Supporting Information

One-pot Double [3+2] Cycloadditions for Diastereoselective Synthesis of Pyrrolidine-based Polycyclic Systems

Xiaofeng Zhang, Weiqi Qiu, Xiaoming Ma, Jason Evans, Manpreet Kaur, Jerry P. Jasinski, Wei Zhang

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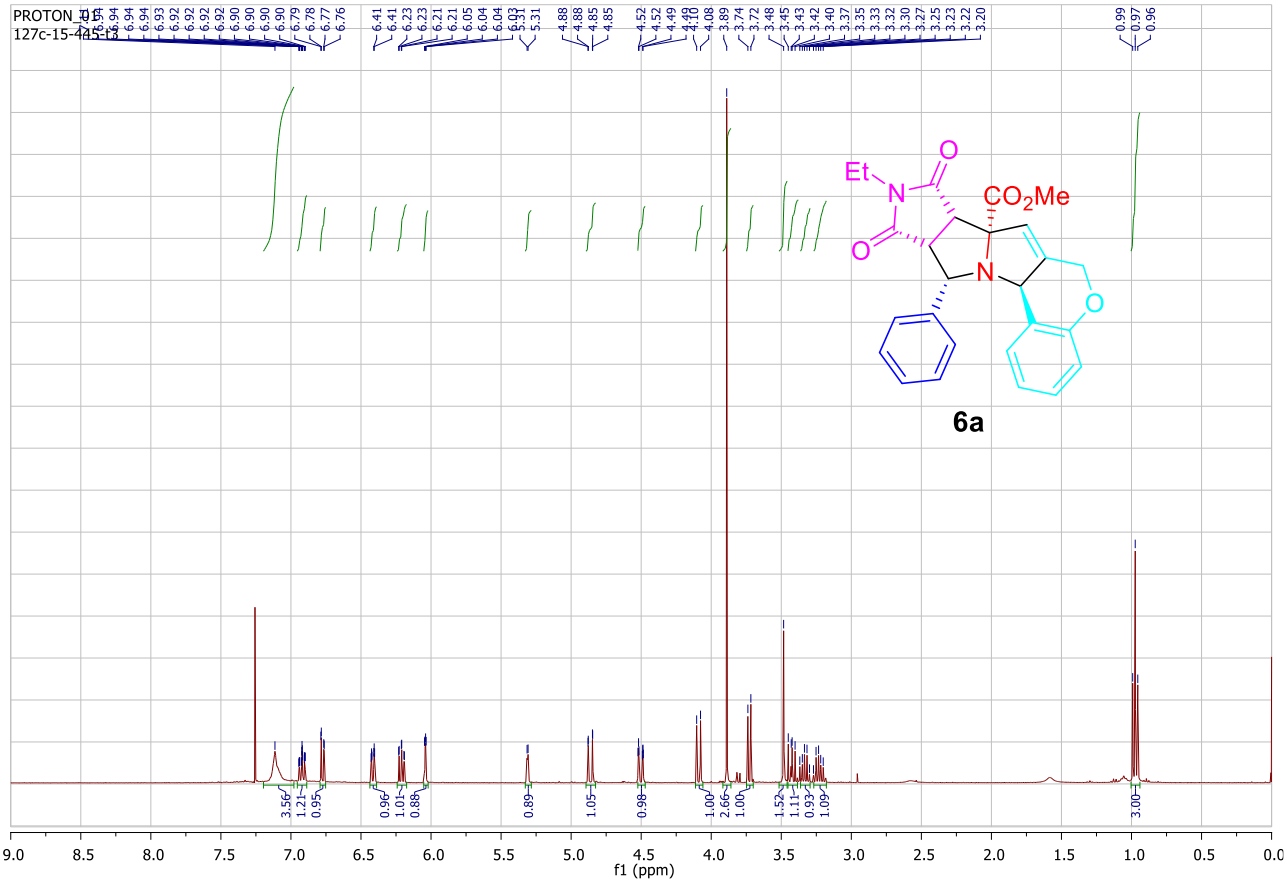
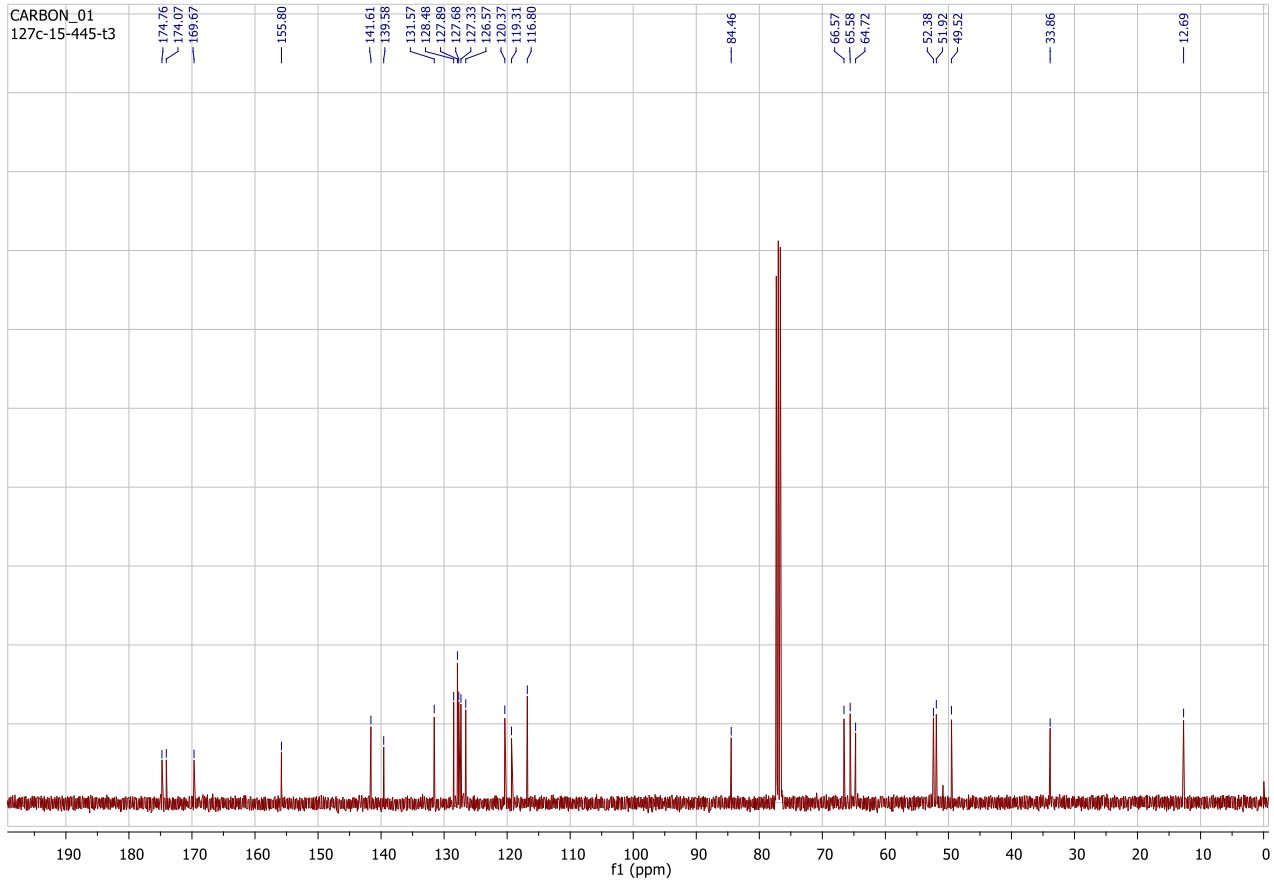
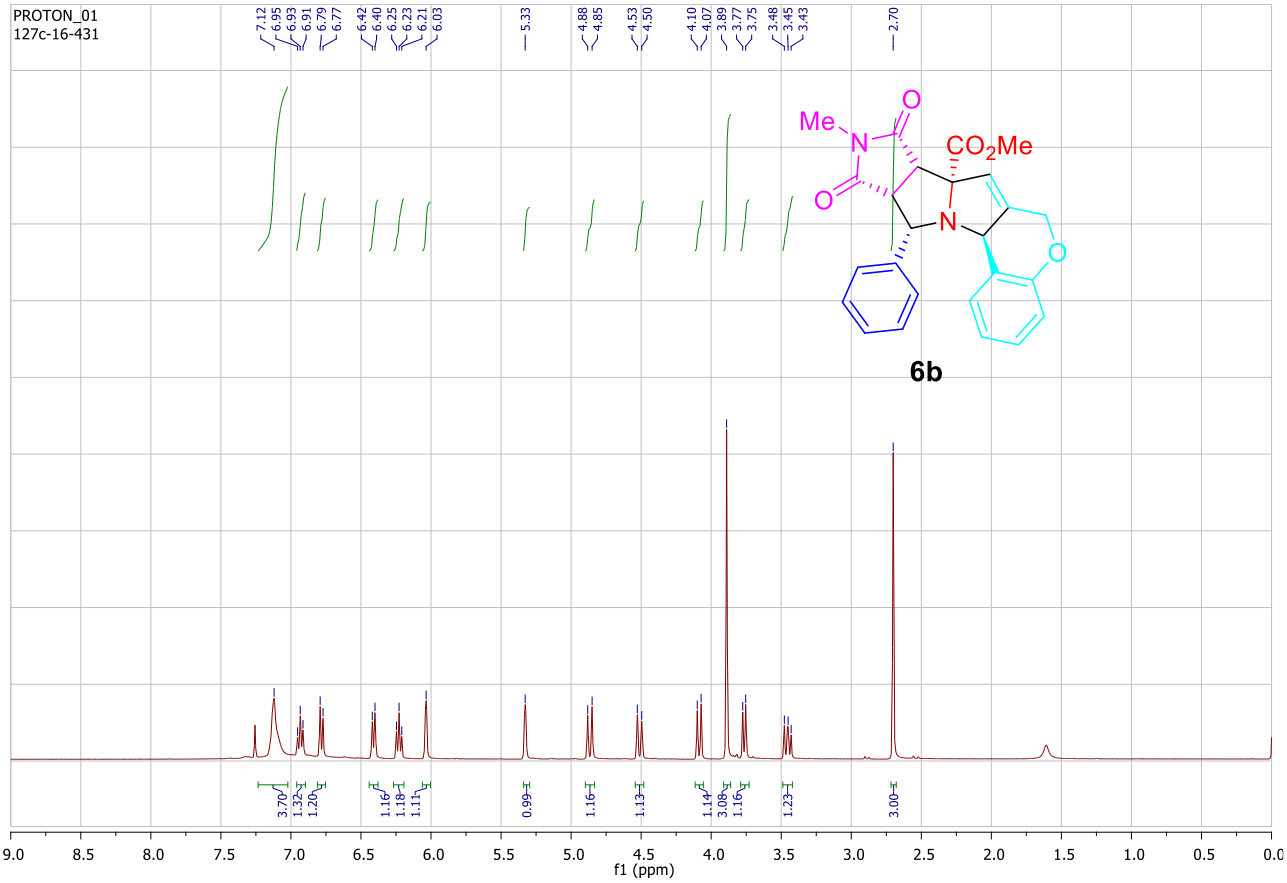
1. NMR Spectra of Products



