

## Supporting Information

### Oxidative esterification via photocatalytic C-H activation

**Sanny Verma<sup>a†</sup>, R. B. Nasir Baig<sup>a†</sup>, Changseok Han<sup>b</sup>, Mallikarjuna N. Nadagouda<sup>b</sup> and Rajender S. Varma<sup>a\*</sup>**

<sup>a</sup>Sustainable Technology Division, National Risk Management Research Laboratory, U. S. Environmental Protection Agency, MS 443, Cincinnati, Ohio 45268, USA. Fax: 513- 569-7677; Tel: 513-487-2701. E-mail:varma.rajender@epa.gov

<sup>b</sup>WQMB, WSWRD, National Risk Management Research Laboratory, U. S. Environmental Protection Agency, MS 443, Cincinnati, Ohio 45268, USA

#### 1. Synthesis and recycling of the catalyst

- a) Synthesis of g-C<sub>3</sub>N<sub>4</sub>
- b) Synthesis of VO@g-C<sub>3</sub>N<sub>4</sub> catalyst
- c) Recycling of VO@C<sub>3</sub>N<sub>4</sub> catalyst

#### 2. TEM image of g-C<sub>3</sub>N<sub>4</sub> support (S2)

#### 3. SEM image of recycled VO@C<sub>3</sub>N<sub>4</sub> catalyst (S3)

#### 4. <sup>1</sup>H and <sup>13</sup>C NMR of the product

## **1. Synthesis of materials**

### **a) Synthesis of g-C<sub>3</sub>N<sub>4</sub>:**

The graphitic carbon nitride, g-C<sub>3</sub>N<sub>4</sub>, was synthesized by calcinations of urea at 500 °C.

### **b) Synthesis of VO@g-C<sub>3</sub>N<sub>4</sub> catalyst:**

g-C<sub>3</sub>N<sub>4</sub> (1.0 g) was dispersed in 200 ml aqueous methanol (50 %) under sonication; to this dispersion the methanolic solution of vanadyl acetylacetonate [VO(acac)<sub>2</sub>; 1 mmol] was added and stirred for 3h at room temperature. The reaction mixture was centrifuged, washed with methanol and dried under vacuum at 50 °C to give the formation of VO@g-C<sub>3</sub>N<sub>4</sub> catalyst as pale yellow solid. The VO@g-C<sub>3</sub>N<sub>4</sub> was isolated and characterized using SEM, TEM, XRD and ICP-AES analysis

### **(c) Recycling of VO@g-C<sub>3</sub>N<sub>4</sub> catalyst:**

To establish the recyclability of the VO@g-C<sub>3</sub>N<sub>4</sub> catalyst for oxidative esterification of alcohol, a set of experiments were performed using benzyl alcohol as a substrate in methanol. After the completion of each reaction the VO@g-C<sub>3</sub>N<sub>4</sub> catalyst was recovered using centrifuge, washed with acetone and reused for the oxidative esterification of benzyl alcohol using fresh reagents. The VO@g-C<sub>3</sub>N<sub>4</sub> catalyst could be recycled and reused up to eight times without losing its activity (S1). The metal leaching of VO@g-C<sub>3</sub>N<sub>4</sub> catalyst was studied by ICP-AES analysis before and after the reaction. The concentration of vanadium was found to be 4.91% before the reaction and 4.88% after the 8<sup>th</sup> cycle. The ICP-AES of the mother liquor did not show the presence of vanadium confirming the fact that the g-C<sub>3</sub>N<sub>4</sub> holds the oxo-vanadium complex tightly which eliminates the vanadium leaching and enables efficient recycling.

