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Supporting Information

A simple entry to tetracyclic oxazepine-fused pyrroles *via* metal-free [3+2] annulation between dibenzo[*b*,*f*][1,4]oxazepines and aqueous succinaldehyde

Sachin Choudhary,[a] Anoop Singh,[a] Jyothi Yadav,[a] Nisar A. Mir,[a] Sumati Anthal,[b] Rajni Kant,[b] and Indresh Kumar*[a]

[a]Department of Chemistry, Birla Institute of Technology and Science, Pilani 333 031, (Rajasthan) India

[b]X-ray Crystallography Laboratory, Post-Graduate Department of Physics & Electronics, University of Jammu, Jammu 180 006, India

E-mail: indresh.chemistry@gmail.com, indresh.kumar@pilani.bits-pilani.ac.in

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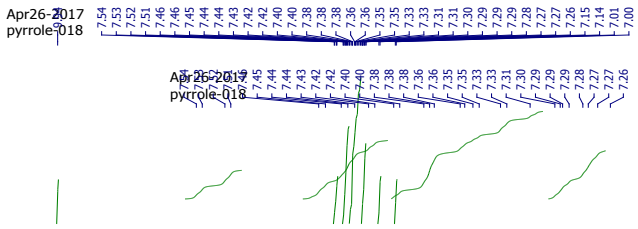
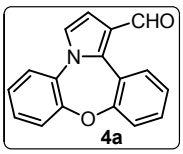

Apr26-2017
pyrrole-018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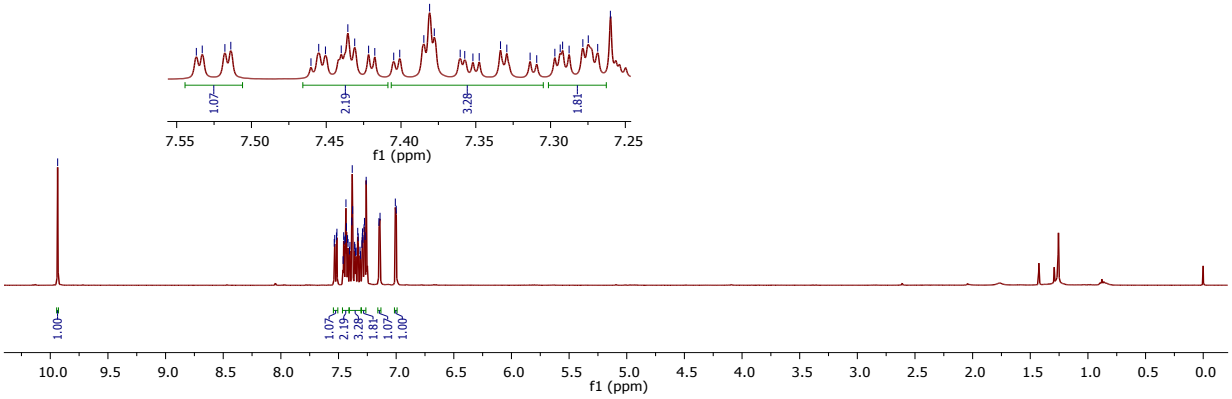
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7.25
f1 (ppm)
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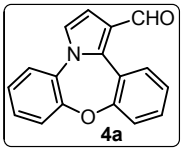
1.07
2.19
3.28
1.81
1.07
1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


186.27
158.86
153.32
137.73
132.11
131.83
131.12
128.63
126.24
125.64
124.74
123.29
122.57
122.32
122.19
121.22
110.13
77.00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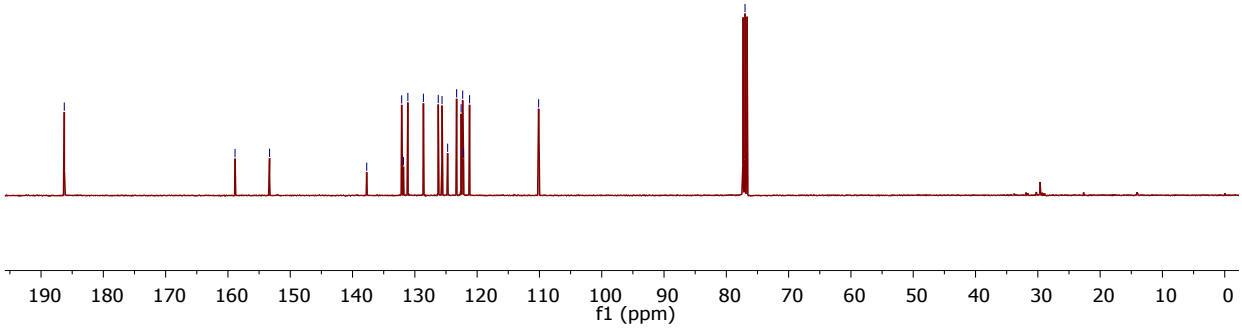
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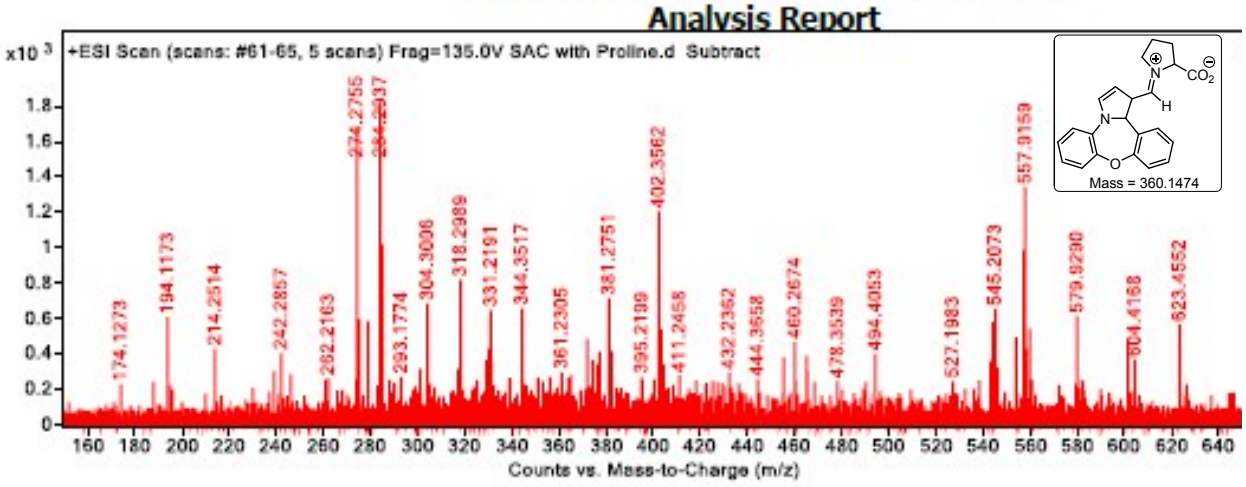
0
f1 (ppm)

HRMS Facility, BITS Pilani, Pilani Campus
Analysis Report



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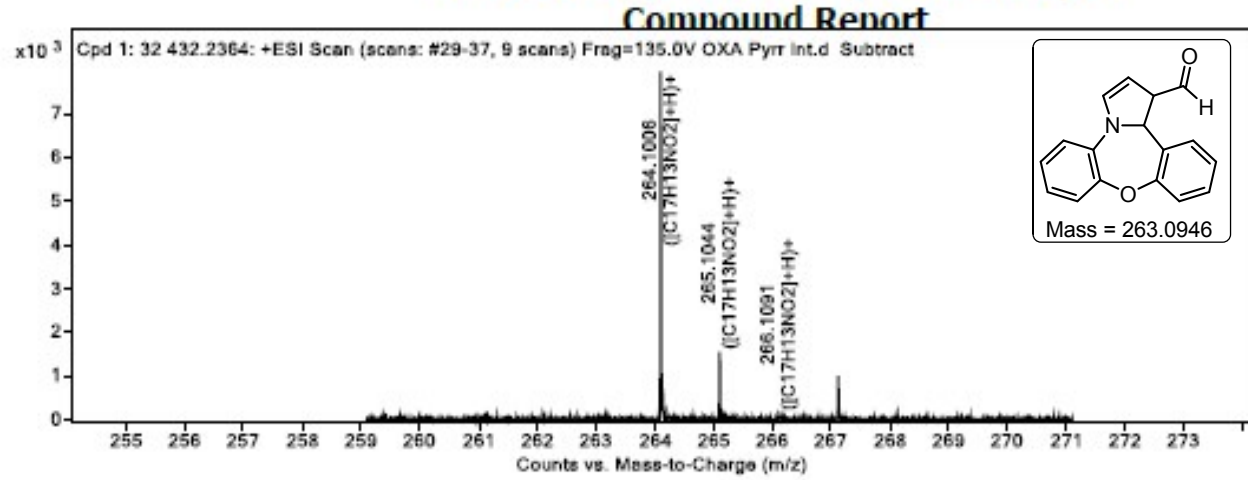
Compound Report



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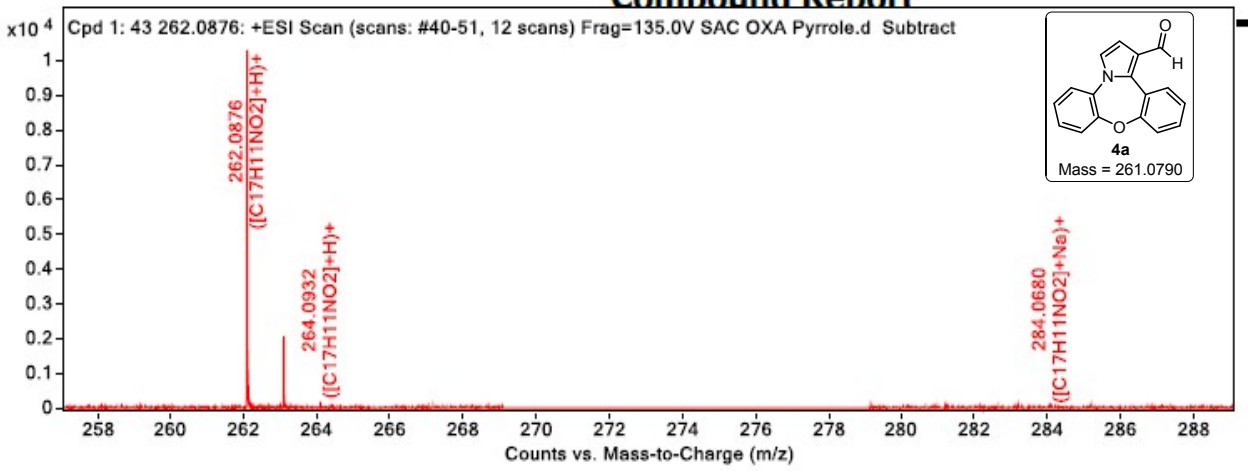
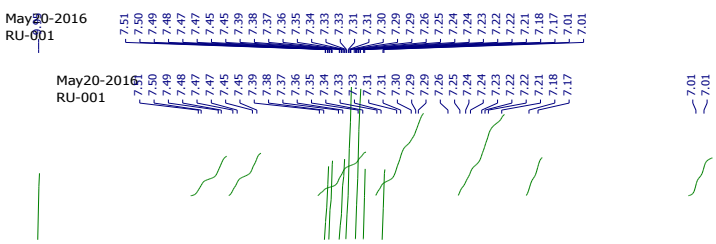
Compound Report

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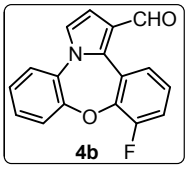
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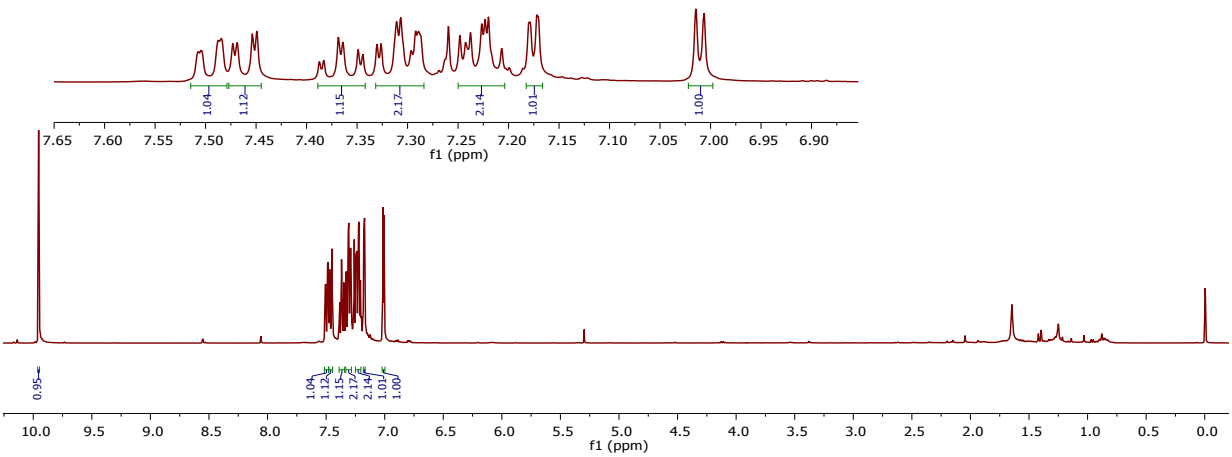
O
4b
F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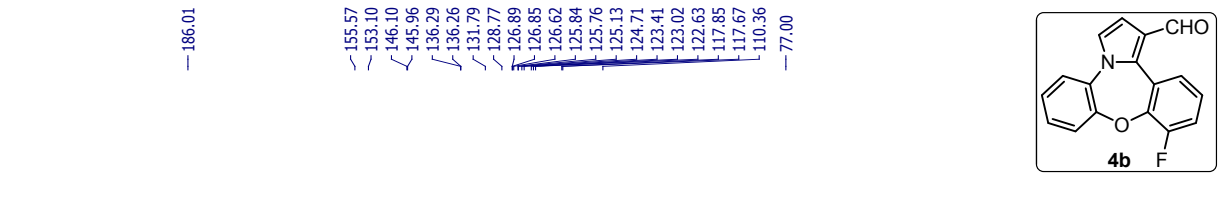
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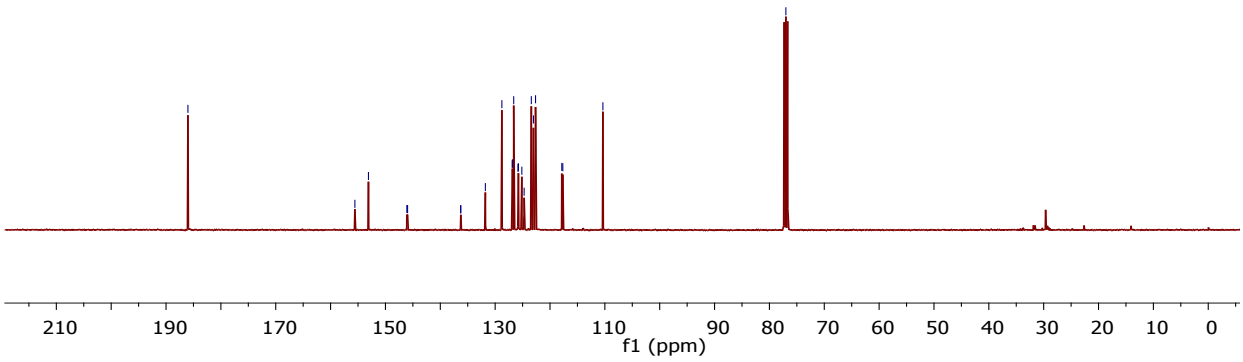
CHO
N
O
4b
F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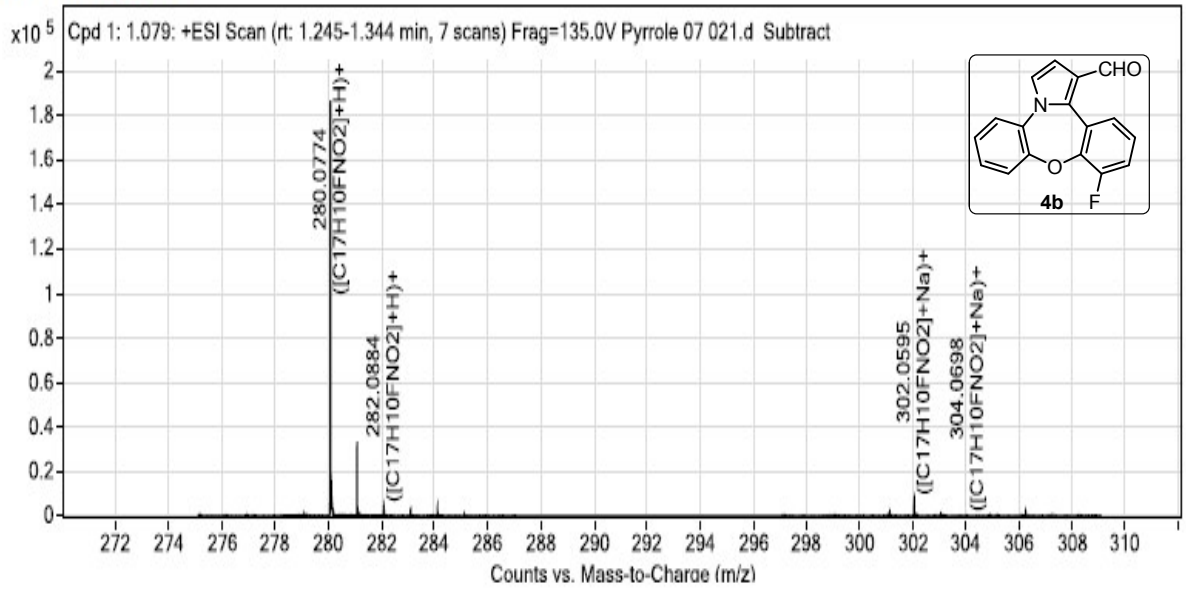
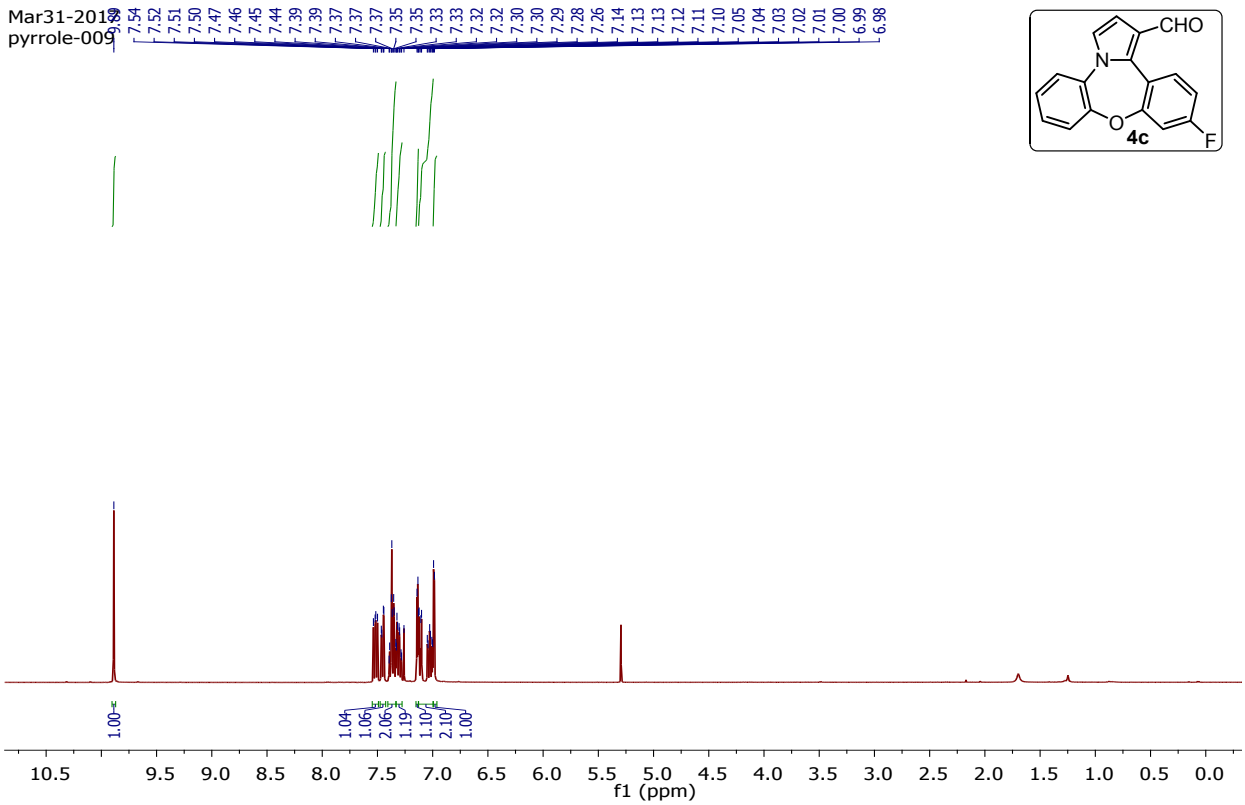
MS Zoomed Spectrum

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7.51
7.50
7.47
7.46
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7.39
7.39
7.37
7.37
7.37
7.35
7.35
7.33
7.33
7.32
7.32
7.30
7.30
7.29
7.28
7.26
7.14
7.13
7.13
7.12
7.11
7.10
7.05
7.04
7.03
7.02
7.01
7.00
6.99
6.98
CHO
N
O
F
4c
1.00
1.04
1.06
2.06
1.19
1.10
2.10
1.00
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9.5
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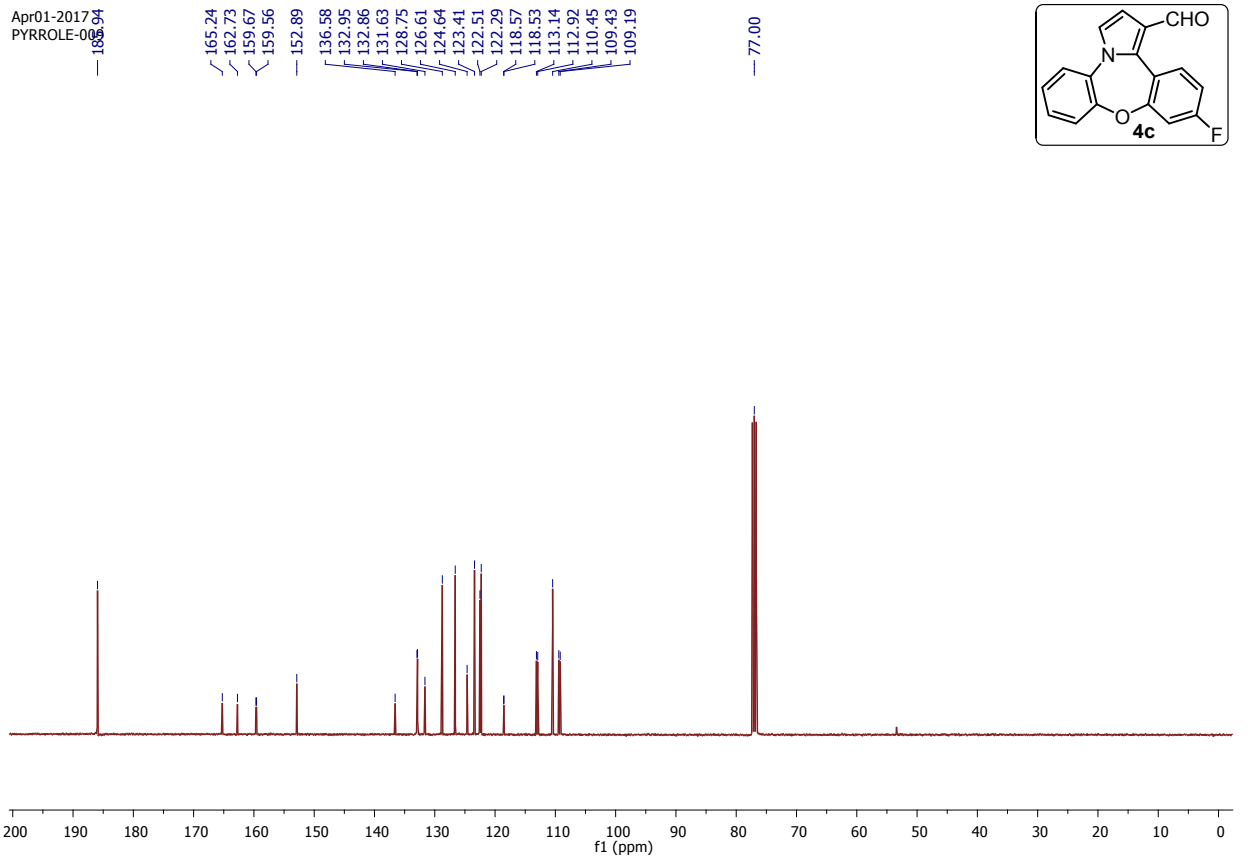
f1 (ppm)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159.56
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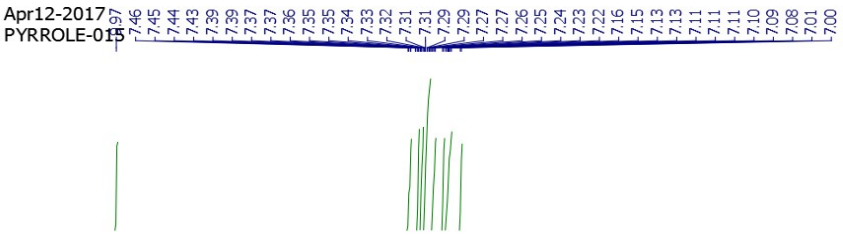
131.63
128.75
126.61
124.64
123.41
122.51
122.29
118.57
118.53
113.14
112.92
110.45
109.43
109.19
77.00
CHO
N
O
F
4c
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.39
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7.36
7.35
7.35
7.34
7.33
7.32
7.31
7.31
7.29
7.29
7.27
7.27
7.26
7.25
7.24
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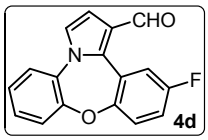
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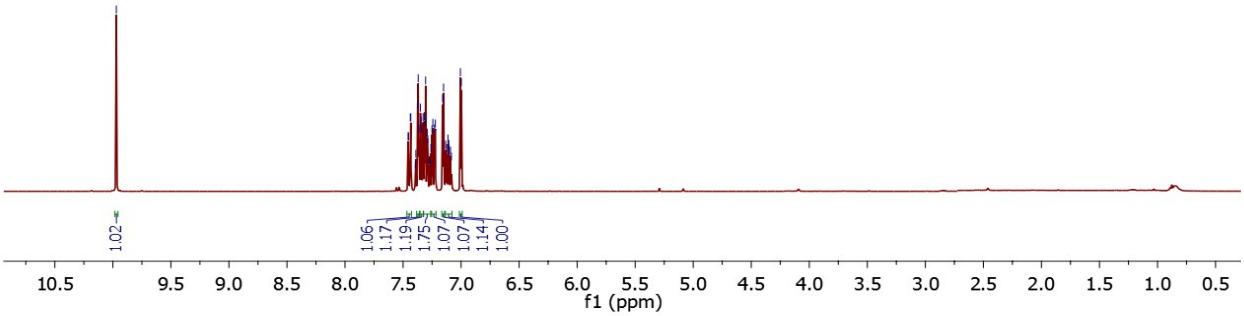
F
O
4d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8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


