

Supplementary Information
For

Efficient Functionalization of Organosulfones via Photoredox Catalysis: Direct Incorporation of α -Carbonyl Alkyl Side Chains into α -Allyl- β -Ketosulfones

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1. General information.

Unless otherwise noted, all reactions were performed under a nitrogen atmosphere and were monitored using TLC and visualized using a UV lamp (254 nm) /or via treatment with a solution of 10 g phosphomolybdic acid and 100 mL EtOH followed by heating. All reagents were used as received from commercial sources without further purification. Silica gel (200-300 mesh) and silica gel GF₂₅₄ (10-40 μ m) were used for column chromatography (CC) and prepare thin layer chromatography (PTLC), respectively. Solvents were dried and purified according to the procedure from "Purification of Laboratory Chemicals book". ¹H NMR and ¹³C NMR spectra were recorded in CDCl₃ on a Varian 500 MHz instrument. Chemical shifts were denoted in ppm (δ) and calibrated using residual undeuterated solvent (CDCl₃ (7.26 ppm), or tetramethylsilane (0.00 ppm)) as internal reference for ¹H NMR and the deuterated solvent (CDCl₃ (77.00 ppm), or tetramethylsilane (0.00 ppm)) as internal standard for ¹³C NMR. The following abbreviations were used to denote the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, br = broad, td = triple doublet, dt = double triplet, m = multiplet. The MS data were obtained with ESI technique, and the relative intensity (%) is given in brackets. High-resolution mass spectral analysis (HRMS) data were measured using a Bruker ApexII mass spectrometer by means of the ESI technique.

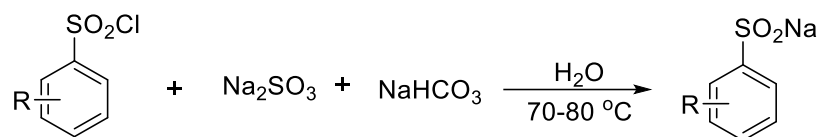
2. Experimental procedures

2.1 Synthesis of starting materials

General Procedure for the Preparation of Sodium Sulfinates¹:

Benzenesulfinic acid sodium salt (1.0 eq.) was prepared by heating sodium sulfite (1.26 g, 10 mmol, 2.0 eq.), benzenesulphonyl chloride (5 mmol, 1.0 eq.), and sodium bicarbonate (0.84 g, 10 mmol, 2.0 eq.) in 10 mL of water at 70-80 °C for 5 h. After cooling to room temperature, water was removed under vacuum and the residue was extracted by ethanol, recrystallization as a white solid, the yield was 60-70%.

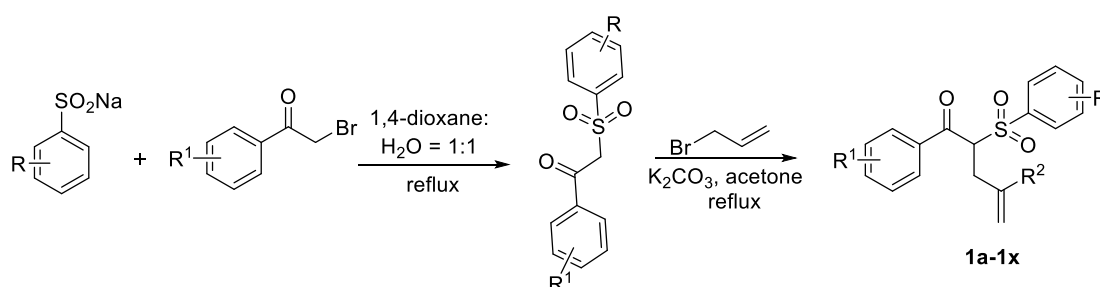
Similarly, other sodium arenesulfonates were prepared from their corresponding sulfonyl chlorides.



General Procedure for the Preparation of substrates **1a-1x**^{2,3}

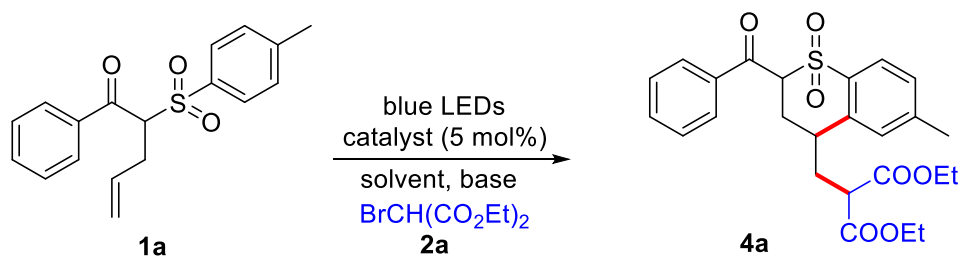
Sodium sulfonates (RSO_2Na , 3.6 mmol, 1.2 eq.) was added to a solution of substituted 2-bromoacetophenones (3.0 mmol, 1.0 eq.) in a cosolvent of 1,4-dioxane and water (20 mL, v/v = 1:1) at rt. The reaction mixture was stirred at reflux for 6 h. After the reaction was completed, the reaction mixture was extracted with CH_2Cl_2 (3×20 mL). The organic layer was combined, dried (MgSO_4), filtered, and evaporated to obtain crude β -ketosulfones² under reduced pressure. Crude β -ketosulfones were recrystallized from petroleum ether/EtOAc in nearly quantitative yields.

K_2CO_3 (0.621 g, 4.5 mmol, 1.5 eq.) was added to a solution of β -ketosulfones (3.0 mmol, 1.0 eq.) in acetone (30 mL) at rt. The reaction mixture was stirred at rt for 10 min. Allyl halides (3.3 mmol, 1.1 eq.) were added to the reaction mixture at rt. The reaction mixture was stirred at reflux for 14 h. After the reaction was completed, the reaction mixture was extracted with EtOAc ($30\text{ mL} \times 3$). The organic layer was combined, dried (MgSO_4), filtered, and concentrated in vacuo. The resulting residue was purified by silica gel (petroleum ether/EtOAc = 15:1) obtained **1a-1x**.



Substrates **1a**,^{3a,c} **1b**,^{3b} **1c-d**,^{3a} **1g**,^{3b} **1o-p**^{3a} are known compounds and the analytical data are consistent with the previous literature.

2.2 Optimization data for compound **4a**

Table S1. Optimization of the reaction conditions^{a,b}

Entry	Photocatalyst	Base	Solvent	Yield (%)
1	<i>fac</i> -Ir(ppy) ₃	K ₂ HPO ₄	DMF	67
2	(Ir[dF(CF ₃)ppy] ₂ (dtbpy))PF ₆	K ₂ HPO ₄	DMF	33
3	Eosin Y	K ₂ HPO ₄	DMF	N. R.
4	Ru(bpy) ₃ Cl ₂	K ₂ HPO ₄	DMF	12
5	<i>fac</i> -Ir(ppy) ₃	K ₂ HPO ₄	CH ₃ CN	29
6	<i>fac</i> -Ir(ppy) ₃	K ₂ HPO ₄	CHCl ₃	37
7	<i>fac</i> -Ir(ppy) ₃	K ₂ HPO ₄	DMSO	49
8	<i>fac</i> -Ir(ppy) ₃	K ₂ HPO ₄	1,4-Dioxane	46
9	<i>fac</i> -Ir(ppy) ₃	K ₂ HPO ₄	MeOH	31
10 ^c	<i>fac</i> -Ir(ppy) ₃	—	DMF	16
11	<i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	DMF	41
12	<i>fac</i> -Ir(ppy) ₃	KOH	DMF	27
13	<i>fac</i> -Ir(ppy) ₃	2,6-lutidine	DMF	35
14	<i>fac</i> -Ir(ppy) ₃	KHCO ₃	DMF	39
15	<i>fac</i> -Ir(ppy) ₃	CsF	DMF	26
16 ^d	<i>fac</i> -Ir(ppy) ₃	K ₂ HPO ₄	DMF	N.R.
17 ^e	<i>fac</i> -Ir(ppy) ₃	K ₂ HPO ₄	DMF	trace
18 ^f	—	K ₂ HPO ₄	DMF	0

^aReaction conditions: **1a** (0.2 mmol, 0.1 M in solvent), **2a** (2.0 equiv.), base (2.0 equiv.), *fac*-Ir(ppy)₃ (0.005 mmol), under N₂ atmosphere irradiated using blue LEDs (5W) at room temperature for **24** h. ^bIsolated yield of **4a**. ^cWithout base. ^dIn the dark. ^eIn the air. ^fWithout catalyst.

2.3 Light Source in Detail and Experimental Set-up Photograph



Small scale reaction

The light source used for photochemical experiments was a 5 W blue Leds belt, purchased from JD.COM

Manufacturer: Gree Think. China

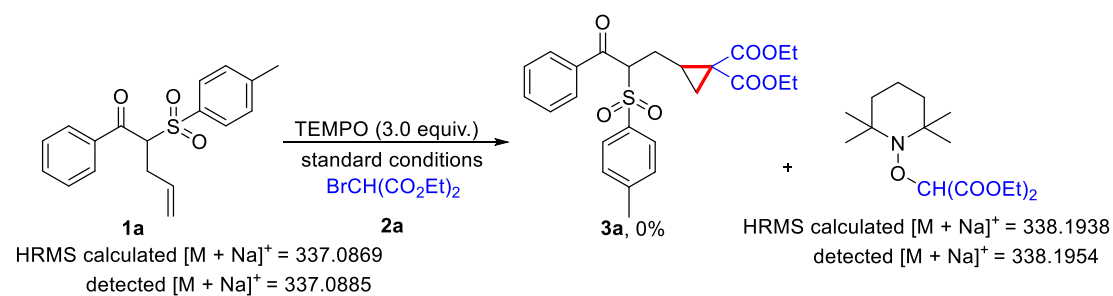
Model: 12v3528

Broadband source: $\lambda = 460\text{-}470\text{ nm}$

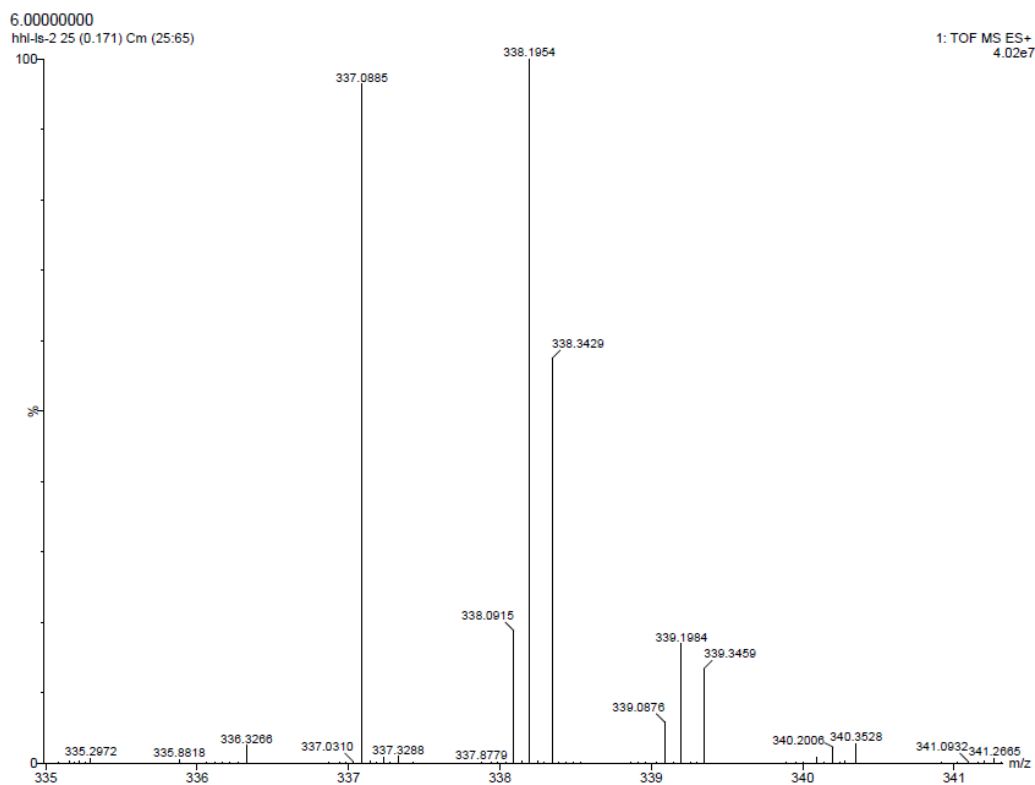
Material of the irradiation vessel: Schlenk tube

Distance from the light source to the irradiation vessel: 6.0 cm (Not use any filters)

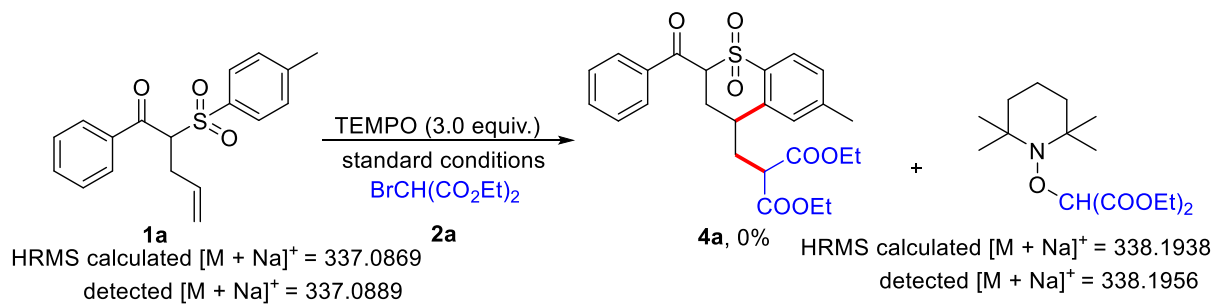
2.4 TEMPO control experiment for compound 3a



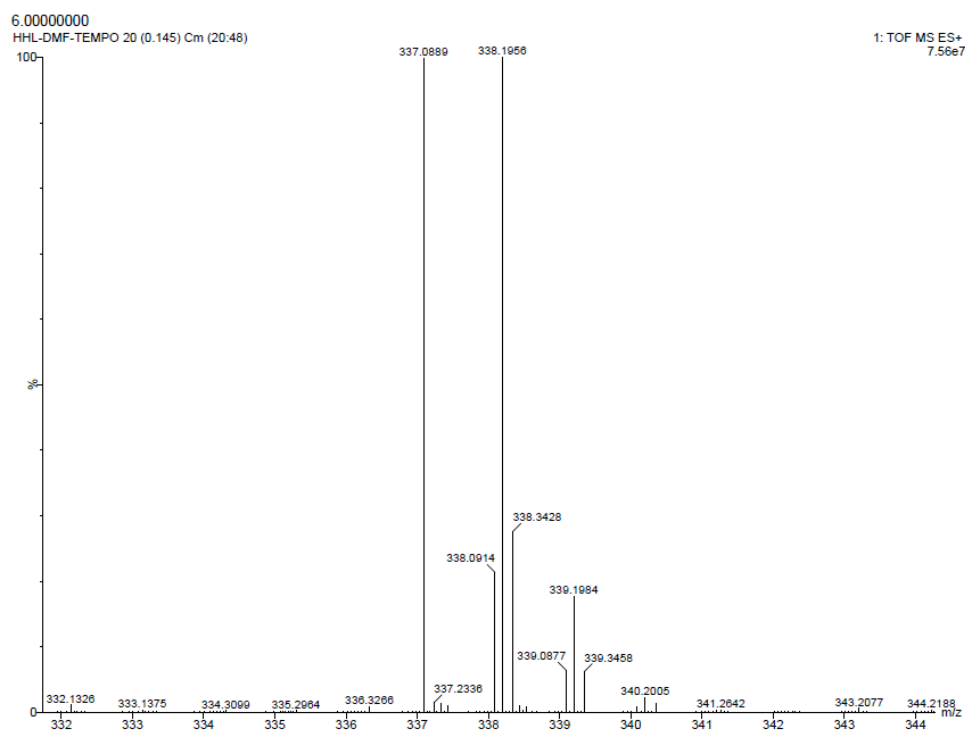
HRMS data:



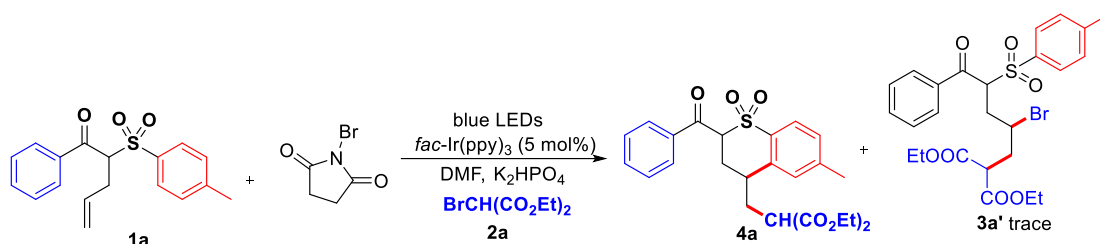
2.5 TEMPO control experiment for compound 4a



HRMS data:



2.6 NBS as bromination reagent for compound 4a.



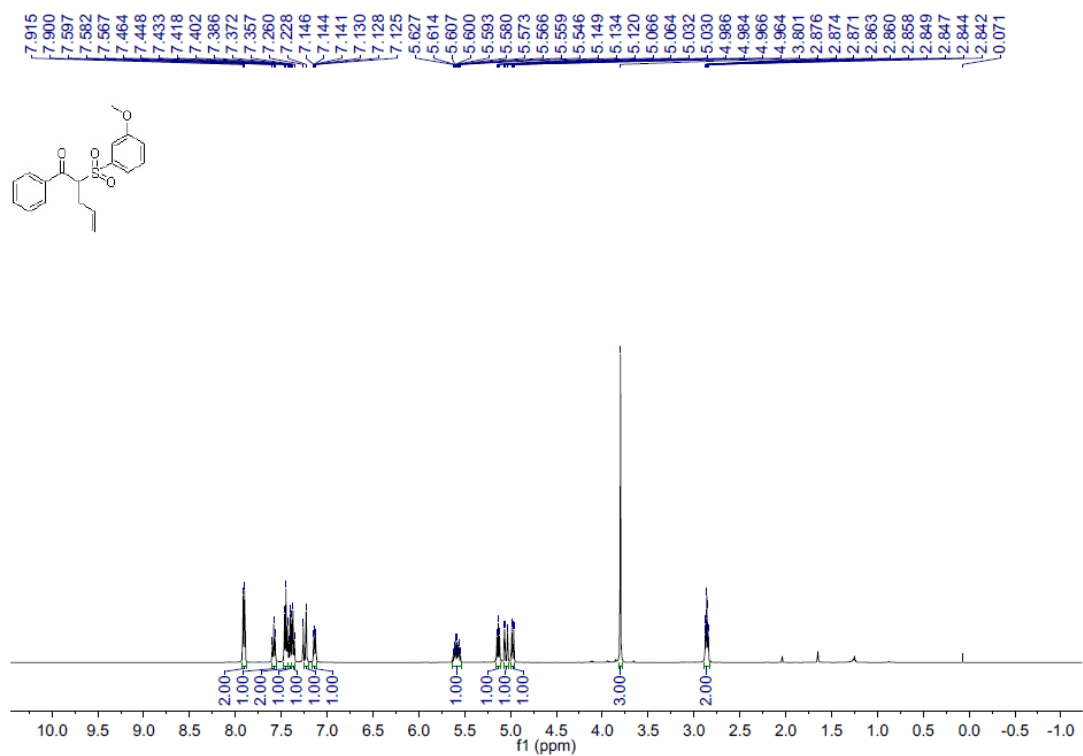
References

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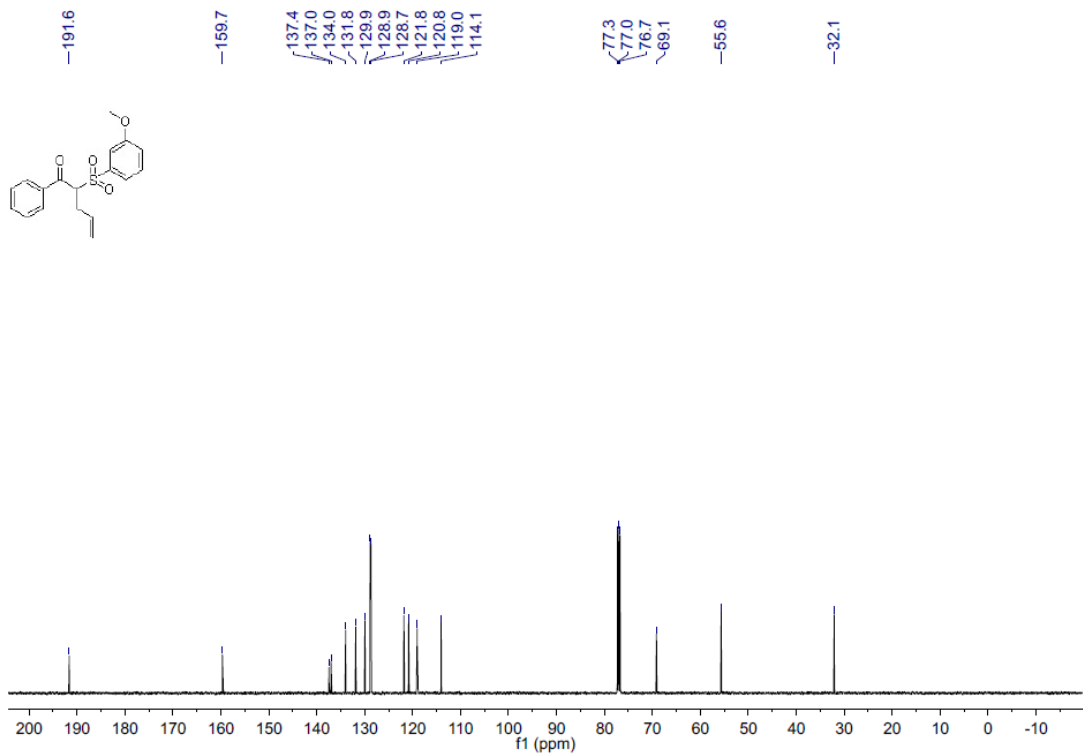
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- [3]. (a) Chang, M.-Y.; Cheng, Y.-C.; Lu, Y.-J. One-Pot Access to Sulfonylmethyl Arylpyrroles via the Domino Aerobic Wacker-Type Aminocyclization/1,4-Sulfonyl Migration. *Org. Lett.*, **2014**, *16*, 6252-6255. (b) Chang, M.-Y.; Hsiao, Y.-T. H₂SO₄-Mediated Stereocontrolled Annulation of Oxygenated Naphthalenes and 4-Alkenols: One-Pot Synthesis of Tetanthrenes. *J. Org. Chem.* **2017**, *82*, 11594-11602. (c) Chang, M.-Y.; Cheng, Y.-C.; Chan, C.-K. Synthesis of Vinylcyclopropanes by Allylation/ring-closing Metathesis/Claisen Rearrangement. *Tetrahedron* **2014**, *70*, 8908-8913.

