WYDZIAŁ CHEMICZNY
KATEDRA CHEMII ORGANICZNEJ, BIOORGANICZNEJ
I BIOTECHNOLOGII

mgr inż. Olga Drosik

ROZPRAWA DOKTORSKA

Badanie reakcji α,β-nienasyconych związków karbonylowych z reagentami binukleofilowymi

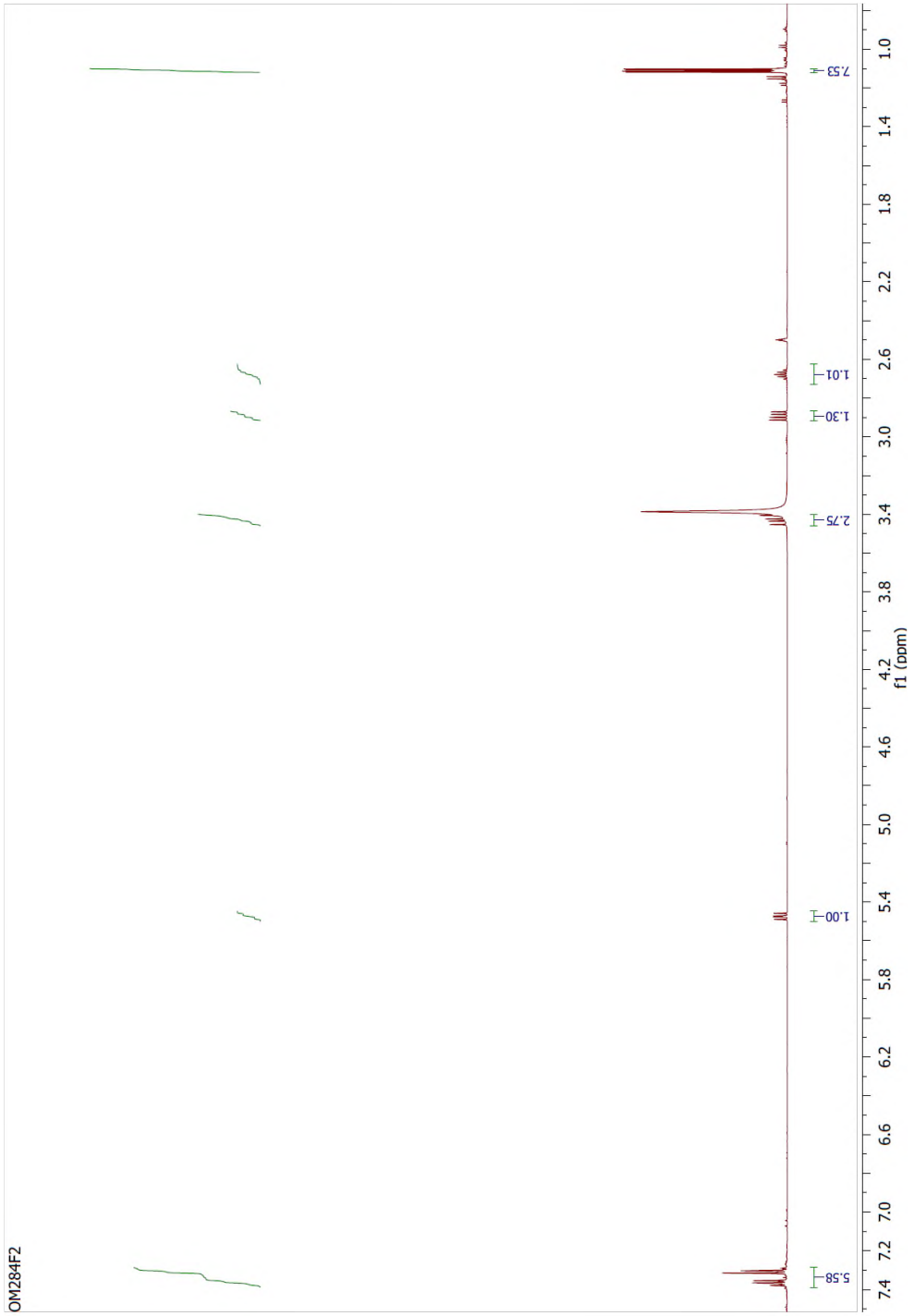
Promotor: dr hab. inż. Wojciech Szczepankiewicz

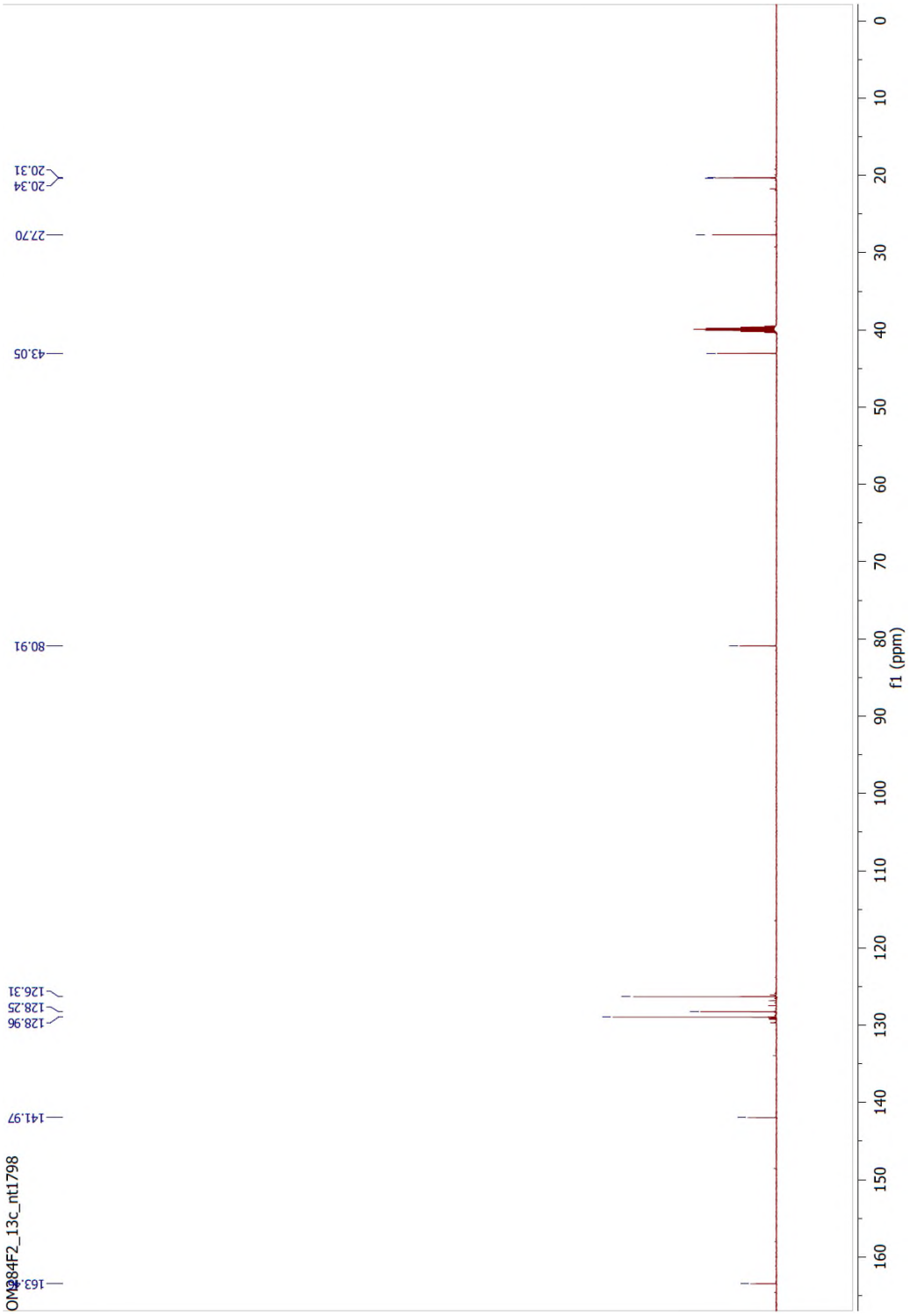
Gliwice, 2023

Załączniki

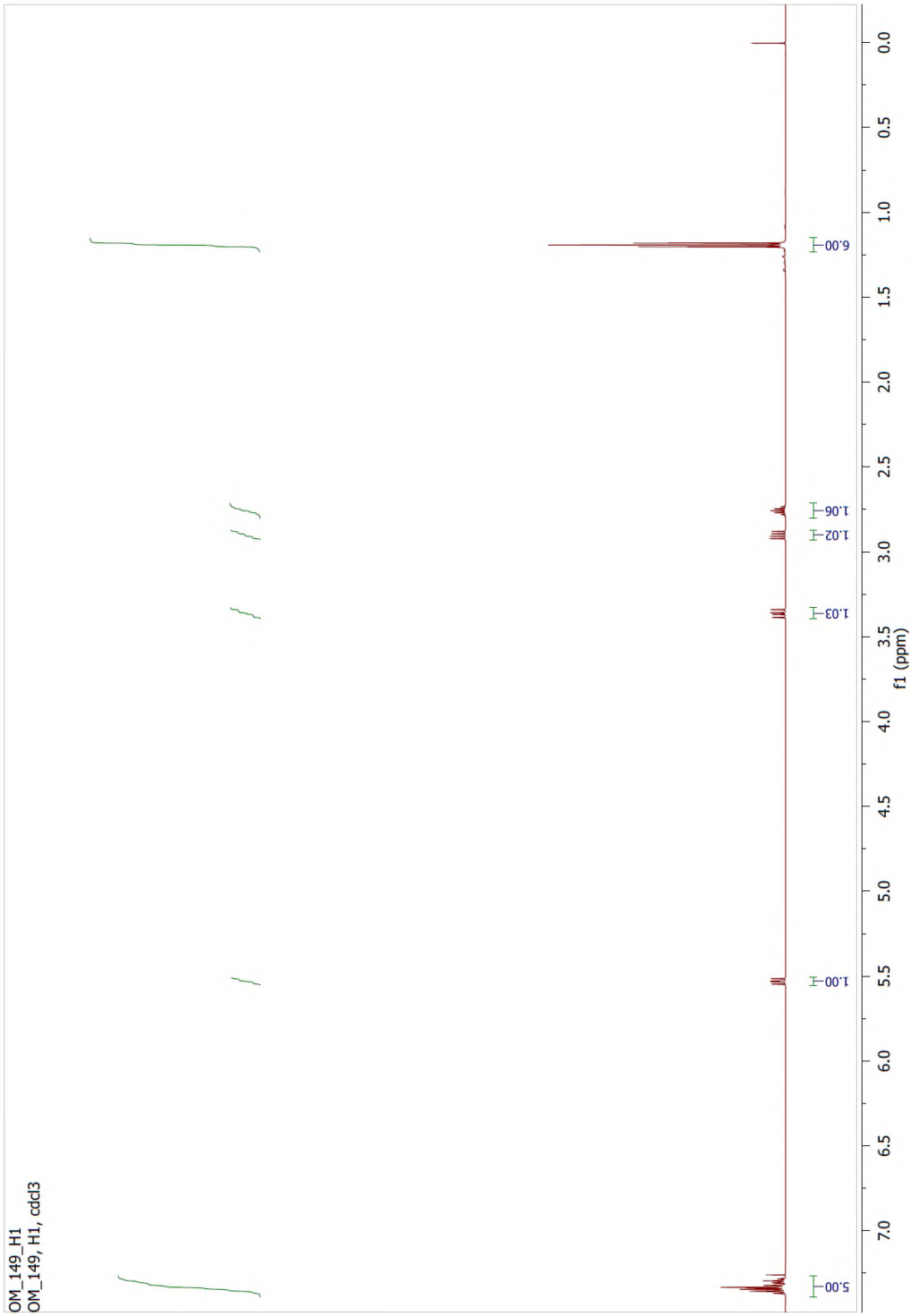
Wybrane najważniejsze widma NMR

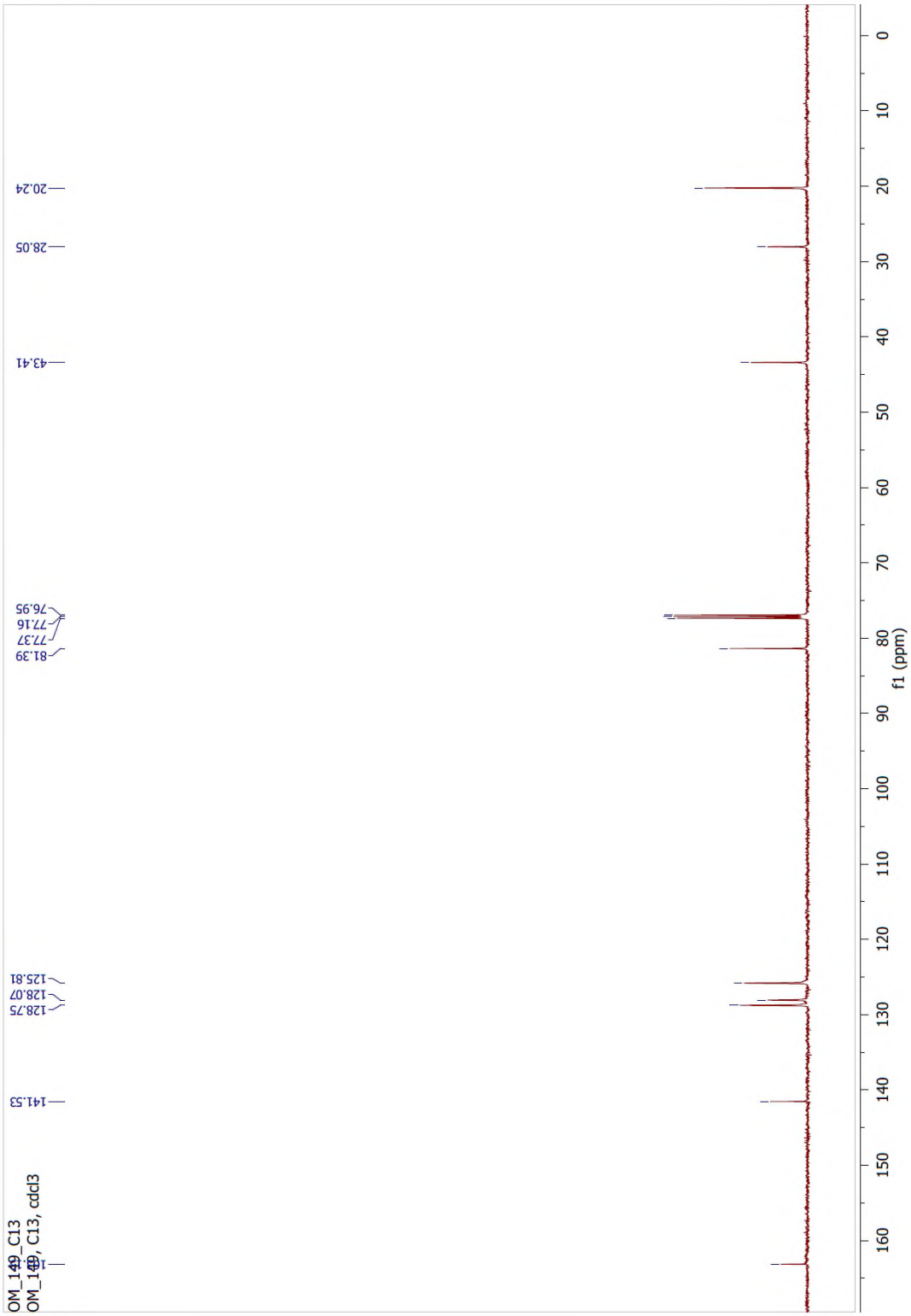
(3b) 3-izopropylo-5-fenylo-2-izoksazolina – metoda A:




20.31
20.34
27.70
43.05
80.91
126.31
128.25
128.96
141.97
163.27
OM384F2_13c_nt1798
f1 (ppm)

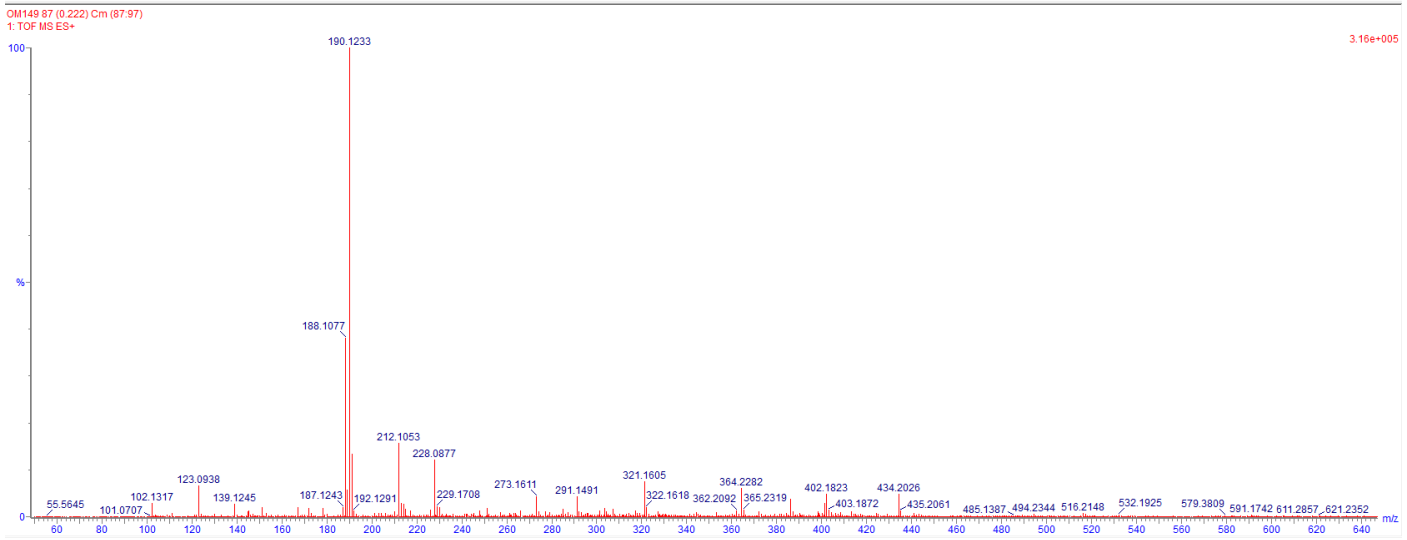
Metoda B:



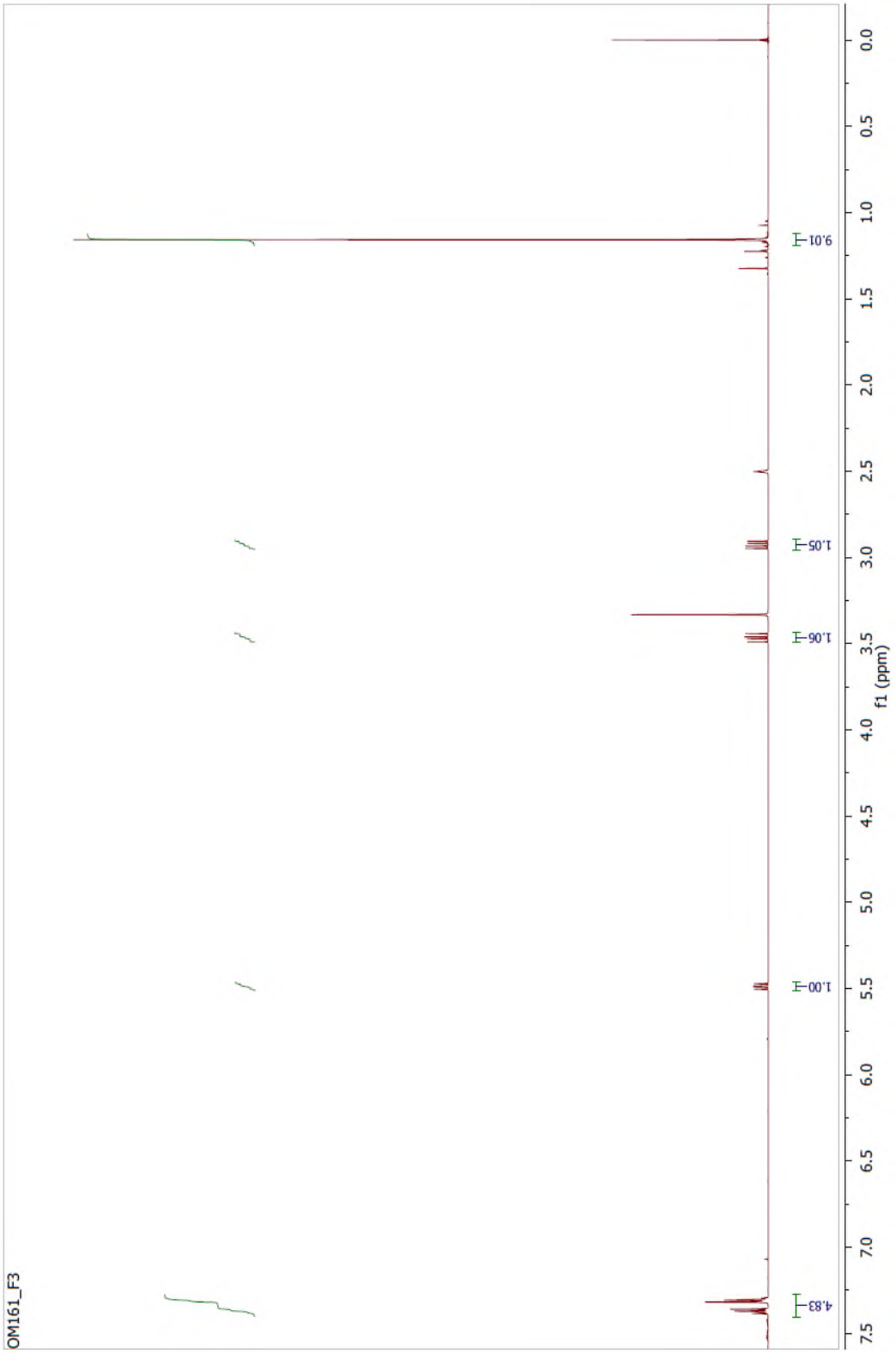

OM_149_C13
OM_149, C13, cdcl3
20.24
28.05
43.41
76.95
77.16
77.37
81.39
125.81
128.07
128.75
141.53
f1 (ppm)

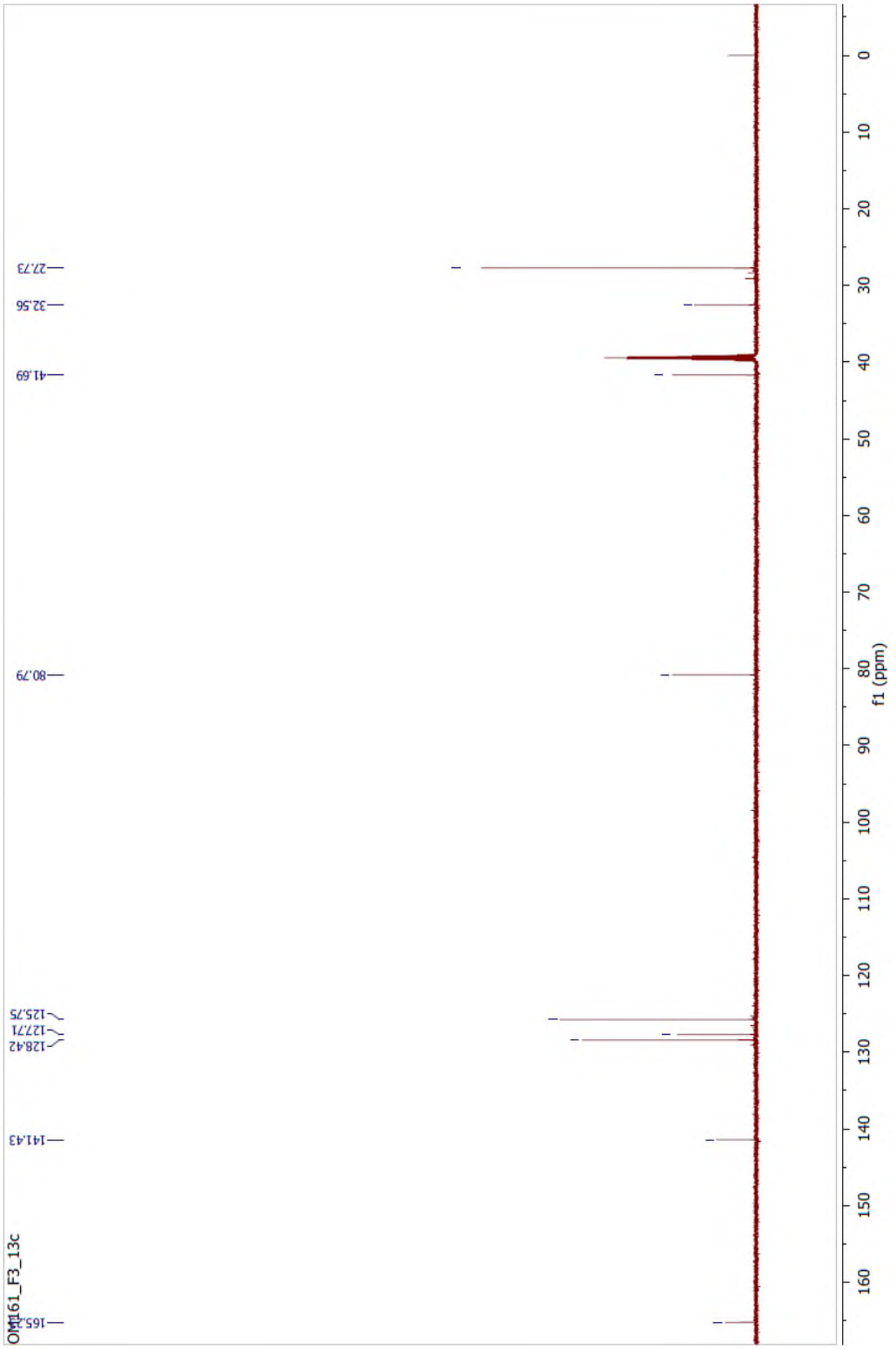
Tolerance = 100.0 mDa / DBE: min = -100.0, max = 200.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
18 formula(e) evaluated with 6 results within limits (up to 3 closest results for each mass)
Elements Used:

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	N	O	23Na
190.1233	100.00	190.1232	0.1	0.5	5.5	C12 H16 N O	328.6	0.002	99.83	12	16	1	1	
		190.1208	2.5	13.1	2.5	C10 H17 N O 23Na	335.0	6.391	0.17	10	17	1	1	1
		190.2147	-91.4	-480.7	-4.5	C9 H29 N O 23Na	341.2	12.609	0.00	9	29	1	1	1



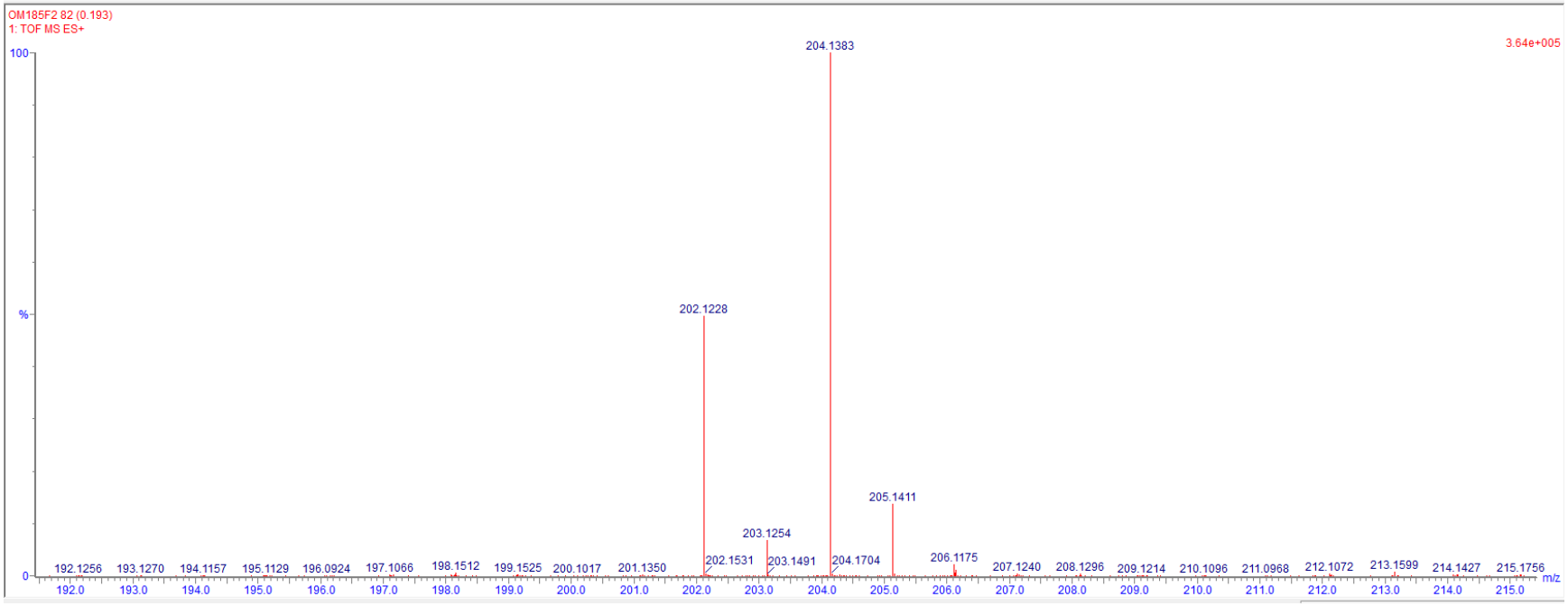
(3c) 3-t-butylo-5-fenylo-2-izoksazolina - Metoda A:



