Supporting Information for

Copper(0)/Selectfluor System-promoted Oxidative Carbon-Carbon Bond Cleavage/Annulation of *o*-Aryl Chalcones: An Unexpected Synthesis of 9,10-Phenanthraquinone Derivatives

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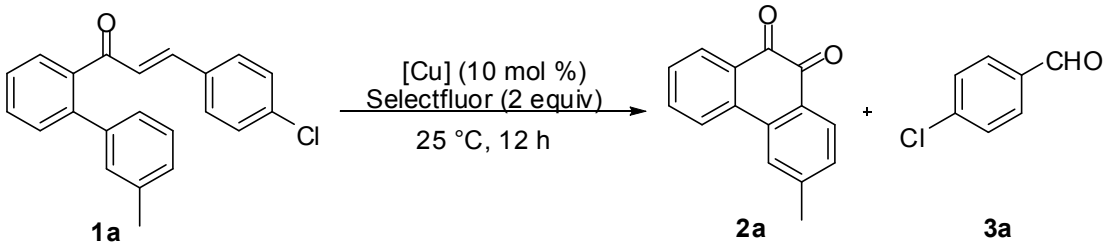
Contents

1. Optimization of Reaction Conditions

Table *S1*. Screening with different catalysts [a]

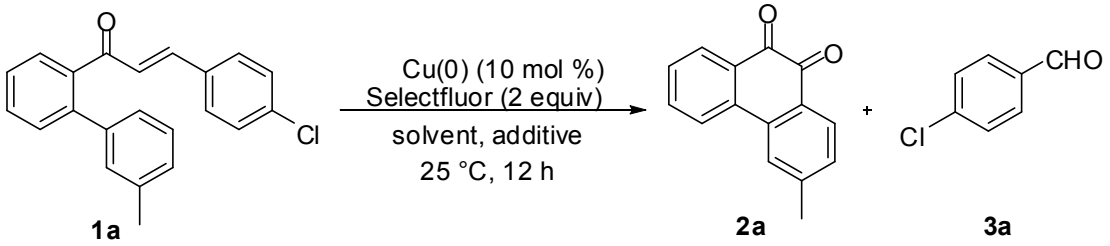


1a **2a** **3a**

entry	[Cu]	solvent	yield of **2a** (%)[b]
1	Cu(0)[c]	CH_3CN	70
2	Cu(0)	CH_3CN	75
3	**Cu(0)**	**CH_3CN/H_2O = 50:1 (V/V)**	**80**
4	Cu(0)[d]	CH_3CN/H_2O = 50:1 (V/V)	69
5	Cu(0)[e]	CH_3CN/H_2O = 50:1 (V/V)	55
6	--	CH_3CN/H_2O = 50:1 (V/V)	0
7	CuBr	CH_3CN/H_2O = 50:1 (V/V)	64
8	CuI	CH_3CN/H_2O = 50:1 (V/V)	57
9	CuCl	CH_3CN/H_2O = 50:1 (V/V)	55
10	$CuCl_2$	CH_3CN/H_2O = 50:1 (V/V)	60
11	$Cu(OAc)_2$	CH_3CN/H_2O = 50:1 (V/V)	50
12	$Cu(NO_3)_2$	CH_3CN/H_2O = 50:1 (V/V)	52

[a] Reactions carried out with **1a** (0.3 mmol), copper catalyst (10 mol % based on **1a**), Selectfluor (2 equiv), solvent (3 mL), 25 ºC for 12 h unless otherwise noted. [b] Isolated yields.[c] Using 5 mol % of Cu(0) powder. [d]Using 20 mol % of Cu(0) powder.[e] The reaction temperature is 50 ºC.

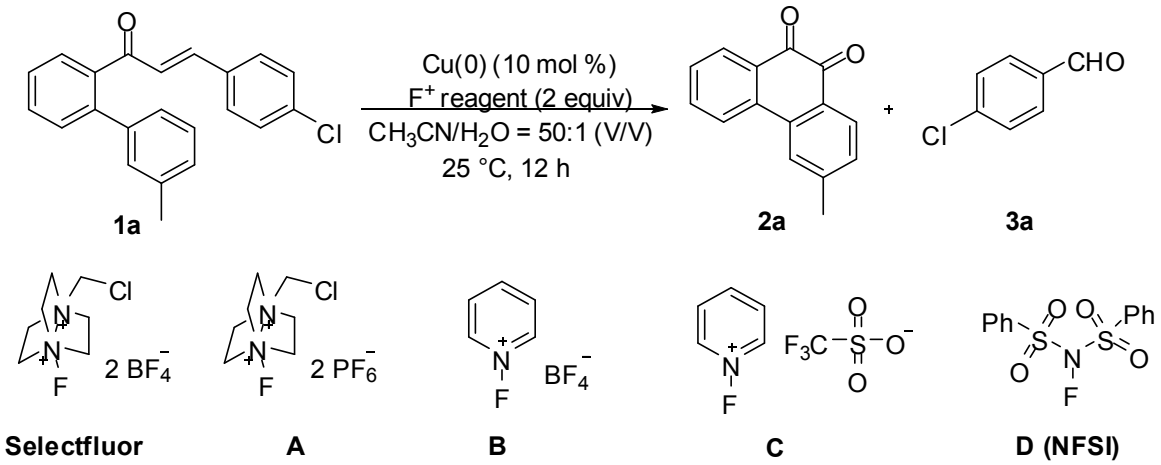
Table *S2*. Screening with different solvents, additives and reaction times [a]



entry	solvent	additive	yield of **2a** (%,)[b]
1	CH_3CN	--	75
2	CH_3CN	K_2CO_3	68
3	CH_3CN	$NaHCO_3$	64
4	CH_3CN	K_2CO_3	67
5	CH_3CN	K_3PO_4	57
6	**CH_3CN/H_2O = 50:1 (V/V)**	**--**	**80**
7	CH_3CN/H_2O = 25:1 (V/V)	--	65
8	CH_3CN/H_2O = 100:1 (V/V)	--	77
9	CH_3CN/H_2O = 200:1 (V/V)	--	75
10	CH_3CN/H_2O = 400:1 (V/V)	--	73
11	Acetone/H_2O = 50:1 (V/V)	--	0
12	EtOAc/H_2O = 50:1 (V/V)	--	0
13	MeOH/H_2O = 50:1 (V/V)	--	0
14	EtOH/H_2O = 50:1 (V/V)	--	0
15	DMF/H_2O = 50:1 (V/V)	--	0
16	toluene/H_2O = 50:1 (V/V)	--	0
17	CH_2Cl_2/H_2O = 50:1 (V/V)	--	0
18	1,4-dioxane/H_2O = 50:1 (V/V)	--	0
19	CH_3CN/H_2O = 50:1 (V/V)	--	70[c]
20	CH_3CN/H_2O = 50:1 (V/V)	--	65[d]
21	CH_3CN/H_2O = 50:1 (V/V)	--	30[e]

[a] Reactions carried out with **1a** (0.3 mmol), copper powder (10 mol % based on **1a**), Selectfluor (2 equiv), solvent (3 mL), 25 ºC for 12 h unless otherwise noted.[b]Isolated yields. [c]The reaction time is 6 h. [d]The reaction time is 4 h. [e]The reaction time is 2 h.

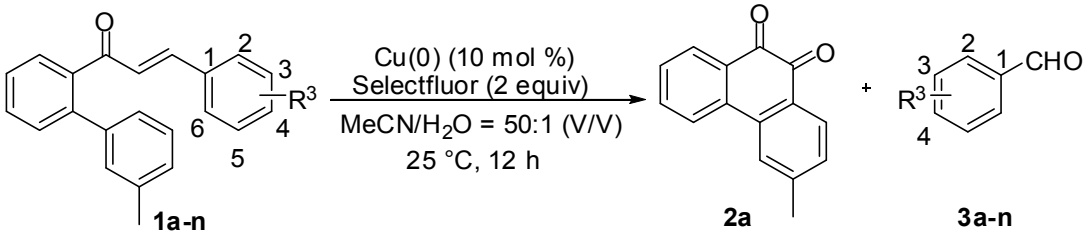
Table *S3*. Screening with different oxidants [a]



entry	F^+ reagent	yield of **2a** (%)[b]
1	**Selectfluor**	**80**
2	Selectfluor	40[c]
3	Selectfluor	62[d]
4	**A**	40
5	**B**	trace
6	**C**	10
7	**D**	50

[a] Reactions carried out with **1a** (0.3 mmol), copper powder (10 mol % based on **1a**), F^+ reagent (2 equiv), CH_3CN/H_2O mixture (50:1 (V/V), 3 mL), 25 ºC for 12 h unless otherwise noted. [b]Isolated yields. [c]The use of 1.0 equivalent of Selectfluor. [d] The use of 3.0 equivalents of Selectfluor.

Table *S4*. Investigation of the effect of different benzylidene moieties in **1a-n** on the formation of **2a**[a]



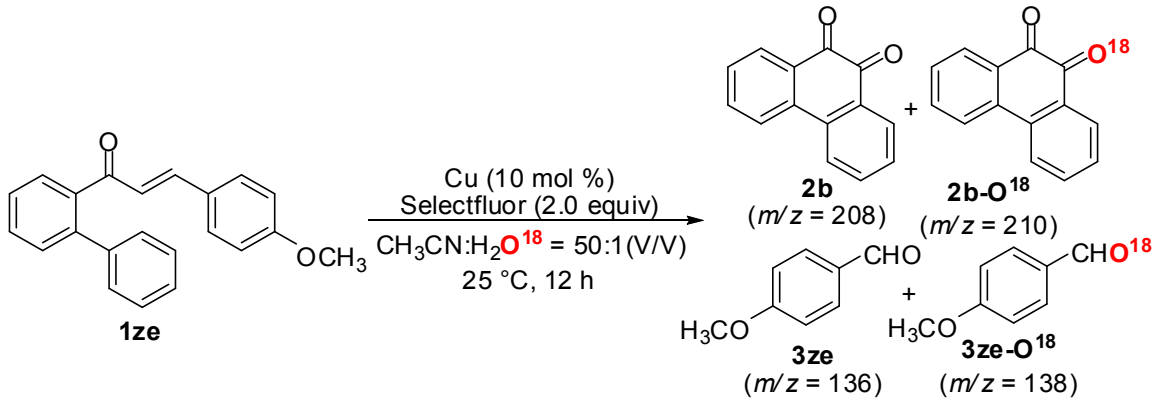
entry	R^3 (**1a-n**)	aldehyde (**3a-n**)	yield of **2a** (%)[b]
1	4-Cl (**1a**)	**3a**	80
2	2-Cl (**1a-1**)	**3a-1**	66
3	3-Cl (**1a-2**)	**3a-2**	60
4	4-Br (**1a-3**)	**3a-3**	61
5	3,5-dichloro (**1a-4**)	**3a-4**	65
6	2,6-dichloro (**1a-5**)	**3a-5**	63
7	4-NO_2 (**1a-6**)	**3a-6**	45
8	4-CH_3 (**1a-7**)	**3a-7**	32
9	H (**1a-8**)	**3a-8**	60

[a] Reactions carried out with **1a-n** (0.3 mmol), copper catalyst (10 mol % based on **1a-n**), Selectfluor (2 equiv), CH_3CN/H_2O = 50:1 (V/V) (3 mL), 25 ºC for 12 h unless otherwise noted.

[b] Isolated yields.

2. Mechanistic Experiments

2.1 Reaction of 1ze in CH_3CN-H_2O^{18} (50:1, V/V)



MS-analytical Results:

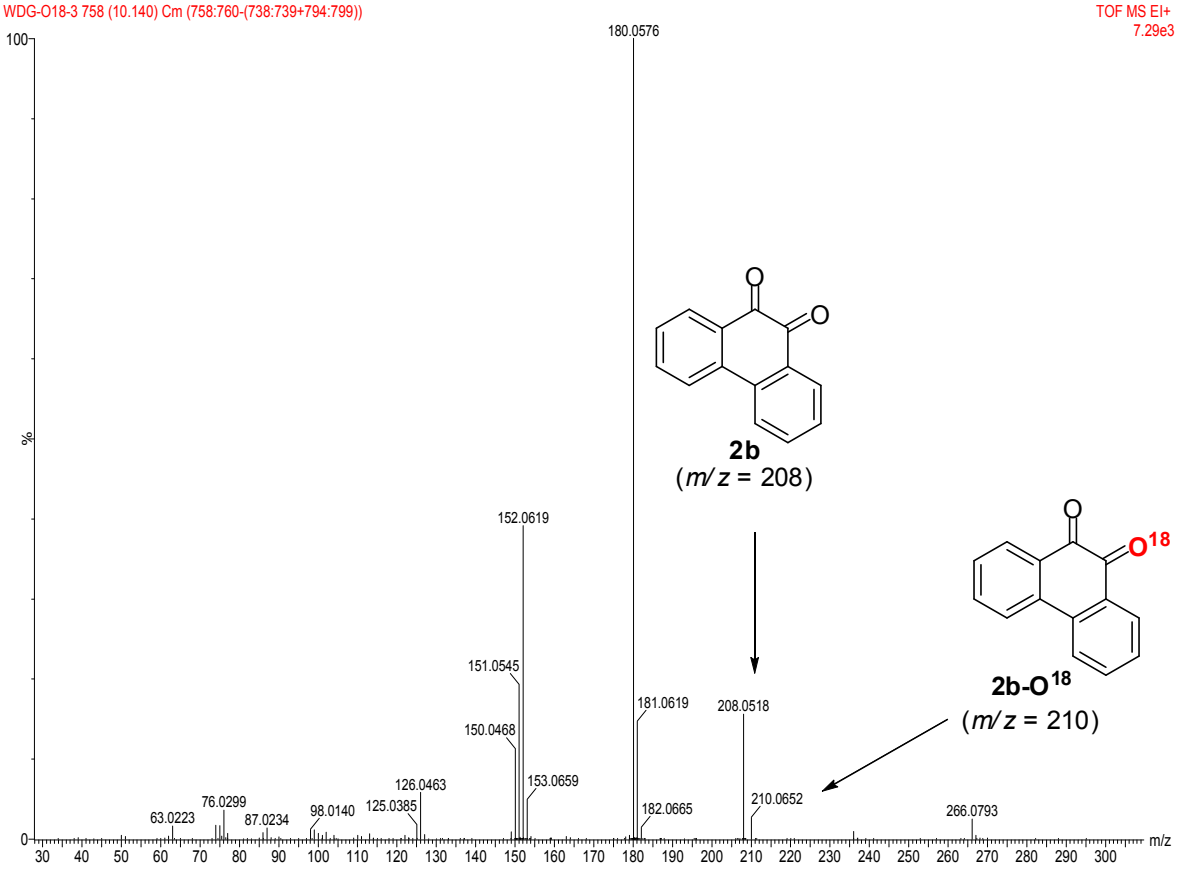


Figure *S1*. The MS-spectrum of **2b**/**2b-O^{18}**

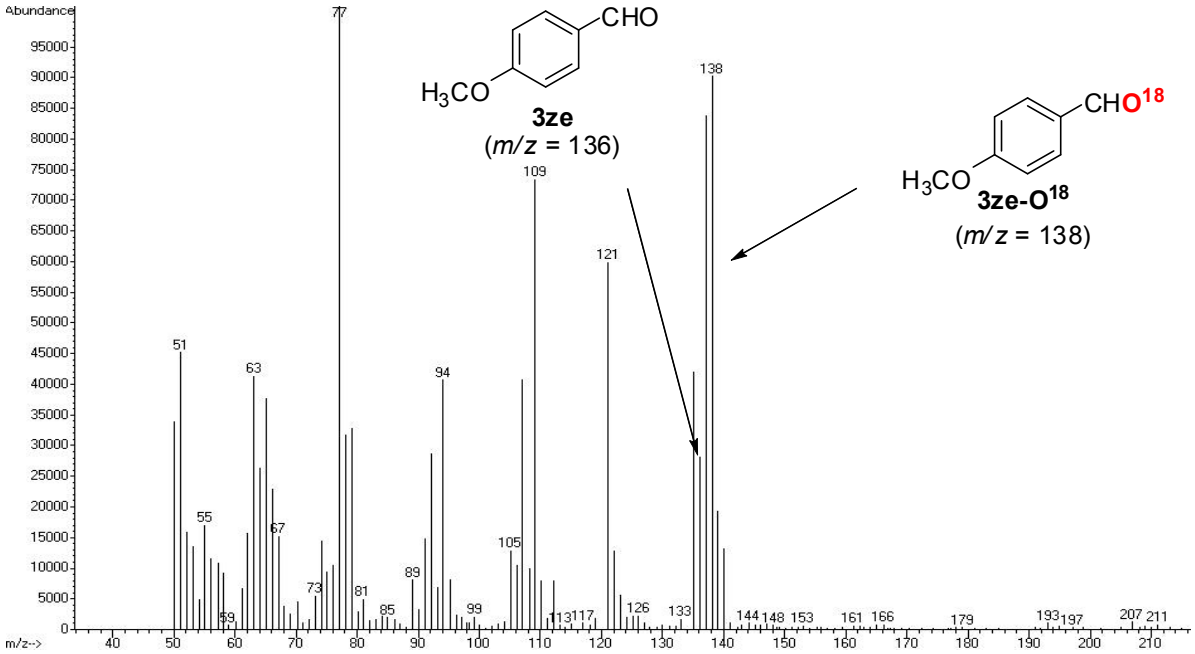


Figure *S2*. The MS-spectrum of **3ze**/**3ze-O^{18}**

2.2 Studies on the Intermolecular Kinetic Isotope Effects (KIE) Based on Substrate 1u and 1u-d_5.

^{1}H NMR Analytical Results:

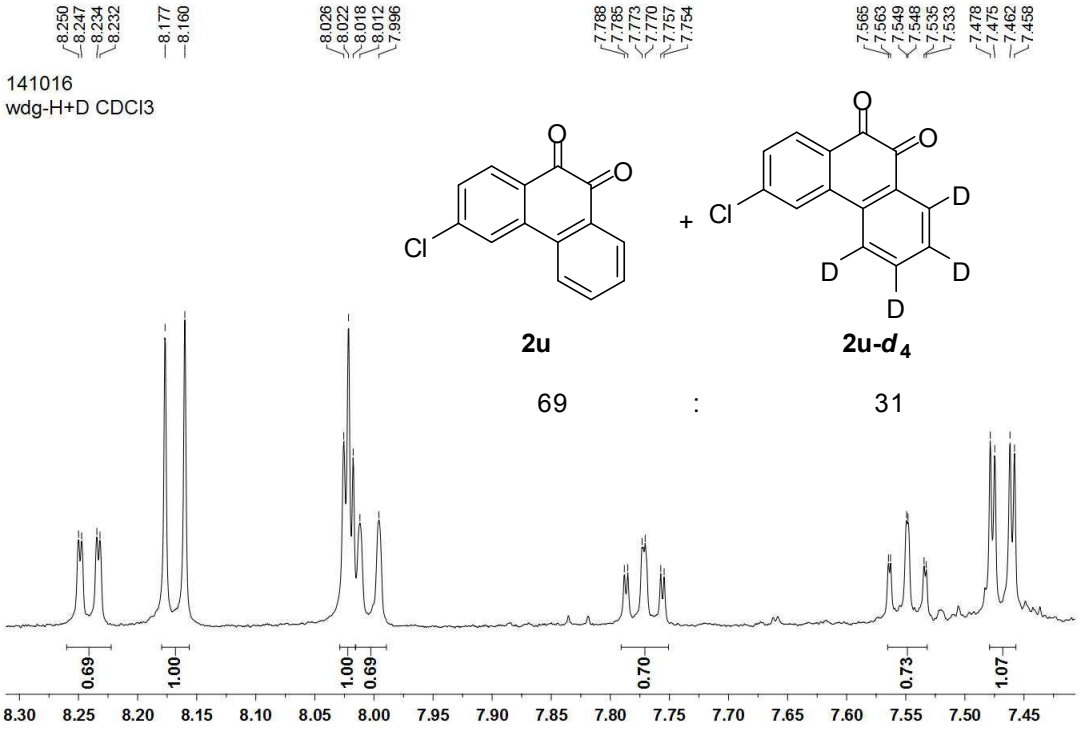
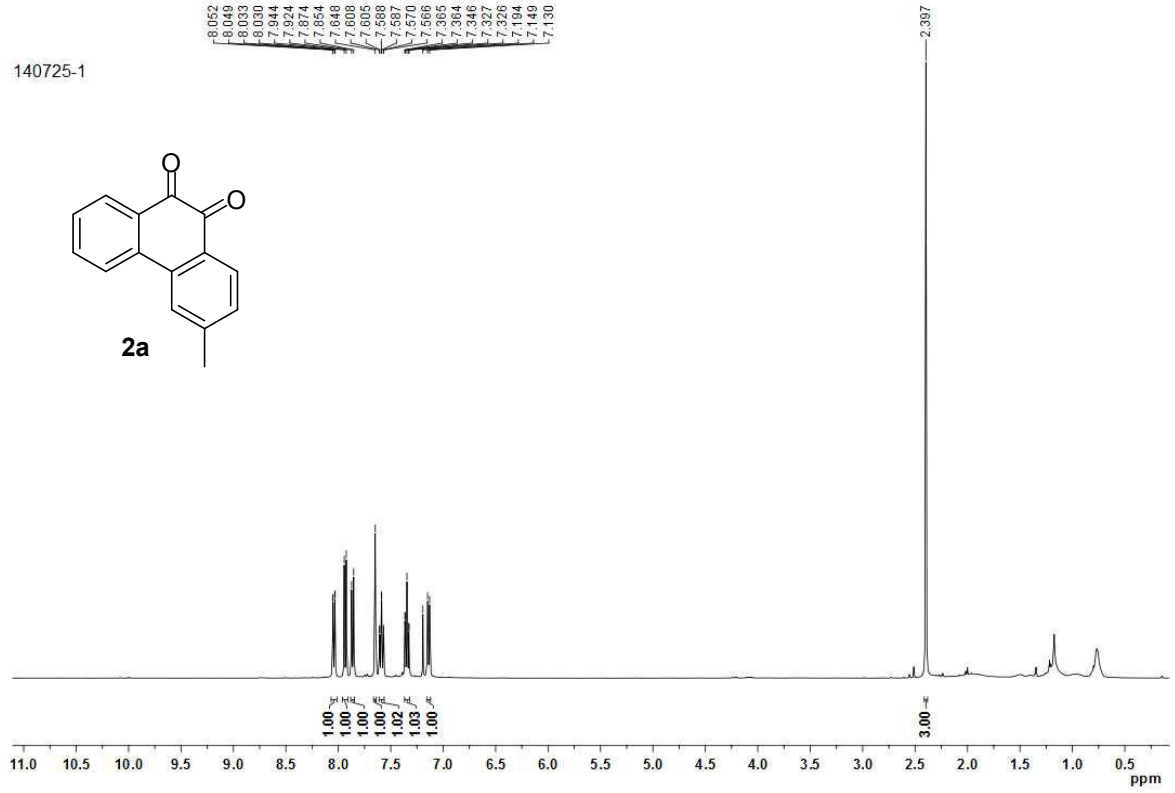
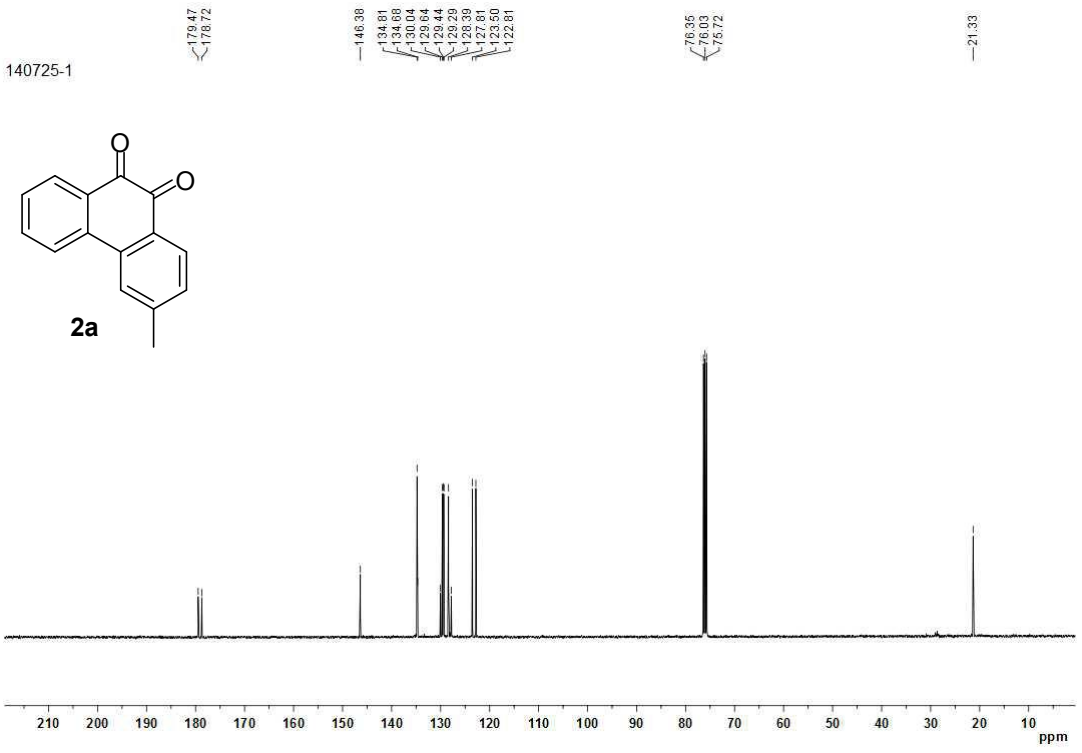
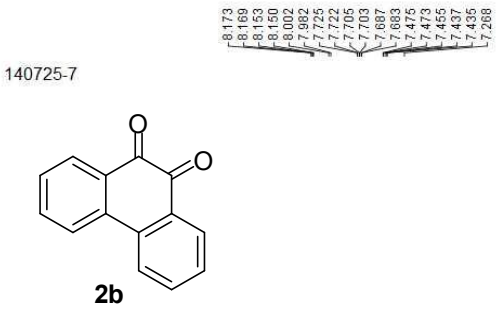


Figure *S3*. ^{1}H NMR spectrum of the mixture of **2u** and **2u-d_4**

3. ^{1}H and ^{13}C NMR spectra of 2a-2zd



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7.722
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7.703
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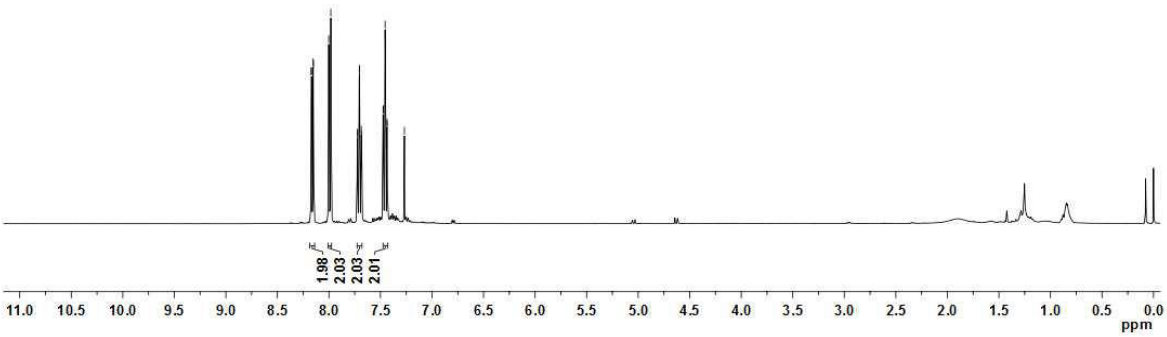
7.475
7.473
7.455
7.437
7.435
7.268
140725-7
O
O
2b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2.03
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10.5
10.0
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6.5
6.0
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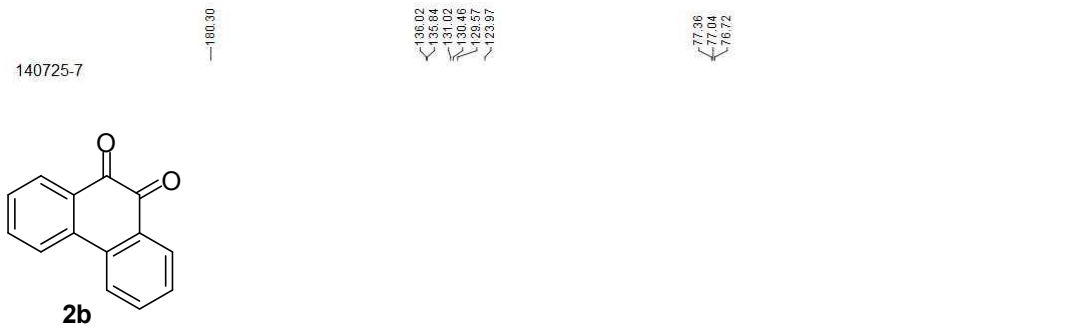
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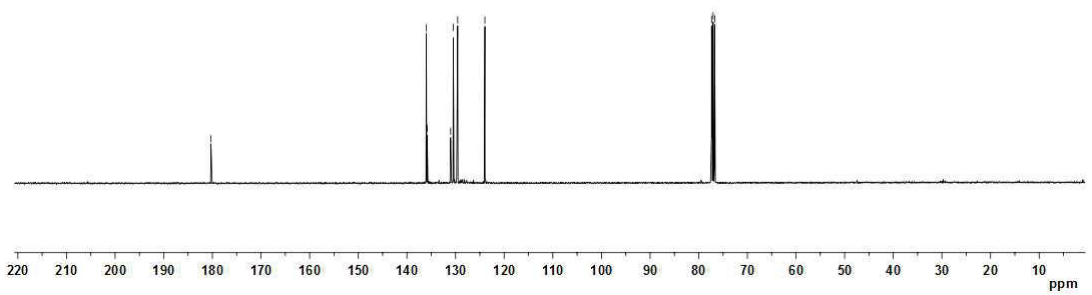
76.72
140725-7
O
O
2b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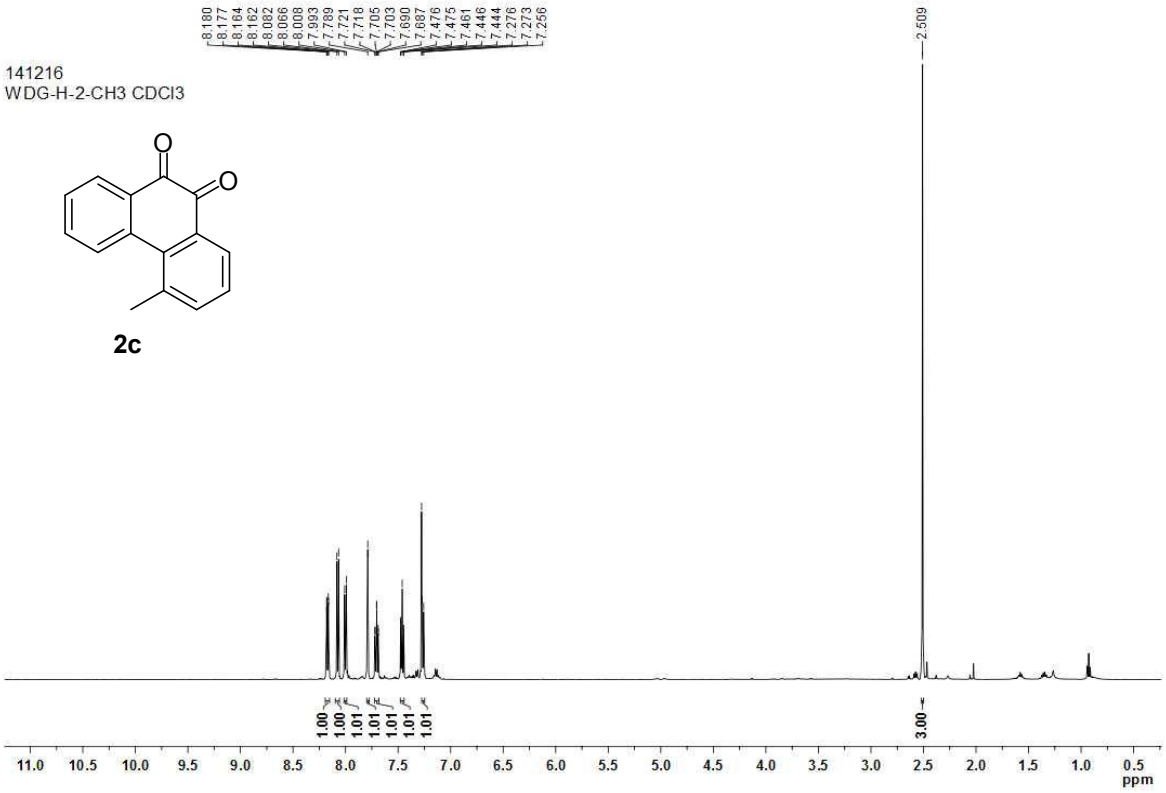
10
ppm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8.082
8.066
8.008
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7.721
7.718
7.705
7.703
7.690
7.687
7.476
7.475
7.461
7.446
7.444
7.276
7.273
7.256
2.509
141216
WDG-H-2-CH3 CDCl3
O
O
1.00
1.00
1.01
1.01
1.01
1.01
1.01
3.00
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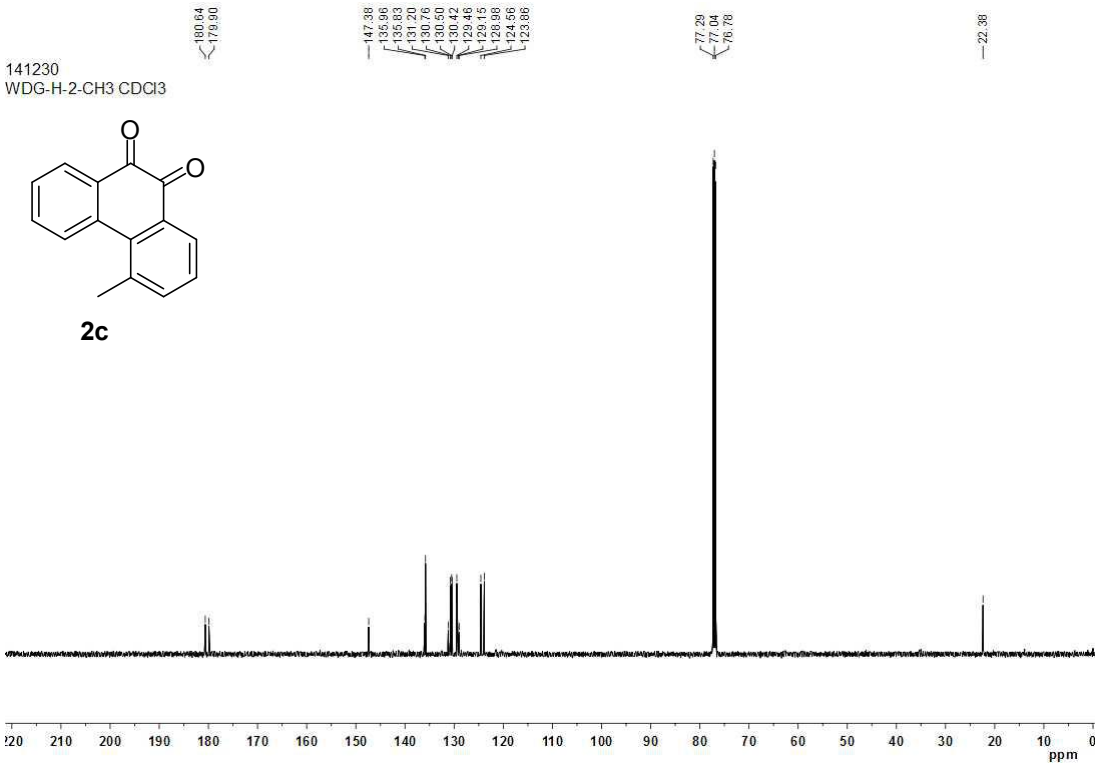
ppm

2c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128.98
124.56
123.86
77.29
77.04
76.78
22.38
141230
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O
O
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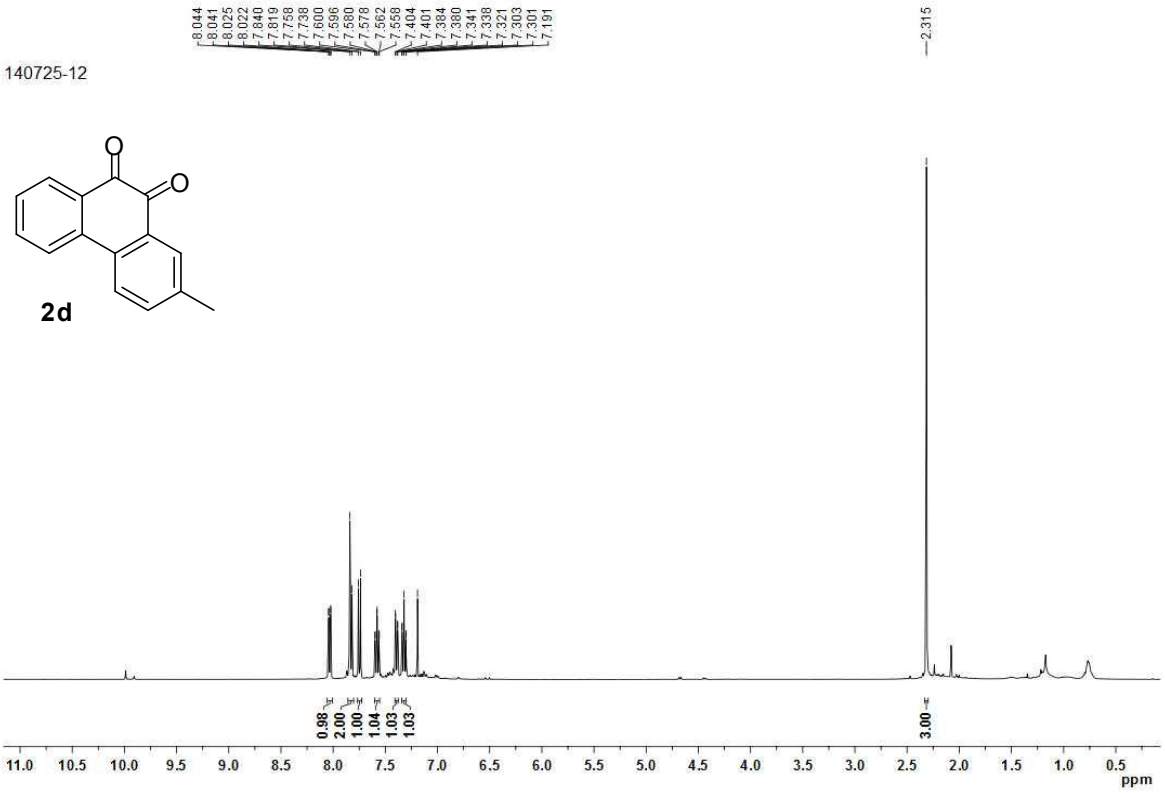
ppm

