Combined Experimental and Computational Study of Al_2O_3 Catalyzed Transamidation of Secondary Amides with Amines.

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Supporting information

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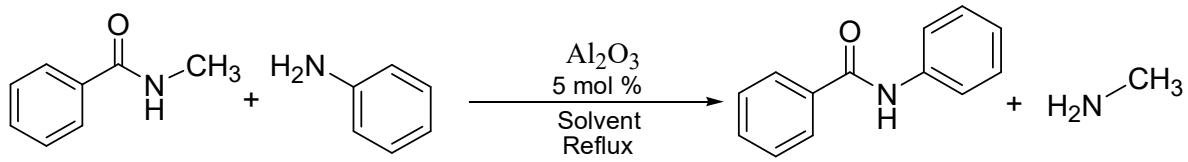
1. Synthesis of N-Phenyl Benzamide

For synthesis of N-Phenyl Benzamide, N- Methyl Benzamide (1 mmol) and aniline (1 mmol) were taken in a RB flask containing 5 mol% Al_2O_3 and then added 5 mL Triethyl amine in the mixture. This reaction mixture was heated on hot plate around at 100 °C for 30 h in a sand bath with continuous stirring of magnetic bar at 300 rpm. The progress of the reaction was monitored by TLC with n-hexane and chloroform. Completion of the reaction was confirmed by TLC. After the completion of the reaction, 4 ml 2-propanol was added to dissolve amides mixture. Then, Al_2O_3 was separated from the mixture by centrifugation followed by washing with acetone and dried at 100°C for 3 h. The recovered Al_2O_3 was reused for three cycles without a marked decrease in the yield of the product. The solvent was removed from mixture by rotatory evaporator. Finally, the amide was purified by column chromatography with chloroform and n-hexane and recrystallization separation technique with ethanol and water. For catalyst screening, solvent screening and temperature screening same reaction procedure were performed.

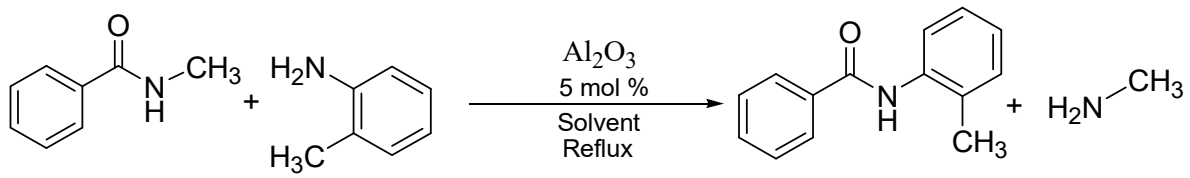


Scheme S1: Synthesis of N-Phenyl Benzamide.

- Molecular weight : 197 g/mol
- Molecular formula : $C_{13}NOH_{11}$
- Solubility : Soluble in Chloroform.
- FT-IR (υ KBr) : 3347, 3055, 1659, 1536, 1439, 1659, 1075 cm-1
- 1H-NMR (400 MHz, CDCl3) : δ 8.02 (br s, 1H, -NH), 7.088(m, 2H), 7.67 (m, 2H), 7.48 (t, 1H), 7.38 (t, 2H), 7.27(m, 2H), 7.17 (t, 1H)
- 13C-NMR (100 MHz, CDCl3) : δ 165.895 (1C, C=O), 137.969 (1C),135.010 (1C), 131.844 (1C), 129.100-129.438 (2C), 128.783-129.074 (2C),127.080 (2C), 124.550 (1C), 120.30 (2C)

2. Synthesis of N-(o-methyl) Phenyl Benzamide

For synthesis of N-(o-methyl)Phenyl Benzamide, N- Methyl Benzamide (1 mmol) and o-Toluedine (1 mmol) were taken in a RB flask containing 5 mol% Al_2O_3 and then added 5 mL Triethyl amine in the mixture. This reaction mixture was heated on hot plate around at 100 °C for 30 hrs in a sand bath with continuous stirring of magnetic bar at 300 rpm. The progress of the reaction was monitored by TLC with n-hexane and chloroform. Completion of the reaction was confirmed by TLC. After the completion of the reaction, 4 ml 2-propanol was added to dissolve amides mixture. Then, Al_2O_3 was separated from the mixture by centrifugation followed by washing with acetone and dried at 100°C for 3 hrs. The recovered Al_2O_3 was reused for three cycles without a marked decrease in the yield of the product. The solvent was removed from mixture by rotatory evaporator. Finally, the amide was purified by column chromatography with chloroform and n-hexane and recrystallization separation technique with ethanol and water. For catalyst screening, solvent screening and temperature screening same reaction procedure were performed.

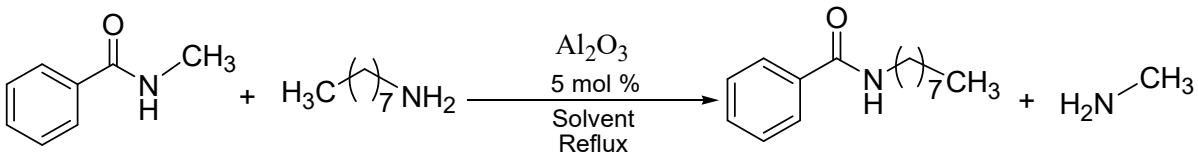


Scheme S2: Synthesis of N-(o-methyl)Phenyl Benzamide.

- Molecular weight : 212 g/mol
- Molecular formula : $C_{14}NOH_{14}$
- Solubility : Soluble in Chloroform.
- FT-IR (υ KBr) : 3227, 3060, 1645, 1592, 1434, 1294, 1074cm-1
- 1H-NMR (400 MHz, CDCl3) : δ 8.067 (br s, 1H, -NH), 7.86-7.88 (m, 2H), 7.53-7.56 (t, 2H), 7.44-7.47 (t, 3H), 7.23-7.28 (m, 2H), 6.69-6.99 (m, 1H), 2.36 (s, 3H)
- 13C-NMR (100 MHz, CDCl3) : δ 165.924 (1C, C=O), 138.989 (1C), 137.913 (1C), 131.764 (1C), 129.391 (1C), 128.86 (1C), 128.729 (2C), 125.401 (1C), 127.092 (2C), 121.031 (1C), 117.462 (1C), 21.51 (1C).

3. Synthesis of N-n-Octyl Benzamide

For synthesis of N-n-Octyl Benzamide, N- Methyl Benzamide (1 mmol) and n-Octyl amine (1 mmol) were taken in a RB flask containing 5 mol% Al_2O_3 and then added 5 mL Triethyl amine in the mixture. This reaction mixture was heated on hot plate around at 100 °C for 30 hrs in a sand bath with continuous stirring of magnetic bar at 300 rpm. The progress of the reaction was monitored by TLC with n-hexane and chloroform. Completion of the reaction was confirmed by TLC. After the completion of the reaction, 4 ml 2-propanol was added to dissolve amides mixture. Then, Al_2O_3 was separated from the mixture by centrifugation followed by washing with acetone and dried at 100°C for 3 hrs. The recovered Al_2O_3 was reused for three cycles without a marked decrease in the yield of the product. The solvent was removed from mixture by rotatory evaporator. Finally, the amide was purified by column chromatography with chloroform and n-hexane and recrystallization separation technique with ethanol and water. For catalyst screening, solvent screening and temperature screening same reaction procedure were performed.

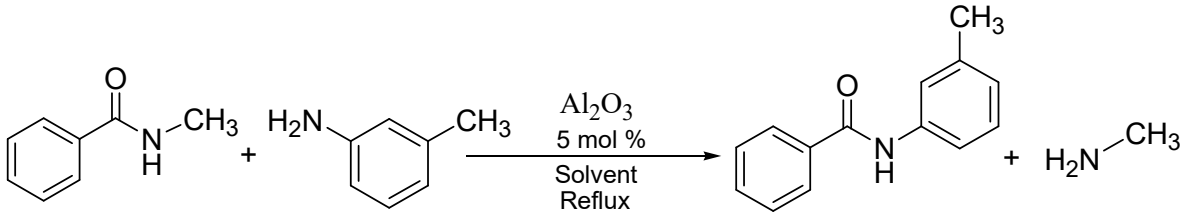


Scheme S3: Synthesis of N-Octyl Benzamide.

- Molecular weight : 233 g/mol
- Molecular formula : $C_{15}NOH_{23}$
- Solubility : Soluble in Chloroform.
- FT-IR (υ KBr) : 3360, 3058, 1653, 1549, 1493, 1447, 1076cm-1
- 1H-NMR (400 MHz, CDCl3) : δ 6.368 (br s, 1H, -NH), 7.77-7.78 (m, 2H), 7.47-7.51 (t, 1H), 7.40-7.44 (t, 2H), 3.42-3.47 (m, 2H), 2.362-2.398 (m, 2H), 1.58-1.65 (m, 2H), 1.28-1.34 (m, 8H), 0.87-.91 (t, 3H)
- 13C-NMR (100 MHz, CDCl3) : δ 167.626 (1C, C=O), 134.811 (1C), 131.313 (1C), 128.525 (1C), 126.889 (1C), 40.181 (1C), 31.806 (1C), 29.675 (1C), 29.225 (1C), 29.308 (1C), 27.029 (1C), 14.097 (1C).

4. Synthesis of N-(m-methyl) Phenyl Benzamide

For synthesis of N-(m-methyl)Phenyl Benzamide, N- Methyl Benzamide (1 mmol) and m-Toluedine (1 mmol) were taken in a RB flask containing 5 mol% Al_2O_3 and then added 5 mL Triethyl amine in the mixture. This reaction mixture was heated on hot plate around at 100 °C for 30 hrs in a sand bath with continuous stirring of magnetic bar at 300 rpm. The progress of the reaction was monitored by TLC with n-hexane and chloroform. Completion of the reaction was confirmed by TLC. After the completion of the reaction, 4 ml 2-propanol was added to dissolve amides mixture. Then, Al_2O_3 was separated from the mixture by centrifugation followed by washing with acetone and dried at 100°C for 3 hrs. The recovered Al_2O_3 was reused for three cycles without a marked decrease in the yield of the product. The solvent was removed from mixture by rotatory evaporator. Finally, the amide was purified by column chromatography with chloroform and n-hexane and recrystallization separation technique with ethanol and water. For catalyst screening, solvent screening and temperature screening same reaction procedure were performed.

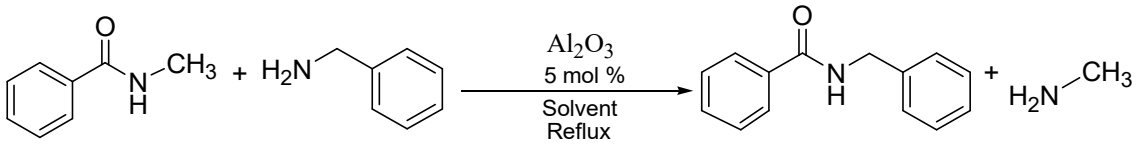


Scheme S4: Synthesis of N-(o-methyl)Phenyl Benzamide.

- Molecular weight : 212 g/mol
- Molecular formula : $C_{14}NOH_{14}$
- Solubility : Soluble in Chloroform.
- FT-IR (υ KBr) : 3240, 3030, 2829, 1651, 1603, 1526, 1440cm-1
- 1H-NMR (400 MHz, CDCl3) : δ 7.722 (br s, 1H, -NH), 7.962-7.982 (m, 1H), 7.907-7.925 (m, 1H), 7.576-7.612 (t, 1H), 7.50-7.54 (m, 3H), 7.24-7.27 (m, 1H), 7.13 (m, 1H), 2.36 (s, 3H)
- 13C-NMR (100 MHz, CDCl3) : δ 165.683 (1C, C=O), 135.789 (1C), 135.039 (1C), 131.885 (1C), 130.591 (1C), 129.244 (1C), 128.873-128.918 (2C), 127.074 (1C), 126.947 (1C), 125.399 (1C), 123.139 (1C), 17.860 (1C).
-

5. Synthesis of N-Benzyl Benzamide

For synthesis of N-Benzyl Benzamide, N- Methyl Benzamide (1 mmol) and Benzylamine (1 mmol) were taken in a RB flask containing 5 mol% Al_2O_3 and then added 5 mL Triethyl amine in the mixture. This reaction mixture was heated on hot plate around at 100 °C for 30 hrs in a sand bath with continuous stirring of magnetic bar at 300 rpm. The progress of the reaction was monitored by TLC with n-hexane and chloroform. Completion of the reaction was confirmed by TLC. After the completion of the reaction, 4 ml 2-propanol was added to dissolve amides mixture. Then, Al_2O_3 was separated from the mixture by centrifugation followed by washing with acetone and dried at 100°C for 3 hrs. The recovered Al_2O_3 was reused for three cycles without a marked decrease in the yield of the product. The solvent was removed from mixture by rotatory evaporator. Finally, the amide was purified by column chromatography with chloroform and n-hexane and recrystallization separation technique with ethanol and water. For catalyst screening, solvent screening and temperature screening same reaction procedure were performed.



Scheme S5: Synthesis of N-Benzyl Benzamide.

- Molecular weight : 212 g/mol
- Molecular formula : $C_{14}NOH_{13}$
- Solubility : Soluble in Chloroform.
- FT-IR (υ KBr) : 3329, 3056, 2940, 1639, 1578, 1555, 1492 cm-1
- 1H-NMR (400 MHz, CDCl3) : δ 9.270 (br s, 1H, -NH), 5.150 (s, 1H), 4.460 (s, 1H), 6.825-6.834 (m, 2H), 7.396-7.404 (m, 2H), 7.185-7.283 (m, 1H), 8.001-8.183 (m, 2H), 7.597-7.641 (m, 2H), 7.826-7.844 (m, 1H)

6. Catalyst Screening

Model reaction:

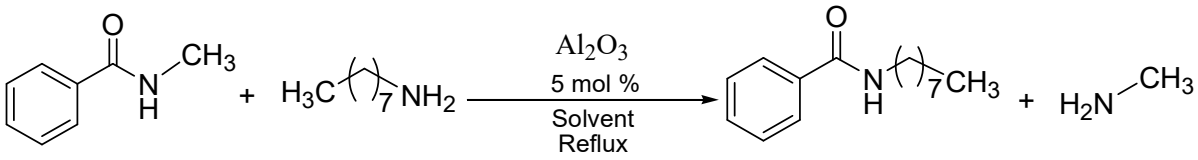


Table S1: Catalyst screening for model reaction

Entry	**Catalyst**	**% of Yields**
01	**-**	**0**
02	SnO_2	5
03	Cu_2O	3
04	Al_2O_3	76
05	Nb_2O_5	8
06	CeO_2	10
07	TiO_2	6

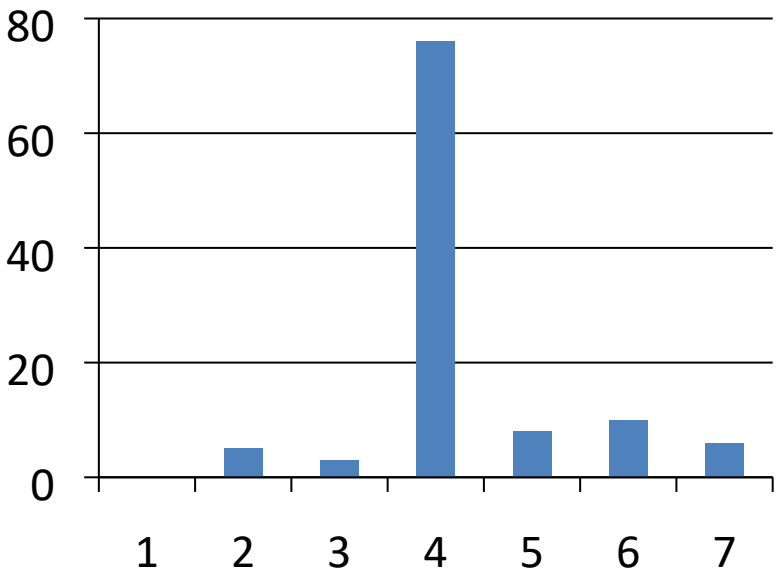


Figure S1: Catalyst screening for model reaction.

7. Solvent Screening

Model reaction.

