Supplementary Information

Synthesis, *In-Silico* Modelling and *In-Vitro* Biological Evaluation of Substituted Pyrazole Derivatives as Potential Anti-Skin Cancer, Anti-Tyrosinase, and Antioxidant Agents

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Running Title: Identification of potent anticancer, antioxidant and anti-tyrosinase pyrazole derivatives

‡ These authors contributed equally to this work.

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Table of Contents Pages

Table S1. The summary IC_{50} values of mushroom tyrosinase inhibition assay of pyrazoles, pyrazolones, isoxazoles and positive control drugs P1-P25 > 500 = IC_{50} Value is greater than 500 μM, and therefore not biologically relevant.

Sample Identity	**IC_{50} Value (μM)**
P1	> 500
P2	> 500
P3	> 500
P4	196.9
P5	> 500
P6	> 500
P7	> 500
P8	190.4
P9	> 500
P10	> 500
P11	172.8
P12	335.5
P13	> 500
P14	15.9
P15	> 500
P16	> 500
P17	165.4
P18	156.0
P19	> 500
P20	> 500
P21	294.8
P22	> 500
P23	> 500
P24	> 500
P25	> 500
Arbutin	91
Kojic acid	31

Table S2. Physicochemical properties of pyrazoles, pyrazolones, and isoxazoles P1-25

Compounds	MW	#Heavy atoms	#Aromatic heavy atoms	Fraction Csp3	#Rotatable bonds		#H-bond acceptors	#H-bond donors	MR
P1	124.18	9	5	0.57	1		1	1	38.29
P2	122.17	9	5	0.57	0		1	1	36.18
P3	158.2	12	6	0.2	1		2	0	57.74
P4	192.64	13	11	0.1	1		1	1	54
P5	184.24	14	11	0.25	0		1	1	56.81
P6	182.22	14	13	0.08	0		1	1	58.57
P7	200.28	15	11	0.31	2		1	0	63.27
P8	198.26	15	11	0.31	1		1	0	61.16
P9	260.33	20	17	0.17	1		1	0	81.78
P10	244.29	18	11	0.29	4		3	0	69.58
P11	159.18	12	11	0.1	1		2	0	46.91
P12	185.22	14	11	0.25	0		2	0	54.72
P13	296.37	23	23	0	3		1	0	94.44
P14	234.3	18	17	0.06	2		1	0	73.97
P15	288.27	21	17	0.06	3		4	0	74
P16	313.19	19	17	0.06	2		1	0	81.67
P17	268.74	19	17	0.06	2		1	0	78.98
P18	264.32	20	17	0.12	3		2	0	80.46
P19	313.37	22	17	0.06	3		4	1	84.96
P20	188.23	14	6	0.27	1		2	0	63.1
P21	236.27	18	12	0.07	2		2	0	77.98
P22	394.06	20	12	0.07	2		2	0	93.37
P23	383.16	23	12	0.12	3		5	0	90.68
P24	315.16	19	12	0.07	2		2	0	85.67
P25	315.16	19	12	0.07	2		2	0	85.67

Table S3: Physicochemical, pharmacokinetics (SwissADME), and toxicologic properties of the synthesized compounds.

			Lipophilicity						**Drug-likeness** (number of violations)					**Water Solubility**		**Pharmacokinetics**	
Comp.	**MW** g/mol	TPSA	ilogP	Xlog P3	Wlog P	Mlog P	Silicos -IT logP	Consensus logP	Lipinski	Ghose	Veber	Egan	Muegge	ESOL Log S	ESOL Class	Log K_p (cm/s)	F
1	124.18	28.68	1.32	1.81	1.59	1.08	2.55	1.67	0	2	0	0	1	-2.1	Soluble	-5.77	0.55
2	122.17	28.68	1.23	1.3	1.21	1.08	2.65	1.49	0	3	0	0	1	-1.83	Very soluble	-6.12	0.55
3	158.2	24.72	1.69	1.76	1.49	1.81	3.79	2.11	0	1	0	0	1	-2.23	Soluble	-6.02	0.55
4	192.64	28.68	1.75	2.91	3.04	2.33	3.66	2.74	0	0	0	0	1	-3.43	Soluble	-5.41	0.55
5	184.24	28.68	1.64	2.57	2.48	2.34	3.7	2.55	0	0	0	0	1	-3.18	Soluble	-5.6	0.55
6	182.22	28.68	1.63	3.2	3.02	2.45	3.53	2.77	0	0	0	0	1	-3.67	Soluble	-5.14	0.55
7	200.28	17.82	2.74	3.49	3.05	2.88	3.27	3.09	1	0	0	0	0	-3.69	Soluble	-5.04	0.55
8	198.26	17.82	2.56	2.98	2.67	2.88	3.13	2.84	0	0	0	0	1	-3.42	Soluble	-5.39	0.55
9	260.33	17.82	3.07	4.25	3.95	3.89	4.19	3.87	0	0	0	0	0	-4.69	Moderate	-4.87	0.55
10	244.29	44.12	2.99	3.13	2.67	2.41	2.69	2.78	0	0	0	0	0	-3.51	Soluble	-5.57	0.55
11	159.18	26.03	2.22	2.43	2.65	1.77	2.93	2.4	0	1	0	0	1	-2.97	Soluble	-5.55	0.55
12	185.22	26.03	2.39	2.73	2.75	2.34	3.57	2.76	0	0	0	0	1	-3.29	Soluble	-5.49	0.55
13	296.37	17.82	3.4	5.22	5.21	4.35	4.58	4.55	1	0	0	0	1	-5.51	Moderate	-4.4	0.55
14	234.3	17.82	2.91	3.95	3.85	3.41	3.55	3.53	0	0	0	0	0	-4.35	Moderate	-4.92	0.55
15	288.27	17.82	2.94	4.47	5.71	4.04	4.02	4.24	0	1	0	0	0	-4.84	Moderate	-4.88	0.55
16	313.19	17.82	3.3	4.64	4.61	4.04	4.19	4.16	0	0	0	0	0	-5.24	Moderate	-4.92	0.55
17	268.74	17.82	3.18	4.58	4.5	3.92	4.16	4.07	0	0	0	0	0	-4.92	Moderate	-4.69	0.55
18	264.32	27.05	3.23	3.92	3.86	3.04	3.55	3.52	0	0	0	0	0	-4.38	Moderate	-5.13	0.55
19	313.37	86.36	2.21	2.52	3.58	1.78	1.58	2.33	0	0	0	0	0	-3.74	Soluble	-6.42	0.55
20	188.23	32.67	2.3	1.84	1.28	1.89	2.24	1.91	0	0	0	0	1	-2.42	Soluble	-6.14	0.55
21	236.27	32.67	2.55	2.93	2.07	2.73	3.27	2.71	1	0	0	0	0	-3.51	Soluble	-5.66	0.55
22	394.06	32.67	3.3	4.31	3.59	3.99	4.57	3.95	0	0	0	0	0	-5.31	Moderate	-5.64	0.55

23	383.16	32.67	3.19	4.5	5	4.22	4.91	4.37	1	0	0	0	0	-5.24	Moderate	-5.44	0.55
24	315.16	32.67	2.9	3.62	2.83	3.37	3.92	3.33	0	0	0	0	0	-4.41	Moderate	-5.65	0.55
25	315.16	32.67	2.96	3.62	2.83	3.37	3.92	3.34	0	0	0	0	0	-4.41	Moderate	-5.65	0.55

Molecular weight (MW), LogP values (an indicator of lipophilicity), aqueous solubility parameters (ESOL), an indicator of skin permeation (Log K_p), and bioavailability score (F)
HBA: number of hydrogen bond acceptor; HBD: number of hydrogen bond donor, LogP: partition coefficient; LogS: lipophilicity of water; PSA: total polar surface area; RO5: Lipinski's rule of 5

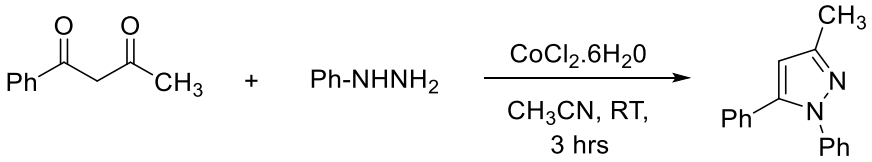
Table S4. Summary IC_{50} data of the DPPH antioxidant activity screening of pyrazoles, pyrazolones and isoxazoles drugs P1-P25. N.A. = No Activity, > 1000 = IC_{50} Value is greater than 1000 μM, and therefore not biologically relevant.

Sample Identity	**IC_{50} Value (μM)**
P1	> 1000
P2	N.A.
P3	N.A.
P4	N.A.
P5	N.A.
P6	30.56
P7	> 1000
P8	N.A.
P9	>1000
P10	N.A.
P11	N.A.
P12	N.A.
P13	>1000
P14	N.A.
P15	N.A.
P16	N.A.
P17	N.A.
P18	N.A.
P19	N.A.
P20	N.A.
P21	354.5
P22	276.3
P23	196.4
P24	444.4
P25	189.6

Table S5. Computational prediction of some pharmacokinetic properties of the pyrazoles, pyrazolones, and isoxazoles P1-P25.

Compound	GI absorption	BBB permeant	Pgp substrate	CYP1A2 inhibitor	CYP2C19 inhibitor	CYP2C9 inhibitor	CYP2D6 inhibitor	CYP3A4 inhibitor
P1	High	Yes	No	No	No	No	No	No
P2	High	Yes	Yes	No	No	No	No	No
P3	High	Yes	No	Yes	No	No	No	No
P4	High	Yes	No	Yes	No	No	No	No
P5	High	Yes	Yes	Yes	No	No	No	No
P6	High	Yes	Yes	Yes	No	No	No	No
P7	High	Yes	No	Yes	Yes	No	Yes	No
P8	High	Yes	Yes	Yes	No	No	No	No
P9	High	Yes	Yes	Yes	Yes	No	Yes	No
P10	High	Yes	No	Yes	Yes	Yes	No	No
P11	High	Yes	No	Yes	No	No	No	No
P12	High	Yes	Yes	Yes	No	No	No	No
P13	High	Yes	Yes	Yes	Yes	No	Yes	Yes
P4	High	Yes	No	Yes	Yes	No	Yes	No
P15	High	No	No	Yes	Yes	No	Yes	No
P16	High	Yes	No	Yes	Yes	Yes	Yes	No
P17	High	Yes	No	Yes	Yes	Yes	Yes	No
P18	High	Yes	No	Yes	Yes	Yes	Yes	Yes
P19	High	No	No	Yes	No	No	No	Yes
P20	High	Yes	No	Yes	No	No	No	No
P21	High	Yes	No	Yes	Yes	No	No	No
P22	High	Yes	No	Yes	Yes	Yes	No	No
P23	High	Yes	No	Yes	Yes	Yes	No	No
P24	High	Yes	No	Yes	Yes	Yes	No	No
P25	High	Yes	No	Yes	Yes	Yes	No	No

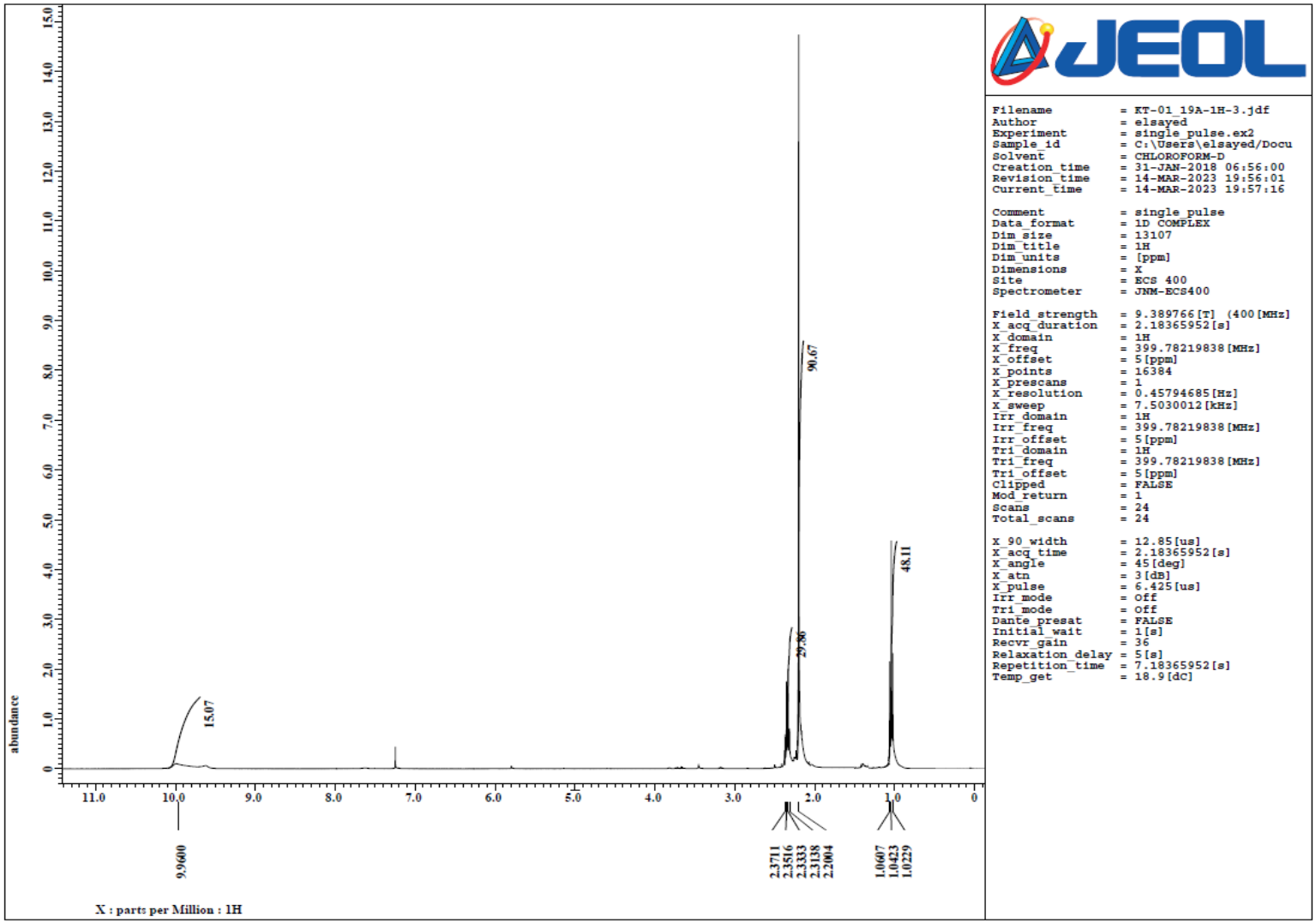
Table S6. Screening of Base-metal catalysts with different solvents



S. No.	Catalyst	Solvent	% Yield
1	$Ni^{II}Cl_2$	CH_3CN	53
2	$Ni^{II}(OTf)_2$	CH_3CN	43
3	$Ni^{II}(acac)_2$	CH_3CN	69
4	$Co^{II}Cl_2.6H_2O$	CH_3CN	~100
5	$Co^{II}SO_4$	CH_3CN	94
6	$Cu^{II}(BF_4)_2.H_2O$	CH_3CN	90
7	$Cu^{II}(OTf)_2$	CH_3CN	94
8	No catalyst	CH_3CN	11
9	$Co^{II}Cl_2.6H_2O$	CH_2Cl_2	94
10	$Co^{II}Cl_2.6H_2O$	Dioxane	86
11	$Co^{II}Cl_2.6H_2O$	DMF	60
12	$Co^{II}Cl_2.6H_2O$	Methanol	>99

^{1}H- and ^{13}C-NMR, and Mass Spectra of the Products P1-P19. We confirmed the products P20-P25 by comparing the data we reported earlier (ref#79 in the manuscript).

4-ethyl-3,5-dimethyl-1H-pyrazole (P1): ^{1}H-NMR ($CDCl_3$)



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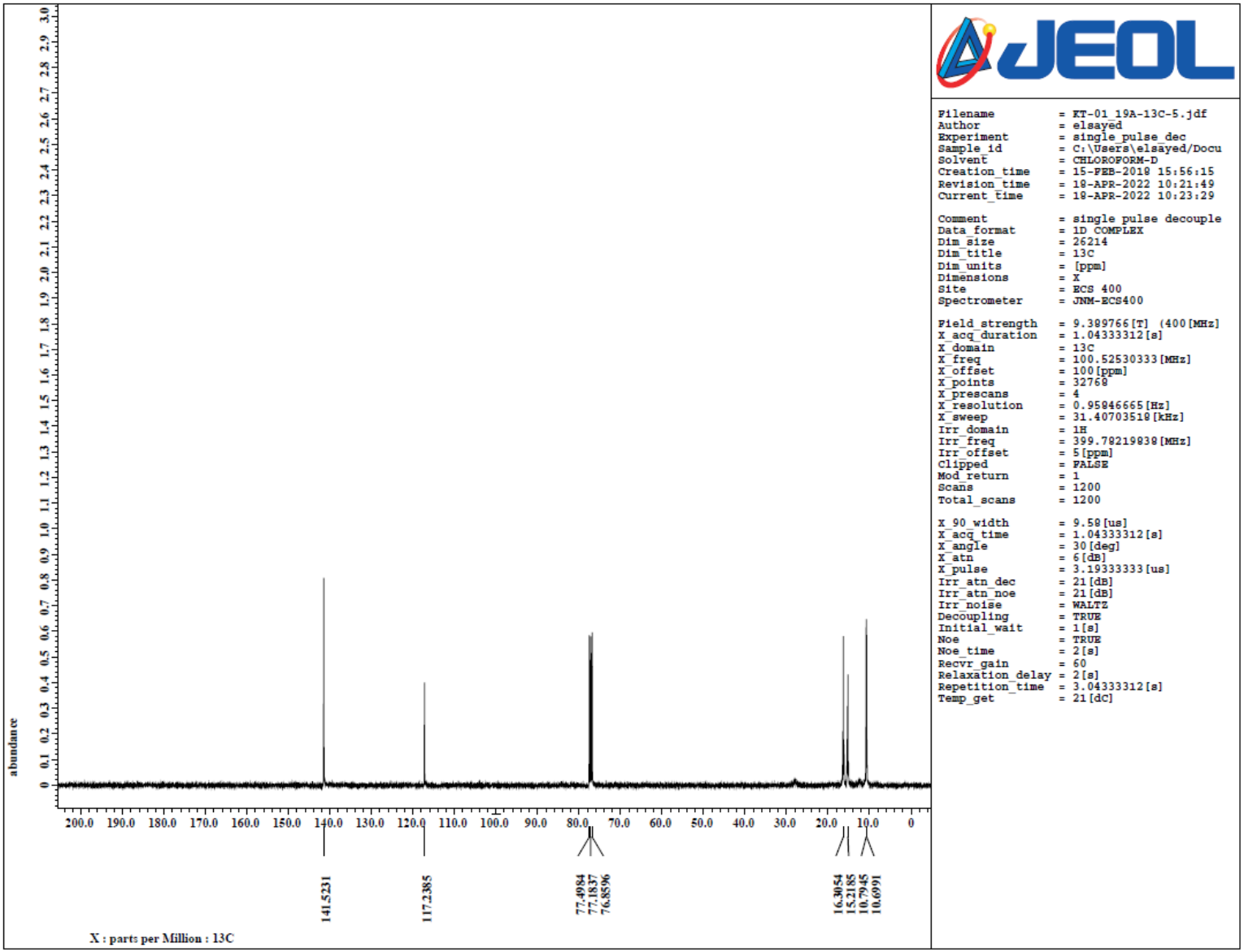


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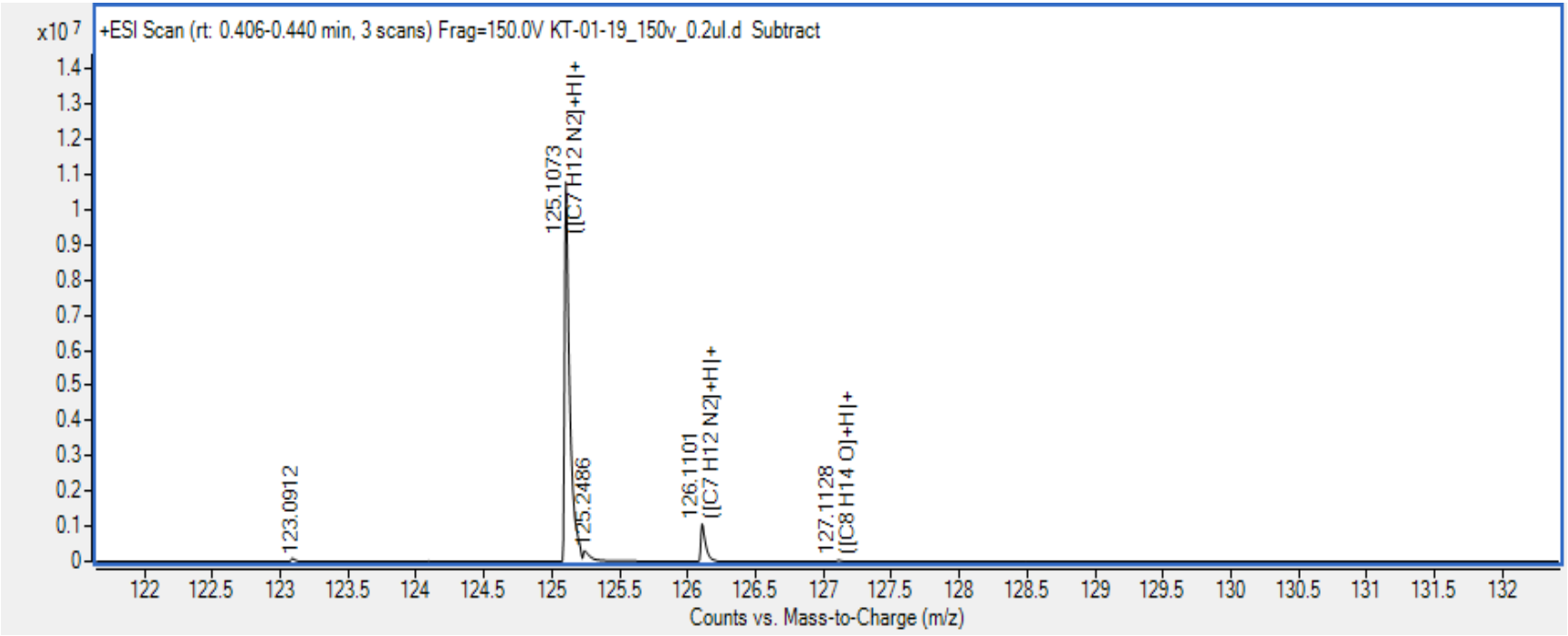

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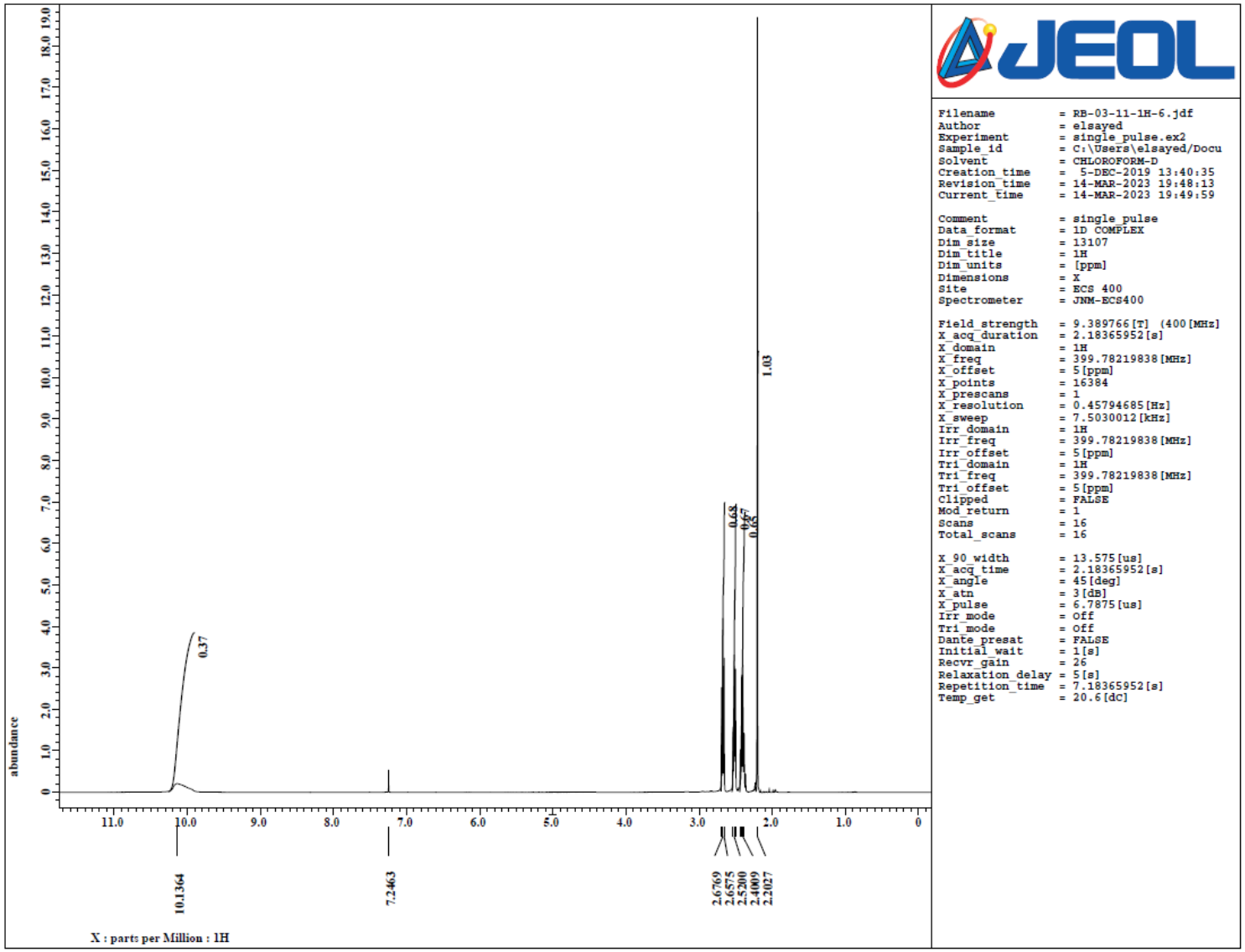
3-methyl-1,4,5,6-tetrahydrocyclopenta[c]pyrazole (P2): ^{1}H-NMR ($CDCl_3$)

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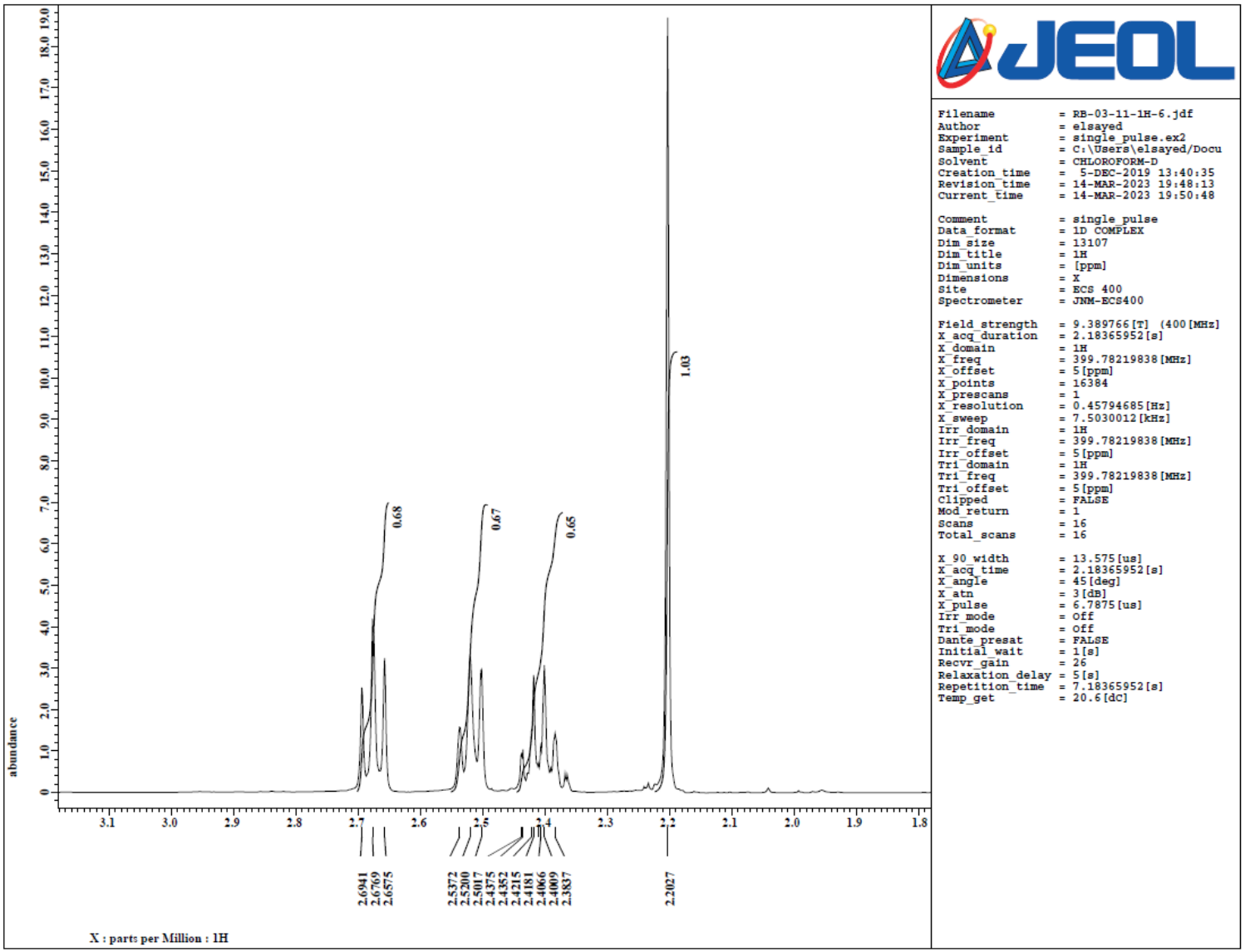
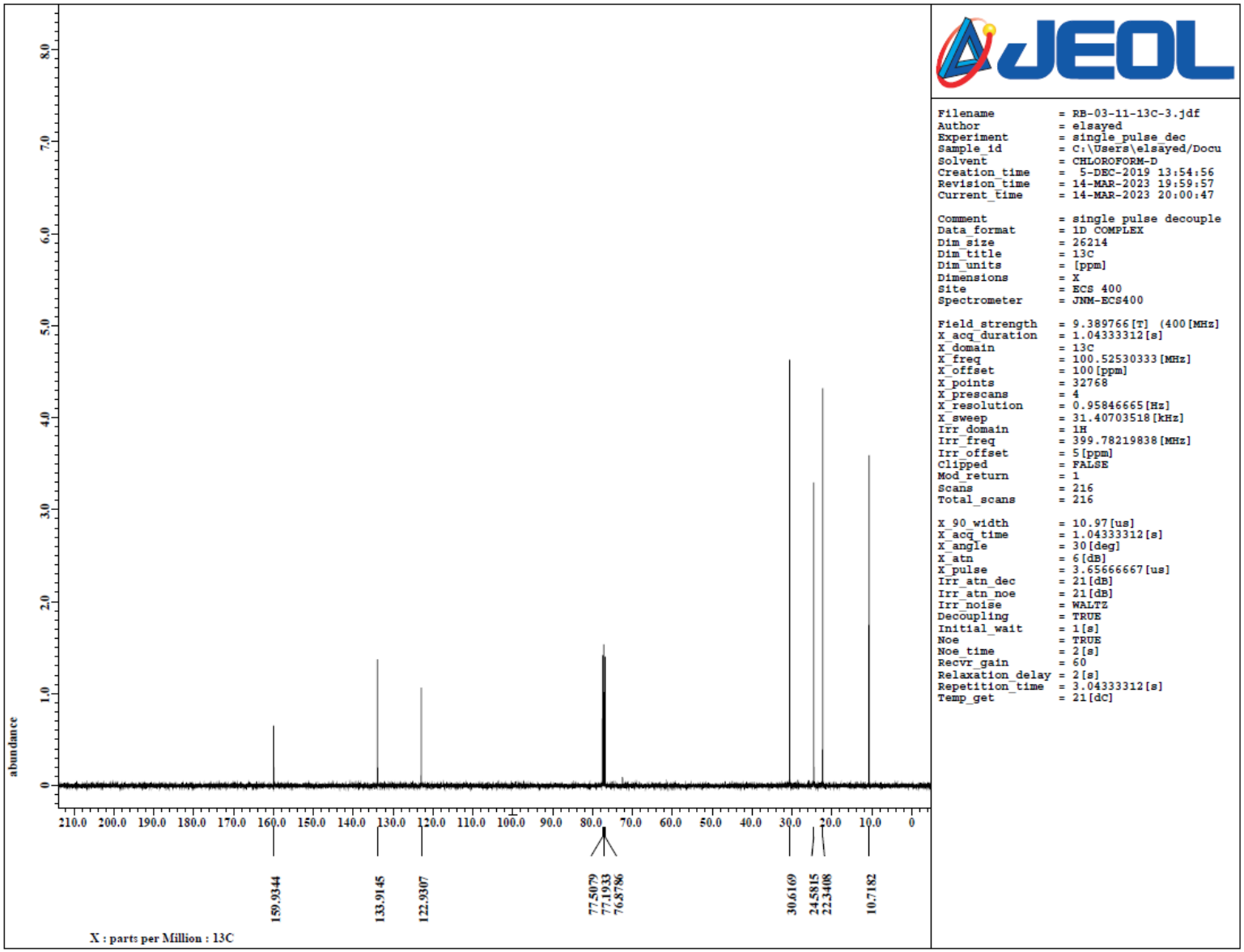
3-methyl-1,4,5,6-tetrahydrocyclopenta[c]pyrazole (P2): ^{1}H-NMR ($CDCl_3$) _ Expanded Aliphatic Region



3-methyl-1,4,5,6-tetrahydrocyclopenta[c]pyrazole (P2): ^{13}C-NMR ($CDCl_3$)



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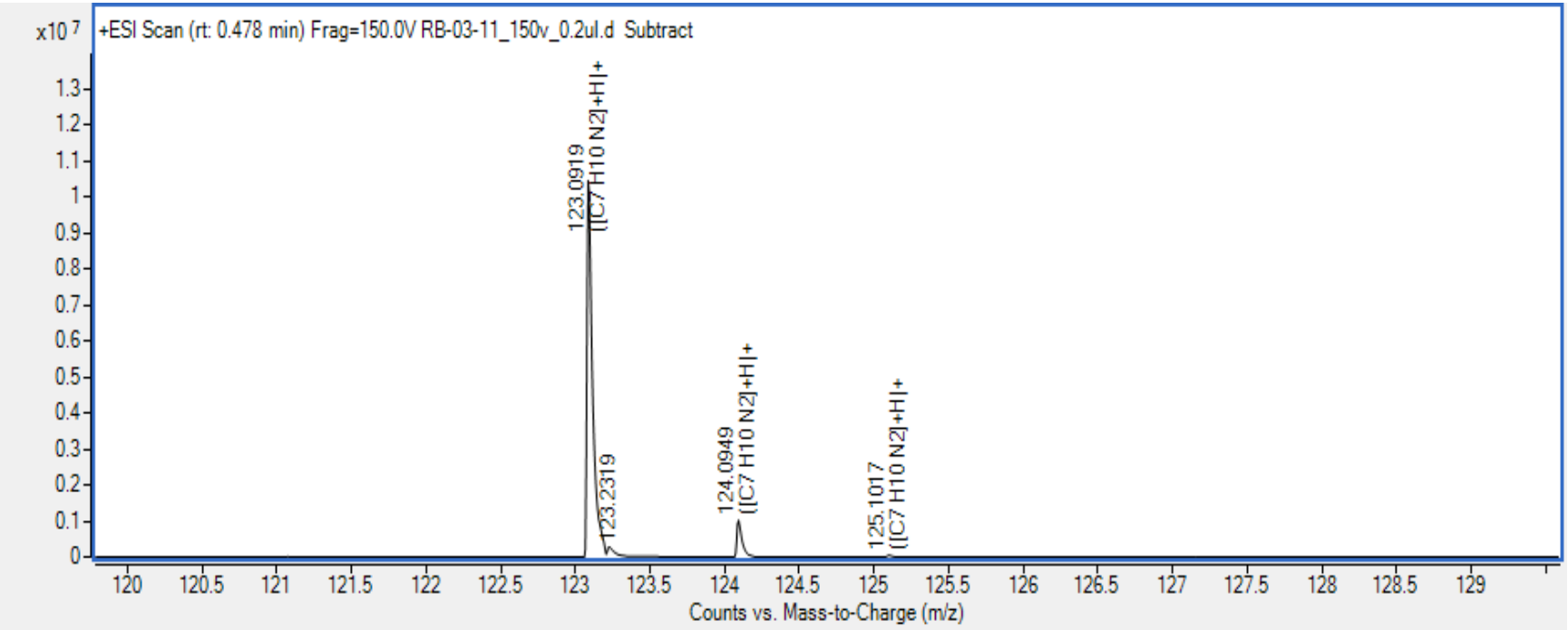

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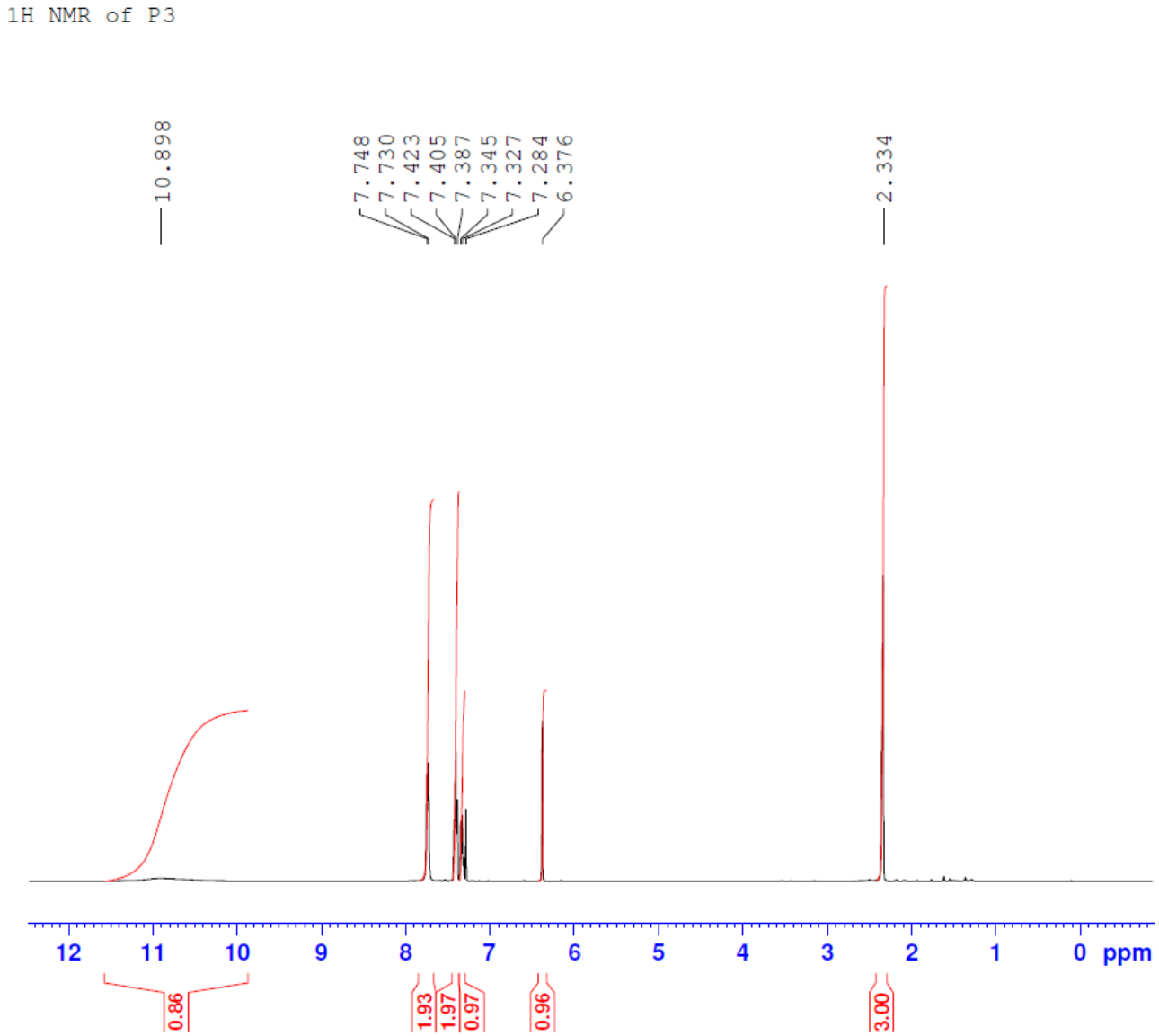

BRUKER

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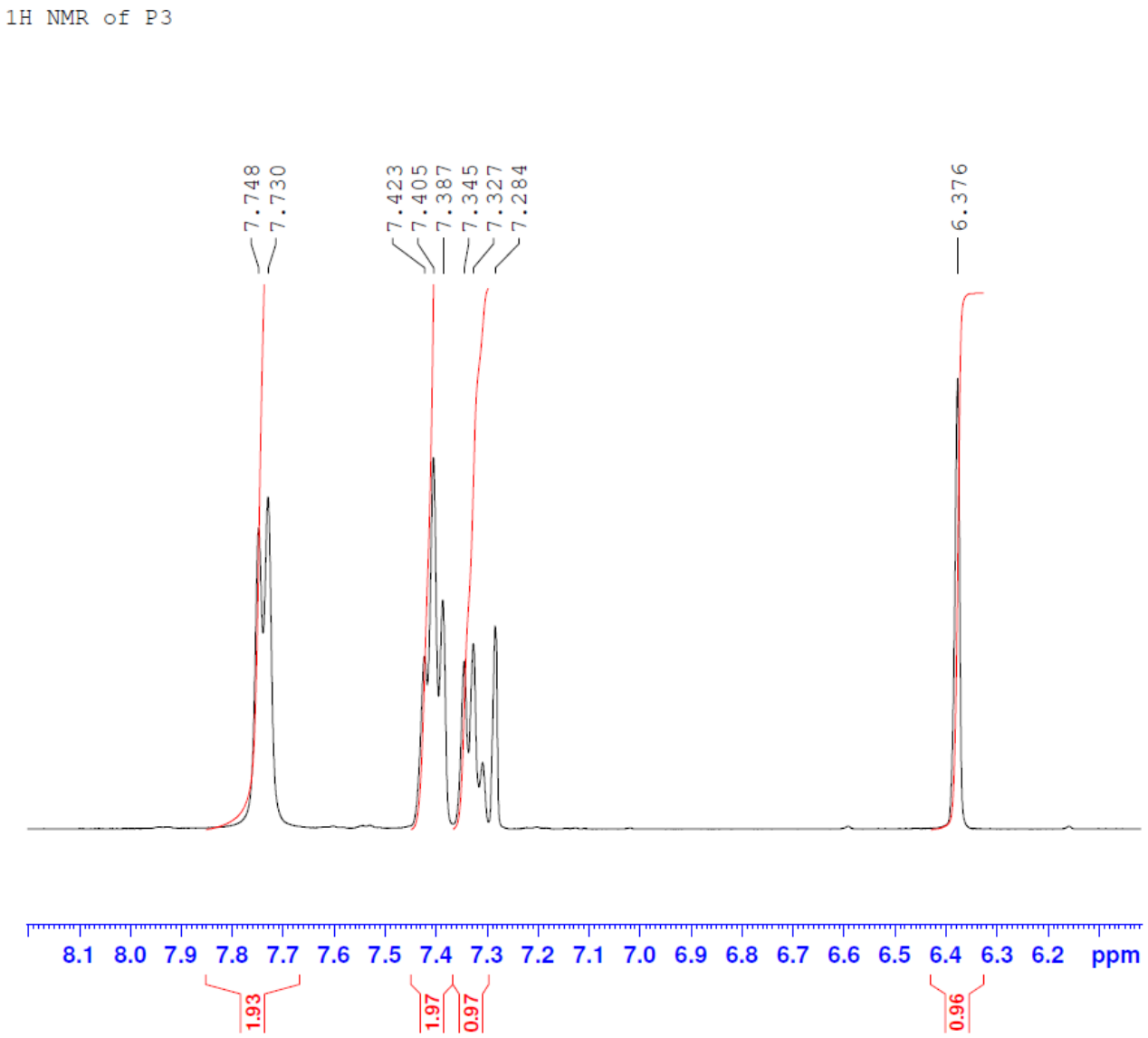


