

Supporting Information

***cis*-2,3-Disubstituted Cyclopropane 1,1-Diesters in [3+2] Annulations with Aldehydes: Highly Diastereoselective Construction of Densely Substituted Tetrahydrofurans**

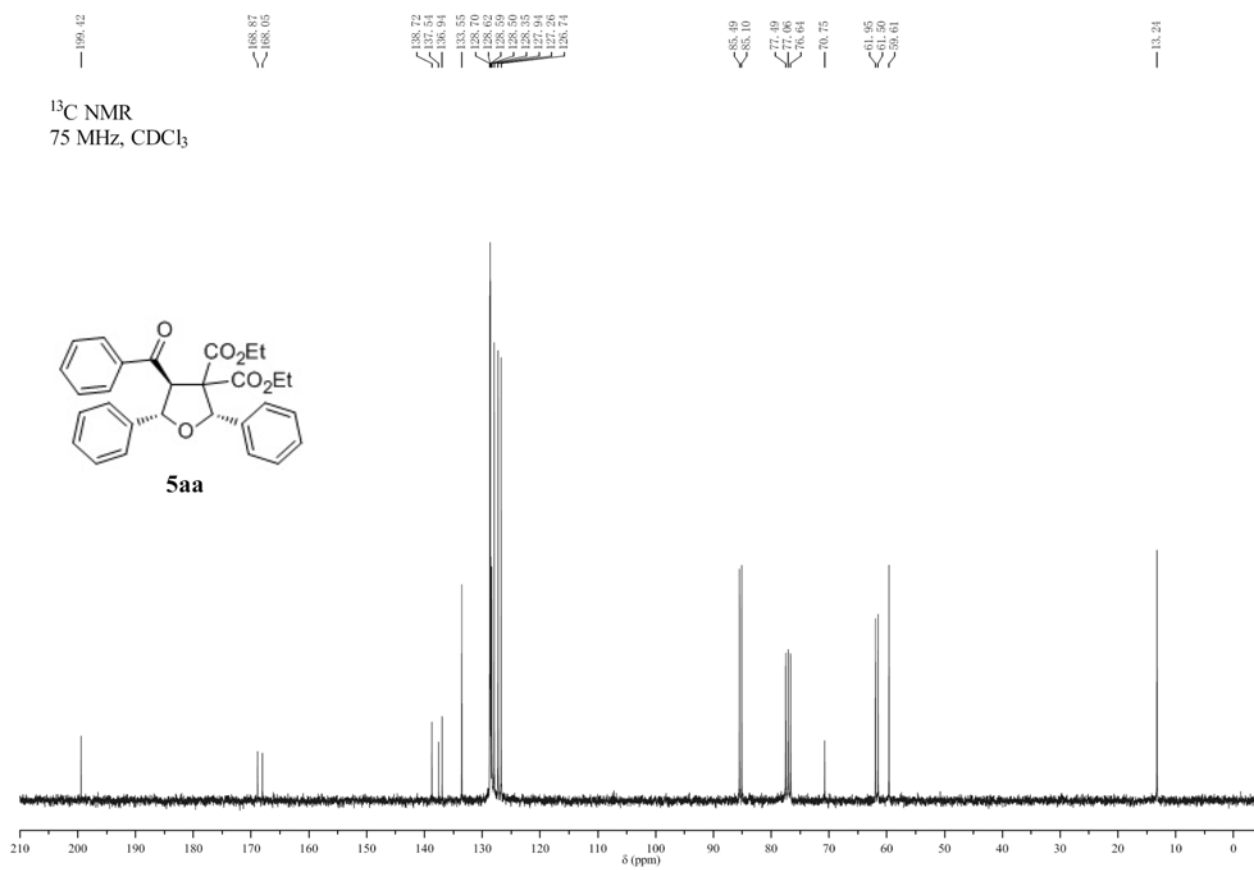
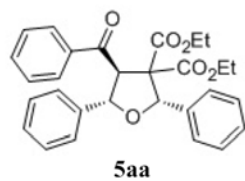
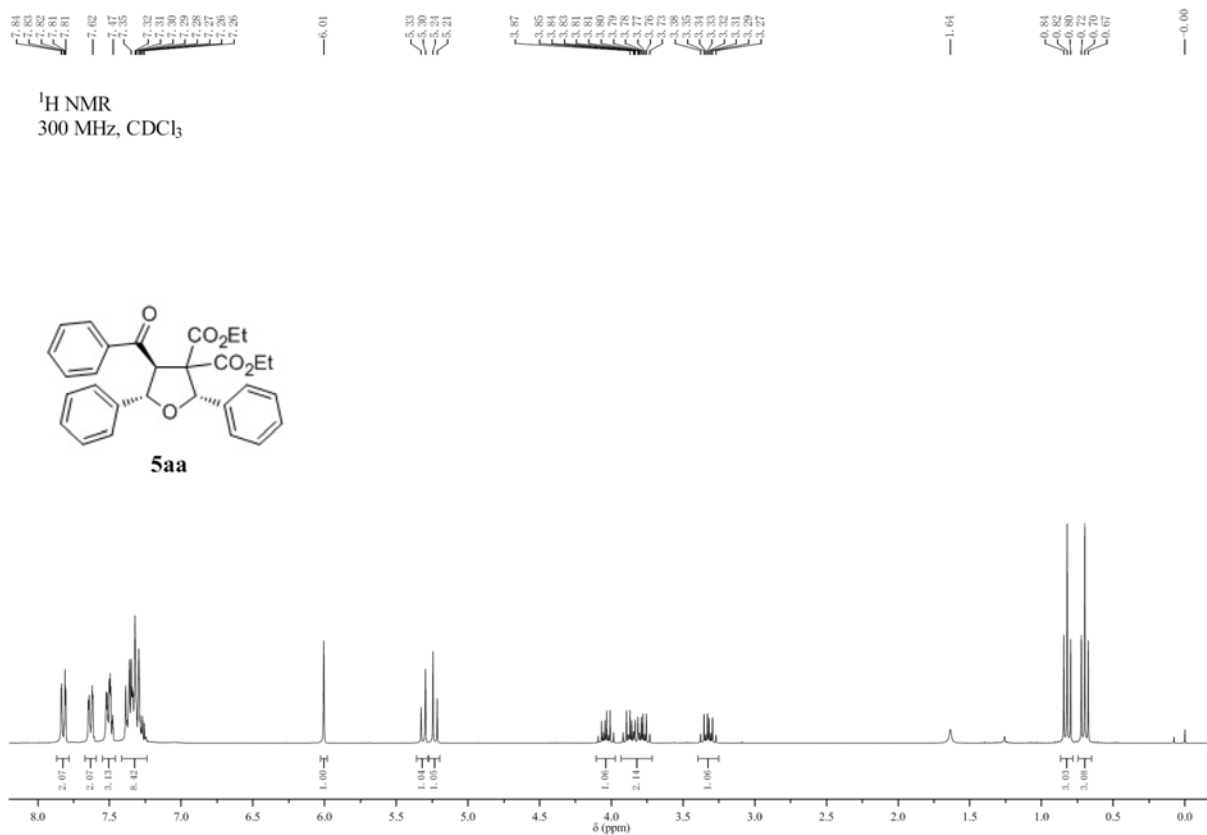
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Laboratory of Functionalized Molecular Solids, Ministry of Education, Anhui Laboratory of Molecule-
Based Materials, Institute of Organic Chemistry, College of Chemistry and Materials Science, Anhui
Normal University, Wuhu, Anhui 241000, China

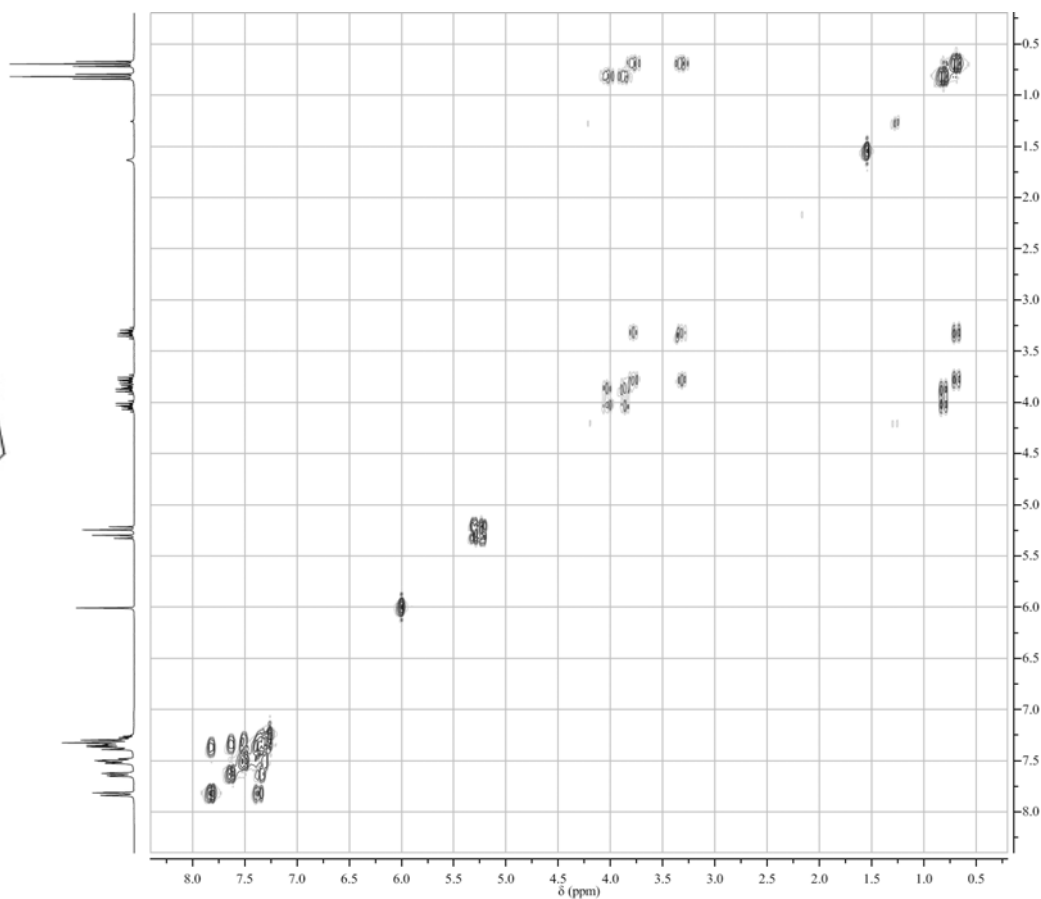
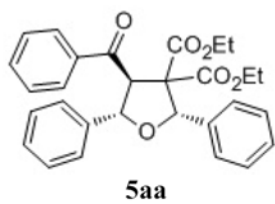
gshyang@mail.ahnu.edu.cn; chaizhuo@mail.ahnu.edu.cn

Content

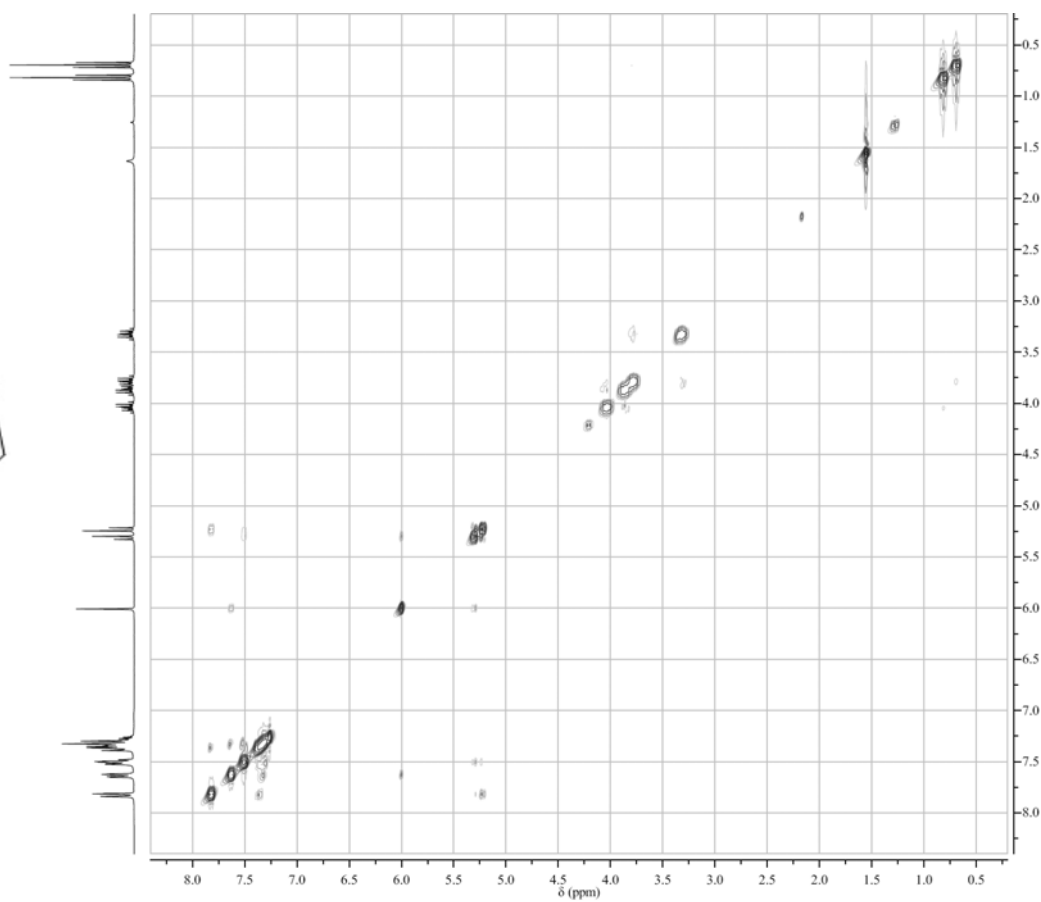
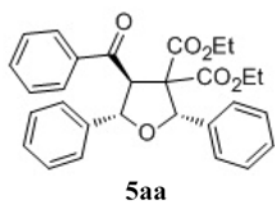
¹ H, ¹³ C, COSY and NOESY NMR Spectra of all products.....	S2–S66
X-ray crystallographic analysis of 5ac	S67–S68

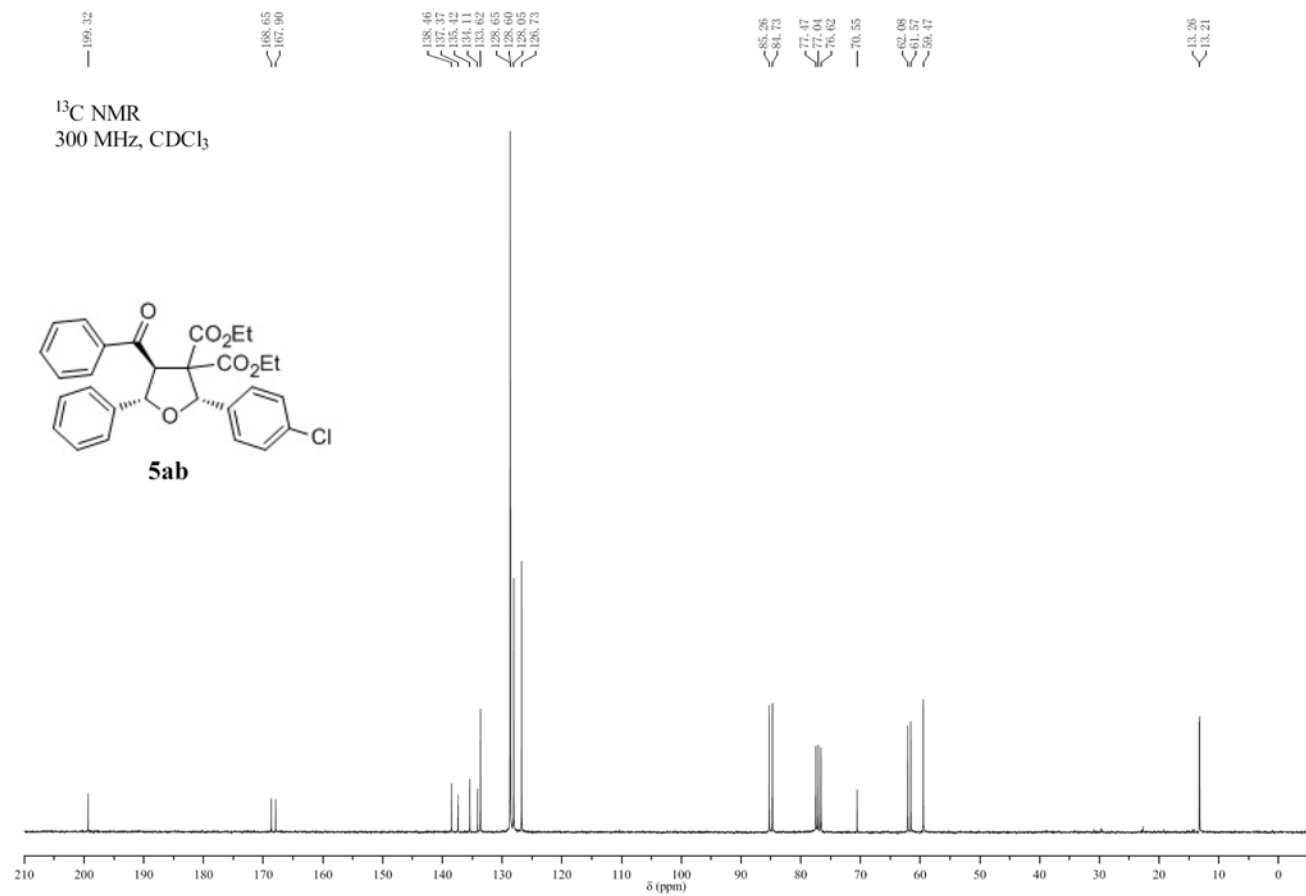
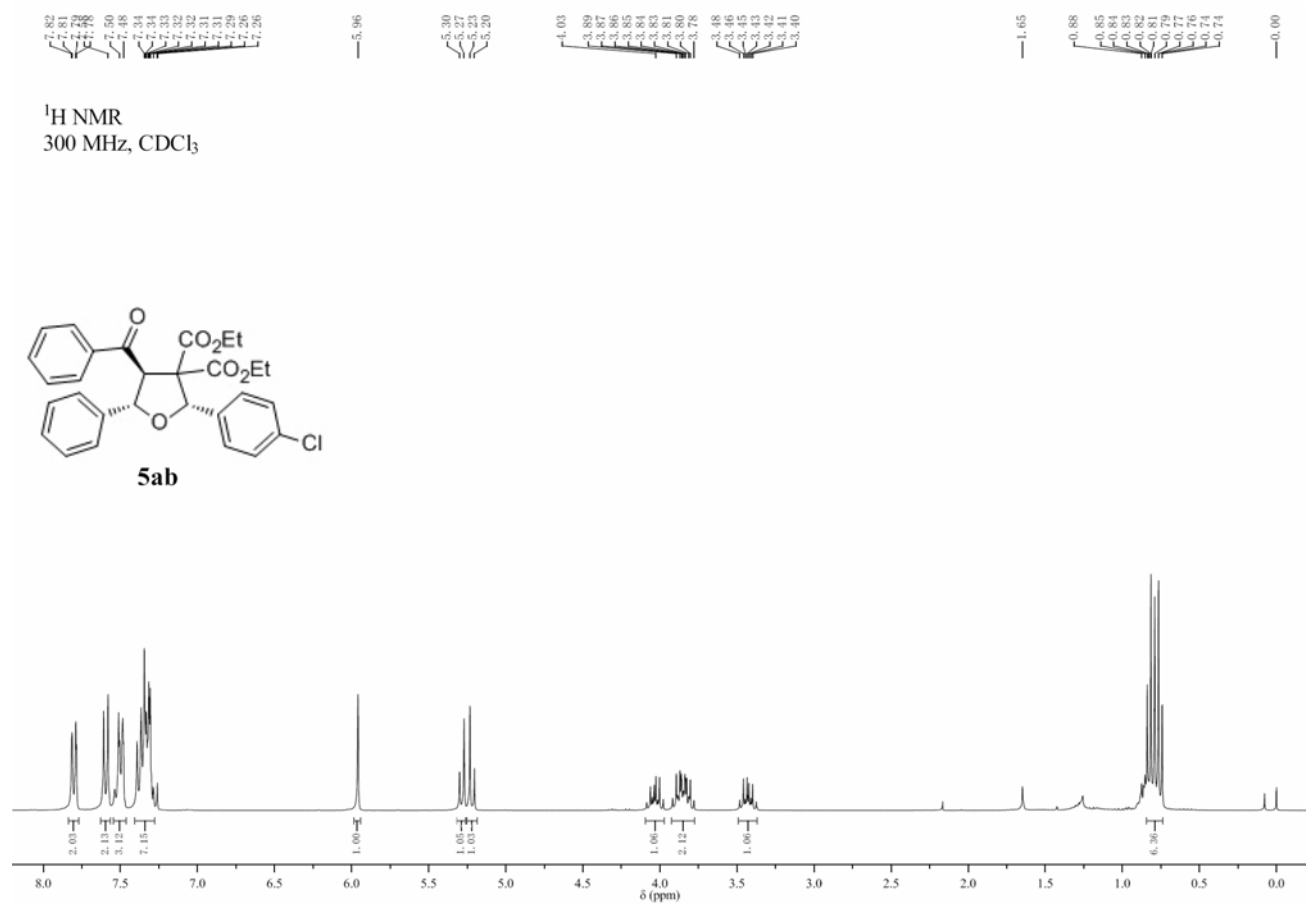
¹H, ¹³C, COSY and NOESY NMR Spectra of all products

2D COSY NMR
300 MHz, CDCl₃

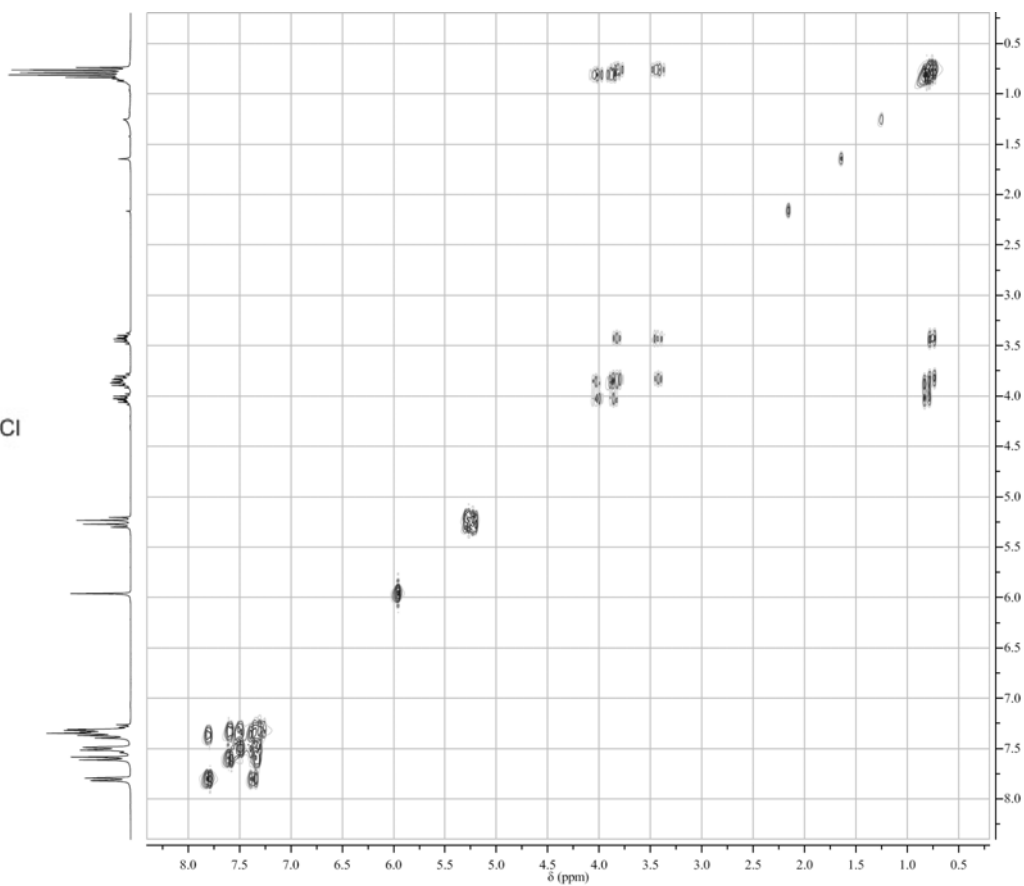
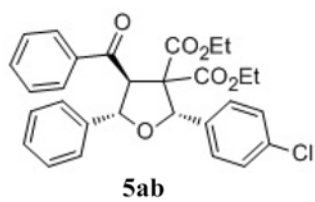


2D NOESY NMR
300 MHz, CDCl₃

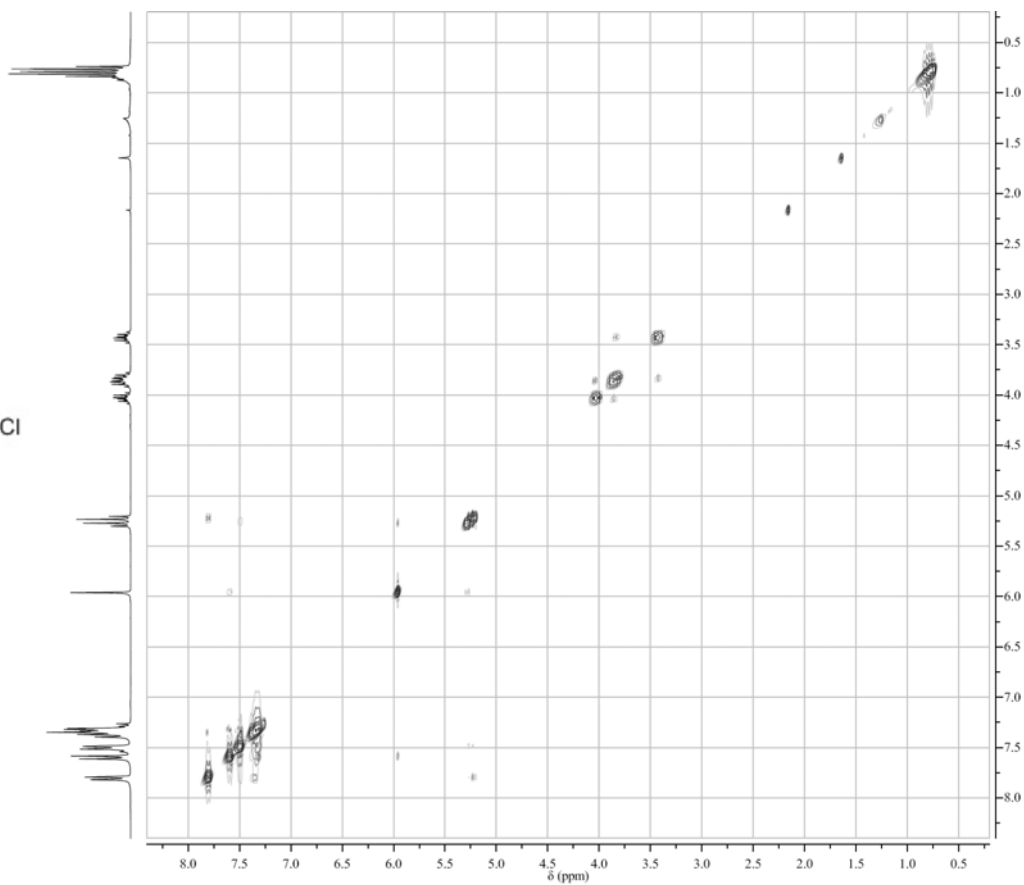
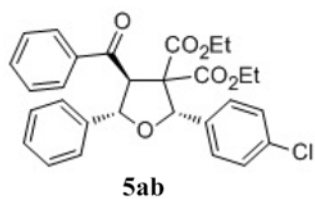


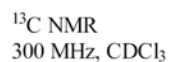
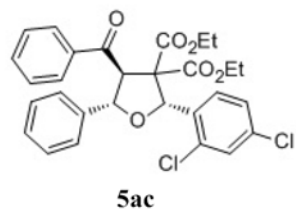
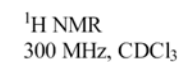


2D COSY NMR
300 MHz, CDCl₃

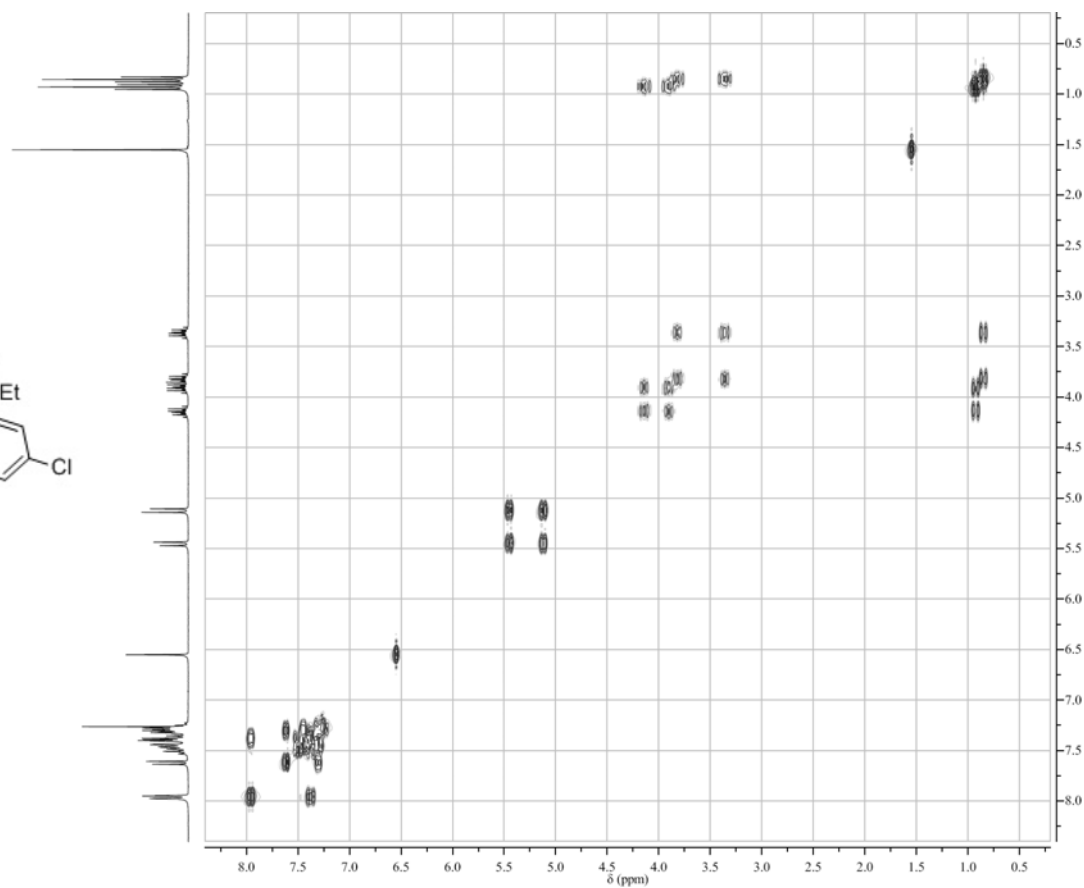
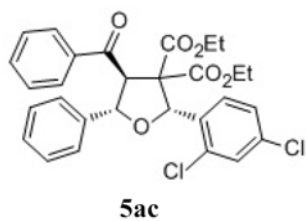


2D NOESY NMR
300 MHz, CDCl₃

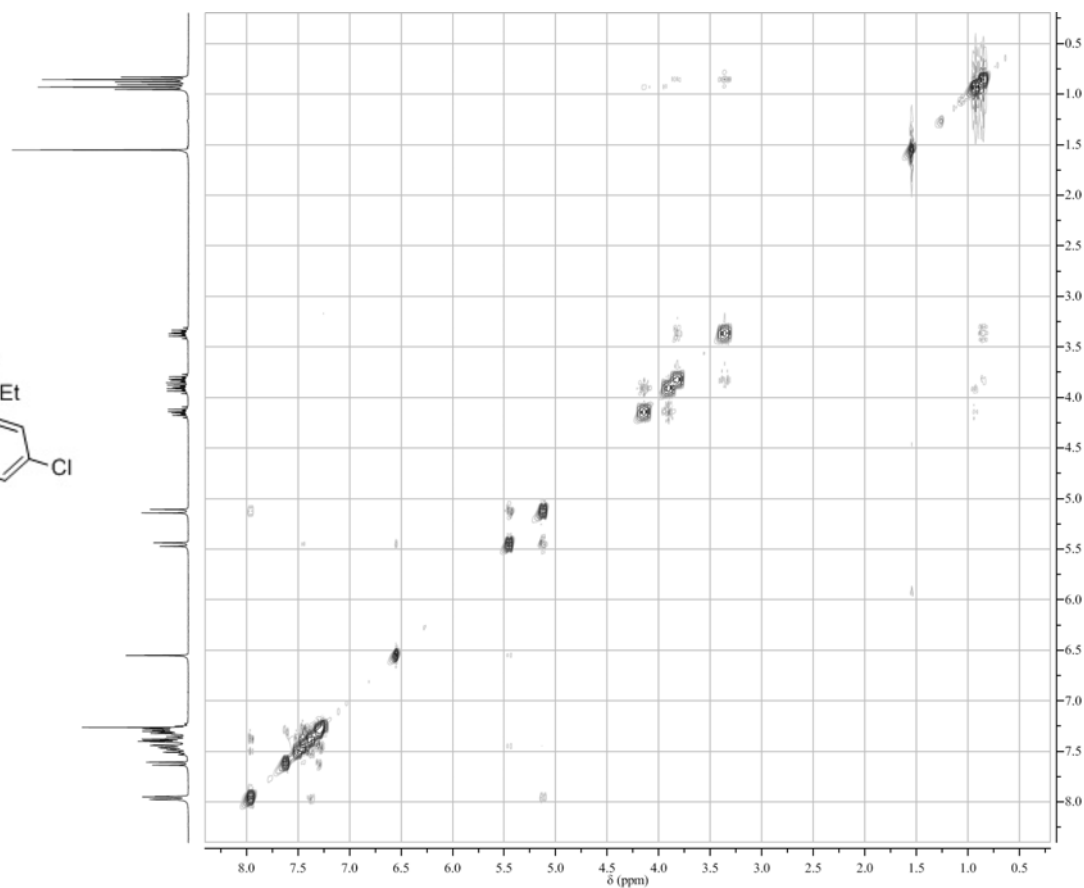
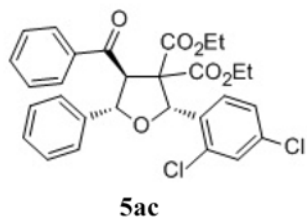


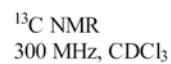
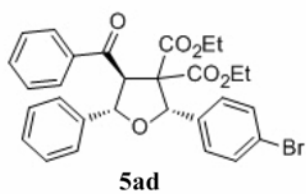
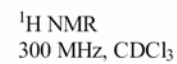


2D COSY NMR
300 MHz, CDCl₃

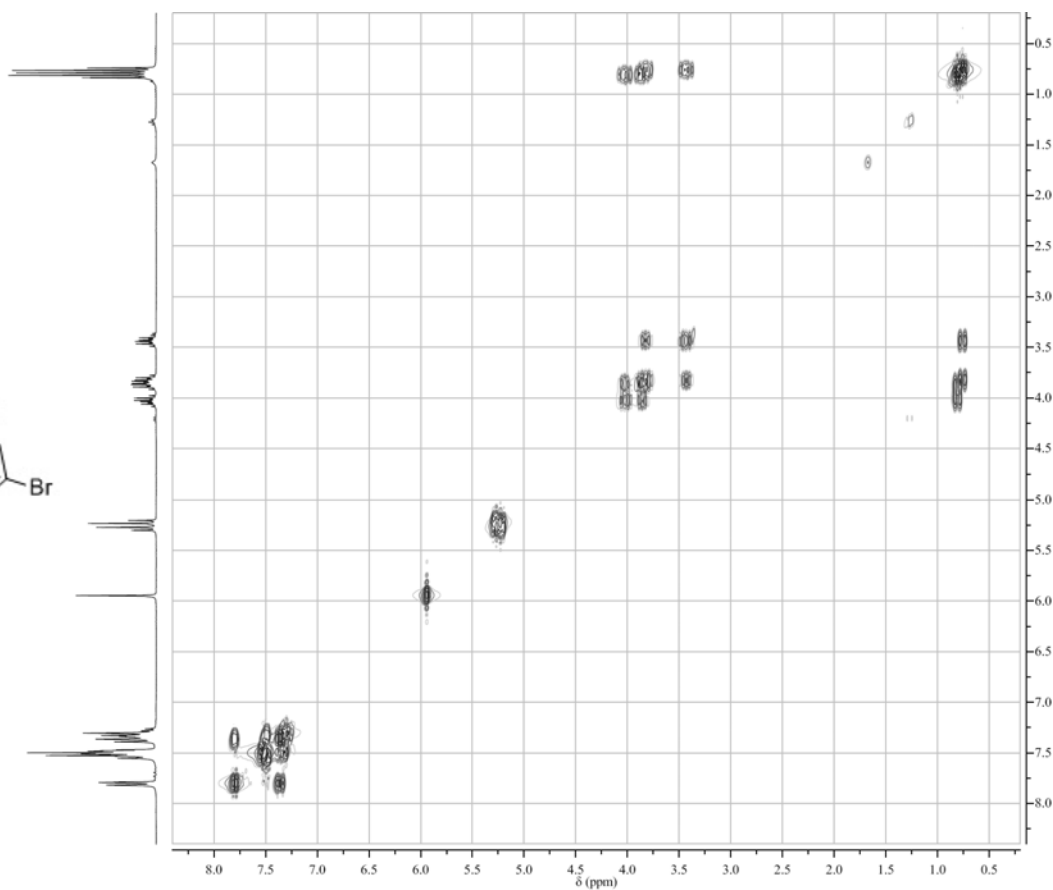
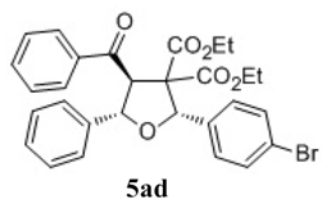


2D NOESY NMR
300 MHz, CDCl₃

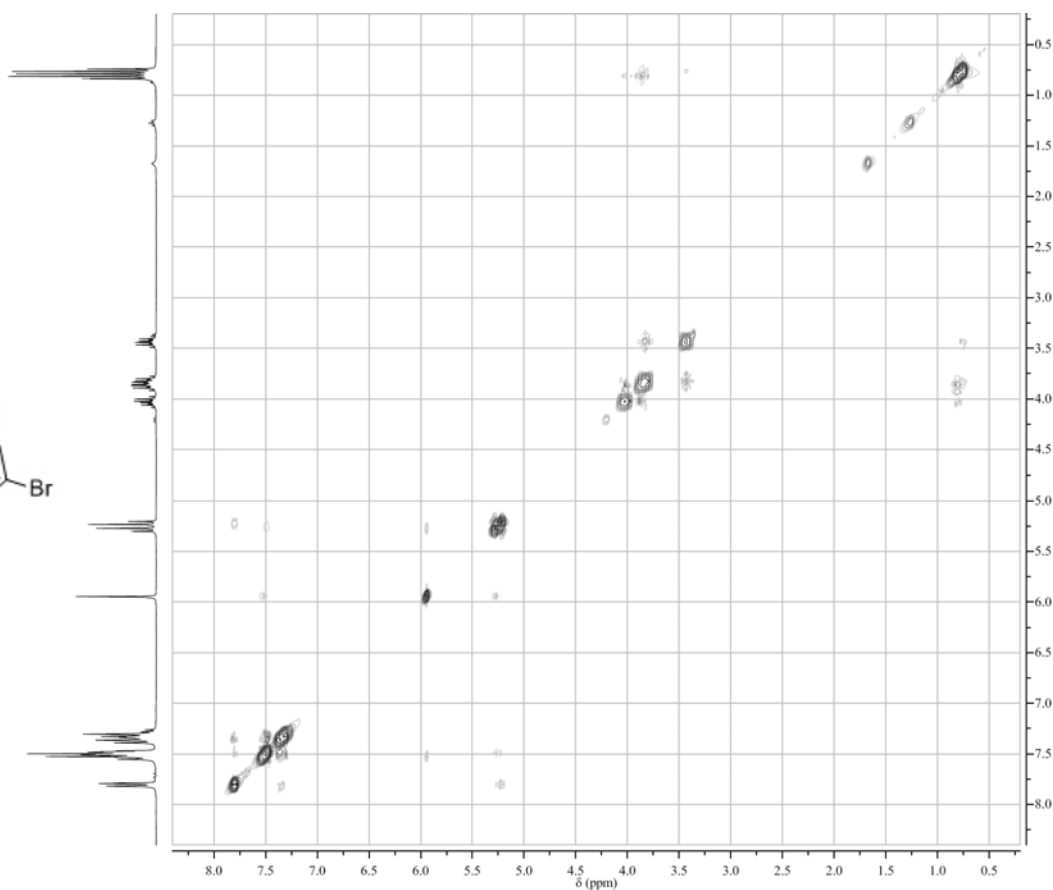
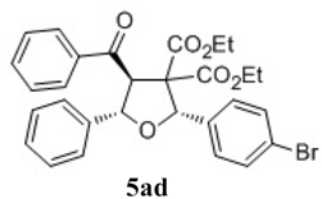


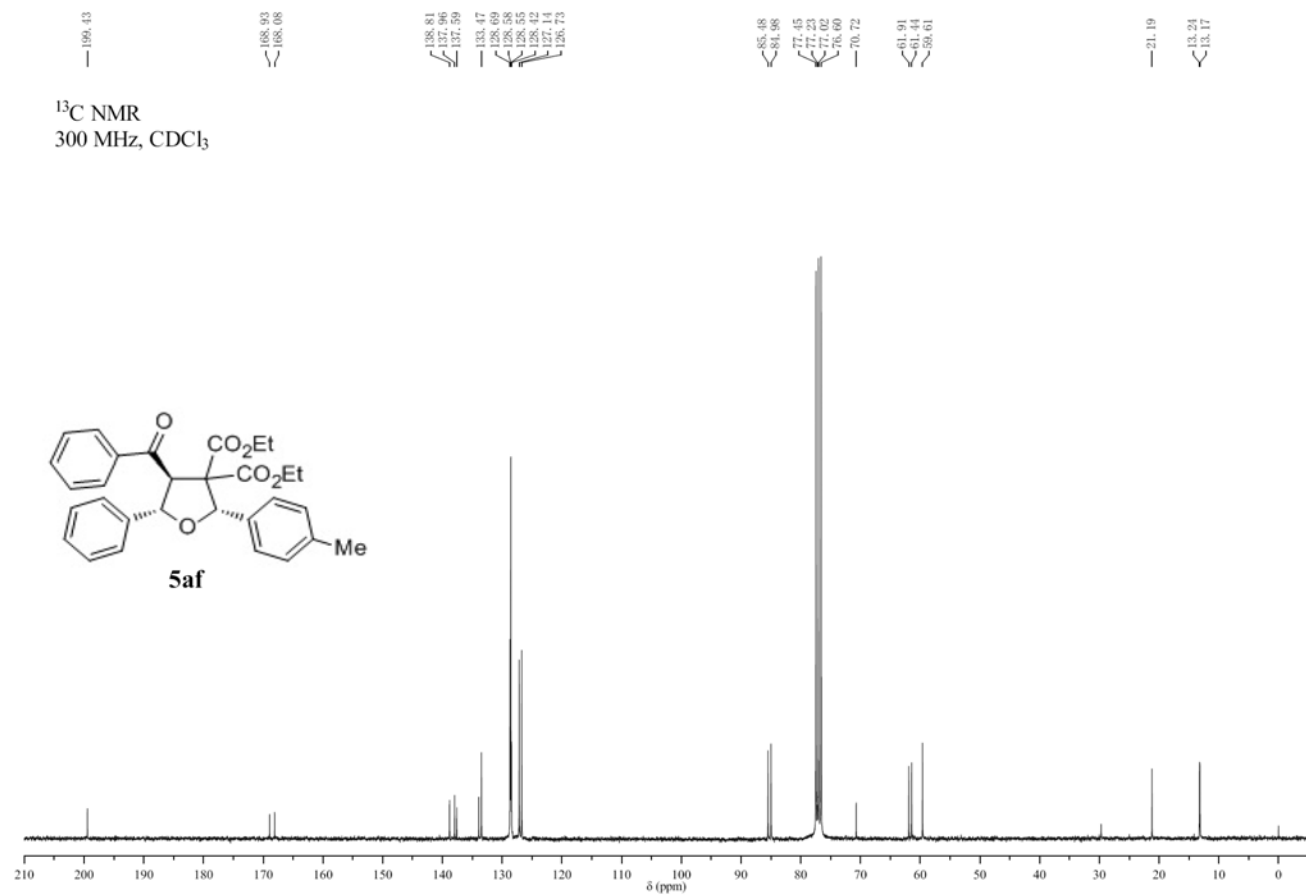
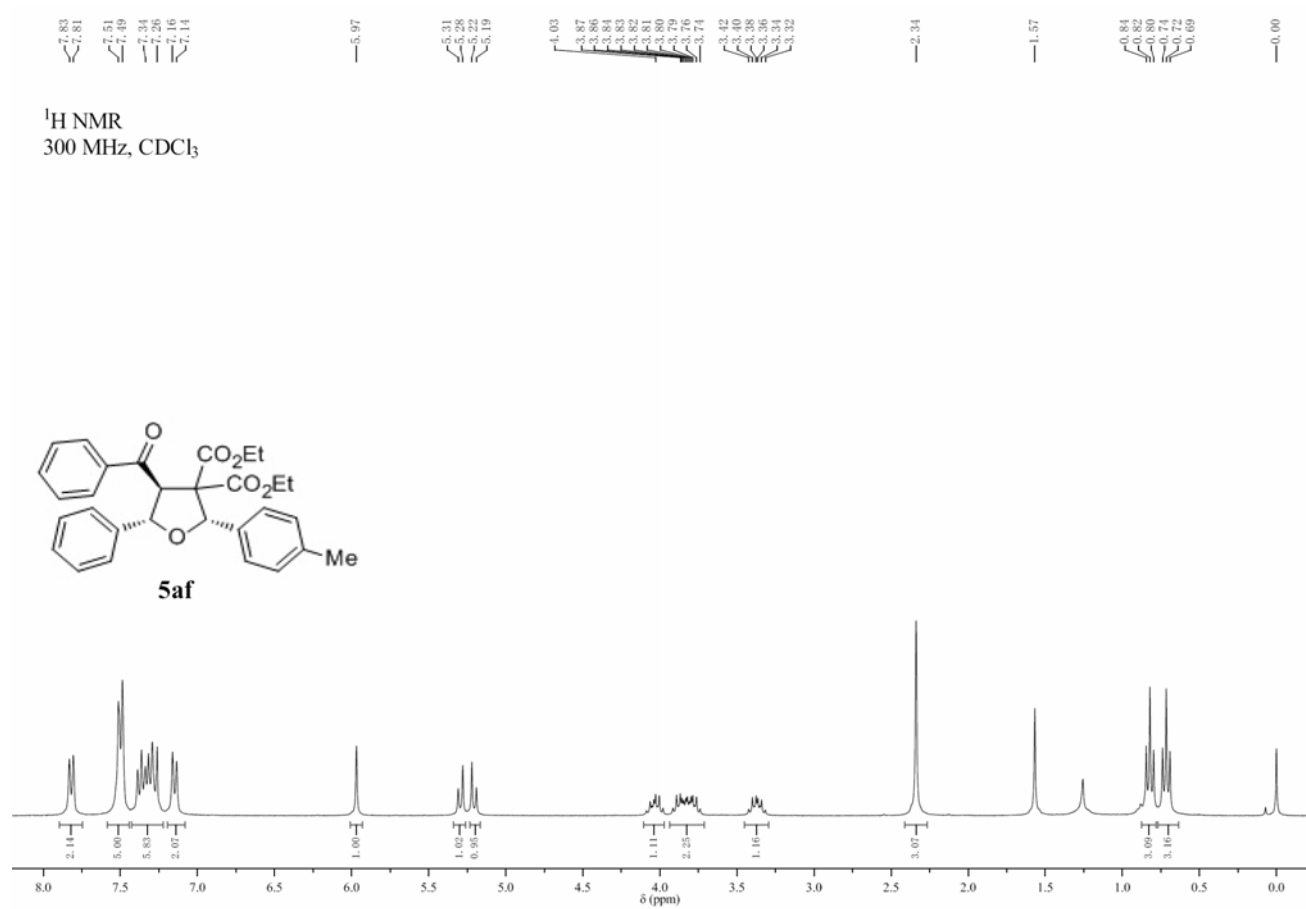


