Electronic Supplementary Information (ESI)

Green Electrochemical Strategy for One-step Synthesis of New Catechol Derivatives

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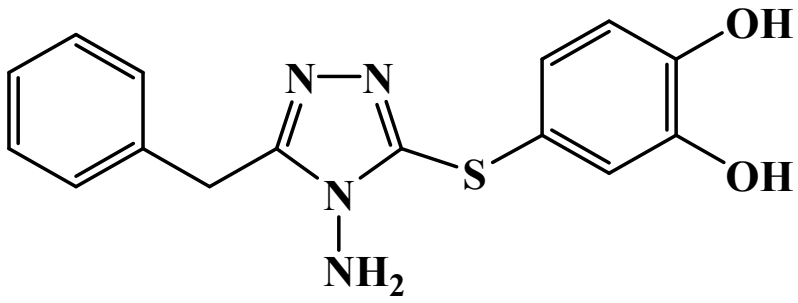
Spectroscopic characterizations of the electro- synthesized products (6, 7, and 8):

After purifications, all the electrochemical synthesized organic products were characterized by using FT-IR (Shimadzu FT-IR 8101 PC). ^{1}H and ^{13}C NMR spectra were recorded in DMSO-d6 at 25 ^{o}C using a 300 MHz and 75 MHz Varian Mercury VX "NMR300" spectrometer, respectively. All chemical shifts for NMR spectra were measured in form of ppm with δ units relative to TMS in DMSO-d6 as an internal standard. Electron impact mass spectra were obtained at 70 eV using a GCMS-QP 1000 EX Shimadzu spectrometer. Both elemental analysis using elemental C,H,N,S Analyzer- Vario EL III Germany, mass (MS) and NMR spectra were carried out at the Microanalytical Center of Cairo University. Melting points (uncorrected) were recorded on melting point apparatus SMP 10. The results indicate that the formed electro-synthesized organic products are mainly depending on the nature of the nucleophilic reagent (3, 4, or 5) used. The spectroscopic data of the electrosynthesized products 6, 7 or 8 via Michael addition reaction are summarized as the following:

Product number 6:

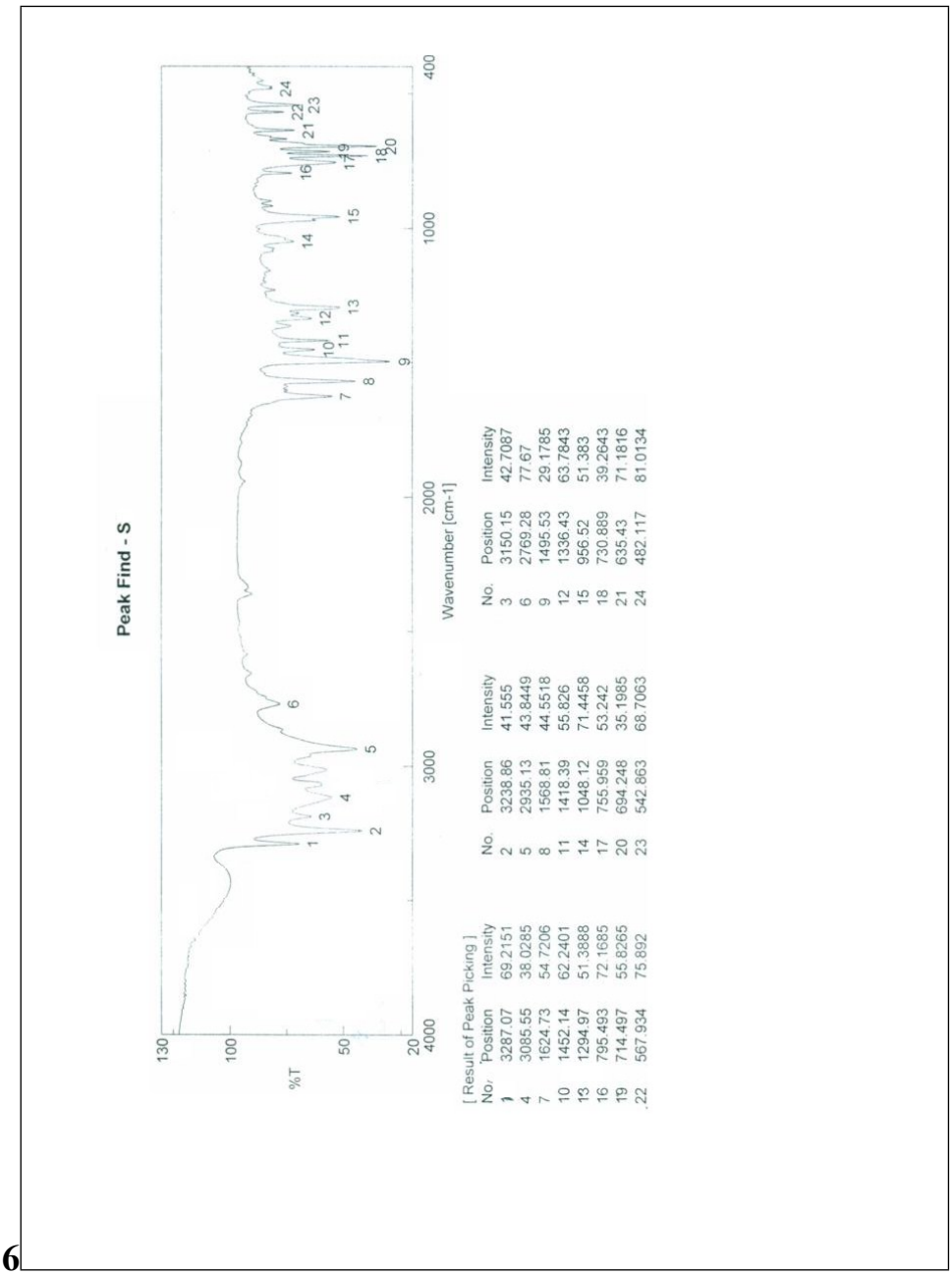
Name: 4-(4-Amino-5-benzyl-4H-[1,2,4]triazol-3-ylsulfanyl)-benzene-1,2-diol

Data: Yellowish white crystals Yield: 0.26 g, 82.8 %, mp: 159 oC. IR spectrum (KBr) , ν, cm-1: 3400 (br. OH), 3287, 3238 (NH_2), 3085 (CH-aromatic), 2935 (CH_2), 1624 (C=N); ^{1}H NMR (DMSO-d6) (δ ppm): 9.4 (br.s, 1H, OH), 9.1 (s, 1H, OH), 7.36-7.21 (m, 8H, Ar'H), 5.54 (s, 2H, NH_2), 4.02 (s, 2H, CH_2); ^{13}C NMR spectrum (75 MHz, DMSO-d6) (δ, ppm): 28.55 (CH_2), 115.27, 120.72, 127.05, 127.19, 128.58, 128.88, 134.00, 134.12 (Ar-C), 144.01 (Ar-OH), 146.95 (Ar-OH), 151.53 (C=N), 160.91 (C=N); Ms: m/z 314 (M^+). Anal. Calcd for C15H14N4O2S; C, 57.31; H, 4.49; N, 17.82; S, 10.20. Found: C, 57.92; H, 4.71; N, 17.16; S, 10.03.