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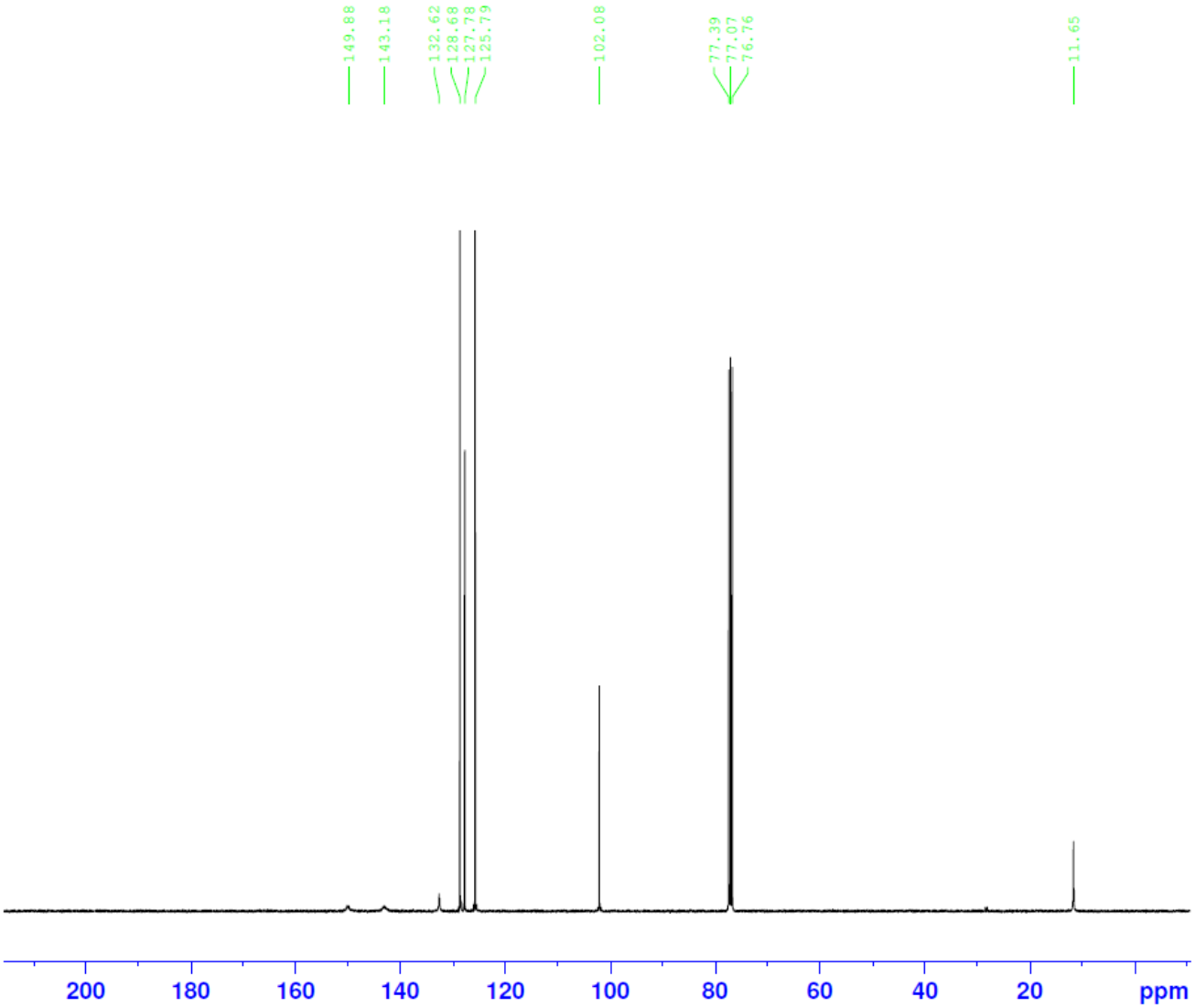
FIDRES                0.250144 Hz
AQ                    3.9976959 sec
RG                         101
DW                      61.000 usec
DE                      13.54 usec
TE                      296.3 K
D1                    1.00000000 sec
TD0                          1
SFO1                400.1424709 MHz
NUC1                        1H
P0                       3.33 usec
P1                      10.00 usec
PLW1                22.64200020 W

F2 - Processing parameters
SI                       65536
SF                  400.1400000 MHz
WDW                         EM
SSB                          0
LB                       0.30 Hz
GB                           0
PC                        1.00
```

5-methyl-3-phenyl-1H-pyrazole (P3): ^{13}C-NMR ($CDCl_3$)

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Current Data Parameters
NAME      SM-P3_1Hand13C
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230318
Time               9.25 h
INSTRUM       Avance Neo
PROBHD   Z140678_0053 (
PULPROG          zgpg30
TD                65536
SOLVENT            CDCl3
NS                 4096
DS                    4
SWH           23809.523 Hz
FIDRES         0.726609 Hz
AQ             1.3762560 sec
RG                   16
DW                21.000 usec
DE                 6.50 usec
TE                296.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         100.6253446 MHz
NUC1                13C
P0                 3.33 usec
P1                10.00 usec
PLW1         77.80000305 W
SFO2         400.1416006 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             90.00 usec
PLW2         22.64200020 W
PLW12         0.27952999 W
PLW13         0.14060000 W

F2 - Processing parameters
SI                 32768
SF           100.6152830 MHz
WDW                   EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```
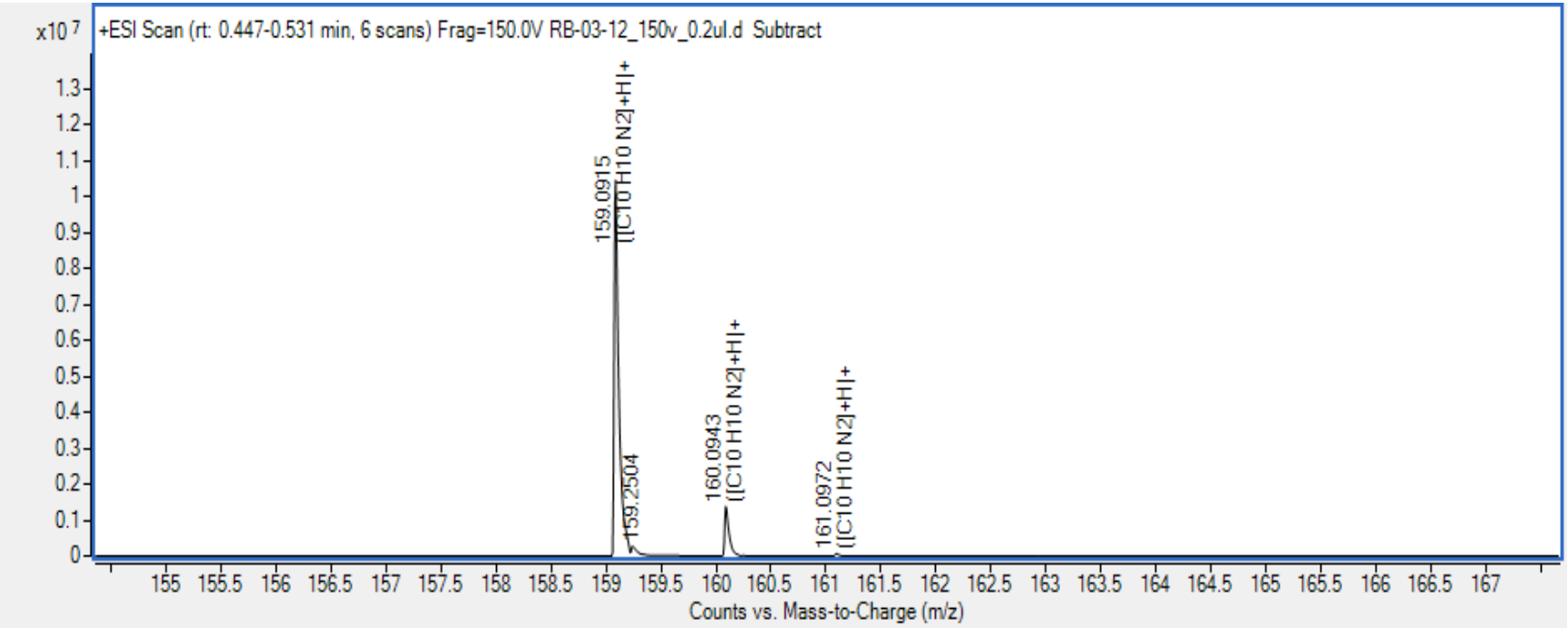
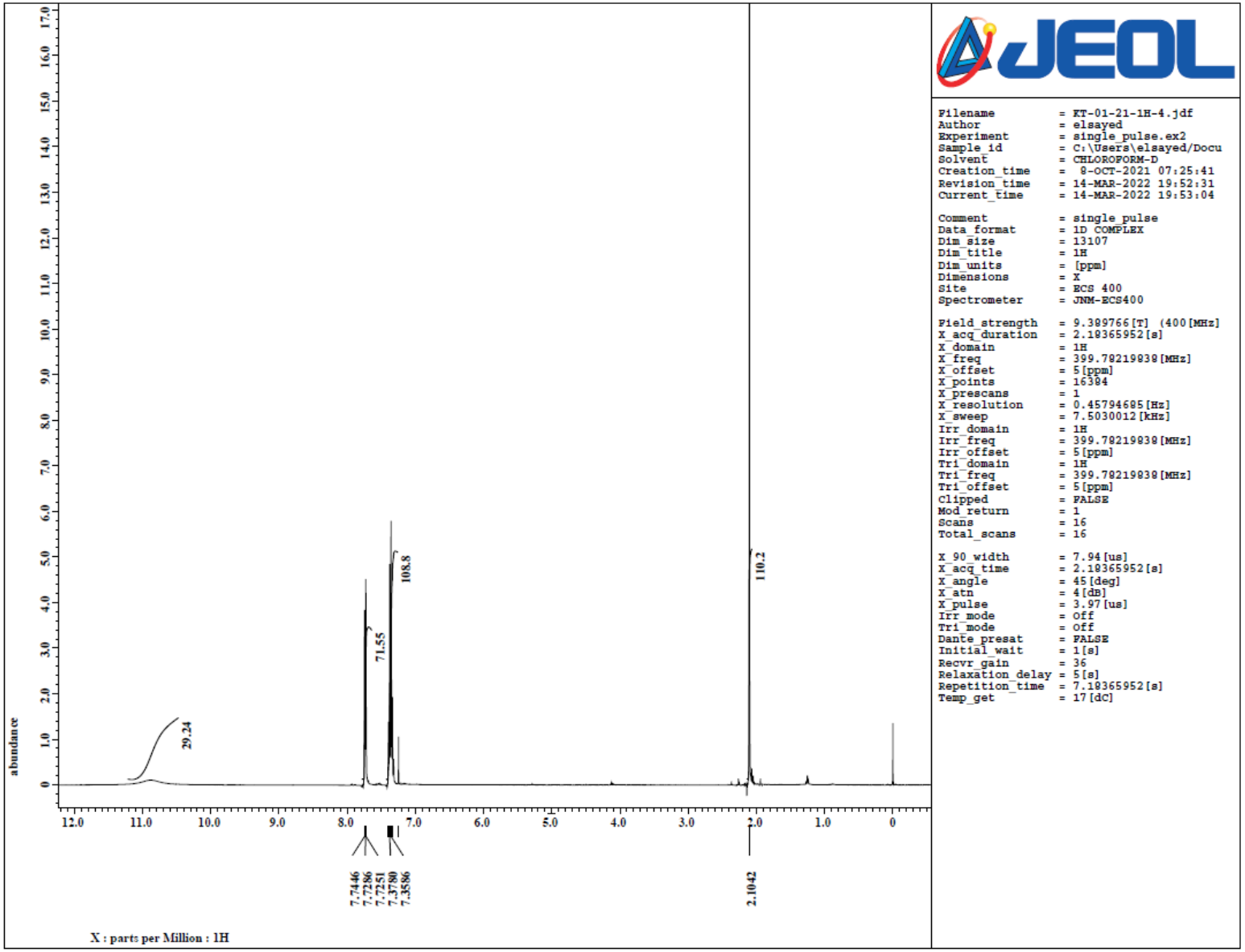

5-methyl-3-phenyl-1H-pyrazole (P3): ESI-MS

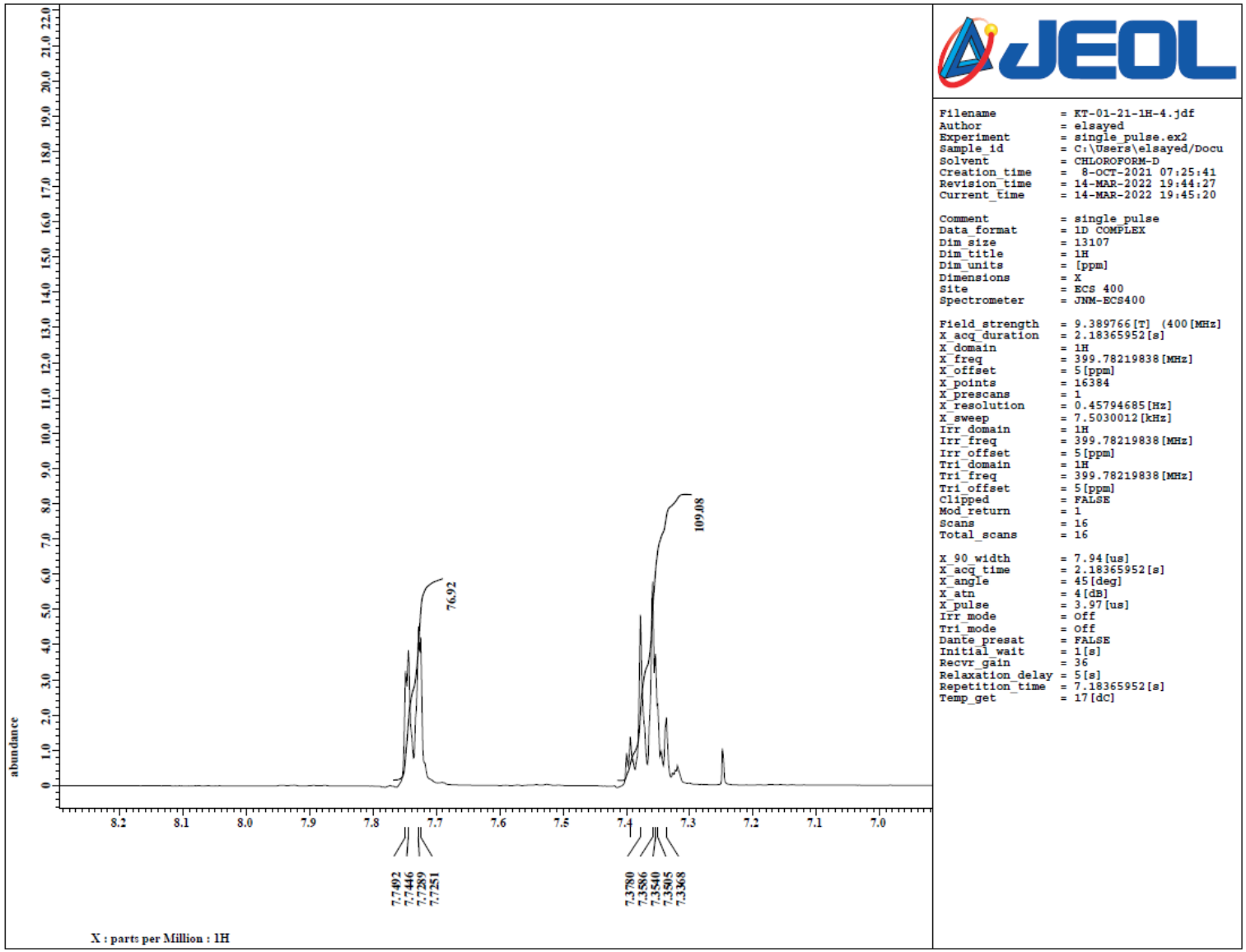


Formula,	Species,	m/z,	Score,	Diff (abs. ppm),	Mass
C10 H10 N2,	(M+H)+,	159.0915,	98.7,	1.37,	158.0842

4-chloro-5-methyl-3-phenyl-1H-pyrazole (P4): ^{1}H-NMR ($CDCl_3$)

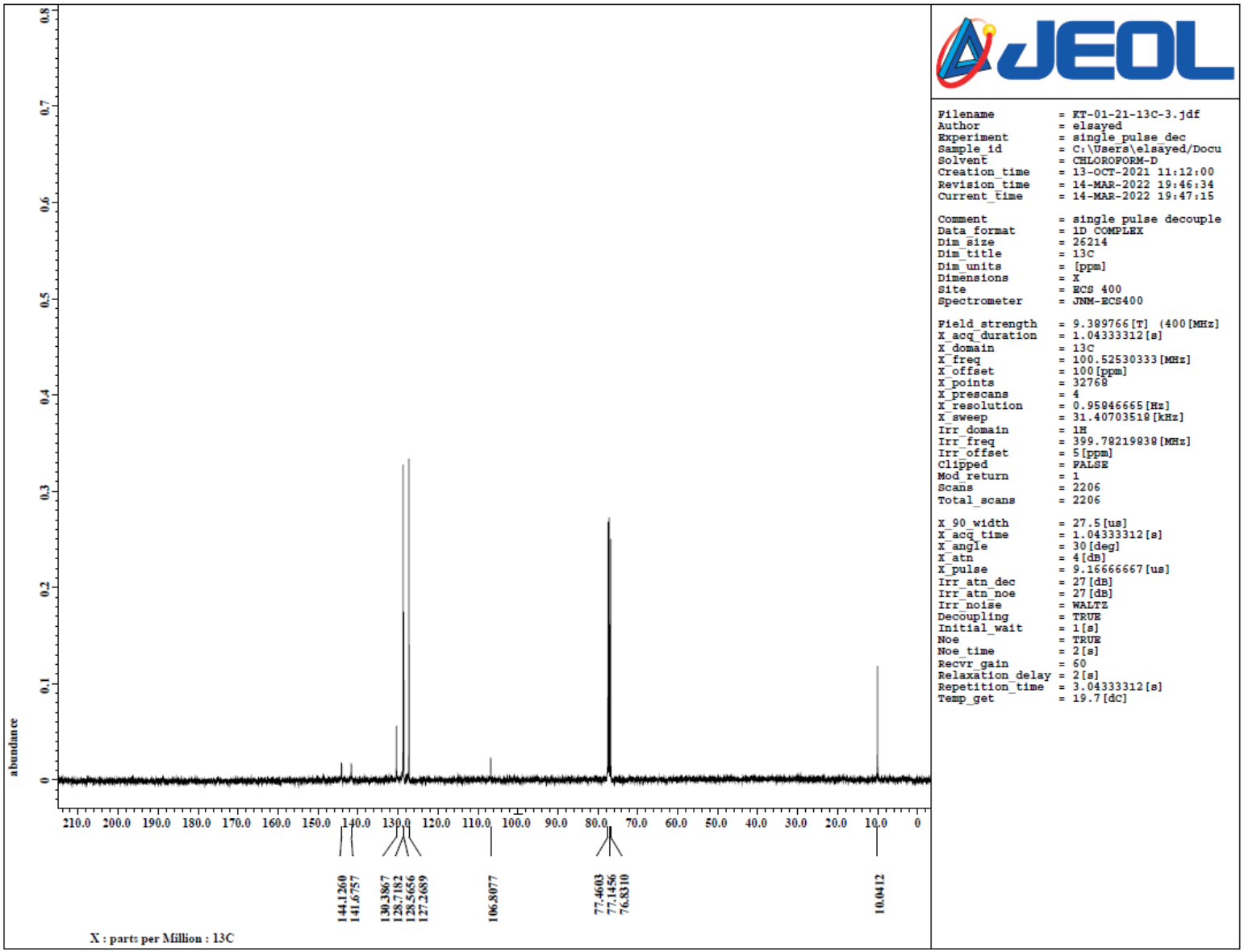


4-chloro-5-methyl-3-phenyl-1H-pyrazole (P4): ^{1}H-NMR ($CDCl_3$) _ Expanded Aromatic Region

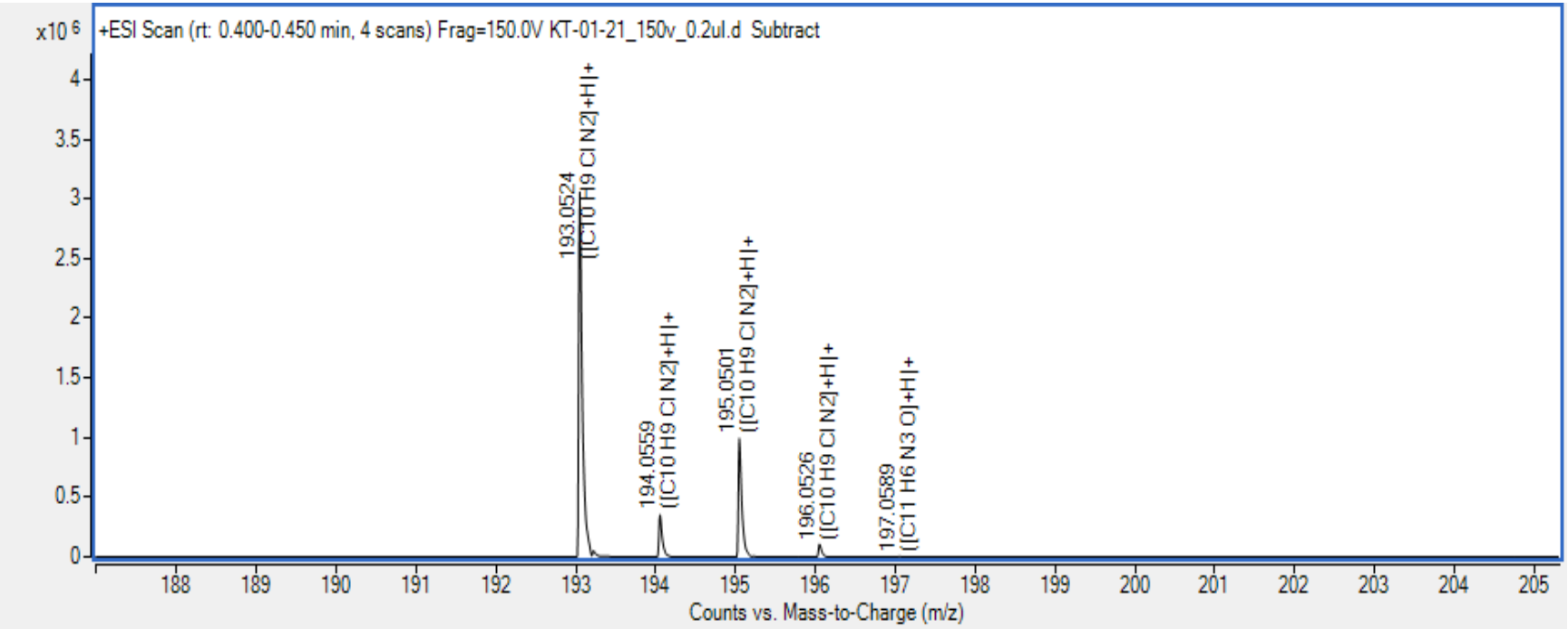



```
Filename          = KT-01-21-1H-4.jdf
Author            = elsayed
Experiment        = single_pulse.ex2
Sample_id         = C:\Users\elsayed/Docu
Solvent           = CHLOROFORM-D
Creation_time     =  8-OCT-2021 07:25:41
Revision_time     = 14-MAR-2022 19:44:27
Current_time      = 14-MAR-2022 19:45:20

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 7.94[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 4[dB]
X_pulse           = 3.97[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 36
Relaxation_delay  = 5[s]
Repetition_time   = 7.18365952[s]
Temp_get          = 17[dC]
```

4-chloro-5-methyl-3-phenyl-1H-pyrazole (P4): ^{13}C-NMR ($CDCl_3$)

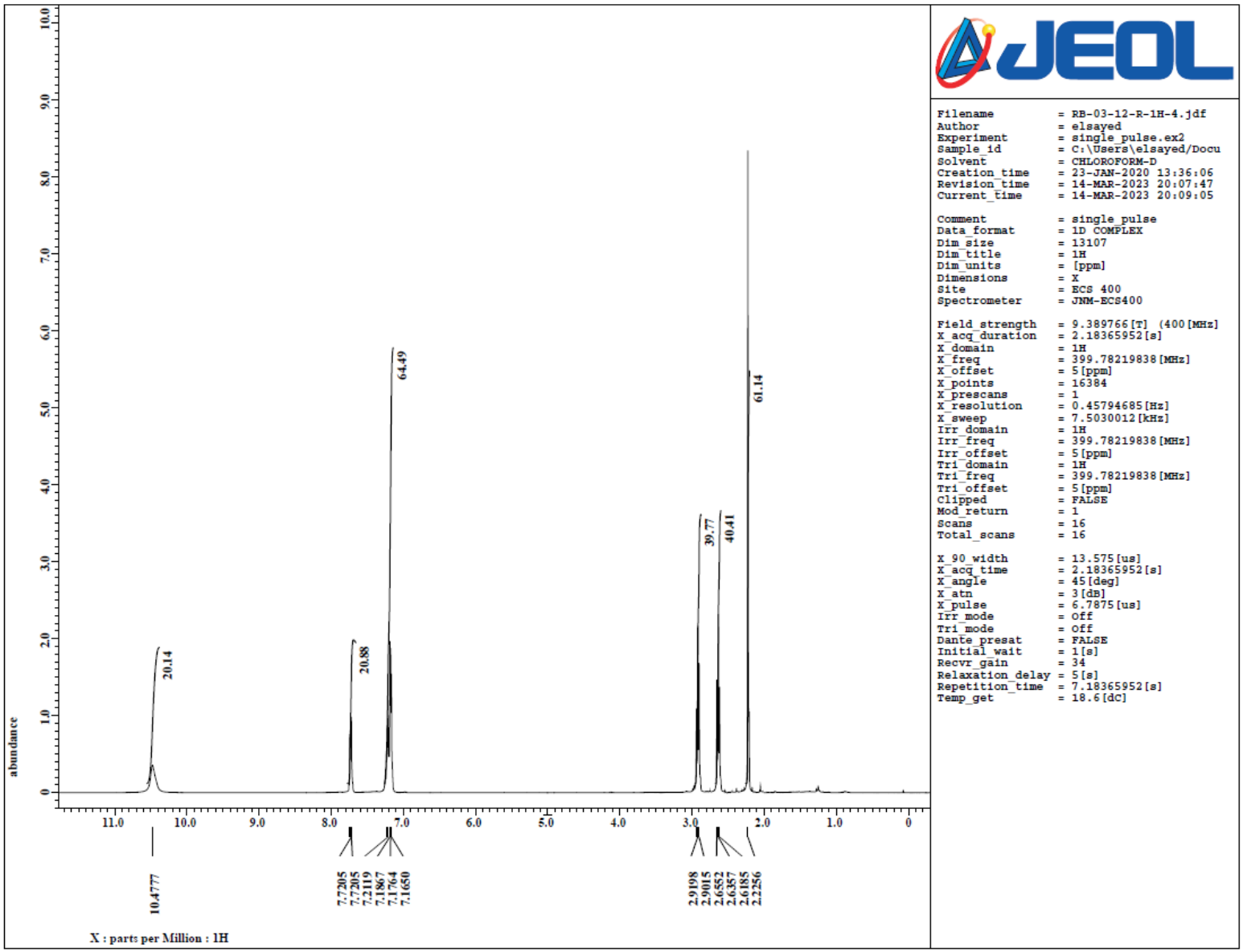


4-chloro-5-methyl-3-phenyl-1H-pyrazole (P4): ESI-MS

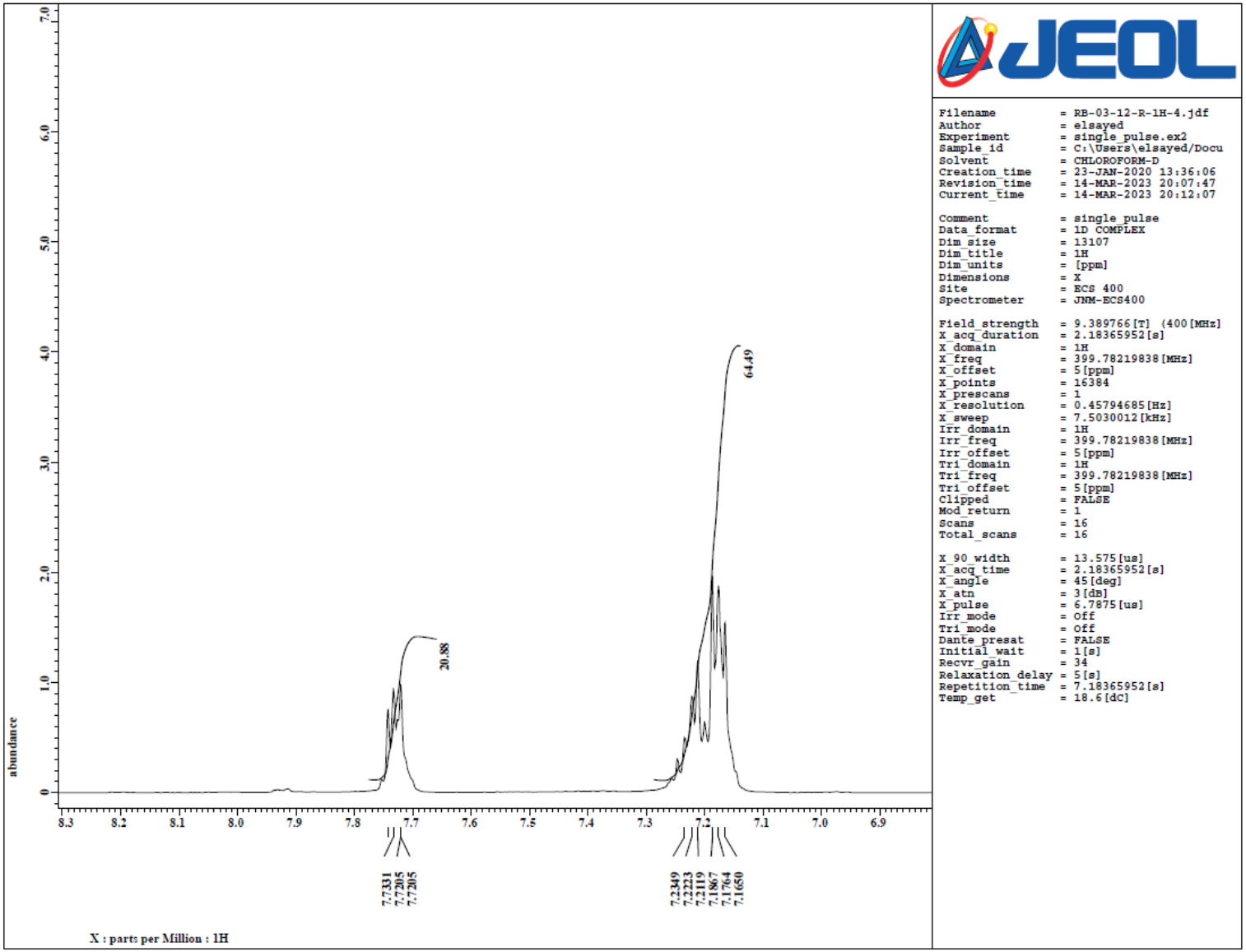


Formula,	Species,	m/z,	Score,	Diff (abs. ppm),	Mass
C10 H9 Cl N2,	(M+H)+,	193.0524,	99.25,	0.82,	192.0453

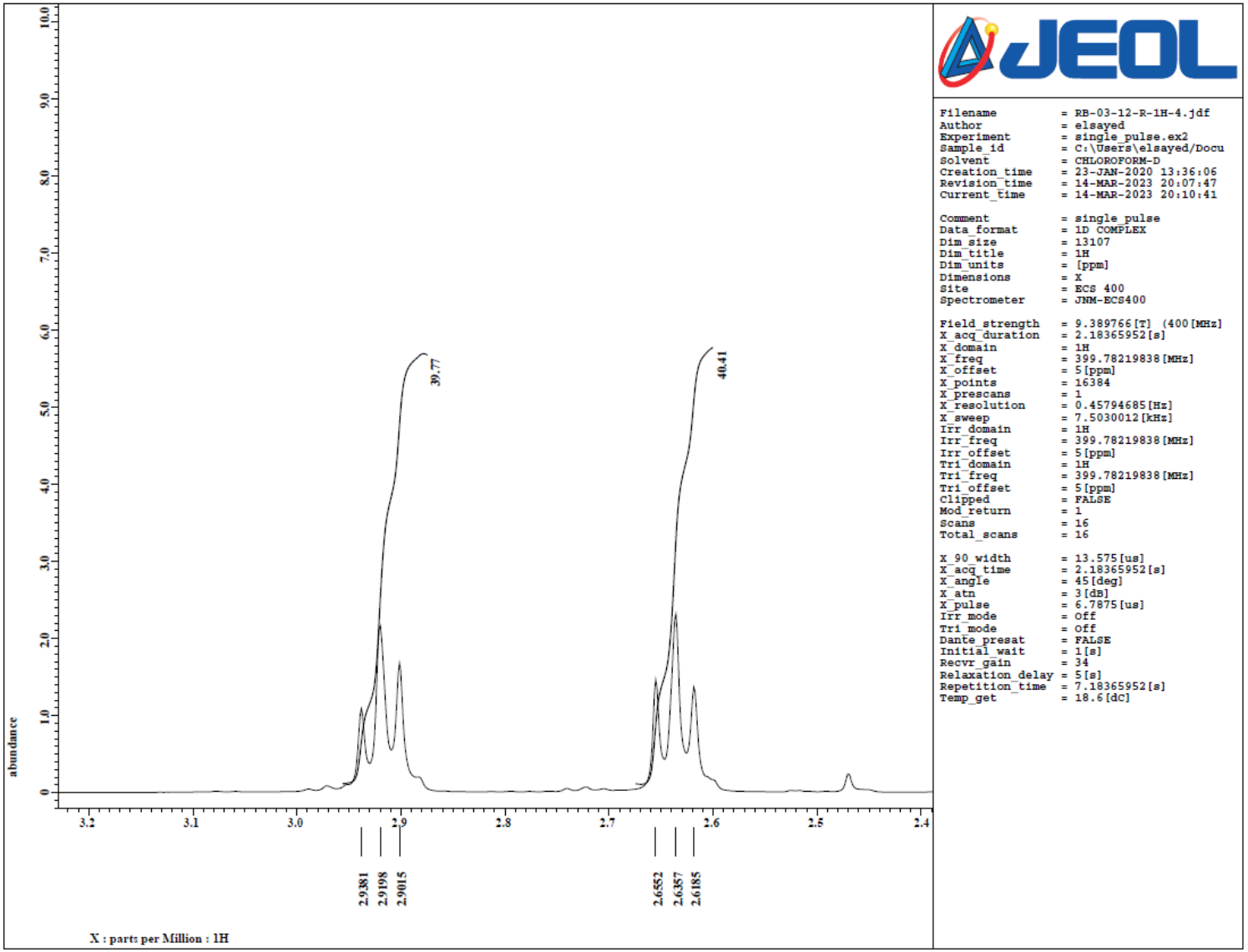
3-methyl-4,5-dihydro-2H-benzo[g]indazole (P5): ^{1}H-NMR ($CDCl_3$)



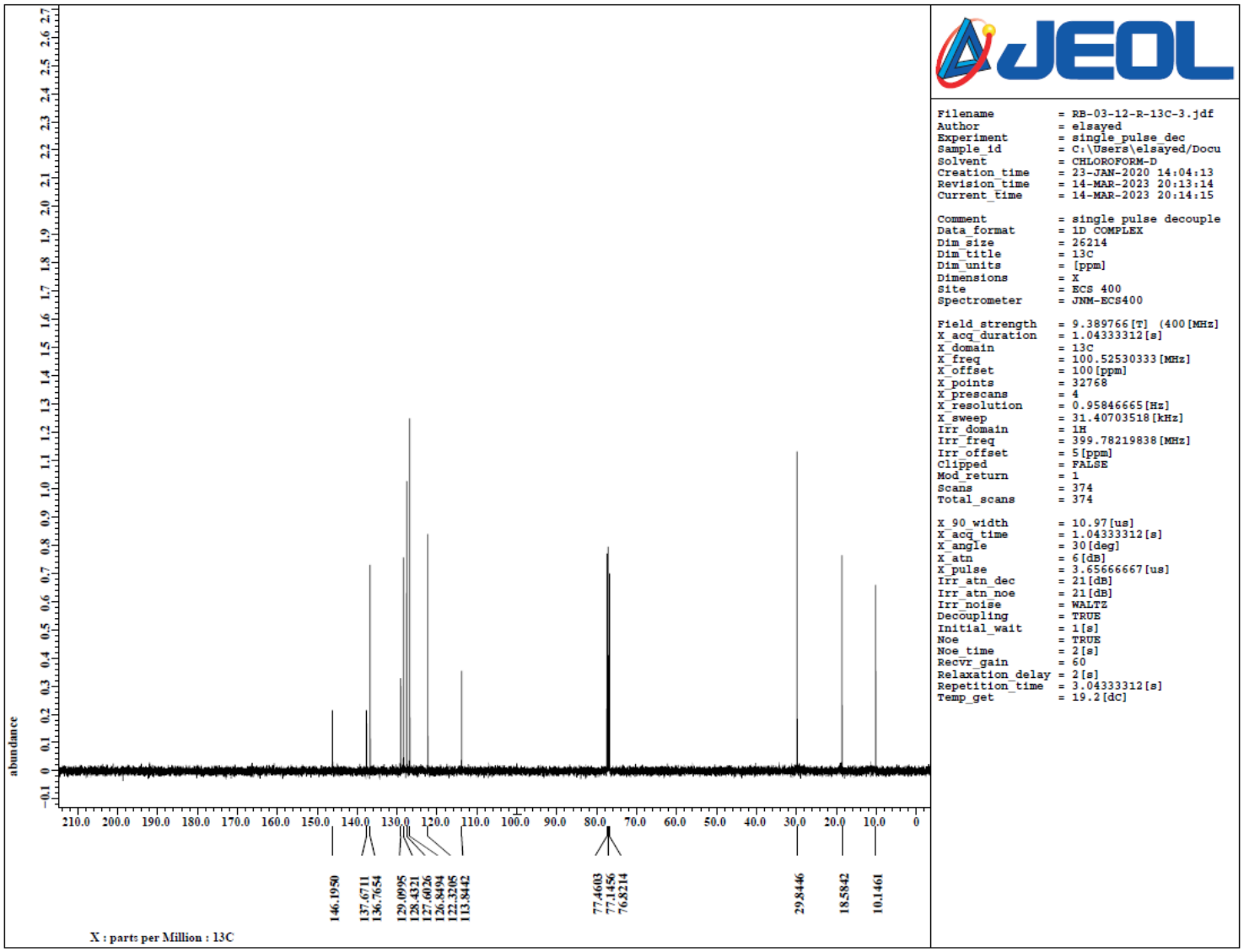
3-methyl-4,5-dihydro-2H-benzo[g]indazole (P5): ^{1}H-NMR ($CDCl_3$) _ Expanded Aromatic Region



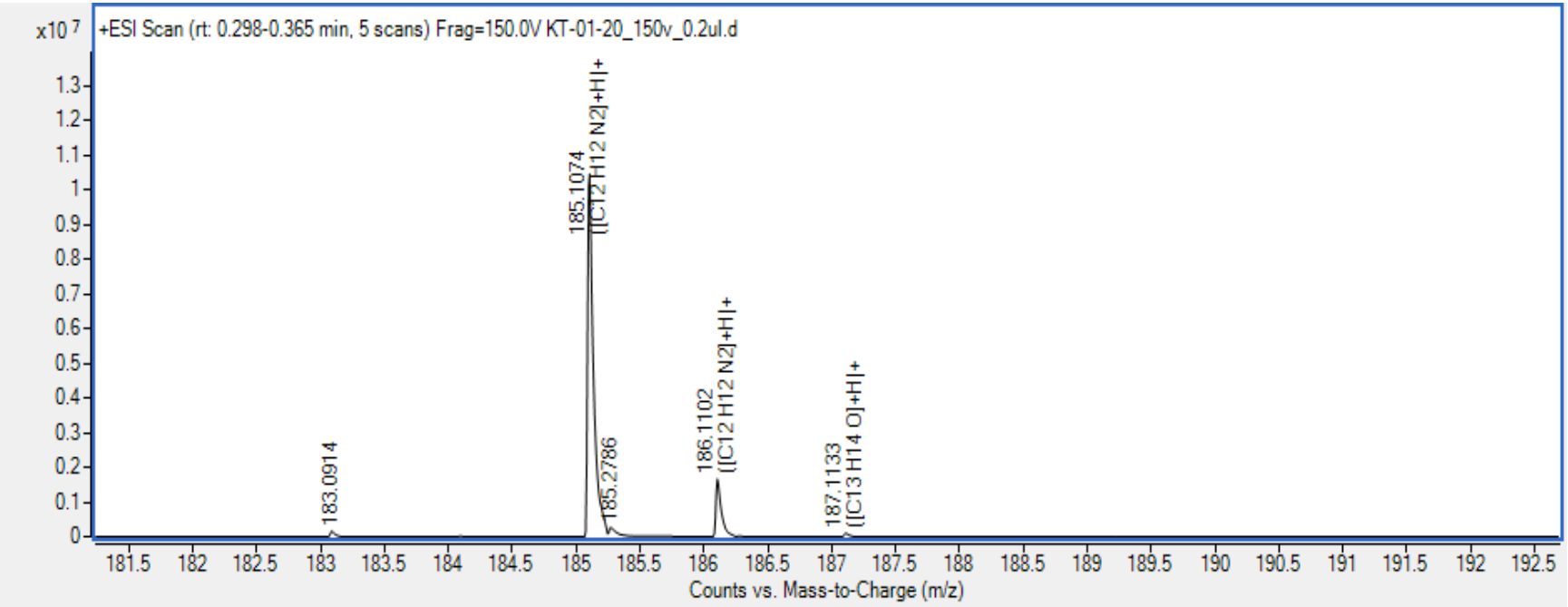
3-methyl-4,5-dihydro-2H-benzo[g]indazole (P5): ^{1}H-NMR ($CDCl_3$) _ Expanded Aliphatic Region



3-methyl-4,5-dihydro-2H-benzo[g]indazole (P5): ^{13}C-NMR ($CDCl_3$)

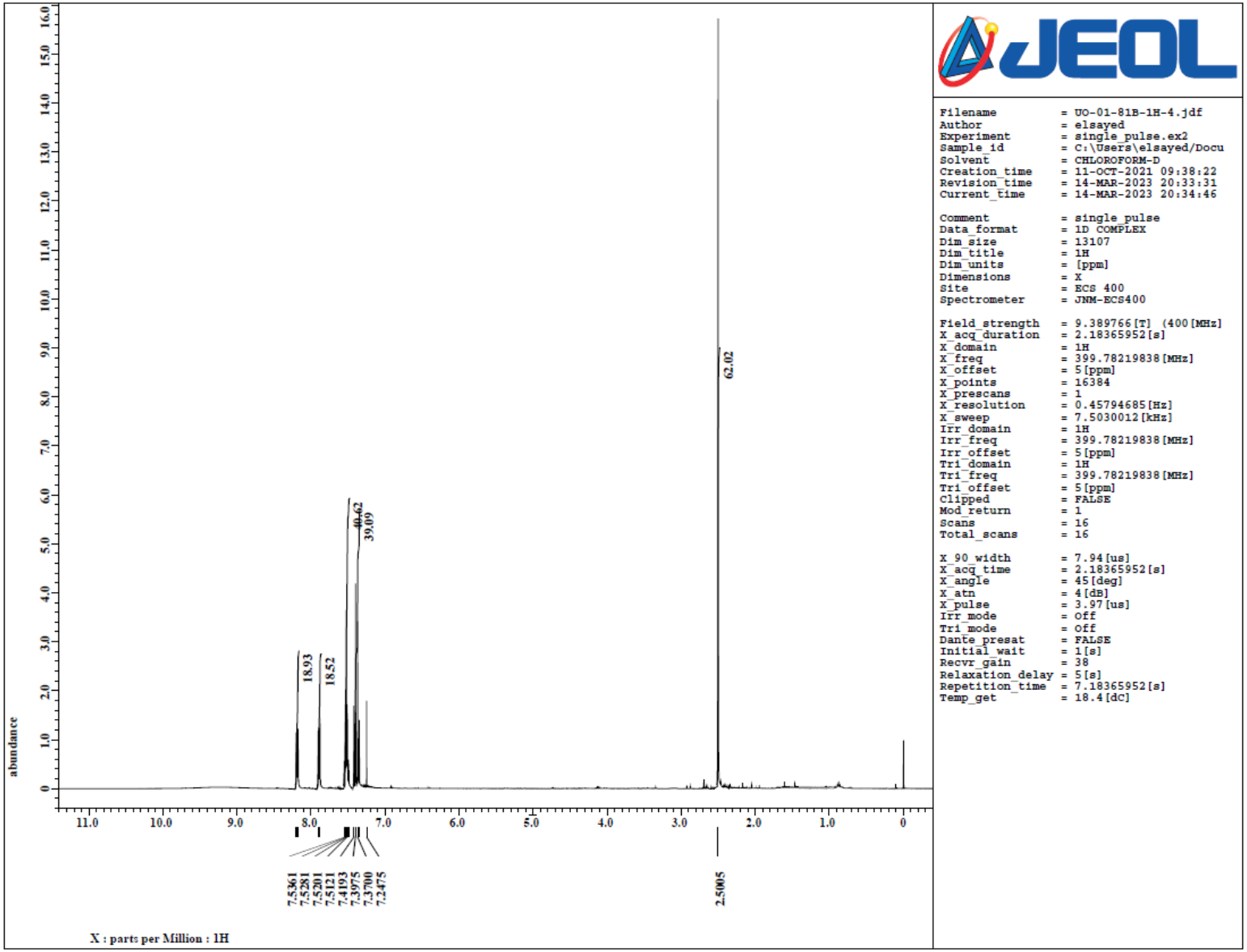


3-methyl-4,5-dihydro-2H-benzo[g]indazole (P5): ESI-MS



Formula,	Species,	m/z,	Score,	Diff (abs. ppm),	Mass
C12 H12 N2,	(M+H)+,	185.1074,	86.32,	0.04,	184.1001

3-methyl-2H-benzo[g]indazole (P6): ^{1}H-NMR ($CDCl_3$)

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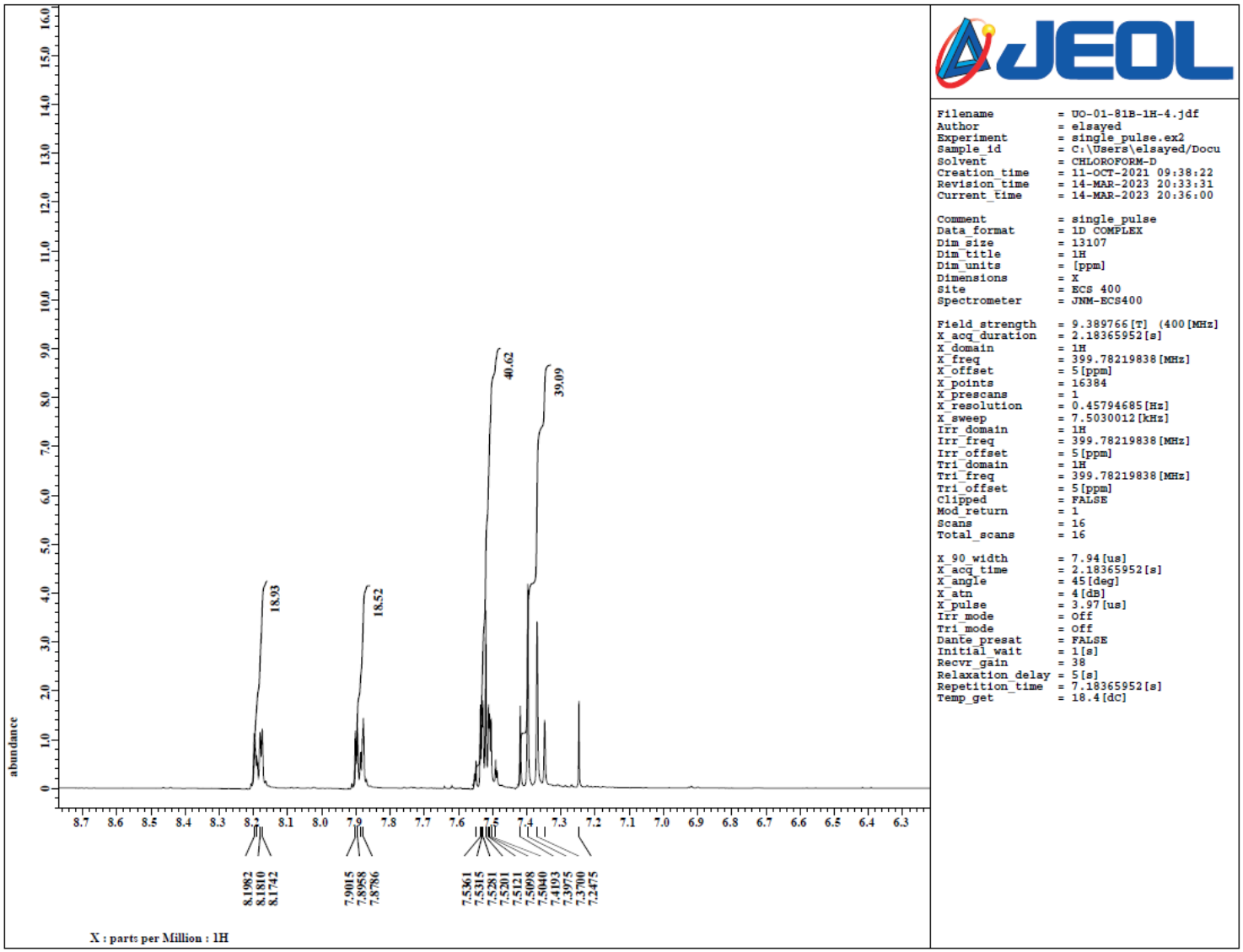
Filename          = UO-01-81B-1H-4.jdf
Author            = elsayed
Experiment        = single_pulse.ex2
Sample_id         = C:\Users\elsayed/Docu
Solvent           = CHLOROFORM-D
Creation_time     = 11-OCT-2021 09:38:22
Revision_time     = 14-MAR-2023 20:33:31
Current_time      = 14-MAR-2023 20:34:46

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

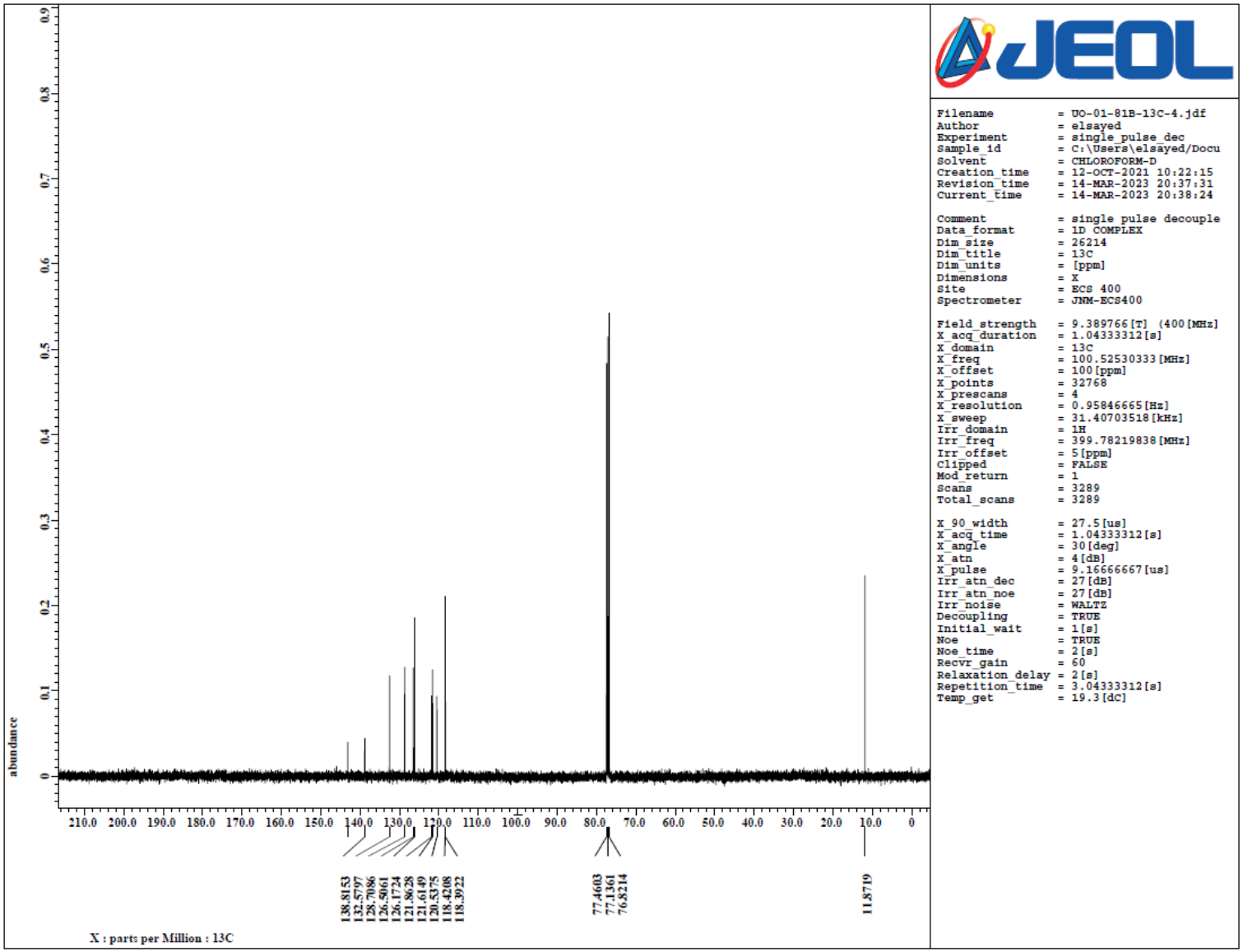
Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 7.94[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 4[dB]
X_pulse           = 3.97[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 38
Relaxation_delay  = 5[s]
Repetition_time   = 7.18365952[s]
Temp_get          = 18.4[dC]
```