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3.70
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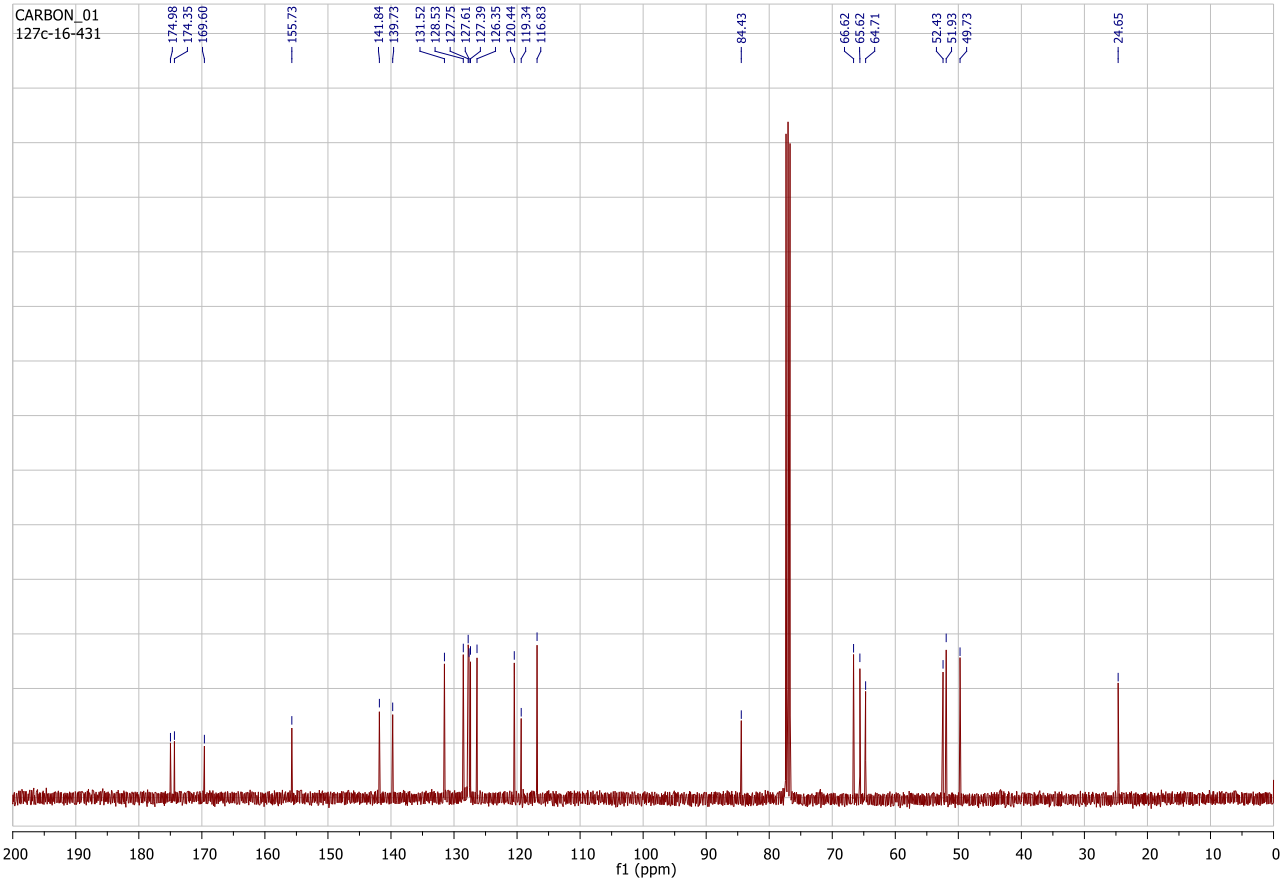
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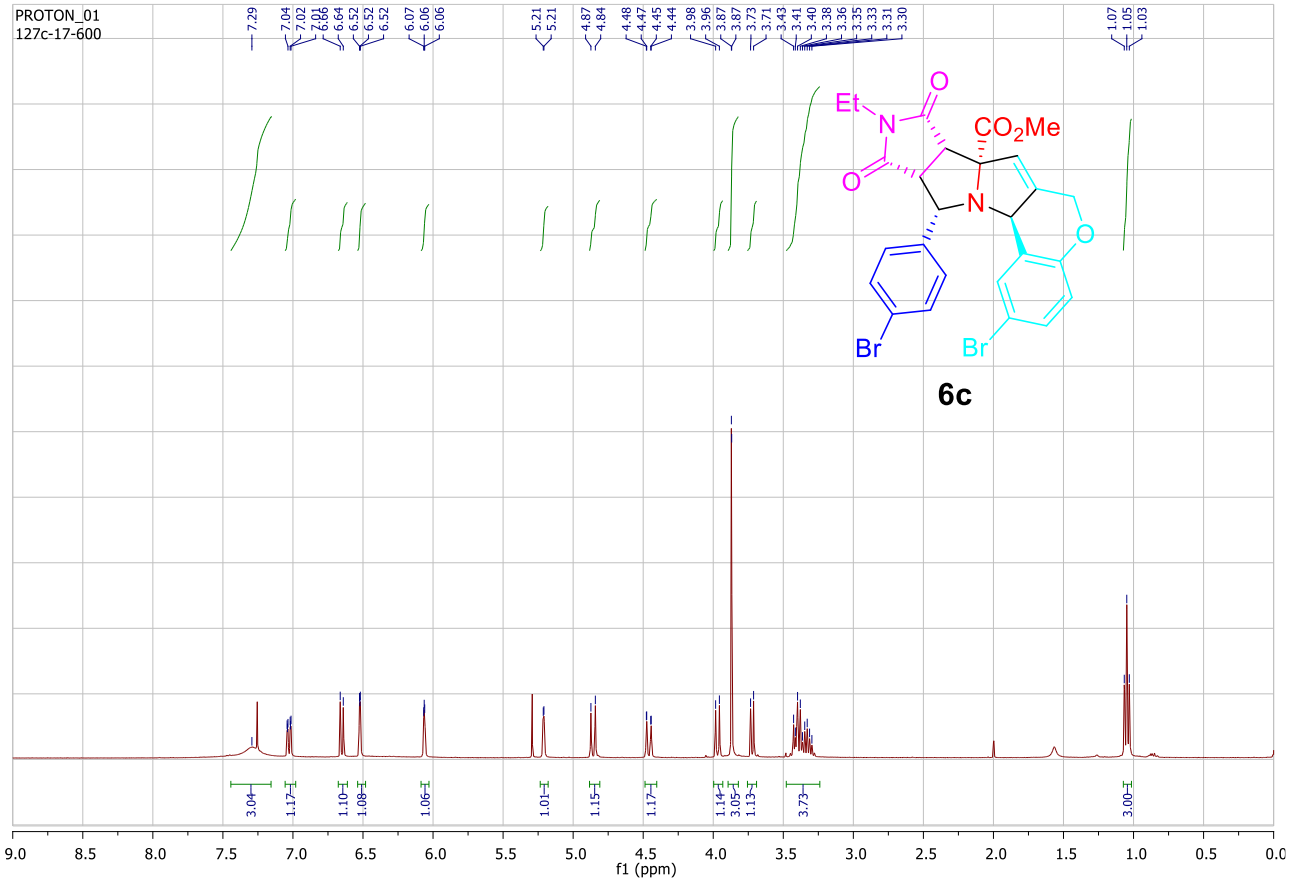
f1 (ppm)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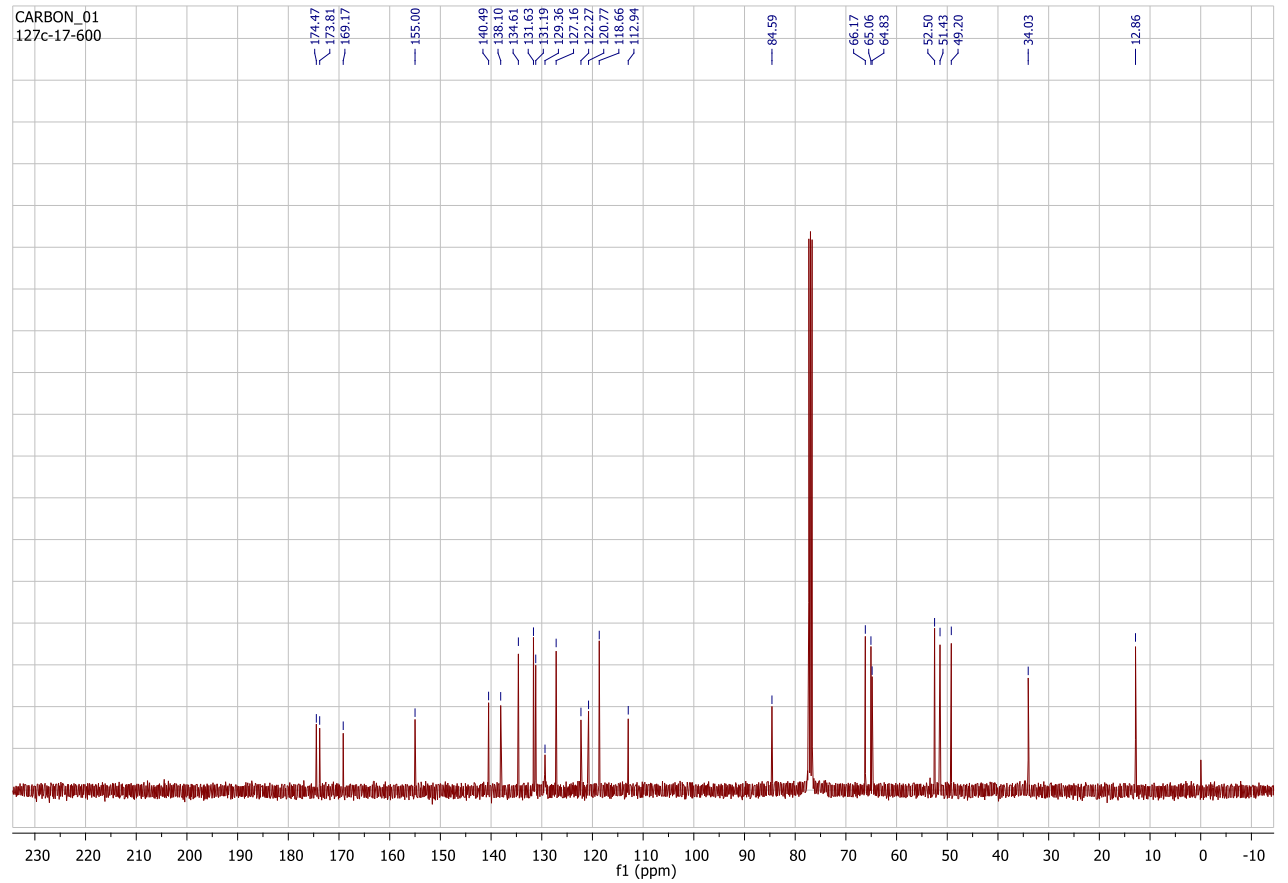
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66.17
65.06
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49.20
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220
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170
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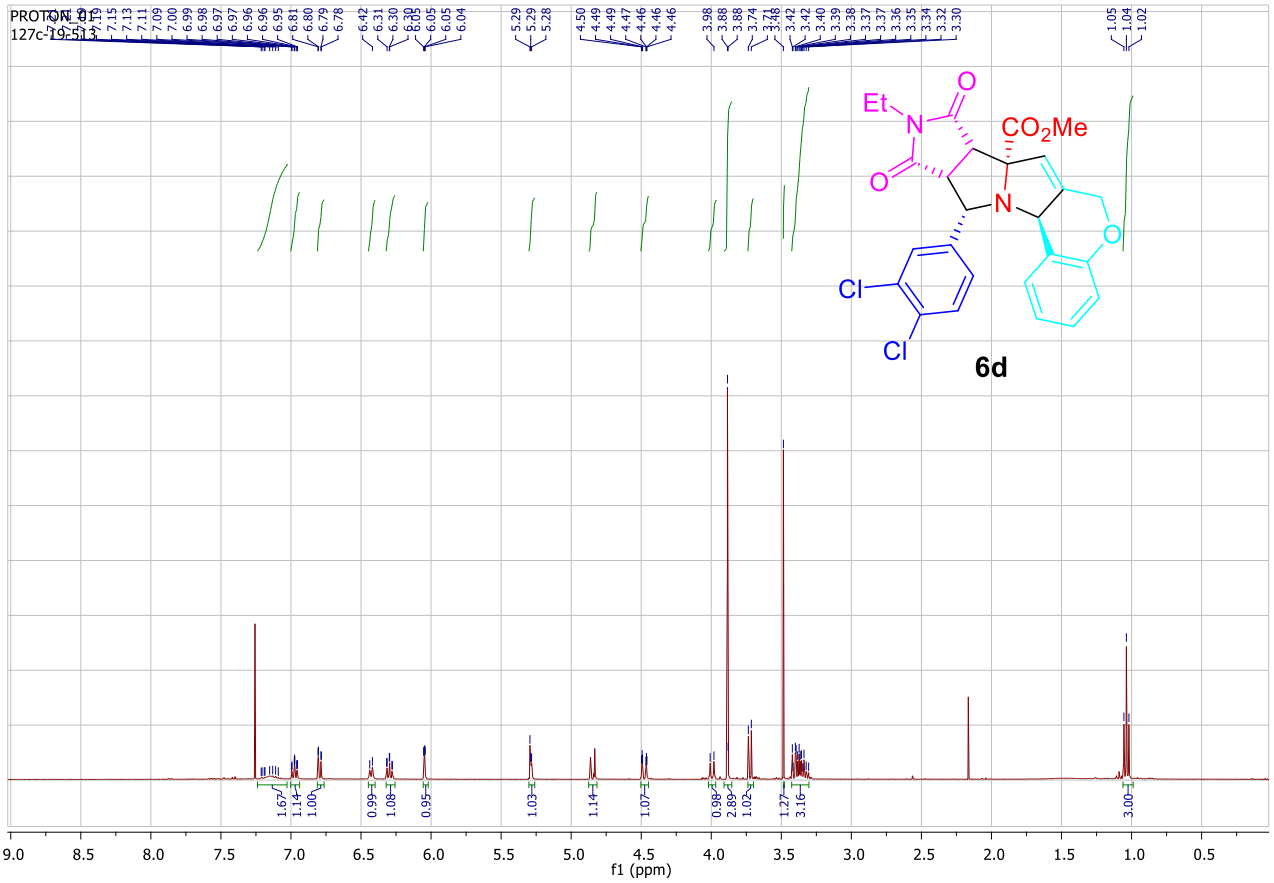
f1 (ppm)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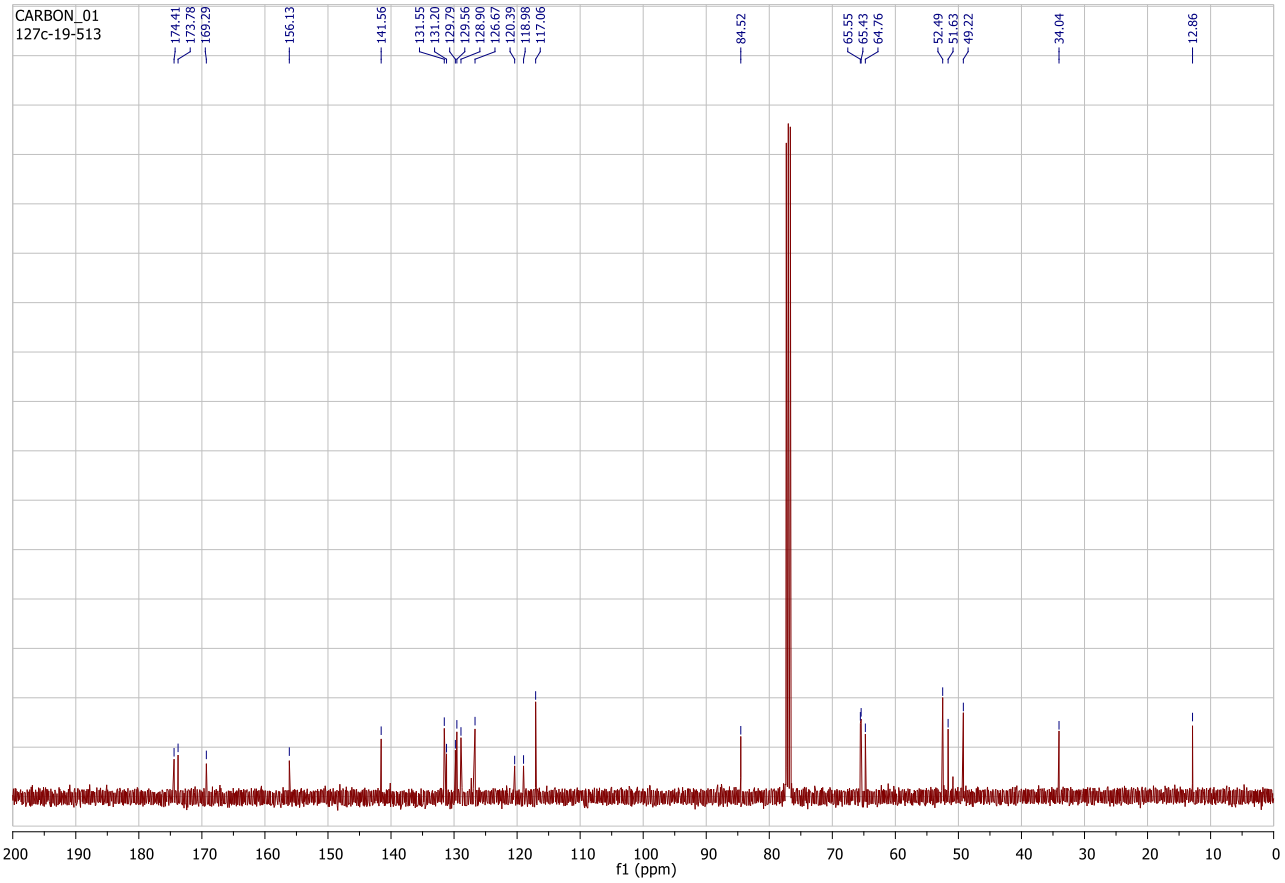
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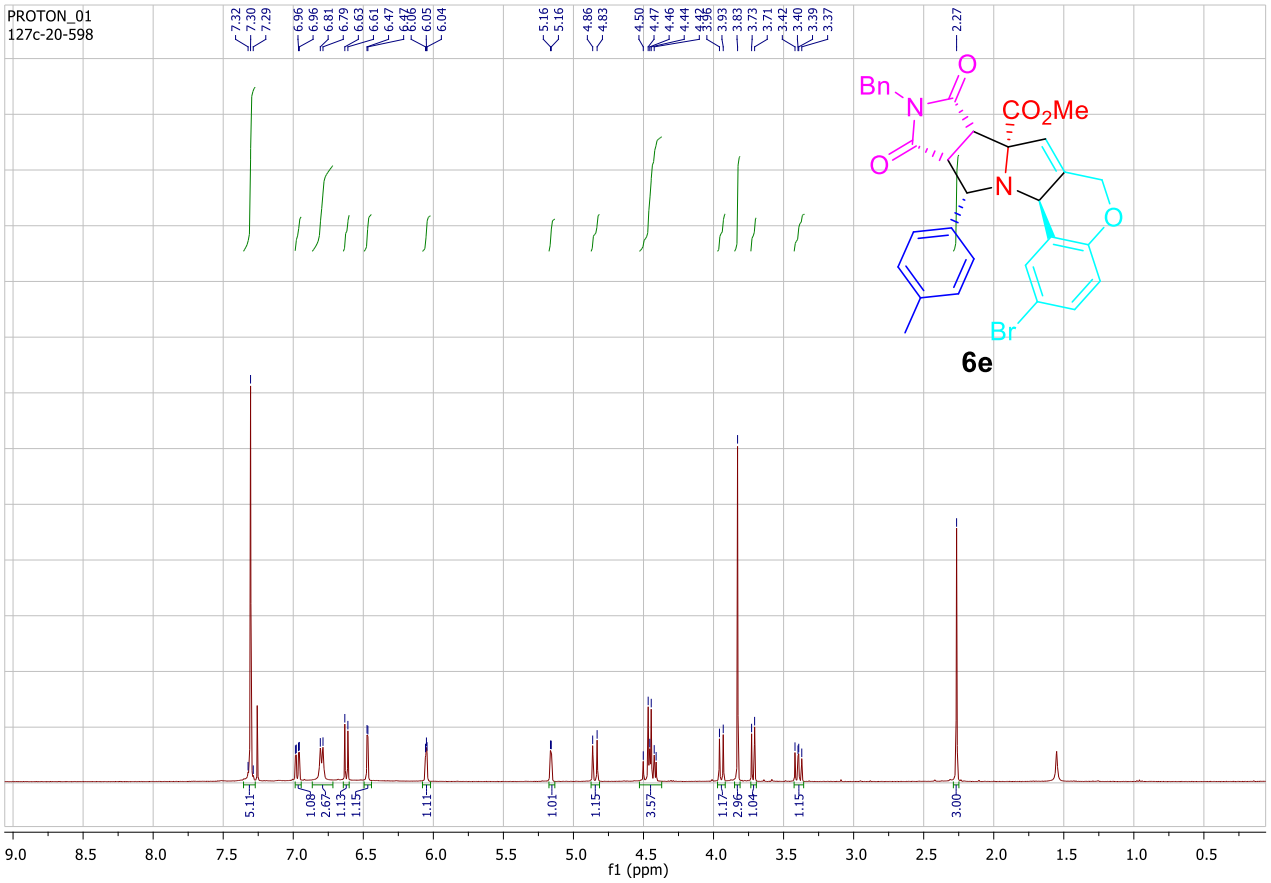
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O
Br
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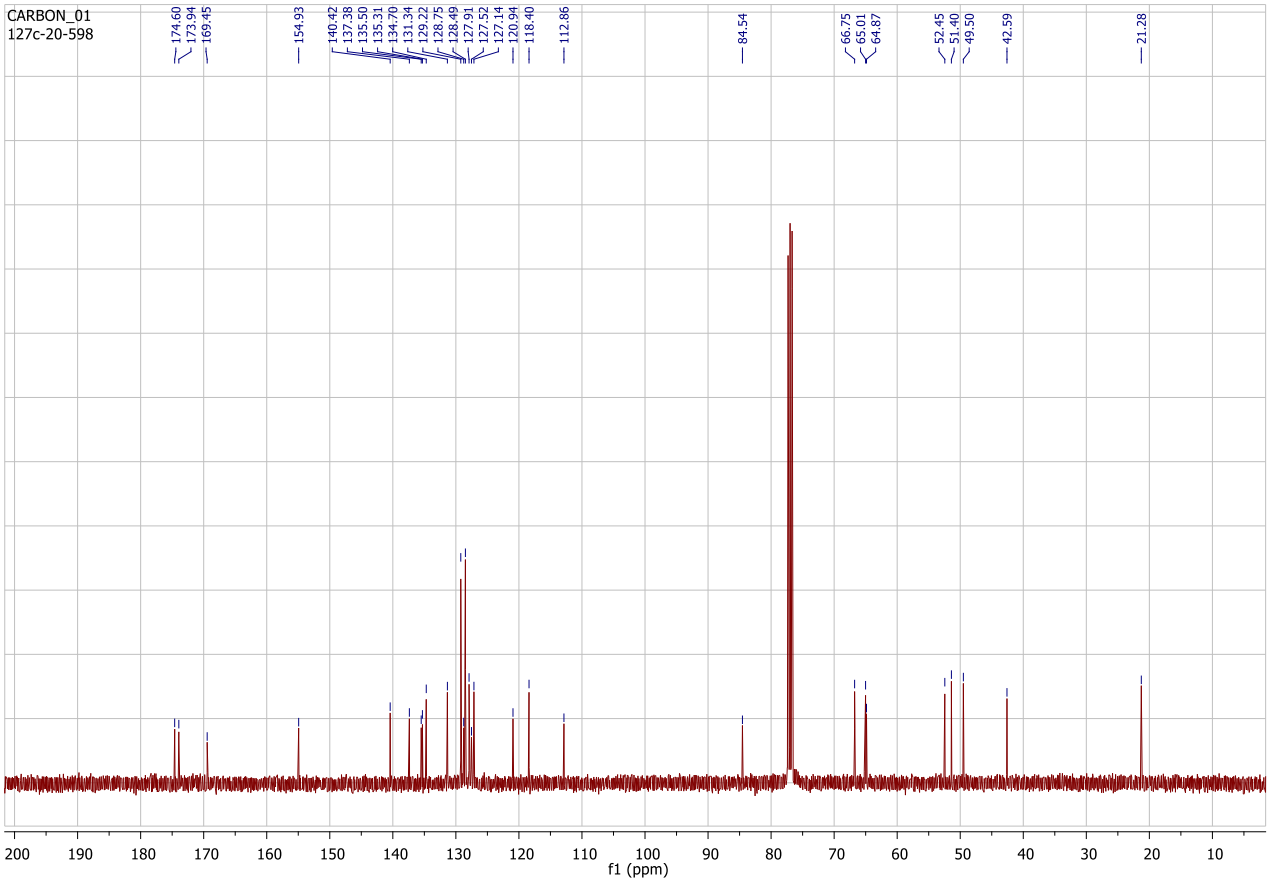
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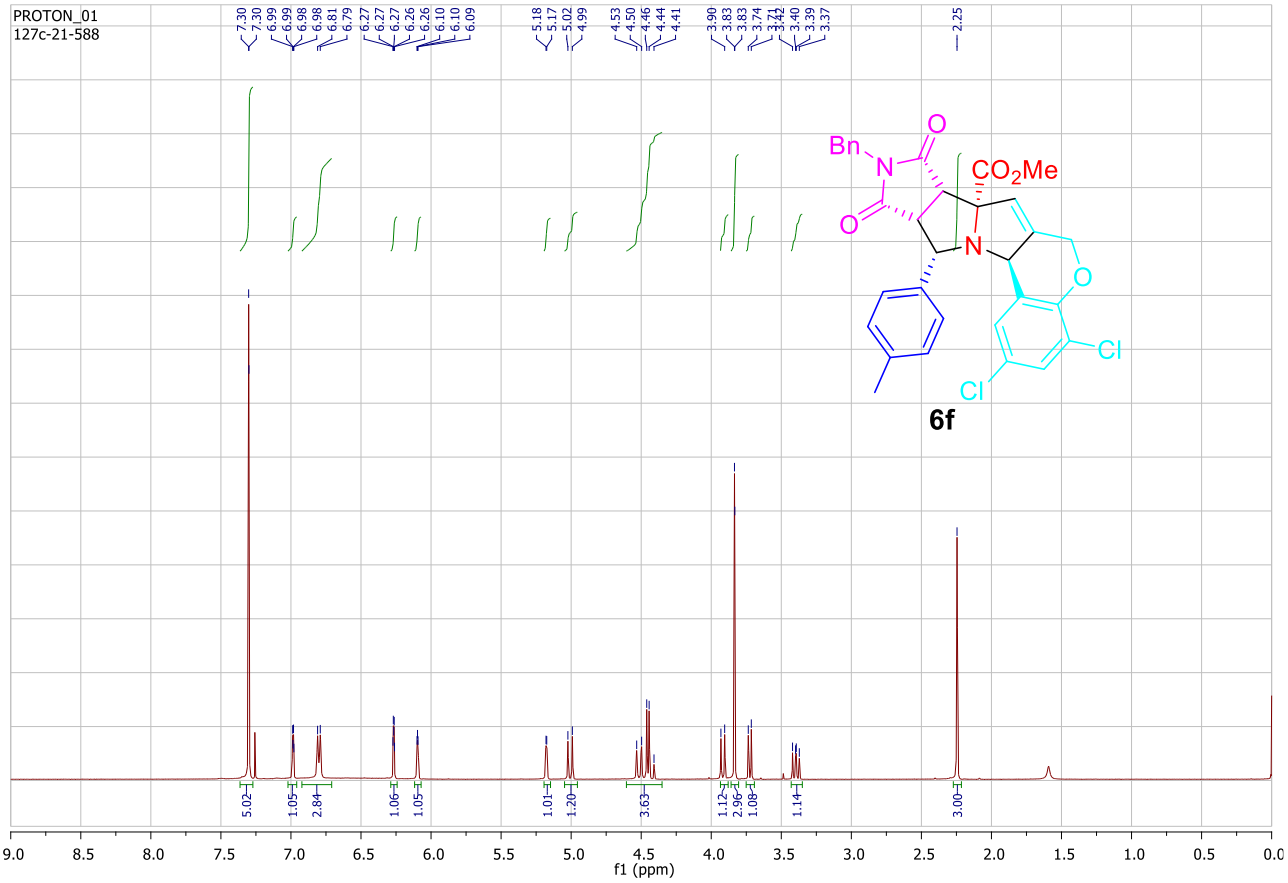
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6.27
6.27
6.26
6.26
6.10
6.10
6.09
5.18
5.17
5.02
4.99
4.53
4.50
4.46
4.44
4.41
3.90
3.83
3.83
3.74
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3.39
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2.25
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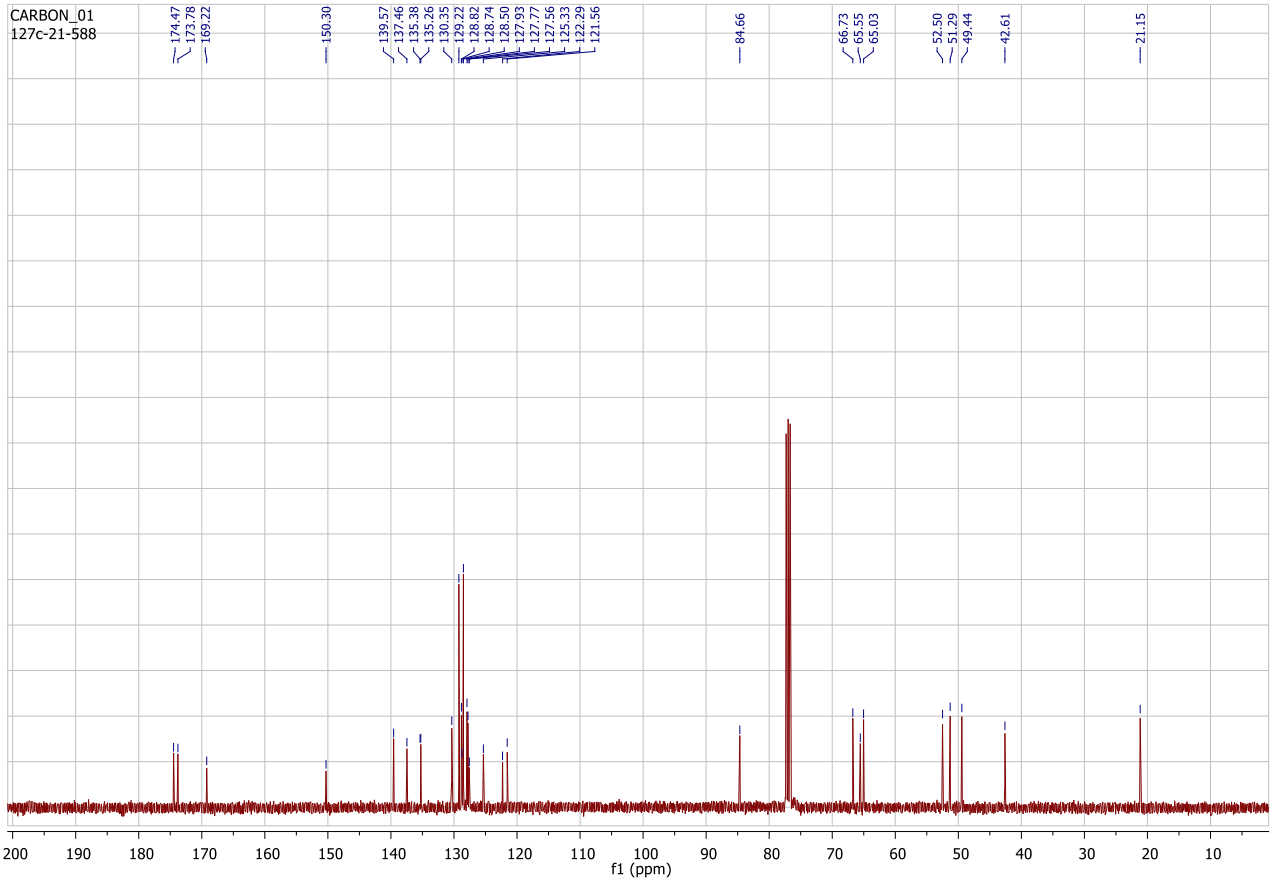
2.84
1.06
1.05
1.01
1.20
3.63
1.12
2.96
1.08
1.14
3.00
f1 (ppm)