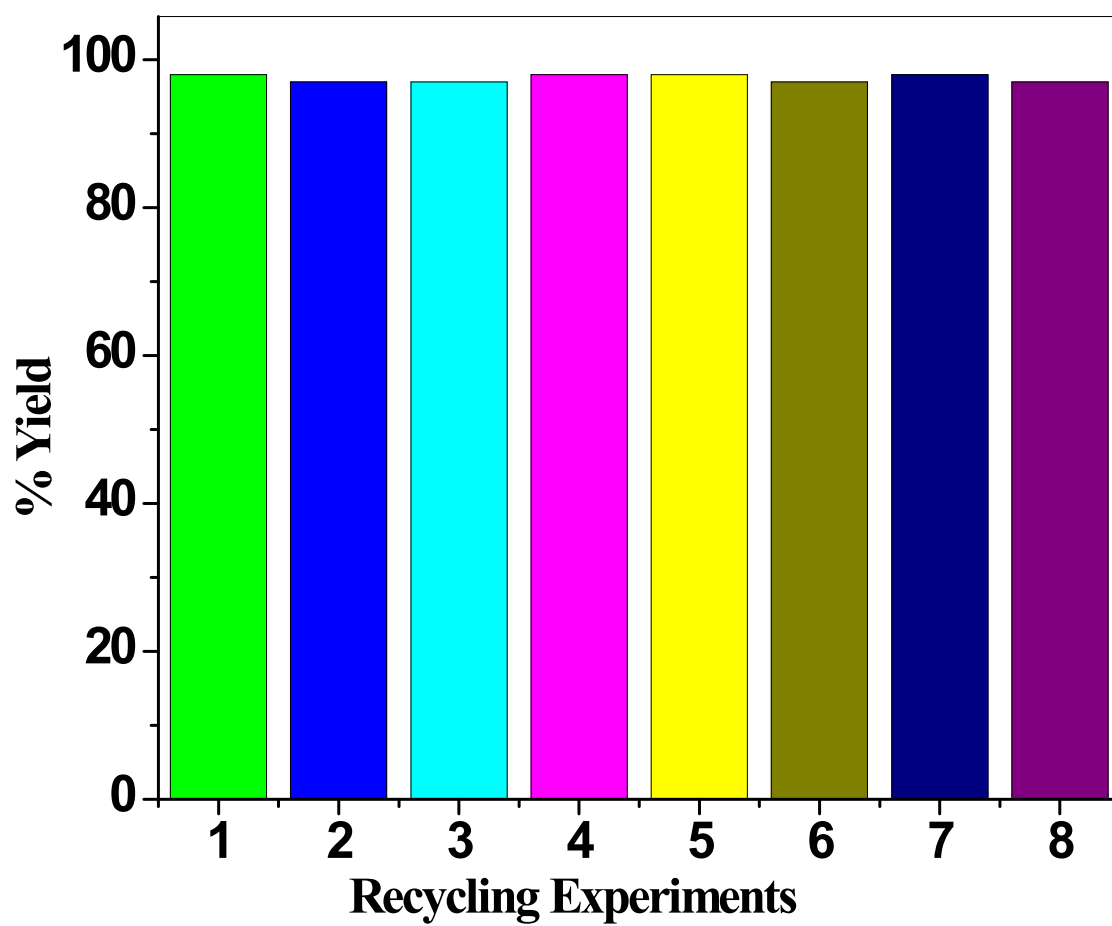


Figure S1: Histogram for recycling experiments

## 2. TEM image g-C<sub>3</sub>N<sub>4</sub> support

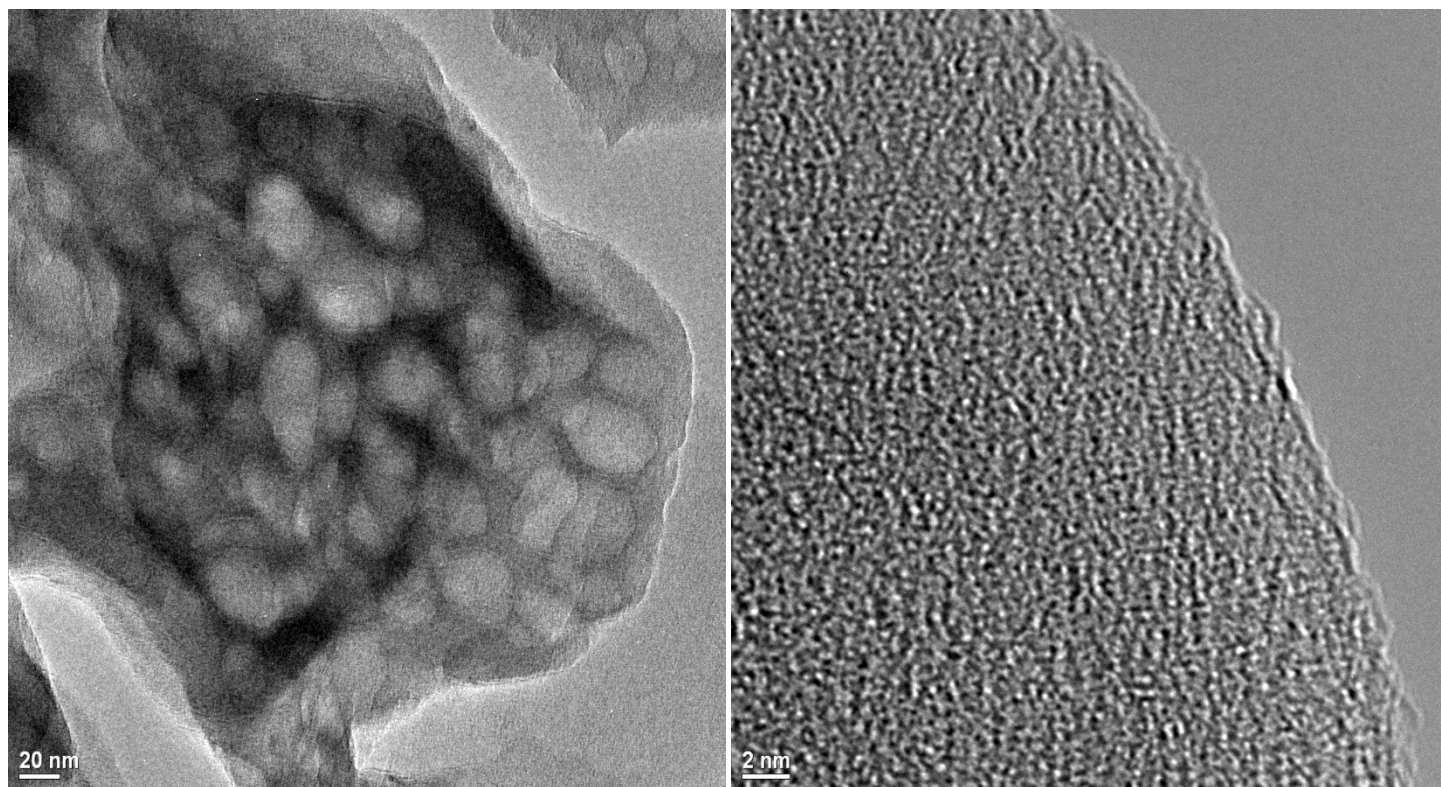


Figure S2: TEM image of g-C<sub>3</sub>N<sub>4</sub> support

### 3. SEM image of recycled VO@C<sub>3</sub>N<sub>4</sub> catalyst