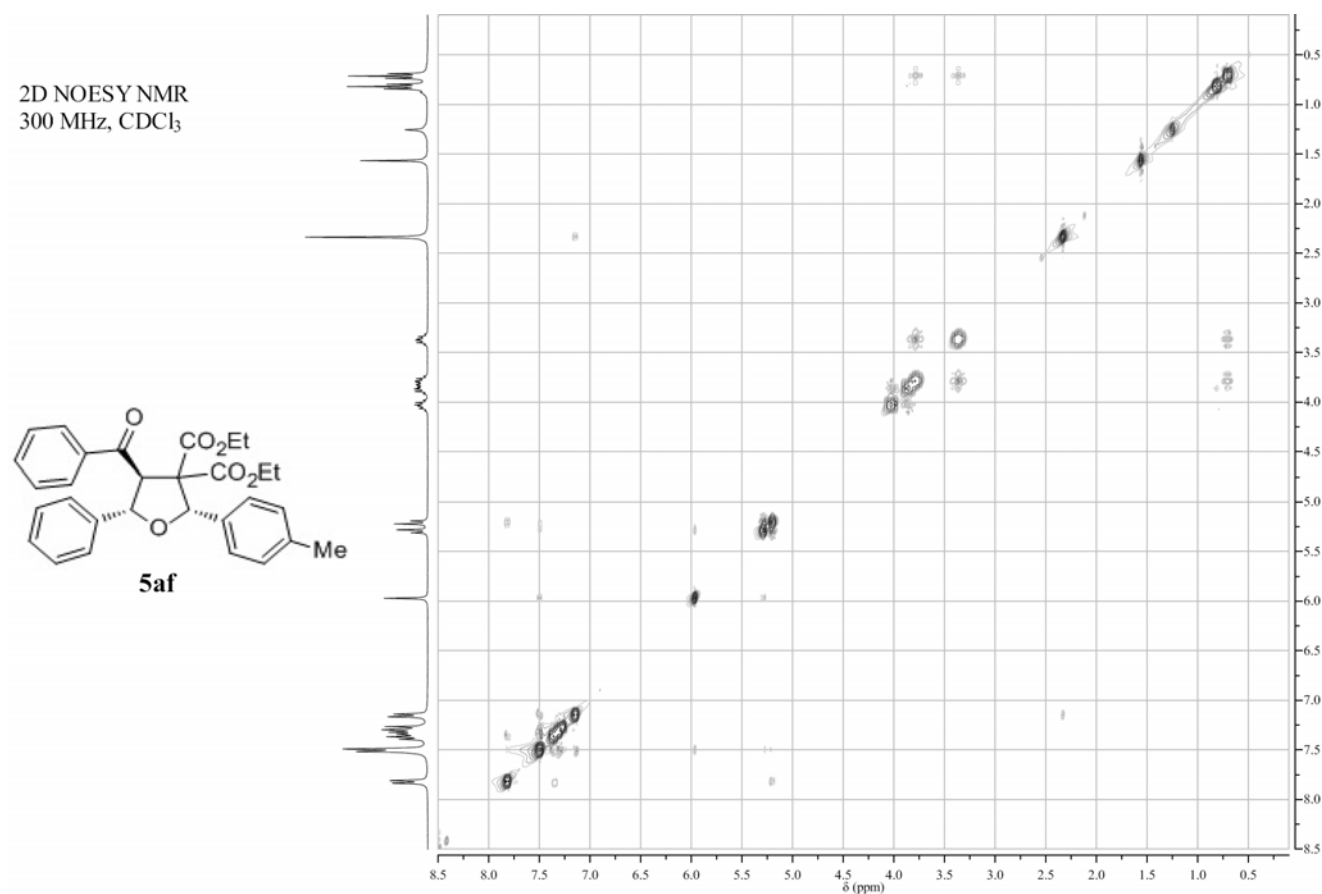
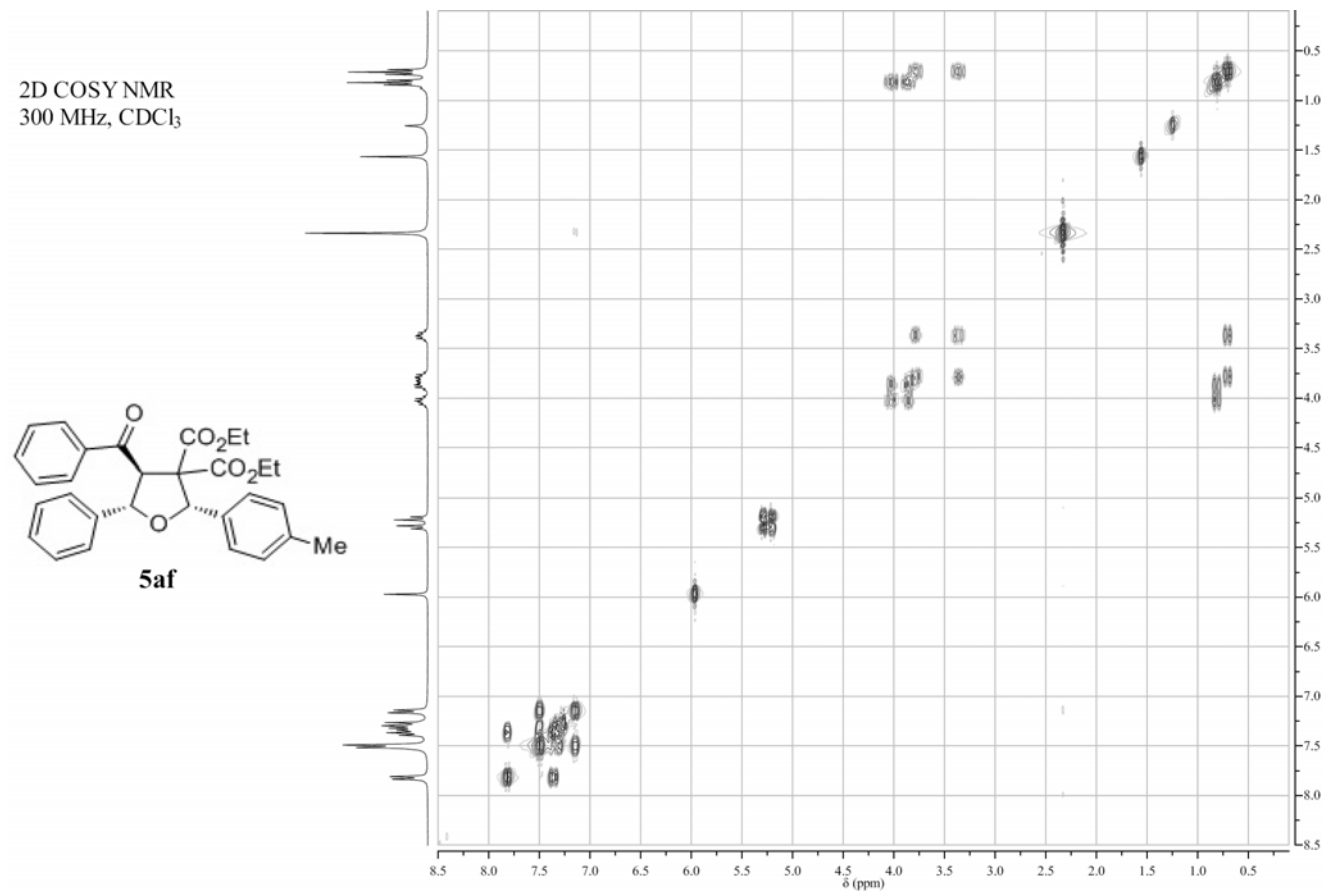
2D COSY NMR
300 MHz, CDCl₃

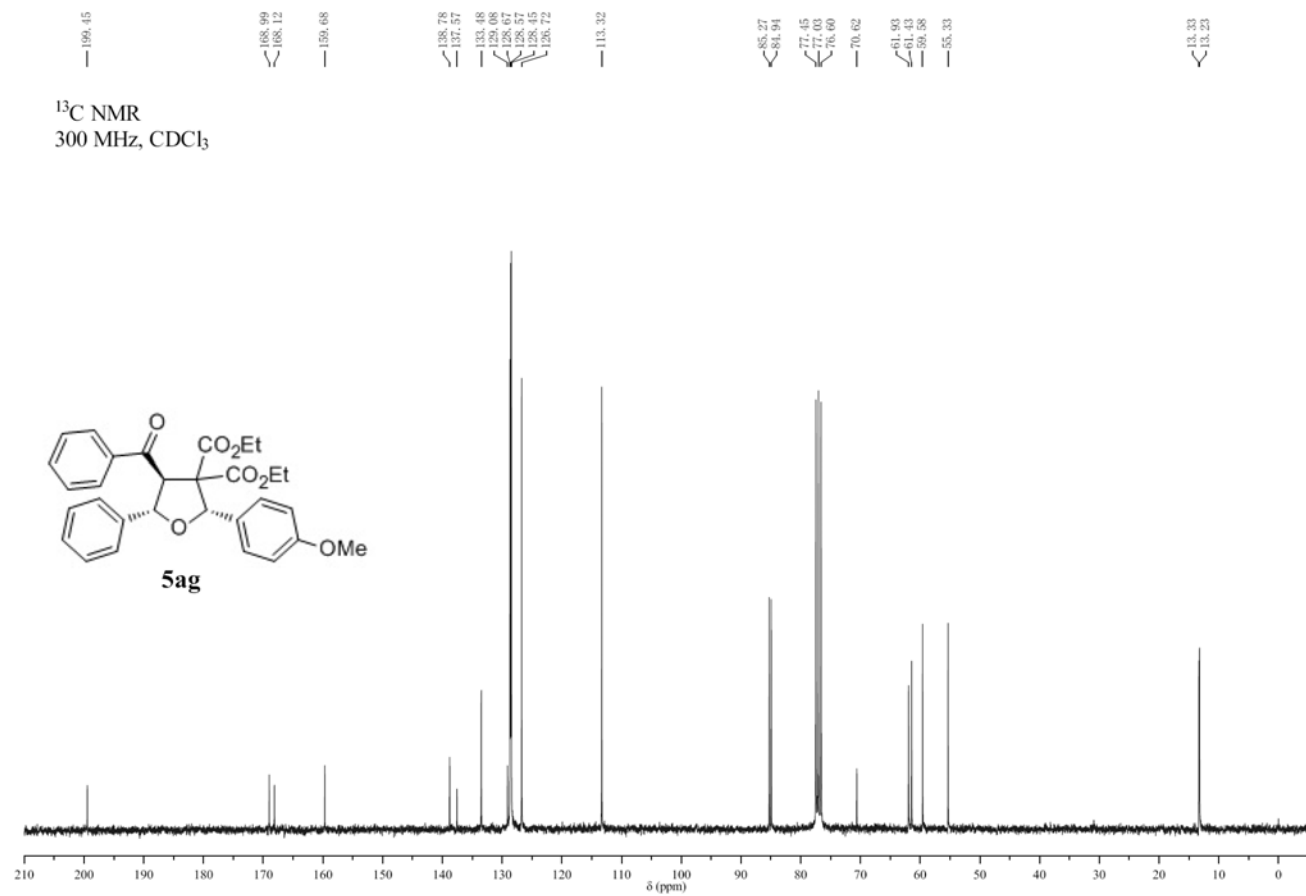
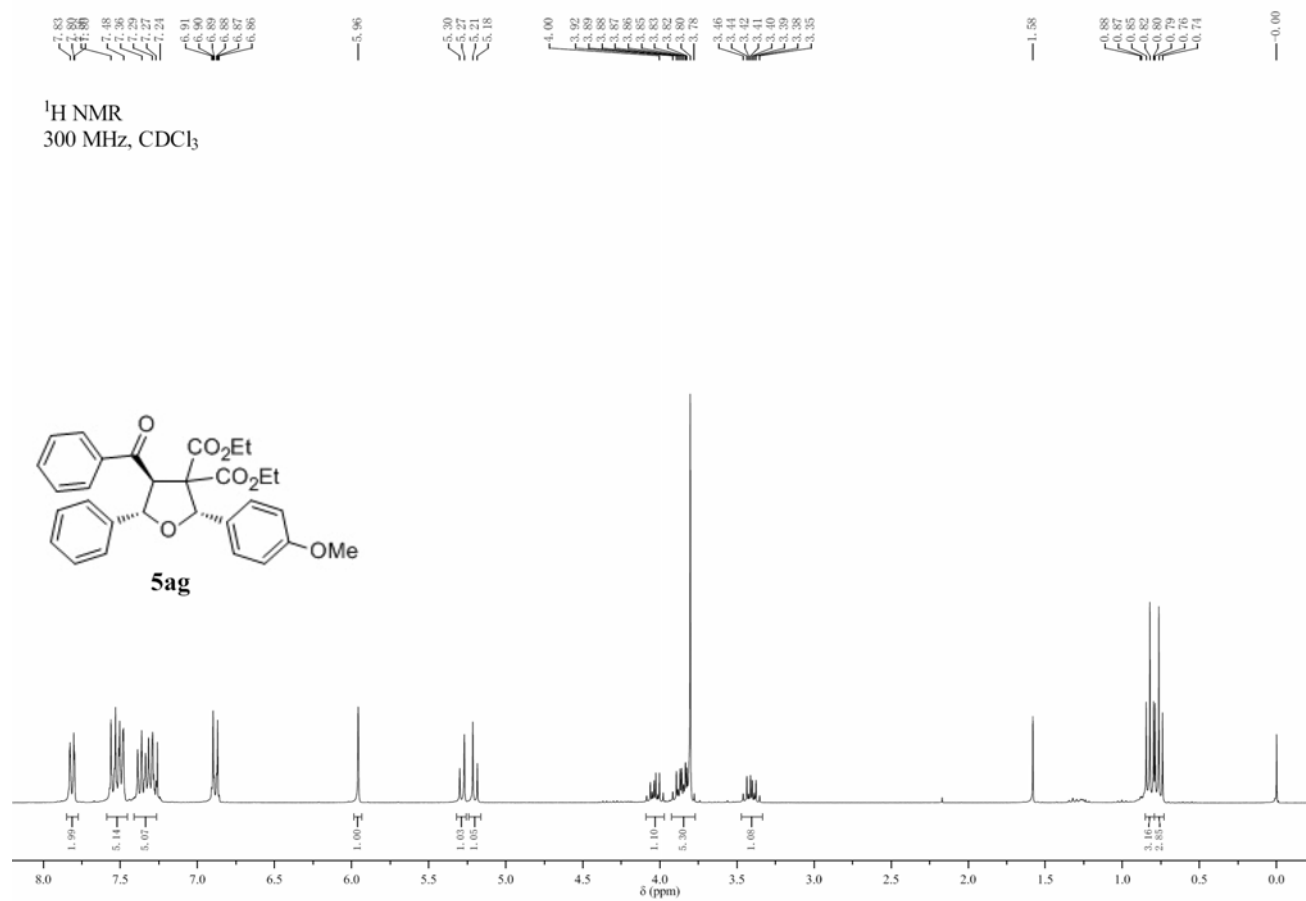


2D NOESY NMR
300 MHz, CDCl₃

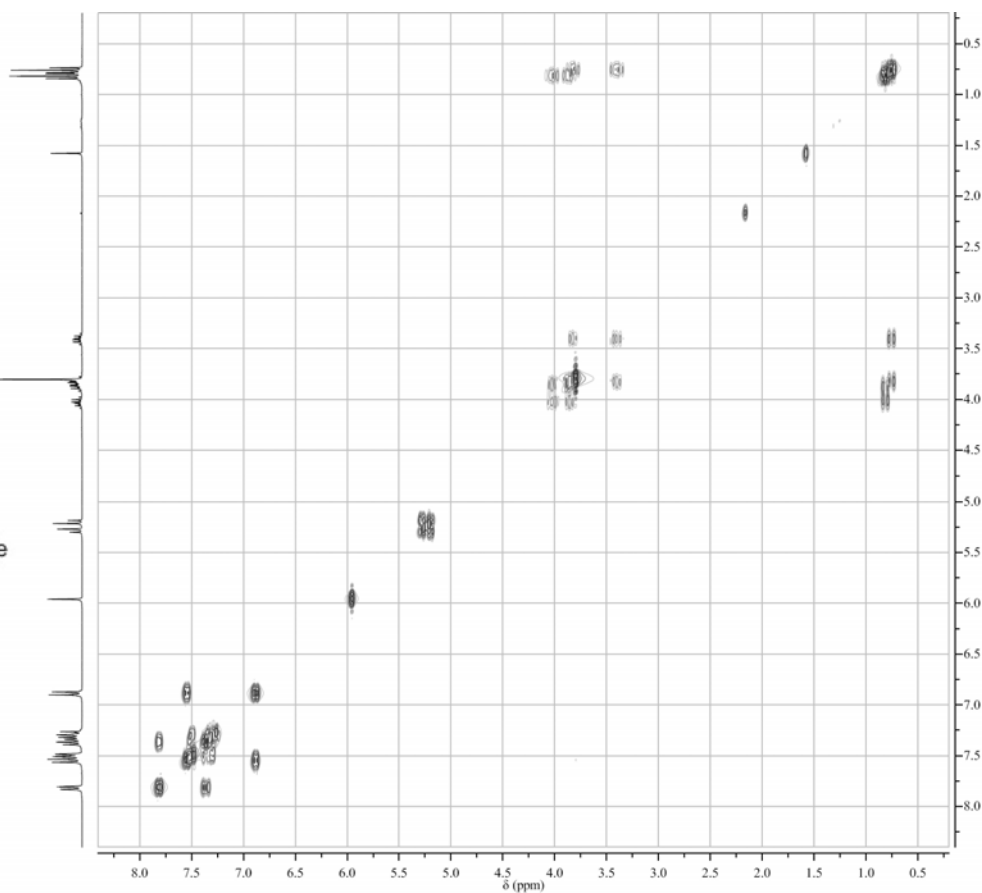
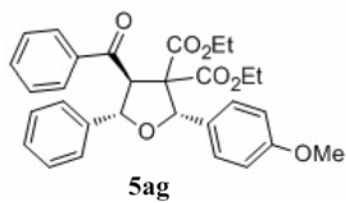




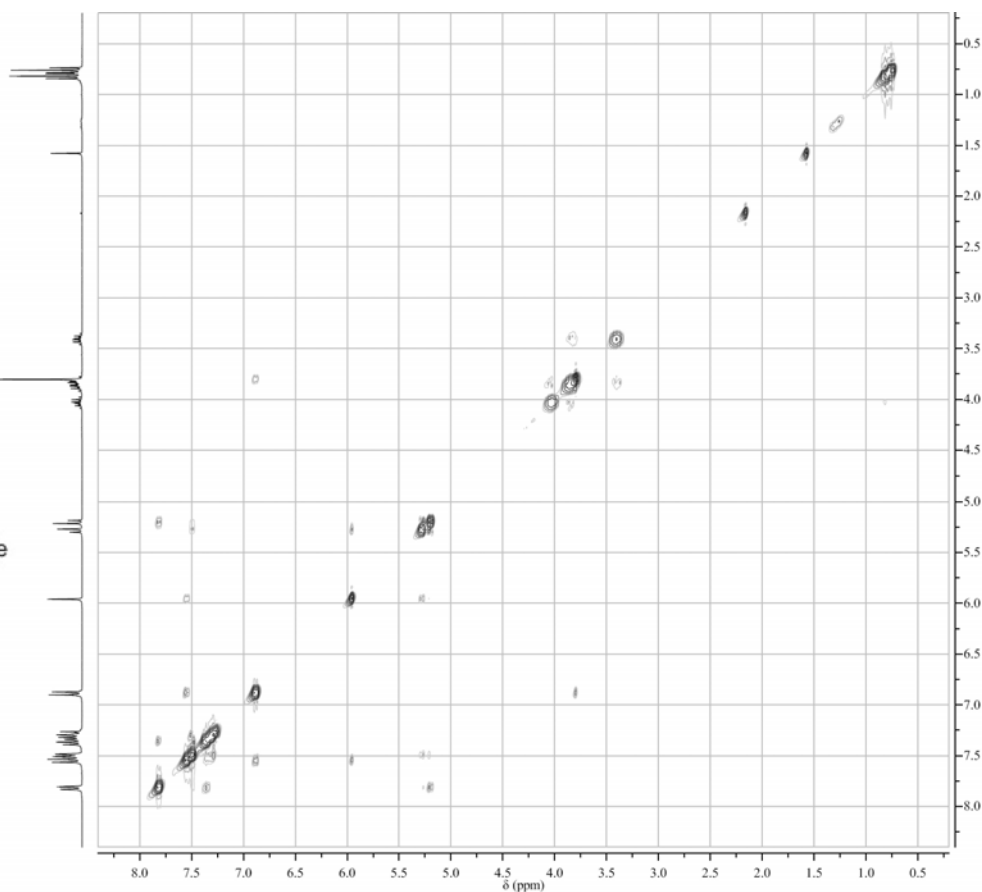
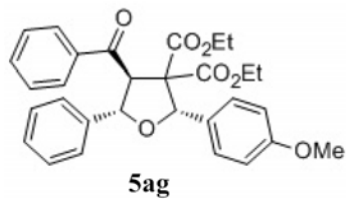




2D COSY NMR
300 MHz, CDCl₃



2D NOESY NMR
300 MHz, CDCl₃



7.83
7.81
7.80
7.51
7.40
7.35
7.33
7.26
7.19
7.16
6.87
6.84

— 5.95

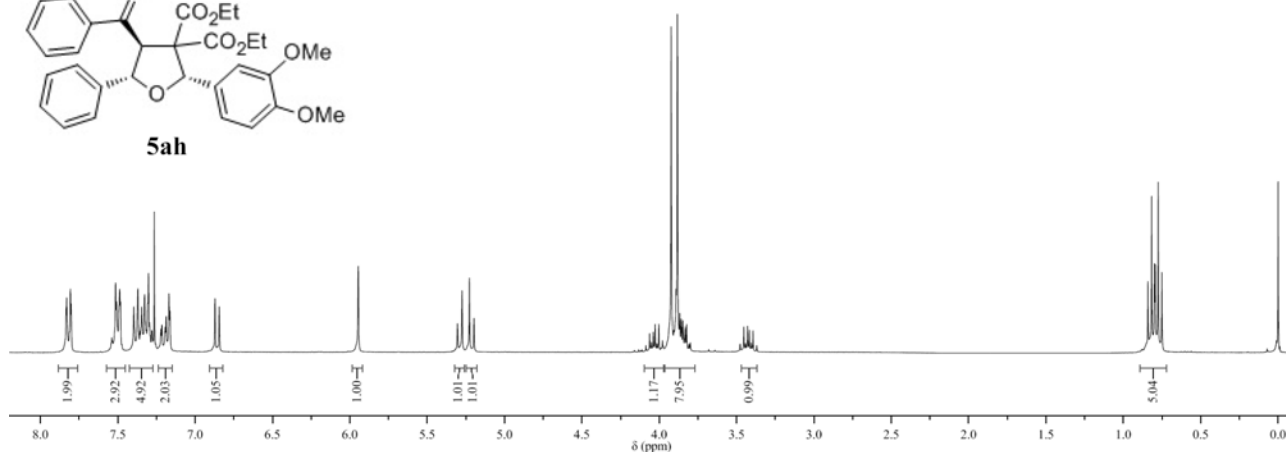
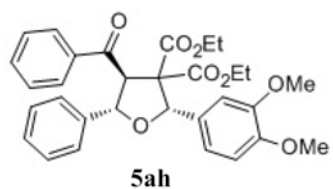
5.30
5.27
5.23
5.20

3.92
3.91
3.89
3.88
3.87
3.86
3.85
3.83
3.82
3.45
3.43
3.42
3.39

0.84
0.82
0.80
0.79
0.77
0.75

— 0.00

^1H NMR
300 MHz, CDCl_3



199.39

169.00
168.08

148.97
148.44

138.67
137.50

133.51

128.65
128.57
126.72

119.74

110.57
110.51
110.37
110.29

85.33
84.93

77.44
77.01
76.59

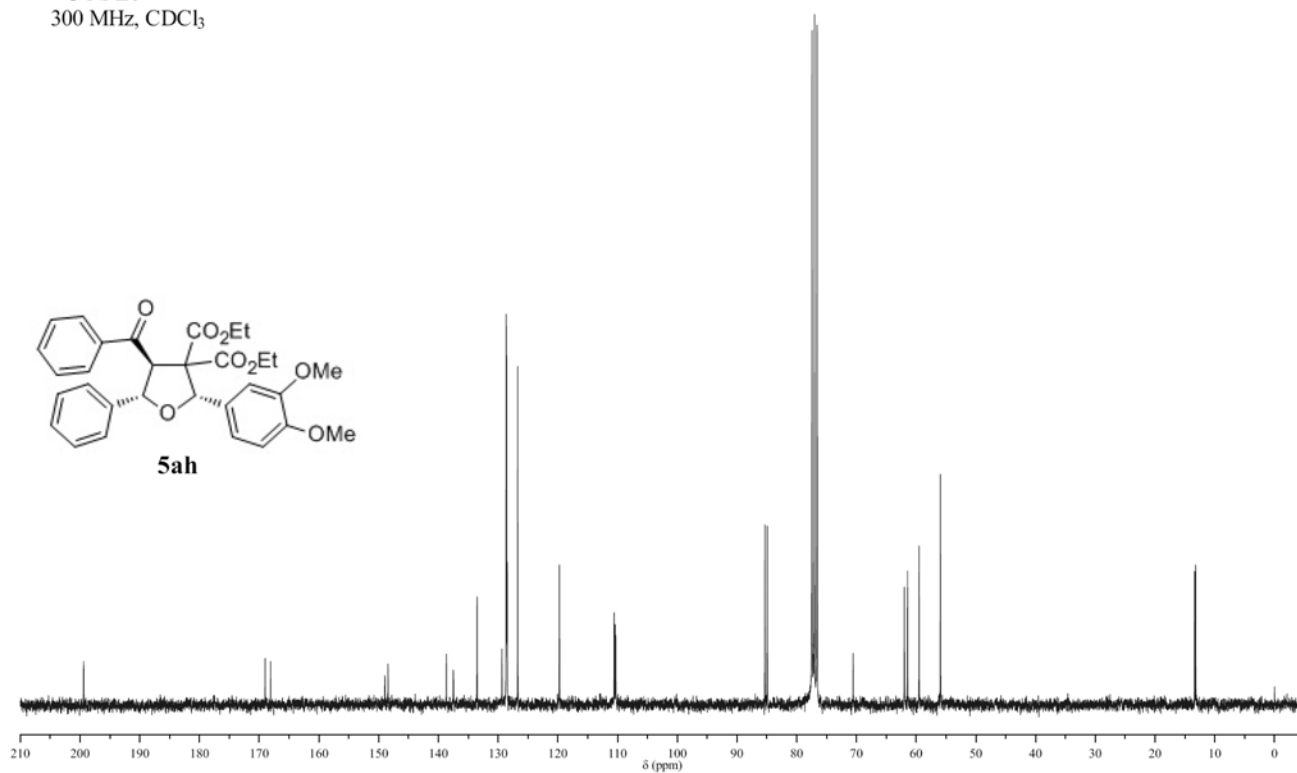
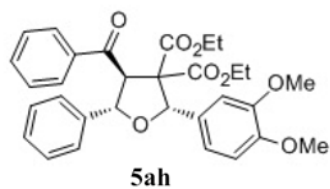
70.55

61.96
61.45
59.51

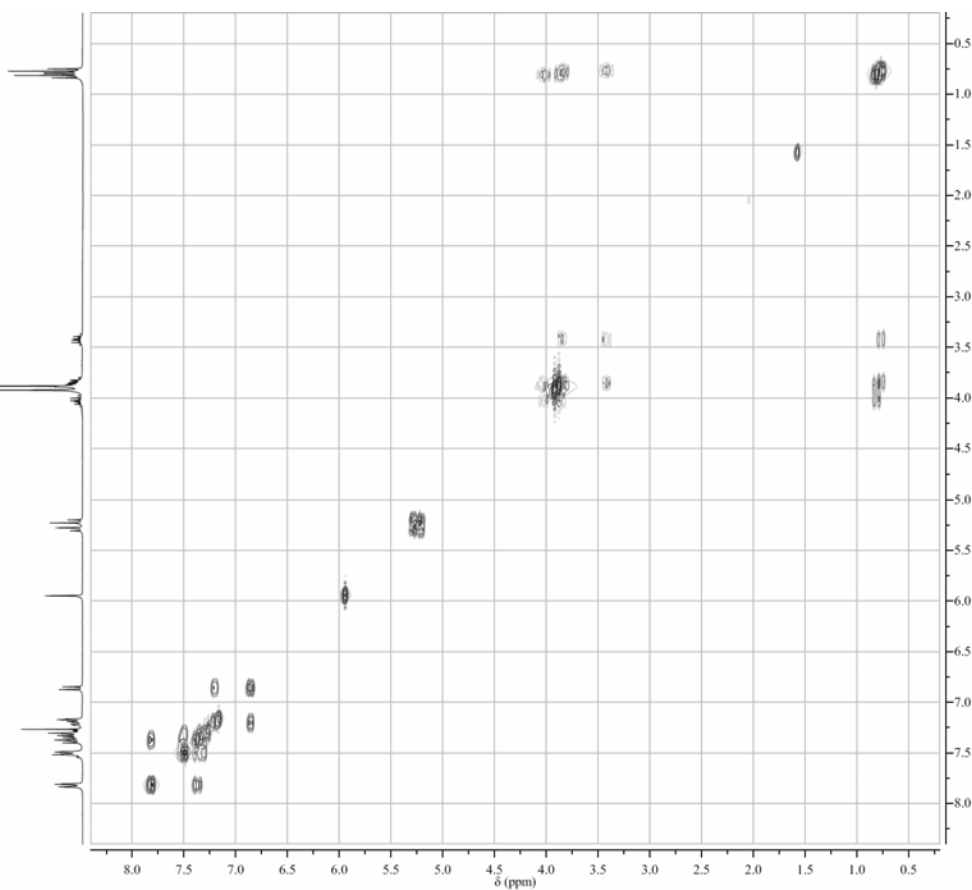
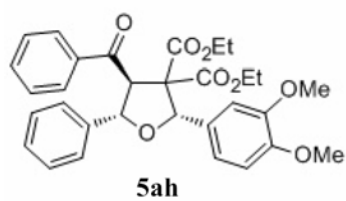
55.94
55.93

13.37
13.21

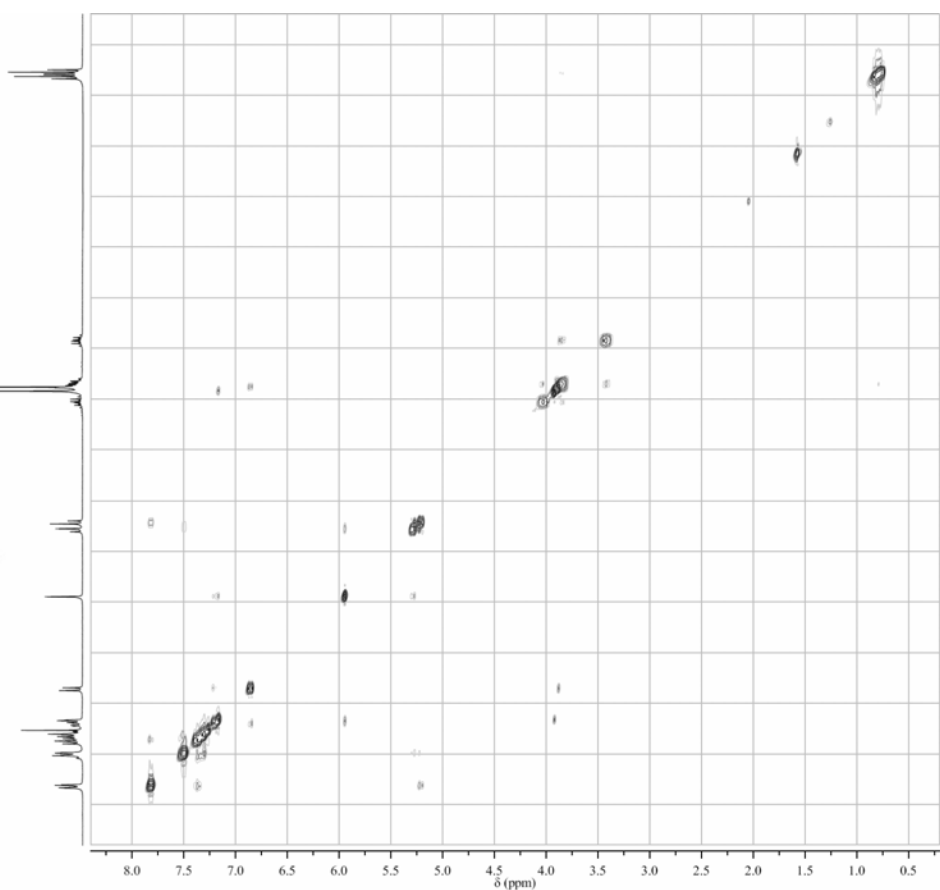
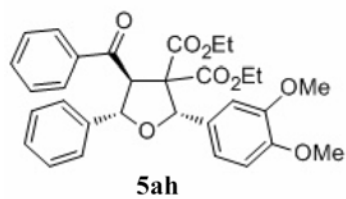
^{13}C NMR
300 MHz, CDCl_3

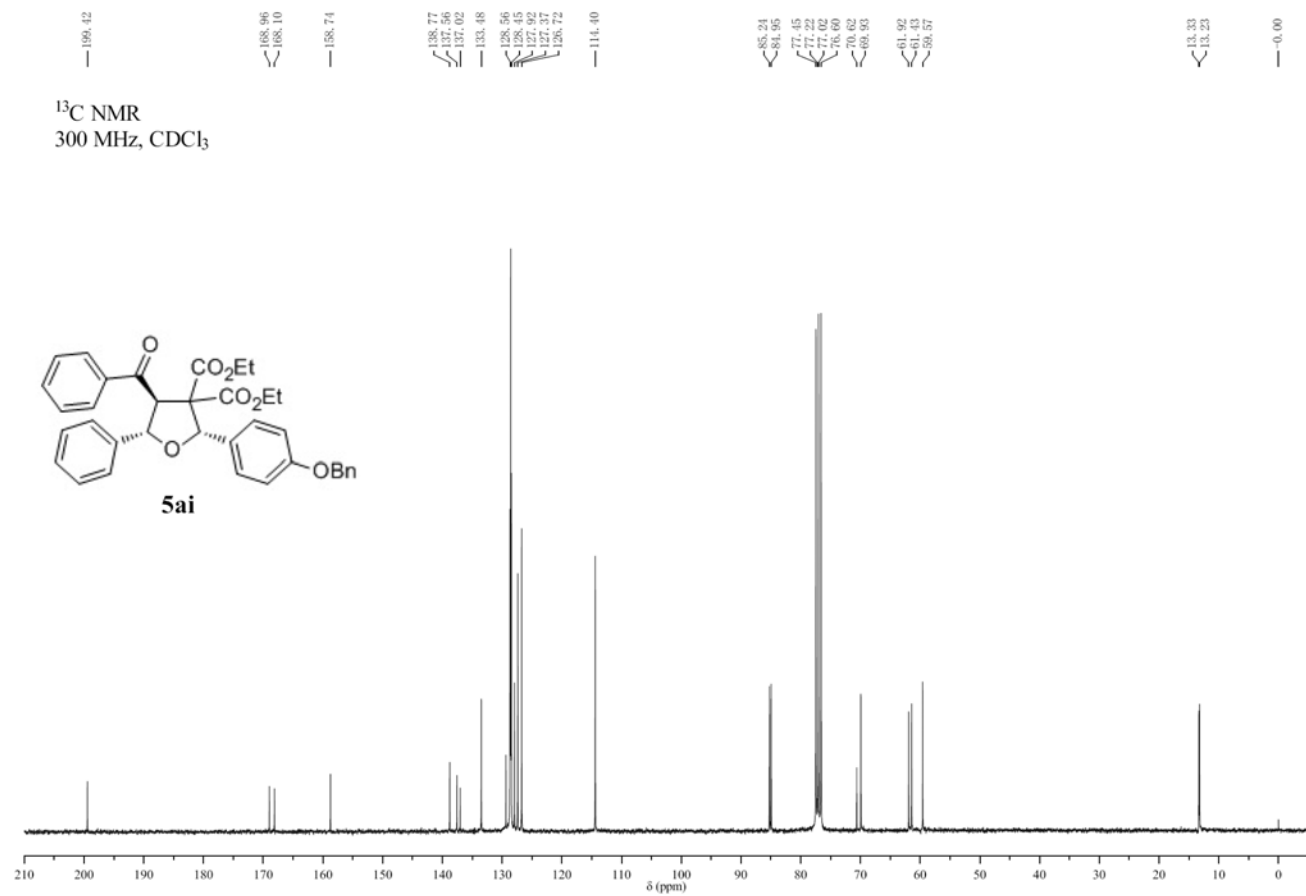
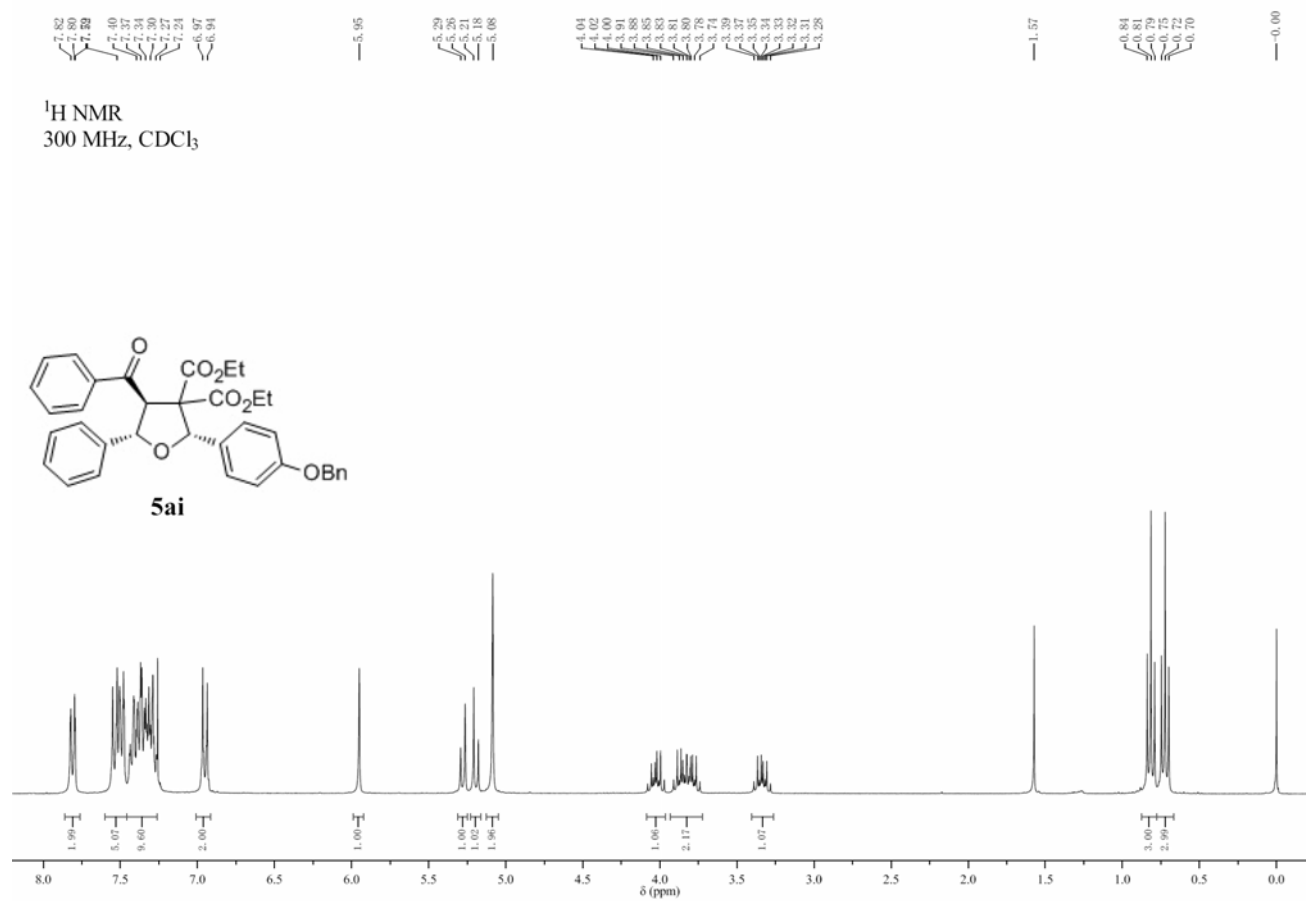


2D COSY NMR
300 MHz, CDCl₃

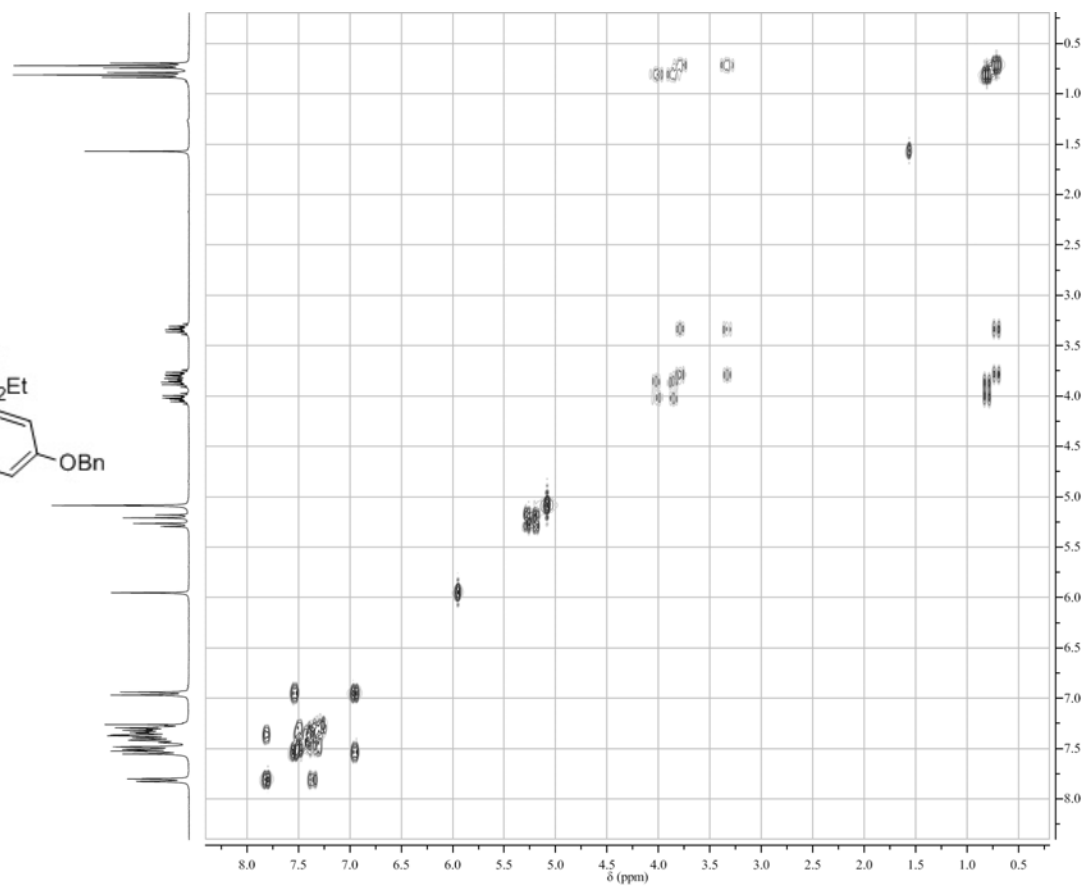
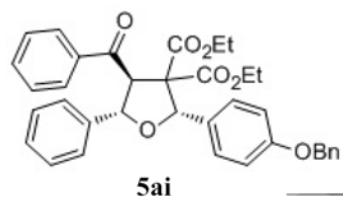


2D NOESY NMR
300 MHz, CDCl₃

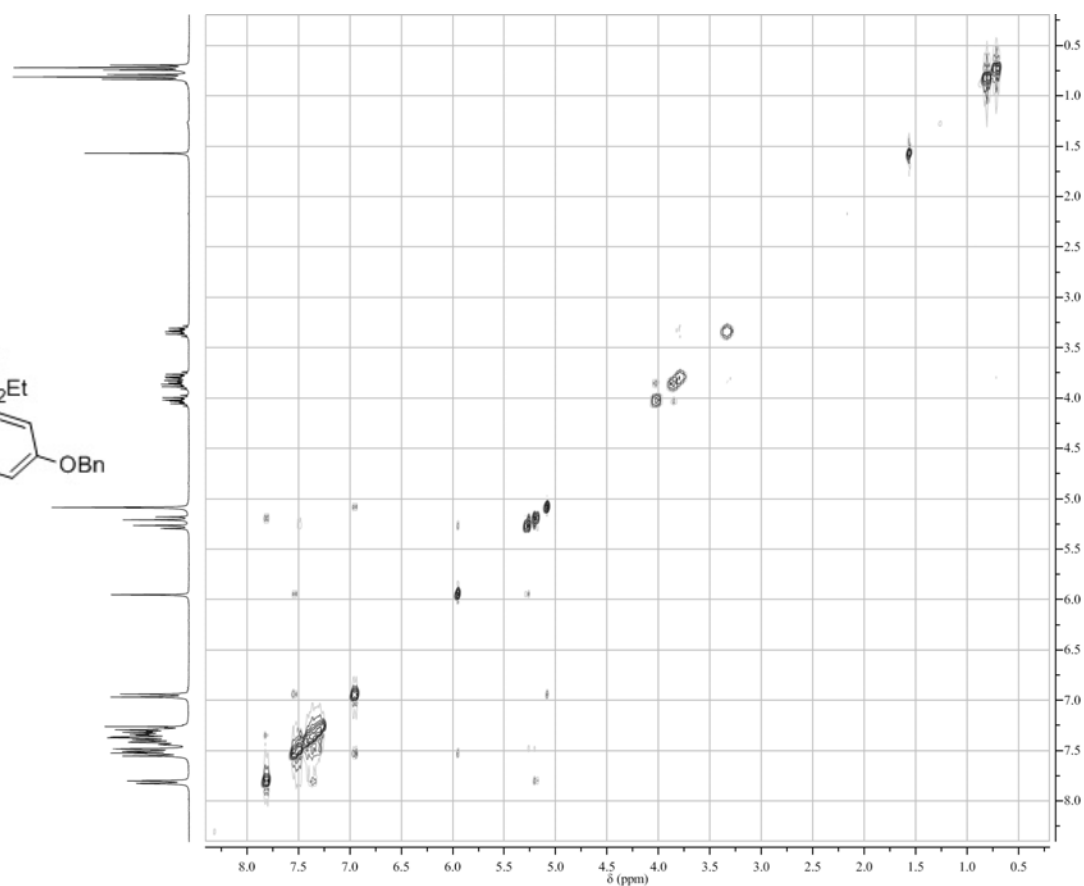
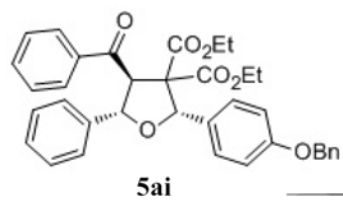


