Chemistry of Heterocyclic Compounds **2024**, *60*(7/8), 390–402

Polysubstituted pyrans, chromenes, and chromenopyridines with isoxazole or isothiazole moiety: synthesis, structure, and antitumor activity

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SUPPLEMENTARY INFORMATION

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1. Methodology and results of biotesting

Materials and methods.

Media and components of culture media: medium DMEM 5648, antibiotics (penicillin, streptomycin, amphotericin B), fetal bovine serum (FBS), trypsin - EDTA (0,25% trypsin – 0,02% EDTA) (Sigma), dimethyl sulfoxide (DMSO).

Cell lines. Hela (cervical cancer, human), glioma *C6* (rat) from the collection of the Republican Research and Practical Center for Epidemiology and Microbiology, Belarus.

Study of the antitumor effect of compounds. Cells were seeded into wells of 96-well plates (Corning) in DMEM supplemented with 10% FBS and antibiotics (penicillin, streptomycin, amphotericin B). A day later, the test compounds were added to the wells. In the control, a solvent was added (dimethyl sulfoxide at a final concentration of 0,1%). Cultivated for 48 h at 37 °C and 5% CO_2. Then, cell samples were analyzed using the MTT assay with 3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyl-tetrazolium bromide (MTT - thiazolyl blue tetrazolium bromide, Glentham Life Sciences).

Medicines.

11a, **12e**, **15a**, **15c** – 0,5 mM stock solutions in 1% DMSO were prepared (first substances were diluted in DMSO, then brought to the required volume with isotonic sodium chloride solution). Further dilutions were made with 1% DMSO. Solutions were added to the wells of the plate with growing cells in a volume ratio 1 (**11a**, **12e**, **15a** *и* **15c**) : 9 (medium with cells).

Carboplatin (Cp) is an alkylating drug, a platinum derivative. Forms cross-links between adjacent guanine pairs in DNA.

Doxorubicin (Dx) is an anthracycline antibiotic with antitumor action. Intercalates into DNA, disrupts DNA synthesis, and also causes other metabolic disorders in cells.

Cyclophosphamide (Ca) is an alkylating agent that alkylates DNA and proteins.

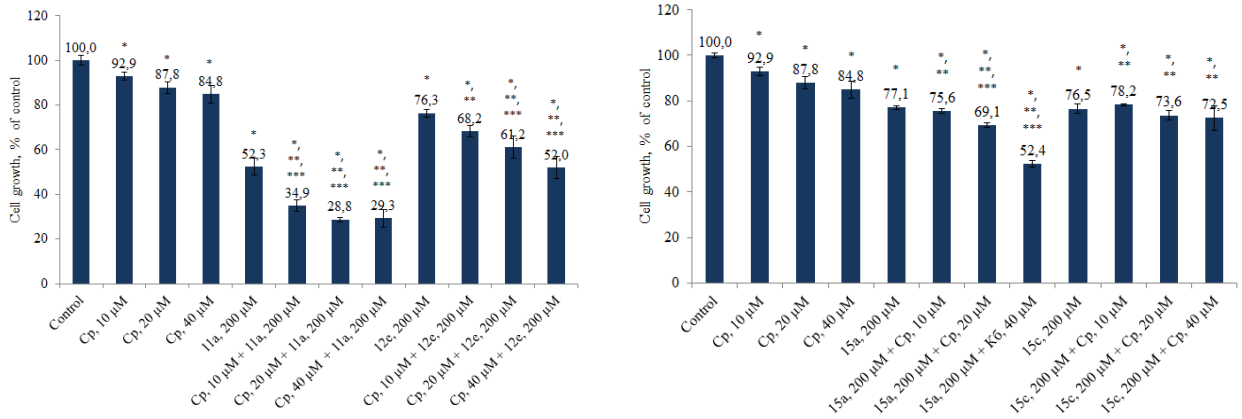
Fluorouracil (Fu) is an antimetabolite, an antagonist of pyrimidines (uracil). Disturbs DNA synthesis, suppresses RNA synthesis.

Ribomustine (Rm) (active component – bendamustine hydrochloride) is an alkylating drug that disrupts the structure and synthesis of DNA.

Results.

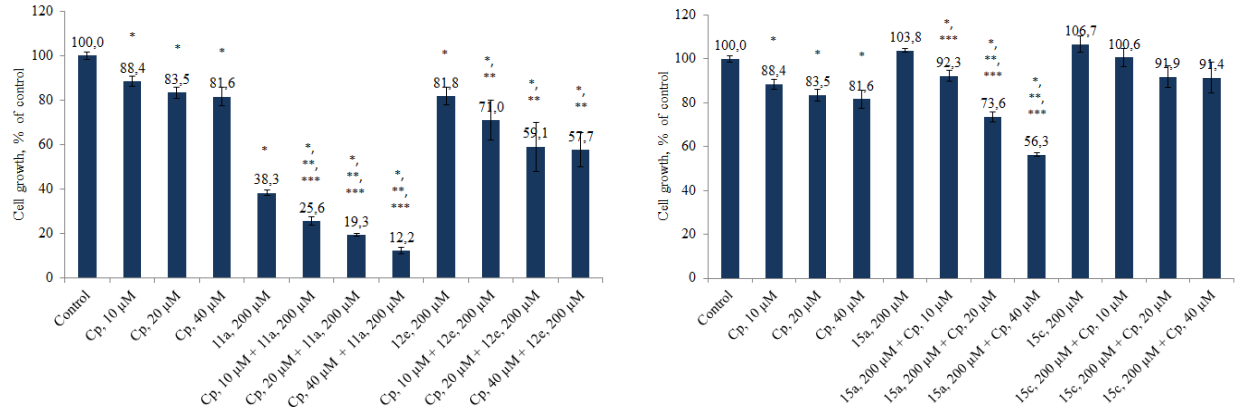
Substances **11a**, **12e**, **15a** and **15c** at a concentration of 200 μM suppressed the growth of *C6* glioma cells (Fig. S1), substances **11a**, **12e** – the growth of *Hela* cells (Fig. S2). Carboplatin at a concentration of 10–40 μM had a dose-dependent inhibitory effect on the growth of tumor

cells. The combined use of compound **11a** with carboplatin led to an enhancing of inhibitory effect. A similar but less pronounced effect was observed when carboplatin was combined with the compound **12e**; the same trend was observed when **12e** acted on *Hela* cells. Co-administration of carboplatin with compound **15a** provided some additional effect only at carboplatin concentrations of 20 and 40 μM, Fig. S2. Co-administration of carboplatin with compound **15c** resulted in a weak tendency to increase the effect compared to compound **15c**.



(a) (b)

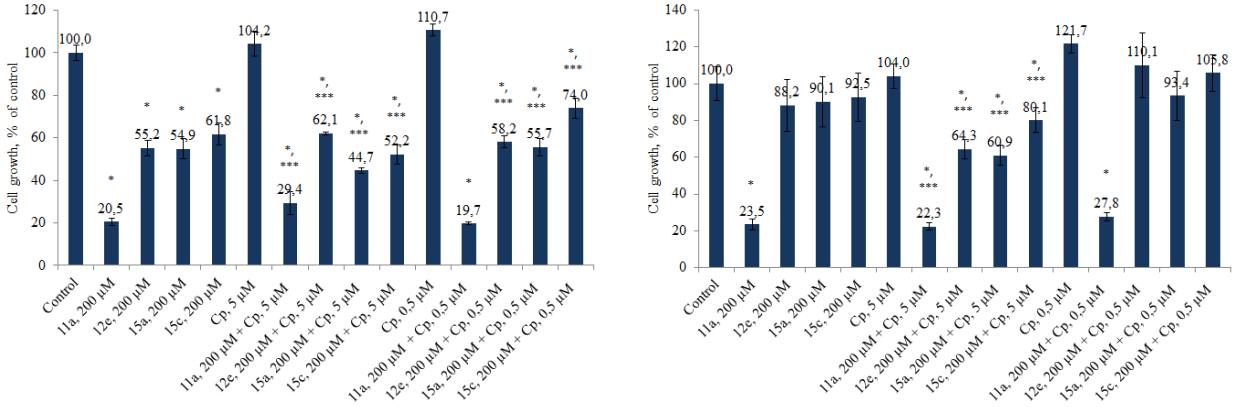
Fig. S1. Effect of carboplatin at a concentrations of 10–40 μM and substances **11a**, **12e** (a) and **15a**, **15c** (b) at a concentration of 200 μM on the growth of *C6* glioma cells; *p < 0,05 when compared with control; **p < 0,05 when compared with the effect of carboplatin; ***p < 0,05 when compared with the effect of a heterocyclic compound (Mann-Whitney test)



(a) (b)

Fig. S2. Effect of carboplatin at a concentrations of 10–40 μM and substances **11a**, **12e** (a) and **15a**, **15c** (b) at a concentration of 200 μM on the growth of *Hela* cells; *p < 0,05 when compared with control; **p < 0,05 when compared with the effect of carboplatin; ***p < 0,05 when compared with the effect of a heterocyclic compound (Mann-Whitney test)

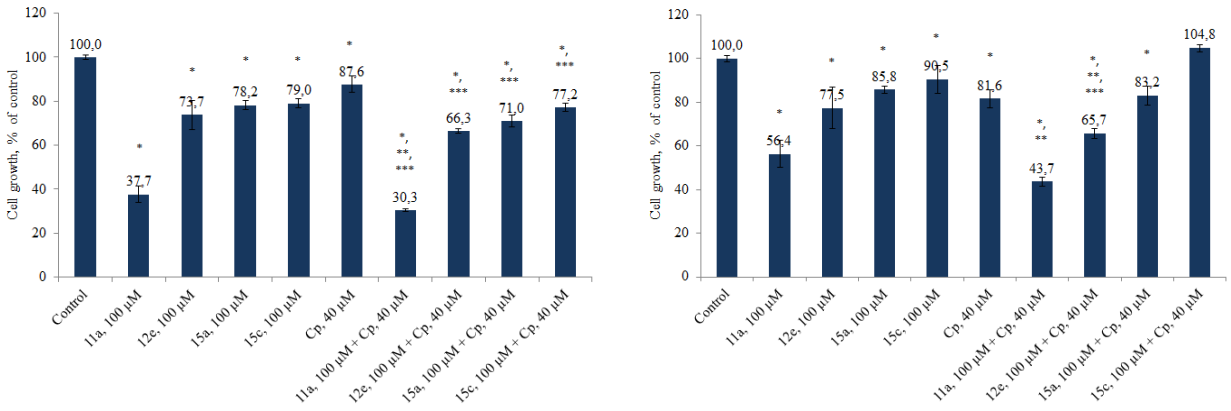
Carboplatin at low doses (5 and 0,5 μM) didn't affect the growth of *C6* glioma and *Hela* cells. At low doses of carboplatin (5 and 0,5 μM) substances **11a**, **12e**, **15a** and **15c** showed their own inhibitory effect against *C6* glioma cells, but no additional effect was detected. There was a weak tendency to enhance the effect of 5 μM carboplatin when combined with **15a** on *C6* glioma cells (Fig. S3a) and a tendency to enhance the effect of 5 μM carboplatin when combined with **12e**, **15a**, **15c** on *Hela* cells (Fig. S3b). In this and the next experiment, compounds **11a**, **12e**, **15a** and **15c** inhibited the growth of *C6* glioma cells more significantly than in the previous experiment. This could be because of some variation in the onset of the exponential phase of cell growth from experiment to experiment. It can be assumed that conducting the experiment in the most rapid growth phase contributes to a better expression of the effect of growth-inhibiting drugs.



(a) (b)

Fig. S3. Effect of carboplatin at a concentrations of 5 and 0,5 μM and substances **11a**, **12e**, **15a** and **15c** at a concentration of 200 μM on the growth of *C6* glioma (a) and *Hela* (b) cells; *p < 0,05 when compared with control; **p < 0,05 when compared with the effect of a heterocyclic compound; ***p < 0,05 when compared with the effect of carboplatin (Mann-Whitney test)

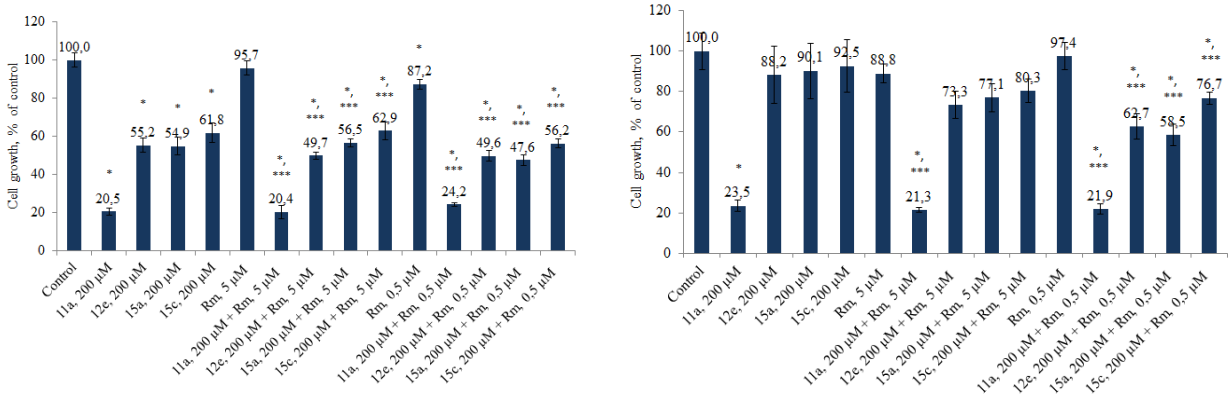
Compounds **11a**, **12e**, **15a** and **15c** at a dose of 100 μM with carboplatin at a concentration of 40 μM tended to show a weak additional effect against *C6* glioma cells (Fig. S4a). For *Hela* cells, a similar effect was observed when using **11a** or **12e** (Fig. S4b).



(a) (b)

Fig. S4. Effect of carboplatin at a concentration of 40 μM and substances **11a**, **12e**, **15a** and **15c** at a concentration of 100 μM on the growth of C*6* glioma (a) and *Hela* (b) cells; *p < 0,05 when compared with control; **p < 0,05 when compared with the effect of a heterocyclic compound; ***p < 0,05 when compared with the effect of carboplatin (Mann-Whitney test)

An experiment with ribomustine was conducted taking it in a small dose of 5 and 0,5 μM. At such doses it had almost no effect on cell growth. The combined use of 5 μM ribomustine with **11a**, **12e**, **15a** and **15c** didn't lead to a significant change in the effect compared to the effect of heterocyclic compounds, and when using 0,5 μM ribomustine together with **12e**, **15a** and **15c**, a tendency to enhance the effect was observed (Fig. S5). We have already noted a similar phenomenon of increasing synergistic effect with decreasing dose.[1]

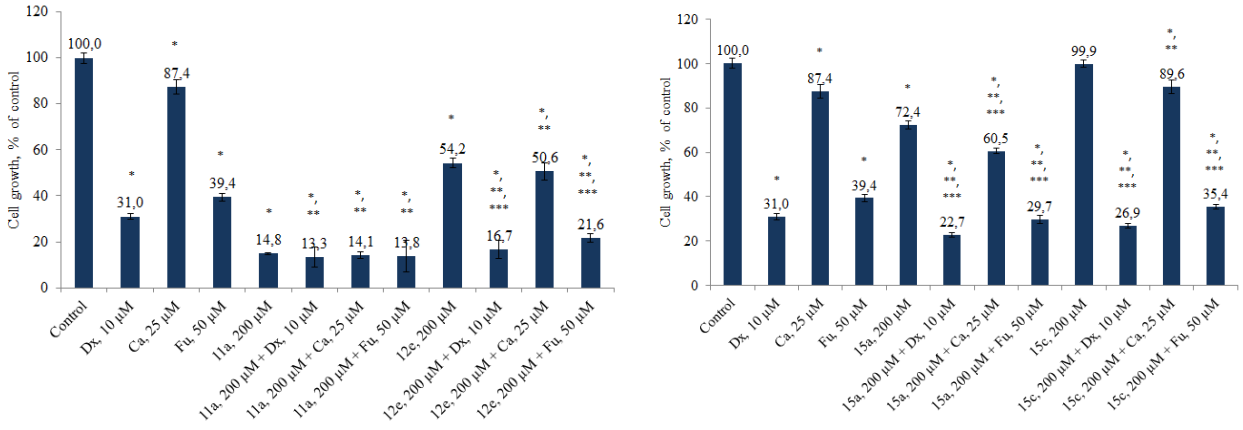


(a) (b)

Fig. S5. Effect of ribomustine at a concentrations of 5 and 0,5 μM and substances **11a**, **12e**, **15a** and **15c** at a concentration of 200 μM on the growth of *C6* glioma (a) and *Hela* (b) cells; *p < 0,05 when compared with control; **p < 0,05 when compared with the effect of a heterocyclic compound; ***p < 0,05 when compared with the effect of ribomustine (Mann-Whitney test)

An experiment was conducted on *C6* glioma cells using other chemotherapy drugs: doxorubicin, cyclophosphamide and fluorouracil. Compound **11a** had a strong inhibitory effect;

against this background, no enhancement of the effect of chemotherapy drugs was detected (compared to **11a**). Derivatives **12e** and **15a** enhanced the effect of doxorubicin, cyclophosphamide and fluorouracil (Fig. S6a, b), and **15c** caused a weak tendency to increase the effect (with doxorubicin and uracil) or had no effect (with cyclophosphamide) (Fig. S6b).



(a) (b)

Fig. S6. The effect of combined use of doxorubicin, cyclophosphamide and fluorouracil and compounds **11a**, **12e** (a) and **15a**, **15c** (b) at a concentration of 200 μM on the growth of *C6* glioma cells; *p < 0,05 when compared with control; ** p < 0,05 when compared with the effect of a chemotherapy drug; *** p < 0,05 when compared with the effect of a heterocyclic compound (Mann-Whitney test)

Conclusion

1. Compounds **11a**, **12e**, **15a**, **15c** can have an inhibitory effect on the growth of tumor cells. The most active one is **11a**, the least active is **15c**.

2. The combined use of compounds **11a**, **12e**, **15a** with chemotherapy drugs leads to an increased inhibitory effect on tumor cells compared to the action of the drugs alone. Compound **15c** weakly enhances or doesn't enhance the effects of drugs.

3. The effects of **11a**, **12e**, **15a**, **15c** are similar for *C6* glioma and *Hela* cells.

4. Experiments with ribomustine revealed an effect of increasing the synergistic effect of compounds **12e**, **15a**, **15c** with a decrease in the dose of the chemotherapy drug.

2. X-Ray diffraction analysis

X-ray diffraction experiments were carried out on an automatic four-circle area-detector diffractometer Bruker KAPPA APEX II (MoKα radiation).[2] The unit cell constants were refined over the whole data set.[3] The experimental intensities were corrected for absorption using the SADABS program.[4] The structure was solved by the intrinsic phasing method (SHELXT[5]) and refined by the full-matrix least squares method (SHELXL-2018/3[6]) on F^2 for all data in the anisotropic approximation for all non-hydrogen atoms. The H atoms of CH, CH_2, and CH_3 groups were introduced at geometrically calculated positions with $U_{iso}(H) = 1.2U_{eq}(C)$ for CH, and CH_2 groups and $U_{iso}(H) = 1.5U_{eq}(C)$ for CH_3 ones. The H atoms of NH_2 group in **9b** were introduced at geometrically calculated positions with $U_{iso}(H) = 1.2U_{eq}(N)$, in **11a** and **12d** the H atoms of NH_2 group were refined with $U_{iso}(H) = 1.2U_{eq}(N)$. The structure **11a** contains, contains, presumably, a half of the disordered dichloromethane molecule per formula unit. The contribution of the disordered solvent was removed using the SQUEEZE procedure in the PLATON program package.[7]

The main crystallographic data and characteristics of X-ray diffraction experiment are given in Table S1. The atomic coordinates have been deposited with the Cambridge Crystallographic Data Centre, depositions CCDC 2333536−2333538.

Table S1. Crystal data and structure refinement.

Identification code	**9b**	**11a**	**12d**
CCDC deposition number	2333536	2333537	2333538
Empirical formula	$C_{17}H_{18}Cl_2N_2O_4S$	$C_{13.5}H_{10}Cl_3N_3O_2S$	$C_{21}H_{19}N_3O_3$
Formula weight	417.29	384.65	361.39
Temperature/K	100(2)	100(2)	100(2)
Crystal system	monoclinic	monoclinic	monoclinic
Space group	C2/c	P2/c	C2/c
a/Å	27.927(3)	11.7288(10)	26.3449(18)
b/Å	8.1910(10)	8.8967(8)	8.6497(6)
c/Å	20.626(4)	16.6618(14)	16.8120(11)
α/°	90	90	90
β/°	129.048(3)	107.973(4)	104.961(3)
γ/°	90	90	90
Volume/$Å^3$	3664.3(9)	1653.8(3)	3701.2(4)
Z	8	4	8
$\rho_{calc}g/cm^3$	1.513	1.545	1.297
μ/mm^{-1}	0.494	0.690	0.089

F(000)	1728.0	780.0	1520.0
Crystal size/mm^3	0.5 × 0.06 × 0.03	0.36 × 0.2 × 0.06	0.18 × 0.16 × 0.08
Radiation	MoKα (λ = 0.71073)	MoKα (λ = 0.71073)	MoKα (λ = 0.71073)
2Θ range for data collection/°	7.516 to 59.998	6.886 to 59.998	8.674 to 59.994
Index ranges	-39 ≤ h ≤ 38, -9 ≤ k ≤ 11, -28 ≤ l ≤ 28	-16 ≤ h ≤ 16, -12 ≤ k ≤ 12, -20 ≤ l ≤ 23	-36 ≤ h ≤ 36, -12 ≤ k ≤ 12, -23 ≤ l ≤ 23
Reflections collected	40315	28436	35141
Independent reflections	5334 [R_{int} = 0.1694, R_{sigma} = 0.1371]	4824 [R_{int} = 0.0892, R_{sigma} = 0.0749]	5379 [R_{int} = 0.0828, R_{sigma} = 0.0720]
Data/restraints/parameters	5334/0/235	4824/0/196	5379/0/252
Goodness-of-fit on F^2	1.001	1.021	1.025
Final R indexes [I>=2σ (I)]	R_1 = 0.0564, wR_2 = 0.0988	R_1 = 0.0464, wR_2 = 0.0981	R_1 = 0.0619, wR_2 = 0.1446
Final R indexes [all data]	R_1 = 0.1469, wR_2 = 0.1260	R_1 = 0.0845, wR_2 = 0.1118	R_1 = 0.1230, wR_2 = 0.1767
Largest diff. peak/hole / e $Å^{-3}$	0.41/-0.47	0.43/-0.36	0.58/-0.27

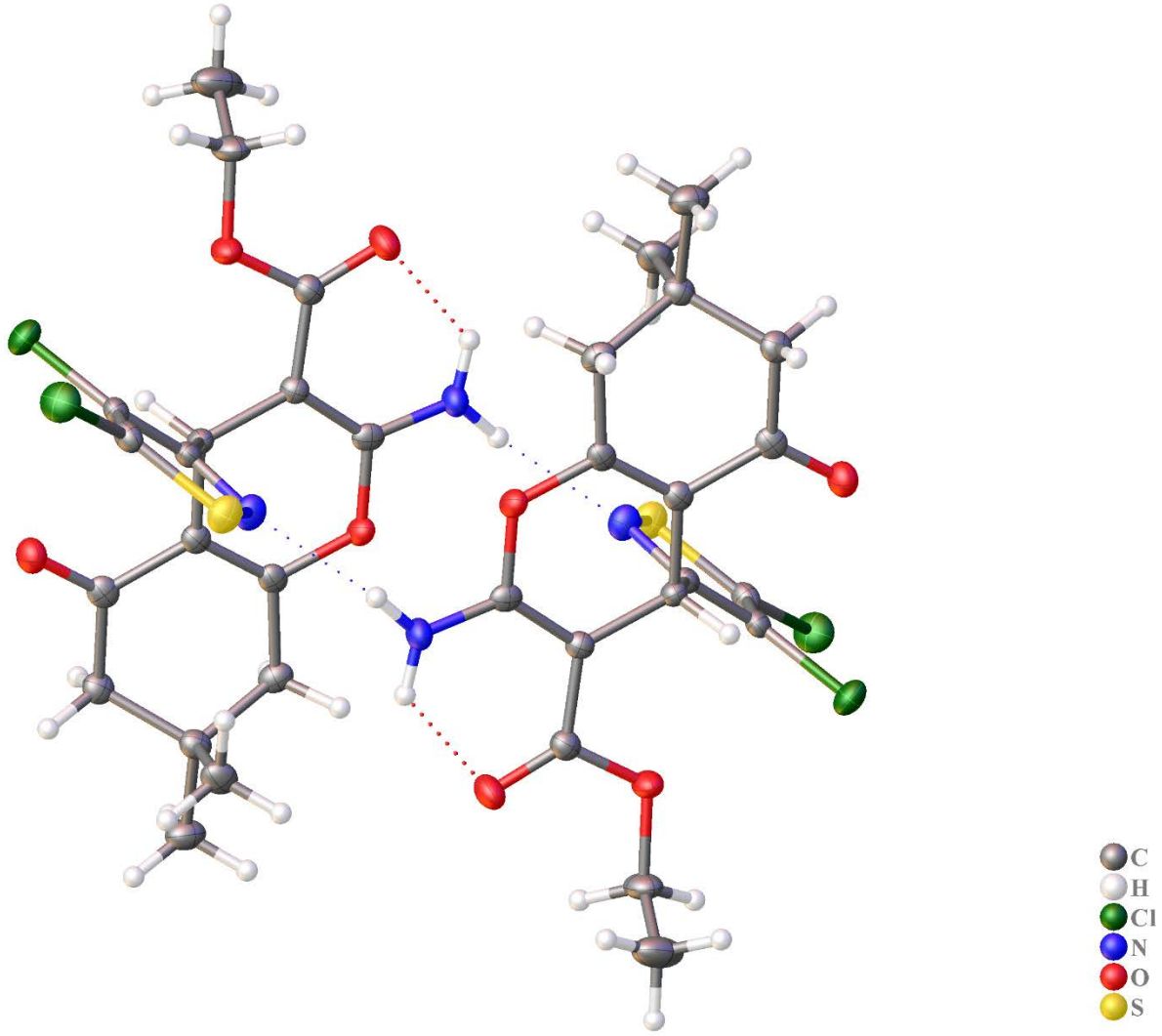


Figure S7. H-Bonding in **9b**.

Table S2. Hydrogen bonds for **9b**.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
N1	H1A	O3	0.88	2.07	2.691(3)	127
N1	H1B	N12*	0.88	2.15	3.008(4)	164

*1-X,1-Y,1-Z

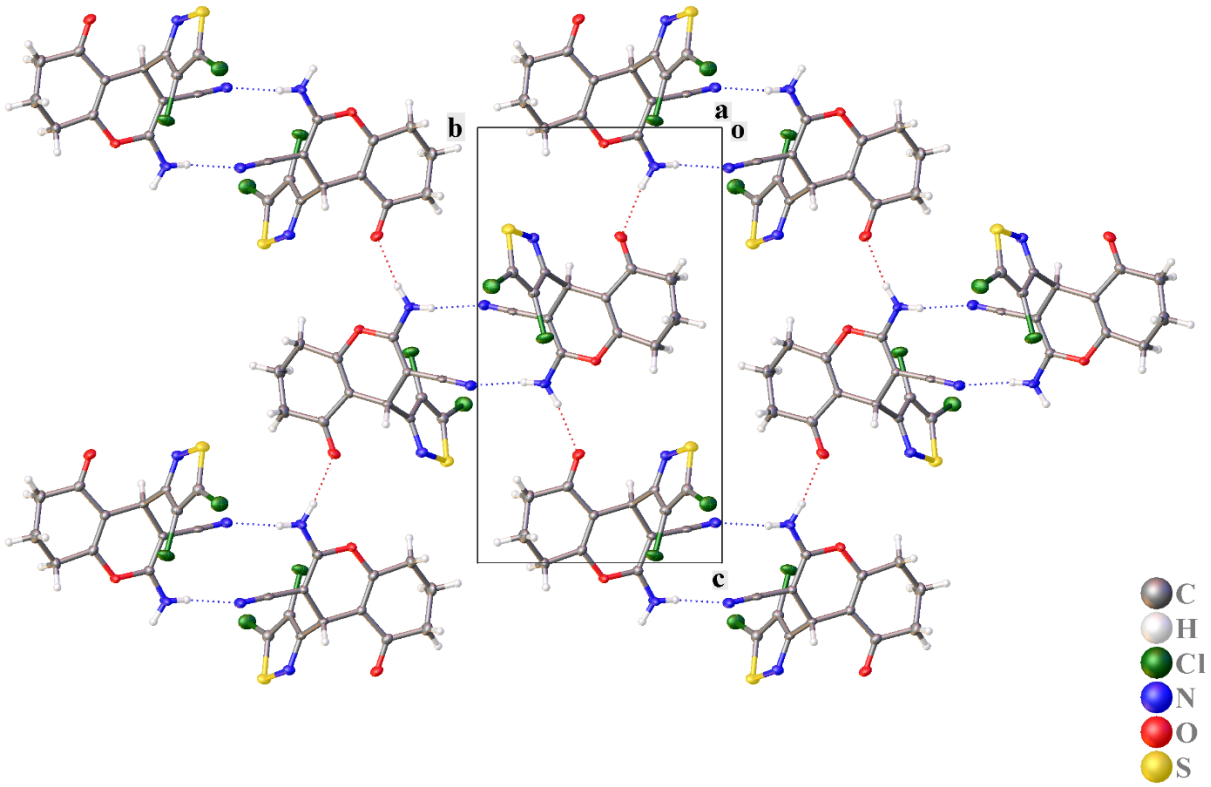


Figure S8. H-Bonding in **11a**.

Table S3. Hydrogen bonds for **11a**.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
N1	H1A	N2*	0.82(2)	2.18(2)	2.991(3)	172(2)
N1	H1B	O2**	0.87(2)	2.06(2)	2.911(2)	167(2)

*-X,2-Y,1-Z; **+X,1-Y,1/2+Z

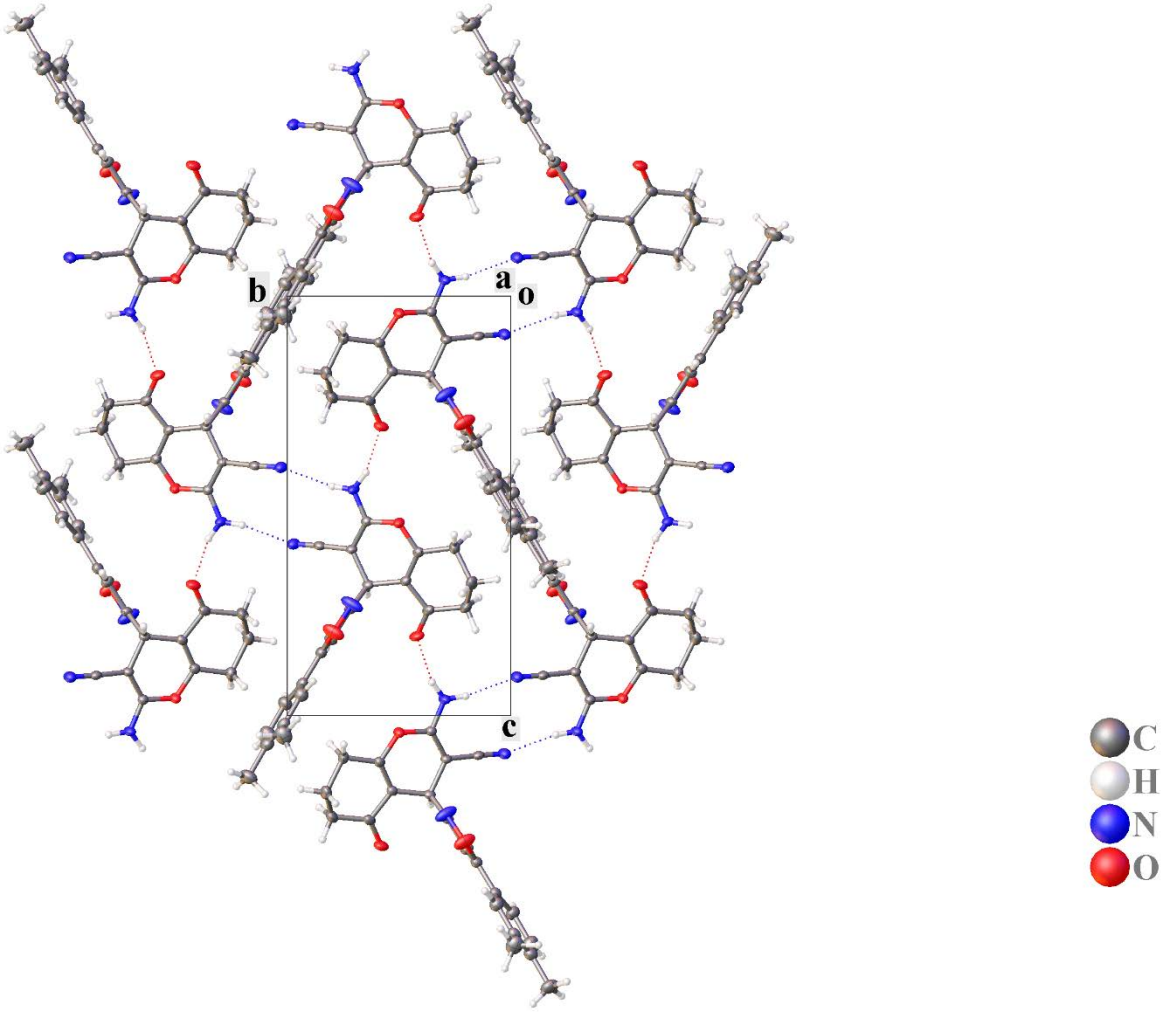


Figure S9. H-Bonding in **12d**.

Table S4. Hydrogen bonds for **11d**.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
N2	H2A	N3*	0.85(2)	2.21(3)	3.054(3)	169(2)
N2	H2B	O5**	0.91(3)	1.99(3)	2.868(2)	162(2)

*1-X,-Y,-Z; **+X,1-Y,-1/2+Z

3. References

1. Potkin V.; Pushkarchuk A.; Zamaro A.; Zhou H.; Kilin S.; Petkevich S.; Kolesnik I.; Michels D.; Lyakhov D.; Kulchitsky V. *SciRep*. **2023**, *13*, 13624.

2. Apex2 // Bruker AXS Inc., Madison, Wisconsin, USA (2008).

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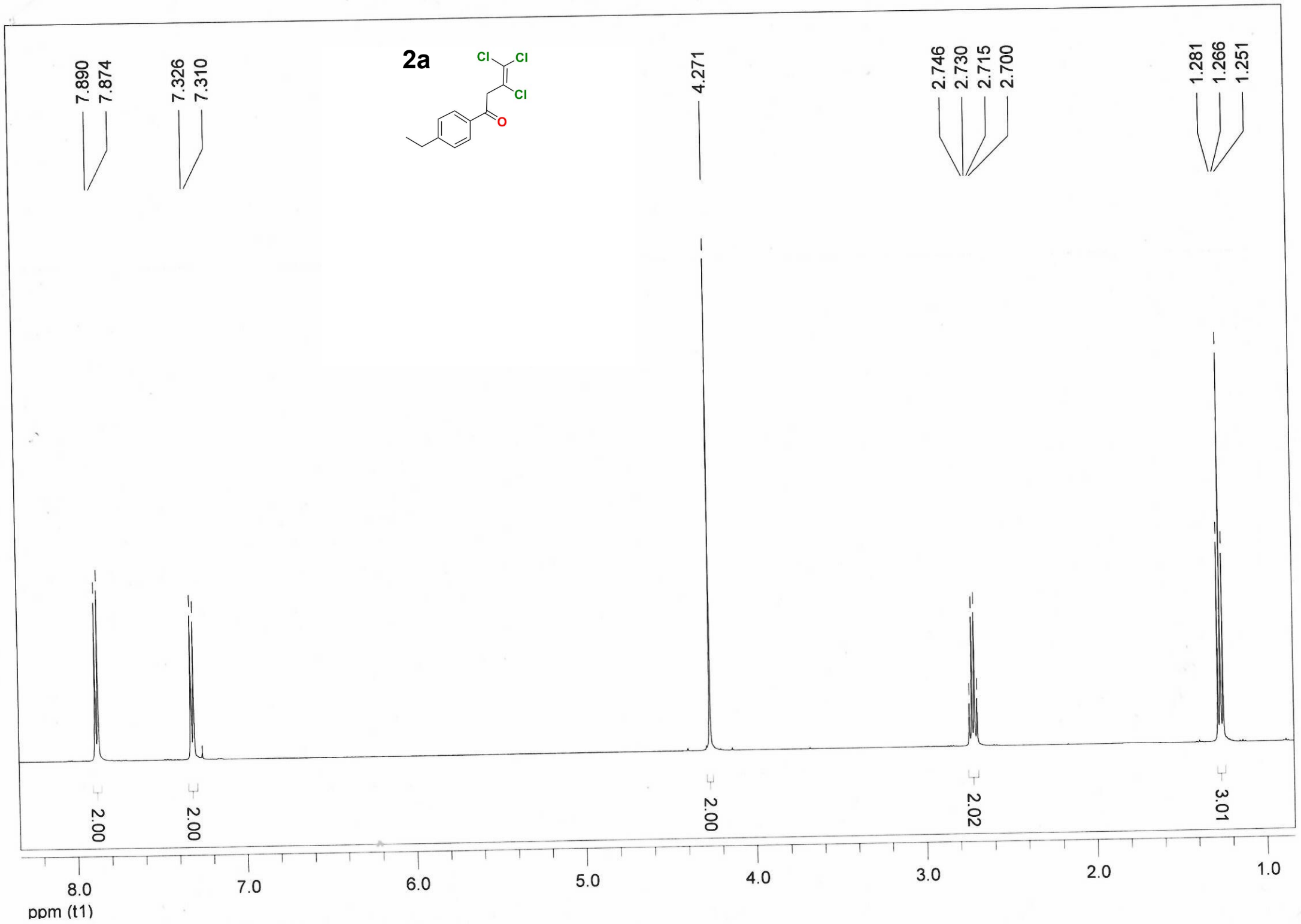
4. Krause L.; Herbst-Irmer R.; Sheldrick G.M.; Stalke D. *J. Appl. Cryst*. **2015**, *48*, 3.

5. Sheldrick G.M. *Acta Crystallogr*. **2015**, *A71*, 3.

6. Sheldrick G.M. *Acta Crystallogr*. **2015**, *C71*, 3.

7. Spek A.L. *Acta Crystallogr*. **2015**, *C71*, 9.

4. Copy of the NMR spectra and the data of mass-spectrometry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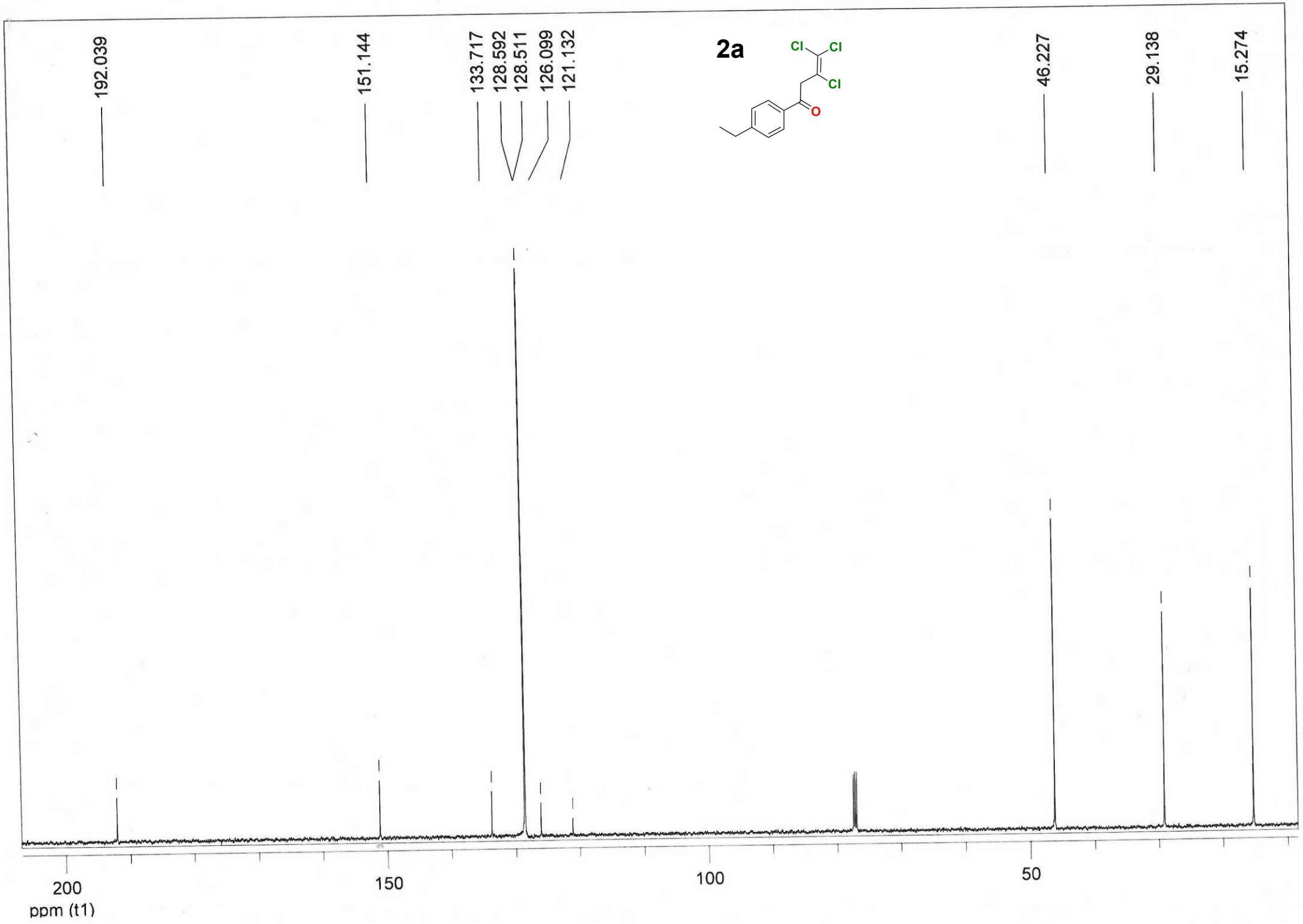
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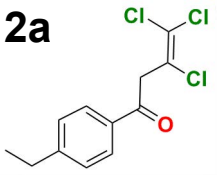

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Comment			

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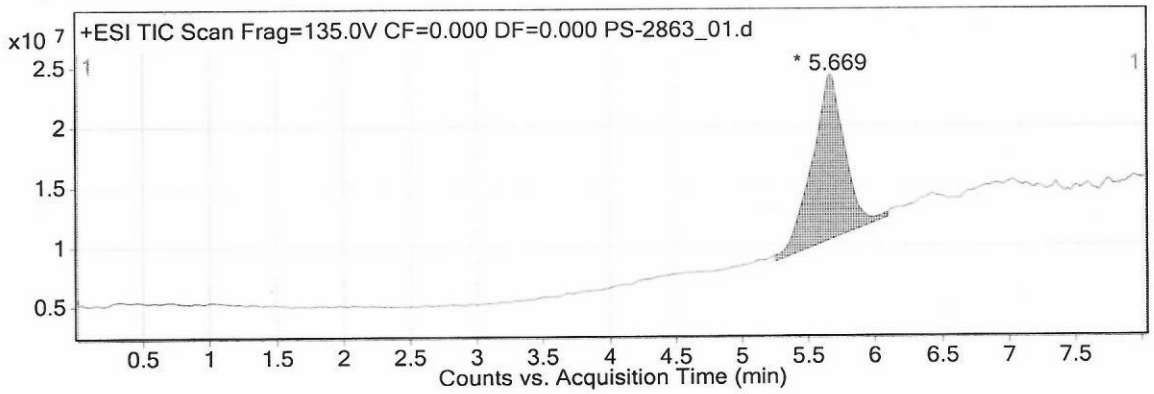

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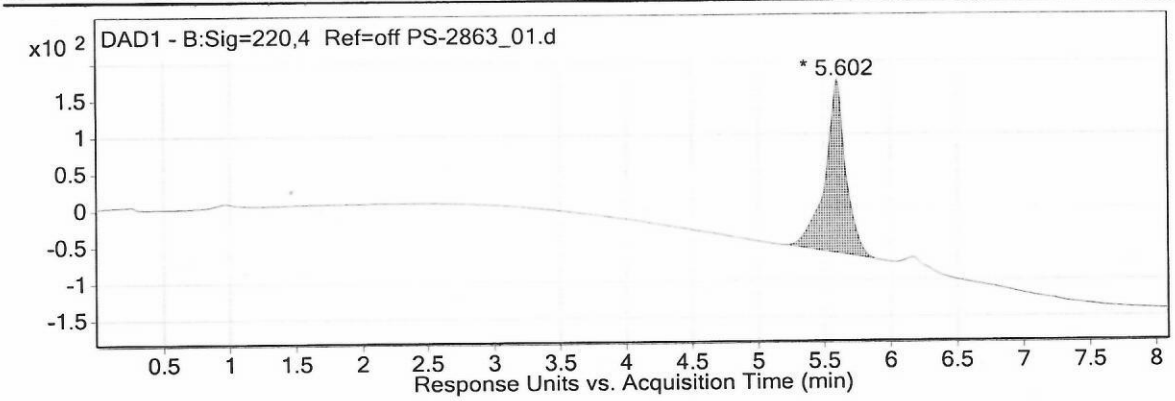

Integration Peak List

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User Spectra

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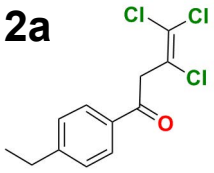
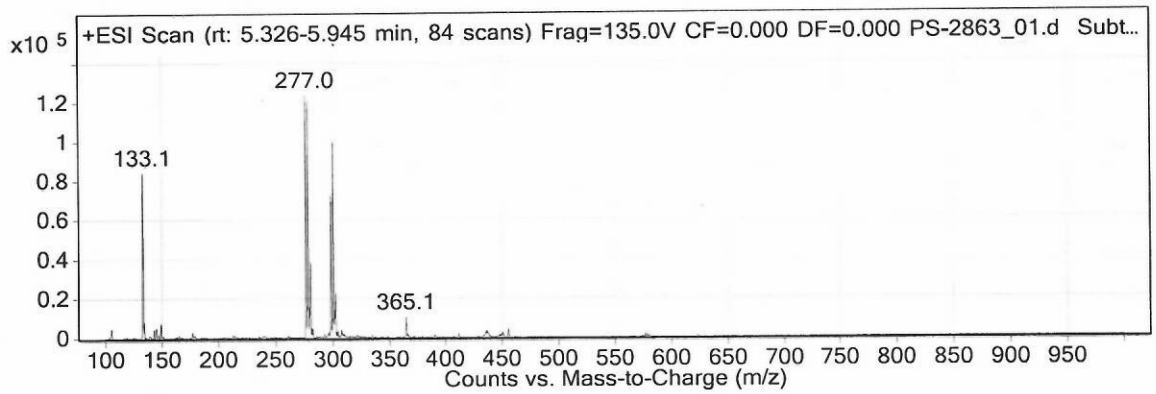


Qualitative Analysis Report



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Spectrum Source

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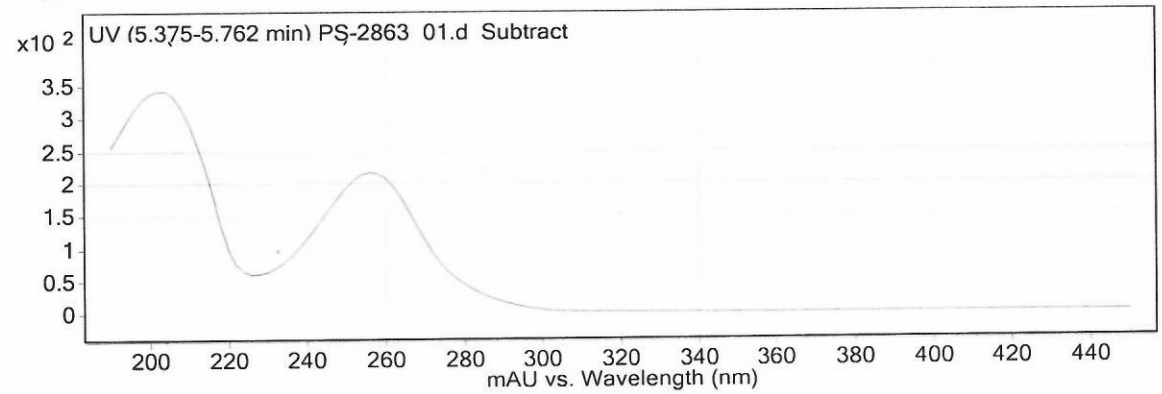

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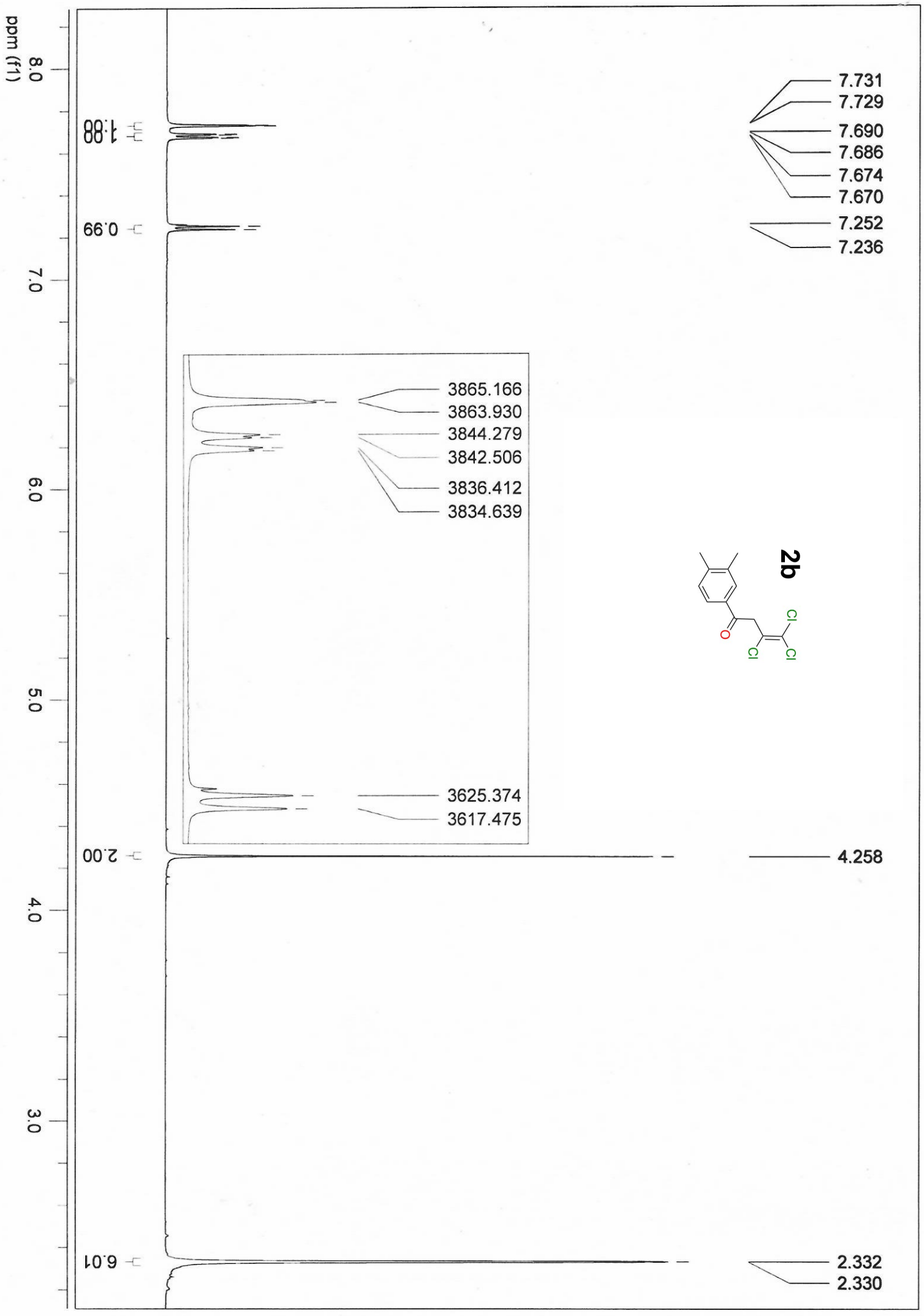
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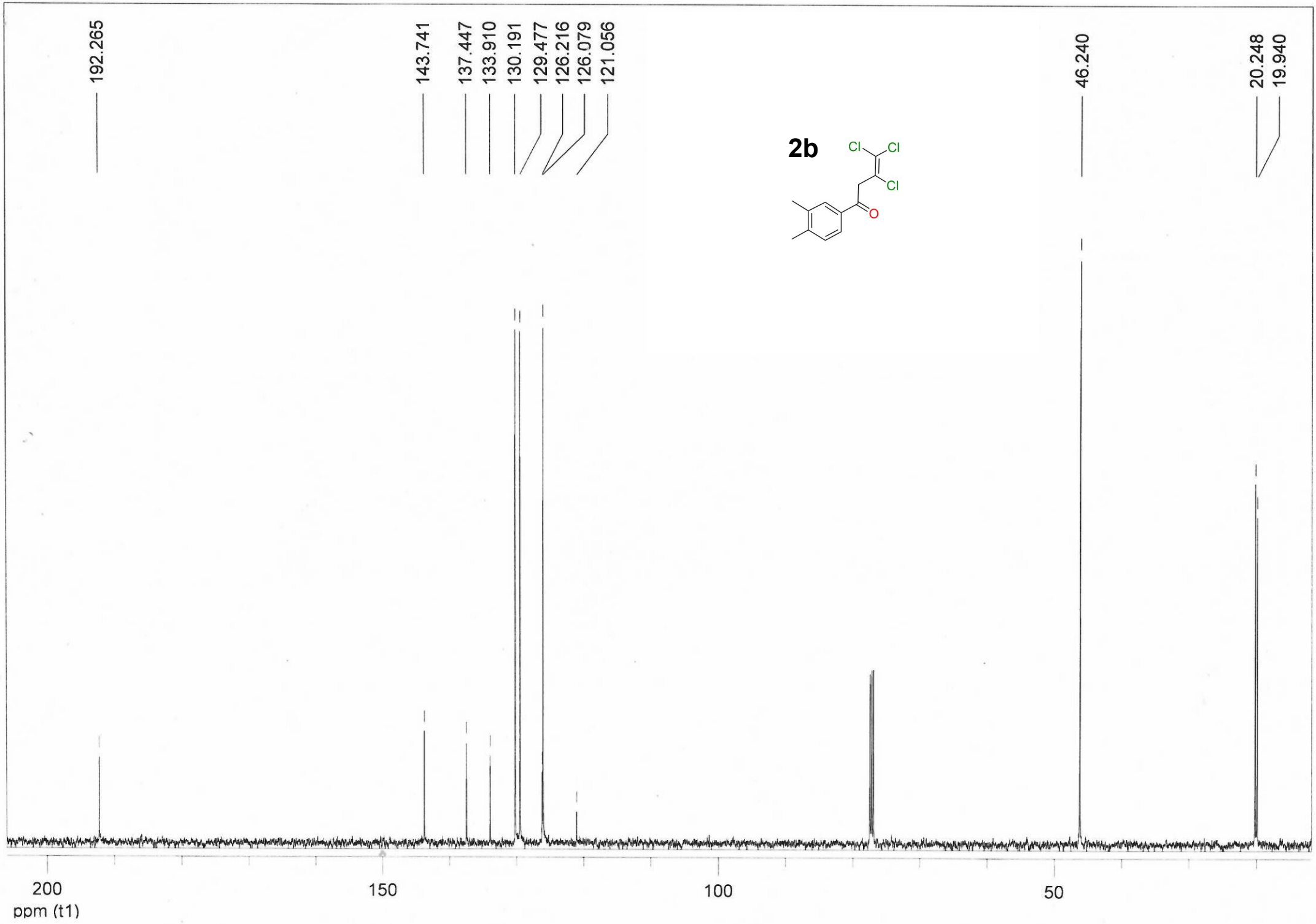
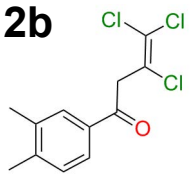
Cl
Cl

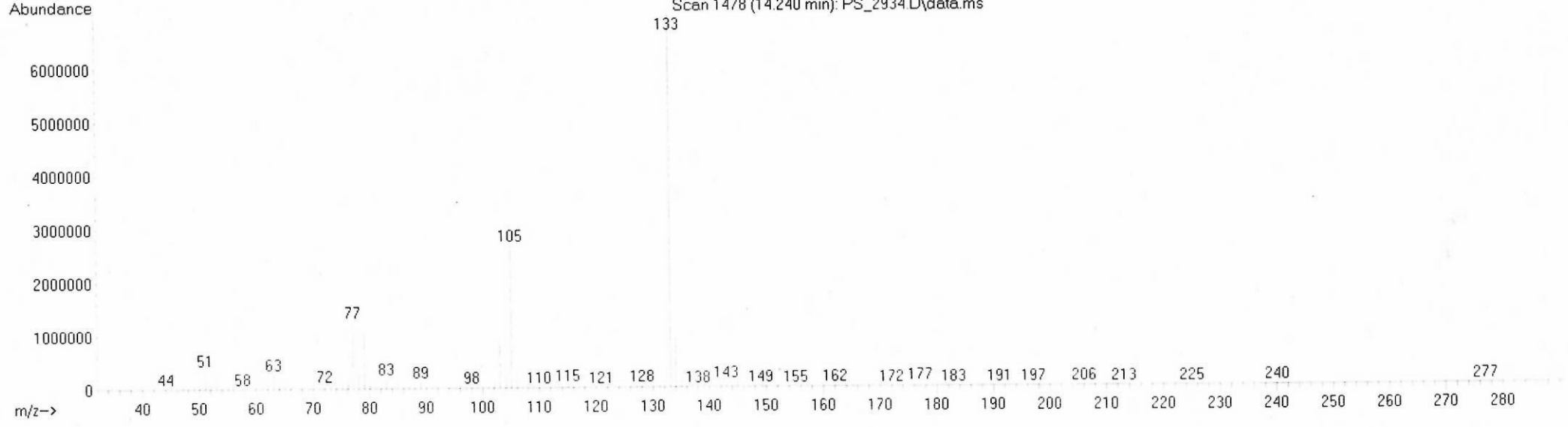

192.265
143.741
137.447
133.910
130.191
129.477
126.216
126.079
121.056
46.240
20.248
19.940
2b
Cl
Cl
Cl
O
200
150
100
50
ppm (t1)

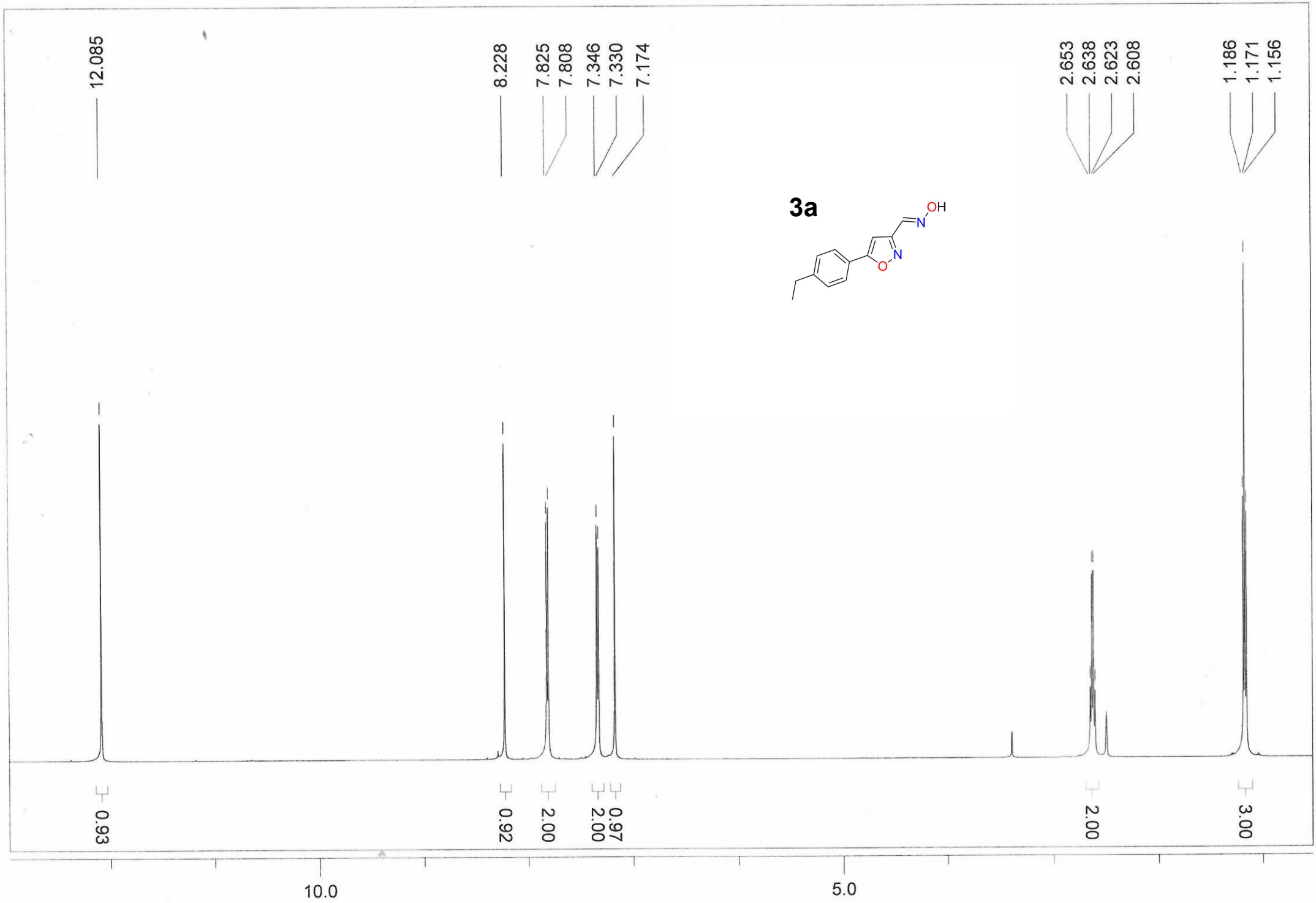
File :D:\msdchem\1\DATA\PV2_0613\PS_2934.D
Operator :
Acquired : 13 Jun 2022 11:55 am using AcqMethod BA_1SL.M
Instrument : Instrument #1
Sample Name: PS-2934
Misc Info :
Vial Number: 2

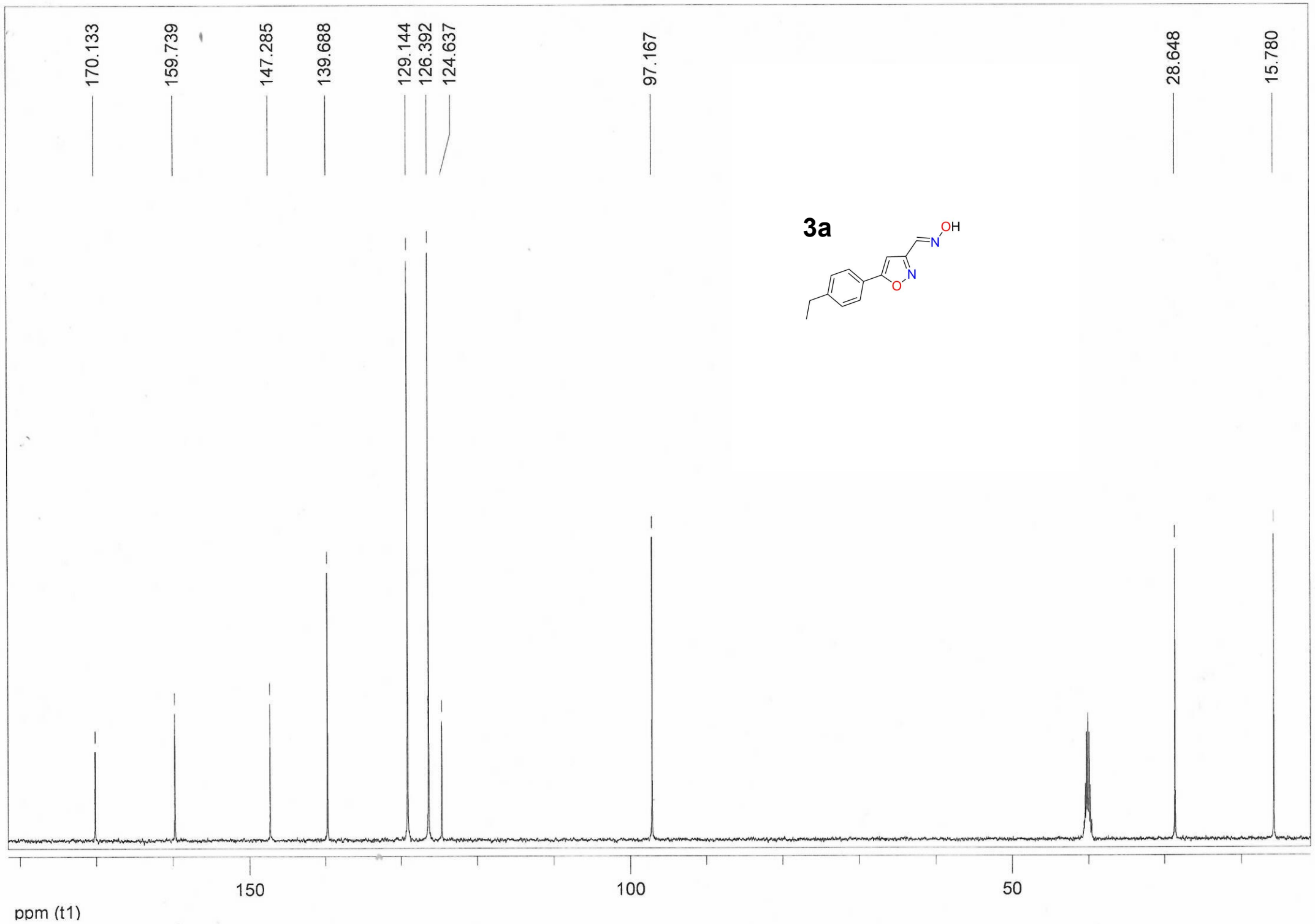
2b



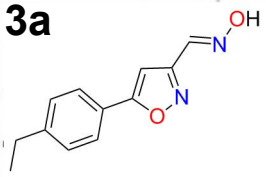
$[M]^+ = 276$




12.085
8.228
7.825
7.808
7.346
7.330
7.174
2.653
2.638
2.623
2.608
1.186
1.171
1.156
3a
OH
N
O
N
0.93
0.92
2.00
2.00
0.97
2.00
3.00
10.0
5.0
ppm (t1)


170.133
159.739
147.285
139.688
129.144
126.392
124.637
97.167
28.648
15.780
3a
OH
N
O
N
150
100
50
ppm (t1)

3a



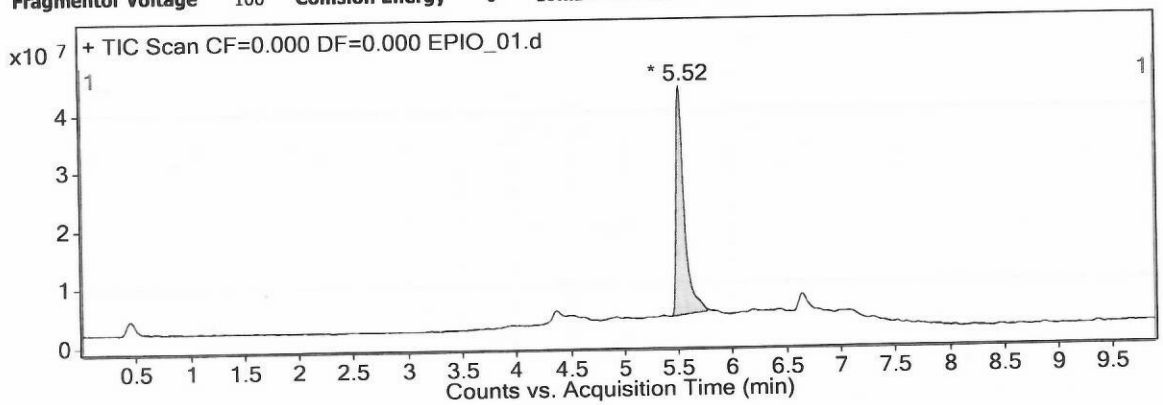
Qualitative Analysis Report

Data Filename	EPIO_01.d	**Sample Name**	EPIO
Sample Type	Sample	**Position**	Vial 3
Instrument Name	Instrument 1	**User Name**	
Acq Method	All_2021_kol 1-6.m	**Acquired Time**	3/21/2024 12:25:20 PM
IRM Calibration Status	Not Applicable	**DA Method**	QualDAMethod.m
Comment			

Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition SW Version**	6400 Series Triple Quadrupole 10.0 (127)

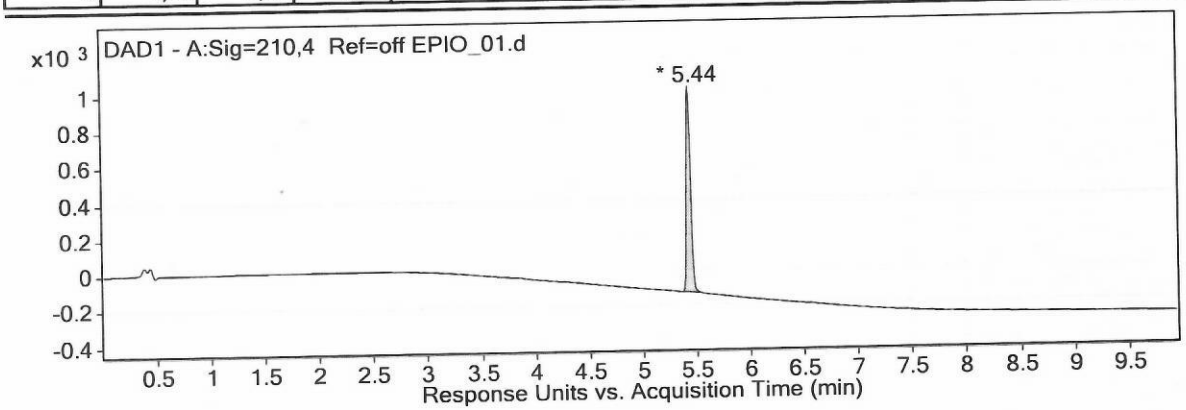
User Chromatograms

Fragmentor Voltage 100 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	5,43	5,52	5,76	39437606,7	206963986,2	100