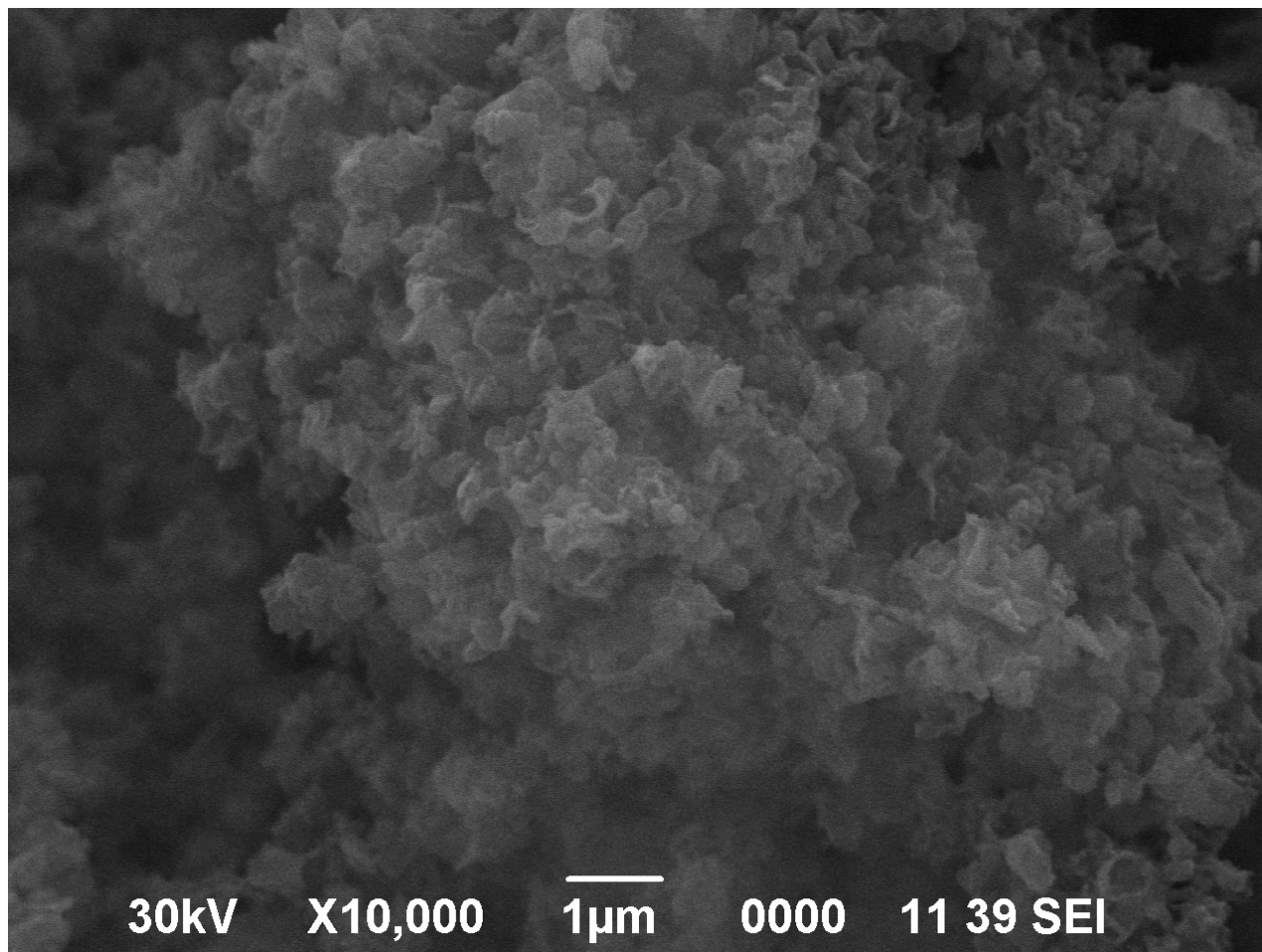
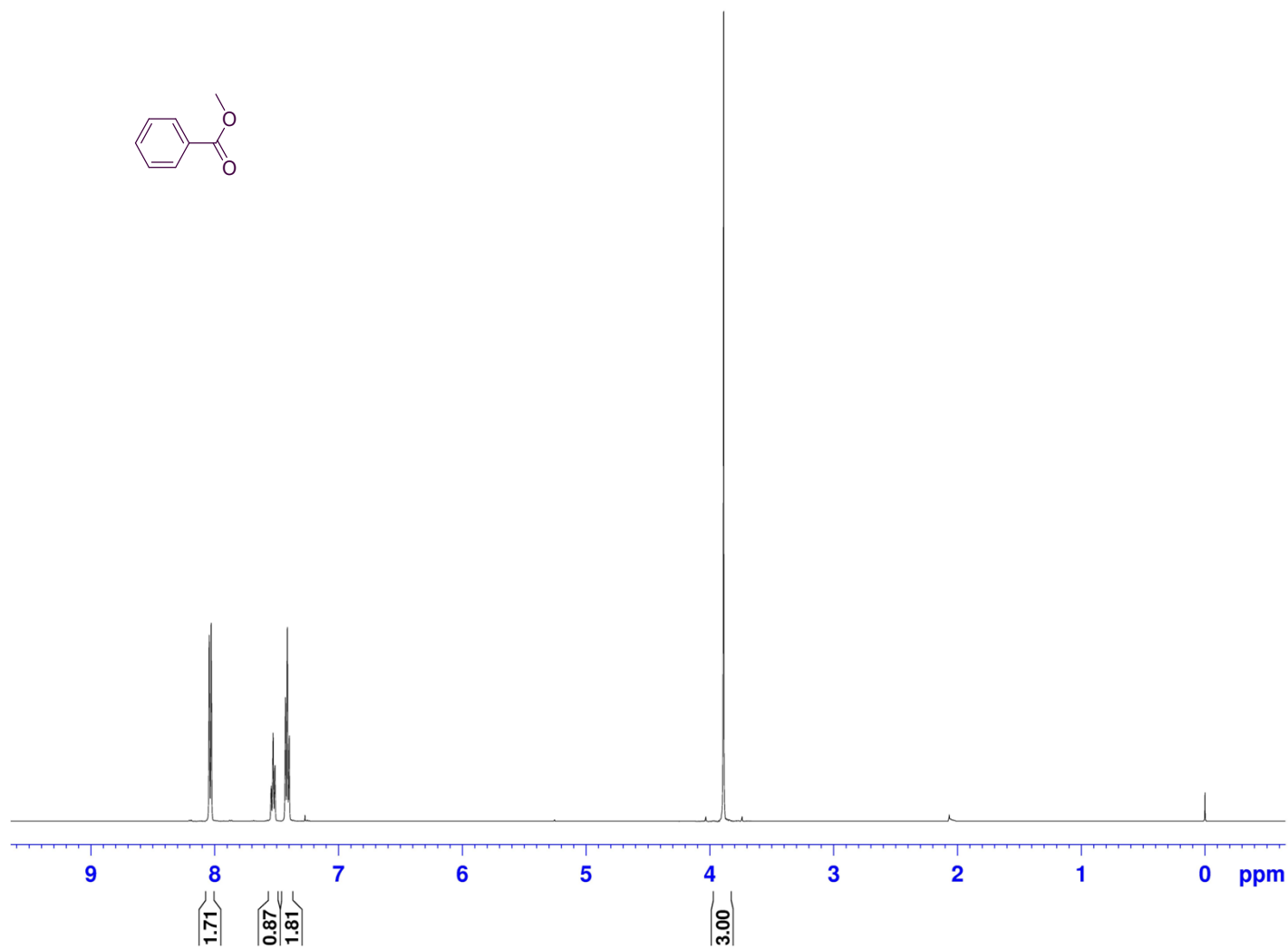
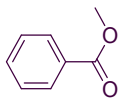
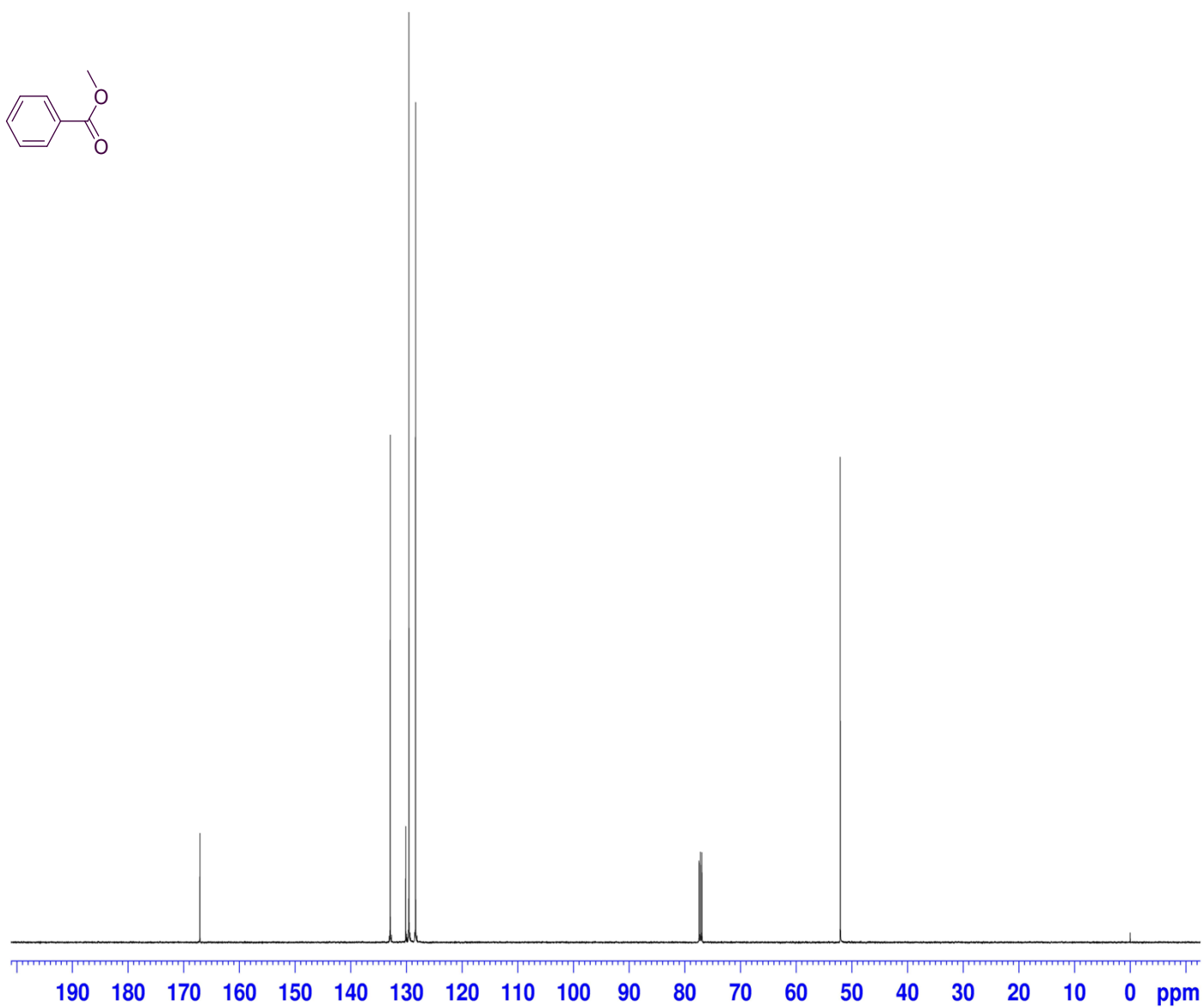
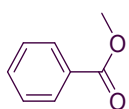
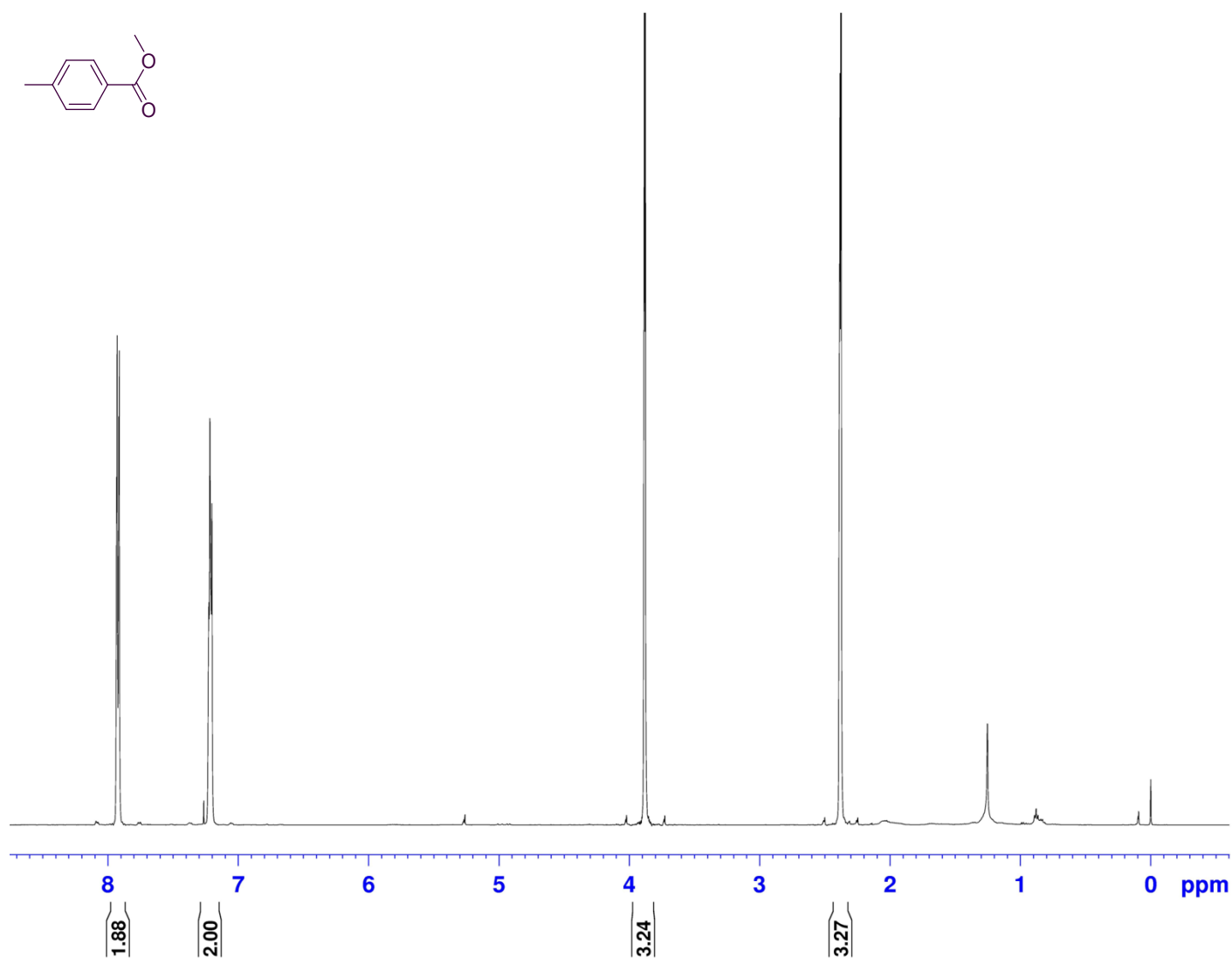
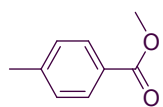