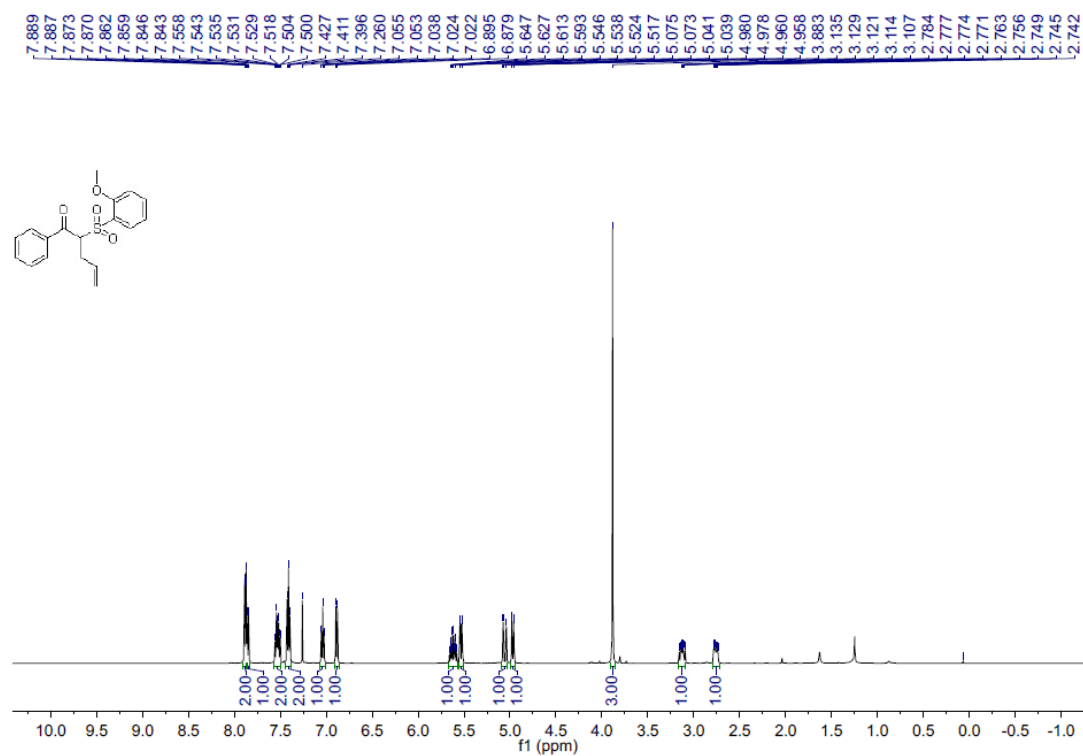
3. Copies of NMR Spectra

¹H NMR (500 MHz, CDCl₃) spectrum of compound **1e**

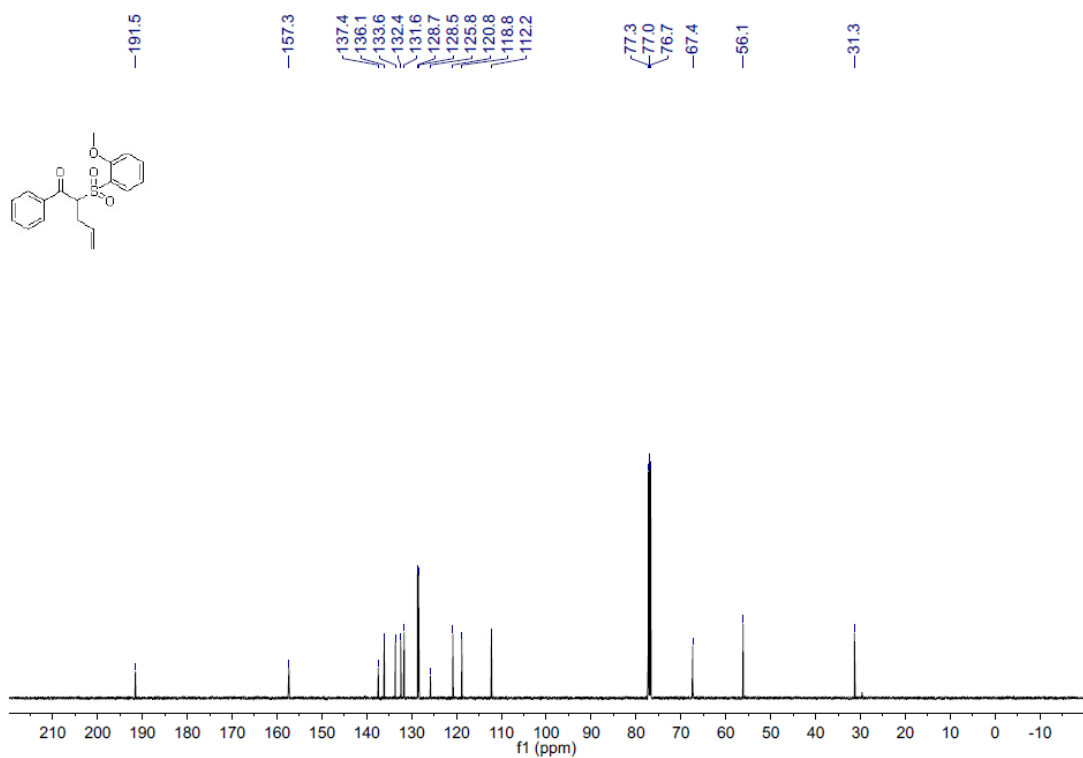


^{13}C $\{^1\text{H}\}$ NMR (126 MHz, CDCl_3) spectrum of compound **1e**

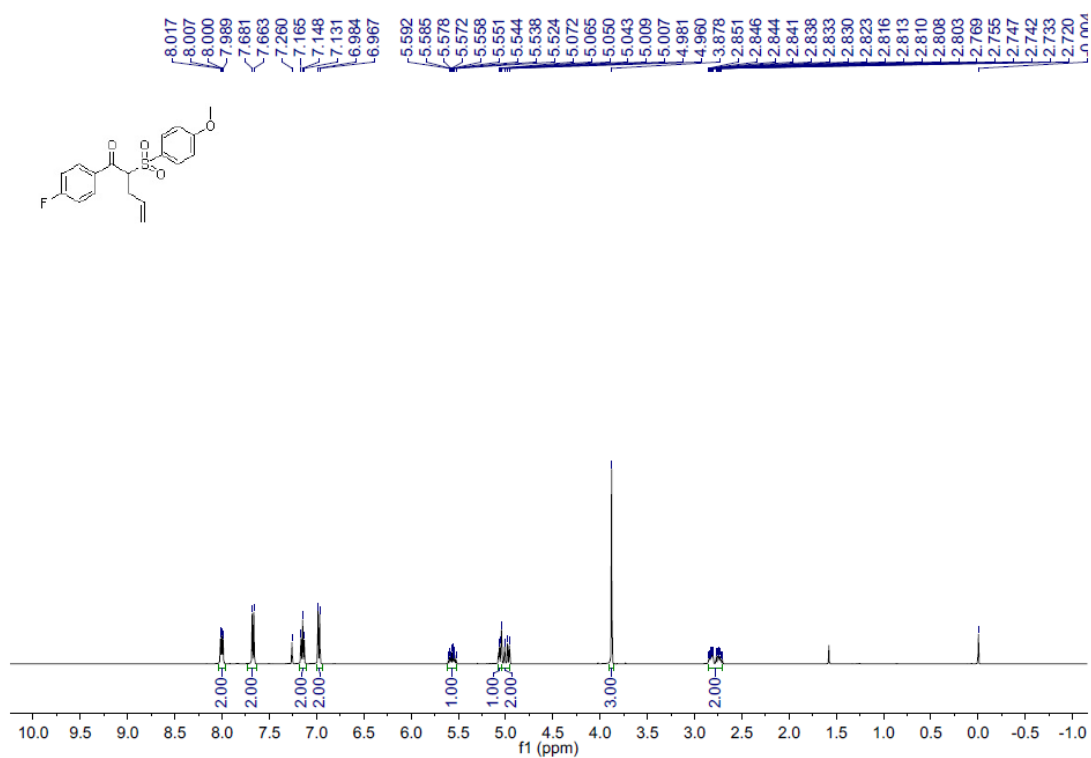


¹H NMR (500 MHz, CDCl₃) spectrum of compound **1f**

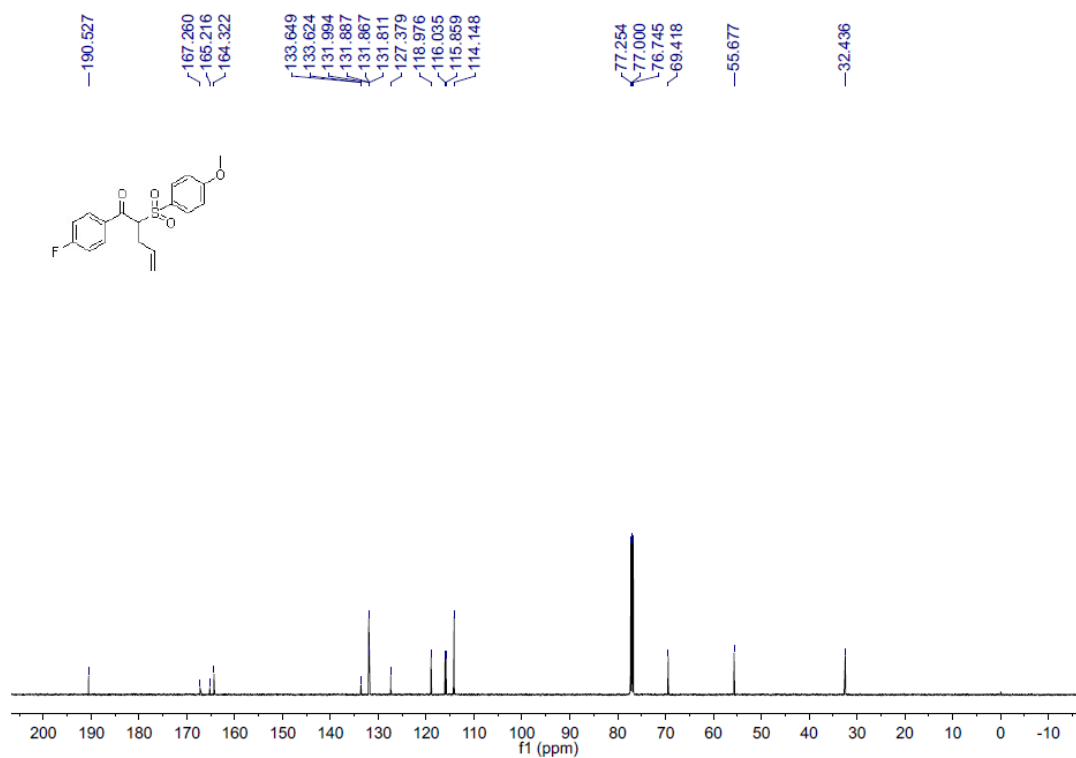
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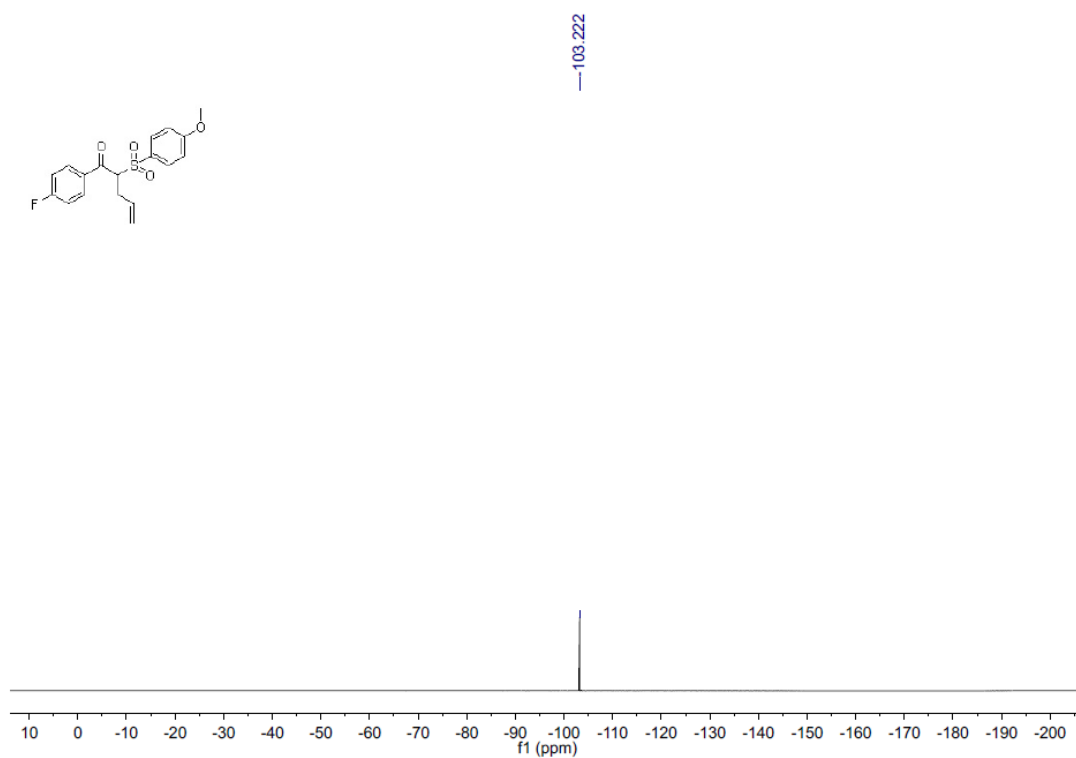
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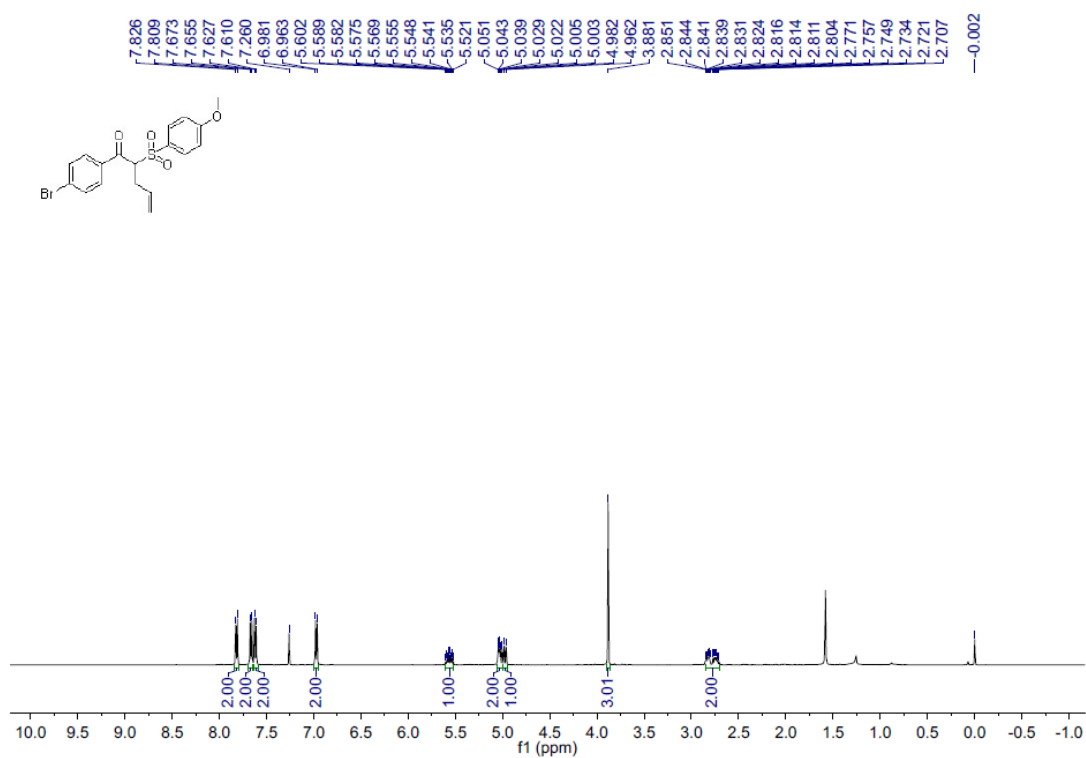
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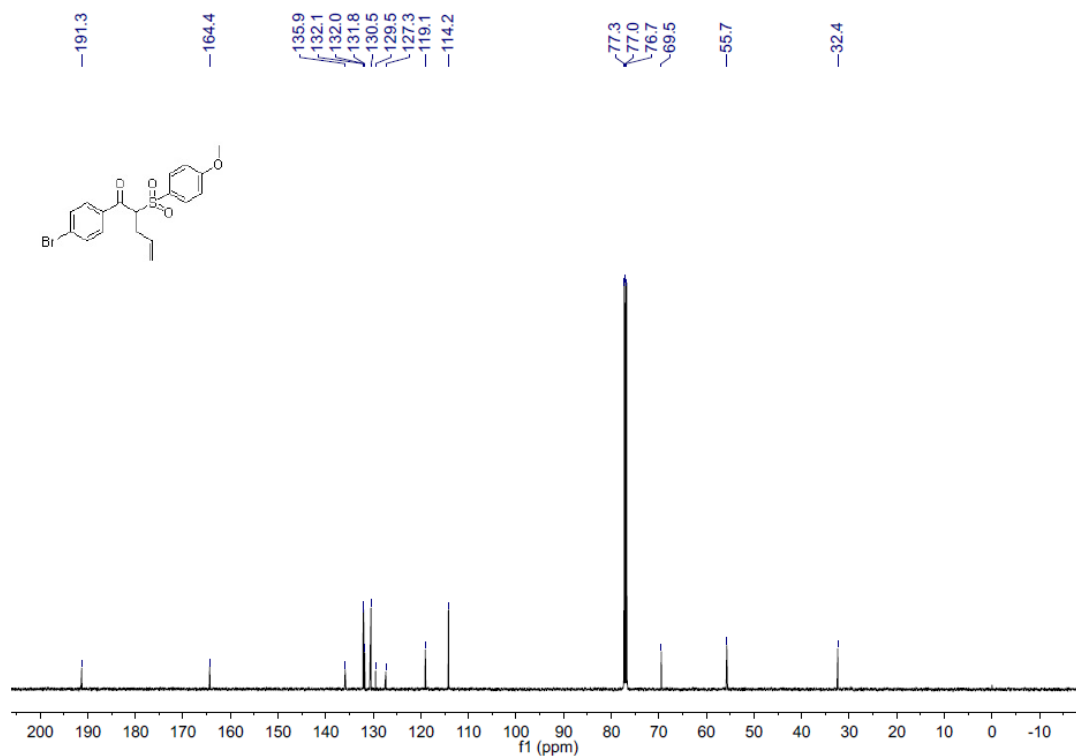
^{19}F NMR (470 MHz, CDCl_3) spectrum of compound **1h**