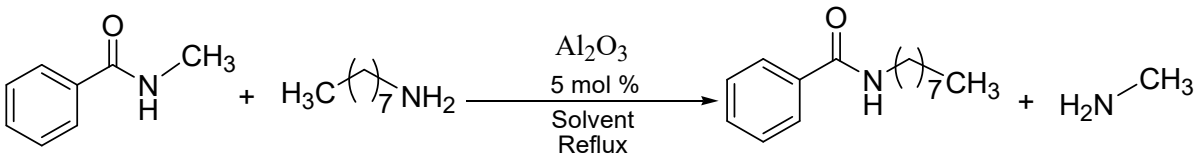


Table S2: Solvent screening for model reaction

Entry	**Solvent**	**% of Yields**
01	**No solvent**	**0**
02	***O*-Xylene**	**9**
03	**Benzene**	**5**
04	**Triethylamine**	**76**
05	**Toluene**	**7**

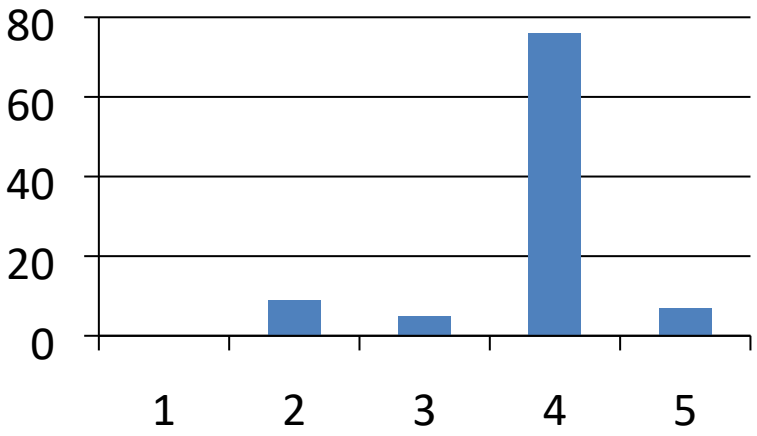


Figure S2: Solvent screening for model reaction.

8. Reusability of Al_2O_3

Model Reaction:

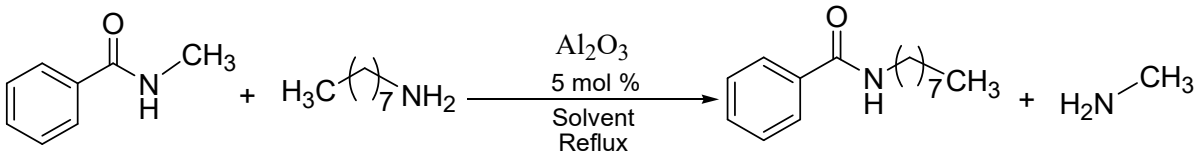


Table S3: Reusability of Al_2O_3 for model reaction.

Cycle Number	Catalyst	% of Yields
01	Al_2O_3	76
02		70
03		63
04		41

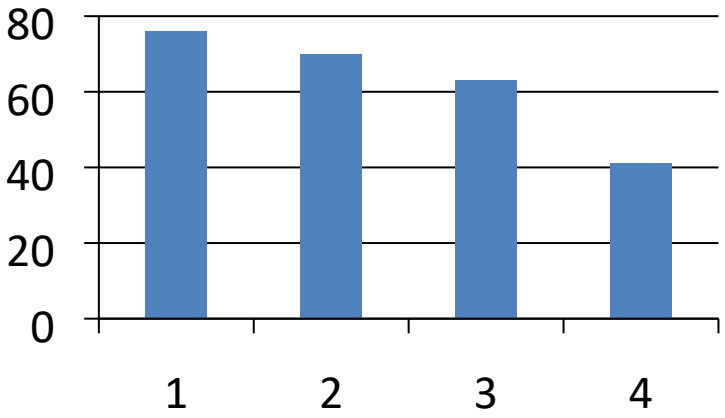
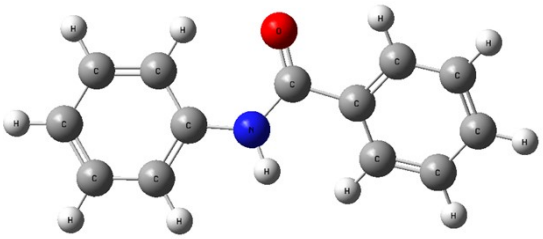
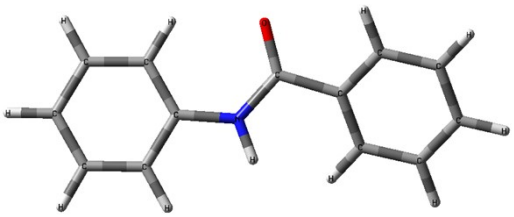
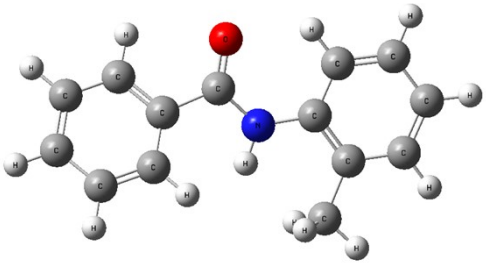
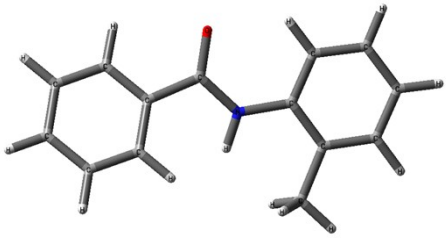
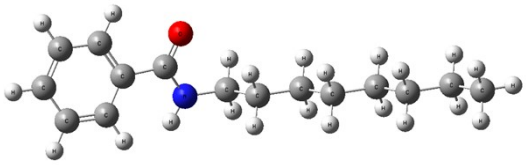
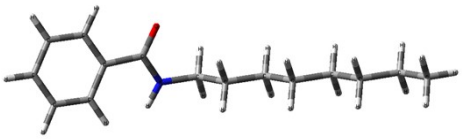
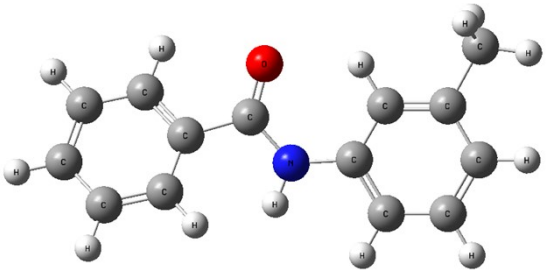
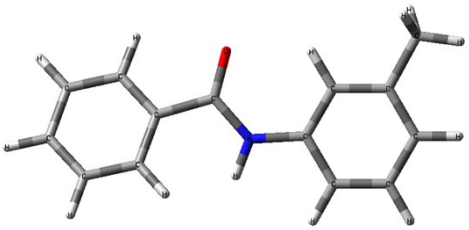
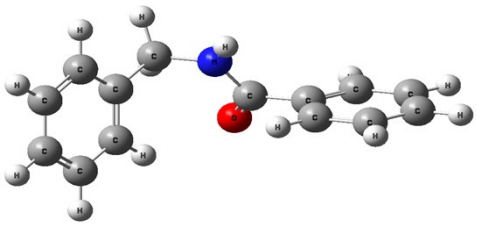
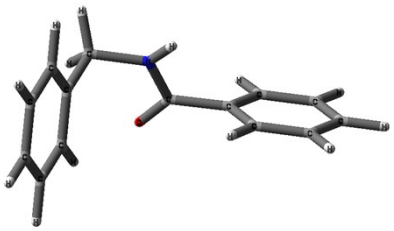


Figure S3: Reusability of Al_2O_3 for model reaction.

9. Optimize structure of Compounds 1-5

Table S4: Optimize Structure of Compounds 1-5

Compnd	Optimize ball and bond type Structure	Optimize tube Structure	Optimize Energy (Hartee)
1			-624.166
2			-663.353
3			-706.818
4			-663.355
5			-663.351

10. DFT Calculation Data

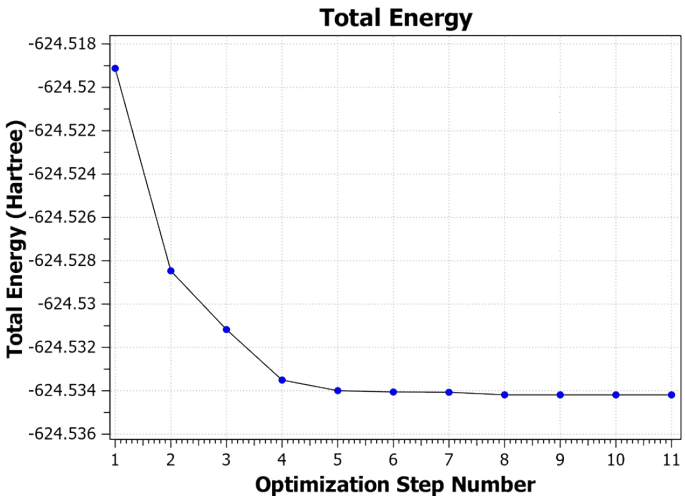
Table S5: Using Catalyst Al_2O_3

Calculation Method	Basis Set	Compounds	Energy (Hartee)	Relative Energy
RB3LYP	6-311+G (D, P)	Amides	-440.380	0.000
RB3LYP	6-311+G (D, P)	Amines-1	-287.631	152.749
RB3LYP	6-311+G (D, P)	Catalyst Al_2O_3	-710.733	-270.353
RB3LYP	6-311+G (D, P)	Solvent $N(C_2H_5)_3$	-174.528	265.853
RB3LYP	6-311+G (D, P)	TS1	-1438.224	-997.844
RB3LYP	6-311+G (D, P)	TS2	-1603.740	-1163.359
RB3LYP	6-311+G (D, P)	Product 1	-624.166	-191.786

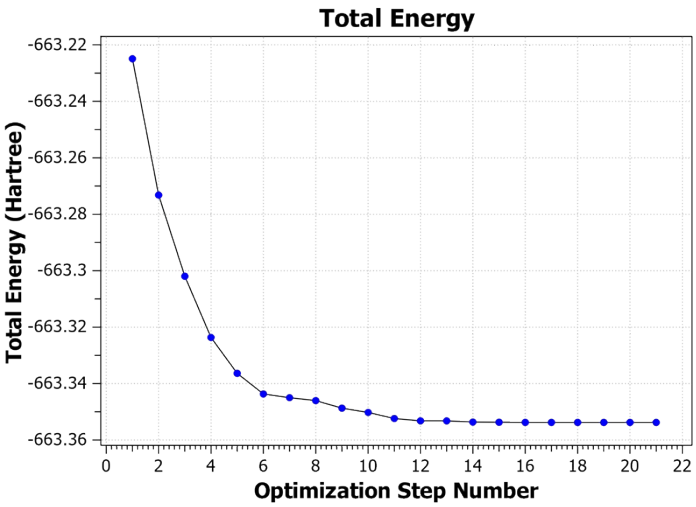
Table S6: Using Catalyst Nb_2O_5

Calculation Method	Basis Set	Compounds	Energy (Hartee)	Relative Energy
RB3LYP	6-311+G (D, P)	Amides	-440.380	0.000
RB3LYP	6-311+G (D, P)	Amines-1	-287.631	152.749
RB3LYP	6-311+G (D, P)	Catalyst Nb_2O_5	-7843.992	-7403.612
RB3LYP	6-311+G (D, P)	Solvent $N(C_2H_5)_3$	-174.528	265.853
RB3LYP	6-311+G (D, P)	TS1	-0.430672	439.950
RB3LYP	6-311+G (D, P)	TS2	-8852.090	-8411.710
RB3LYP	6-311+G (D, P)	Product 1	-624.166	-191.786

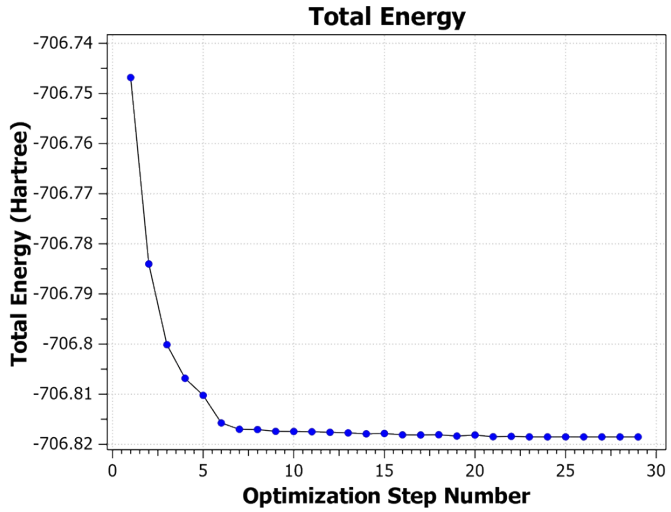
Total energy plot for compounds 1-5



Compound 1



Compound 2



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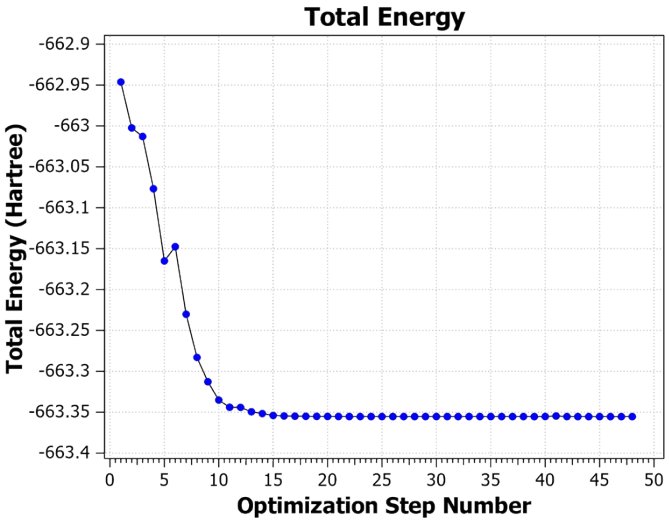

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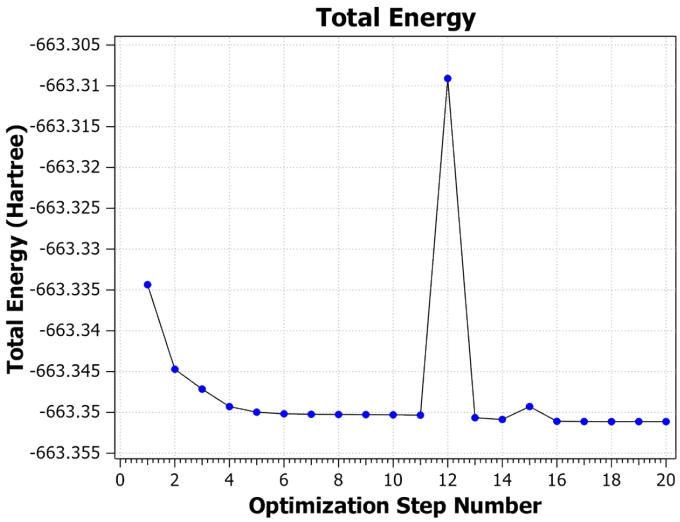


