677490 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```