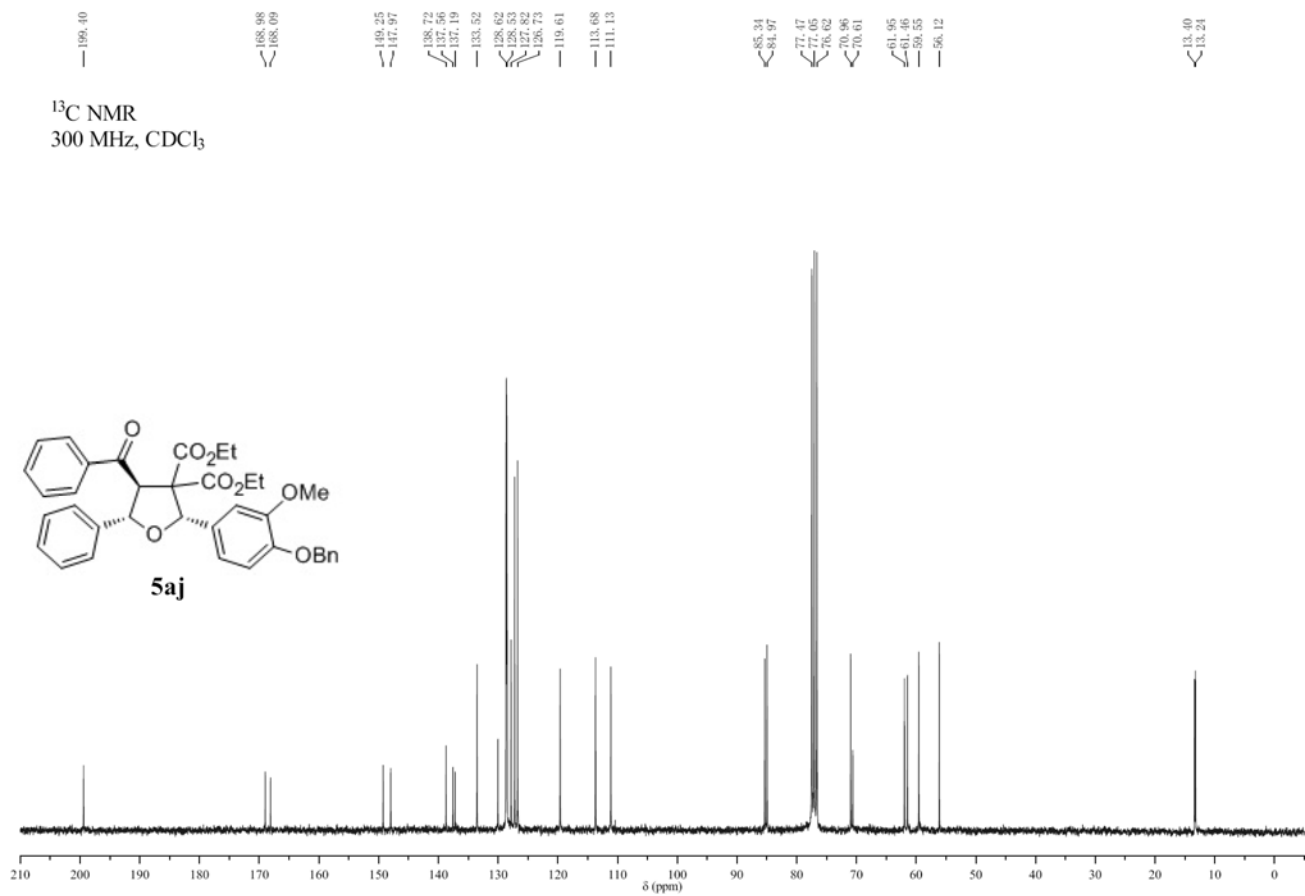
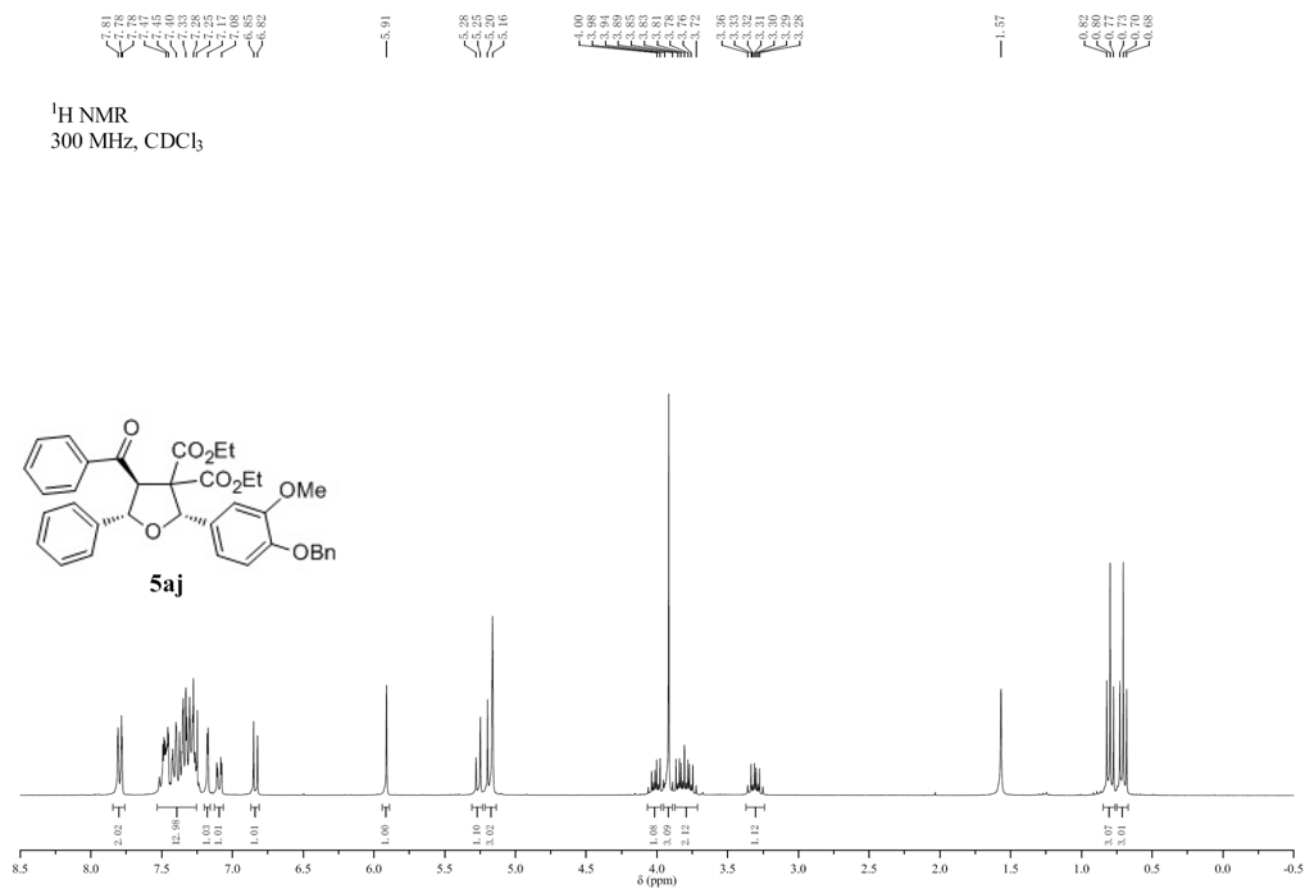


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300 MHz, CDCl₃

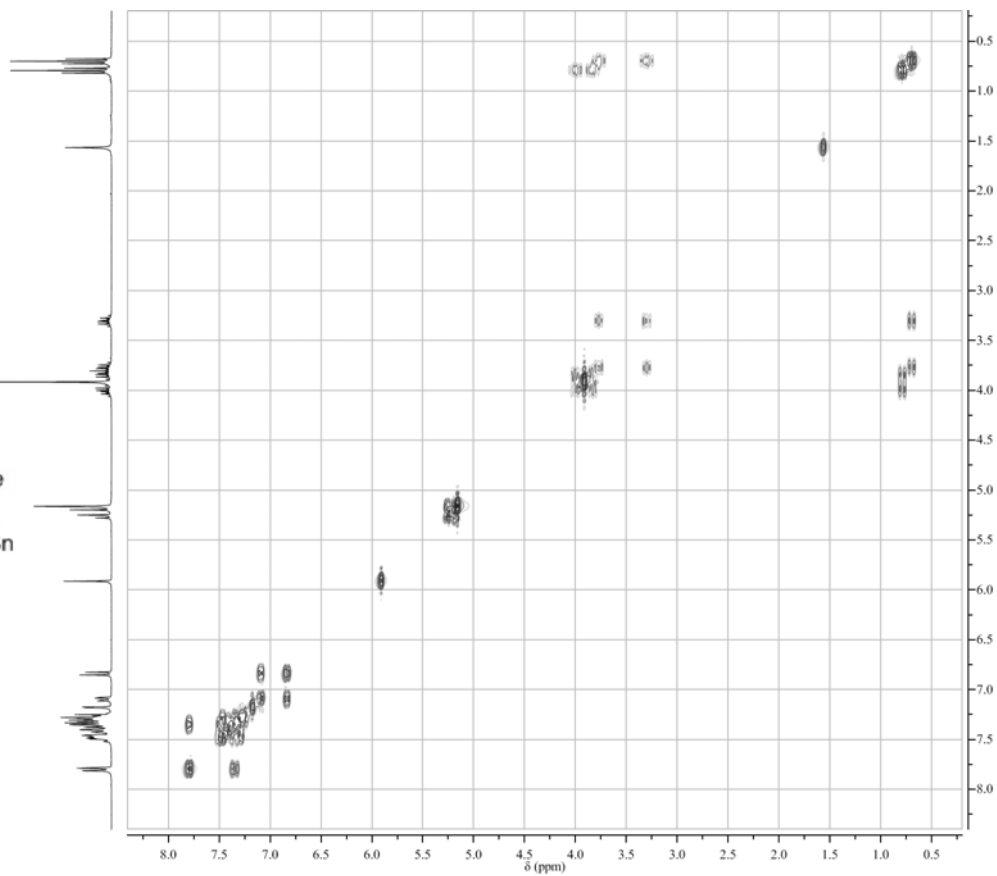
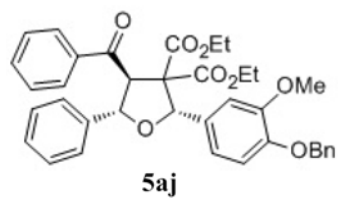


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300 MHz, CDCl₃

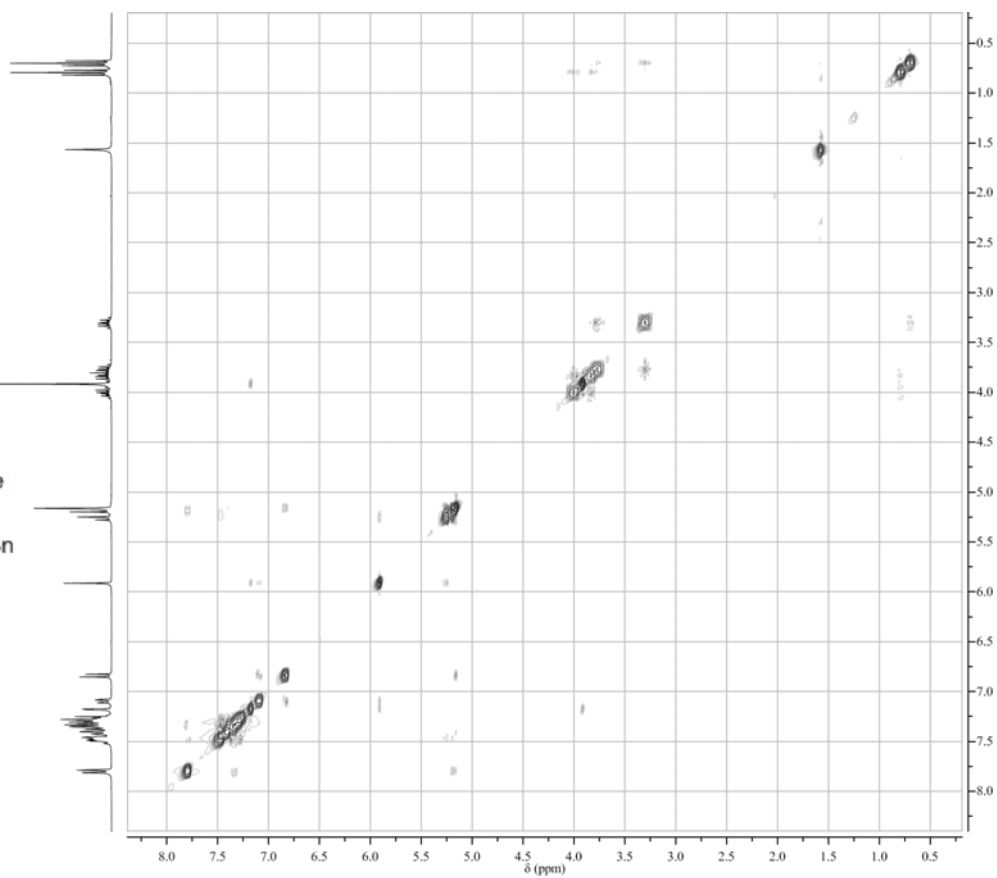
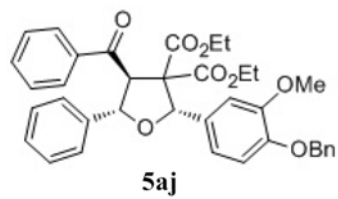


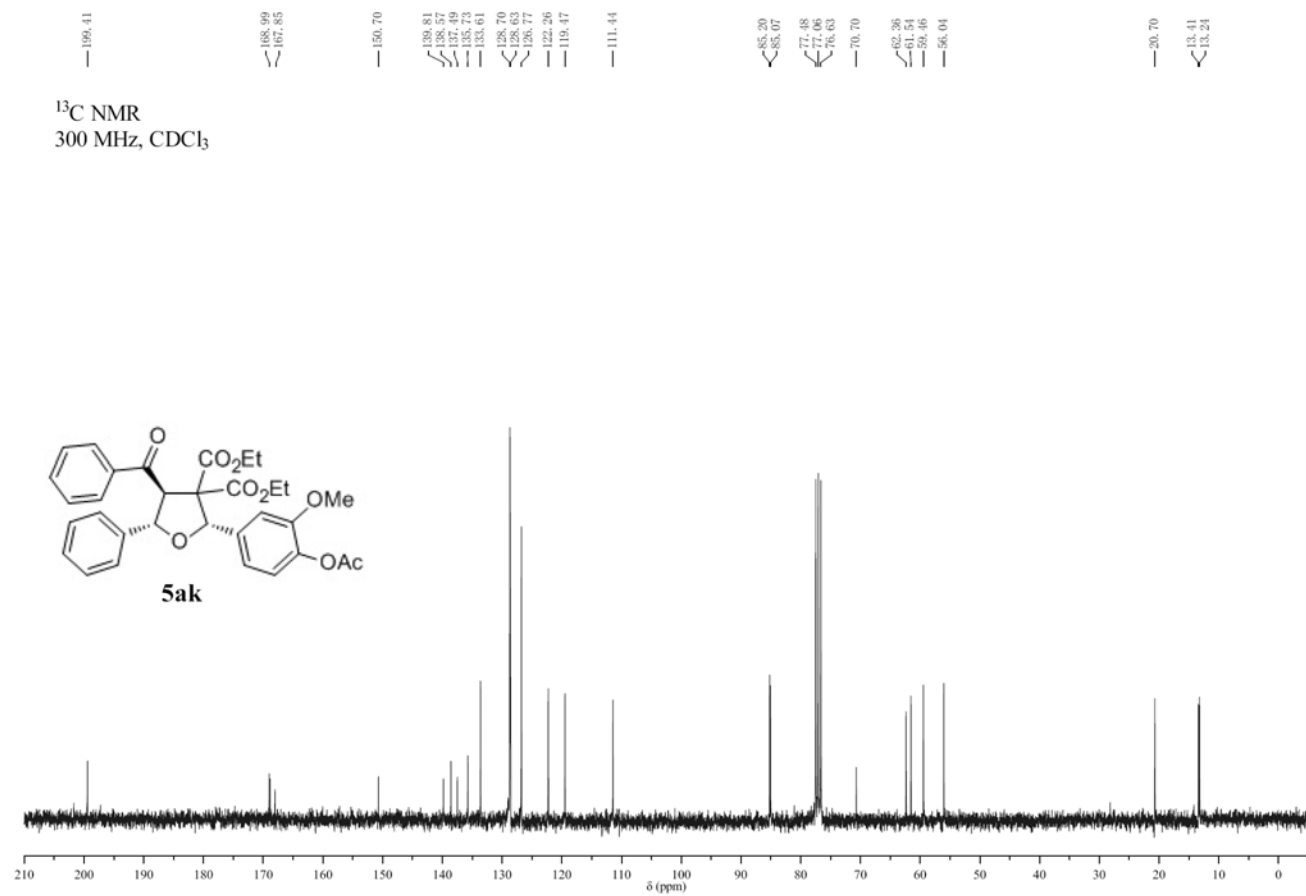
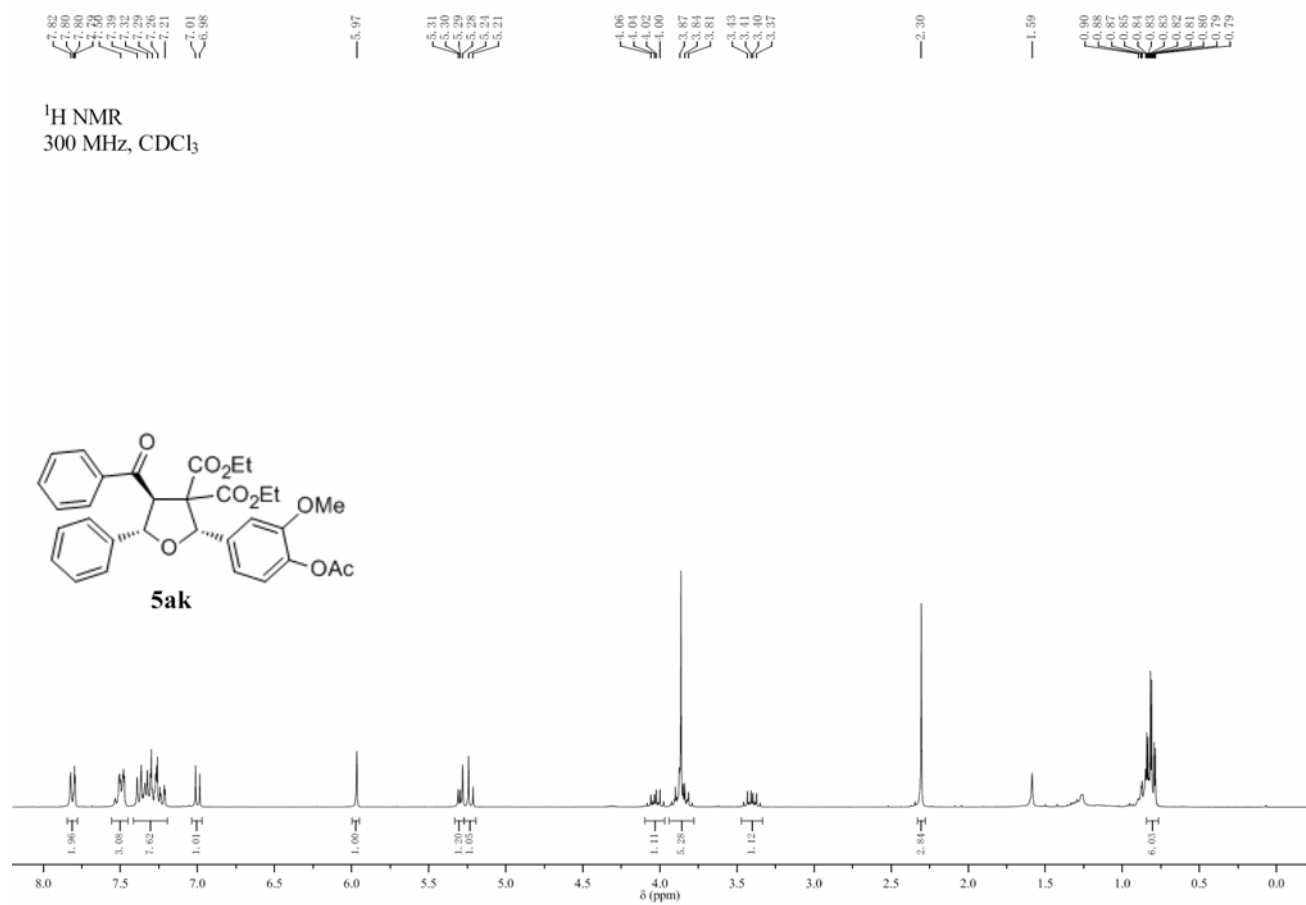


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300 MHz, CDCl₃

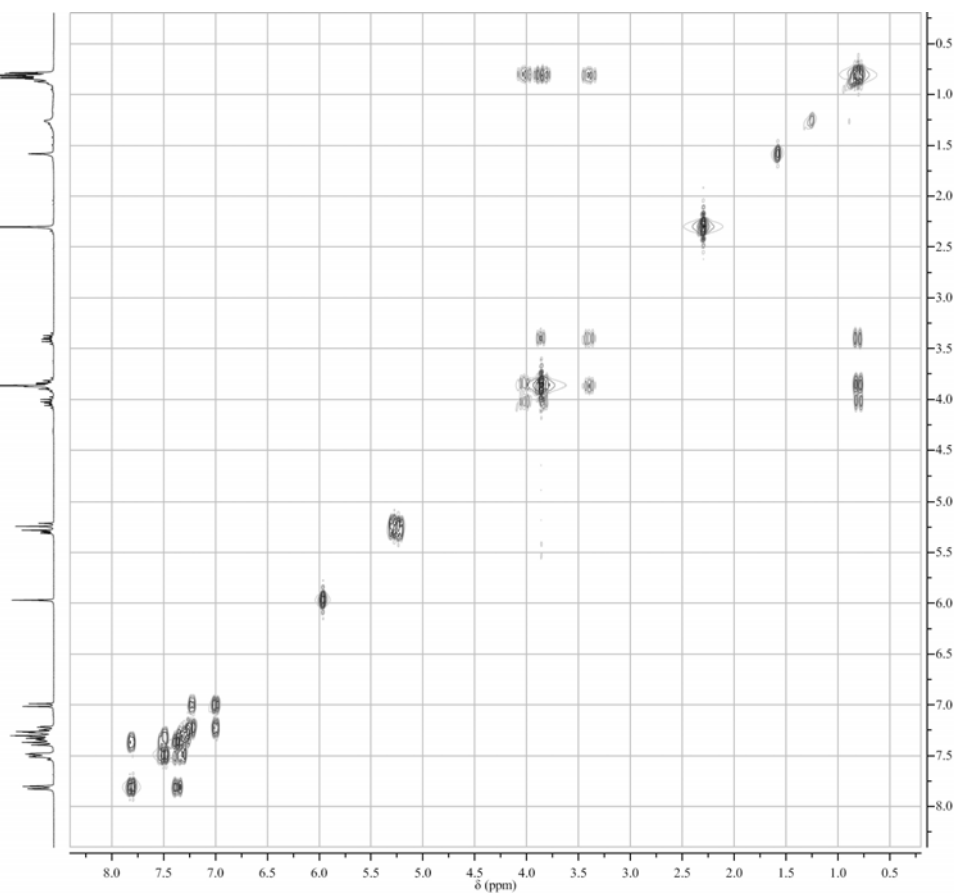
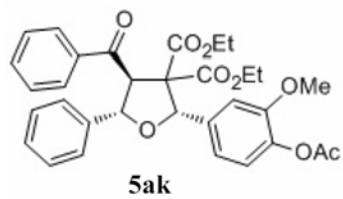


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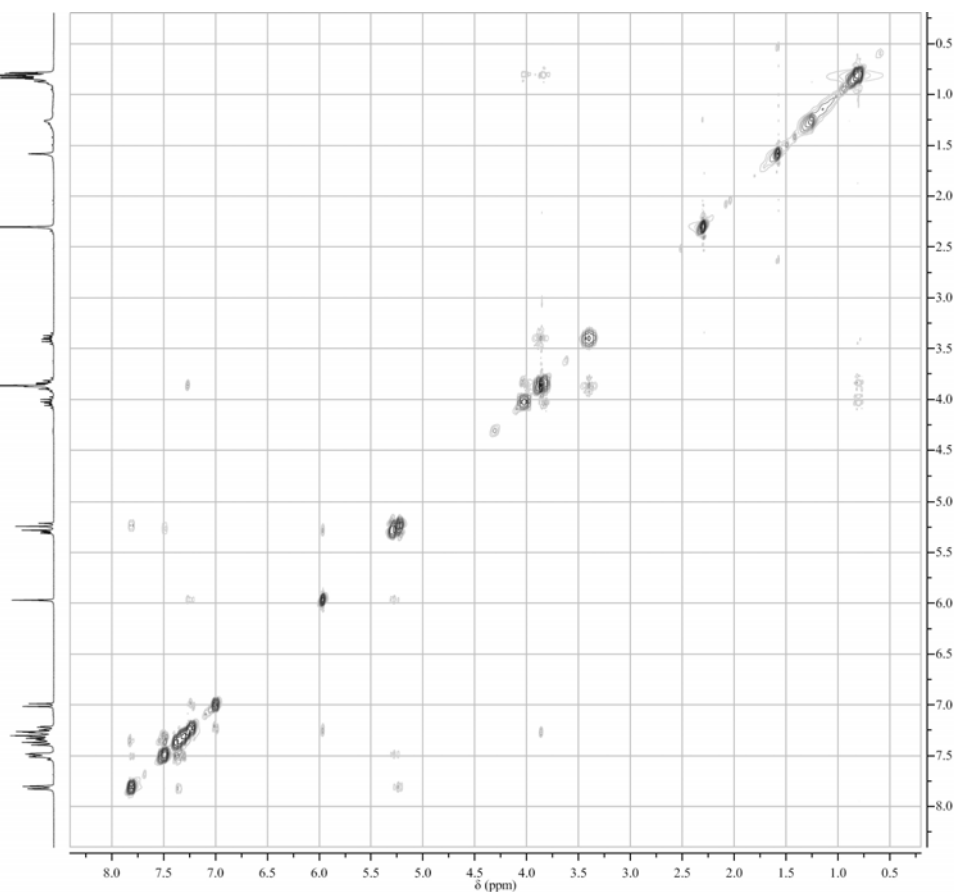
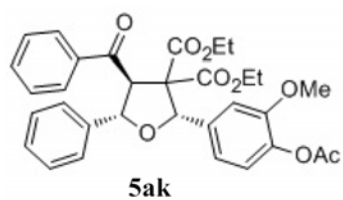




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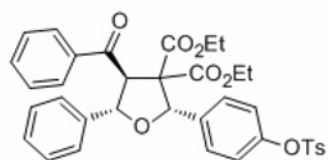


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300 MHz, CDCl₃

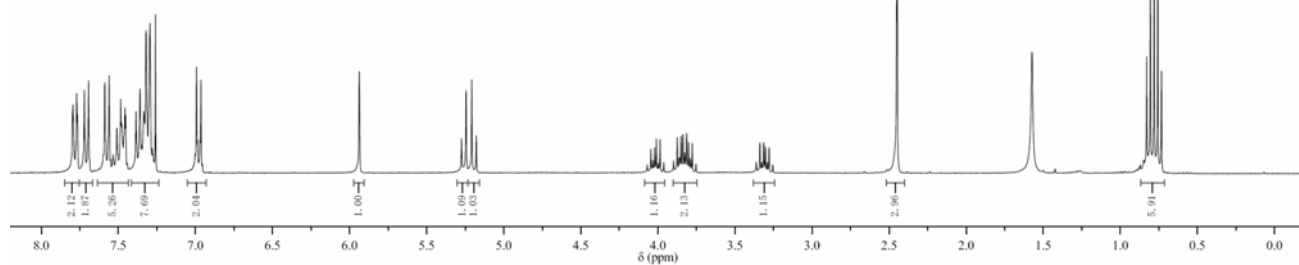




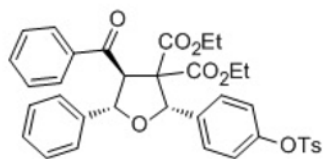
¹H NMR
300 MHz, CDCl₃



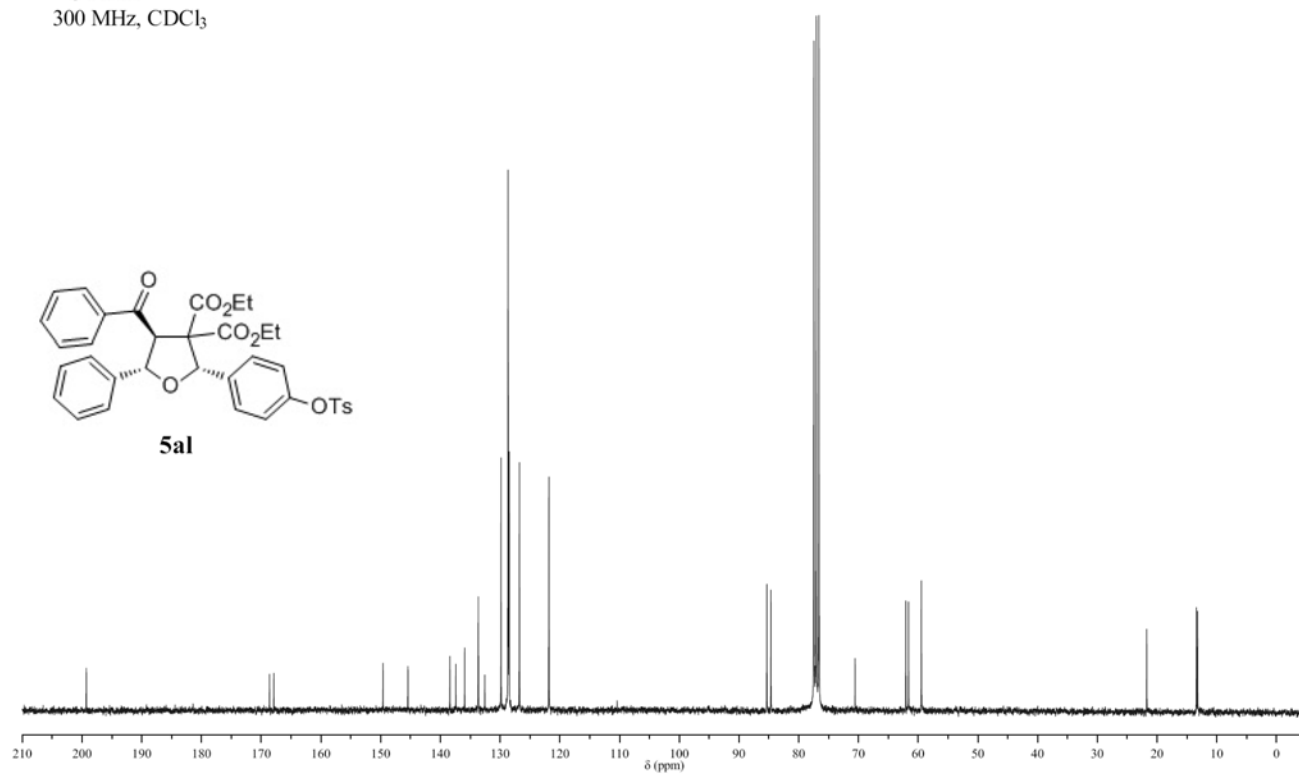
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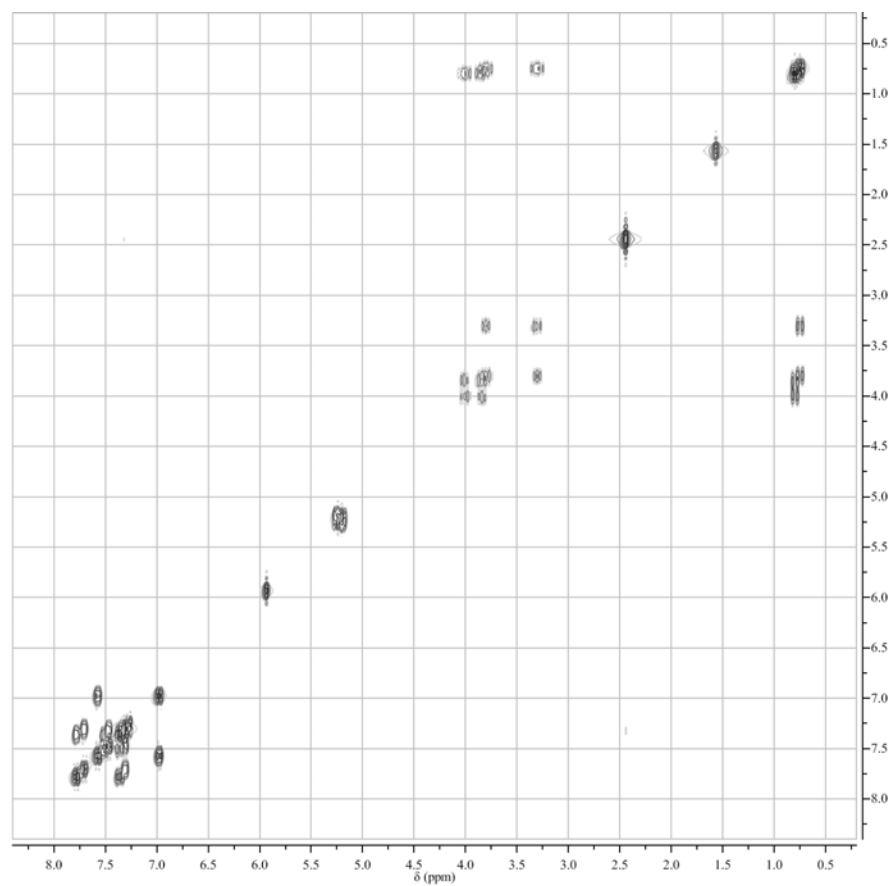
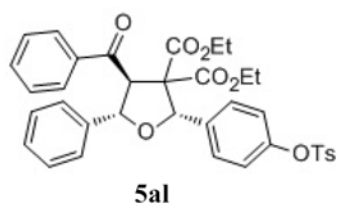
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300 MHz, CDCl₃



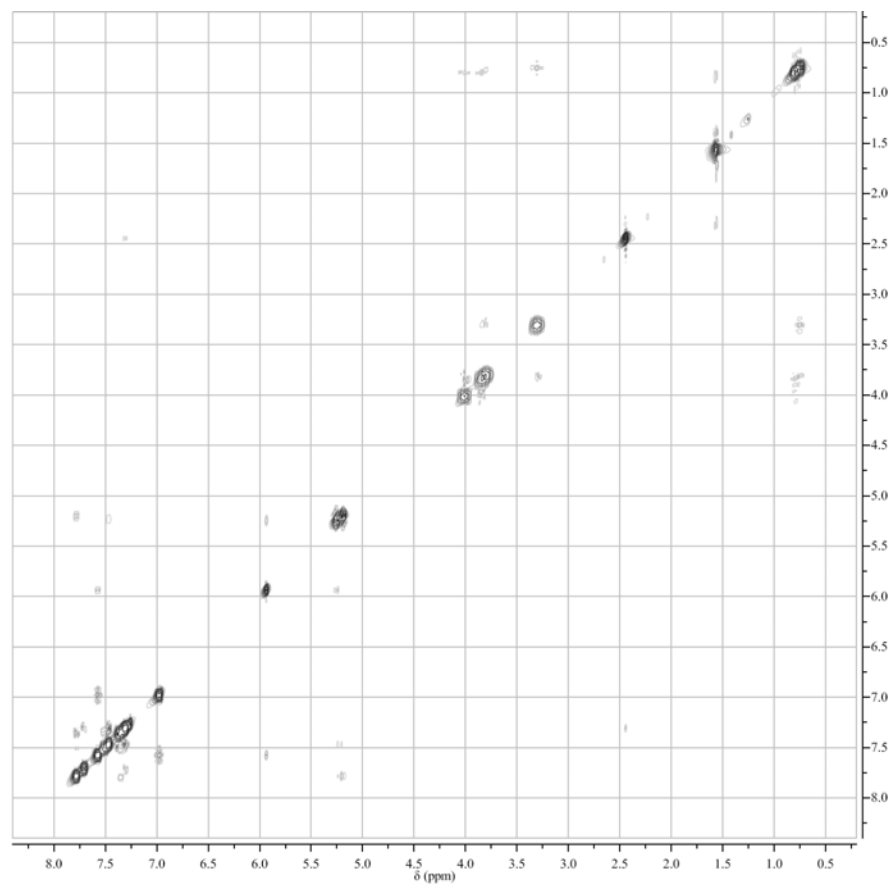
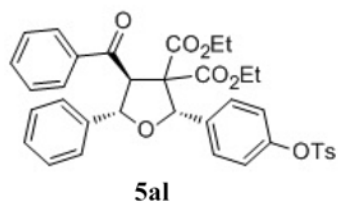
5al



2D COSY NMR
300 MHz, CDCl₃



2D NOESY NMR
300 MHz, CDCl₃



7.60
7.58
7.57
7.55
7.54
7.50
7.49
7.48
7.28
7.26
7.18
7.17
7.16
7.10
7.07

5.91

5.28
5.25
5.22
5.19

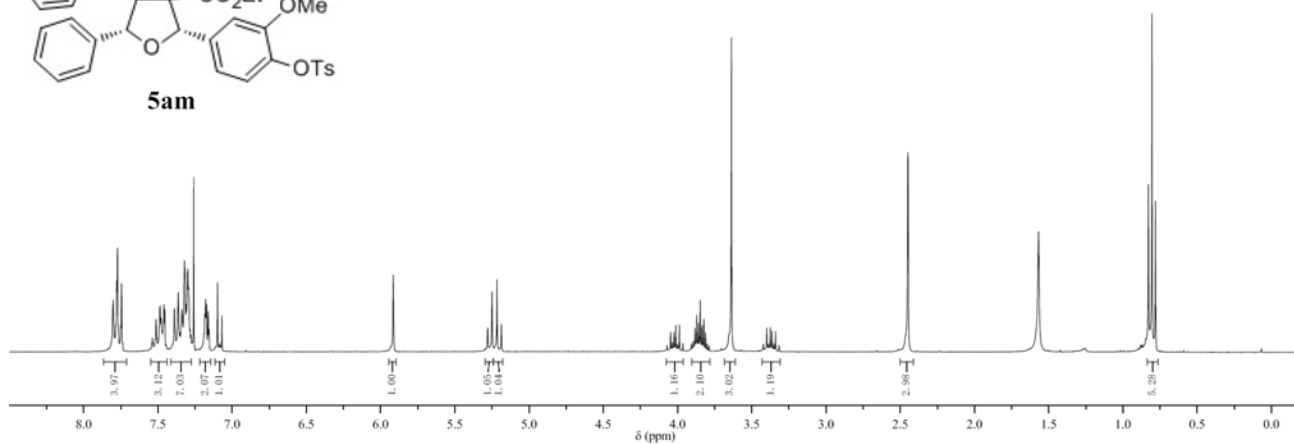
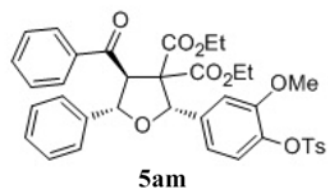
3.99
3.89
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3.84
3.82
3.81
3.80
3.64
3.42
3.40
3.39
3.38
3.36
3.35
3.34
3.32

2.45

1.57

0.83
0.80
0.78

¹H NMR
300 MHz, CDCl₃



199.27

168.68
167.91

151.45

138.40
137.42
136.96

133.50

128.63
126.77

123.29

119.40

111.88

85.29
81.81

77.46
77.24
77.04

76.61
70.58

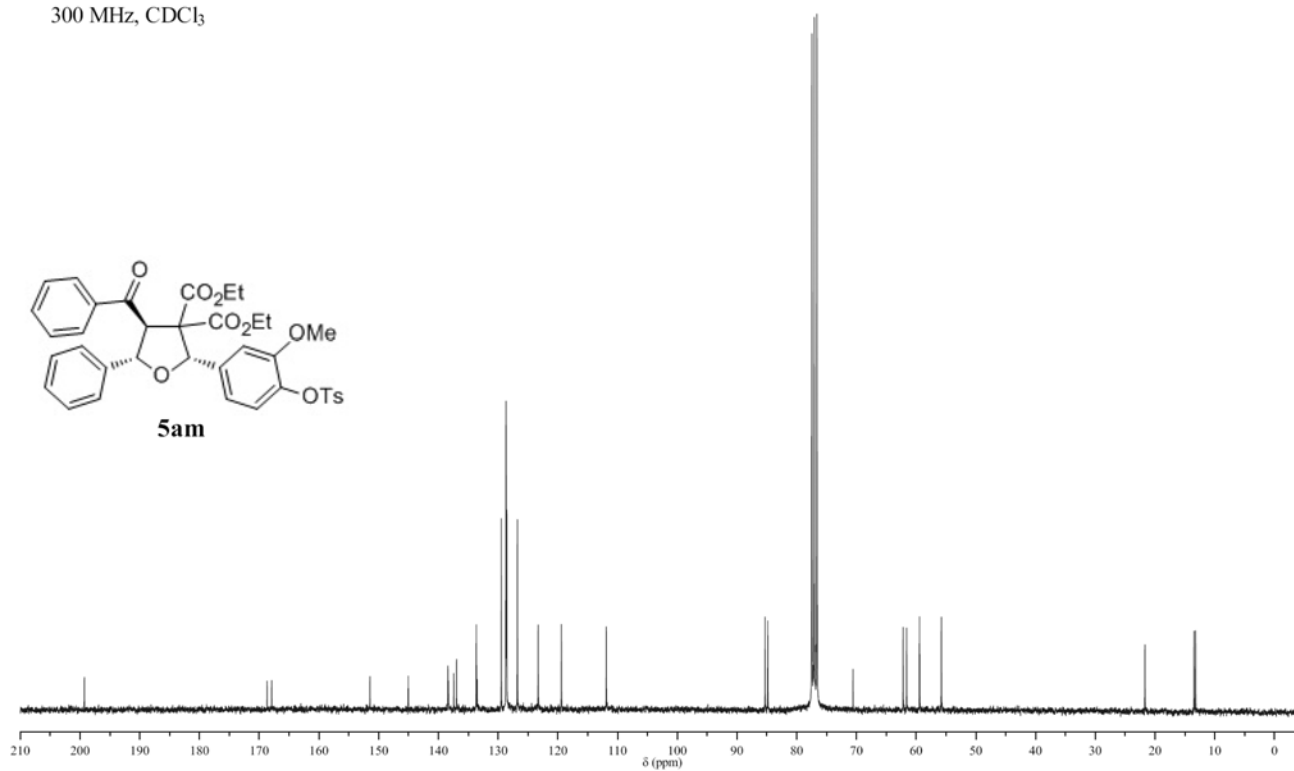
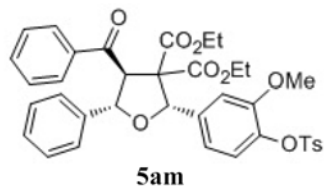
62.18
61.60
59.43

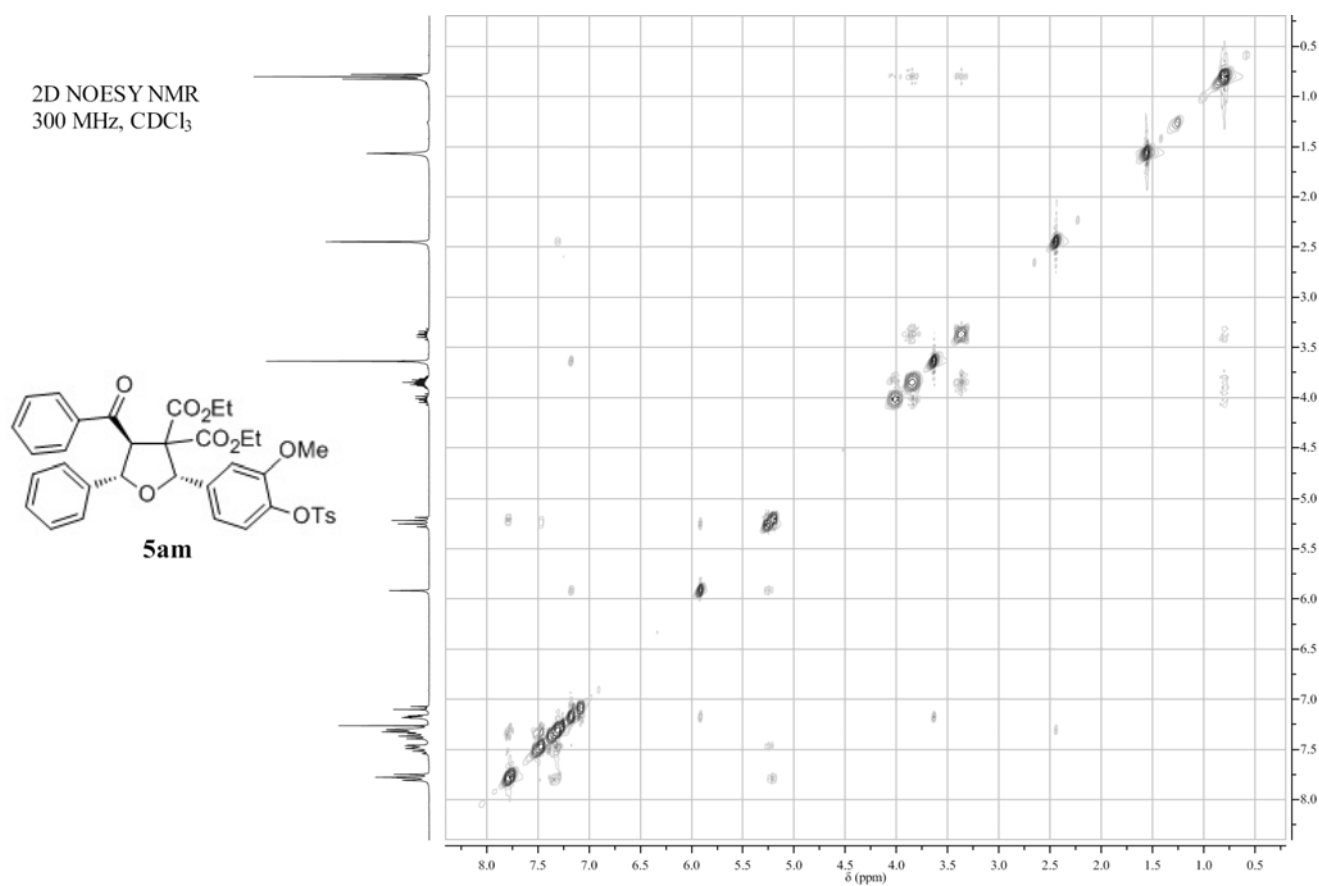
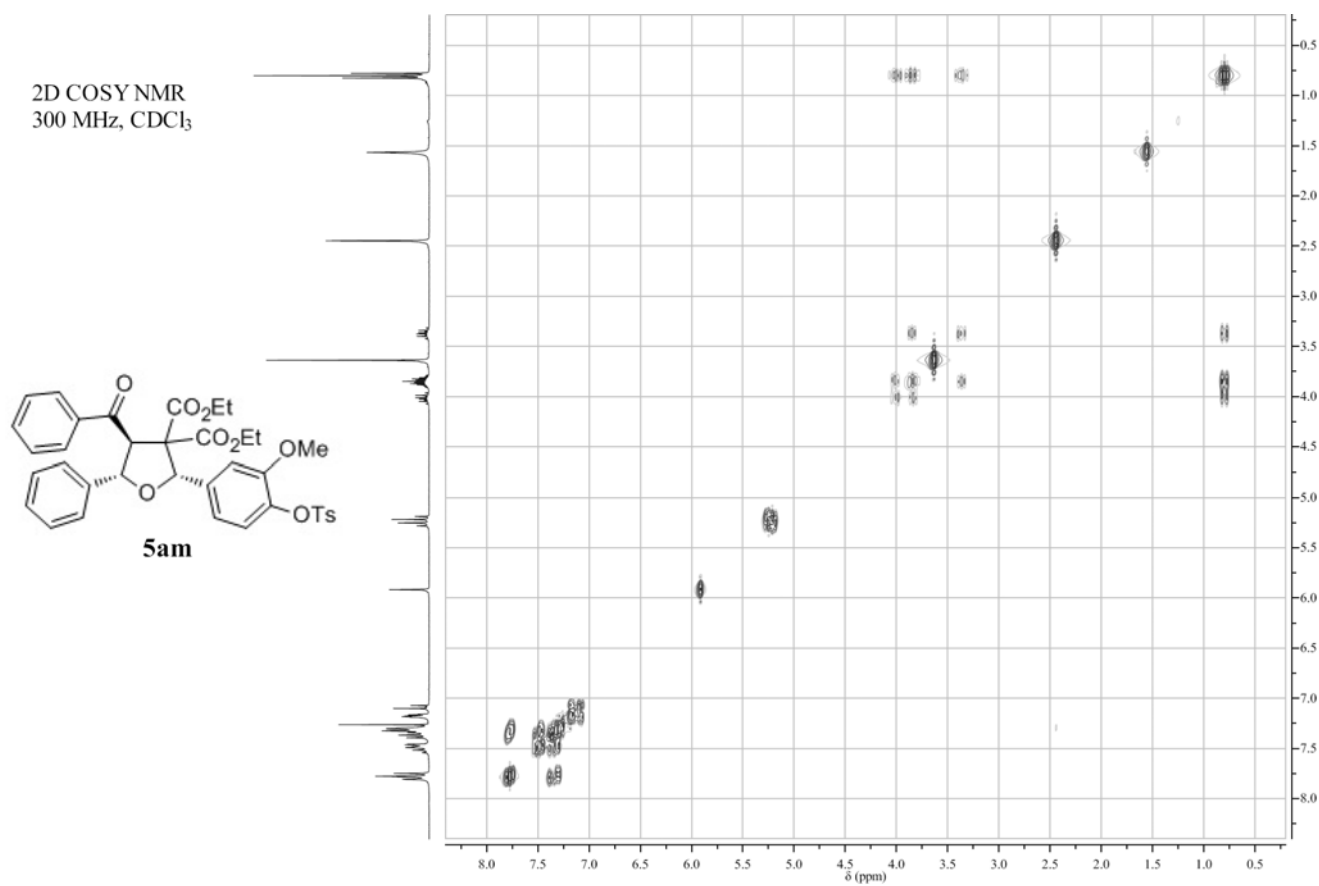
55.78