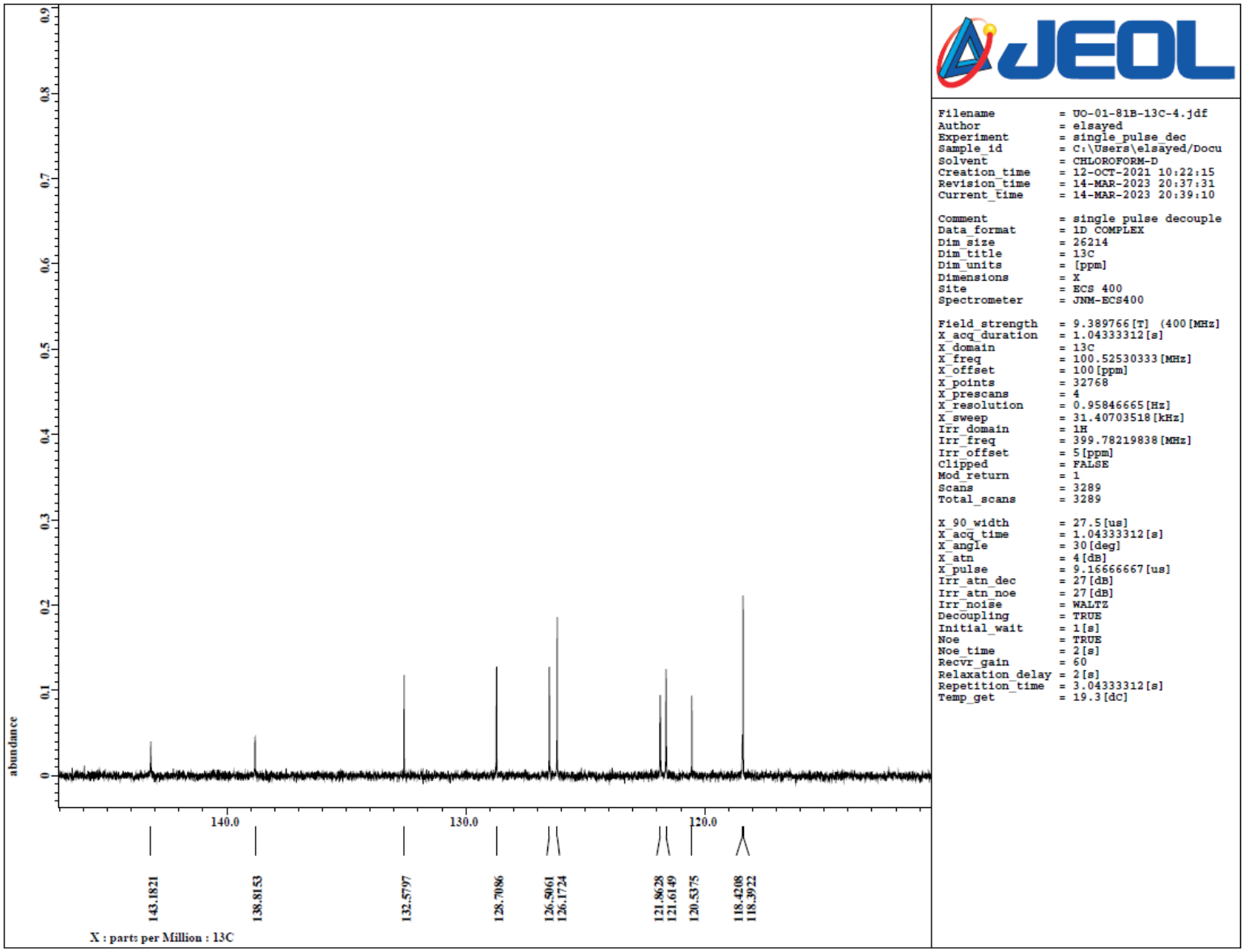
3-methyl-2H-benzo[g]indazole (P6): ^{1}H-NMR ($CDCl_3$) _ Expanded Aromatic Region



3-methyl-2H-benzo[g]indazole (P6): ^{13}C-NMR ($CDCl_3$)



3-methyl-2H-benzo[g]indazole (P6): ^{13}C-NMR ($CDCl_3$) _ Expanded Aromatic Region



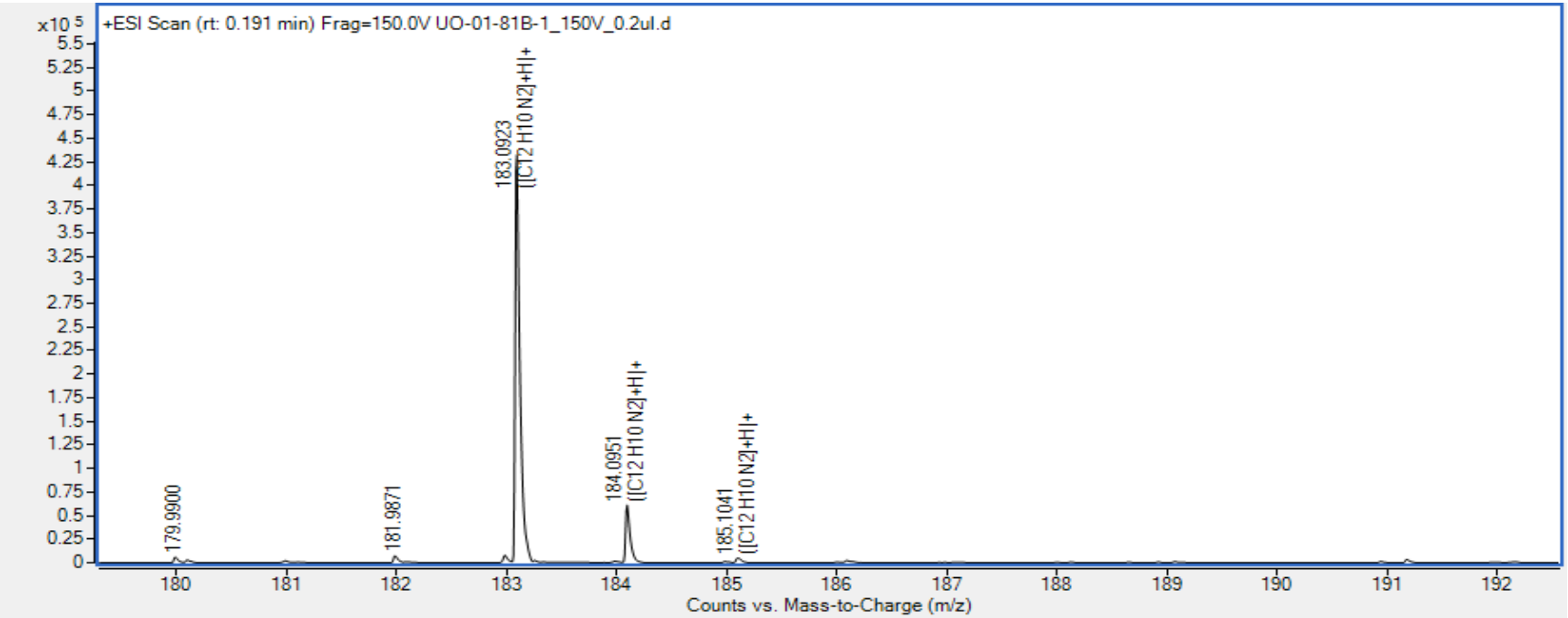

```
Filename          = UO-01-81B-13C-4.jdf
Author            = elsayed
Experiment        = single_pulse_dec
Sample_id         = C:\Users\elsayed/Docu
Solvent           = CHLOROFORM-D
Creation_time     = 12-OCT-2021 10:22:15
Revision_time     = 14-MAR-2023 20:37:31
Current_time      = 14-MAR-2023 20:39:10

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 3289
Total_scans       = 3289

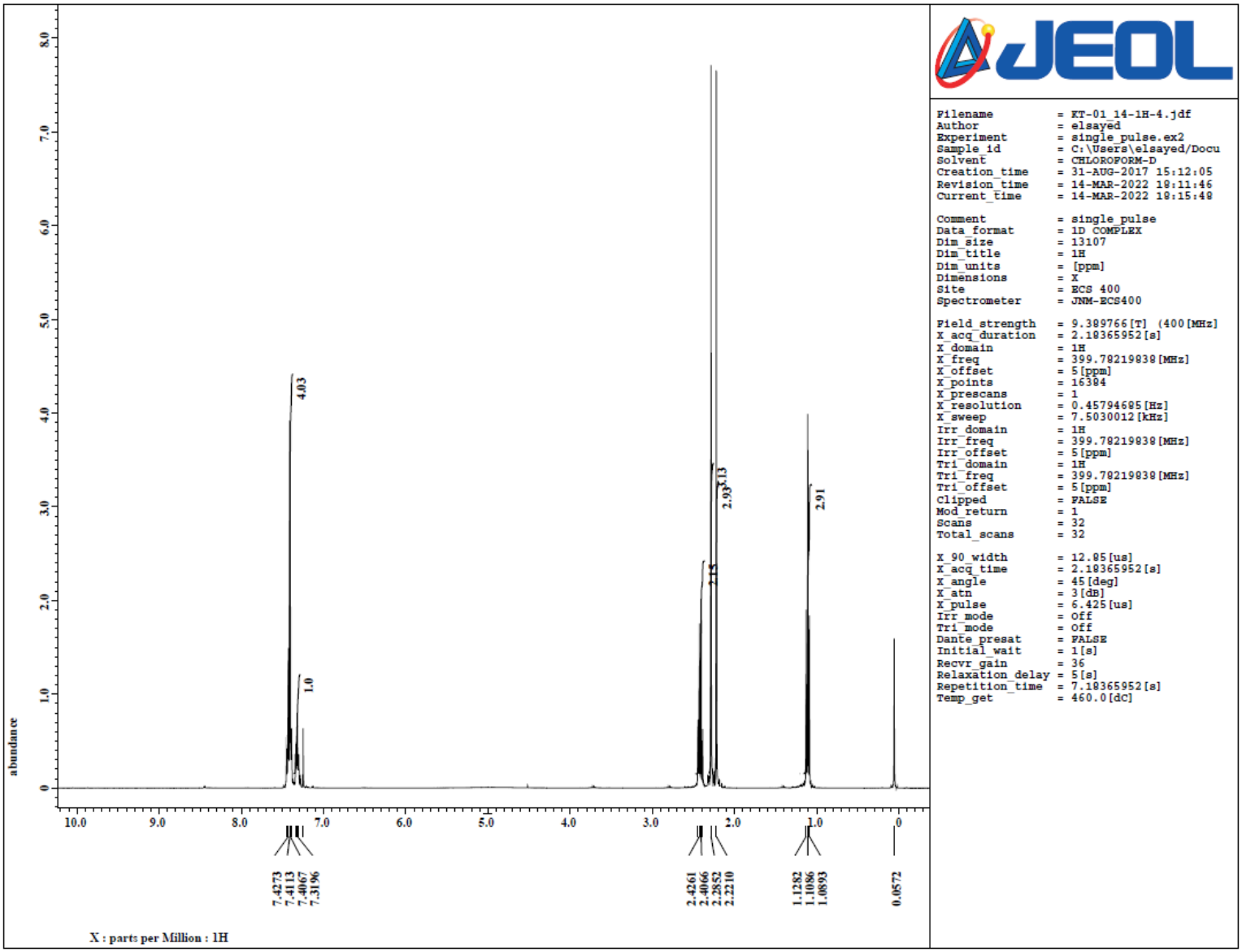
X_90_width        = 27.5[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 4[dB]
X_pulse           = 9.16666667[us]
Irr_atn_dec       = 27[dB]
Irr_atn_noe       = 27[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 3.04333312[s]
Temp_get          = 19.3[dC]
```

3-methyl-2H-benzo[g]indazole (P6): ESI-MS

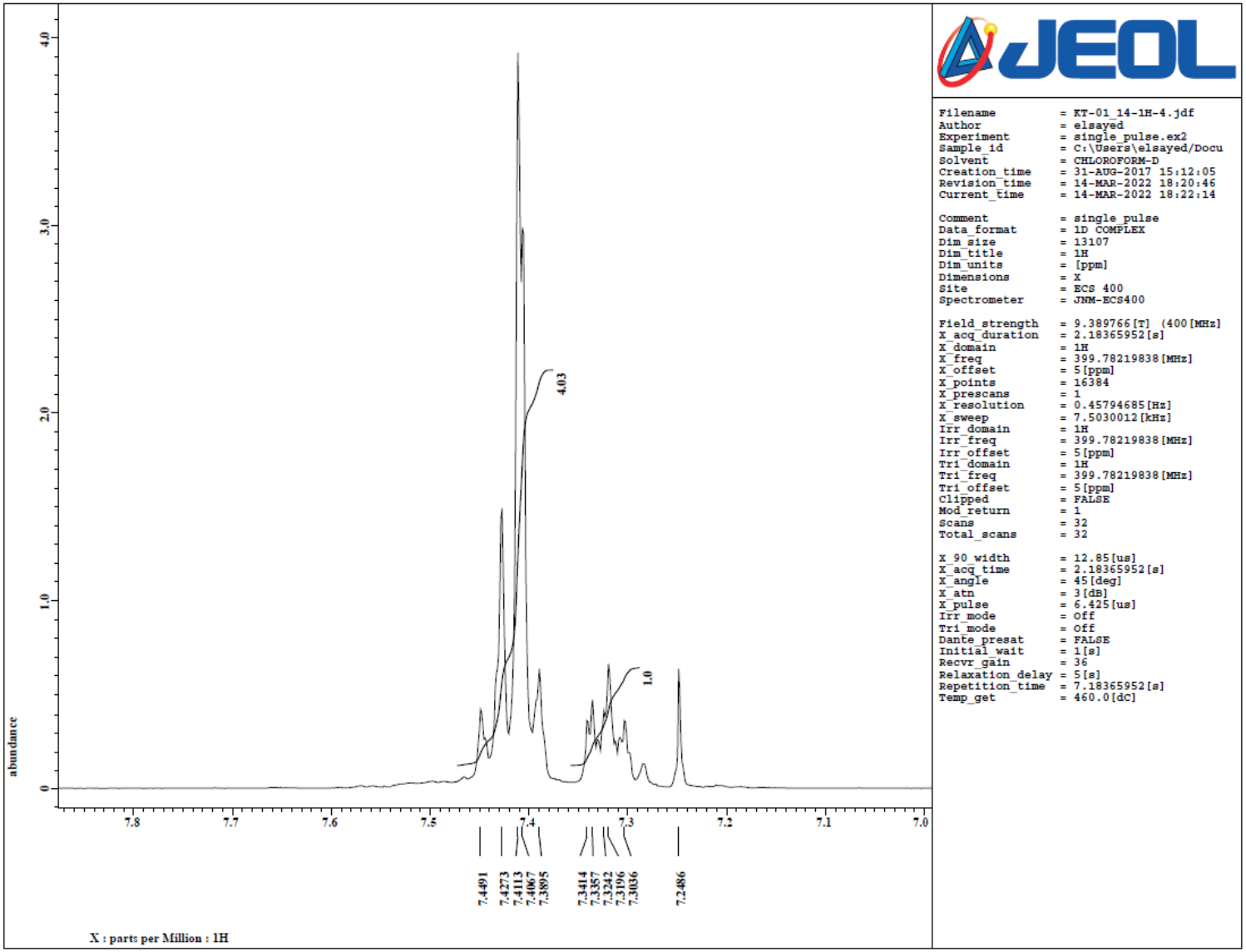


Formula,	Species,	m/z,	Diff (abs. ppm),	Score,	Mass
C12 H10 N2,	(M+H)+,	183.0923,	3.55,	97.29,	182.085

4-ethyl-3,5-dimethyl-1-phenyl-1H-pyrazole (P7): ^{1}H-NMR ($CDCl_3$)



4-ethyl-3,5-dimethyl-1-phenyl-1H-pyrazole (P7): ^{1}H-NMR ($CDCl_3$) _ Expanded Aromatic Region



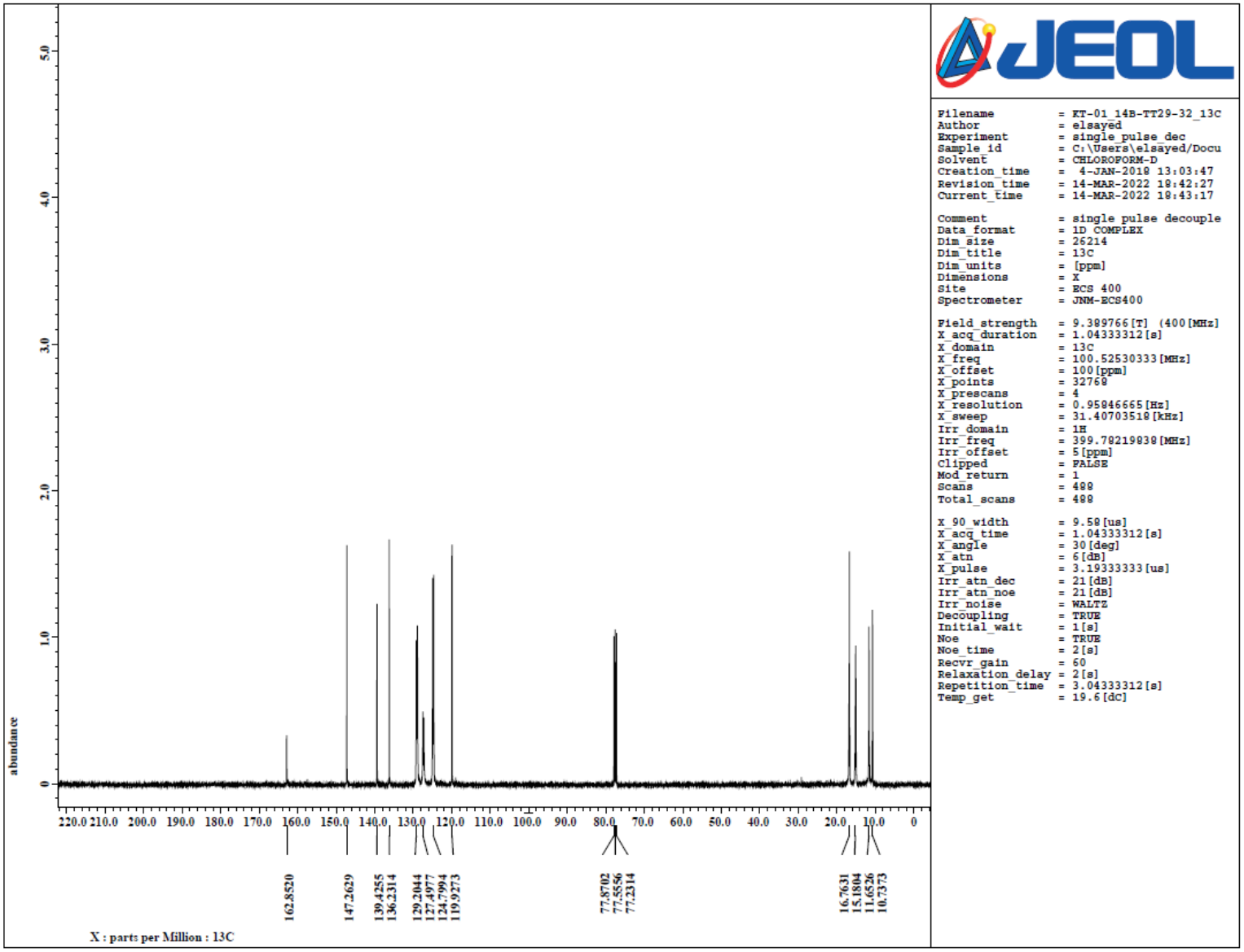

```
Filename          = KT-01_14-1H-4.jdf
Author            = elsayed
Experiment        = single_pulse.ex2
Sample_id         = C:\Users\elsayed/Docu
Solvent           = CHLOROFORM-D
Creation_time     = 31-AUG-2017 15:12:05
Revision_time     = 14-MAR-2022 18:20:46
Current_time      = 14-MAR-2022 18:22:14

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.389766[T]  (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 32
Total_scans       = 32

X_90_width        = 12.85[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.425[us]
Irr_mode          = off
Tri_mode          = off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 36
Relaxation_delay  = 5[s]
Repetition_time   = 7.18365952[s]
Temp_get          = 460.0[dC]
```

4-ethyl-3,5-dimethyl-1-phenyl-1H-pyrazole (P7): ^{13}C-NMR ($CDCl_3$)



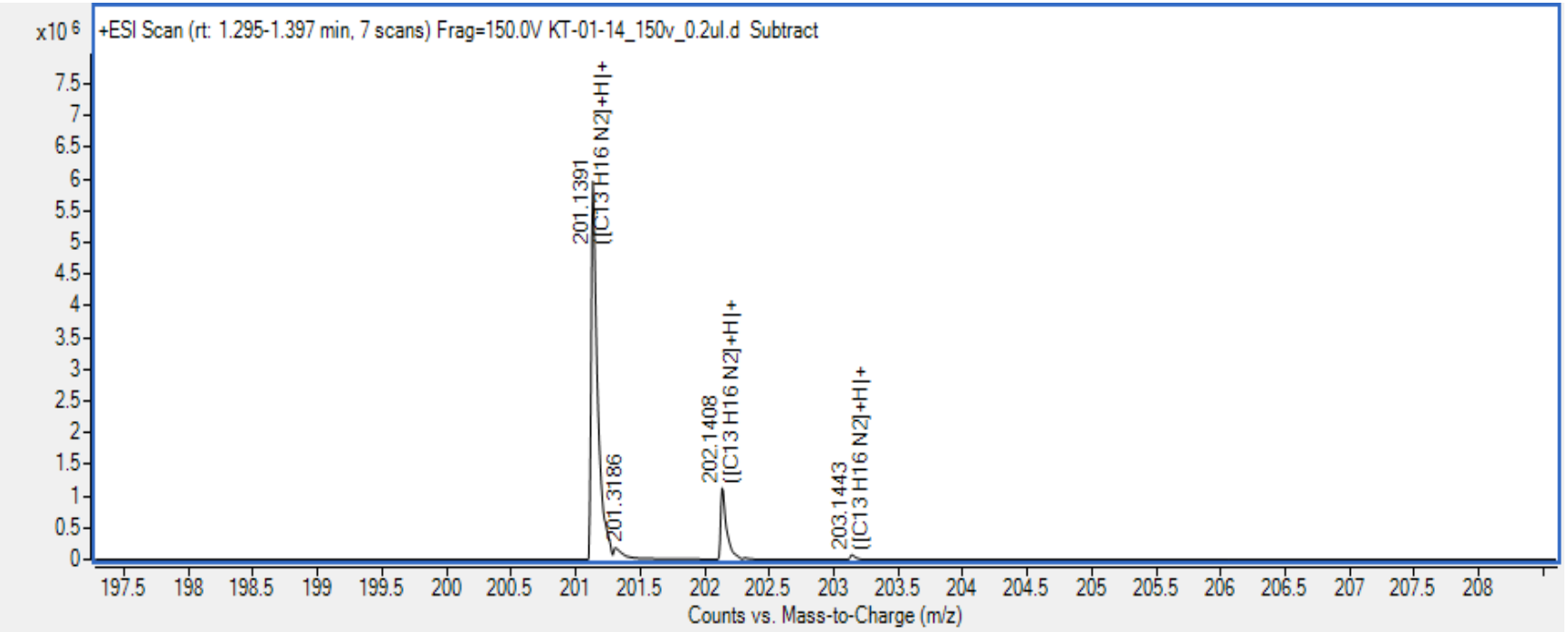

```
Filename          = KT-01_14B-TT29-32_13C
Author            = elsayed
Experiment        = single_pulse_dec
Sample_id         = C:\Users\elsayed/Docu
Solvent           = CHLOROFORM-D
Creation_time     =  4-JAN-2018 13:03:47
Revision_time     = 14-MAR-2022 10:42:27
Current_time      = 14-MAR-2022 10:43:17

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.79219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 488
Total_scans       = 488

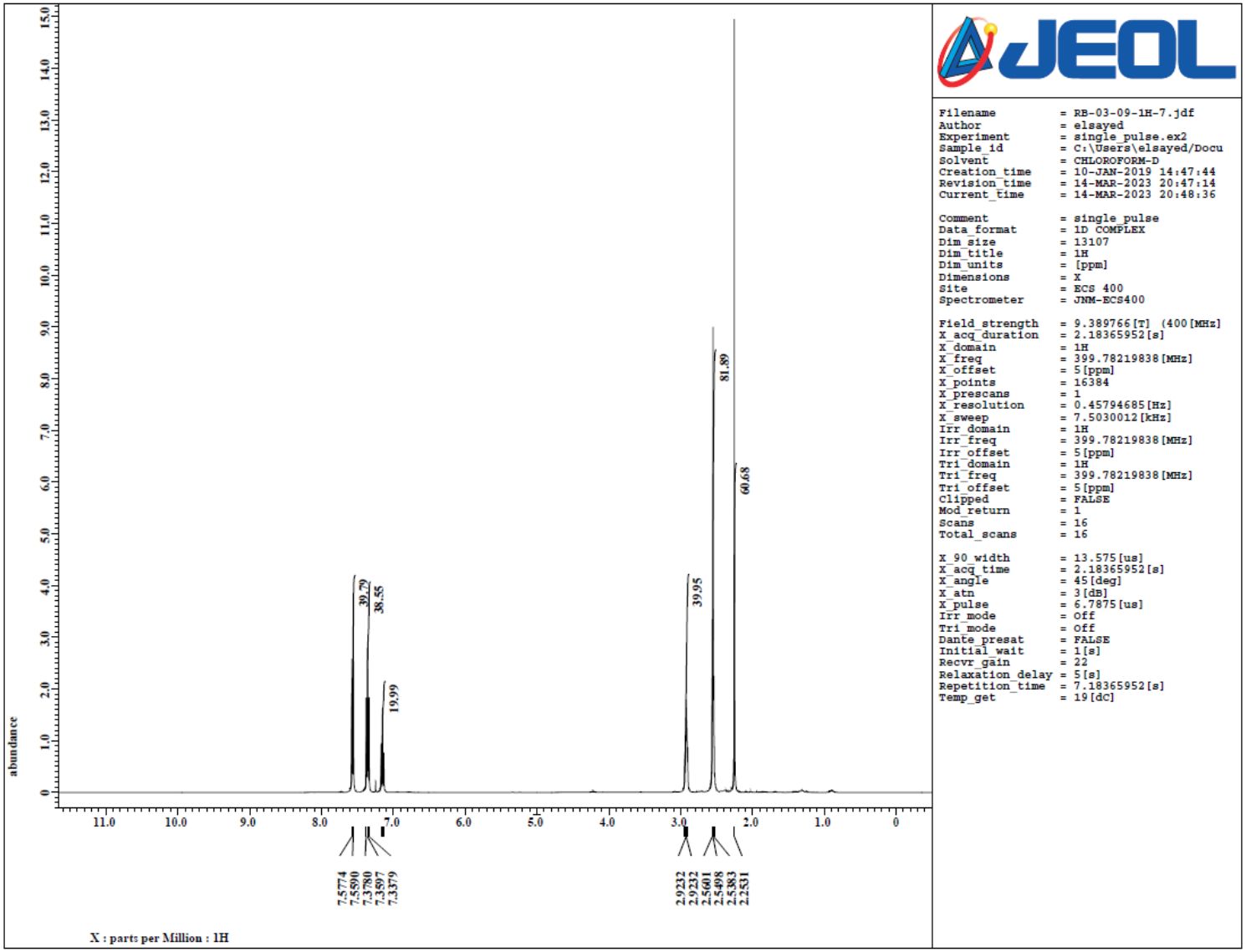
X_90_width        = 9.50[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6[dB]
X_pulse           = 3.19333333[us]
Irr_atn_dec       = 21[dB]
Irr_atn_noe       = 21[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 3.04333312[s]
Temp_get          = 19.6[dC]
```

4-ethyl-3,5-dimethyl-1-phenyl-1H-pyrazole (P7): ESI-MS

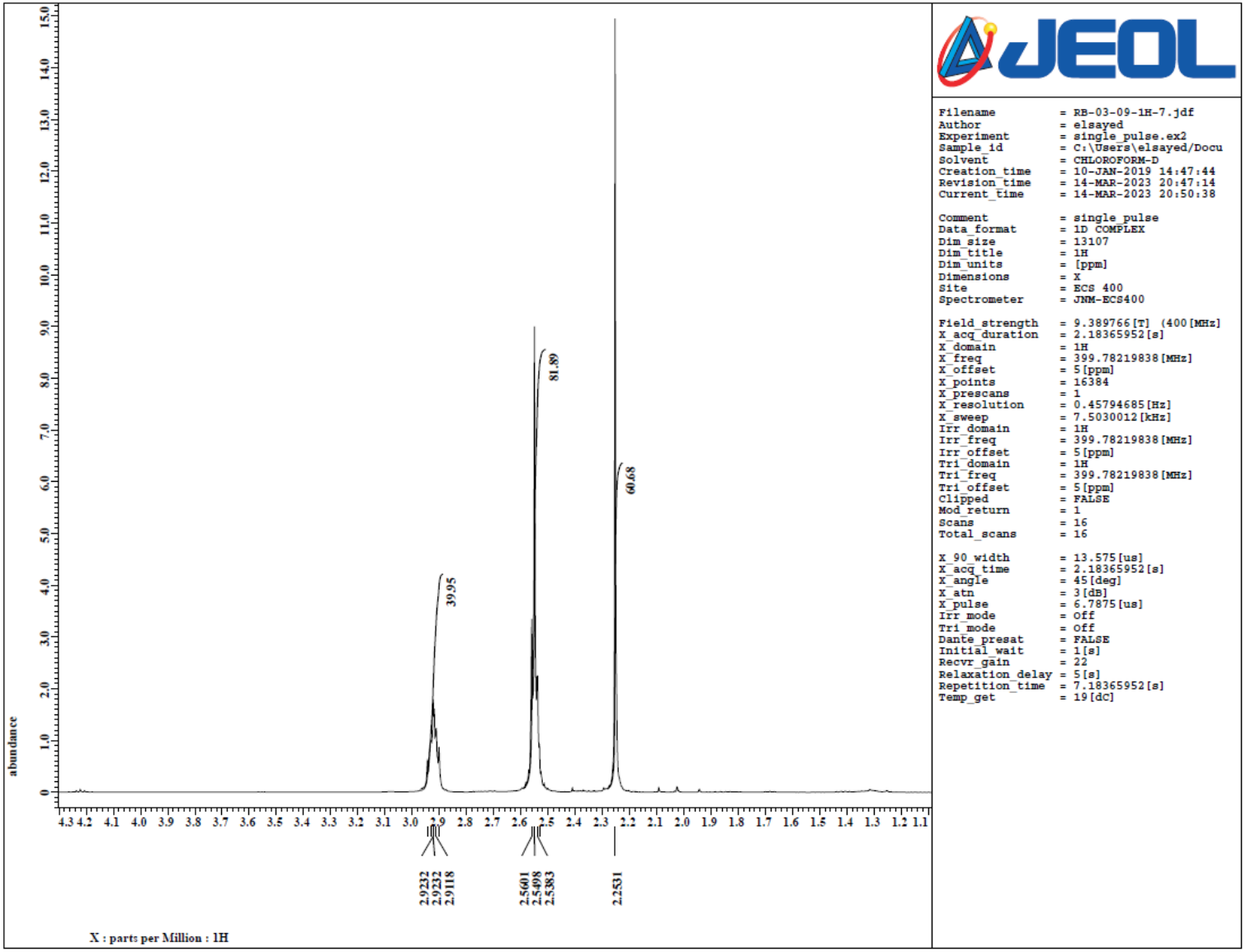


Formula,	Species,	m/z,	Score,	Diff (abs. ppm),	Mass
C13 H16 N2,	(M+H)+,	201.1391,	92.34,	1.02,	200.1316

3-methyl-1-phenyl-1,4,5,6-tetrahydrocyclopenta[c]pyrazole (P8): ^{1}H-NMR ($CDCl_3$)



3-methyl-1-phenyl-1,4,5,6-tetrahydrocyclopenta[c]pyrazole (P8): ^{1}H-NMR ($CDCl_3$) _ Expanded Aliphatic Region



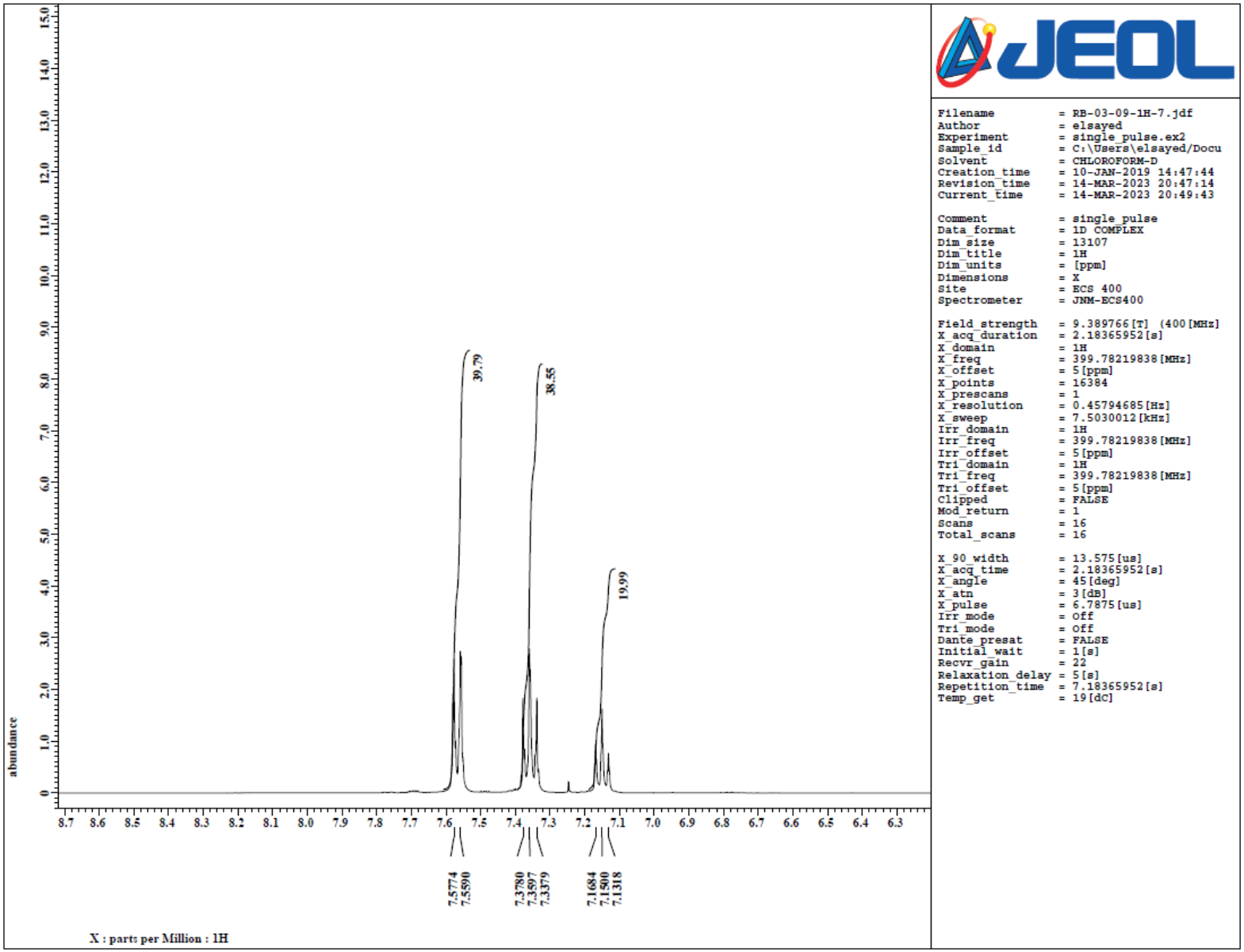

```
Filename          = RB-03-09-1H-7.jdf
Author            = elsayed
Experiment        = single_pulse.ex2
Sample_id         = C:\Users\elsayed/Docu
Solvent           = CHLOROFORM-D
Creation_time     = 10-JAN-2019 14:47:44
Revision_time     = 14-MAR-2023 20:47:14
Current_time      = 14-MAR-2023 20:50:38

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 13.575[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.7875[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 22
Relaxation_delay  = 5[s]
Repetition_time   = 7.18365952[s]
Temp_get          = 19[dC]
```