FID FILES FOR OXA PYRROLE 13
pyrrole-015
185.74
160.74
158.33
154.70
154.62
153.38
135.86
135.80
131.65
128.85
126.42
125.04
123.56
123.46
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122.92
122.50
122.41
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118.08
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117.29
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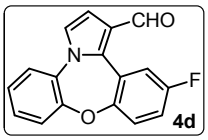
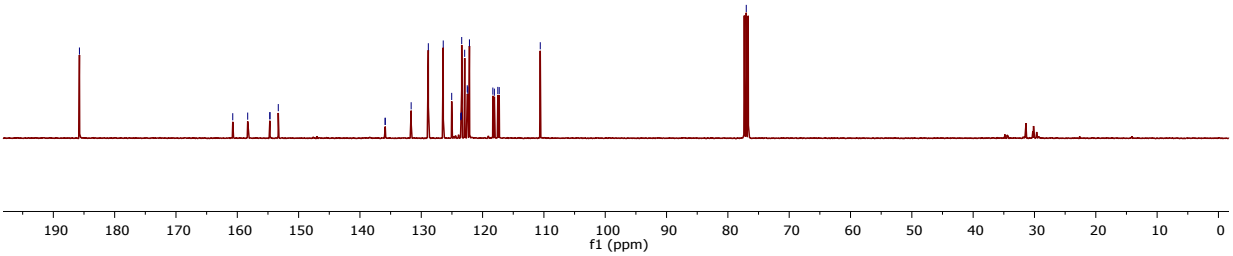
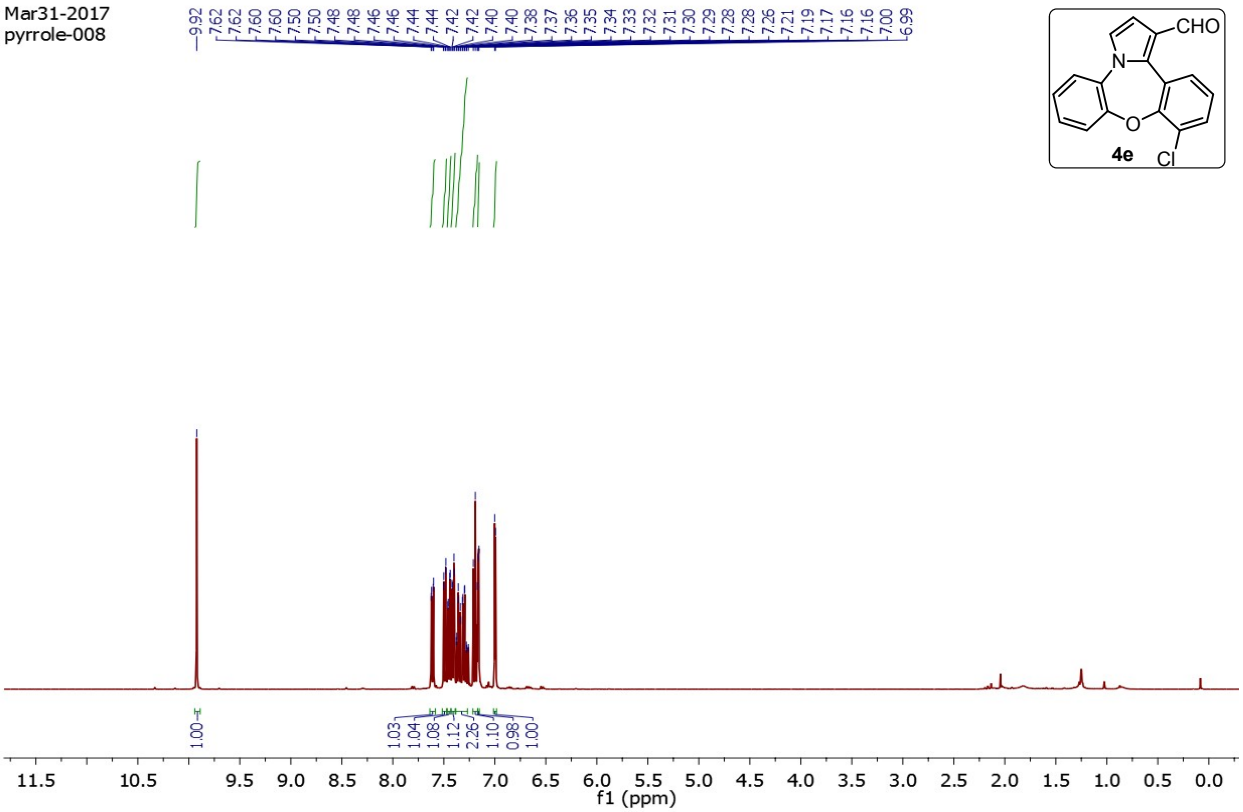
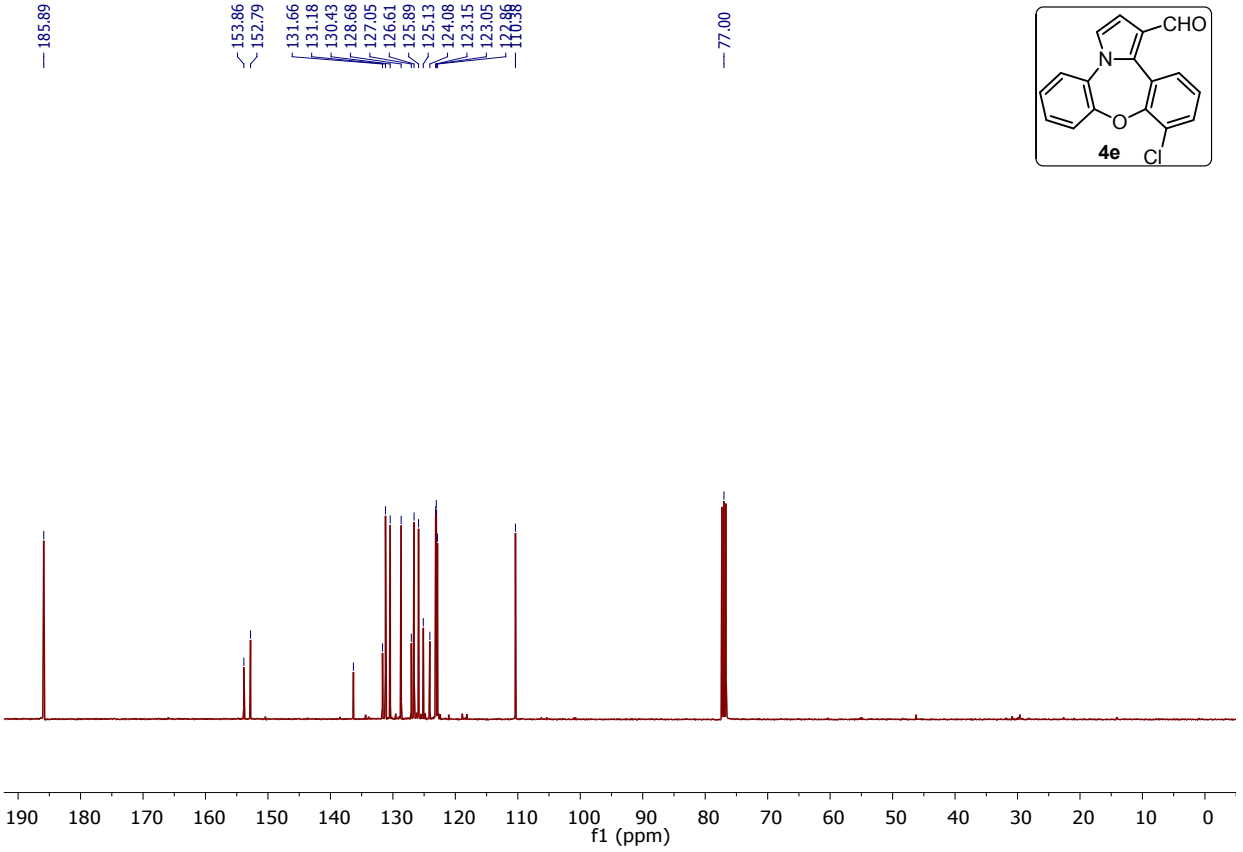
77.00

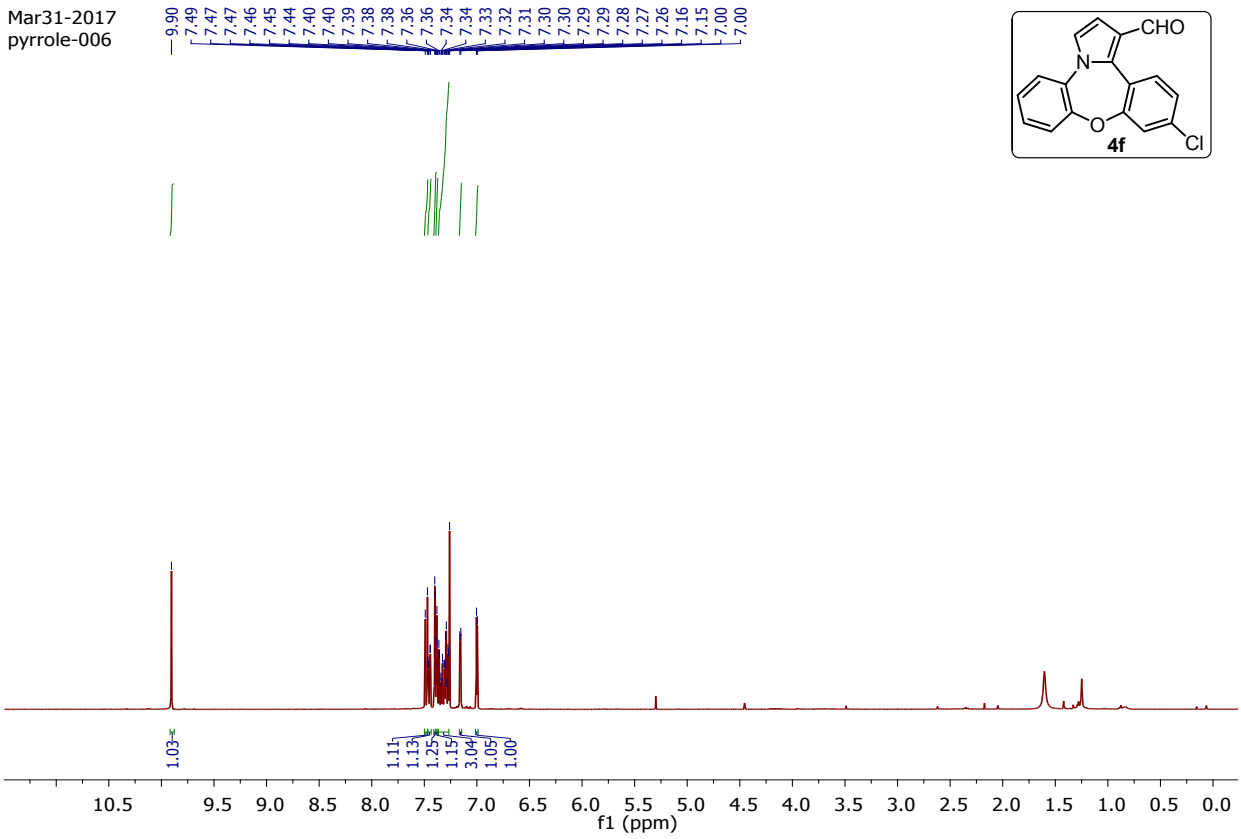

CHO
N
F
O
4d


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

Mar31-2017
pyrrole-008



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9.90
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7.46
7.45
7.44
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7.40
7.39
7.38
7.38
7.36
7.36
7.34
7.34
7.33
7.32
7.31
7.30
7.30
7.29
7.29
7.28
7.27
7.26
7.16
7.15
7.00
7.00
CHO
N
O
Cl
4f
1.03
1.11
1.13
1.25
1.15
3.04
1.05
1.00
10.5
9.5
9.0
8.5
8.0
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7.0
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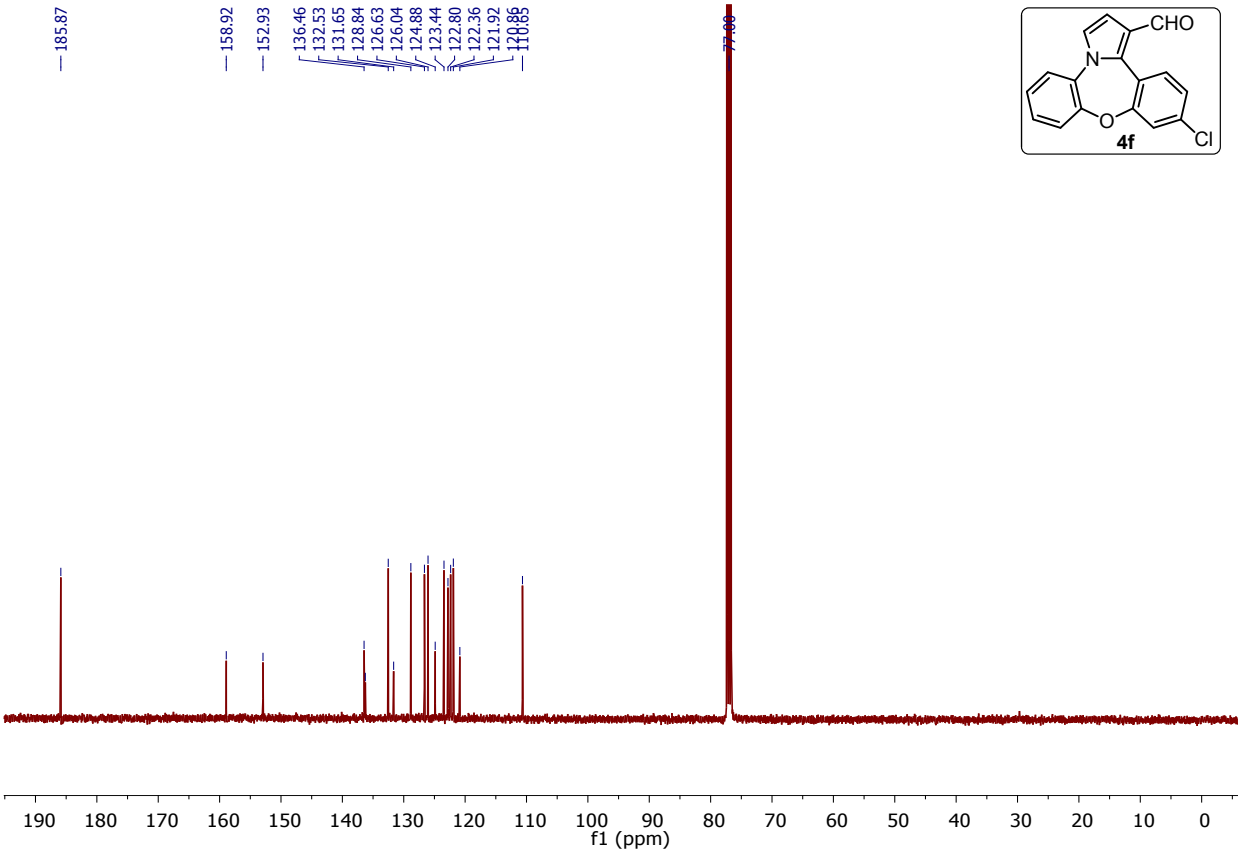
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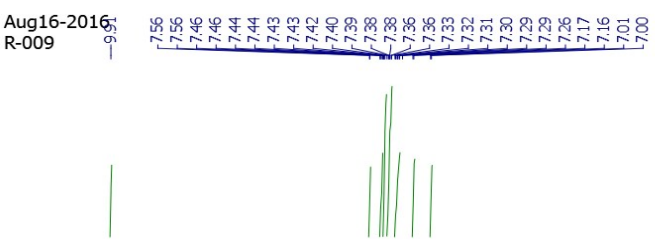
123.44
122.80
122.36
121.92
120.86
110.85
77.00
CHO
N
O
Cl
4f
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Aug16-2016
R-009
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7.40
7.39
7.38
7.38
7.36
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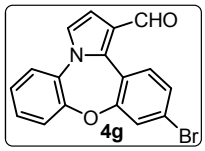
7.16
7.01
7.00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4g
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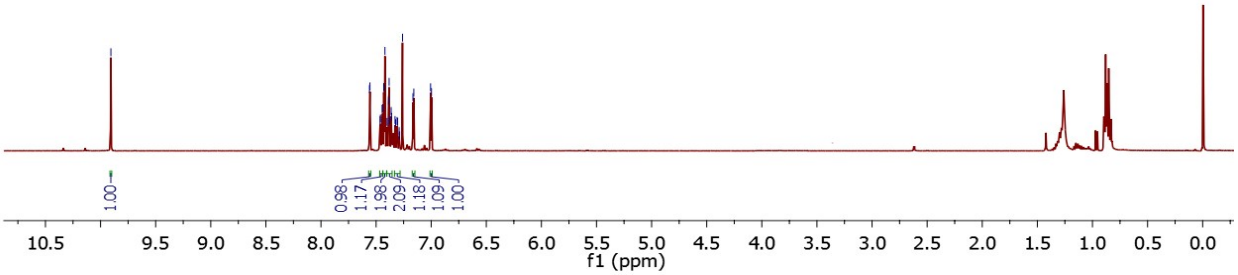
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8.0
7.5
7.0
6.5
6.0
5.5
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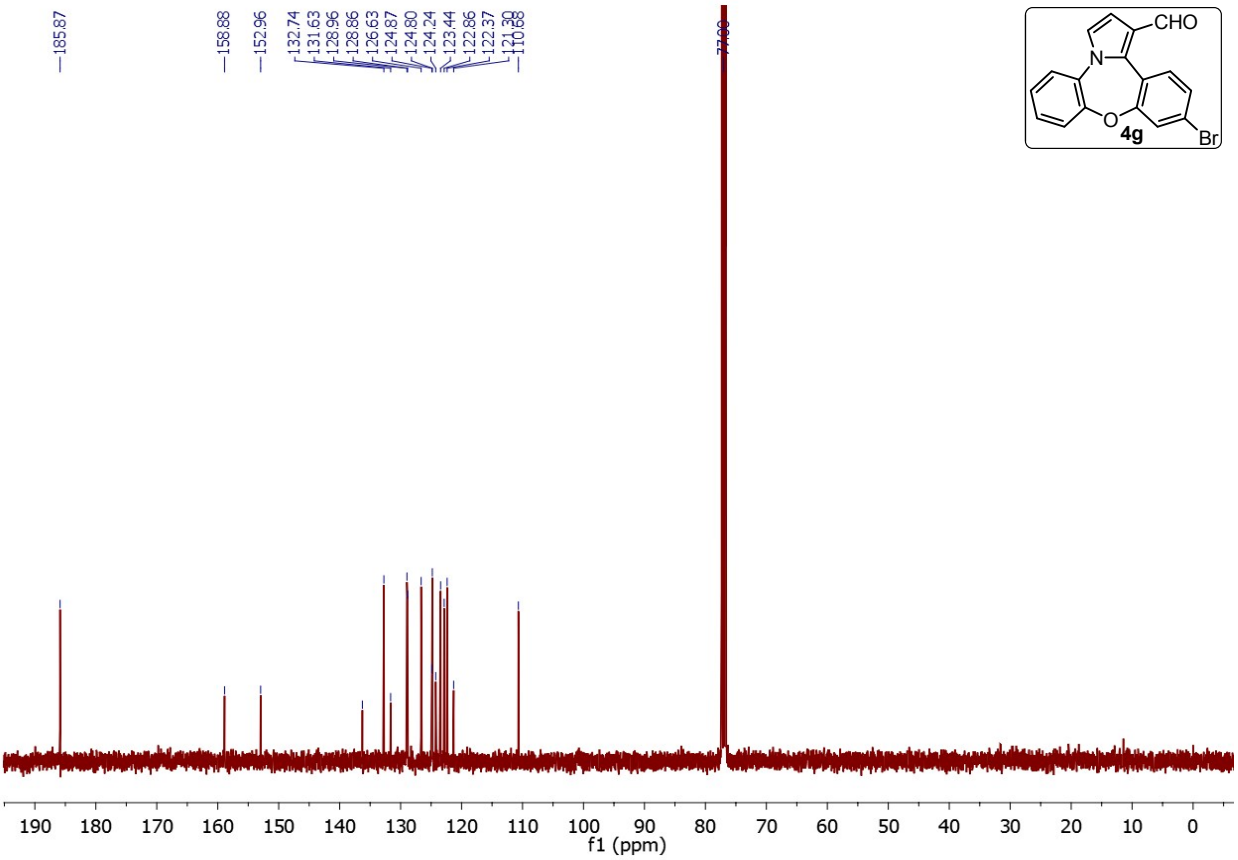
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122.86
122.37
121.30
110.68
77.00
190
180
170
160
150
140
130
120
110
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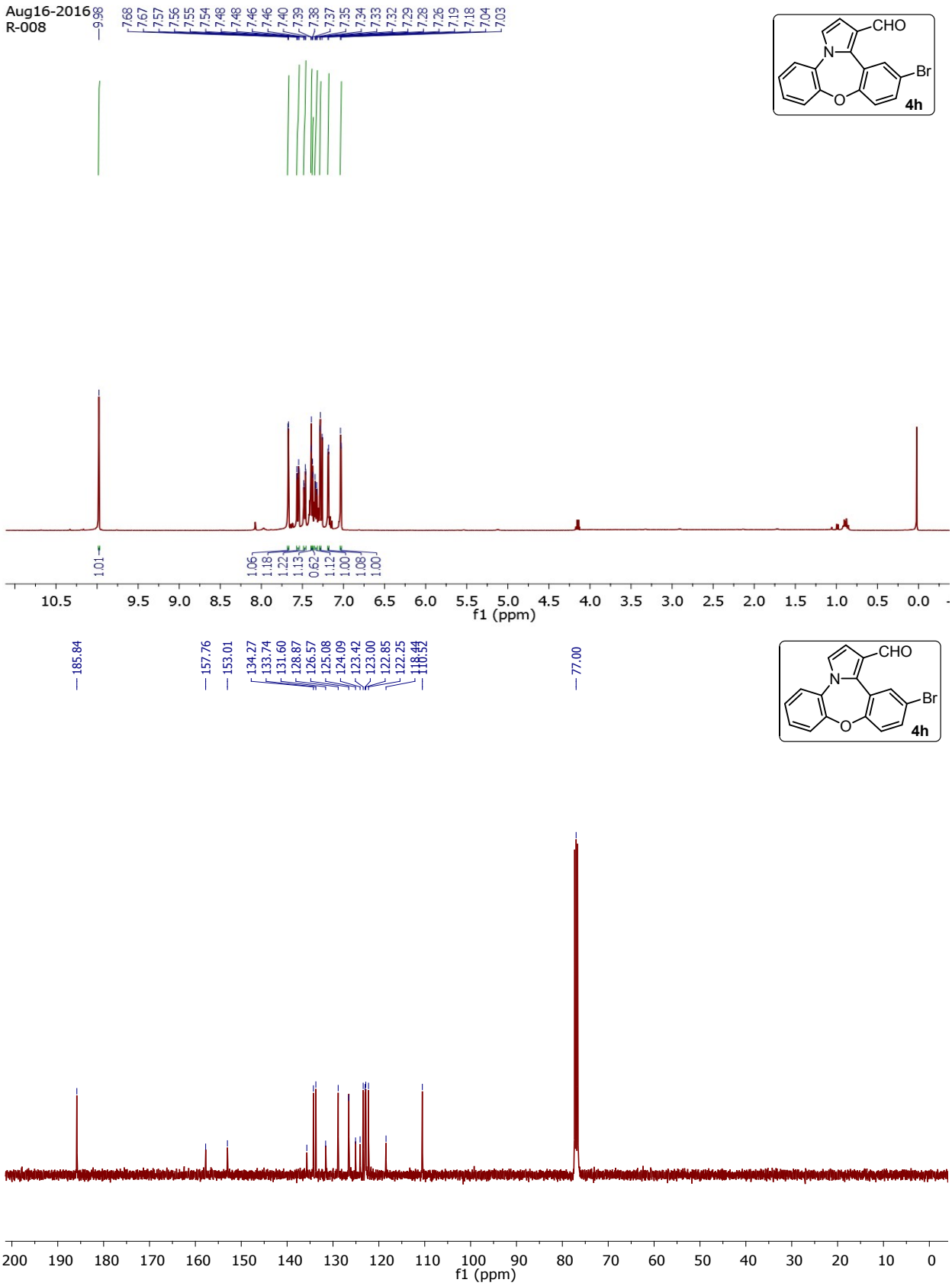
80
70
60
50
40
30
20
10
0
f1 (ppm)

