

Phosphine-Catalyzed Dearomative (3+2) Annulation of 2-Nitrobenzofurans and Nitrobenzothiophenes with Allenolate

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^c Chengdu Institute of Biology, Chinese Academy of Sciences, Chengdu 610041, China

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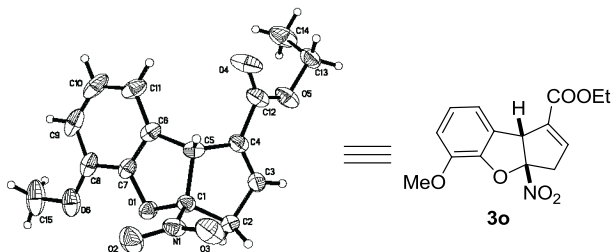
*E-mail: yuanwc@cioc.ac.cn

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1. X-ray crystal structure of compound **3o**



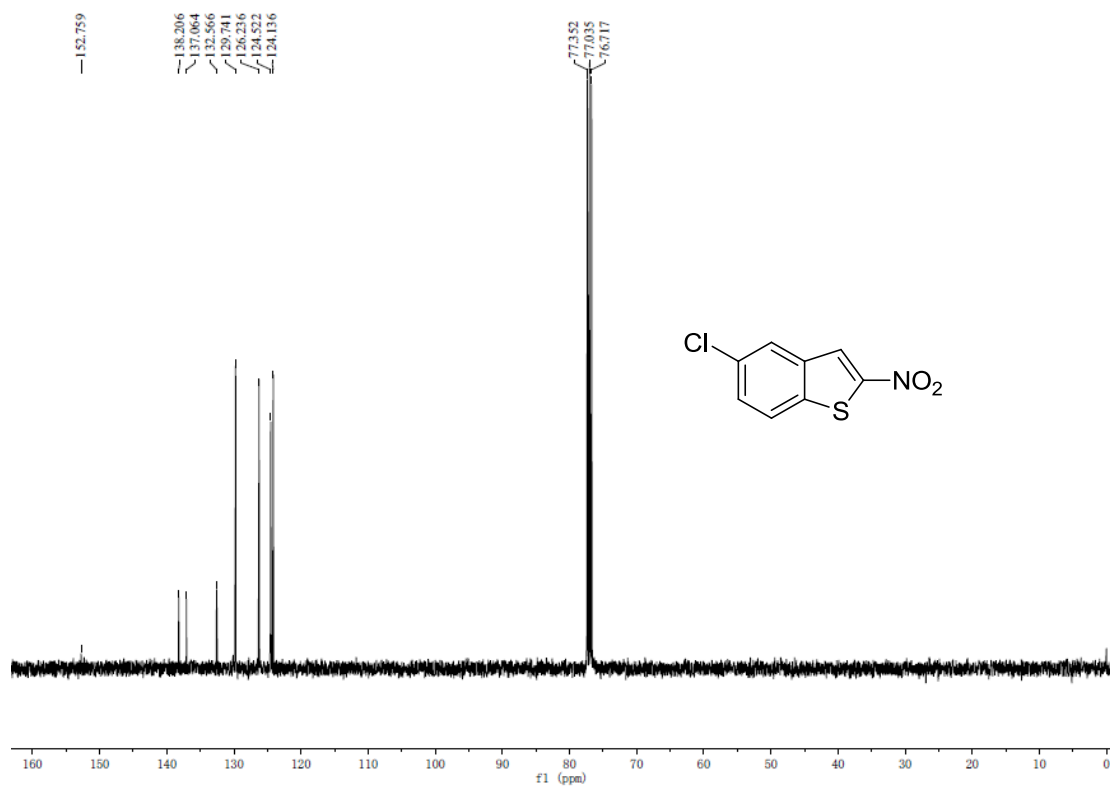
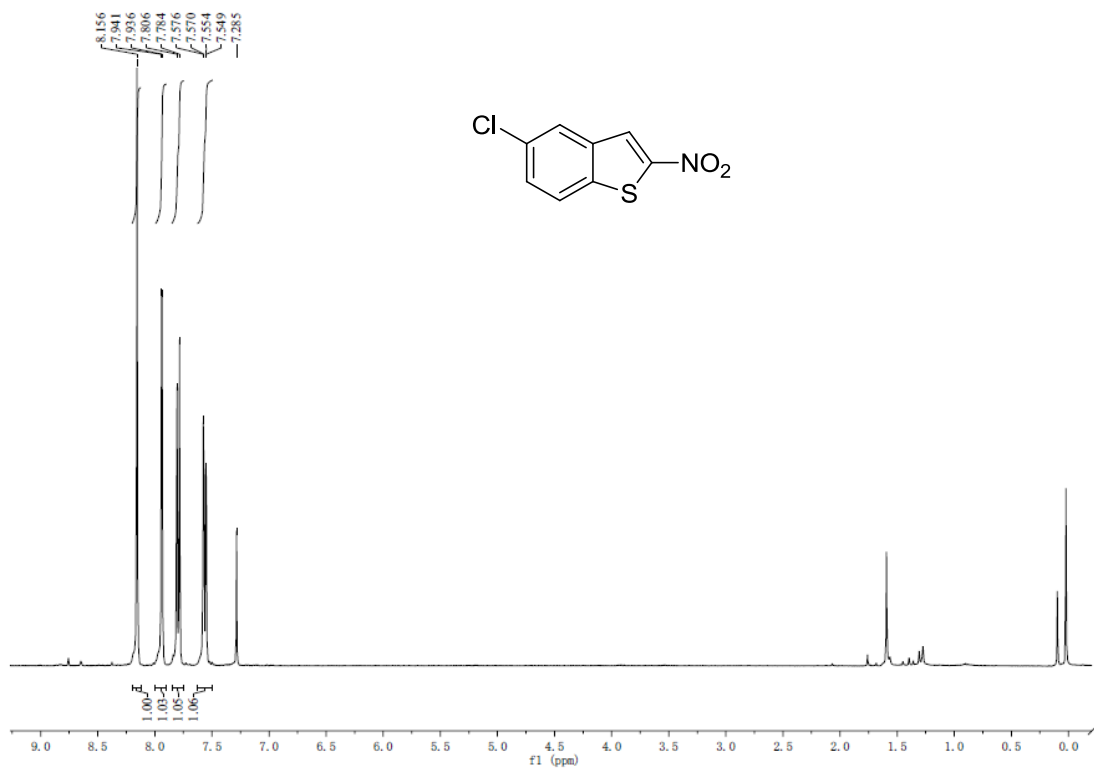
ORTEP of **3o** (at 50% level)

Crystal data and structure refinement for **3o** (CCDC-1900800)

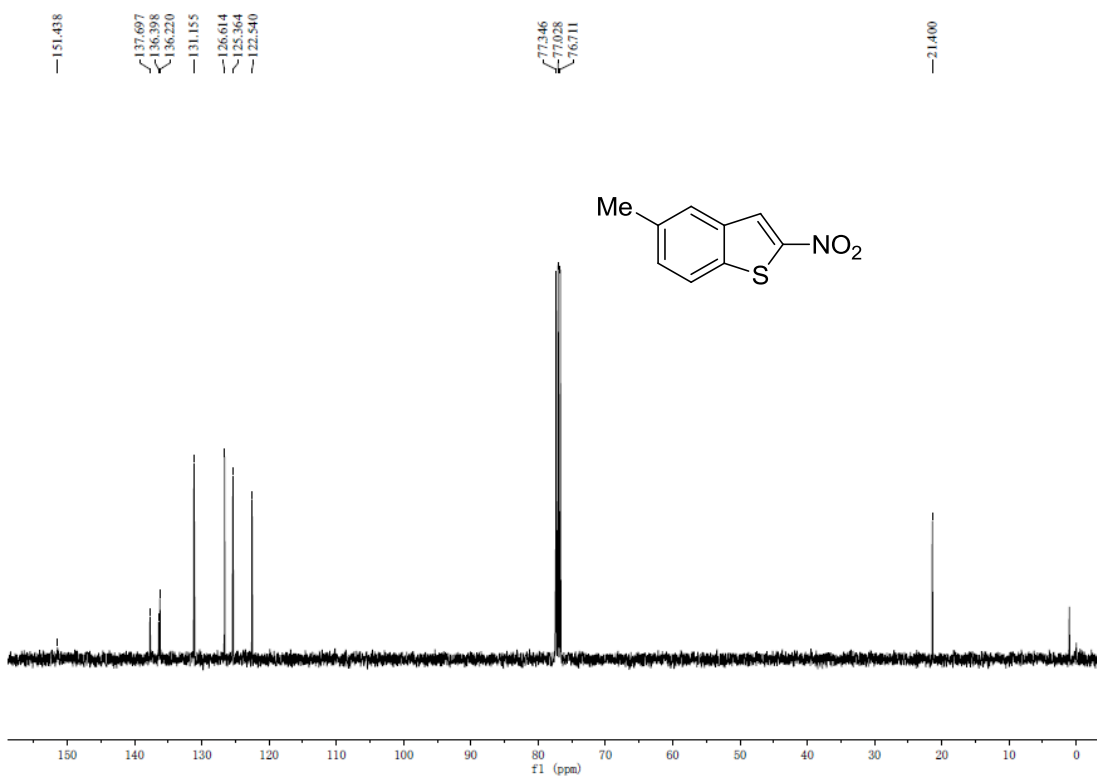
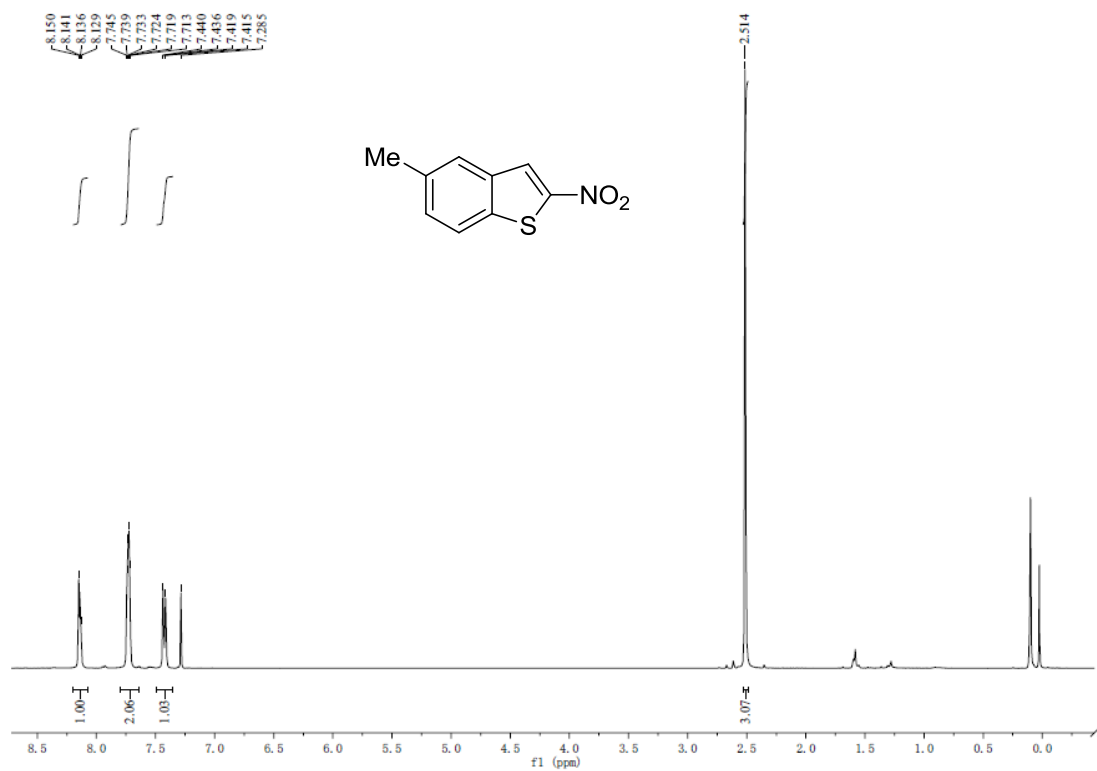
Identification code	3o
Empirical formula	C ₁₅ H ₁₅ NO ₆
Formula weight	305.28
Temperature/K	293(2)
Crystal system	triclinic
Space group	P-1
a/Å	6.9301(3)
b/Å	12.5301(7)
c/Å	17.0571(10)
α/°	85.888(5)
β/°	88.194(4)
γ/°	79.004(4)
Volume/Å ³	1449.97(14)
Z	4
ρ _{calc} /cm ³	1.398
μ/mm ⁻¹	0.926
F(000)	640.0
Crystal size/mm ³	0.2 × 0.14 × 0.1
Radiation	CuKα (λ = 1.54184)
2θ range for data collection/°	7.204 to 134.152
Index ranges	-8 ≤ h ≤ 5, -14 ≤ k ≤ 14, -20 ≤ l ≤ 20
Reflections collected	10304
Independent reflections	5176 [R _{int} = 0.0235, R _{sigma} = 0.0373]
Data/restraints/parameters	5176/4/418
Goodness-of-fit on F ²	1.039
Final R indexes [I ≥ 2σ (I)]	R ₁ = 0.0496, wR ₂ = 0.1333
Final R indexes [all data]	R ₁ = 0.0633, wR ₂ = 0.1480
Largest diff. peak/hole / e Å ⁻³	0.20/-0.20

2. ^1H and ^{13}C for new substrates 4 and 6

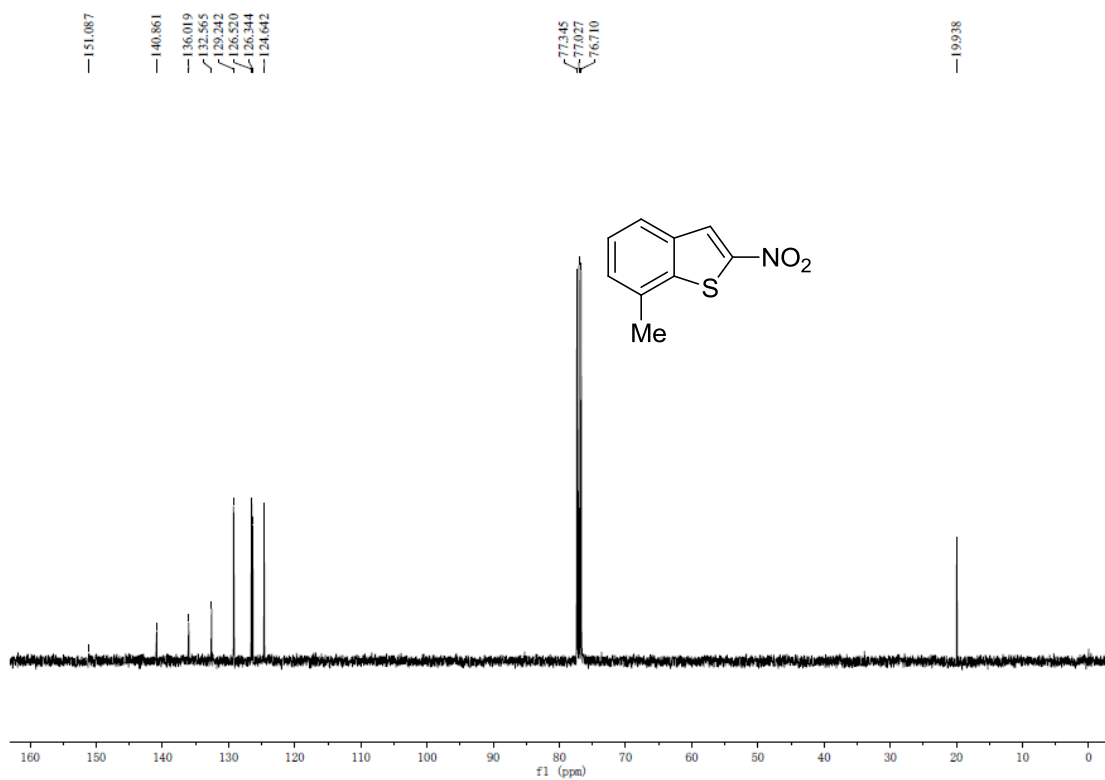
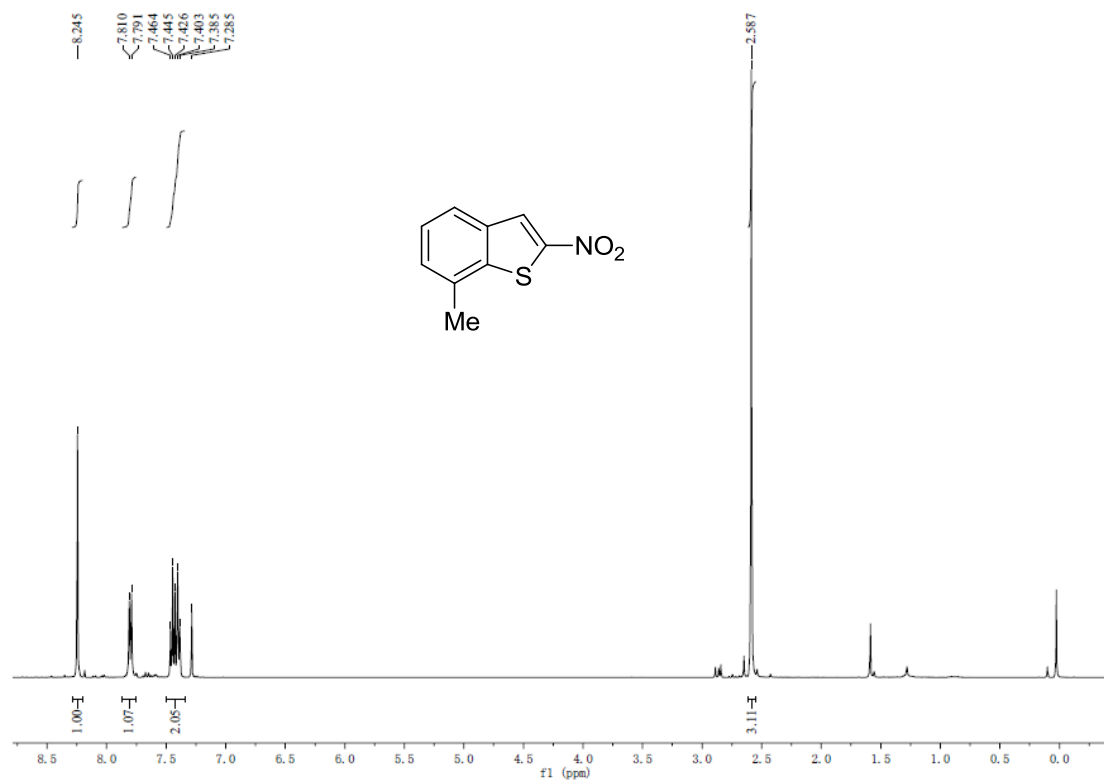
^1H and ^{13}C NMR of **4b**



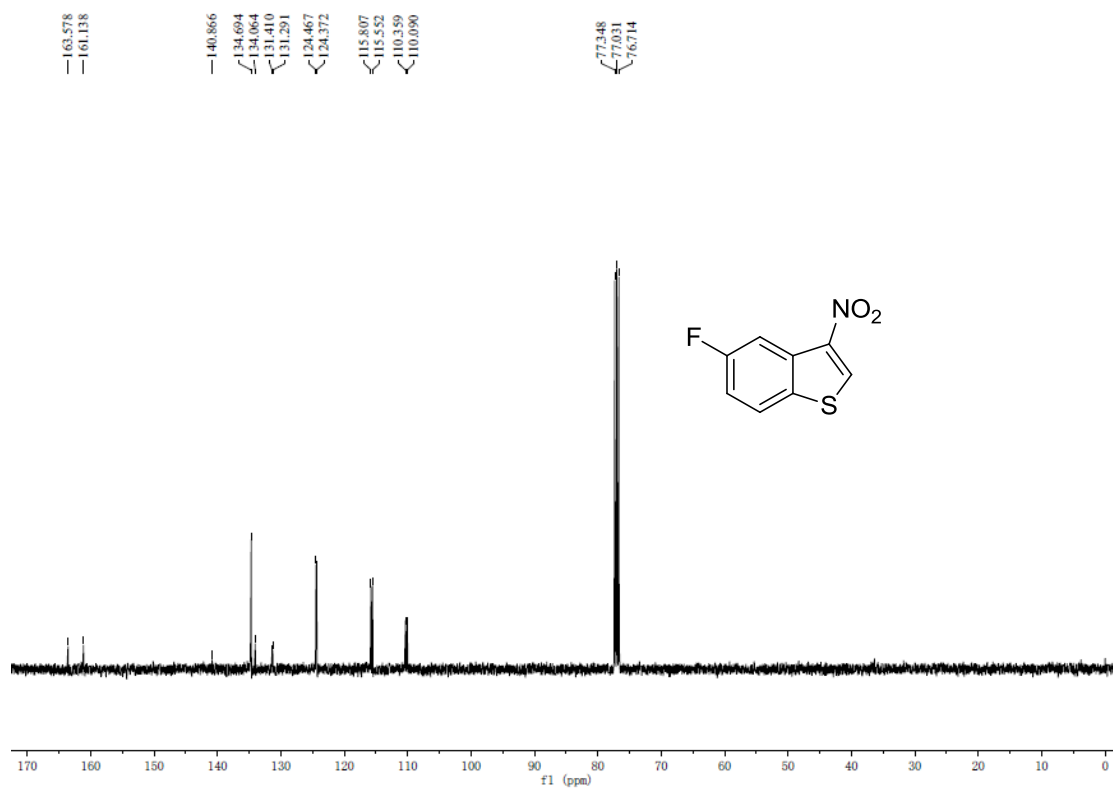
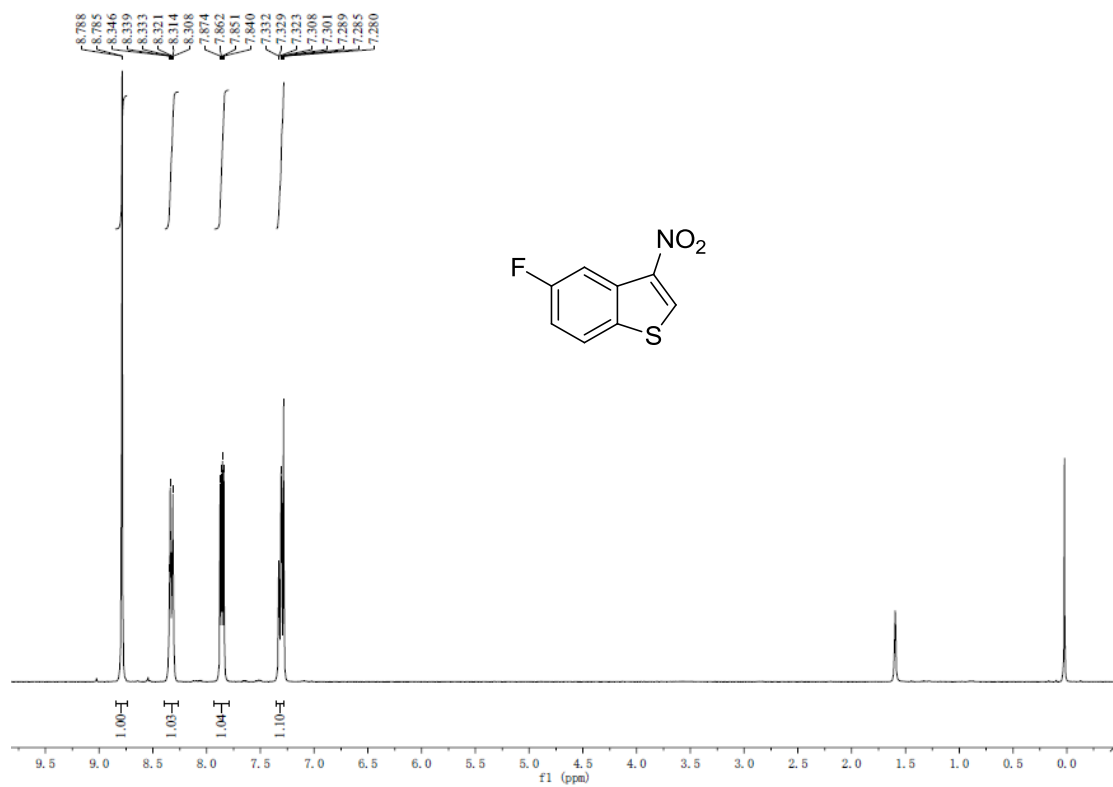
^1H and ^{13}C NMR of **4c**



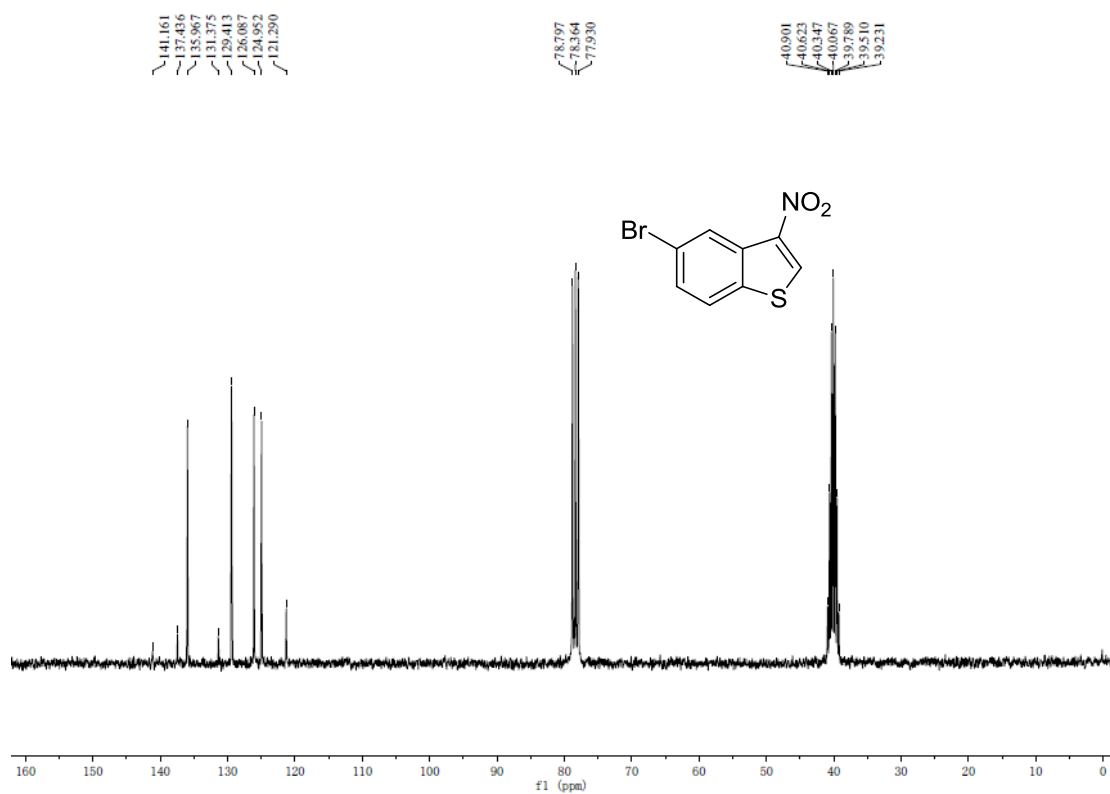
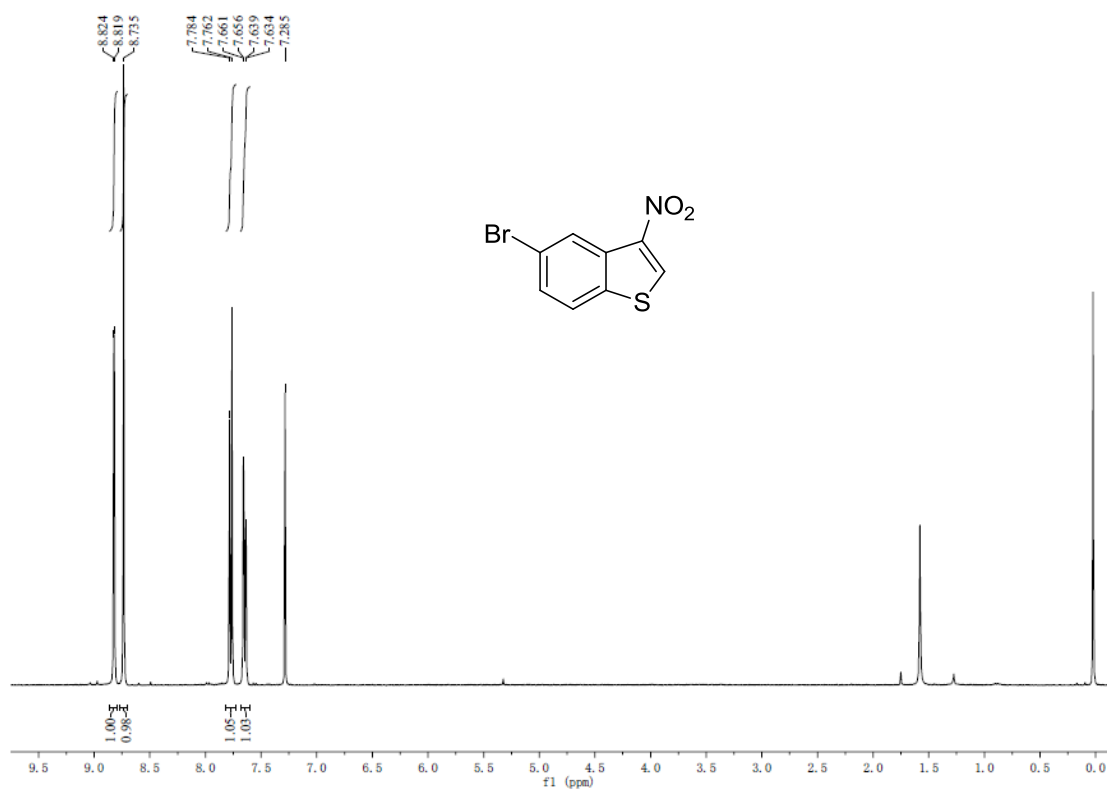
^1H and ^{13}C NMR of **4d**