3-methyl-1-phenyl-1,4,5,6-tetrahydrocyclopenta[c]pyrazole (P8): ^{1}H-NMR ($CDCl_3$) _ Expanded Aromatic Region



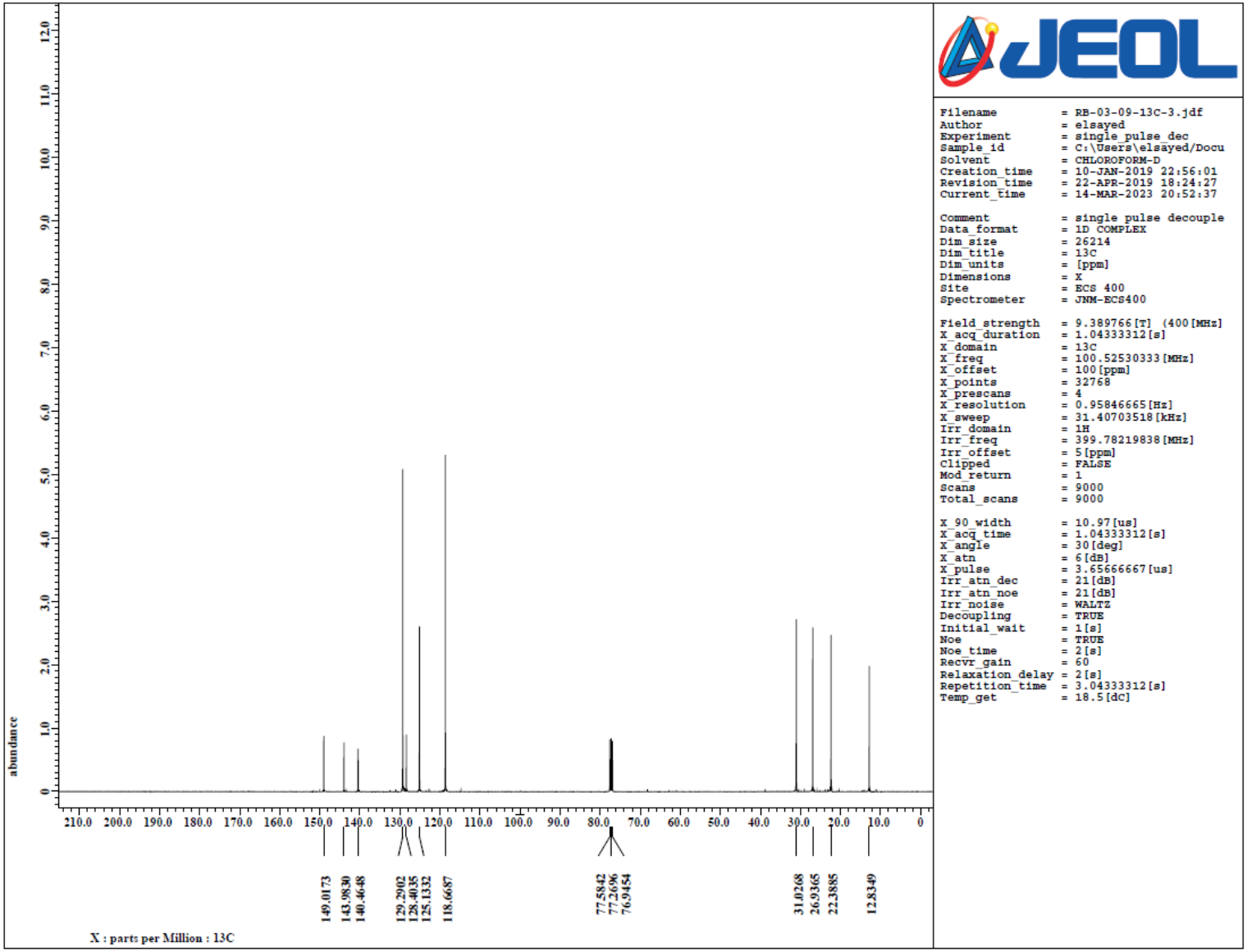

```
Filename          = RB-03-09-1H-7.jdf
Author            = elsayed
Experiment        = single_pulse.ex2
Sample_id         = C:\Users\elsayed/Docu
Solvent           = CHLOROFORM-D
Creation_time     = 10-JAN-2019 14:47:44
Revision_time     = 14-MAR-2023 20:47:14
Current_time      = 14-MAR-2023 20:49:43

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 13.575[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.7875[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 22
Relaxation_delay  = 5[s]
Repetition_time   = 7.18365952[s]
Temp_get          = 19[dC]
```

3-methyl-1-phenyl-1,4,5,6-tetrahydrocyclopenta[c]pyrazole (P8): ^{13}C-NMR ($CDCl_3$)

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Filename          = RB-03-09-13C-3.jdf
Author            = elsayed
Experiment        = single_pulse_dec
Sample_id         = C:\Users\elsayed/Docu
Solvent           = CHLOROFORM-D
Creation_time     = 10-JAN-2019 22:56:01
Revision_time     = 22-APR-2019 18:24:27
Current_time      = 14-MAR-2023 20:52:37

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 9000
Total_scans       = 9000

X_90_width        = 10.97[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6[dB]
X_pulse           = 3.65666667[us]
Irr_atn_dec       = 21[dB]
Irr_atn_noe       = 21[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 3.04333312[s]
Temp_get          = 18.5[dC]
```
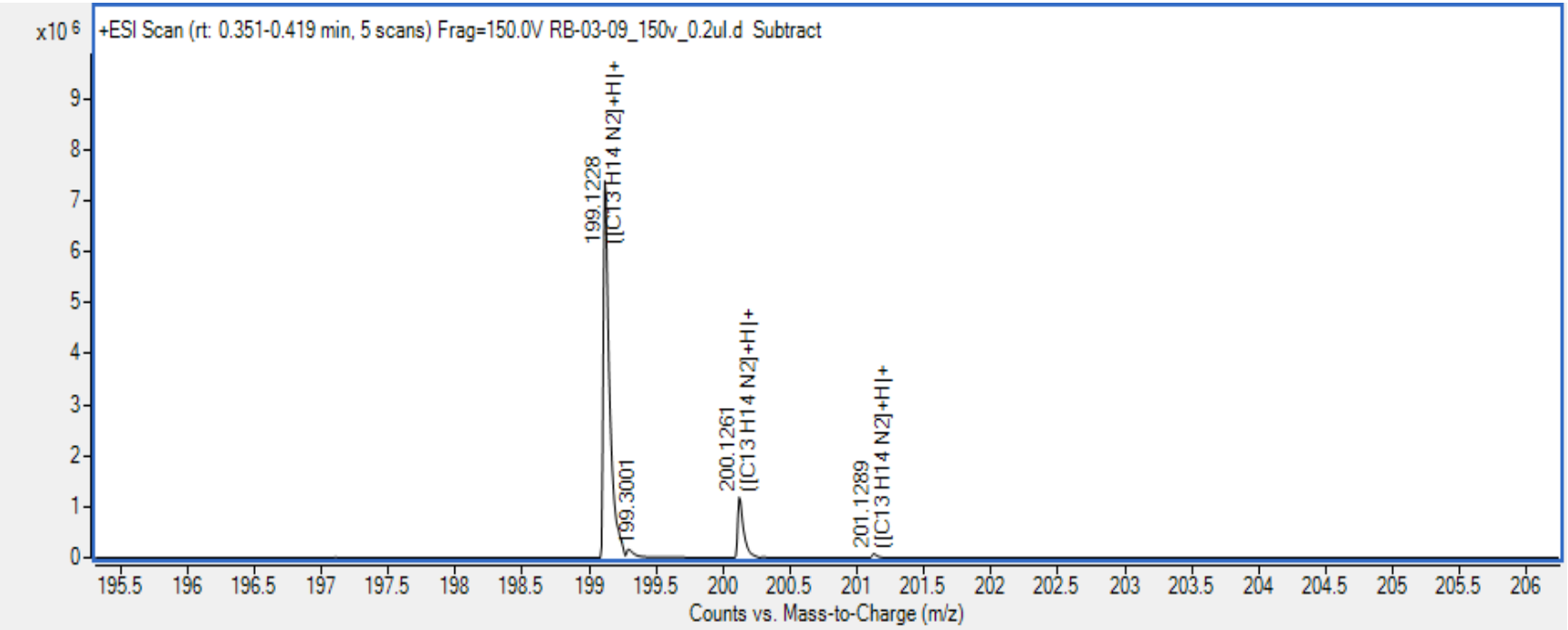
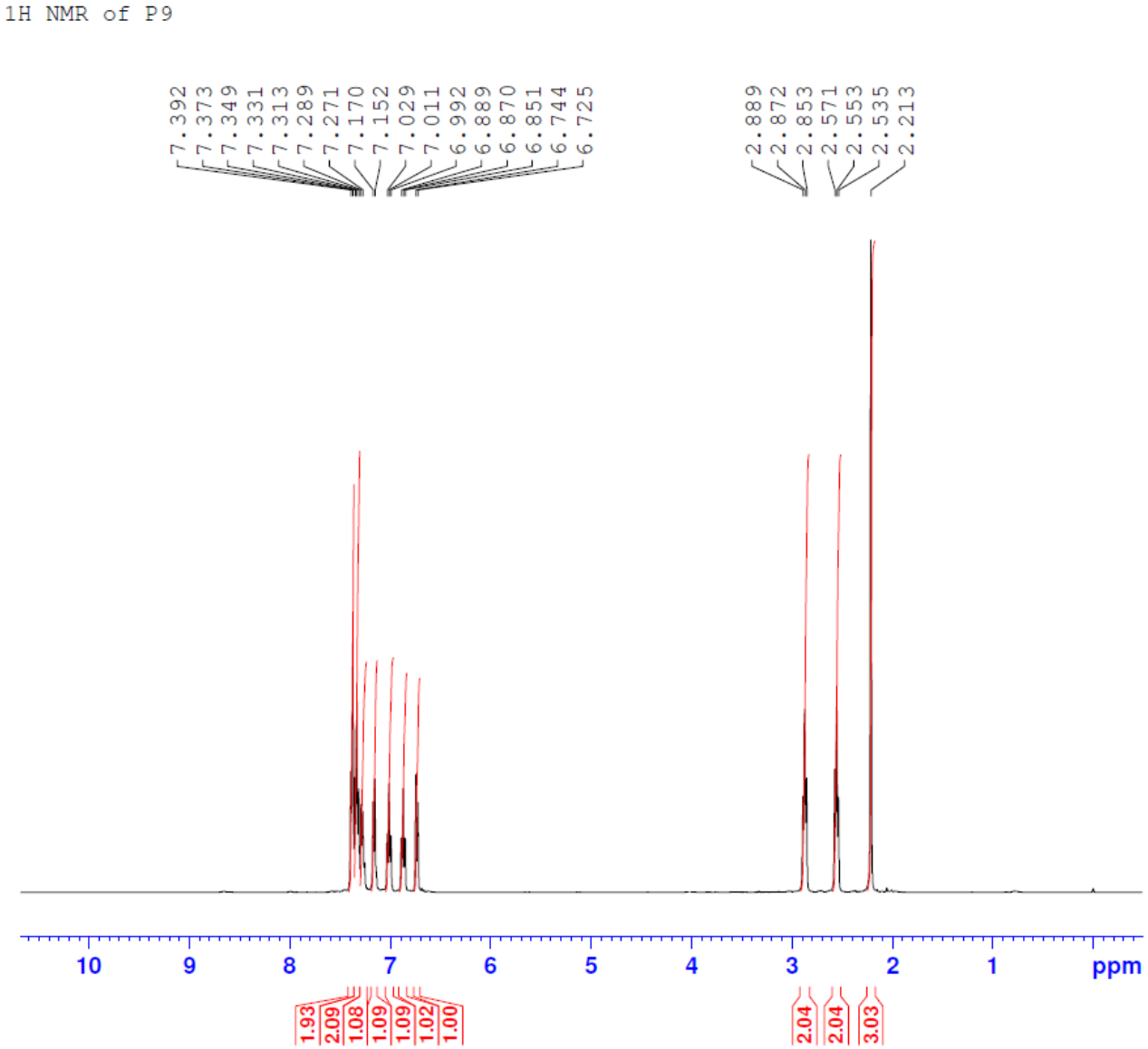

3-methyl-1-phenyl-1,4,5,6-tetrahydrocyclopenta[c]pyrazole (P8): ESI-MS



Formula,	Species,	m/z,	Score,	Diff (abs. ppm),	Mass
C13 H14 N2,	(M+H)+,	199.1228,	99.52,	0.57,	198.1156

3-methyl-2-phenyl-4,5-dihydro-2H-benzo[g]indazole (P9): ^{1}H-NMR ($CDCl_3$)



BRUKER

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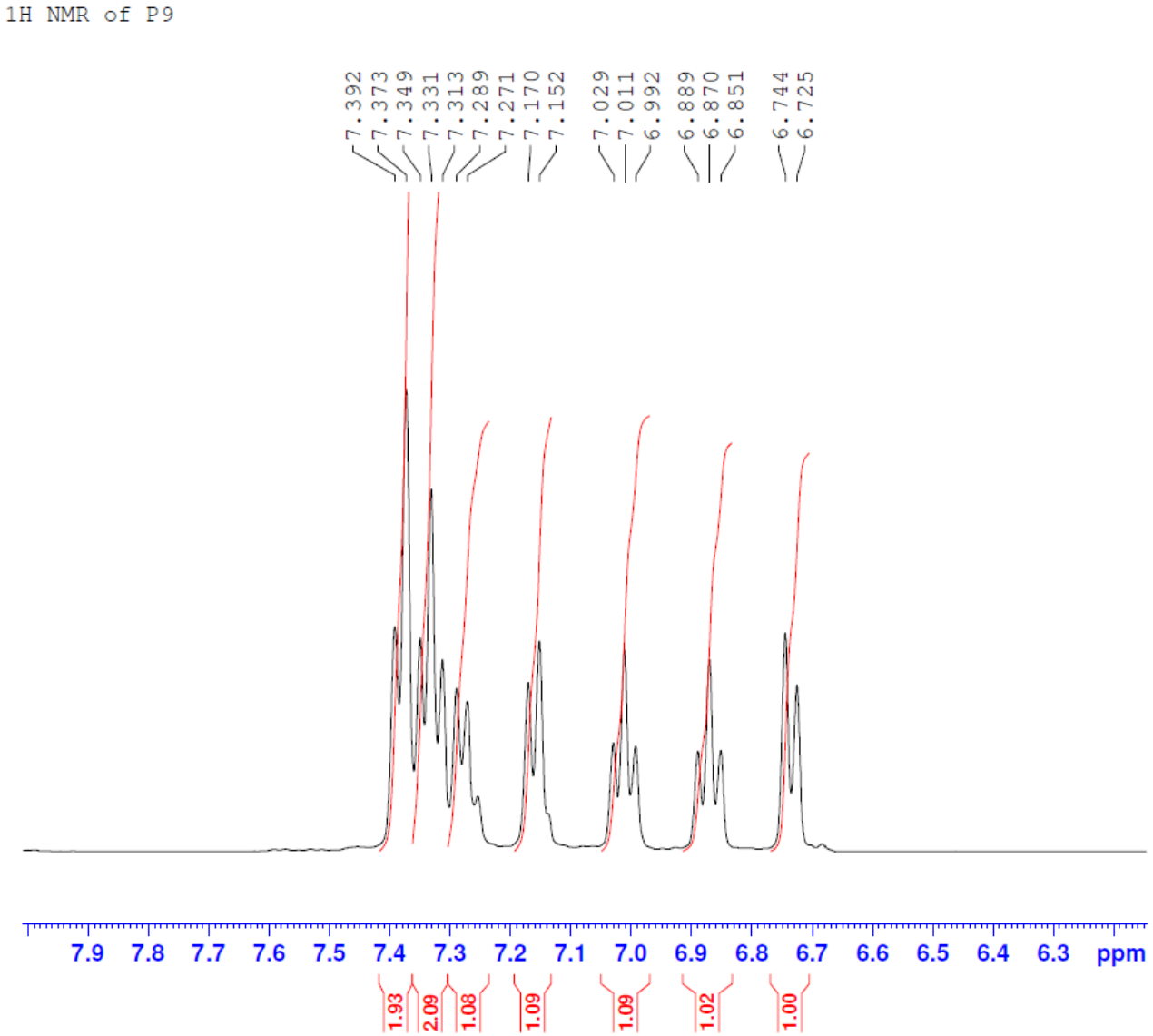
Current Data Parameters
NAME                  SM-P9_1H
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20230317
Time                     16.15 h
INSTRUM              Avance Neo
PROBHD    Z140678_0053 (
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                   8196.722 Hz
FIDRES                0.250144 Hz
AQ                    3.9976959 sec
RG                         45.2
DW                       61.000 usec
DE                       13.54 usec
TE                       296.3 K
D1                  1.00000000 sec
TD0                          1
SFO1                400.1424709 MHz
NUC1                        1H
P0                        3.33 usec
P1                       10.00 usec
PLW1                22.64200020 W

F2 - Processing parameters
SI                       65536
SF                  400.1400589 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```

3-methyl-2-phenyl-4,5-dihydro-2H-benzo[g]indazole (P9): ^{1}H-NMR ($CDCl_3$) _ Expanded Aromatic Region



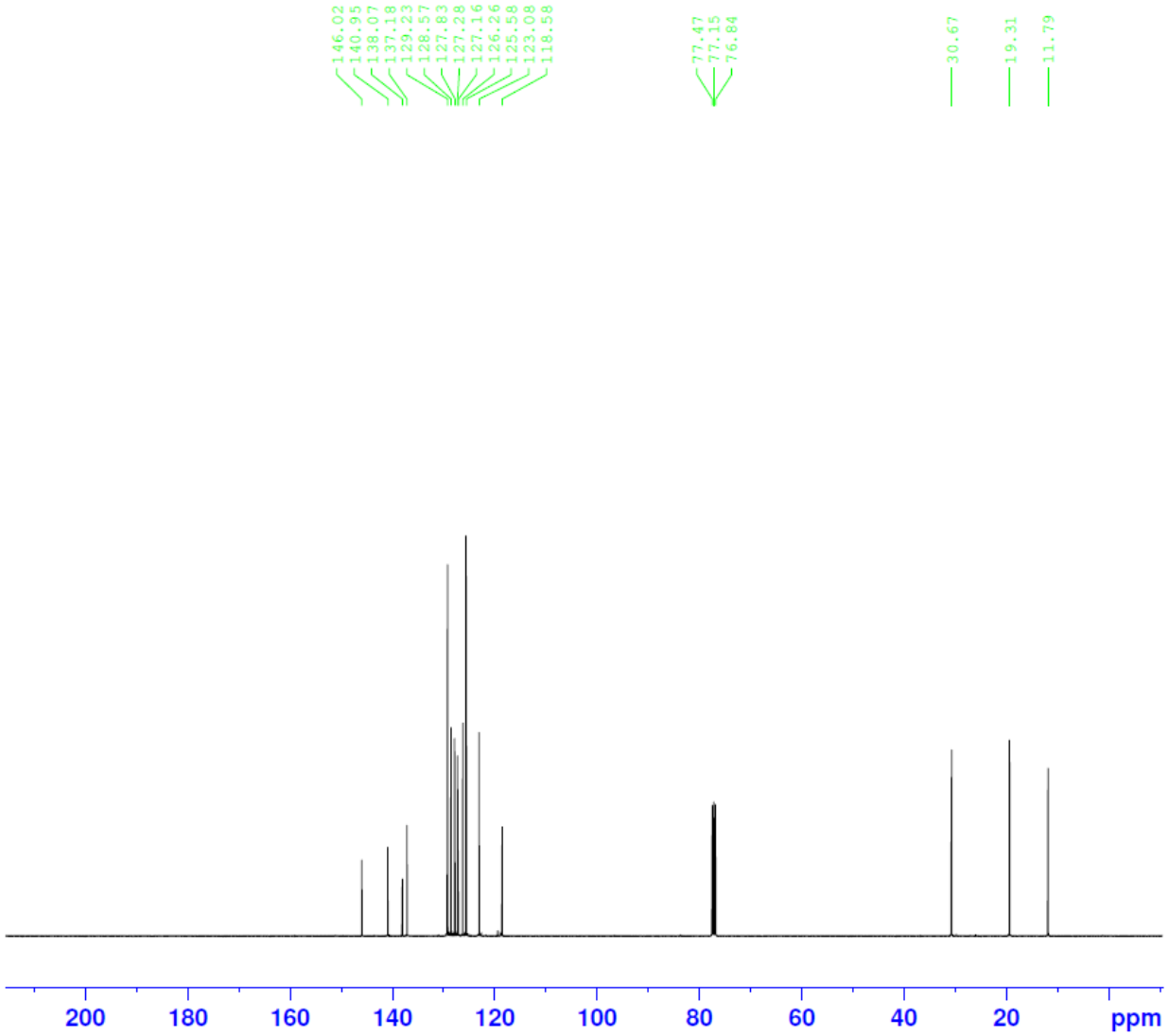
BRUKER

```
Current Data Parameters
NAME                  SM-P9_1H
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20230317
Time                     16.15 h
INSTRUM              Avance Neo
PROBHD           Z140678_0053 (
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                   8196.722 Hz
FIDRES                0.250144 Hz
AQ                    3.9976959 sec
RG                         45.2
DW                       61.000 usec
DE                       13.54 usec
TE                       296.3 K
D1                    1.00000000 sec
TD0                          1
SFO1                400.1424709 MHz
NUC1                         1H
P0                        3.33 usec
P1                       10.00 usec
PLW1                22.64200020 W

F2 - Processing parameters
SI                       65536
SF                  400.1400589 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```

3-methyl-2-phenyl-4,5-dihydro-2H-benzo[g]indazole (P9): ^{13}C-NMR ($CDCl_3$)

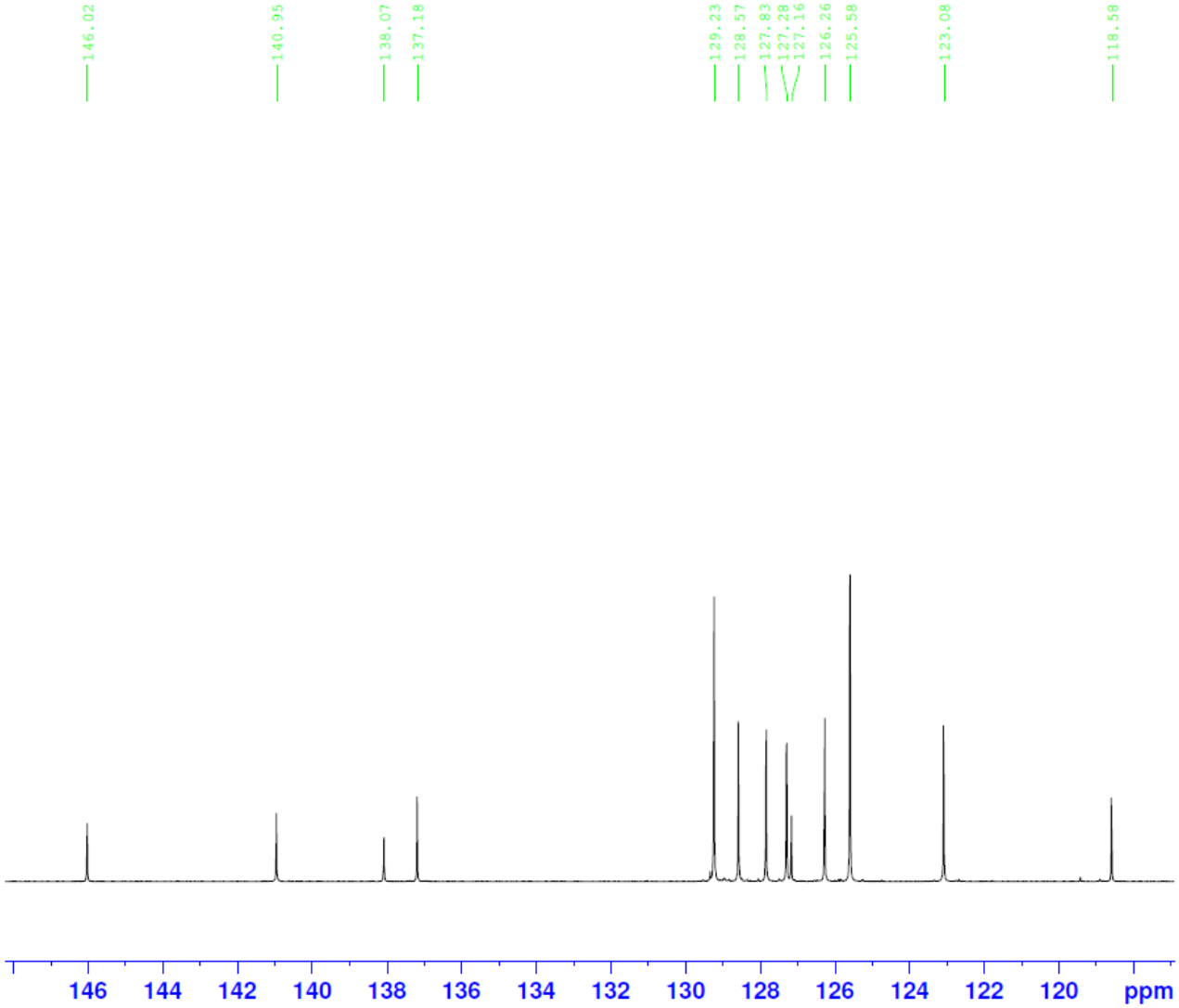



```
Current Data Parameters
NAME      SM-P-9_1Hand13C
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230318
Time               1.22 h
INSTRUM       Avance Neo
PROBHD   Z140678_0053 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           23809.523 Hz
FIDRES         0.726609 Hz
AQ            1.3762560 sec
RG                   16
DW                21.000 usec
DE                 6.50 usec
TE                296.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6253446 MHz
NUC1               13C
P0                 3.33 usec
P1                10.00 usec
PLW1        77.80000305 W
SFO2        400.1416006 MHz
NUC2               1H
CPDPRG[2        waltz65
PCPD2             90.00 usec
PLW2        22.64200020 W
PLW12        0.27952999 W
PLW13        0.14060000 W

F2 - Processing parameters
SI                32768
SF          100.6152830 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

3-methyl-2-phenyl-4,5-dihydro-2H-benzo[g]indazole (P9): ^{13}C-NMR ($CDCl_3$) _ Expanded Aromatic Region

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Current Data Parameters
NAME      SM-P-9_1Hand13C
EXPNO                  11
PROCNO                  1

F2 - Acquisition Parameters
Date_           20230318
Time                1.22 h
INSTRUM       Avance Neo
PROBHD    Z140678_0053 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           23809.523 Hz
FIDRES         0.726609 Hz
AQ            1.3762560 sec
RG                   16
DW                21.000 usec
DE                 6.50 usec
TE                296.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         100.6253446 MHz
NUC1                 13C
P0                 3.33 usec
P1                10.00 usec
PLW1         77.80000305 W
SFO2         400.1416006 MHz
NUC2                 1H
CPDPRG[2         waltz65
PCPD2              90.00 usec
PLW2         22.64200020 W
PLW12         0.27952999 W
PLW13         0.14060000 W

F2 - Processing parameters
SI                 32768
SF           100.6152830 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```
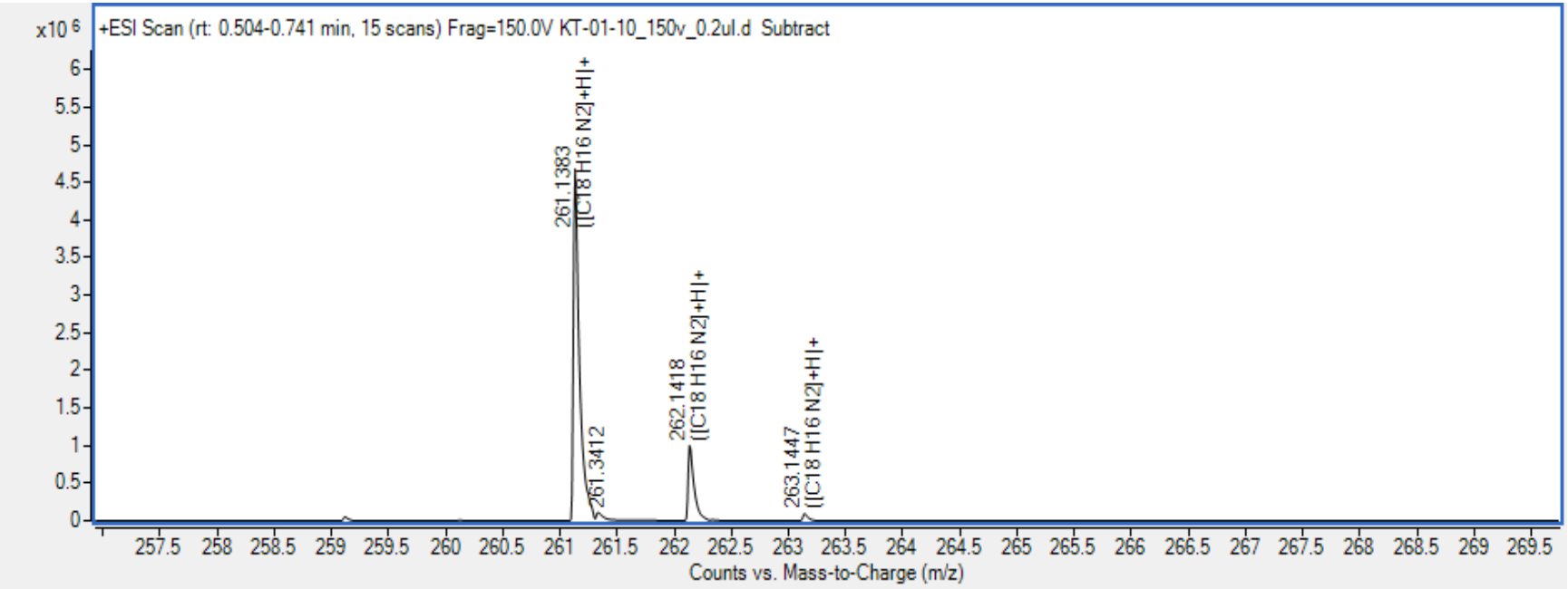
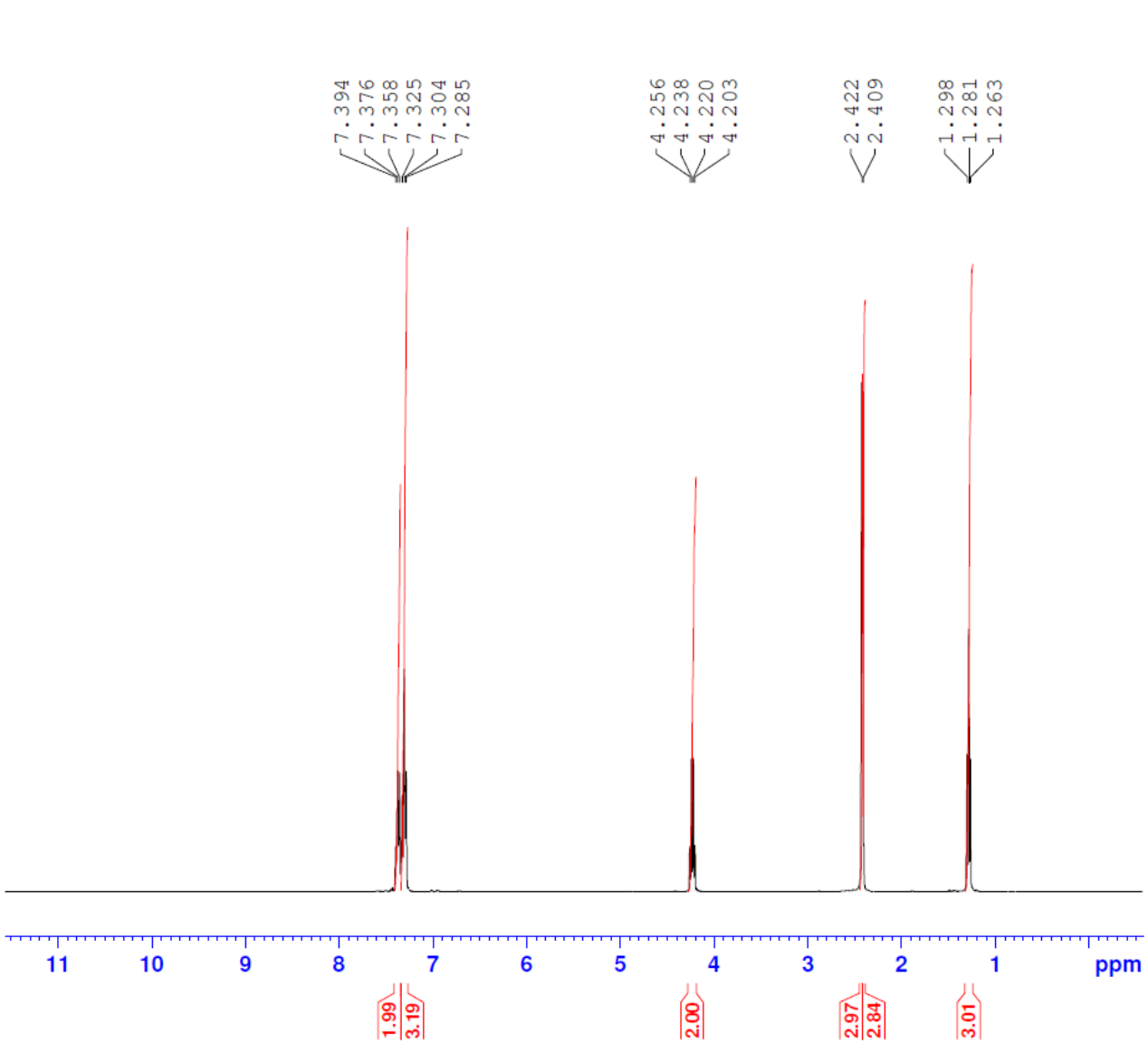

3-methyl-2-phenyl-4,5-dihydro-2H-benzo[g]indazole (P9): ESI-MS



Formula,	Species,	m/z,	Score,	Diff (abs. ppm),	Mass
C18 H16 N2,	(M+H)+,	261.1383,	98.93,	1.11,	260.1311

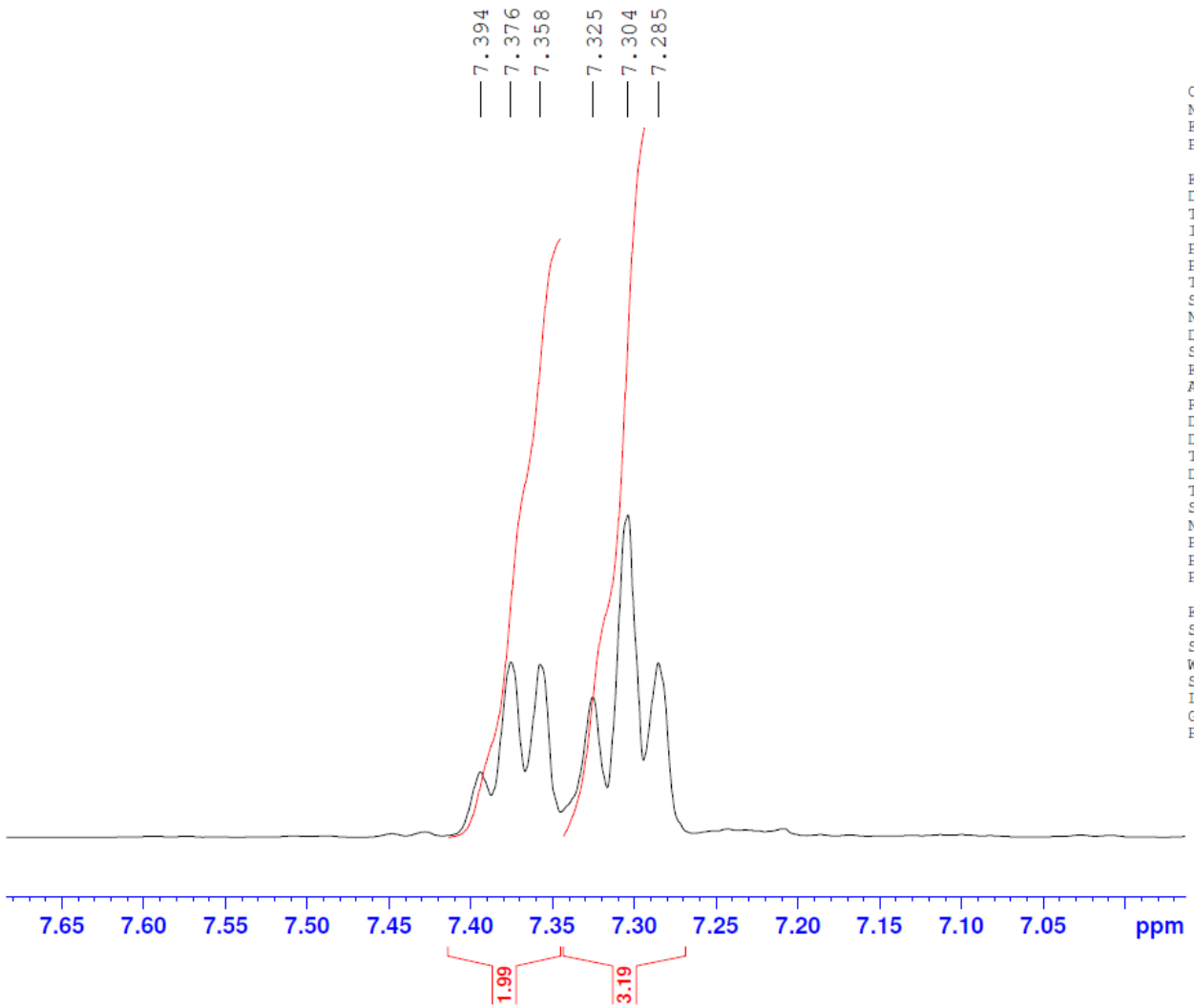
Ethyl 3,5-dimethyl-1-phenyl-1H-pyrazole-4-carboxylate (P10): ^{1}H-NMR ($CDCl_3$)



BRUKER

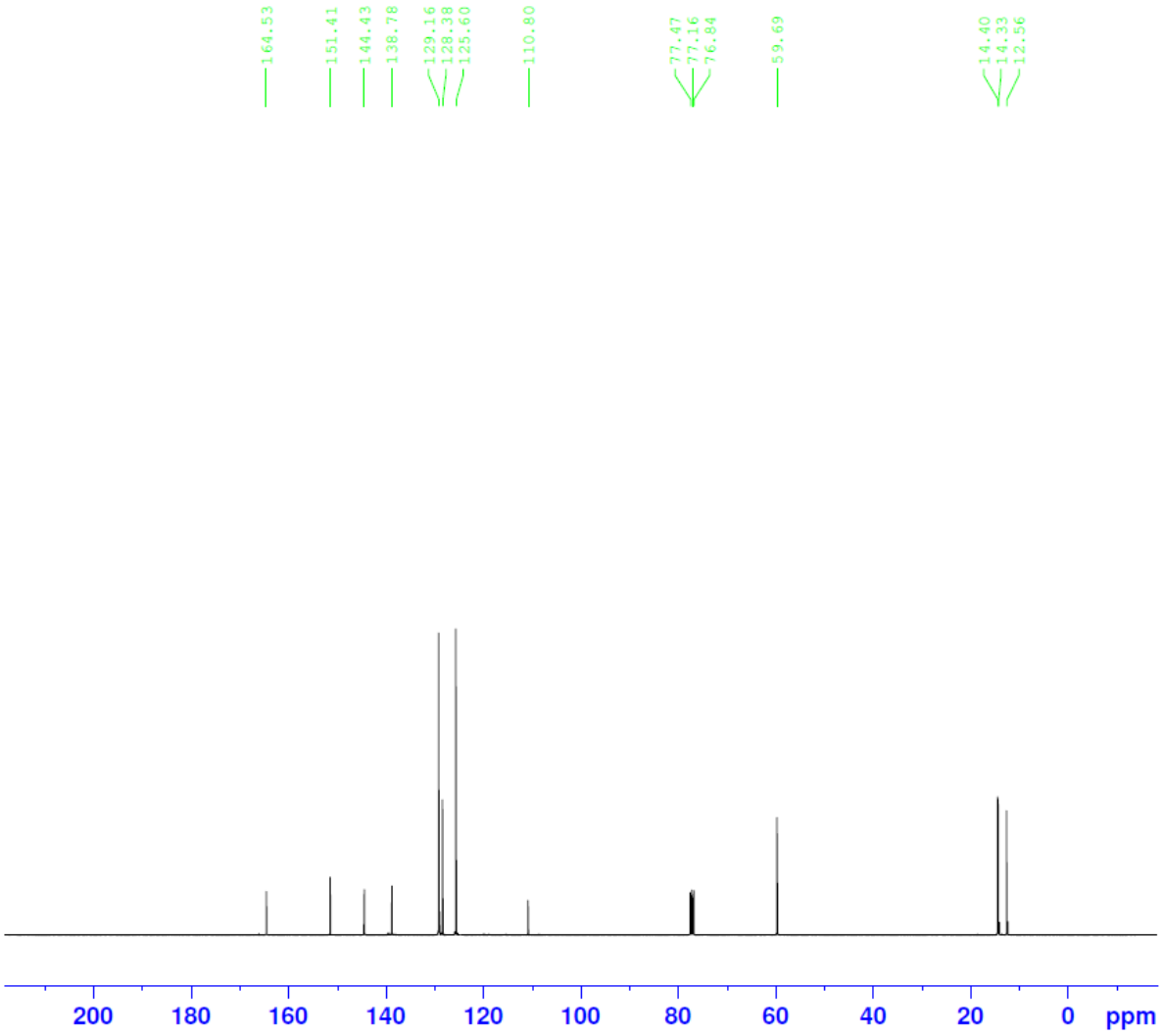
```
Current Data Parameters
NAME      SM-P10_1Hand13C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230318
Time                1.28 h
INSTRUM       Avance Neo
PROBHD   Z140678_0053 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8196.722 Hz
FIDRES          0.250144 Hz
AQ              3.9976959 sec
RG                    36
DW                61.000 usec
DE                13.54 usec
TE                296.0 K
D1           1.00000000 sec
TD0                    1
SFO1         400.1424709 MHz
NUC1                  1H
P0                 3.33 usec
P1                10.00 usec
PLW1         22.64200020 W

F2 - Processing parameters
SI                 65536
SF           400.1400291 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

Ethyl 3,5-dimethyl-1-phenyl-1H-pyrazole-4-carboxylate (P10): ^{1}H-NMR ($CDCl_3$) _ Expanded Aromatic Region