2c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7.562
7.558
7.404
7.401
7.384
7.380
7.341
7.338
7.321
7.303
7.301
7.191
2.315
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2.00
1.00
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1.03
3.00
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10.0
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9.0
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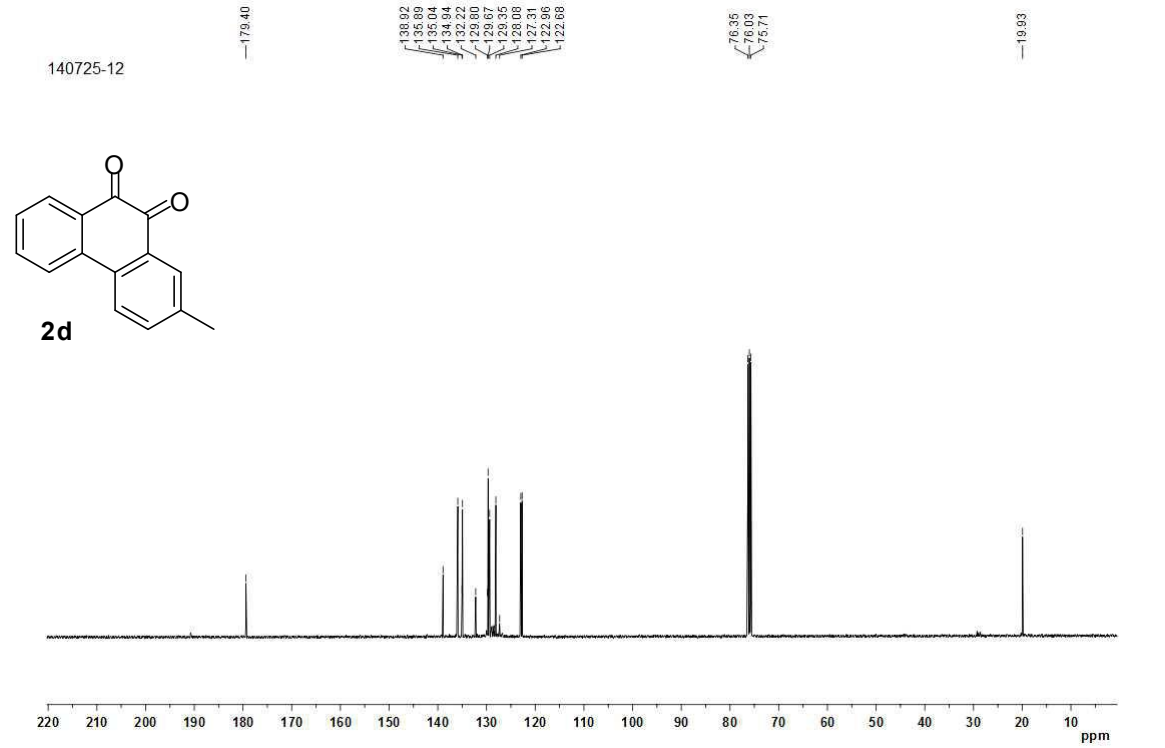
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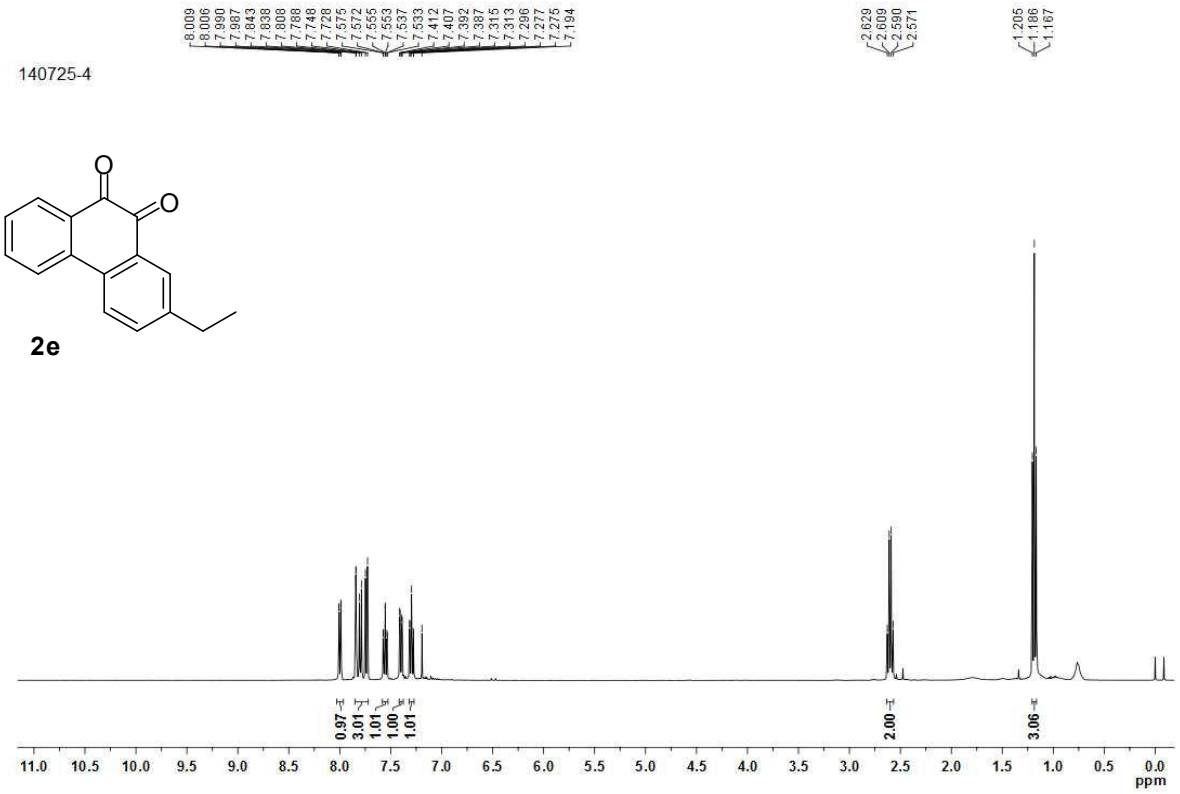
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7.387
7.315
7.313
7.296
7.277
7.275
7.194
2.629
2.609
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1.167
140725-4
O
O
2e
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3.01
1.01
1.00
1.01
2.00
3.06
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 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
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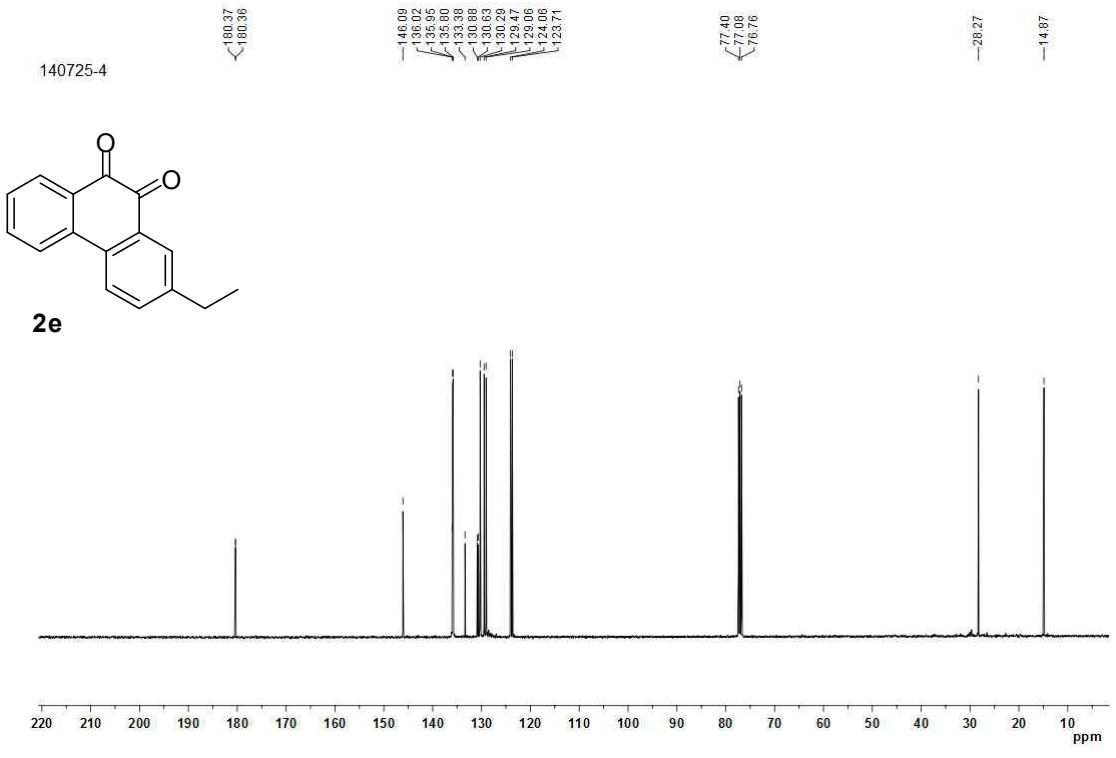
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136.02
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123.71
77.40
77.08
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140725-4
O
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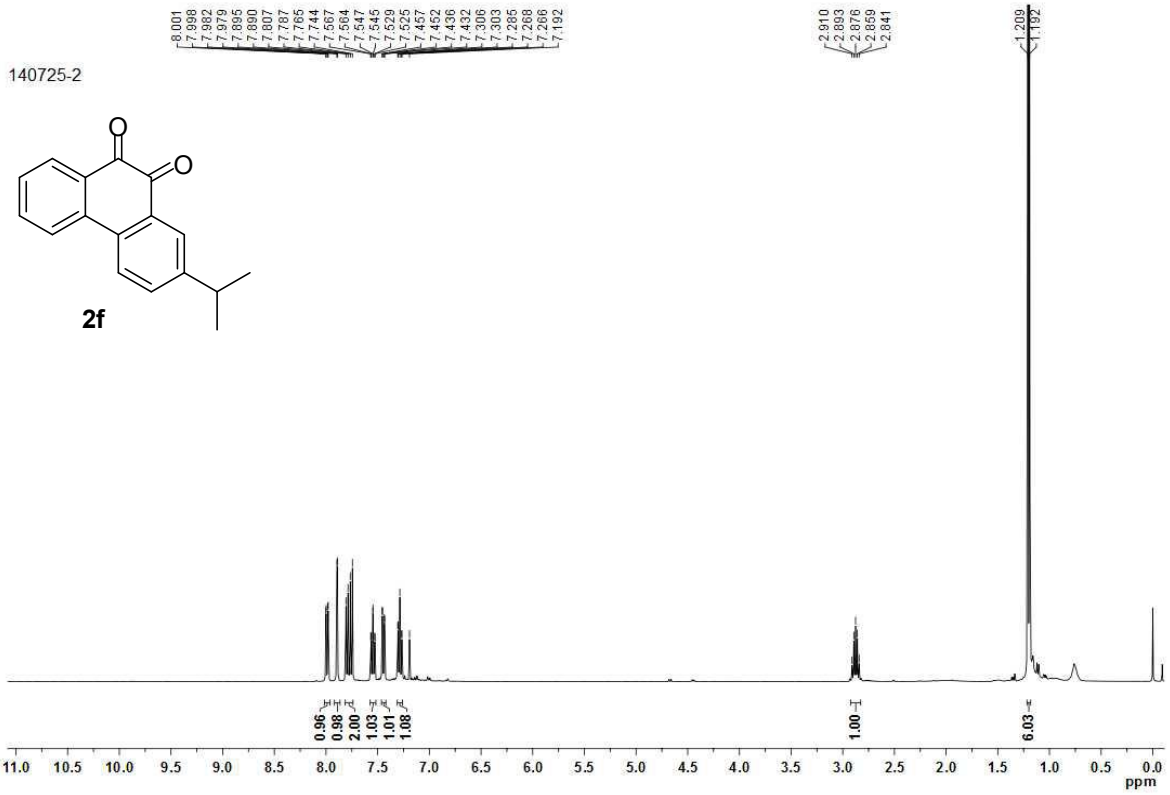
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7.306
7.303
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7.268
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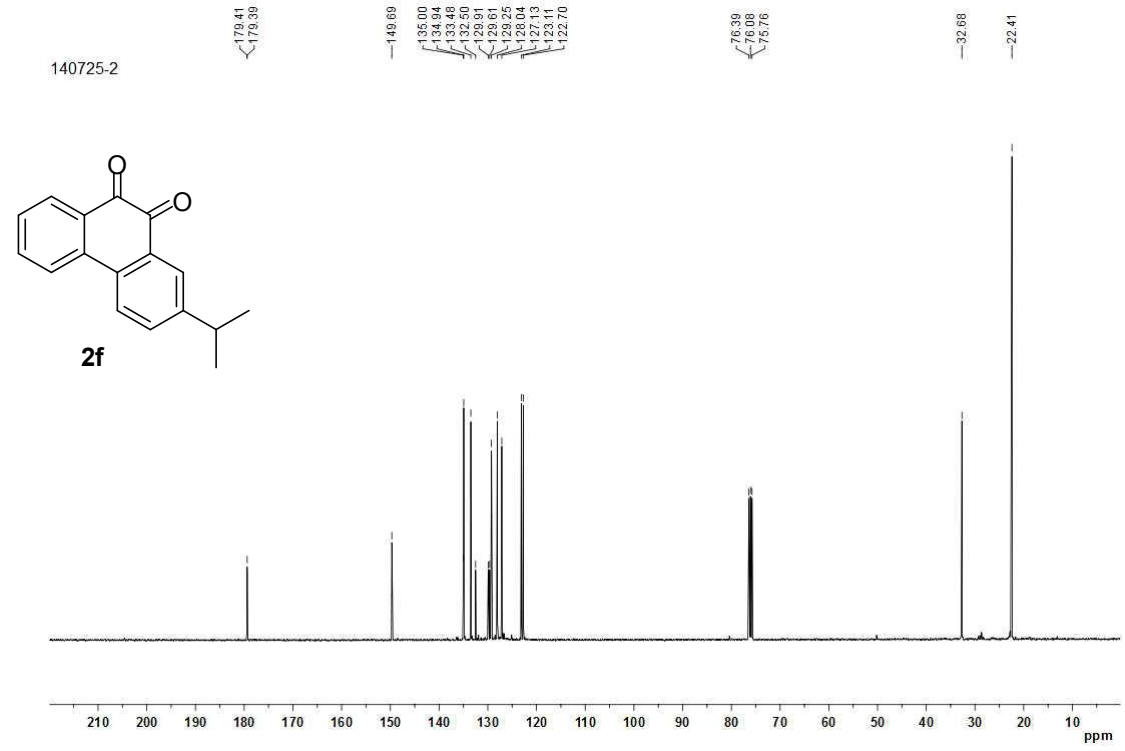
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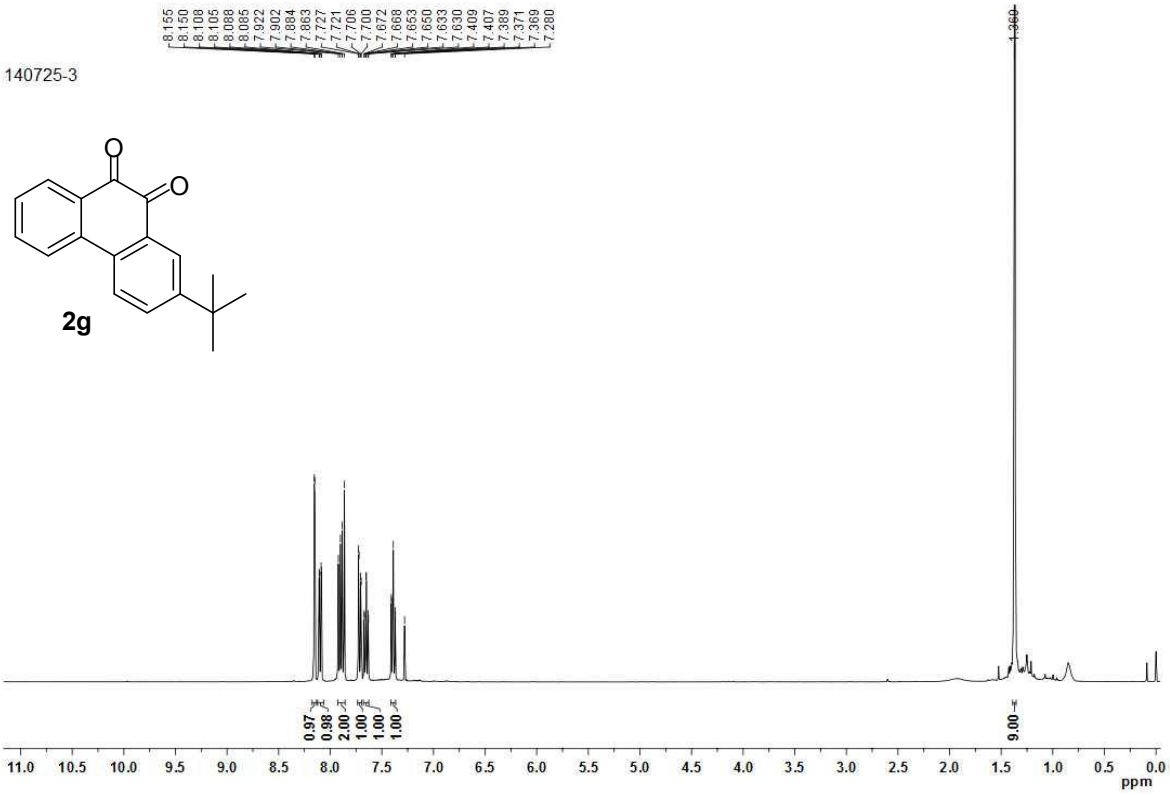
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7.668
7.653
7.650
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7.630
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7.407
7.389
7.371
7.369
7.280
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0.97
0.98
2.00
1.00
1.00
1.00
9.00
11.0 10.5 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
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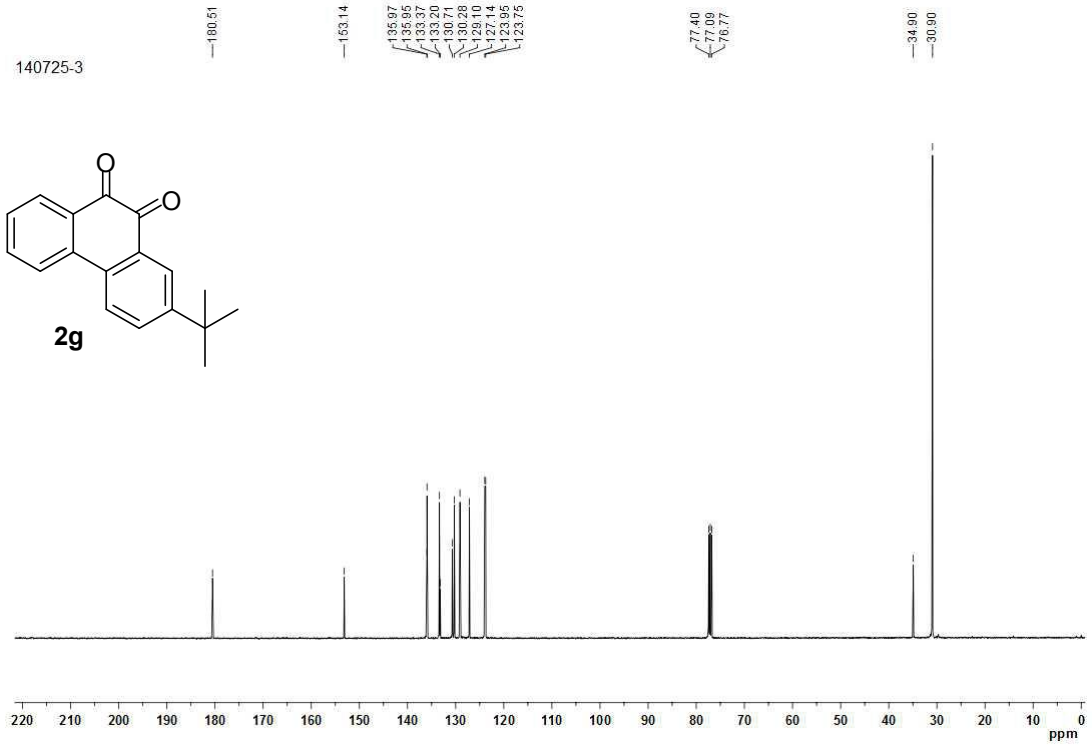
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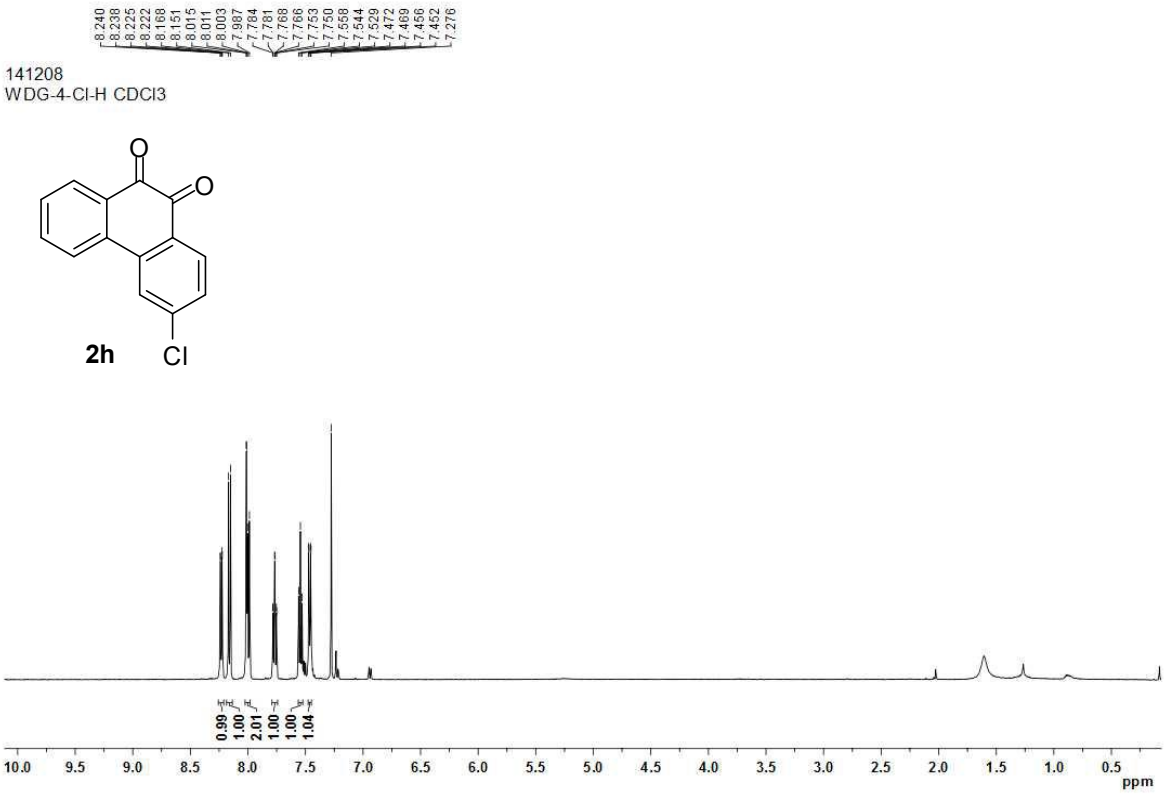
ppm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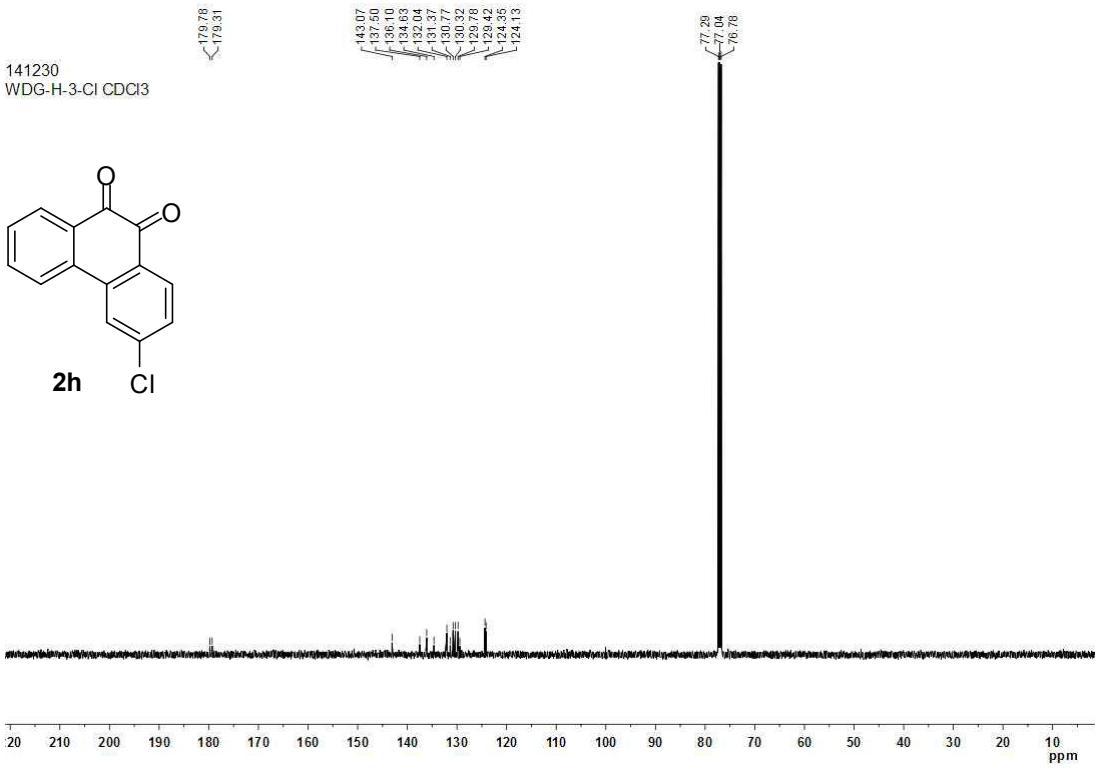
7.529
7.472
7.469
7.456
7.452
7.276
141208
WDG-4-Cl-H CDCl3
O
O
2h
Cl
0.99
1.00
2.01
1.00
1.00
1.04
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm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131.37
130.77
130.32
129.78
129.42
124.35
124.13
77.29
77.04
76.78
141230
WDG-H-3-Cl CDCl3
O
O
2h
Cl
220
210
200
190
180
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150
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110
100
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70
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30
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10
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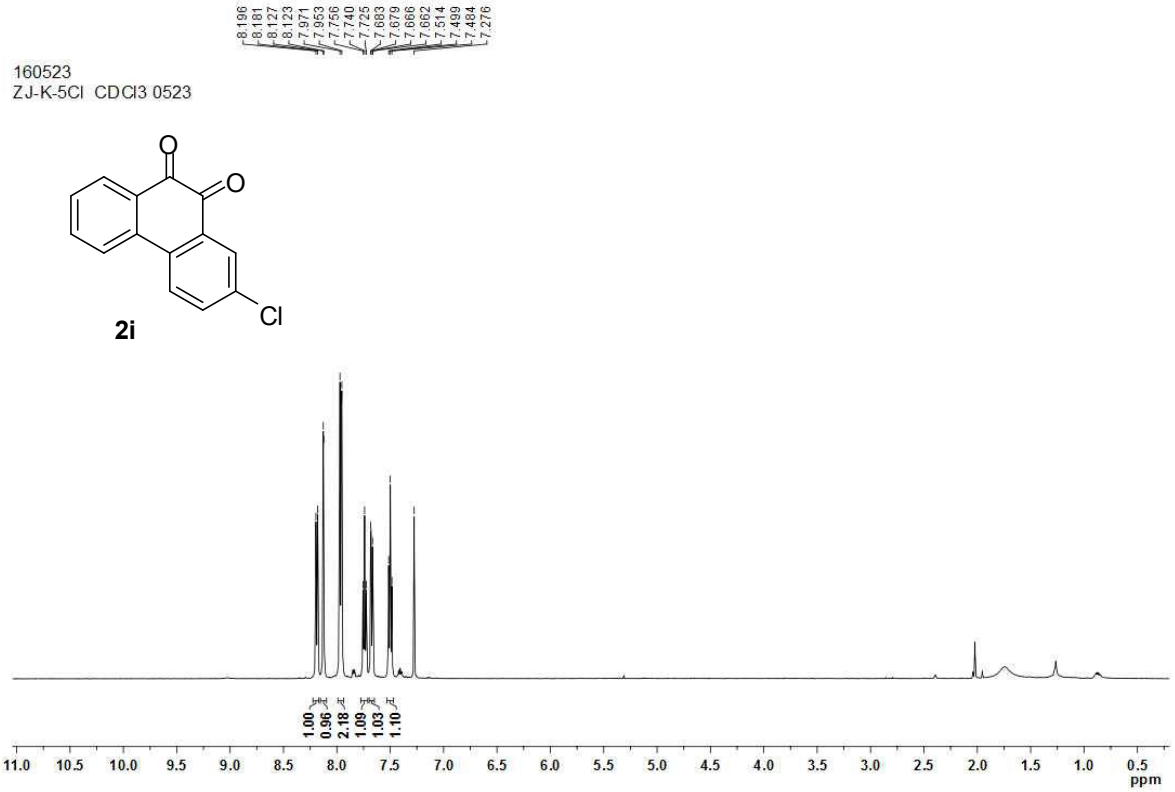
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7.662
7.514
7.499
7.484
7.276
160523
ZJ-K-5Cl CDCl3 0523
O
O
Cl
2i
1.00
0.96
2.18
1.09
1.03
1.10
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
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4.0
3.5
3.0
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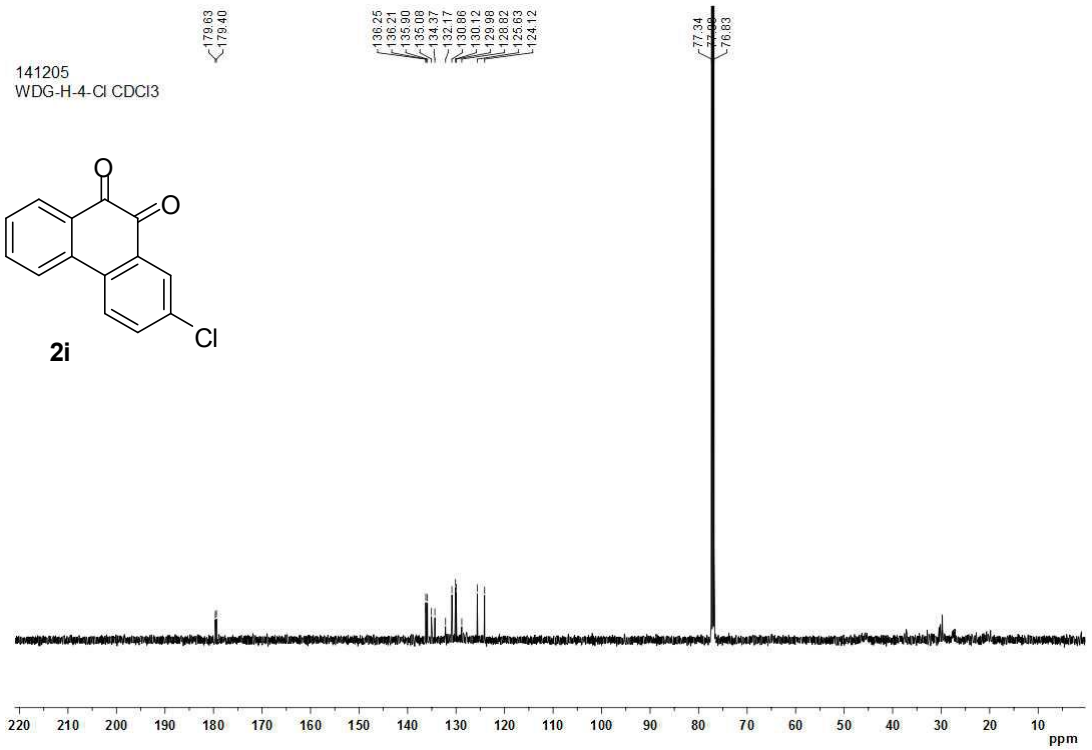
2.0
1.5
1.0
0.5
ppm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128.82
125.63
124.12
77.34
77.00
76.83
141205
WDG-H-4-Cl CDCl3
O
O
Cl
2i
220
210
200
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150
140
