Supplementary File

Green Synthesis and Molecular docking Study of Some New Thiazoles using Terephthalohydrazide Chitosan Hydrogel as Ecofriendly Biopolymeric Catalyst

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2.1. Chemicals and Materials

Chitosan (degree of deacetylation 88% and molecular weight of 200000 g mol^{-1}) was purchased from Funakoshi Co. LTD (Japan). Benzaldehyde, epichlorohydrin, terephthalohydrazide, 2-acetylthiophene, hydrazine hydrate, phenylisothiocyanate, thiosemicarbazide, phenacylbromide, acetylacetone, ethylacetoacetate, aniline, 4-chloroaniline, 4-bromoaniline, 4-anisidine, 4-toluidine, sulphuryl chloride, ether, absolute ethanol, dimethylformamide, 4-chloroacetophenone, 4-bromoacetophenone, 4-methylacetophenone, 4-methoxyacetophenone, 3-nitoacetophenone, glacial acetic acid were provided by Alfa Aesar (UK) and Sigma-Aldrich (Germany) and were of analytical grade and used as received. All chemicals and reagents were purchased from Funakoshi Co. LTD (Japan), Alfa Aesar (UK) and Sigma-Aldrich (Germany) and were of analytical grade and used as received.

2.2. Measurements

IR spectra were recorded in potassium bromide discs on Pye Unicam SP 3300 and Shimadzu FTIR 8101 PC infrared spectrophotometers. The Shimadzu GCMS-QP1000 EX mass spectrometer was used to record mass spectra at 70 eV. . The ^{1}H-NMR and ^{13}C-NMR spectra were recorded using a Jeol-500 spectrometer (500 MHz for ^{1}H-NMR and 125 MHz for ^{13}C-NMR). On a digital melting point device from the Electrothermal IA 9000 series, melting points were calculated. FTIR spectra of chitosan and TcsSB were given using KBr pellets with a range of 400 to 4000 cm^{-1} by a Tescan Shimadzu FTIR spectrophotometer, Model 8000, Japan. X-ray diffractometer (Brucker's D-8) was used to determine the internal structure of chitosan and TCsSB hydrogel. The samples were scanned at a various angle, 2θ, in the range between 3º and 70º at 80 min^{-1} speed at room temperature. The source of the X-ray (1.5406 ˚A, 40 kV, 30 mA) was made by CuKα radiation filtered by a nickel. Chitosan and TCsSB surface morphology was scanned using a Quanta scanning electron microscope (model FEG 250). Before scanning the samples were sputtered with gold.

The physical constants and analytical information for the known products 5a-d, 9a-c, and 11a are listed below.

4-Methyl-5-(phenyldiazenyl)-2-(2-(1-(thiophen-2-yl)ethylidene)hydrazinyl)thiazole (5a)

Red solid; mp 183-185 °C (DMF); IR (KBr) ν_{max}: 3427 (NH), 3043 (=C-H), 2931 (-C-H), 1603 (C=N) cm^{-1}; ^1H NMR (DMSO-d_6) δ: 2.50 (s, 3H, CH_3), 2.58 (s, 3H, CH_3), 6.99-7.75 (m, 8H, ArH), 10.67 (s, 1H, D_2O-exchangeable, NH); ^{13}C NMR (75 MHz, DMSO-d_6) δ: 8.5, 15.5 (CH_3), 114.2, 122.1, 128.0, 128.3, 129.2, 130.2, 130.8, 137.9, 143.4 (Ar-C), 160.4, 170.6, 177.6 (C=N) ppm; MS *m/z* (%): 341 (M^+, 44), 238 (71), 106 (53), 78 (82), 51 (100); Anal. Calcd. for $C_{16}H_{15}N_5S_2$ (341.08): C, 56.28; H, 4.43; N, 20.51; Found: C, 56.22; H, 4.36; N, 20.40%.

4-Methyl-2-(2-(1-(thiophen-2-yl)ethylidene)hydrazinyl)-5-(*p*-tolyldiazenyl)thiazole (5b)

Red solid; mp 169-170 °C (DMF); IR (KBr) ν_{max}: 3427 (NH), 3042 (=C-H), 2928 (-C-H), 1602 (C=N) cm^{-1}; ^1H NMR (DMSO-d_6) δ: 2.25 (s, 3H, CH_3), 2.50 (s, 3H, CH_3), 2.56 (s, 3H, CH_3), 7.12-7.73 (m, 7H, Ar-H), 10.60 (s, 1H, D_2O-exchangeable, NH) ppm; ^{13}C-NMR (75 MHz, DMSO-d_6) δ: 8.5, 15.5, 20.3 (CH_3), 114.2, 128.0, 129.6, 130.0, 130.7, 131.1, 137.3, 141.1, 142.7 (Ar-C), 160.1, 177.3, 170.8 (C=N); MS *m/z* (%): 355 (M^+, 20), 238 (53), 185 (77), 78 (69), 51 (100); Anal. Calcd. for $C_{17}H_{17}N_5S_2$ (355.09): C, 57.44; H, 4.82; N, 19.70; Found: C, 57.36; H, 4.77; N, 19.63%.

5-((4-Chlorophenyl)diazenyl)-4-methyl-2-(2-(1-(thiophen-2-yl)ethylidene)hydrazinyl)thiazole (5c)

Red solid; mp 216-218 °C (DMF); IR (KBr) ν_{max}: 3427 (NH), 3043 (=C-H), 2931 (-C-H), 1603 (C=N) cm^{-1}; ^1H NMR (DMSO-d_6) δ: 2.51 (s, 3H, CH_3), 2.57 (s, 3H, CH_3), 7.17-7.74 (m, 7H, Ar-H), 10.71 (s, 1H, D_2O-exchangeable, NH) ppm; ^{13}C-NMR (75 MHz, DMSO-d_6) δ: 8.5, 15.4 (CH_3), 115.6, 125.6, 128.0, 128.3, 129.1, 129.3, 138.7, 142.4, 142.5 (Ar-C), 160.6, 170.4, 177.6 (C=N) ppm; MS *m/z* (%): 377 (M^+ + 2, 32), 375 (M^+, 100), 222 (54), 186 (72), 78 (69), 51 (80); Anal. Calcd. for $C_{16}H_{14}ClN_5S_2$ (375.04): C, 51.12; H, 3.75; N, 18.63; Found: C, 51.03; H, 3.66; N, 18.51%.

5-((4-Bromophenyl)diazenyl)-4-methyl-2-(2-(1-(thiophen-2-yl)ethylidene)hydrazinyl) thiazole (5d)

Red solid; mp 203-205 °C (DMF); IR (KBr) ν_{max}: 3441 (NH), 3043 (=C-H), 2937 (-C-H), 1601 (C=N) cm^{-1}; ^1H NMR (DMSO-d_6) δ: 2.51(s, 3H, CH_3), 2.57 (s, 3H, CH_3), 7.17-7.75 (m, 7H, Ar-H), 10.72 (s, 1H, D_2O-exchangeable, NH) ppm; ^{13}C-NMR (75 MHz, DMSO-d_6) δ: 8.5, 15.5 (CH_3),

116.1, 128.0, 130.3, 130.9, 131.9, 132.1, 138.8, 142.5, 142.8 (Ar-C), 160.6, 170.4, 177.6 (C=N) ppm; MS *m/z* (%): 421 (M^+ + 2, 18), 419 (M^+, 19), 302 (100), 185 (83), 78 (82), 51 (74); Anal. Calcd. for $C_{16}H_{14}BrN_5S_2$ (418.99): C, 45.72; H, 3.36; N, 16.66; Found: C, 45.55; H, 3.30; N, 16.42%.

4-Phenyl-2-(2-(1-(thiophen-2-yl)ethylidene)hydrazineyl)thiazole (9a).

Yellow crystals; mp 258-260 °C (DMF); IR (KBr) ν_{max}: 3219, 3116 (NH), 3030 (=C-H), 2977 (-C-H), 1613 (C=N) cm^{-1}; ^{1}H-NMR (DMSO-d_6) δ: 2.33 (s, 3H, CH_3), 7.05–7.07 (m, 1H, Ar-H), 7.29-7.31 (m, 2H, Ar-H), 7.38–7.40 (m, 3H, Ar-H), 7.52 (s, 1H, Thiazole-H_5), 7.85–7.87 (m, 2H, Ar-H) ppm; Anal. Calcd. for $C_{15}H_{13}N_3S_2$ (299.4): C, 60.17; H, 4.38; N, 14.03. Found: C, 59.88; H, 4.28; N, 13.89%.

4-(4-Methoxyphenyl)-2-(2-(1-(thiophen-2-yl)ethylidene)hydrazineyl)thiazole (9b).

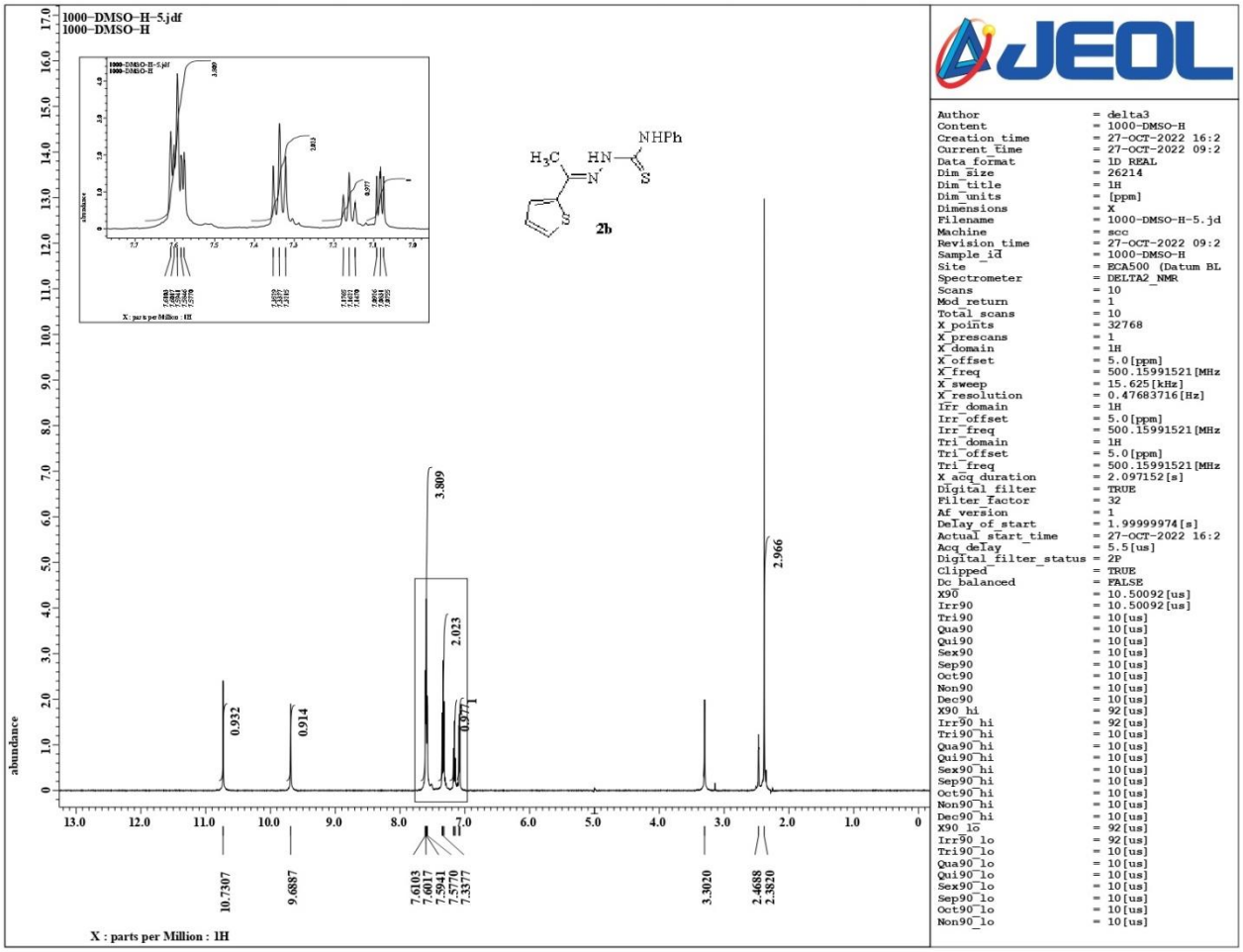
Yellow crystals; mp 249-251 °C (DMF); IR (KBr) ν_{max}: 3219, 3116 (NH), 3030 (=C-H), 2977 (-C-H), 1613 (C=N) cm^{-1}; ^{1}H-NMR (DMSO-d_6) δ: 2.33 (s, 3H, CH_3), 2.49 (s, 3H, OCH_3), 6.95-7.07 (m, 3H, Ar-H), 7.12 (s, 1H, Thiazole-H_5), 7.38-7.40 (m, 2H, Ar-H), 7.77-7.79 (m, 2H, Ar-H), 11.02 (bs, 1H, NH, D_2O-exchangeable) ppm. Anal. Calcd. for $C_{15}H_{13}N_3S_2$ (329.44): C, 58.33; H, 4.59; N, 12.76. Found: C, 58.24; H, 4.39; N, 12.67%.

4-(3-Nitrophenyl)-2-(2-(1-(thiophen-2-yl)ethylidene)hydrazineyl)thiazole (9c).