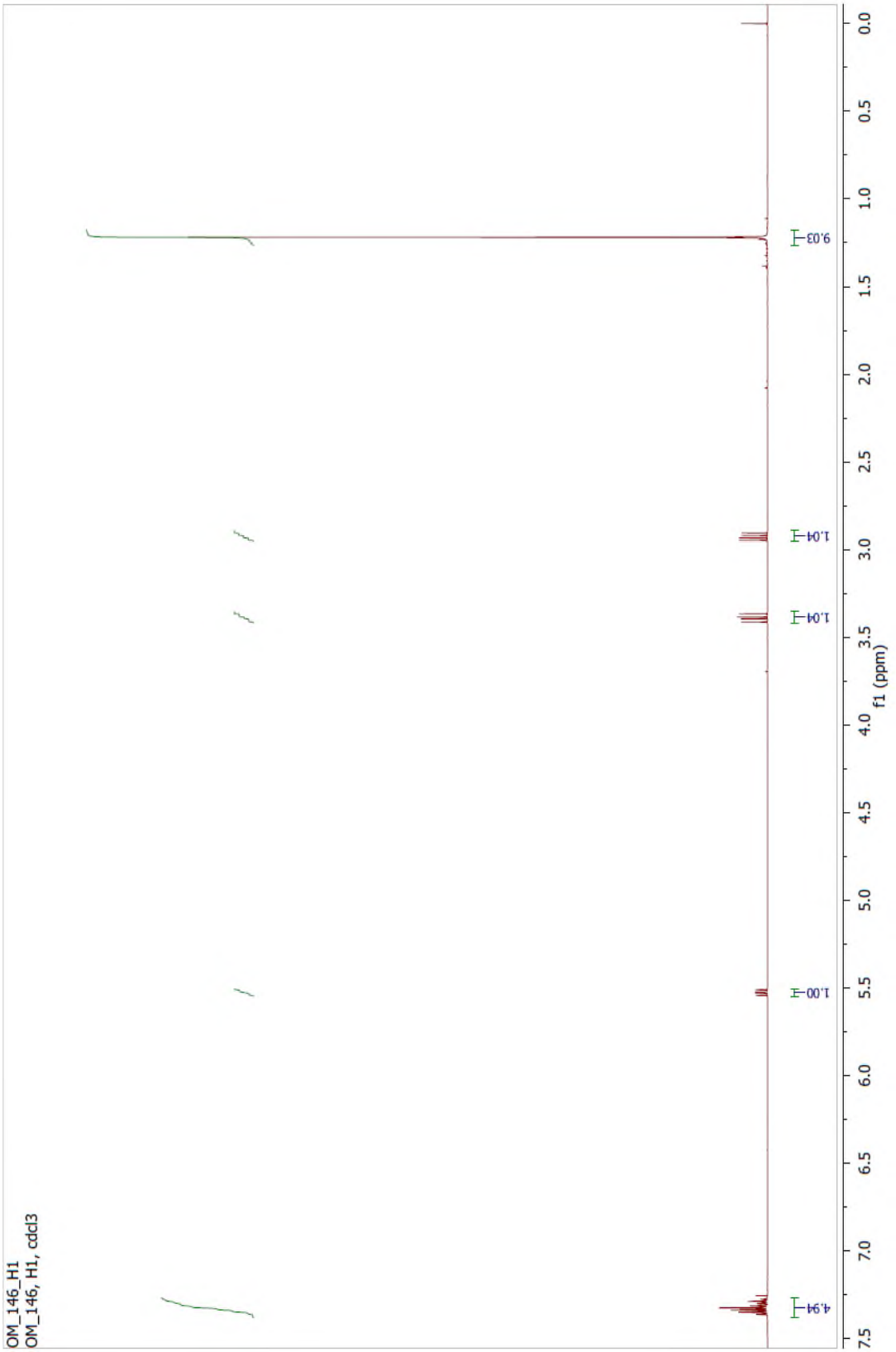

OM161_F3_13c
165,14
141,43
128,42
127,71
125,75
80,79
41,69
32,56
27,73
f1 (ppm)

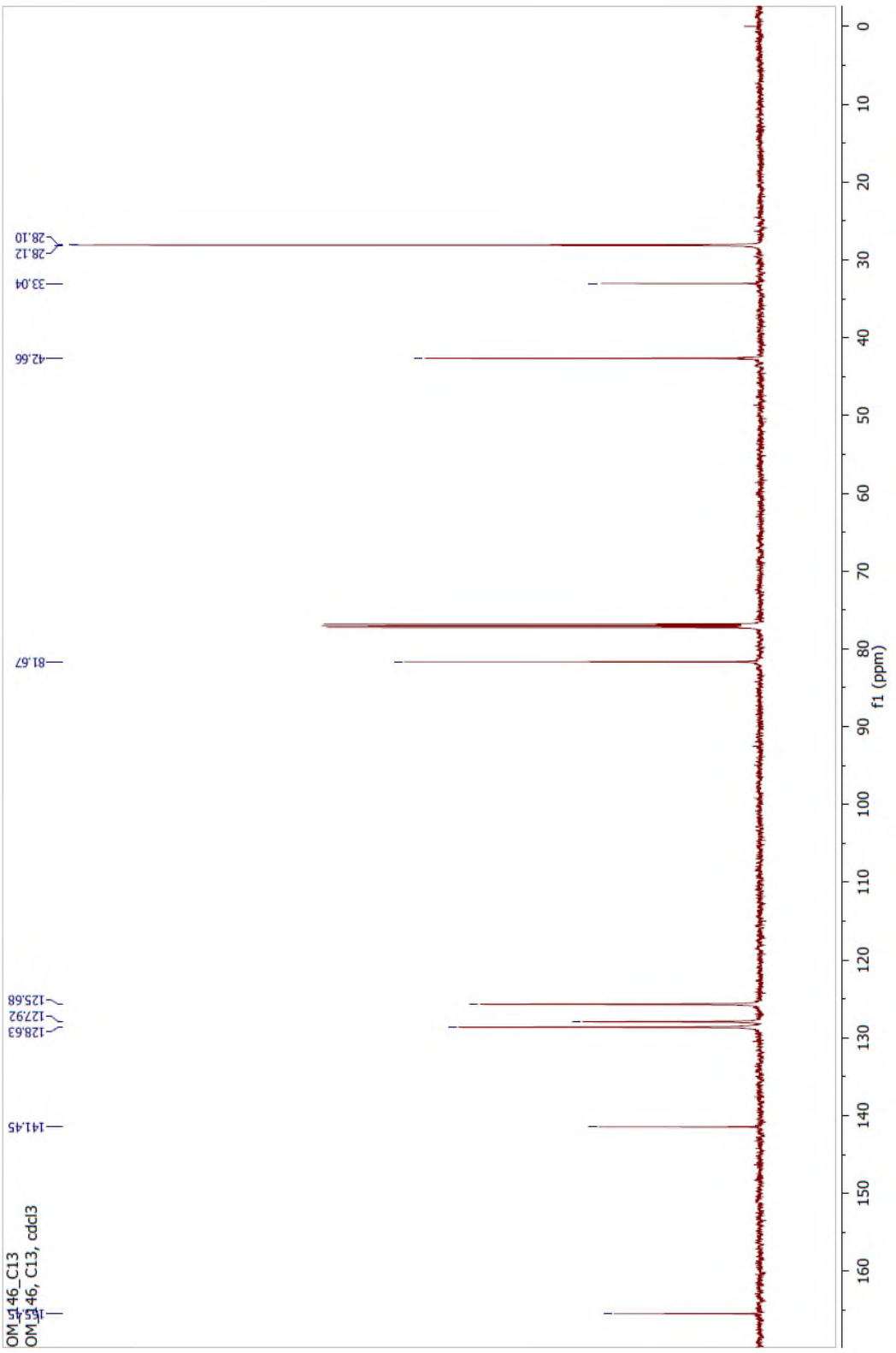
Multiple Mass Analysis: 2 mass(es) processed
Tolerance = 100.0 mDa / DBE: min = -100.0, max = 200.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
34 formula(e) evaluated with 12 results within limits (up to 3 closest results for each mass)
Elements Used:

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	N	O	23Na
202.1228	49.73	202.1232	-0.4	-2.0	6.5	C13 H16 N O	464.9	0.972	37.84	13	16	1	1	
		202.1208	2.0	9.9	3.5	C11 H17 N O 23Na	465.0	1.032	35.65	11	17	1	1	1
		202.2147	-91.9	-454.7	-3.5	C10 H29 N O 23Na	465.3	1.327	26.52	10	29	1	1	1
204.1383	100.00	204.1388	-0.5	-2.4	5.5	C13 H18 N O	417.5	0.263	76.90	13	18	1	1	
		204.1364	1.9	9.3	2.5	C11 H19 N O 23Na	418.7	1.483	22.71	11	19	1	1	1
		204.2303	-92.0	-450.7	-4.5	C10 H31 N O 23Na	422.8	5.548	0.39	10	31	1	1	1



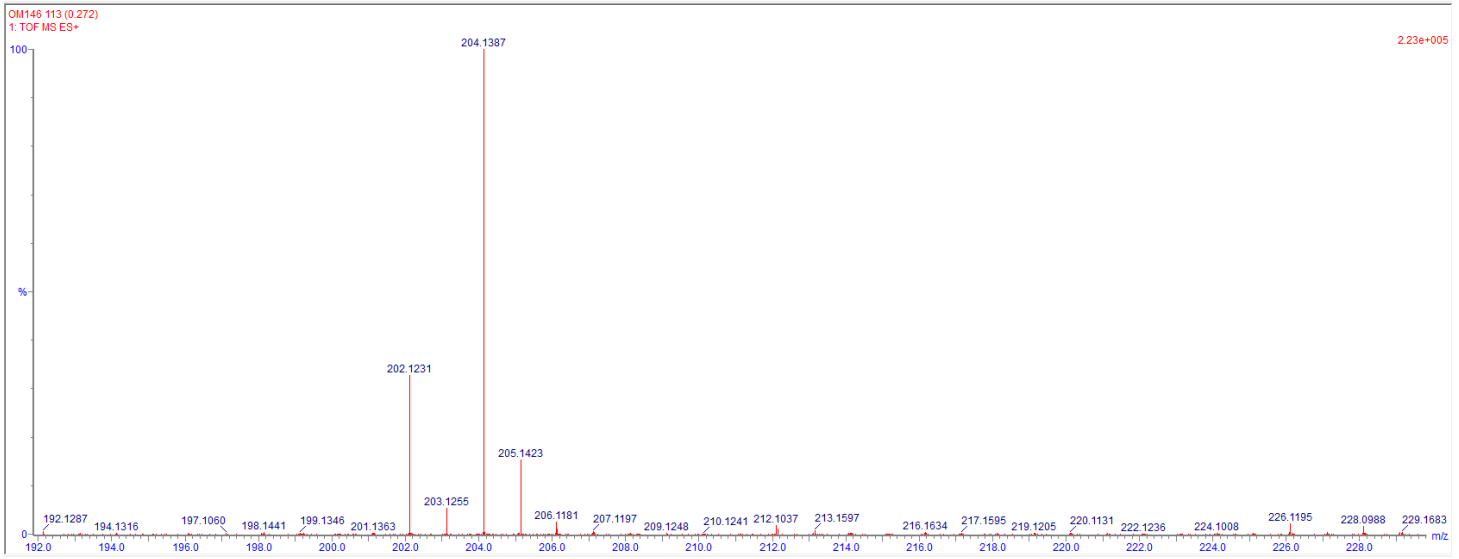
Metoda B:



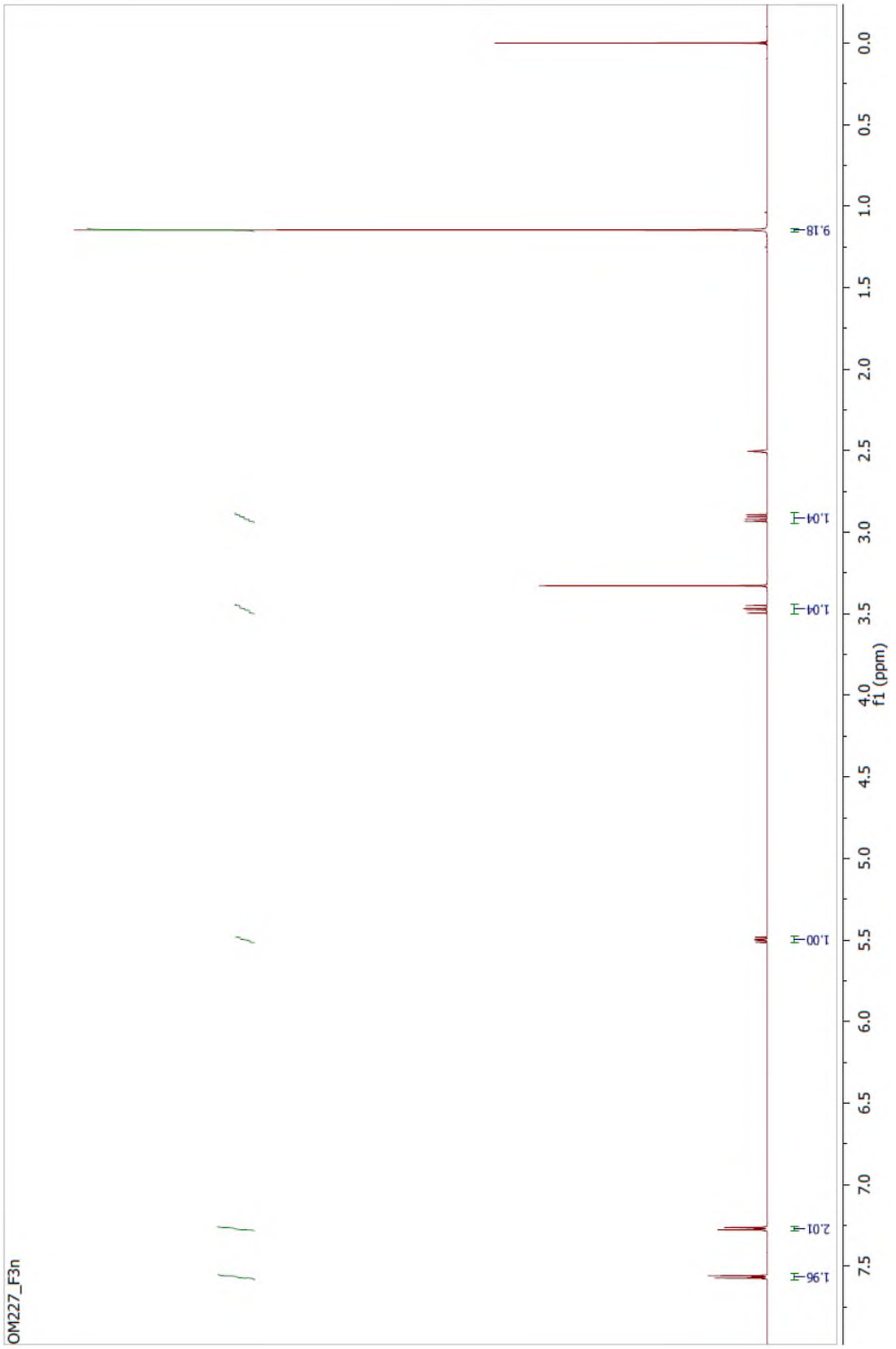

OM_5146_C13
OM_5146, C13, cdcl3
141.45
128.63
127.92
125.68
81.67
42.66
33.04
28.12
28.10
f1 (ppm)

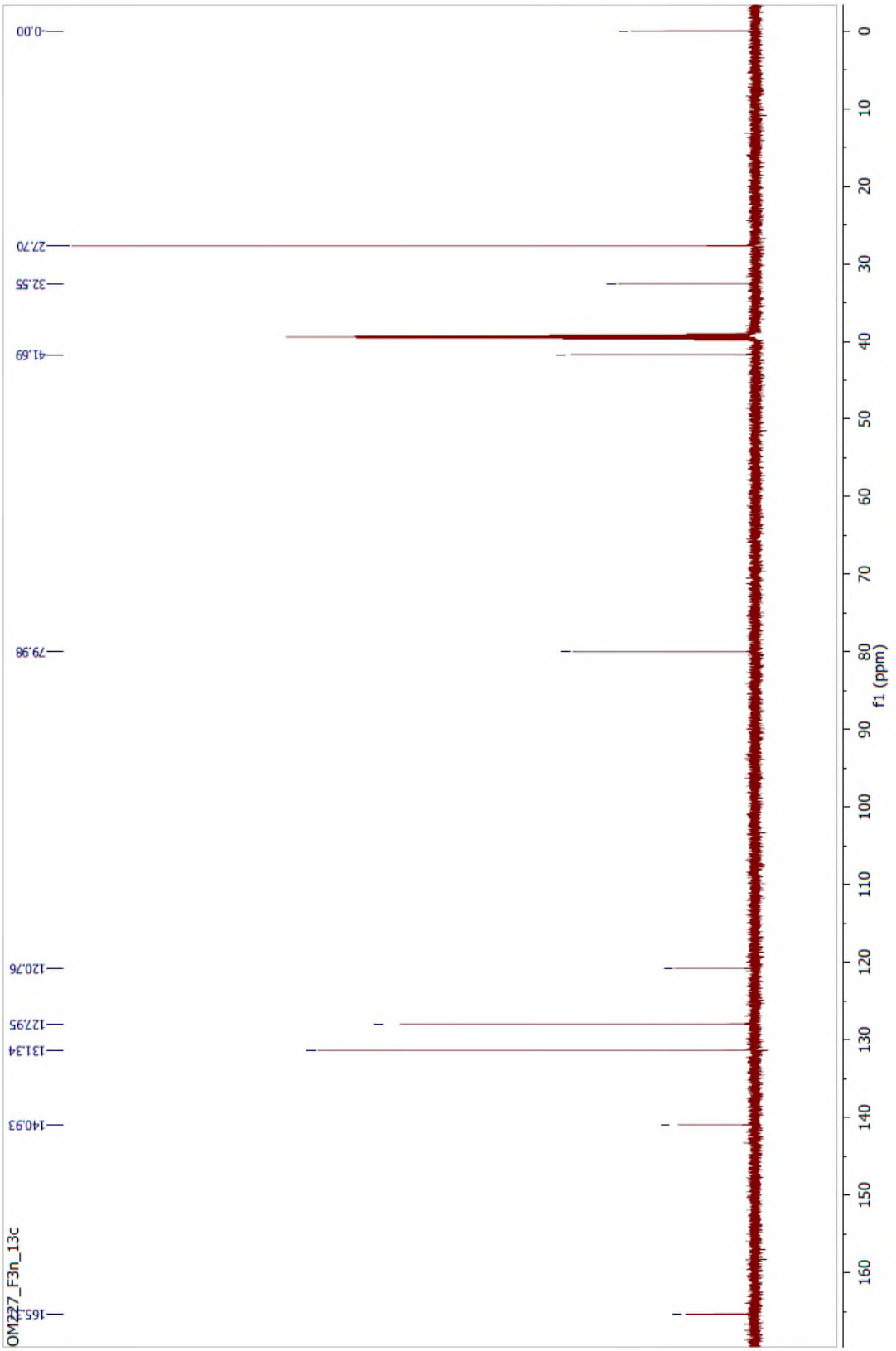
Tolerance = 100.0 mDa / DBE: min = -100.0, max = 200.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
16 formula(e) evaluated with 6 results within limits (up to 3 closest results for each mass)
Elements Used:

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	N	O	23Na
204.1387	100.00	204.1388	-0.1	-0.5	5.5	C13 H18 N O	398.1	0.040	96.10	13	18	1	1	
		204.1364	2.3	11.3	2.5	C11 H19 N O 23Na	401.8	3.705	2.46	11	19	1	1	1
		204.2303	-91.6	-448.7	-4.5	C10 H31 N O 23Na	402.3	4.237	1.45	10	31	1	1	1



(3d) 3-t-butylo-5-(4-bromofenylo)-2-izoksazolina - Metoda A:




0.00
27.70
32.55
41.69
79.98
120.76
127.95
131.34
140.93
165.27
OM227_F3n_13c
f1 (ppm)

Multiple Mass Analysis: 2 mass(es) processed

Tolerance = 100.0 mDa / DBE: min = -100.0, max = 200.0

Element prediction: Off

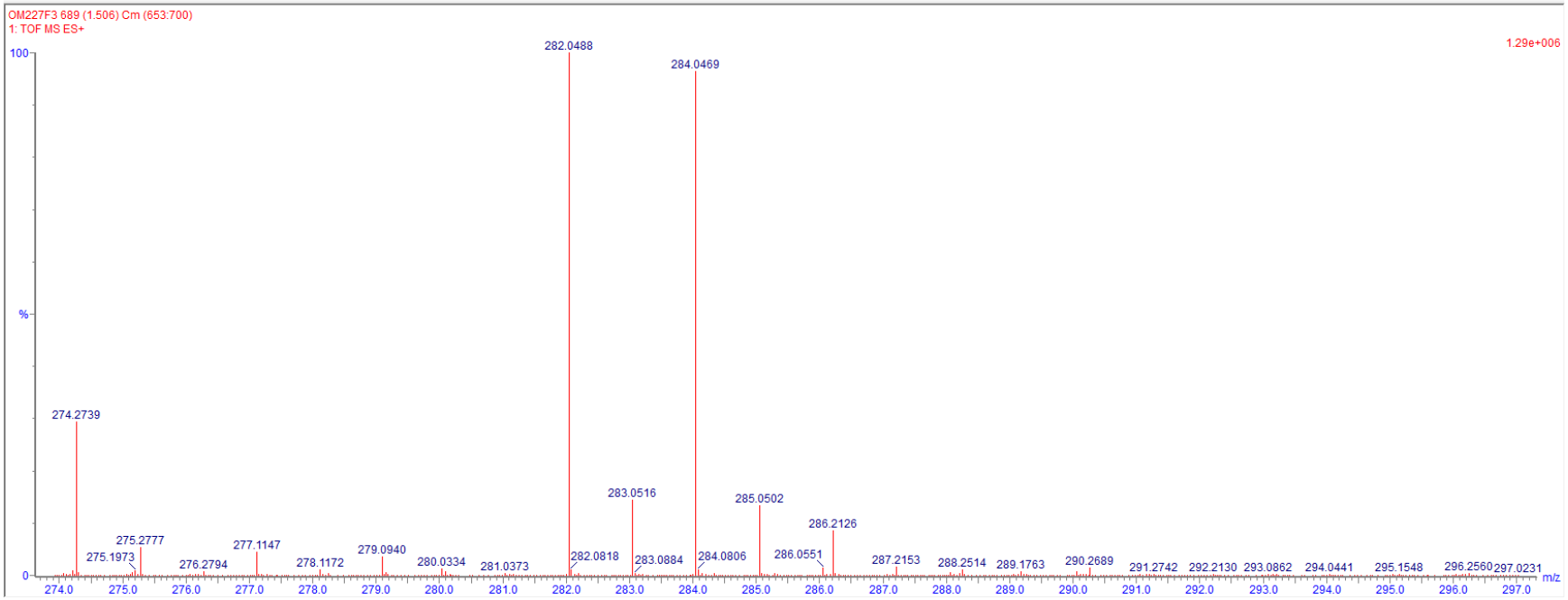
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

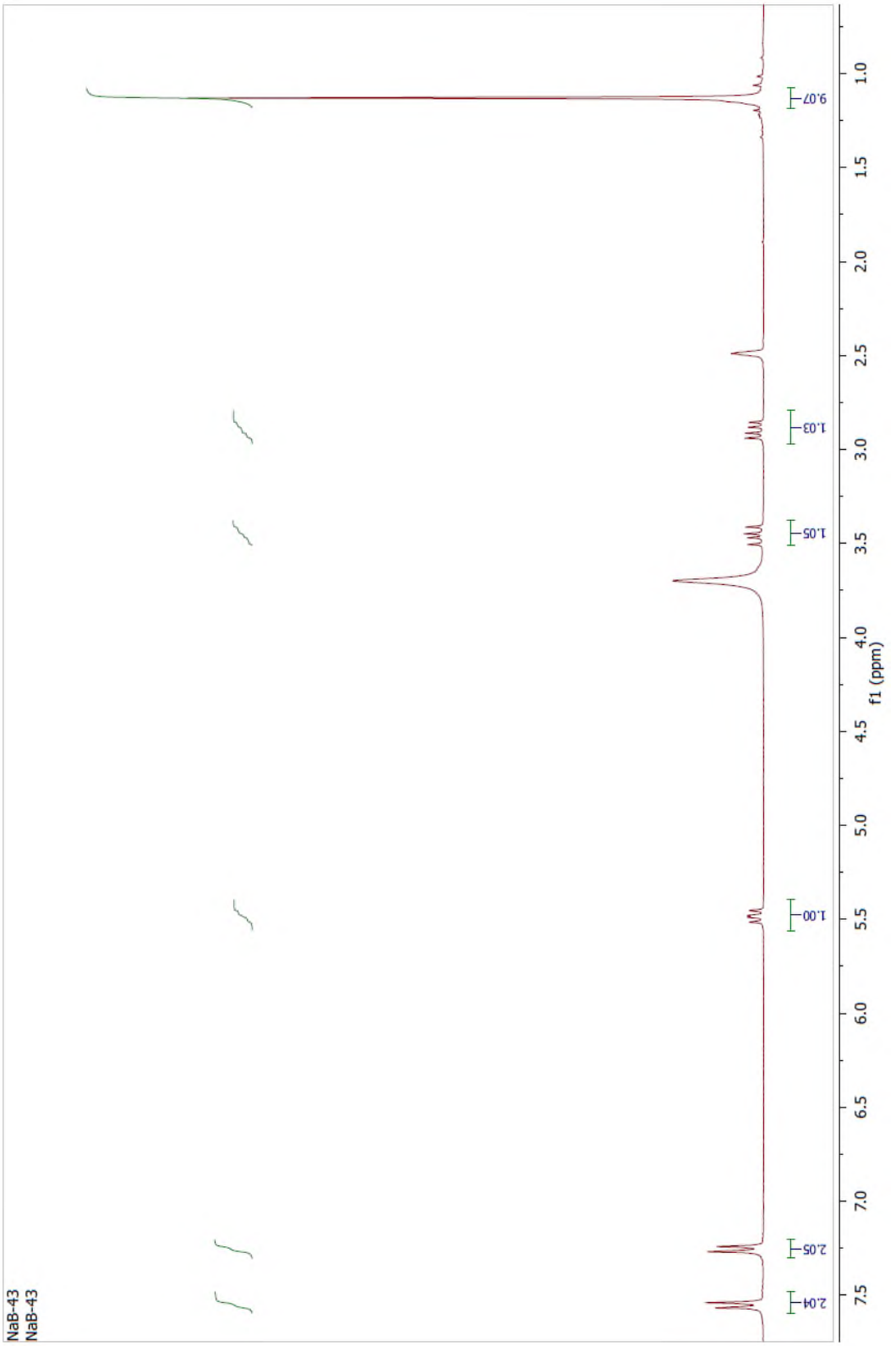
68 formula(e) evaluated with 10 results within limits (up to 3 closest results for each mass)

Elements Used:

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	N	O	23Na	Br
282.0488	100.00	282.0494	-0.6	-2.1	5.5	C13 H17 N O Br	688.7	0.008	99.21	13	17	1	1		1
		282.0469	1.9	6.7	2.5	C11 H18 N O 23Na Br	693.5	4.845	0.79	11	18	1	1	1	1
		282.1408	-92.0	-326.2	-4.5	C10 H30 N O 23Na Br	698.2	9.571	0.01	10	30	1	1	1	1
284.0469	96.43	284.0626	-15.7	-55.3	1.5	C11 H20 N O 23Na Br	695.5	1.175	30.88	11	20	1	1	1	1
		284.0650	-18.1	-63.7	4.5	C13 H19 N O Br	695.5	1.170	31.04	13	19	1	1		1
		283.9711	75.8	266.9	11.5	C14 H7 N O Br	695.2	0.966	38.08	14	7	1	1		1

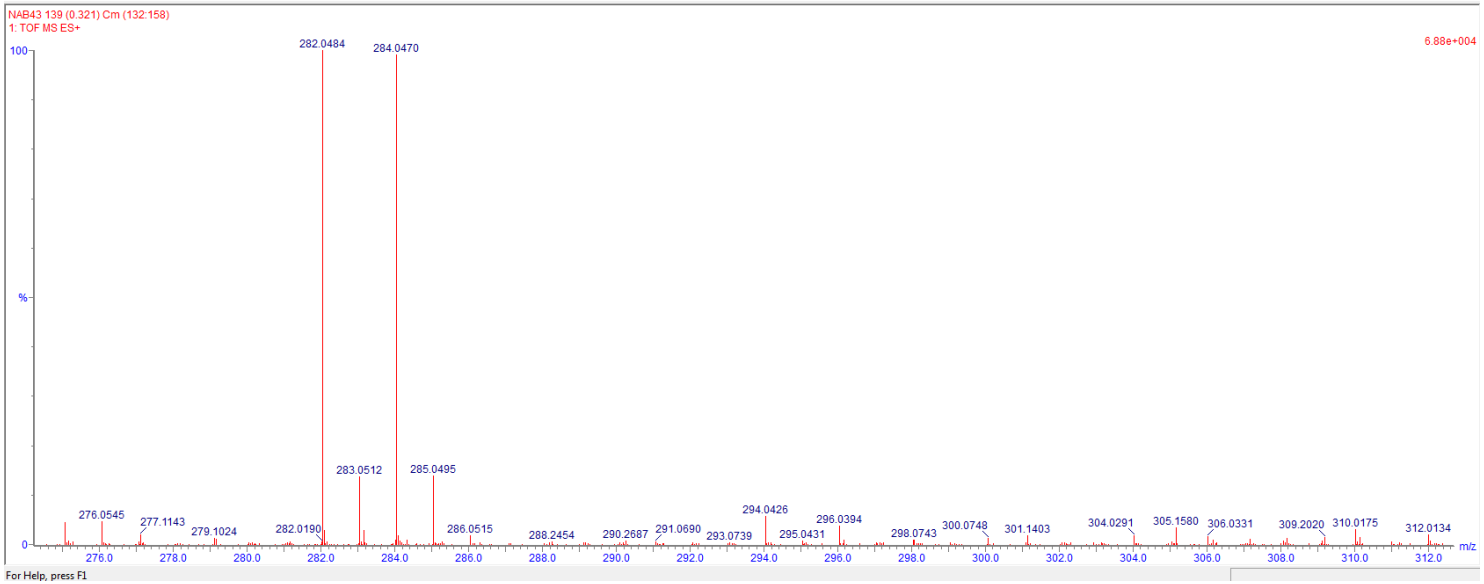


Metoda B:

