

Supporting Information (2) for Total Synthesis of the Proposed Structure for Aromin and its Structural Revision

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Yamaguchi,[§] Takemichi Nakamura,[†] and Hiroyuki Koshino[†]

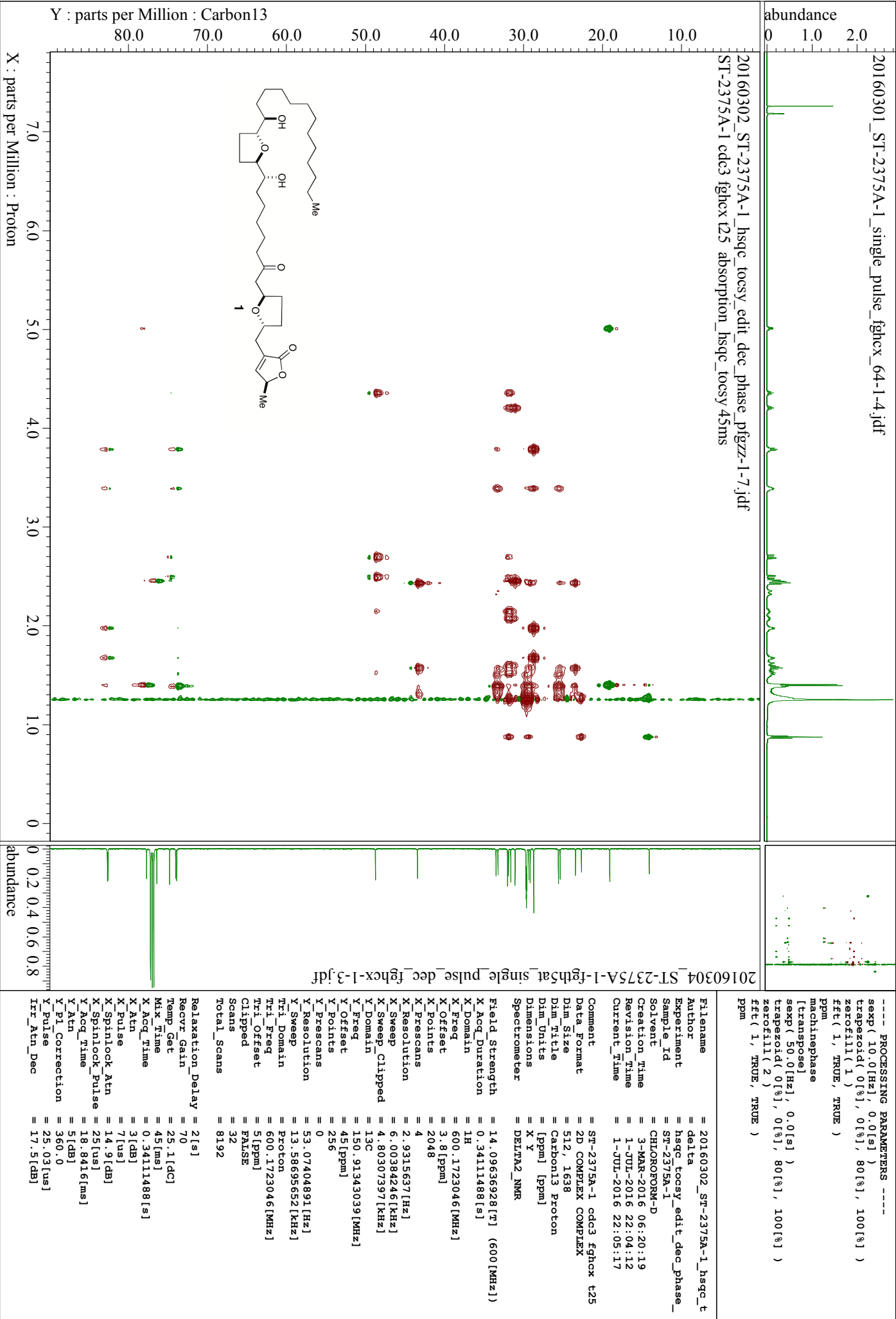
[†]*RIKEN Center for Sustainable Resource Science, Wako, Saitama 351-0198, Japan*

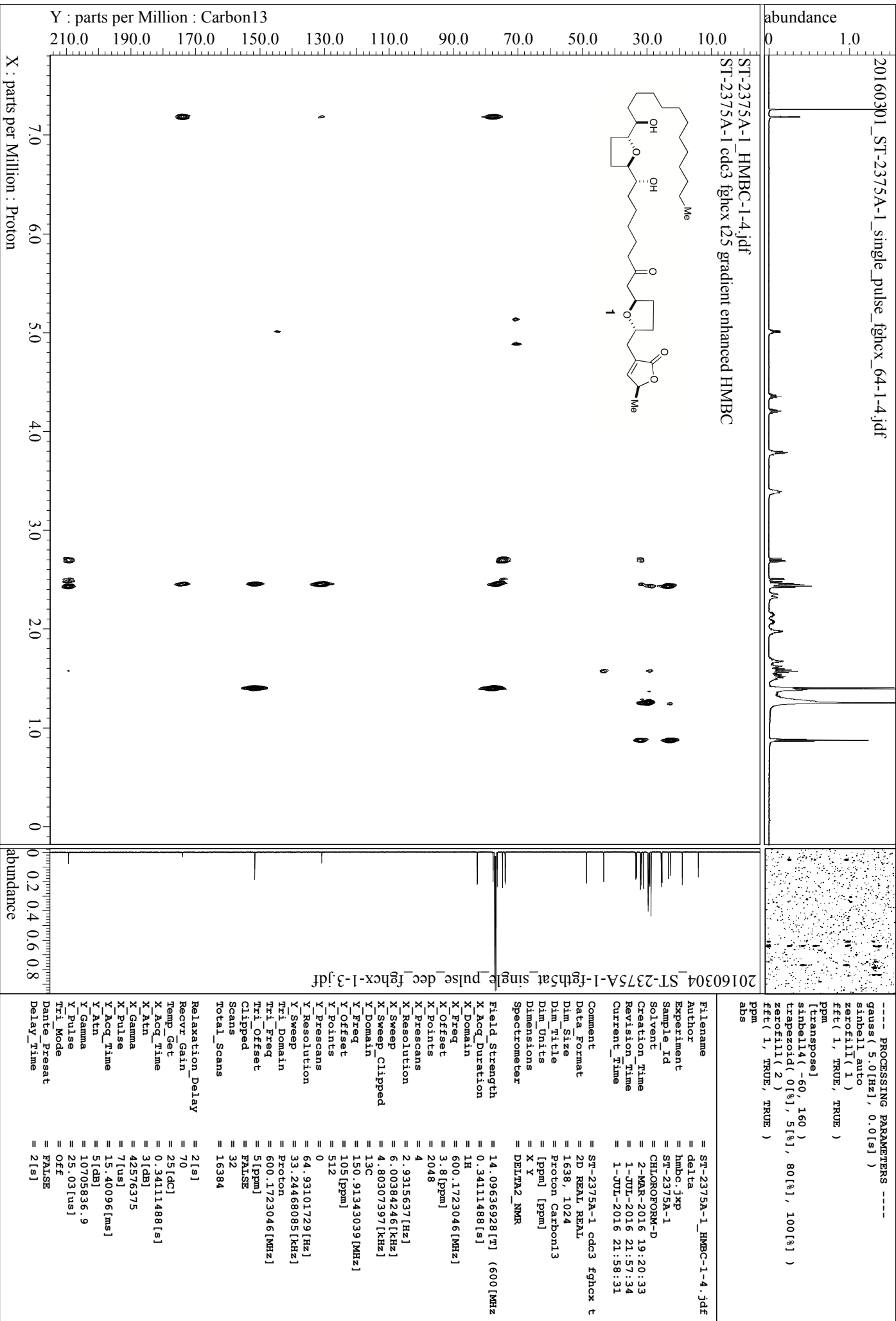
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Medical and Pharmaceutical University, Sendai 981-8558, Japan*

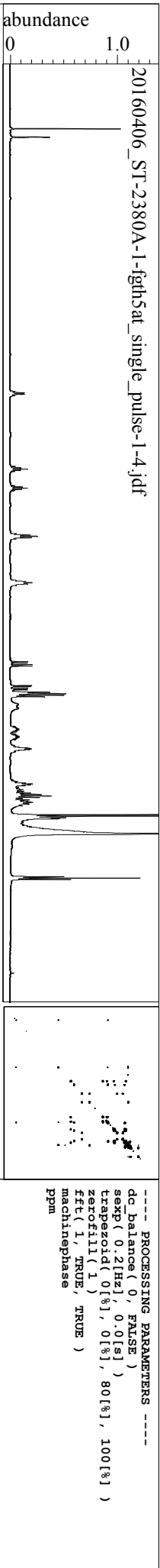
[§]*Division of Molecular and Cellular Oncology, Miyagi Cancer Center Research Institute,
Natori 981-1293, Japan*

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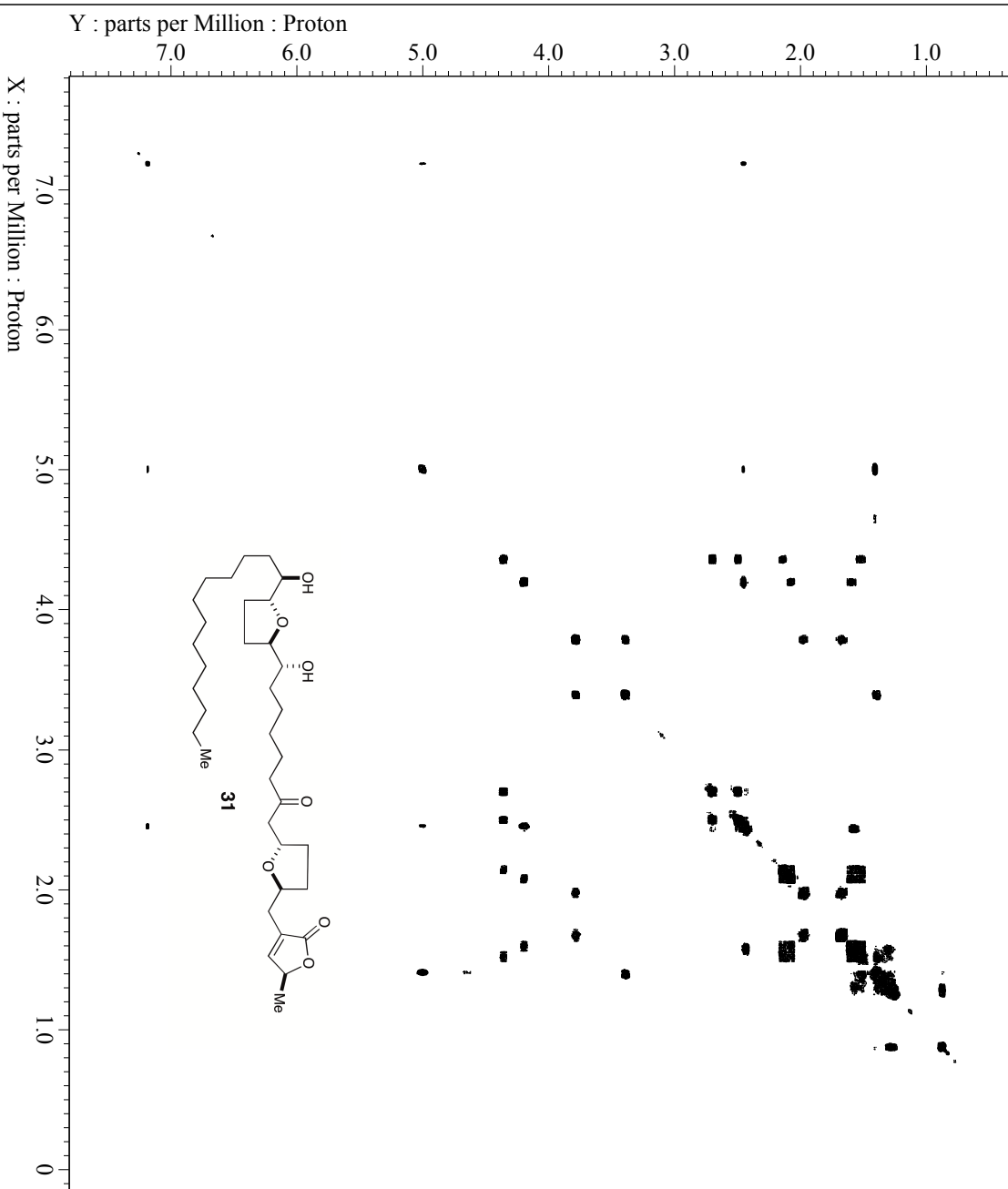
2D-NMR spectra of 1	S3
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20160406_ST-2380A-1-fgth5at_dqf_cosy_pfg-1-4.jdf
ST-2380A-1 cdc3 fgth5at t25 gradient absolute value DQF Cosy



