Supporting information

For

A mild and efficient method for the synthesis of pyrroles using MIL-53(Al) as a catalyst under solvent-free sonication.

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Section S1. Materials

Aniline (ACS reagent, ≥ 99.5%), *o*-toluidine (assay ≥ 99%), 3,5-dichoroaniline (assay ≥ 98%), 2,5-dichoroaniline (assay ≥ 99%), 3,4-dichoroaniline (assay ≥ 99%), 2,5-dibromoaniline (assay ≥ 98%), triethylenetetramine (assay ≥ 97.0% (T)), tetraethylenepentamine (technical grade), phenylhydrazine (assay 97%), 2,4-dinitrophenylhydrazine (reagent grade, 97%), 4-nitroaniline (assay ≥ 99%), 4-nitro-*o*-phenylenediamine (assay 98%), 2-amino-4-nitrophenol (assay ≥ 99.0% (NT)), 2-amino-*p*-cresol (assay 97%), 4-aminobenzonitrile (assay 98%), 4-iodoaniline (assay 98%), 2-aminobiphenyl (assay 97%), methyl 4-aminobenzoate (assay 98%), and 4-aminophenol (assay 99%) were purchased from Sigma-Aldrich. Acetonylacetone (analysis EMSURE®), anhydrous glycerol (excipient EMPROVE®), anhydrous oxalic acid (for synthesis), and TLC (silica gel 60 F254) were obtained from Merck. Silica gel 230–400 mesh (for flash chromatography) was obtained from Merck. Ethyl acetate (purity ≥ 99.5%), *n*-hexane, and chloroform (purity ≥ 99%) were obtained from Xilong Chemical Co., Ltd (China). Chloroform-*d*, 99.8 Atom %D, stab. with Ag was obtained from Armar (Switzerland). All starting materials, reagents and solvents were used without further purification.

Section S2: Characterization of MIL-53(Al)

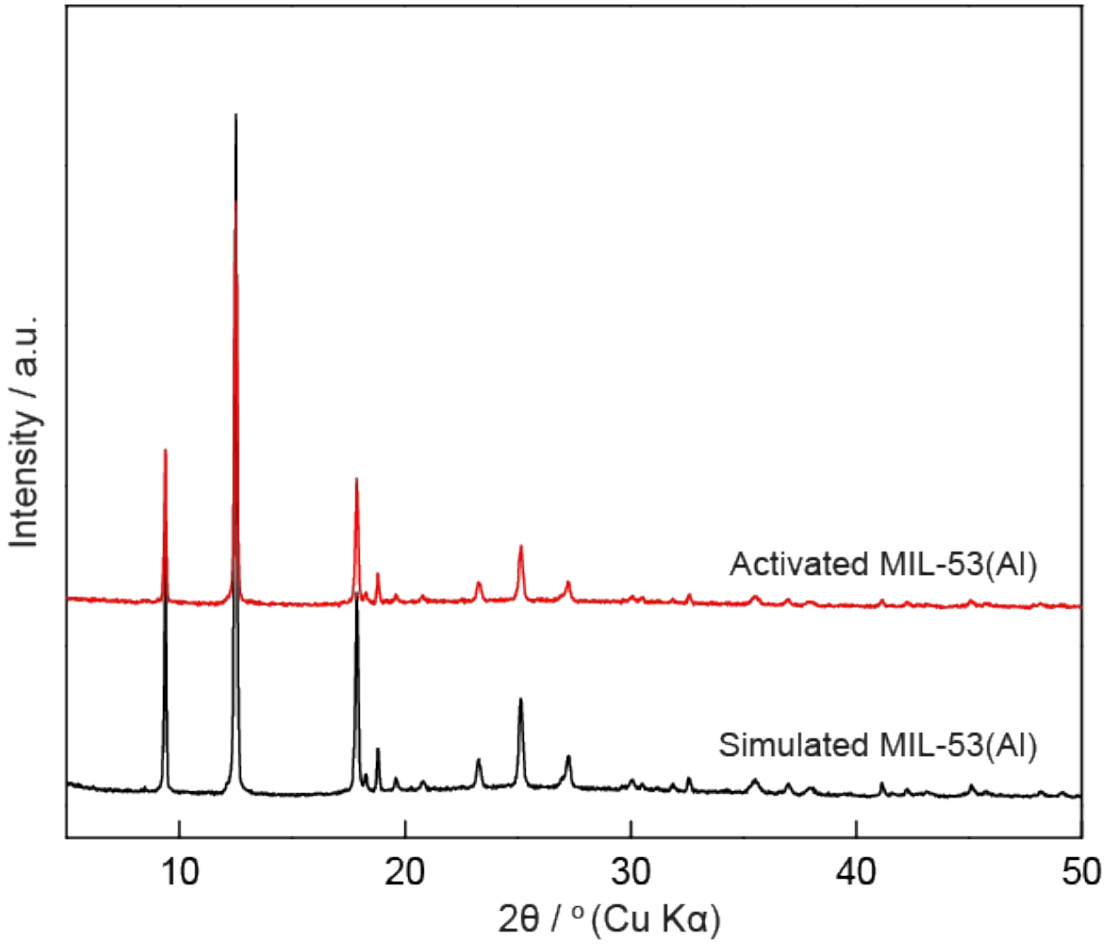


Fig. S1. PXRD analysis of MIL-53(Al). The calculated pattern from single crystal data (black) is compared to the activated powder sample (red).

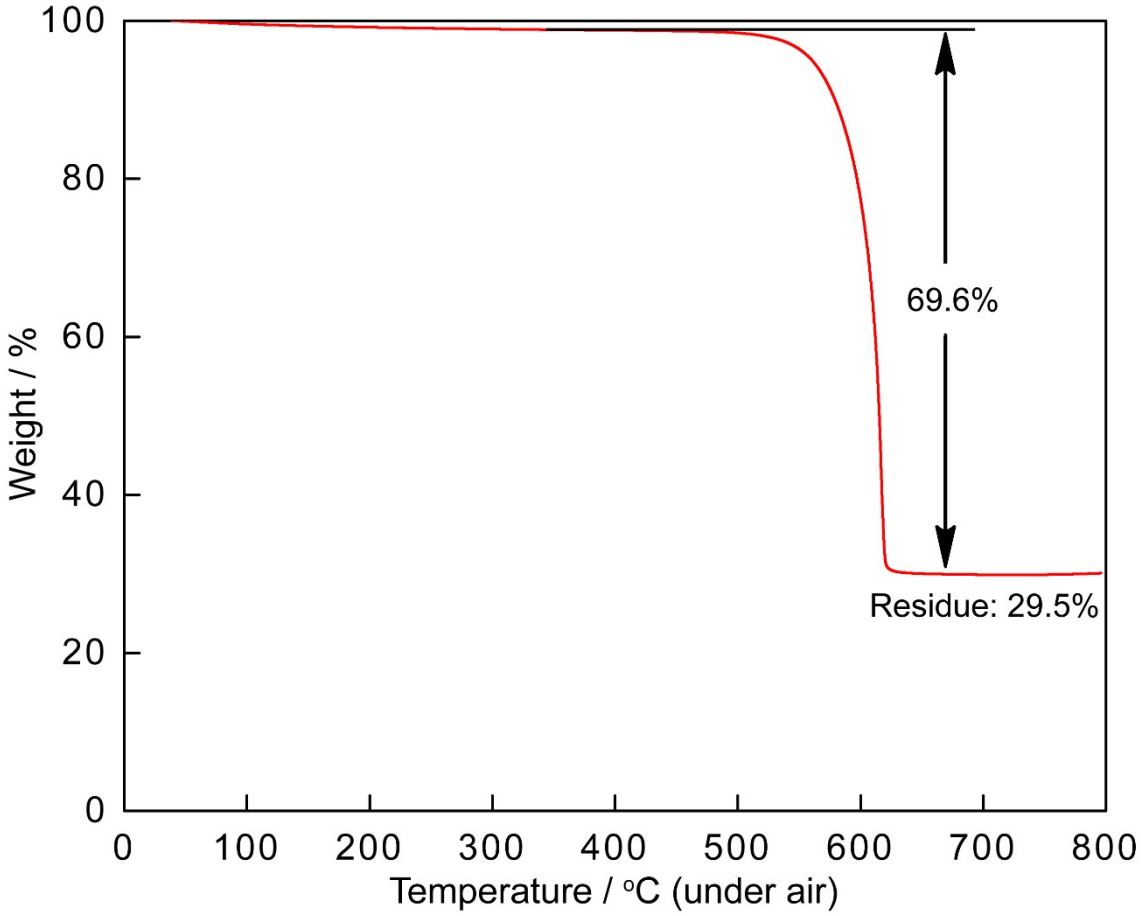


Fig. S2. Thermal gravimetric analysis of activated MIL-53(Al) under airflow.

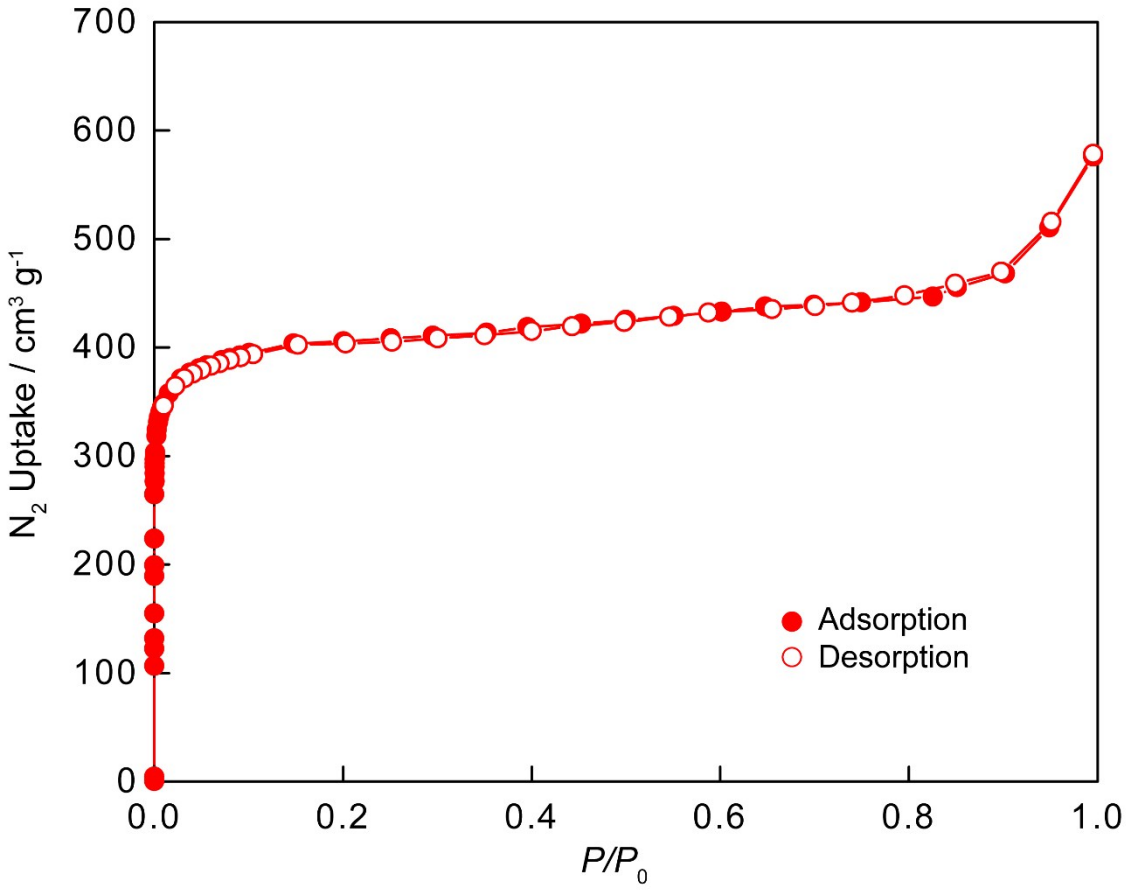


Fig. S3. N_2 isotherm at 77 K for activated MIL-53(Al). Closed and open circles represent the adsorption and desorption branches, respectively.

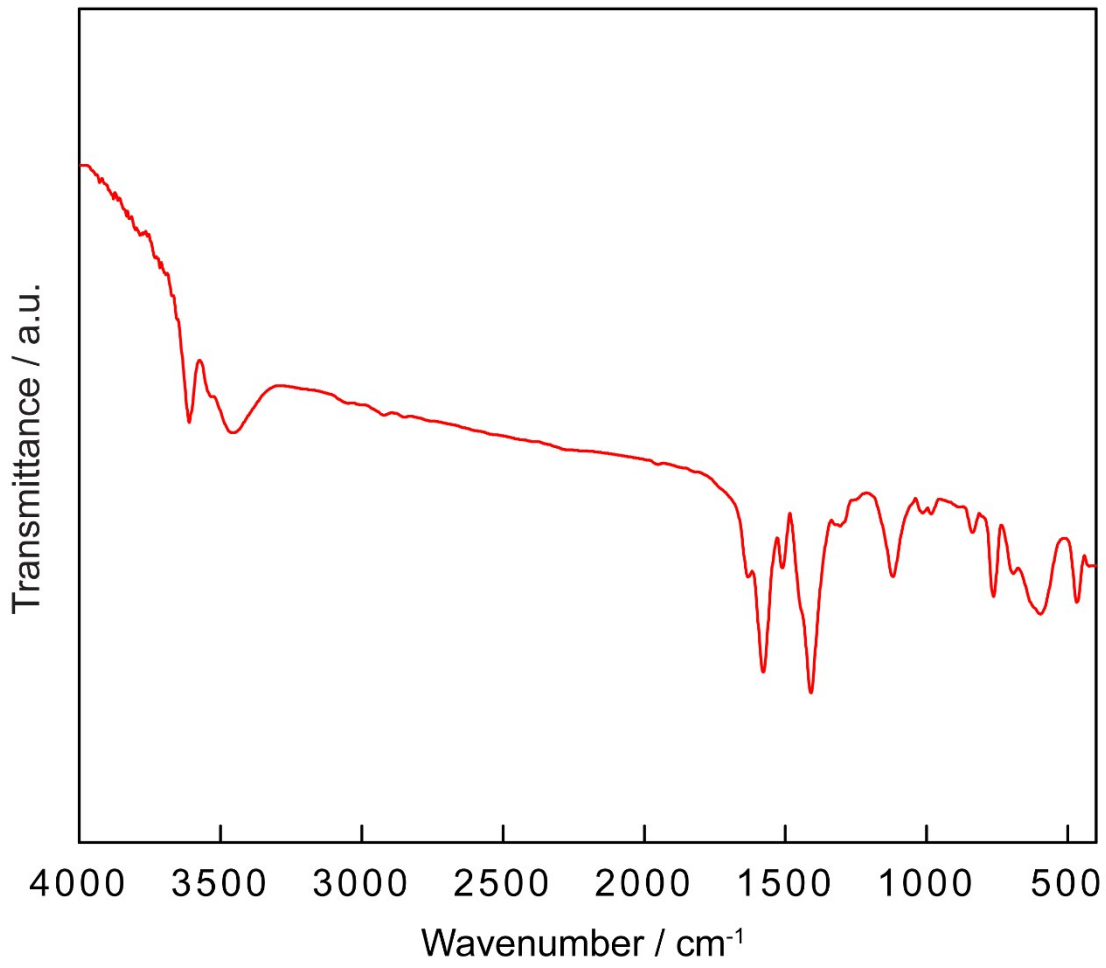


Fig. S4. Infrared spectra of activated MIL-53(Al) (red) in dry KBr.

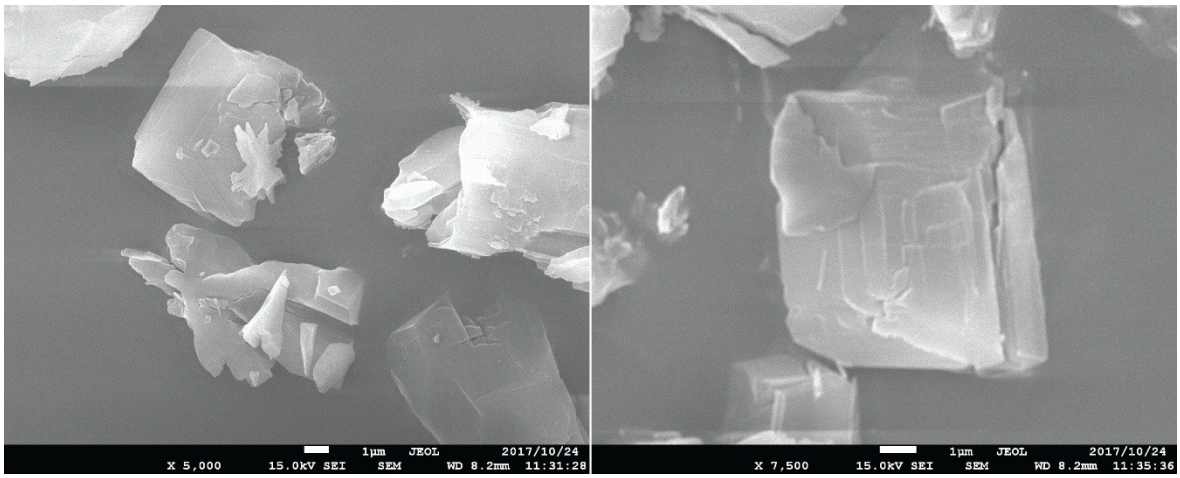


Fig. S5. SEM images of activated MIL-53(Al).

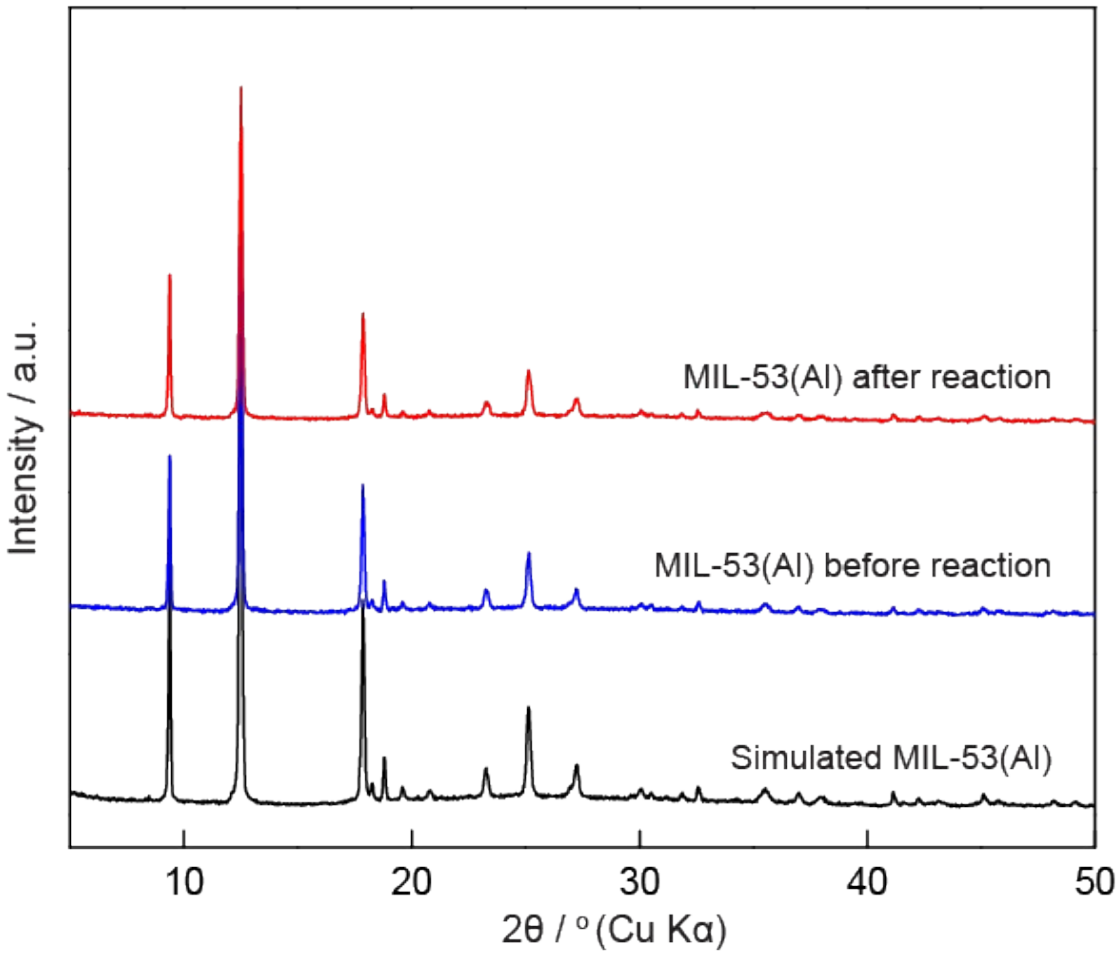
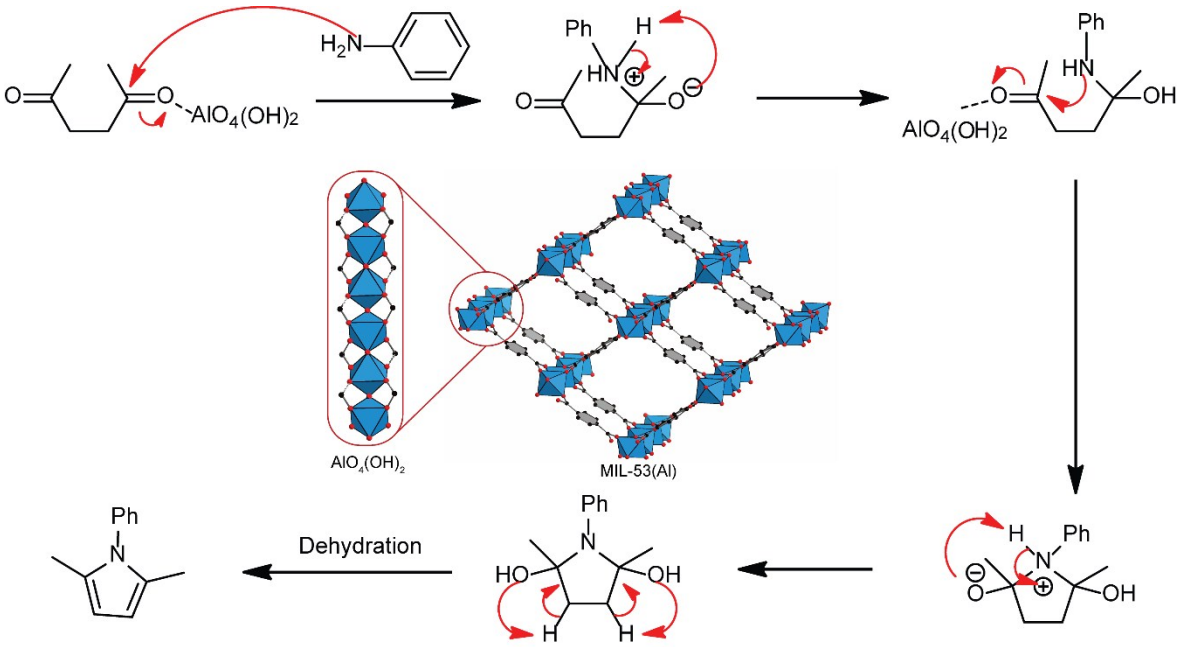


Fig. S6. PXRD analysis of MIL-53(Al) before (blue) and after (red) 4th time recycling.

Section S3. Optimization of the reaction condition



Scheme S1. Proposed reaction mechanism

Table S1. Effect of the reaction time.[a]

Entry	Time (min)	Isolated yield (%)[b]
1	1	45
2	5	53
3	10	55
4	**15**	**96**
5	30	95
6	45	97

[a]Reaction condition: Aniline (1.0 mmol), acetonylacetone (1.2 mmol) in the presence of MIL-53(Al)-catalyzed (5 %mol) without solvent.

The effect of catalyst amount on the model reaction was also carried out by varying the MIL-53(Al) amount to 0 %mol, 1 %mol, 5 %mol, 7 %mol, 10 %mol and 15 %mol. The optimum amount of catalyst for the model reaction was found to be 5 %mol, about 96% (Table 2, entry 3). The effect of the molar ratio of substrates on the model reaction was investigated in Table 3. As can be seen from Table 3, the molar ratio of substrates had a remarkable effect on the yield. When the ratio of

aniline and acetonylacetone was 1:1.2, the product was obtained in the best yield of 96% (Table 3, entry 3). Thus, we chose the molar ratio as the optimal ratio for further studies.

Table S2. Effect of the ratio of MIL-53(Al) with solventless.[a]

Entry	Amount of MIL-53(Al) (mol%)	Isolated yield (%)
1	0	65
2	1	80
3	**5**	**96**
4	7	96
5	10	90
6	15	90

[a]Reaction condition: Aniline (1.0 mmol), acetonylacetone (1.2 mmol) in the presence of catalyst (%mol) without solvent.

Table S3. Effect of the ratio aniline/acetonylacetone.[a]

Entry	Molar ratios of reactants	Isolated yield (%)
1	1:1	80
2	1:1.1	87
3	**1:1.2**	**96**
4	1:1.3	96
5	1:1.4	96
6	1:1.5	97
7	1:2	98
8	1:3	98
9	1:4	98
10	1:5	98

[a]Reaction condition: Aniline (1.0 mmol), acetonylacetone (mmol) in the presence of MIL-53(Al) (5 %mol) without solvent.

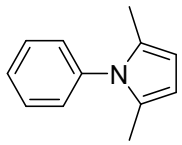
Table S4. Effect of various solvents.[a]

Entry	Solvent	Isolated yield (%)
1	**None-solvent**	**96**
2	Dichloromethane	85
3	Tetrahydrofurane	79
4	Ethanol	77
5	*n*-Butanol	75
6	*N,N*-Dimethylformamide	82
7	Acetone	90
8	Acetonitrile	75
9	Dimethyl sulfoxide	80
10	Hexane	83
11	Dioxane	84
12	Toluene	81
13	Cyclopentyl methyl ether	76

[a]Reaction condition: Aniline (1.0 mmol), acetonylacetone (1.2 mmol) and MIL-53(Al) (5 %mol) in the presence of solvent (1.5 mL).