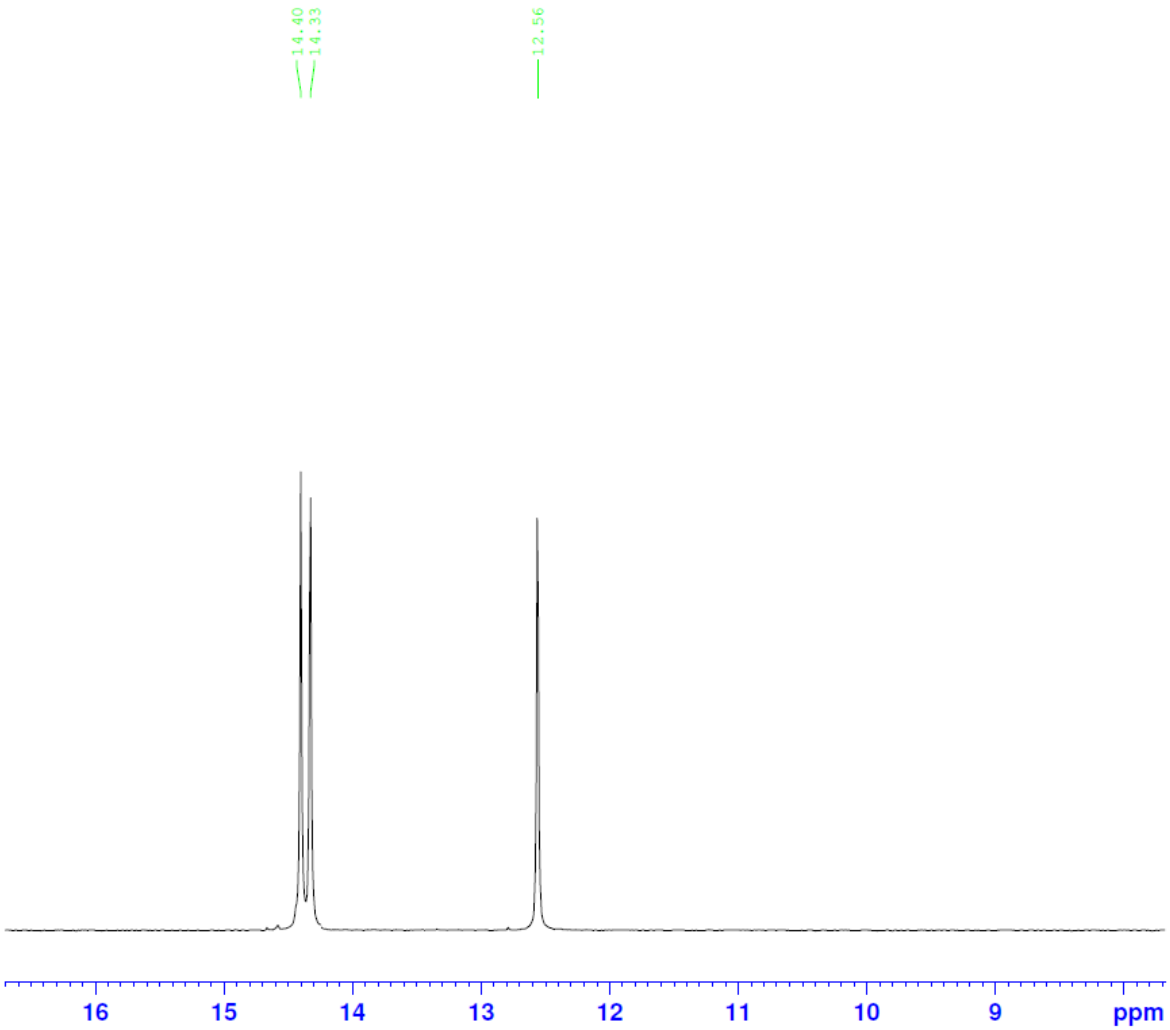

```
Current Data Parameters
NAME      SM-P10_1Hand13C
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230318
Time                1.28 h
INSTRUM       Avance Neo
PROBHD    Z140678_0053 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8196.722 Hz
FIDRES          0.250144 Hz
AQ              3.9976959 sec
RG                    36
DW                61.000 usec
DE                13.54 usec
TE                296.0 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1424709 MHz
NUC1                  1H
P0                 3.33 usec
P1                10.00 usec
PLW1          22.64200020 W

F2 - Processing parameters
SI                 65536
SF            400.1400291 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

Ethyl 3,5-dimethyl-1-phenyl-1H-pyrazole-4-carboxylate (P10): ^{13}C-NMR ($CDCl_3$)



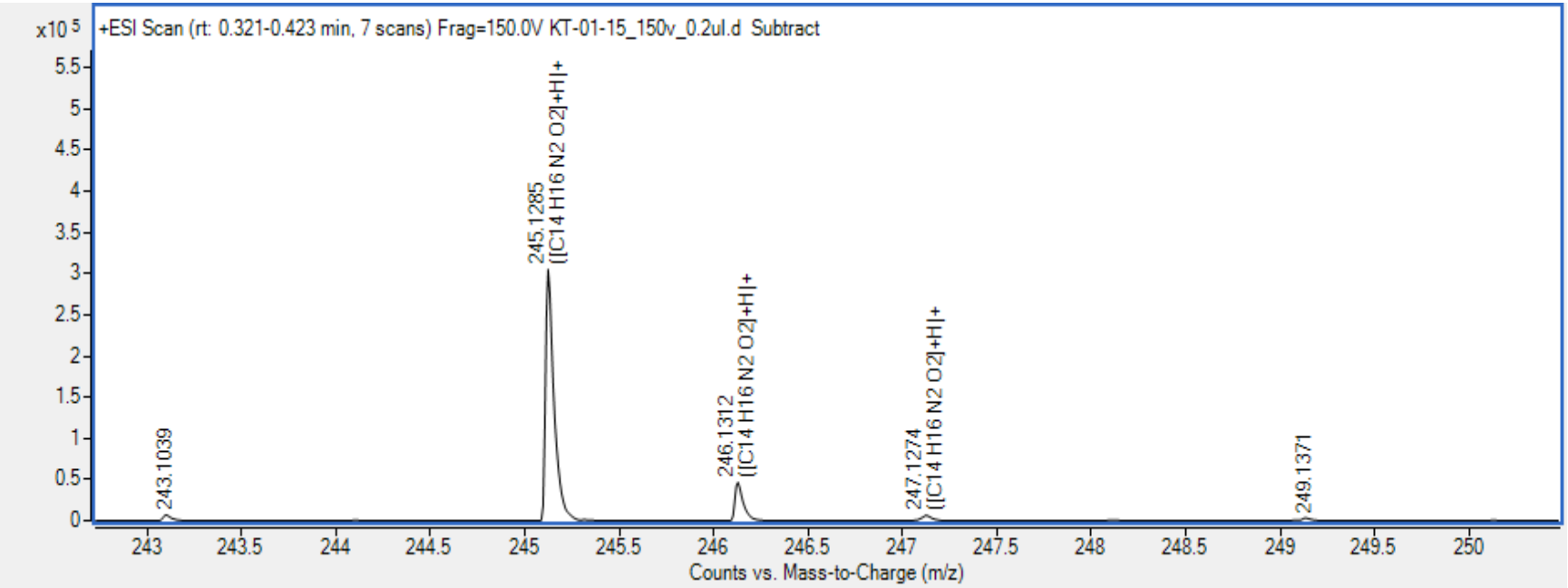

```
Current Data Parameters
NAME      SM-P10_1Hand13C
EXPNO                  11
PROCNO                  1

F2 - Acquisition Parameters
Date_           20230318
Time                5.24 h
INSTRUM       Avance Neo
PROBHD   Z140678_0053 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           23809.523 Hz
FIDRES         0.726609 Hz
AQ            1.3762560 sec
RG                 12.7
DW               21.000 usec
DE                6.50 usec
TE                296.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6253446 MHz
NUC1               13C
P0                3.33 usec
P1               10.00 usec
PLW1        77.80000305 W
SFO2        400.1416006 MHz
NUC2               1H
CPDPRG[2        waltz65
PCPD2             90.00 usec
PLW2        22.64200020 W
PLW12        0.27952999 W
PLW13        0.14060000 W

F2 - Processing parameters
SI                32768
SF          100.6152830 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

Ethyl 3,5-dimethyl-1-phenyl-1H-pyrazole-4-carboxylate (P10): ^{13}C-NMR ($CDCl_3$) _ Expanded Aliphatic Region



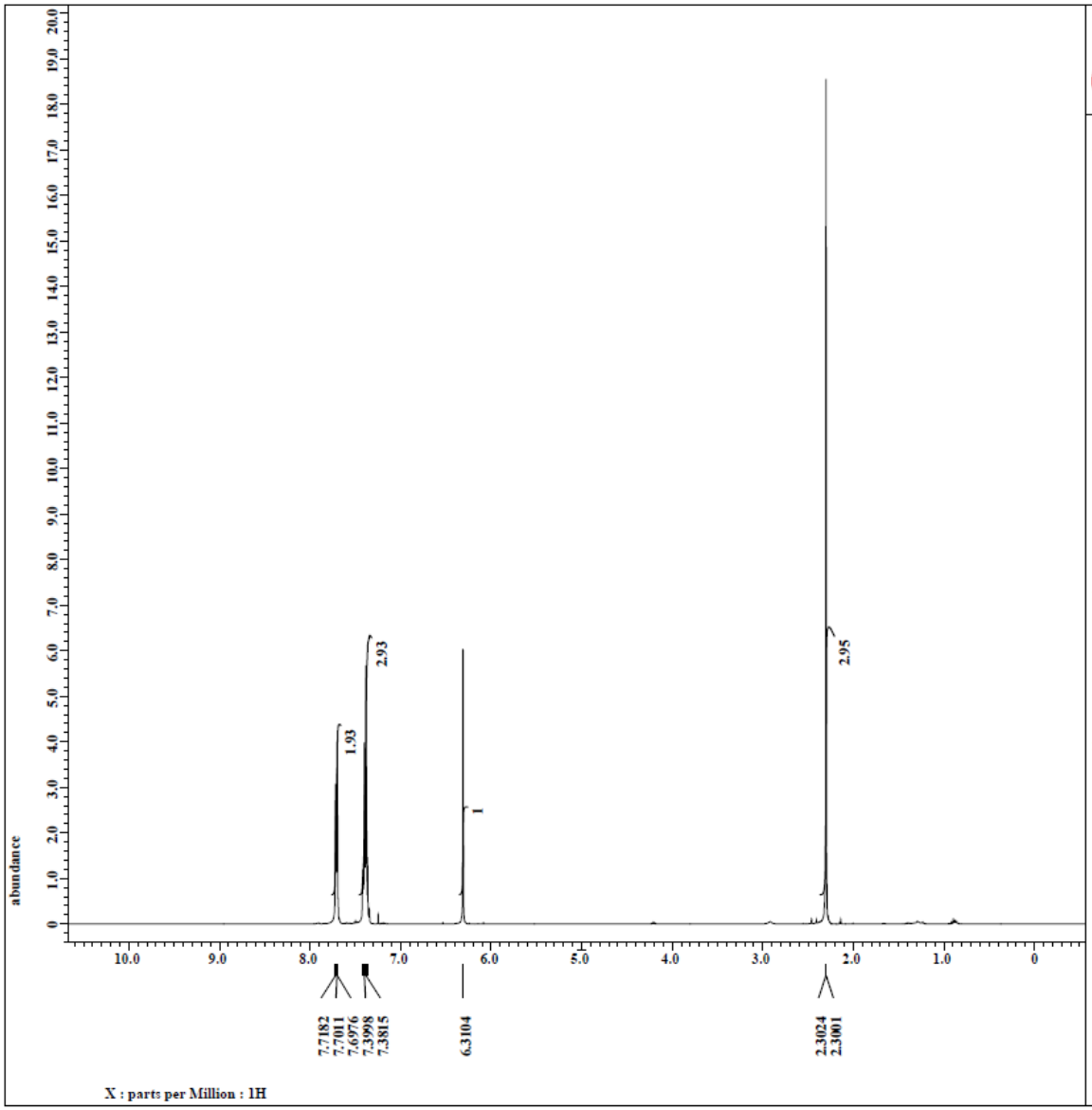

```
Current Data Parameters
NAME      SM-P10_1Hand13C
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230318
Time                5.24 h
INSTRUM       Avance Neo
PROBHD    Z140678_0053 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           23809.523 Hz
FIDRES         0.726609 Hz
AQ             1.3762560 sec
RG                 12.7
DW                21.000 usec
DE                 6.50 usec
TE                296.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6253446 MHz
NUC1               13C
P0                 3.33 usec
P1                10.00 usec
PLW1        77.80000305 W
SFO2        400.1416006 MHz
NUC2               1H
CPDPRG[2         waltz65
PCPD2             90.00 usec
PLW2        22.64200020 W
PLW12        0.27952999 W
PLW13        0.14060000 W

F2 - Processing parameters
SI                32768
SF          100.6152830 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

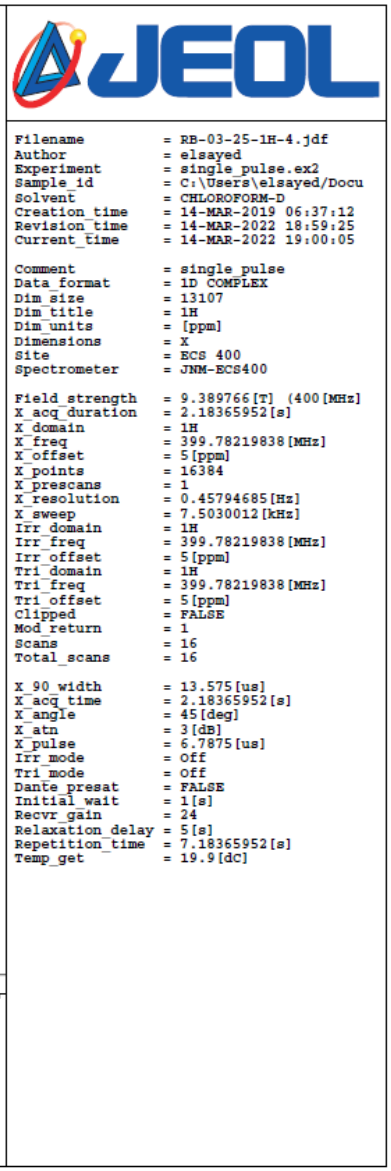
Ethyl 3,5-dimethyl-1-phenyl-1H-pyrazole-4-carboxylate (P10): ESI-MS



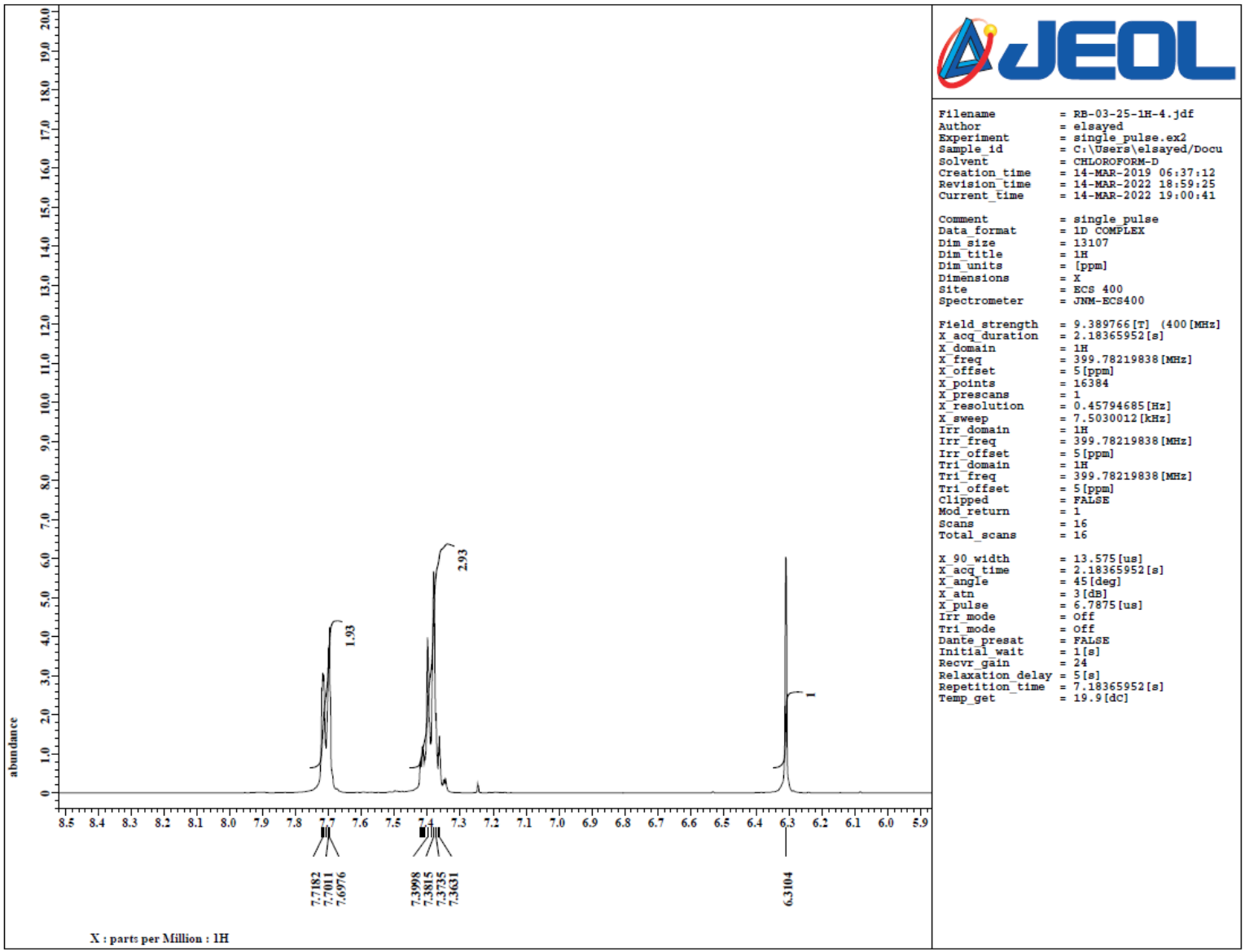
Formula,	Species,	m/z,	Score,	Diff (abs. ppm),	Mass
C14 H16 N2 O2,	(M+H)+,	245.1285,	97.86,	0.62,	244.121

5-methyl-3-phenylisoxazole (P11): ^{1}H-NMR ($CDCl_3$)





5-methyl-3-phenylisoxazole (P11): ^{1}H-NMR ($CDCl_3$) _ Aromatic Region Expanded

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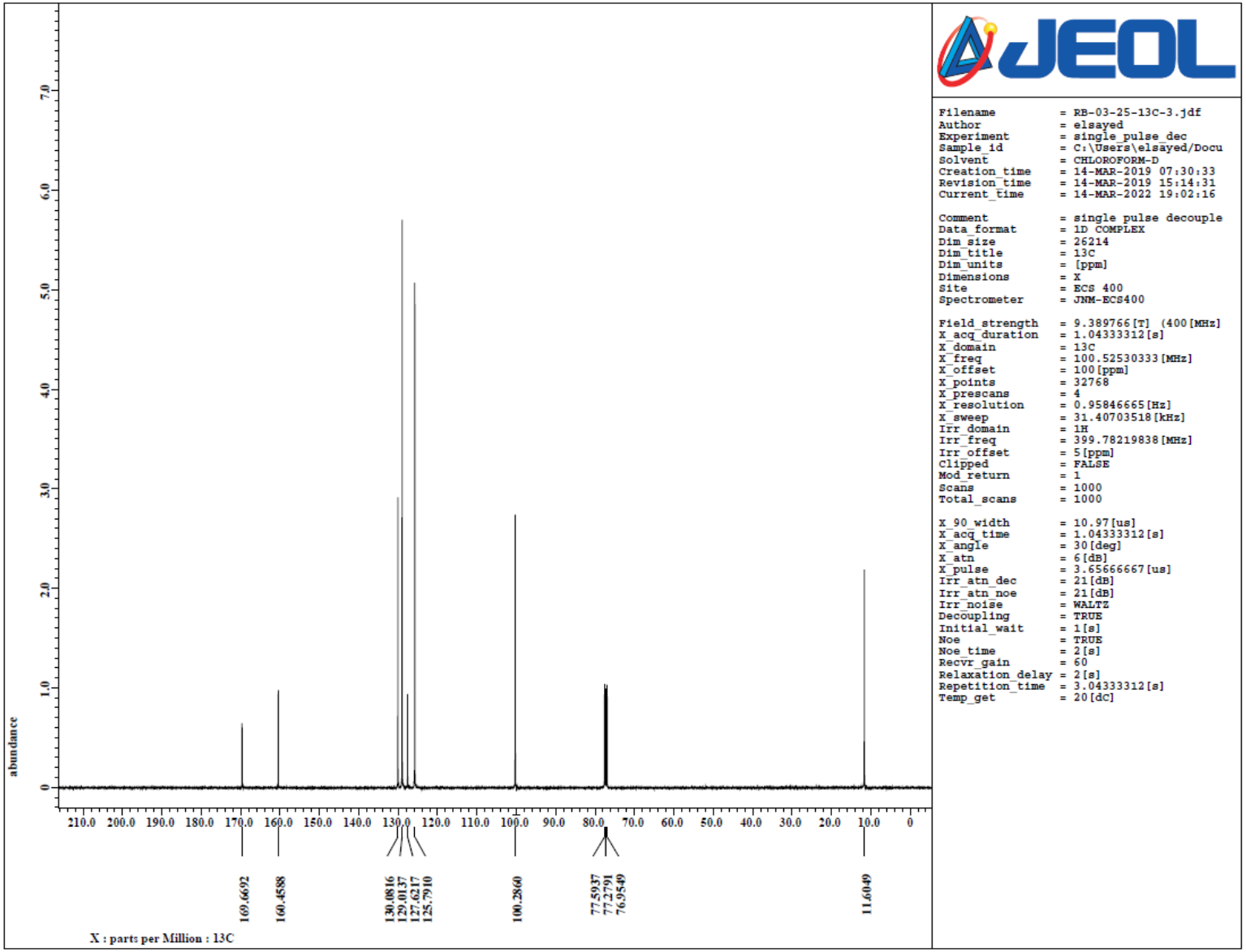
Filename          = RB-03-25-1H-4.jdf
Author            = elsayed
Experiment        = single_pulse.ex2
Sample_id         = C:\Users\elsayed/Docu
Solvent           = CHLOROFORM-D
Creation_time     = 14-MAR-2019 06:37:12
Revision_time     = 14-MAR-2022 18:59:25
Current_time      = 14-MAR-2022 19:00:41

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

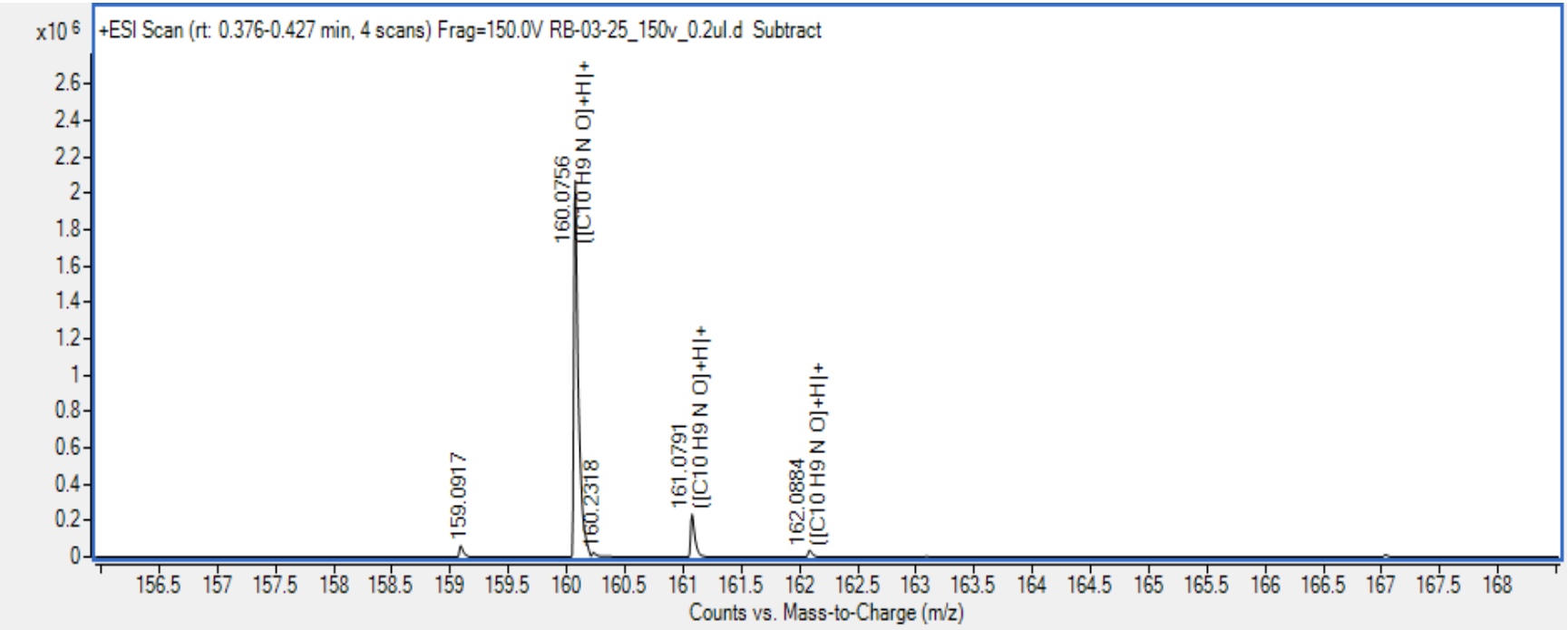
Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 13.575[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.7875[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 24
Relaxation_delay  = 5[s]
Repetition_time   = 7.18365952[s]
Temp_get          = 19.9[dC]
```

5-methyl-3-phenylisoxazole (P11): ^{13}C-NMR ($CDCl_3$)

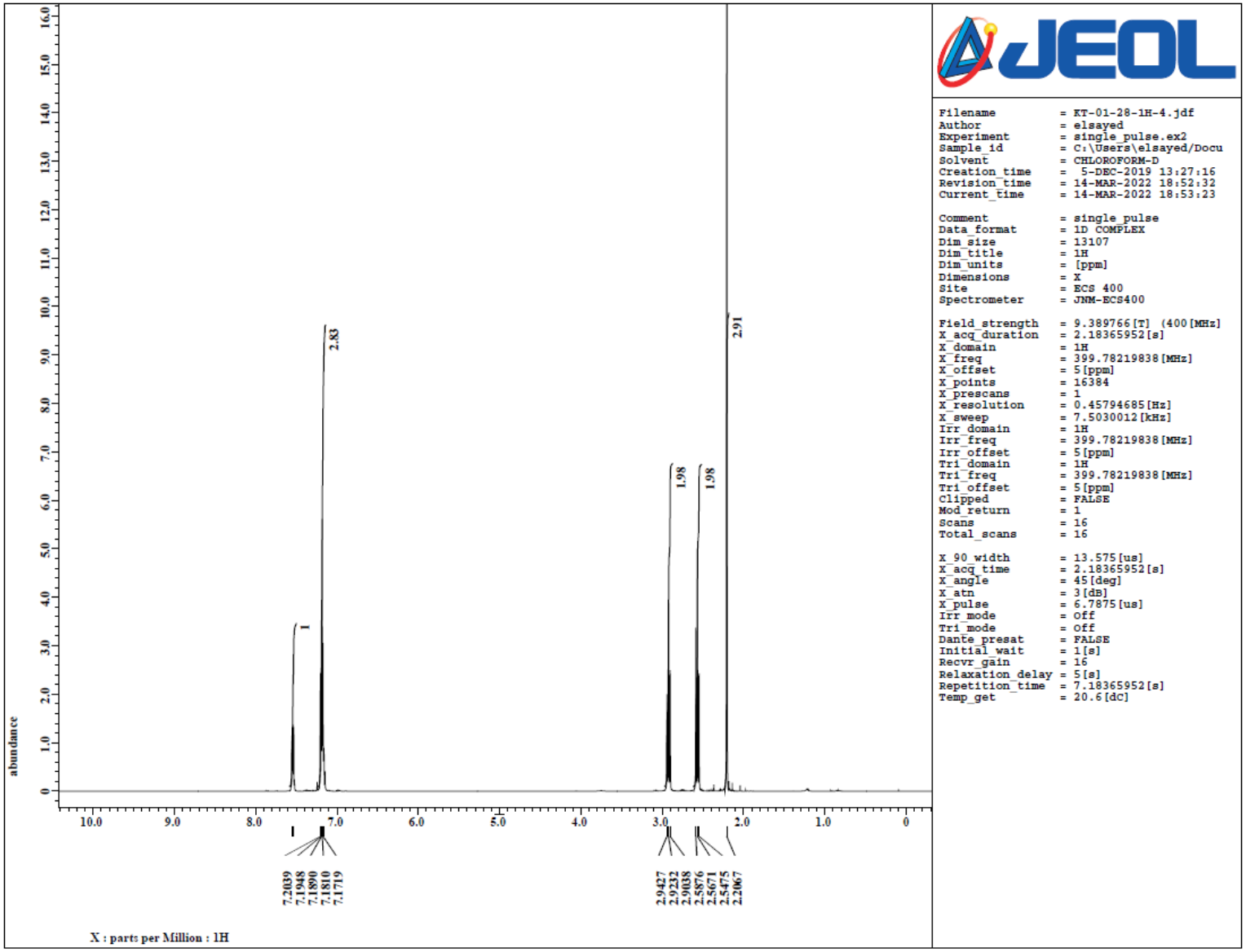


5-methyl-3-phenylisoxazole (P11): ESI-MS



Formula,	Species,	m/z,	Score,	Diff (abs. ppm),	Mass
C10 H9 N O,	(M+H)+,	160.0756,	97.86,	0.38,	159.0685

3-methyl-4,5-dihydronaphtho[1,2-c]isoxazole (P12): ^{1}H-NMR ($CDCl_3$)

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Filename          = KT-01-28-1H-4.jdf
Author            = elsayed
Experiment        = single_pulse.ex2
Sample_id         = C:\Users\elsayed/Docu
Solvent           = CHLOROFORM-D
Creation_time     =  5-DEC-2019 13:27:16
Revision_time     = 14-MAR-2022 18:52:32
Current_time      = 14-MAR-2022 18:53:23

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 13.575[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.7875[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 16
Relaxation_delay  = 5[s]
Repetition_time   = 7.18365952[s]
Temp_get          = 20.6[dC]
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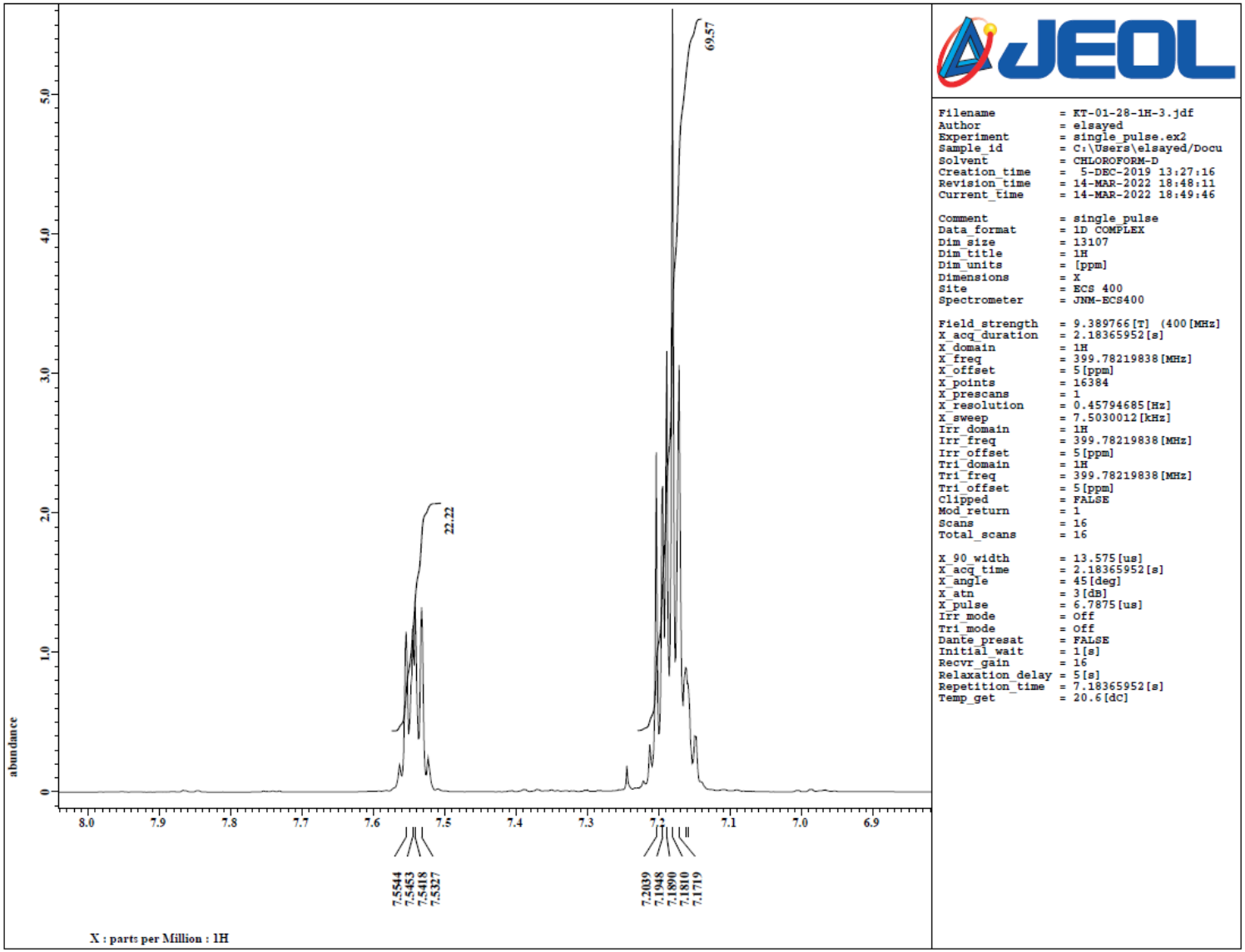
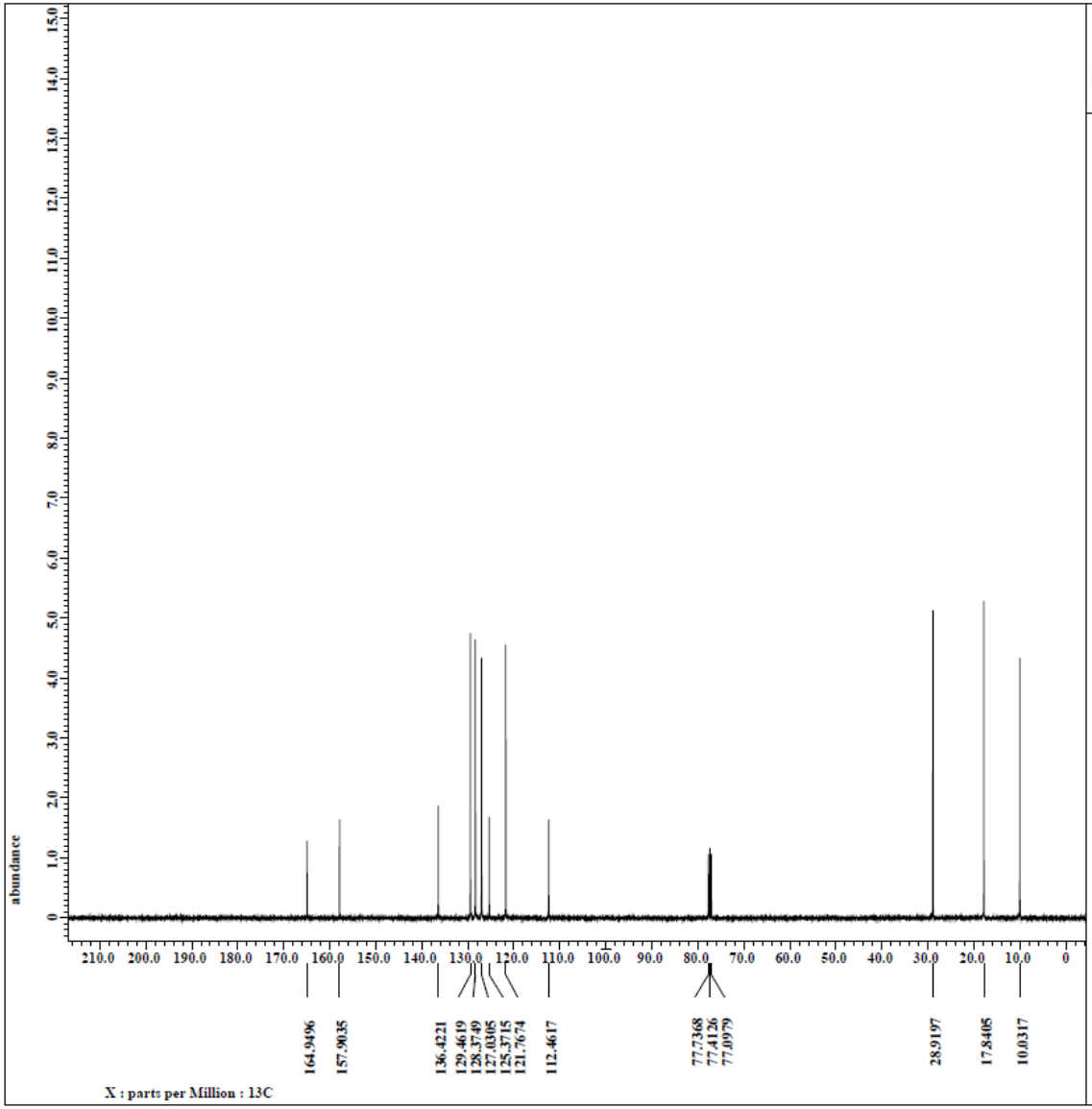
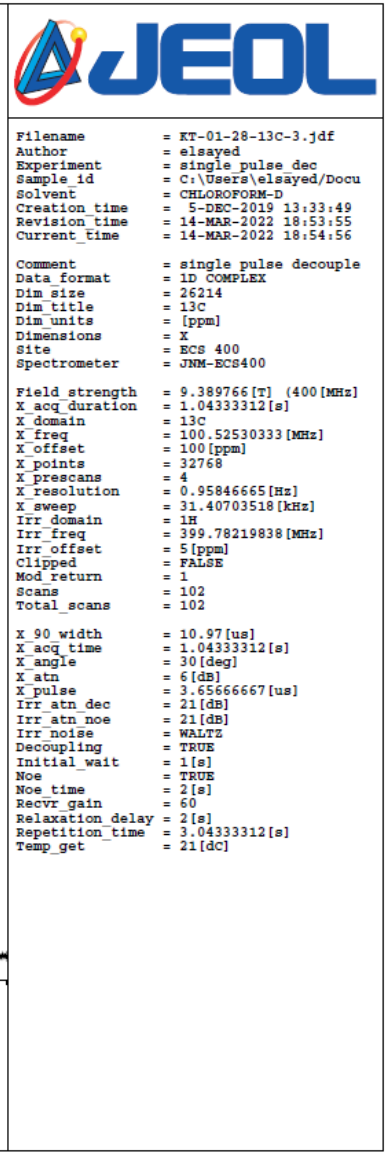
3-methyl-4,5-dihydronaphtho[1,2-c]isoxazole (P12): ^{1}H-NMR ($CDCl_3$) _ Expanded Aromatic Region

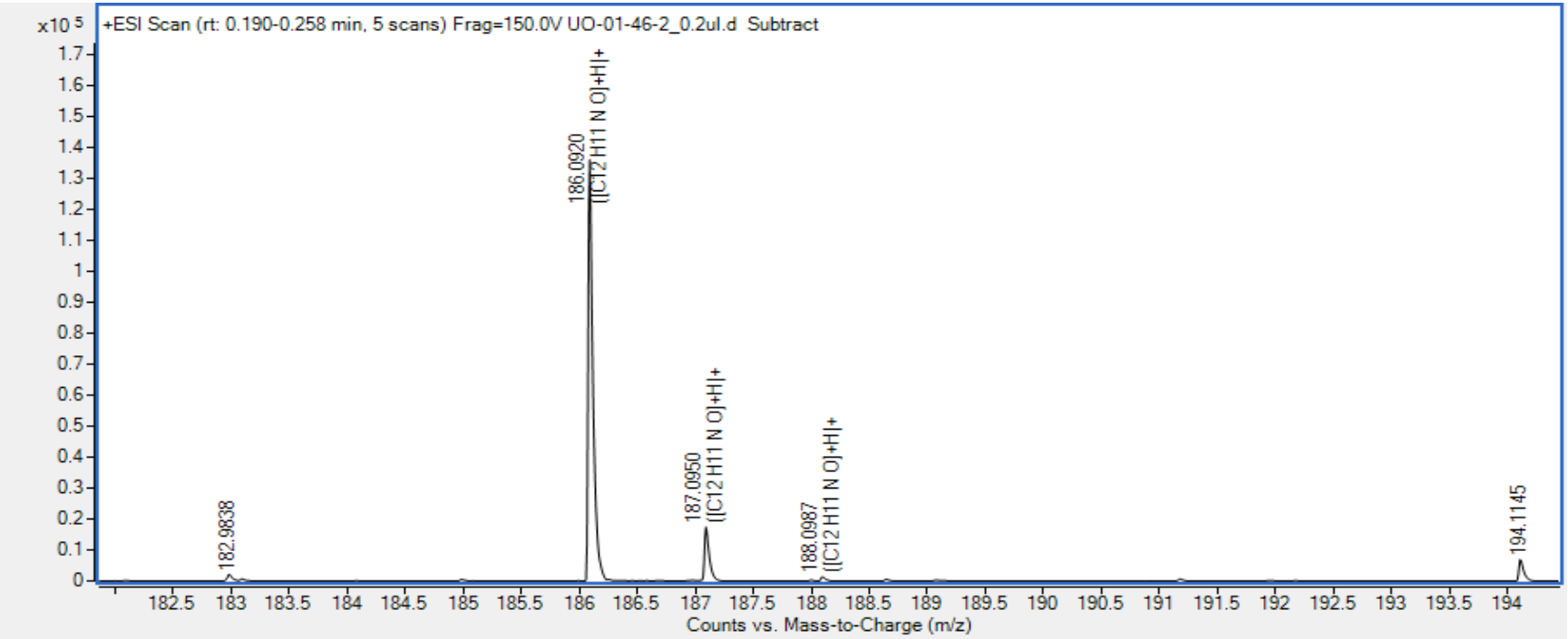


3-methyl-4,5-dihydronaphtho[1,2-c]isoxazole (P12): ^{13}C-NMR ($CDCl_3$)



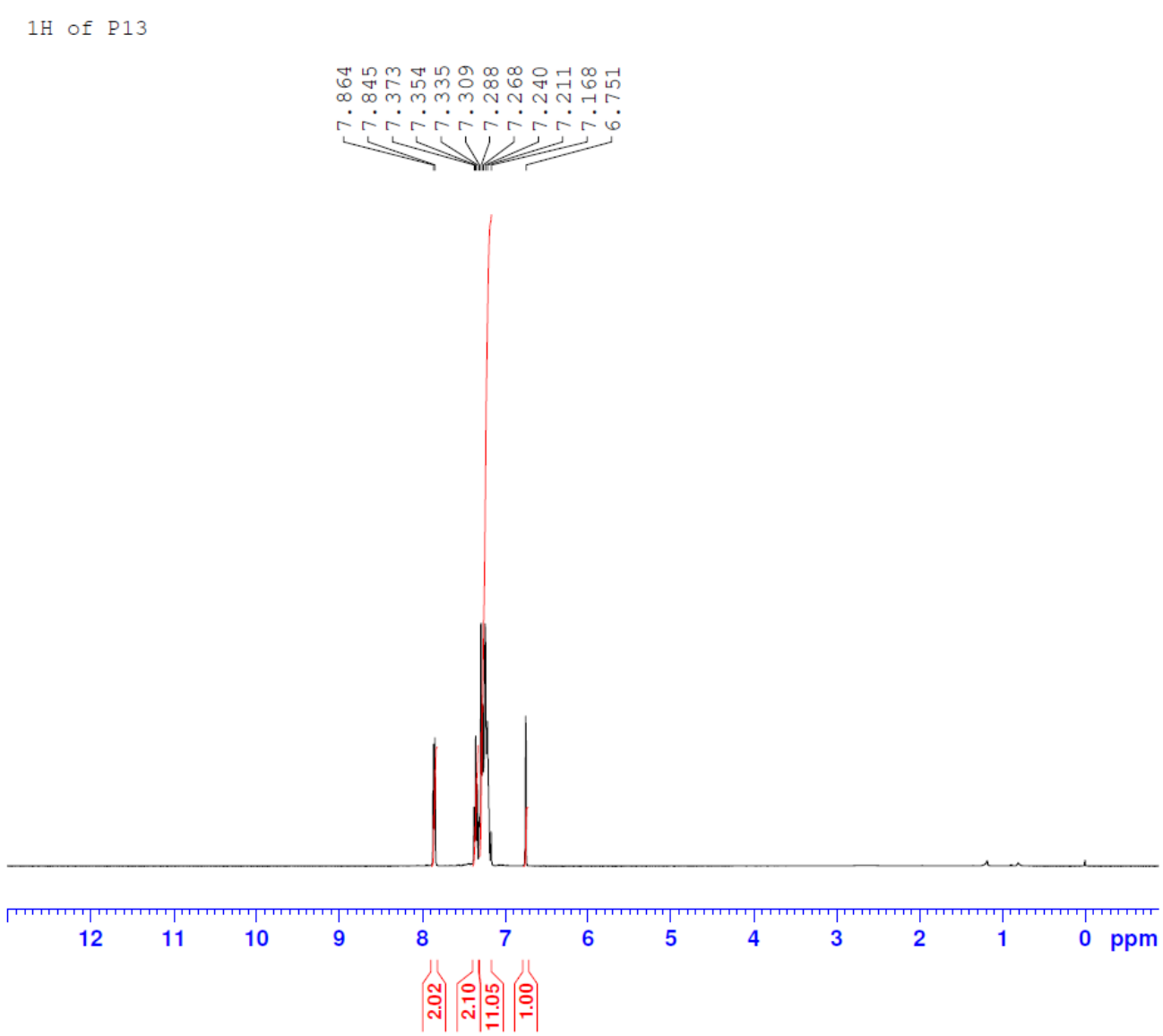


3-methyl-4,5-dihydronaphtho[1,2-c]isoxazole (P12): ESI-MS



Formula,	Species,	m/z,	Diff (abs. ppm),	Score,	Mass
C12 H11 N O,	(M+H)+,	186.0920,	3.59,	97.51,	185.0847