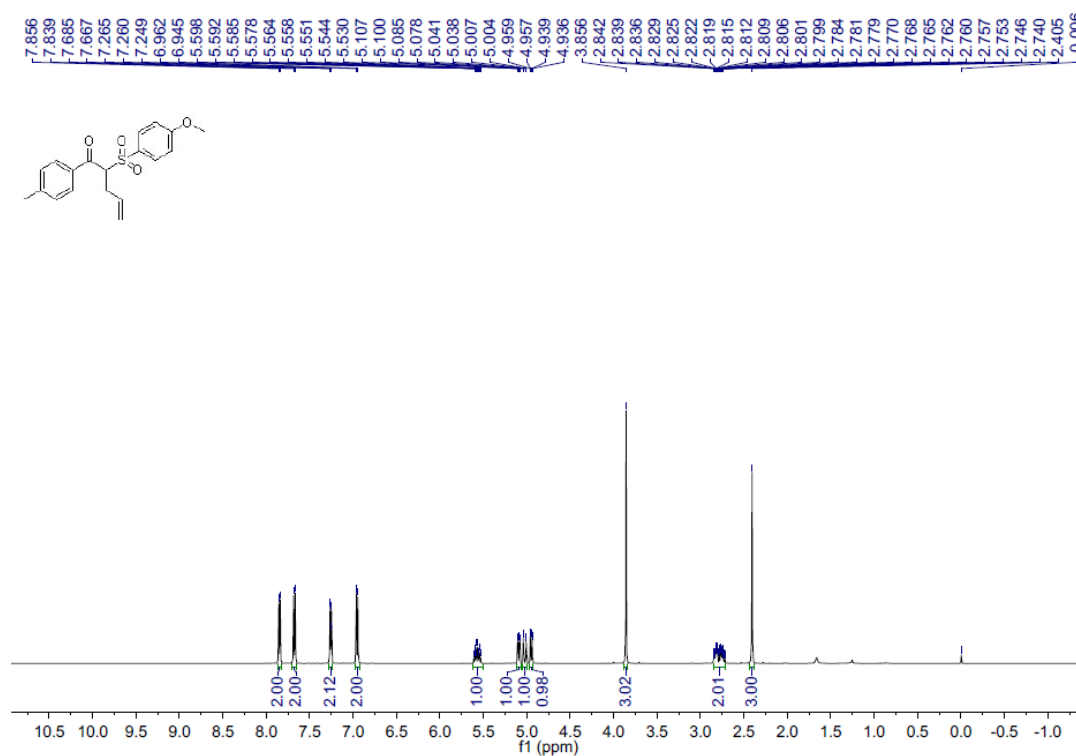
^1H NMR (500 MHz, CDCl_3) spectrum of compound **1i**



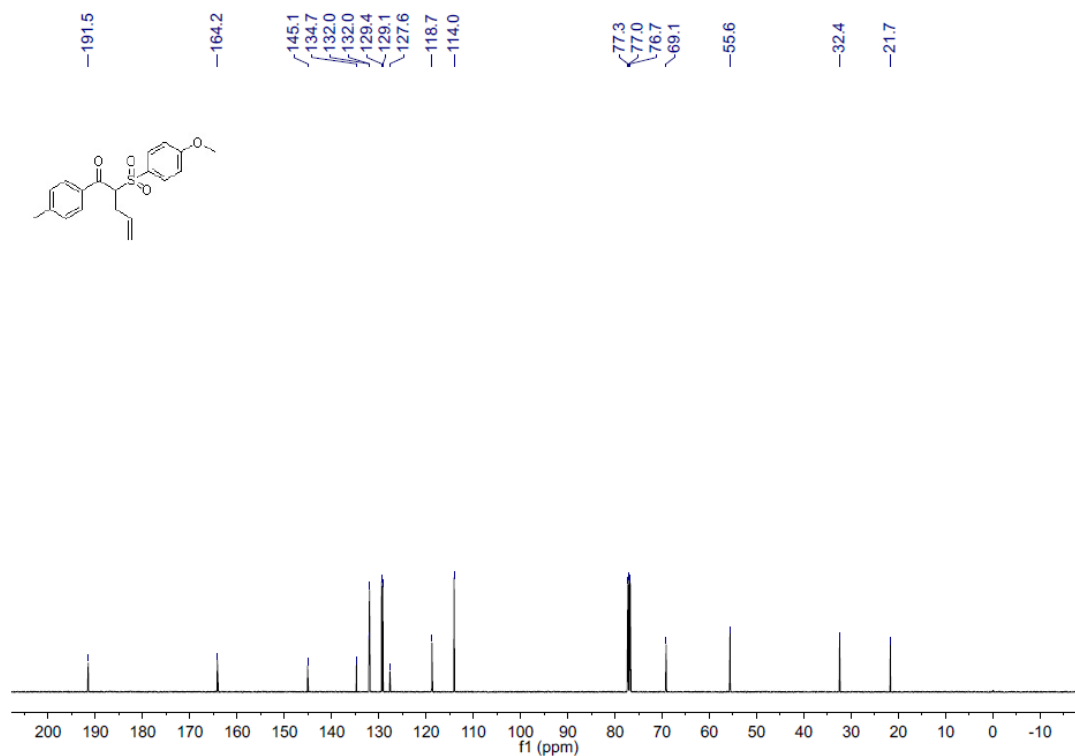
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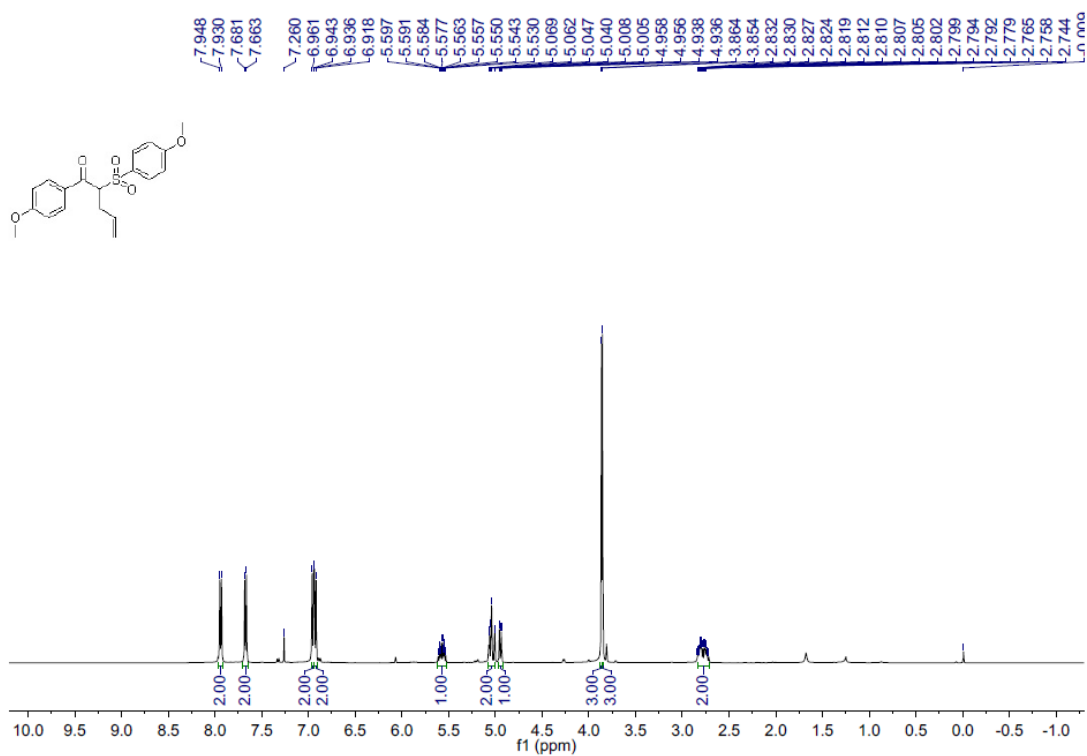
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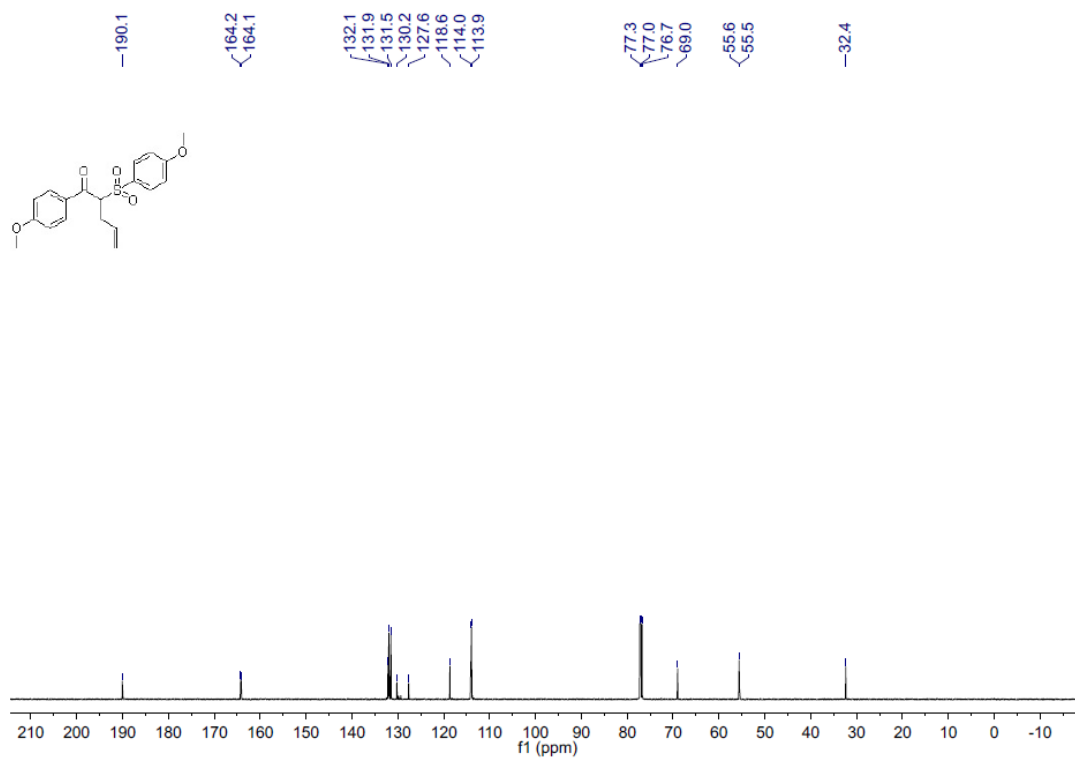
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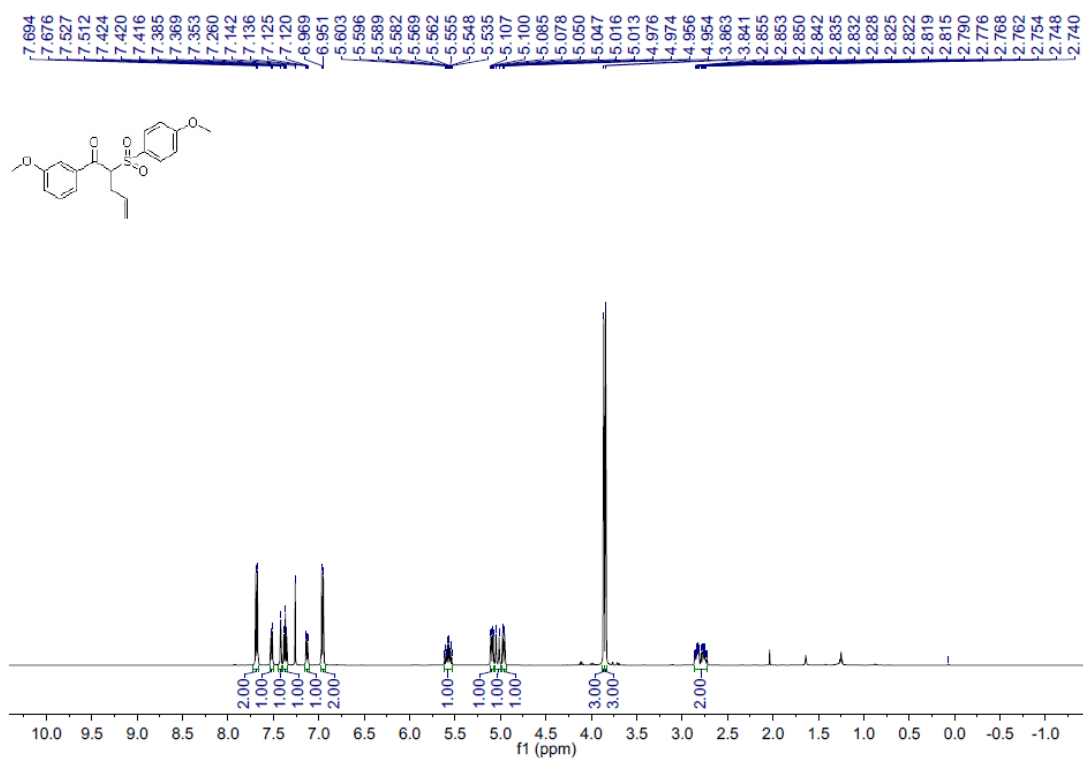
^1H NMR (500 MHz, CDCl_3) spectrum of compound **1k**



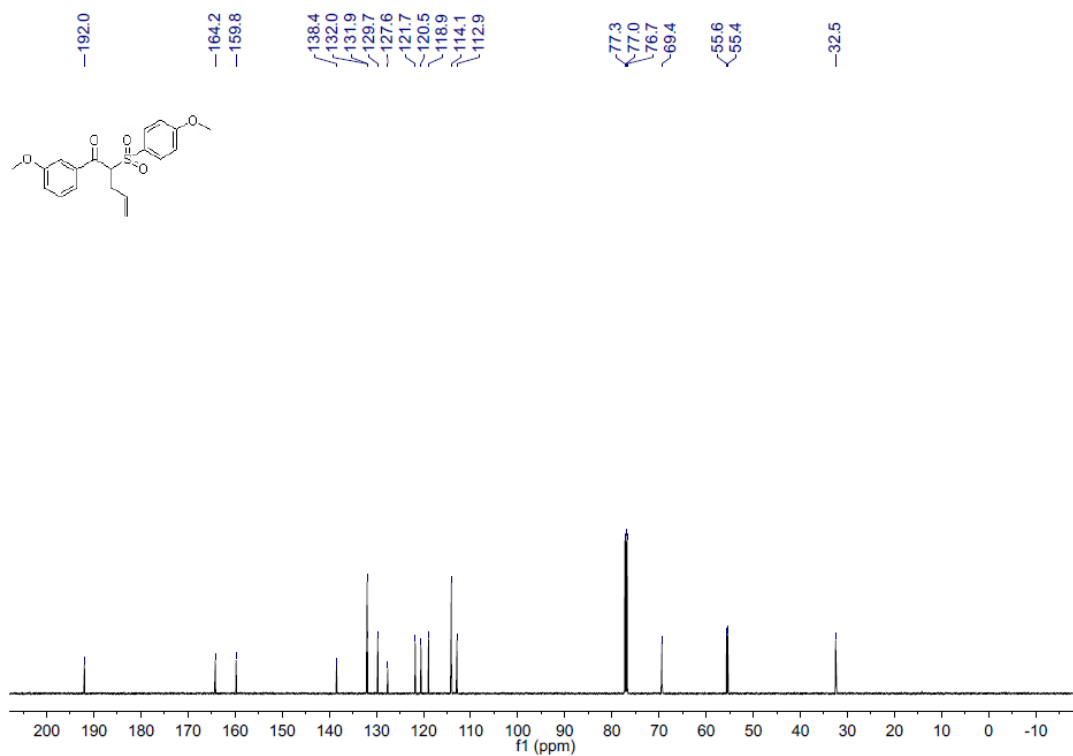
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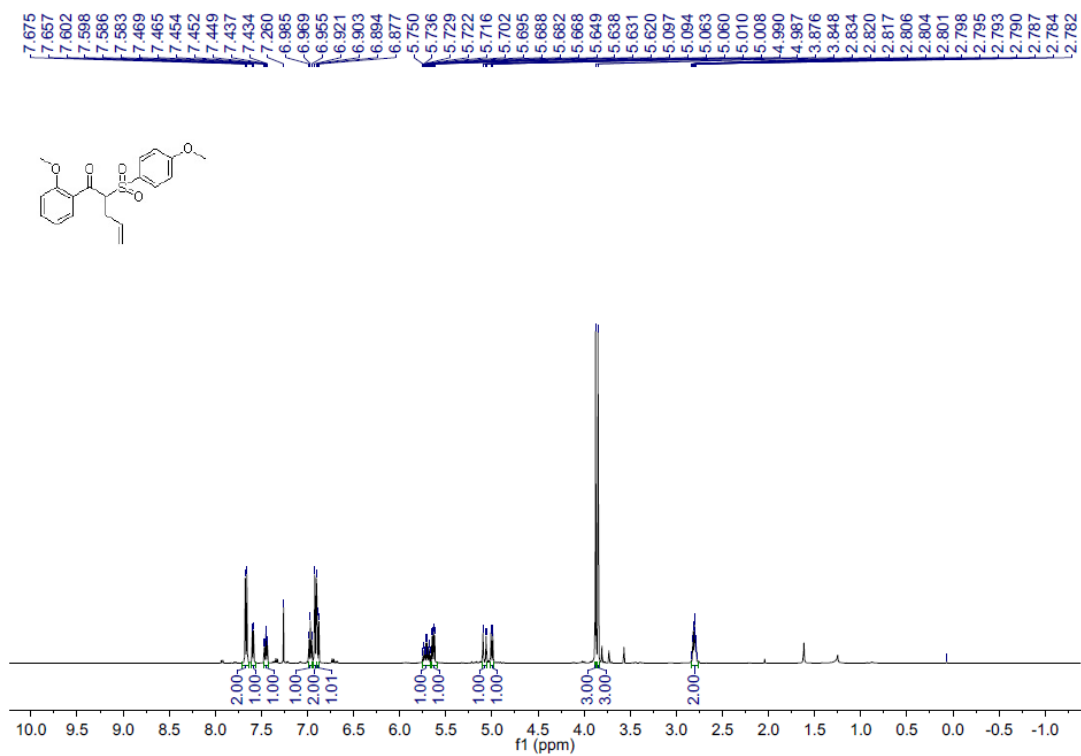
^1H NMR (500 MHz, CDCl_3) spectrum of compound **1l**