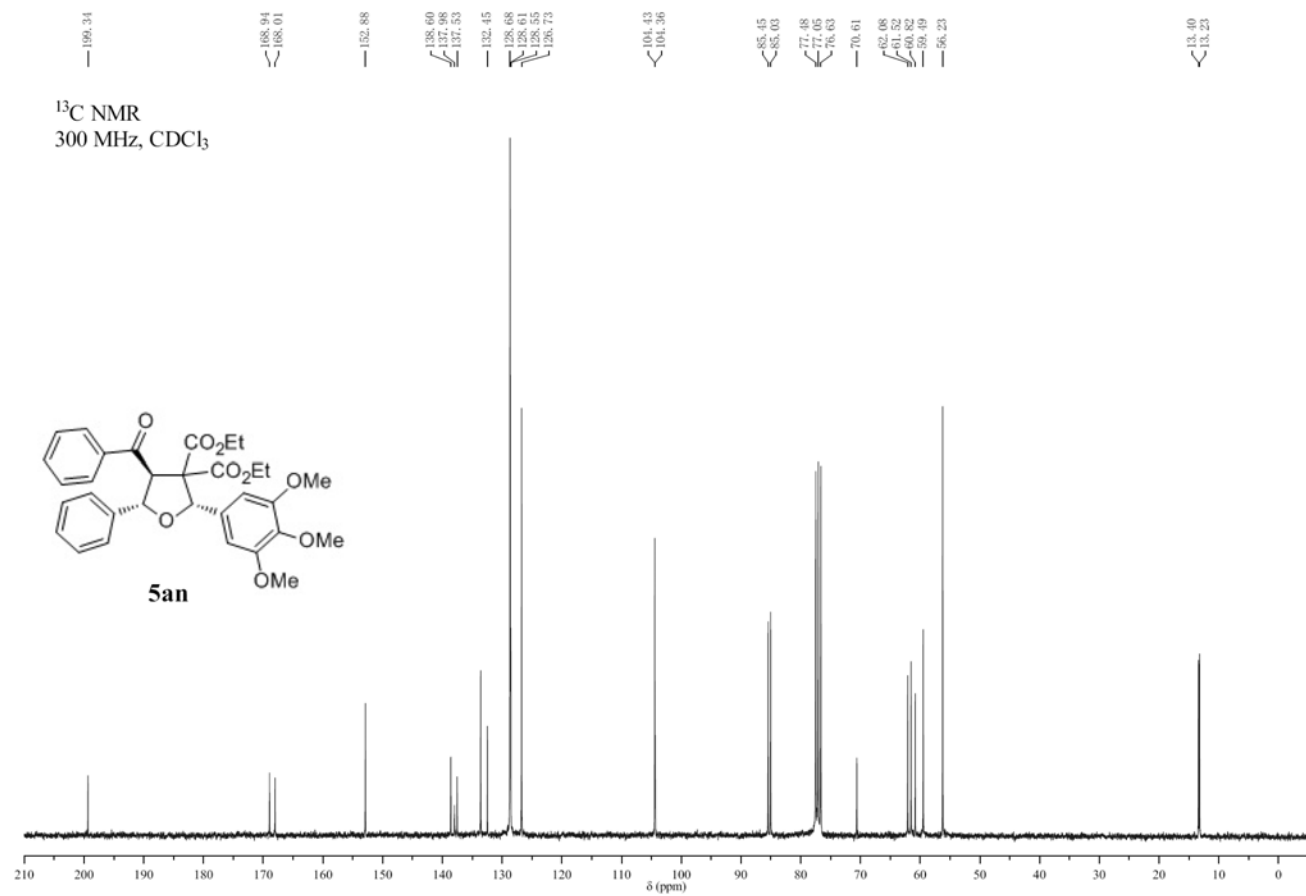
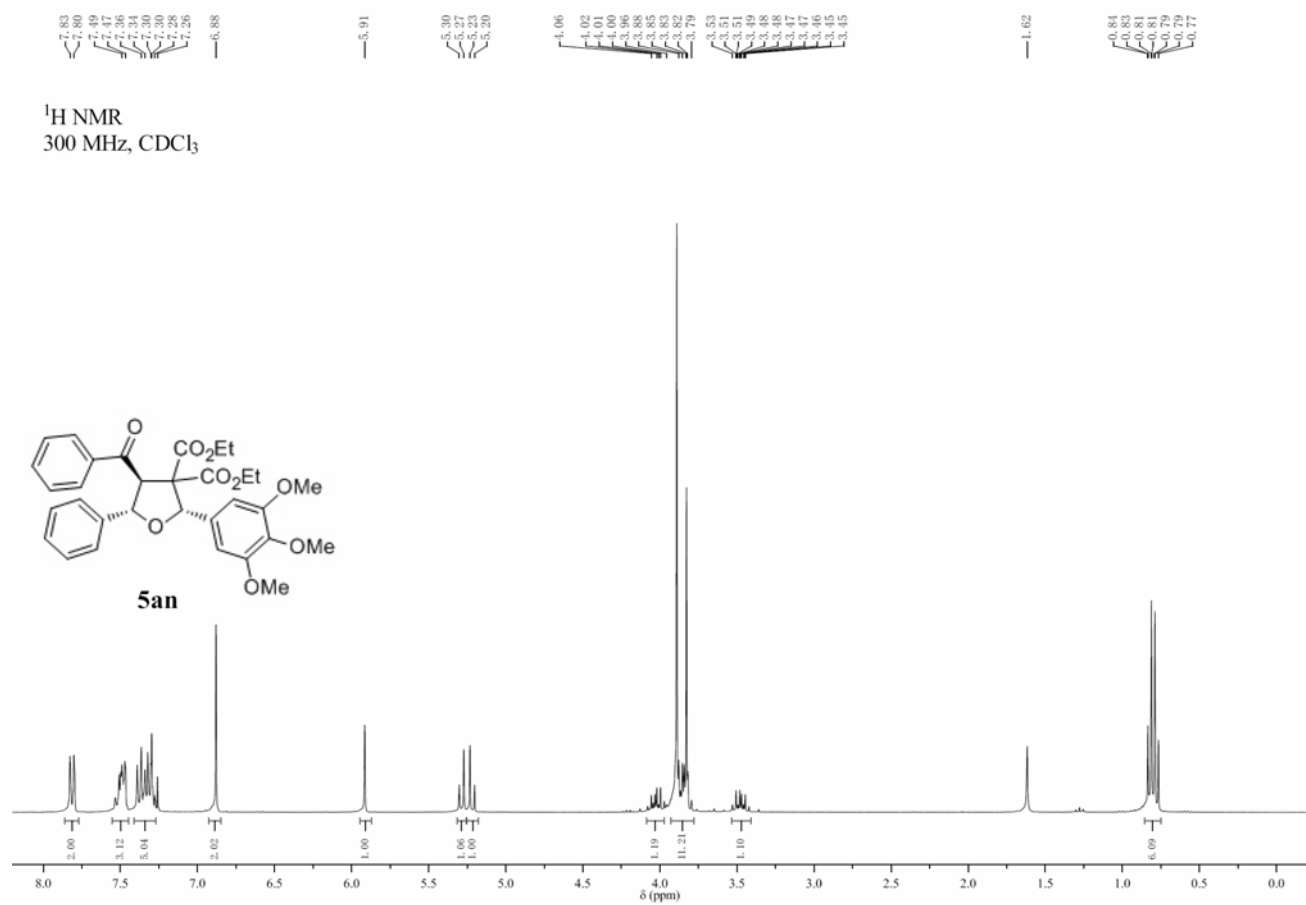
21.70

13.48
13.22

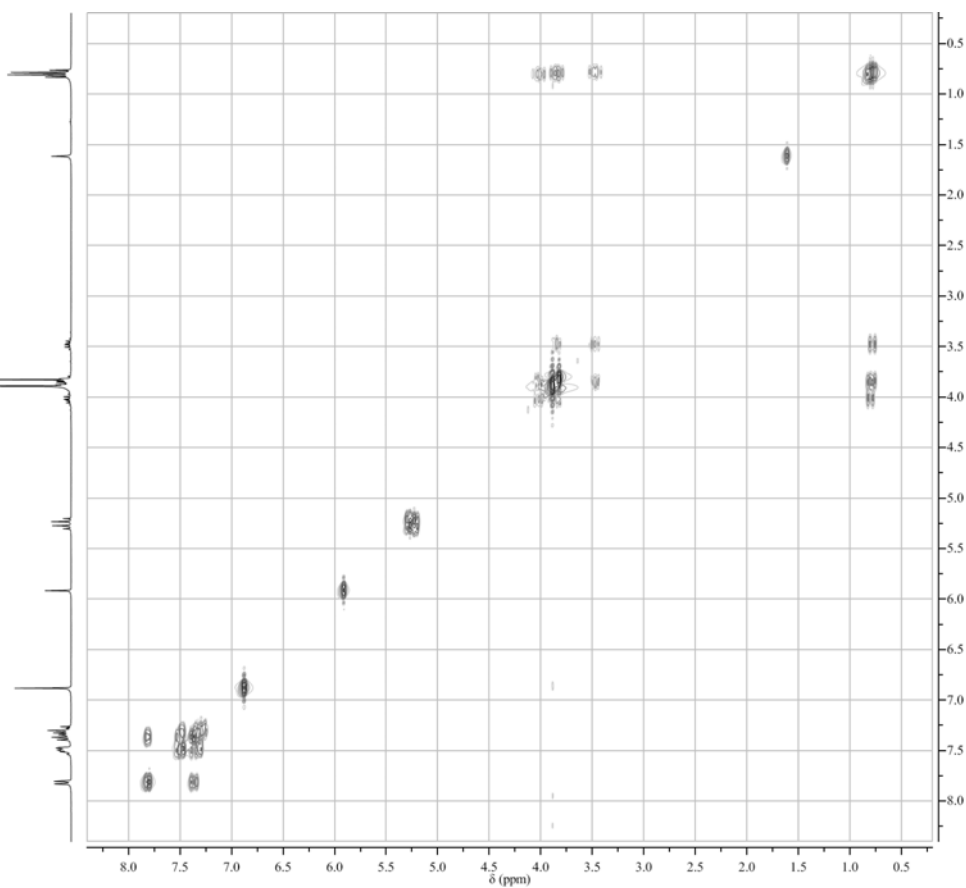
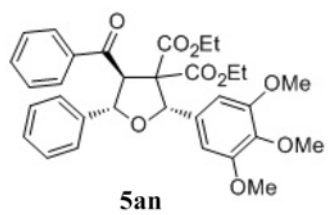
¹³C NMR
300 MHz, CDCl₃



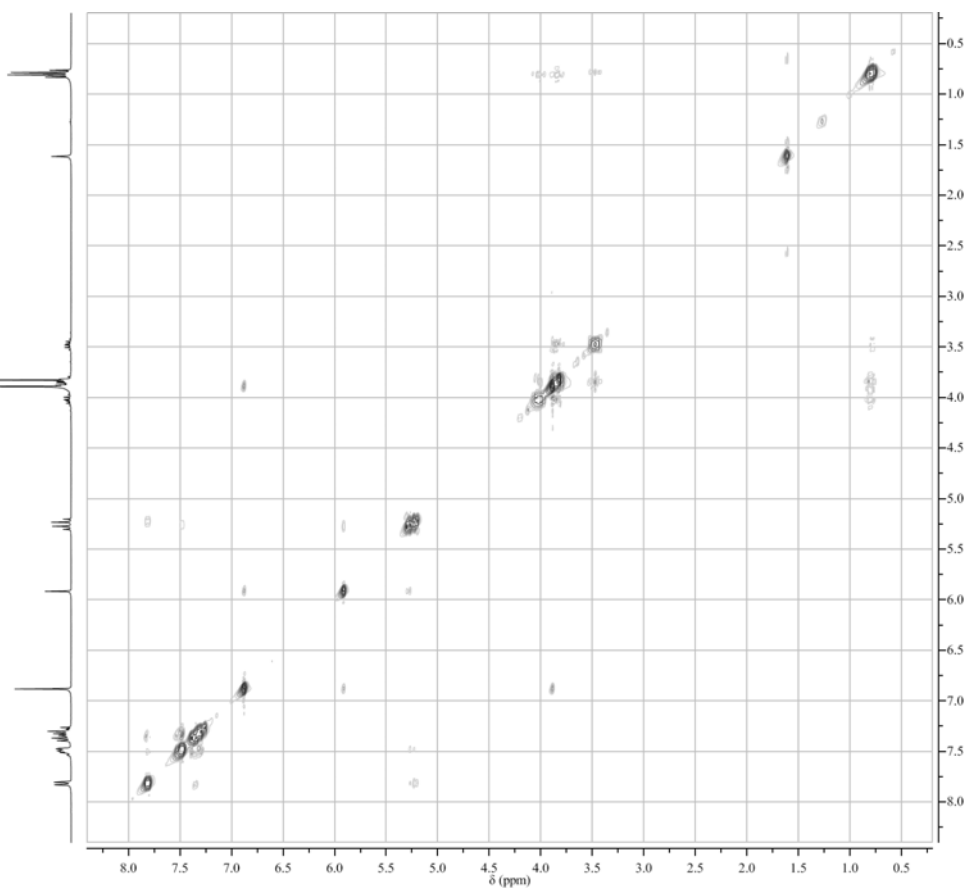
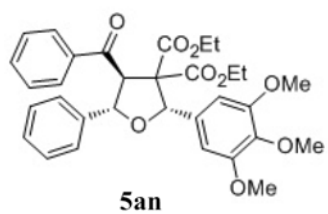


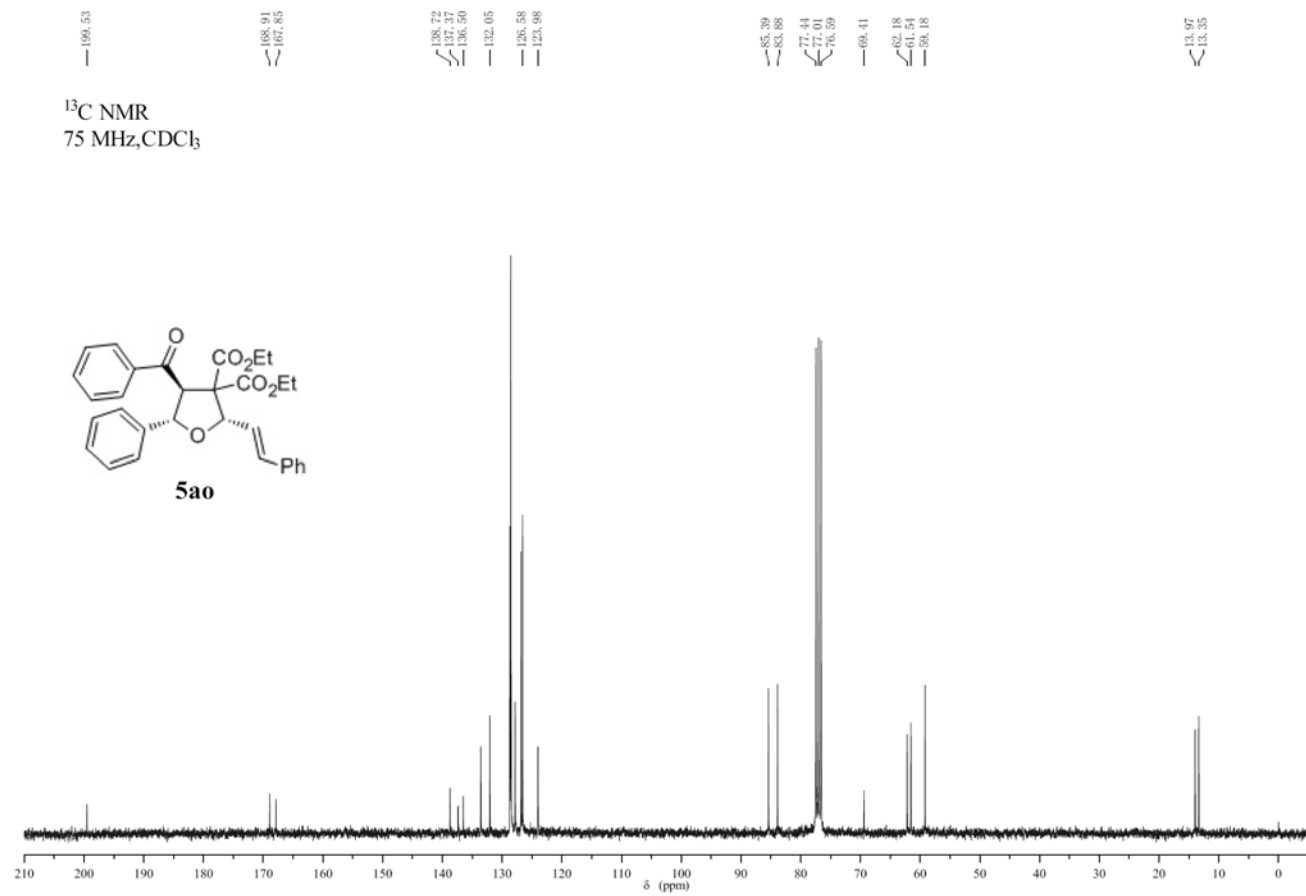
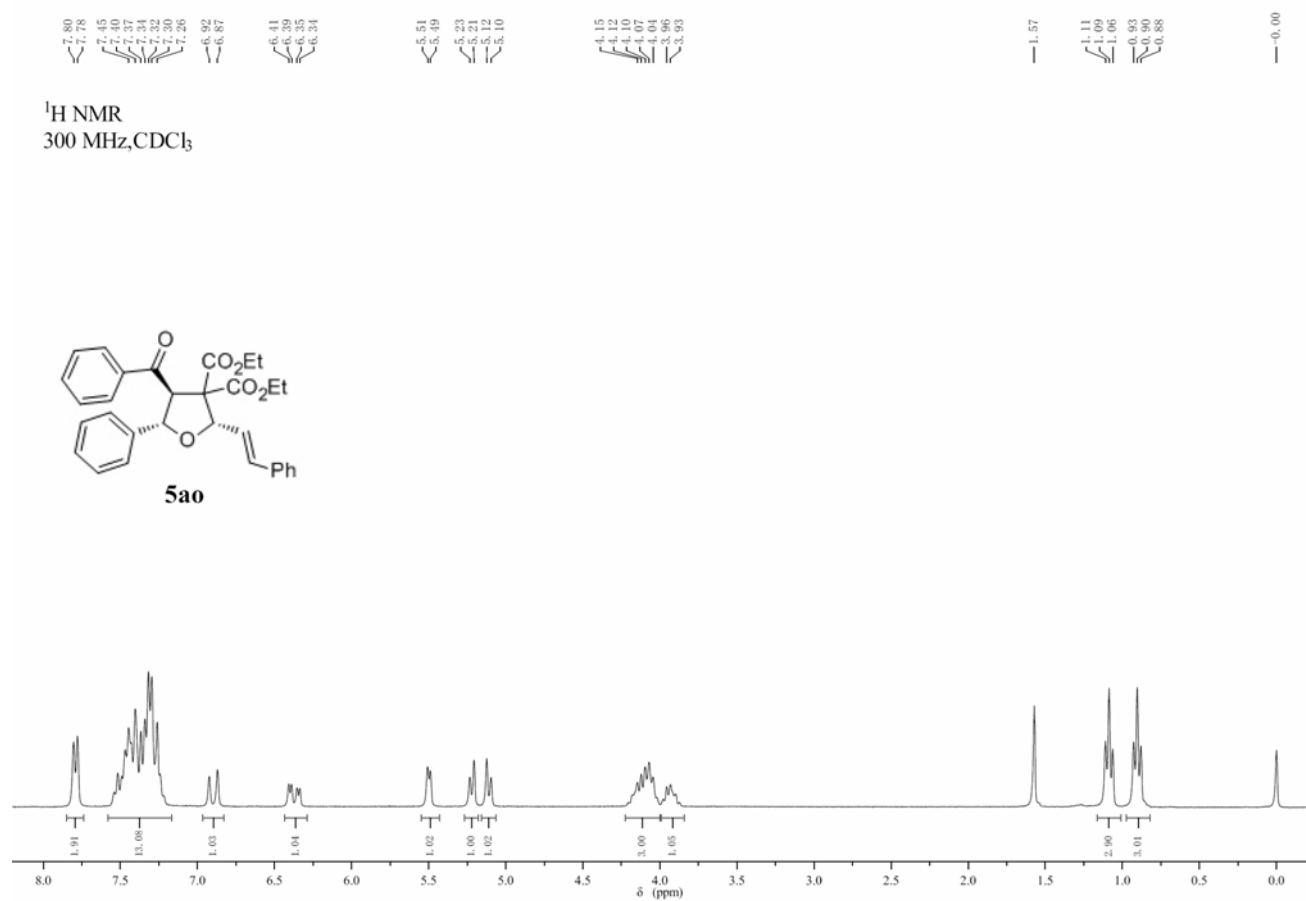


2D COSY NMR
300 MHz, CDCl₃

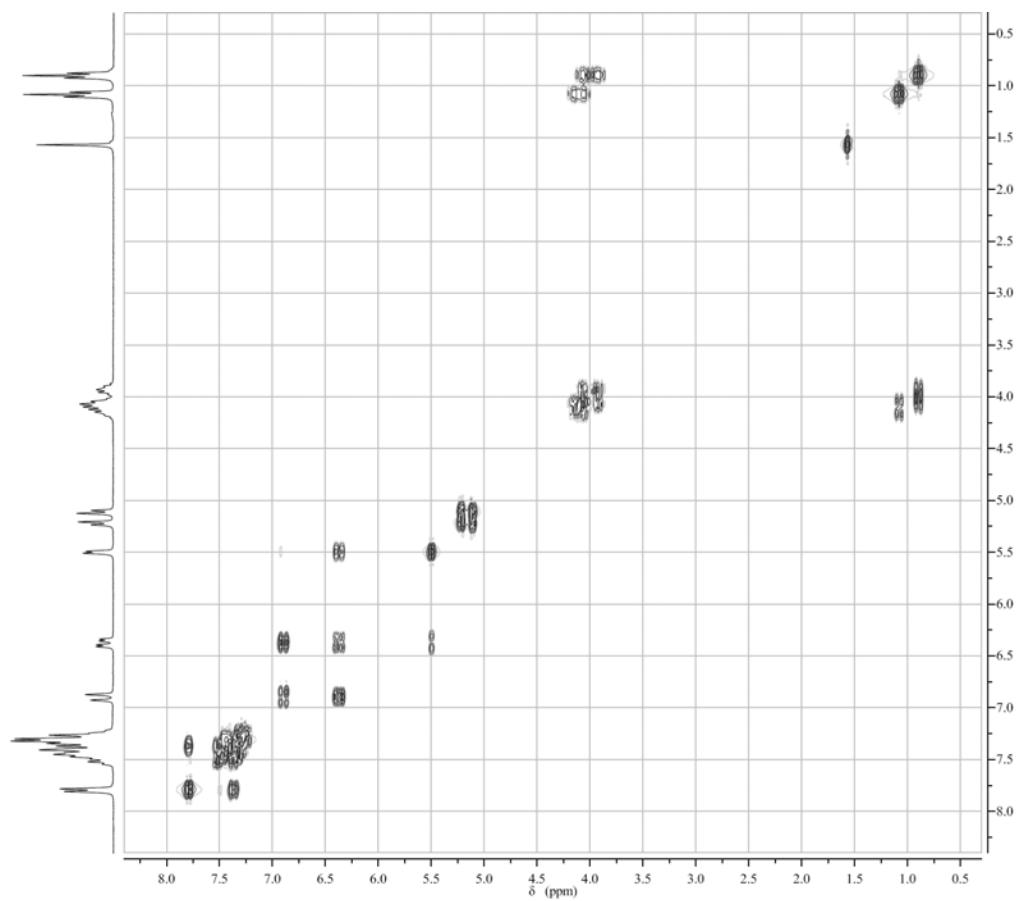
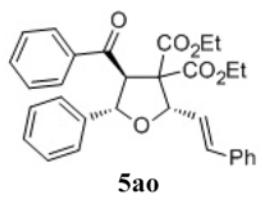


2D NOESY NMR
300 MHz, CDCl₃

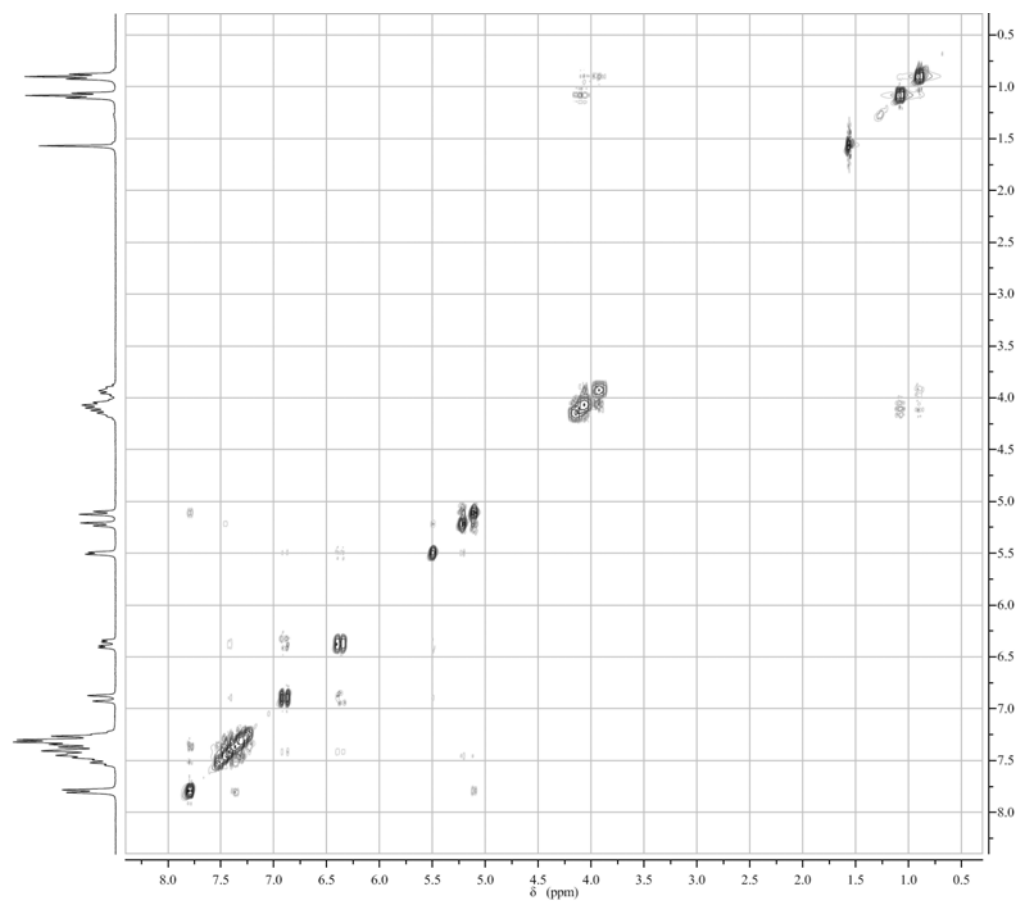
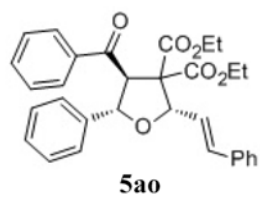




2D COSY NMR
300 MHz, CDCl₃



2D NOESY NMR
300 MHz, CDCl₃



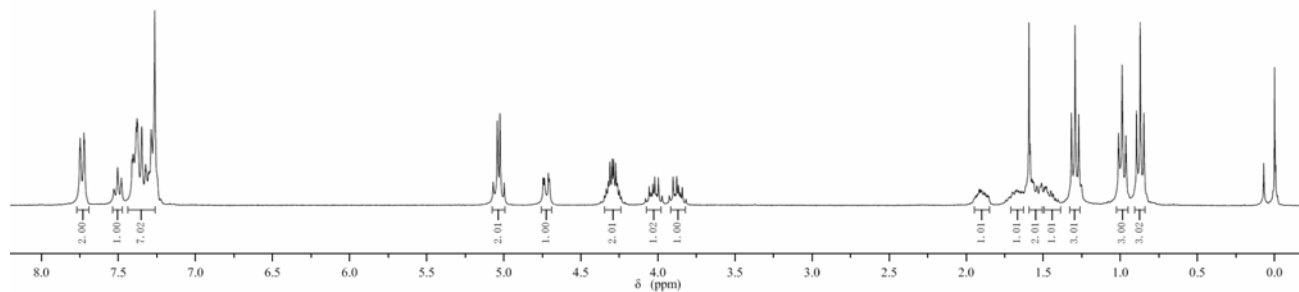
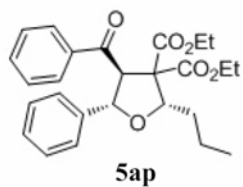
7.75
7.72
7.72
7.51
7.48
7.41
7.40
7.38
7.35
7.32
7.29
7.26

5.07
5.04
5.02
5.00
4.74
4.71
4.70
4.51
4.26
4.24
4.08
4.04
4.03
4.02
4.00
3.97
3.93
3.90
3.88
3.87
3.85
3.84
3.82

1.92
1.91
1.89
1.88
1.57
1.57
1.48
1.44
1.27
1.01
0.99
0.98
0.90
0.87
0.85

-0.00

¹H NMR
300 MHz, CDCl₃



199.83

162.35
162.90

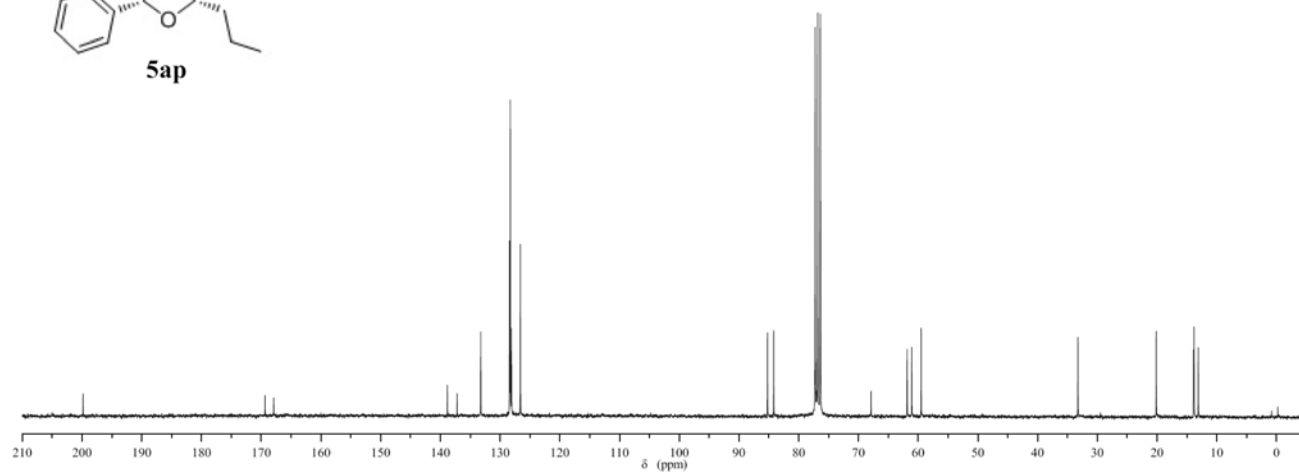
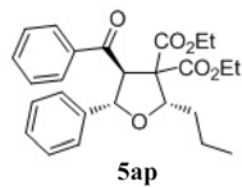
138.83
137.20
133.24
128.42
128.30
128.10
126.61

85.23
84.20
77.23
77.01
76.80
76.58
67.88
61.85
61.67
59.69

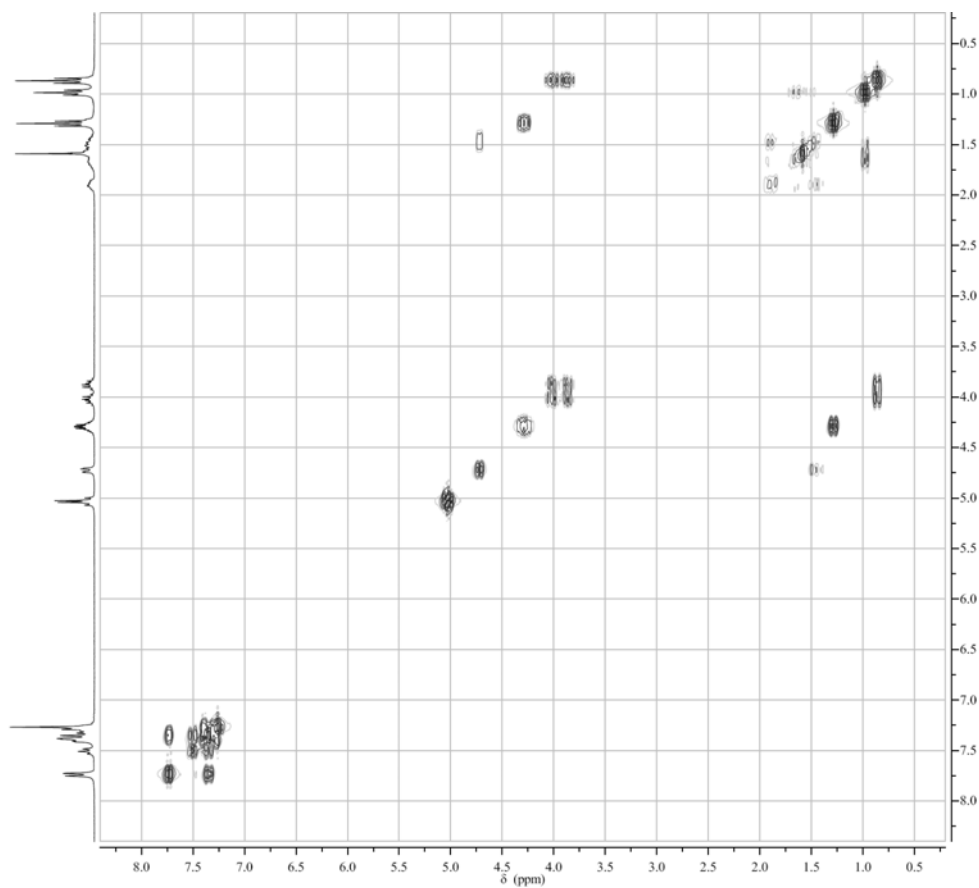
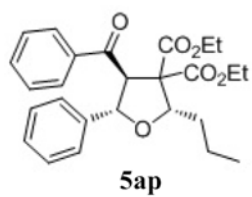
33.26

20.14
13.89
13.80
13.10

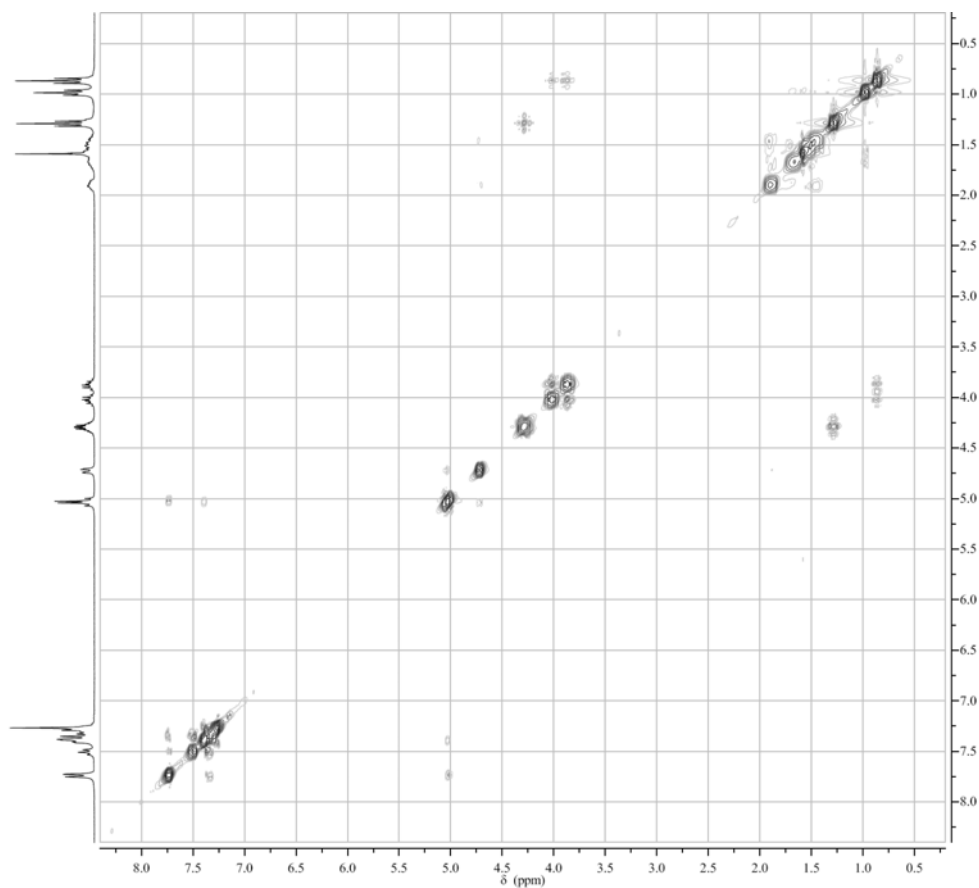
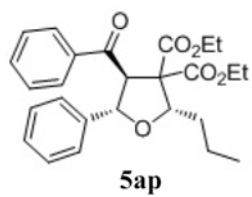
¹³C NMR
75 MHz, CDCl₃



2D COSY NMR
300 MHz, CDCl₃



2D NOESY NMR
300 MHz, CDCl₃



7.84
7.82
7.81
7.45
7.43
7.42
7.39
7.38
7.35
7.33
7.32
7.32
7.28
7.26

5.98

5.30
5.27
5.17
5.14

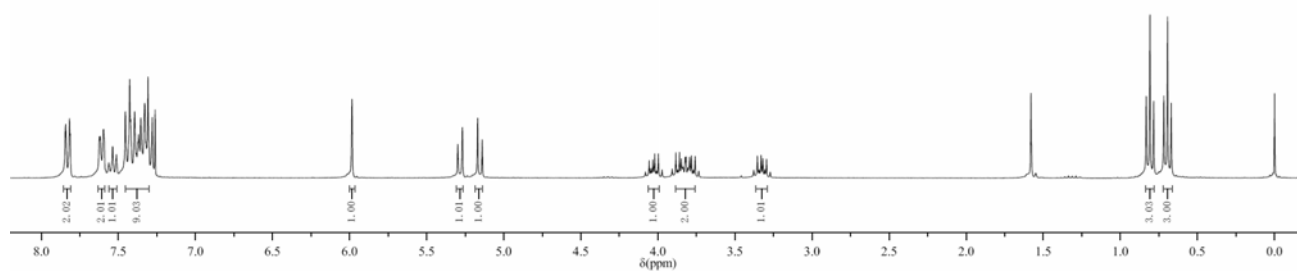
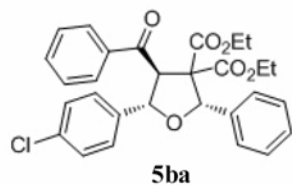
4.06
4.03
4.02
4.00
3.86
3.85
3.79
3.76
3.35
3.32
3.30

1.58
1.58

0.83
0.82
0.78
0.72
0.69
0.67

-0.00

^1H NMR
300 MHz, CDCl_3



199.19

168.81
167.81

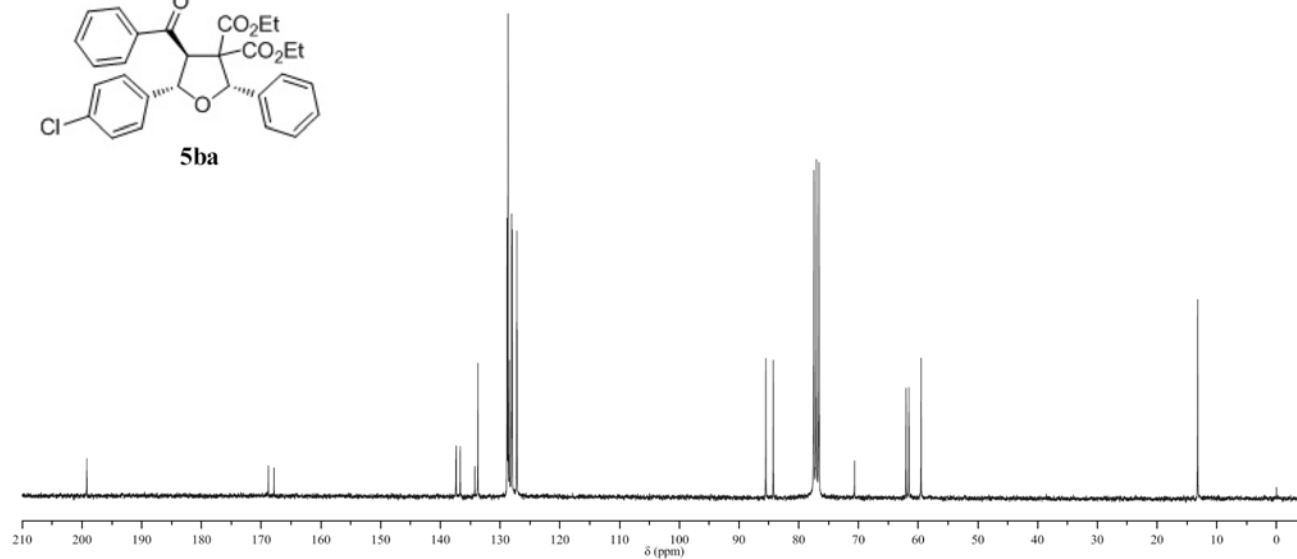
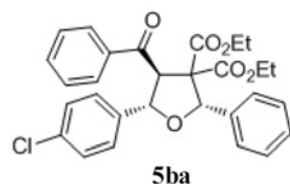
137.38
137.34
136.68
133.72
128.84
128.68
128.43
128.38
127.98
127.19

85.50
84.26
77.45
77.02
76.60
70.66
62.02
61.55
59.51

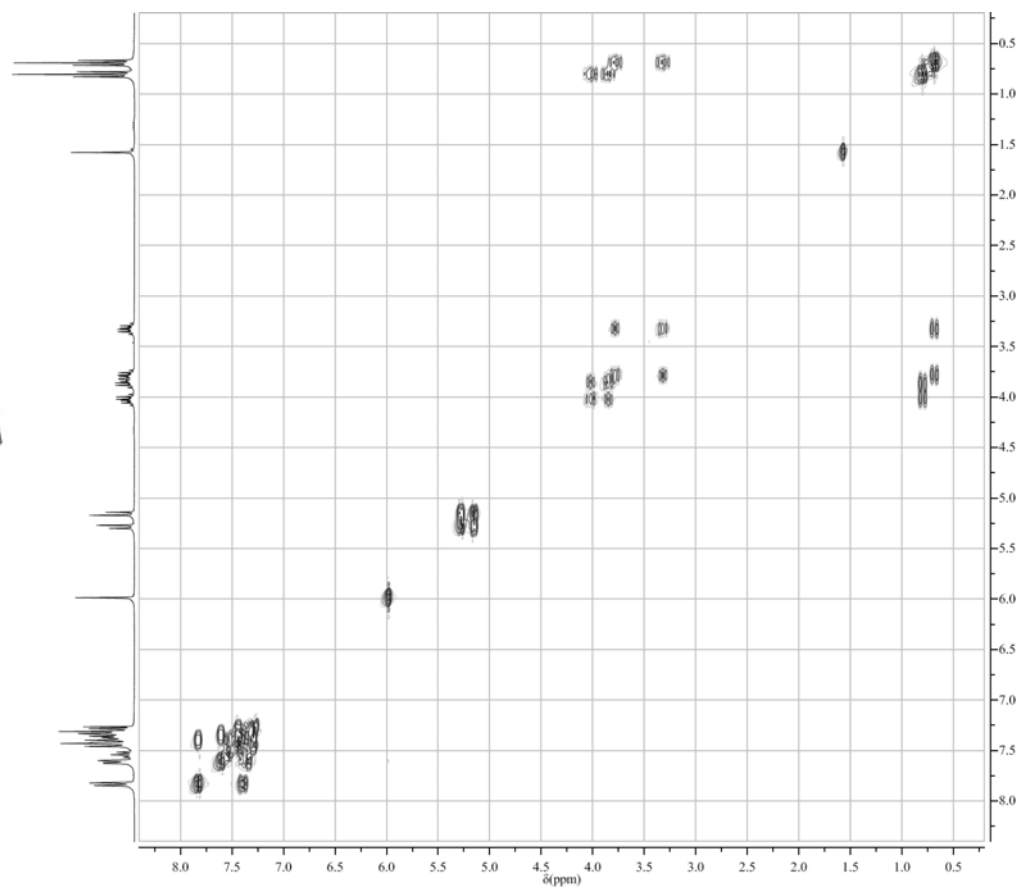
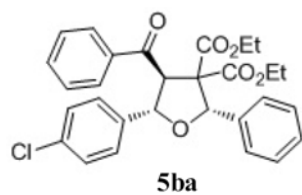
13.20

-0.00

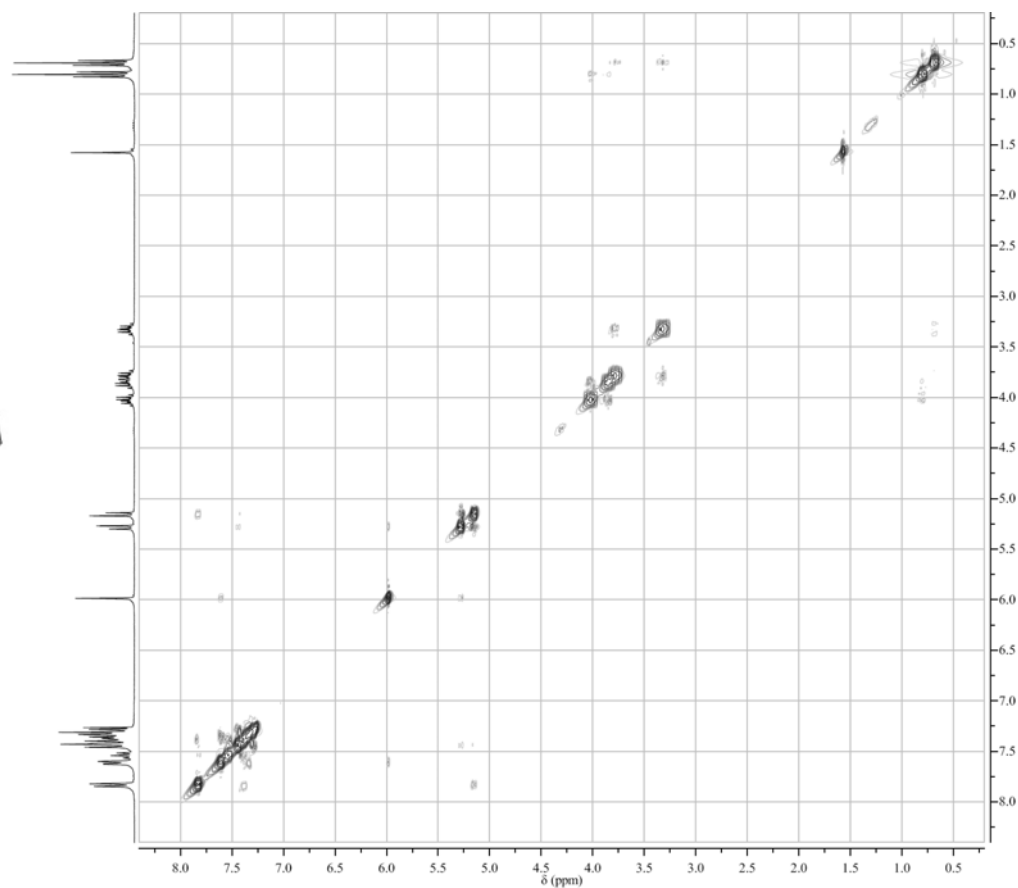
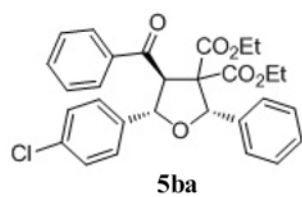
^{13}C NMR
75 MHz, CDCl_3

