Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supporting information

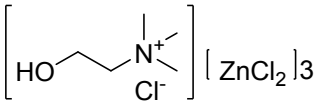
For

A green and efficient method for the synthesis of pyrroles using a deep eutectic solvent ([CholineCl][$ZnCl_2$]$_3$) under solvent-free sonication.

Table of contents

Section S1. Spectral data

[CholineCl][$ZnCl_2$]$_3$



IR (KBr, cm^{-1})3543, 3033, 2966, 1619, 1475, 1130, 954, 863 cm^{-1}.

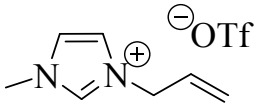
HRMS (ESI) *m/z* [Choline + H]$^+$ 105.0504

m/z [$ZnCl_3$]$^-$ 170.8356, [Zn_2Cl_5]$^-$ 306.6968, [Zn_3Cl_7]$^-$ 444.5604

^{1}H NMR (500 MHz, DMSO-*d6*) δ 5.22 (s, 1H), 3.81 (m, 2H), 3.38 – 3.36 (m, 2H), 3.09 (s, 9H).

^{13}C NMR (125 MHz, DMSO-*d6*) δ 67.8, 55.9, 54.2, 54.1, 54.0.

1-Allyl-3-methylimidazolium tetrafluoromethanesulfonate

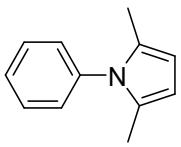


^{1}H NMR (500 Hz, $CDCl_3$) δ 9.62 (s, 1H), 7.42 (s, 1H), 7.33 (s, 1H), 6.01–5.95 (q, *J* = 10.5 Hz, 17 Hz, 1H), 5.46–5.42 (t, *J* = 8.5 Hz, 2H), 4.88–4.87 (d, *J* = 6.5 Hz, 2H), 4.00 (s, 3H).

^{13}C NMR (125 Hz, $CDCl_3$) δ 135.9, 127.8, 121.6, 121.0, 119.8, 114.5, 50.3, 34.9.

HR MS (ESI) *m/z* 123.0971 ([M]$^+$)

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



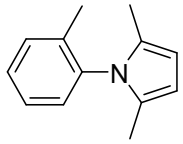
Yellow solid, mp 52-54 ˚C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49 – 7.46 (t, *J* = 7 Hz, 2H), 7.43 – 7.40 (t, *J* = 7.5 Hz, 1H), 7.24 – 7.23 (d, *J* = 7 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]



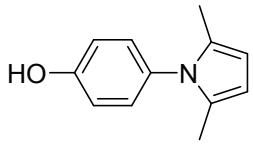
Yellow oil

^{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]



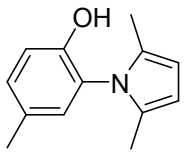
Yellow solid, mp 105-107 ˚C

^{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



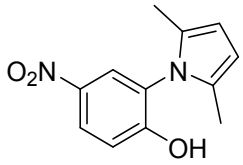
Black oil

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.14 – 7.12 (dd, J = 2 Hz, 2 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



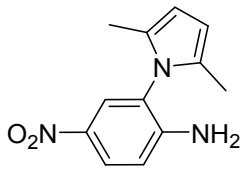
Orange solid, mp 167-170 ˚C

^{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 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



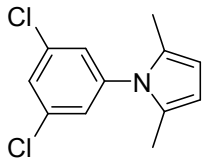
Yellow solid, m.p. = 128-130 ˚C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.65 – 7.63 (m, 2H), 7.21 – 7.19 (d, J = 9 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]



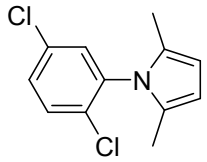
Orange solid, mp 79-81 ˚C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42 – 7.41 (t, J = 2 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]



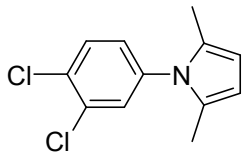
Black solid, mp 136-137 ˚C

^{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]



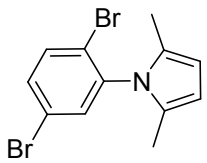
Yellow solid, mp 101-103 ˚C

^{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



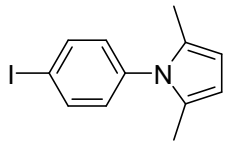
Yellow oil

^{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]



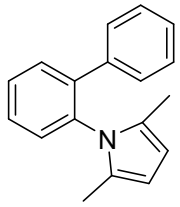
Yellow solid, mp 63-65 ˚C

^{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 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]



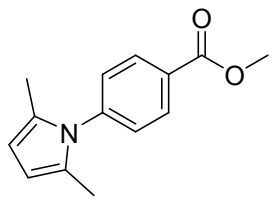
Yellow solid, mp 98-99 ˚C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.55 – 7.53 (dd, J = 1.5 Hz, 8 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 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]



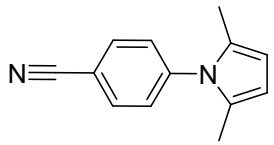
White solid, mp 100-102 ˚C

^{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]



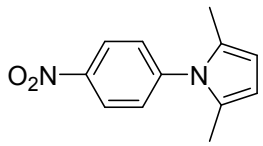
White solid, mp 93-94 ˚C

^{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]



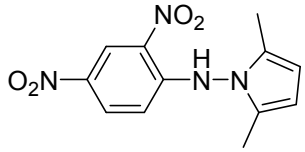
Yellow solid, mp 144-146 ˚C

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.35 – 8.34 (d, J = 9 Hz, 2H), 7.40 – 7.38 (d, J = 9 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]**

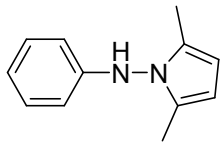


Yellow solid, mp 182-184 ˚C

^{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]



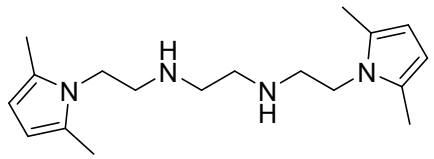
Yellow solid, mp 82-85 ˚C

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32 – 7.29 (t, J = 9 Hz, 2H), 6.92 – 6.90 (t, J = 7.5 Hz, 1H), 6.86 – 6.83 (t, J = 7 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

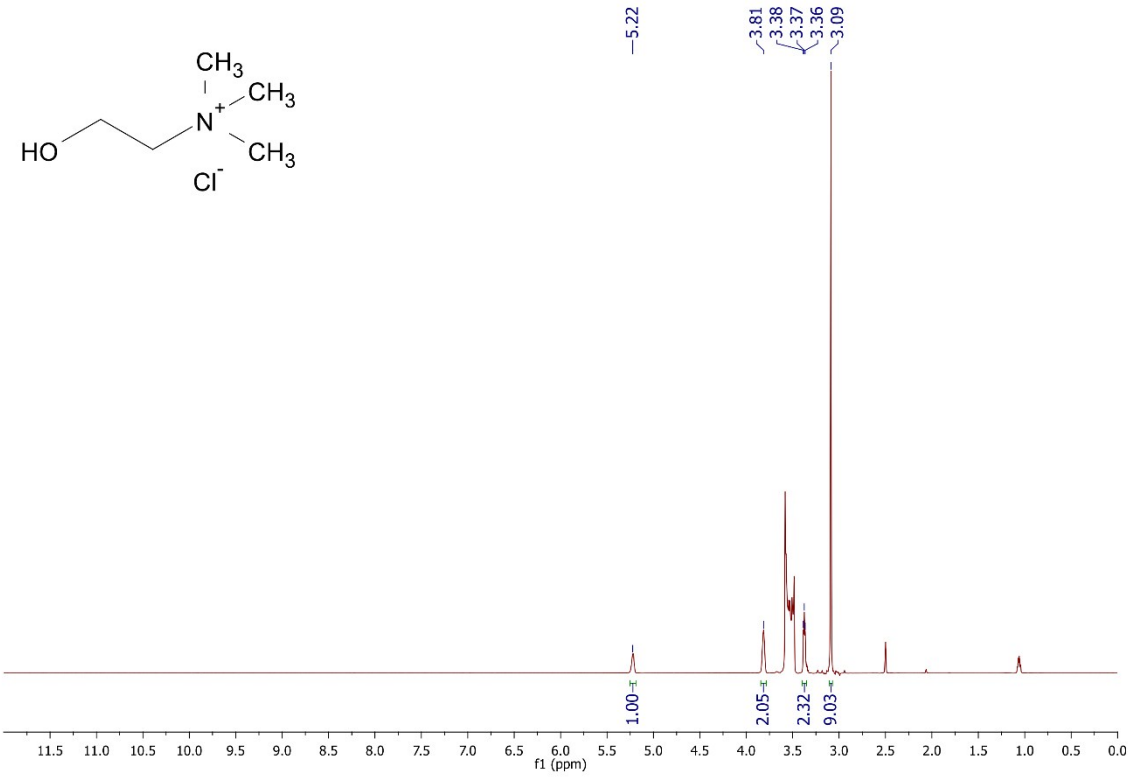
^{1}H NMR (500 MHz, $CDCl_3$) δ 5.77 – 5.76 (d, J = 5 Hz, 4H), 3.88 – 3.85 (t, J = 7 Hz, 4H), 2.83 – 2.81 (t, J = 7 Hz, 4H), 2.71 (s, 4H), 2.23 (s, 12H).

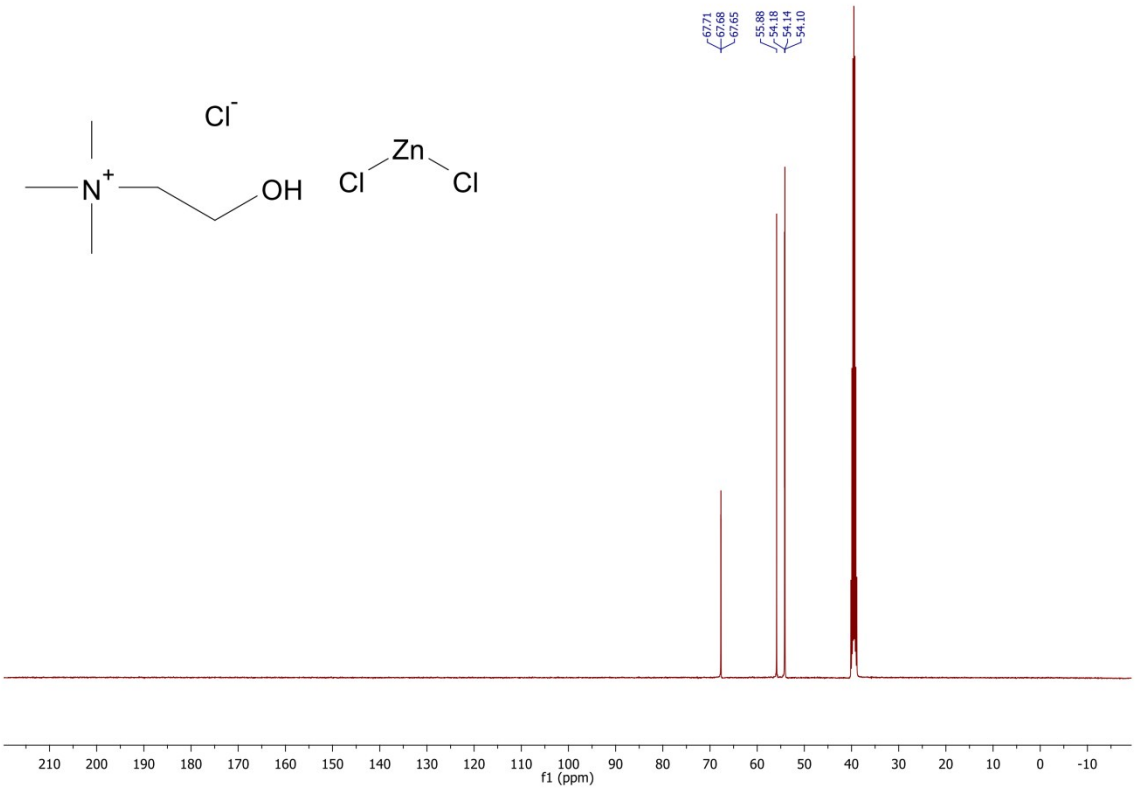
^{13}C NMR (125 MHz, $CDCl_3$) δ 127.6, 105.4, 49.7, 49.0, 43.7, 12.6.

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

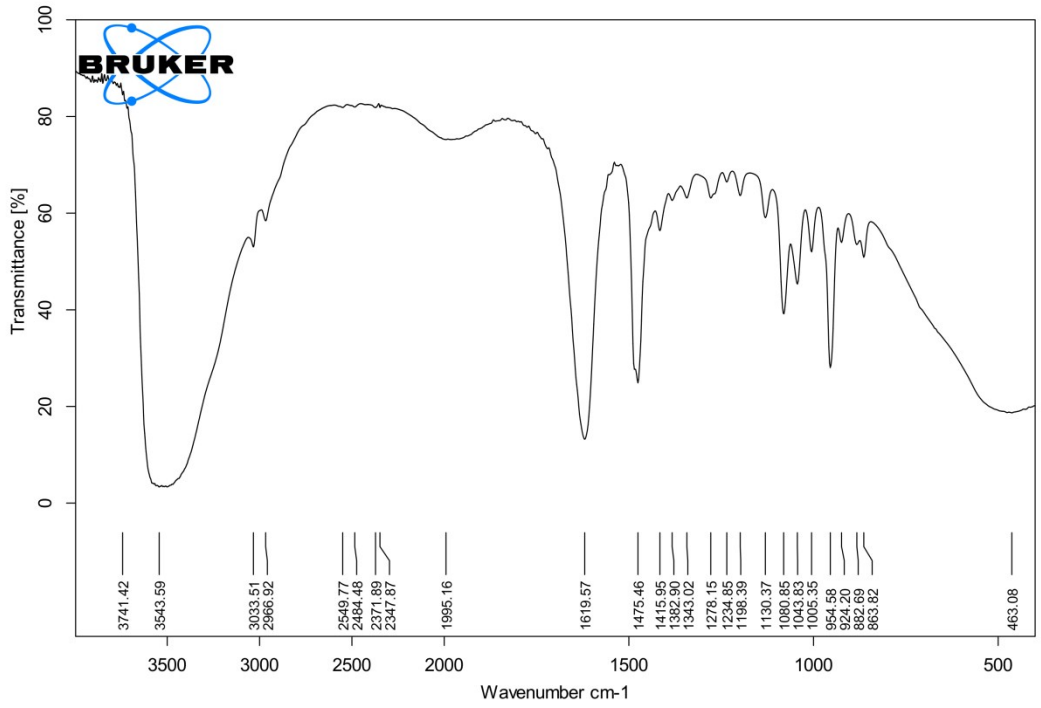
Section S2. ^{1}H, ^{13}C NMR and HRMS spectroscopy

^{1}H NMR, ^{13}C NMR, IR and HRMS of $[CholineCl][ZnCl_2]_3$



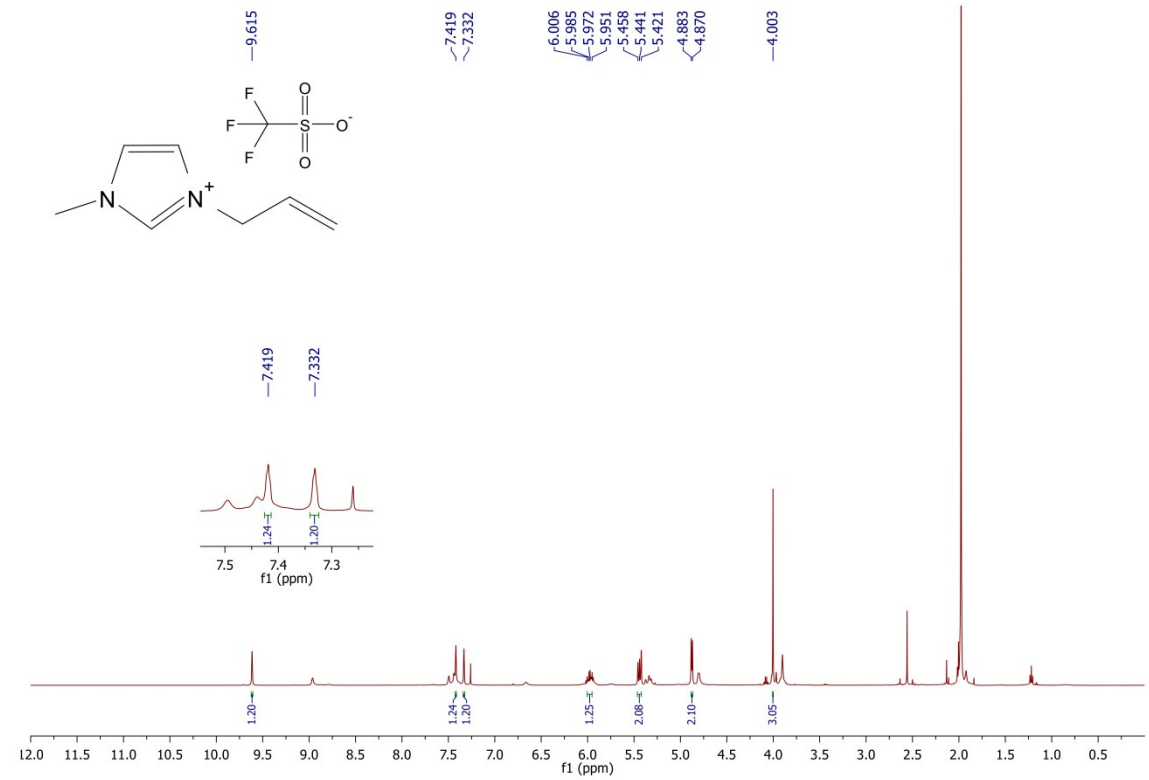


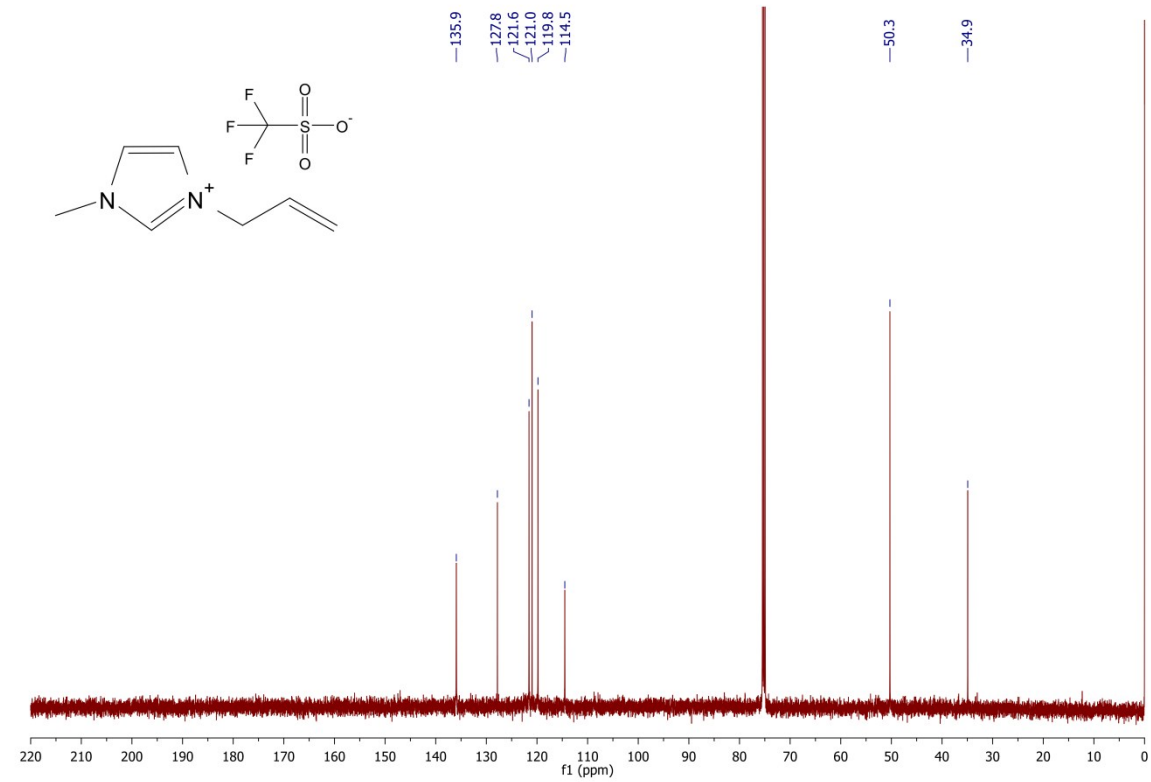
C:\OPUS_7.0.122\MEAS DATA\DICH VU\dh KHTN\10072015\DES V.1 7/10/2015 4:45:01 PM



Seite 1 von 1

^{1}H NMR, ^{13}C NMR, and HRMS of 3-allyl-1-methylimidazolium trifluoromethanesulfonate [AMIm](OTf)





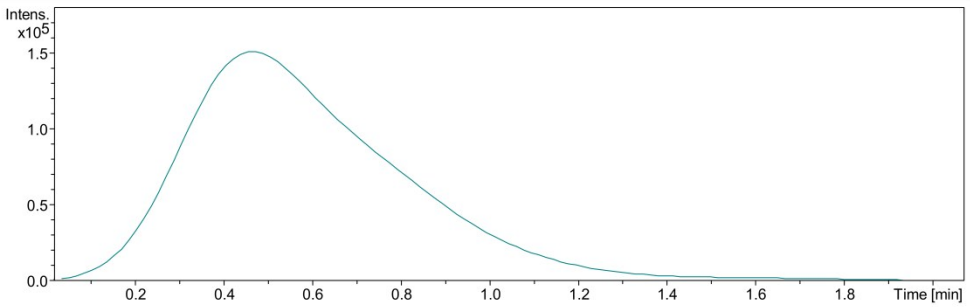
Display Report

Analysis Info

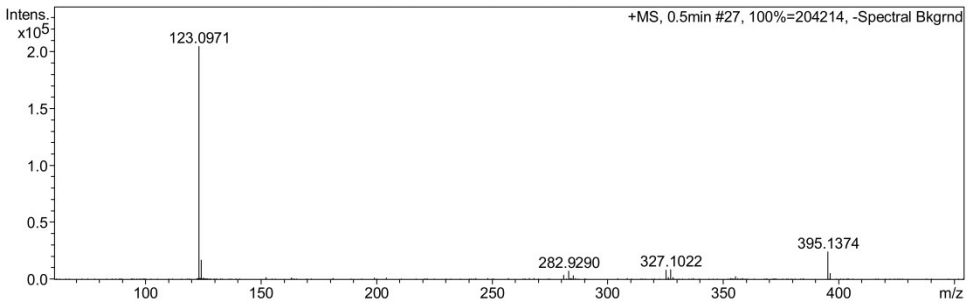
Analysis Name	D:\Data\dmm 2015\AMIM _1-F,2_01_16187.d	Acquisition Date	5/25/2015 9:39:53 PM
Method	dmm.m	Operator	Mai
Sample Name	AMIM	Instrument	micrOTOF-Q 10187
Comment			

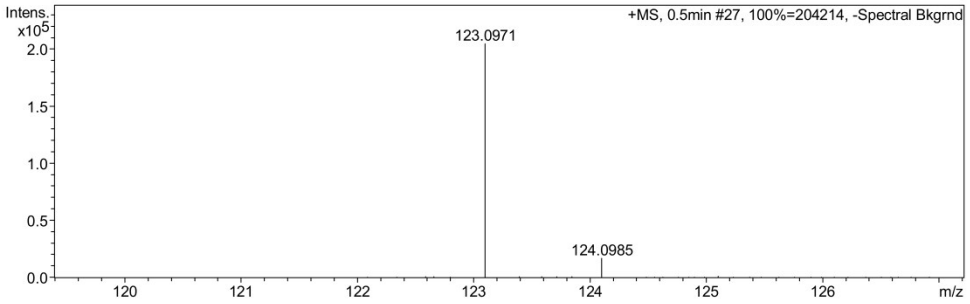
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	2.0 Bar
Focus	Not active	Set Capillary	4500 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	3000 m/z	Set Collision Cell RF	250.0 Vpp	Set Divert Valve	Source