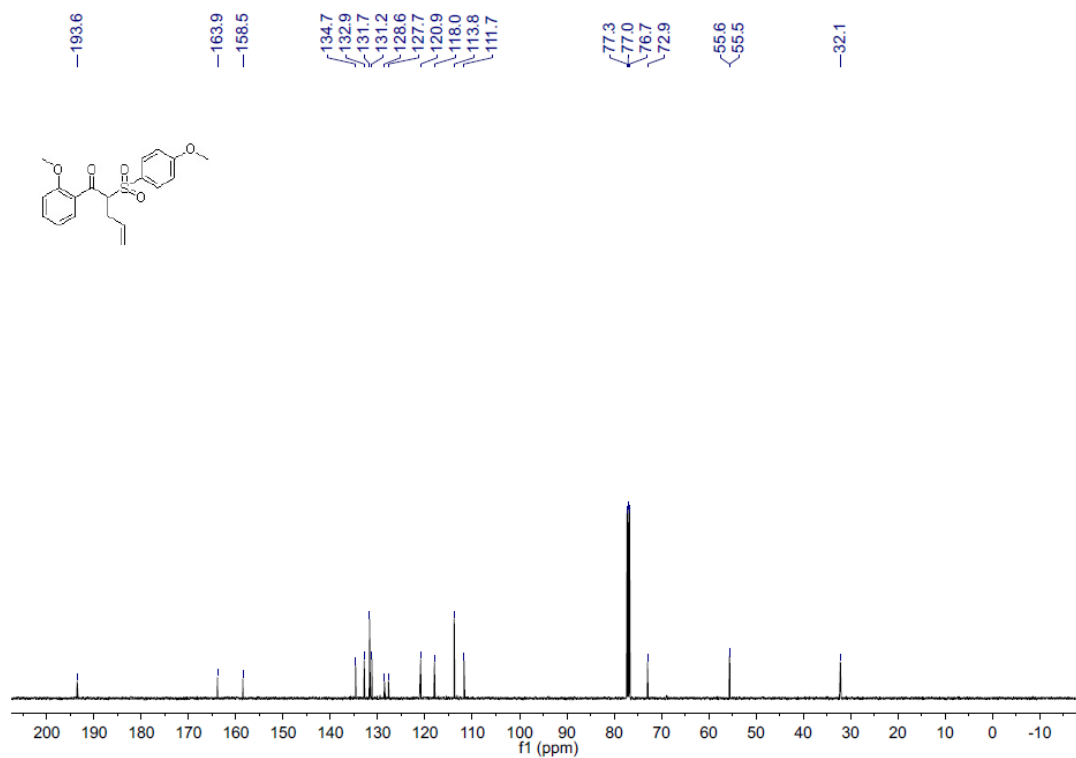
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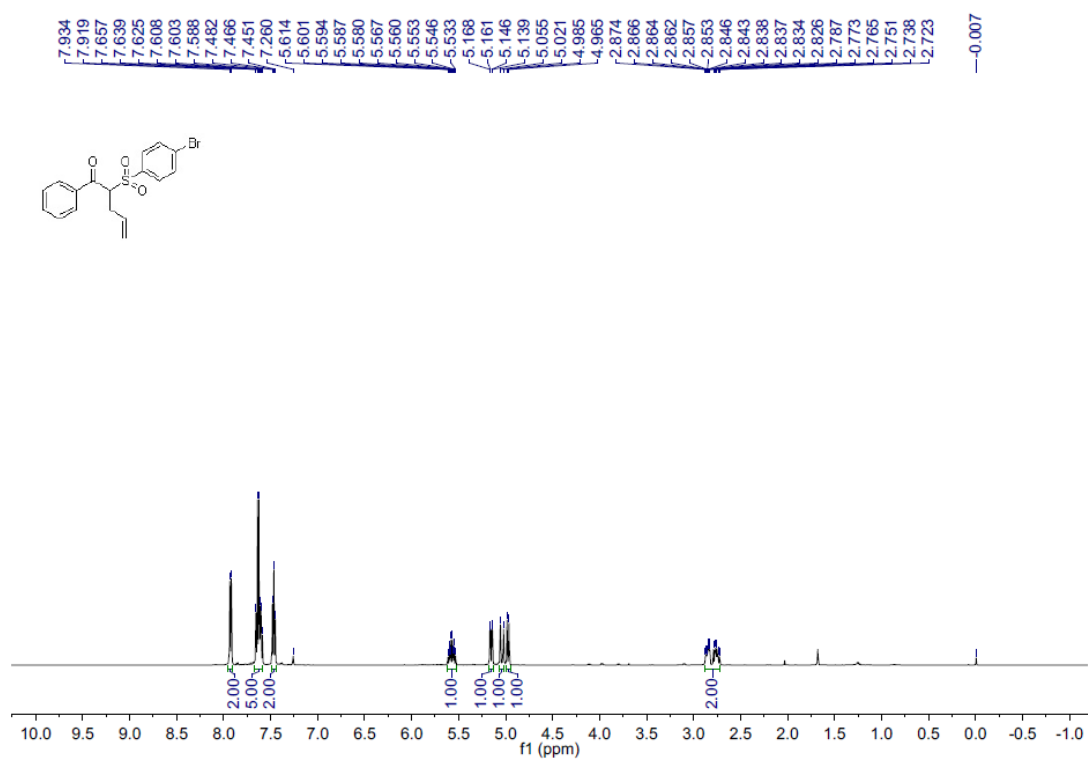
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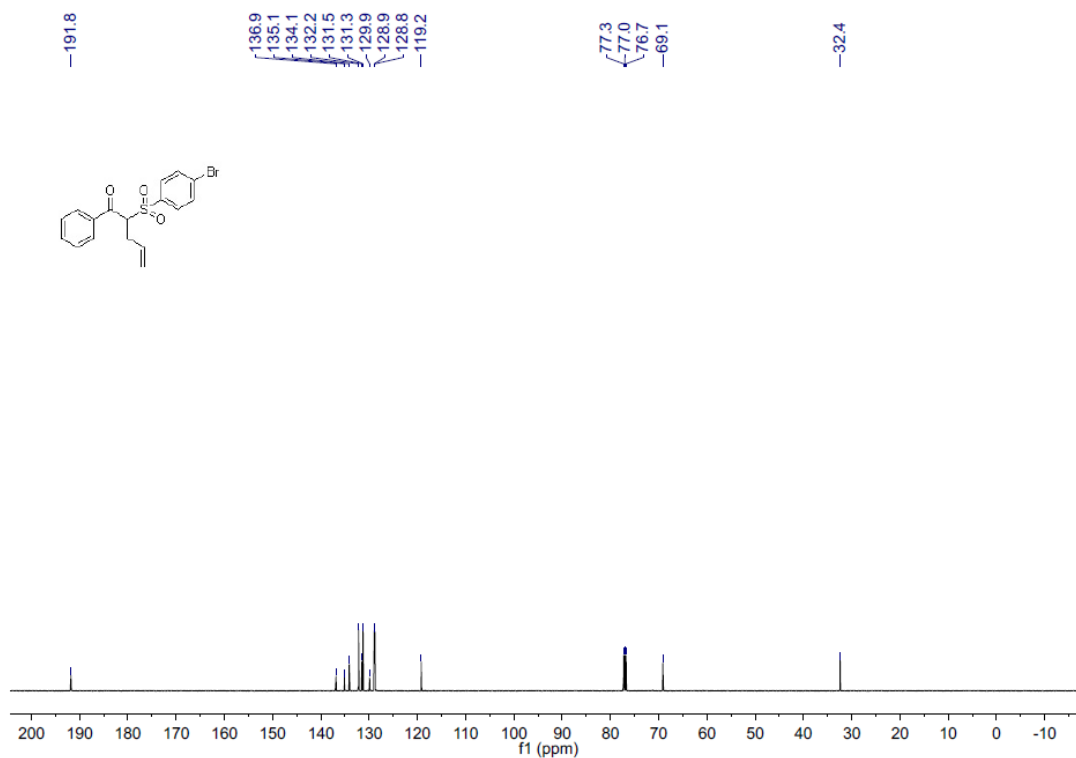
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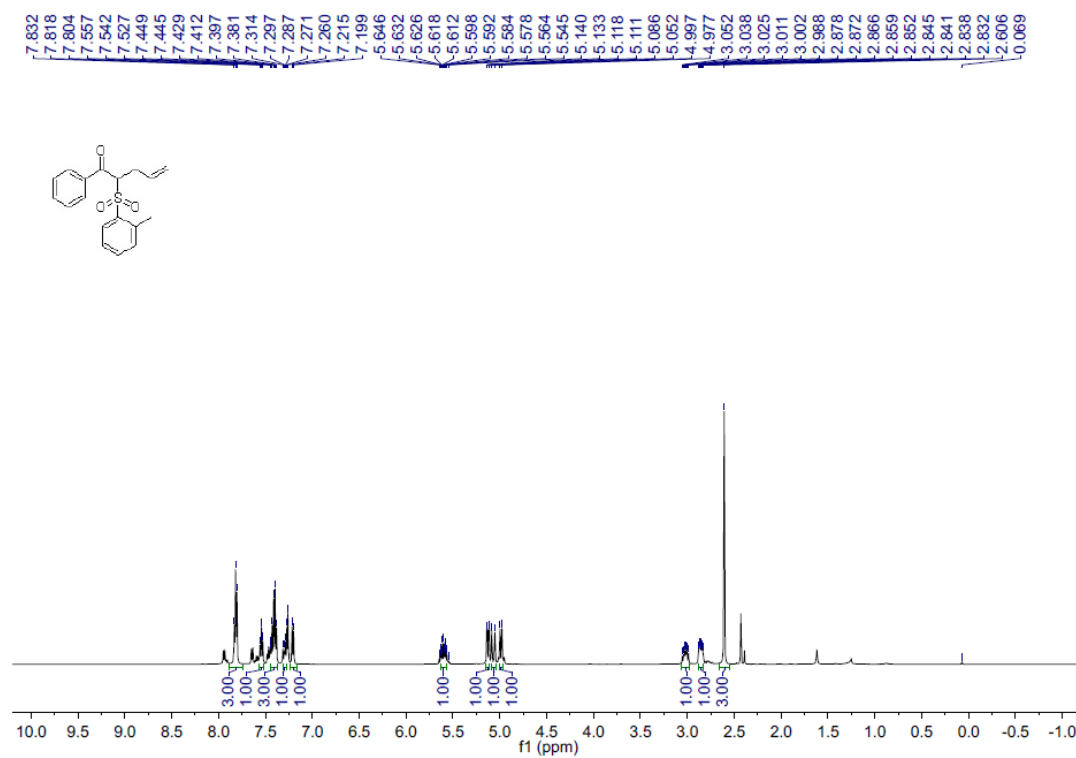
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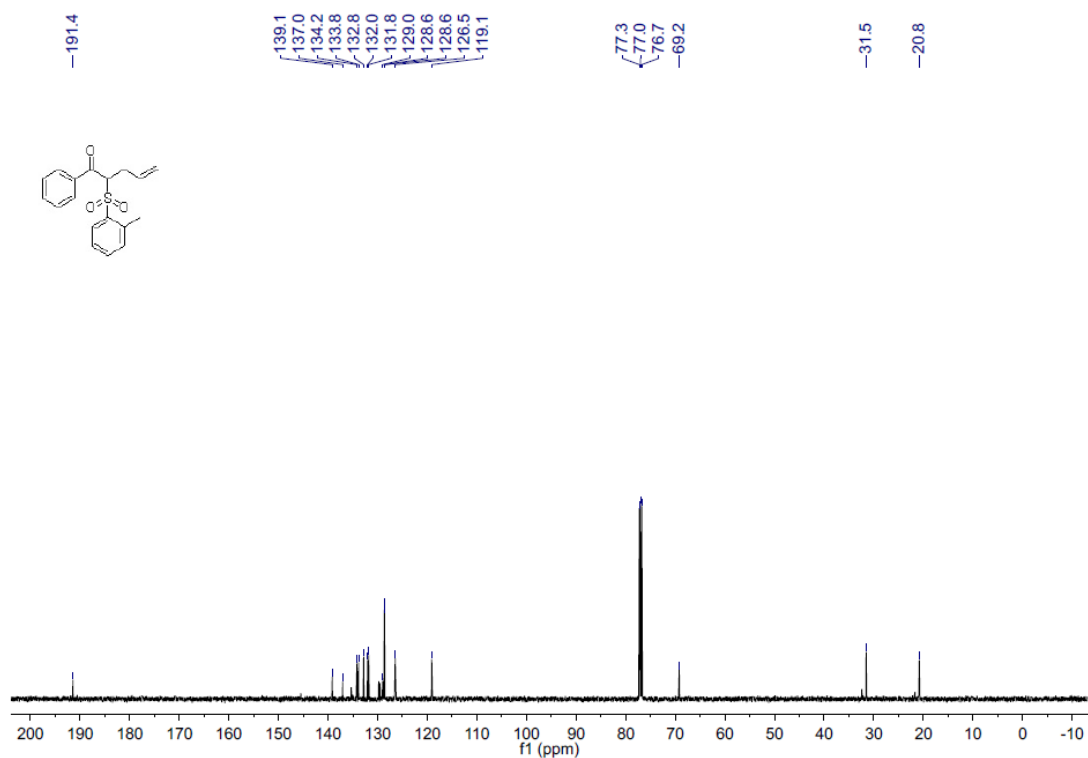
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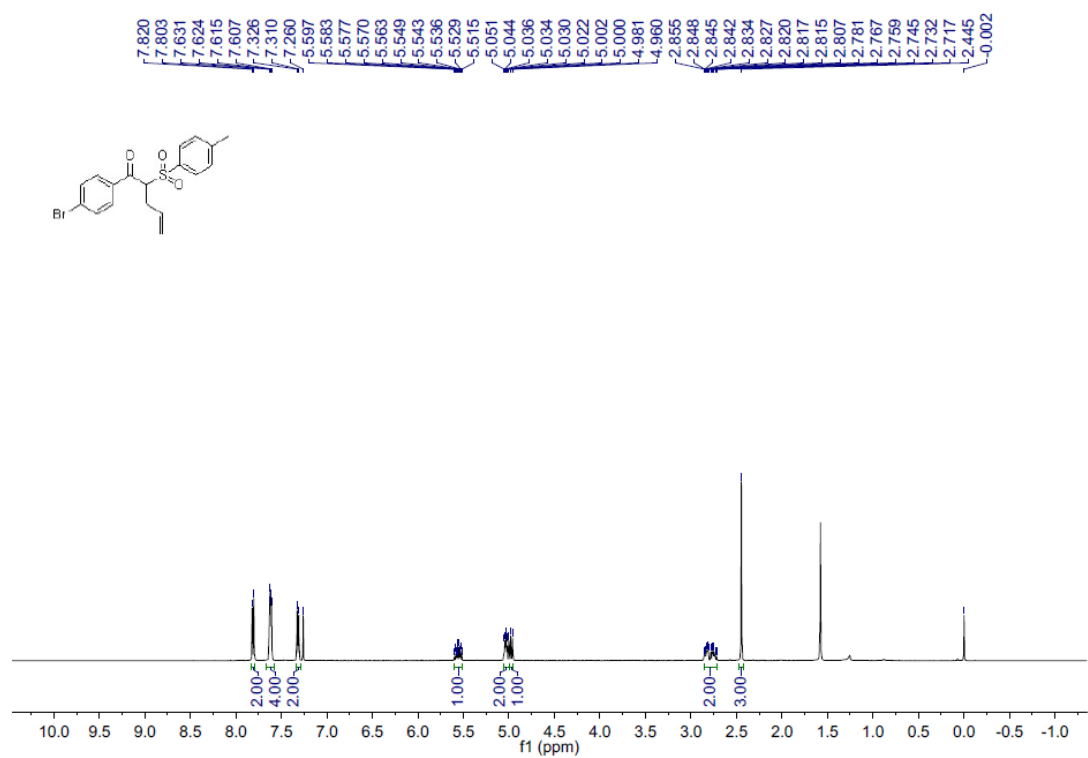
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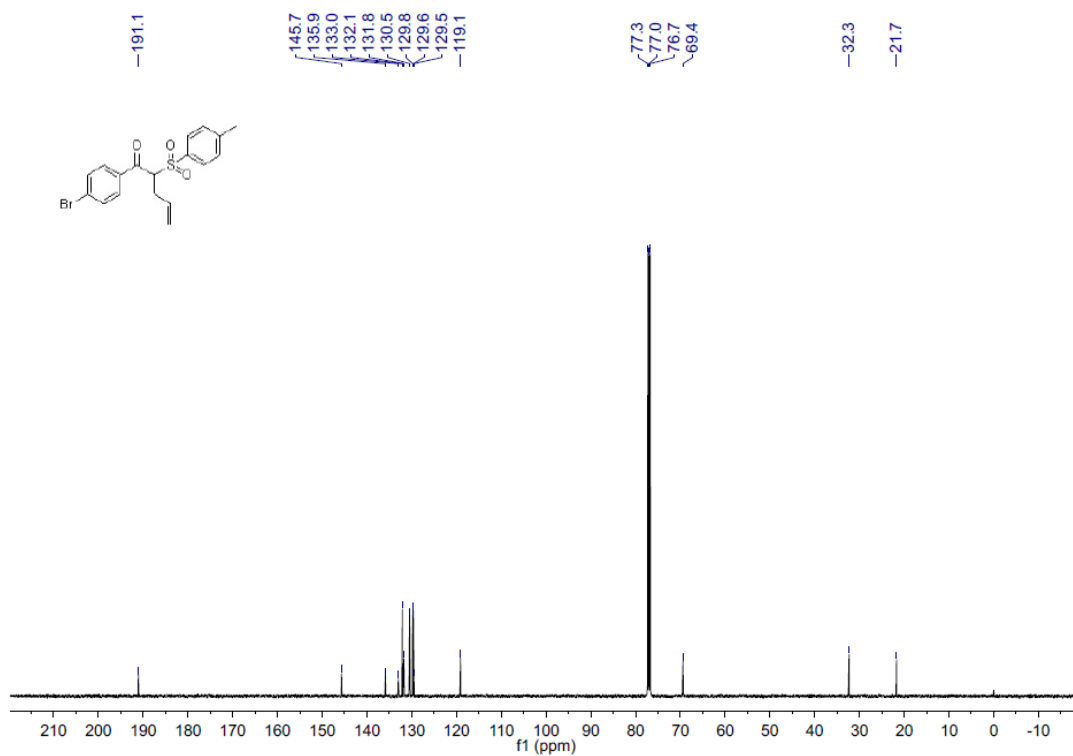
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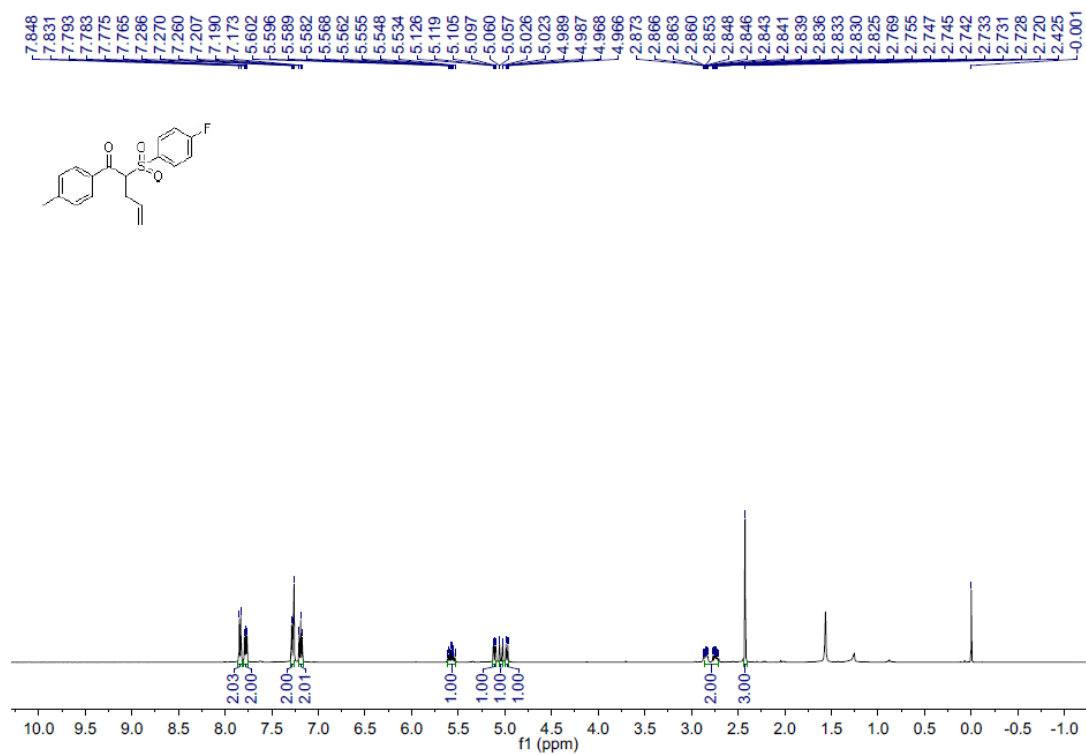
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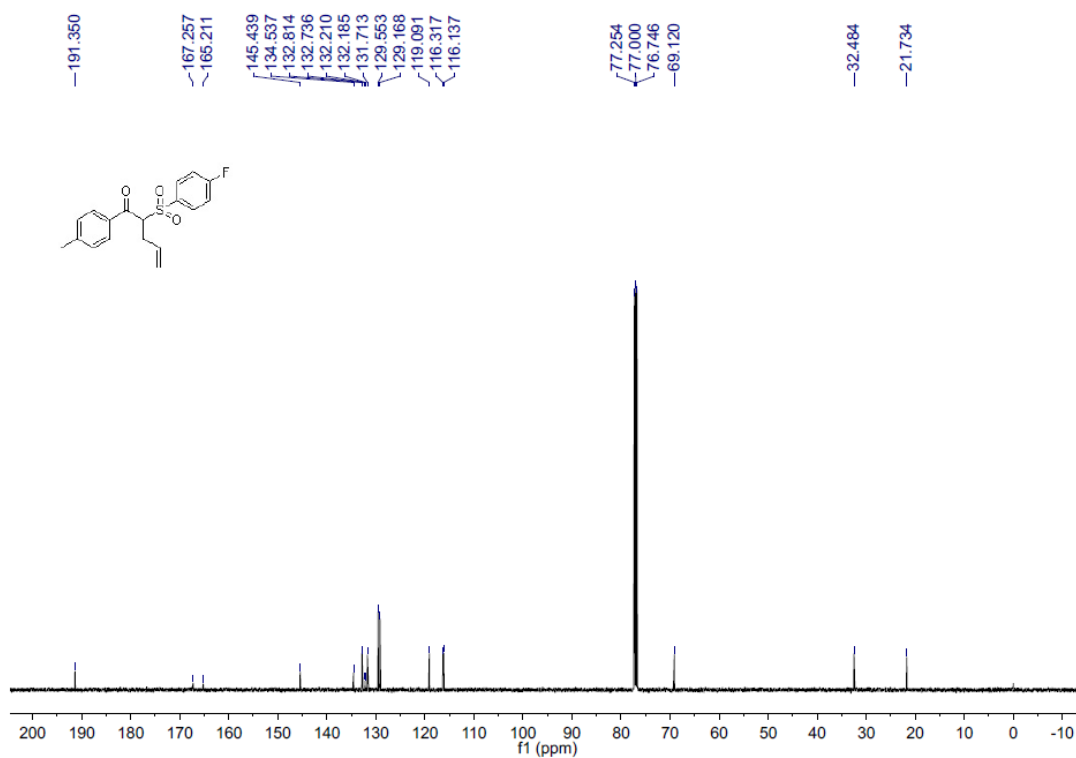
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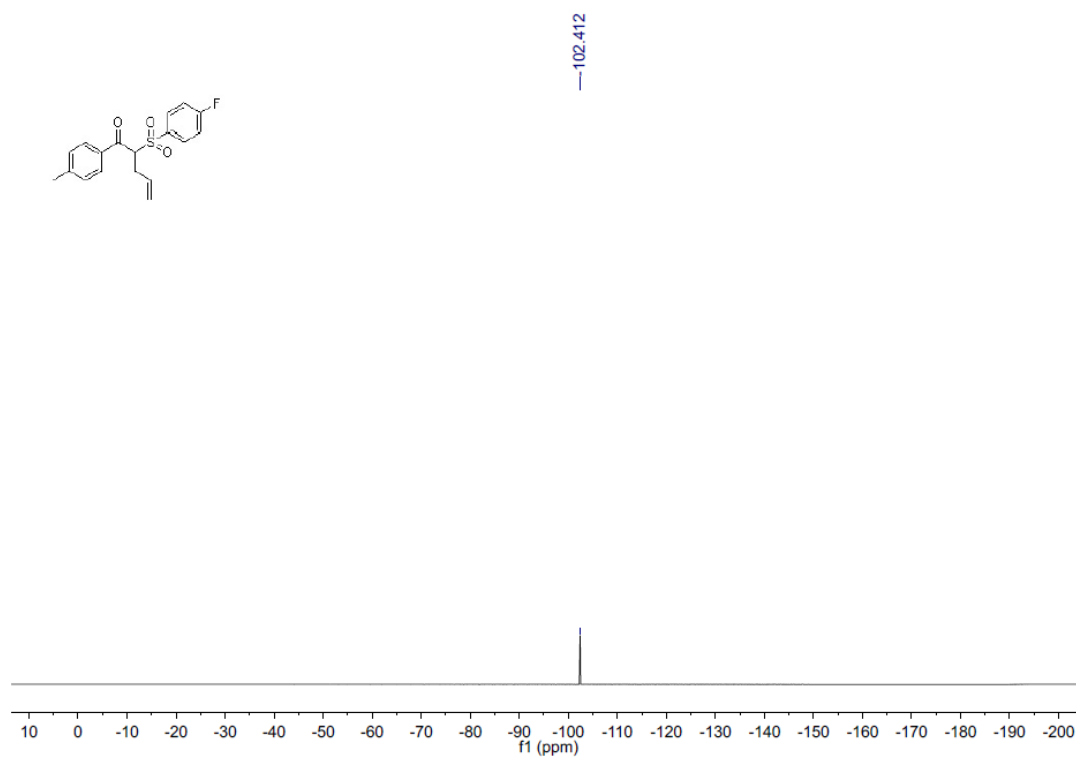
^1H NMR (500 MHz, CDCl_3) spectrum of compound **1t**