----- PROCESSING PARAMETERS -----
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machinephase
ppm

20160406_ST-2380A-1-fgth5at_single_pulse-1-4.jdf

abundance

1.0

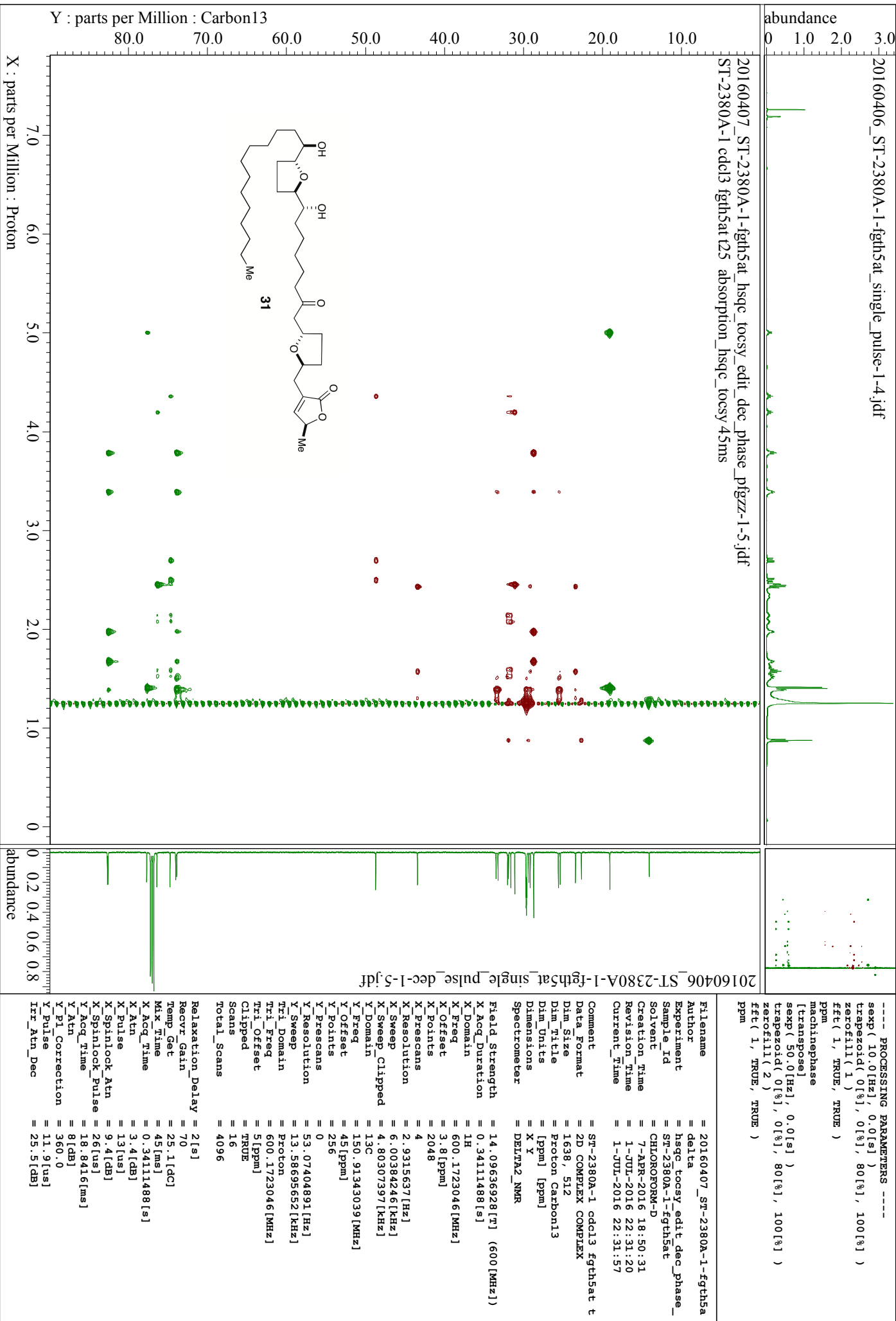
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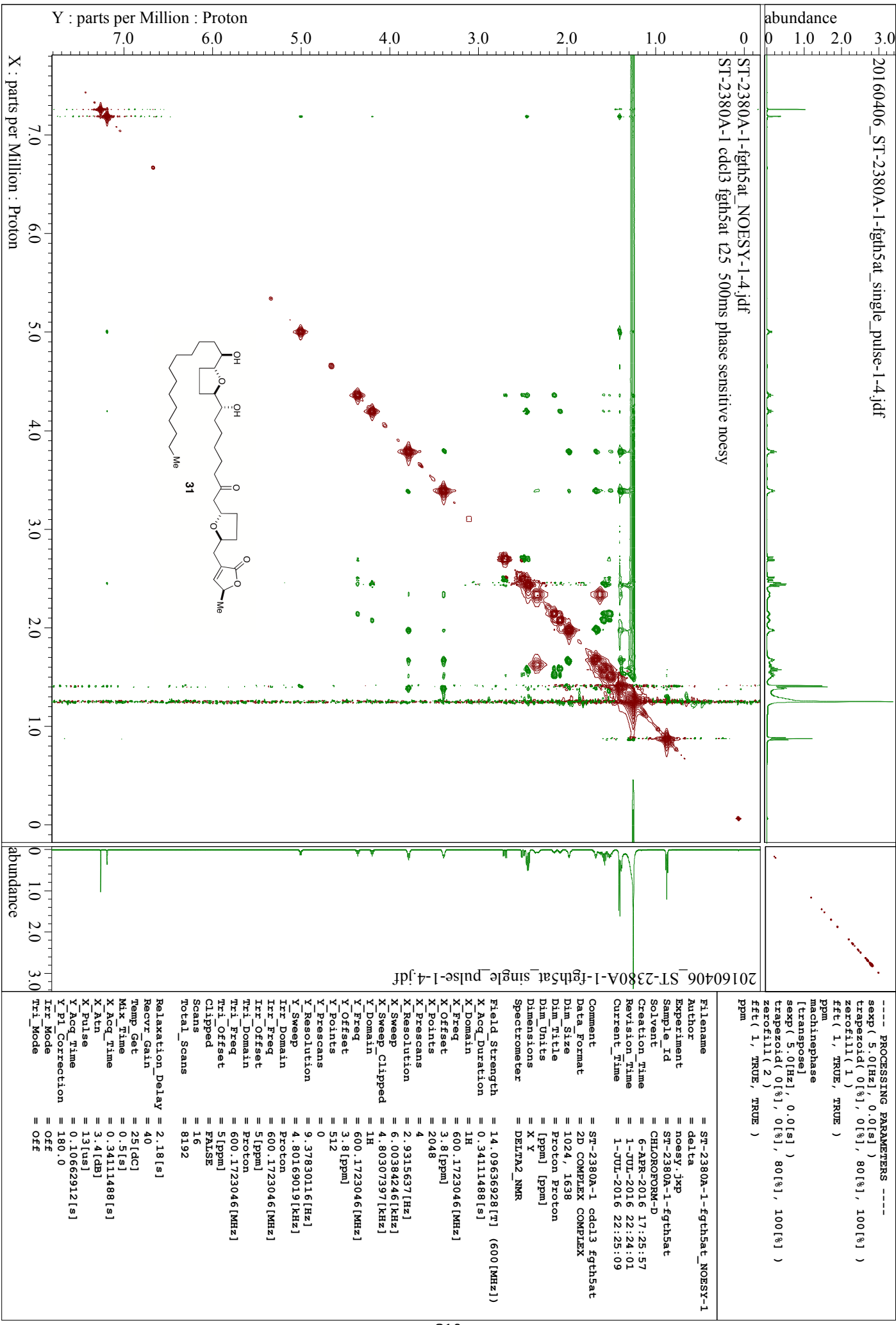
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Current_Time = 1-JUL-2016 22:21:35