CHO
N
O
4g
Br


Aug16-2016
R-008
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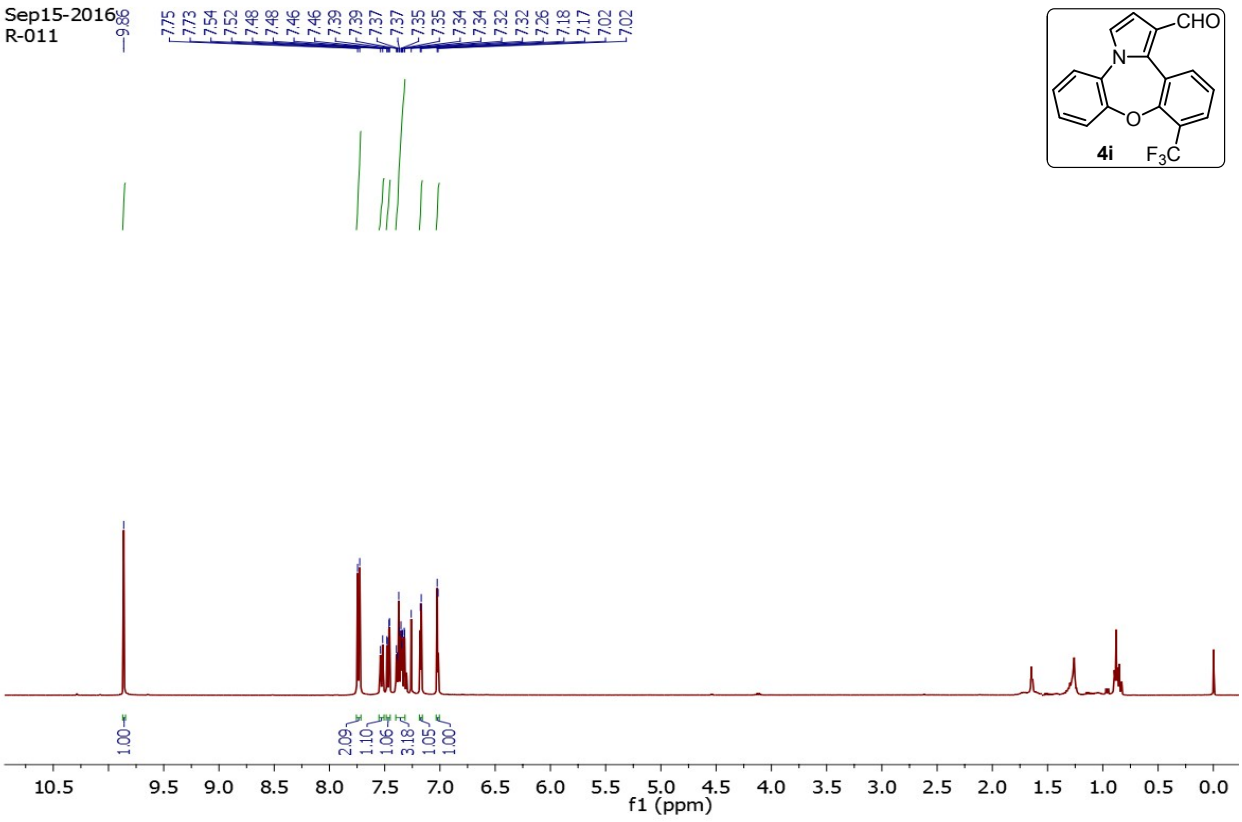
7.46
7.40
7.39
7.38
7.37
7.35
7.34
7.33
7.32
7.29
7.28
7.26
7.19
7.18
7.04
7.03
CHO
N
Br
O
4h
1.01
1.06
1.18
1.22
1.13
0.62
1.12
1.00
1.08
1.00
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
185.84
157.76
153.01
134.27
133.74
131.60
128.87
126.57
125.08
124.09
123.42
123.00
122.85
122.25
118.44
110.52
77.00
CHO
N
Br
O
4h
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Sep15-2016
R-011
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7.75
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7.52
7.48
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7.46
7.46
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7.39
7.37
7.37
7.35
7.35
7.34
7.34
7.32
7.32
7.26
7.18
7.17
7.02
7.02
CHO
N
O
4i
F3C
1.00
2.09
1.10
1.06
3.18
1.05
1.00
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
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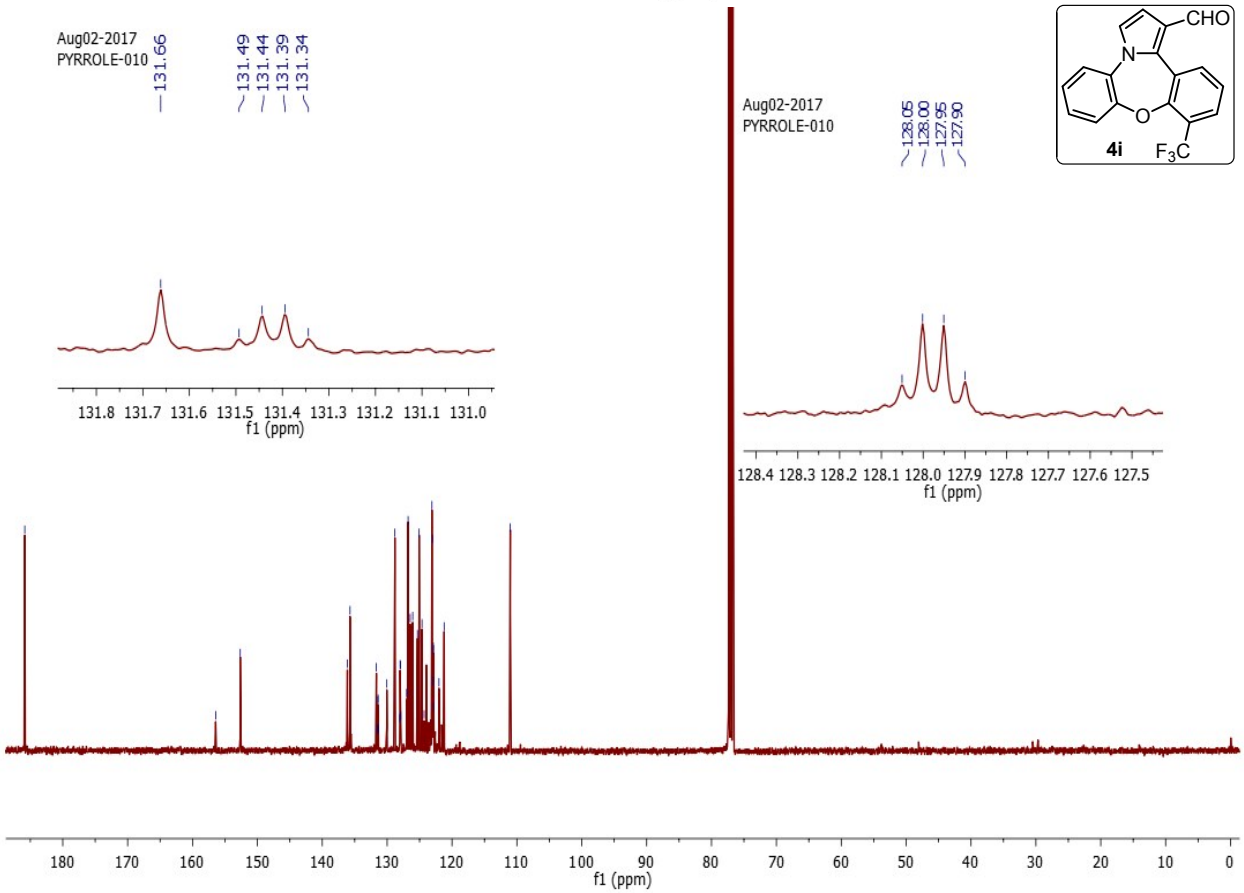
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131.34
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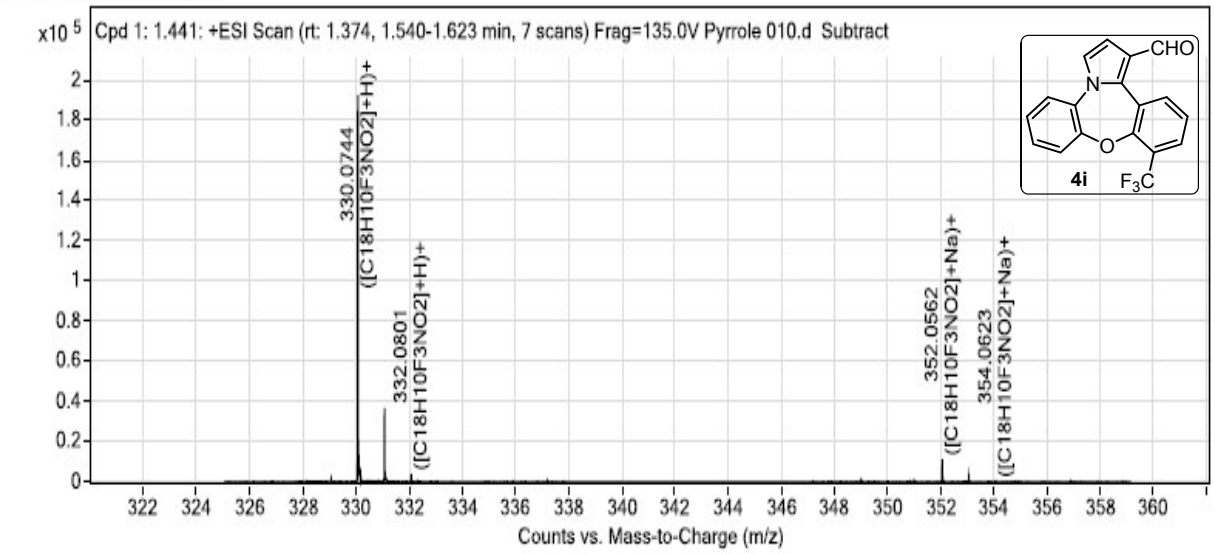
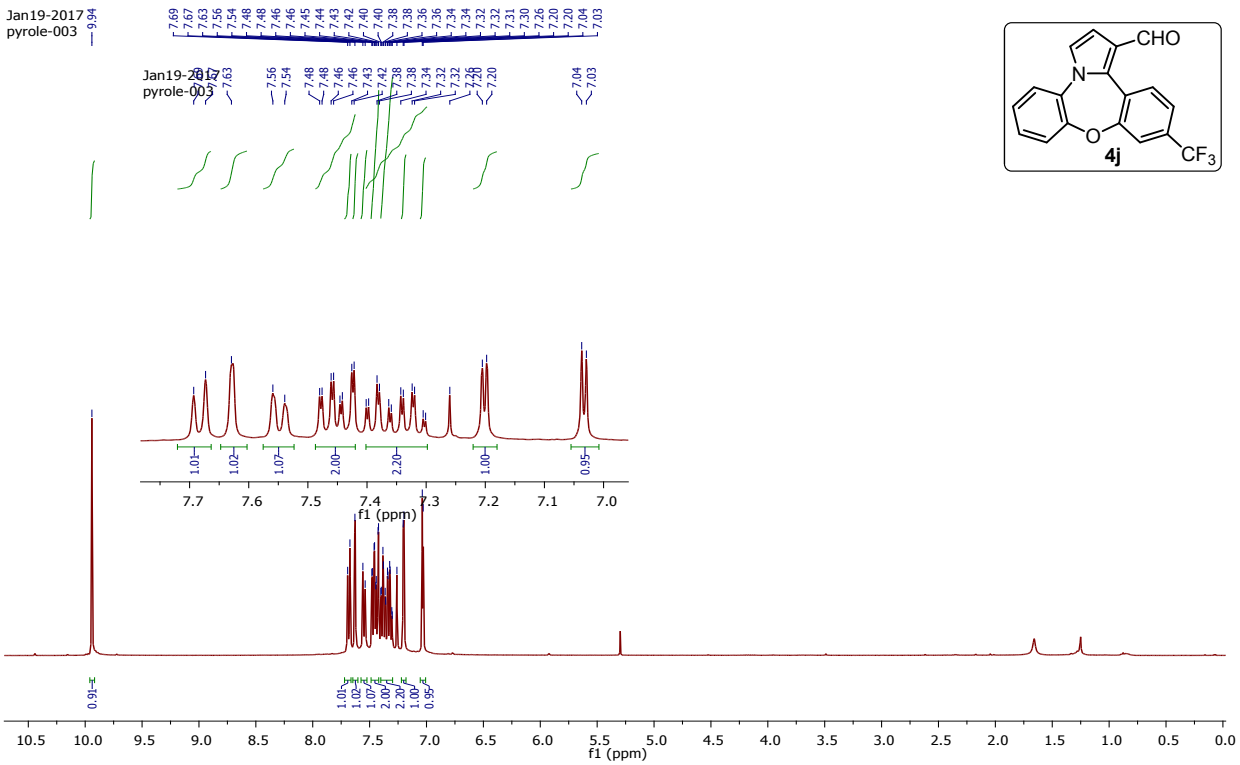
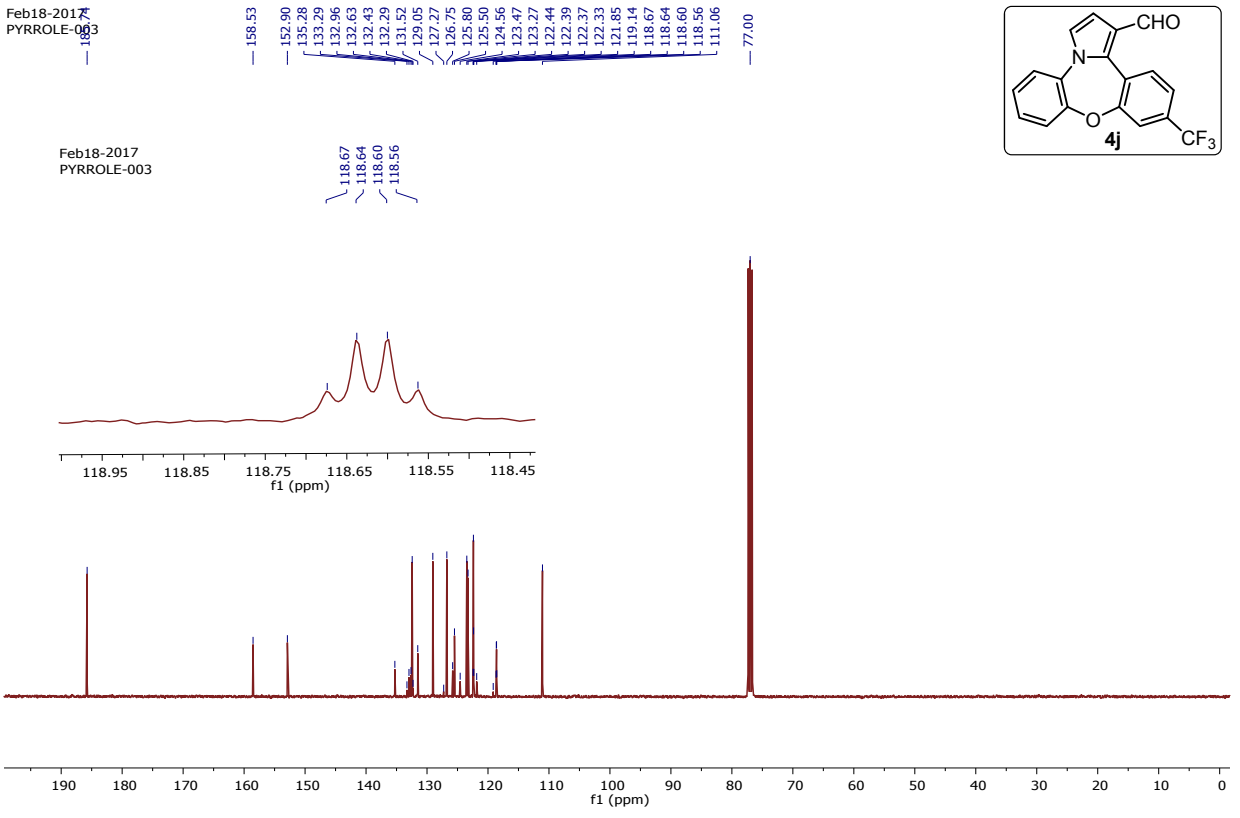
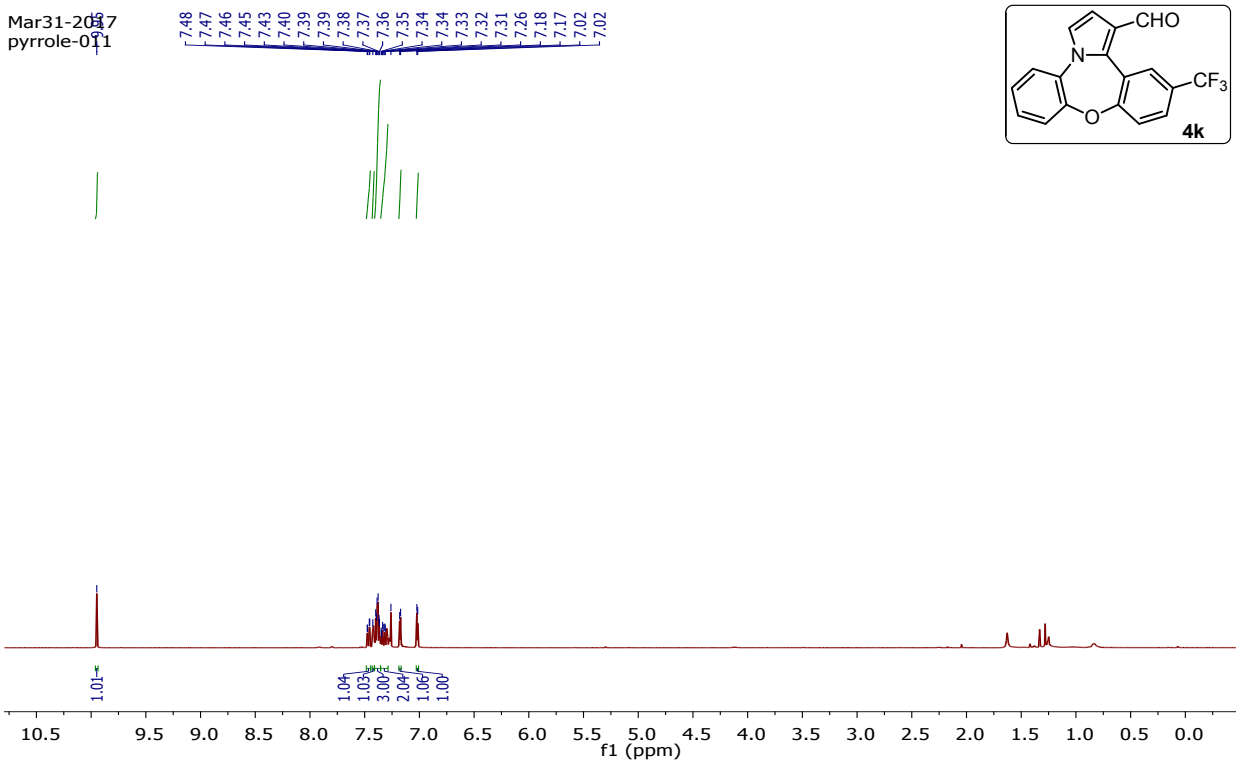
N
O
4i
F3C
131.8 131.7 131.6 131.5 131.4 131.3 131.2 131.1 131.0
f1 (ppm)
Aug02-2017
PYRROLE-010
128.05
128.00
127.95
127.90
128.4 128.3 128.2 128.1 128.0 127.9 127.8 127.7 127.6 127.5
f1 (ppm)
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

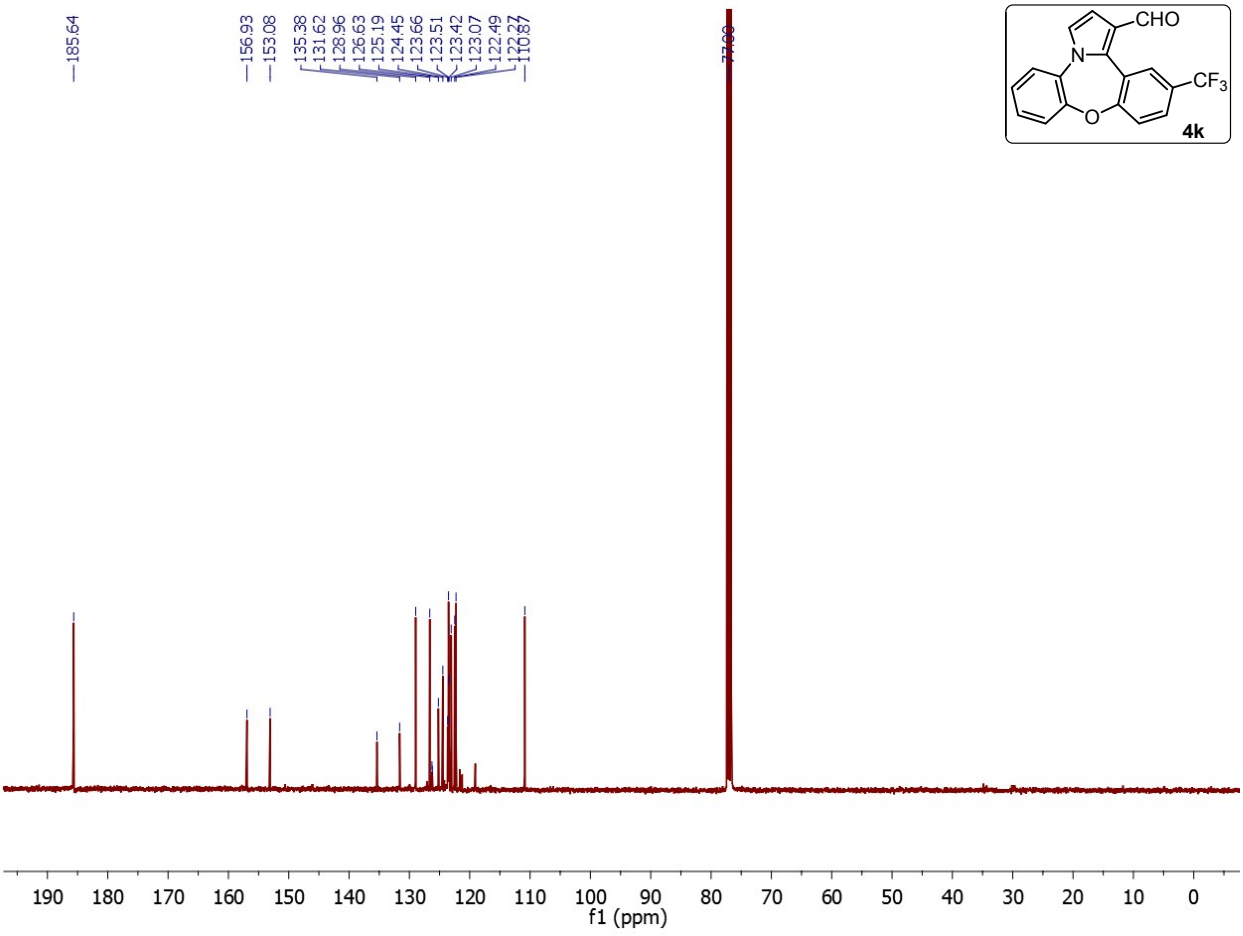
MS Zoomed Spectrum

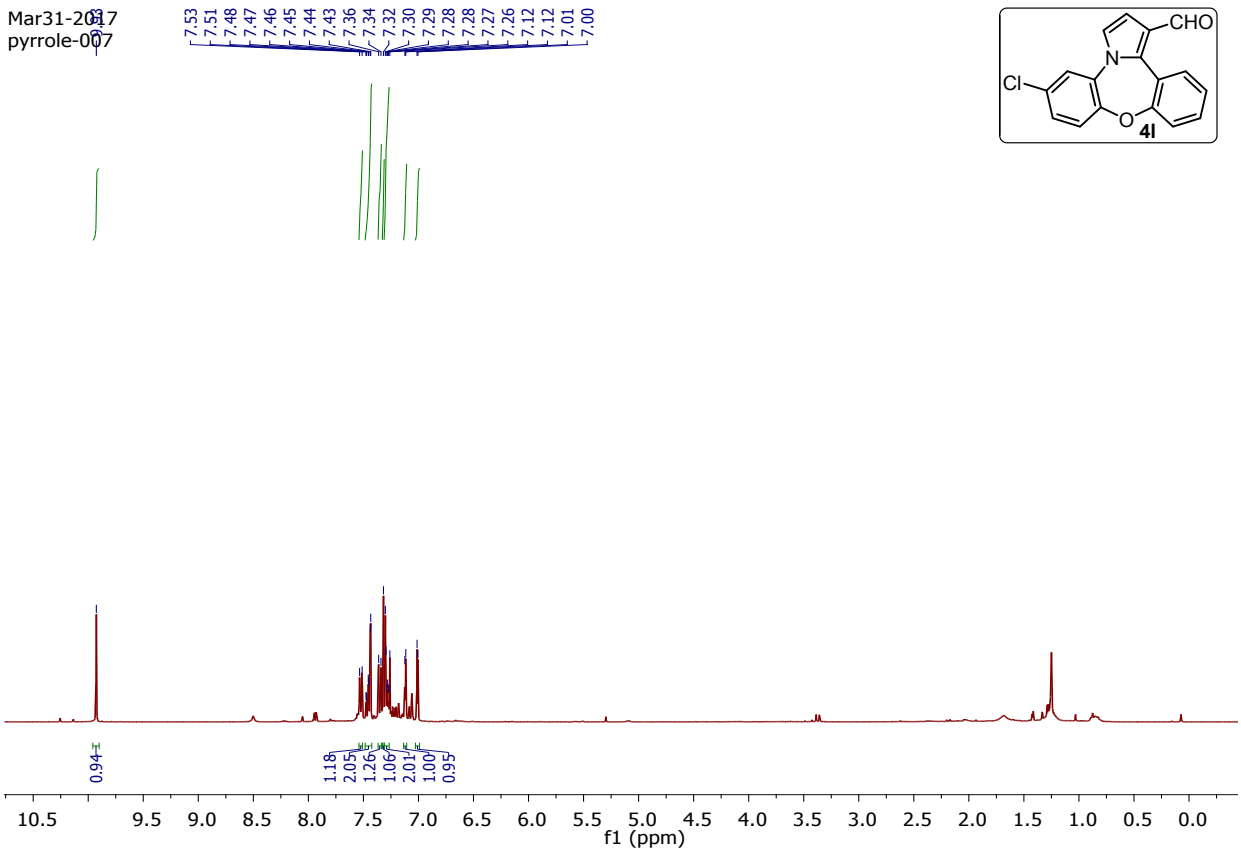


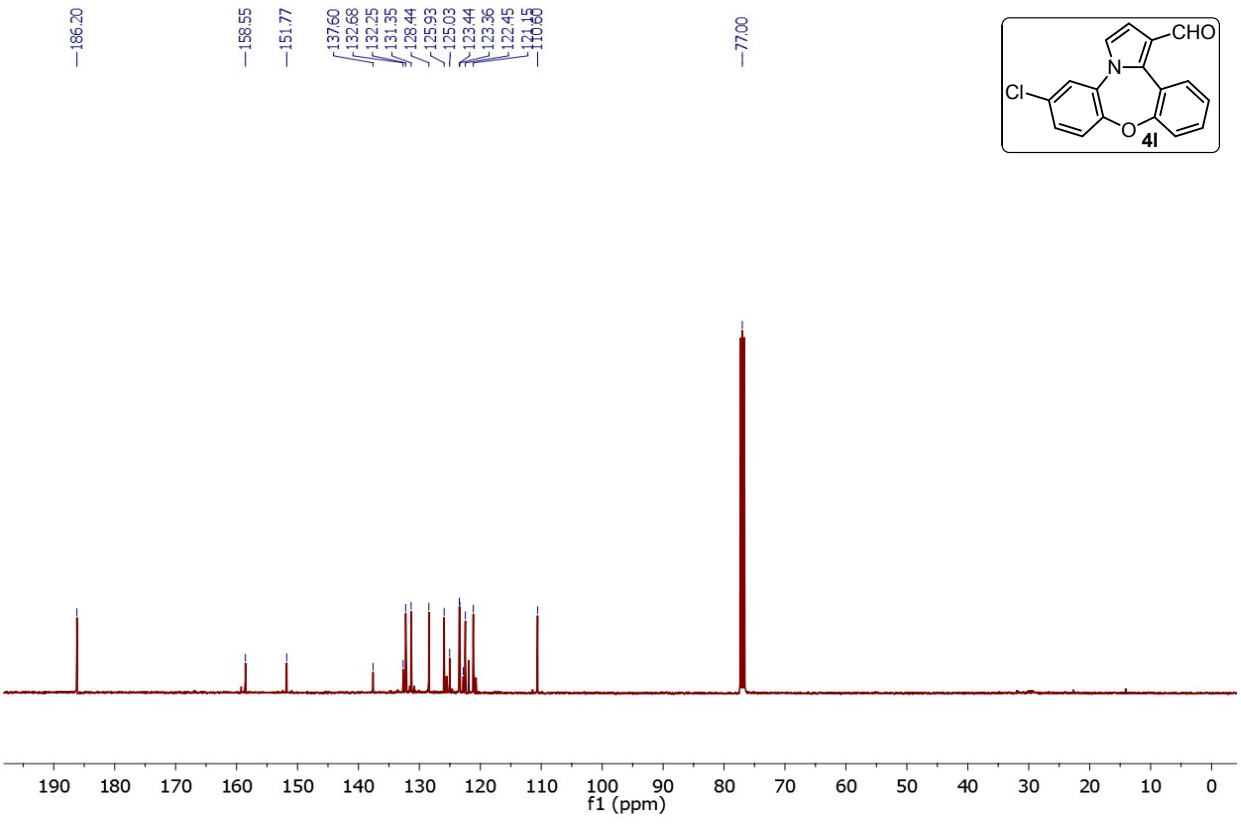

Jan19-2017
pyrole-003
CHO
N
O
CF3
4j
f1 (ppm)