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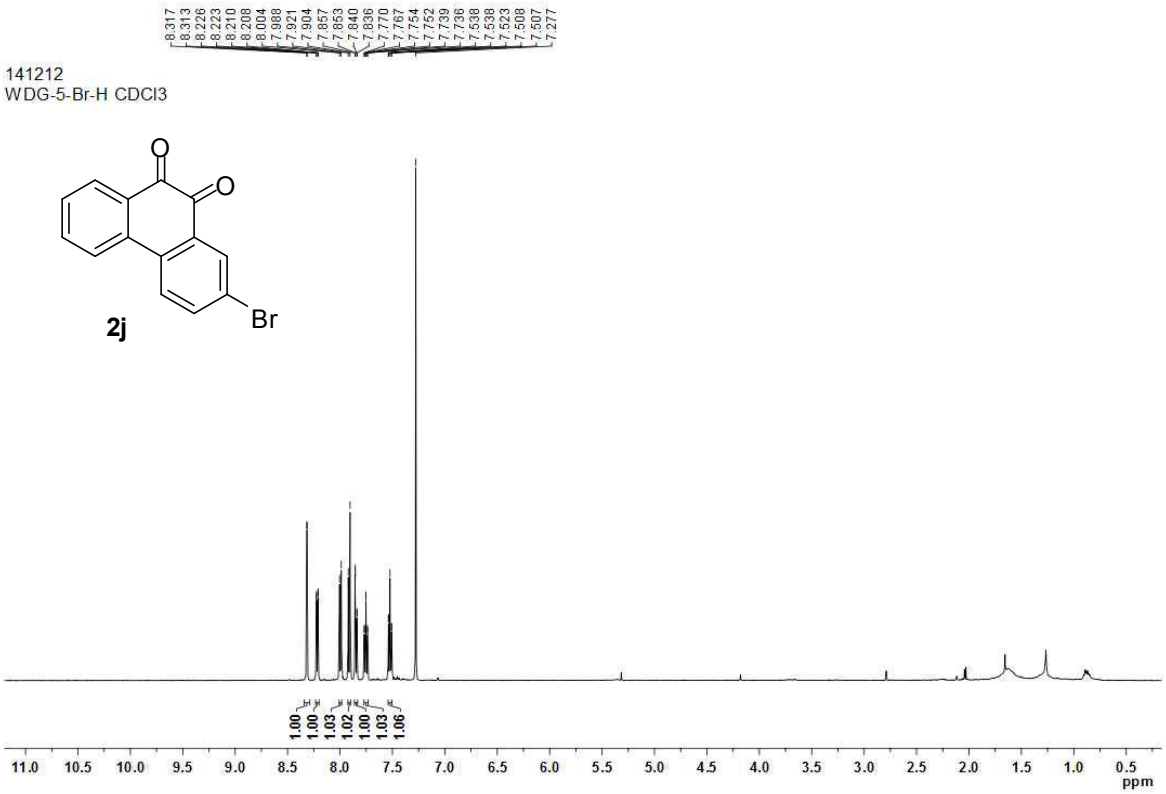
30
20
10
ppm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7.840
7.836
7.770
7.767
7.754
7.752
7.739
7.736
7.538
7.523
7.508
7.507
7.277
141212
WDG-5-Br-H CDCl3
O
O
Br
2j
1.00
1.00
1.03
1.02
1.00
1.03
1.06
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
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4.5
4.0
3.5
3.0
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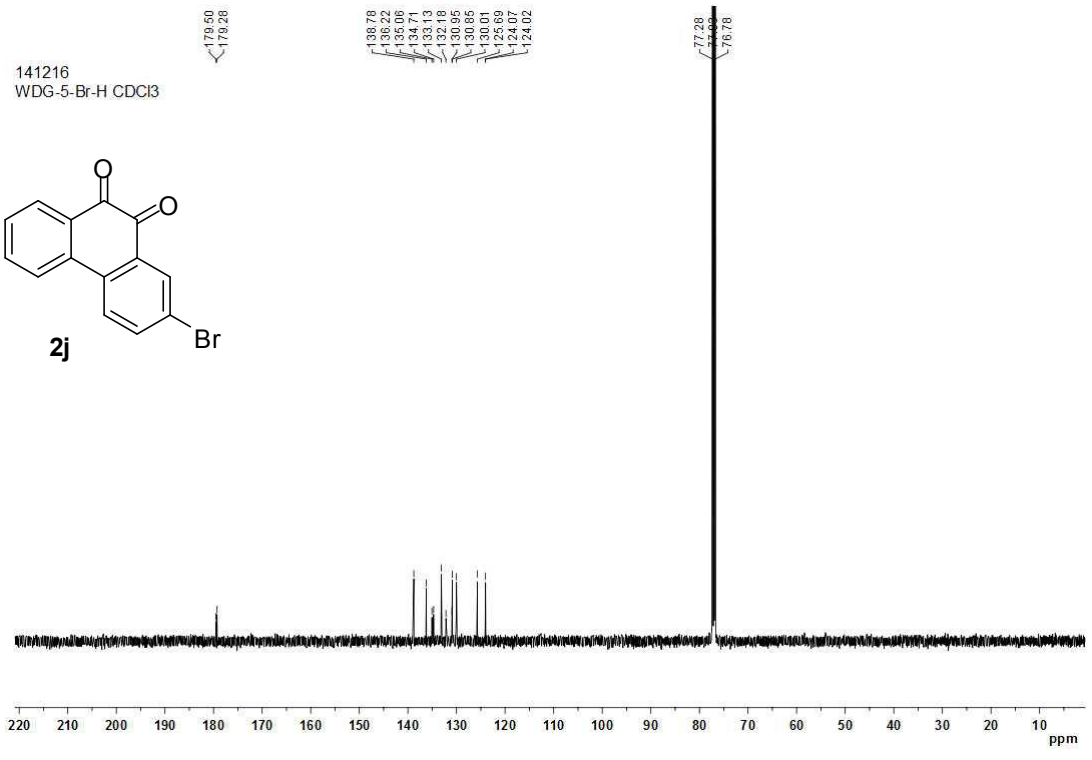
2.0
1.5
1.0
0.5
ppm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132.18
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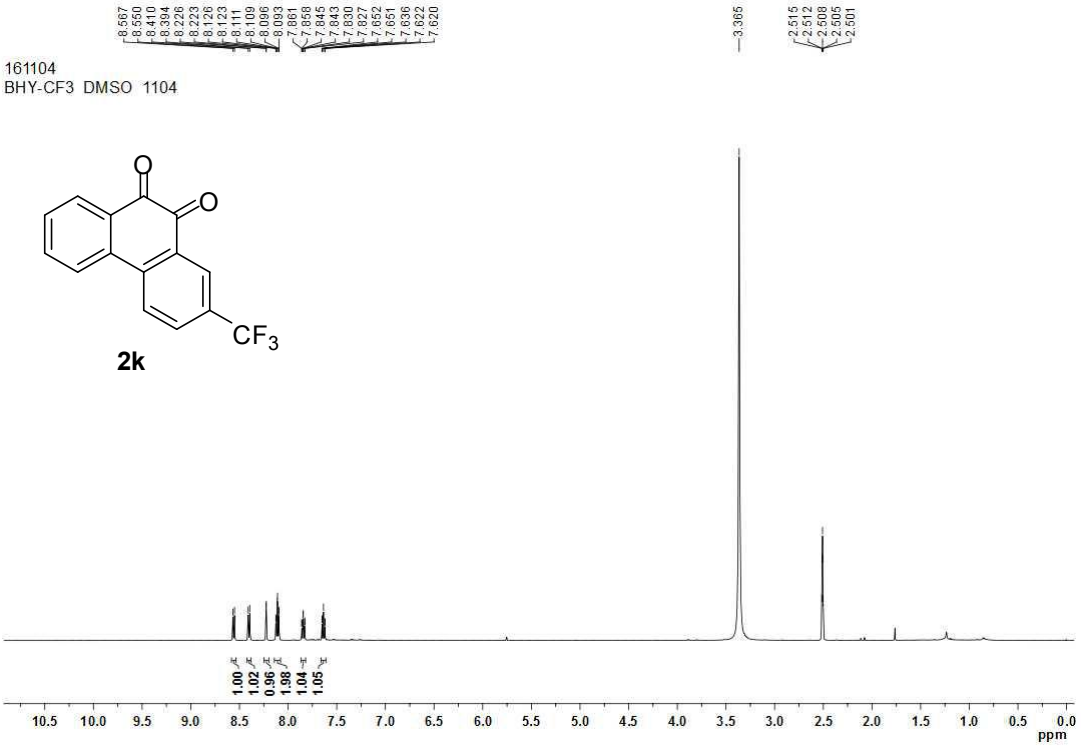
130.01
125.69
124.07
124.02
77.28
77.03
76.78
141216
WDG-5-Br-H CDCl3
O
O
Br
2j
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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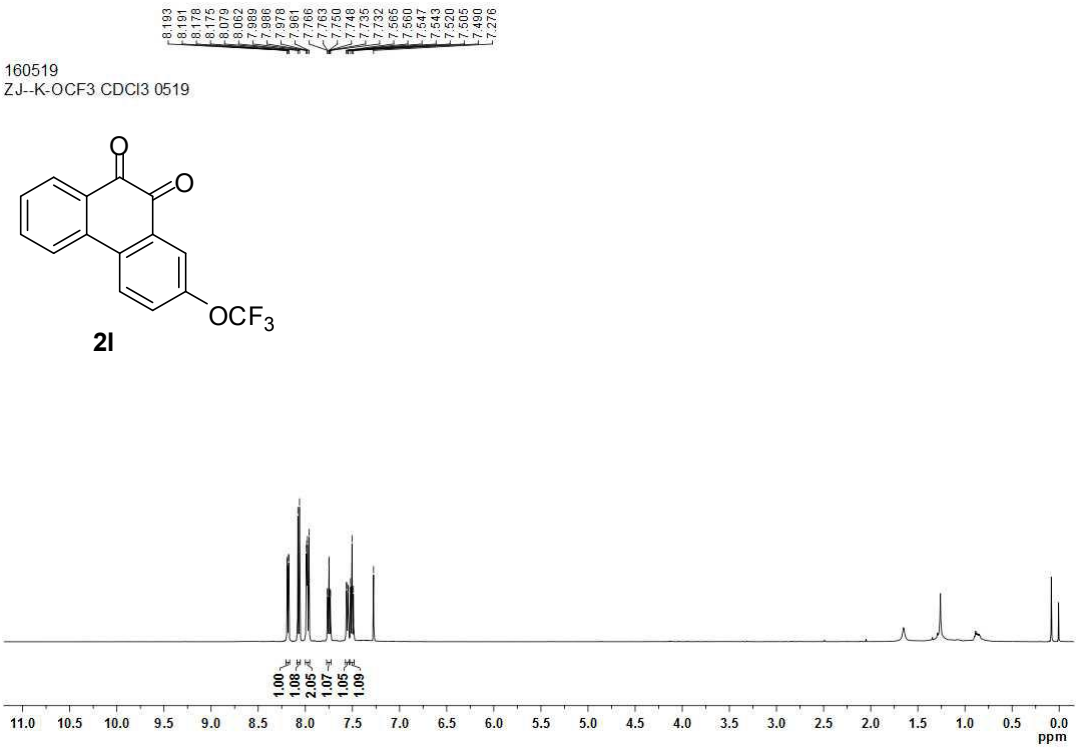
8.223
8.126
8.123
8.111
8.109
8.096
8.093
7.861
7.858
7.845
7.843
7.830
7.827
7.652
7.651
7.636
7.622
7.620
3.365
2.515
2.512
2.508
2.505
2.501
161104
BHY-CF3 DMSO 1104
O
O
CF3
2k
1.00
1.02
0.96
1.98
1.04
1.05
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
ppm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8.062
7.989
7.986
7.978
7.961
7.766
7.763
7.750
7.748
7.735
7.732
7.565
7.560
7.547
7.543
7.520
7.505
7.490
7.276
160519
ZJ--K-OCF3 CDCl3 0519
O
O
OCF3
2l
1.00
1.08
2.05
1.07
1.05
1.09
11.0 10.5 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
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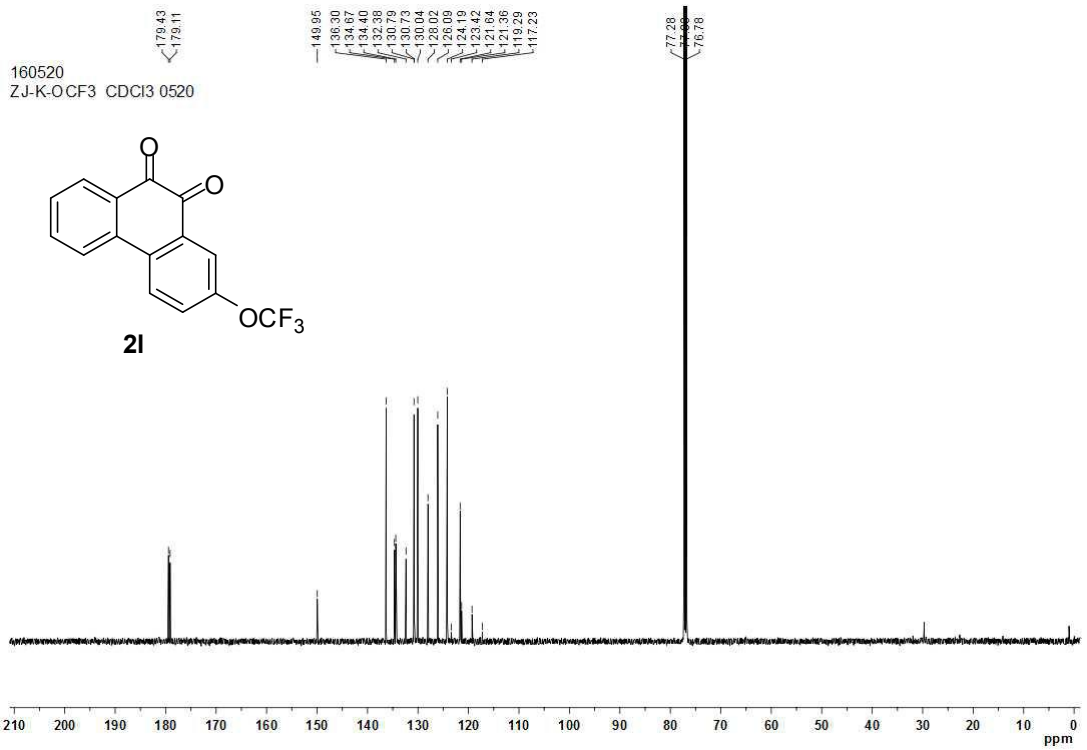
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149.95
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132.38
130.79
130.73
130.04
128.02
126.09
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121.36
119.29
117.23
77.28
76.78
160520
ZJ-K-O CF3 CDCl3 0520
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O
OCF3
2l
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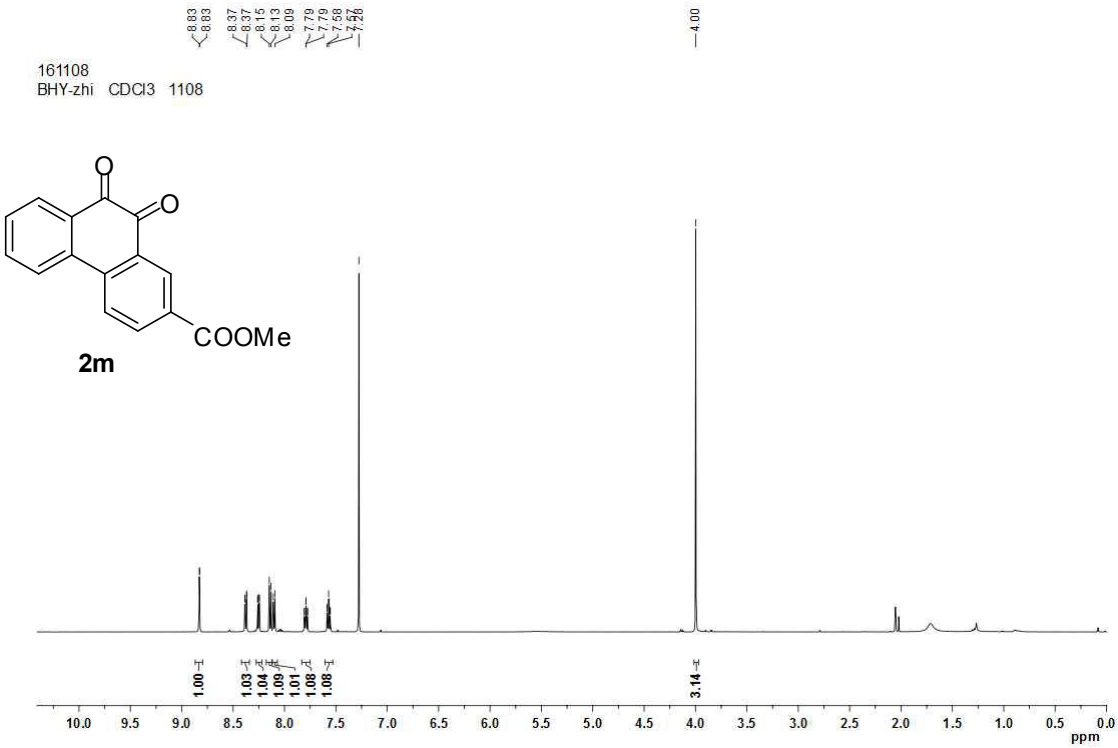
ppm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8.15
8.13
8.09
7.79
7.79
7.68
7.67
7.26
4.00
161108
BHY-zhi CDCl3 1108
O
O
COOMe
2m
1.00
1.03
1.04
1.09
1.01
1.08
1.08
3.14
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
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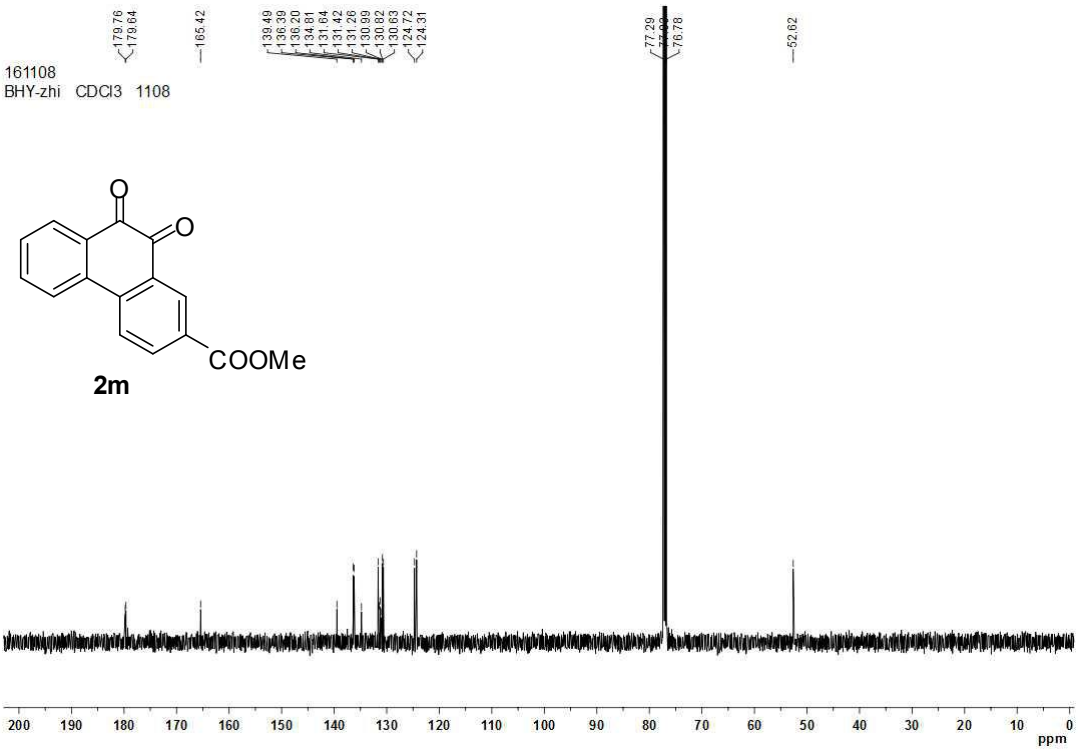
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165.42
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136.39
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131.64
131.42
131.26
130.99
130.82
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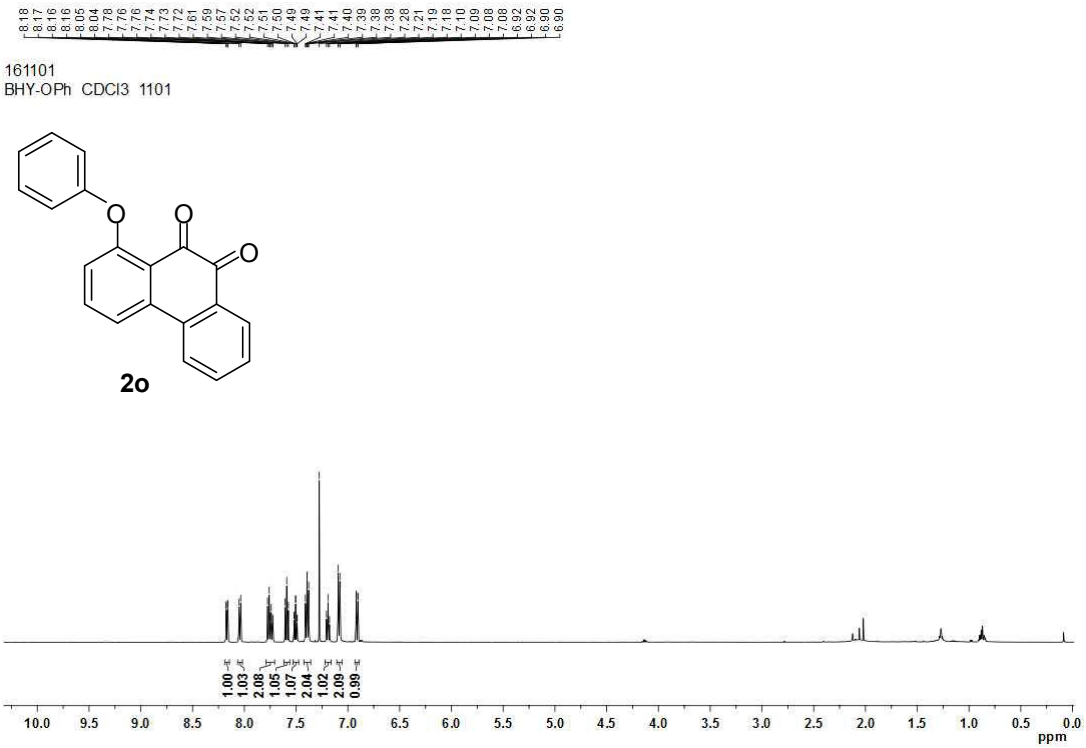
124.72
124.31
77.29
76.78
52.62
161108
BHY-zhi CDCl3 1108
O
O
COOMe
2m
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm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8.16
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7.76
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7.73
7.72
7.61
7.59
7.57
7.52
7.52
7.51
7.50
7.49
7.49
7.41
7.41
7.40
7.39
7.38
7.38
7.28
7.21
7.19
7.18
7.10
7.09
7.08
7.08
6.92
6.92
6.90
6.90
161101
BHY-OPh CDCl3 1101
O
O
O
2o
1.00
1.03
2.08
1.05
1.07
2.04
1.02
2.09
0.99
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
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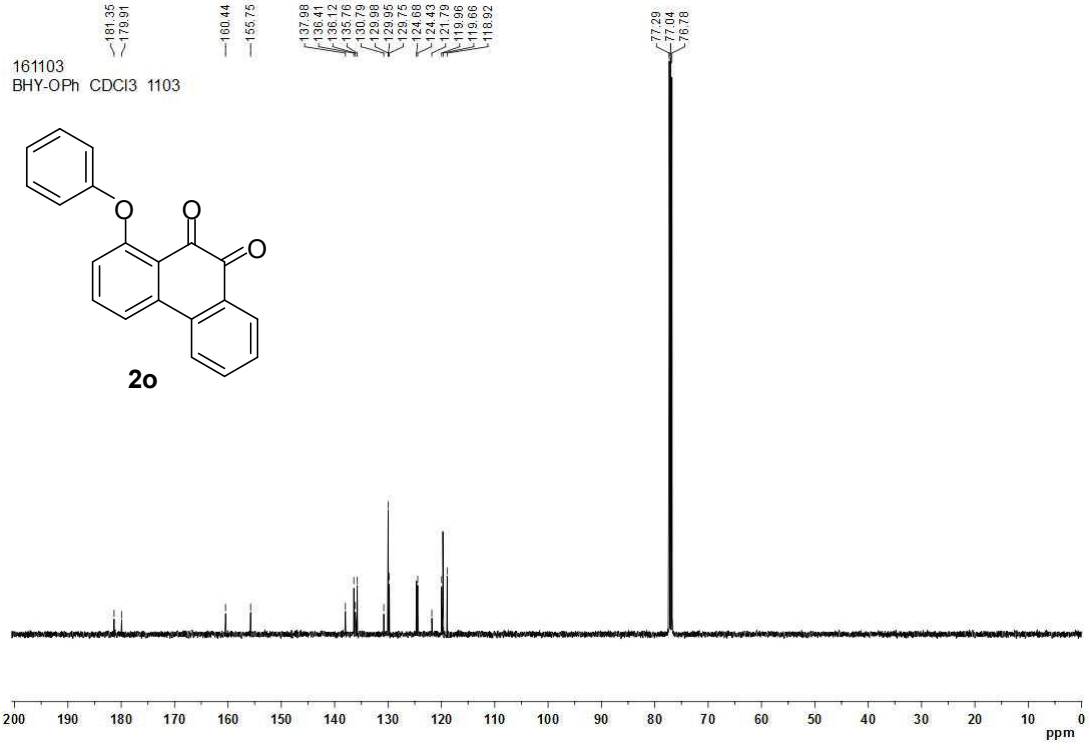
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129.75
124.68
124.43
121.79
119.96
119.66
118.92
77.29
77.04
76.78
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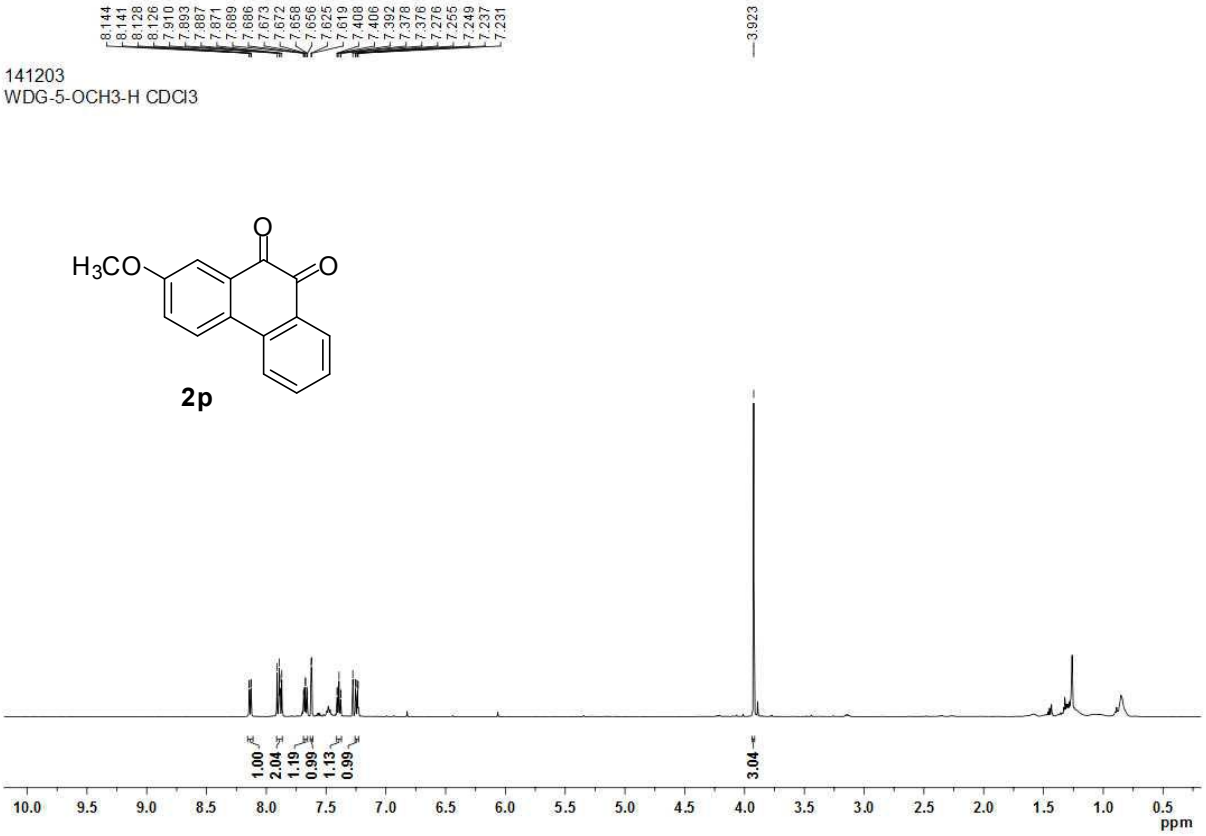
BHY-OPh CDCl3 1103
O
O
O
2o