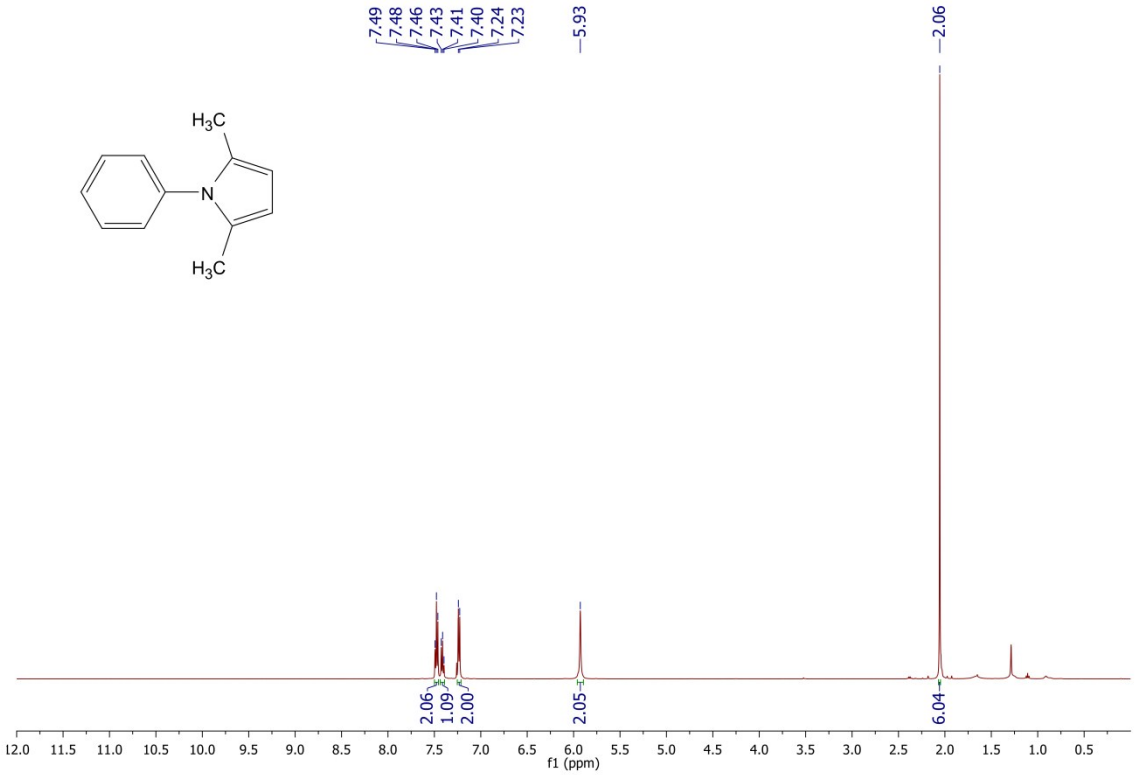


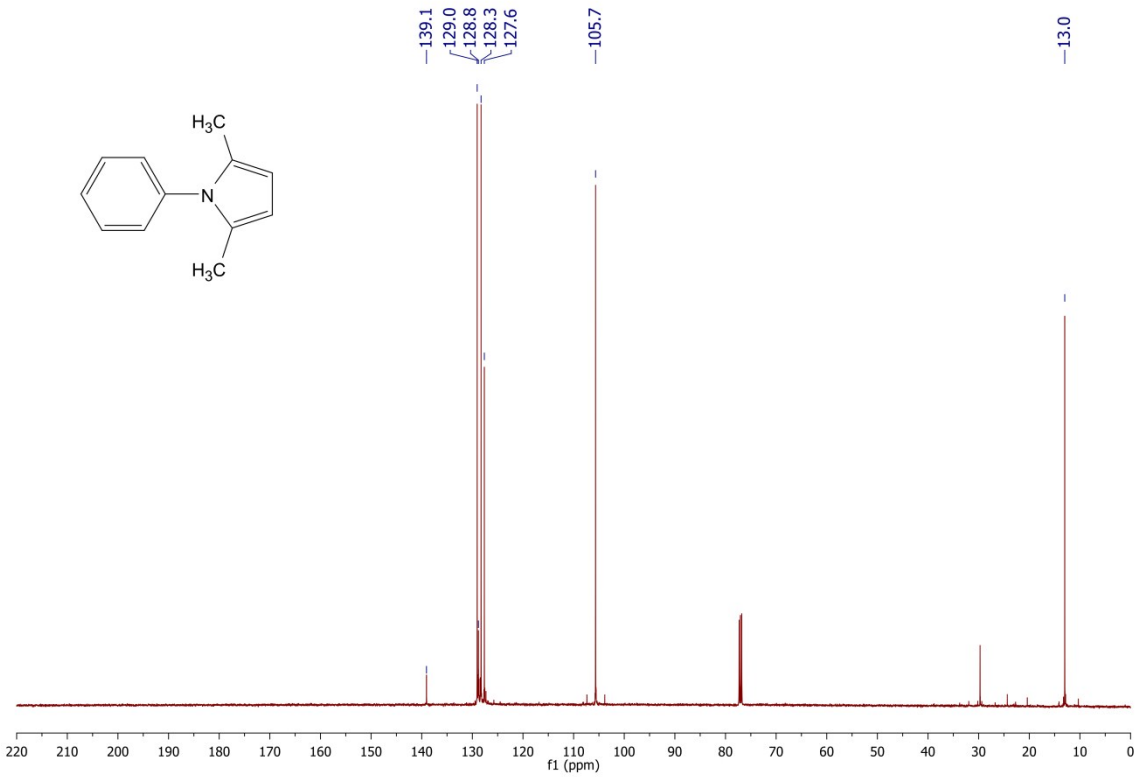
EIC 123.0000±0.3000 +All MS, -Spectral Bkgrnd, Smoothed (1.01,50,GA)



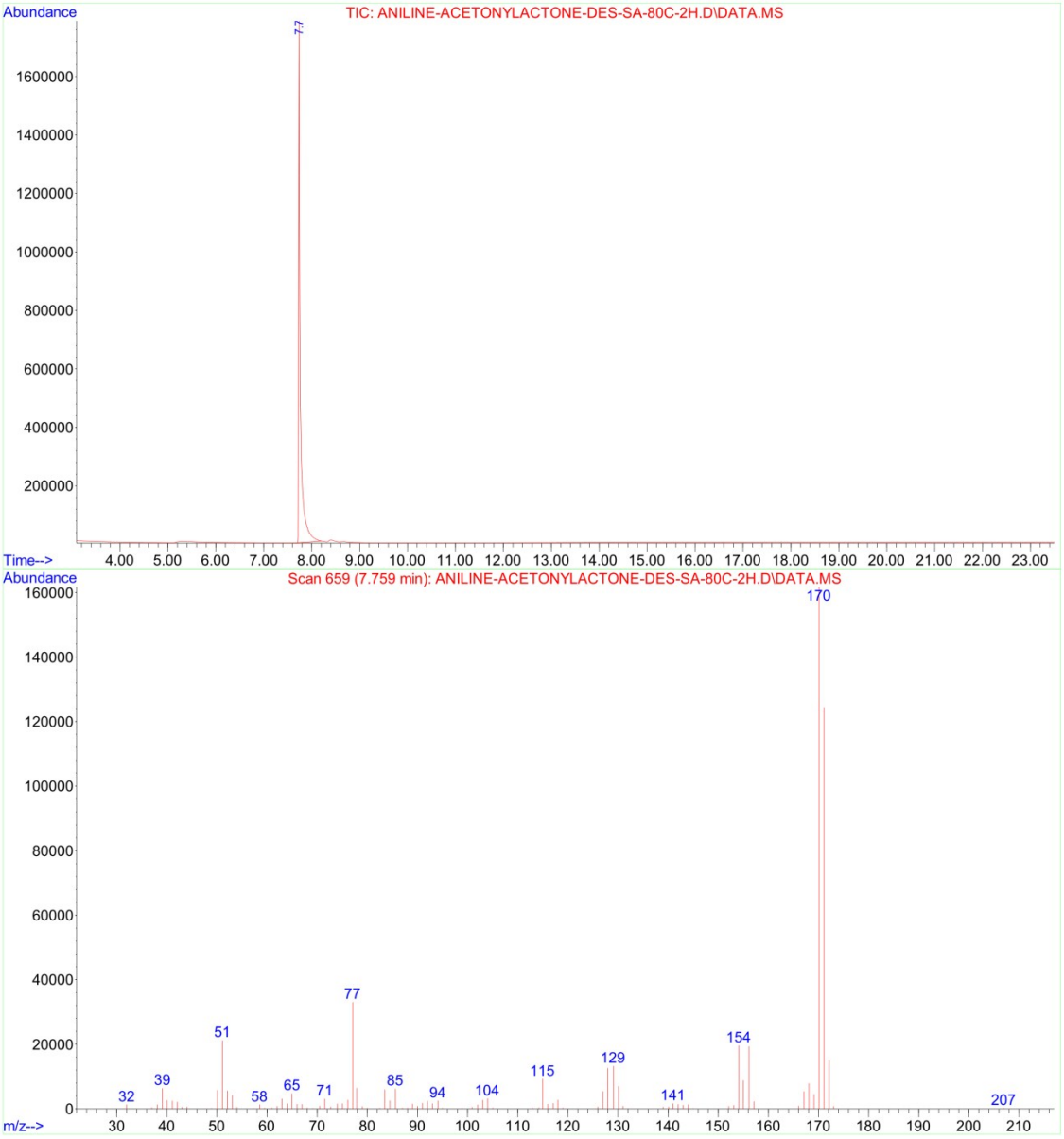


^{1}H NMR, ^{13}C NMR**, and GC-MS of 2,5-Dimethyl-1-phenyl-1*H*-pyrrole**

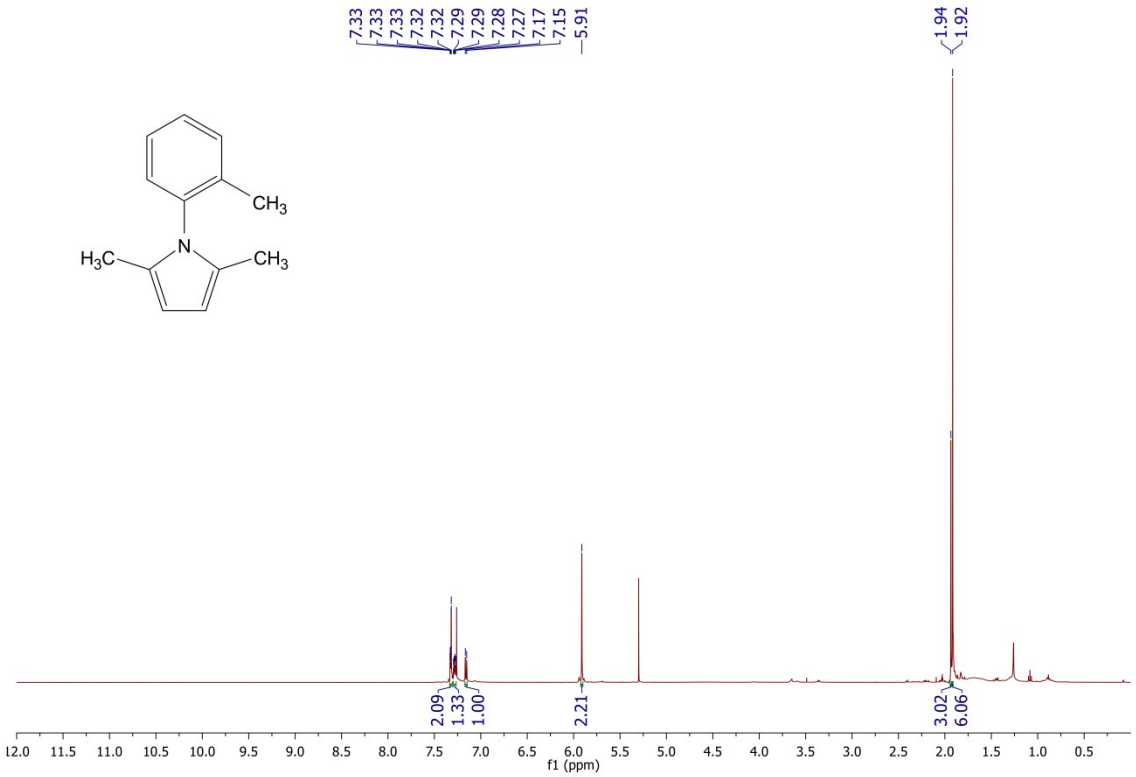


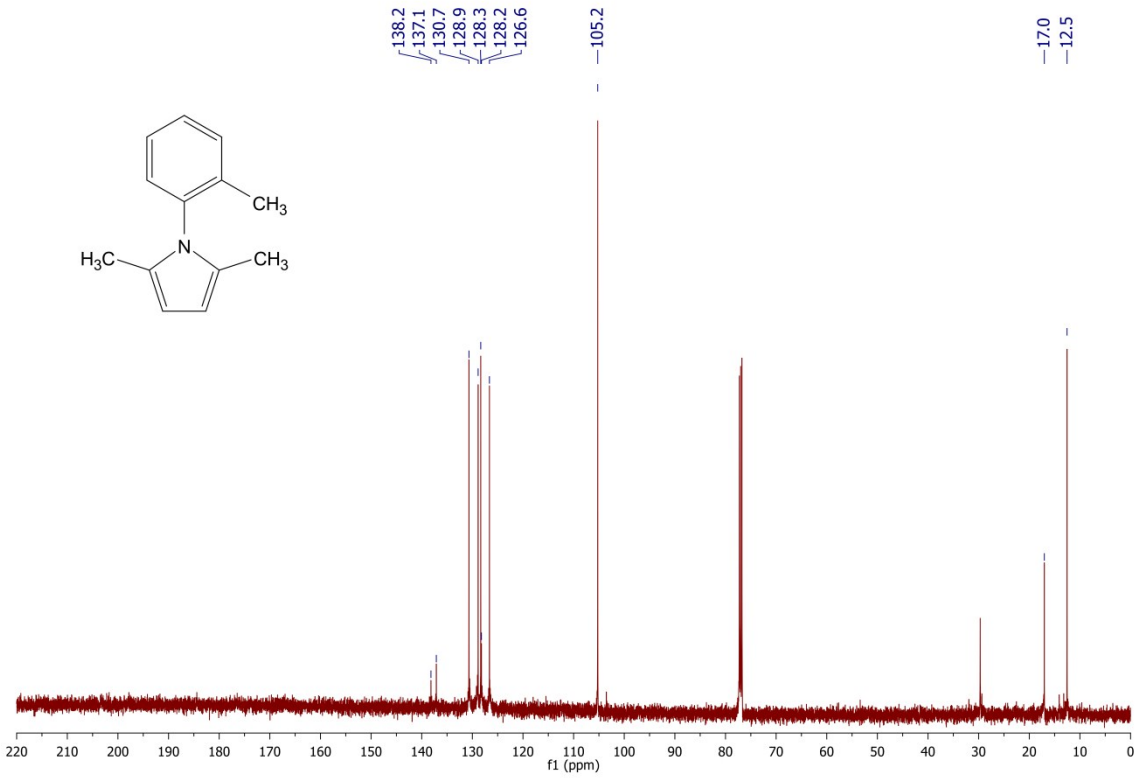


File :C:\GC-MS\2016\08.01.2016\ANILINE-ACETONYLACTONE-DES-SA-80C-2
... H.D
Operator : TRUONG HAI
Instrument : GCMSD
Acquired : 1 Aug 2016 16:44 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Sample Name: ANILINE-ACETONYLACTONE-DES-SA-80C-2H
Misc Info :

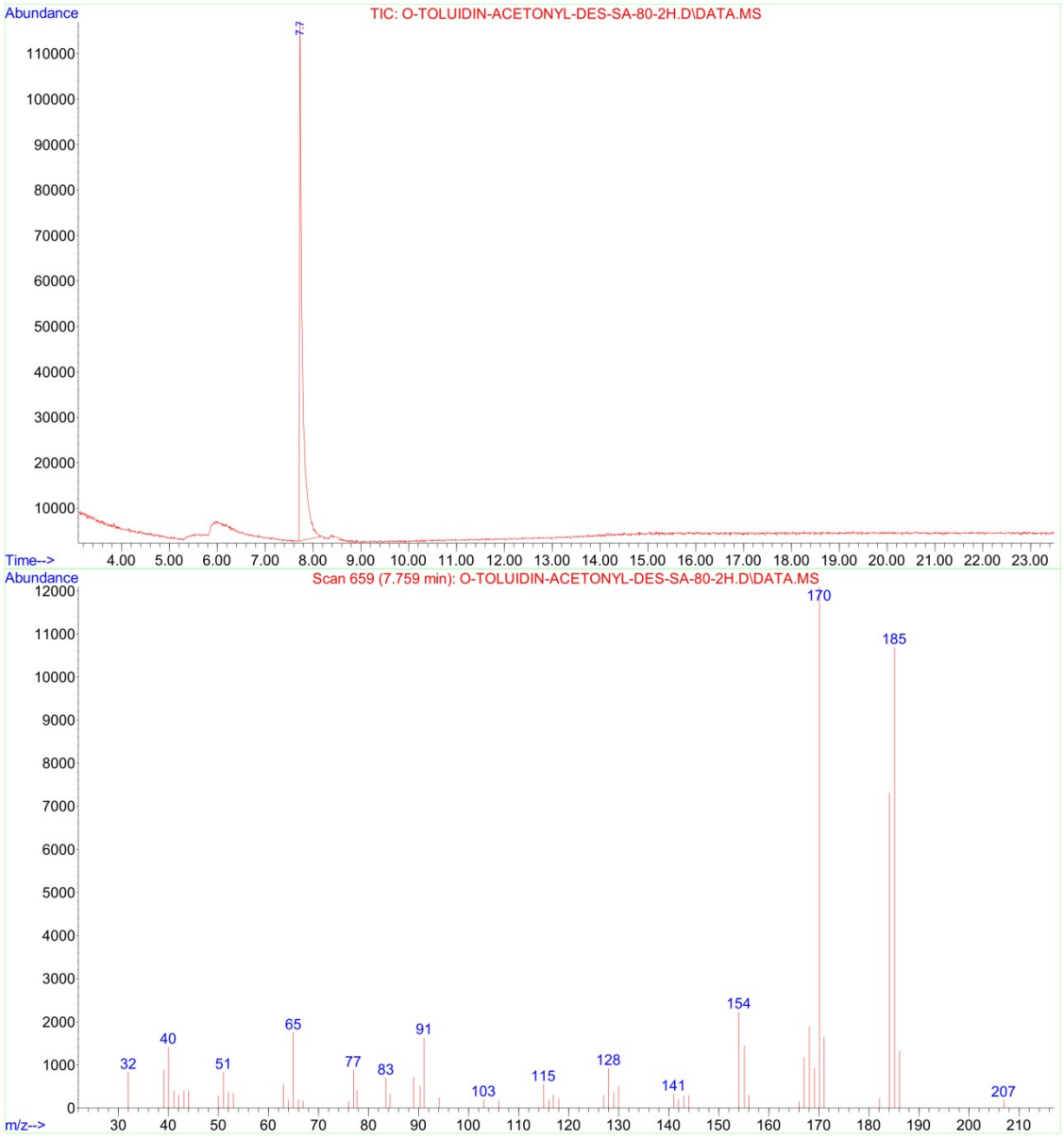


^{1}H NMR, ^{13}C NMR**, and GC-MS of 2,5-Dimethyl-1-(*o*-tolyl)-1*H*-pyrrole**

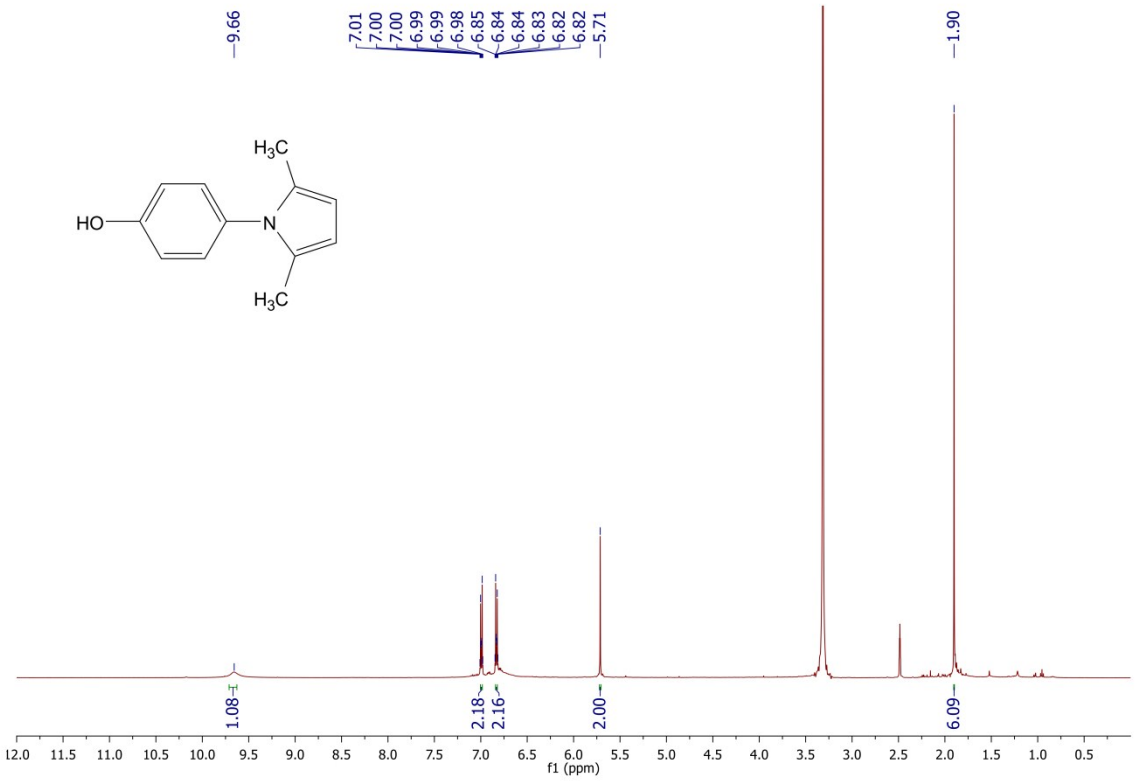


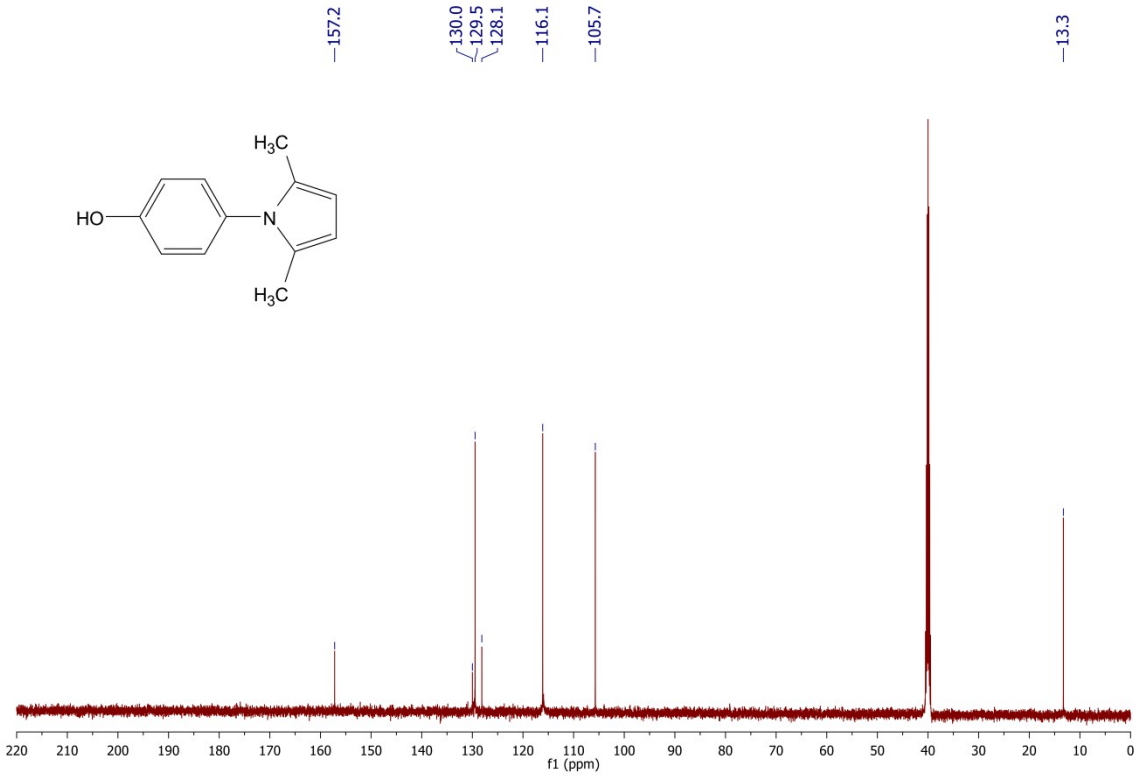


File :C:\GC-MS\2016\08.03.2016\O-TOLUIDIN-ACETONYL-DES-SA-80-2H.D
Operator : TRUONG HAI
Acquired : 4 Aug 2016 11:12 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Instrument : GCMSD
Sample Name: O-TOLUIDIN-ACETONYL-DES-SA-80-2H
Misc Info :
Vial Number: 3