1,3,5-triphenyl-1H-pyrazole (P13): ^{1}H-NMR ($CDCl_3$)

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Current Data Parameters
NAME      SM_Mar21_13_1H
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230321
Time              19.49 h
INSTRUM       Avance Neo
PROBHD   Z140678_0053 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8196.722 Hz
FIDRES          0.250144 Hz
AQ              3.9976959 sec
RG                   101
DW                61.000 usec
DE                13.54 usec
TE                296.4 K
D1            1.00000000 sec
TD0                    1
SFO1         400.1424709 MHz
NUC1                  1H
P0                 3.33 usec
P1                10.00 usec
PLW1         22.64200020 W

F2 - Processing parameters
SI                 65536
SF           400.1400465 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
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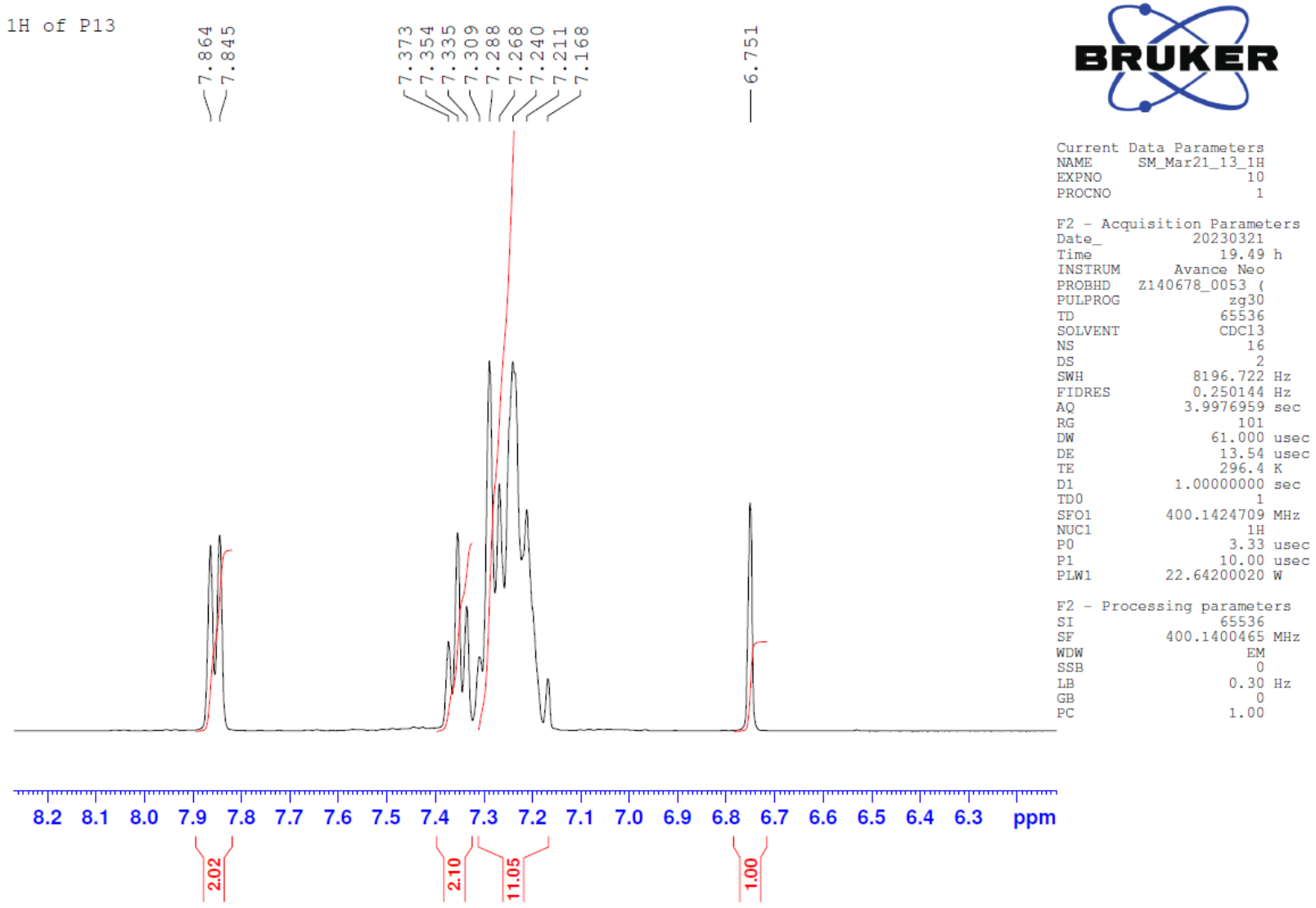
1,3,5-triphenyl-1H-pyrazole (P13): ^{1}H-NMR ($CDCl_3$) _ Expanded Aromatic Region

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Current Data Parameters
NAME      SM_Mar21_13_1H
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230321
Time              19.49 h
INSTRUM       Avance Neo
PROBHD   Z140678_0053 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8196.722 Hz
FIDRES          0.250144 Hz
AQ              3.9976959 sec
RG                   101
DW                61.000 usec
DE                13.54 usec
TE                296.4 K
D1           1.00000000 sec
TD0                    1
SFO1         400.1424709 MHz
NUC1                  1H
P0                 3.33 usec
P1                10.00 usec
PLW1         22.64200020 W

F2 - Processing parameters
SI                 65536
SF           400.1400465 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```
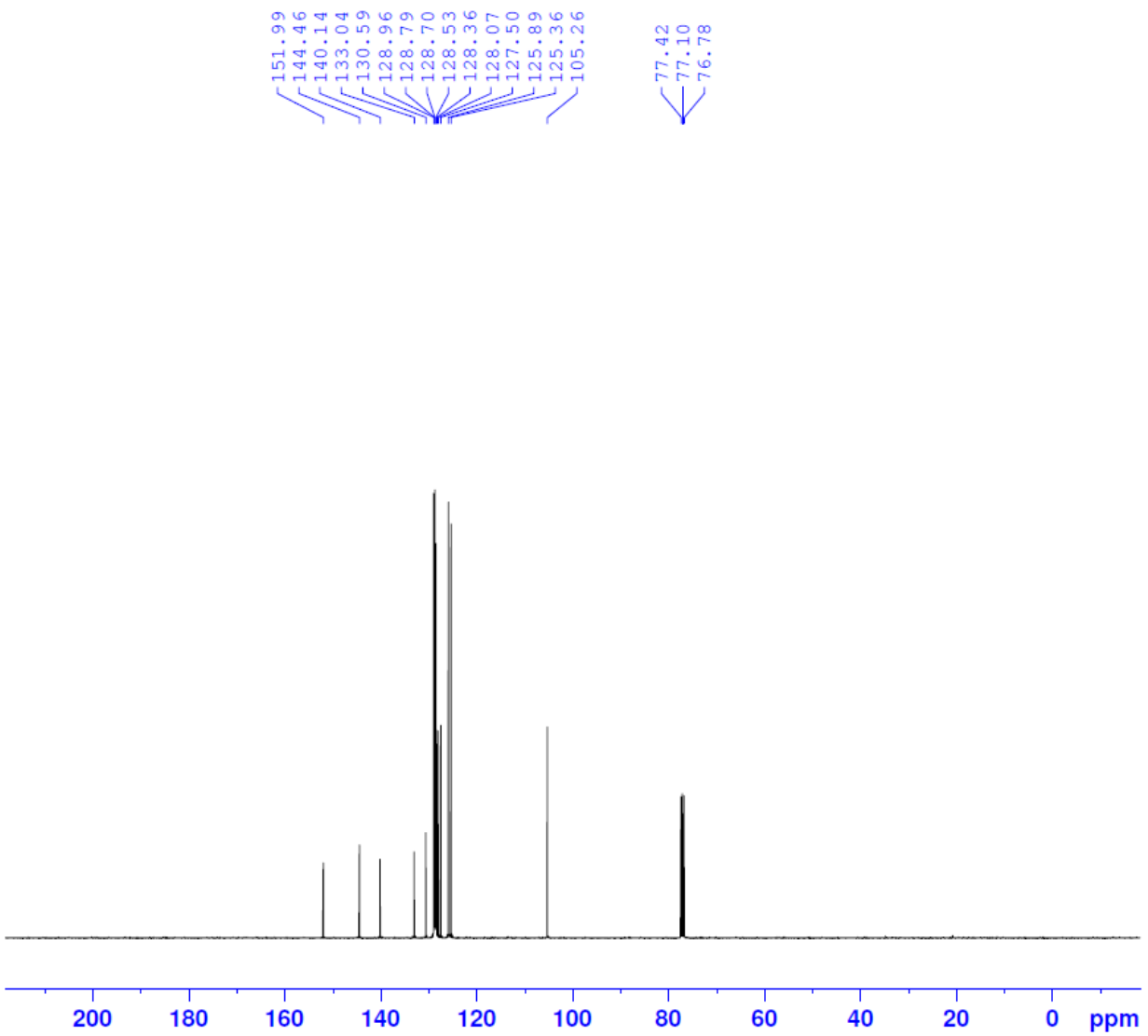

1,3,5-triphenyl-1H-pyrazole (P13): ^{13}C-NMR ($CDCl_3$)

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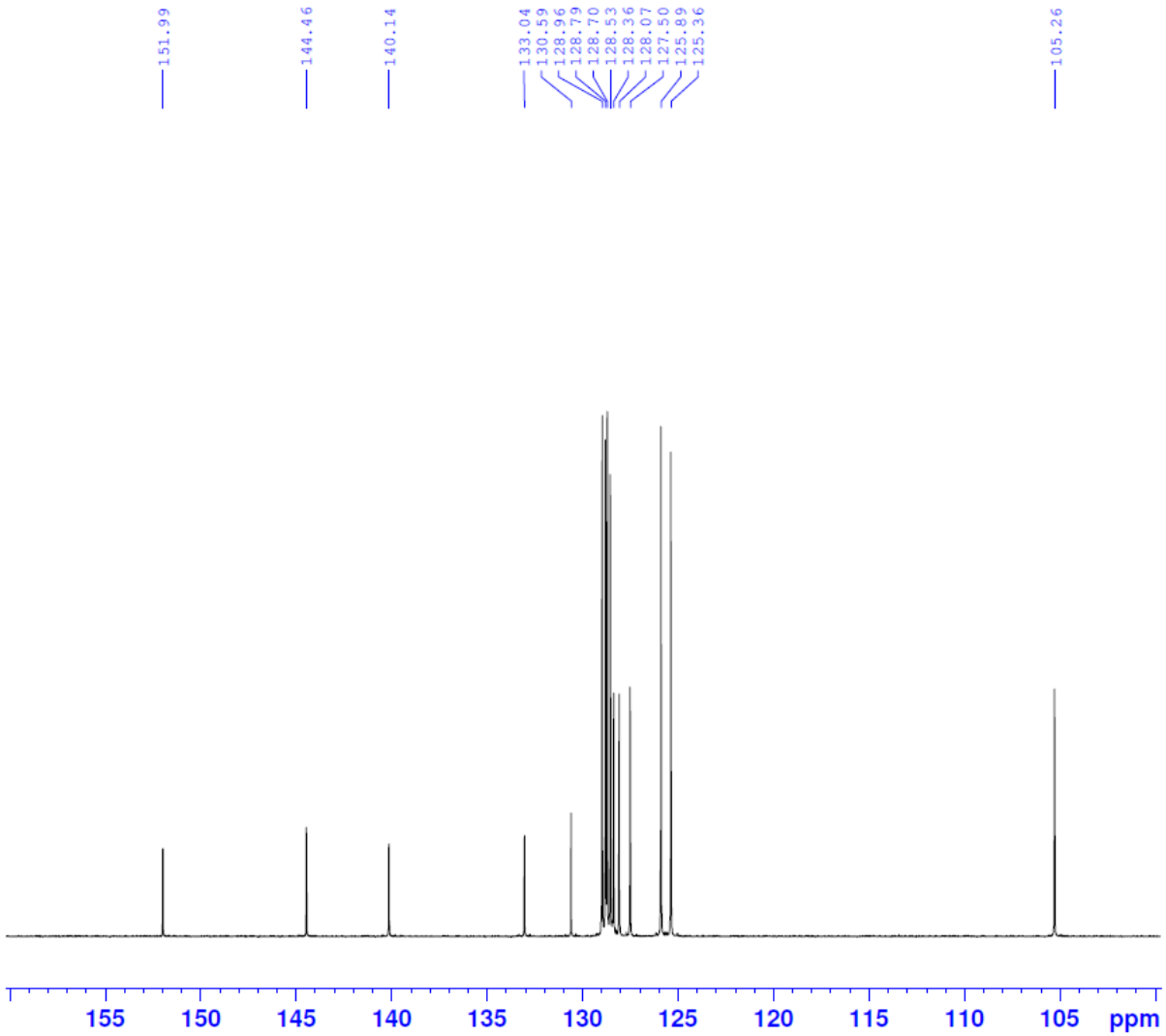
Current Data Parameters
NAME      SM_Mar21_P131Hand13C
EXPNO                   11
PROCNO                   1

F2 - Acquisition Parameters
Date_           20230321
Time                19.42 h
INSTRUM       Avance Neo
PROBHD    Z140678_0053 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           23809.523 Hz
FIDRES         0.726609 Hz
AQ             1.3762560 sec
RG                 12.7
DW                21.000 usec
DE                 6.50 usec
TE                296.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6253446 MHz
NUC1               13C
P0                 3.33 usec
P1                10.00 usec
PLW1        77.80000305 W
SFO2        400.1416006 MHz
NUC2               1H
CPDPRG[2        waltz65
PCPD2             90.00 usec
PLW2        22.64200020 W
PLW12        0.27952999 W
PLW13        0.14060000 W

F2 - Processing parameters
SI                32768
SF          100.6152830 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

1,3,5-triphenyl-1H-pyrazole (P13): ^{13}C-NMR ($CDCl_3$) _ Expanded Aromatic Region



BRUKER

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Current Data Parameters
NAME      SM_Mar21_P131Hand13C
EXPNO                   11
PROCNO                   1

F2 - Acquisition Parameters
Date_           20230321
Time              19.42 h
INSTRUM       Avance Neo
PROBHD    Z140678_0053 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           23809.523 Hz
FIDRES         0.726609 Hz
AQ             1.3762560 sec
RG                12.7
DW               21.000 usec
DE                6.50 usec
TE               296.9 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1
SFO1         100.6253446 MHz
NUC1                13C
P0                3.33 usec
P1               10.00 usec
PLW1         77.80000305 W
SFO2         400.1416006 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             90.00 usec
PLW2         22.64200020 W
PLW12         0.27952999 W
PLW13         0.14060000 W

F2 - Processing parameters
SI                32768
SF           100.6152830 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```
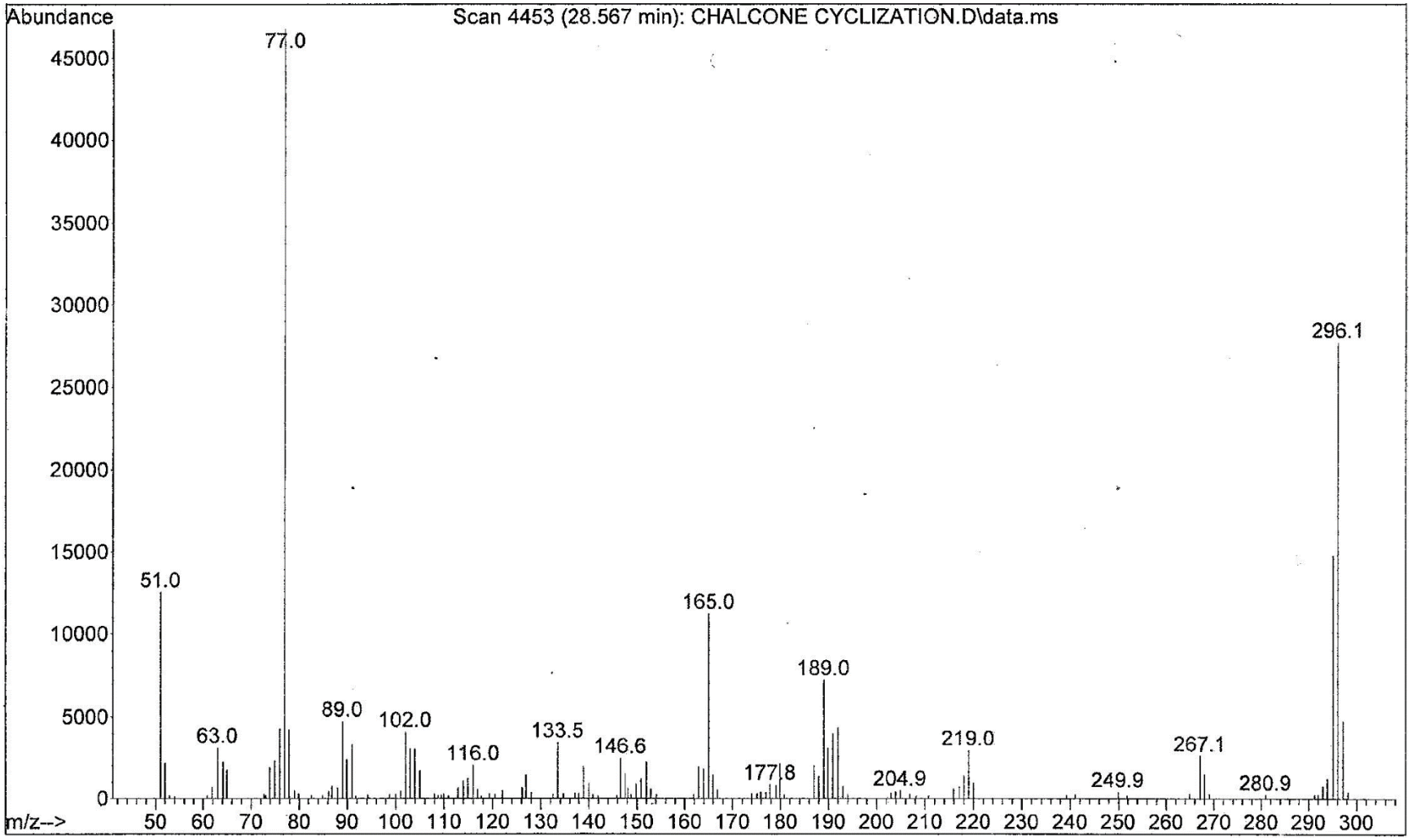
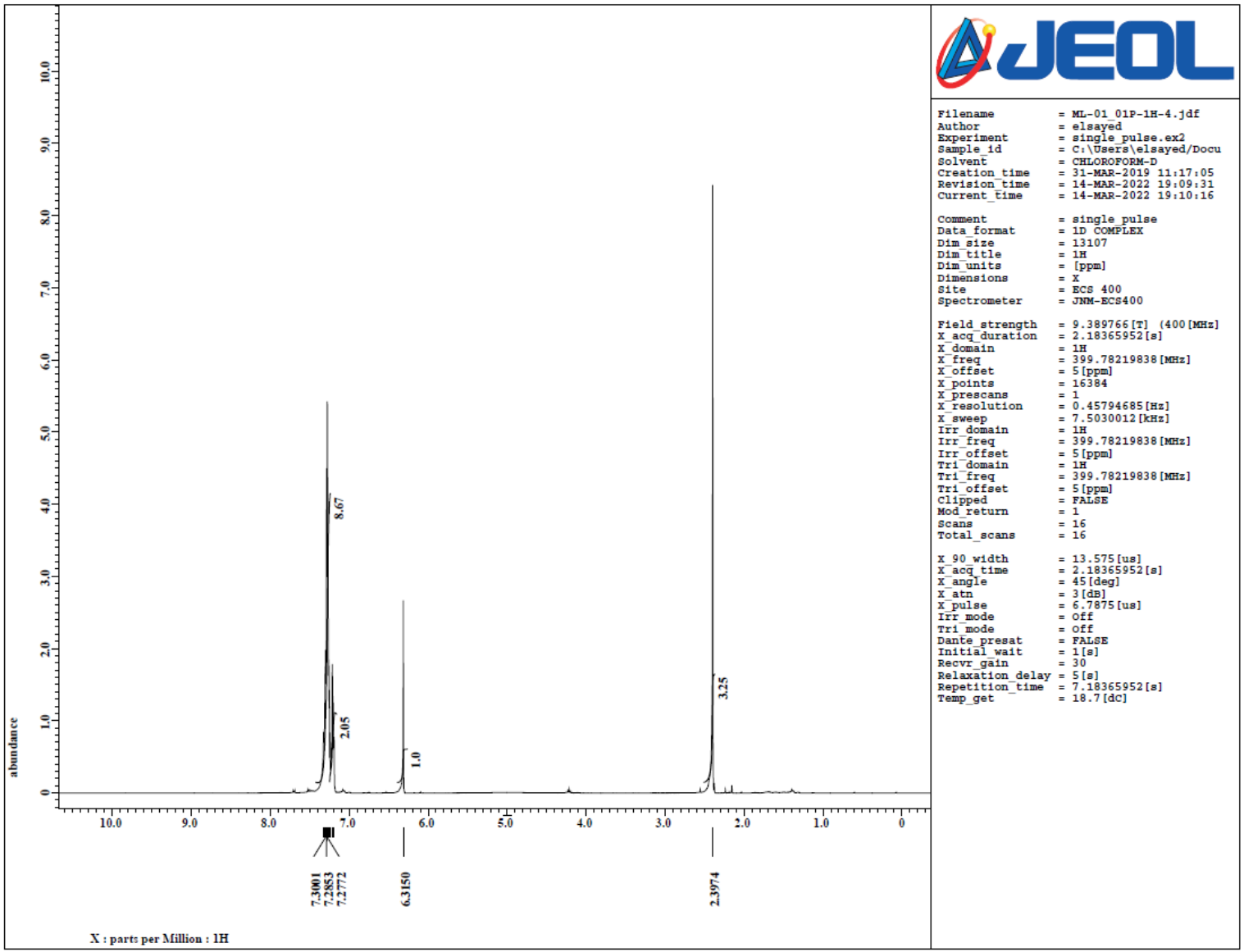
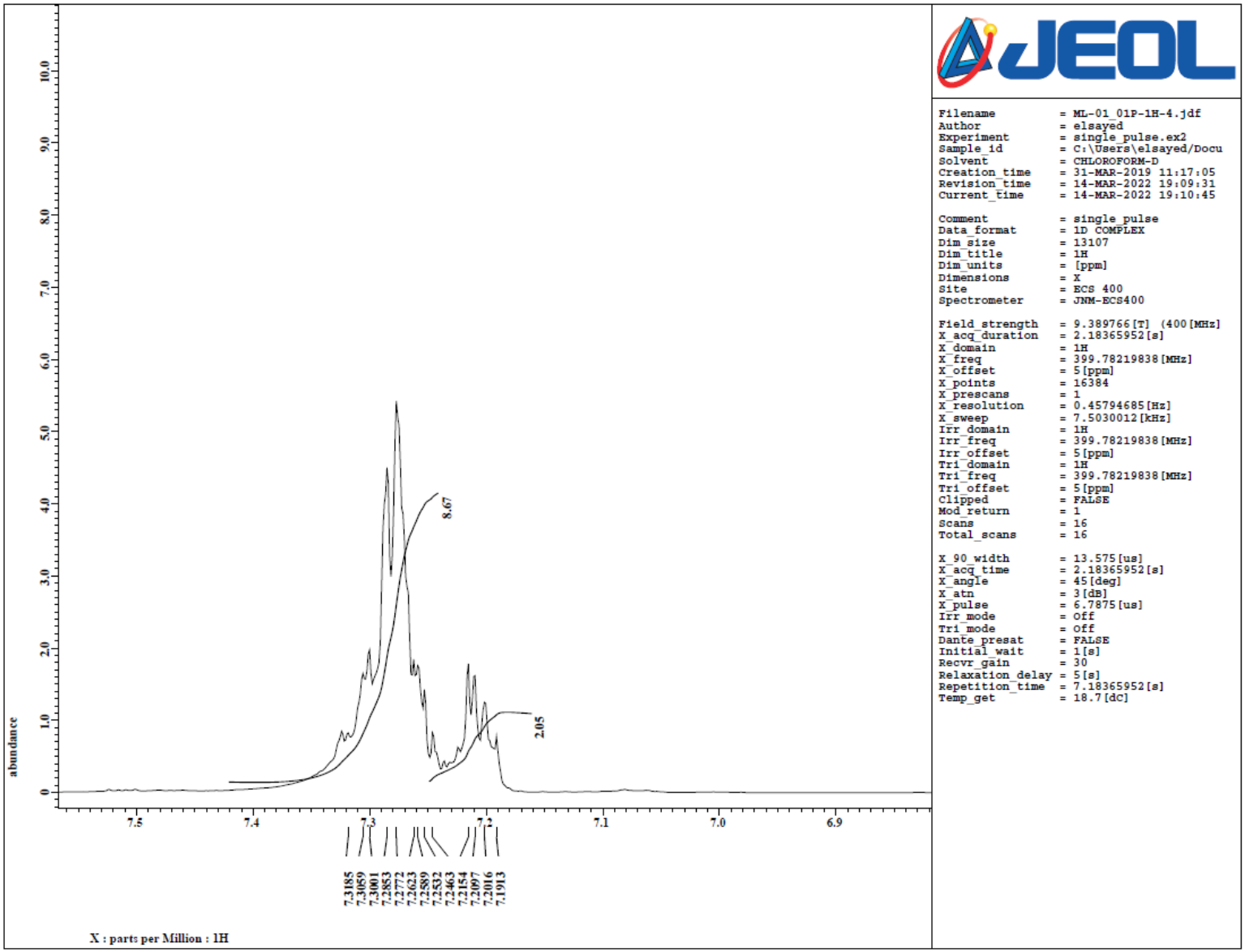

1,3,5-triphenyl-1H-pyrazole (P13): GC-MS



3-methyl-1,5-diphenyl-1H-pyrazole (P14): ^{1}H-NMR ($CDCl_3$)



3-methyl-1,5-diphenyl-1H-pyrazole (P14): ^{1}H-NMR ($CDCl_3$) _ Expanded Aromatic Region

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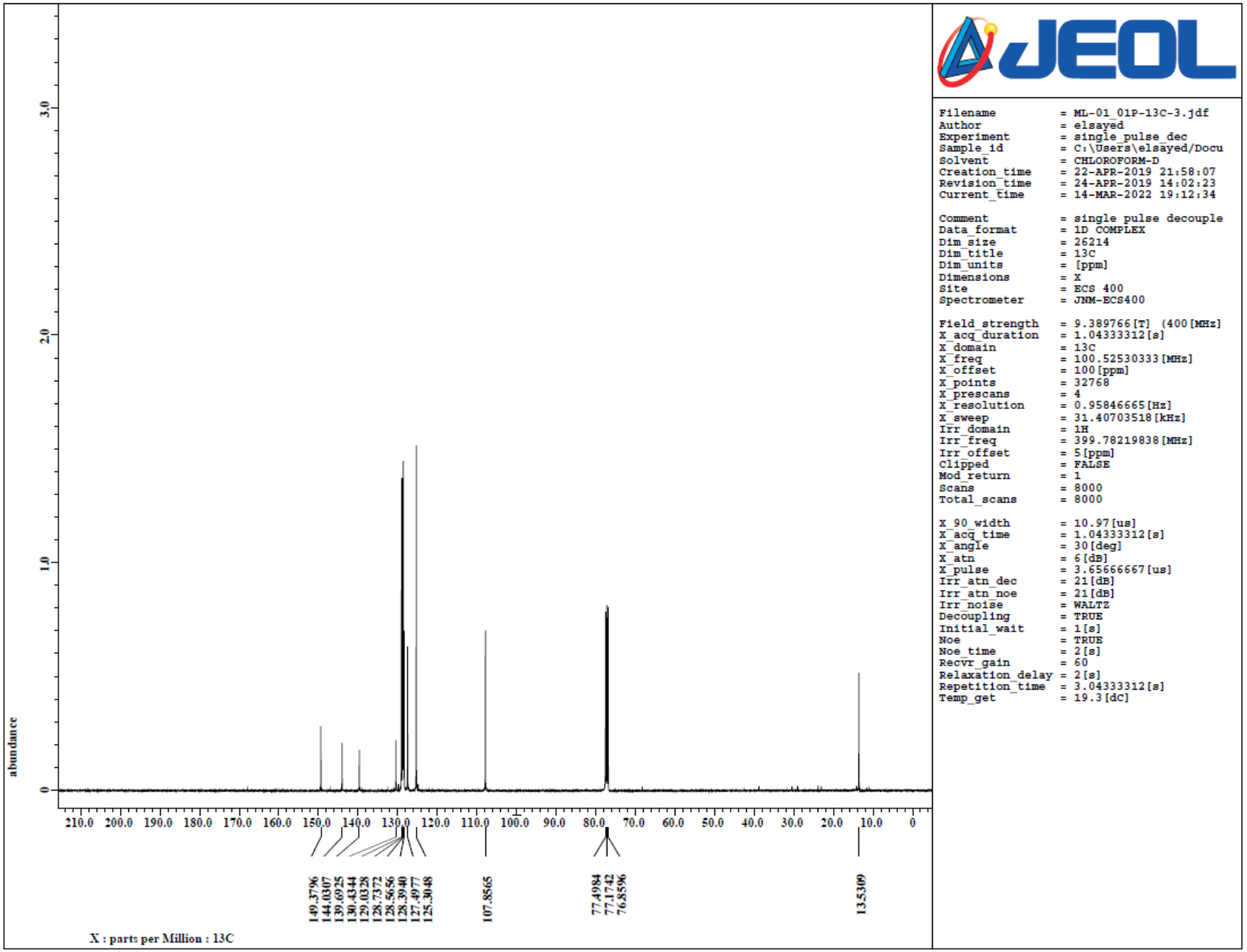
Filename          = ML-01_01P-1H-4.jdf
Author            = elsayed
Experiment        = single_pulse.ex2
Sample_id         = C:\Users\elsayed/Docu
Solvent           = CHLOROFORM-D
Creation_time     = 31-MAR-2019 11:17:05
Revision_time     = 14-MAR-2022 19:09:31
Current_time      = 14-MAR-2022 19:10:45

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

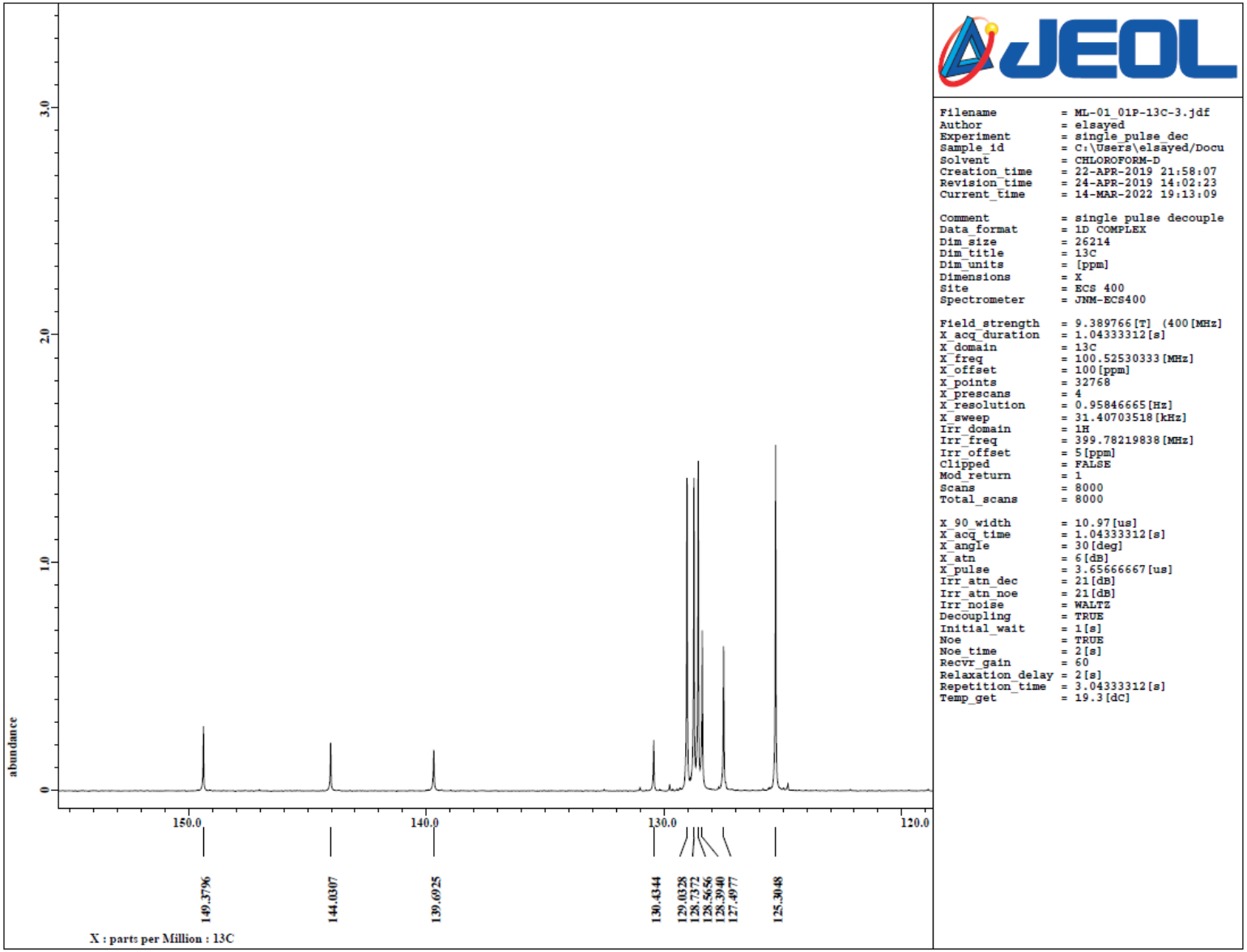
Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 13.575[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.7875[us]
Irr_mode          = off
Tri_mode          = off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 30
Relaxation_delay  = 5[s]
Repetition_time   = 7.18365952[s]
Temp_get          = 18.7[dC]
```

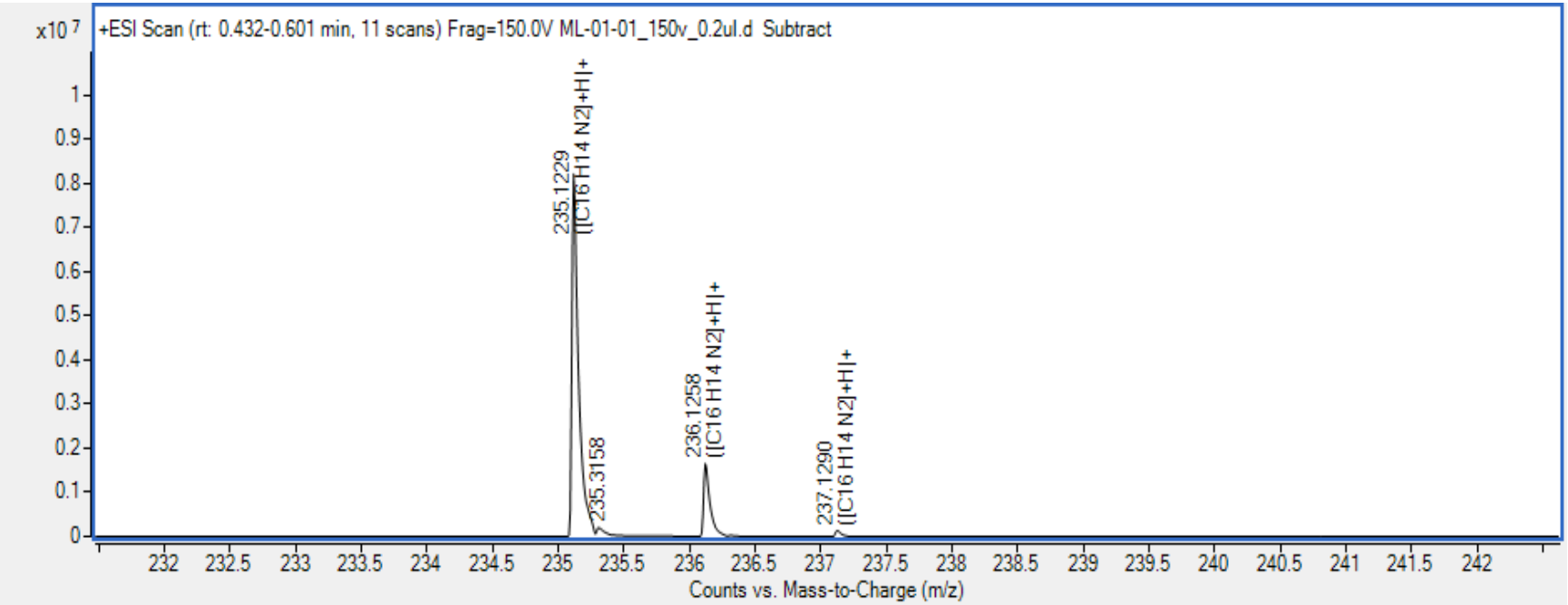
3-methyl-1,5-diphenyl-1H-pyrazole (P14): ^{13}C-NMR ($CDCl_3$)



3-methyl-1,5-diphenyl-1H-pyrazole (P14): ^{13}C-NMR ($CDCl_3$) _ Expanded Aromatic Region



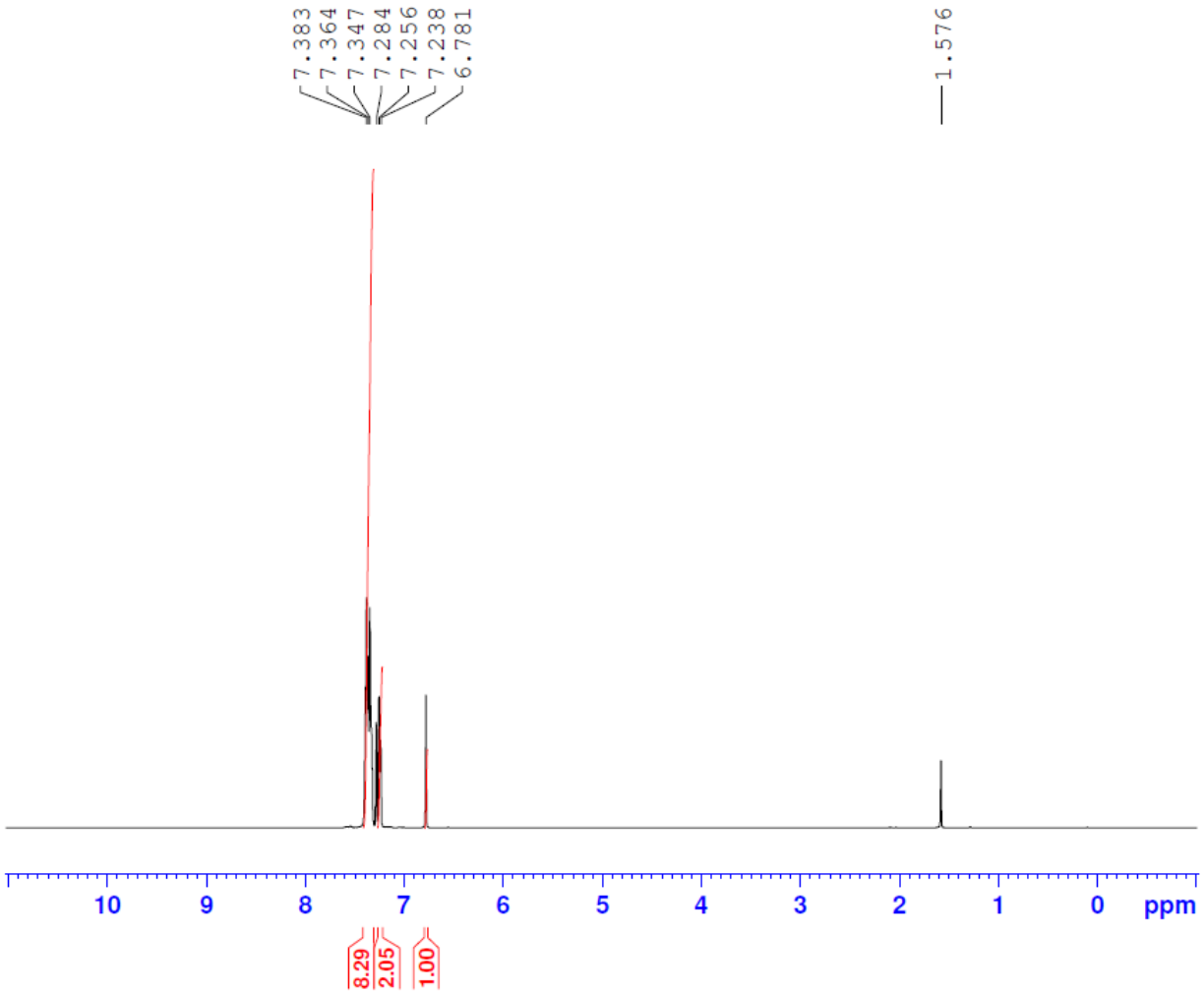
3-methyl-1,5-diphenyl-1H-pyrazole (P14): ESI-MS



Formula,	Species,	m/z,	Score,	Diff (abs. ppm),	Mass
C16 H14 N2,	(M+H)+,	235.1229,	98.87,	0.54,	234.1156

1,5-diphenyl-3-(trifluoromethyl)-1H-pyrazole (P15): ^{1}H-NMR ($CDCl_3$)

1H NMR of 84



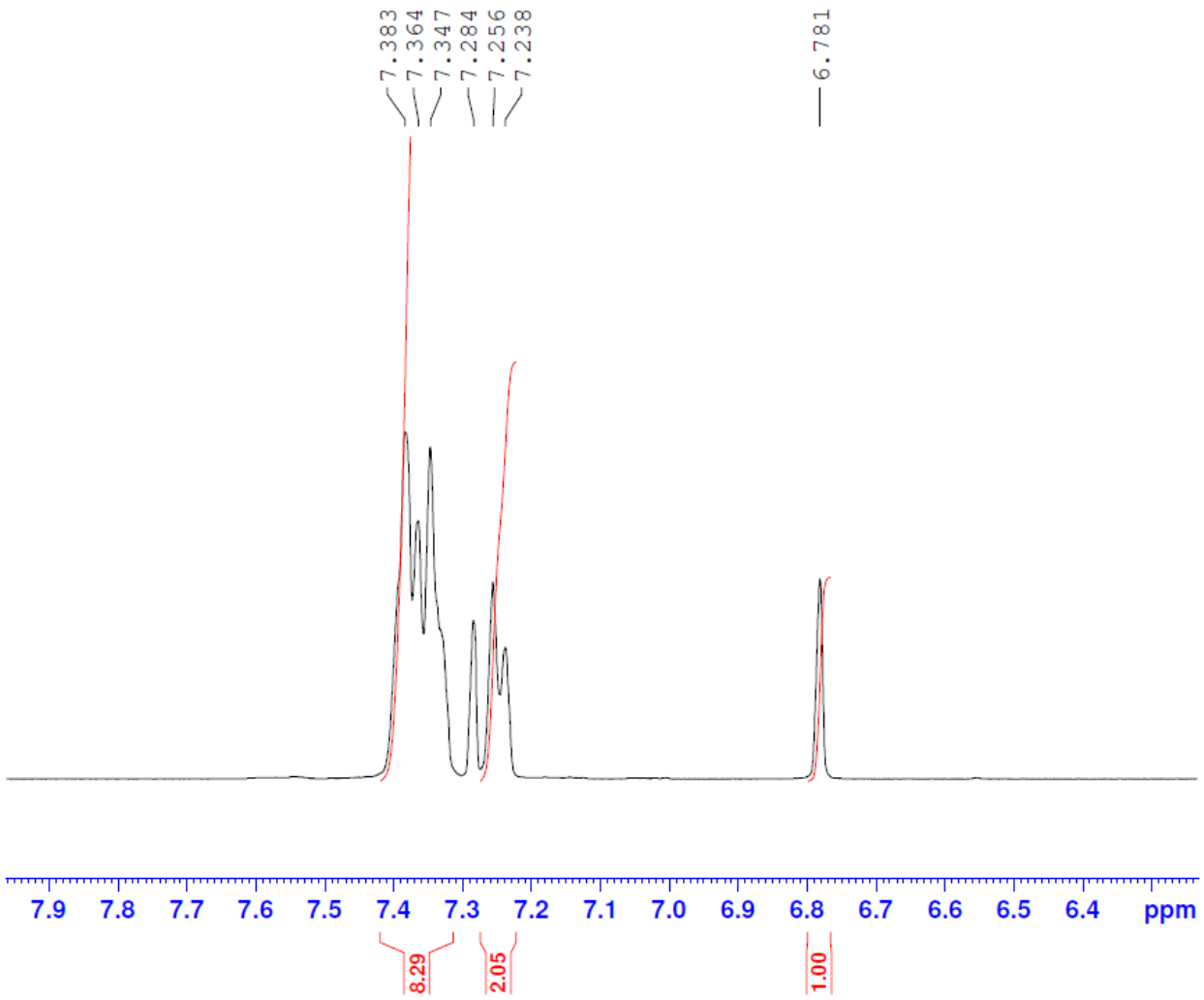
Current Data Parameters
NAME SM-84_1H
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230317
Time 16.39 h
INSTRUM Avance Neo
PROBHD Z140678_0053 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8196.722 Hz
FIDRES 0.250144 Hz
AQ 3.9976959 sec
RG 101
DW 61.000 usec
DE 13.54 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1
SFO1 400.1424709 MHz
NUC1 1H
P0 3.33 usec
P1 10.00 usec
PLW1 22.64200020 W

F2 - Processing parameters
SI 65536
SF 400.1400000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1,5-diphenyl-3-(trifluoromethyl)-1H-pyrazole (P15): ^{1}H-NMR ($CDCl_3$) _ Expanded Aromatic Region

1H NMR of 84



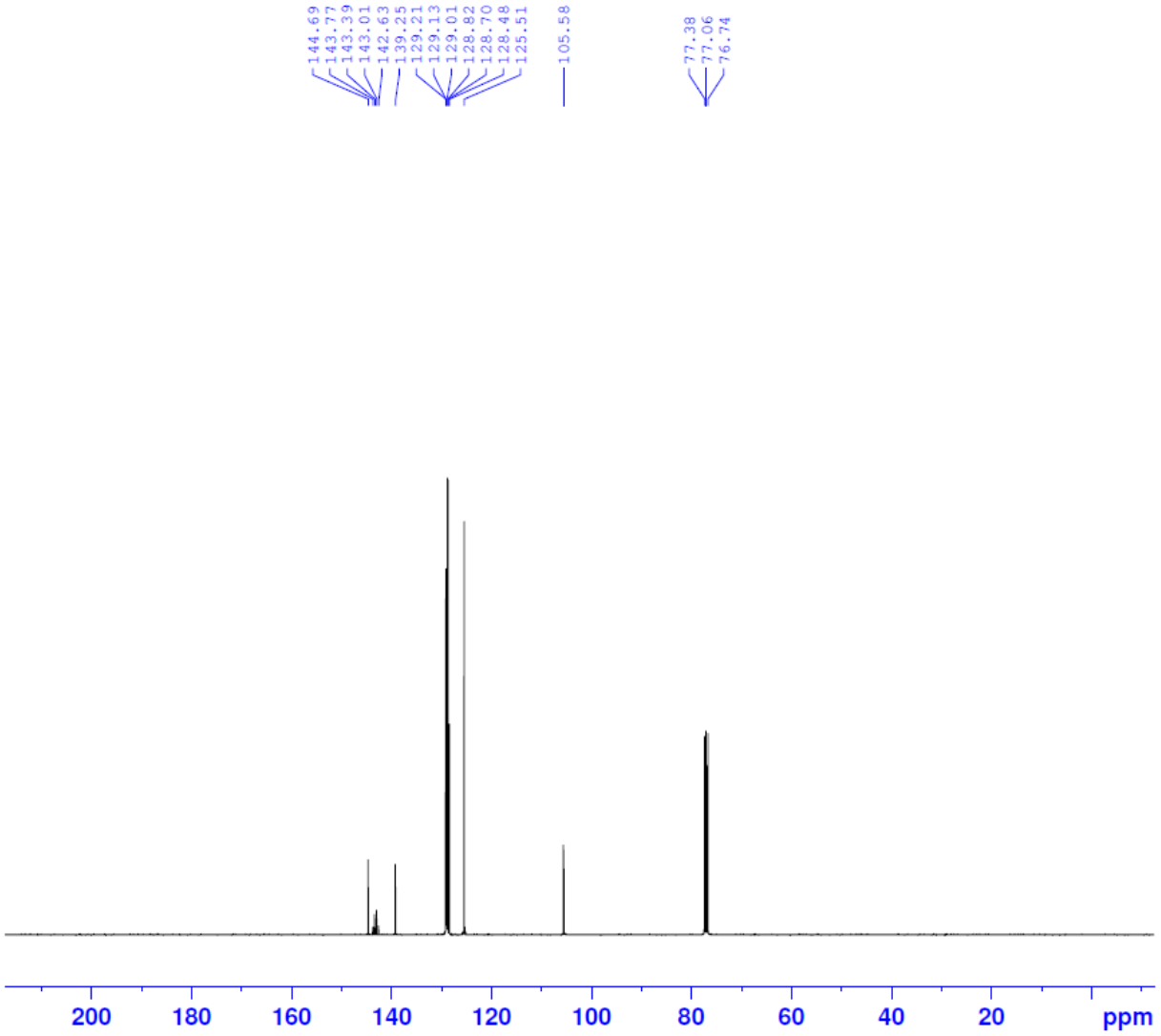
BRUKER