6

6



[Result of Peak Picking]

No.	Position	Intensity	No.	Position	Intensity	No.	Position	Intensity
1	3287.07	69.2151	2	3238.86	41.555	3	3150.15	42.7087
4	3085.55	38.0285	5	2935.13	43.8449	6	2769.28	77.67
7	1624.73	54.7206	8	1568.81	44.5518	9	1495.53	29.1785
10	1452.14	62.2401	11	1418.39	55.826	12	1336.43	63.7843
13	1294.97	51.3888	14	1048.12	71.4458	15	956.52	51.383
16	795.493	72.1685	17	755.959	53.242	18	730.889	39.2643
19	714.497	55.8265	20	694.248	35.1985	21	635.43	71.1816
22	567.934	75.892	23	542.863	68.7063	24	482.117	81.0134

FT-IR results for the product number **6.**

Cairo University
Micro Analytical Center

DI Analysis
Shimadzu Qp-2010 Plus

Sample Information

Analyzed by	: Mai Younis
Analyzed	: 10/09/2014 04:47:44 م
Sample Name	: S1
Sample ID	:
Customer Name	: Dr.Ahmed Mohamed Abo Bakr - El-wadi South
Data File	: C:\GCMSsolution\Data\Project1\B32.QGD
Org Data File	: C:\GCMSsolution\Data\Project1\B32.QGD
Method File	: C:\GCMSsolution\Data\Project1\A.GABR.qgm
Org Method File	: C:\GCMSsolution\Data\Project1\A.GABR.qgm
Report File	:
Tuning File	: C:\GCMSsolution\System\Tune1_default1.qgt
$Endiff$Modified by	: Mai Younis
Modified	: 10/09/2014 04:51:12 م

Method

Analytical Line 1

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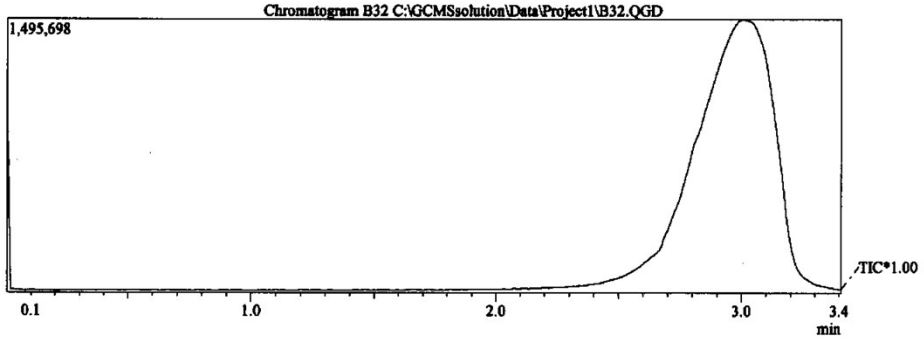
Start Time	:0.00min
End Time	:10.00min
ACQ Mode	:Scan
Event Time	:0.50sec
Scan Speed	:1428
Start m/z	:50.00
End m/z	:700.00
Electron Voltage	: 70 eV
Ionization Mode	: EI

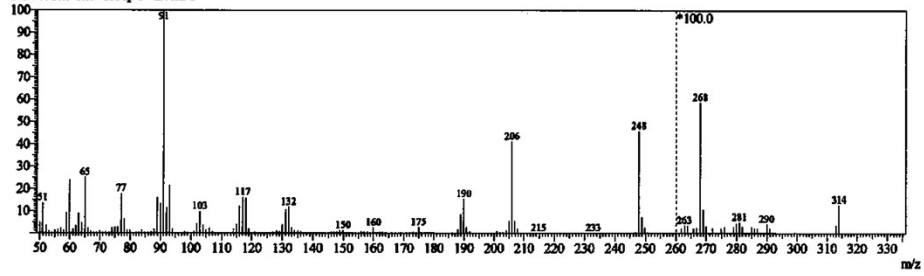
C:\GCMSsolution\Data\Project1\B32.QGD

Chromatogram B32 C:\GCMSsolution\Data\Project1\B32.QGD



Spectrum

Line#:1 R.Time:3.0(Scan#:364)
MassPeaks:225
RawMode:Single 3.0(364) BasePeak:91(246006)
BG Mode:None Group 1 - Event 1