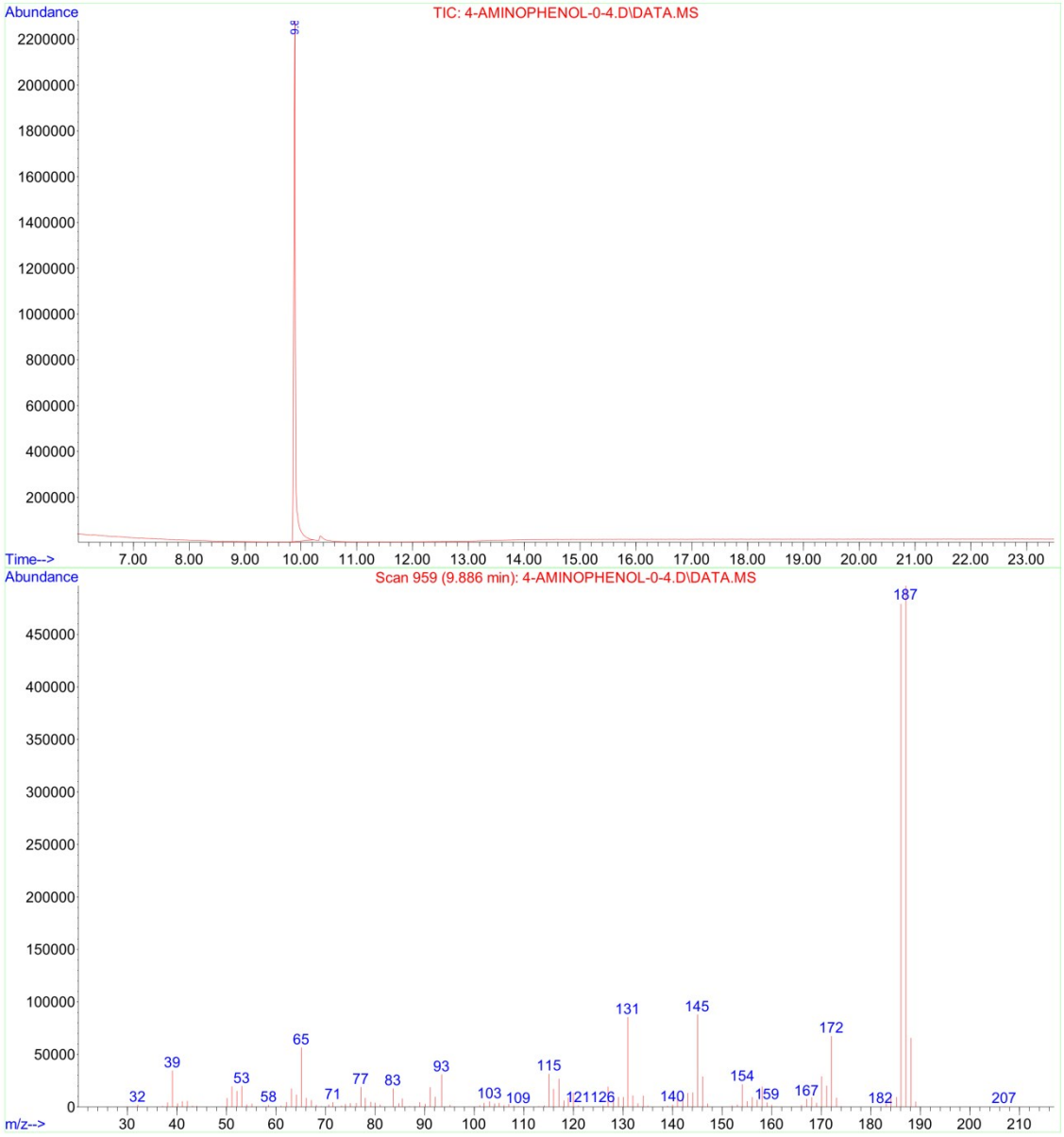


^{1}H NMR, ^{13}C NMR**, and GC-MS of 1-(4-Hydroxyphenyl)-2,5-dimethyl-1*H*-pyrrole**

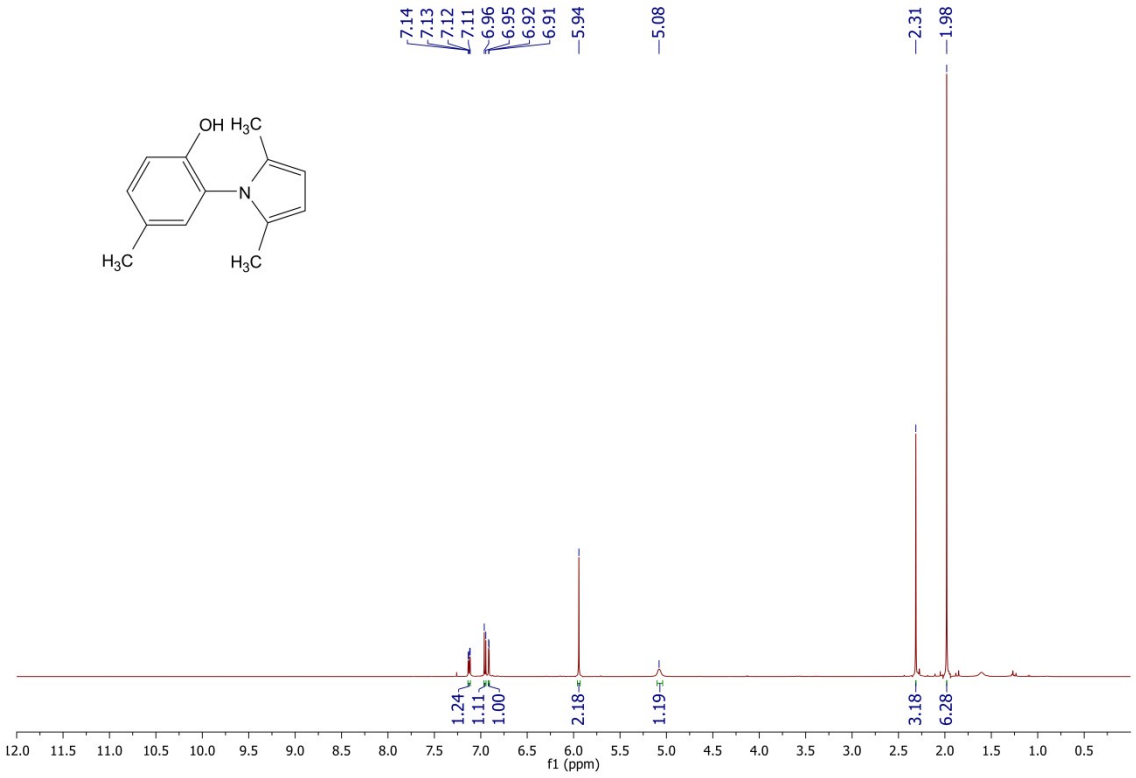


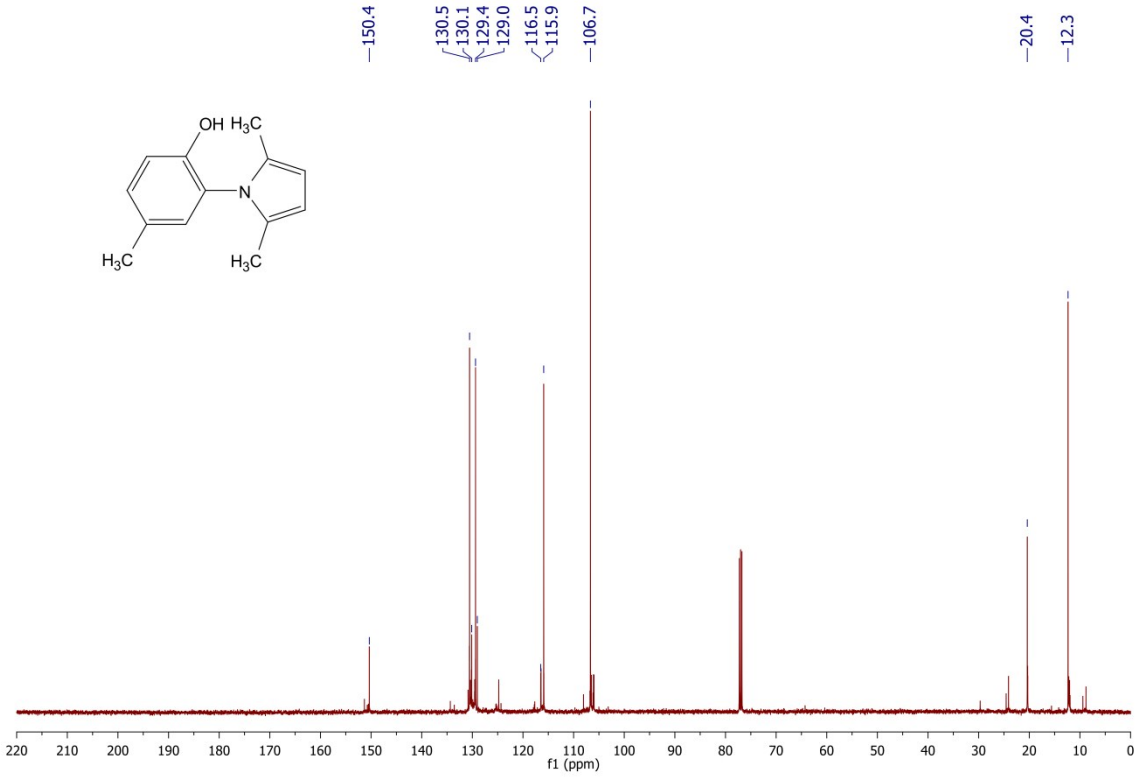


File :C:\GC-MS\2016\11.16.2016\4-AMINOPHENOL-0-4.D
Operator : TRUONG HAI
Acquired : 16 Nov 2016 16:21 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Instrument : GCMSD
Sample Name: 4-AMINOPHENOL-0-4
Misc Info :
Vial Number: 2



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





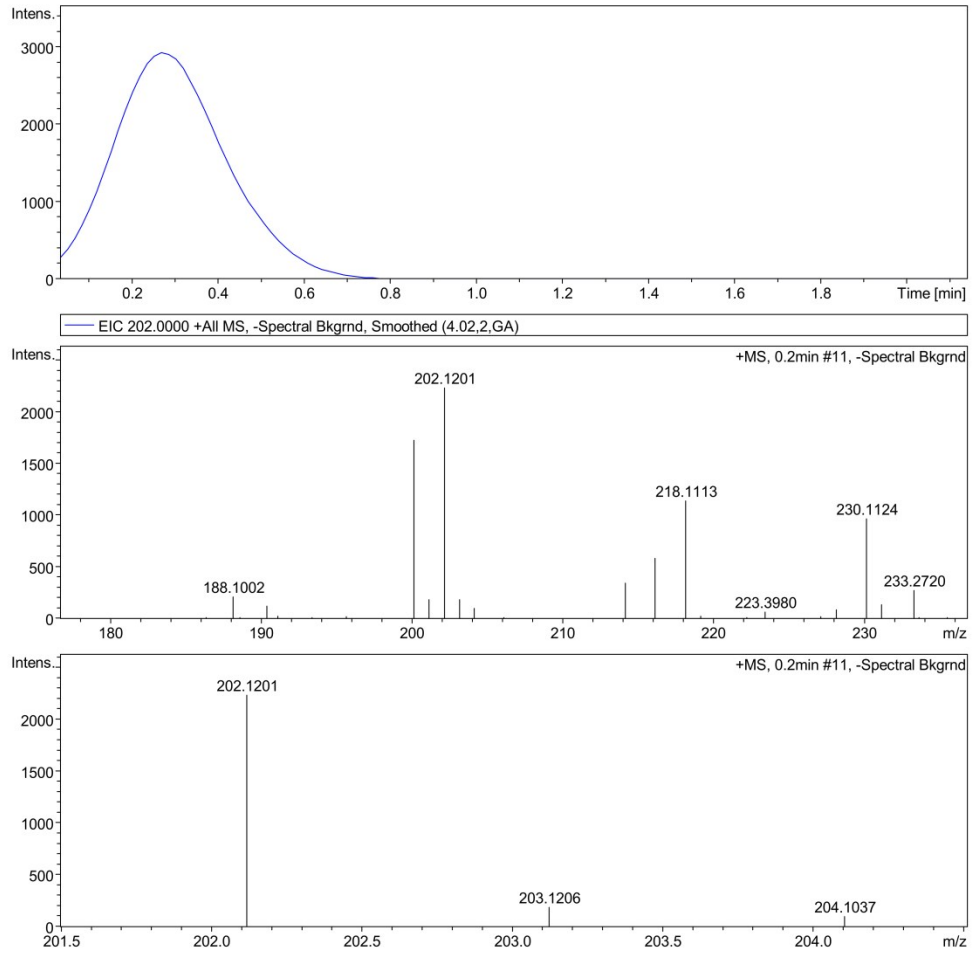
Display Report

Analysis Info

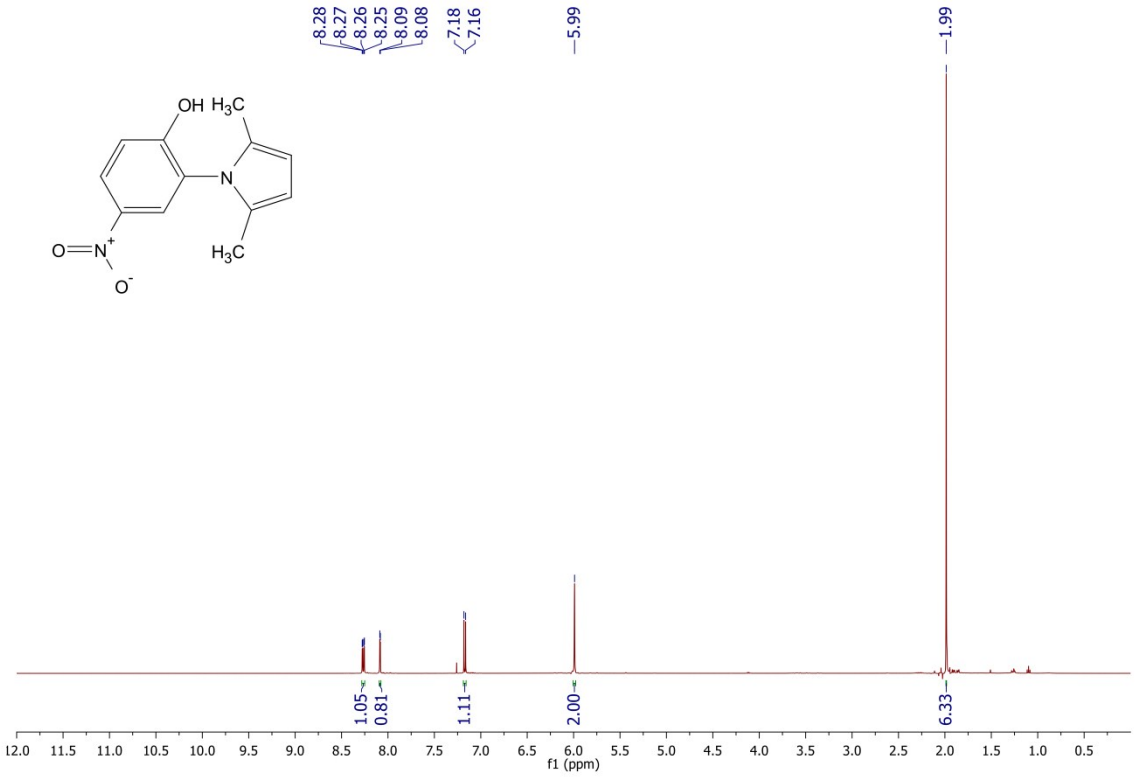
Analysis Name	D:\Data\2016\2 ami_1-b,1_01_2267.d	Acquisition Date	12/30/2016 3:54:34 PM
Method	dmm 2017.m	Operator	Anh Mai
Sample Name	2 ami	Instrument	micrOTOF-Q 10187
Comment			

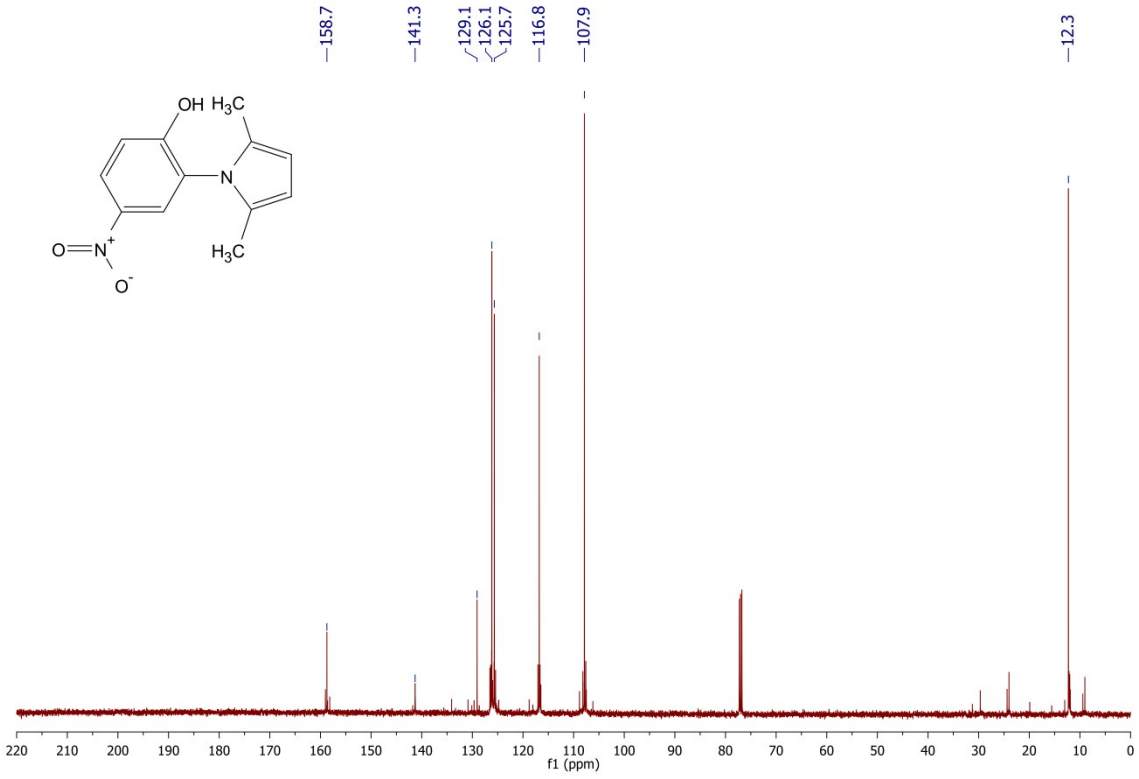
Acquisition Parameter

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



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





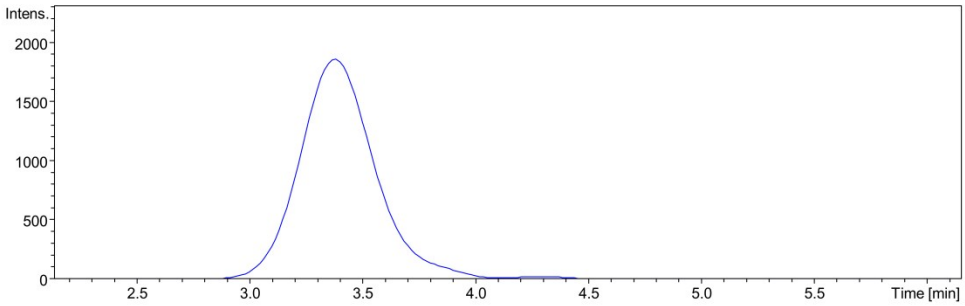
Display Report

Analysis Info

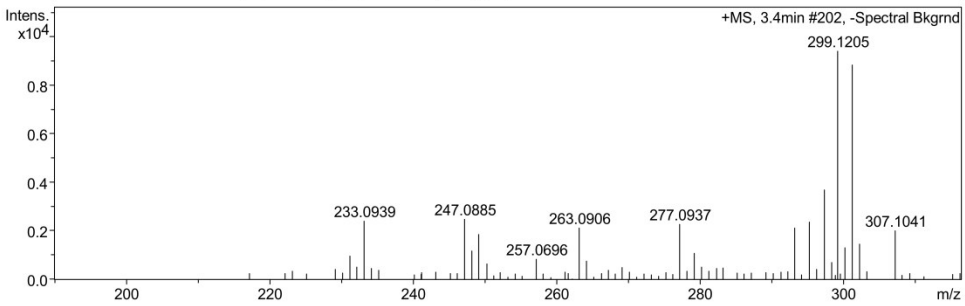
Analysis Name	D:\Data\2016\2 amino 4 ntro_1-b,3_01_2264.d	Acquisition Date	12/29/2016 6:23:13 PM
Method	dmm 2017.m	Operator	Anh Mai
Sample Name	2 amino 4 ntro	Instrument	micrOTOF-Q 10187
Comment			

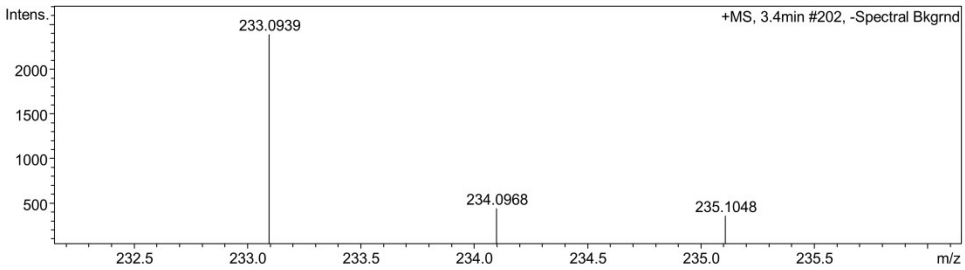
Acquisition Parameter

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

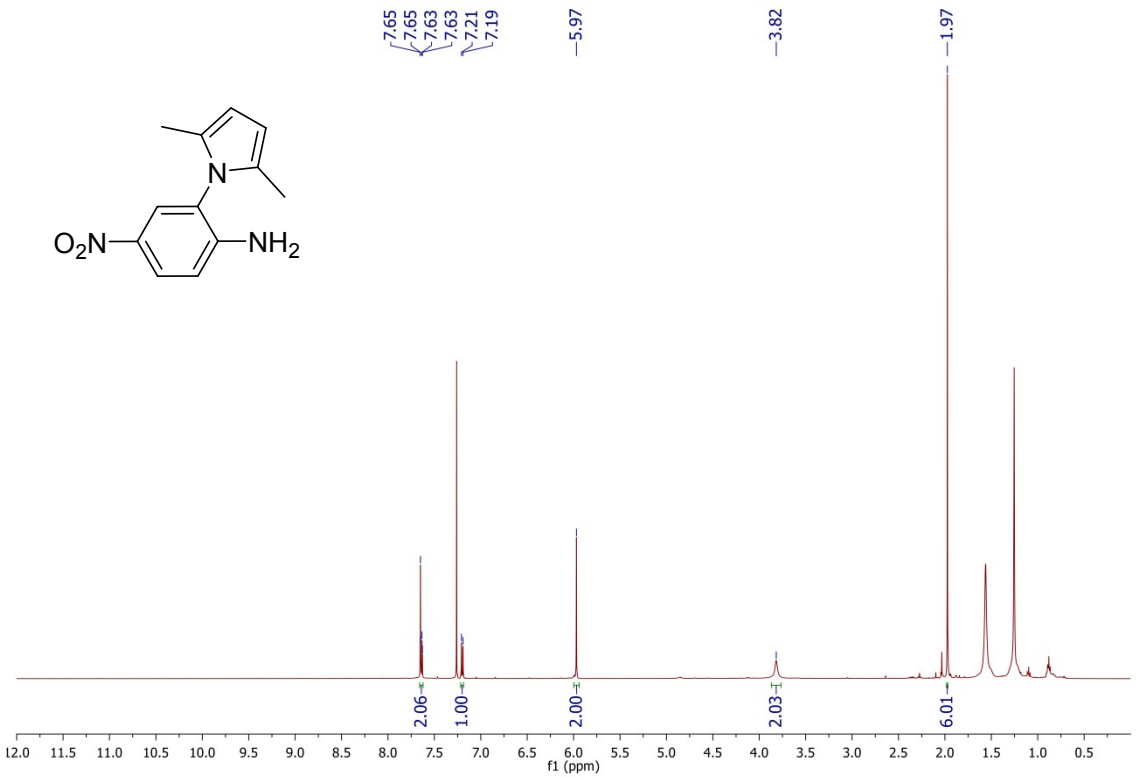


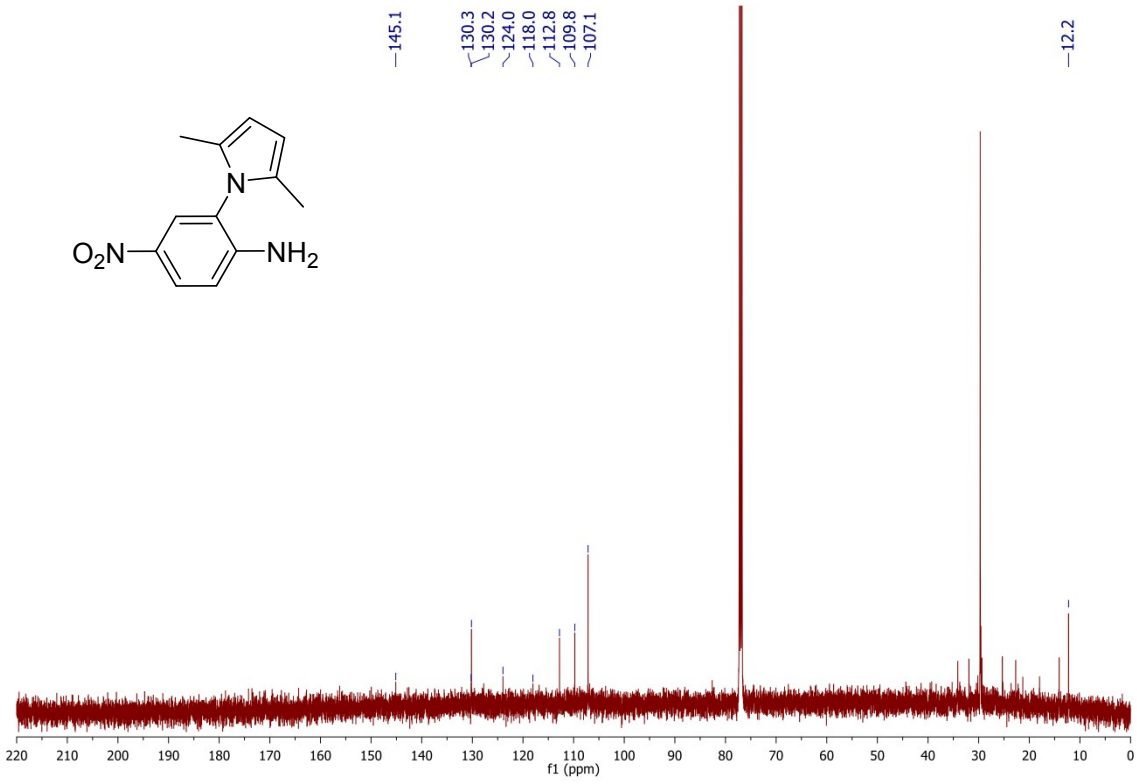
EIC 233.0000 +All MS, -Spectral Bkgrnd, Smoothed (4.01,2,GA)