```
Current Data Parameters
NAME                  SM-84_1H
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20230317
Time                     16.39 h
INSTRUM             Avance Neo
PROBHD          Z140678_0053 (
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                   8196.722 Hz
FIDRES                0.250144 Hz
AQ                    3.9976959 sec
RG                        101
DW                       61.000 usec
DE                       13.54 usec
TE                       296.3 K
D1                    1.00000000 sec
TD0                         1
SFO1                400.1424709 MHz
NUC1                        1H
P0                       3.33 usec
P1                      10.00 usec
PLW1                22.64200020 W

F2 - Processing parameters
SI                      65536
SF                  400.1400000 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```

1,5-diphenyl-3-(trifluoromethyl)-1H-pyrazole (P15): ^{13}C-NMR ($CDCl_3$)

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Current Data Parameters
NAME      SM-84_1Hand13C
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230318
Time              13.27 h
INSTRUM       Avance Neo
PROBHD    Z140678_0053 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           23809.523 Hz
FIDRES         0.726609 Hz
AQ            1.3762560 sec
RG                   16
DW                21.000 usec
DE                 6.50 usec
TE                296.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6253446 MHz
NUC1               13C
P0                3.33 usec
P1               10.00 usec
PLW1        77.80000305 W
SFO2        400.1416006 MHz
NUC2               1H
CPDPRG[2        waltz65
PCPD2            90.00 usec
PLW2        22.64200020 W
PLW12        0.27952999 W
PLW13        0.14060000 W

F2 - Processing parameters
SI                32768
SF          100.6152830 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```
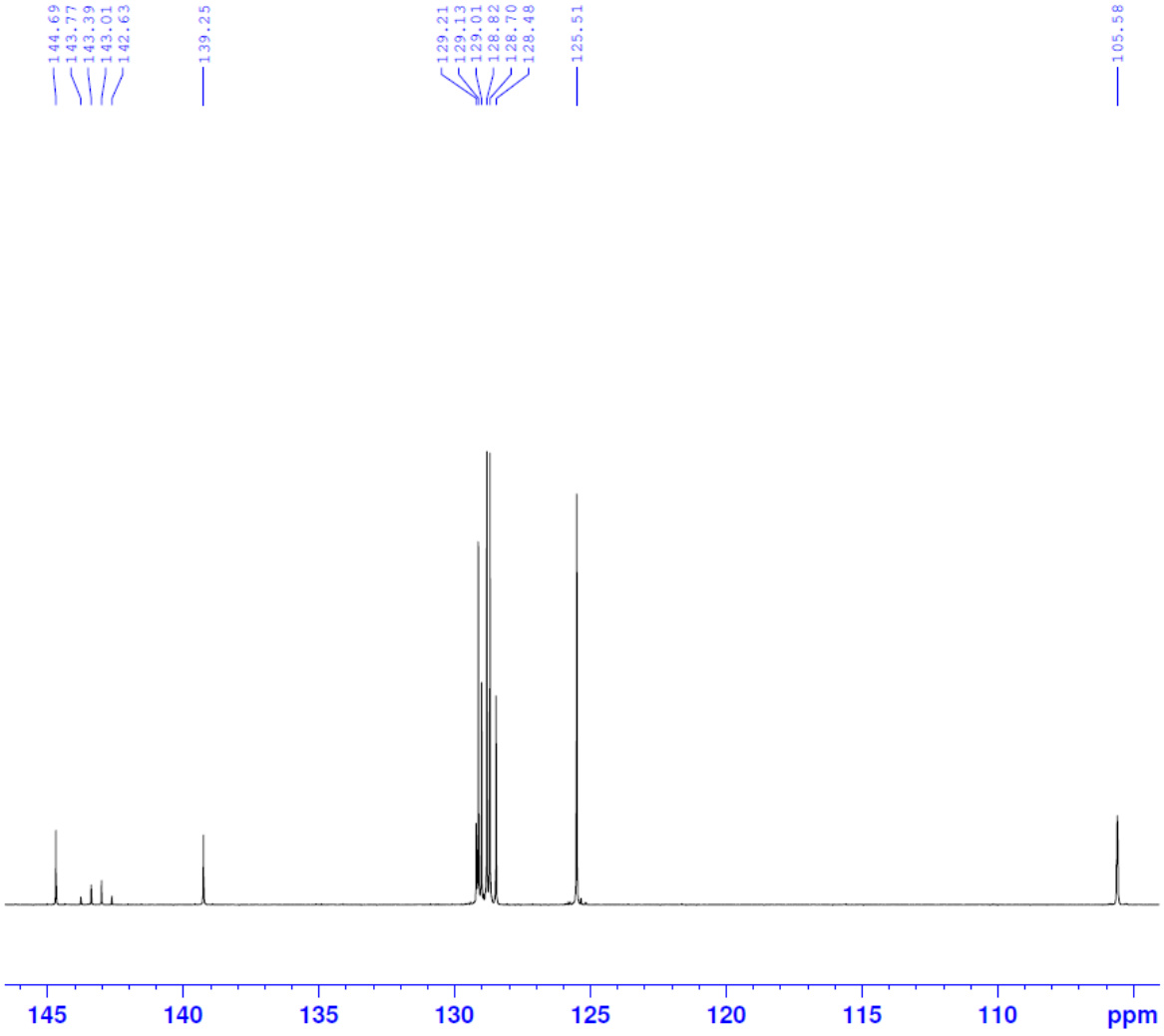

1,5-diphenyl-3-(trifluoromethyl)-1H-pyrazole (P15): ^{13}C-NMR ($CDCl_3$) _ Expanded Aromatic Region



BRUKER

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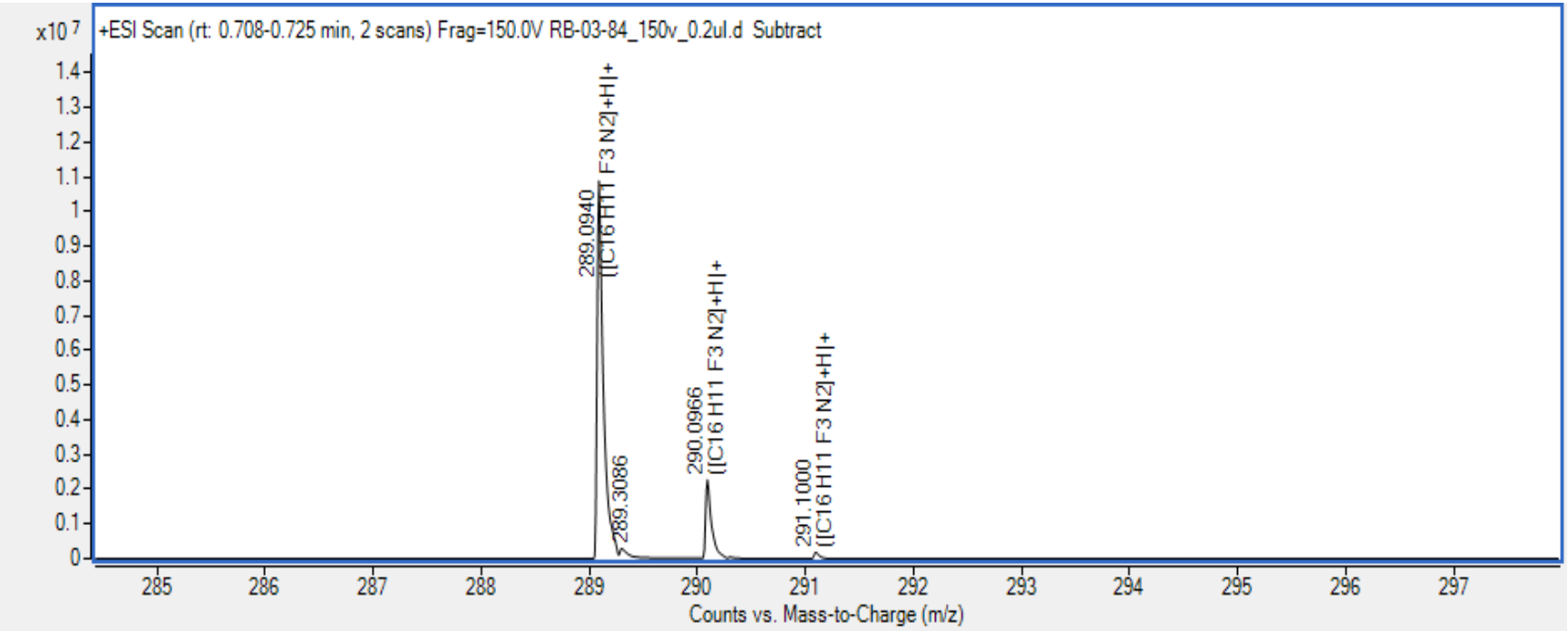
Current Data Parameters
NAME      SM-84_1Hand13C
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230318
Time              13.27 h
INSTRUM       Avance Neo
PROBHD   Z140678_0053 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           23809.523 Hz
FIDRES         0.726609 Hz
AQ            1.3762560 sec
RG                   16
DW                21.000 usec
DE                 6.50 usec
TE                296.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6253446 MHz
NUC1                13C
P0                 3.33 usec
P1                10.00 usec
PLW1        77.80000305 W
SFO2        400.1416006 MHz
NUC2                1H
CPDPRG[2        waltz65
PCPD2             90.00 usec
PLW2        22.64200020 W
PLW12        0.27952999 W
PLW13        0.14060000 W

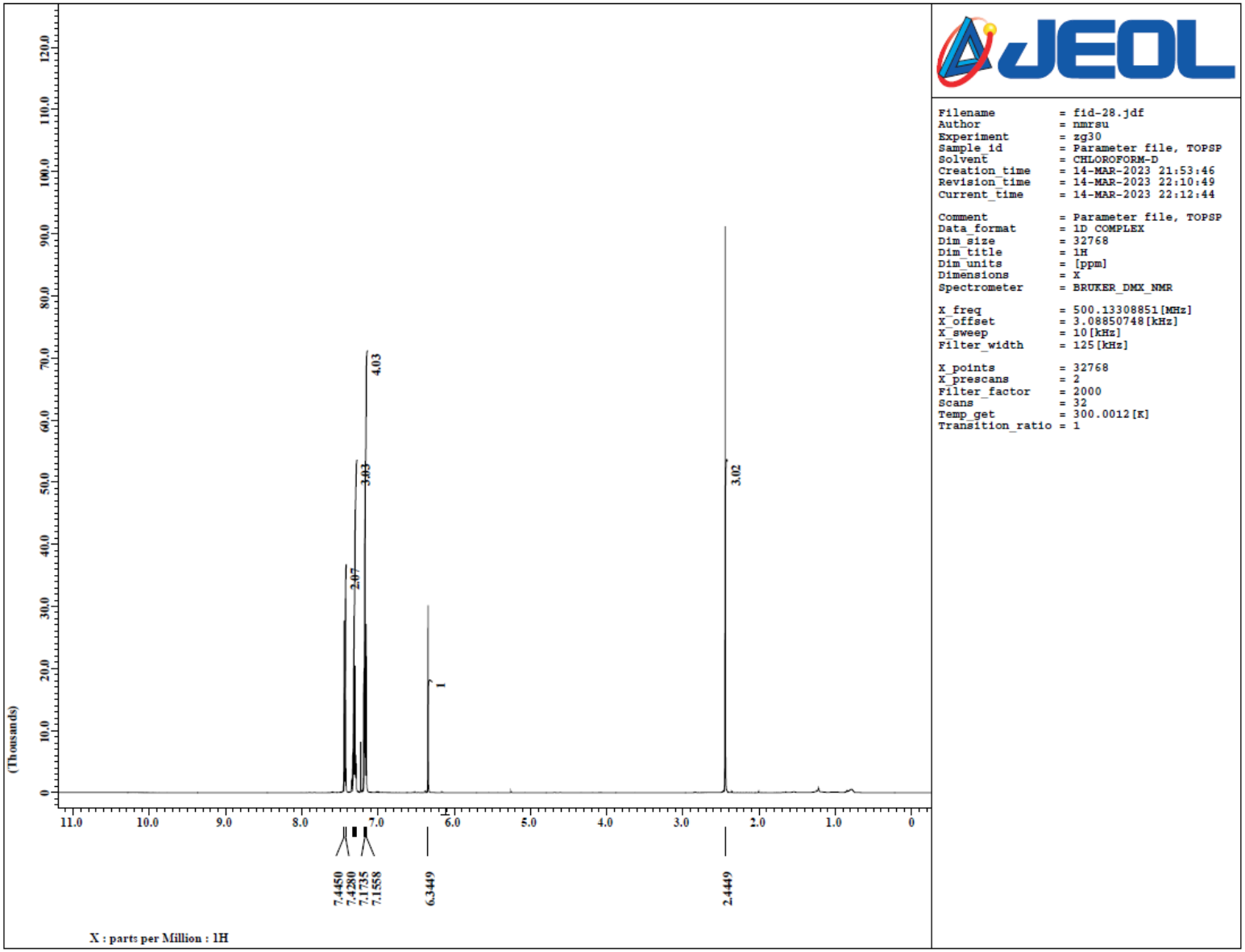
F2 - Processing parameters
SI                32768
SF          100.6152830 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

1,5-diphenyl-3-(trifluoromethyl)-1H-pyrazole (P15): ESI-MS

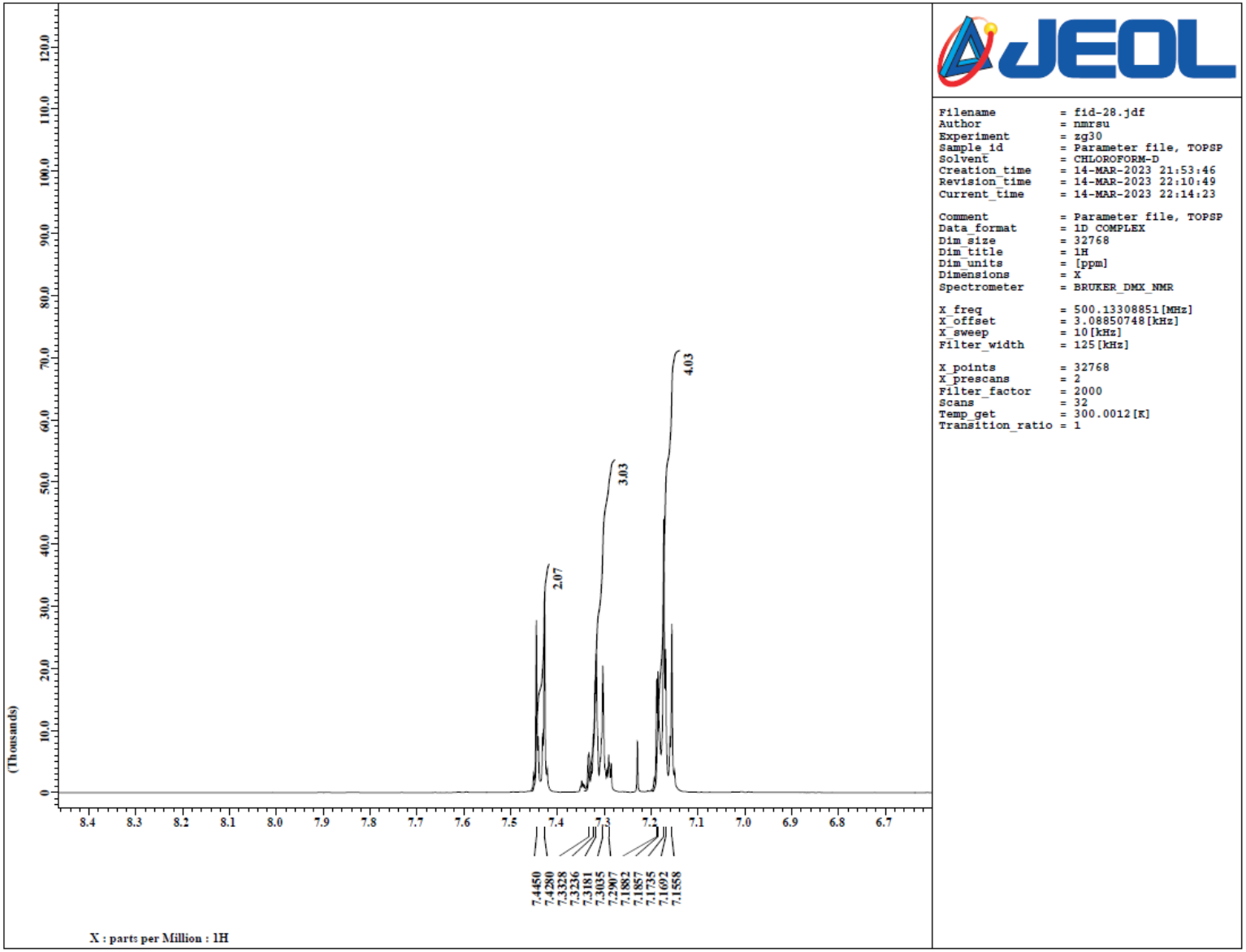


Formula,	Species,	m/z,	Score,	Diff (abs. ppm),	Mass
C16 H11 F3 N2,	(M+H)+,	289.0940,	95.7,	2.66,	288.0867

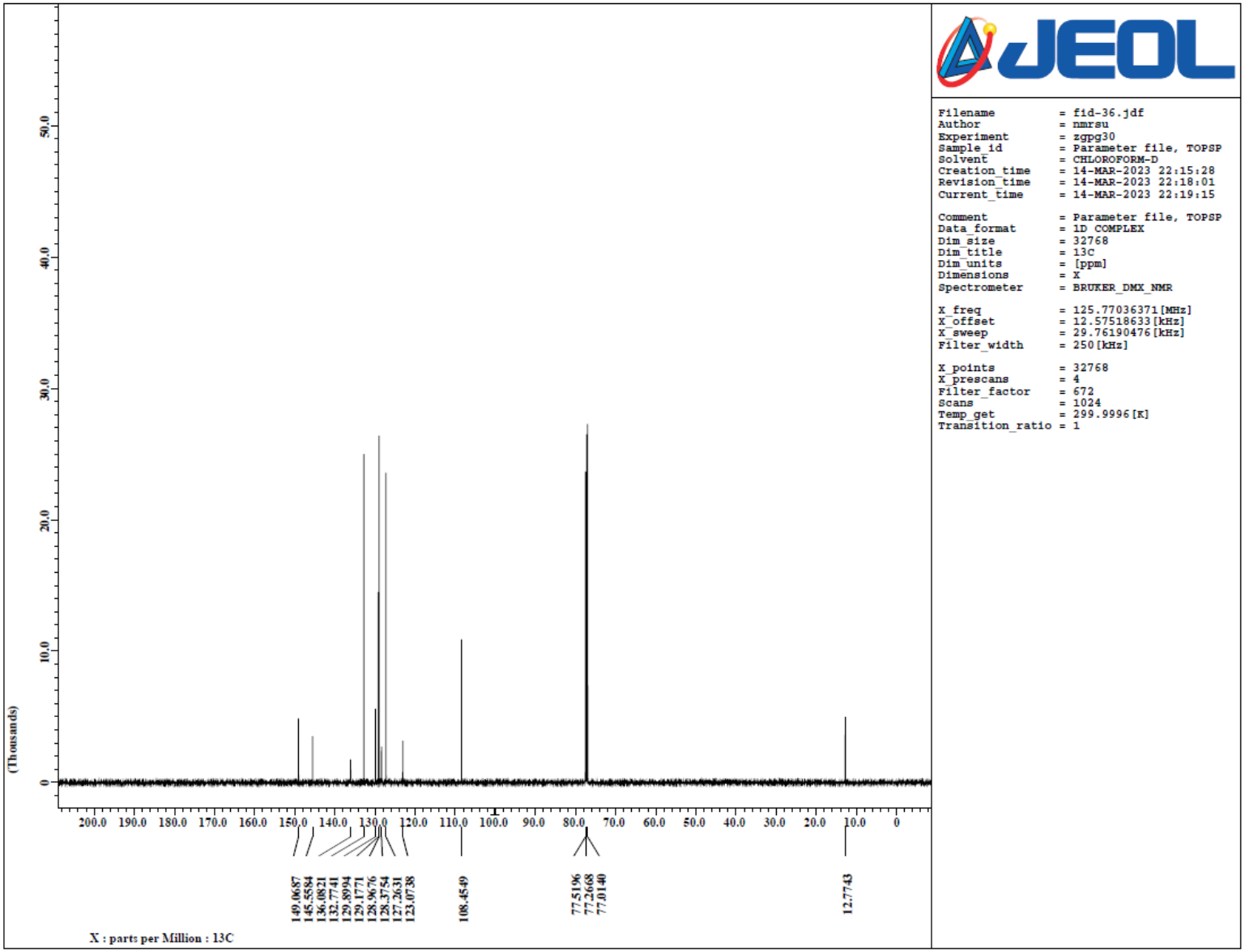
1-(4-bromophenyl)-3-methyl-5-phenyl-1H-pyrazole (P16): ^{1}H-NMR ($CDCl_3$)



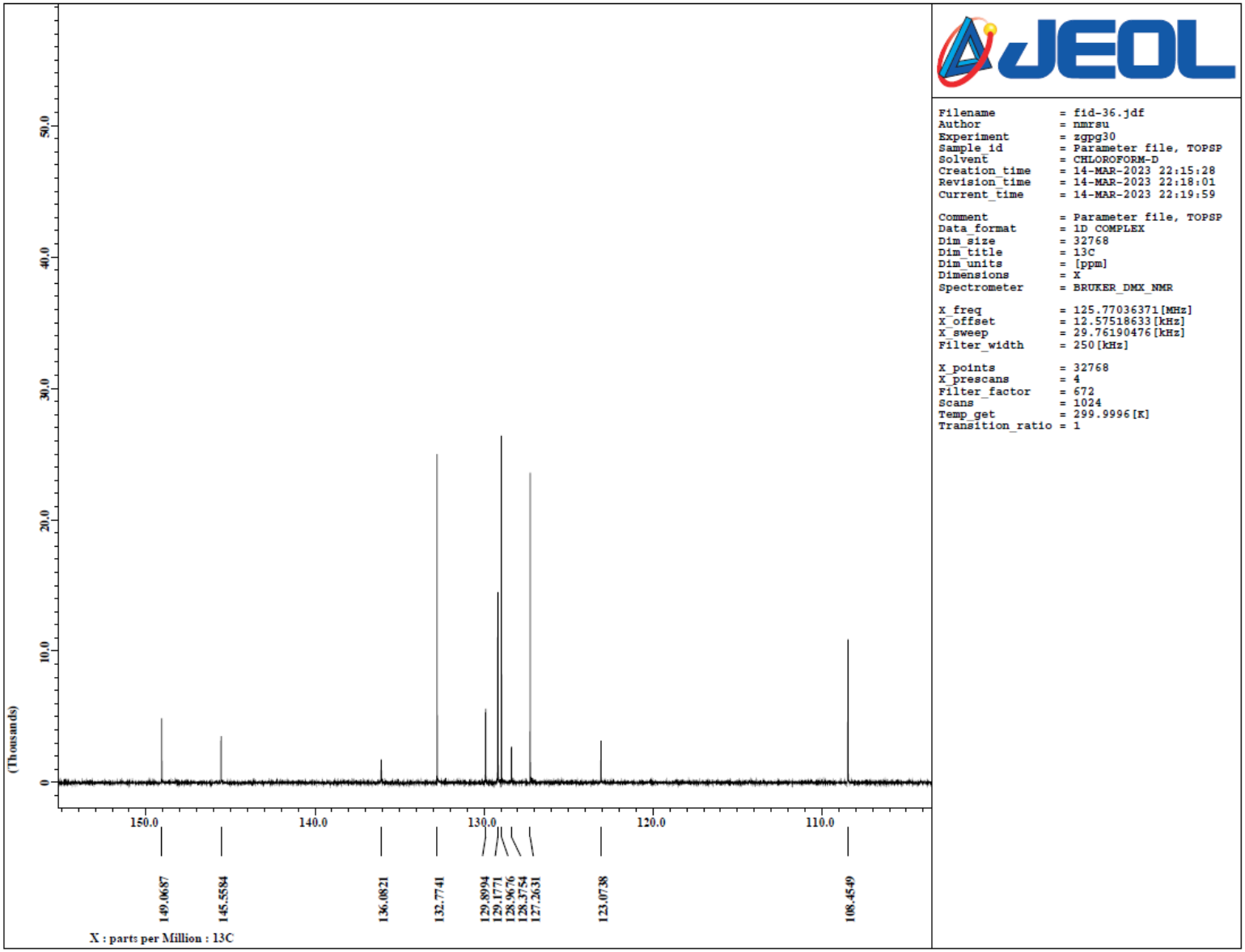
1-(4-bromophenyl)-3-methyl-5-phenyl-1H-pyrazole (P16): ^{1}H-NMR ($CDCl_3$) _ Expanded Aromatic Region



1-(4-bromophenyl)-3-methyl-5-phenyl-1H-pyrazole (P16): ^{13}C-NMR ($CDCl_3$)



1-(4-bromophenyl)-3-methyl-5-phenyl-1H-pyrazole (P16): ^{13}C-NMR ($CDCl_3$) _ Expanded Aromatic Region

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Filename          = fid-36.jdf
Author            = nmrsu
Experiment        = zgpg30
Sample_id         = Parameter file, TOPSP
Solvent           = CHLOROFORM-D
Creation_time     = 14-MAR-2023 22:15:28
Revision_time     = 14-MAR-2023 22:18:01
Current_time      = 14-MAR-2023 22:19:59

Comment           = Parameter file, TOPSP
Data_format       = 1D COMPLEX
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Spectrometer      = BRUKER_DMX_NMR

X_freq            = 125.77036371[MHz]
X_offset          = 12.57518633[kHz]
X_sweep           = 29.76190476[kHz]
Filter_width      = 250[kHz]

X_points          = 32768
X_prescans        = 4
Filter_factor     = 672
Scans             = 1024
Temp_get          = 299.9996[K]
Transition_ratio = 1
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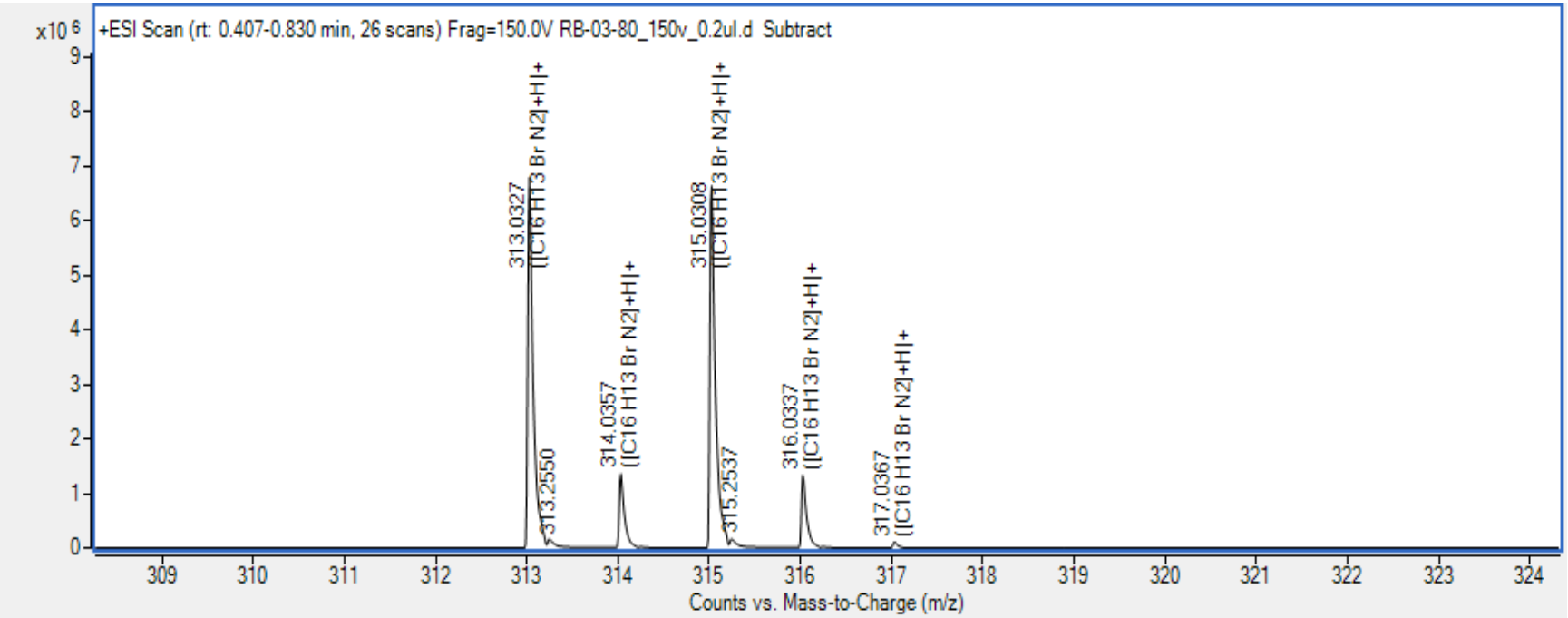
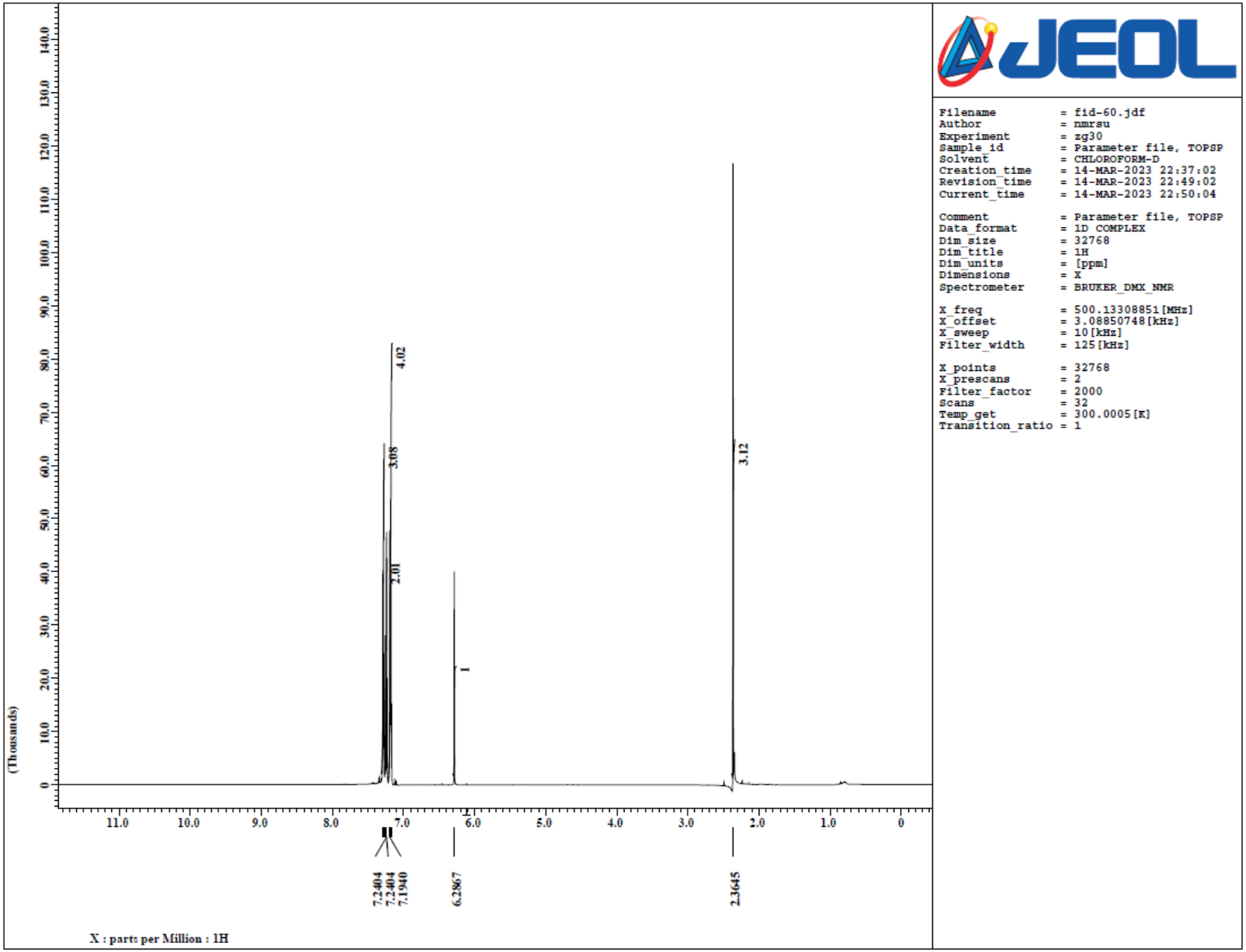
1-(4-bromophenyl)-3-methyl-5-phenyl-1H-pyrazole (P16): ESI-MS

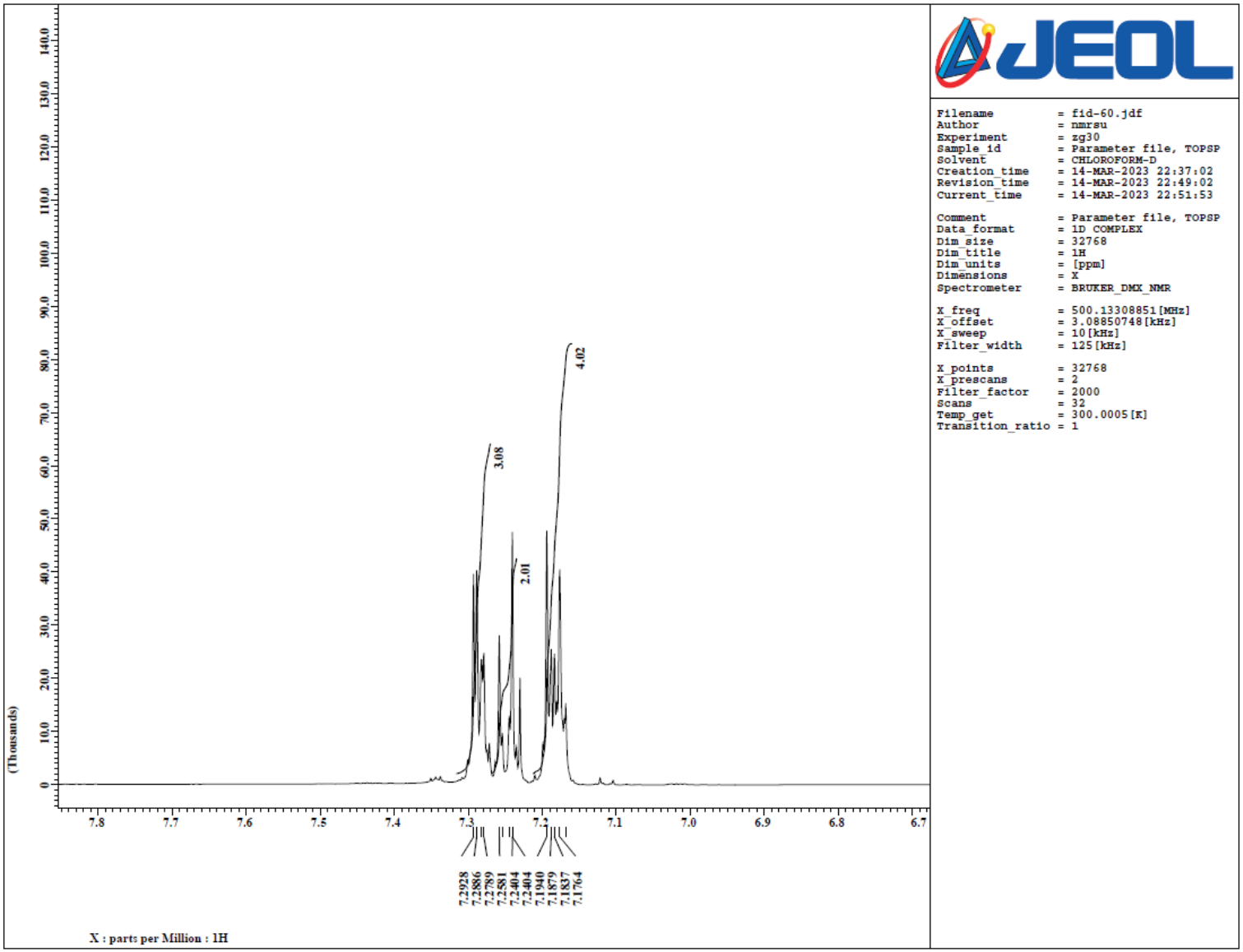


Formula,	Species,	m/z,	Score,	Diff (abs. ppm),	Mass
C16 H13 Br N2,	(M+H)+,	313.0327,	96.99,	2.58,	312.0254

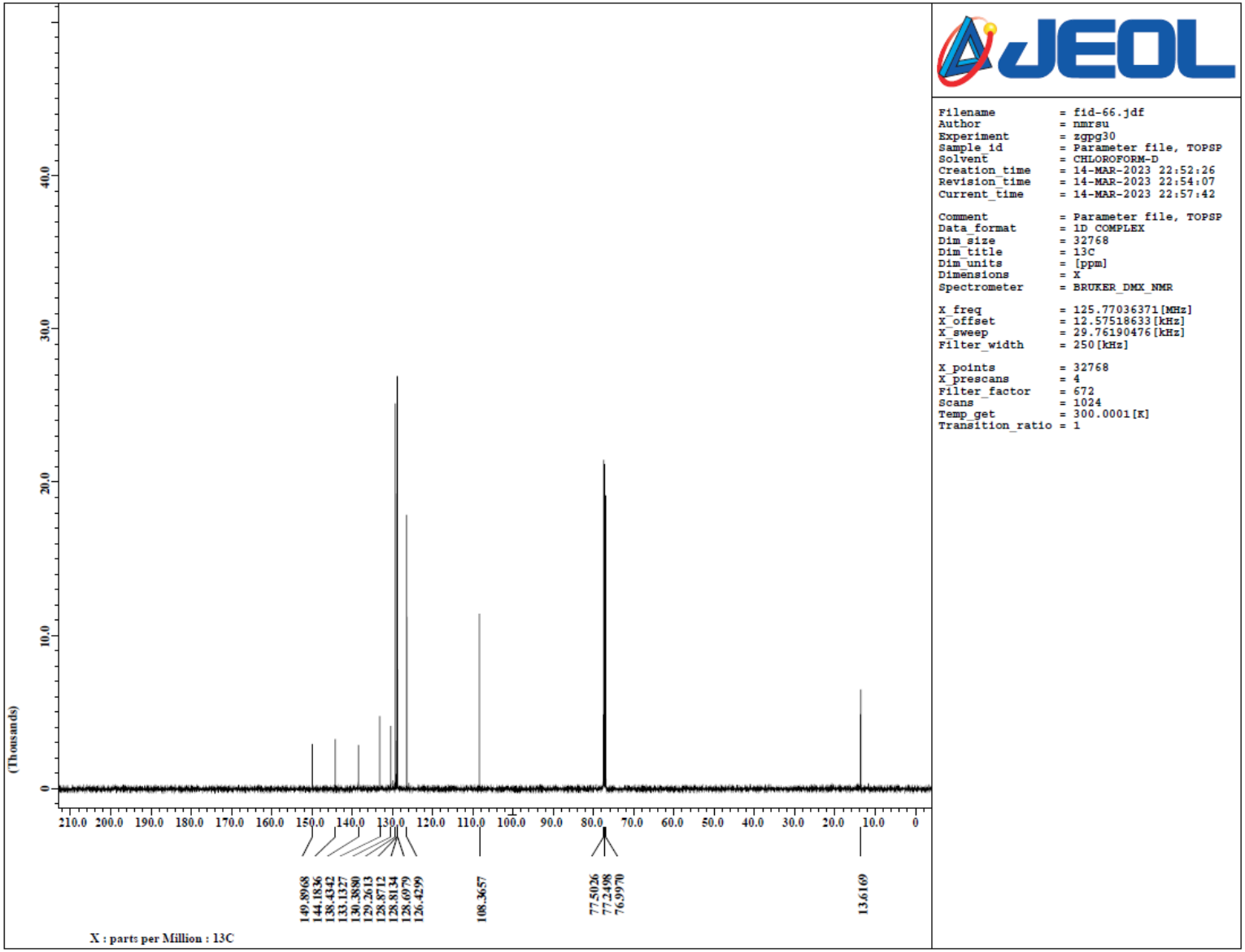
1-(4-chlorophenyl)-3-methyl-5-phenyl-1H-pyrazole (P17): ^{1}H-NMR ($CDCl_3$)



