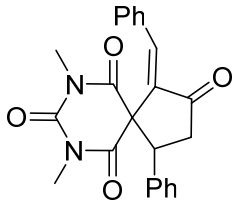
Electronic Supplementary Information

Phosphine-Catalyzed [3+2] and [4+2] Annulation Reactions of Ynones with Barbiturate-derived Alkenes

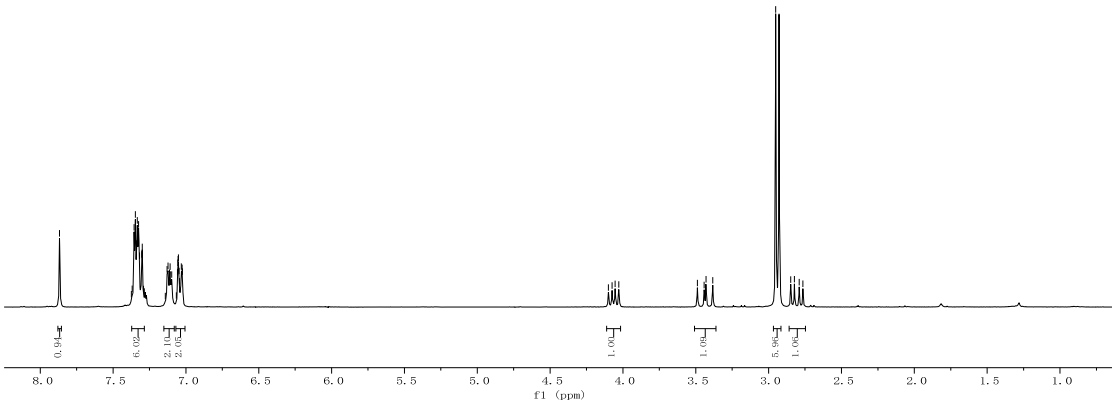
Xing Gao, Zhen Li, Wenjun Yang, Yang Liu, Wufeng Chen, Cheng Zhang, Lufei Zheng,* and Hongchao Guo*

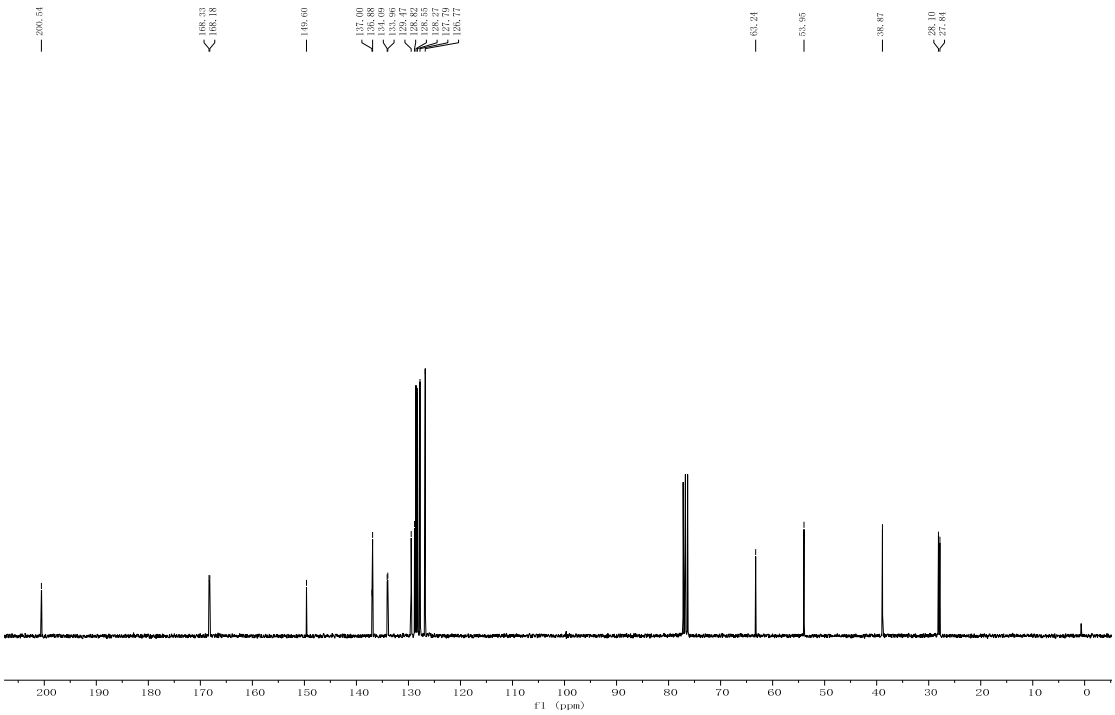
Contents

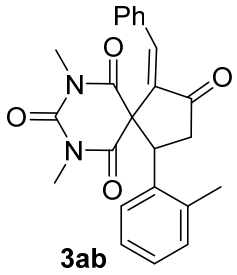
^{1}H and ^{13}C NMR Spectra of All Products



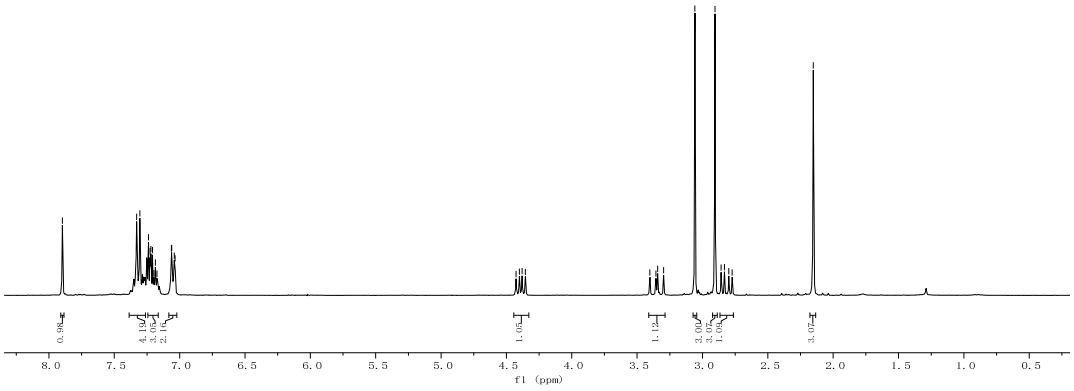
3aa

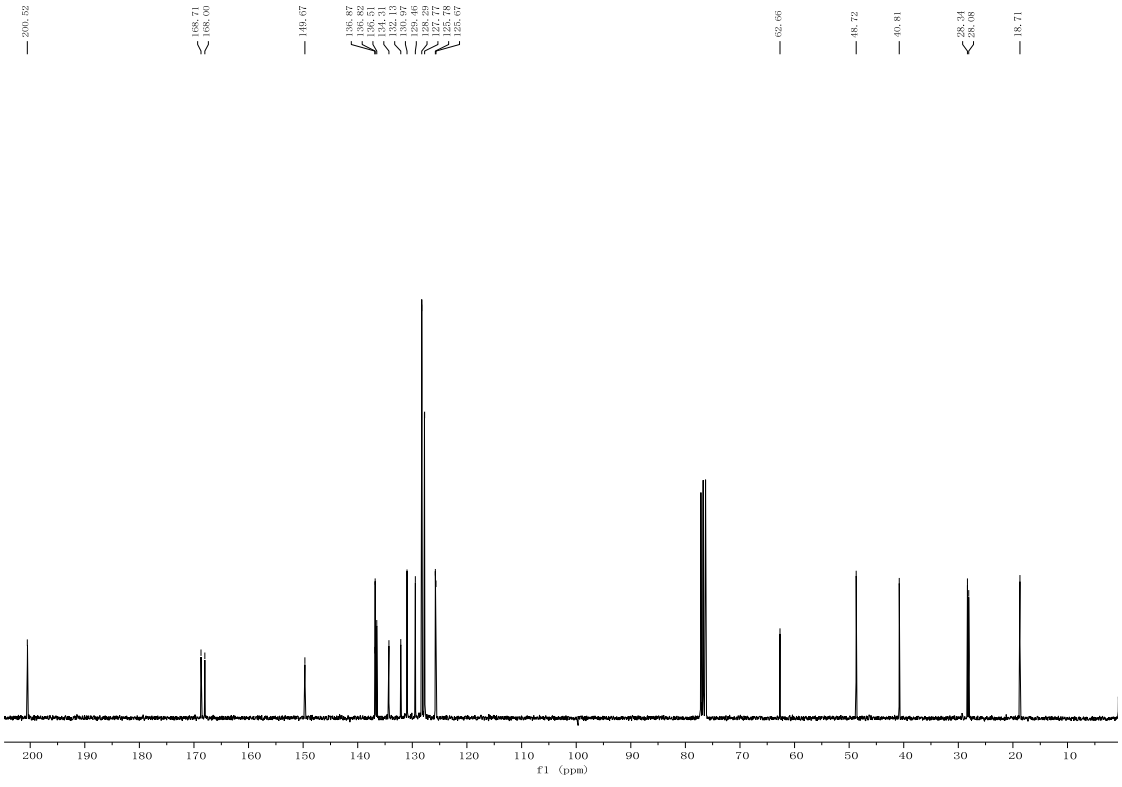


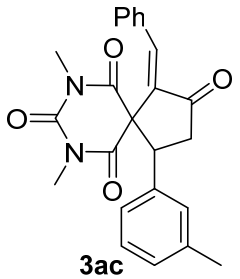




3ab

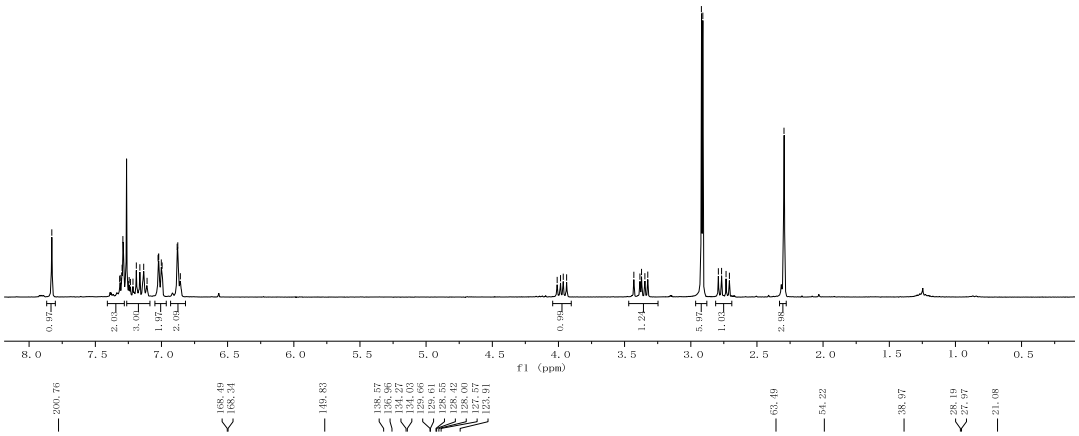


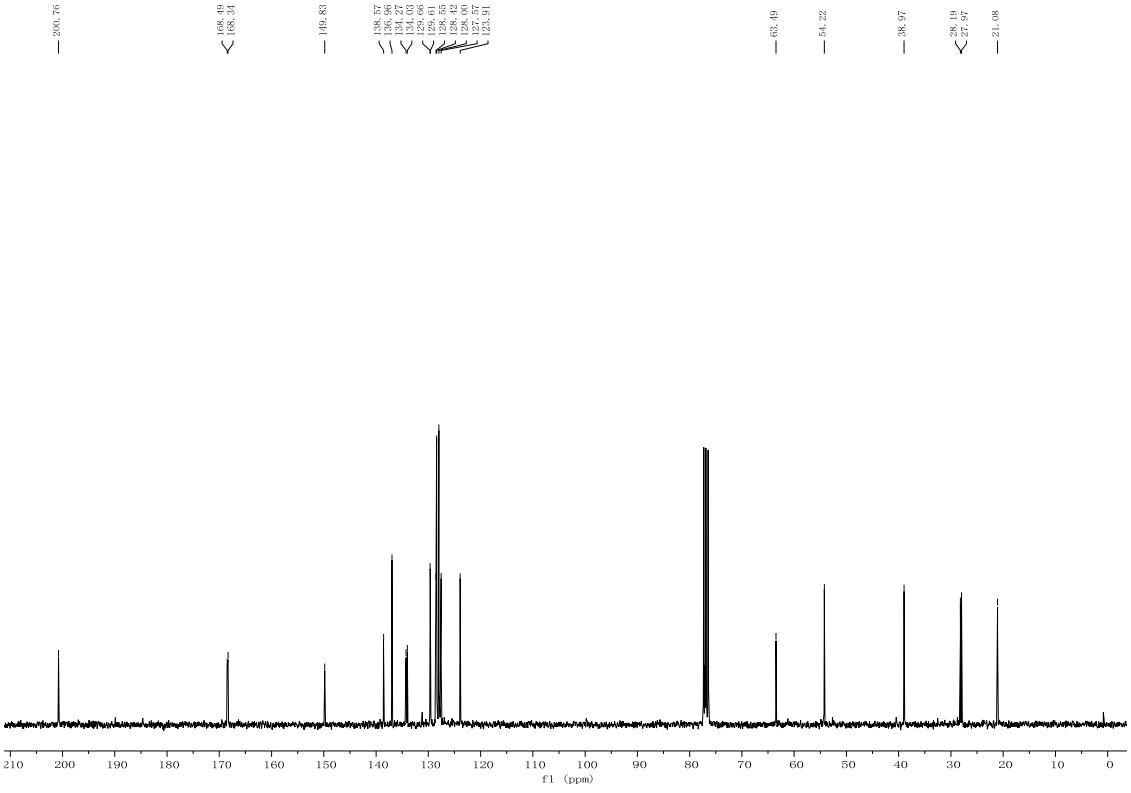


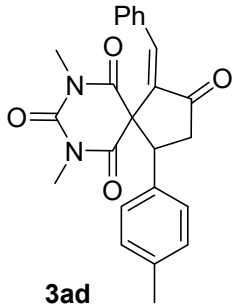

Ph
O
N
O
N
O
O
O

3ac

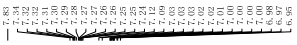

7.83
7.32
7.31
7.30
7.30
7.29
7.25
7.24
7.24
7.22
7.19
7.16
7.13
7.11
7.11
7.02
7.02
7.00
7.00
6.88
6.88
6.86
4.01
3.98
3.96
3.94
3.43
3.38
3.37
3.35
3.33
2.92
2.91
2.79
2.77
2.73
2.71
2.29

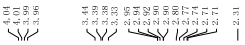

0.97
2.02
3.00
1.97
2.09
0.89
1.24
5.97
1.03
2.98
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

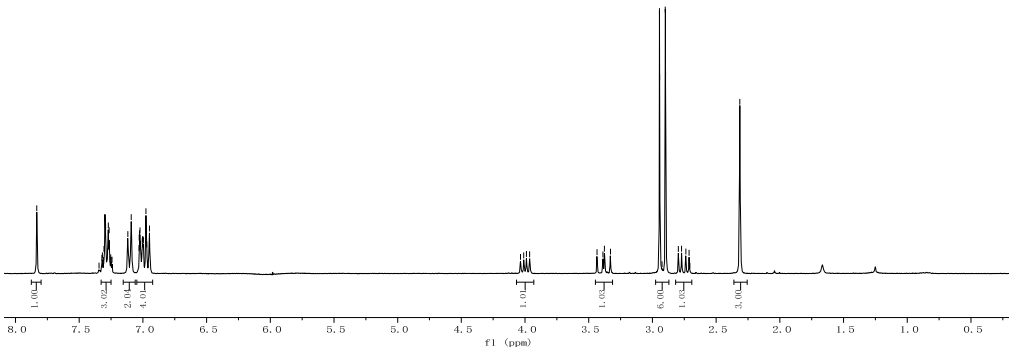

200.76
168.49
168.34
149.83
138.57
136.96
134.27
134.03
129.66
129.61
128.55
128.42
128.00
127.57
123.91
63.49
54.22
38.97
28.19
27.97
21.08
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

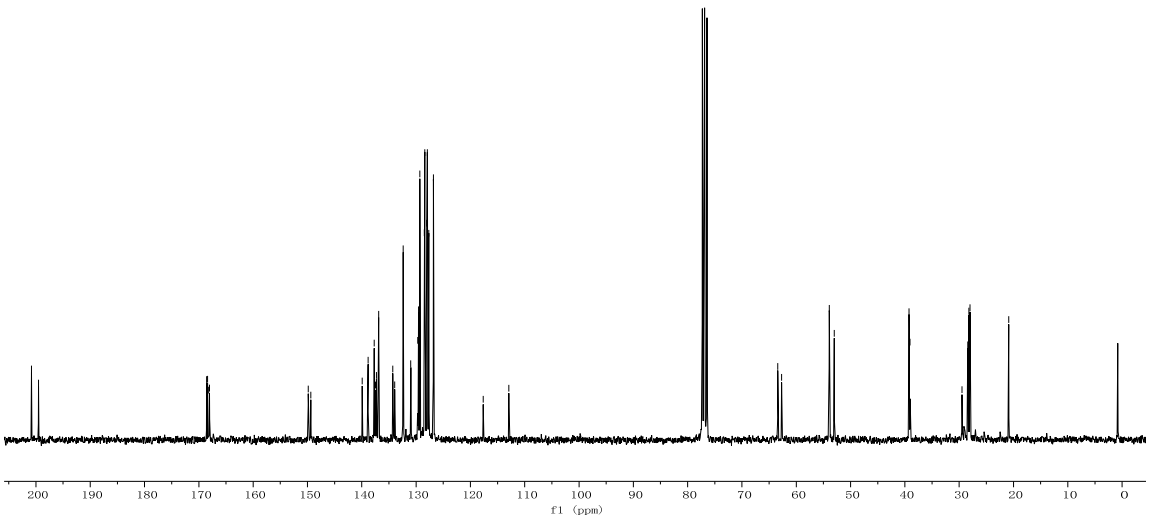


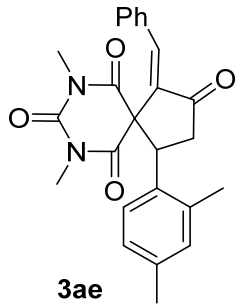
3ad










Ph
O
N
O
N
O
O
O

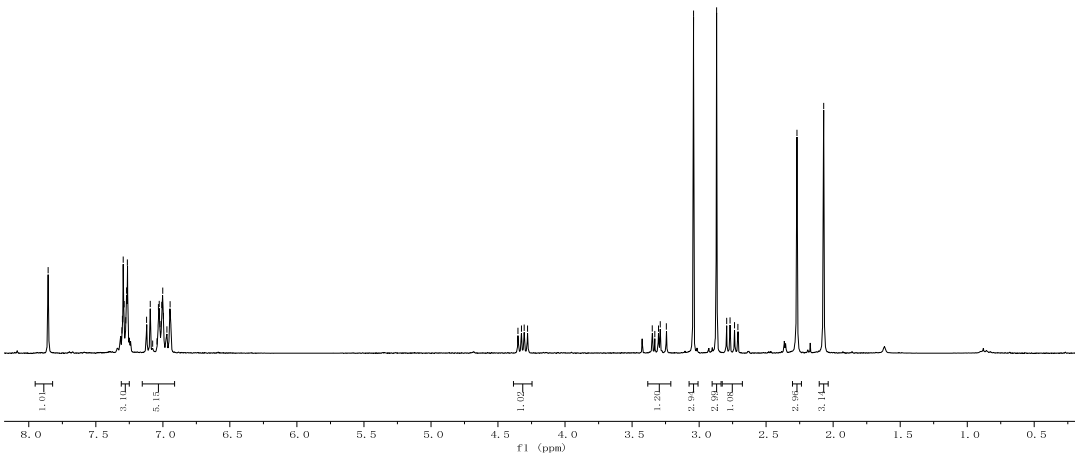
3ae


7.86
7.31
7.30
7.29
7.28
7.27
7.26
7.12
7.10
7.08
7.04
7.03
7.02
7.01
7.00
6.97
6.95

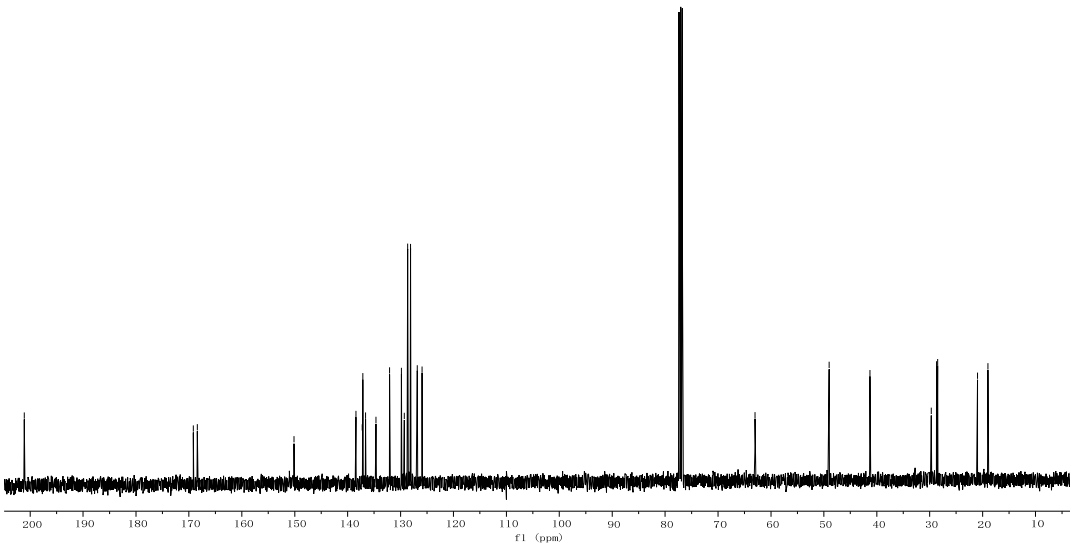

4.35
4.33
4.30
4.28

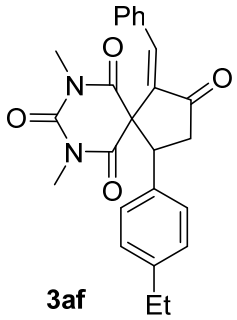

3.35
3.33
3.30
3.29
3.24
3.04
2.87
2.79
2.77
2.74
2.71


2.27
2.07

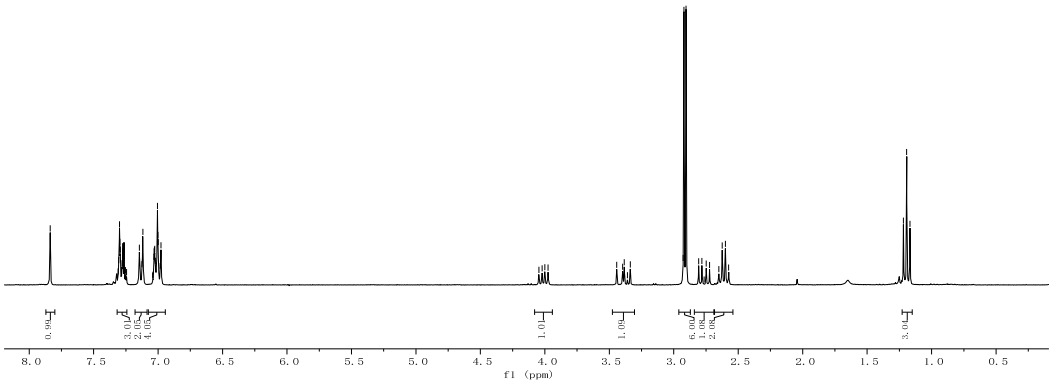

1.01
3.10
5.15
1.02
1.29
2.94
2.99
1.08
2.99
3.14
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