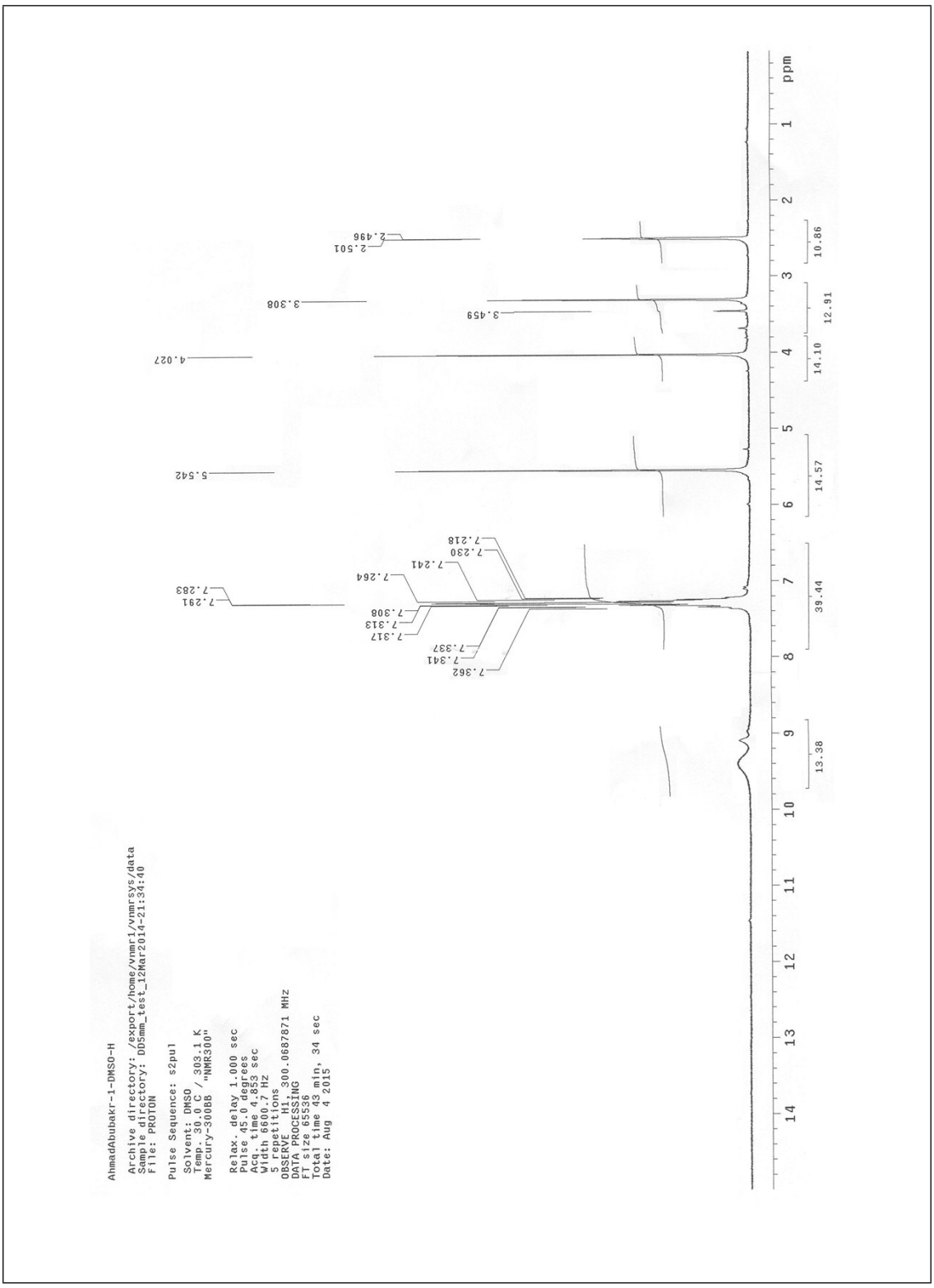


Mass Table

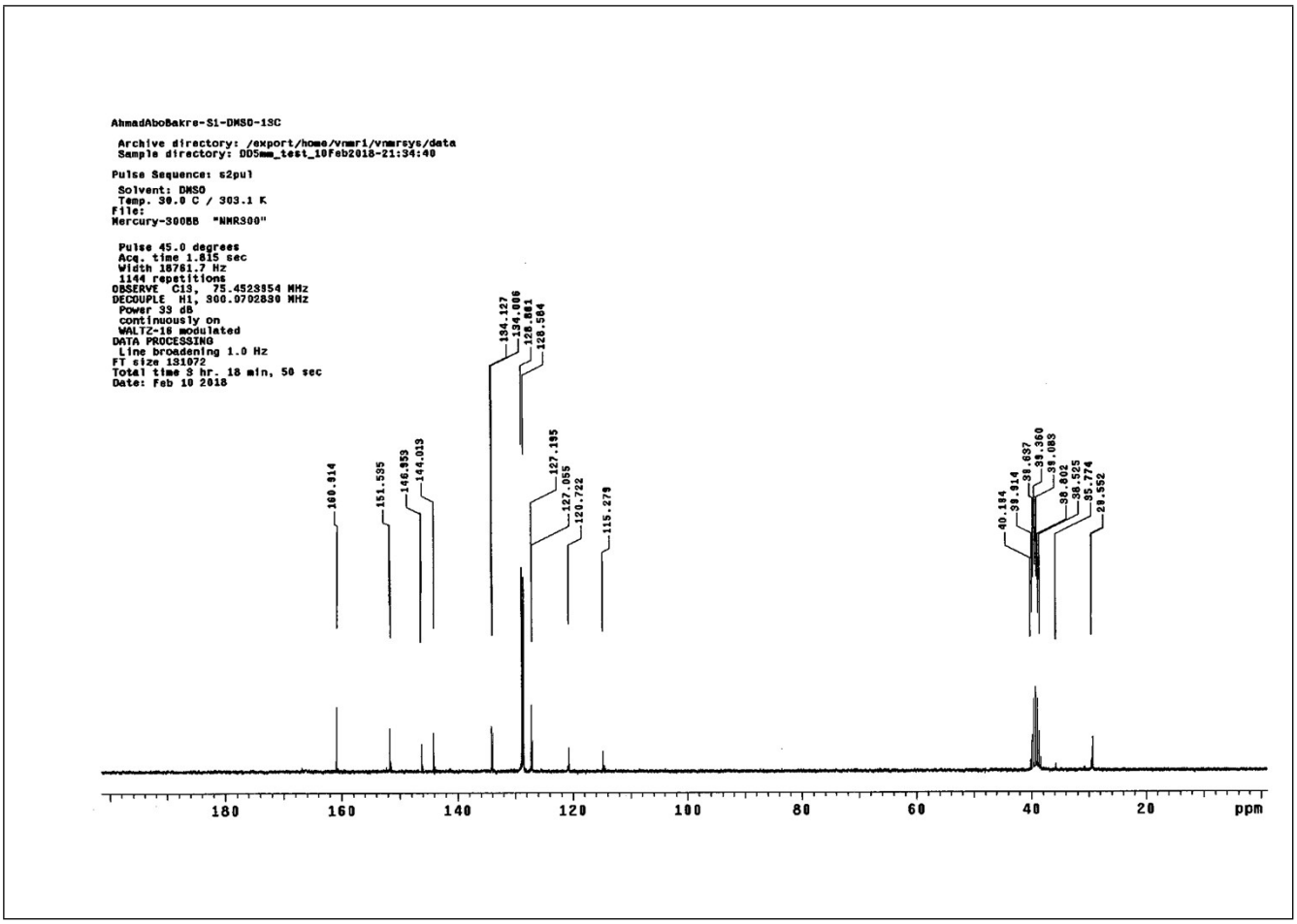
Line#:1 R.Time:3.0(Scan#:364)
MassPeaks:225
RawMode:Single 3.0(364) BasePeak:91(246006)
BG Mode:None Group 1 - Event 1

#	m/z	Abs. In	Rel. Int.	#	m/z	Abs. In	Rel. Int.	#	m/z	Abs. In	Rel. Int.
1	50.00	12151	4.94	4	53.00	2575	1.05	7	56.00	3956	1.61
2	50.95	33376	13.57	5	54.00	791	0.32	8	56.95	5393	2.19
3	52.00	8281	3.37	6	55.00	3945	1.60	9	57.95	3636	1.48

MS results for the product number **6**.



^{1}H NMR results for the product number **6**.

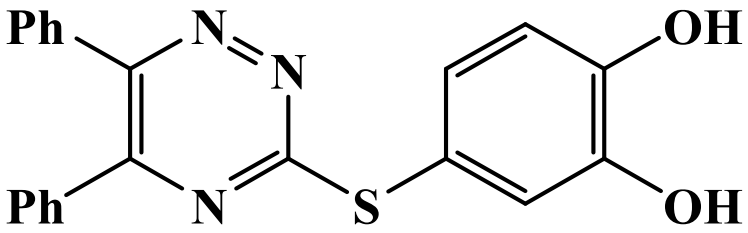


^{13}C NMR results for the product number **6**.