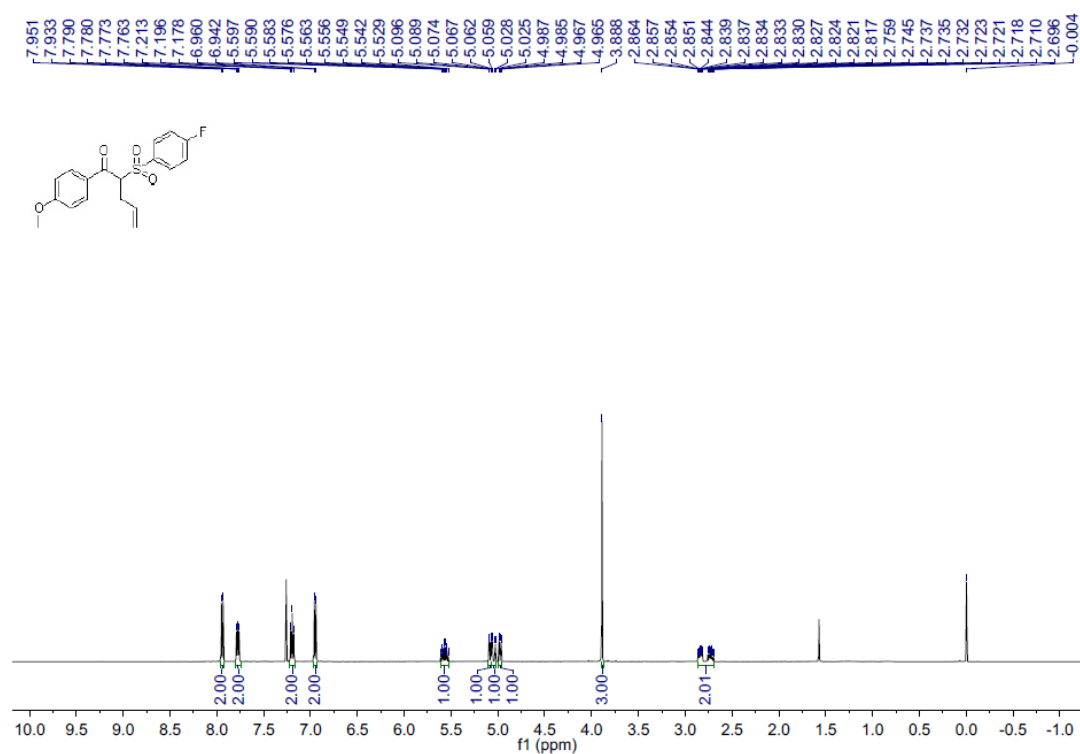
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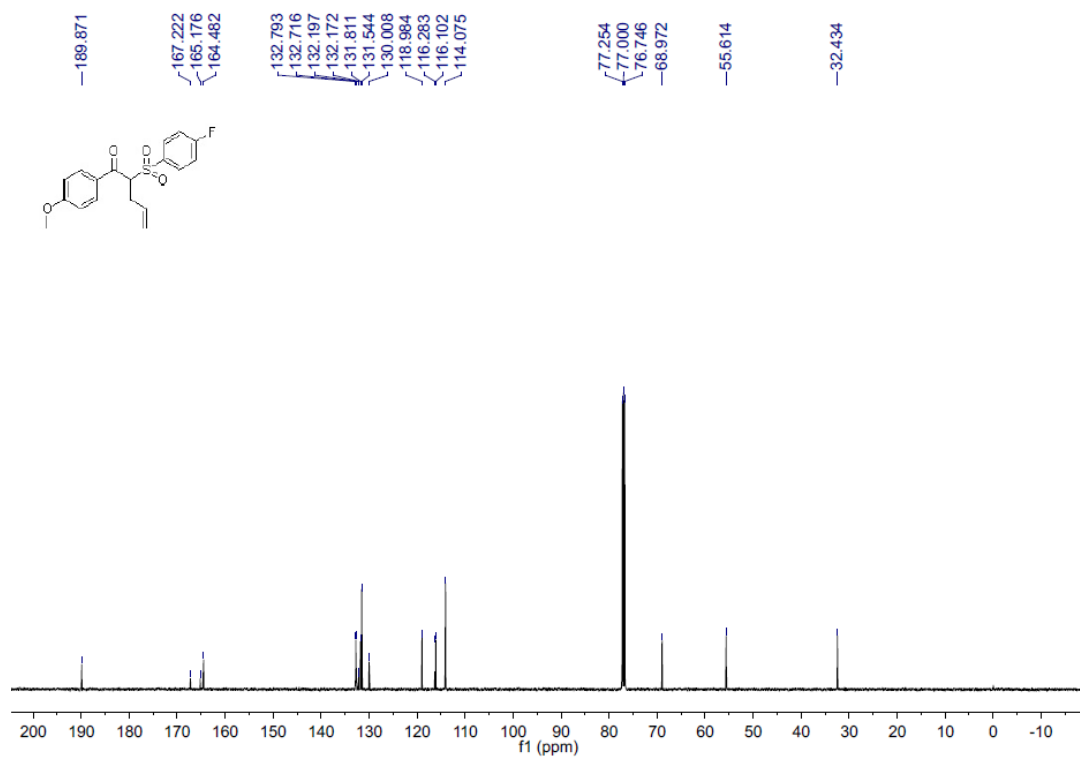
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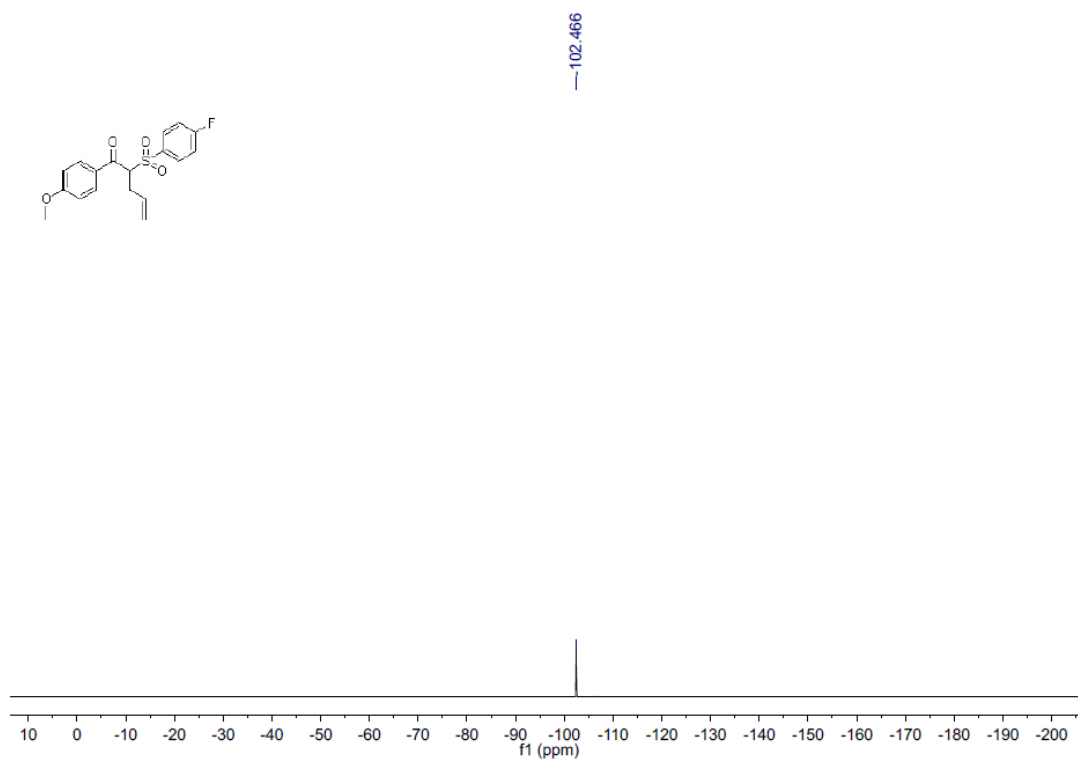
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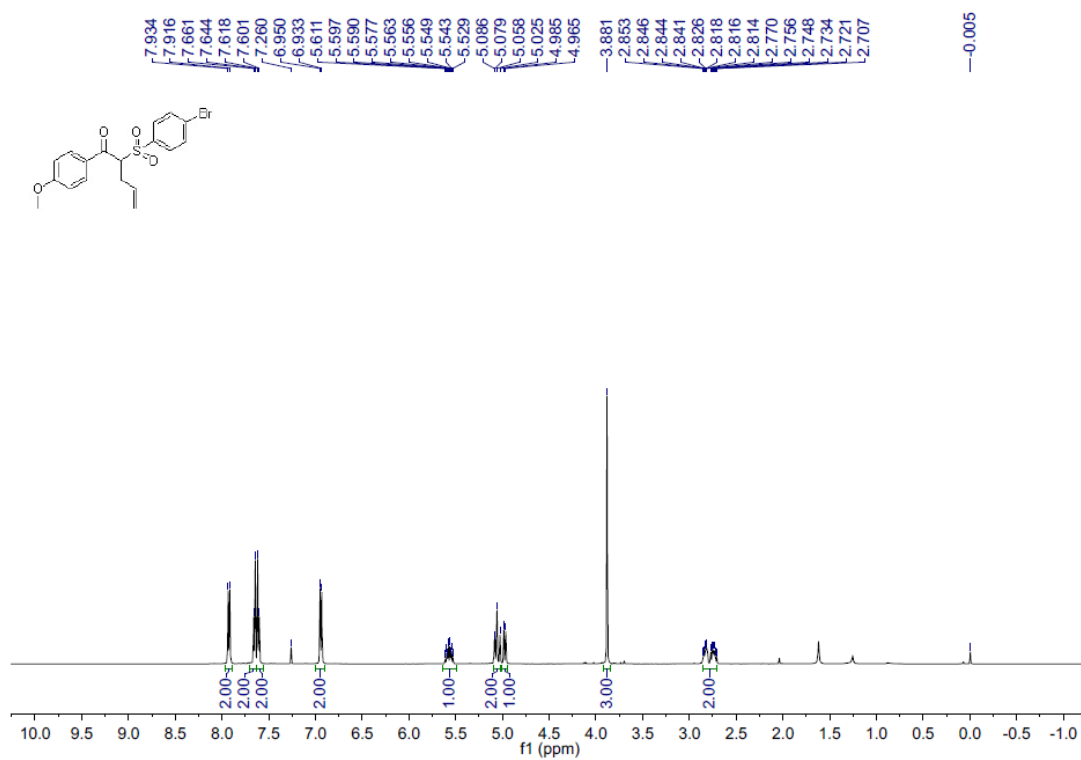
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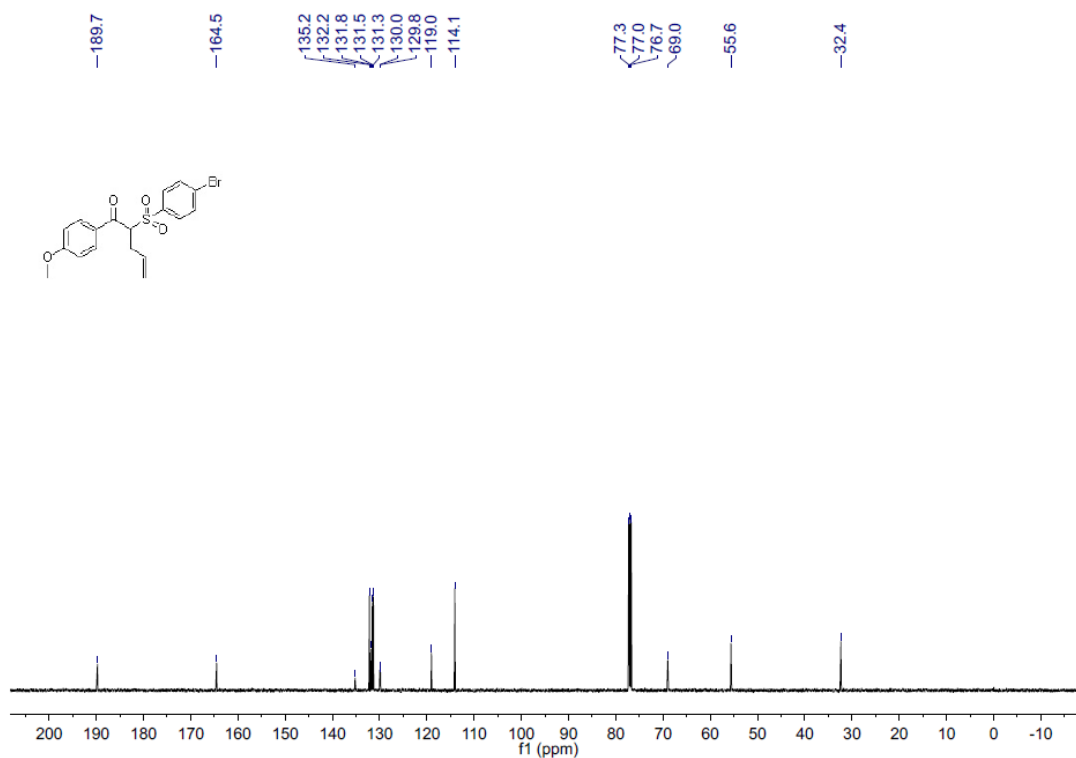
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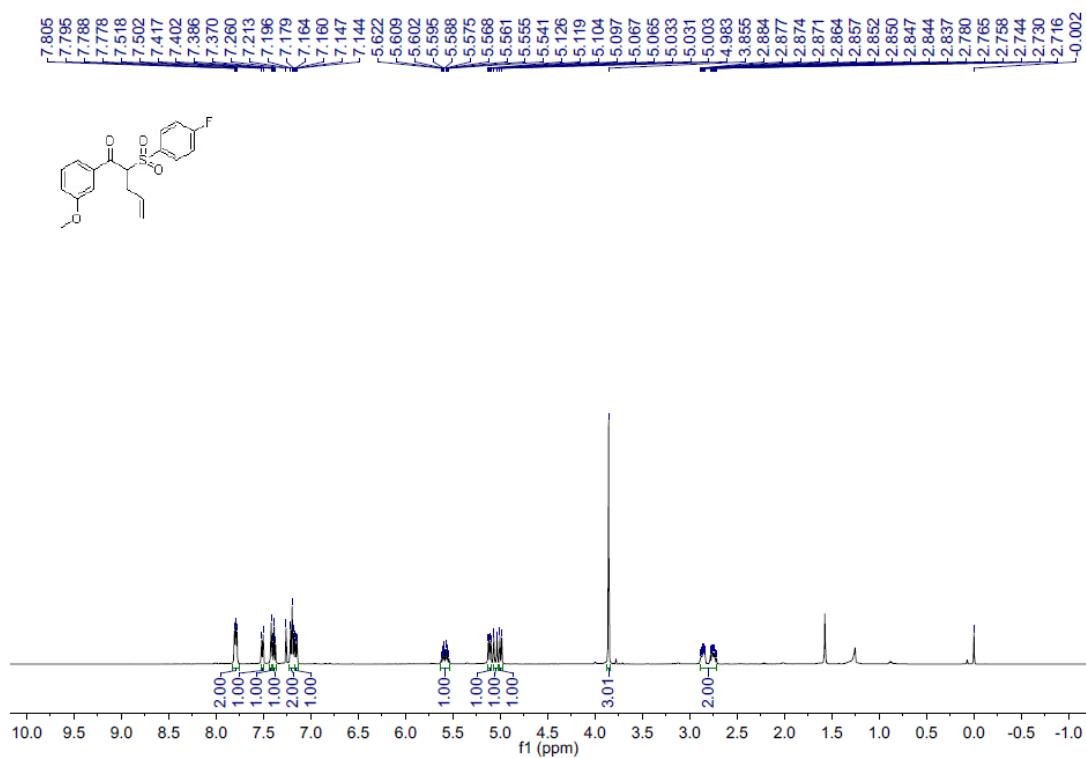
^1H NMR (500 MHz, CDCl_3) spectrum of compound **1v**