Section S4. Spectral data

2,5-Dimethyl-1-phenyl-1*H*-pyrrole[1-6]



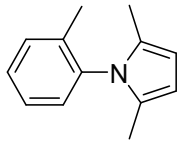
Yellow solid, mp 52-54 ˚C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49 – 7.46 (t, J = 7.0 Hz, 2H), 7.43 – 7.40 (t, J = 7.5 Hz, 1H), 7.24 – 7.23 (d, J = 7.0 Hz, 2H), 5.93 (s, 2H), 2.06 (s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 139.1, 129.0, 128.8, 128.3, 127.6, 105.6, 13.0.

GC-MS (EI, 70 eV) *m/z* 171 ($[M]^+$)

2,5-Dimethyl-1-(*o*-tolyl)-1*H*-pyrrole[1, 2, 4, 7]



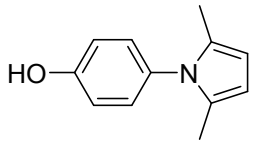
Yellow oil

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 – 7.32 (m, 2H), 7.29 – 7.27 (m, 1H), 7.17 – 7.15 (d, J = 7.5 Hz, 2H), 5.91 (s, 2H), 1.94 (s, 3H), 1.92 (s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 137.1, 130.7, 128.9, 128.3, 128.2, 126.6, 105.2, 29.7, 17.0, 12.5.

GC-MS (EI, 70 eV) *m/z* 185 ($[M]^+$)

1-(4-Hydroxyphenyl)-2,5-dimethyl-1*H*-pyrrole[7, 17, 19, 20]



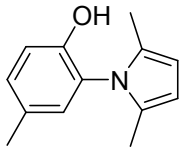
Yellow solid, mp 105-107 ˚C

^{1}H-NMR (500 MHz, DMSO-d_6) δ 9.66 (s, 1H), 7.01 – 6.98 (m, 2H), 6.85 – 6.82 (m, 2H), 5.71 (s, 2H), 1.90 (s, 6H).

^{13}C-NMR (125 MHz, DMSO-d_6) δ 157.2, 130.0, 129.5, 128.1, 116.1, 105.7, 13.3.

GC-MS (EI, 70 eV) *m/z* 187 ($[M]^+$).

1-(2'-Hydroxy-5'-methylphenyl)-2,5-dimethyl-1*H*-pyrrole



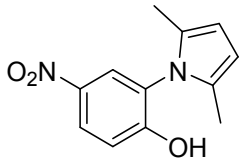
Black oil

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.14 – 7.12 (dd, J = 2.0 Hz, 2.0 Hz, 1H), 6.96 – 6.95 (d, J = 8.5 Hz, 1H), 6.92 – 6.91 (d, J = 1.5 Hz, 1H), 5.94 (s, 2H), 5.08 (s, 1H), 2.31 (s, 3H), 1.98 (s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 150.4, 130.5, 130.1, 129.4, 129.0, 116.5, 115.9, 106.7, 20.4, 12.3.

HRMS (ESI) *m/z* calcd for $[M + H]^+$ $C_{13}H_{16}NO^+$ 202.1226, found 202.1201.

1-(2'-Hydroxy-5'-nitrophenyl)-2,5-dimethyl-1*H*-pyrrole



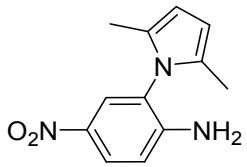
Orange solid, mp 167-170 ˚C

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.28 – 8.24 (dd, J = 2.5 Hz, 2.5 Hz, 1H), 8.09 – 8.08 (d, J = 3.0 Hz, 1H), 7.18 – 7.16 (d, J = 9.5 Hz, 1H), 5.99 (s, 2H), 1.99 (s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 158.7, 141.3, 129.1, 126.1, 125.7, 116.8, 107.9, 12.3.

HRMS (ESI) *m/z* calcd for $[M + H]^+$ $C_{12}H_{13}N_2O_3^+$ 233.0920, found 233.0939.

1-(2'-Amino-4'-nitrophenyl)-2,5-dimethyl-1*H*-pyrrole



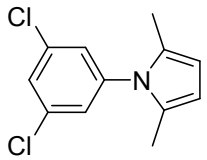
Yellow solid, m.p. = 128-130 ˚C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.65 – 7.63 (m, 2H), 7.21 – 7.19 (d, J = 9.0 Hz, 1H), 5.97 (s, 2H), 3.82 (s, 2H), 1.97 (s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 145.1, 130.3, 130.2, 124.0, 118.0, 112.8, 109.8, 107.1, 12.2.

HRMS (ESI) *m/z* calcd for $[M + H]^+$ $C_{12}H_{14}N_3O_2^+$ 230.1049, found 230.1011.

1-(3,5-Dichlorophenyl)-2,5-dimethyl-1*H*-pyrrole[8]



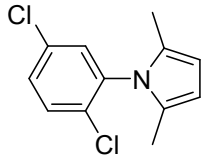
Orange solid, mp 79-81 ˚C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42 – 7.41 (t, J = 2.0 Hz, 1H), 7.15 – 7.14 (d, J = 1.5 Hz, 2H), 5.90 (s, 2H), 2.06 (s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 141.0, 135.2, 128.6, 128.6, 127.0, 106.7, 29.7, 13.0.

GC-MS (EI, 70 eV) *m/z* 239 ($[M]^+$)

1-(2,5-Dichlorophenyl)-2,5-dimethyl-1*H*-pyrrole[9]



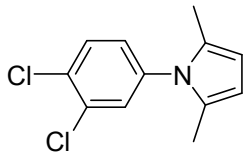
Black solid, mp 136-137 ˚C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.51 – 7.50 (d, J = 8.5 Hz, 1H), 7.42 – 7.39 (dd, J = 2.5 Hz, 2.5 Hz, 1H), 7.36 – 7.35 (d, J = 2.5 Hz, 1H), 5.97 (s, 2H), 2.01 (s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 138.1, 133.0, 132.7, 131.0, 130.8, 129.8, 128.6, 106.2, 12.5.

GC-MS (EI, 70 eV) *m/z* 239 ($[M]^+$)

1-(3,4-Dichlorophenyl)-2,5-dimethyl-1*H*-pyrrole[1, 2, 4, 5]



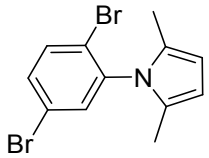
Yellow solid, mp 101-103 ˚C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.55 – 7.54 (d, J = 8.5 Hz, 1H), 7.35 (d, J = 2.5 Hz, 1H), 7.10 – 7.08 (m, 1H), 5.91 (s, 2H), 2.05 (s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 138.5, 133.0, 132.0, 130.8, 130.2, 128.7, 127.6, 106.5, 13.0.

GC-MS (EI, 70 eV) *m/z* 239 ($[M]^+$)

1-(2,5-Dibromophenyl)-2,5-dimethyl-1*H*-pyrrole



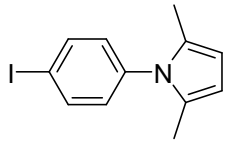
Yellow oil

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 – 7.57 (d, J = 8.5 Hz, 1H), 7.47 – 7.44 (m, 2H), 5.92 (s, 2H), 1.97 (s, 6H).

^{13}C-NMR (125 MHz, $CDCl_3$) δ 140.0, 134.3, 133.6, 133.0, 128.4, 123.5, 121.3, 106.1, 12.6.

GC-MS (EI, 70 eV) *m/z* 326 ($[M]^+$)

1-(4-Iodophenyl)-2,5-dimethyl-1*H*-pyrrole[18, 19]



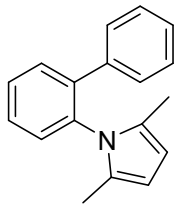
Yellow solid, mp 63-65 ˚C

^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.80 – 7.79 (d, J = 8.5 Hz, 2H), 6.97 – 6.96 (d, J = 8.0 Hz, 2H), 5.90 (s, 2H), 2.03 (s, 6H).

^{13}C-NMR (125 MHz, $CDCl_3$) δ 138.8, 138.3, 130.2, 128.6, 106.2, 92.9, 13.0.

GC-MS (EI, 70 eV) *m/z* 297 ($[M]^+$).

1-([1,1'-Biphenyl]-2-yl)-2,5-dimethyl-1*H*-pyrrole[21]



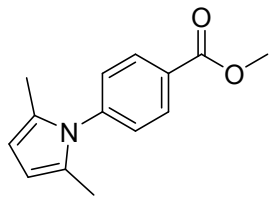
Yellow solid, mp 98-99 ˚C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.55 – 7.53 (dd, J = 1.5 Hz, 8.0 Hz, 1H), 7.48 – 7.45 (dt, J = 1.5 Hz, 1H), 7.43 – 7.39 (dt, J = 1.5 Hz, 1H), 7.25 – 7.22 (m, 4H), 7.01 – 6.99 (dd, J = 2.0 Hz, 2H), 5.76 (s, 2H), 1.84 (s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 140.4, 138.7, 136.4, 130.82, 129.9, 128.5, 128.5, 128.3, 128.2, 128.0, 127.3, 105.8, 12.9.

GC-MS (EI, 70 eV) *m/z* 247 ($[M]^+$)

Methyl 4-(2,5-dimethyl-1*H*-pyrrol-1-yl)benzoate[22-25]



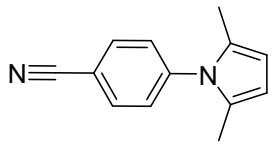
White solid, mp 100-102 ˚C

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.16 – 8.13 (m, 2H), 7.30 – 7.27 (m, 2H), 5.92 (s, 2H), 3.96 (s, 3H), 2.05 (s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 166.4, 143.2, 130.5, 129.3, 128.6, 128.1, 106.5, 52.3, 13.0.

GC-MS (EI, 70 eV) *m/z* 229 ($[M]^+$)

1-(4-Cyanophenyl)-2,5-Dimethyl-1*H*-pyrrole[1, 2, 5, 7]



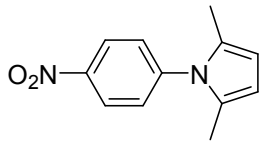
White solid, mp 93-94 ˚C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.79 – 7.77 (m, 2H), 7.35 – 7.33 (m, 2H), 5.94 (s, 2H), 2.05 (s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 143.1, 133.1, 129.0, 128.5, 118.2, 111.5, 107.2, 13.1.

GC-MS (EI, 70 eV) *m/z* 196 ($[M]^+$)

2,5-Dimethyl-1-(4-nitrophenyl)-1*H*-pyrrole[2, 4, 5, 7, 17]



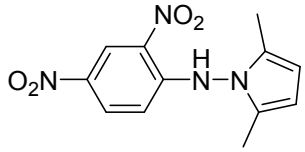
Yellow solid, mp 144-146 ˚C

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.35 – 8.34 (d, J = 9.0 Hz, 2H), 7.40 – 7.38 (d, J = 9.0 Hz, 2H), 5.96 (s, 2H), 2.07 (s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 146.8, 144.8, 128.8, 124.6, 109.0, 107.4, 29.7.

GC-MS (EI, 70 eV) *m/z* 216 ($[M]^+$)

***N*-(2,4-Dinitrophenyl)-2,5-dimethyl-1*H*-pyrrol-1-amine[10, 14-16]**

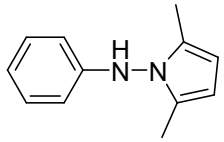


Yellow solid, mp 182-184 ˚C

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.96 (s, 1H), 9.19 – 9.18 (d, J = 2.5 Hz, 1H), 8.27 – 8.24 (m, 1H), 6.22 – 6.20 (d, J = 9.5 Hz, 1H), 5.94 (s, 2H), 2.08 (s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 148.7, 139.2, 130.9, 127.4, 123.5, 114.6, 105.7, 11.1.

2,5-Dimethyl-*N*-phenyl-1*H*-pyrrol-1-amine[10-13]



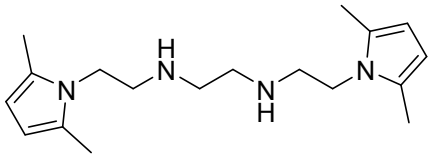
Yellow solid, mp 82-85 ˚C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32 – 7.29 (t, J = 9.0 Hz, 2H), 6.92 – 6.90 (t, J = 7.5 Hz, 1H), 6.86 – 6.83 (t, J = 7.0 Hz, 1H), 6.47 – 6.46 (d, J = 7.5 Hz, 2H), 6.32 (s, 1H), 5.87 (s, 2H), 2.14 (s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 142.4, 120.5, 118.6, 113.2, 112.0, 103.5, 14.8.

GC-MS (EI, 70 eV) *m/z* 186 ($[M]^+$)

***N*^{1},*N*^{2}-bis(2-(2,5-Dimethyl-1*H*-pyrrol-1-yl)ethyl)ethane-1,2-diamine**



Yellow oil

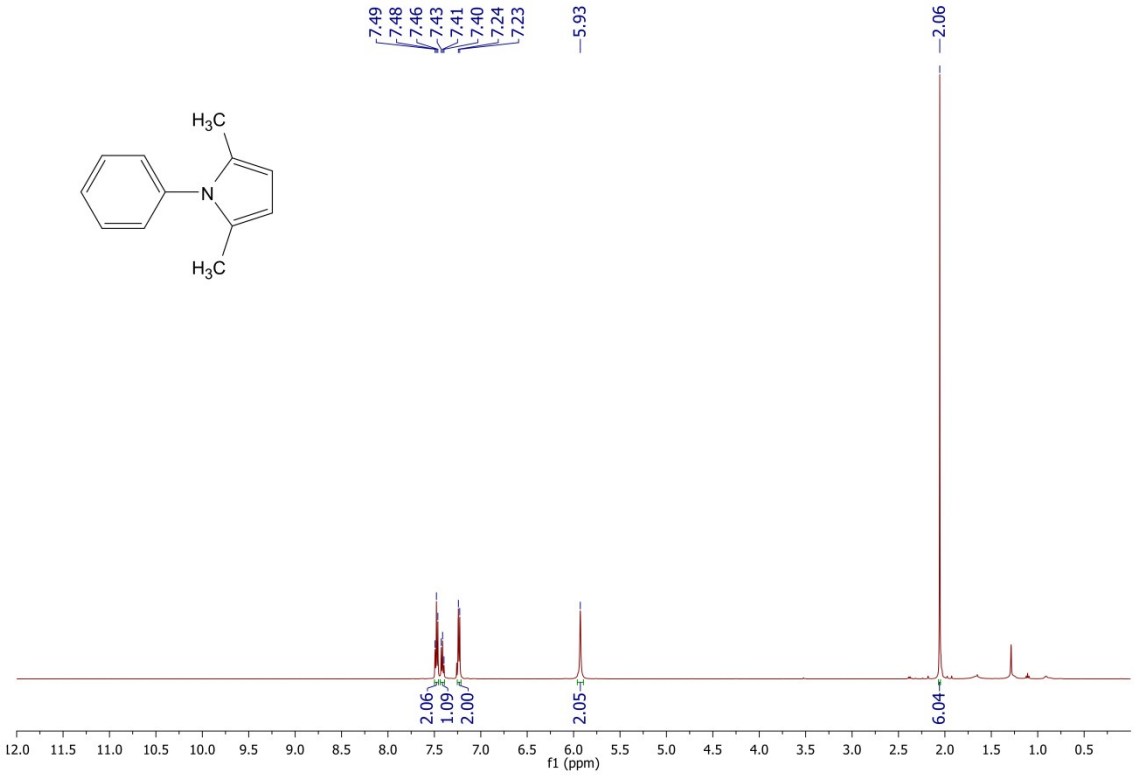
^{1}H NMR (500 MHz, $CDCl_3$) δ 5.77 – 5.76 (d, J = 5.0 Hz, 4H), 3.88 – 3.85 (t, J = 7.0 Hz, 4H), 2.83 – 2.81 (t, J = 7.0 Hz, 4H), 2.71 (s, 4H), 2.23 (s, 12H).

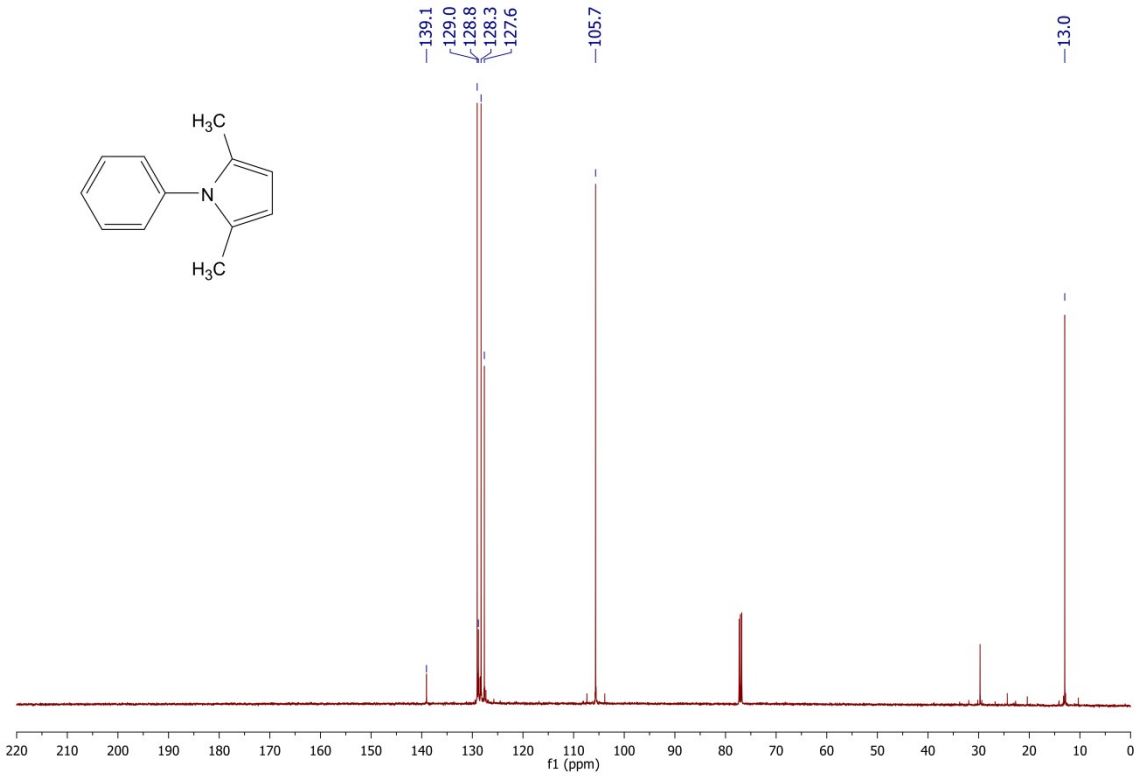
^{13}C NMR (125 MHz, $CDCl_3$) δ 127.6, 105.4, 49.7, 49.0, 43.7, 12.6.

HRMS (ESI) *m/z* calcd for $[M + H]^+$ $C_{18}H_{31}N_4^+$ 303.2543, found 303.2575.

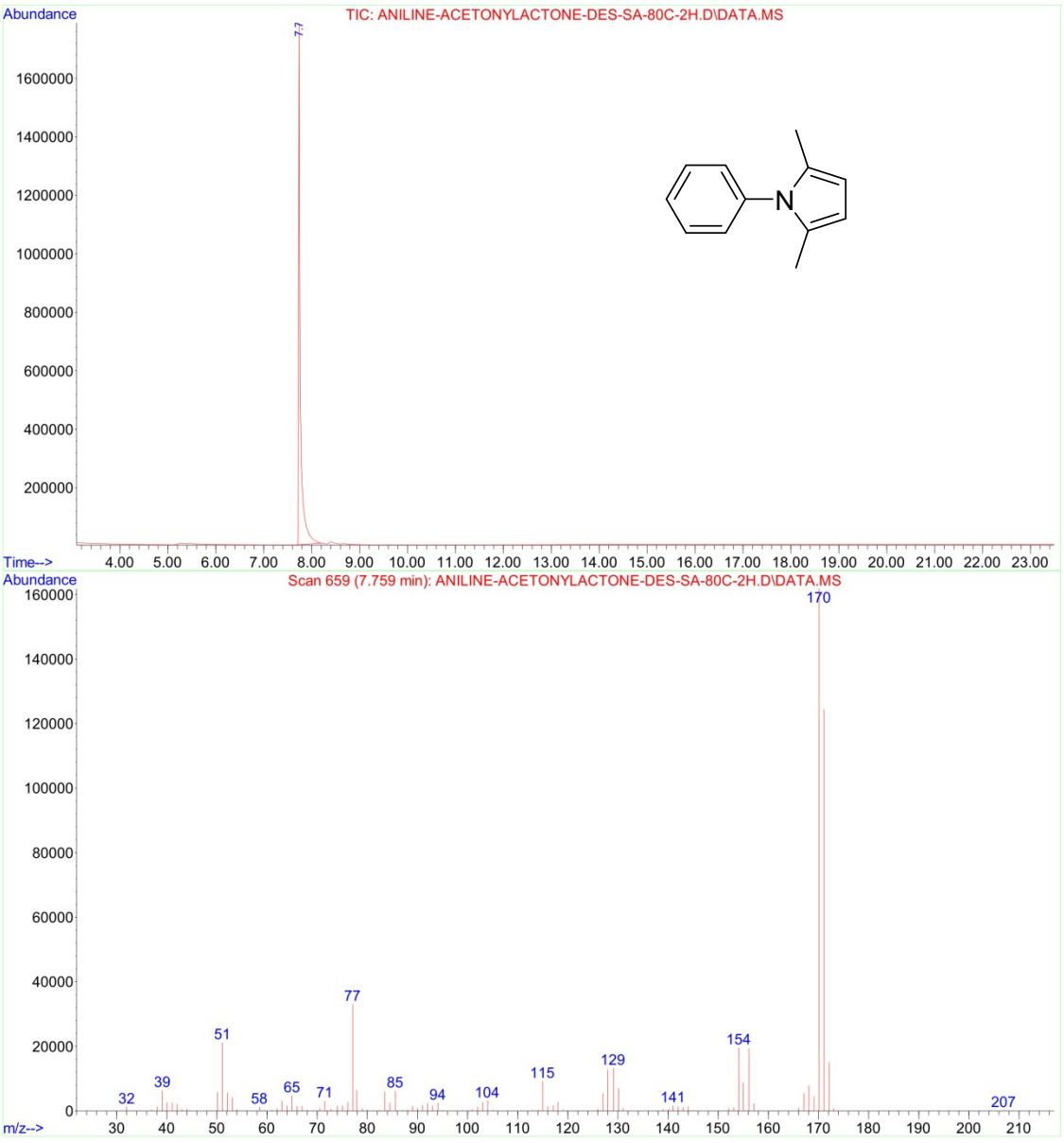
Section S5. ^{1}H, ^{13}C NMR and HRMS spectroscopy

^{1}H NMR, ^{13}C NMR, and GC-MS of 2,5-Dimethyl-1-phenyl-1*H*-pyrrole

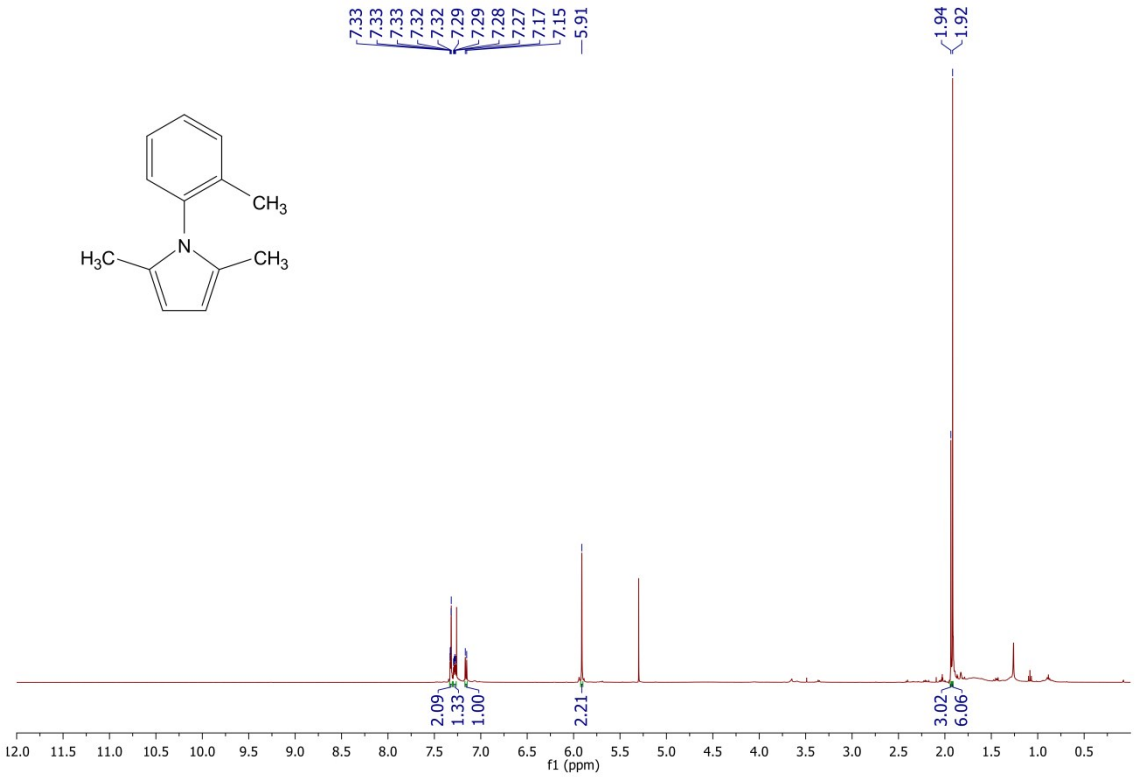


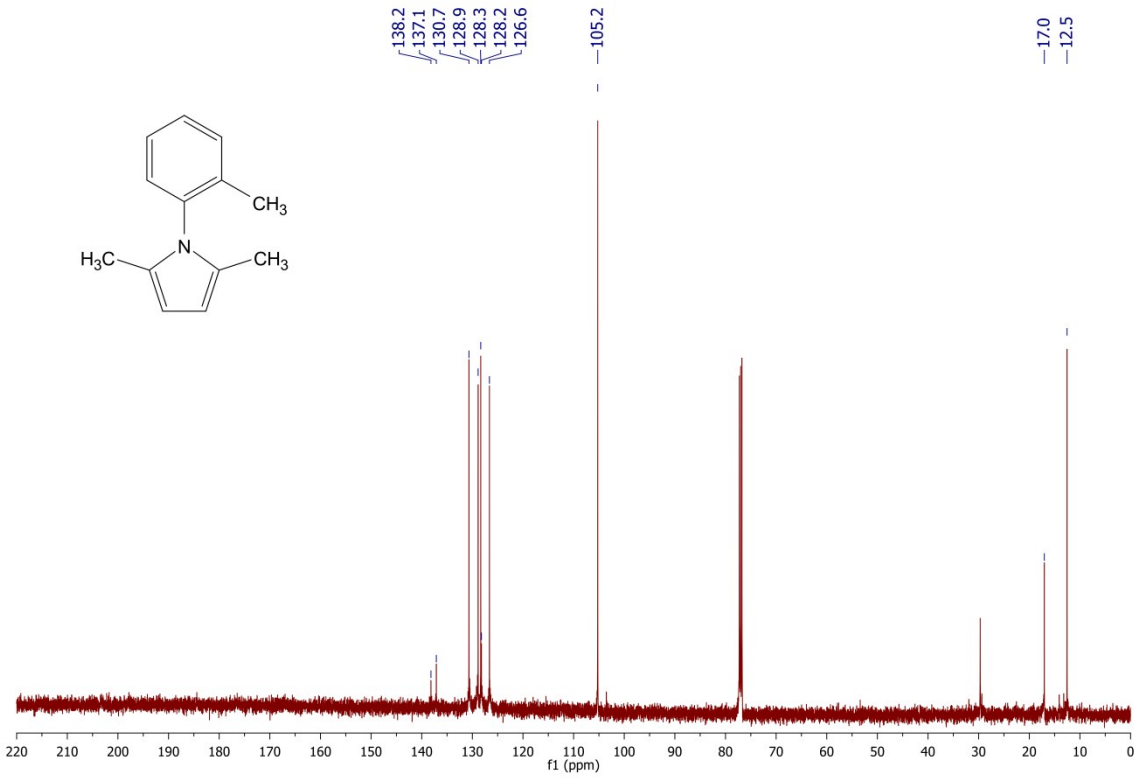


File :C:\GC-MS\2016\08.01.2016\ANILINE-ACETONYLACTONE-DES-SA-80C-2
... H.D
Operator : TRUONG HAI
Instrument : GCMSD
Acquired : 1 Aug 2016 16:44 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Sample Name: ANILINE-ACETONYLACTONE-DES-SA-80C-2H
Misc Info :