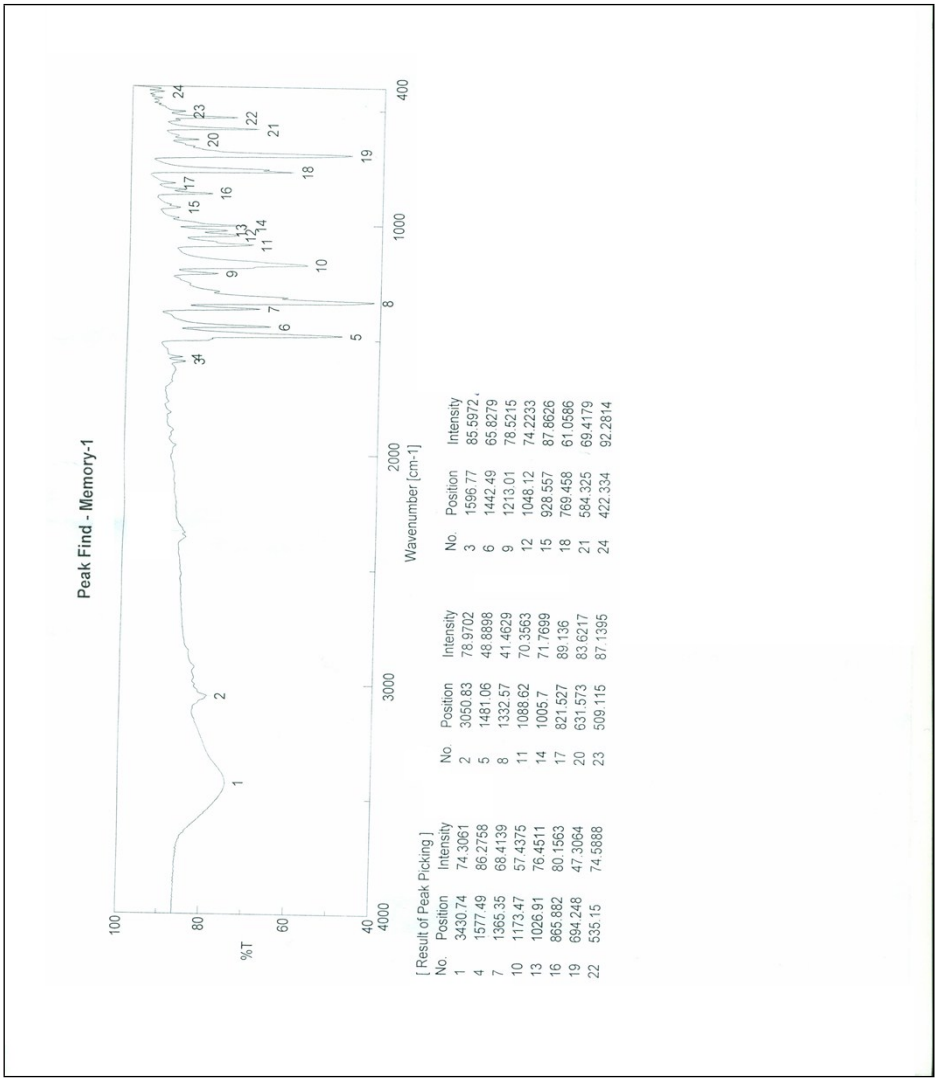
Product number 7:

Name: 5,6-(Diphenyl-[1,2,4]triazin-3-ylsulfanyl)-benzene-1,2-diol

Data: Pale yellow crystals, Yield: 0.23 g, 61.6 %, mp: 175 oC. IR spectrum (KBr) , ν, cm-1: 3430 (br. OH), 3050 (CH-aromatic), 1596 (C=N); ^{1}H NMR (DMSO-d6) (δ ppm): 9.04 (br.s, 2H, 2OH), 7.46-7.29 (m, 13H, Ar'H); ^{13}C NMR spectrum (75 MHz, DMSO-d6) (δ, ppm): 113.88, 120.52, 121.00, 126.86, 128.47, 128.67, 132.12, 135.05 (Ar-C), 144.38 (Ar-OH), 147.17 (Ar-OH), 153.18 (C=N), 153.83 (C=N), 157.84 (N=C-S); Ms: m/z 373 ($M^{+\cdot}$). Anal. Calcd for C21H15N3O2S; C, 67.54; H, 4.05; N, 11.25; S, 8.59. Found: C, 67.17; H, 4.86; N, 11.34; S, 8.06.



7



FT-IR results for the product number **7**.

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Micro Analytical Center

DI Analysis
Shimadzu Qp-2010 Plus

Sample Information

Analyzed by	: A.GABR
Analyzed	: 05/03/2014 05:37:45 م
Sample Name	: S2
Sample ID	
Customer Name	: [illegible]
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Org Data File	: C:\GCMSsolution\Data\Project1\B8.QGD
Method File	: C:\GCMSsolution\Data\Project1\A.GABR.qgm
Org Method File	: C:\GCMSsolution\Data\Project1\A.GABR.qgm
Report File	:
Tuning File	: C:\GCMSsolution\System\Tune1_default1.qgt
Saved Modified by	: A.GABR
Modified	: 05/03/2014 05:40:04 م

Method

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End Time	:10.00min
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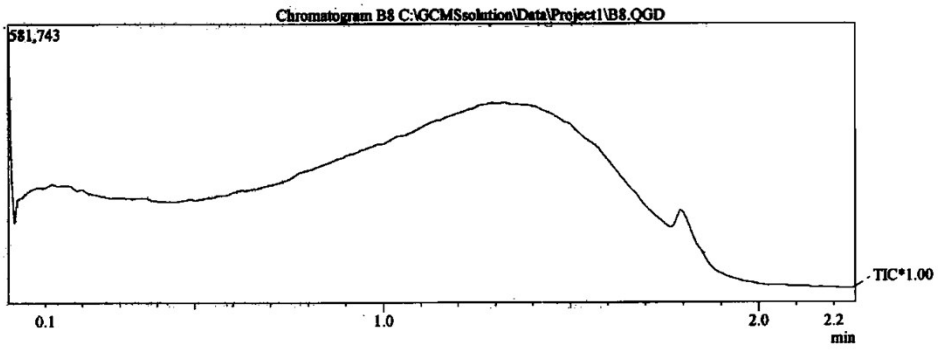
Event Time	:0.50sec
Scan Speed	:769
Start m/z	:50.00
End m/z	:400.00
Electron Voltage	:70 eV
Ionization Mode	: EI