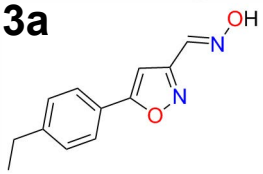
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	5,34	5,44	5,54	1149,99	3224,94	100

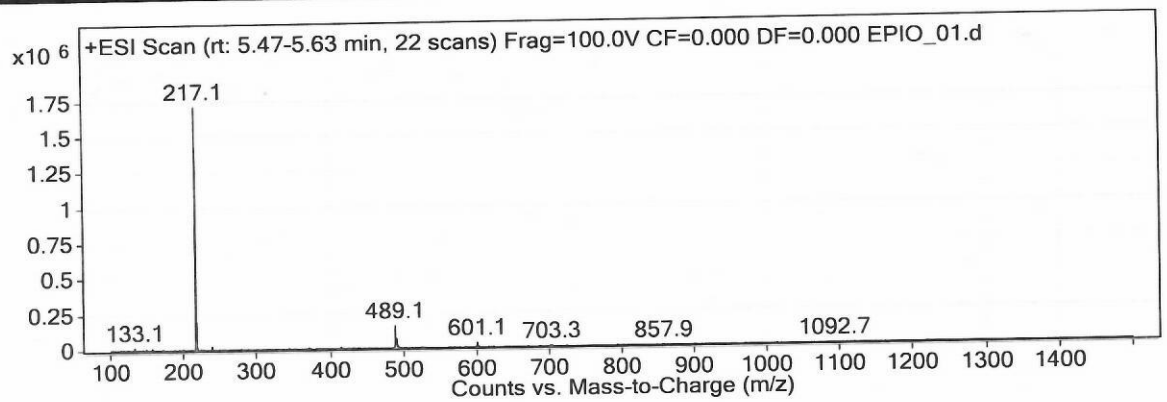
User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan"	100	0	ESI

3a



Qualitative Analysis Report

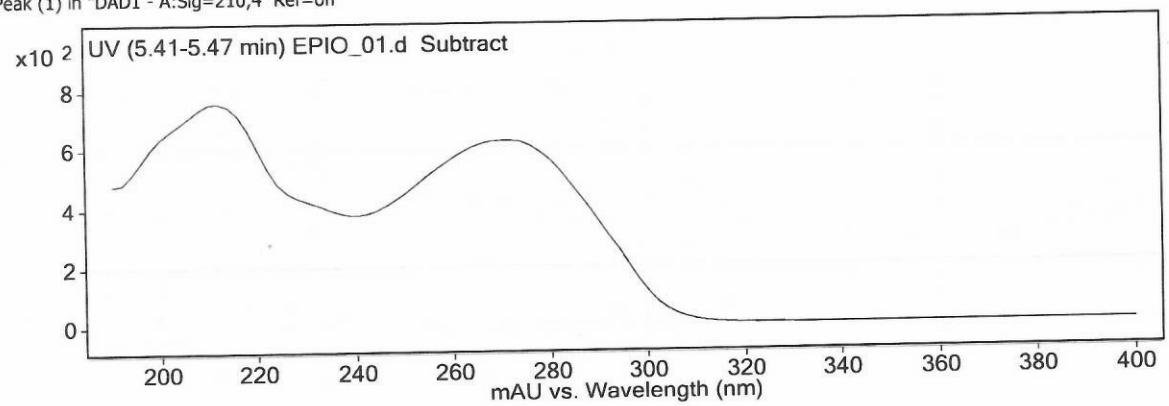


Peak List

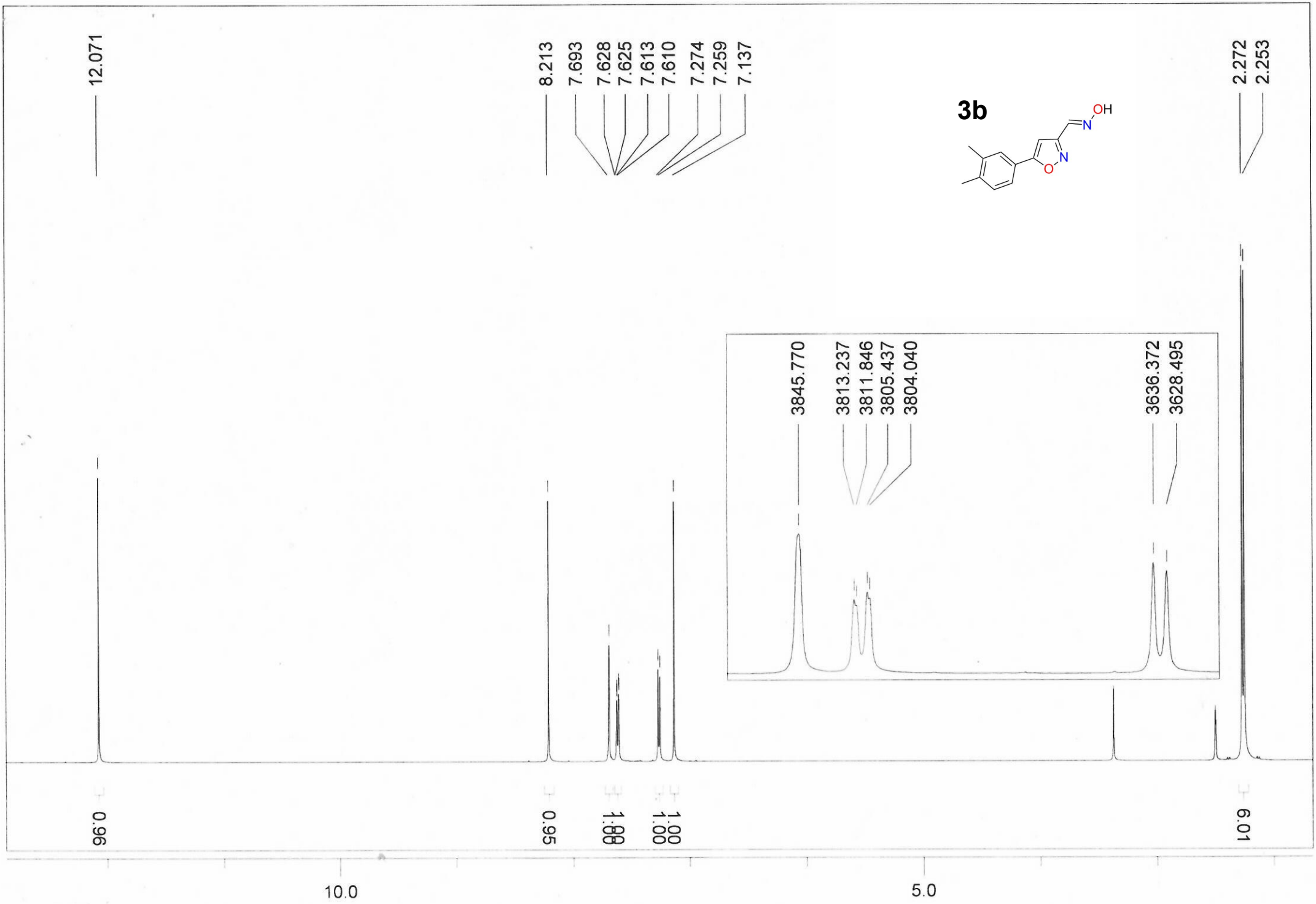
m/z	z	Abund	
133.1	1	21265.13	
217.1	1	1718778.5	[M+H]+
218.1	1	198049.86	
219.1	1	19869.02	
239.1		26483.62	
489.1		158748.41	
490.2		44358.15	
491.1	1	68609.38	
492.1	1	20025.01	
601.1		33846.95	

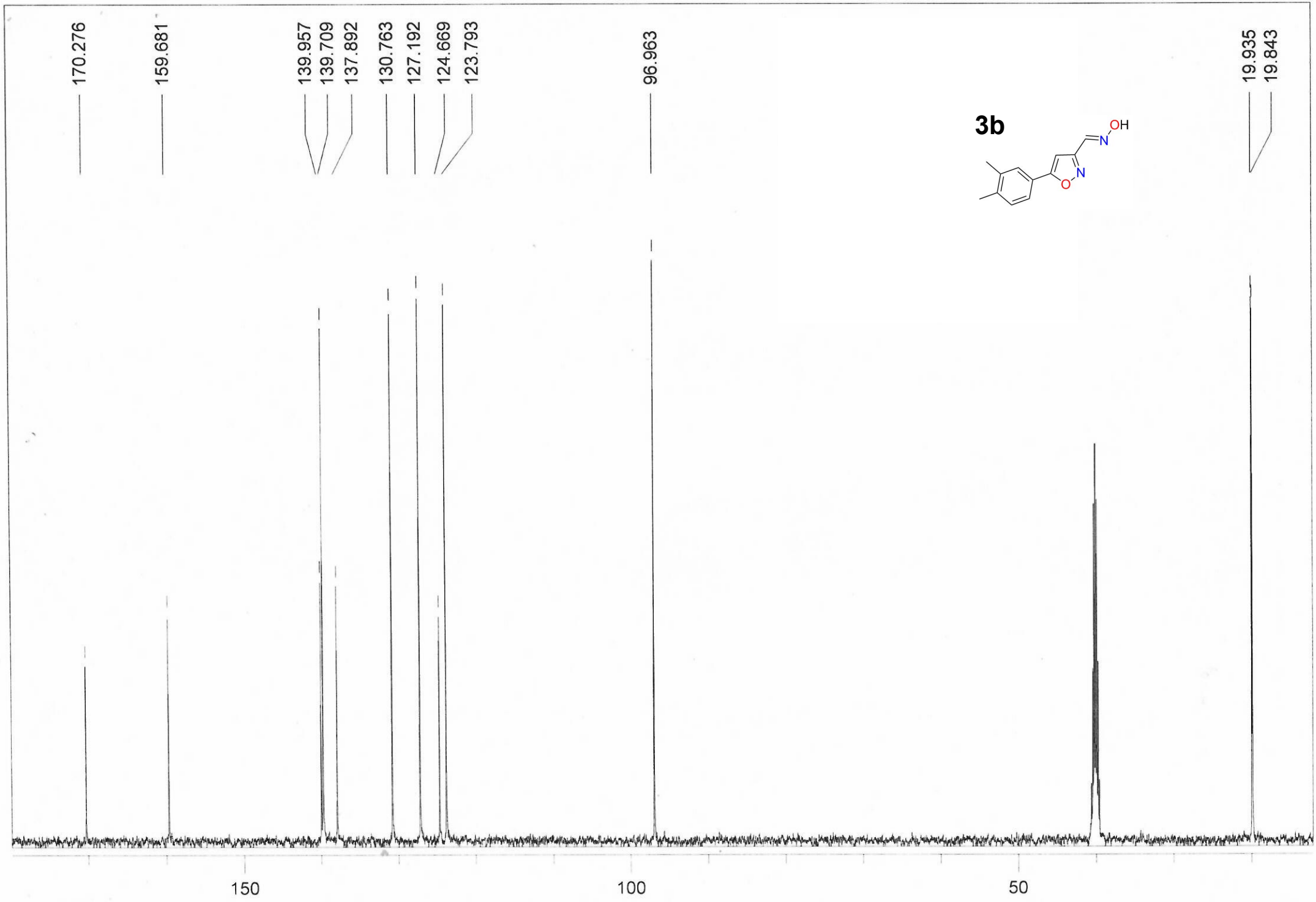
Spectrum Source

Peak (1) in "DAD1 - A:Sig=210,4 Ref=off"

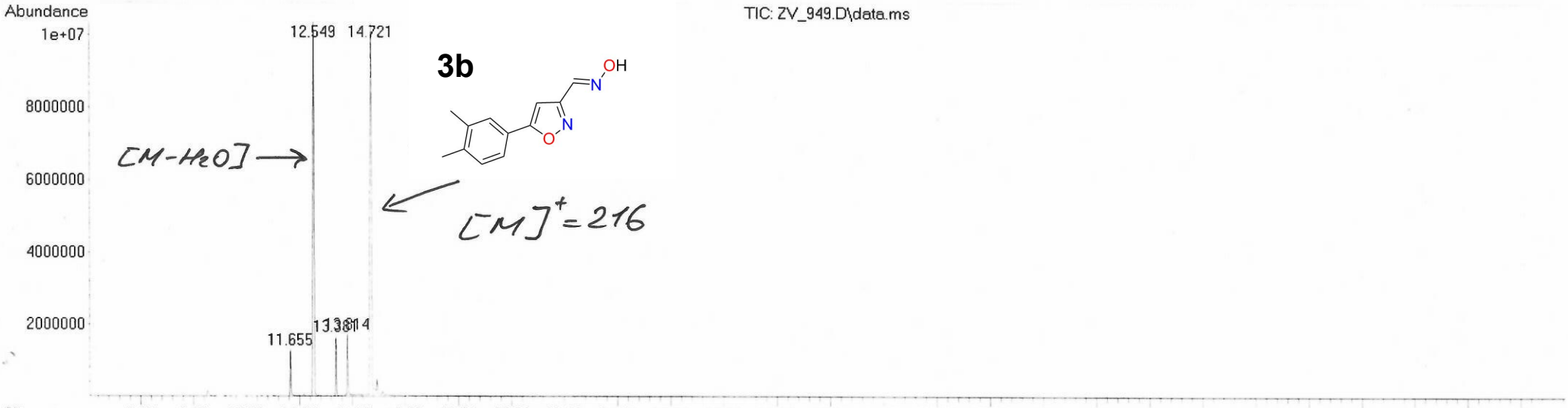


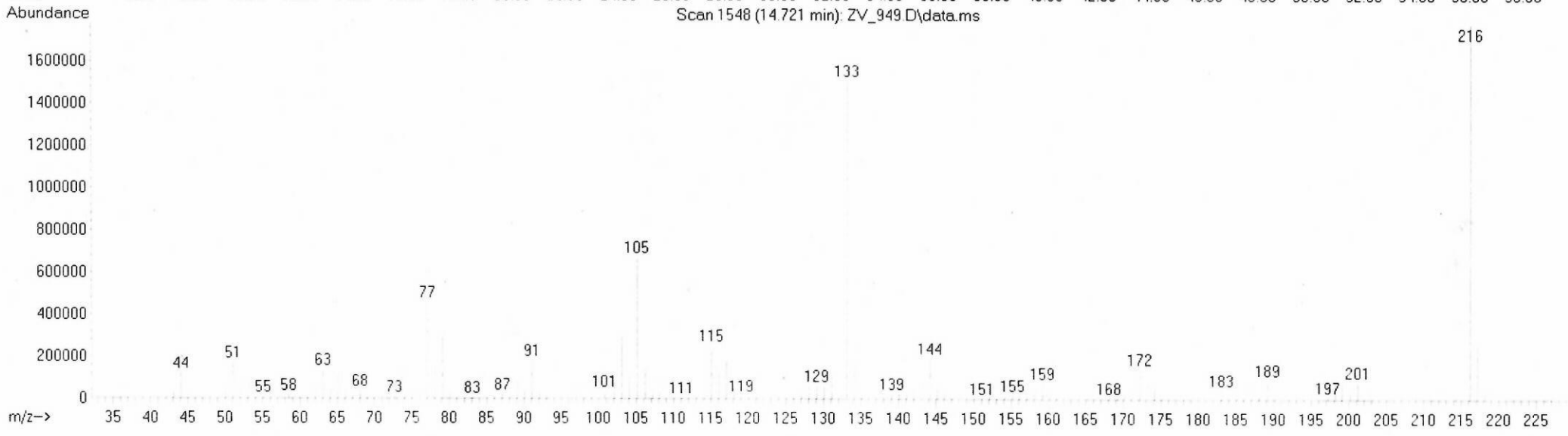
--- End Of Report ---

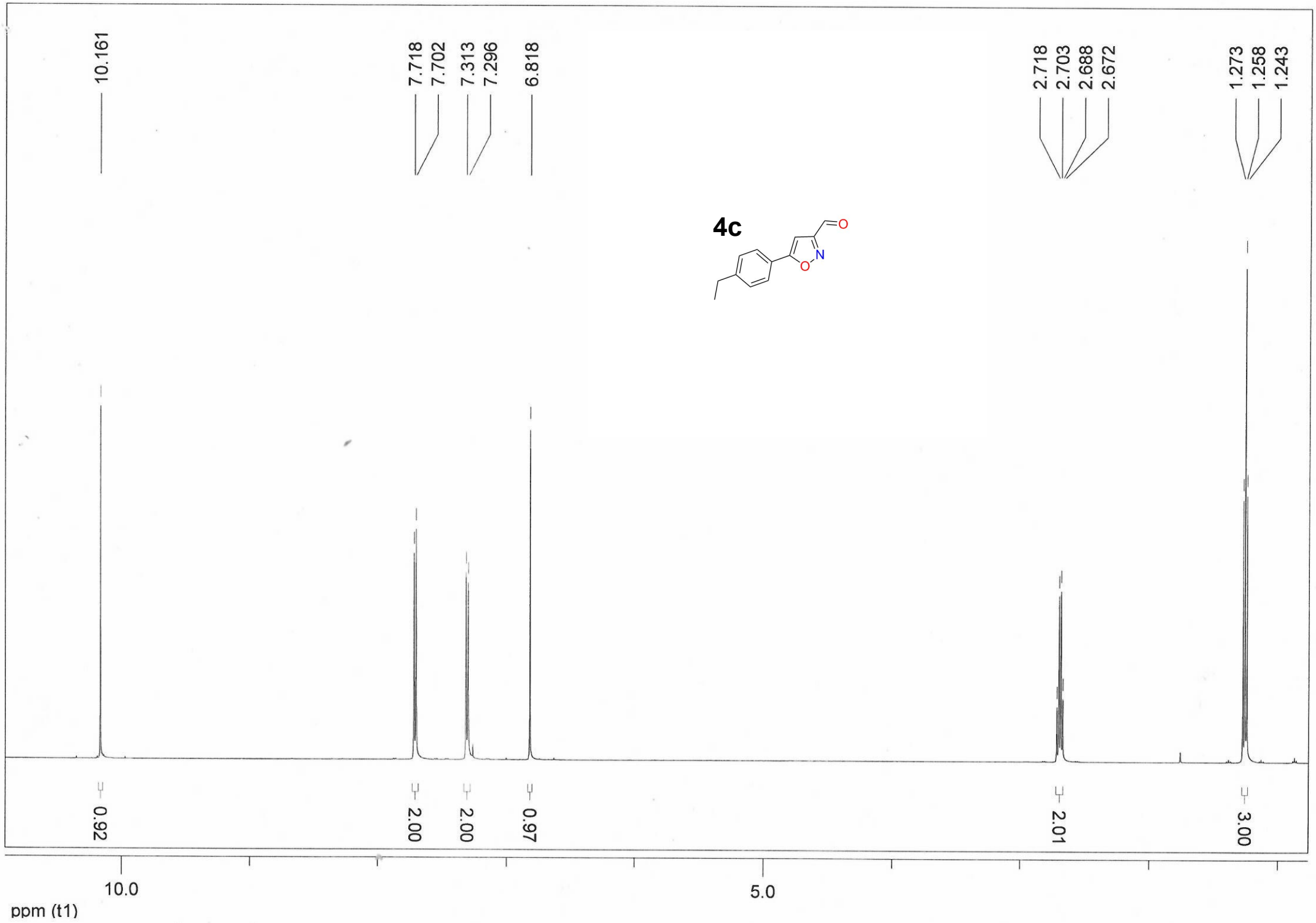

12.071
8.213
7.693
7.628
7.625
7.613
7.610
7.274
7.259
7.137
2.272
2.253
3b
OH
N
O
N
3845.770
3813.237
3811.846
3805.437
3804.040
3636.372
3628.495
0.96
0.95
1.00
1.00
6.01
10.0
5.0
ppm (t1)

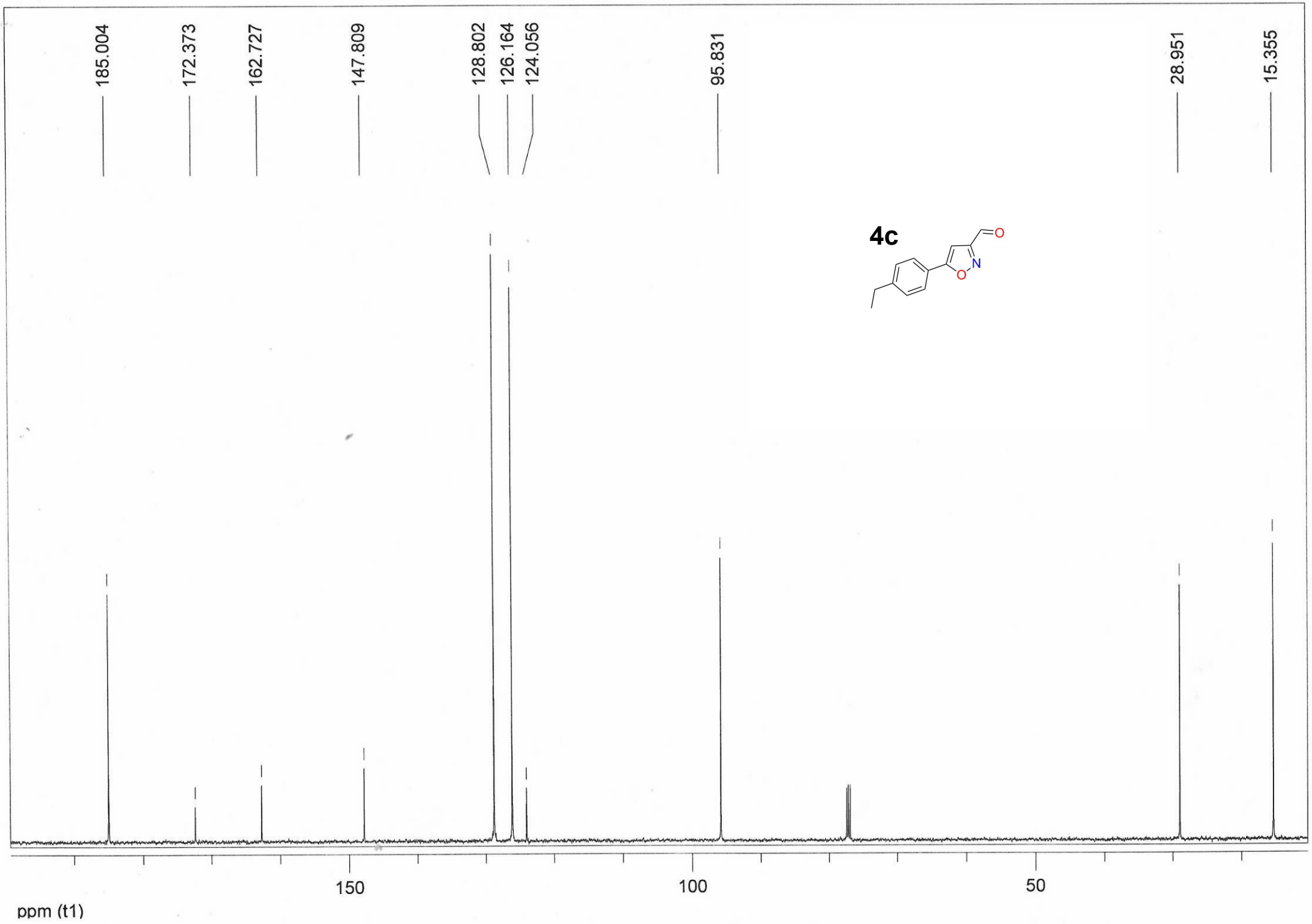

170.276
159.681
139.957
139.709
137.892
130.763
127.192
124.669
123.793
96.963
19.935
19.843
3b
OH
N
O
N
150
100
50
ppm (t1)

File :D:\msdchem\1\DATA\PV2_0721\ZV_949.D
Operator :
Acquired : 20 Jul 2022 9:47 pm using AcqMethod BA_1SL.M
Instrument : Instrument #1
Sample Name: ZV-949
Misc Info :
Vial Number: 8

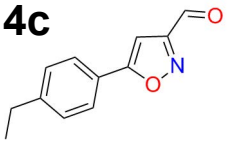





10.161
7.718
7.702
7.313
7.296
6.818
2.718
2.703
2.688
2.672
1.273
1.258
1.243
4c
O
N
O
0.92
2.00
2.00
0.97
2.01
3.00
10.0
5.0
ppm (t1)


185.004
172.373
162.727
147.809
128.802
126.164
124.056
95.831
28.951
15.355
4c
O
N
O
150
100
50
ppm (t1)

4c



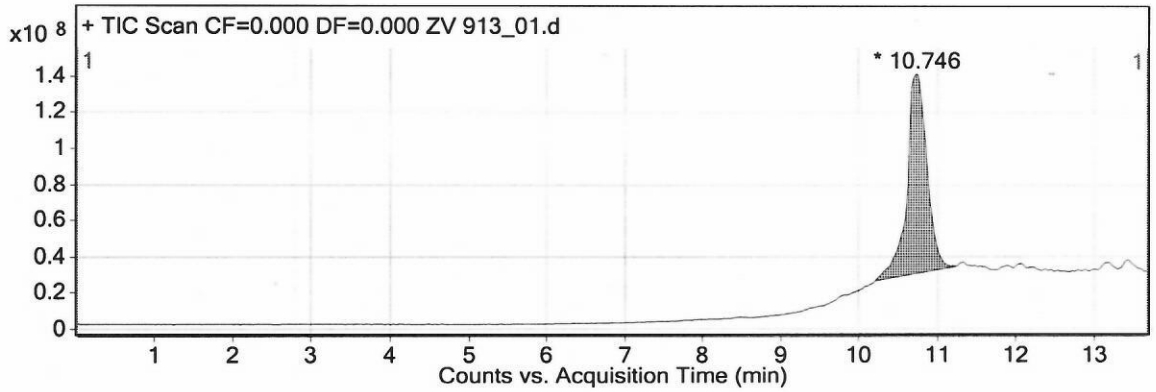
Qualitative Analysis Report

Data Filename	ZV 913_01.d	**Sample Name**	ZV 913
Sample Type	Sample	**Position**	Vial 2
Instrument Name	Instrument 1	**User Name**	
Acq Method	All_2021_kol 1-6.m	**Acquired Time**	12/1/2021 1:45:48 PM
IRM Calibration Status	Not Applicable	**DA Method**	ChromPeakSurvey-Default.m
Comment			

Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition SW Version**	6400 Series Triple Quadrupole 10.0 (127)

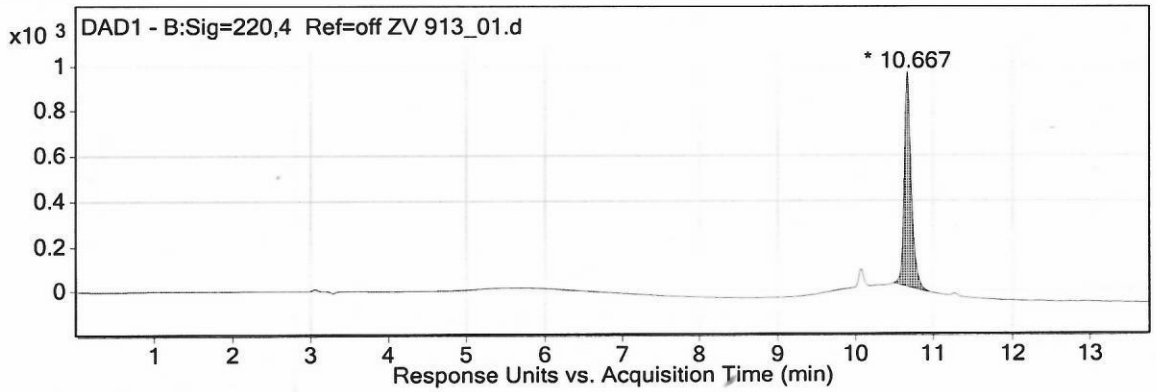
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	10,217	10,746	11,243	110618059	1972444149	100



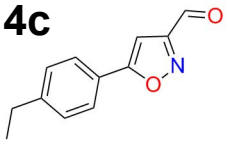
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	10,494	10,667	11,02	953,86	6079,46	100

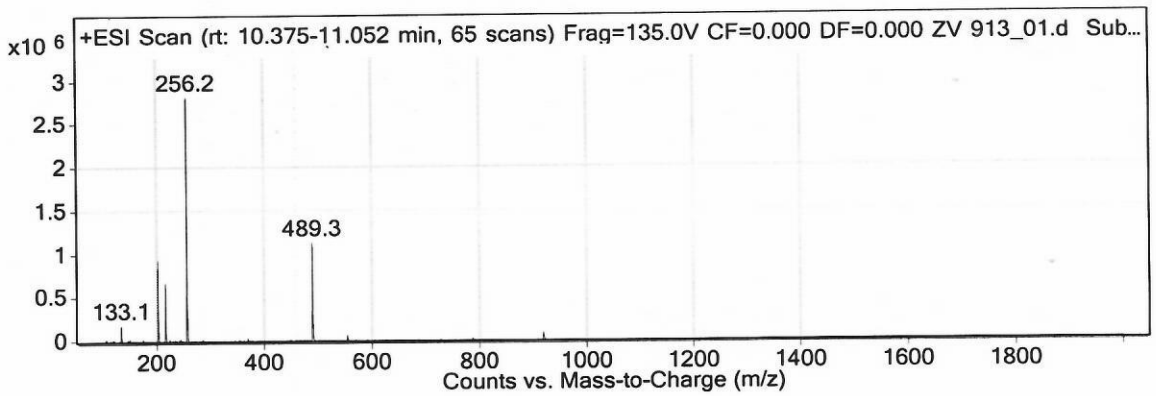
User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan"	135	0	ESI

4c



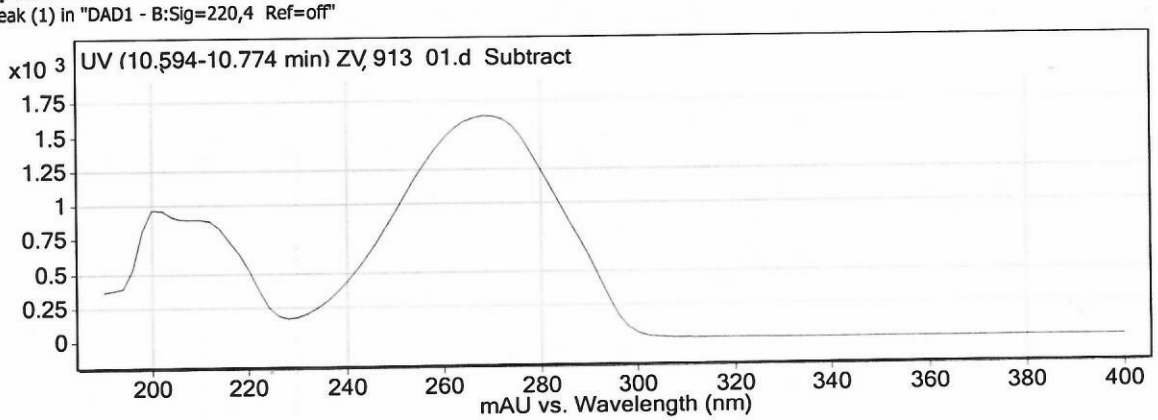
Qualitative Analysis Report



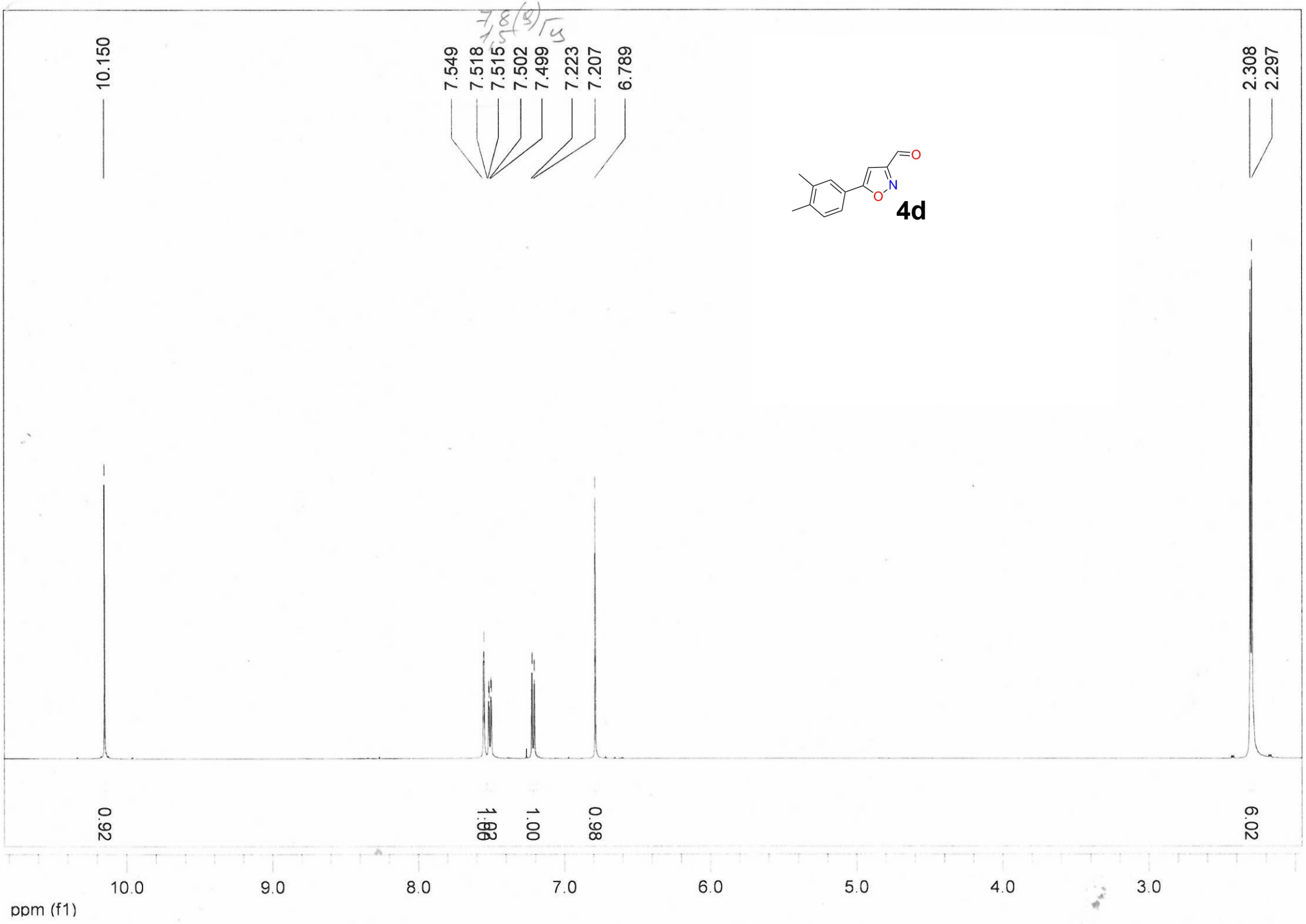
Peak List

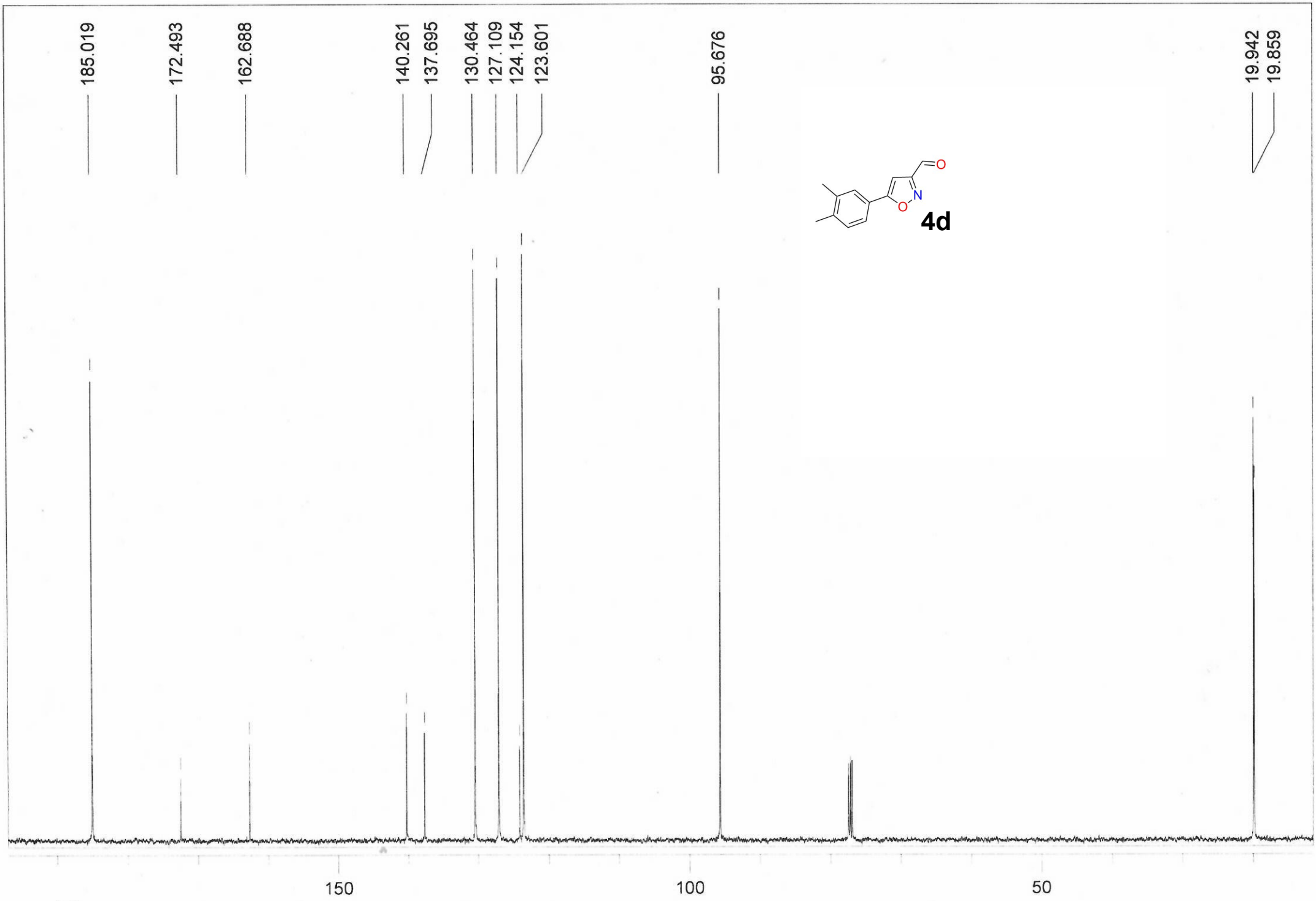
m/z	z	Abund
133.1		175774.55
202.1		928609.69
216.1		663932.06
256.2		2811573.75
257.1		358430.03
489.3	1	1123075.63
490.3	1	298654.78

Spectrum Source
Peak (1) in "DAD1 - B:Sig=220,4 Ref=off"

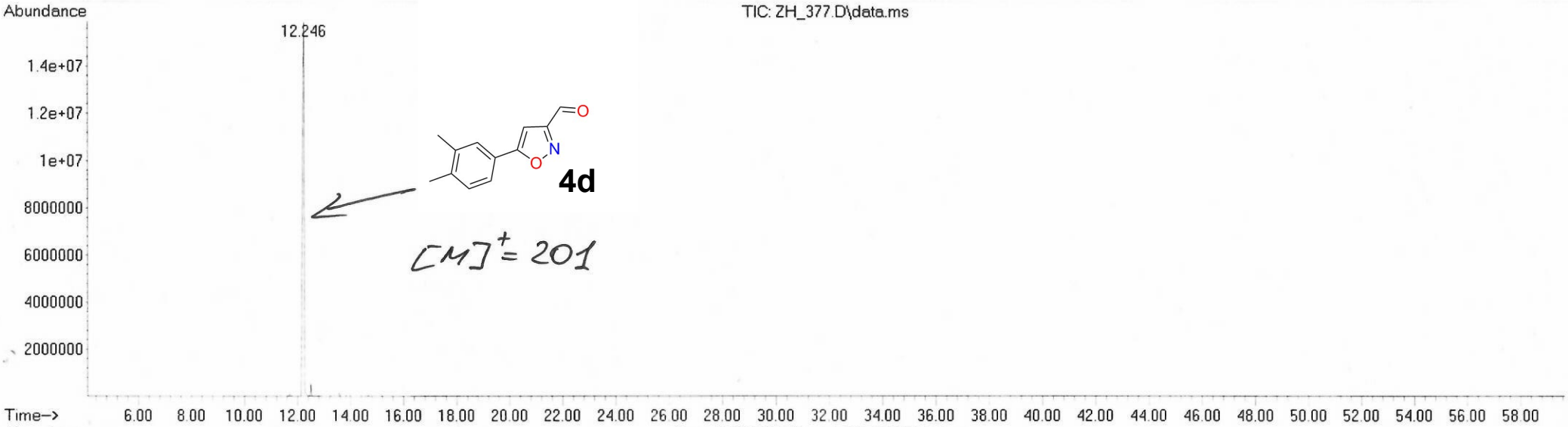


--- End Of Report ---


10.150
7.549
7.518
7.515
7.502
7.499
7.223
7.207
6.789
2.308
2.297
O
N
O
4d
0.92
1.00
1.00
0.98
6.02
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
ppm (f1)

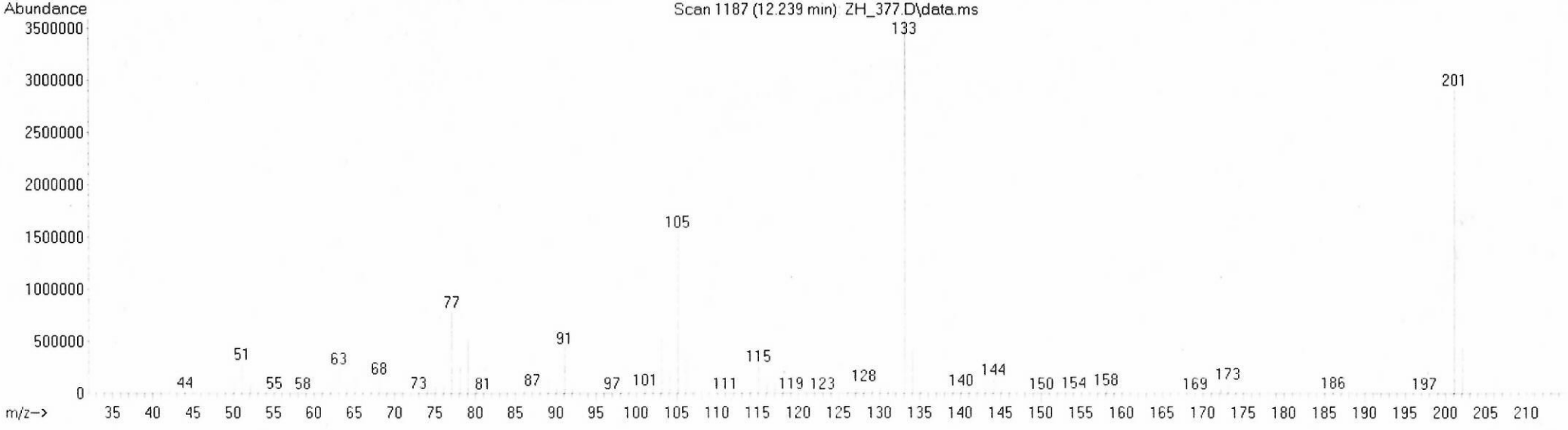

185.019
172.493
162.688
140.261
137.695
130.464
127.109
124.154
123.601
95.676
19.942
19.859
O
N
O
4d
150
100
50
ppm (t1)

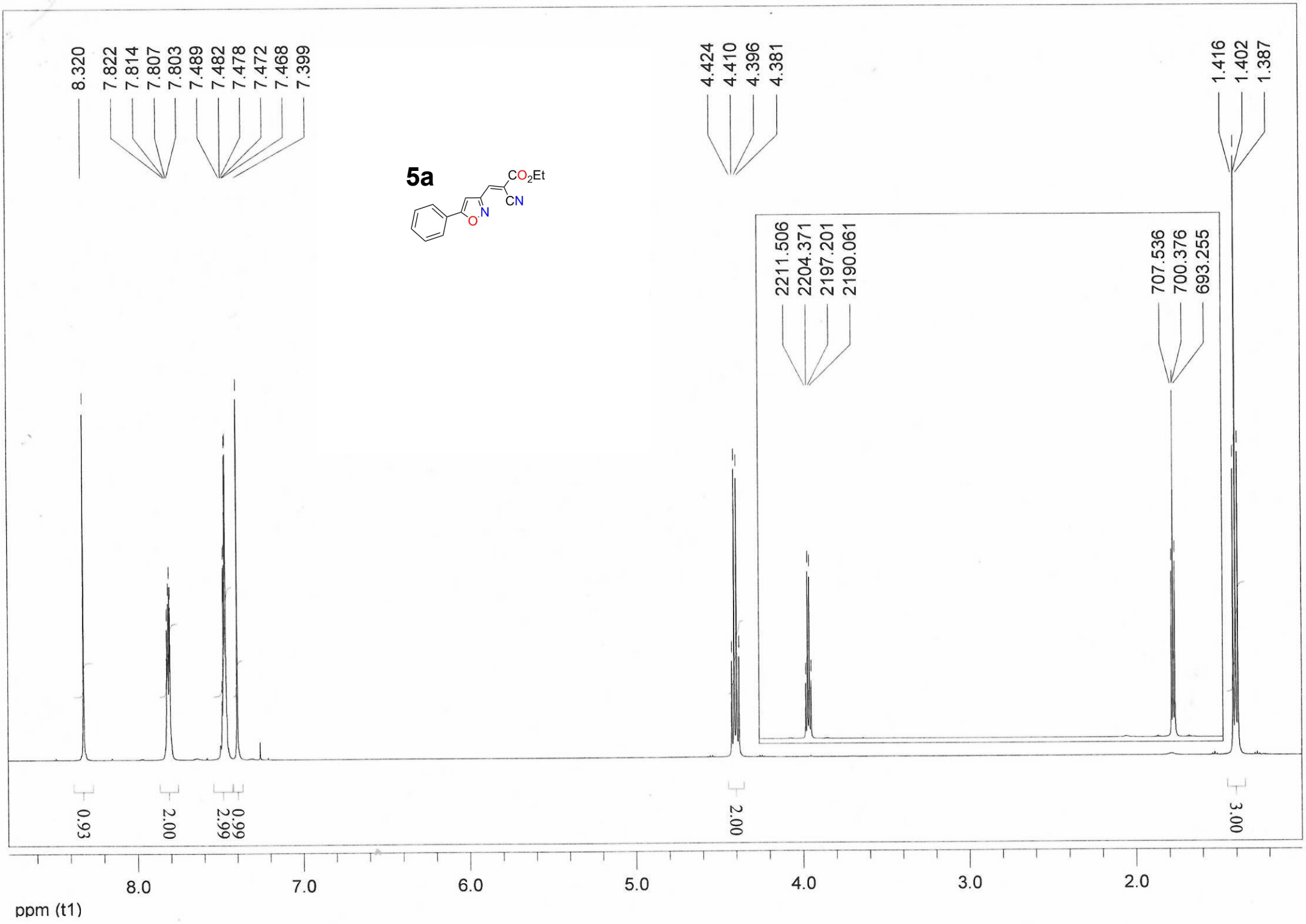
File :D:\msdchem\1\DATA\PV2_0721\ZH_377.D
Operator :
Acquired : 20 Jul 2022 10:53 pm using AcqMethod BA_1SL.M
Instrument : Instrument #1
Sample Name: ZH-377
Misc Info :
Vial Number: 1

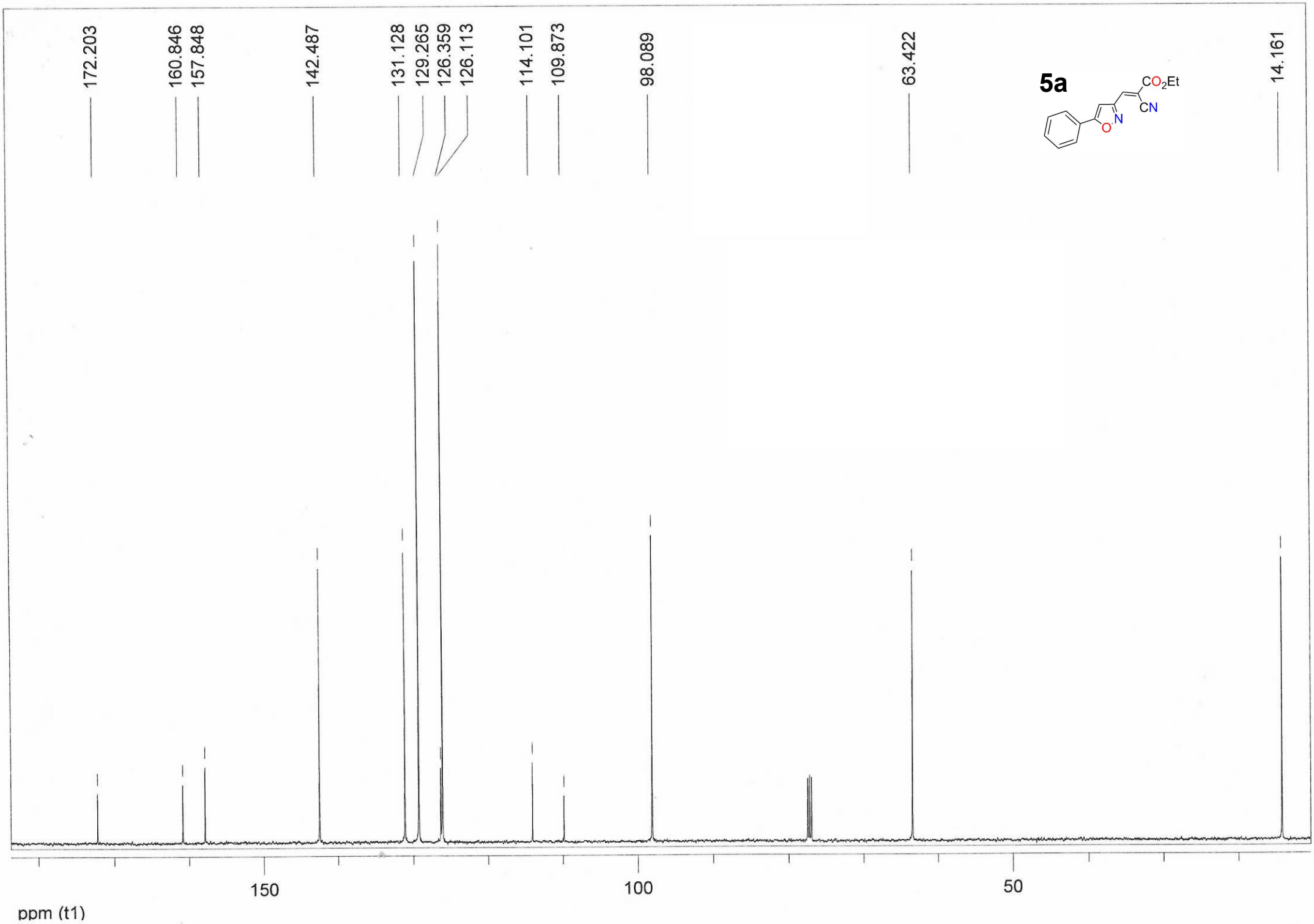


4d

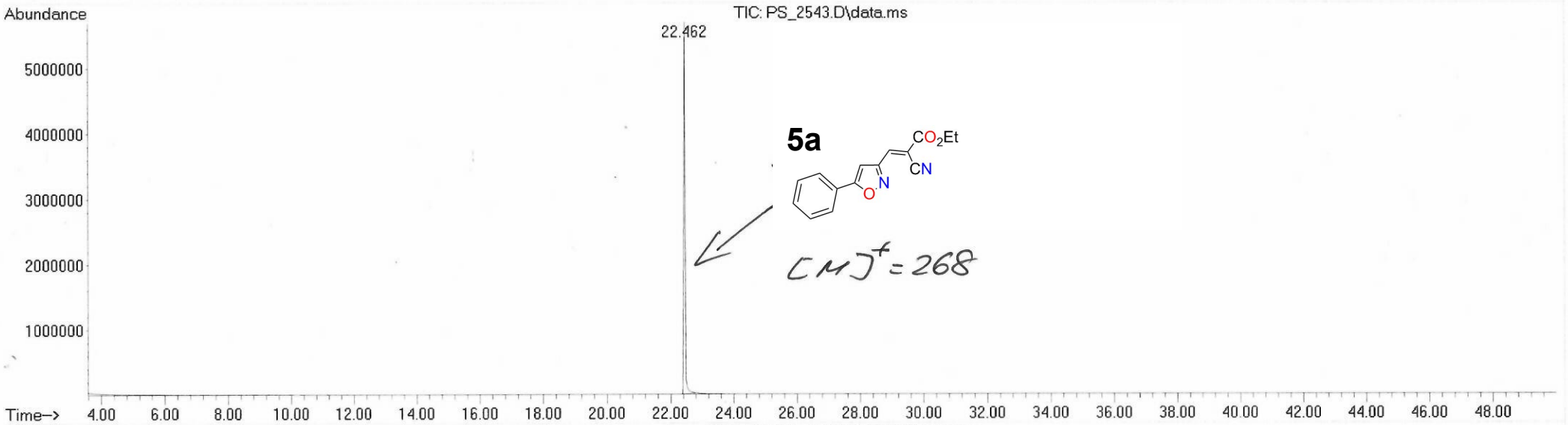
$[M]^+ = 201$




8.320
7.822
7.814
7.807
7.803
7.489
7.482
7.478
7.472
7.468
7.399
4.424
4.410
4.396
4.381
1.416
1.402
1.387
5a
CO2Et
CN
O
N
2211.506
2204.371
2197.201
2190.061
707.536
700.376
693.255
0.93
2.00
2.99
0.99
2.00
3.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
ppm (t1)

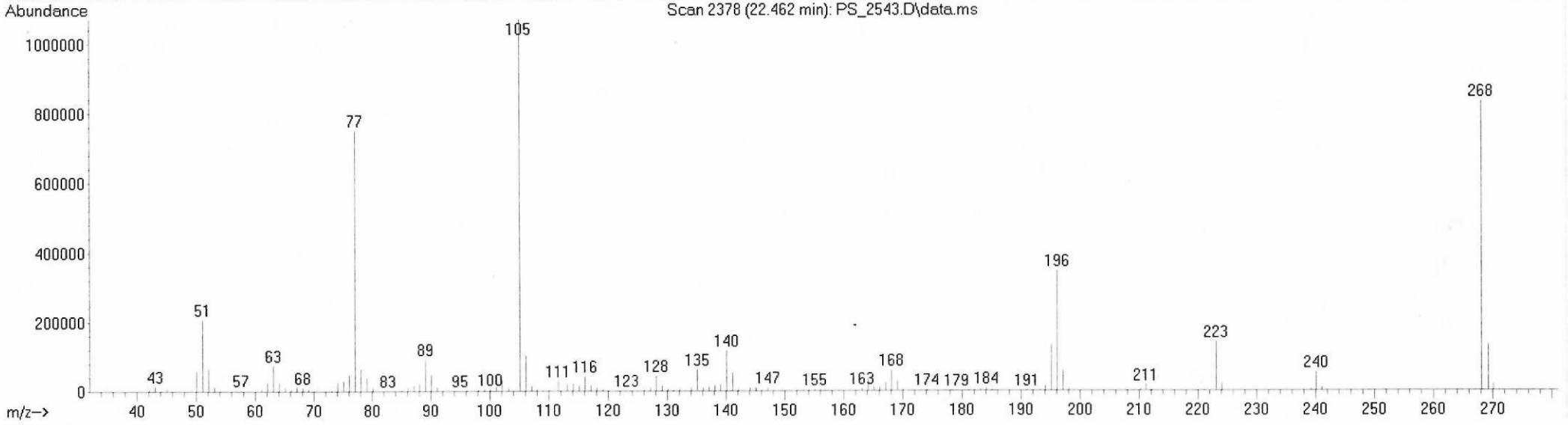

172.203
160.846
157.848
142.487
131.128
129.265
126.359
126.113
114.101
109.873
98.089
63.422
14.161
5a
CO2Et
CN
O
N
150
100
50
ppm (t1)

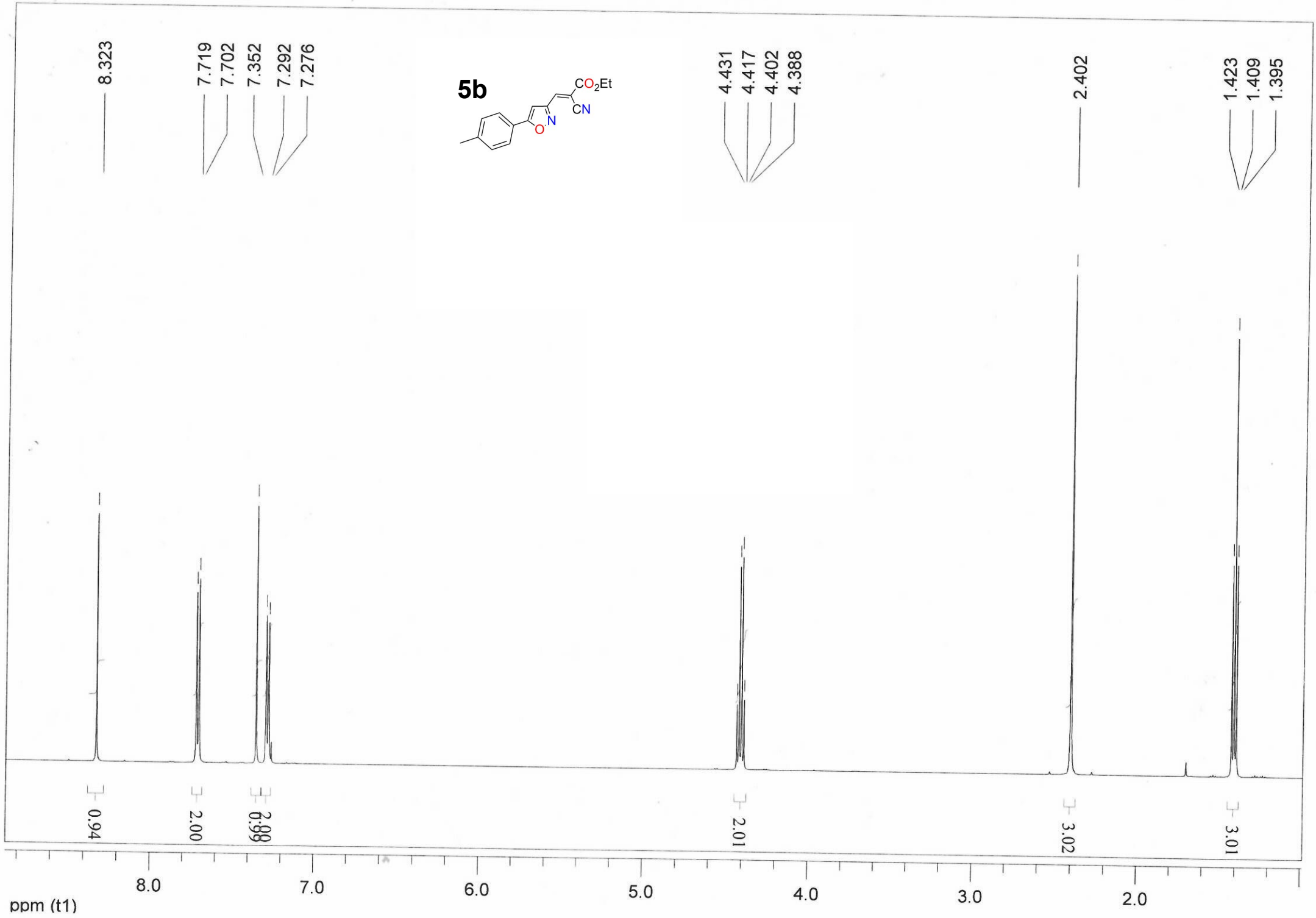
File :C:\msdchem\1\DATA\PV8_0313\PS_2543.D
Operator :
Acquired : 13 Mar 2018 3:18 pm using AcqMethod PVI_1N.M
Instrument : Instrument #1
Sample Name: PS-2543
Misc Info :
Vial Number: 1

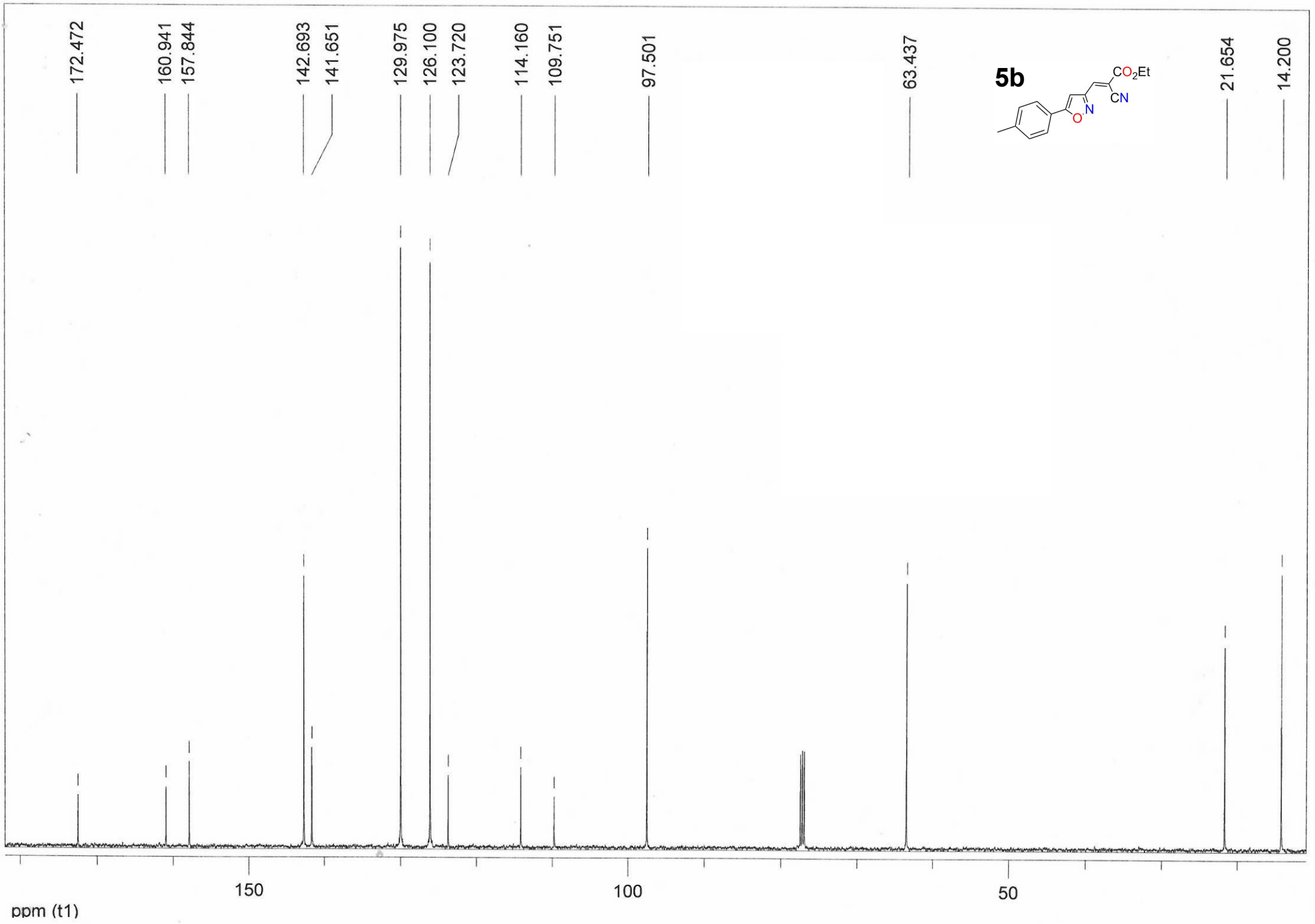


5a

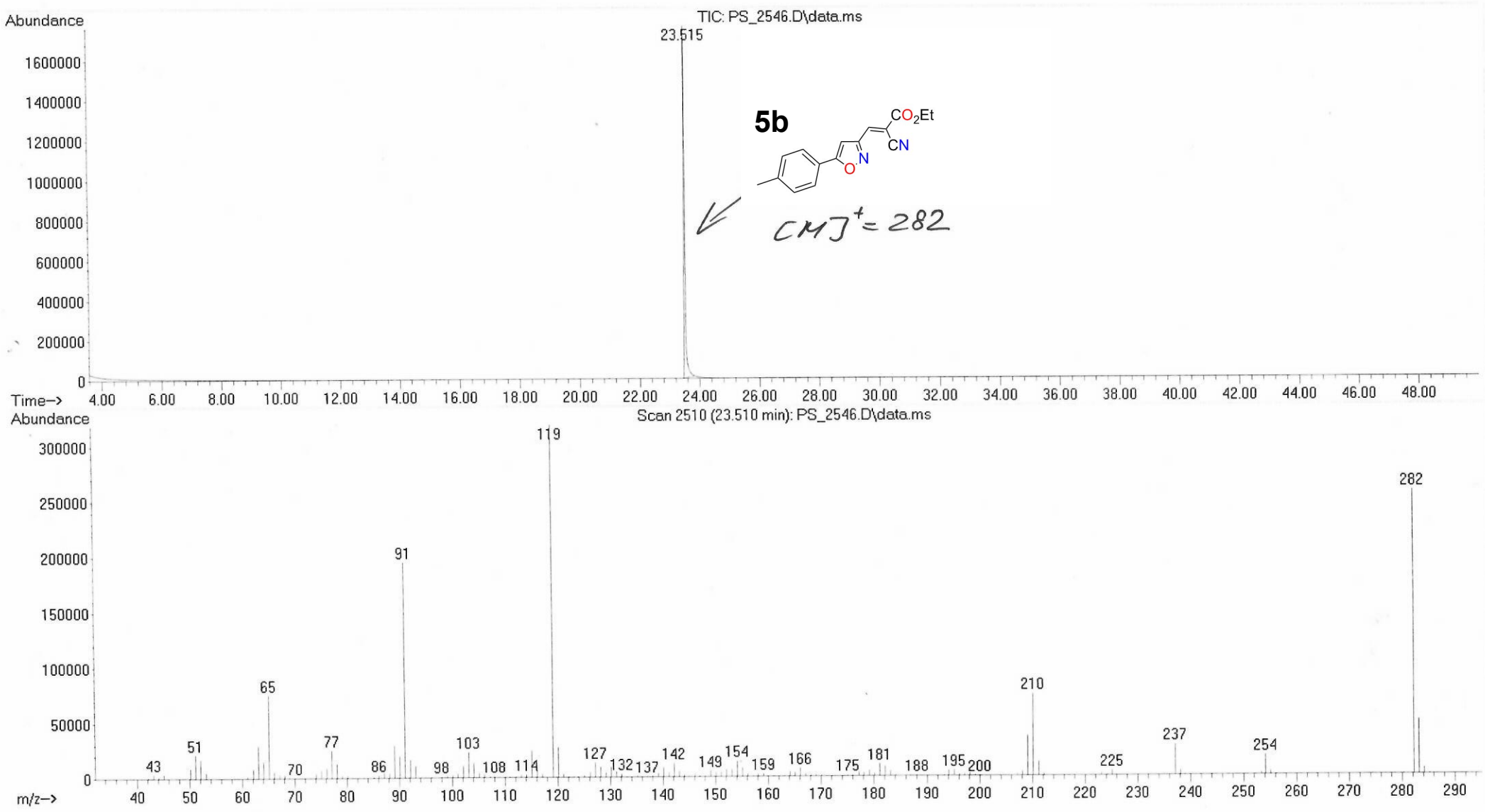
$[M]^+ = 268$




8.323
7.719
7.702
7.352
7.292
7.276
5b
CO2Et
CN
O
N
4.431
4.417
4.402
4.388
2.402
1.423
1.409
1.395
0.94
2.00
2.88
2.01
3.02
3.01
8.0
7.0
6.0
5.0
4.0
3.0
2.0
ppm (t1)

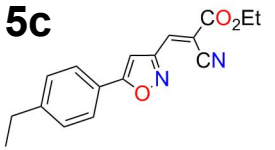

172.472
160.941
157.844
142.693
141.651
129.975
126.100
123.720
114.160
109.751
97.501
63.437
21.654
14.200
5b
CO2Et
CN
N
O
150
100
50
ppm (t1)

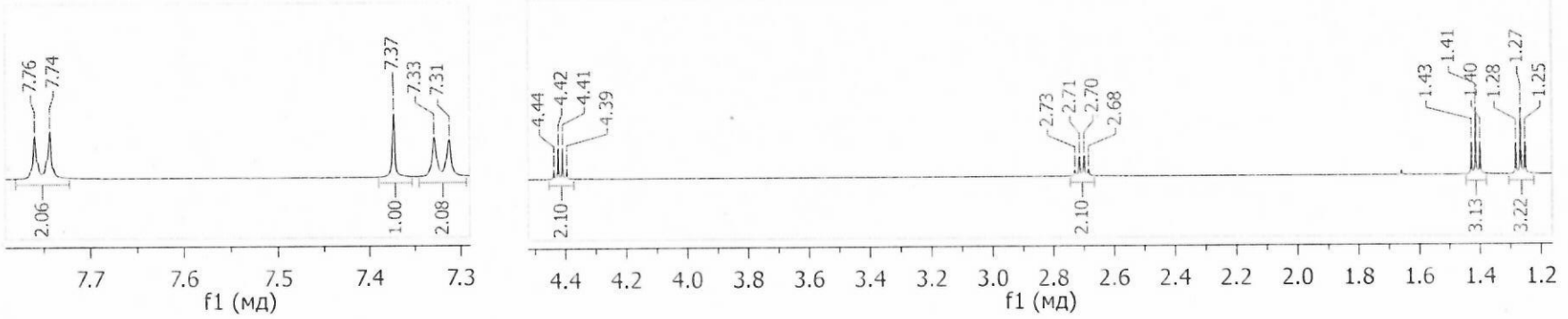
File :C:\msdchem\1\DATA\PV8_0313\PS_2546.D
Operator :
Acquired : 13 Mar 2018 5:16 pm using AcqMethod PVI_1N.M
Instrument : Instrument #1
Sample Name: PS-2546
Misc Info :
Vial Number: 3

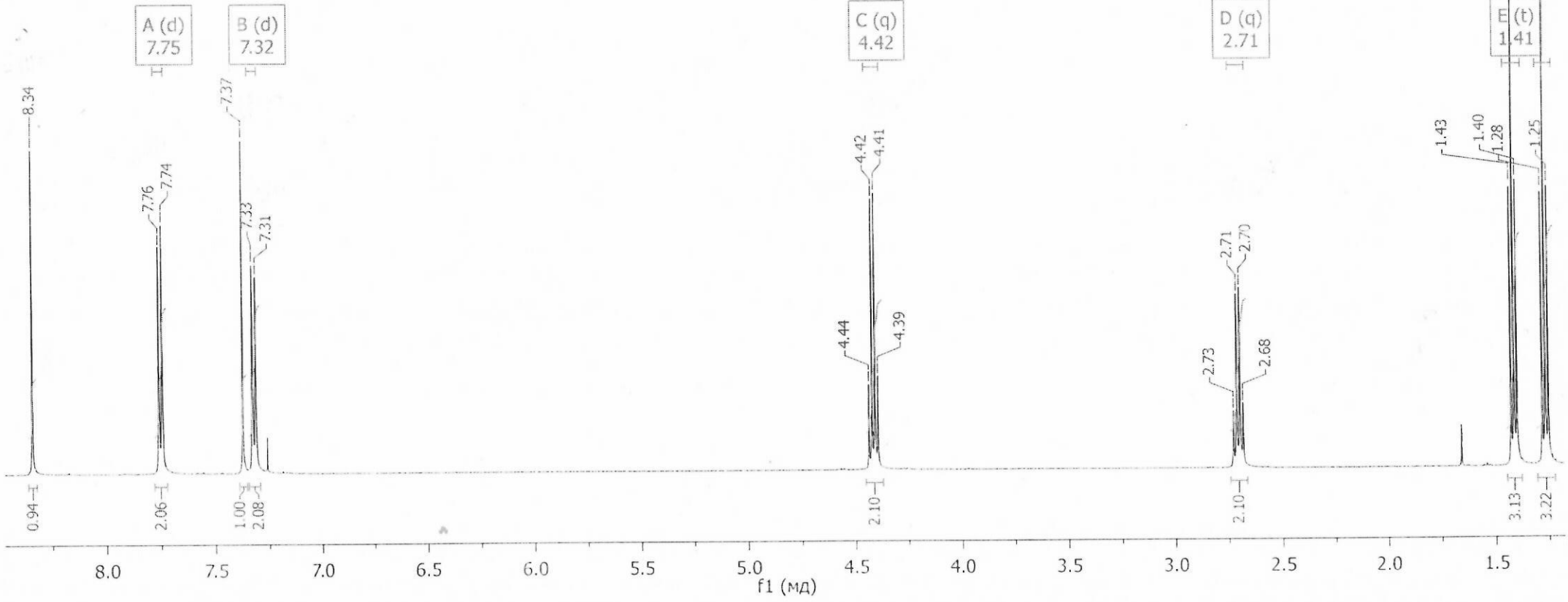


^{1}H NMR (500 MHz, $CDCl_3$) δ = 7.75 (d, *J*=8.3, 1H), 7.32 (d, *J*=8.4, 1H), 4.42 (q, *J*=7.1, 1H), 2.71 (q, *J*=7.6, 1H), 1.41 (t, *J*=7.1, 2H), 1.27 (t, *J*=7.6, 2H).