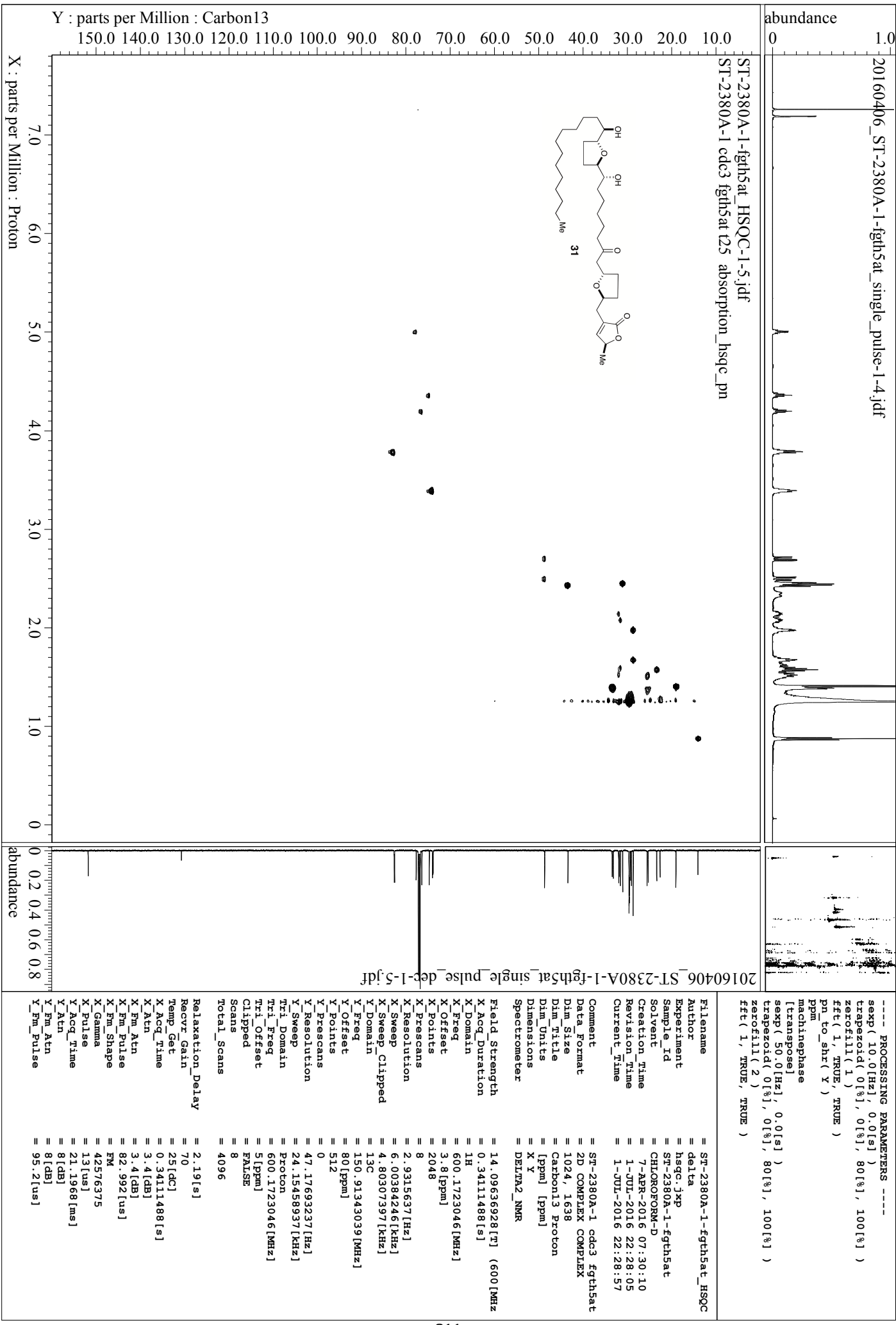
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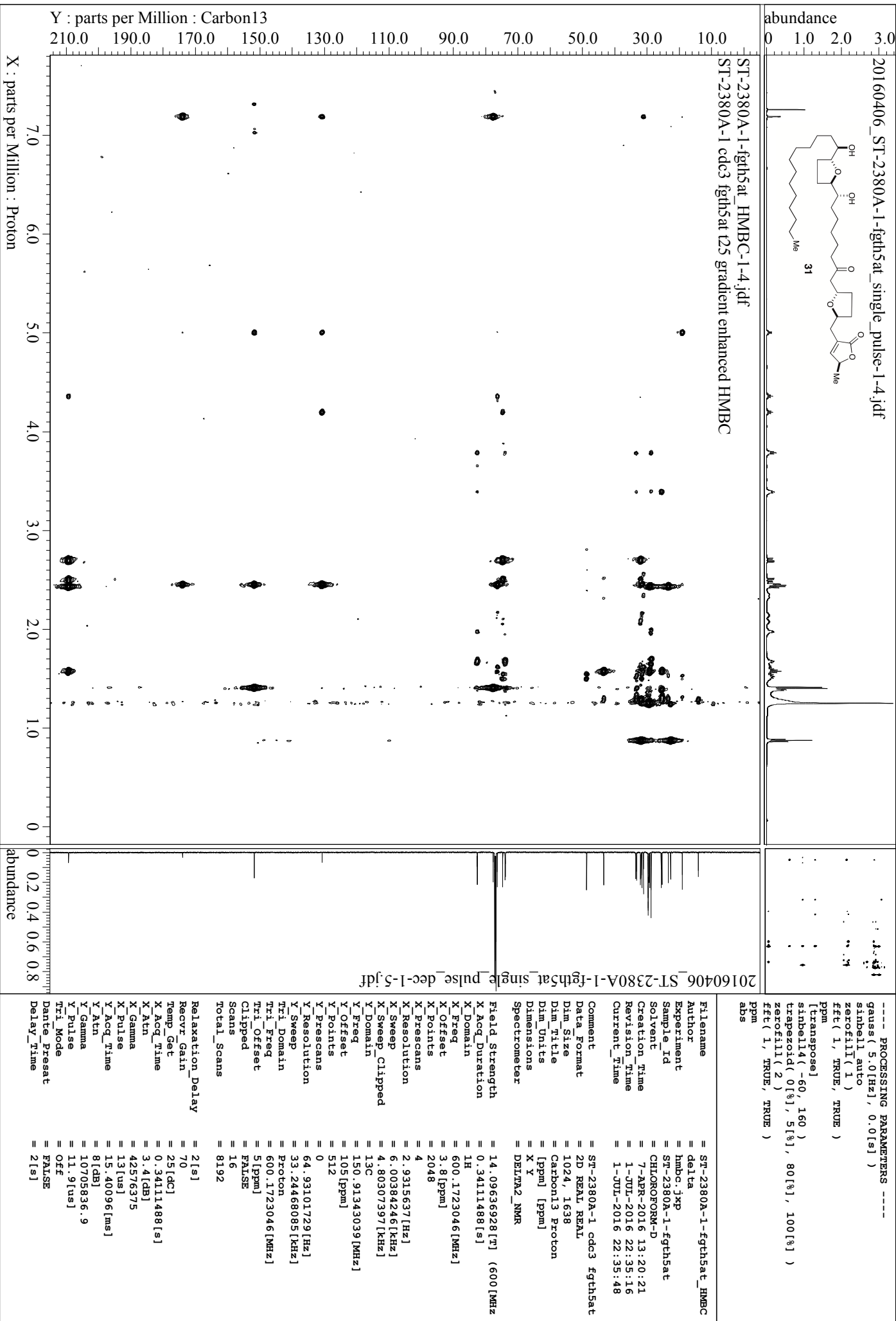
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X_Points = 2560
X_Prescans = 4
X_Resolution = 2.34525096 [Hz]
X_Sweep = 6.00384246 [kHz]
X_Sweep_Clippped = 4.80307397 [kHz]
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Y_Offset = 3.8 [ppm]
Y_Points = 512
Y_Prescans = 0
Y_Resolution = 9.37830116 [Hz]
Y_Sweep = 4.80169019 [kHz]
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Yr_Freq = 600.1723046 [MHz]
Yr_Offset = 5 [ppm]
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Yr_Freq = 600.1723046 [MHz]
Yr_Offset = 5 [ppm]
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Total_Scans = 4096

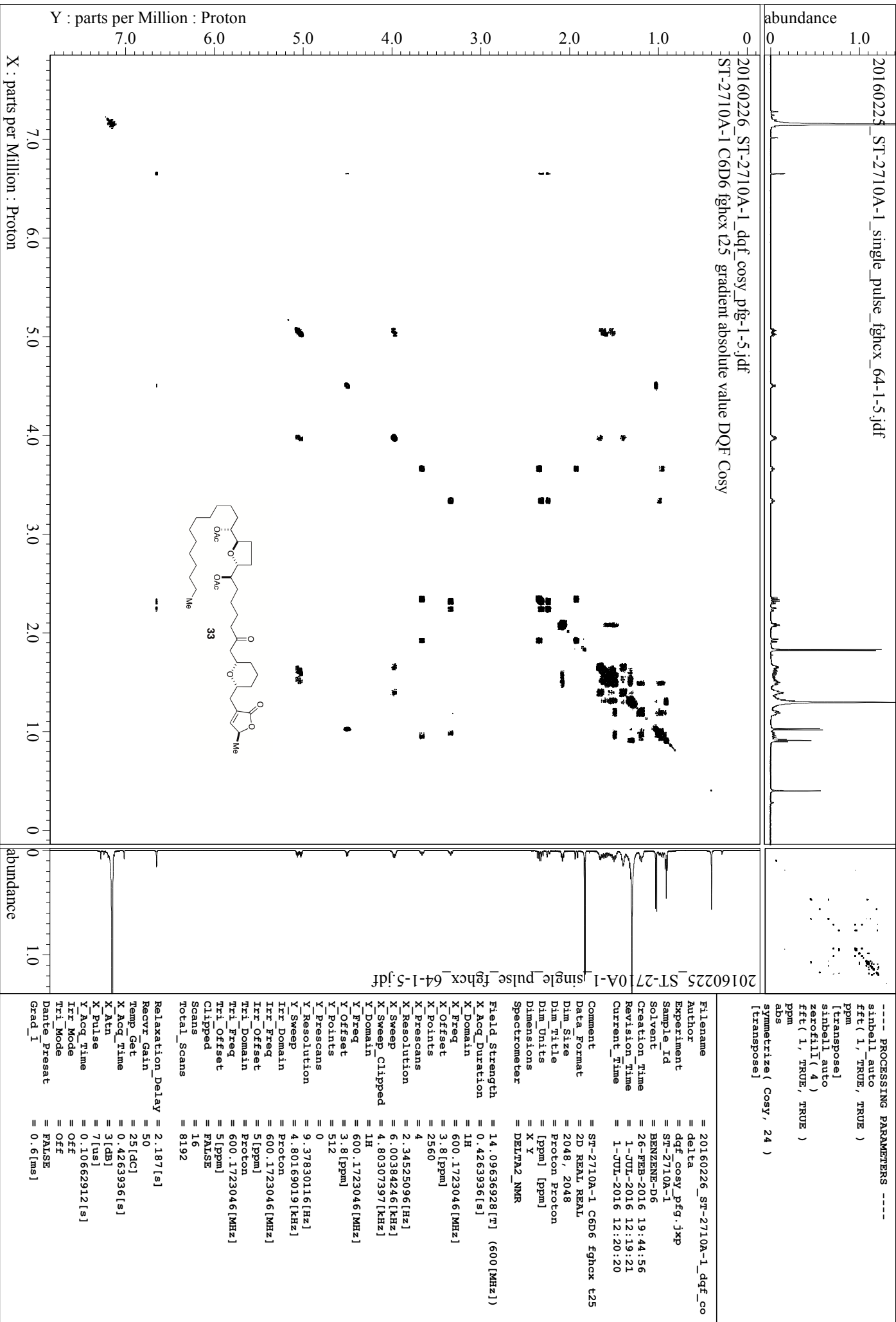
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X_Pulse = 13 [us]
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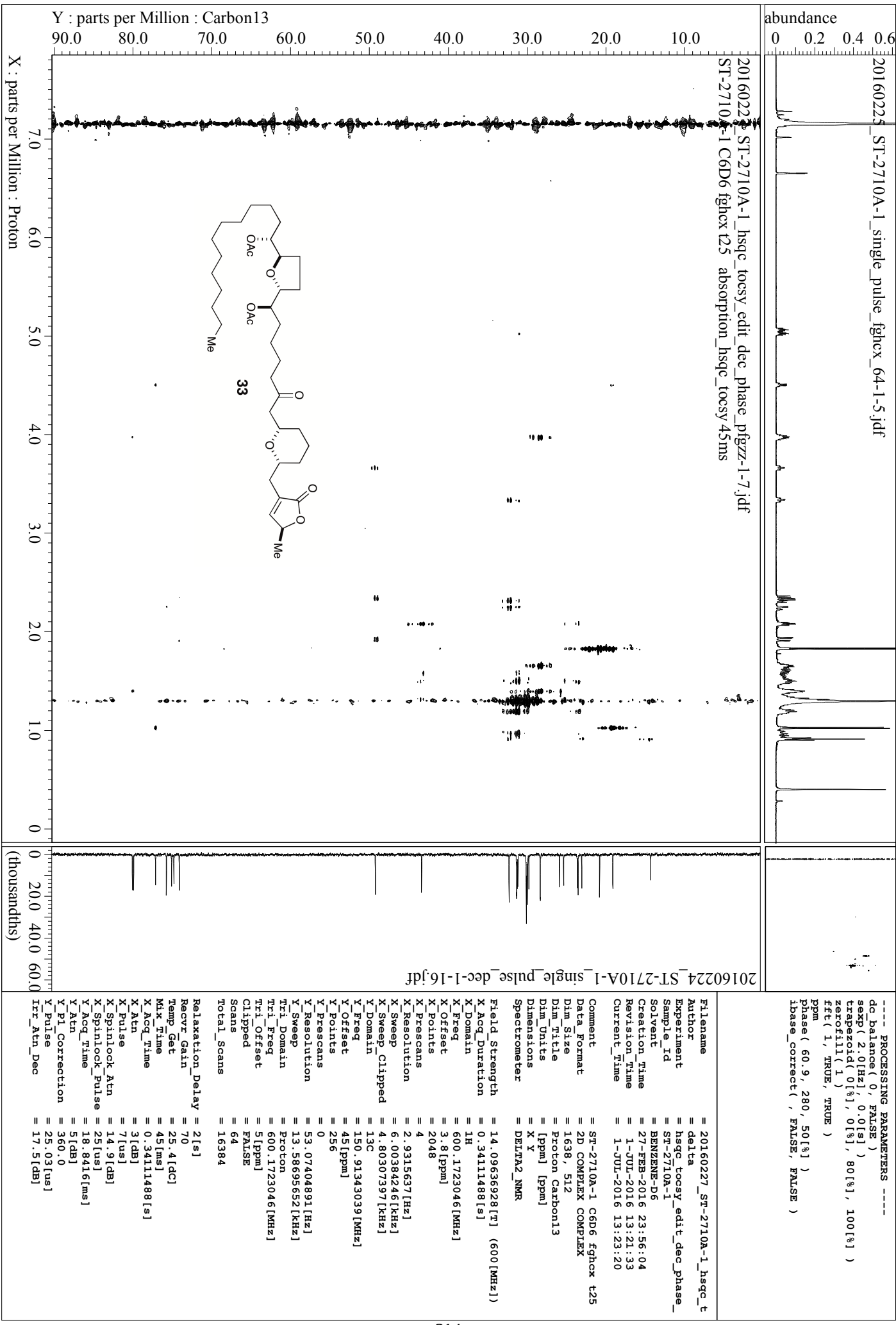