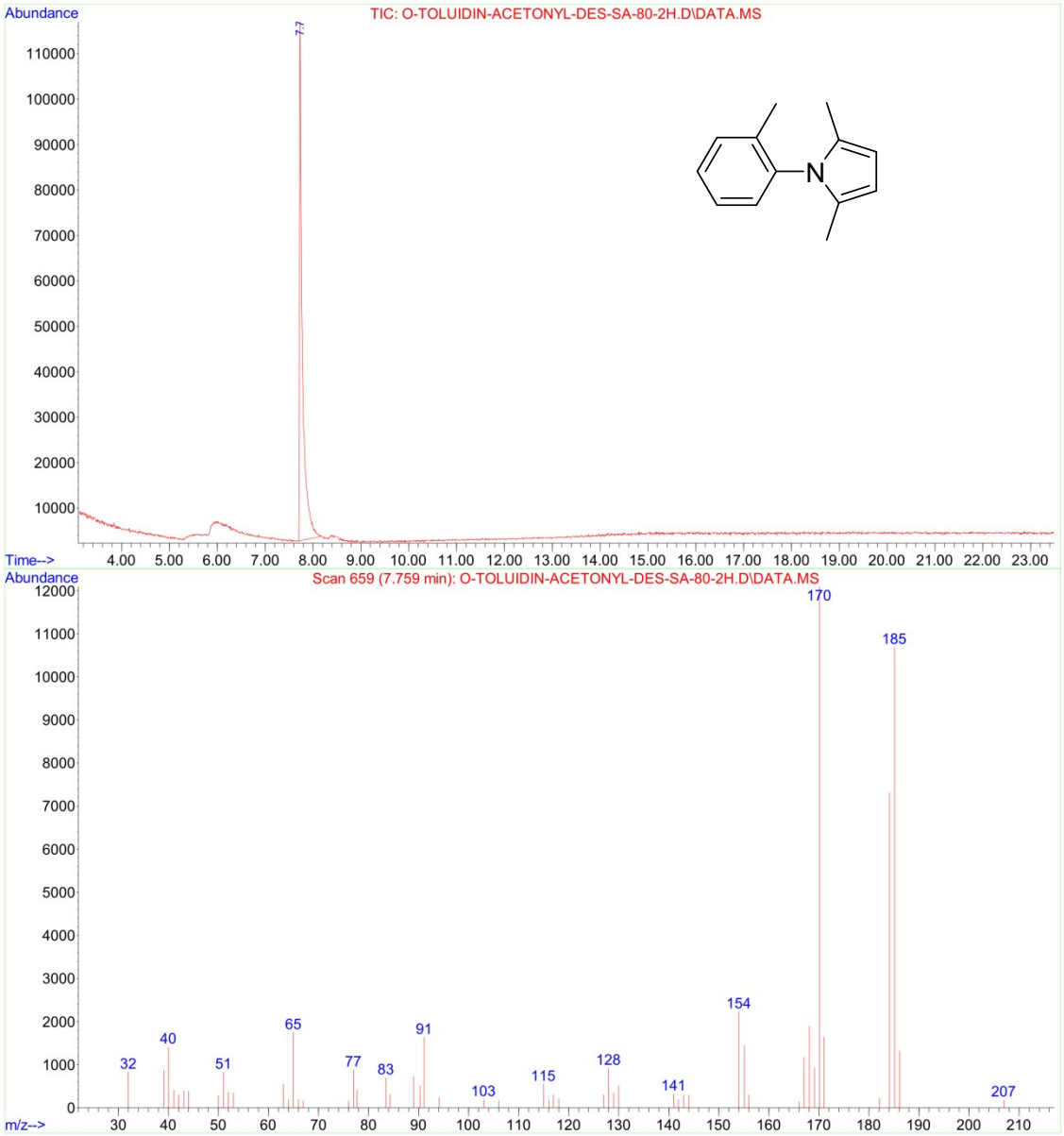


^{1}H NMR, ^{13}C NMR**, and GC-MS of 2,5-Dimethyl-1-(*o*-tolyl)-1*H*-pyrrole**

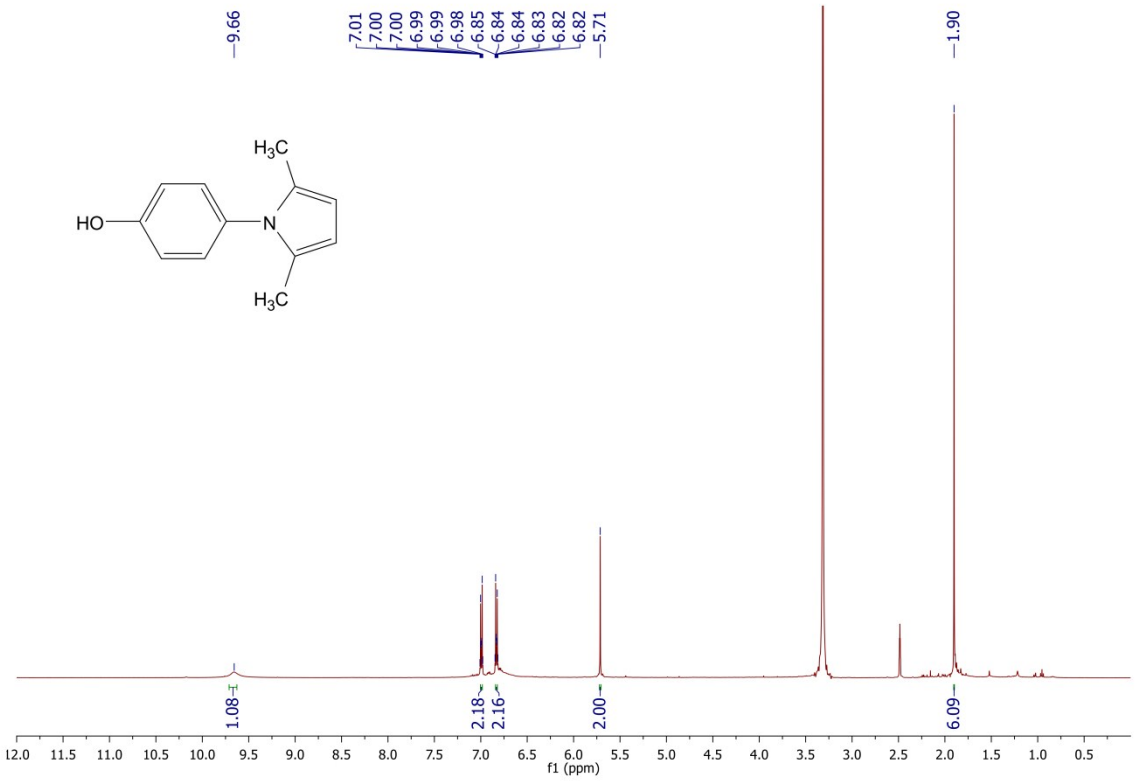


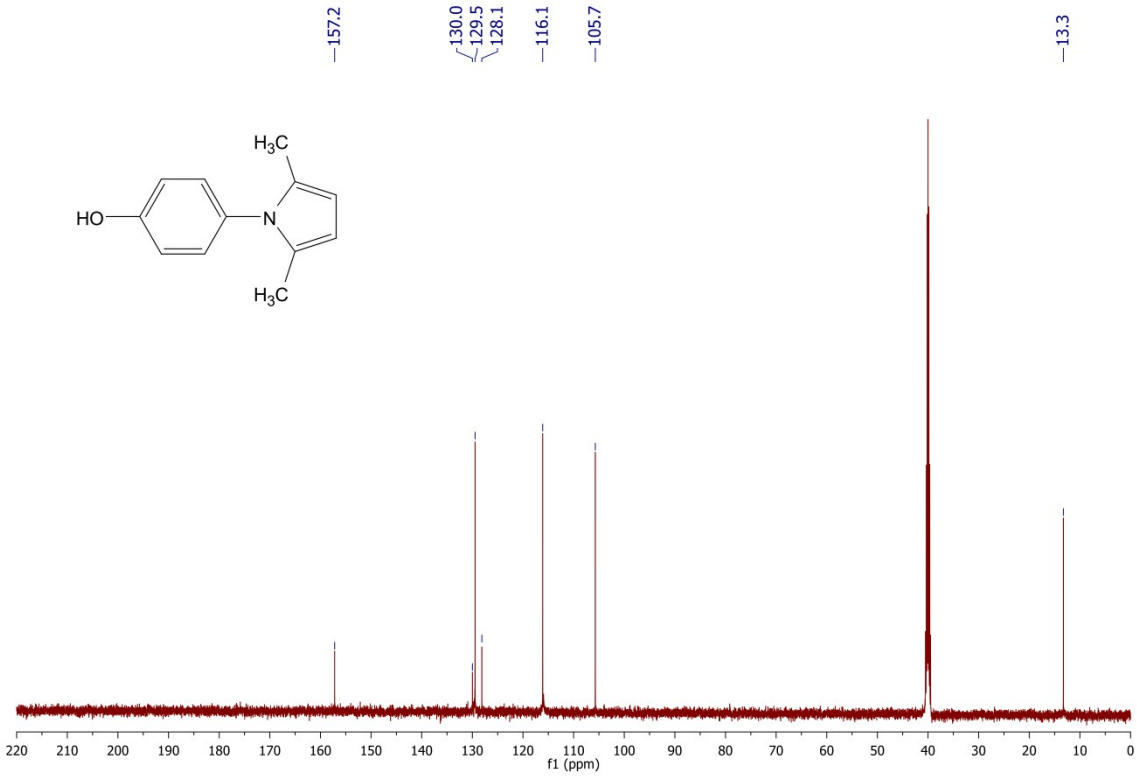


File :C:\GC-MS\2016\08.03.2016\O-TOLUIDIN-ACETONYL-DES-SA-80-2H.D
Operator : TRUONG HAI
Acquired : 4 Aug 2016 11:12 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Instrument : GCMSD
Sample Name: O-TOLUIDIN-ACETONYL-DES-SA-80-2H
Misc Info :
Vial Number: 3

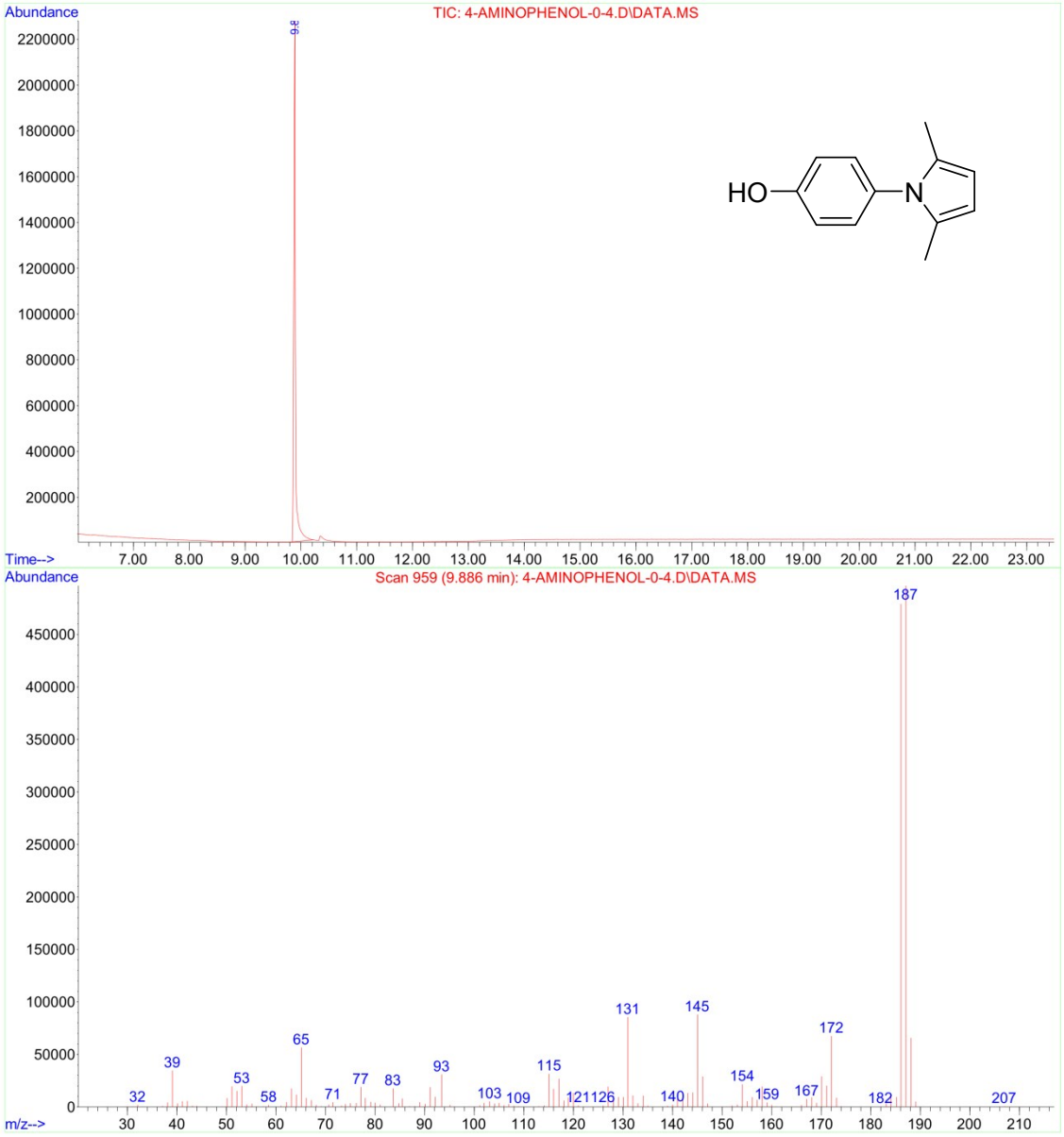


^{1}H NMR, ^{13}C NMR**, and GC-MS of 1-(4-Hydroxyphenyl)-2,5-dimethyl-1*H*-pyrrole**

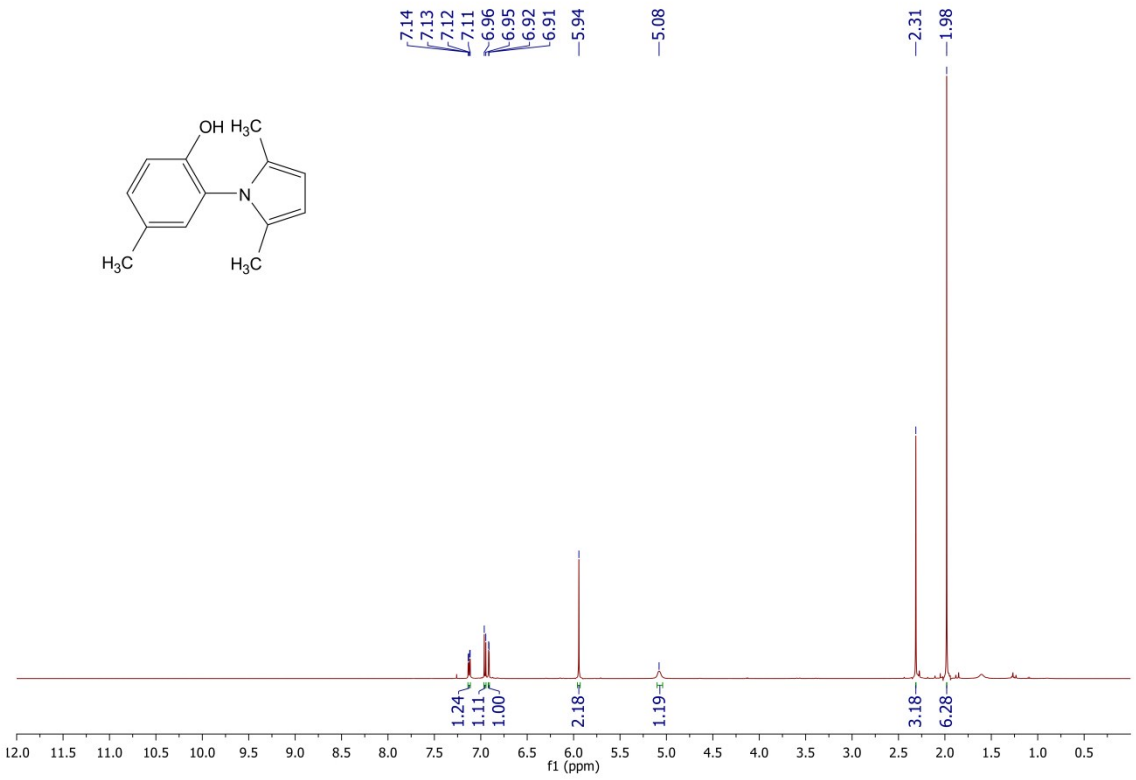


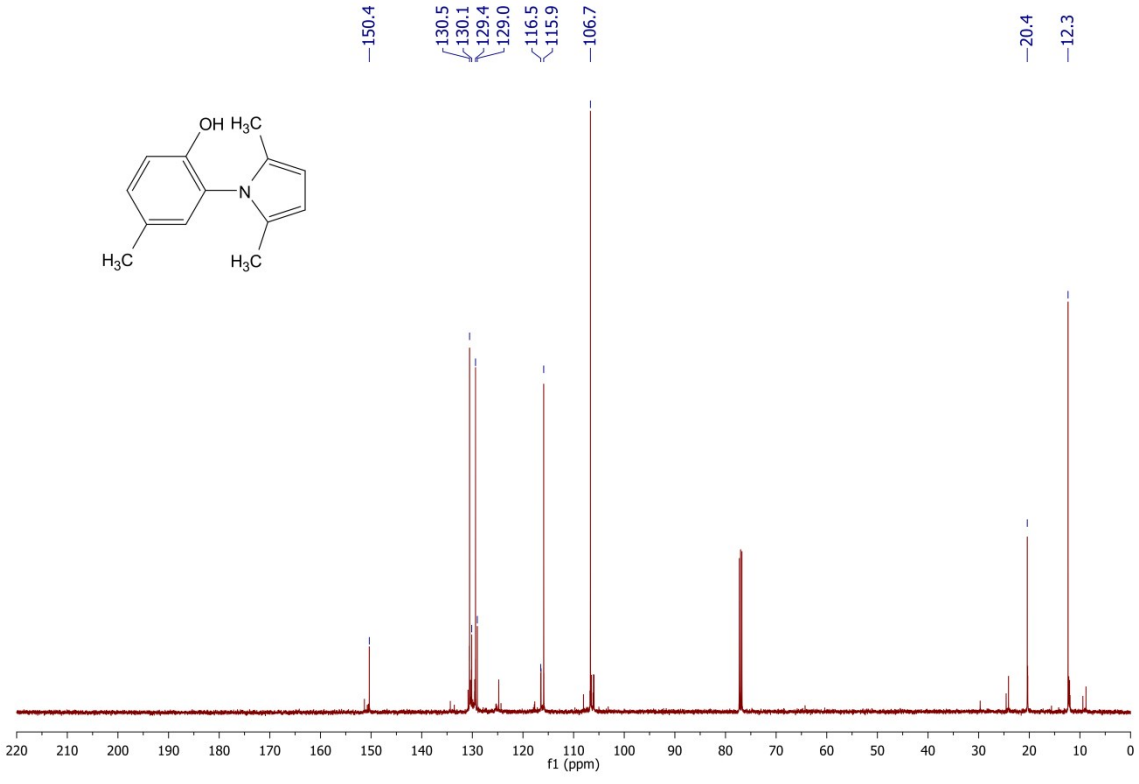


File :C:\GC-MS\2016\11.16.2016\4-AMINOPHENOL-0-4.D
Operator : TRUONG HAI
Acquired : 16 Nov 2016 16:21 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Instrument : GCMSD
Sample Name: 4-AMINOPHENOL-0-4
Misc Info :
Vial Number: 2



^{1}H NMR, ^{13}C NMR**, and HRMS of 1-(2'-Hydroxy-5'-methylphenyl)-2,5-dimethyl-1*H*-pyrrole**





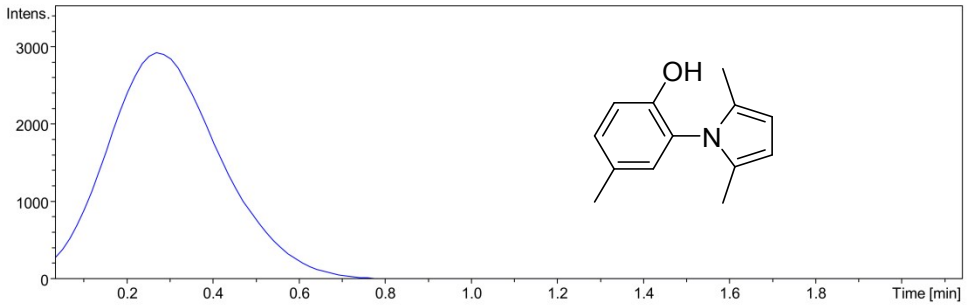
Display Report

Analysis Info

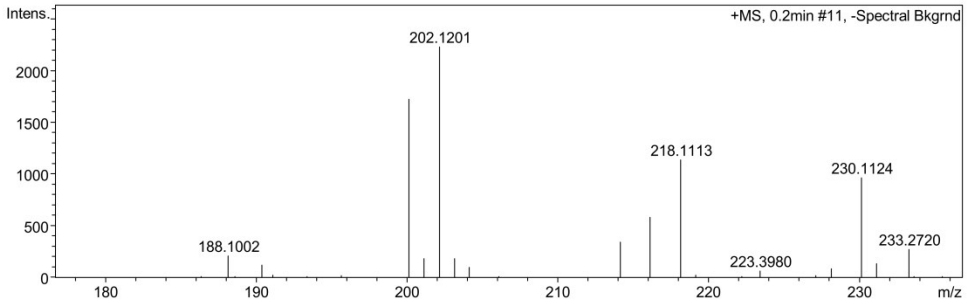
Analysis Name	D:\Data\2016\2 ami_1-b,1_01_2267.d	Acquisition Date	12/30/2016 3:54:34 PM
Method	dmm 2017.m	Operator	Anh Mai
Sample Name	2 ami	Instrument	micrOTOF-Q 10187
Comment			

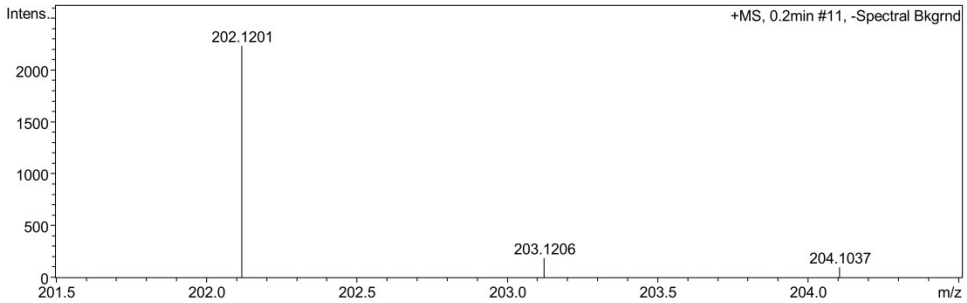
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4000 V	Set Dry Heater	200 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	9.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Source