5c



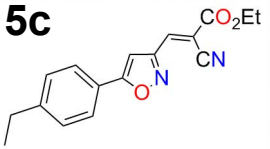


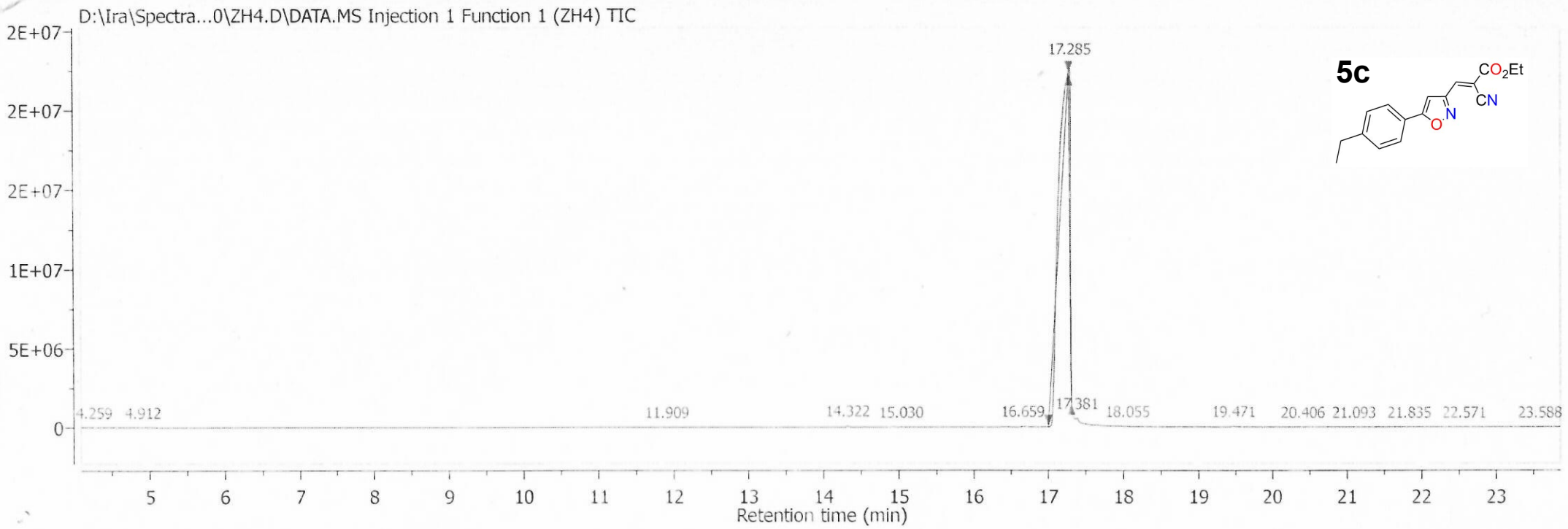


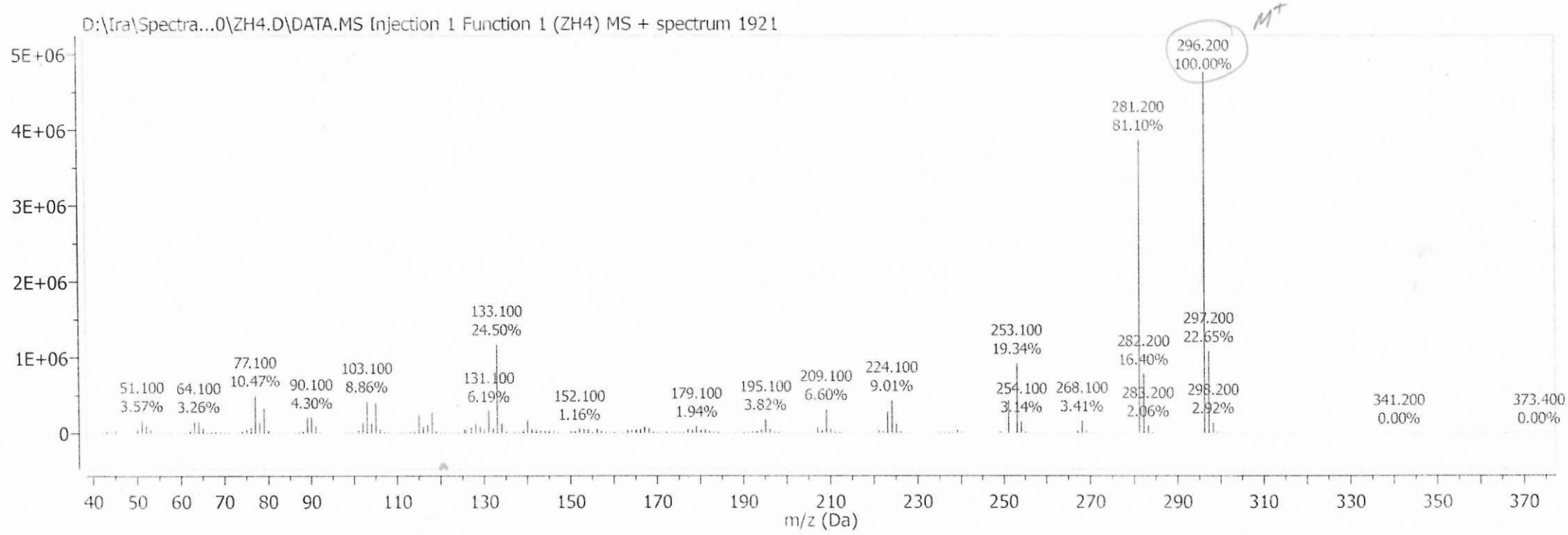
SK_zh4_03222023

bbo_13CF_bar CDCl3 /v nmrsu 16

5c

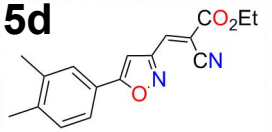


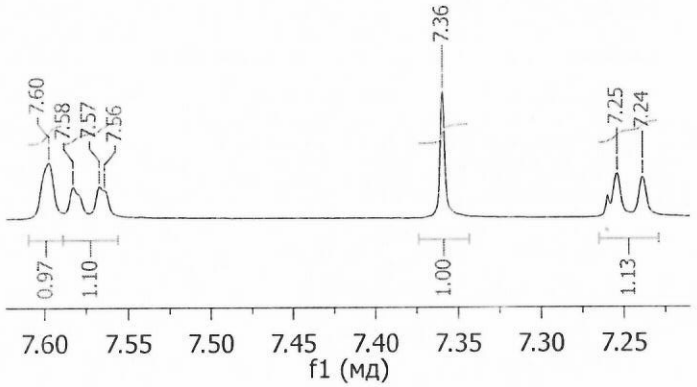

D:\Ira\Spectra...0\ZH4.D\DATA.MS Injection 1 Function 1 (ZH4) TIC
2E+07
2E+07
2E+07
1E+07
5E+06
0
17.285
4.259
4.912
11.909
14.322
15.030
16.659
17.381
18.055
19.471
20.406
21.093
21.835
22.571
23.588
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
Retention time (min)
5c
CO2Et
CN
N
O

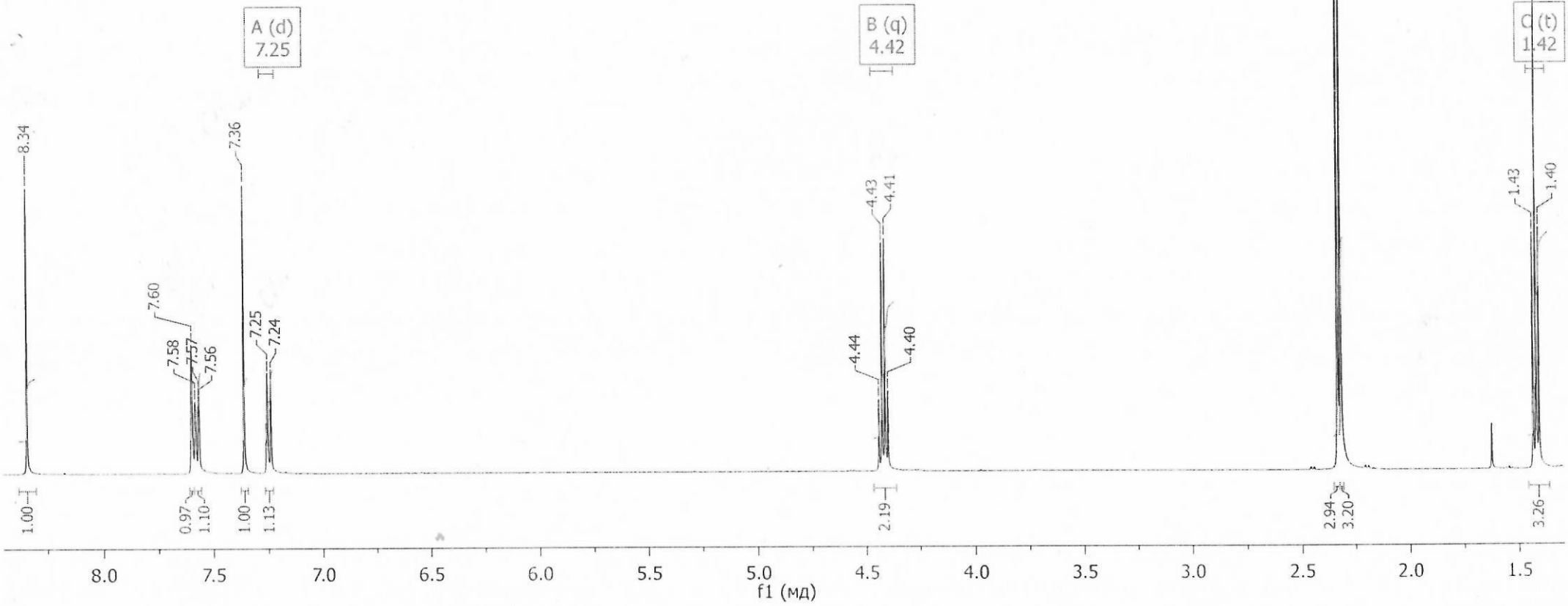

D:\Ira\Spectra...0\ZH4.D\DATA.MS Injection 1 Function 1 (ZH4) MS + spectrum 1921
M+
5E+06
4E+06
3E+06
2E+06
1E+06
0
296.200
100.00%
281.200
81.10%
133.100
24.50%
253.100
19.34%
297.200
22.65%
282.200
16.40%
77.100
10.47%
103.100
8.86%
224.100
9.01%
209.100
6.60%
51.100
3.57%
64.100
3.26%
90.100
4.30%
131.100
6.19%
152.100
1.16%
179.100
1.94%
195.100
3.82%
254.100
3.14%
268.100
3.41%
283.200
2.06%
298.200
2.92%
341.200
0.00%
373.400
0.00%
40
50
60
70
80
90
110
130
150
170
190
210
230
250
270
290
310
330
350
370
m/z (Da)

^{1}H NMR (500 MHz, $CDCl_3$) δ = 7.25 (d, *J*=7.8, 1H), 4.42 (q, *J*=7.1, 2H), 1.42 (t, *J*=7.1, 3H).

5d



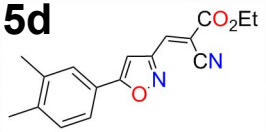


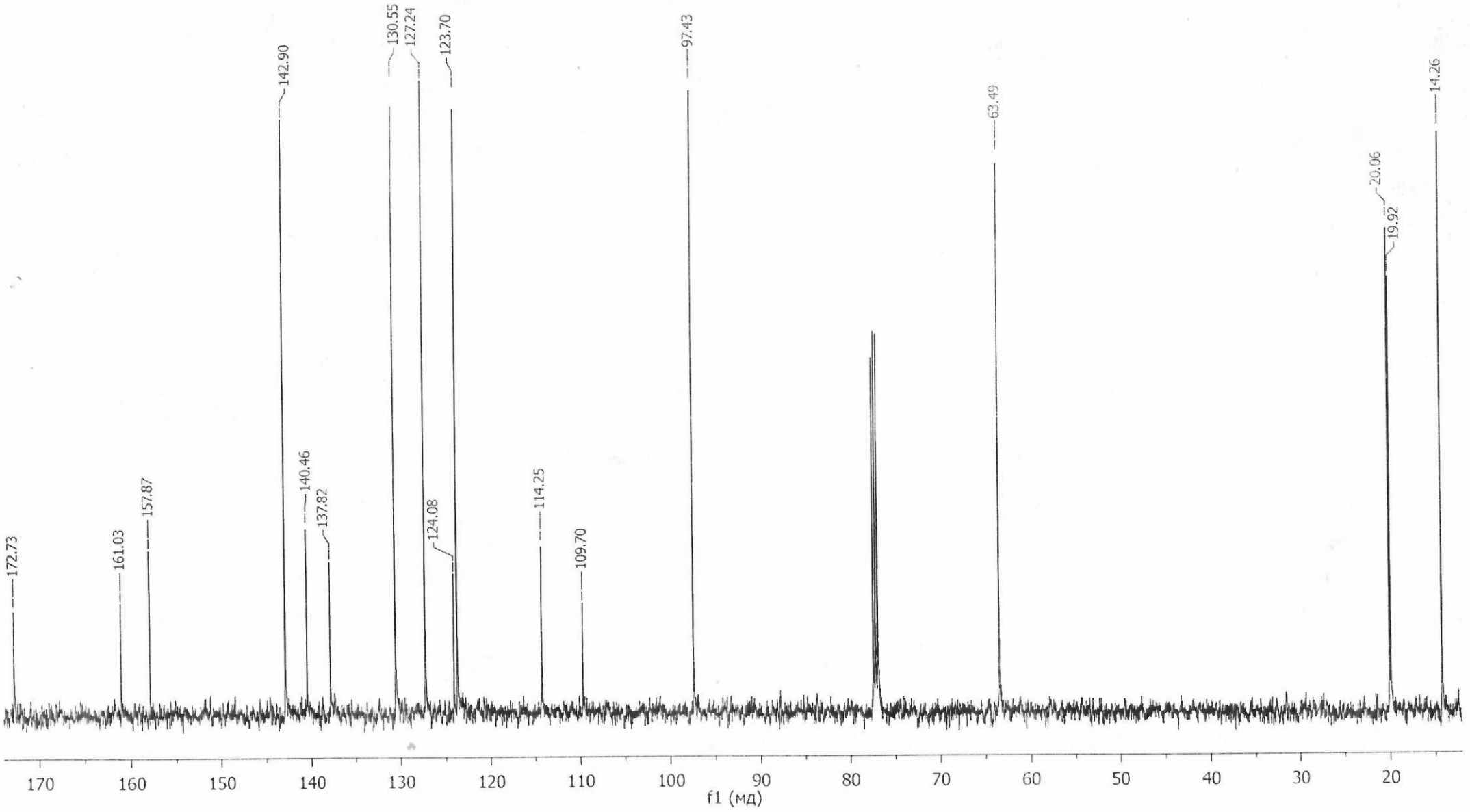


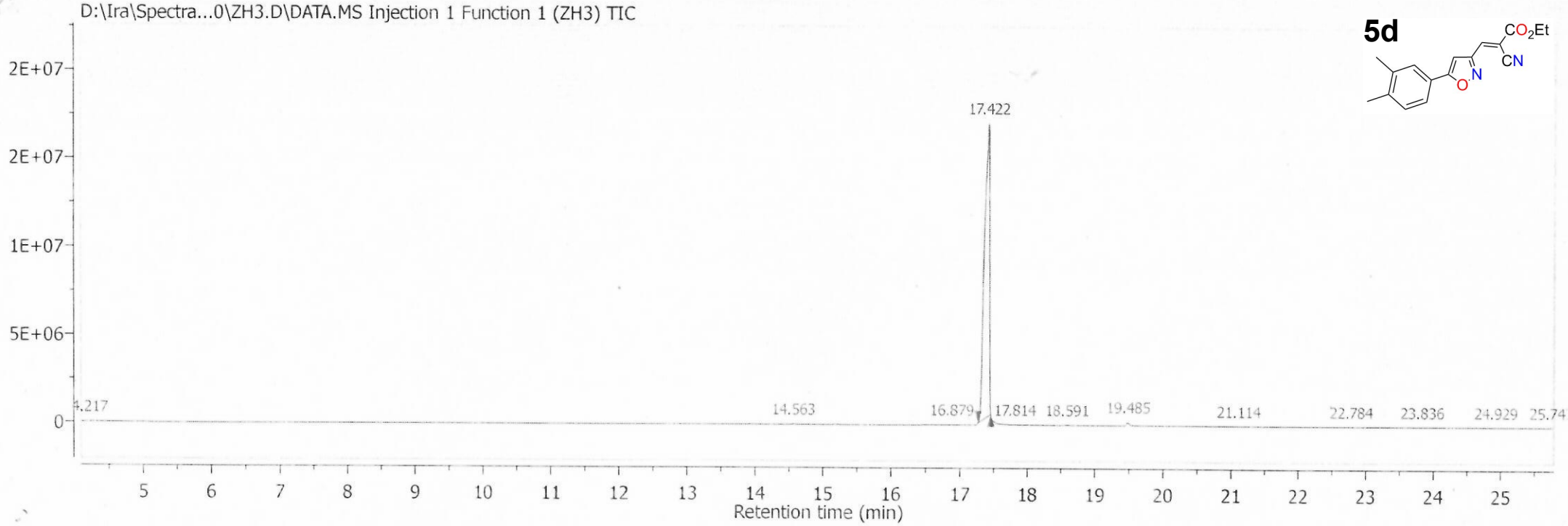
SK_zh3_03222023

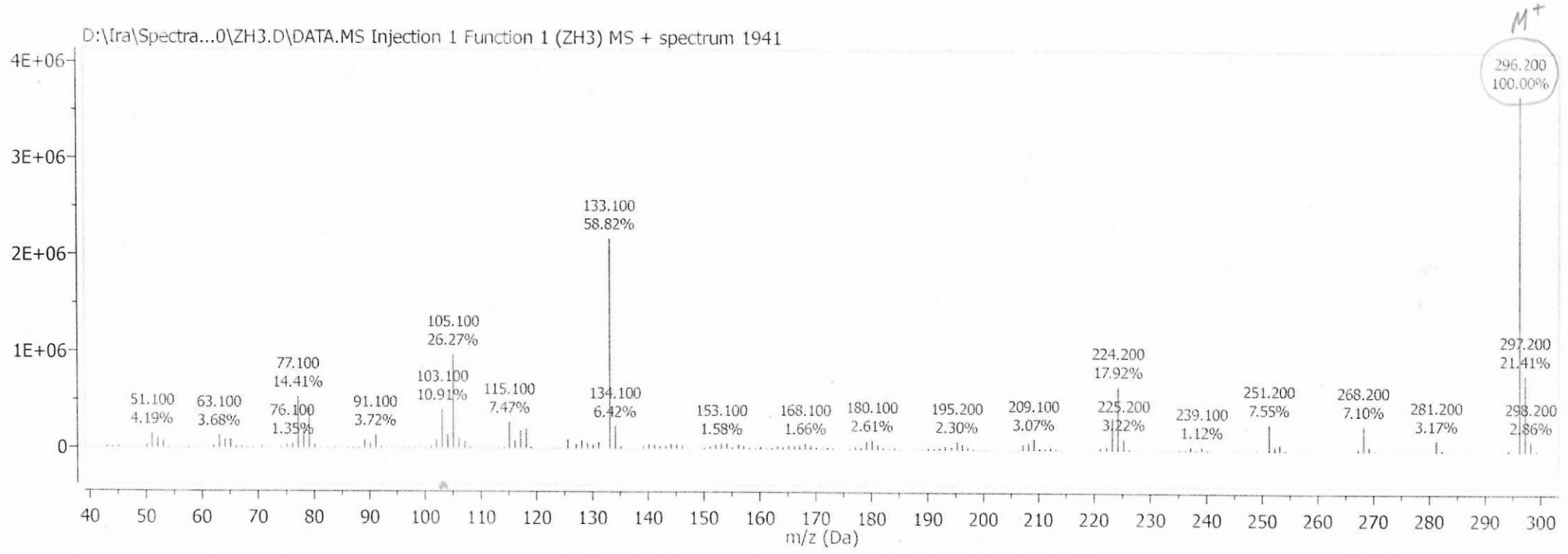
bbo_13CF_bar CDCl3 /v nmrsu 15

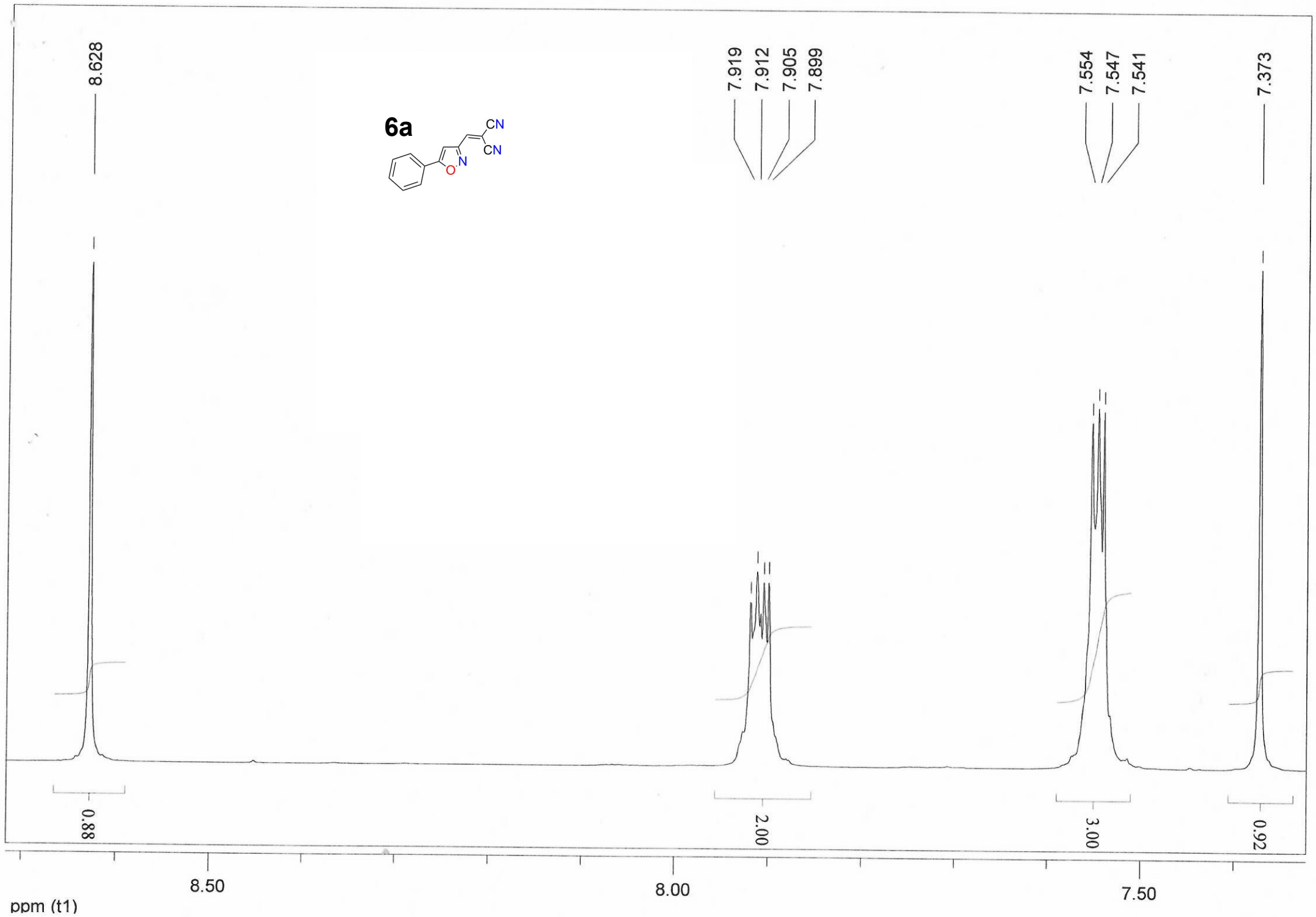
5d

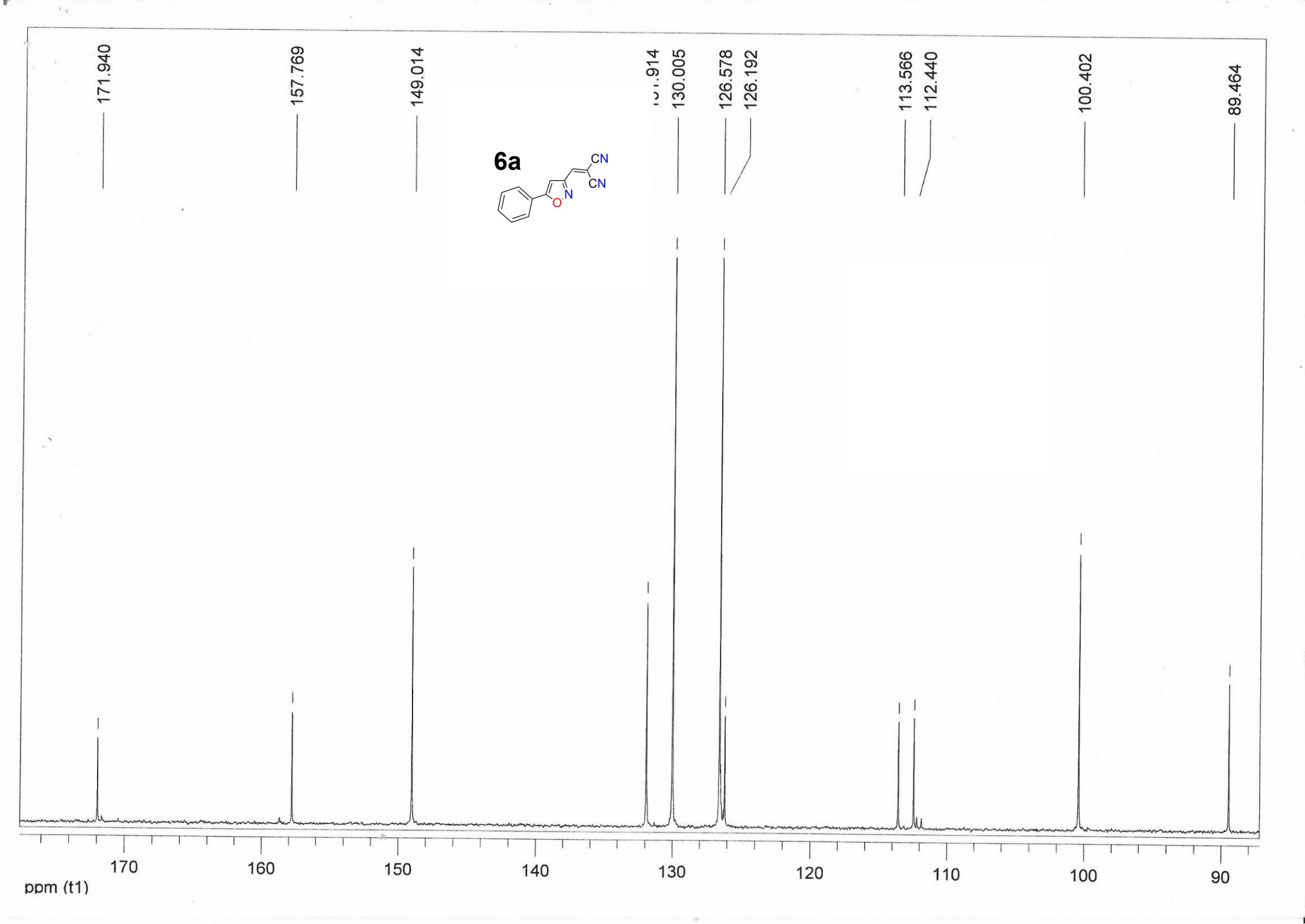




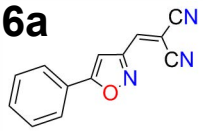

D:\Ira\Spectra...0\ZH3.D\DATA.MS Injection 1 Function 1 (ZH3) TIC
5d
CO2Et
CN
N
O
2E+07
2E+07
1E+07
5E+06
0
17.422
4.217
14.563
16.879
17.814
18.591
19.485
21.114
22.784
23.836
24.929
25.74
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
24
25
Retention time (min)


D:\Ira\Spectra...0\ZH3.D\DATA.MS Injection 1 Function 1 (ZH3) MS + spectrum 1941
M+
4E+06
3E+06
2E+06
1E+06
0
296.200
100.00%
133.100
58.82%
105.100
26.27%
77.100
14.41%
103.100
10.91%
115.100
7.47%
134.100
6.42%
51.100
4.19%
63.100
3.68%
76.100
1.35%
91.100
3.72%
153.100
1.58%
168.100
1.66%
180.100
2.61%
195.200
2.30%
209.100
3.07%
224.200
17.92%
225.200
3.22%
239.100
1.12%
251.200
7.55%
268.200
7.10%
281.200
3.17%
297.200
21.41%
298.200
2.86%
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
220
230
240
250
260
270
280
290
300
m/z (Da)


6a
CN
CN
O
N
8.628
7.919
7.912
7.905
7.899
7.554
7.547
7.541
7.373
0.88
2.00
3.00
0.92
8.50
8.00
7.50
ppm (t1)


171.940
157.769
149.014
130.005
126.578
126.192
113.566
112.440
100.402
89.464
6a
CN
CN
O
N
170
160
150
140
130
120
110
100
90
ppm (t1)

6a



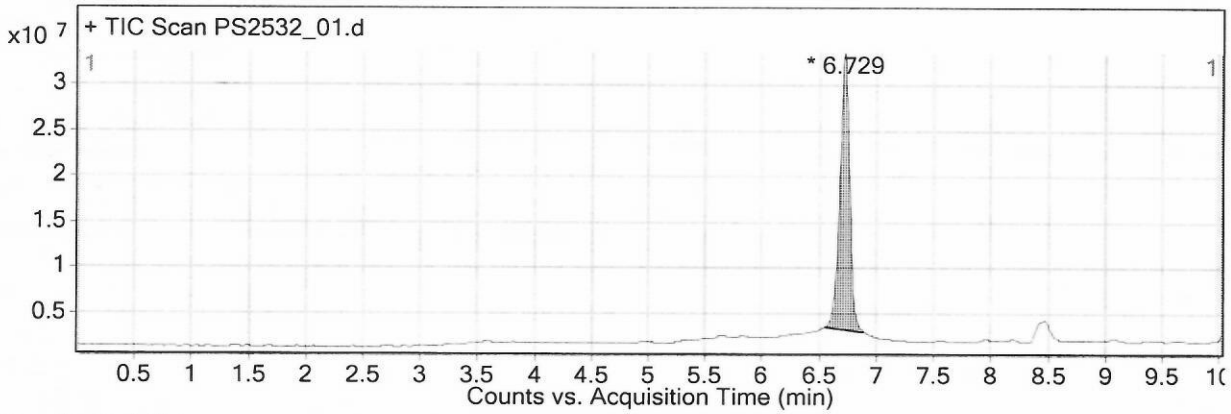
Qualitative Analysis Report

Data Filename	PS2532_01.d	**Sample Name**	Unavailable
Sample Type	Unavailable	**Position**	Unavailable
Instrument Name	Unavailable	**User Name**	Unavailable
Acq Method		**IRM Calibration Status**	Success
DA Method	Default.m	**Comment**	Sample information is unavailable

$[M]^+ = 221$

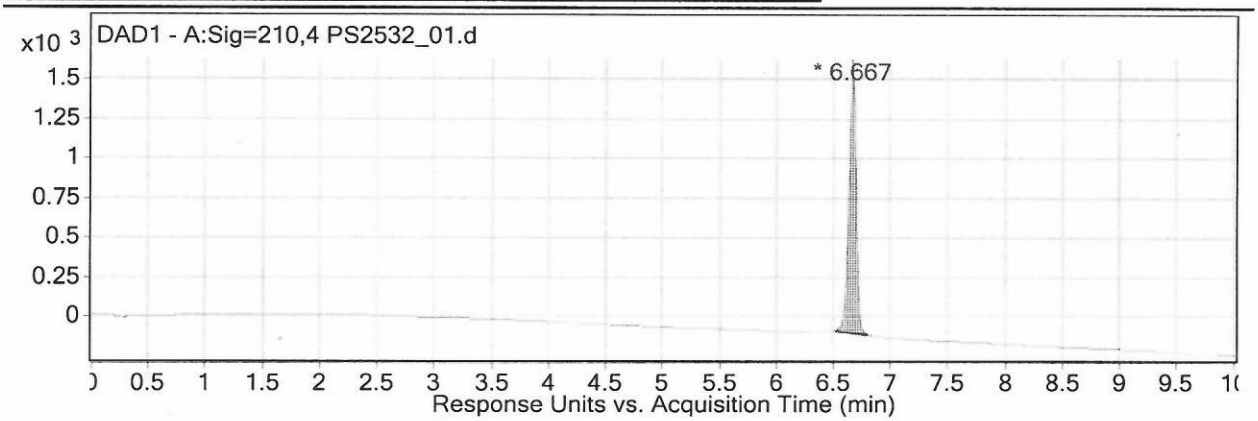
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** Esi



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	6,549	6,729	6,893	30333718	182127295	100

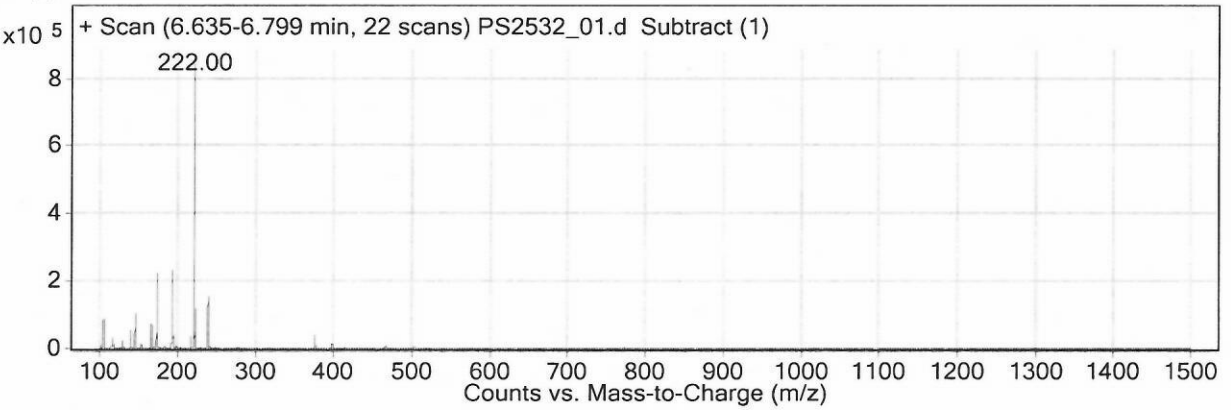


Integration Peak List

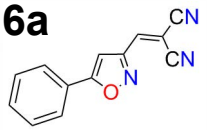
Peak	Start	RT	End	Height	Area	Area %
1	6,513	6,667	6,807	1734,33	7705,43	100

User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan"	135	0	Esi



6a



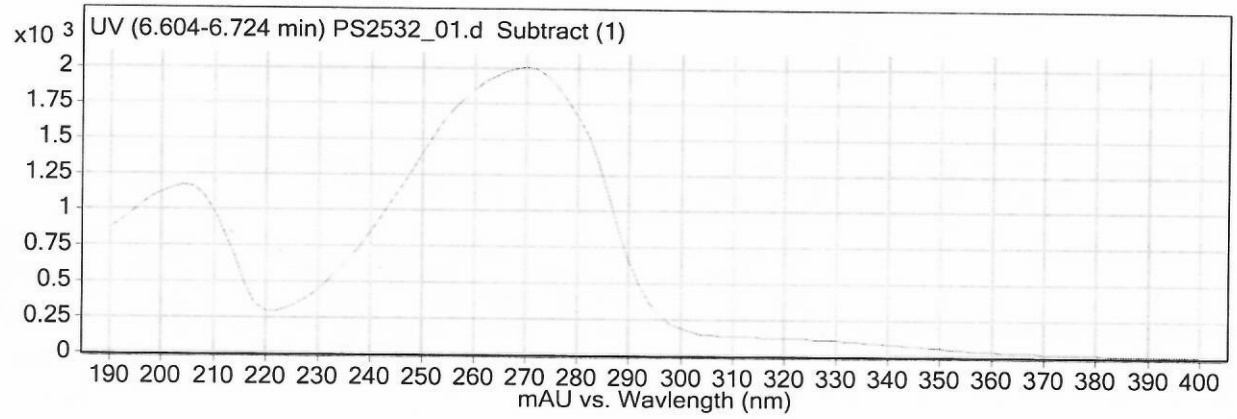
Qualitative Analysis Report

Peak List

m/z	z	Abund.
105		84578
140		54485
146		103453
167		73977
174	1	222866
175	1	55138
194		234144
222	1	842317
223	1	118929
240		153418

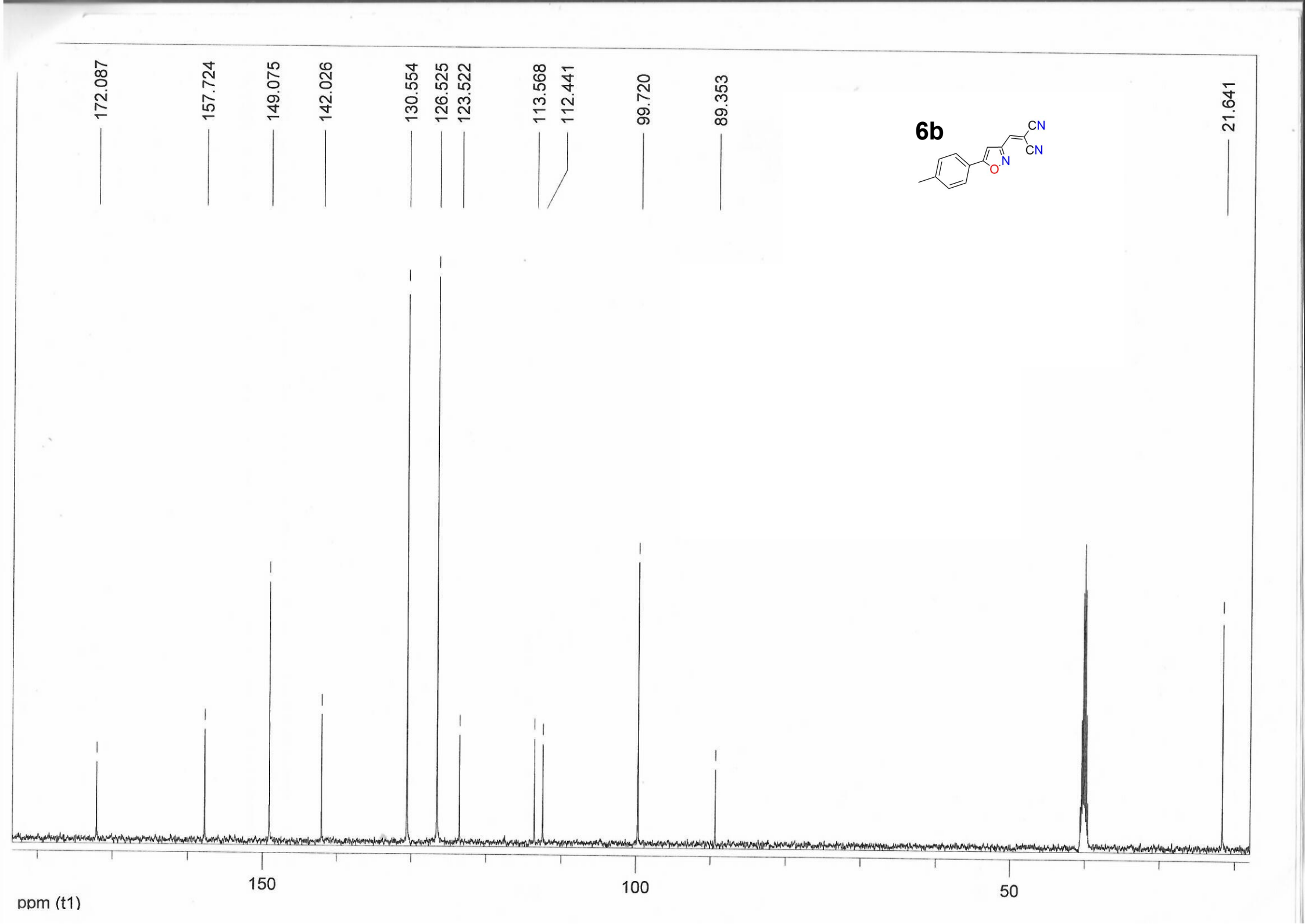
Spectrum Source

Peak (1) in "DAD1 - A:Sig=210,4"

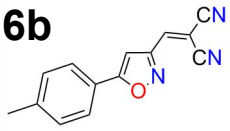


--- End Of Report ---

8.633
7.833
7.816
7.379
7.363
7.327
2.373
6b
CN
CN
O
N
3915.441
3907.344
3688.499
3680.464
3662.915
0.90
2.00
1.88
1.00
3.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
ppm (t1)


172.087
157.724
149.075
142.026
130.554
126.525
123.522
113.568
112.441
99.720
89.353
21.641
6b
CN
CN
N
O
150
100
50
ppm (t1)

6b

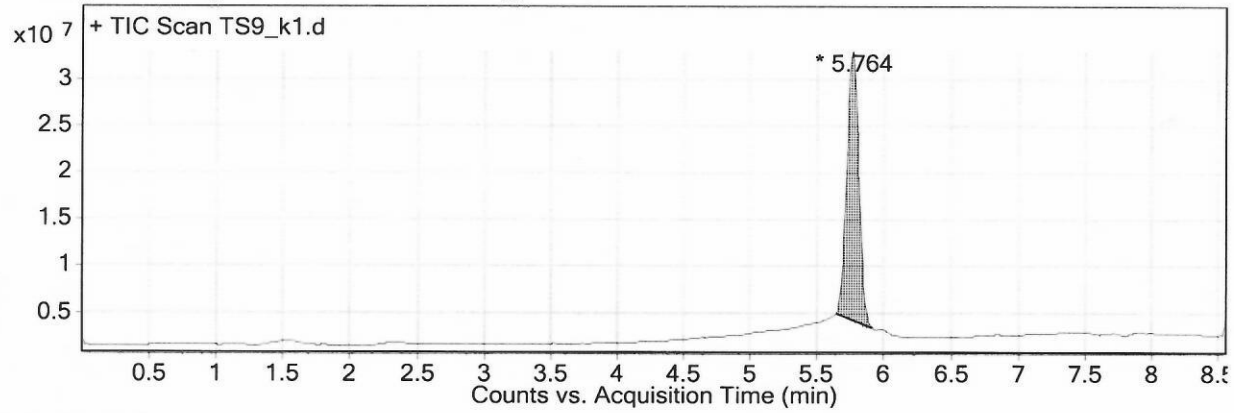


Qualitative Analysis Report

Data Filename	TS9_k1.d	**Sample Name**	Unavailable
Sample Type	Unavailable	**Position**	Unavailable
Instrument Name	Unavailable	**User Name**	Unavailable
Acq Method		**IRM Calibration Status**	Success
DA Method	Default.m	**Comment**	Sample information is unavailable

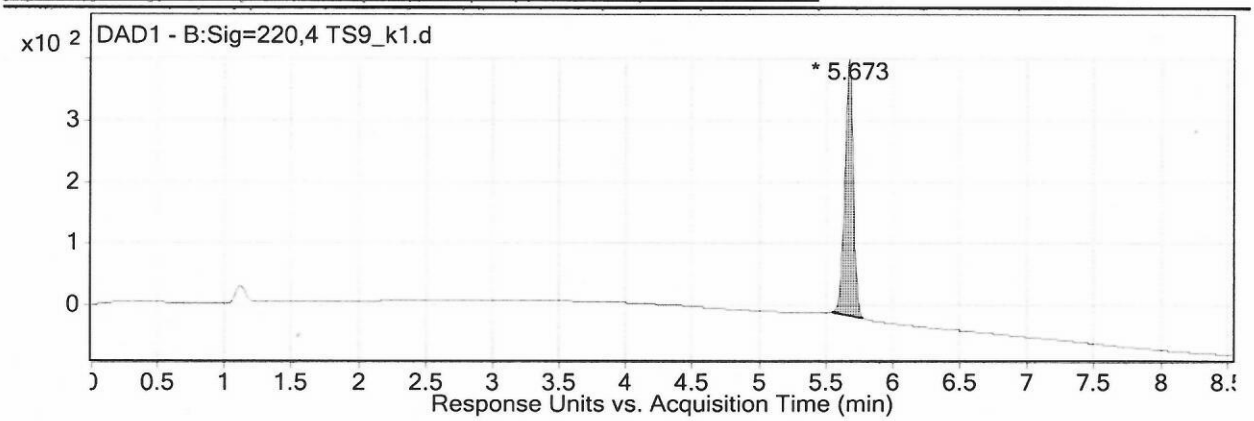
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** Esi



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	5,643	5,764	5,913	28919097	180887399	100

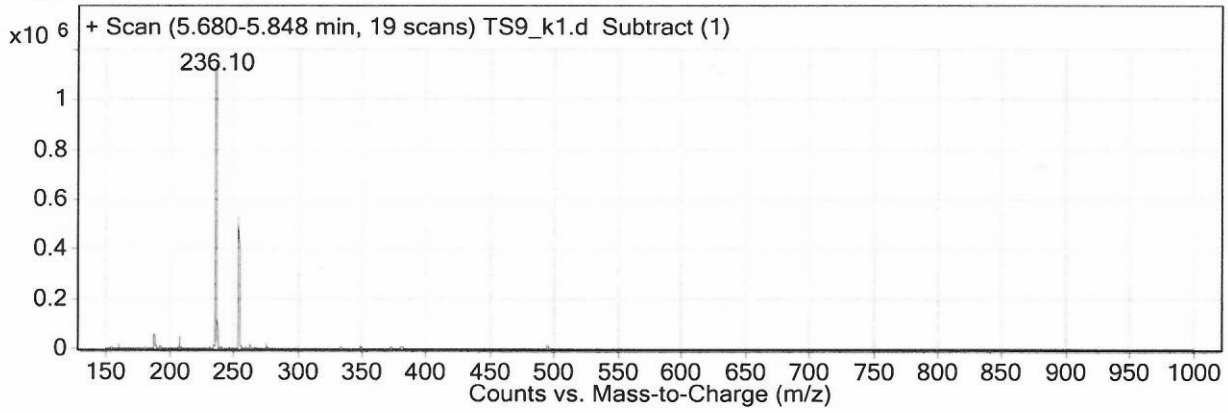


Integration Peak List

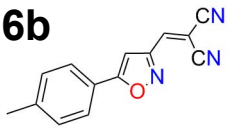
Peak	Start	RT	End	Height	Area	Area %
1	5,547	5,673	5,780	416,69	1863,74	100

User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan"	135	0	Esi



6b



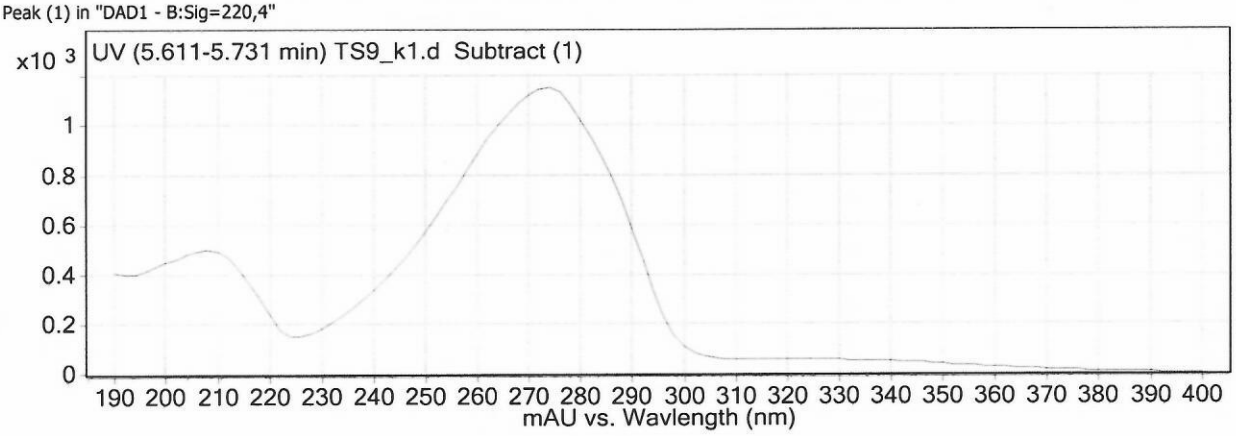
Qualitative Analysis Report

Peak List

m/z	z	Abund.
236,1	1	1143691
237,1	1	172393
254,1	1	532860
255,1	1	96979

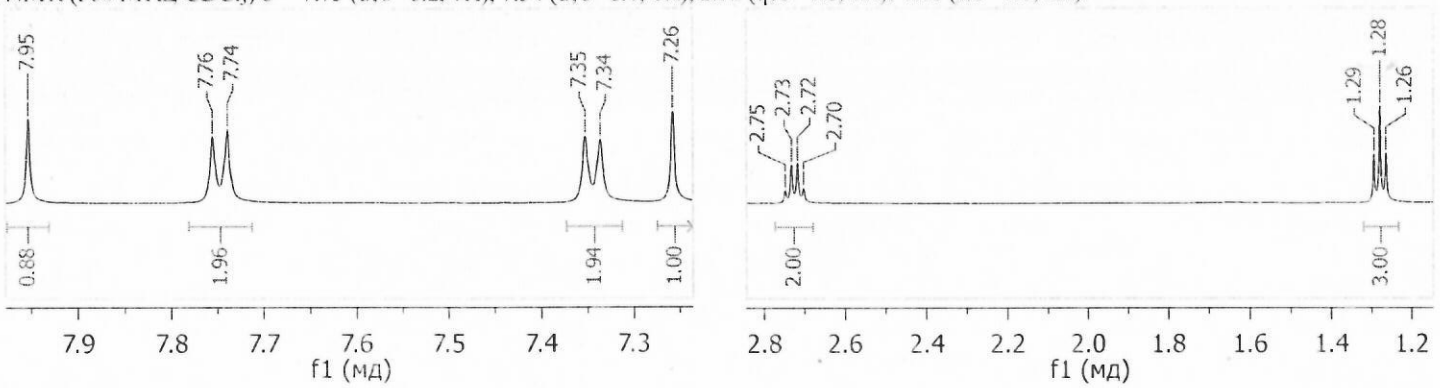
Spectrum Source

Peak (1) in "DAD1 - B:Sig=220,4"

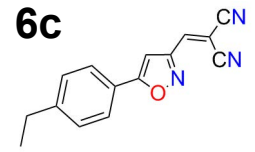


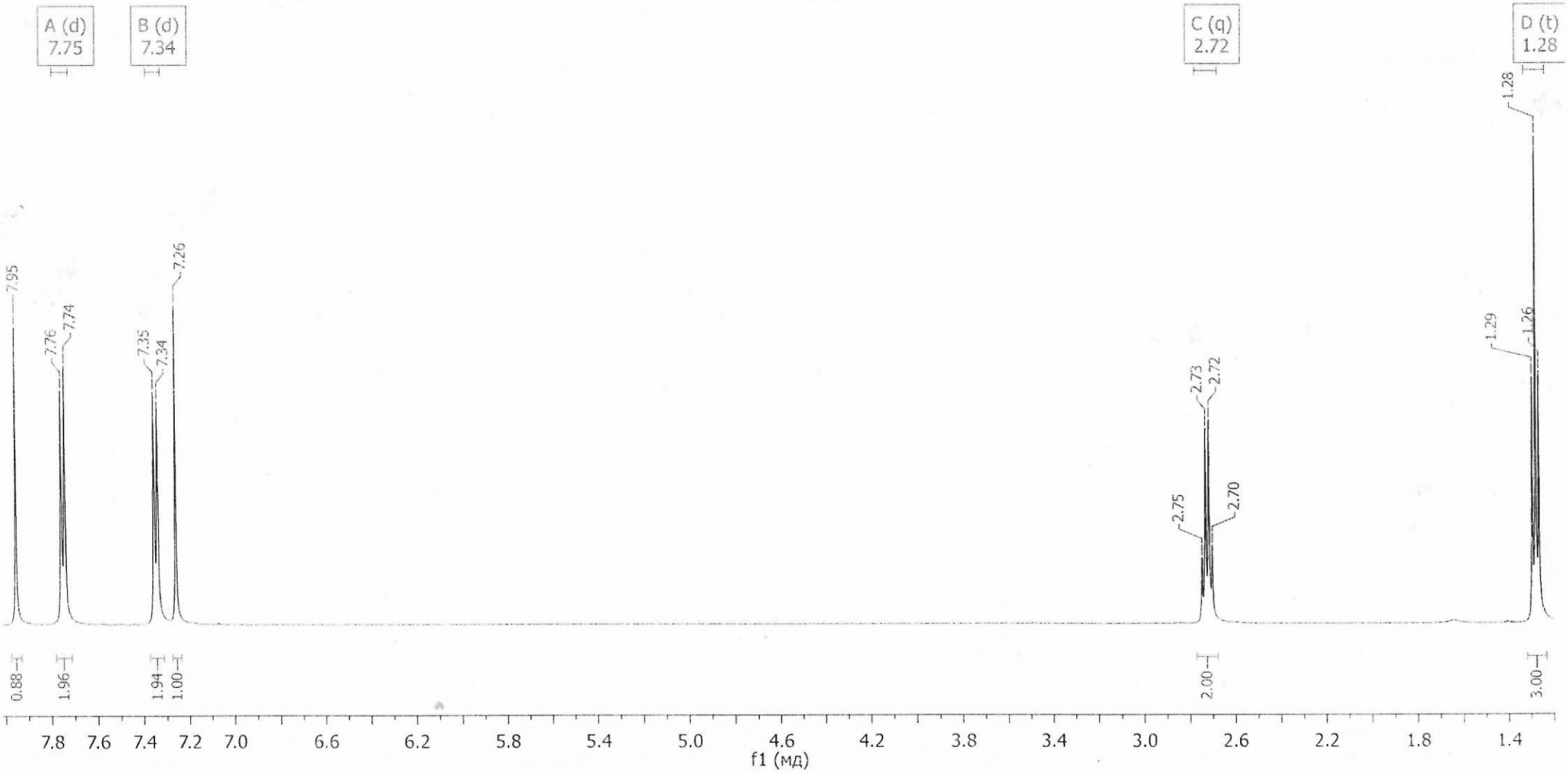
--- End Of Report ---

^{1}H NMR (500 MHz, $CDCl_3$) δ = 7.75 (d, *J*=8.2, 1H), 7.34 (d, *J*=8.1, 1H), 2.72 (q, *J*=7.6, 1H), 1.28 (t, *J*=7.6, 2H).



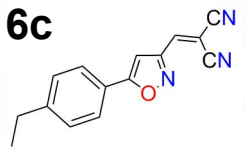
6c



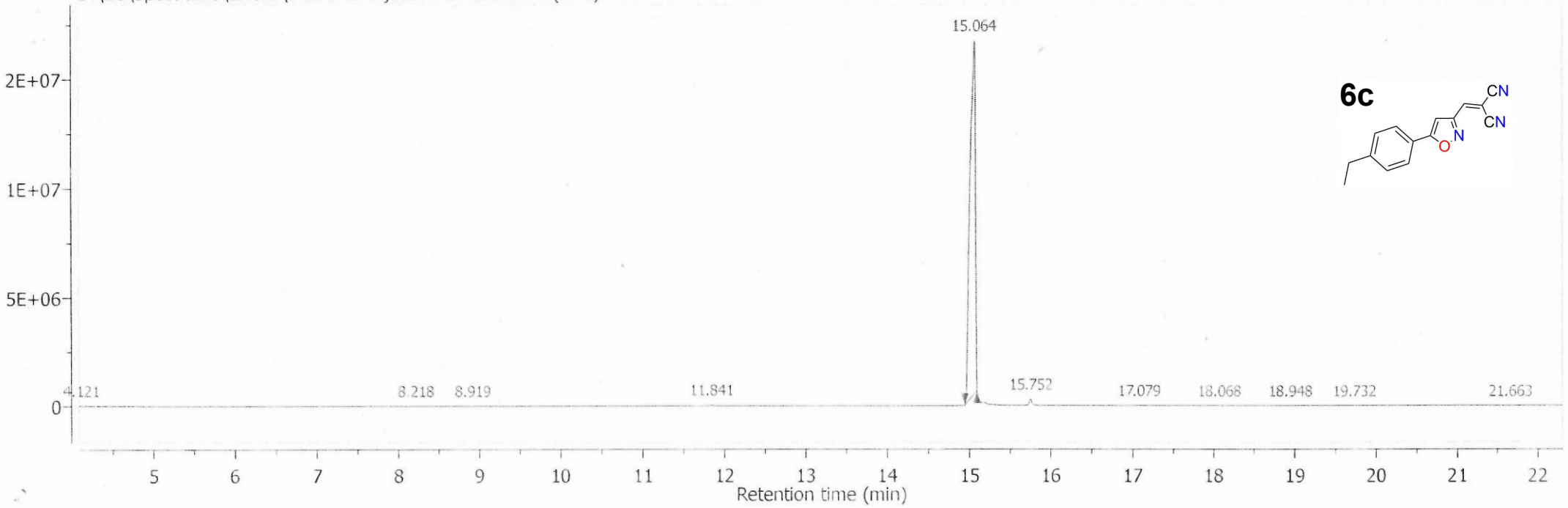


SK_zh-8_03292023

bbo_13CF_bar CDCl3 /v nmrsu 4

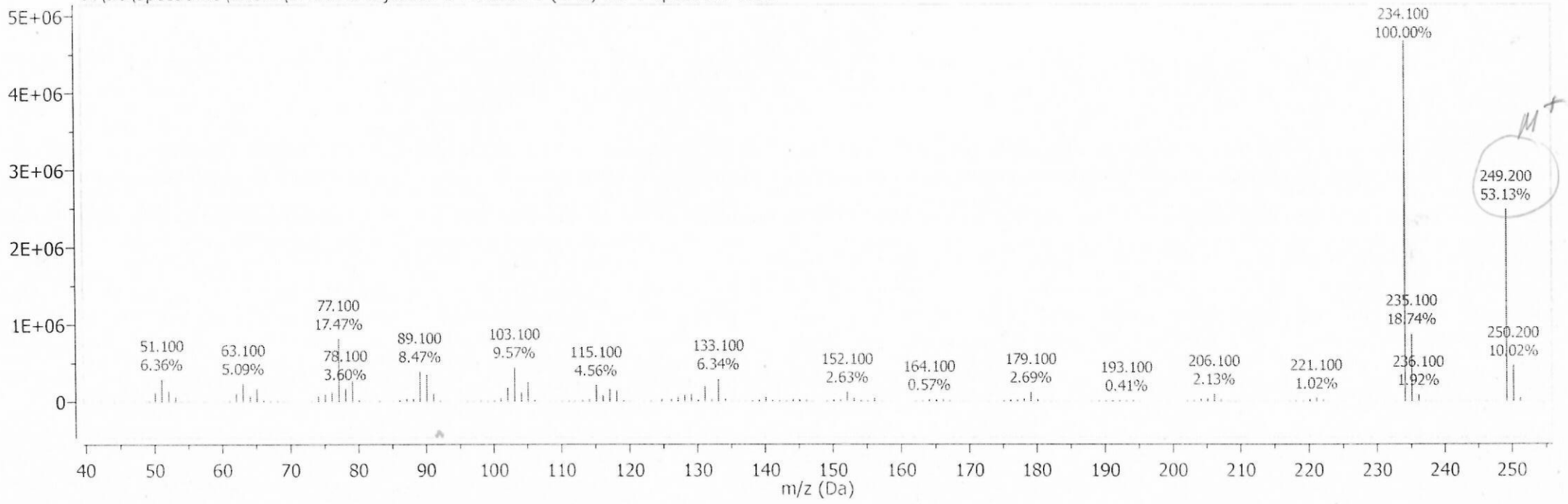


D:\Ira\Spectra...0\ZH8.D\DATA.MS Injection 1 Function 1 (ZH8) TIC

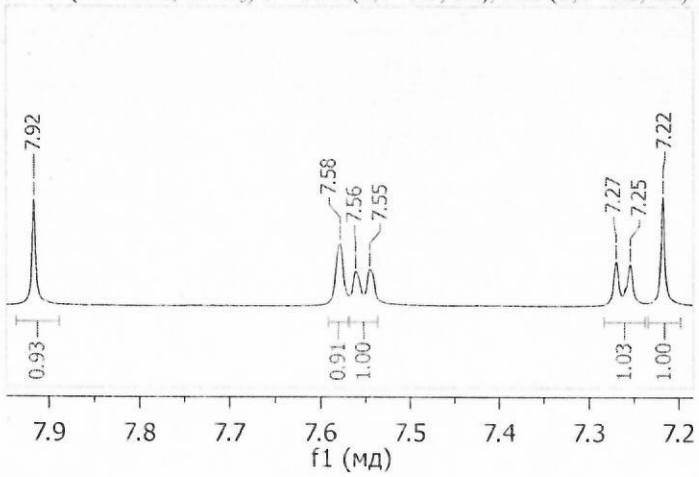


6c

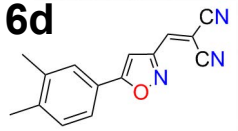
D:\Ira\Spectra...0\ZH8.D\DATA.MS Injection 1 Function 1 (ZH8) MS + spectrum 1598



^{1}H NMR (500 MHz, $CDCl_3$) δ = 7.55 (d, *J*=7.9, 1H), 7.26 (d, *J*=7.8, 2H).