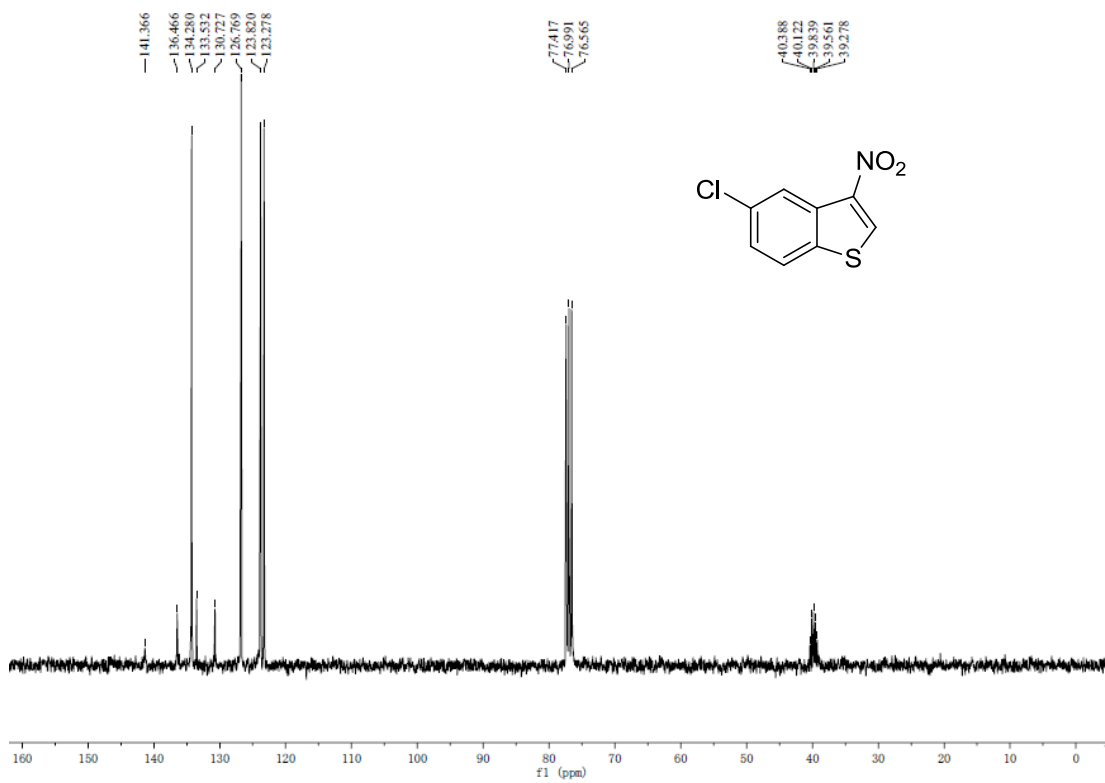
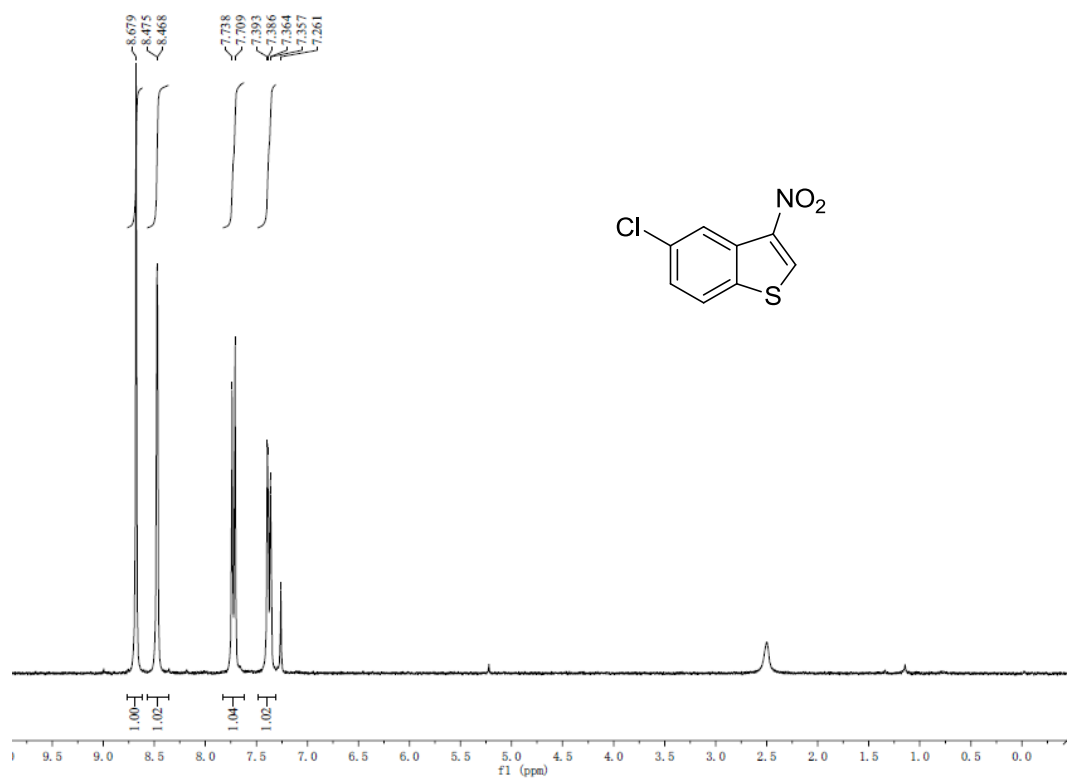
¹H and ¹³C NMR of 6b



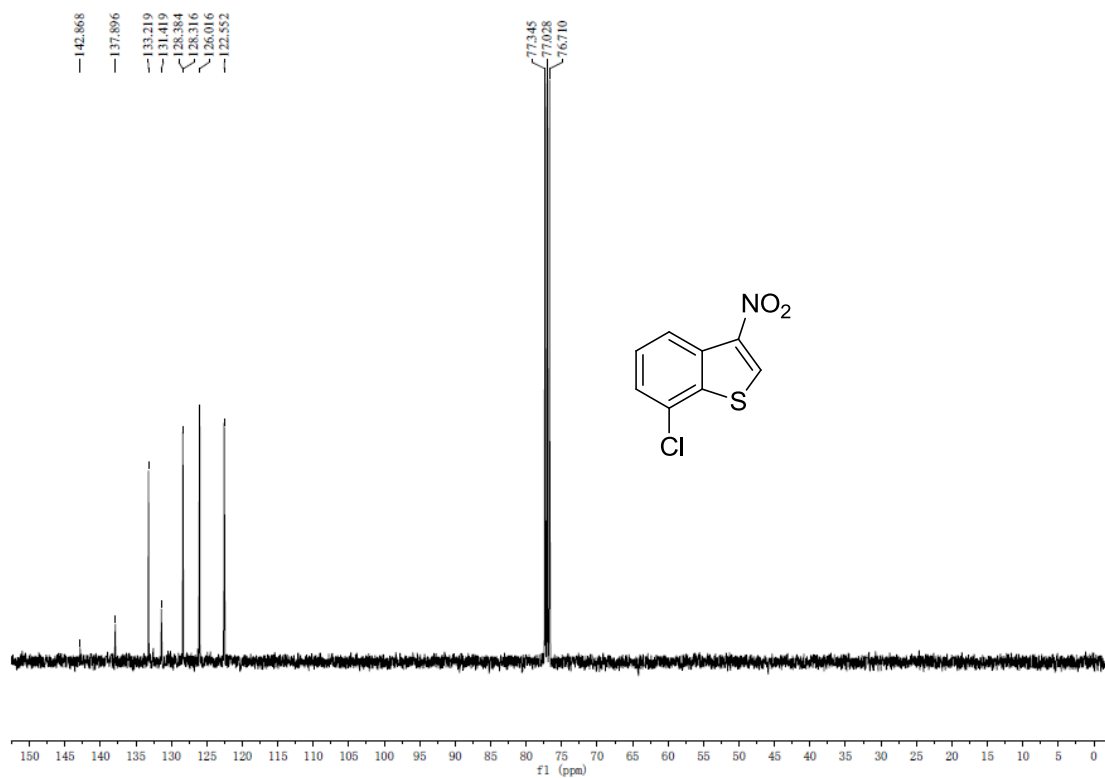
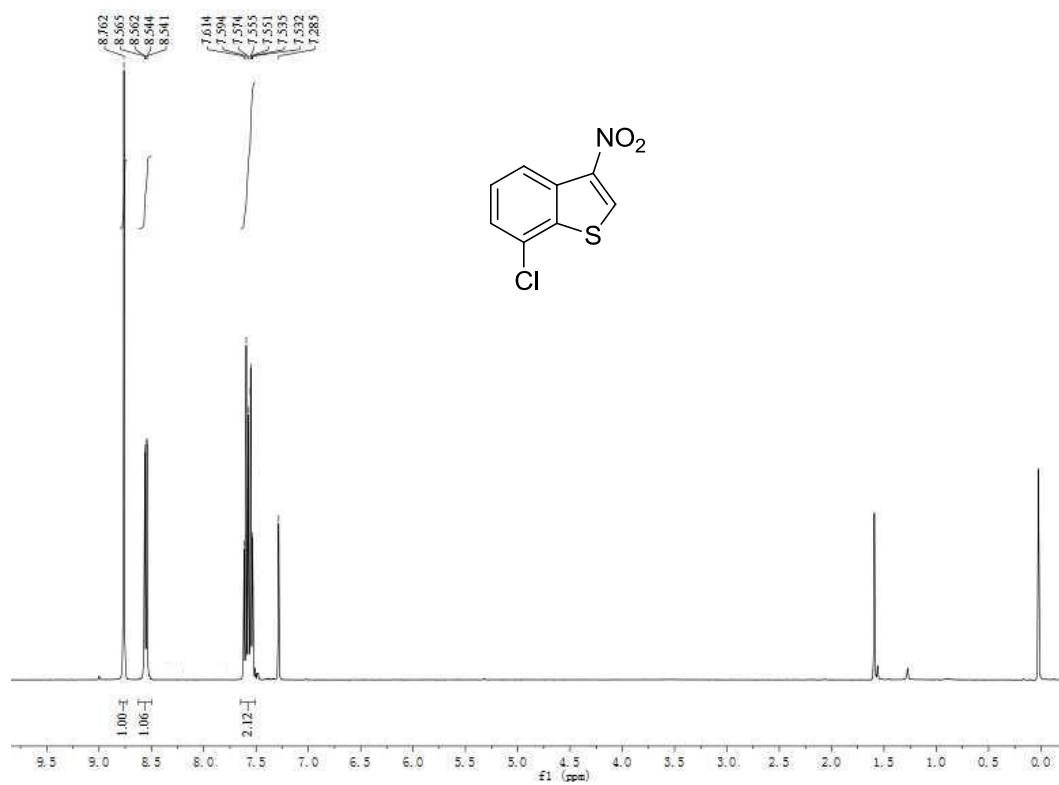
^1H and ^{13}C NMR of 6c



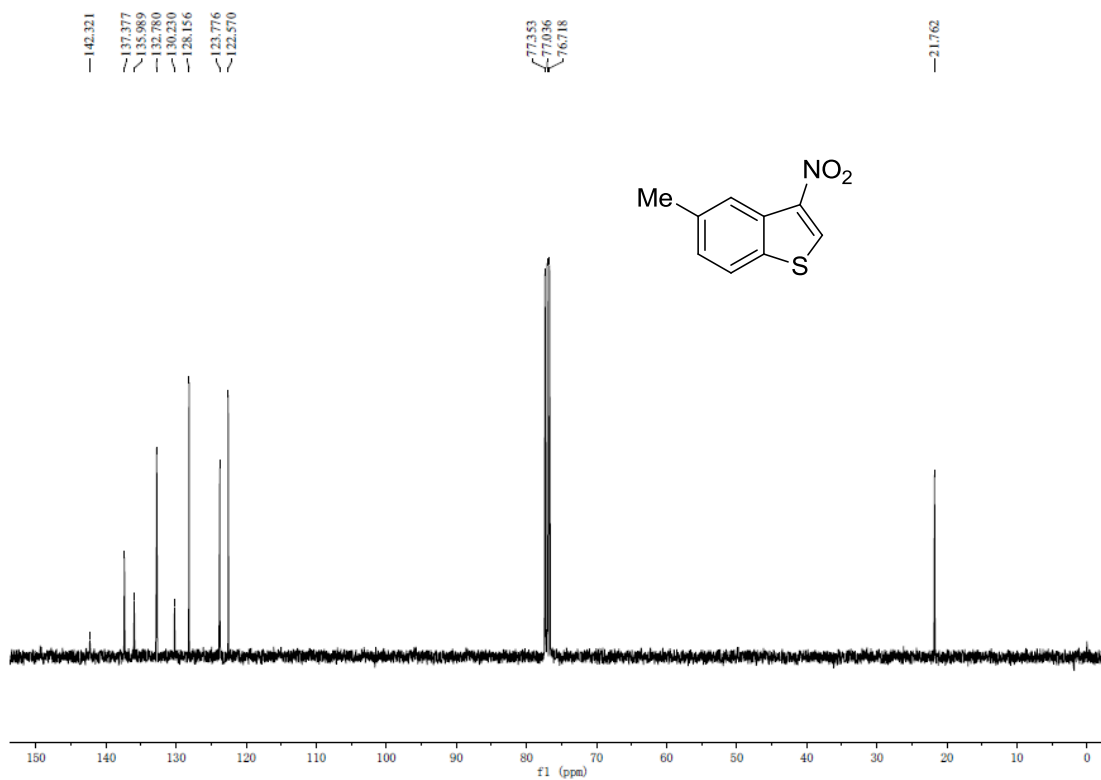
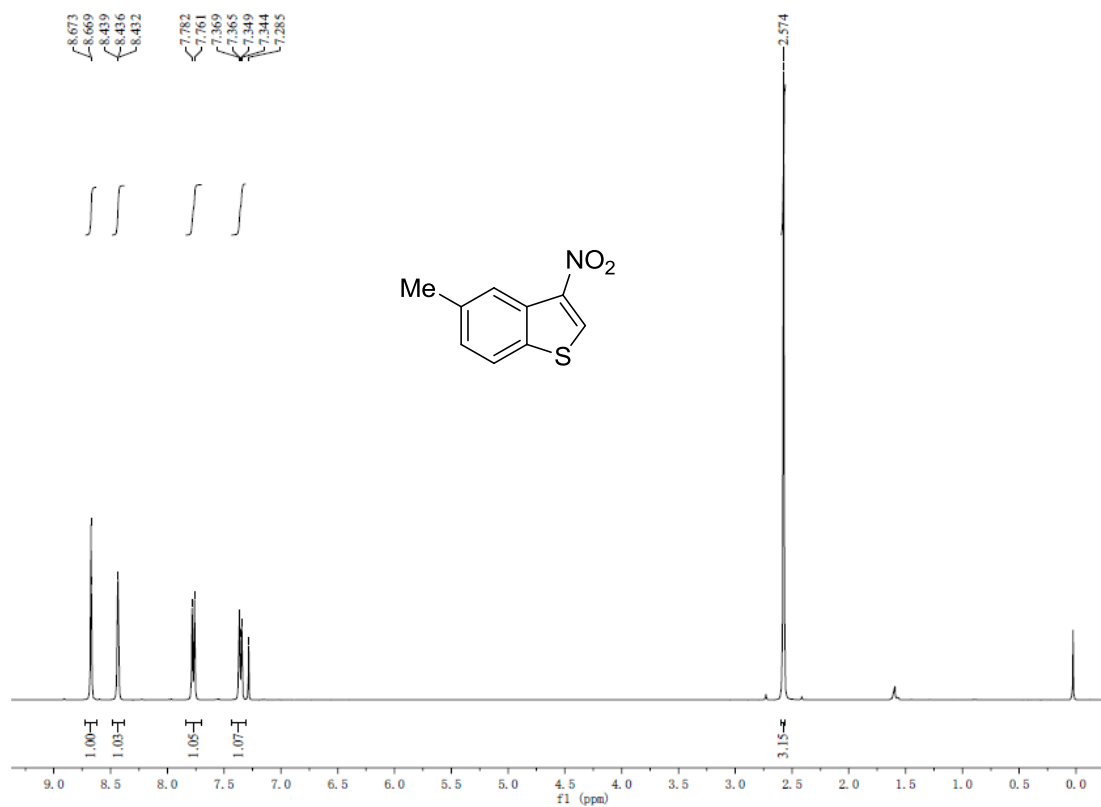
¹H and ¹³C NMR of 6d



^1H and ^{13}C NMR of 6e

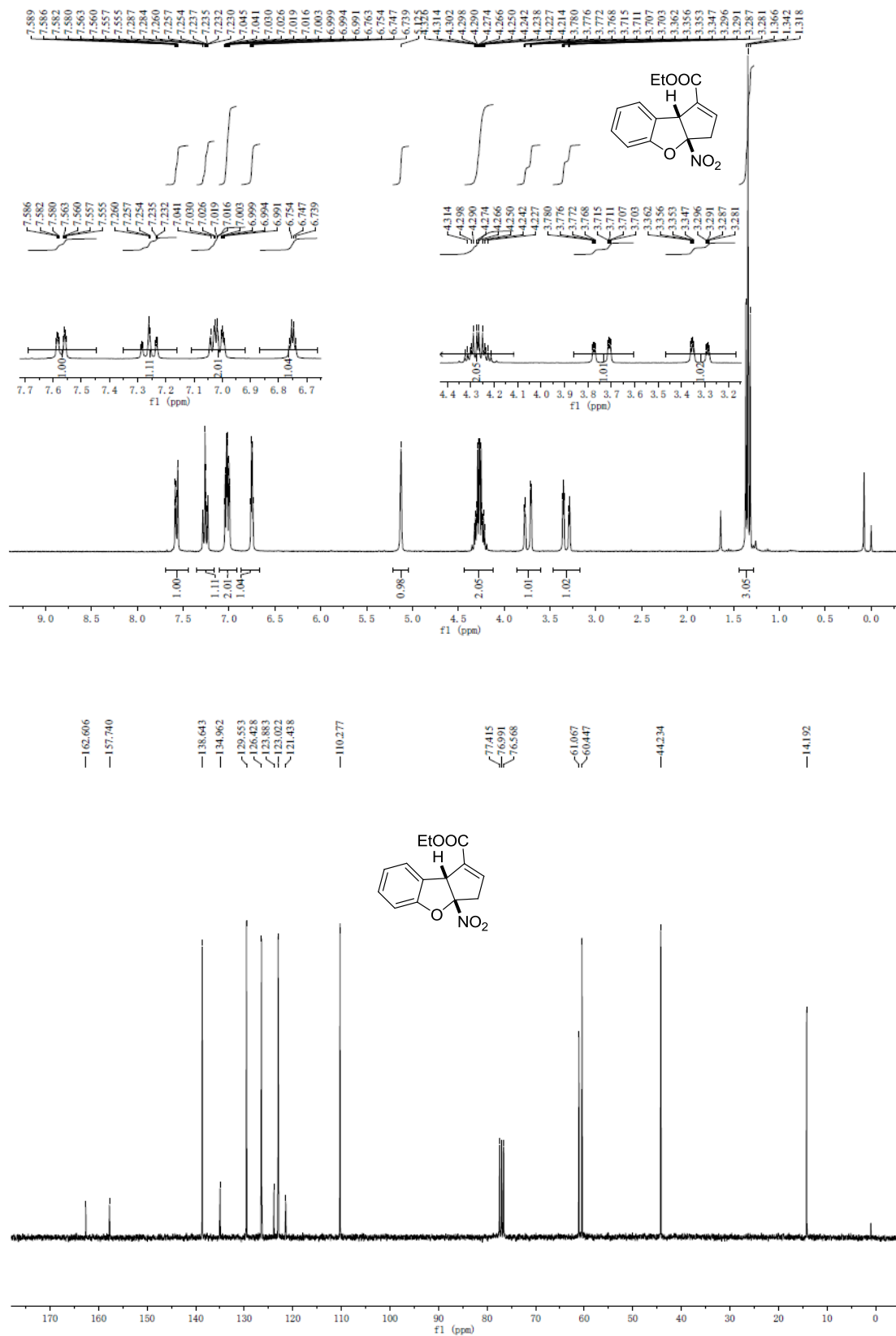


^1H and ^{13}C NMR of **6f**

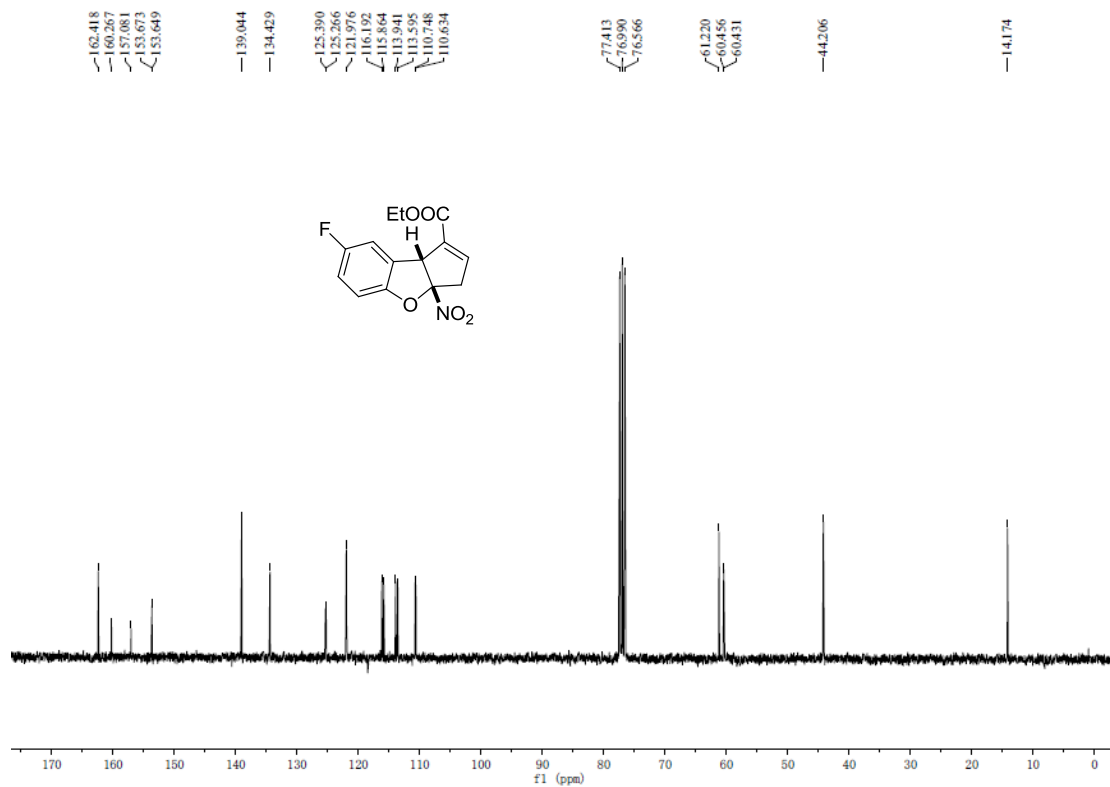
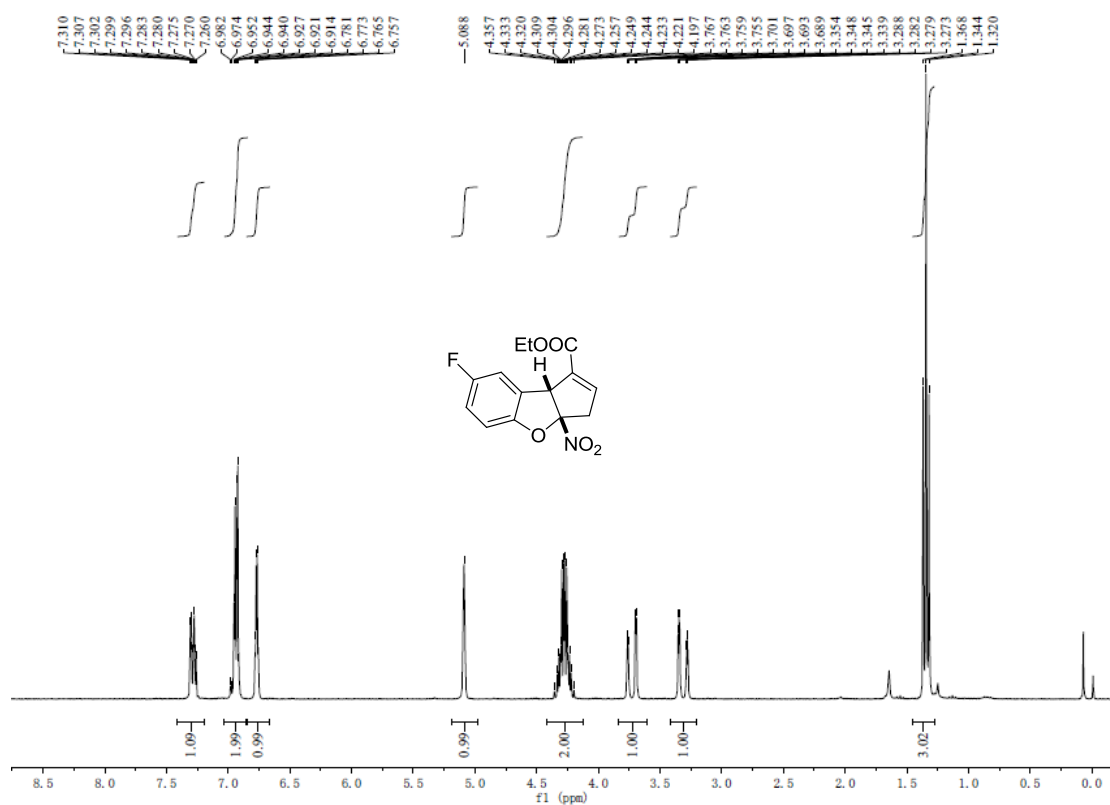