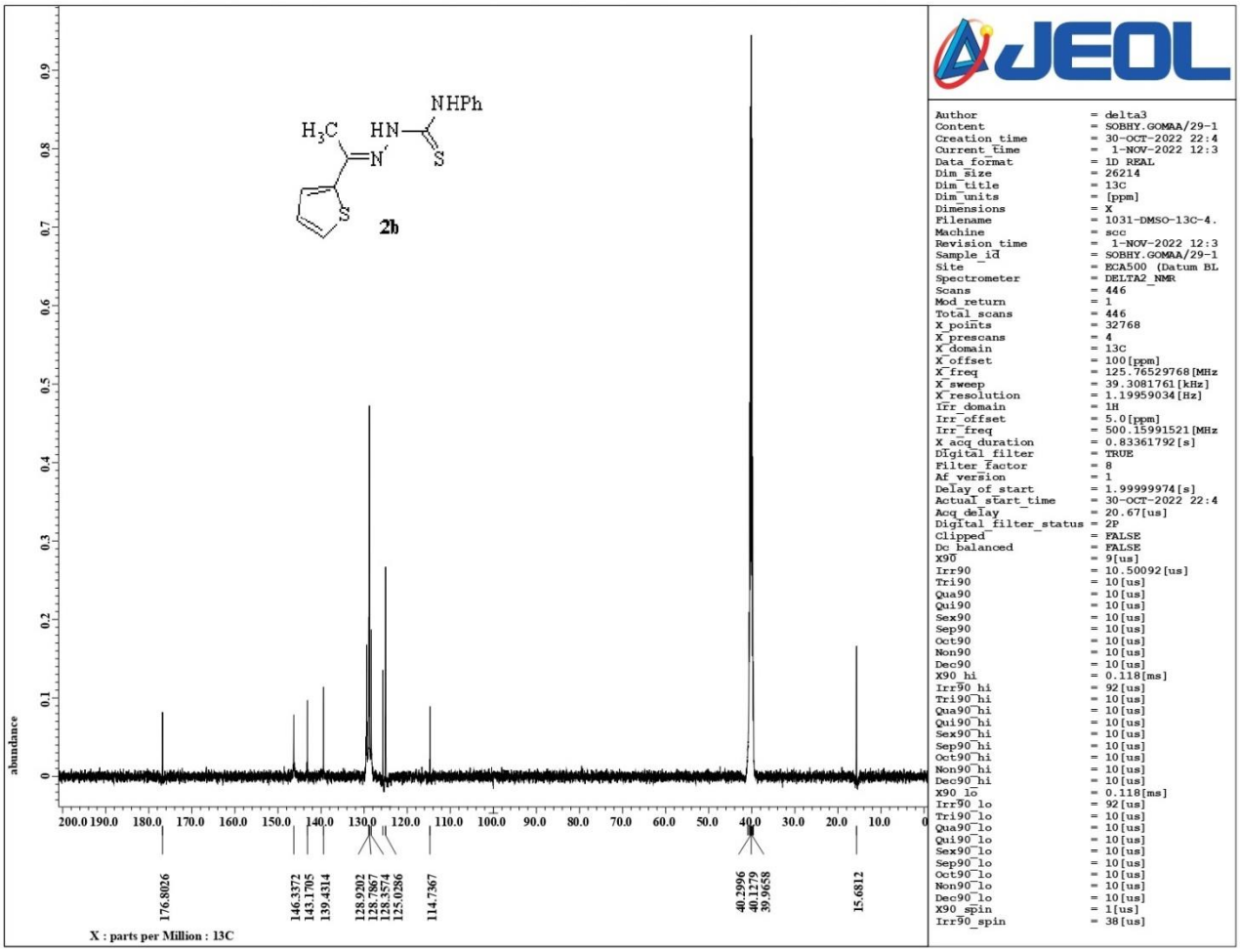
Yellow crystals; mp 246-248 °C (DMF); IR (KBr) ν_{max}: 3215 (NH), 3042, (=C-H), 2924 (-C-H), 1614 (C=N) cm^{-1}; ^{1}H-NMR (DMSO-d_6) δ: 2.28 (s, 3H, CH_3), 7.04 (s, 1H, Thiazole-H_5), 7.32–7.41 (m, 1H, Ar-H), 7.45–7.52 (m, 1H, Ar-H), 7.62–7.82 (m, 1H, Ar-H), 7.89 (d, 1H, Ar-H), 8.15–8.30 (m, 1H, Ar-H), 8.35 (d, 1H, Ar-H), 8.57–8.78 (m, 1H, Ar-H) ppm; Anal. Calcd. for $C_{15}H_{12}N_4O_2S_2$ (344.4): C, 52.31; H, 3.51; N, 16.27. Found: C, 52.09; H, 3.59; N, 16.16%.

1-(4-Methyl-2-(2-(1-(thiophen-2-yl)ethylidene)hydrazineyl)thiazol-5-yl)ethan-1-one (11a).

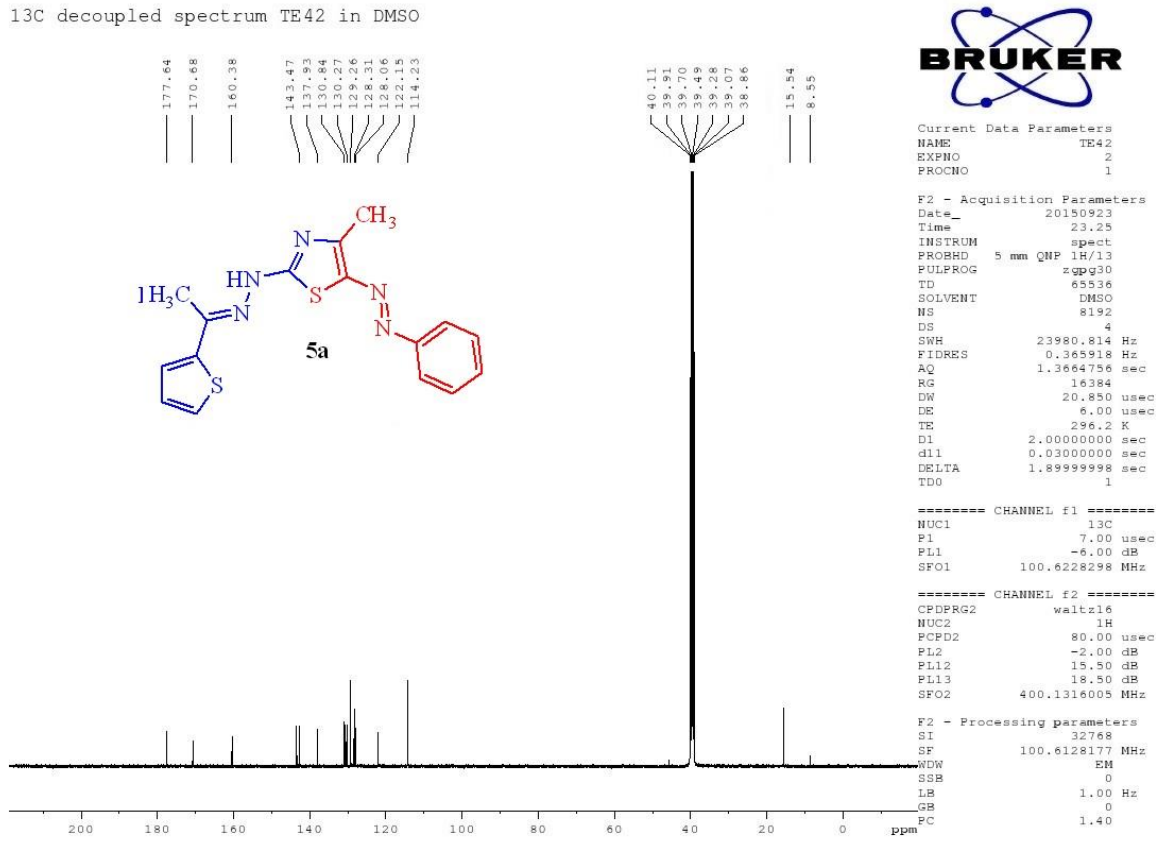
Yellow crystals, mp 218-220 °C (DMF); IR (KBr) ν_{max}: 3342 (NH), 2919 (-C-H), 1652 (C=O), 1611 (C=N) cm^{-1}; ^{1}H-NMR (DMSO-d_6) δ: 2.33 (s, 3H, CH_3), 2.39 (s, 3H, CH_3), 2.48 (s, 3H, CH_3), 7.06–7.08 (dd, 1H, Ar-H), 7.42 (d, 1H, Ar-H), 7.54–7.55 (dd, 1H, Ar-H) ppm; Anal. Calcd. for $C_{12}H_{13}N_3OS_2$ (279.4): C, 51.59; H, 4.69; N, 15.04. Found: C, 51.34; H, 4.60; N, 14.89%.