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm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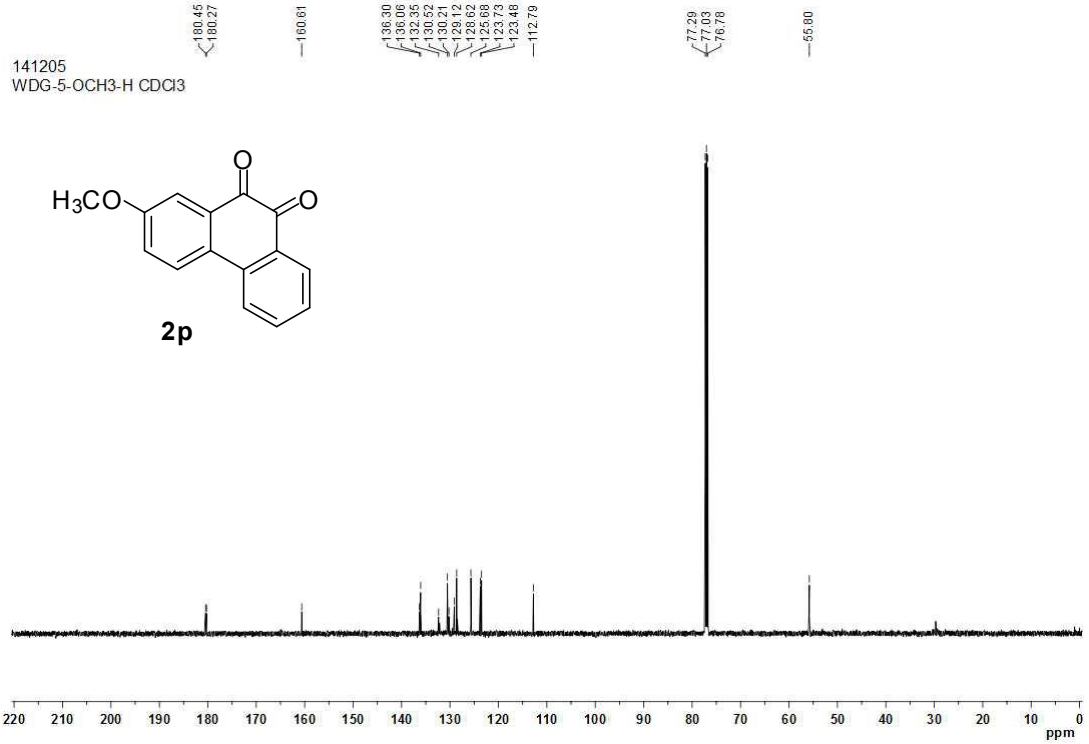
7.673
7.672
7.658
7.656
7.625
7.619
7.408
7.406
7.392
7.378
7.376
7.276
7.255
7.249
7.237
7.231
3.923
141203
WDG-5-OCH3-H CDCl3
O
H3CO
O
2p
1.00
2.04
1.19
0.99
1.13
0.99
3.04
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
ppm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130.21
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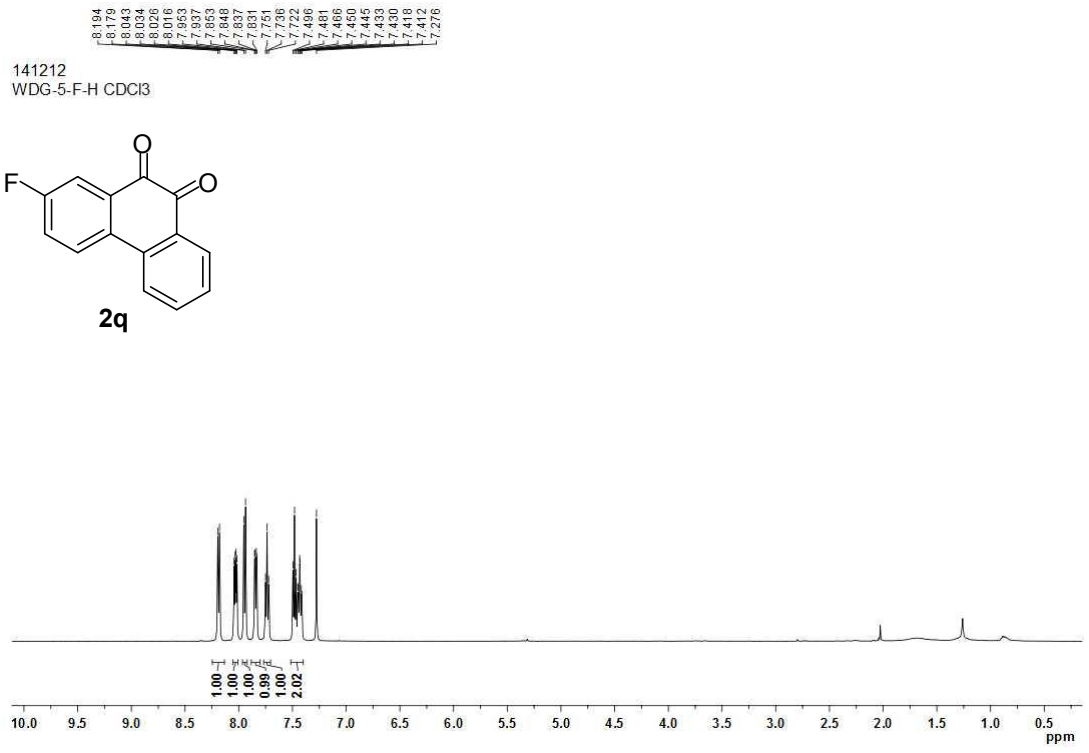
128.62
125.68
123.73
123.48
112.79
77.29
77.03
76.78
55.80
141205
WDG-5-OCH3-H CDCl3
H3CO
O
O
2p
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm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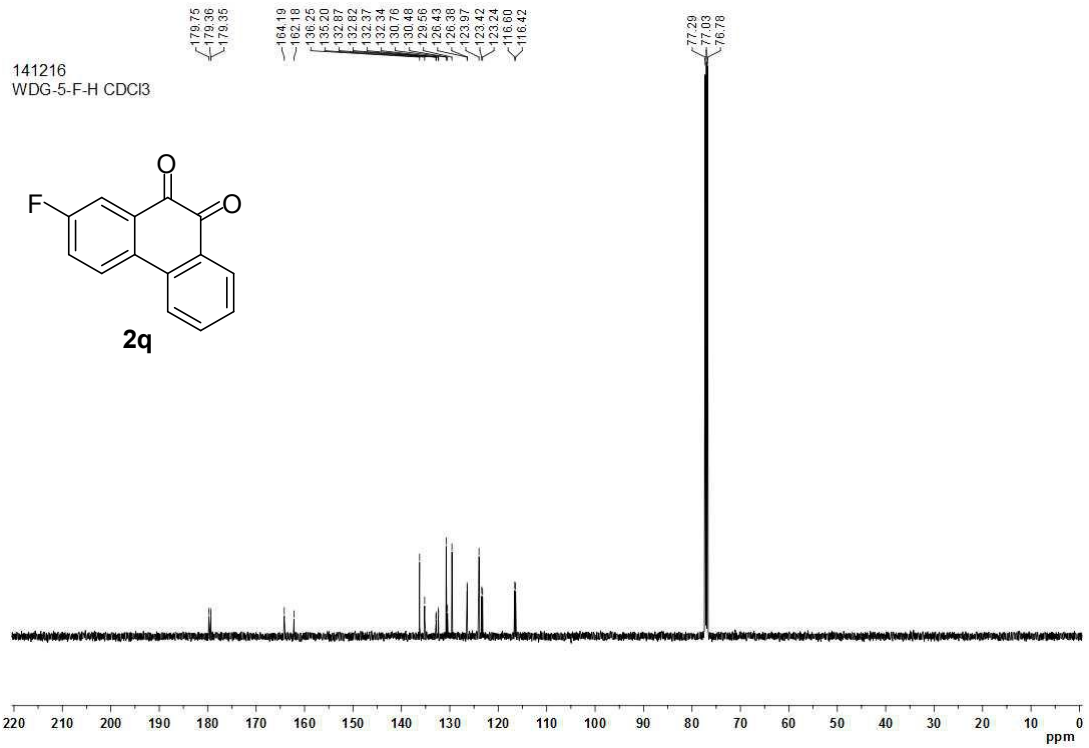
7.937
7.853
7.848
7.837
7.831
7.751
7.736
7.722
7.496
7.481
7.466
7.450
7.445
7.433
7.430
7.418
7.412
7.276
141212
WDG-5-F-H CDCl3
F
O
O
2q
1.00
1.00
1.00
0.99
1.00
2.02
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
ppm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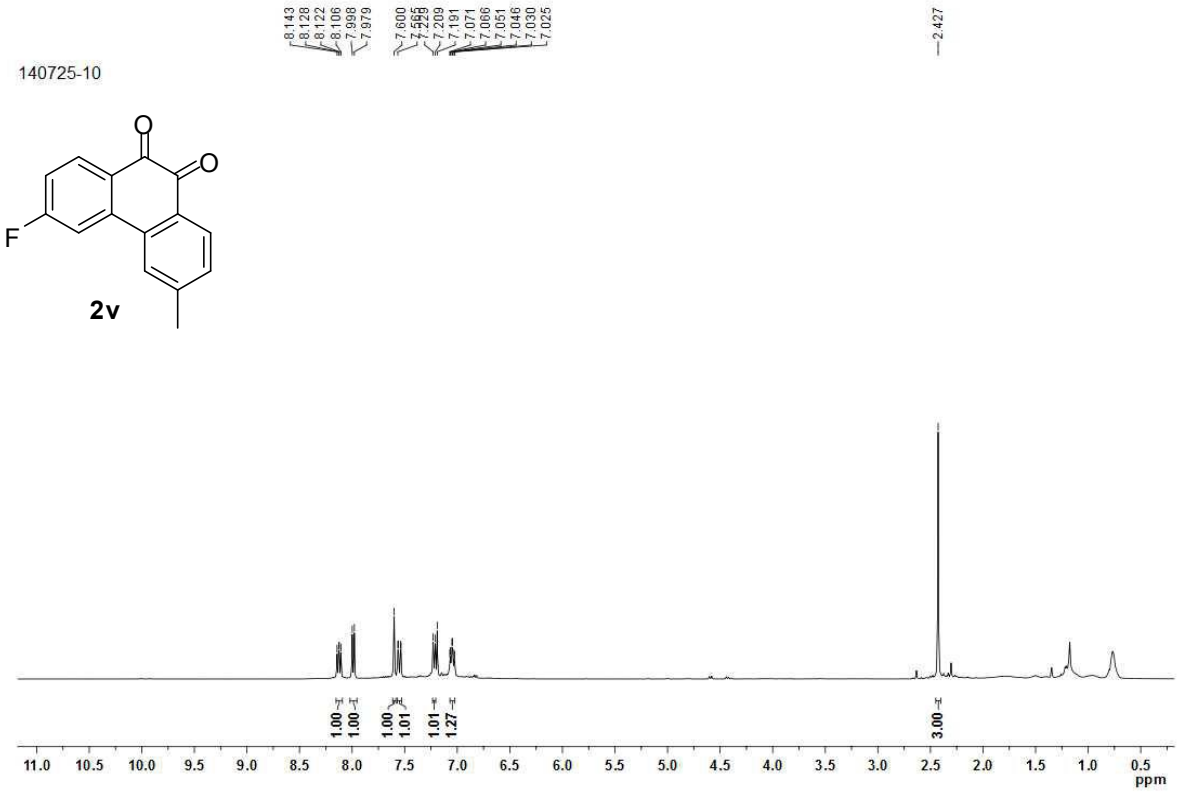
132.82
132.37
132.34
130.76
130.48
129.56
126.43
126.38
123.97
123.42
123.24
116.60
116.42
77.29
77.03
76.78
F
O
O
2q
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm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7.565
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7.209
7.191
7.071
7.066
7.051
7.046
7.030
7.025
2.427
140725-10
F
O
O
2v
1.00
1.00
1.00
1.01
1.01
1.27
3.00
11.0 10.5 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
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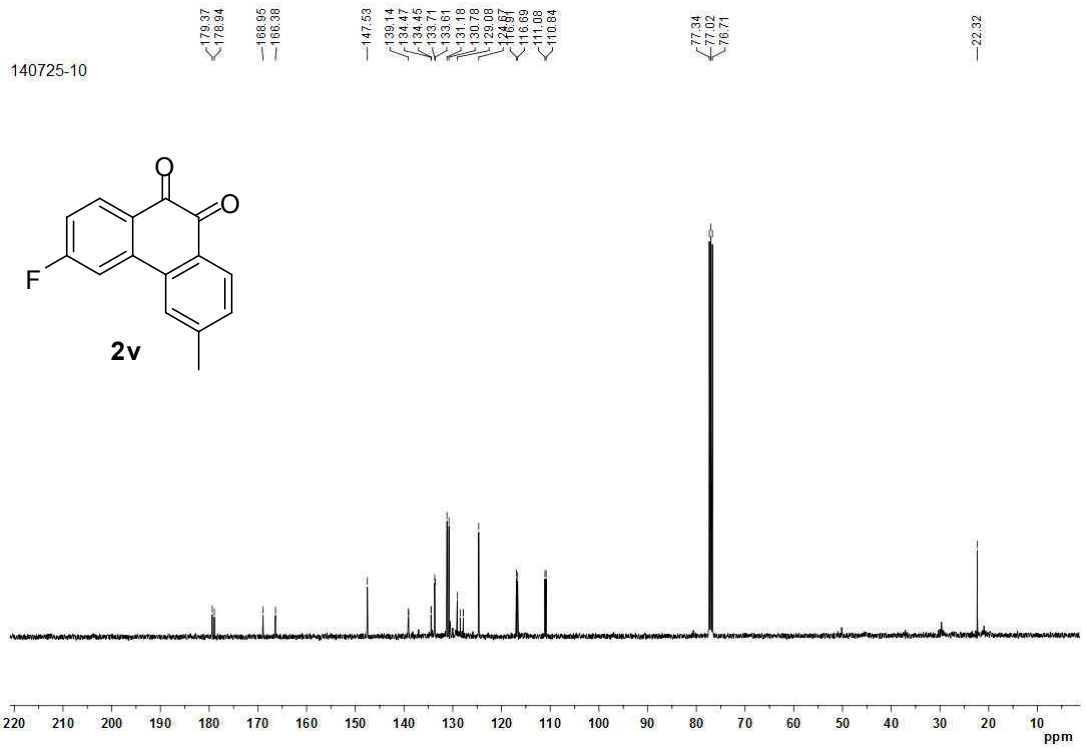
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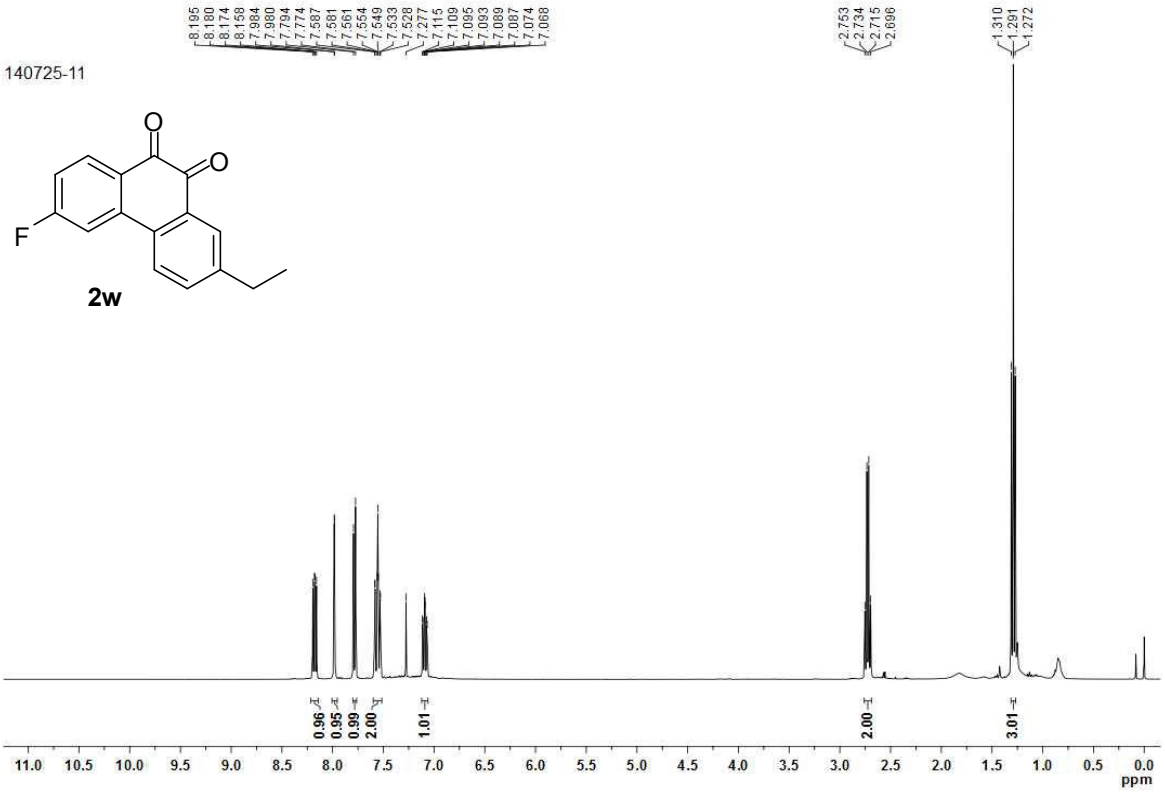
133.61
131.18
130.78
129.08
124.67
116.91
116.69
111.08
110.84
77.34
77.02
76.71
22.32
140725-10
O
O
F
2v
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm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7.774
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7.554
7.549
7.533
7.528
7.277
7.115
7.109
7.095
7.093
7.089
7.087
7.074
7.068
2.753
2.734
2.715
2.696
1.310
1.291
1.272
140725-11
O
O
F
2w
0.96
0.95
0.99
2.00
1.01
2.00
3.01
11.0 10.5 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
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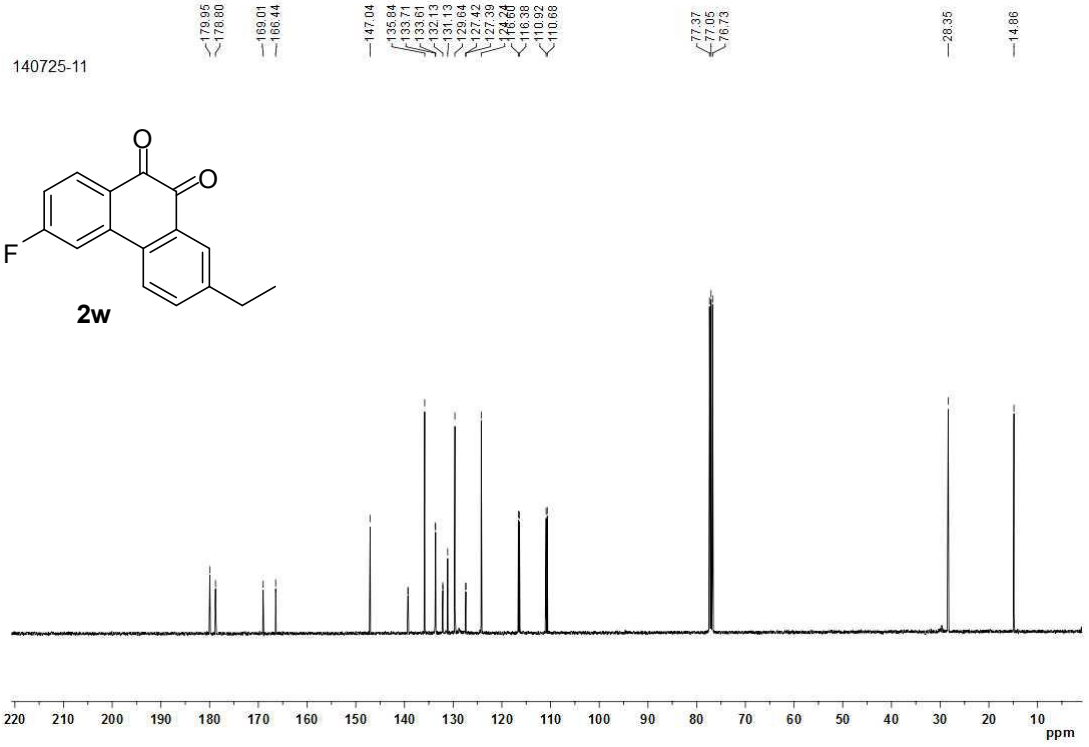
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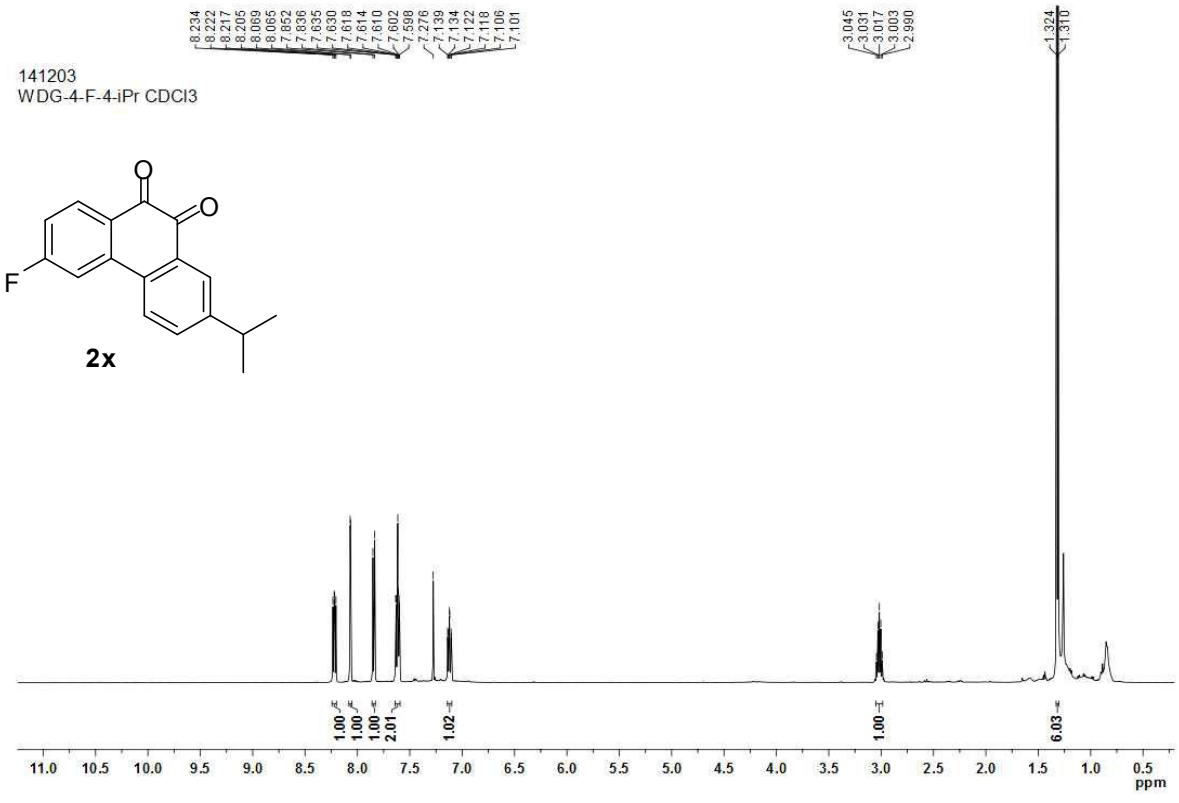
132.13
131.13
129.64
127.42
127.39
124.24
116.60
116.38
110.92
110.68
77.37
77.05
76.73
28.35
14.86
O
O
F
2w
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm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7.630
7.618
7.614
7.610
7.602
7.598
7.276
7.139
7.134
7.122
7.118
7.106
7.101
3.045
3.031
3.017
3.003
2.990
1.324
1.310
141203
WDG-4-F-4-iPr CDCl3
O
O
F
2x
1.00
1.00
1.00
2.01
1.02
1.00
6.03
11.0 10.5 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
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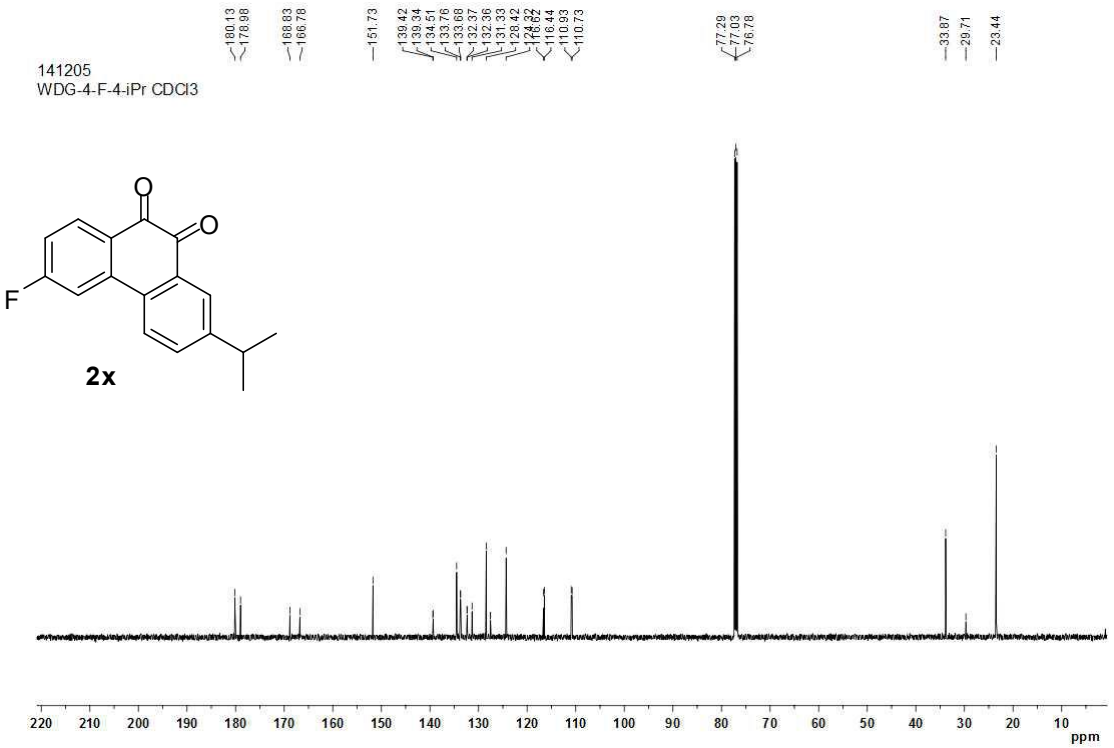
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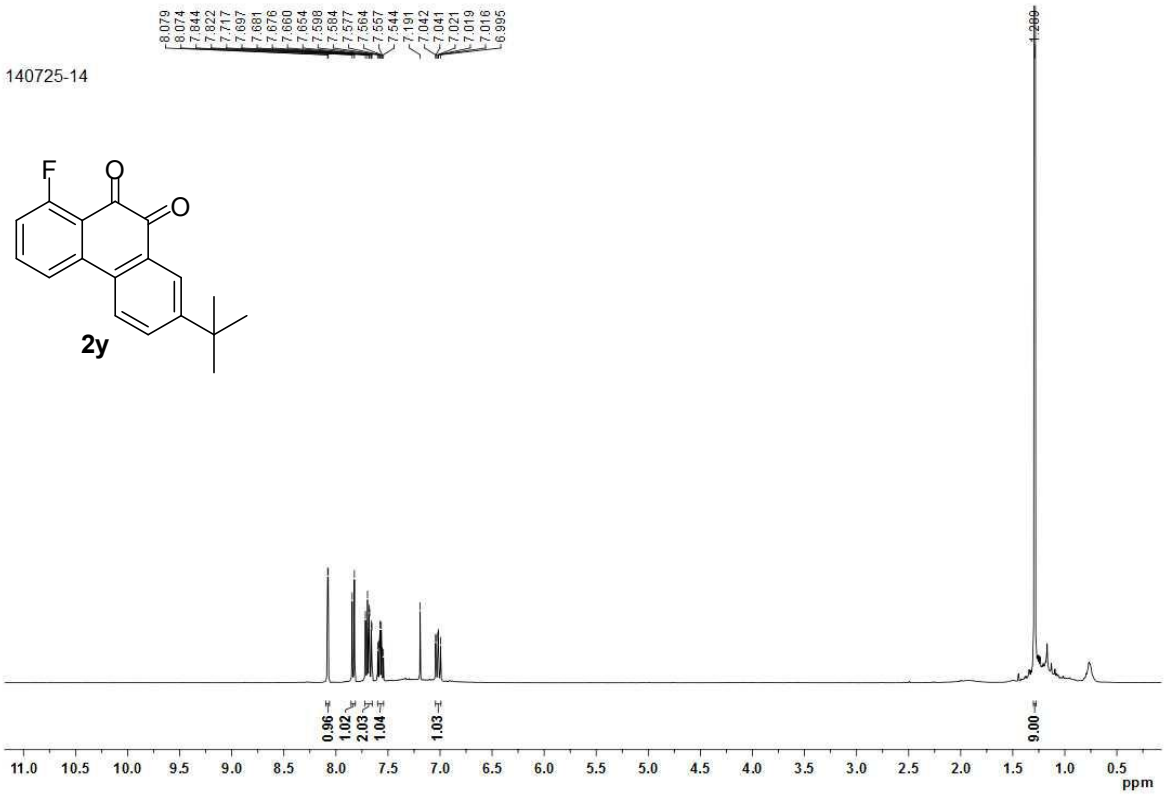
133.68
132.37
132.36
131.33
128.42
124.32
116.62
116.44
110.93
110.73
77.29
77.03
76.78
33.87
29.71
23.44
141205
WDG-4-F-4-iPr CDCl3
O
O
F
2x
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm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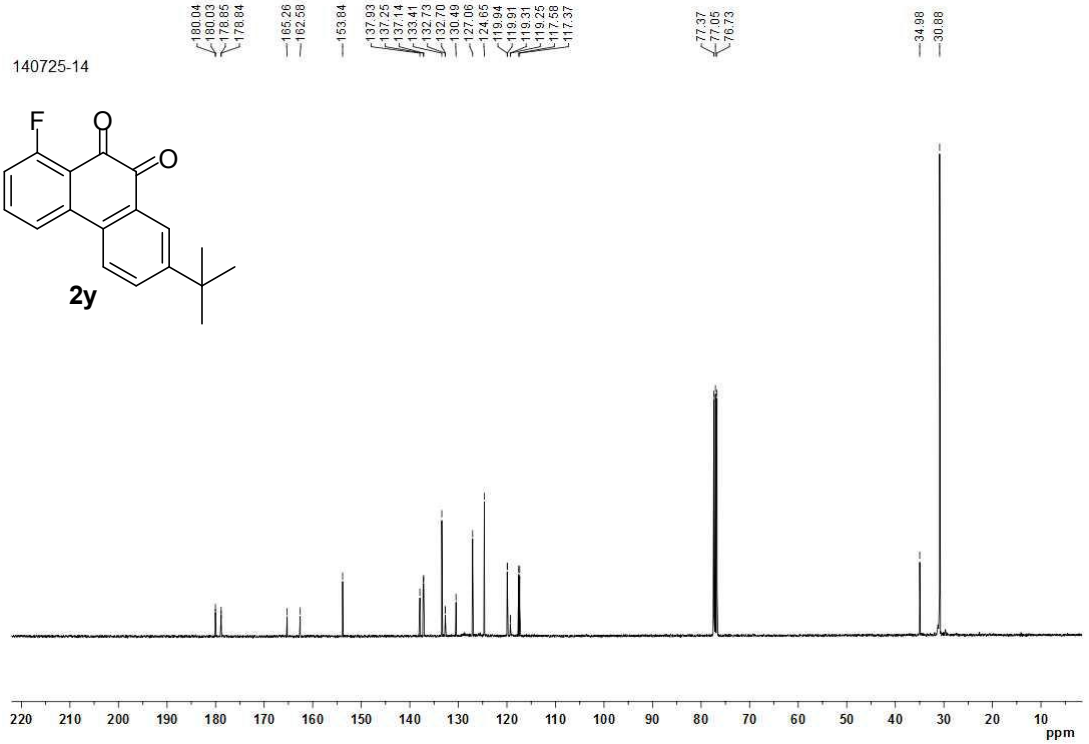
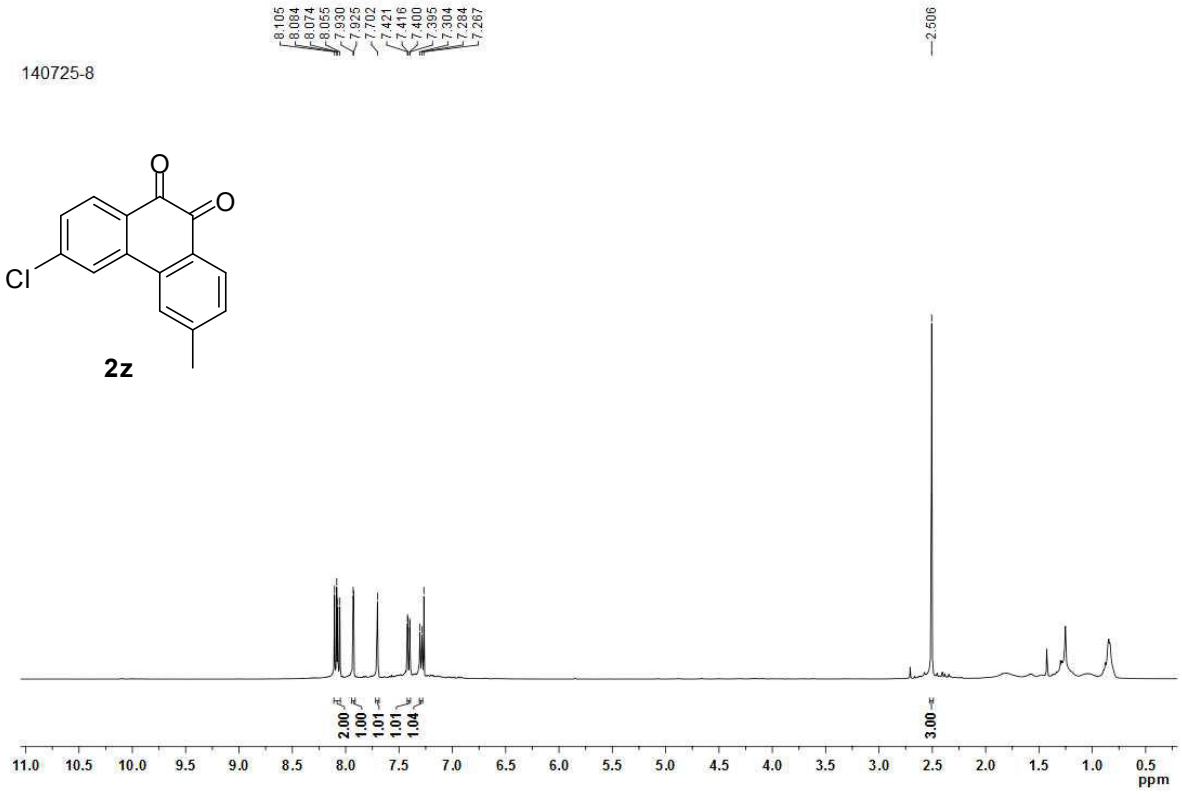
7.598
7.584
7.577
7.564
7.557
7.544
7.191
7.042
7.041
7.021
7.019
7.016
6.995
1.280
140725-14
F
O
O
2y
0.96
1.02
2.03
1.04
1.03
9.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
ppm