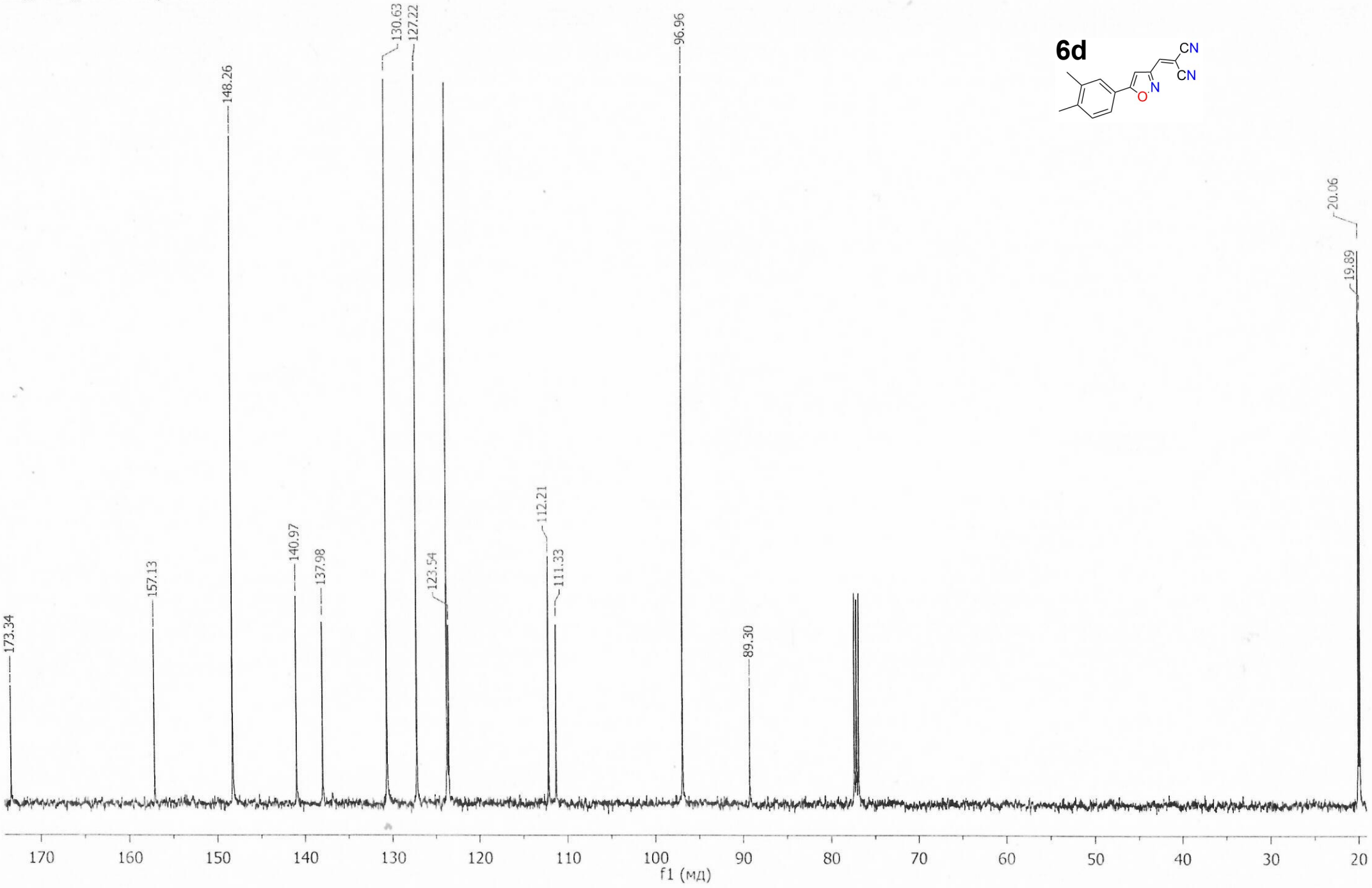


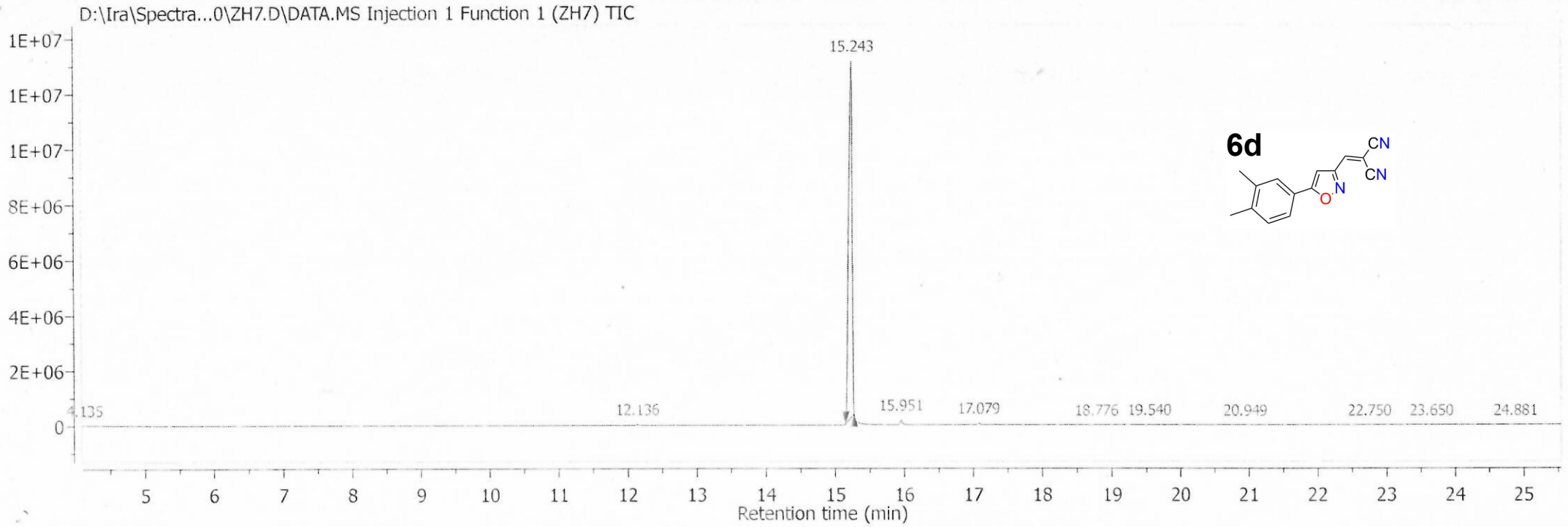
6d

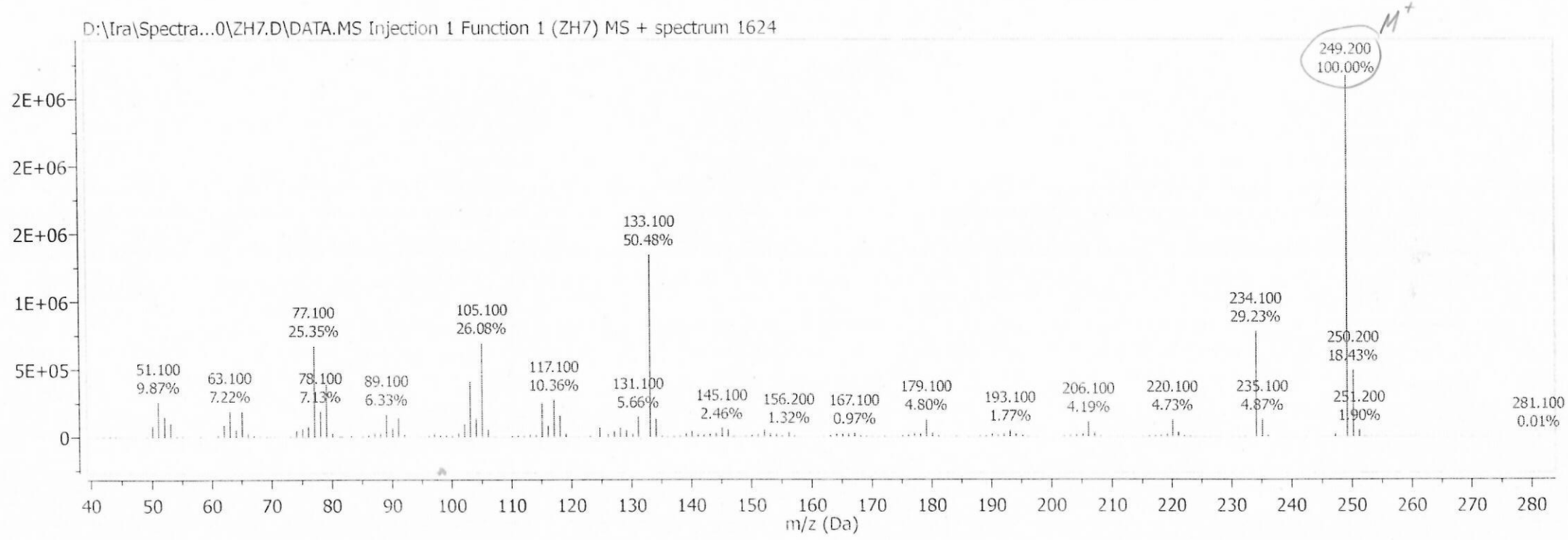


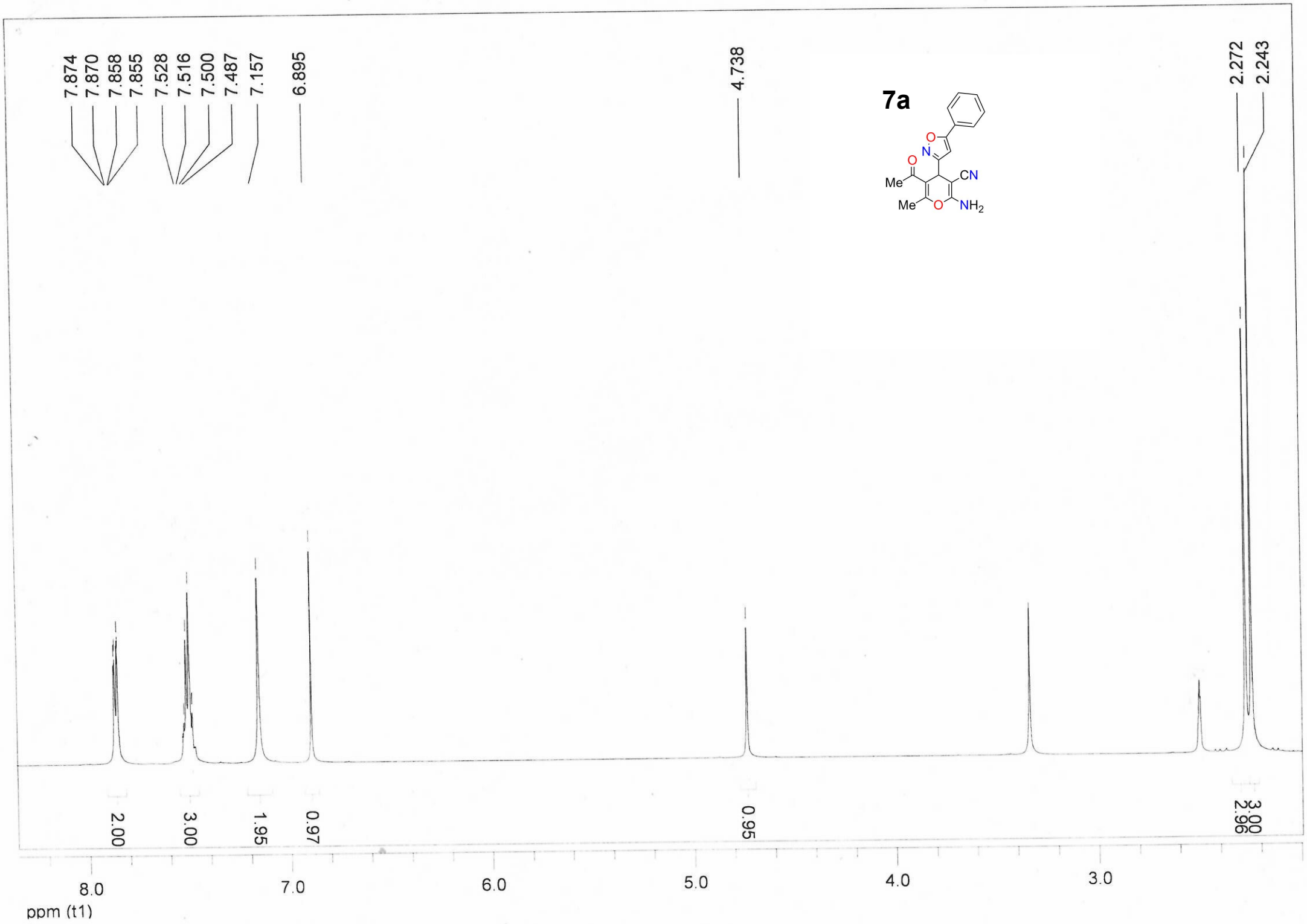
SK_zh-7_03292023

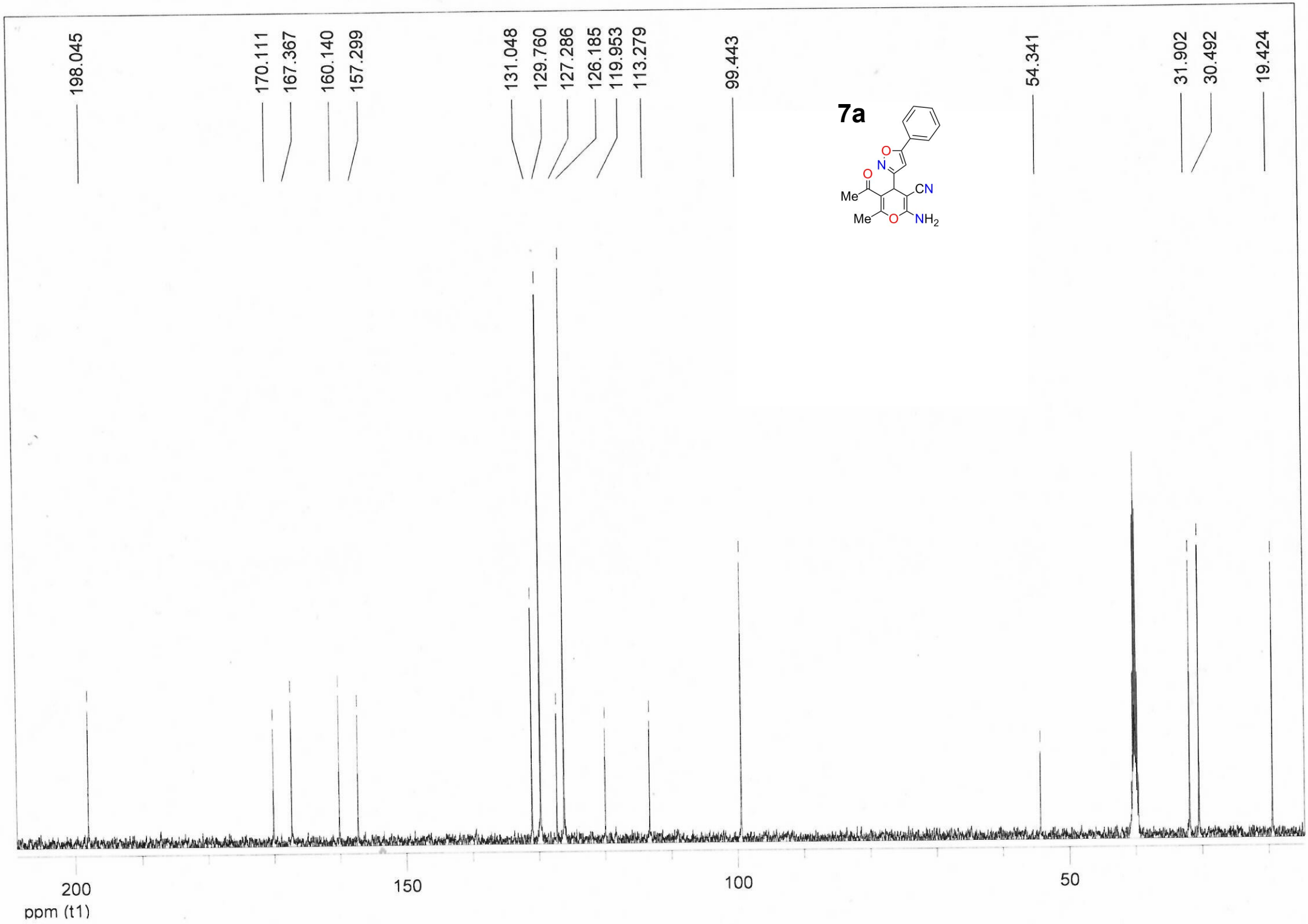
bbo_13CF_bar CDCl3 /v nmrsu 3




D:\Ira\Spectra...0\ZH7.D\DATA.MS Injection 1 Function 1 (ZH7) TIC
6d
CN
CN
N
O
15.243
4.135
12.136
15.951
17.079
18.776
19.540
20.949
22.750
23.650
24.881
Retention time (min)


D:\Ira\Spectra...0\ZH7.D\DATA.MS Injection 1 Function 1 (ZH7) MS + spectrum 1624
M+
249.200
100.00%
133.100
50.48%
77.100
25.35%
105.100
26.08%
234.100
29.23%
250.200
18.43%
51.100
9.87%
63.100
7.22%
78.100
7.13%
89.100
6.33%
117.100
10.36%
131.100
5.66%
145.100
2.46%
156.200
1.32%
167.100
0.97%
179.100
4.80%
193.100
1.77%
206.100
4.19%
220.100
4.73%
235.100
4.87%
251.200
1.90%
281.100
0.01%
m/z (Da)


7.874
7.870
7.858
7.855
7.528
7.516
7.500
7.487
7.157
6.895
4.738
2.272
2.243
7a
O
N
O
Me
CN
Me
O
NH2
2.00
3.00
1.95
0.97
0.95
3.00
2.96
8.0
7.0
6.0
5.0
4.0
3.0
ppm (t1)

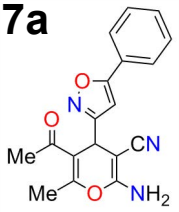

198.045
170.111
167.367
160.140
157.299
131.048
129.760
127.286
126.185
119.953
113.279
99.443
54.341
31.902
30.492
19.424
7a
O
N
O
Me
CN
Me
O
NH2
200
150
100
50
ppm (t1)

Qualitative Analysis Report

Data Filename	PS2813_01.D	**Sample Name**	
Sample Type	Sample	**Position**	Vial 2
Instrument Name	Instrument 1	**User Name**	
Acq Method	ALL_2020_KOL2.M	**Acquired Time**	10/15/2020 2:16:09 PM
IRM Calibration Status	Not Applicable	**DA Method**	ChromPeakSurvey-Default.m
Comment			

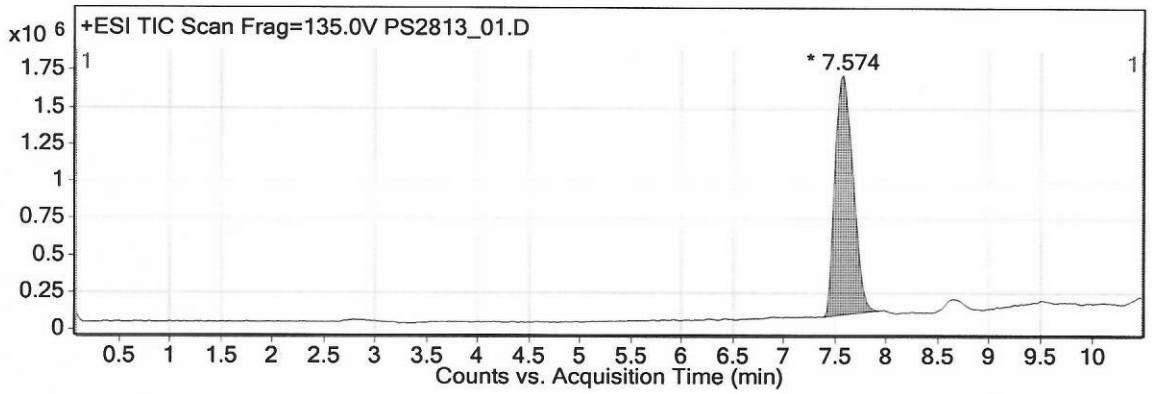
OperatorName **RunCompletedFlag** True

7a



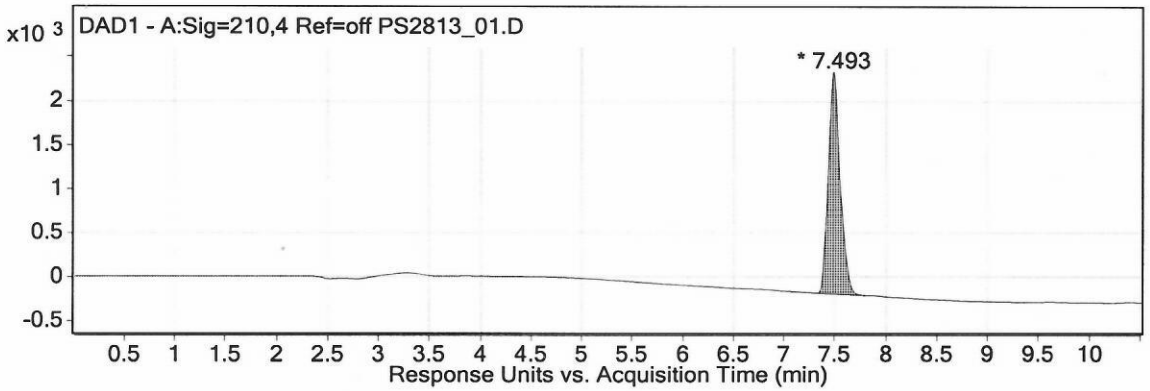
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,374	7,574	7,933	1619479	19933823	100



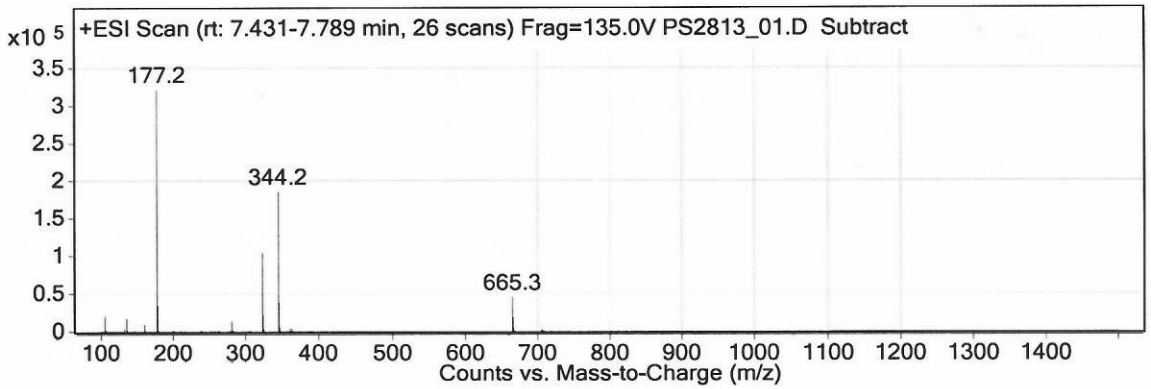
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,246	7,493	7,813	2526,35	20151,26	100

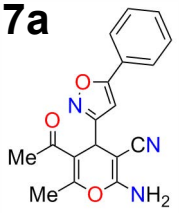
User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan"	135	0	ESI

Qualitative Analysis Report



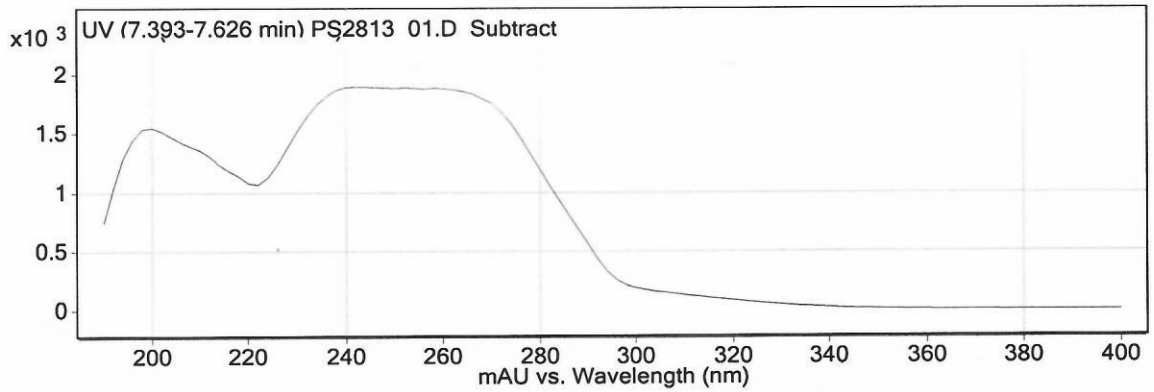
7a



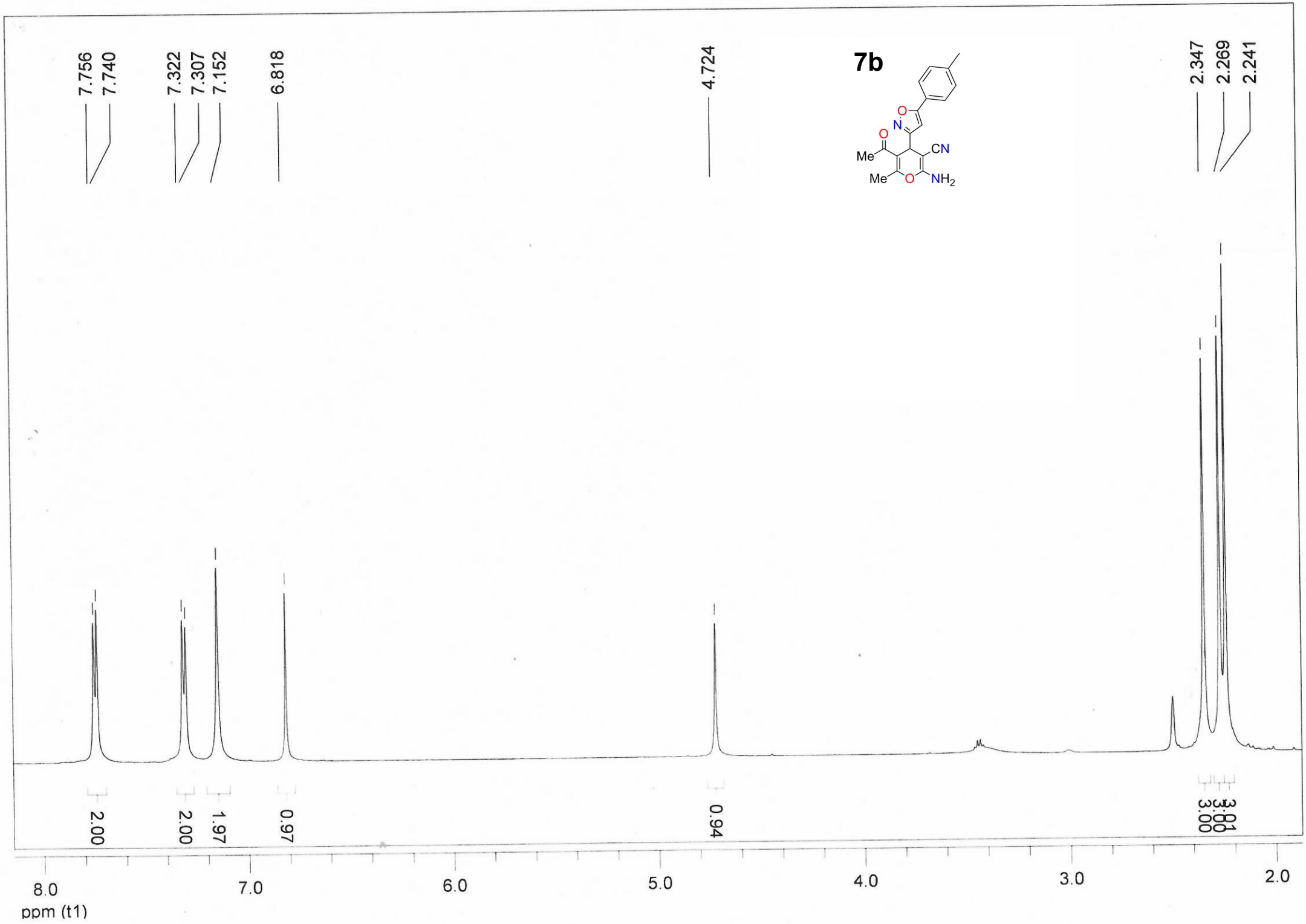
Peak List

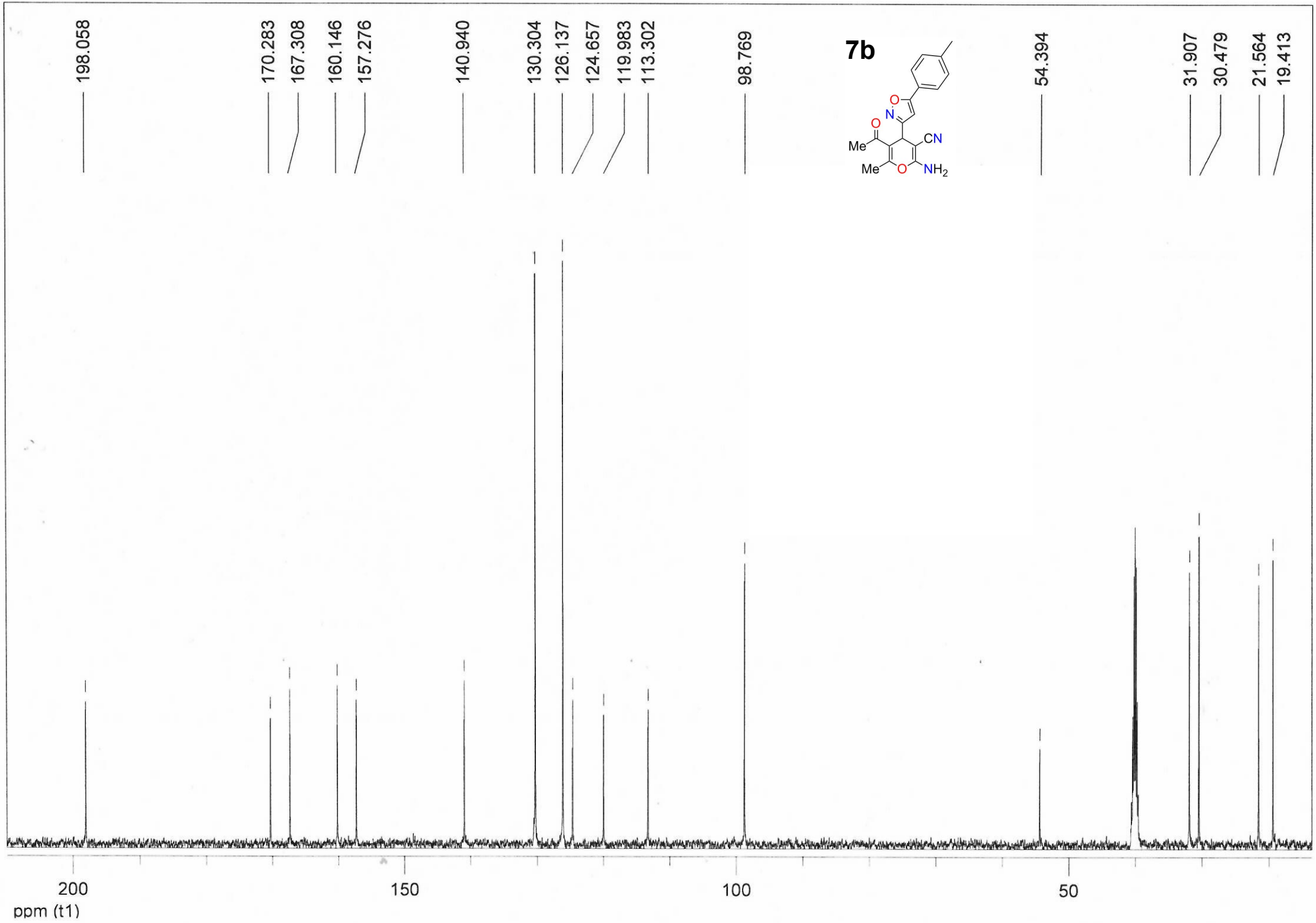
m/z	z	Abund
105.2		21053.46
135.2		17740.36
177.2		321999.59
178.2		34744.21
322.2		104192.57
323.2		21916.67
344.2	1	185685.17
345.2	1	37806.77
665.3	1	45190.74
666.3	1	18744.09

Spectrum Source
Peak (1) in "DAD1 - A:Sig=210,4 Ref=off"



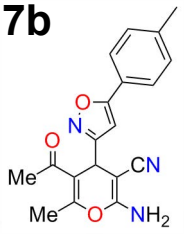
--- End Of Report ---


7.756
7.740
7.322
7.307
7.152
6.818
4.724
2.347
2.269
2.241
7b
O
N
O
Me
CN
Me
O
NH2
2.00
2.00
1.97
0.97
0.94
3.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
ppm (t1)


198.058
170.283
167.308
160.146
157.276
140.940
130.304
126.137
124.657
119.983
113.302
98.769
54.394
31.907
30.479
21.564
19.413
7b
O
N
O
Me
CN
Me
O
NH2
200
150
100
50
ppm (t1)

Qualitative Analysis Report

7b

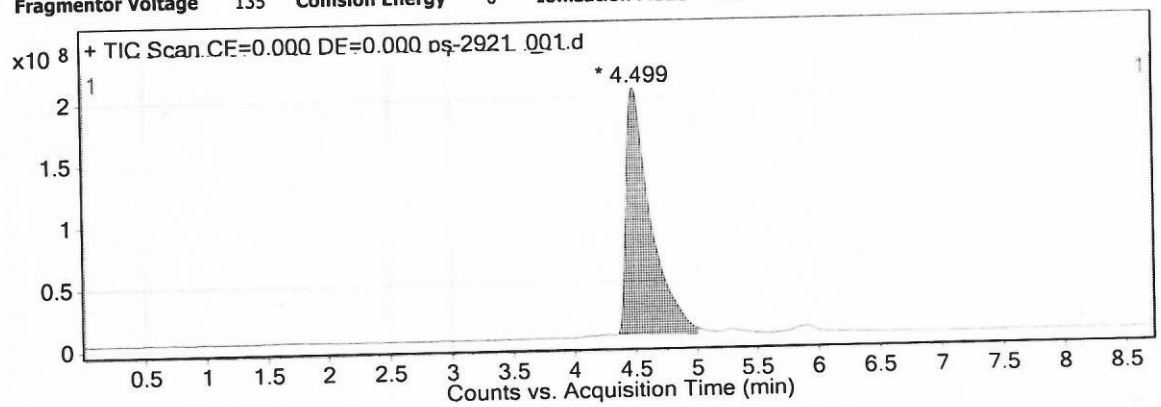


Data Filename	ps-2921_001.d	**Sample Name**	ps-2921
Sample Type	Sample	**Position**	Vial 2
Instrument Name	Instrument 1	**User Name**	
Acq Method	All_2021_kol 1-2.m	**Acquired Time**	5/11/2022 12:16:18 PM
IRM Calibration Status	Not Applicable	**DA Method**	Defaul1t.m
Comment			

Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition SW Version**	6400 Series Triple Quadrupole 10.0 (127)

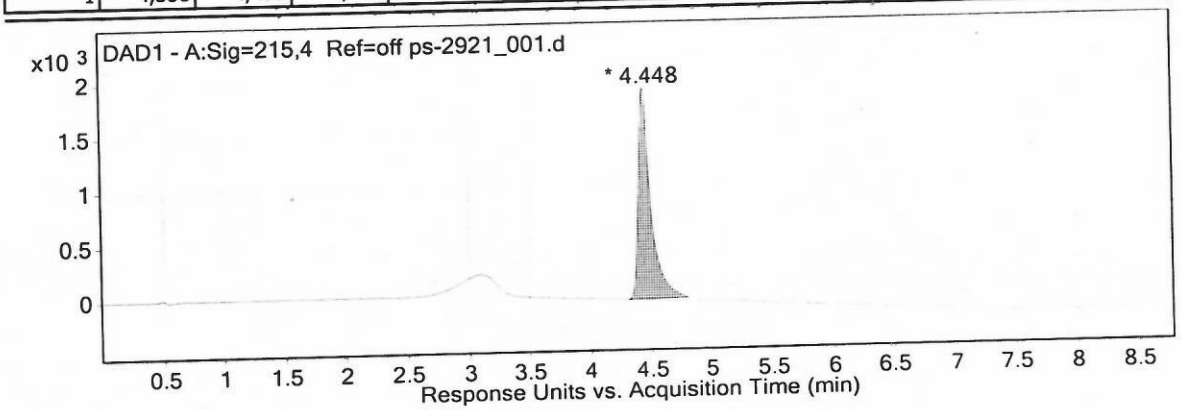
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	4,358	4,499	5,006	198895770,9	2801014288	100



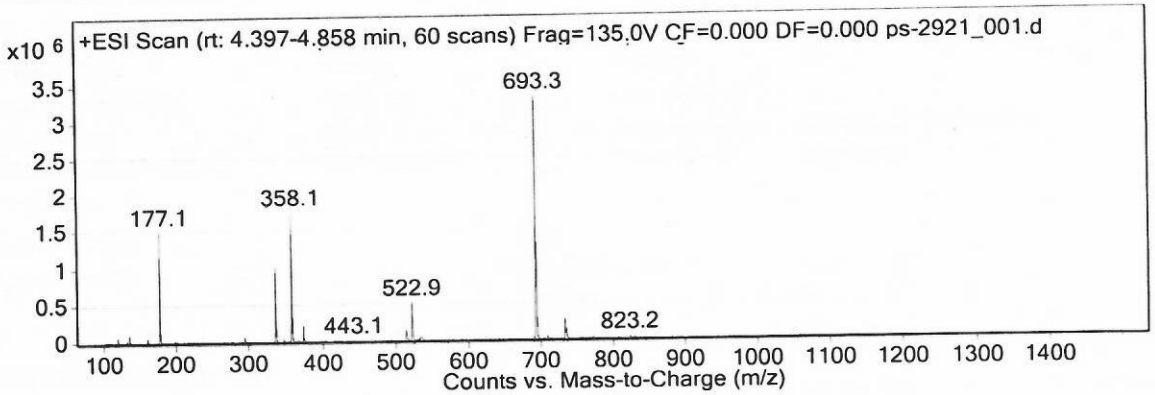
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	4,308	4,448	4,815	1946,31	13879,29	100

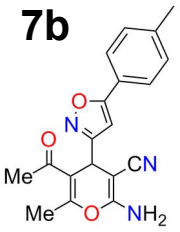
User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan"	135	0	ESI

Qualitative Analysis Report



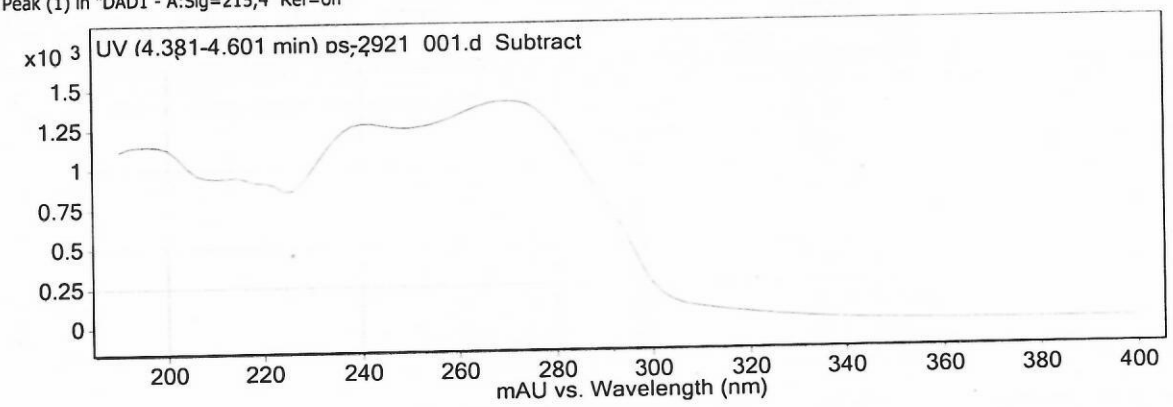
7b



Peak List

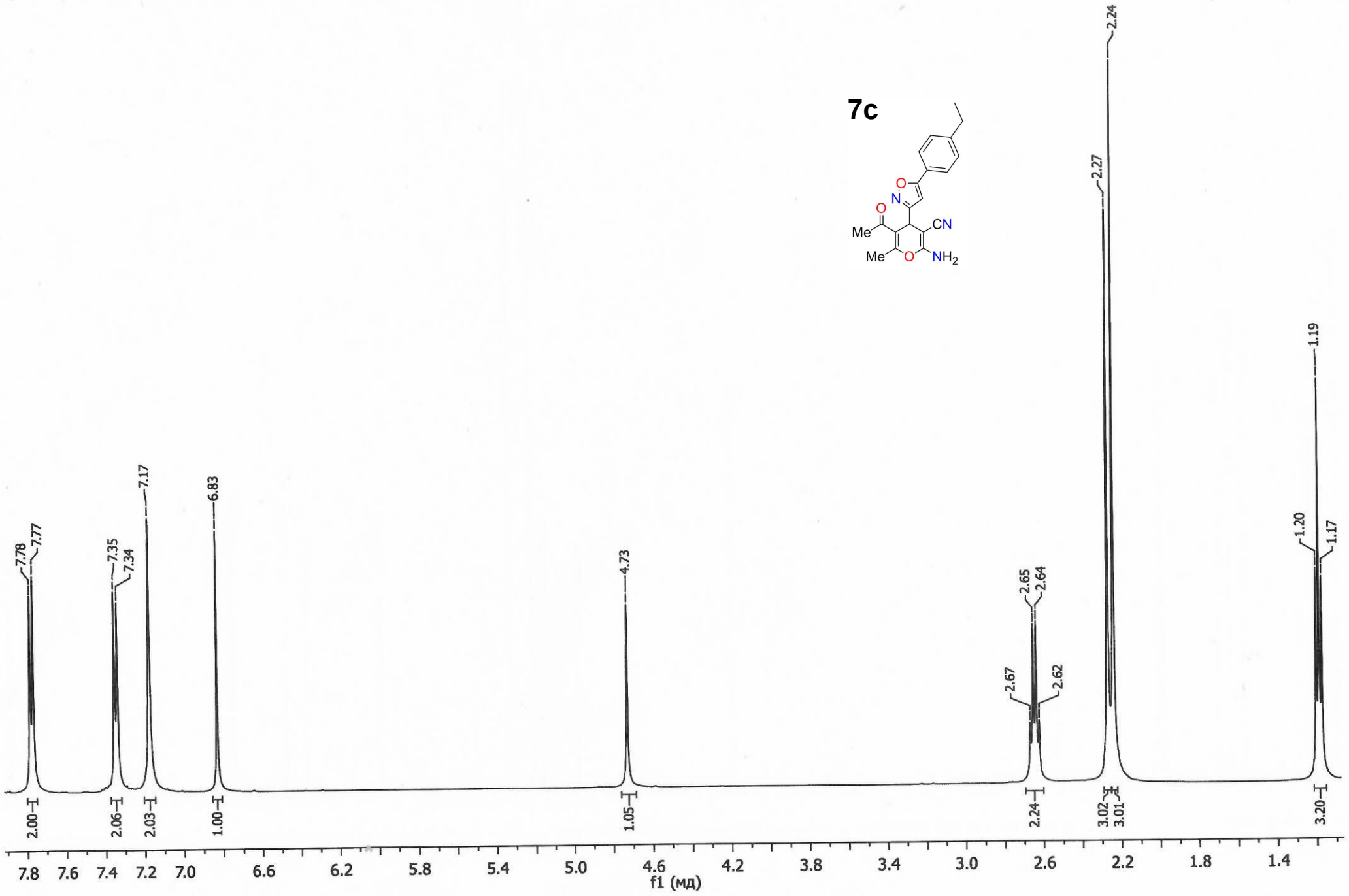
m/z	z	Abund
177.1	1	1503561.25
336.2	1	1008723.69
358.1	1	1761692.75
359.1	1	339073.28
374.1	1	214288.27
522.9		513855.19
693.3	1	3321244.5
694.3	1	1316276.88
695.3	1	288386.06
733.2		271985.53

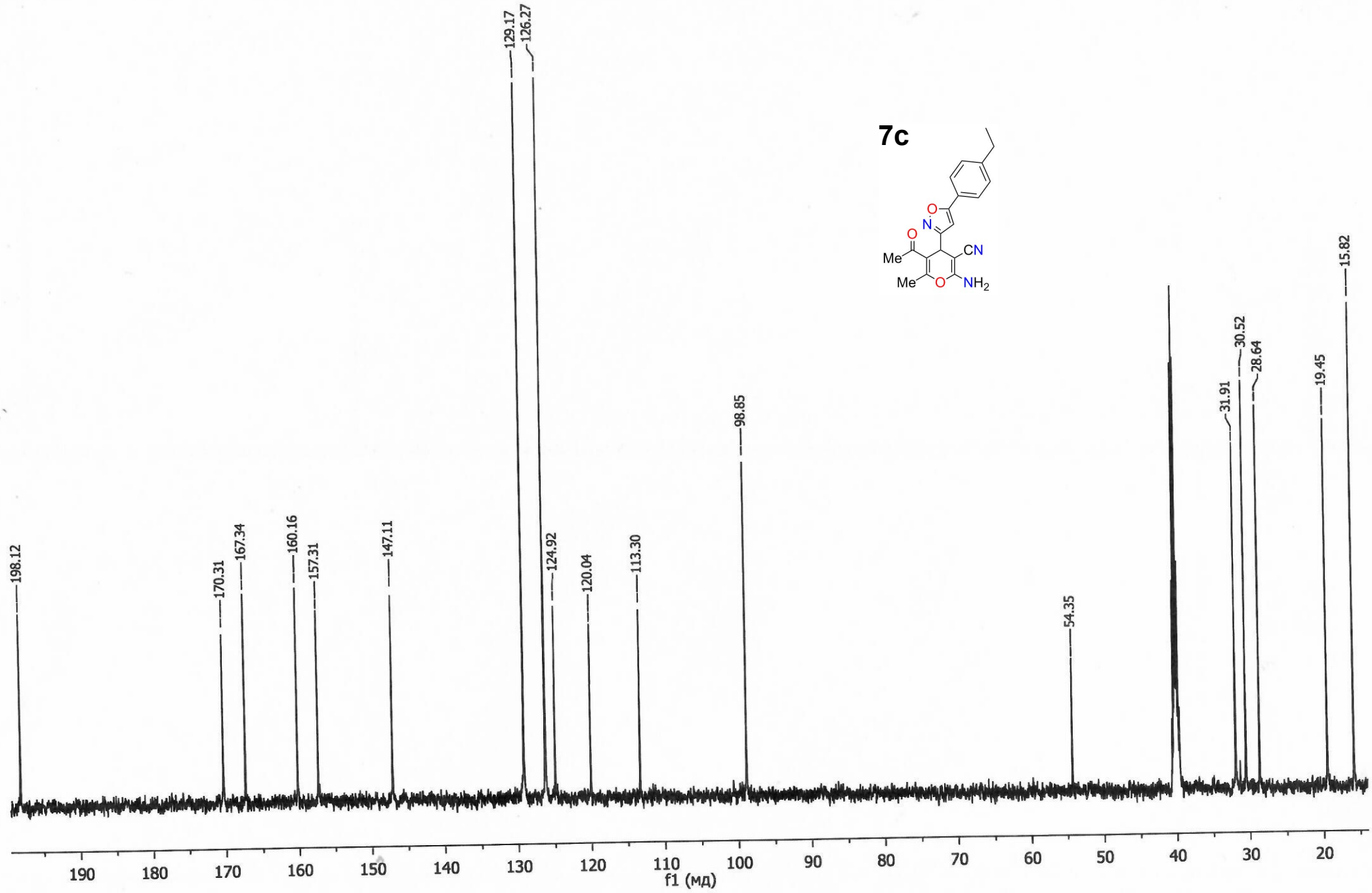
Spectrum Source
Peak (1) in "DAD1 - A:Sig=215,4 Ref=off"



--- End Of Report ---

^{1}H NMR (500 MHz, DMSO) δ = 7.77 (d, *J*=8.1, 1H), 7.35 (d, *J*=8.1, 1H), 2.64 (q, *J*=7.5, 1H), 1.19 (t, *J*=7.6, 1H).




7c
O
N
O
Me
CN
Me
O
NH2
198.12
170.31
167.34
160.16
157.31
147.11
129.17
126.27
124.92
120.04
113.30
98.85
54.35
31.91
30.52
28.64
19.45
15.82
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (мд)

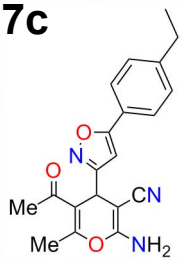
Qualitative Analysis Report

Data Filename	I-738_01.d	**Sample Name**	I-738
Sample Type	Sample	**Position**	Vial 2
Instrument Name	Instrument 1	**User Name**	
Acq Method	All_2021_kol 1-2.m	**Acquired Time**	5/29/2023 11:31:04 AM
IRM Calibration Status	Not Applicable	**DA Method**	Defaul1t.m
Comment			

Sample Group
Stream Name LC 1

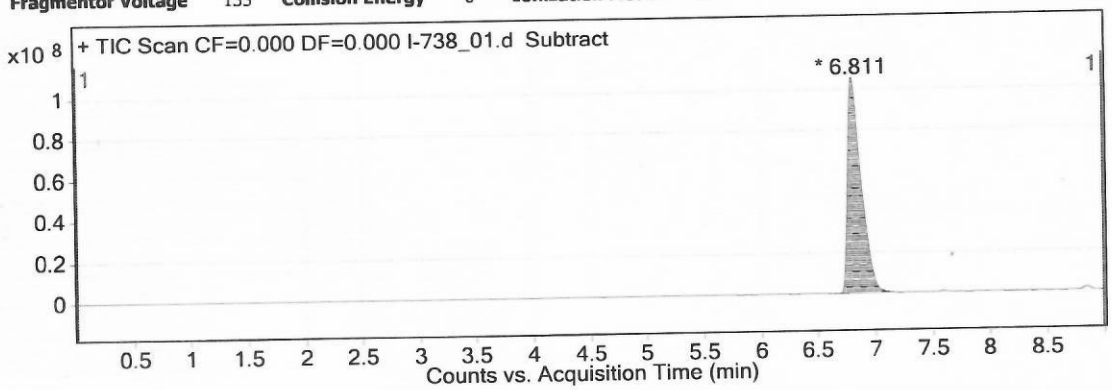
Info.
Acquisition SW Version 6400 Series Triple Quadrupole 10.0 (127)

7c



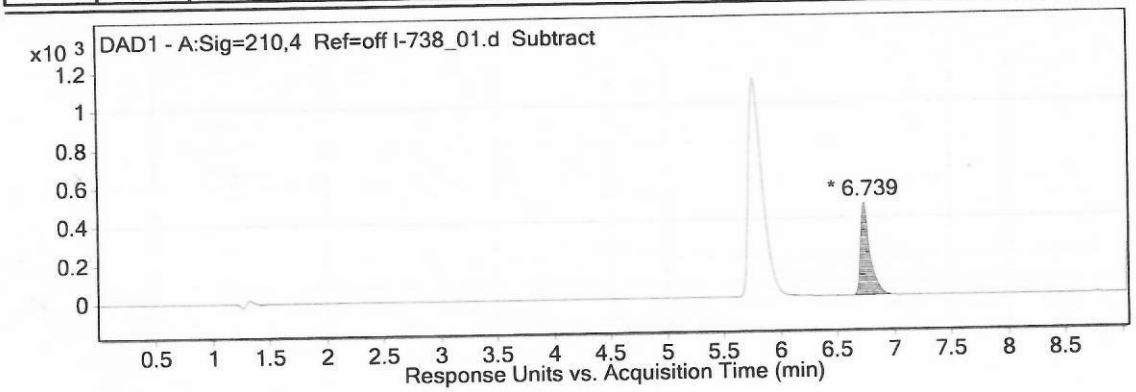
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	6,693	6,811	7,123	105782930,5	853859858,8	100



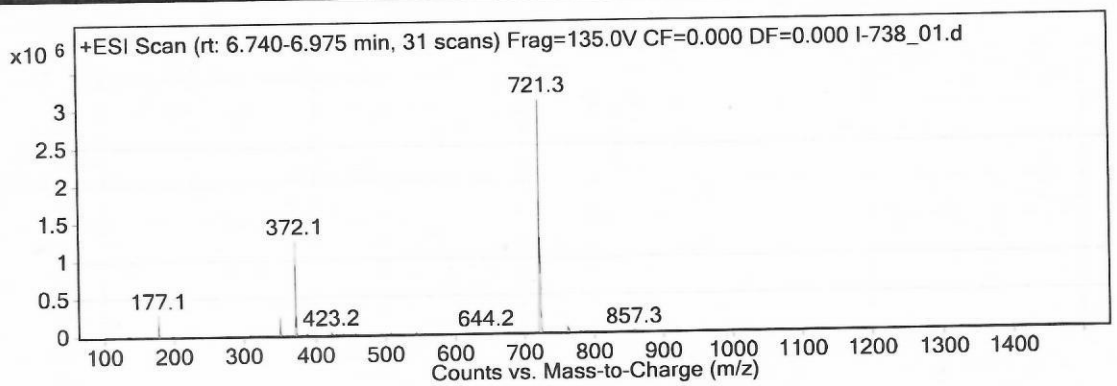
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	6,652	6,739	7,005	476,01	2591,32	100

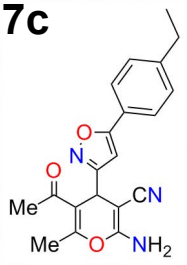
User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan Sub"	135	0	ESI

Qualitative Analysis Report



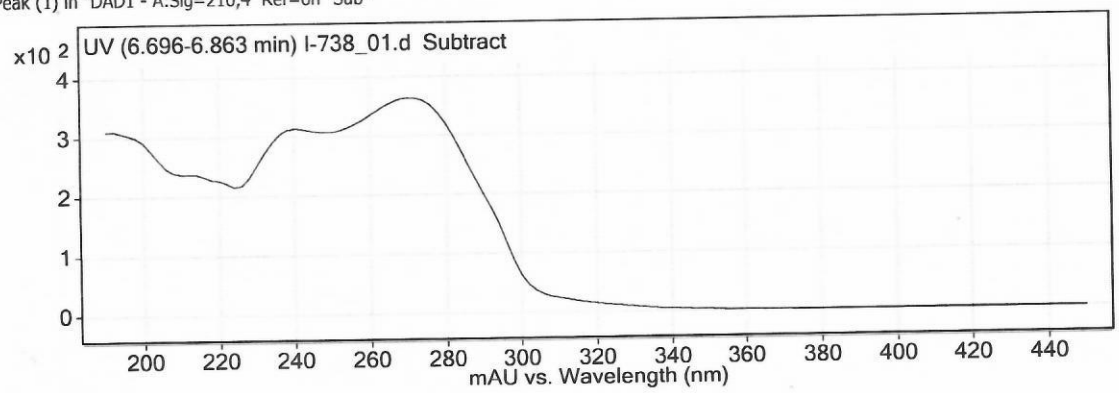
7c



Peak List

m/z	z	Abund
177.1	1	282175.72
350.2	1	253849.27
351.2	1	57109.17
372.1	1	1256493.88
373.1	1	259610.81
721.3	1	3099698.5
722.3	1	1264411.5
723.3	1	292381.69
724.3	1	51502.52
761.2		78049.34

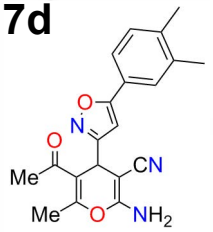
Spectrum Source
Peak (1) in "DAD1 - A:Sig=210,4 Ref=off Sub"



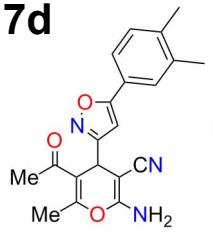
--- End Of Report ---

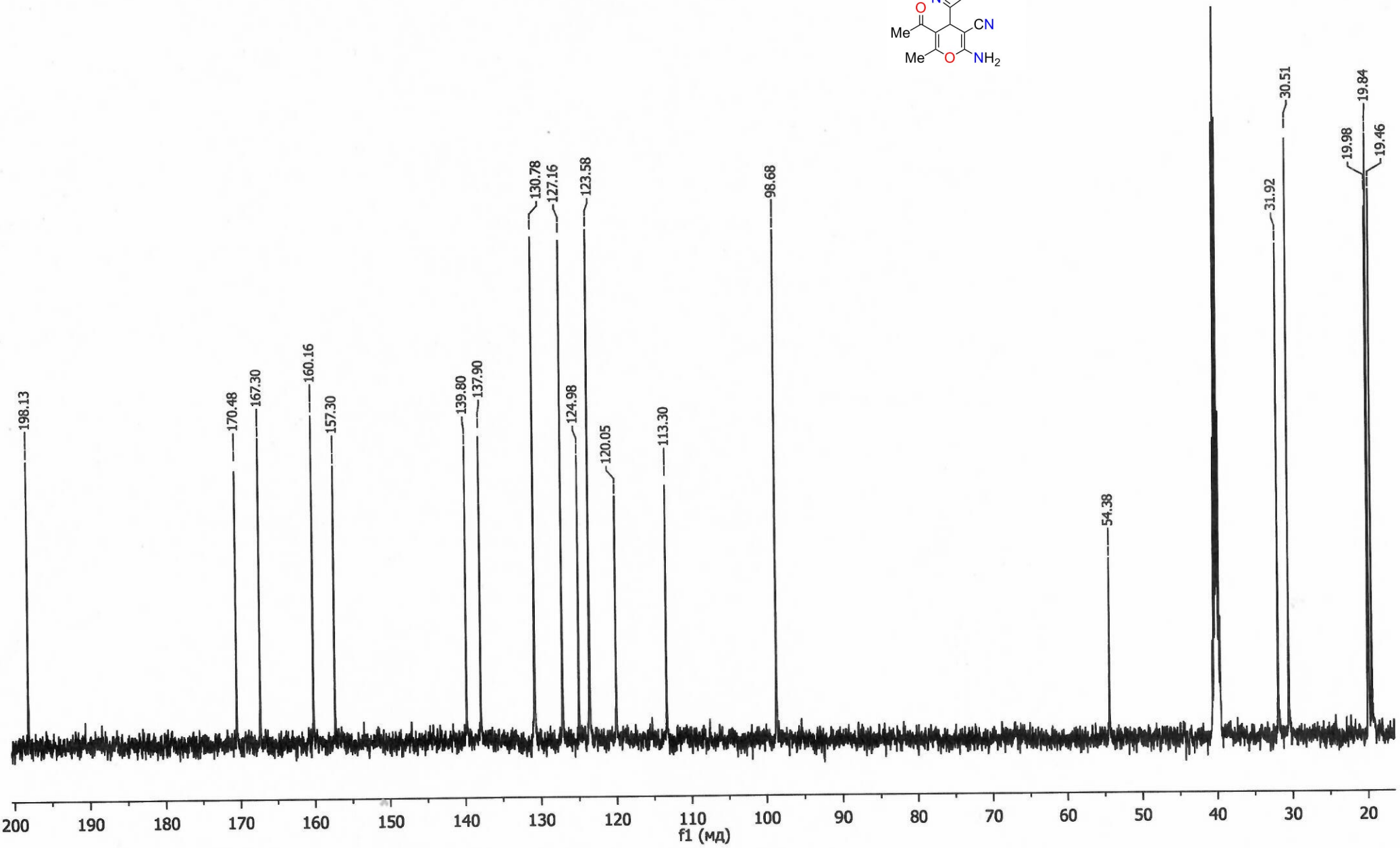
^{1}H NMR (500 MHz, DMSO) δ = 7.57 (d, *J*=7.7, 1H), 7.26 (d, *J*=7.9, 1H).

7d



7d

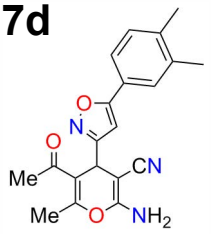

O
N
O
Me
CN
Me
O
NH2


198.13
170.48
167.30
160.16
157.30
139.80
137.90
130.78
127.16
124.98
123.58
120.05
113.30
98.68
54.38
31.92
30.51
19.98
19.84
19.46
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (мд)

Qualitative Analysis Report

Data Filename	I-737_01.d	**Sample Name**	I-737
Sample Type	Sample	**Position**	Vial 2
Instrument Name	Instrument 1	**User Name**	
Acq Method	All_2021_kol 1-2.m	**Acquired Time**	5/29/2023 11:15:48 AM
IRM Calibration Status	Not Applicable	**DA Method**	Defaul1t.m
Comment			

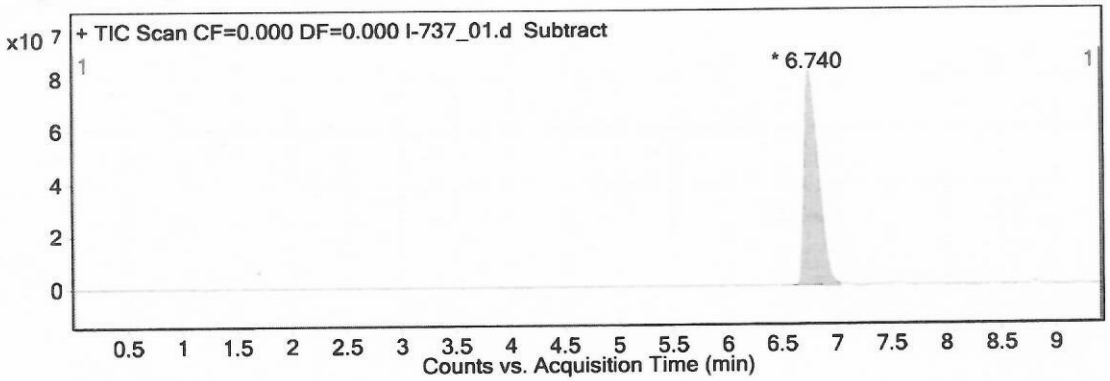
7d



Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition SW Version**	6400 Series Triple Quadrupole 10.0 (127)

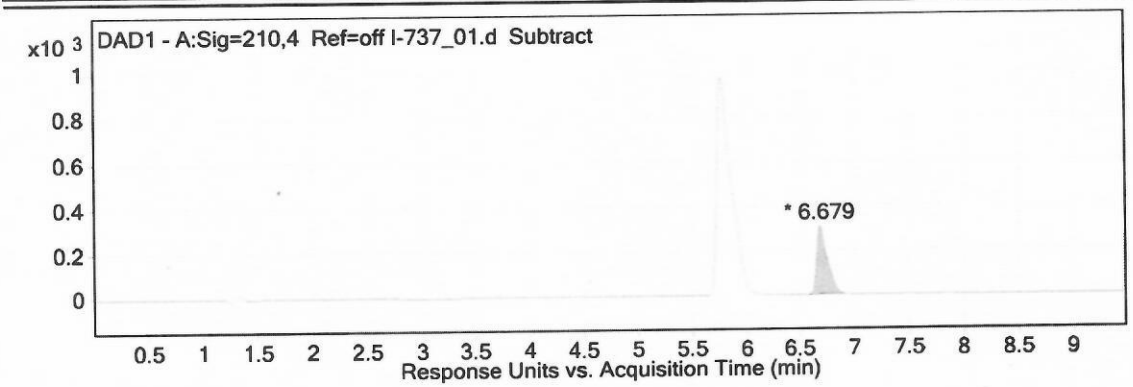
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	6,592	6,74	7,029	81612543,93	712732304,9	100



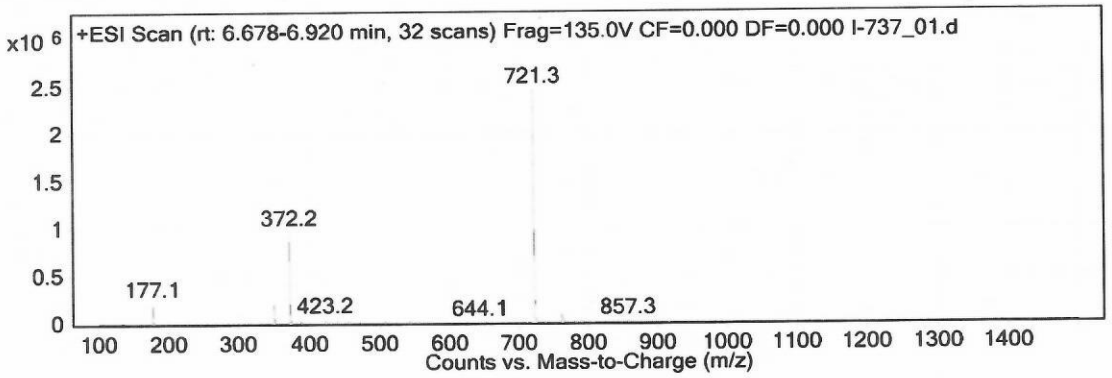
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	6,579	6,679	6,905	306,04	2022,04	100

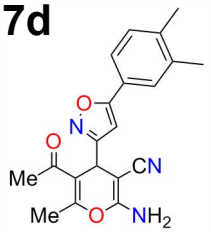
User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan Sub"	135	0	ESI

Qualitative Analysis Report



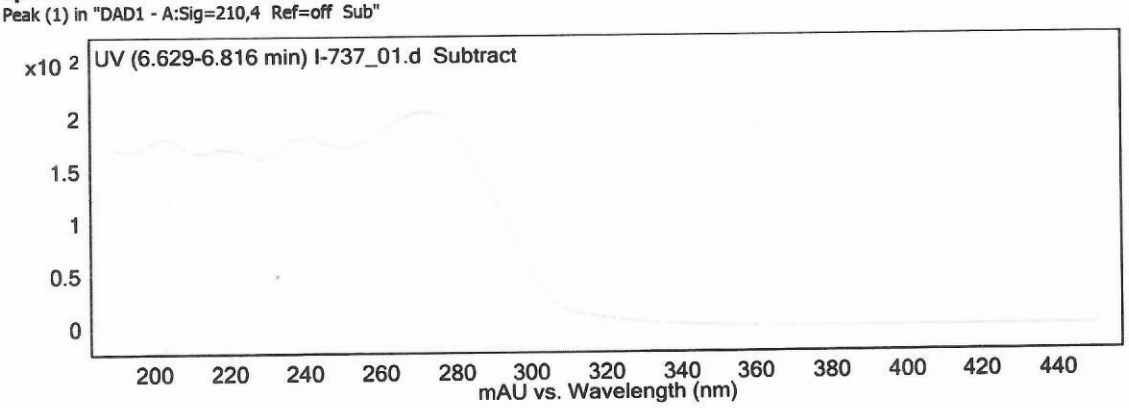
7d



Peak List

m/z	z	Abund
177.1	1	216357.47
350.2	1	211756.36
351.2	1	49207.43
372.2	1	961951.19
373.2	1	207183.81
721.3	1	2457911.5
722.3	1	1003938
723.3	1	232311.84
761.3	1	83831.52
763.3	1	46807.84

Spectrum Source
Peak (1) in "DAD1 - A:Sig=210,4 Ref=off Sub"

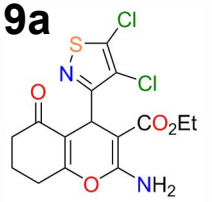


--- End Of Report ---

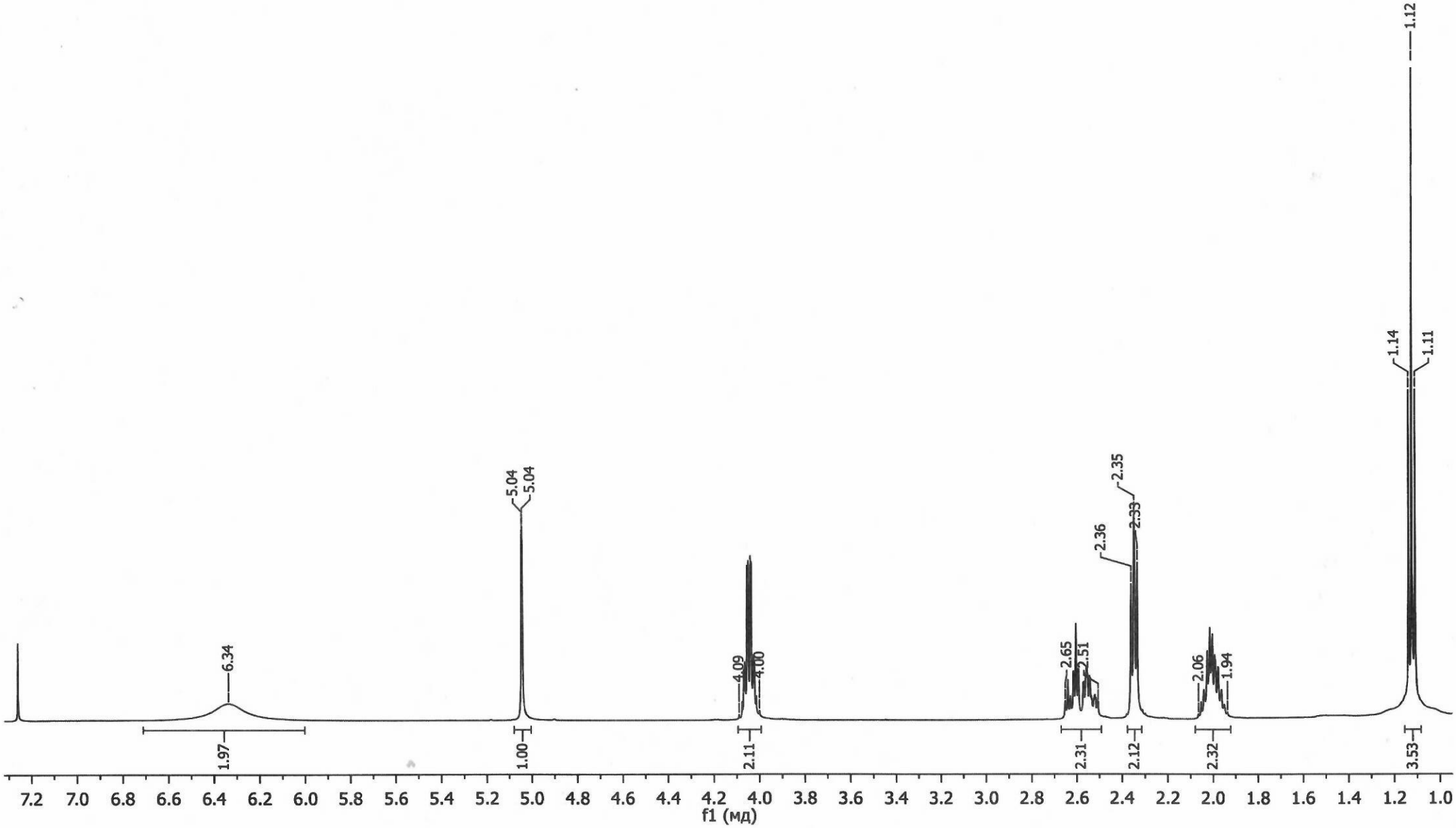
SK_i709_12212022

bbo_1H_bar CDCl3 /v nmrsu 14

9a



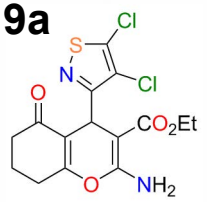
^{1}H NMR (500 MHz, $CDCl_3$) δ = 5.04 (d, *J*=0.8, 1H), 4.05 (m, 1H), 2.35 (m, 1H), 1.12 (t, *J*=7.1, 2H).