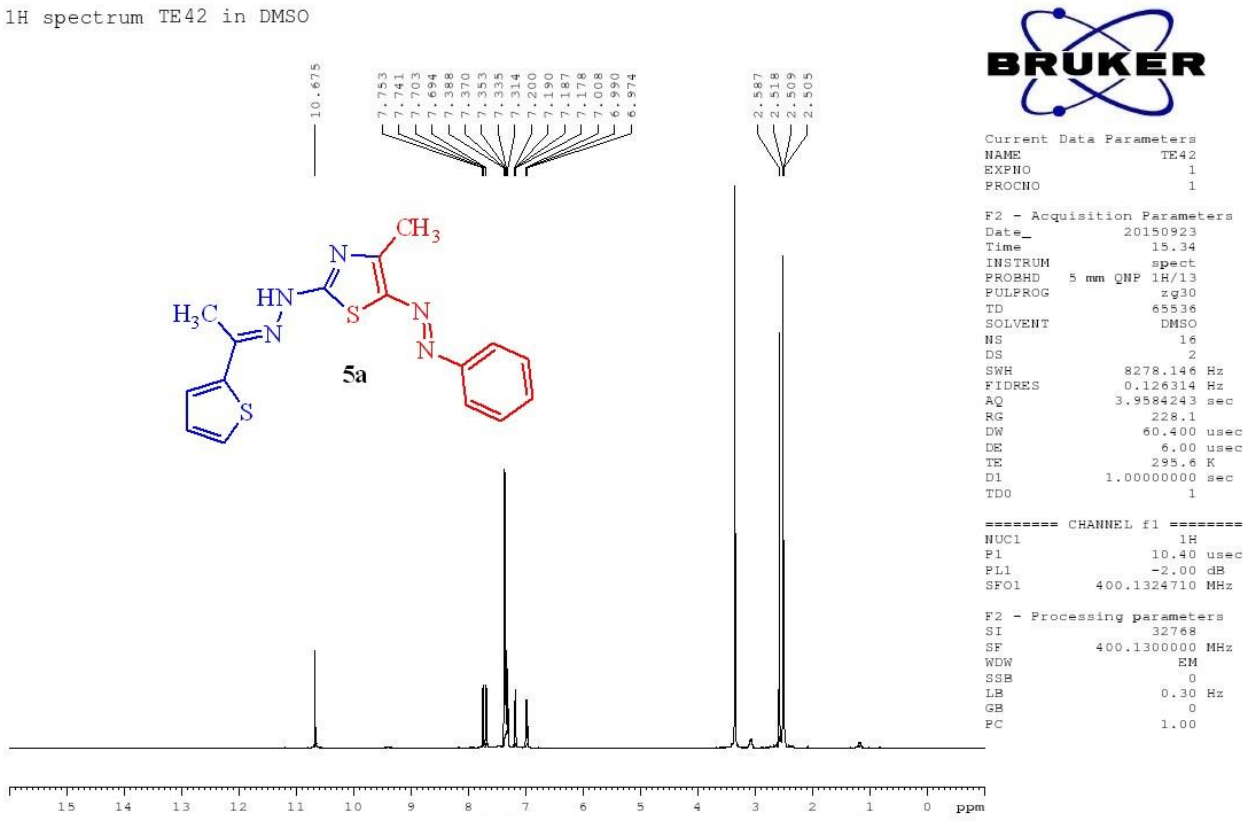
^{1}H-NMR spectra of compound **2b**



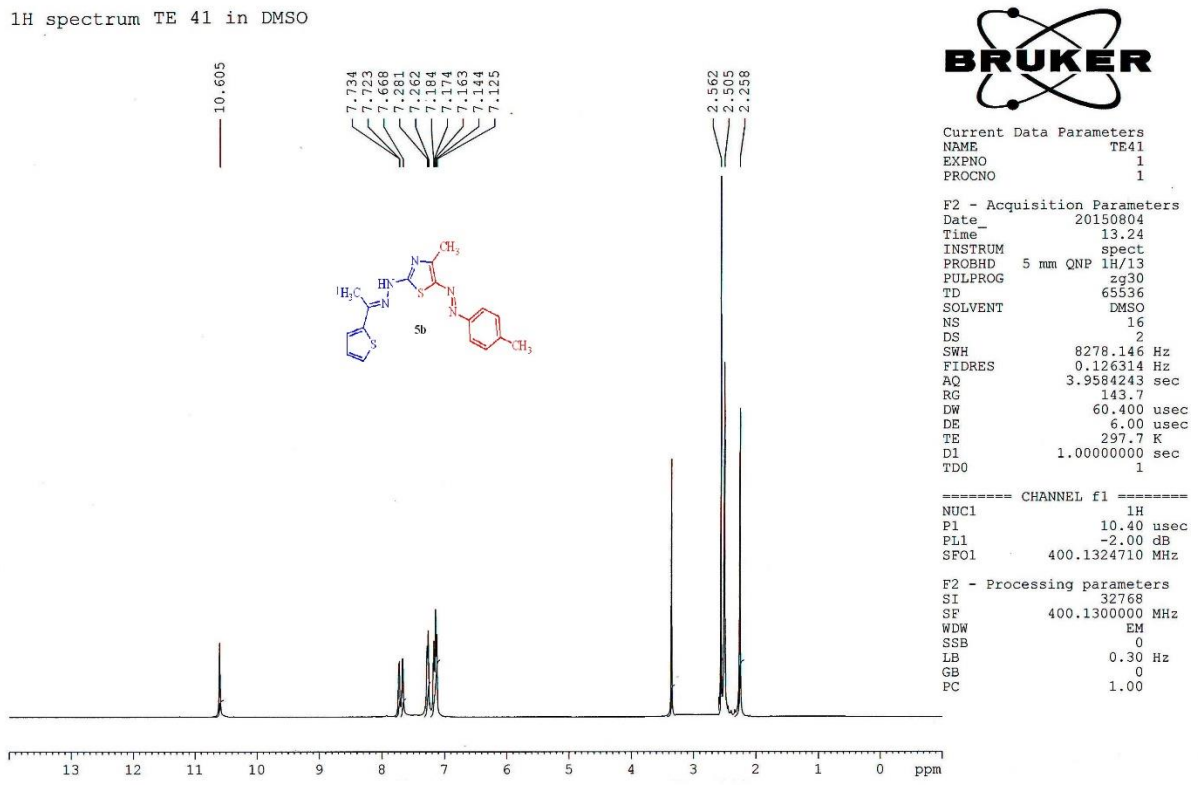
^{13}C-NMR spectra of compound **2b**



^{13}C-NMR spectra of compound **5a**



^{1}H-NMR spectra of compound **5a**



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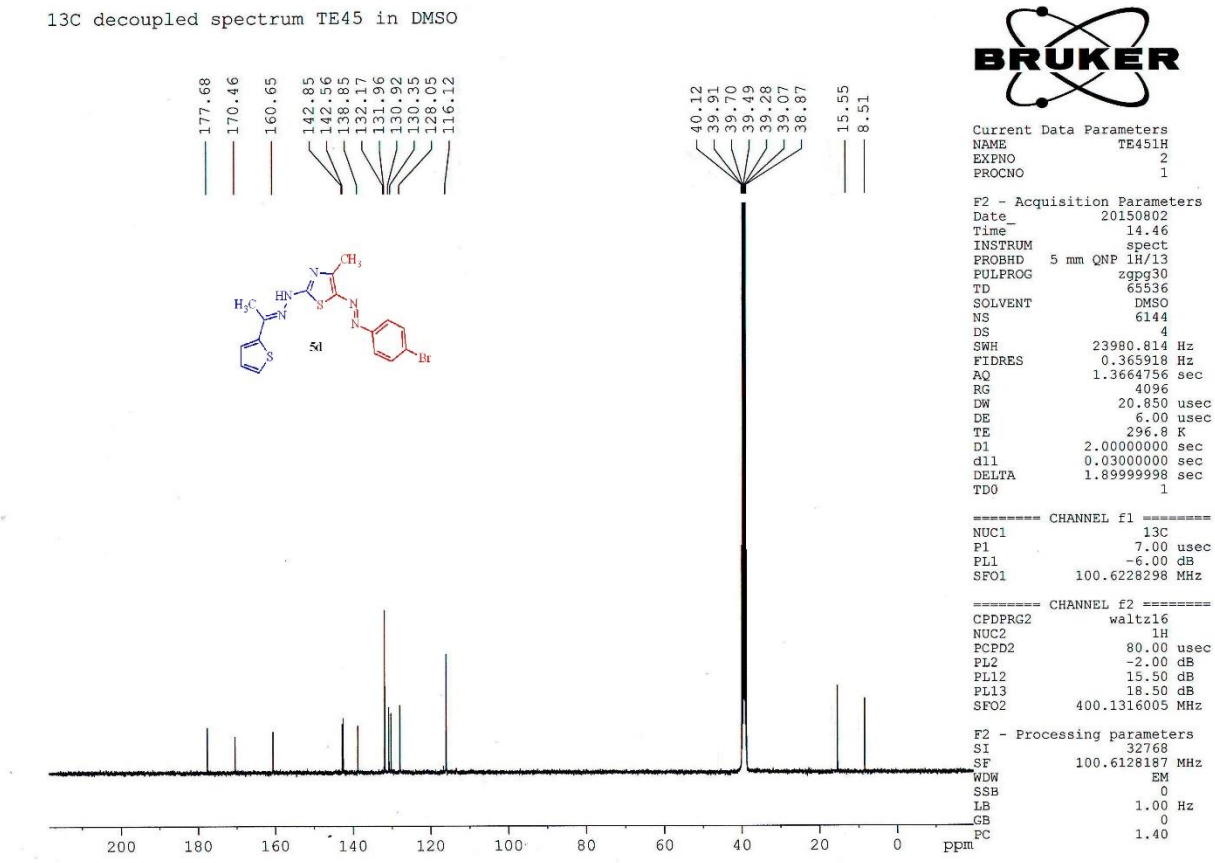
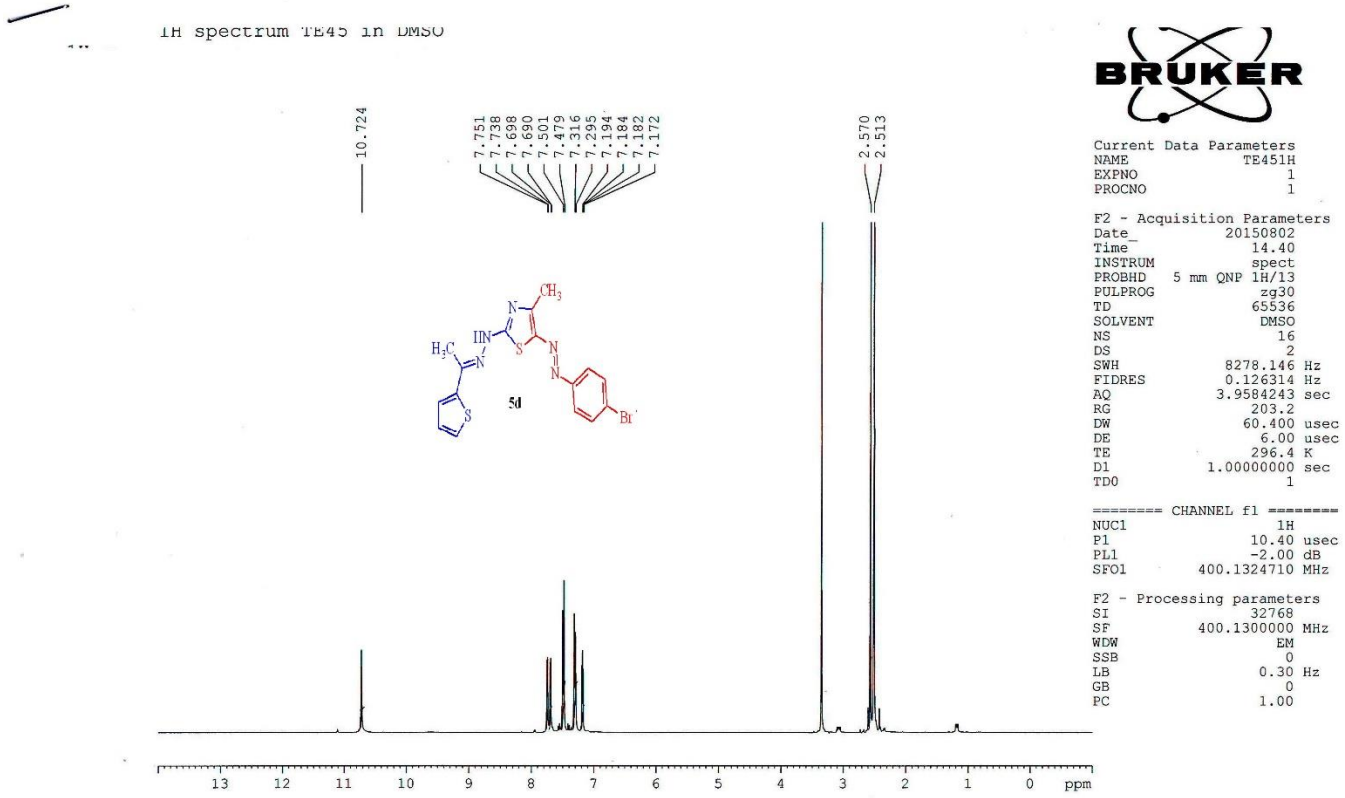


^{13}C-NMR spectra of compound **5d**



BRUKER

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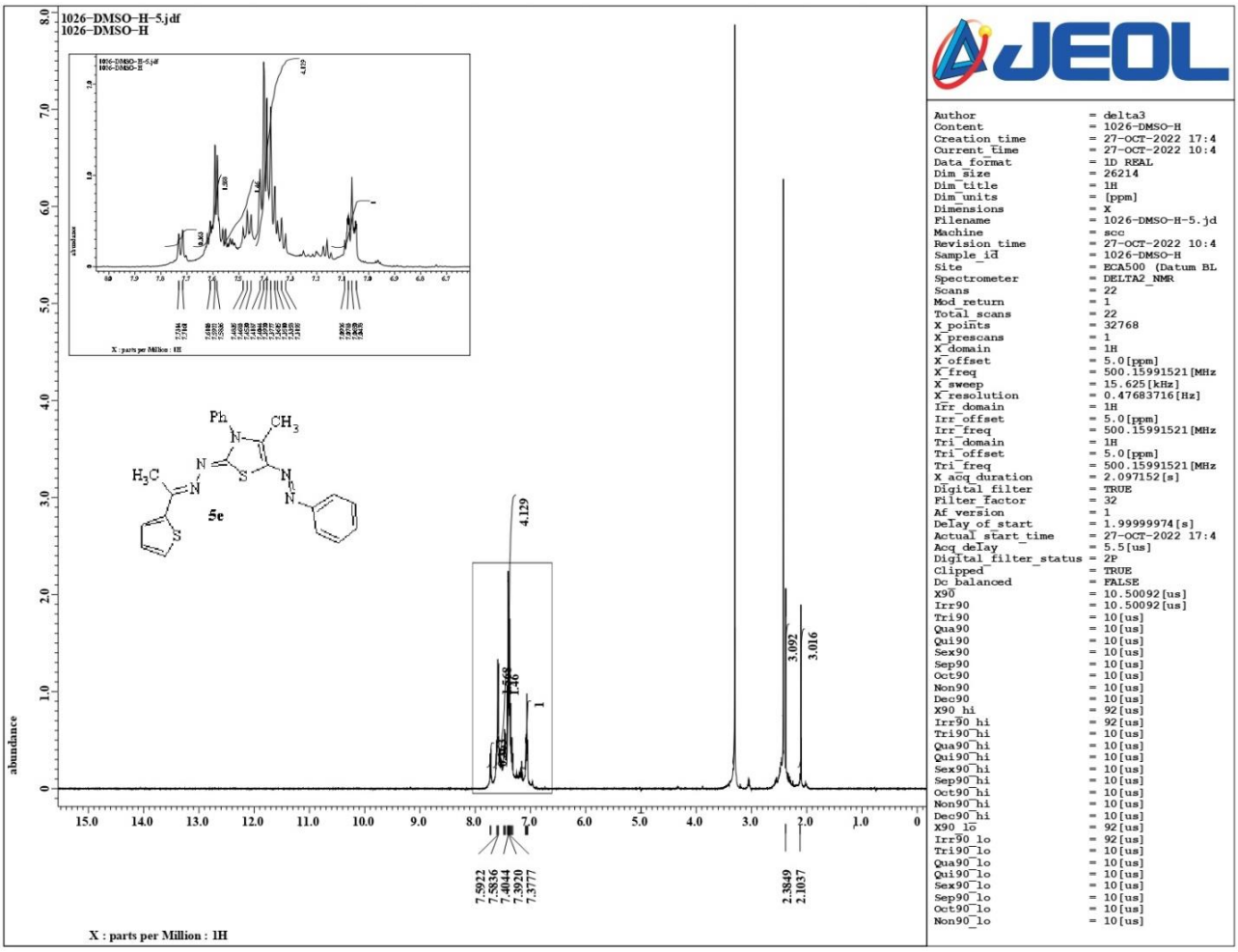
^{1}H-NMR spectra of compound **5d**



^{1}H-NMR spectra of compound **5e**

Cairo University
Micro Analytical Center

DI Analysis
Shimadzu Qp-2010 Plus

Sample Information

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Sample ID	:
Customer Name	: Dr. Sobhy Goma - Science - Cairo
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Modified	:

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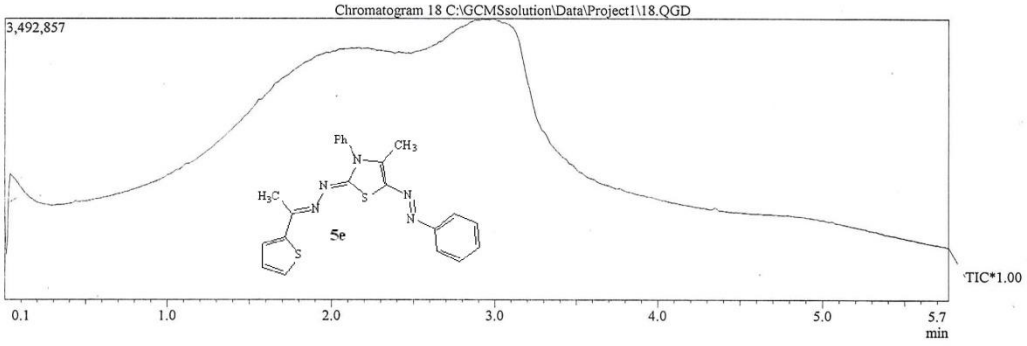
Chromatogram 18 C:\GCMSsolution\Data\Project1\18.QGD



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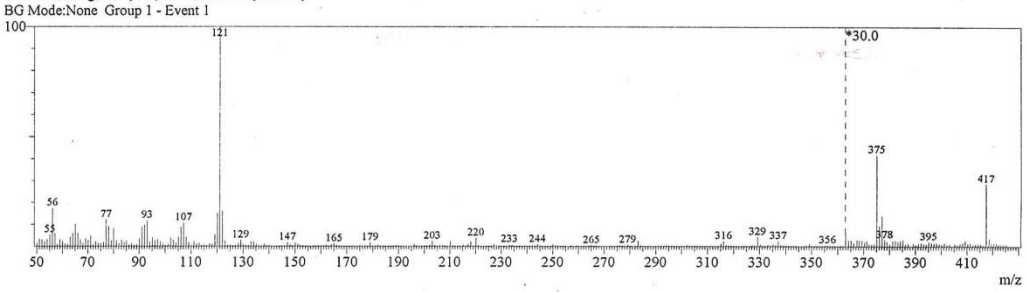

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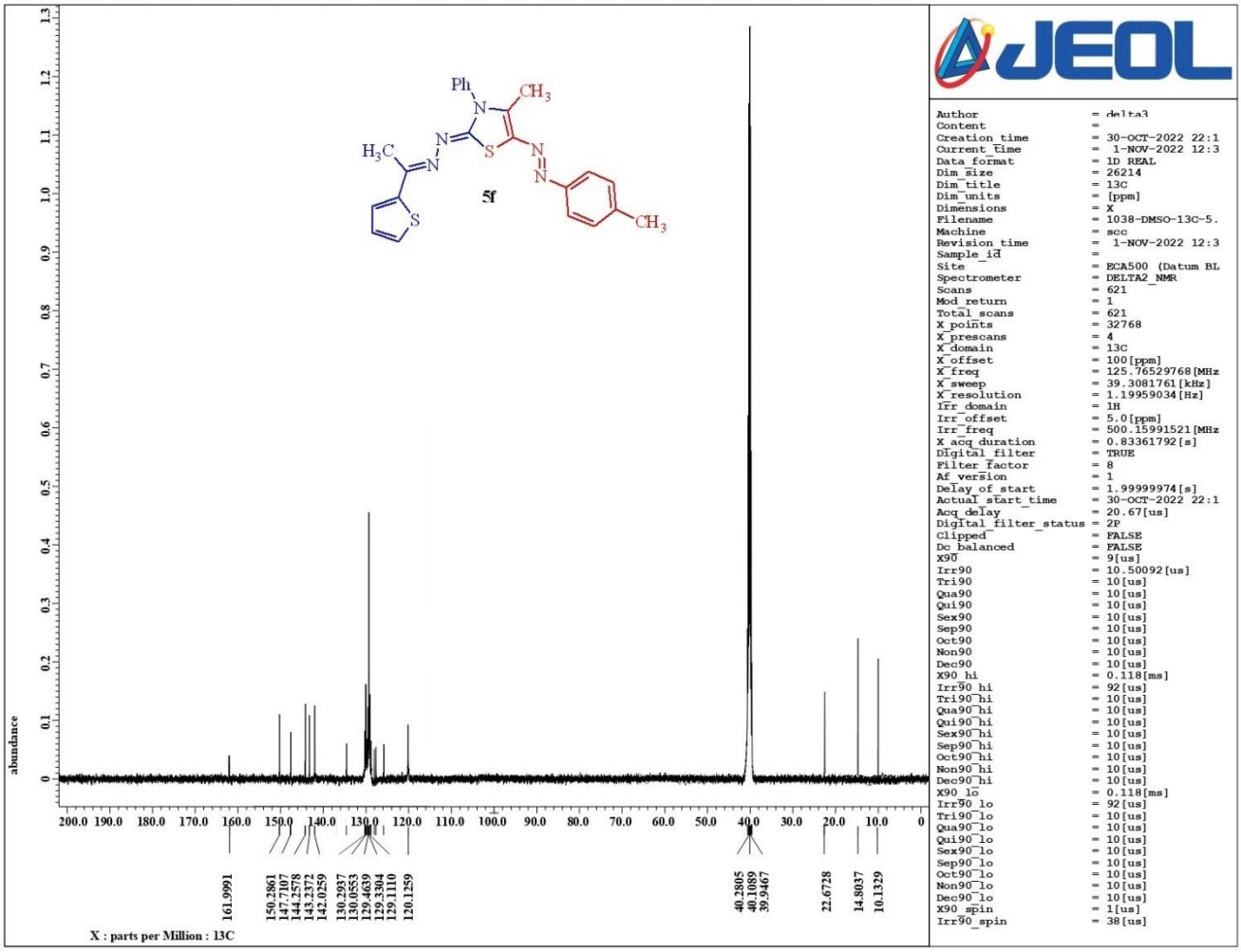
Mass spectra of compound **5e**