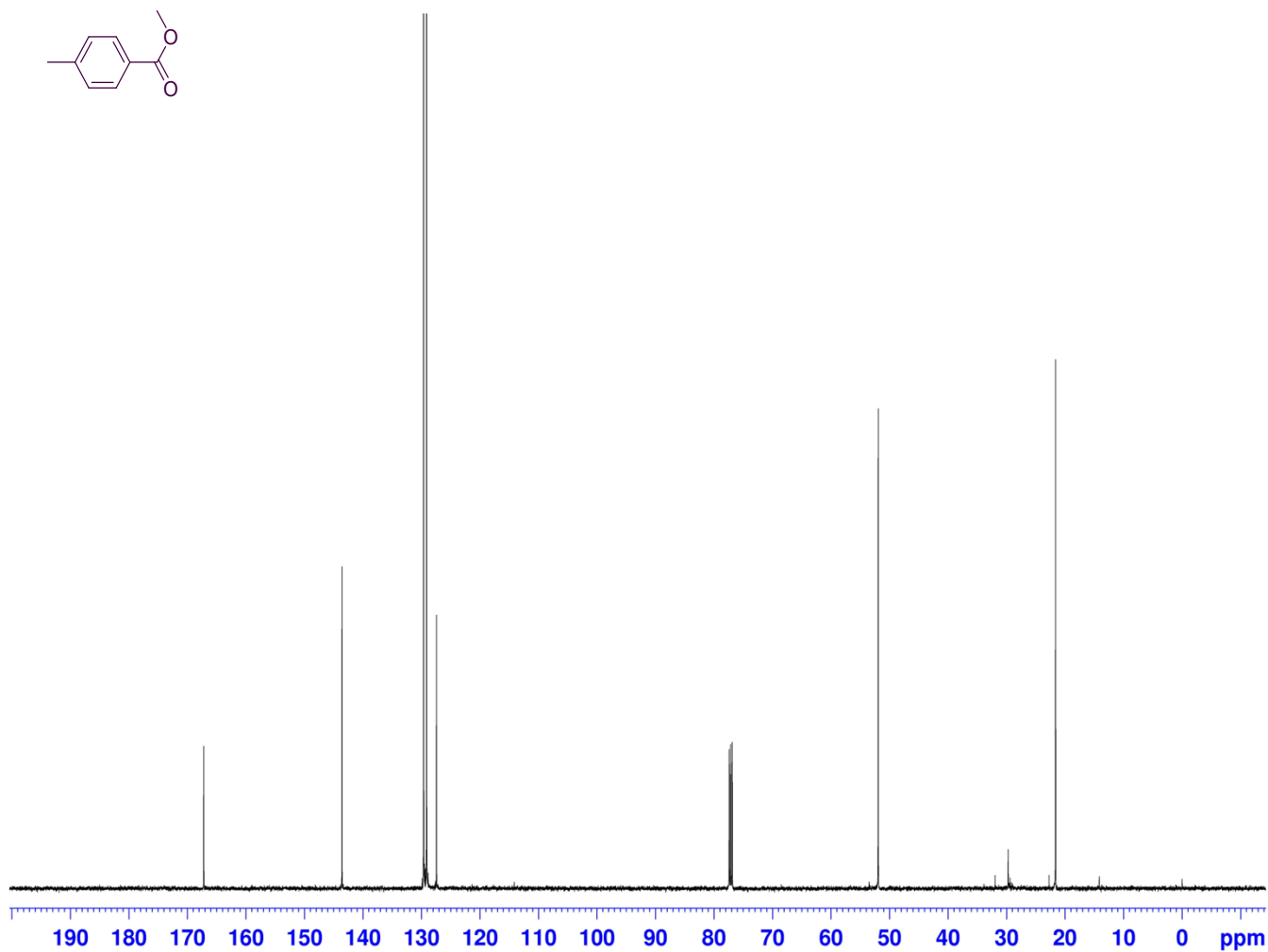
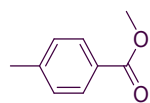
Figure S3: SEM image of recycled VO@g-C<sub>3</sub>N<sub>4</sub> catalyst

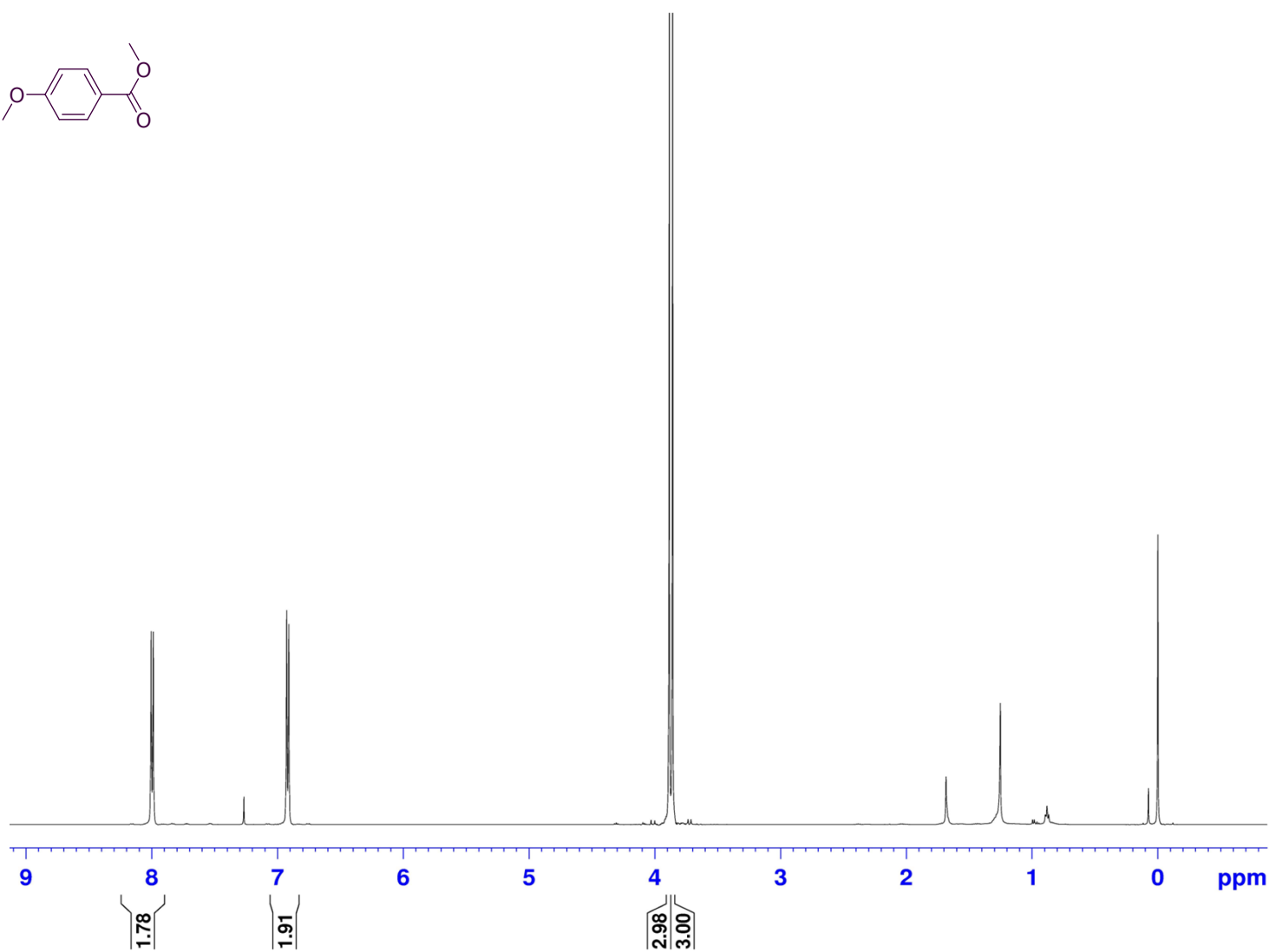
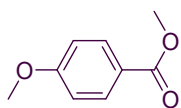