Compound 5

11. Spectra of compound 1

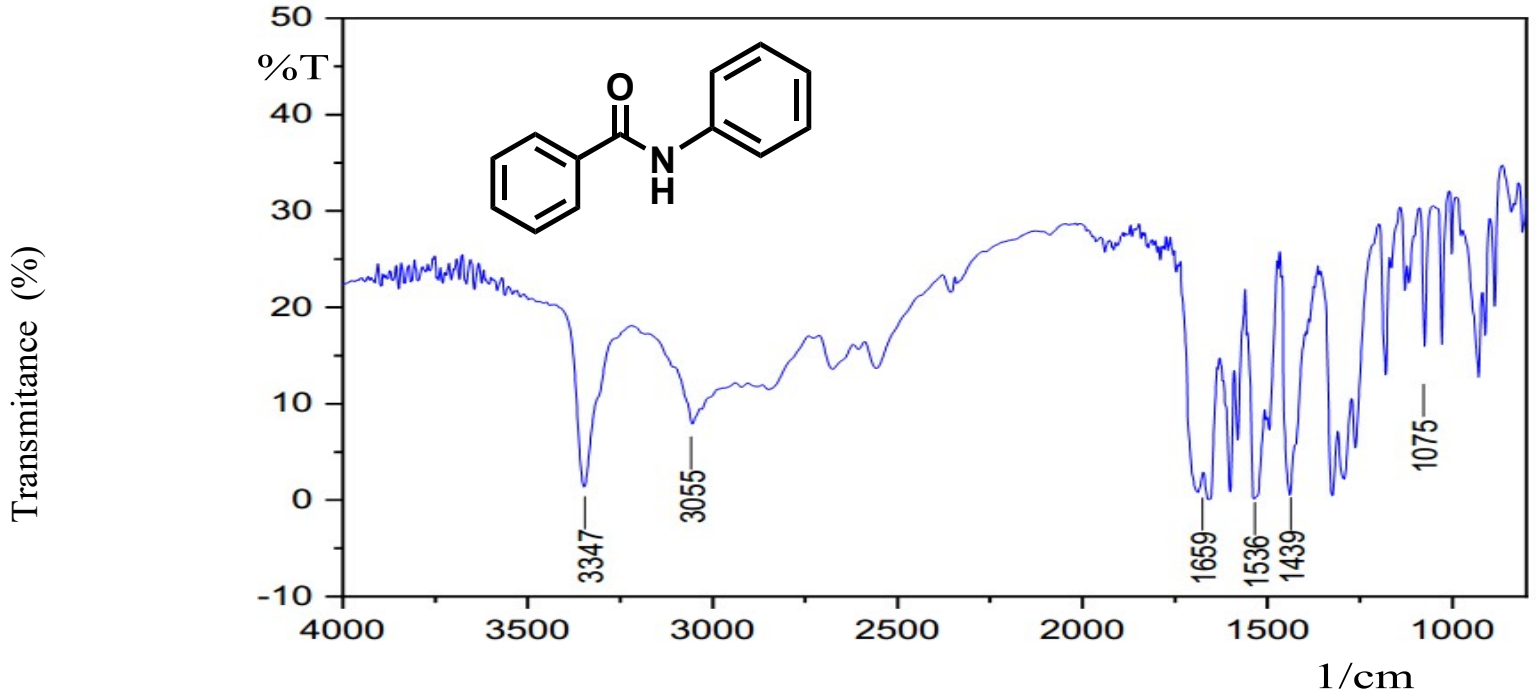
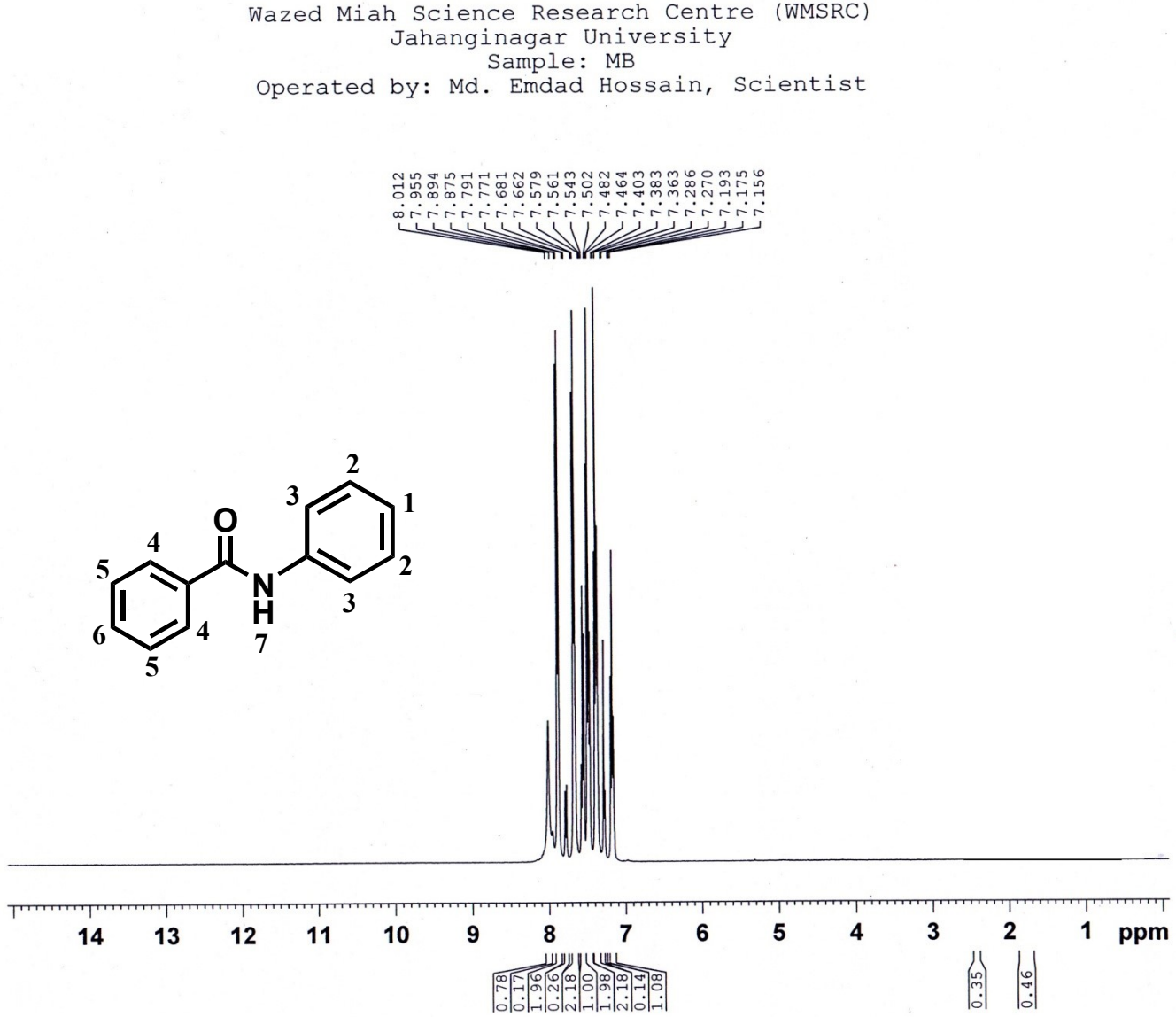


Figure S4: FT-IR spectrum of Compound 1



BRUKER

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PROCNO                      1

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DE                  6.50 usec
TE                 298.0 K
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TD0                      1

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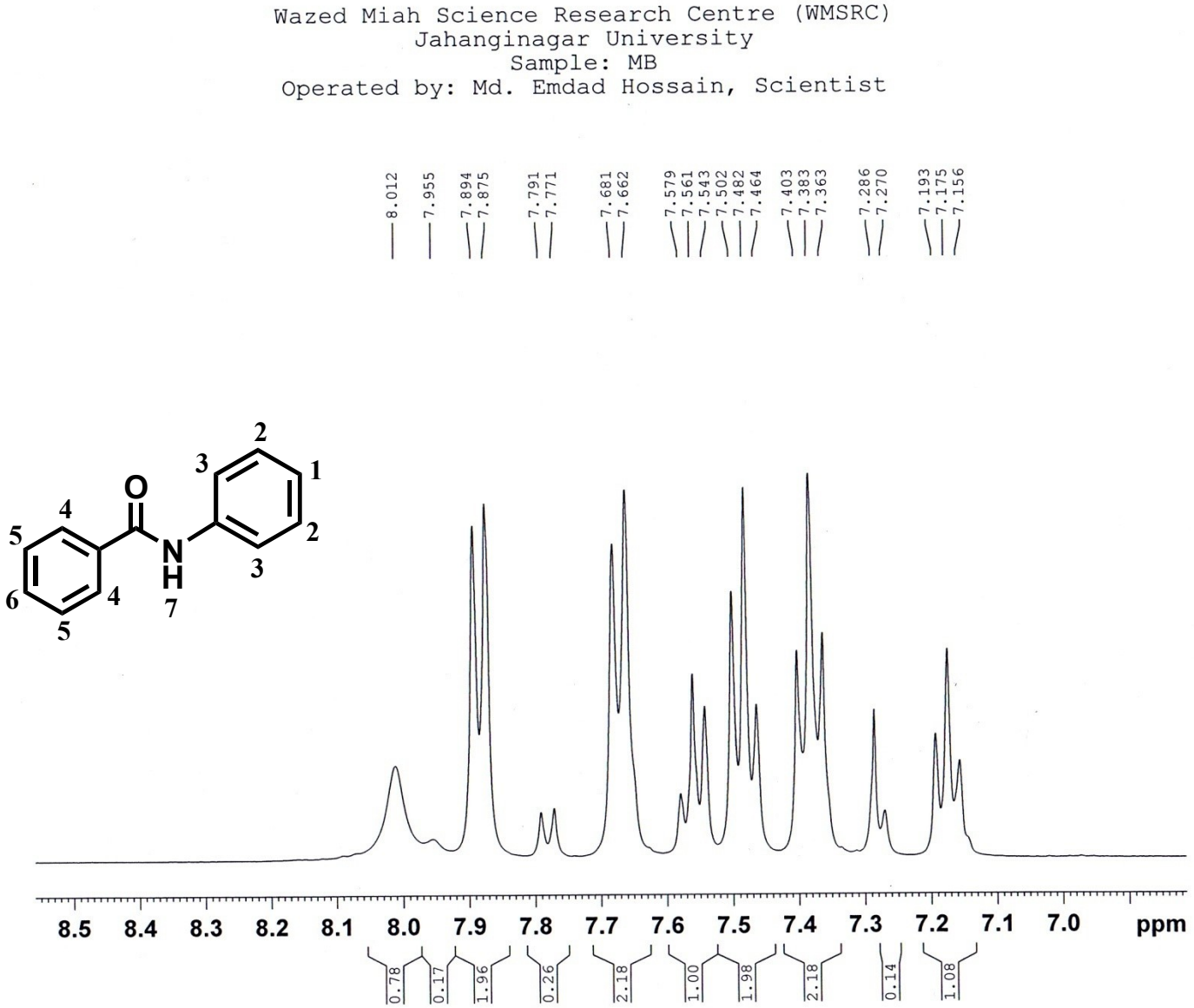
P1                  11.20 usec
PLW1           12.00000000 W

F2 - Processing parameters
SI                   131072
SF              400.2300000 MHz
WDW                     EM
SSB         0
LB                   1.00 Hz
GB          0
PC                   1.00
```

Figure S5: ^{1}H-NMR spectrum of compound 1




```
Current Data Parameters
NAME                  BUET_MB
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20200223
Time                   11.36
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                   zg
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         0
SWH               12019.230 Hz
FIDRES             0.183399 Hz
AQ                 2.7262976 sec
RG                     66.24
DW                    41.600 usec
DE                     6.50 usec
TE                    298.0 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1            400.2340023 MHz
NUC1                      1H
P1                     11.20 usec
PLW1           12.00000000 W