3. ^1H and ^{13}C for compounds 3, 5, 7 and 8

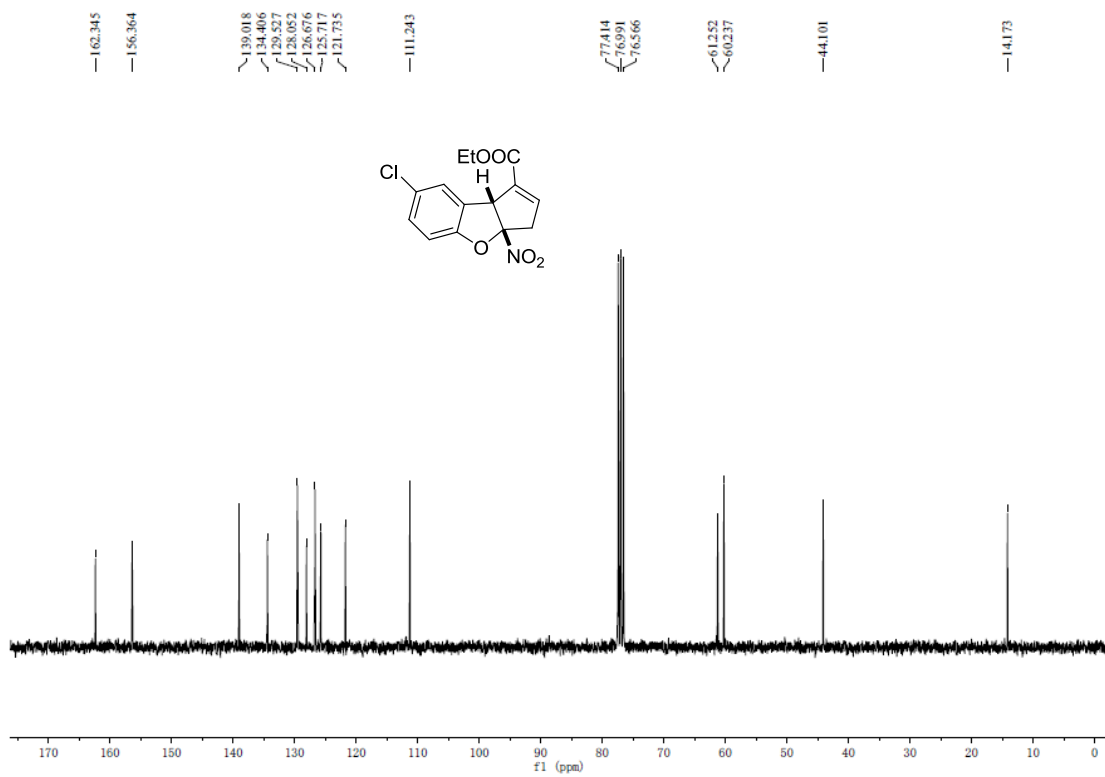
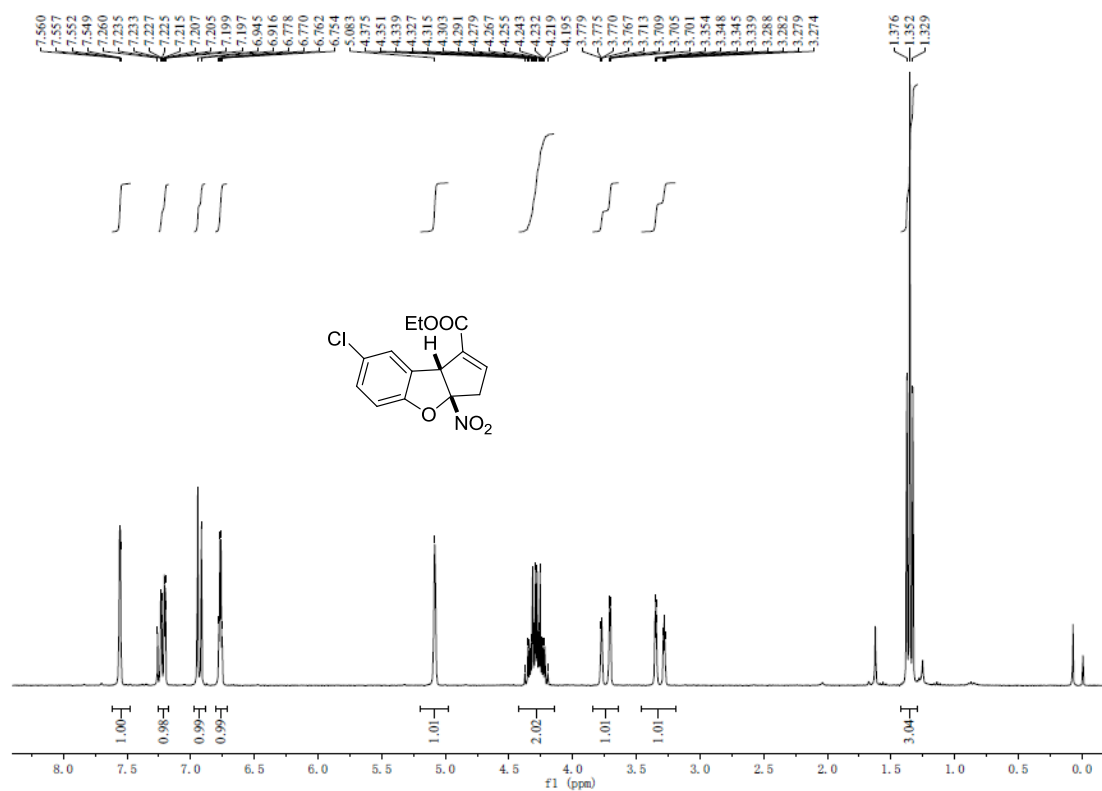
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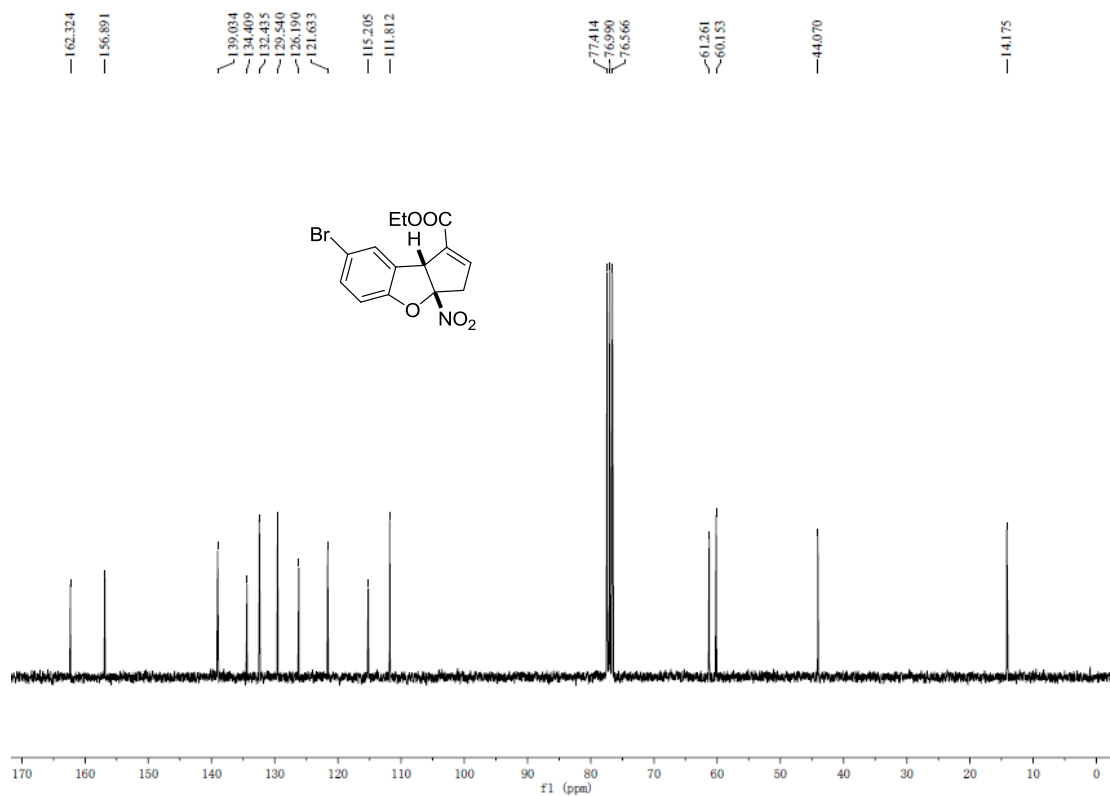
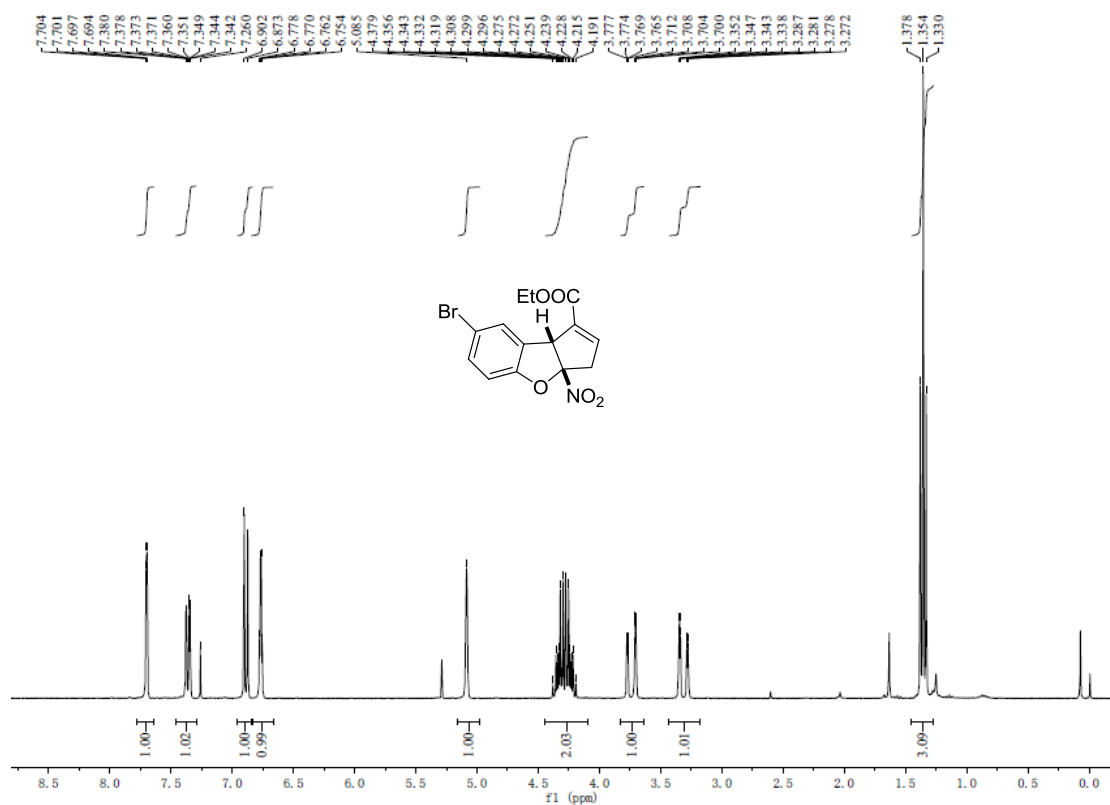
^1H and ^{13}C NMR of **3b**



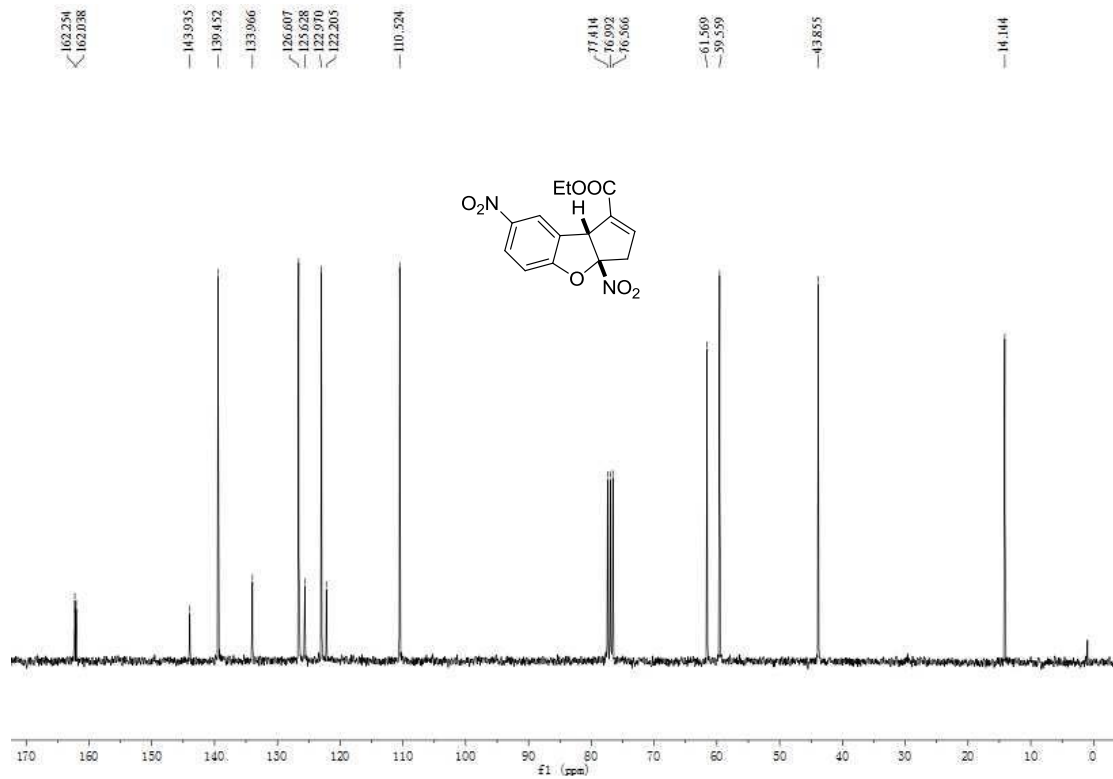
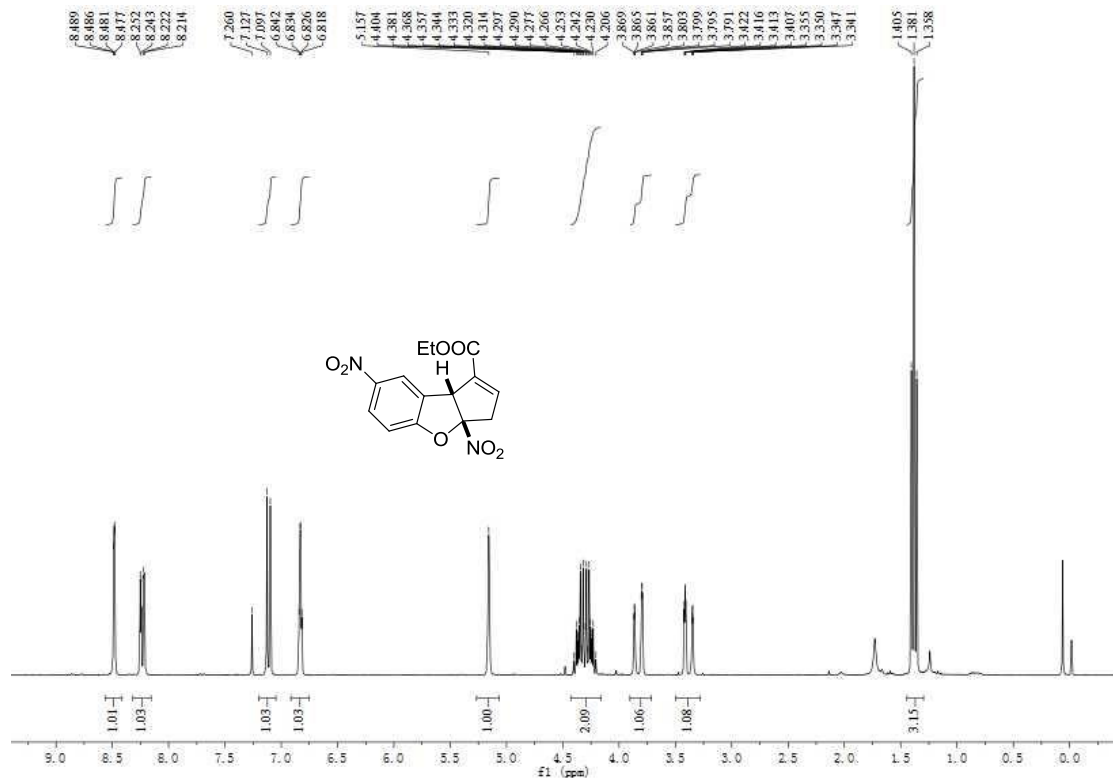
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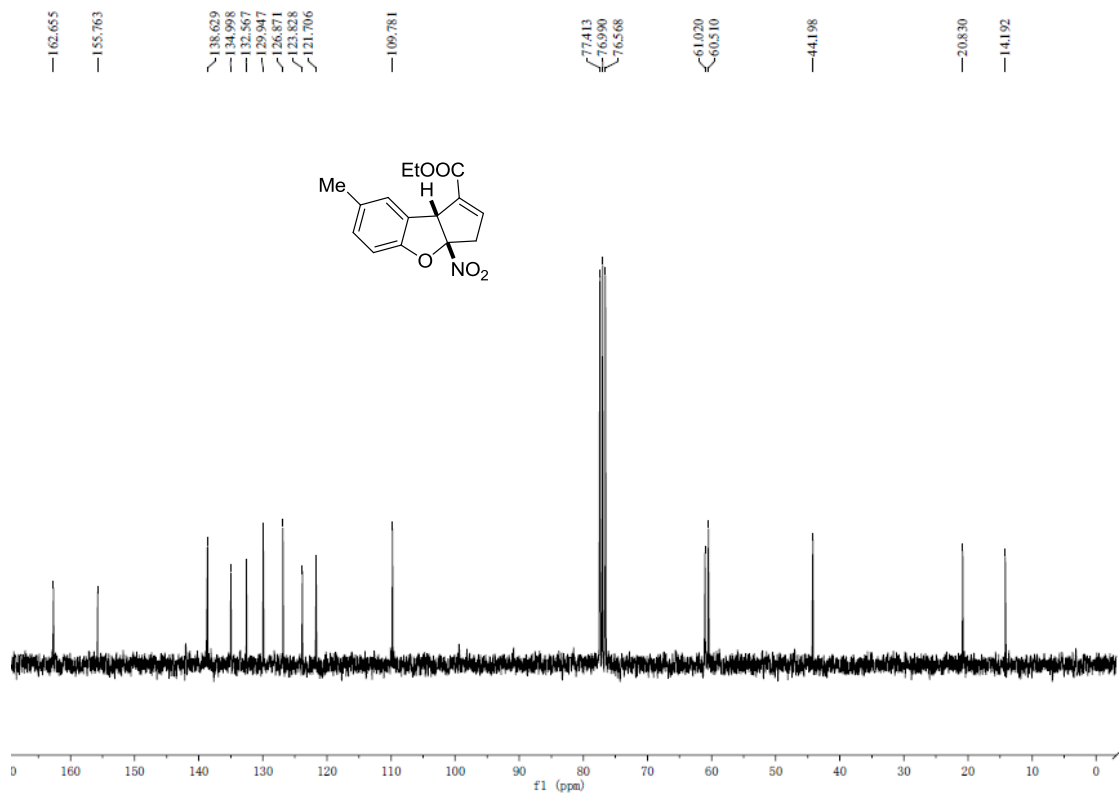
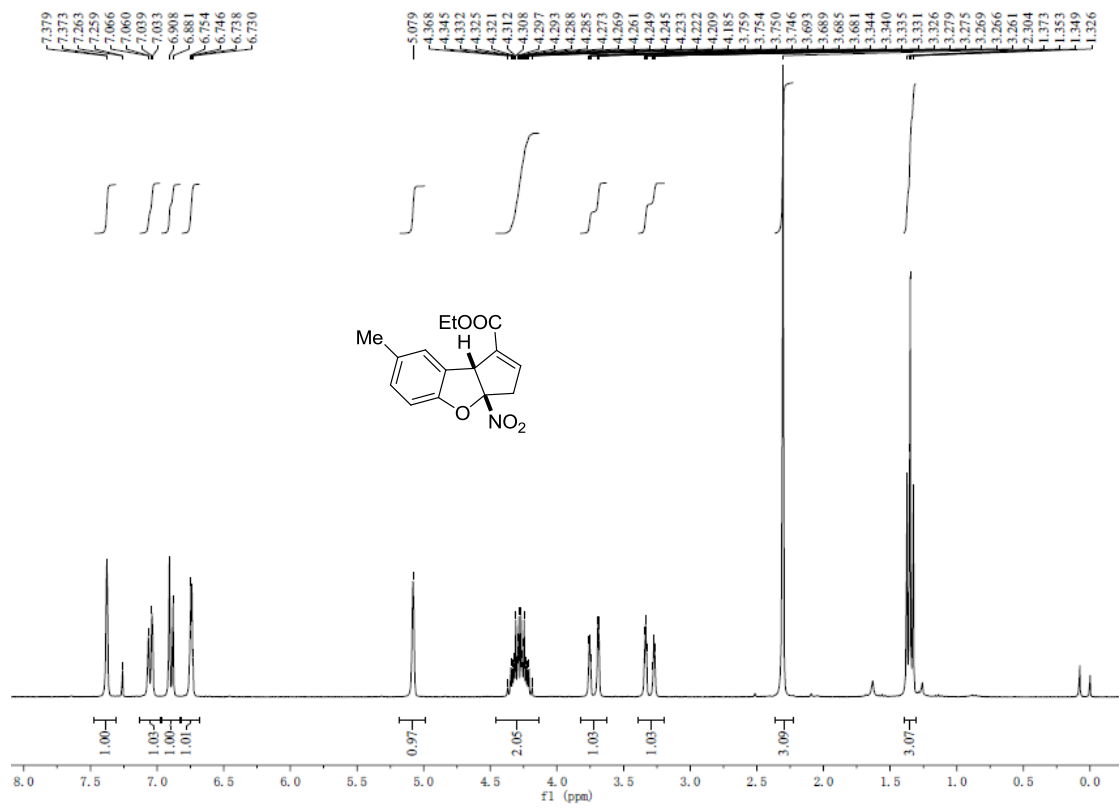
^1H and ^{13}C NMR of **3d**



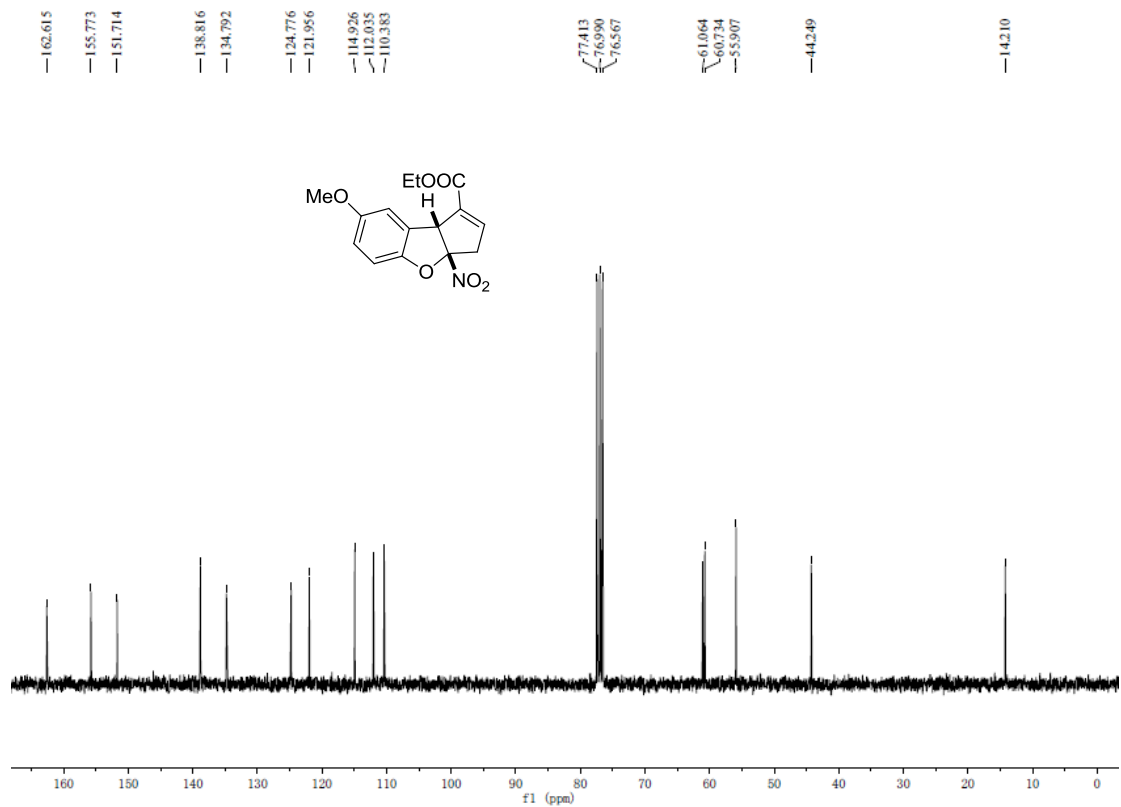
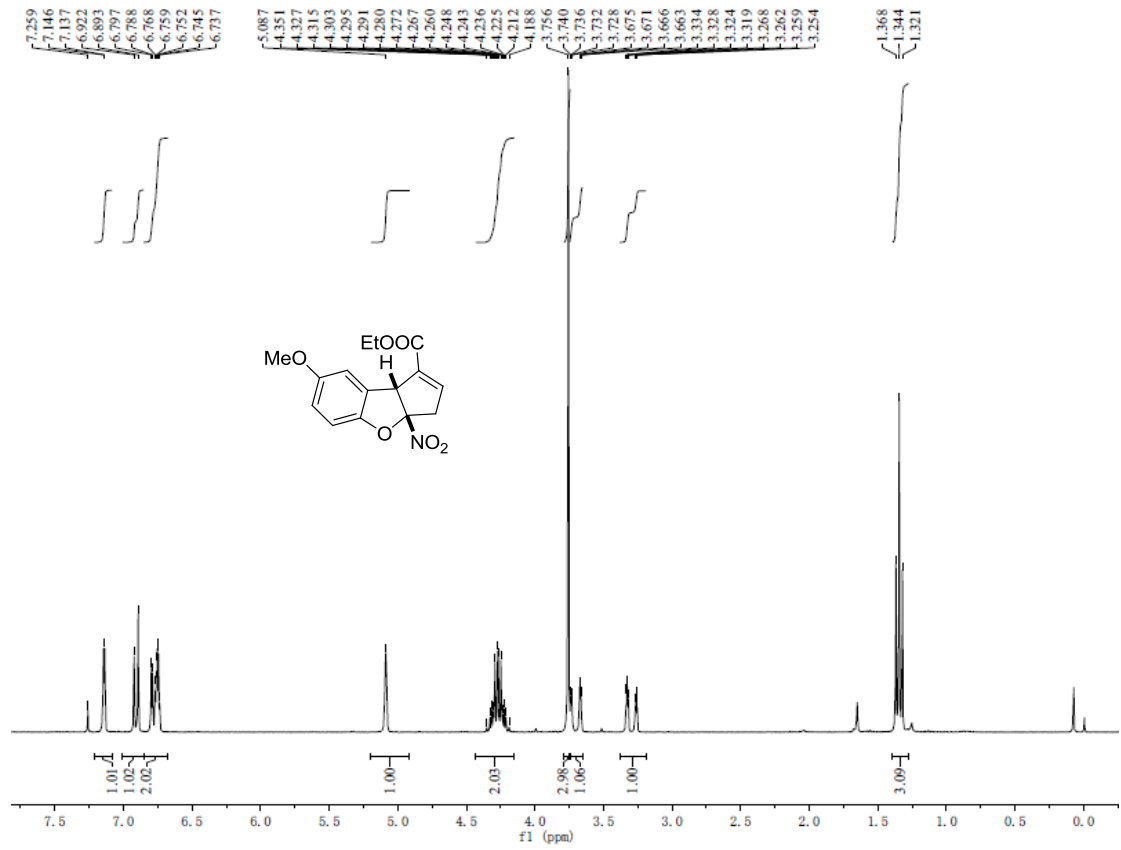
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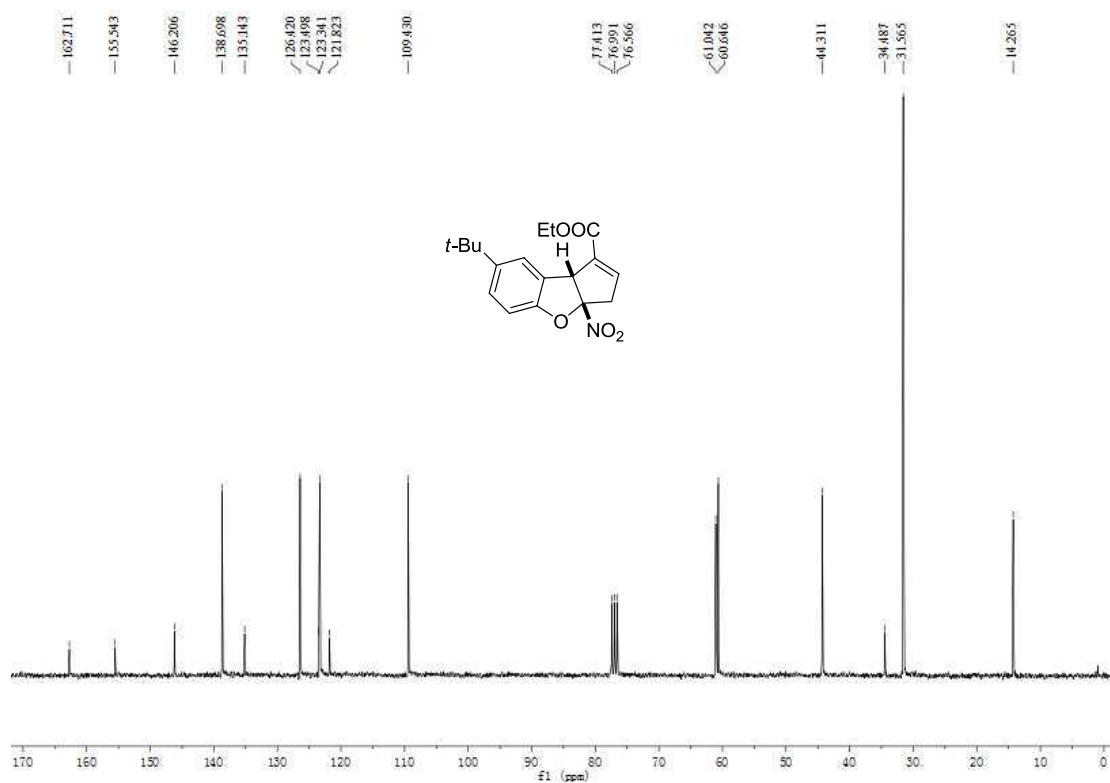
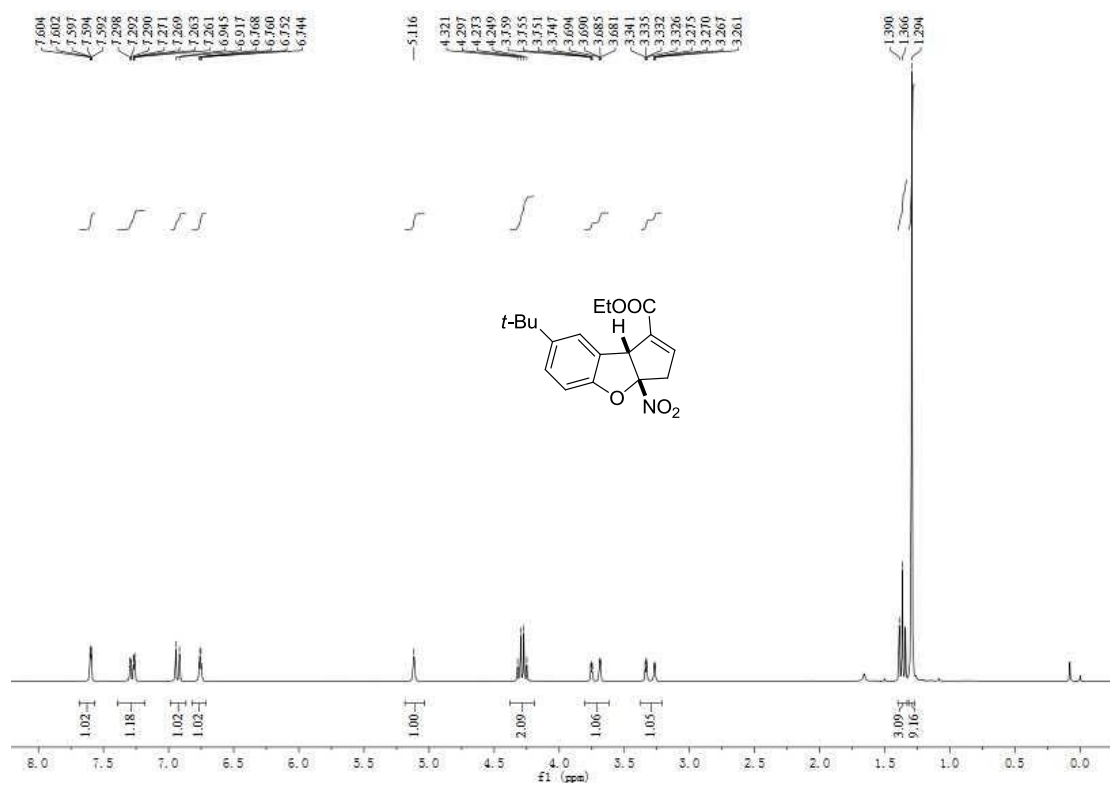
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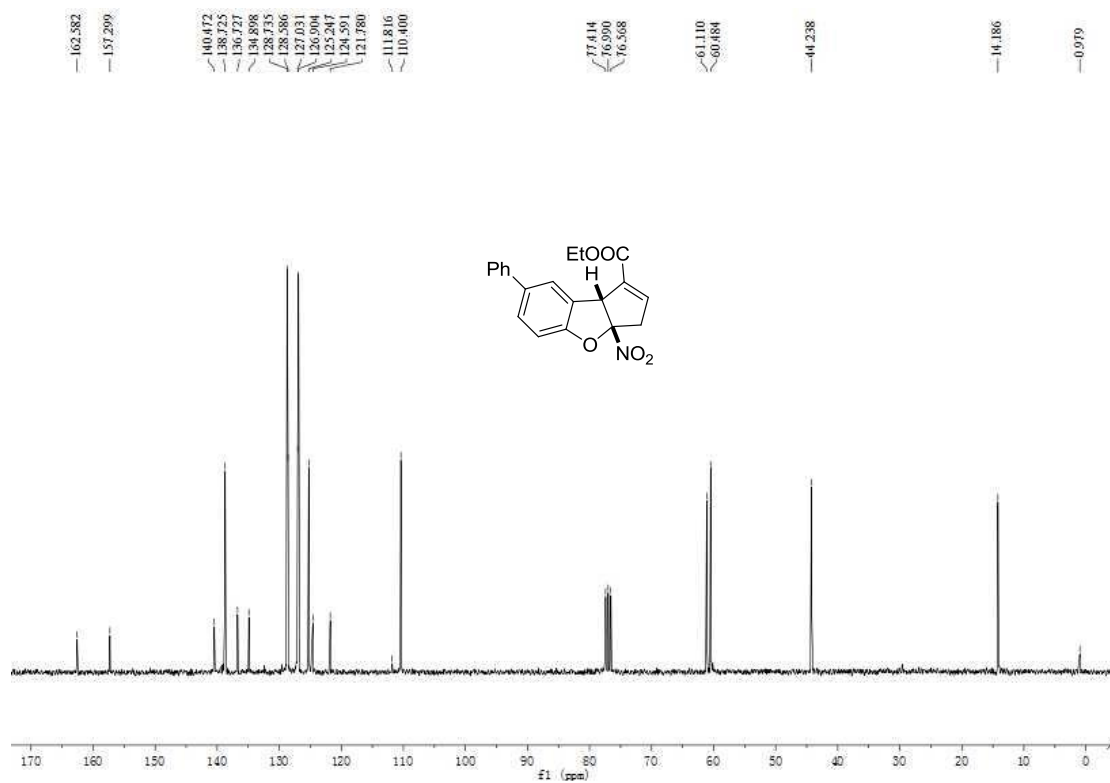
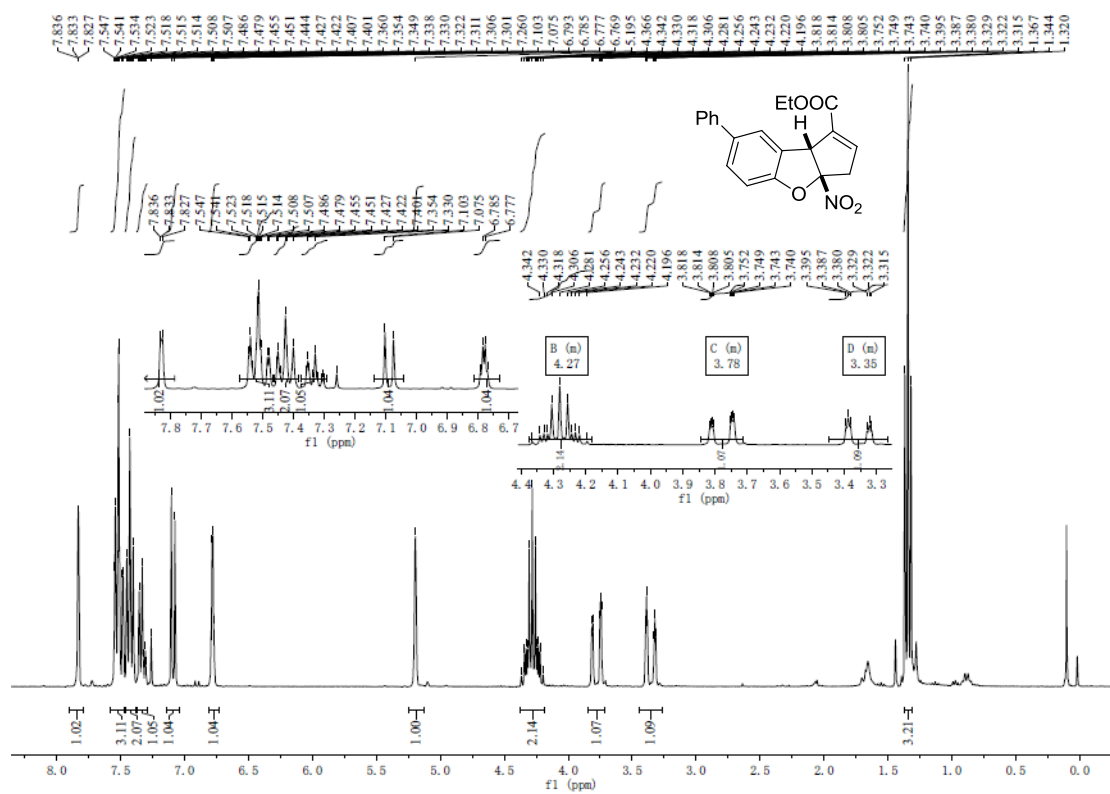
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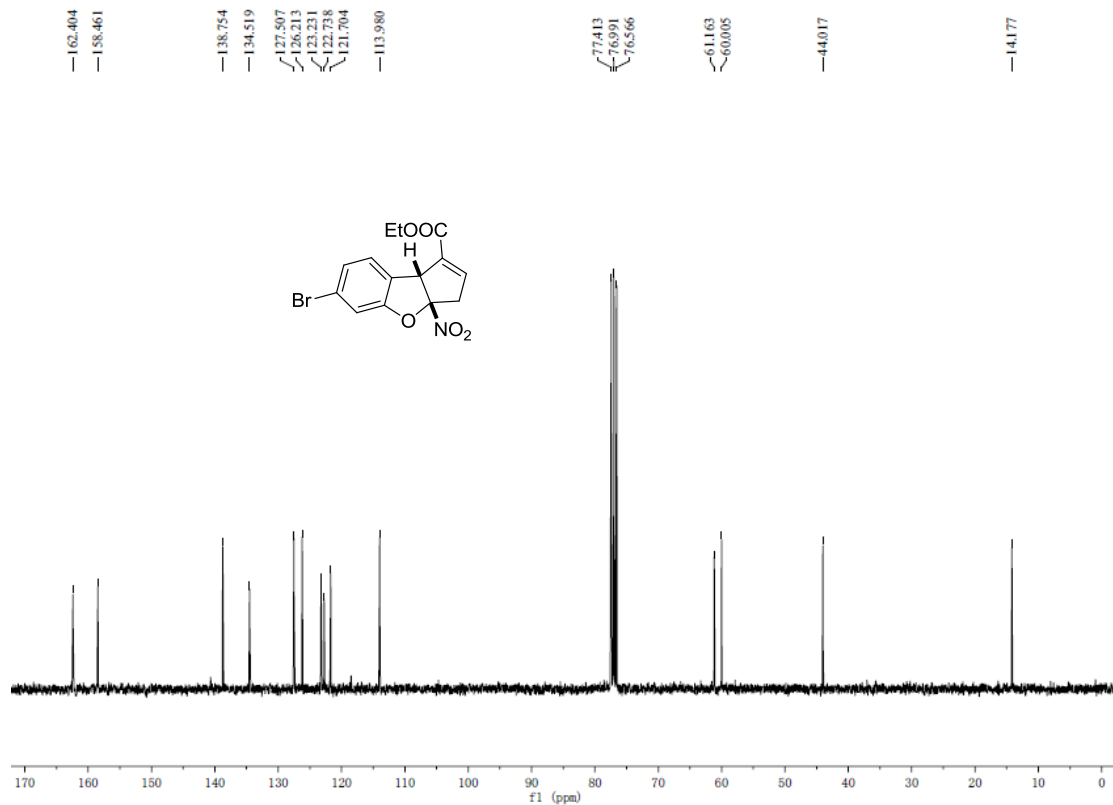
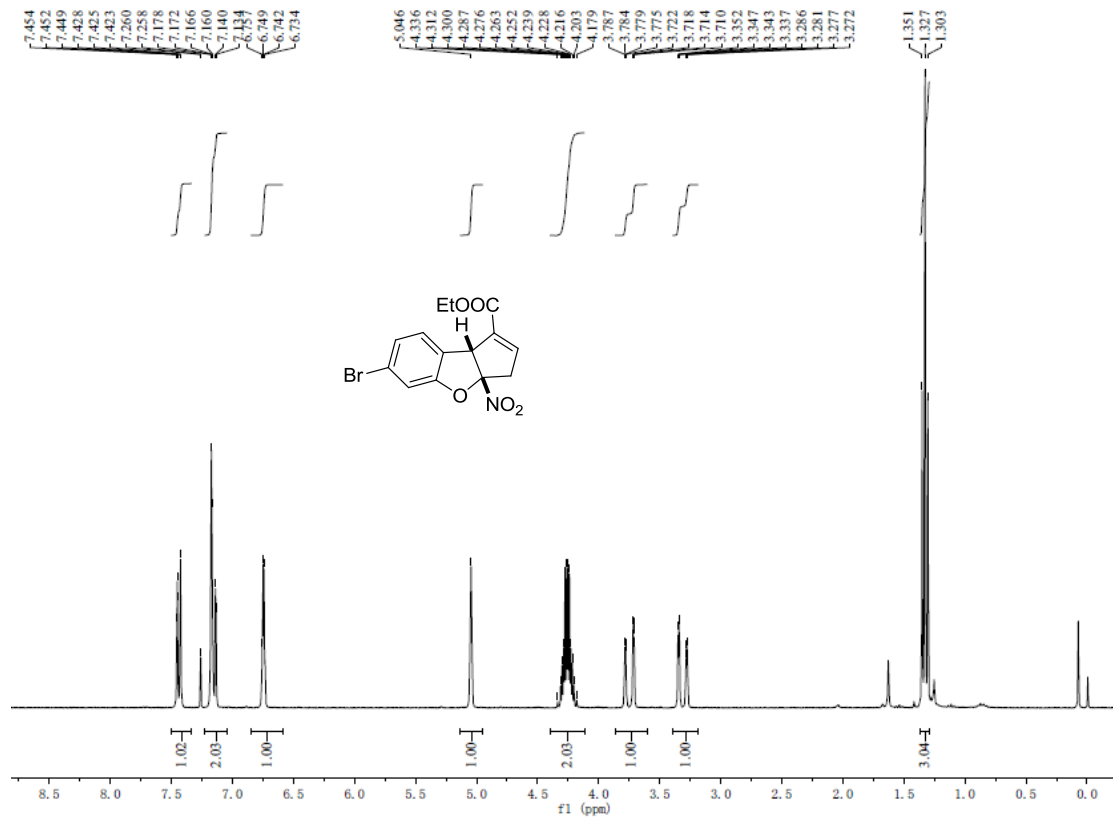
^1H and ^{13}C NMR of **3h**