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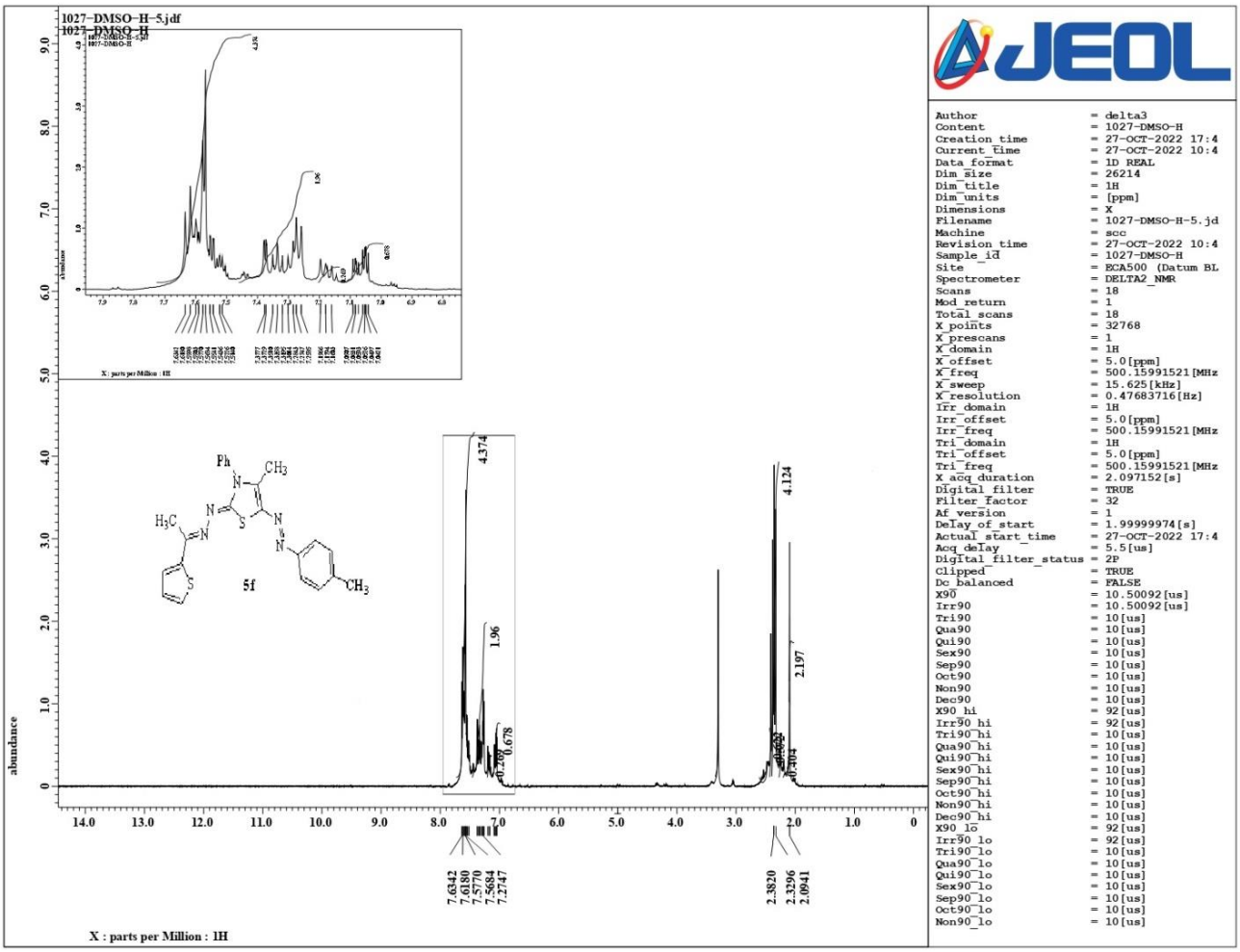
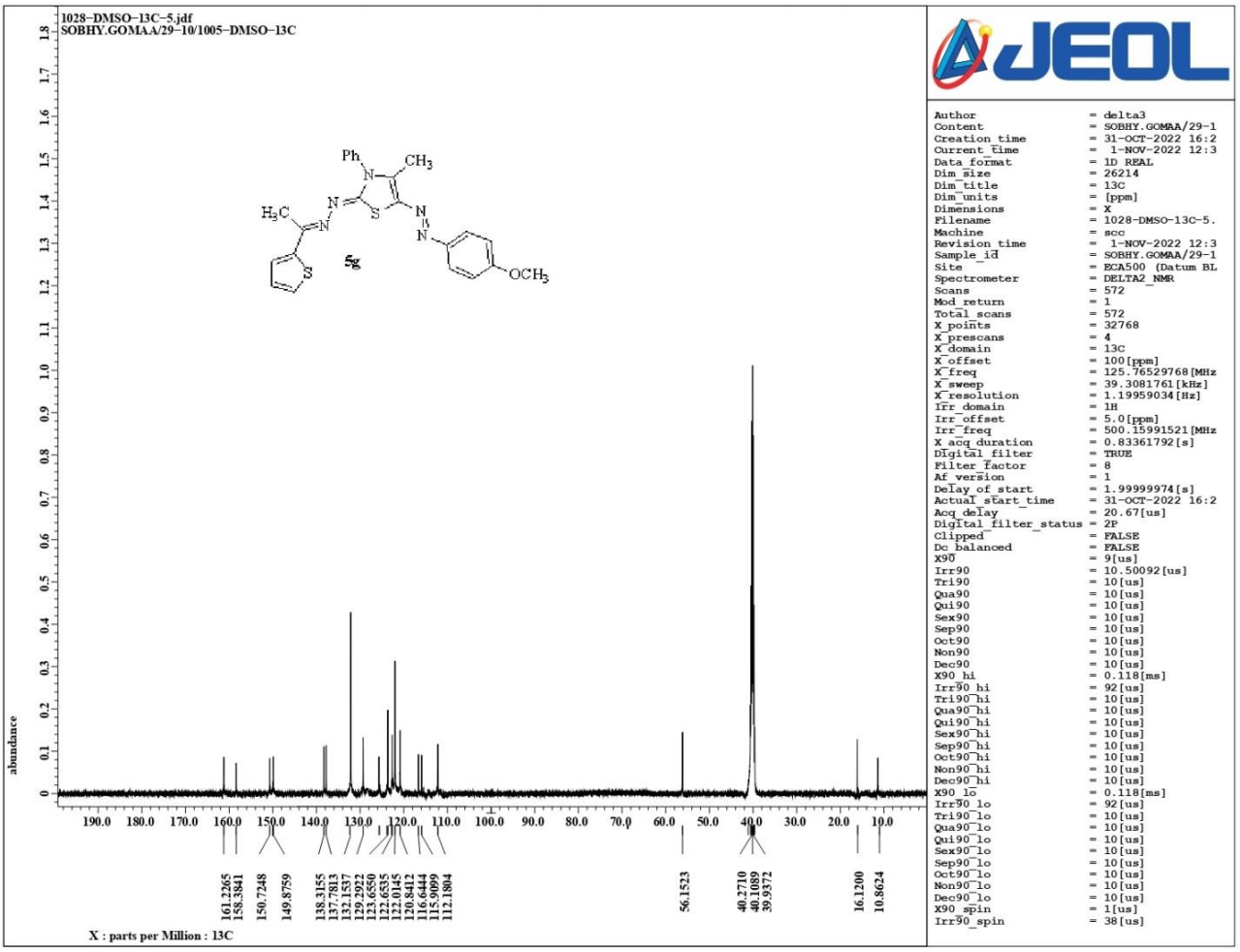
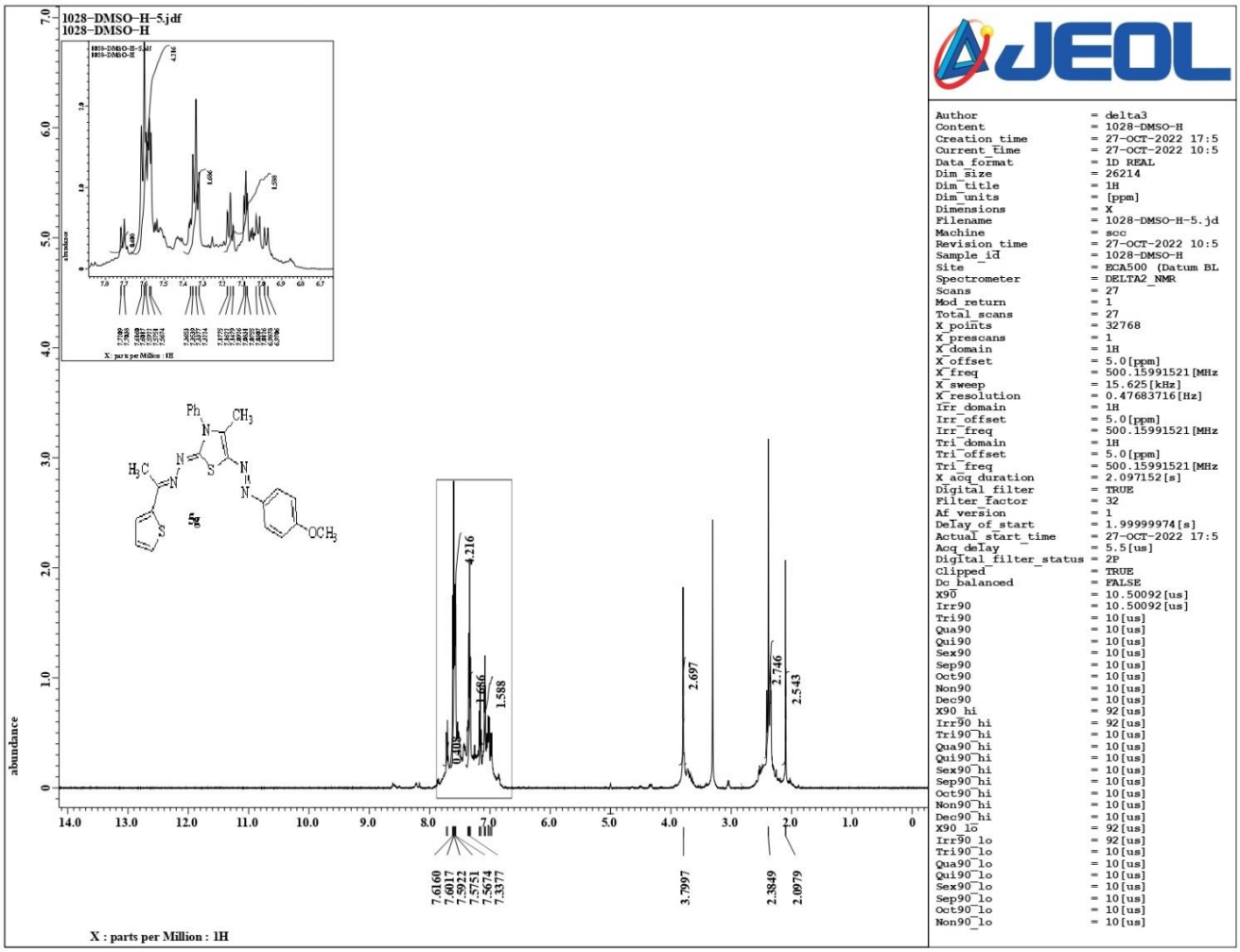


^{1}H-NMR spectra of compound **5f**



^{13}C-NMR spectra of compound **5g**



^{1}H-NMR spectra of compound **5g**

Cairo University
Micro Analytical Center

DI Analysis
Shimadzu Qp-2010 Plus

Sample Information

Analyzed by	: Dr. Mai Younis
Analyzed	:
Sample Name	: 16
Sample ID	:
Customer Name	: Dr. Sobhy Goma - Science - Cairo
Data File	: C:\GCMSsolution\Data\Project1\16.QGD
Org Data File	: C:\GCMSsolution\Data\Project1\16.QGD
Method File	: C:\GCMSsolution\Data\Project1\High Temperature Op
Org Method File	: C:\GCMSsolution\Data\Project1\High Temperature Op
Report File	:
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Modified	:

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	:1250
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End m/z	:600.00

Electron Voltage : 70 eV
Ionization Mode : EI

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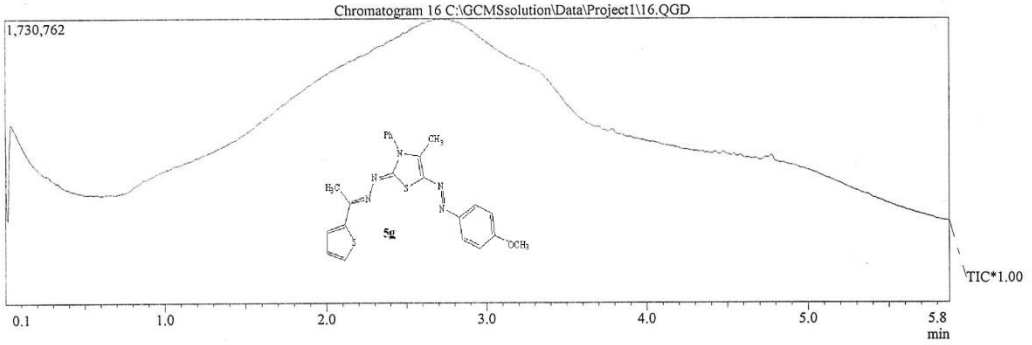
Chromatogram 16 C:\GCMSsolution\Data\Project1\16.QGD



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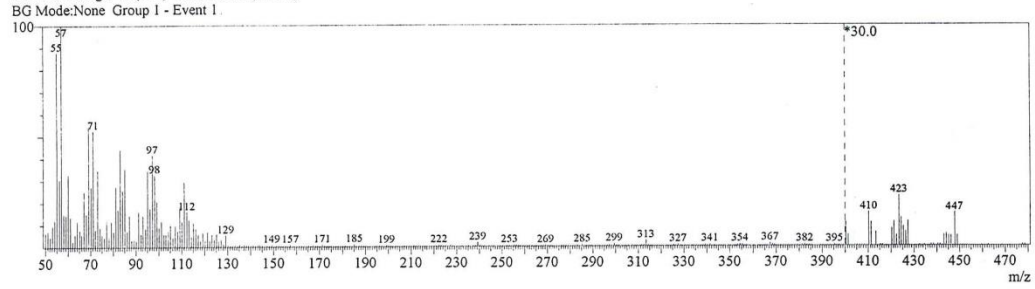