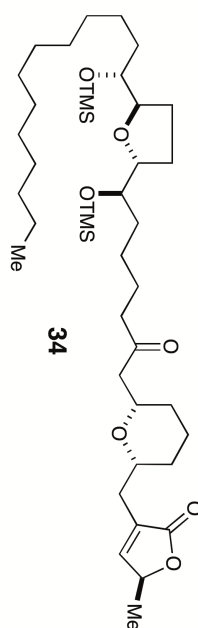
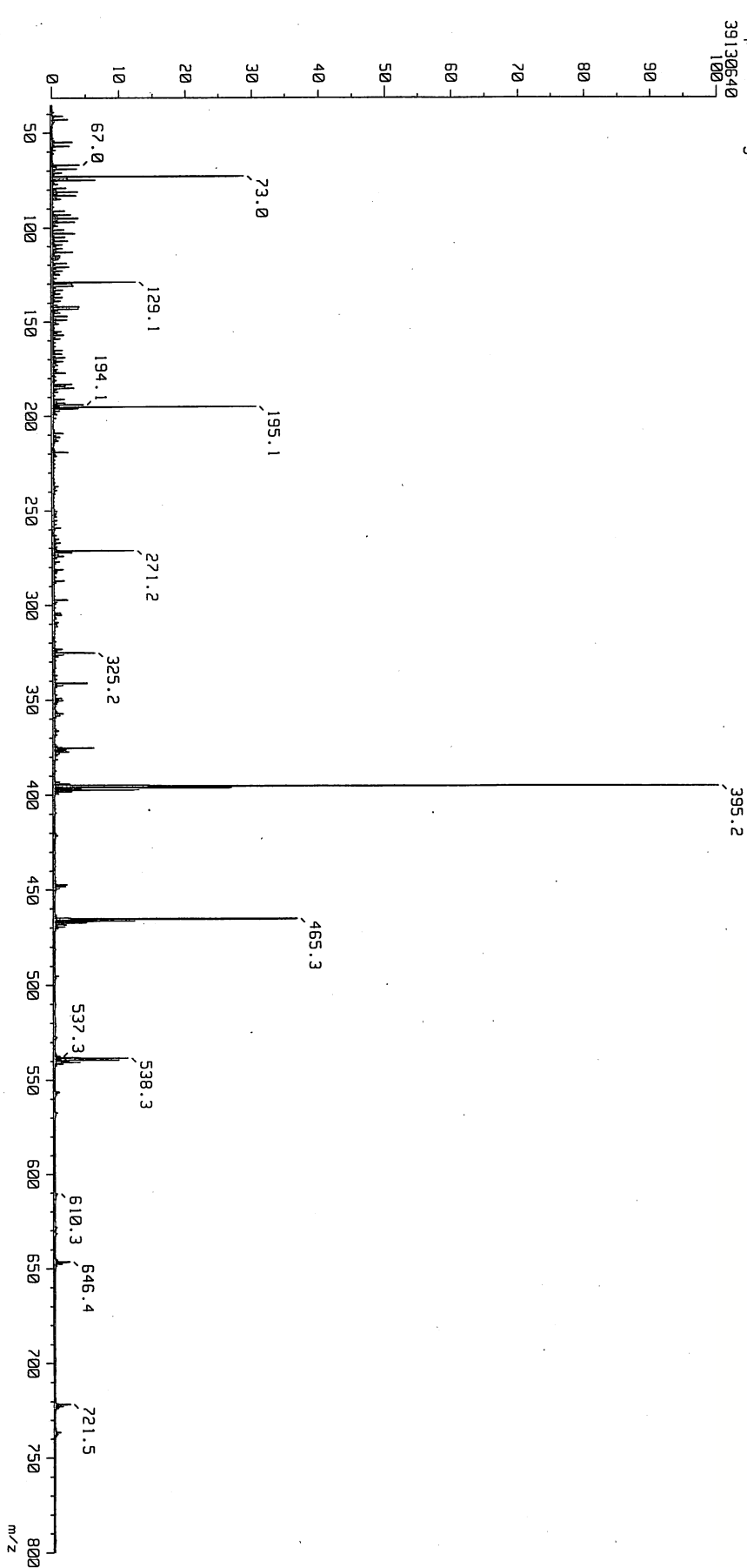








[Mass Spectrum]
 Date : 20160527 takahashi, tn005 Date : 27-May-2016 15:57
 Sample: ST-2711R-1 (in C6D6) PT:160 CT:200 PT:35 (1.2) ->128/m -> 350
 Note : DB-5MS, 122-5532, 30m x 0.25mm id, film 0.25um, ser# USR244957H (12/10/
 Inlet : Direct Ion Mode : EI+
 Spectrum Type : Normal Ion [MF-Linear]
 RT : 2.34 min Scan# : (70,72)
 BP : m/z 395.2137 Int. : 1243.93
 Output m/z range : 35.0000 to 800.0000 Cut Level : 0.00 %
 39130640



[Mass Spectrum]

Page: 1

Data : 20160527 takahashi tn005

Date : 27-May-2016 15:57

Sample: ST-2711A-1 (in C6D6) PT:160 CT:200 PT:35 (1.2) ->128/m -> 350

Note : DB-5MS,122-5532, 30m x 0.25mm id, film 0.25um, ser# USA244957H (12/10/

Inlet : Direct

Ion Mode : EI+

Spectrum Type : Normal Ion [MF-Linear]

RT : 2.34 min Scan# : (70,72)

BP : m/z 395.2137 Int. : 1243.93

Output m/z range : 35.0000 to 800.0000

Cut Level : 0.00 %

m/z	Int.	Norm.	m/z	Int.	Norm.
39.0211	4.28	0.34	109.0710	19.25	1.55
41.0376	21.13	1.70	110.0539	7.63	0.61
42.0370	4.04	0.32	111.0672	19.20	1.54
43.0351	30.24	2.43	112.0536	8.28	0.67
44.0072	4.99	0.40	113.0480	38.48	3.09
45.0051	3.89	0.31	114.0486	5.02	0.40
53.0287	3.34	0.27	115.0482	15.00	1.21
54.0355	5.05	0.41	116.0549	14.73	1.18
55.0292	38.70	3.11	117.0540	12.25	0.98
56.0378	5.53	0.44	119.0758	27.70	2.23
57.0458	32.91	2.65	120.0776	6.01	0.48
59.0141	7.52	0.60	121.0910	31.12	2.50
60.9948	3.27	0.26	122.0854	6.96	0.56
66.0378	3.20	0.26	123.0783	19.36	1.56
67.0493	51.77	4.16	124.0836	5.57	0.45
68.0527	9.02	0.73	125.0751	14.38	1.16
69.0658	46.53	3.74	127.0661	8.08	0.65
70.0586	6.48	0.52	128.0666	3.60	0.29
71.0621	18.99	1.53	129.0690	155.11	12.47
72.0379	5.40	0.43	130.0754	37.57	3.02
73.0327	358.15	28.79	131.0786	39.56	3.18
74.0300	29.80	2.40	132.0781	6.59	0.53
75.0080	80.54	6.47	133.0795	20.10	1.62
76.0051	5.64	0.45	134.0779	5.63	0.45
76.9983	11.50	0.92	135.0869	15.07	1.21
79.0246	26.60	2.14	136.0771	4.58	0.37
80.0311	8.74	0.70	137.0856	18.61	1.50
81.0270	47.82	3.84	138.0890	5.16	0.41
82.0360	11.14	0.90	139.0663	15.93	1.28
83.0435	45.38	3.65	140.0770	2.69	0.22
84.0390	9.96	0.80	141.0703	9.72	0.78
85.0461	16.73	1.34	142.0781	50.50	4.06
89.0240	3.56	0.29	143.0812	48.89	3.93
91.0454	23.64	1.90	144.0778	11.27	0.91
92.0531	3.75	0.30	145.0849	14.67	1.18
93.0675	34.65	2.79	146.0874	3.40	0.27
94.0568	10.38	0.83	147.0852	27.74	2.23
95.0662	48.96	3.94	148.0895	7.52	0.60
96.0701	10.32	0.83	149.0819	27.67	2.22
97.0776	42.68	3.43	150.0817	6.86	0.55
98.0752	6.77	0.54	151.0817	12.80	1.03
99.0550	10.60	0.85	152.1026	2.81	0.23
101.0388	22.21	1.79	153.0855	4.80	0.39
102.0372	2.69	0.22	154.0841	3.54	0.28
103.0430	42.57	3.42	155.0825	17.32	1.39
104.0452	4.93	0.40	156.0863	9.69	0.78
105.0522	24.83	2.00	157.0808	20.99	1.69
106.0579	4.95	0.40	158.0848	5.64	0.45
107.0605	29.19	2.35	159.0846	14.72	1.18
108.0599	5.79	0.47	160.0907	3.00	0.24

[Mass Spectrum]

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Data : 20160527 takahashi tn005

Date : 27-May-2016 15:57

Sample: ST-2711A-1 (in C6D6) PT:160 CT:200 PT:35 (1.2) ->128/m -> 350

Note : DB-5MS,122-5532, 30m x 0.25mm id, film 0.25um, ser# USA244957H (12/10/

Inlet : Direct

Ion Mode : EI+

Spectrum Type : Normal Ion [MF-Linear]

RT : 2.34 min Scan# : (70,72)

BP : m/z 395.2137 Int. : 1243.93

Output m/z range : 35.0000 to 800.0000

Cut Level : 0.00 %

m/z	Int.	Norm.	m/z	Int.	Norm.
161.0957	5.98	0.48	219.1026	28.57	2.30
163.0982	6.11	0.49	220.1142	4.96	0.40
164.1009	2.67	0.21	221.1170	6.99	0.56
165.0959	18.94	1.52	223.1296	3.88	0.31
166.1025	4.77	0.38	225.1414	4.54	0.37
167.0939	17.54	1.41	227.1411	4.55	0.37
168.0914	6.04	0.49	229.1360	2.62	0.21
169.0840	23.69	1.90	233.1821	3.07	0.25
170.0884	7.41	0.60	235.1484	4.15	0.33
171.0914	20.34	1.64	237.1254	10.68	0.86
172.0951	7.06	0.57	238.1347	4.18	0.34
173.0996	10.72	0.86	239.1589	7.80	0.63
174.1004	3.88	0.31	240.1484	3.17	0.26
175.1008	9.44	0.76	241.1539	4.48	0.36
176.0886	7.13	0.57	247.1545	2.81	0.23
177.0903	24.68	1.98	249.1837	3.51	0.28
178.0982	4.11	0.33	250.2007	7.33	0.59
179.1100	5.60	0.45	251.2068	7.08	0.57
180.1168	3.58	0.29	252.1460	6.16	0.50
181.0982	7.62	0.61	253.1612	6.46	0.52
182.1010	3.86	0.31	255.1804	7.13	0.57
183.0908	36.73	2.95	256.1839	2.86	0.23
184.0923	24.07	1.94	257.1737	6.62	0.53
185.0999	40.00	3.22	259.1603	14.87	1.20
186.1000	8.08	0.65	260.1584	3.71	0.30
187.1086	10.24	0.82	263.1936	6.49	0.52
189.1053	4.50	0.36	264.1795	3.28	0.26
190.1014	3.06	0.25	265.1460	10.53	0.85
191.1012	22.38	1.80	266.1409	5.02	0.40
192.1071	8.52	0.69	267.1483	14.32	1.15
193.1038	24.20	1.95	268.1483	4.18	0.34
194.0929	57.06	4.59	269.1740	7.88	0.63
195.0972	379.38	30.50	270.2133	6.11	0.49
196.1006	47.61	3.83	271.2399	149.61	12.03
197.1217	13.70	1.10	272.2401	34.29	2.76
198.1117	6.00	0.48	273.2260	9.87	0.79
199.1158	8.23	0.66	274.1541	19.82	1.59
201.1047	6.64	0.53	275.1623	6.25	0.50
203.1117	2.81	0.23	277.1708	12.20	0.98
207.1109	3.65	0.29	278.1669	4.68	0.38
208.1159	4.47	0.36	279.1703	2.98	0.24
209.1126	20.08	1.61	281.1587	18.70	1.50
210.1165	6.78	0.54	282.1602	8.16	0.66
211.1227	13.42	1.08	283.1717	6.33	0.51
212.1169	7.50	0.60	285.1986	5.33	0.43
213.1135	10.38	0.83	286.1797	3.19	0.26
214.1209	3.53	0.28	287.1707	20.62	1.66
215.1234	2.88	0.23	288.1745	5.42	0.44
217.1134	3.33	0.27	297.2470	27.02	2.17
218.1095	4.23	0.34	298.2456	7.13	0.57

[Mass Spectrum]