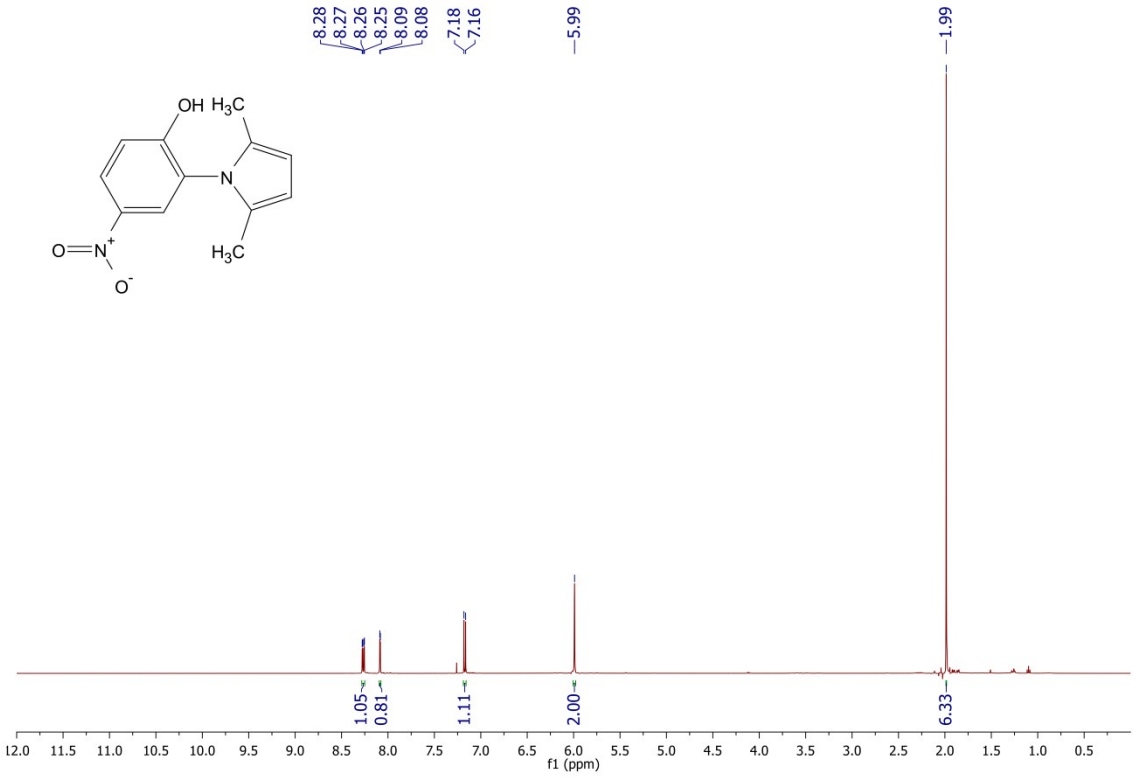


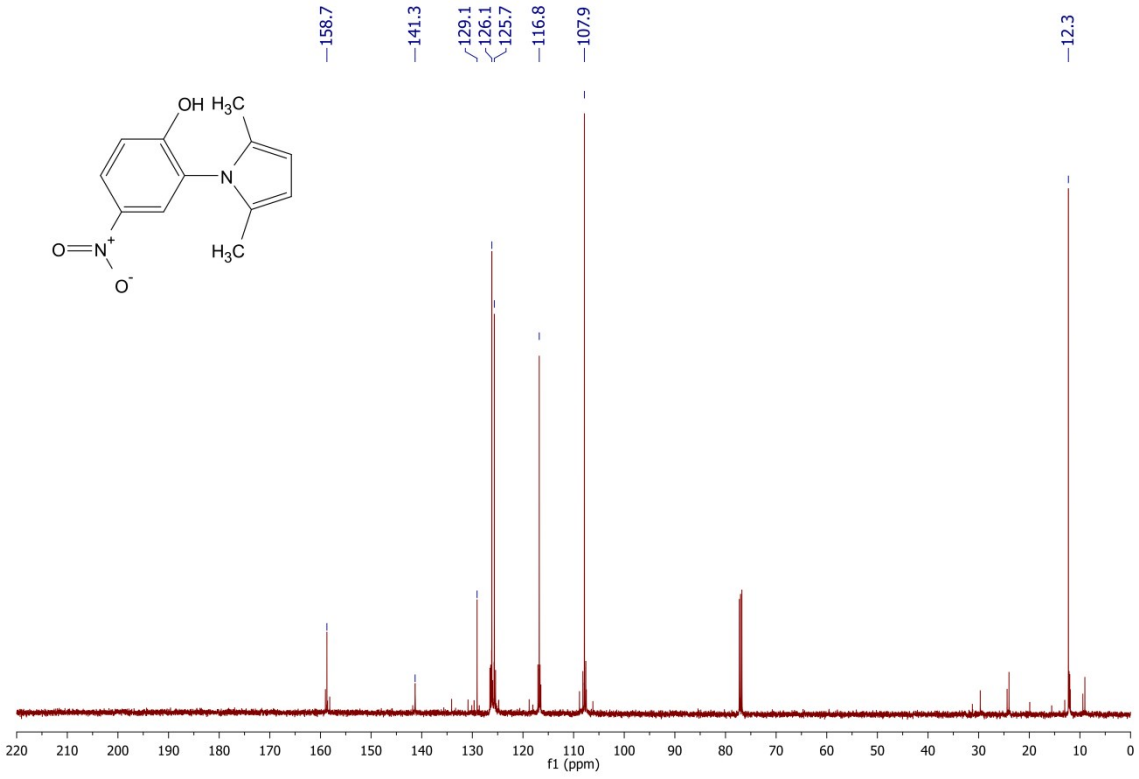
EIC 202.0000 +All MS, -Spectral Bkgrnd, Smoothed (4.02,2,GA)





^{1}H NMR, ^{13}C NMR**, and HR-MS of 1-(2'-Hydroxy-5'-nitrophenyl)-2,5-dimethyl-1*H*-pyrrole**





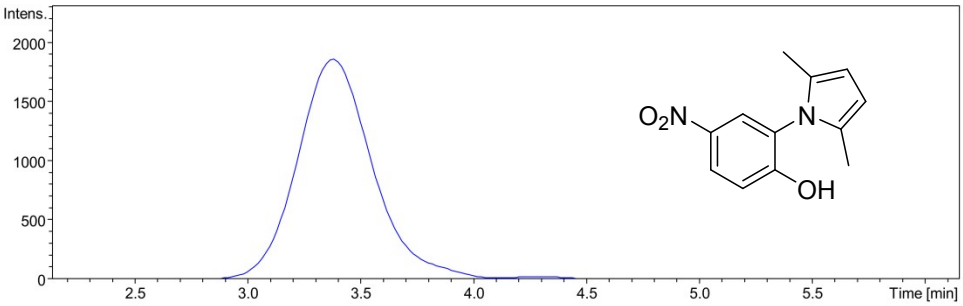
Display Report

Analysis Info

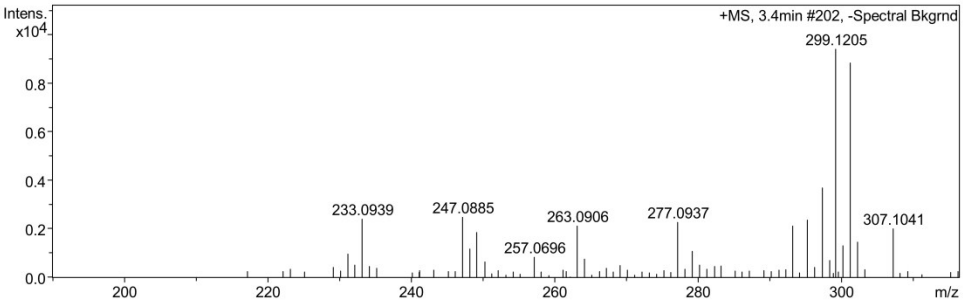
Analysis Name	D:\Data\2016\2 amino 4 ntro_1-b,3_01_2264.d	Acquisition Date	12/29/2016 6:23:13 PM
Method	dmm 2017.m	Operator	Anh Mai
Sample Name	2 amino 4 ntro	Instrument	micrOTOF-Q 10187
Comment			

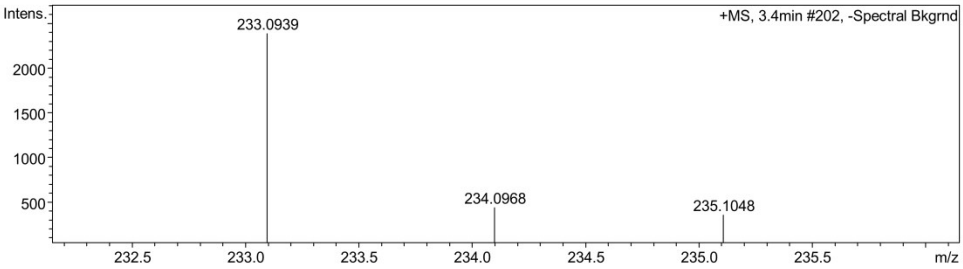
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4000 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	9.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	450.0 Vpp	Set Divert Valve	Source

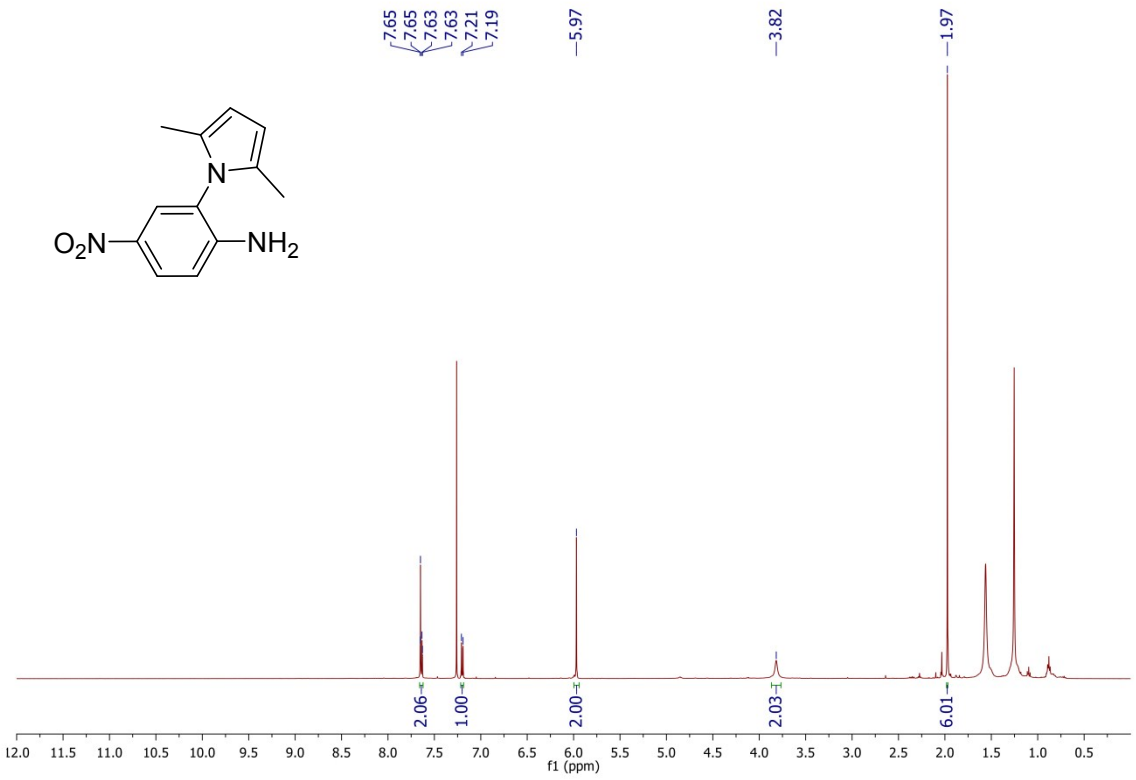


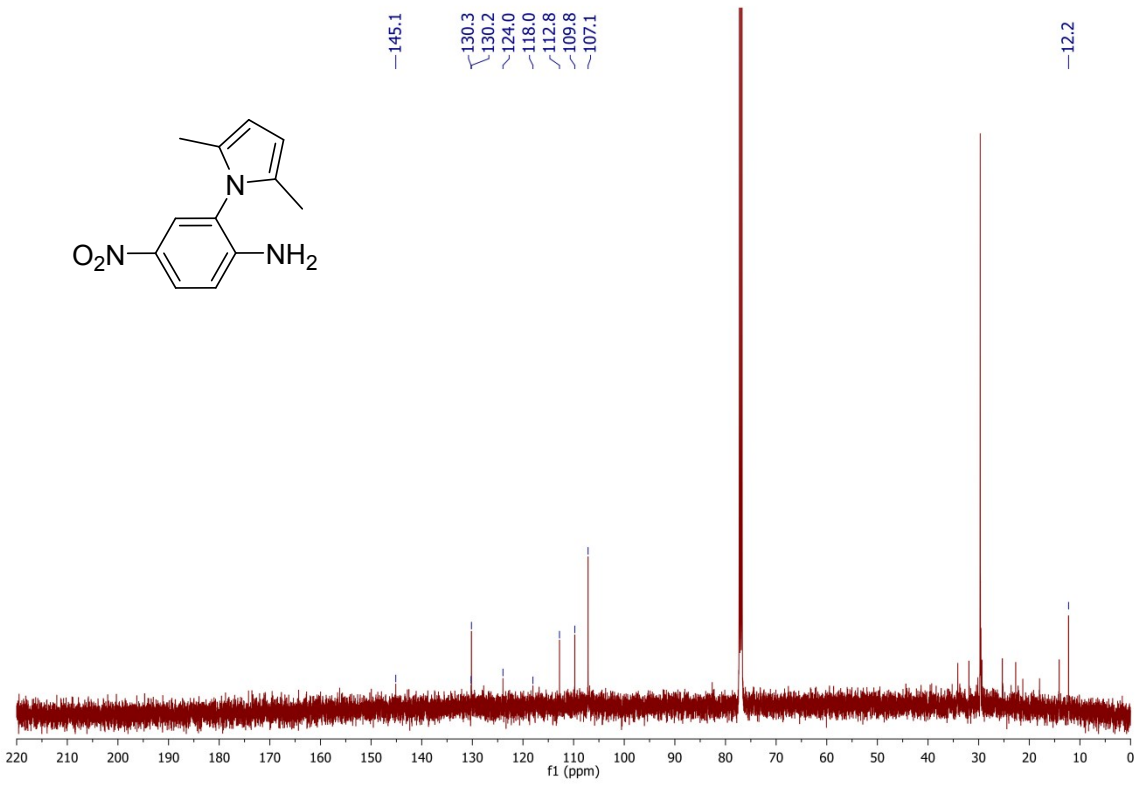
EIC 233.0000 +All MS, -Spectral Bkgrnd, Smoothed (4.01,2,GA)





^{1}H NMR, ^{13}C NMR**, and GC-MS of 1-(2'-Amino-4'-nitrophenyl)-2,5-dimethyl-1*H*-pyrrole**





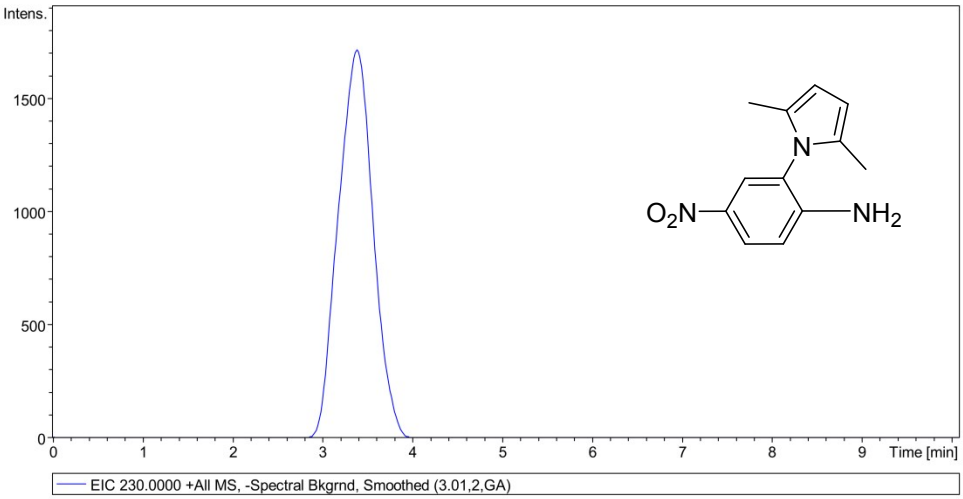
Display Report

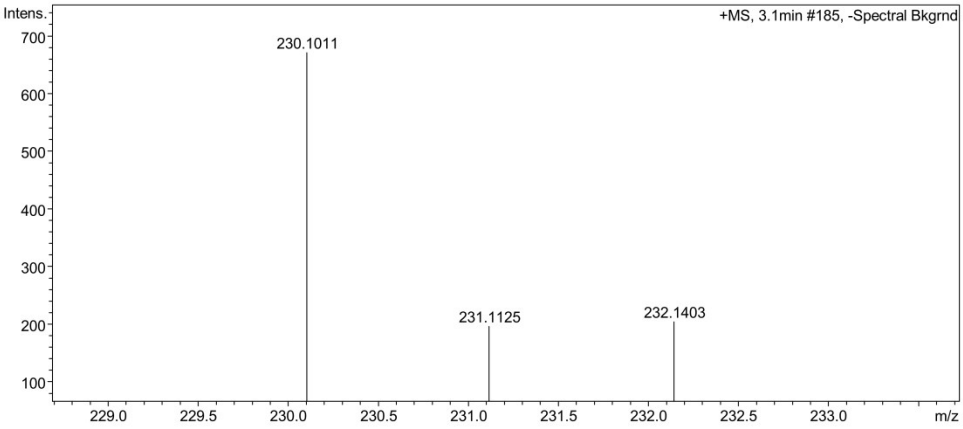
Analysis Info

Analysis Name	D:\Data\2016\4-ni_1-b,2_01_2263.d	Acquisition Date	12/29/2016 6:12:14 PM
Method	dmm 2017.m	Operator	Anh Mai
Sample Name	4-ni	Instrument	micrOTOF-Q 10187
Comment			

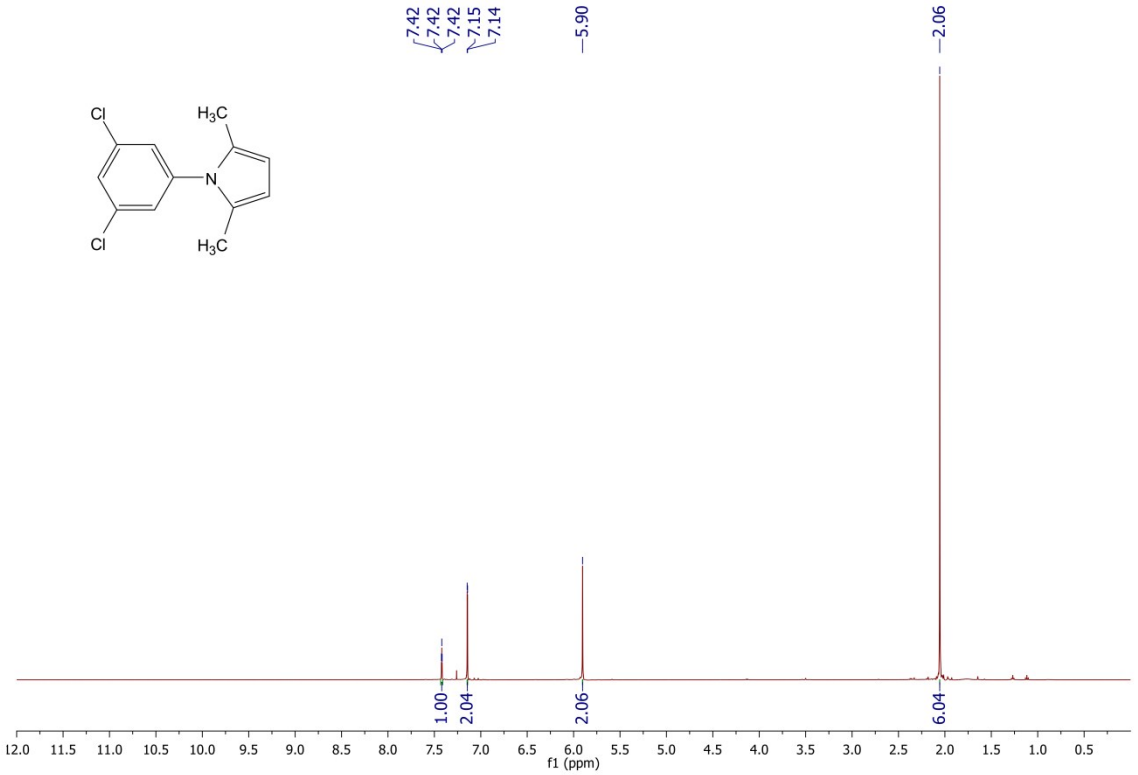
Acquisition Parameter

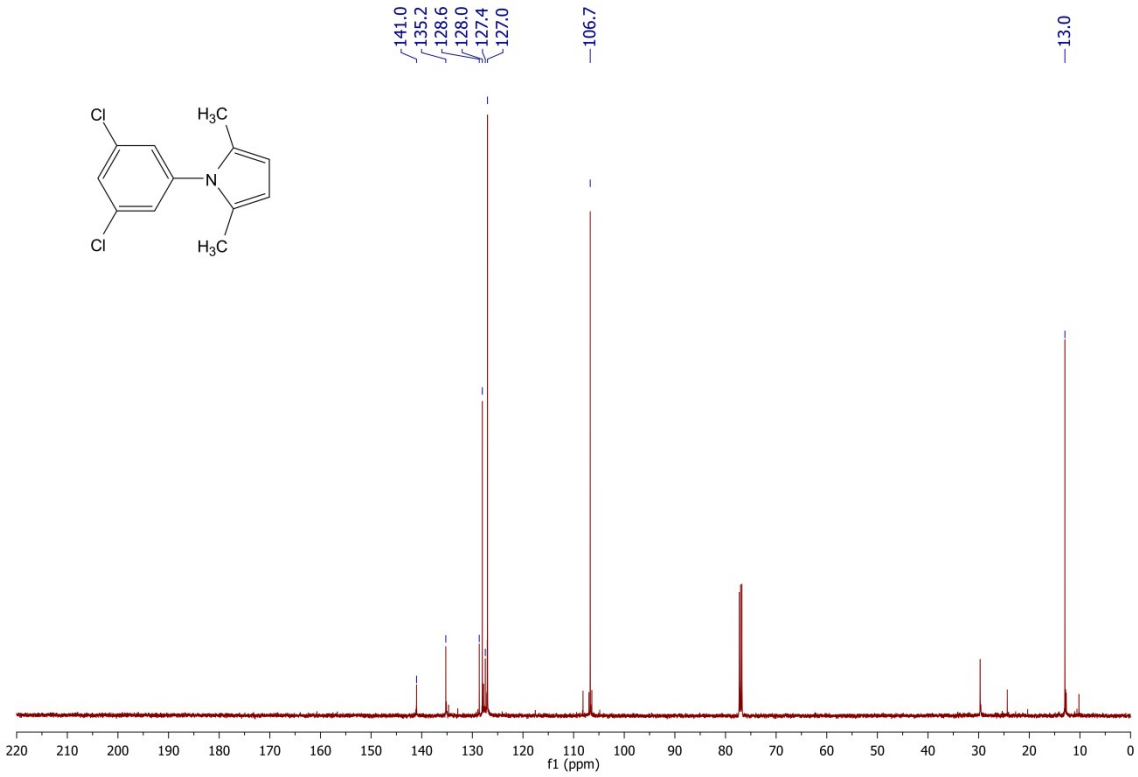
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4000 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	9.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	450.0 Vpp	Set Divert Valve	Source



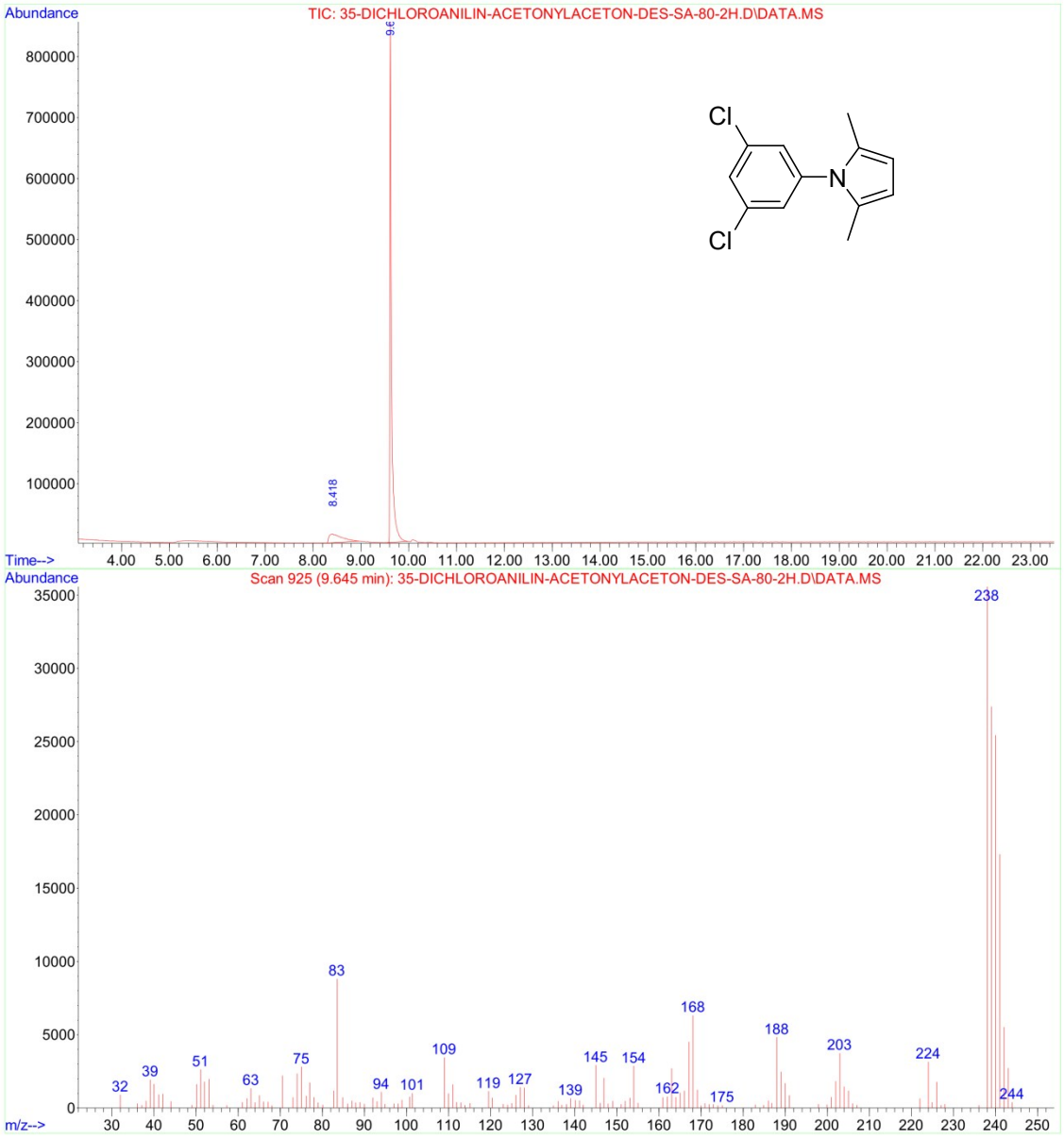


^{1}H NMR, ^{13}C NMR**, and GC-MS of 1-(3,5-Dichlorophenyl)-2,5-dimethyl-1*H*-pyrrole**