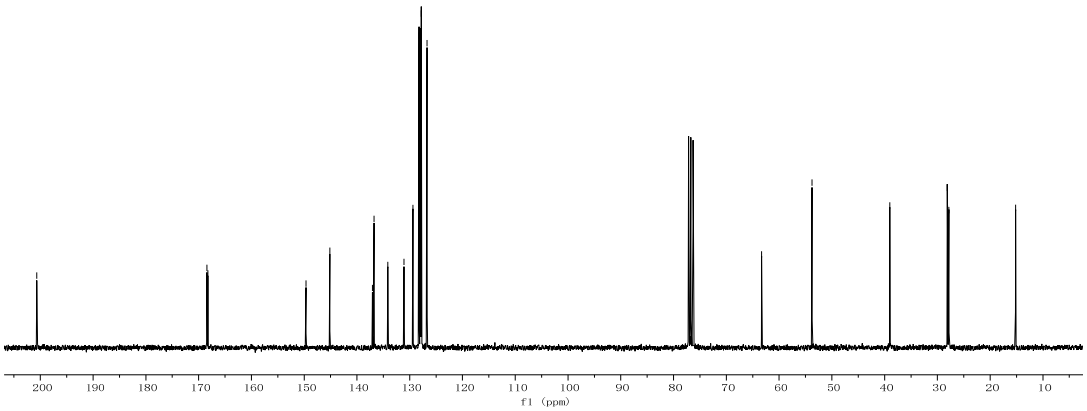

201.11
169.15
168.41
150.13
138.43
137.26
137.12
135.62
134.65
132.98
131.86
129.30
128.65
128.41
126.86
125.92
63.01
49.01
41.30
29.71
28.70
28.48
20.97
18.99

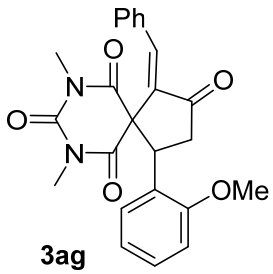

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)



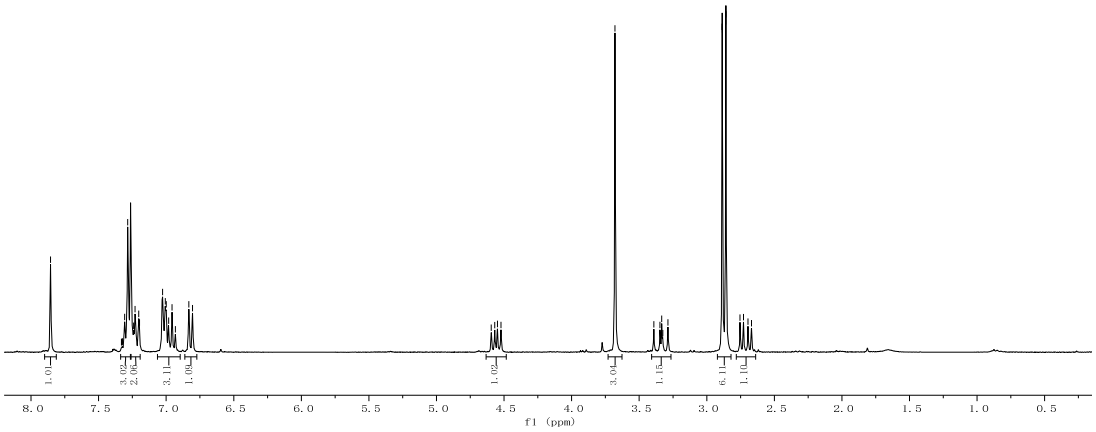
3af

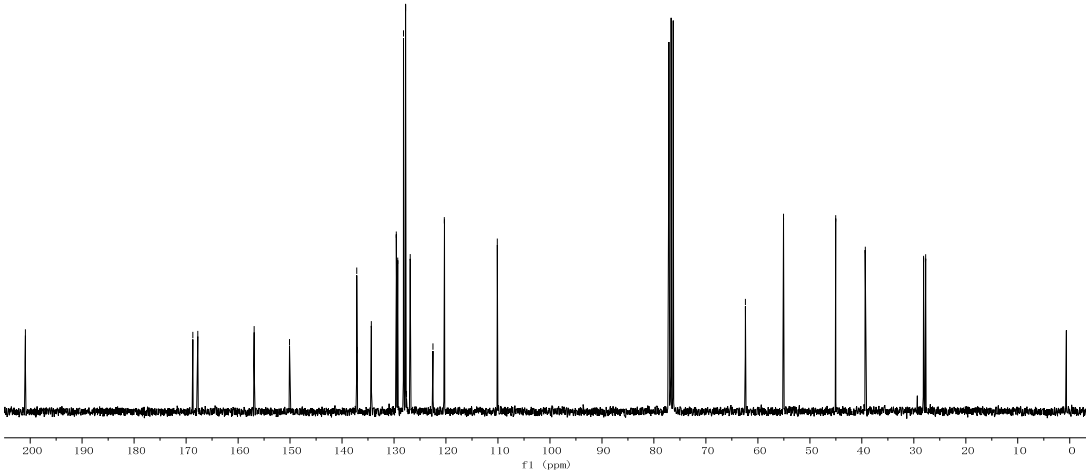


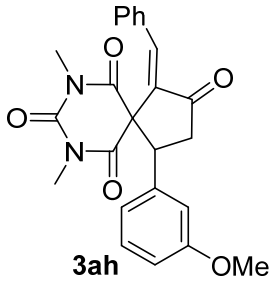


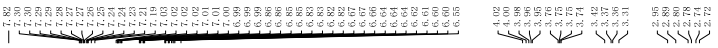

Ph
O
N
O
N
O
O
O
OMe

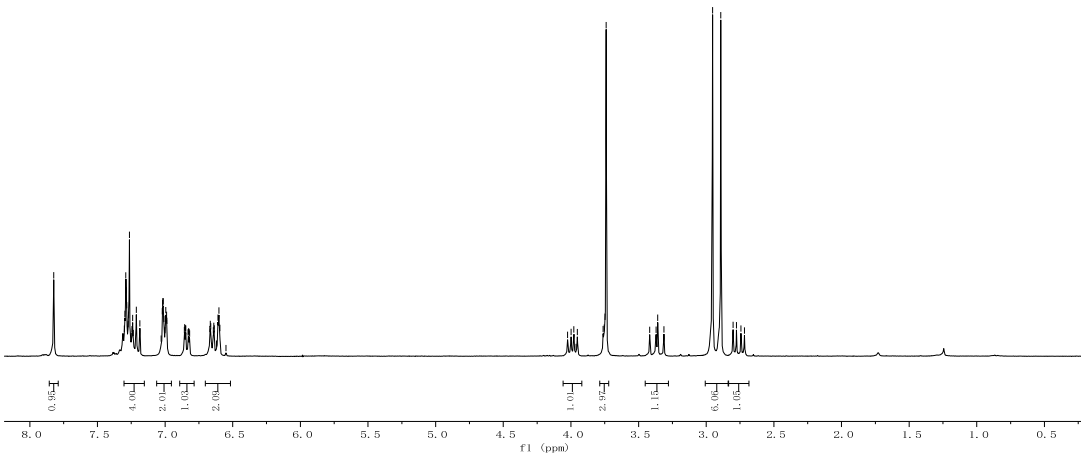
3ag

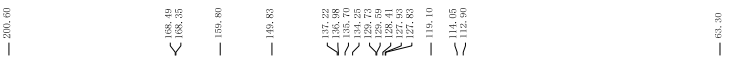

1.01
3.02
2.06
3.11
1.09
1.02
3.04
1.15
6.11
1.10
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

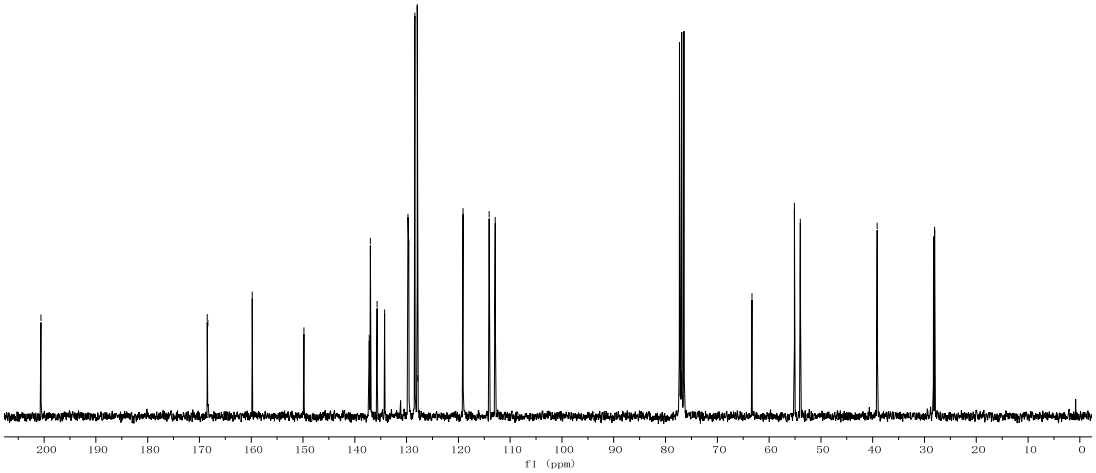

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

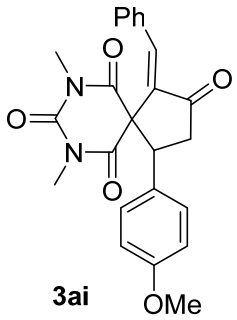

Ph
O
N
O
N
O
O
O
3ah
OMe



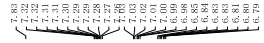

0.95
4.04
2.01
1.03
2.09
1.01
2.92
1.15
6.06
1.05
f1 (ppm)

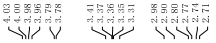


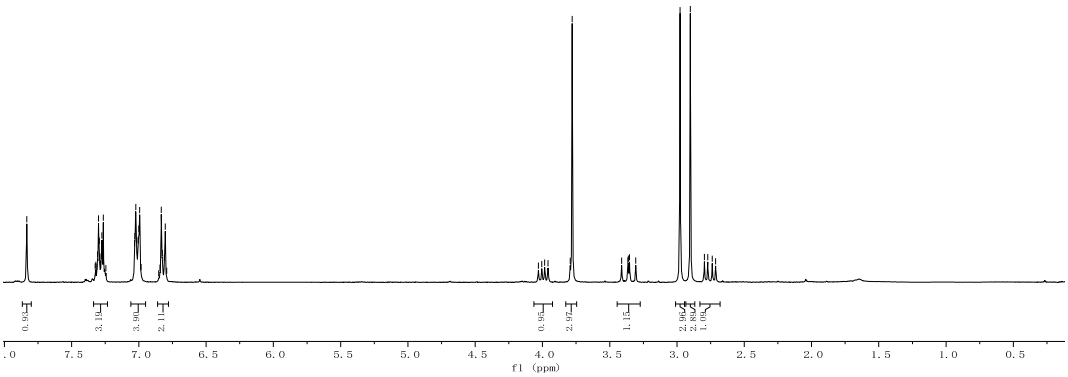

f1 (ppm)

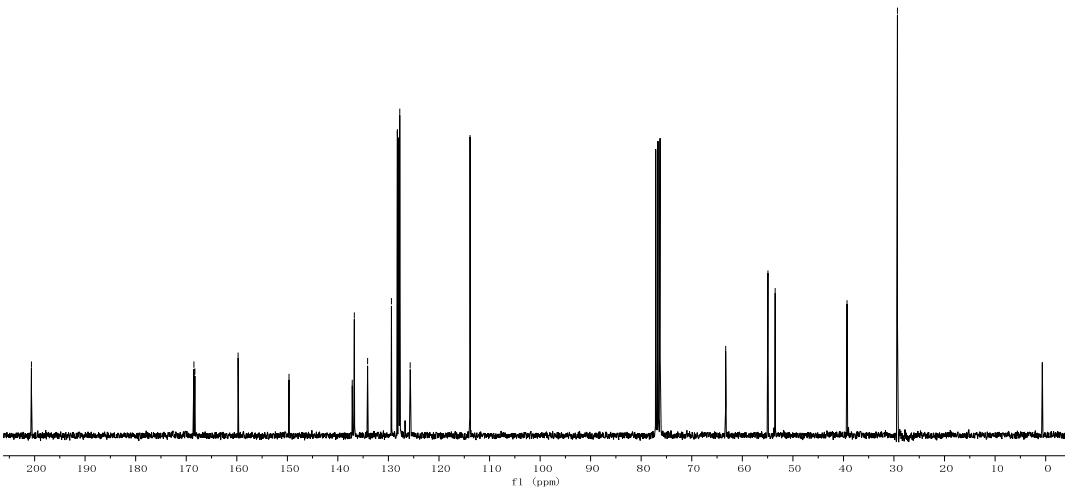


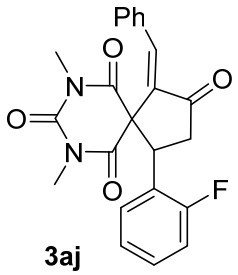
3ai





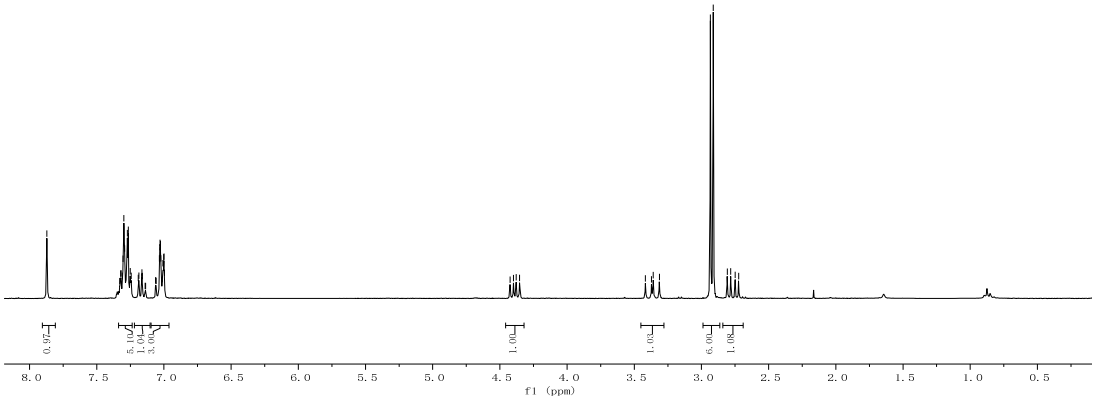




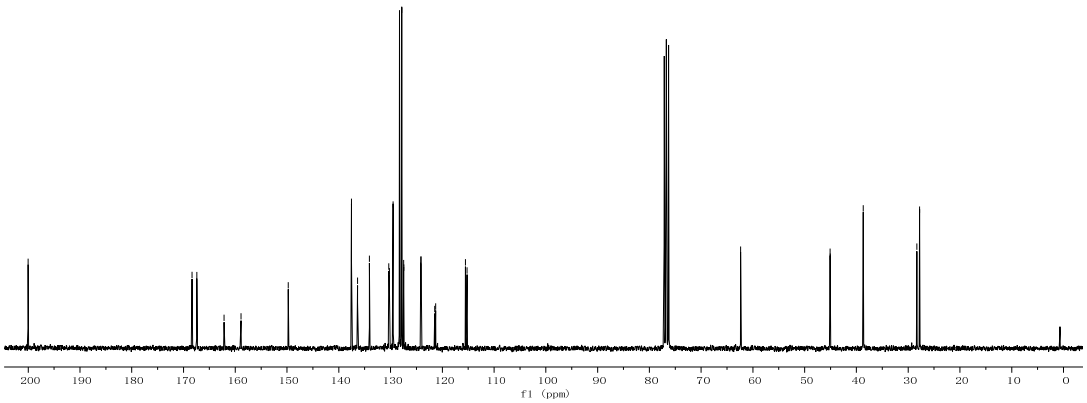

Ph
O
N
O
N
O
O
O
F

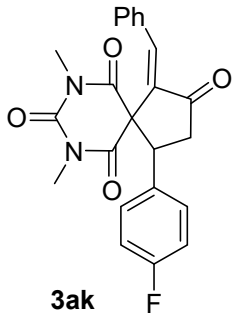
3aj


7.87
7.33
7.32
7.31
7.30
7.29
7.27
7.26
7.25
7.24
7.19
7.18
7.17
7.16
7.16
7.14
7.14
7.06
7.05
7.03
7.03
7.02
7.02
7.01
7.00
7.00
7.00
7.00
4.42
4.40
4.38
4.35
3.42
3.37
3.36
3.31
2.93
2.91
2.81
2.78
2.75
2.72

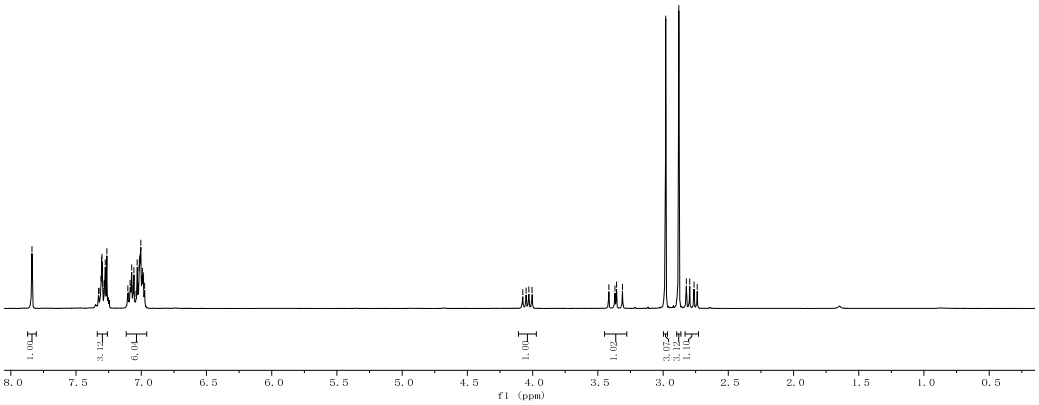

0.97
5.10
1.04
3.00
1.00
1.03
6.00
1.08
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

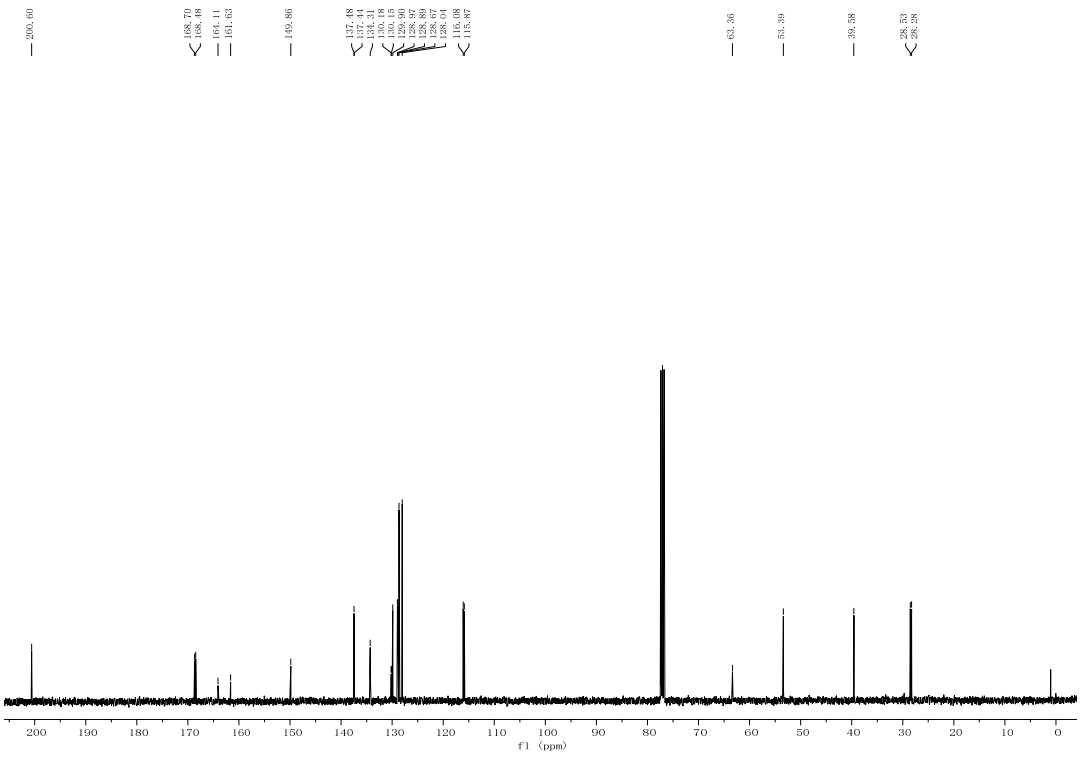

200.01
168.54
167.42
162.16
158.89
149.76
137.54
135.39
134.08
130.35
130.24
129.71
128.28
127.82
127.48
127.43
124.15
124.10
121.47
121.29
115.52
115.23
62.36
45.10
45.07
38.70
28.34
27.81