Feb18-2017
PYRROLE-003
CHO
N
O
CF3
4j
f1 (ppm)

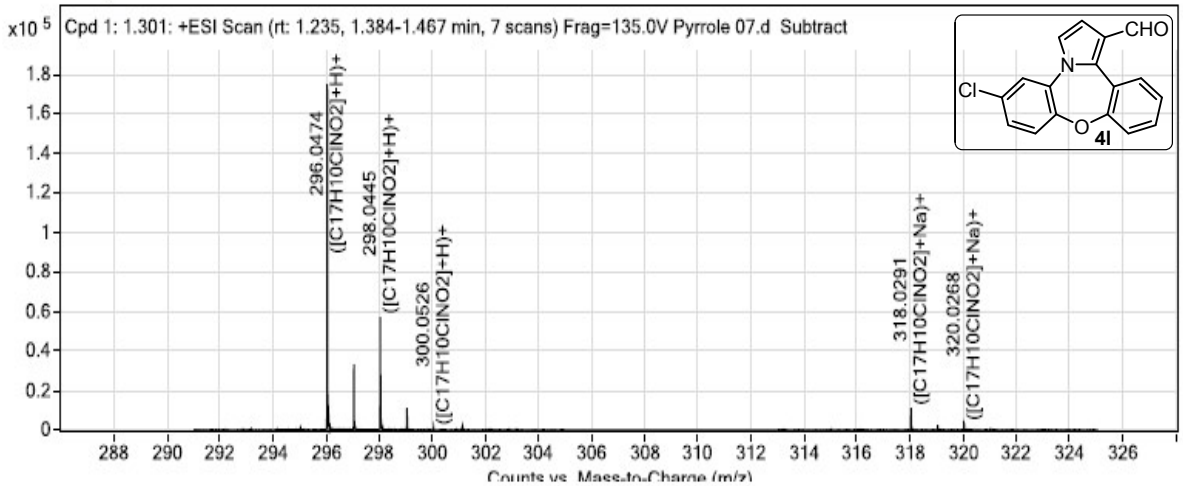

Mar31-2017
pyrrole-011
9.56
7.48
7.47
7.46
7.45
7.43
7.40
7.39
7.39
7.38
7.37
7.36
7.35
7.34
7.34
7.33
7.32
7.31
7.26
7.18
7.17
7.02
7.02
CHO
N
CF3
O
4k
1.01
1.04
1.03
3.00
2.04
1.06
1.00
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

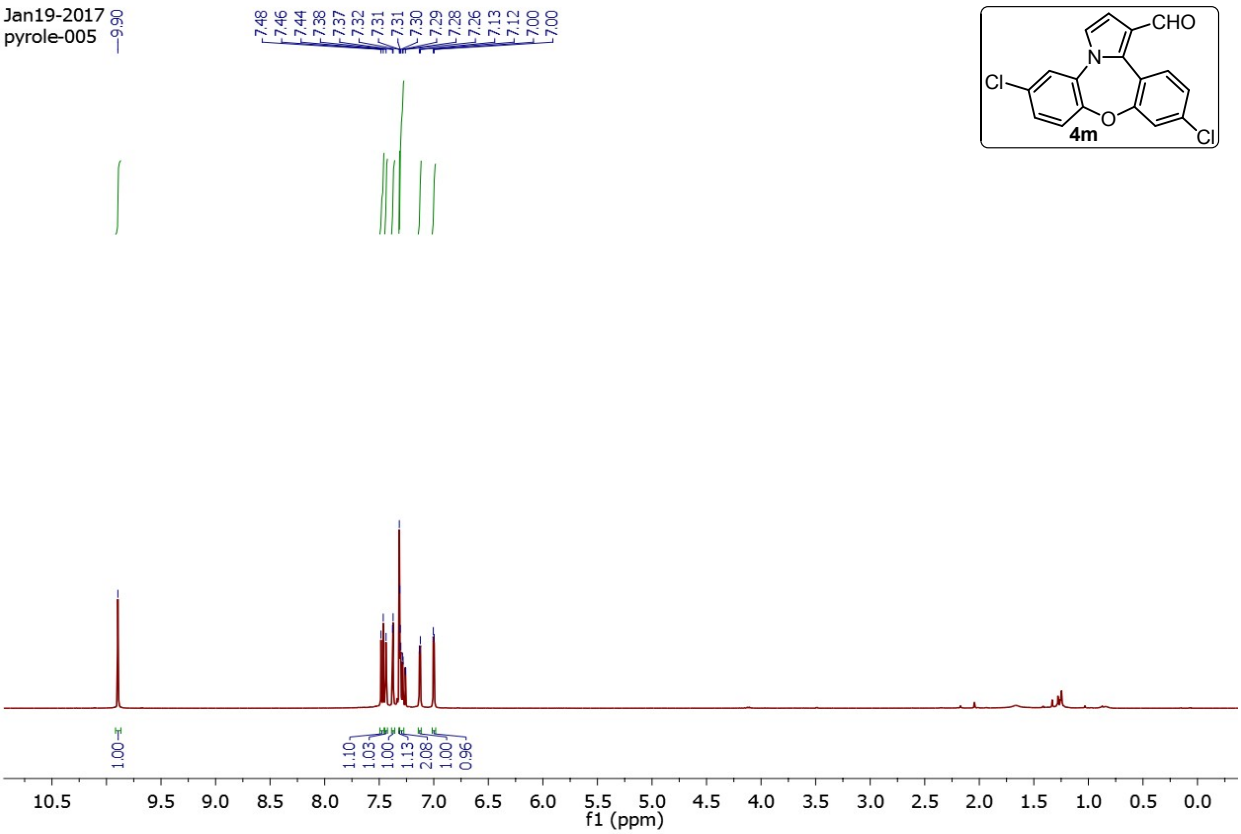

185.64
156.93
153.08
135.38
131.62
128.96
126.63
125.19
124.45
123.66
123.51
123.42
123.07
122.49
122.27
110.87
77.00
CHO
N
CF3
O
4k
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

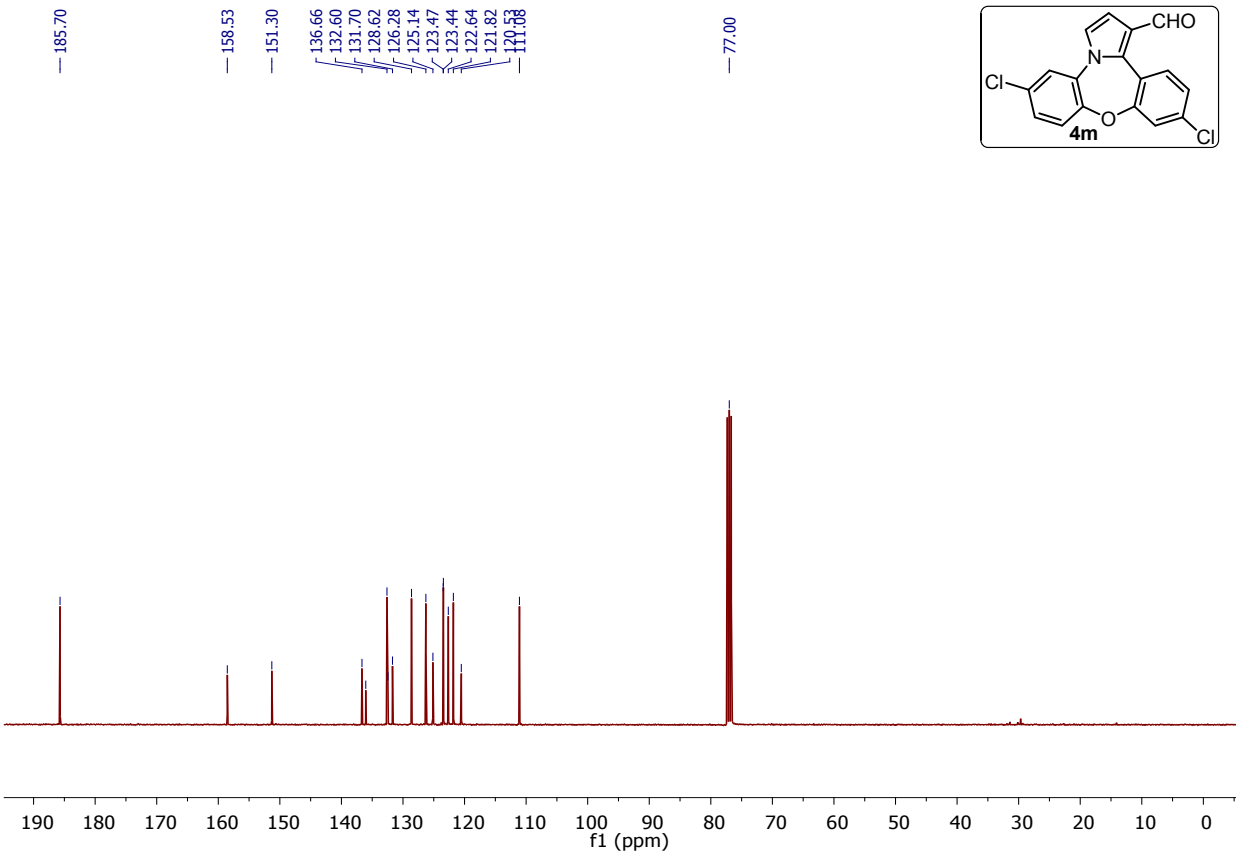

Mar31-2017
pyrrole-007
9.63
7.53
7.51
7.48
7.47
7.46
7.45
7.44
7.43
7.36
7.34
7.32
7.30
7.29
7.28
7.28
7.27
7.26
7.12
7.12
7.01
7.00
CHO
N
Cl
O
4l
0.94
1.18
2.05
1.26
1.06
2.01
1.00
0.95
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

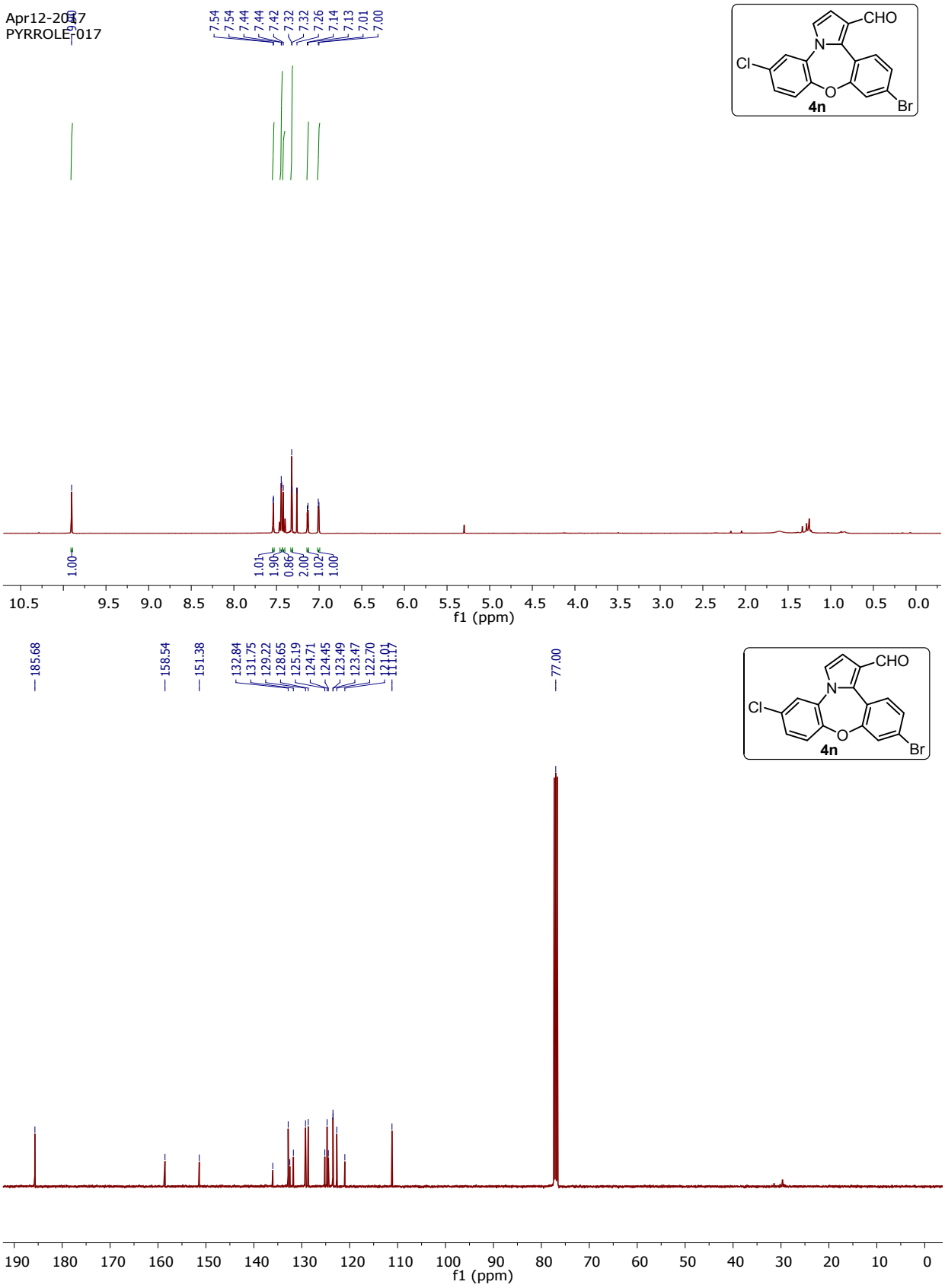

186.20
158.55
151.77
137.60
132.68
132.25
131.35
128.44
125.93
125.03
123.44
123.36
122.45
121.15
110.60
77.00
CHO
N
Cl
O
4l
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

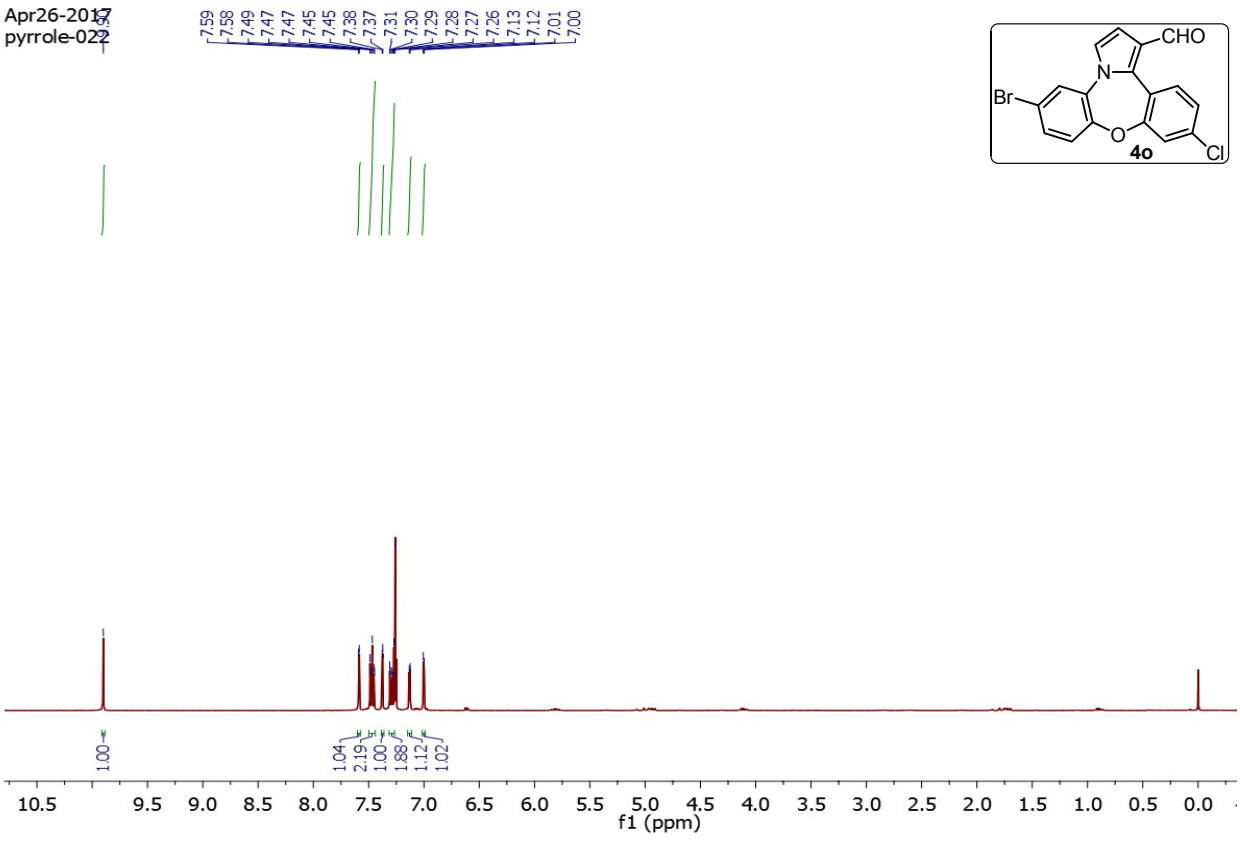
MS Zoomed Spectrum

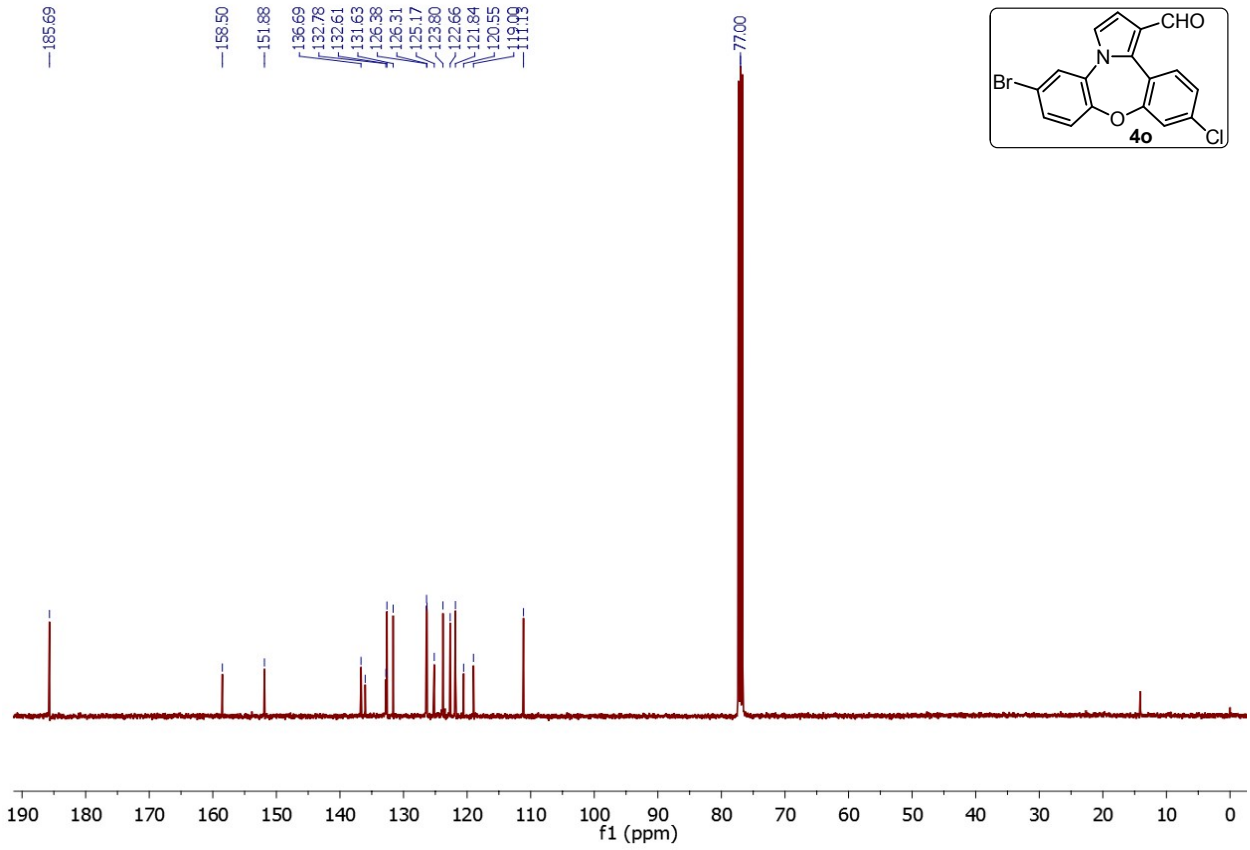


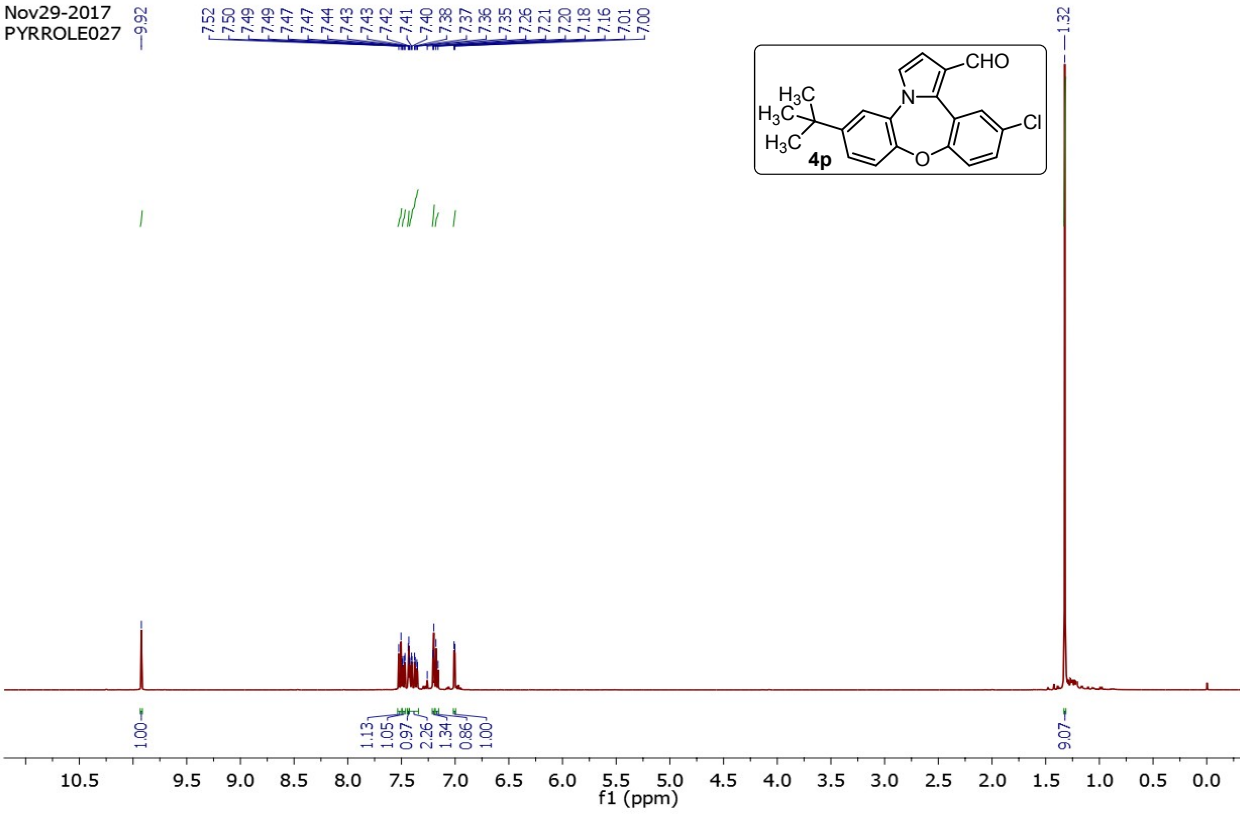

Jan19-2017
pyrole-005
9.90
7.48
7.46
7.44
7.38
7.37
7.32
7.31
7.31
7.30
7.29
7.28
7.26
7.13
7.12
7.00
7.00
CHO
N
Cl
O
Cl
4m
1.00
1.10
1.03
1.00
1.13
2.08
1.00
0.96
f1 (ppm)