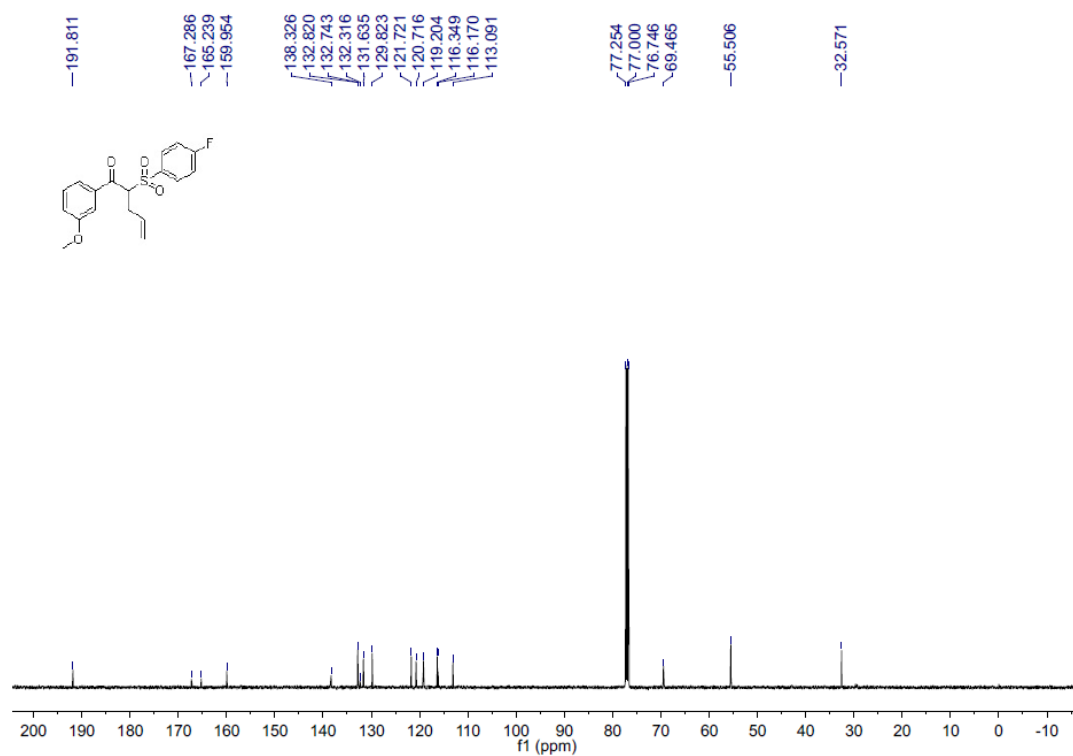
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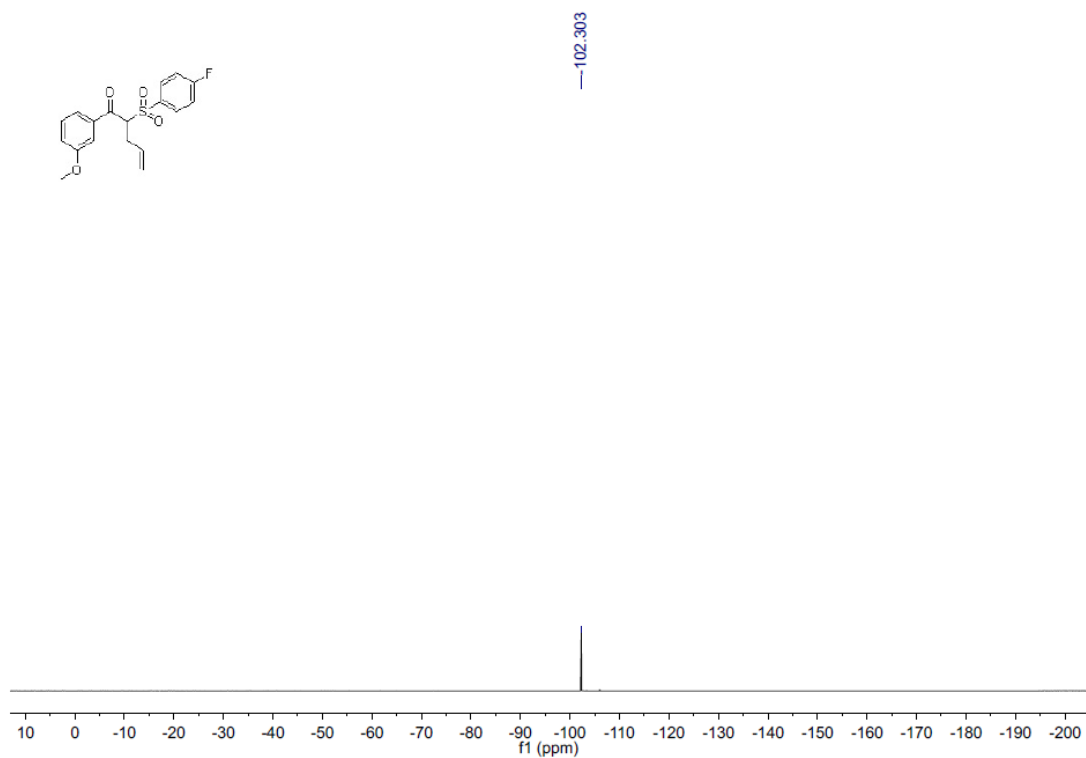
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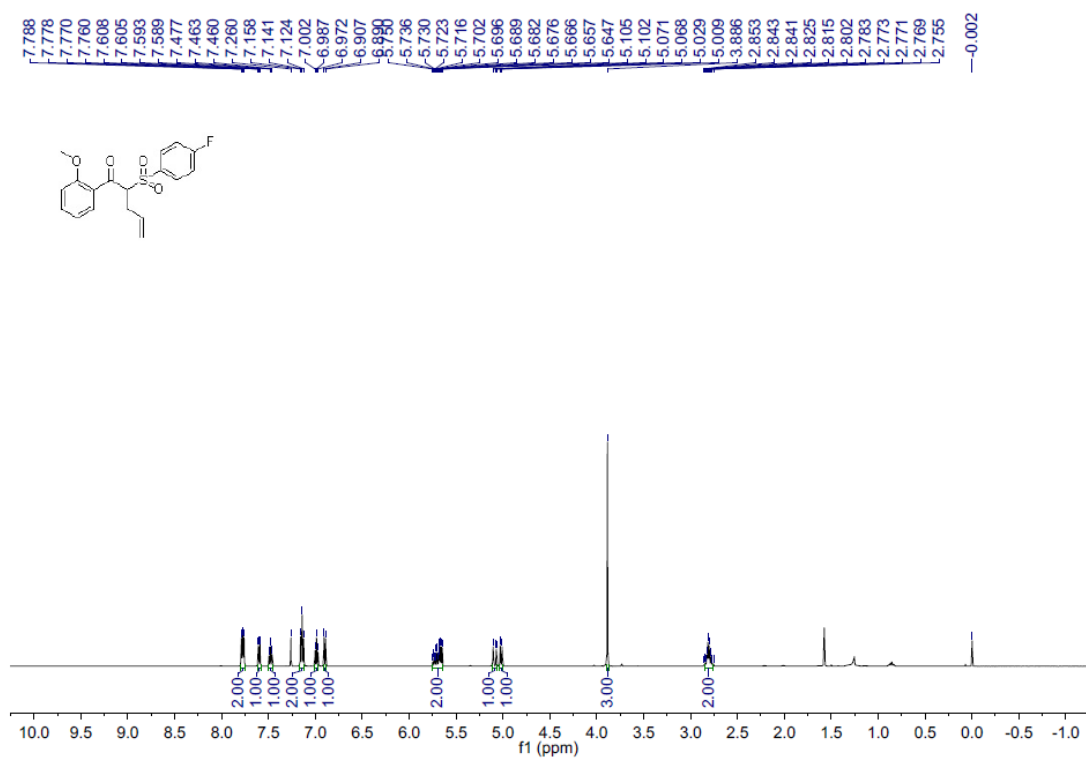
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **1w**



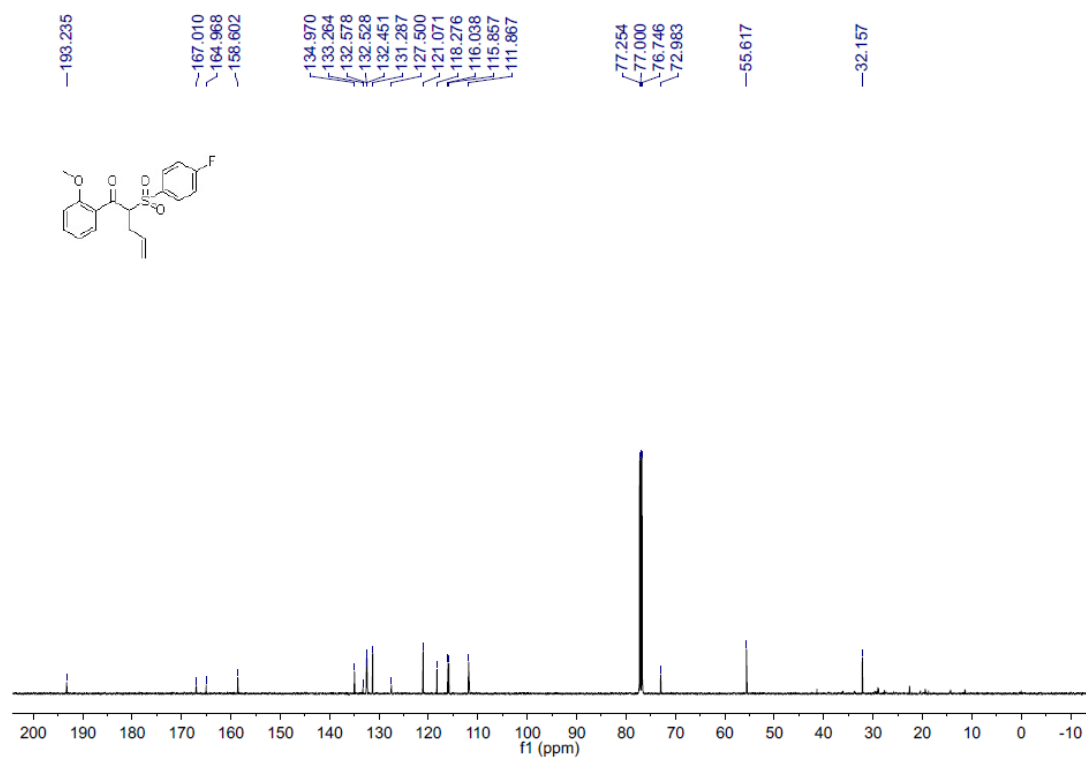
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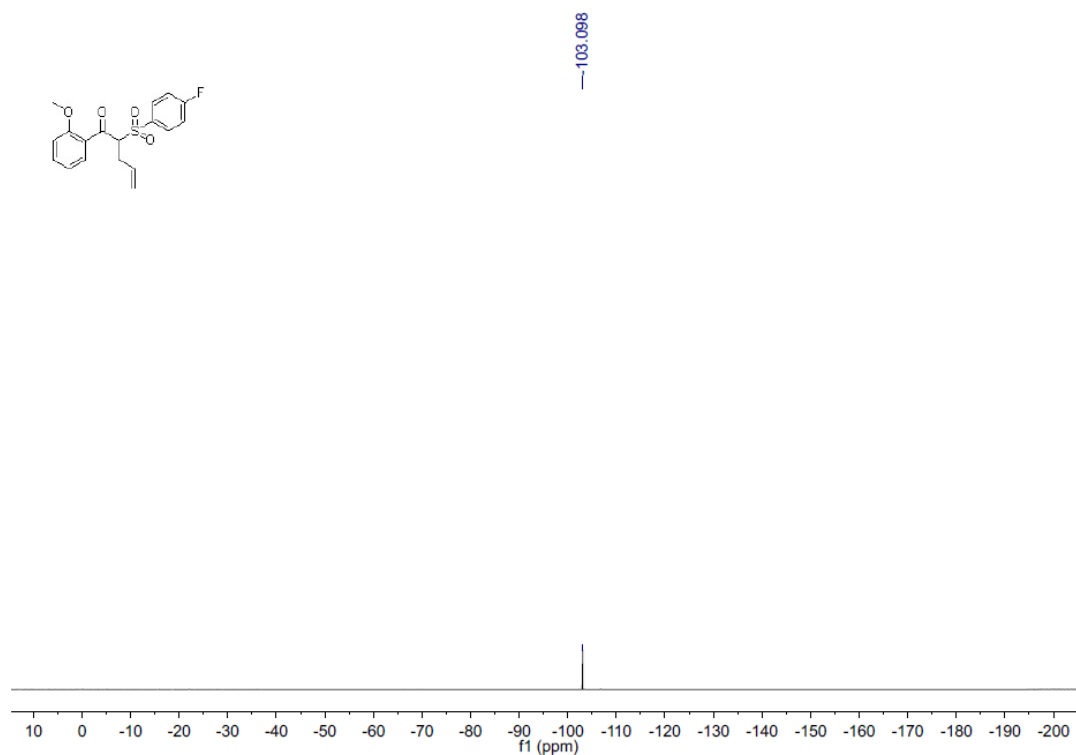
^1H NMR (500 MHz, CDCl_3) spectrum of compound **1x**



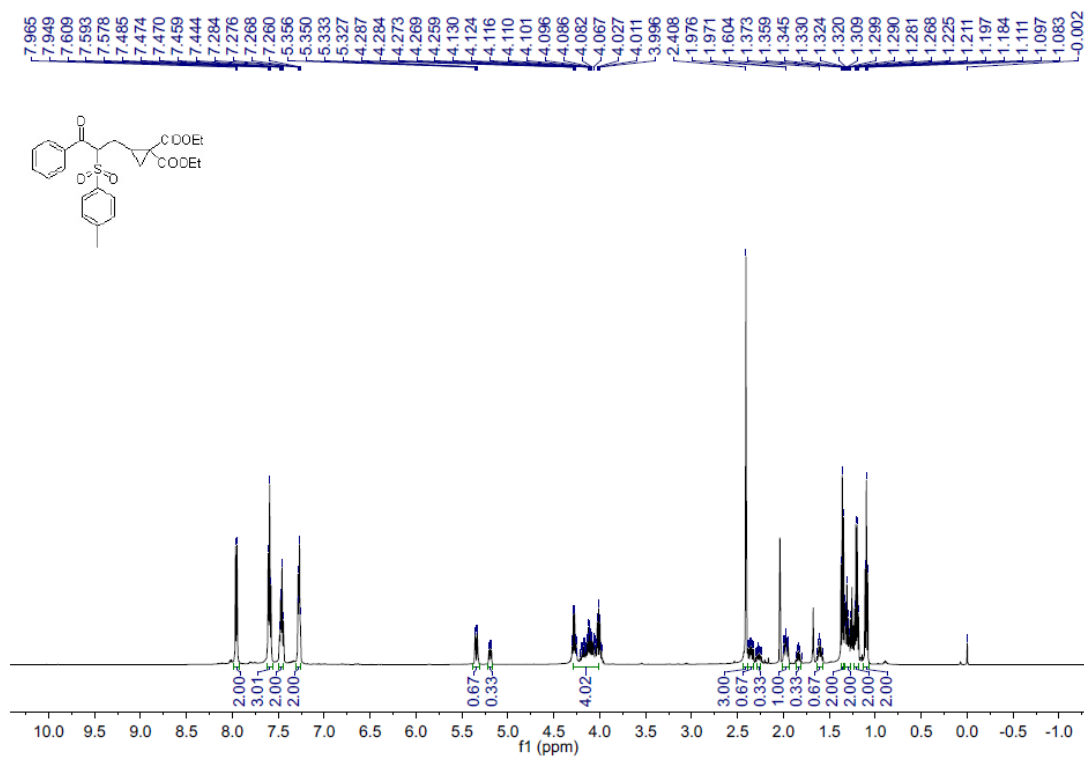
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **1x**