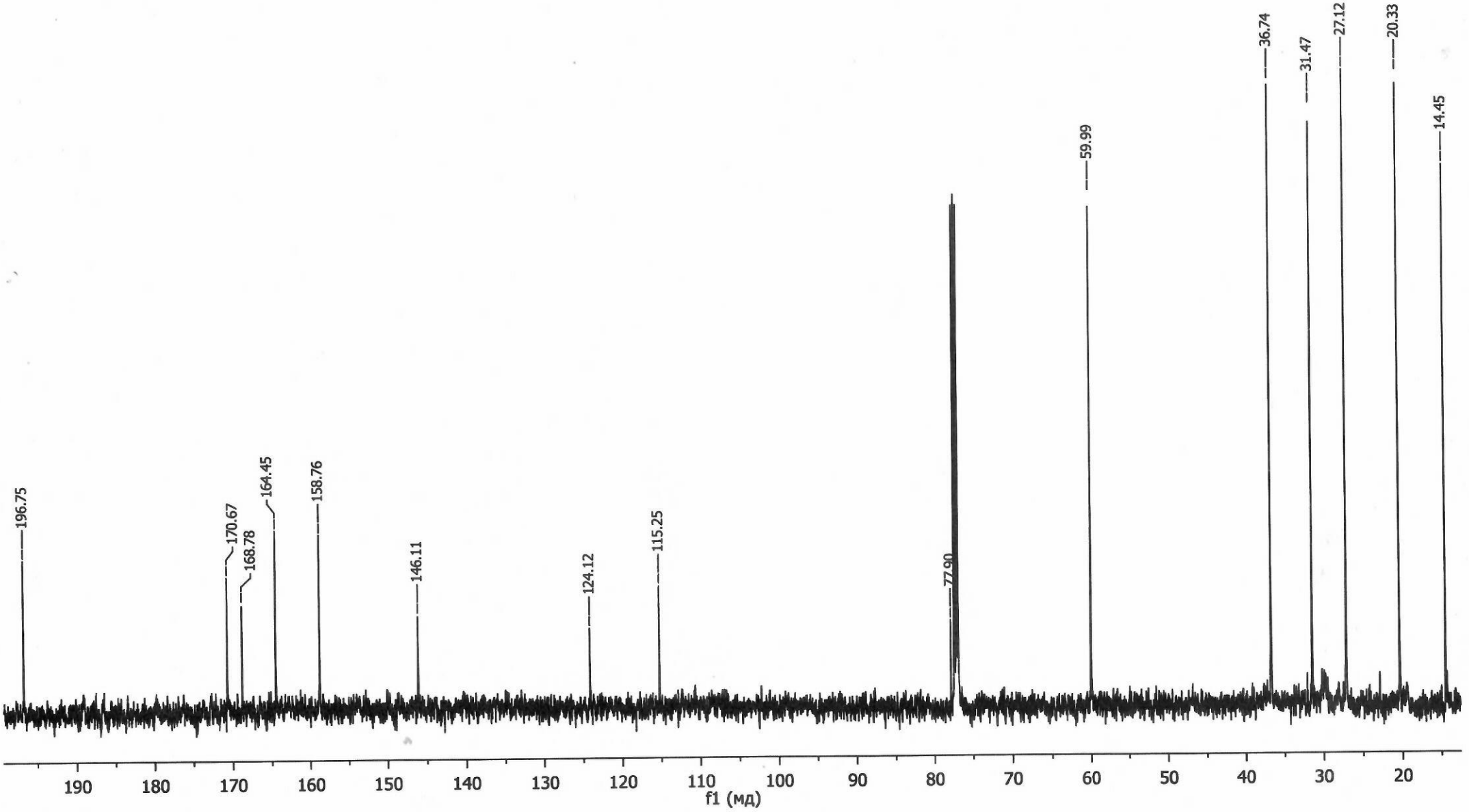


SK_i709_12212022

bbo_13CF_bar CDCl3 /v nmrsu 14

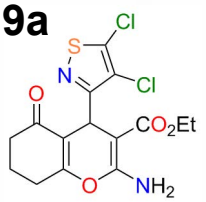
9a





Qualitative Analysis Report

9a

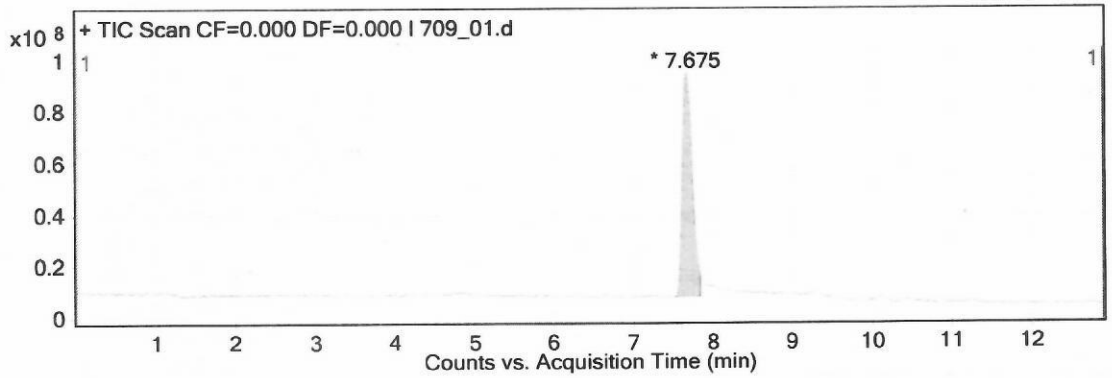


Data Filename	I 709_01.d	**Sample Name**	I 709
Sample Type	Sample	**Position**	Vial 2
Instrument Name	Instrument 1	**User Name**	
Acq Method	All_2021_kol 1-2.m	**Acquired Time**	3/30/2023 11:51:58 AM
IRM Calibration Status	Not Applicable	**DA Method**	Defaul1t.m
Comment			

Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition SW Version**	6400 Series Triple Quadrupole 10.0 (127)

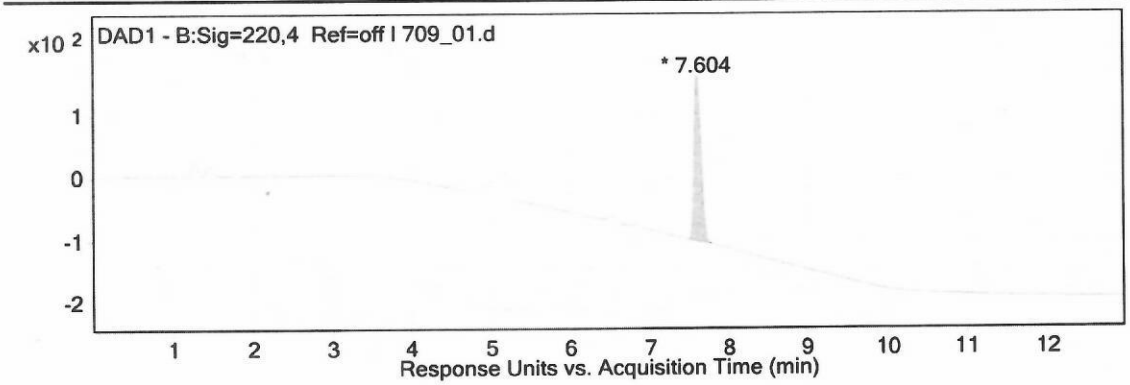
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,514	7,675	7,842	86625862,85	748713142,6	100



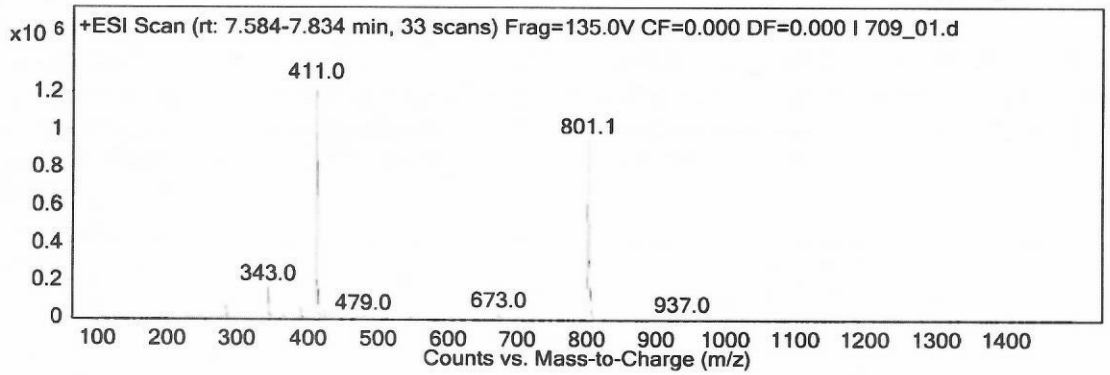
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,484	7,604	7,784	263,68	1519,14	100

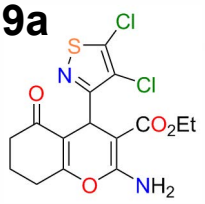
User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan"	135	0	ESI

Qualitative Analysis Report



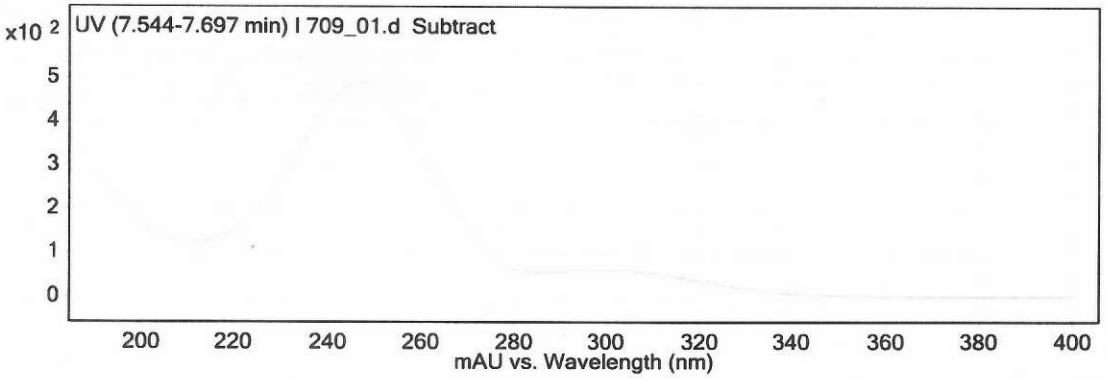
9a



Peak List

m/z	z	Abund
343	1	166750.39
411	1	1236359.63
412	1	208687.8
413	1	867368.81
415	1	166961.47
799.1		638002.06
801.1	1	946129.81
802.1	1	317153.31
803.1	1	534421.06
804.1	1	174360.19

Spectrum Source
Peak (1) in "DAD1 - B:Sig=220,4 Ref=off"



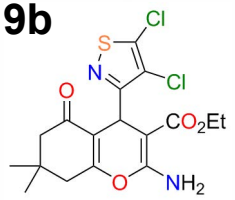
--- End Of Report ---

SK_i705_12142022

bbo_1H_bar CDCl3 /v nmrsu 3

^{1}H NMR (500 MHz, $CDCl_3$) δ = 4.06 (q, *J*=7.0, 1H), 2.21 (dd, *J*=39.9, 16.2, 1H), 1.14 (t, *J*=7.1, 1H).

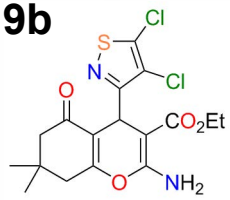
9b

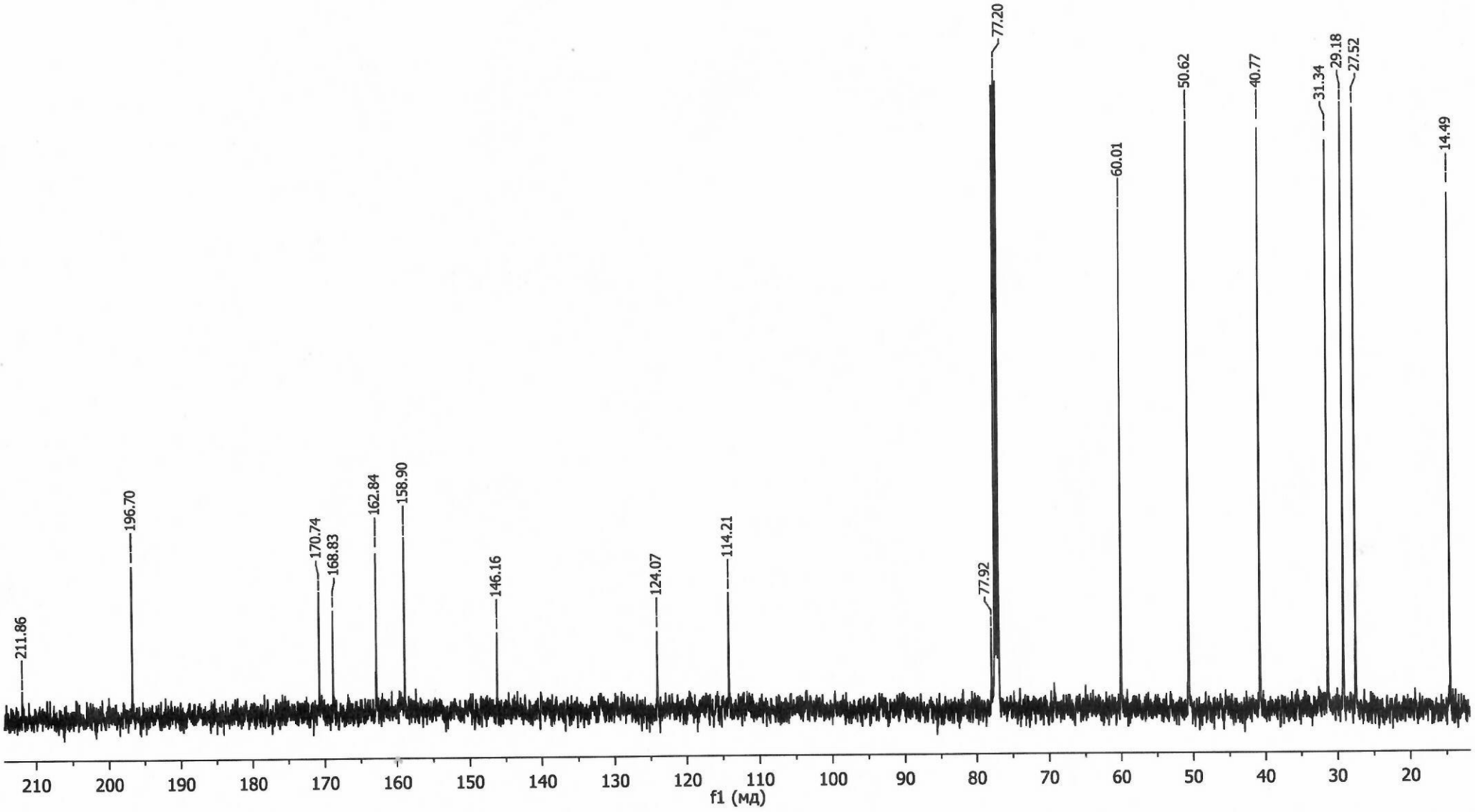


SK_i705_12142022

bbo_13CF_bar CDCl3 /v nmrsu 3

9b

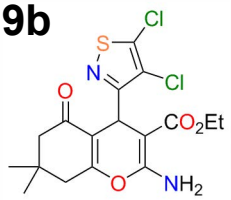




Qualitative Analysis Report

Data Filename	I-705_01.d	**Sample Name**	I-705
Sample Type	Sample	**Position**	Vial 2
Instrument Name	Instrument 1	**User Name**	
Acq Method	All_2021_kol 1-2.m	**Acquired Time**	12/14/2022 10:04:16 AM
IRM Calibration Status	Not Applicable	**DA Method**	Defaul1t.m
Comment			

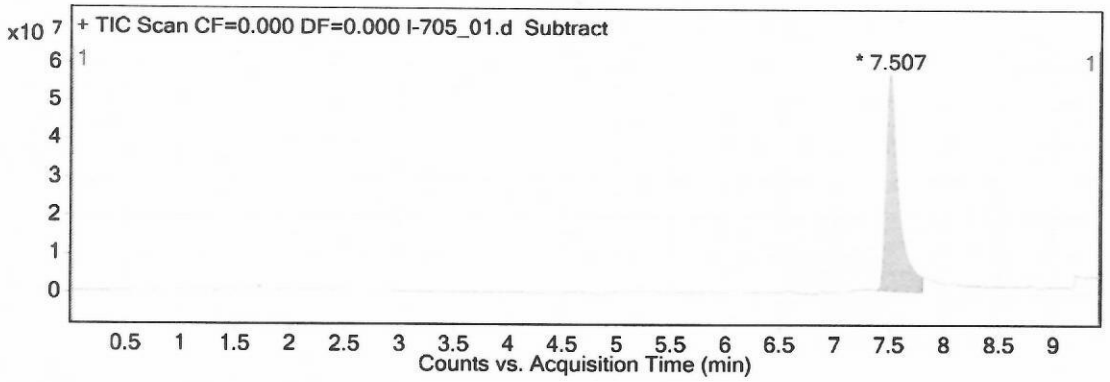
9b



Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition SW Version**	6400 Series Triple Quadrupole 10.0 (127)

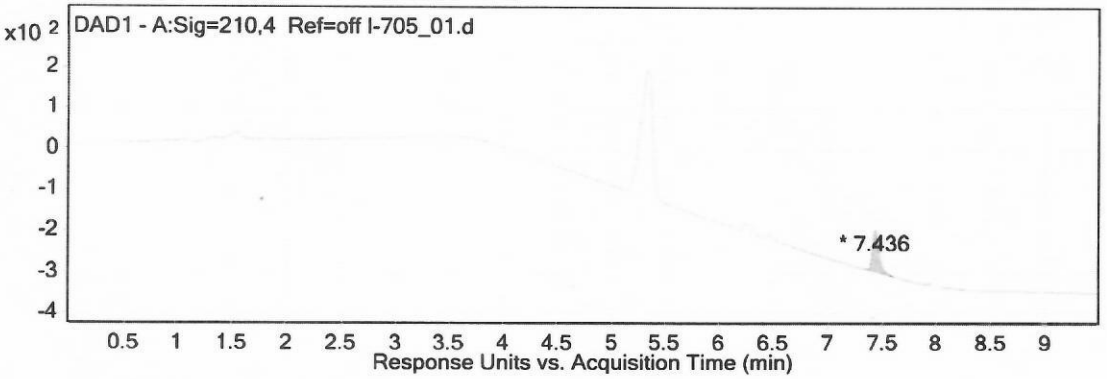
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,395	7,507	7,806	57290314,21	453619140,4	100



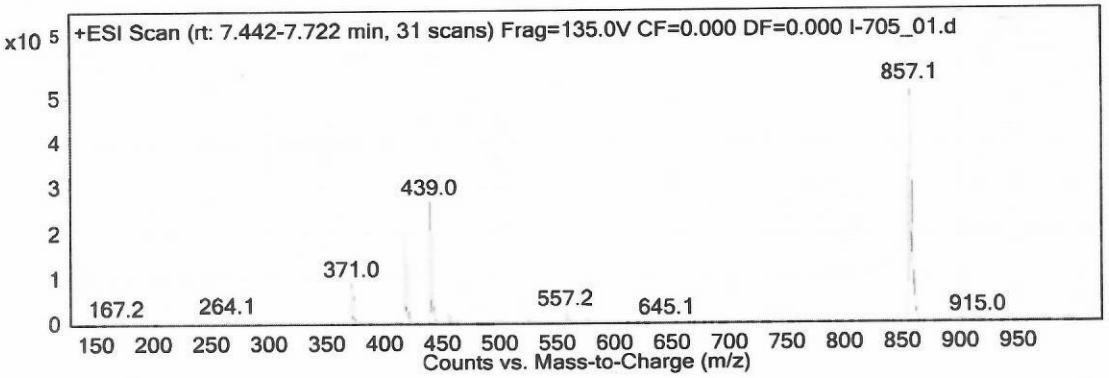
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,362	7,436	7,616	98,98	404,15	100

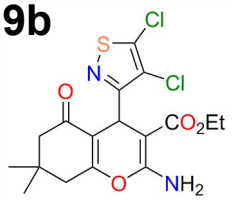
User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan Sub"	135	0	ESI

Qualitative Analysis Report



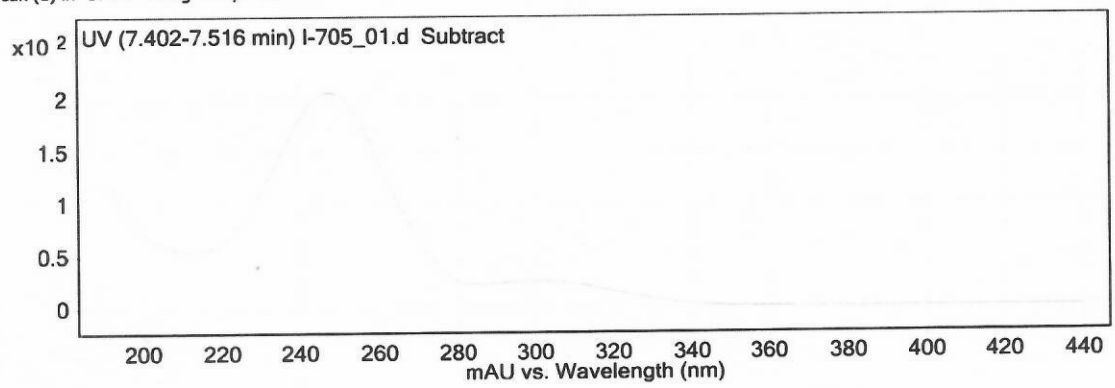
9b



Peak List

m/z	z	Abund
417.1	1	184456.27
419		132333.55
439	1	268196.84
441	1	196784.38
855.1		340666.22
857.1	1	519135.03
858.1	1	199419
859.1	1	308212.75
860.1	1	111508.02
861.1	1	91189.52

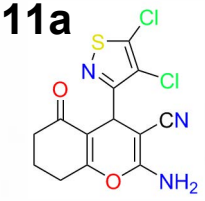
Spectrum Source
Peak (1) in "DAD1 - A:Sig=210,4 Ref=off"



--- End Of Report ---

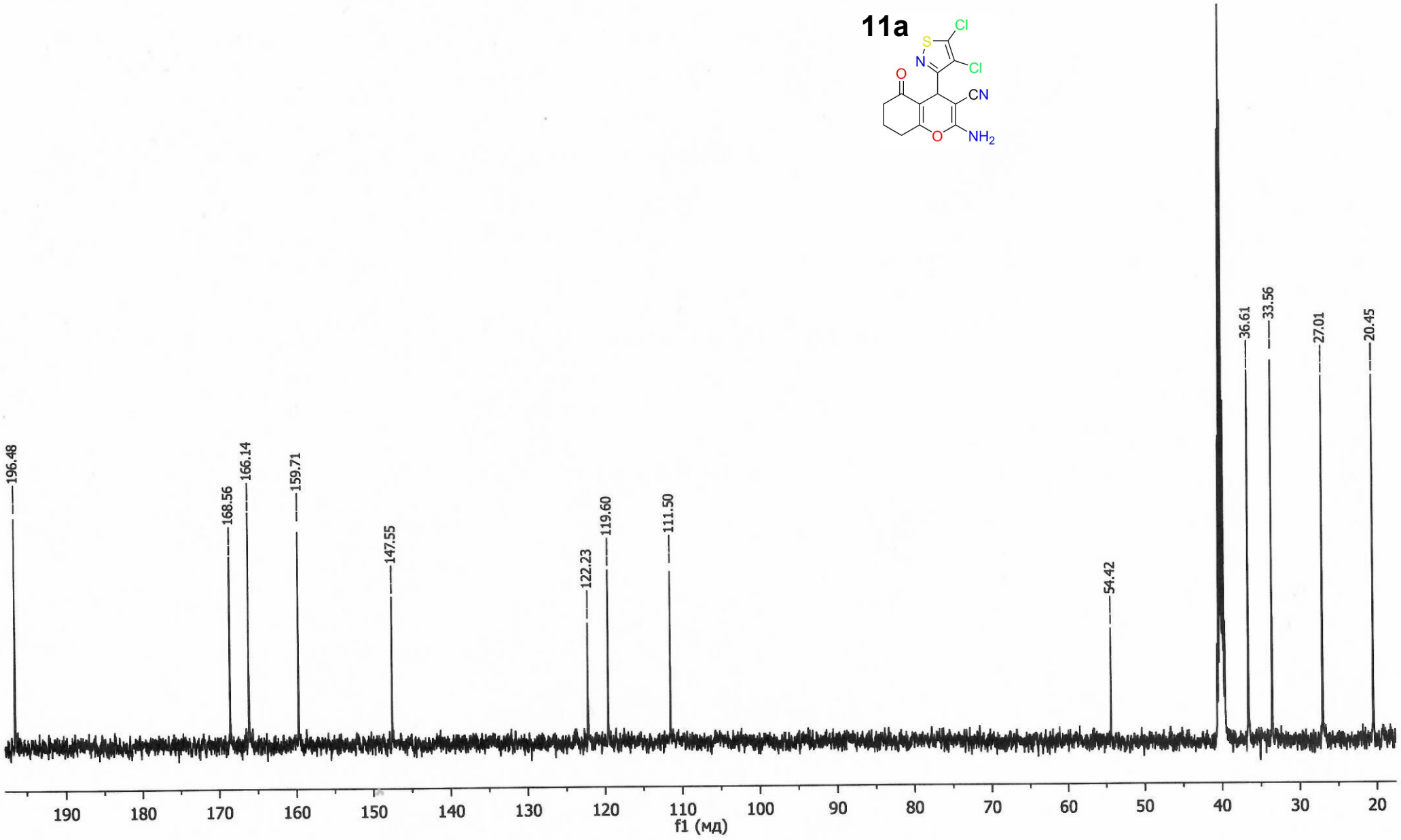
^{1}H NMR (500 MHz, DMSO) δ = 4.36 (t, *J*=4.8, 1H), 2.61 (t, *J*=5.9, 2H).

11a



SK_i723_03222023

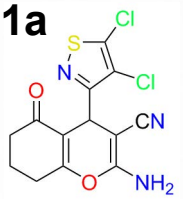
bbo_13CF_bar DMSO /v nmrsu 7



Qualitative Analysis Report

Data Filename	I 723_01.d	**Sample Name**	I 723
Sample Type	Sample	**Position**	Vial 3
Instrument Name	Instrument 1	**User Name**	
Acq Method	All_2021_kol 1-2.m	**Acquired Time**	3/30/2023 12:37:20 PM
IRM Calibration Status	Not Applicable	**DA Method**	Defaul1t.m
Comment			

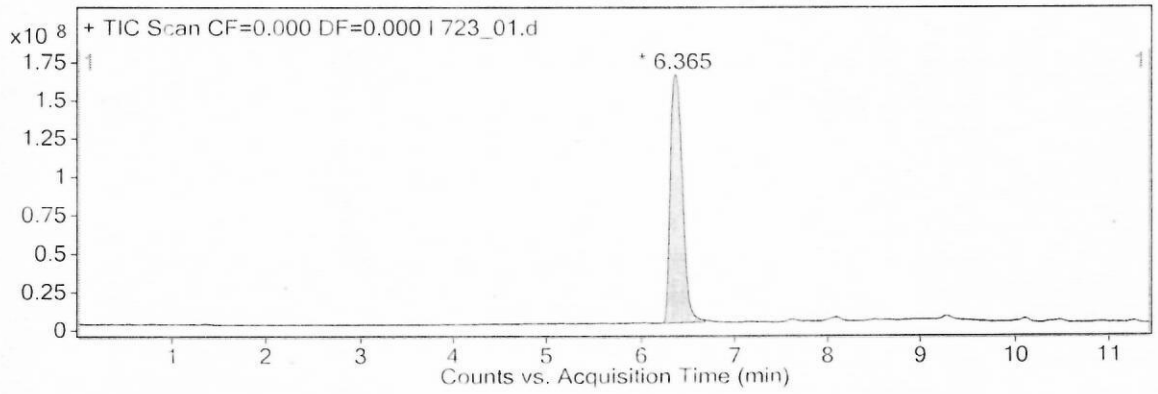
11a



Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition SW Version**	6400 Series Triple Quadrupole 10.0 (127)

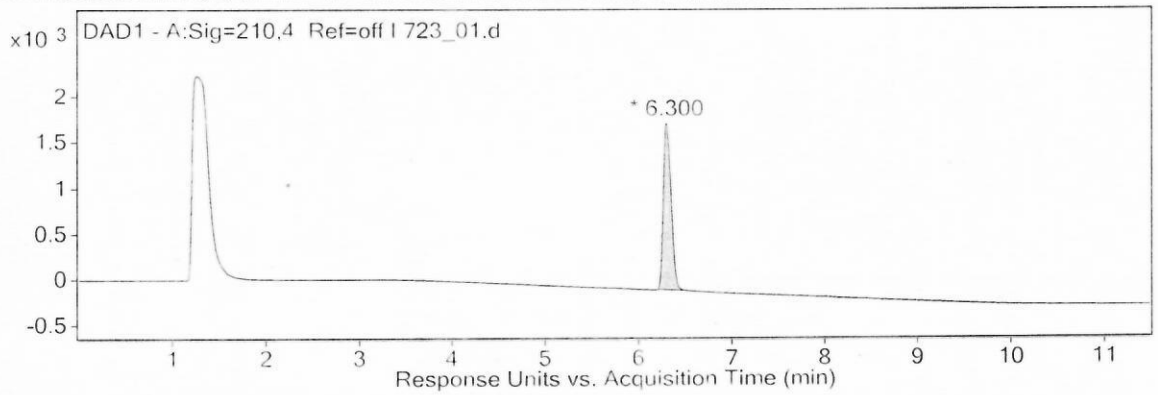
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	6,194	6,365	6,686	161949331,8	1450839461	100



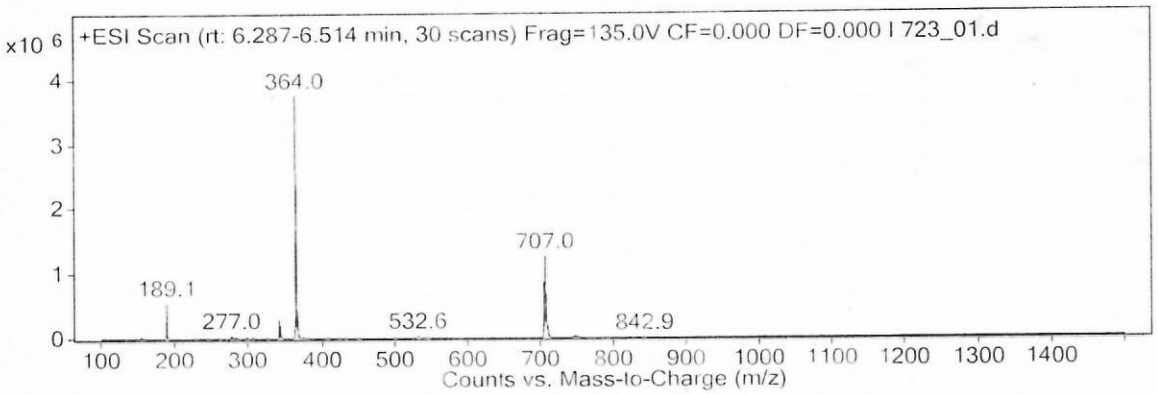
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	6,207	6,3	6,507	1818,76	10449,9	100

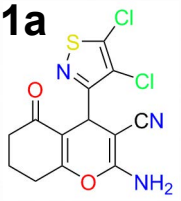
User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan"	135	0	ESI

Qualitative Analysis Report



11a



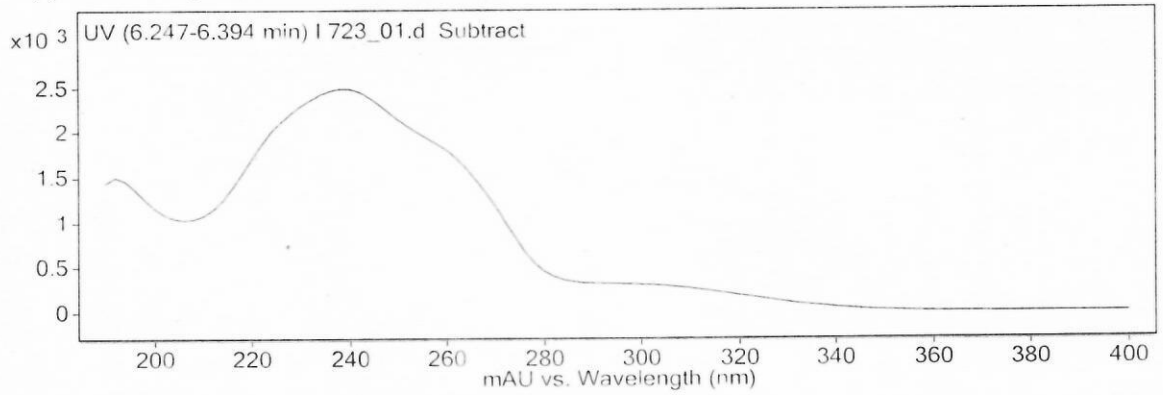
Peak List

m/z	z	Abund
189.1	1	544201.94
364	1	3760455.75
365	1	498165.03
366	1	2555208
367	1	341198.34
368	1	426182.44
705		844164.31
707		1249904.13
708.1		362644.34
709	1	674271.63

$[M+Na]^+$

Spectrum Source

Peak (1) in "DAD1 - A:Sig=210,4 Ref=off"



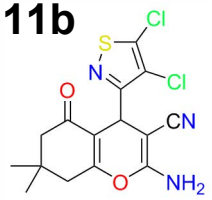
--- End Of Report ---

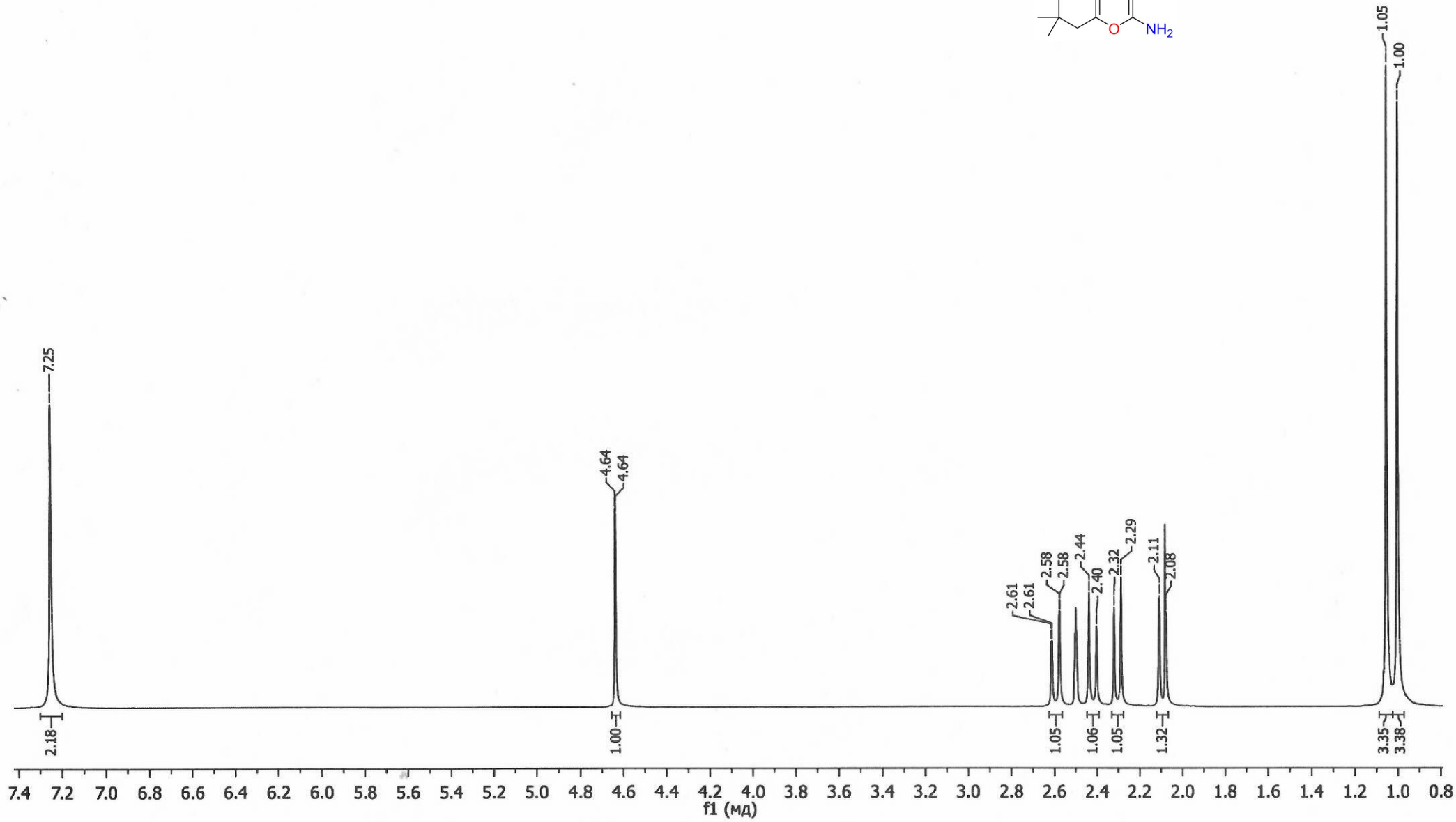
SK_i722_03222023

bbo_1H_bar DMSO /v nmrsu 5

^{1}H NMR (500 MHz, DMSO) δ = 2.59 (dd, *J*=17.6, 1.2, 1H), 2.42 (d, *J*=17.6, 1H), 2.30 (d, *J*=16.1, 1H), 2.09 (d, *J*=15.8, 1H).

11b

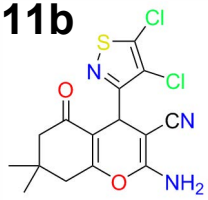




SK_i722_03222023

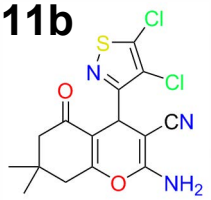
bbo_13CF_bar DMSO /v nmrsu 5

11b



Qualitative Analysis Report

11b

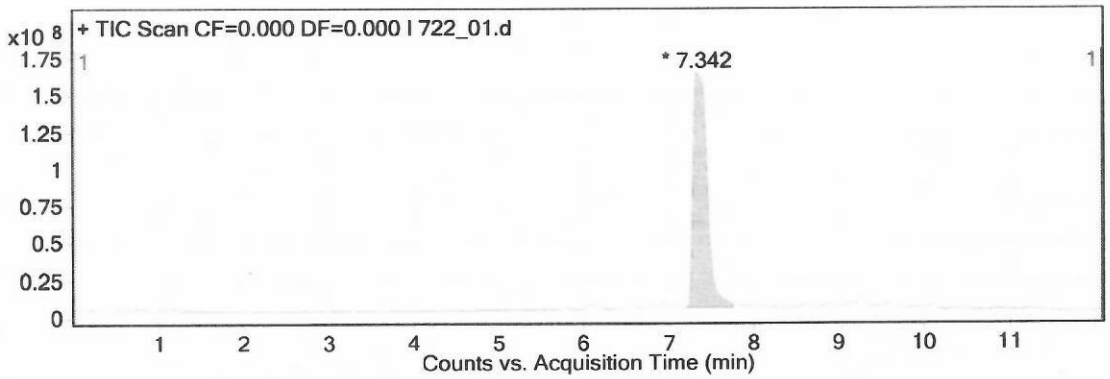


Data Filename	I 722_01.d	**Sample Name**	I 722
Sample Type	Sample	**Position**	Vial 2
Instrument Name	Instrument 1	**User Name**	
Acq Method	All_2021_kol 1-2.m	**Acquired Time**	3/30/2023 12:15:31 PM
IRM Calibration Status	Not Applicable	**DA Method**	Defaul1t.m
Comment			

Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition SW Version**	6400 Series Triple Quadrupole 10.0 (127)

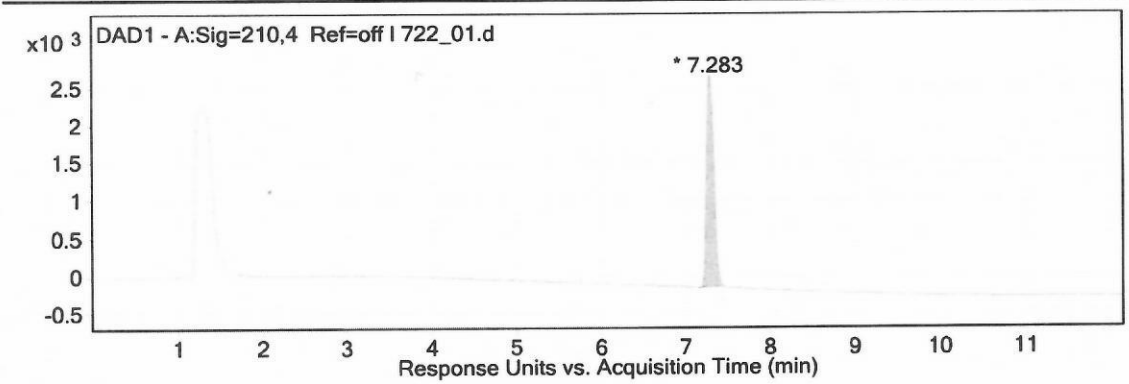
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,209	7,342	7,756	158970777,8	1769827340	100



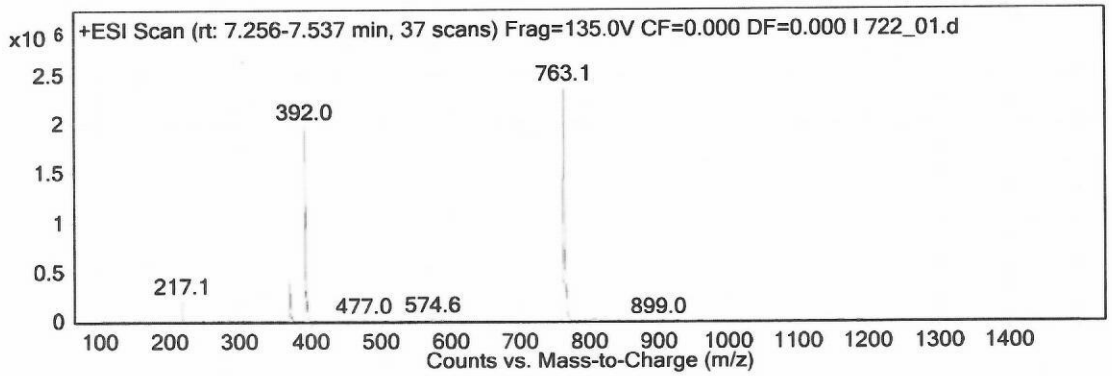
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,156	7,283	7,449	2805,48	16589,06	100

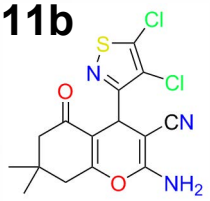
User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan"	135	0	ESI

Qualitative Analysis Report



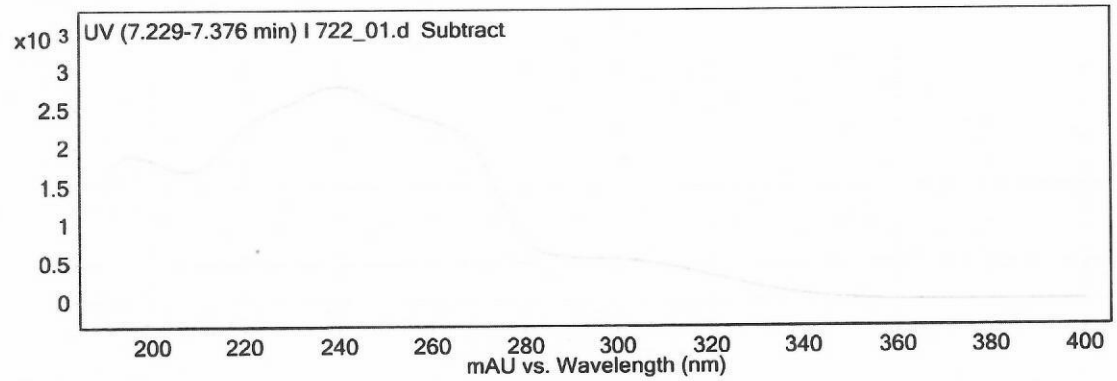
11b



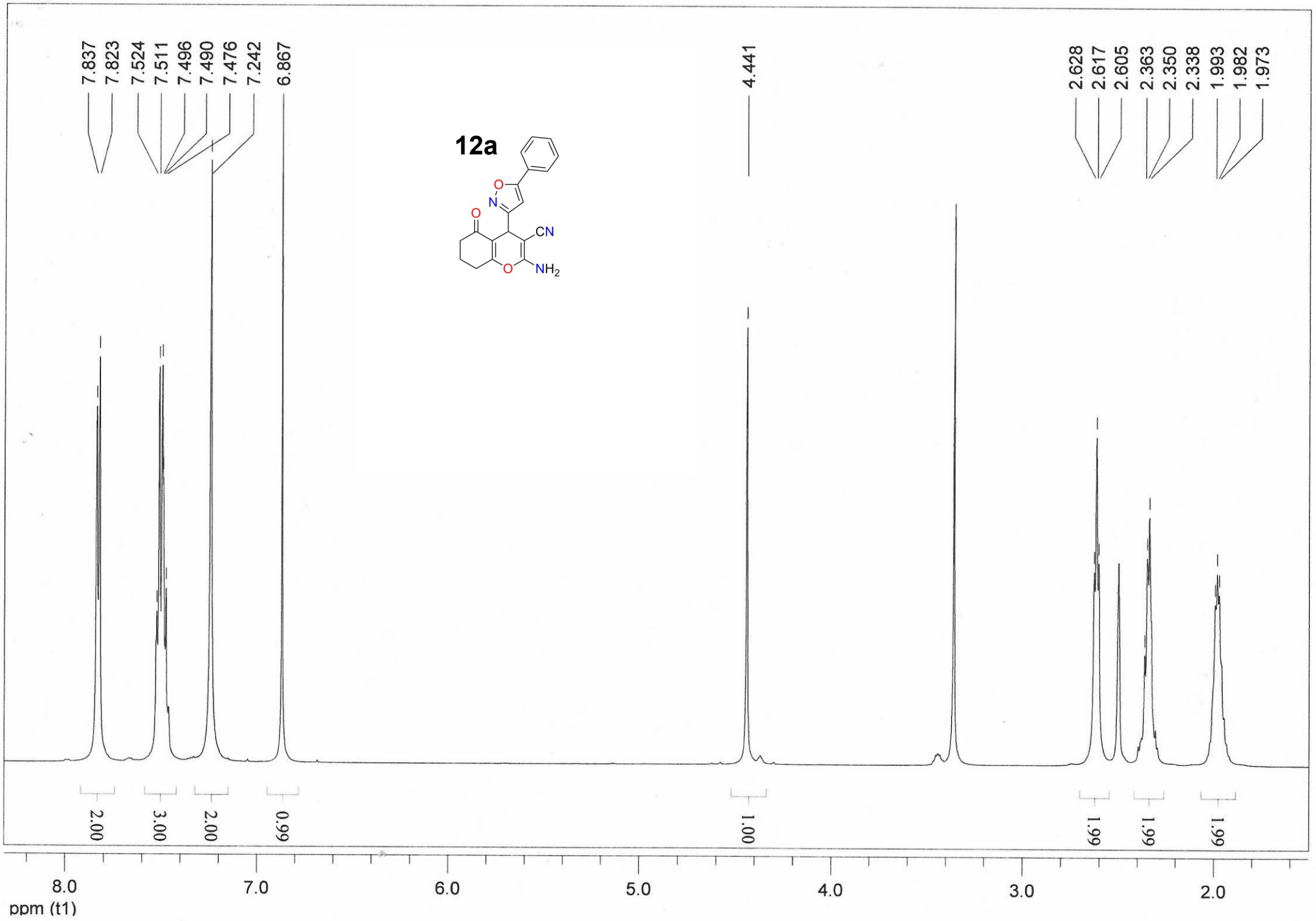
Peak List

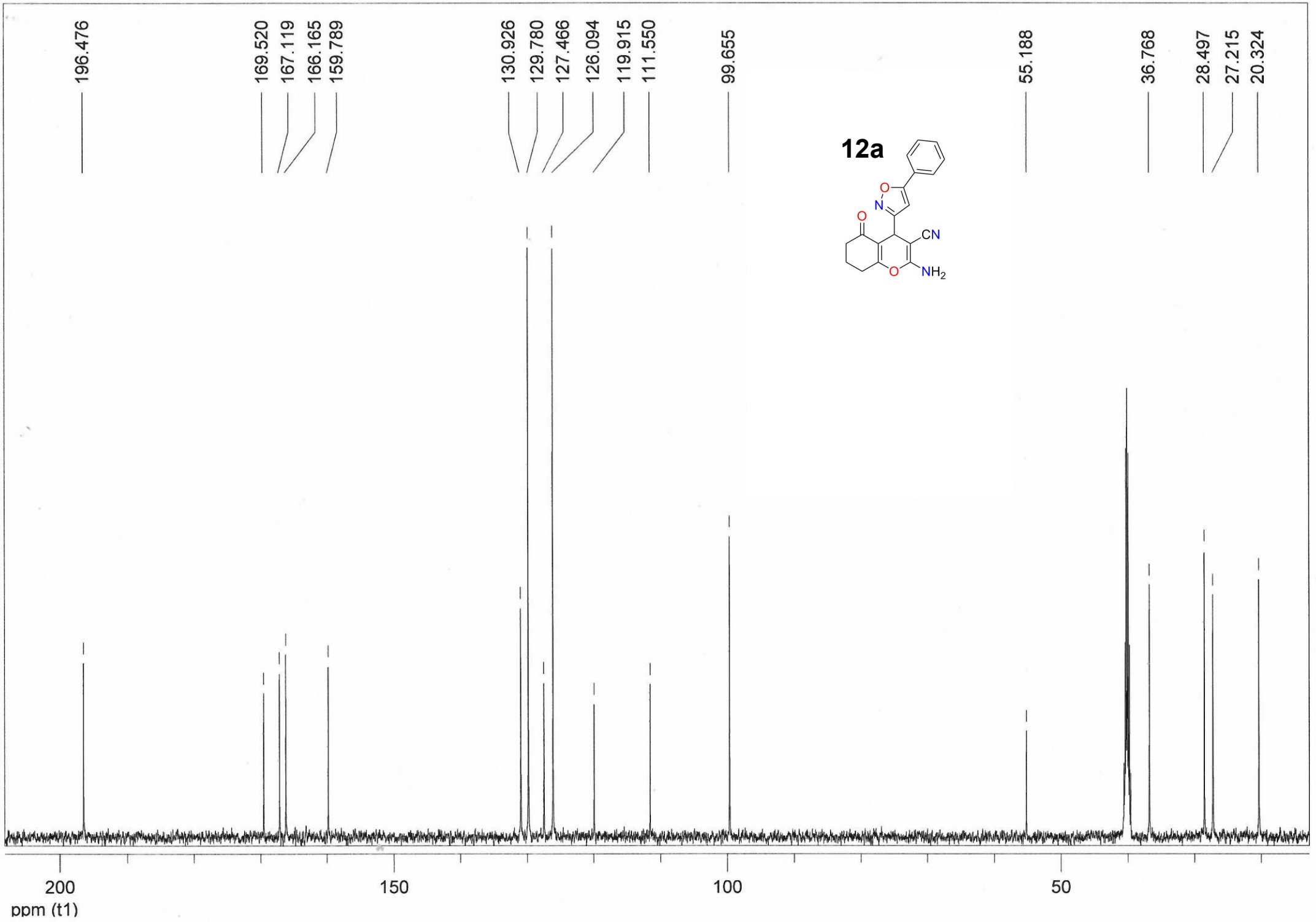
m/z	z	Abund
370.1	1	432107.47
392		1984513.75
393.1		316456.5
394	1	1359416.75
761.1		1550426
763.1	1	2371739.25
764.1	1	725546.06
765.1	1	1246296.5
766.1	1	382616.53
767.1	1	330915.34

Spectrum Source
Peak (1) in "DAD1 - A:Sig=210,4 Ref=off"



--- End Of Report ---

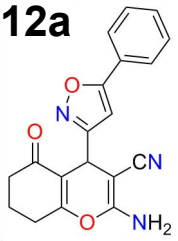

12a
7.837
7.823
7.524
7.511
7.496
7.490
7.476
7.242
6.867
4.441
2.628
2.617
2.605
2.363
2.350
2.338
1.993
1.982
1.973
O
N
O
CN
O
NH2
2.00
3.00
2.00
0.99
1.00
1.99
1.99
1.99
8.0
7.0
6.0
5.0
4.0
3.0
2.0
ppm (t1)


196.476
169.520
167.119
166.165
159.789
130.926
129.780
127.466
126.094
119.915
111.550
99.655
55.188
36.768
28.497
27.215
20.324
12a
O
N
O
O
CN
NH2
200
150
100
50
ppm (t1)

Qualitative Analysis Report

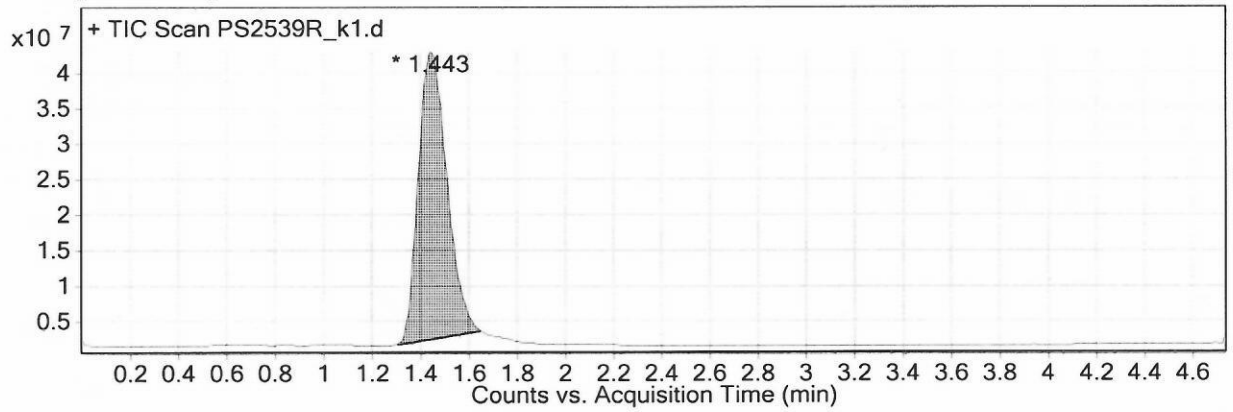
Data Filename	PS2539R_k1.d	**Sample Name**	Unavailable
Sample Type	Unavailable	**Position**	Unavailable
Instrument Name	Unavailable	**User Name**	Unavailable
Acq Method		**IRM Calibration Status**	Success
DA Method	Default.m	**Comment**	Sample information is unavailable

12a



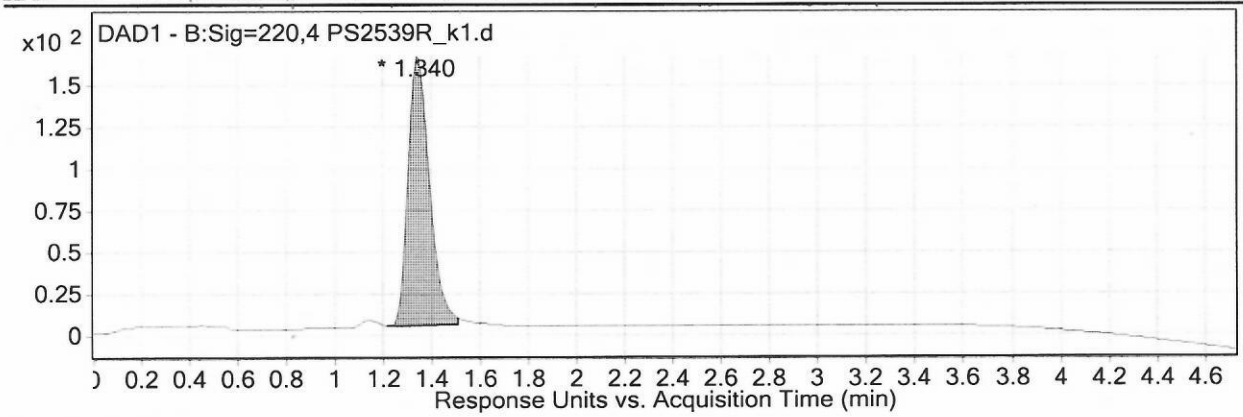
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** Esi



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	1,304	1,443	1,648	40567832	333111398	100

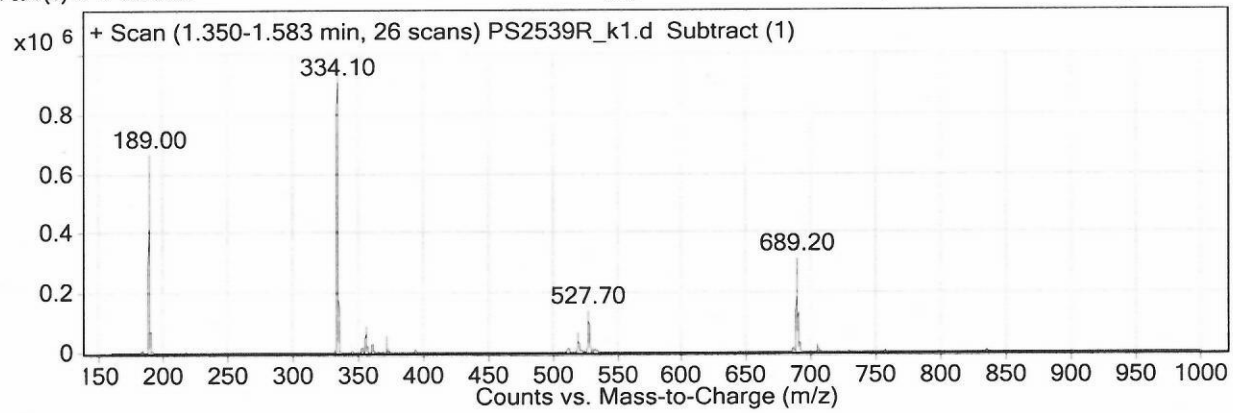


Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	1,213	1,340	1,507	161,41	1022,17	100

User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan"	135	0	Esi

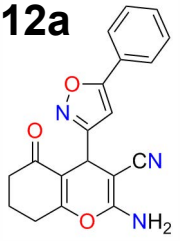


Qualitative Analysis Report

Peak List

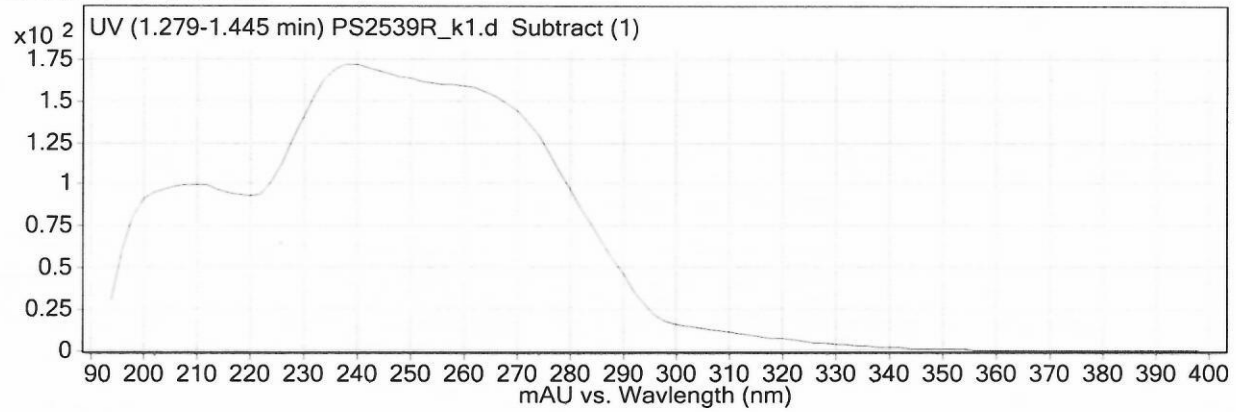
m/z	z	Abund.
189	1	663504
190	1	73061
334,1	1	953474
335,1	1	194203
356,1		88484
372,1		56765
519,8		68331
527,7		137753
689,2	1	315807
690,2	1	133929

12a

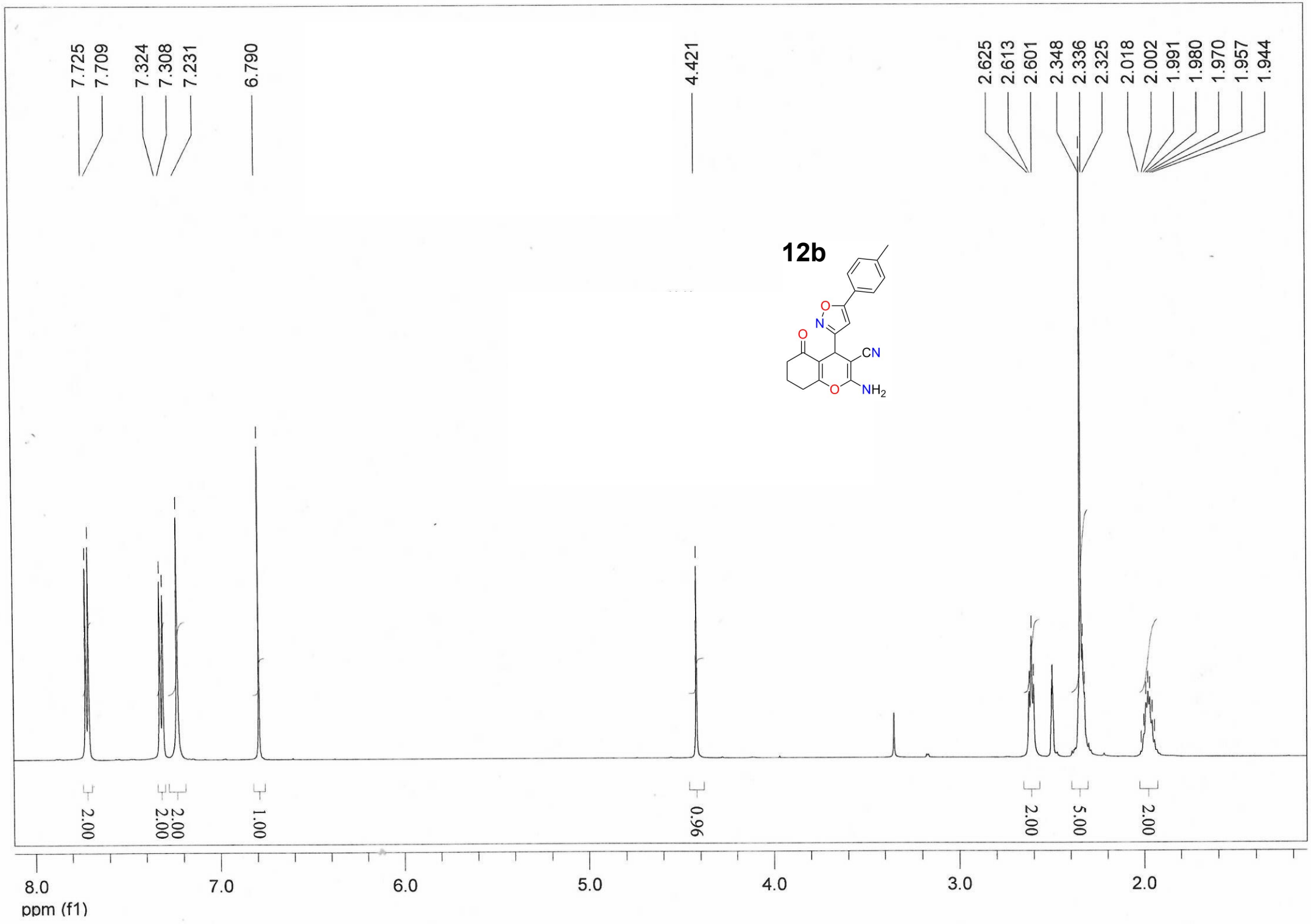


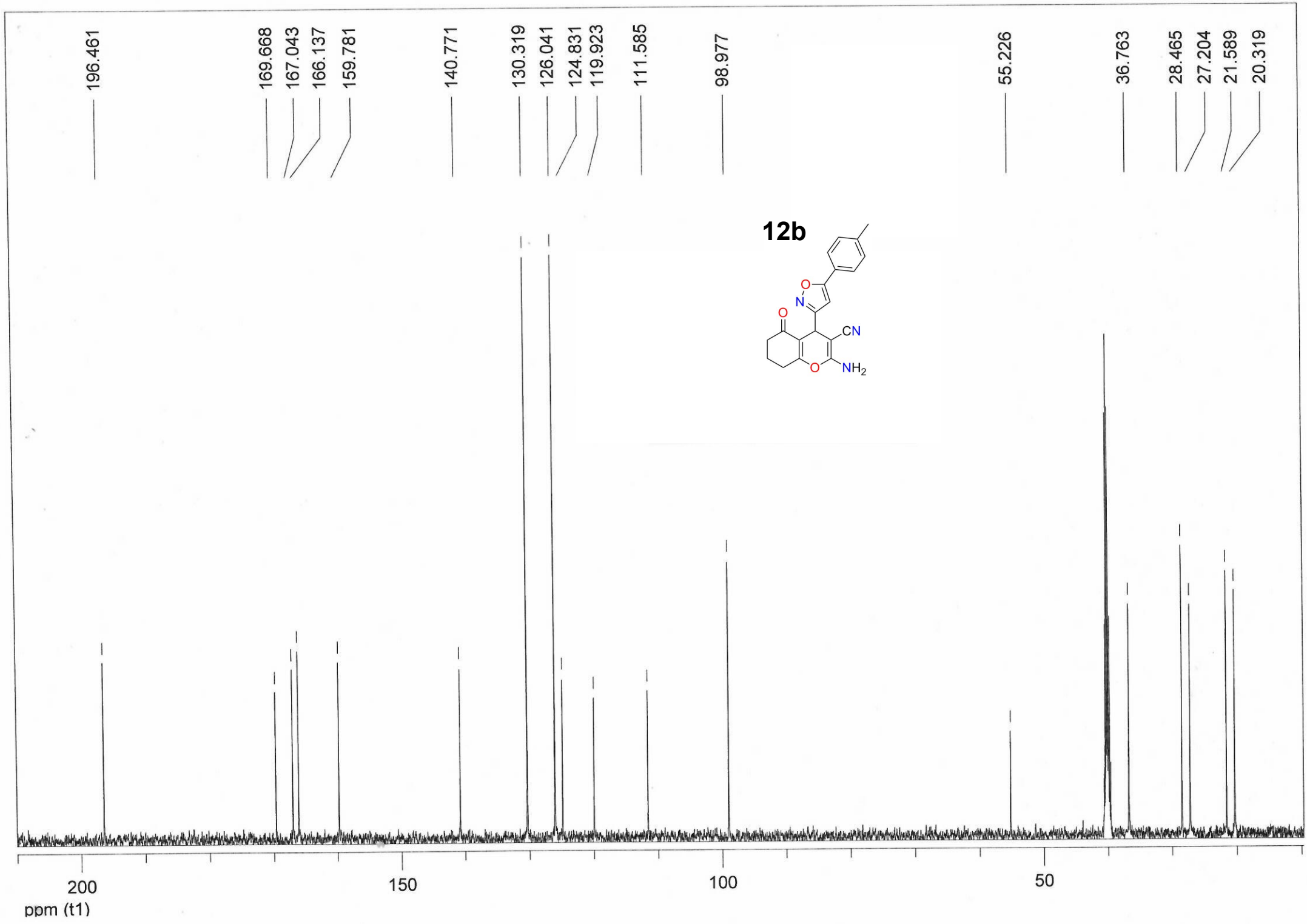
Spectrum Source

Peak (1) in "DAD1 - B:Sig=220,4"



--- End Of Report ---

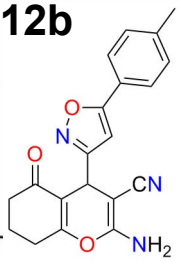

7.725
7.709
7.324
7.308
7.231
6.790
4.421
2.625
2.613
2.601
2.348
2.336
2.325
2.018
2.002
1.991
1.980
1.970
1.957
1.944
12b
O
N
O
CN
O
NH2
2.00
2.00
2.00
1.00
0.96
2.00
5.00
2.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
ppm (f1)


196.461
169.668
167.043
166.137
159.781
140.771
130.319
126.041
124.831
119.923
111.585
98.977
55.226
36.763
28.465
27.204
21.589
20.319
12b
O
N
O
CN
O
NH2
200
150
100
50
ppm (t1)

Qualitative Analysis Report

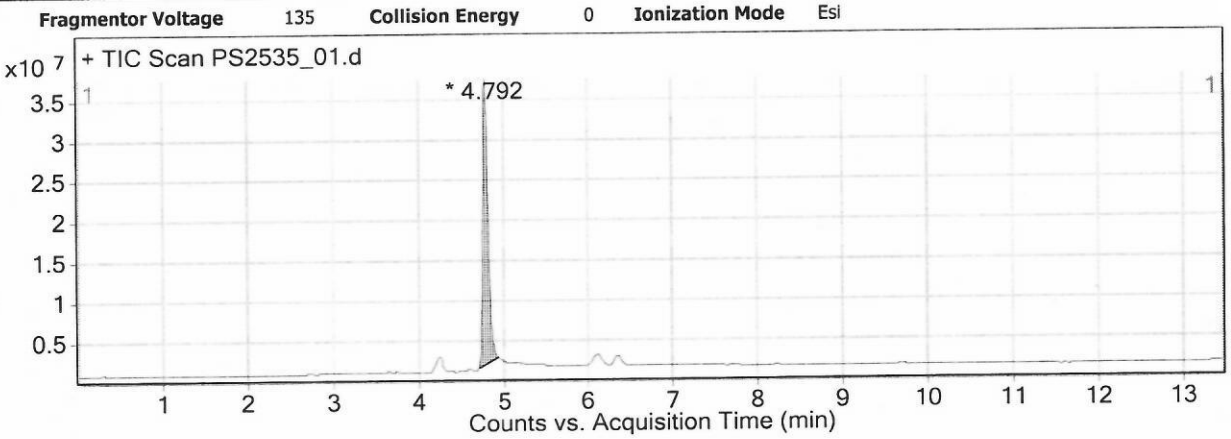
Data Filename	PS2535_01.d	**Sample Name**	Unavailable
Sample Type	Unavailable	**Position**	Unavailable
Instrument Name	Unavailable	**User Name**	Unavailable
Acq Method		**IRM Calibration Status**	Success
DA Method	Default.m	**Comment**	Sample information is unavailable

12b



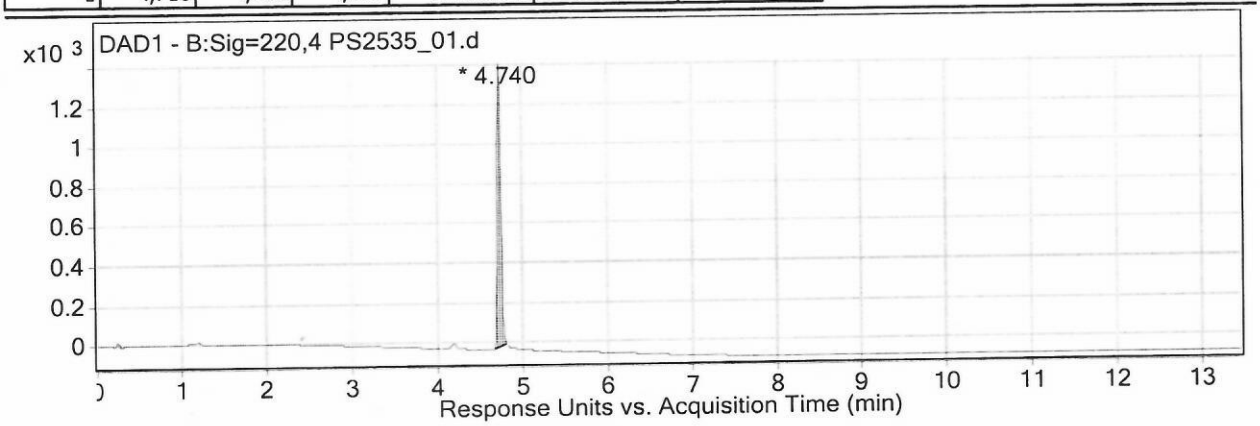
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** Esi



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	4,710	4,792	4,937	35598573	159197249	100

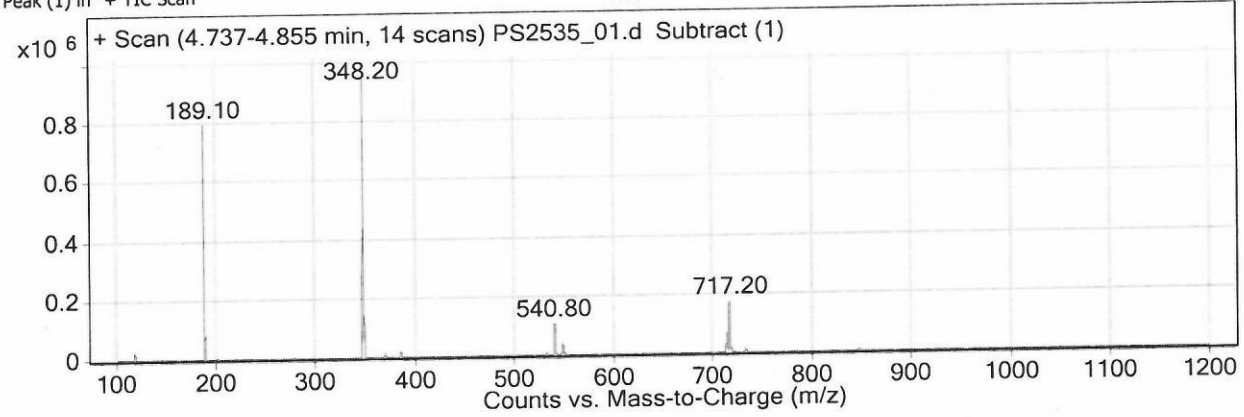


Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	4,673	4,740	4,807	1430,75	4297,93	100

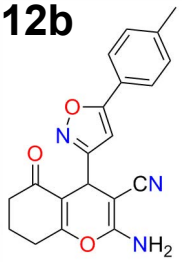
User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan"	135	0	Esi



Qualitative Analysis Report

12b

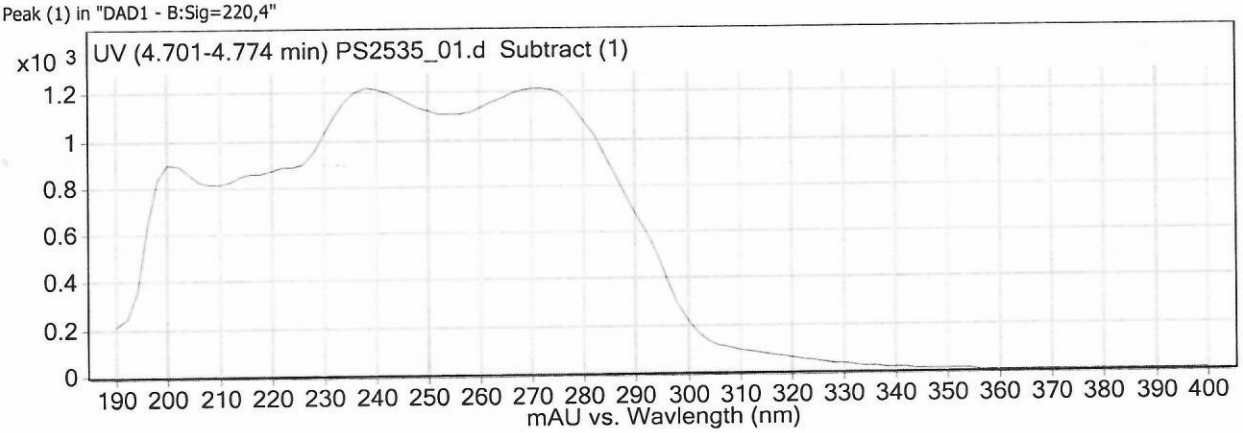


Peak List

m/z	z	Abund.
189,1	1	796983
190,1	1	84013
348,2	1	967384
349,2	1	206095
540,8		106456
714,4		65592
717,2		175214
718,3		80006

Spectrum Source

Peak (1) in "DAD1 - B:Sig=220,4"



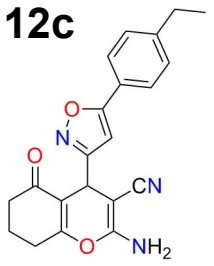
--- End Of Report ---

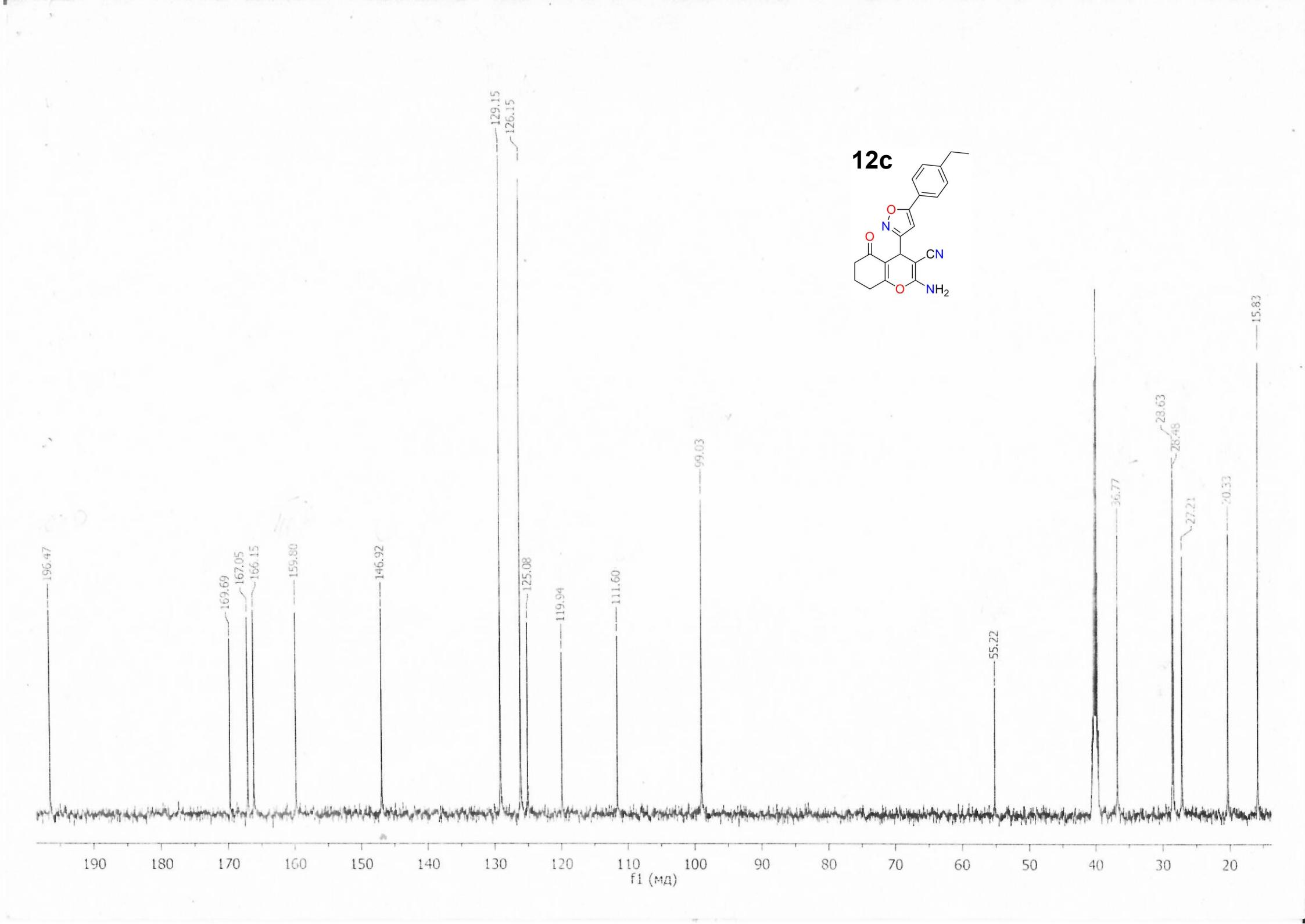
SK_zh12_05032023

bbo_1H_bar DMSO /v nmrsu 11

^{1}H NMR (500 MHz, DMSO) δ = 7.74 (d, *J*=8.1, 1H), 7.34 (d, *J*=8.1, 1H), 1.19 (t, *J*=7.6, 1H).