Bn
N
O
O
CO2Me
N
O
Cl
Cl

6f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128.50
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127.77
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49.44
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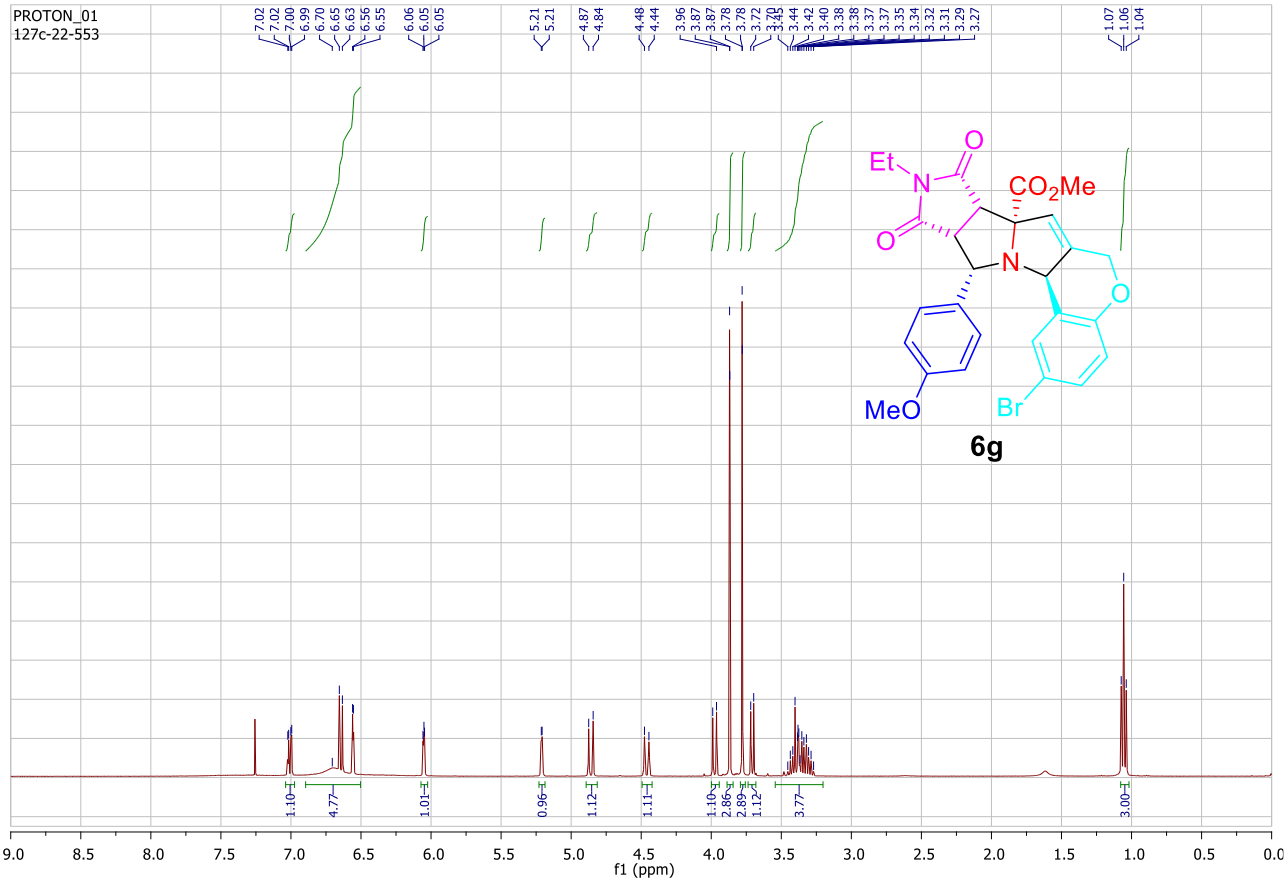
21.15
f1 (ppm)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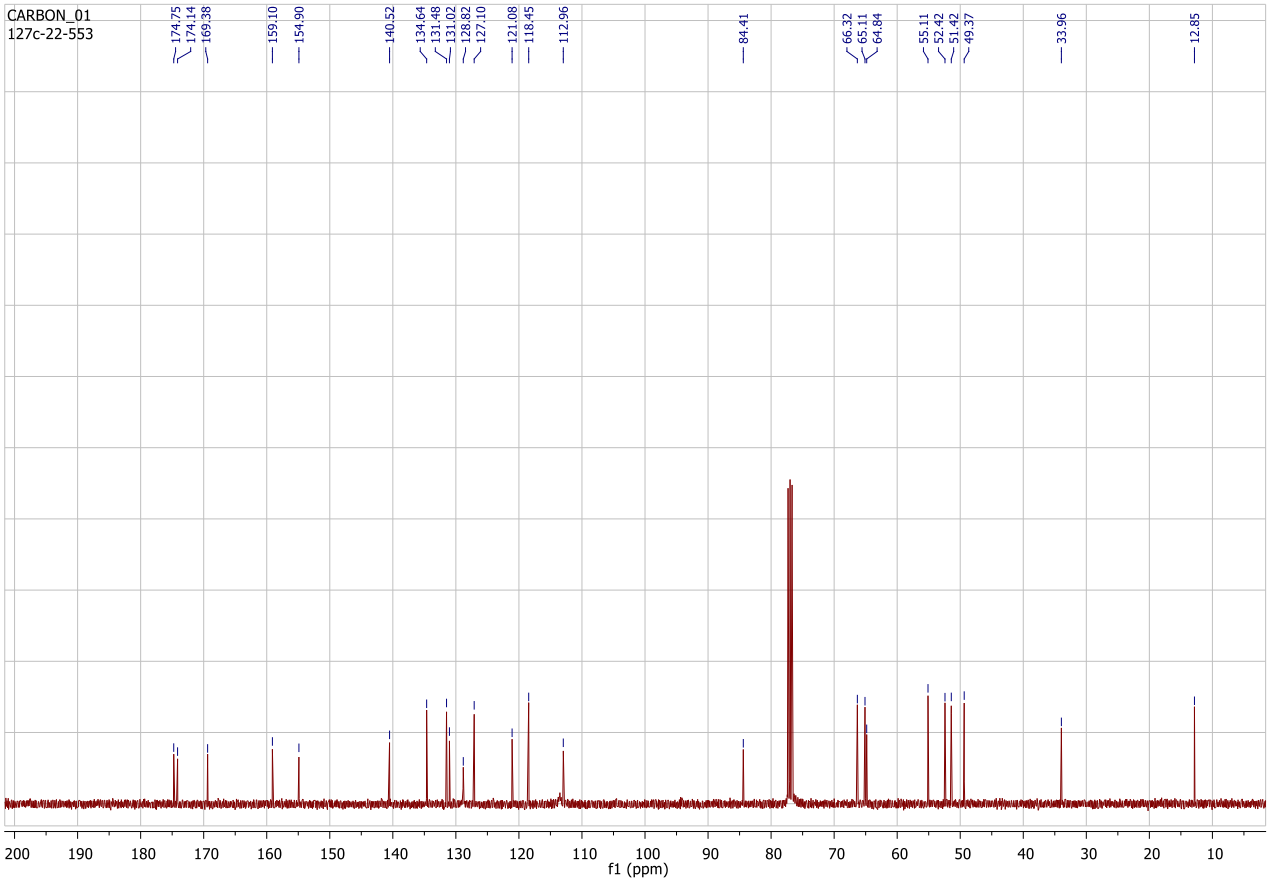
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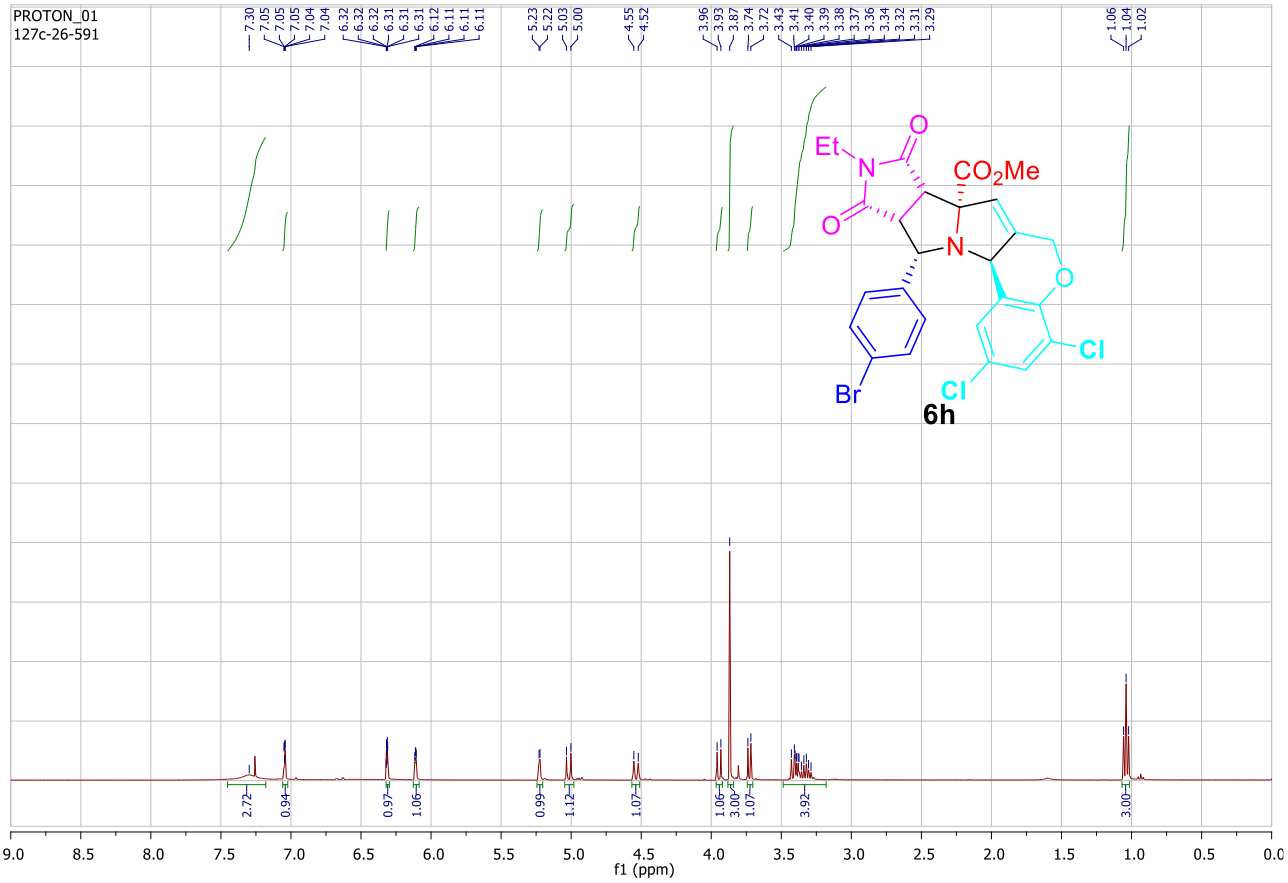
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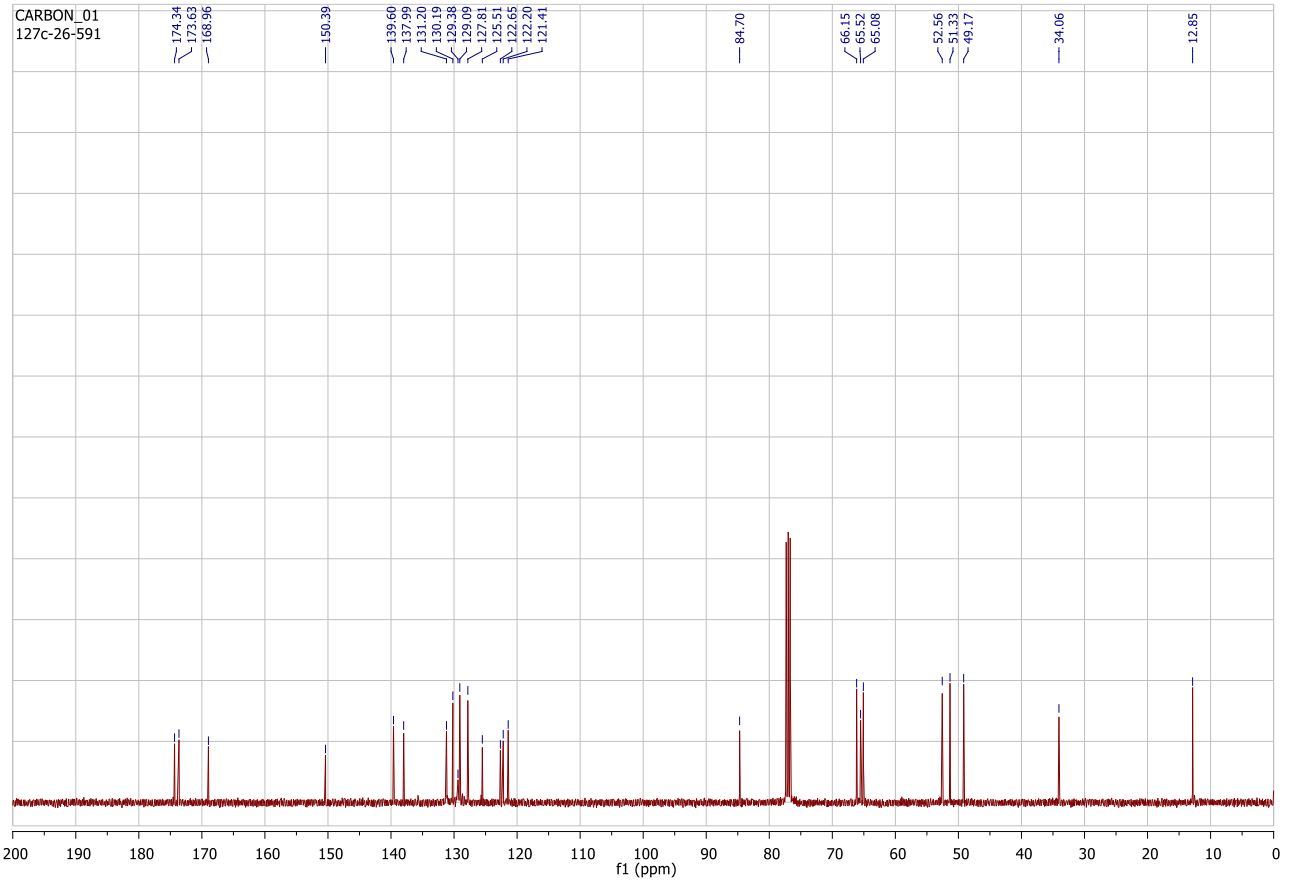
Cl
Br
6h
f1 (ppm)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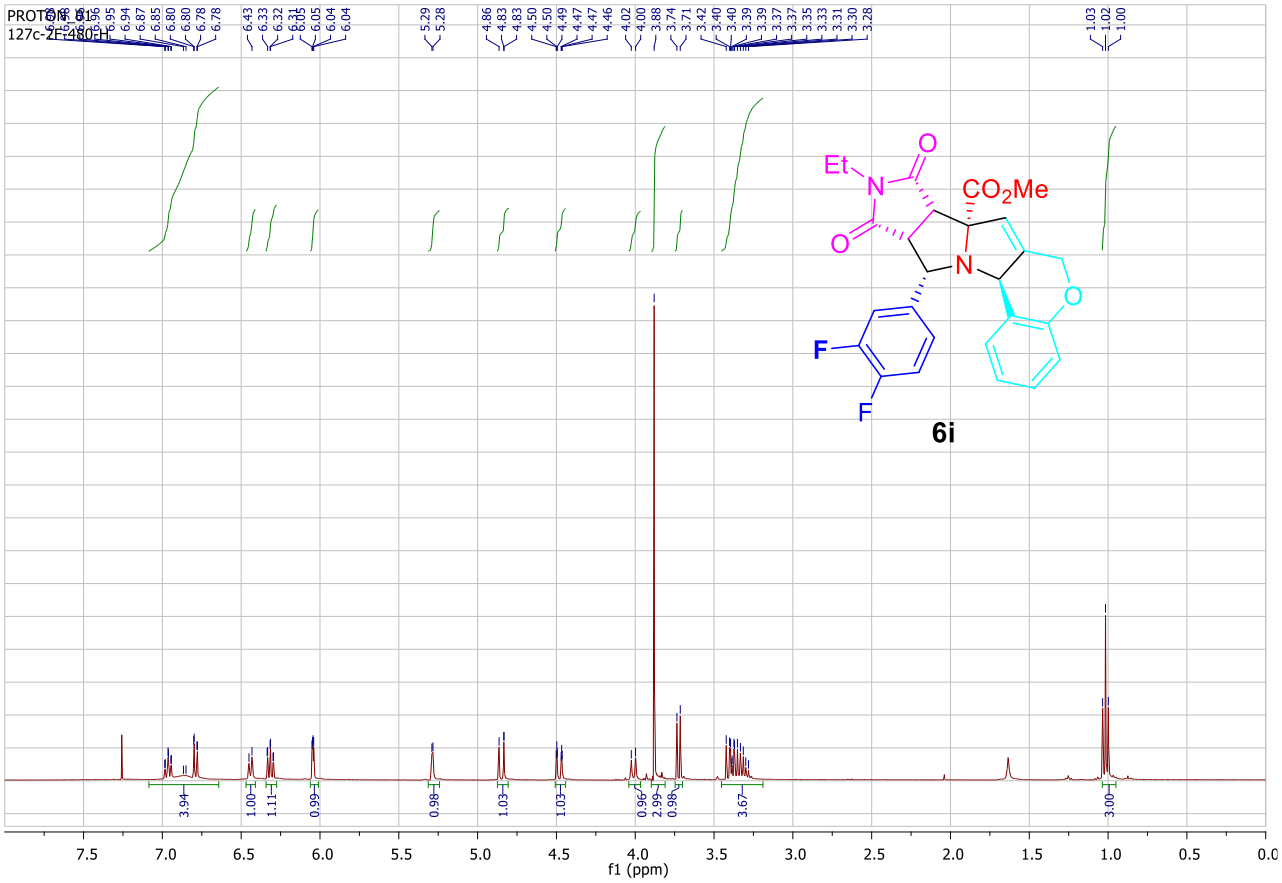
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O
F
F
6i
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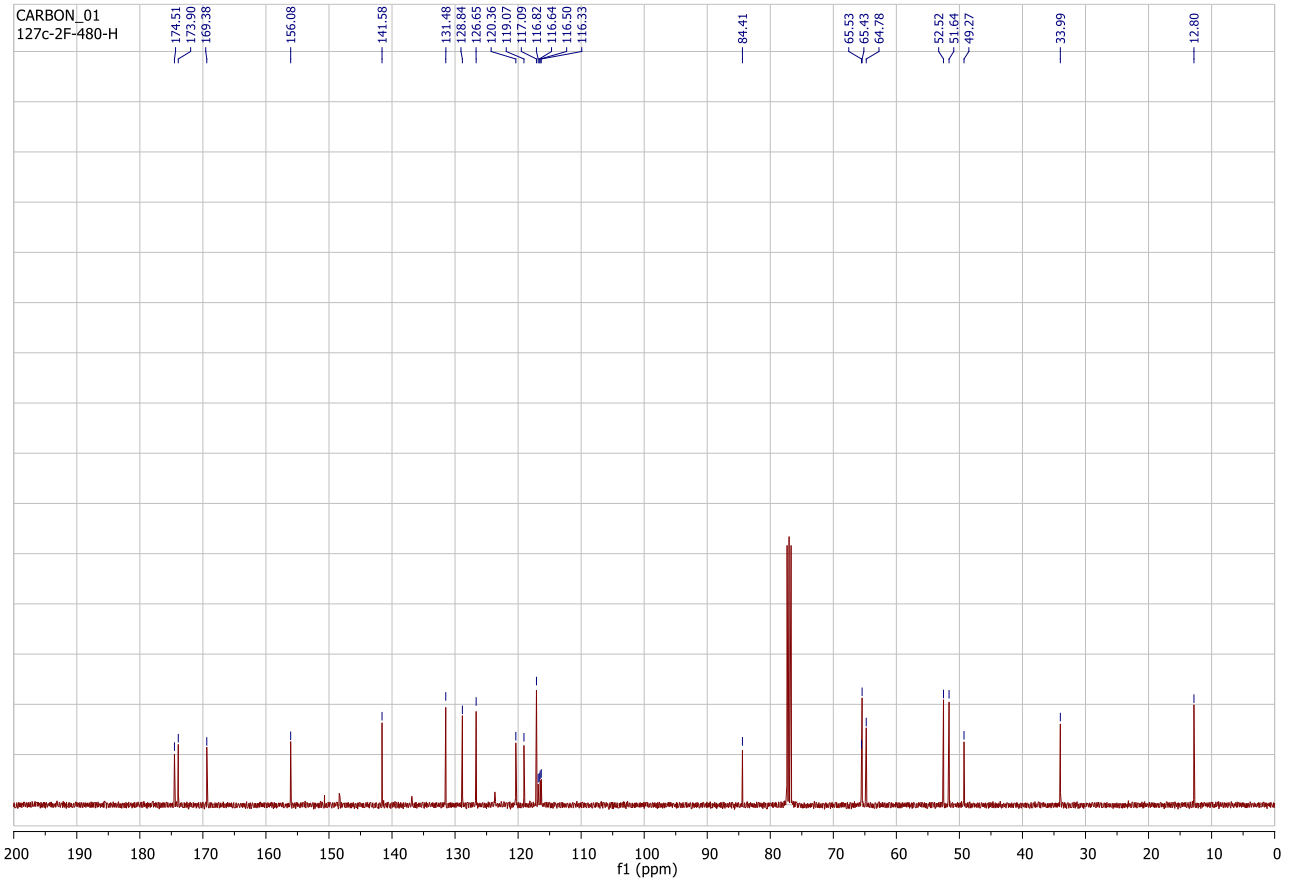
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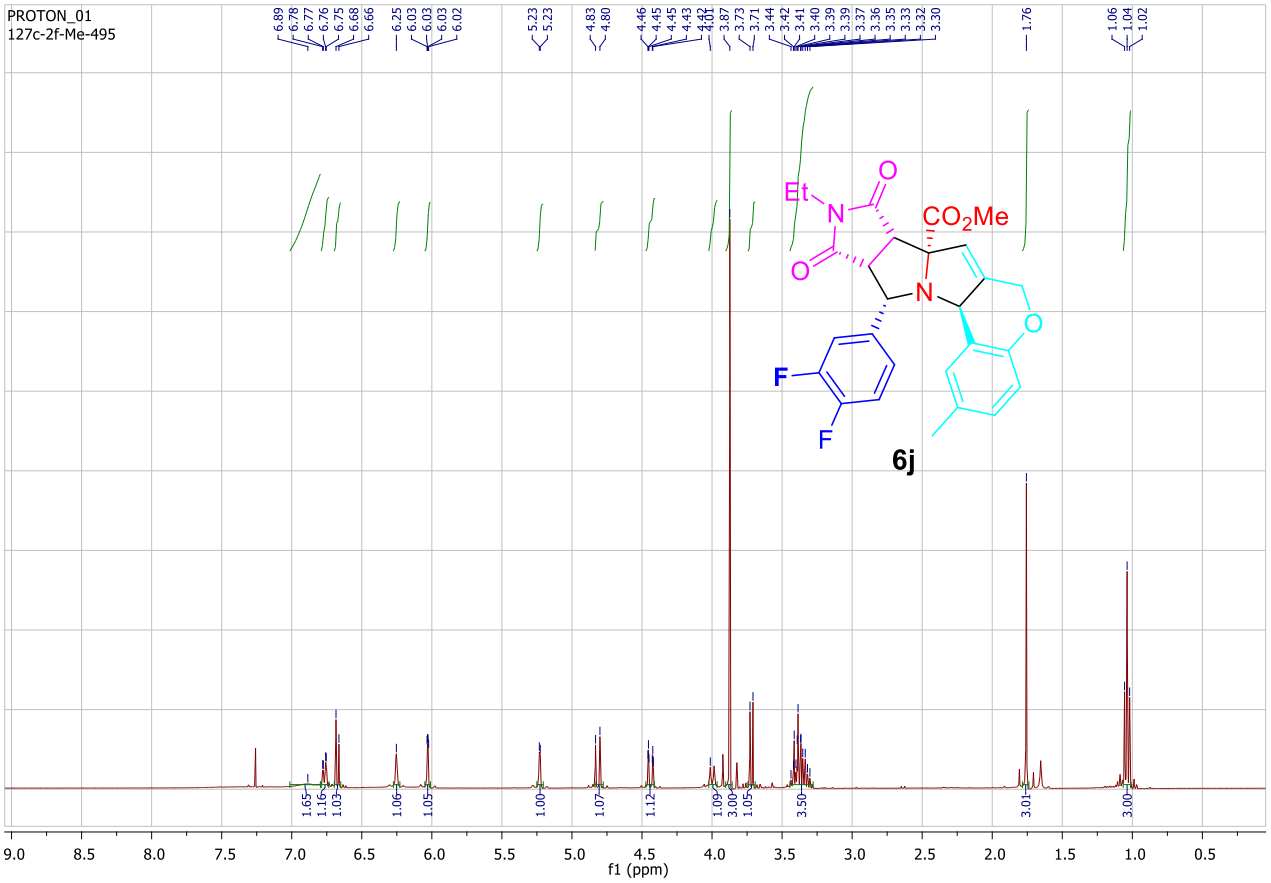