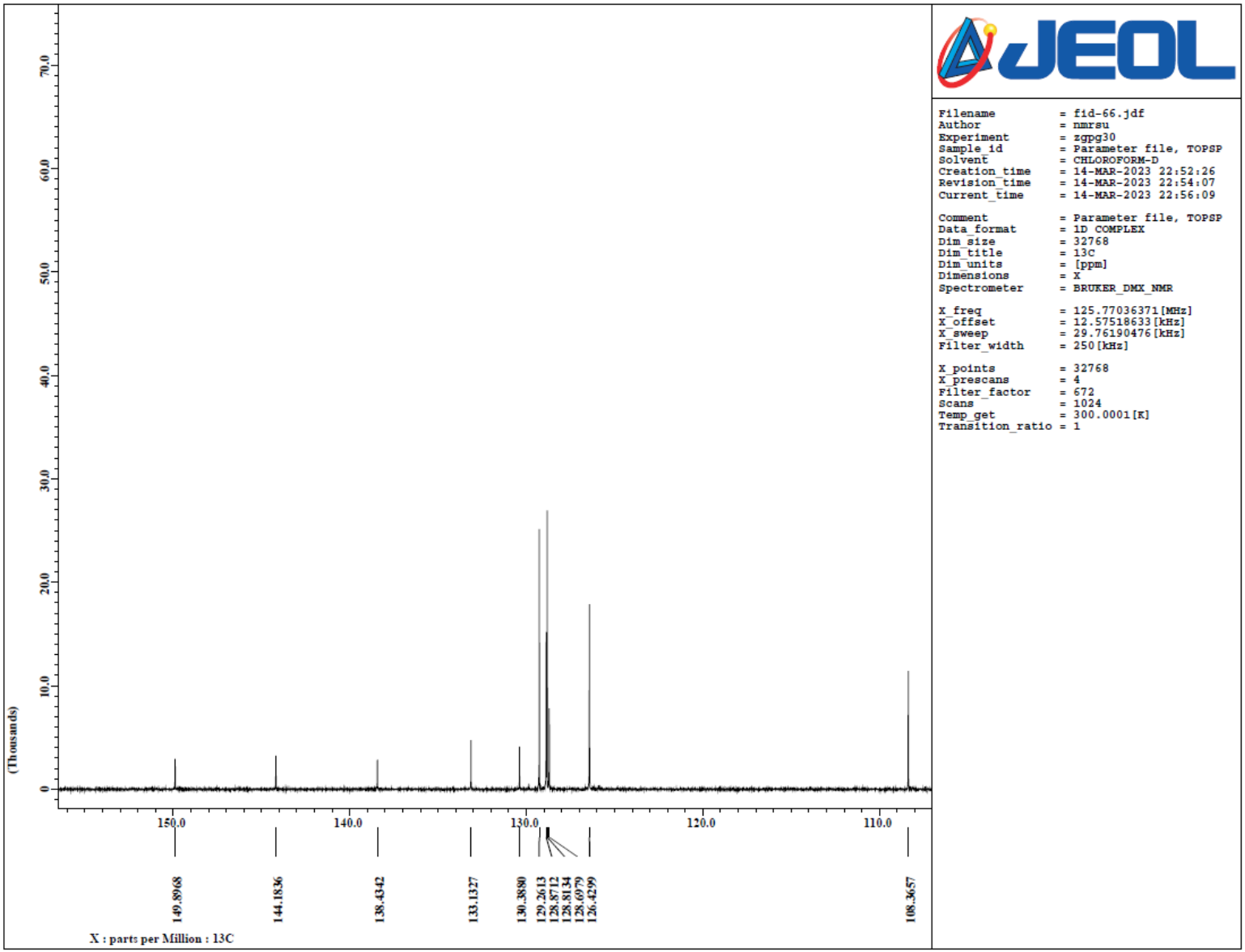
1-(4-chlorophenyl)-3-methyl-5-phenyl-1H-pyrazole (P17): ^{1}H-NMR ($CDCl_3$) _ Expanded Aromatic Region



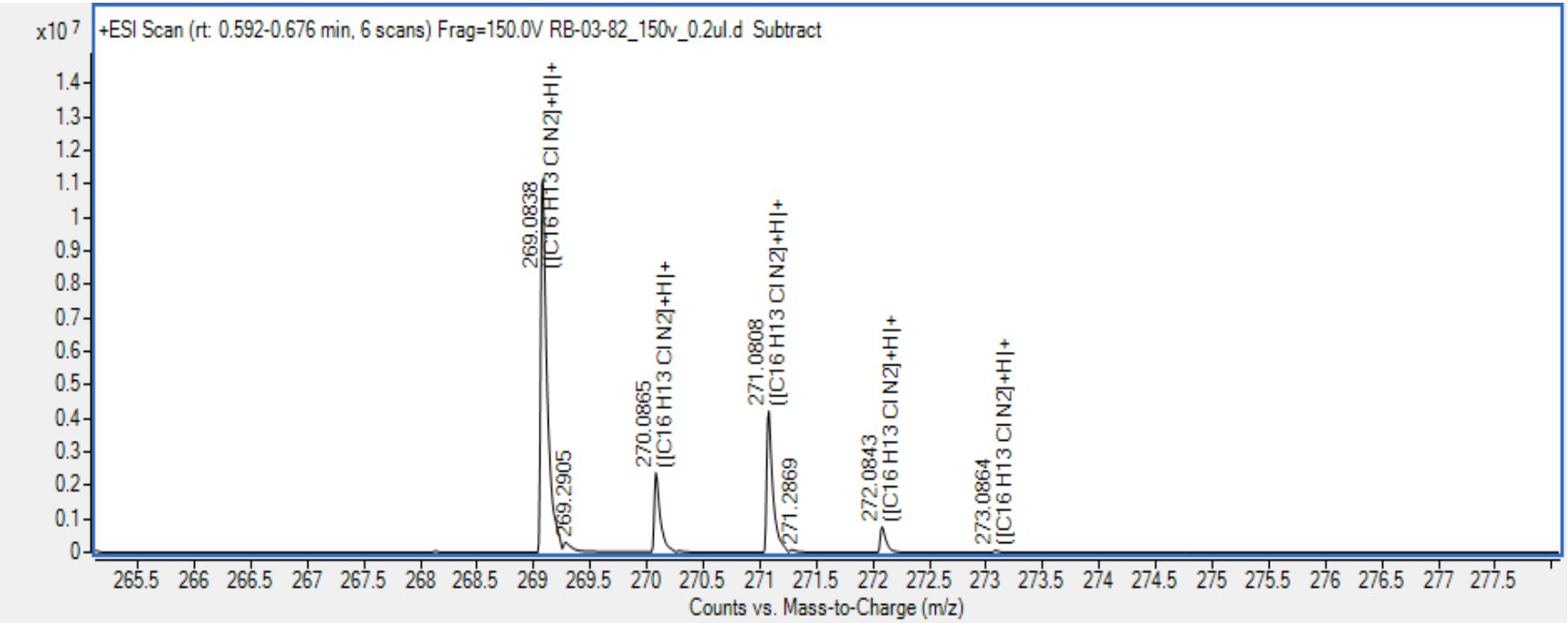
1-(4-chlorophenyl)-3-methyl-5-phenyl-1H-pyrazole (P17): ^{13}C-NMR ($CDCl_3$)



1-(4-chlorophenyl)-3-methyl-5-phenyl-1H-pyrazole (P17): ^{13}C-NMR ($CDCl_3$) _ Expanded Aromatic Region

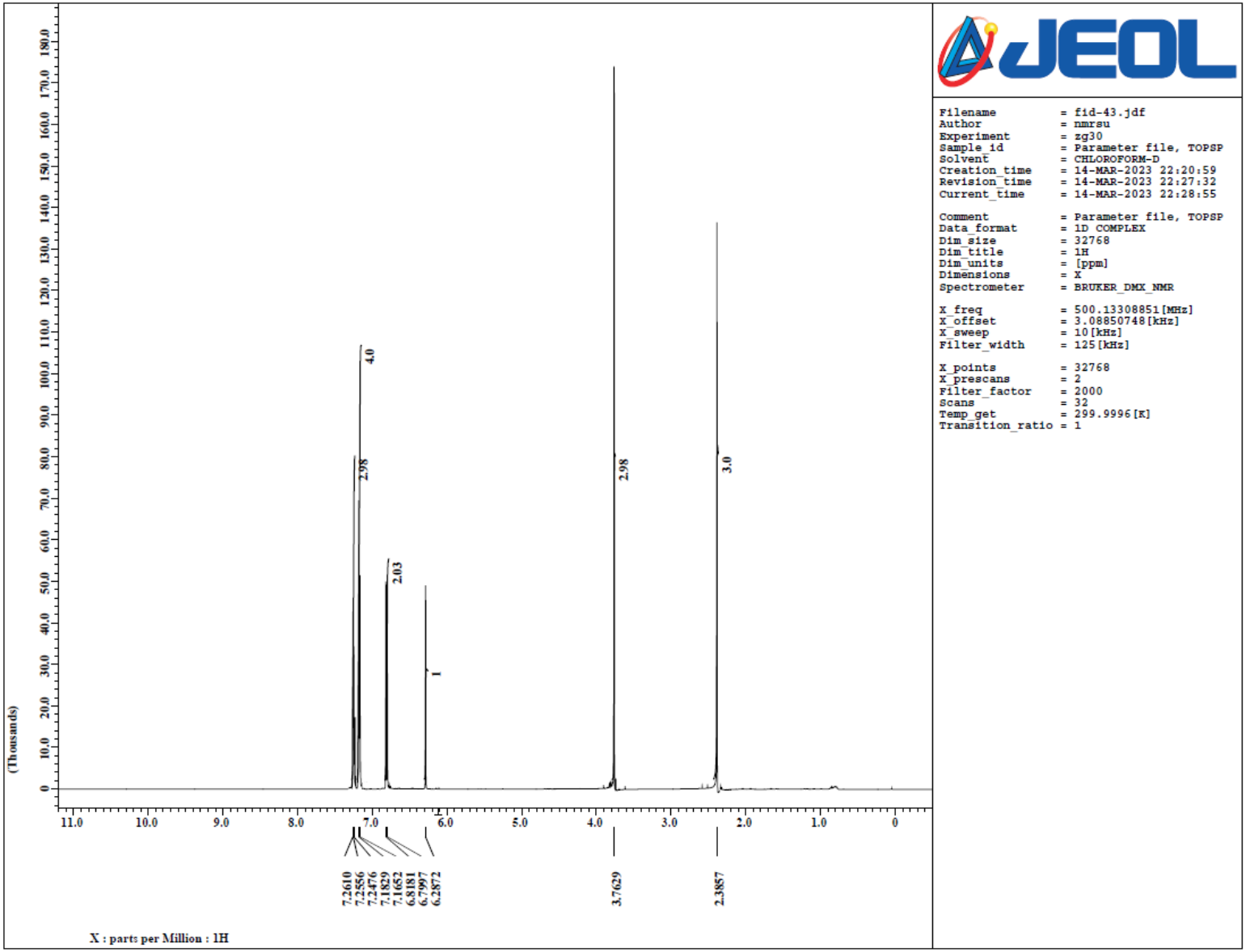


1-(4-chlorophenyl)-3-methyl-5-phenyl-1H-pyrazole (P17): ESI-MS

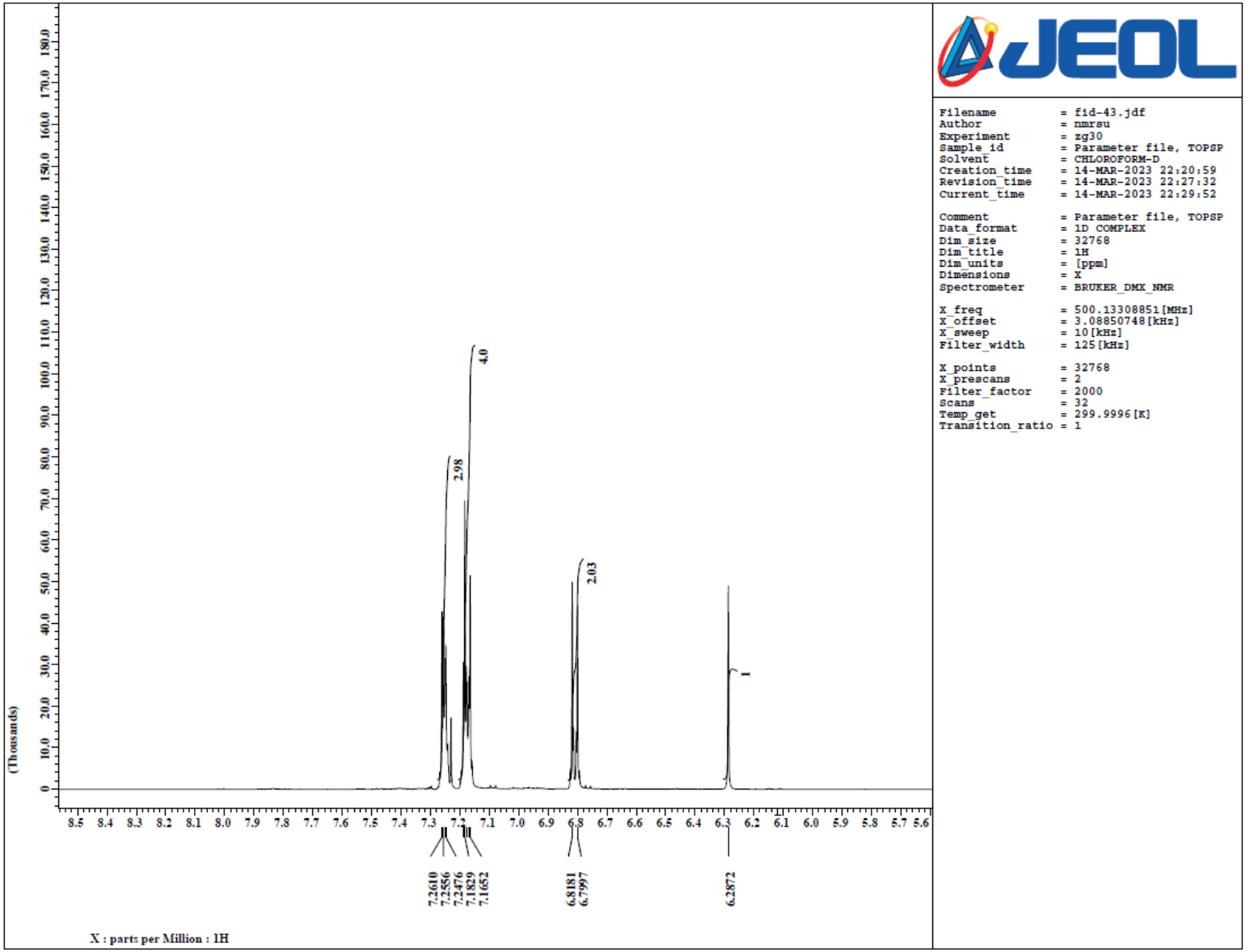


Formula,	Species,	m/z,	Score,	Diff (abs. ppm),	Mass
C16 H13 Cl N2,	(M+H)+,	269.0838,	97.12,	1.28,	268.0764

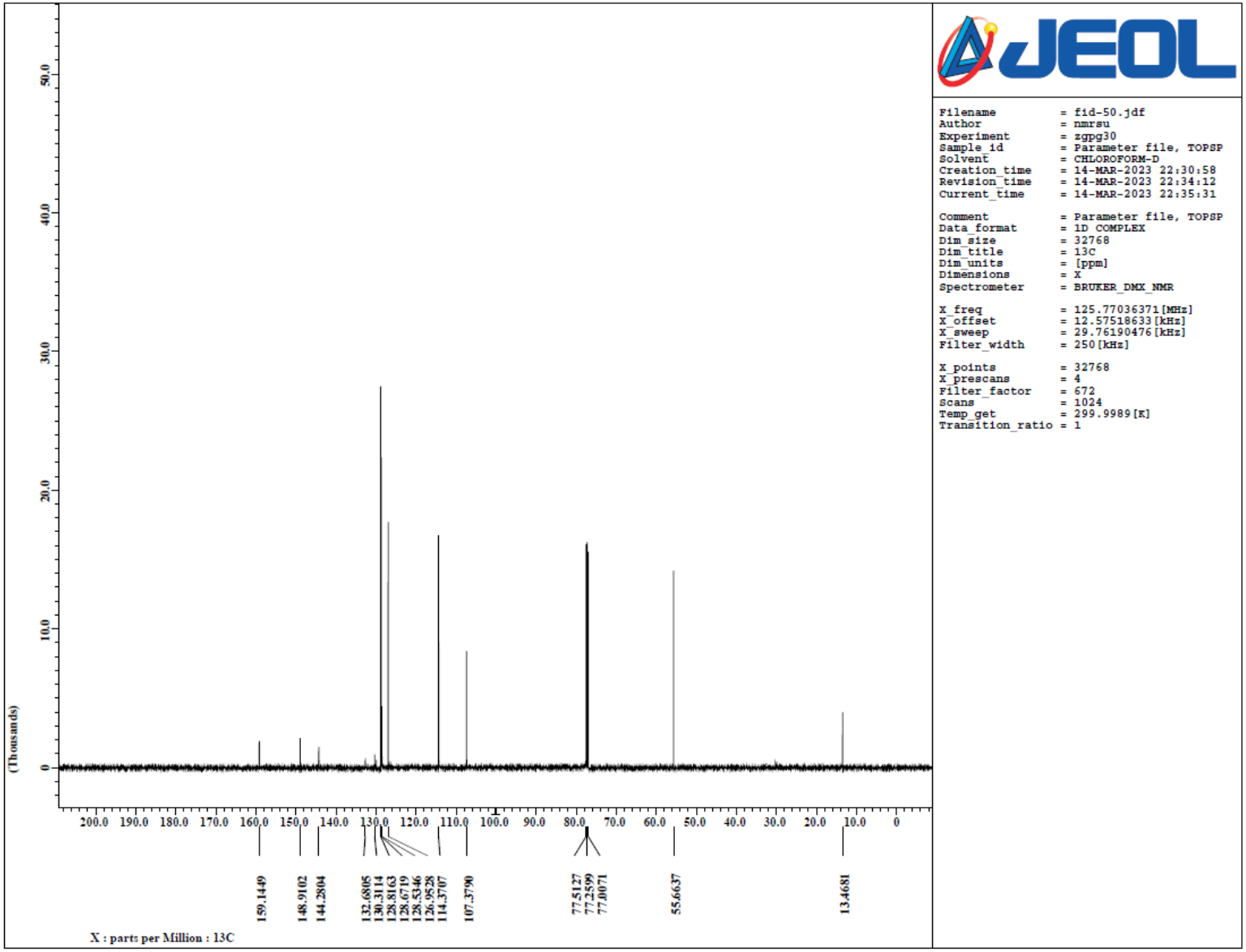
1-(4-methoxyphenyl)-3-methyl-5-phenyl-1H-pyrazole (P18): ^{1}H-NMR ($CDCl_3$)



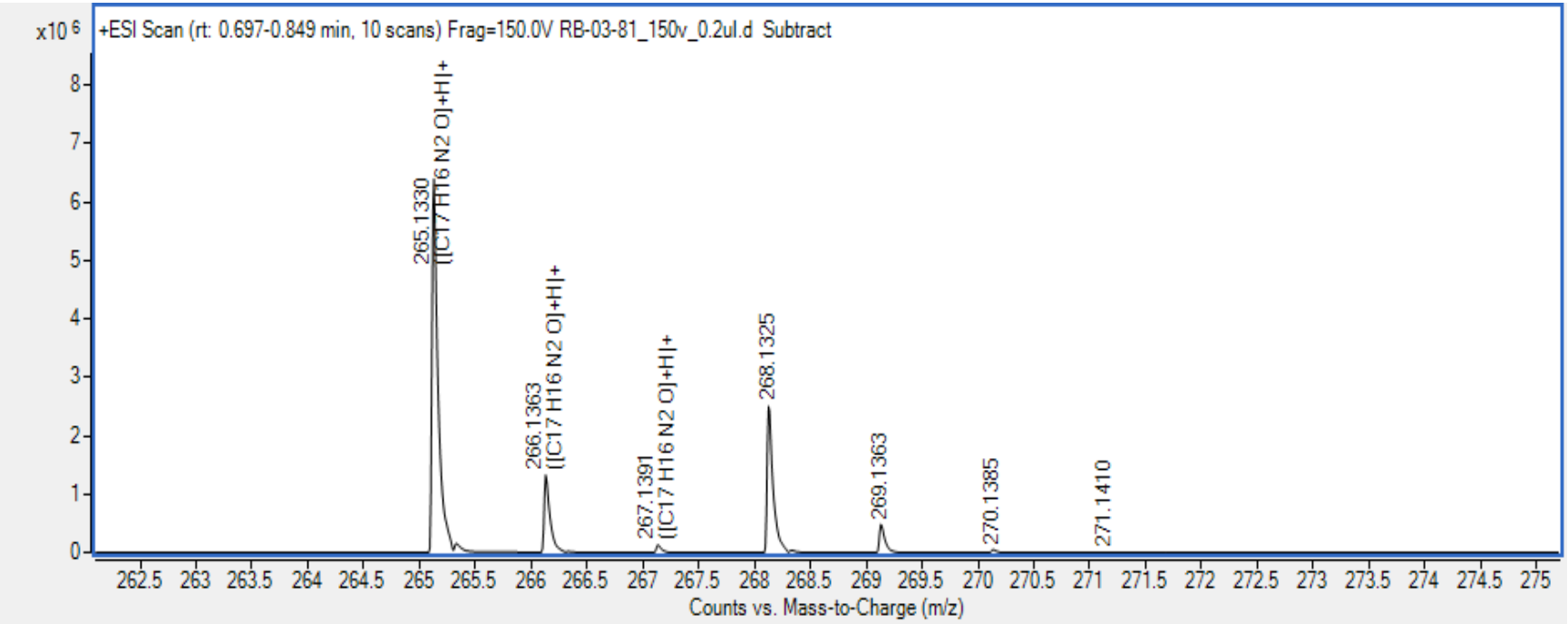
1-(4-methoxyphenyl)-3-methyl-5-phenyl-1H-pyrazole (P18): ^{1}H-NMR ($CDCl_3$) _ Expanded Aromatic Region



1-(4-methoxyphenyl)-3-methyl-5-phenyl-1H-pyrazole (P18): ^{13}C-NMR ($CDCl_3$)

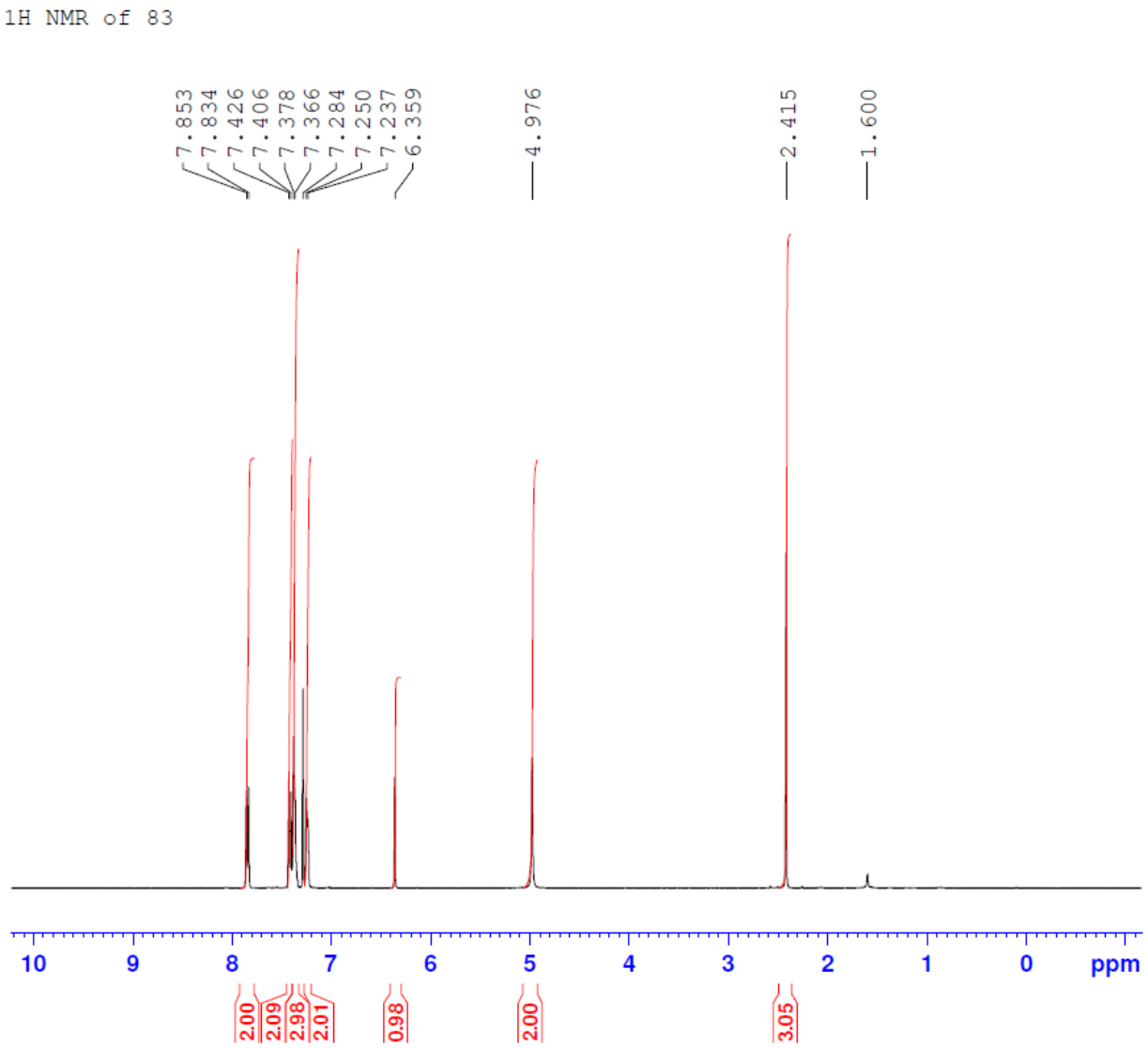


1-(4-methoxyphenyl)-3-methyl-5-phenyl-1H-pyrazole (P18): ESI-MS



Formula,	Species,	m/z,	Score,	Diff (abs. ppm),	Mass
C17 H16 N2 O,	(M+H)+,	265.1330,	98.3,	1.94,	264.1258

4-(3-methyl-5-phenyl-1H-pyrazol-1-yl)benzenesulfonamide (P19): ^{1}H-NMR ($CDCl_3$)

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Current Data Parameters
NAME            SM-83_1H
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230317
Time              16.44 h
INSTRUM       Avance Neo
PROBHD    Z140678_0053 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8196.722 Hz
FIDRES          0.250144 Hz
AQ              3.9976959 sec
RG                   101
DW                61.000 usec
DE                13.54 usec
TE                296.3 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1424709 MHz
NUC1                  1H
P0                  3.33 usec
P1                 10.00 usec
PLW1          22.64200020 W

F2 - Processing parameters
SI                 65536
SF            400.1400000 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
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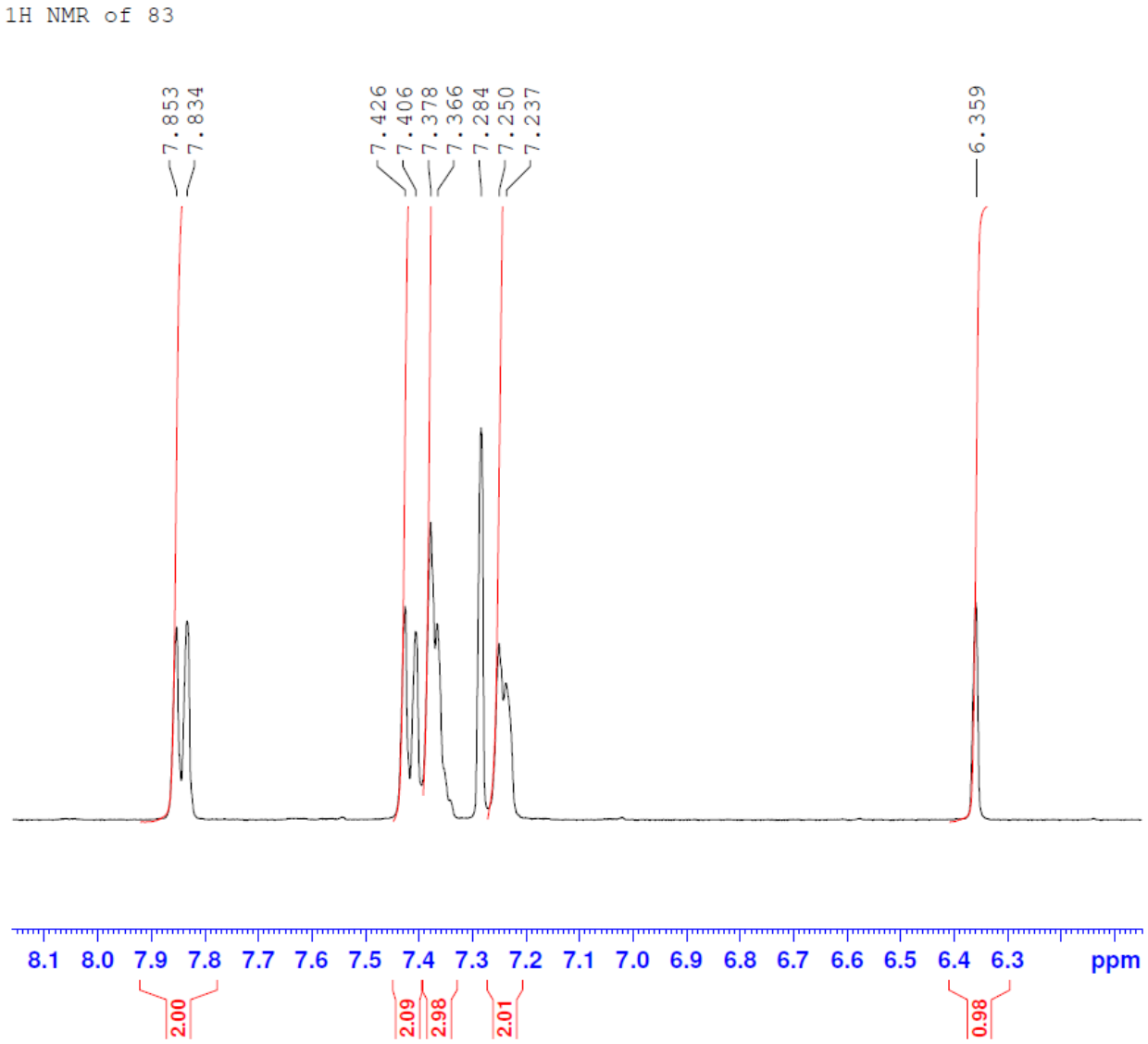
4-(3-methyl-5-phenyl-1H-pyrazol-1-yl)benzenesulfonamide (P19): ^{1}H-NMR ($CDCl_3$) _ Expanded Aromatic Region

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Current Data Parameters
NAME                  SM-83_1H
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20230317
Time                     16.44 h
INSTRUM             Avance Neo
PROBHD          Z140678_0053 (
PULPROG                  zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                   8196.722 Hz
FIDRES                0.250144 Hz
AQ                    3.9976959 sec
RG                         101
DW                      61.000 usec
DE                      13.54 usec
TE                      296.3 K
D1                    1.00000000 sec
TD0                          1
SFO1                400.1424709 MHz
NUC1                        1H
P0                       3.33 usec
P1                      10.00 usec
PLW1                22.64200020 W

F2 - Processing parameters
SI                       65536
SF                  400.1400000 MHz
WDW                         EM
SSB                          0
LB                       0.30 Hz
GB                           0
PC                        1.00
```
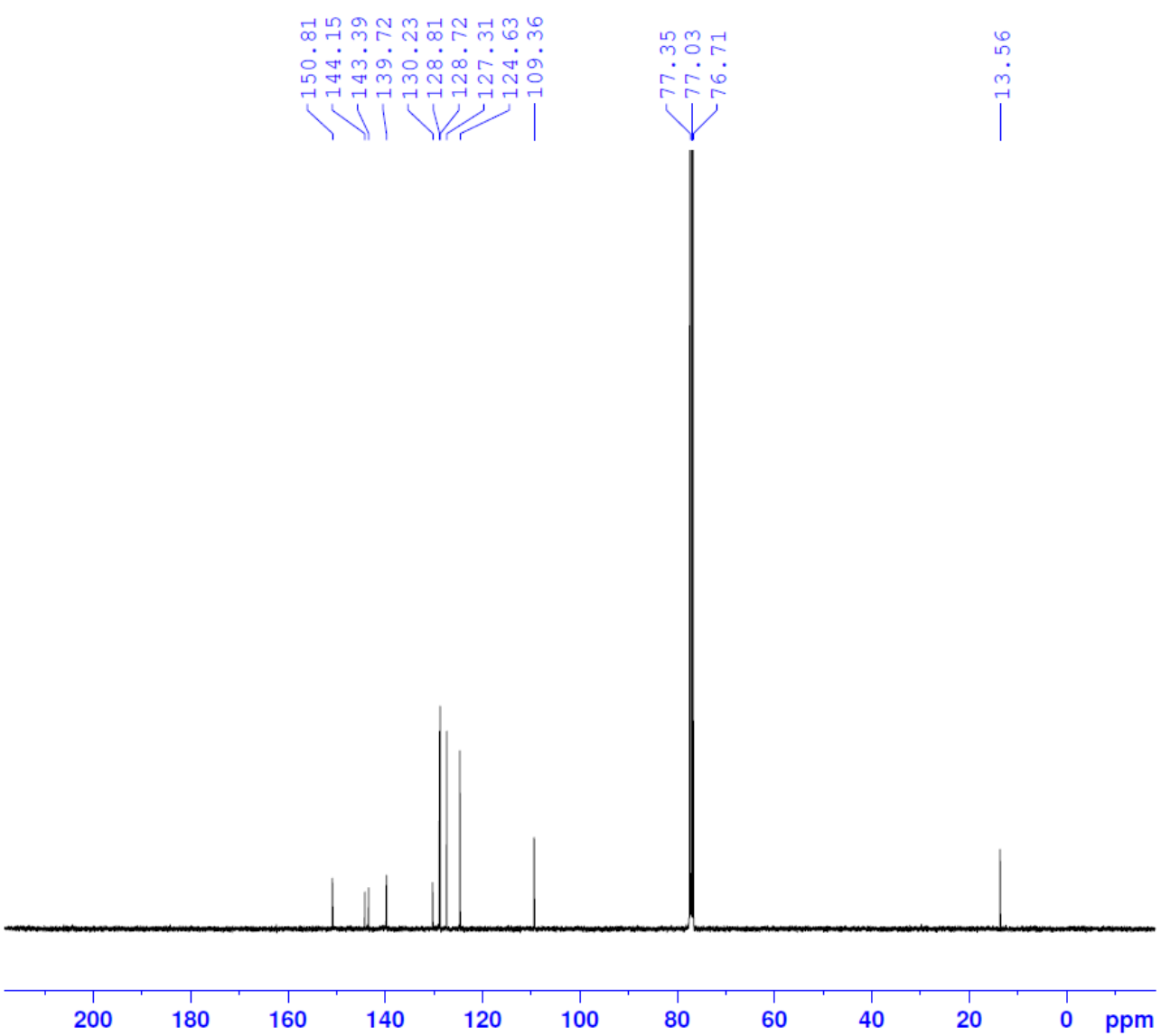

4-(3-methyl-5-phenyl-1H-pyrazol-1-yl)benzenesulfonamide (P19): ^{13}C-NMR ($CDCl_3$)

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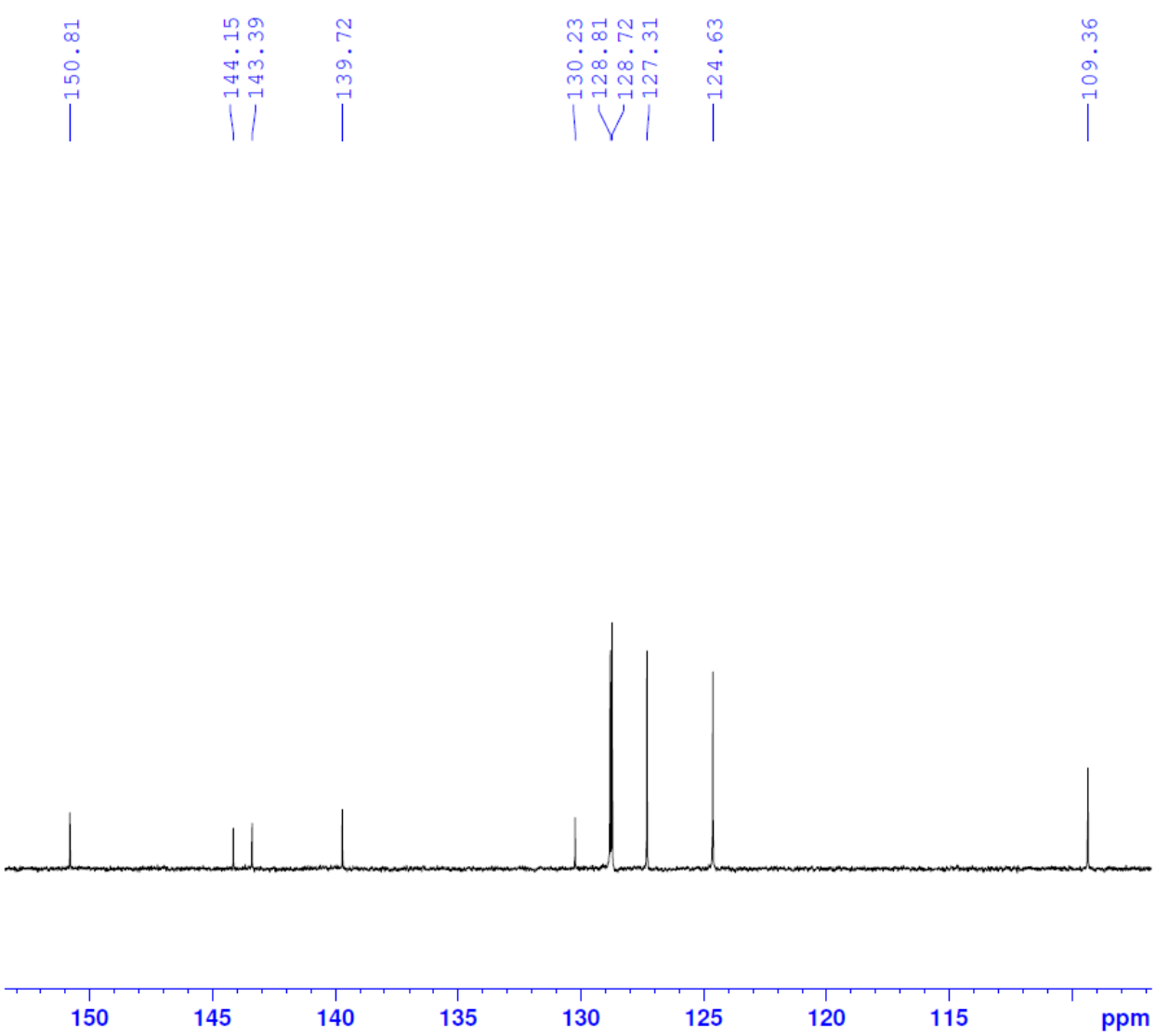
Current Data Parameters
NAME      SM-83_1Hand13C
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230317
Time              21.20 h
INSTRUM       Avance Neo
PROBHD    Z140678_0053 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           23809.523 Hz
FIDRES         0.726609 Hz
AQ             1.3762560 sec
RG                   16
DW                21.000 usec
DE                 6.50 usec
TE                297.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         100.6253446 MHz
NUC1                 13C
P0                 3.33 usec
P1                10.00 usec
PLW1         77.80000305 W
SFO2         400.1416006 MHz
NUC2                 1H
CPDPRG[2         waltz65
PCPD2             90.00 usec
PLW2         22.64200020 W
PLW12         0.27952999 W
PLW13         0.14060000 W

F2 - Processing parameters
SI                32768
SF           100.6152830 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

4-(3-methyl-5-phenyl-1H-pyrazol-1-yl)benzenesulfonamide (P19): ^{13}C-NMR ($CDCl_3$) _ Expanded Aromatic Region

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Current Data Parameters
NAME      SM-83_1Hand13C
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20230317
Time                21.20 h
INSTRUM       Avance Neo
PROBHD   Z140678_0053 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           23809.523 Hz
FIDRES         0.726609 Hz
AQ            1.3762560 sec
RG                   16
DW                21.000 usec
DE                 6.50 usec
TE                297.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         100.6253446 MHz
NUC1                 13C
P0                3.33 usec
P1               10.00 usec
PLW1         77.80000305 W
SFO2         400.1416006 MHz
NUC2                 1H
CPDPRG[2         waltz65
PCPD2             90.00 usec
PLW2         22.64200020 W
PLW12         0.27952999 W
PLW13         0.14060000 W

F2 - Processing parameters
SI                32768
SF           100.6152830 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```
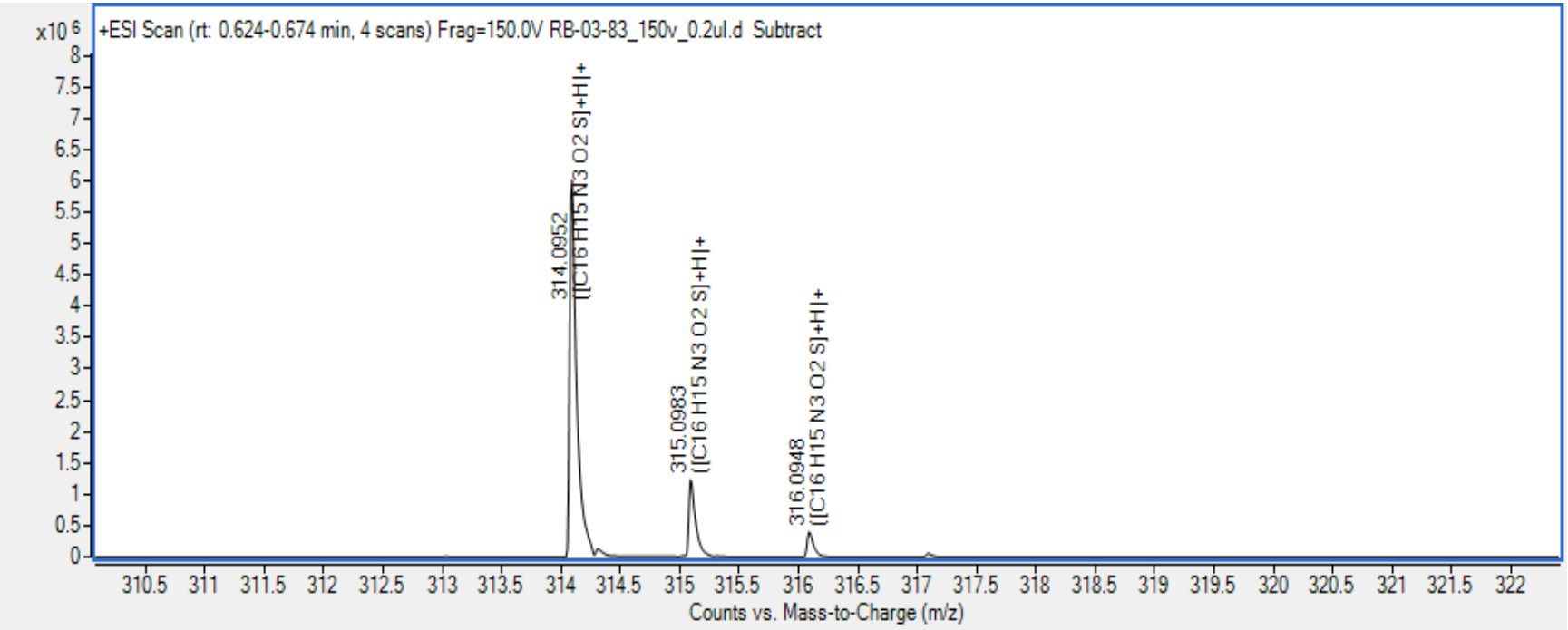

4-(3-methyl-5-phenyl-1H-pyrazol-1-yl)benzenesulfonamide (P19): ESI-MS



Formula,	Species,	m/z,	Score,	Diff (abs. ppm),	Mass
C16 H15 N3 O2 S,	(M+H)+,	314.0952,	98.14,	1.61,	313.088