F2 - Processing parameters
SI                    131072
SF              400.2300000 MHz
WDW                       EM
SSB       0
LB                     1.00 Hz
GB        0
PC                     1.00
```

Figure S6: Extended ^{1}H-NMR spectrum of compound 1.

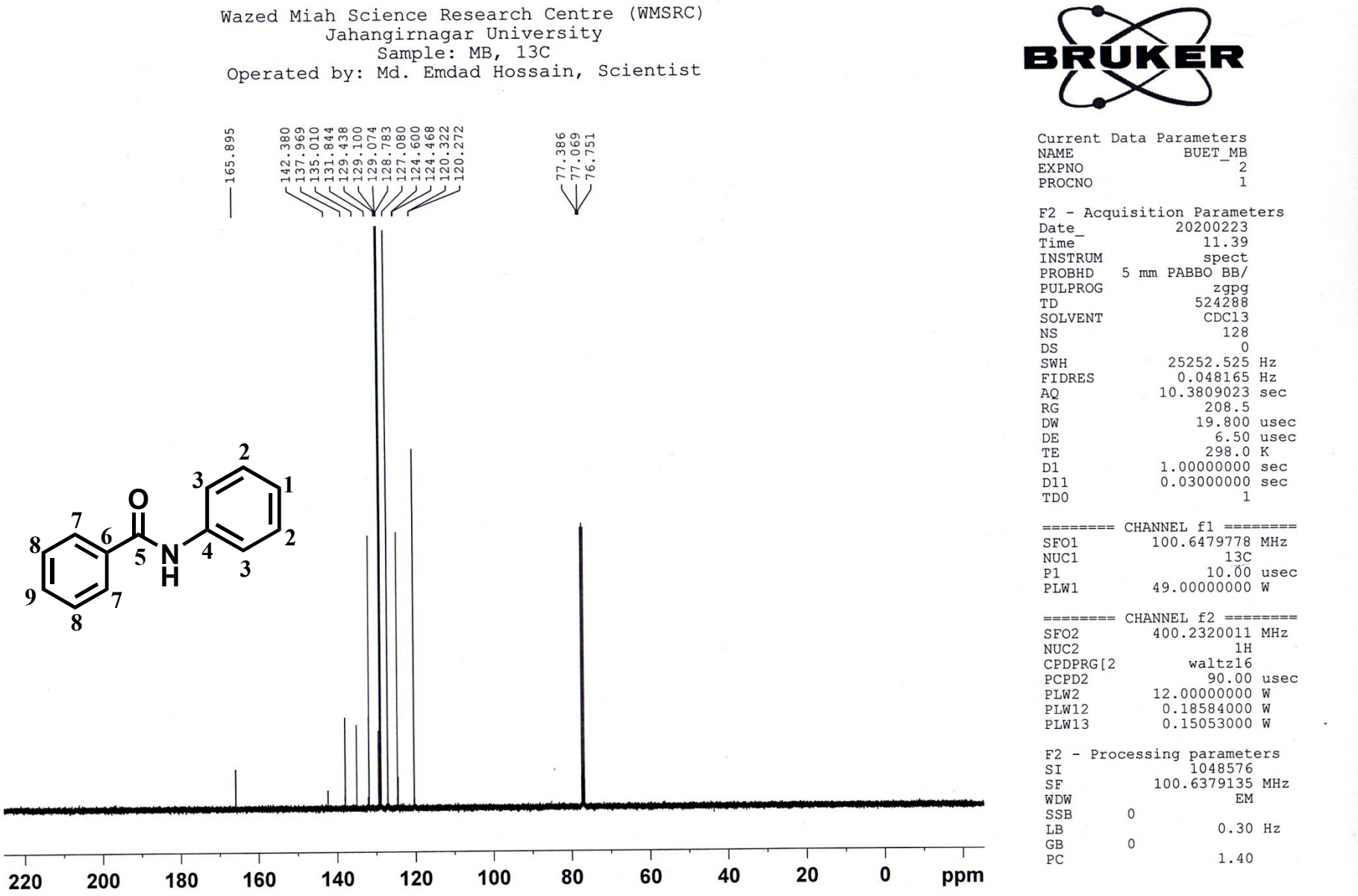


Figure S7: ^{13}C-NMR spectrum of compound 1.

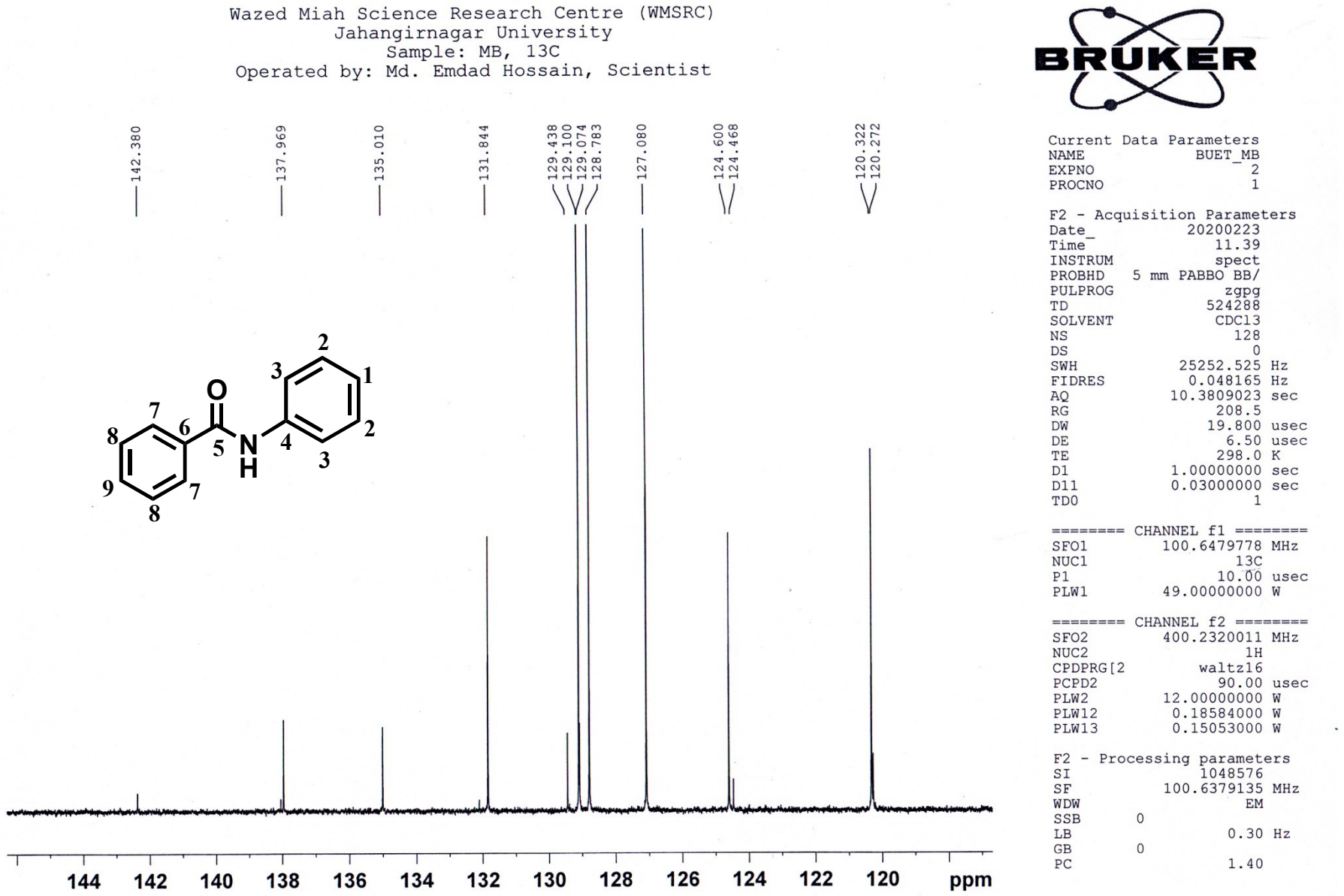


Figure S8: Extended ^{13}C-NMR spectrum of compound 1.

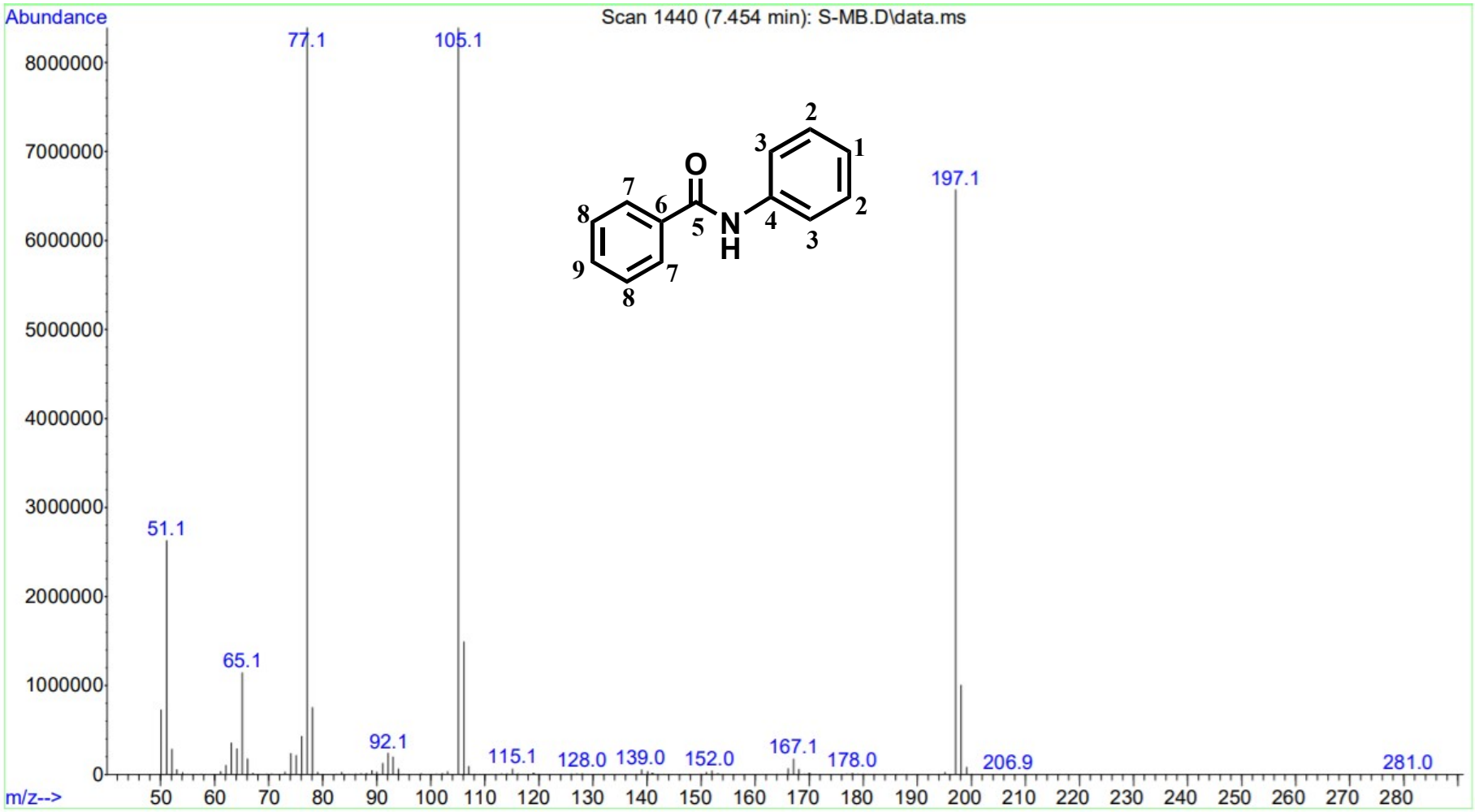


Figure S9: GC-MS spectrum of compound 1.

12. Spectra of compound 2

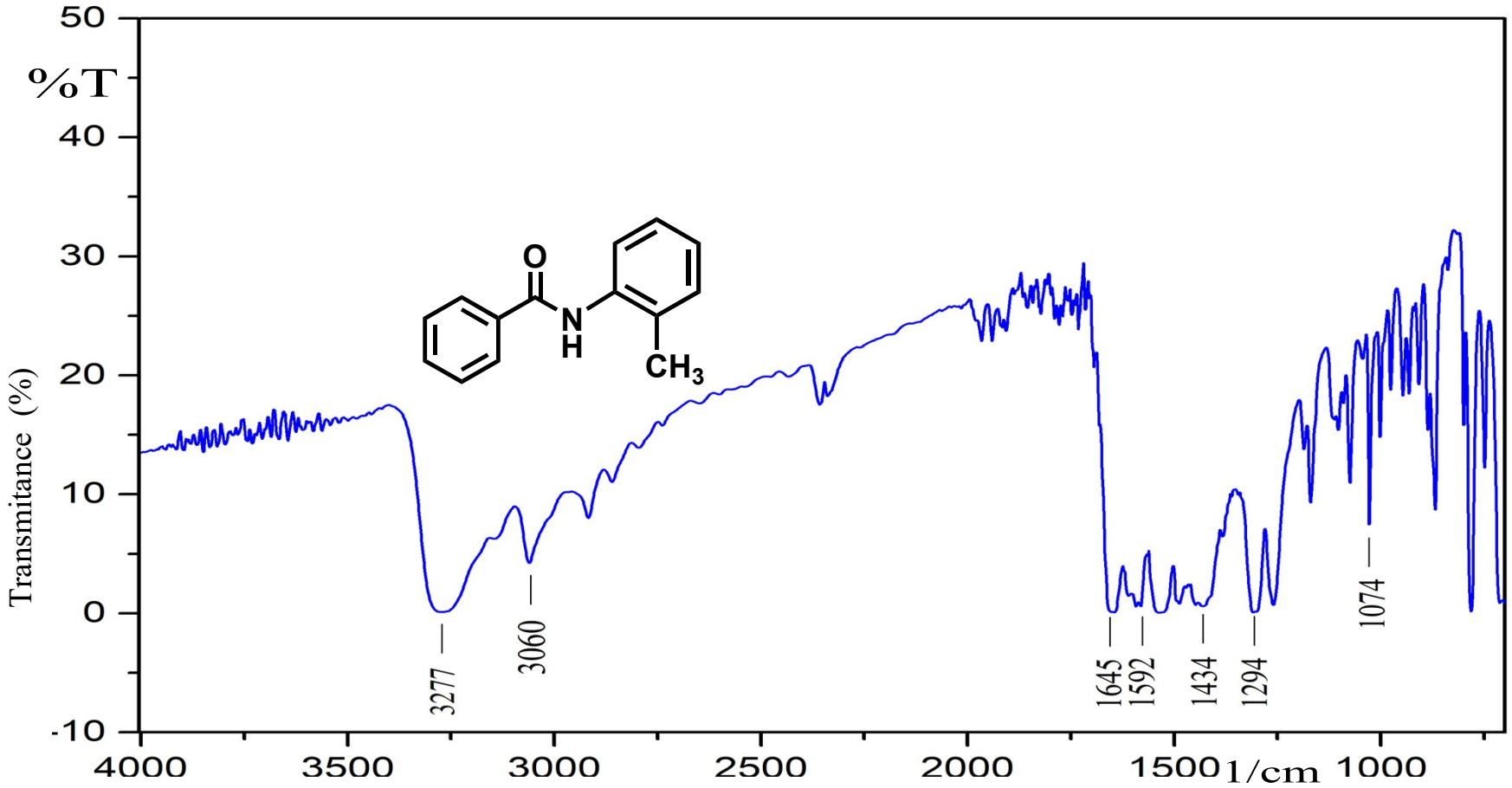


Figure S10: FT-IR spectrum of compound 2

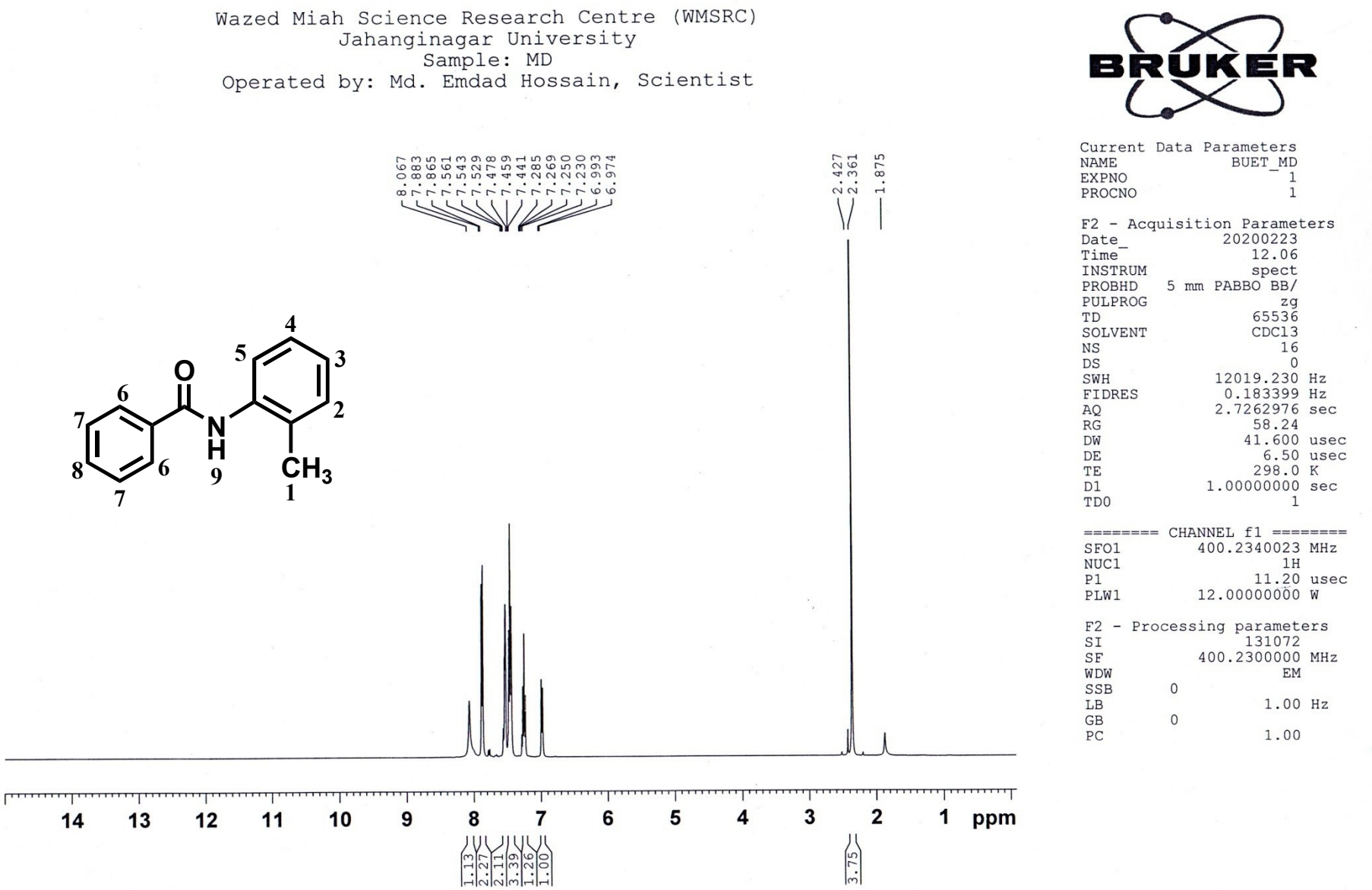


Figure S11: ^{1}H-NMR spectrum of compound 2

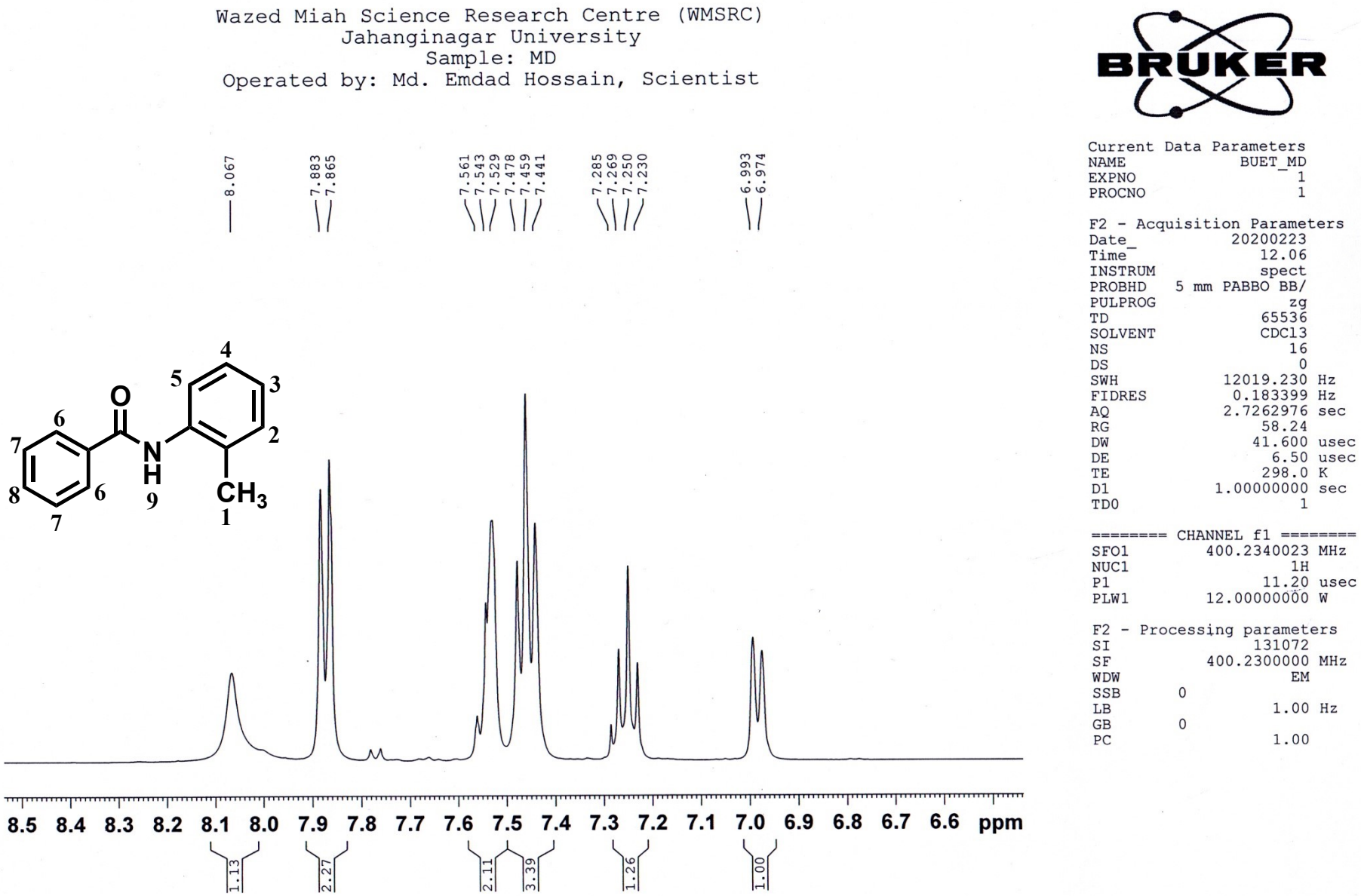
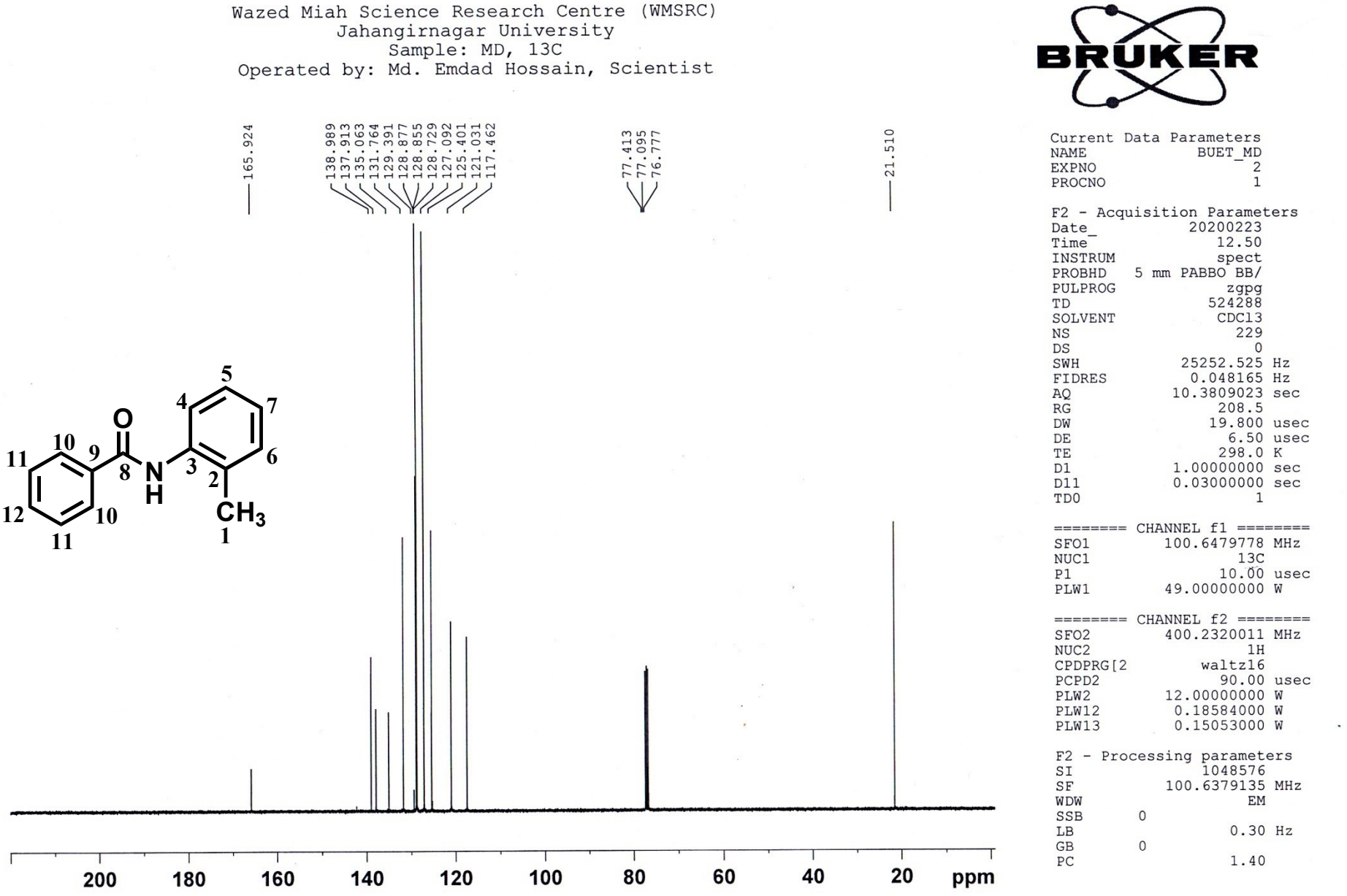


Figure S12: Extended ^{1}H-NMR spectrum of compound 2



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```
Current Data Parameters
NAME                  BUET_MD
EXPNO                       2
PROCNO                      1

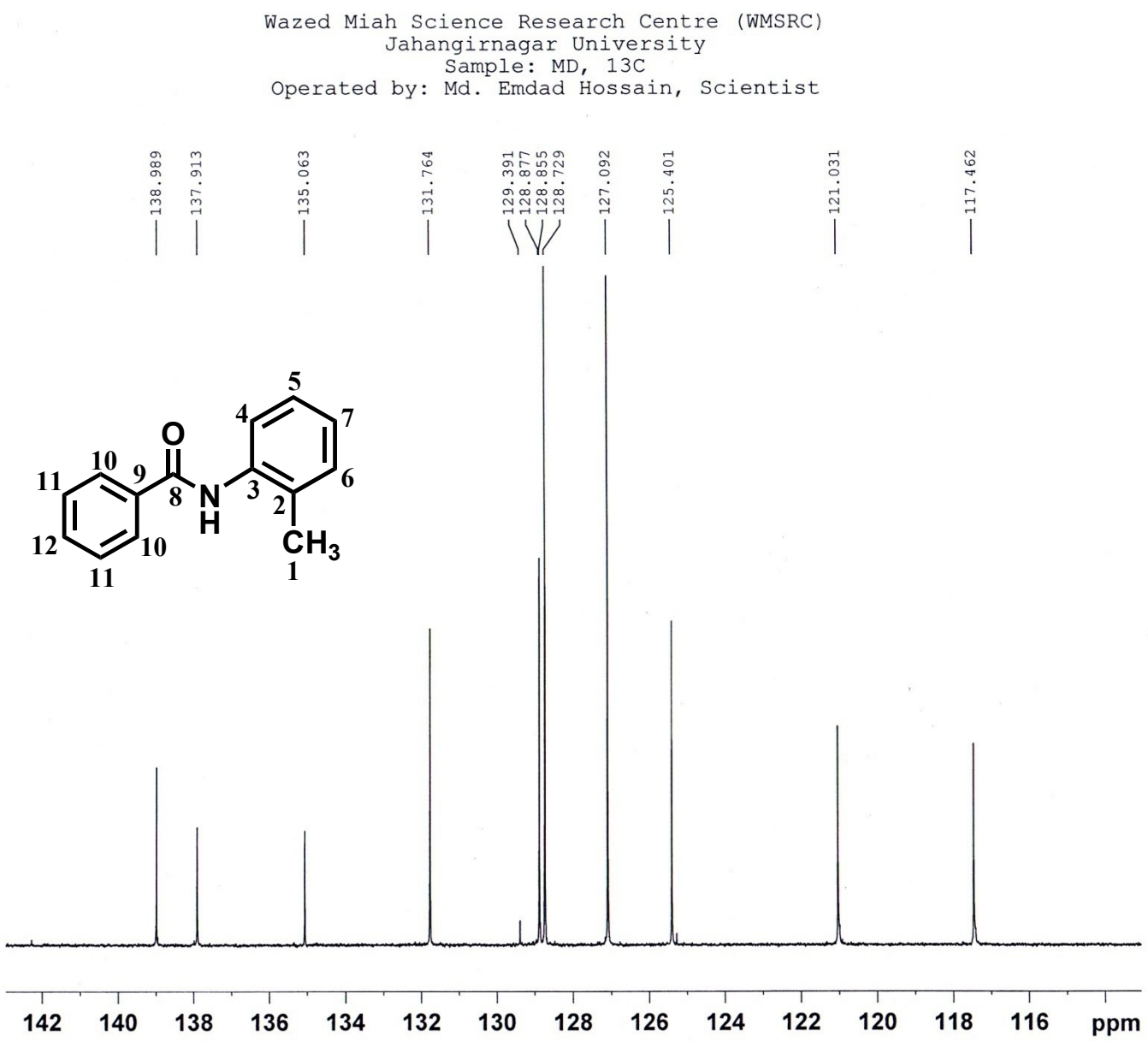
F2 - Acquisition Parameters
Date_              20200223
Time                  12.50
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zgpg
TD                   524288
SOLVENT               CDCl3
NS                      229
DS                        0
SWH               25252.525 Hz
FIDRES             0.048165 Hz
AQ                10.3809023 sec
RG                     208.5
DW                  19.800 usec
DE                   6.50 usec
TE                   298.0 K
D1                1.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            100.6479778 MHz
NUC1                    13C
P1                   10.00 usec
PLW1             49.00000000 W

======== CHANNEL f2 ========
SFO2            400.2320011 MHz
NUC2                    1H
CPDPRG[2             waltz16
PCPD2                90.00 usec
PLW2             12.00000000 W
PLW12             0.18584000 W
PLW13             0.15053000 W

F2 - Processing parameters
SI                    1048576
SF               100.6379135 MHz
WDW                        EM
SSB          0
LB                    0.30 Hz
GB           0
PC                    1.40
```