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Data : 20160527 takahashi tn005 Date : 27-May-2016 15:57
Sample: ST-2711A-1 (in C6D6) PT:160 CT:200 PT:35 (1.2) ->128/m -> 350
Note : DB-5MS,122-5532, 30m x 0.25mm id, film 0.25um, ser# USA244957H (12/10/
Inlet : Direct Ion Mode : EI+
Spectrum Type : Normal Ion [MF-Linear]
RT : 2.34 min Scan# : (70,72)
BP : m/z 395.2137 Int. : 1243.93
Output m/z range : 35.0000 to 800.0000 Cut Level : 0.00 %

m/z	Int.	Norm.	m/z	Int.	Norm.
299.2247	4.06	0.33	374.1978	6.04	0.49
301.2513	3.88	0.31	375.2071	74.43	5.98
303.2111	2.79	0.22	376.2114	22.44	1.80
304.1657	14.16	1.14	377.2061	27.84	2.24
305.1726	15.08	1.21	378.2116	10.01	0.80
306.1771	4.51	0.36	379.2184	5.96	0.48
307.1819	5.05	0.41	380.2360	2.72	0.22
309.1619	8.67	0.70	381.2183	6.44	0.52
310.2202	6.81	0.55	387.2176	3.64	0.29
311.2186	8.23	0.66	393.2107	10.51	0.85
312.2230	2.76	0.22	394.2140	29.76	2.39
313.2303	3.17	0.25	395.2137	1243.93	100.00
315.1865	2.83	0.23	396.2147	331.33	26.64
319.2546	3.83	0.31	397.2202	157.74	12.68
321.1887	2.96	0.24	398.2237	33.61	2.70
323.1899	16.38	1.32	399.2382	8.43	0.68
324.1855	6.44	0.52	407.2435	2.94	0.24
325.1754	76.18	6.12	409.2489	4.22	0.34
326.1799	18.42	1.48	420.2584	3.27	0.26
327.1977	8.11	0.65	421.2471	6.48	0.52
329.2411	3.73	0.30	422.2485	2.86	0.23
332.1949	2.74	0.22	437.3004	3.03	0.24
333.1968	4.21	0.34	445.3035	2.58	0.21
337.1987	5.95	0.48	447.2450	24.01	1.93
338.2022	2.80	0.23	448.2496	21.14	1.70
339.2041	5.06	0.41	449.2502	9.59	0.77
340.2180	2.73	0.22	450.2571	3.19	0.26
341.2769	61.95	4.98	463.2509	3.60	0.29
342.2774	17.14	1.38	464.2568	4.62	0.37
343.2670	5.50	0.44	465.2552	454.08	36.50
345.2440	2.81	0.23	466.2581	150.13	12.07
347.2613	5.72	0.46	467.2531	60.28	4.85
348.2359	2.97	0.24	468.2569	22.16	1.78
349.2134	14.23	1.14	469.2653	20.03	1.61
350.1893	17.27	1.39	470.2644	7.06	0.57
351.1968	7.50	0.60	471.2767	2.80	0.23
352.2073	5.33	0.43	481.2854	2.82	0.23
353.2406	3.58	0.29	495.2877	7.76	0.62
355.2381	3.87	0.31	496.2983	3.16	0.25
356.2012	6.09	0.49	510.3277	2.65	0.21
357.1992	17.54	1.41	520.3057	3.31	0.27
358.2048	11.39	0.92	521.3436	3.02	0.24
359.2087	3.94	0.32	527.3783	4.97	0.40
363.2275	2.65	0.21	528.3829	2.66	0.21
365.2322	4.16	0.33	535.3899	2.78	0.22
366.2165	8.58	0.69	536.3292	3.42	0.28
367.2390	3.92	0.32	537.3082	10.13	0.81
368.3086	5.29	0.42	538.2991	136.75	10.99
369.2773	2.81	0.23	539.3039	119.93	9.64
373.1992	3.29	0.26	540.3059	48.01	3.86

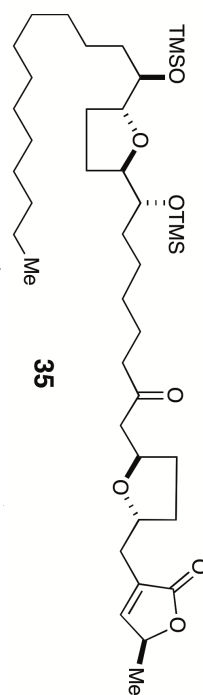
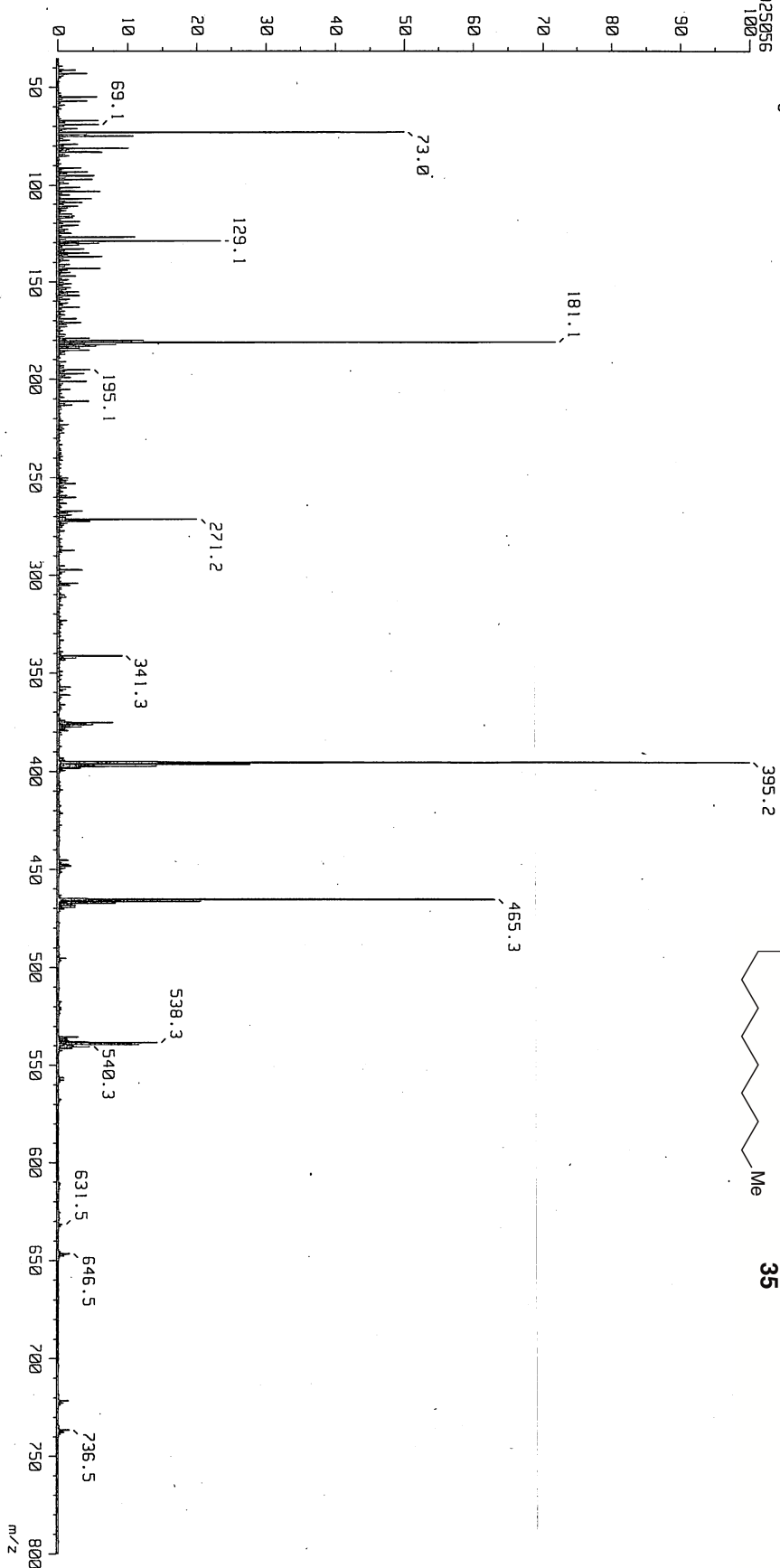
[Mass Spectrum]

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Data : 20160527 takahashi tn005 Date : 27-May-2016 15:57
Sample: ST-2711A-1 (in C6D6) PT:160 CT:200 PT:35 (1.2) ->128/m -> 350
Note : DB-5MS,122-5532, 30m x 0.25mm id, film 0.25um, ser# USA244957H (12/10/
Inlet : Direct Ion Mode : EI+
Spectrum Type : Normal Ion [MF-Linear]
RT : 2.34 min Scan# : (70,72)
BP : m/z 395.2137 Int. : 1243.93
Output m/z range : 35.0000 to 800.0000 Cut Level : 0.00 %

m/z	Int.	Norm.
541.3070	15.63	1.26
542.3202	3.97	0.32
556.3930	9.04	0.73
557.4115	5.39	0.43
567.3038	5.94	0.48
568.3218	2.74	0.22
610.3392	4.97	0.40
611.3500	4.13	0.33
628.4406	4.51	0.36
629.4379	2.83	0.23
631.4245	4.70	0.38
645.4468	4.75	0.38
646.4437	28.03	2.25
647.4514	13.80	1.11
648.4558	4.59	0.37
721.4667	28.55	2.29
722.4774	15.56	1.25
723.4696	6.57	0.53
736.4930	10.43	0.84
737.4910	5.92	0.48

[Mass Spectrum]
 Date : 20160801 takahashi tn004 Date : 01-Aug-2016 18:27
 Sample: ST-2761A-1 (in C6D6) PT:210 CT:200 PT: 35 (1.2) ->128/m -> 350
 Note : DB-SMS,122-5532, 30m x 0.25mm id, film 0.25um, ser# USR24957H (12/10/
 Inlet : Direct Ion Mode : EI+
 Spectrum Type : Normal Ion [MF-Linear]
 RT : 2.82 min Scan# : (84, 87)
 BP : m/z 395.2230 Int. : 713.47
 Output m/z range : 35.0000 to 800.0000 Cut Level : 0.00 %
 2925056



[Mass Spectrum]

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Data : 20160801 takahashi tn004

Date : 01-Aug-2016 18:27

Sample: ST-2761A-1 (in C6D6) PT:210 CT:200 PT: 35 (1.2) ->128/m -> 350

Note : DB-5MS,122-5532, 30m x 0.25mm id, film 0.25um, ser# USA244957H (12/10/

Inlet : Direct

Ion Mode : EI+

Spectrum Type : Normal Ion [MF-Linear]

RT : 2.82 min Scan# : (84,87)

BP : m/z 395.2230 Int. : 713.47

Output m/z range : 35.0000 to 800.0000

Cut Level : 0.00 %

m/z	Int.	Norm.	m/z	Int.	Norm.
39.0204	4.55	0.64	103.0448	43.52	6.10
41.0369	18.27	2.56	104.0449	4.55	0.64
42.0355	3.94	0.55	105.0481	12.48	1.75
43.0329	29.74	4.17	106.0570	2.97	0.42
43.9985	6.13	0.86	107.0655	34.09	4.78
45.0059	3.83	0.54	108.0646	7.66	1.07
46.9861	1.86	0.26	109.0702	24.81	3.48
53.0283	3.00	0.42	110.0608	5.55	0.78
54.0342	4.04	0.57	111.0658	20.23	2.84
55.0309	40.02	5.61	112.0510	9.76	1.37
56.0373	5.05	0.71	113.0506	8.30	1.16
57.0451	30.20	4.23	114.0482	2.88	0.40
58.0314	2.63	0.37	115.0493	14.10	1.98
59.0142	7.00	0.98	116.0575	16.54	2.32
60.9951	3.20	0.45	117.0592	15.36	2.15
65.0258	1.74	0.24	118.0651	3.12	0.44
66.0384	3.23	0.45	119.0693	22.46	3.15
67.0511	41.52	5.82	120.0791	5.22	0.73
68.0522	7.41	1.04	121.0904	20.59	2.89
69.0636	42.13	5.90	122.0835	4.17	0.58
70.0568	6.34	0.89	123.0845	10.34	1.45
71.0607	20.12	2.82	124.0764	6.71	0.94
72.0398	4.51	0.63	125.0807	13.62	1.91
73.0336	356.58	49.98	126.0825	3.86	0.54
74.0313	29.50	4.13	127.0731	78.88	11.06
75.0097	77.36	10.84	128.0732	8.55	1.20
76.0065	5.43	0.76	129.0706	167.44	23.47
76.9994	12.11	1.70	130.0776	41.83	5.86
78.0108	1.65	0.23	131.0718	21.12	2.96
79.0258	20.34	2.85	132.0768	4.52	0.63
80.0328	5.96	0.84	133.0886	26.72	3.74
81.0315	72.36	10.14	134.0847	7.09	0.99
82.0371	12.10	1.70	135.0924	31.69	4.44
83.0474	45.40	6.36	136.0734	10.33	1.45
84.0418	7.67	1.07	137.0659	45.26	6.34
85.0470	11.43	1.60	138.0778	6.12	0.86
87.0175	1.97	0.28	139.0696	11.43	1.60
89.0261	3.31	0.46	140.0779	1.91	0.27
91.0460	23.40	3.28	141.0729	11.91	1.67
92.0551	3.98	0.56	142.0744	6.82	0.96
93.0683	30.43	4.26	143.0822	43.40	6.08
94.0613	9.30	1.30	144.0815	9.70	1.36
95.0696	37.34	5.23	145.0803	12.60	1.77
96.0732	7.85	1.10	146.0835	2.77	0.39
97.0820	35.45	4.97	147.0831	17.86	2.50
98.0767	5.94	0.83	148.0882	4.25	0.60
99.0613	11.05	1.55	149.0793	11.10	1.56
100.0521	1.62	0.23	150.0911	5.96	0.84
101.0394	22.33	3.13	151.0992	14.07	1.97
102.0392	2.75	0.39	152.1034	4.85	0.68