f1 (ppm)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1.65 1.16 1.03 1.06 1.05 1.00 1.07 1.12 1.09 3.00 1.05 3.50 3.01 3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
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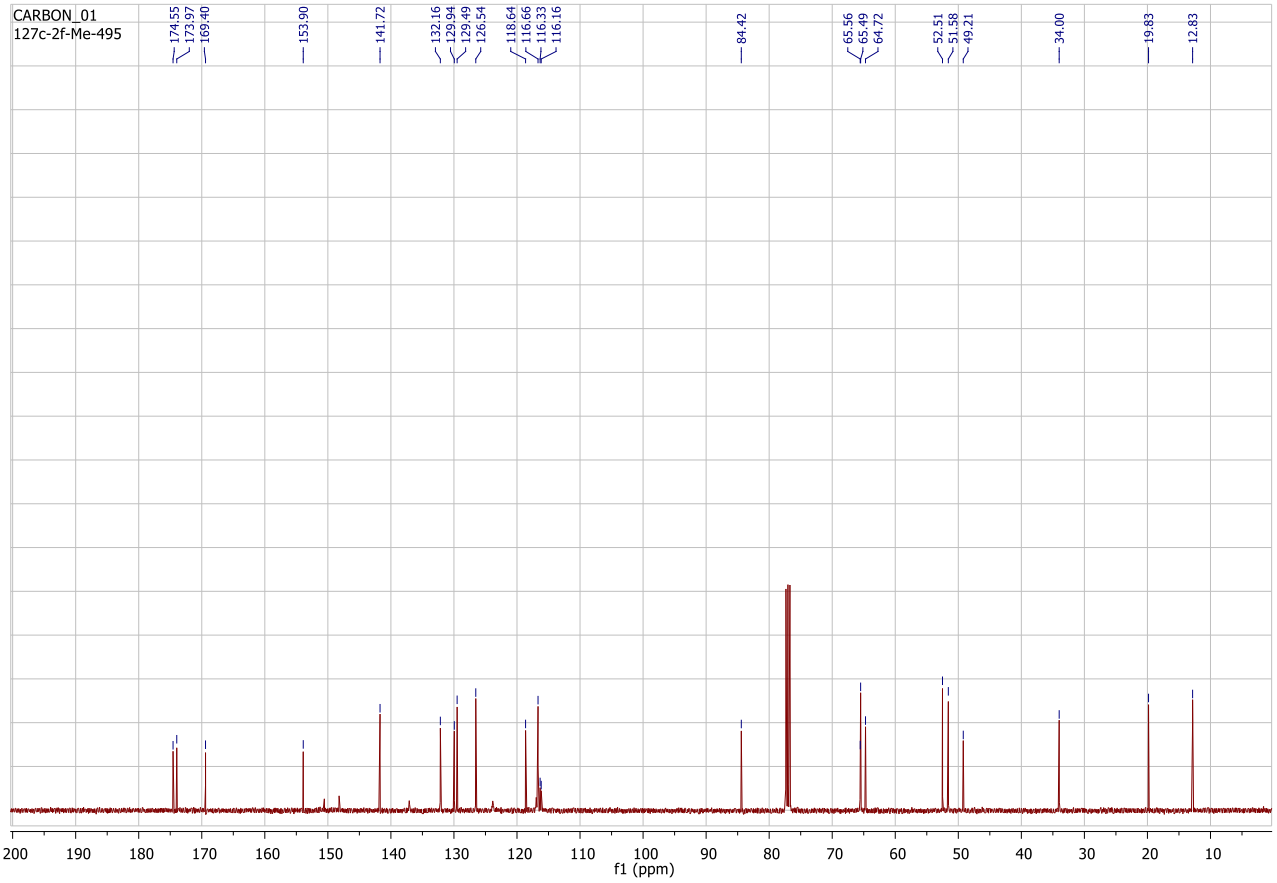
O
O
CO2Me
N
O
F
F
6j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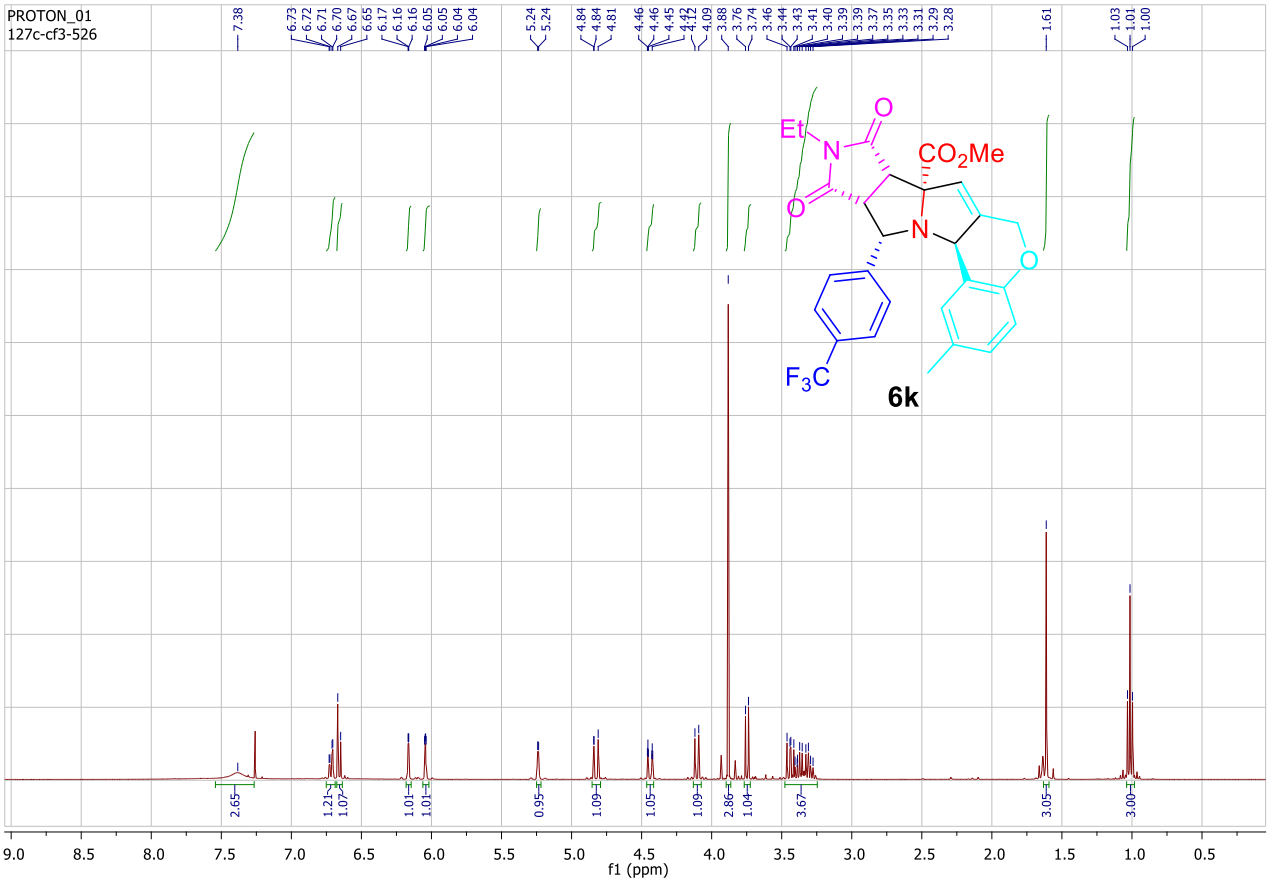
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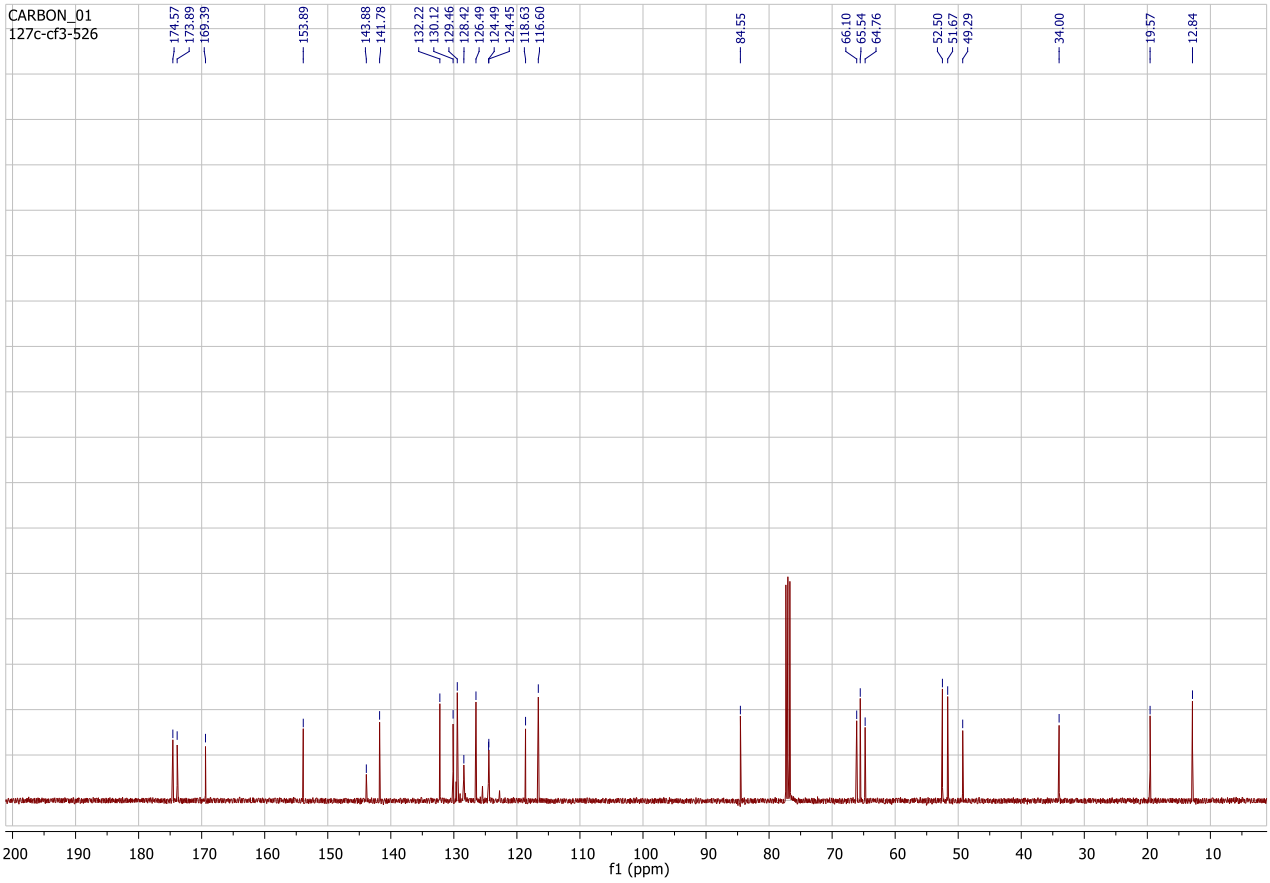
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124.49
124.45
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116.60
84.55
66.10
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64.76
52.50
51.67
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12.84
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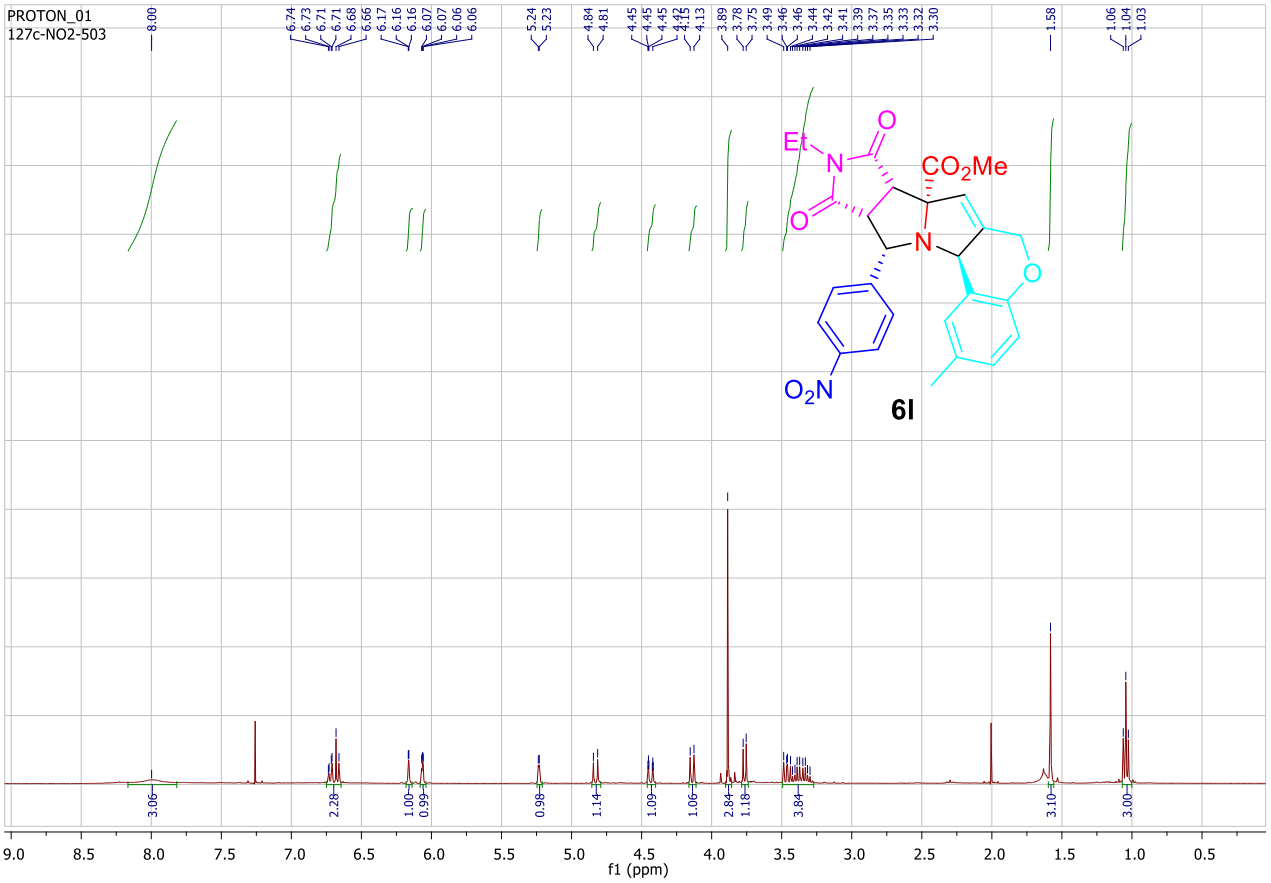