Figure S13: ^{13}C-NMR spectrum of compound 2.



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Current Data Parameters
NAME BUET_MD
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20200223
Time 12.50
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg
TD 524288
SOLVENT CDCl3
NS 229
DS 0
SWH 25252.525 Hz
FIDRES 0.048165 Hz
AQ 10.3809023 sec
RG 208.5
DW 19.800 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6479778 MHz
NUC1 13C
P1 10.00 usec
PLW1 49.00000000 W

======== CHANNEL f2 ========
SFO2 400.2320011 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.18584000 W
PLW13 0.15053000 W

F2 - Processing parameters
SI 1048576
SF 100.6379135 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

Figure S14: Extended ^{13}C-NMR spectrum of compound 2.

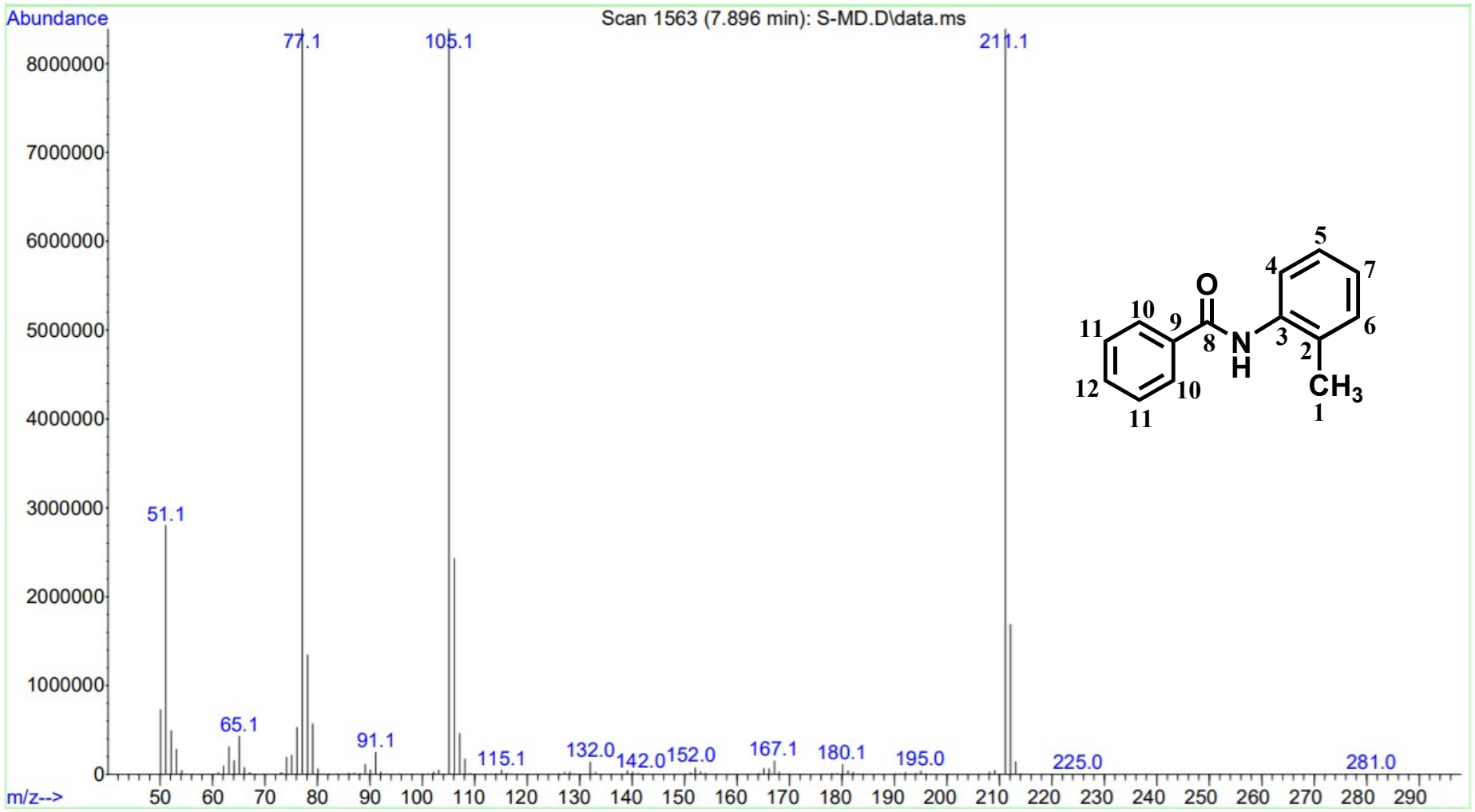


Figure S15: GC-MS spectrum of compound 2.

13. Spectra of compound 3

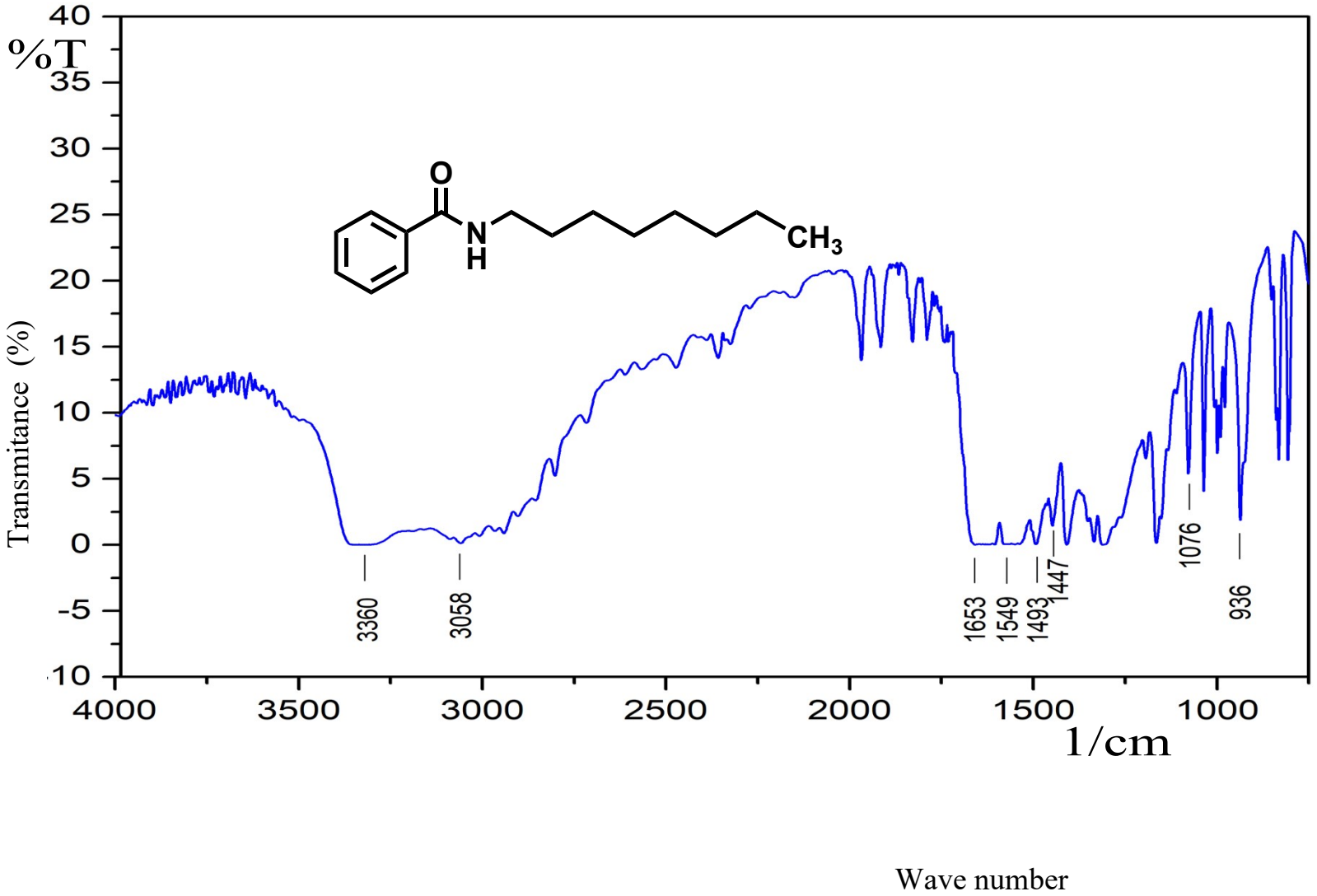


Figure S16: FT-IR spectrum of compound 3.