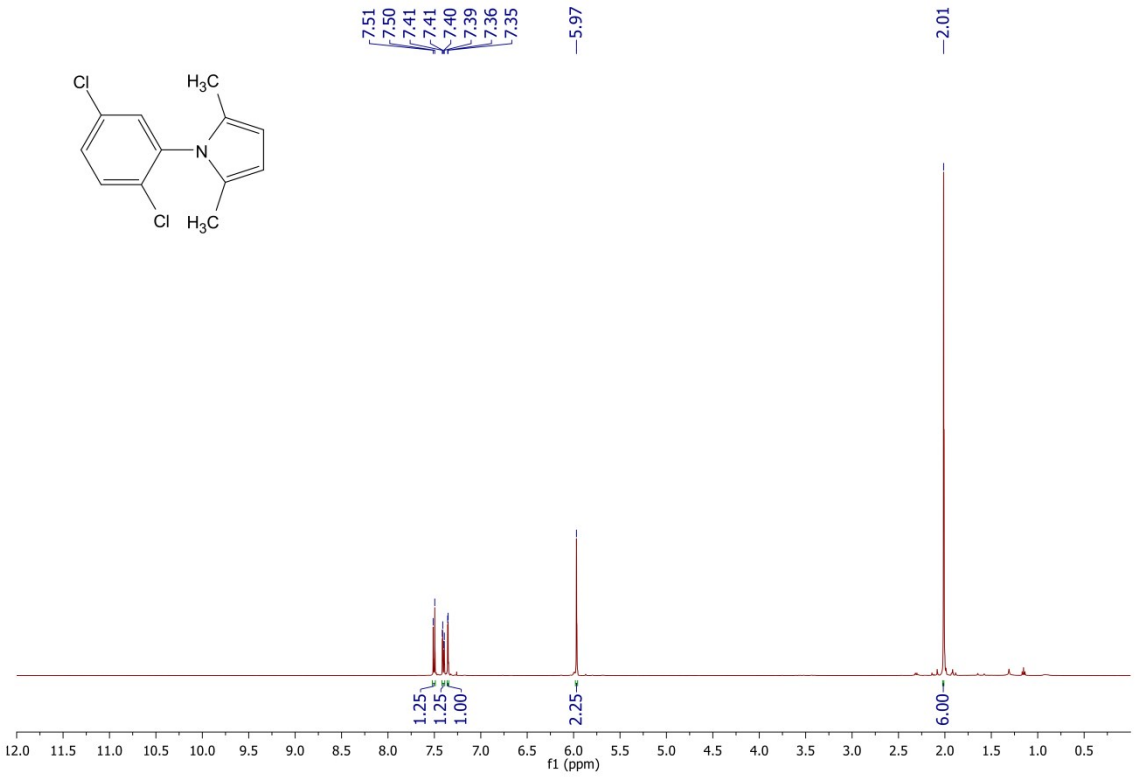


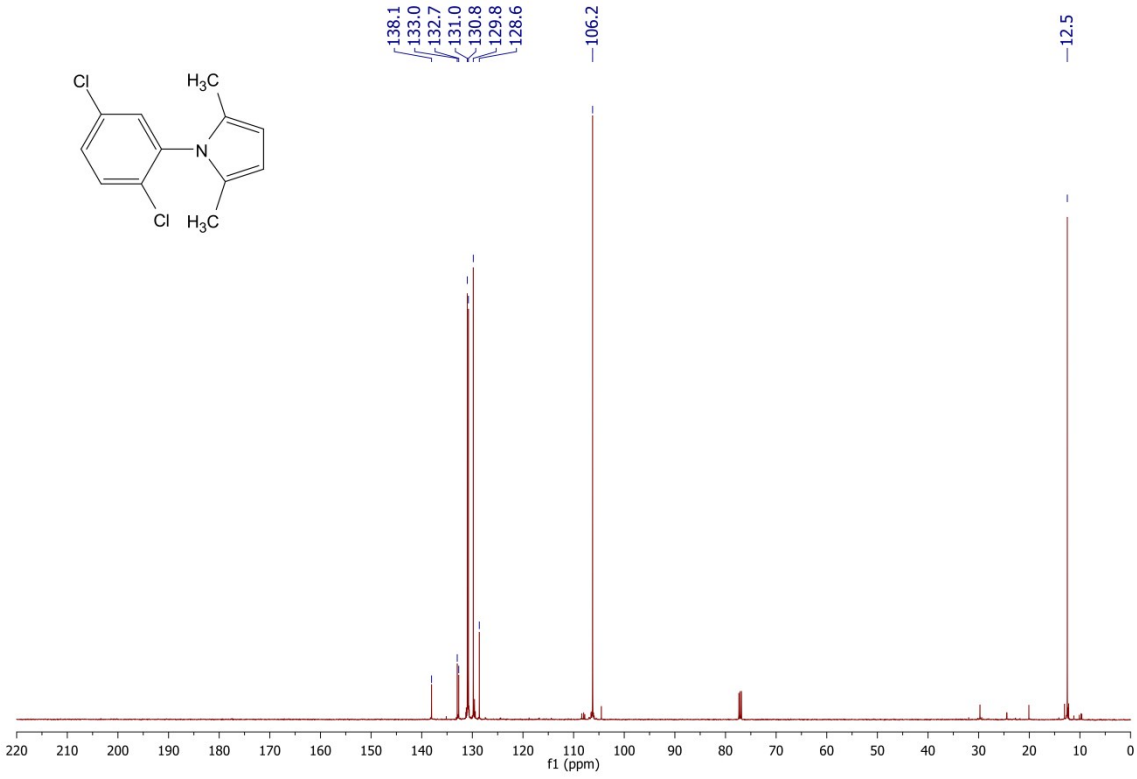


File :C:\GC-MS\2016\08.03.2016\35-DICHLOROANILIN-ACETONYLACETON-DE
... S-SA-80-2H.D
Operator : TRUONG HAI
Instrument : GCMSD
Acquired : 3 Aug 2016 18:03 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Sample Name: 35-DICHLOROANILIN-ACETONYLACETON-DES-SA-80-2H
Misc Info :

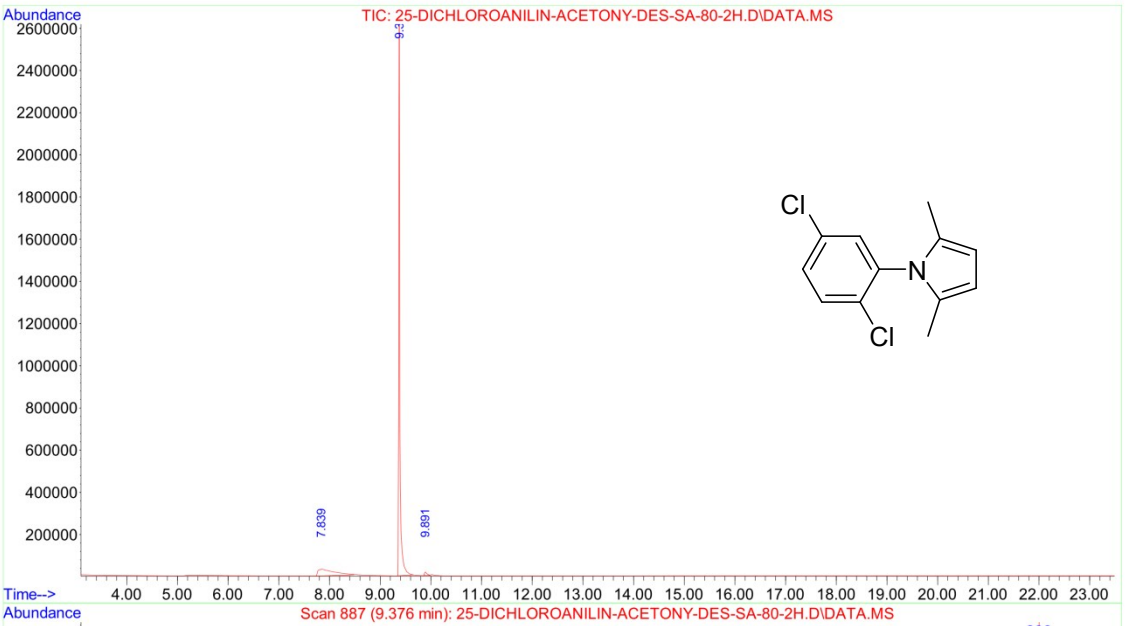


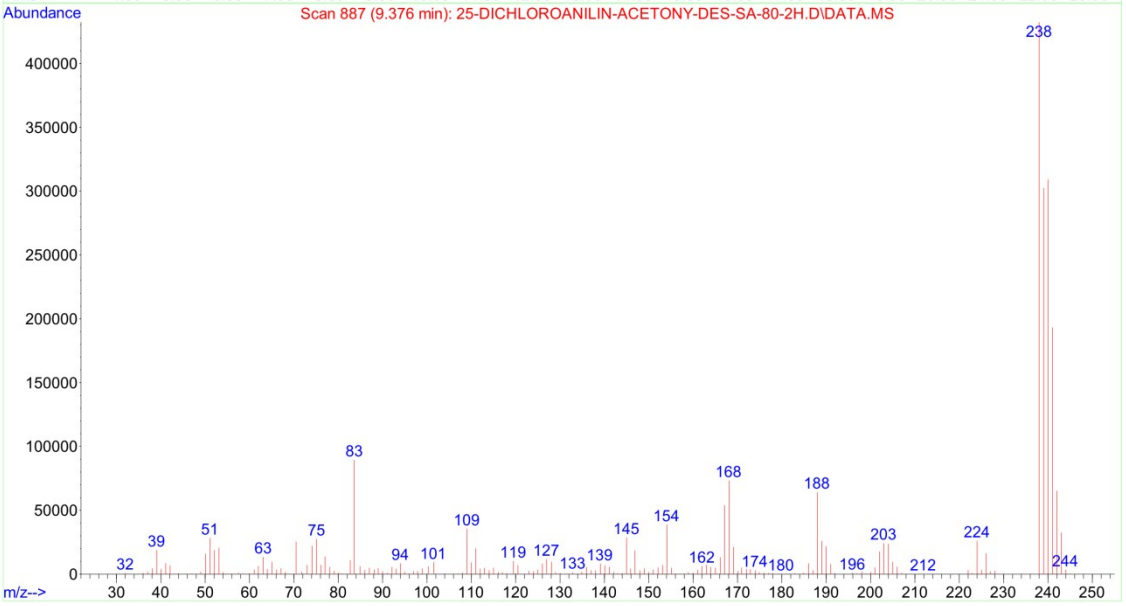
^{1}H NMR, ^{13}C NMR**, and GC-MS of 1-(2,5-Dichlorophenyl)-2,5-dimethyl-1*H*-pyrrole**



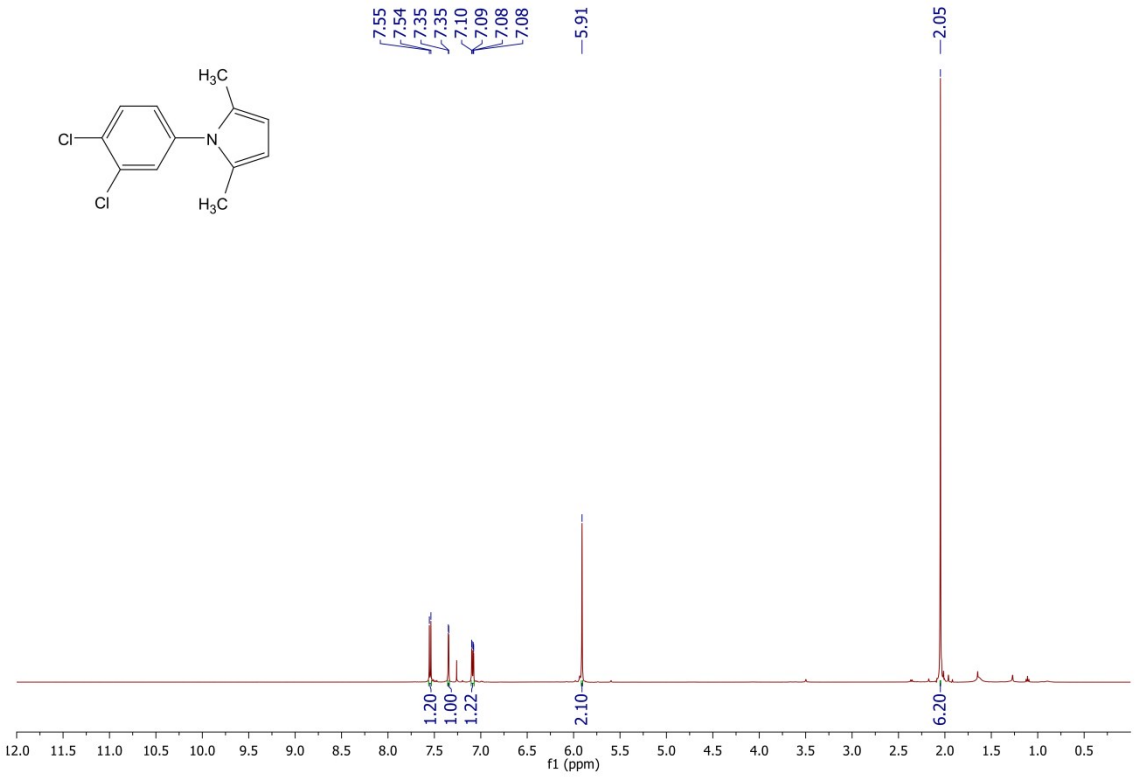


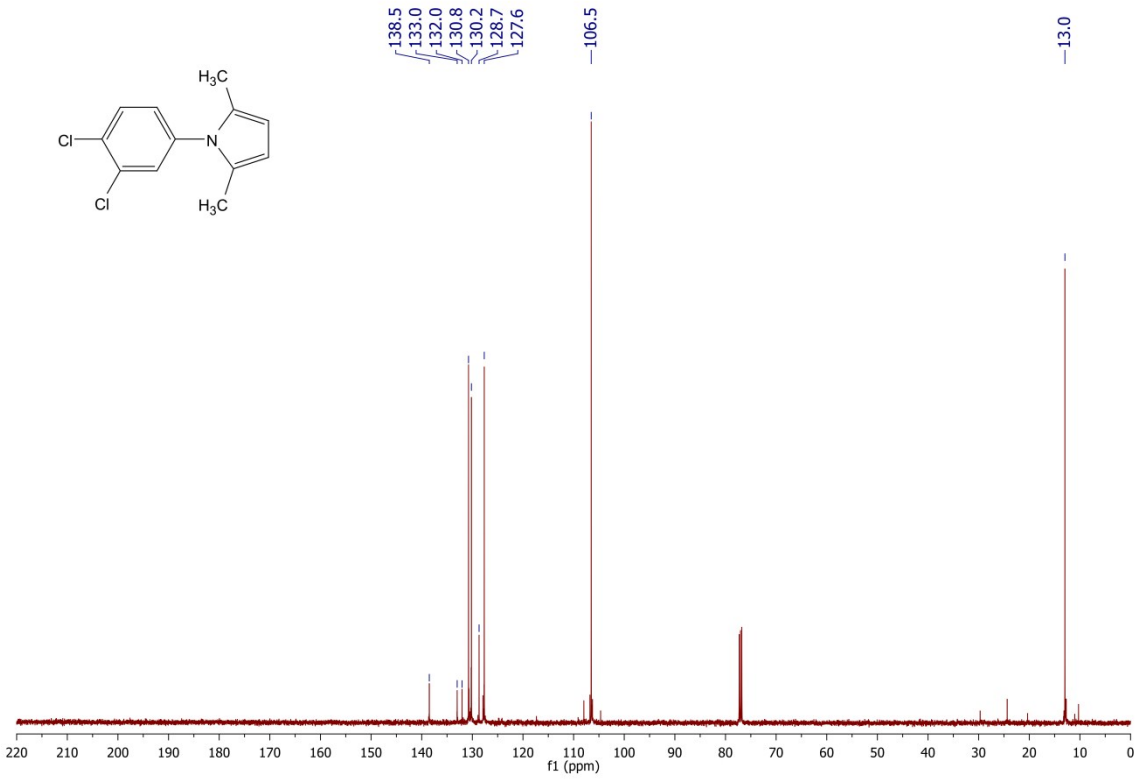
File :C:\GC-MS\2016\08.03.2016\25-DICHLOROANILIN-ACETONY-DES-SA-80
... -2H.D
Operator : TRUONG HAI
Instrument : GCMSD
Acquired : 8 Aug 2016 16:19 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Sample Name: 25-DICHLOROANILIN-ACETONY-DES-SA-80-2H
Misc Info :



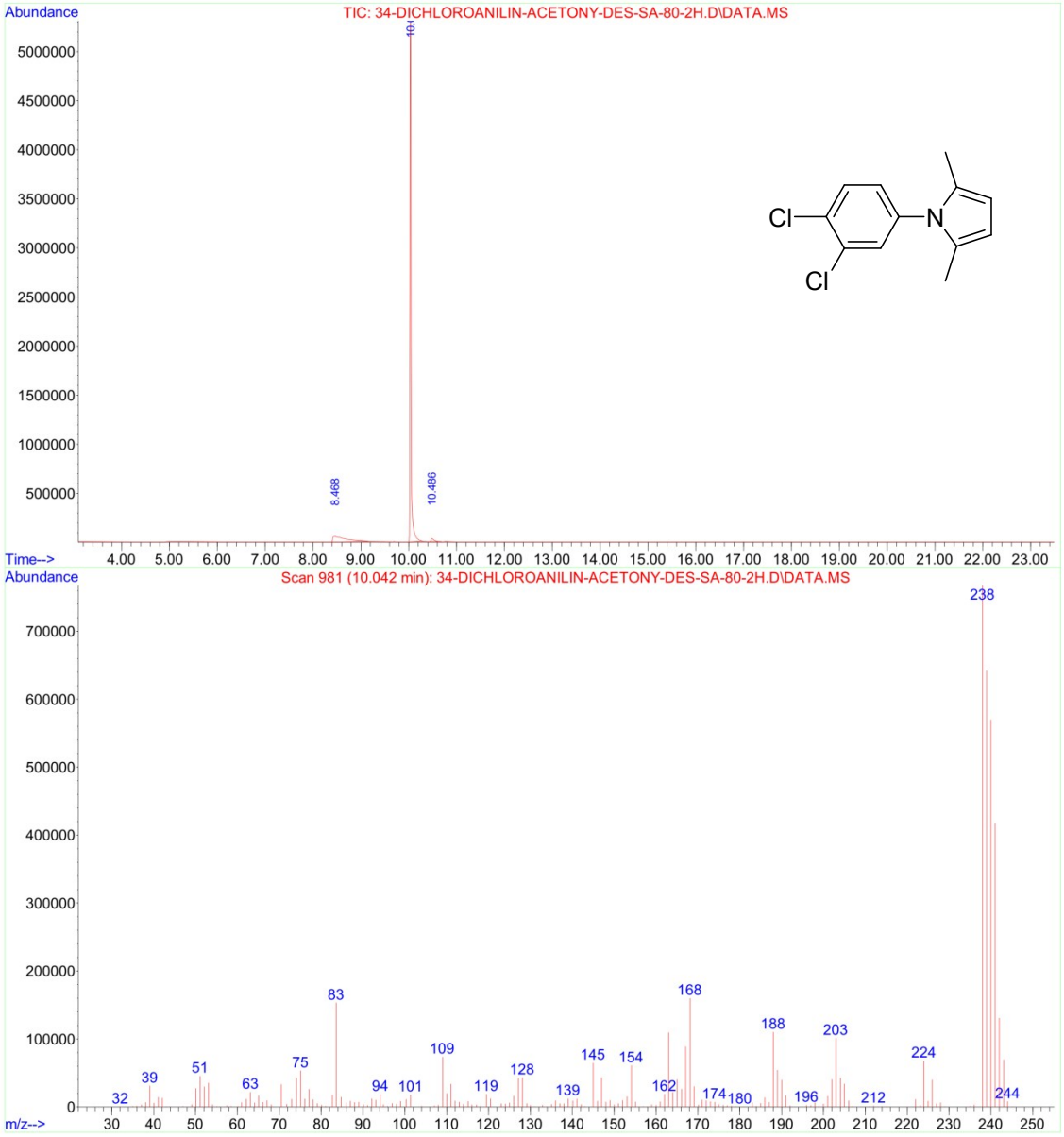


^{1}H NMR, ^{13}C NMR**, and GC-MS of 1-(3,4-Dichlorophenyl)-2,5-dimethyl-1*H*-pyrrole**

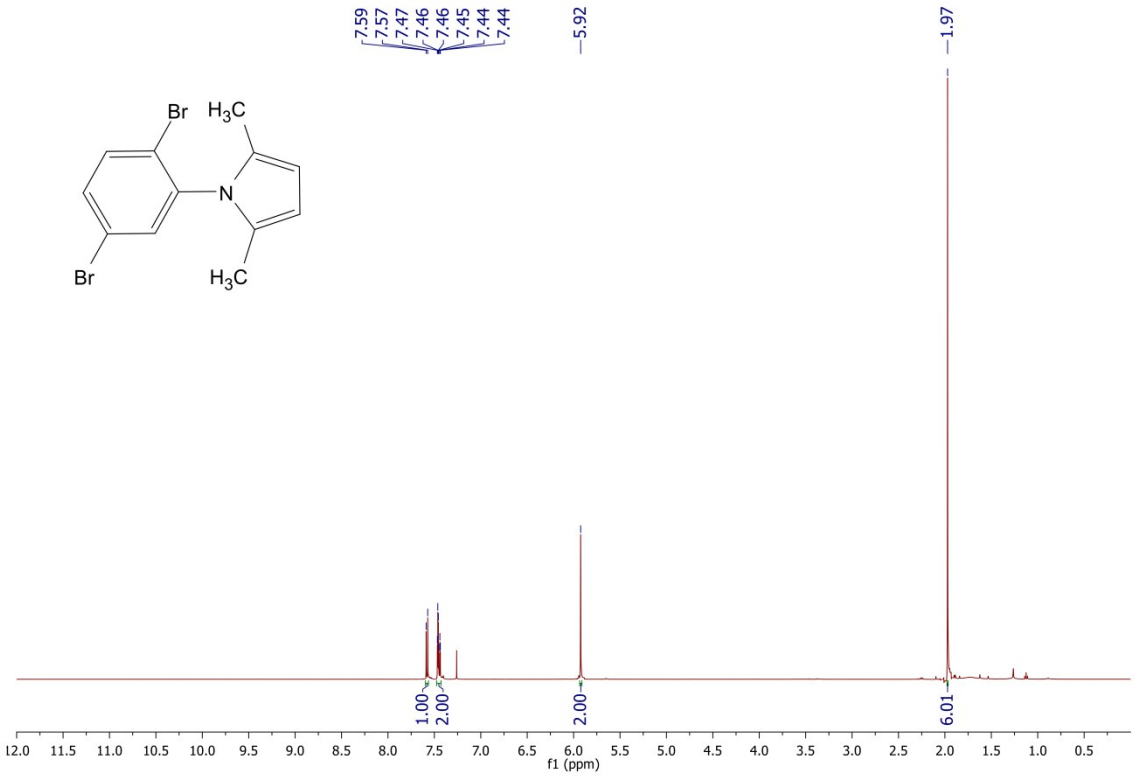


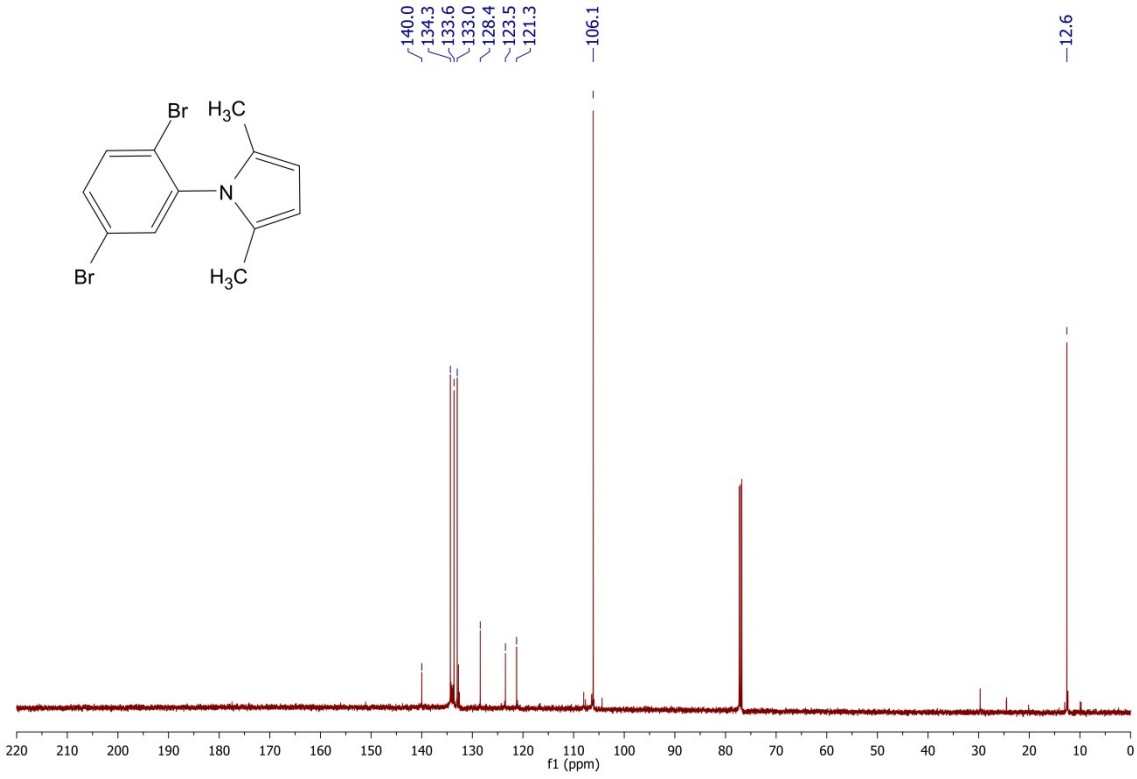


File :C:\GC-MS\2016\08.03.2016\34-DICHLOROANILIN-ACETONY-DES-SA-80
... -2H.D
Operator : TRUONG HAI
Instrument : GCMSD
Acquired : 8 Aug 2016 16:54 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Sample Name: 34-DICHLOROANILIN-ACETONY-DES-SA-80-2H
Misc Info :