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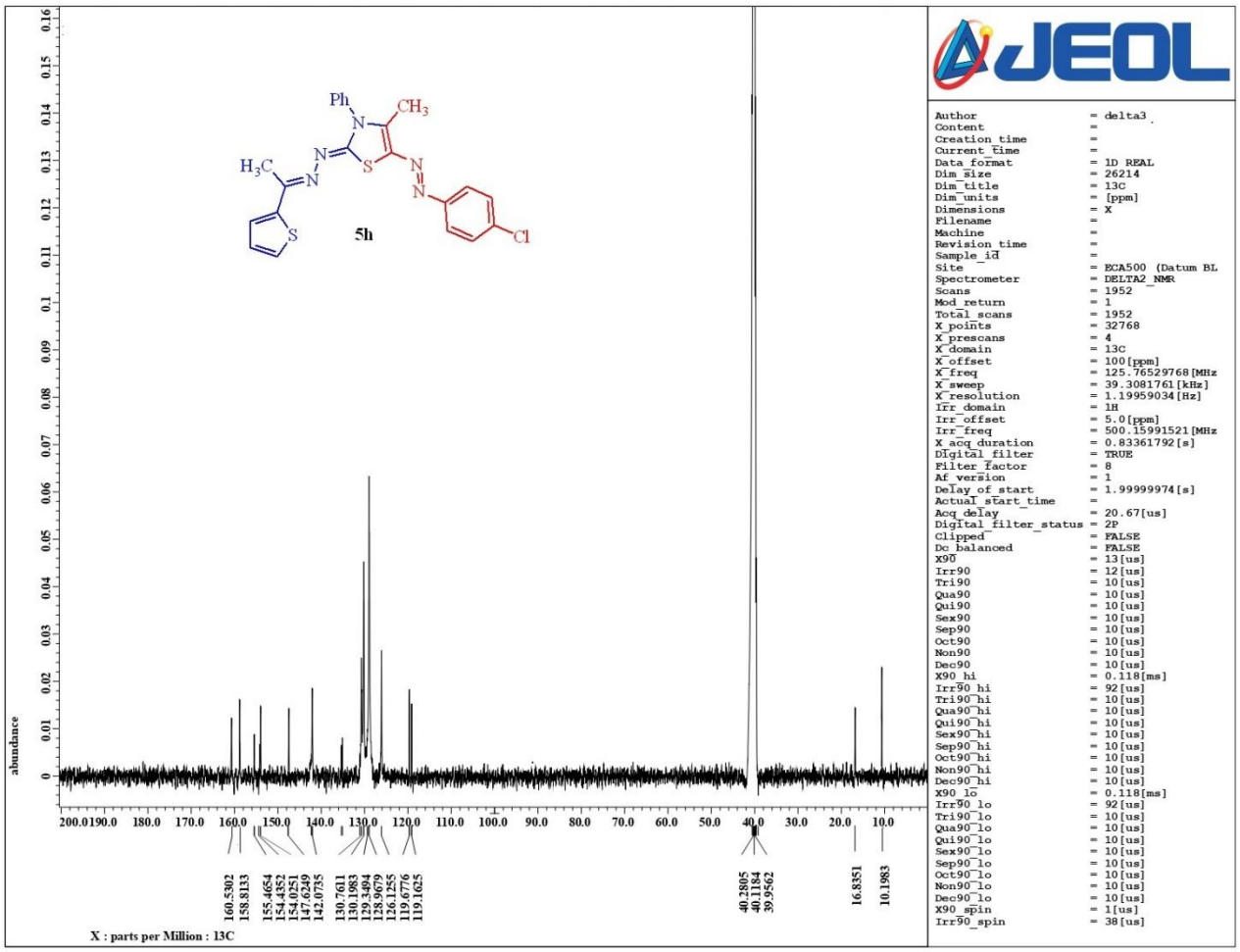
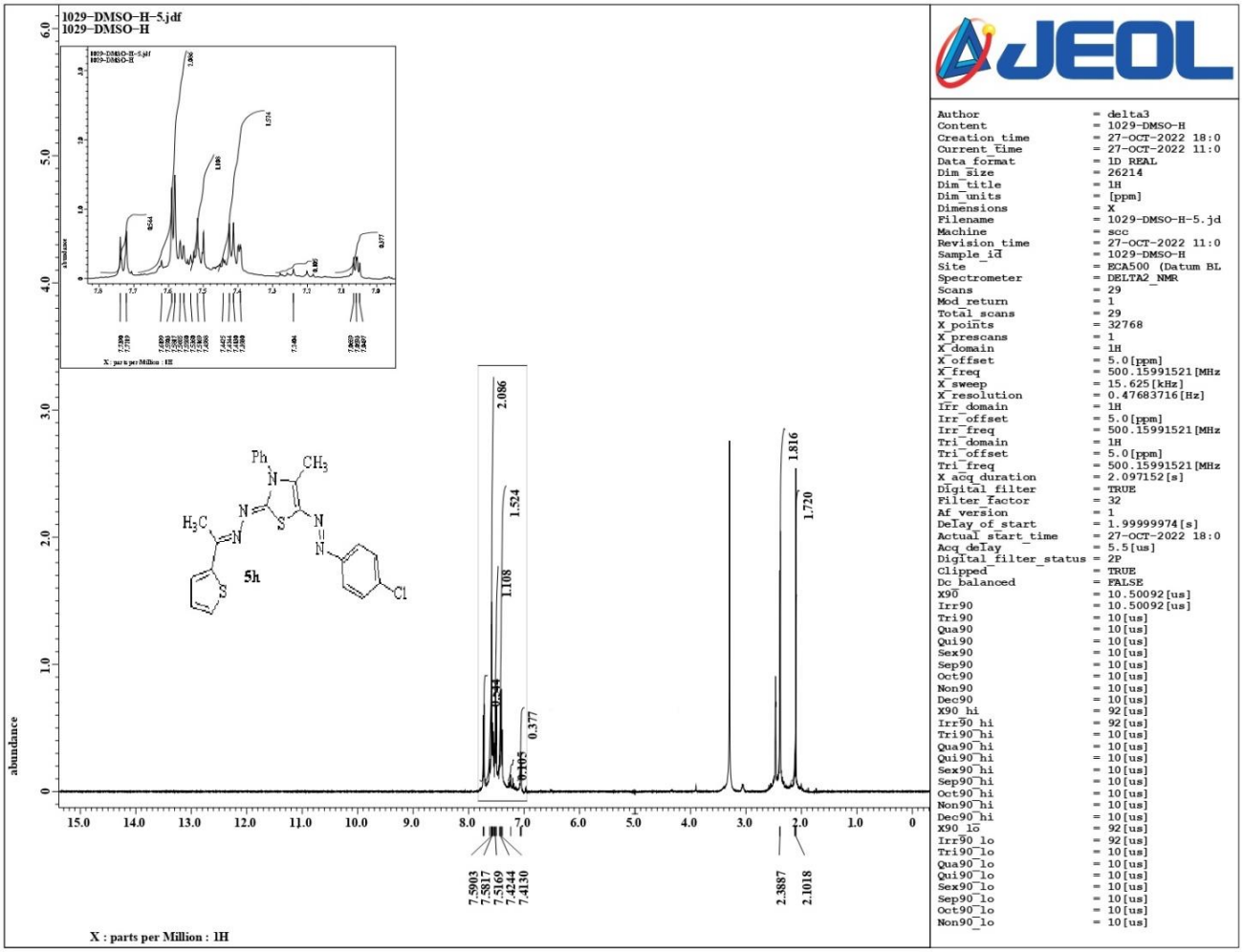
Mass spectra of compound **5g**



^{13}C-NMR spectra of compound **5h**



^{1}H-NMR spectra of compound **5h**

Cairo University
Micro Analytical Center

DI Analysis
Shimadzu Qp-2010 Plus

Sample Information

Analyzed by	: Dr. Mai Younis
Analyzed	:
Sample Name	: 15
Sample ID	:
Customer Name	: Dr. Sobhy Goma - Science - Cairo
Data File	: C:\GCMSsolution\Data\Project1\15.QGD
Org Data File	: C:\GCMSsolution\Data\Project1\15.QGD
Method File	: C:\GCMSsolution\Data\Project1\High Temperature Op
Org Method File	: C:\GCMSsolution\Data\Project1\High Temperature Op
Report File	:
Tuning File	: C:\GCMSsolution\System\Tune1_default.qgt
$EndIf$Modified by	: Dr. Mai Younis
Modified	:

Method

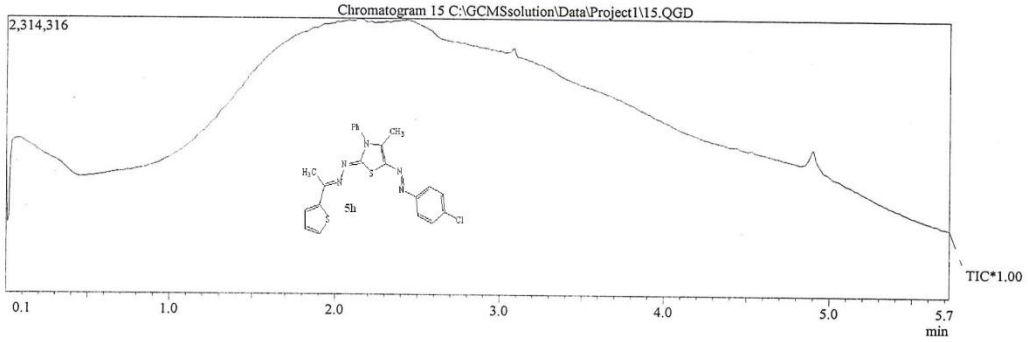
====== Analytical Line 1 ======

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	:1250
Start m/z	:50.00
End m/z	:600.00

Electron Voltage : 70 eV
Ionization Mode : EI

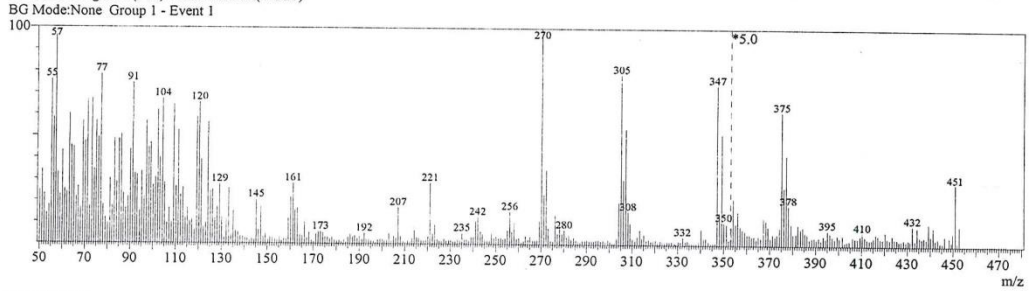
C:\GCMSsolution\Data\Project1\15.QGD

Chromatogram 15 C:\GCMSsolution\Data\Project1\15.QGD



Spectrum

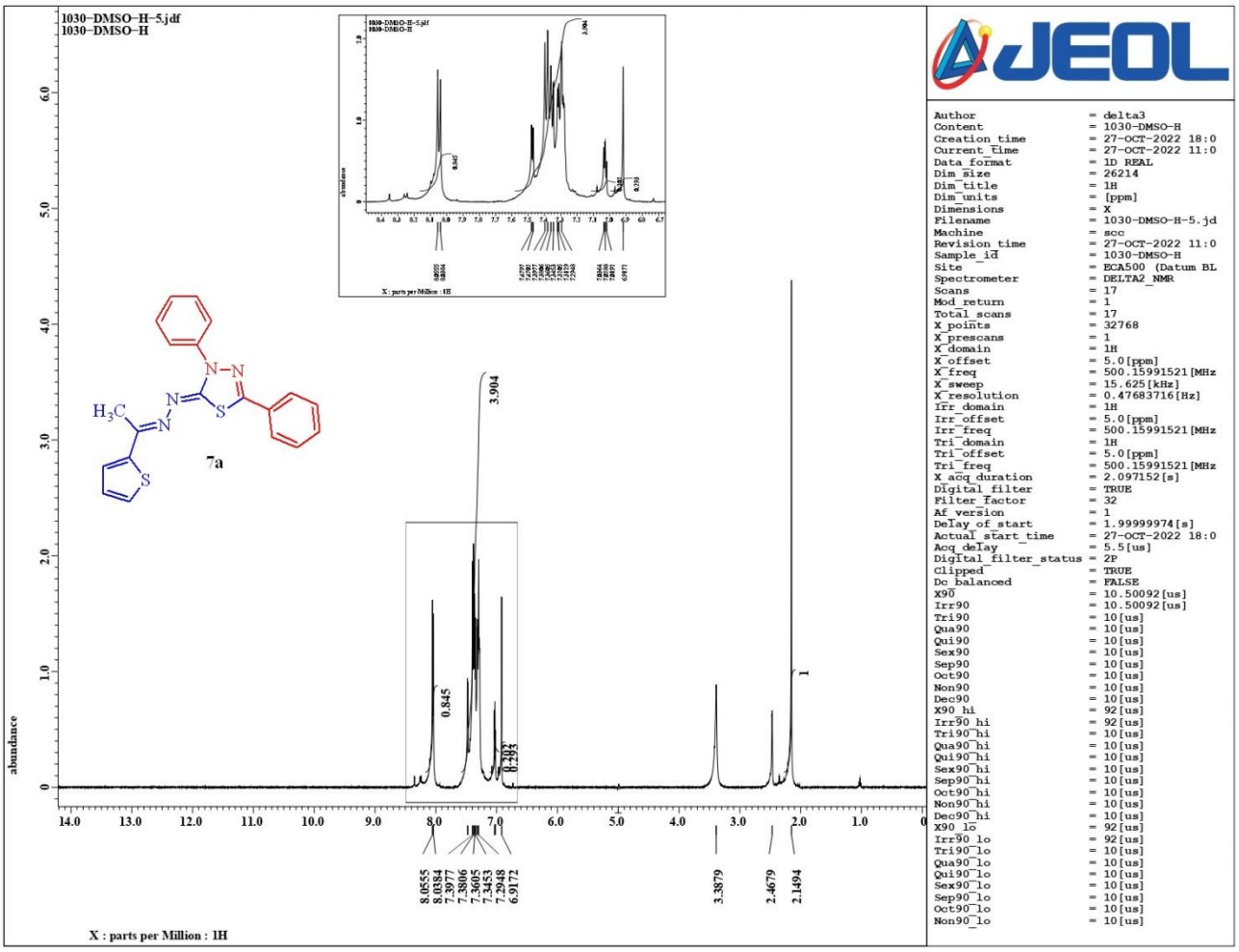
Line#:1 R.Time:3.1(Scan#:368)
MassPeaks:421
RawMode:Single 3.1(368) BasePeak:270(47320)
BG Mode:None Group 1 - Event 1