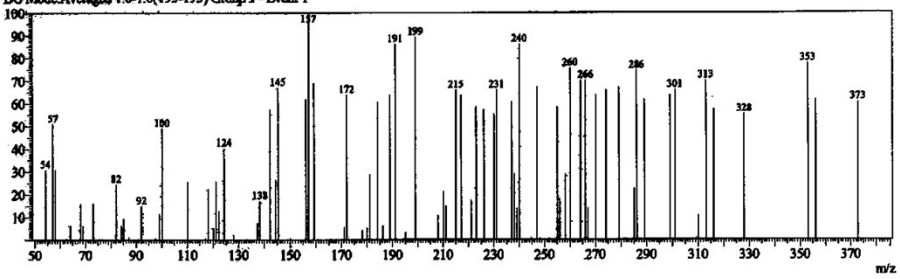
C:\GCMSsolution\Data\Project1\B8.QGD

Chromatogram B8 C:\GCMSsolution\Data\Project1\B8.QGD



Spectrum

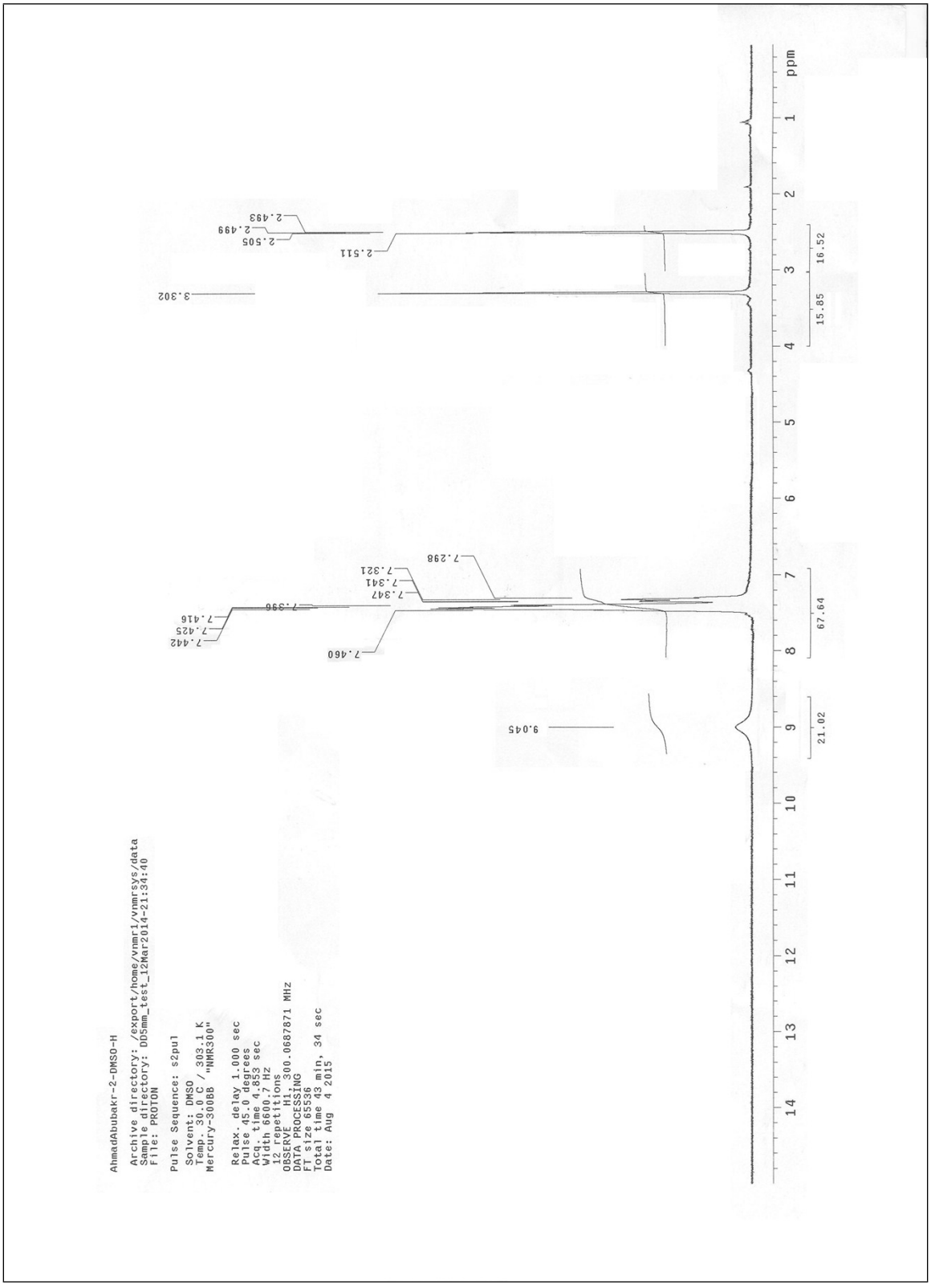
Line#:1 R.Time:1.6(Scan#:197)
MassPeaks:79
RawMode:Single 1.6(197) BasePeak:157(94)
BG Mode:Averaged 1.6-1.6(195-195) Group 1 - Event 1