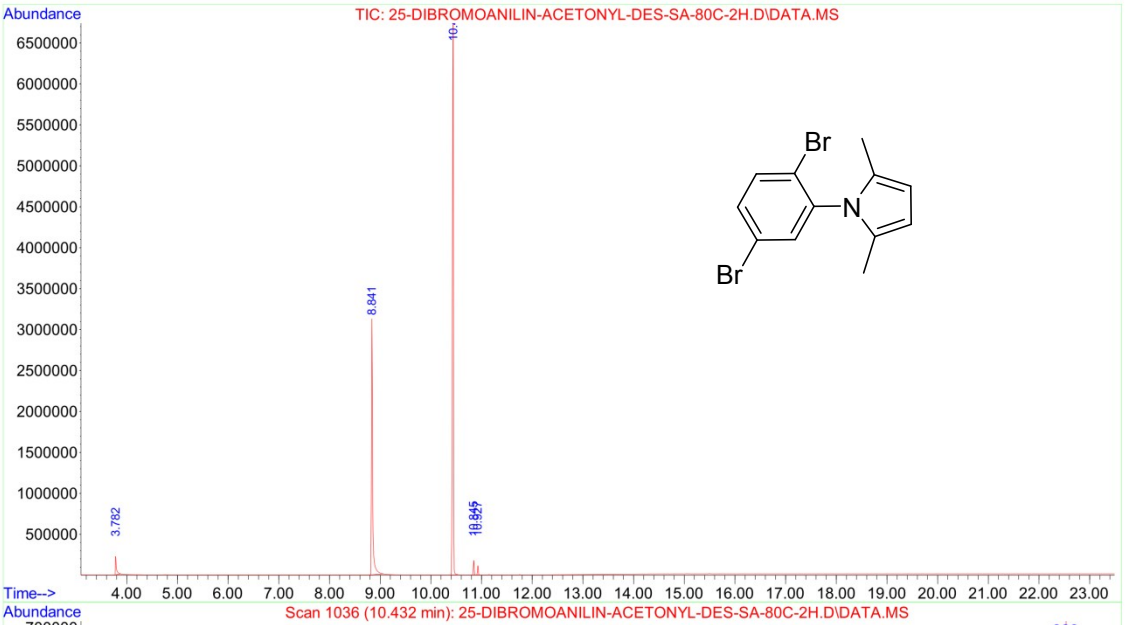


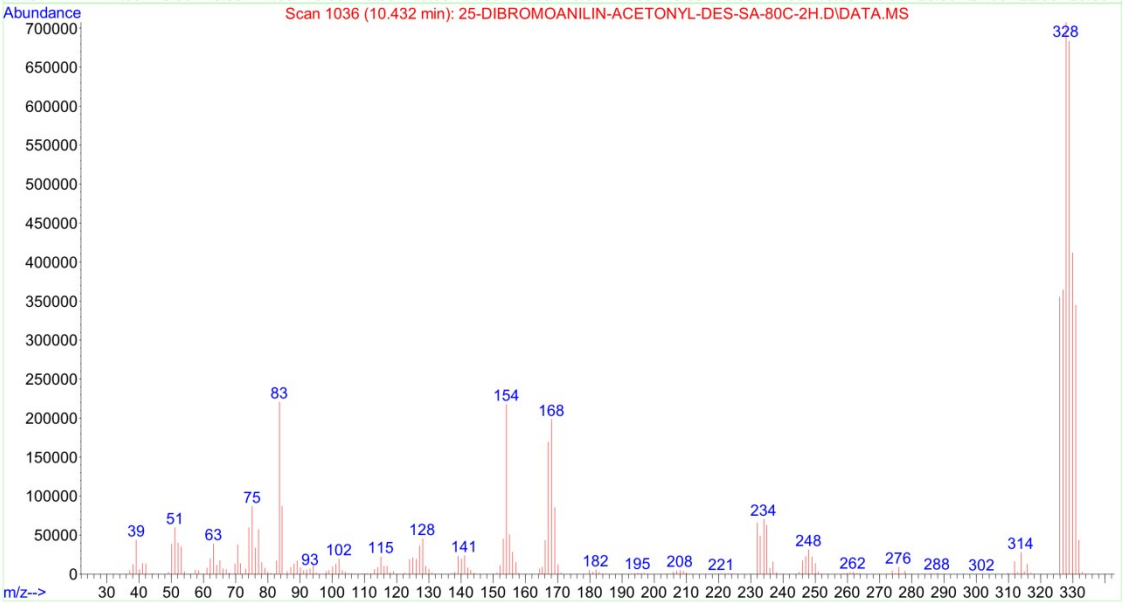
^{1}H NMR, ^{13}C NMR**, and GC-MS of 1-(2,5-Dibromophenyl)-2,5-dimethyl-1*H*-pyrrole**



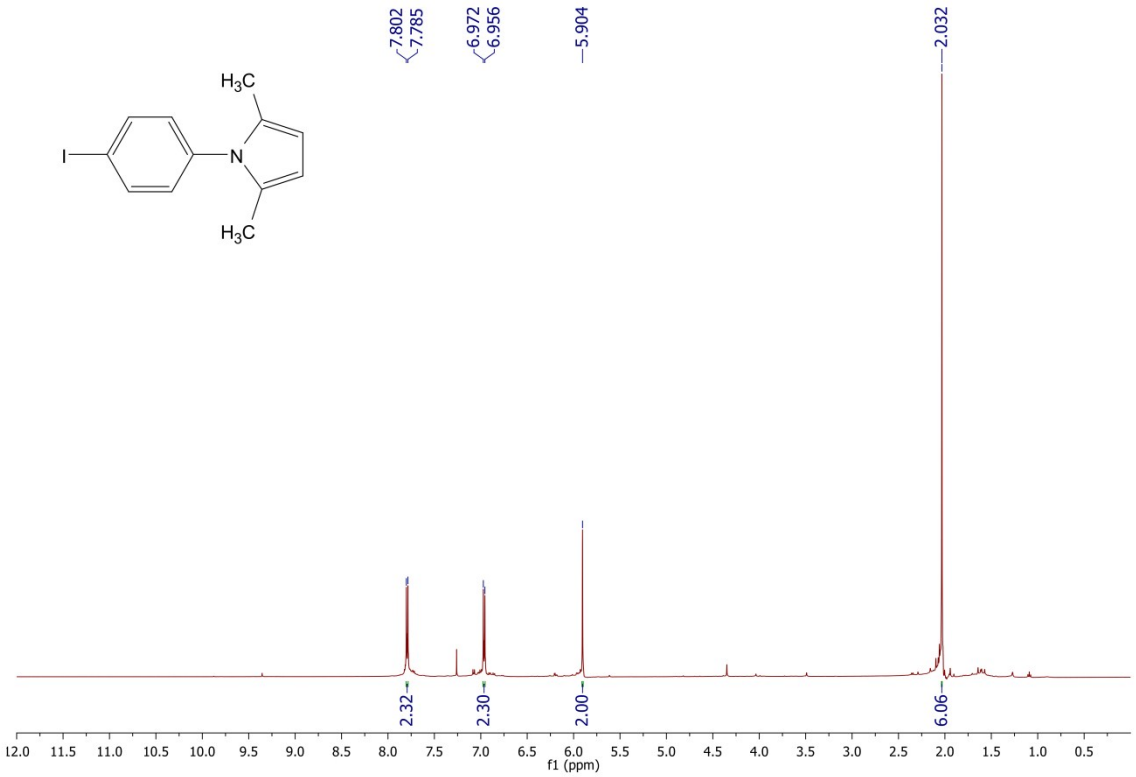


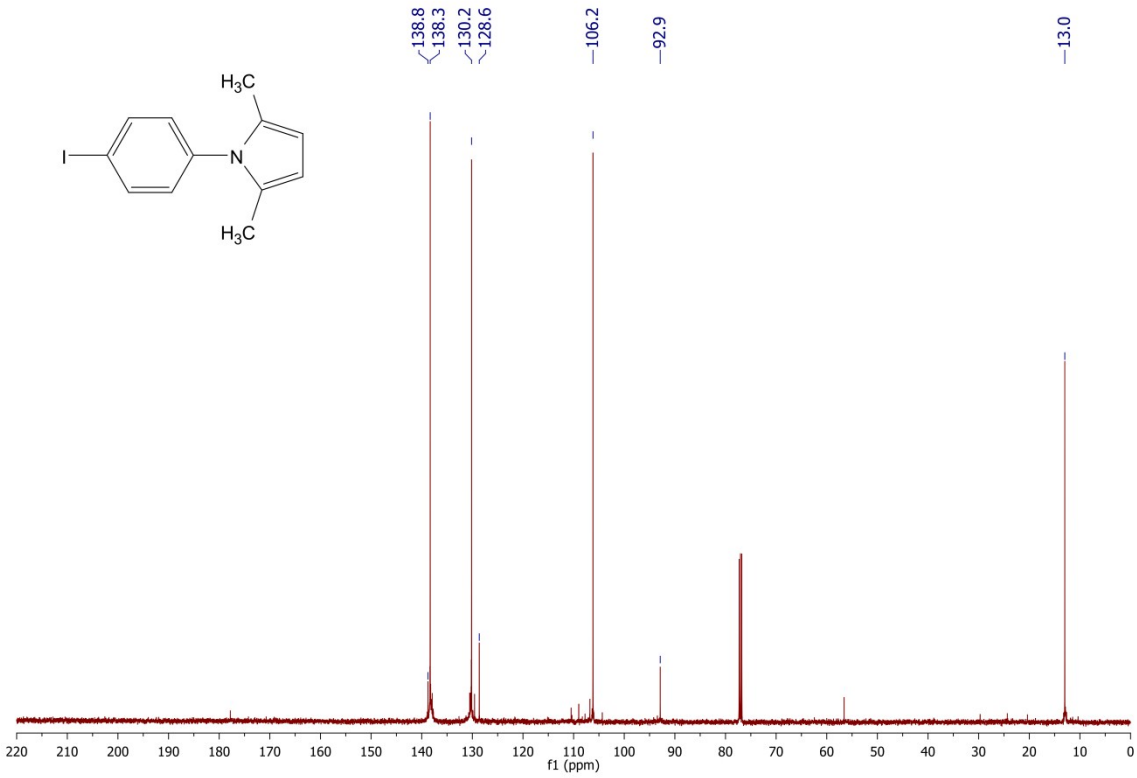
File :C:\GC-MS\2016\08.17.2016\25-DIBROMOANILIN-ACETONYL-DES-SA-80
... C-2H.D
Operator : TRUONG HAI
Instrument : GCMSD
Acquired : 17 Aug 2016 13:03 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Sample Name: 25-DIBROMOANILIN-ACETONYL-DES-SA-80C-2H
Misc Info :



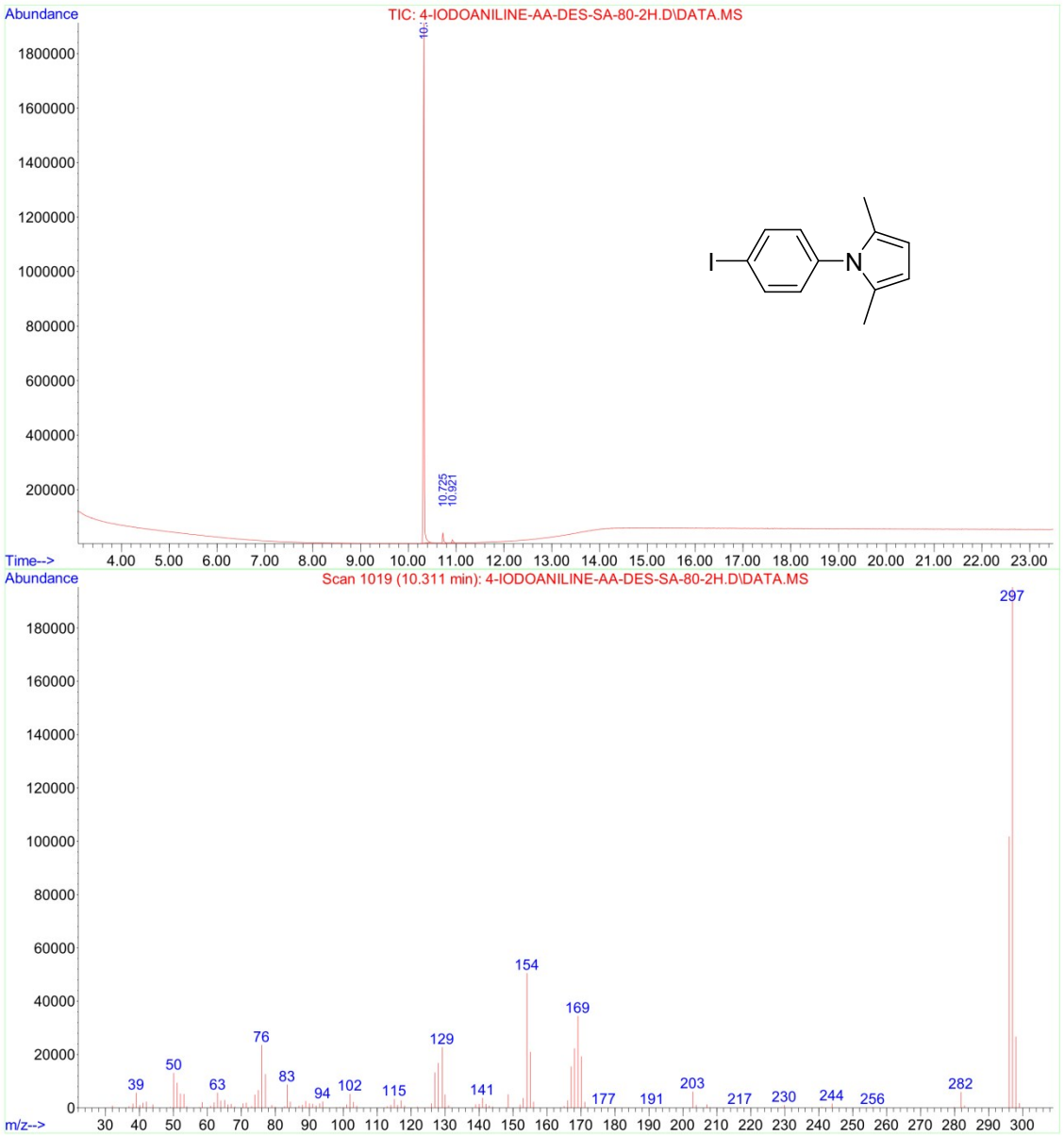


^{1}H NMR, ^{13}C NMR**, and GC-MS of 1-(4-Iodophenyl)-2,5-dimethyl-1*H*-pyrrole**

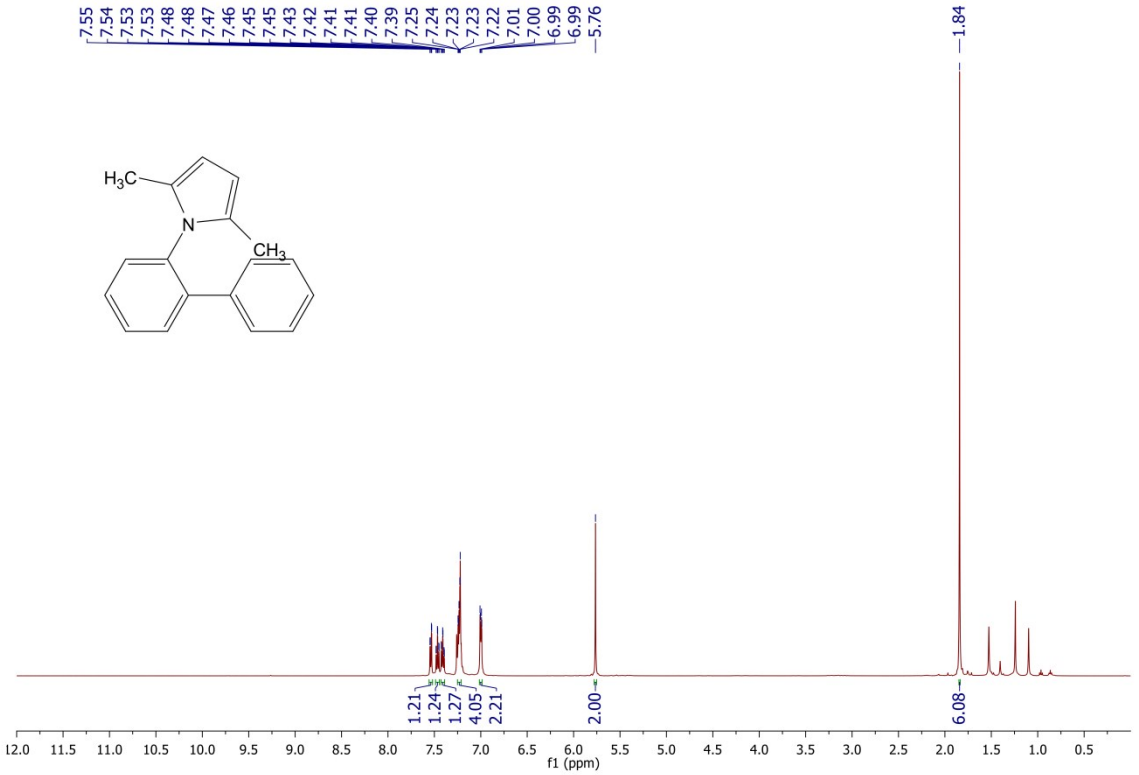


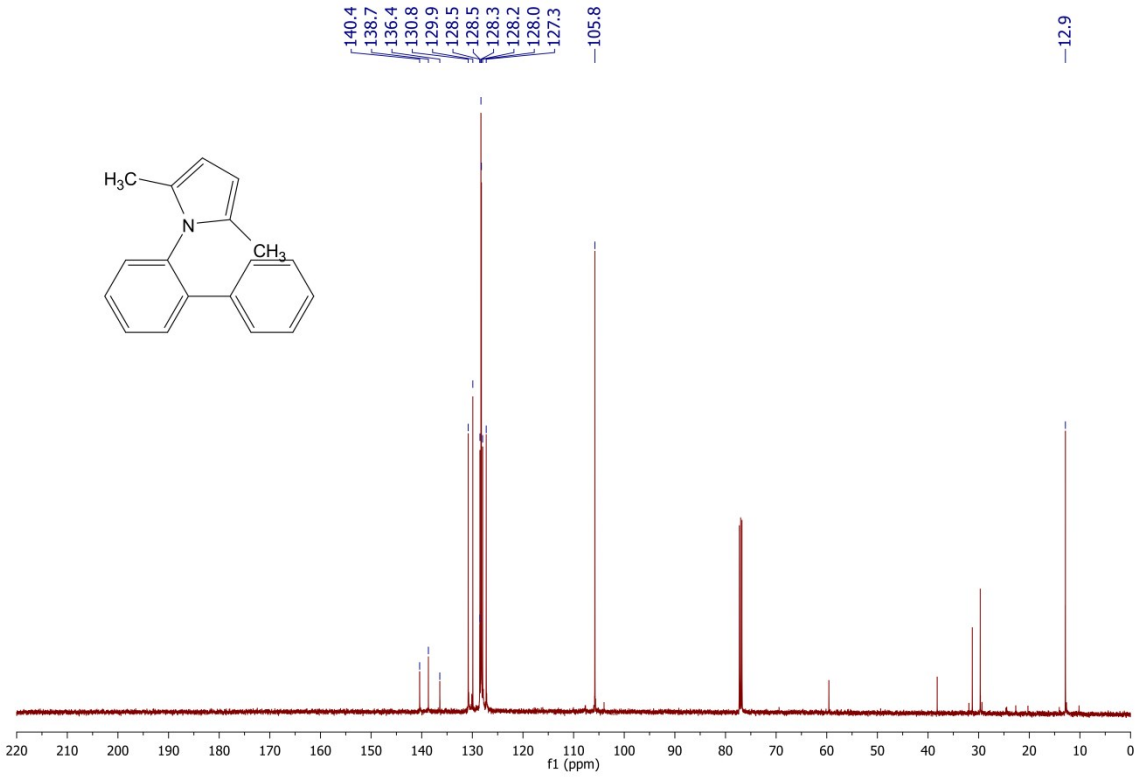


File :C:\GC-MS\2016\11.29.2016\4-IODOANILINE-AA-DES-SA-80-2H.D
Operator : THAO TRAN
Acquired : 29 Nov 2016 14:10 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Instrument : GCMSD
Sample Name: 4-IODOANILINE-AA-DES-SA-80-2H
Misc Info :
Vial Number: 2

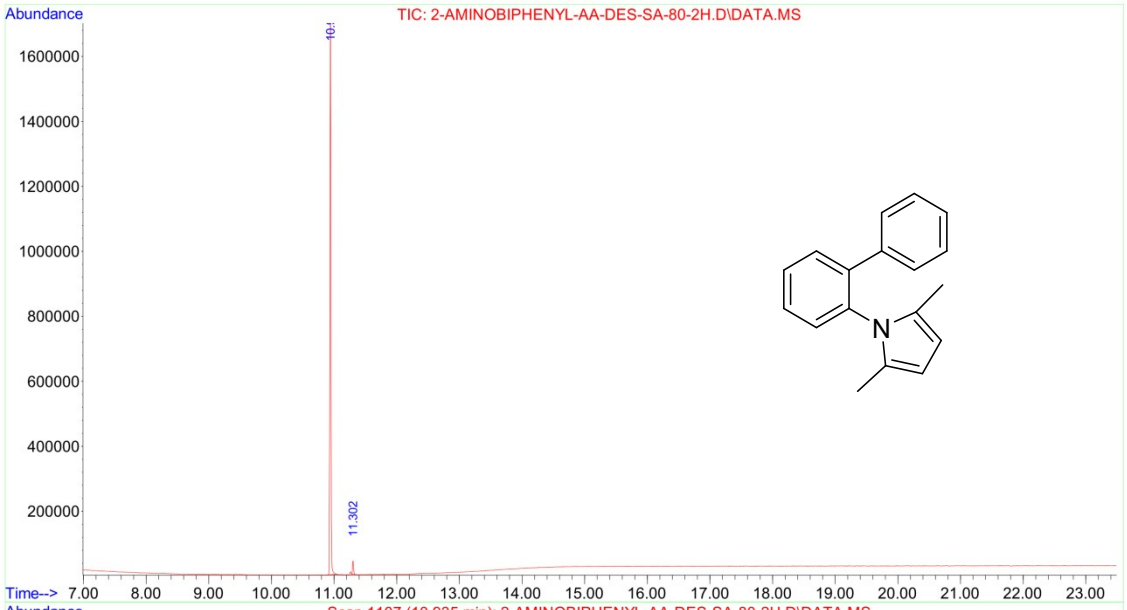


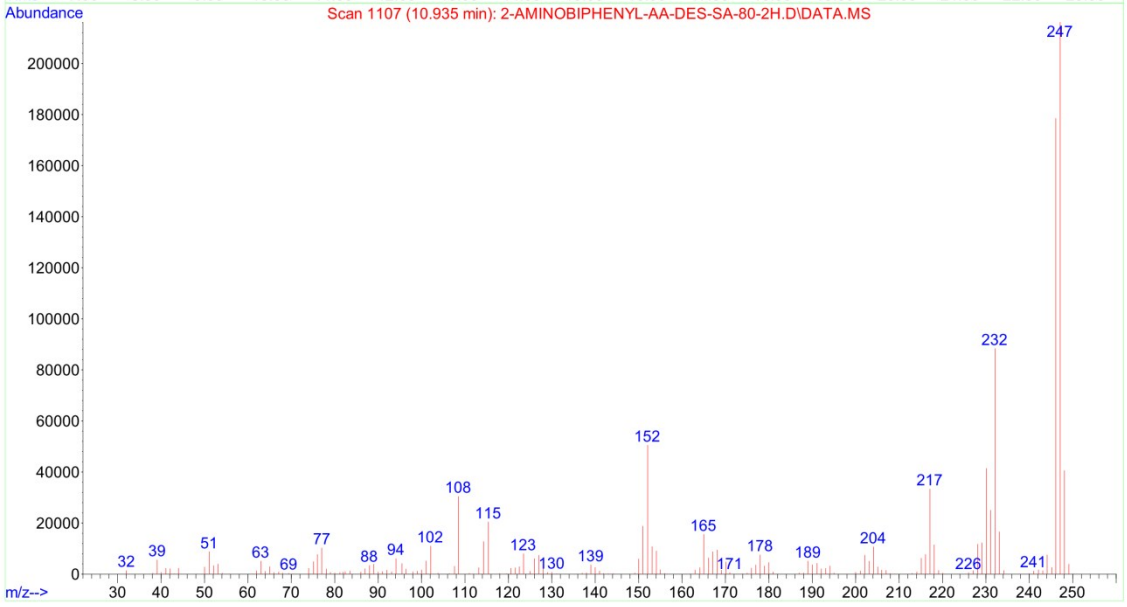
^{1}H NMR, ^{13}C NMR**, and GC-MS of 1-([1,1'-Biphenyl]-2-yl)-2,5-dimethyl-1*H*-pyrrole**