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





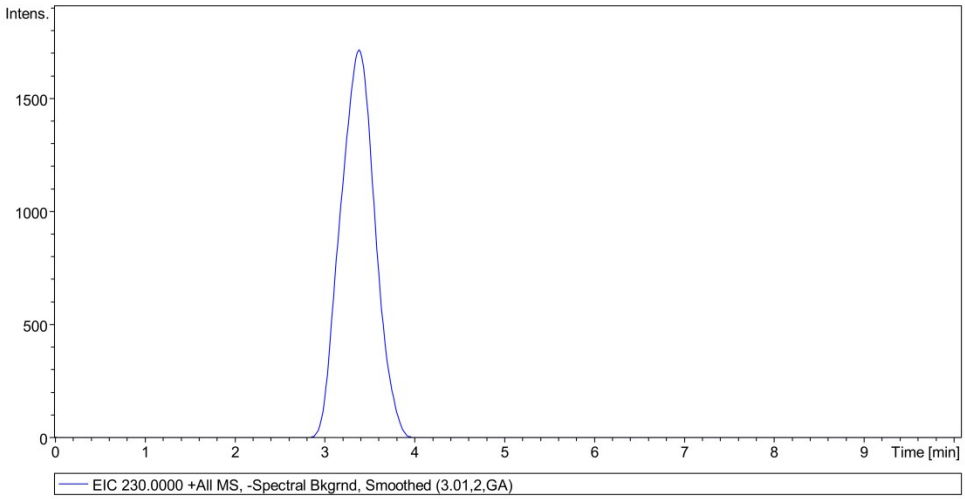
Display Report

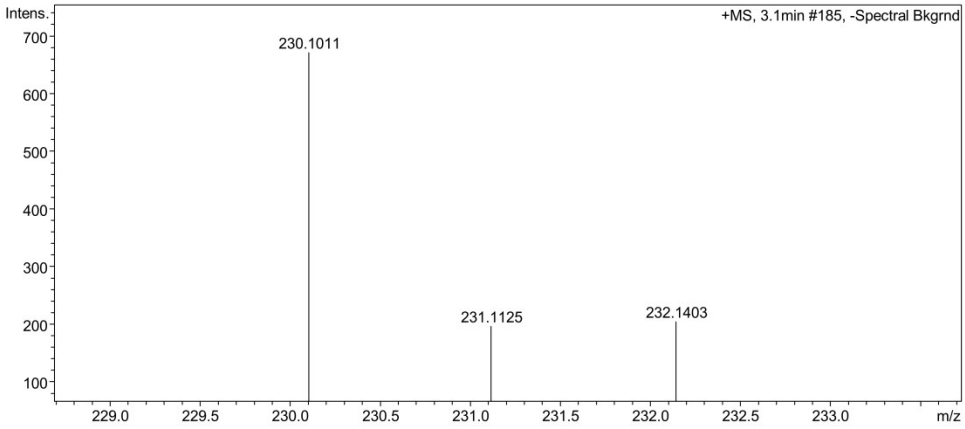
Analysis Info

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			

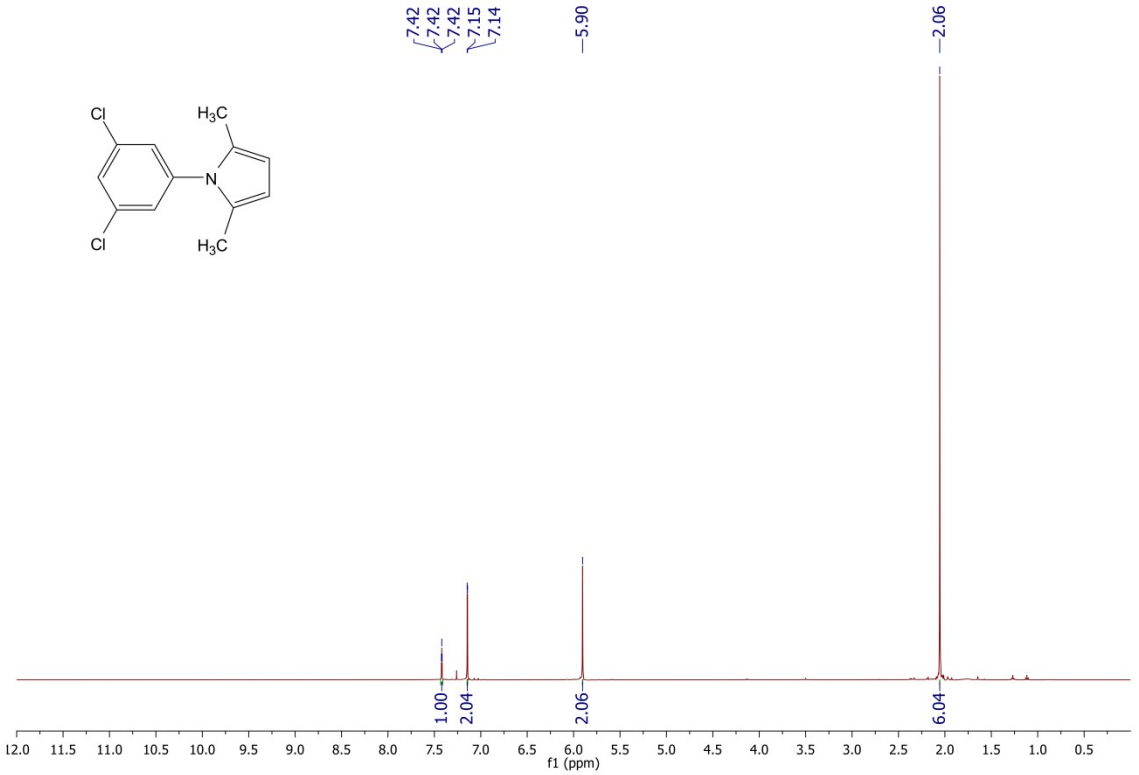
Acquisition Parameter

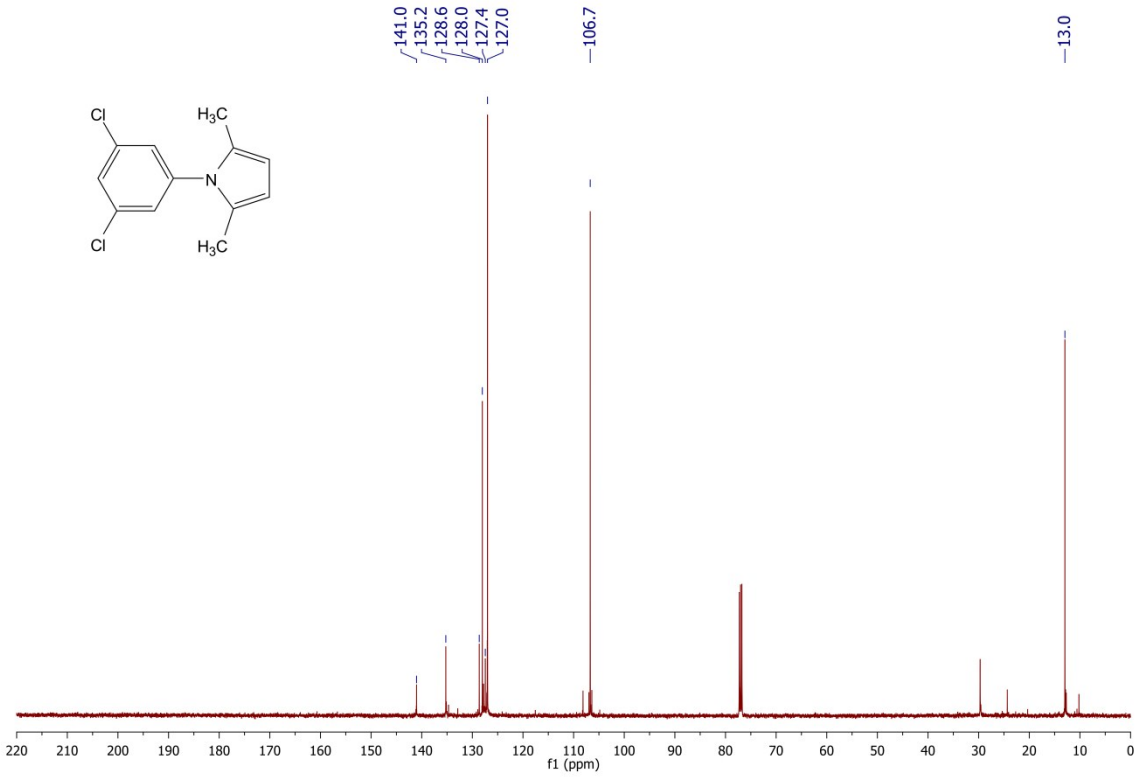
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4000 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	9.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	450.0 Vpp	Set Divert Valve	Source



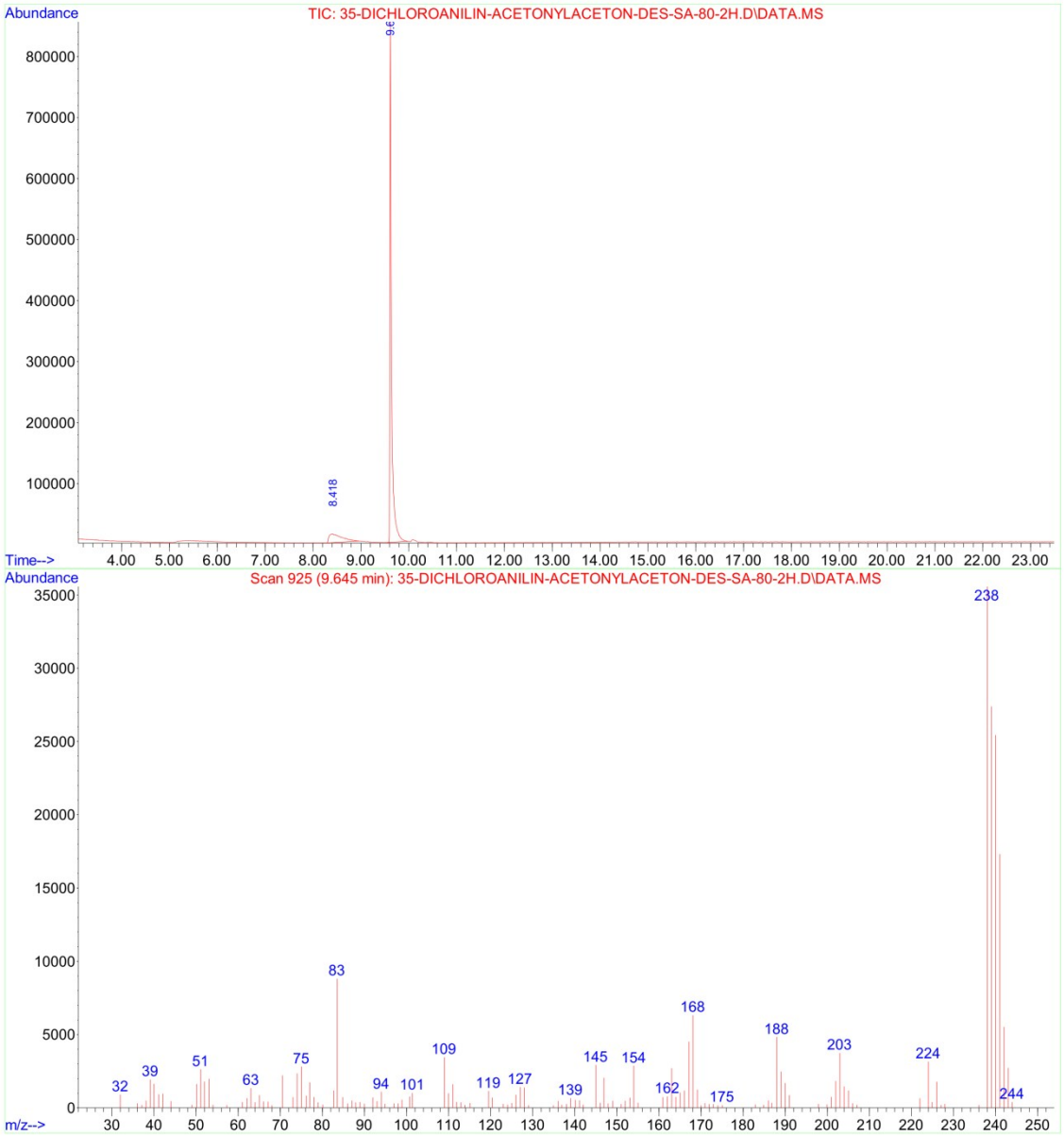


^{1}H NMR, ^{13}C NMR**, and GC-MS of 1-(3,5-Dichlorophenyl)-2,5-dimethyl-1*H*-pyrrole**

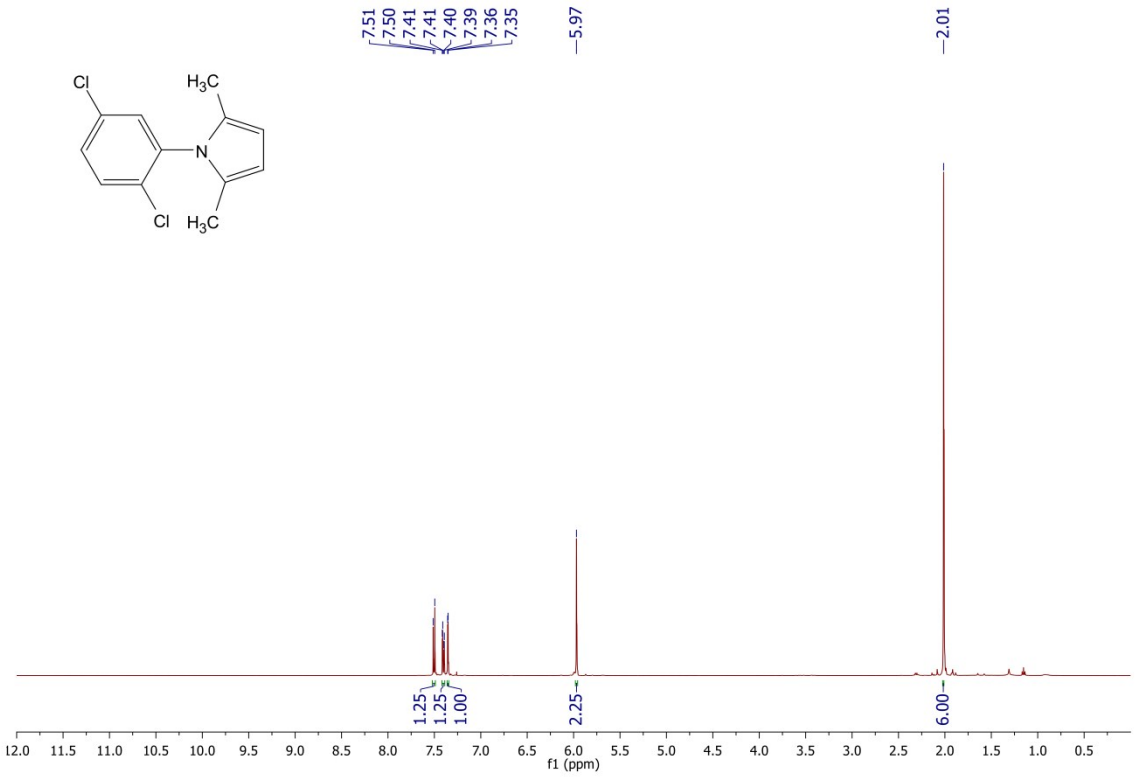


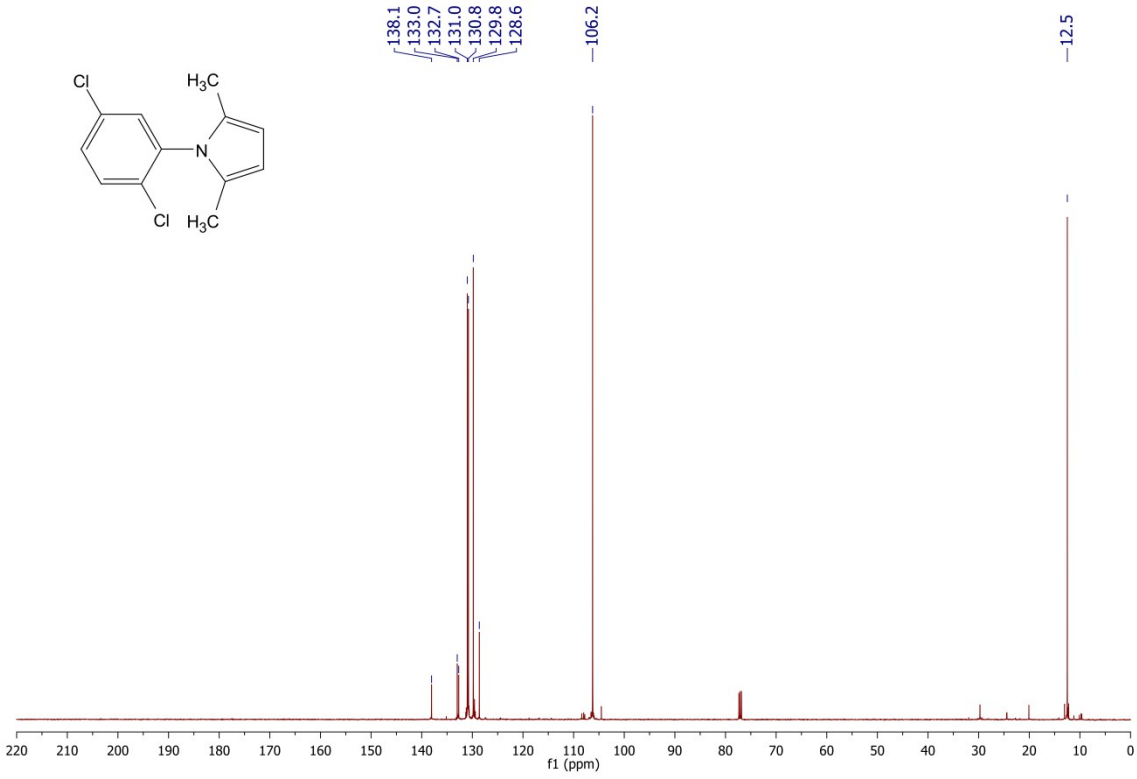


File :C:\GC-MS\2016\08.03.2016\35-DICHLOROANILIN-ACETONYLACETON-DE
... S-SA-80-2H.D
Operator : TRUONG HAI
Instrument : GCMSD
Acquired : 3 Aug 2016 18:03 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Sample Name: 35-DICHLOROANILIN-ACETONYLACETON-DES-SA-80-2H
Misc Info :

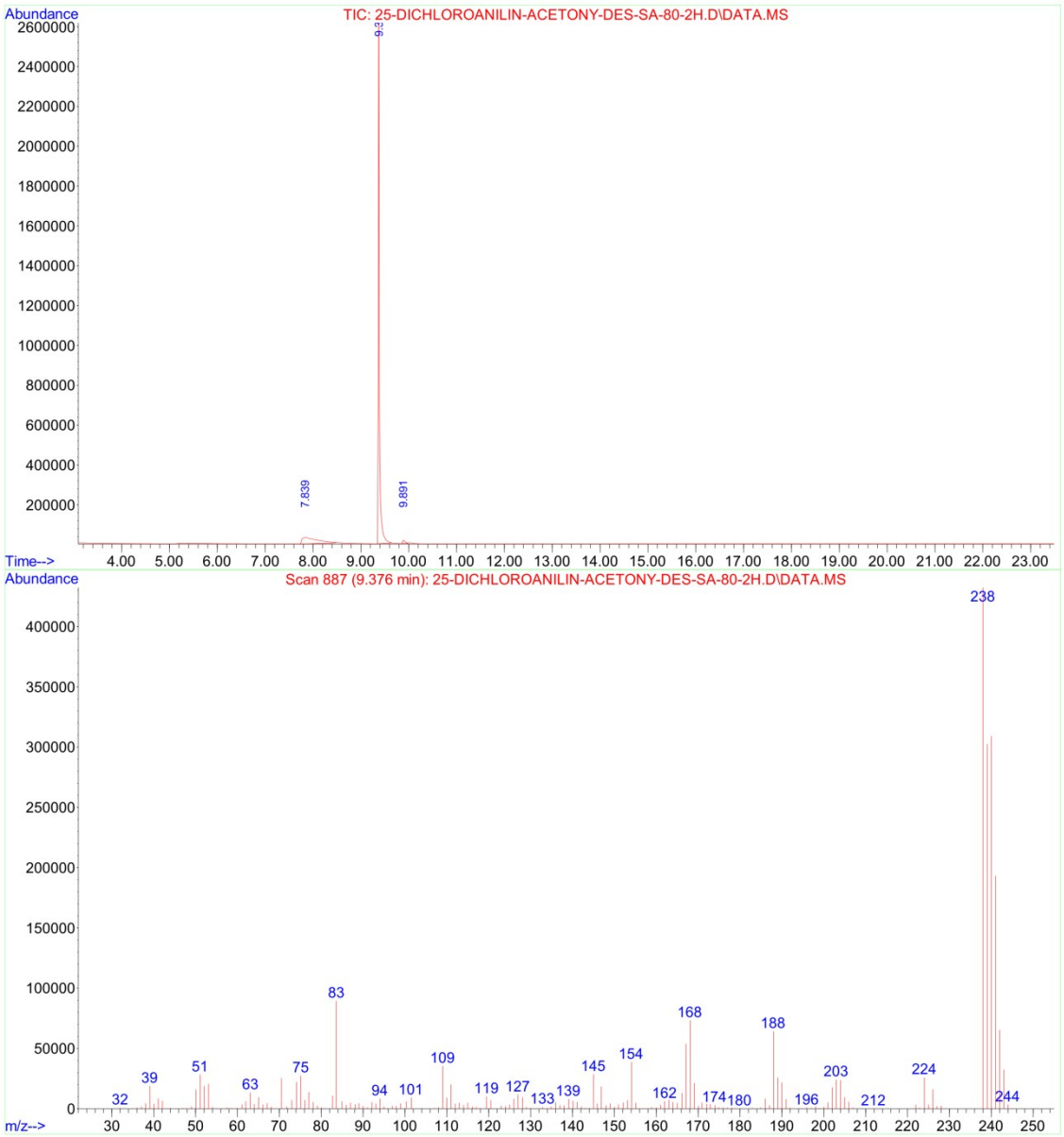


^{1}H NMR, ^{13}C NMR, and GC-MS of 1-(2,5-Dichlorophenyl)-2,5-dimethyl-1*H*-pyrrole