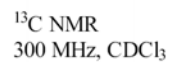
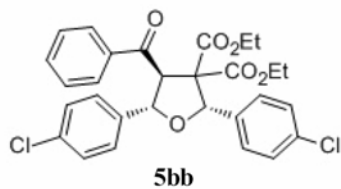
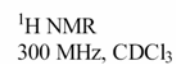


2D COSY NMR
300 MHz, CDCl₃

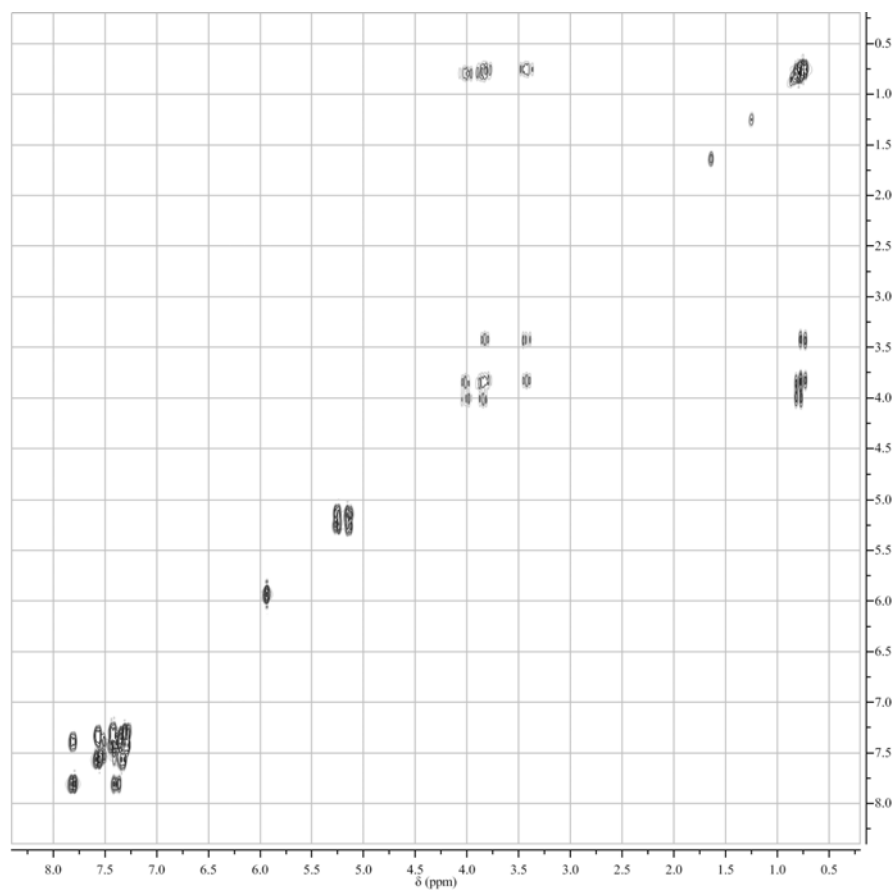
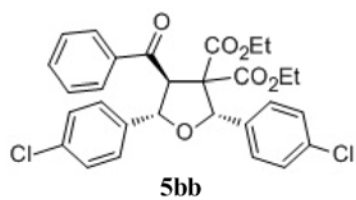


2D NOESY NMR
300 MHz, CDCl₃

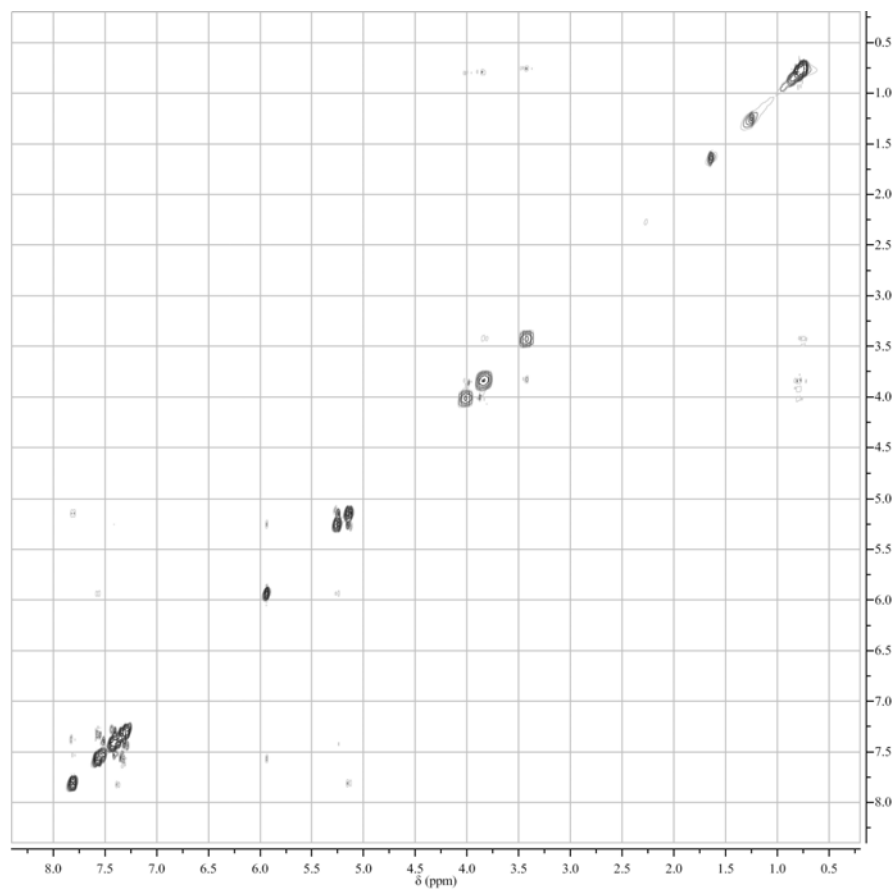
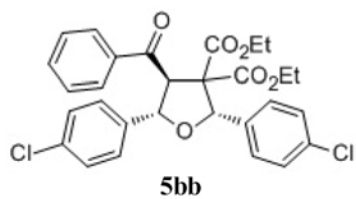


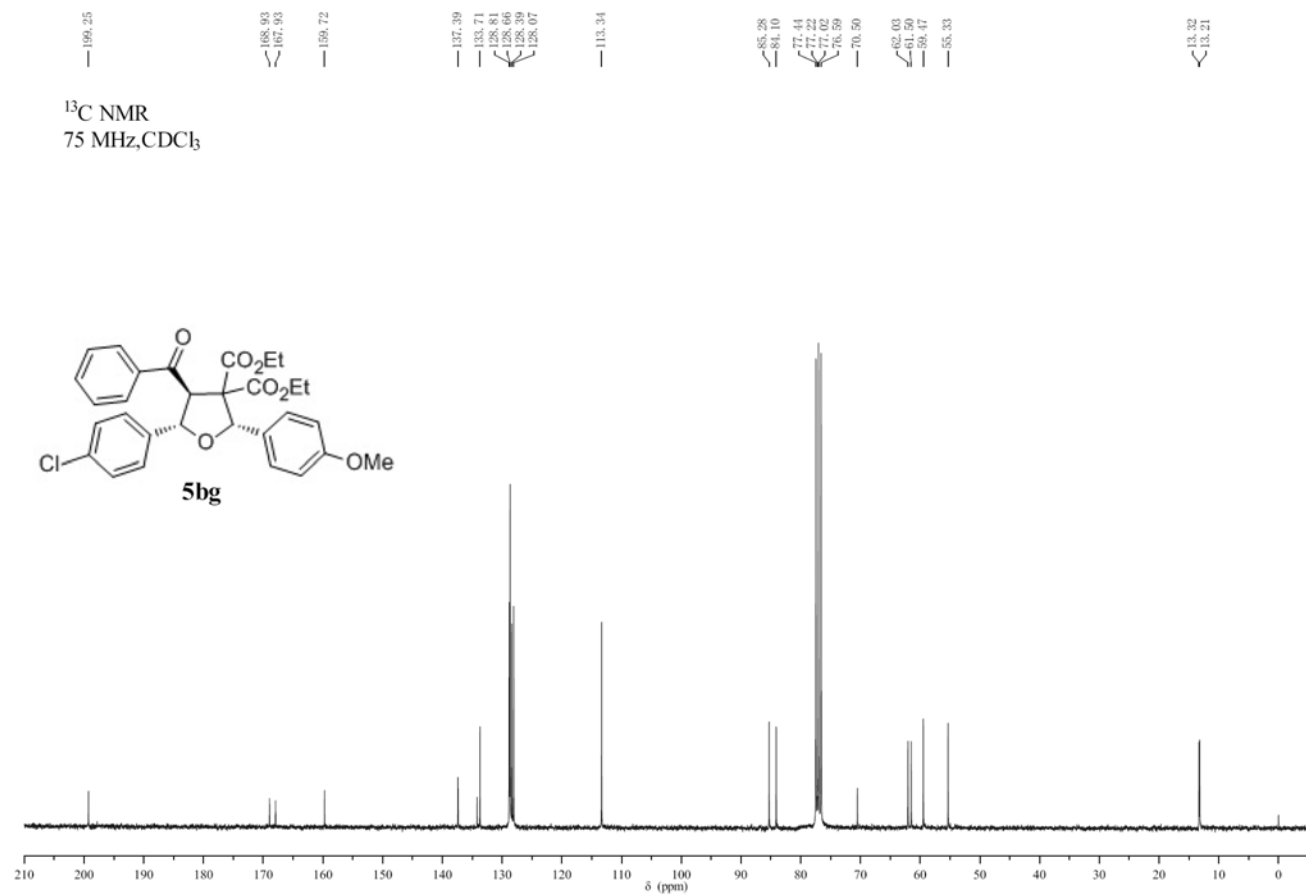
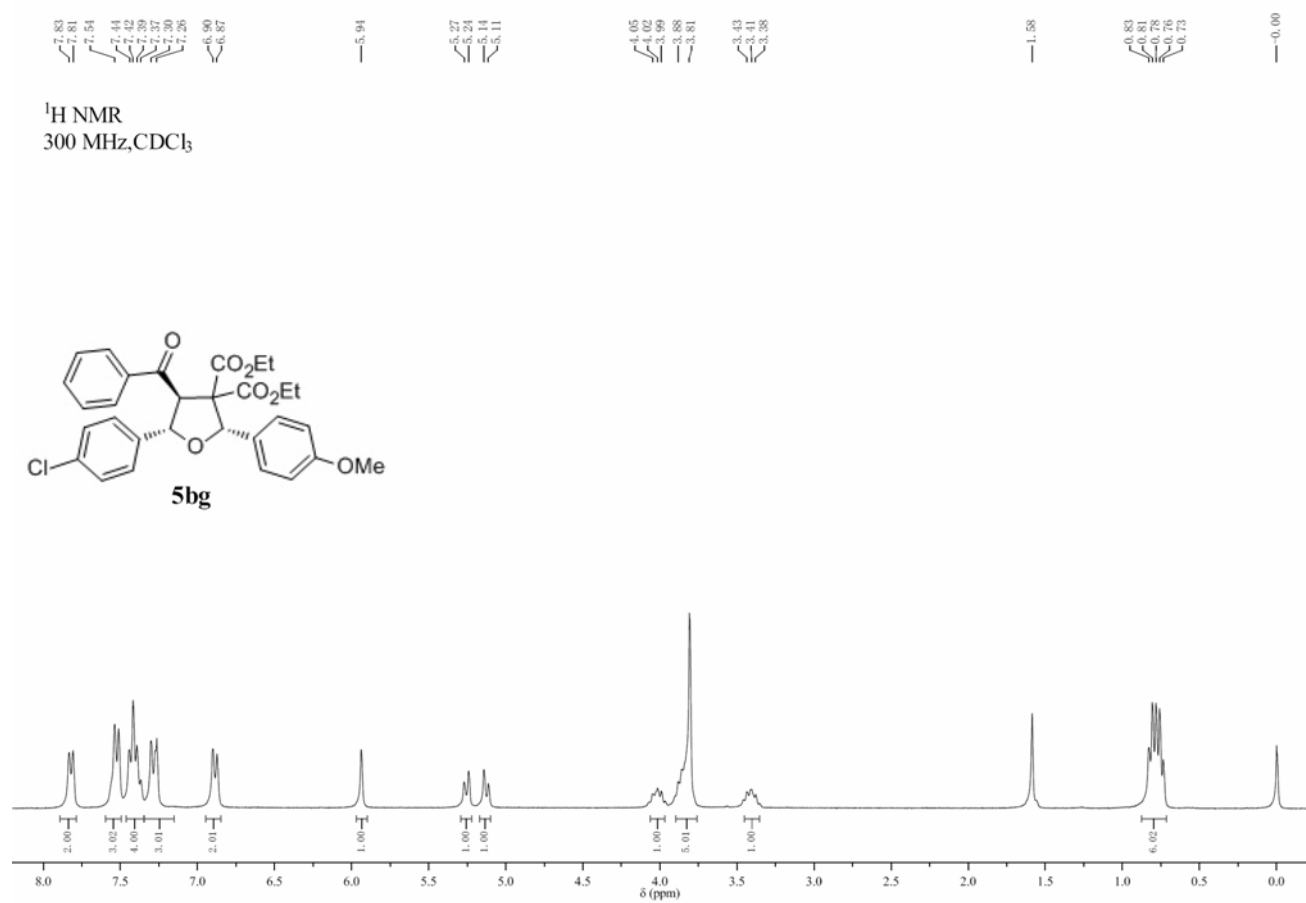


2D COSY NMR
300 MHz, CDCl₃

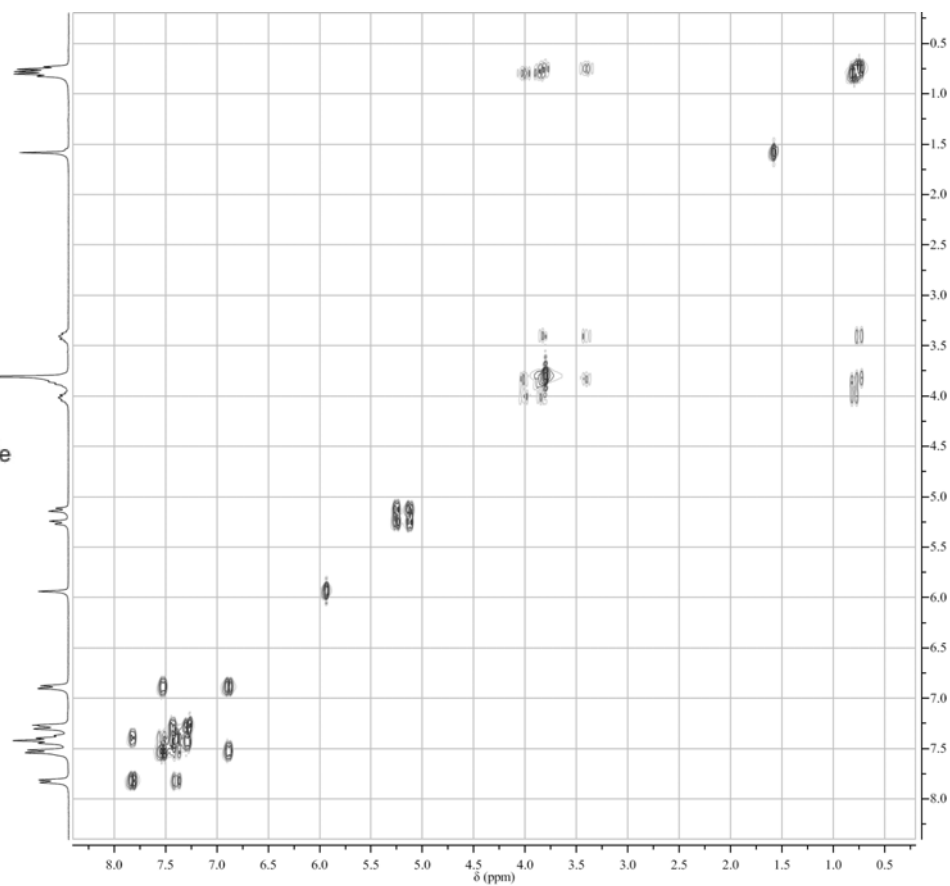
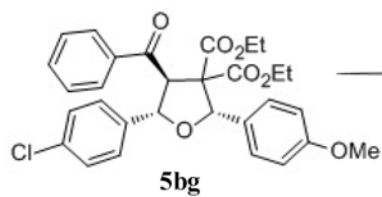


2D NOESY NMR
300 MHz, CDCl₃

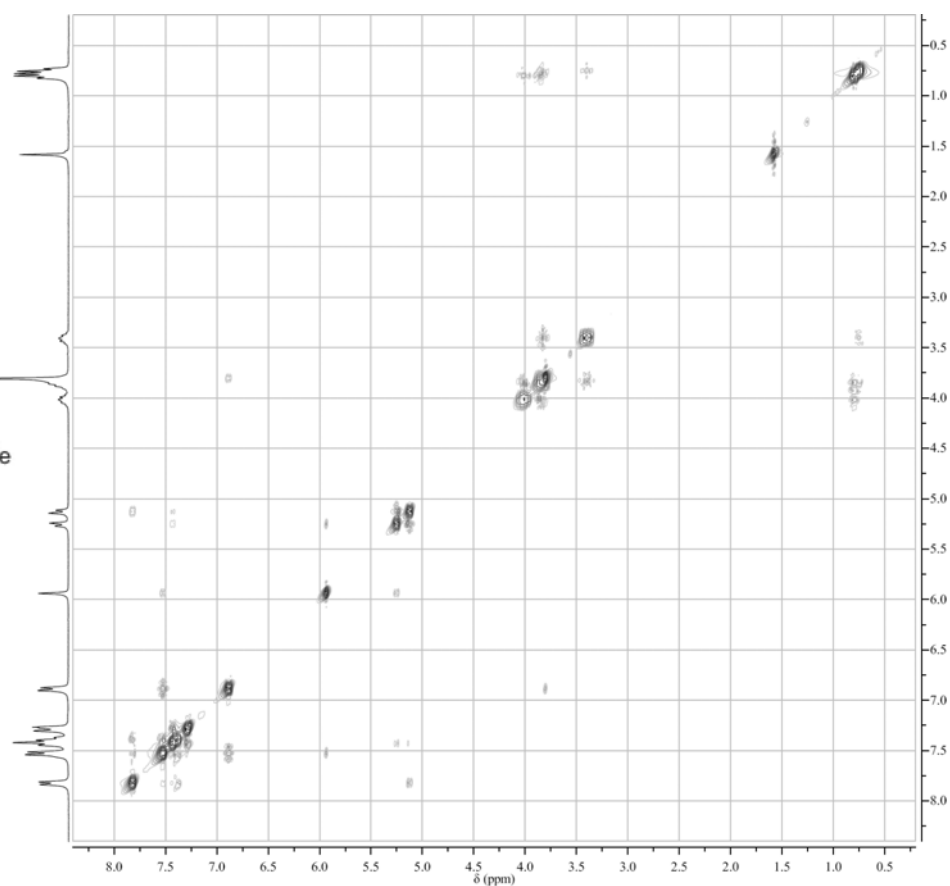
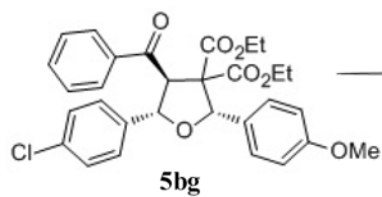


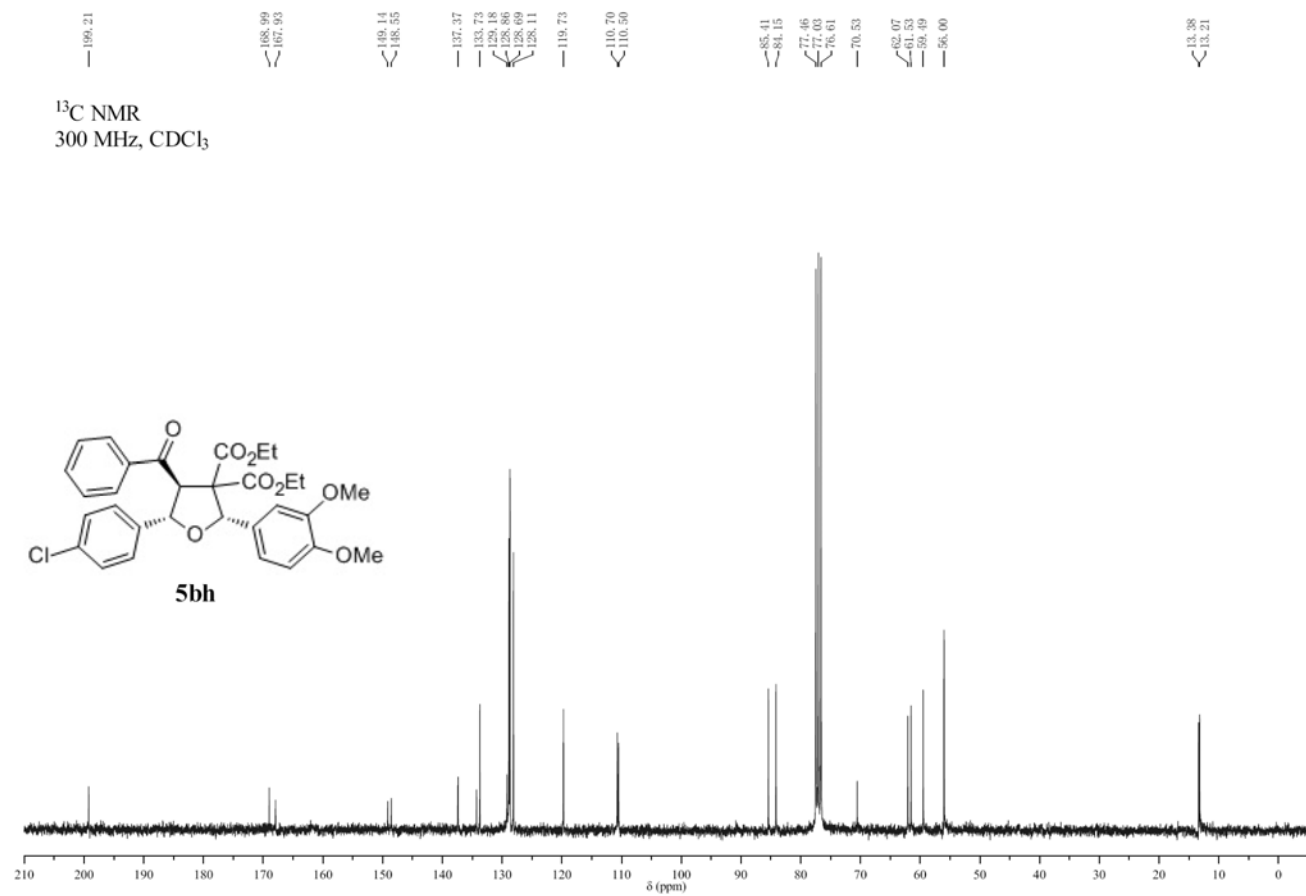
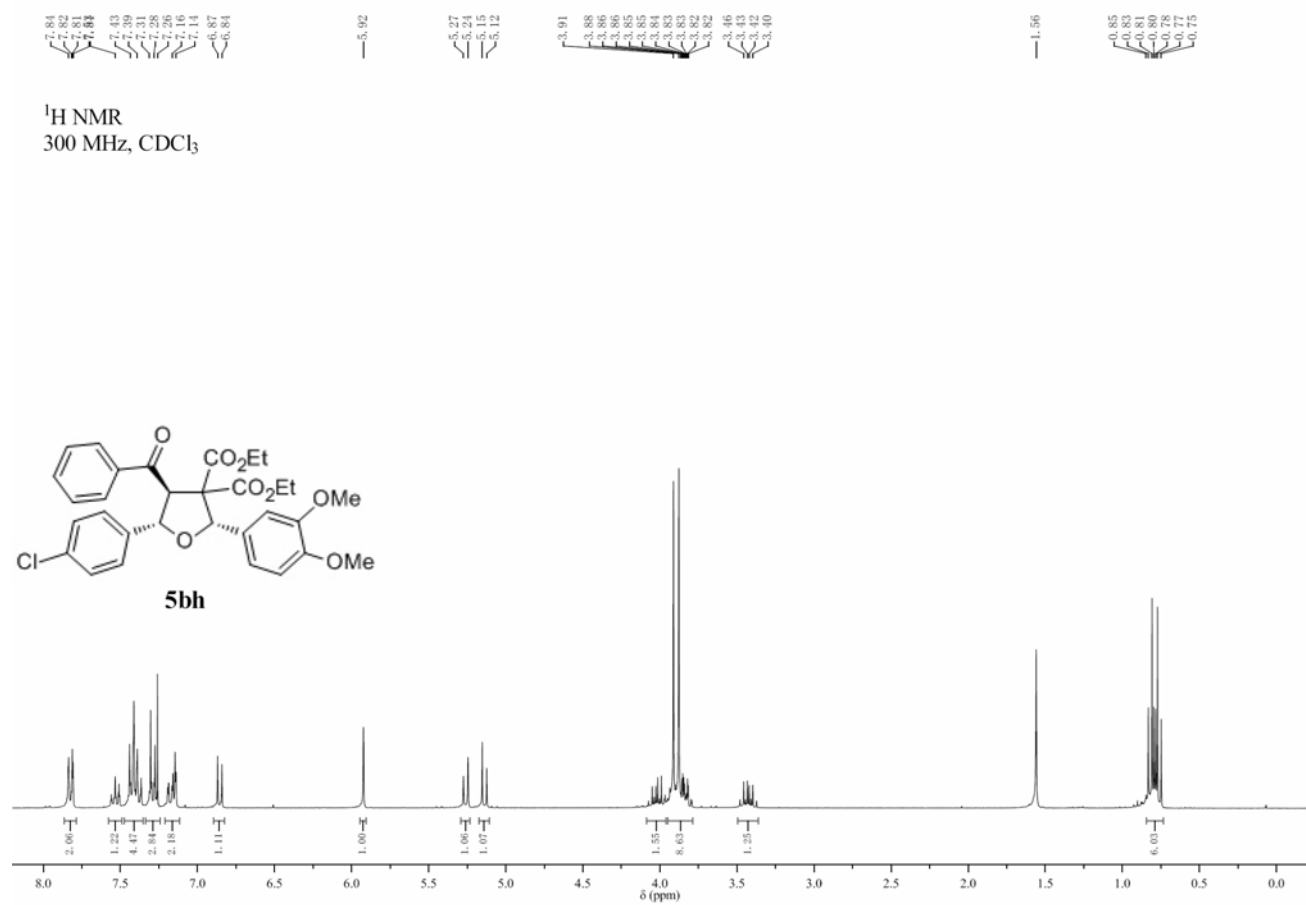


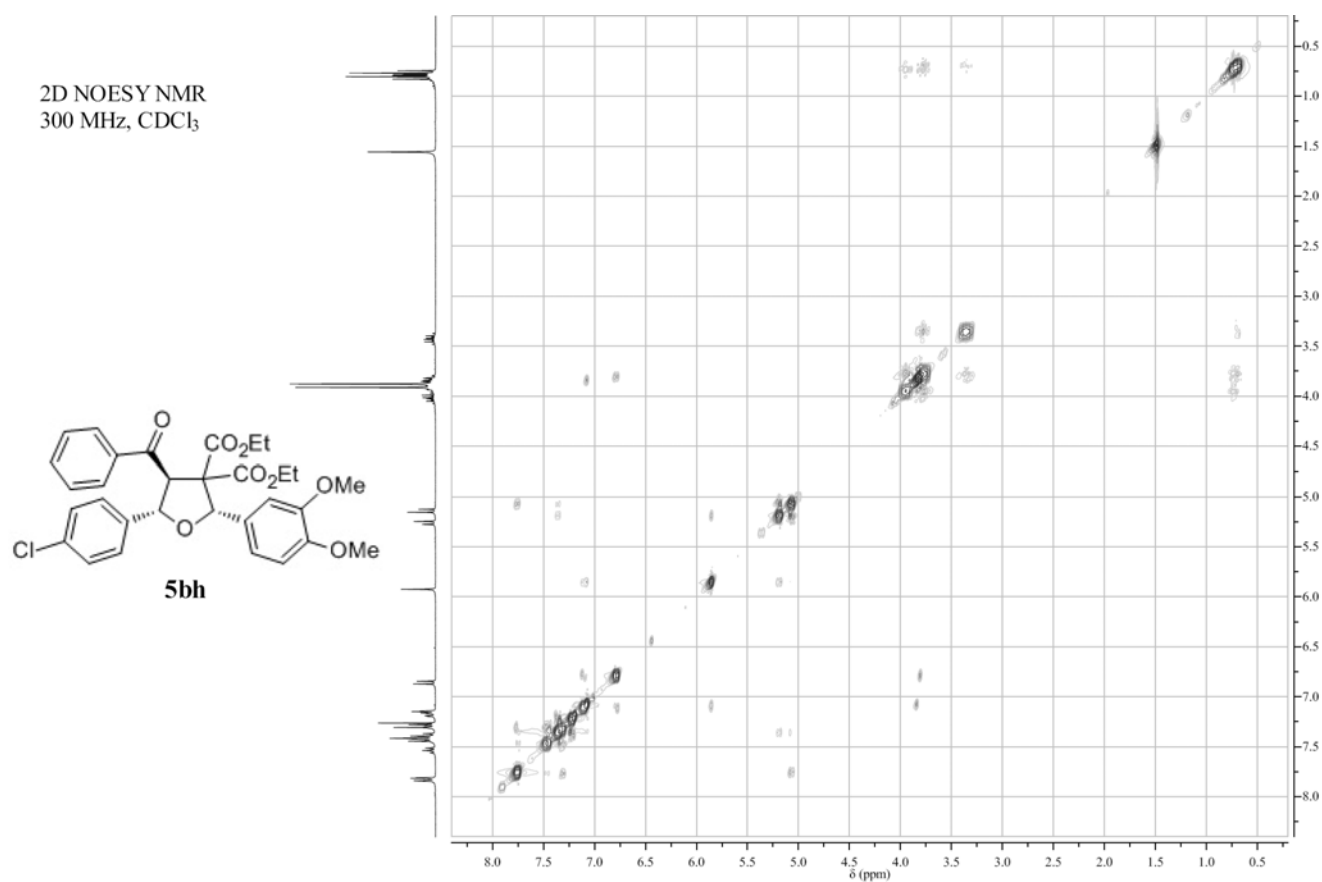
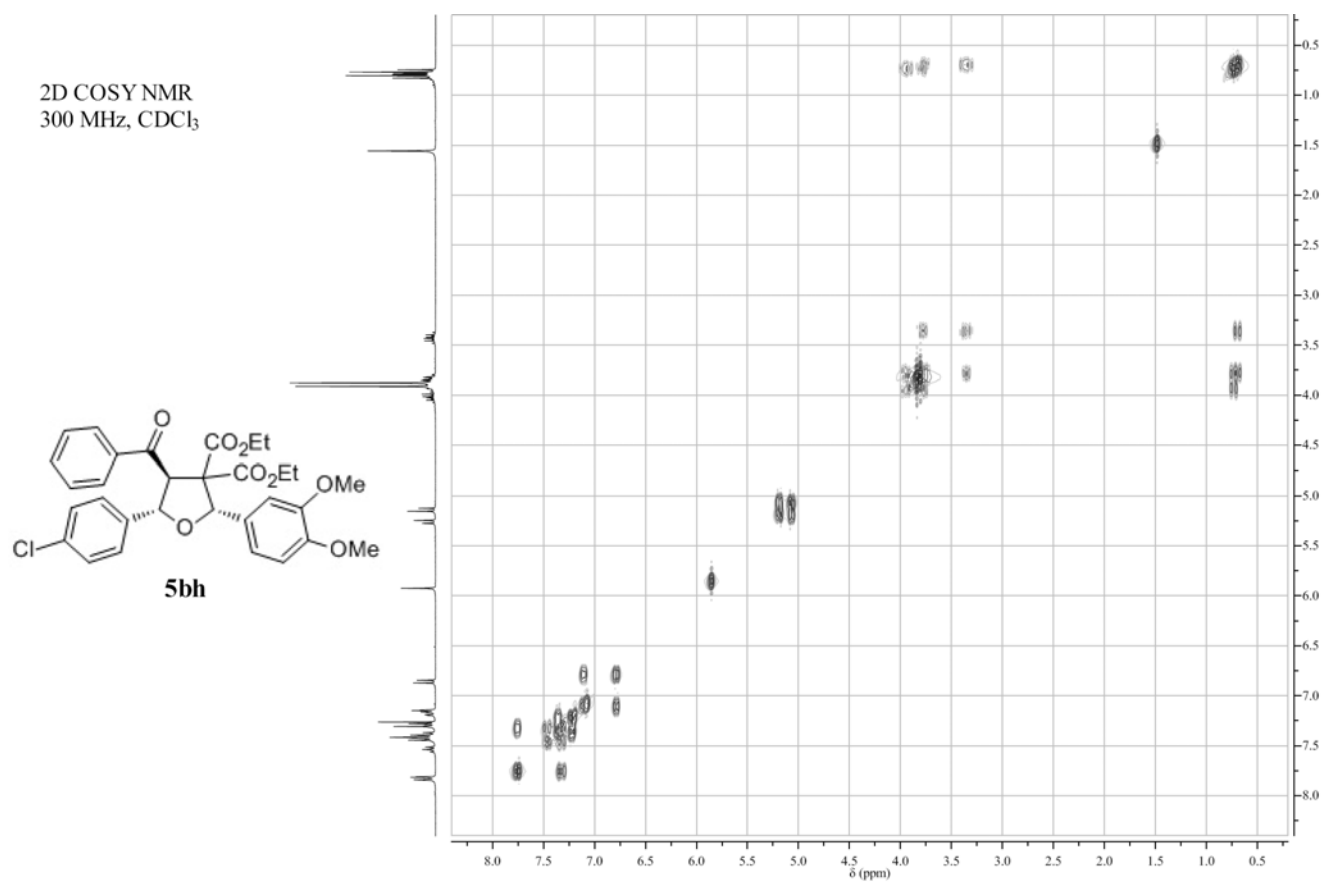
2D COSY NMR
300 MHz, CDCl₃



2D NOESY NMR
300 MHz, CDCl₃







8.29
8.17
7.86
7.83
7.56
7.54
7.36
7.26

6.03

5.45
5.42
5.14
5.11

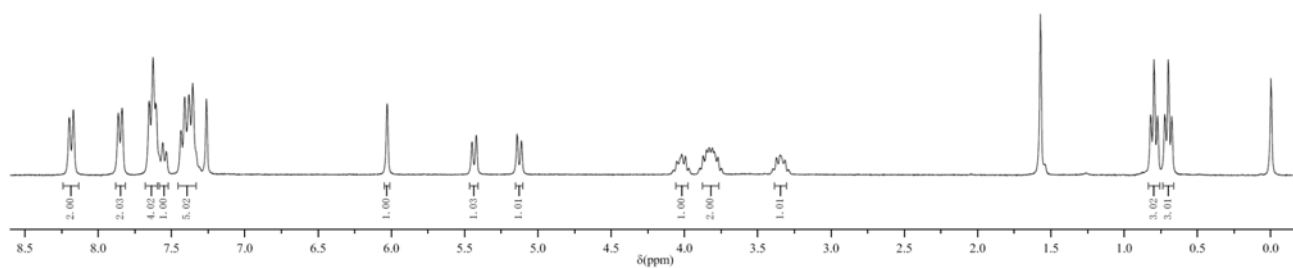
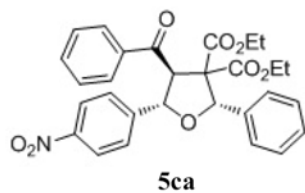
4.05
4.02
3.99
3.85
3.83
3.81
3.77
3.37
3.35
3.31

1.57

0.82
0.80
0.77
0.72
0.70
0.68

0.00

¹H NMR
300 MHz, CDCl₃



198.82

168.64
167.54

147.92
146.16

137.20
136.34
134.01

128.62
127.16
123.91

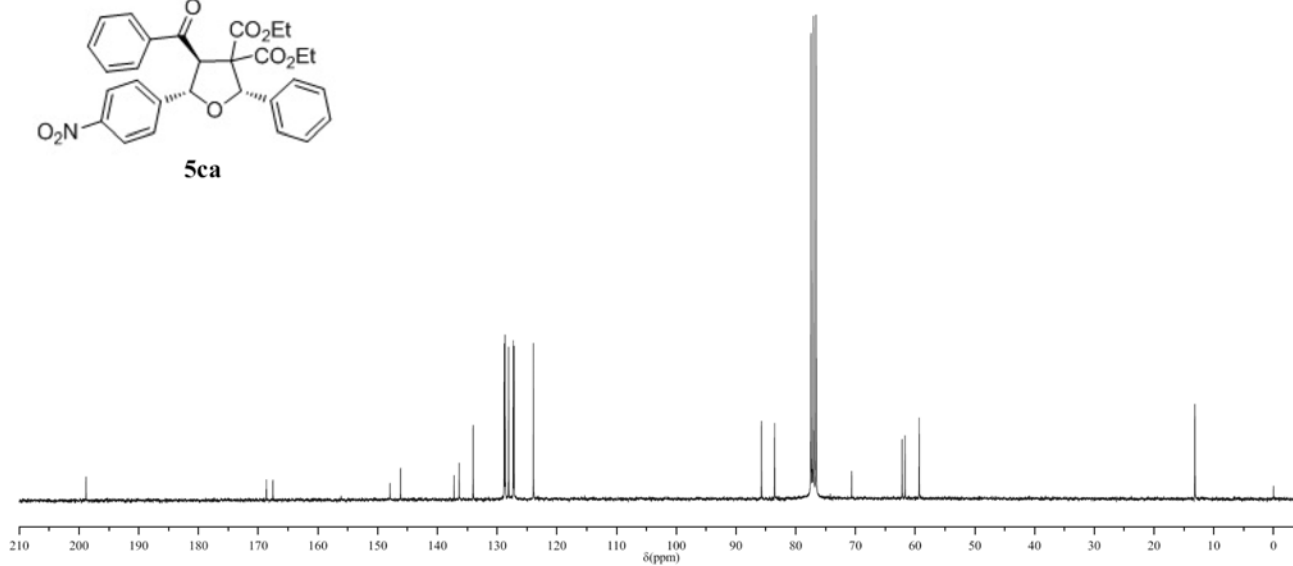
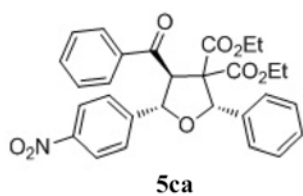
85.73
83.54

77.44
77.22
77.02
76.89
70.64

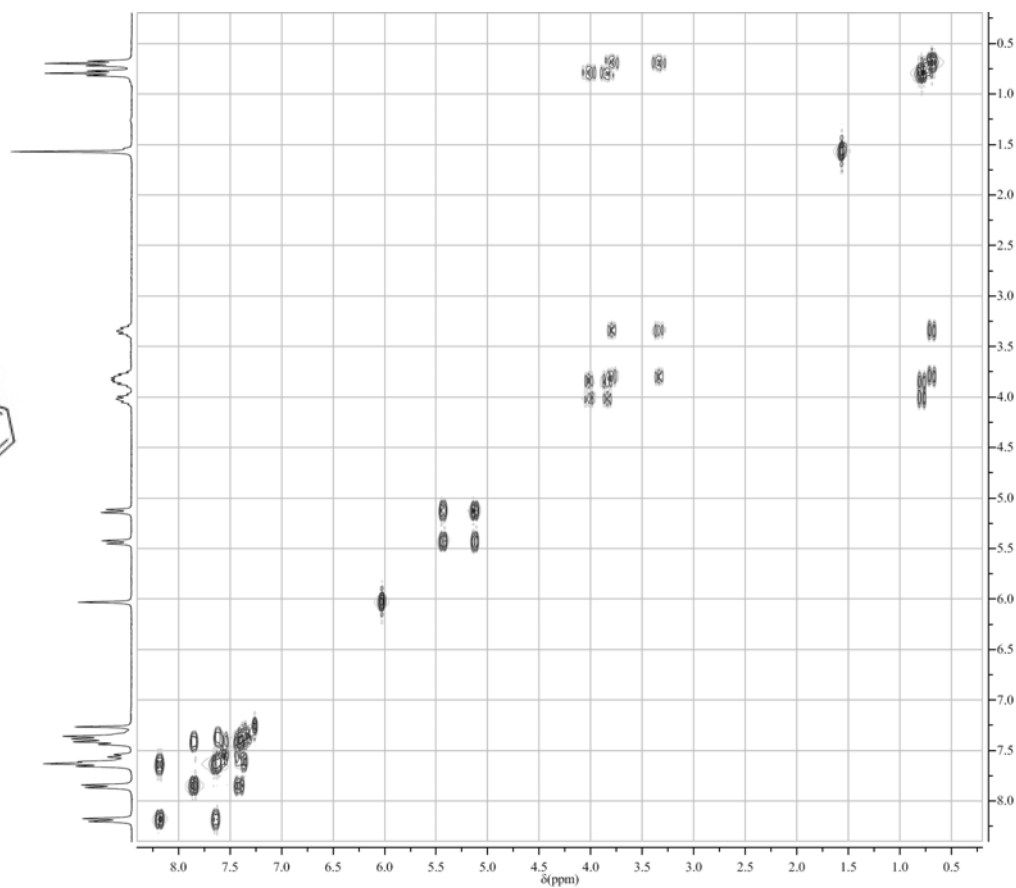
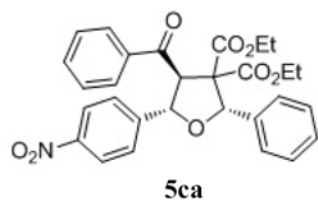
62.19
61.70
59.32

13.17

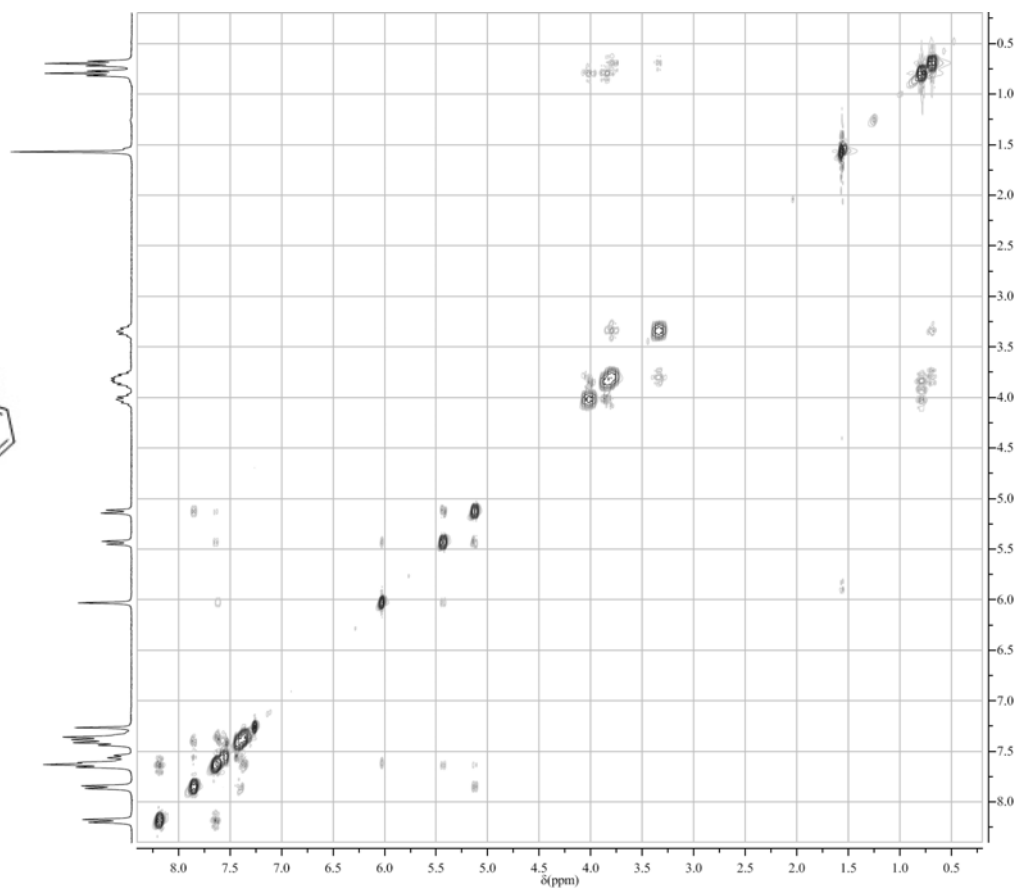
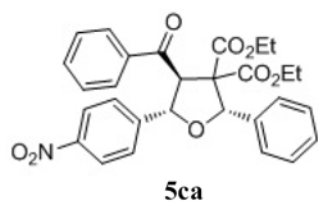
¹³C NMR
75 MHz, CDCl₃



2D COSY NMR
300 MHz, CDCl₃



2D NOESY NMR
300 MHz, CDCl₃



8.29
8.17
7.84
7.82
7.56
7.44
7.41
7.37
7.34
7.26

5.98

5.42
5.39
5.13
5.10

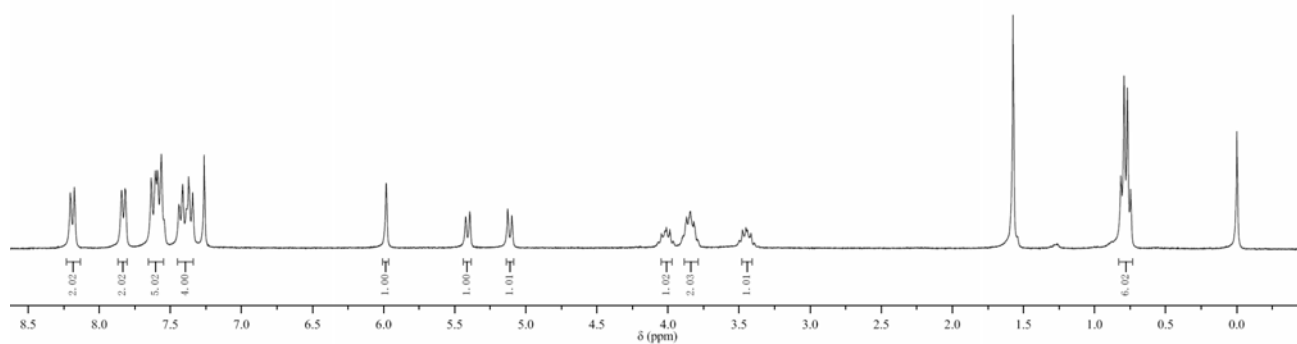
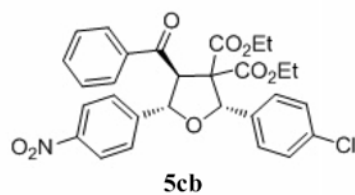
4.05
4.02
3.99
3.87
3.84
3.82
3.48
3.45
3.42