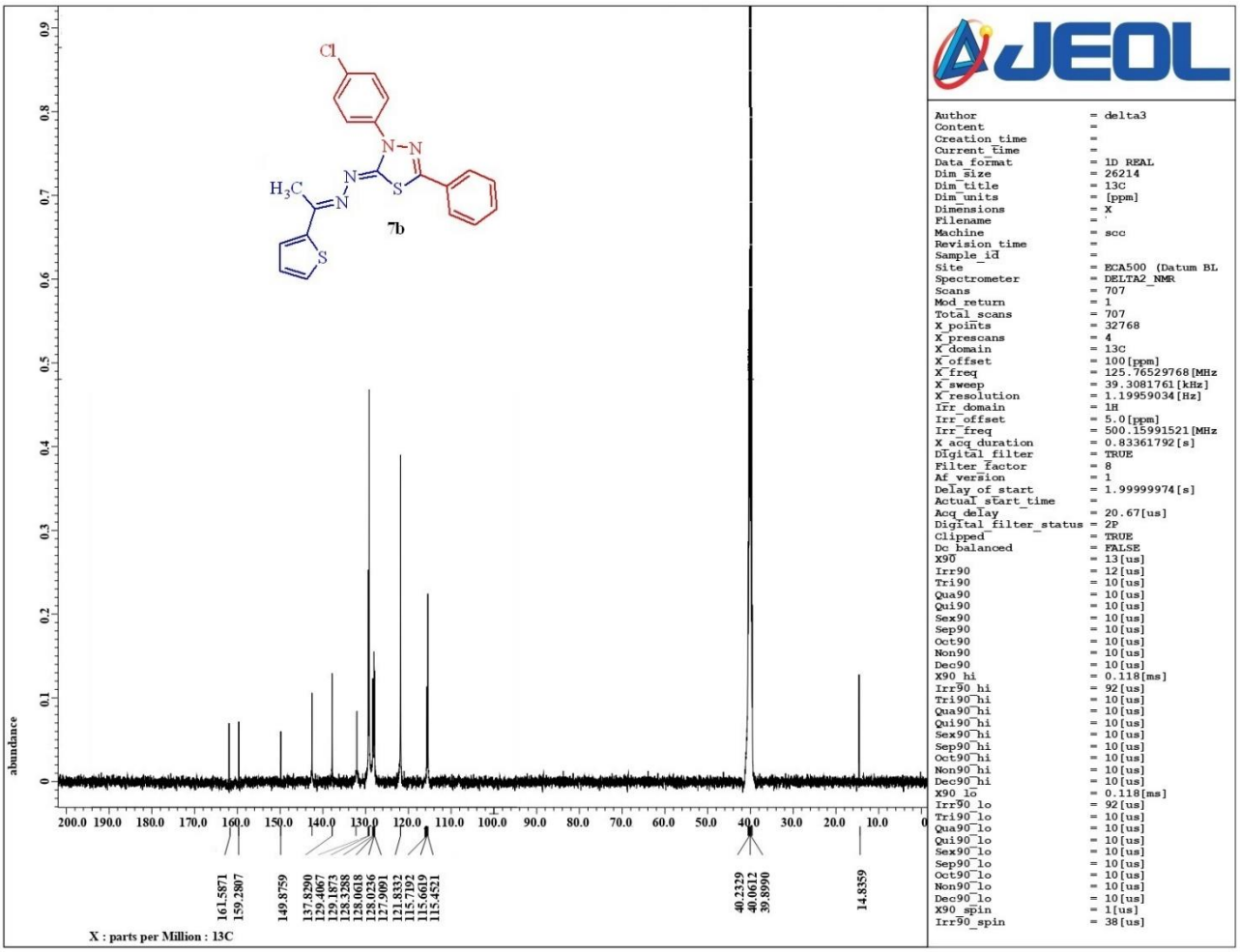
Mass Table
Line#:1 R.Time:3.1(Scan#:368)
MassPeaks:
RawMode:Single 3.1(368) BasePeak:270(47320)
BG Mode:None Group 1 - Event 1

#	m/z	Abs. In	Rel. Int.	#	m/z	Abs. In	Rel. Int.	#	m/z	Abs. In	Rel. Int.
1	50.05	11702	24.73	4	53.05	6522	13.78	7	56.10	27644	58.42
2	51.00	16204	34.24	5	54.10	8295	17.53	8	57.10	45449	96.05
3	52.05	10901	23.04	6	55.10	35964	76.00	9	58.10	15687	33.15

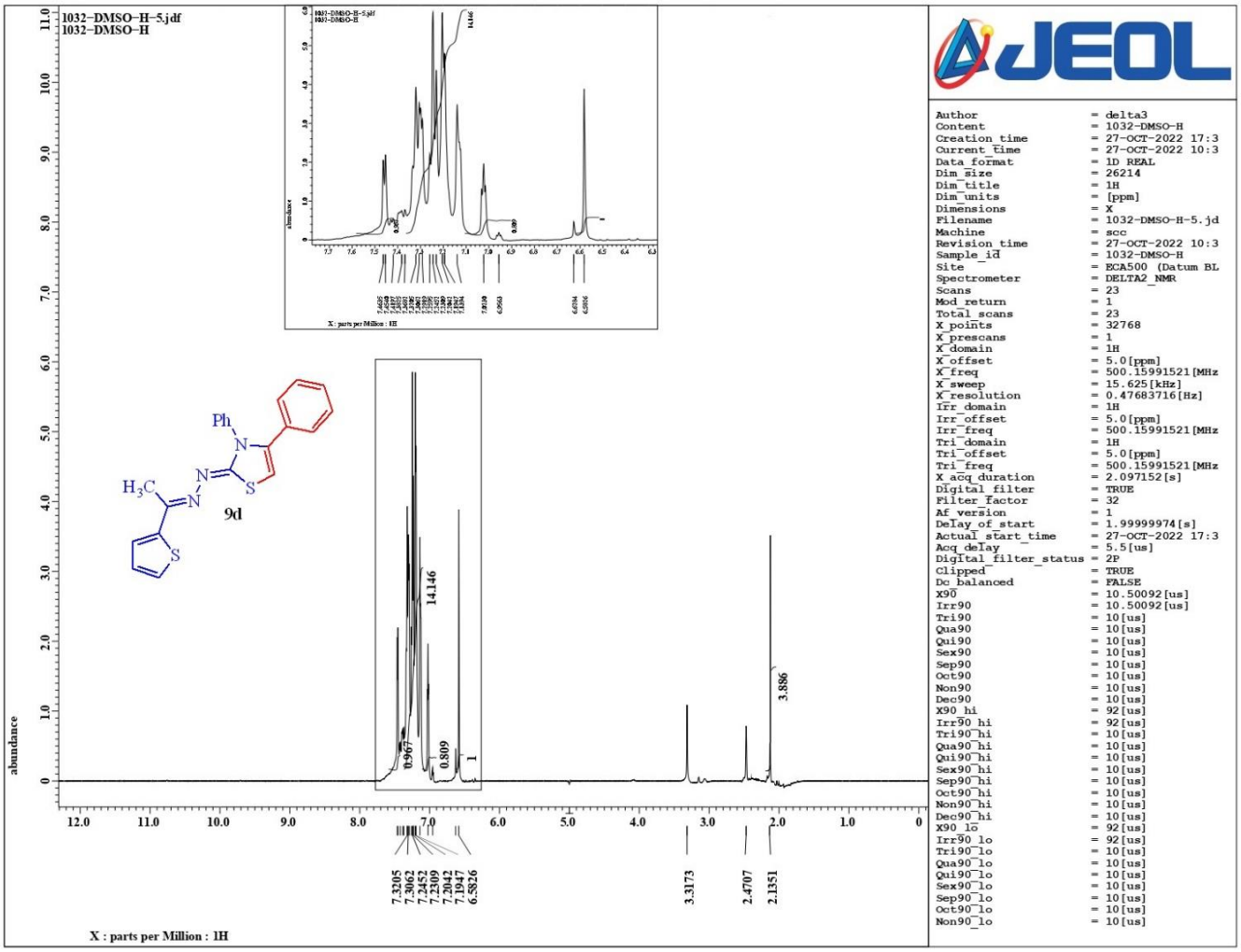
Mass spectra of compound **5h**



^{1}H-NMR spectra of compound **7a**



^{13}C-NMR spectra of compound **7b**



^{1}H-NMR spectra of compound **9d**

Cairo University
Micro Analytical Center

DI Analysis
Shimadzu Qp-2010 Plus

Sample Information

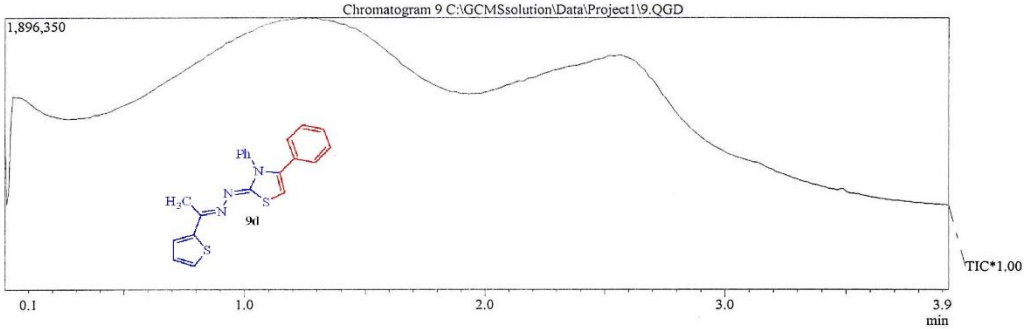
Analyzed by	: Dr. Mai Younis
Analyzed	:
Sample Name	: 9
Sample ID	:
Customer Name	: Dr. Sobhy Goma - Science - Cairo
Data File	: C:\GCMSsolution\Data\Project1\9.QGD
Org Data File	: C:\GCMSsolution\Data\Project1\9.QGD
Method File	: C:\GCMSsolution\Data\Project1\High Temperature Op
Org Method File	: C:\GCMSsolution\Data\Project1\High Temperature Op
Report File	:
Tuning File	: C:\GCMSsolution\System\Tune1_default.qgt
$EndIf$Modified by	: Dr. Mai Younis
Modified	:

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	:1250
Start m/z	:50.00
End m/z	:600.00
Electron Voltage	: 70 eV
Ionization Mode	: EI

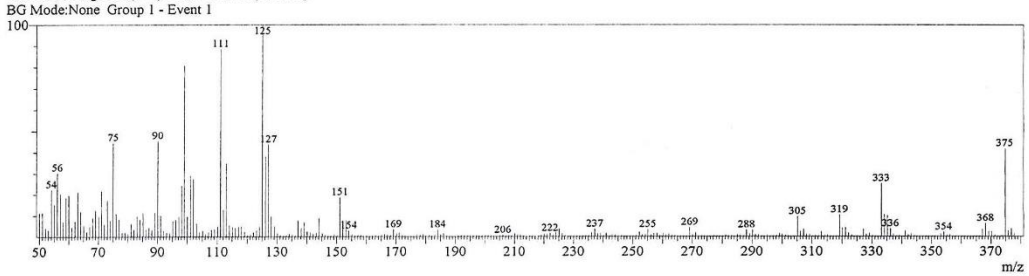
C:\GCMSsolution\Data\Project1\9.QGD

Chromatogram 9 C:\GCMSsolution\Data\Project1\9.QGD



Spectrum

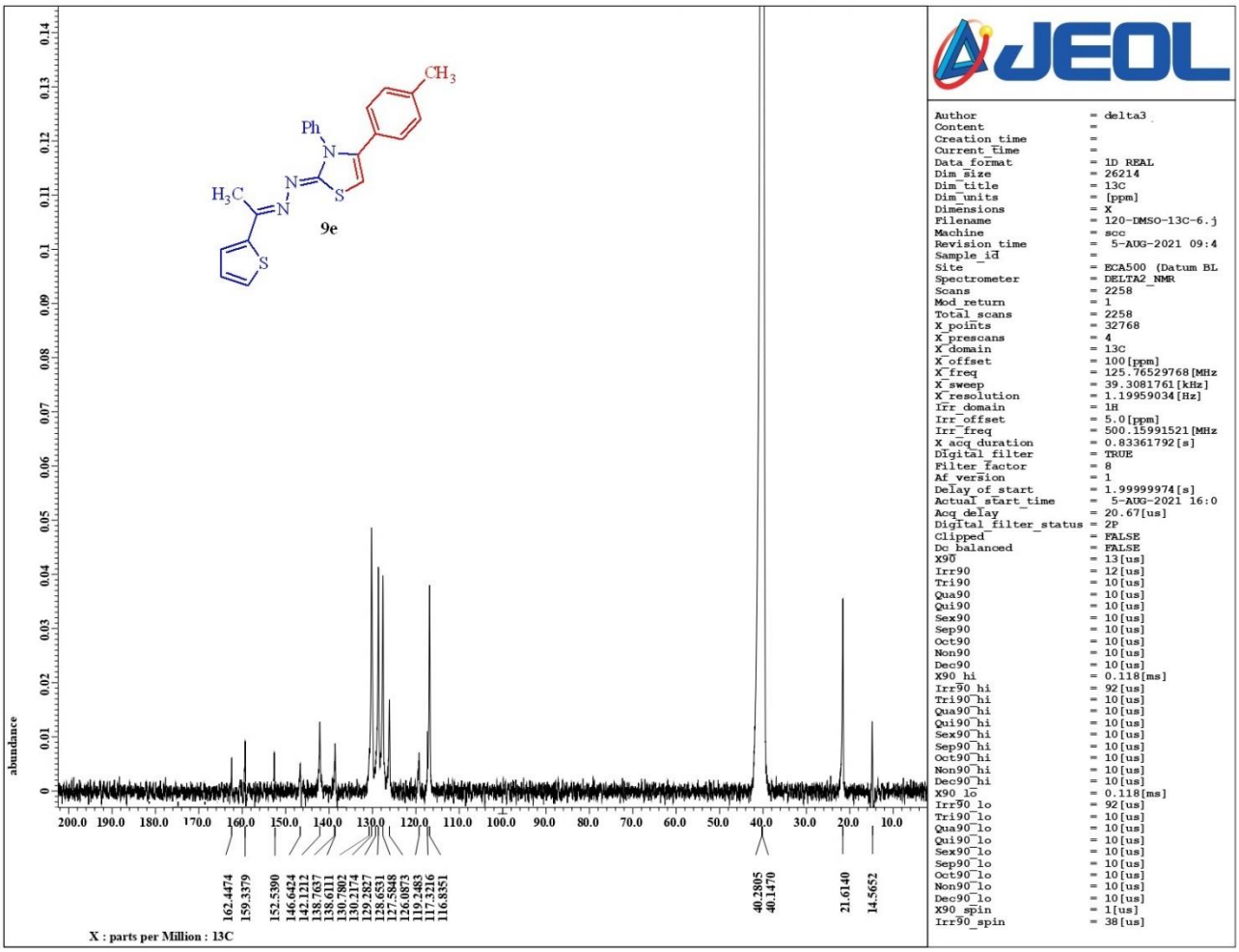
Line#:1 R.Time:1.2(Scan#:147)
MassPeaks:323
RawMode:Single 1.2(147) BasePeak:125(130775)
BG Mode:None Group 1 - Event 1