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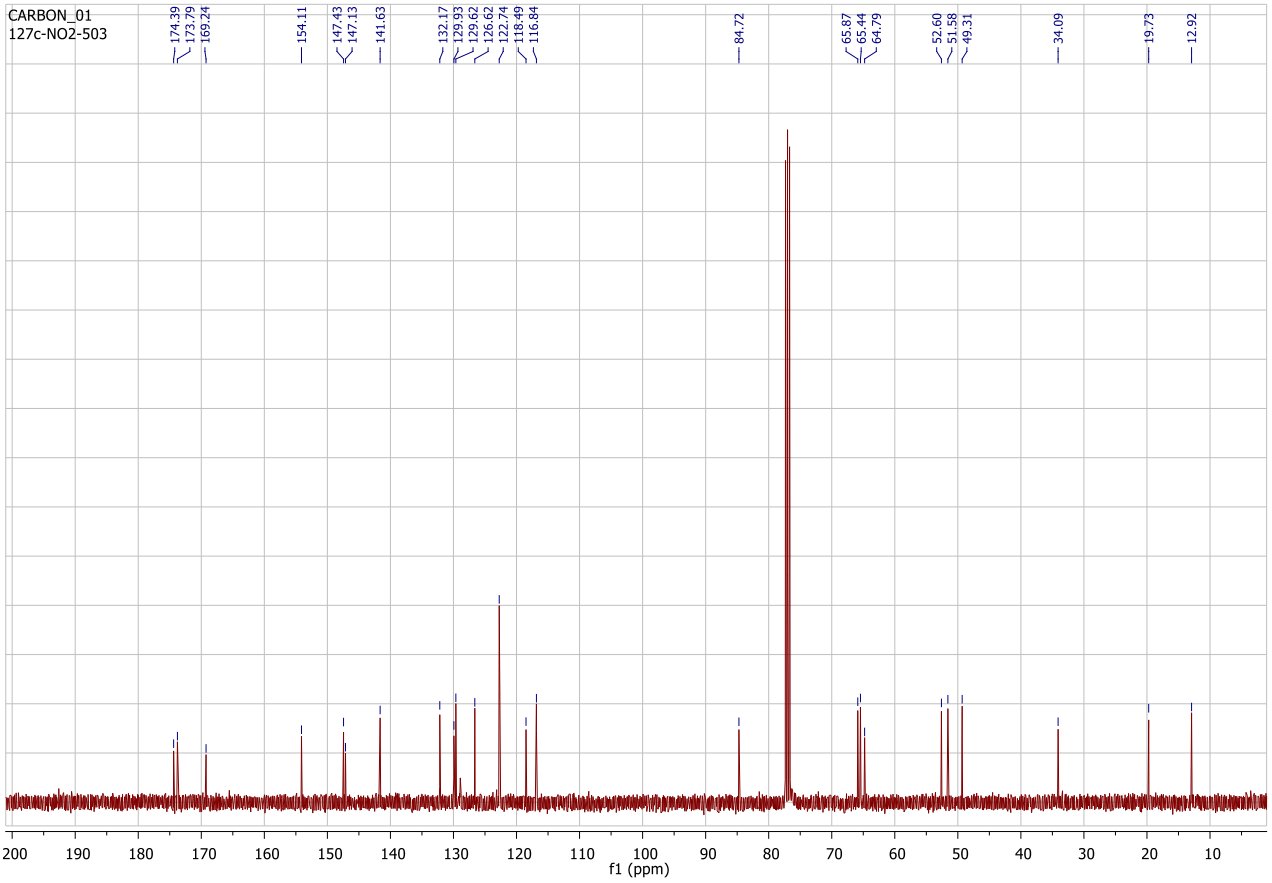
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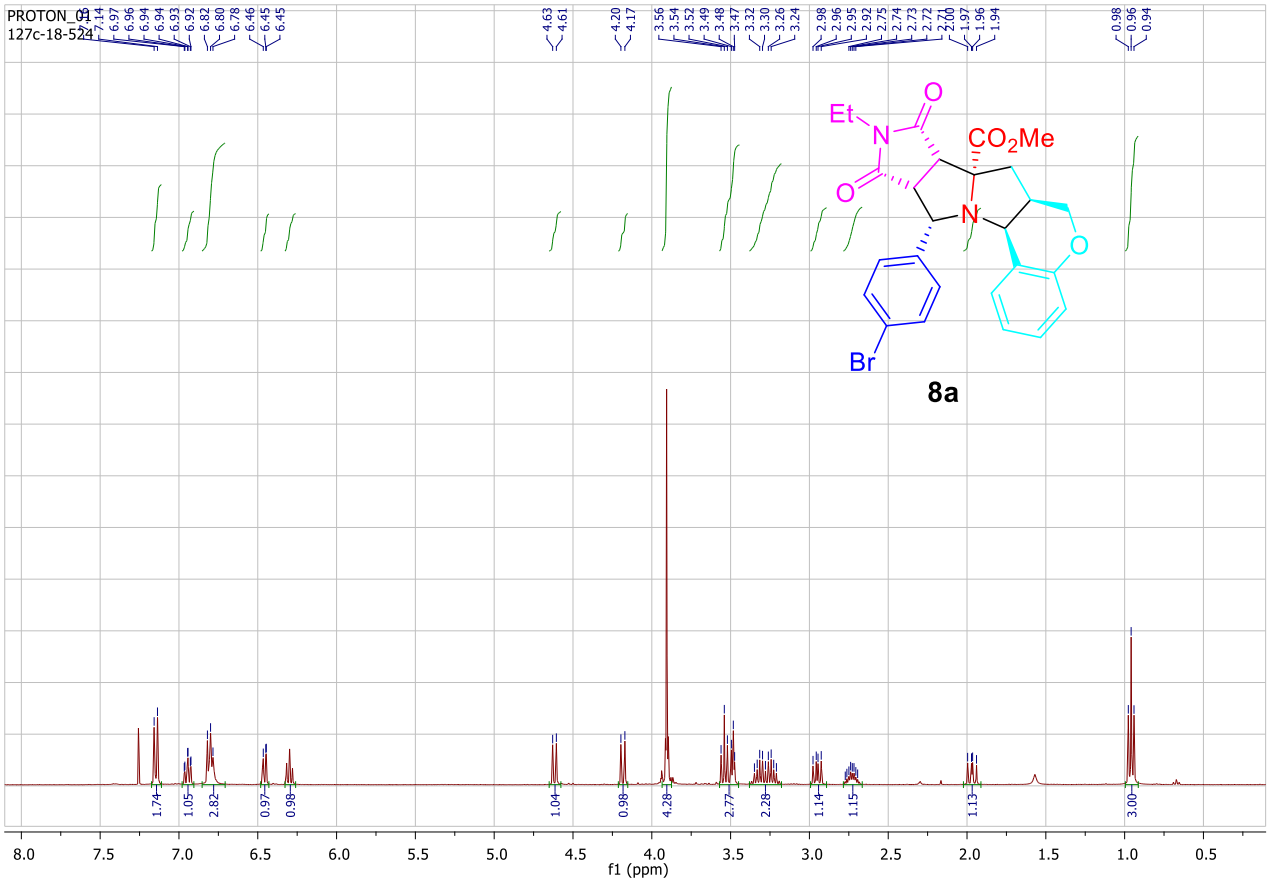
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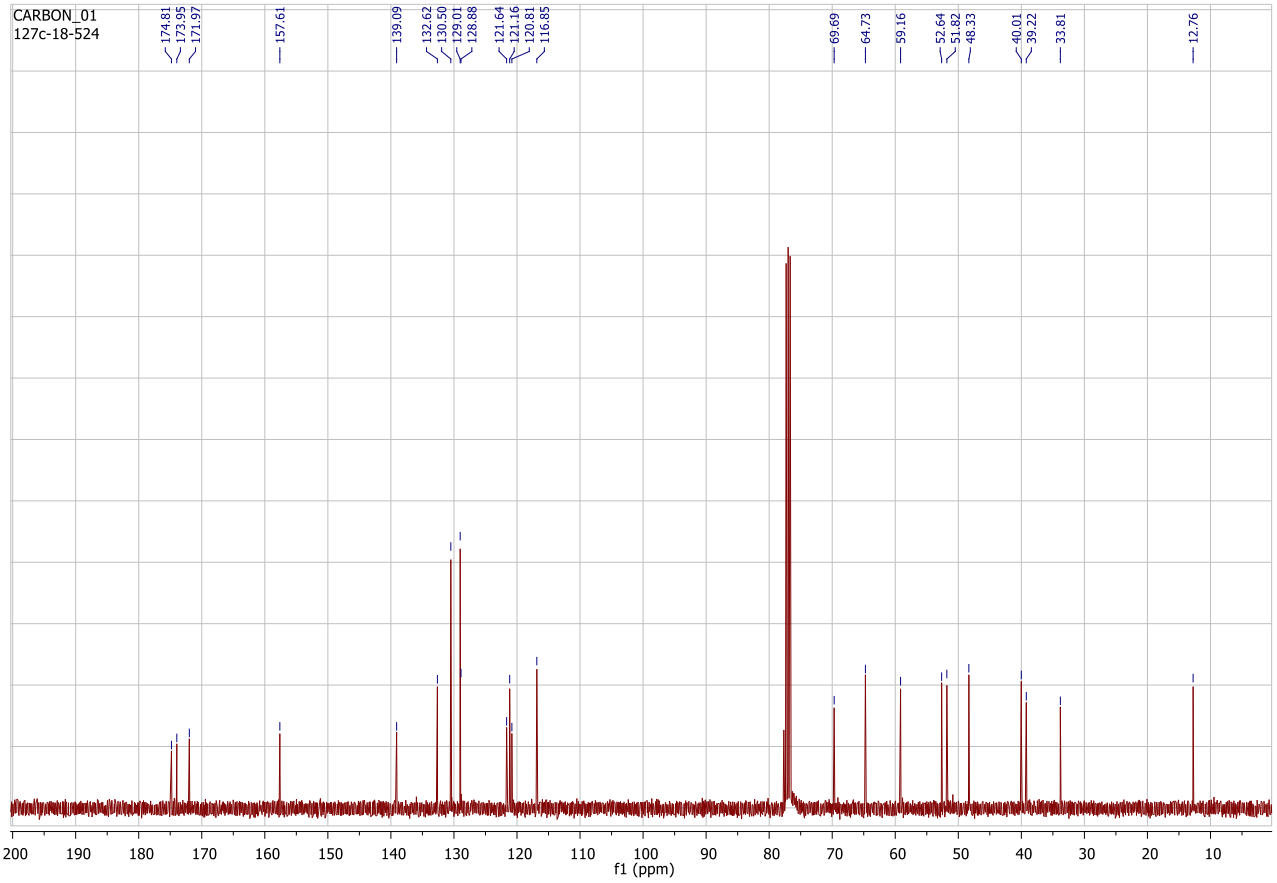
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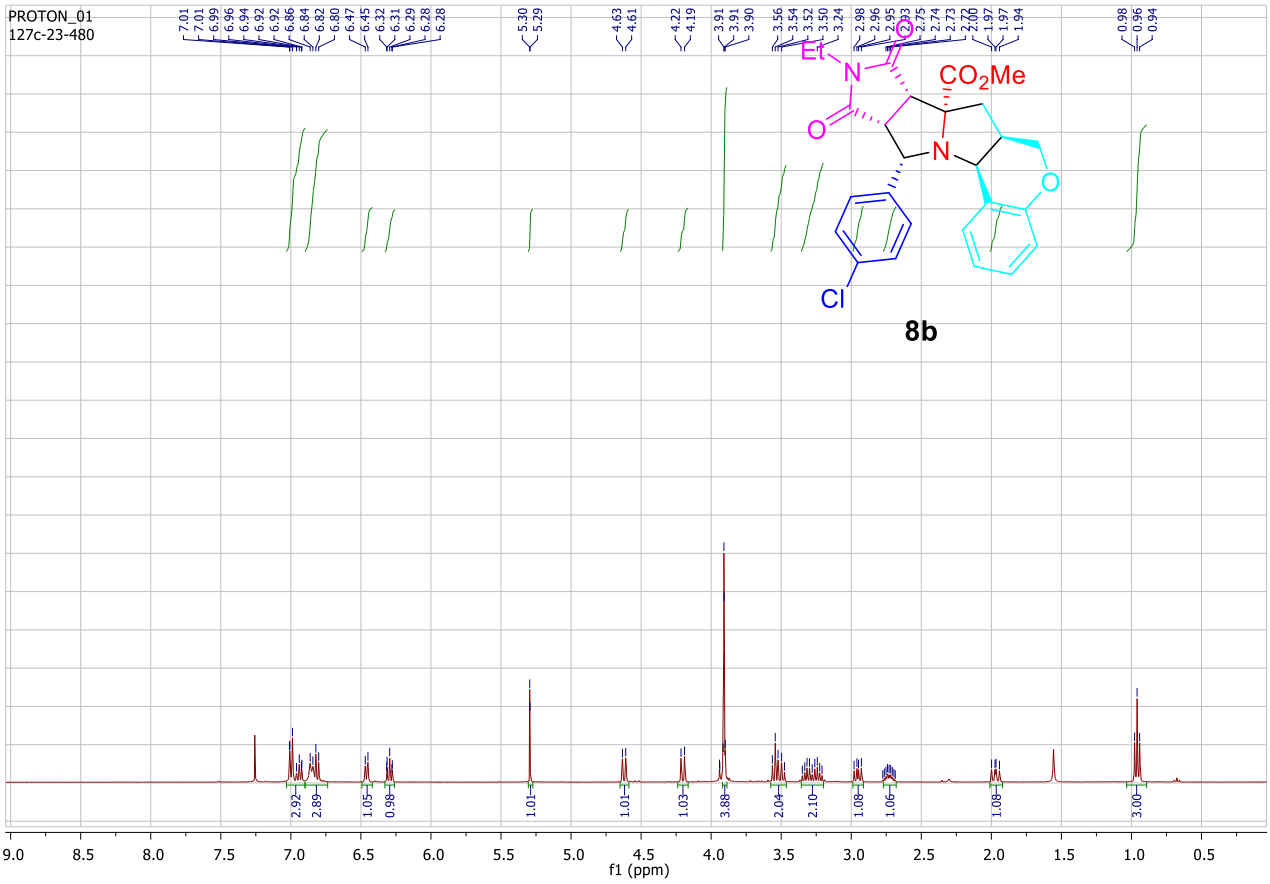
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CO2Me
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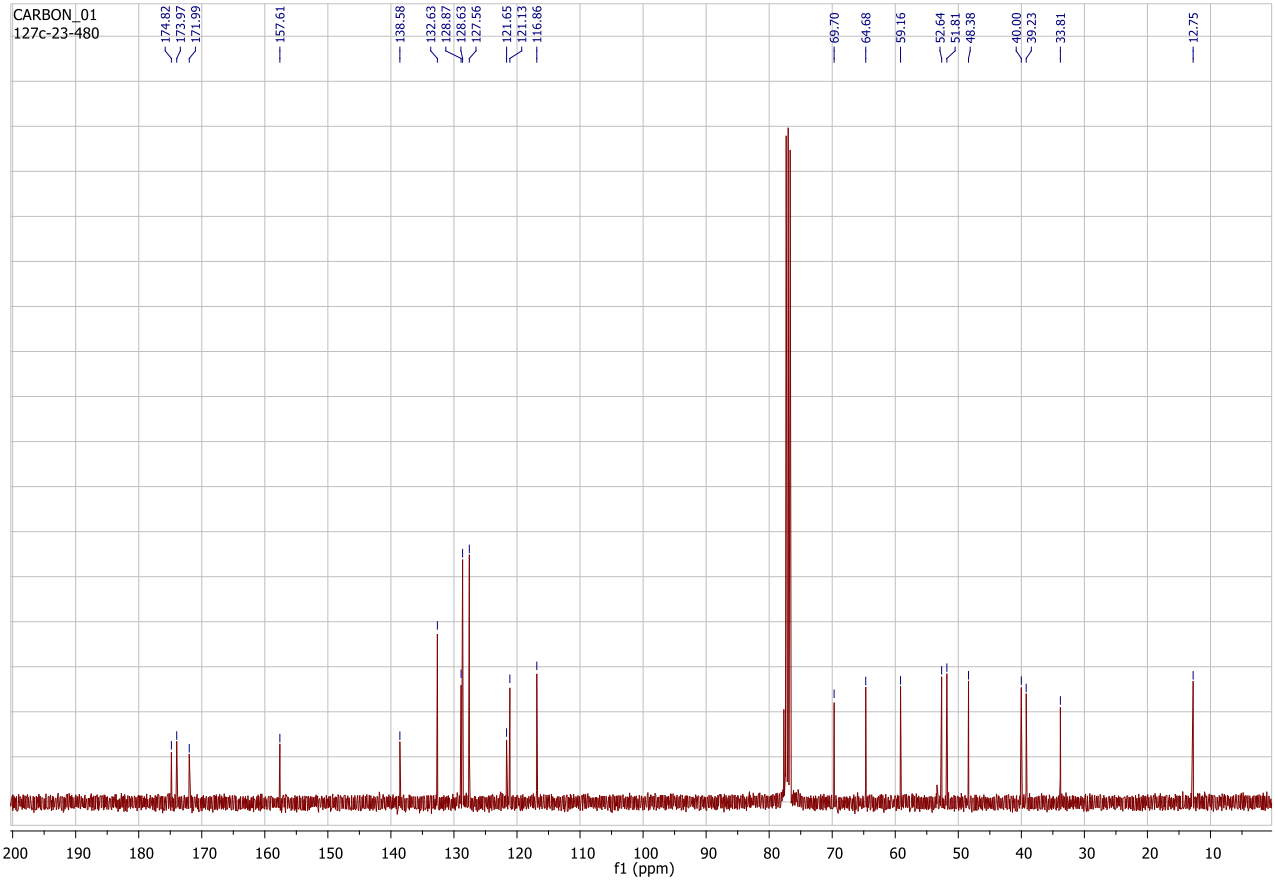
O
Cl
8b
f1 (ppm)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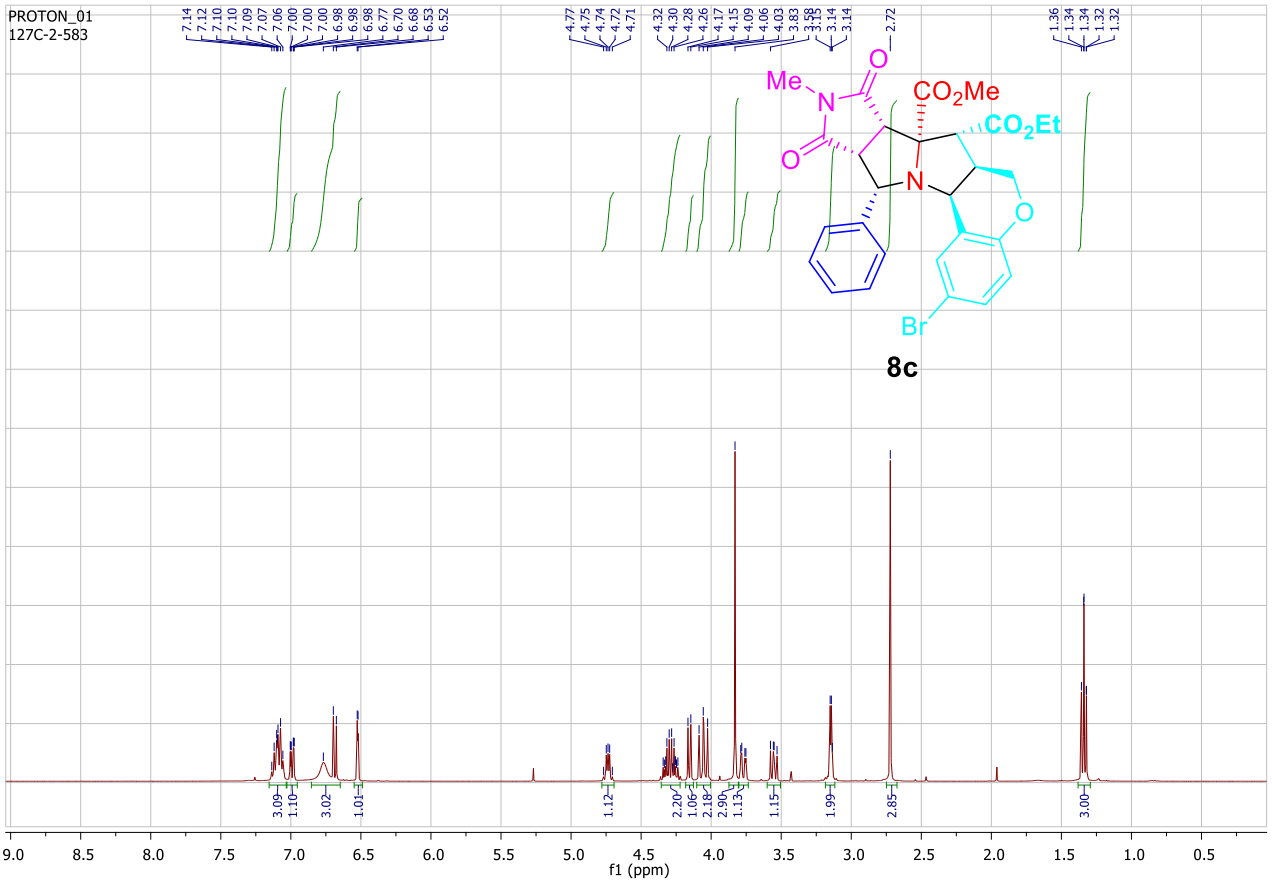
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4.77 4.75 4.74 4.72 4.71
4.32 4.30 4.28 4.26 4.17 4.15 4.09 4.06 4.03 3.83 3.58 3.55 3.14 3.14
2.72
1.36 1.34 1.34 1.32 1.32
3.09 1.10 3.02 1.01 1.12 2.20 1.06 2.18 2.90 1.13 1.15 1.99 2.85 3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)