1.57

0.82
0.79
0.77
0.75

0.00

¹H NMR
300 MHz, CDCl₃



198.66

168.36
167.32

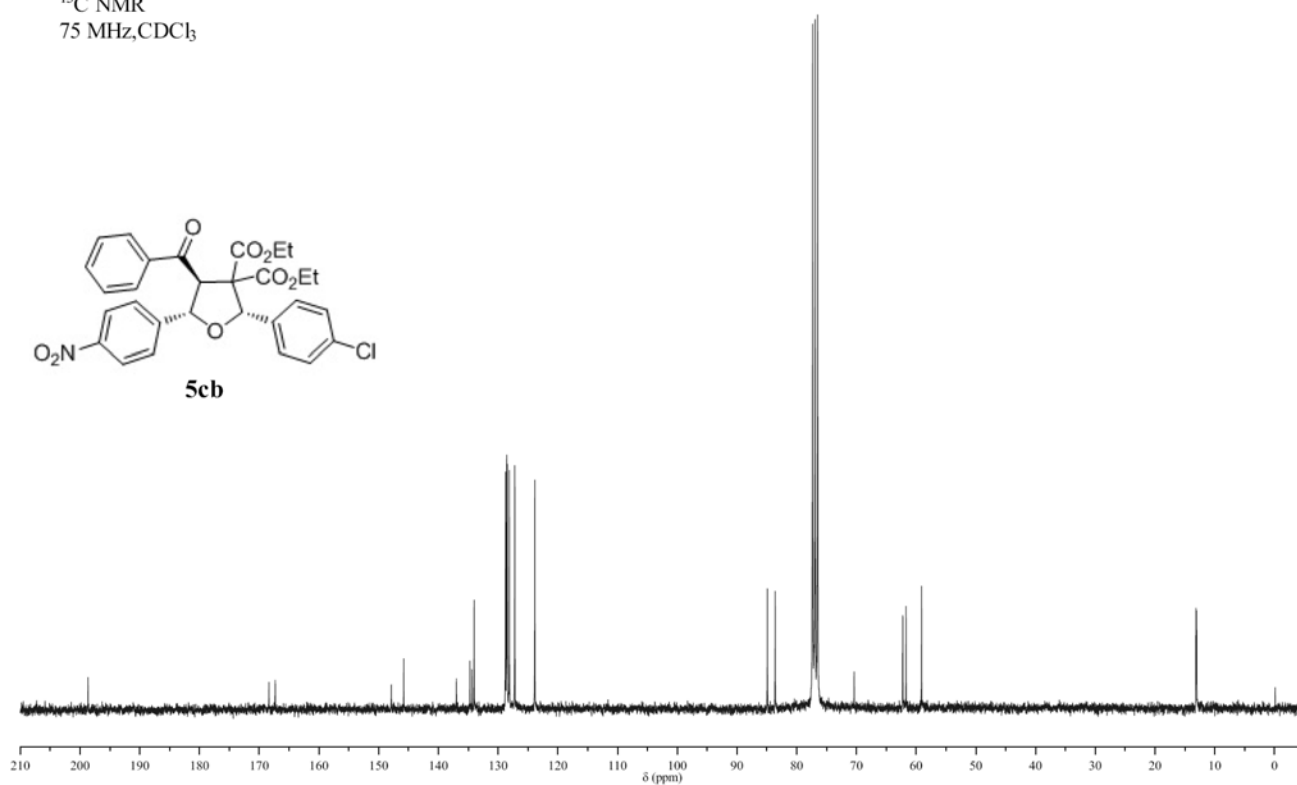
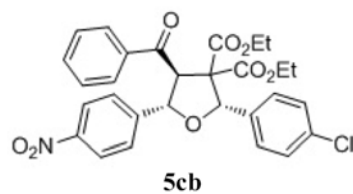
147.88
145.80

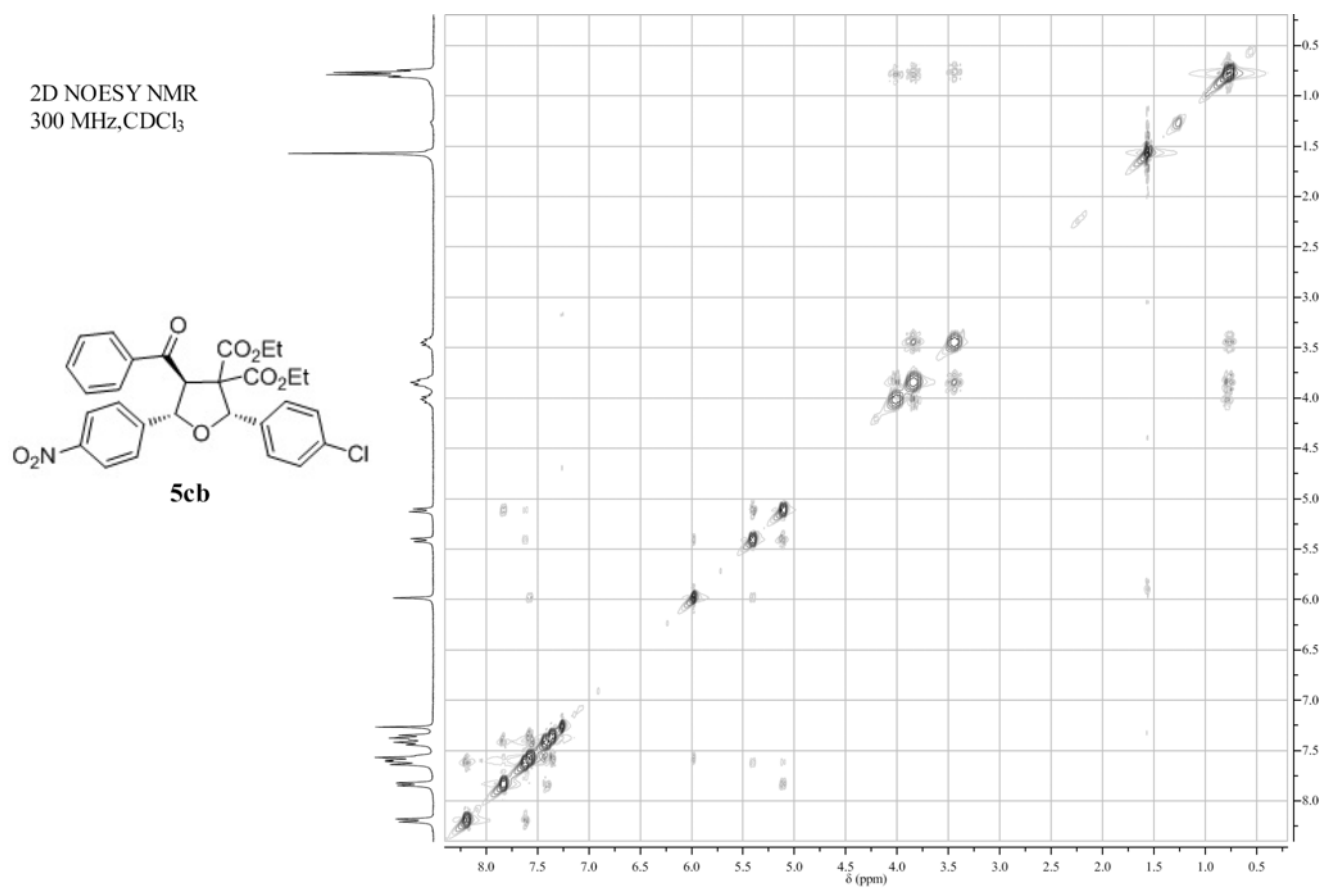
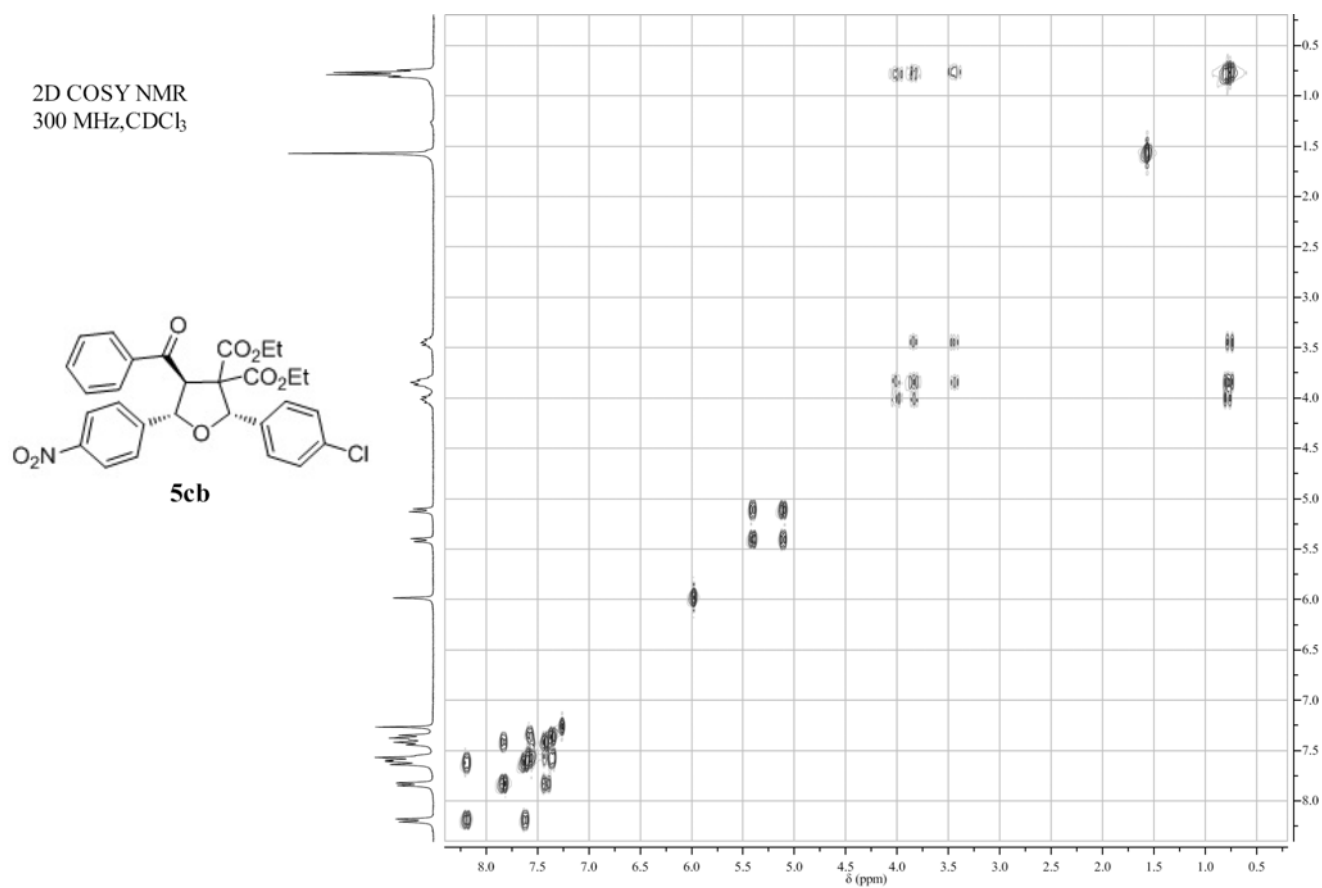
138.98
138.12
134.01
128.46
127.21
123.85

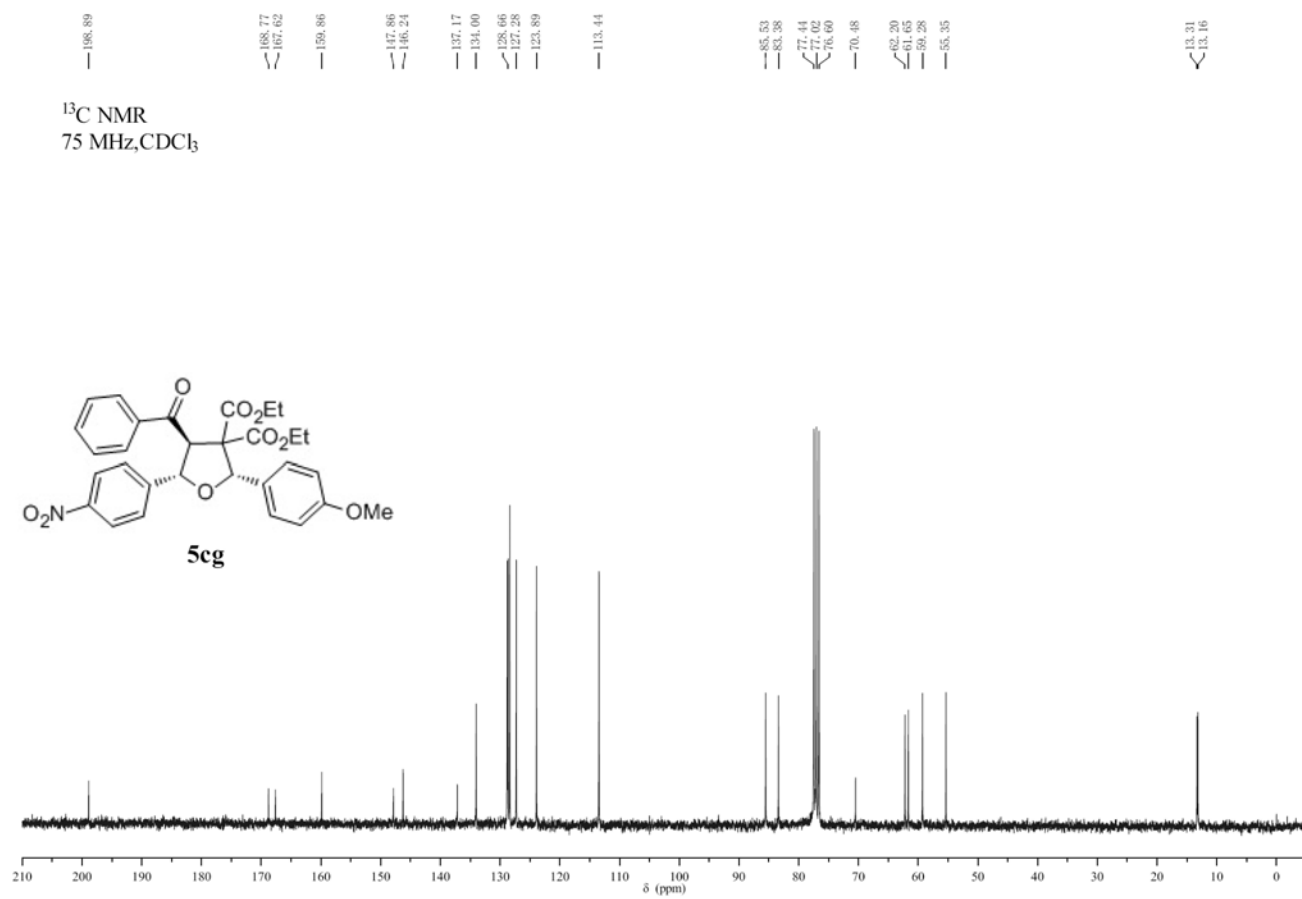
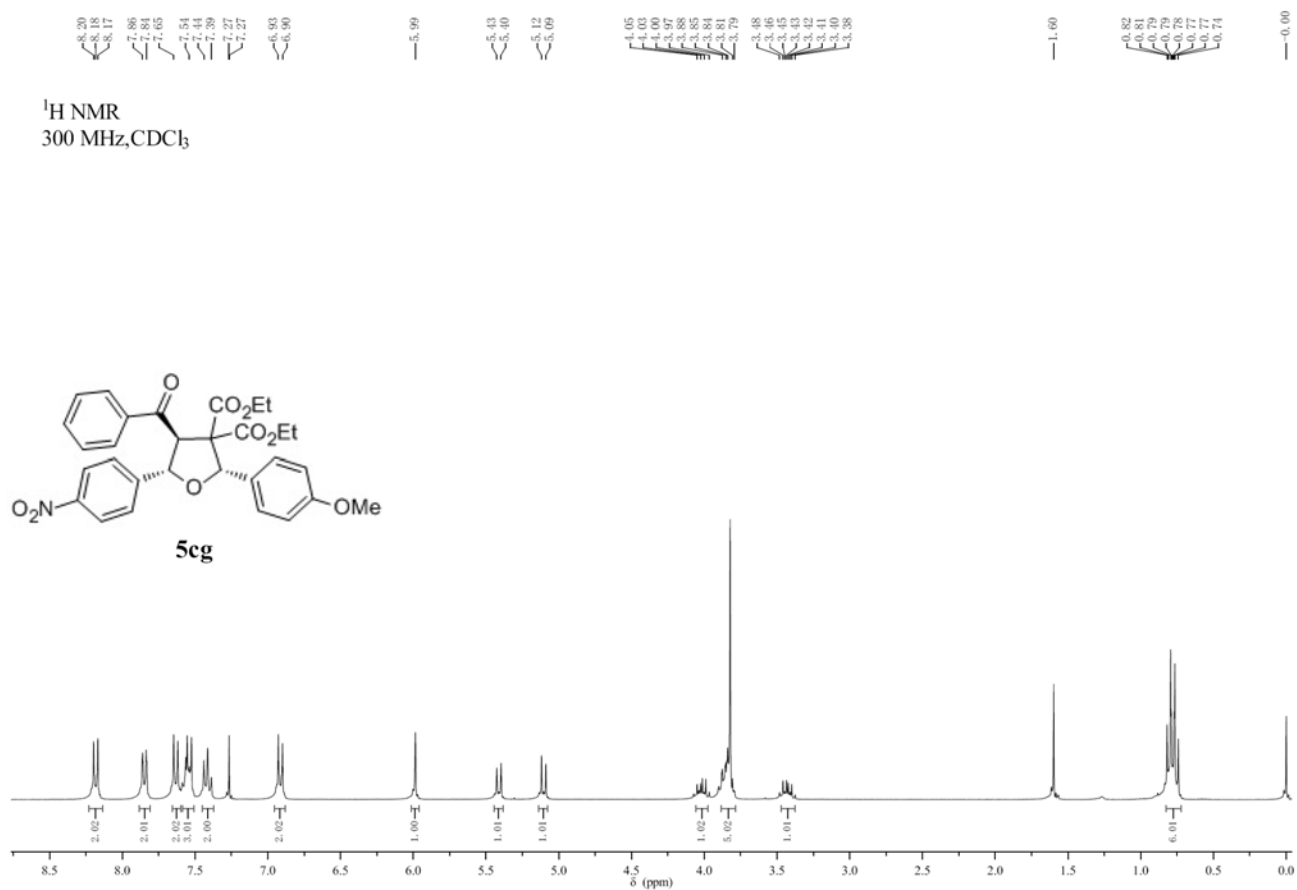
84.90
83.60
77.34
76.91
76.49
70.38
62.25
61.69
59.10

13.15
13.04

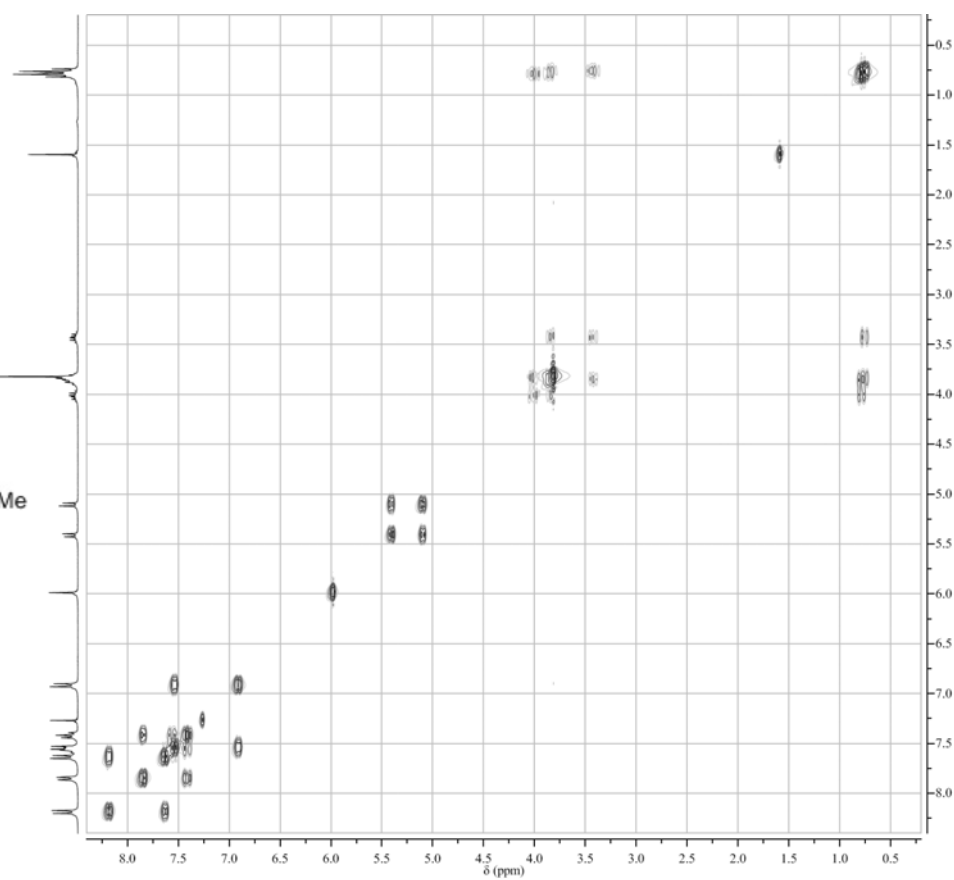
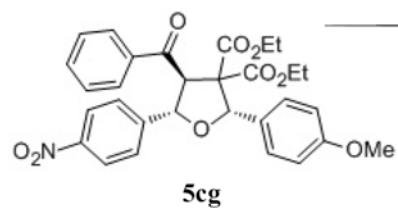
¹³C NMR
75 MHz, CDCl₃



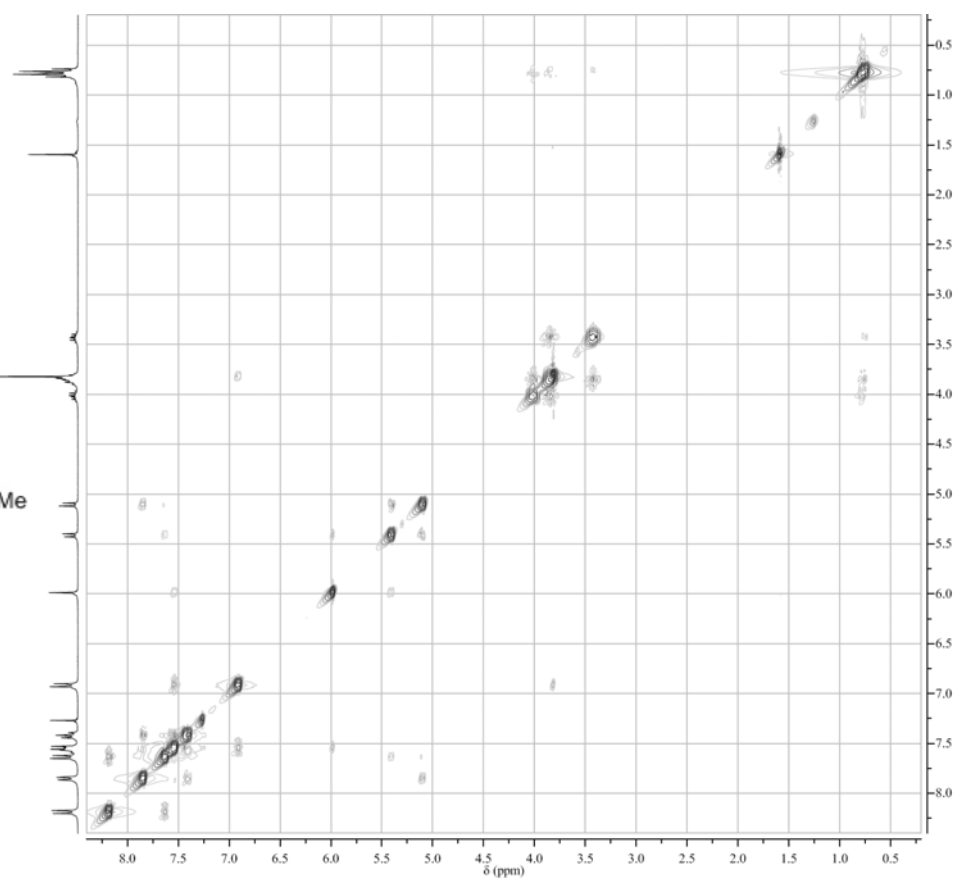
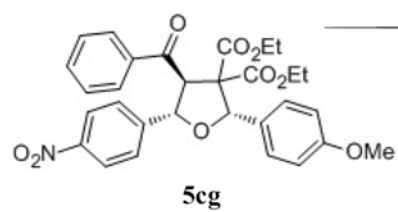


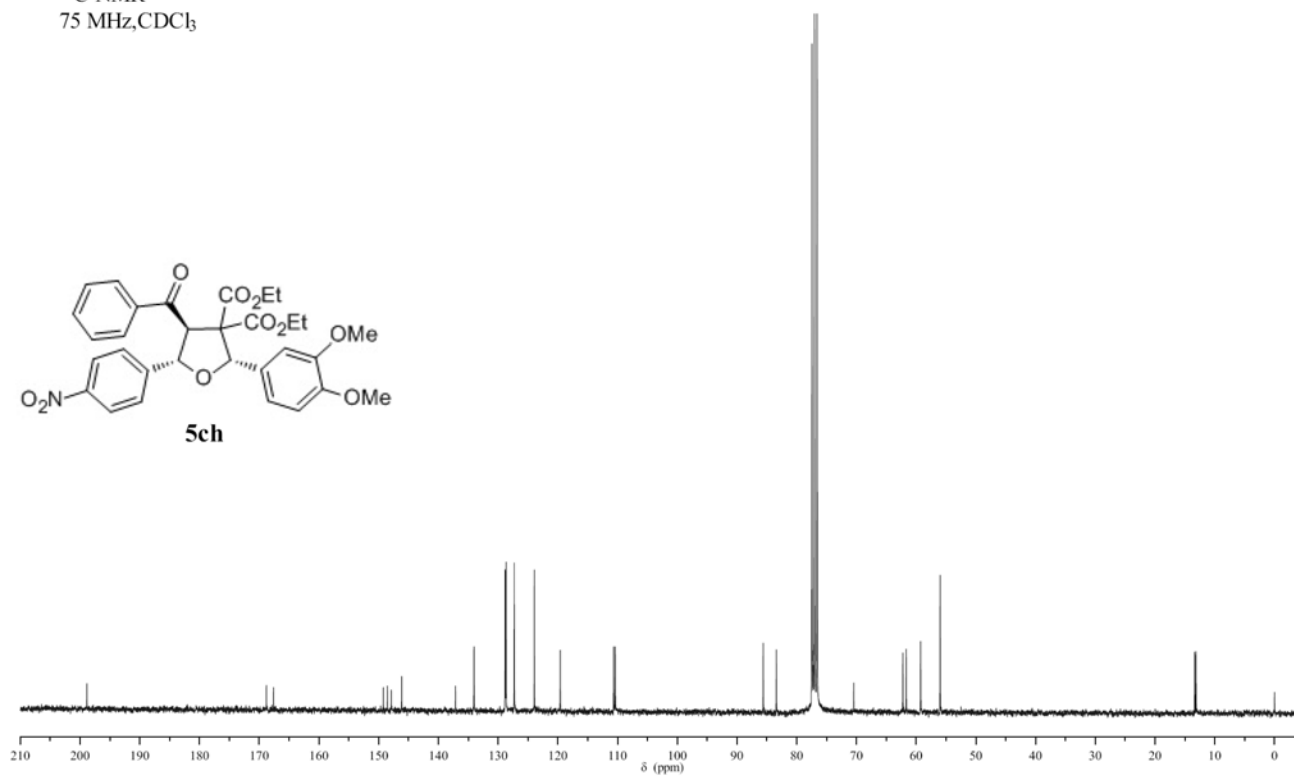
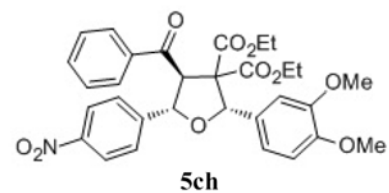
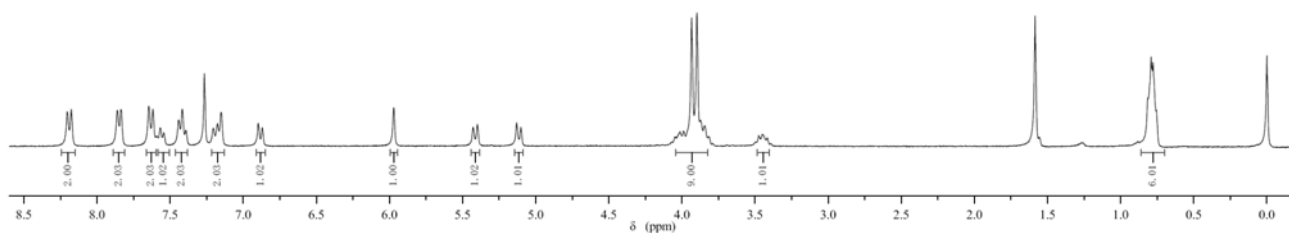
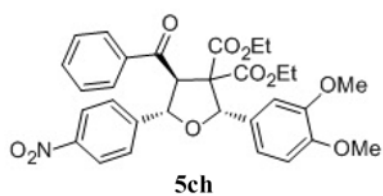


2D COSY NMR
300 MHz, CDCl₃

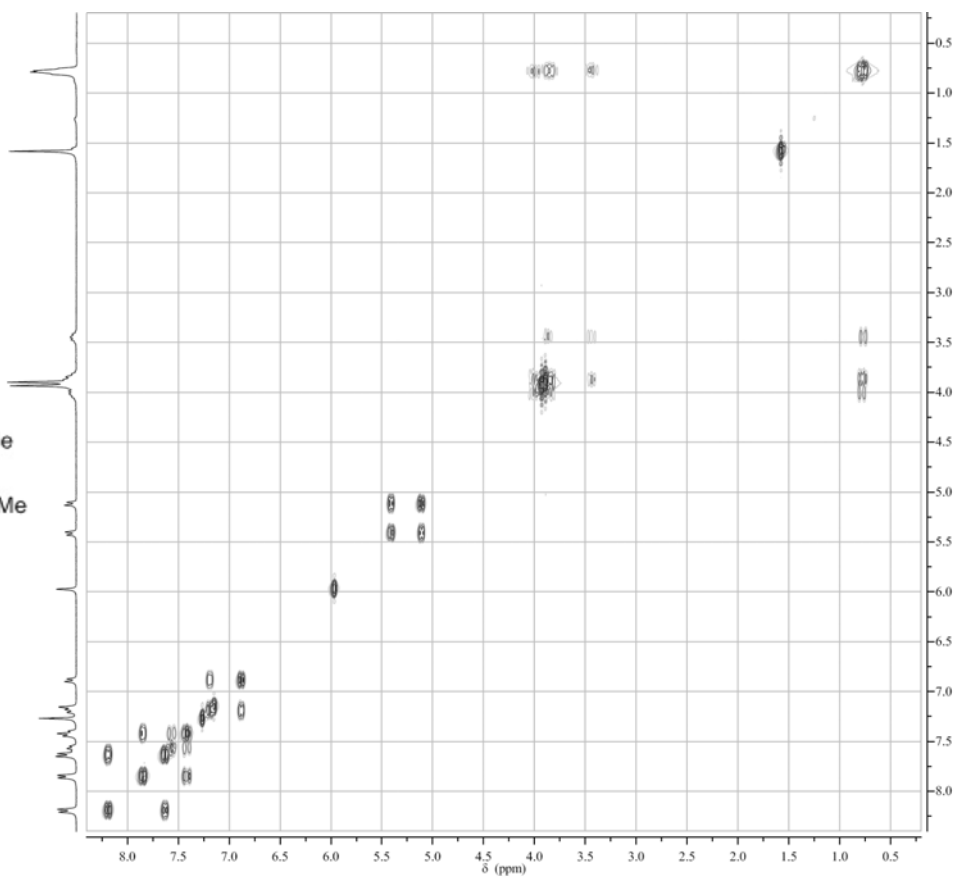
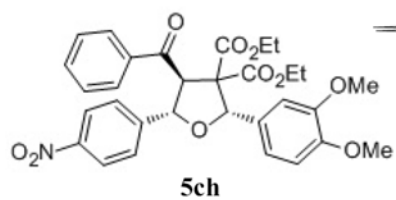


2D NOESY NMR
300 MHz, CDCl₃

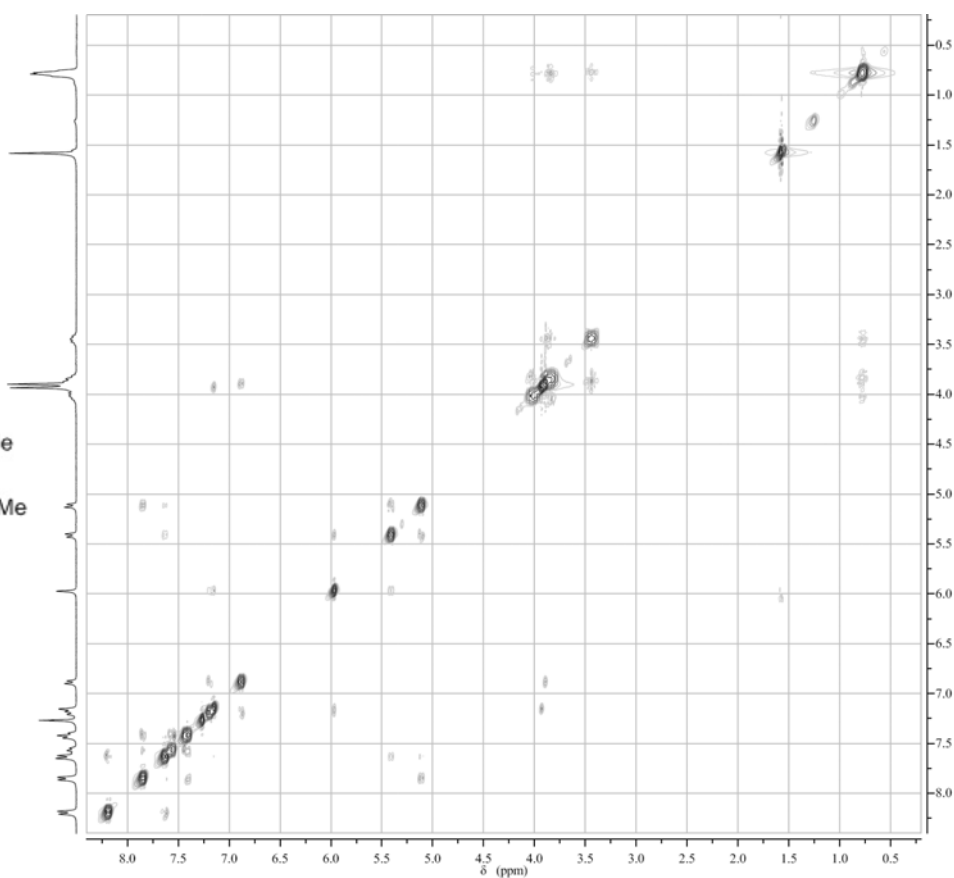
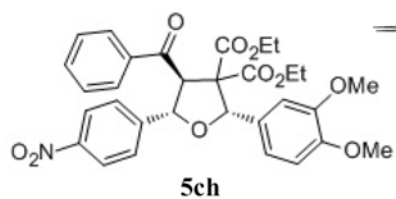


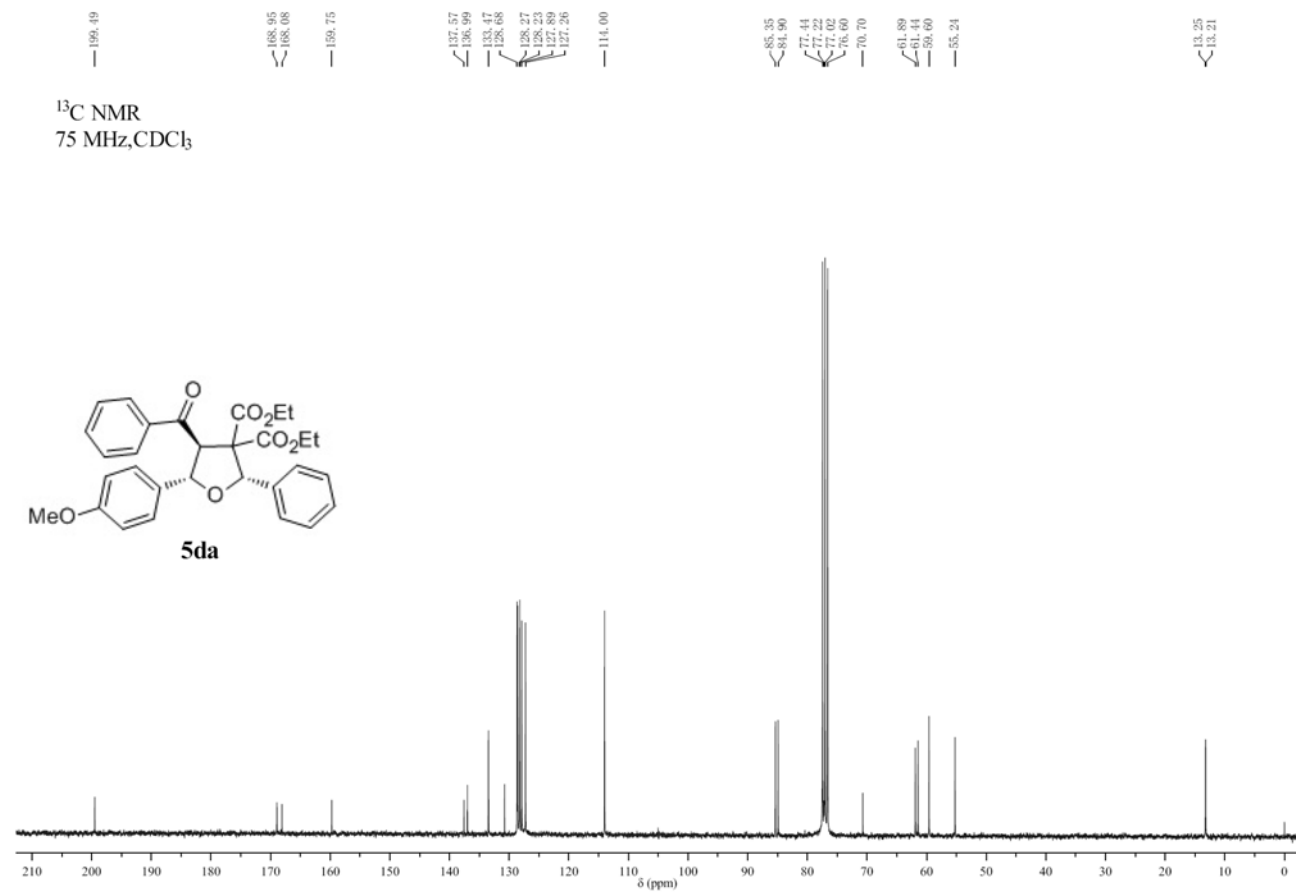
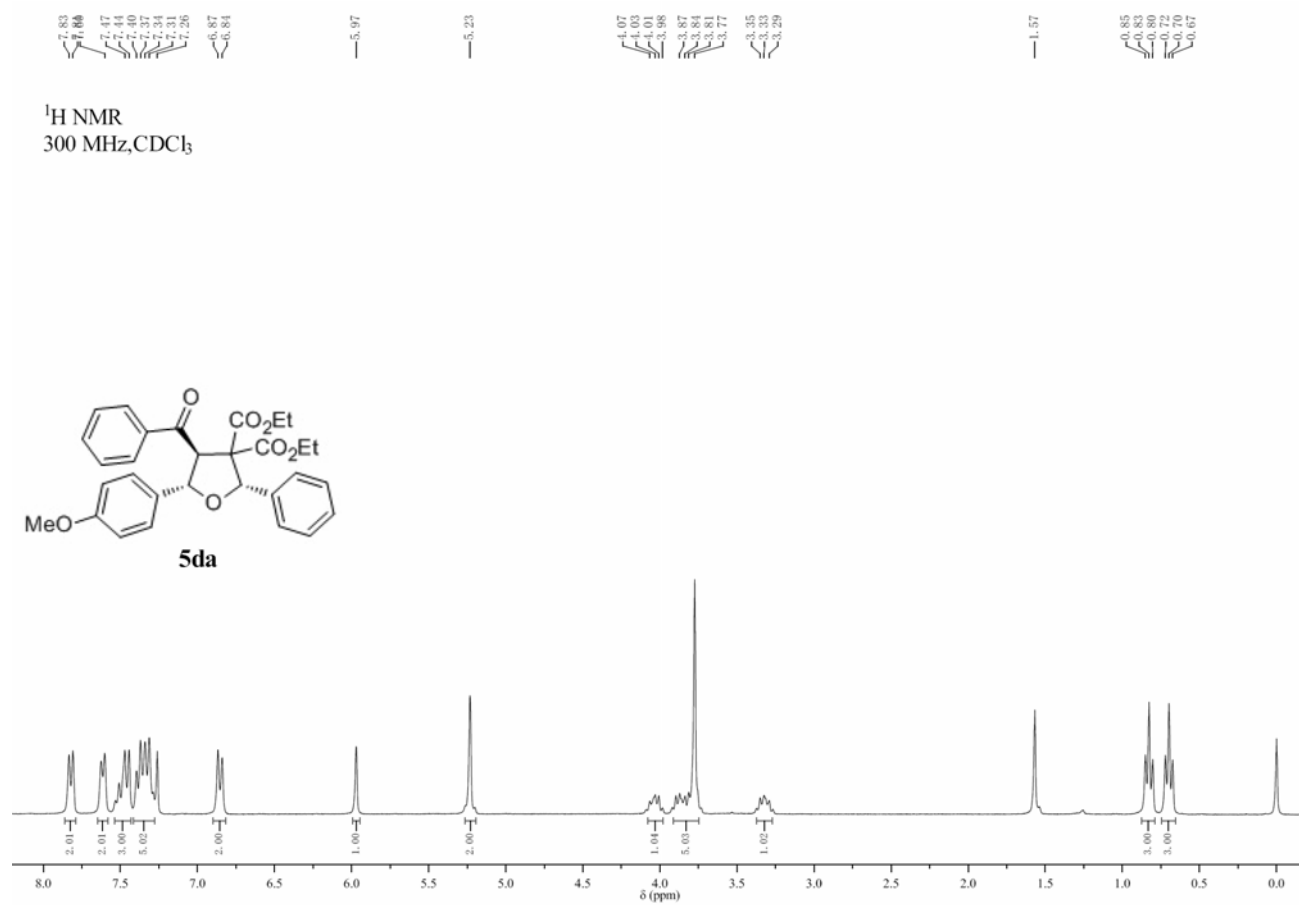


2D COSY NMR
300 MHz, CDCl₃

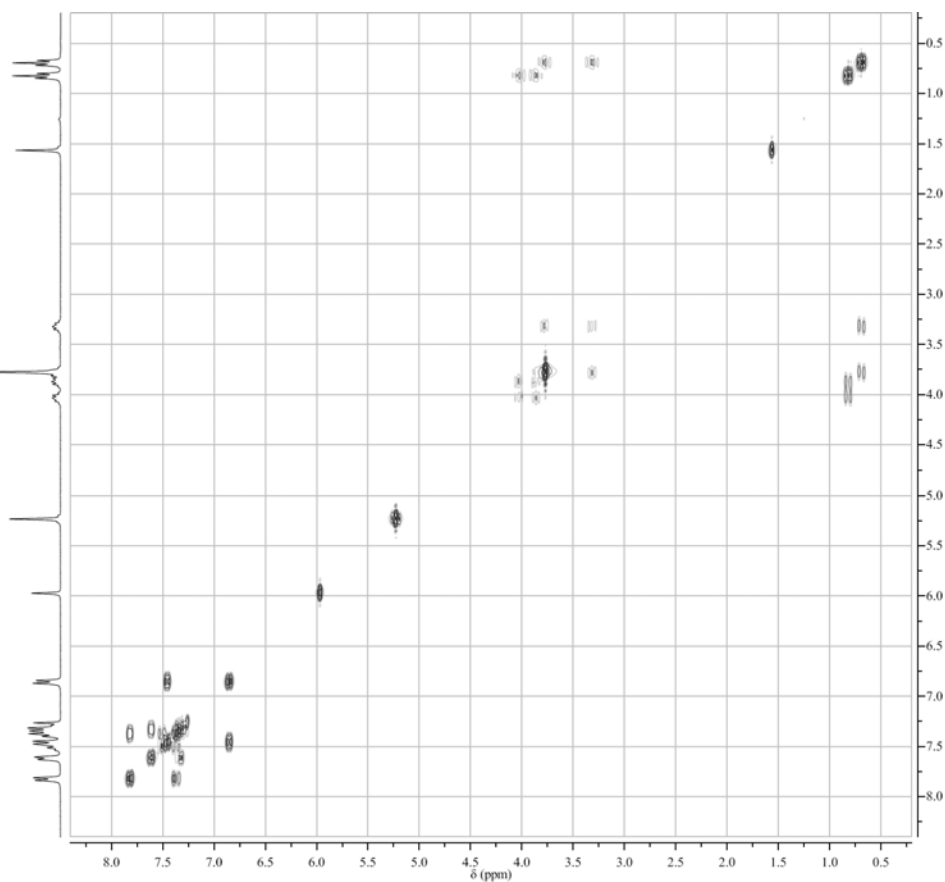
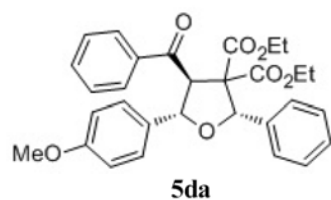


2D NOESY NMR
300 MHz, CDCl₃

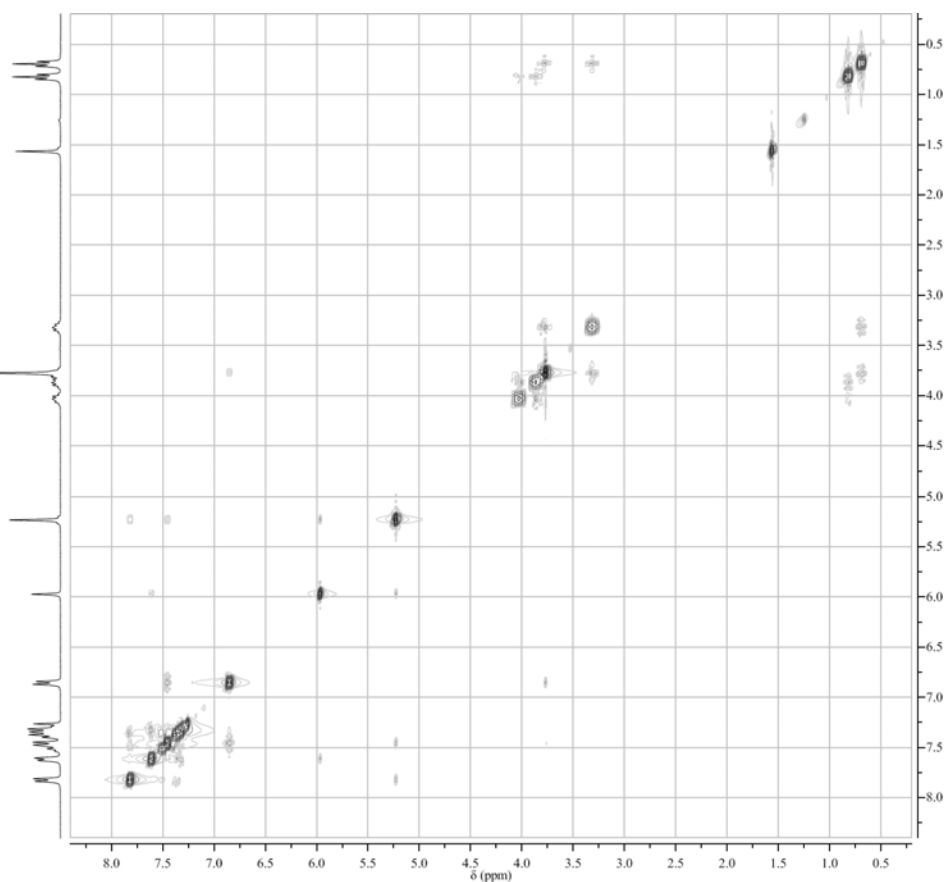
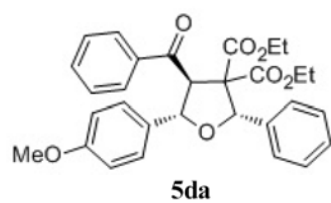