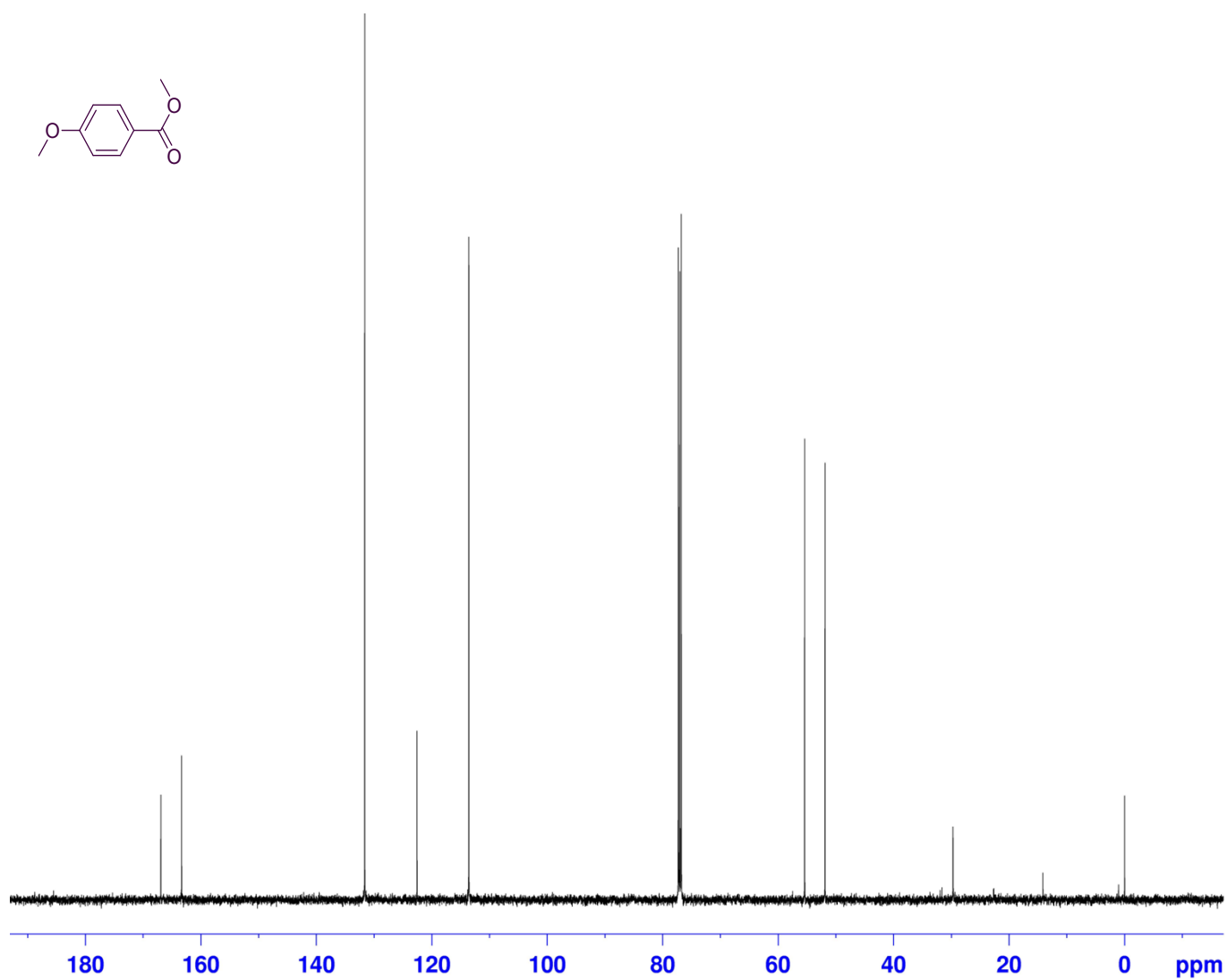
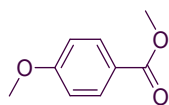


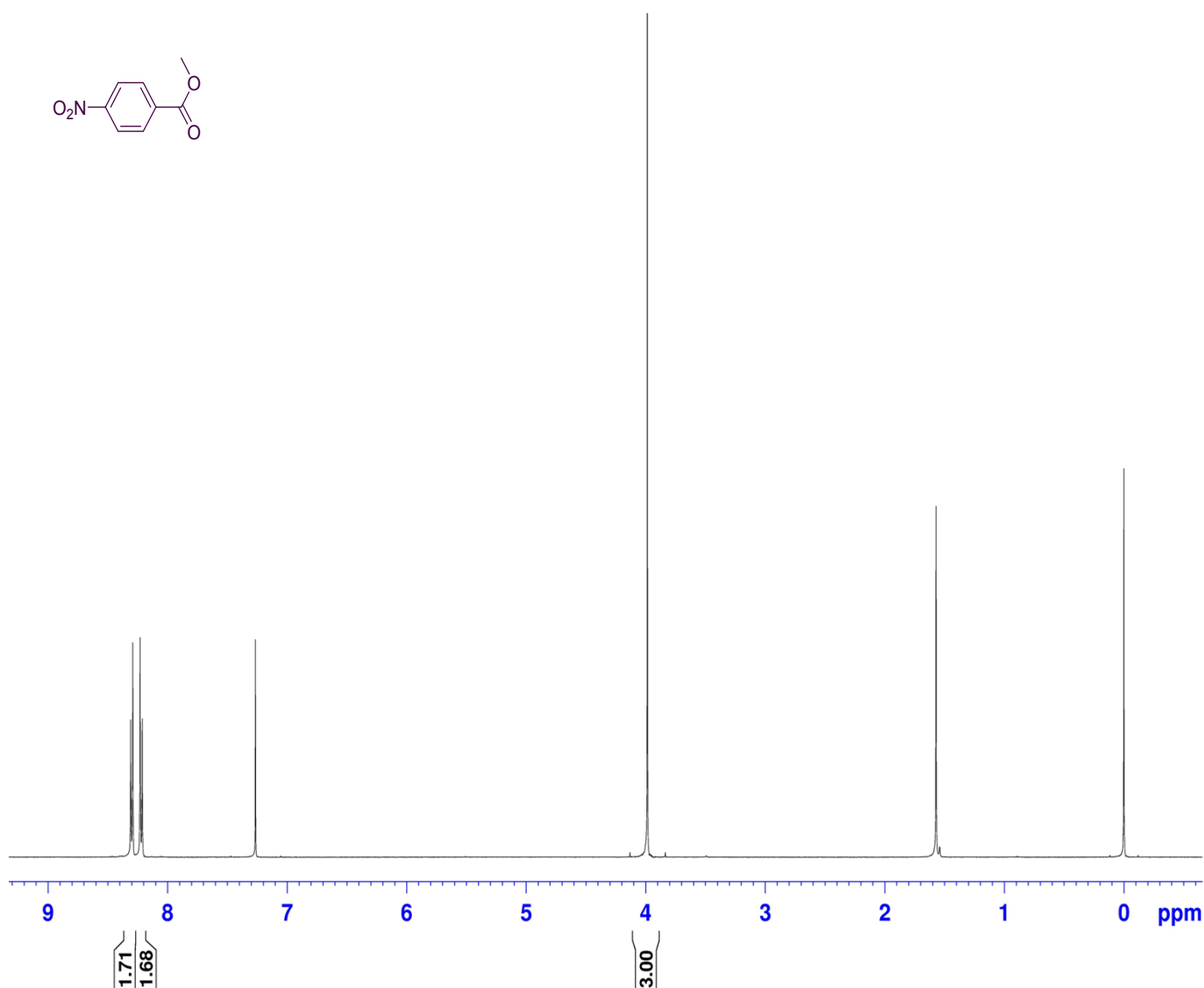
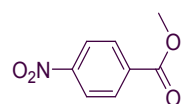


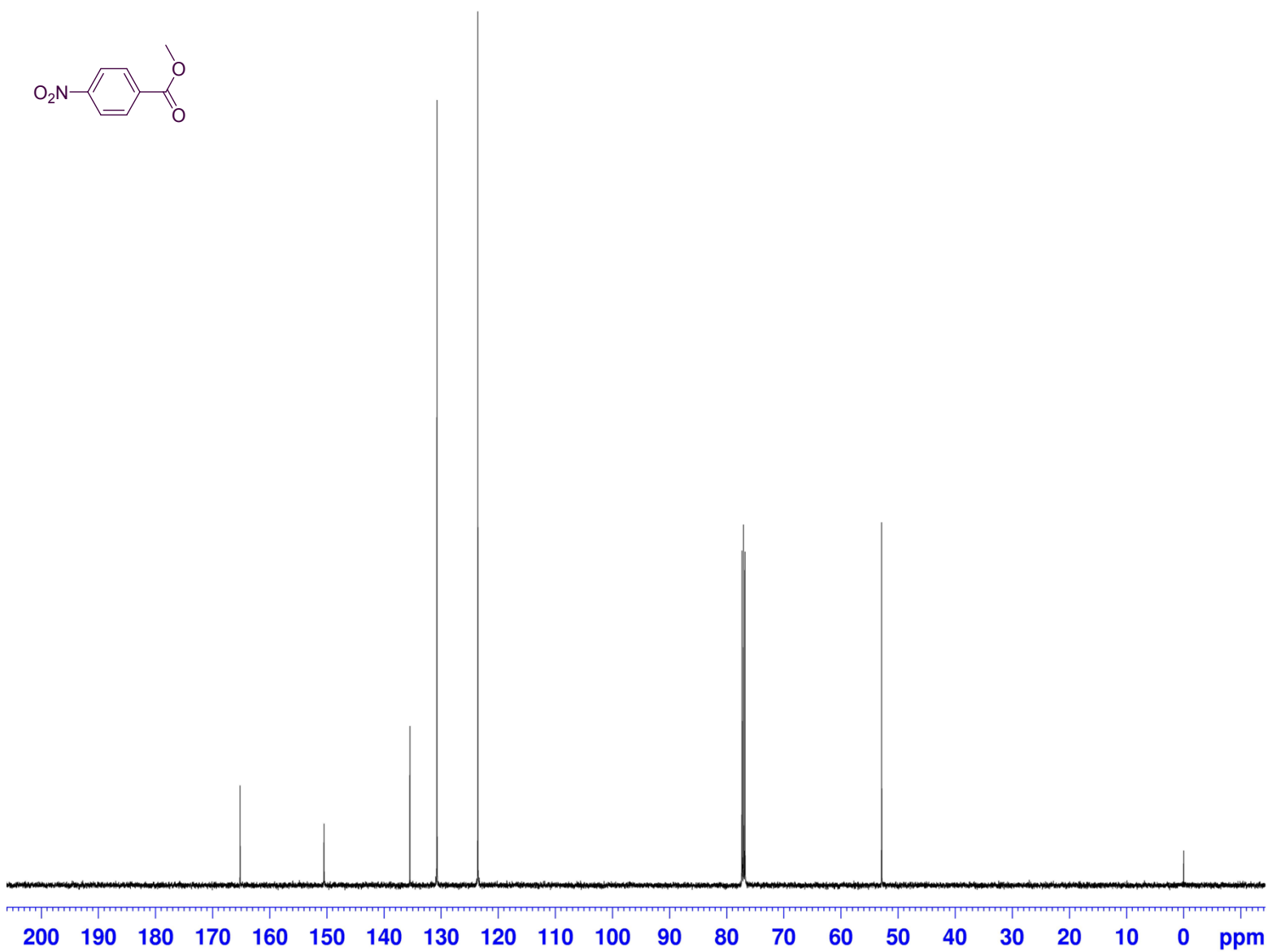
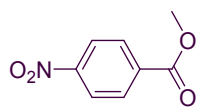