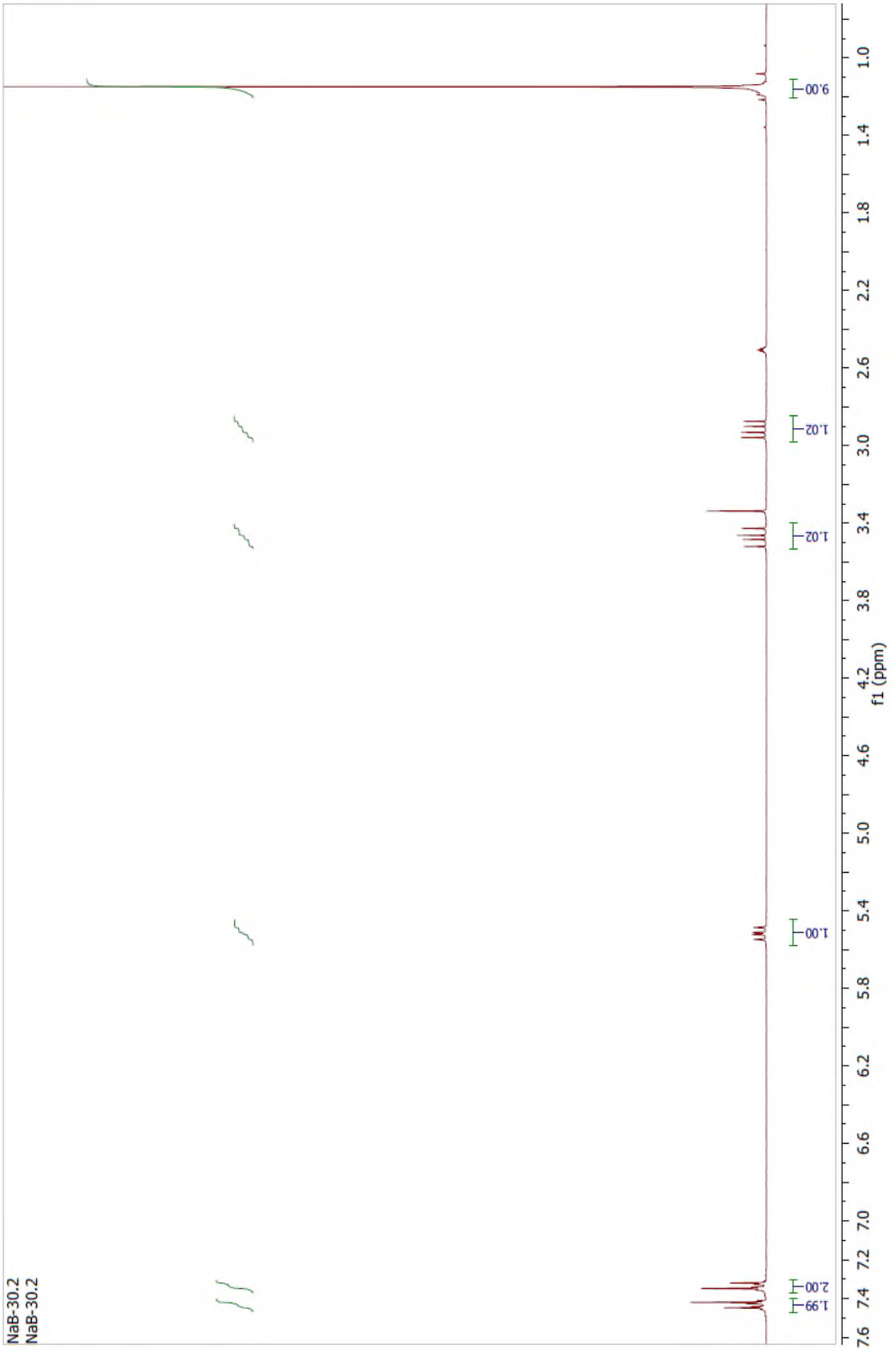


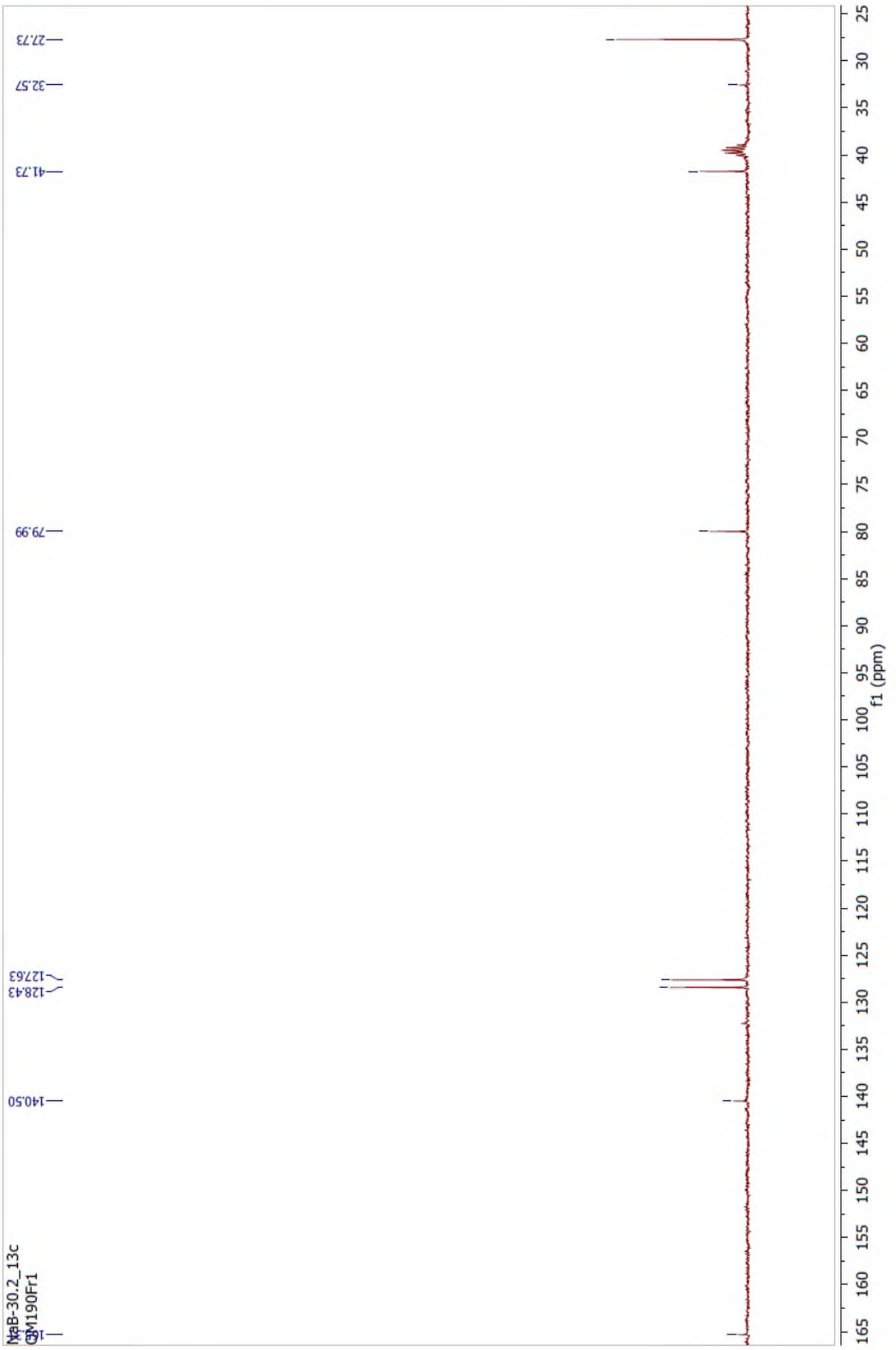
Multiple Mass Analysis: 2 mass(es) processed
Tolerance = 100.0 mDa / DBE: min = -100.0, max = 200.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
68 formula(e) evaluated with 10 results within limits (up to 3 closest results for each mass)
Elements Used:

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	N	O	23Na	Br
282.0484	100.00	282.0494	-1.0	-3.5	5.5	C13 H17 N O Br	288.3	0.443	64.19	13	17	1	1		1
		282.0469	1.5	5.3	2.5	C11 H18 N O 23Na Br	288.9	1.035	35.53	11	18	1	1	1	1
		282.1408	-92.4	-327.6	-4.5	C10 H30 N O 23Na Br	293.7	5.863	0.28	10	30	1	1	1	1
284.0470	99.19	284.0626	-15.6	-54.9	1.5	C11 H20 N O 23Na Br	265.0	2.230	10.75	11	20	1	1	1	1
		284.0650	-18.0	-63.4	4.5	C13 H19 N O Br	263.0	0.254	77.56	13	19	1	1		1
		283.9711	75.9	267.2	11.5	C14 H7 N O Br	264.9	2.146	11.69	14	7	1	1		1



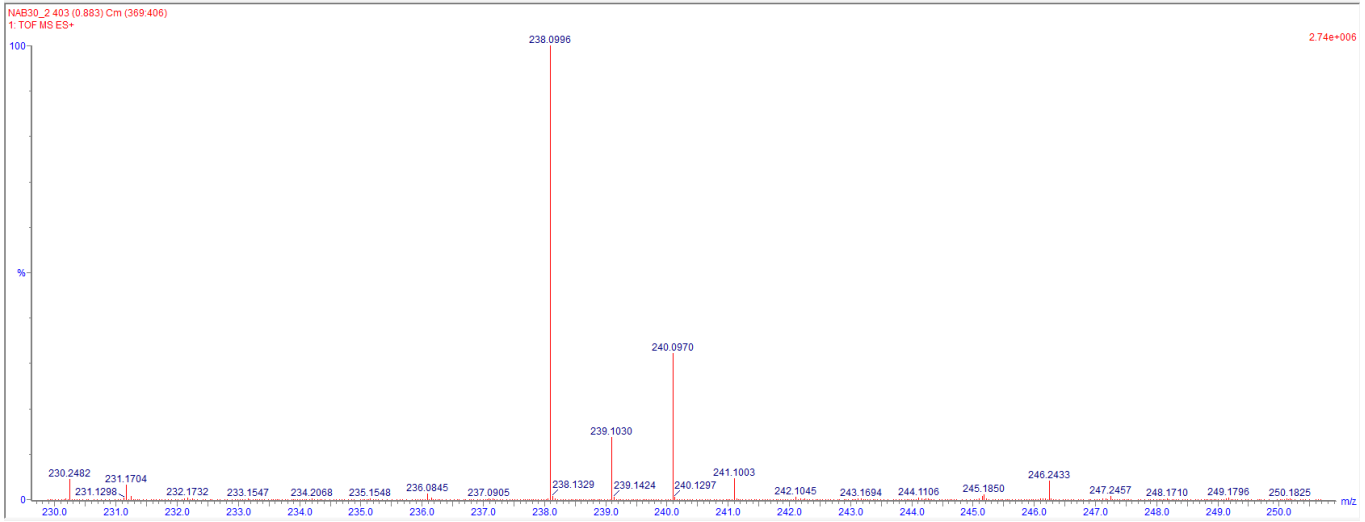
(3e) 3-t-butylo-5-(4-chlorofenylo)-2-izoksazolina - Metoda A:



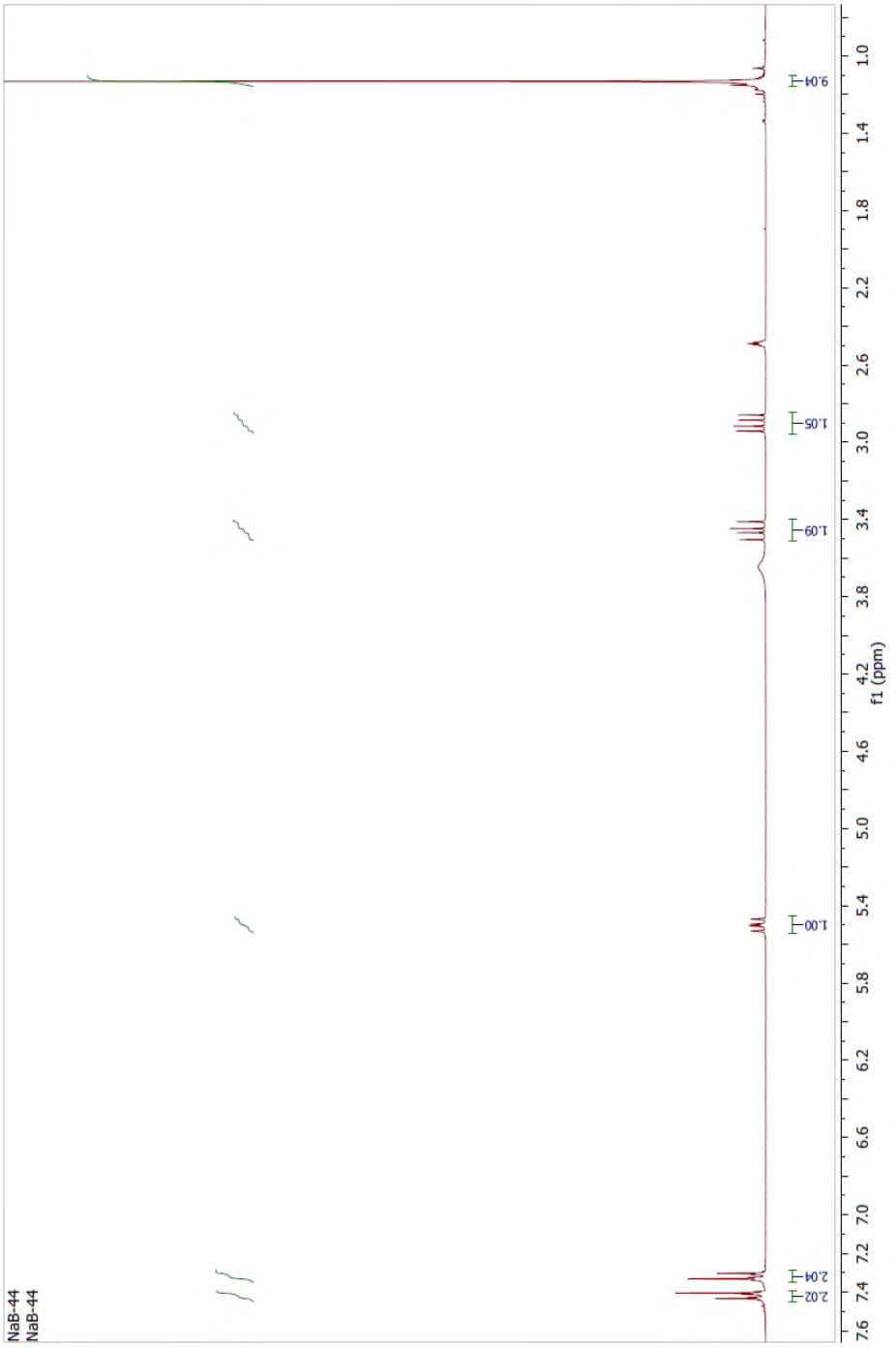

27.73
32.57
41.73
79.99
127.63
128.43
140.50
NaB-30.2_13c
CM190Fr1
f1 (ppm)

Tolerance = 100.0 mDa / DBE: min = -100.0, max = 200.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
68 formula(e) evaluated with 10 results within limits (up to 3 closest results for each mass)
Elements Used:

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	N	O	23Na	Cl
238.0996	100.00	238.0999	-0.3	-1.3	5.5	C13 H17 N O Cl	697.9	0.258	77.29	13	17	1	1		1
		238.0975	2.1	8.8	2.5	C11 H18 N O 23Na Cl	699.1	1.482	22.71	11	18	1	1	1	1
		238.0765	23.1	97.0	1.5	C10 H18 N O Cl2	709.1	11.429	0.00	10	18	1	1		2



Metoda B:



Multiple Mass Analysis: 2 mass(es) processed

Tolerance = 100.0 mDa / DBE: min = -100.0, max = 200.0

Element prediction: Off

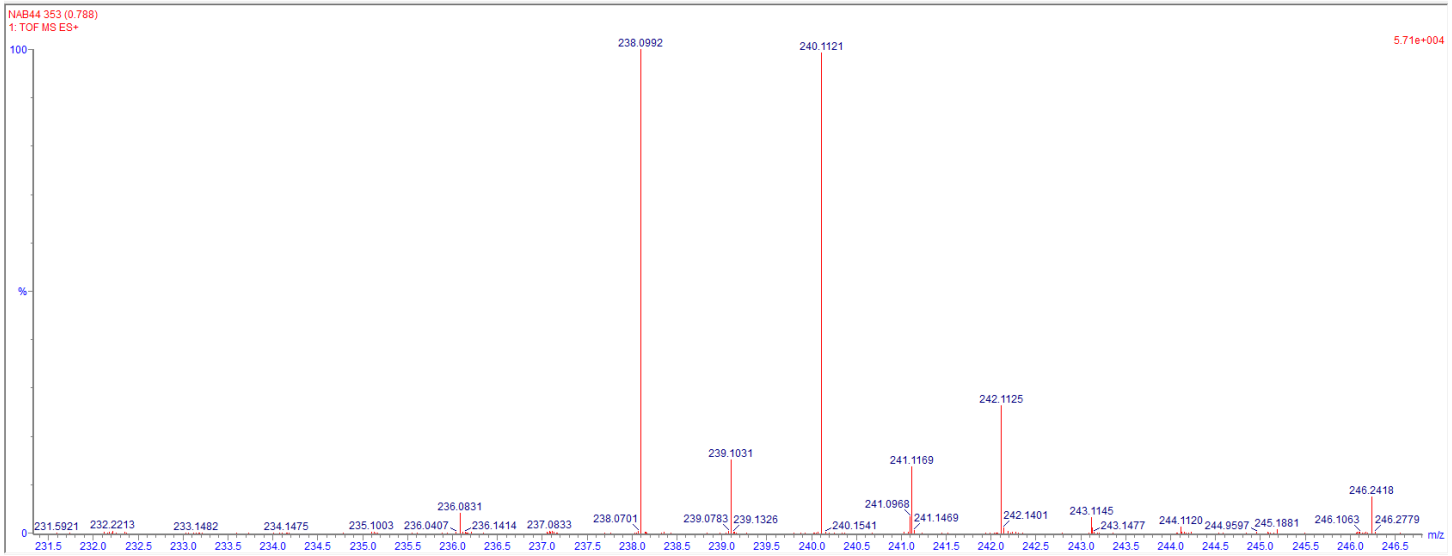
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

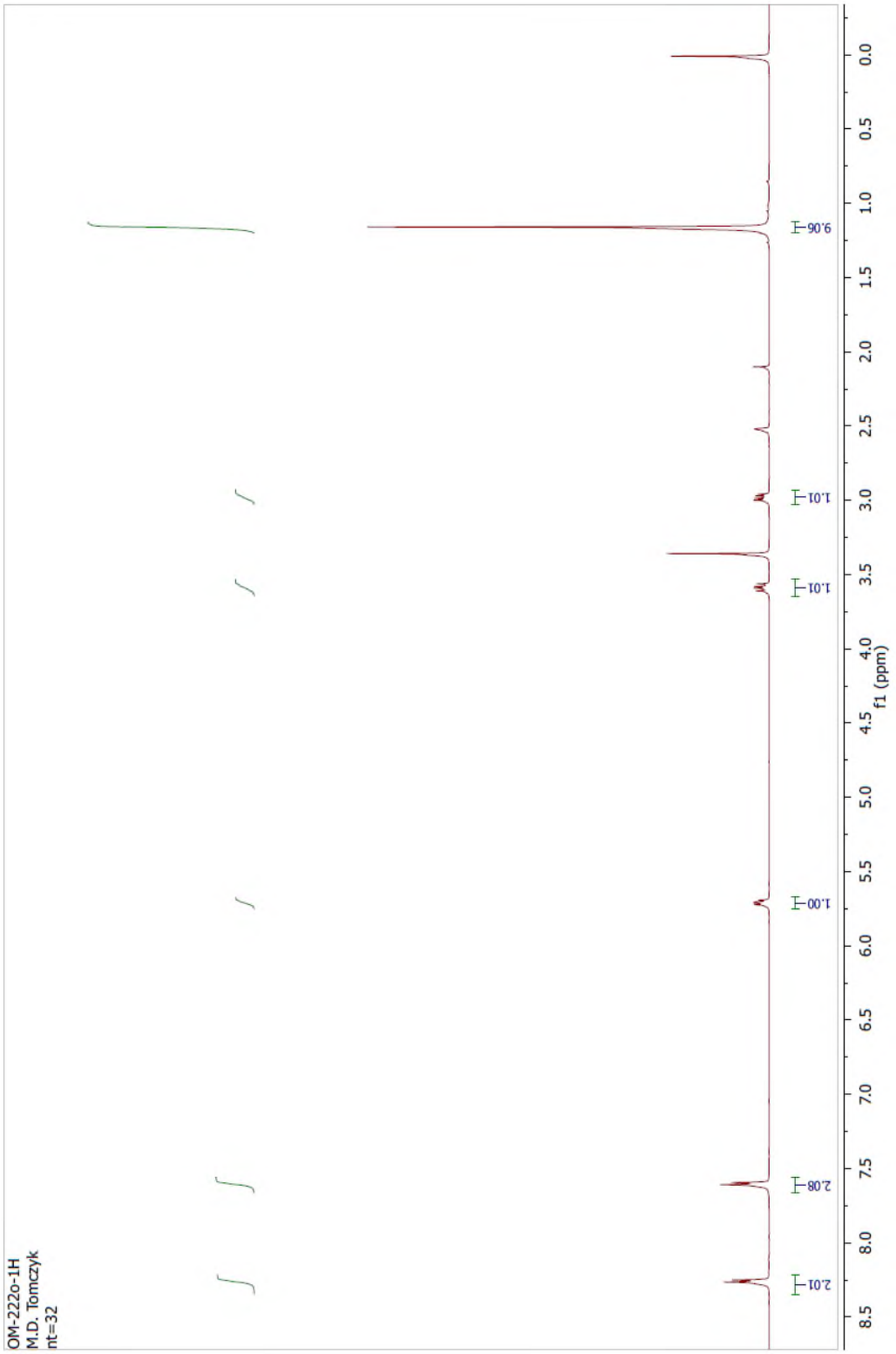
133 formula(e) evaluated with 20 results within limits (up to 3 closest results for each mass)

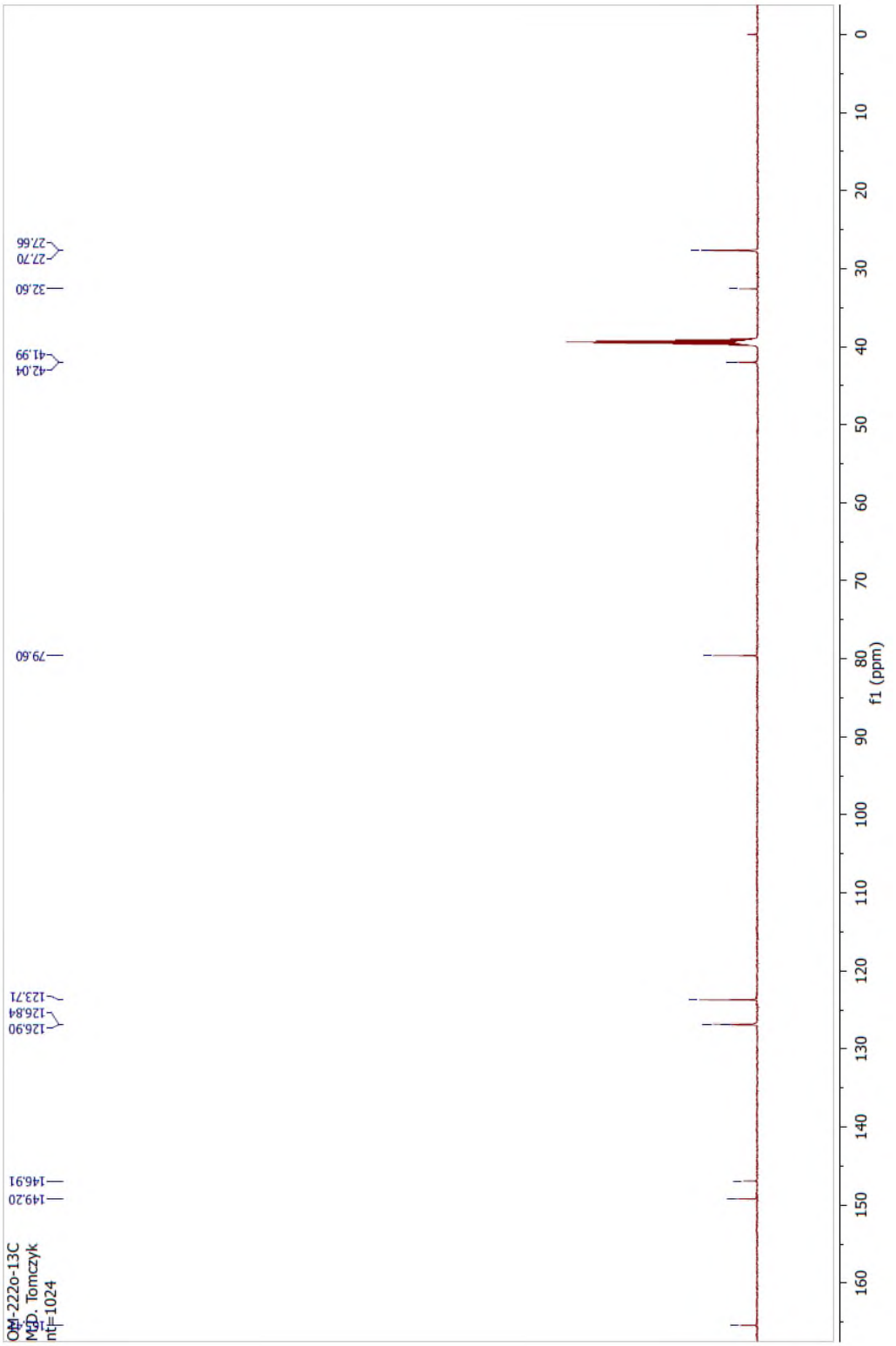
Elements Used:

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	N	O	23Na	Cl
238.0992	100.00	238.0999	-0.7	-2.9	5.5	C13 H17 N O Cl	232.9	3.205	4.05	13	17	1	1		1
		238.0975	1.7	7.1	2.5	C11 H18 N O 23Na Cl	233.0	3.223	3.98	11	18	1	1	1	1
		238.0765	22.7	95.3	1.5	C10 H18 N O Cl2	229.8	0.084	91.96	10	18	1	1		2
240.1121	99.37	240.1131	-1.0	-4.2	1.5	C11 H20 N O 23Na Cl	233.0	0.893	40.94	11	20	1	1	1	1
		240.1155	-3.4	-14.2	4.5	C13 H19 N O Cl	232.7	0.529	58.94	13	19	1	1		1
		240.0922	19.9	82.9	0.5	C10 H20 N O Cl2	238.9	6.727	0.12	10	20	1	1		2



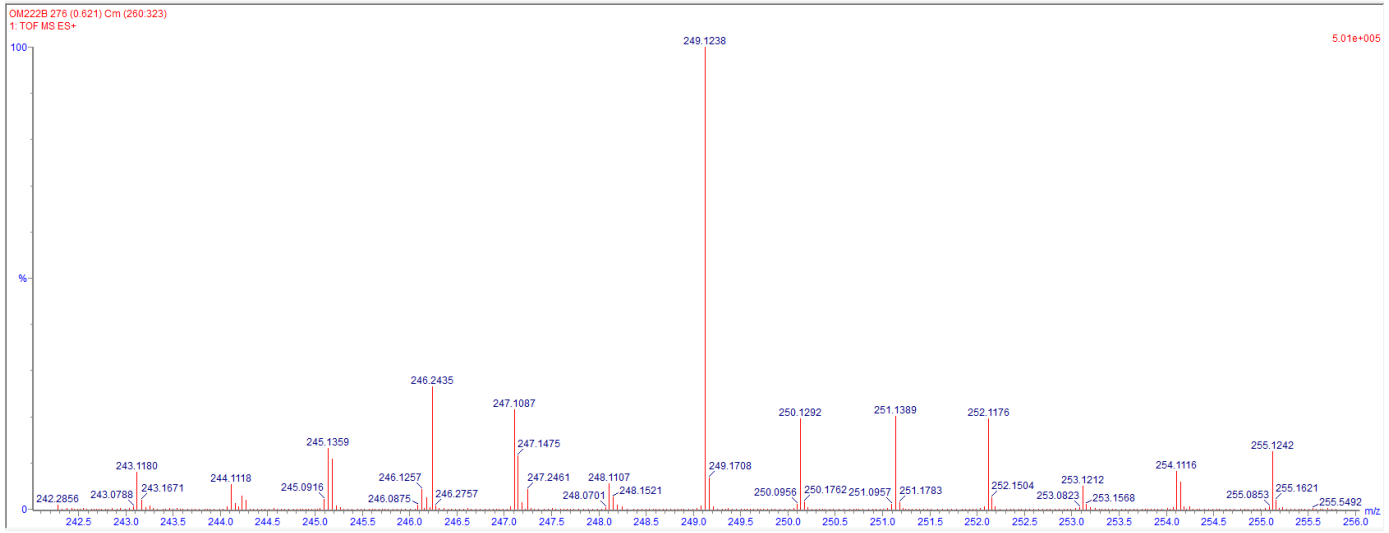
(3f) 3-t-butylo-5-(4-nitrofenylo)-2-izoksazolina - Metoda A:



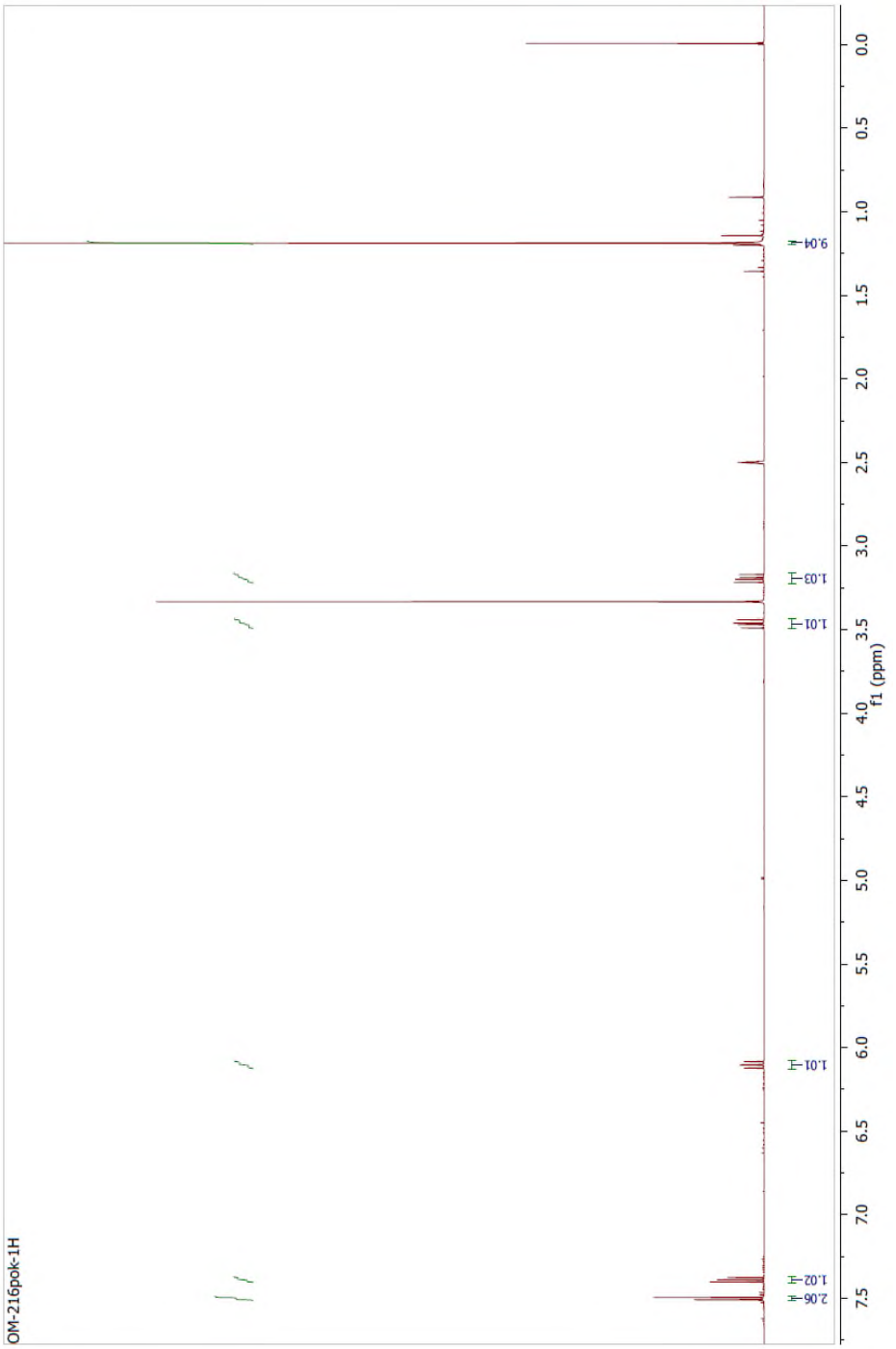

OM-222o-13C
M2D, Tomczyk
nt=1024
146.91
149.20
126.90
126.84
123.71
79.60
42.04
41.99
32.60
27.70
27.66
f1 (ppm)

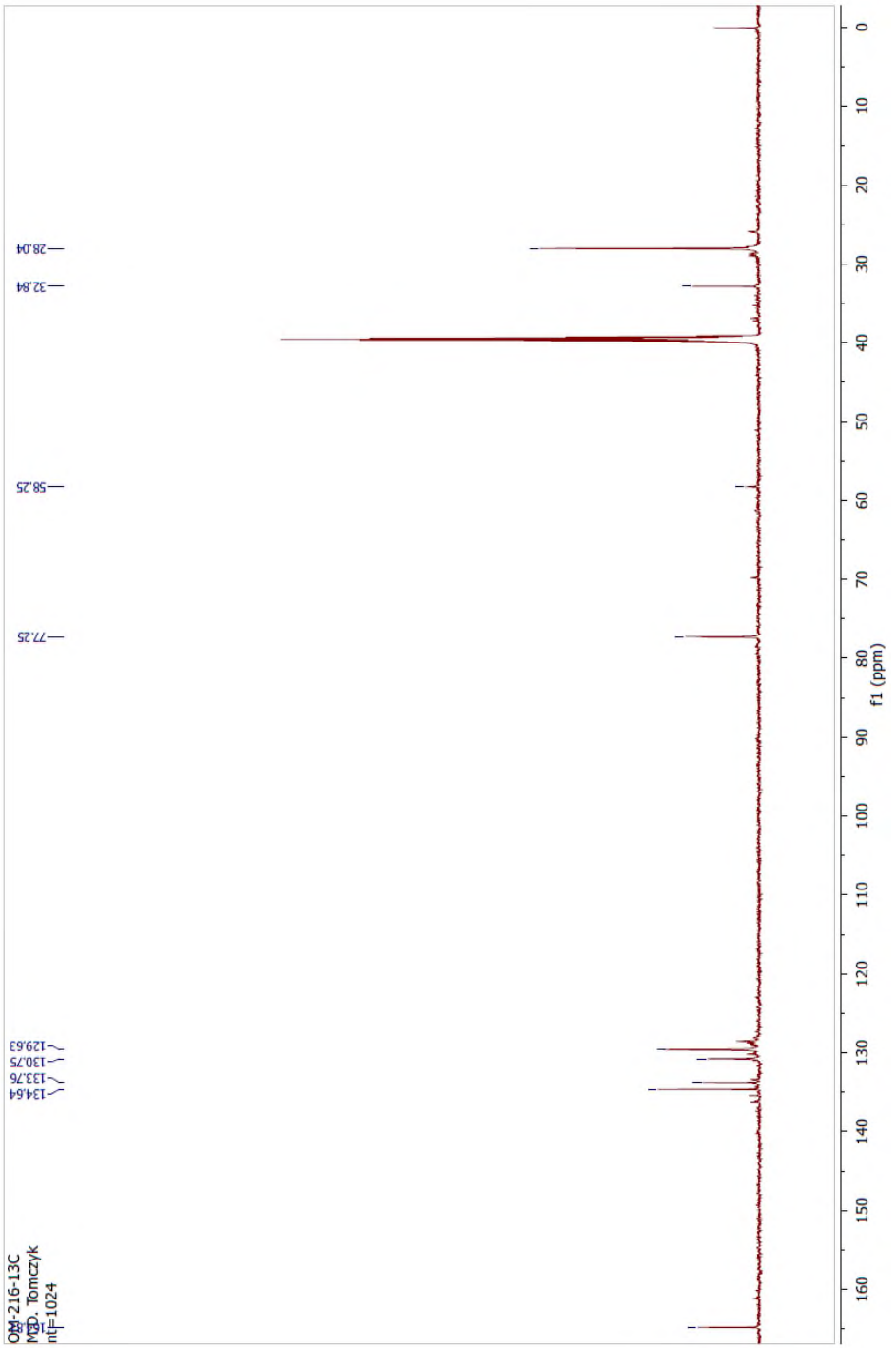
Tolerance = 100.0 mDa / DBE: min = -100.0, max = 200.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
101 formula(e) evaluated with 14 results within limits (up to 3 closest results for each mass)
Elements Used:

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	N	O	23Na
249.1238	100.00	249.1239	-0.1	-0.4	6.5	C13 H17 N2 O3	691.2	1.196	30.25	13	17	2	3	
		249.1215	2.3	9.2	3.5	C11 H18 N2 O3 23Na	691.4	1.458	23.26	11	18	2	3	1
		249.1028	21.0	84.3	11.5	C16 H13 N2 O	690.8	0.766	46.48	16	13	2	1	



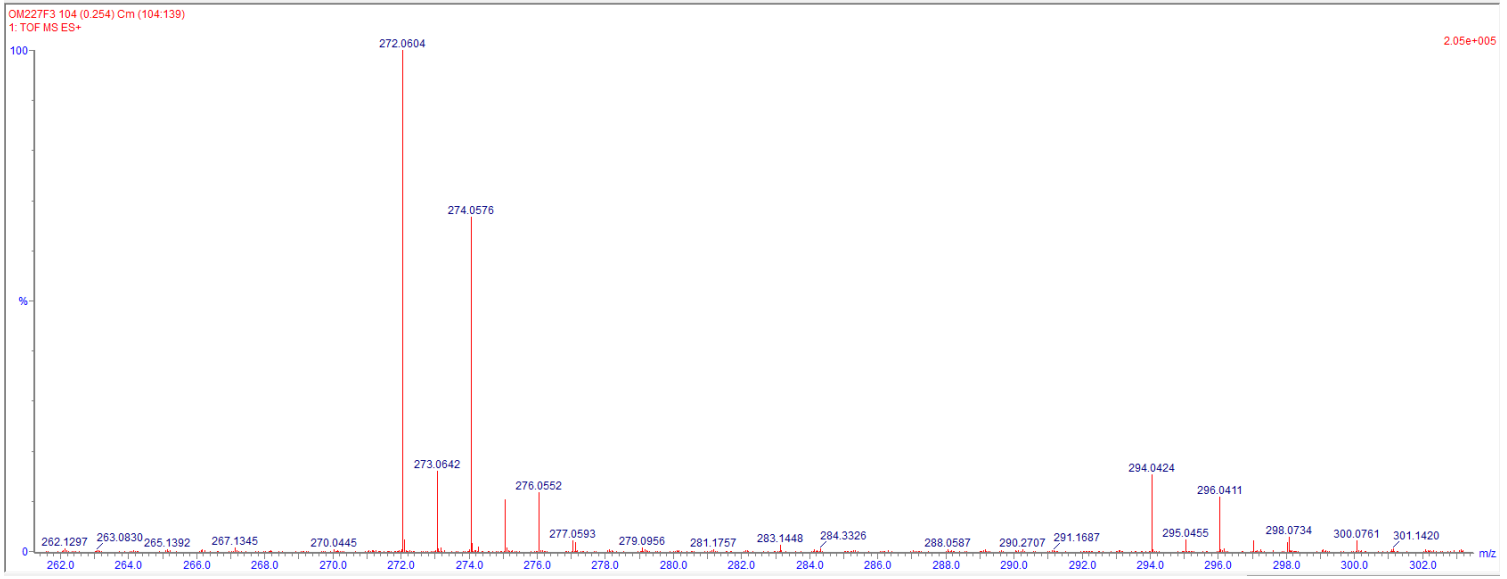
(3g) 3-t-butylo-5-(2,6-dichlorofenylo)-2-izoksazolina - metoda A:



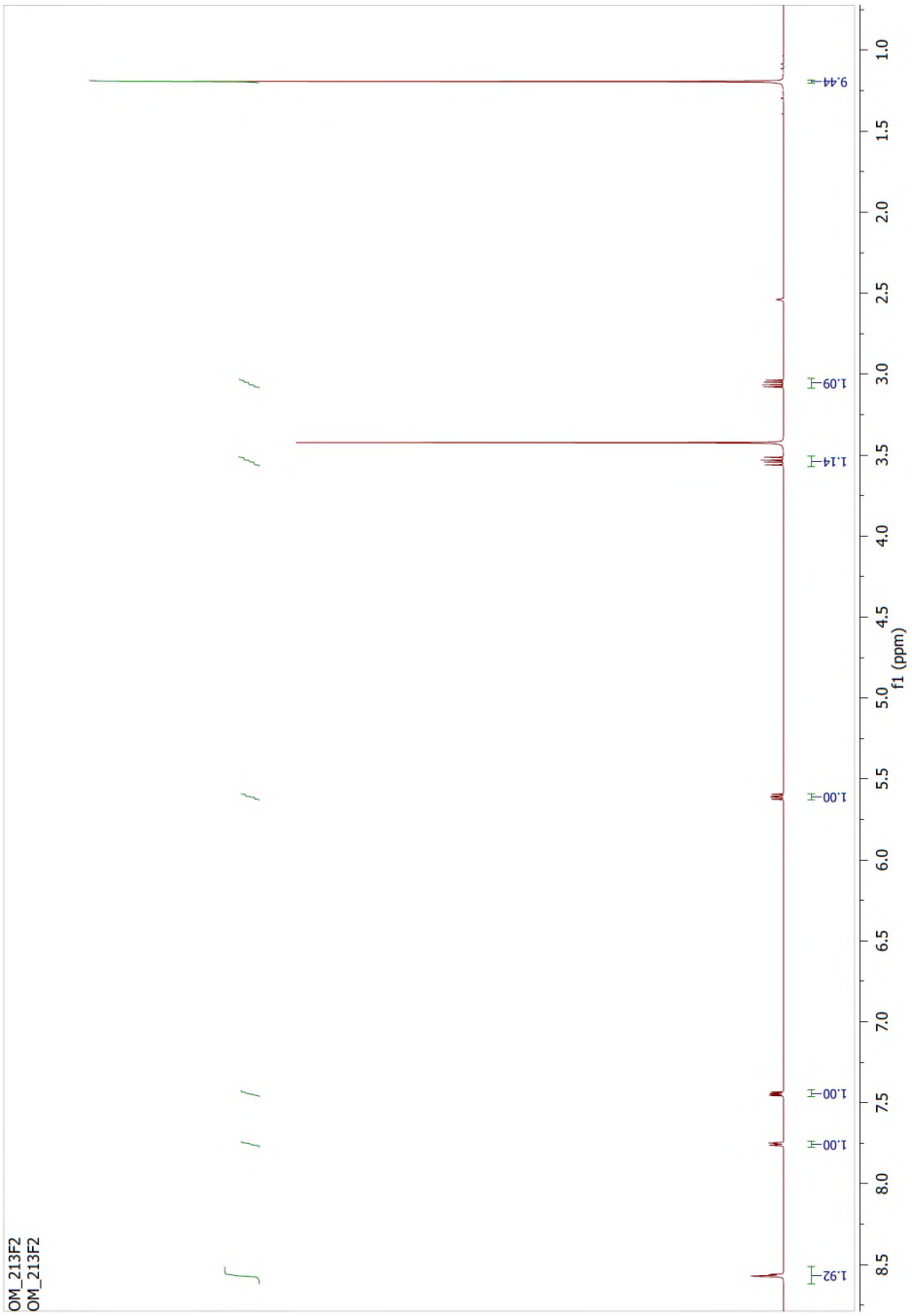

OM-216-13C
MD. Tomczyk
nt=1024
28.04
32.84
58.25
77.25
129.63
130.75
133.76
134.64
f1 (ppm)

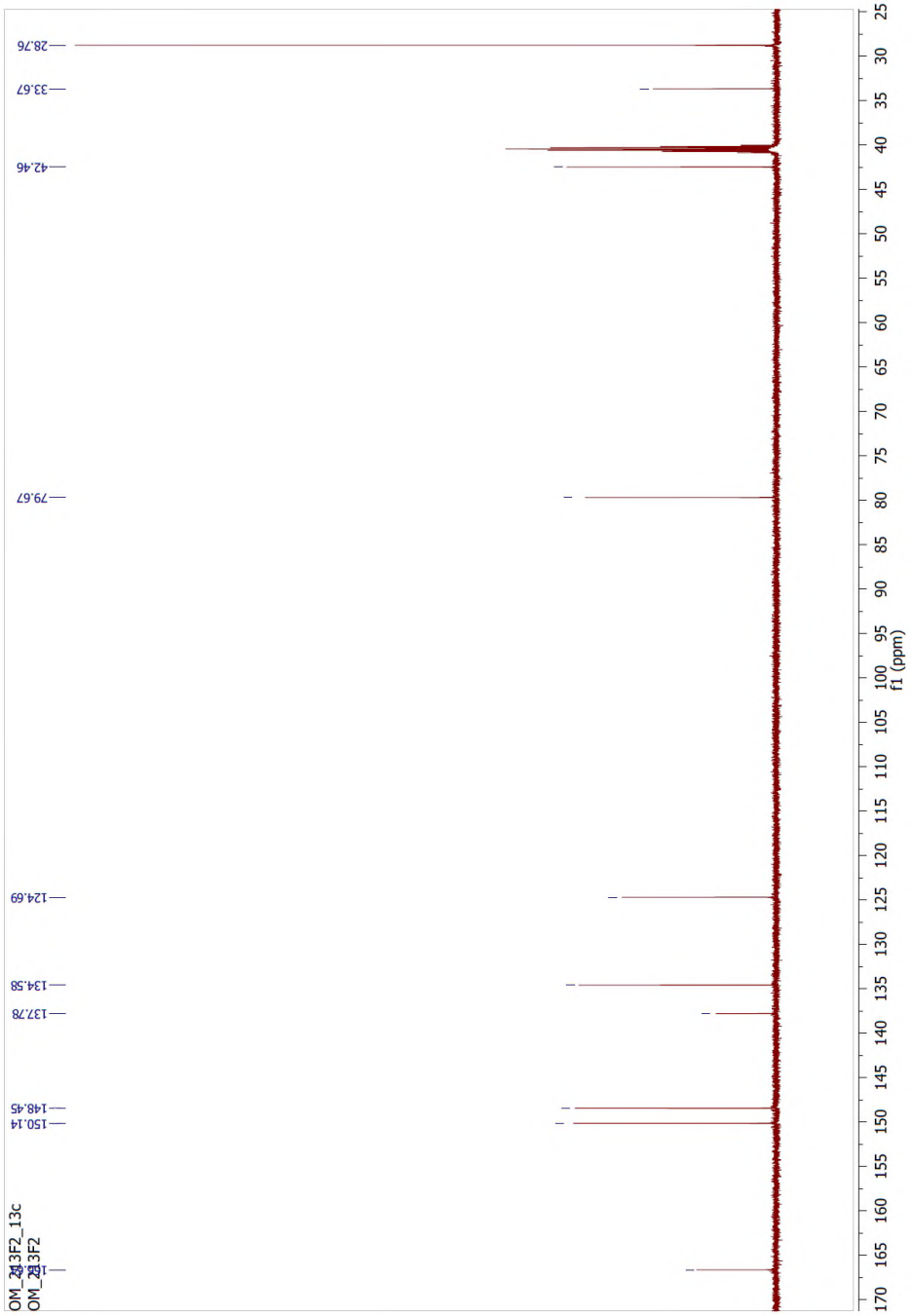
Multiple Mass Analysis: 2 mass(es) processed
Tolerance = 100.0 mDa / DBE: min = -100.0, max = 200.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
141 formula(e) evaluated with 18 results within limits (up to 3 closest results for each mass)
Elements Used:

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	N	O	23Na	Cl
272.0604	100.00	272.0609	-0.5	-1.8	5.5	C13 H16 N O Cl2	379.6	0.021	97.93	13	16	1	1		2
		272.0585	1.9	7.0	2.5	C11 H17 N O 23Na Cl2	383.4	3.878	2.07	11	17	1	1	1	2
		272.0818	-21.4	-78.7	6.5	C14 H16 N O 23Na Cl	390.2	10.676	0.00	14	16	1	1	1	1
274.0576	66.70	274.0741	-16.5	-60.2	1.5	C11 H19 N O 23Na Cl2	322.2	2.917	5.41	11	19	1	1	1	2
		274.0765	-18.9	-69.0	4.5	C13 H18 N O Cl2	322.2	2.858	5.74	13	18	1	1		2
		274.0975	-39.9	-145.6	5.5	C14 H18 N O 23Na Cl	319.4	0.118	88.85	14	18	1	1	1	1



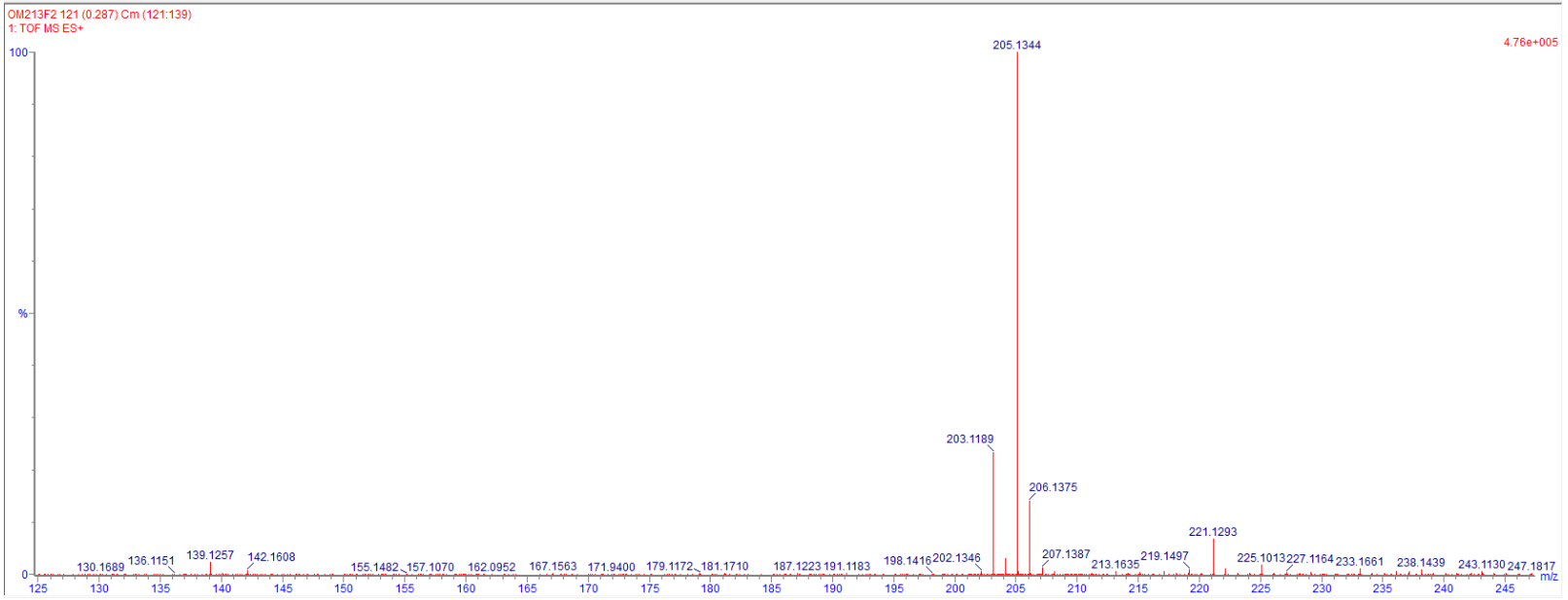
(3h) 3-t-butylo-5-(3-pirydynylo)-2-izoksazolina - Metoda A:



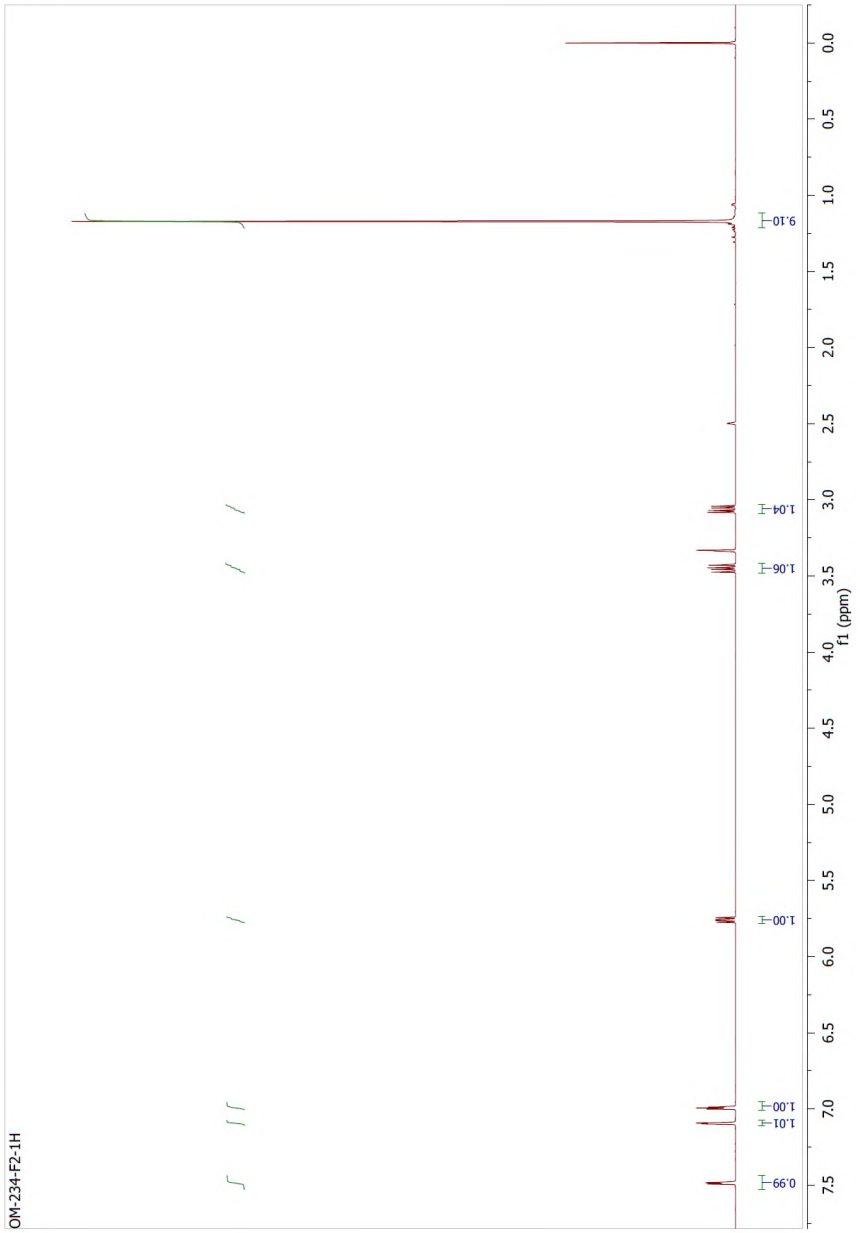

28.76
33.67
42.46
79.67
124.69
134.58
137.78
148.45
150.14
OM_243F2_13c
OM_243F2
f1 (ppm)

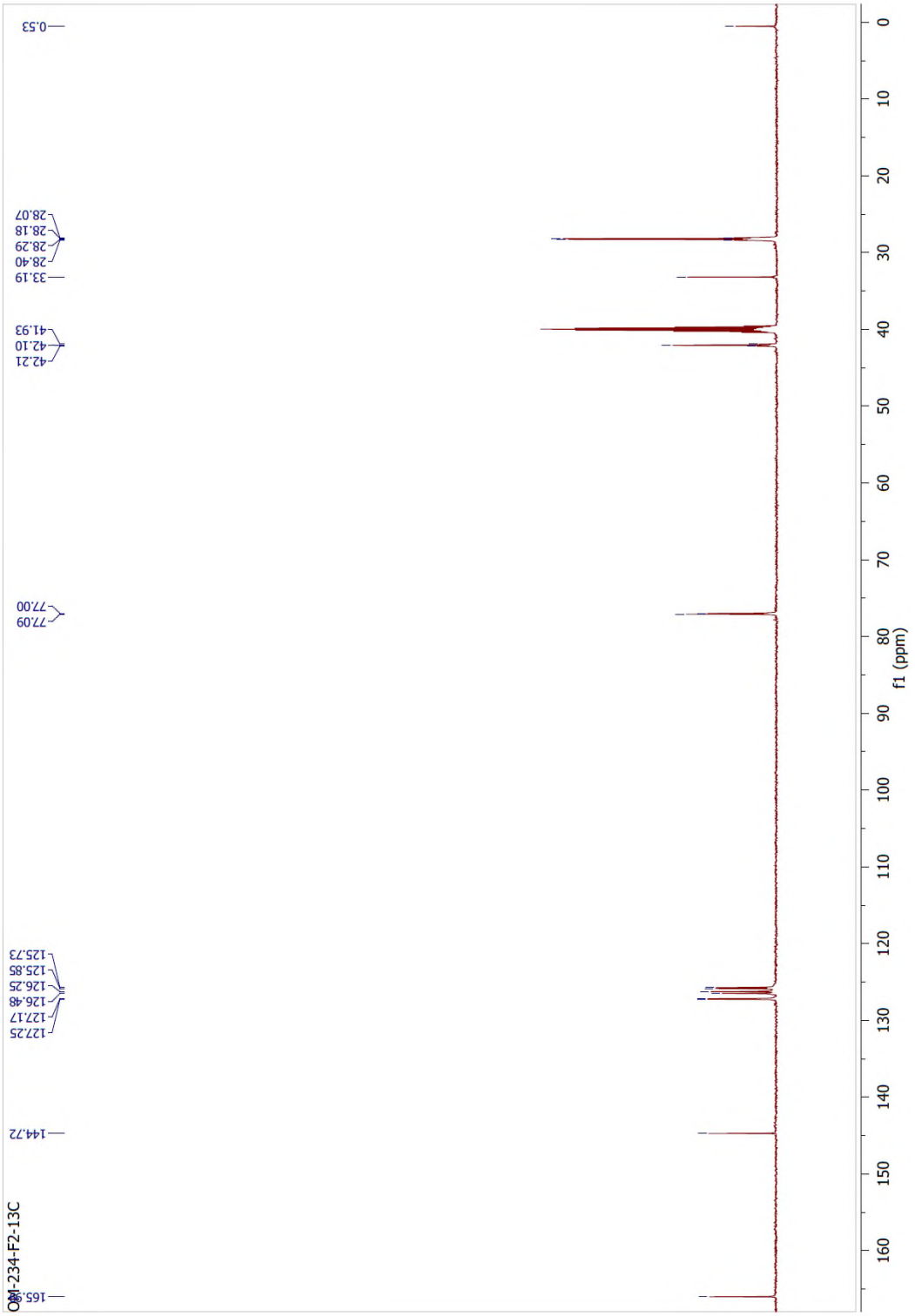
Tolerance = 100.0 mDa / DBE: min = -100.0, max = 200.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
33 formula(e) evaluated with 6 results within limits (up to 3 closest results for each mass)
Elements Used:

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	N	O	23Na
205.1344	100.00	205.1341	0.3	1.5	5.5	C12 H17 N2 O	318.5	0.001	99.93	12	17	2	1	
		205.1317	2.7	13.2	2.5	C10 H18 N2 O 23Na	326.4	7.952	0.04	10	18	2	1	1
		205.2256	-91.2	-444.6	-4.5	C9 H30 N2 O 23Na	326.6	8.118	0.03	9	30	2	1	1



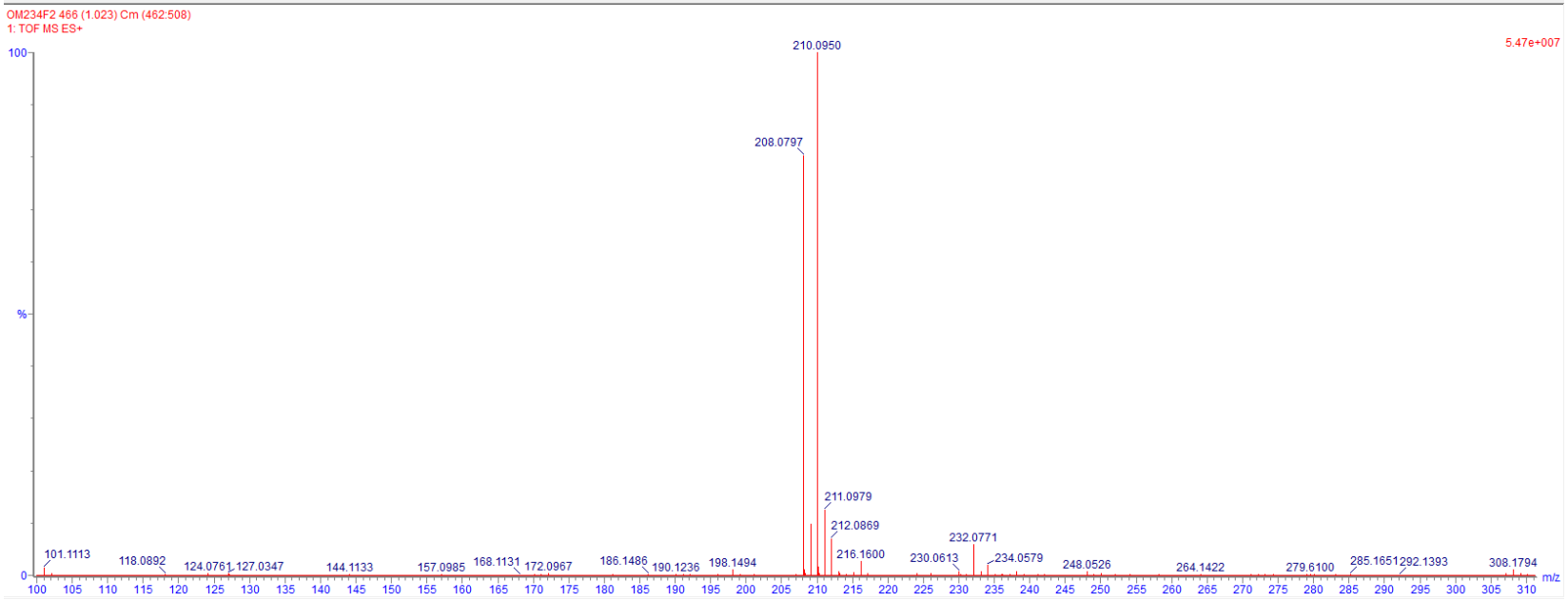
(3j) 3-t-butylo-5-(2-tienylo)-2-izoksazolina - Metoda A:



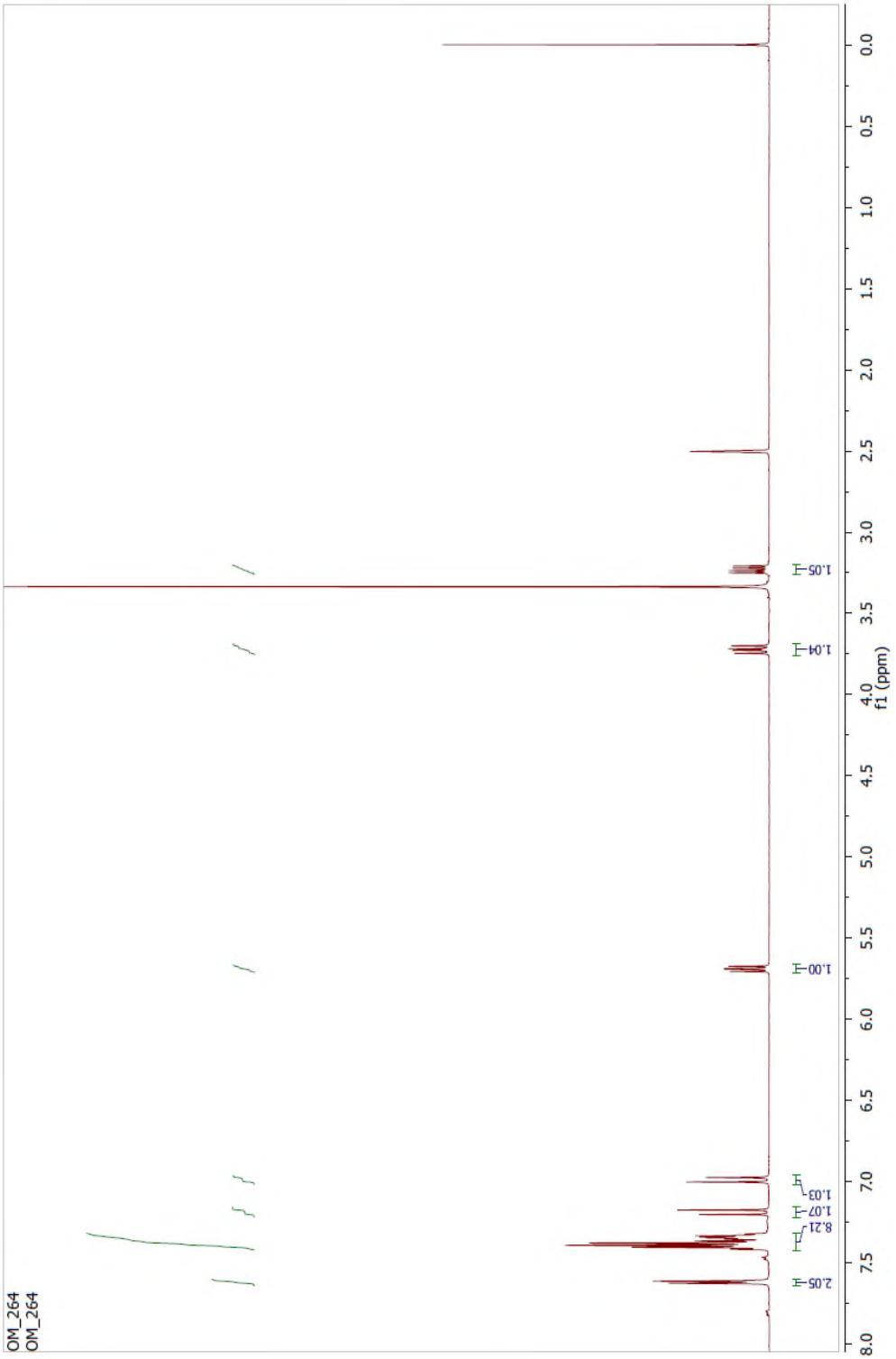

OM-234-F2-13C
165,98
144,72
127,25
127,17
126,48
126,25
125,85
125,73
77,00
77,09
42,21
42,10
41,93
33,19
28,40
28,29
28,18
28,07
0,53
f1 (ppm)

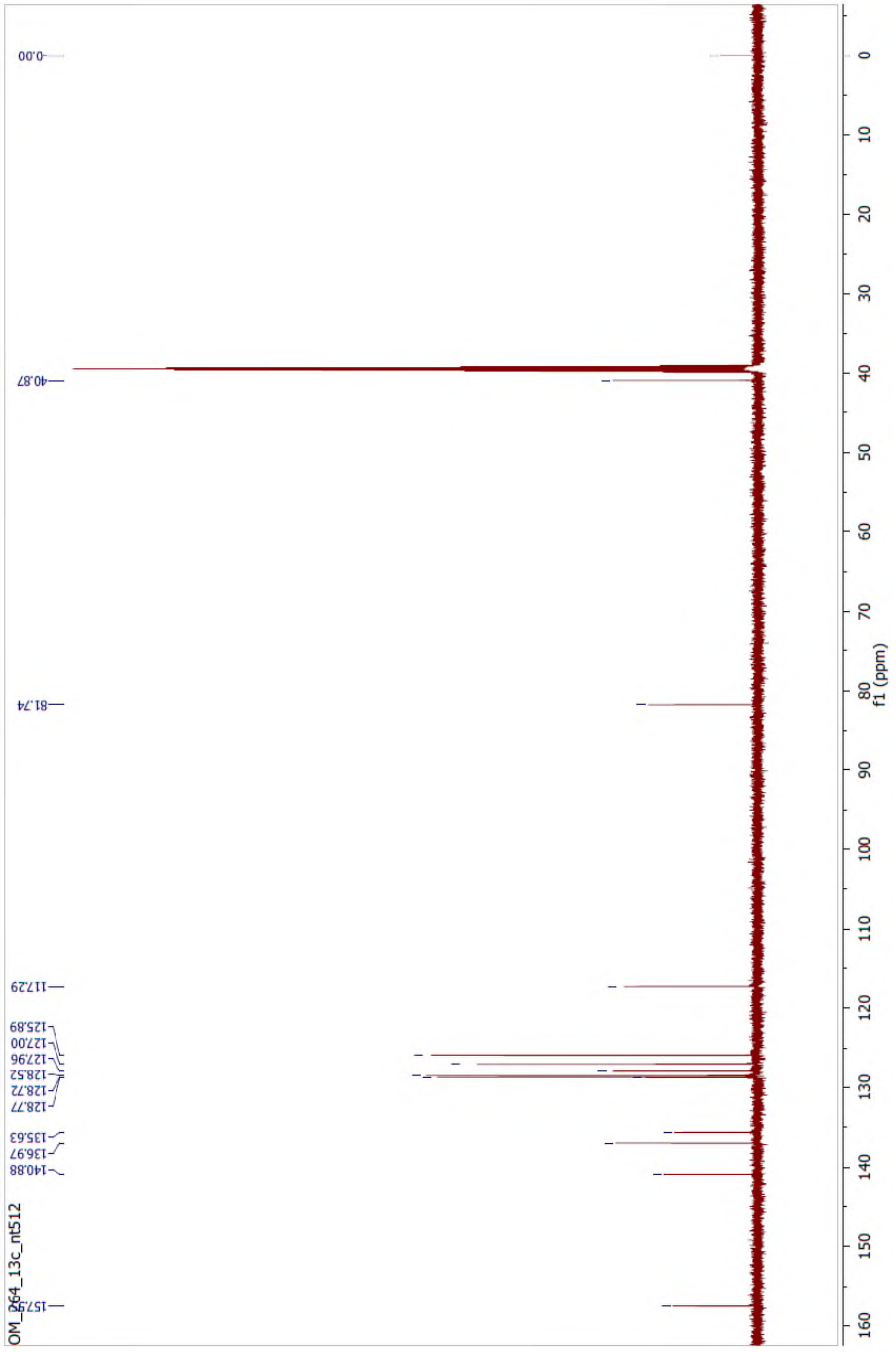
Multiple Mass Analysis: 2 mass(es) processed
Tolerance = 100.0 mDa / DBE: min = -100.0, max = 200.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
192 formula(e) evaluated with 28 results within limits (up to 3 closest results for each mass)
Elements Used:

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	N	O	23Na	S
208.0797	80.24	208.0796	0.1	0.5	5.5	C11 H14 N O S	1156.6	1.033	35.61	11	14	1	1		1
		208.0772	2.5	12.0	2.5	C9 H15 N O 23Na S	1156.7	1.123	32.53	9	15	1	1	1	1
		208.0983	-18.6	-89.4	-2.5	C6 H19 N O3 23Na S	1156.8	1.144	31.86	6	19	1	3	1	1
210.0950	100.00	210.0953	-0.3	-1.4	4.5	C11 H16 N O S	1071.1	0.526	59.11	11	16	1	1		1
		210.0929	2.1	10.0	1.5	C9 H17 N O 23Na S	1071.6	0.974	37.77	9	17	1	1	1	1
		210.1140	-19.0	-90.4	-3.5	C6 H21 N O3 23Na S	1074.1	3.466	3.12	6	21	1	3	1	1



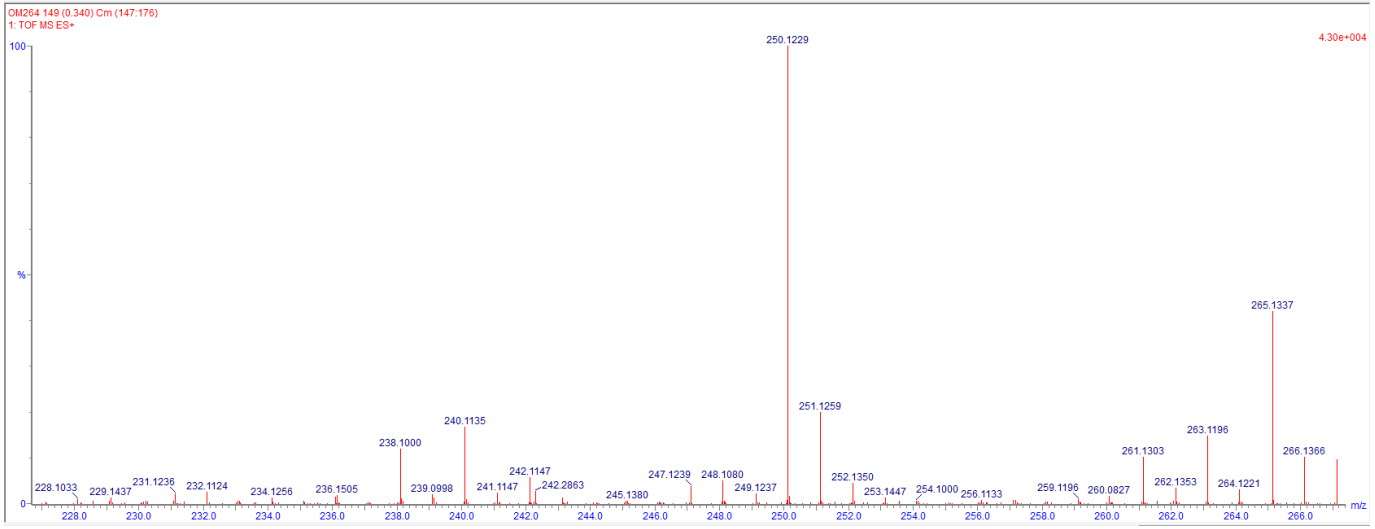
(3k) 5-fenylo-3-styrylo-2-izoksazolina - Metoda A:



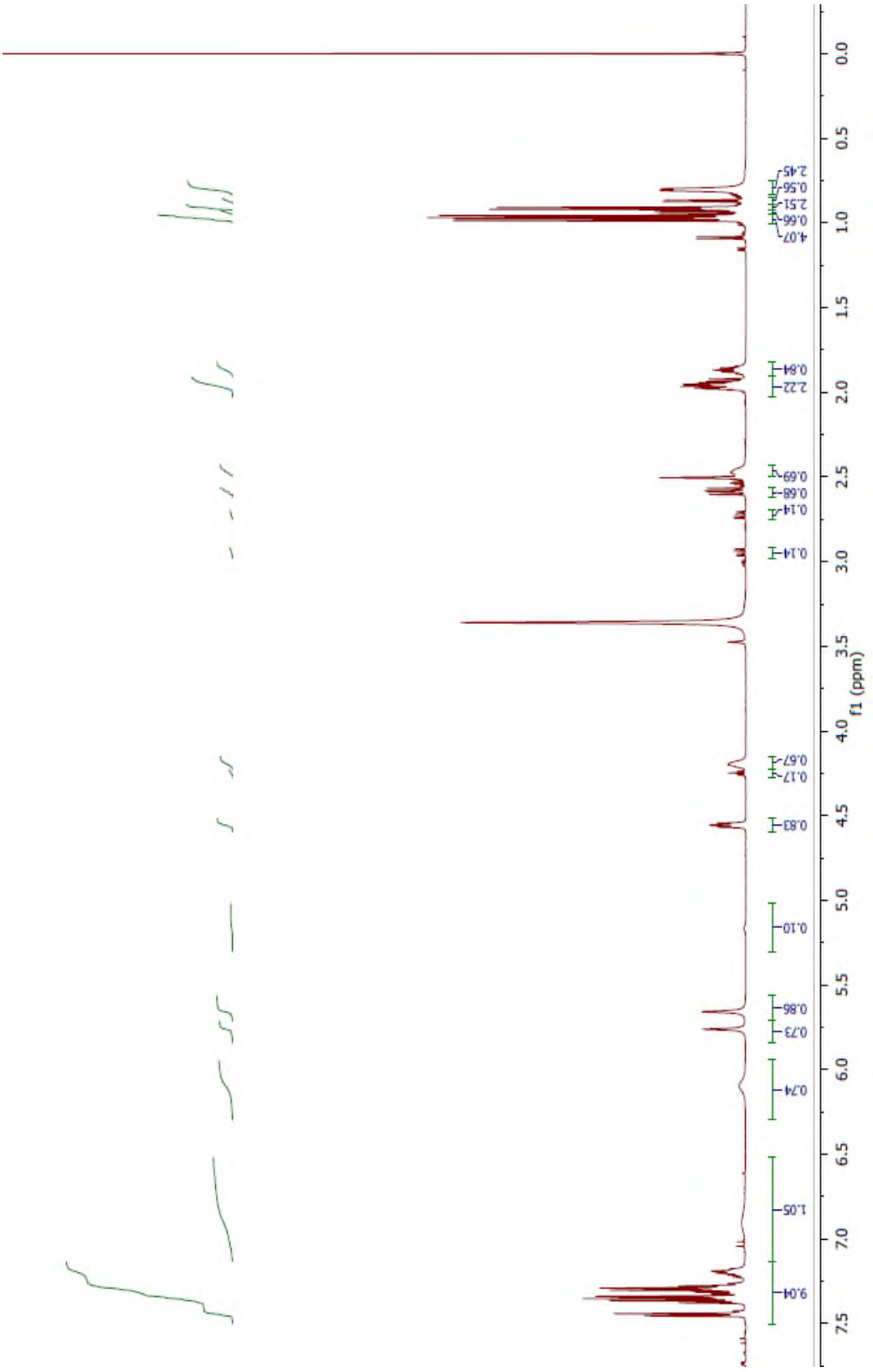

0.00
40.87
81.74
117.29
125.89
127.00
127.96
128.52
128.72
128.77
135.63
136.97
140.88
157.29
OM_264_13c_nt512
f1 (ppm)

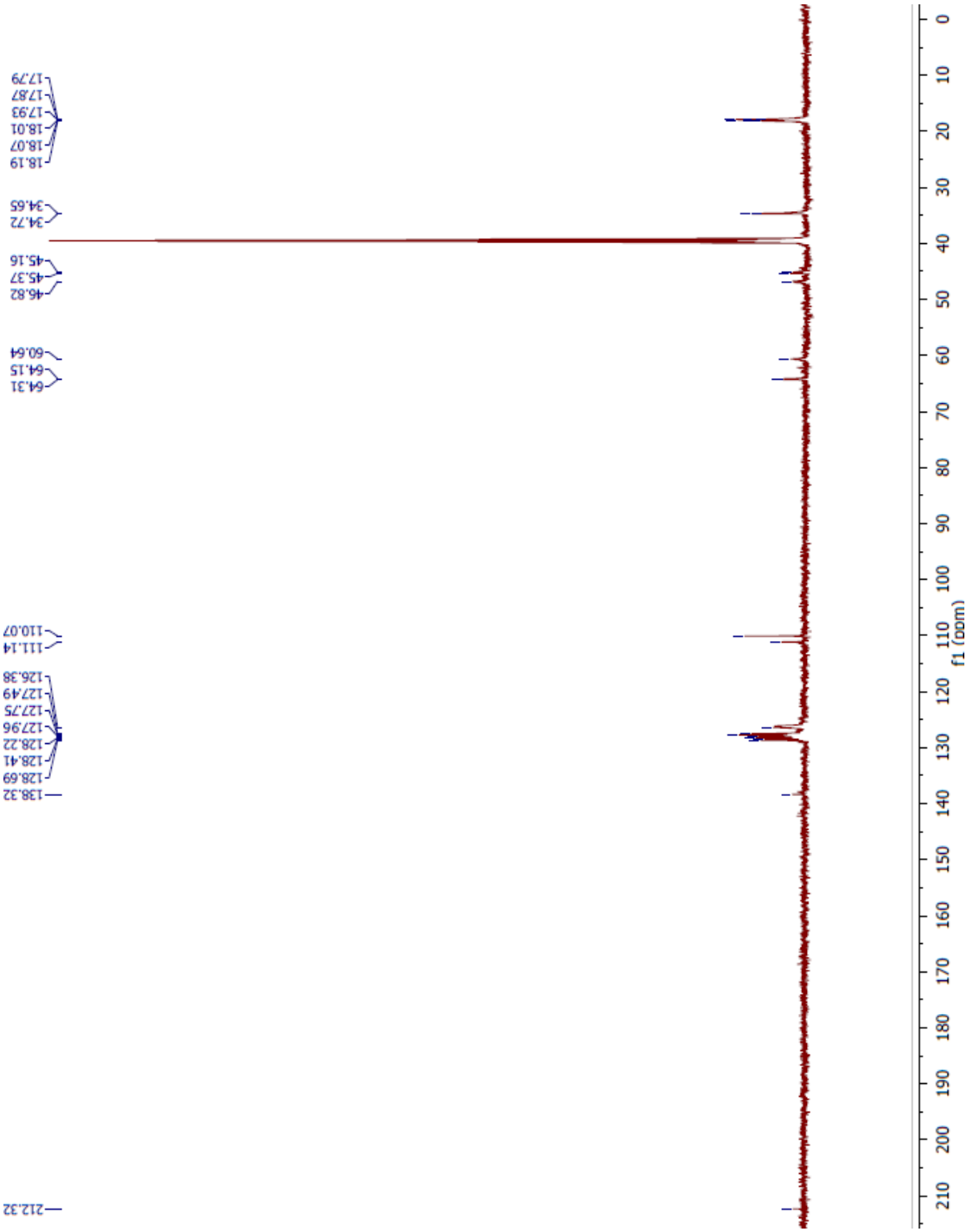
Multiple Mass Analysis: 2 mass(es) processed
Tolerance = 100.0 mDa / DBE: min = -100.0, max = 200.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
34 formula(e) evaluated with 6 results within limits (up to 3 closest results for each mass)
Elements Used:

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	N	O	23Na
250.1229	100.00	250.1232	-0.3	-1.2	10.5	C17 ...	1...	0.454	63.48	17	16	1	1	
		250.1208	2.1	8.4	7.5	C15 ...	1...	1.381	25.13	15	17	1	1	1
		250.2147	-91.8	-3...	0.5	C14 ...	1...	2.172	11.39	14	29	1	1	1
265.1337	41.98	---												

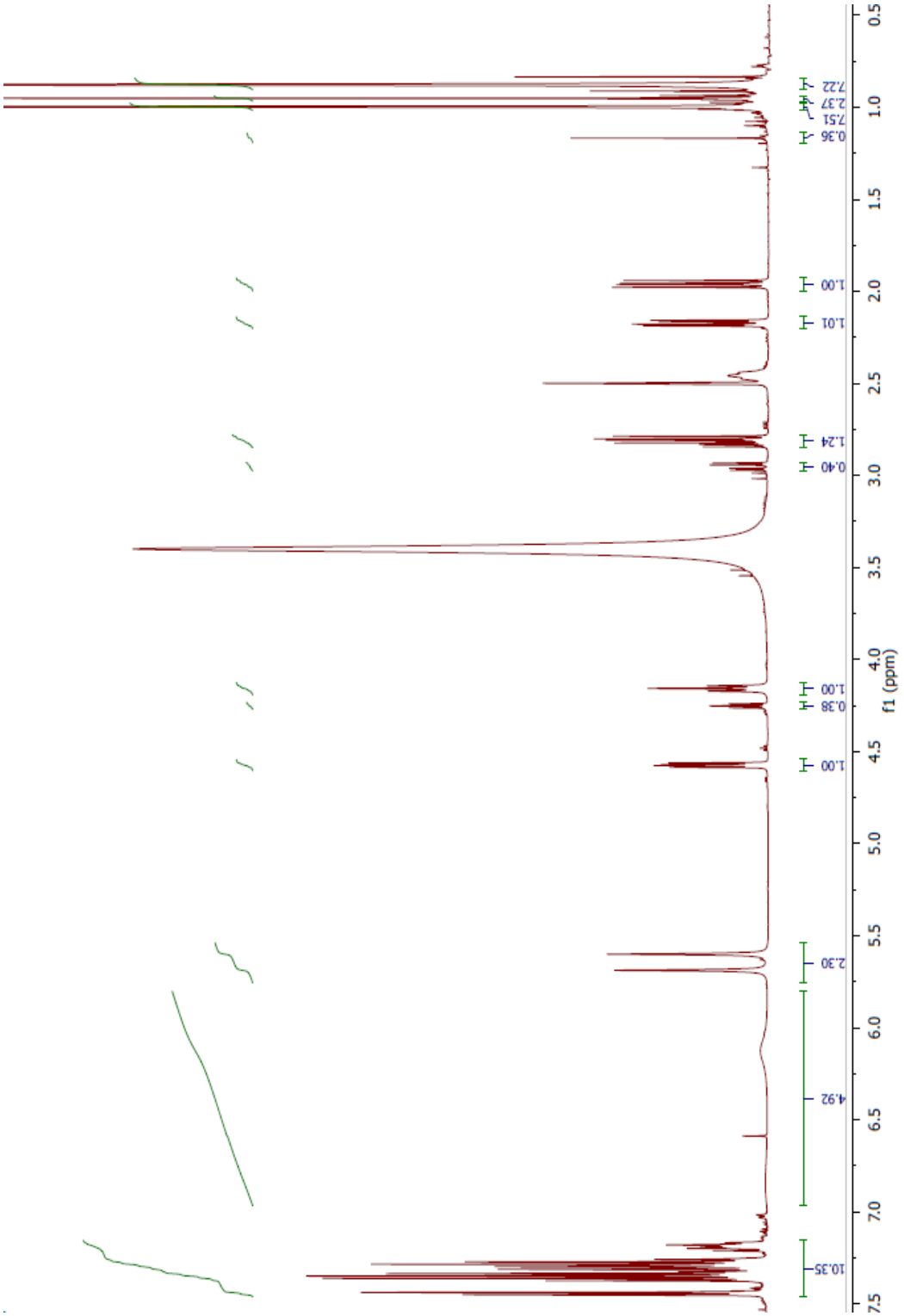


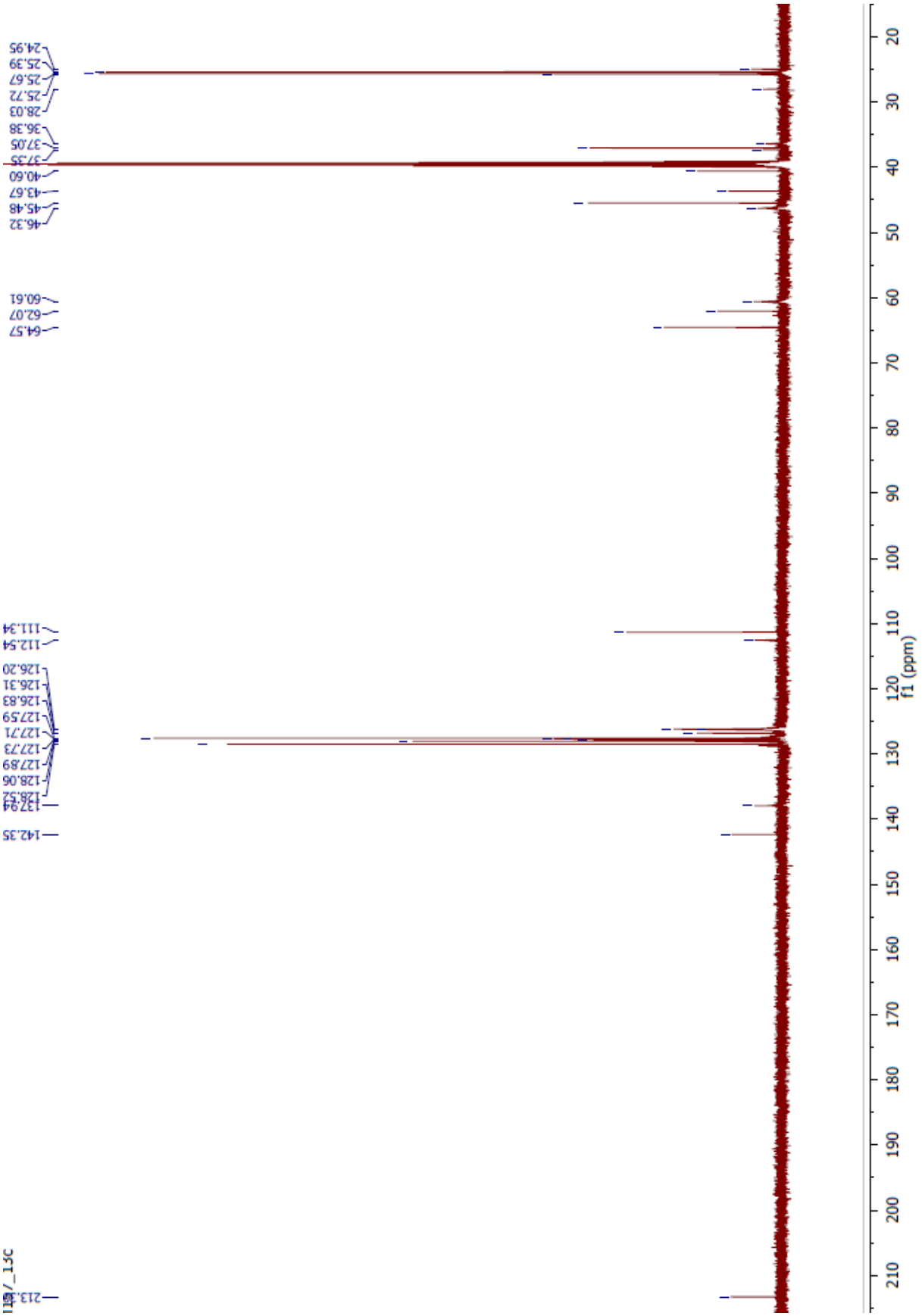
(4b) 5-izopropylo-5-hydroksy-3-fenyloizoksazolidina