12c

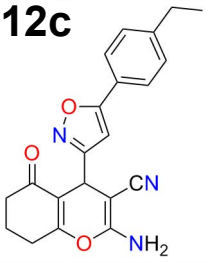



12c
O
N
O
CN
O
NH2
196.47
169.69
167.05
166.15
159.80
146.92
129.15
126.15
125.08
119.94
111.60
99.03
55.22
36.77
28.63
28.48
27.21
20.33
15.83
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (мд)

Qualitative Analysis Report

Data Filename	ZH-12_t1.d	**Sample Name**	ZH-12
Sample Type	Sample	**Position**	Vial 2
Instrument Name	Instrument 1	**User Name**	
Acq Method	All_2021_kol 1-2.m	**Acquired Time**	5/12/2023 10:45:01 AM
IRM Calibration Status	Not Applicable	**DA Method**	Defaul1t.m
Comment			

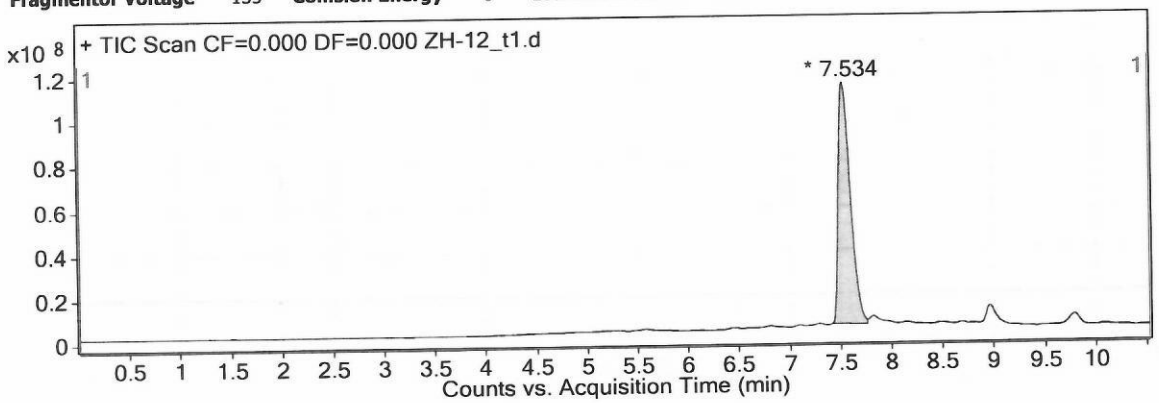
12c



Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition SW Version**	6400 Series Triple Quadrupole 10.0 (127)

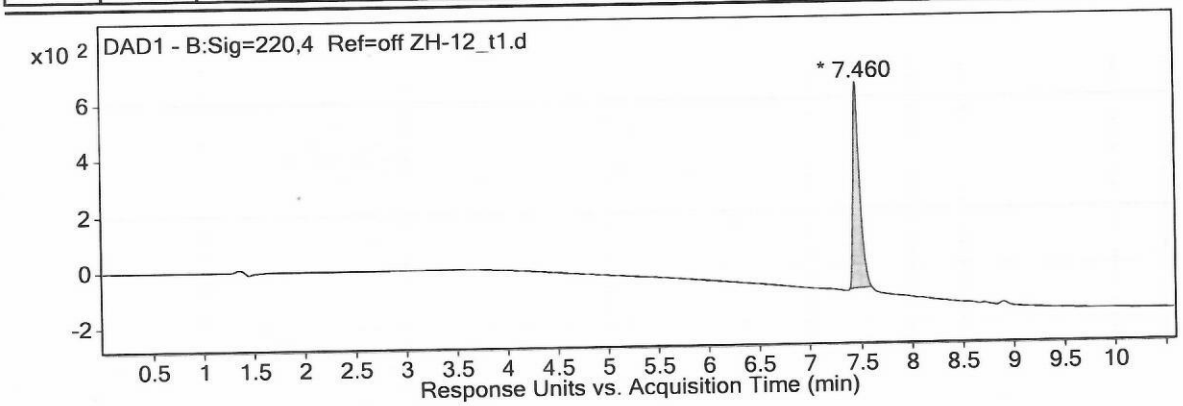
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,421	7,534	7,76	109373582,4	897169452,5	100



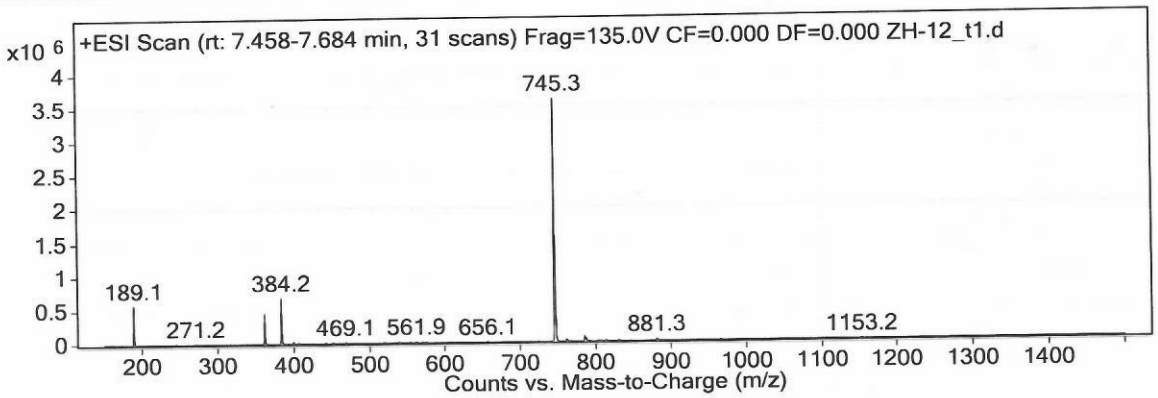
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,393	7,46	7,606	738,61	3354,19	100

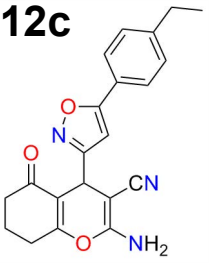
User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan"	135	0	ESI

Qualitative Analysis Report



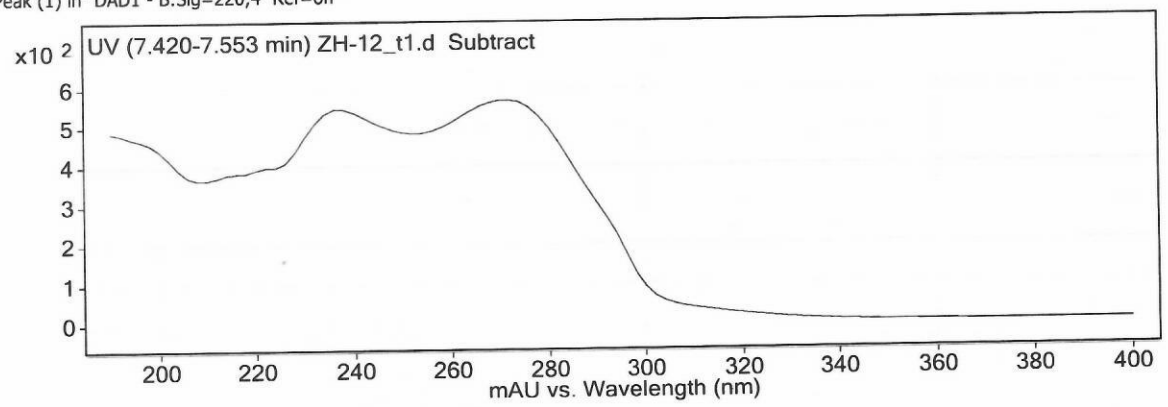
12c



Peak List

m/z	z	Abund
189.1	1	575217.81
362.2	1	451348.34
363.2	1	104135.53
384.2	1	681424.13
385.2	1	152590.22
745.3	1	3612887.25
746.3	1	1578503.63
747.3	1	368622.03
748.3	1	67051.02
785.3	1	81401.53

Spectrum Source
Peak (1) in "DAD1 - B:Sig=220,4 Ref=off"



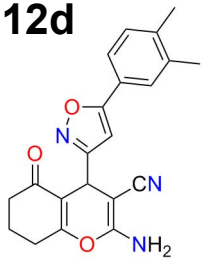
--- End Of Report ---

SK_zh11_04192023

bbo_1H_bar DMSO /v nmrsu 2

^{1}H NMR (500 MHz, DMSO) δ = 7.54 (d, *J*=7.8, 1H), 7.26 (d, *J*=7.9, 1H), 2.62 (t, *J*=5.8, 2H).

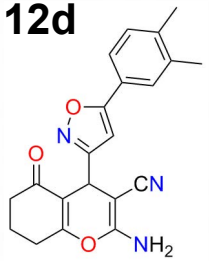
12d



SK_zh11_04192023

bbo_13CF_bar DMSO /v nmrsu 2

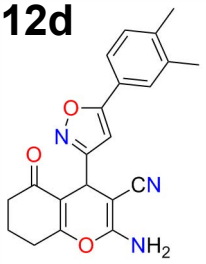
12d



Qualitative Analysis Report

Data Filename	ZH-11_t1.d	**Sample Name**	ZH-11
Sample Type	Sample	**Position**	Vial 2
Instrument Name	Instrument 1	**User Name**	
Acq Method	All_2021_kol 1-2.m	**Acquired Time**	5/12/2023 10:22:29 AM
IRM Calibration Status	Not Applicable	**DA Method**	Defaul1t.m
Comment			

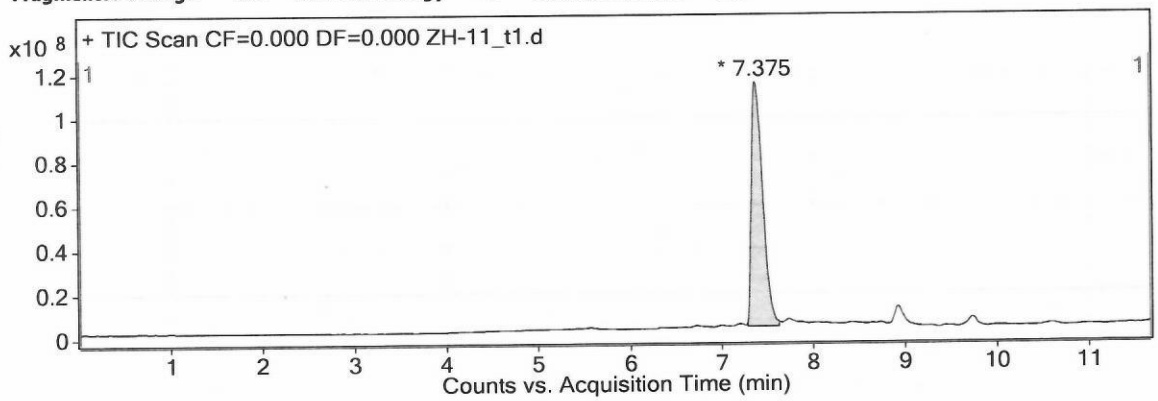
12d



Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition SW Version**	6400 Series Triple Quadrupole 10.0 (127)

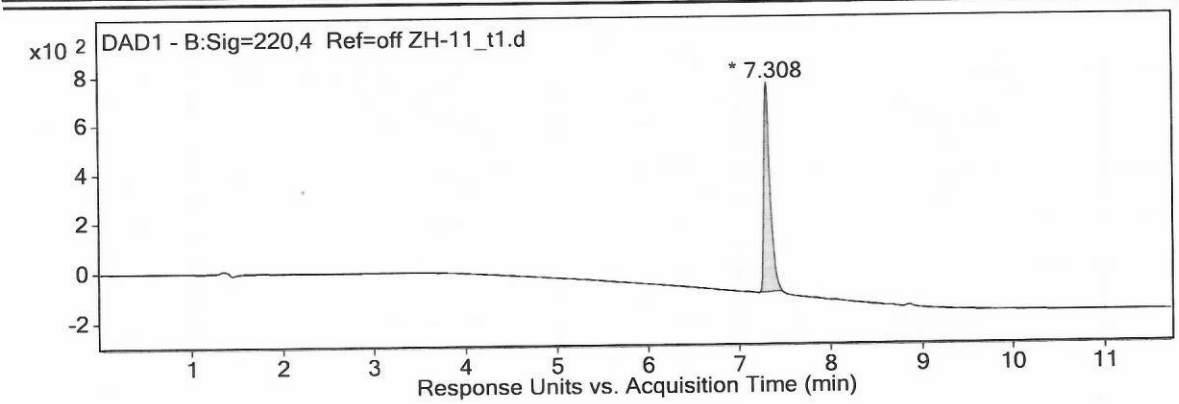
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,285	7,375	7,632	111280717,8	959829711,1	100



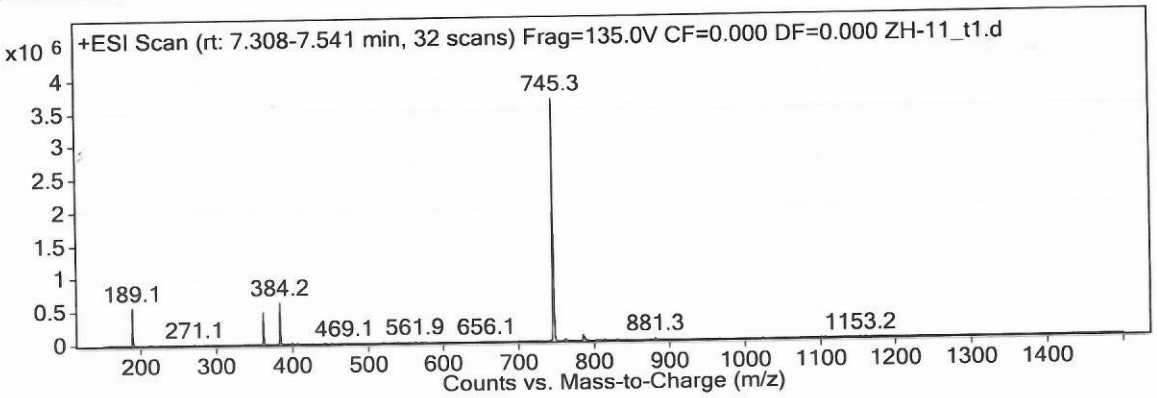
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,228	7,308	7,475	859,08	4074,86	100

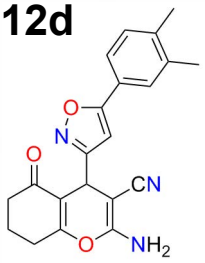
User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan"	135	0	ESI

Qualitative Analysis Report



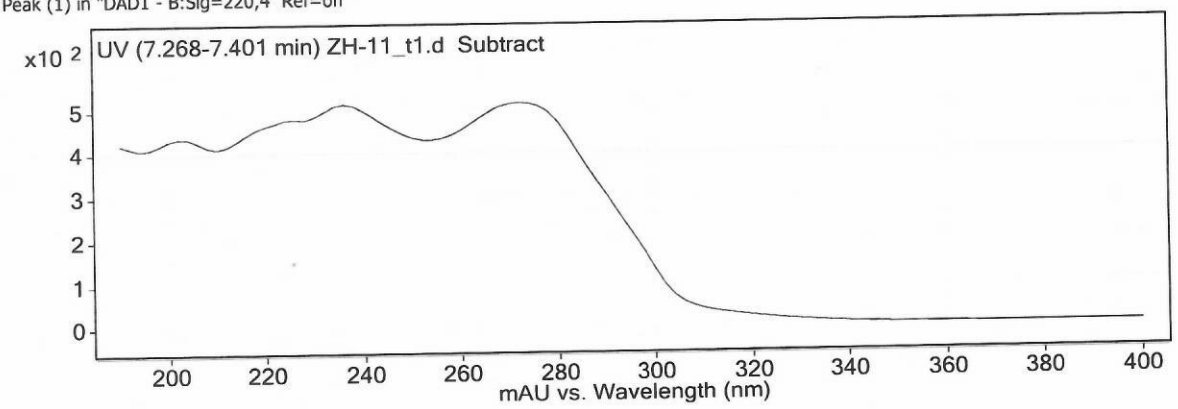
12d



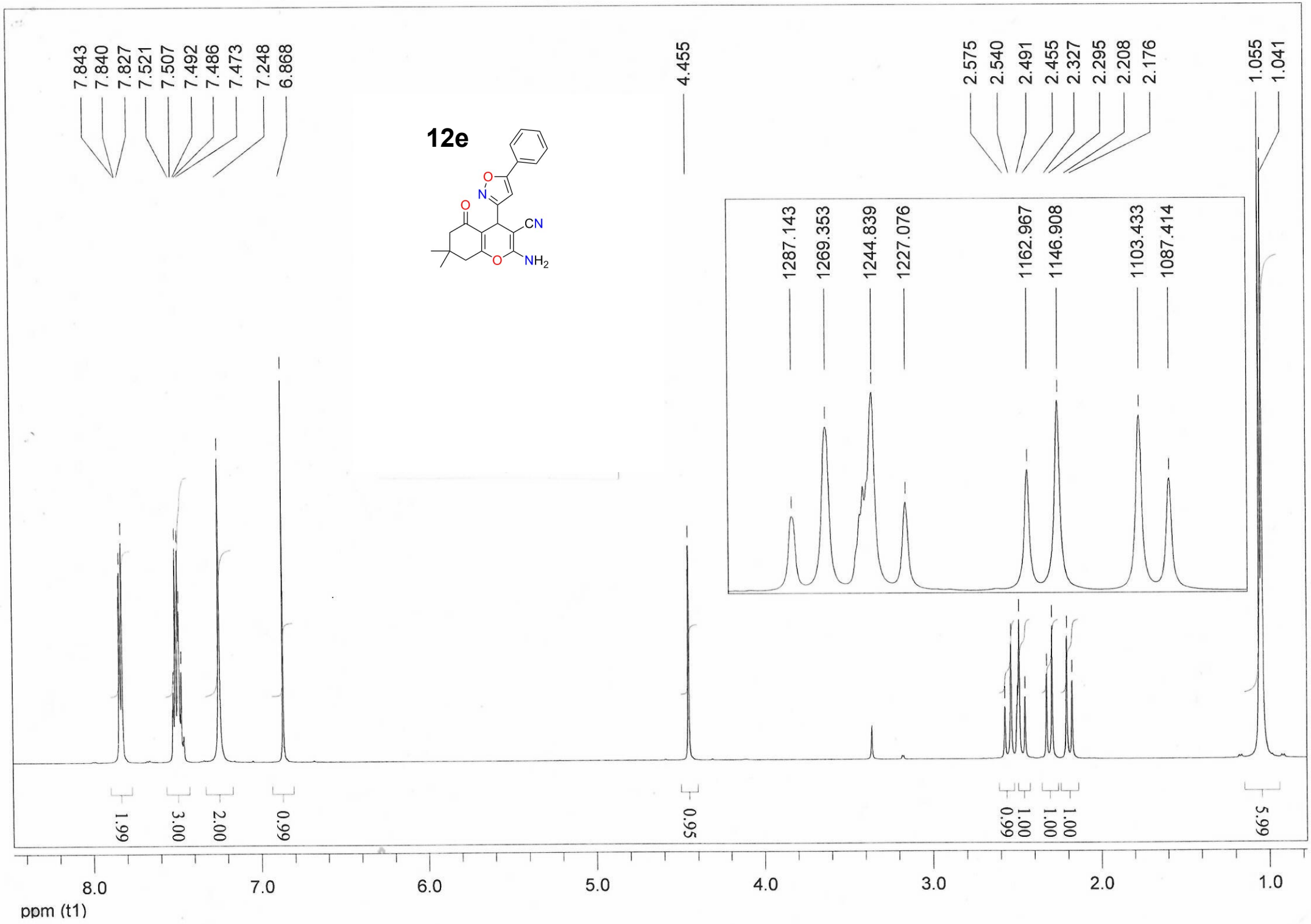
Peak List

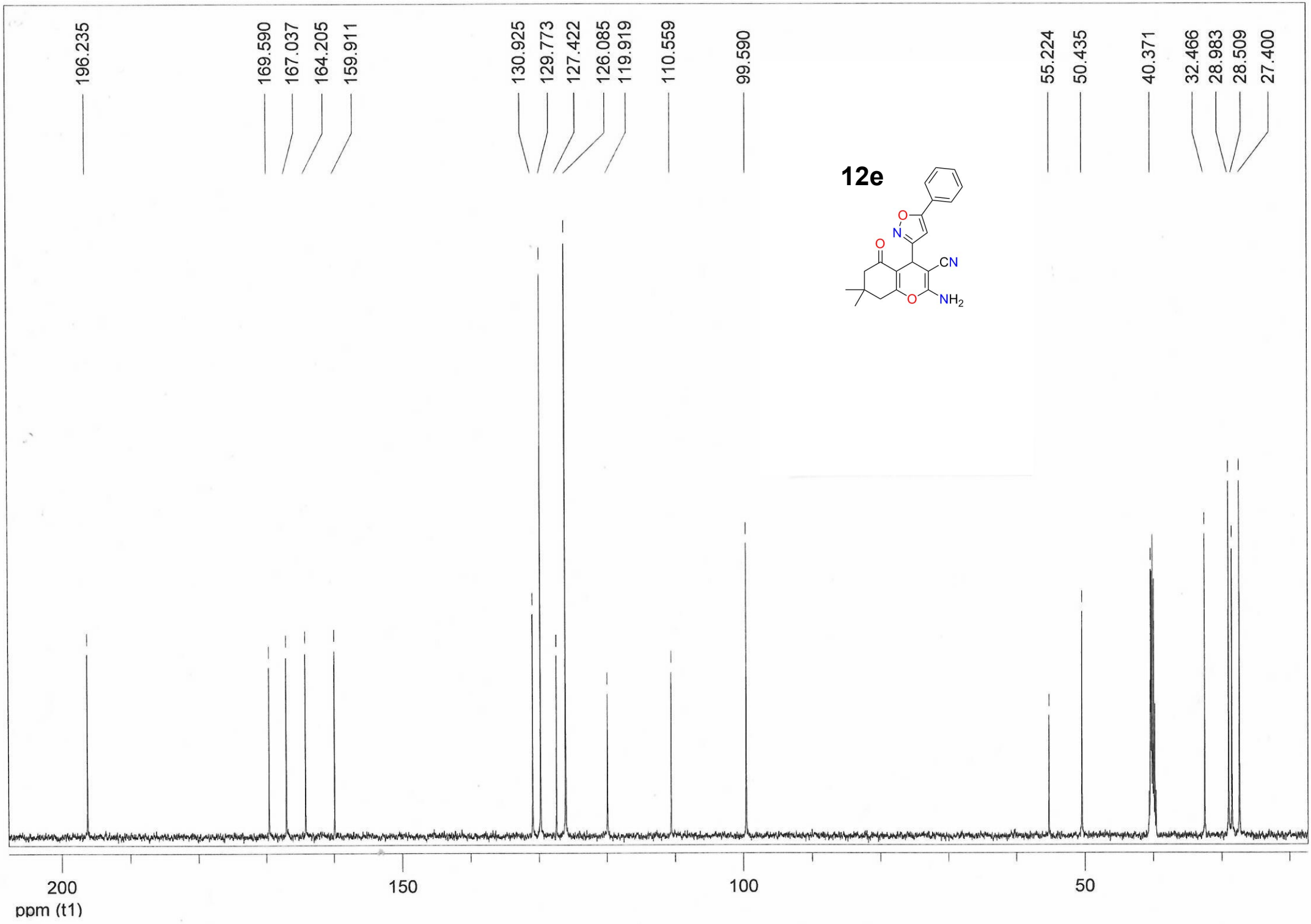
m/z	z	Abund
189.1	1	569036.56
362.2	1	488253.97
363.2	1	113170.16
384.2		634524.81
385.1		147476.25
745.3	1	3671770.75
746.3	1	1611261.88
747.3	1	371363.16
748.3	1	68150.44
785.3	1	93351.94

Spectrum Source
Peak (1) in "DAD1 - B:Sig=220,4 Ref=off"



--- End Of Report ---

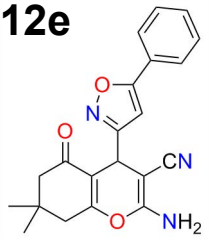

12e
7.843
7.840
7.827
7.521
7.507
7.492
7.486
7.473
7.248
6.868
4.455
2.575
2.540
2.491
2.455
2.327
2.295
2.208
2.176
1.055
1.041
1287.143
1269.353
1244.839
1227.076
1162.967
1146.908
1103.433
1087.414
1.99
3.00
2.00
0.99
0.95
0.99
1.00
1.00
1.00
5.99
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)


196.235
169.590
167.037
164.205
159.911
130.925
129.773
127.422
126.085
119.919
110.559
99.590
55.224
50.435
40.371
32.466
28.983
28.509
27.400
12e
O
N
O
O
CN
NH2
200
150
100
50
ppm (t1)

Qualitative Analysis Report

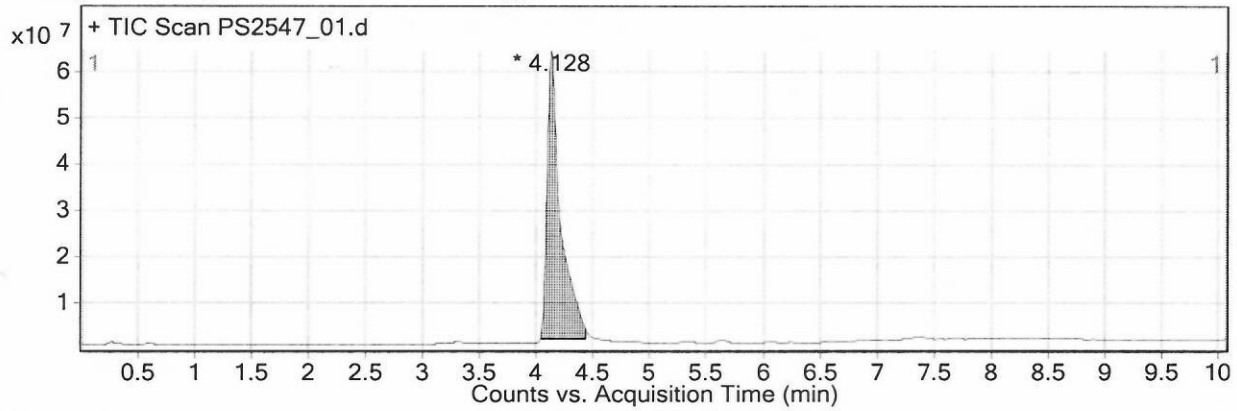
Data Filename	PS2547_01.d	**Sample Name**	Unavailable
Sample Type	Unavailable	**Position**	Unavailable
Instrument Name	Unavailable	**User Name**	Unavailable
Acq Method		**IRM Calibration Status**	Success
DA Method	Default.m	**Comment**	Sample information is unavailable

12e



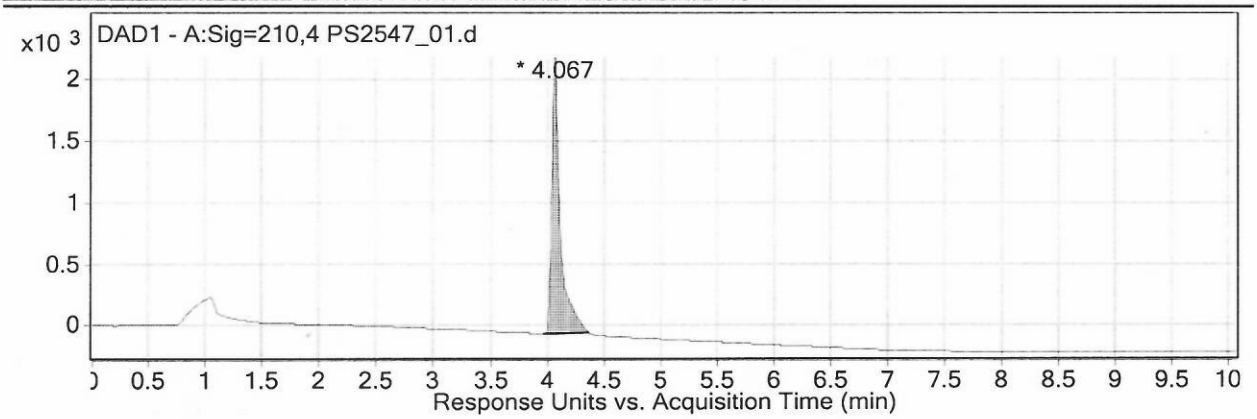
User Chromatograms

Fragmentor Voltage	90	**Collision Energy**	0	**Ionization Mode**	Esi



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	4,039	4,128	4,434	62064161	514376014	100

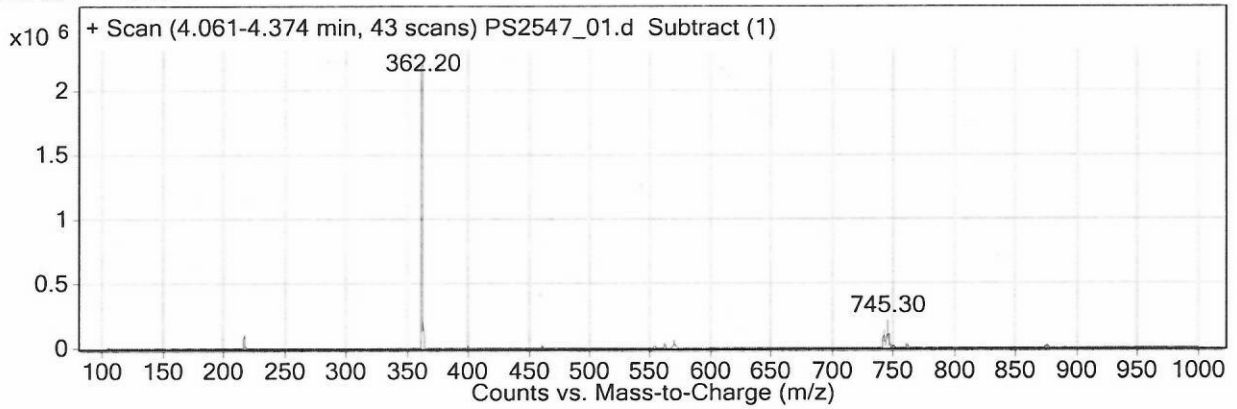


Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	3,967	4,067	4,367	2239,04	11725,81	100

User Spectra

Spectrum Source	**Fragmentor Voltage**	**Collision Energy**	**Ionization Mode**
Peak (1) in "+ TIC Scan"	90	0	Esi

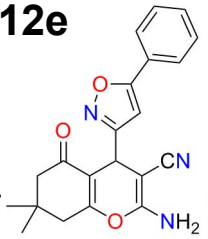


Qualitative Analysis Report

Peak List

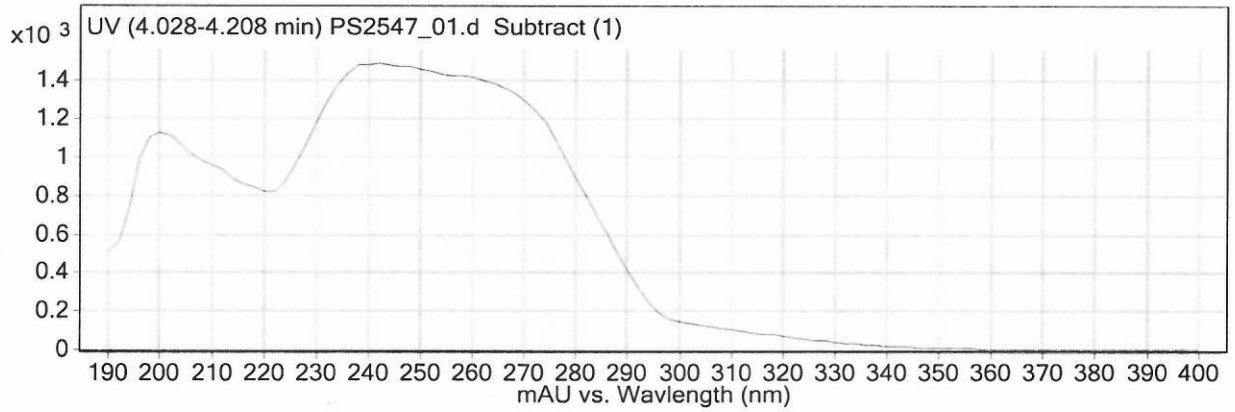
m/z	z	Abund.
362,2	1	2204545
363,2	1	464837
742,5		126107
745,3		217685

12e

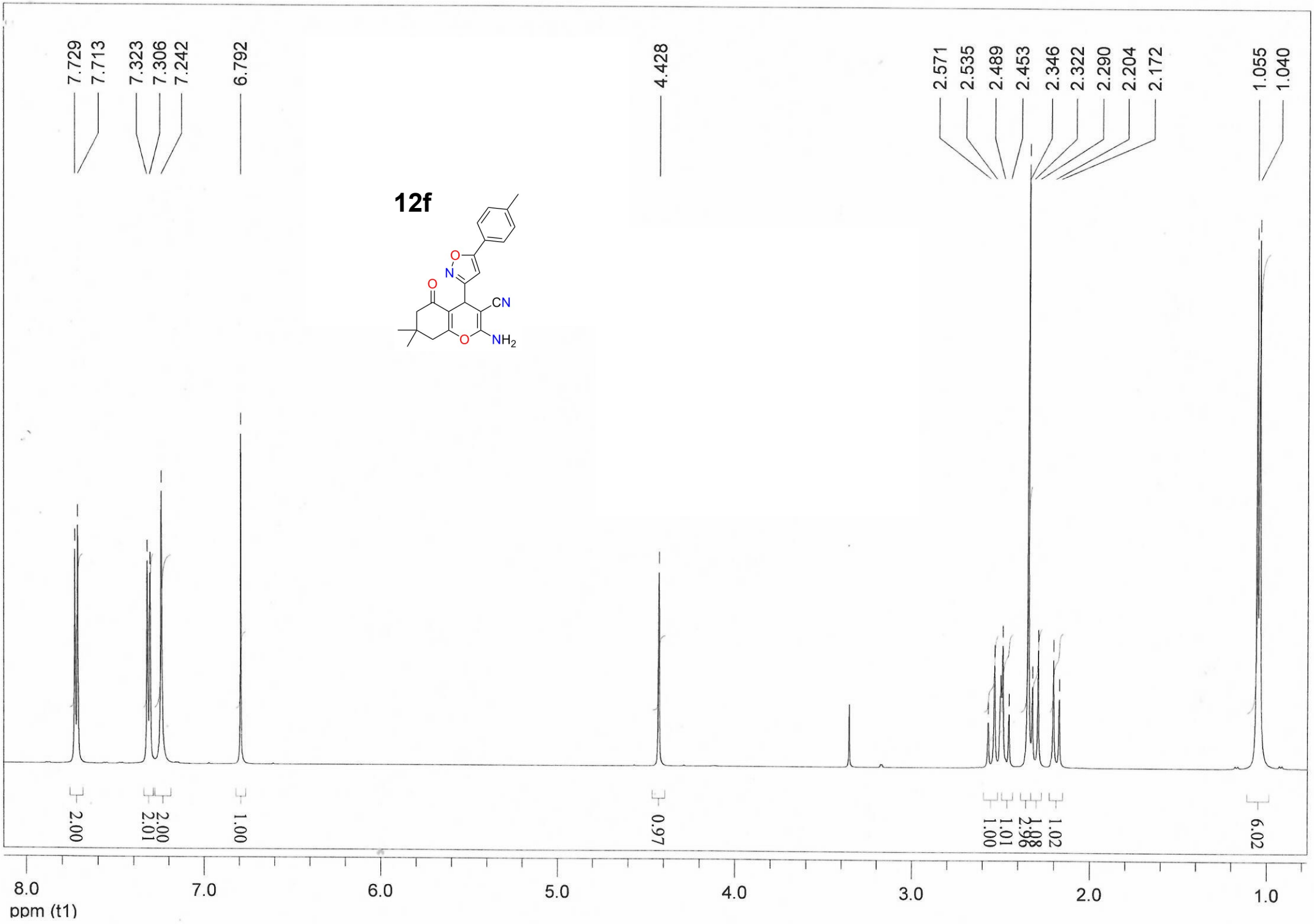


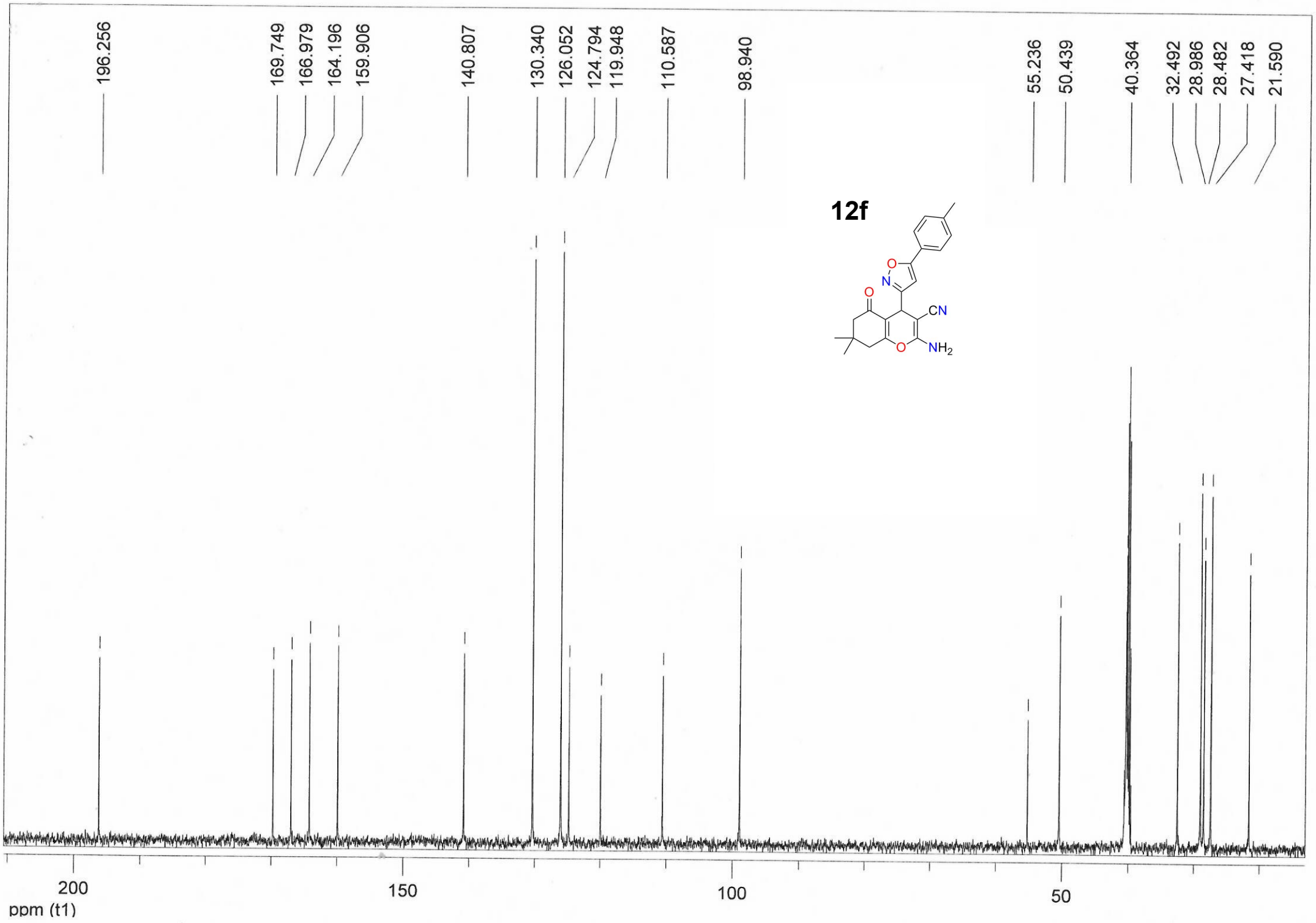
Spectrum Source

Peak (1) in "DAD1 - A:Sig=210,4"



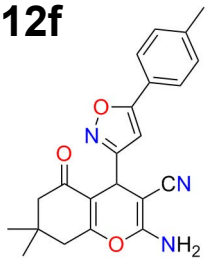
--- End Of Report ---


7.729
7.713
7.323
7.306
7.242
6.792
4.428
2.571
2.535
2.489
2.453
2.346
2.322
2.290
2.204
2.172
1.055
1.040
12f
O
N
O
CN
O
NH2
2.00
2.00
2.01
1.00
0.97
1.00
1.01
2.98
1.02
6.02
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)


196.256
169.749
166.979
164.196
159.906
140.807
130.340
126.052
124.794
119.948
110.587
98.940
55.236
50.439
40.364
32.492
28.986
28.482
27.418
21.590
12f
O
N
O
CN
O
NH2
200
150
100
50
ppm (t1)

Qualitative Analysis Report

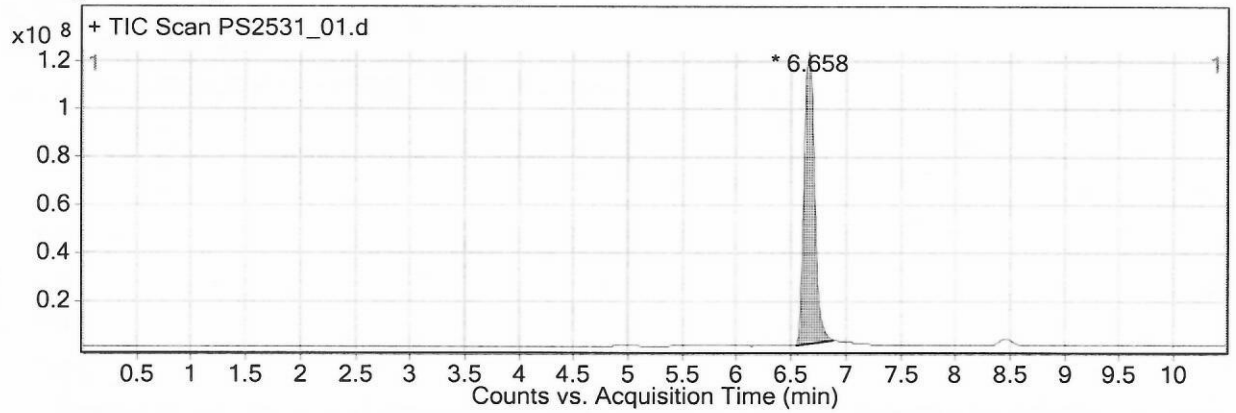
12f



Data Filename	PS2531_01.d	**Sample Name**	
Sample Type	Unavailable	**Position**	
Instrument Name	Unavailable	**User Name**	
Acq Method		**IRM Calibration Status**	
DA Method	Default.m	**Comment**	

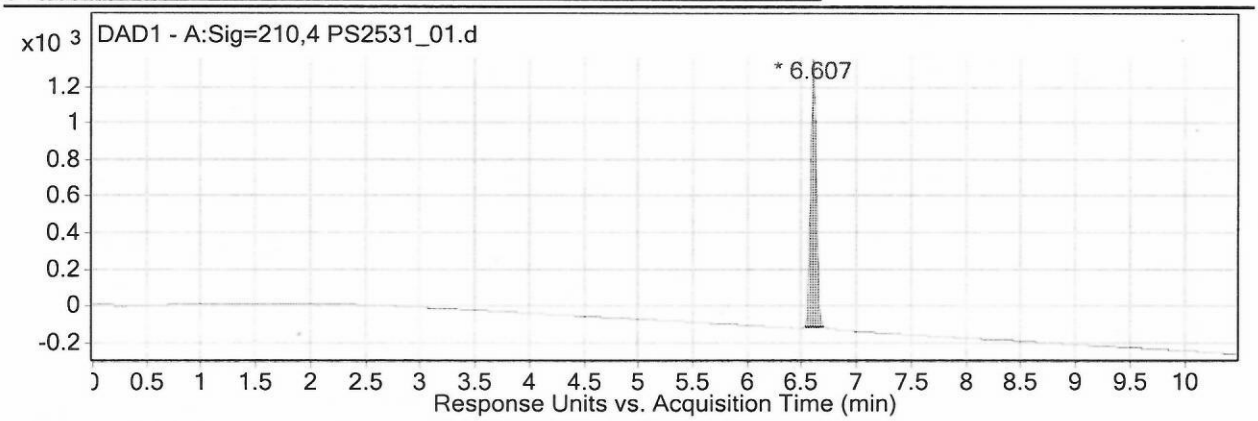
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** Esi



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	6,541	6,658	6,893	122152087	781421380	100

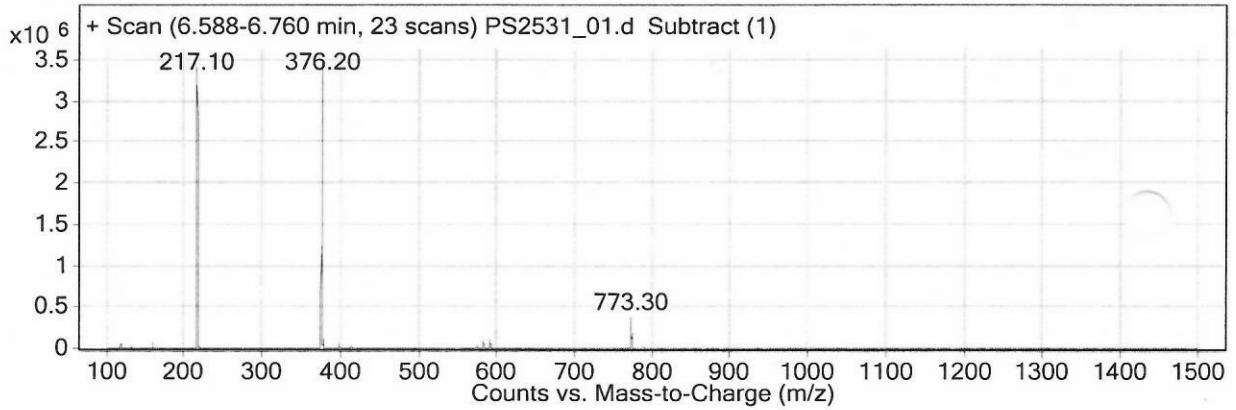


Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	6,527	6,607	6,707	1473,02	5461,03	100

User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan"	135	0	Esi

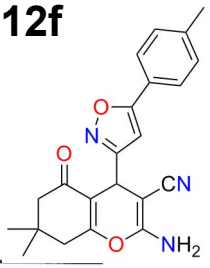


Qualitative Analysis Report

Peak List

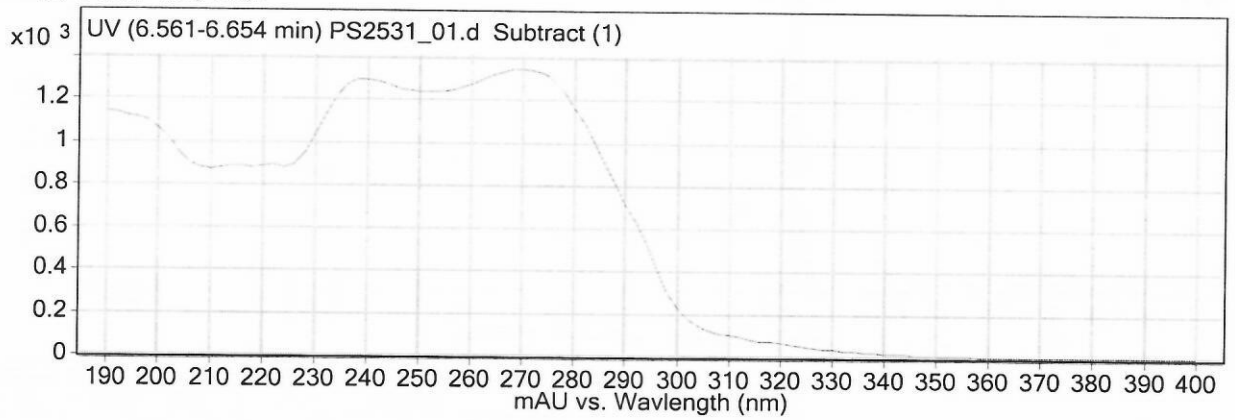
m/z	z	Abund.
217,1	1	3405041
218,1	1	398484
376,2		3458335
377,1		782424
773,3	1	365842
774,3	1	177876

12f



Spectrum Source

Peak (1) in "DAD1 - A:Sig=210,4"



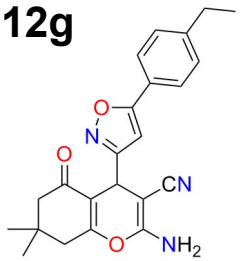
--- End Of Report ---

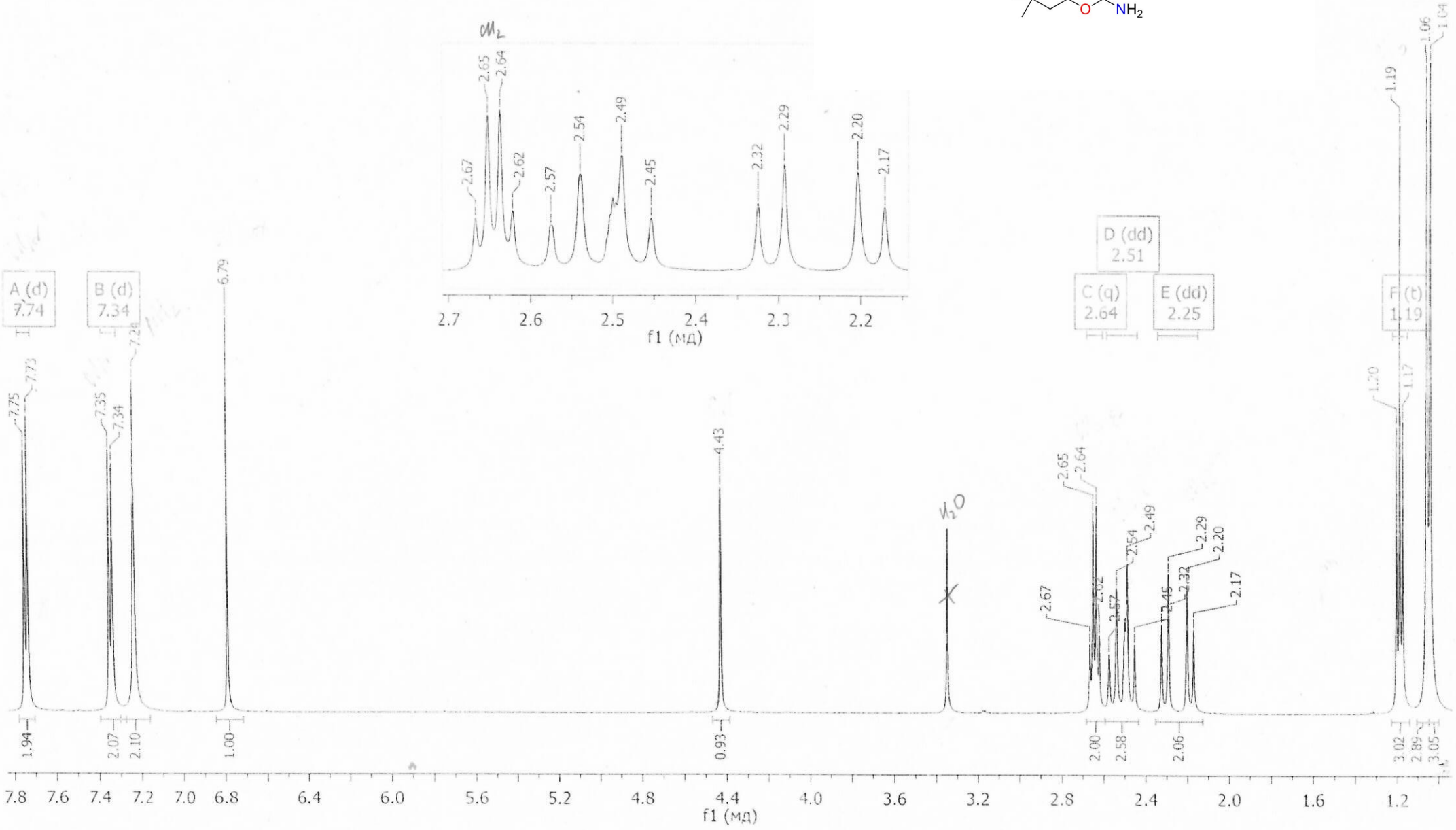
SK_zh10_04192023

bbo_1H_bar DMSO /v nmrsu 16

^{1}H NMR (500 MHz, DMSO) δ = 7.74 (d, *J*=8.2, 1H), 7.34 (d, *J*=8.2, 1H), 2.64 (q, *J*=7.6, 1H), 2.51 (dd, *J*=43.0, 17.7, 1H), 2.25 (dd, *J*=60.7, 16.0, 1H), 1.19 (t, *J*=7.6, 2H).

12g

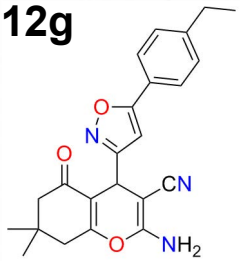




SK_zh10_04192023

bbo_13CF_bar DMSO /v nmrsu 16

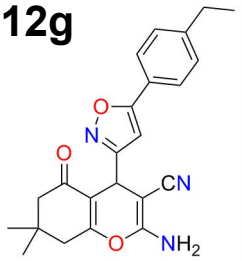
12g



Qualitative Analysis Report

Data Filename	ZH-10_t1.d	**Sample Name**	ZH-10
Sample Type	Sample	**Position**	Vial 2
Instrument Name	Instrument 1	**User Name**	
Acq Method	All_2021_kol 1-2.m	**Acquired Time**	5/12/2023 10:01:36 AM
IRM Calibration Status	Not Applicable	**DA Method**	Defaul1t.m
Comment			

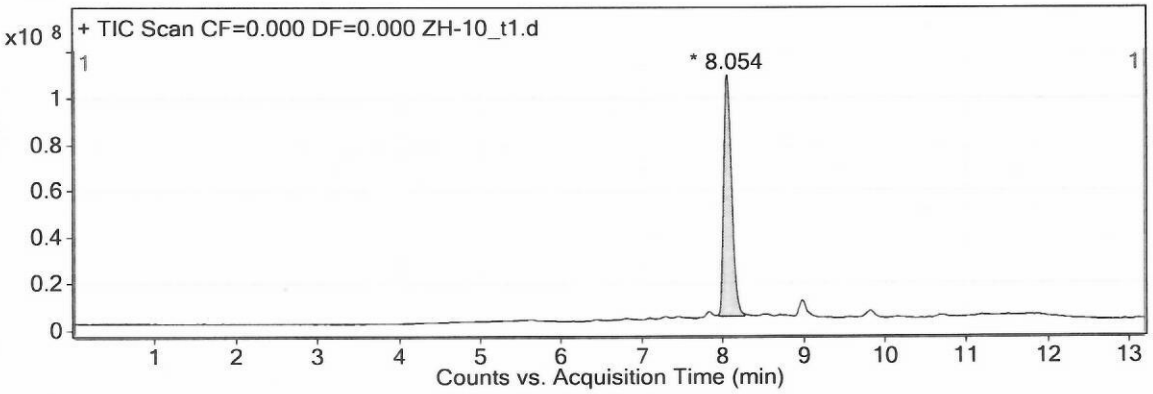
12g



Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition SW Version**	6400 Series Triple Quadrupole 10.0 (127)

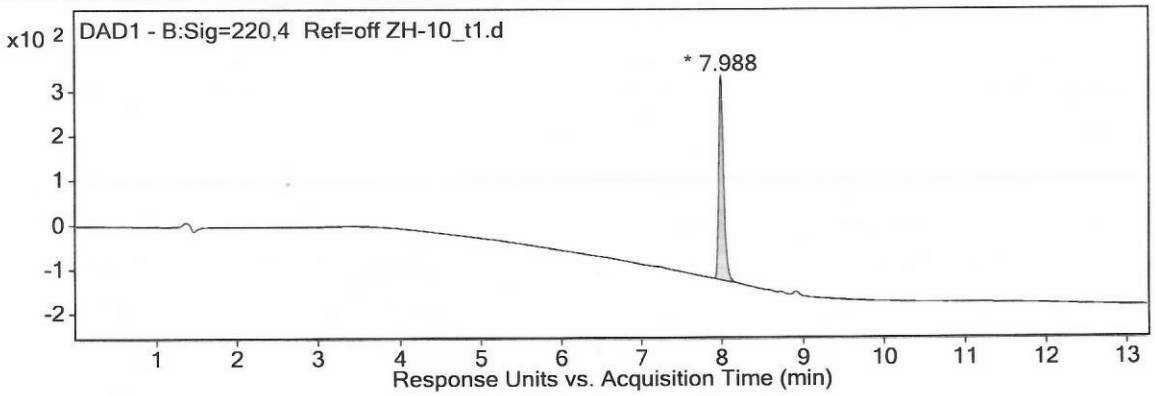
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,948	8,054	8,272	104163473,5	715338023,6	100



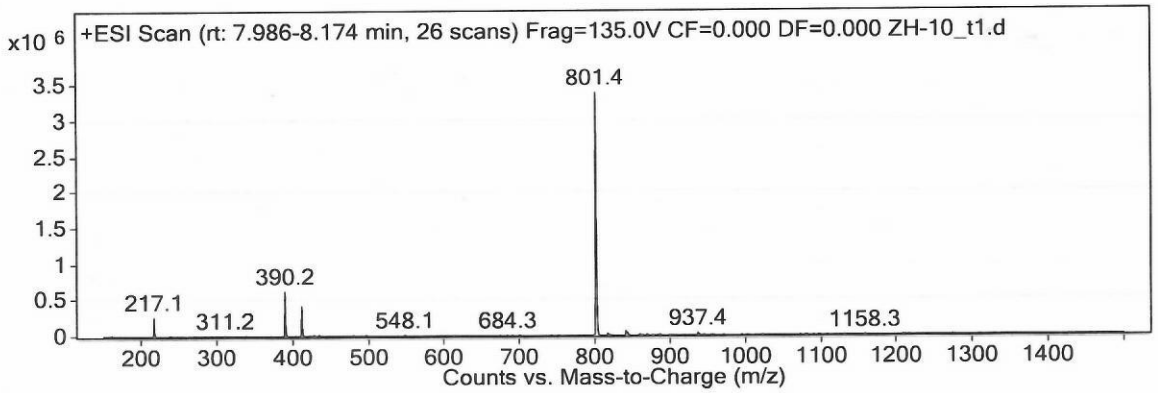
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,895	7,988	8,195	461,43	1951,78	100

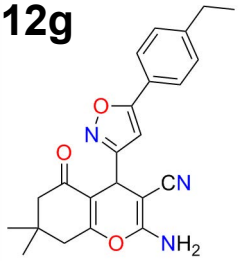
User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan"	135	0	ESI

Qualitative Analysis Report



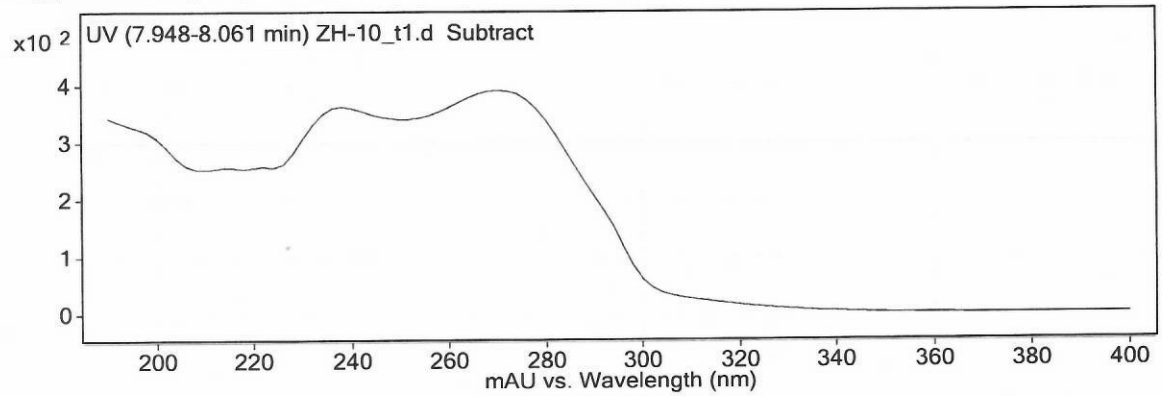
12g



Peak List

m/z	z	Abund
217.1	1	264546.41
390.2	1	622332.44
391.2	1	155738.61
412.2	1	421707.53
413.2	1	110807.05
801.4	1	3389641
802.4	1	1628730.13
803.4	1	405797.53
804.4	1	75748.55
841.3	1	64117.92

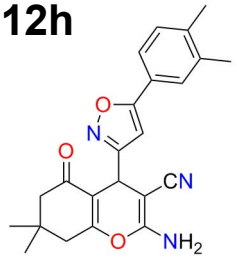
Spectrum Source
Peak (1) in "DAD1 - B:Sig=220,4 Ref=off"

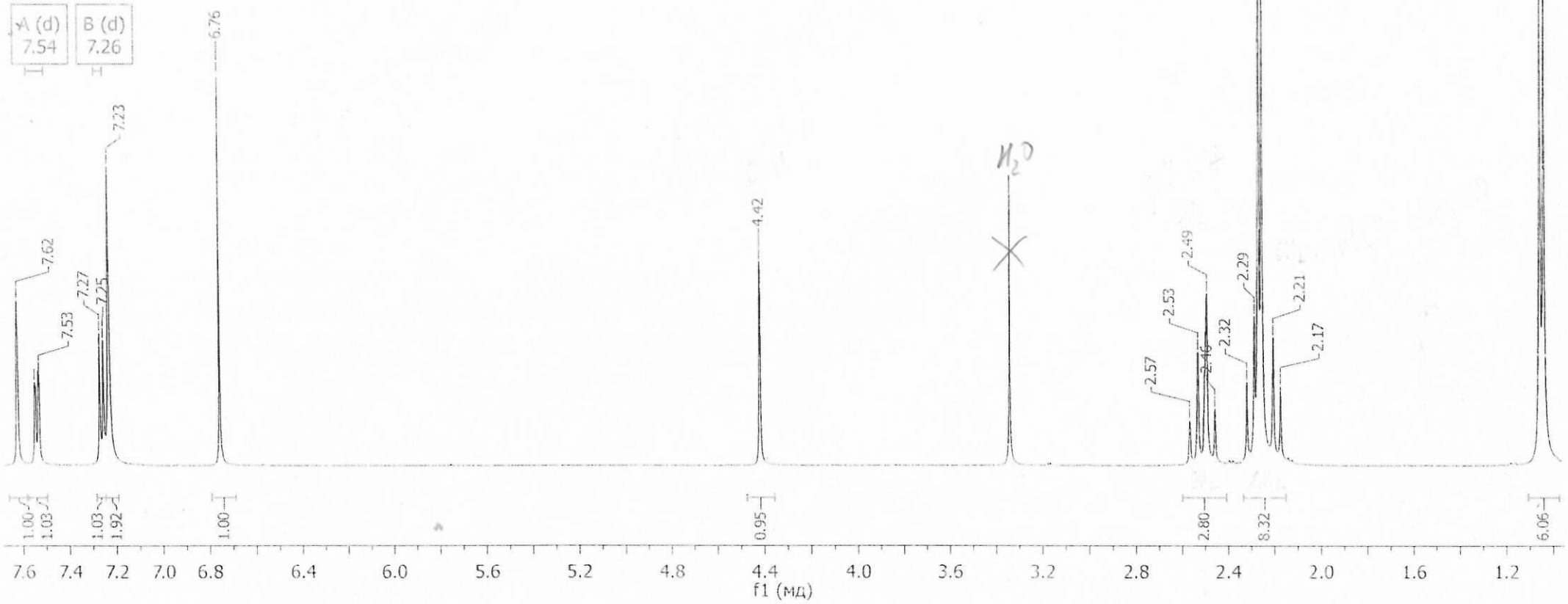


--- End Of Report ---

^{1}H NMR (500 MHz, DMSO) δ = 7.54 (d, *J*=9.2, 1H), 7.26 (d, *J*=7.9, 1H).