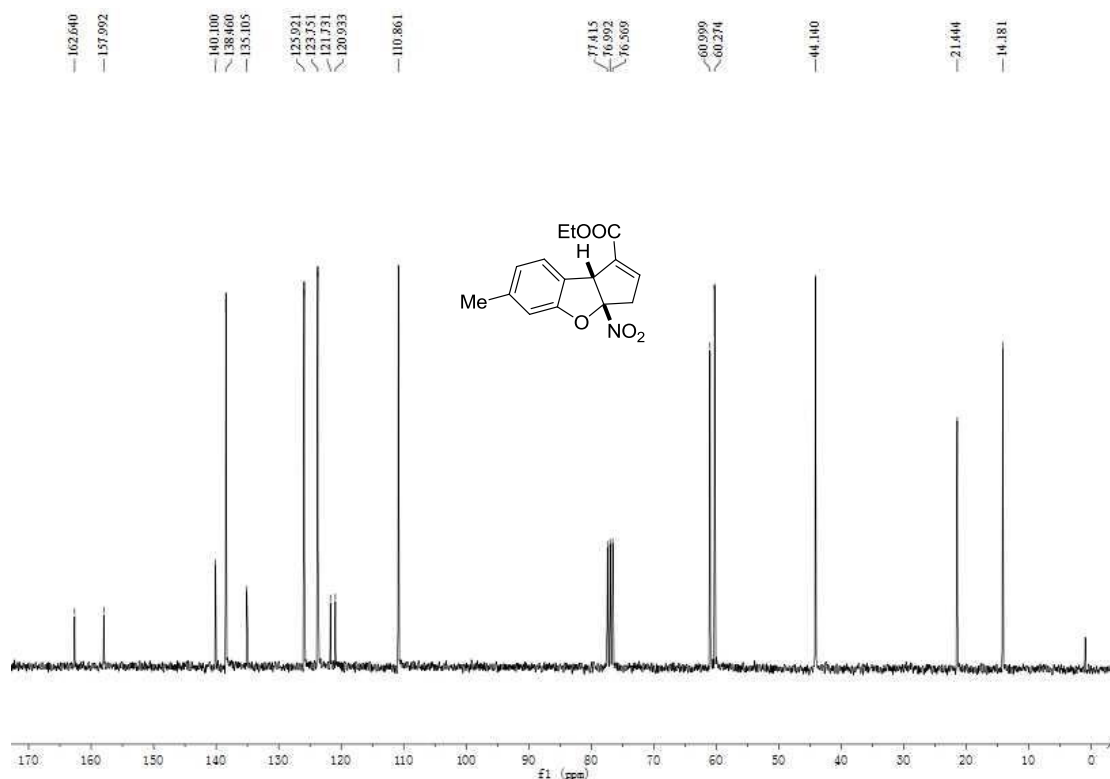
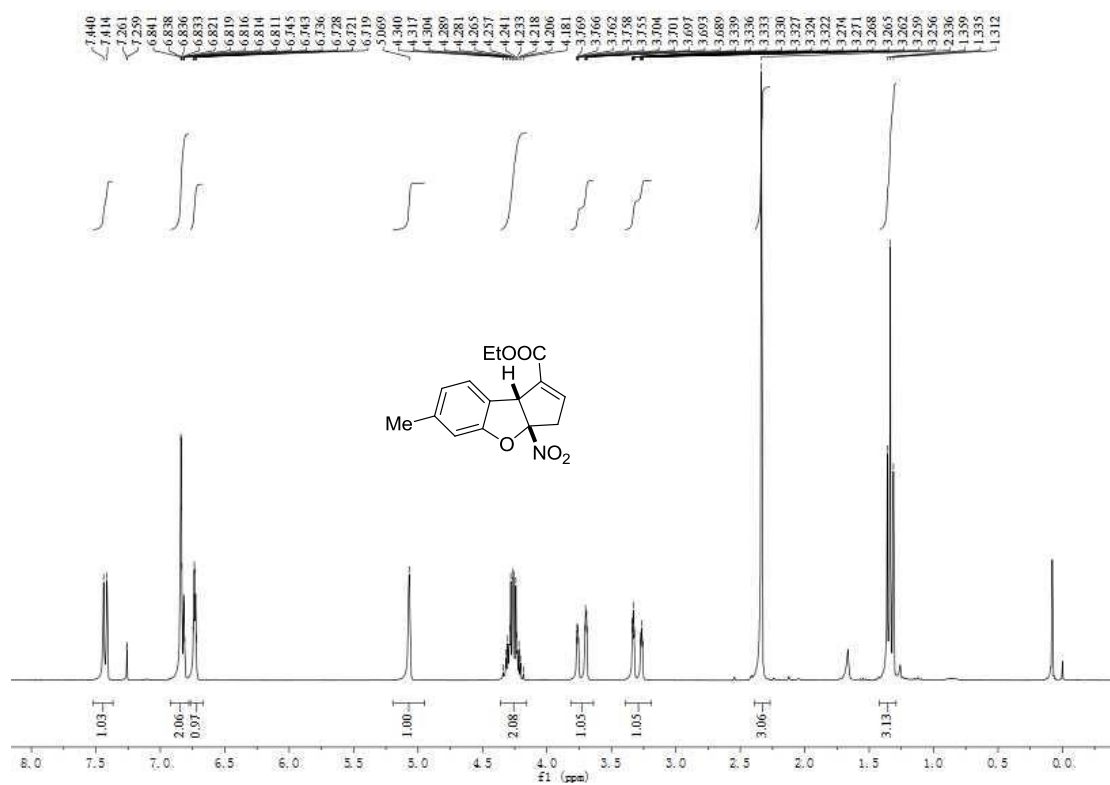
^1H and ^{13}C NMR of **3i**



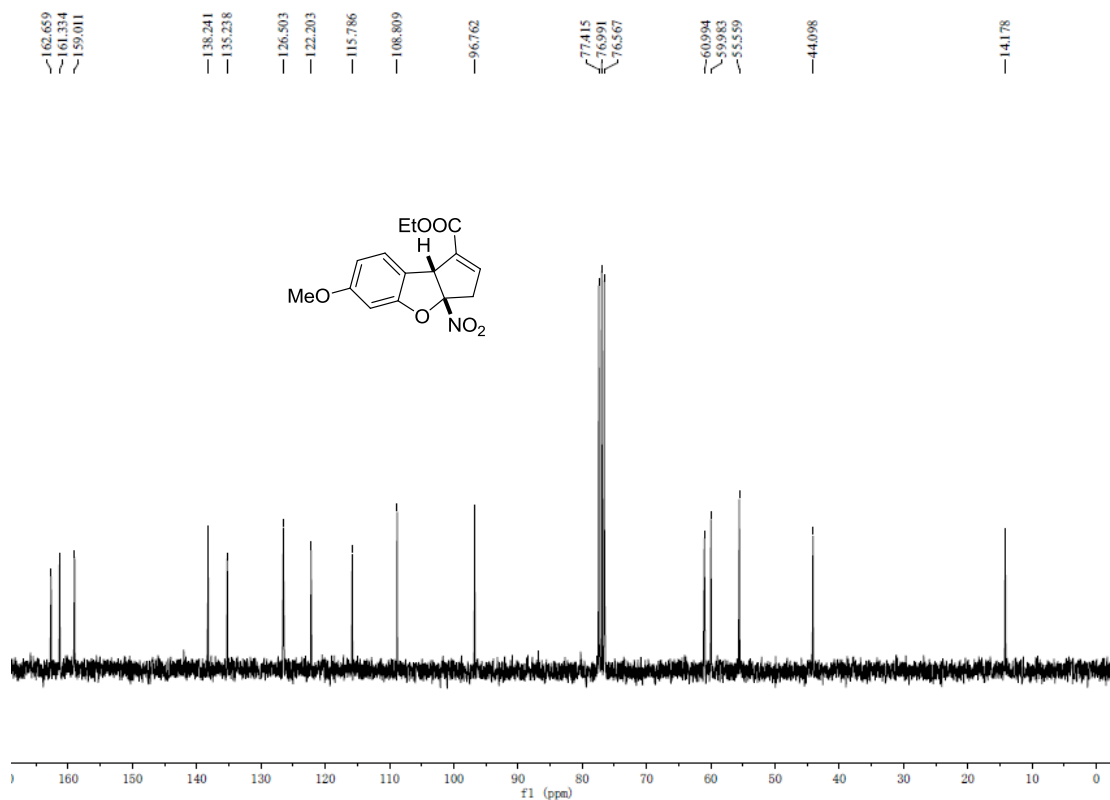
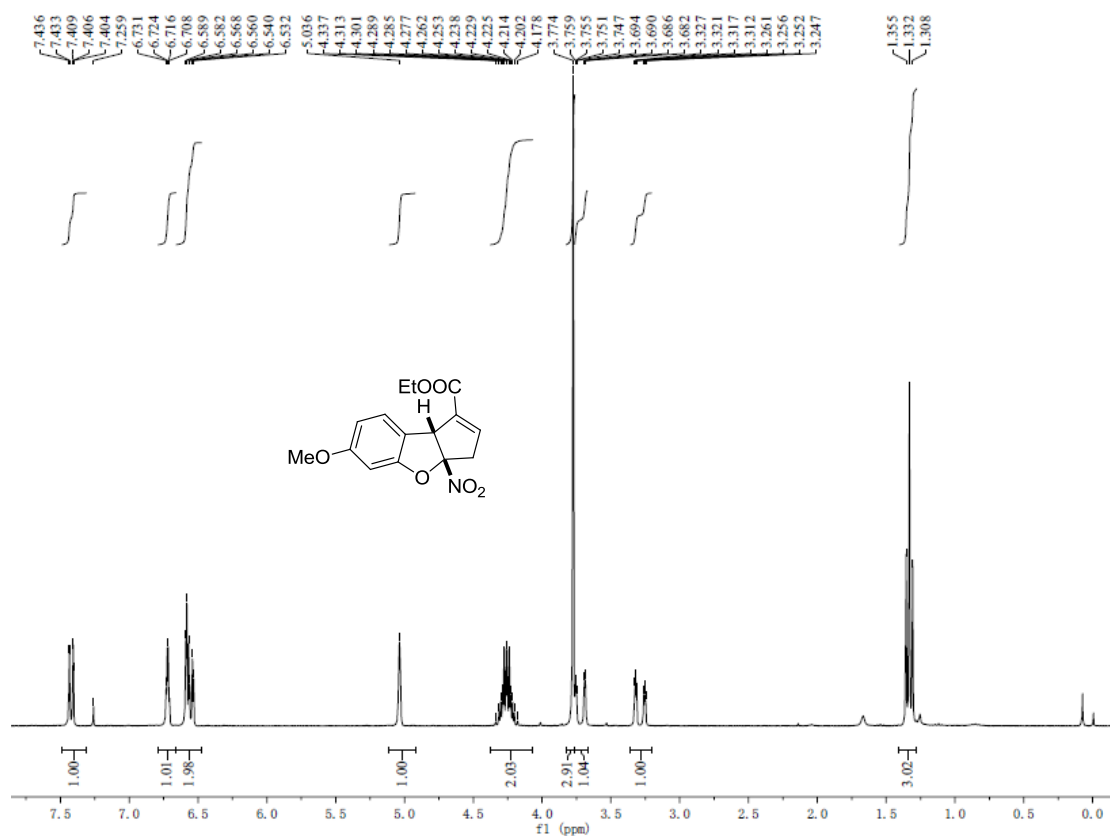
^1H and ^{13}C NMR of **3j**



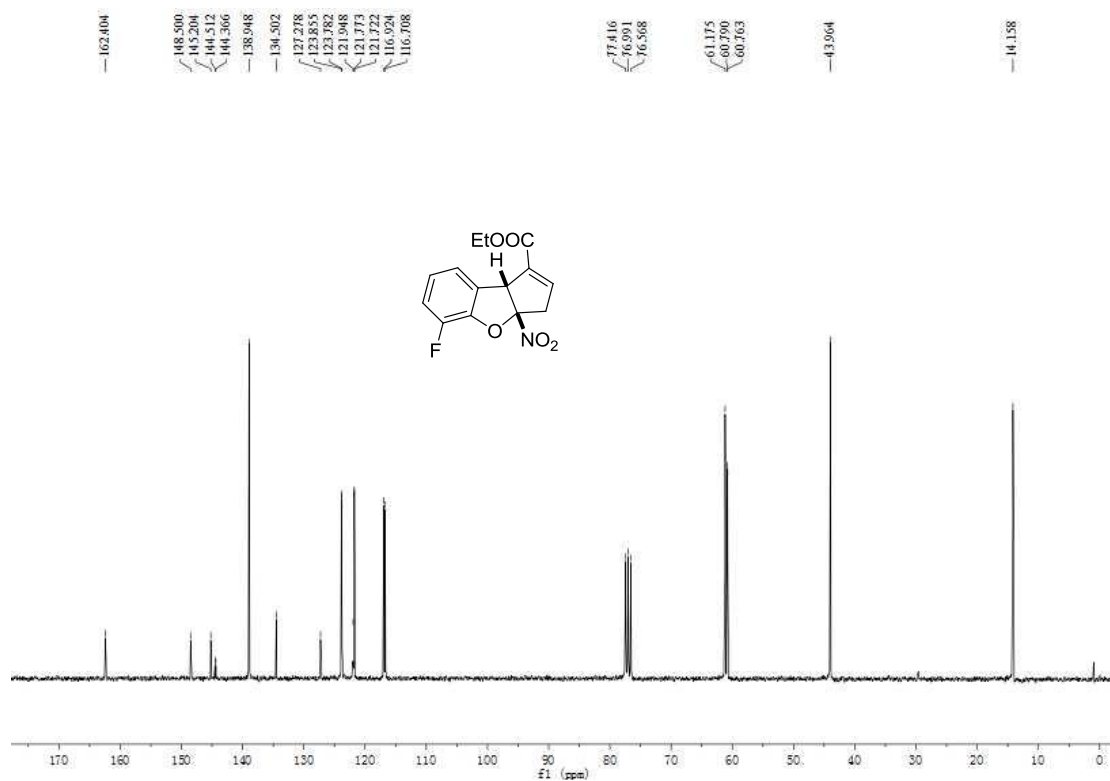
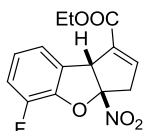
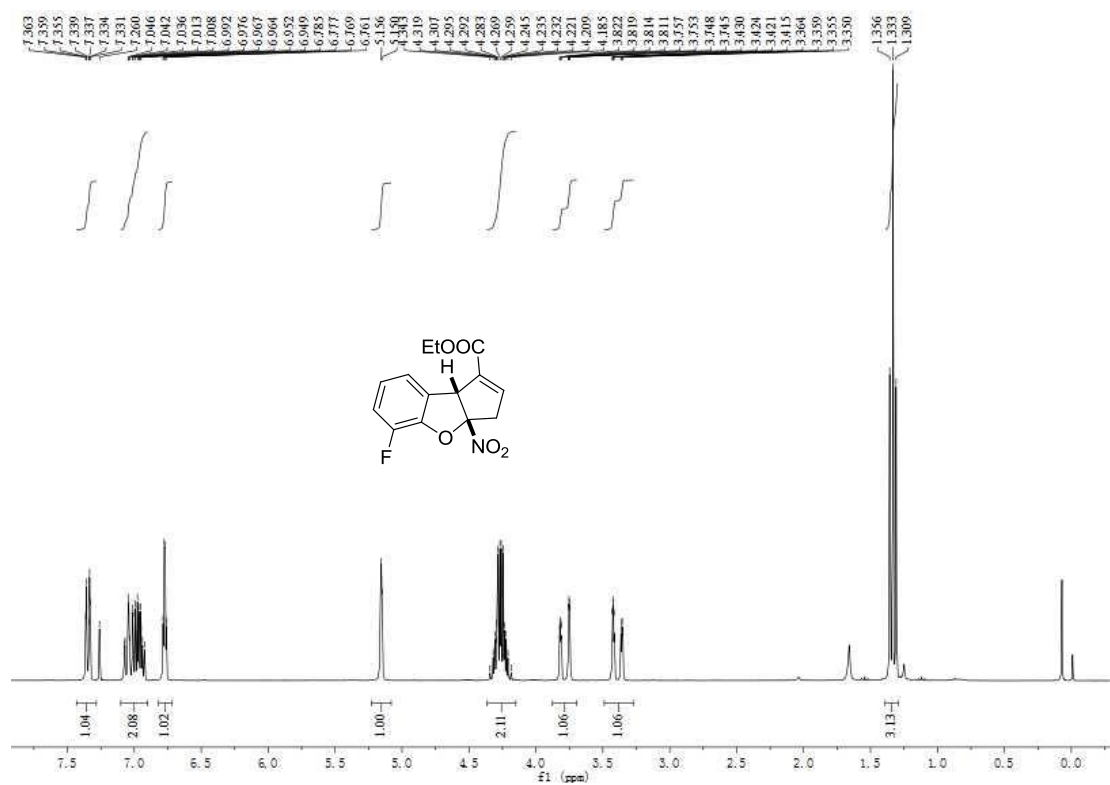
¹H and ¹³C NMR of **3k**