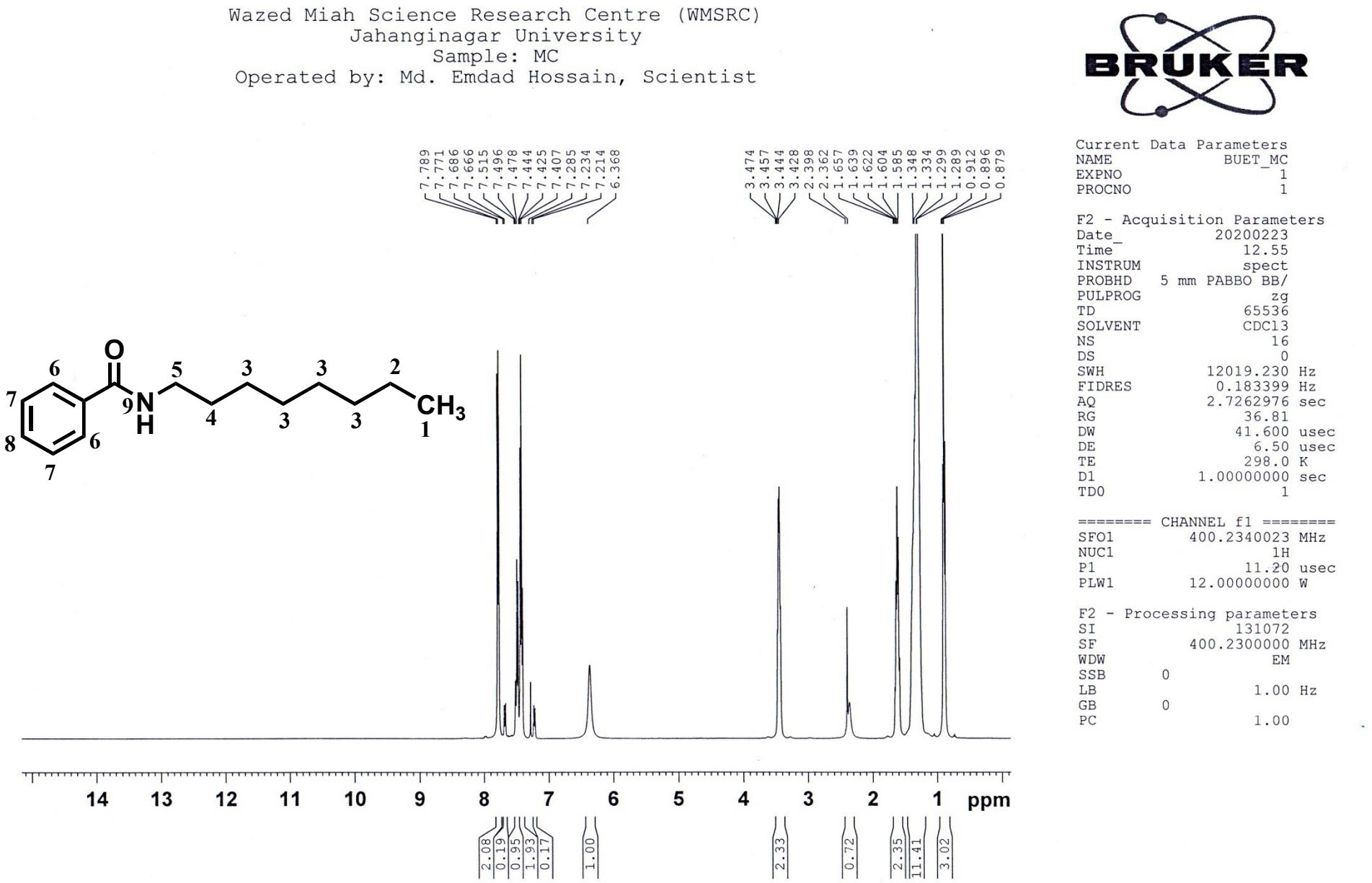
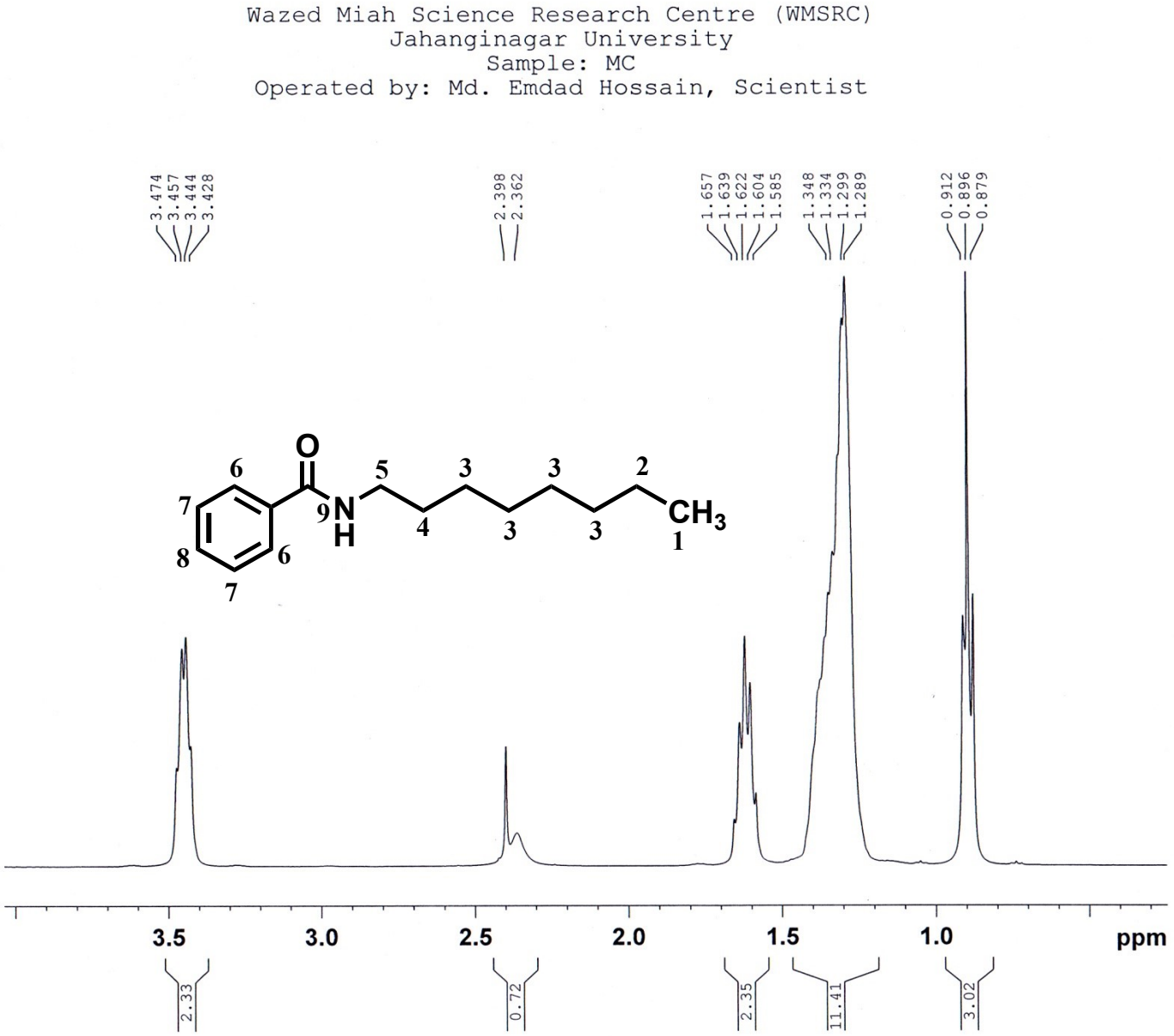


Figure S17: ^{1}H-NMR spectrum of compound 3




```
Current Data Parameters
NAME                  BUET_MC
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20200223
Time                   12.55
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                   zg
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         0
SWH               12019.230 Hz
FIDRES             0.183399 Hz
AQ                 2.7262976 sec
RG                     36.81
DW                    41.600 usec
DE                     6.50 usec
TE                    298.0 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1            400.2340023 MHz
NUC1                      1H
P1                     11.20 usec
PLW1           12.00000000 W

F2 - Processing parameters
SI                    131072
SF              400.2300000 MHz
WDW                       EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.00
```

Figure S18: Extended ^{1}H-NMR spectrum of compound 3

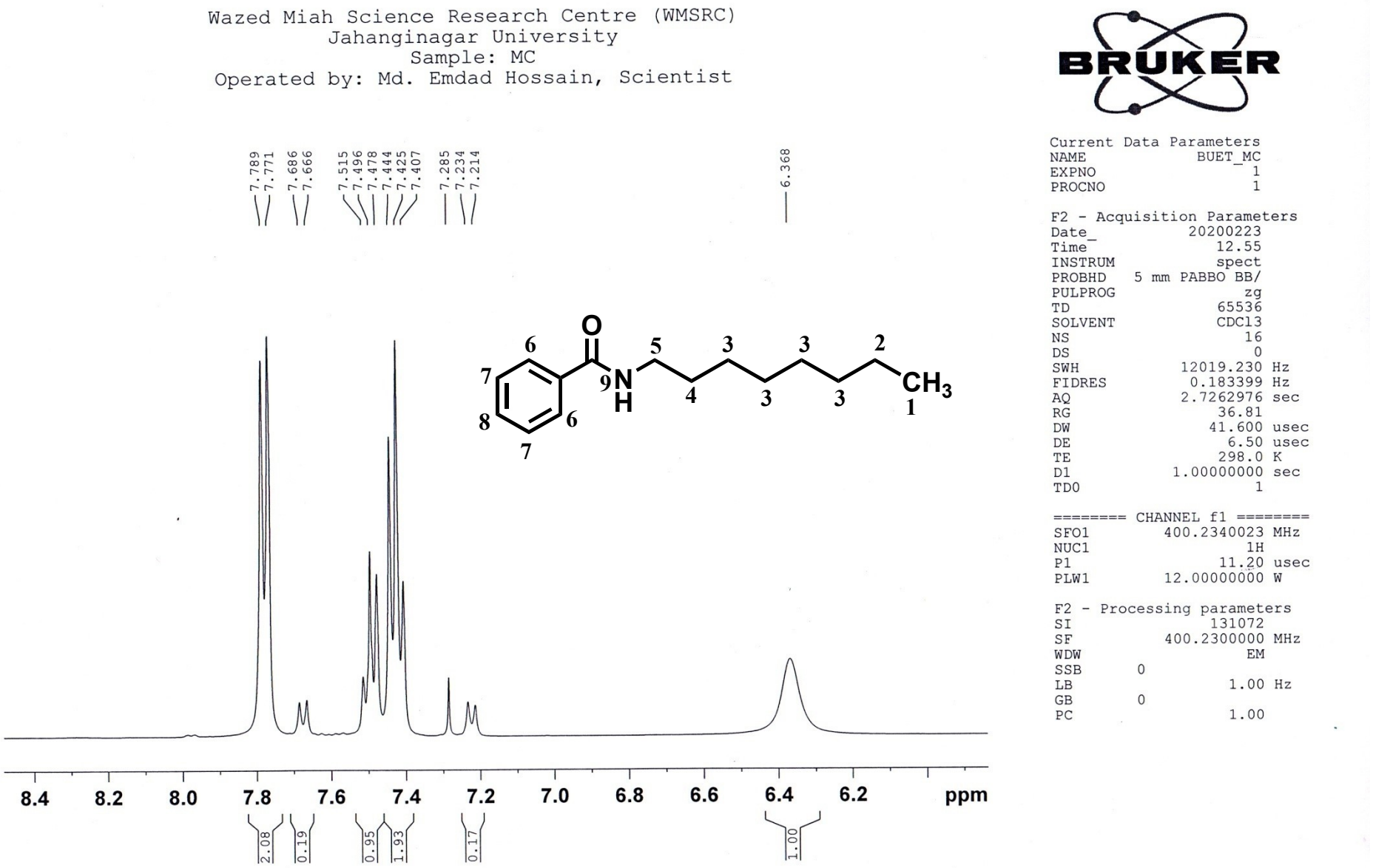


Figure S19: Extended ^{1}H-NMR spectrum of compound 3.