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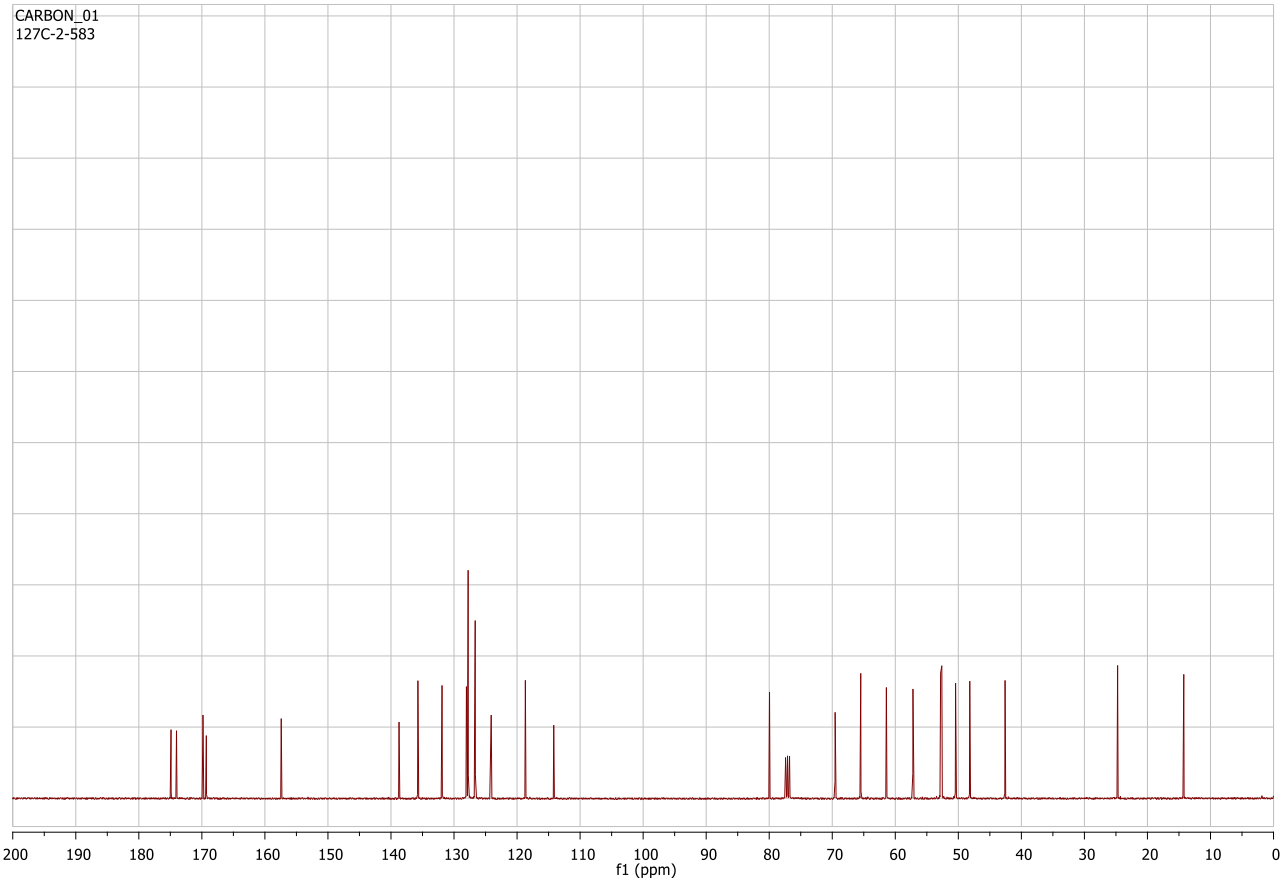
O
O
CO2Me
CO2Et
N
O
Br
8c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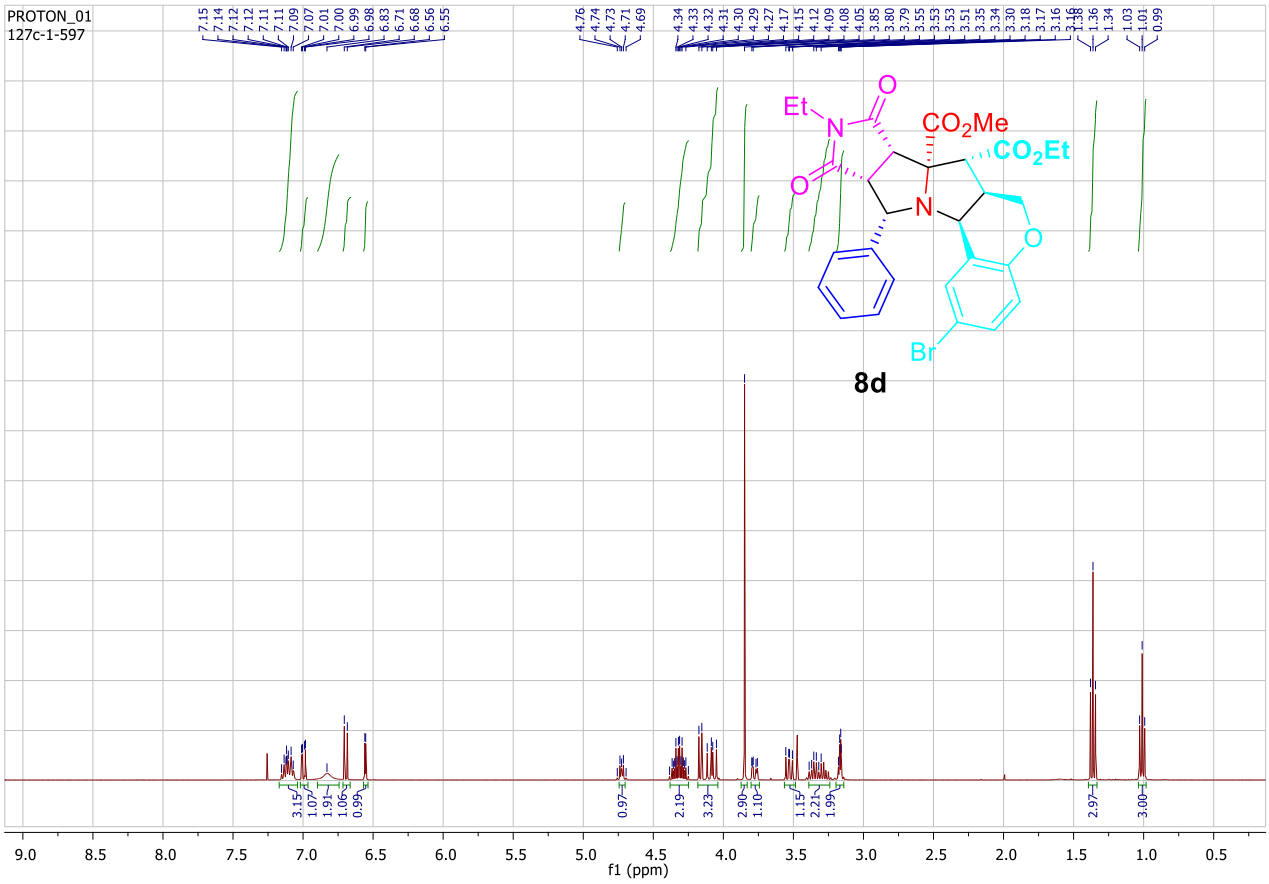
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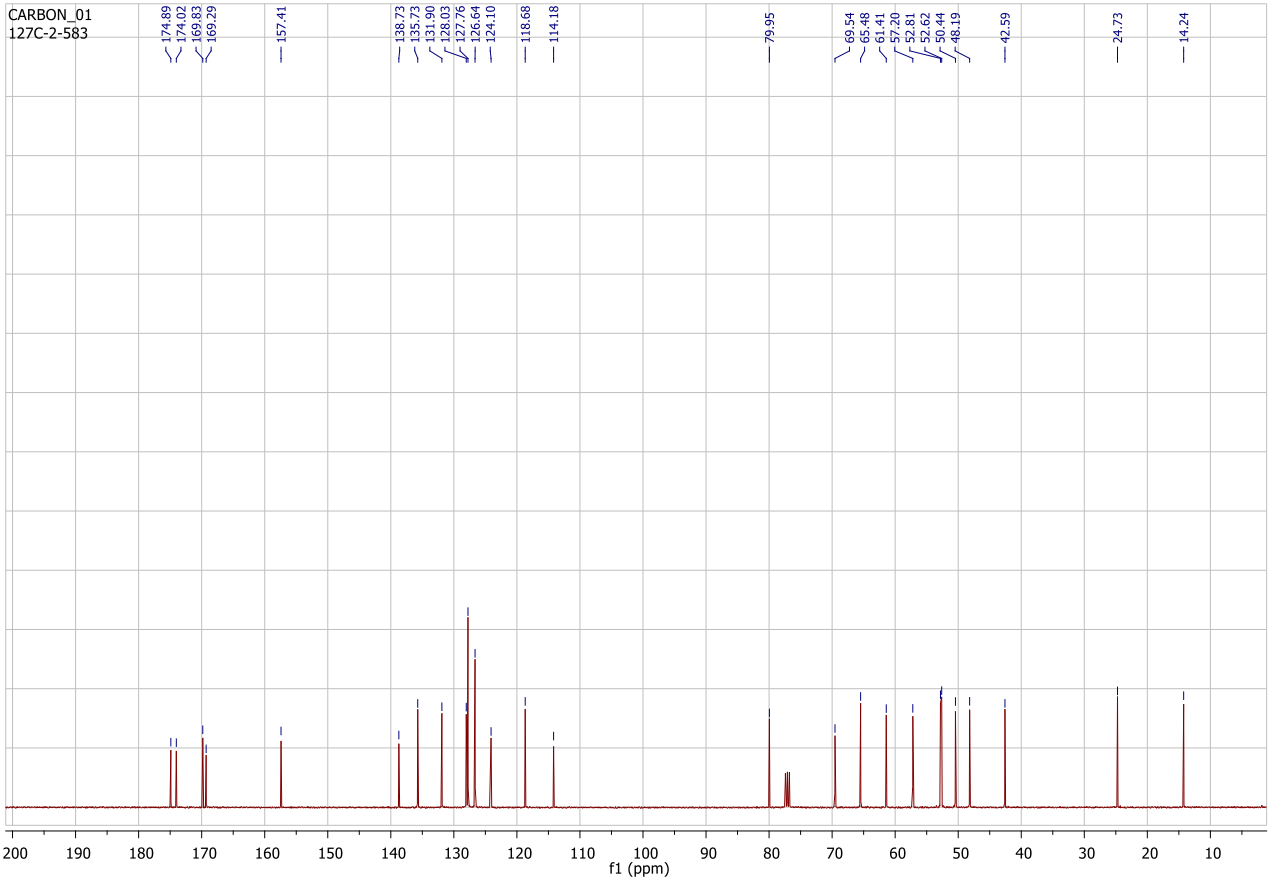
8d
f1 (ppm)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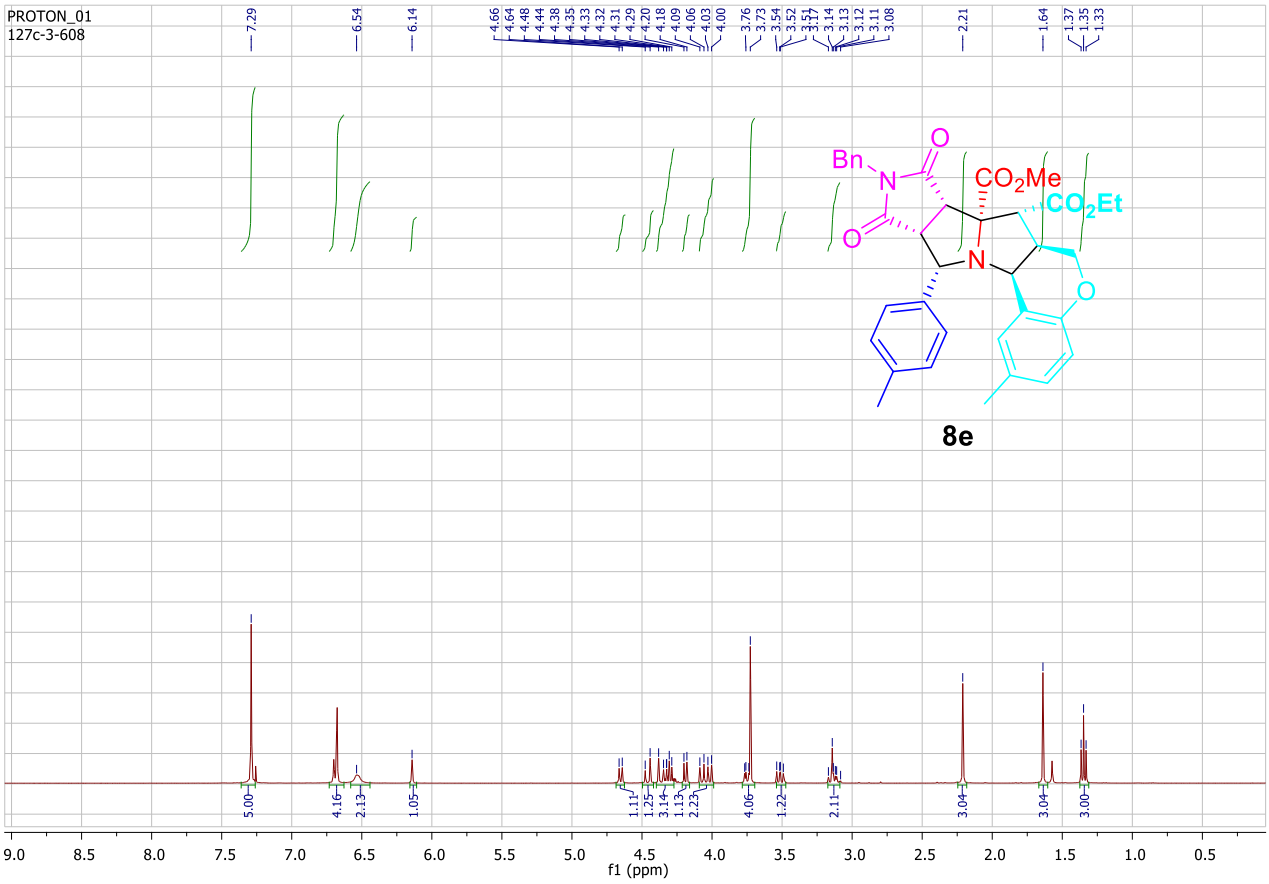
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4.32
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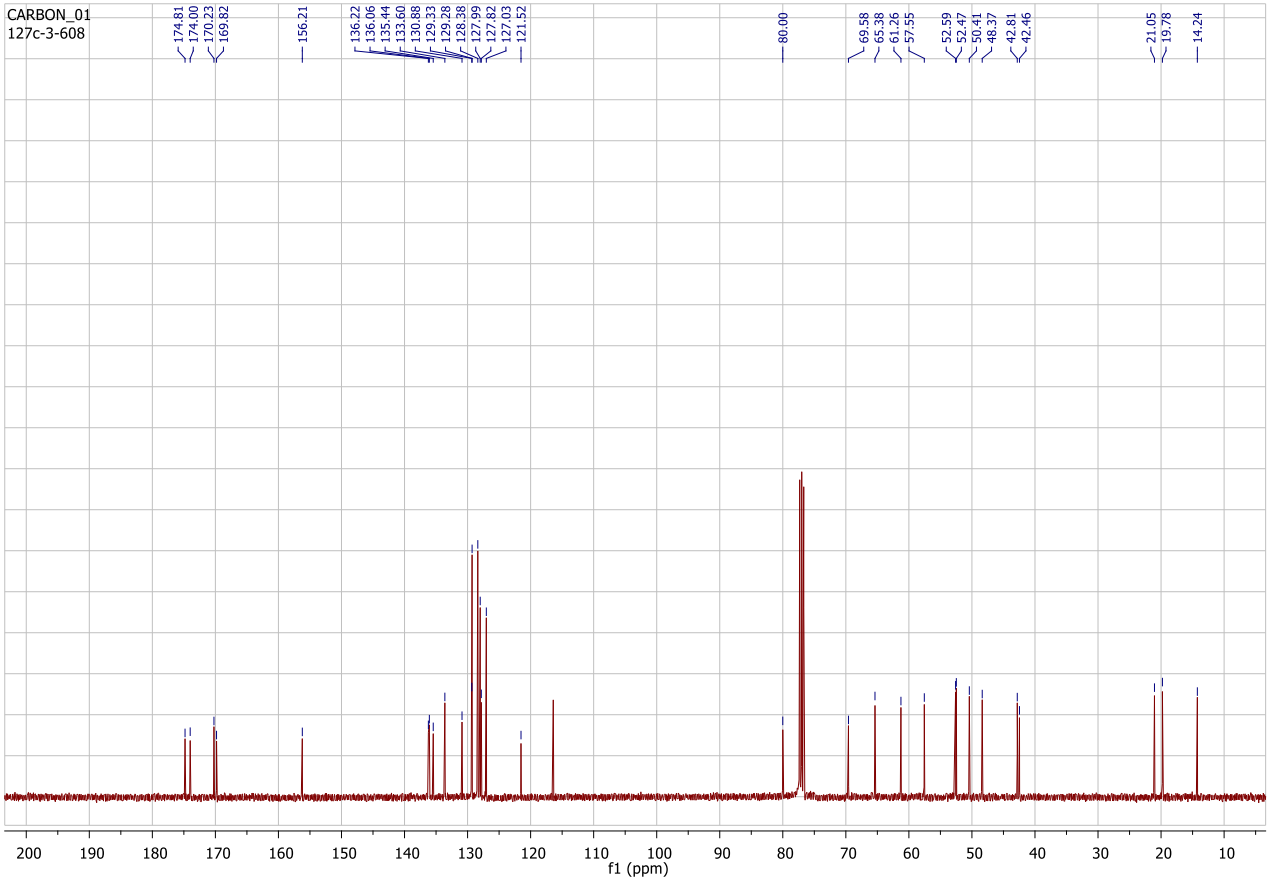
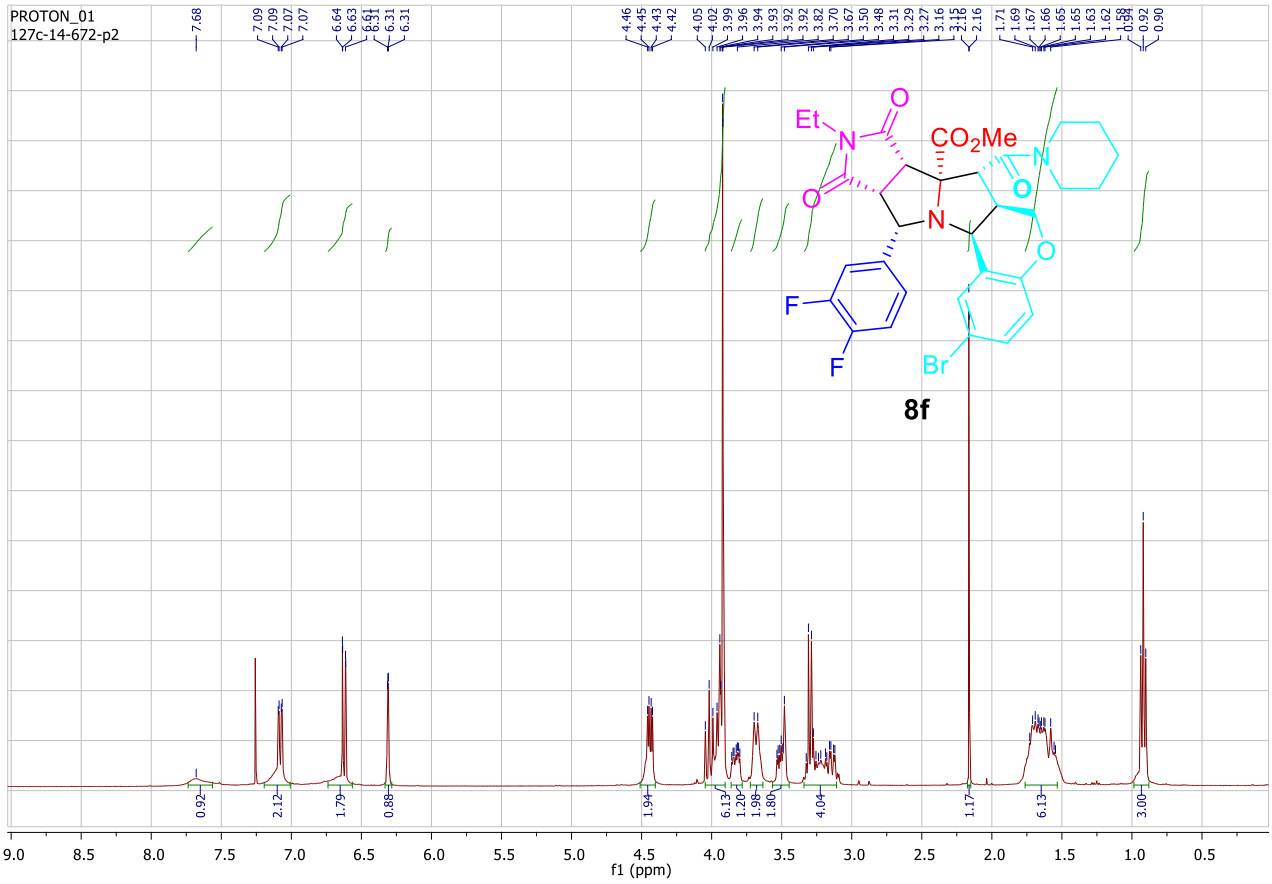
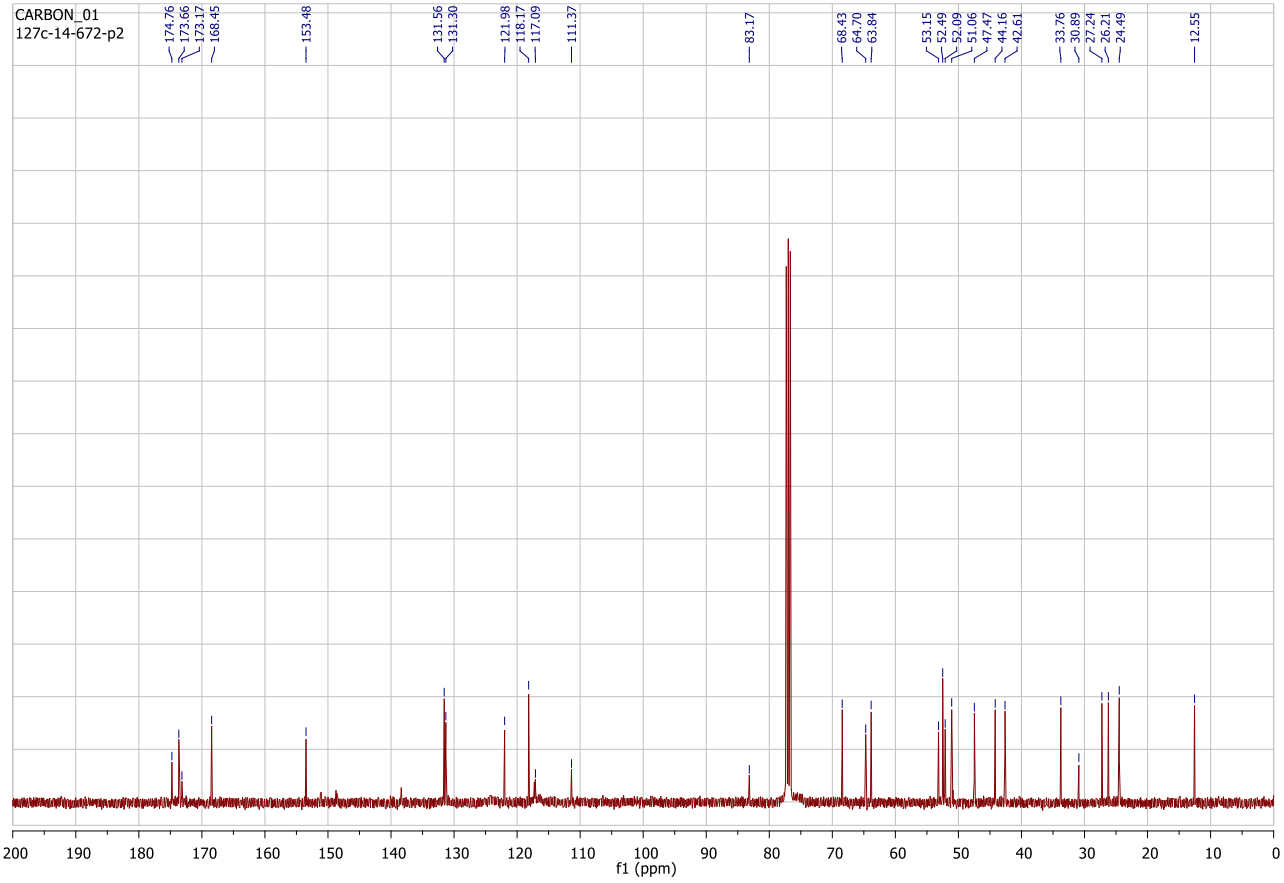
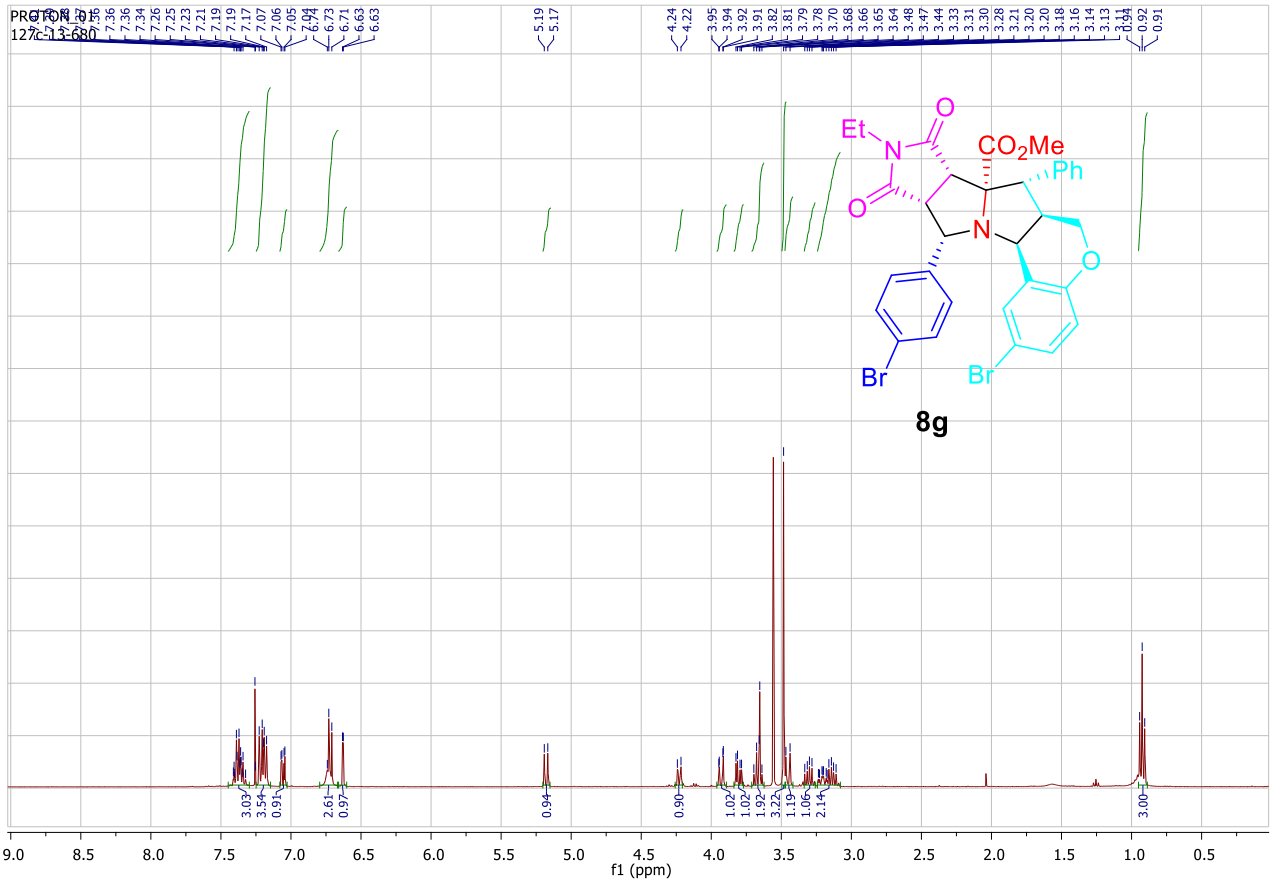
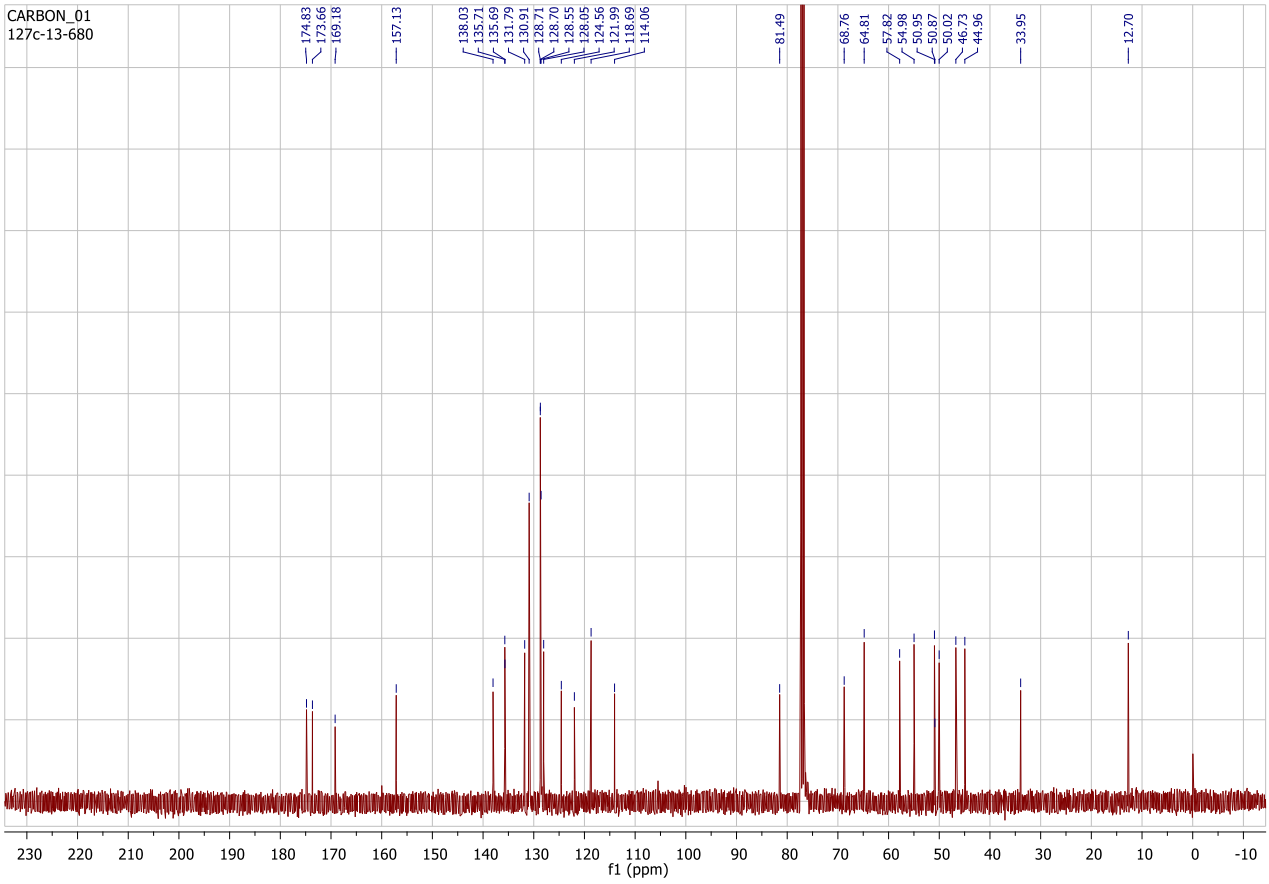
4.18
4.09
4.06
4.03
4.00
3.76
3.73
3.54
3.52
3.14
3.13
3.12
3.11
3.08
2.21
1.64
1.37
1.35
1.33
Bn
N
O
O
CO2Me
CO2Et
N
O
8e
5.00
4.16
2.13
1.05
1.11
1.25
3.14
1.13
2.23
4.06
1.22
2.11
3.04
3.04
3.00
f1 (ppm)