2D COSY NMR
300 MHz, CDCl₃

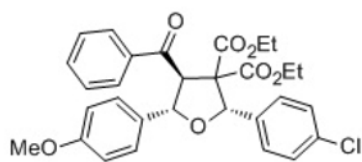


2D NOESY NMR
300 MHz, CDCl₃

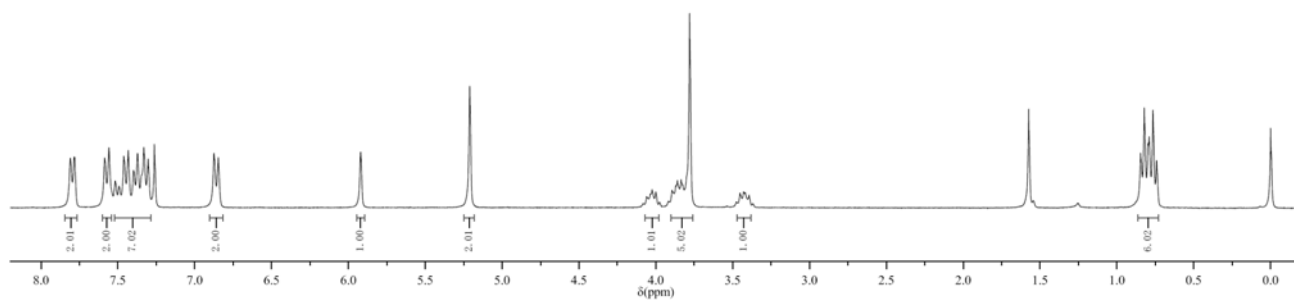




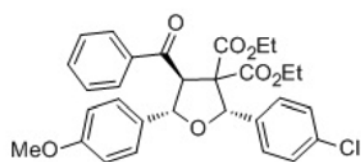
^1H NMR
300 MHz, CDCl_3



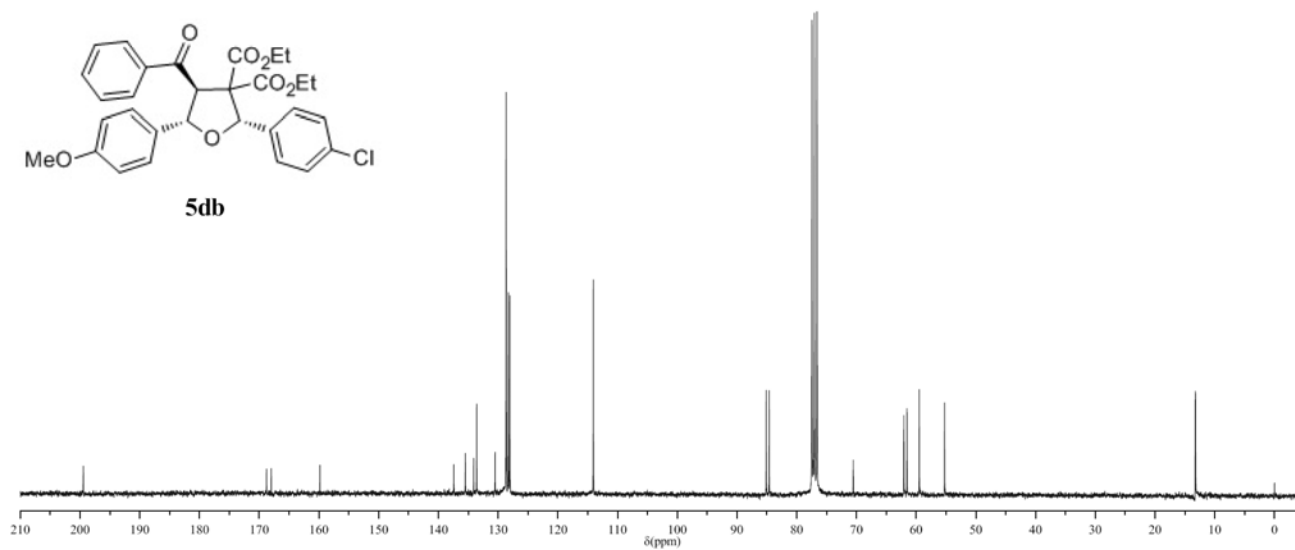
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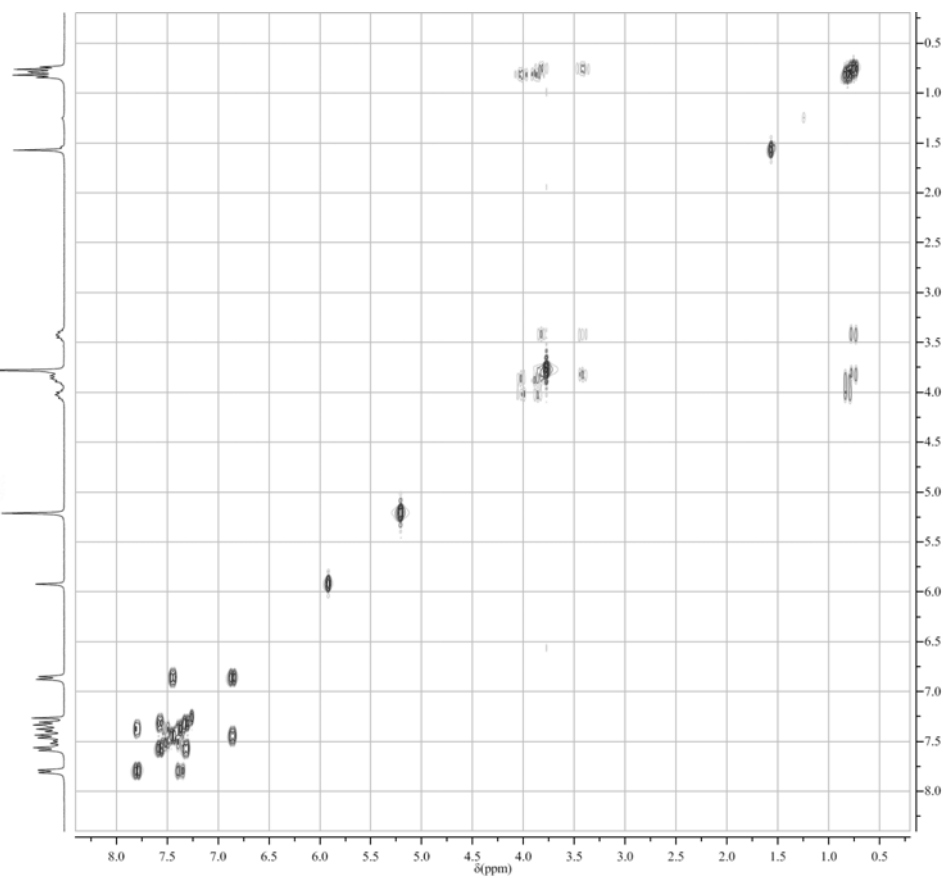
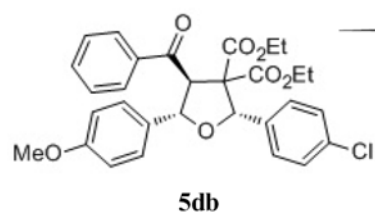
^{13}C NMR
75 MHz, CDCl_3



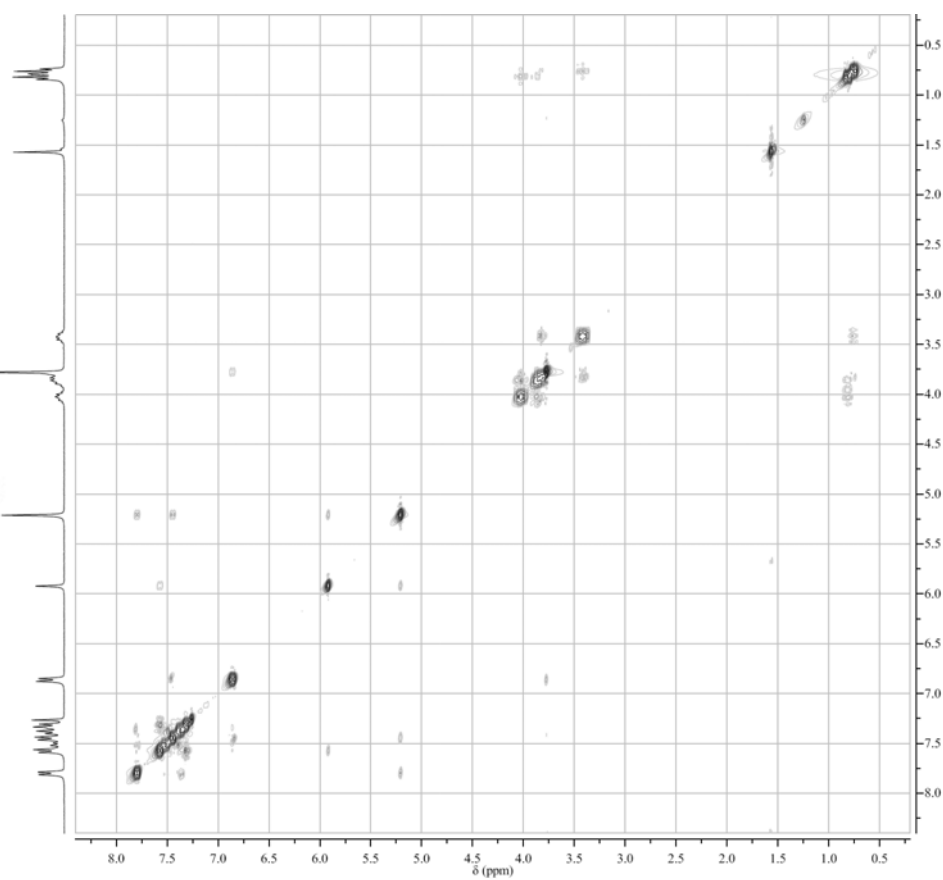
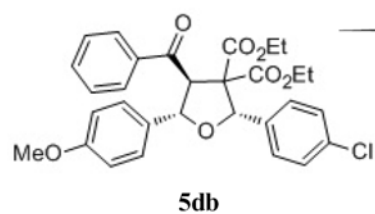
5db



2D COSY NMR
300 MHz, CDCl₃

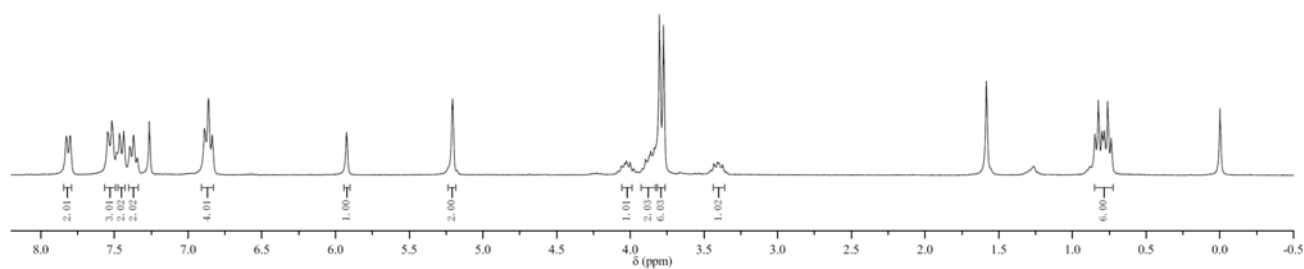
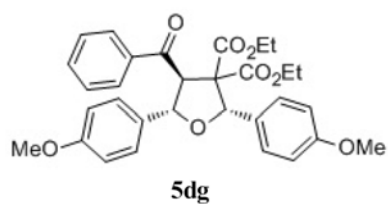


2D NOESY NMR
300 MHz, CDCl₃

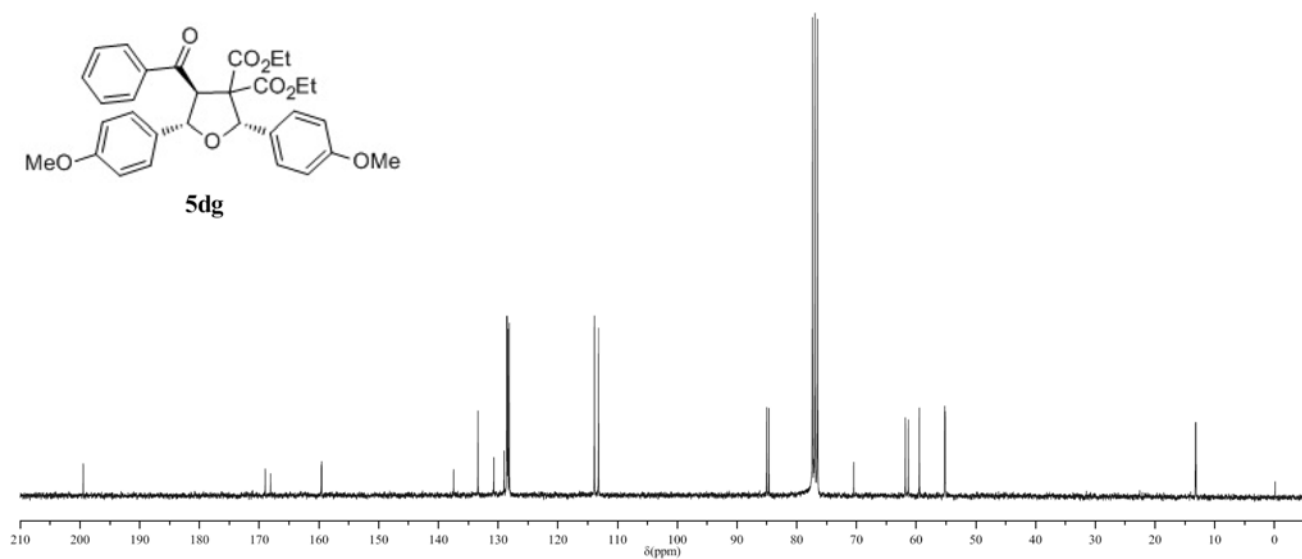
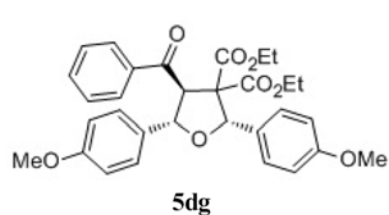




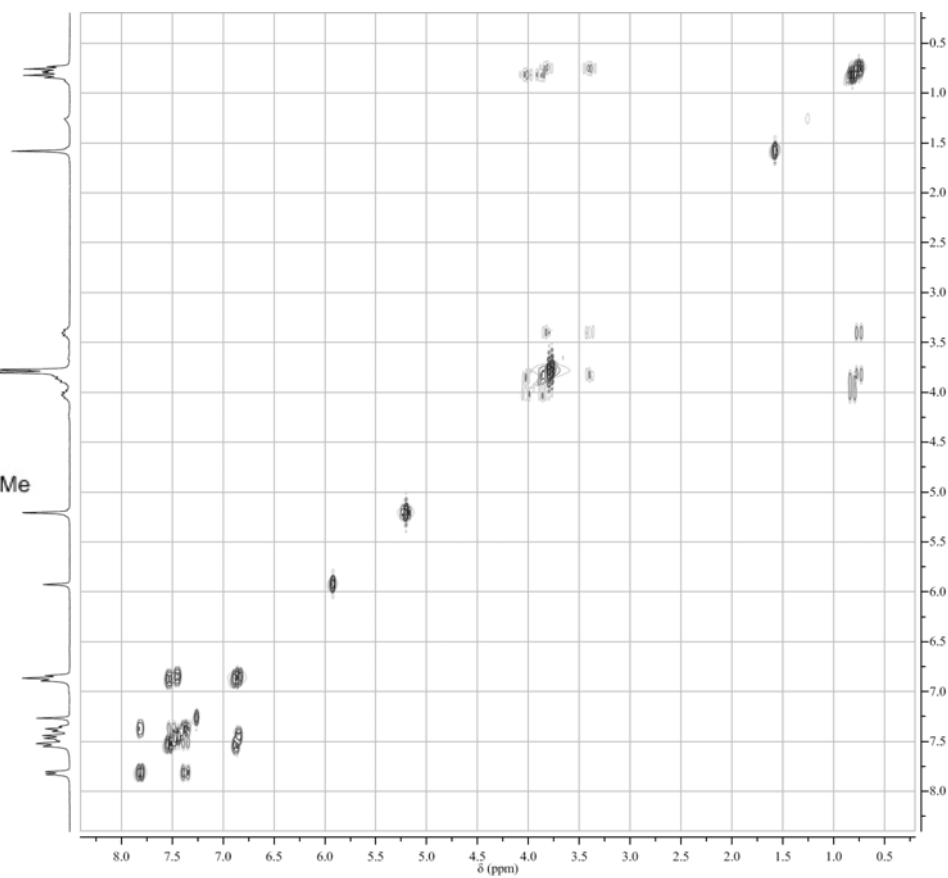
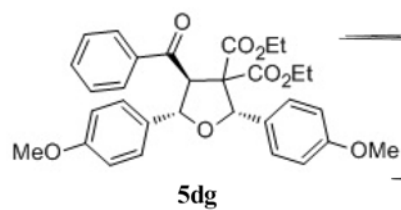
¹H NMR
300 MHz, CDCl₃



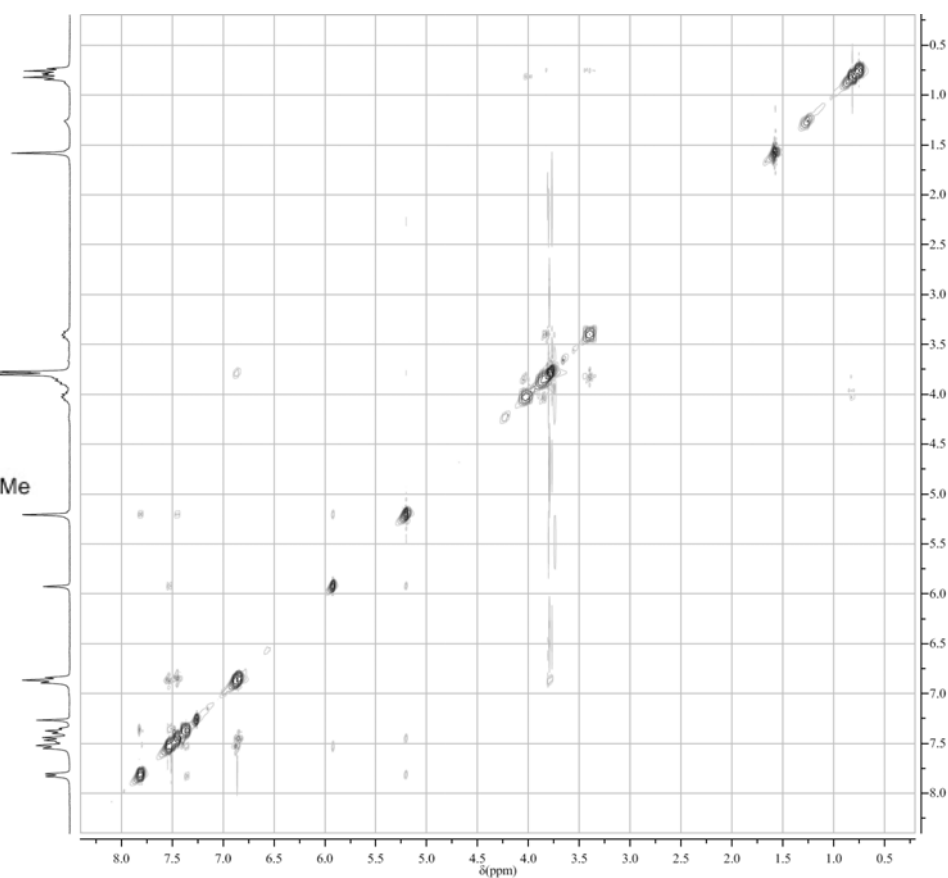
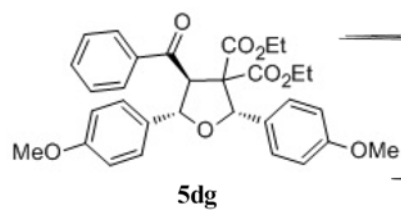
¹³C NMR
75 MHz, CDCl₃

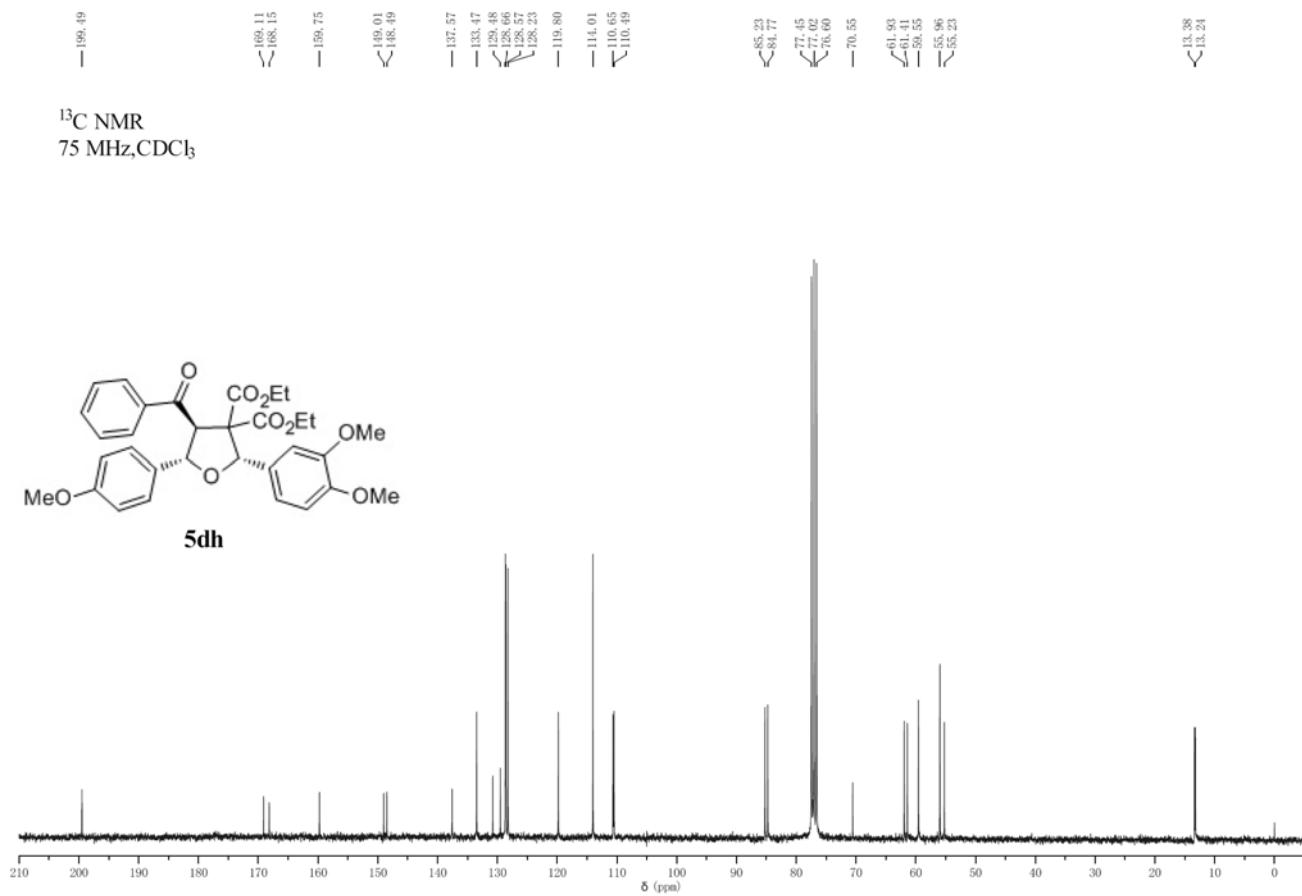
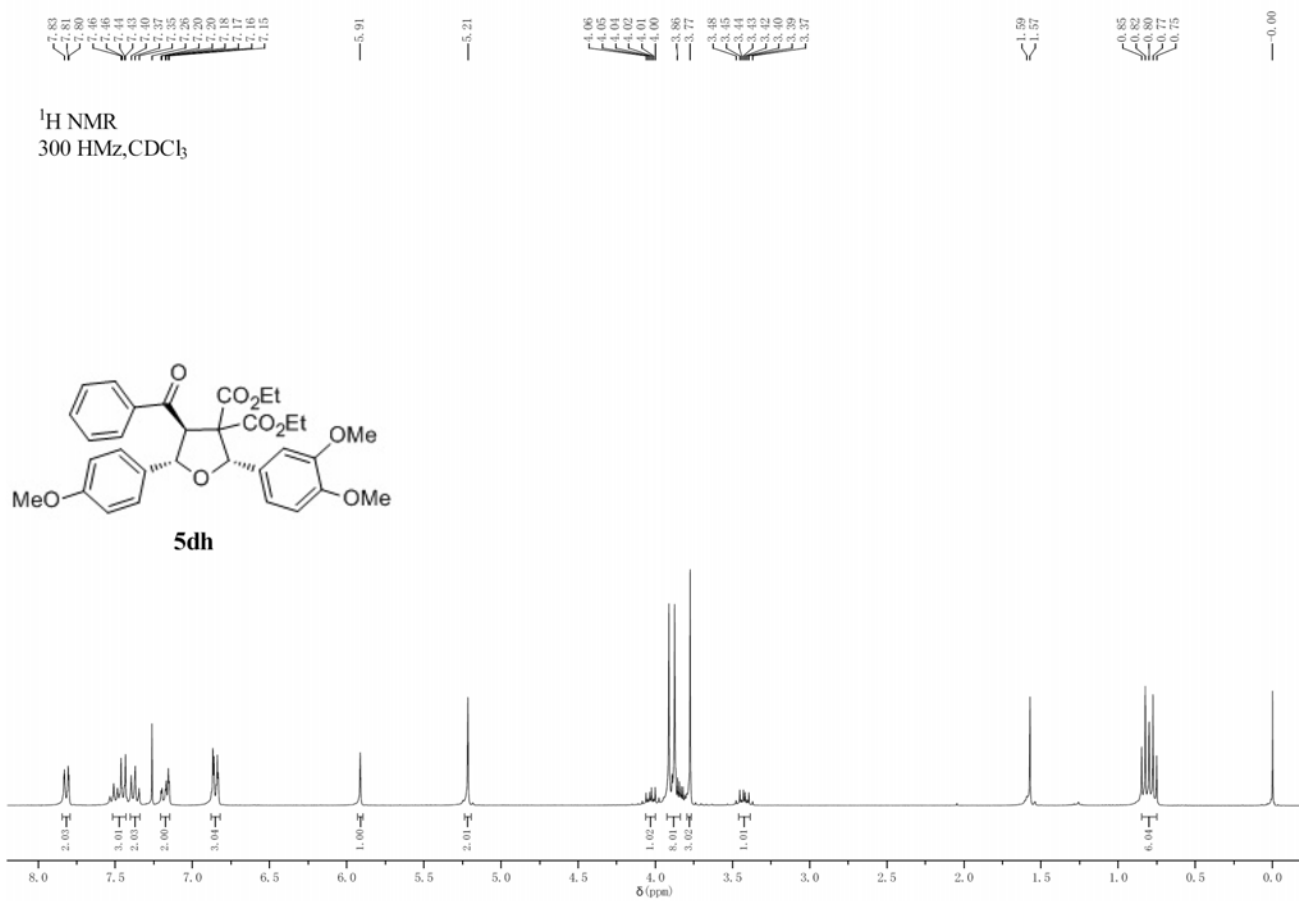


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300 MHz, CDCl₃

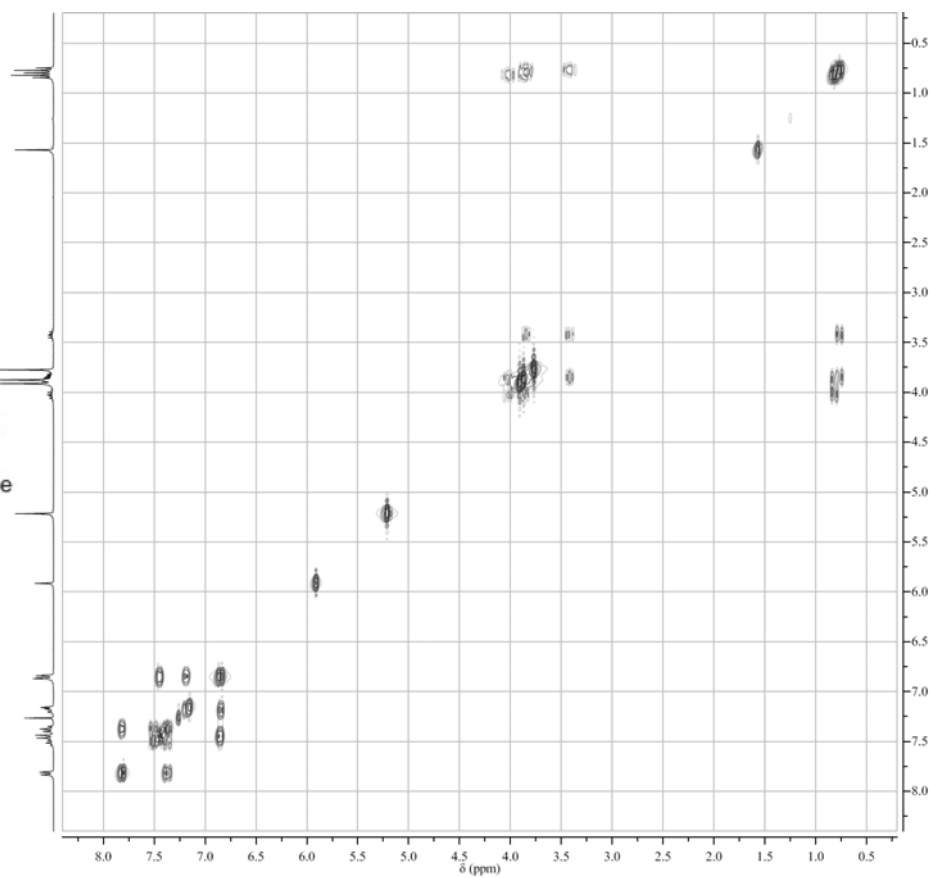
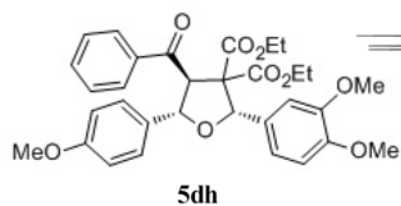


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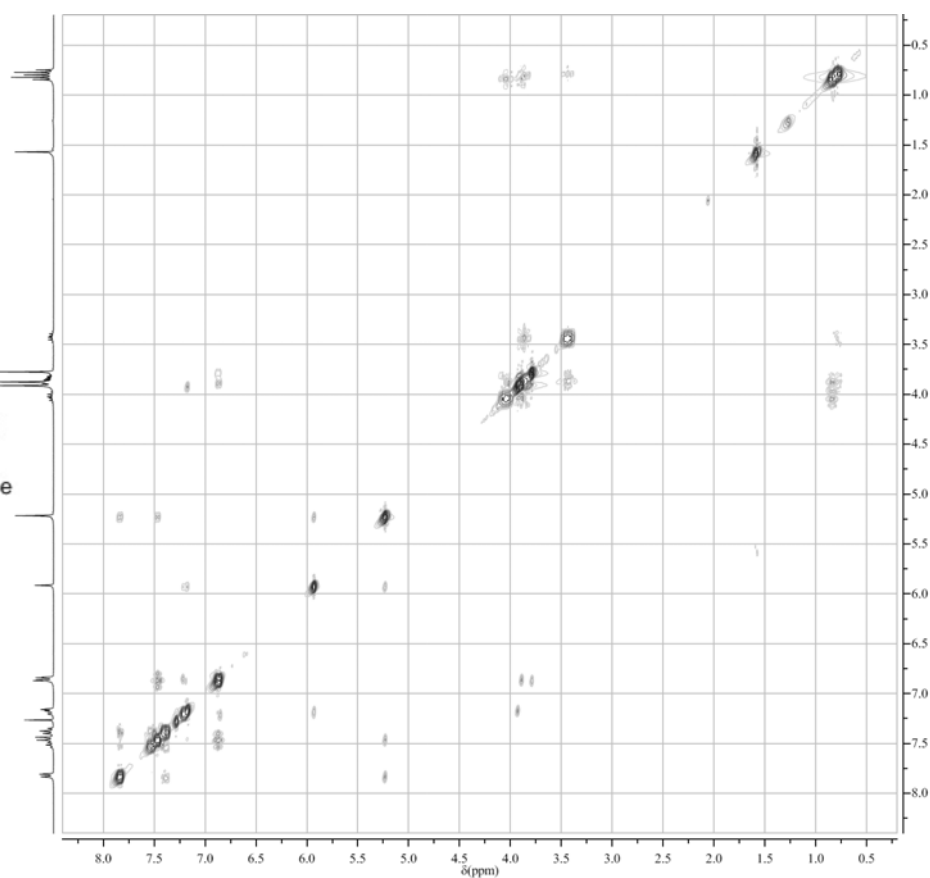
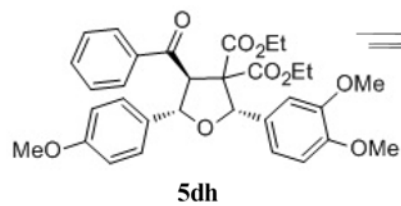




2D COSY NMR
300 MHz, CDCl₃



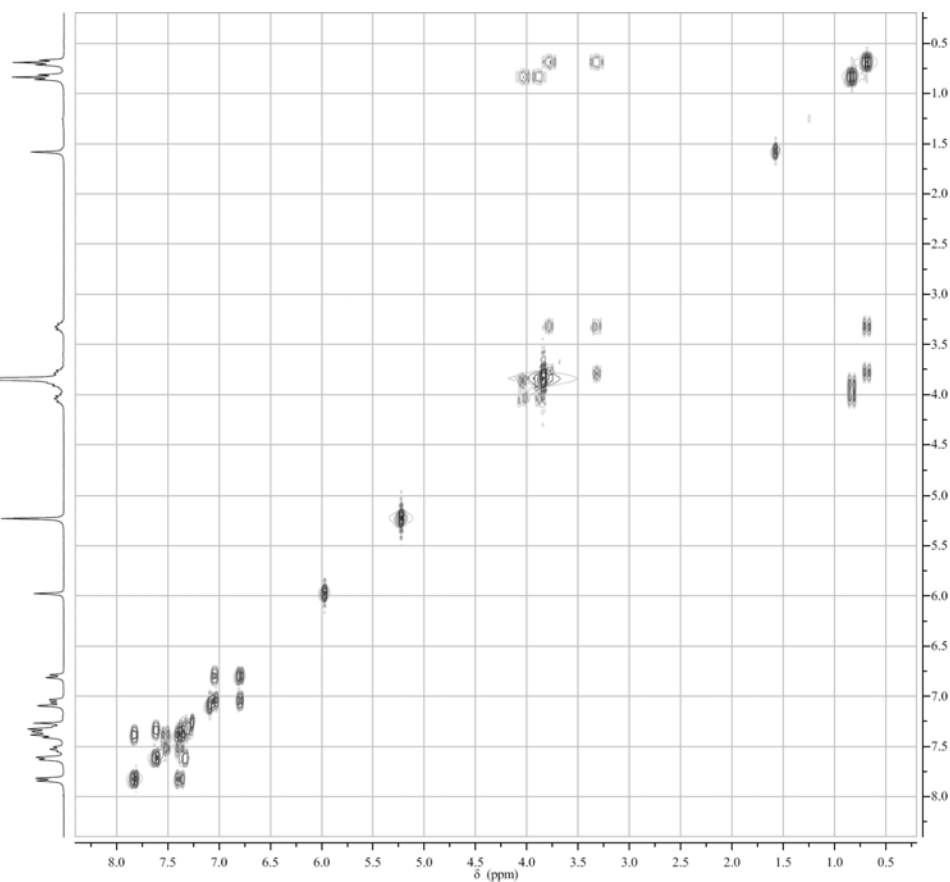
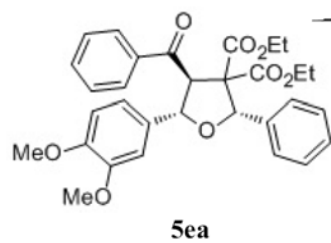
2D NOESY NMR
300 MHz, CDCl₃



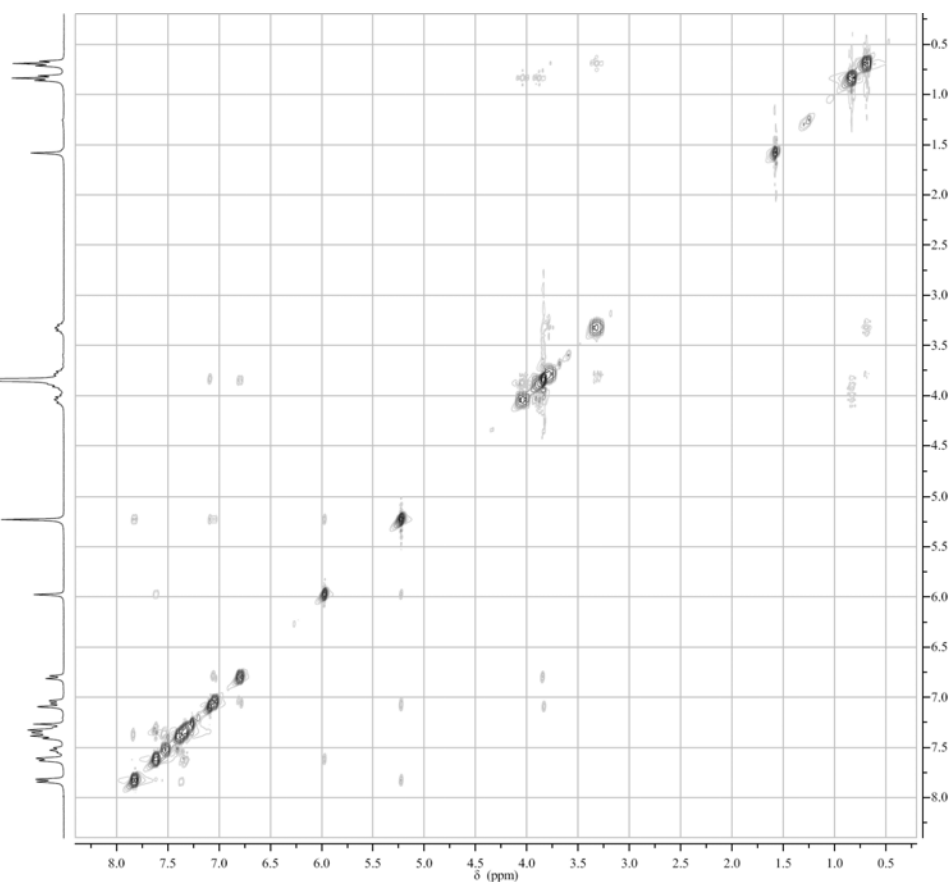
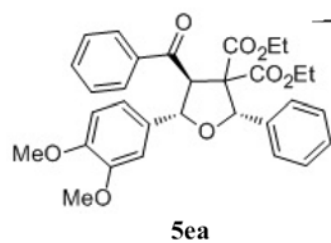
-0.00

-13.27

2D COSY NMR
300 MHz, CDCl₃



2D NOESY NMR
300 MHz, CDCl₃



7.81
7.79
7.51
7.51
7.36
7.32
7.27
7.06
7.05
6.82
6.79

5.92

5.24
5.21
5.20
5.17

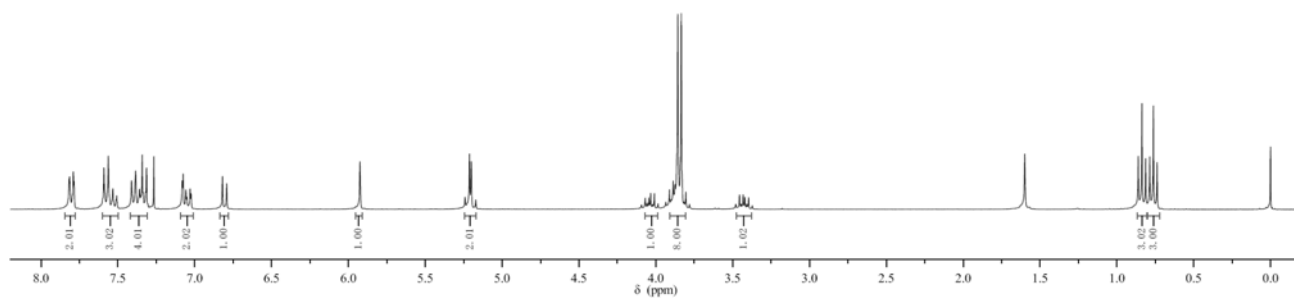
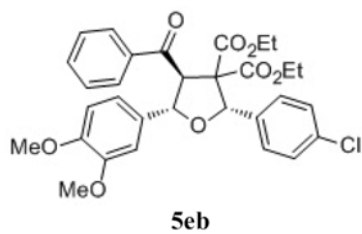
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3.91
3.90
3.89
3.87
3.86
3.83
3.82
3.80
3.78
3.48
3.46
3.44
3.43
3.42
3.41
3.40
3.37

1.60

0.86
0.84
0.81
0.79
0.74

0.00

^1H NMR
300 MHz, CDCl_3



199.57

168.72
167.95

149.16
149.04

137.38
136.10
134.10
133.66
130.92
128.65
128.05

119.32

110.97
109.87

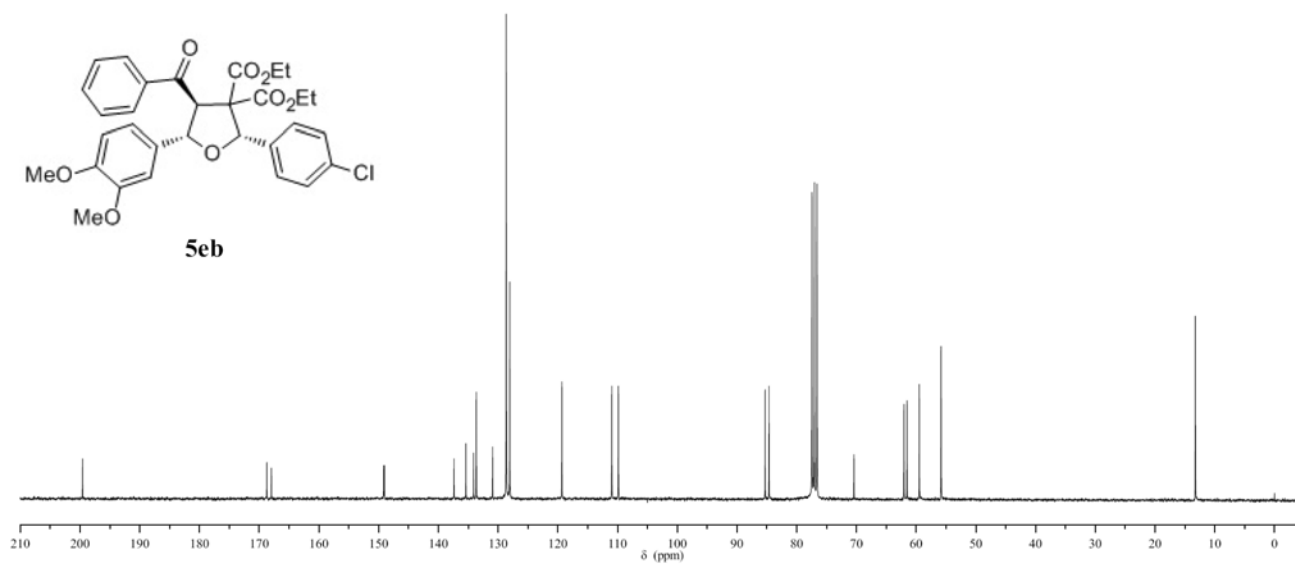
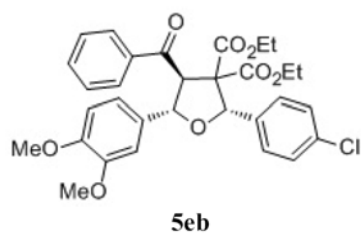
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84.04
77.45
77.22
77.02
76.60
70.41

62.04
61.53
59.47

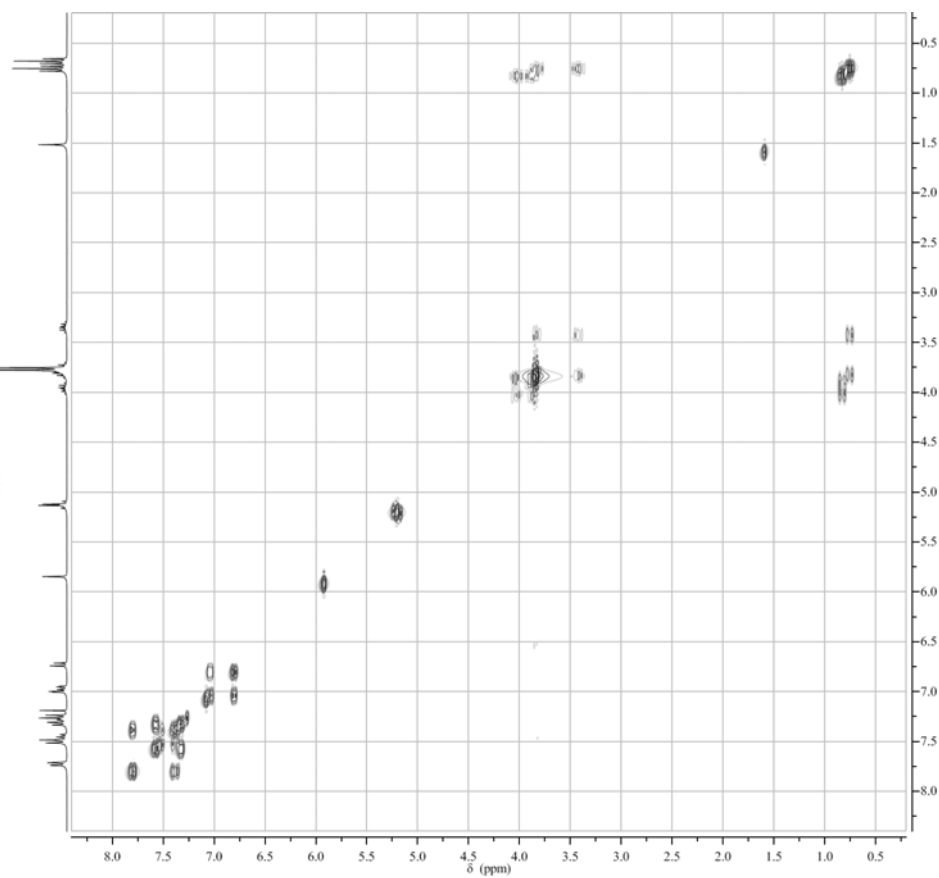
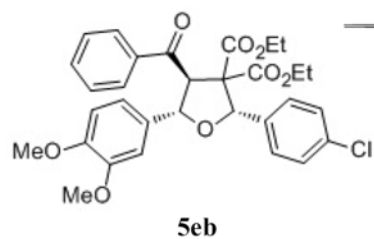
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13.26