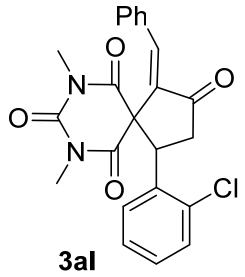

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



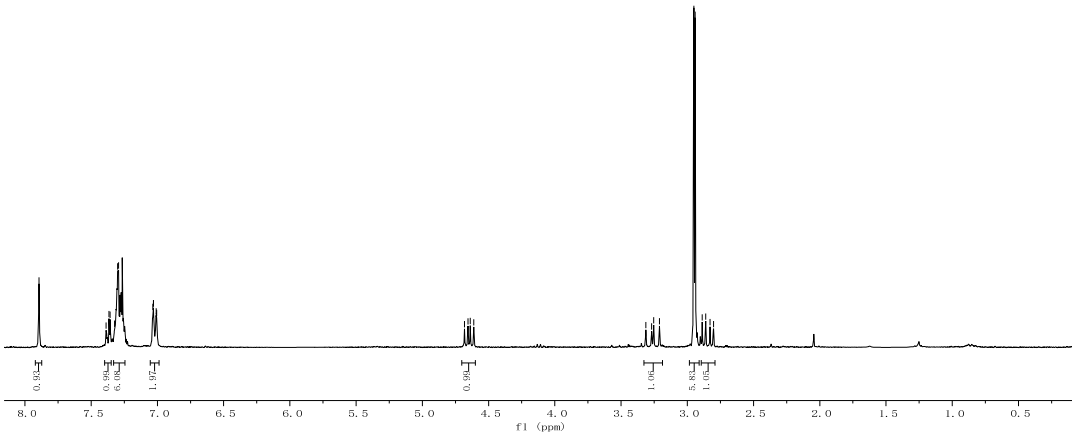
3ak

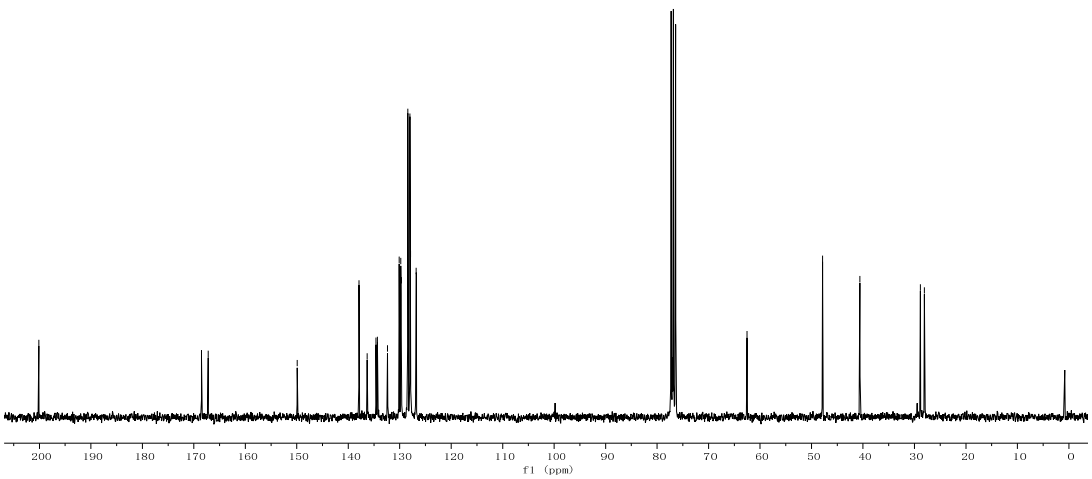


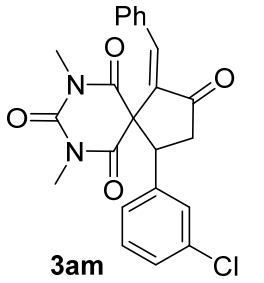




3al



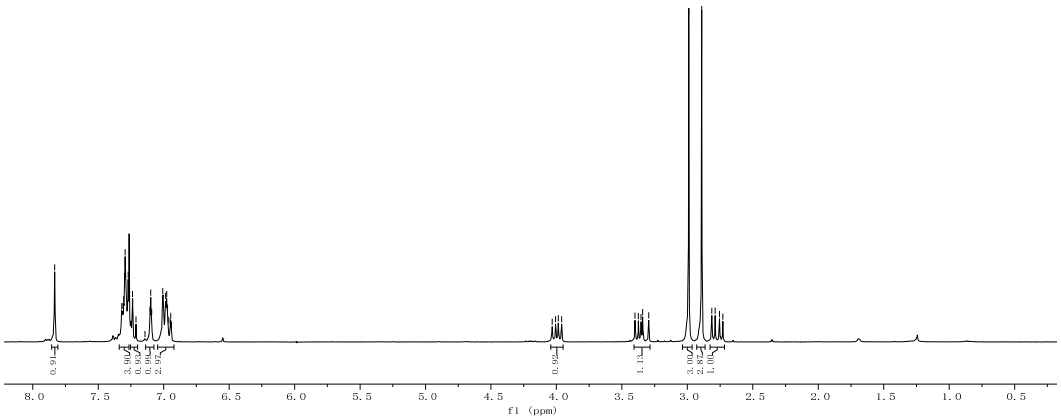



Ph
O
N
O
N
O
O
O
Cl

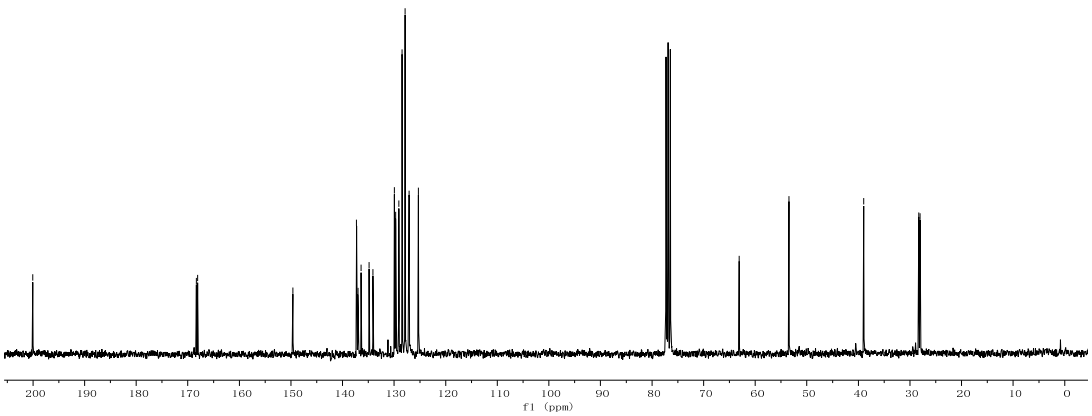
3am

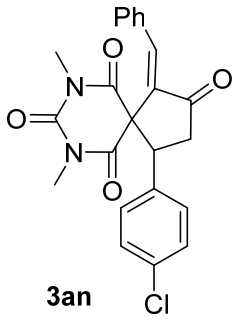

7.83
7.32
7.31
7.31
7.30
7.29
7.27
7.24
7.21
7.14
7.10
7.10
7.09
7.01
6.99
6.98
6.97
6.97
6.95
6.95
6.94


4.03
4.01
3.98
3.96
3.40
3.37
3.35
3.34
3.30
2.99
2.88
2.81
2.79
2.76
2.73

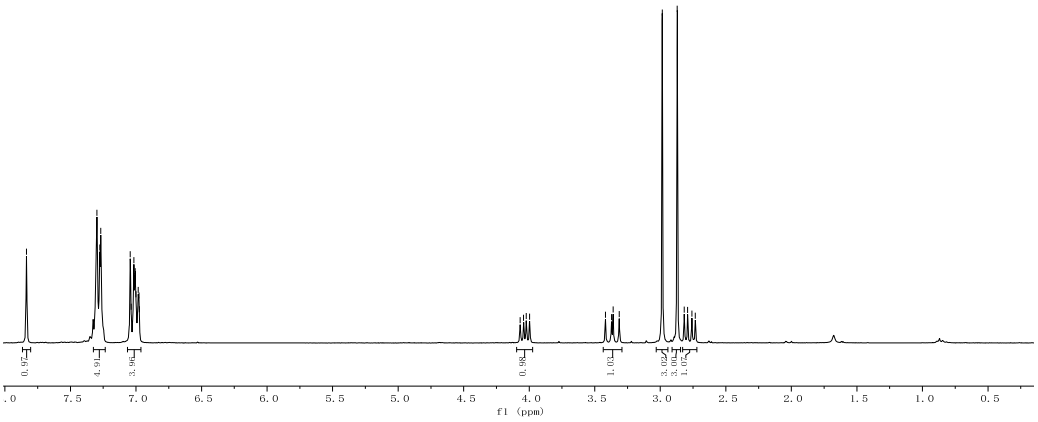

0.91
3.90
0.93
0.99
2.97
0.92
1.13
3.02
2.82
1.00
f1 (ppm)

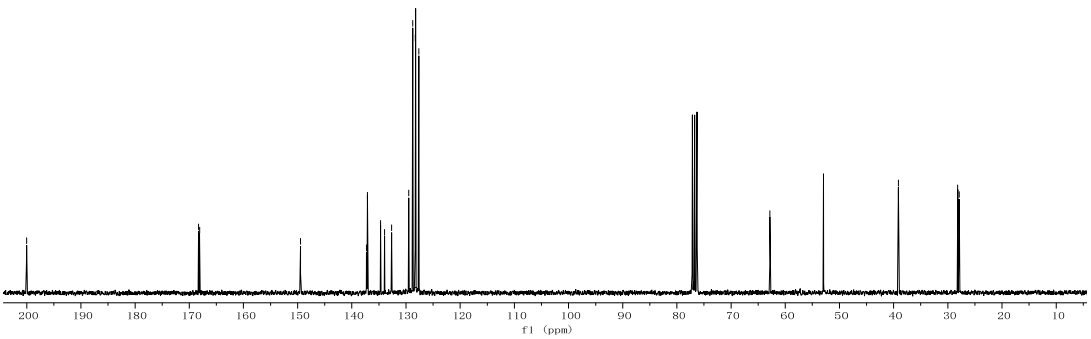

200.05
168.53
168.10
149.62
137.29
137.01
136.40
134.85
134.11
129.95
129.69
129.08
128.46
127.87
127.11
125.29
63.11
53.46
38.97
28.30
28.06

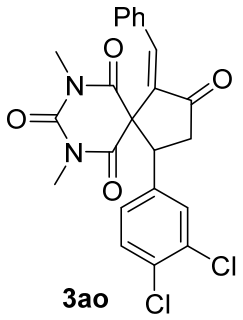

f1 (ppm)



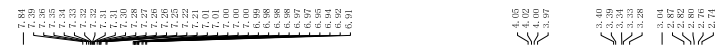
3an

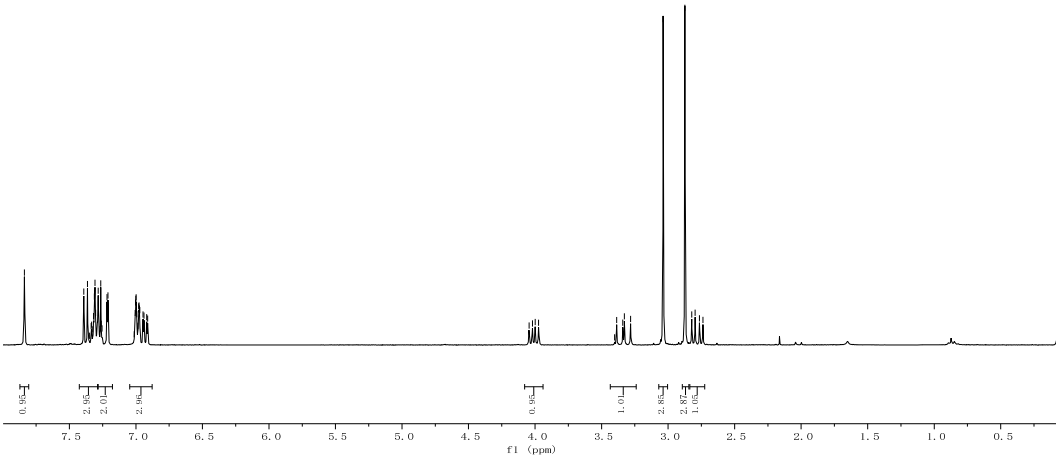


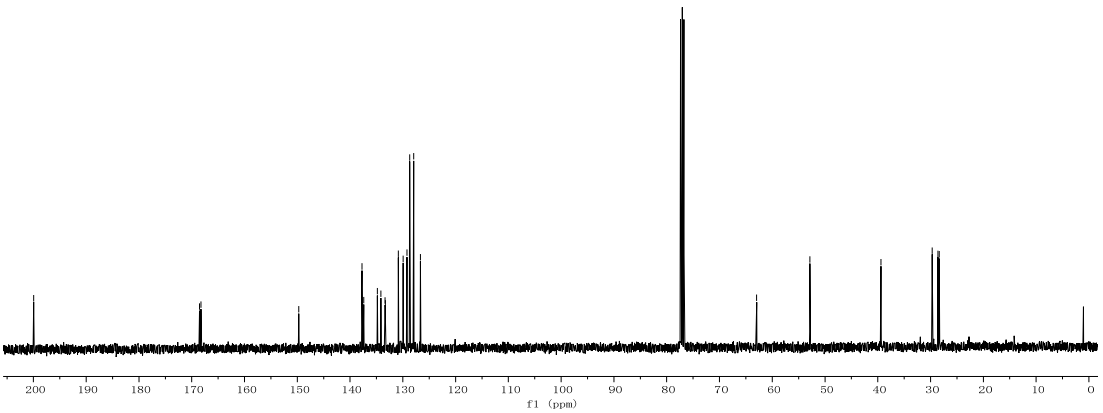


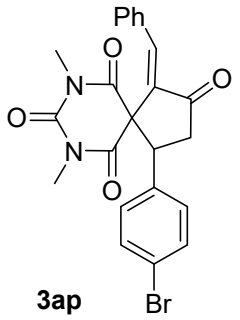


3ao

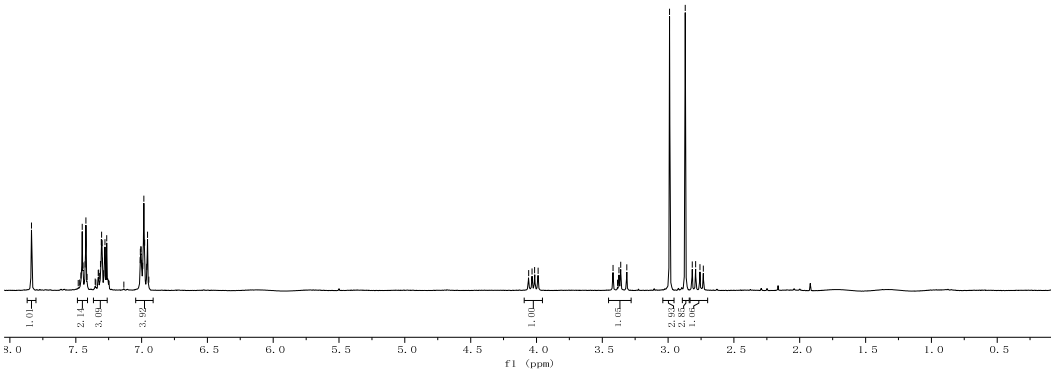


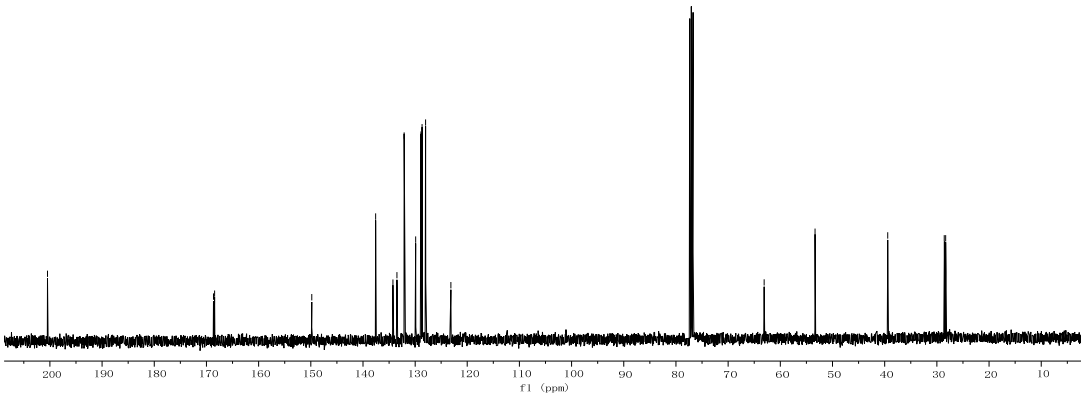


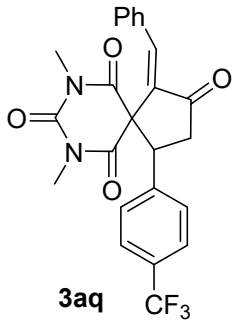




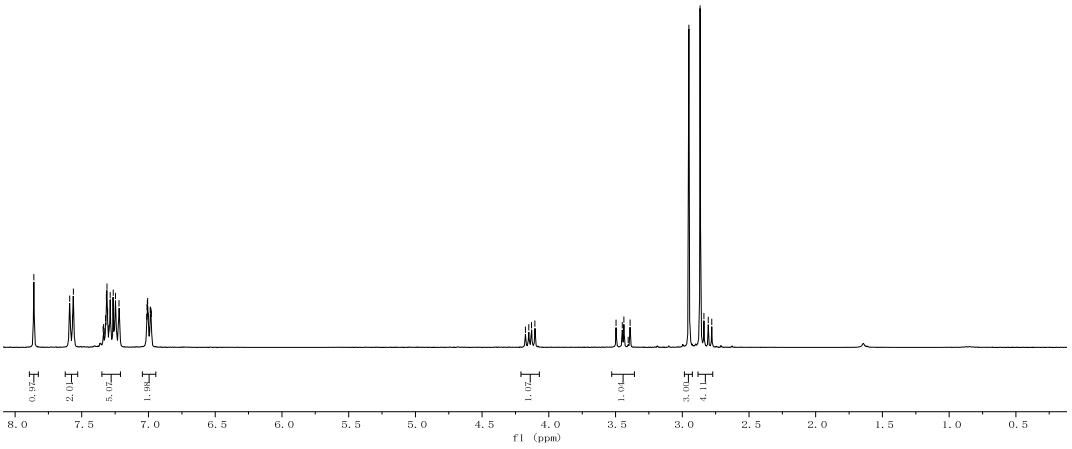
3ap



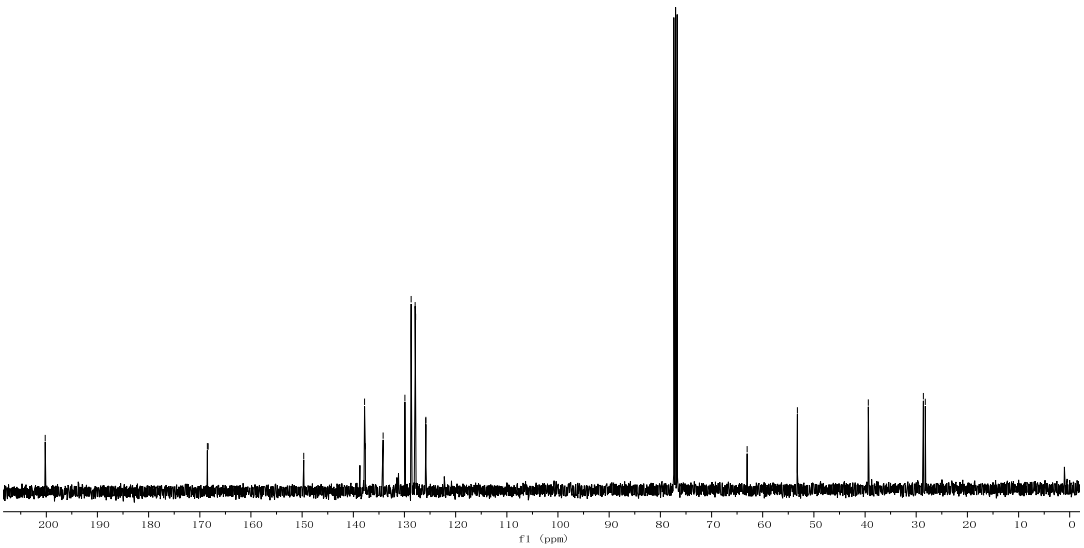


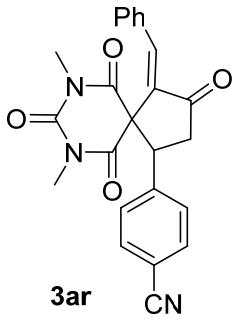

Ph
O
N
O
O
N
O
O
3aq
CF3

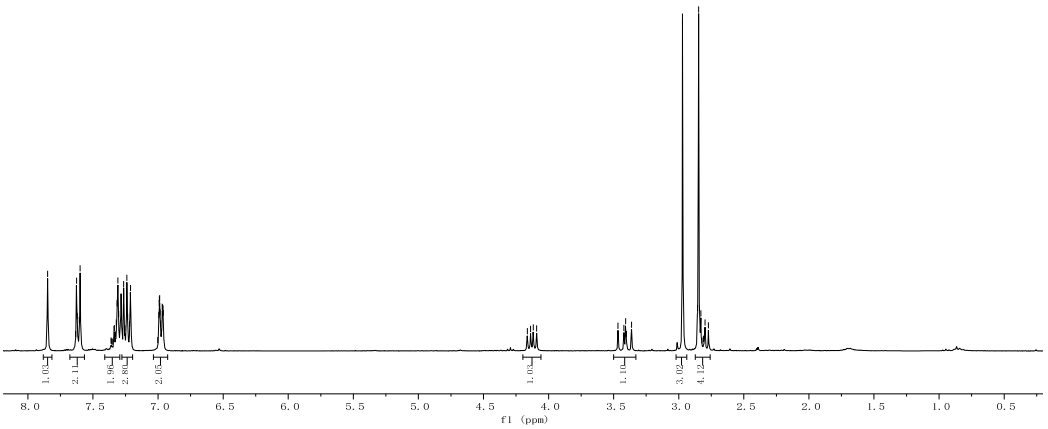

7.86
7.59
7.57
7.56
7.34
7.33
7.32
7.31
7.30
7.29
7.26
7.25
7.22
7.01
7.01
7.00
6.99
6.98
6.98
6.98
4.18
4.15
4.13
4.10
3.50
3.45
3.44
3.41
2.39
2.95
2.87
2.84
2.81
2.78