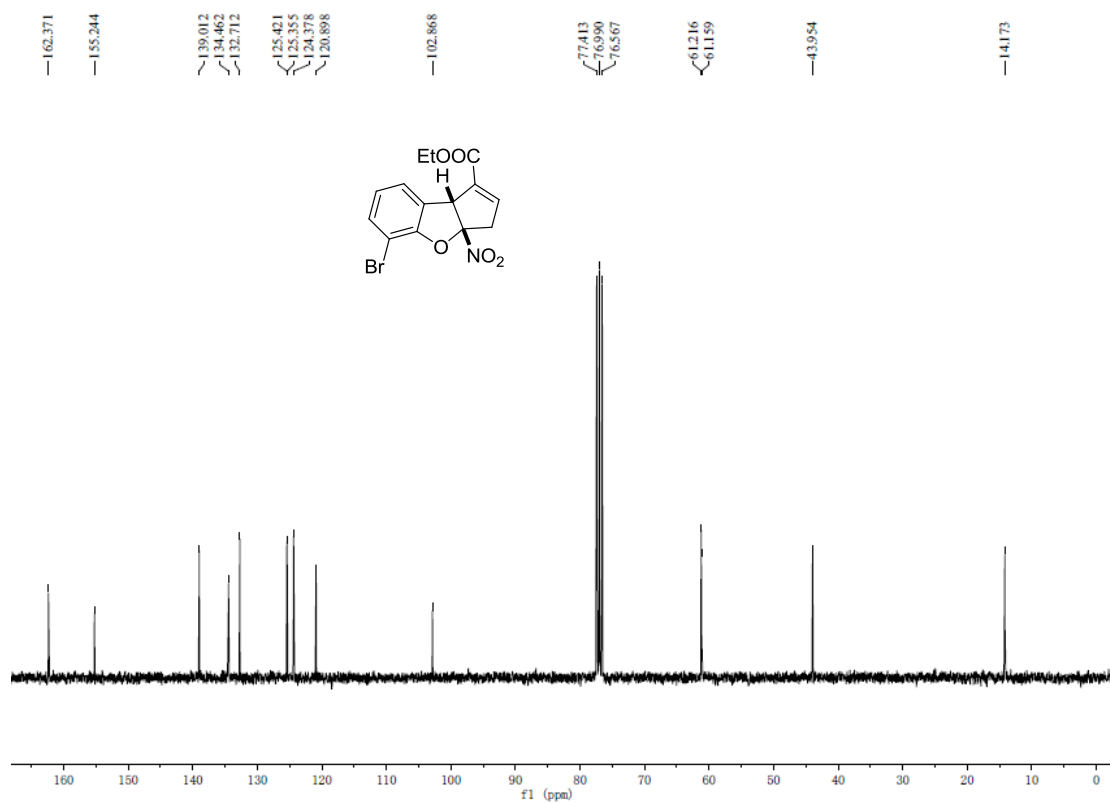
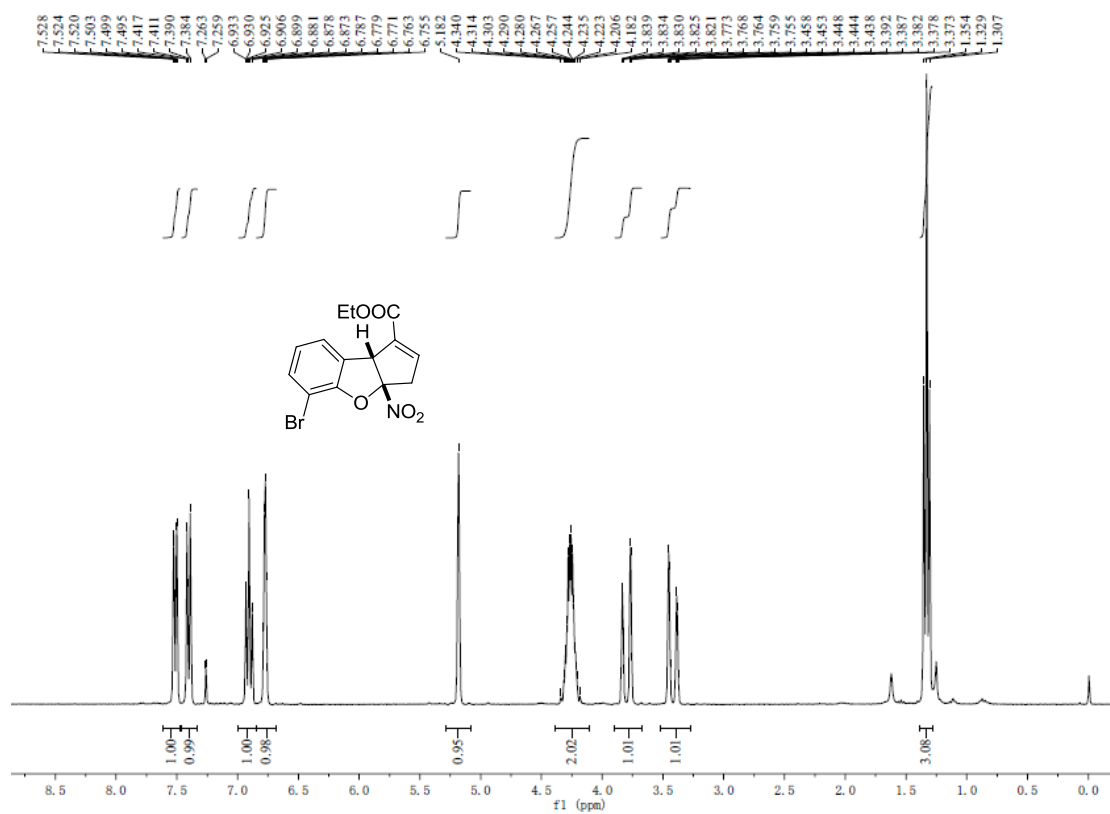
^1H and ^{13}C NMR of **31**



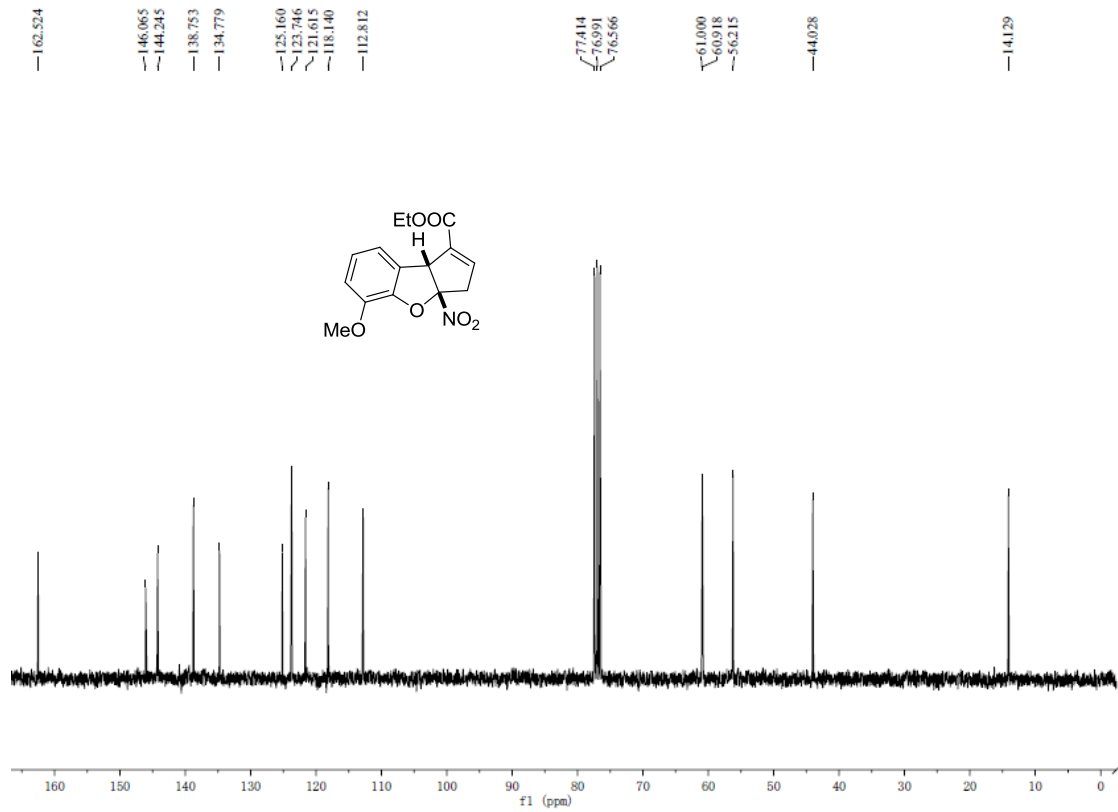
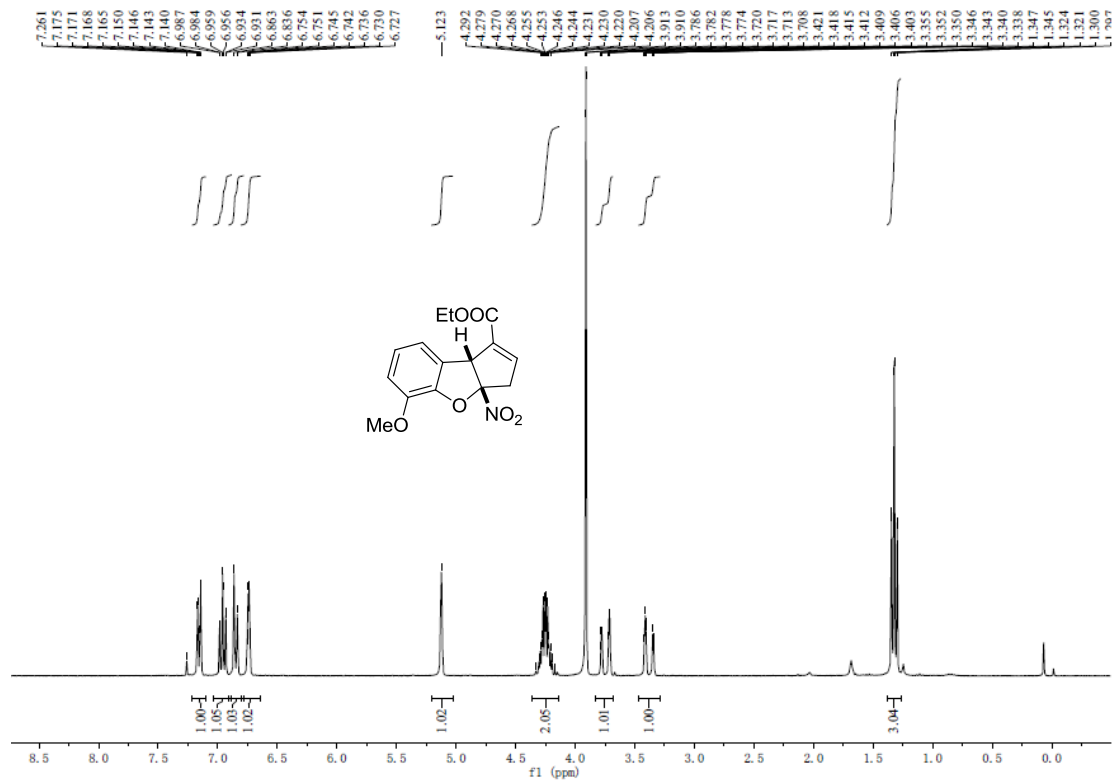
^1H and ^{13}C NMR of **3m**



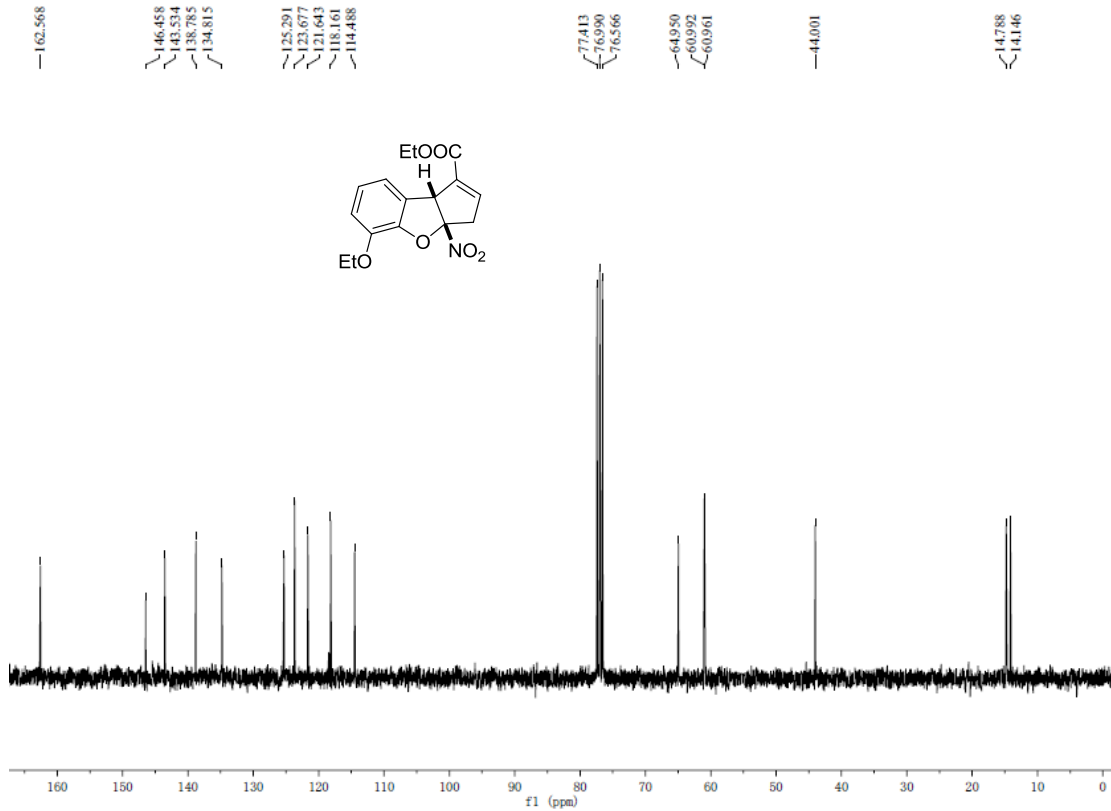
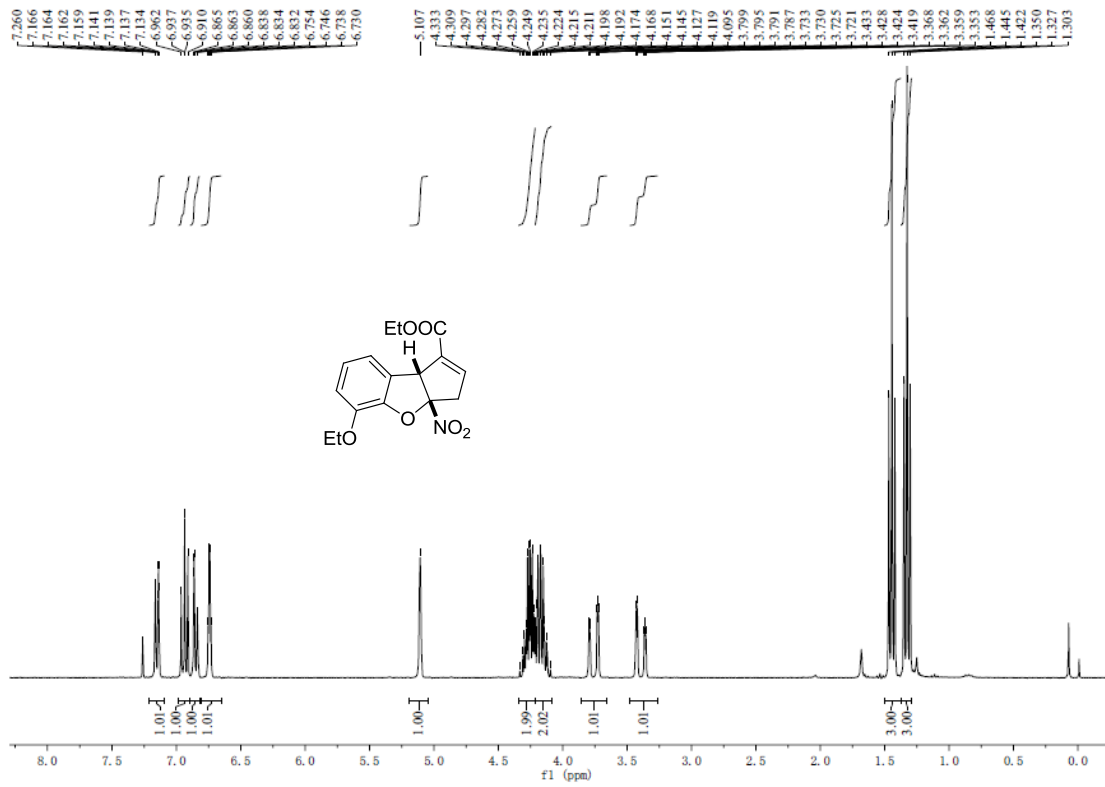
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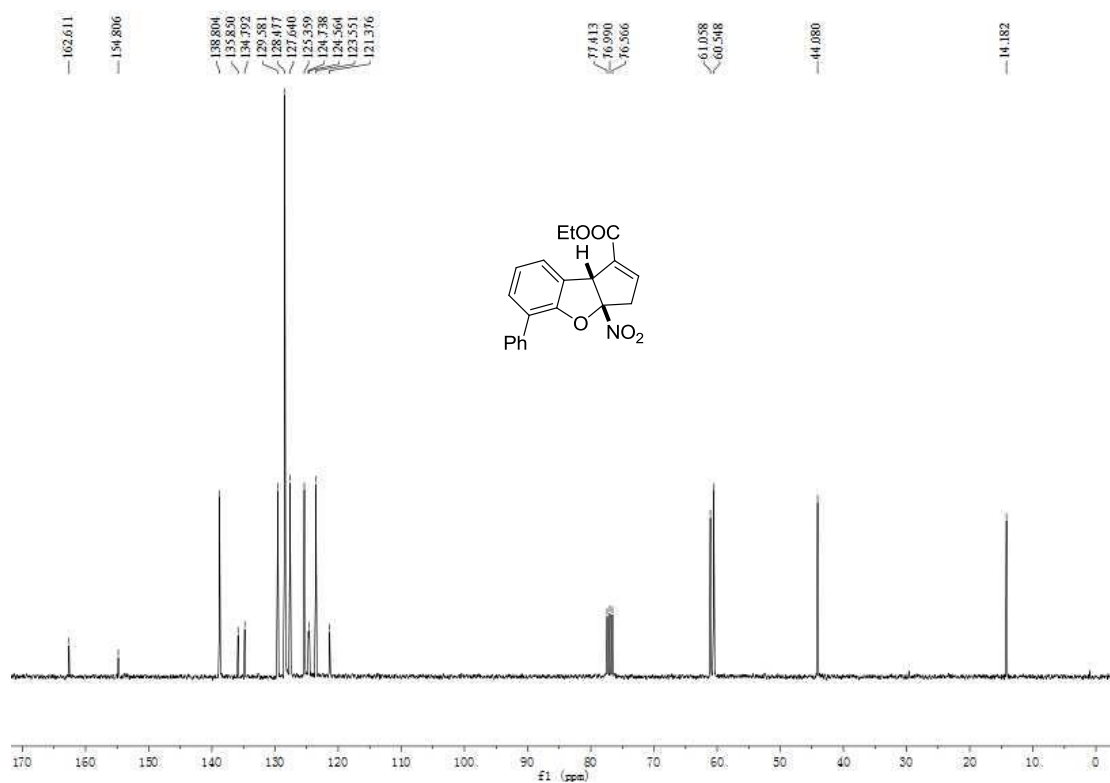
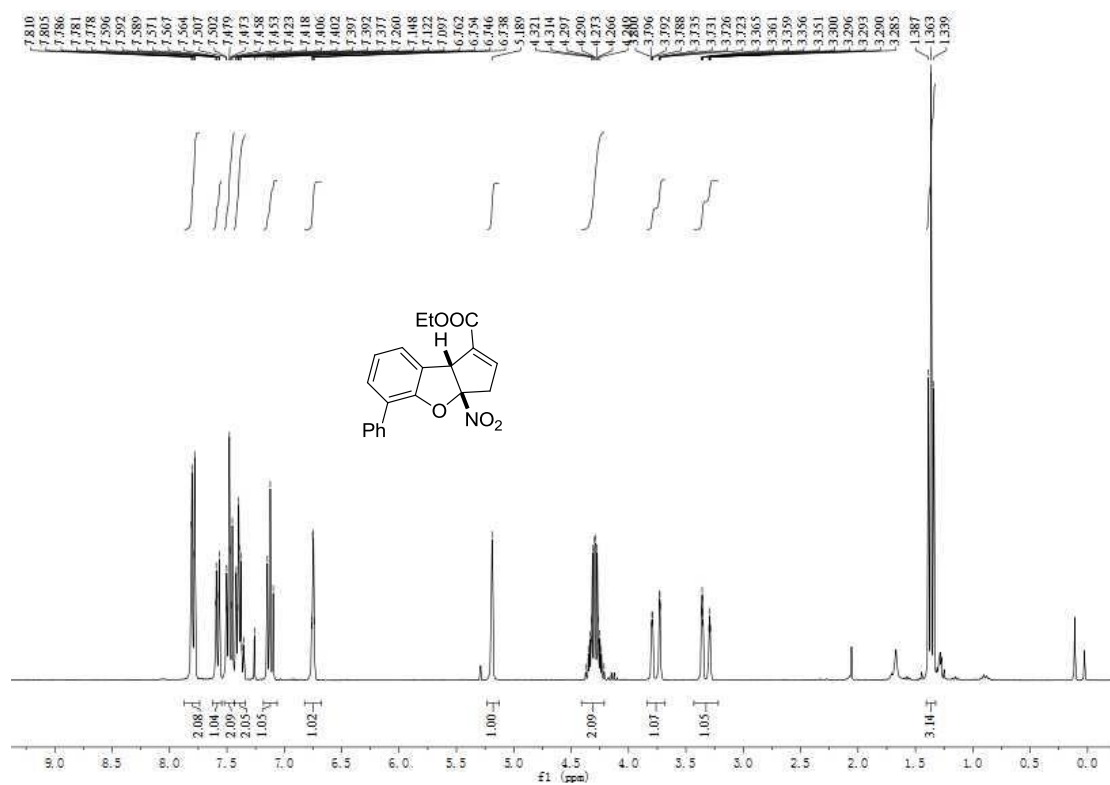
^1H and ^{13}C NMR of **30**



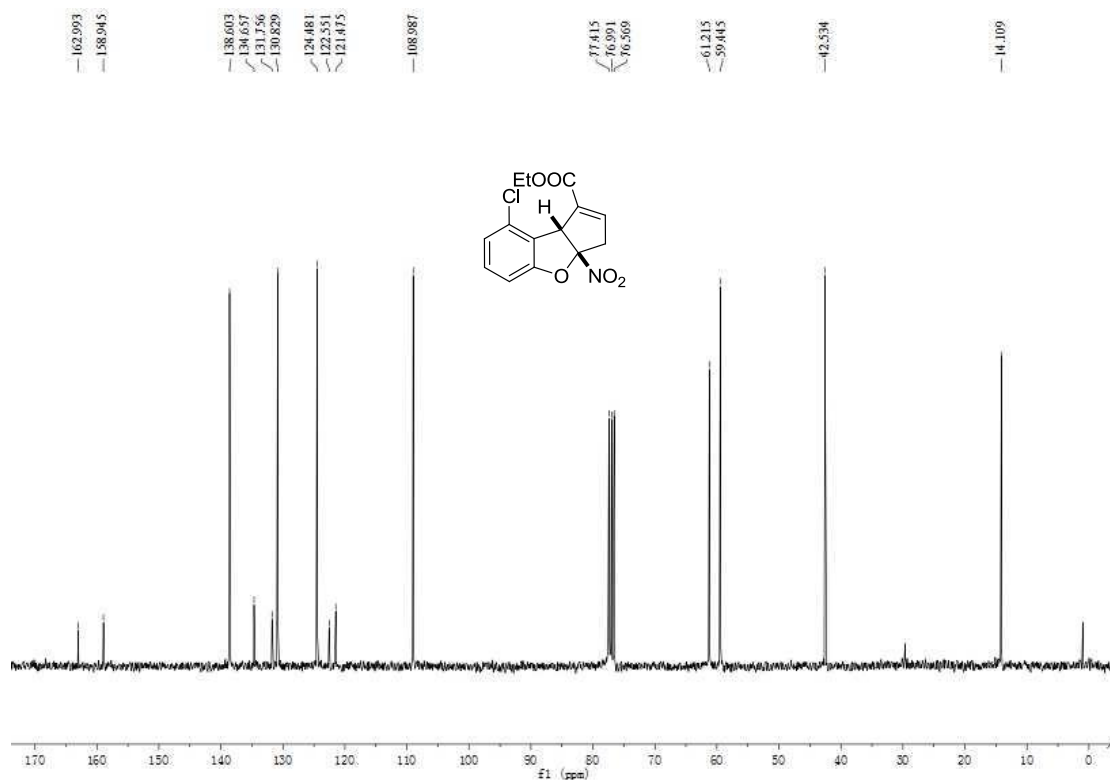
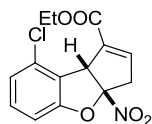
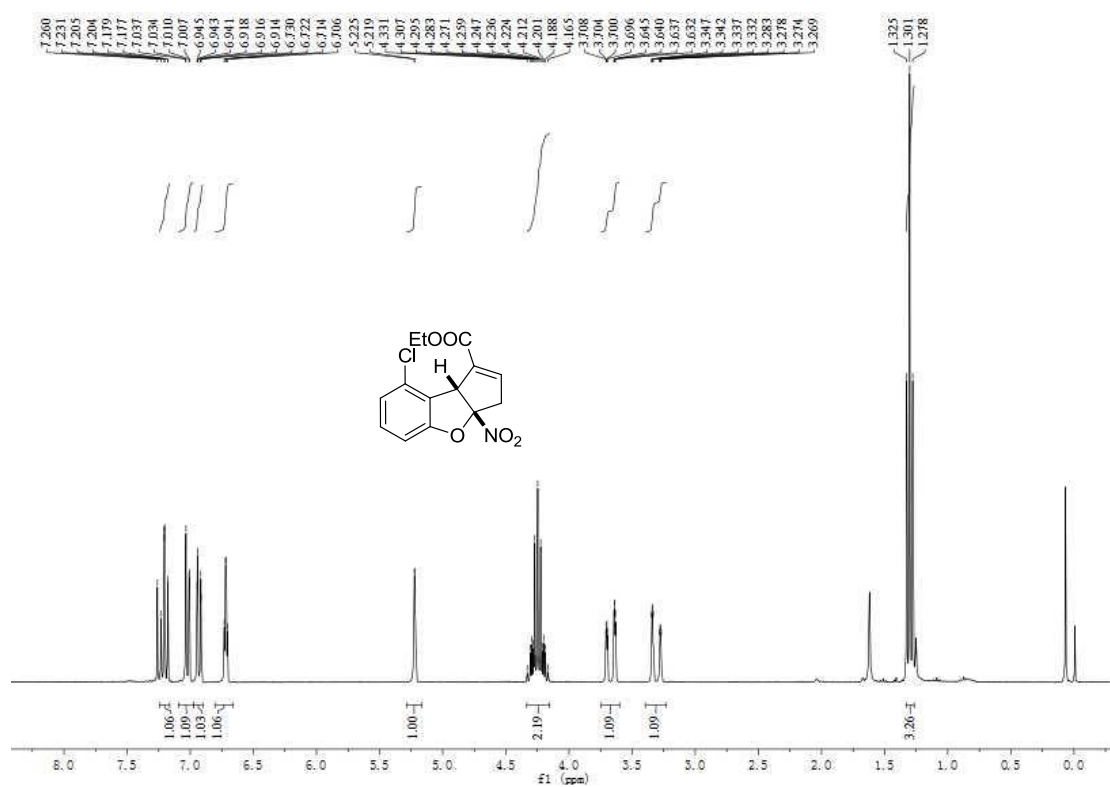
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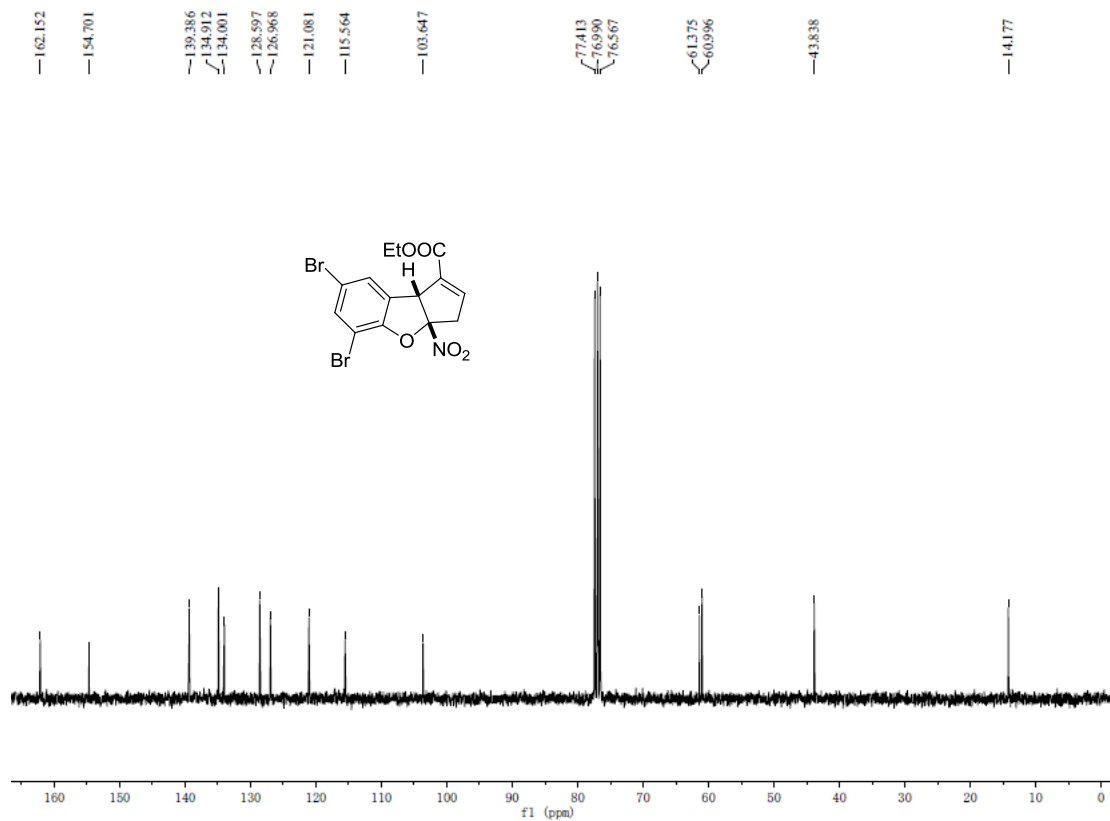
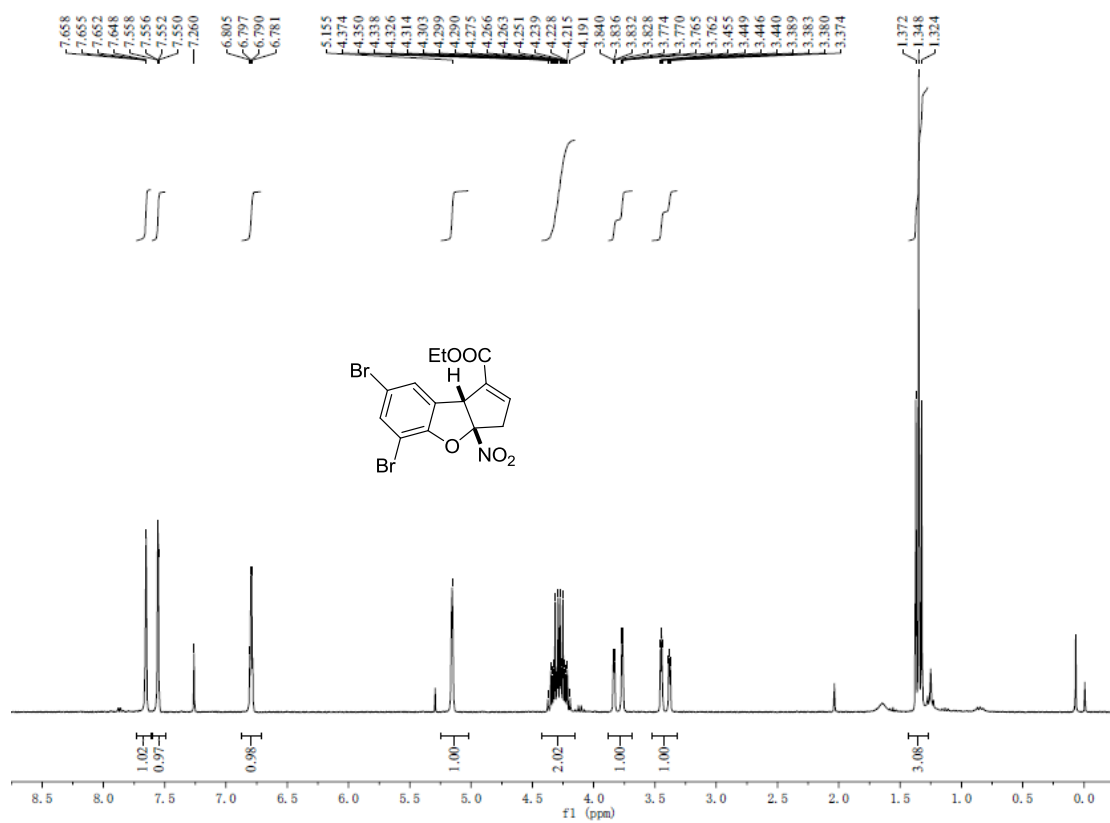
^1H and ^{13}C NMR of **3q**