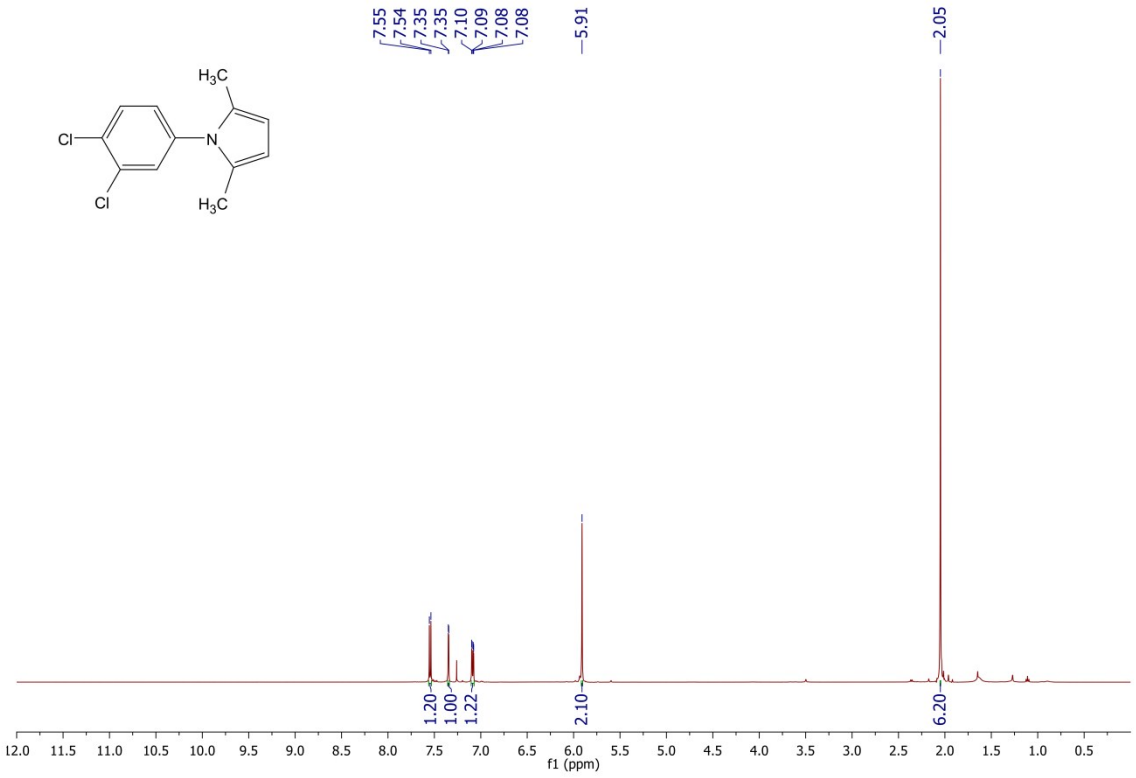


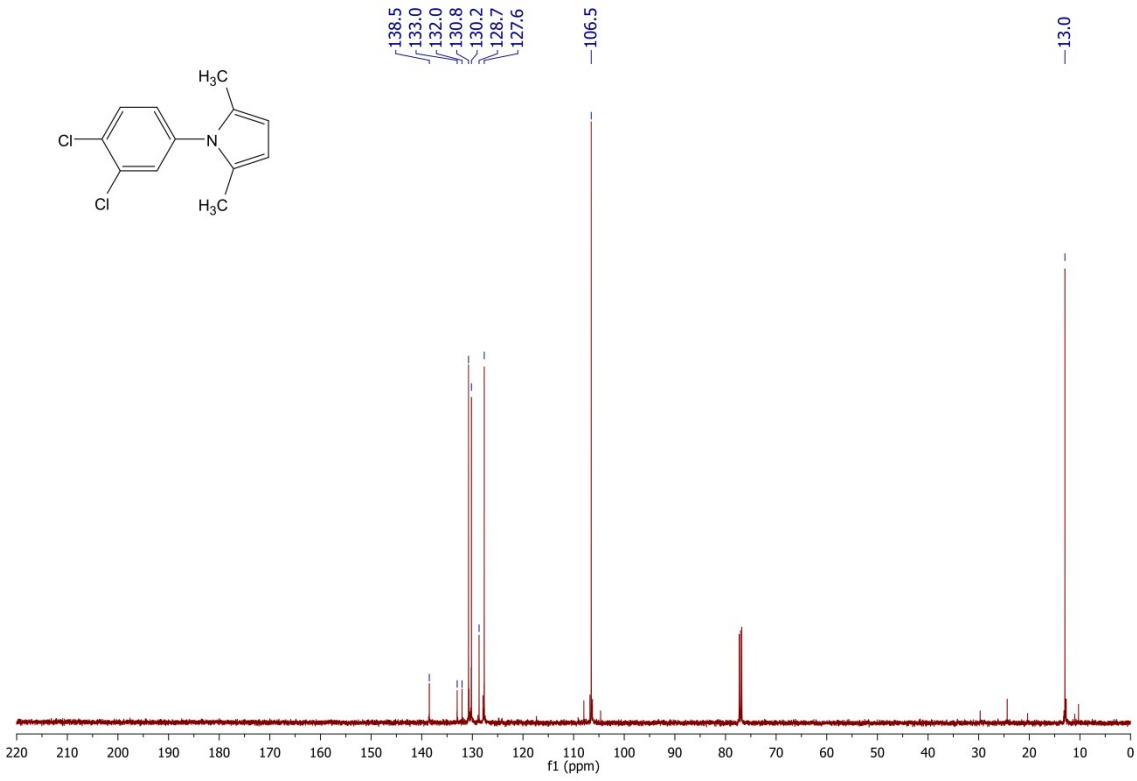


File :C:\GC-MS\2016\08.03.2016\25-DICHLOROANILIN-ACETONY-DES-SA-80
... -2H.D
Operator : TRUONG HAI
Instrument : GCMSD
Acquired : 8 Aug 2016 16:19 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Sample Name: 25-DICHLOROANILIN-ACETONY-DES-SA-80-2H
Misc Info :

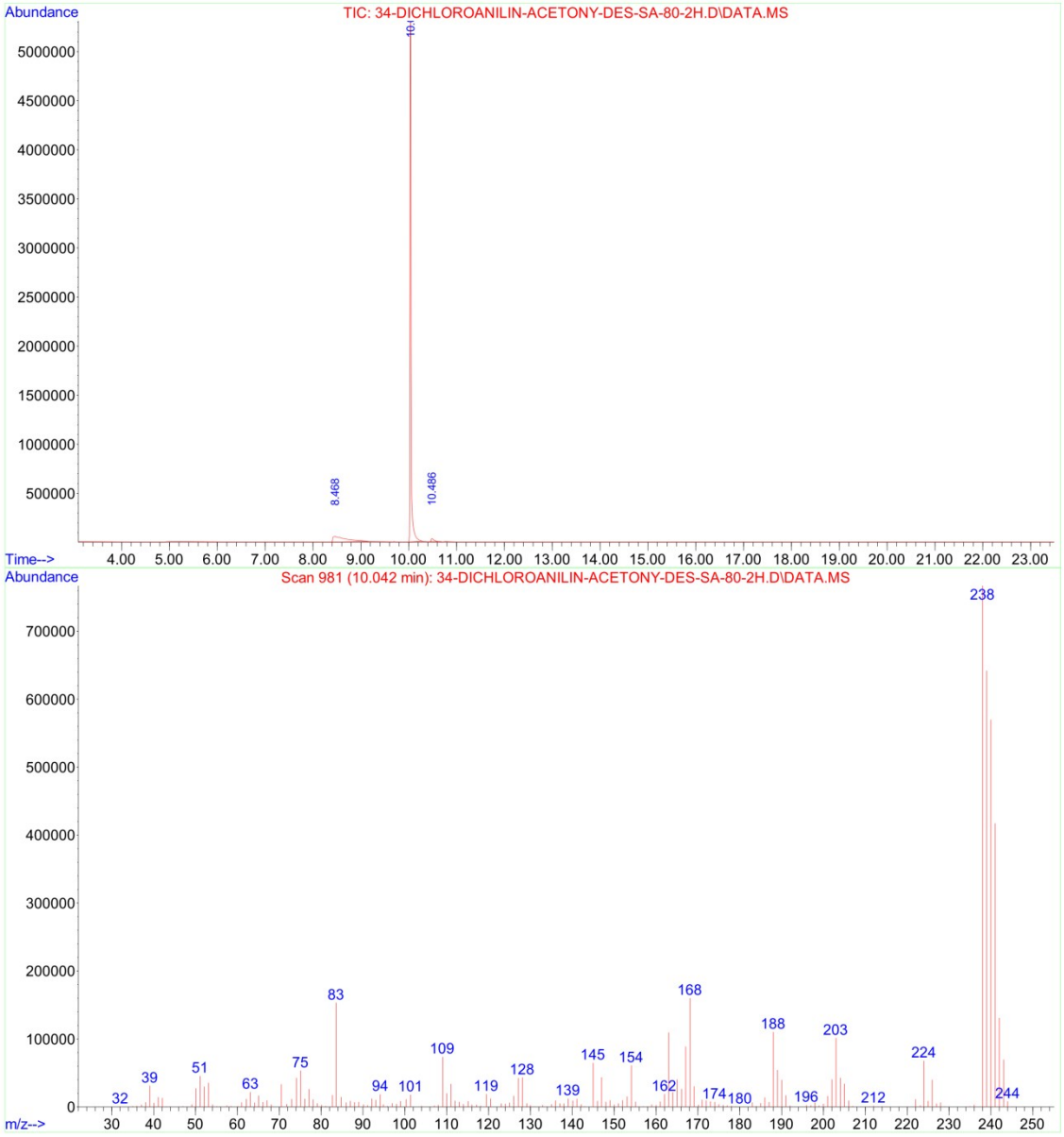


^{1}H NMR, ^{13}C NMR**, and GC-MS of 1-(3,4-Dichlorophenyl)-2,5-dimethyl-1*H*-pyrrole**

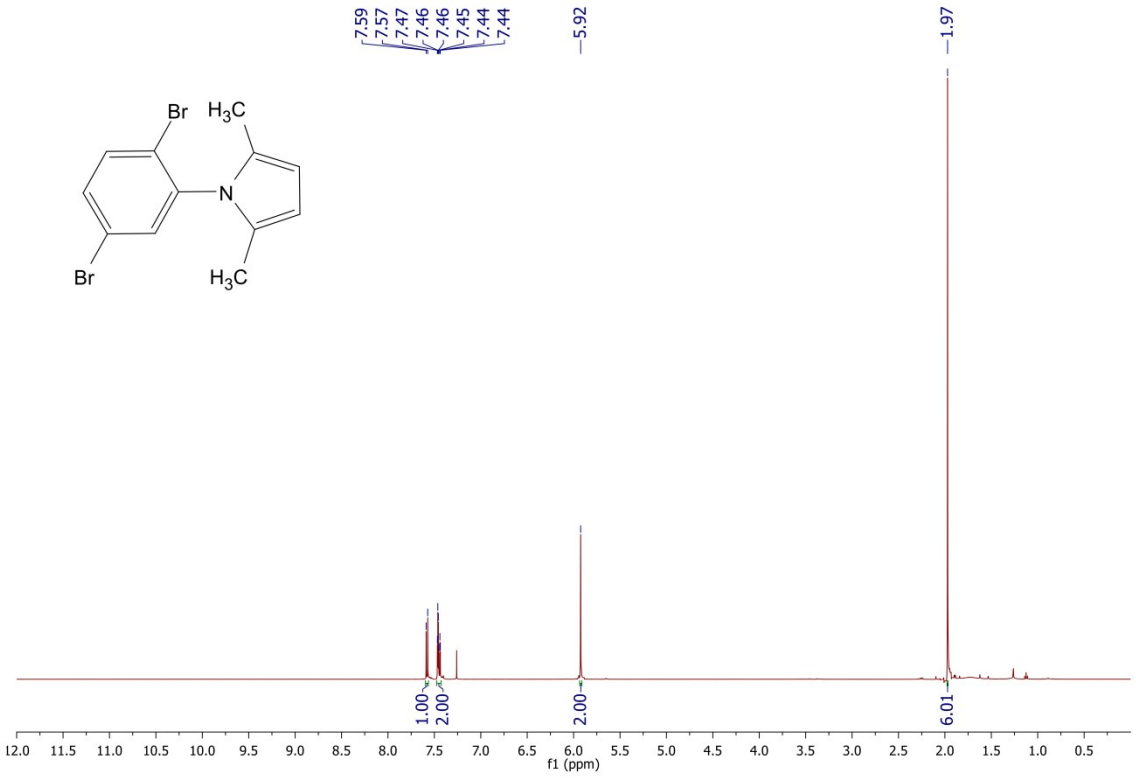


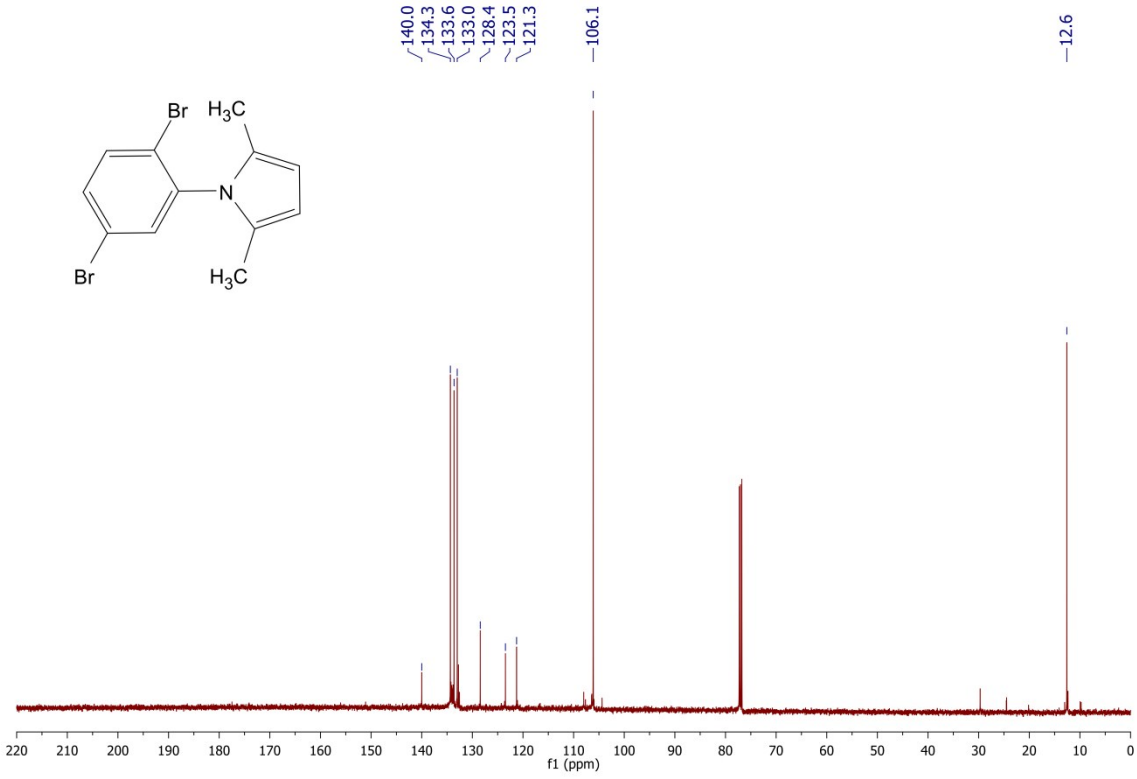


File :C:\GC-MS\2016\08.03.2016\34-DICHLOROANILIN-ACETONY-DES-SA-80
... -2H.D
Operator : TRUONG HAI
Instrument : GCMSD
Acquired : 8 Aug 2016 16:54 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Sample Name: 34-DICHLOROANILIN-ACETONY-DES-SA-80-2H
Misc Info :

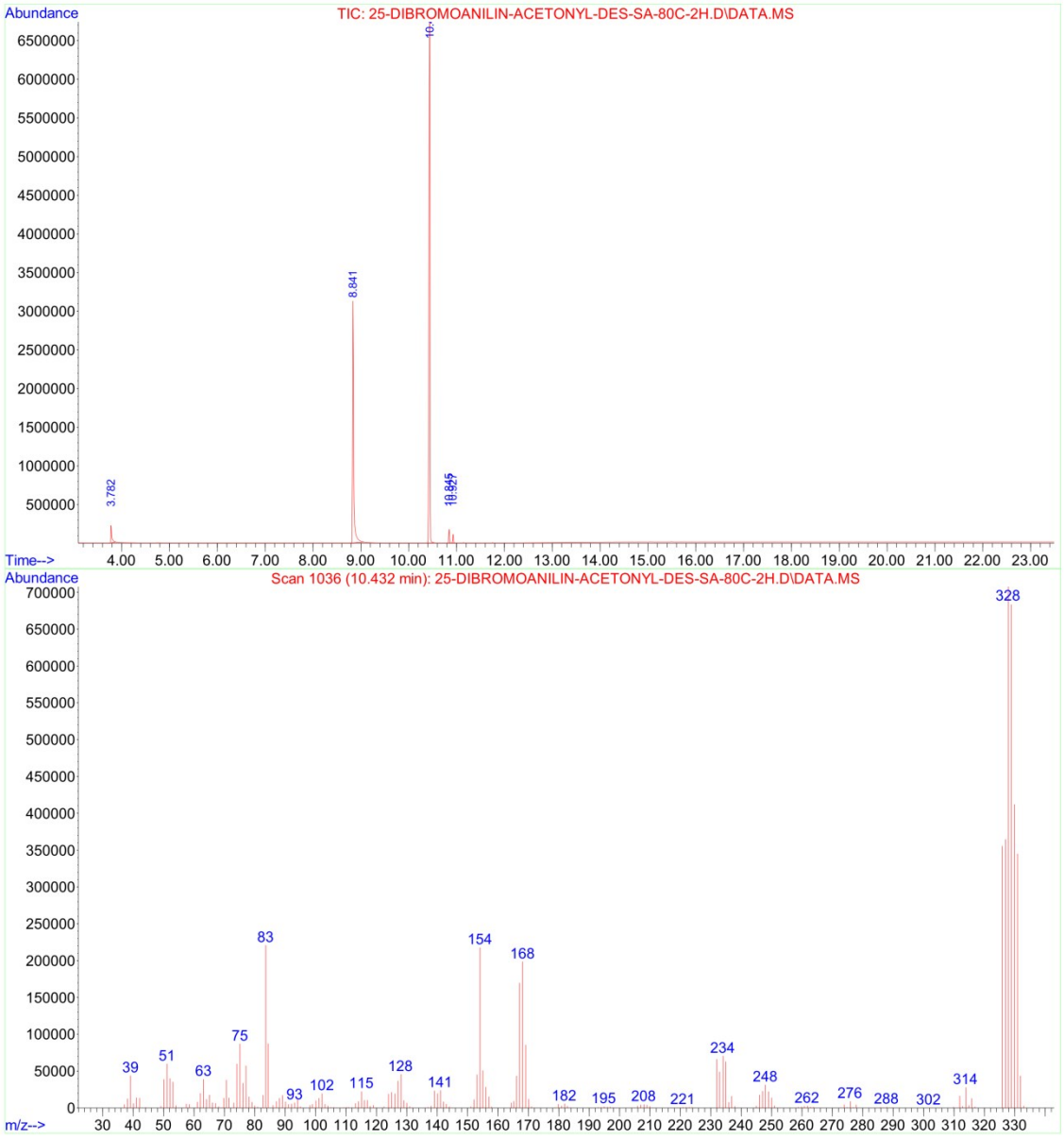


^{1}H NMR, ^{13}C NMR**, and GC-MS of 1-(2,5-Dibromophenyl)-2,5-dimethyl-1*H*-pyrrole**

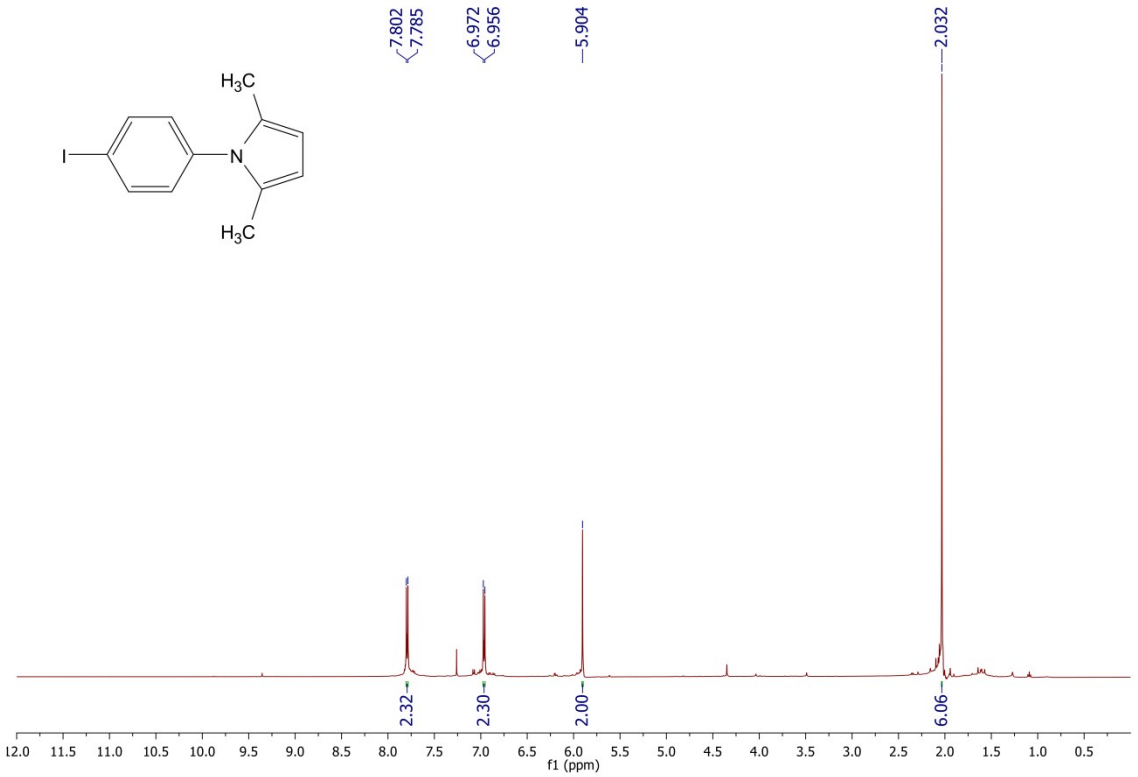


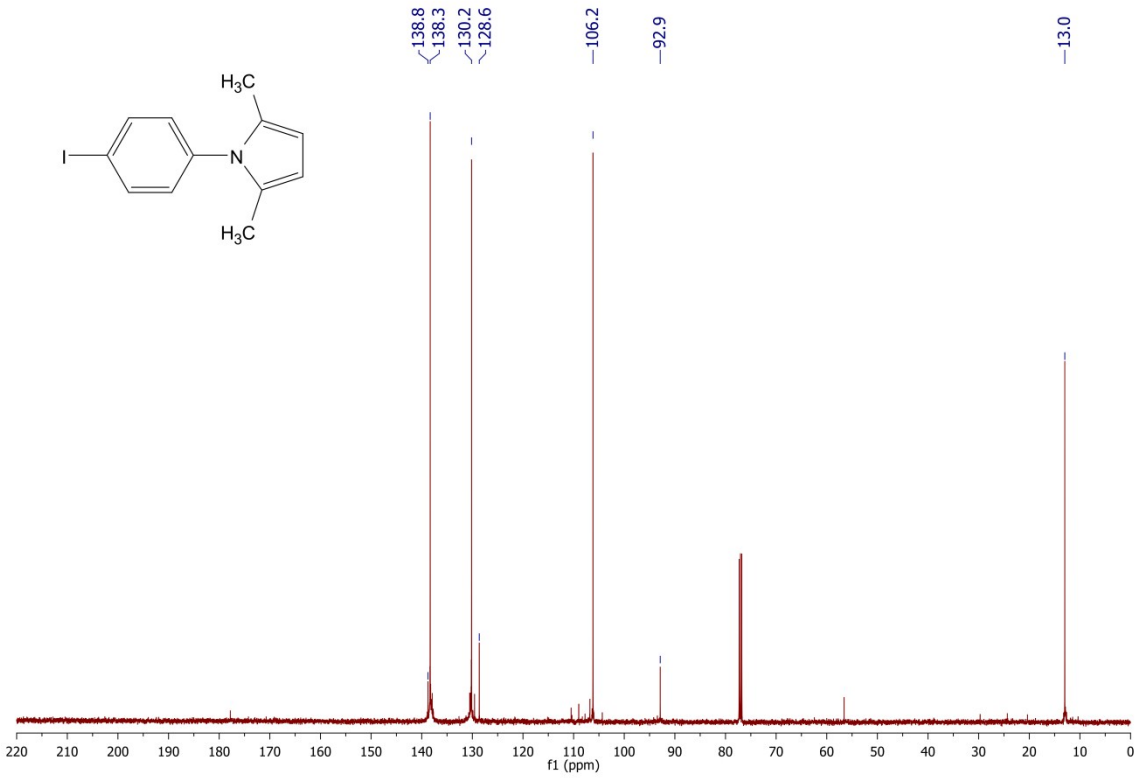


File :C:\GC-MS\2016\08.17.2016\25-DIBROMOANILIN-ACETONYL-DES-SA-80
... C-2H.D
Operator : TRUONG HAI
Instrument : GCMSD
Acquired : 17 Aug 2016 13:03 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Sample Name: 25-DIBROMOANILIN-ACETONYL-DES-SA-80C-2H
Misc Info :