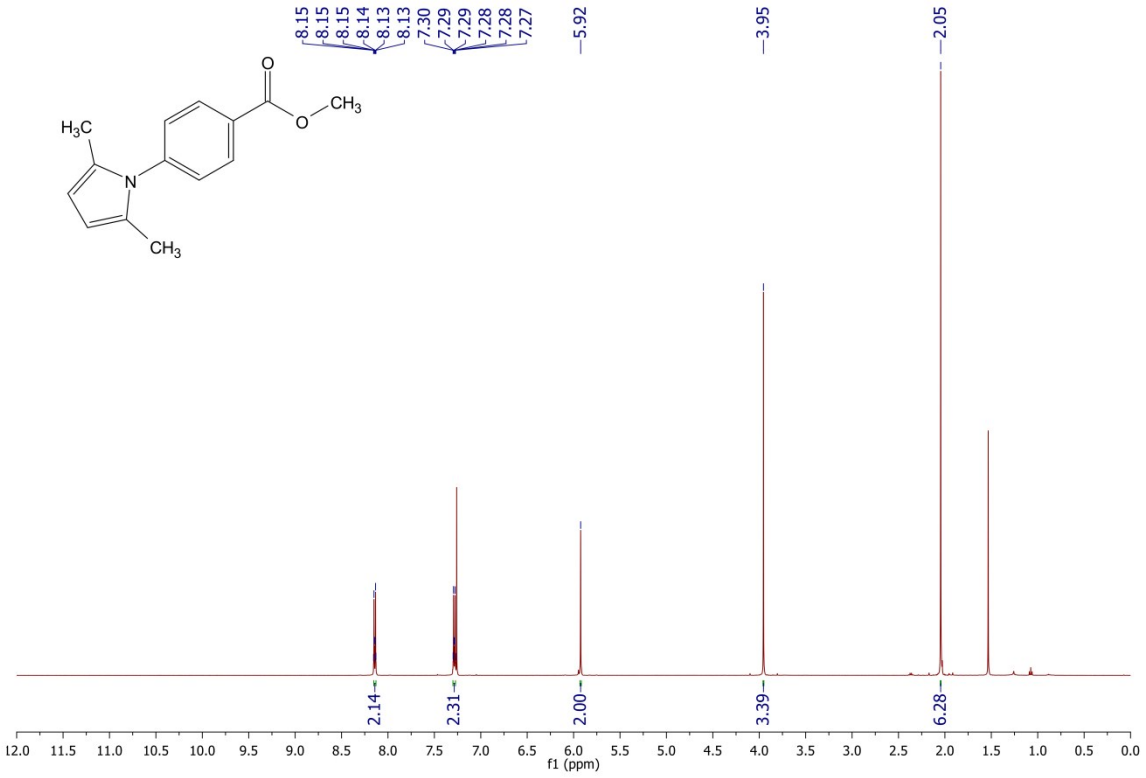


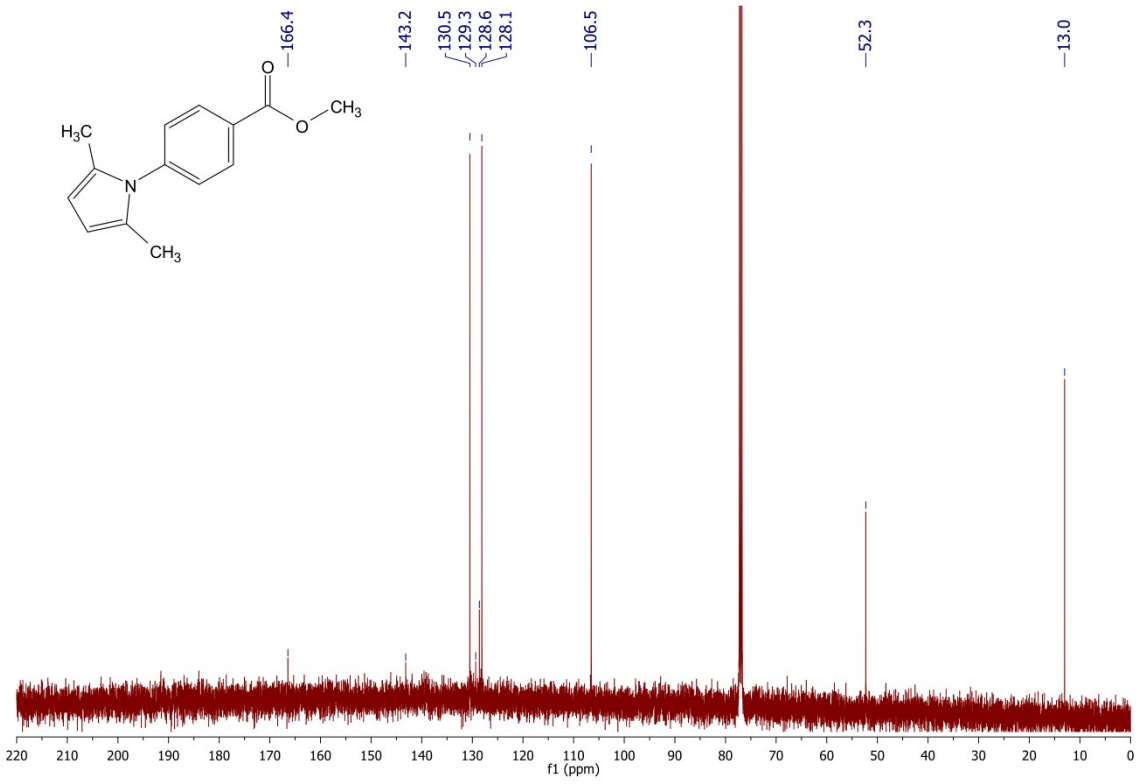
File :C:\GC-MS\2016\11.03.2016\2-AMINOBIPHENYL-AA-DES-SA-80-2H.D
Operator : TRUONG HAI
Acquired : 3 Nov 2016 14:21 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Instrument : GCMSD
Sample Name: 2-AMINOBIPHENYL-AA-DES-SA-80-2H
Misc Info :
Vial Number: 8



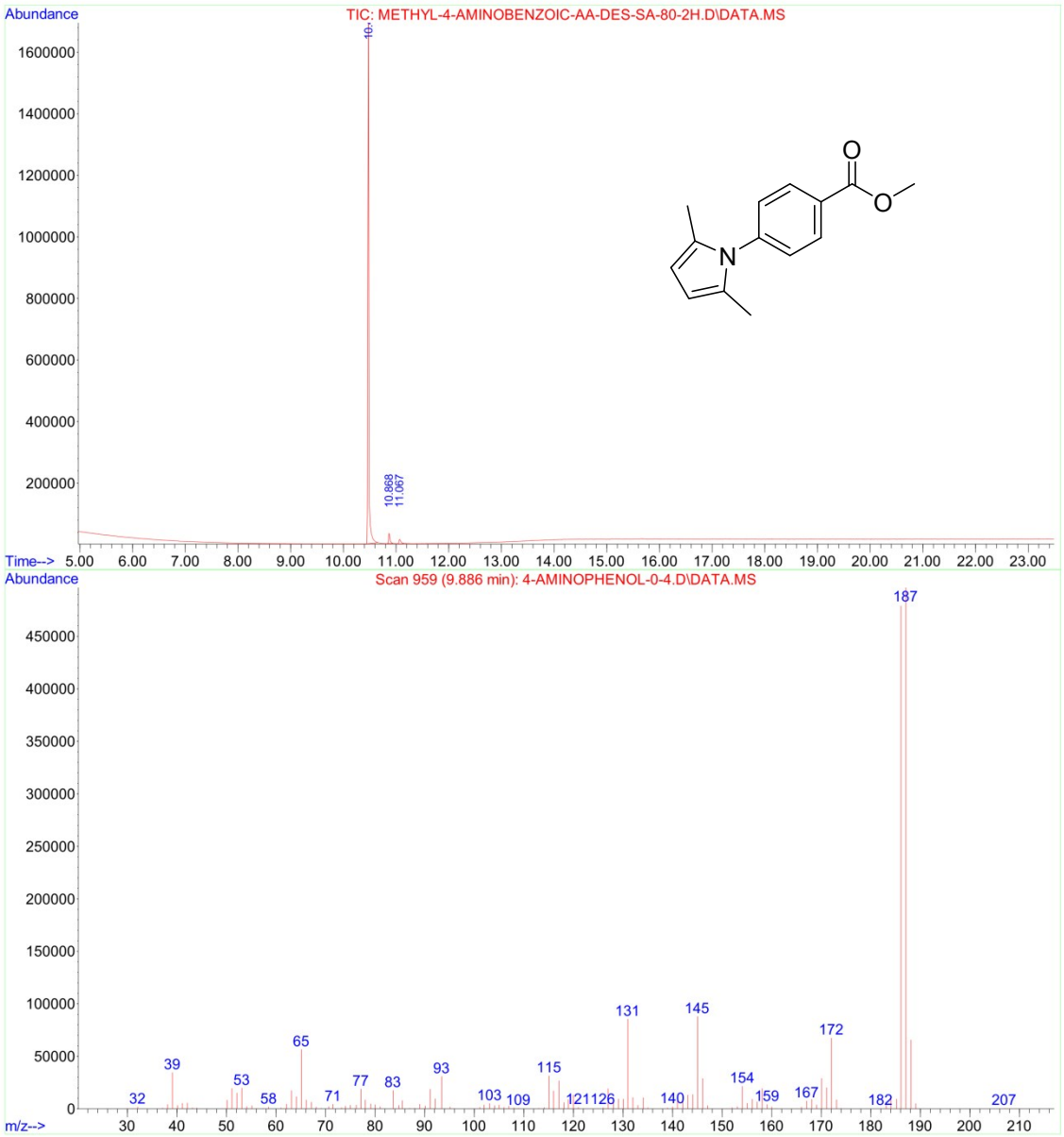


^{1}H NMR, ^{13}C NMR**, and GC-MS of methyl 4-(2,5-dimethyl-1*H*-pyrrol-1-yl)benzoate**

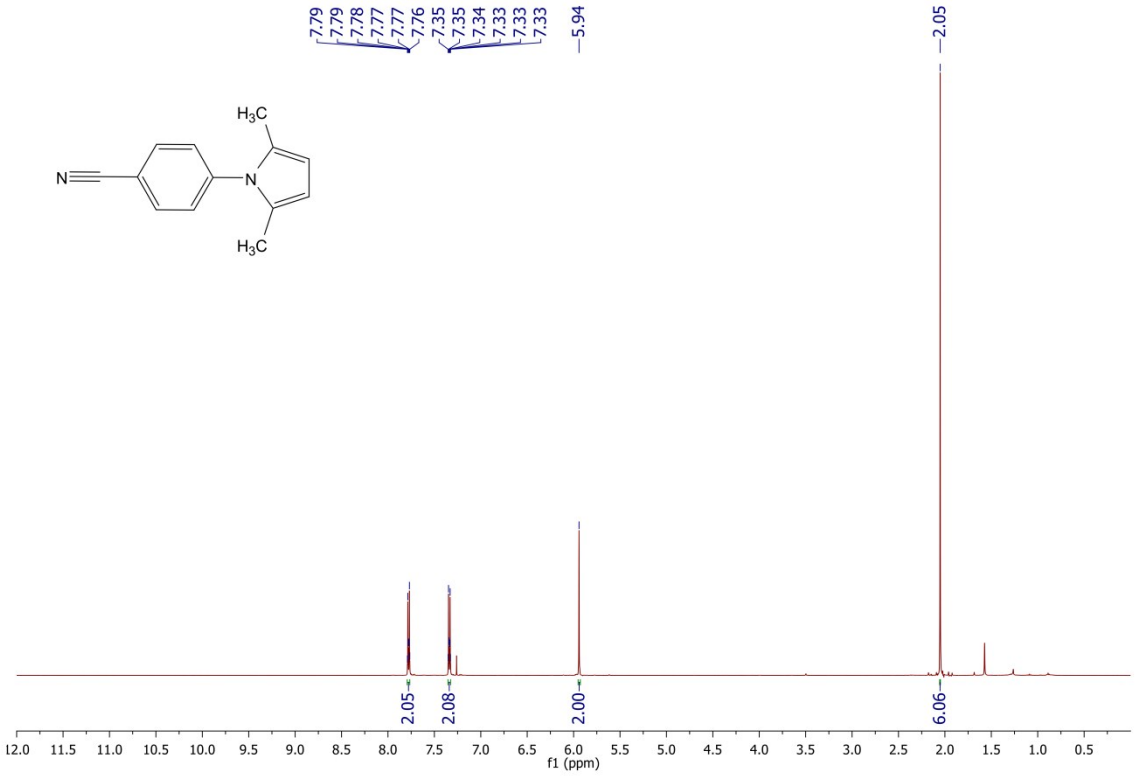


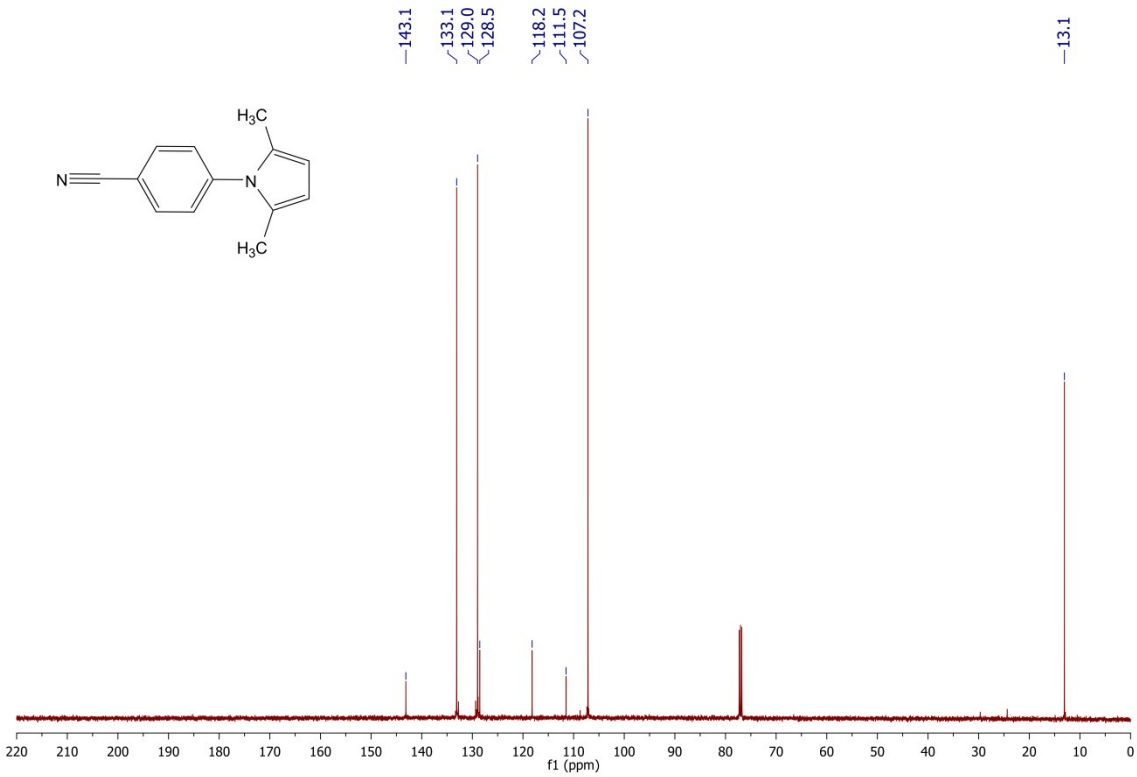


File :C:\GC-MS\2016\11.22.2016\METHYL-4-AMINOBENZOIC-AA-DES-SA-80-
... 2H.D
Operator : TRUONG HAI
Instrument : GCMSD
Acquired : 22 Nov 2016 15:13 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Sample Name: METHYL-4-AMINOBENZOIC-AA-DES-SA-80-2H
Misc Info :

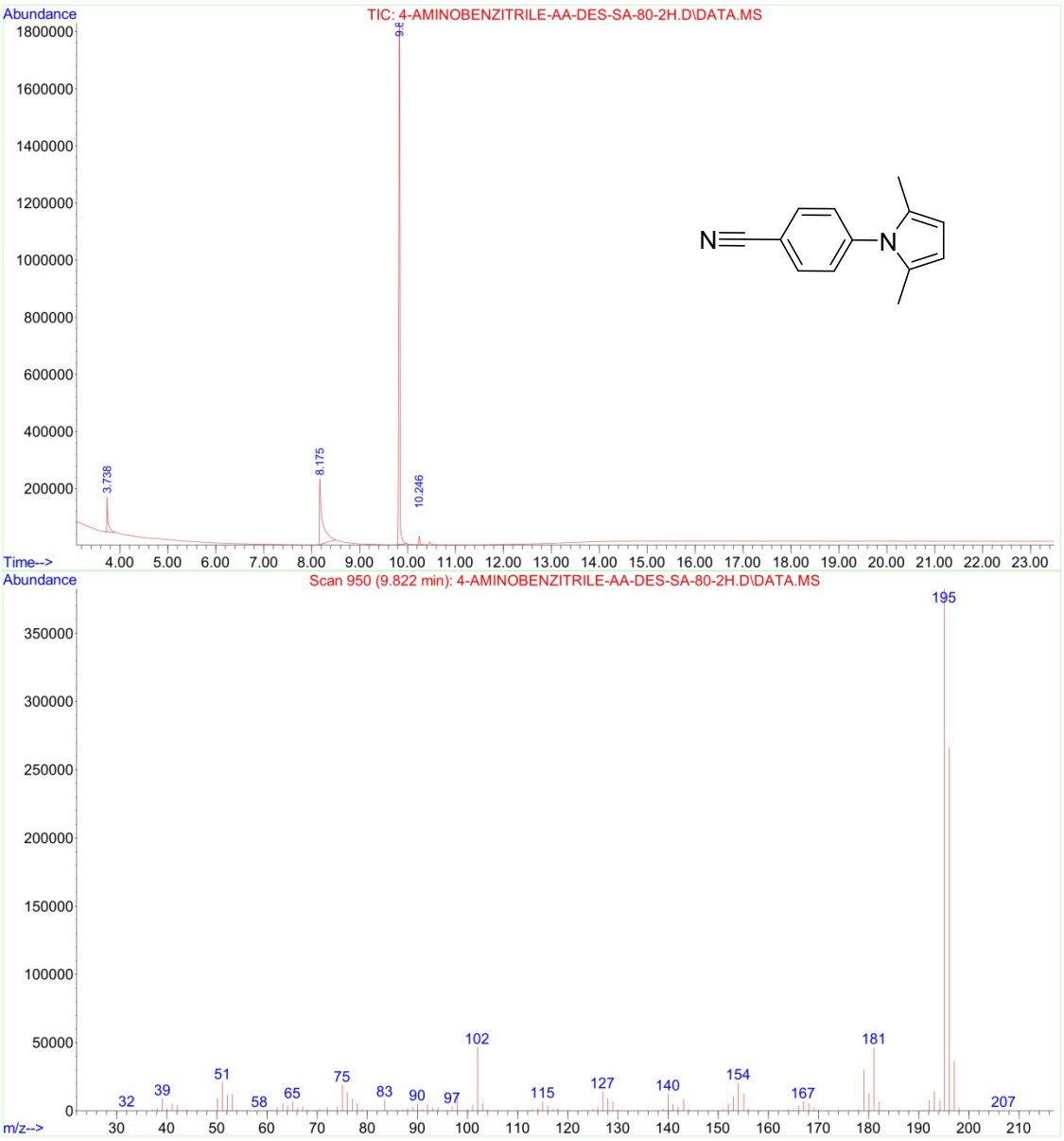


^{1}H NMR, ^{13}C NMR**, and GC-MS of 1-(4-cyanophenyl)-2,5-Dimethyl-1*H*-pyrrole**

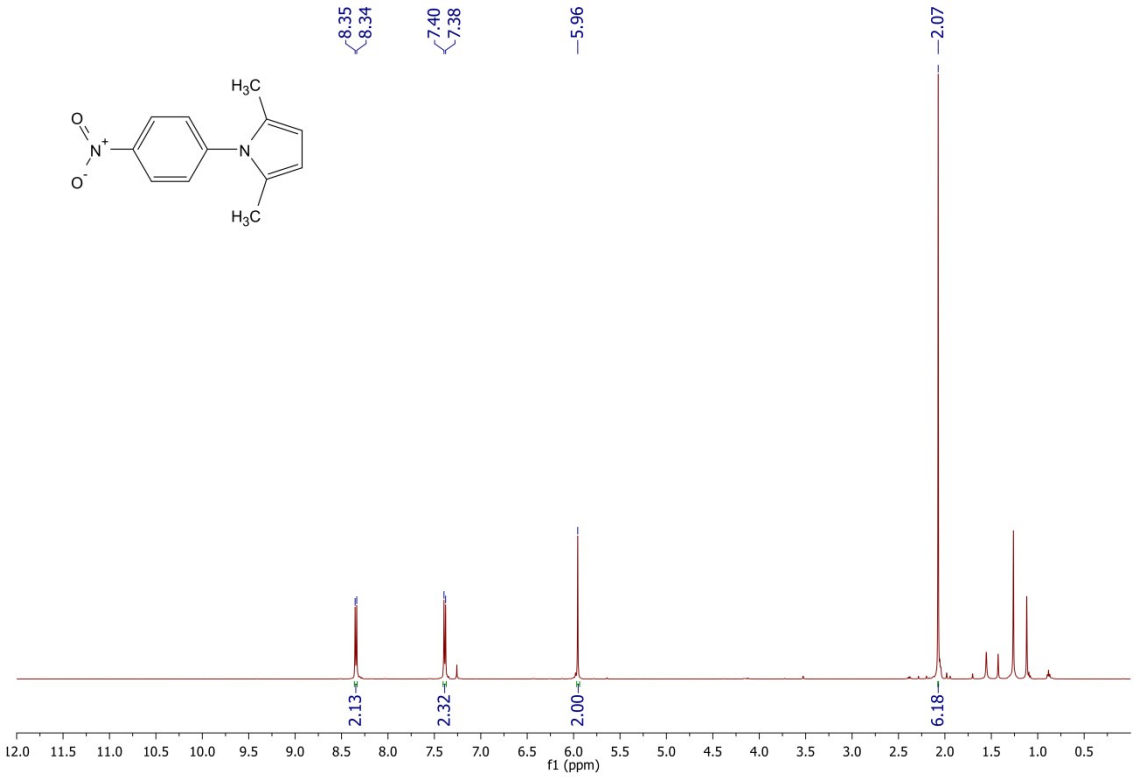


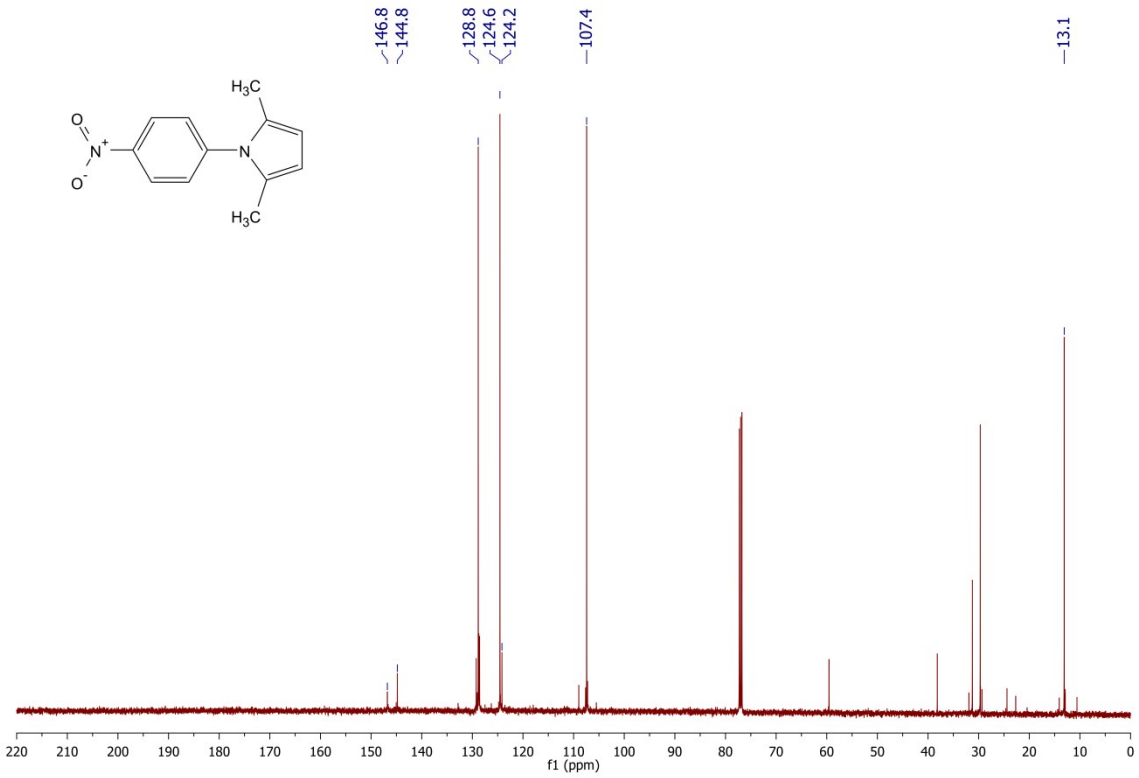


File :C:\GC-MS\2016\11.23.2016\4-AMINOBENZITRILE-AA-DES-SA-80-2H.D
Operator : TRUONG HAI
Acquired : 24 Nov 2016 8:50 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Instrument : GCMSD
Sample Name: 4-AMINOBENZITRILE-AA-DES-SA-80-2H
Misc Info :
Vial Number: 1