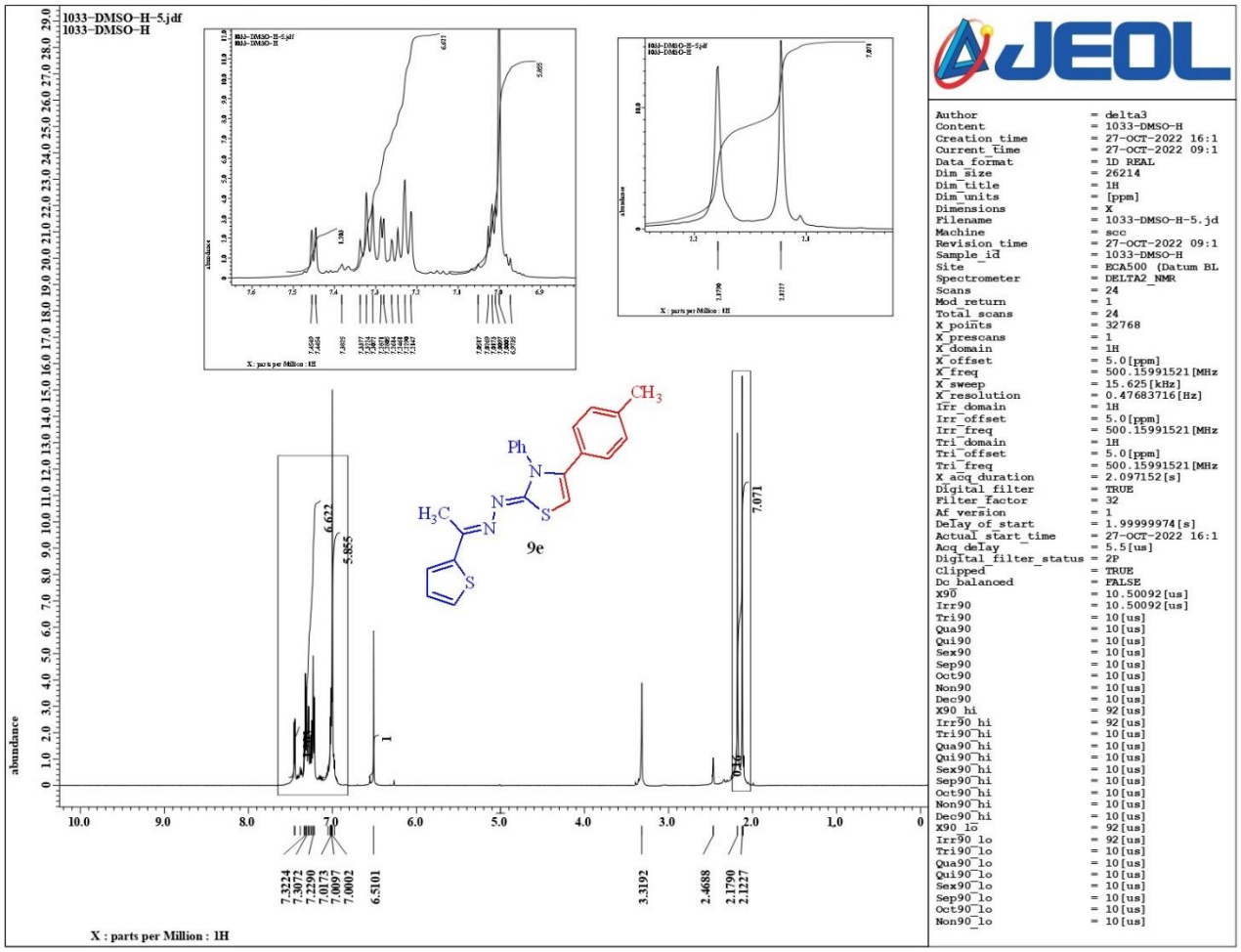
Mass Table
Line#:1 R.Time:1.2(Scan#:147)
MassPeaks:323
RawMode:Single 1.2(147) BasePeak:125(130775)
BG Mode:None Group 1 - Event 1

#	m/z	Abs. In	Rel. Int.	#	m/z	Abs. In	Rel. Int.	#	m/z	Abs. In	Rel. Int.
1	50.00	14993	11.46	4	53.05	3980	3.04	7	56.05	39539	30.23
2	51.05	14814	11.33	5	54.05	29093	22.25	8	57.10	26513	20.27
3	52.05	5476	4.19	6	55.10	19772	15.12	9	58.05	9223	7.05

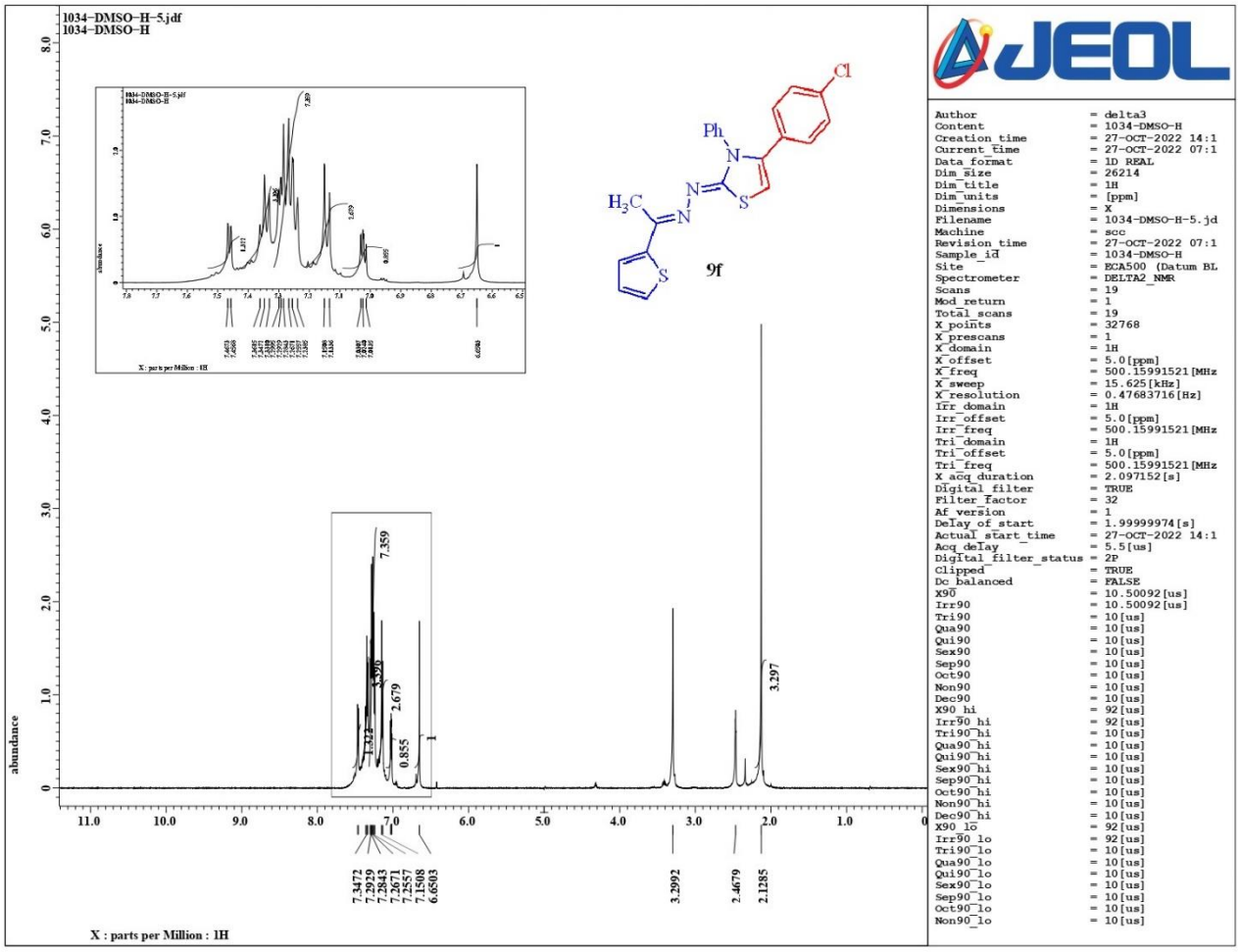
Mass spectra of compound **9d**



^{13}C-NMR spectra of compound **9e**



^{1}H-NMR spectra of compound **9e**



[1]H-NMR spectra of compound **9f**

Cairo University
Micro Analytical Center

DI Analysis
Shimadzu Qp-2010 Plus

Sample Information

Analyzed by	: Dr. Mai Younis
Analyzed	:
Sample Name	: 12
Sample ID	:
Customer Name	: Dr. Sobhy Goma - Science - Cairo
Data File	: C:\GCMSsolution\Data\Project1\12.QGD
Org Data File	: C:\GCMSsolution\Data\Project1\12.QGD
Method File	: C:\GCMSsolution\Data\Project1\High Temperature Op
Org Method File	: C:\GCMSsolution\Data\Project1\High Temperature Op
Report File	:
Tuning File	: C:\GCMSsolution\System\Tune1_default.qgt
$EndIf$Modified by	: Dr. Mai Younis
Modified	:

Method

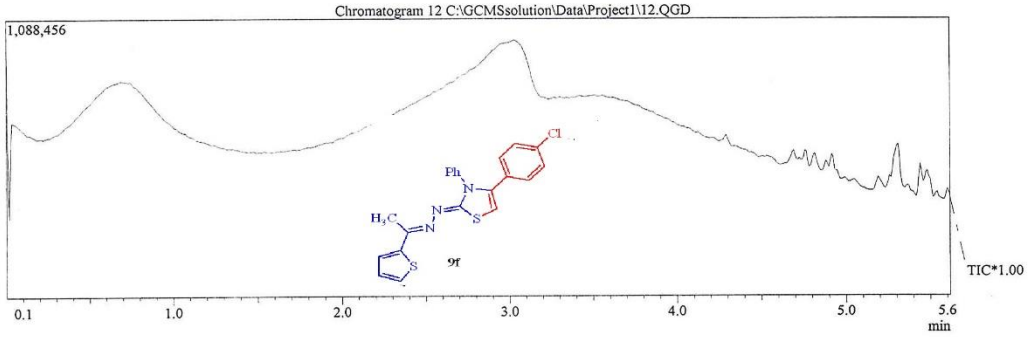
===== Analytical Line 1 =====

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	:1250
Start m/z	:50.00
End m/z	:600.00

Electron Voltage : 70 eV
Ionization Mode : EI

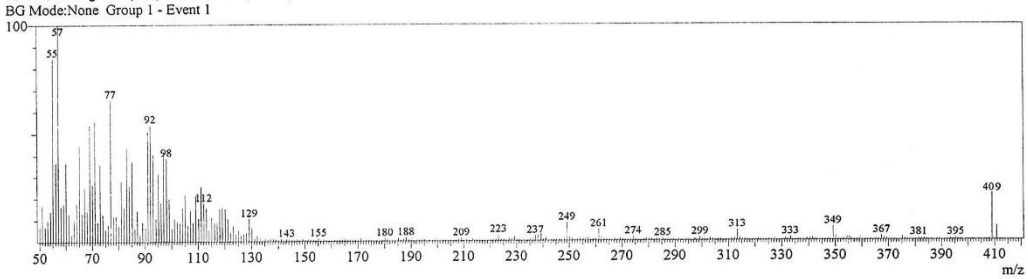
C:\GCMSsolution\Data\Project1\12.QGD

Chromatogram 12 C:\GCMSsolution\Data\Project1\12.QGD



Spectrum

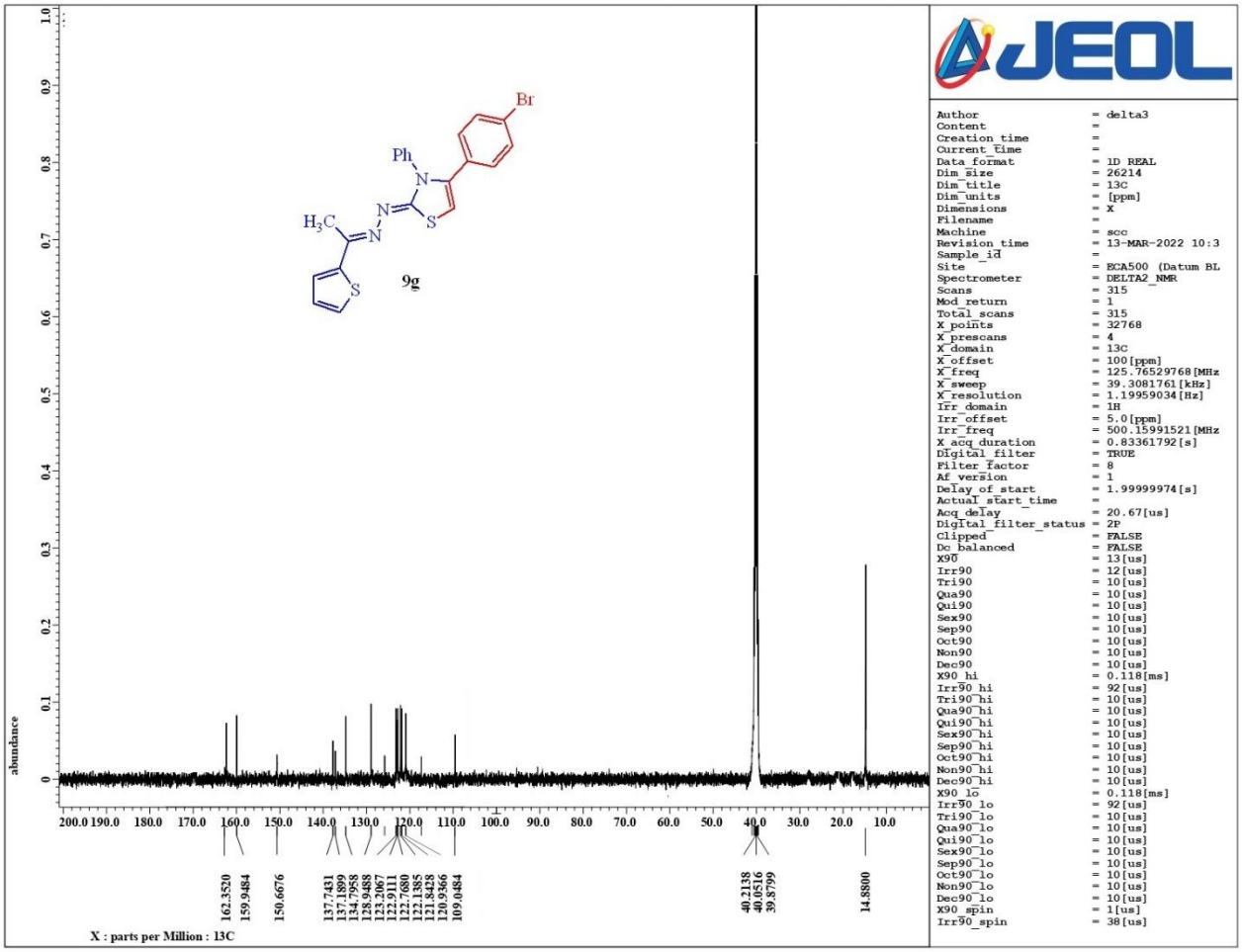
Line#:1 R.Time:3.0(Scan#:366)
MassPeaks:
RawMode:Single 3.0(366) BasePeak:57(53964)
BG Mode:None Group 1 - Event 1