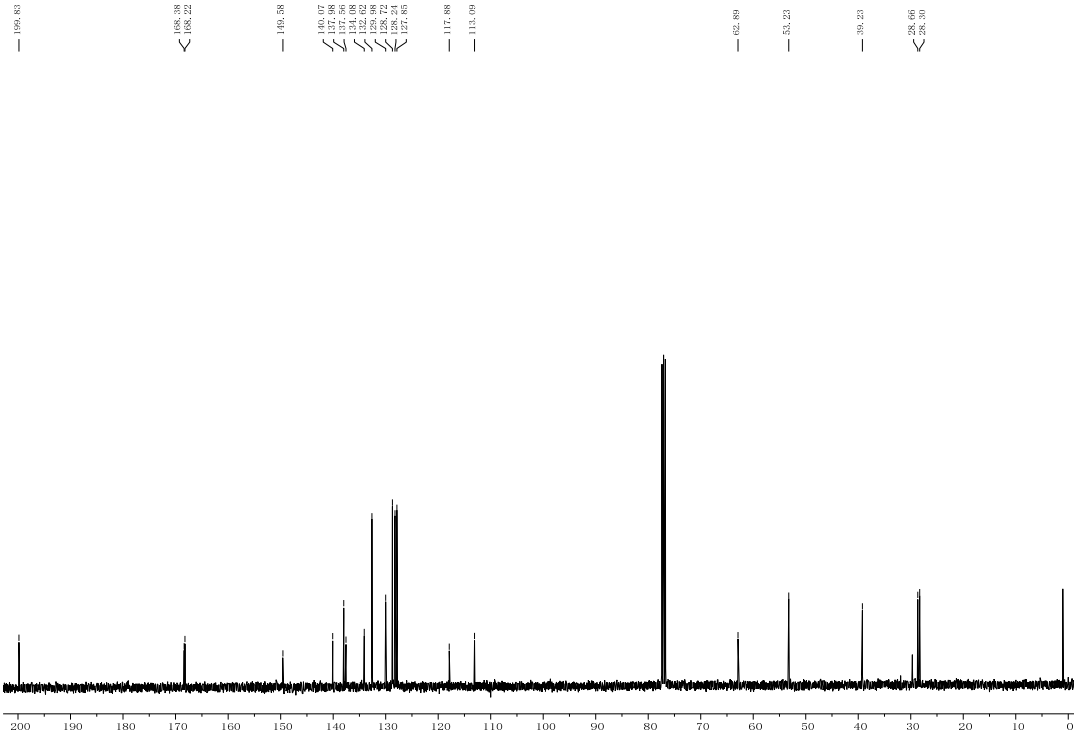

0.97
2.01
5.07
1.98
1.07
1.04
3.00
4.11
f1 (ppm)

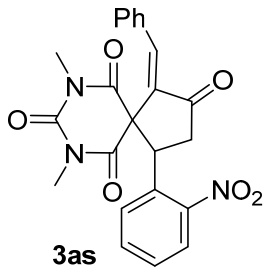

200.20
168.53
168.38
149.68
137.80
137.65
134.19
129.59
128.69
127.91
127.82
125.86
125.82
63.04
53.23
39.36
28.61
28.23


f1 (ppm)

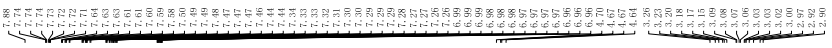

Ph
O
N
O
N
O
O
O
3ar
CN

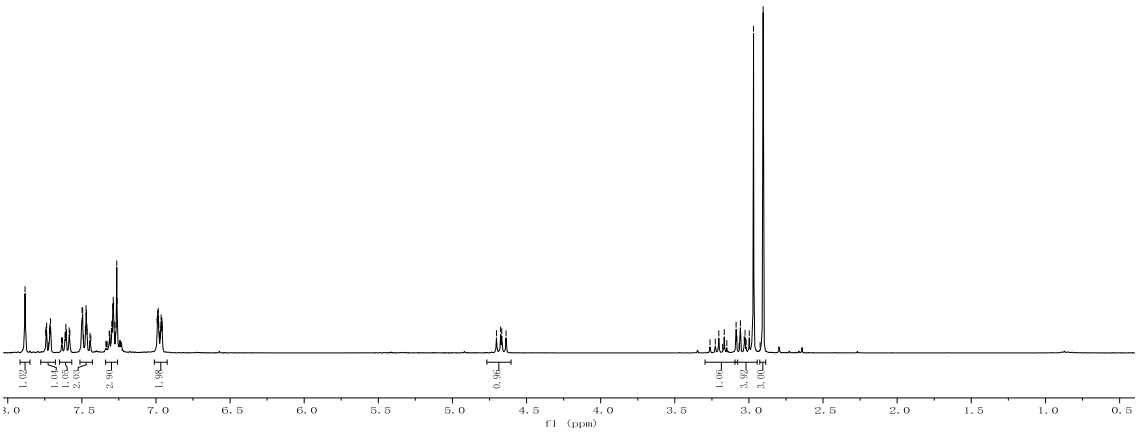

1.03
2.14
1.96
2.80
2.05
1.03
1.10
3.05
4.13
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

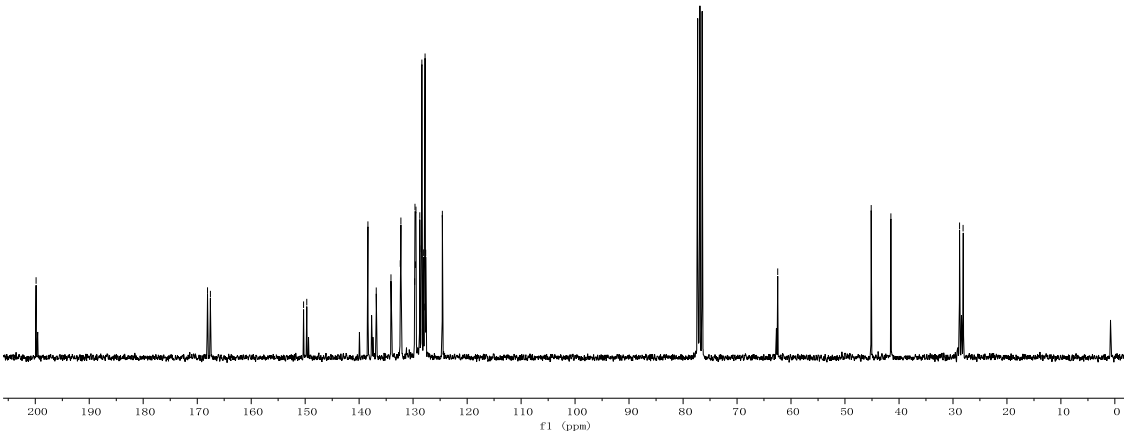

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

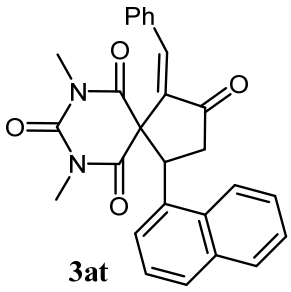

Ph
O
N
O
O
N
O
O
NO2

3as

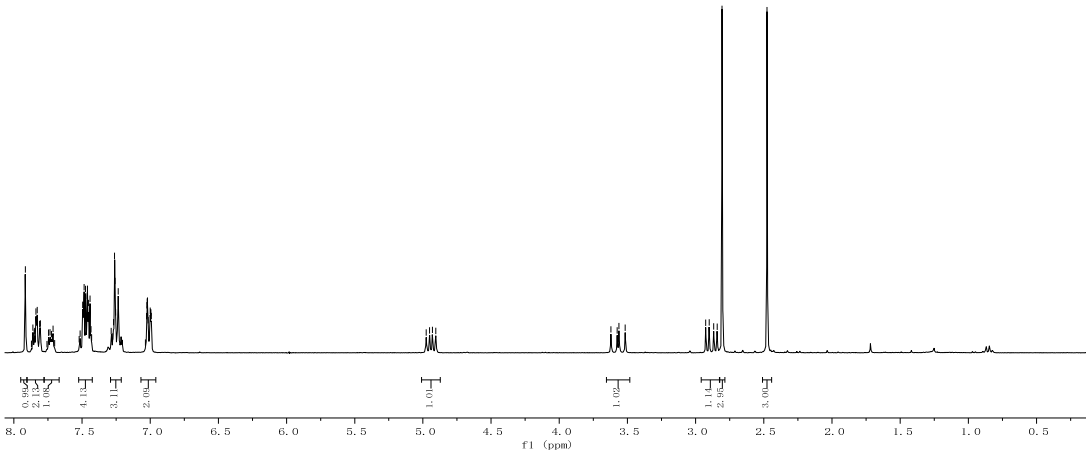


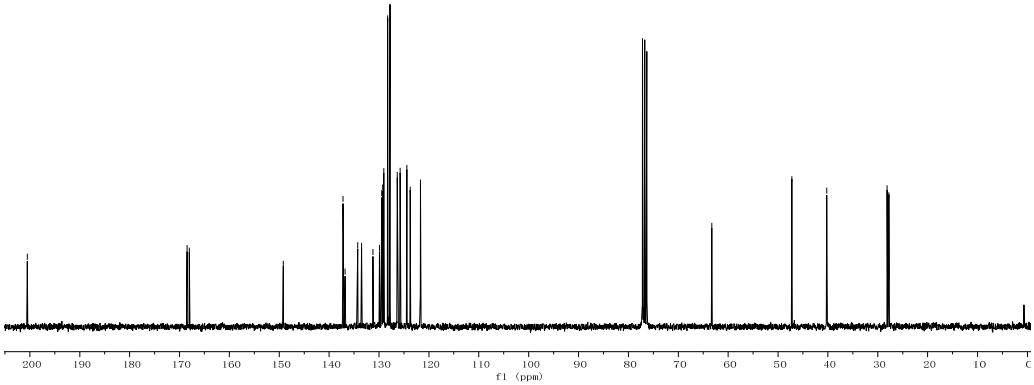

1.02
1.04
1.05
2.03
2.90
1.98
0.96
1.06
3.92
3.00
f1 (ppm)

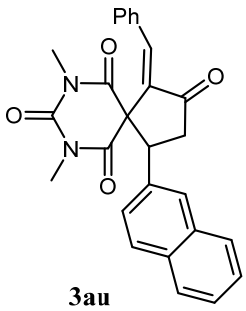

f1 (ppm)



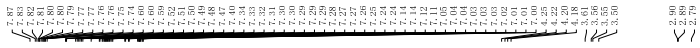
3at

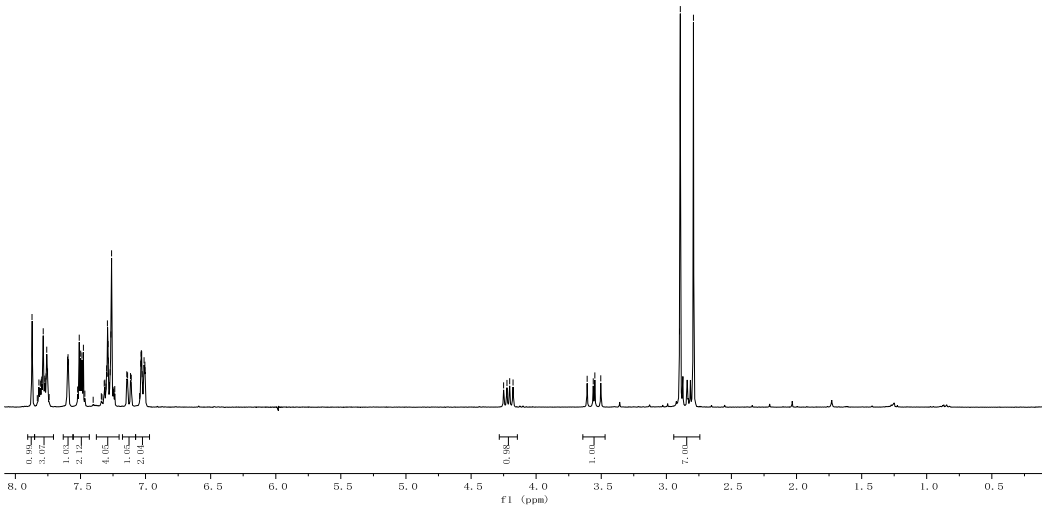


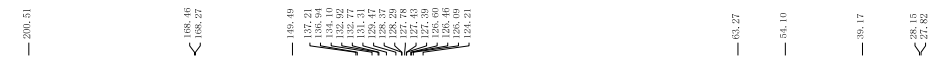


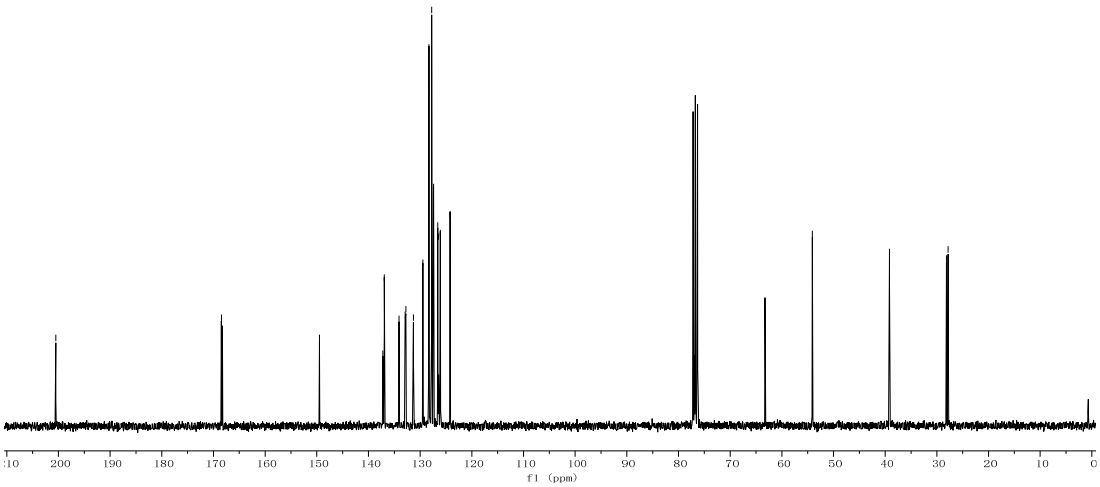


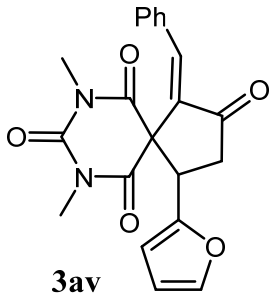
3au



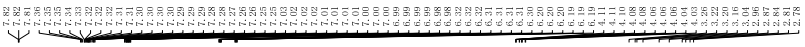


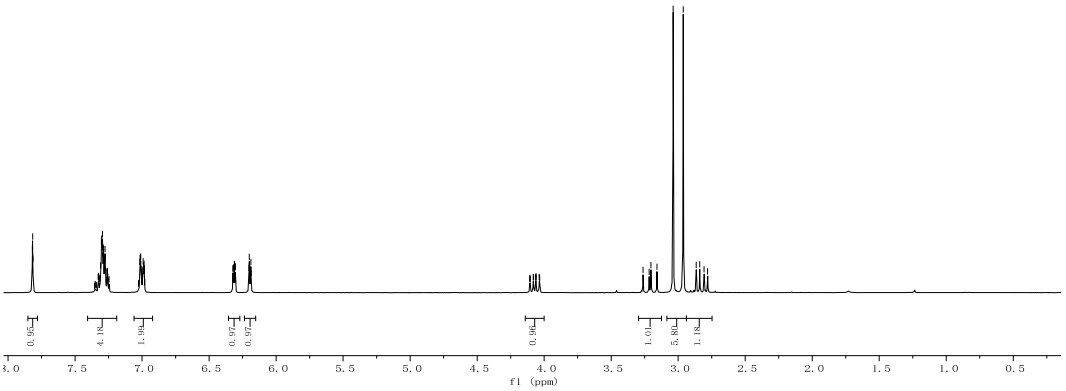


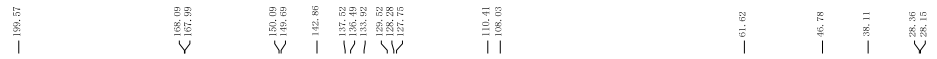


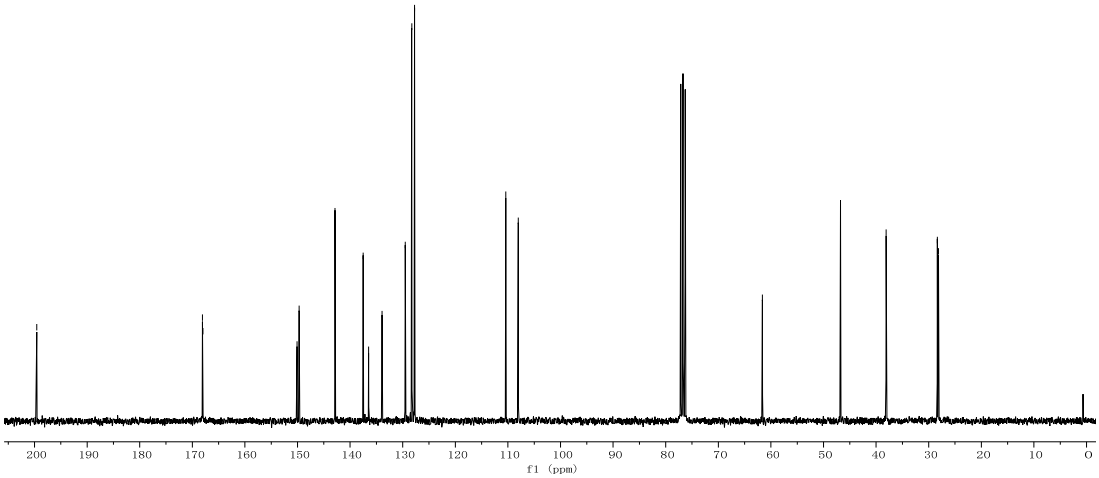

Ph
O
N
O
N
O
O
O

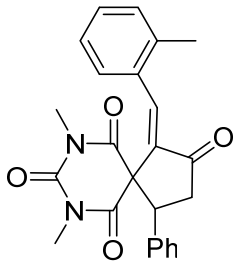
3av



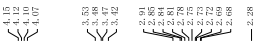

0.95
4.18
1.99
0.97
0.97
0.96
1.01
5.80
1.18
f1 (ppm)

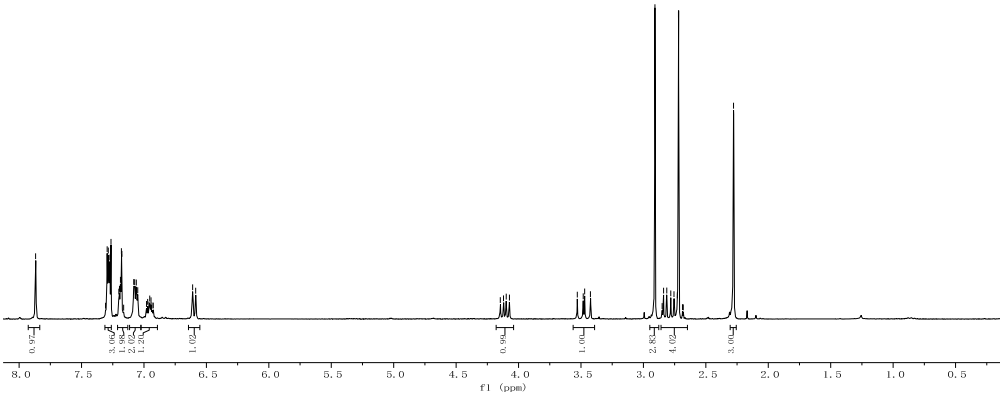

199.57
168.09
167.99
150.09
149.69
142.86
137.52
136.49
133.92
129.52
128.28
127.75
110.41
108.03
61.62
46.78
38.11
28.36
28.15

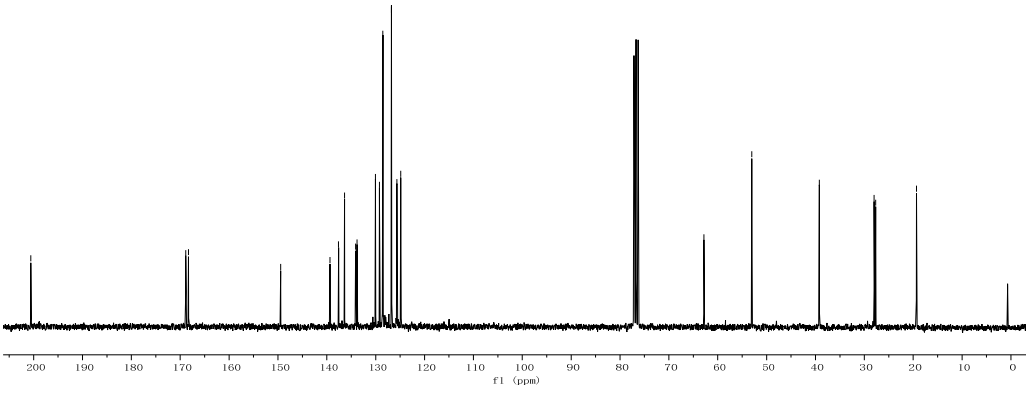

f1 (ppm)