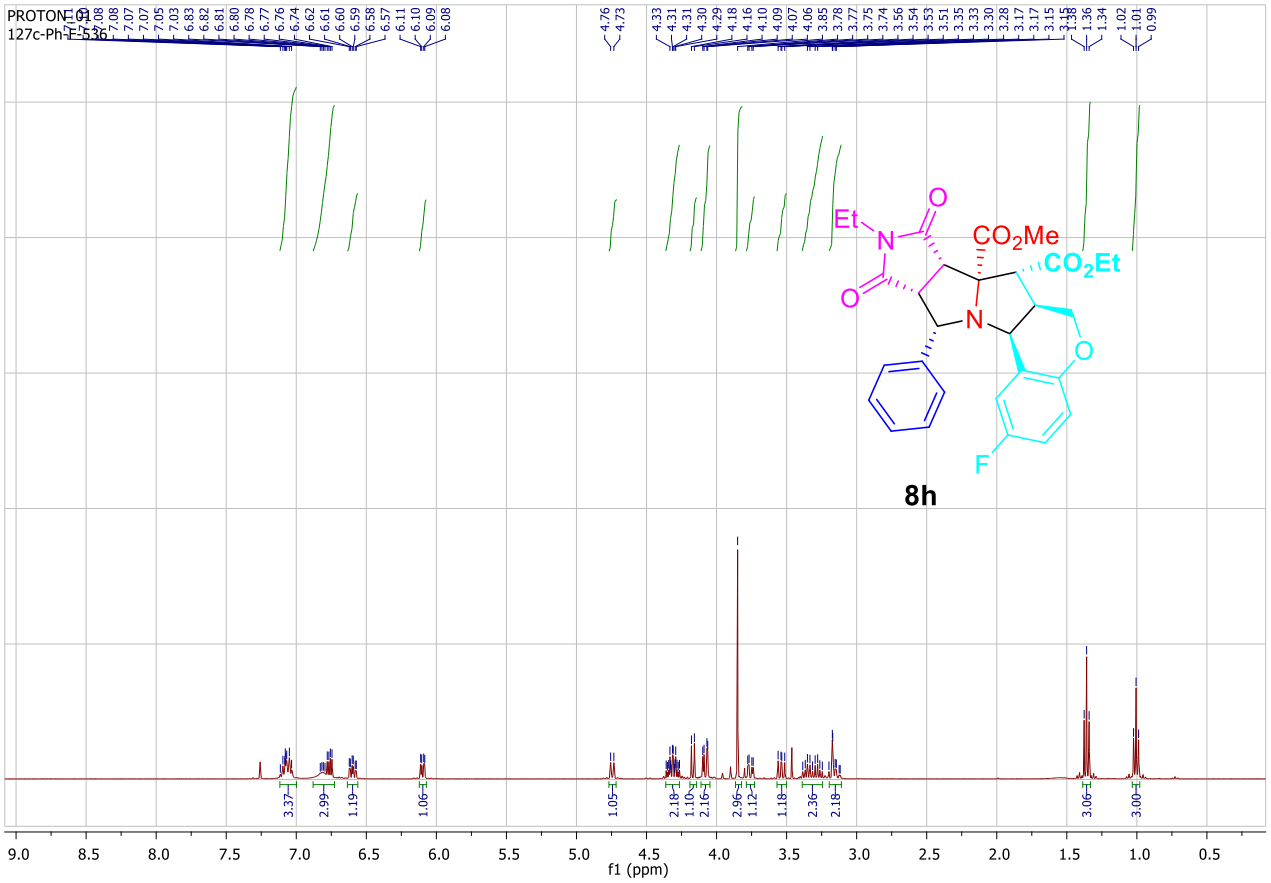

CARBON_01
127c-3-608
174.81
174.00
170.23
169.82
156.21
136.22
136.06
135.44
133.60
130.88
129.33
129.28
128.38
127.99
127.82
127.03
121.52
80.00
69.58
65.38
61.26
57.55
52.59
52.47
50.41
48.37
42.81
42.46
21.05
19.78
14.24
f1 (ppm)

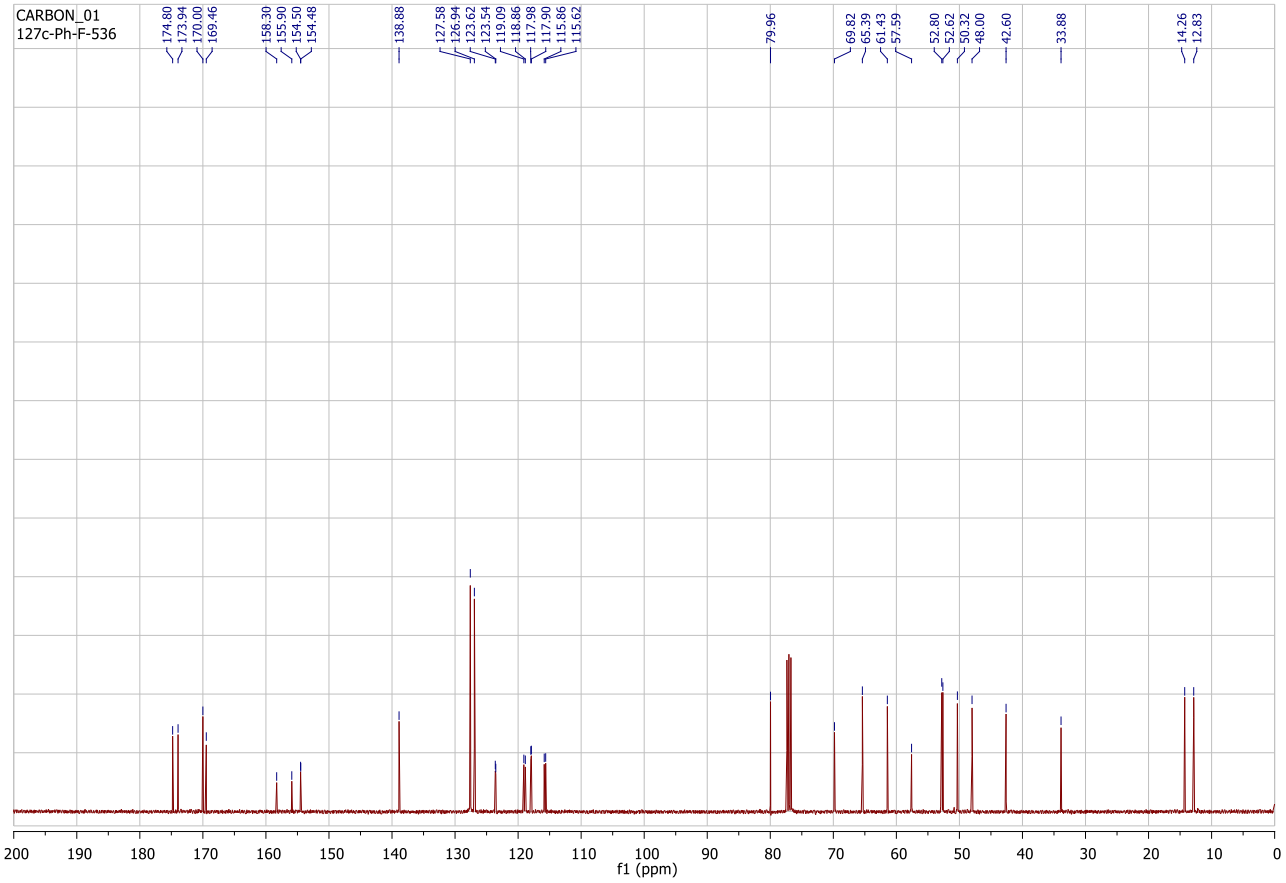

PROTON_01
127c-14-672-p2
Et
CO2Me
F
F
Br
8f
f1 (ppm)