12h

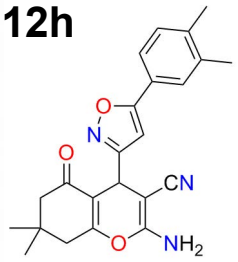


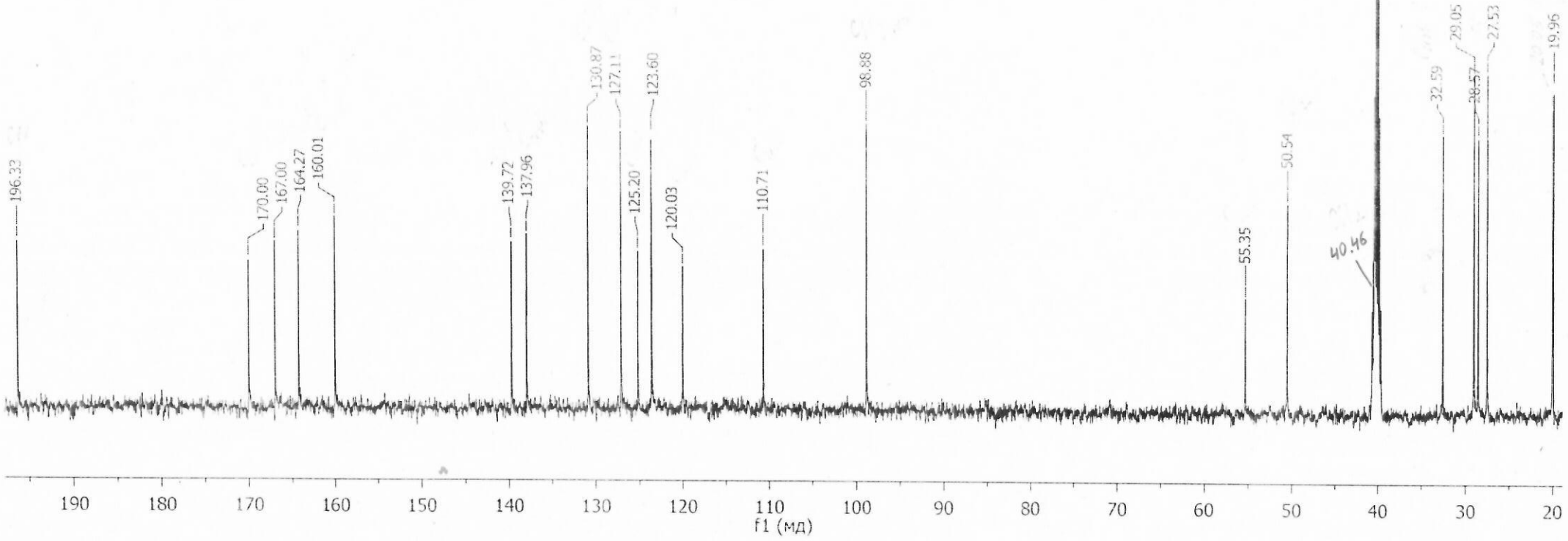


SK_zh9_04192023

bbo_13CF_bar DMSO /v nmrsu 3

12h

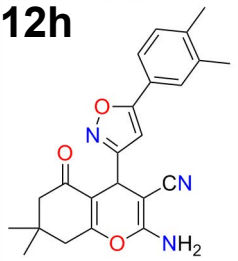




Qualitative Analysis Report

Data Filename	ZH-9_t1.d	**Sample Name**	ZH-9
Sample Type	Sample	**Position**	Vial 2
Instrument Name	Instrument 1	**User Name**	
Acq Method	All_2021_kol 1-2.m	**Acquired Time**	5/12/2023 9:41:38 AM
IRM Calibration Status	Not Applicable	**DA Method**	Defaul1t.m
Comment			

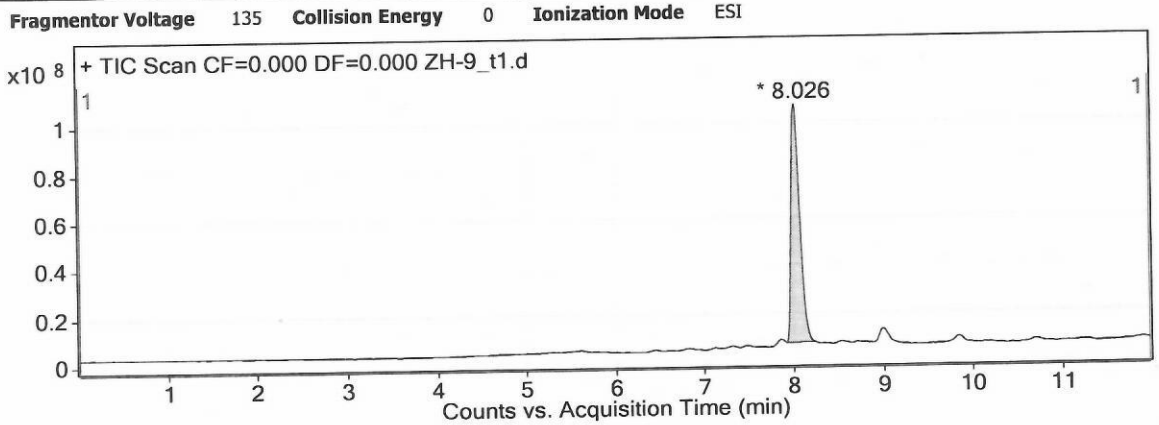
12h



Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition SW Version**	6400 Series Triple Quadrupole 10.0 (127)

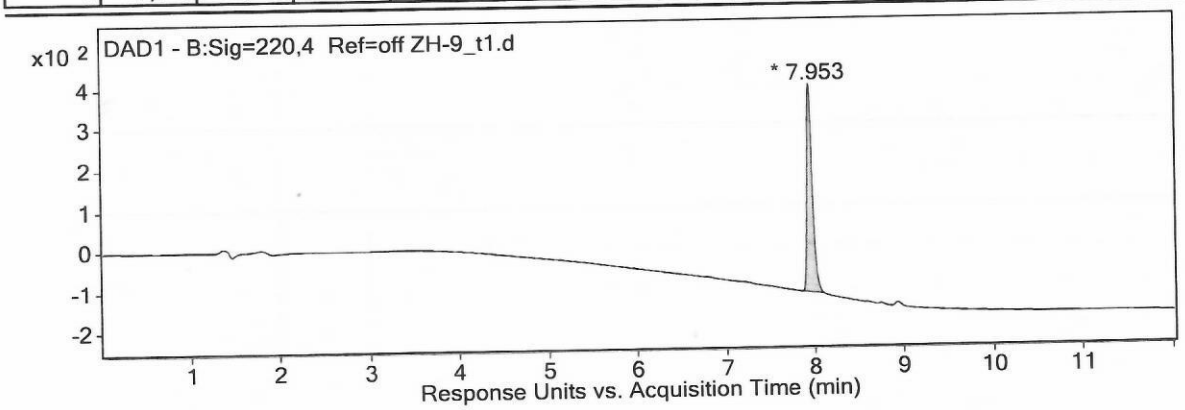
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,918	8,026	8,234	100192715,8	676627325,4	100



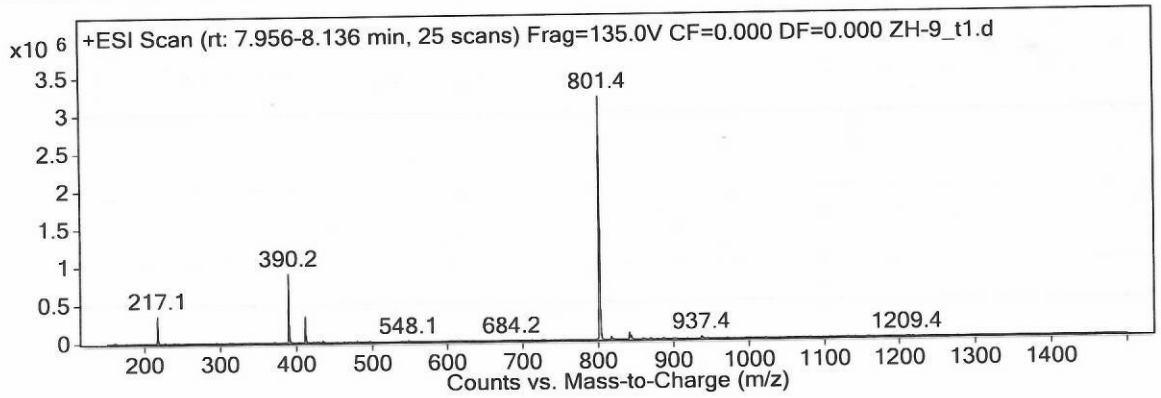
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,866	7,953	8,106	514,42	2196,68	100

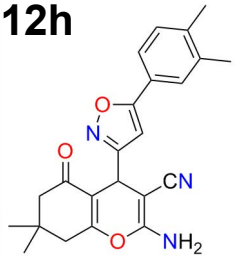
User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan"	135	0	ESI

Qualitative Analysis Report



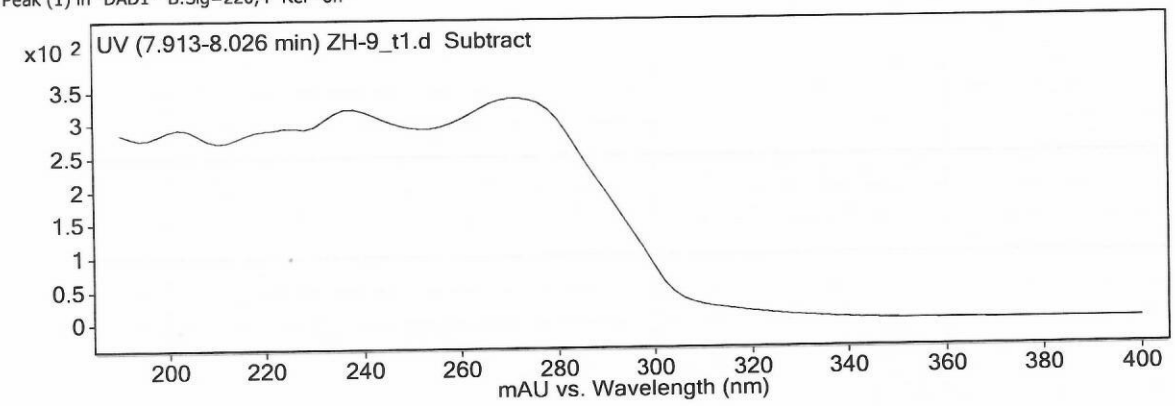
12h



Peak List

m/z	z	Abund
217.1	1	360304.88
390.2	1	905105.63
391.2	1	222456.55
412.2	1	347667.56
413.2	1	89409.32
801.4	1	3206206.5
802.4	1	1517856.5
803.4	1	375397.59
804.4	1	72159.23
841.4	1	91376.82

Spectrum Source
Peak (1) in "DAD1 - B:Sig=220,4 Ref=off"

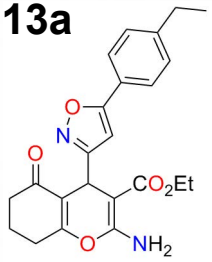


--- End Of Report ---

SK_zh-20_05172023

bbo_1H_bar DMSO /v nmrsu 4

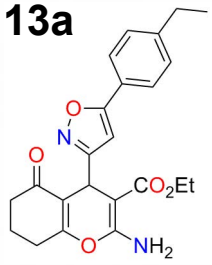
13a



SK_zh-20_05172023

bbo_13CF_bar DMSO /v nmrsu 4

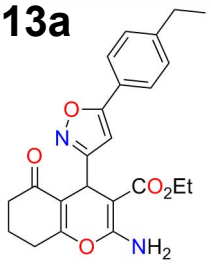
13a



Qualitative Analysis Report

Data Filename	ZH20_01.d	**Sample Name**	ZH20
Sample Type	Sample	**Position**	Vial 2
Instrument Name	Instrument 1	**User Name**	
Acq Method	All_2021_kol 1-2.m	**Acquired Time**	5/29/2023 10:43:36 AM
IRM Calibration Status	Not Applicable	**DA Method**	Defaul1t.m
Comment			

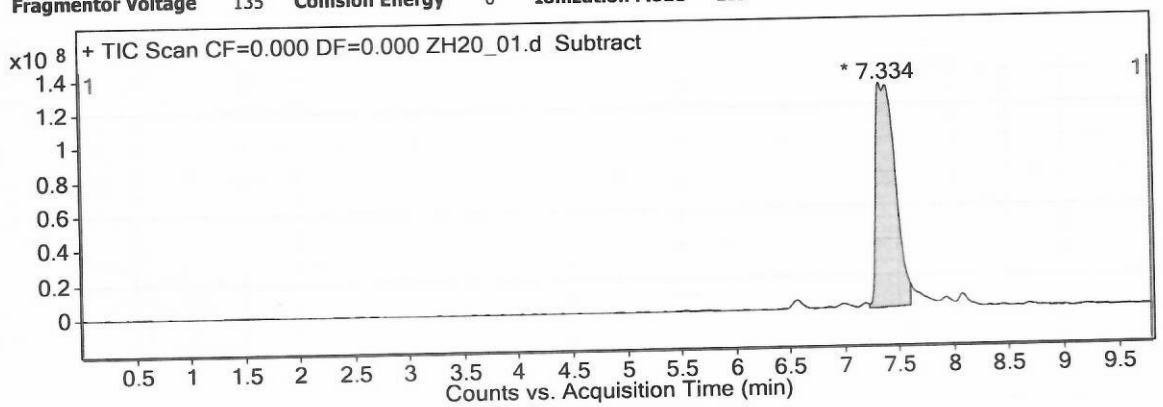
13a



Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition SW Version**	6400 Series Triple Quadrupole 10.0 (127)

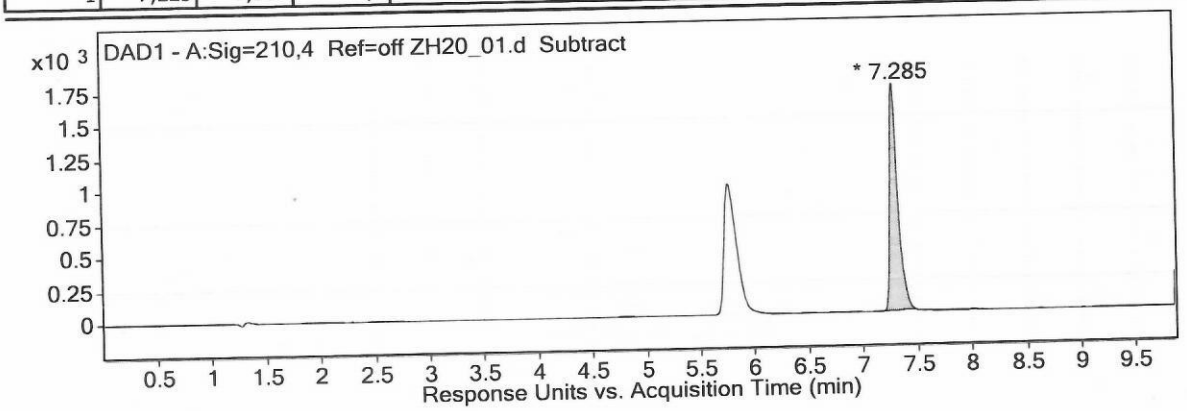
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,225	7,334	7,6	131778479,3	1619848058	100



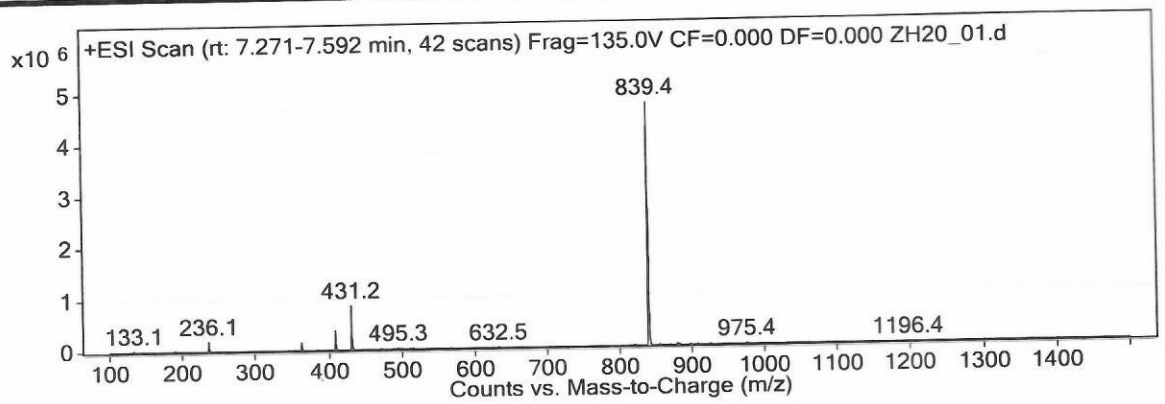
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,192	7,285	7,472	1733,59	8837,84	100

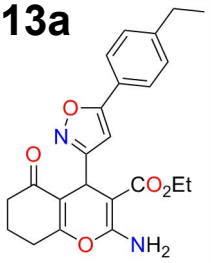
User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan Sub"	135	0	ESI

Qualitative Analysis Report



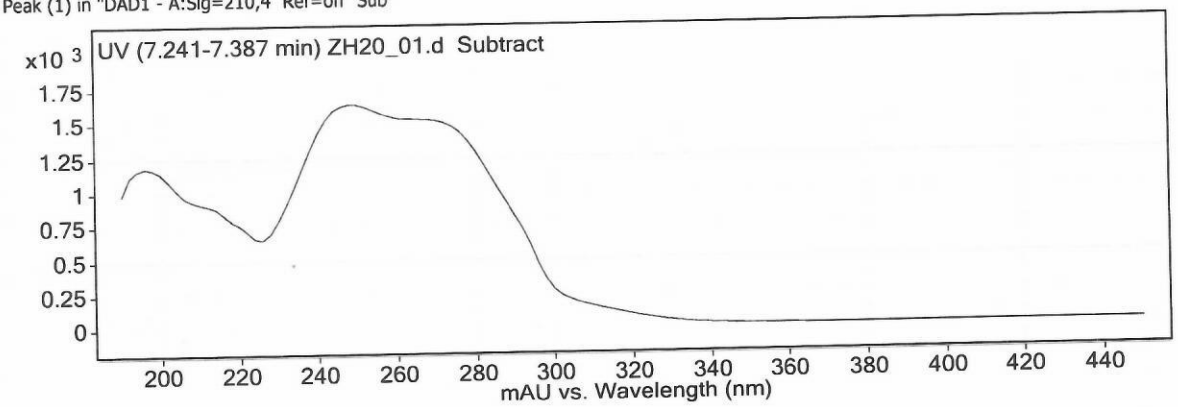
13a



Peak List

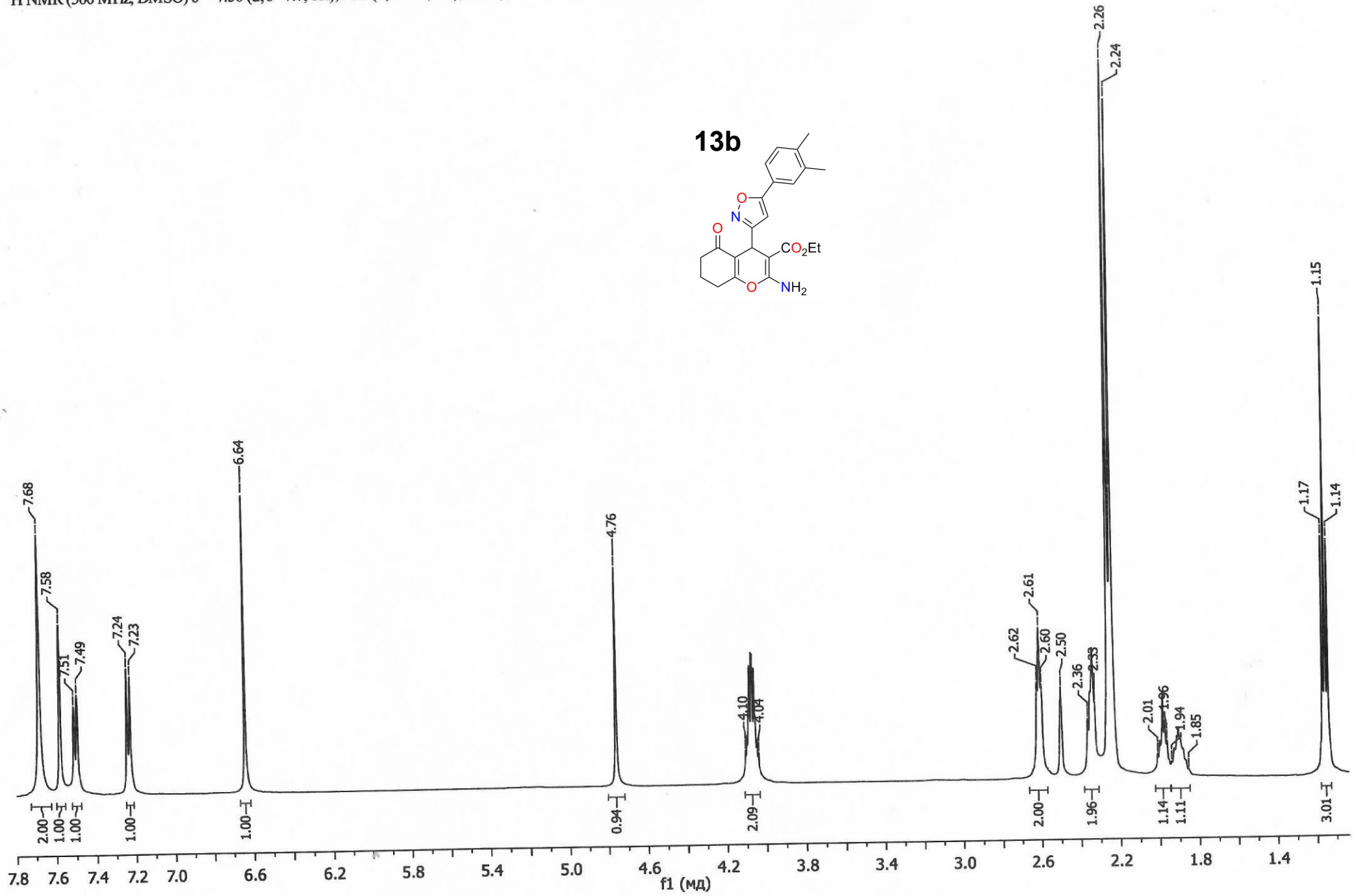
m/z	z	Abund
236.1	1	187663.42
363.2	1	152994.13
409.2	1	379000.94
410.2	1	97657.85
431.2	1	850581.88
432.2	1	212601.22
839.4	1	4725600
840.4	1	2613605.75
841.4	1	639827.13
842.4	1	130995.91

Spectrum Source
Peak (1) in "DAD1 - A:Sig=210,4 Ref=off Sub"

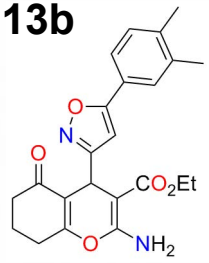


--- End Of Report ---

^{1}H NMR (500 MHz, DMSO) δ = 7.50 (d, *J*=7.7, 1H), 7.23 (d, *J*=7.9, 1H), 2.61 (t, *J*=5.8, 2H), 1.15 (t, *J*=7.1, 3H).



13b

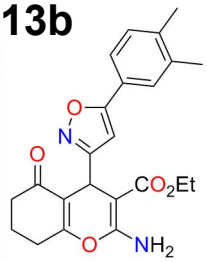

O
N
O
O
CO2Et
O
NH2

196.63
169.03
168.28
168.06
165.99
160.31
139.37
137.81
130.73
126.92
125.31
123.44
114.54
99.81
75.12
59.56
36.75
27.01
26.62
19.94
19.87
15.03
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (мд)

Qualitative Analysis Report

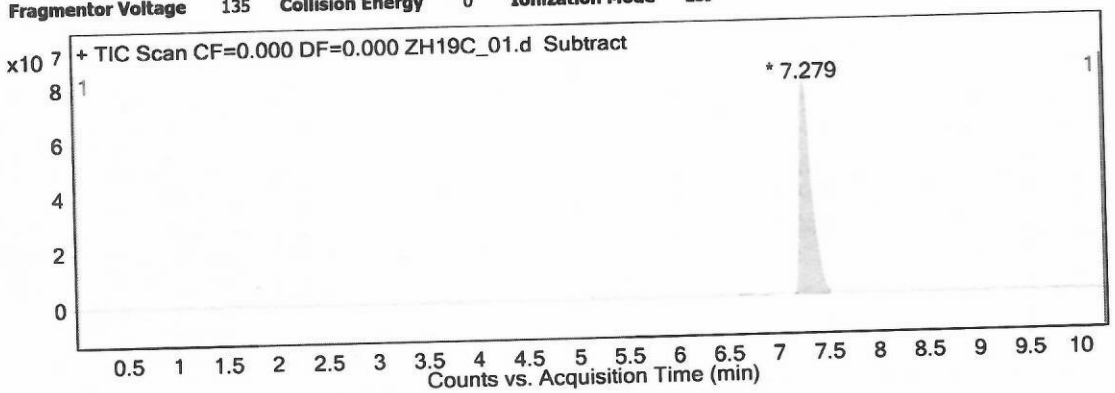
Data Filename	ZH19C_01.d	**Sample Name**	ZH19C
Sample Type	Sample	**Position**	Vial 2
Instrument Name	Instrument 1	**User Name**	
Acq Method	All_2021_kol 1-2.m	**Acquired Time**	5/29/2023 10:27:13 AM
IRM Calibration Status	Not Applicable	**DA Method**	Defaul1t.m
Comment			
Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition SW Version**	6400 Series Triple Quadrupole 10.0 (127)

13b



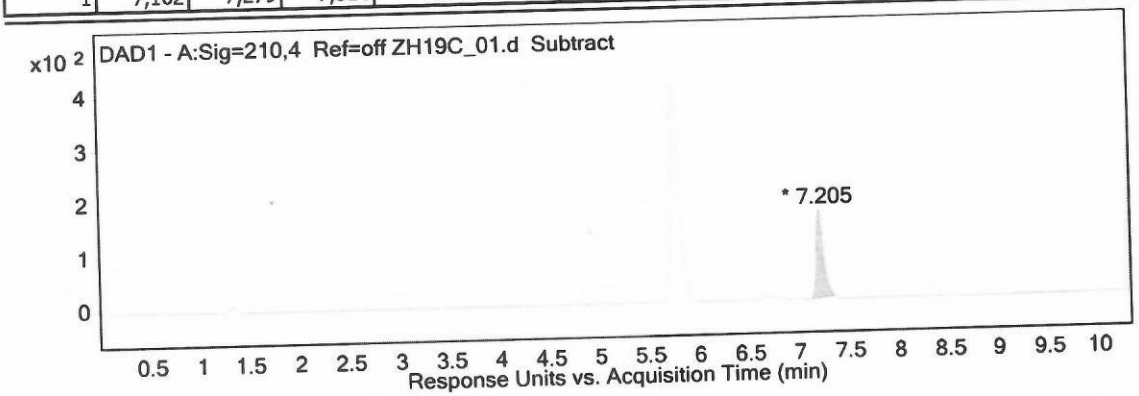
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,162	7,279	7,514	77600986,83	585234776,9	100



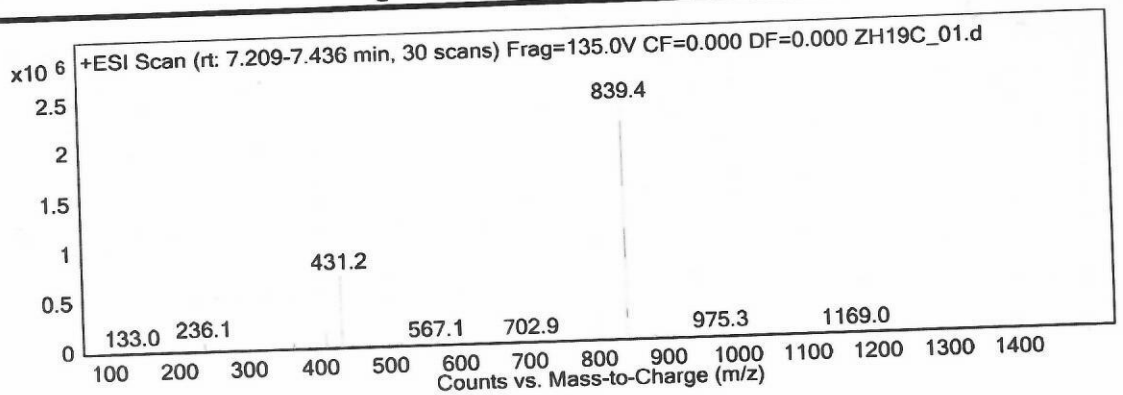
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,132	7,205	7,359	164,9	789,32	100

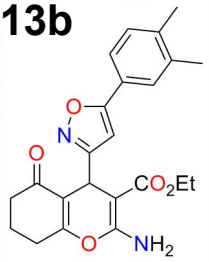
User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan Sub"	135	0	ESI

Qualitative Analysis Report



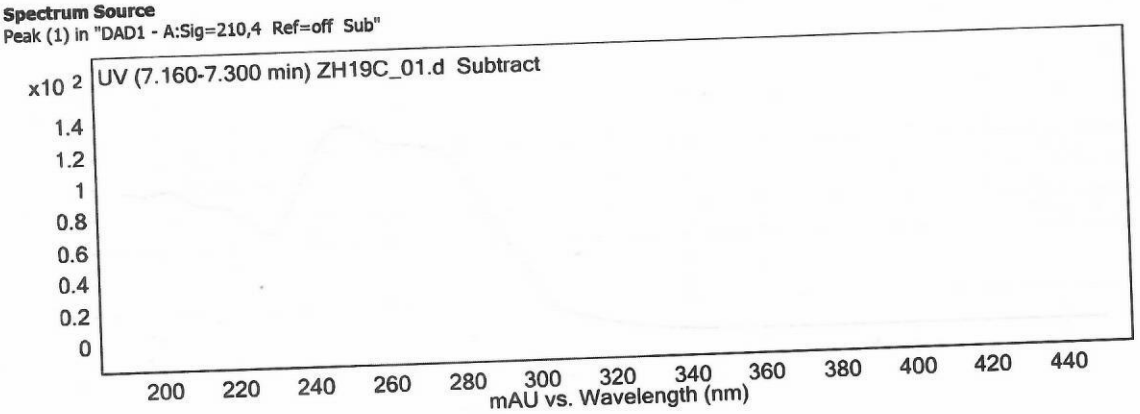
13b



Peak List

m/z	z	Abund
236.1	1	73888.09
363.1	1	48471.92
409.2	1	151166
410.2	1	37995.34
431.2	1	713686.69
432.2	1	177063.5
839.4	1	2341839.25
840.4	1	1101613.38
841.4	1	299019.13
842.4	1	62567.76

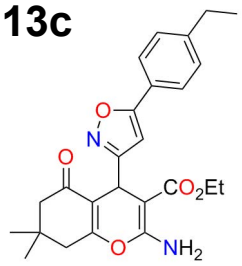
Spectrum Source
Peak (1) in "DAD1 - A:Sig=210,4 Ref=off Sub"



--- End Of Report ---

^{1}H NMR (500 MHz, DMSO) δ = 7.71 (d, *J*=7.8, 1H), 7.33 (d, *J*=7.5, 1H), 2.64 (dd, *J*=14.2, 6.9, 1H), 2.58 (d, *J*=18.0, 1H), 2.44 (d, *J*=17.7, 1H), 2.34 (d, *J*=16.1, 1H), 2.16 (d, *J*=16.0, 1H).

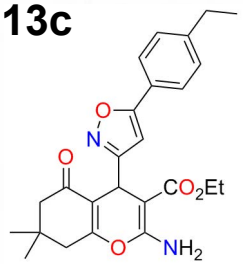
13c



SK_zh-16_05102023

bbo_13CF_bar DMSO /v nmrsu 10

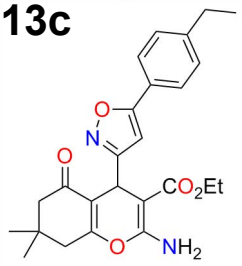
13c



Qualitative Analysis Report

Data Filename	ZH6C_01.d	**Sample Name**	ZH6C
Sample Type	Sample	**Position**	Vial 2
Instrument Name	Instrument 1	**User Name**	
Acq Method	All_2021_kol 1-2.m	**Acquired Time**	5/29/2023 10:10:20 AM
IRM Calibration Status	Not Applicable	**DA Method**	Defaul1t.m
Comment			

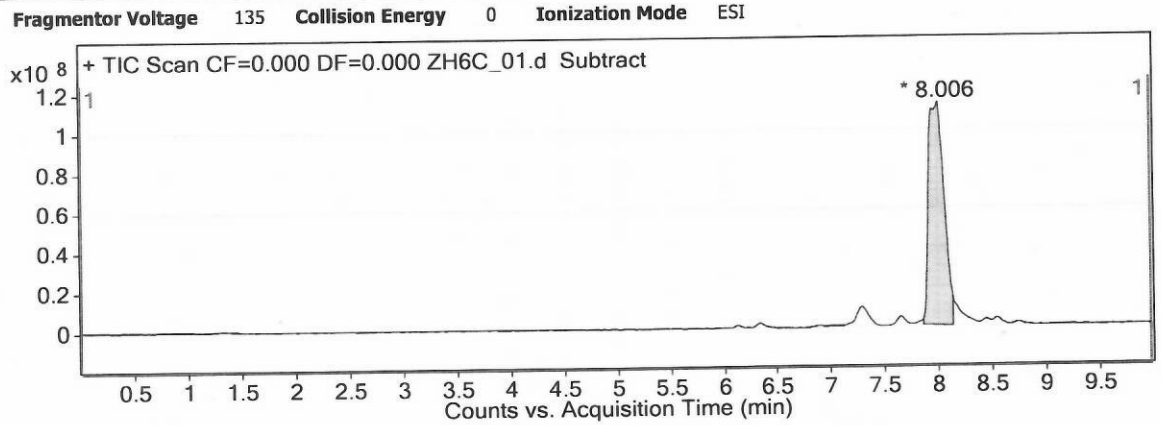
13c



Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition SW Version**	6400 Series Triple Quadrupole 10.0 (127)

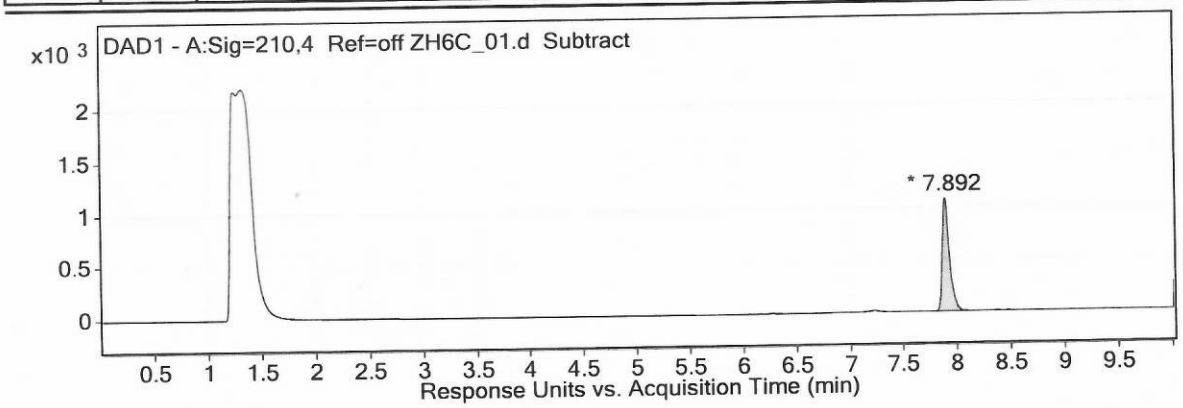
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,857	8,006	8,131	112508888,3	1113805576	100



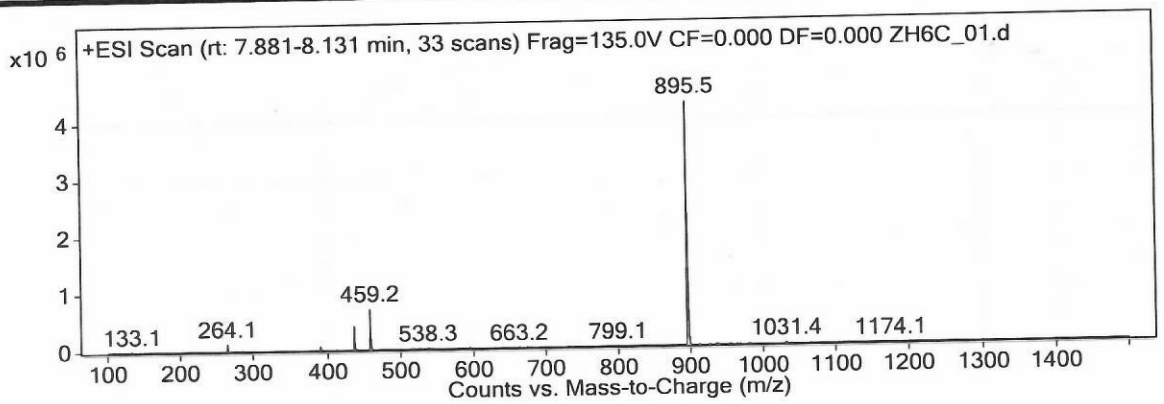
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,805	7,892	8,079	1077,45	4904,71	100

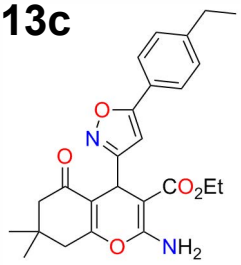
User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan Sub"	135	0	ESI

Qualitative Analysis Report



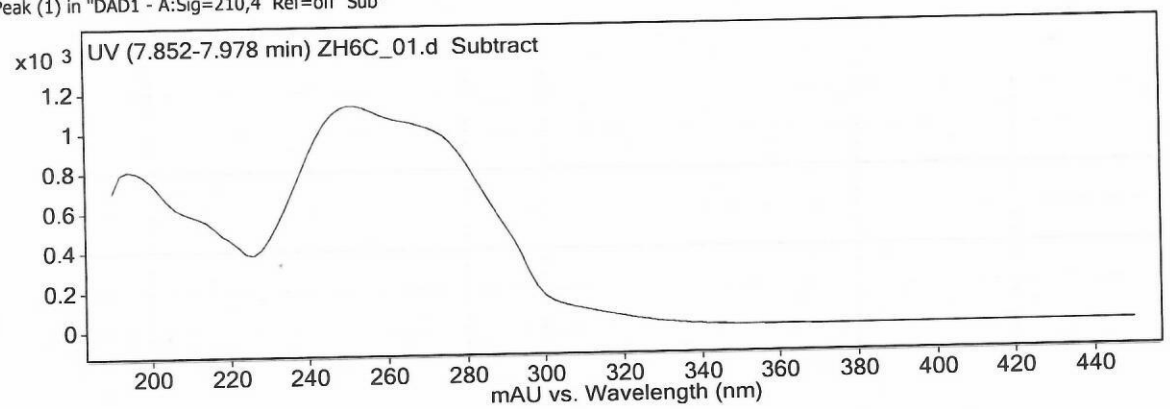
13c



Peak List

m/z	z	Abund
264.1	1	120717.73
391.2	1	63457.22
437.2	1	418541.94
438.2	1	114552.61
459.2	1	710954.56
460.2	1	189415.41
895.5	1	4291627
896.5	1	2314255
897.5	1	620106.69
898.5	1	137651.42

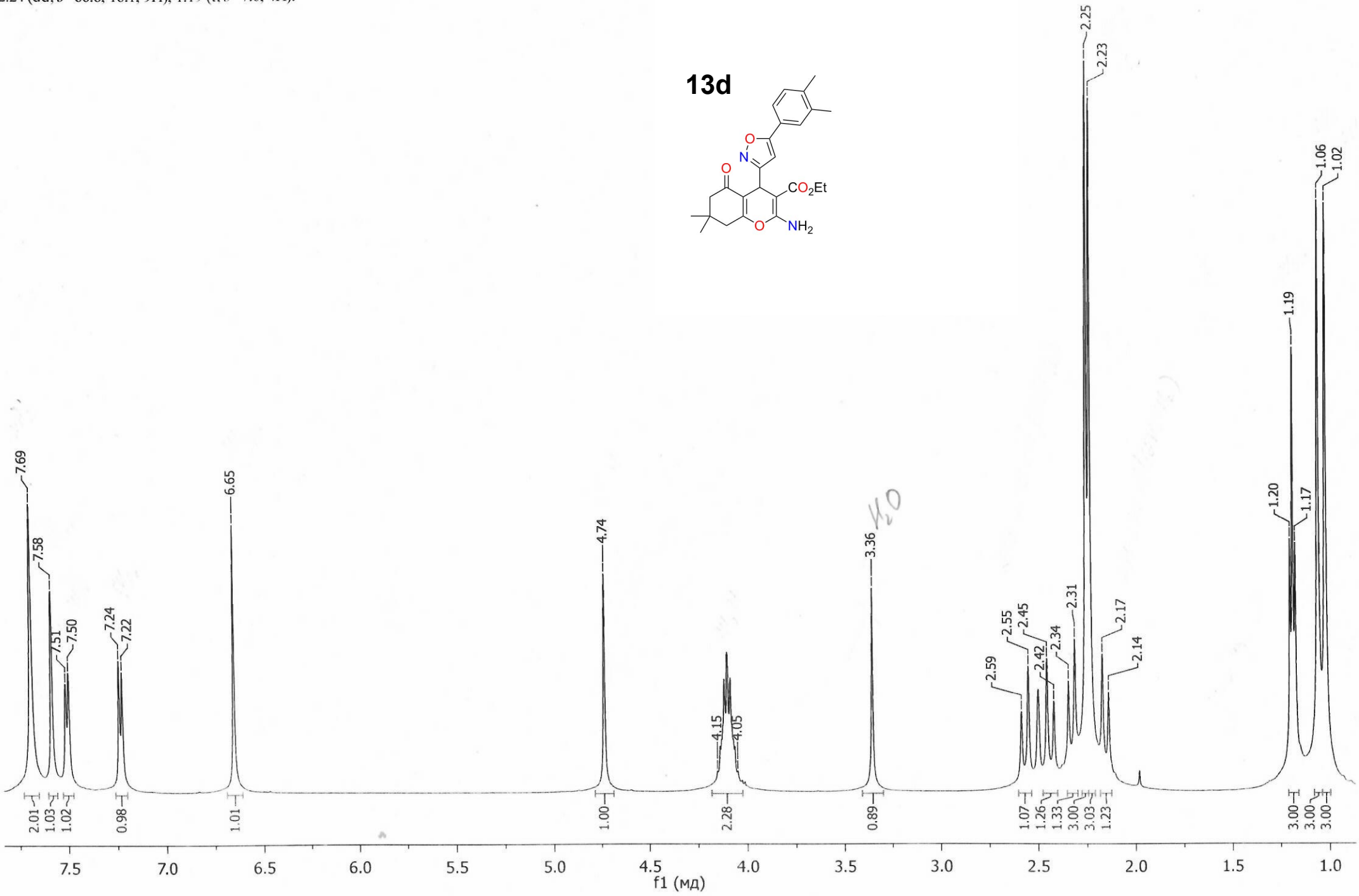
Spectrum Source
Peak (1) in "DAD1 - A:Sig=210,4 Ref=off Sub"



--- End Of Report ---

^{1}H NMR (500 MHz, DMSO) δ = 7.50 (d, *J*=7.6, 1H), 7.23 (d, *J*=7.8, 1H), 2.50 (dd, *J*=65.5, 17.6, 3H), 2.24 (dd, *J*=86.8, 16.1, 9H), 1.19 (t, *J*=7.0, 4H).

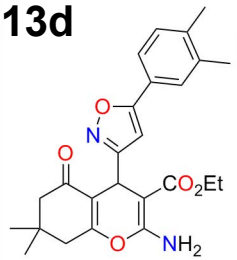
13d



SK_zh5c_05242023

bbo_13CF_bar DMSO /v nmrsu 12

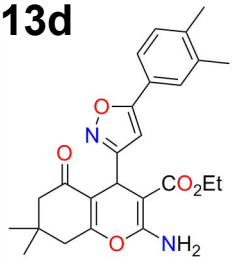
13d



Qualitative Analysis Report

Data Filename	ZH5C_01.d	**Sample Name**	ZH5C
Sample Type	Sample	**Position**	Vial 2
Instrument Name	Instrument 1	**User Name**	
Acq Method	All_2021_kol 1-2.m	**Acquired Time**	5/29/2023 9:54:12 AM
IRM Calibration Status	Not Applicable	**DA Method**	Defaul1t.m
Comment			

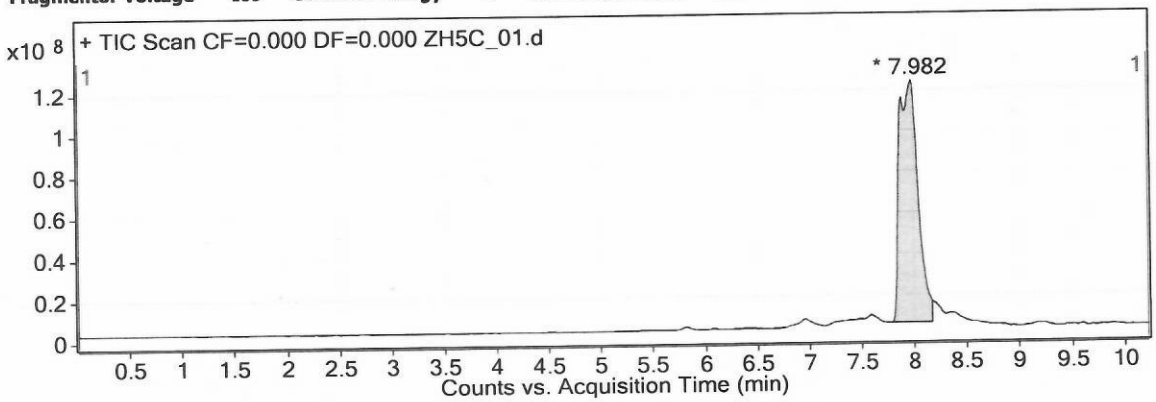
13d



Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition SW Version**	6400 Series Triple Quadrupole 10.0 (127)

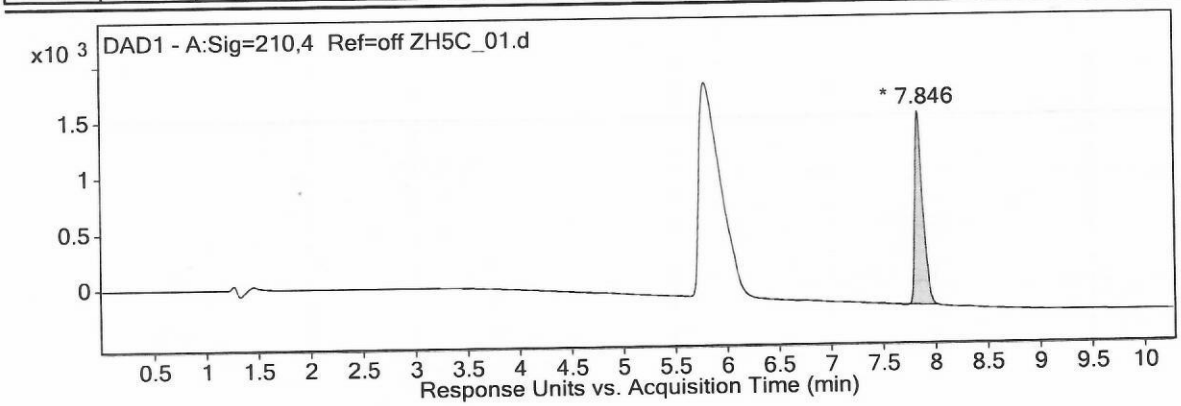
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,779	7,982	8,17	118128859,7	1458320768	100



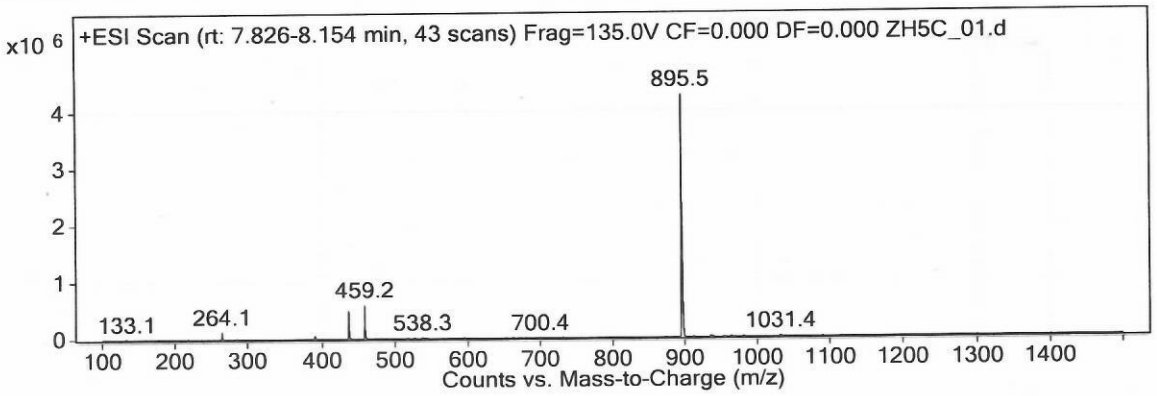
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	7,759	7,846	8,032	1738,86	9135,32	100

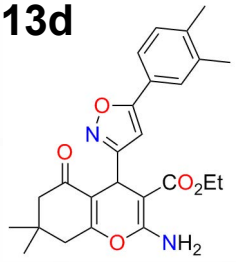
User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan"	135	0	ESI

Qualitative Analysis Report



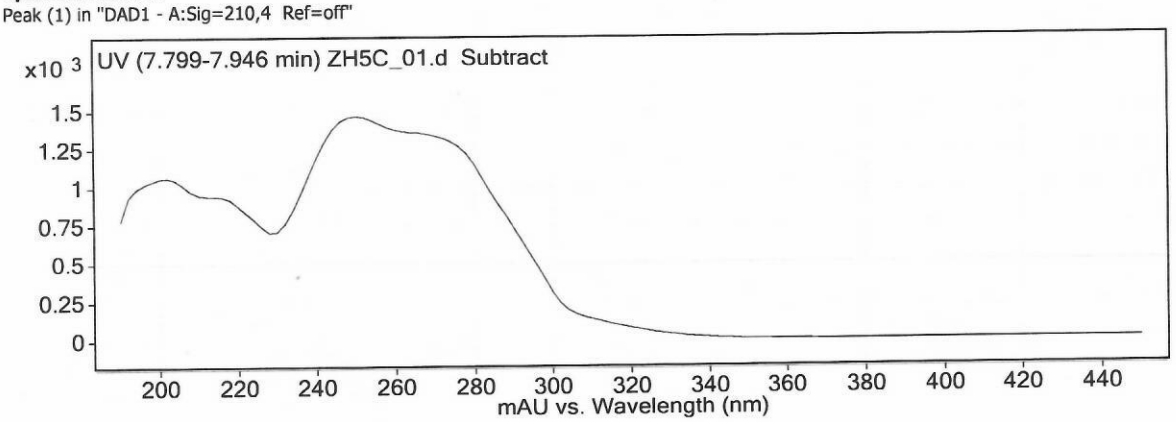
13d



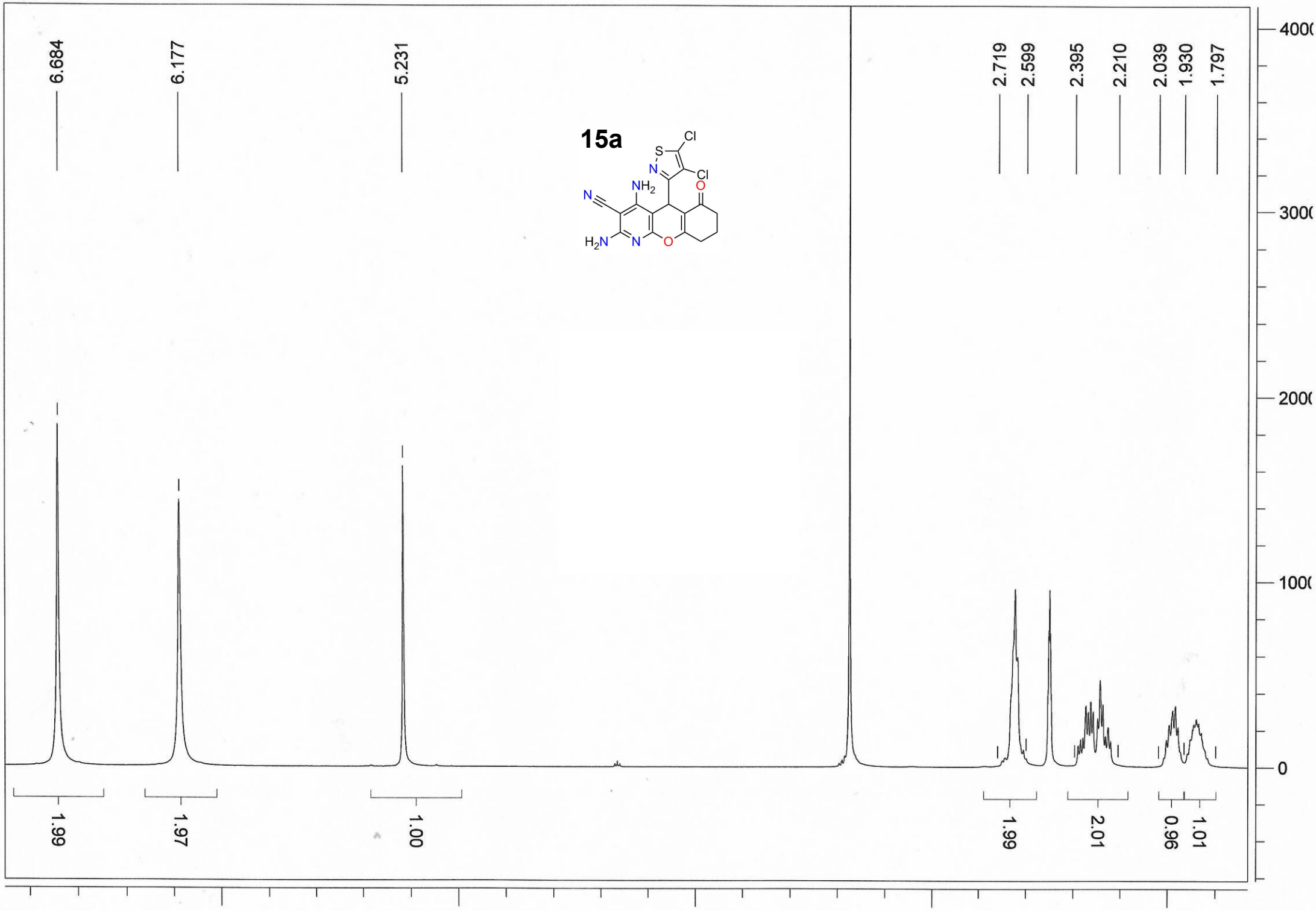
Peak List

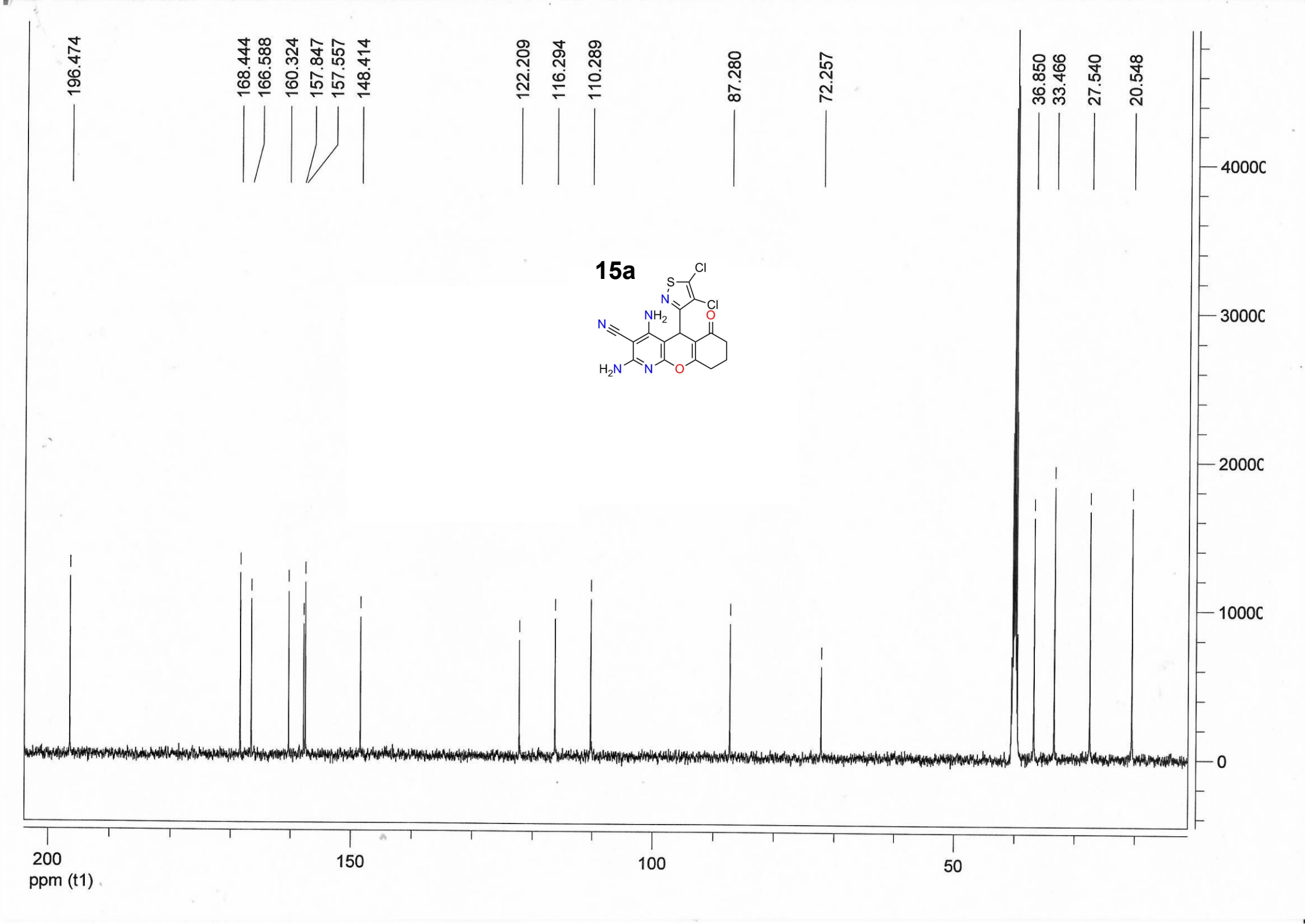
m/z	z	Abund
264.1	1	128387.64
391.2	1	56554.41
437.2	1	500249.72
438.2	1	131228.06
459.2	1	602584
460.2	1	163088.91
895.5	1	4295738
896.5	1	2353243.25
897.5	1	633835.63
898.5	1	133971.08

Spectrum Source
Peak (1) in "DAD1 - A:Sig=210,4 Ref=off"



--- End Of Report ---

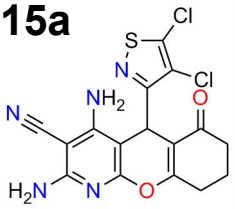

6.684
6.177
5.231
2.719
2.599
2.395
2.210
2.039
1.930
1.797
15a
Cl
S
N
Cl
NH2
O
N
H2N
N
O
1.99
1.97
1.00
1.99
2.01
0.96
1.01
4000
3000
2000
1000
0


196.474
168.444
166.588
160.324
157.847
157.557
148.414
122.209
116.294
110.289
87.280
72.257
36.850
33.466
27.540
20.548
15a
Cl
S
N
Cl
NH2
O
N
H2N
N
O
4000
3000
2000
1000
0
200
150
100
50
ppm (t1)

Qualitative Analysis Report

Data Filename	HP-9_t1.d	**Sample Name**	HP-9
Sample Type	Sample	**Position**	Vial 3
Instrument Name	Instrument 1	**User Name**	
Acq Method	All_2021_kol 1-6.m	**Acquired Time**	2/27/2024 12:49:38 PM
IRM Calibration Status	Not Applicable	**DA Method**	Defaul1t.m
Comment			

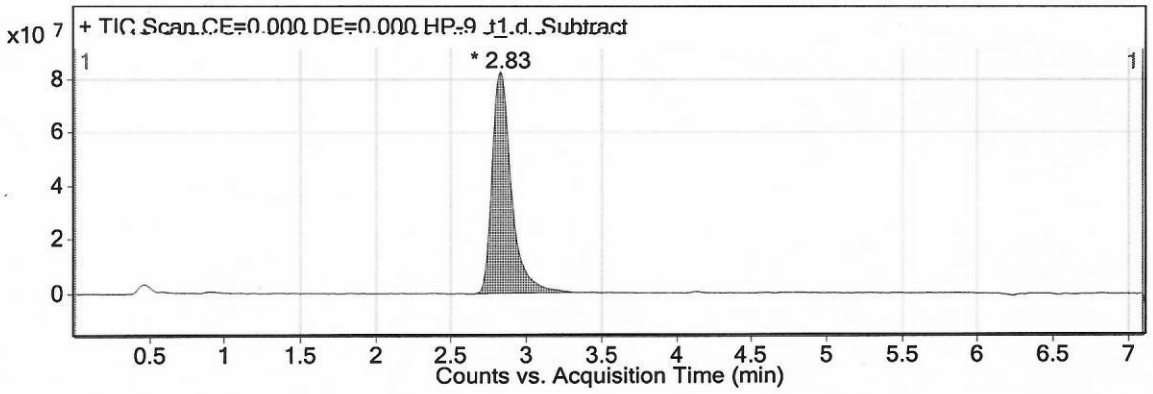
15a



Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition SW Version**	6400 Series Triple Quadrupole 10.0 (127)

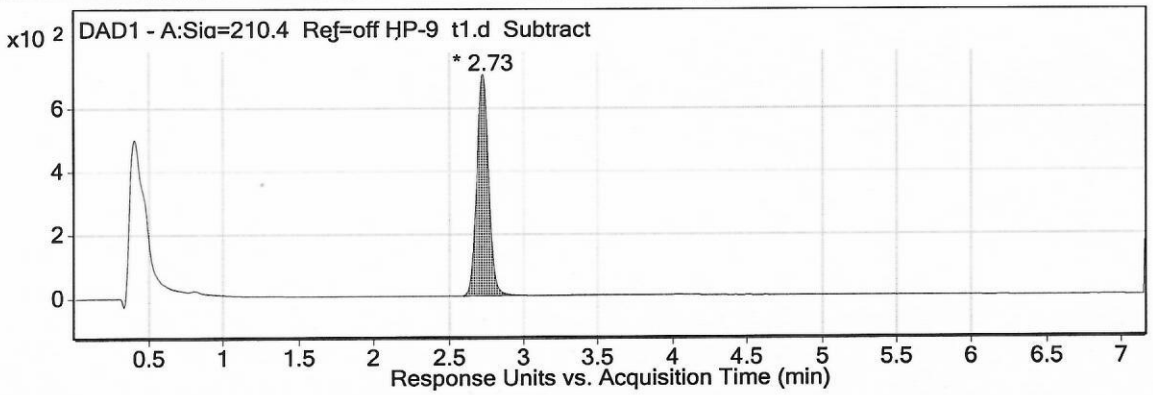
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	2,66	2,83	3,3	82614705,09	730795846,2	100



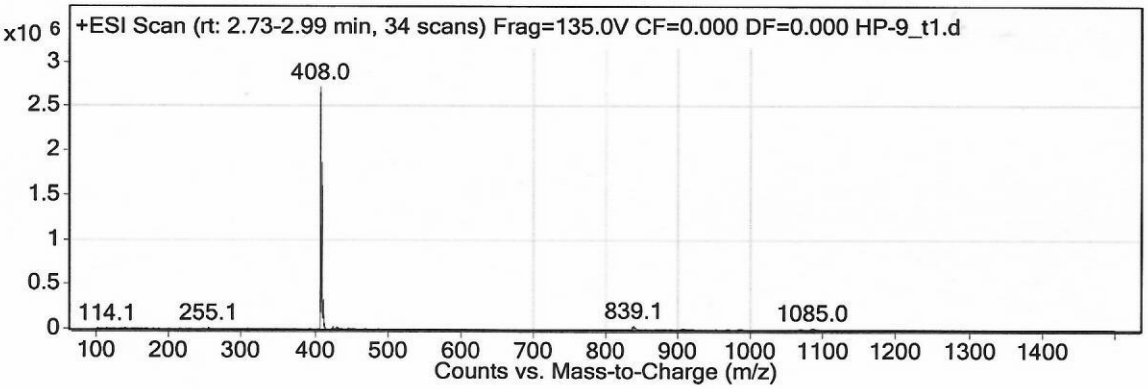
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	2,53	2,73	2,96	701,22	3868,69	100

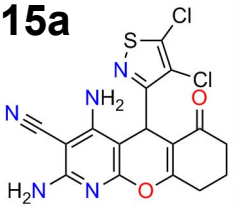
User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan Sub"	135	0	ESI

Qualitative Analysis Report



15a

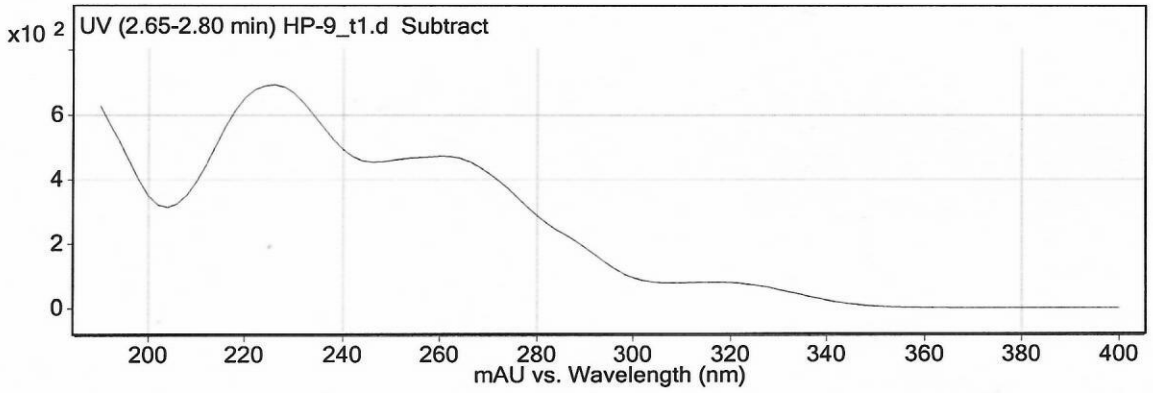


Peak List

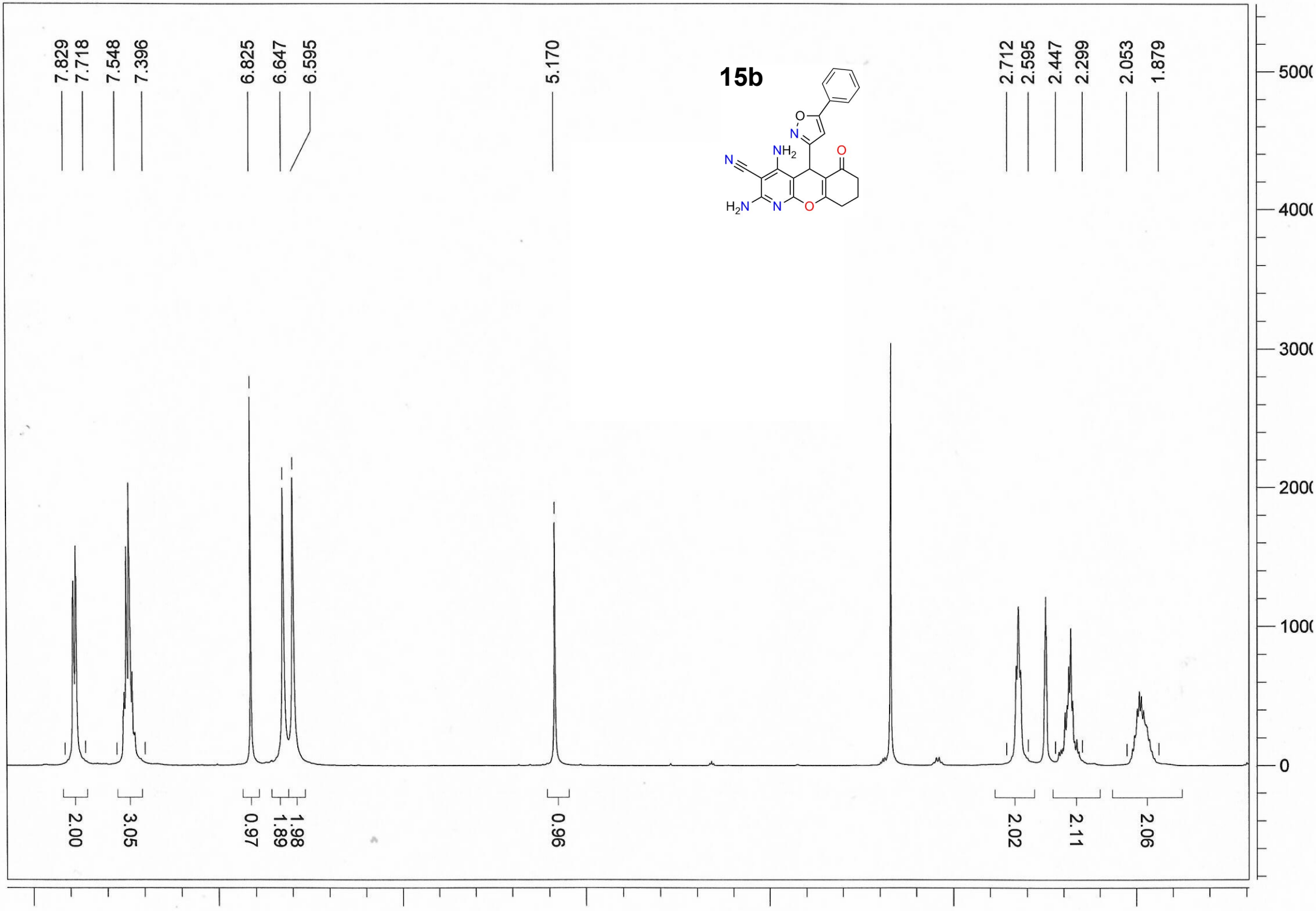
m/z	z	Abund
408		2719980
409.1		492485.25
410	1	1865634.5
411	1	313240.31
412	1	331431.59
413	1	62470.07
424.9	1	27148.55
837		28895.22
839.1	1	40868.66
841	1	24664.24