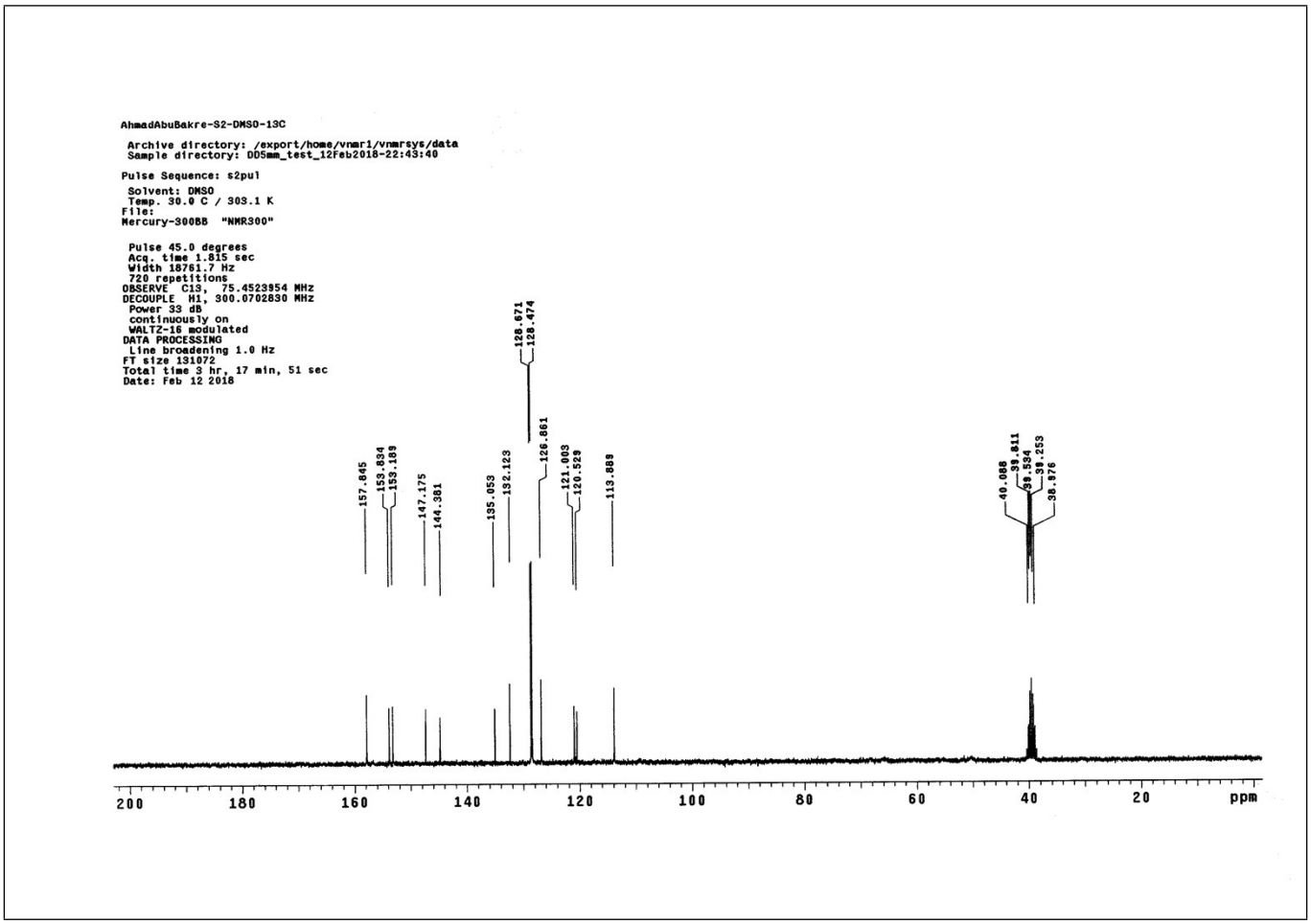
Mass Table
Line#:1 R.Time:1.6(Scan#:197)
MassPeaks:79
RawMode:Single 1.6(197) BasePeak:157(94)
BG Mode:Averaged 1.6-1.6(195-195) Group 1 - Event 1

#	m/z	Abs. In	Rel. Int.	#	m/z	Abs. In	Rel. Int.	#	m/z	Abs. In	Rel. Int.
1	54.15	29	30.85	4	64.10	6	6.38	7	73.05	15	15.96
2	57.10	48	51.06	5	68.10	15	15.96	8	82.15	23	24.47
3	58.05	29	30.85	6	69.10	6	6.38	9	84.10	6	6.38

MS results for the product number **7**.



^{1}H NMR results for the product number **7**.

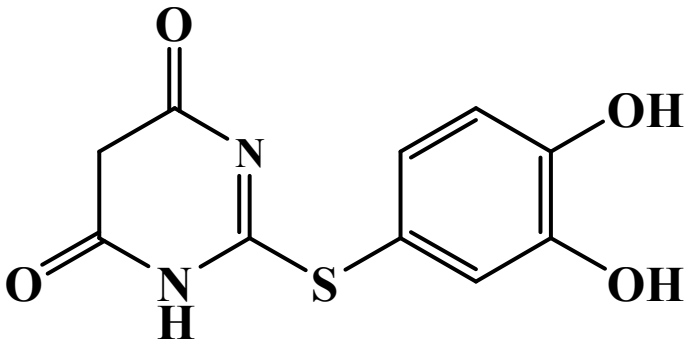


^{13}C NMR results for the product number **7**.

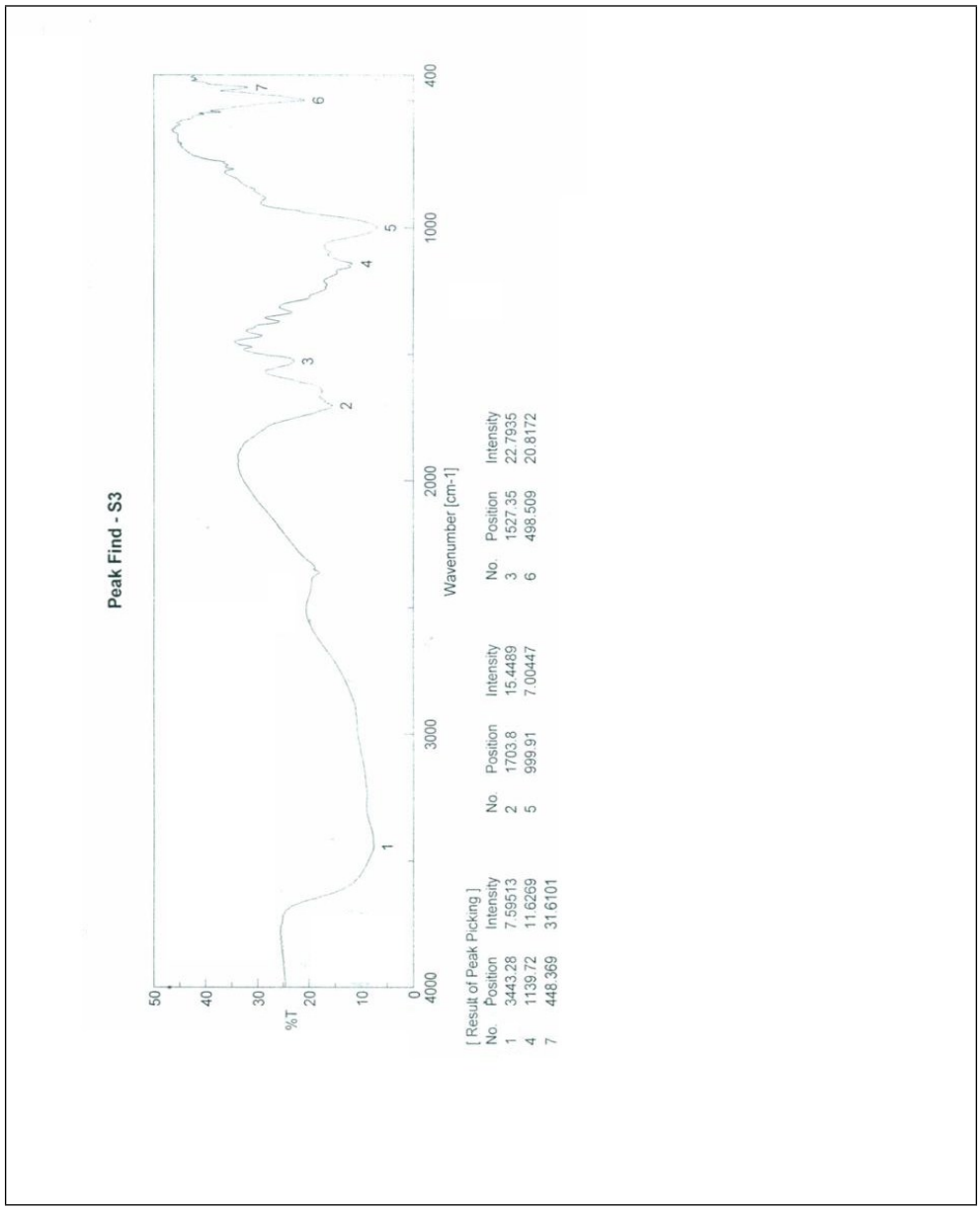
Product number 8:

Name: 6-(3,4-Dihydroxy-phenylsulfanyl)-1H-pyrimidine-2,4-dione

Data: Brown crystals Yield: 0.2 g, 79.3 %, mp: 193 oC. IR spectrum (KBr) , ν, cm-1: 3443 (br. OH), 1703 (C=O); ^{1}H NMR (DMSO-d6) (δ ppm): 11.71 (br.s, 2H, 2OH), 11.08 (s, 1H, NH), 6.96-6.55 (m, 3H, Ar'H), 1.90 (s, 2H, CH_2); ^{13}C NMR spectrum (75 MHz, DMSO-d6) (δ, ppm): 31.83 (CH_2), 116.20, 120.72, 122.33, 127.69 (Ar-C), 145.32 (Ar-OH), 147.65 (Ar-OH), 162.36 (S-C=N)), 166.55 (C=O), 167.98 (C=O); Ms: m/z 252 (M^+). Anal. Calcd for C10H8N2O4S; C, 47.61; H, 3.20; N, 11.11; S, 12.71. Found: C, 47.56; H, 3.13; N, 11.45; S, 12.49.



8



FT-IR results for the product number **8**.

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DI Analysis
Shimadzu Qp-2010 Plus

Sample Information

Analyzed by	: A.GABR
Analyzed	: 02/01/2014 11:33:10 ص
Sample Name	: S3
Sample ID	:
Customer Name	: أحمد محمد ابو بكر - جامعة جنزب الوادى
Data File	: C:\GCMSsolution\Data\Project1\1.QGD
Org Data File	: C:\GCMSsolution\Data\Project1\1.QGD
Method File	: C:\GCMSsolution\Data\Project1\A.GABR.qgm
Org Method File	: C:\GCMSsolution\Data\Project1\A.GABR.qgm
Report File	:
Tuning File	: C:\GCMSsolution\System\Tune1_default1.qgt
EndModified by	: A.GABR
Modified	: 02/01/2014 11:38:14 ص

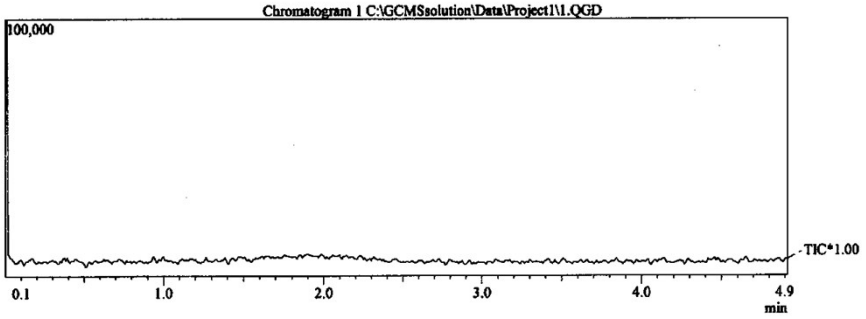
Method

—— Analytical Line 1 ——

IonSourceTemp	:250.00 °C
[MS Table]	
--Group 1 - Event 1--	
Start Time	:0.00min
End Time	:10.00min
ACQ Mode	:Scan
Event Time	:0.50sec
Scan Speed	: 625
Start m/z	:50.00
End m/z	:350.00
Electron Voltage	: 70 eV
Ionization Mode	: EI

C:\GCMSsolution\Data\Project1\1.QGD

Chromatogram 1 C:\GCMSsolution\Data\Project1\1.QGD



Spectrum

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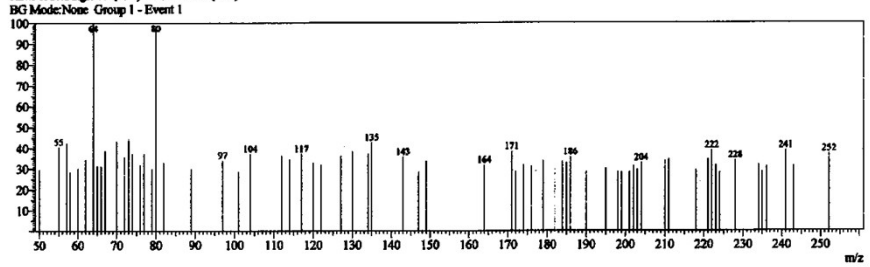
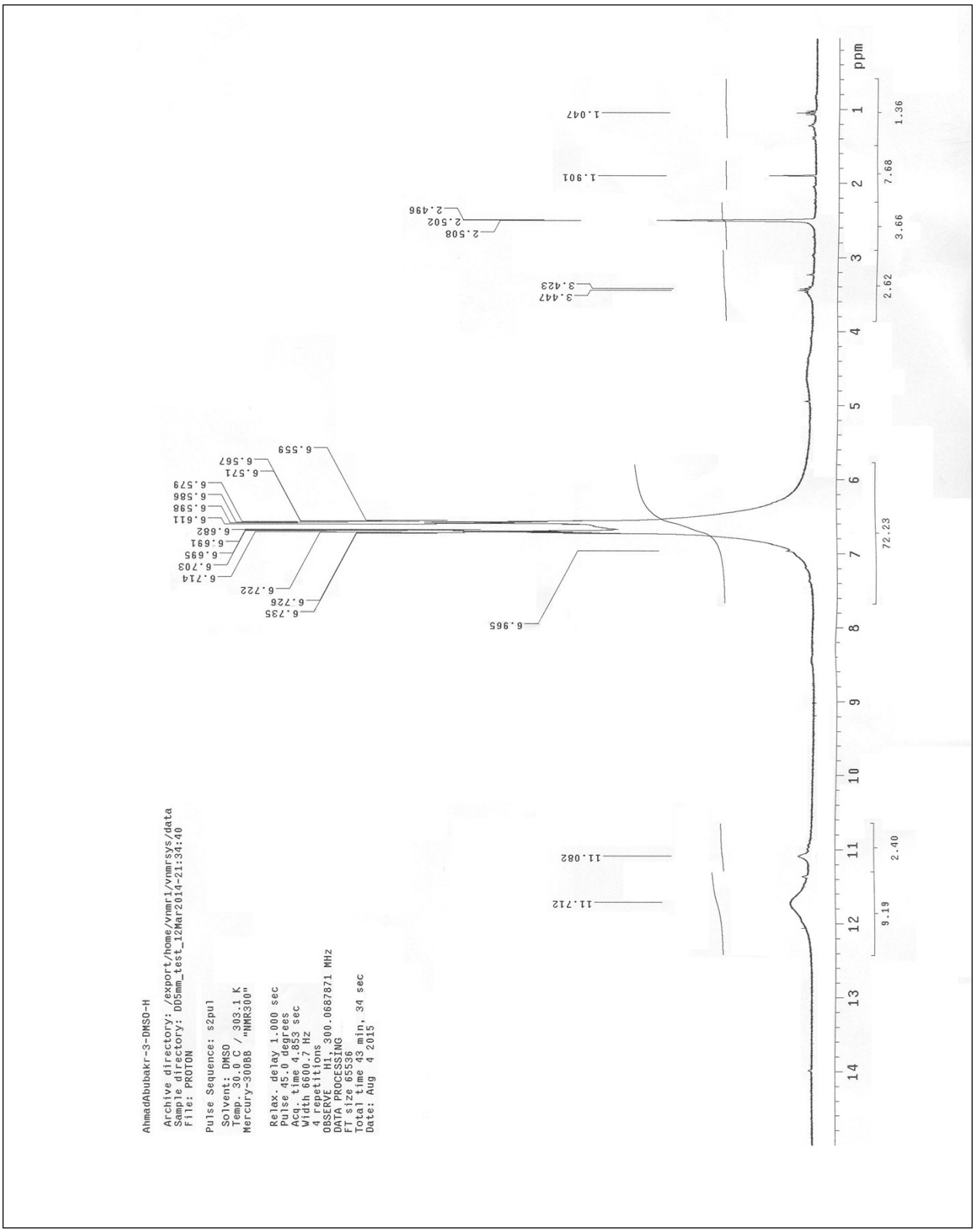
BG Mode:None Group 1 - Event 1