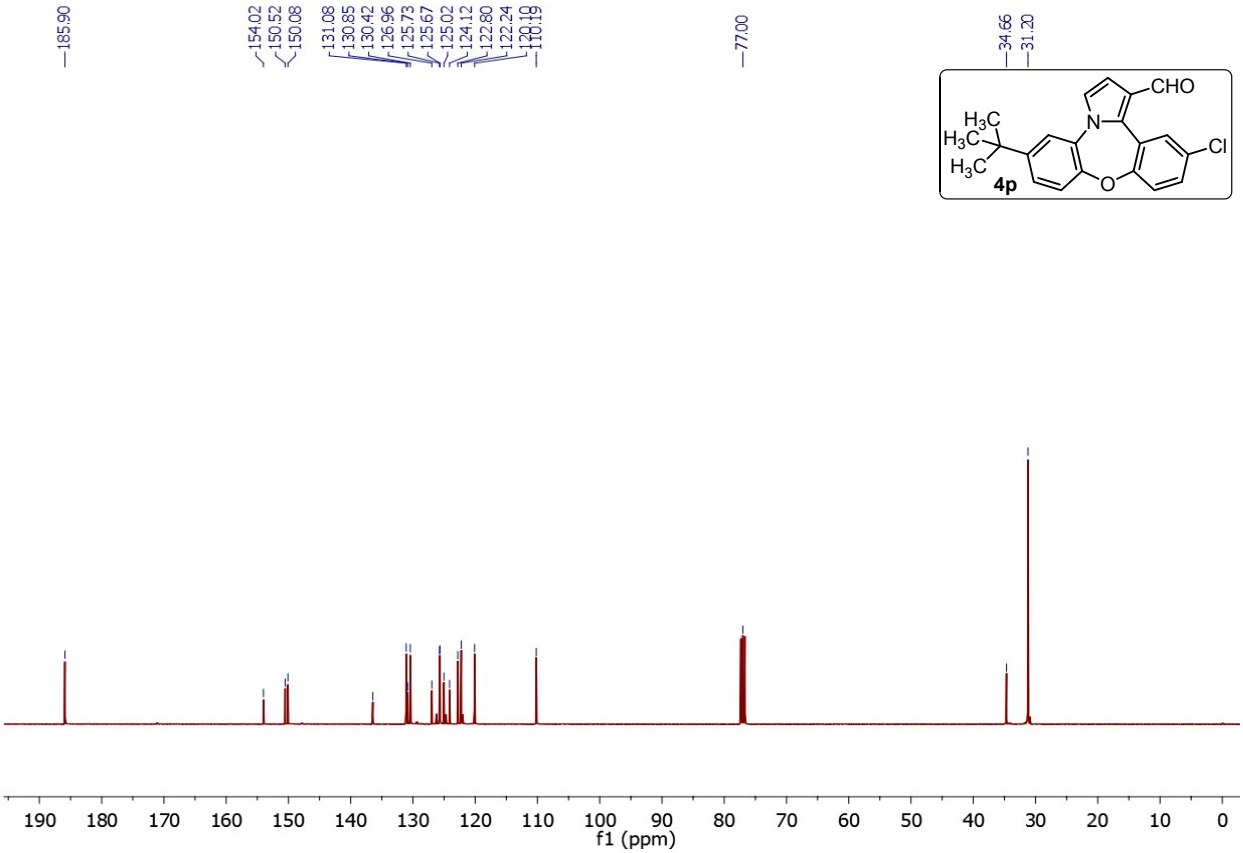

185.70
158.53
151.30
136.66
132.60
131.70
128.62
126.28
125.14
123.47
123.44
122.64
121.82
120.53
111.08
77.00
CHO
N
Cl
O
Cl
4m
f1 (ppm)

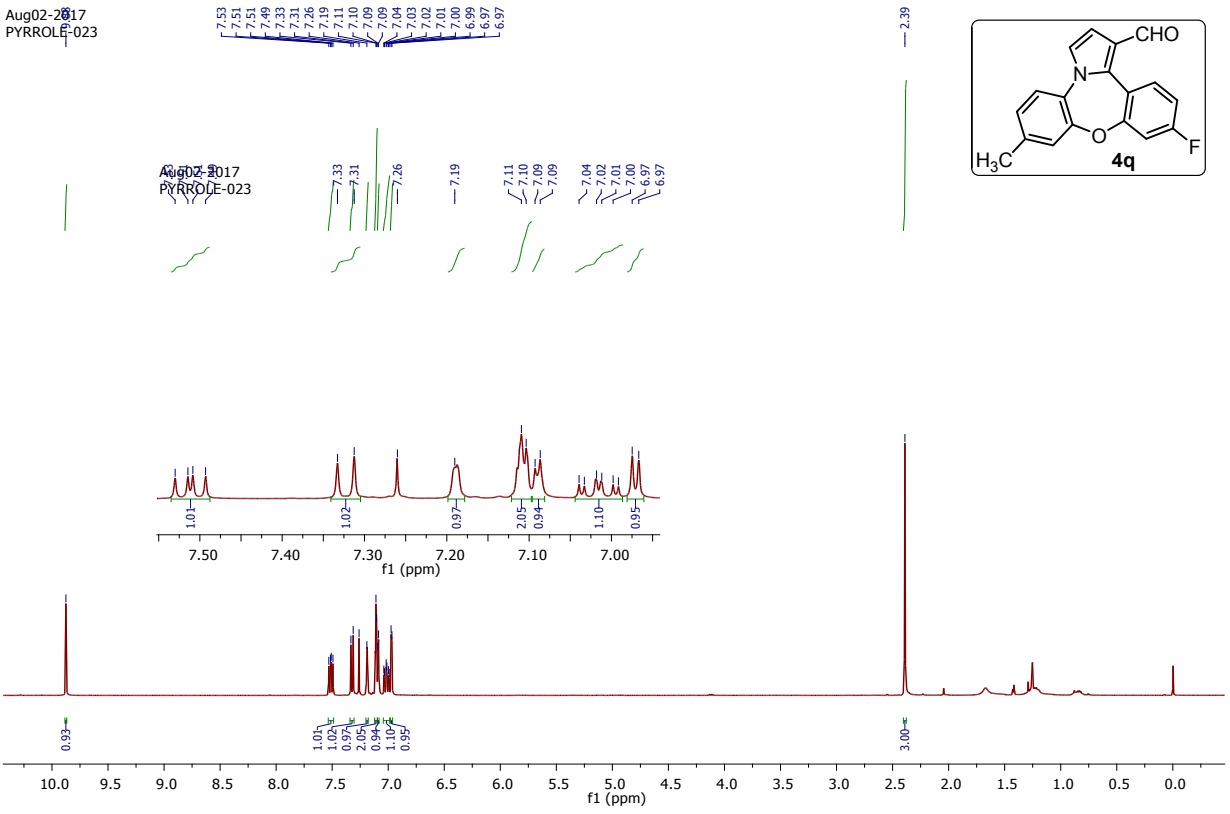

Apr12-2017
PYRROLE-017
7.54
7.54
7.44
7.44
7.42
7.32
7.32
7.26
7.14
7.13
7.01
7.00
CHO
N
Cl
O
Br
4n
1.00
1.01
1.90
0.86
2.00
1.02
1.00
f1 (ppm)
185.68
158.54
151.38
132.84
131.75
129.22
128.65
125.19
124.71
124.45
123.49
123.47
122.70
111.17
77.00
f1 (ppm)

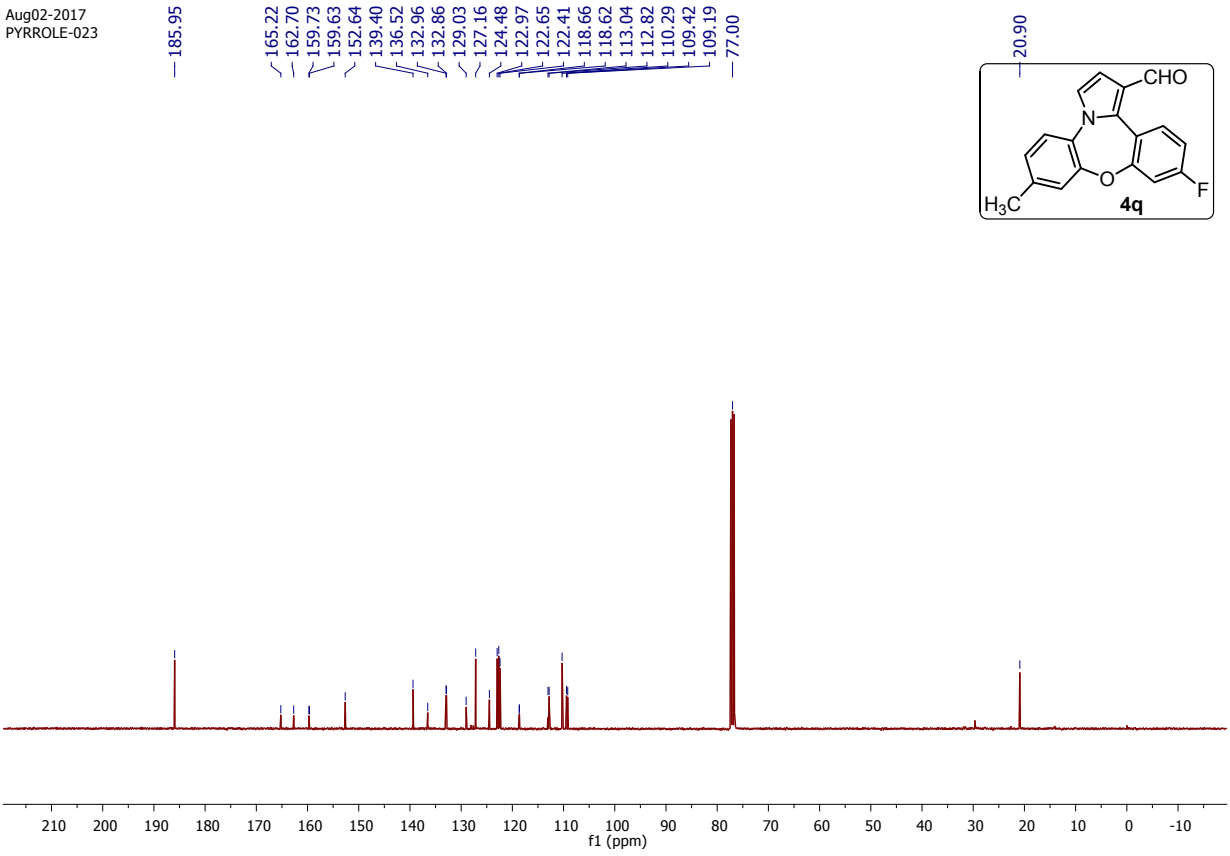

Apr26-2017
pyrrole-022
7.59
7.58
7.49
7.47
7.47
7.45
7.45
7.38
7.37
7.31
7.30
7.29
7.28
7.27
7.26
7.13
7.12
7.01
7.00
CHO
N
Br
O
Cl
4o
1.00
1.04
2.19
1.00
1.88
1.12
1.02
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


185.69
158.50
151.88
136.69
132.78
132.61
131.63
126.38
126.31
125.17
123.80
122.66
121.84
120.55
119.09
111.13
77.00
CHO
N
Br
O
Cl
4o
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

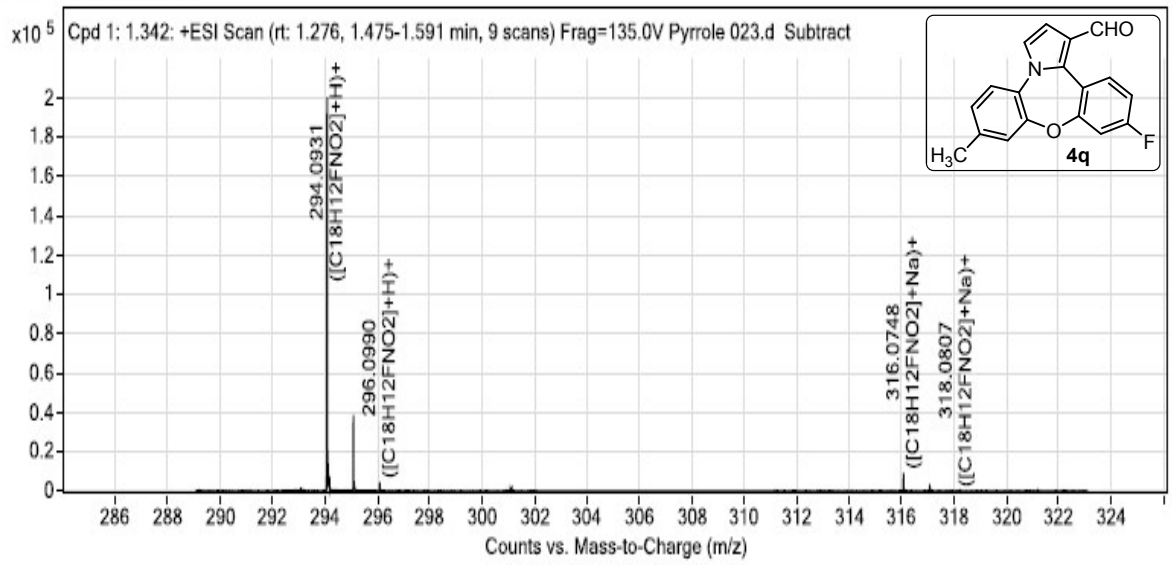

Nov29-2017
PYRROLE027
9.92
7.52
7.50
7.49
7.49
7.47
7.47
7.44
7.43
7.43
7.42
7.41
7.40
7.38
7.37
7.36
7.35
7.26
7.21
7.20
7.18
7.16
7.01
7.00
1.32
CHO
N
Cl
O
H3C
H3C
H3C
4p
1.00
1.13
1.05
0.97
2.26
1.34
0.86
1.00
9.07
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

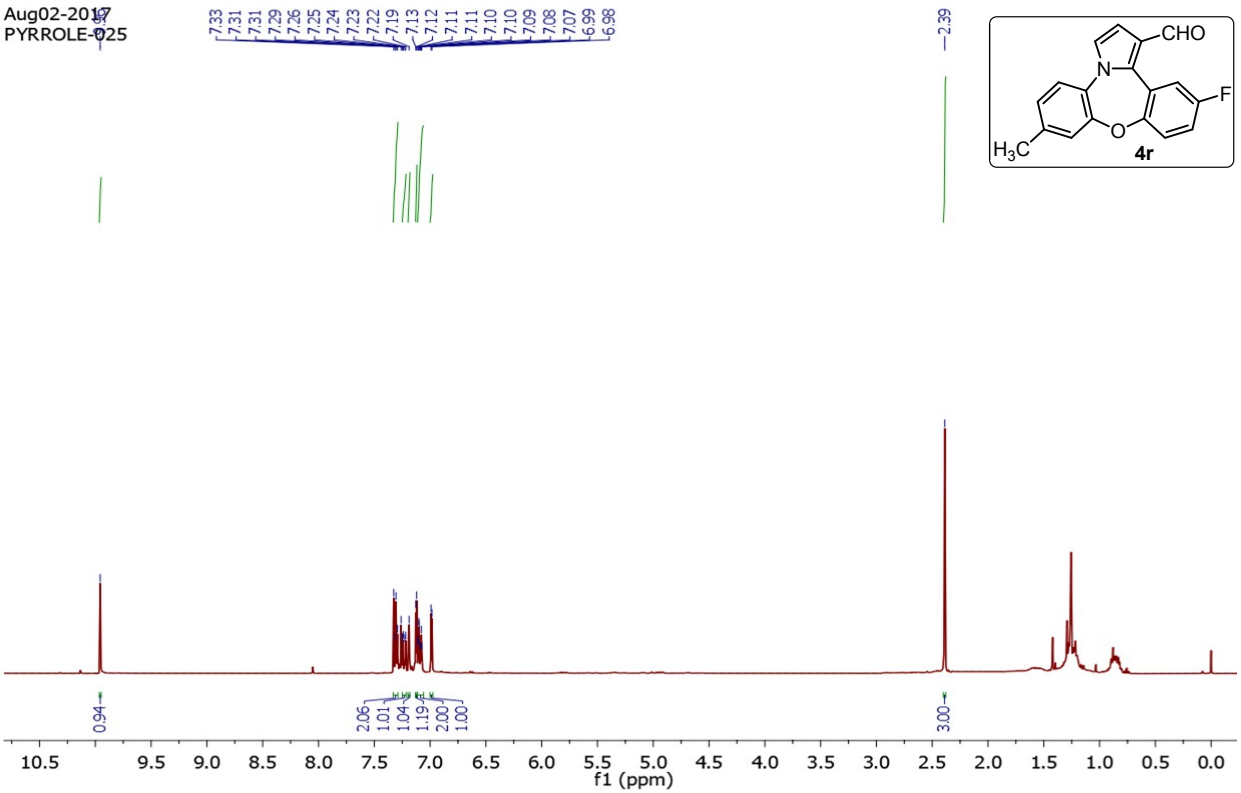

185.90
154.02
150.52
150.08
131.08
130.85
130.42
126.96
125.73
125.67
125.02
124.12
122.80
122.24
120.10
119.19
77.00
34.66
31.20
CHO
N
Cl
O
H3C
H3C
H3C
4p
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

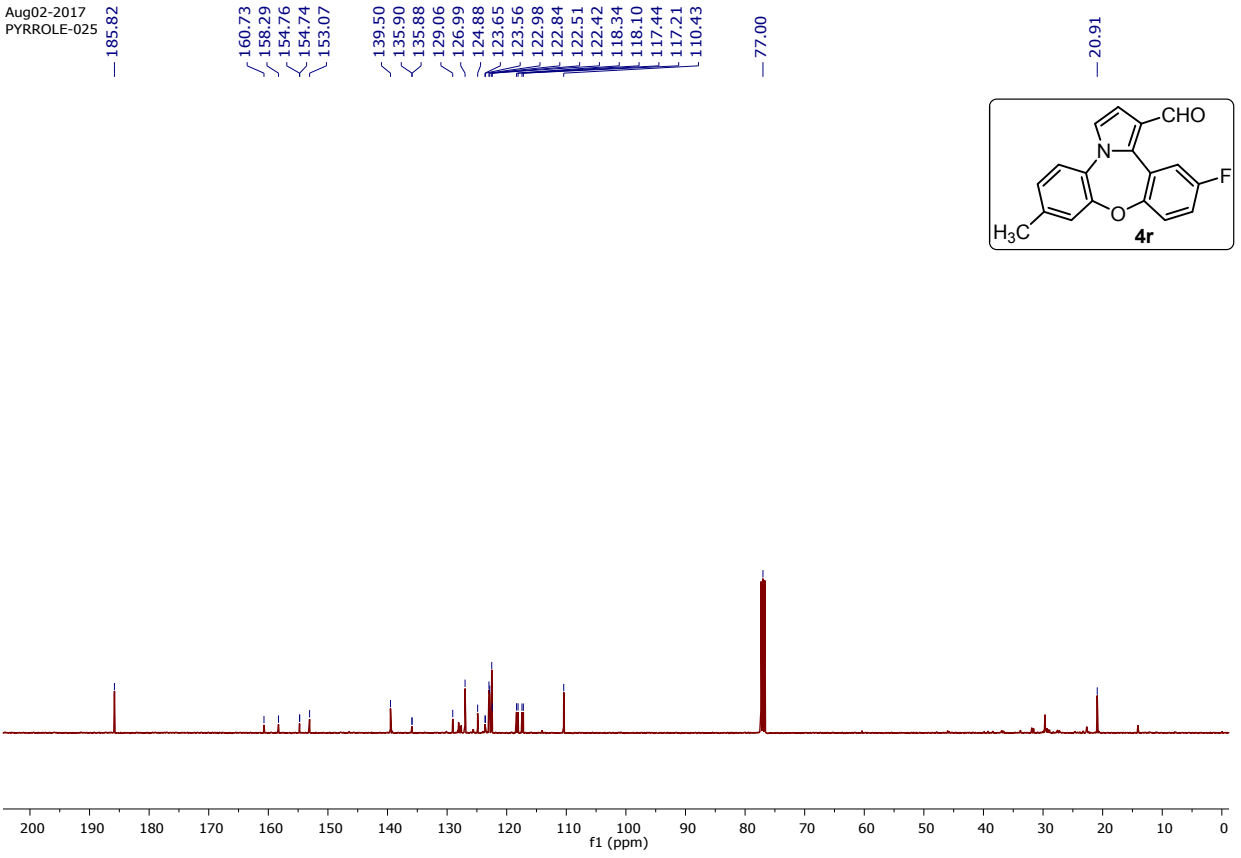

Aug02-2017
PYRROLE-023
CHO
N
O
F
H3C
4q
f1 (ppm)

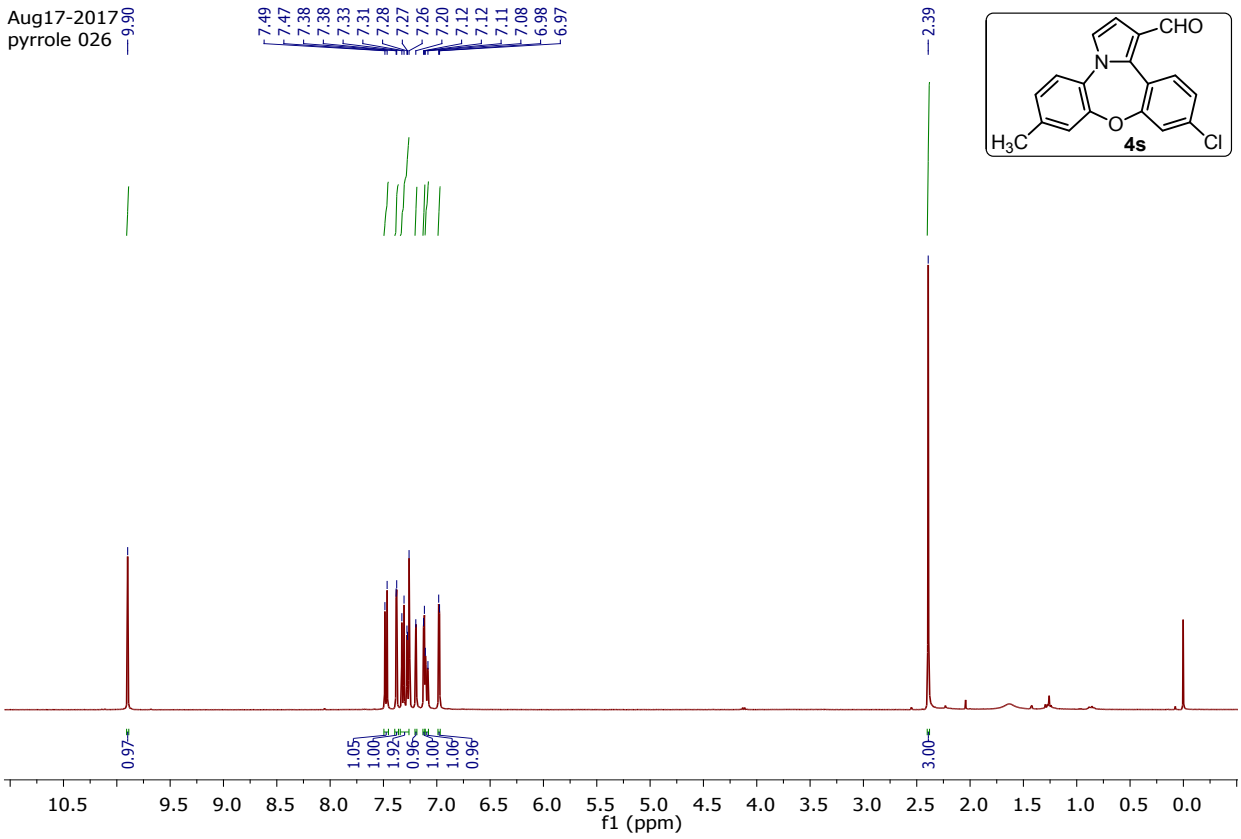

Aug02-2017
PYRROLE-023
CHO
N
O
F
H3C
4q
f1 (ppm)