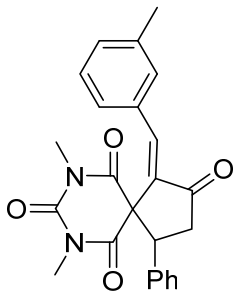


3ba

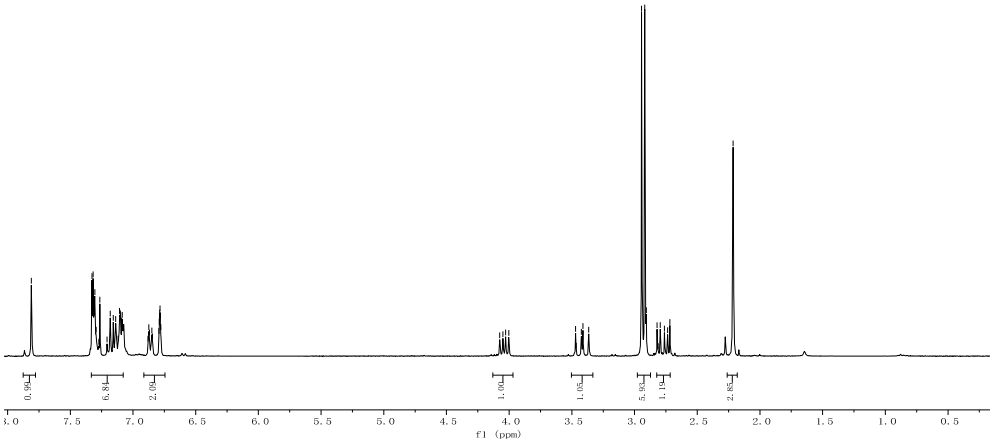


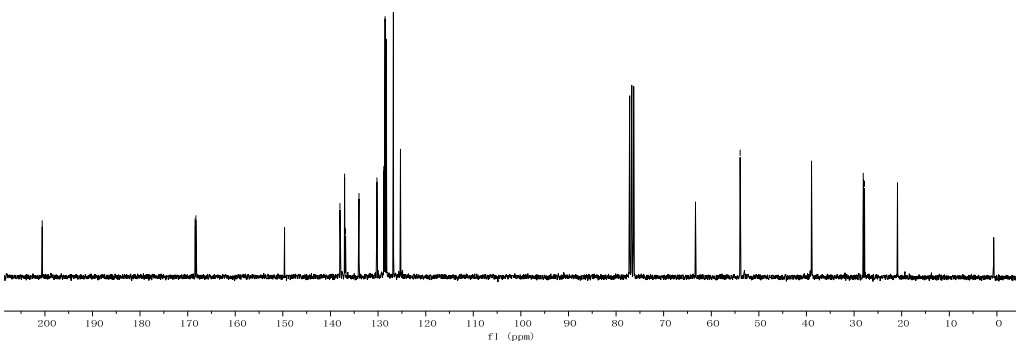


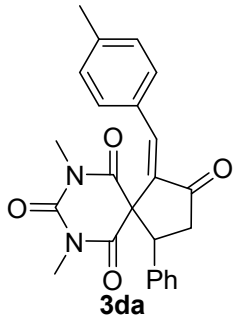




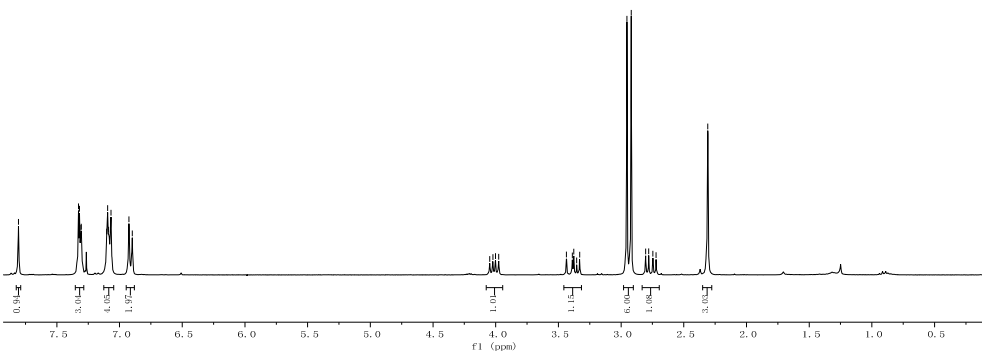
3ca

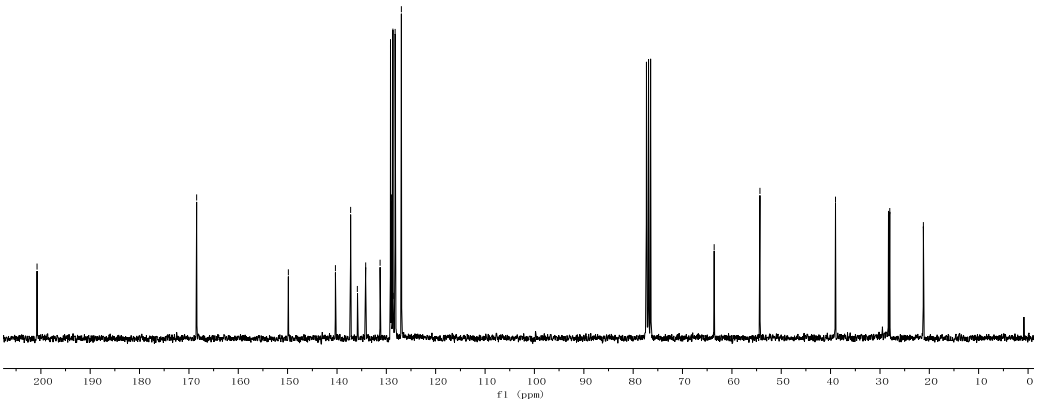


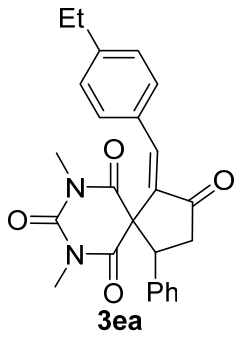



O
N
O
O
N
O
O
Ph

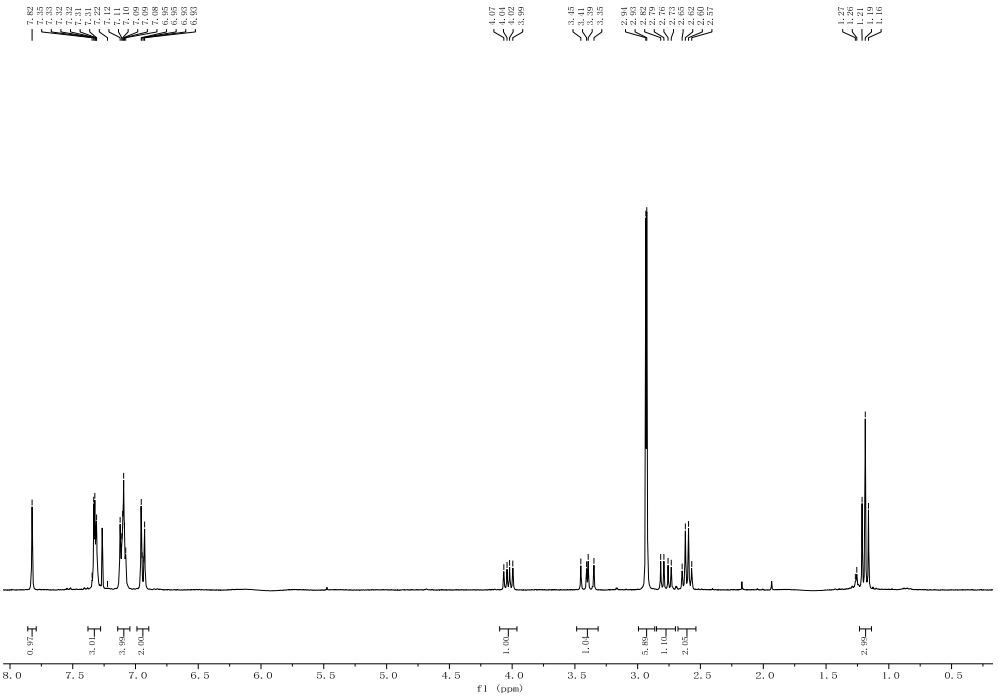
3da

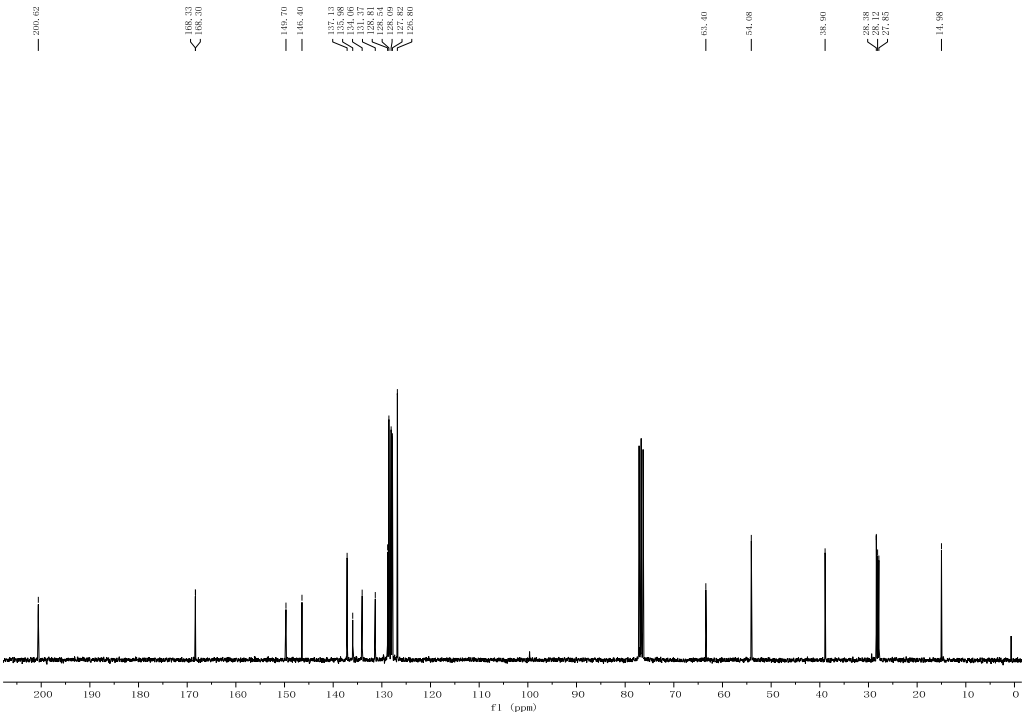

0.94
3.04
4.05
1.97
1.01
1.15
6.00
1.08
3.03
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

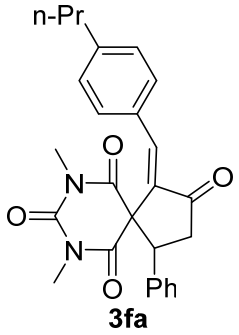

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



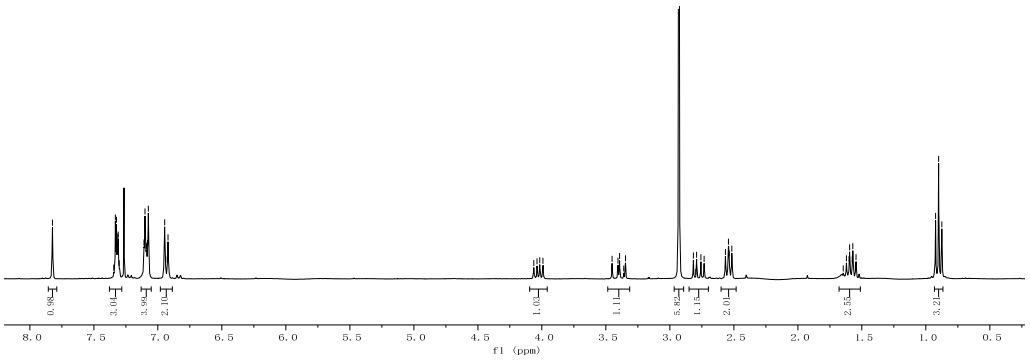
3ea

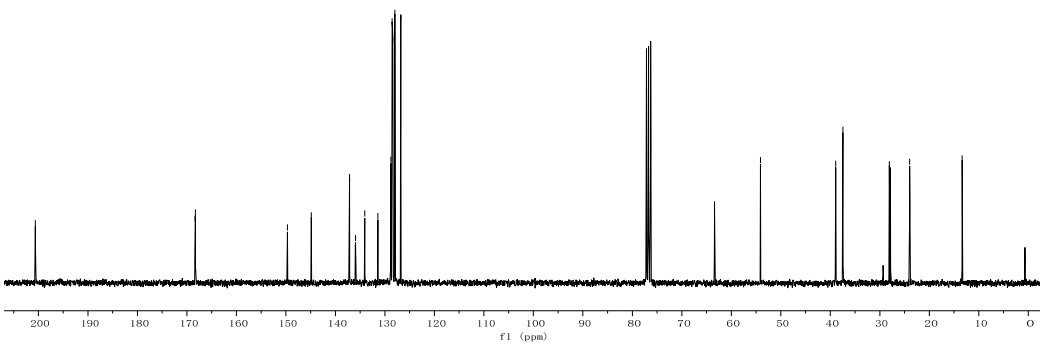


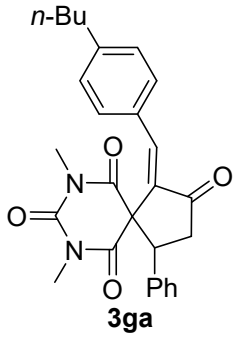



n-Pr
O
N
O
O
N
O
O
Ph

3fa

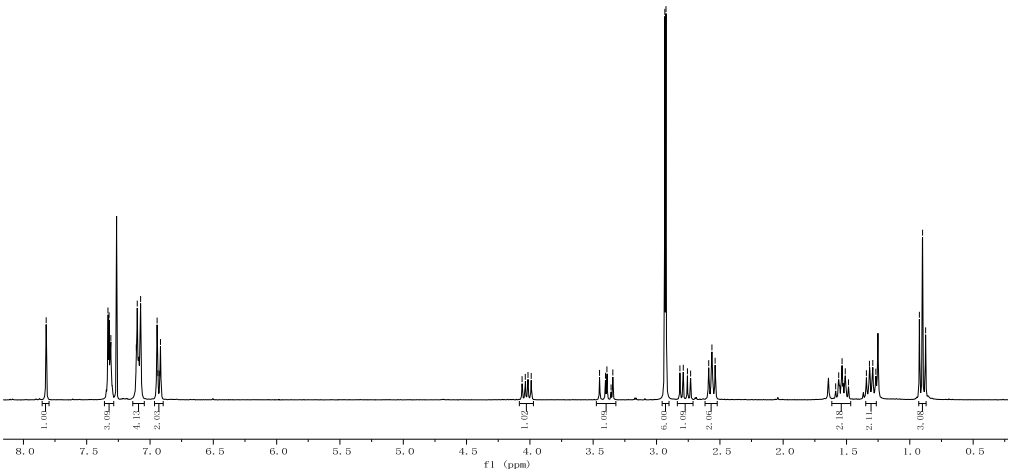

0.98
3.04
3.99
2.10
1.03
1.11
5.82
1.15
2.01
2.55
3.21
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


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

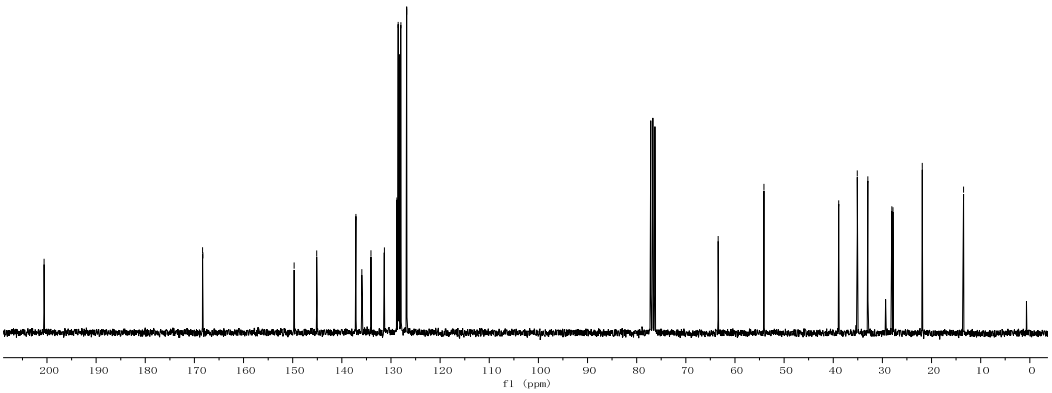

n-Bu
O
N
O
N
O
O
O
Ph

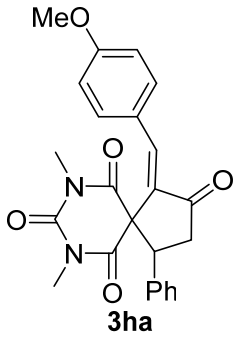
3ga


7.82
7.33
7.32
7.31
7.11
7.10
7.07
6.94
6.94
6.92
6.92
4.06
4.04
4.02
3.99
3.45
3.40
3.39
3.38
3.34
2.93
2.92
2.81
2.79
2.75
2.73
2.59
2.56
2.54
1.59
1.56
1.54
1.51
1.48
1.34
1.32
1.29
1.27
0.92
0.90
0.88

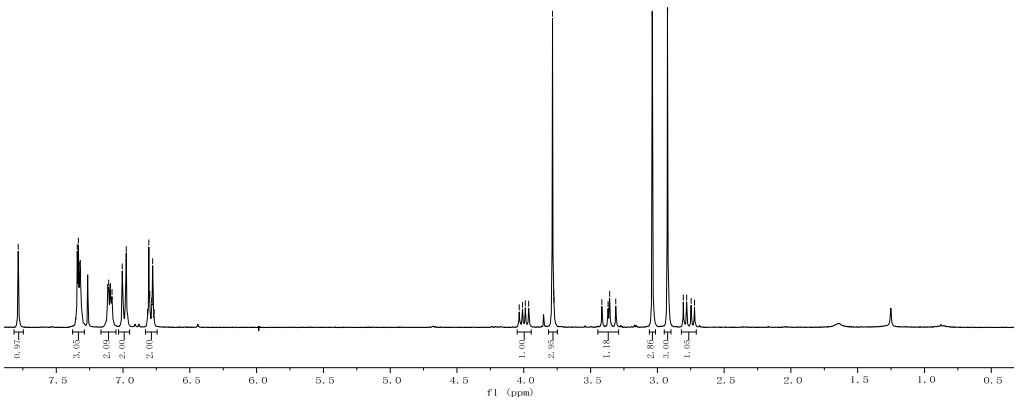

1.00
3.00
4.13
2.00
1.00
1.00
6.00
1.00
2.00
2.18
2.11
3.08
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

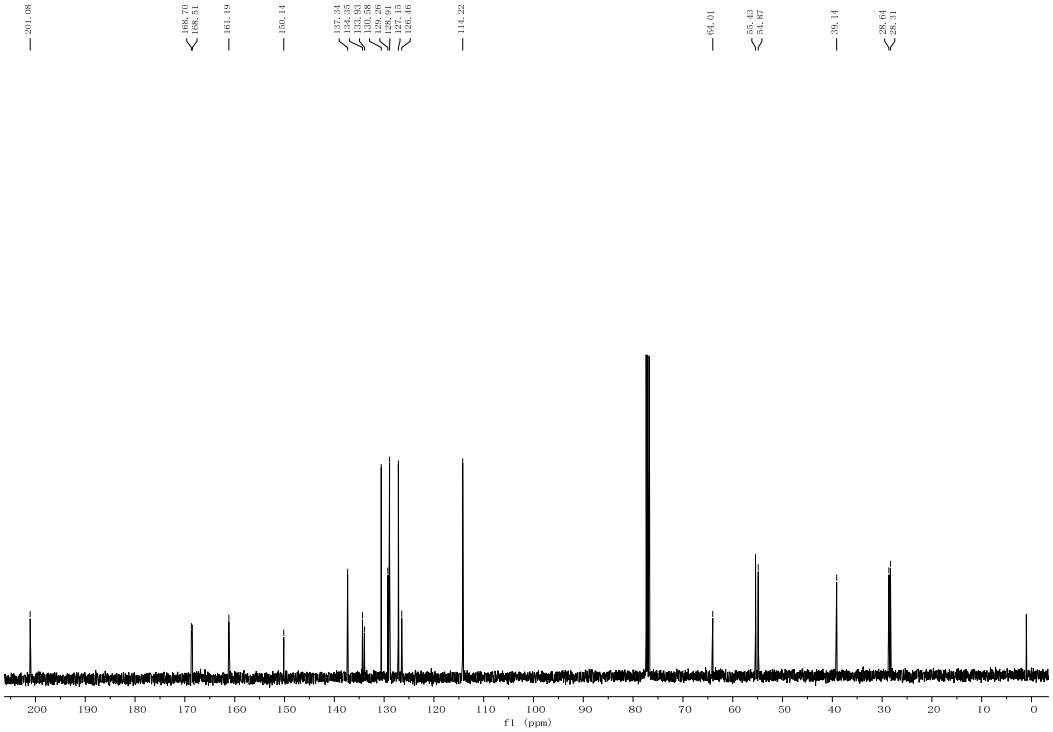

200.60
168.33
168.30
149.70
145.11
137.44
135.55
134.07
131.35
129.80
128.53
128.36
128.00
126.80
63.41
54.11
38.90
35.09
32.96
28.07
27.83
21.89
13.49