[Mass Spectrum]

Page: 2

Data : 20160801_takahashi tn004

Date : 01-Aug-2016 18:27

Sample: ST-2761A-1 (in C6D6) PT:210 CT:200 PT: 35 (1.2) ->128/m -> 350

Note : DB-5MS,122-5532, 30m x 0.25mm id, film 0.25um, ser# USA244957H (12/10/

Inlet : Direct

Ion Mode : EI+

Spectrum Type : Normal Ion [MF-Linear]

RT : 2.82 min Scan# : (84,87)

BP : m/z 395.2230 Int. : 713.47

Output m/z range : 35.0000 to 800.0000

Cut Level : 0.00 %

m/z	Int.	Norm.	m/z	Int.	Norm.
153.0933	12.86	1.80	204.1037	2.44	0.34
154.0750	8.68	1.22	205.0987	12.53	1.76
155.0845	21.25	2.98	206.1163	2.42	0.34
156.0892	10.30	1.44	207.1199	4.39	0.62
157.0835	22.29	3.12	208.1294	2.00	0.28
158.0883	5.43	0.76	209.1187	6.62	0.93
159.0847	12.25	1.72	210.1187	2.82	0.40
160.0906	2.83	0.40	211.1247	31.52	4.42
161.0954	10.19	1.43	212.1259	7.91	1.11
162.0800	5.06	0.71	213.1090	14.81	2.08
163.0814	22.35	3.13	214.1173	3.29	0.46
164.0934	4.12	0.58	215.1231	3.95	0.55
165.1129	7.34	1.03	217.1285	3.44	0.48
166.0978	3.17	0.44	218.1377	1.57	0.22
167.0868	6.98	0.98	219.1379	2.35	0.33
168.0960	3.30	0.46	220.1309	3.55	0.50
169.0851	19.18	2.69	221.1237	5.06	0.71
170.0924	6.25	0.88	222.1286	2.15	0.30
171.1006	23.66	3.32	223.1111	11.03	1.55
172.0986	8.90	1.25	224.1217	5.07	0.71
173.1083	9.26	1.30	225.1420	6.53	0.91
174.1089	2.27	0.32	226.1212	4.01	0.56
175.1123	5.33	0.75	227.1349	6.53	0.91
176.1054	2.01	0.28	228.1396	1.80	0.25
177.0991	8.20	1.15	229.1353	2.61	0.37
178.0978	6.87	0.96	231.1380	1.93	0.27
179.1027	31.99	4.48	233.1767	4.39	0.62
180.0836	88.16	12.36	234.1598	1.76	0.25
181.0892	513.21	71.93	235.1702	2.67	0.37
182.0928	59.71	8.37	236.1385	3.17	0.44
183.0924	38.73	5.43	237.1557	3.52	0.49
184.0925	22.15	3.11	238.1345	3.33	0.47
185.1010	32.28	4.52	239.1737	5.31	0.74
186.1076	7.06	0.99	240.1579	2.50	0.35
187.1091	7.43	1.04	241.1594	5.22	0.73
188.1110	1.86	0.26	242.1579	1.78	0.25
189.1155	2.64	0.37	243.1428	3.18	0.45
191.1017	8.31	1.17	245.1576	2.99	0.42
192.1085	2.57	0.36	247.1717	1.87	0.26
193.1095	6.55	0.92	249.1668	4.01	0.56
194.1042	5.79	0.81	250.2056	10.14	1.42
195.1037	33.09	4.64	251.1882	10.05	1.41
196.1081	7.13	1.00	252.1375	9.06	1.27
197.1267	27.15	3.81	253.1420	18.09	2.54
198.1188	7.93	1.11	254.1460	6.57	0.92
199.1204	13.18	1.85	255.1777	8.34	1.17
200.1222	3.35	0.47	256.1797	2.84	0.40
201.1265	29.49	4.13	257.1809	5.35	0.75
202.1286	5.58	0.78	258.1697	1.92	0.27
203.1191	3.89	0.55	259.1670	9.57	1.34

[Mass Spectrum]

Page: 3

Data : 20160801 takahashi tn004

Date : 01-Aug-2016 18:27

Sample: ST-2761A-1 (in C6D6) PT:210 CT:200 PT: 35 (1.2) ->128/m -> 350

Note : DB-5MS,122-5532, 30m x 0.25mm id, film 0.25um, ser# USA244957H (12/10/

Inlet : Direct

Ion Mode : EI+

Spectrum Type : Normal Ion [MF-Linear]

RT : 2.82 min Scan# : (84,87)

BP : m/z 395.2230 Int. : 713.47

Output m/z range : 35.0000 to 800.0000

Cut Level : 0.00 %

m/z	Int.	Norm.	m/z	Int.	Norm.
260.1411	18.86	2.64	325.2375	4.83	0.68
261.1567	5.09	0.71	326.2362	1.92	0.27
263.2005	8.95	1.25	327.2275	2.53	0.35
264.1982	2.64	0.37	329.2089	2.55	0.36
265.1790	3.31	0.46	330.2122	1.60	0.22
266.1590	2.00	0.28	331.2445	2.33	0.33
267.1502	25.13	3.52	333.2894	5.61	0.79
268.1526	9.97	1.40	334.2868	1.84	0.26
269.1699	13.93	1.95	335.2367	2.23	0.31
270.2104	8.12	1.14	337.2328	2.53	0.35
271.2434	143.23	20.08	339.2155	4.53	0.63
272.2448	32.56	4.56	340.2247	2.89	0.40
273.2250	9.58	1.34	341.2848	65.43	9.17
274.1718	5.76	0.81	342.2881	18.22	2.55
275.1718	4.05	0.57	343.2819	7.38	1.03
276.1708	2.17	0.30	344.2732	1.62	0.23
277.1812	6.78	0.95	345.2599	1.70	0.24
278.1825	1.77	0.25	349.2255	4.27	0.60
279.1829	2.45	0.34	350.2223	1.66	0.23
281.2065	4.51	0.63	351.2220	3.57	0.50
282.1996	1.68	0.24	353.2623	2.77	0.39
283.1945	2.90	0.41	355.2639	1.57	0.22
284.2144	1.80	0.25	356.2087	2.28	0.32
285.2004	4.76	0.67	357.2112	12.61	1.77
286.1688	3.91	0.55	358.2166	7.97	1.12
287.1679	16.70	2.34	359.2449	4.24	0.59
288.1728	4.15	0.58	361.3044	12.23	1.71
289.1961	1.94	0.27	362.3047	3.65	0.51
293.1844	1.82	0.26	363.2834	1.79	0.25
295.1635	6.56	0.92	365.2294	2.31	0.32
296.1737	2.22	0.31	366.2244	6.99	0.98
297.2488	25.47	3.57	367.2467	3.47	0.49
298.2500	7.24	1.02	368.3081	2.94	0.41
299.2289	3.61	0.51	369.2781	1.92	0.27
301.2564	2.53	0.36	373.2127	2.15	0.30
303.1817	3.15	0.44	374.2094	6.98	0.98
304.1659	20.61	2.89	375.2184	56.12	7.87
305.1768	12.20	1.71	376.2146	35.71	5.01
306.1810	3.50	0.49	377.2215	23.52	3.30
307.1926	3.09	0.43	378.2235	7.11	1.00
309.2106	2.70	0.38	379.2756	10.05	1.41
310.2114	6.73	0.94	380.2740	2.98	0.42
311.2310	8.69	1.22	381.2780	3.61	0.51
312.2308	2.84	0.40	387.2238	3.27	0.46
313.2235	2.72	0.38	393.2234	5.66	0.79
315.2670	4.52	0.63	394.2250	6.76	0.95
317.2477	1.78	0.25	395.2230	713.47	100.00
321.1914	2.57	0.36	396.2255	197.31	27.66
323.2040	9.41	1.32	397.2308	100.77	14.12
324.2068	2.99	0.42	398.2359	22.95	3.22

[Mass Spectrum]

Page: 4

Data : 20160801 takahashi tn004

Date : 01-Aug-2016 18:27

Sample: ST-2761A-1 (in C6D6) PT:210 CT:200 PT: 35 (1.2) ->128/m -> 350

Note : DB-5MS,122-5532, 30m x 0.25mm id, film 0.25um, ser# USA244957H (12/10/

Inlet : Direct

Ion Mode : EI+

Spectrum Type : Normal Ion [MF-Linear]

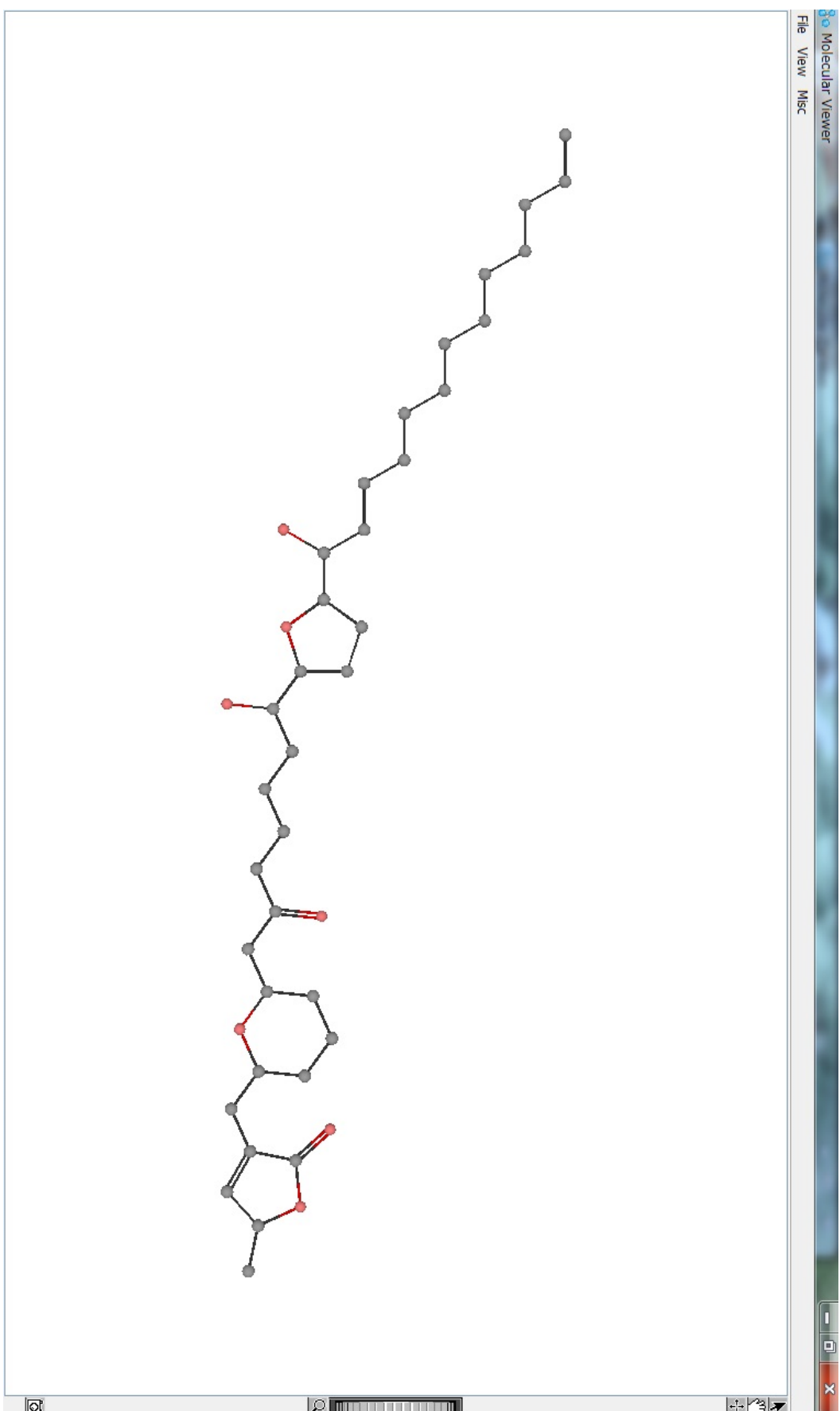
RT : 2.82 min Scan# : (84,87)