180.04
180.03
178.85
178.84
165.26
162.58
153.84
137.93
137.25
137.14
133.41
132.73
132.70
130.49
127.06
124.65
119.94
119.91
119.31
119.25
117.58
117.37
77.37
77.05
76.73
34.98
30.88
140725-14
F
O
O
2y
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm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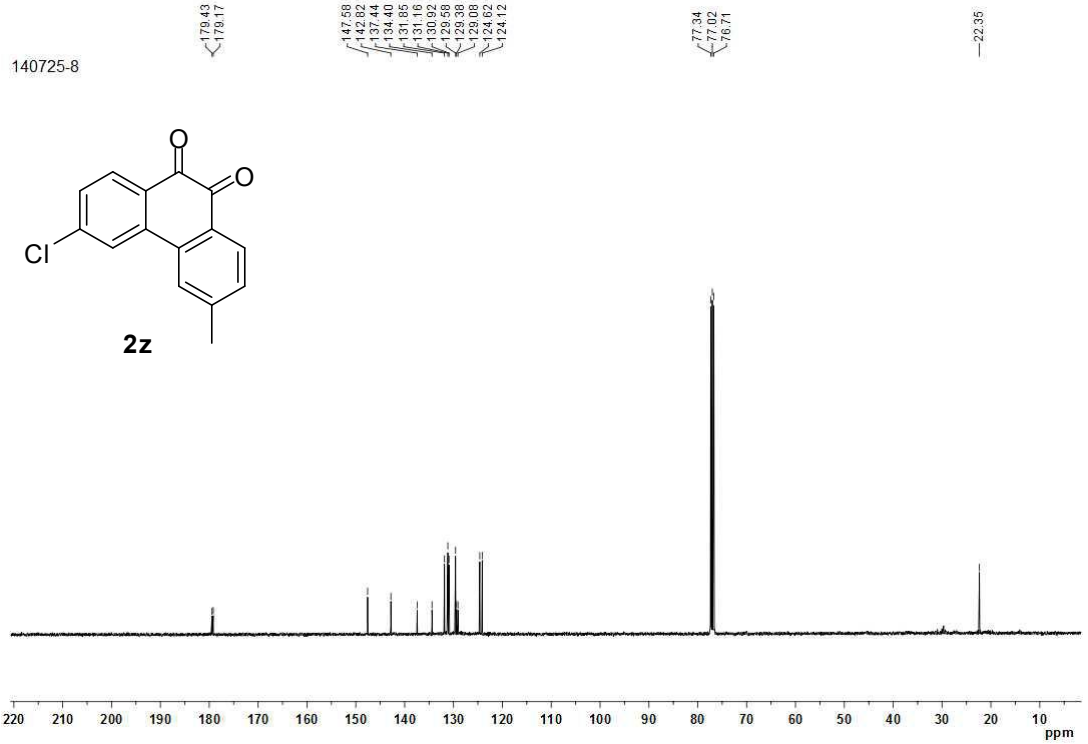
8.084
8.074
8.055
7.930
7.925
7.702
7.421
7.416
7.400
7.395
7.304
7.284
7.267
2.506
140725-8
O
O
Cl
2z
2.00
1.00
1.01
1.01
1.04
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
ppm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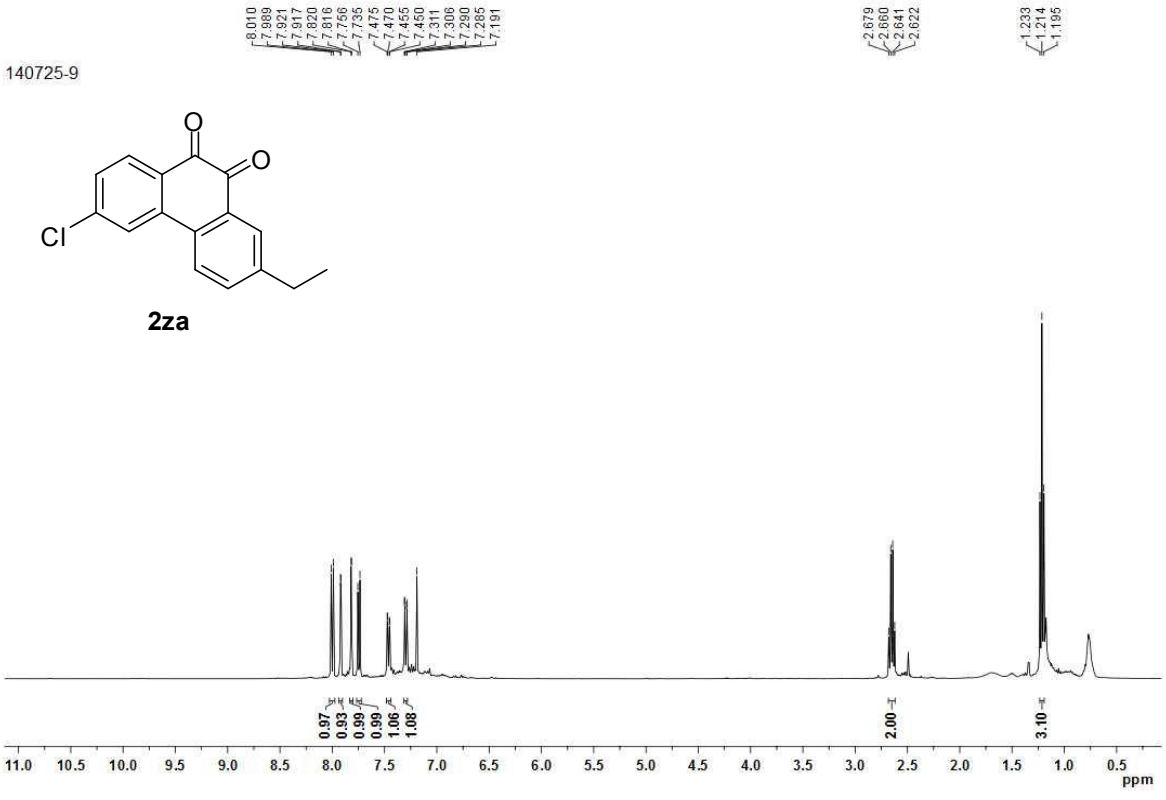
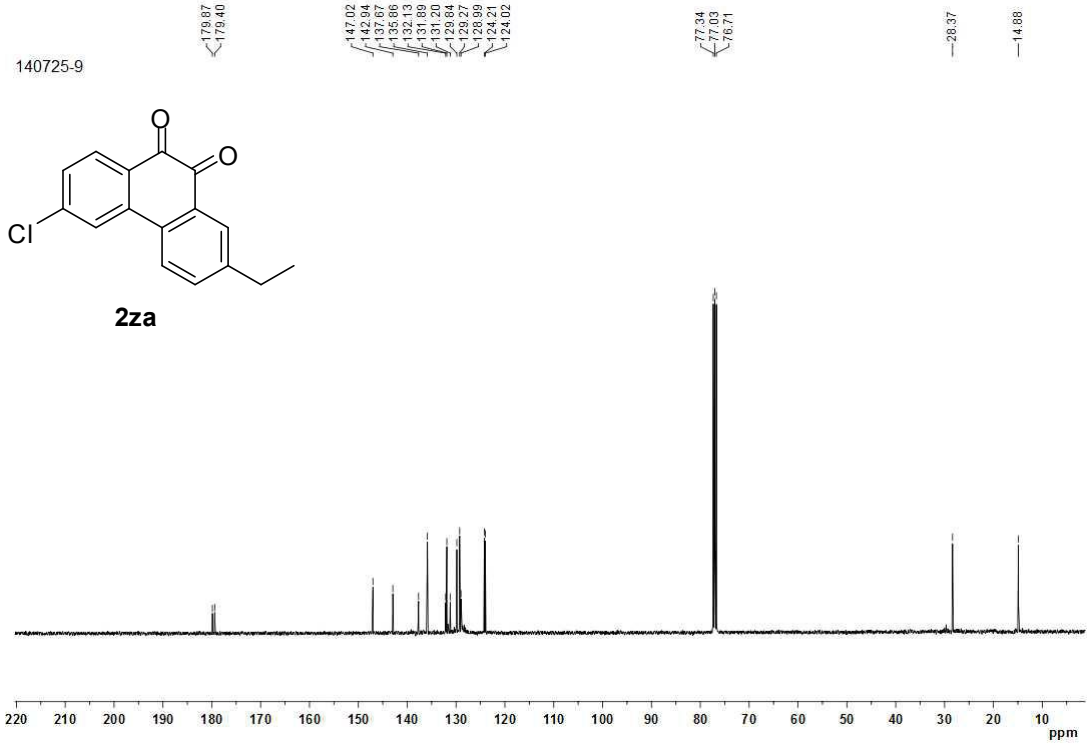
179.17
147.58
142.82
137.44
134.40
131.85
131.16
130.92
129.58
129.38
129.08
124.62
124.12
77.34
77.02
76.71
22.35
140725-8
O
O
Cl
2z
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm


8.010
7.989
7.921
7.917
7.820
7.816
7.756
7.735
7.475
7.470
7.455
7.450
7.311
7.306
7.290
7.285
7.191
2.679
2.660
2.641
2.622
1.233
1.214
1.195
140725-9
O
O
Cl
2za
0.97
0.93
0.99
0.99
1.06
1.08
2.00
3.10
11.0 10.5 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
ppm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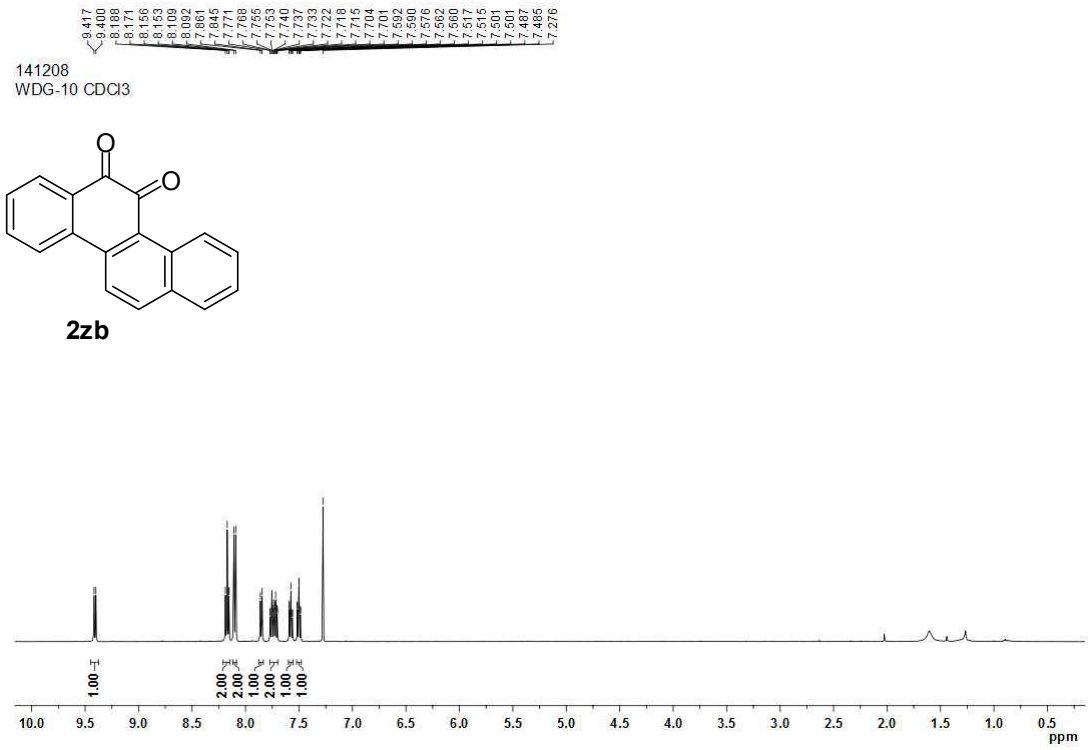
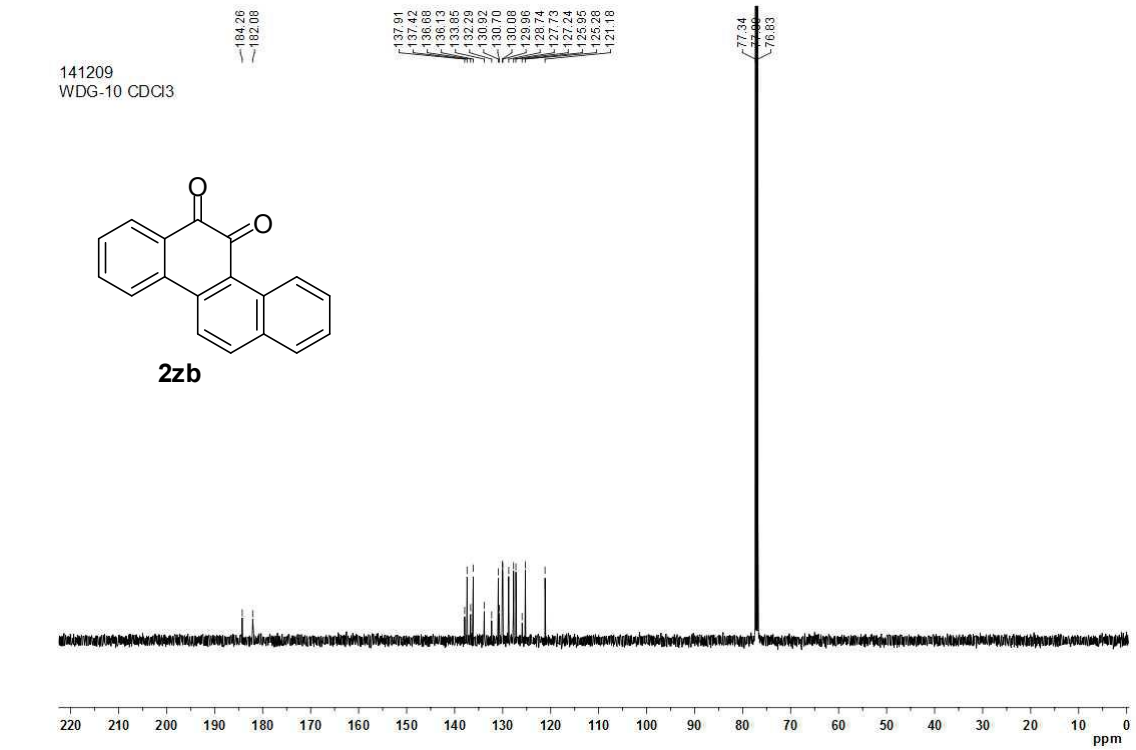
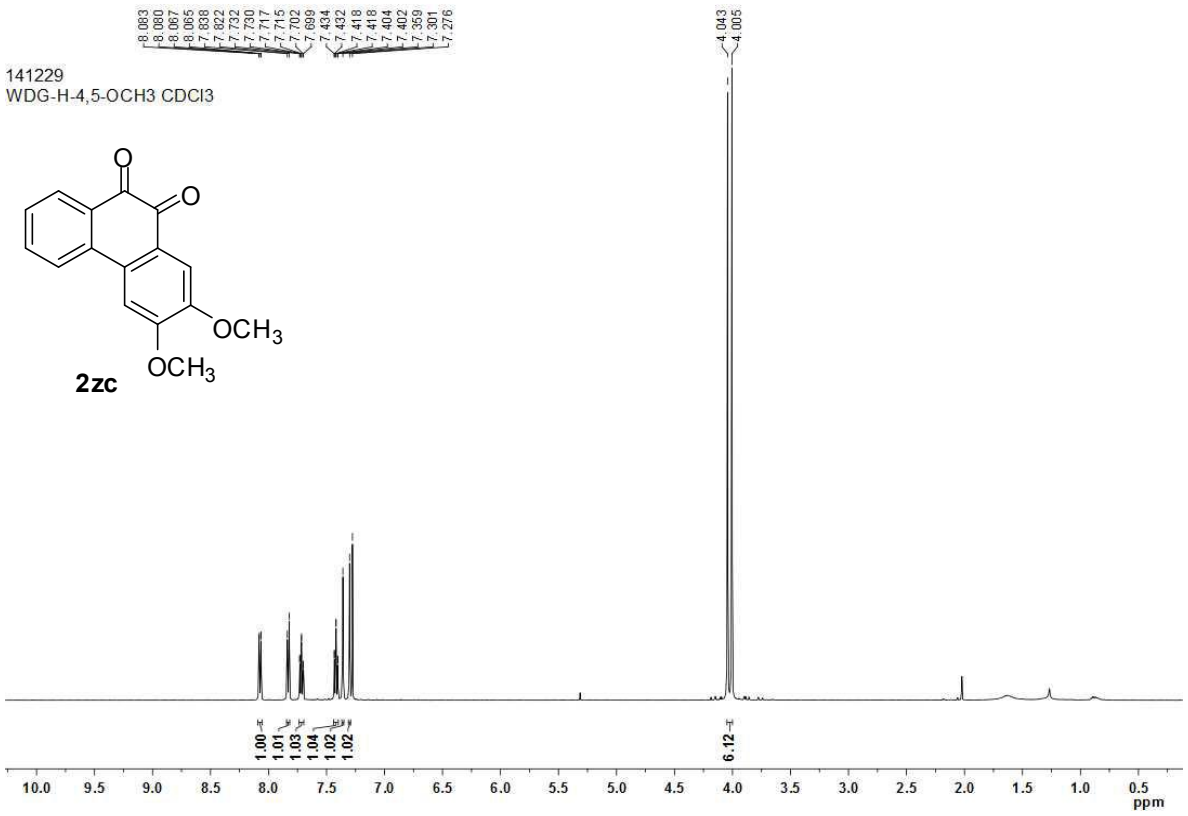
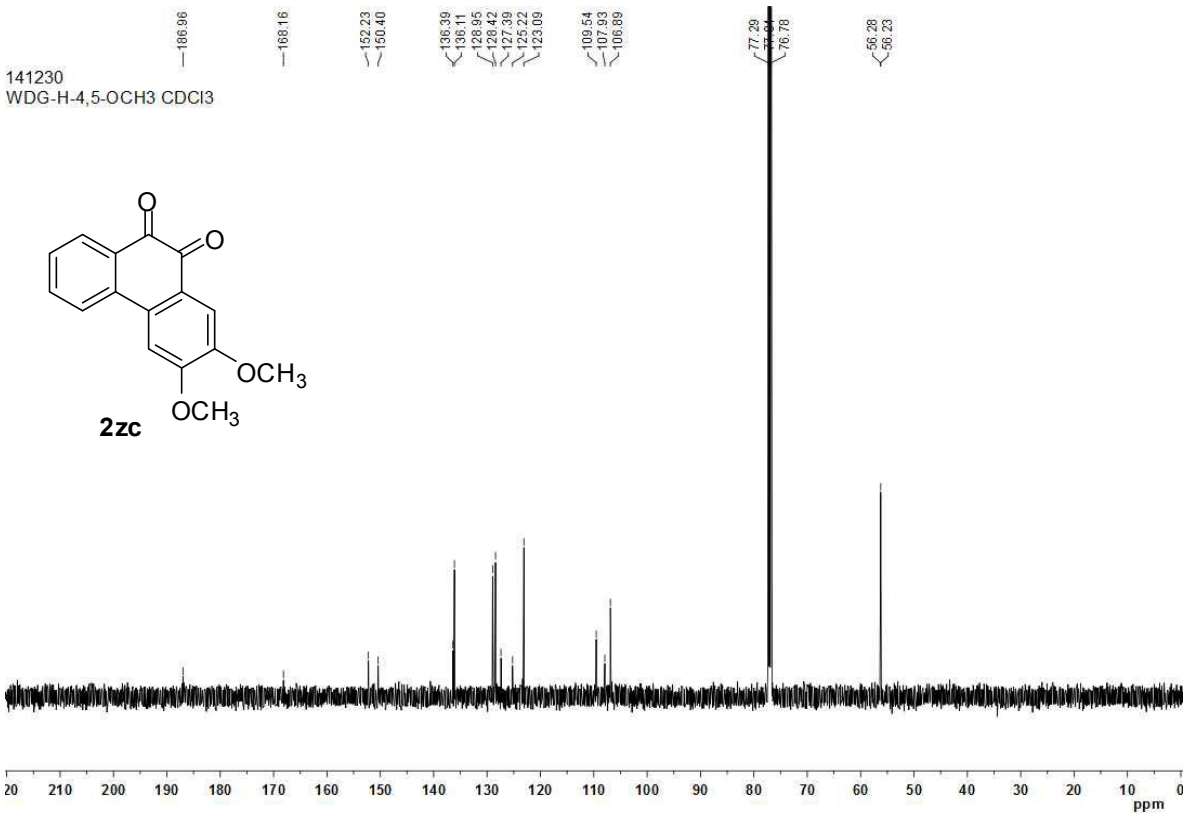
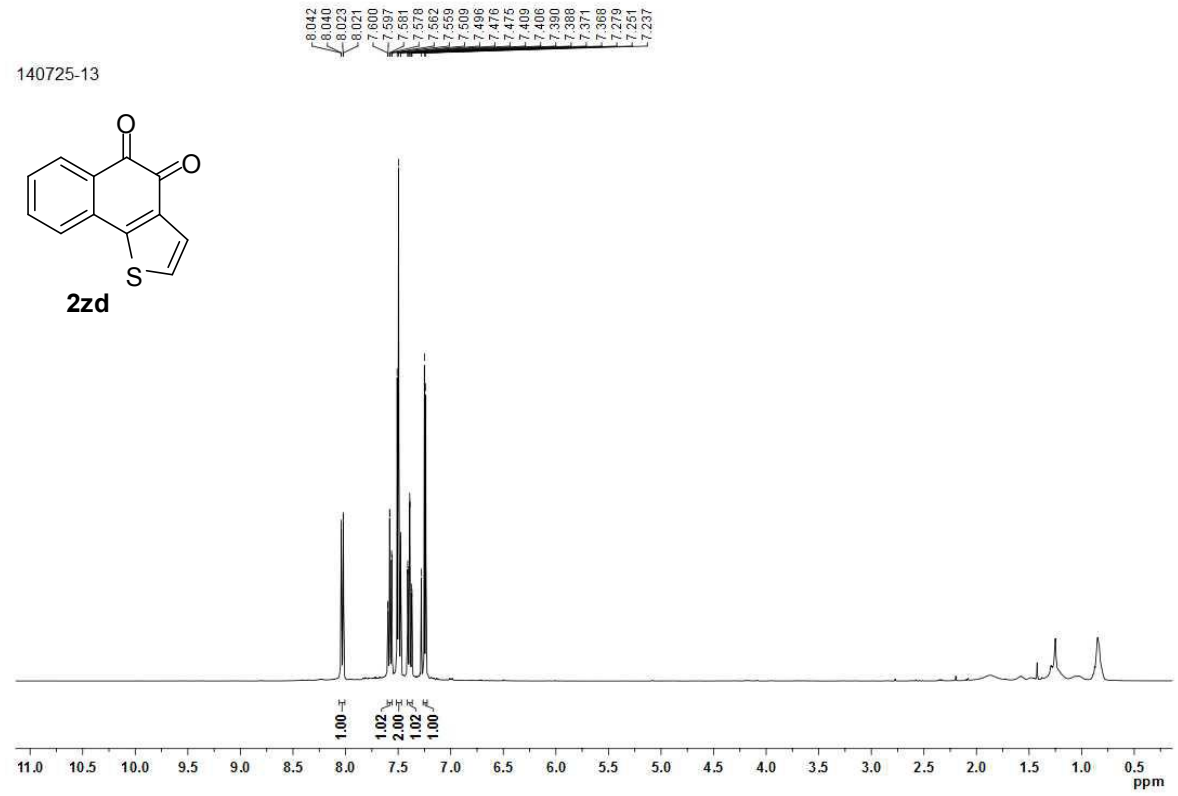
179.40
147.02
142.94
137.67
135.86
132.13
131.89
131.20
129.84
129.27
128.99
124.21
124.02
77.34
77.03
76.71
28.37
14.88
140725-9
O
O
Cl
2za
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
ppm