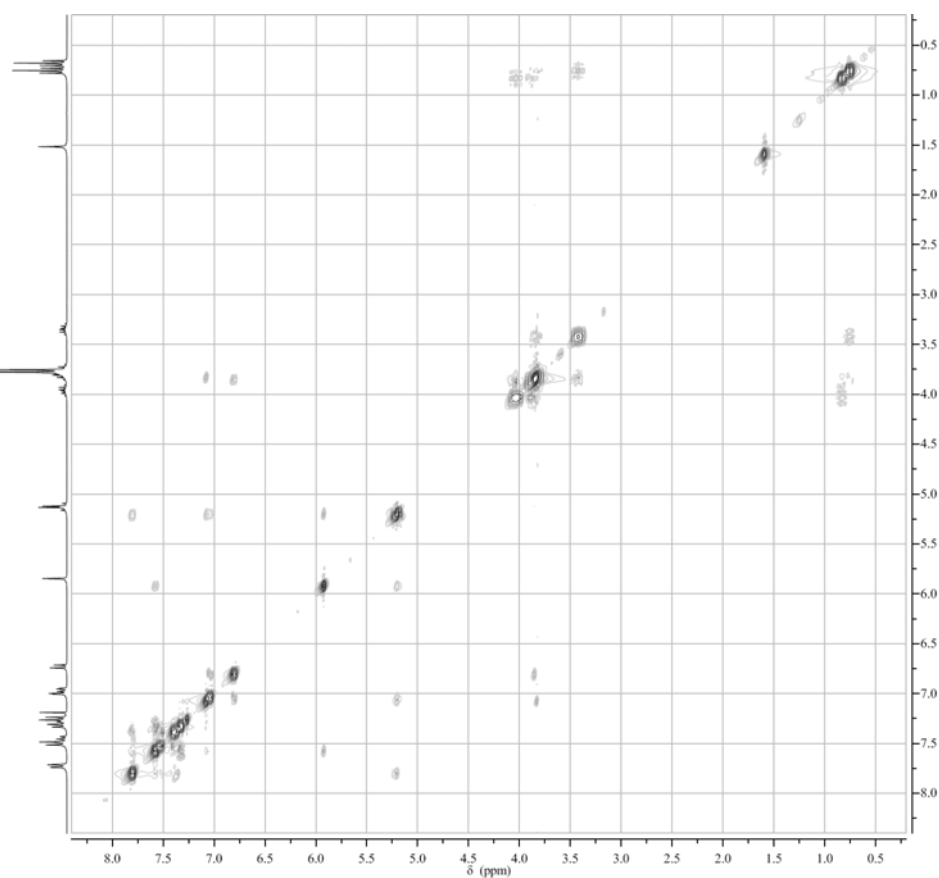
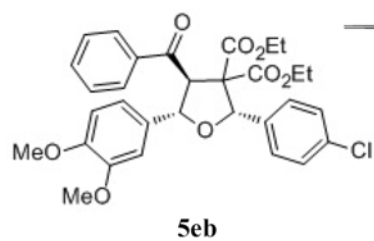
^{13}C NMR
75 MHz, CDCl_3



2D COSY NMR
300 MHz, CDCl₃



2D NOESY NMR
300 MHz, CDCl₃



7.82
7.80
7.55
7.52
7.36
7.35
7.02
6.90
6.87
6.81
6.78

5.93

5.20

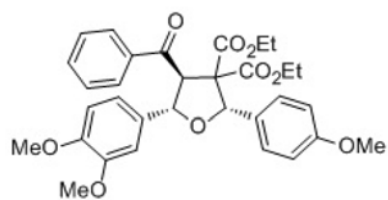
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3.44
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1.60
1.58

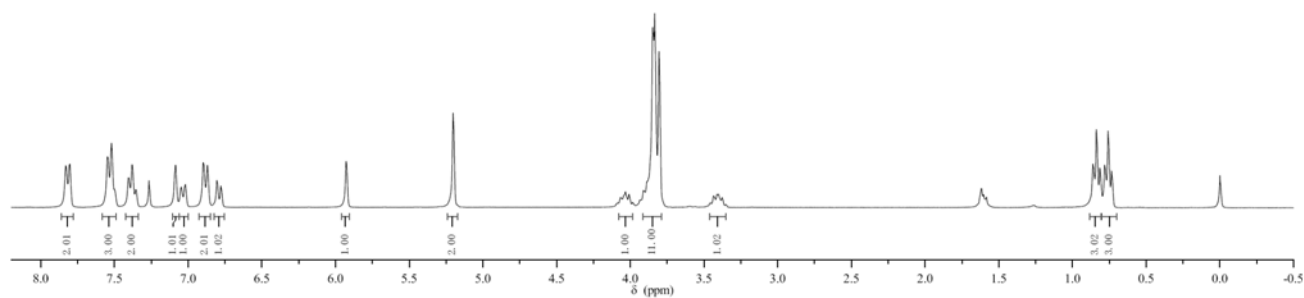
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0.84
0.81
0.78
0.75
0.73

0.00

¹H NMR
300 MHz, CDCl₃



5eg



199.69

168.04
168.16

159.64

149.01
148.97

137.52

133.54
131.23

128.65
128.60
128.41

119.24

113.29
100.91
100.80

85.16
84.93

77.45
77.03
76.61

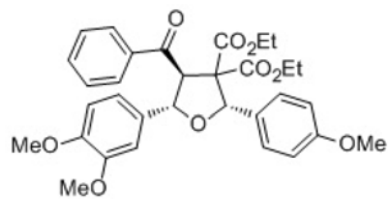
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61.90
61.40
59.54

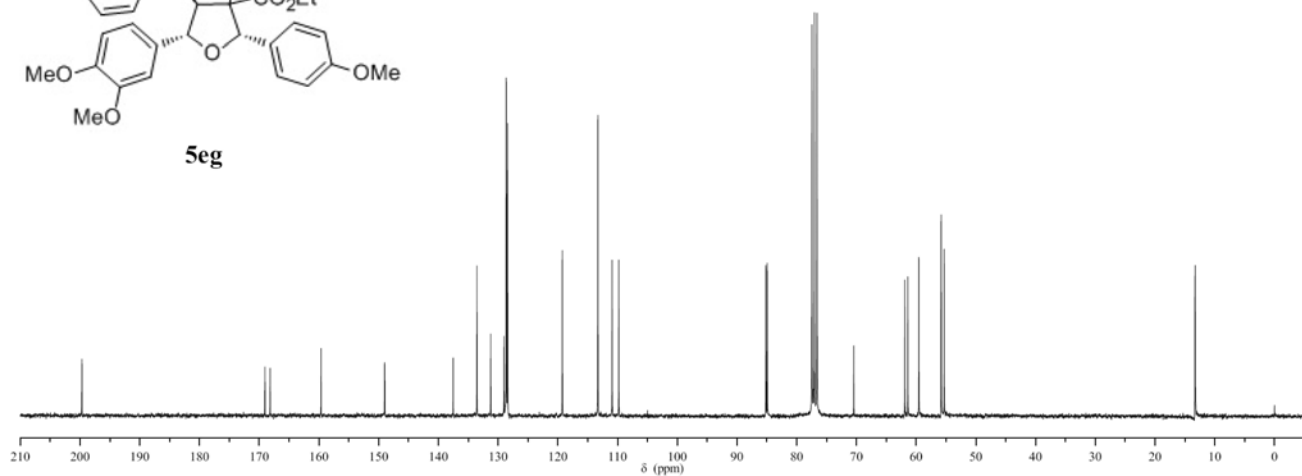
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55.80
55.31

13.33
13.27

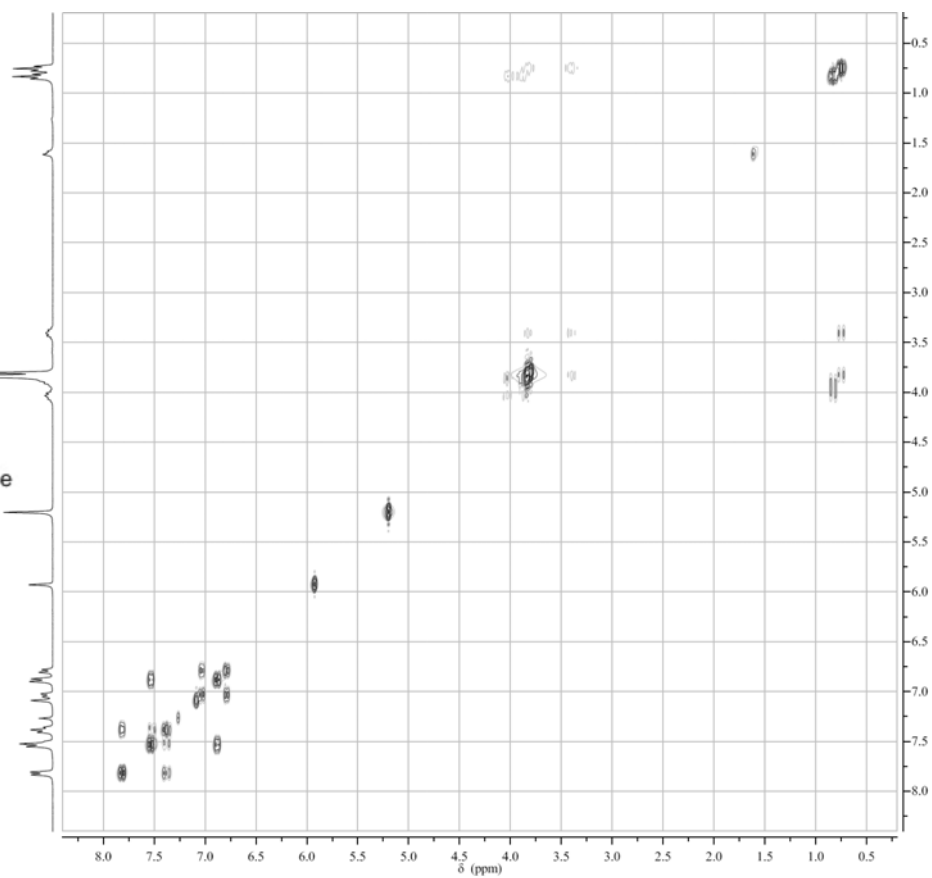
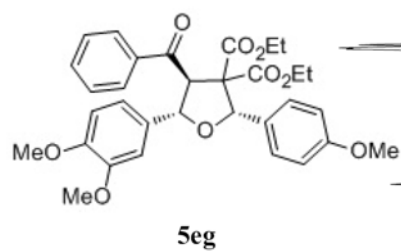
¹³C NMR
75 MHz, CDCl₃



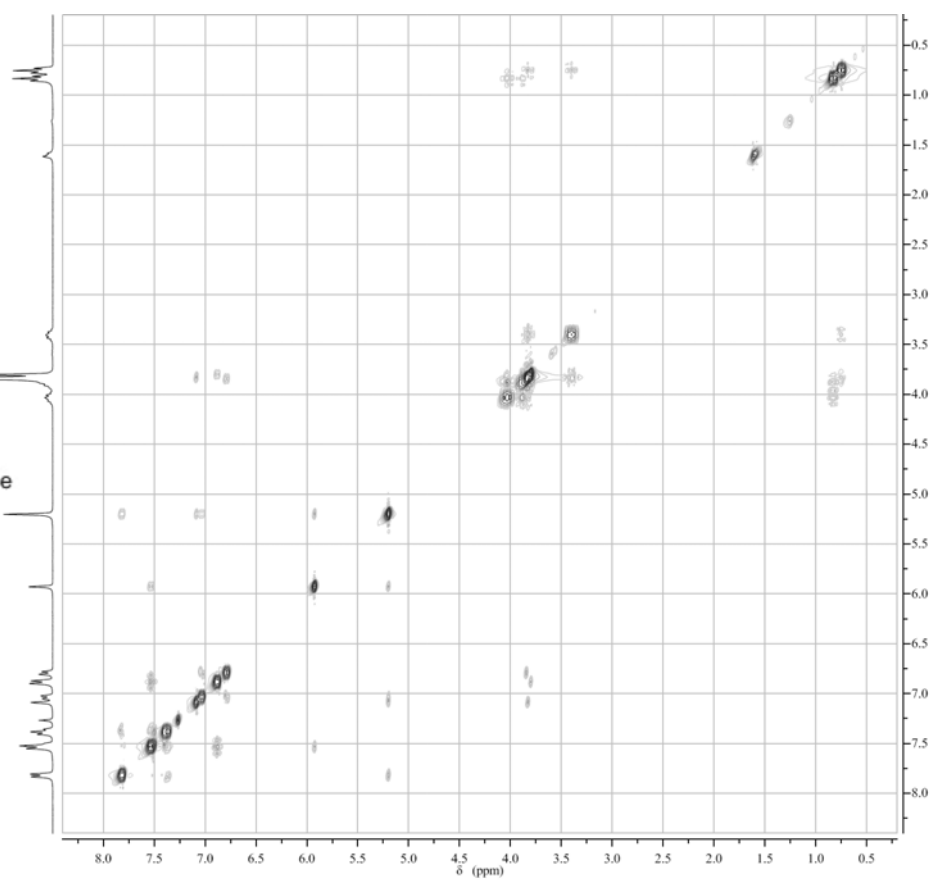
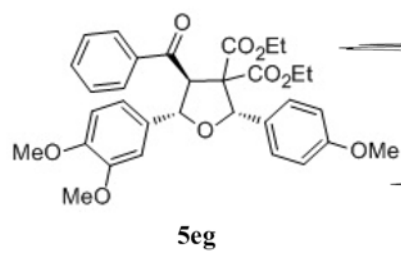
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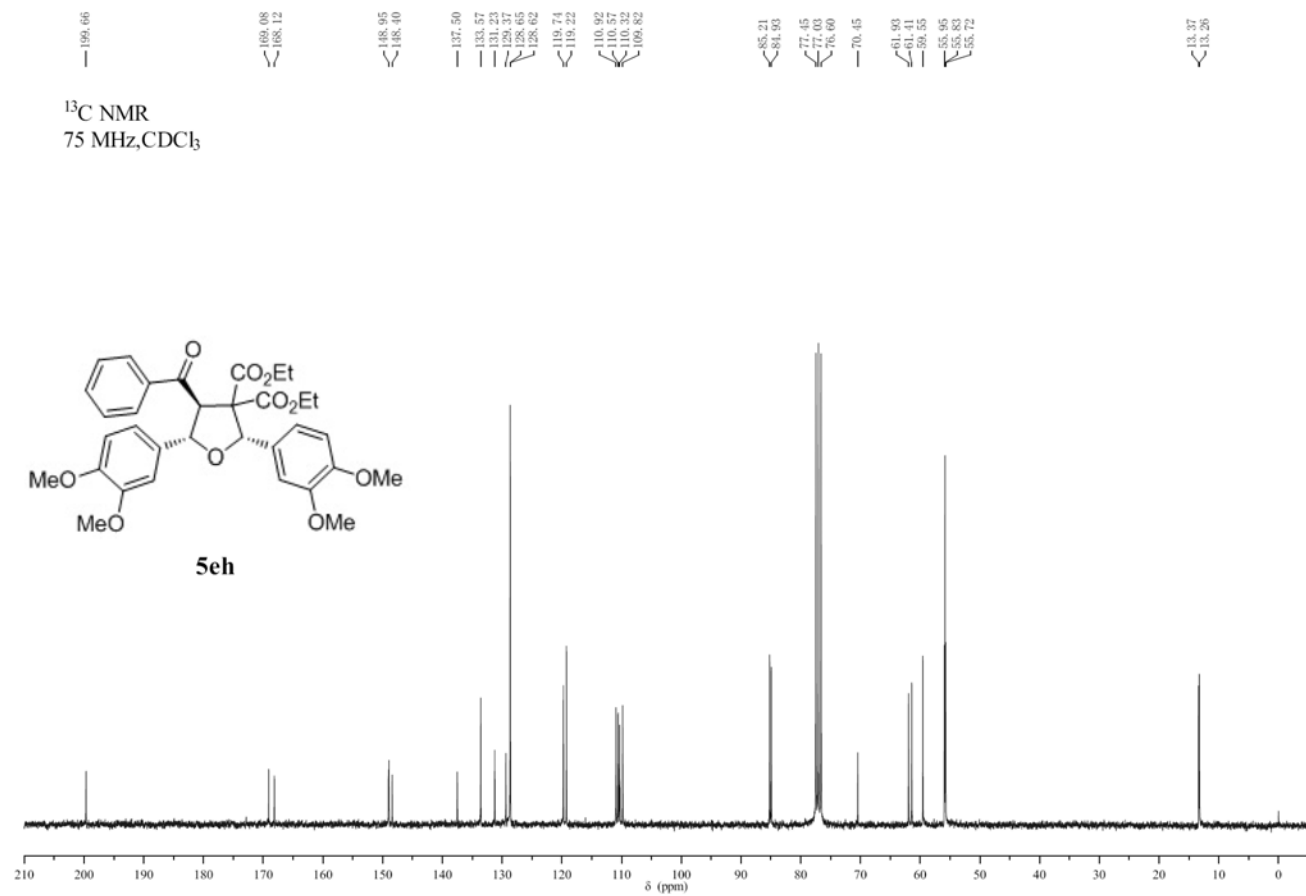
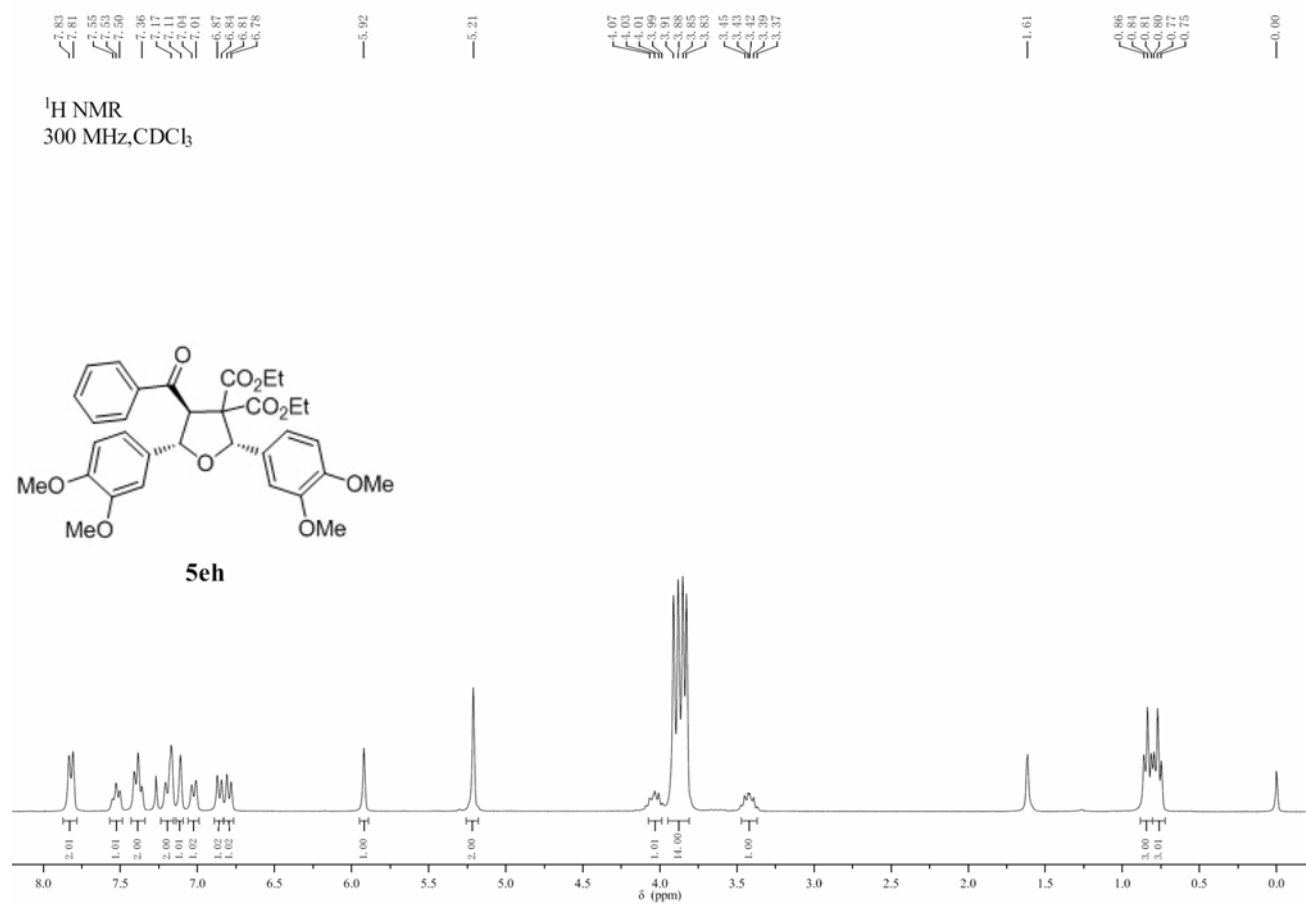


2D COSY NMR
300 MHz, CDCl₃

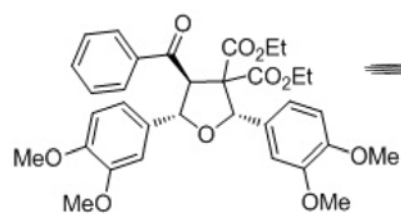


2D NOESY NMR
300 MHz, CDCl₃

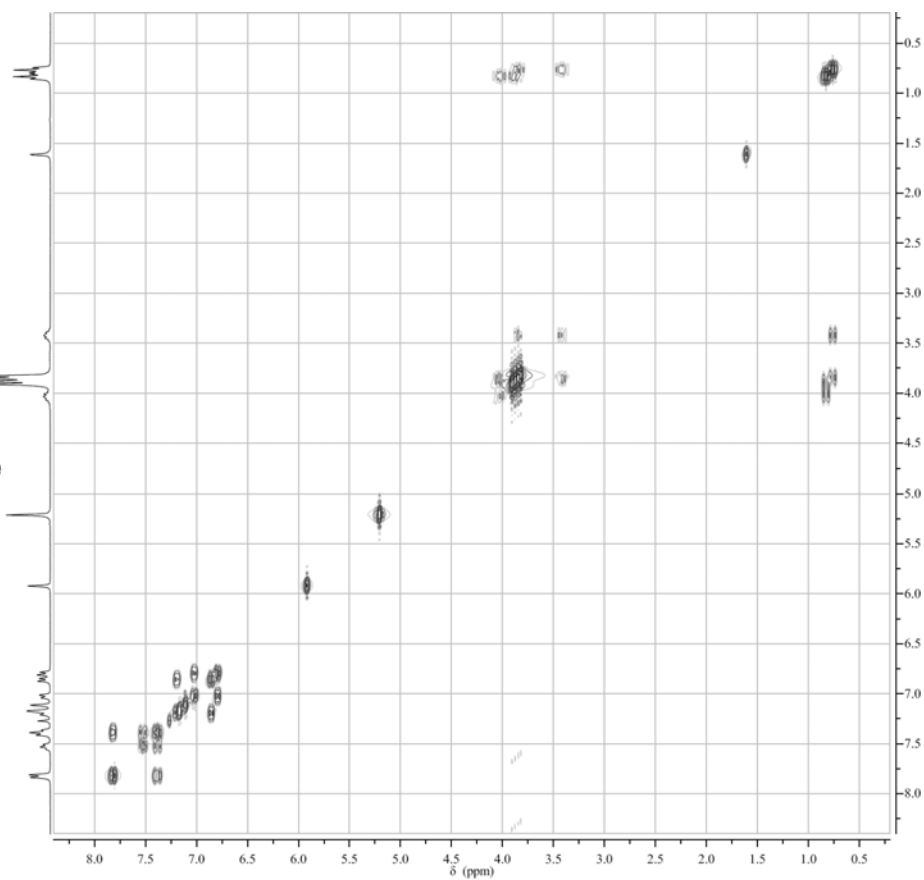




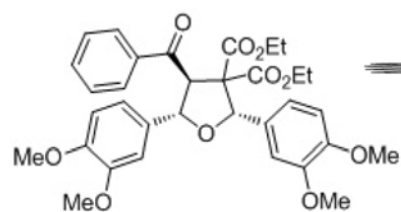
2D COSY NMR
300 MHz, CDCl₃



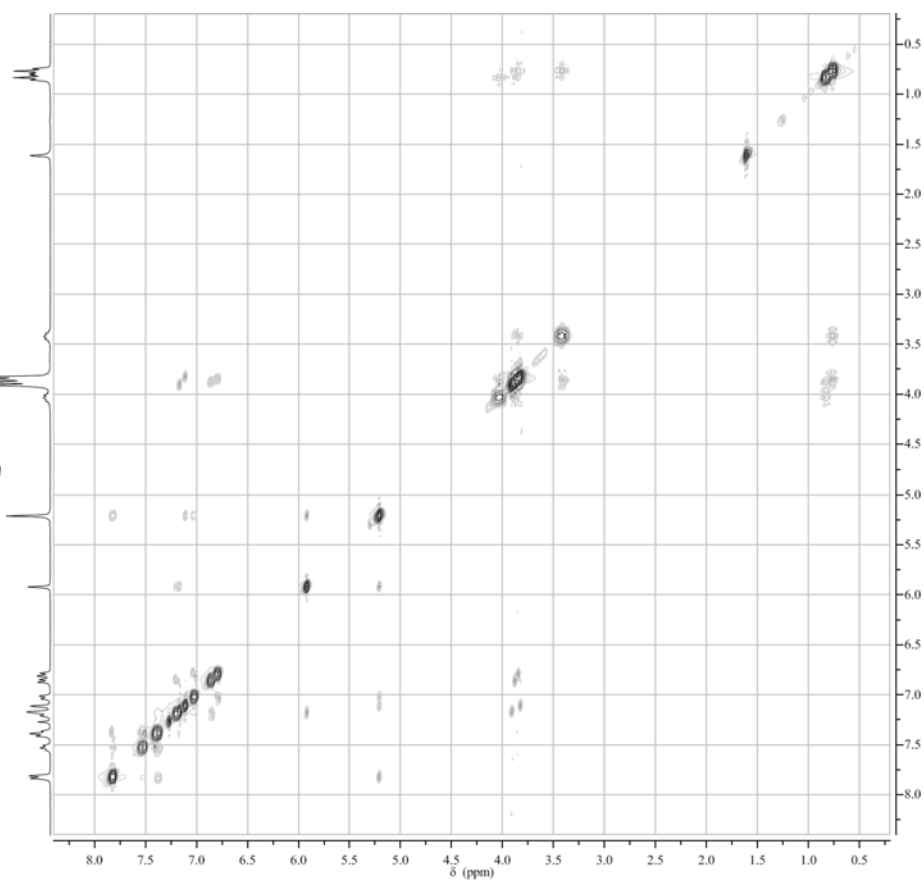
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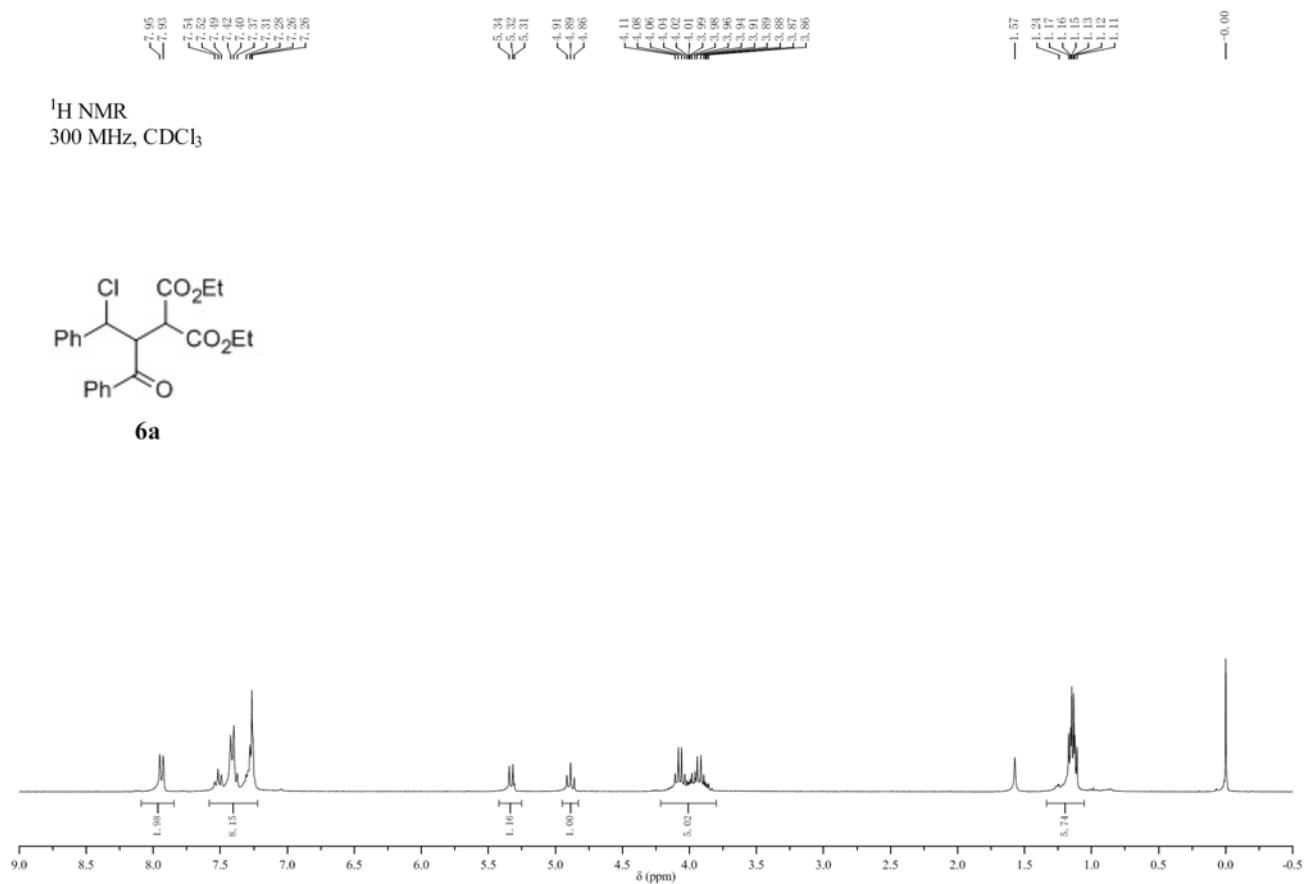
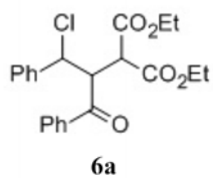
2D NOESY NMR
300 MHz, CDCl₃



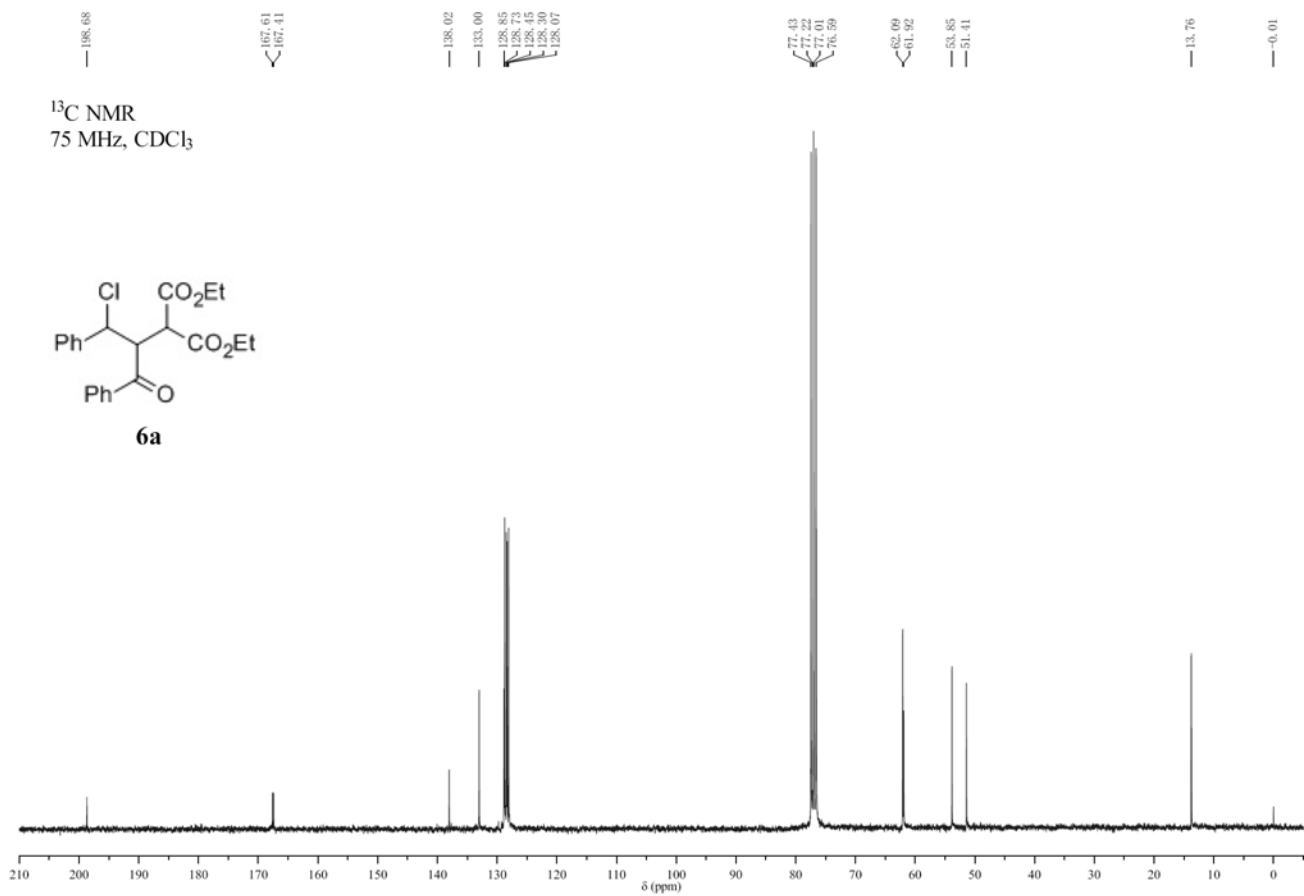
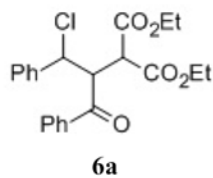
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¹H NMR
300 MHz, CDCl₃

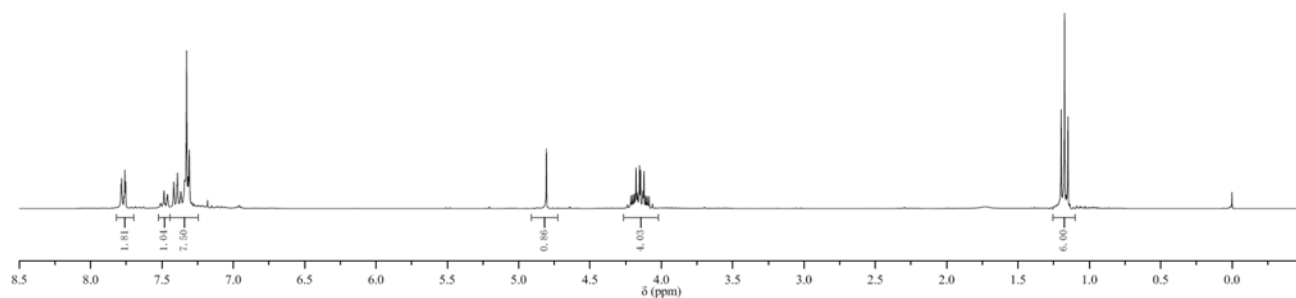
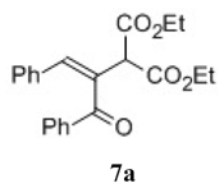


¹³C NMR
75 MHz, CDCl₃

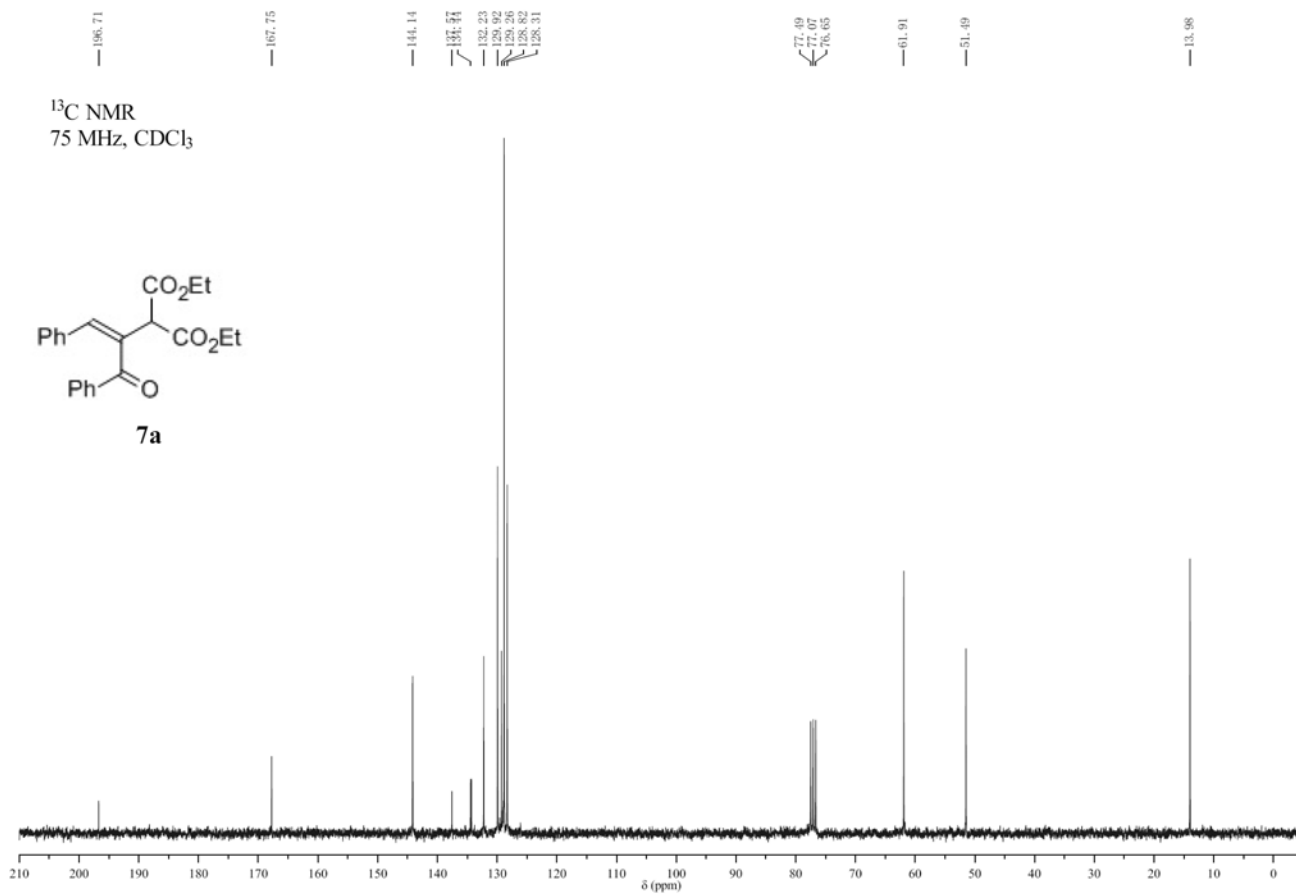
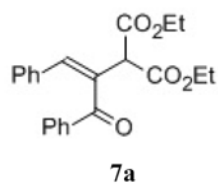




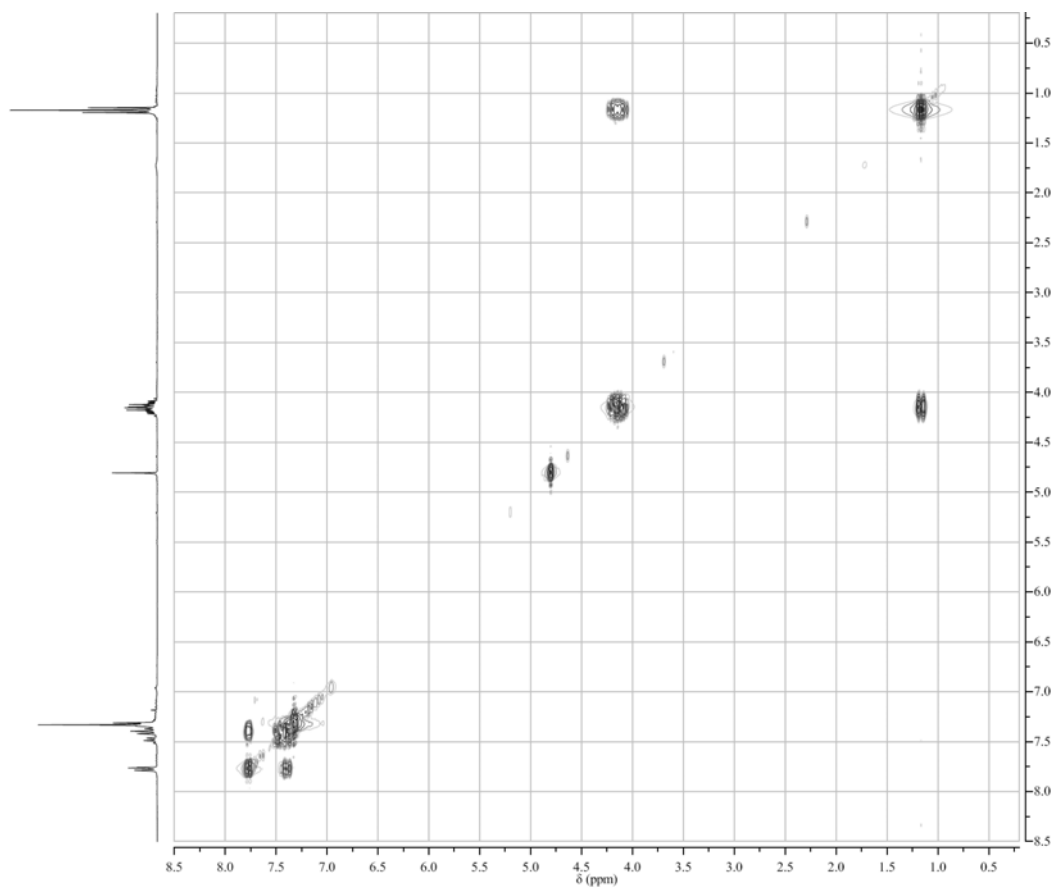
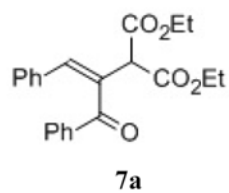
^1H NMR
300 MHz, CDCl_3



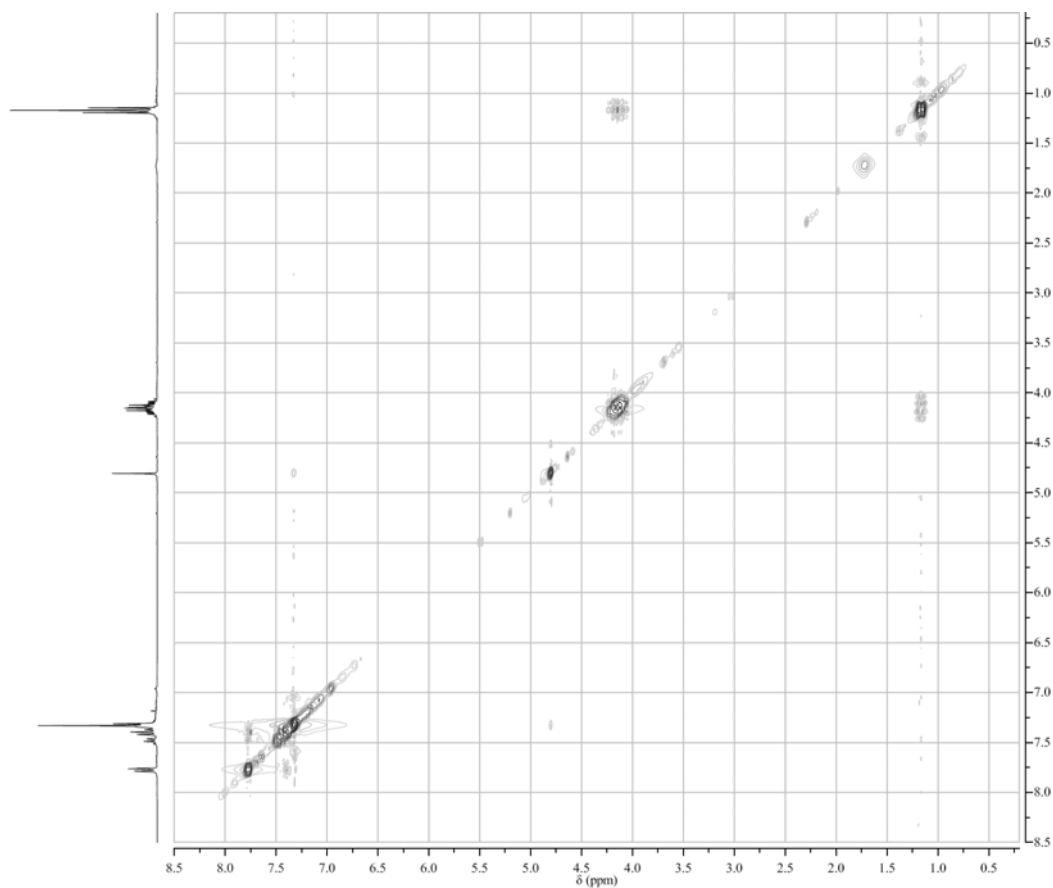
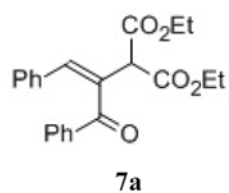
^{13}C NMR
75 MHz, CDCl_3



2D COSY NMR
300 MHz, CDCl₃



2D NOESY NMR
300 MHz, CDCl₃



X-ray crystallographic analysis of **5ac**

The single crystal of **5ac** suitable for X-ray crystallographic analysis was obtained by recrystallization from a mixed solvent of dichloromethane and petroleum ether. The single crystal X-ray diffraction data for the **5ac** was collected on a diffractometer with graphite monochromated Mo K α radiation ($\lambda = 0.71073$ Å) at room temperature. Saint program and SADABS program carried out the data integration. The structure was solved by a direct method and refined on F² using SHELXTL suite of program. All non-hydrogen atoms were anisotropically refined by full-matrix least squares methods. All hydrogen atoms were geometrically generated and isotropically refined using a riding model. The details of the X-ray data collection, structure solution and structure refinements are given in Table S1. The crystal structure of **5ac** is given in Figure S1.

Table S1 Crystallographic data and structural refinement details for compound **5ac**.

Compound	5ac
Formula	C ₂₉ H ₂₆ O ₆ Cl ₂
Formula weight	541.40
Temperature/K	293(2)
Crystal system	Triclinic
Space group	P-1
<i>a</i> , Å	8.330(1)
<i>b</i> , Å	11.589(2)
<i>c</i> , Å	15.560(2)
α , deg	69.6(2)
β , deg	87.9(2)
γ , deg	77.0 (2)
<i>V</i> / Å ³	1370.2(3)
<i>Z</i>	2
μ (Mo Ka), mm ⁻¹	0.277
θ range for data collection, deg	1.9 to 27.6
Calculated density/(g·cm ⁻³)	1.312
Reflections collected	11796
Unique reflections/R _{int}	6154 / R(int) = 0.025
<i>F</i> (000)	564
Goodness-of-fit on F ²	1.04
Crystal dimension/mm ³	0.20×0.25×0.30
R, R _w [<i>I</i> > 2 σ (<i>I</i>)]	0.0601, 0.2130
Residual ρ /(e·Å ⁻³)	0.390, −0.310

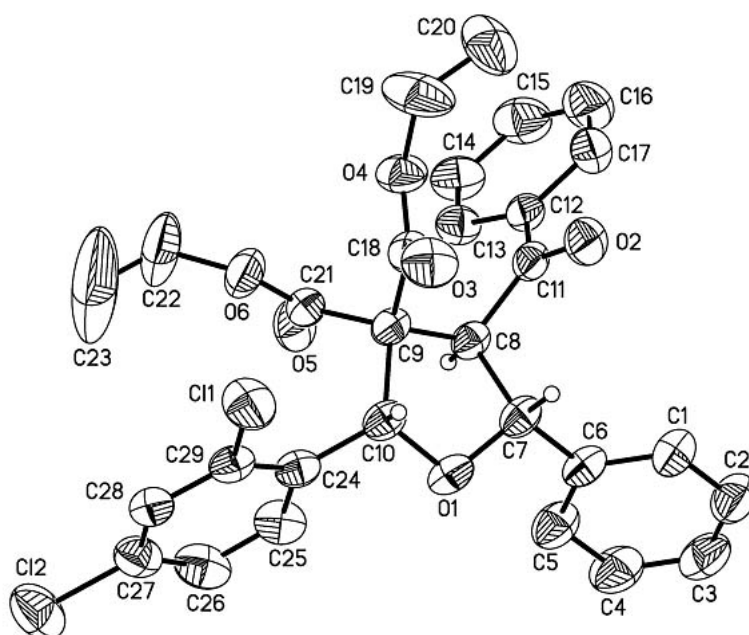


Figure S1 X-ray structure (50 % probability ellipsoids) of **5ac**.