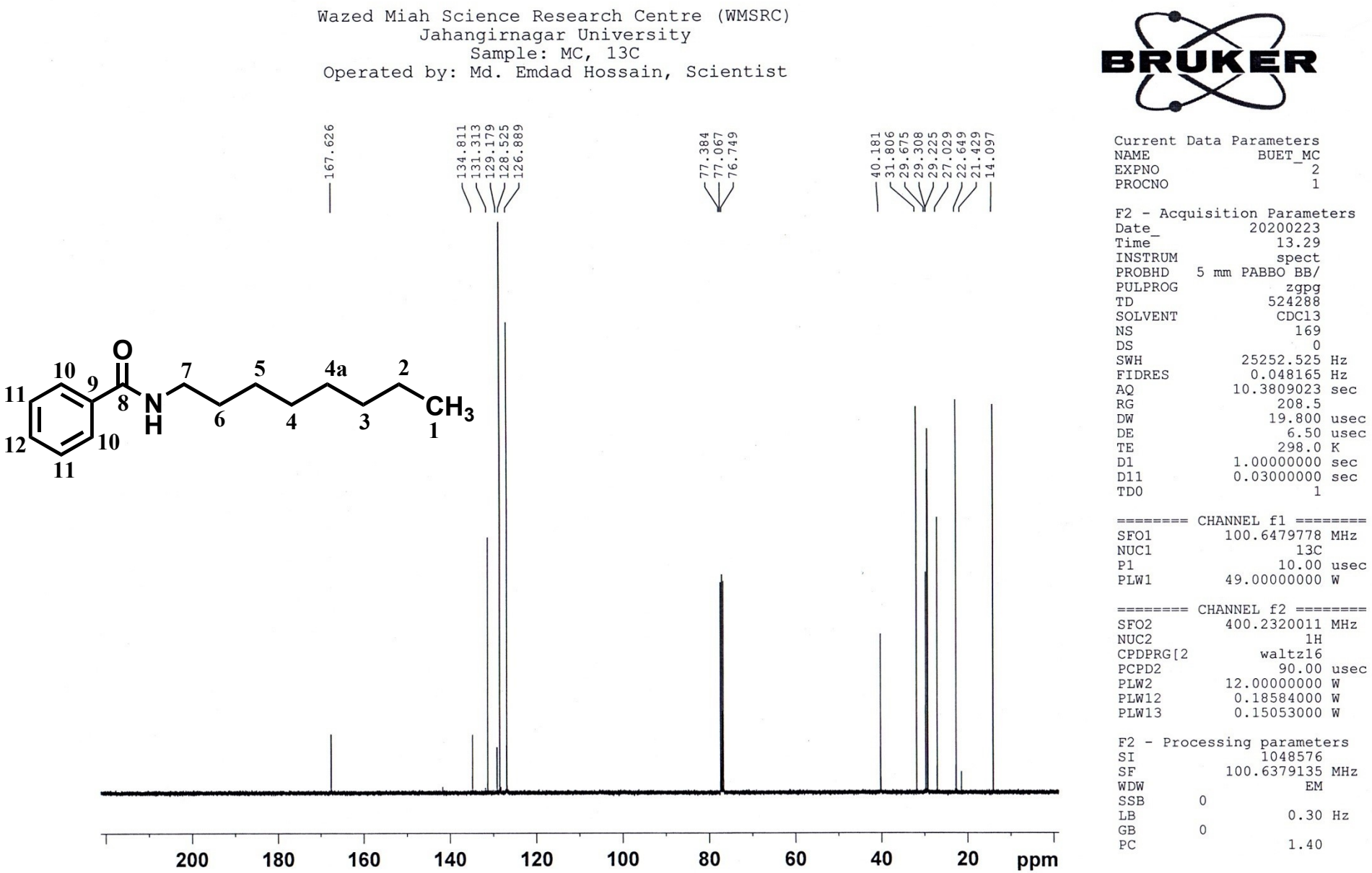


Figure S20: ^{13}C-NMR spectrum of compound 3

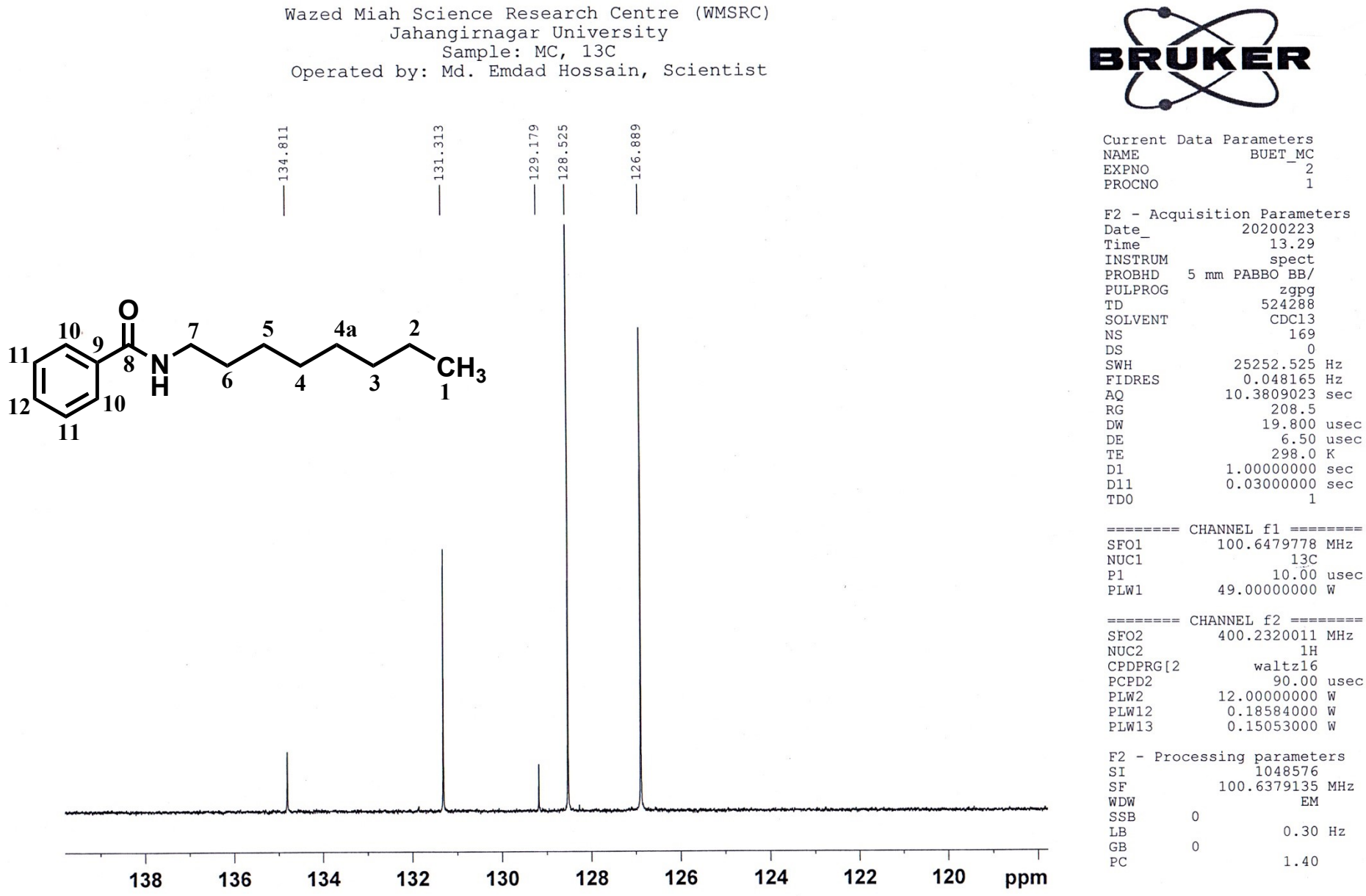


Figure S21: Extended ^{13}C-NMR spectrum of compound 3

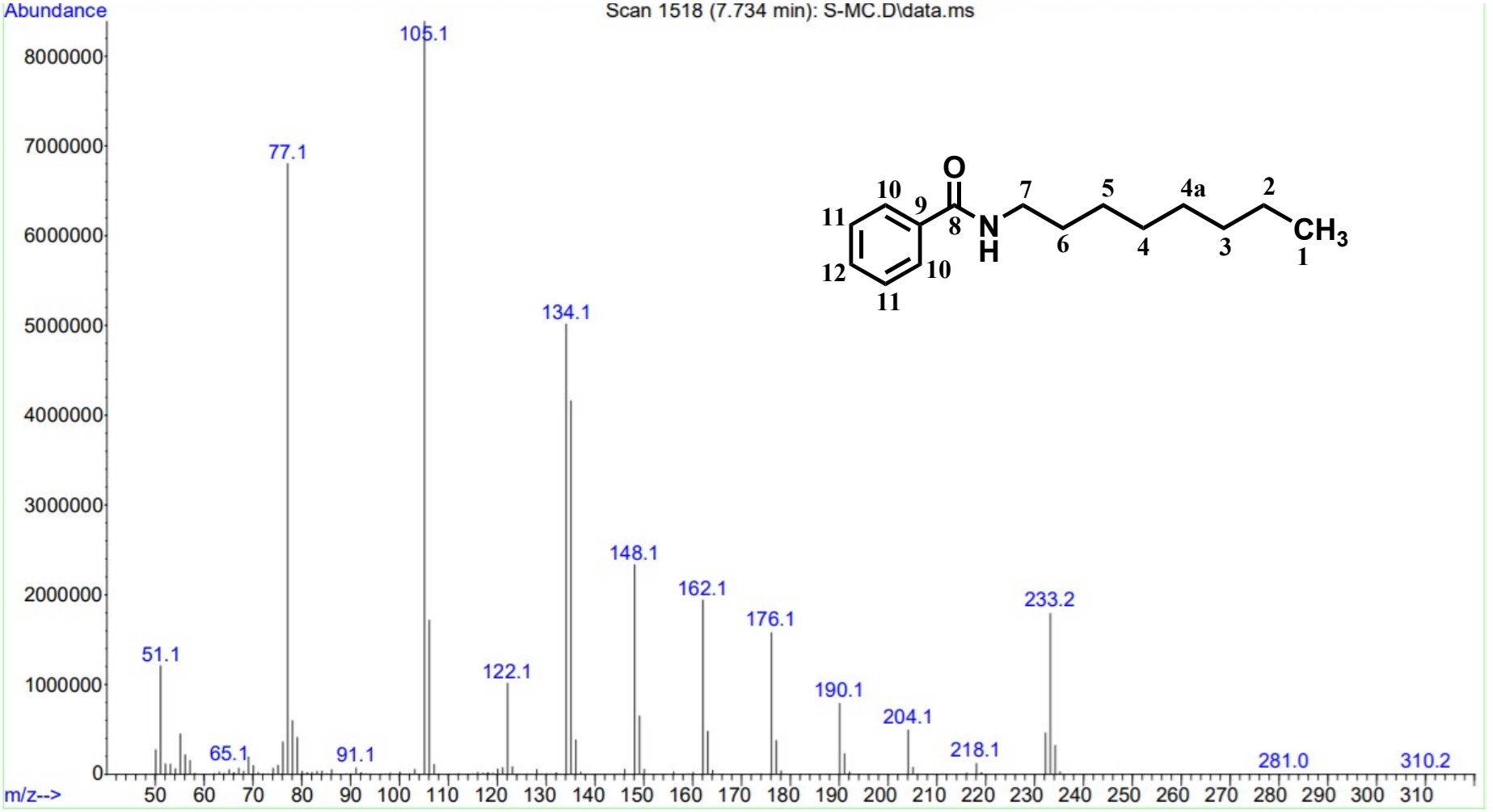


Figure S22: GC-MS spectrum of compound 3

14. Spectra of compound 4

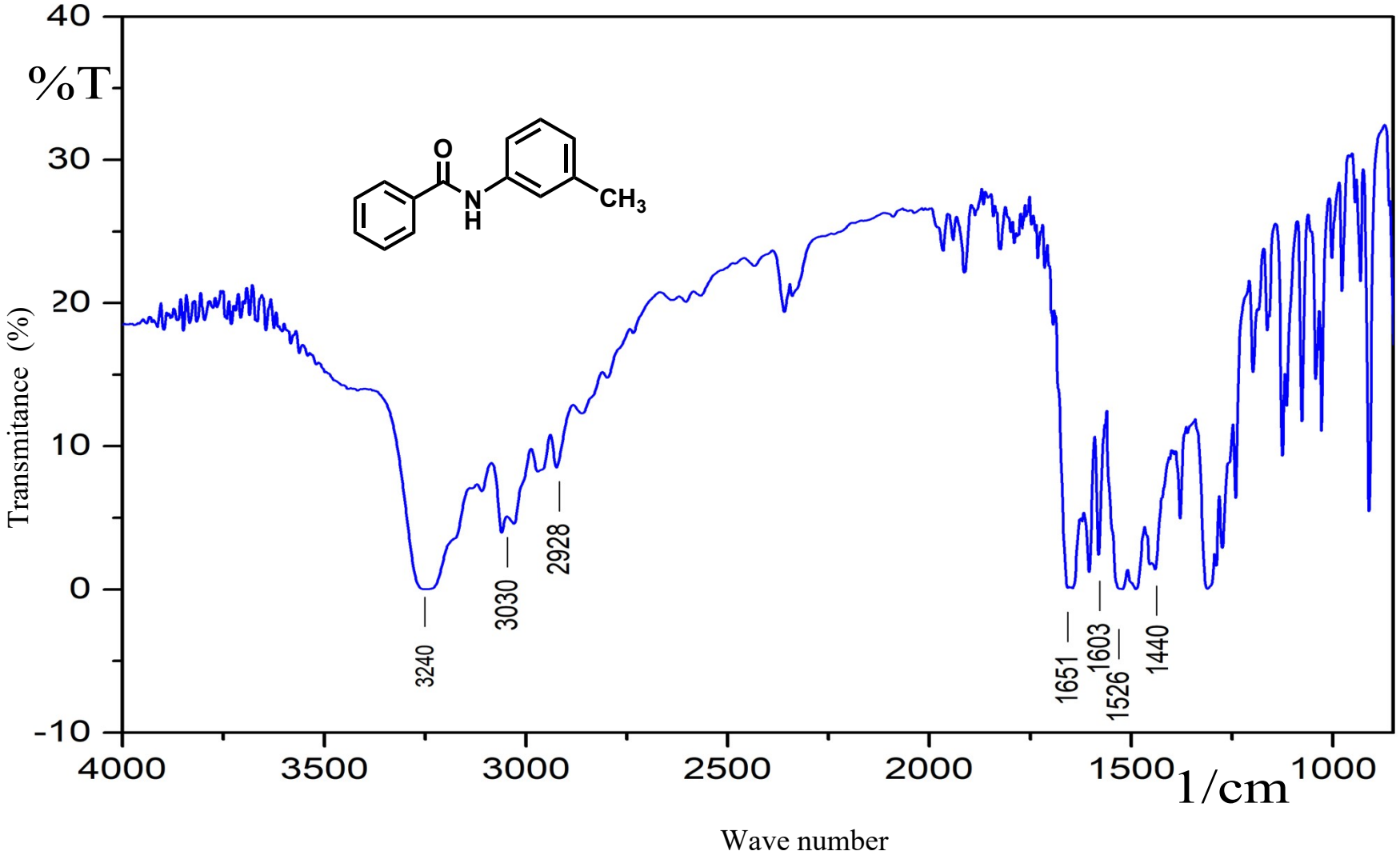


Figure S23: FT-IR spectrum of compound 4.

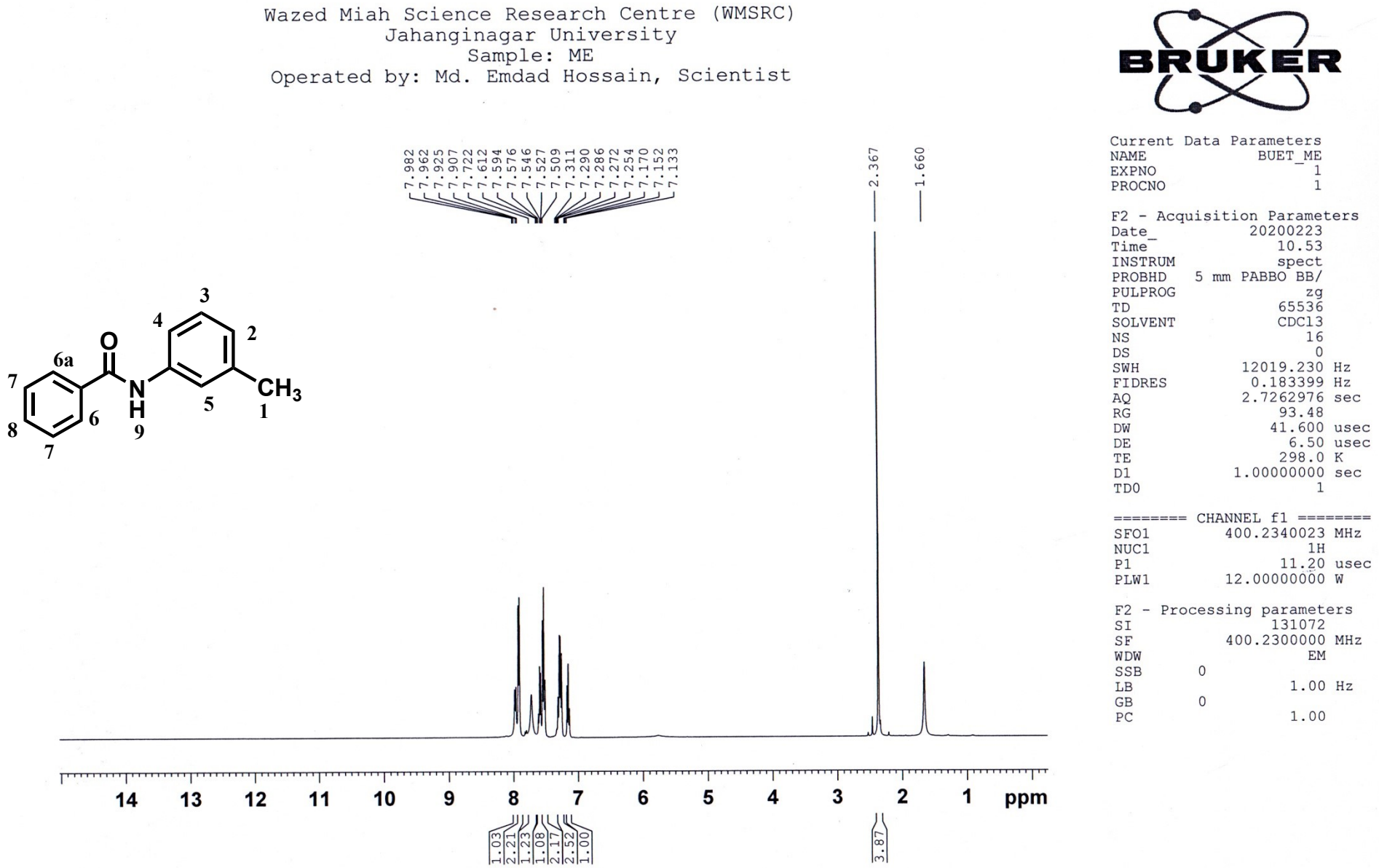
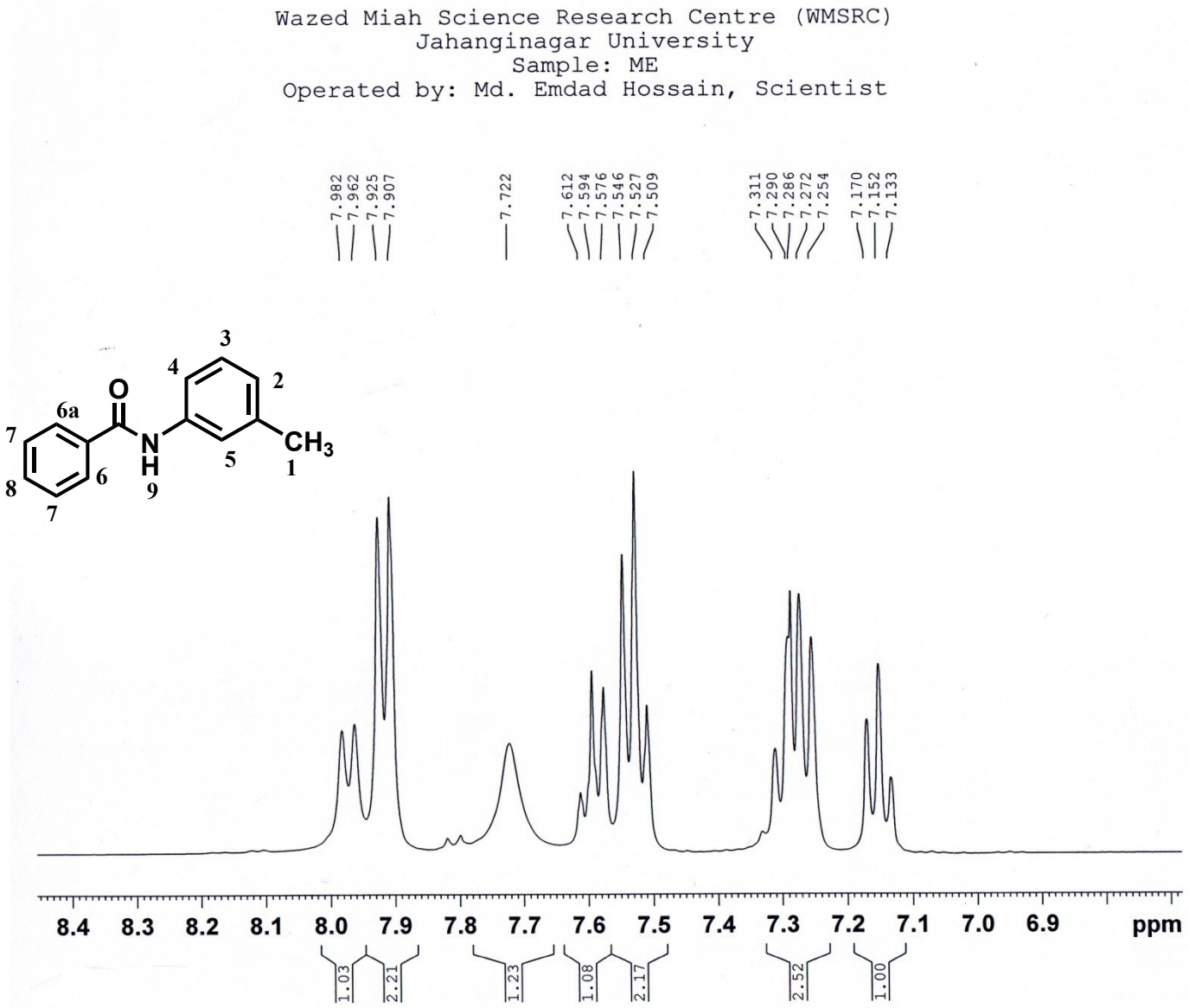