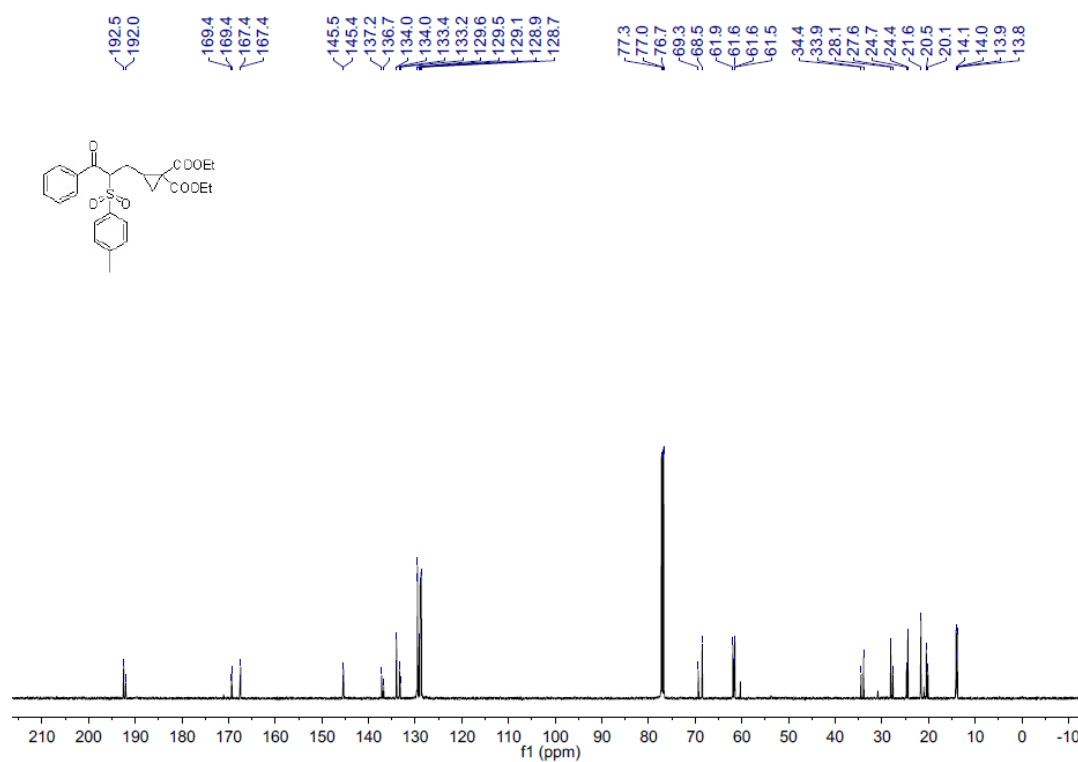
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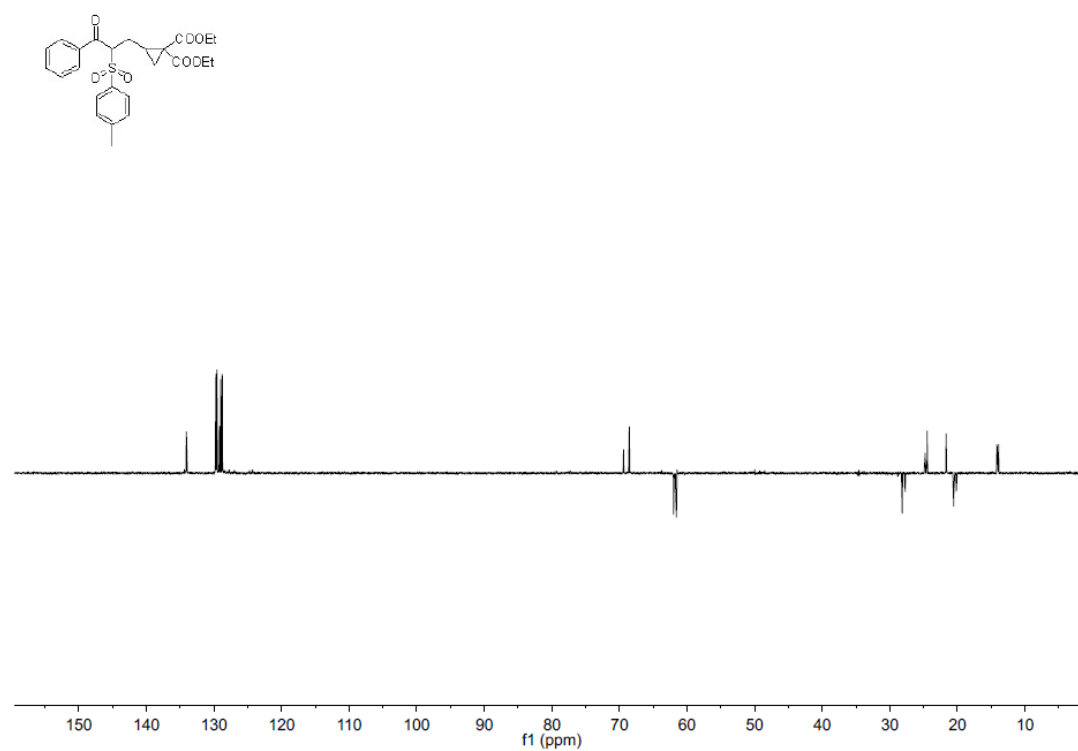
^1H NMR (500 MHz, CDCl_3) spectrum of compound **3a**



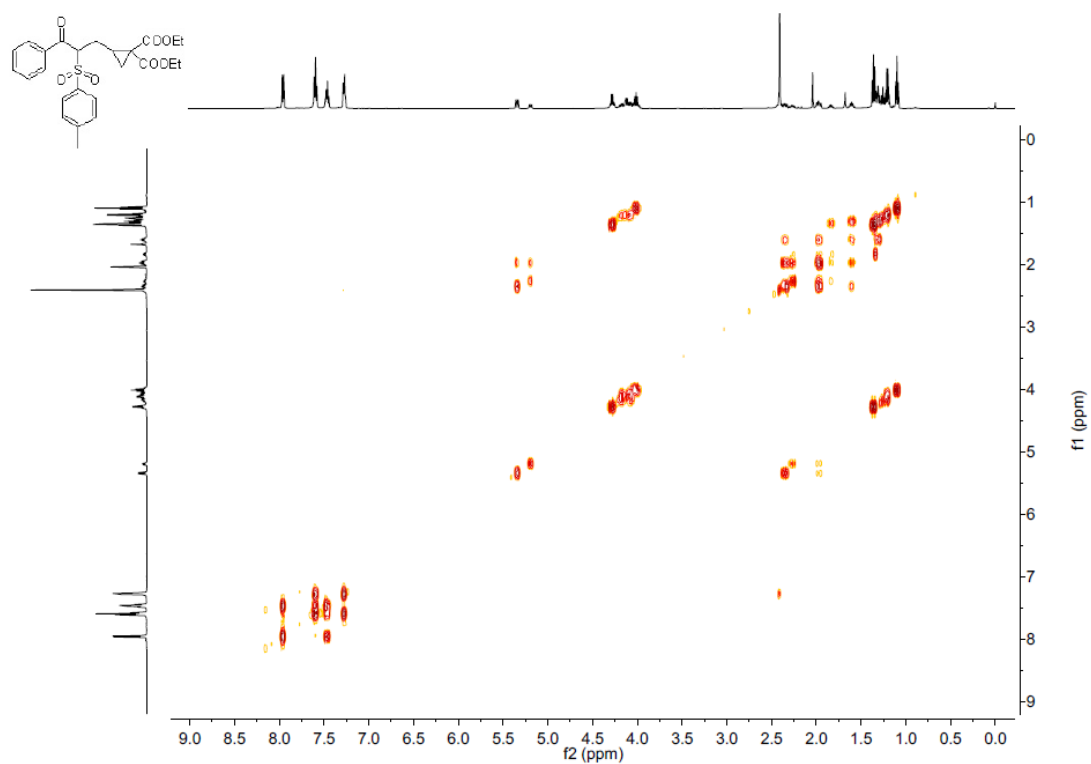
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **3a**



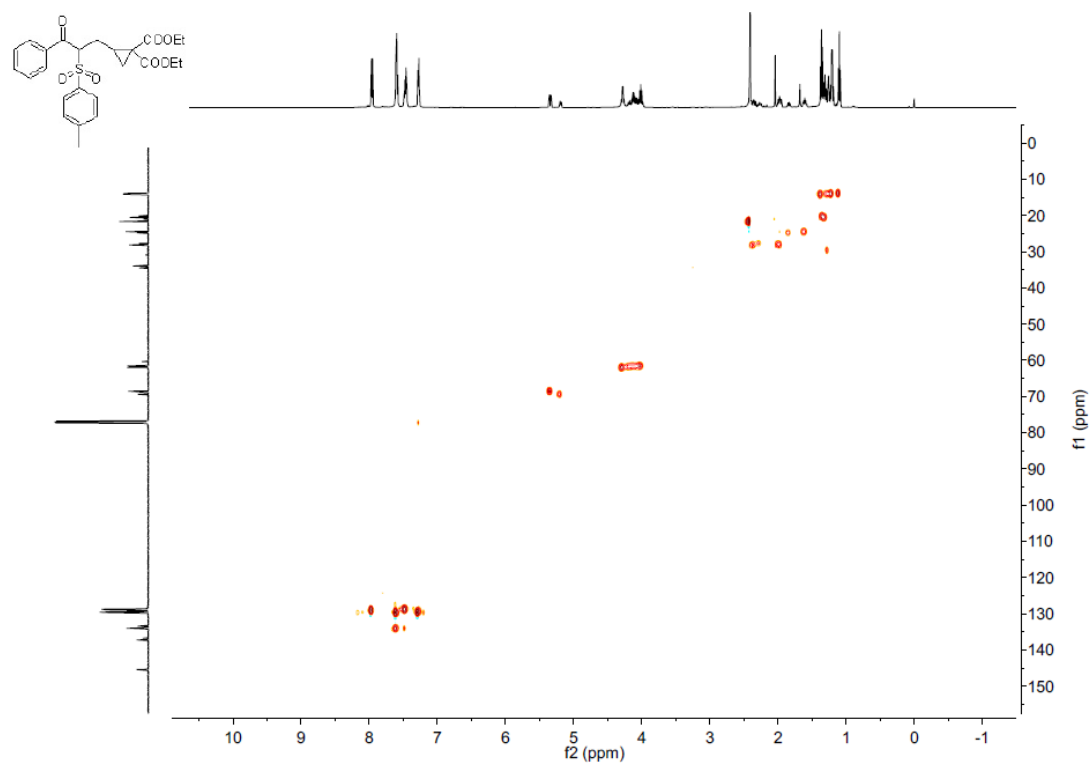
DEPT 135 NMR (CDCl_3) spectrum of compound **3a**



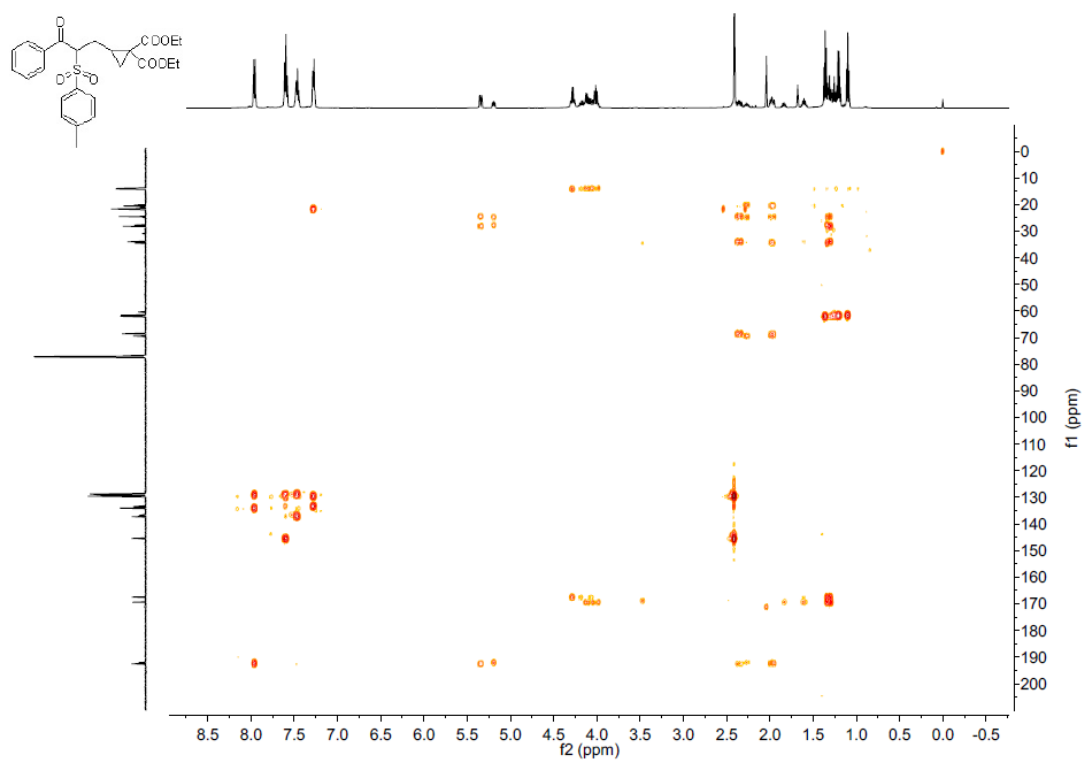
^1H - ^1H COSY NMR (CDCl_3) spectrum of compound **3a**



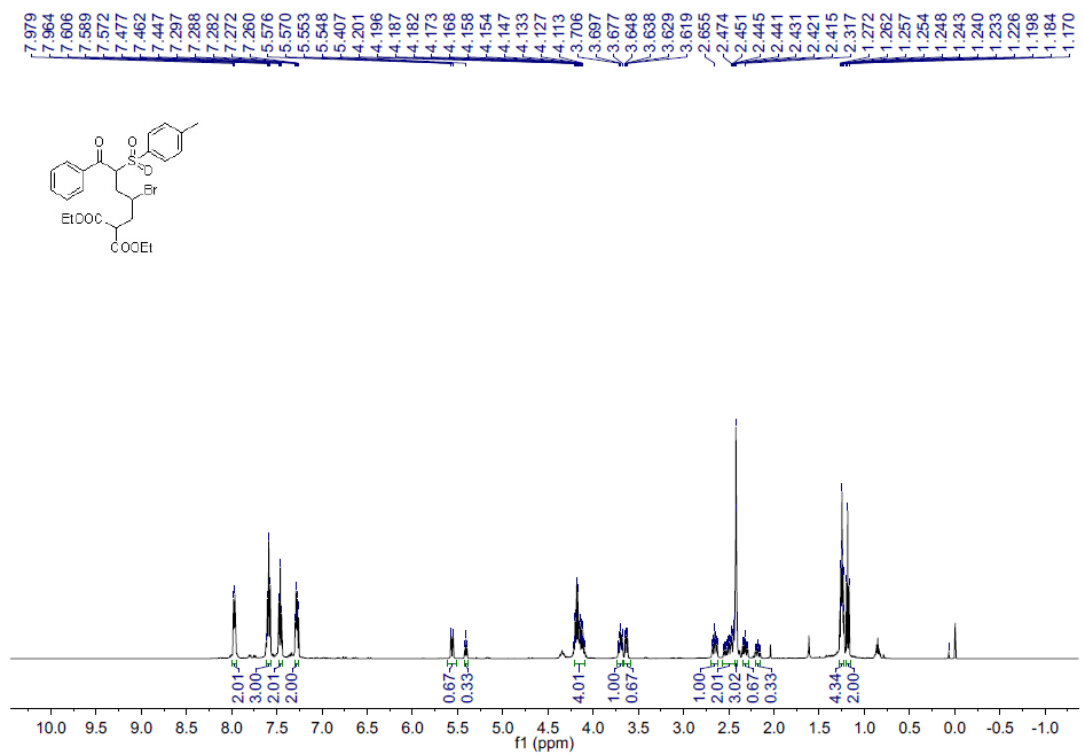
HSQC NMR (CDCl_3) spectrum of compound **3a**



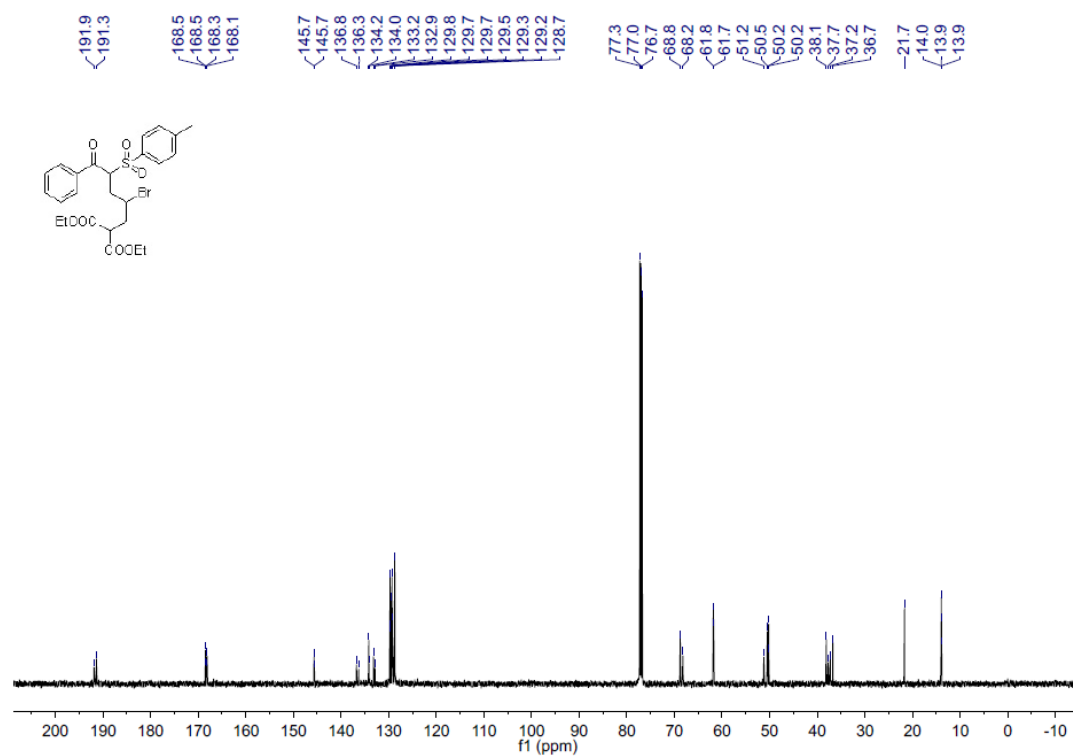
HMBC NMR (CDCl₃) spectrum of compound **3a**



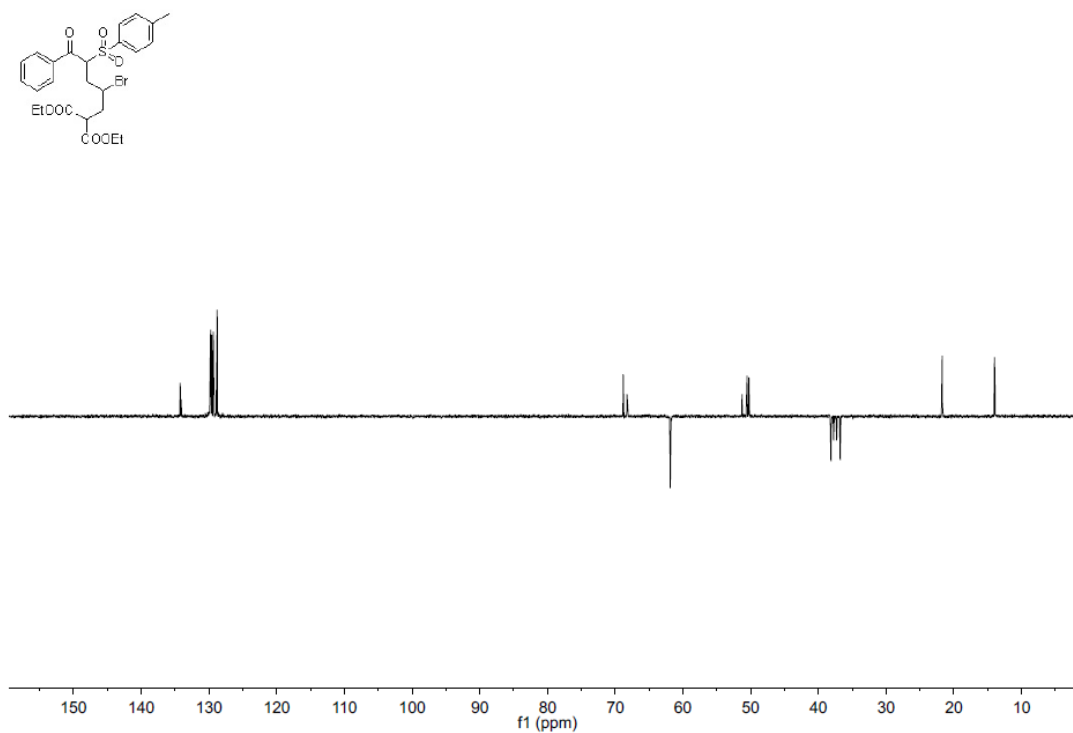
¹H NMR (500 MHz, CDCl₃) spectrum of compound **3a'**



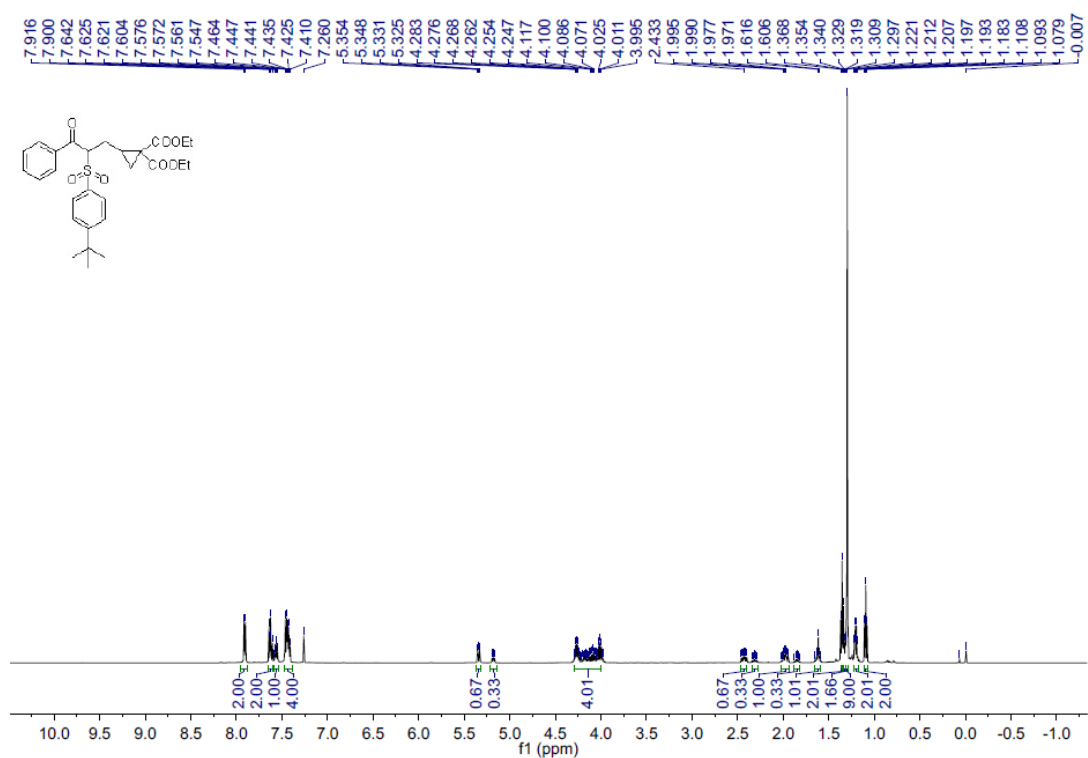
^{13}C NMR (126 MHz, CDCl_3) spectrum of compound **3a'**