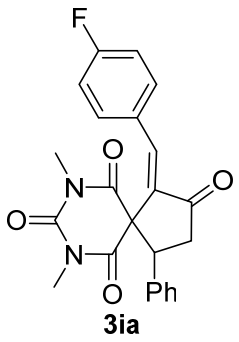

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



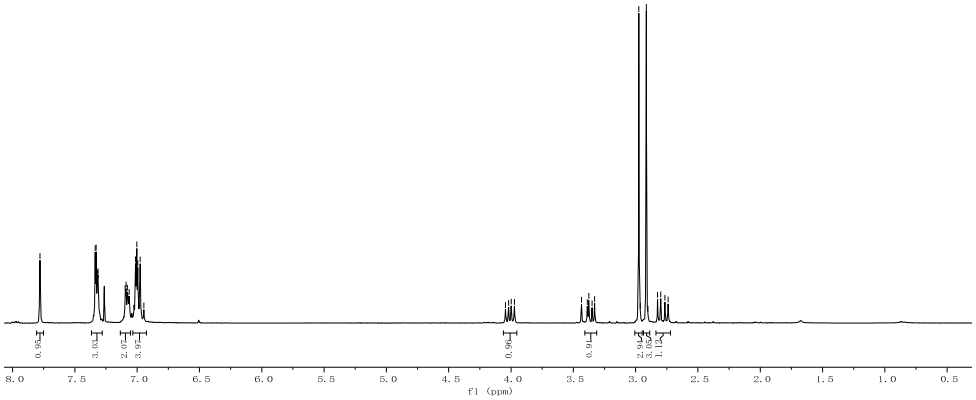
3ha

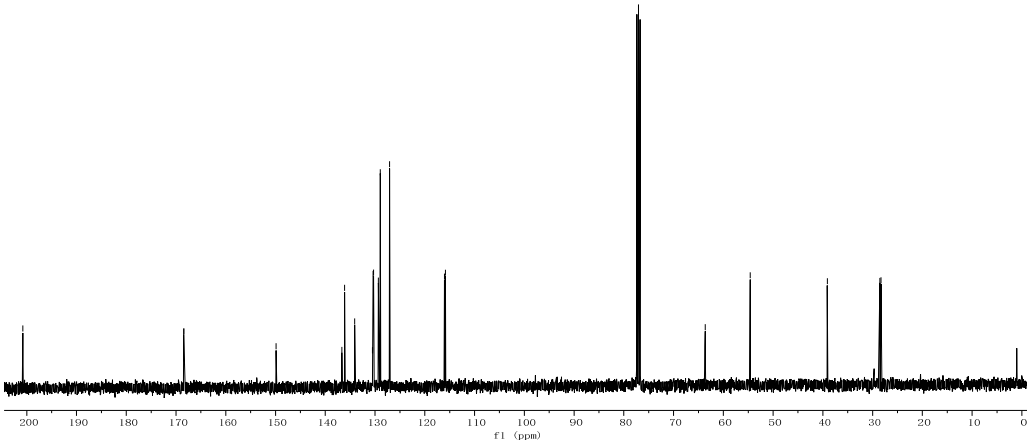


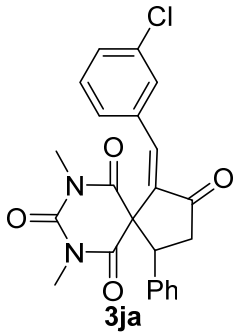



F
O
N
O
N
O
O
O
Ph

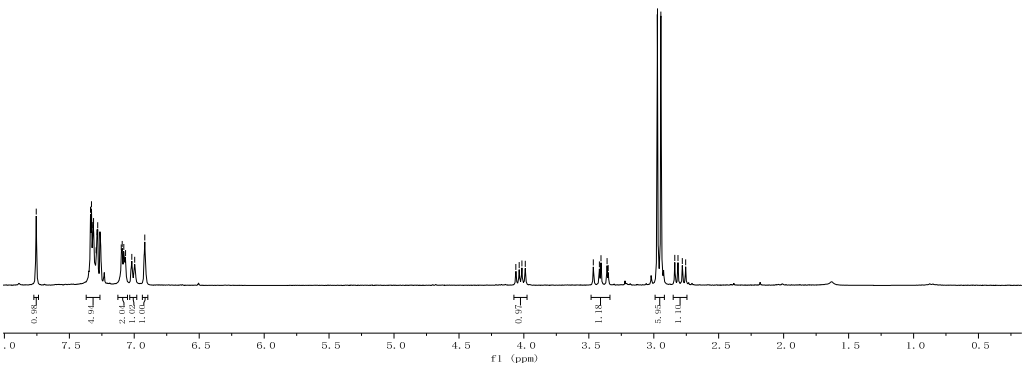
3ia

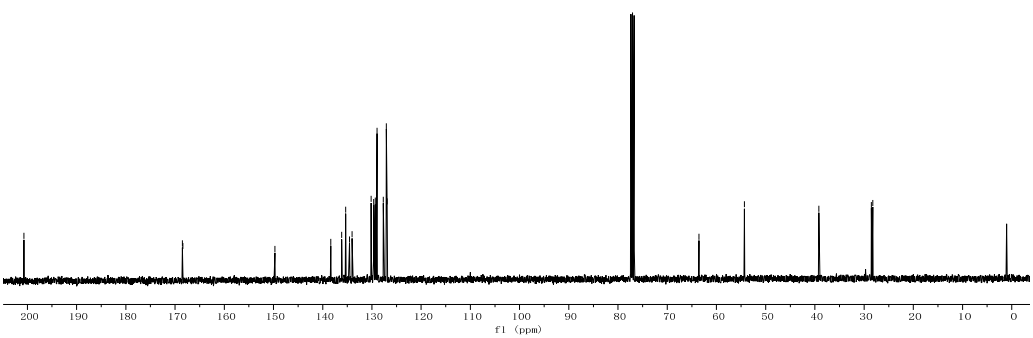

0.98
3.04
2.07
3.97
0.96
0.94
2.94
3.05
1.15
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

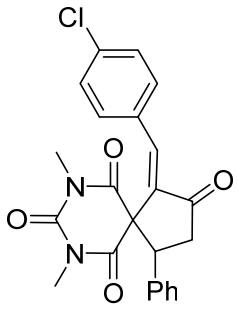

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



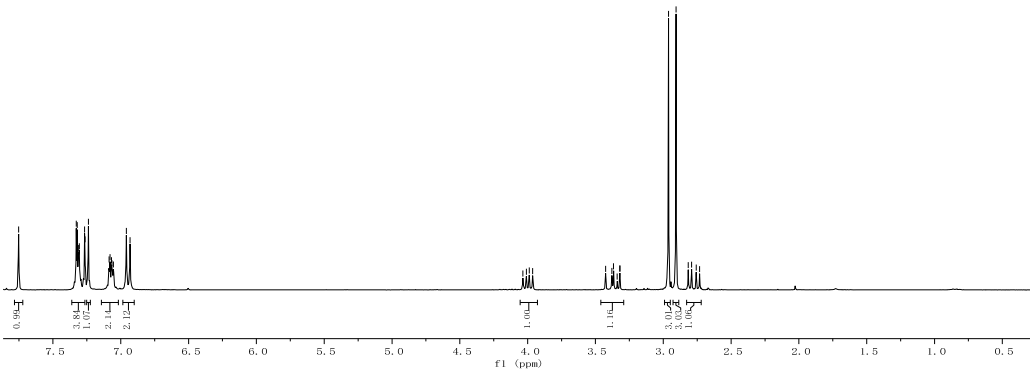
3ja

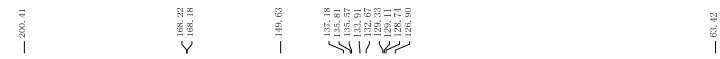


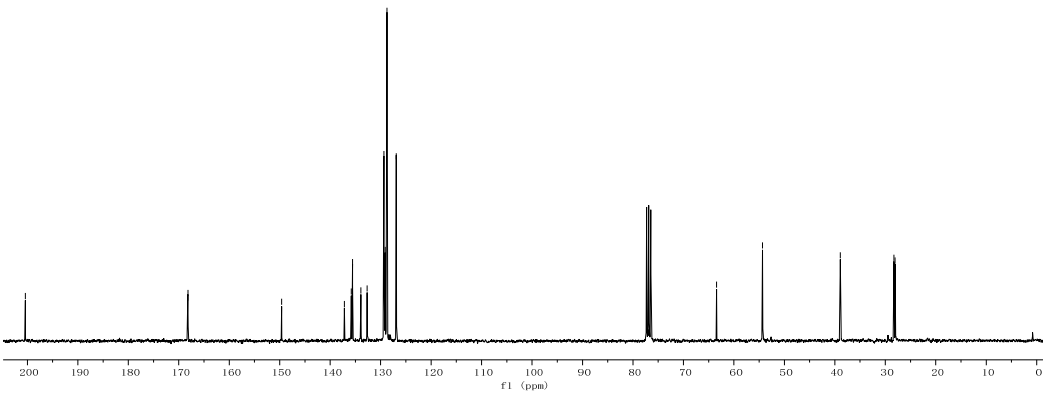


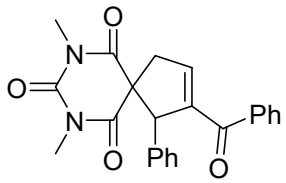


3ka

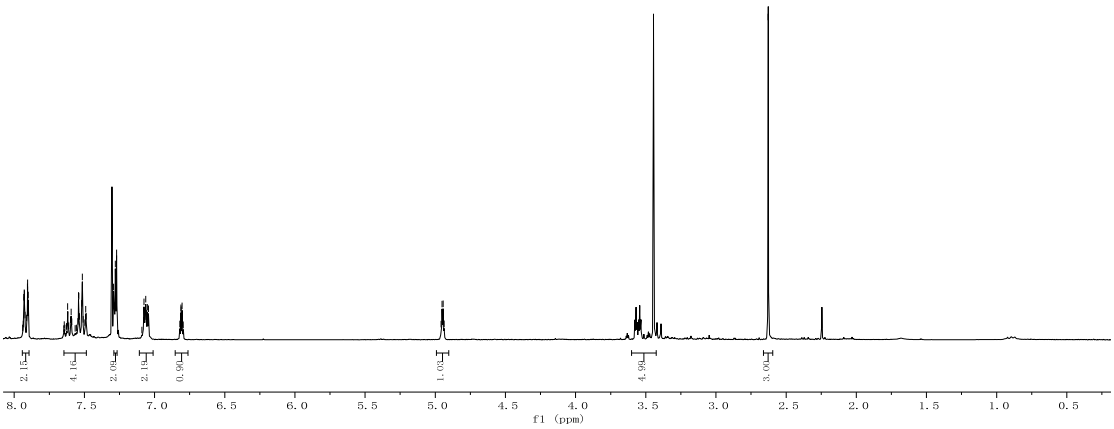


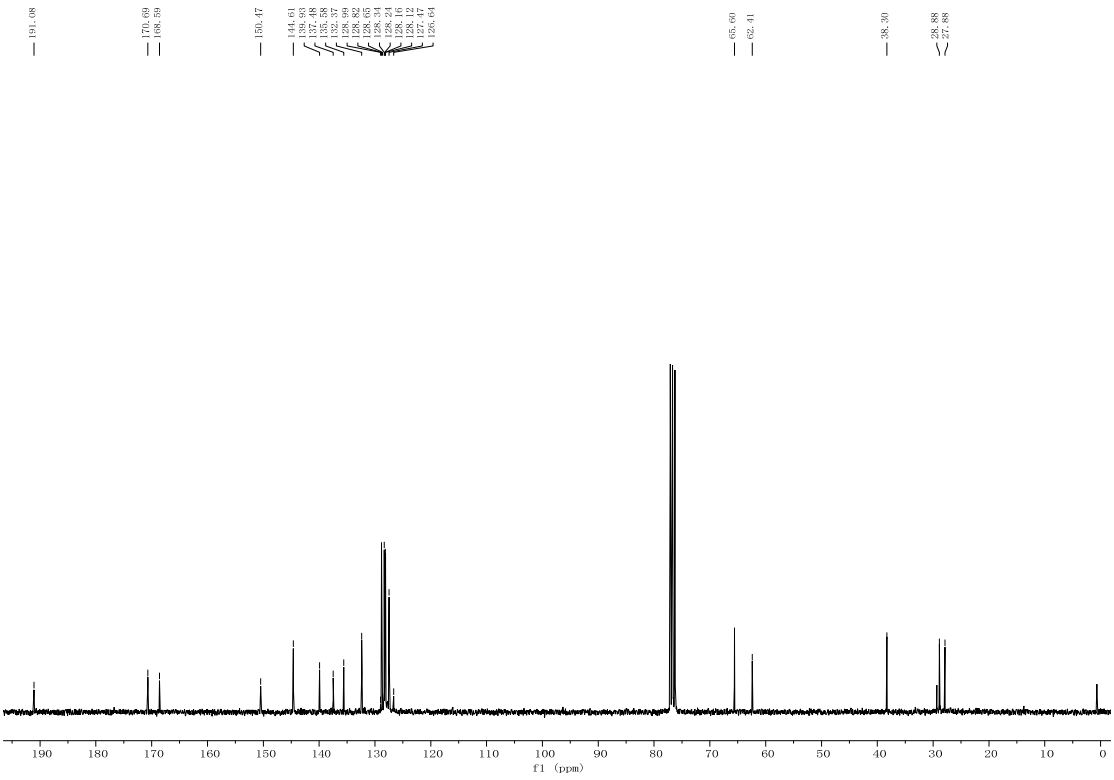


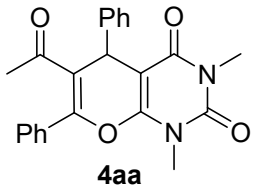




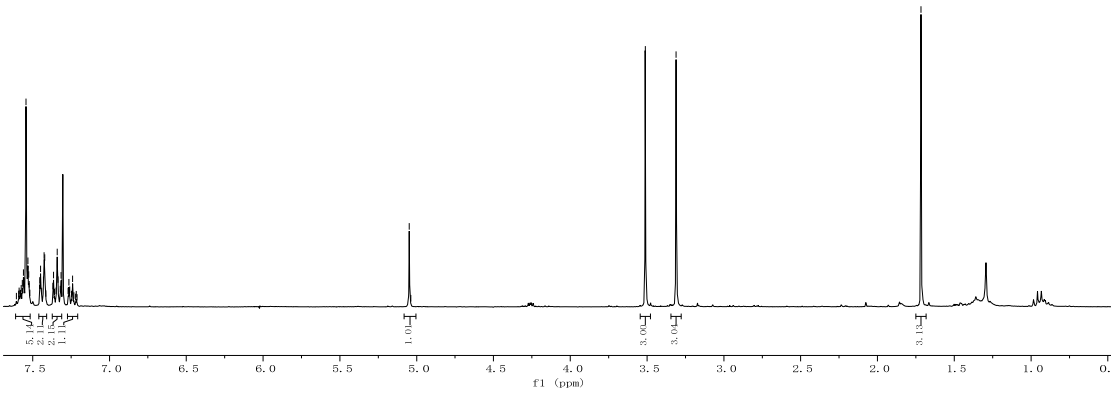
3ma

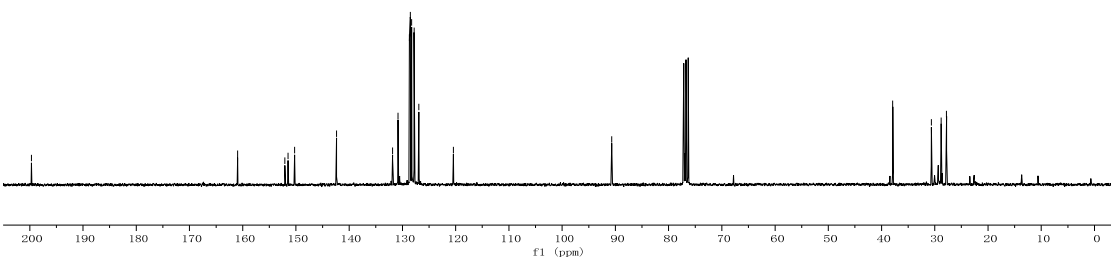


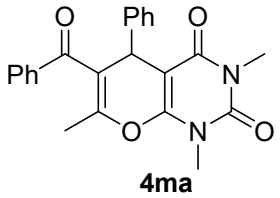




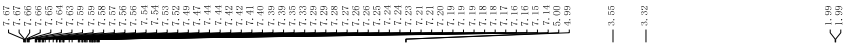
4aa

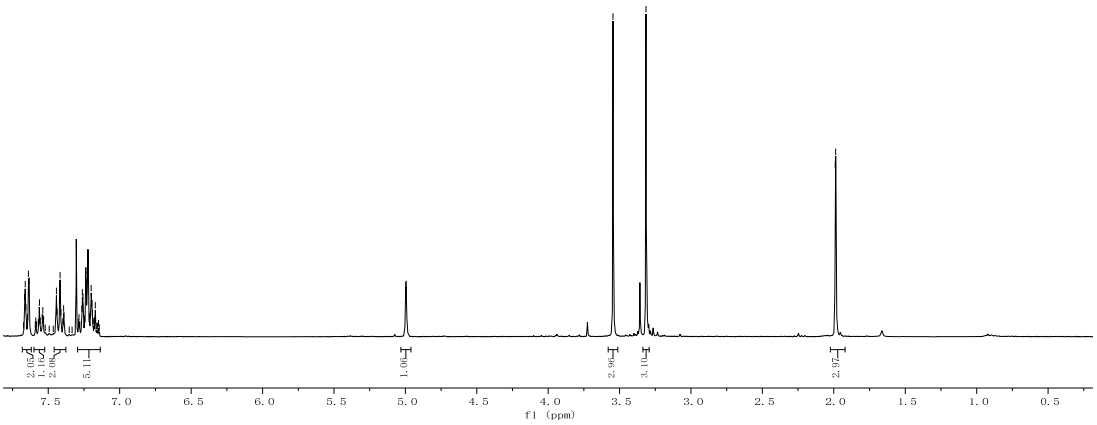


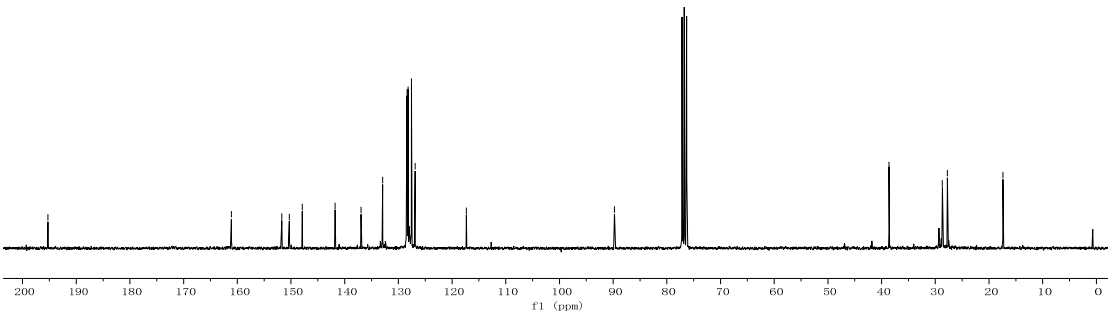




4ma

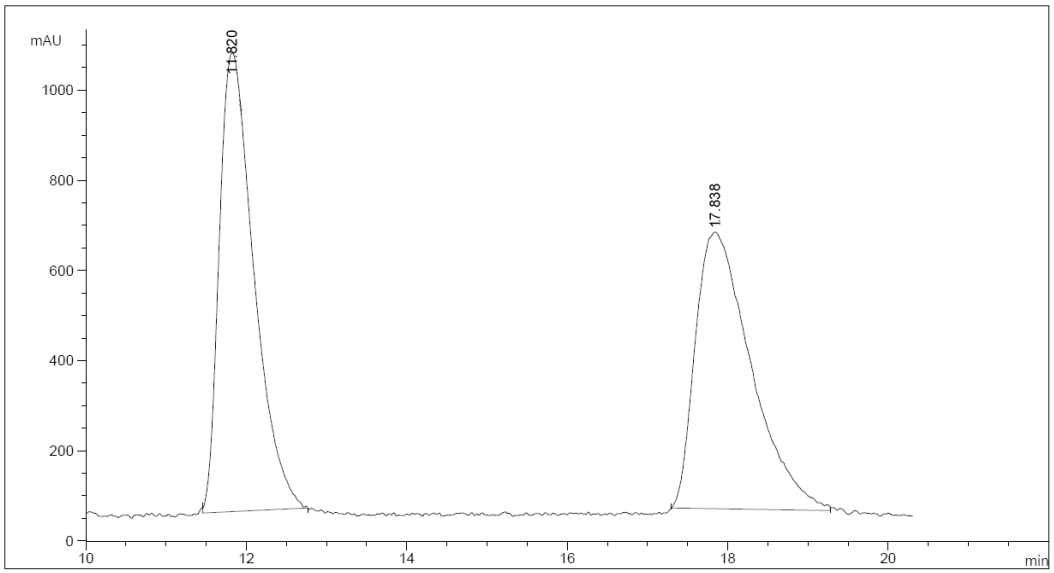






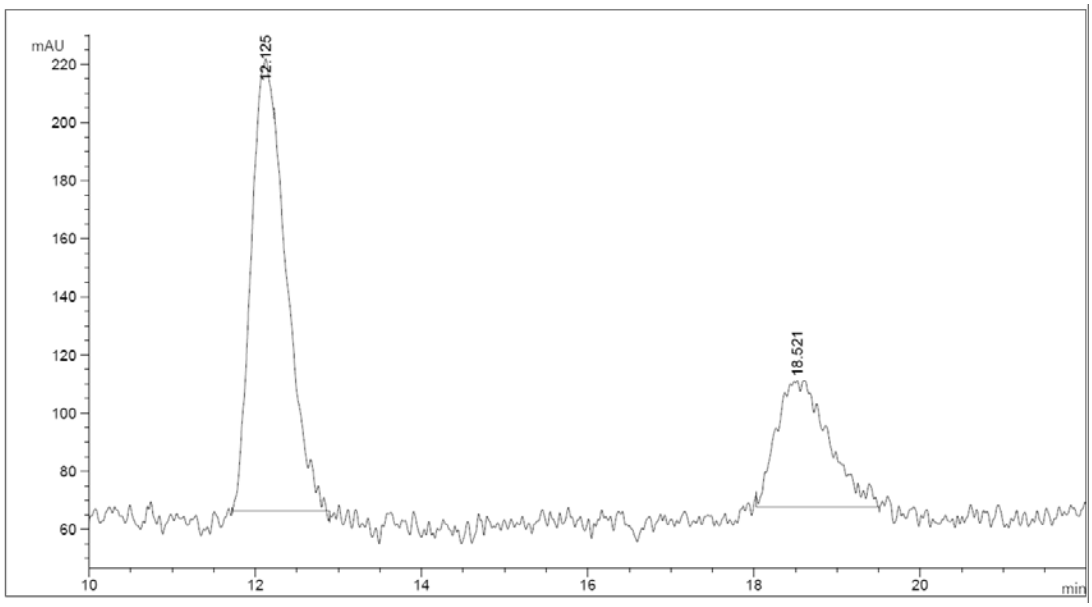
Chiral HPLC analysis of chiral catalyst entry

HPLC chromatogram of racemic product 3aa



Peak #	Ret Time [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	11.820	MM	0.4996	3.05099e4	1017.89349	50.7918
2	17.838	MM	0.8026	2.95587e4	613.79730	49.2082

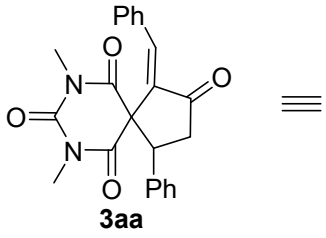
HPLC chromatogram of chiral product 3aa



Peak #	Ret Time [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	12.125	MM	0. 4823	4496.48340	155.39626	70.2471
2	18.521	MM	0.7304	1904.46936	43.45869	29.7529

X-Ray Crystallographic Data

Crystallographic data for **3aa**, **3ma** and **4ma** has been deposited with the Cambridge Crystallographic Data Centre as deposition number CCDC 1474966, 1525361 and 1525356, respectively. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.