BP : m/z 395.2230 Int. : 713.47

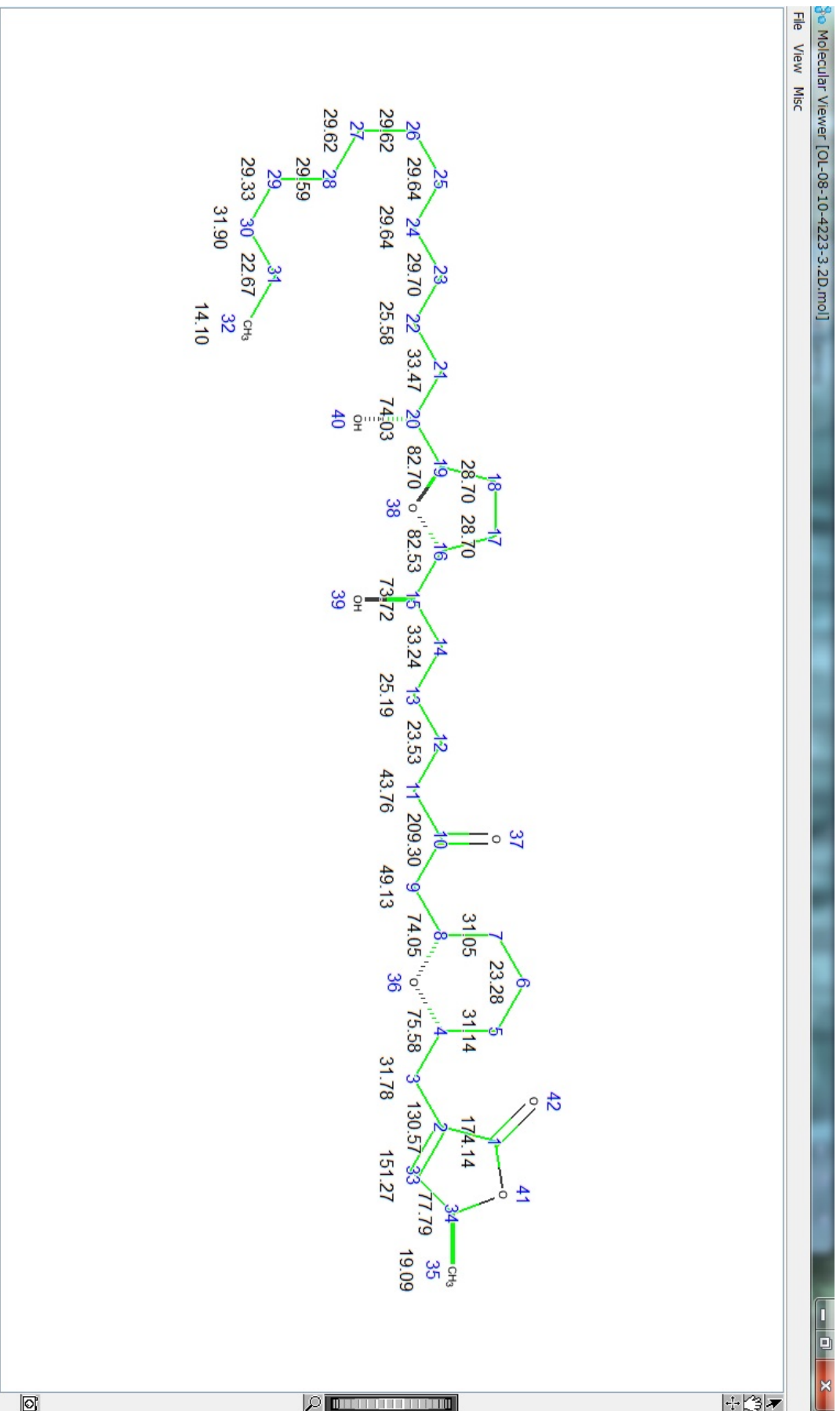
Output m/z range : 35.0000 to 800.0000

Cut Level : 0.00 %

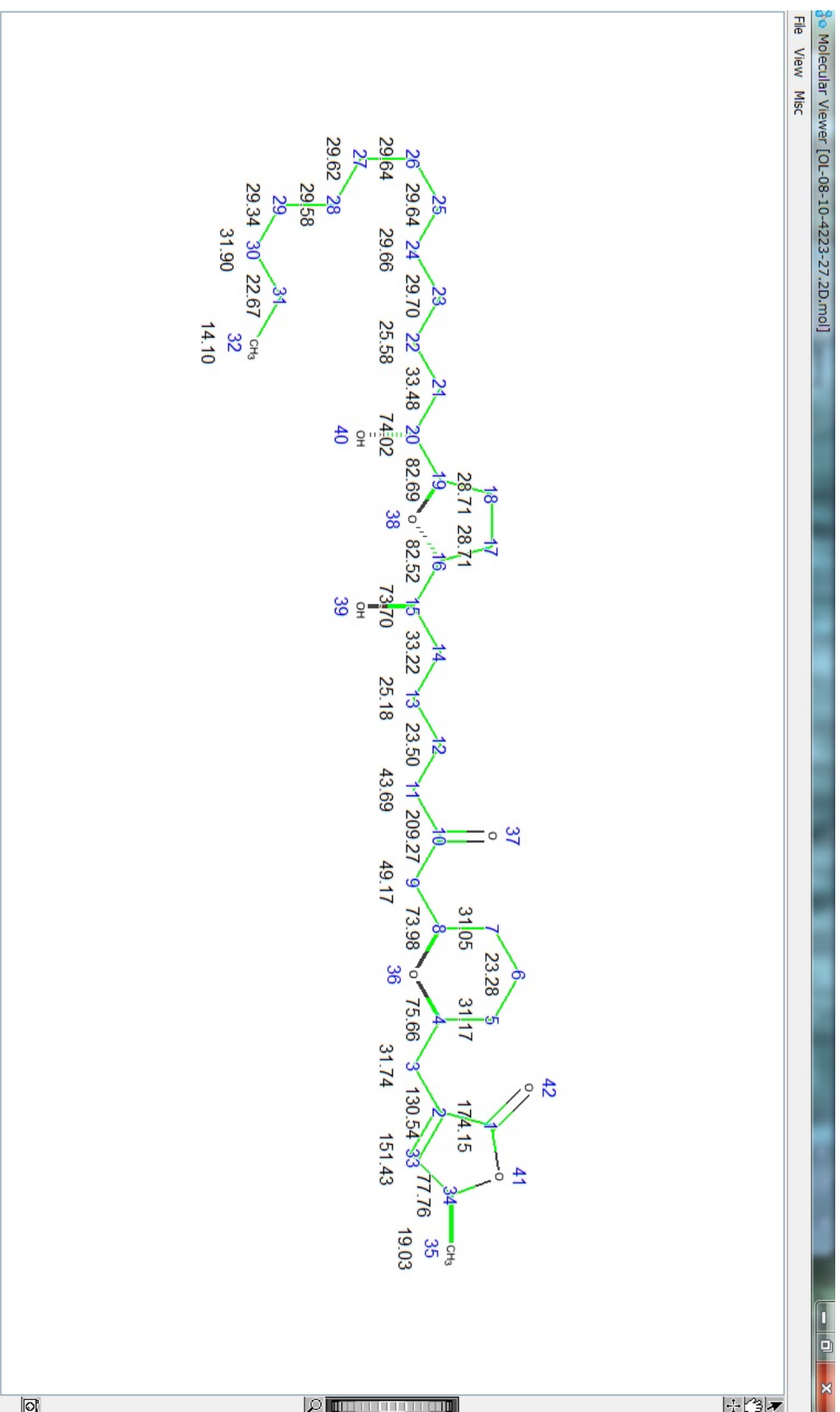
m/z	Int.	Norm.	m/z	Int.	Norm.
399.2856	7.32	1.03	541.3444	14.44	2.02
400.3077	1.91	0.27	542.3593	4.42	0.62
405.2577	1.87	0.26	556.4135	6.23	0.87
407.2657	2.36	0.33	557.4205	6.53	0.92
409.2737	4.68	0.66	558.4303	2.17	0.30
410.2829	1.69	0.24	567.3264	3.53	0.49
417.3632	2.71	0.38	610.3610	2.51	0.35
420.2696	1.79	0.25	611.3676	2.54	0.36
421.2581	4.26	0.60	613.4188	1.80	0.25
422.2562	1.79	0.25	625.4683	2.41	0.34
425.3186	1.75	0.24	628.4569	2.11	0.30
427.3470	3.40	0.48	631.4564	4.74	0.66
428.3355	1.58	0.22	632.4537	2.37	0.33
434.2750	1.53	0.21	645.4753	2.49	0.35
435.2903	1.64	0.23	646.4646	12.08	1.69
443.3323	2.09	0.29	647.4667	6.11	0.86
445.3604	10.50	1.47	648.4778	2.07	0.29
446.3405	3.90	0.55	721.4929	10.84	1.52
447.2625	11.58	1.62	722.4927	5.91	0.83
448.2634	13.12	1.84	723.4995	2.51	0.35
449.2722	7.63	1.07	736.5081	12.09	1.69
450.2788	2.71	0.38	737.5145	6.66	0.93
451.3223	3.95	0.55	738.5196	2.74	0.38
452.3295	1.57	0.22			
463.2756	3.26	0.46			
464.2814	2.54	0.36			
465.2675	450.44	63.13			
466.2703	147.18	20.63			
467.2703	59.15	8.29			
468.2739	17.78	2.49			
469.2769	17.66	2.48			
470.2809	6.16	0.86			
471.2756	2.44	0.34			
493.3866	2.07	0.29			
495.3033	8.34	1.17			
496.3103	3.44	0.48			
510.3379	1.61	0.23			
517.4087	3.78	0.53			
518.4091	1.75	0.24			
519.3958	1.57	0.22			
520.3290	3.49	0.49			
521.3448	3.08	0.43			
522.3545	1.57	0.22			
523.3596	1.96	0.27			
535.4205	21.27	2.98			
536.4114	9.53	1.34			
537.3441	11.07	1.55			
538.3146	102.19	14.32			
539.3193	83.07	11.64			
540.3193	32.81	4.60			