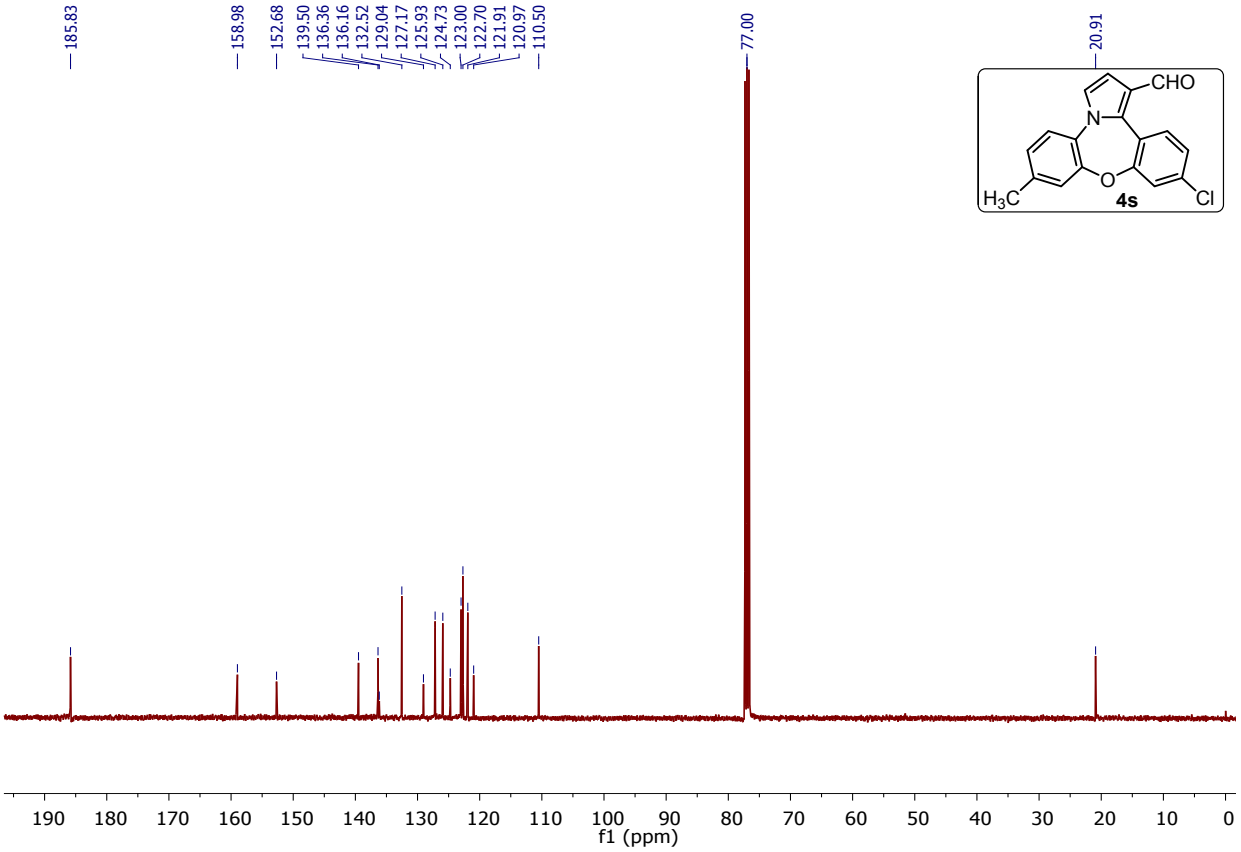
MS Zoomed Spectrum

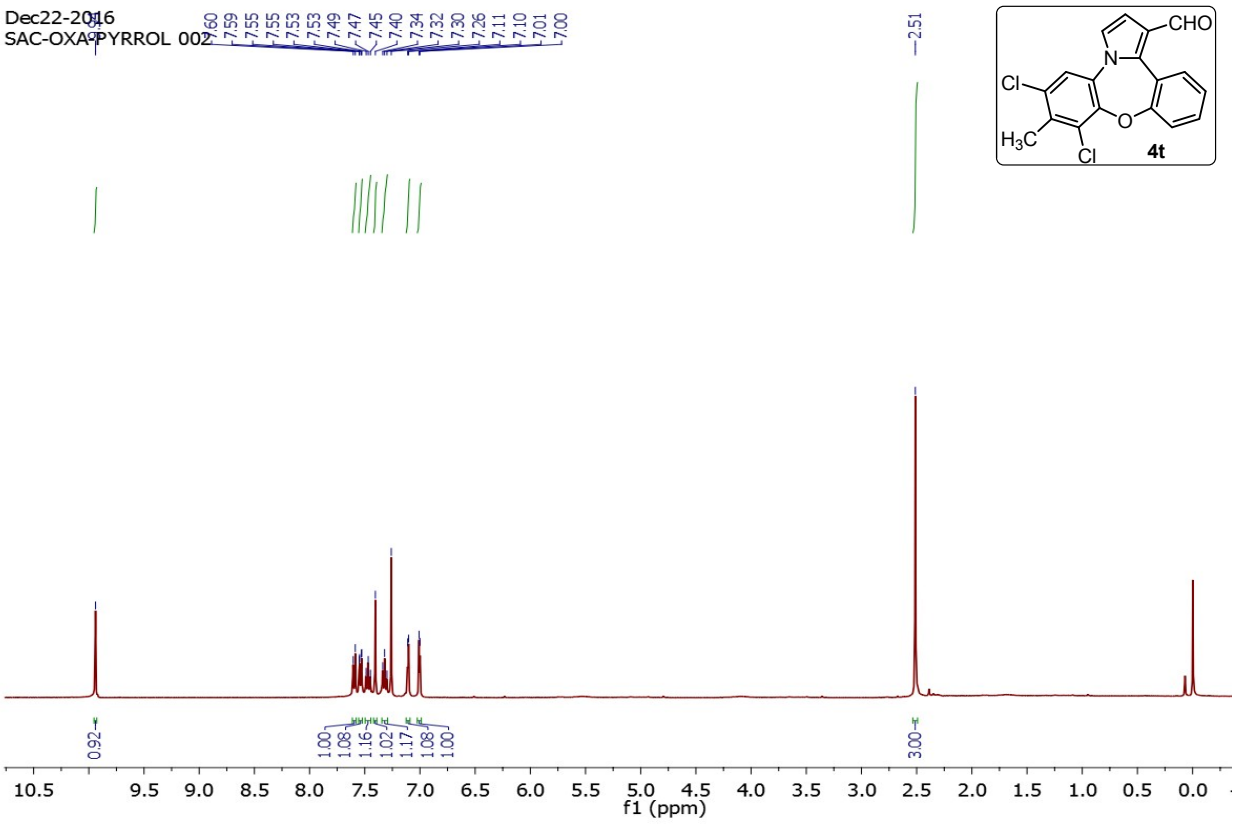


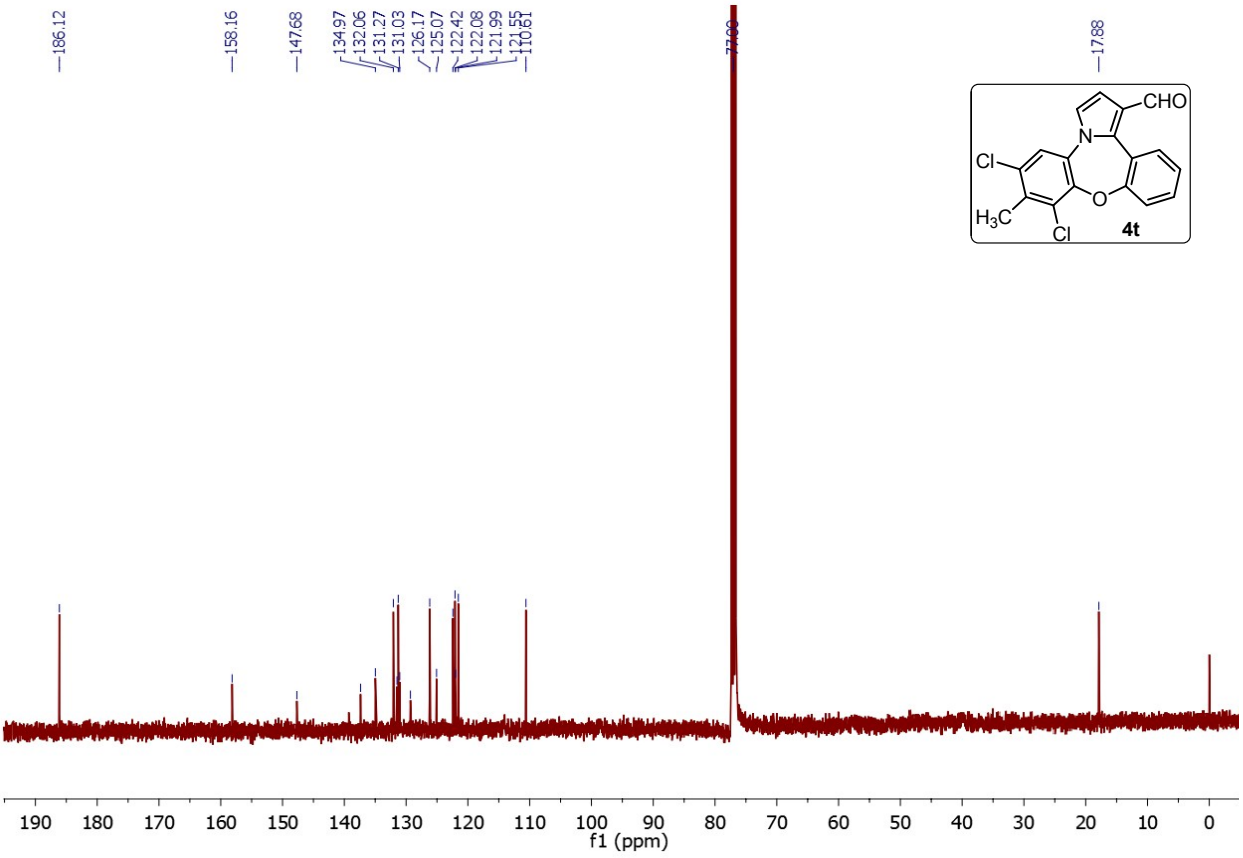

Aug02-2017
PYRROLE-025
9.87
7.33
7.31
7.31
7.29
7.26
7.25
7.24
7.23
7.22
7.19
7.13
7.12
7.11
7.11
7.10
7.10
7.09
7.08
7.07
6.99
6.98
2.39
CHO
N
F
O
H3C
4r
0.94
2.06
1.01
1.04
1.19
2.00
1.00
3.00
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

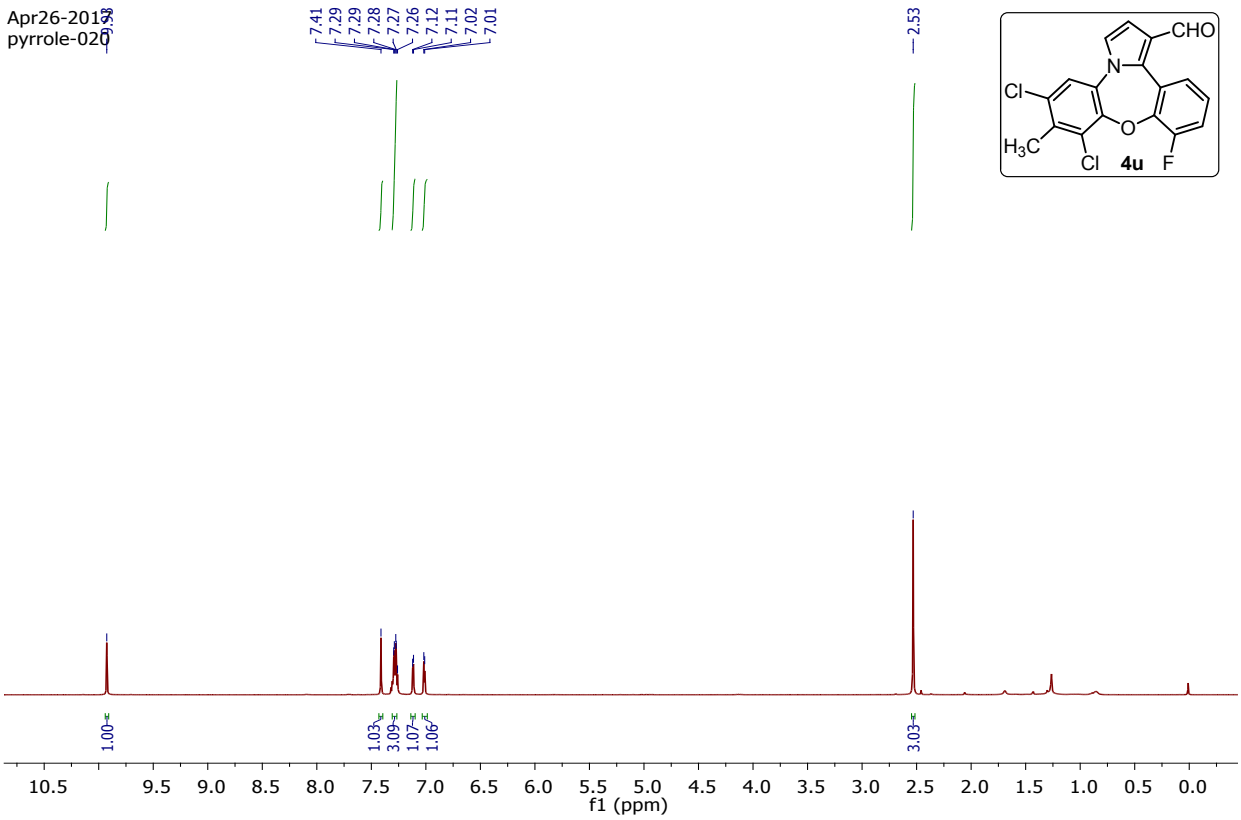

Aug02-2017
PYRROLE-025
185.82
160.73
158.29
154.76
154.74
153.07
139.50
135.90
135.88
129.06
126.99
124.88
123.65
123.56
122.98
122.84
122.51
122.42
118.34
118.10
117.44
117.21
110.43
77.00
20.91
CHO
N
F
O
H3C
4r
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

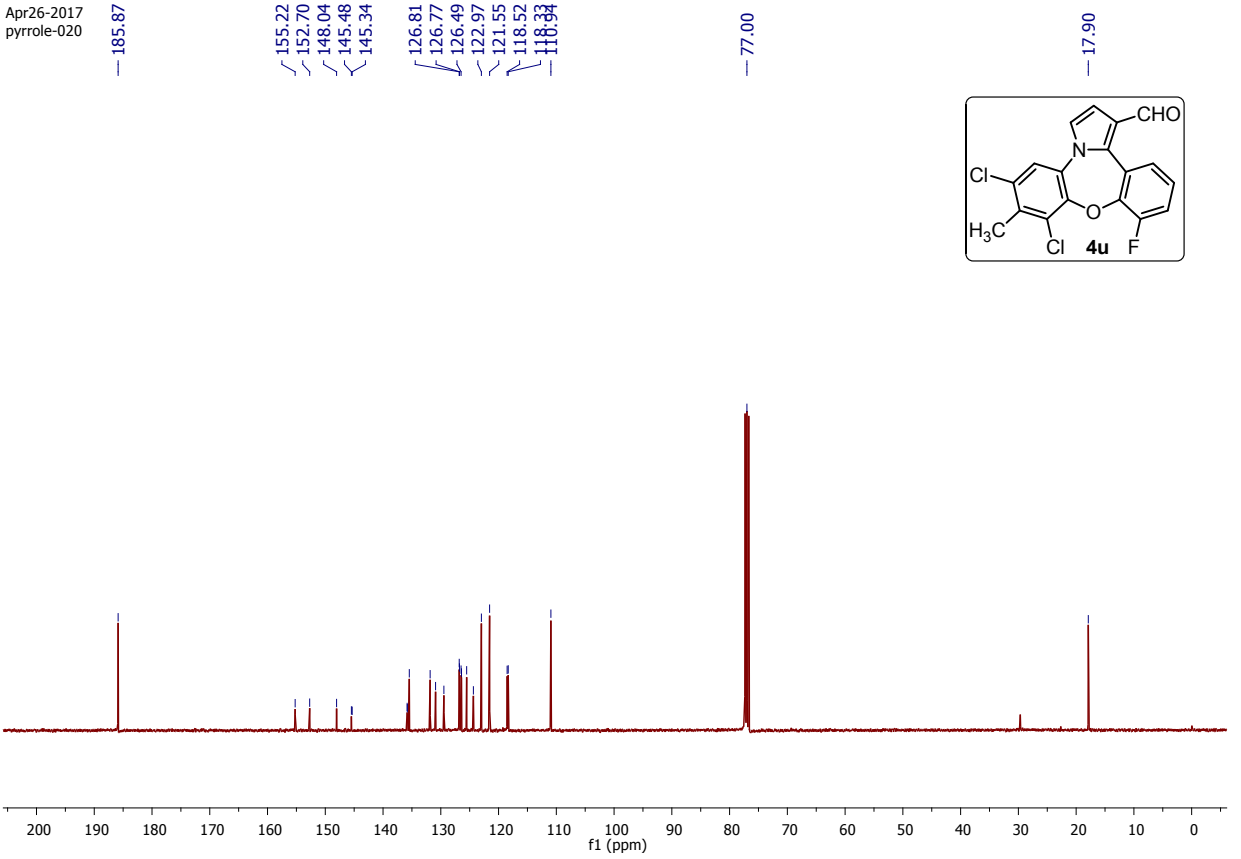

Aug17-2017
pyrrole 026
9.90
7.49
7.47
7.38
7.38
7.33
7.31
7.28
7.27
7.26
7.20
7.12
7.12
7.11
7.08
6.98
6.97
2.39
CHO
N
O
H3C
Cl
4s
0.97
1.05
1.00
1.92
0.96
1.00
1.06
0.96
3.00
f1 (ppm)

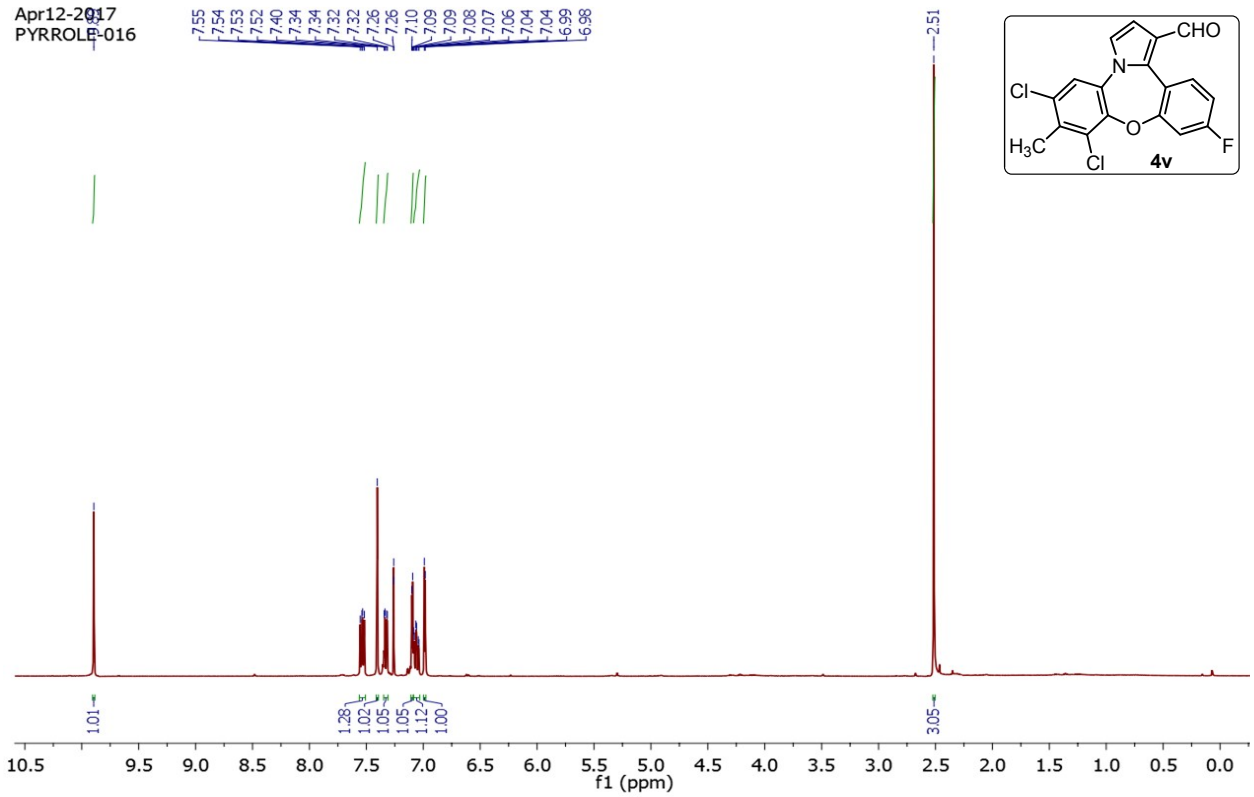

185.83
158.98
152.68
139.50
136.36
136.16
132.52
129.04
127.17
125.93
124.73
123.00
122.70
121.91
120.97
110.50
77.00
20.91
CHO
N
O
H3C
Cl
4s
f1 (ppm)

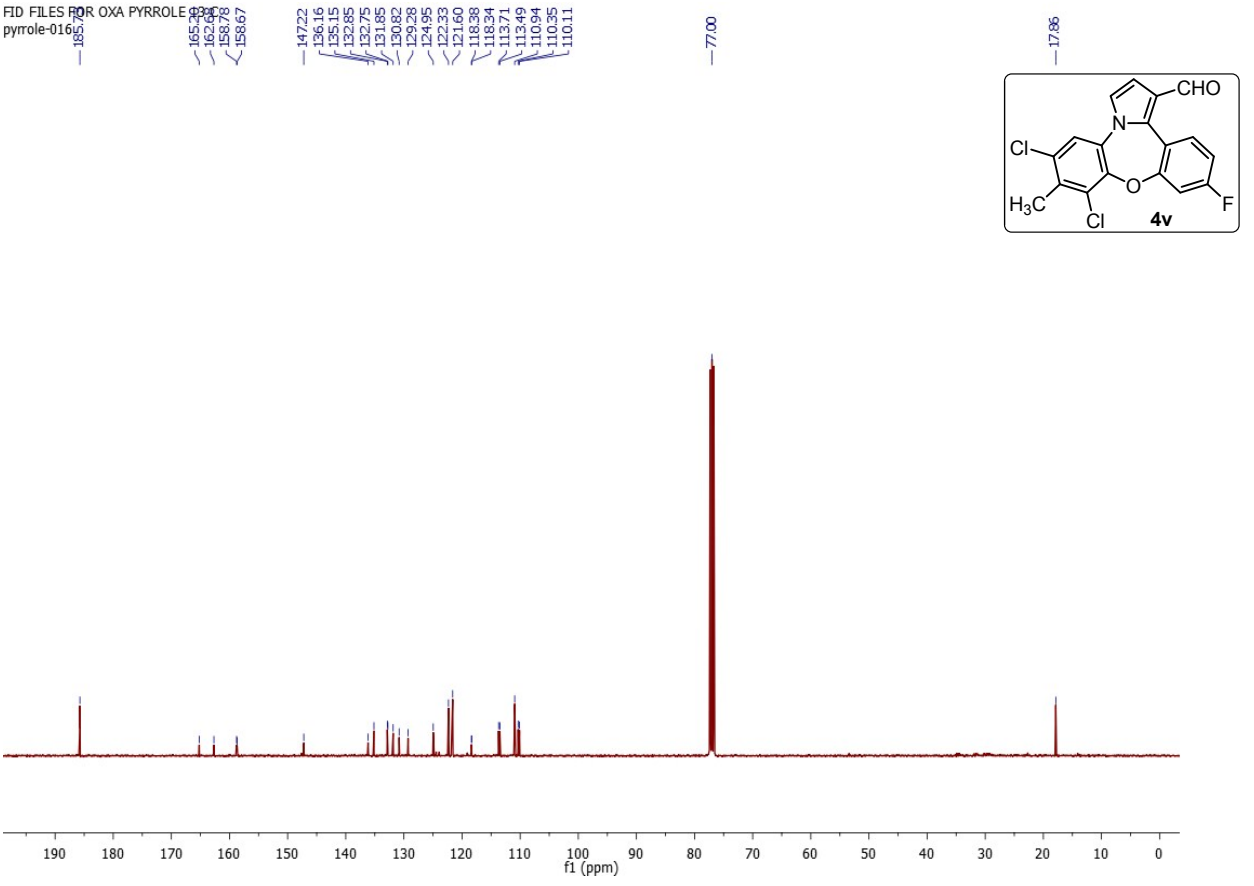

Dec22-2016
SAC-OXA-PYRROL 002
9.94
7.60
7.59
7.55
7.55
7.53
7.53
7.49
7.47
7.45
7.40
7.34
7.32
7.30
7.26
7.11
7.10
7.01
7.00
2.51
CHO
N
Cl
O
H3C
Cl
4t
0.92
1.00
1.08
1.16
1.02
1.17
1.08
1.00
3.00
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


186.12
158.16
147.68
134.97
132.06
131.27
131.03
126.17
125.07
122.42
122.08
121.99
77.00
17.88
CHO
N
Cl
O
H3C
Cl
4t
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Apr26-2017
pyrrole-020
9.58
7.41
7.29
7.29
7.28
7.27
7.26
7.12
7.11
7.02
7.01
2.53
CHO
N
Cl
O
H3C
Cl
4u
F
1.00
1.03
3.09
1.07
1.06
3.03
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


Apr26-2017
pyrrole-020
185.87
155.22
152.70
148.04
145.48
145.34
126.81
126.77
126.49
122.97
121.55
118.52
118.33
110.34
77.00
17.90
CHO
N
Cl
O
H3C
Cl
4u
F
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Apr12-2017
PYRROLE-016
7.55
7.54
7.53
7.52
7.40
7.34
7.34
7.32
7.32
7.26
7.26
7.10
7.09
7.09
7.08
7.07
7.06
7.04
7.04
6.99
6.98
2.51
CHO
N
Cl
O
F
H3C
Cl
4v
1.01
1.28
1.02
1.05
1.05
1.12
1.00
3.05
f1 (ppm)