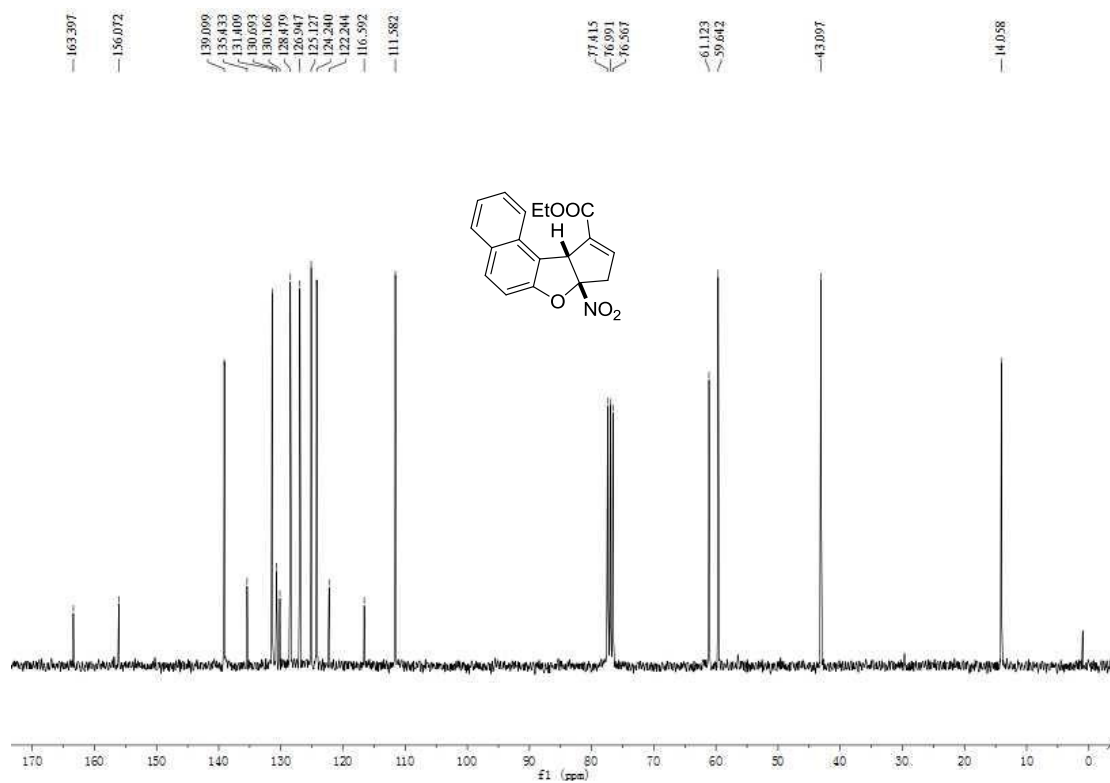
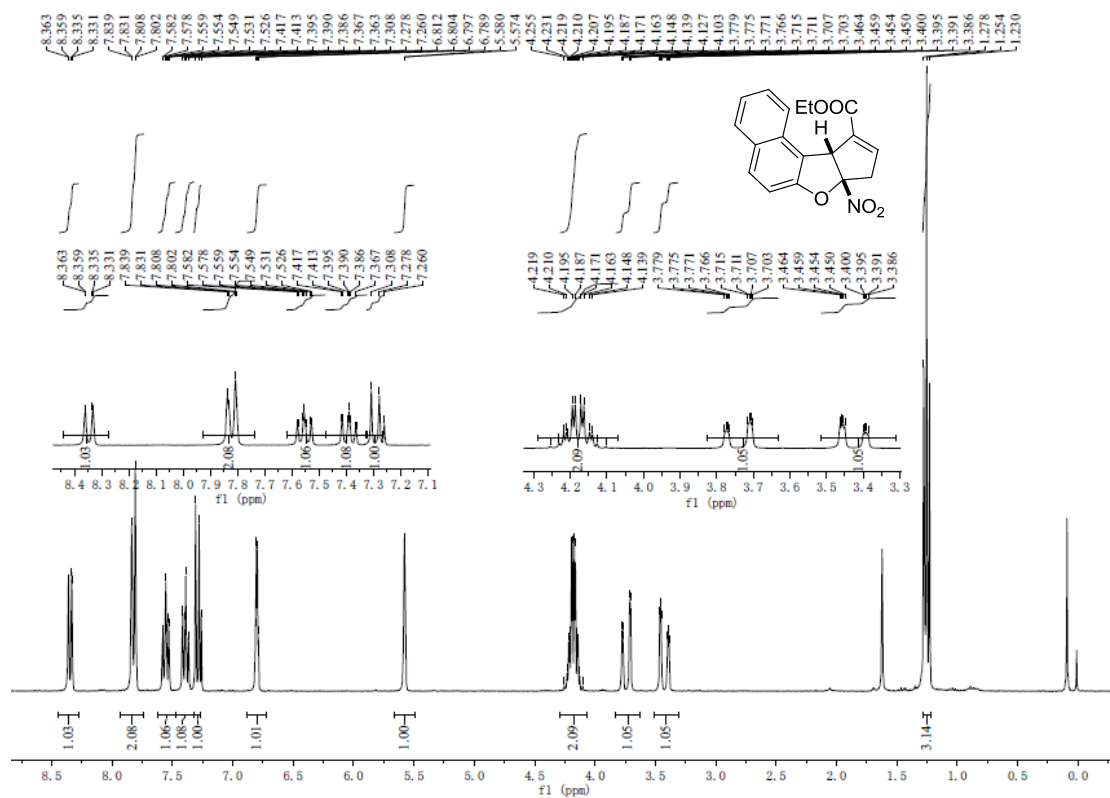
^1H and ^{13}C NMR of **3r**



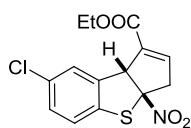
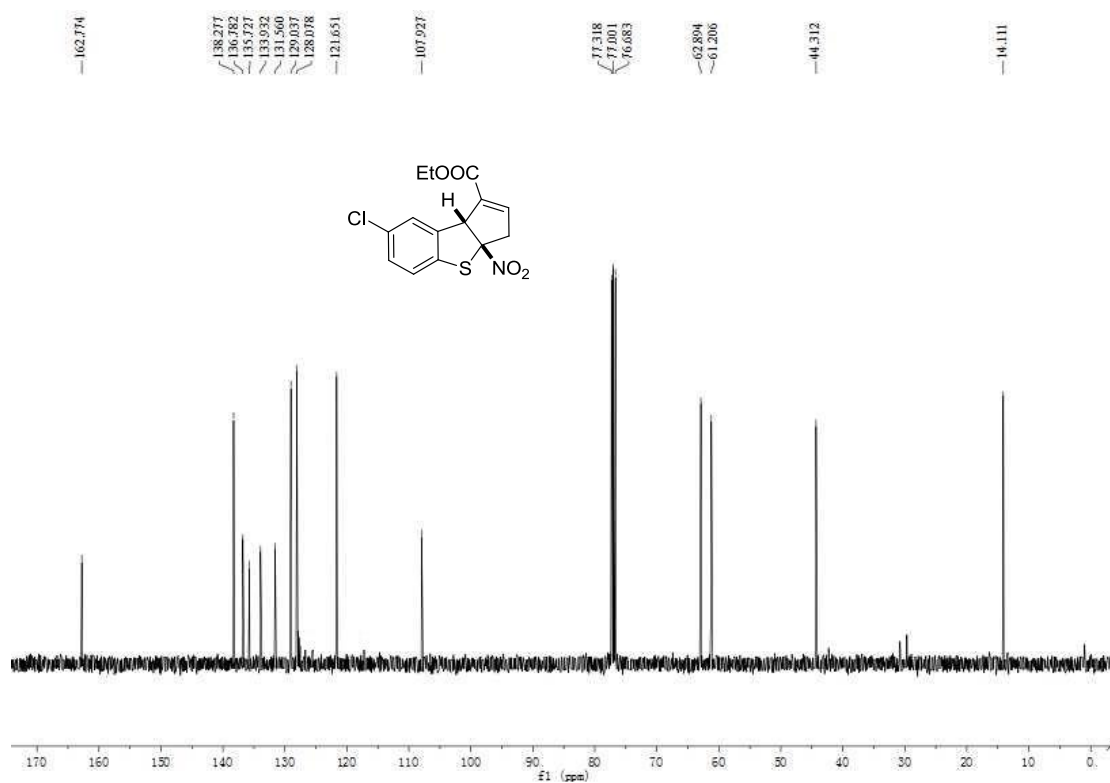
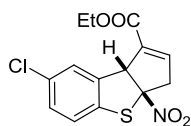
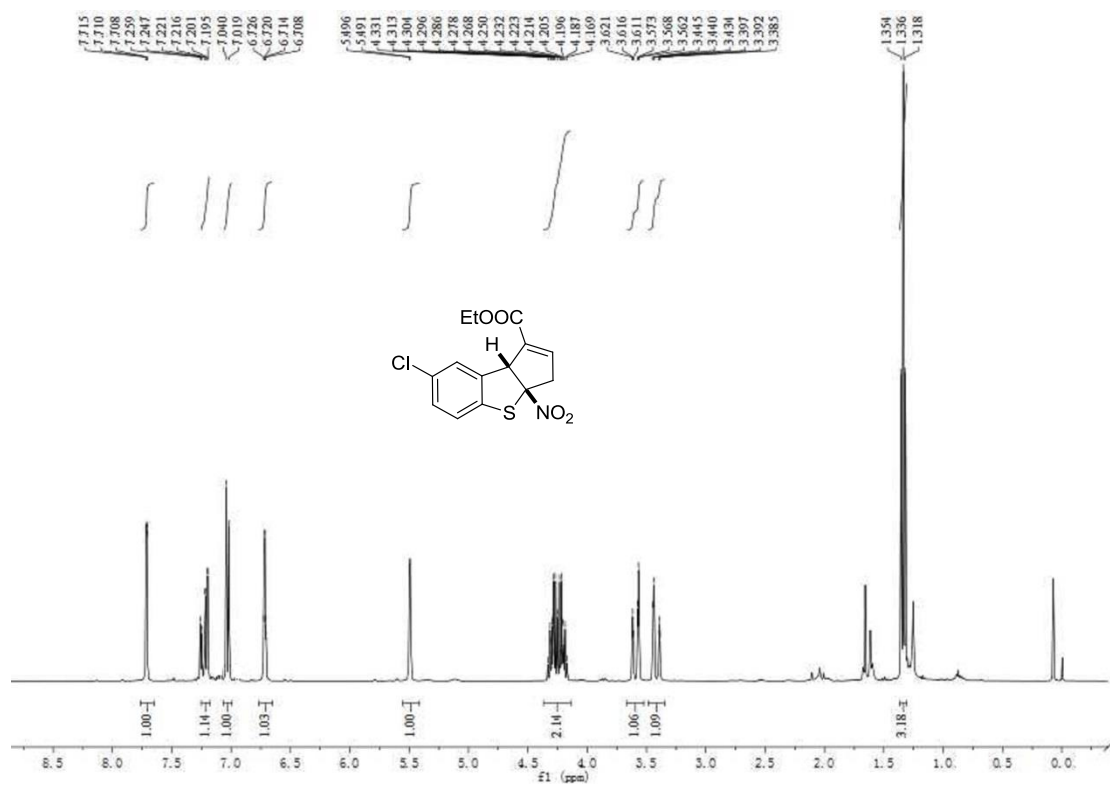
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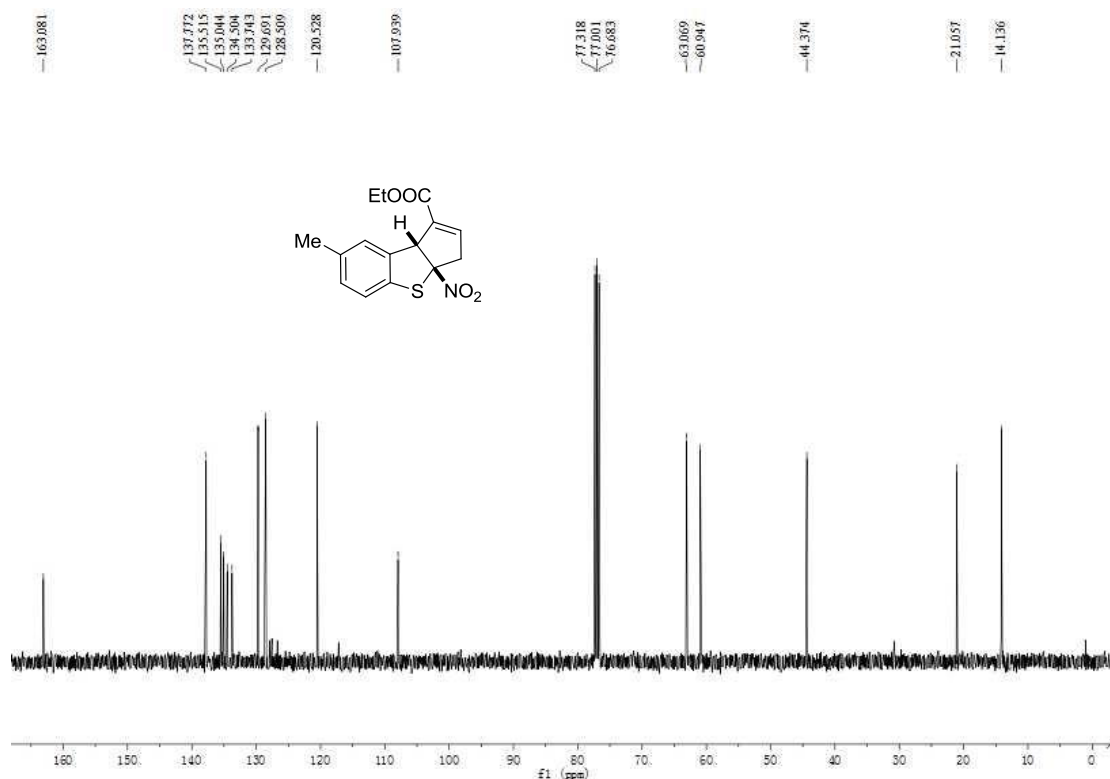
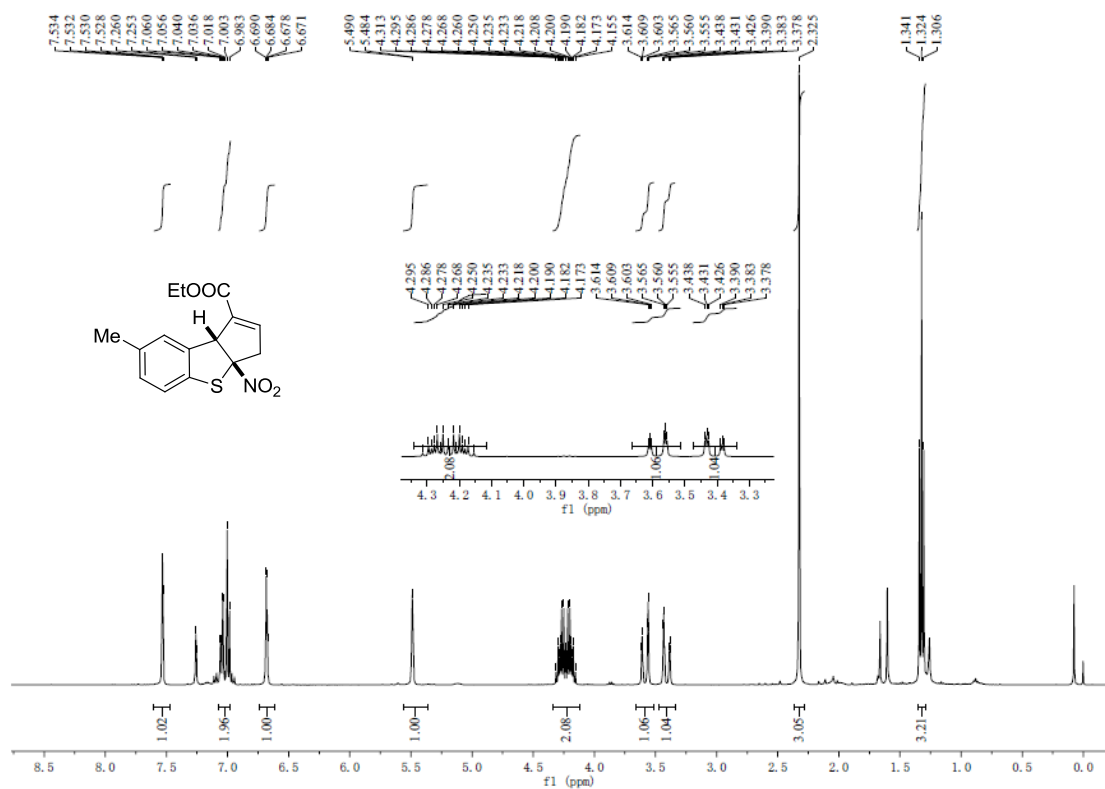
^1H and ^{13}C NMR of **3t**



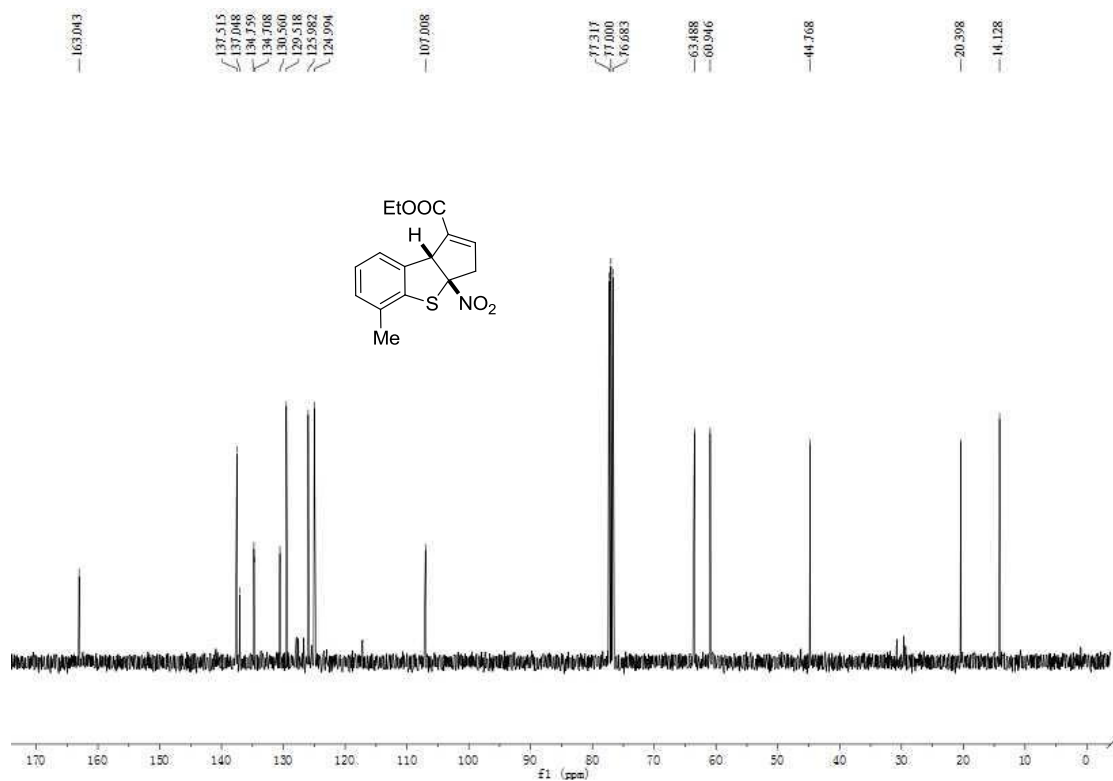
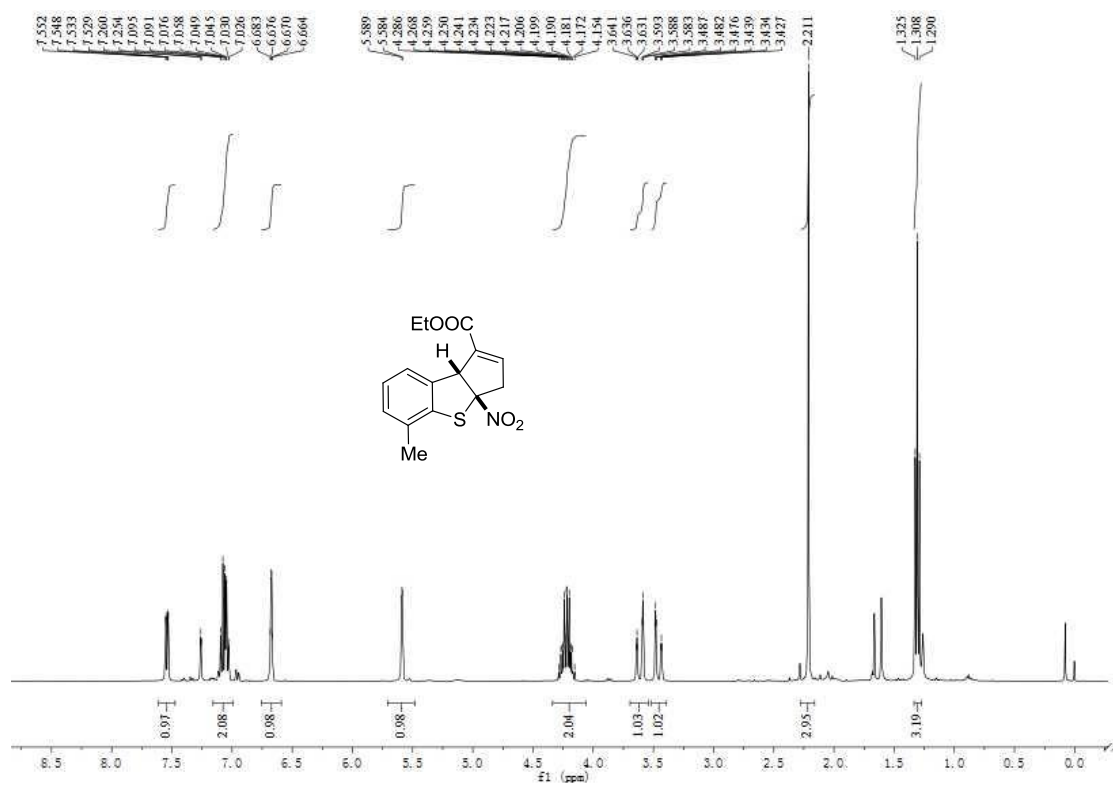
^1H and ^{13}C NMR of **5b**



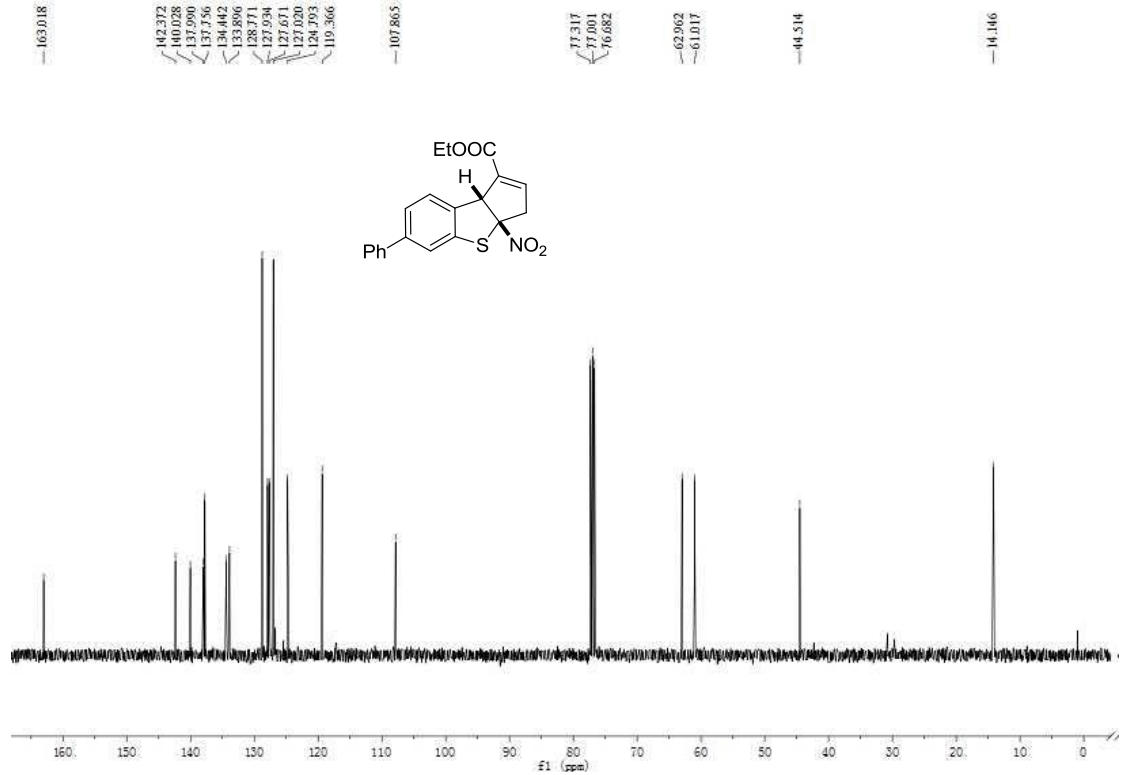
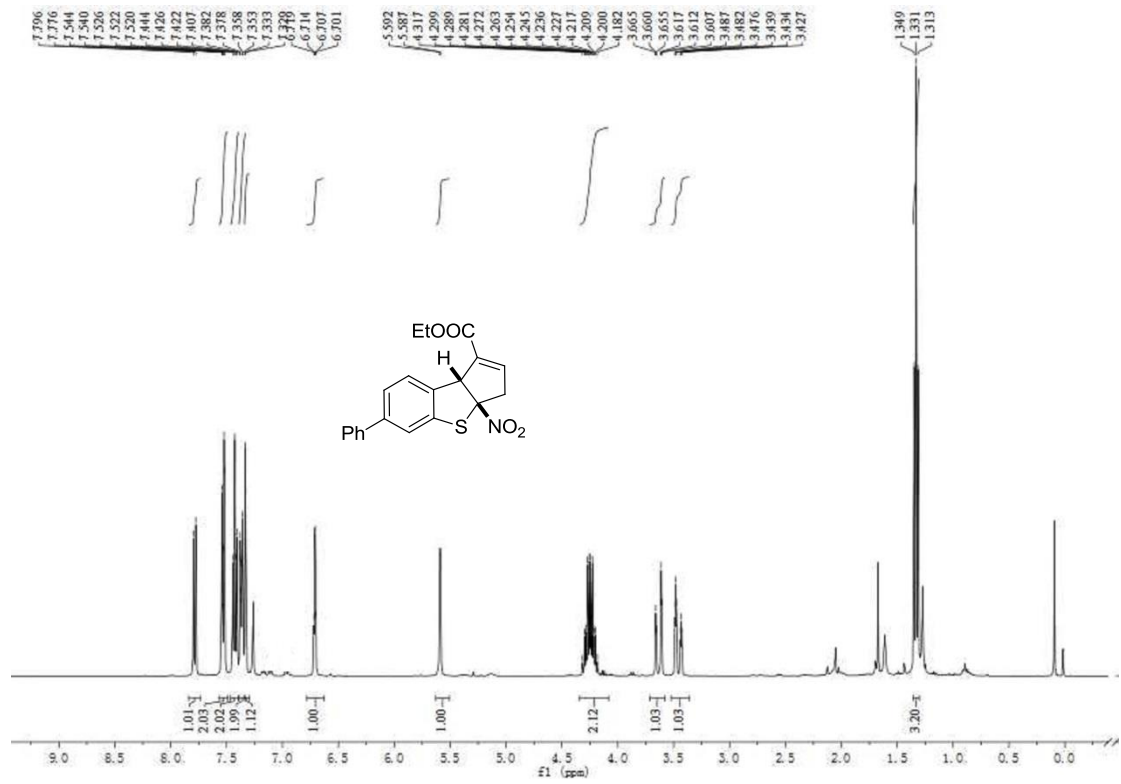
¹H and ¹³C NMR of 5c