3aa

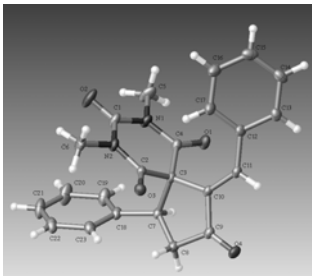


Table 1. Crystal data and structure refinement for **3aa**

Identification code	**3aa**	
Empirical formula	$C_{23}H_{20}N_2O_4$	
Formula weight	388.41	
Temperature	173.1500 K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 8.5462(17) Å	a= 90.29(3)°.
	b = 9.7412(19) Å	b= 104.04(3)°.
	c = 12.085(2) Å	g = 94.61(3)°.
Volume	972.6(4) $Å^3$	
Z	2	
Density (calculated)	1.326 Mg/m^3	
Absorption coefficient	0.092 mm^{-1}	

F(000)	408
Crystal size	0.33 x 0.21 x 0.16 mm^3
Theta range for data collection	2.465 to 27.490°.
Index ranges	-11<=h<=11, -12<=k<=12, -15<=l<=14
Reflections collected	10072
Independent reflections	4384 [R(int) = 0.0319]
Completeness to theta = 26.000°	98.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.7894
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4384 / 0 / 264
Goodness-of-fit on F^2	1.152
Final R indices [I>2sigma(I)]	R1 = 0.0644, wR2 = 0.1352
R indices (all data)	R1 = 0.0734, wR2 = 0.1425
Extinction coefficient	n/a
Largest diff. peak and hole	0.270 and -0.253 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **3aa**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O1	5089(2)	747(2)	3620(1)	37(1)
O2	5770(2)	-1442(2)	486(1)	60(1)
O3	1544(2)	-3006(1)	1913(1)	32(1)
O4	1019(2)	-1828(2)	5124(1)	39(1)
N1	5489(2)	-376(2)	2087(1)	33(1)
N2	3679(2)	-2262(2)	1210(1)	30(1)
C1	5035(2)	-1373(2)	1218(2)	36(1)
C2	2787(2)	-2263(2)	2020(2)	25(1)

C3	3495(2)	-1387(2)	3086(2)	24(1)
C4	4716(2)	-222(2)	2951(2)	28(1)
C5	6884(3)	598(3)	2062(2)	54(1)
C6	3210(3)	-3322(2)	289(2)	41(1)
C7	4452(2)	-2384(2)	4036(2)	28(1)
C8	3137(2)	-3001(2)	4592(2)	32(1)
C9	1986(2)	-1902(2)	4541(2)	30(1)
C10	2194(2)	-925(2)	3631(2)	26(1)
C11	1325(2)	165(2)	3425(2)	29(1)
C12	1266(2)	1197(2)	2536(2)	30(1)
C13	952(2)	2537(2)	2765(2)	36(1)
C14	879(3)	3529(2)	1939(2)	43(1)
C15	1074(3)	3189(3)	871(2)	47(1)
C16	1338(3)	1860(3)	617(2)	48(1)
C17	1439(3)	868(2)	1448(2)	42(1)
C18	5435(2)	-3374(2)	3580(2)	31(1)
C19	7048(3)	-2994(3)	3606(2)	46(1)
C20	7935(3)	-3849(3)	3115(2)	64(1)
C21	7223(3)	-5088(3)	2607(2)	59(1)
C22	5646(3)	-5486(2)	2590(2)	45(1)
C23	4756(3)	-4637(2)	3079(2)	36(1)

Table 3. Bond lengths [Å] and angles [°] for **3aa**.

O1-C4	1.209(2)
O2-C1	1.208(2)
O3-C2	1.215(2)
O4-C9	1.215(2)
N1-C1	1.388(3)

N1-C4	1.378(2)
N1-C5	1.469(3)
N2-C1	1.388(3)
N2-C2	1.379(2)
N2-C6	1.473(2)
C2-C3	1.510(2)
C3-C4	1.516(3)
C3-C7	1.617(3)
C3-C10	1.519(2)
C5-H5A	0.9600
C5-H5B	0.9600
C5-H5C	0.9600
C6-H6A	0.9600
C6-H6B	0.9600
C6-H6C	0.9600
C7-H7	0.9800
C7-C8	1.529(3)
C7-C18	1.513(3)
C8-H8A	0.9700
C8-H8B	0.9700
C8-C9	1.503(3)
C9-C10	1.491(3)
C10-C11	1.334(3)
C11-H11	0.9300
C11-C12	1.469(3)
C12-C13	1.393(3)
C12-C17	1.397(3)
C13-H13	0.9300
C13-C14	1.387(3)
C14-H14	0.9300

C14-C15	1.383(4)
C15-H15	0.9300
C15-C16	1.377(4)
C16-H16	0.9300
C16-C17	1.390(3)
C17-H17	0.9300
C18-C19	1.390(3)
C18-C23	1.388(3)
C19-H19	0.9300
C19-C20	1.392(3)
C20-H20	0.9300
C20-C21	1.377(4)
C21-H21	0.9300
C21-C22	1.368(4)
C22-H22	0.9300
C22-C23	1.387(3)
C23-H23	0.9300

C1-N1-C5	116.95(17)
C4-N1-C1	125.01(16)
C4-N1-C5	118.04(17)
C1-N2-C6	116.95(16)
C2-N2-C1	125.03(16)
C2-N2-C6	117.95(16)
O2-C1-N1	121.25(19)
O2-C1-N2	121.44(19)
N2-C1-N1	117.29(16)
O3-C2-N2	121.31(16)
O3-C2-C3	121.63(16)
N2-C2-C3	116.89(15)

C2-C3-C4	114.68(15)
C2-C3-C7	106.99(14)
C2-C3-C10	112.01(14)
C4-C3-C7	106.46(14)
C4-C3-C10	113.19(15)
C10-C3-C7	102.38(14)
O1-C4-N1	121.25(17)
O1-C4-C3	121.89(17)
N1-C4-C3	116.63(16)
N1-C5-H5A	109.5
N1-C5-H5B	109.5
N1-C5-H5C	109.5
H5A-C5-H5B	109.5
H5A-C5-H5C	109.5
H5B-C5-H5C	109.5
N2-C6-H6A	109.5
N2-C6-H6B	109.5
N2-C6-H6C	109.5
H6A-C6-H6B	109.5
H6A-C6-H6C	109.5
H6B-C6-H6C	109.5
C3-C7-H7	107.1
C8-C7-C3	103.46(14)
C8-C7-H7	107.1
C18-C7-C3	114.05(15)
C18-C7-H7	107.1
C18-C7-C8	117.47(16)
C7-C8-H8A	110.7
C7-C8-H8B	110.7
H8A-C8-H8B	108.8

C9-C8-C7	105.35(15)
C9-C8-H8A	110.7
C9-C8-H8B	110.7
O4-C9-C8	126.04(18)
O4-C9-C10	125.21(18)
C10-C9-C8	108.73(15)
C9-C10-C3	109.08(15)
C11-C10-C3	130.27(17)
C11-C10-C9	120.64(17)
C10-C11-H11	115.3
C10-C11-C12	129.50(17)
C12-C11-H11	115.3
C13-C12-C11	119.06(18)
C13-C12-C17	118.56(19)
C17-C12-C11	122.32(18)
C12-C13-H13	119.9
C14-C13-C12	120.3(2)
C14-C13-H13	119.9
C13-C14-H14	119.9
C15-C14-C13	120.3(2)
C15-C14-H14	119.9
C14-C15-H15	119.9
C16-C15-C14	120.3(2)
C16-C15-H15	119.9
C15-C16-H16	120.2
C15-C16-C17	119.6(2)
C17-C16-H16	120.2
C12-C17-H17	119.5
C16-C17-C12	120.9(2)
C16-C17-H17	119.5

C19-C18-C7	119.77(19)
C23-C18-C7	121.90(18)
C23-C18-C19	118.25(19)
C18-C19-H19	119.8
C18-C19-C20	120.5(2)
C20-C19-H19	119.8
C19-C20-H20	120.0
C21-C20-C19	120.1(2)
C21-C20-H20	120.0
C20-C21-H21	119.9
C22-C21-C20	120.1(2)
C22-C21-H21	119.9
C21-C22-H22	120.0
C21-C22-C23	120.0(2)
C23-C22-H22	120.0
C18-C23-H23	119.5
C22-C23-C18	121.0(2)
C22-C23-H23	119.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2$ x 10^3) for **3aa**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O1	40(1)	37(1)	35(1)	-13(1)	14(1)	-6(1)
O2	50(1)	92(1)	44(1)	-26(1)	30(1)	-15(1)
O3	29(1)	31(1)	35(1)	-2(1)	7(1)	-1(1)
O4	38(1)	48(1)	39(1)	4(1)	24(1)	7(1)
N1	29(1)	44(1)	29(1)	-6(1)	14(1)	-7(1)
N2	30(1)	36(1)	24(1)	-7(1)	8(1)	2(1)
C1	31(1)	51(1)	29(1)	-9(1)	14(1)	0(1)
C2	25(1)	26(1)	24(1)	0(1)	6(1)	6(1)

C3	24(1)	28(1)	22(1)	-2(1)	8(1)	4(1)
C4	27(1)	32(1)	26(1)	-3(1)	8(1)	2(1)
C5	43(1)	73(2)	46(1)	-11(1)	21(1)	-27(1)
C6	43(1)	48(1)	32(1)	-16(1)	12(1)	-1(1)
C7	26(1)	33(1)	24(1)	-1(1)	5(1)	7(1)
C8	37(1)	34(1)	29(1)	4(1)	13(1)	8(1)
C9	28(1)	35(1)	26(1)	-3(1)	9(1)	2(1)
C10	25(1)	31(1)	26(1)	-3(1)	10(1)	2(1)
C11	27(1)	33(1)	31(1)	-4(1)	12(1)	4(1)
C12	24(1)	32(1)	38(1)	2(1)	11(1)	6(1)
C13	29(1)	33(1)	47(1)	-2(1)	11(1)	4(1)
C14	34(1)	29(1)	65(2)	7(1)	10(1)	3(1)
C15	32(1)	53(1)	59(2)	22(1)	13(1)	6(1)
C16	44(1)	63(2)	43(1)	14(1)	18(1)	22(1)
C17	46(1)	44(1)	41(1)	5(1)	17(1)	20(1)
C18	29(1)	39(1)	25(1)	-2(1)	4(1)	12(1)
C19	29(1)	62(2)	45(1)	-19(1)	4(1)	9(1)
C20	30(1)	97(2)	65(2)	-30(2)	10(1)	16(1)
C21	47(1)	78(2)	51(2)	-25(1)	6(1)	30(1)
C22	50(1)	45(1)	39(1)	-9(1)	1(1)	21(1)
C23	36(1)	37(1)	34(1)	0(1)	4(1)	12(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **3aa**.

	x	y	z	U(eq)
H5A	7708	113	1853	81
H5B	7306	1025	2803	81
H5C	6550	1292	1513	81
H6A	2306	-3048	-284	62
H6B	2913	-4183	596	62

H6C	4106	-3424	-46	62
H7	5213	-1790	4614	33
H8A	2586	-3828	4178	39
H8B	3601	-3232	5377	39
H11	660	290	3916	35
H13	791	2767	3474	43
H14	697	4428	2104	52
H15	1028	3860	322	57
H16	1448	1628	-106	57
H17	1625	-28	1278	50
H19	7537	-2163	3955	55
H20	9010	-3584	3130	76
H21	7815	-5655	2274	70
H22	5169	-6325	2251	55
H23	3688	-4920	3072	43

Table 6. Torsion angles [°] for **3aa**.

O3-C2-C3-C4	-162.13(16)
O3-C2-C3-C7	80.1(2)
O3-C2-C3-C10	-31.4(2)
O4-C9-C10-C3	-178.79(18)
O4-C9-C10-C11	2.5(3)
N2-C2-C3-C4	22.6(2)
N2-C2-C3-C7	-95.20(18)
N2-C2-C3-C10	153.38(16)
C1-N1-C4-O1	-173.2(2)
C1-N1-C4-C3	12.2(3)
C1-N2-C2-O3	174.58(19)
C1-N2-C2-C3	-10.1(3)
C2-N2-C1-O2	179.1(2)
C2-N2-C1-N1	-2.6(3)

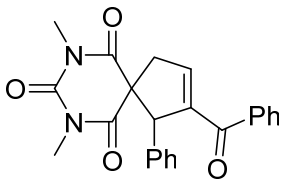
C2-C3-C4-O1	161.92(17)
C2-C3-C4-N1	-23.6(2)
C2-C3-C7-C8	-86.66(16)
C2-C3-C7-C18	42.1(2)
C2-C3-C10-C9	95.05(18)
C2-C3-C10-C11	-86.4(2)
C3-C7-C8-C9	-31.98(18)
C3-C7-C18-C19	91.3(2)
C3-C7-C18-C23	-85.5(2)
C3-C10-C11-C12	5.7(3)
C4-N1-C1-O2	179.7(2)
C4-N1-C1-N2	1.4(3)
C4-C3-C7-C8	150.28(15)
C4-C3-C7-C18	-80.94(19)
C4-C3-C10-C9	-133.41(16)
C4-C3-C10-C11	45.1(3)
C5-N1-C1-O2	0.9(3)
C5-N1-C1-N2	-177.5(2)
C5-N1-C4-O1	5.7(3)
C5-N1-C4-C3	-168.88(19)
C6-N2-C1-O2	2.0(3)
C6-N2-C1-N1	-179.68(19)
C6-N2-C2-O3	-8.4(3)
C6-N2-C2-C3	166.91(17)
C7-C3-C4-O1	-80.0(2)
C7-C3-C4-N1	94.53(18)
C7-C3-C10-C9	-19.24(18)
C7-C3-C10-C11	159.32(19)
C7-C8-C9-O4	-160.46(19)
C7-C8-C9-C10	20.9(2)
C7-C18-C19-C20	-175.3(2)
C7-C18-C23-C22	175.16(19)
C8-C7-C18-C19	-147.4(2)

C8-C7-C18-C23	35.8(3)
C8-C9-C10-C3	-0.1(2)
C8-C9-C10-C11	-178.87(17)
C9-C10-C11-C12	-175.86(18)
C10-C3-C4-O1	31.7(2)
C10-C3-C4-N1	-153.78(16)
C10-C3-C7-C8	31.26(17)
C10-C3-C7-C18	160.03(15)
C10-C11-C12-C13	-149.1(2)
C10-C11-C12-C17	33.8(3)
C11-C12-C13-C14	-179.60(17)
C11-C12-C17-C16	178.44(19)
C12-C13-C14-C15	1.7(3)
C13-C12-C17-C16	1.4(3)
C13-C14-C15-C16	0.2(3)
C14-C15-C16-C17	-1.3(3)
C15-C16-C17-C12	0.5(3)
C17-C12-C13-C14	-2.5(3)
C18-C7-C8-C9	-158.62(16)
C18-C19-C20-C21	-0.6(4)
C19-C18-C23-C22	-1.7(3)
C19-C20-C21-C22	-0.5(5)
C20-C21-C22-C23	0.4(4)
C21-C22-C23-C18	0.7(3)
C23-C18-C19-C20	1.6(4)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for **3aa** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)



3ma

≡

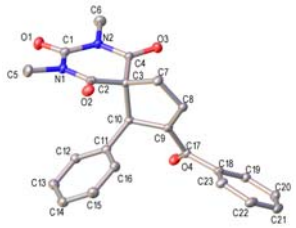


Table 1. Crystal data and structure refinement for **3ma**.

Identification code	**3ma**	
Empirical formula	C23 H20 N2 O4	
Formula weight	388.41	
Temperature	173 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 1 21/c 1	
Unit cell dimensions	a = 8.989(4) Å	α= 90°.
	b = 5.730(3) Å	β= 96.682(6)°.
	c = 36.156(16) Å	γ = 90°.
Volume	1849.6(15) $Å^3$	
Z	4	
Density (calculated)	1.395 Mg/m^3	
Absorption coefficient	0.096 mm^{-1}	
F(000)	816	
Crystal size	0.24 x 0.07 x 0.07 mm^3	
Theta range for data collection	1.134 to 27.500°.	
Index ranges	-11<=h<=11, -6<=k<=7, -46<=l<=46	
Reflections collected	15481	
Independent reflections	4233 [R(int) = 0.1260]	
Completeness to theta = 26.000°	99.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.000 and 0.815	
Refinement method	Full-matrix least-squares on F^2	

Data / restraints / parameters	4233 / 0 / 264
Goodness-of-fit on F^2	1.181
Final R indices [I>2sigma(I)]	R1 = 0.0714, wR2 = 0.1666
R indices (all data)	R1 = 0.0774, wR2 = 0.1711
Extinction coefficient	n/a
Largest diff. peak and hole	0.314 and -0.274 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **3ma**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O1	-358(2)	4771(3)	6960(1)	38(1)
O2	2317(2)	11131(3)	6687(1)	35(1)
O3	686(2)	5679(3)	5774(1)	36(1)
O4	5415(2)	3402(3)	5897(1)	33(1)
N1	1040(2)	7919(3)	6835(1)	28(1)
N2	103(2)	5265(3)	6362(1)	28(1)
C1	247(2)	5885(4)	6736(1)	27(1)
C2	1819(2)	9215(4)	6598(1)	26(1)
C3	2084(2)	8045(4)	6236(1)	24(1)
C4	894(2)	6283(4)	6097(1)	25(1)
C5	949(3)	8837(5)	7212(1)	38(1)
C6	-944(3)	3357(5)	6247(1)	39(1)
C7	2307(3)	9815(4)	5924(1)	30(1)
C8	3554(2)	8796(4)	5735(1)	26(1)
C9	4222(2)	7016(4)	5922(1)	24(1)
C10	3616(2)	6566(4)	6290(1)	23(1)
C11	4718(2)	7267(4)	6622(1)	24(1)
C12	4825(3)	5919(4)	6944(1)	30(1)

C13	5808(3)	6537(5)	7253(1)	36(1)
C14	6713(3)	8486(5)	7242(1)	36(1)
C15	6603(3)	9851(4)	6923(1)	36(1)
C16	5619(3)	9248(4)	6614(1)	31(1)
C17	5343(2)	5437(4)	5791(1)	24(1)
C18	6363(2)	6294(4)	5523(1)	25(1)
C19	6740(2)	4799(4)	5246(1)	30(1)
C20	7754(3)	5483(5)	5008(1)	37(1)
C21	8423(3)	7646(5)	5050(1)	38(1)
C22	8062(3)	9152(4)	5323(1)	37(1)
C23	7007(2)	8505(4)	5559(1)	32(1)

Table 3. Bond lengths [Å] and angles [°] for **3ma**.

O1-C1	1.209(3)
O2-C2	1.214(3)
O3-C4	1.212(3)
O4-C17	1.227(3)
N1-C1	1.391(3)
N1-C2	1.385(3)
N1-C5	1.471(3)
N2-C1	1.391(3)
N2-C4	1.386(3)
N2-C6	1.470(3)
C2-C3	1.515(3)
C3-C4	1.513(3)
C3-C7	1.546(3)
C3-C10	1.609(3)
C5-H5A	0.9600

C5-H5B	0.9600
C5-H5C	0.9600
C6-H6A	0.9600
C6-H6B	0.9600
C6-H6C	0.9600
C7-H7A	0.9700
C7-H7B	0.9700
C7-C8	1.498(3)
C8-H8	0.9300
C8-C9	1.329(3)
C9-C10	1.516(3)
C9-C17	1.473(3)
C10-H10	0.9800
C10-C11	1.517(3)
C11-C12	1.391(3)
C11-C16	1.397(3)
C12-H12	0.9300
C12-C13	1.389(3)
C13-H13	0.9300
C13-C14	1.385(4)
C14-H14	0.9300
C14-C15	1.386(4)
C15-H15	0.9300
C15-C16	1.386(3)
C16-H16	0.9300
C17-C18	1.493(3)
C18-C19	1.389(3)
C18-C23	1.393(3)
C19-H19	0.9300
C19-C20	1.382(3)

C20-H20	0.9300
C20-C21	1.378(4)
C21-H21	0.9300
C21-C22	1.378(4)
C22-H22	0.9300
C22-C23	1.396(3)
C23-H23	0.9300

C1-N1-C5	117.15(18)
C2-N1-C1	124.75(18)
C2-N1-C5	117.96(19)
C1-N2-C6	116.66(19)
C4-N2-C1	124.54(19)
C4-N2-C6	118.79(19)
O1-C1-N1	121.7(2)
O1-C1-N2	121.3(2)
N2-C1-N1	116.89(19)
O2-C2-N1	121.2(2)
O2-C2-C3	122.5(2)
N1-C2-C3	116.16(19)
C2-C3-C7	112.76(18)
C2-C3-C10	110.22(17)
C4-C3-C2	113.86(17)
C4-C3-C7	110.08(18)
C4-C3-C10	104.52(17)
C7-C3-C10	104.68(16)
O3-C4-N2	120.7(2)
O3-C4-C3	122.23(19)
N2-C4-C3	116.92(18)
N1-C5-H5A	109.5

N1-C5-H5B	109.5
N1-C5-H5C	109.5
H5A-C5-H5B	109.5
H5A-C5-H5C	109.5
H5B-C5-H5C	109.5
N2-C6-H6A	109.5
N2-C6-H6B	109.5
N2-C6-H6C	109.5
H6A-C6-H6B	109.5
H6A-C6-H6C	109.5
H6B-C6-H6C	109.5
C3-C7-H7A	110.9
C3-C7-H7B	110.9
H7A-C7-H7B	109.0
C8-C7-C3	104.12(17)
C8-C7-H7A	110.9
C8-C7-H7B	110.9
C7-C8-H8	123.7
C9-C8-C7	112.61(19)
C9-C8-H8	123.7
C8-C9-C10	112.98(18)
C8-C9-C17	126.34(19)
C17-C9-C10	120.48(18)
C3-C10-H10	109.3
C9-C10-C3	101.10(16)
C9-C10-H10	109.3
C9-C10-C11	112.32(17)
C11-C10-C3	115.31(17)
C11-C10-H10	109.3
C12-C11-C10	119.31(19)

C12-C11-C16	118.9(2)
C16-C11-C10	121.79(19)
C11-C12-H12	119.7
C13-C12-C11	120.5(2)
C13-C12-H12	119.7
C12-C13-H13	119.9
C14-C13-C12	120.2(2)
C14-C13-H13	119.9
C13-C14-H14	120.2
C13-C14-C15	119.6(2)
C15-C14-H14	120.2
C14-C15-H15	119.8
C16-C15-C14	120.4(2)
C16-C15-H15	119.8
C11-C16-H16	119.8
C15-C16-C11	120.3(2)
C15-C16-H16	119.8
O4-C17-C9	119.76(19)
O4-C17-C18	120.06(19)
C9-C17-C18	120.17(18)
C19-C18-C17	119.0(2)
C19-C18-C23	119.6(2)
C23-C18-C17	121.26(19)
C18-C19-H19	119.7
C20-C19-C18	120.6(2)
C20-C19-H19	119.7
C19-C20-H20	120.1
C21-C20-C19	119.8(2)
C21-C20-H20	120.1
C20-C21-H21	119.8

C20-C21-C22	120.4(2)
C22-C21-H21	119.8
C21-C22-H22	119.9
C21-C22-C23	120.3(2)
C23-C22-H22	119.9
C18-C23-C22	119.3(2)
C18-C23-H23	120.4
C22-C23-H23	120.4

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2$ x 10^3) for **3ma**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O1	41(1)	41(1)	35(1)	6(1)	13(1)	-6(1)
O2	33(1)	29(1)	45(1)	-7(1)	10(1)	-2(1)
O3	29(1)	53(1)	26(1)	-4(1)	1(1)	-5(1)
O4	32(1)	27(1)	43(1)	5(1)	13(1)	3(1)
N1	26(1)	33(1)	24(1)	-3(1)	6(1)	-1(1)
N2	22(1)	34(1)	28(1)	-1(1)	2(1)	-5(1)
C1	23(1)	32(1)	28(1)	-1(1)	5(1)	1(1)
C2	21(1)	27(1)	31(1)	0(1)	4(1)	3(1)
C3	21(1)	26(1)	26(1)	1(1)	4(1)	2(1)
C4	20(1)	33(1)	24(1)	2(1)	4(1)	1(1)
C5	40(1)	46(1)	29(1)	-8(1)	9(1)	-2(1)
C6	31(1)	44(1)	40(1)	-4(1)	2(1)	-12(1)
C7	28(1)	29(1)	33(1)	6(1)	9(1)	6(1)
C8	23(1)	28(1)	27(1)	3(1)	5(1)	-2(1)

C9	19(1)	25(1)	27(1)	0(1)	5(1)	-3(1)
C10	18(1)	22(1)	28(1)	1(1)	4(1)	2(1)
C11	21(1)	25(1)	27(1)	0(1)	5(1)	3(1)
C12	26(1)	31(1)	33(1)	4(1)	4(1)	-1(1)
C13	32(1)	43(1)	31(1)	6(1)	1(1)	1(1)
C14	28(1)	46(1)	31(1)	-2(1)	0(1)	0(1)
C15	31(1)	38(1)	38(1)	-2(1)	2(1)	-8(1)
C16	31(1)	32(1)	30(1)	4(1)	4(1)	-3(1)
C17	21(1)	25(1)	26(1)	-2(1)	2(1)	0(1)
C18	20(1)	28(1)	28(1)	2(1)	3(1)	4(1)
C19	24(1)	35(1)	31(1)	-2(1)	2(1)	2(1)
C20	30(1)	53(2)	29(1)	-2(1)	8(1)	7(1)
C21	26(1)	53(2)	37(1)	14(1)	11(1)	6(1)
C22	24(1)	37(1)	51(1)	13(1)	11(1)	0(1)
C23	25(1)	29(1)	42(1)	1(1)	9(1)	2(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **3ma**.

	x	y	z	U(eq)
H5A	89	8187	7309	57
H5B	860	10506	7202	57
H5C	1838	8415	7371	57
H6A	-563	1922	6358	58
H6B	-1051	3211	5981	58
H6C	-1902	3691	6328	58
H7A	1402	9966	5751	35
H7B	2581	11338	6028	35

H8	3830	9348	5511	31
H10	3378	4904	6309	27
H12	4233	4593	6952	36
H13	5859	5640	7469	43
H14	7391	8876	7447	43
H15	7194	11180	6917	43
H16	5557	10168	6401	37
H19	6306	3326	5221	36
H20	7985	4486	4820	44
H21	9122	8092	4893	46
H22	8523	10604	5351	44
H23	6737	9540	5737	38

Table 6. Torsion angles [°] for **3ma**.