Figure S24: ^{1}H-NMR spectrum of compound 4




```
Current Data Parameters
NAME                  BUET_ME
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_               20200223
Time                   10.53
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                   zg
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         0
SWH               12019.230 Hz
FIDRES             0.183399 Hz
AQ                 2.7262976 sec
RG                     93.48
DW                    41.600 usec
DE                     6.50 usec
TE                    298.0 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1            400.2340023 MHz
NUC1                      1H
P1                     11.20 usec
PLW1           12.00000000 W

F2 - Processing parameters
SI                     131072
SF              400.2300000 MHz
WDW                       EM
SSB         0
LB                     1.00 Hz
GB          0
PC                     1.00
```

Figure S25: Extended ^{1}H-NMR spectrum of compound 4

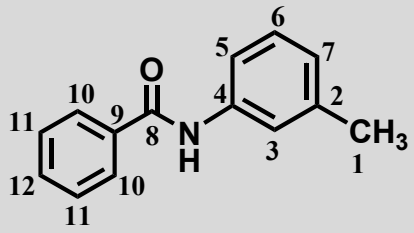
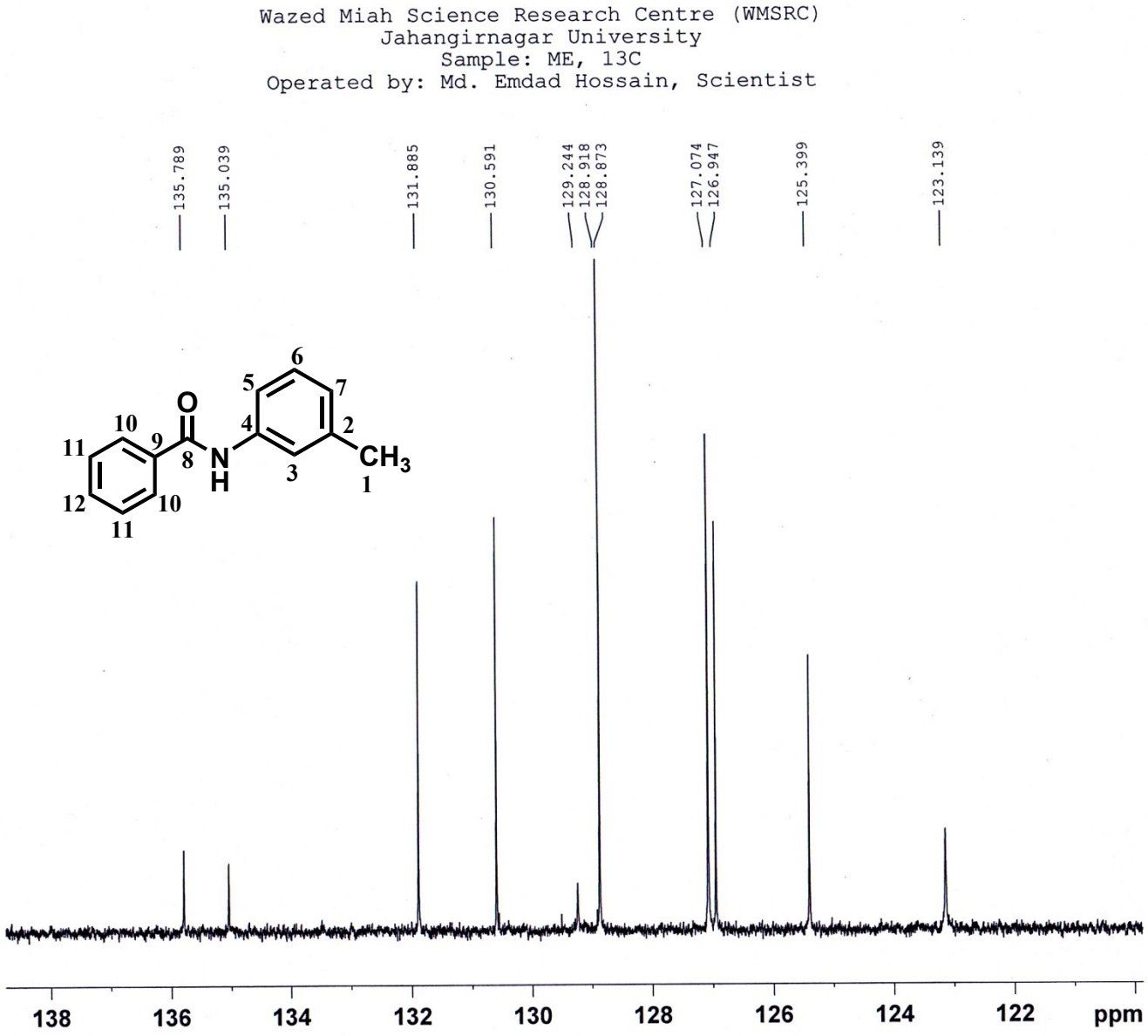


Figure S26: ^{13}C-NMR spectrum of compound 4



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```
Current Data Parameters
NAME                  BUET_ME
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20200223
Time                  11.29
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG                zgpg
TD                   524288
SOLVENT               CDCl3
NS                      183
DS                        0
SWH               25252.525 Hz
FIDRES             0.048165 Hz
AQ                10.3809023 sec
RG                     208.5
DW                  19.800 usec
DE                   6.50 usec
TE                   298.0 K
D1                1.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            100.6479778 MHz
NUC1                    13C
P1                    10.00 usec
PLW1           49.00000000 W

======== CHANNEL f2 ========
SFO2            400.2320011 MHz
NUC2                    1H
CPDPRG[2            waltz16
PCPD2                 90.00 usec
PLW2           12.00000000 W
PLW12           0.18584000 W
PLW13           0.15053000 W

F2 - Processing parameters
SI                    1048576
SF              100.6379135 MHz
WDW                       EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.40
```

Figure S27: Extended ^{13}C-NMR spectrum of compound 4

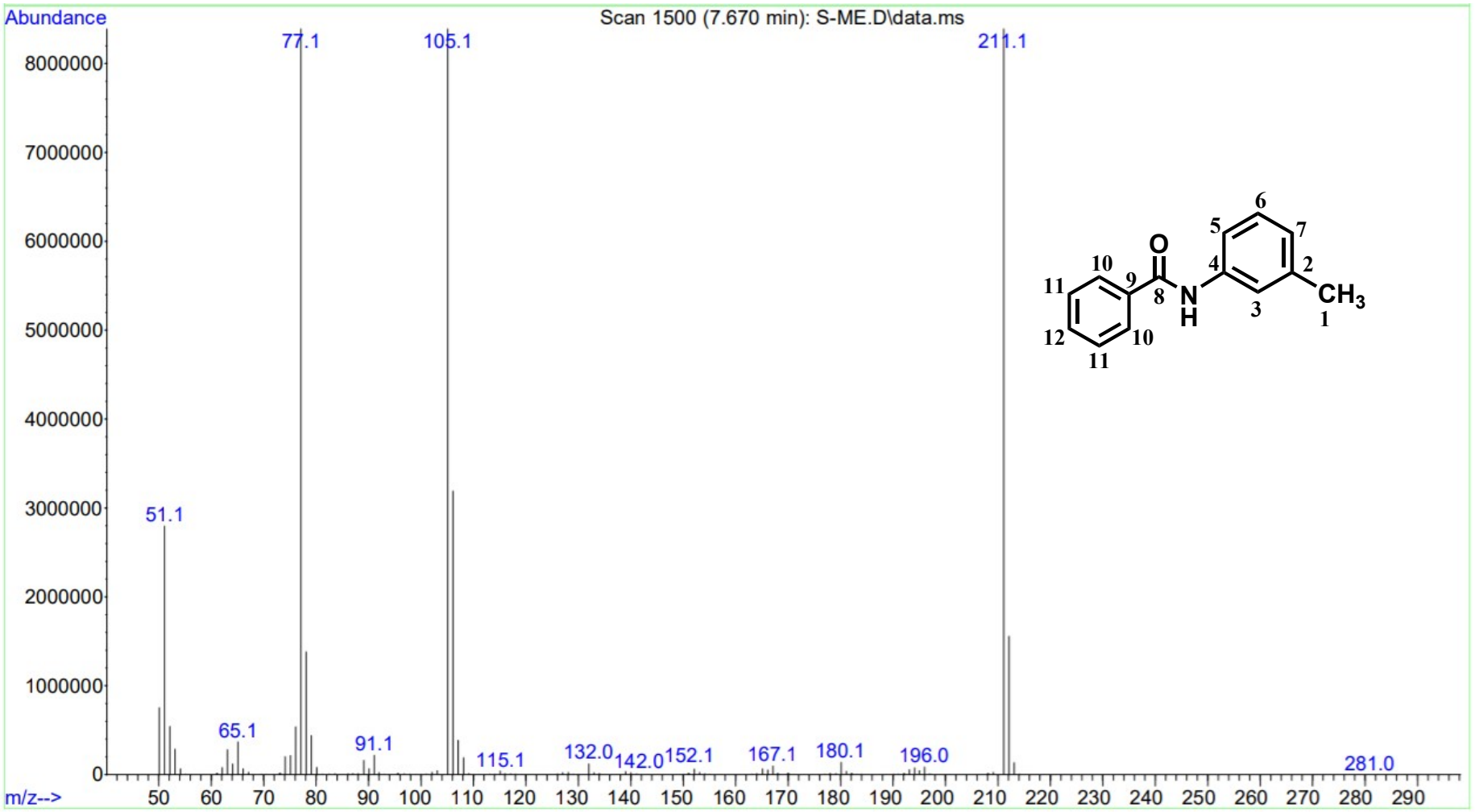

Figure S28: GC-MS spectrum of compound 4

15. Spectra of compound 5

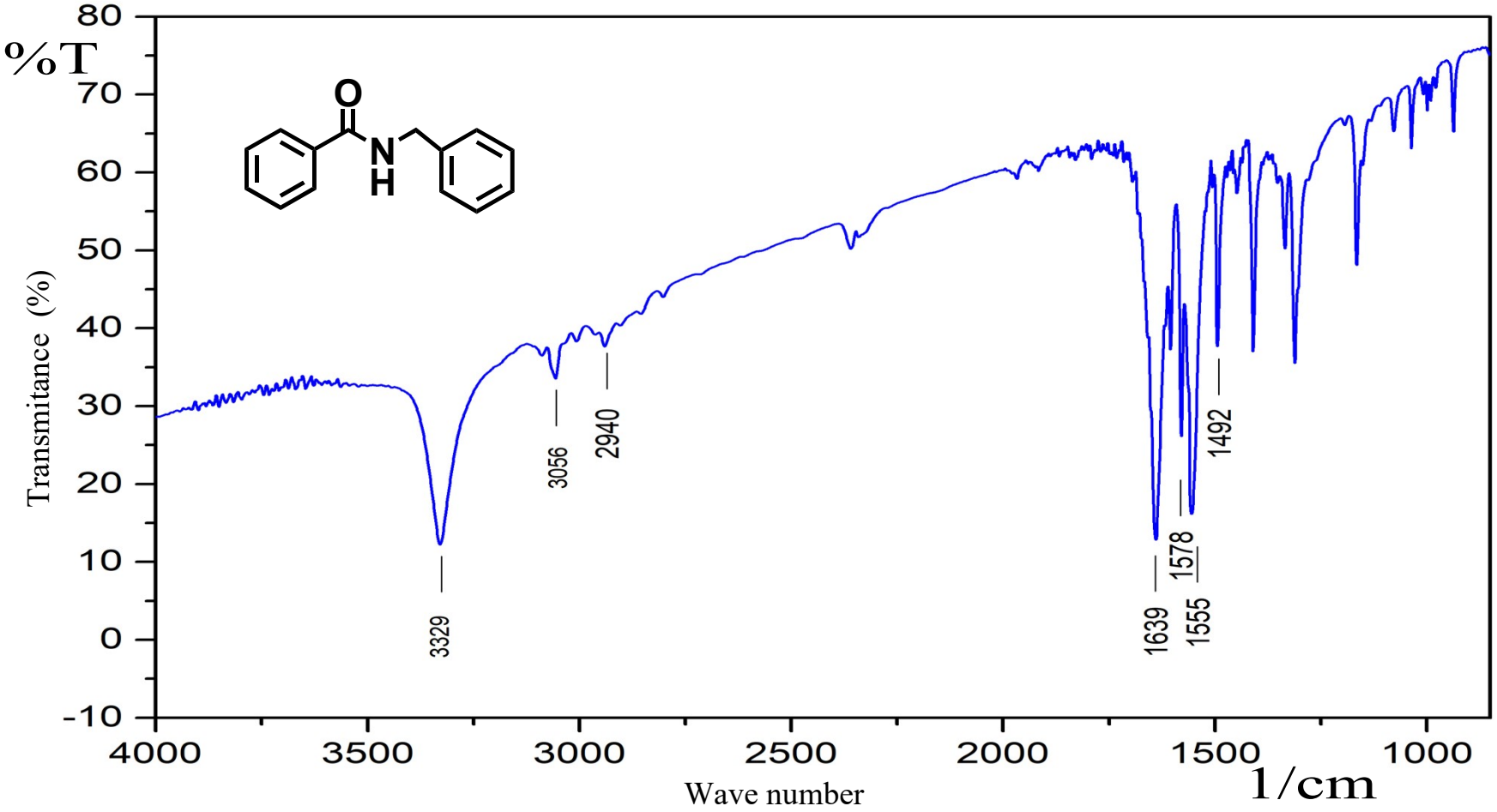

Figure S29: FT-IT spectrum of compound 5