FID FILES FOR OXA PYRROLE 4336
pyrrole-016
185.78
165.43
162.68
158.78
158.67
147.22
136.16
135.15
132.85
132.75
131.85
130.82
129.28
124.95
122.33
121.60
118.38
118.34
113.71
113.49
110.94
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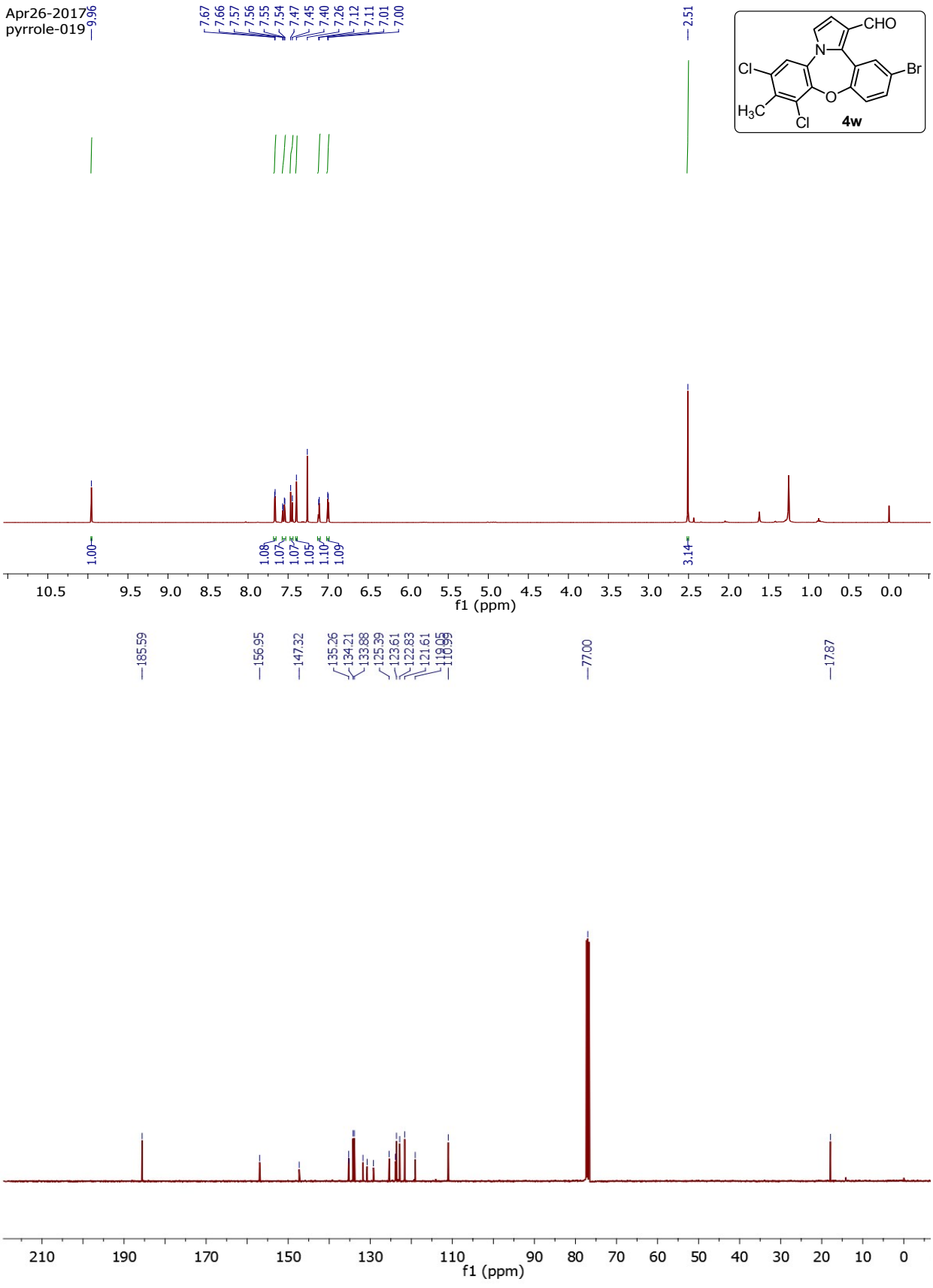
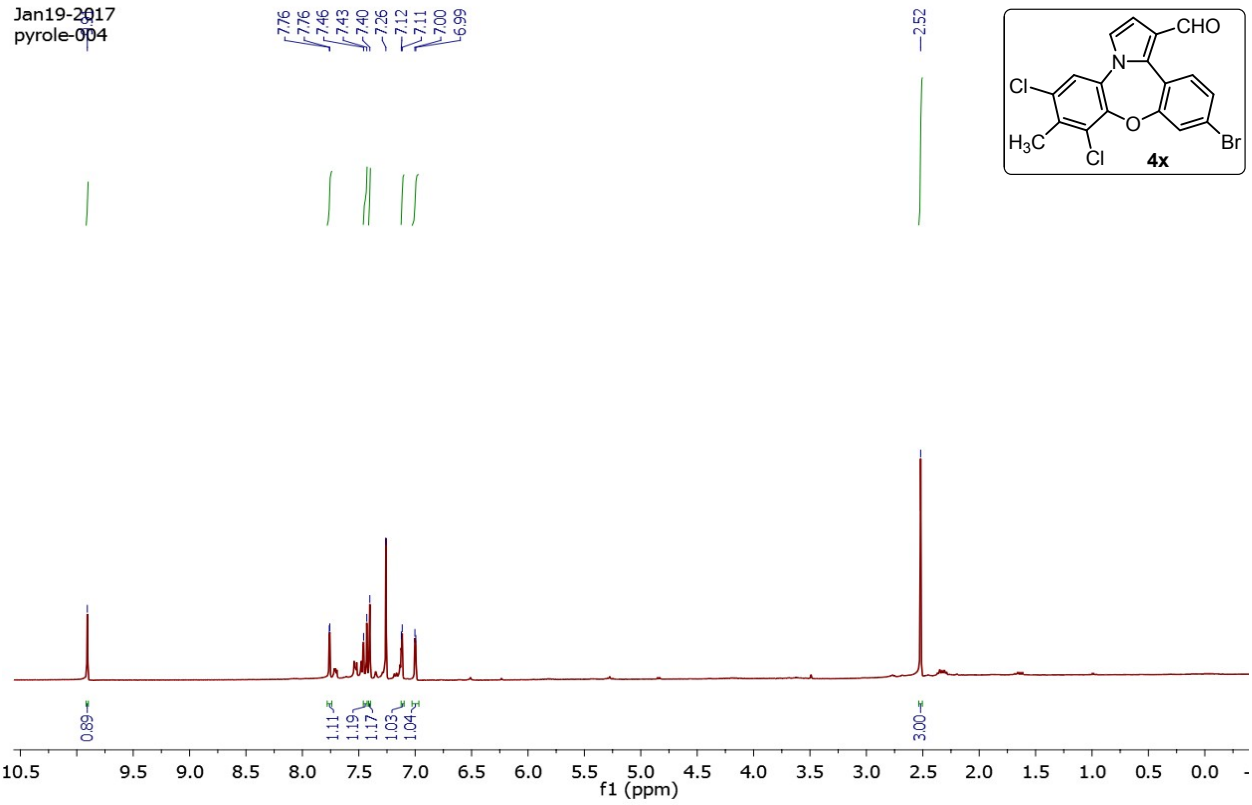
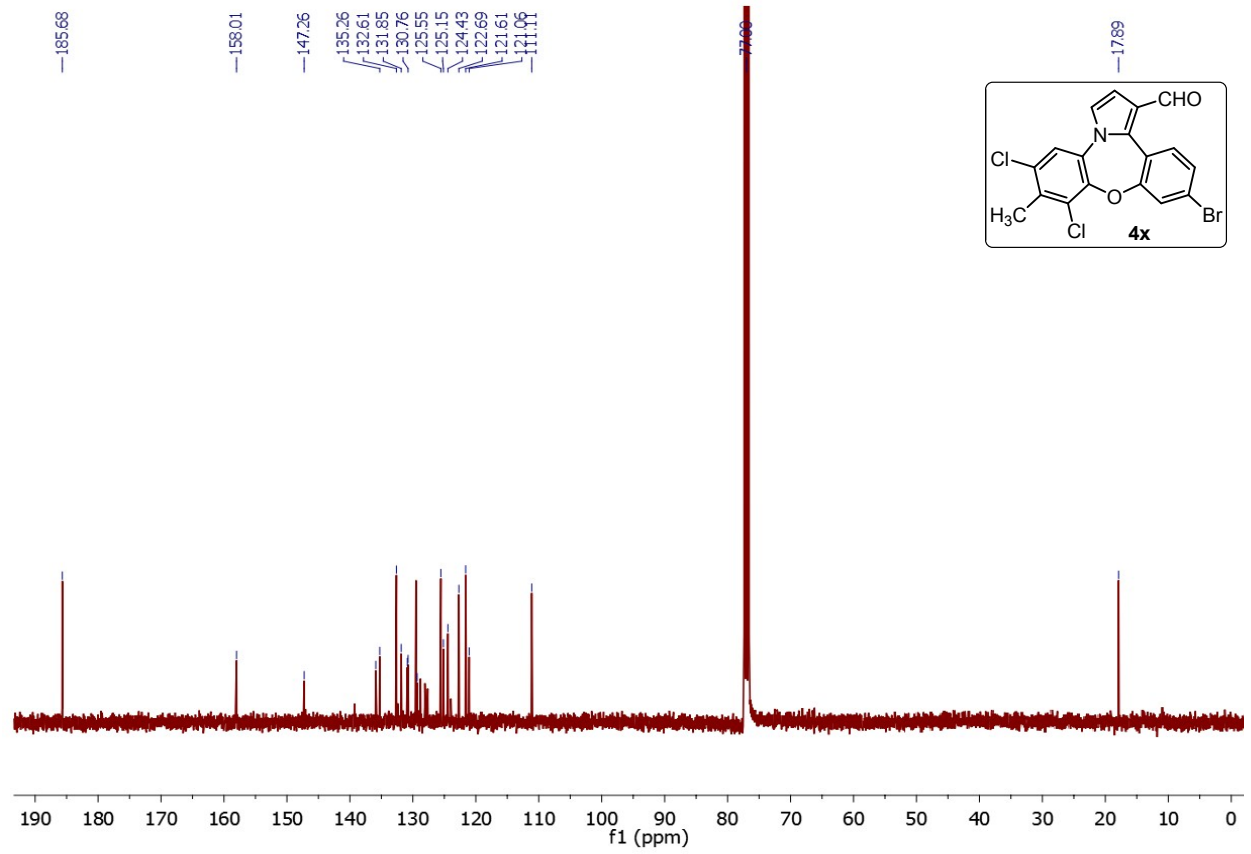
Cl
O
F
H3C
Cl
4v
f1 (ppm)


Apr26-2017
pyrrole-019
9.96
7.67
7.66
7.57
7.56
7.55
7.54
7.47
7.45
7.40
7.26
7.12
7.11
7.01
7.00
2.51
CHO
N
Cl
Br
O
H3C
Cl
4w
1.00
1.08
1.07
1.07
1.05
1.10
1.09
3.14
f1 (ppm)
185.59
156.95
147.32
135.26
134.21
133.88
125.39
123.61
122.83
121.61
119.05
110.99
77.00
17.87
f1 (ppm)


Jan19-2017
pyrole-004
7.76
7.76
7.46
7.43
7.40
7.26
7.12
7.11
7.00
6.99
2.52
CHO
N
Cl
O
H3C
Cl
Br
4x
0.89
1.11
1.19
1.17
1.03
1.04
3.00
f1 (ppm)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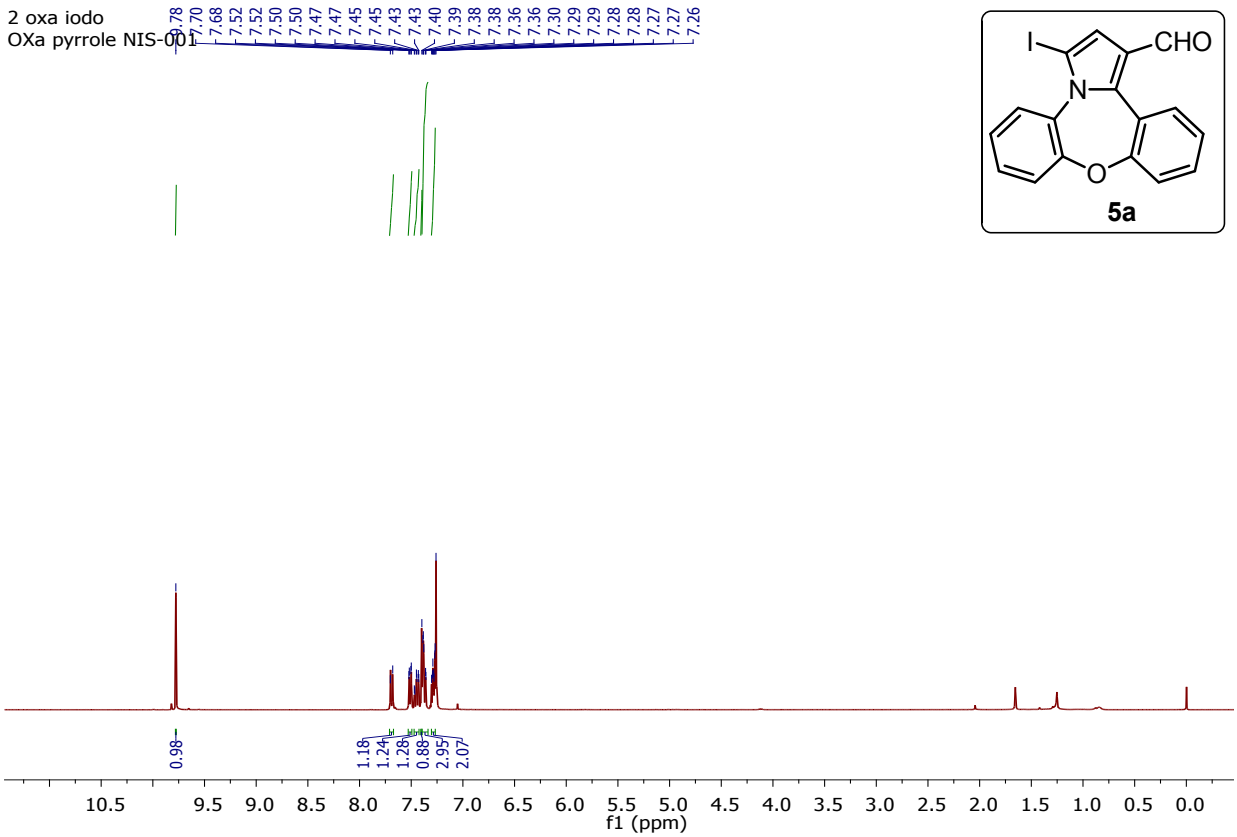
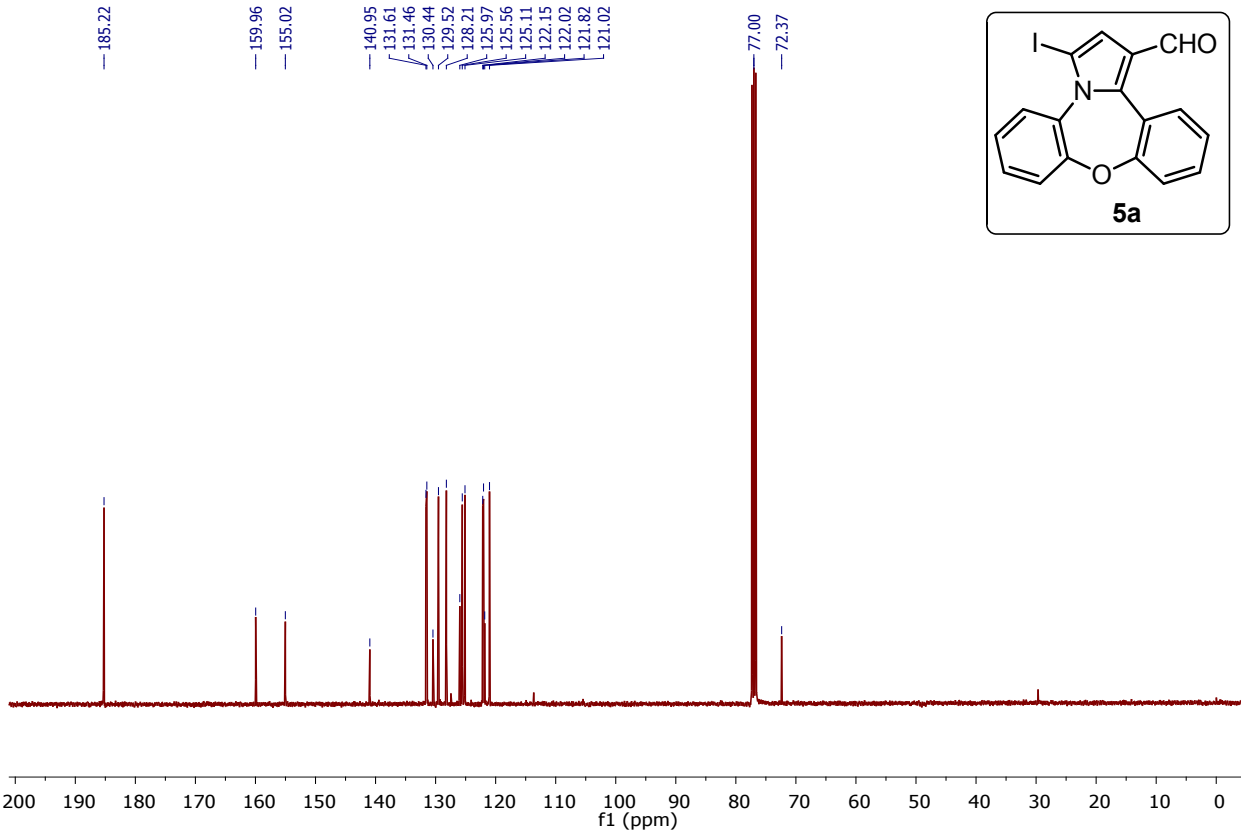
135.26
132.61
131.85
130.76
125.55
125.15
124.43
122.69
121.61
121.06
111.11
77.00
17.89
CHO
N
Cl
O
H3C
Cl
Br
4x
f1 (ppm)


2 oxa iodo
OXa pyrrole NIS-001
7.78
7.70
7.68
7.52
7.52
7.50
7.50
7.47
7.47
7.45
7.45
7.43
7.43
7.40
7.39
7.38
7.38
7.36
7.36
7.30
7.29
7.29
7.28
7.28
7.27
7.27
7.26
I
CHO
N
O
5a
0.98
1.18
1.24
1.28
0.88
2.95
2.07
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


185.22
159.96
155.02
140.95
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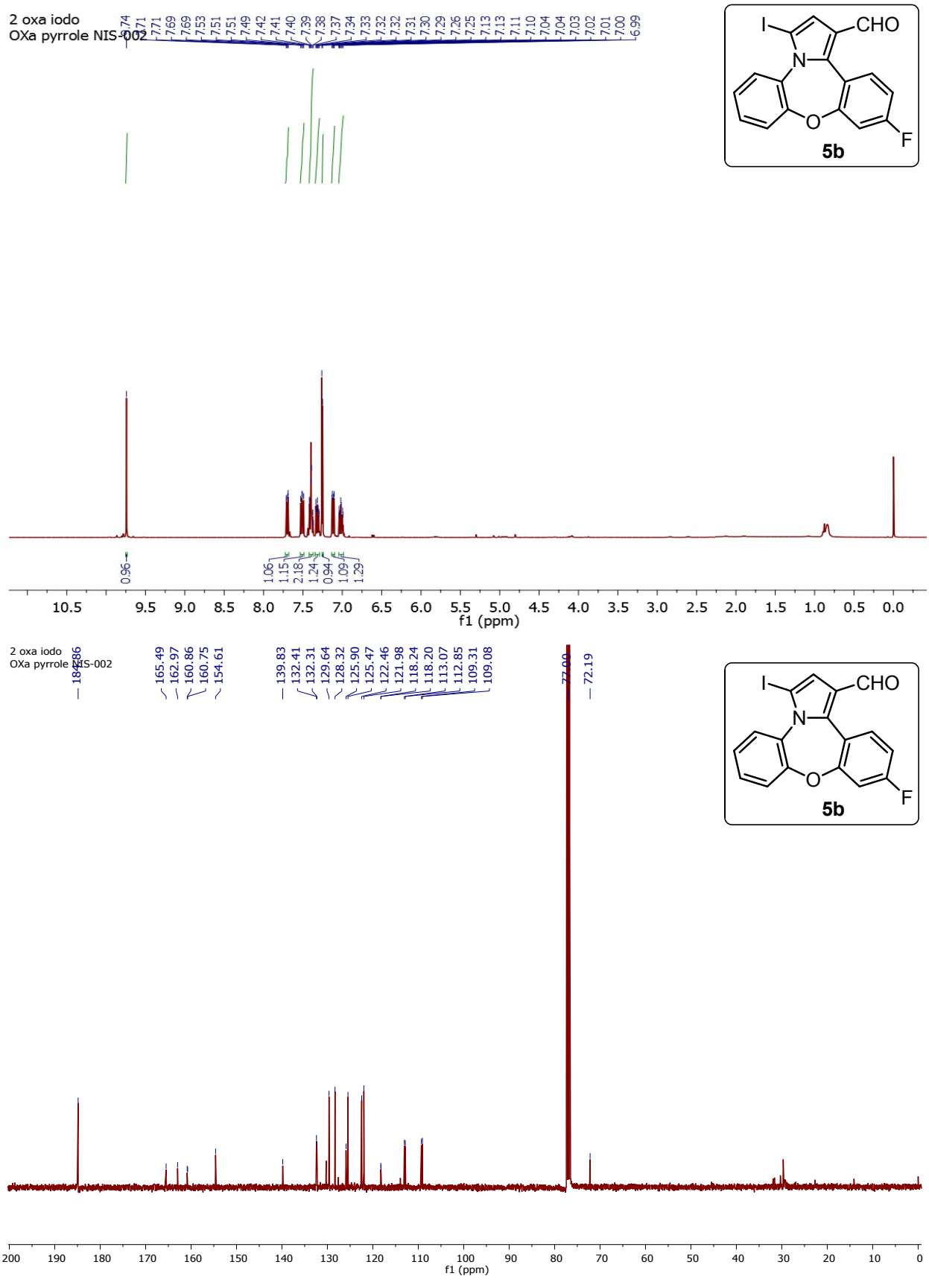
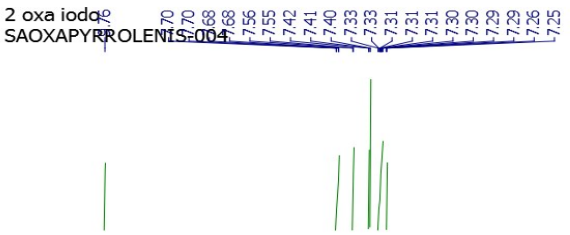
131.46
130.44
129.52
128.21
125.97
125.56
125.11
122.15
122.02
121.82
121.02
77.00
72.37
I
CHO
N
O
5a
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


2 oxa iodo
OXa pyrrole NIS-002
7.74
7.71
7.71
7.69
7.69
7.53
7.51
7.51
7.49
7.42
7.41
7.40
7.39
7.38
7.37
7.34
7.33
7.32
7.32
7.31
7.30
7.29
7.26
7.25
7.13
7.11
7.10
7.04
7.04
7.03
7.02
7.01
7.00
6.99
I
CHO
N
O
F
5b
0.96
1.06
1.15
2.18
1.24
0.94
1.09
1.29
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
2 oxa iodo
OXa pyrrole NIS-002
184.86
165.49
162.97
160.86
160.75
154.61
139.83
132.41
132.31
129.64
128.32
125.90
125.47
122.46
121.98
118.24
118.20
113.07
112.85
109.31
109.08
77.00
72.19
I
CHO
N
O
F
5b
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


2 oxa iodo
SAOXAPYRROLENIS-004
7.70
7.70
7.68
7.68
7.56
7.55
7.42
7.41
7.40
7.33
7.33
7.31
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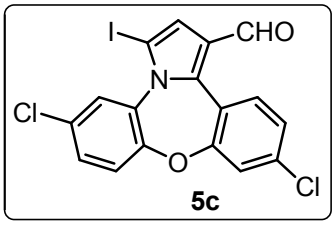
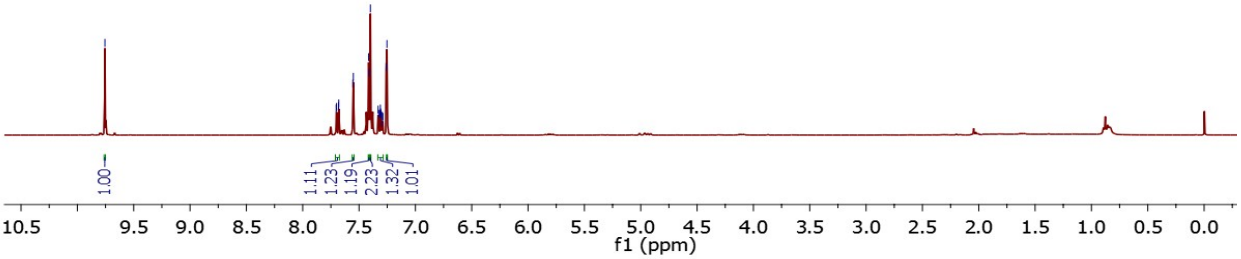
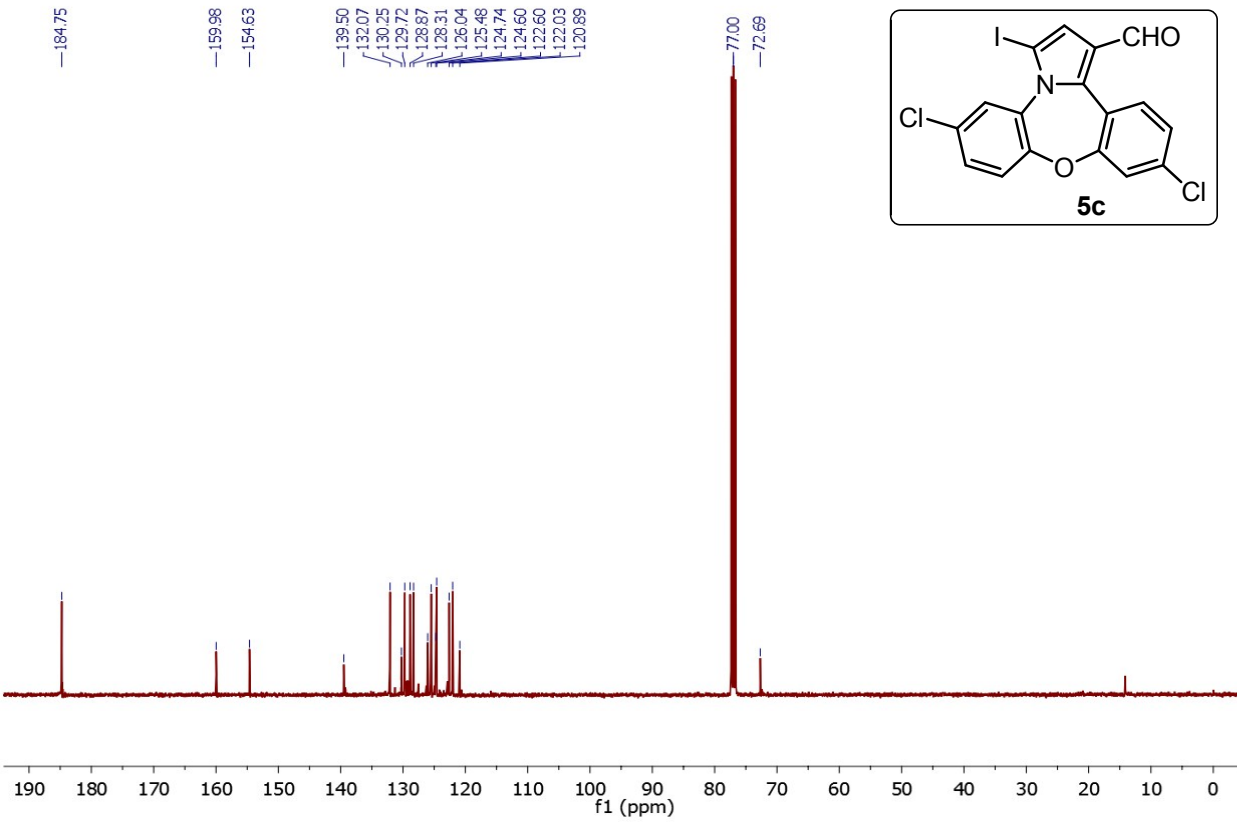
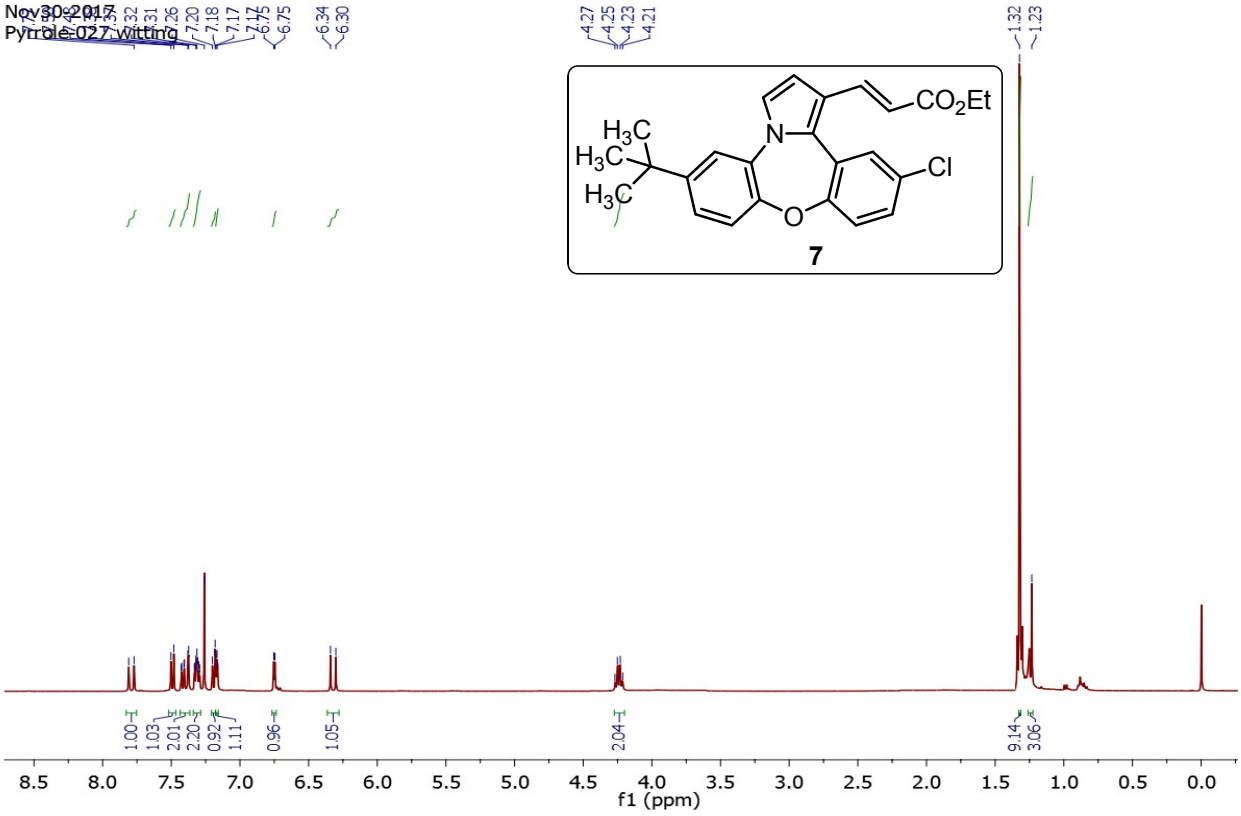
7.30
7.30
7.29
7.29
7.26
7.25