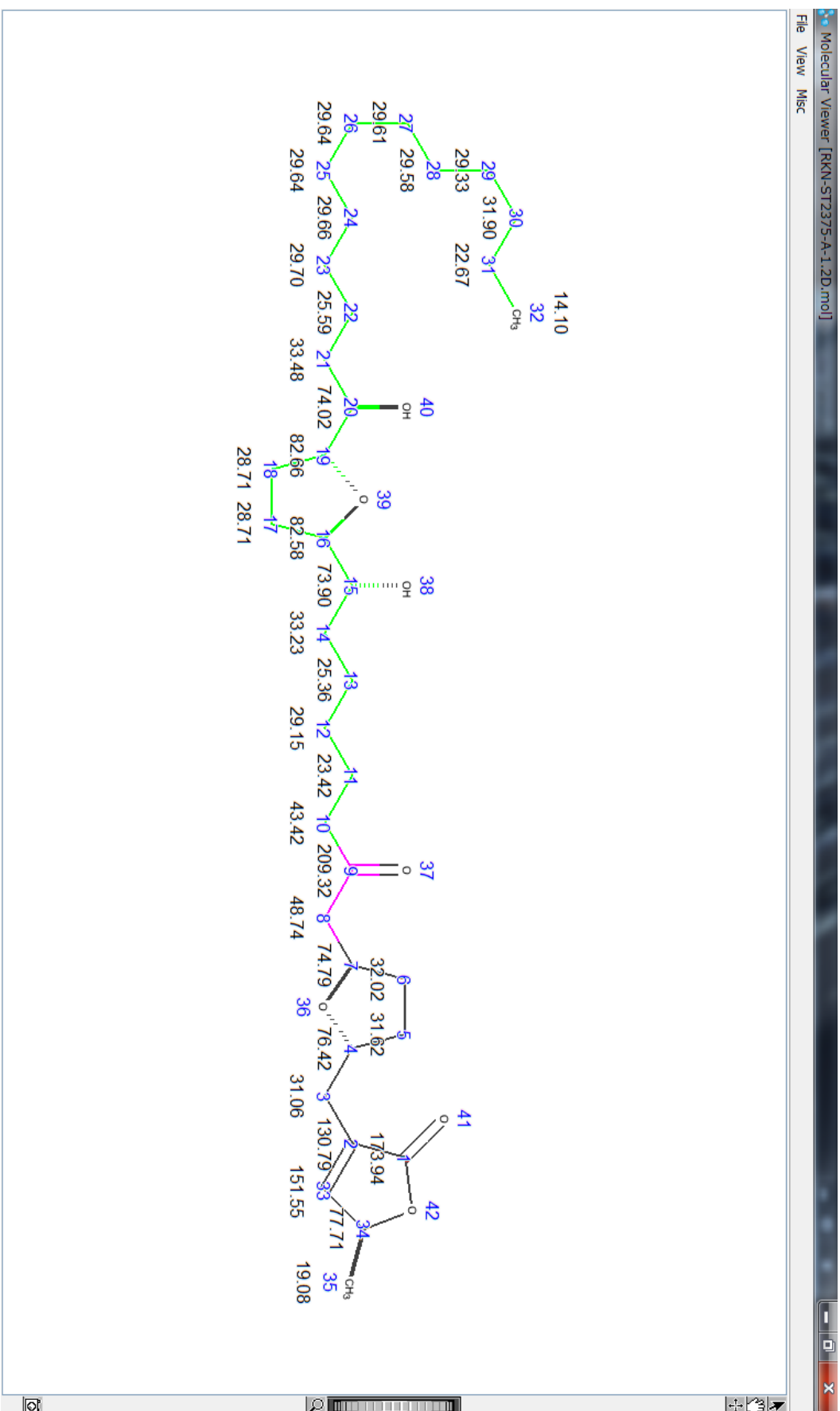
This is the proposed structure obtained by CAST/CNMR Structure Elucidator¹⁾ for query of natural aromin ¹³C NMR data²⁾ in the condition within 0.5 ppm error. This result was based on the complete matching for data of montanacin D (**32**) and its 4,8-di-epimer.



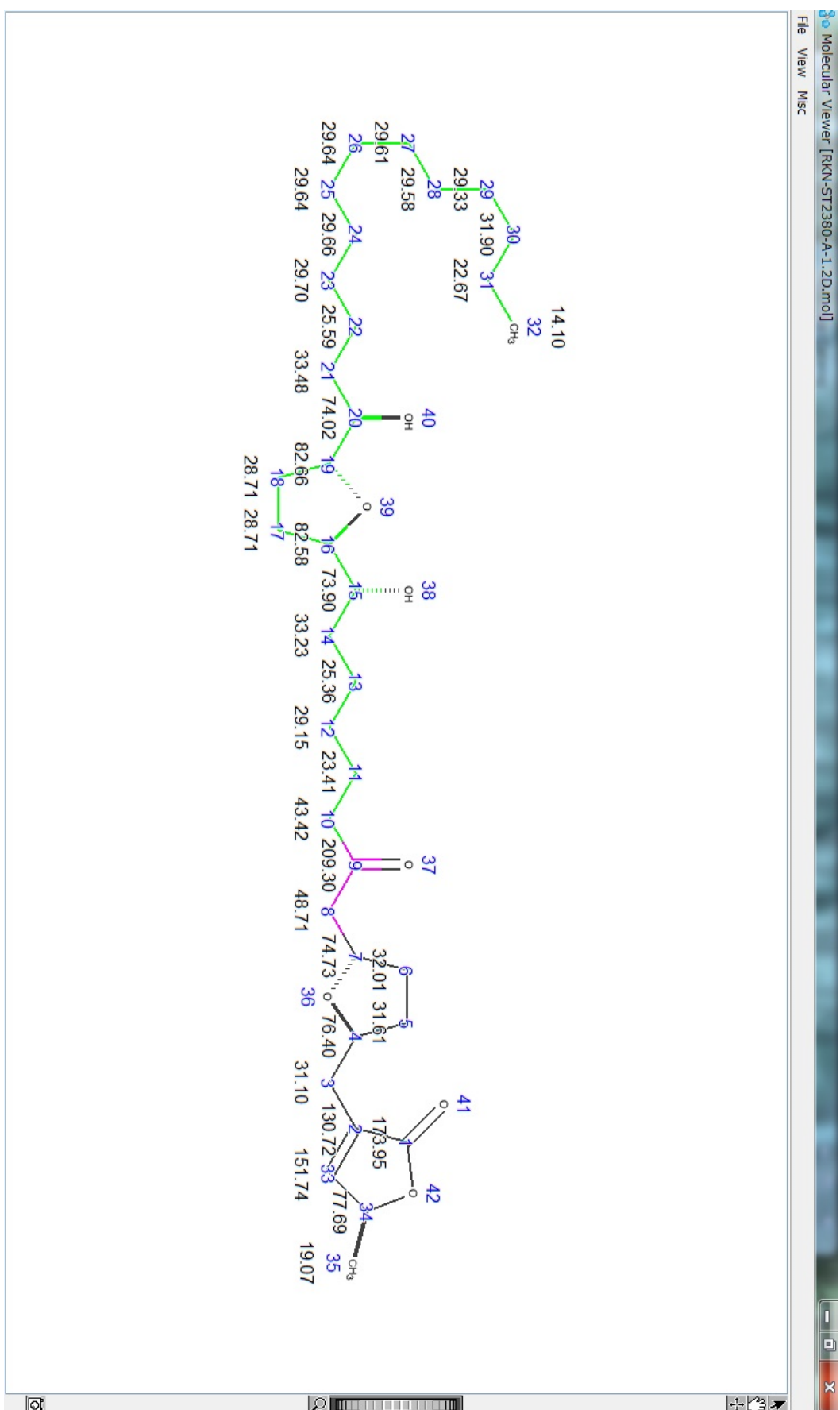
Montanacin D (**32**)³ was found in the search system by CAST/CNMR Structure Elucidator for the query of aromin ^{13}C NMR data. The green lines mean complete matched carbons.

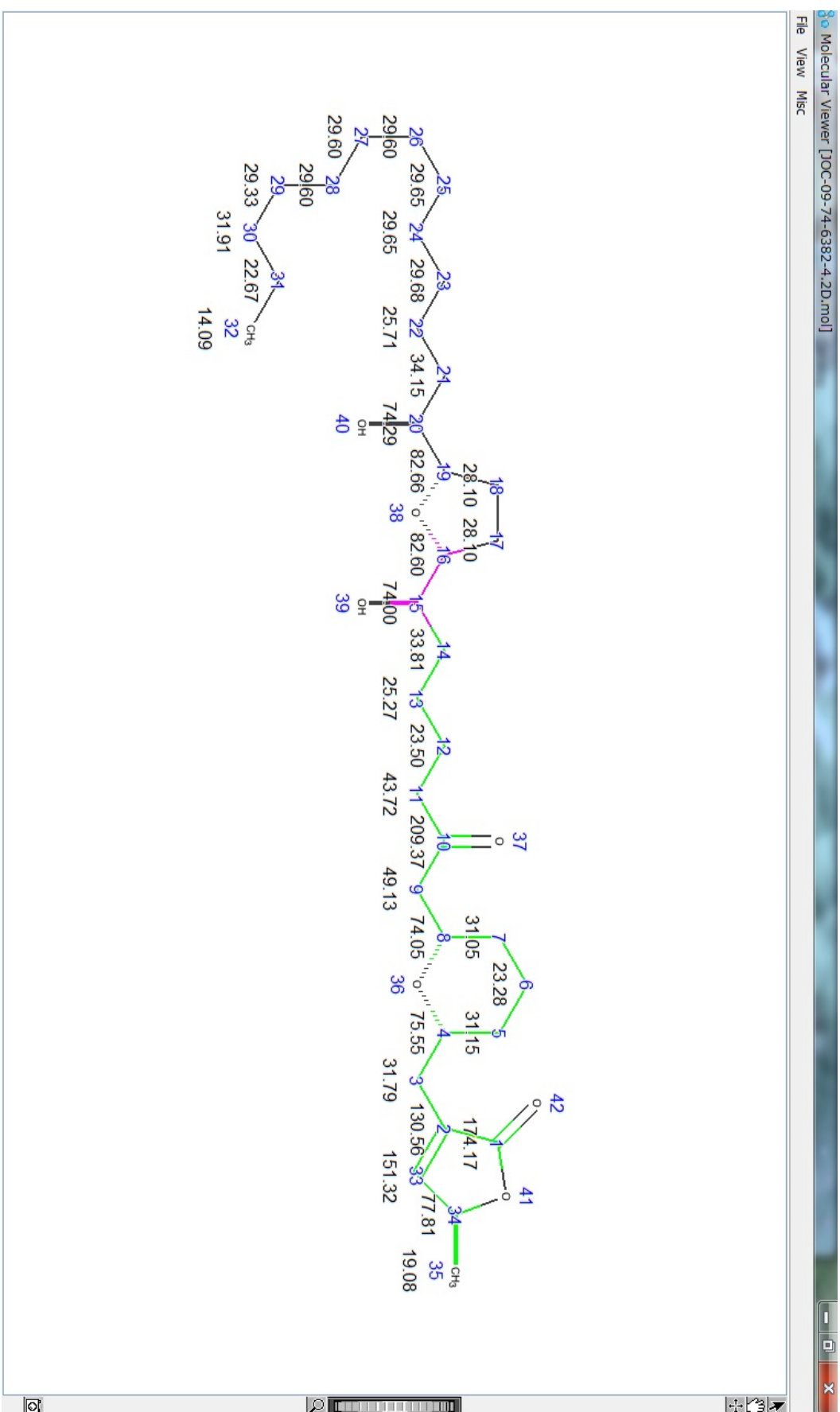


4,8-Di-*epi*-montanacin D³) was also found by the search system in the same condition as for montanin D (**32**).



^{13}C NMR data of synthesized **1** did not match with those reported of the natural arabin because of small difference on the oxygenated methin carbon (C-4). The balck lines except for the butenolide region⁴⁾ mean miss-matched carbons.





¹³C NMR data of montanacin E (and other related synthetic diastereoisomers)⁵⁾ did not match with those reported of the natural aramin.

References and note

1. Koichi, S.; Arisaka, M.; Koshino, H.; Aoki, A.; Iwata, S.; Uno, T.; Satoh, H. *J. Chem. Inf. Model.* **2014**, *54*, 1027.
2. Alfonso, D.; Colman-Saizarbitoria, T.; Zhao, G. -X.; Shi, G.; Ye, Q.; Schwedler, J. T.; McLaughlin, J. L. *Tetrahedron*, **1996**, *52*, 4215-4224.
3. Takahashi, S.; Hongo, Y.; Tsukagoshi, Y.; Koshino H. *Org. Lett.* **2008**, *10*, 4223-4226.
4. The ^{13}C -NMR data of the terminal lactone moiety was consistent with those reported of the natural product. It might be a bug of this program that the butenolide region doesn't show green color.
5. Takahashi, S., Takahashi, R.; Hongo Y., Koshino H., Yamaguchi, K.; Miyagi, T. *J. Org. Chem.* **2009**, *74*, 6382-6385.