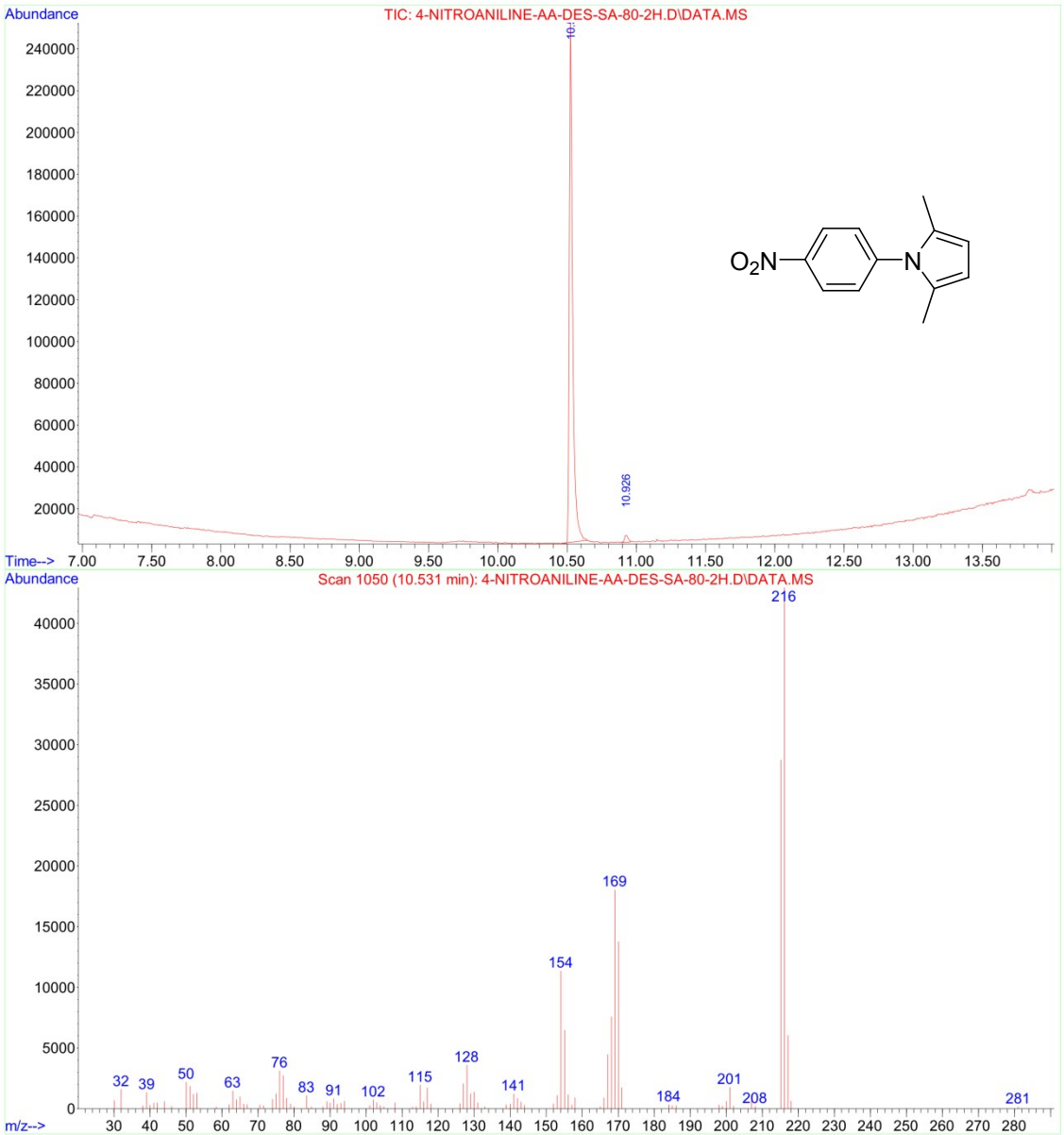


^{1}H NMR, ^{13}C NMR**, and GC-MS of 2,5-dimethyl-1-(4-nitrophenyl)-1*H*-pyrrole**

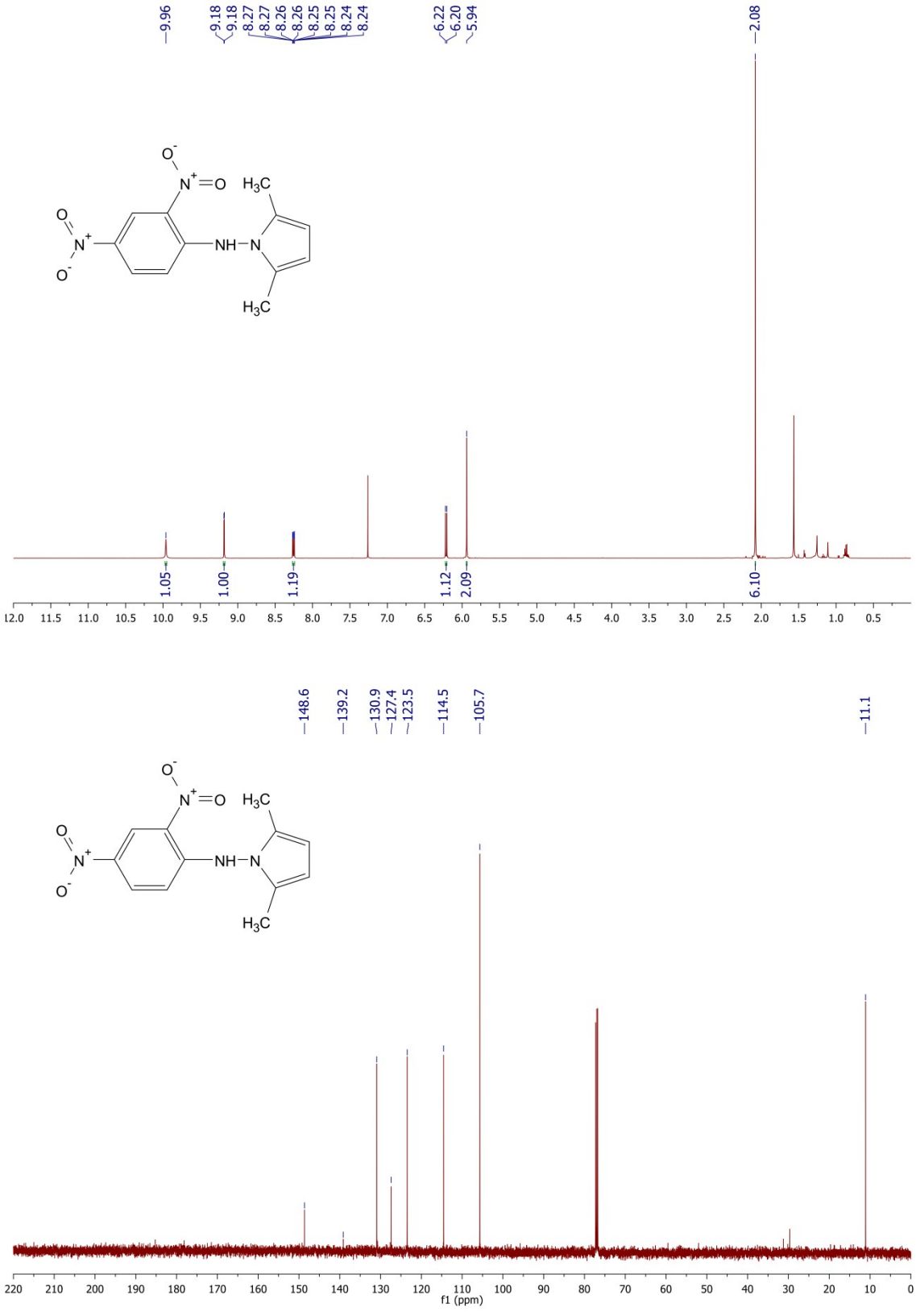




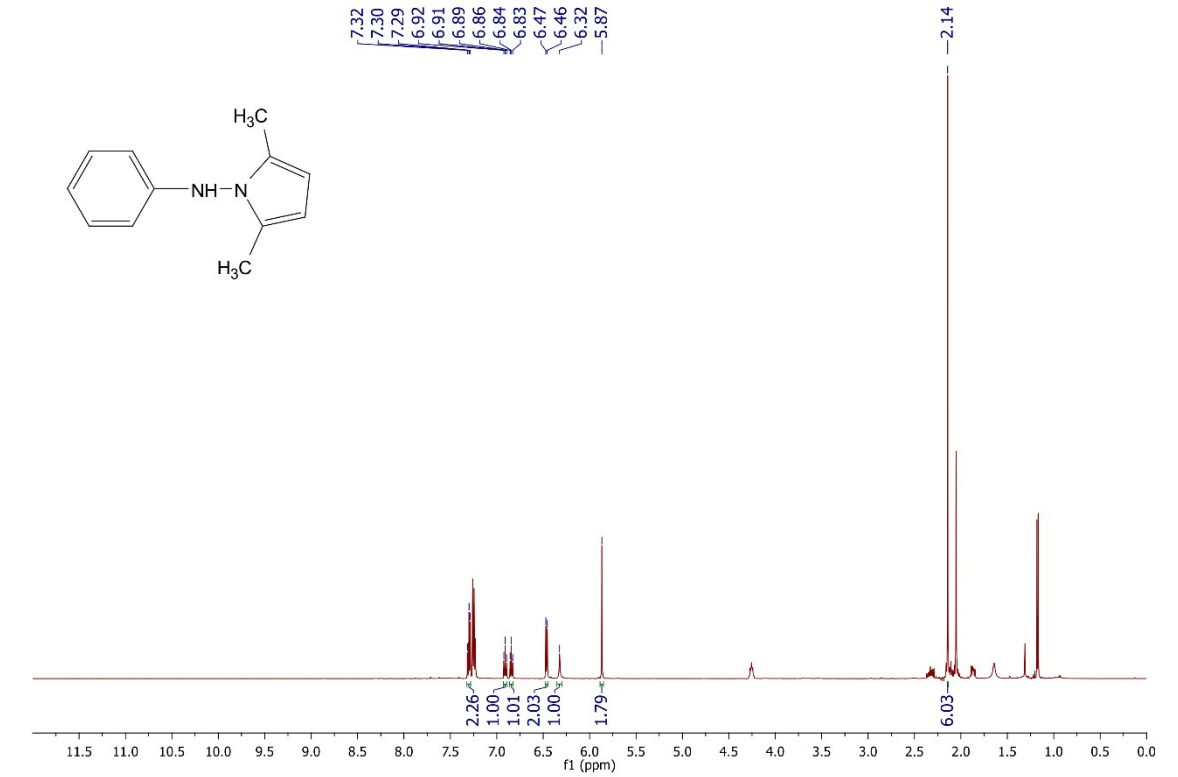
File :C:\GC-MS\2016\11.07.2016\4-NITROANILINE-AA-DES-SA-80-2H.D
Operator : Thao Tran
Acquired : 7 Nov 2016 18:27 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Instrument : GCMSD
Sample Name: 4-NITROANILINE-AA-DES-SA-80-2H
Misc Info :
Vial Number: 1

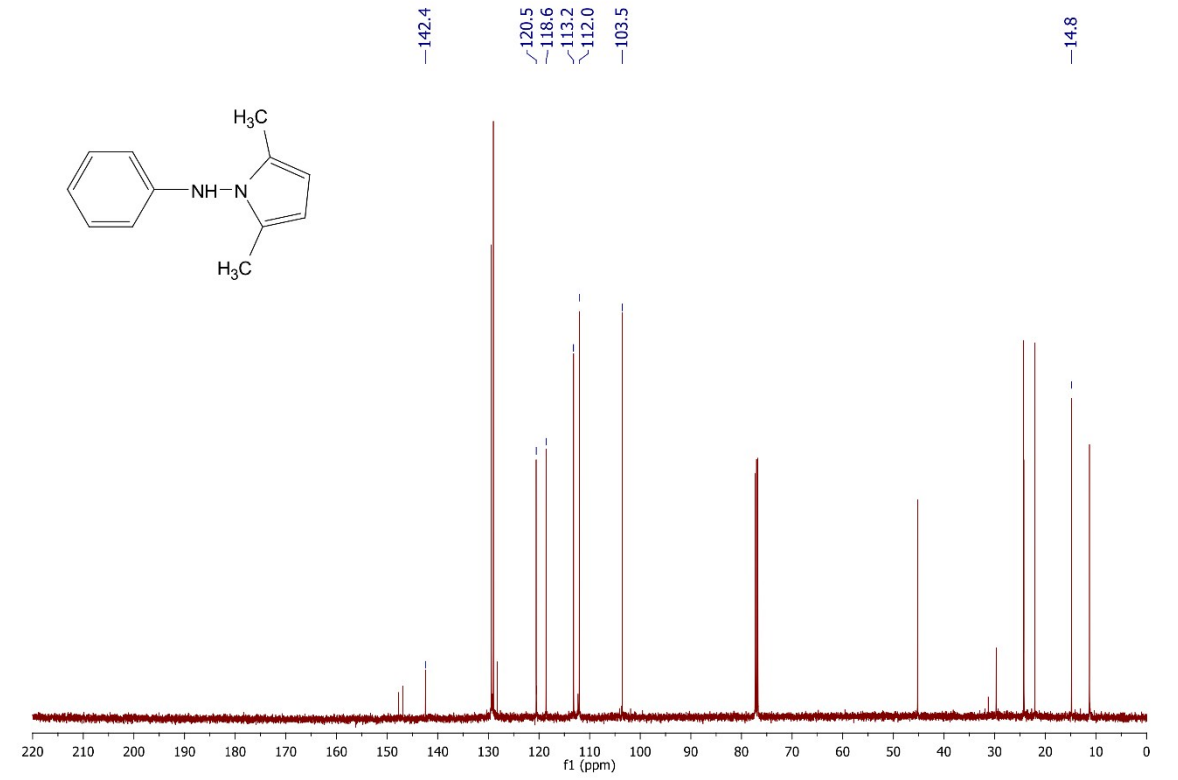


^{1}H NMR, ^{13}C NMR, and GC-MS of *N*-(2,4-dinitrophenyl)-2,5-dimethyl-1*H*-pyrrol-1-amine

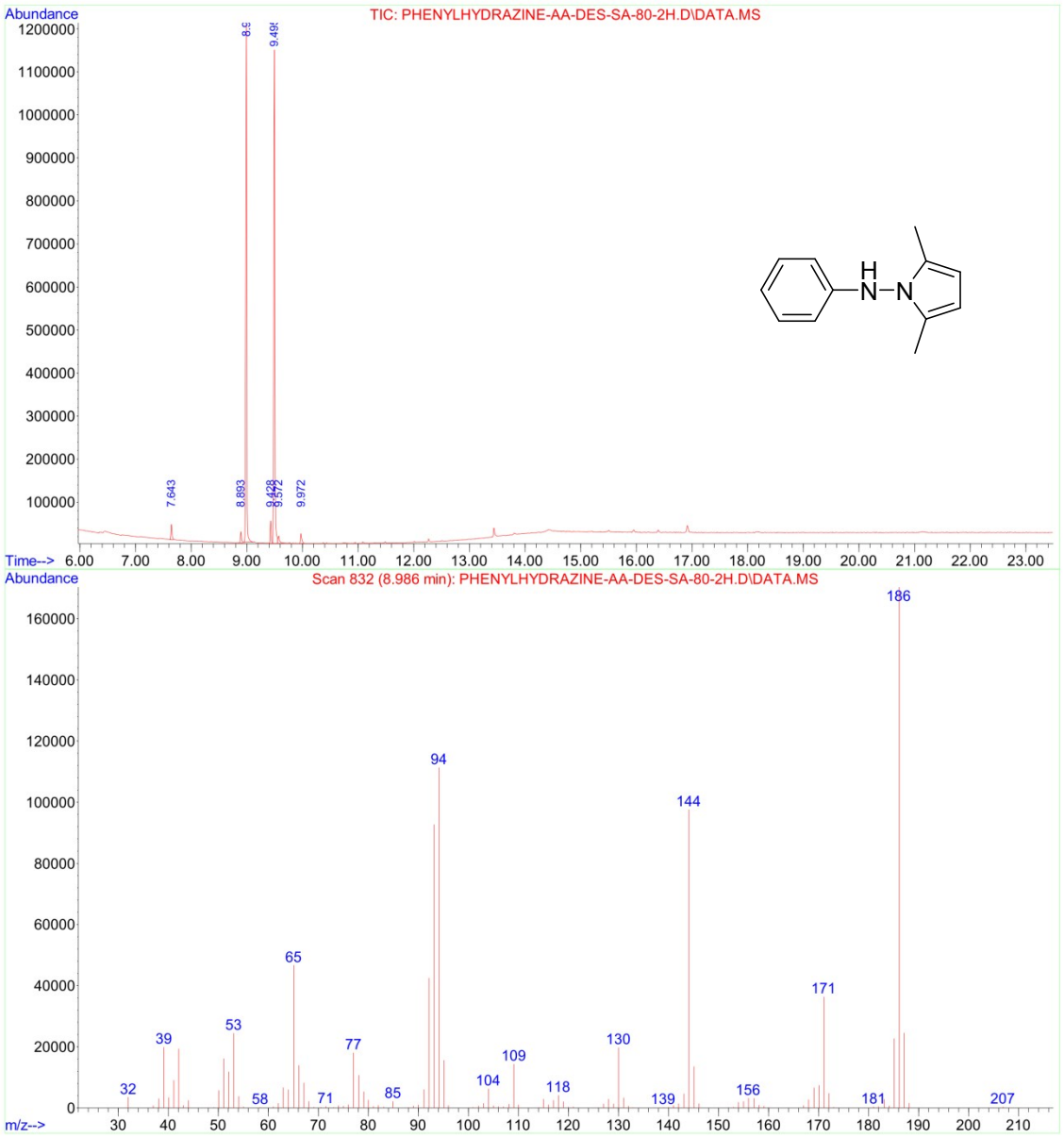


^{1}H NMR, ^{13}C NMR**, and GC-MS of 2,5-dimethyl-*N*-phenyl-1*H*-pyrrol-1-amine**

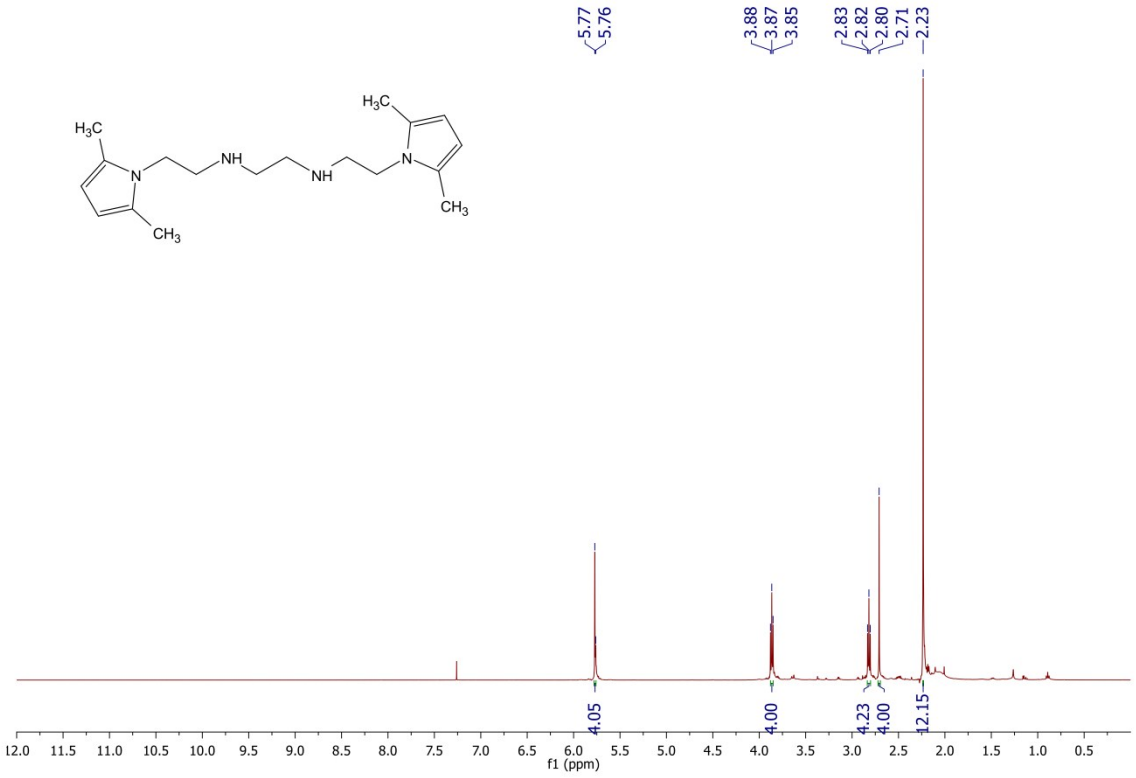


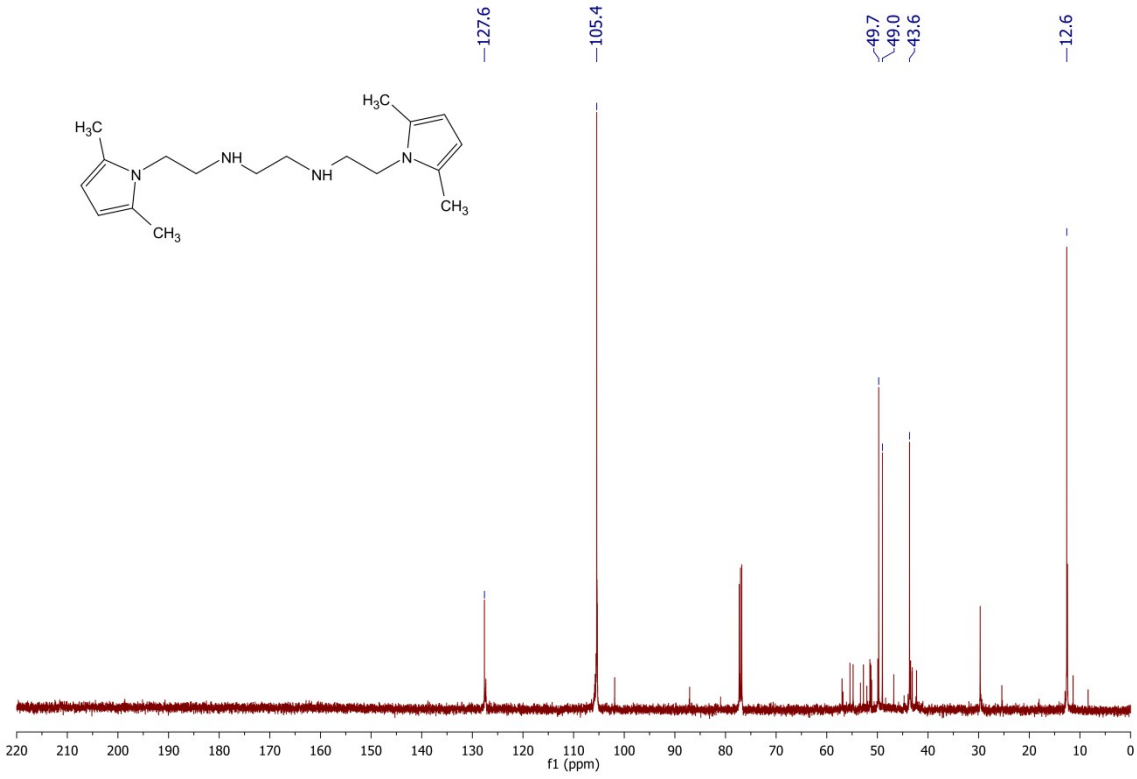


File :C:\GC-MS\2016\11.13.2016\PHENYLHYDRAZINE-AA-DES-SA-80-2H.D
Operator : TRUONG HAI
Acquired : 13 Nov 2016 12:51 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Instrument : GCMSD
Sample Name: PHENYLHYDRAZINE-AA-DES-SA-80-2H
Misc Info :
Vial Number: 3



^{1}H NMR, ^{13}C NMR**, and HRMS of N_1,N_2-bis(2-(2,5-Dimethyl-1*H*-pyrrol-1-yl)ethyl)ethane-1,2-diamine**





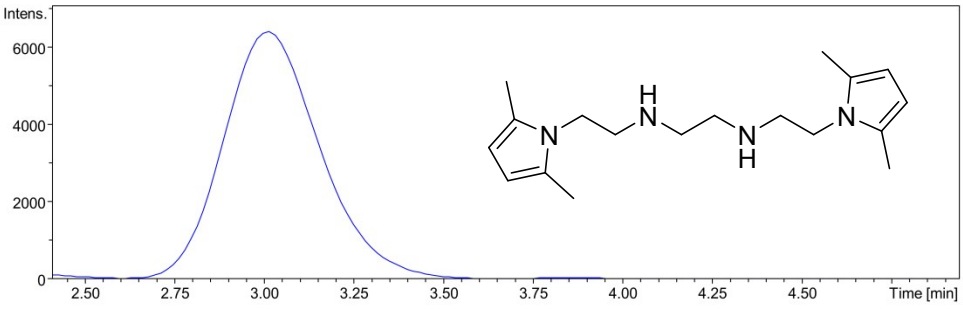
Display Report

Analysis Info

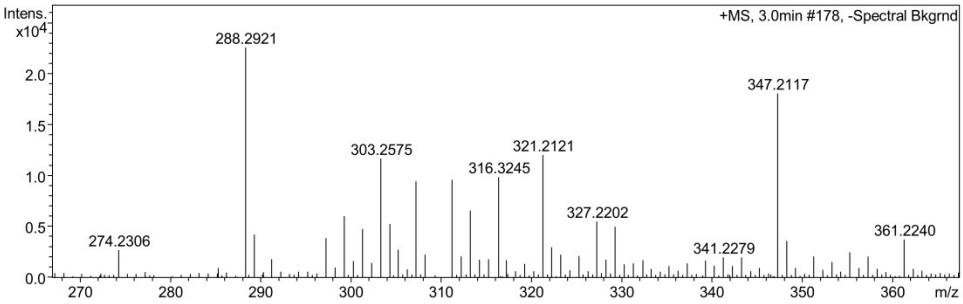
Analysis Name	D:\Data\2016\triet_1-a,8_01_2245.d	Acquisition Date	12/28/2016 11:47:51 PM
Method	dmm 2017.m	Operator	Anh Mai
Sample Name	triet	Instrument	micrOTOF-Q 10187
Comment			

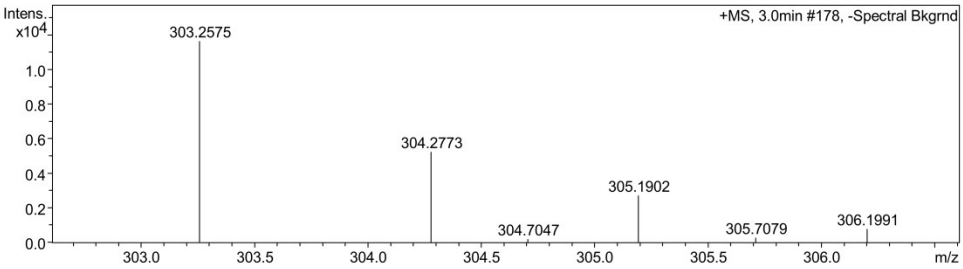
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4000 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	9.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	450.0 Vpp	Set Divert Valve	Source



EIC 303.0000 +All MS, -Spectral Bkgrnd, Smoothed (4.01,2,GA)





Section S6. References

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