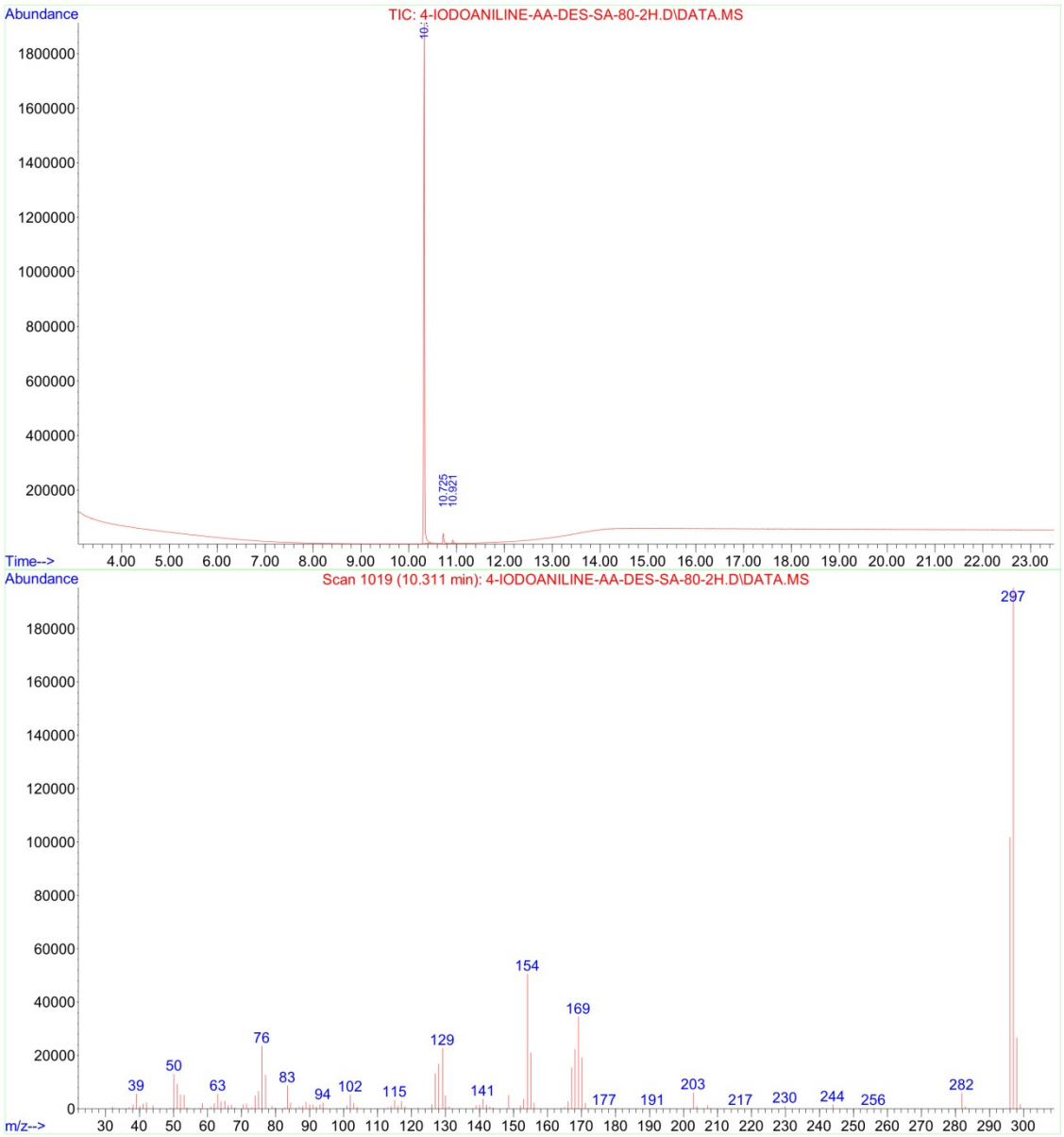


^{1}H NMR, ^{13}C NMR**, and GC-MS of 1-(4-Iodophenyl)-2,5-dimethyl-1*H*-pyrrole**

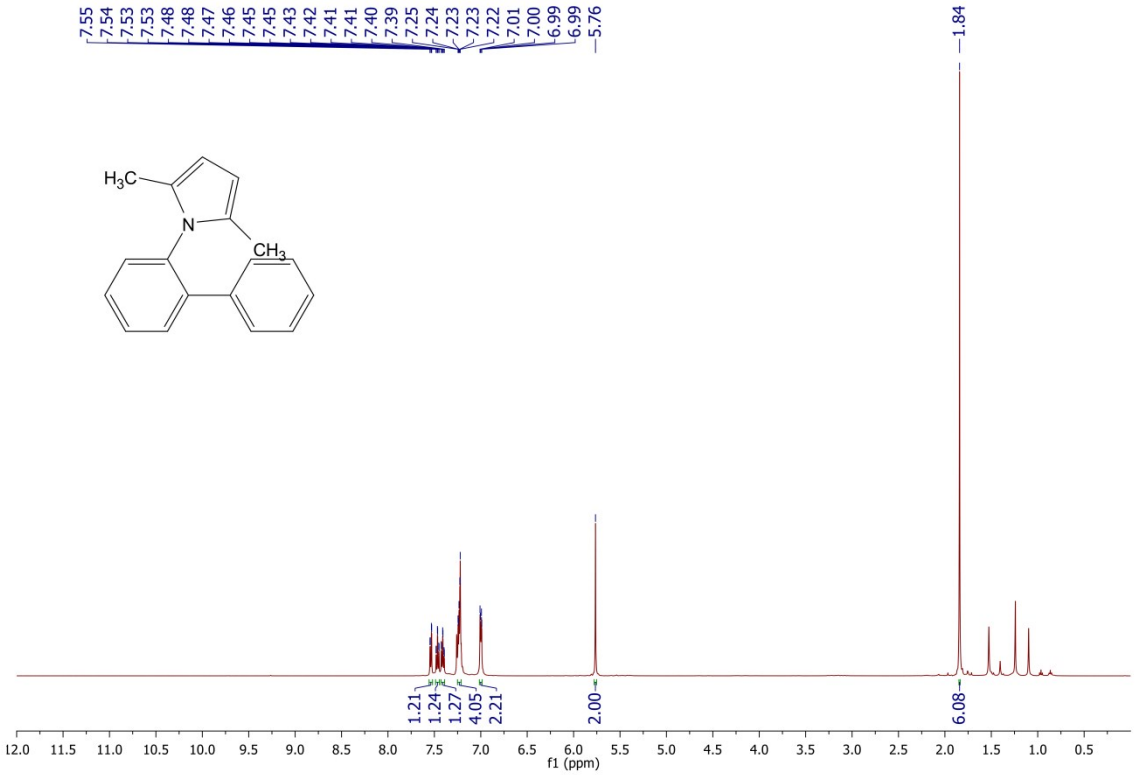


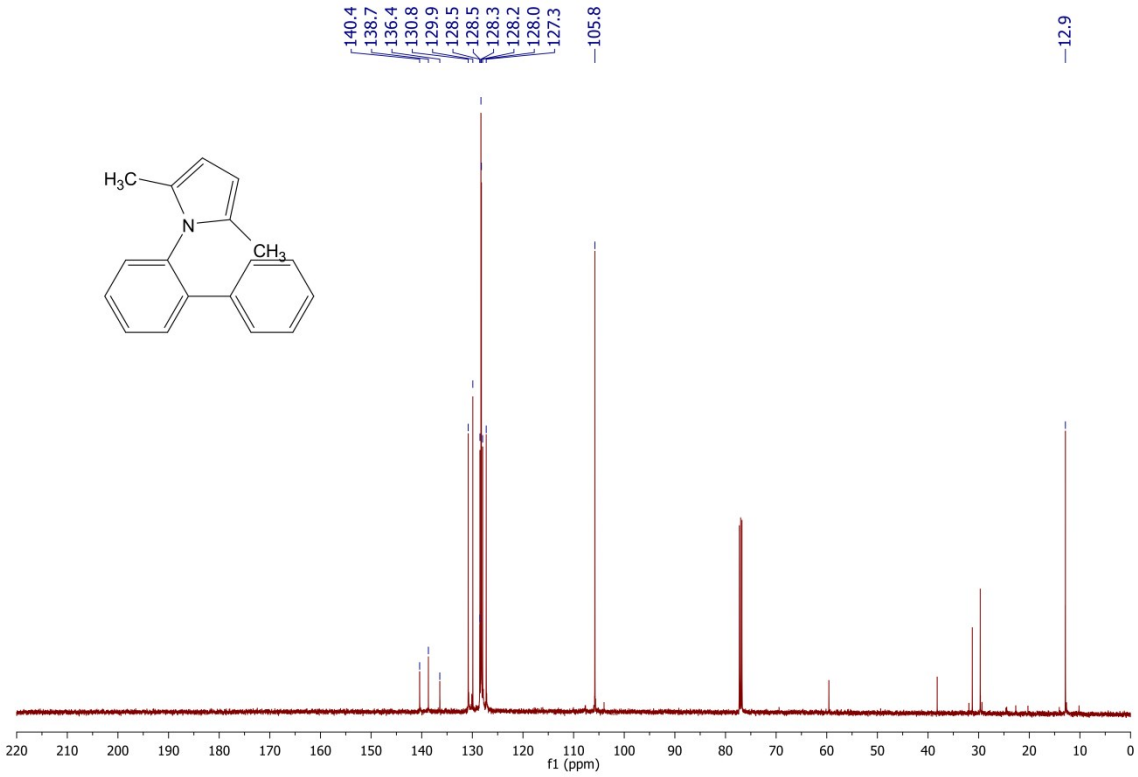


File :C:\GC-MS\2016\11.29.2016\4-IODOANILINE-AA-DES-SA-80-2H.D
Operator : THAO TRAN
Acquired : 29 Nov 2016 14:10 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Instrument : GCMSD
Sample Name: 4-IODOANILINE-AA-DES-SA-80-2H
Misc Info :
Vial Number: 2

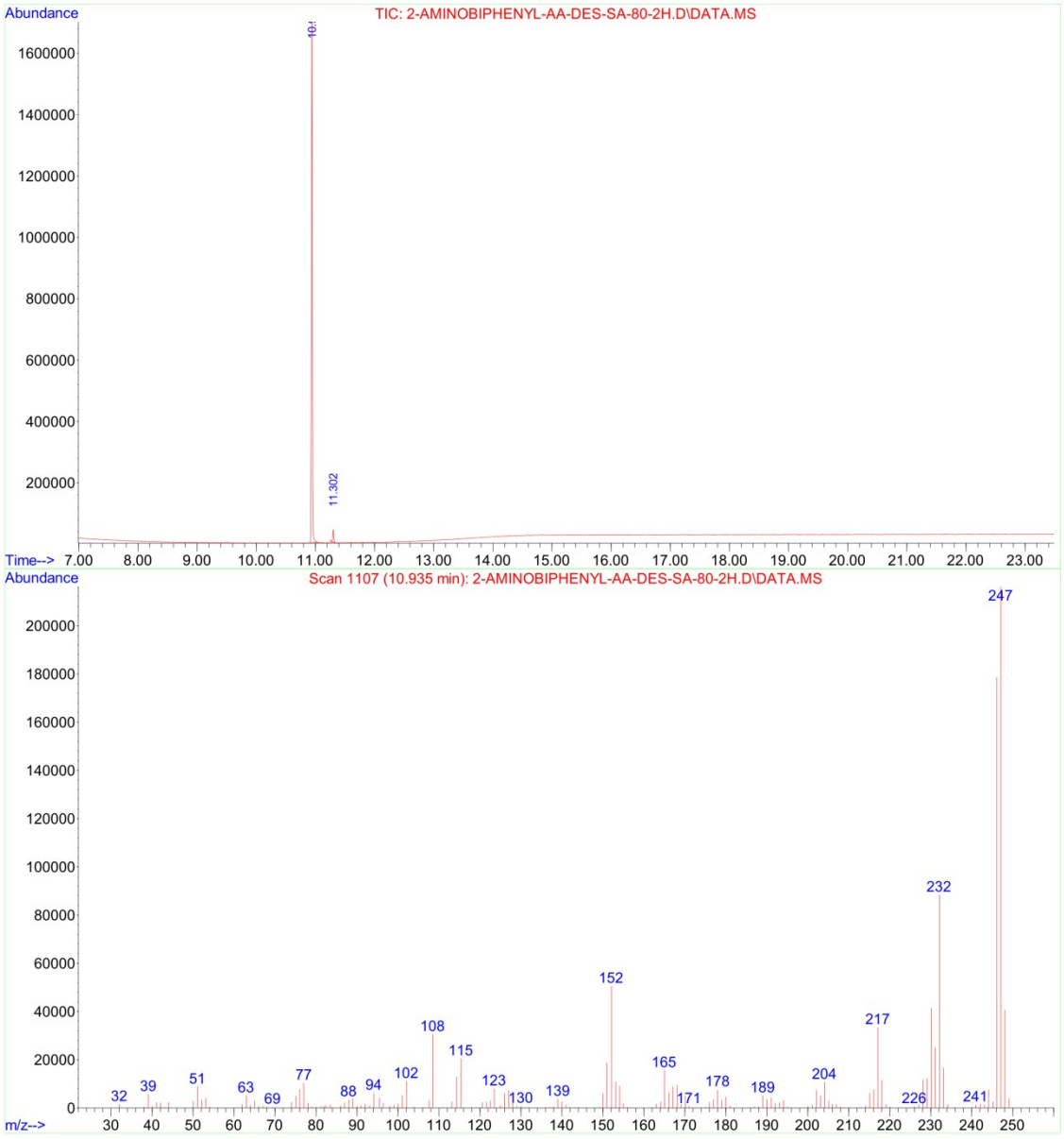


^{1}H NMR, ^{13}C NMR**, and GC-MS of 1-([1,1'-Biphenyl]-2-yl)-2,5-dimethyl-1*H*-pyrrole**

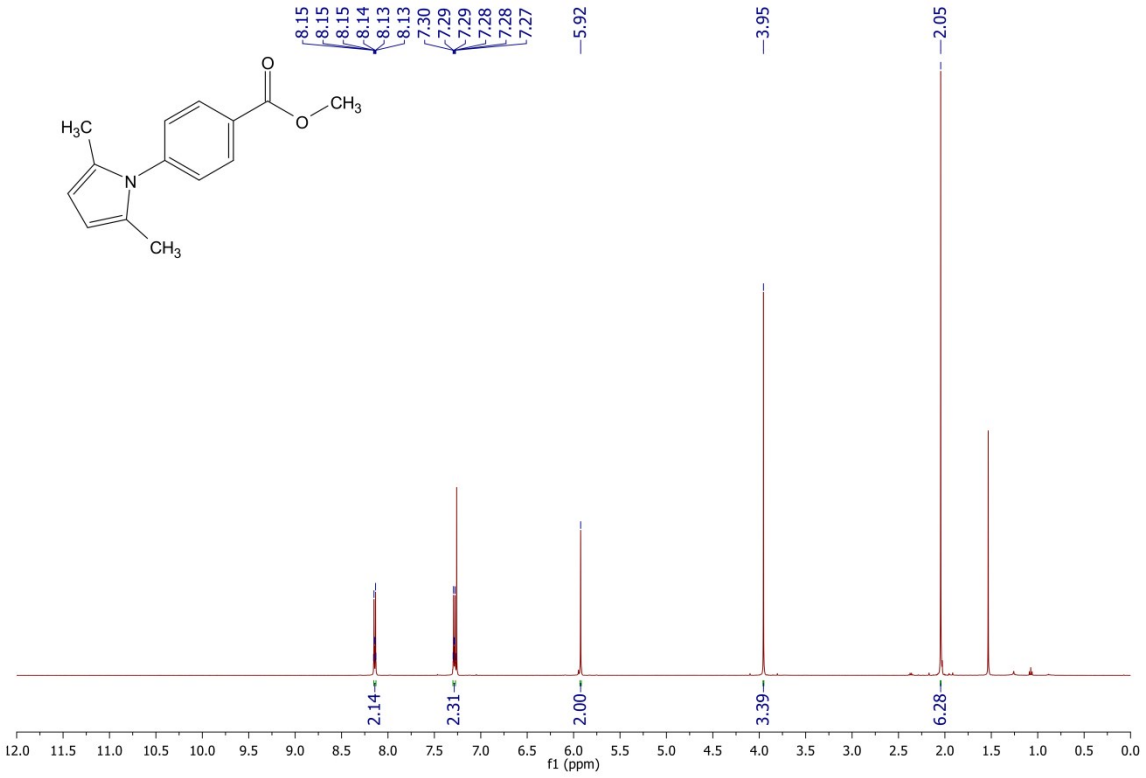


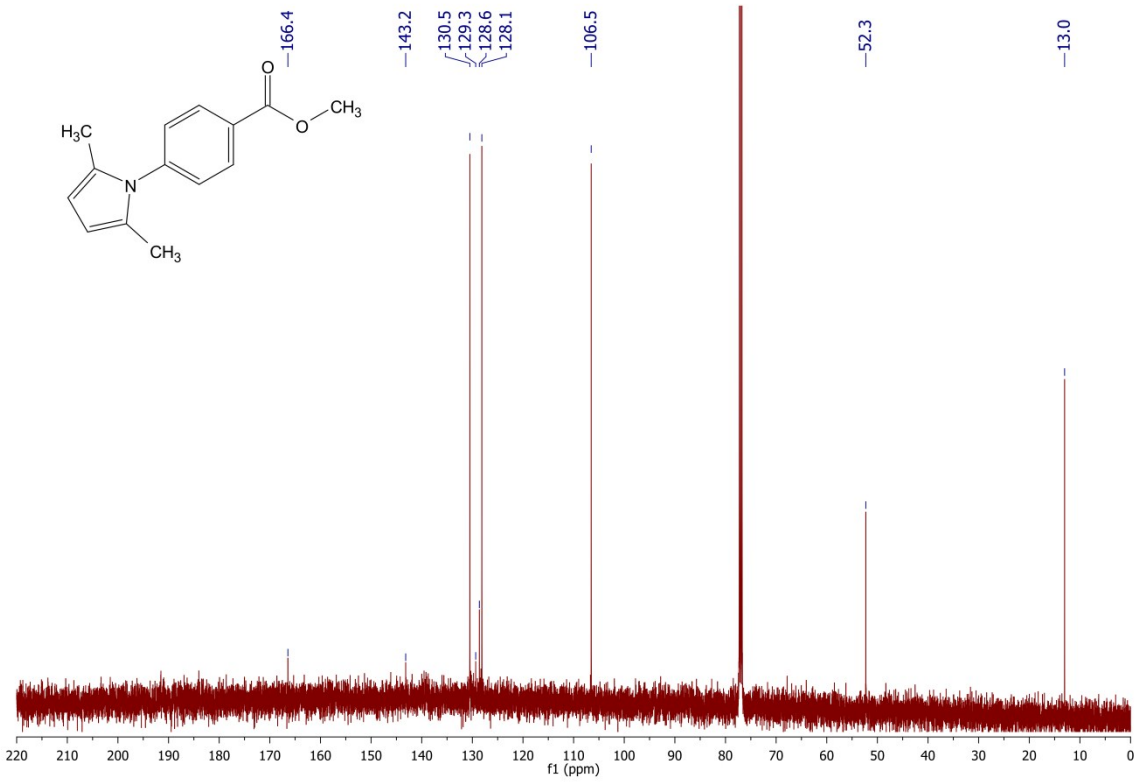


File :C:\GC-MS\2016\11.03.2016\2-AMINOBIPHENYL-AA-DES-SA-80-2H.D
Operator : TRUONG HAI
Acquired : 3 Nov 2016 14:21 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Instrument : GCMSD
Sample Name: 2-AMINOBIPHENYL-AA-DES-SA-80-2H
Misc Info :
Vial Number: 8

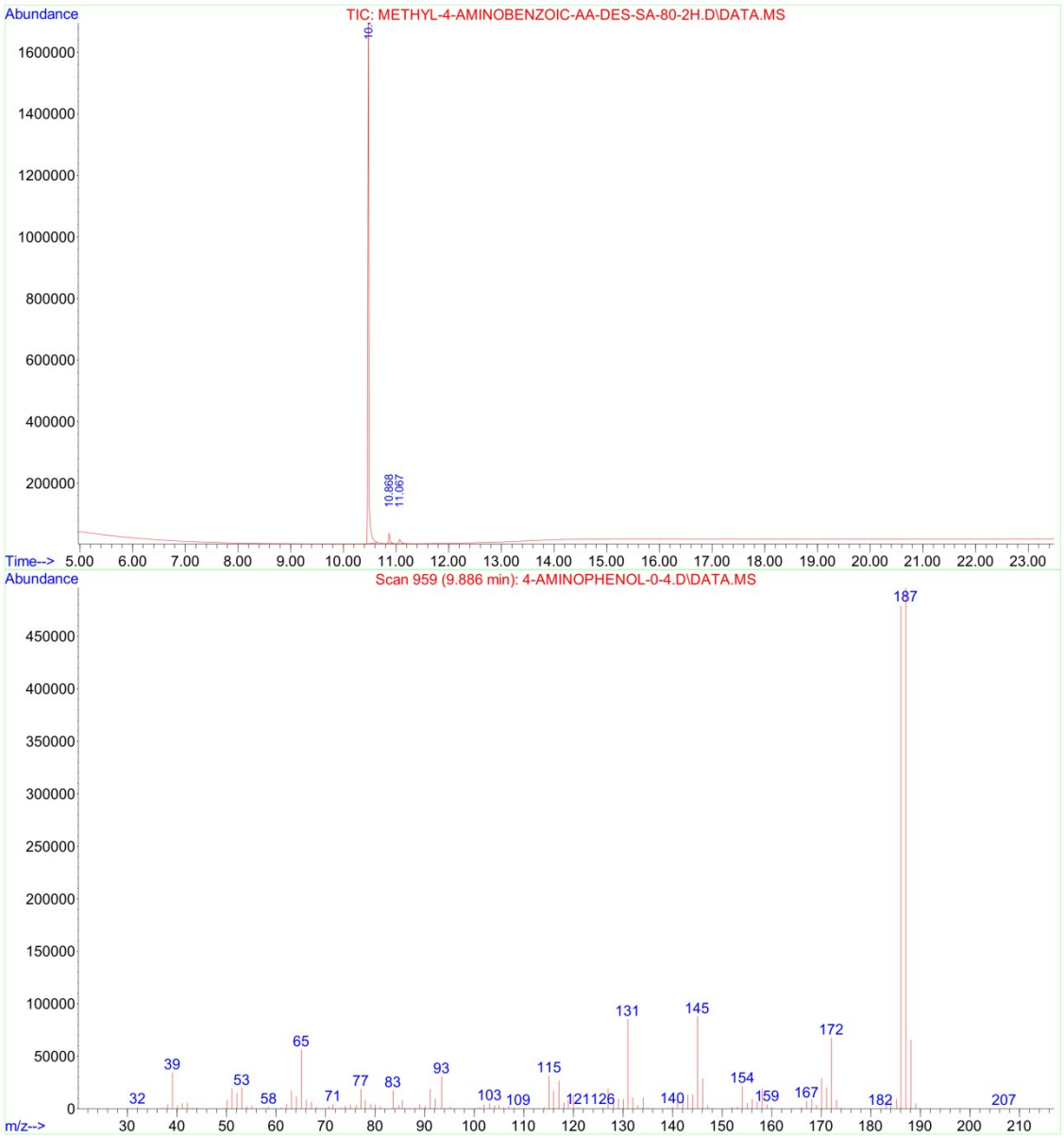


^{1}H NMR, ^{13}C NMR**, and GC-MS of methyl 4-(2,5-dimethyl-1*H*-pyrrol-1-yl)benzoate**