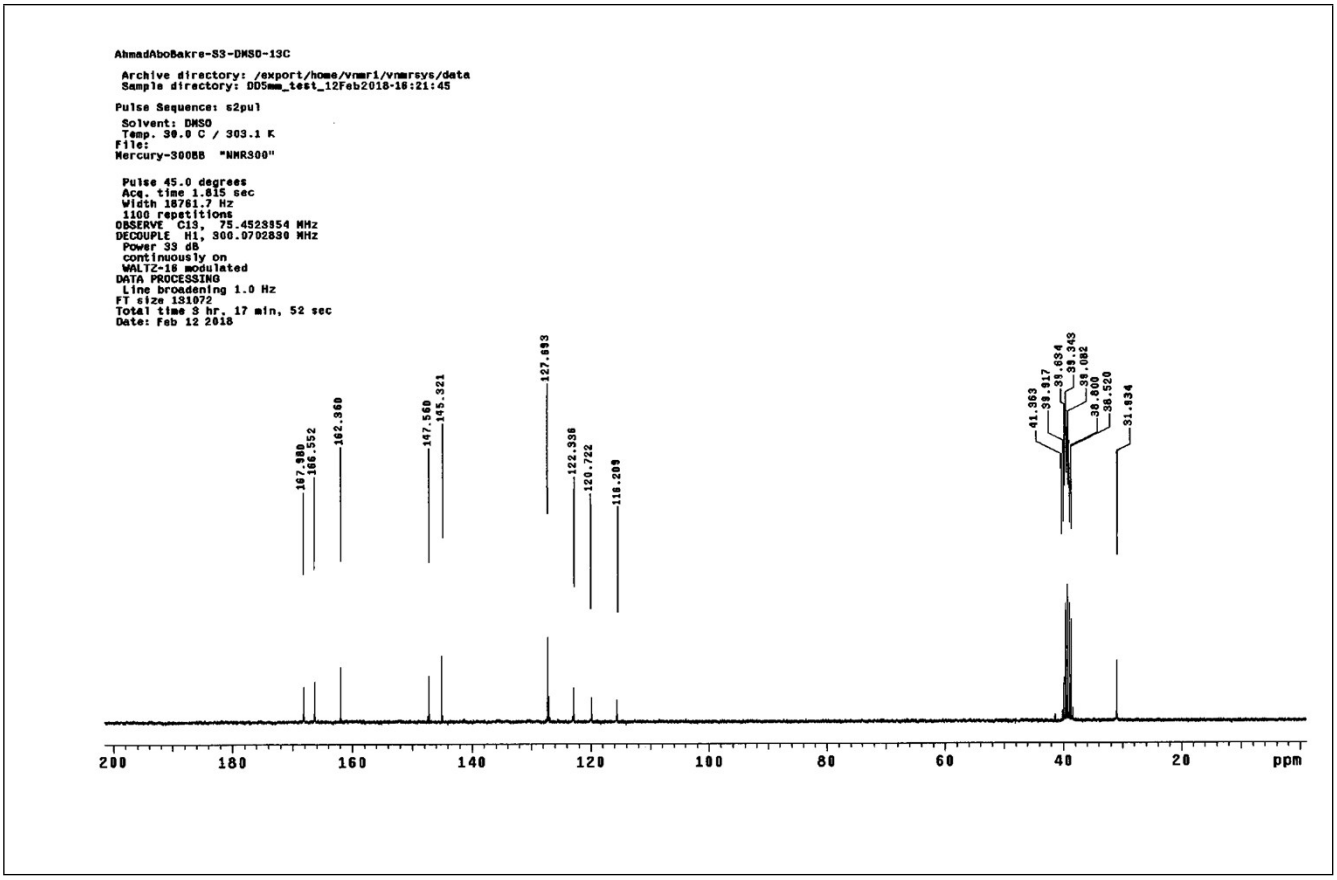
Mass Table
Line#:1 R.Time:3.5(Scan#:423)
MassPeaks:69
RawMode:Single 3.5(423) BasePeak:64(183)
BG Mode:None Group 1 - Event 1

#	m/z	Abs. In	Rel. Int.	#	m/z	Abs. In	Rel. Int.	#	m/z	Abs. In	Rel. Int.
1	50.00	54	29.51	4	58.00	52	28.42	7	64.00	183	100.00
2	55.00	74	40.44	5	60.00	55	30.05	8	65.00	57	31.15
3	57.00	78	42.62	6	62.00	63	34.43	9	66.00	57	31.15

MS results for the product number **8**.



^{1}H NMR results for the product number **8**.



^{13}C NMR results for the product number **8**.