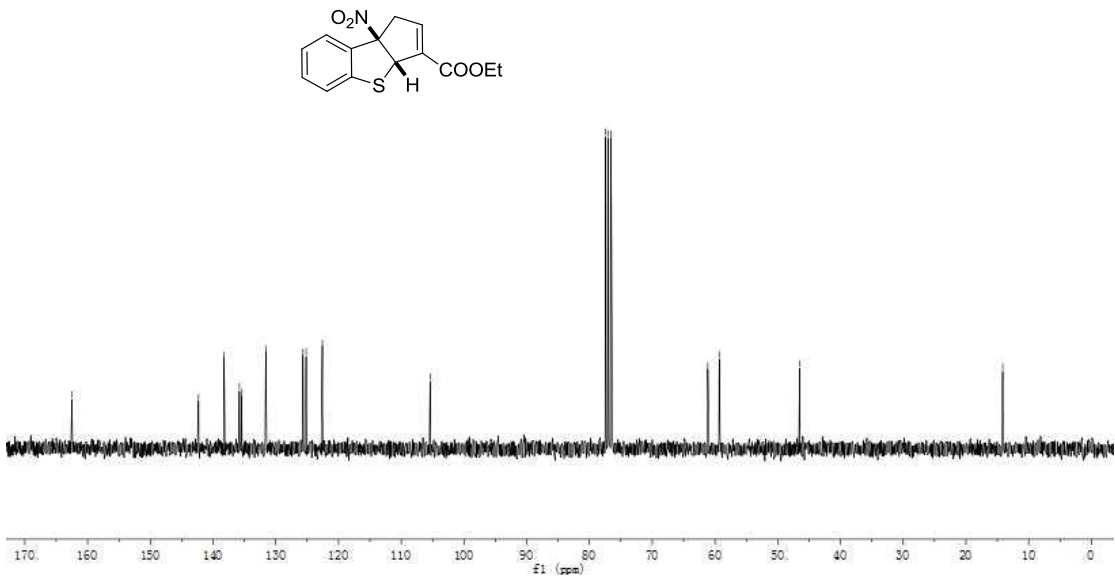
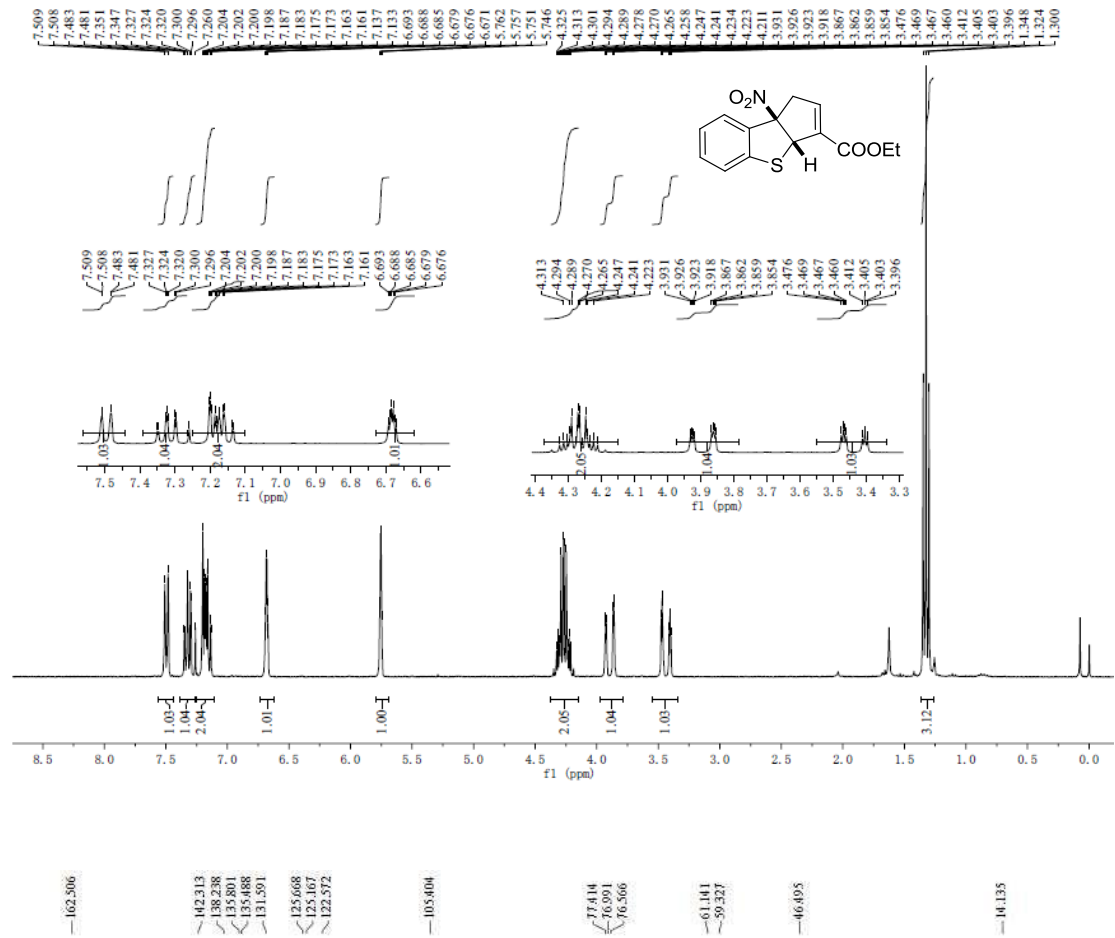
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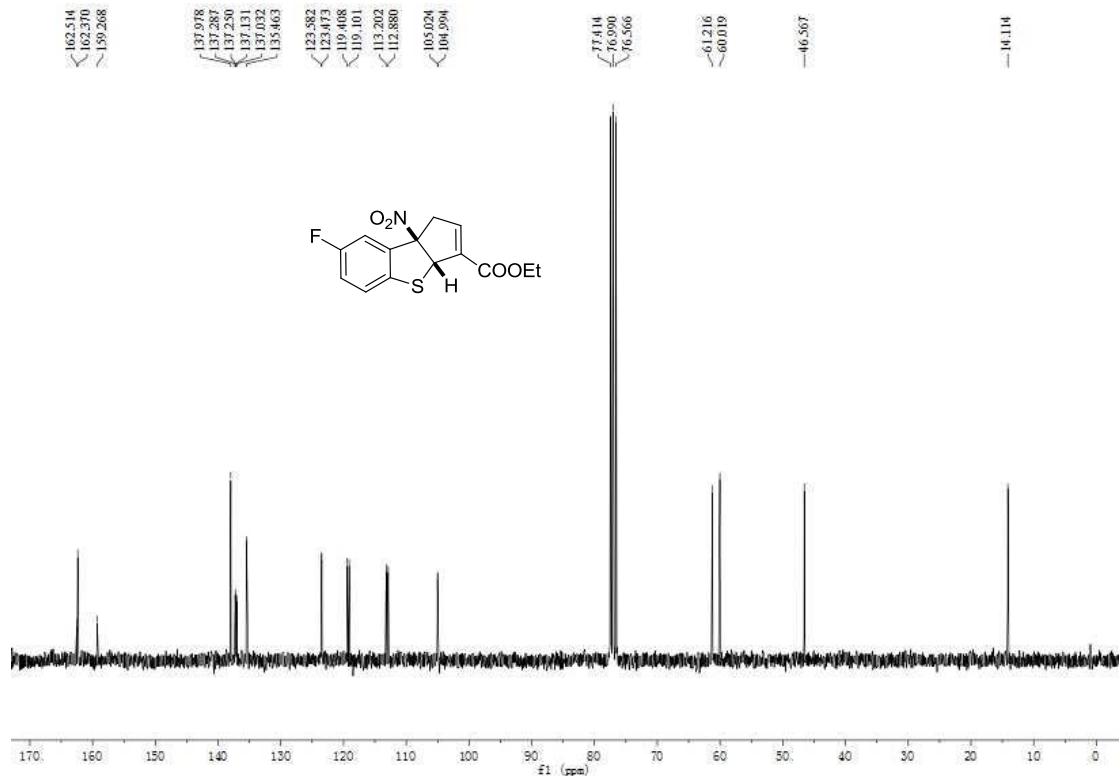
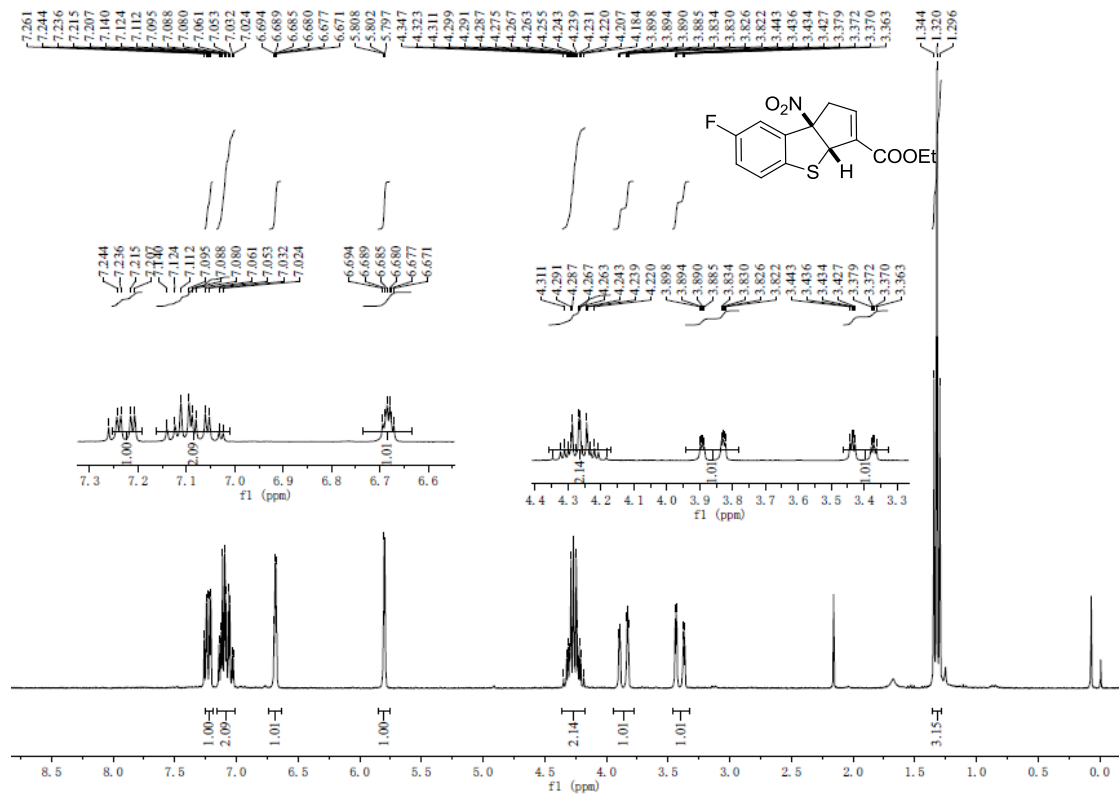
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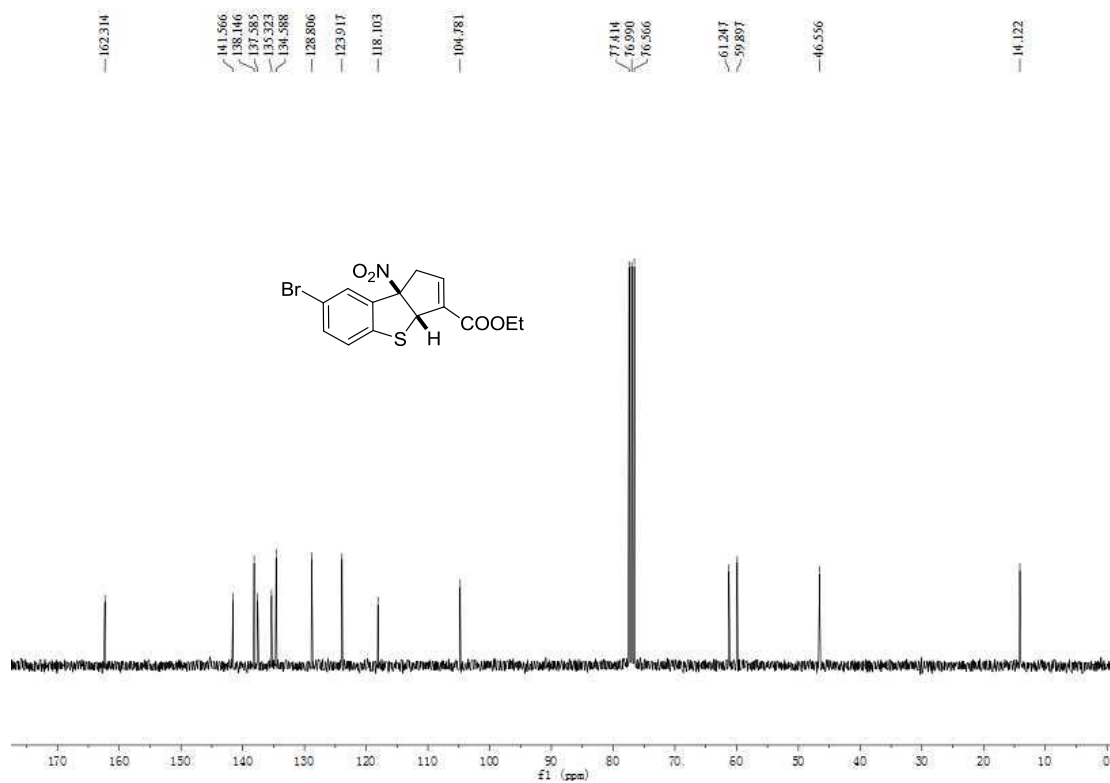
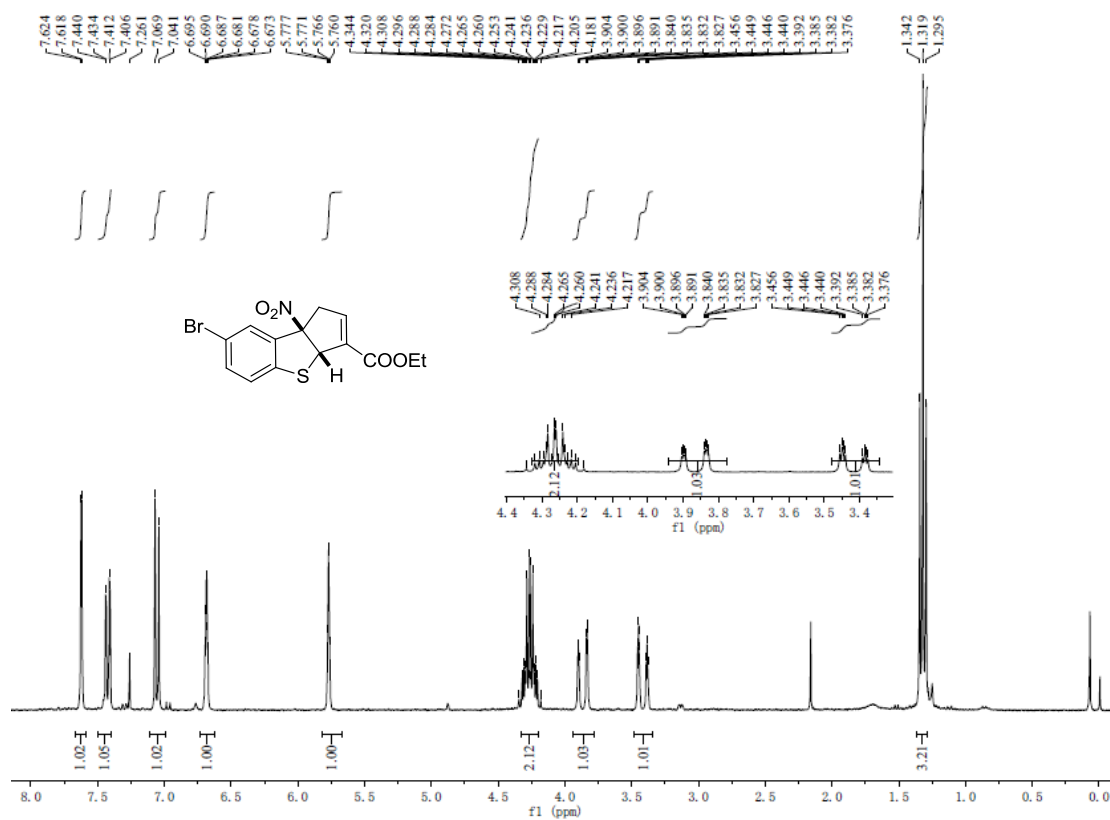
¹H and ¹³C NMR of **7a**



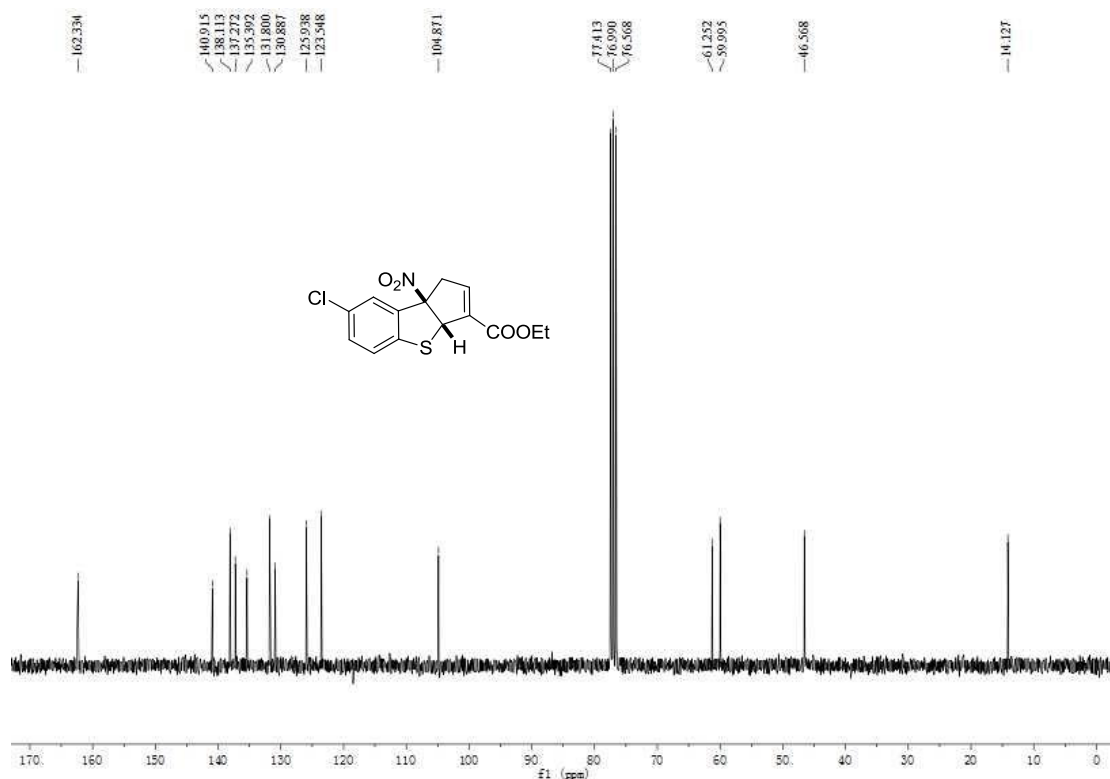
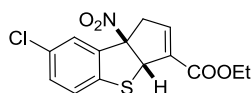
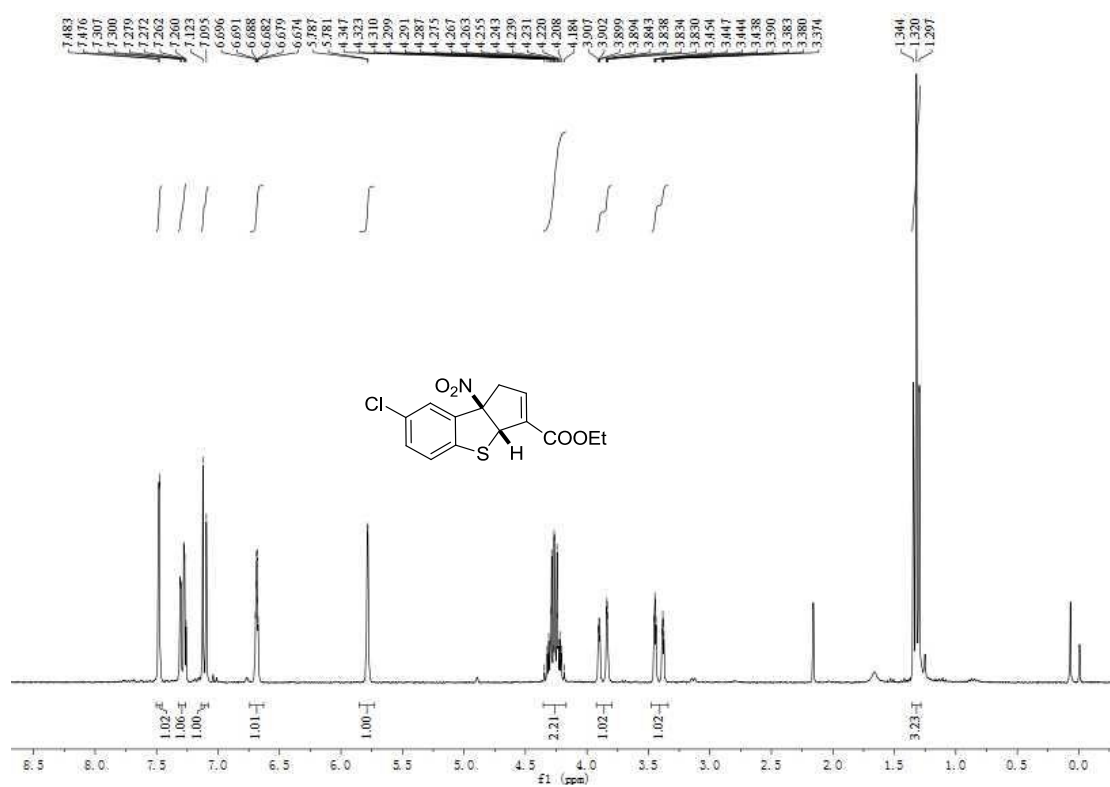
¹H and ¹³C NMR of **7b**



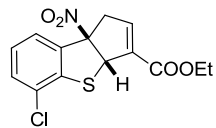
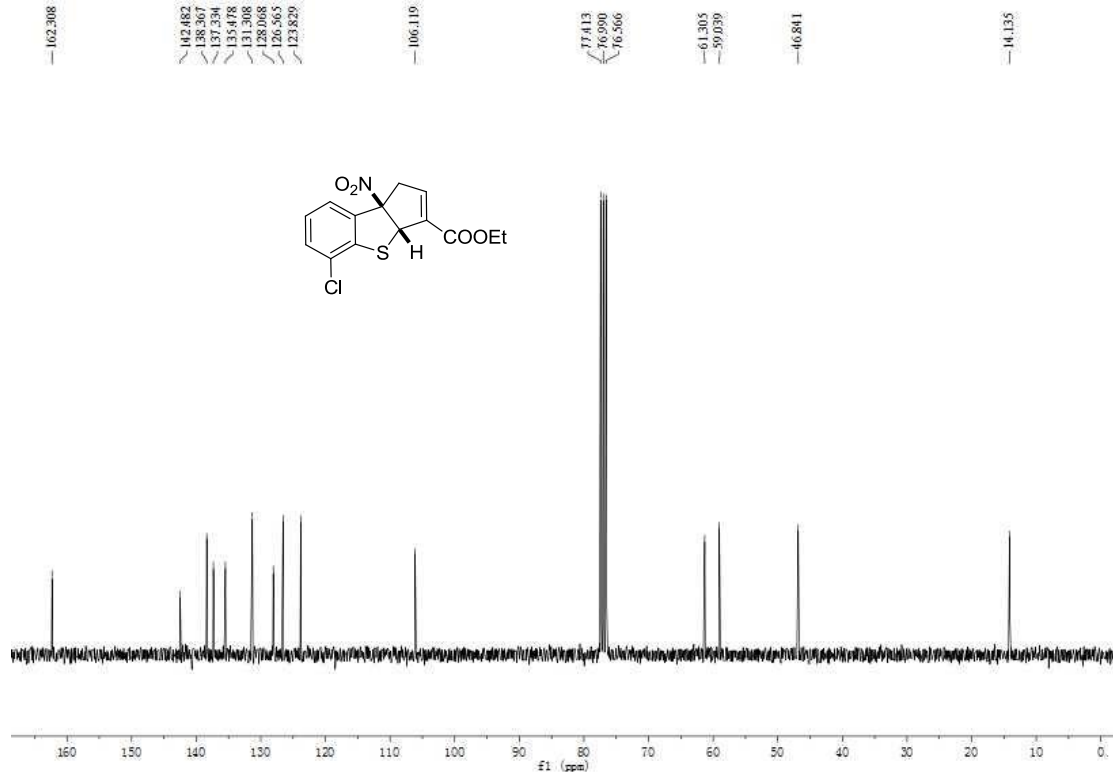
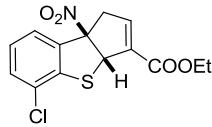
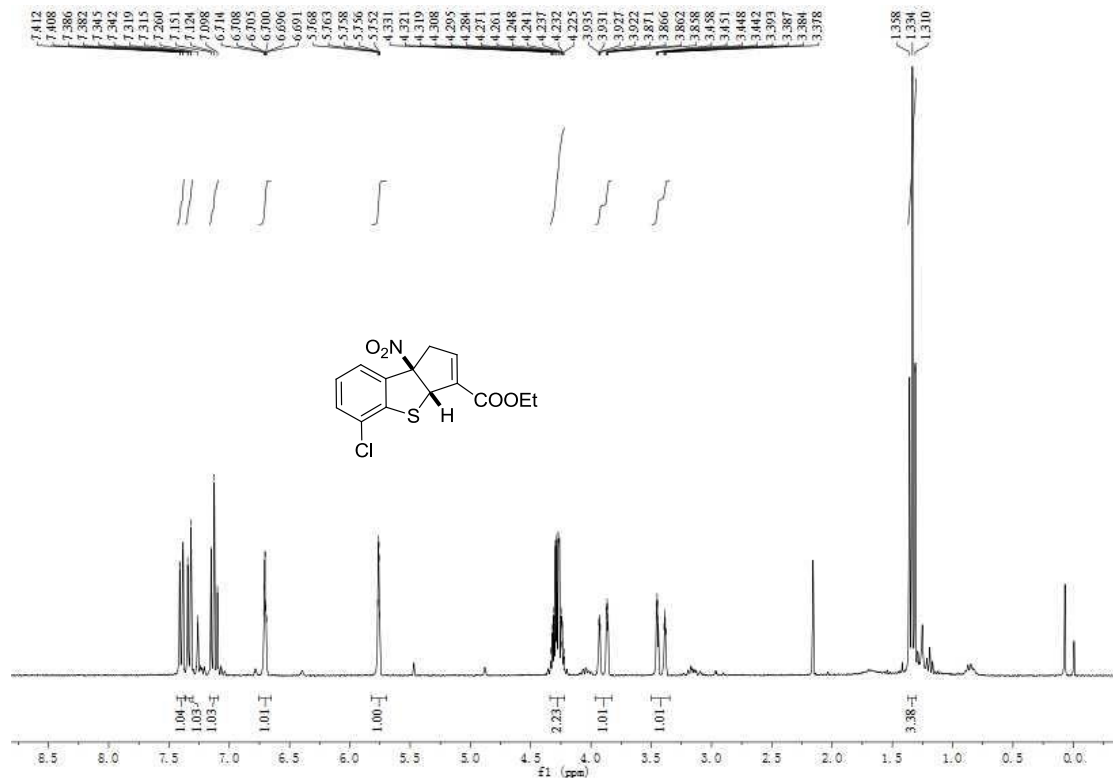
^1H and ^{13}C NMR of **7c**