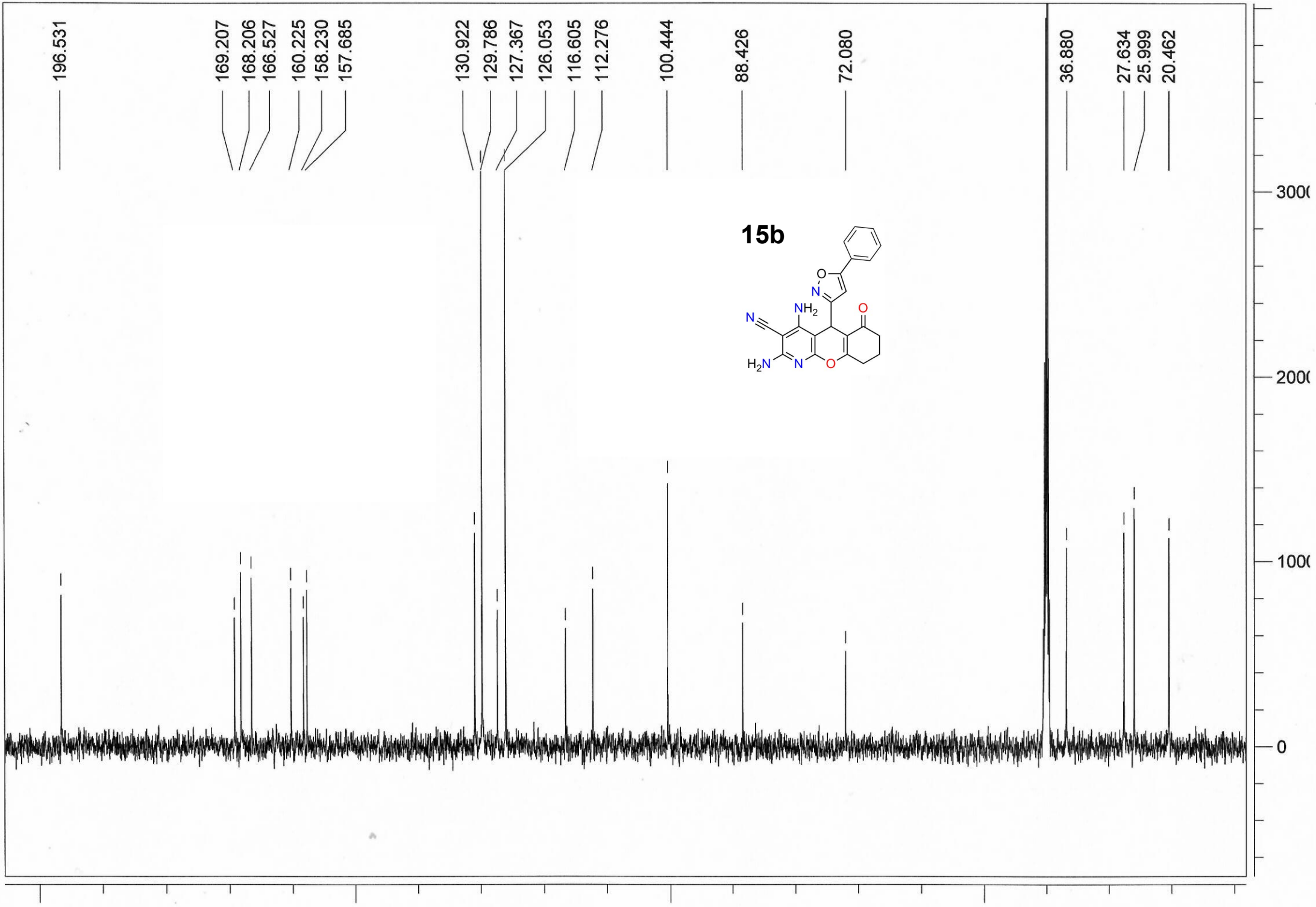
Spectrum Source

Peak (1) in "DAD1 - A:Sig=210,4 Ref=off Sub"



--- End Of Report ---

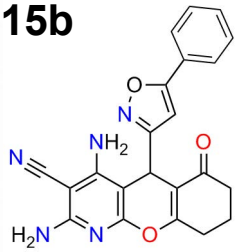

15b
7.829
7.718
7.548
7.396
6.825
6.647
6.595
5.170
2.712
2.595
2.447
2.299
2.053
1.879
2.00
3.05
0.97
1.89
1.98
0.96
2.02
2.11
2.06
500
400
300
200
100
0


196.531
169.207
168.206
166.527
160.225
158.230
157.685
130.922
129.786
127.367
126.053
116.605
112.276
100.444
88.426
72.080
36.880
27.634
25.999
20.462
15b
300
200
100
0

Qualitative Analysis Report

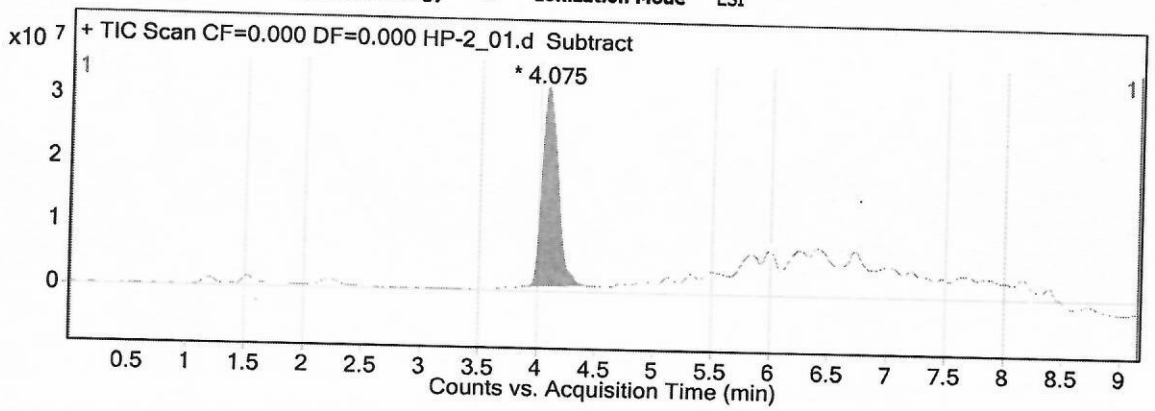
Data Filename	HP-2_01.d	**Sample Name**	HP-2
Sample Type	Sample	**Position**	Vial 3
Instrument Name	Instrument 1	**User Name**	
Acq Method	All_2021_kol 1-2.m	**Acquired Time**	4/13/2023 10:46:28 AM
IRM Calibration Status	Not Applicable	**DA Method**	Defaul1t.m
Comment			
Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition SW Version**	6400 Series Triple Quadrupole 10.0 (127)

15b



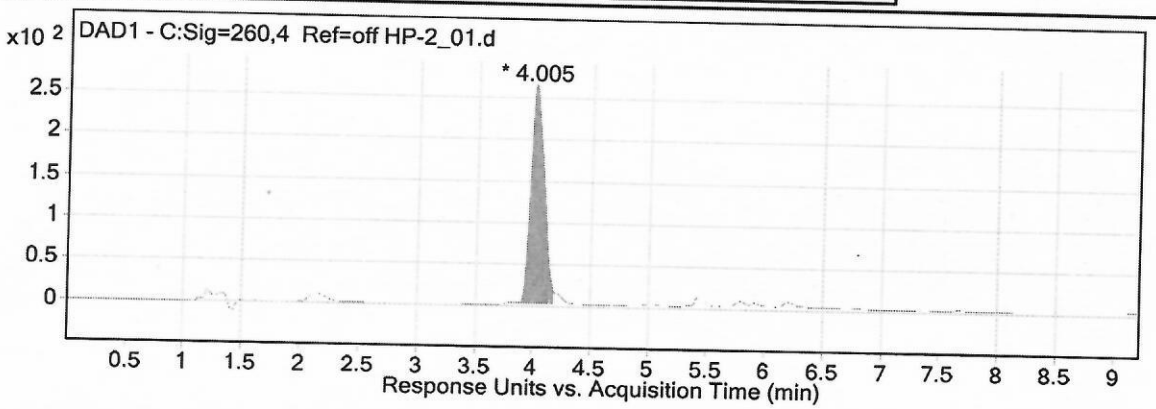
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	3,913	4,075	4,405	31619332,67	291820617,5	100



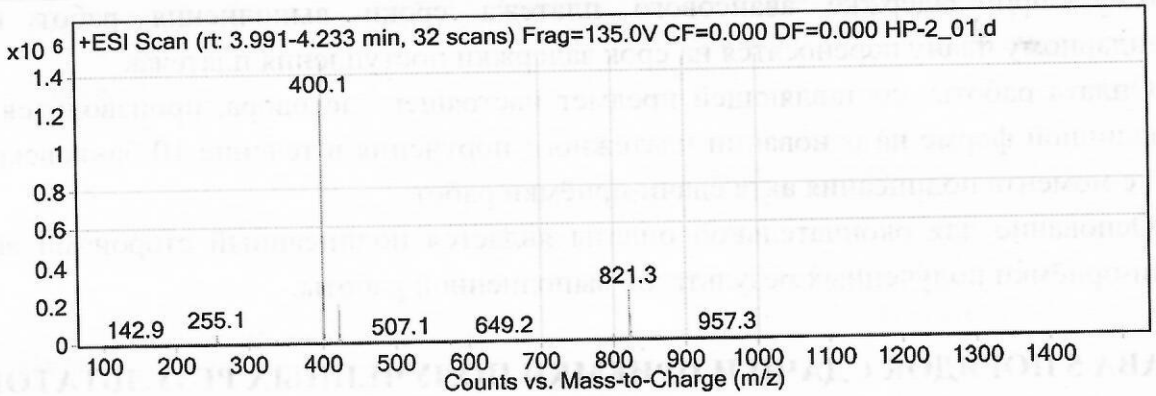
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	3,885	4,005	4,171	261,99	2065,8	100

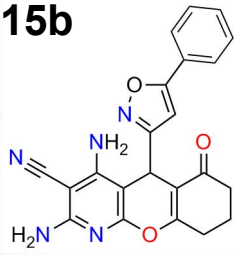
User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan Sub"	135	0	ESI

Qualitative Analysis Report



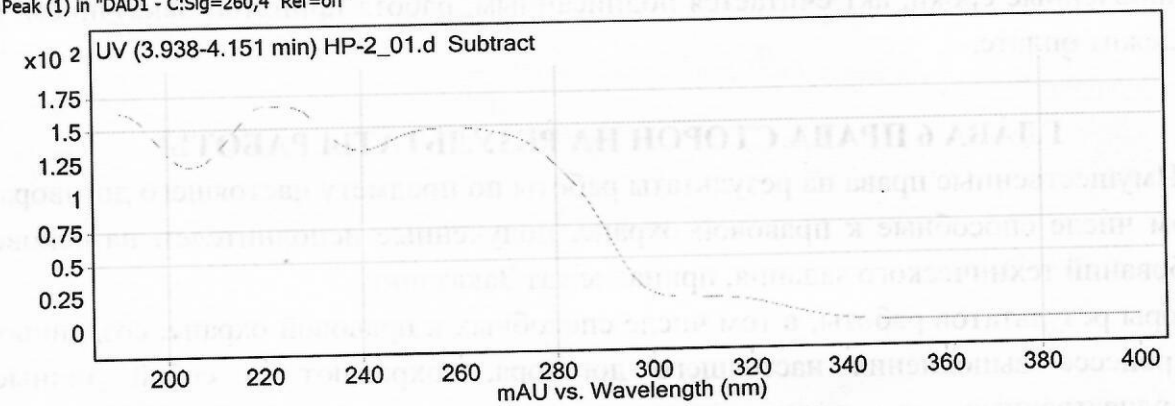
15b



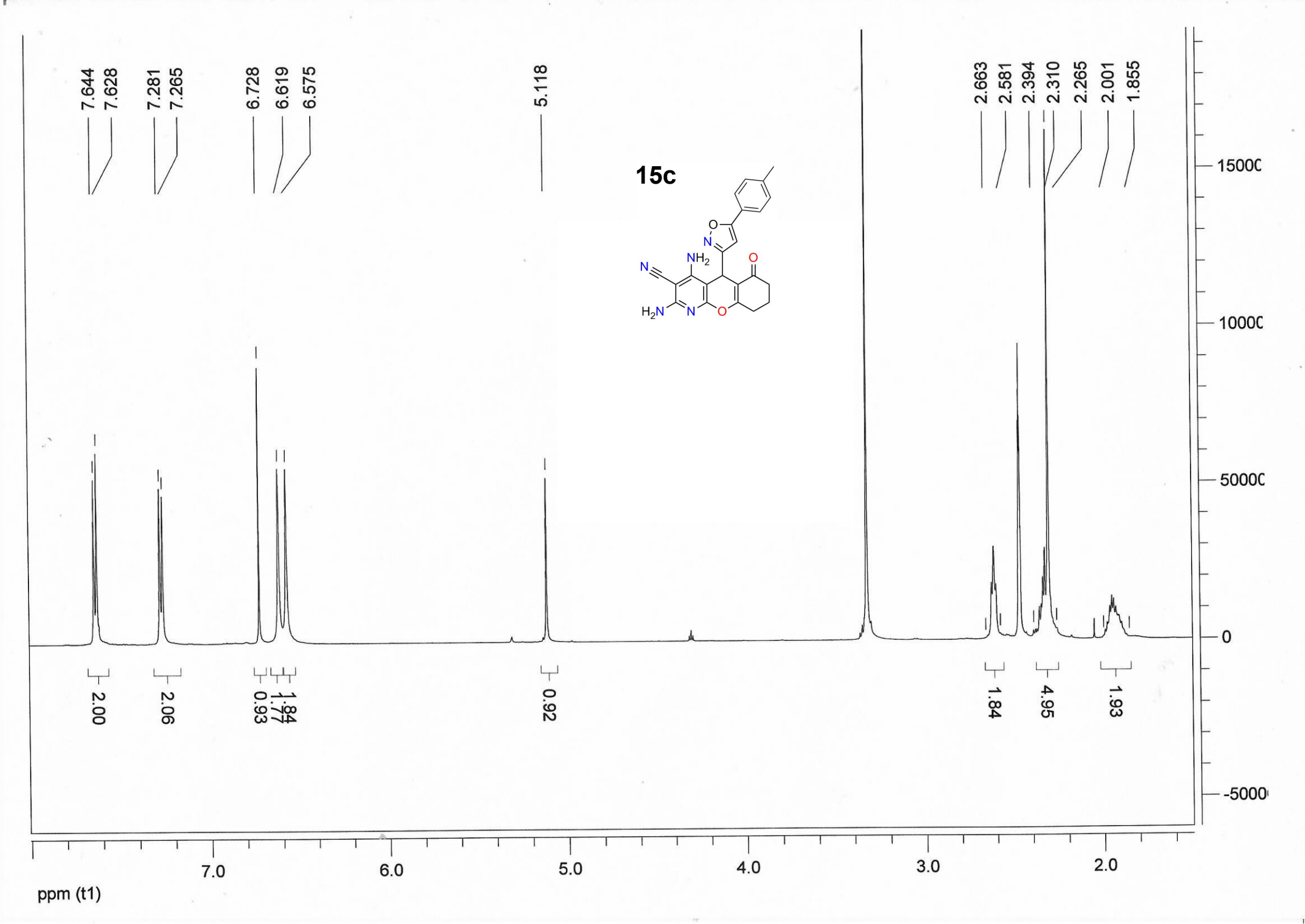
Peak List

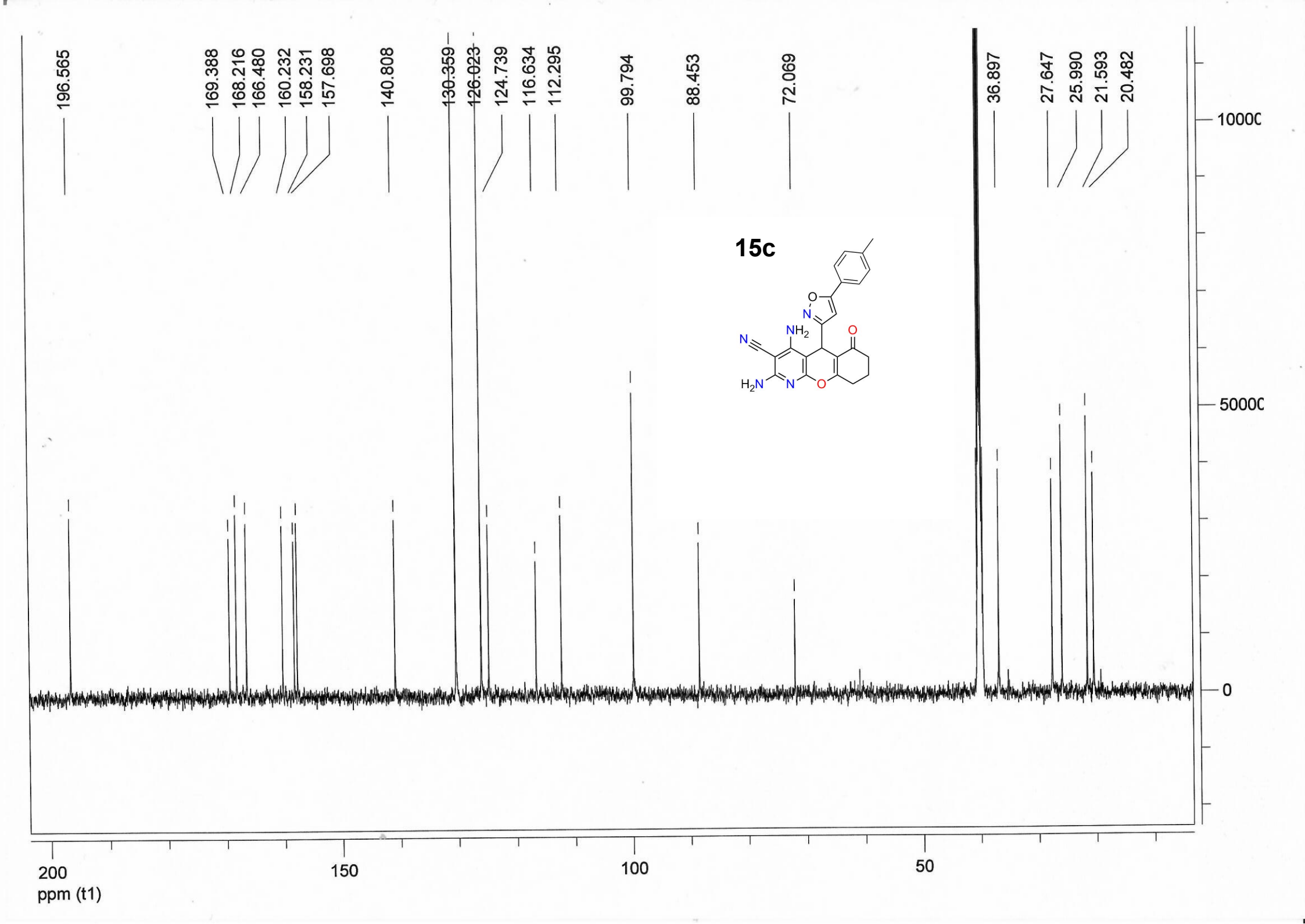
m/z	z	Abund
255.1	1	47228.23
400.1	1	1274338.25
401.1	1	300627.16
402.1	1	44883.64
422.1	1	197416.97
423.1	1	50234.55
799.3	1	19844.97
821.3	1	251334.08
822.3	1	125602.36
823.3	1	35859.36

Spectrum Source
Peak (1) in "DAD1 - C:Sig=260,4 Ref=off"



--- End Of Report ---

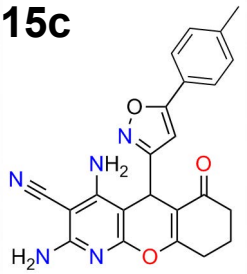

7.644
7.628
7.281
7.265
6.728
6.619
6.575
5.118
2.663
2.581
2.394
2.310
2.265
2.001
1.855
15c
NH2
N
O
N
O
H2N
N
O
2.00
2.06
0.93
1.77
1.84
0.92
1.84
4.95
1.93
15000
10000
5000
0
-5000
7.0
6.0
5.0
4.0
3.0
2.0
ppm (t1)


196.565
169.388
168.216
166.480
160.232
158.231
157.698
140.808
130.359
126.023
124.739
116.634
112.295
99.794
88.453
72.069
36.897
27.647
25.990
21.593
20.482
15c
10000
50000
0
200
150
100
50
ppm (t1)

Qualitative Analysis Report

Data Filename	HP-12_k01.d	**Sample Name**	HP-12
Sample Type	Sample	**Position**	Vial 2
Instrument Name	Instrument 1	**User Name**	
Acq Method	All_2021_kol 1-6.m	**Acquired Time**	2/19/2024 12:18:34 PM
IRM Calibration Status	Not Applicable	**DA Method**	Defaul1t.m
Comment			

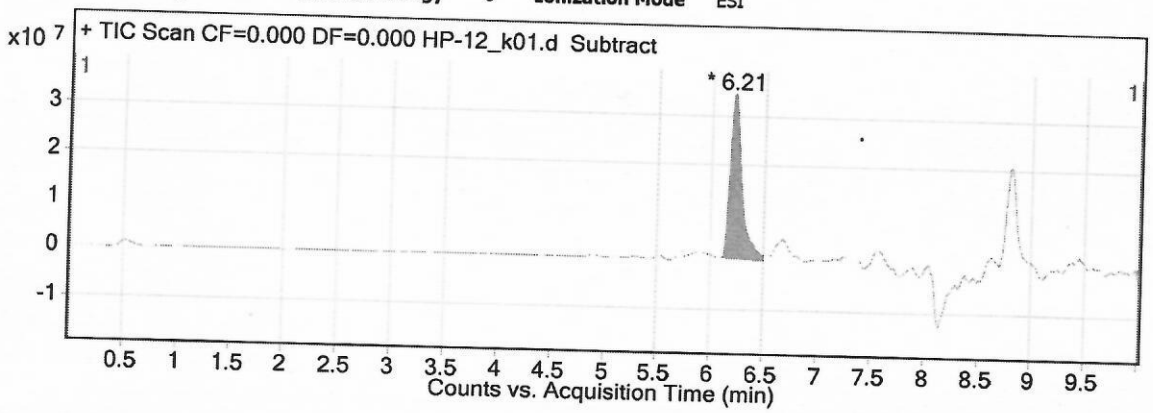
15c



Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition SW Version**	6400 Series Triple Quadrupole 10.0 (127)

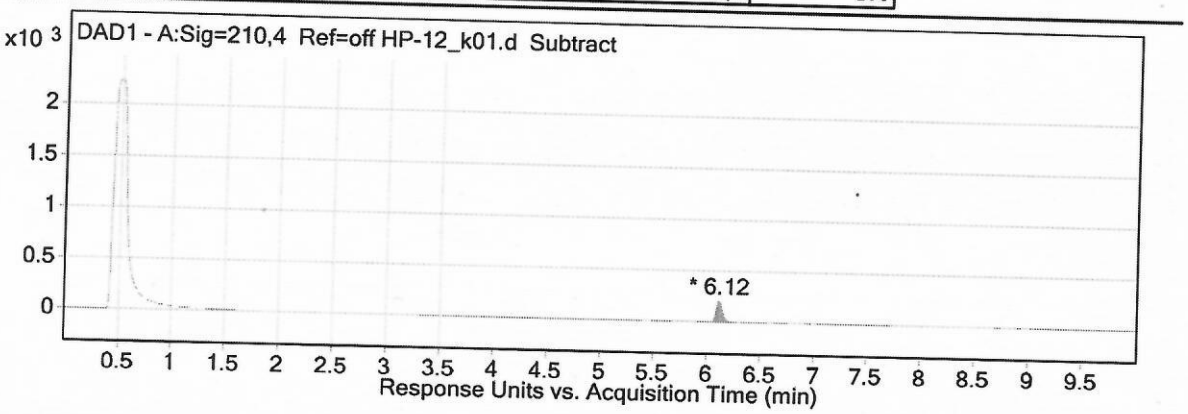
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	6,1	6,21	6,5	34068581,15	268022536,3	100



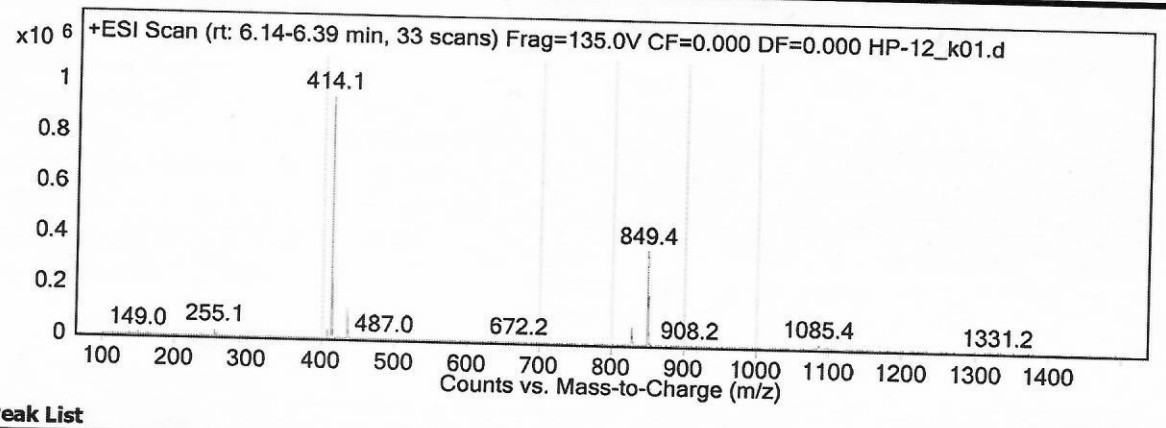
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	6,04	6,12	6,24	202,2	789,34	100

User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan Sub"	135	0	ESI

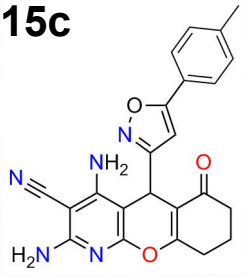
Qualitative Analysis Report



Peak List

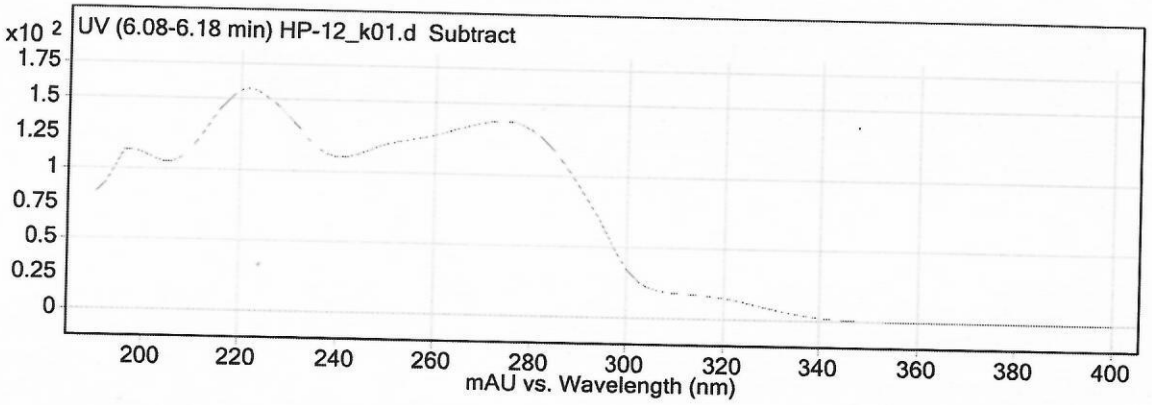
m/z	z	Abund
414.1		955572.75
415.2	1	236250.86
416.2	1	36652.49
436.1	1	126425.88
437.1	1	35644.69
827.4	1	70220.91
828.4	1	37023.97
849.4	1	370789.28
850.4	1	197895.2
851.4	1	55879.56

15c

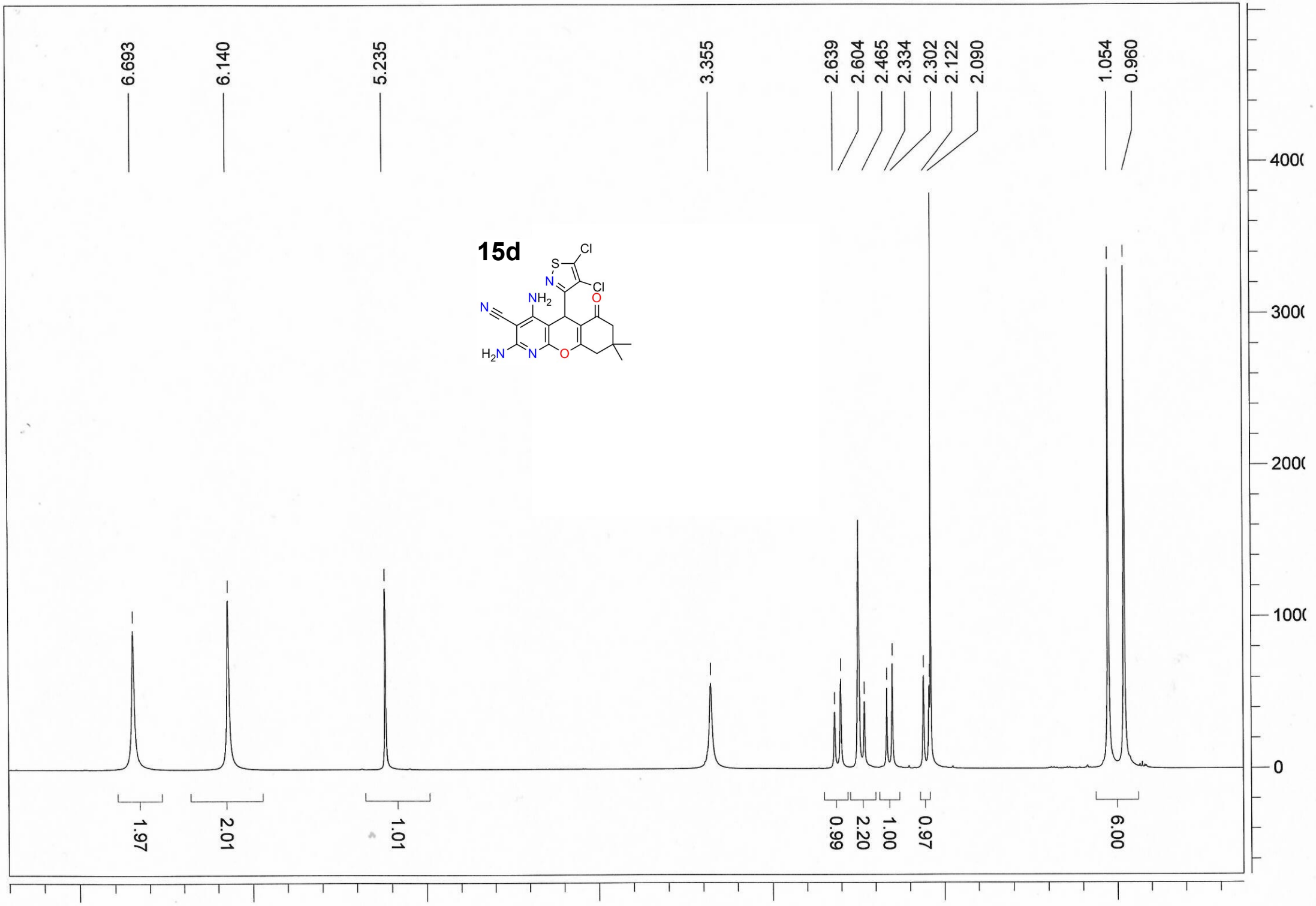


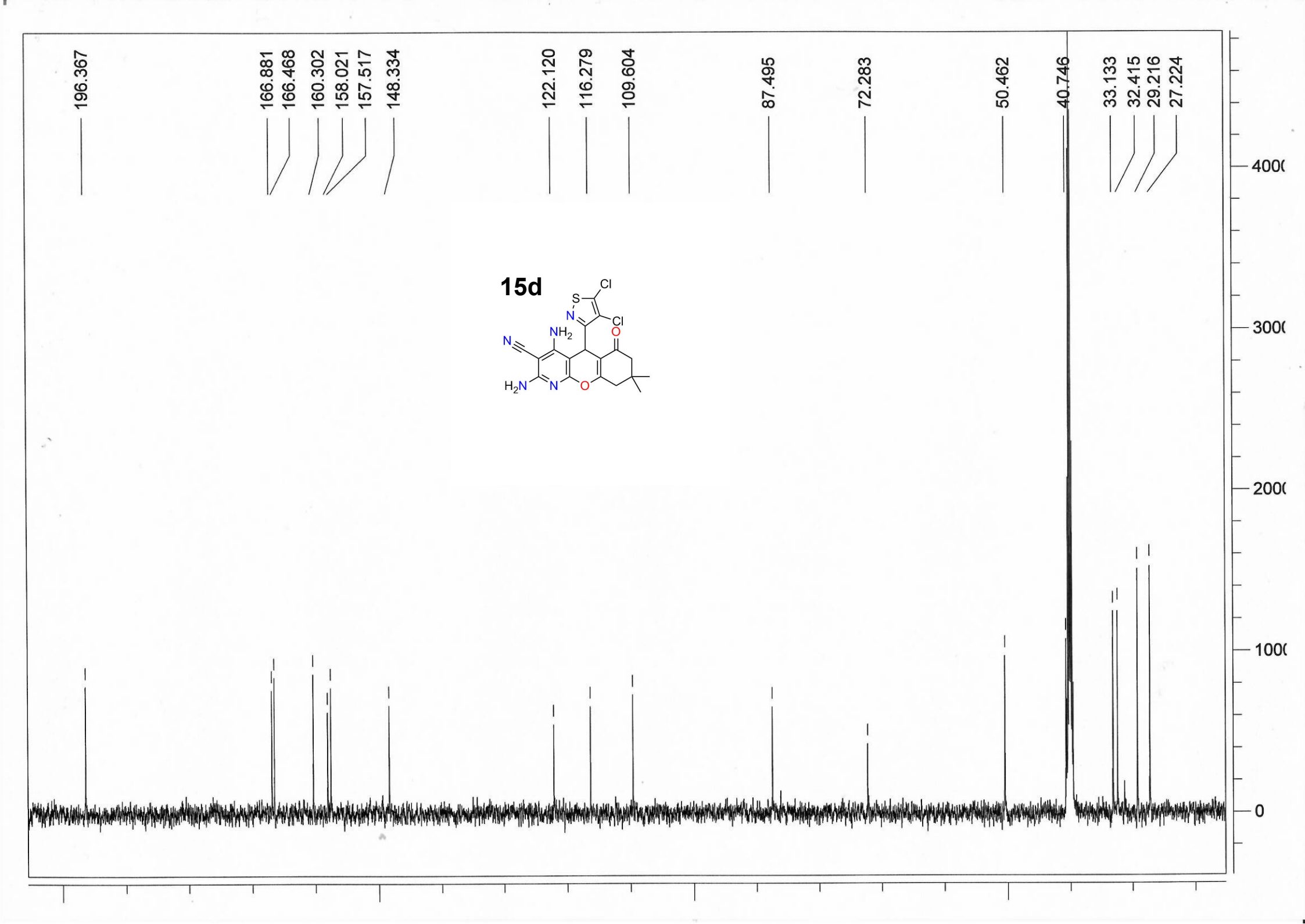
Spectrum Source

Peak (1) in "DAD1 - A:Sig=210,4 Ref=off Sub"



--- End Of Report ---

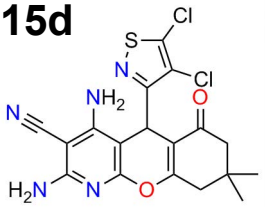

6.693
6.140
5.235
3.355
2.639
2.604
2.465
2.334
2.302
2.122
2.090
1.054
0.960
15d
Cl
S
N
Cl
NH2
O
N
H2N
N
O
400
300
200
100
0
1.97
2.01
1.01
0.99
2.20
1.00
0.97
6.00


196.367
166.881
166.468
160.302
158.021
157.517
148.334
122.120
116.279
109.604
87.495
72.283
50.462
40.746
33.133
32.415
29.216
27.224
15d
Cl
S
N
Cl
NH2
N
O
H2N
N
O
400
300
200
100
0

Qualitative Analysis Report

Data Filename	HP-10_k01.d	**Sample Name**	HP-10
Sample Type	Sample	**Position**	Vial 2
Instrument Name	Instrument 1	**User Name**	
Acq Method	All_2021_kol 1-6.m	**Acquired Time**	2/19/2024 11:45:23 AM
IRM Calibration Status	Not Applicable	**DA Method**	Defaul1t.m
Comment			

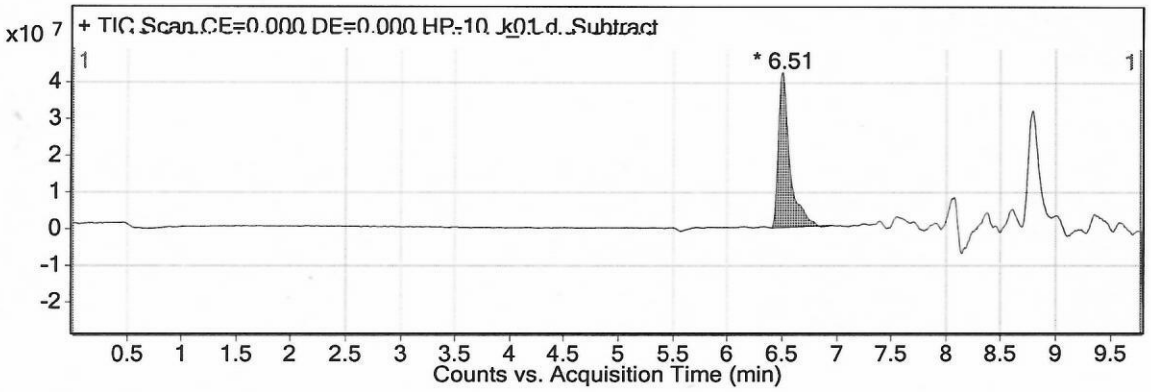
15d



Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition SW Version**	6400 Series Triple Quadrupole 10.0 (127)

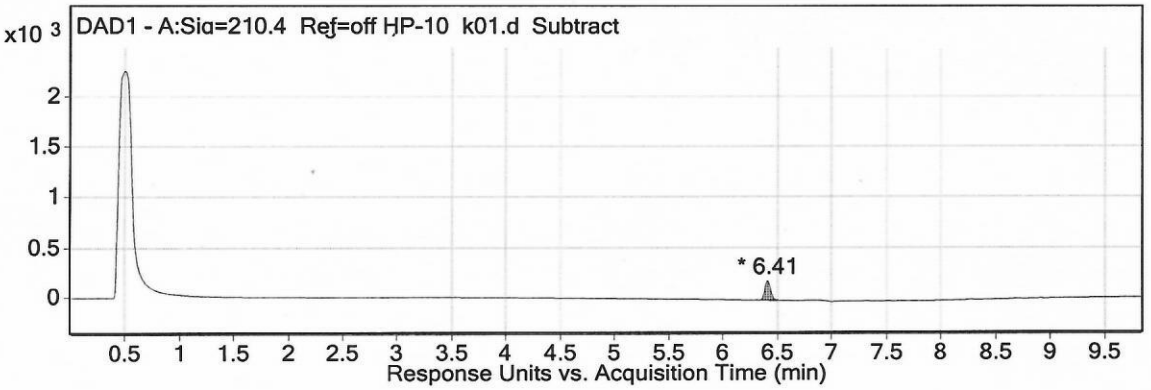
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	6,4	6,51	6,97	42483030,16	307213165,9	100



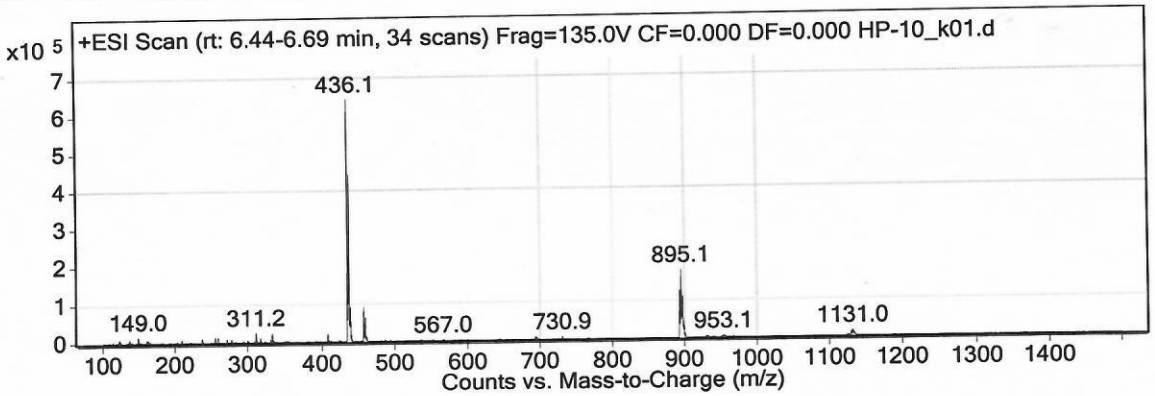
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	6,35	6,41	6,5	192,41	694,8	100

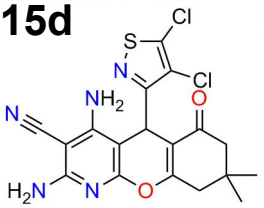
User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan Sub"	135	0	ESI

Qualitative Analysis Report



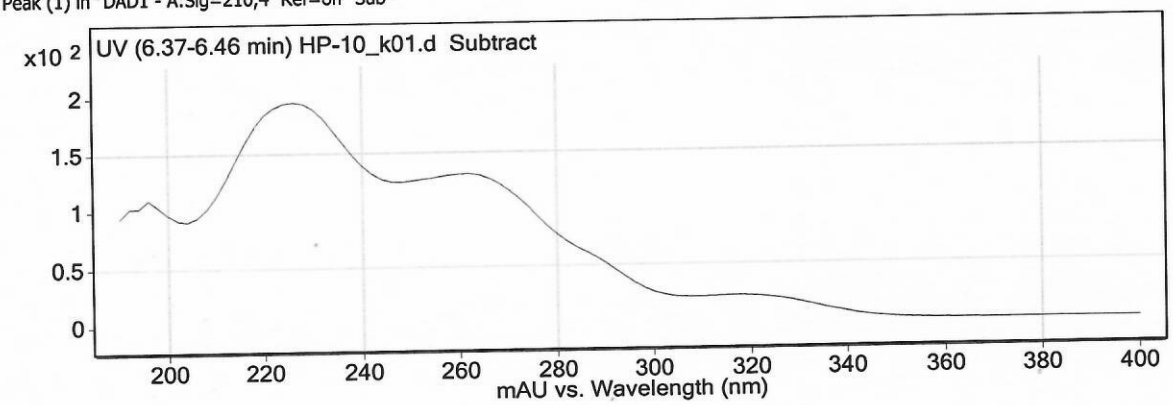
15d



Peak List

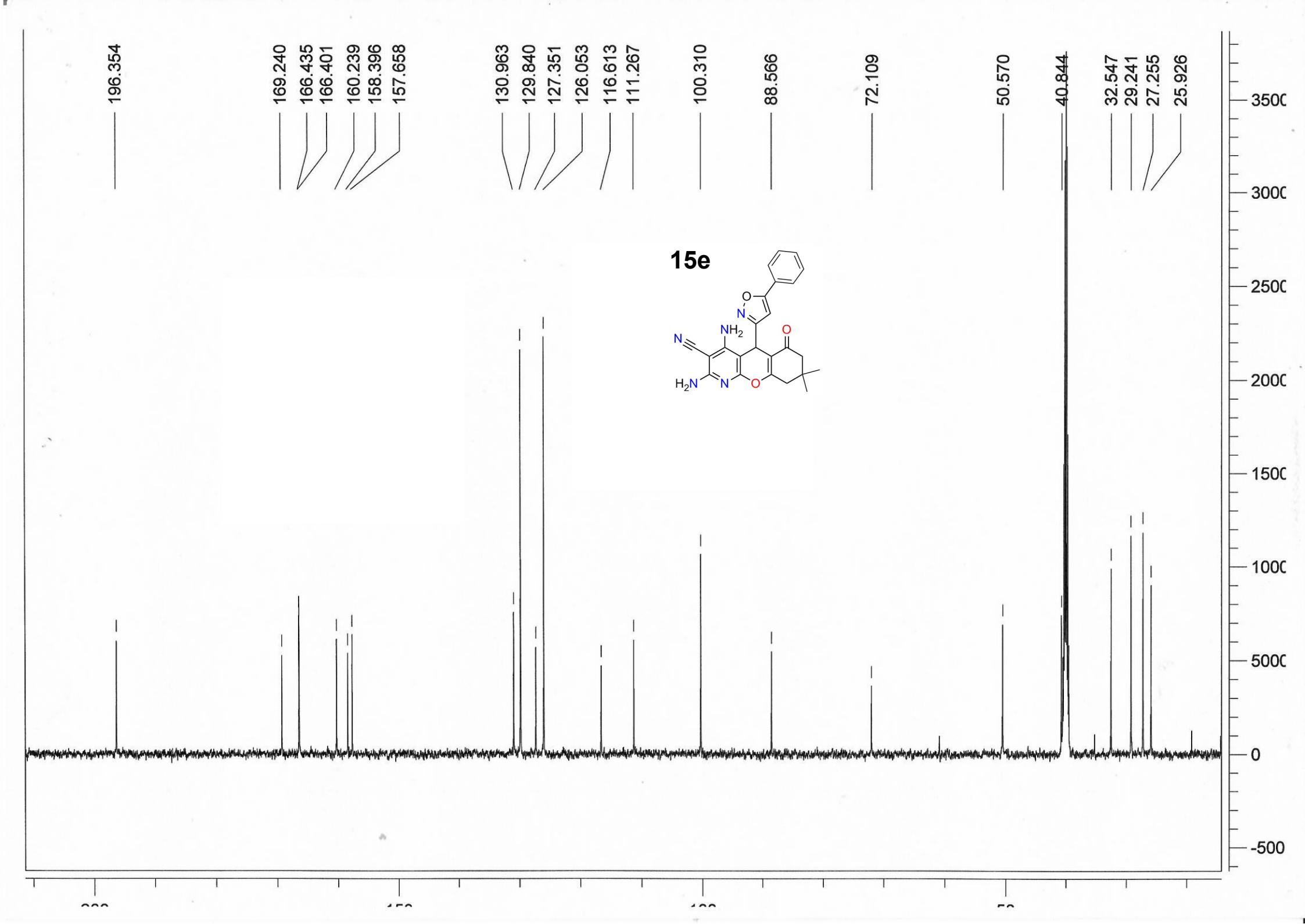
m/z	z	Abund
436.1	1	644394.81
437.1	1	134816.67
438		443152.06
439.1		89733.87
440.1	1	91473.16
458	1	90970.45
893.2		126572.48
895.1		180619.44
896.2		78564.46
897.1	1	110026.41

Spectrum Source
Peak (1) in "DAD1 - A:Sig=210,4 Ref=off Sub"



--- End Of Report ---

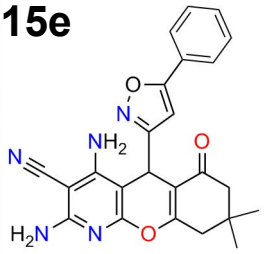
7.796
7.759
7.740
7.525
7.432
6.822
6.685
6.615
5.185
2.615
2.580
2.528
2.493
2.370
2.338
2.209
2.177
1.062
1.010
15e
2.02
3.10
0.99
2.11
1.94
1.00
0.99
2.11
1.04
1.03
6.09


196.354
169.240
166.435
166.401
160.239
158.396
157.658
130.963
129.840
127.351
126.053
116.613
111.267
100.310
88.566
72.109
50.570
40.844
32.547
29.241
27.255
25.926
15e
3500
3000
2500
2000
1500
1000
500
0
-500

Qualitative Analysis Report

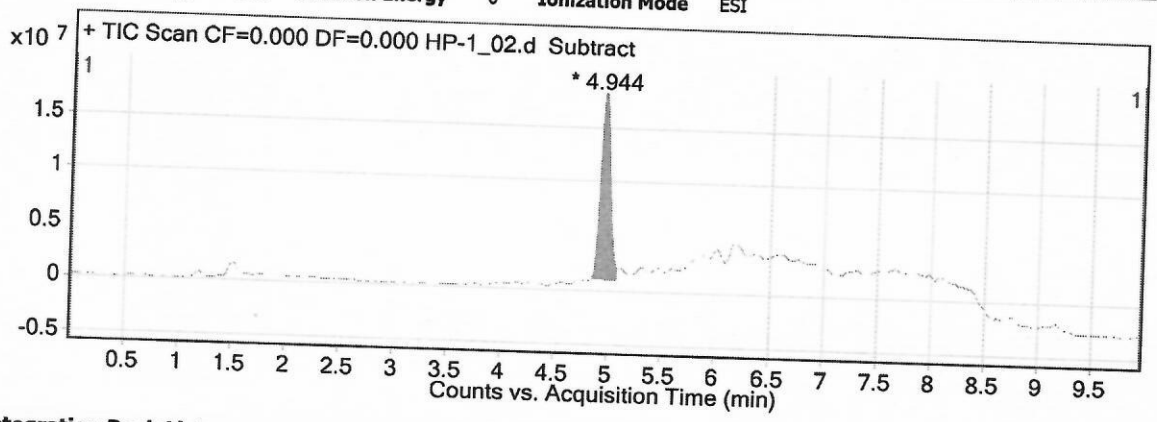
Data Filename	HP-1_02.d	**Sample Name**	HP-1
Sample Type	Sample	**Position**	Vial 2
Instrument Name	Instrument 1	**User Name**	
Acq Method	All_2021_kol 1-2.m	**Acquired Time**	4/13/2023 10:29:49 AM
IRM Calibration Status	Not Applicable	**DA Method**	Defaul1t.m
Comment			
Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition SW Version**	6400 Series Triple Quadrupole 10.0 (127)

15e



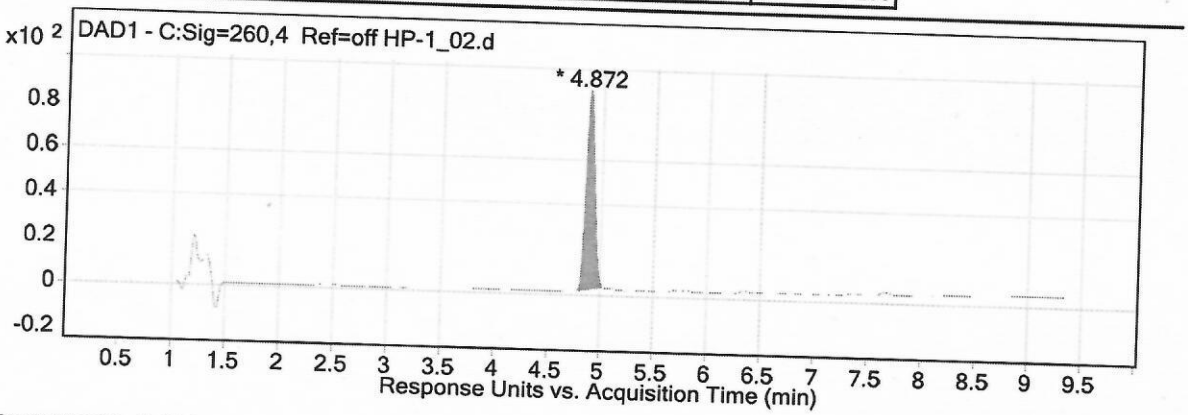
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	4,842	4,944	5,077	17124448,33	109471978	100



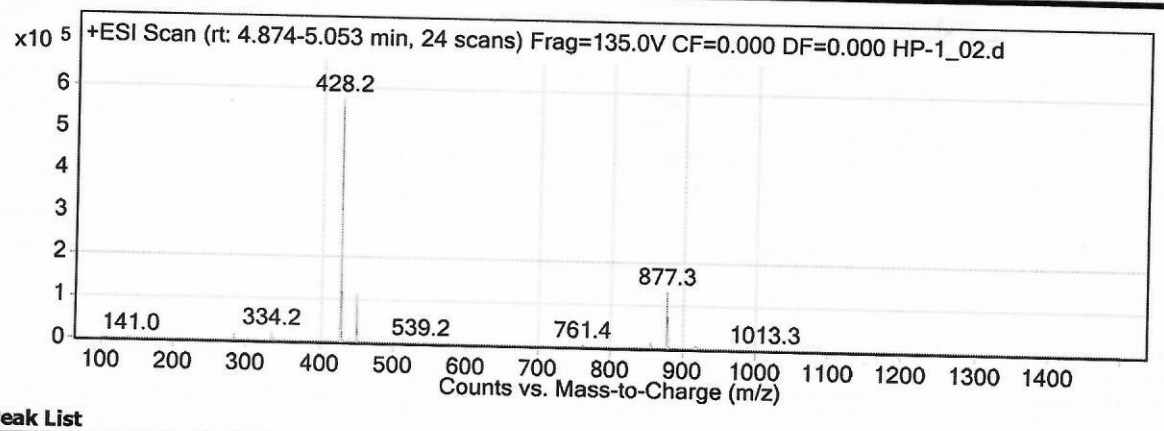
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	4,779	4,872	5,026	88,69	494,54	100

User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan Sub"	135	0	ESI

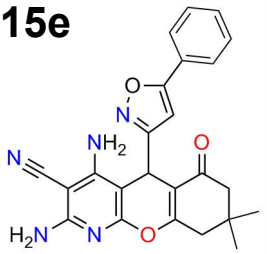
Qualitative Analysis Report



Peak List

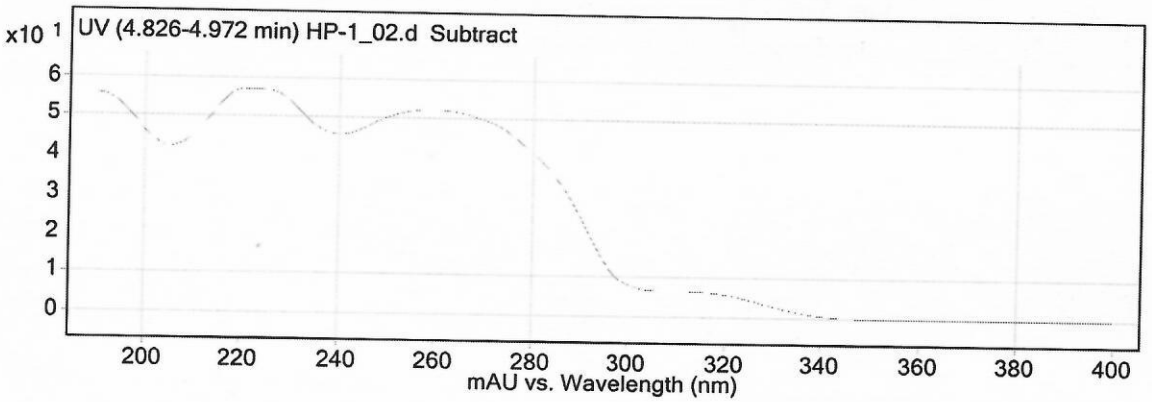
m/z	z	Abund
283.1		15472.87
334.2	1	20856.69
428.2	1	577019.94
429.2	1	158924.31
430.2	1	24846.27
450.1	1	114902.99
451.1	1	33331.02
877.3		134735.14
878.4	1	75853.62
879.4	1	21416.2

15e

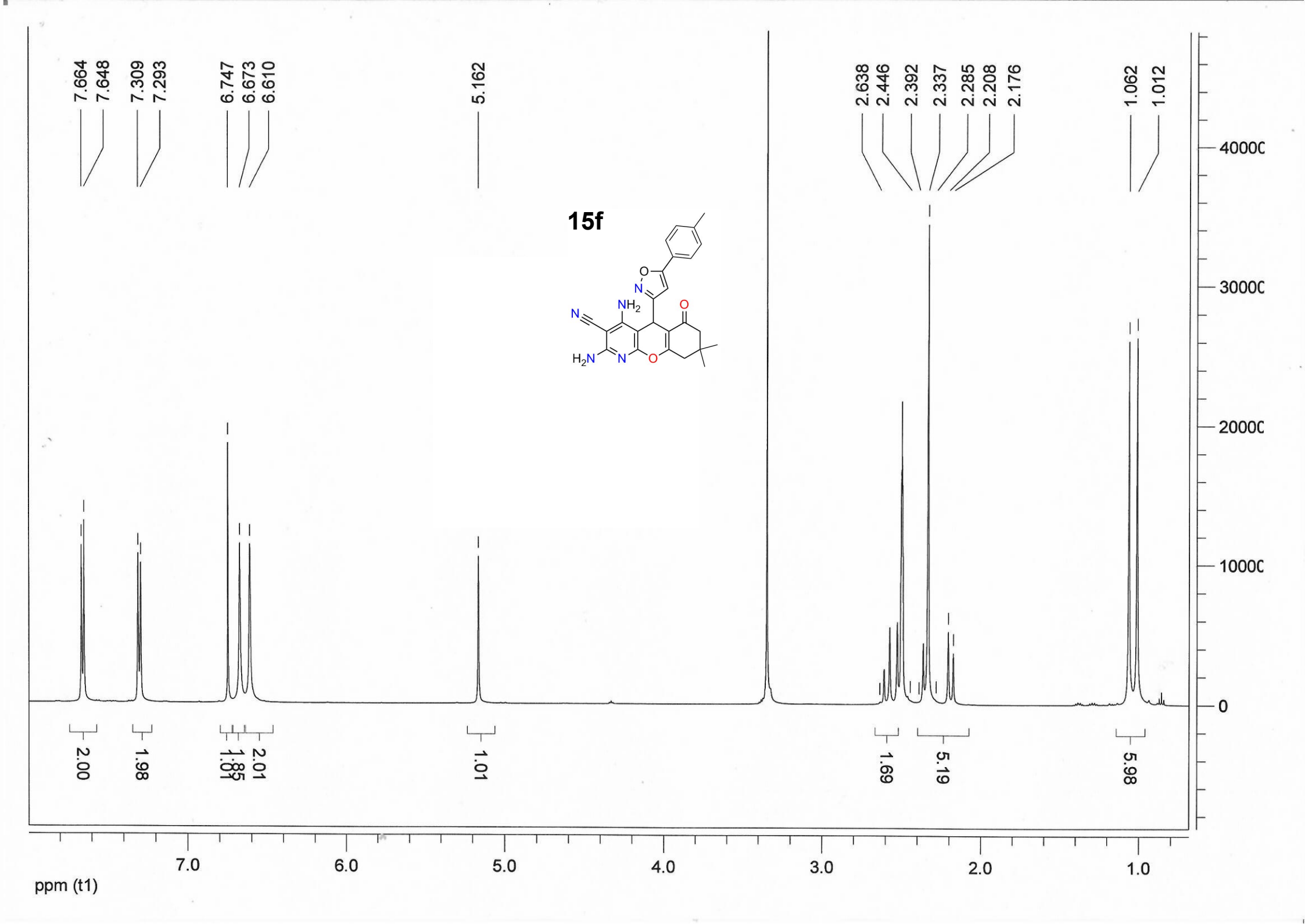


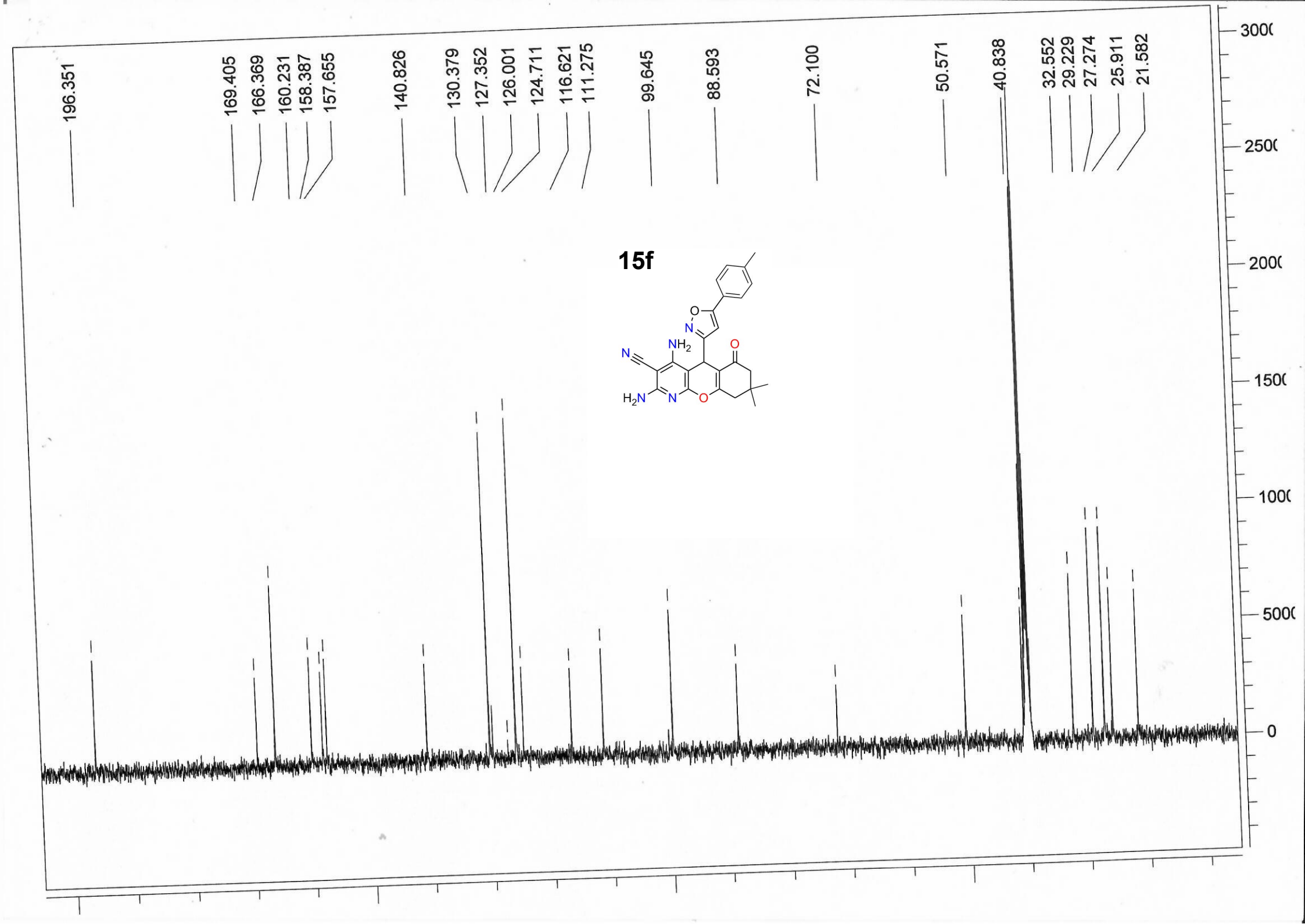
Spectrum Source

Peak (1) in "DAD1 - C:Sig=260,4 Ref=off"



--- End Of Report ---

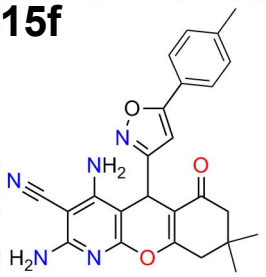

7.664
7.648
7.309
7.293
6.747
6.673
6.610
5.162
2.638
2.446
2.392
2.337
2.285
2.208
2.176
1.062
1.012
15f
NH2
O
N
N
H2N
N
O
O
4000
3000
2000
1000
0
2.00
1.98
1.01
1.85
2.01
1.01
1.69
5.19
5.98
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)


196.351
169.405
166.369
160.231
158.387
157.655
140.826
130.379
127.352
126.001
124.711
116.621
111.275
99.645
88.593
72.100
50.571
40.838
32.552
29.229
27.274
25.911
21.582
15f
3000
2500
2000
1500
1000
500
0

Qualitative Analysis Report

Data Filename	HP-11_k01.d	**Sample Name**	HP-11
Sample Type	Sample	**Position**	Vial 2
Instrument Name	Instrument 1	**User Name**	
Acq Method	All_2021_kol 1-6.m	**Acquired Time**	2/19/2024 12:02:29 PM
IRM Calibration Status	Not Applicable	**DA Method**	Defaul1t.m
Comment			

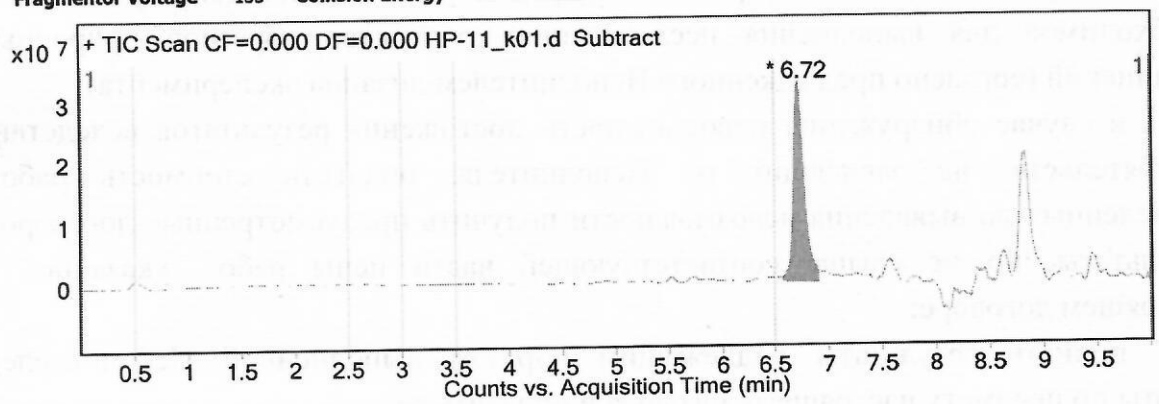
15f



Sample Group		**Info.**	
Stream Name	LC 1	**Acquisition SW Version**	6400 Series Triple Quadrupole 10.0 (127)

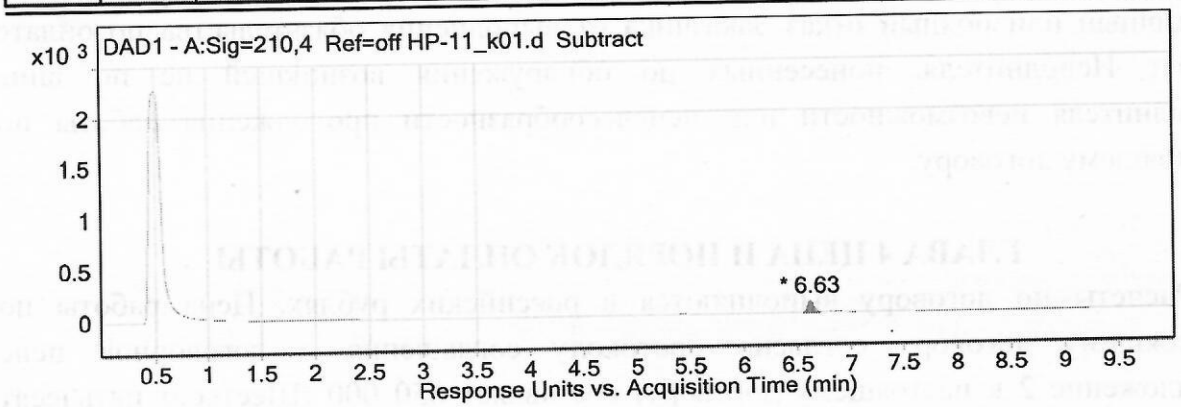
User Chromatograms

Fragmentor Voltage 135 **Collision Energy** 0 **Ionization Mode** ESI



Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	6,57	6,72	6,9	33035628,01	228452506,5	100



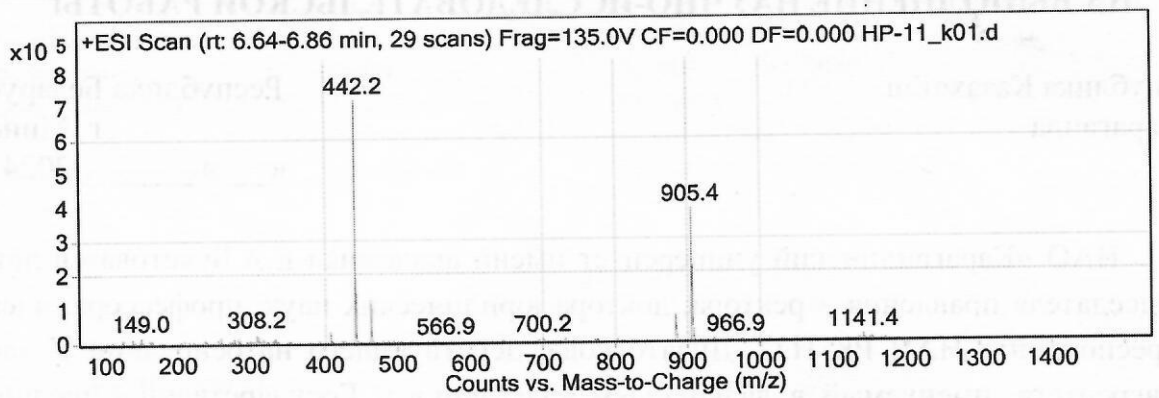
Integration Peak List

Peak	Start	RT	End	Height	Area	Area %
1	6,56	6,63	6,72	124,21	478,86	100

User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
Peak (1) in "+ TIC Scan Sub"	135	0	ESI

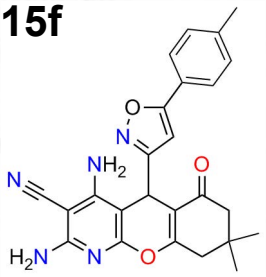
Qualitative Analysis Report



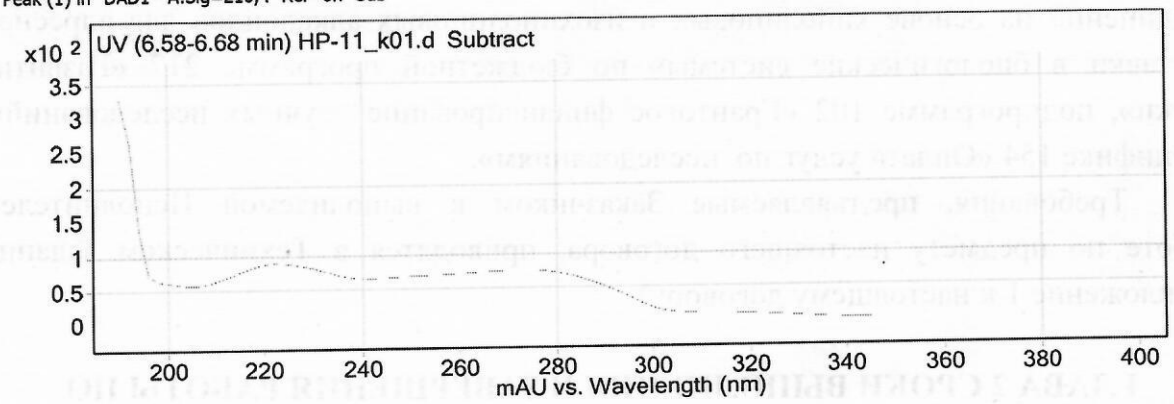
Peak List

m/z	z	Abund
442.2	1	718932.75
443.2	1	191088.22
444.2	1	30020.13
464.2		130716.3
465.1		36964.34
883.4	1	77452.52
884.4	1	44096.52
905.4	1	389273.06
906.4	1	221593.94
907.4	1	68230.75

15f



Spectrum Source
Peak (1) in "DAD1 - A:Sig=210,4 Ref=off Sub"



--- End Of Report ---