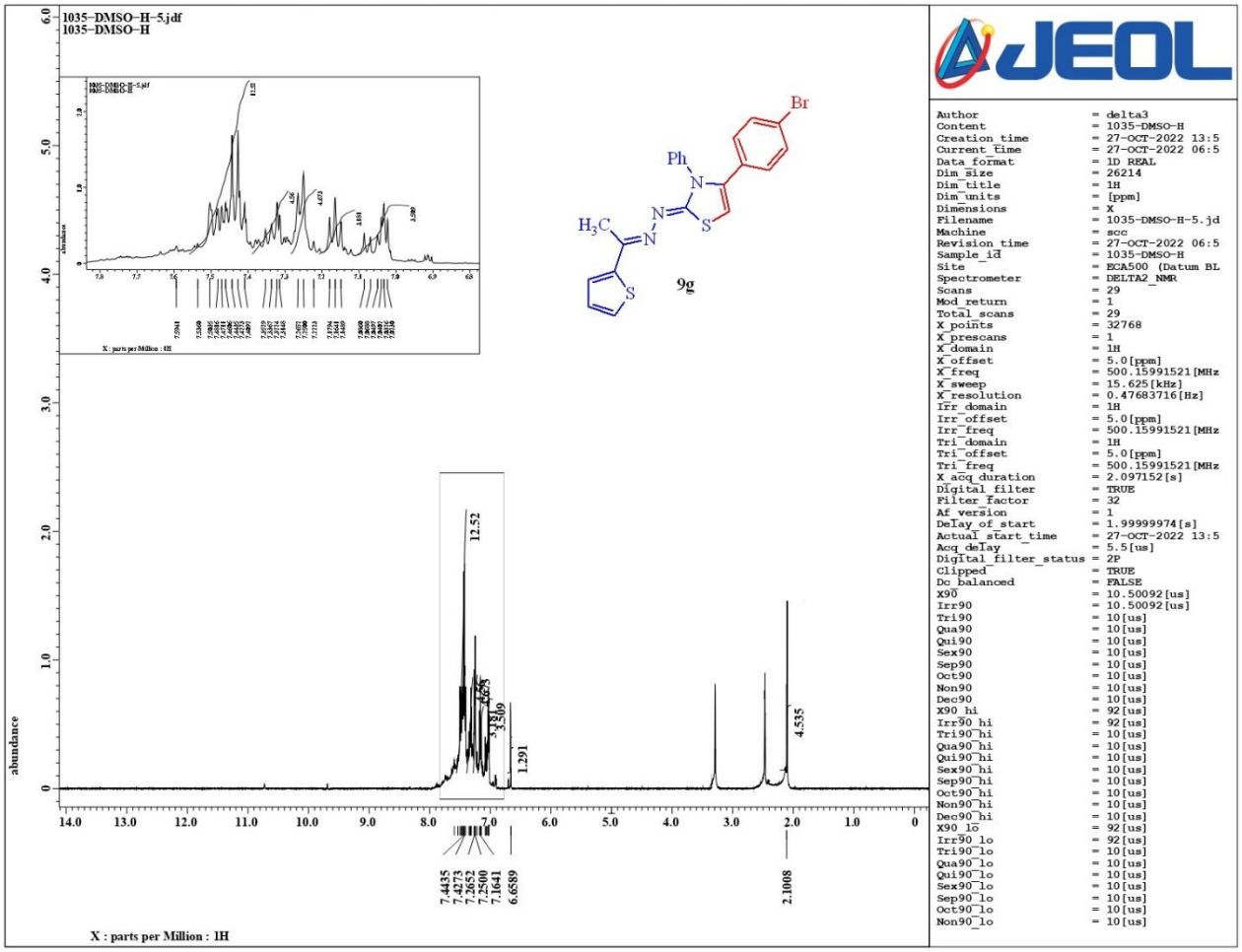
Mass Table
Line#:1 R.Time:3.0(Scan#:366)
MassPeaks:
RawMode:Single 3.0(366) BasePeak:57(53964)
BG Mode:None Group 1 - Event 1

#	m/z	Abs. In	Rel. Int.	#	m/z	Abs. In	Rel. Int.	#	m/z	Abs. In	Rel. Int.
1	50.00	3625	6.72	4	53.05	5268	9.76	7	56.10	19774	36.64
2	51.00	9014	16.70	5	54.10	7558	14.01	8	57.10	53964	100.00
3	52.05	3762	6.97	6	55.05	45651	84.60	9	58.10	8633	16.00

Mass spectra of compound **9f**



^{13}C-NMR spectra of compound **9g**



^{1}H-NMR spectra of compound **9g**

Cairo University
Micro Analytical Center

DI Analysis
Shimadzu Qp-2010 Plus

Sample Information

Analyzed by	: Dr. Mai Younis
Analyzed	:
Sample Name	: 14
Sample ID	:
Customer Name	: Dr. Sobhy Goma - Science - Cairo
Data File	: C:\GCMSsolution\Data\Project1\14.QGD
Org Data File	: C:\GCMSsolution\Data\Project1\14.QGD
Method File	: C:\GCMSsolution\Data\Project1\High Temperature Op
Org Method File	: C:\GCMSsolution\Data\Project1\High Temperature Op
Report File	:
Tuning File	: C:\GCMSsolution\System\Tune1_default.qgt
$EndIf$Modified by	: Dr. Mai Younis
Modified	:

Method

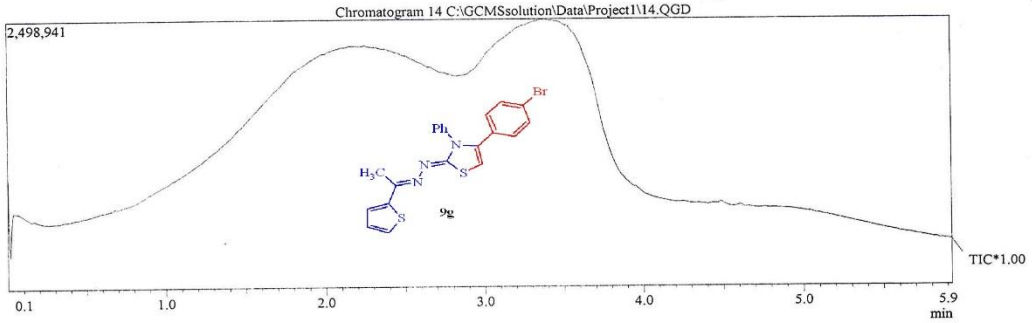
====== Analytical Line 1 ======

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	:1250
Start m/z	:50.00
End m/z	:600.00

Electron Voltage : 70 eV
Ionization Mode : EI

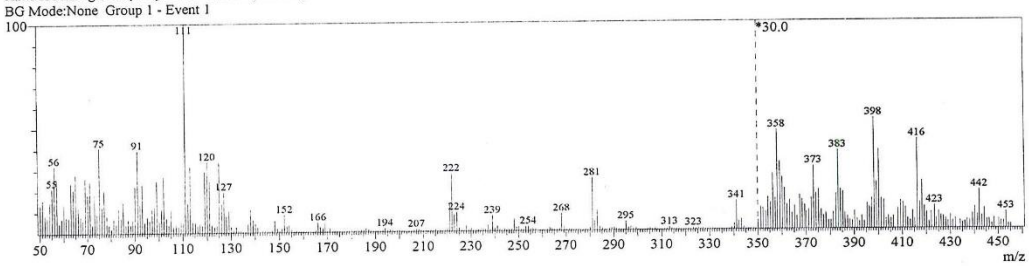
C:\GCMSsolution\Data\Project1\14.QGD

Chromatogram 14 C:\GCMSsolution\Data\Project1\14.QGD



Spectrum

Line#:1 R.Time:3.5(Scan#:416)
MassPeaks:
RawMode:Single 3.5(416) BasePeak:111(161583)
BG Mode:None Group 1 - Event 1

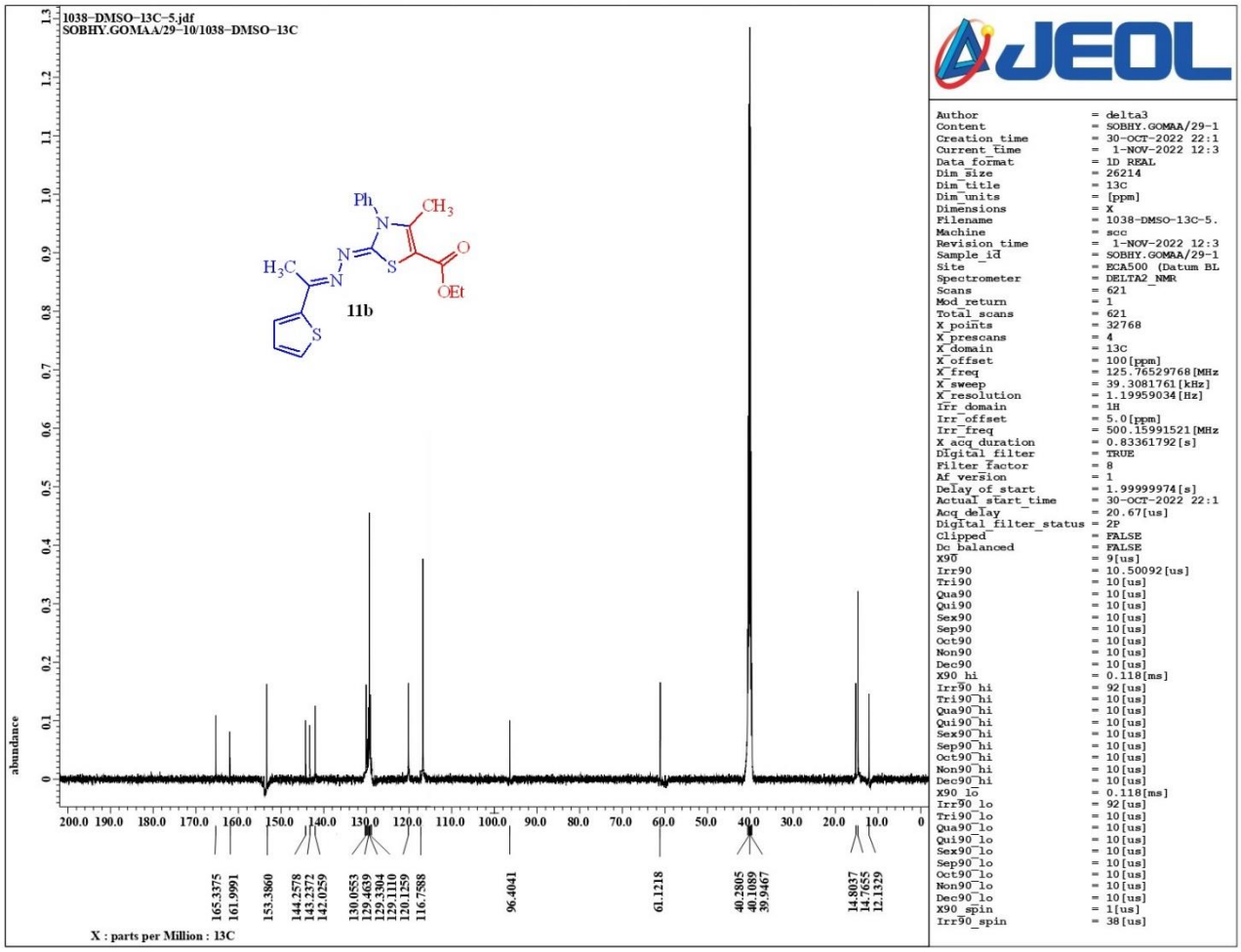


Mass Table
Line#:1 R.Time:3.5(Scan#:416)
MassPeaks:
RawMode:Single 3.5(416) BasePeak:111(161583)
BG Mode:None Group 1 - Event 1

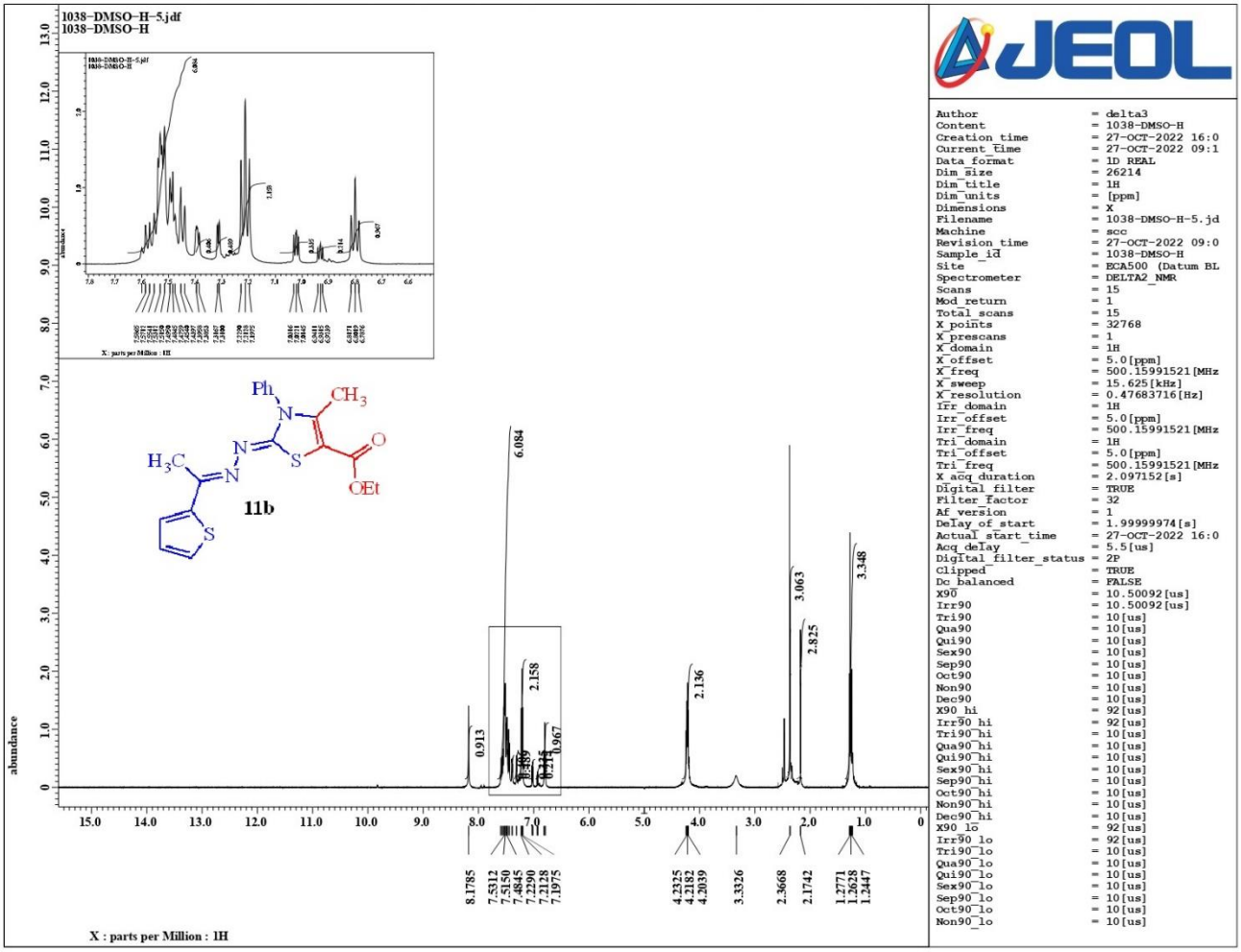
#	m/z	Abs. In	Rel. Int.	#	m/z	Abs. In	Rel. Int.	#	m/z	Abs. In	Rel. Int.
1	50.05	21798	13.49	4	53.05	12176	7.54	7	56.05	51948	32.15
2	51.05	25816	15.98	5	54.05	23840	14.75	8	57.10	42182	26.11
3	52.05	14096	8.72	6	55.05	34620	21.43	9	58.05	7632	4.72

1 / 3

Mass spectra of compound **9g**



^{13}C-NMR spectra of compound **11b**



^{1}H-NMR spectra of compound **11b**

السيد الاستاذ الدكتور عميد كلية العلوم – جامعة القاهرة

تحية طيبة وبعد

أفيد سيادتكم علما بأن نتيجة التحاليل المرسلة من السيد / أ. د. صبحى جمعة

جاءت على النحو التالى:

Results of analysis

Sample Cpd	C, %	H, %	N, %	Cl, %	S, %	Remarks
5e	63.15	4.38	16.57			
5g	61.53	4.86	15.47			
5h	58.37	4.14	15.35			
7a	63.64	4.19	14.81			
9d	67.04	4.41	11.13			
9e	67.96	4.82	10.68			
9g	55.39	3.40	9.16			
11b	59.12	4.79	10.74			

وتفضلوا بقبول فائق الاحترام ،،،،،

التاريخ : 2022-9-18

مدير مركز التحاليل الدقيقة

أ.د. رهام تمام حلمى

Micro Analytical Center
Faculty of Science - Cairo University
Tel.: 35676897-35676898 - 35676899 - 35713208 - Fax: +202 35713208
e-mail: microcentercairo@hotmail.com