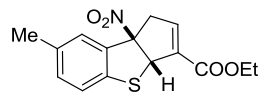
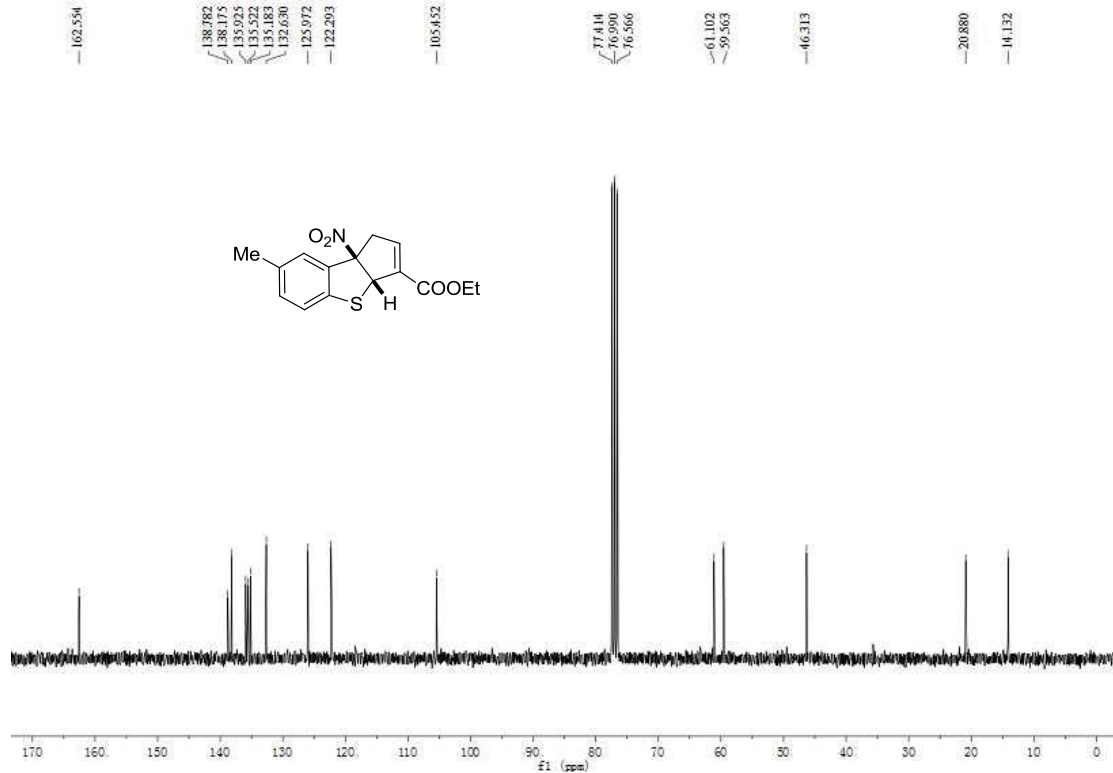
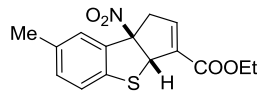
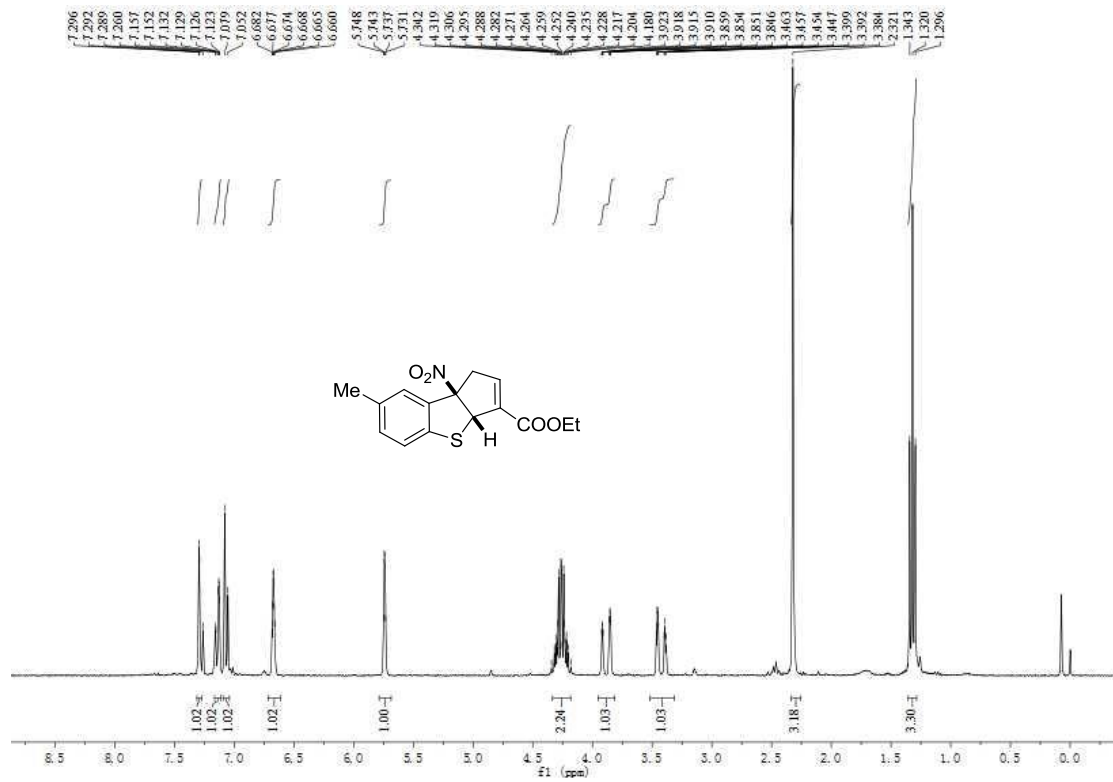
^1H and ^{13}C NMR of 7d



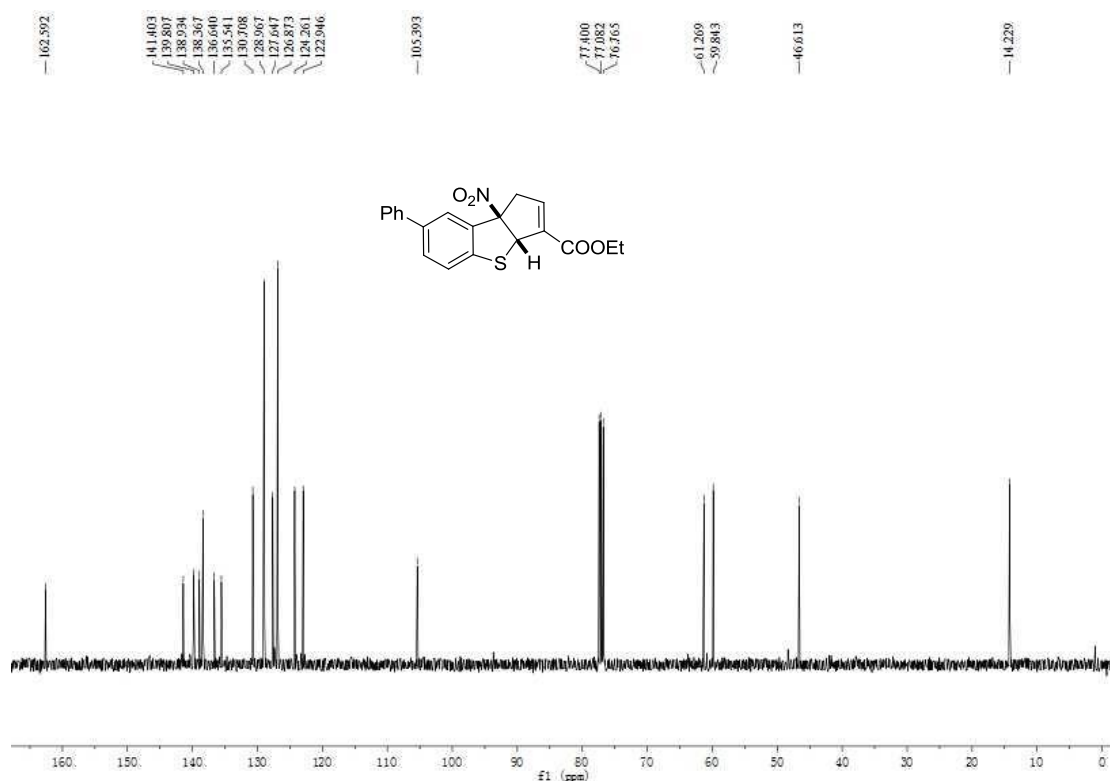
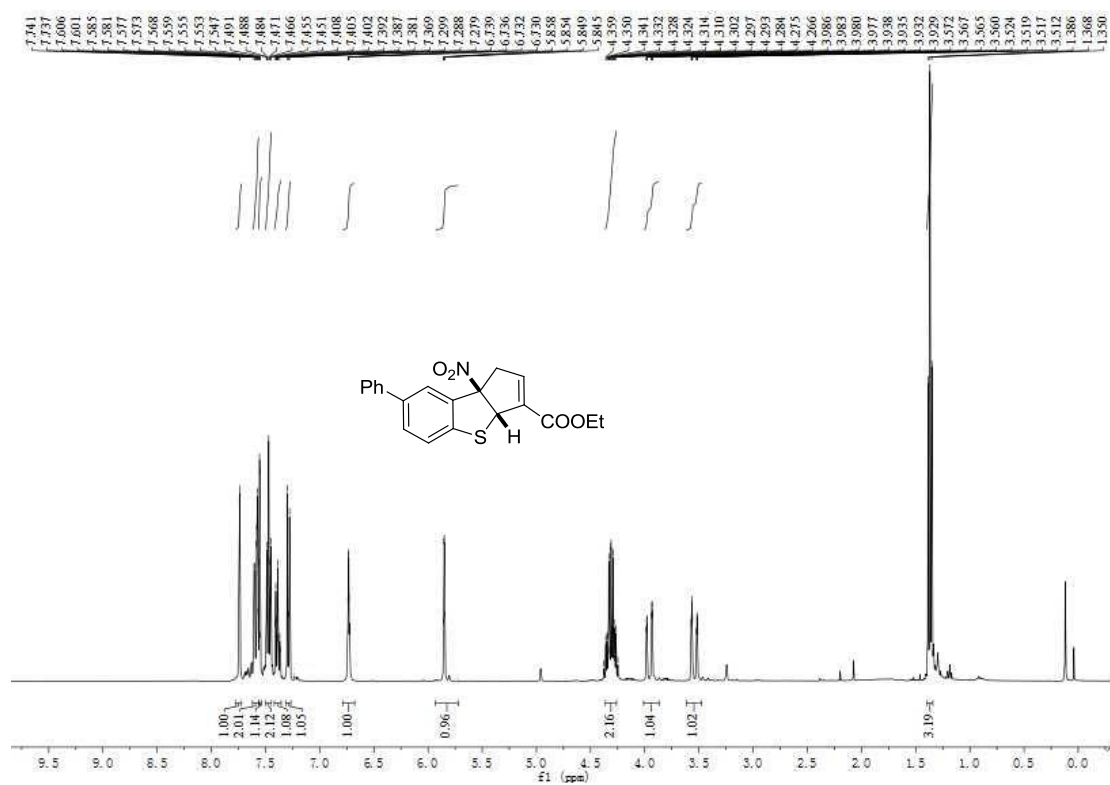
¹H and ¹³C NMR of 7e



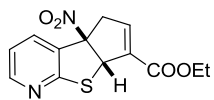
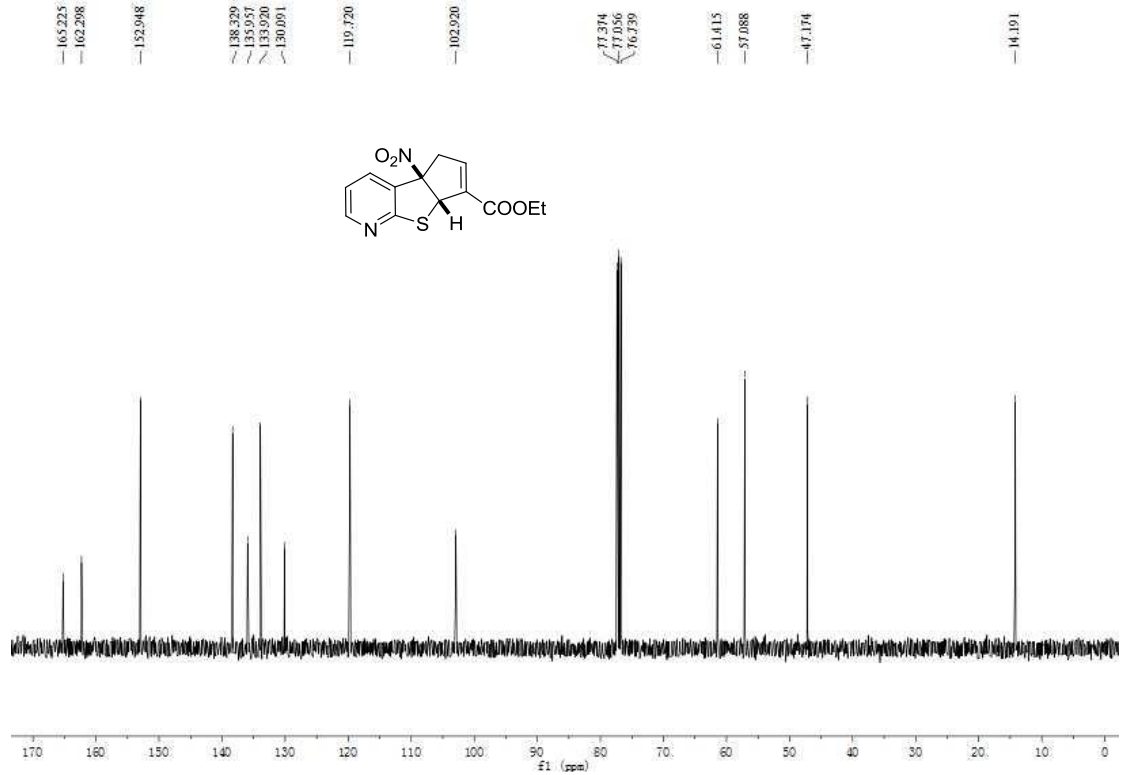
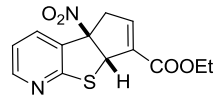
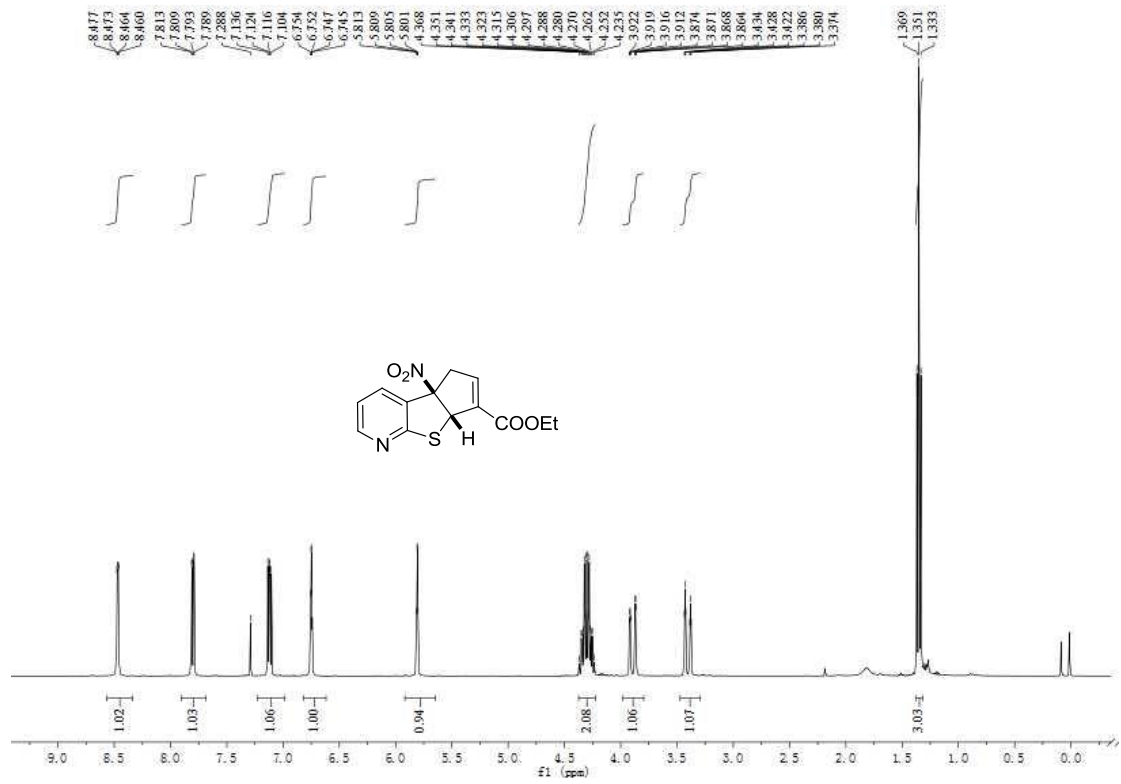
^1H and ^{13}C NMR of **7f**



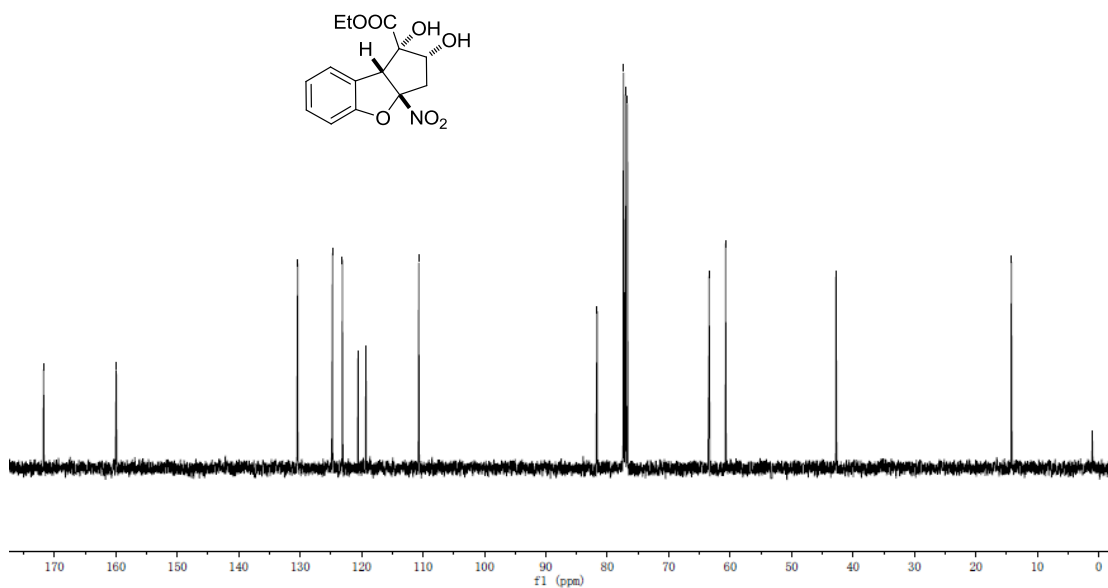
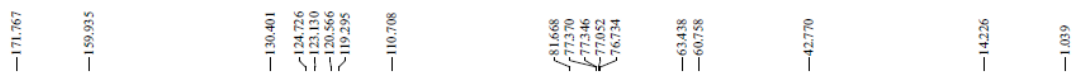
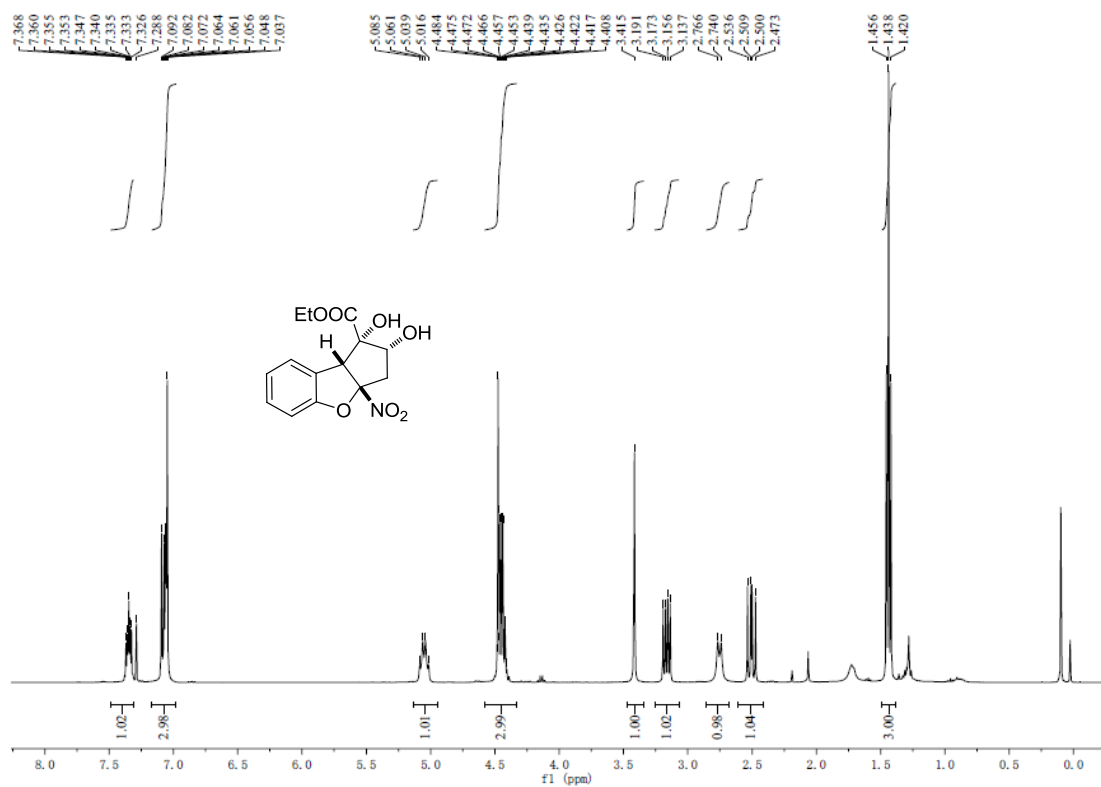
^1H and ^{13}C NMR of **7g**



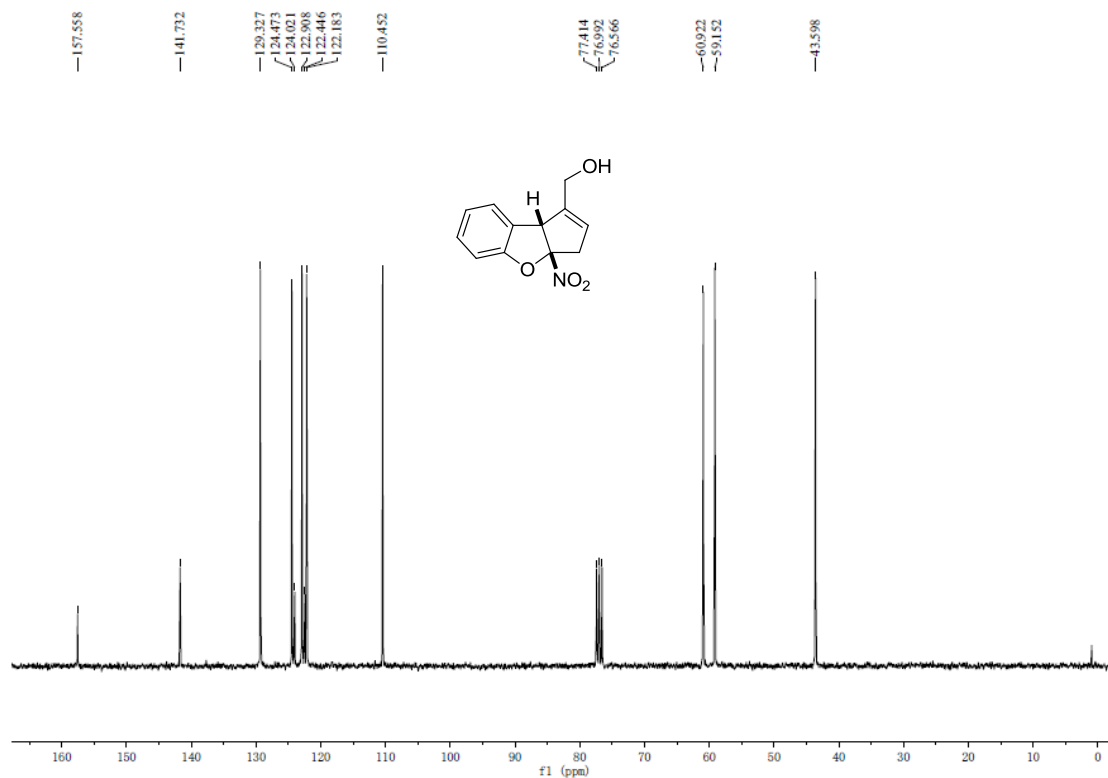
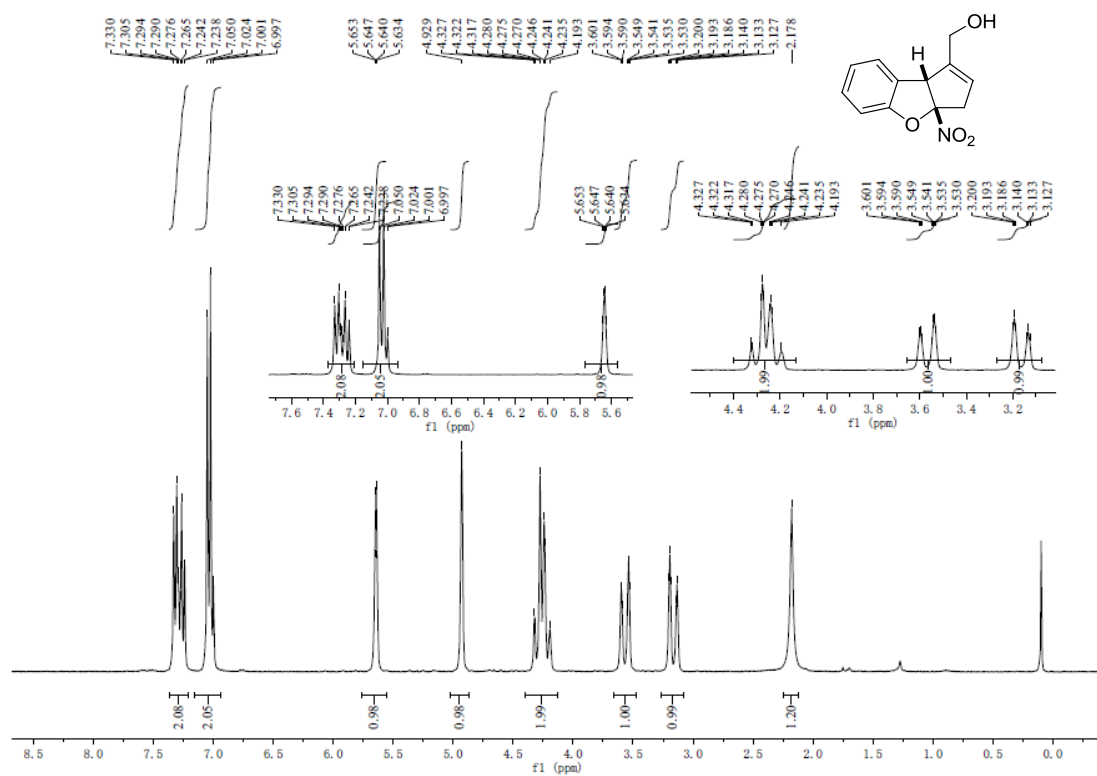
¹H and ¹³C NMR of 7h



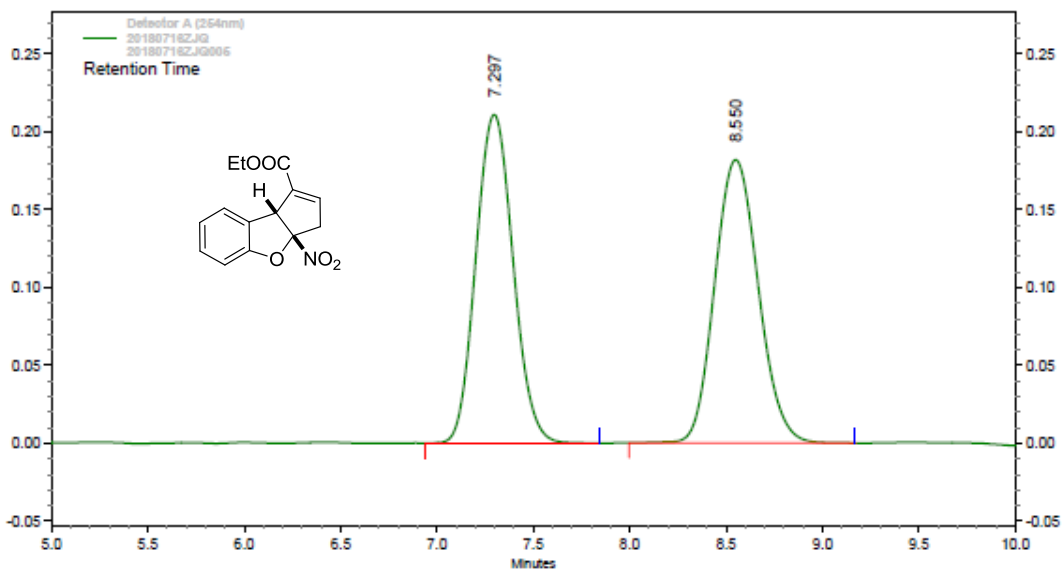
¹H and ¹³C NMR of 8



¹H and ¹³C NMR of 9



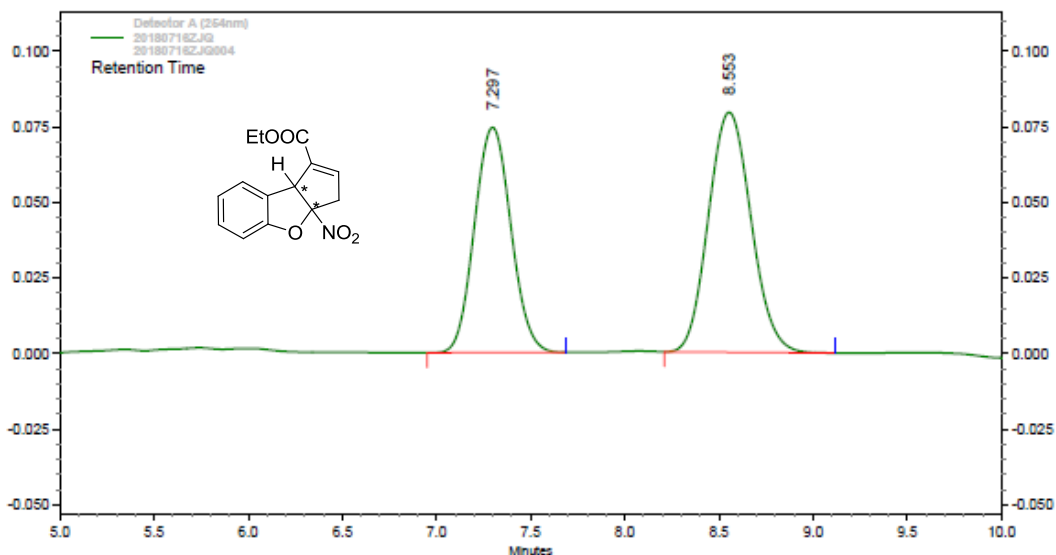
HPLC of 3a



Detector
A (254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	7.297	210877	53.71	2833387	49.76
2	8.550	181773	46.29	2860294	50.24

Totals		392650	100.00	5693681	100.00
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Detector
A (254nm)

Pk #	Retention Time	Height	Height Percent	Area	Area Percent
1	7.297	74516	48.41	1005196	44.51
2	8.553	79407	51.59	1253011	55.49

Totals		153923	100.00	2258207	100.00
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