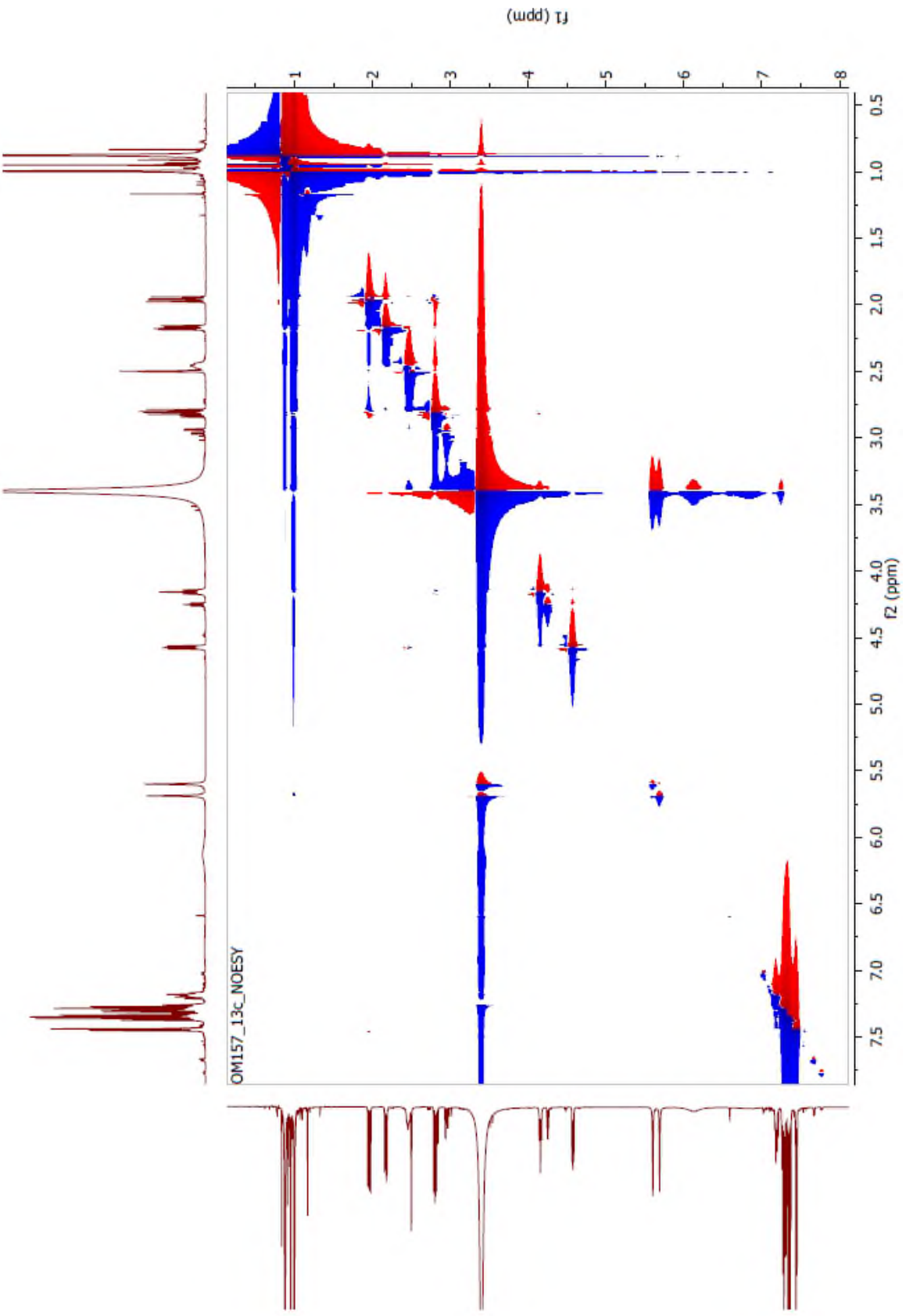


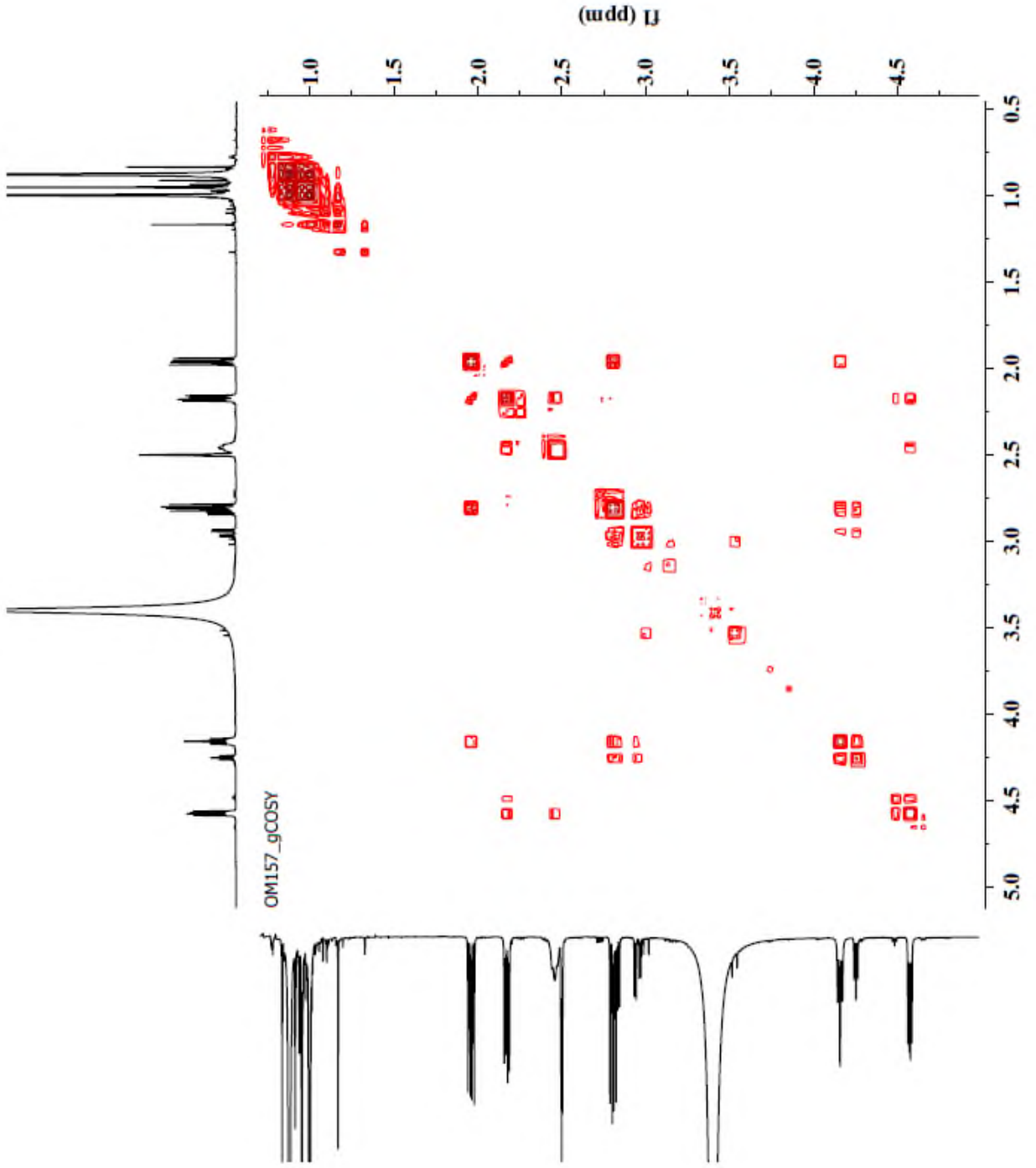

212.32
138.32
128.69
128.41
128.22
127.96
127.75
127.49
126.38
111.14
110.07
64.31
64.15
60.64
46.82
45.37
45.16
34.72
34.65
18.19
18.07
18.01
17.93
17.87
17.79
f1 (ppm)

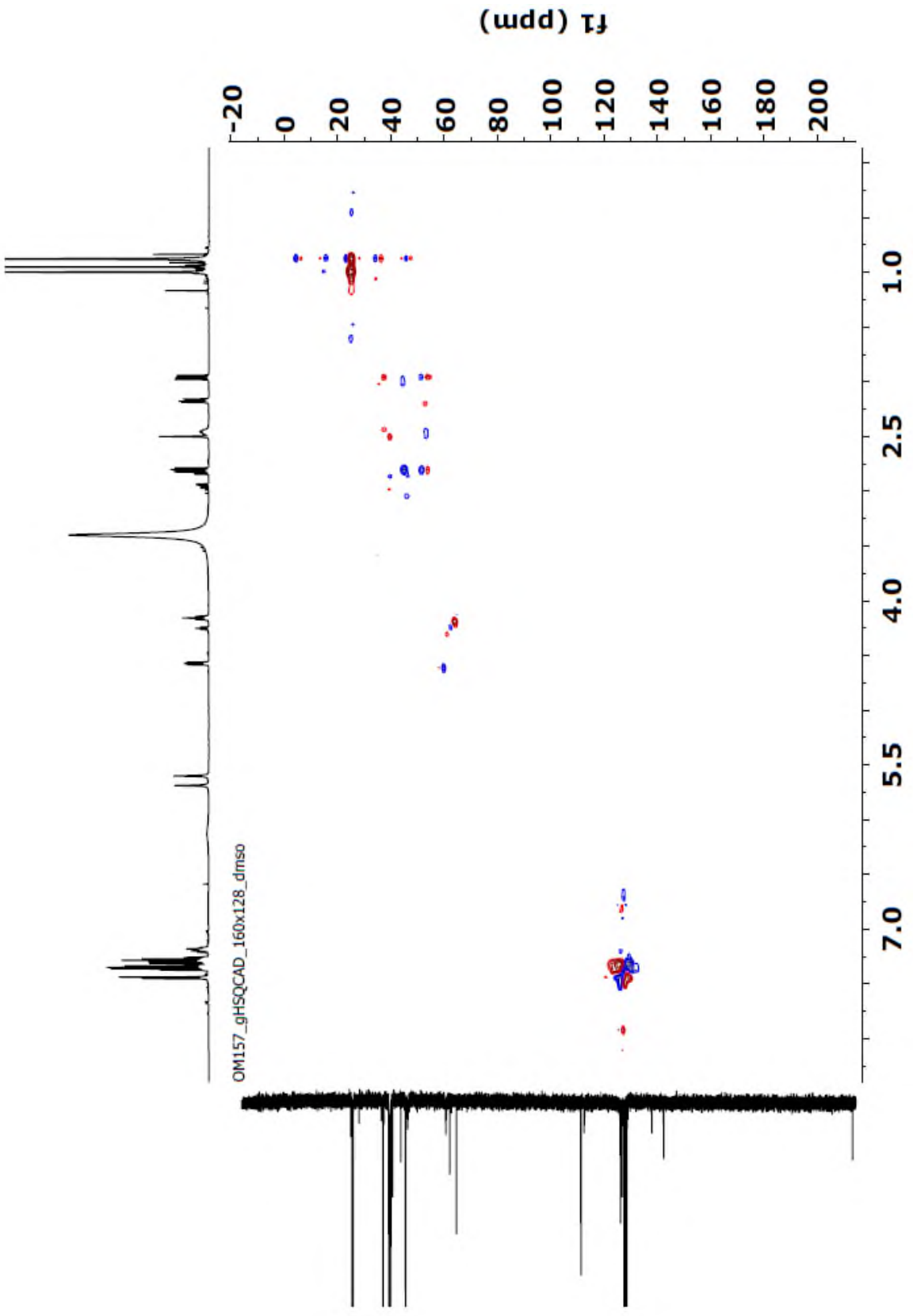
(4c) 5-tert-butylo-5-hydroksy-3-fenyloizoksazolidina




213.38
142.35
137.94
128.52
128.06
127.89
127.73
127.71
127.59
126.83
126.31
126.20
112.54
111.34
64.57
62.07
60.61
46.32
45.48
43.67
40.60
37.35
37.05
36.38
28.03
25.72
25.67
25.39
24.95
f1 (ppm)

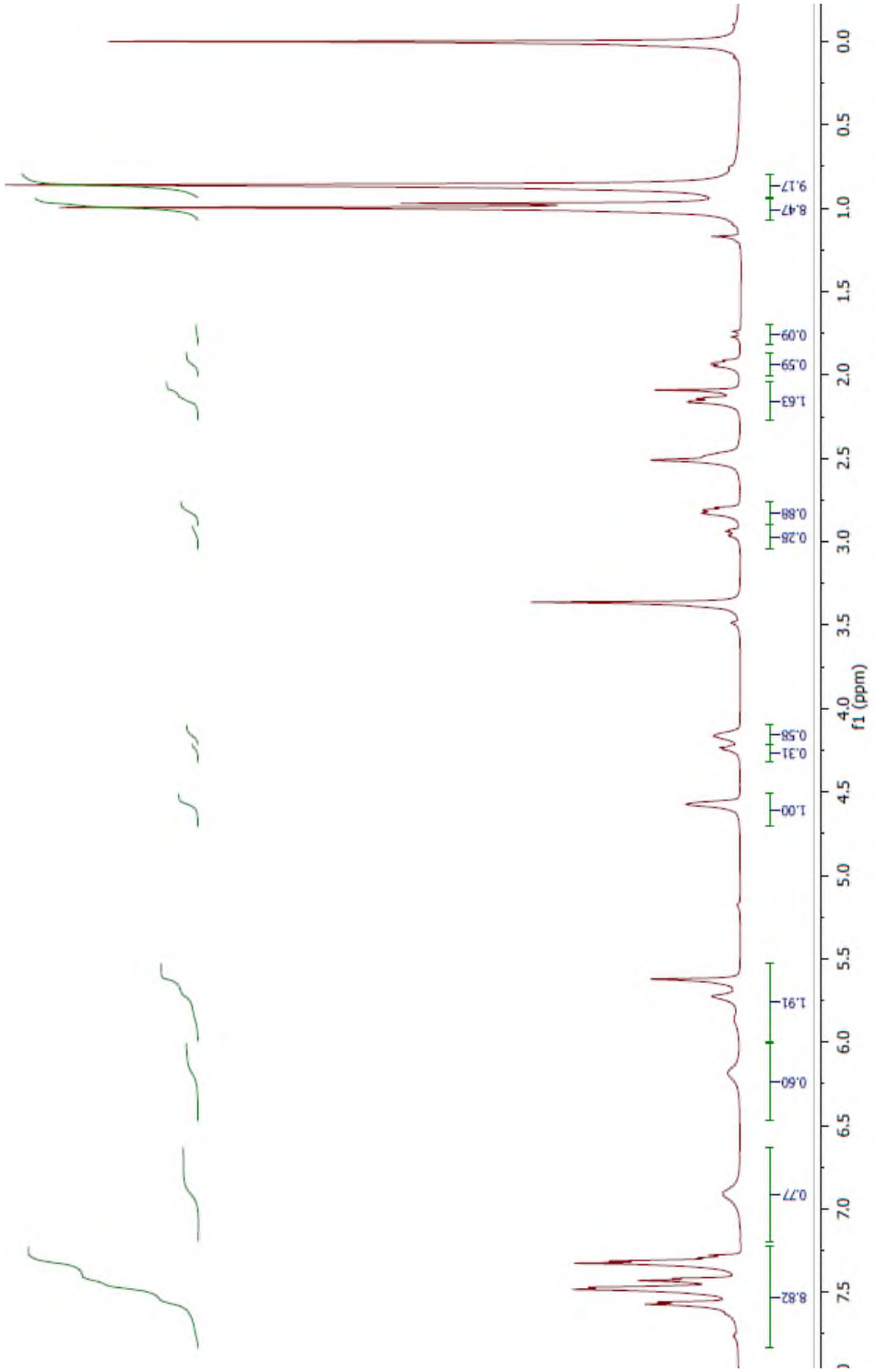

f1 (ppm)
f2 (ppm)
OM157_13c_NOESY

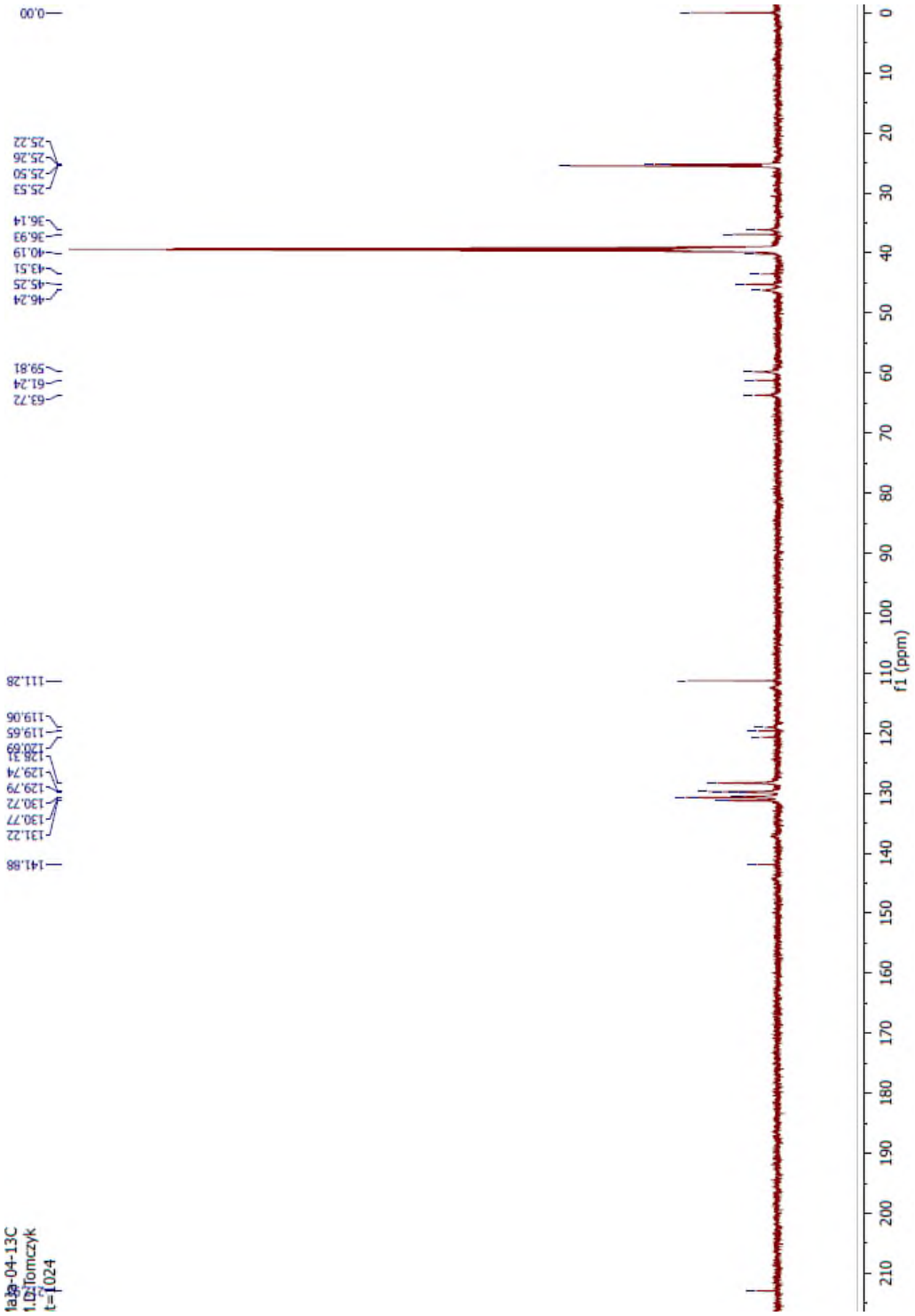

f1 (ppm)
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
0.5
OM157_gCOSY


f1 (ppm)
-20
0
20
40
60
80
100
120
140
160
180
200
1.0
2.5
4.0
5.5
7.0
OM157_gHSQCAD_160x128_dmso

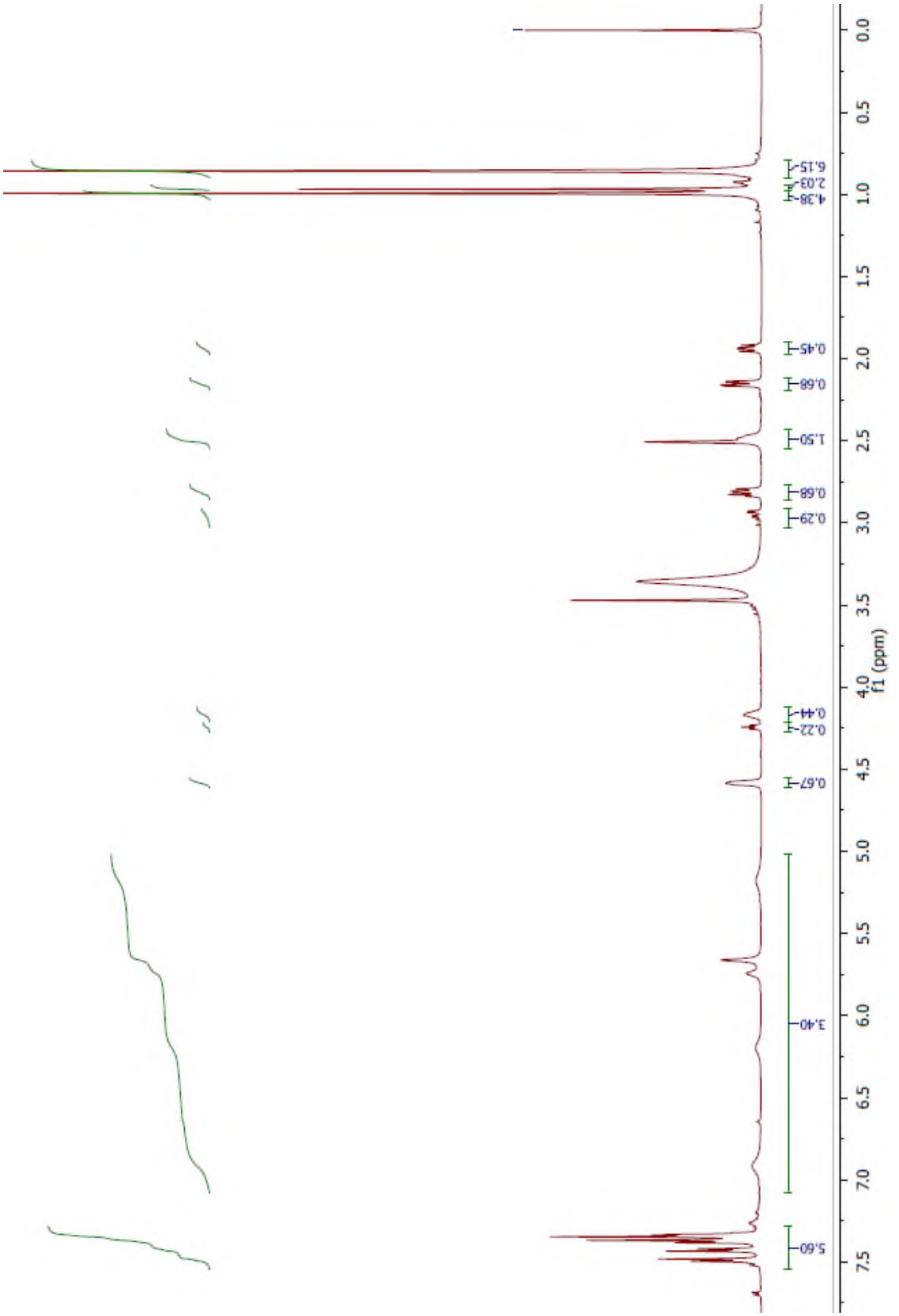
f1 (ppm)
0
20
40
60
80
100
120
140
160
180
200
220
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
f2 (ppm)

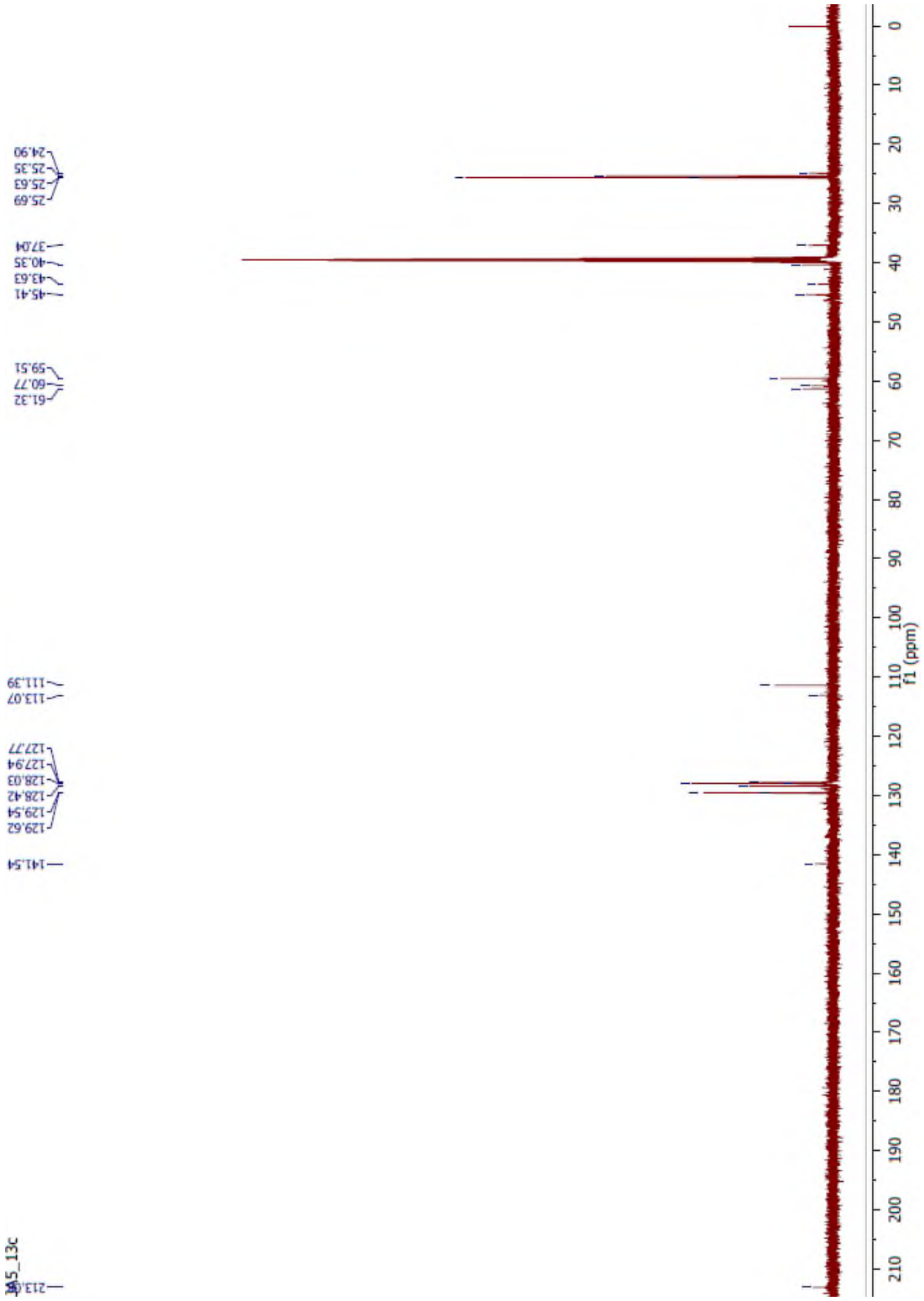
(4d) 5-tert-butylo-5-hydroksy-3-(4-bromofenylo)izoksazolidina