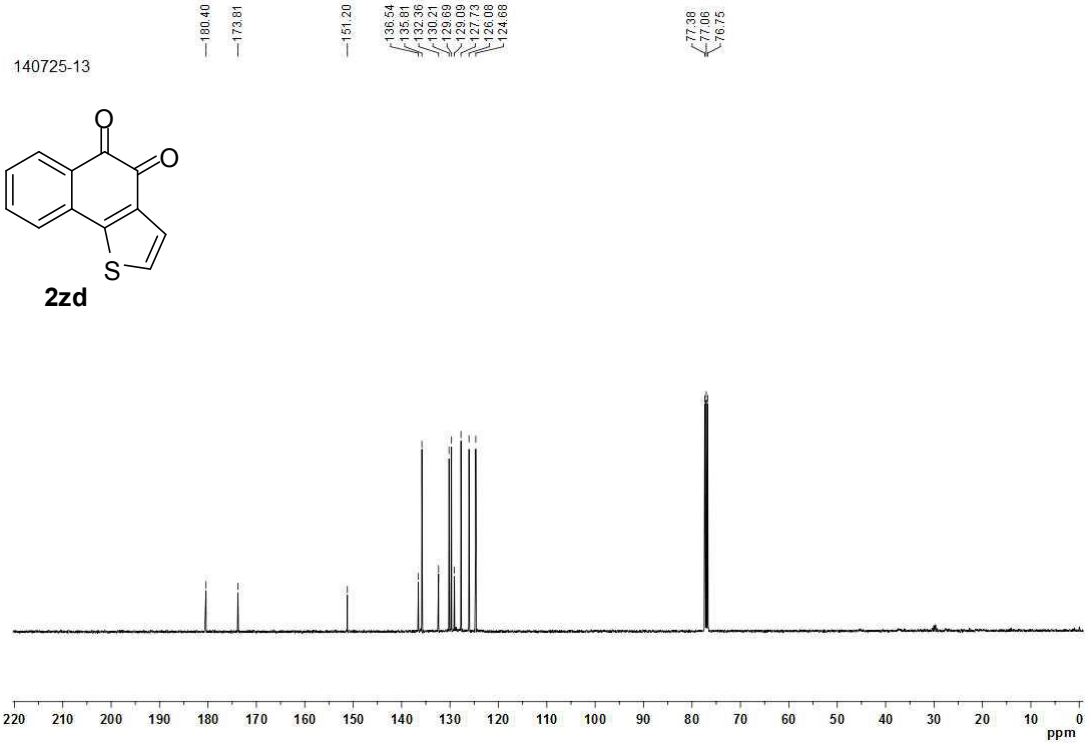

9.417
9.400
8.188
8.171
8.156
8.153
8.109
8.092
7.861
7.845
7.771
7.768
7.755
7.753
7.740
7.737
7.733
7.722
7.718
7.715
7.704
7.701
7.592
7.590
7.576
7.562
7.560
7.517
7.515
7.501
7.501
7.487
7.485
7.276
141208
WDG-10 CDCl3
O
O
2zb
1.00
2.00
2.00
1.00
2.00
1.00
1.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
ppm


184.26
182.08
137.91
137.42
136.68
136.13
133.85
132.29
130.92
130.70
130.08
129.96
128.74
127.73
127.24
125.95
125.28
121.18
77.34
77.00
76.83
141209
WDG-10 CDCl3
O
O
2zb
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm


8.083
8.080
8.067
8.065
7.838
7.822
7.732
7.730
7.717
7.715
7.702
7.699
7.434
7.432
7.418
7.418
7.404
7.402
7.359
7.301
7.276
4.043
4.005
141229
WDG-H-4,5-OCH3 CDCl3
O
O
OCH3
OCH3
2zc
1.00
1.01
1.03
1.04
1.02
1.02
6.12
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
ppm


186.96
168.16
152.23
150.40
136.39
136.11
128.95
128.42
127.39
125.22
123.09
109.54
107.93
106.89
77.29
76.78
56.28
56.23
141230
WDG-H-4,5-OCH3 CDCl3
O
O
OCH3
OCH3
2zc
ppm


8.042
8.040
8.023
8.021
7.600
7.597
7.581
7.578
7.562
7.559
7.509
7.496
7.476
7.475
7.409
7.406
7.390
7.388
7.371
7.368
7.279
7.251
7.237
140725-13
O
O
S
2zd
1.00
1.02
2.00
1.02
1.00
ppm



4. ^{1}H spectrum of 1u-d_5