O2-C2-C3-C4	-154.8(2)
O2-C2-C3-C7	-28.4(3)
O2-C2-C3-C10	88.2(2)
O4-C17-C18-C19	35.9(3)
O4-C17-C18-C23	-140.4(2)
N1-C2-C3-C4	28.6(3)
N1-C2-C3-C7	155.00(19)
N1-C2-C3-C10	-88.4(2)
C1-N1-C2-O2	170.0(2)
C1-N1-C2-C3	-13.4(3)
C1-N2-C4-O3	-178.5(2)
C1-N2-C4-C3	5.3(3)
C2-N1-C1-O1	176.7(2)
C2-N1-C1-N2	-7.2(3)
C2-C3-C4-O3	158.9(2)
C2-C3-C4-N2	-25.0(3)

C2-C3-C7-C8	138.95(18)
C2-C3-C10-C9	-141.98(18)
C2-C3-C10-C11	-20.6(2)
C3-C7-C8-C9	-10.7(3)
C3-C10-C11-C12	101.8(2)
C3-C10-C11-C16	-77.5(2)
C4-N2-C1-O1	-172.3(2)
C4-N2-C1-N1	11.6(3)
C4-C3-C7-C8	-92.7(2)
C4-C3-C10-C9	95.29(18)
C4-C3-C10-C11	-143.32(18)
C5-N1-C1-O1	-7.8(3)
C5-N1-C1-N2	168.3(2)
C5-N1-C2-O2	-5.5(3)
C5-N1-C2-C3	171.2(2)
C6-N2-C1-O1	6.3(3)
C6-N2-C1-N1	-169.8(2)
C6-N2-C4-O3	2.9(3)
C6-N2-C4-C3	-173.25(19)
C7-C3-C4-O3	31.2(3)
C7-C3-C4-N2	-152.70(18)
C7-C3-C10-C9	-20.5(2)
C7-C3-C10-C11	100.9(2)
C7-C8-C9-C10	-3.5(3)
C7-C8-C9-C17	171.3(2)
C8-C9-C10-C3	15.3(2)
C8-C9-C10-C11	-108.2(2)
C8-C9-C17-O4	-148.9(2)
C8-C9-C17-C18	29.5(3)
C9-C10-C11-C12	-143.13(19)
C9-C10-C11-C16	37.6(3)
C9-C17-C18-C19	-142.6(2)

C9-C17-C18-C23	41.2(3)
C10-C3-C4-O3	-80.7(2)
C10-C3-C4-N2	95.4(2)
C10-C3-C7-C8	19.1(2)
C10-C9-C17-O4	25.5(3)
C10-C9-C17-C18	-156.04(19)
C10-C11-C12-C13	-179.1(2)
C10-C11-C16-C15	179.4(2)
C11-C12-C13-C14	-1.1(4)
C12-C11-C16-C15	0.1(3)
C12-C13-C14-C15	1.7(4)
C13-C14-C15-C16	-1.4(4)
C14-C15-C16-C11	0.5(4)
C16-C11-C12-C13	0.2(3)
C17-C9-C10-C3	-159.85(18)
C17-C9-C10-C11	76.7(2)
C17-C18-C19-C20	-175.9(2)
C17-C18-C23-C22	174.0(2)
C18-C19-C20-C21	1.4(4)
C19-C18-C23-C22	-2.2(3)
C19-C20-C21-C22	-1.4(4)
C20-C21-C22-C23	-0.4(4)
C21-C22-C23-C18	2.2(4)
C23-C18-C19-C20	0.4(3)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for **3ma** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)

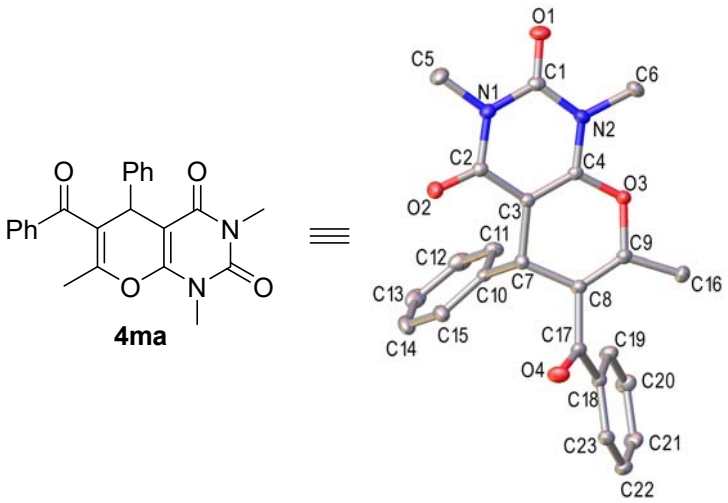


Table 1. Crystal data and structure refinement for **4ma**.

Identification code	**4ma**	
Empirical formula	C23 H20 N2 O4	
Formula weight	388.41	
Temperature	173 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 1 21/c 1	
Unit cell dimensions	a = 8.989(4) Å	α= 90°.
	b = 5.730(3) Å	β= 96.682(6)°.
	c = 36.156(16) Å	γ = 90°.
Volume	1849.6(15) $Å^3$	
Z	4	
Density (calculated)	1.395 Mg/m^3	
Absorption coefficient	0.096 mm^{-1}	
F(000)	816	
Crystal size	0.24 x 0.07 x 0.07 mm^3	
Theta range for data collection	1.134 to 27.500°.	
Index ranges	-11<=h<=11, -6<=k<=7, -46<=l<=46	
Reflections collected	15481	
Independent reflections	4233 [R(int) = 0.1260]	
Completeness to theta = 26.000°	99.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.000 and 0.815	

Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4233 / 0 / 264
Goodness-of-fit on F^2	1.181
Final R indices [I>2sigma(I)]	R1 = 0.0714, wR2 = 0.1666
R indices (all data)	R1 = 0.0774, wR2 = 0.1711
Extinction coefficient	n/a
Largest diff. peak and hole	0.314 and -0.274 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **4ma**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O1	-358(2)	4771(3)	6960(1)	38(1)
O2	2317(2)	11131(3)	6687(1)	35(1)
O3	686(2)	5679(3)	5774(1)	36(1)
O4	5415(2)	3402(3)	5897(1)	33(1)
N1	1040(2)	7919(3)	6835(1)	28(1)
N2	103(2)	5265(3)	6362(1)	28(1)
C1	247(2)	5885(4)	6736(1)	27(1)
C2	1819(2)	9215(4)	6598(1)	26(1)
C3	2084(2)	8045(4)	6236(1)	24(1)
C4	894(2)	6283(4)	6097(1)	25(1)
C5	949(3)	8837(5)	7212(1)	38(1)
C6	-944(3)	3357(5)	6247(1)	39(1)
C7	2307(3)	9815(4)	5924(1)	30(1)
C8	3554(2)	8796(4)	5735(1)	26(1)
C9	4222(2)	7016(4)	5922(1)	24(1)
C10	3616(2)	6566(4)	6290(1)	23(1)
C11	4718(2)	7267(4)	6622(1)	24(1)
C12	4825(3)	5919(4)	6944(1)	30(1)

C13	5808(3)	6537(5)	7253(1)	36(1)
C14	6713(3)	8486(5)	7242(1)	36(1)
C15	6603(3)	9851(4)	6923(1)	36(1)
C16	5619(3)	9248(4)	6614(1)	31(1)
C17	5343(2)	5437(4)	5791(1)	24(1)
C18	6363(2)	6294(4)	5523(1)	25(1)
C19	6740(2)	4799(4)	5246(1)	30(1)
C20	7754(3)	5483(5)	5008(1)	37(1)
C21	8423(3)	7646(5)	5050(1)	38(1)
C22	8062(3)	9152(4)	5323(1)	37(1)
C23	7007(2)	8505(4)	5559(1)	32(1)

Table 3. Bond lengths [Å] and angles [°] for **4ma**.

O1-C1	1.209(3)
O2-C2	1.214(3)
O3-C4	1.212(3)
O4-C17	1.227(3)
N1-C1	1.391(3)
N1-C2	1.385(3)
N1-C5	1.471(3)
N2-C1	1.391(3)
N2-C4	1.386(3)
N2-C6	1.470(3)
C2-C3	1.515(3)
C3-C4	1.513(3)
C3-C7	1.546(3)
C3-C10	1.609(3)
C5-H5A	0.9600

C5-H5B	0.9600
C5-H5C	0.9600
C6-H6A	0.9600
C6-H6B	0.9600
C6-H6C	0.9600
C7-H7A	0.9700
C7-H7B	0.9700
C7-C8	1.498(3)
C8-H8	0.9300
C8-C9	1.329(3)
C9-C10	1.516(3)
C9-C17	1.473(3)
C10-H10	0.9800
C10-C11	1.517(3)
C11-C12	1.391(3)
C11-C16	1.397(3)
C12-H12	0.9300
C12-C13	1.389(3)
C13-H13	0.9300
C13-C14	1.385(4)
C14-H14	0.9300
C14-C15	1.386(4)
C15-H15	0.9300
C15-C16	1.386(3)
C16-H16	0.9300
C17-C18	1.493(3)
C18-C19	1.389(3)
C18-C23	1.393(3)
C19-H19	0.9300
C19-C20	1.382(3)

C20-H20	0.9300
C20-C21	1.378(4)
C21-H21	0.9300
C21-C22	1.378(4)
C22-H22	0.9300
C22-C23	1.396(3)
C23-H23	0.9300

C1-N1-C5	117.15(18)
C2-N1-C1	124.75(18)
C2-N1-C5	117.96(19)
C1-N2-C6	116.66(19)
C4-N2-C1	124.54(19)
C4-N2-C6	118.79(19)
O1-C1-N1	121.7(2)
O1-C1-N2	121.3(2)
N2-C1-N1	116.89(19)
O2-C2-N1	121.2(2)
O2-C2-C3	122.5(2)
N1-C2-C3	116.16(19)
C2-C3-C7	112.76(18)
C2-C3-C10	110.22(17)
C4-C3-C2	113.86(17)
C4-C3-C7	110.08(18)
C4-C3-C10	104.52(17)
C7-C3-C10	104.68(16)
O3-C4-N2	120.7(2)
O3-C4-C3	122.23(19)
N2-C4-C3	116.92(18)
N1-C5-H5A	109.5

N1-C5-H5B	109.5
N1-C5-H5C	109.5
H5A-C5-H5B	109.5
H5A-C5-H5C	109.5
H5B-C5-H5C	109.5
N2-C6-H6A	109.5
N2-C6-H6B	109.5
N2-C6-H6C	109.5
H6A-C6-H6B	109.5
H6A-C6-H6C	109.5
H6B-C6-H6C	109.5
C3-C7-H7A	110.9
C3-C7-H7B	110.9
H7A-C7-H7B	109.0
C8-C7-C3	104.12(17)
C8-C7-H7A	110.9
C8-C7-H7B	110.9
C7-C8-H8	123.7
C9-C8-C7	112.61(19)
C9-C8-H8	123.7
C8-C9-C10	112.98(18)
C8-C9-C17	126.34(19)
C17-C9-C10	120.48(18)
C3-C10-H10	109.3
C9-C10-C3	101.10(16)
C9-C10-H10	109.3
C9-C10-C11	112.32(17)
C11-C10-C3	115.31(17)
C11-C10-H10	109.3
C12-C11-C10	119.31(19)

C12-C11-C16	118.9(2)
C16-C11-C10	121.79(19)
C11-C12-H12	119.7
C13-C12-C11	120.5(2)
C13-C12-H12	119.7
C12-C13-H13	119.9
C14-C13-C12	120.2(2)
C14-C13-H13	119.9
C13-C14-H14	120.2
C13-C14-C15	119.6(2)
C15-C14-H14	120.2
C14-C15-H15	119.8
C16-C15-C14	120.4(2)
C16-C15-H15	119.8
C11-C16-H16	119.8
C15-C16-C11	120.3(2)
C15-C16-H16	119.8
O4-C17-C9	119.76(19)
O4-C17-C18	120.06(19)
C9-C17-C18	120.17(18)
C19-C18-C17	119.0(2)
C19-C18-C23	119.6(2)
C23-C18-C17	121.26(19)
C18-C19-H19	119.7
C20-C19-C18	120.6(2)
C20-C19-H19	119.7
C19-C20-H20	120.1
C21-C20-C19	119.8(2)
C21-C20-H20	120.1
C20-C21-H21	119.8

C20-C21-C22	120.4(2)
C22-C21-H21	119.8
C21-C22-H22	119.9
C21-C22-C23	120.3(2)
C23-C22-H22	119.9
C18-C23-C22	119.3(2)
C18-C23-H23	120.4
C22-C23-H23	120.4

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2$ x 10^3) for **4ma**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O1	41(1)	41(1)	35(1)	6(1)	13(1)	-6(1)
O2	33(1)	29(1)	45(1)	-7(1)	10(1)	-2(1)
O3	29(1)	53(1)	26(1)	-4(1)	1(1)	-5(1)
O4	32(1)	27(1)	43(1)	5(1)	13(1)	3(1)
N1	26(1)	33(1)	24(1)	-3(1)	6(1)	-1(1)
N2	22(1)	34(1)	28(1)	-1(1)	2(1)	-5(1)
C1	23(1)	32(1)	28(1)	-1(1)	5(1)	1(1)
C2	21(1)	27(1)	31(1)	0(1)	4(1)	3(1)
C3	21(1)	26(1)	26(1)	1(1)	4(1)	2(1)
C4	20(1)	33(1)	24(1)	2(1)	4(1)	1(1)
C5	40(1)	46(1)	29(1)	-8(1)	9(1)	-2(1)
C6	31(1)	44(1)	40(1)	-4(1)	2(1)	-12(1)
C7	28(1)	29(1)	33(1)	6(1)	9(1)	6(1)
C8	23(1)	28(1)	27(1)	3(1)	5(1)	-2(1)

C9	19(1)	25(1)	27(1)	0(1)	5(1)	-3(1)
C10	18(1)	22(1)	28(1)	1(1)	4(1)	2(1)
C11	21(1)	25(1)	27(1)	0(1)	5(1)	3(1)
C12	26(1)	31(1)	33(1)	4(1)	4(1)	-1(1)
C13	32(1)	43(1)	31(1)	6(1)	1(1)	1(1)
C14	28(1)	46(1)	31(1)	-2(1)	0(1)	0(1)
C15	31(1)	38(1)	38(1)	-2(1)	2(1)	-8(1)
C16	31(1)	32(1)	30(1)	4(1)	4(1)	-3(1)
C17	21(1)	25(1)	26(1)	-2(1)	2(1)	0(1)
C18	20(1)	28(1)	28(1)	2(1)	3(1)	4(1)
C19	24(1)	35(1)	31(1)	-2(1)	2(1)	2(1)
C20	30(1)	53(2)	29(1)	-2(1)	8(1)	7(1)
C21	26(1)	53(2)	37(1)	14(1)	11(1)	6(1)
C22	24(1)	37(1)	51(1)	13(1)	11(1)	0(1)
C23	25(1)	29(1)	42(1)	1(1)	9(1)	2(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **4ma**.

	x	y	z	U(eq)
H5A	89	8187	7309	57
H5B	860	10506	7202	57
H5C	1838	8415	7371	57
H6A	-563	1922	6358	58
H6B	-1051	3211	5981	58
H6C	-1902	3691	6328	58
H7A	1402	9966	5751	35
H7B	2581	11338	6028	35

H8	3830	9348	5511	31
H10	3378	4904	6309	27
H12	4233	4593	6952	36
H13	5859	5640	7469	43
H14	7391	8876	7447	43
H15	7194	11180	6917	43
H16	5557	10168	6401	37
H19	6306	3326	5221	36
H20	7985	4486	4820	44
H21	9122	8092	4893	46
H22	8523	10604	5351	44
H23	6737	9540	5737	38

Table 6. Torsion angles [°] for **4ma**.

O2-C2-C3-C4	-154.8(2)
O2-C2-C3-C7	-28.4(3)
O2-C2-C3-C10	88.2(2)
O4-C17-C18-C19	35.9(3)
O4-C17-C18-C23	-140.4(2)
N1-C2-C3-C4	28.6(3)
N1-C2-C3-C7	155.00(19)
N1-C2-C3-C10	-88.4(2)
C1-N1-C2-O2	170.0(2)
C1-N1-C2-C3	-13.4(3)
C1-N2-C4-O3	-178.5(2)
C1-N2-C4-C3	5.3(3)
C2-N1-C1-O1	176.7(2)
C2-N1-C1-N2	-7.2(3)

C2-C3-C4-O3	158.9(2)
C2-C3-C4-N2	-25.0(3)
C2-C3-C7-C8	138.95(18)
C2-C3-C10-C9	-141.98(18)
C2-C3-C10-C11	-20.6(2)
C3-C7-C8-C9	-10.7(3)
C3-C10-C11-C12	101.8(2)
C3-C10-C11-C16	-77.5(2)
C4-N2-C1-O1	-172.3(2)
C4-N2-C1-N1	11.6(3)
C4-C3-C7-C8	-92.7(2)
C4-C3-C10-C9	95.29(18)
C4-C3-C10-C11	-143.32(18)
C5-N1-C1-O1	-7.8(3)
C5-N1-C1-N2	168.3(2)
C5-N1-C2-O2	-5.5(3)
C5-N1-C2-C3	171.2(2)
C6-N2-C1-O1	6.3(3)
C6-N2-C1-N1	-169.8(2)
C6-N2-C4-O3	2.9(3)
C6-N2-C4-C3	-173.25(19)
C7-C3-C4-O3	31.2(3)
C7-C3-C4-N2	-152.70(18)
C7-C3-C10-C9	-20.5(2)
C7-C3-C10-C11	100.9(2)
C7-C8-C9-C10	-3.5(3)
C7-C8-C9-C17	171.3(2)
C8-C9-C10-C3	15.3(2)
C8-C9-C10-C11	-108.2(2)
C8-C9-C17-O4	-148.9(2)
C8-C9-C17-C18	29.5(3)
C9-C10-C11-C12	-143.13(19)

C9-C10-C11-C16	37.6(3)
C9-C17-C18-C19	-142.6(2)
C9-C17-C18-C23	41.2(3)
C10-C3-C4-O3	-80.7(2)
C10-C3-C4-N2	95.4(2)
C10-C3-C7-C8	19.1(2)
C10-C9-C17-O4	25.5(3)
C10-C9-C17-C18	-156.04(19)
C10-C11-C12-C13	-179.1(2)
C10-C11-C16-C15	179.4(2)
C11-C12-C13-C14	-1.1(4)
C12-C11-C16-C15	0.1(3)
C12-C13-C14-C15	1.7(4)
C13-C14-C15-C16	-1.4(4)
C14-C15-C16-C11	0.5(4)
C16-C11-C12-C13	0.2(3)
C17-C9-C10-C3	-159.85(18)
C17-C9-C10-C11	76.7(2)
C17-C18-C19-C20	-175.9(2)
C17-C18-C23-C22	174.0(2)
C18-C19-C20-C21	1.4(4)
C19-C18-C23-C22	-2.2(3)
C19-C20-C21-C22	-1.4(4)
C20-C21-C22-C23	-0.4(4)
C21-C22-C23-C18	2.2(4)
C23-C18-C19-C20	0.4(3)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for **4ma** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)