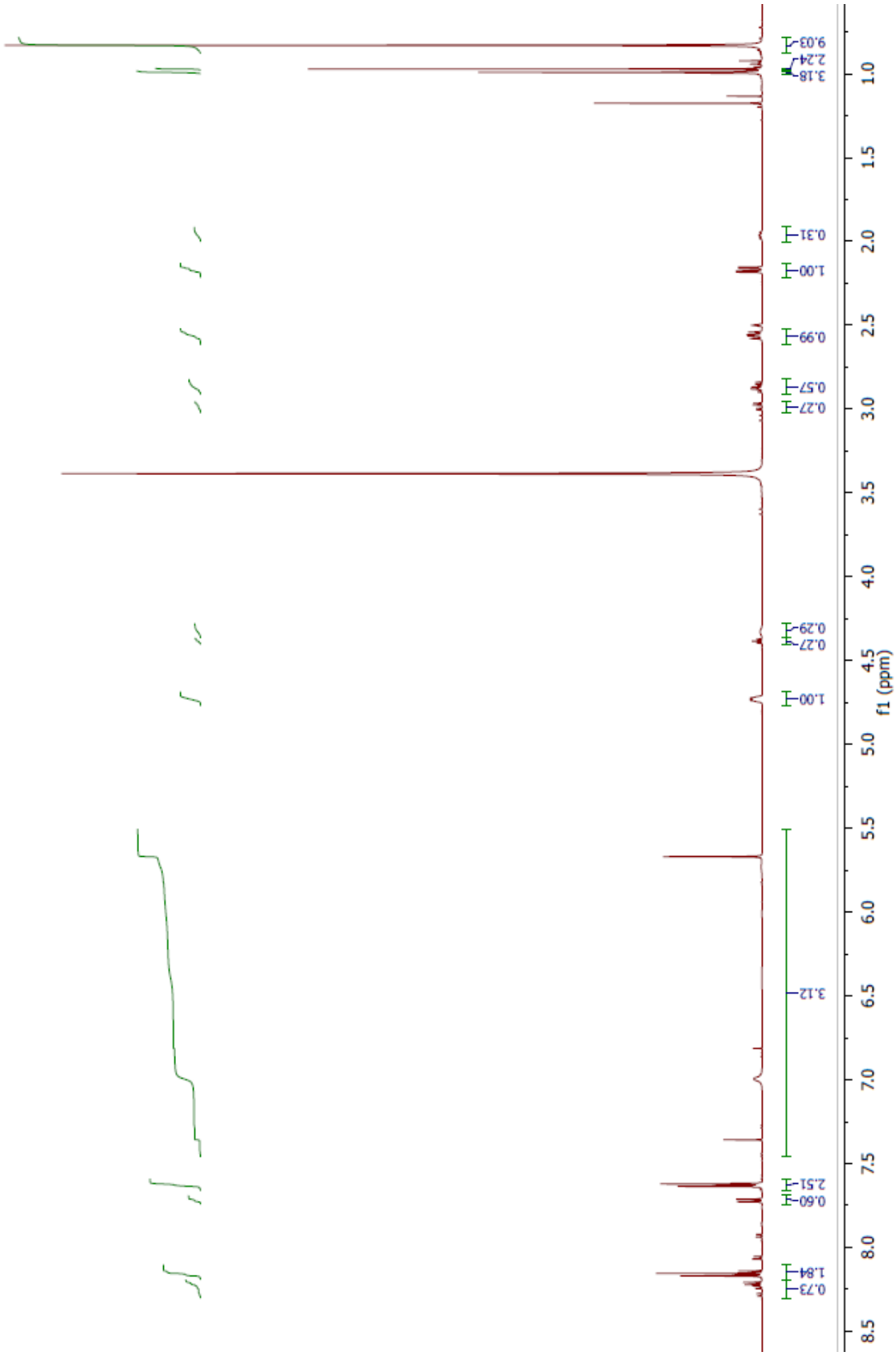

0.00
25.22
25.26
25.50
25.53
36.14
36.93
40.19
43.51
45.25
46.24
59.81
61.24
63.72
111.28
119.06
119.65
120.89
128.31
129.74
129.79
130.72
130.77
131.22
141.88
f1 (ppm)

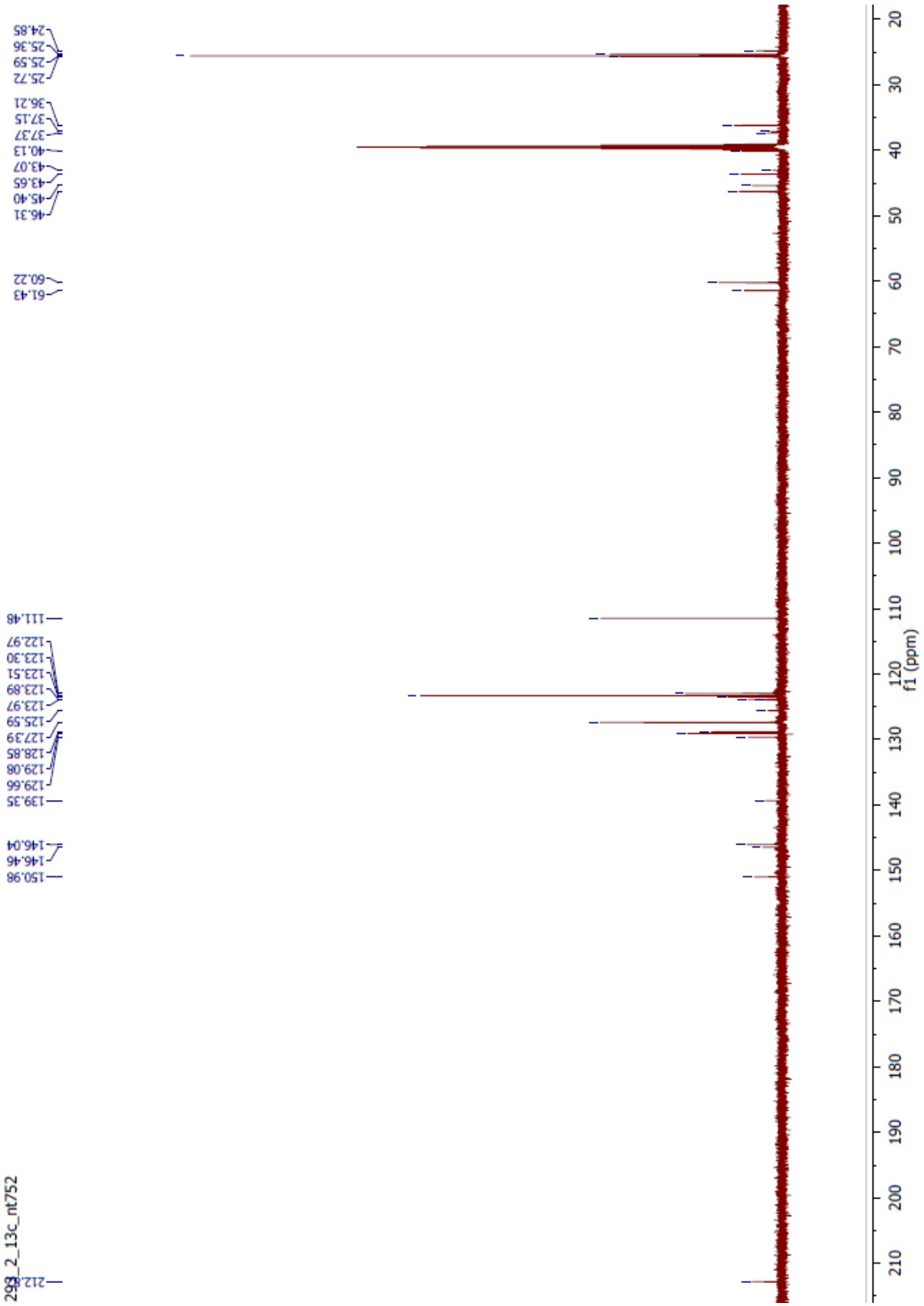
(4e) 5-tert-butylo-5-hydroksy-3-(4-chlorofenylo)izoksazolidina



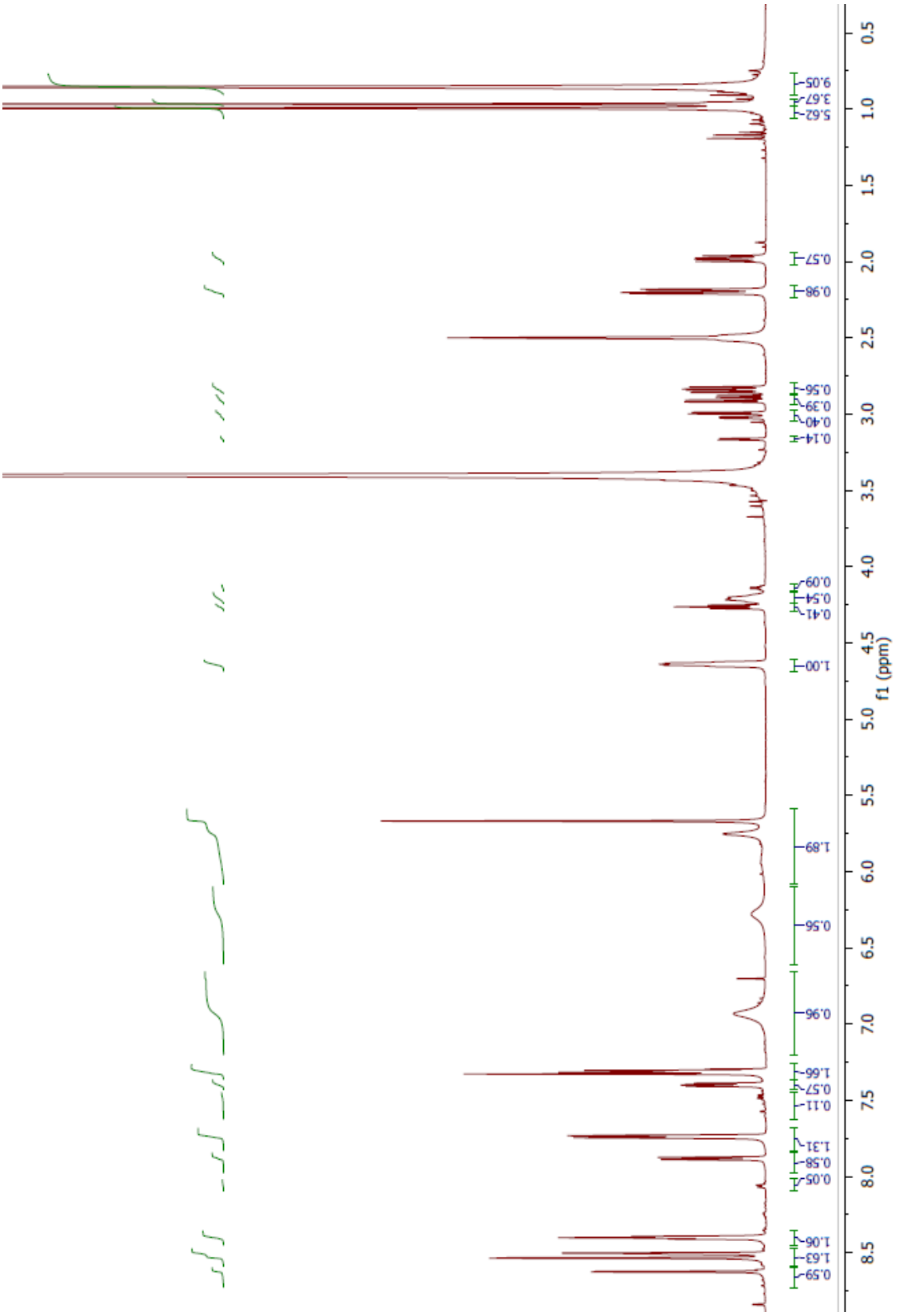

213.65
141.54
129.62
129.54
128.42
128.03
127.94
127.77
113.07
111.39
61.32
60.77
59.51
45.41
43.63
40.35
37.04
25.69
25.63
25.35
24.90
f1 (ppm)

(4f) 5-tert-butylo-5-hydroksy-3-(4-nitrofenylo)izoksazolidina

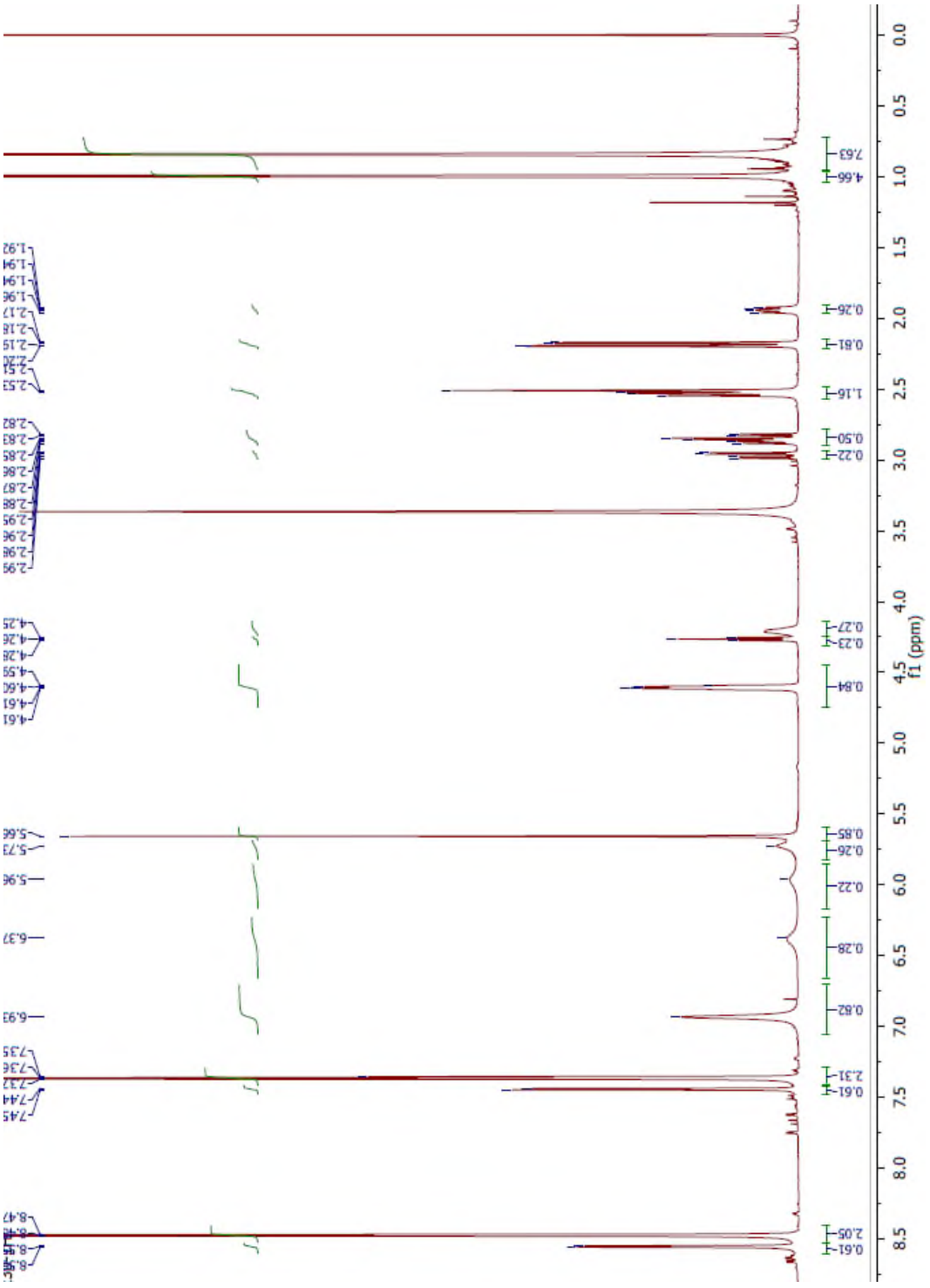



293_2_13c_nt752
212.83
150.98
146.46
146.04
139.35
129.66
129.08
128.85
127.39
125.59
123.97
123.89
123.51
123.30
122.97
111.48
61.43
60.22
46.31
45.40
43.65
43.07
40.13
37.37
37.15
36.21
25.72
25.59
25.36
24.85
f1 (ppm)

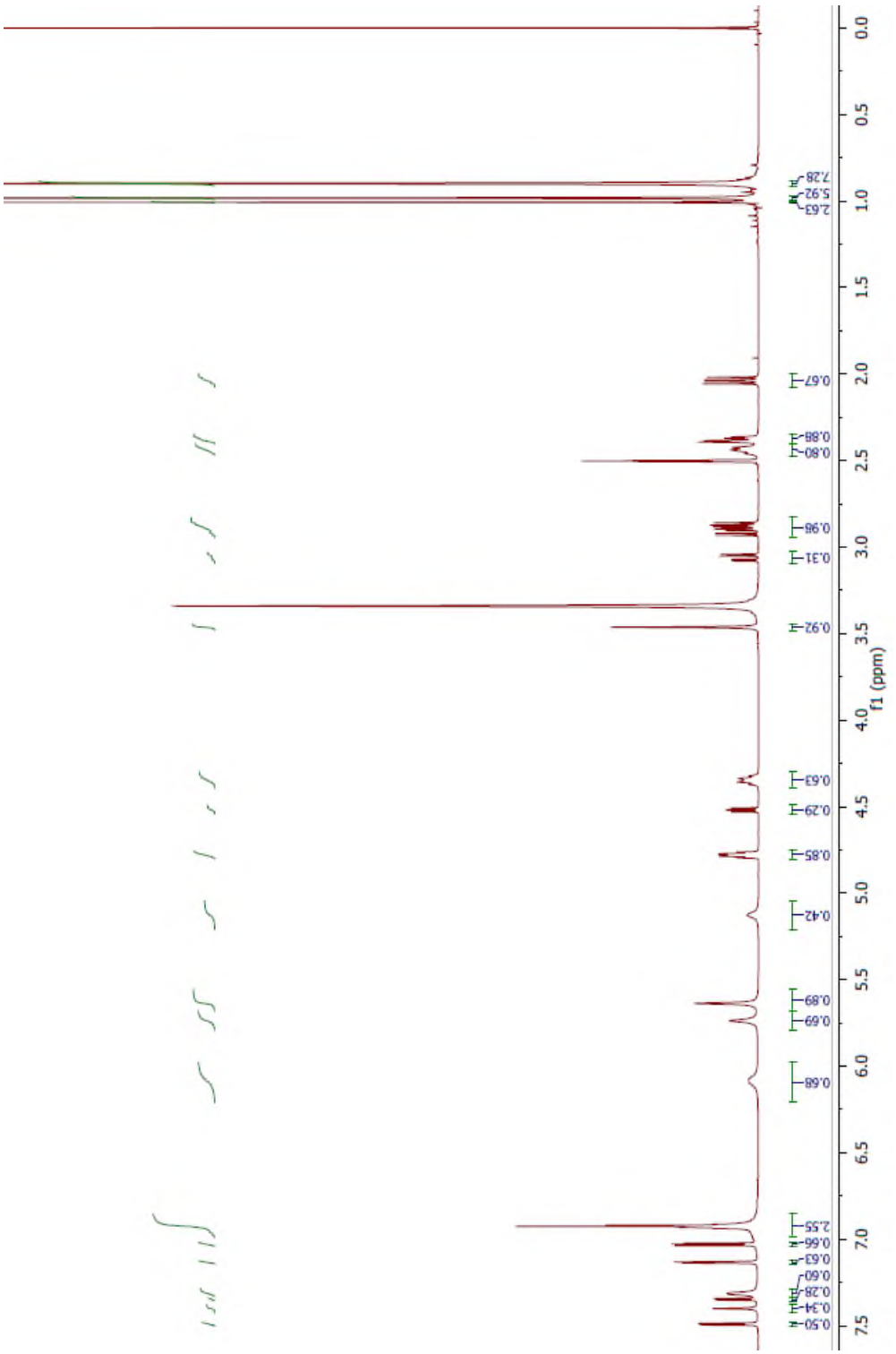
(4h) 5-tert-butylo-5-hydroksy-3-(3-pirydylo)izoksazolidina

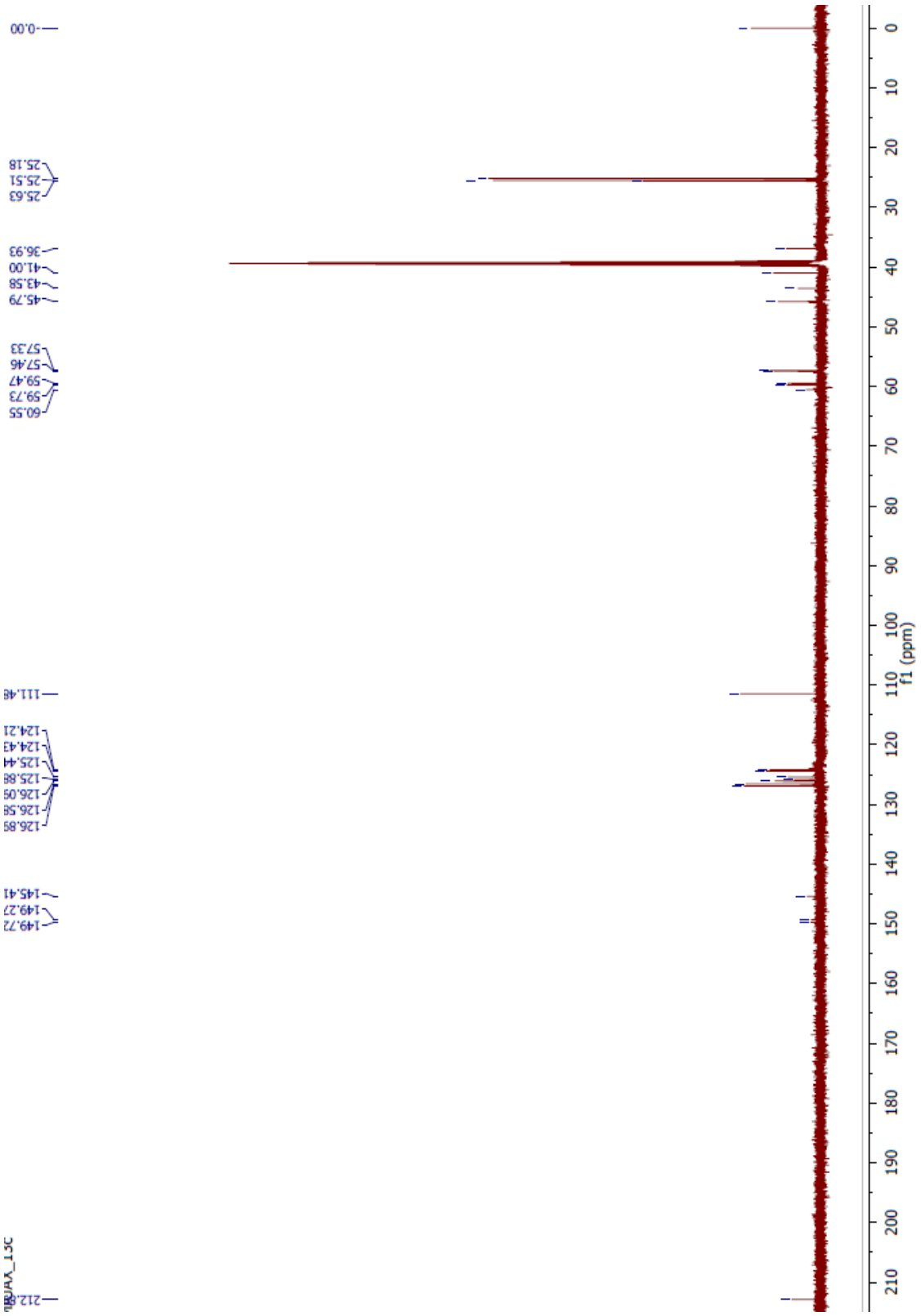


(4i) 5-tert-butylo-5-hydroksy-3-(4-pirydylo)izoksazolidina

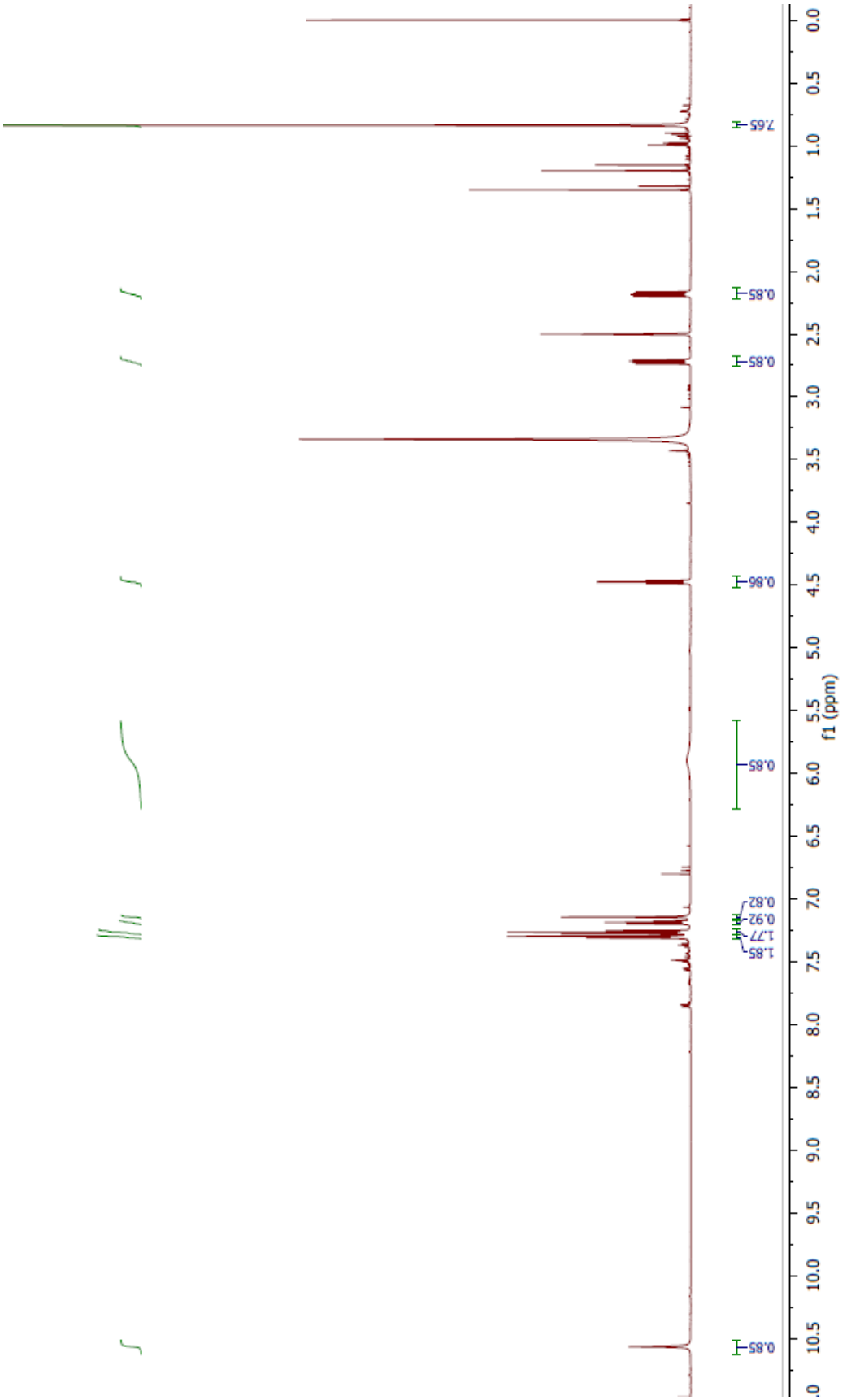


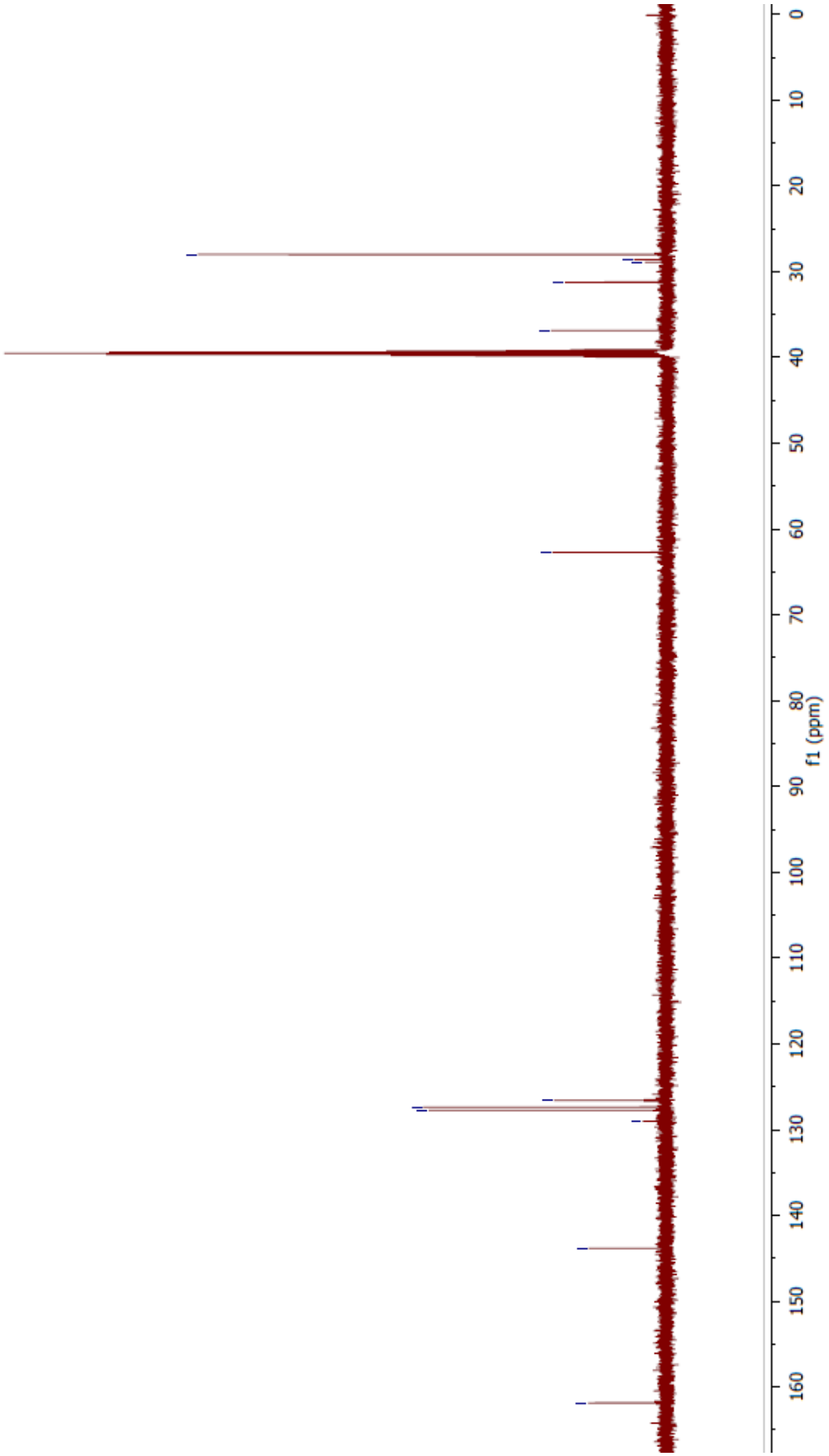
(4j) 5-tert-butylo-5-hydroksy-3-(2-tienylo)izoksazolidina