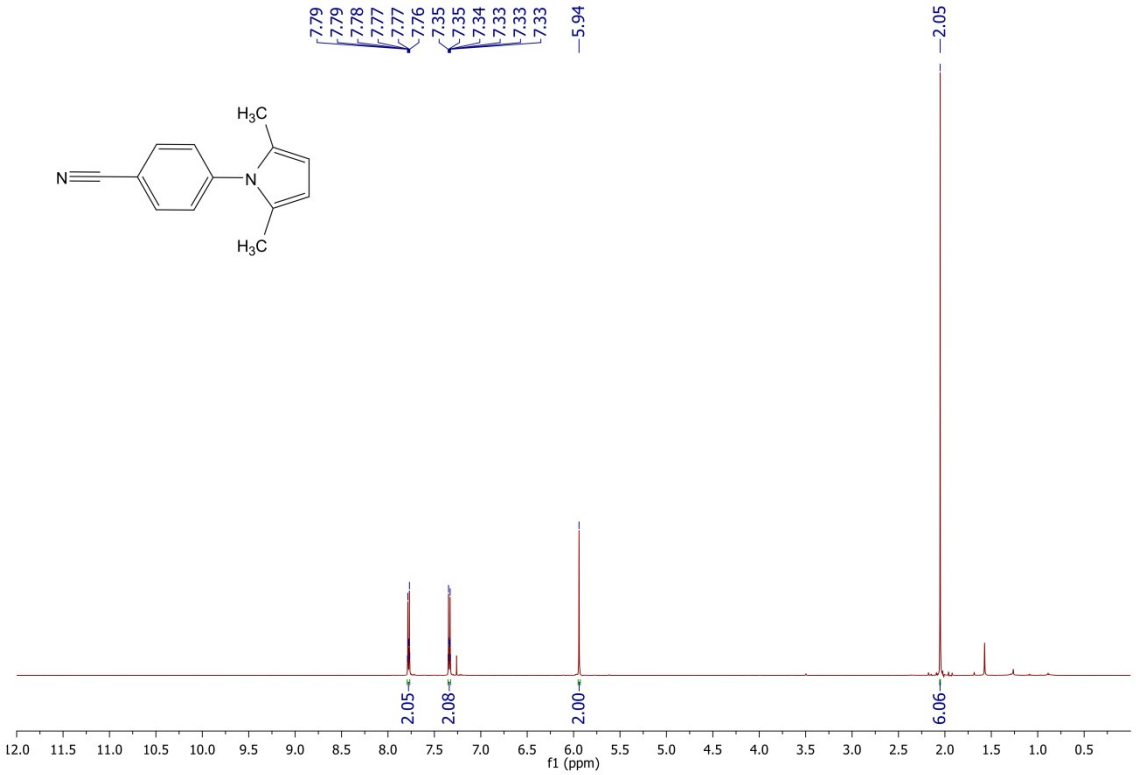


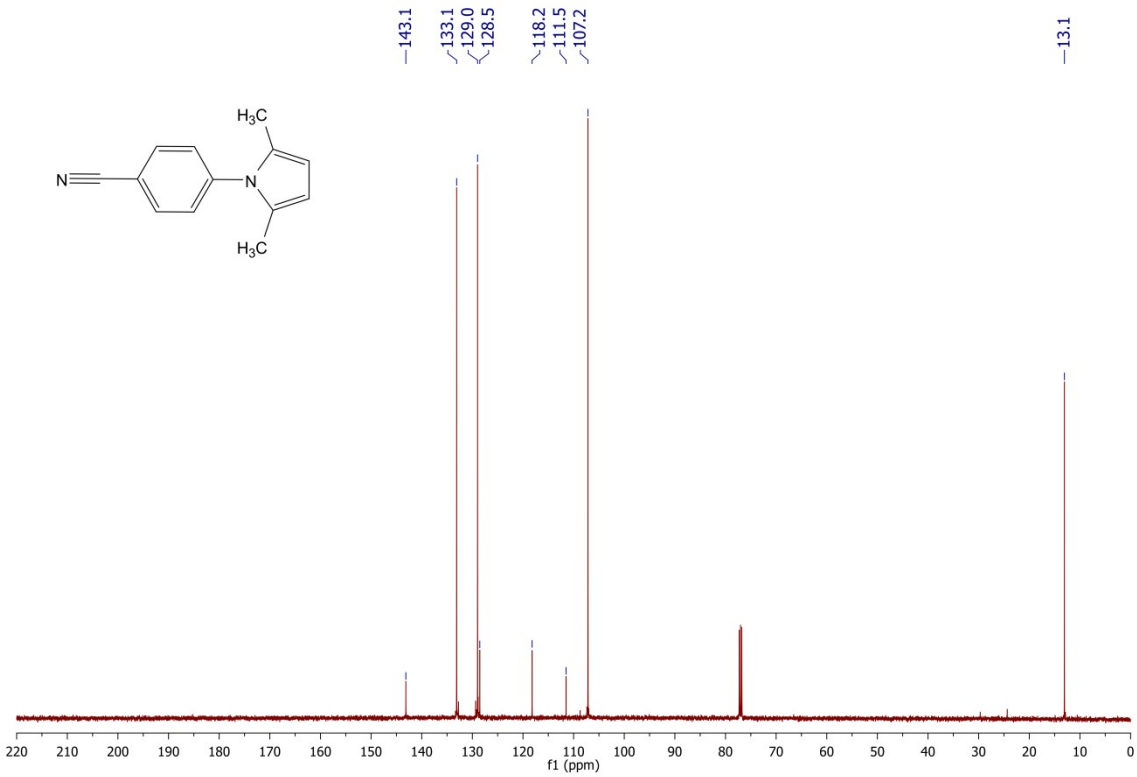


File :C:\GC-MS\2016\11.22.2016\METHYL-4-AMINOBENZOIC-AA-DES-SA-80-
... 2H.D
Operator : TRUONG HAI
Instrument : GCMSD
Acquired : 22 Nov 2016 15:13 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Sample Name: METHYL-4-AMINOBENZOIC-AA-DES-SA-80-2H
Misc Info :

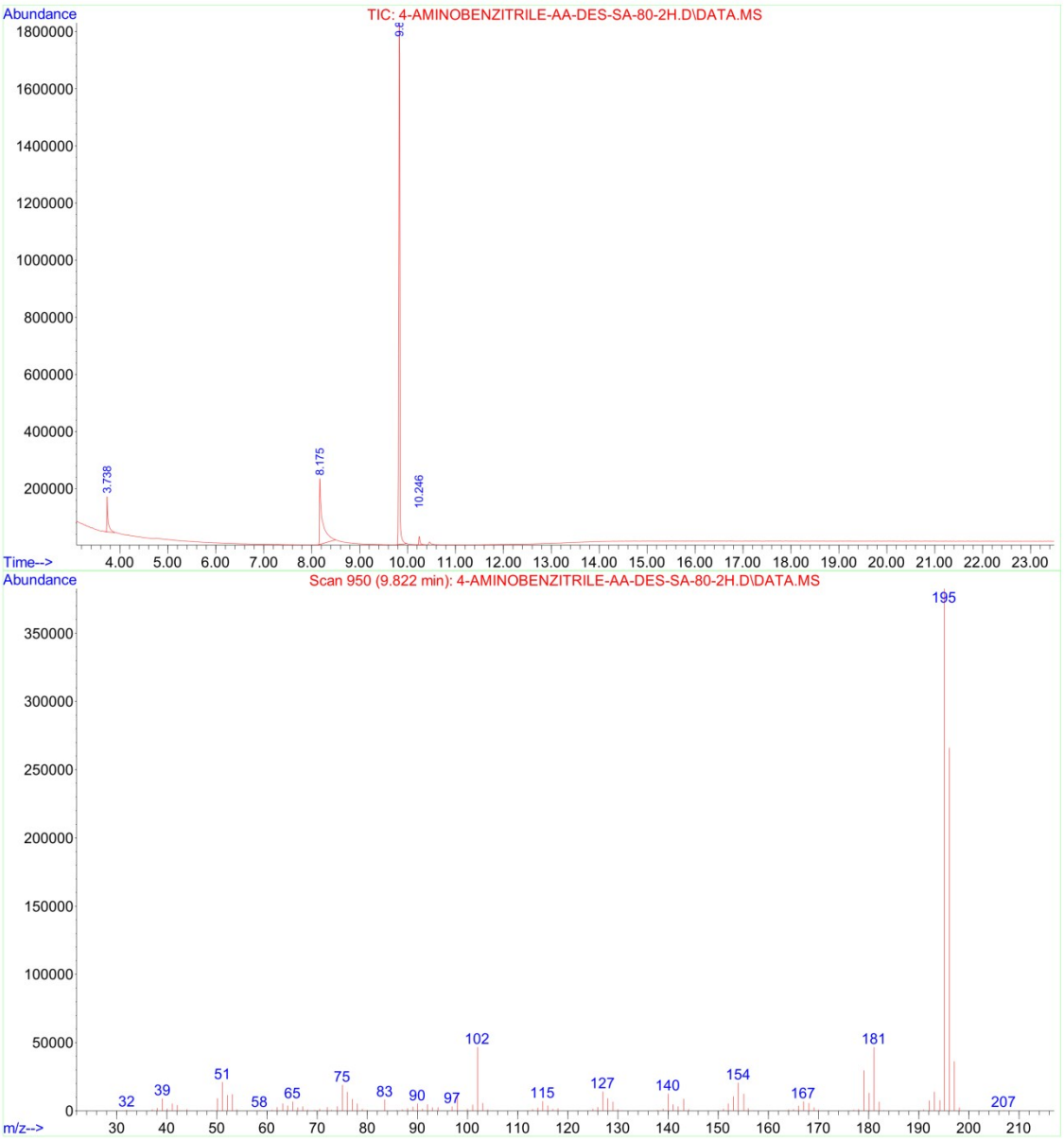


^{1}H NMR, ^{13}C NMR**, and GC-MS of 1-(4-cyanophenyl)-2,5-Dimethyl-1*H*-pyrrole**

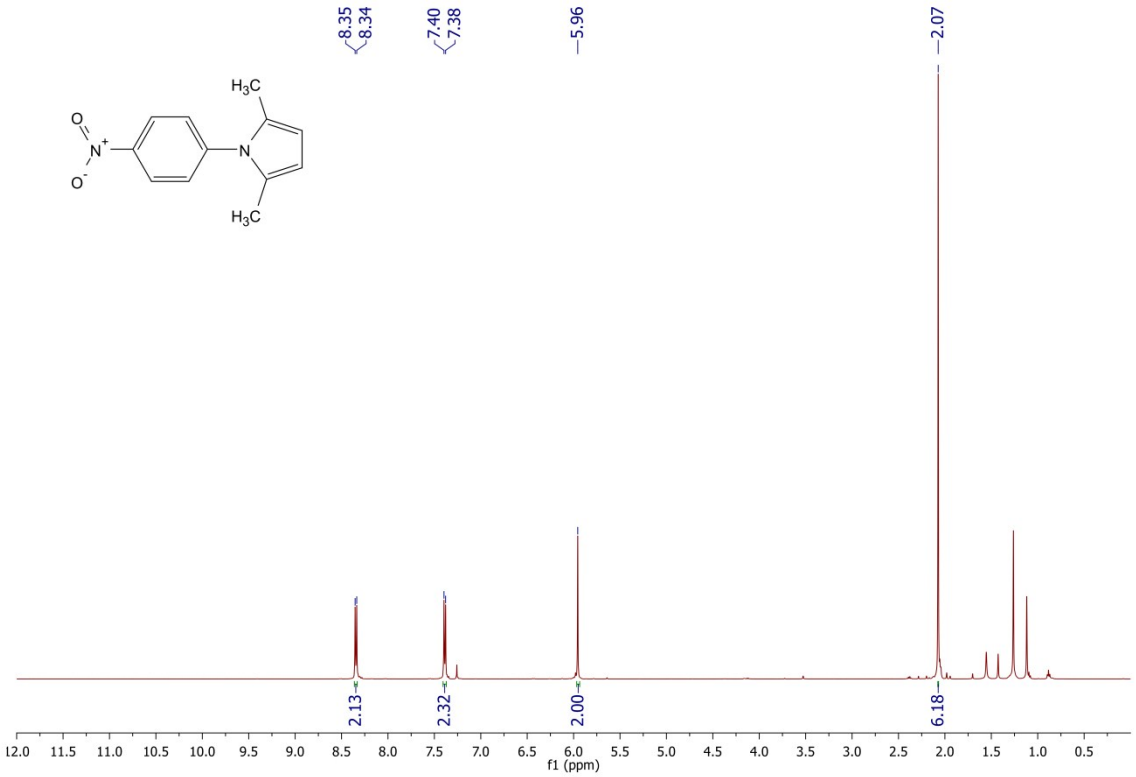


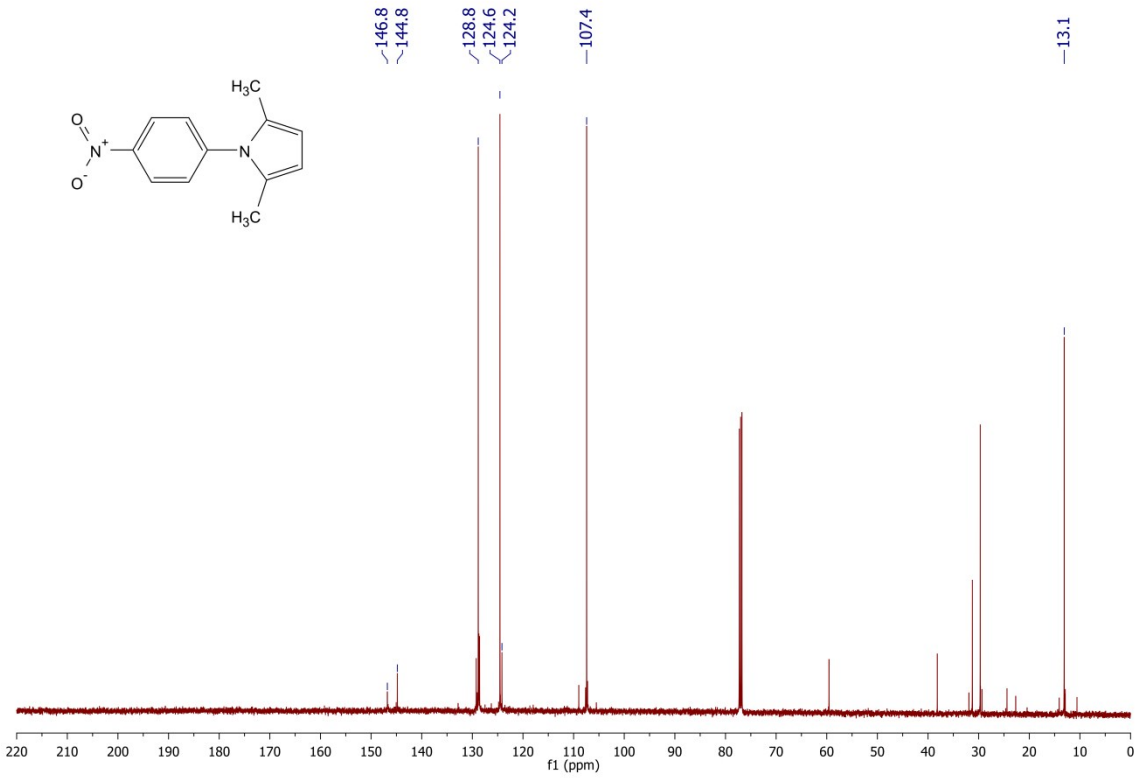


File :C:\GC-MS\2016\11.23.2016\4-AMINOBENZITRILE-AA-DES-SA-80-2H.D
Operator : TRUONG HAI
Acquired : 24 Nov 2016 8:50 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Instrument : GCMSD
Sample Name: 4-AMINOBENZITRILE-AA-DES-SA-80-2H
Misc Info :
Vial Number: 1

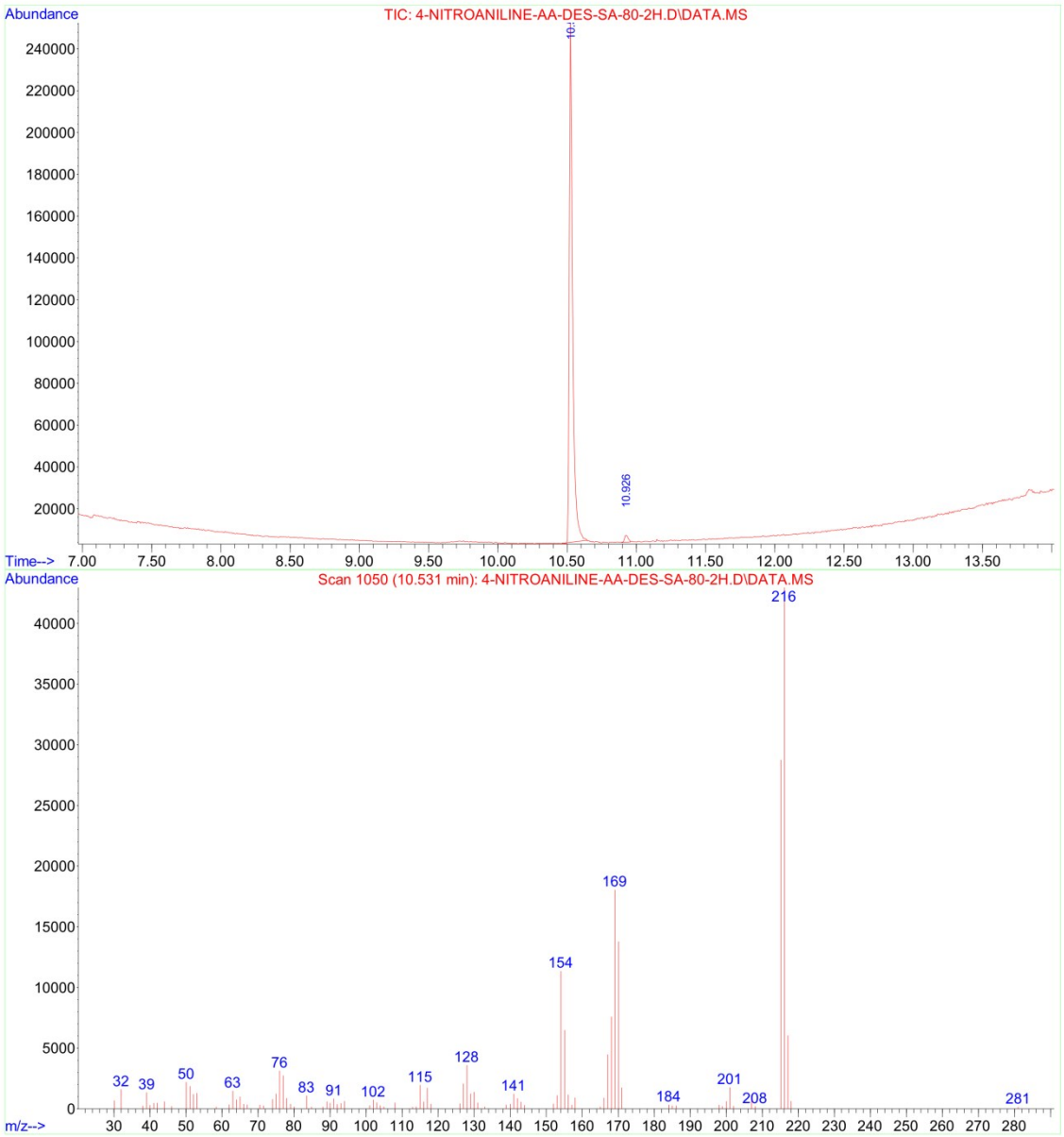


^{1}H NMR, ^{13}C NMR**, and GC-MS of 2,5-dimethyl-1-(4-nitrophenyl)-1*H*-pyrrole**

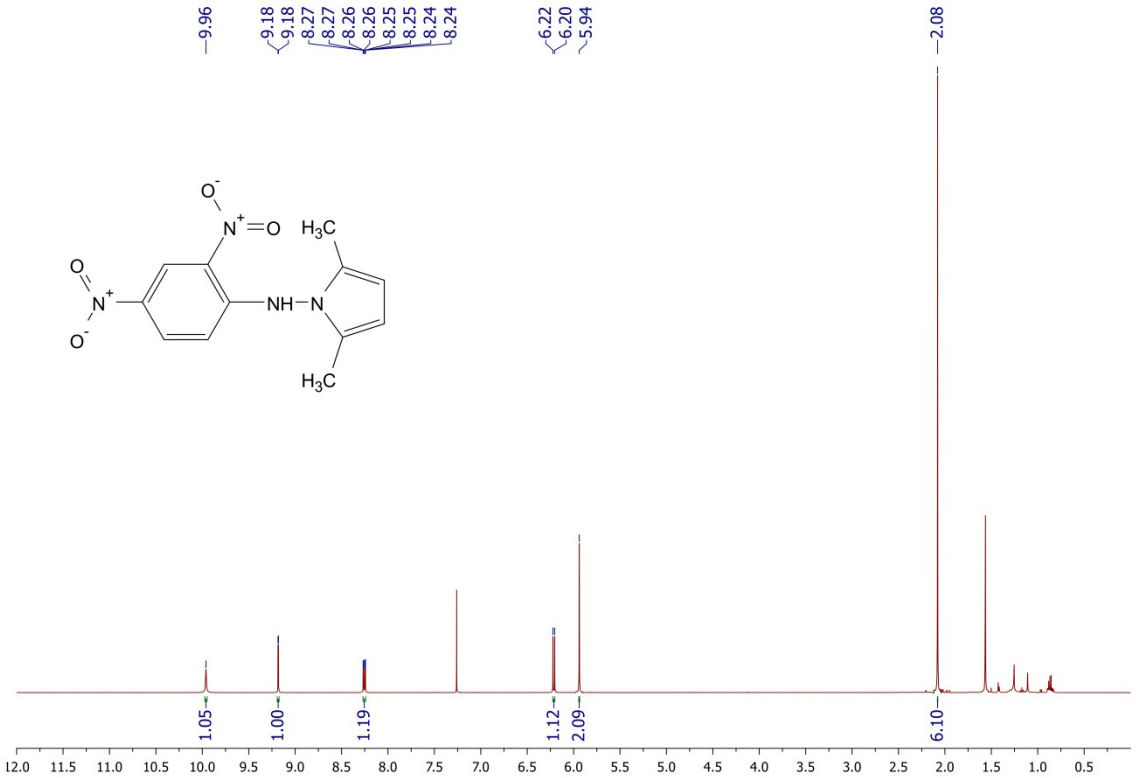


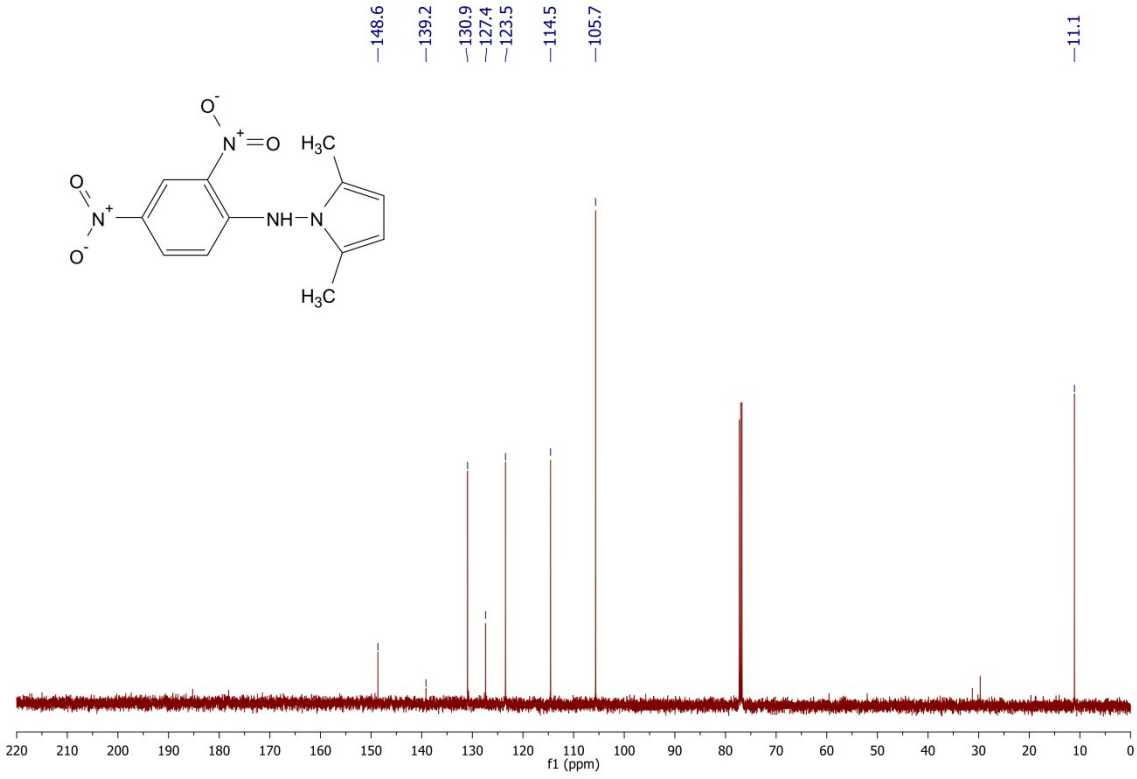


File :C:\GC-MS\2016\11.07.2016\4-NITROANILINE-AA-DES-SA-80-2H.D
Operator : Thao Tran
Acquired : 7 Nov 2016 18:27 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Instrument : GCMSD
Sample Name: 4-NITROANILINE-AA-DES-SA-80-2H
Misc Info :
Vial Number: 1