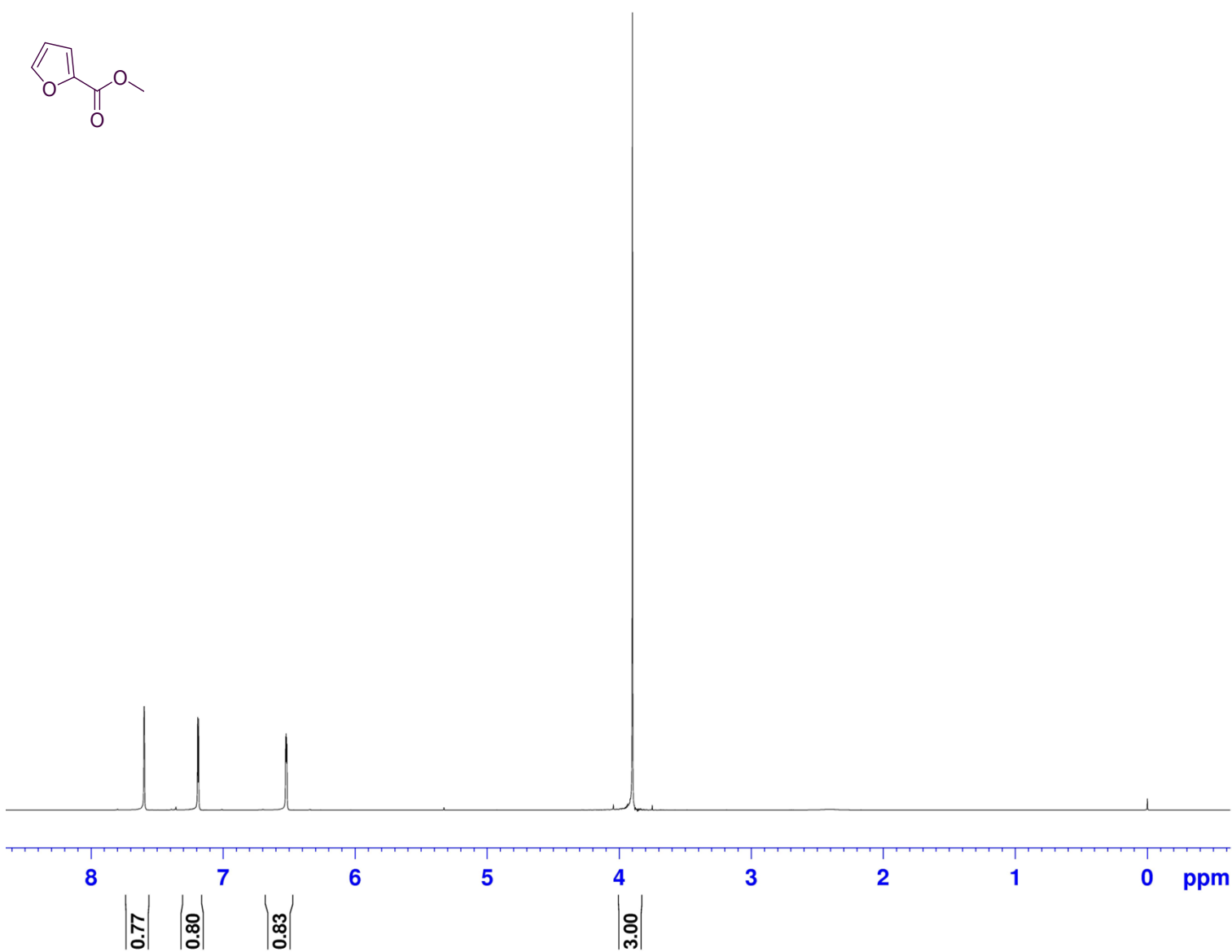
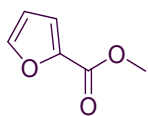


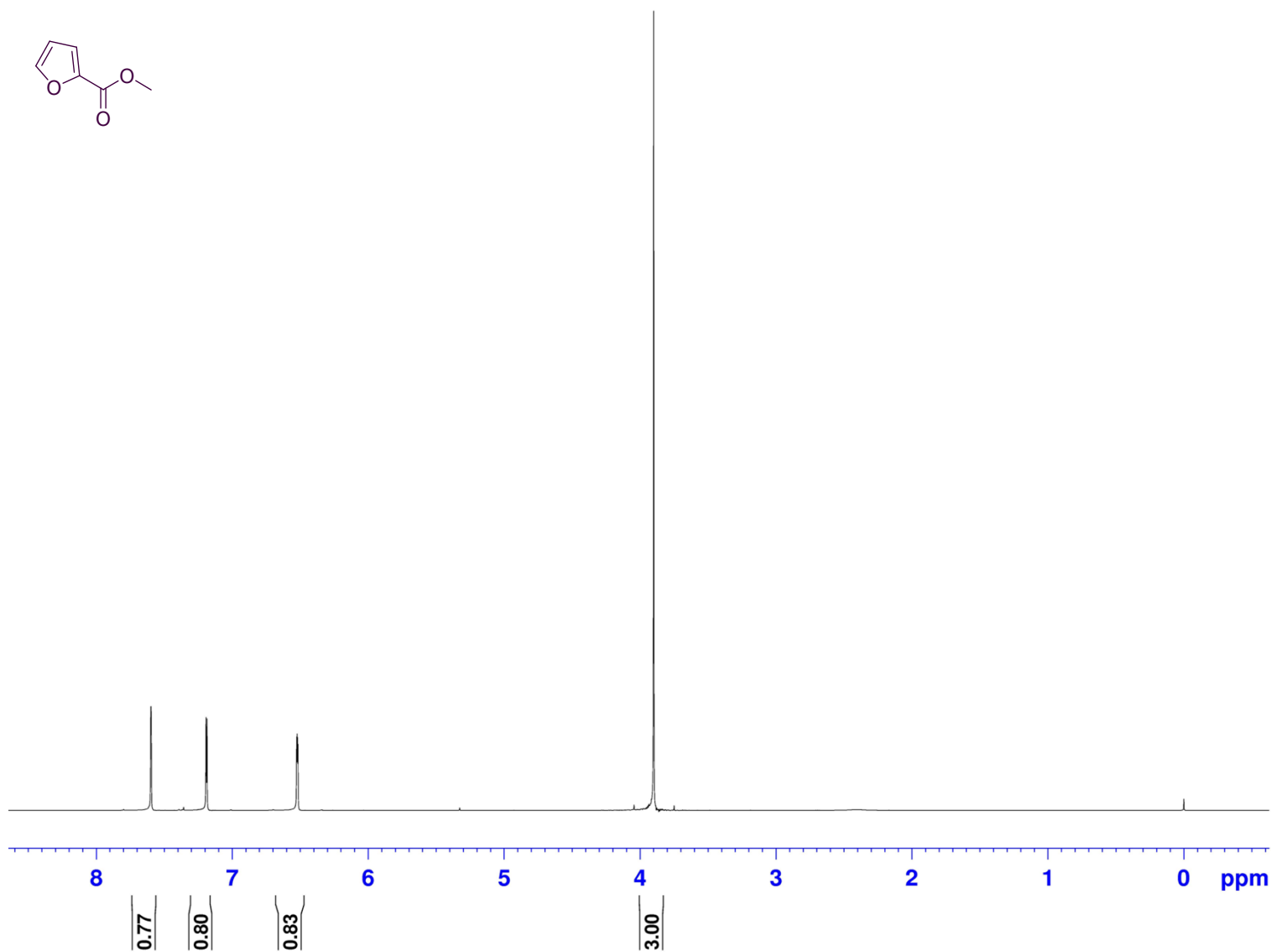
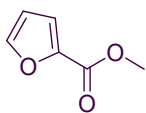




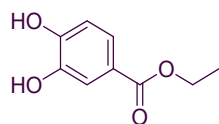








NS 08



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7.621  
7.614  
7.593  
7.587  
7.281  
6.943  
6.915