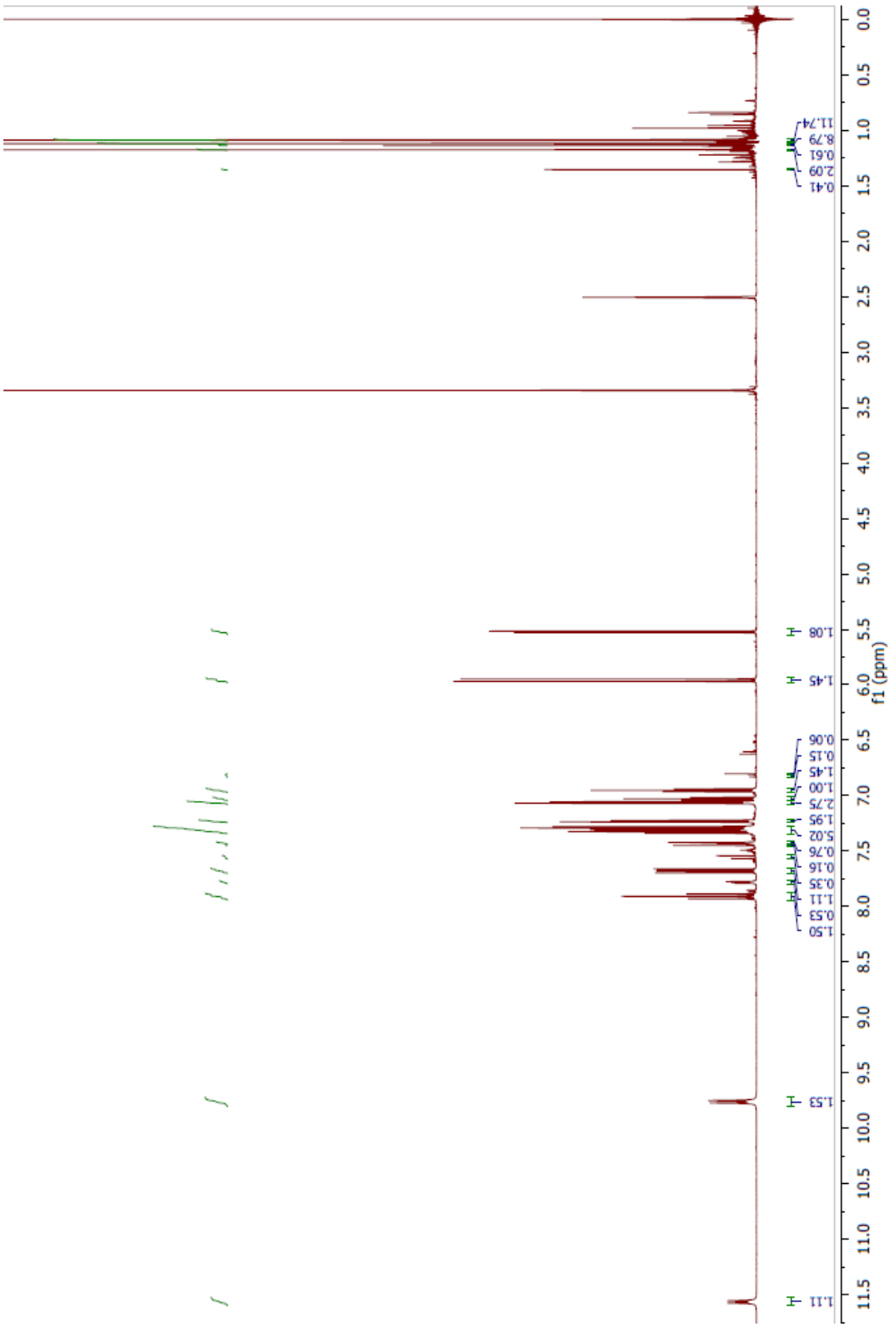

0.00
25.18
25.51
25.63
36.93
41.00
43.58
45.79
57.33
57.46
59.47
59.73
60.55
111.46
124.21
124.43
125.44
125.88
126.05
126.58
126.85
145.41
149.27
149.72
212.88
f1 (ppm)

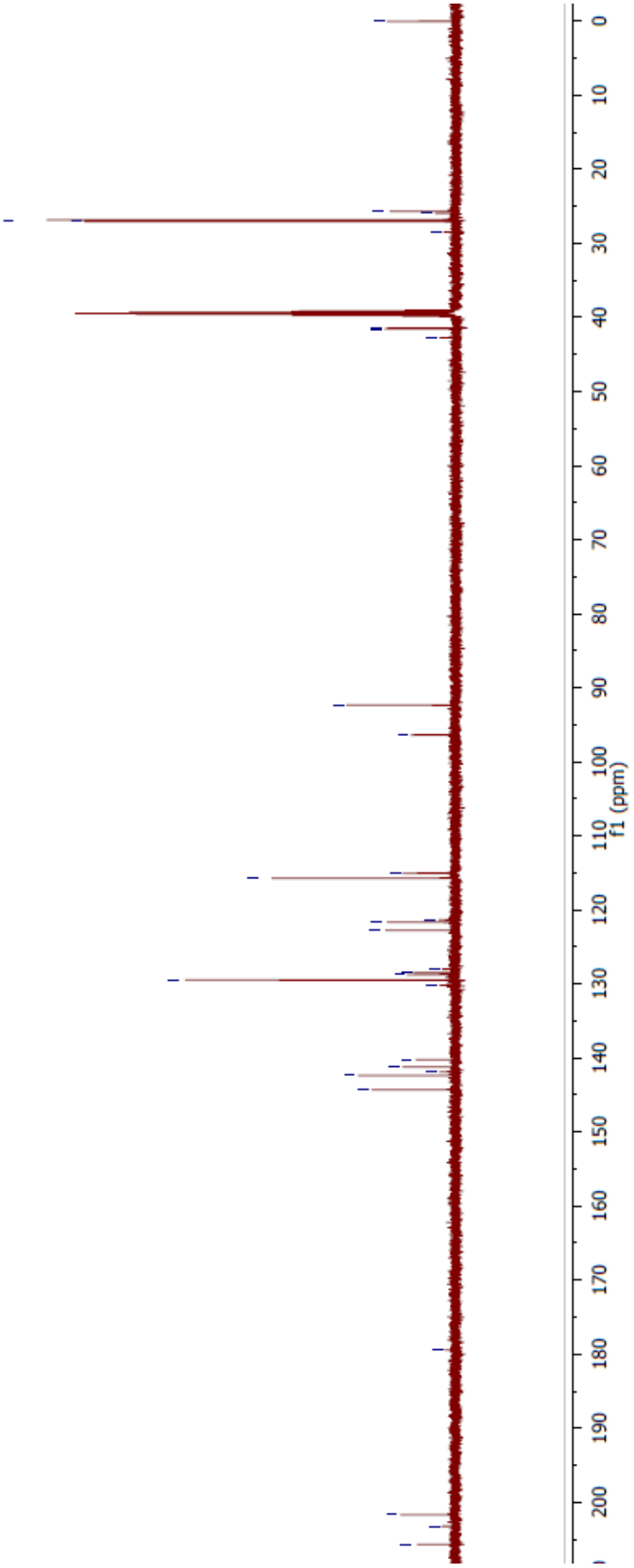
(6) oksym 5-hydroksyamino-2,2-dimetylo-5-fenylopentan-3-onu

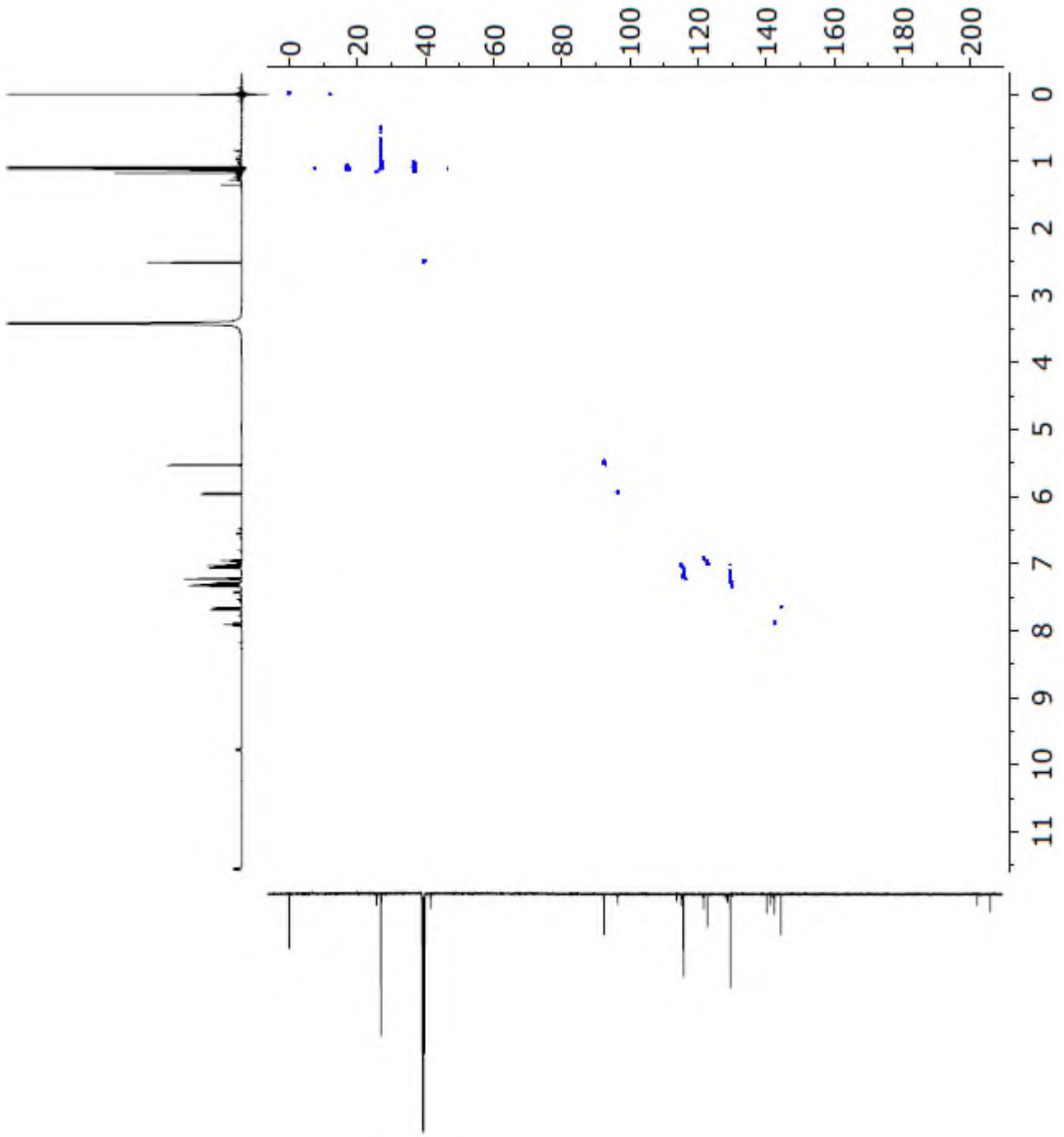


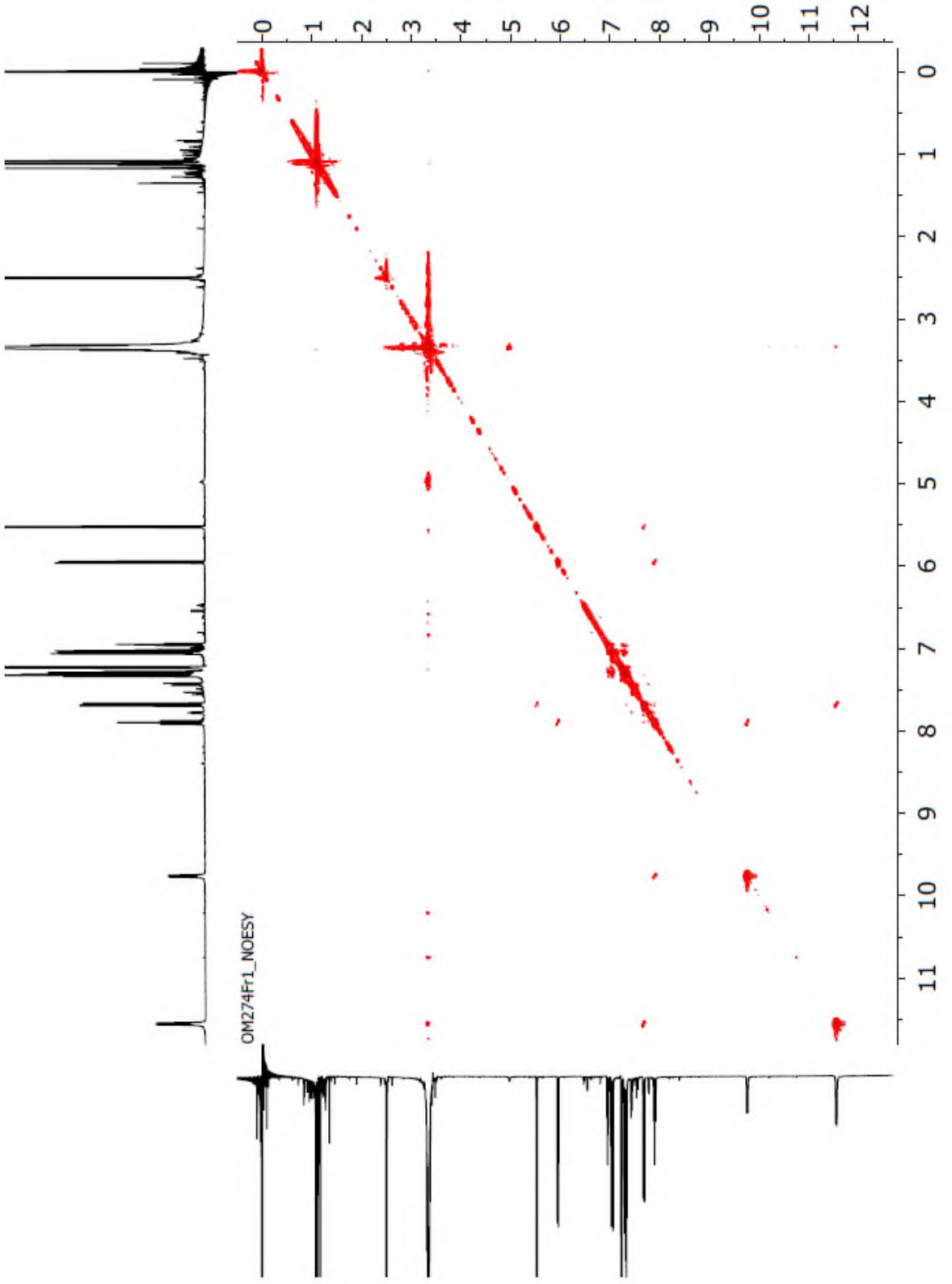

0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
f1 (ppm)

(7) 5-*tert*-butylo-3-fenylo-4-izoksazolidina

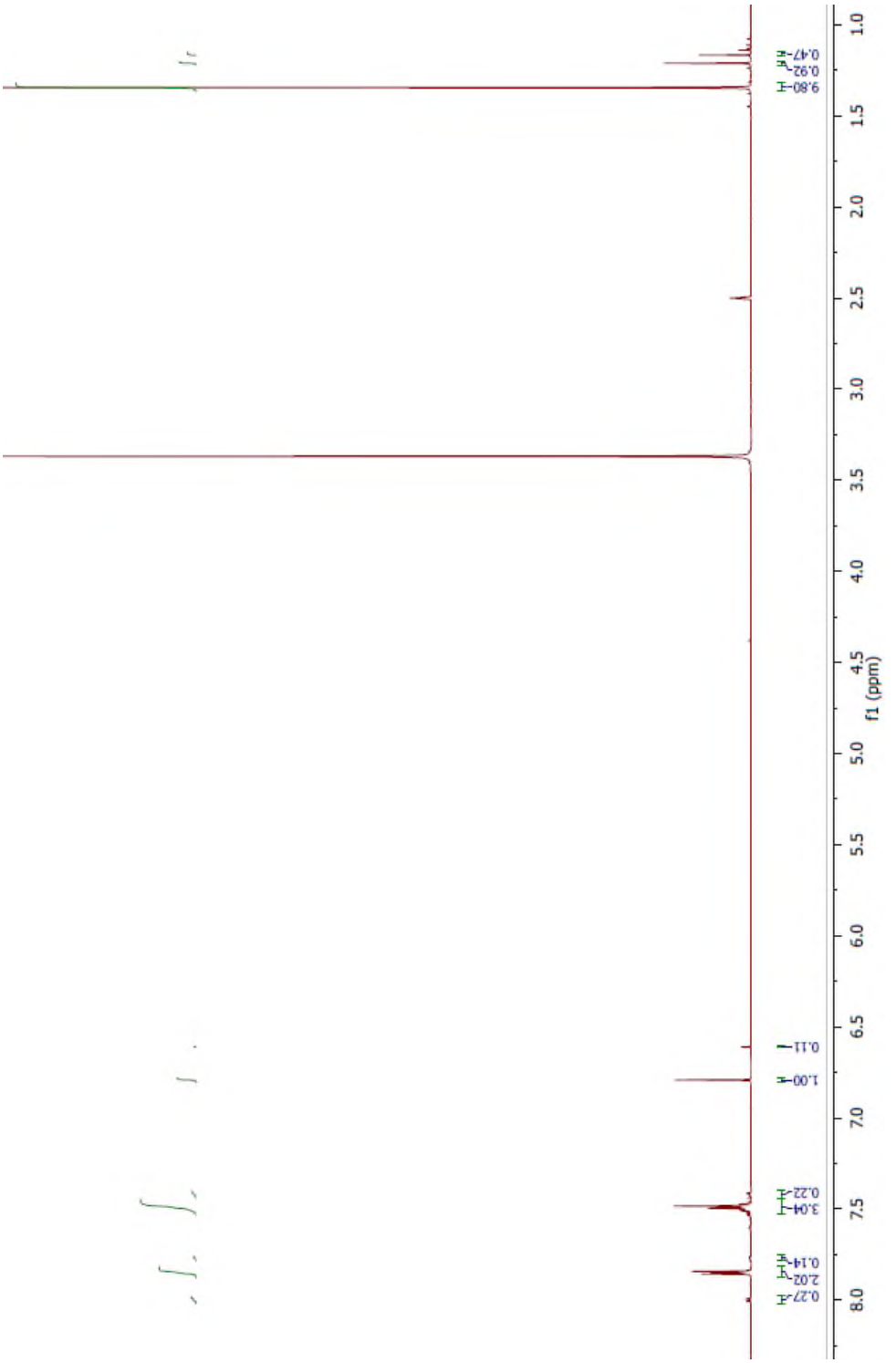


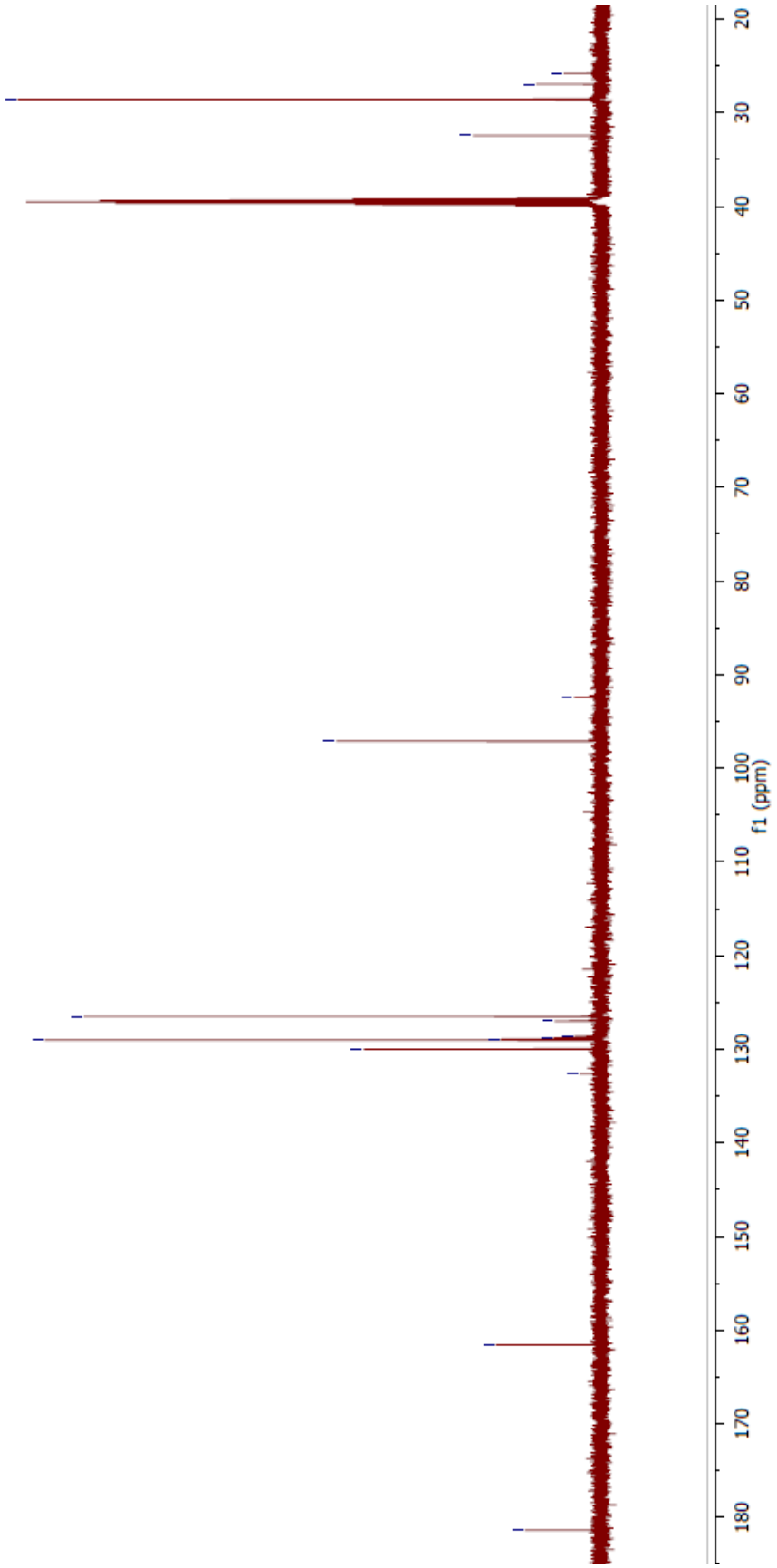

0
10
20
30
40
50
60
70
80
90
100
110
f1 (ppm)
120
130
140
150
160
170
180
190
200


0
20
40
60
80
100
120
140
160
180
200
0
1
2
3
4
5
6
7
8
9
10
11

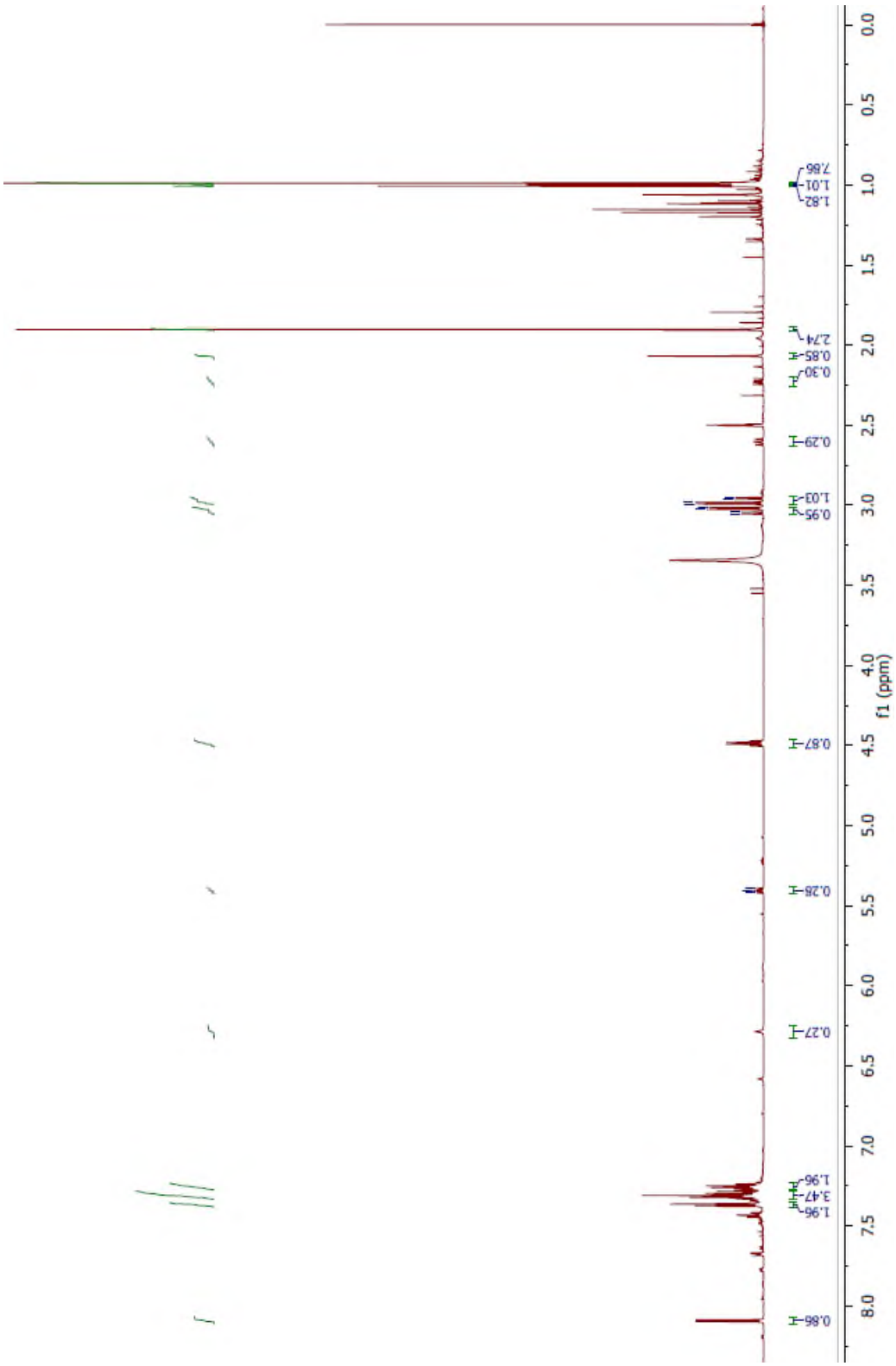

OM274Fr1_NOESY
0
1
2
3
4
5
6
7
8
9
10
11
12

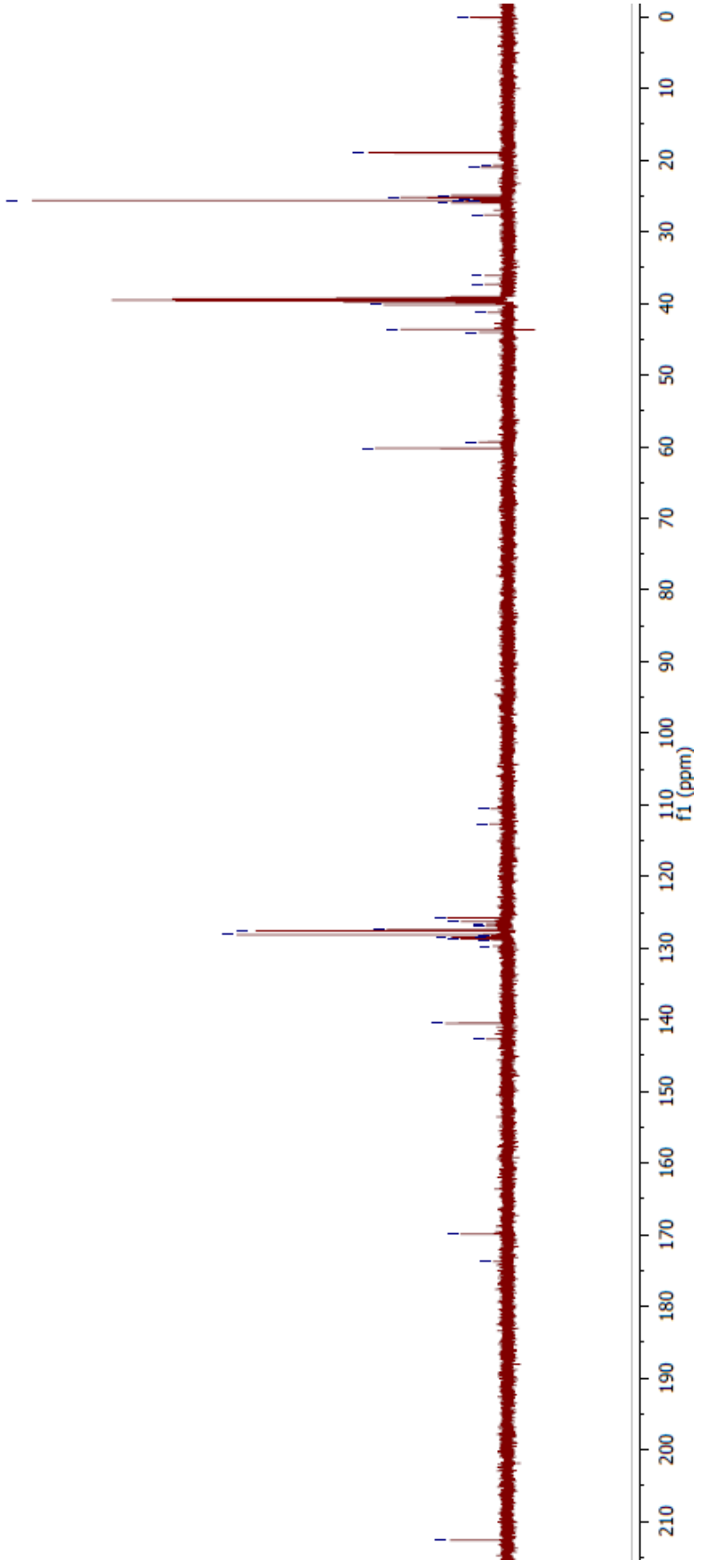
(8) 3(*5*)-fenylo-5(*3*)-*t*-butyloizoksazol




20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
f1 (ppm)

(9) 5-(*N*-acetoksy)amino-2,2-dimetylo-5-fenylopentan-3-on i 2-(*N*-acetoksy)-5-*tert*-butylo-5-hydroksy-3-fenyloizoksazolidina

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