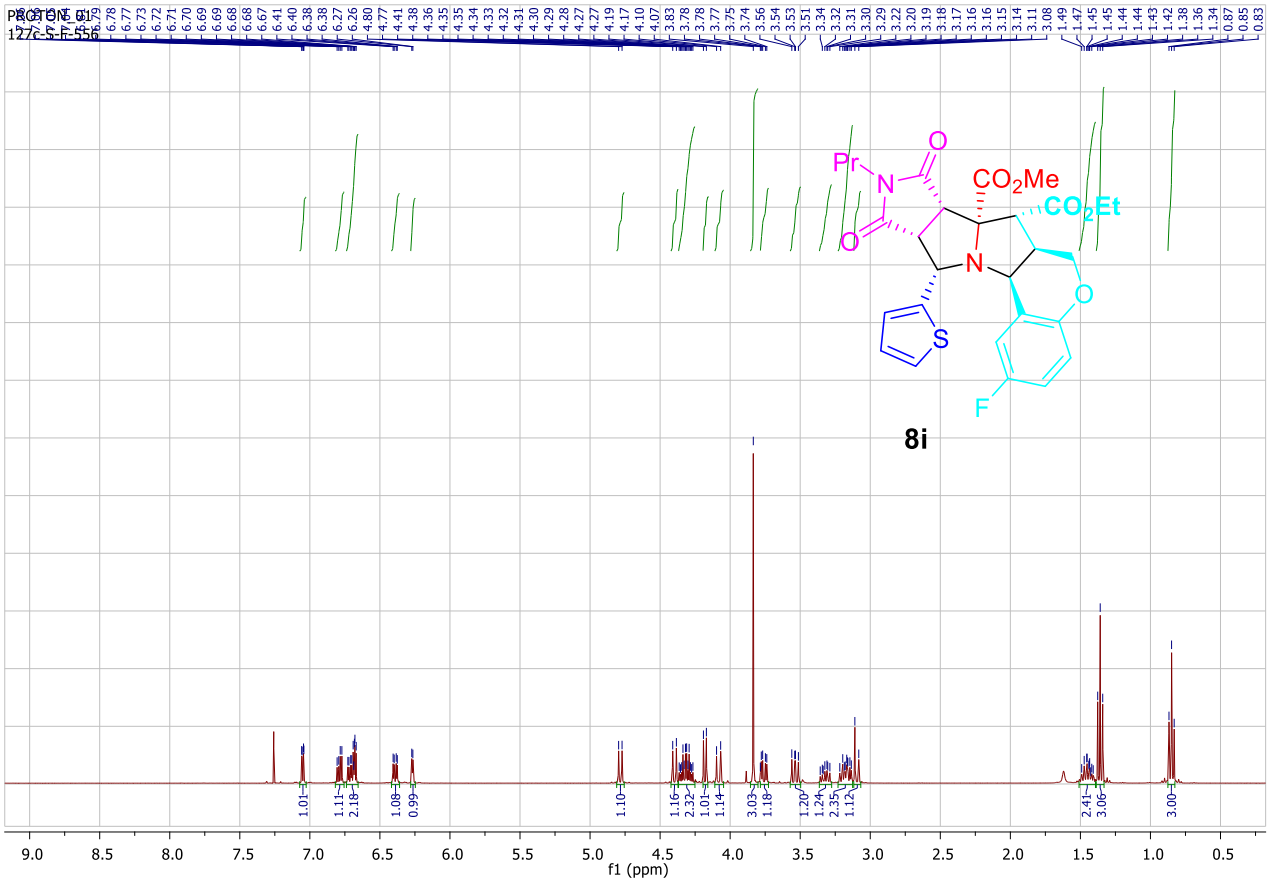

CARBON_01
127c-14-672-p2
f1 (ppm)

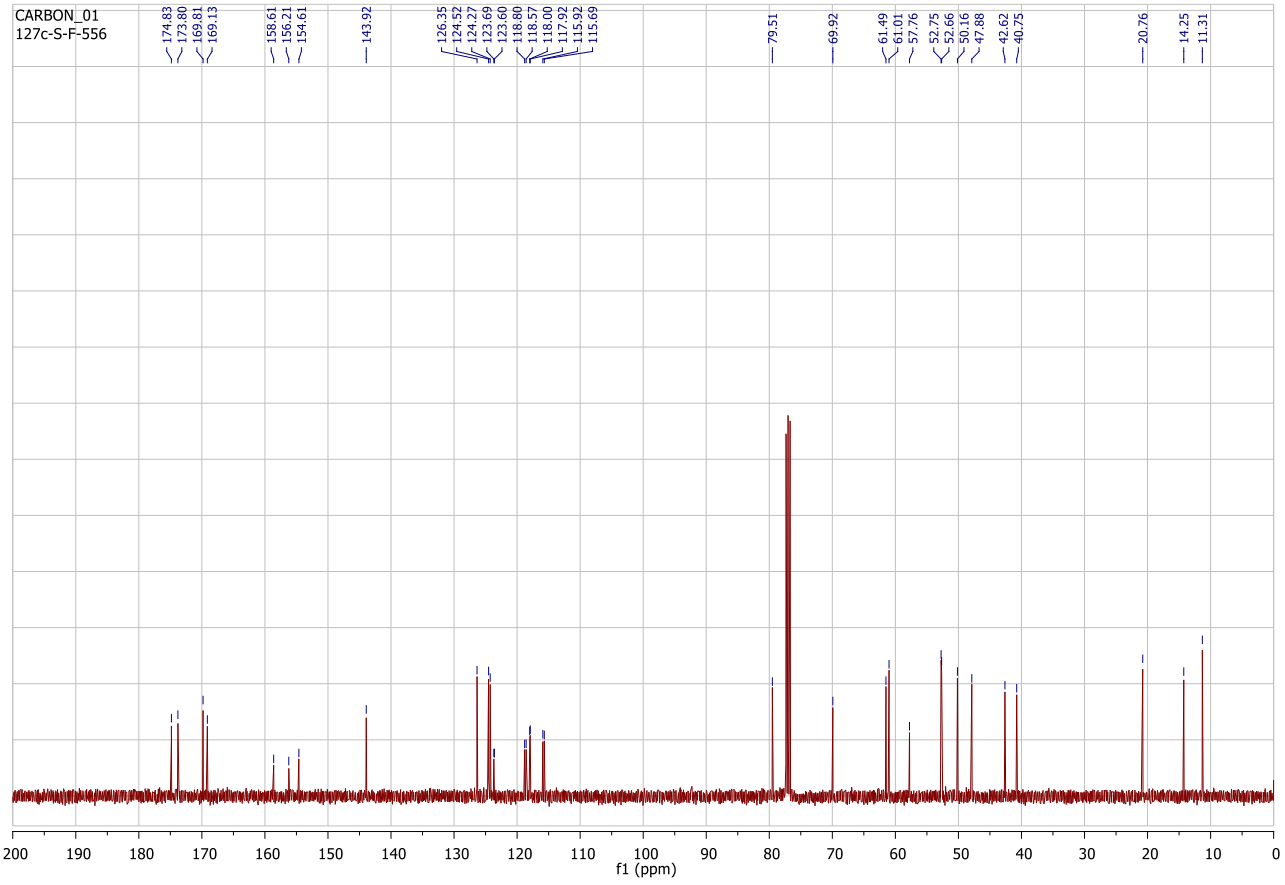

PROTON_01
127c-13-680
Et
N
O
O
CO2Me
Ph
N
O
Br
Br
8g
f1 (ppm)

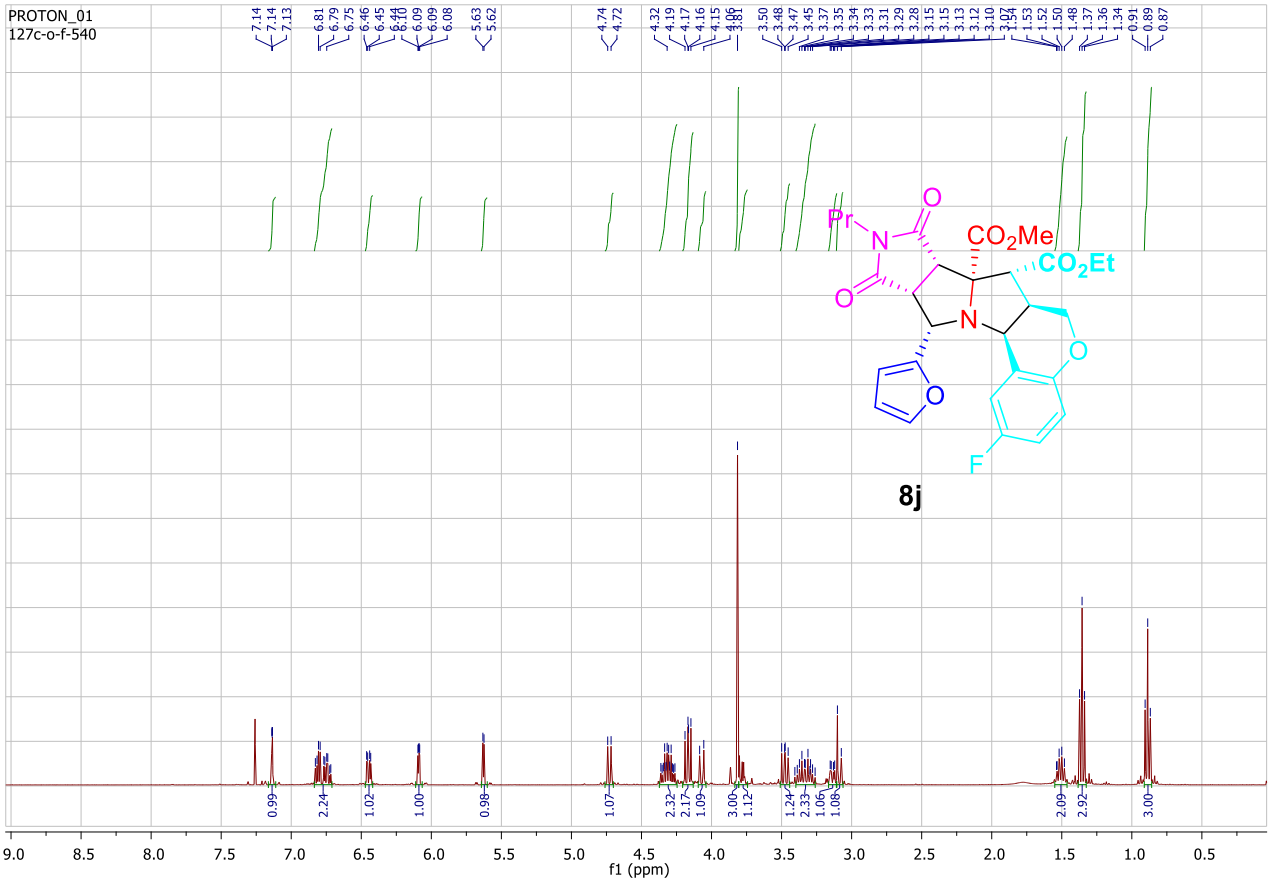

CARBON_01
127c-13-680
f1 (ppm)


PROTON_01
127c-Ph-F-536
Et
N
O
O
CO2Me
CO2Et
N
O
F
8h
f1 (ppm)

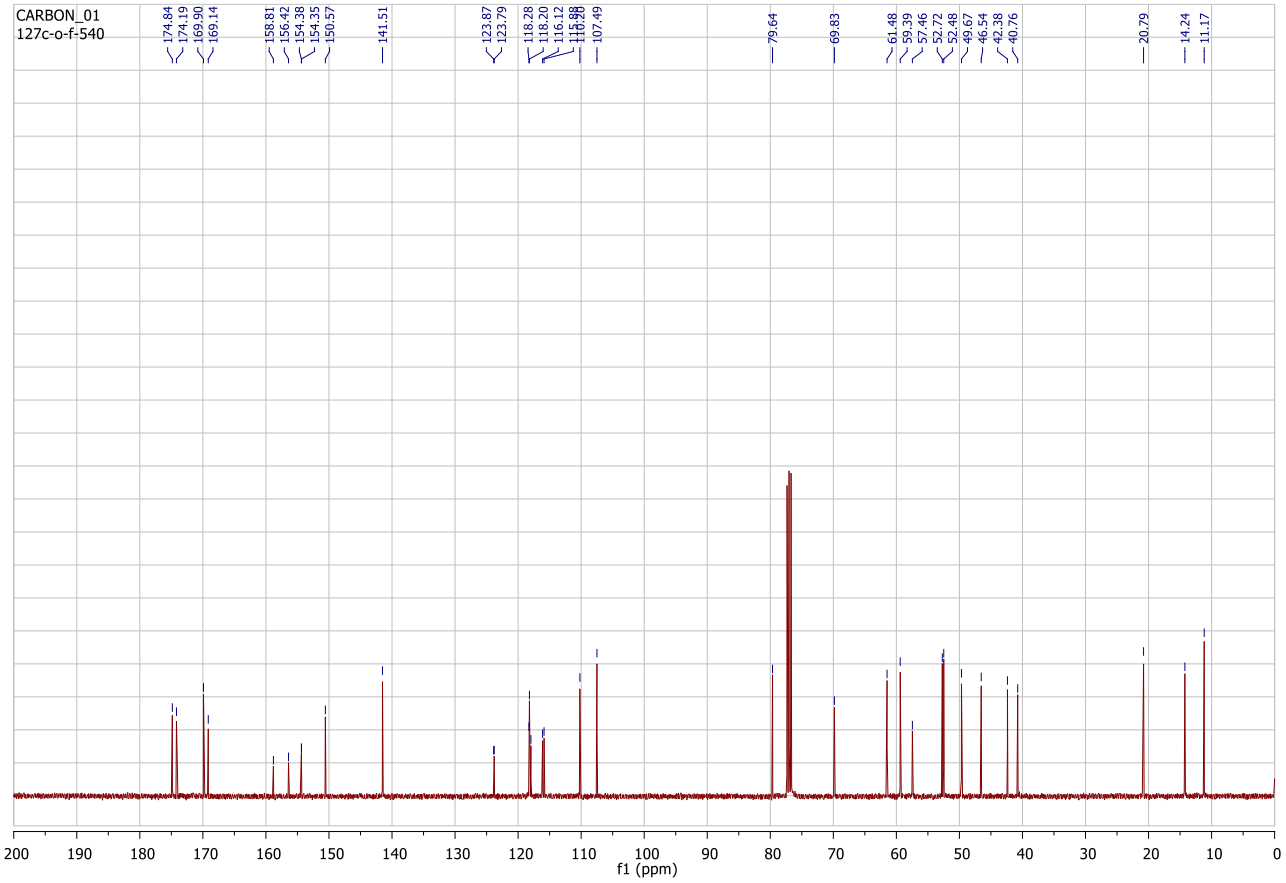

CARBON_01
127c-Ph-F-536
f1 (ppm)


PROTON_01
127c-S-F-556
Pr
N
O
O
CO2Me
CO2Et
N
O
S
F
8i
f1 (ppm)


CARBON_01
127c-S-F-556
f1 (ppm)

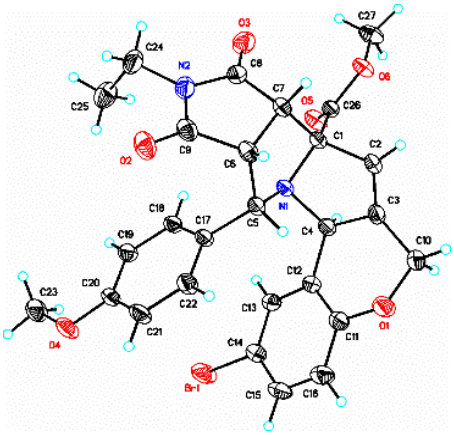

PROTON_01
127c-o-f-540
Pr
N
O
O
CO2Me
CO2Et
N
O
O
F
f1 (ppm)

8j


CARBON_01
127c-o-f-540
f1 (ppm)

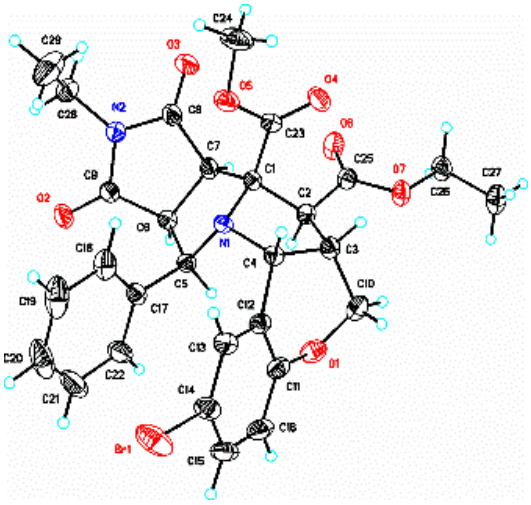
2. X-Ray Crystal Structure Analysis of 6g and 8d

X-ray crystal structure of compound **6g** (Ellipsoid contour probability 30%)



complex	**6g**
Formula	$C_{27}H_{25}BrN_2O_6$
Formula weight	553.40
Crystal system	triclinic
space group	*P*-1
a (Å)	6.1480(3)
b (Å)	9.3553(7)
c (Å)	21.4639(14)
α (º)	97.508(6)
β (º)	91.047(5)
γ (º)	93.474(5)
Volume(Å^3)	1221.27(13)
Z	2
T (K)	293(2)
D_{calcd} (g/m^3)	1.505
F(000)	568
Crystal size/mm3	0.24 × 0.12 × 0.08
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	8.312 to 142.384
Index ranges	-5 ≤ h ≤ 7, -11 ≤ k ≤ 11, -23 ≤ l ≤ 26
Reflections collected	8057
Goodness-of-fit on F^2 1.037	1.037
Largest diff. peak/hole / e Å^{-3}	0.55/-0.51
Data/restraints/parameters	4658/0/329
CCDC NO.	**1852574**

X-ray crystal structure of compound **8d** (Ellipsoid contour probability 30%)



complex	**8d**
Formula	$C_{29}H_{29}BrN_2O_7$
Formula weight	597.45
Crystal system	monoclinic
space group	$P2_1/n$
a (Å)	14.6471(3)
b (Å)	9.8270(2)
c (Å)	19.3981(3)
α (º)	90
β (º)	104.564(2)
γ (º)	90
Volume(Å^3)	2702.39(9)
Z	4
T (K)	293(2)
D_{calcd} (g/m^3)	1.468
F(000)	1232
Crystal size/mm^3	0.36 × 0.24 × 0.22
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	6.802 to 142.666
Index ranges	-18 ≤ h ≤ 17, -9 ≤ k ≤ 11, -17 ≤ l ≤ 23
Reflections collected	11287
Goodness-of-fit on F^2 1.037	1.032
Largest diff. peak/hole / e Å^{-3}	0.63/-0.61
Data/restraints/parameters	5160/0/356
CCDC NO.	**1852575**