— 5.885

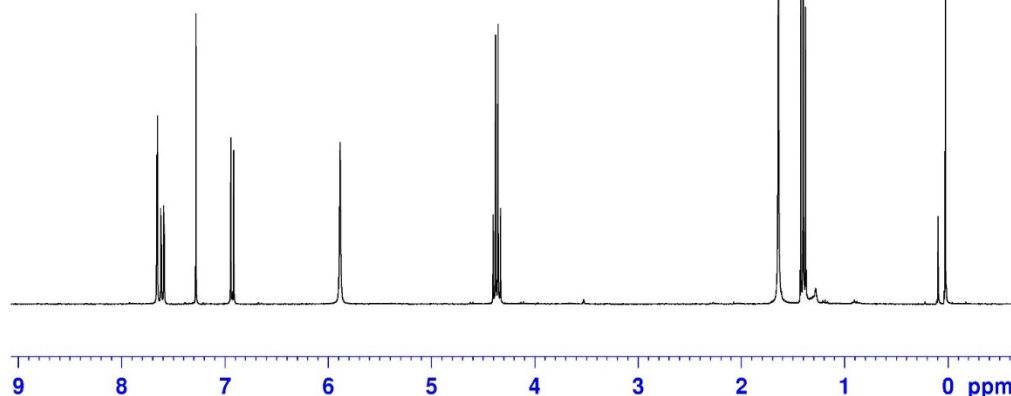
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4.355  
4.331

1.423  
1.400  
1.376



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Time 15.27  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg  
TD 32768  
SOLVENT CDCl3  
NS 32  
DS 2  
SWH 6188.119 Hz  
FIDRES 0.188846 Hz  
AQ 2.6477852 sec  
RG 456.1  
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  
SF01 300.1318534 MHz  
SI 32768  
SF 300.1300000 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



0.87  
0.85

0.89

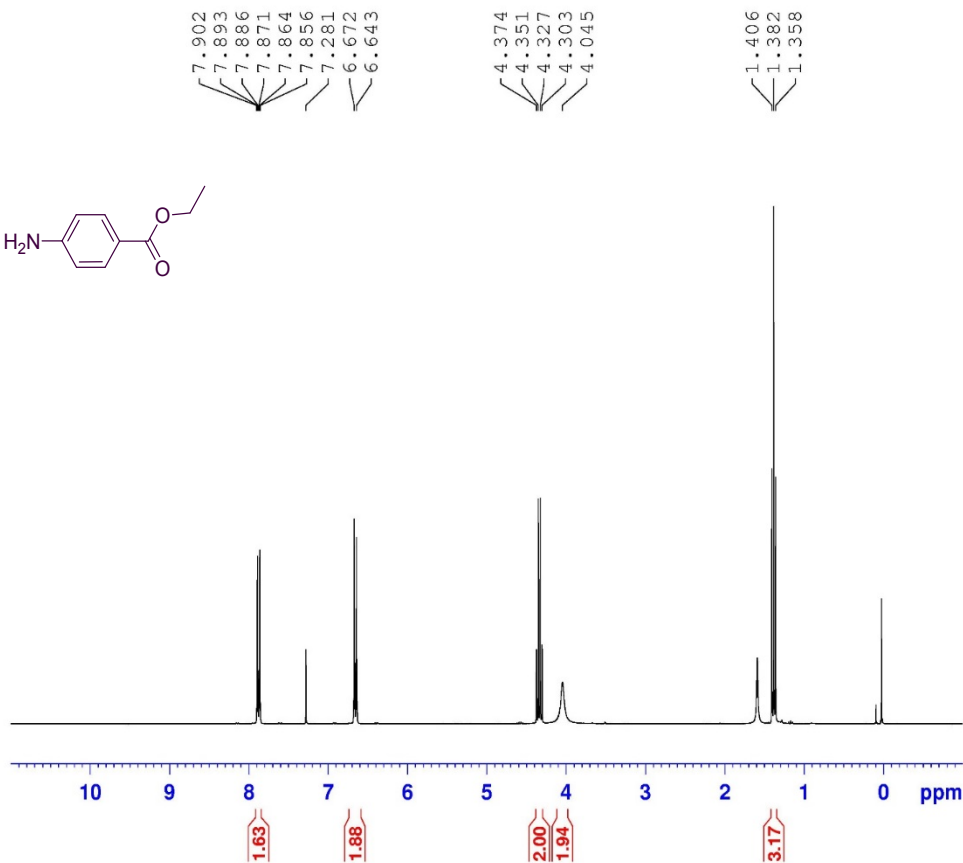
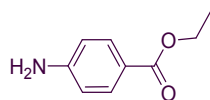
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2.04

3.00



NS 09

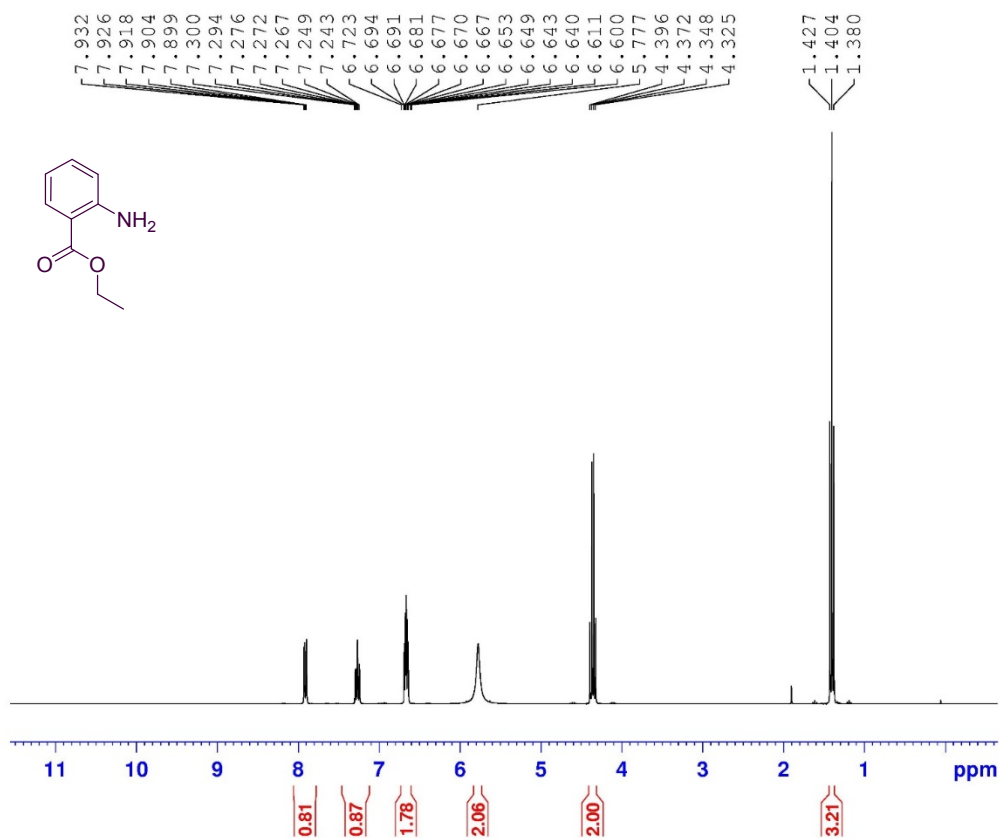
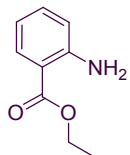


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TD            32768
SOLVENT       CDC13
NS            16
DS            2
SWH           6188.119 Hz
FIDRES        0.188846 Hz
AQ            2.6477852 sec
RG            322.5
DW            80.800 usec
DE            6.50 usec
TE            303.0 K
D1            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
  
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NS 10

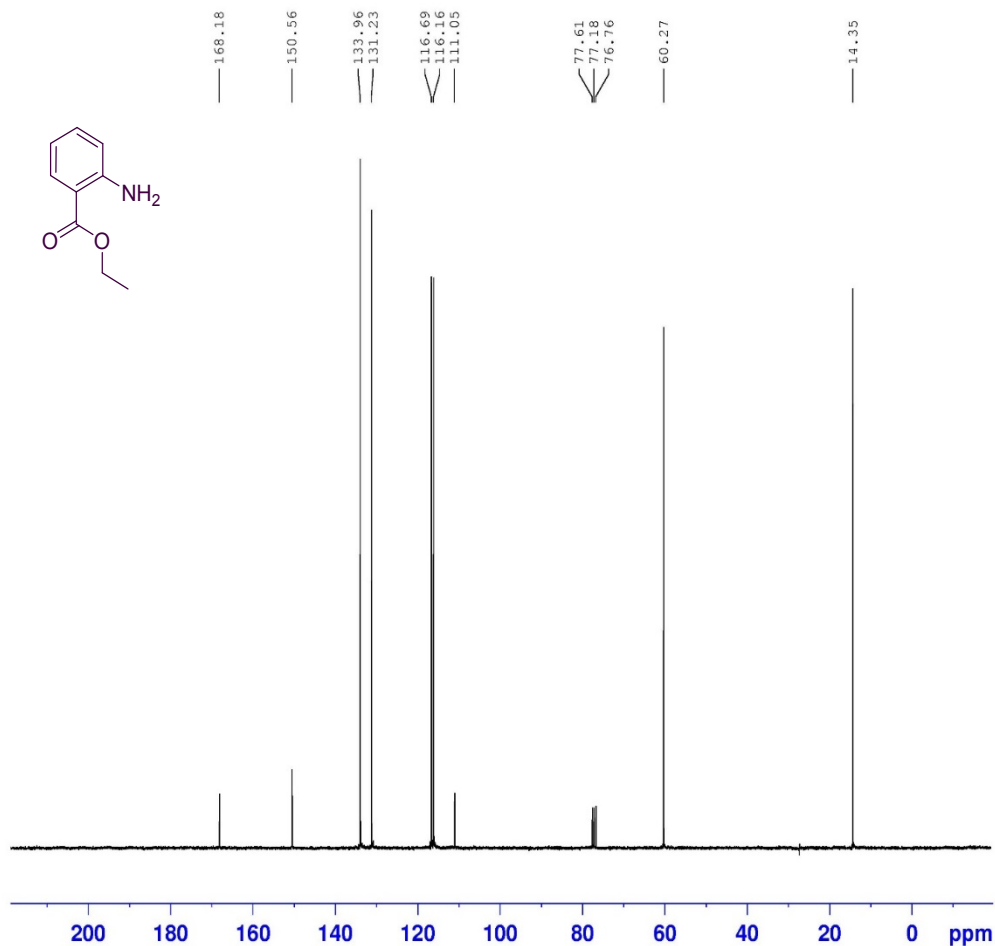
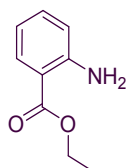