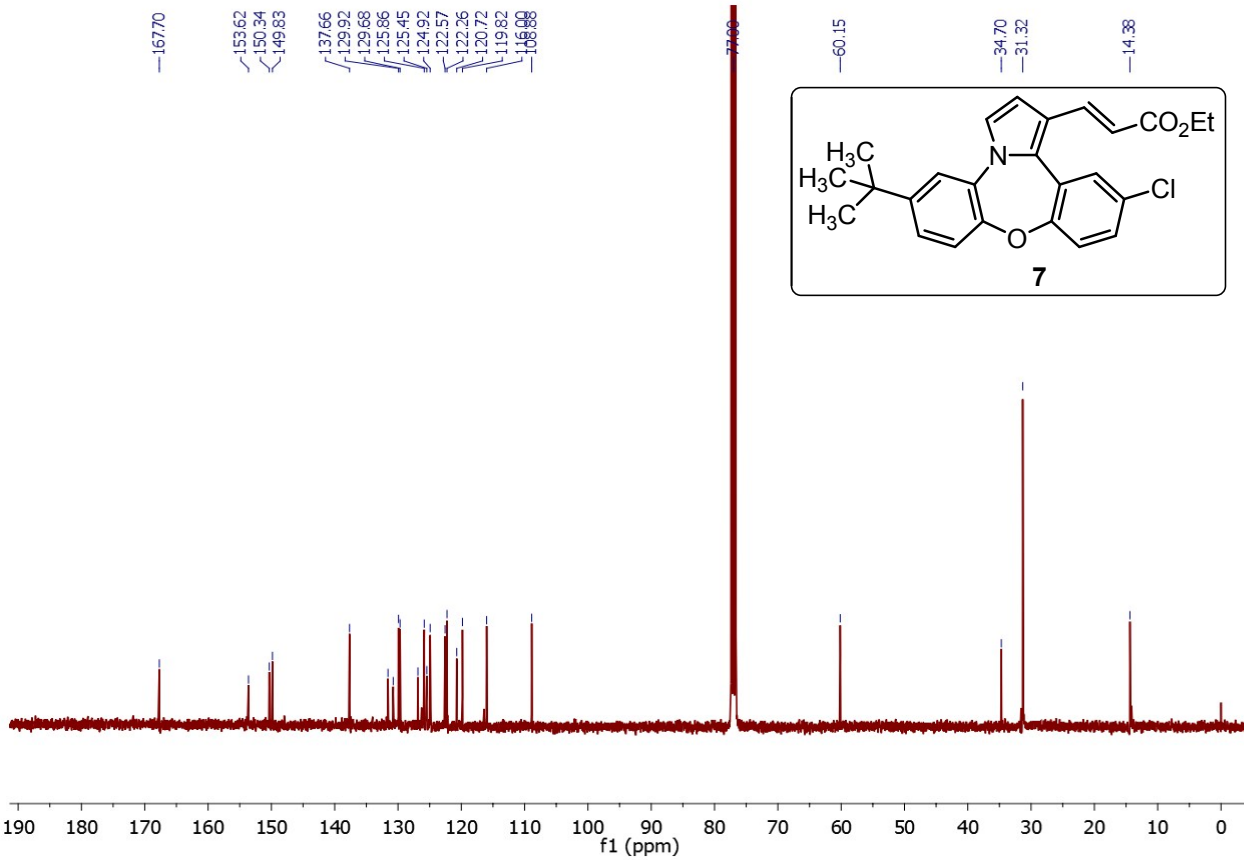

I
CHO
N
Cl
O
Cl
5c

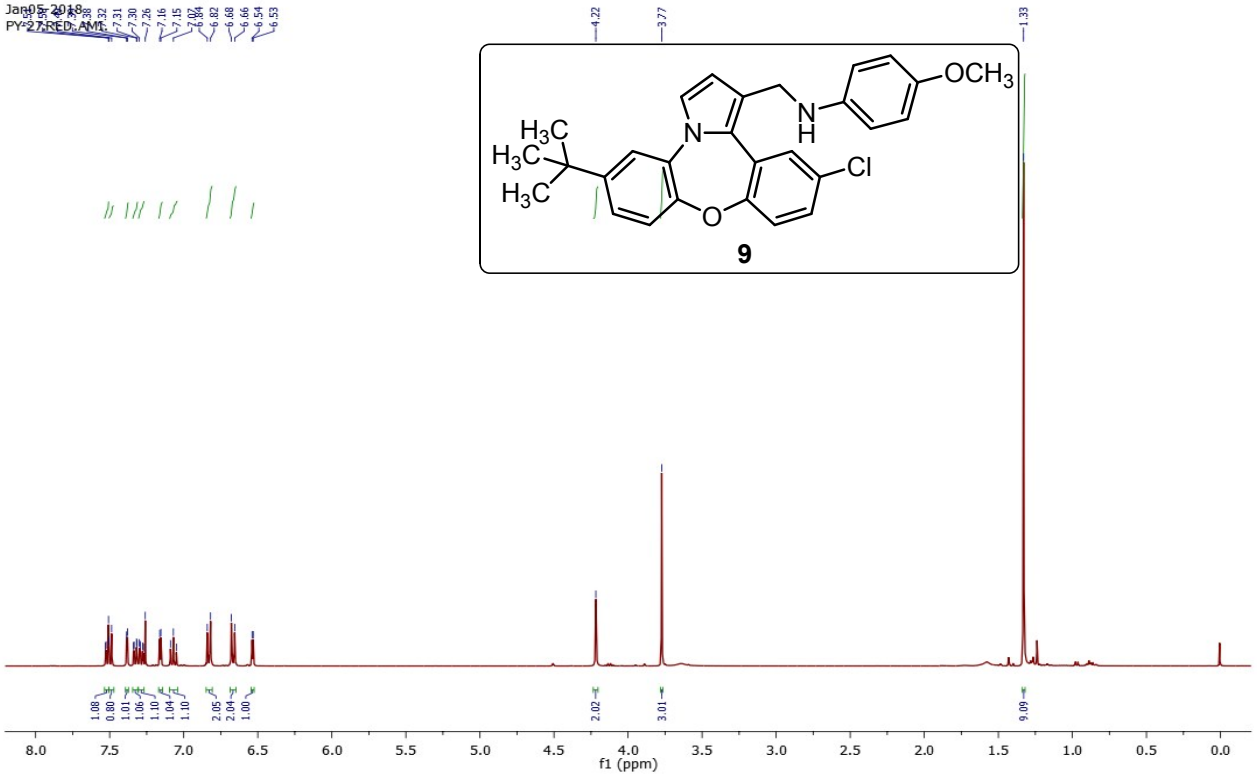

1.00
1.11
1.23
1.19
2.23
1.32
1.01
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

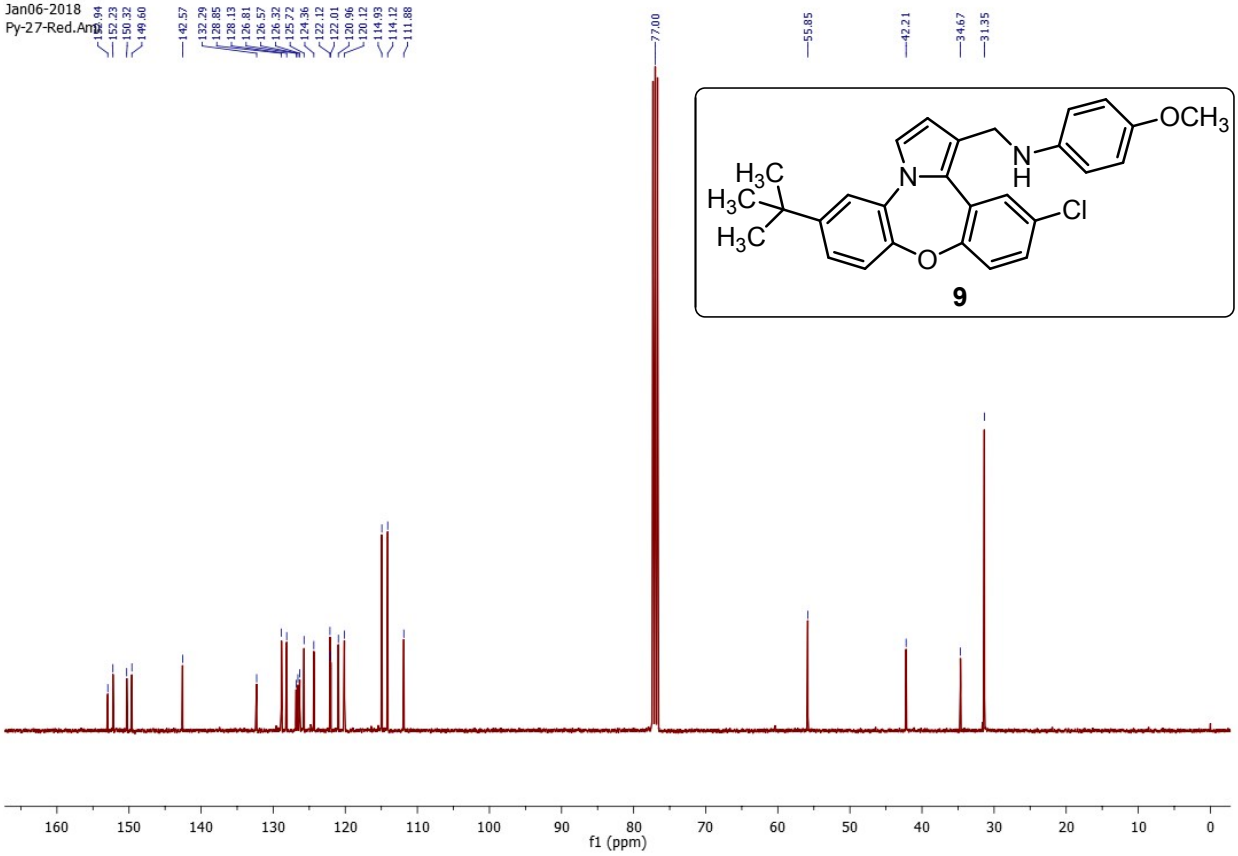

184.75
159.98
154.63
139.50
132.07
130.25
129.72
128.87
128.31
126.04
125.48
124.74
124.60
122.60
122.03
120.89
77.00
72.69
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

I
CHO
N
Cl
O
Cl
5c


Nov30-2017
Pyrrole-027-witting
7.32
7.31
7.26
7.20
7.18
7.17
7.17
6.75
6.75
6.34
6.30
4.27
4.25
4.23
4.21
1.32
1.23
CO2Et
N
H3C
H3C
H3C
Cl
O
7
1.00
1.03
2.01
2.20
0.92
1.11
0.96
1.05
2.04
9.14
3.06
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


167.70
153.62
150.34
149.83
137.66
129.92
129.68
125.86
125.45
124.92
122.57
122.26
120.72
119.82
77.00
60.15
34.70
31.32
14.38
CO2Et
N
H3C
H3C
H3C
Cl
O
7
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

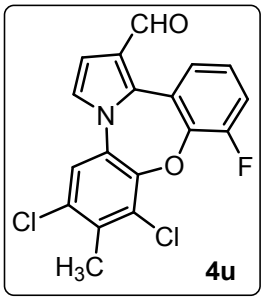

Jan05-2018
PY-27-RED. AMT
4.22
3.77
1.33
OCH3
N
H
H3C
H3C
H3C
N
Cl
O
9
1.08
0.80
1.01
1.06
1.10
1.04
1.10
2.05
2.04
1.00
2.02
3.01
9.09
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


Jan06-2018
Py-27-Red.Amt
152.23
150.32
149.60
142.57
132.29
128.85
128.13
126.81
126.57
126.32
125.72
124.36
122.12
122.01
120.96
120.12
114.93
114.12
111.88
77.00
55.85
42.21
34.67
31.35
OCH3
N
H
H3C
H3C
H3C
N
Cl
O
9
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

Crystal structure data for (4u)

6,8-dichloro-10-fluoro-7-methyldibenzo[*b*,*f*]pyrrolo[1,2-*d*][1,4]oxazepine-1-carbaldehyde

(CCDC NO. 1573097)



The title compound, 6,8-dichloro-10-fluoro-7-methyldibenzo[b,f]pyrrolo[1,2-d][1,4]oxazepine-1-carbaldehyde(**4u**), $C_{18}H_{10}Cl_2FNO_2$, crystallizes in the monoclinic space group $P2_1/c$with unit cell parameters: a =16.6493(13), b = 13.1167(11), c = 7.0767(7)Å, β = 98.220(9)° and Z= 4. The crystal structure was solved by direct methods using single-crystal X-ray diffraction data and refined to R = 0.0626 for 2041 observed reflections.

Crystal Structure Determination and Refinement

X-ray intensity data of the crystal of the dimension 0.20 X 0.20 X 0.10 mm^3having well defined morphology was collected on *X'calibur*CCD area-detector diffractometer equipped with graphite monochromated Mo*Kα* radiation (λ = 0.71073 Å). X-ray intensity data of 6007 reflections (of which 3003 unique) were collected at 293(2) K. The cell dimensions were determined by least-squares fit of angular settings of 1757 reflections in theθ range 3.88 to 27.55°.The intensities were measured by ω scan mode forθ ranges 3.66 to 25.99°. 2041 reflections were treated as observed ($I > 2\sigma(I)$). Data were corrected for Lorentz and polarisation factors. The structure was solved by direct methods using SHELXS97 [1]. All non-hydrogen atoms of the molecule were located in the best E-map. Full-matrix least-squares refinement was carried out using SHELXL97 [2]. All the hydrogen atoms were geometrically fixed and allowed to ride on their parent carbon atoms with C-H= 0.93 – 0.96 Å with *U*iso = 1.5*U*eq of the attached C atom for methyl H atoms and1.2*U*eq for other H atoms. The final refinement cycles converged to an R = 0.0626 and wR (F^2) = 0.1607 for the observed data. Residual electron densities ranged from -0.325 to 0.377 $eÅ^{-3}$. Atomic scattering factors were taken from International Tables for X-ray Crystallography (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4). The crystallographic data are summarized in Table 1.An ORTEP view of the title compound with atomic labeling is shown in Fig.1 [3]. The geometry of the molecule was

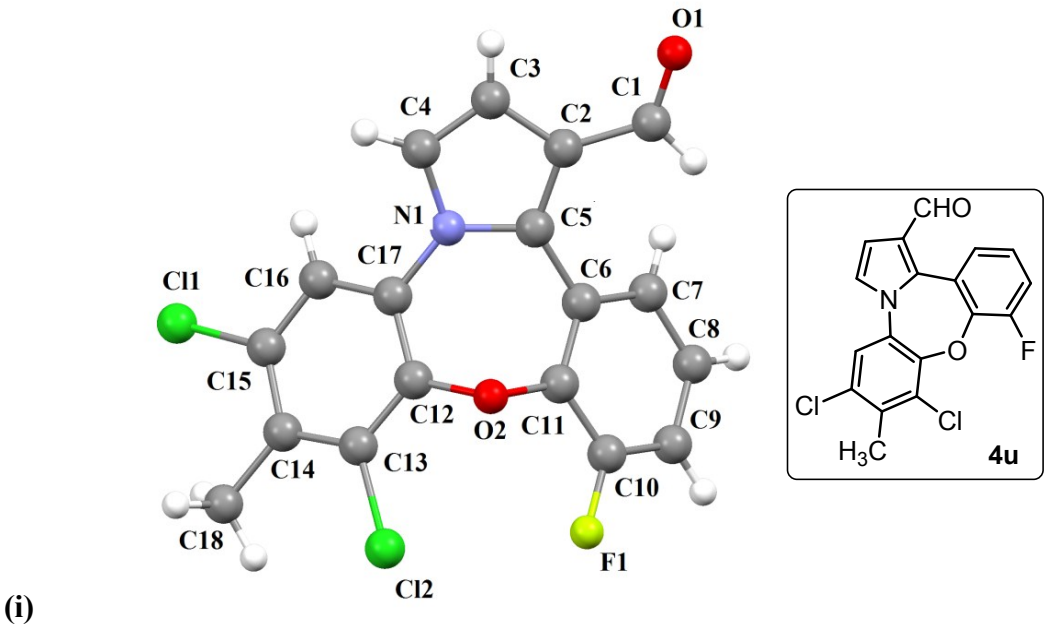
calculated using the PLATON [4] and PARST [5] software's. Bond lengths and bond angles are within expected values [6].

Table 1 Crystal and experimental data

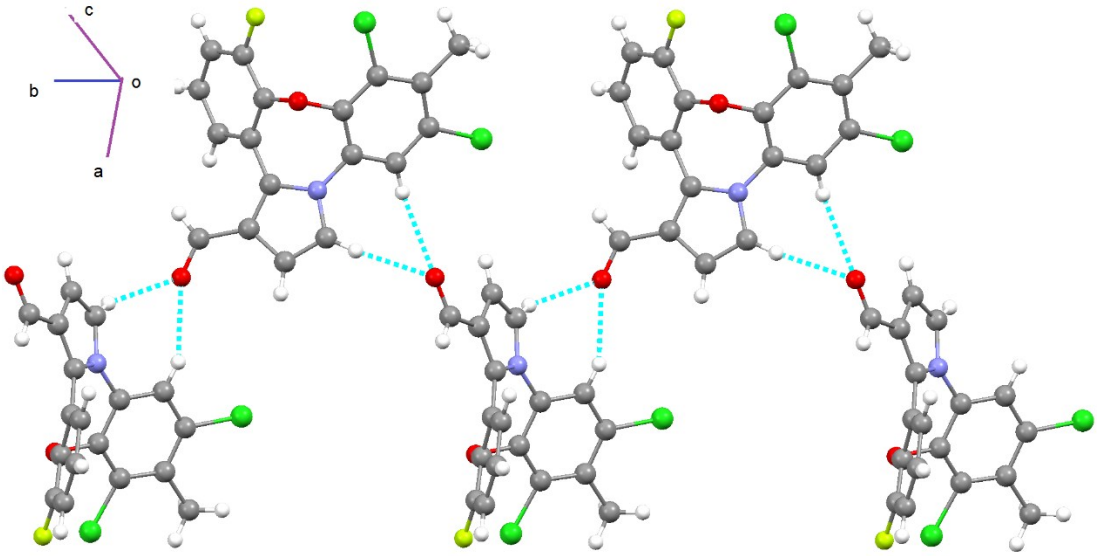
CCDC	**1573097**
Crystal description	Plate shaped
Crystal colour	Orange
Crystal size	0.2 x 0.2 x 0.1 mm
Empirical formula	$C_{18}H_{10}Cl_2FNO_2$
Formula weight	362.17
Radiation, Wavelength	Mo *K*α, 0.71073 Å
Unit cell dimensions	a =16.6493(13), b = 13.1167(11), c = 7.0767(7) Å, β= 98.220(9)°
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell volume	1529.6(2)
No. of molecules per unit cell, Z	4
Temperature	293(2)
Absorption coefficient	0.446 mm^{-1}
F(000)	736
Scan mode	ω scan
θ range for entire data collection	3.66<θ<25.99 °
Range of indices	h= -20 to 20, k= -16 to 11, l= -8 to 4

Reflections collected / unique	6007 / 3003
Reflections observed ($I > 2\sigma(I)$)	2041
R_{int}	0.0605
R_{sigma}	0.0430
Structure determination	Direct methods
Refinement	Full-matrix least-squares on F^2
No. of parameters refined	219
Final R	0.0626
$wR(F^2)$	0.1607
Weight	$1/[\sigma^2(F_o^2)+(0.0725P)^2+ 0.8139P]$ where $P=[F_o^2 +2F_c^2] / 3$
Goodness-of-fit	1.104
$(\Delta /\sigma)_{max}$	0.002 (for tors H18A)
Final residual electron density	$-0.325 < \Delta\rho < 0.377$ eÅ^{-3}
Measurement	*X'calibur system – Oxford diffraction make, U.K.*
Software for structure solution:	SHELXS97 (Sheldrick, 2008)
Software for refinement:	SHELXL97 (Sheldrick, 2015)
Software for molecular plotting:	ORTEP-3 (Farrugia, 2012) PLATON (Spek, 2009)

Software for geometrical calculation PLATON (Spek, 2009)



(i)

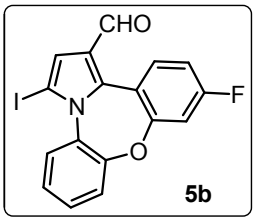


(ii)

Figure 1: ***(i)*** *ORTEP* view of the molecule (**4u**), with Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. **(CCDC NO. 1573097)** ***(ii)*** The crystal packing viewed down a-axis

11-fluoro-3-iododibenzo[b,f]pyrrolo[1,2-d][1,4]oxazepine-1-carbaldehyde (5b):

(CCDC NO. 1573098)



The title compound 11-fluoro-3-iododibenzo[b,f]pyrrolo[1,2-d][1,4]oxazepine-1-carbaldehyde **(7)**,$C_{17}H_9N_1O_2F_1I_1$,crystallizes in the orthorhombic space group Pbcawith unit cell parameters: a = 17.5729(13), b = 9.1432(10), c = 17.8218(13)Å, and Z= 8. The crystal structure was solved by direct methods using single-crystal X-ray diffraction data and refined to R = 0.0406 for 2081 observed reflections.

Crystal Structure Determination and Refinement

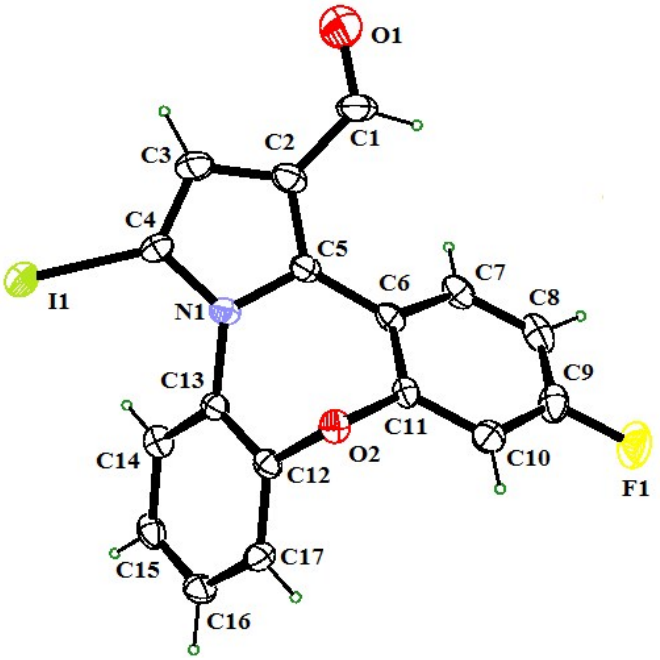
X-ray intensity data of the crystal of the dimension 0.30 X 0.20 X 0.20 mm^3having well defined morphology was collected on *X'calibur*CCD area-detector diffractometer equipped with graphite monochromatedMo*Kα* radiation (λ = 0.71073 Å).X-ray intensity data of 7316 reflections (of which 2808 unique) were collected at 293(2)K. The cell dimensions were determined by least-squares fit of angular settings of 2367 reflections in theθ range 4.11 to 28.88°**.**The intensities were measured byωscan mode forθ ranges 3.95 to 26.00°. 2081 reflections were treated as observed ($I > 2\sigma(I)$). Data were corrected for Lorentz and polarisation factors. The structure was solved by direct methods using SHELXS97 [1]. All non-hydrogen atoms of the molecule were located in the best E-map. Full-matrix least-squares refinement was carried out using SHELXL97 [2]. All the hydrogen atoms were geometrically fixed and allowed to ride on their parent carbon atomswith C-H= 0.93 Å with $U_{iso}(H) = 1.2U_{eq}(C)$. The final refinement cycles converged to an R = 0.0406 and wR (F^2) = 0.0914 for the observed data. Residual electron densities ranged from -0.571 to 0.665e$Å^{-3}$. Atomic scattering factors were taken from International Tables for X-ray Crystallography (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4). The crystallographic data are summarized in Table 2.

An ORTEP view of the title compound with atomic labeling is shown in Fig.2 [3]. The geometry of the molecule was calculated using the PLATON [4] and PARST [5] softwares. Bond lengths and bond angles are within expected values [6].

Table 2 Crystal and experimental data

CCDC	**1573098**
Crystal description	Block shaped
Crystal colour	Orange
Crystal size	0.3 x 0.2 x 0.2 mm
Empirical formula	$C_{17}H_9N_1O_2F_1I_1$
Formula weight 405.15	
Radiation, Wavelength	Mo *K*α, 0.71073 Å
Unit cell dimensions	a= 17.5729(13), b= 9.1432(10), c= 17.8218(13)Å,
Crystal system	Orthorhombic
Space group	Pbca
Unit cell volume	2863.5(4)
No. of molecules per unit cell, Z	8
Temperature	293(2)
Absorption coefficient	2.254 mm^{-1}
F(000)	1568
Scan mode	ω scan
θrange for entire data collection	3.95<θ<26.00 º
Range of indices	h= -21 to 14, k= -11 to 11, l= -17 to 21
Reflections collected / unique	7316 / 2808
Reflections observed (I > 2σ(I))	2081
R_{int}	0.0423
R_{sigma}	0.0361

Structure determination	Direct methods
Refinement	Full-matrix least-squares on F^2
No. of parameters refined	200
Final R	0.0406
wR(F^2)	0.0914
Weight	$1/[\sigma^2(F_o^2)+(0.0363\ P)^2+3.3228P]$
	where $P=[F_o^2 +2F_c^2] / 3$
Goodness-of-fit	1.125
$(\Delta /\sigma)_{max}$	0.002 (forU22 C5)
Final residual electron density	$-0.571 < \Delta\rho < 0.665$ eÅ^{-3}
Measurement	*X'calibur system – Oxford diffraction make, U.K.*
Software for structure solution:	SHELXS97 (Sheldrick, 2008)
Software for refinement:	SHELXL97 (Sheldrick, 2015)
Software for molecular plotting:	ORTEP-3 (Farrugia, 2012) PLATON (Spek, 2009)
Software for geometrical calculation	PLATON (Spek, 2009)



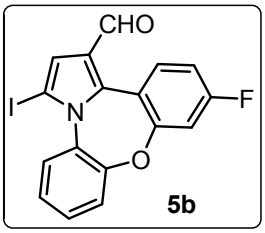


FIGURE 2: *ORTEP* view of the molecule (**5b**), with Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. **(CCDC NO. 1573098)**

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