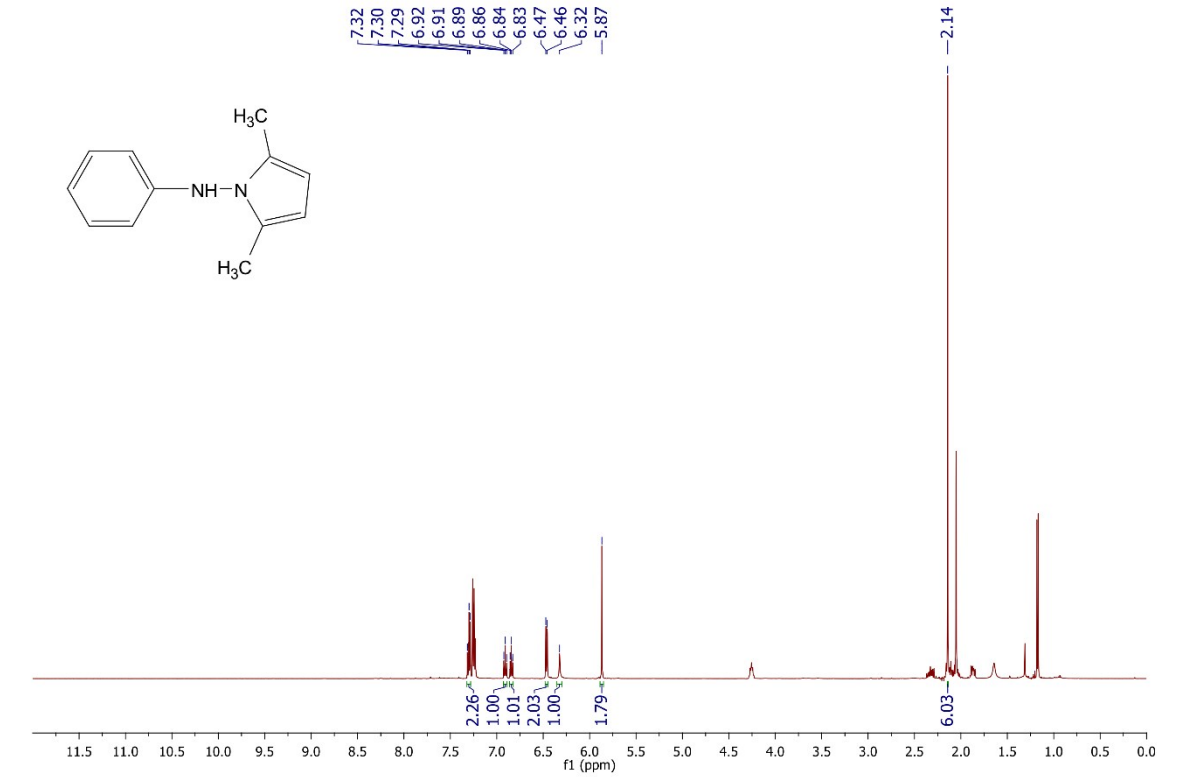


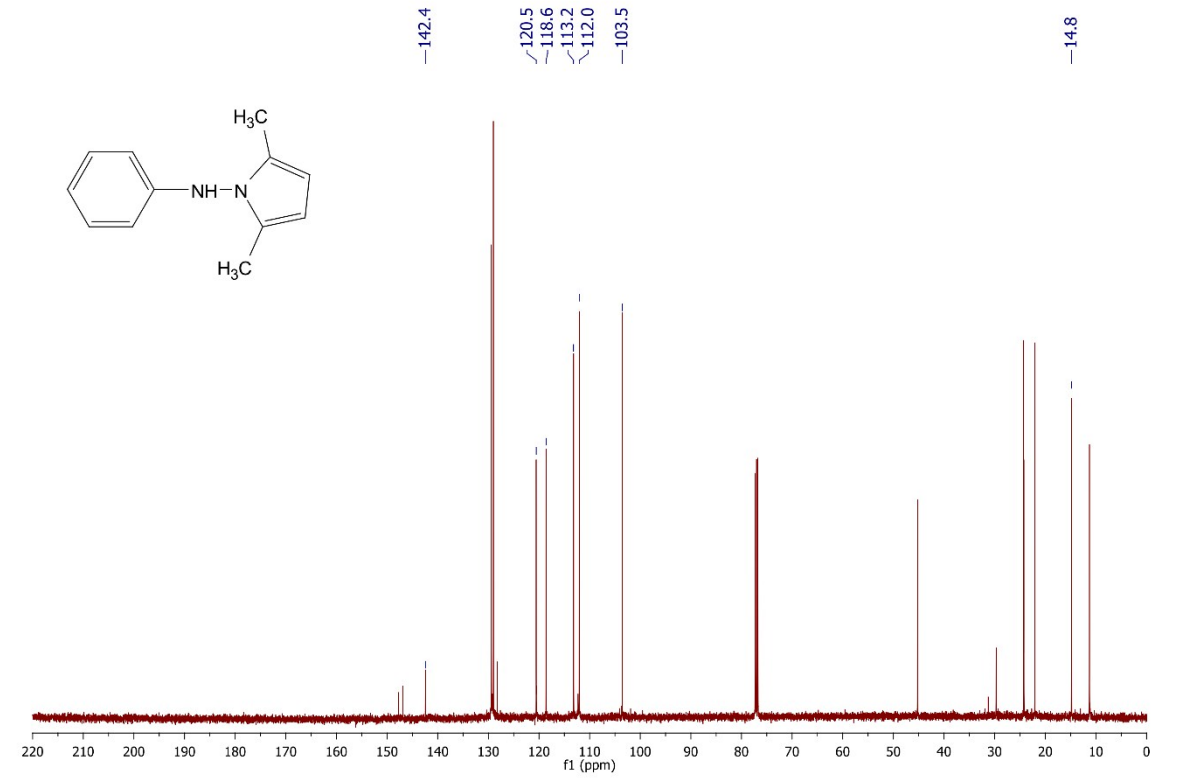
^{1}H NMR, ^{13}C NMR**, and GC-MS of *N*-(2,4-dinitrophenyl)-2,5-dimethyl-1*H*-pyrrol-1-amine**



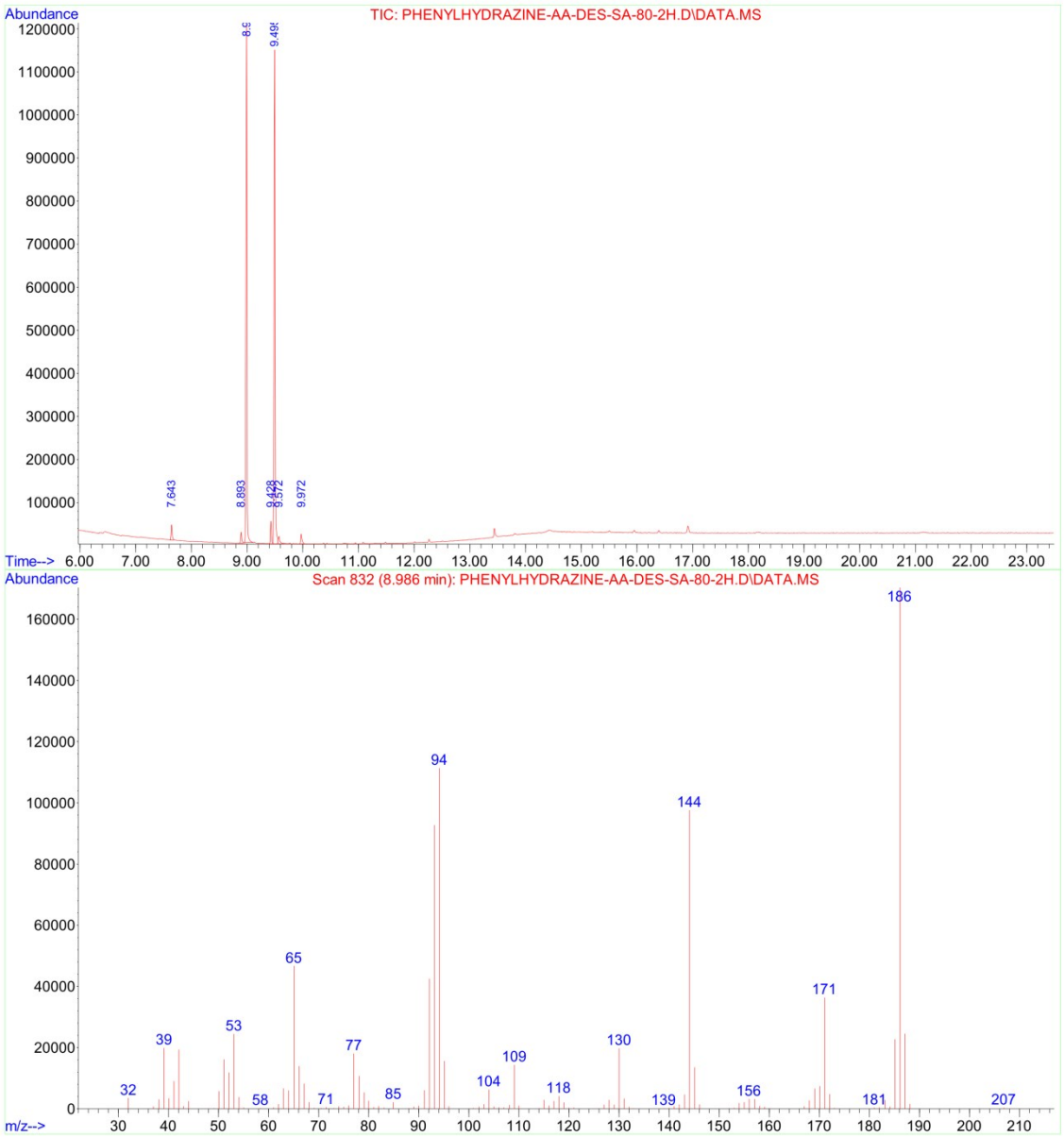


^{1}H NMR, ^{13}C NMR**, and GC-MS of 2,5-dimethyl-*N*-phenyl-1*H*-pyrrol-1-amine**

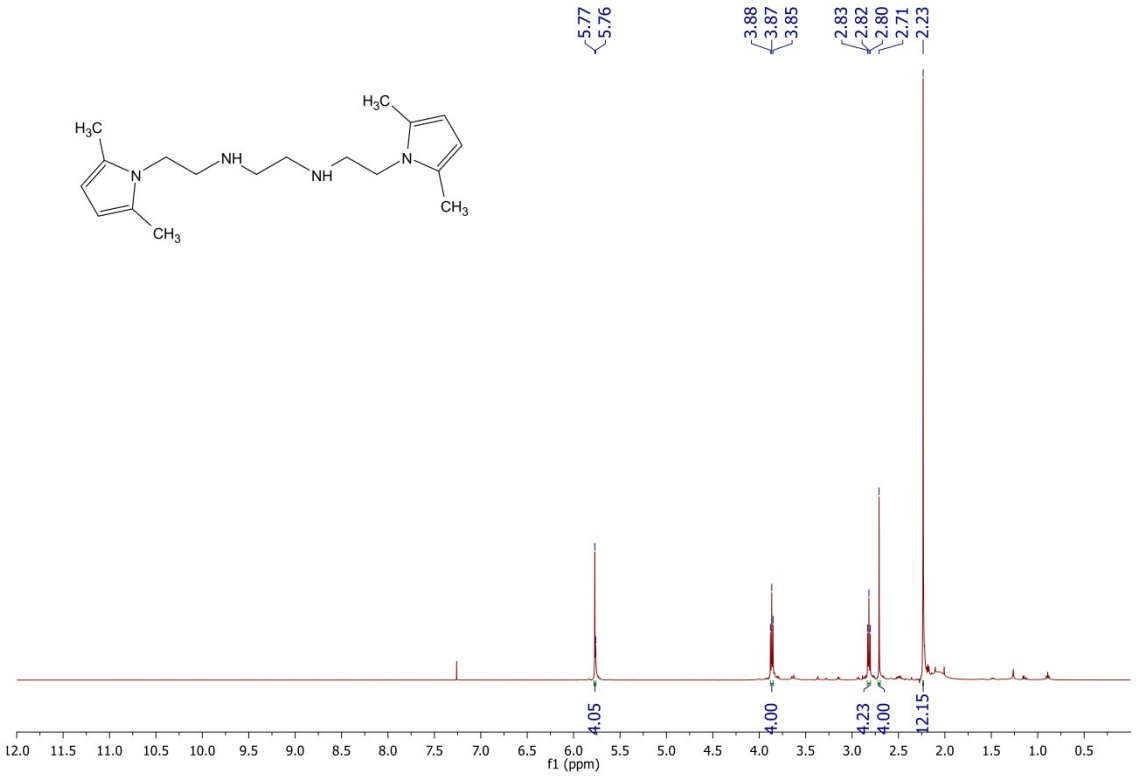


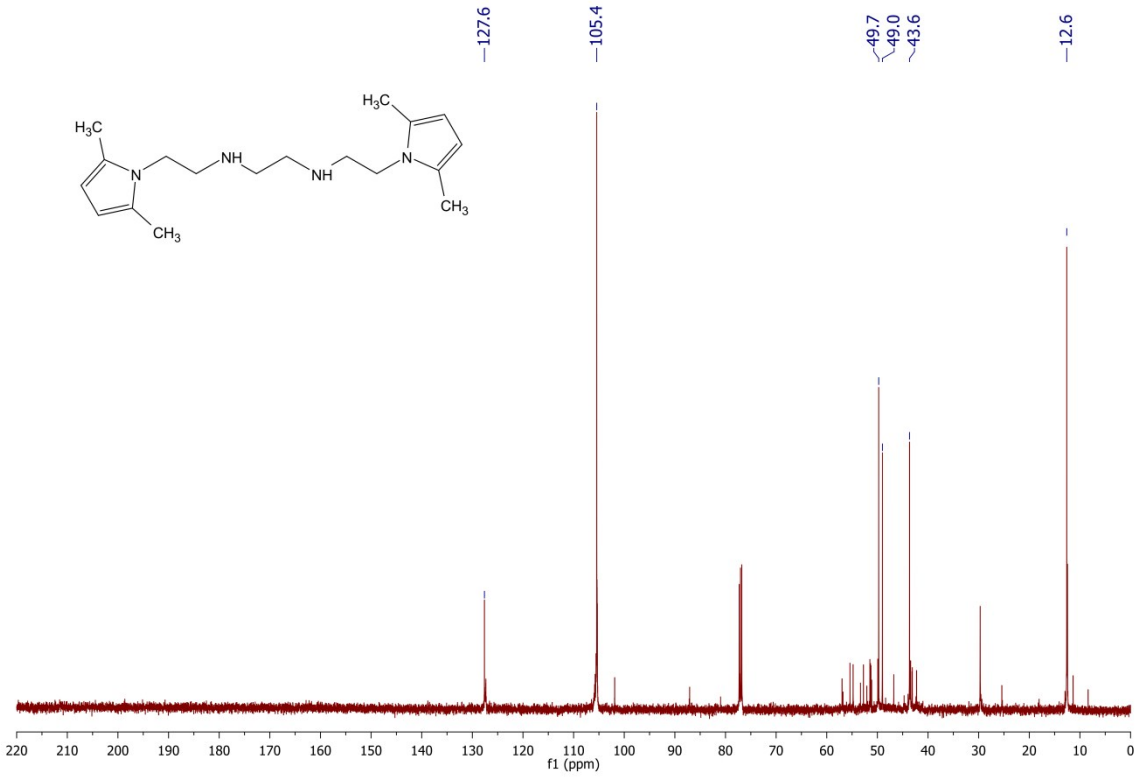


File :C:\GC-MS\2016\11.13.2016\PHENYLHYDRAZINE-AA-DES-SA-80-2H.D
Operator : TRUONG HAI
Acquired : 13 Nov 2016 12:51 using AcqMethod ACYLATION-SHORT-DELAY-3MIN.M
Instrument : GCMSD
Sample Name: PHENYLHYDRAZINE-AA-DES-SA-80-2H
Misc Info :
Vial Number: 3



^{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**





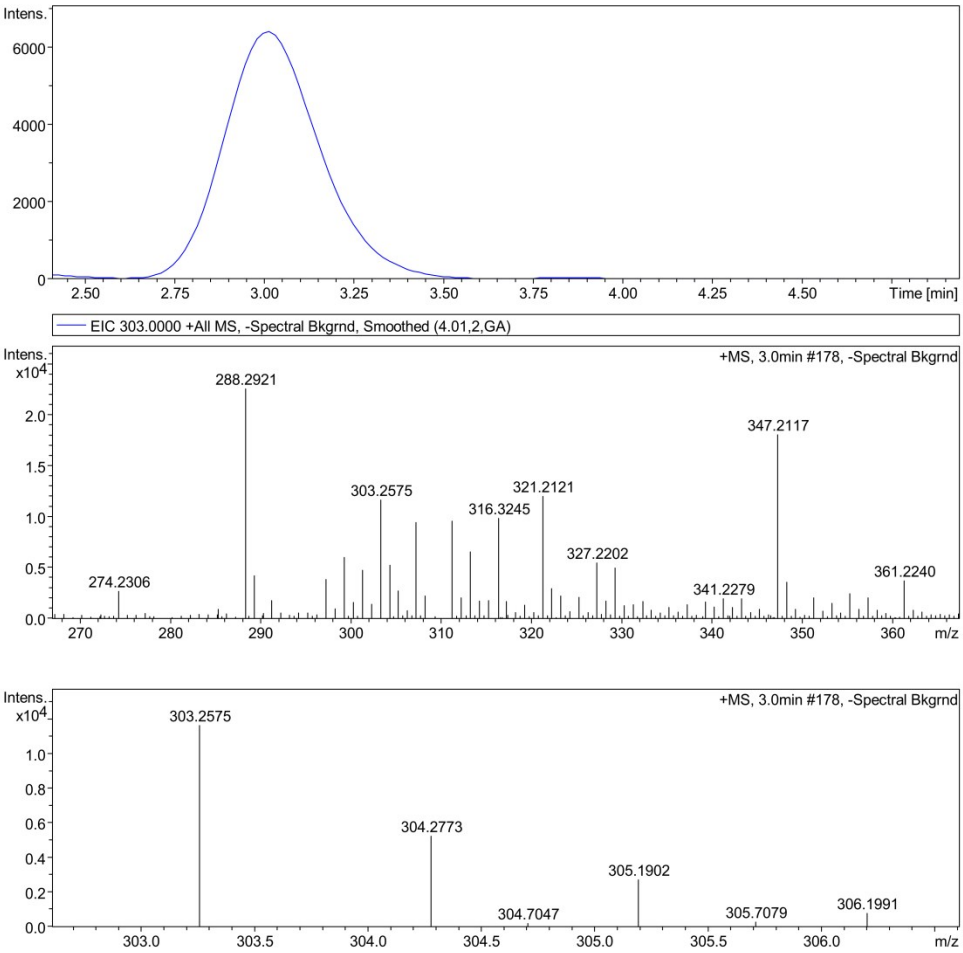
Display Report

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



Section S3. References

1. K. Aghapoor, L. Ebadi-Nia, F. Mohsenzadeh, M. Mohebi Morad, Y. Balavar and H. R. Darabi, *J. Organomet. Chem.*, 2012, **708–709**, 25-30.
2. K. A. Hossein Reza Darabi, Abbas Darestani Farahani, Farshid Mohsenzadeh, *Environ. Chem. Lett.*, 2012, **10**, 369-375.
3. H. Cho, R. Madden, B. Nisanci and B. Torok, *Green Chem.*, 2015, **17**, 1088-1099.
4. F. M. Kioumars Aghapoor, Hossein Reza Darabi, Hani Sayahi, Yadollah Balavar, *Res. Chem. Intermed.*, 2016, **42**, 407-415.
5. M. R. P. Hossein Reza Darabi, Kioumars Aghapoor, Asyeh Mirzaee, Farshid Mohsenzadeh, Nazafarin Asadollahnejad, Hossein Taherzadeh, Yadollah Balavar, *Environ. Chem. Lett.*, 2012, **10**, 5–12.
6. S. S. Bimal K. Banik , Indrani Banik, *J. Org. Chem.*, 2004, **69**, 213-216.
7. A. Rahmatpour, *J. Organomet. Chem.*, 2012, **712**, 15-19.
8. M. A. V. R. d. S. Ana Filipa L. O. M. Santos, *Struct. Chem.*, 2013, **24**, 1981-1992.
9. S. J. H. Hazlewood, Gordon K.; Lions, Francis; Baldick, Kenric J.; Cornforth, John W.; Graves, John N.; Maunsell, James J.; Wilkinson, Thomas; Birch, Arthur J.; Harradence, Rita H., *Journal and Proceedings of the Royal Society of New South Wales*, 1937, **71**, 92-102.
10. P. Grammaticakis, *Comptes Rendus des Seances de l'Academie des Sciences, Serie C: Sciences Chimiques* 1971, **272**, 1574-1547.
11. H. A. Akio Ohsawa, Hiroshi Igeta, Toshio Akimoto, Akio Tsuji, Yoichi Iitaka, *J. Org. Chem.*, 1979, **44**, 3524–3529.
12. S. S. Haifeng Duan, Jeffrey L. Petersen, Novruz G. Akhmedov, Xiaodong Shi, *J. Am. Chem. Soc.*, 2009, **131**, 12100–12102.
13. P.-Y. J. Ming-Zhong Zhang, Yu-Feng Liu, and Can-Cheng Guo, *J. Org. Chem.*, 2015, **80**, 10777–10786.
14. T. D. Binns and R. Brettle, *Journal of the Chemical Society C: Organic*, 1966, DOI: 10.1039/J39660000341, 341-343.
15. T. Zsolnai, *Biochem. Pharmacol.*, 1961, **5**, 387-304.

16. K. N. D. Zelenin, J., *Zhurnal Organicheskoi Khimii* 1973, **9**, 1295-1304.
17. J. Chen, H. Wu, Z. Zheng, C. Jin, X. Zhang and W. Su, *Tetrahedron Lett.*, 2006, **47**, 5383-5387.
18. Liudvikas Akelis, Jolanta Rousseau, Robertas Juskenas, Jelena Dodonova, Cyril Rousseau, Stéphane Menuel, Dominique Prevost, Sigitas Tumkevičius, Eric Monflier and F. Hapiot, *Eur. J. Org. Chem.*, 2015, **2016**, 31-35.
19. S. Handy and K. Lavender, *Tetrahedron Lett.*, 2013, **54**, 4377-4379.
20. M. Curini, F. Montanari, O. Rosati, E. Lioy and R. Margarita, *Tetrahedron Lett.*, 2003, **44**, 3923-3925.
21. H. M. Abbas Ali Jafari, *Environ. Chem. Lett.*, 2013, **11**, 157-162.
22. X. Chen, M. Yang and M. Zhou, *Tetrahedron Letters*, 2016, **57**, 5215-5218.
23. S. K. Amit Walia, Richard B Silverman, *J. Org. Chem.*, 2013, **78**, 10931–10937.
24. K. M. Noberini R, Peddibhotla S, Dahl R, Su Y, Cosford ND, Roth GP, Pasquale EB., *J Biol Chem.*, 2008, **283**, 29461-29472.
25. P. D. A. Marina Porcelloni, Cristina Rossi, Alessandro Sisto, Alessandro Ettorre, Andrea Madami, Maria Altamura, Sandro Giuliani, Stefania Meini, Daniela Fattori, *ChemMedChem*, 2008, **3**, 1048–1060.