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EXPNO         1
PROCNO        1
Date_         20150821
Time          12.35
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg
TD            32768
SOLVENT       CDCl3
NS            8
DS            2
SWH           6188.119 Hz
FIDRES        0.188846 Hz
AQ            2.6477852 sec
RG            32
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
SF01          300.1318534 MHz
SI            32768
SF            300.1300000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

NS 10



```

NAME           NS 10
EXPNO           3
PROCNO          1
Date_          20150821
Time            13.30
INSTRUM         spect
PROBHD          5 mm BBO BB-1H
PULPROG         zgpg
TD              32768
SOLVENT         CDCl3
NS              1024
DS              4
SWH             17985.611 Hz
FIDRES          0.548877 Hz
AQ              0.9110282 sec
RG              35.9
DW              27.800 usec
DE              6.50 usec
TE              303.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0             1
  
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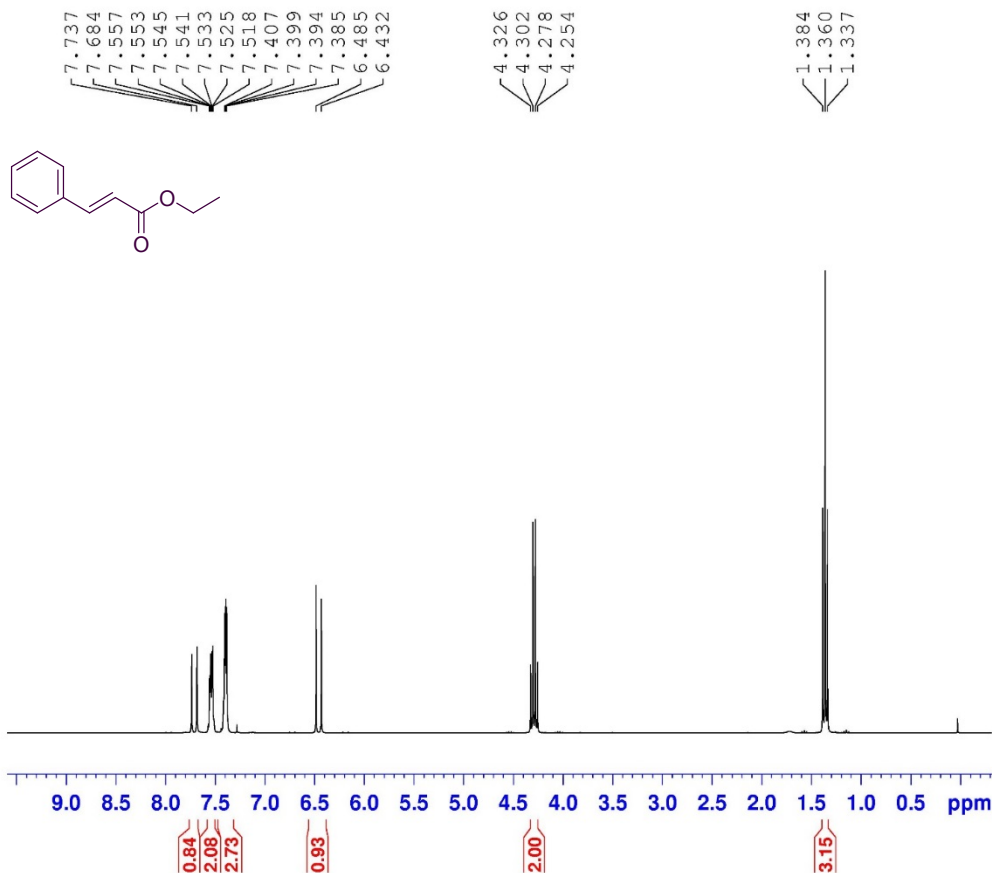
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===== CHANNEL f1 =====
NUC1             13C
P1              10.00 usec
PL1             1.80 dB
PL1W            49.78760910 W
SF01            75.4752953 MHz
  
```

```

===== 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
SF02            300.1312005 MHz
SI              32768
SF              75.4677490 MHz
WDW             EM
SSB             0
LB              1.00 Hz
GB              0
PC              1.40
  
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NS 11



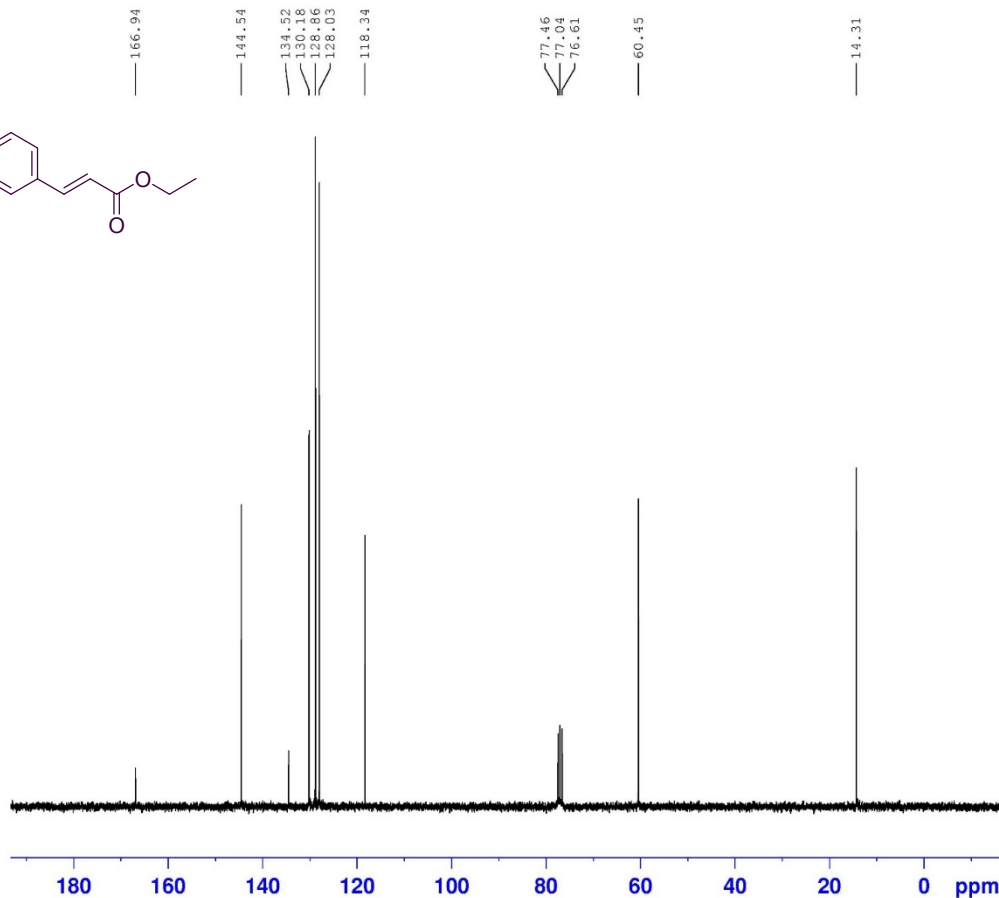
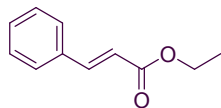
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PULPROG        zg
TD             32768
SOLVENT        CDCl3
NS             16
DS             2
SWH            6188.119 Hz
FIDRES         0.188846 Hz
AQ            2.6477852 sec
RG            114
DW            80.800 usec
DE            6.50 usec
TE            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
  
```

NS 11



```

NAME          NS 11
EXPNO         2
PROCNO        1
Date_         20150821
Time          12.24
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgig
TD            32768
SOLVENT       CDCl3
NS            300
DS            4
SWH           17985.611 Hz
FIDRES        0.548877 Hz
AQ            0.9110282 sec
RG            8192
DW            27.800 usec
DE            10.00 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
  
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