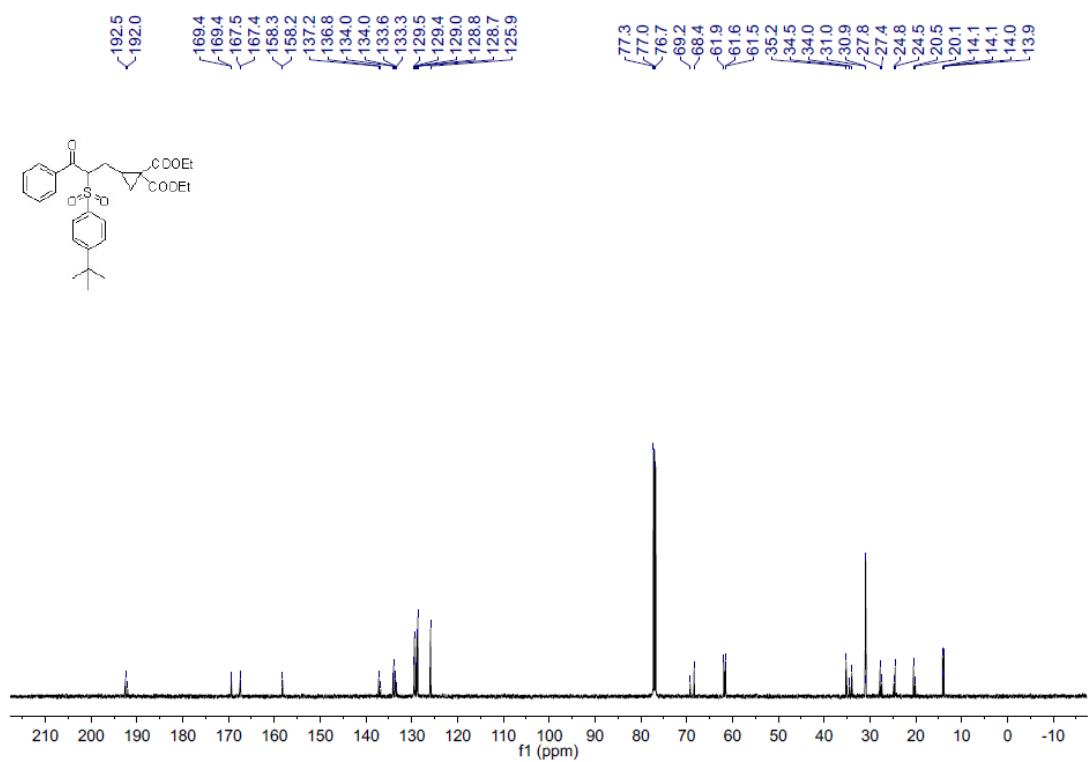
DEPT 135 NMR (CDCl_3) spectrum of compound **3a'**



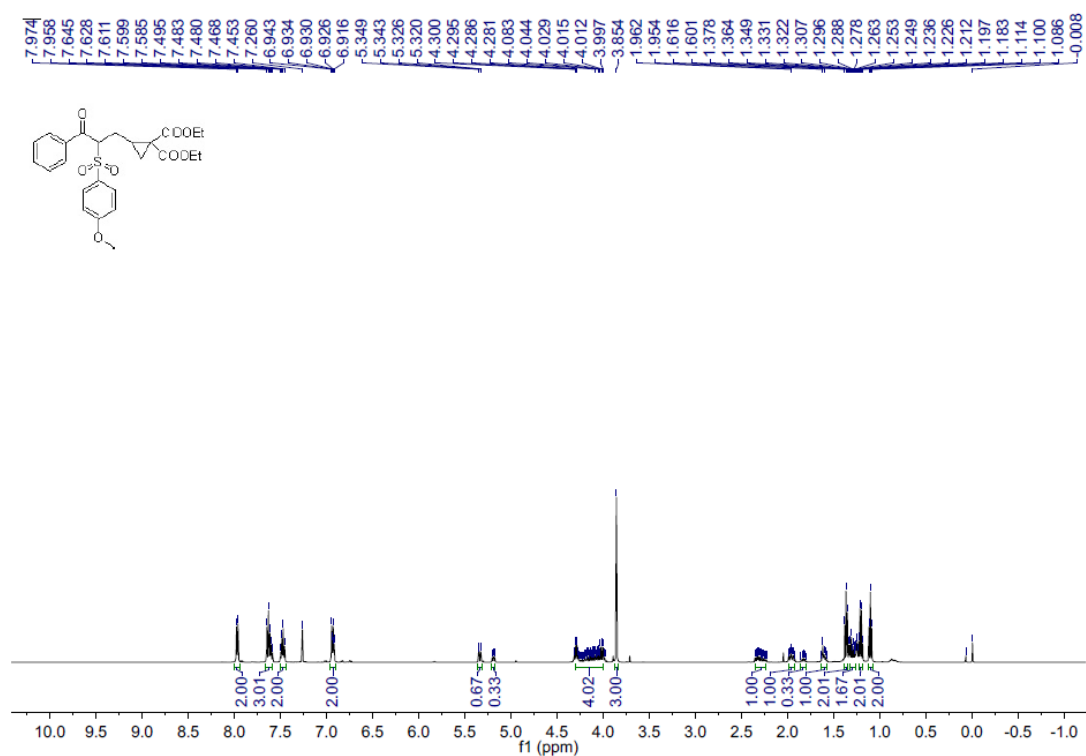
^1H NMR (500 MHz, CDCl_3) spectrum of compound **3b**



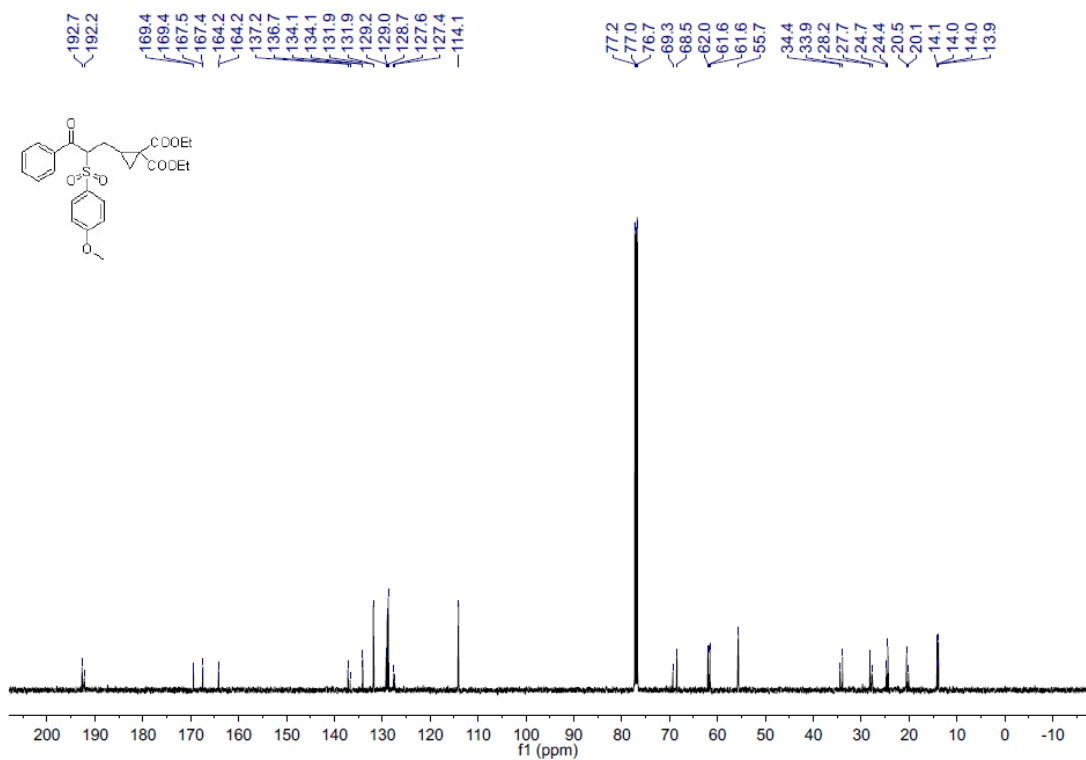
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **3b**



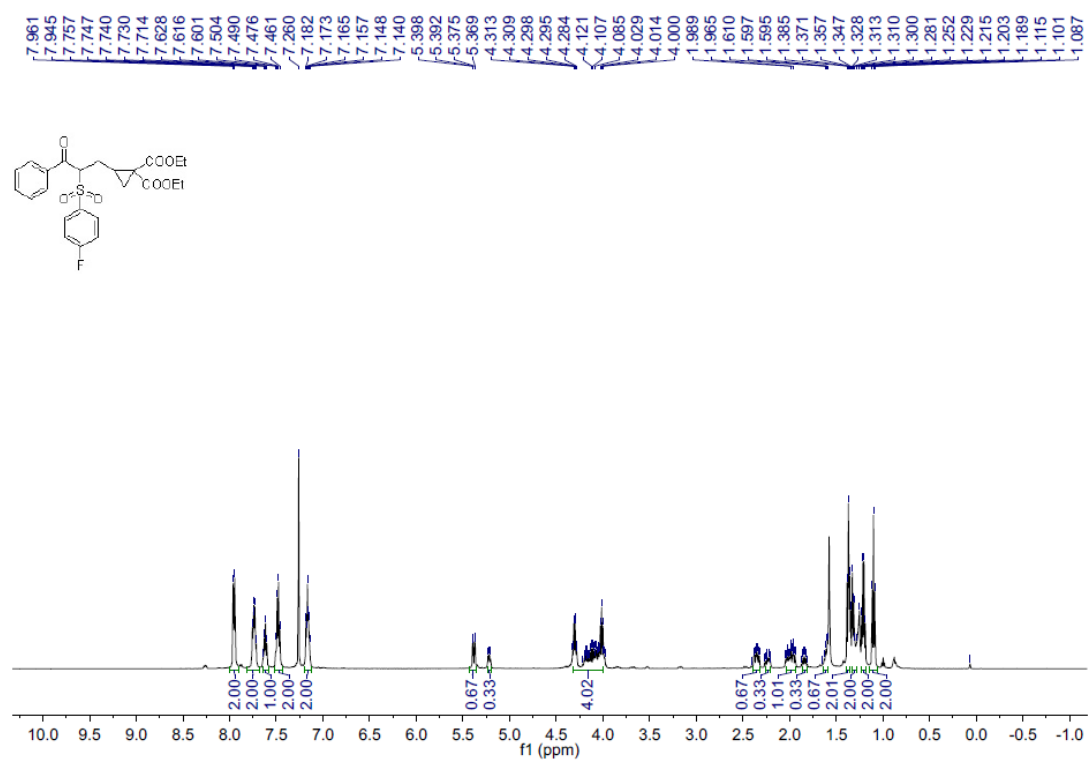
^1H NMR (500 MHz, CDCl_3) spectrum of compound **3c**



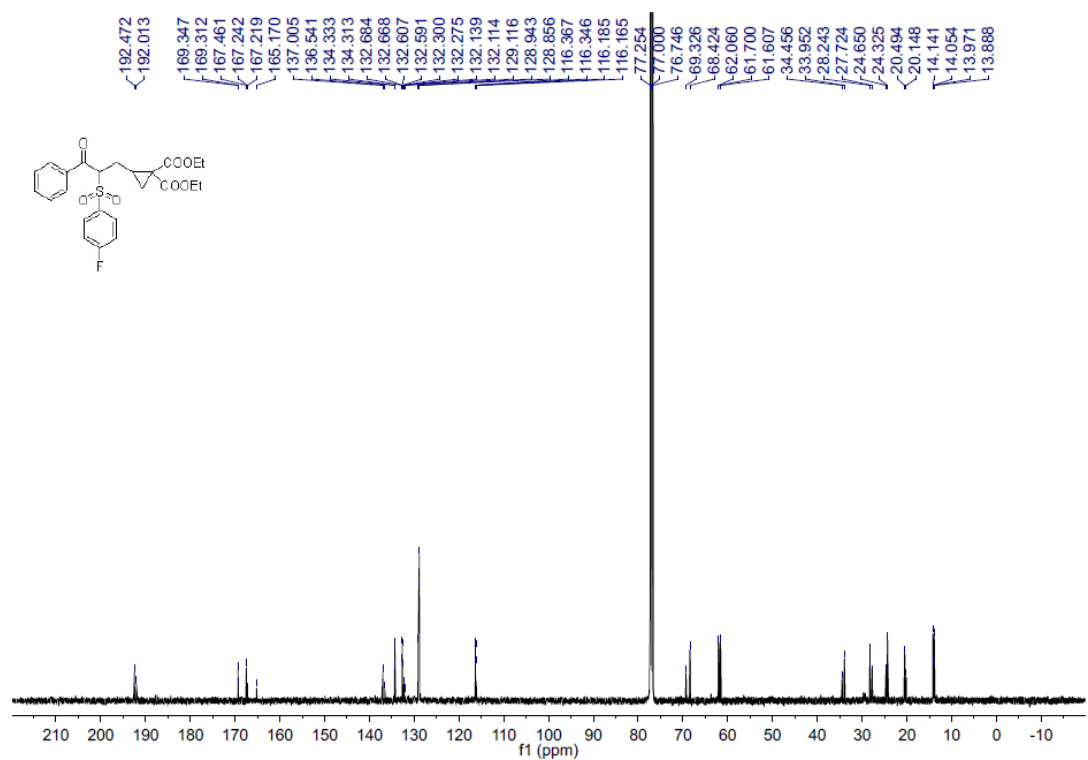
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **3c**



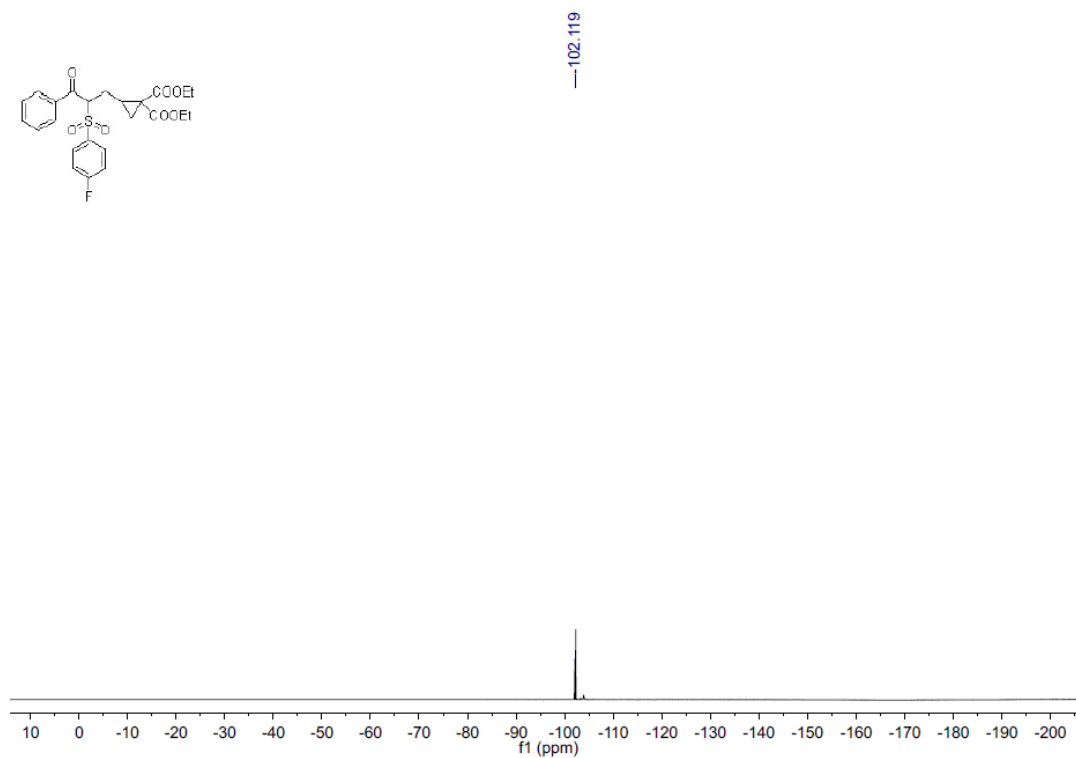
^1H NMR (500 MHz, CDCl_3) spectrum of compound **3d**



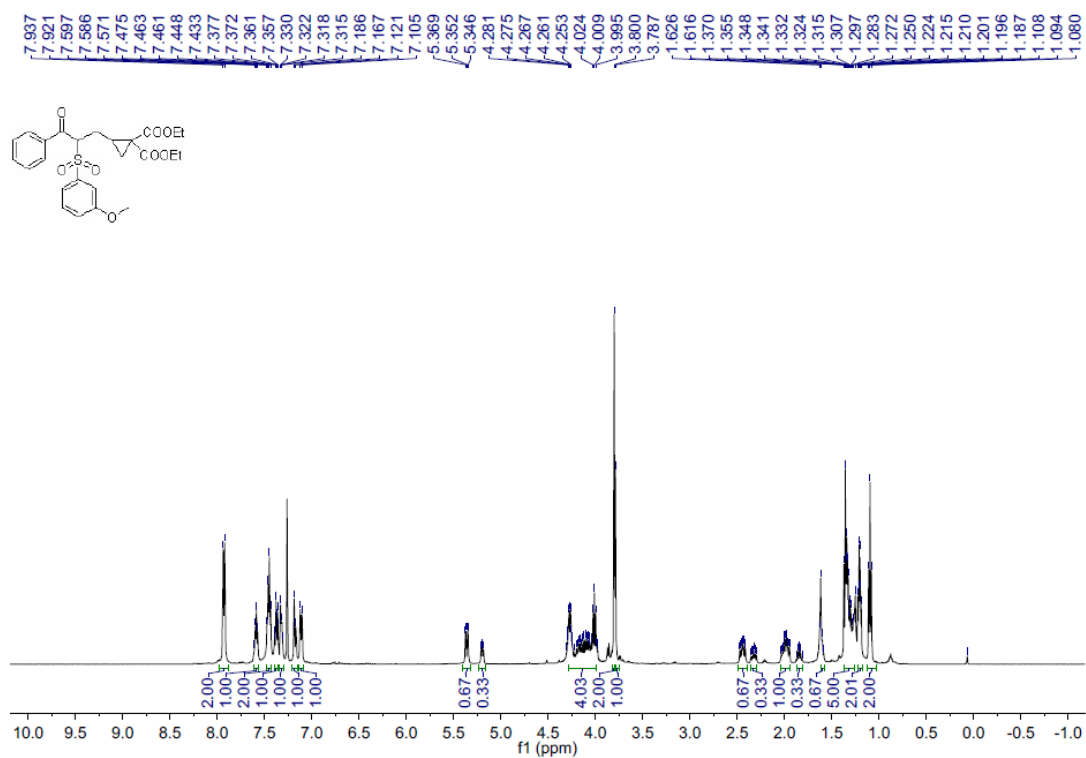
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **3d**



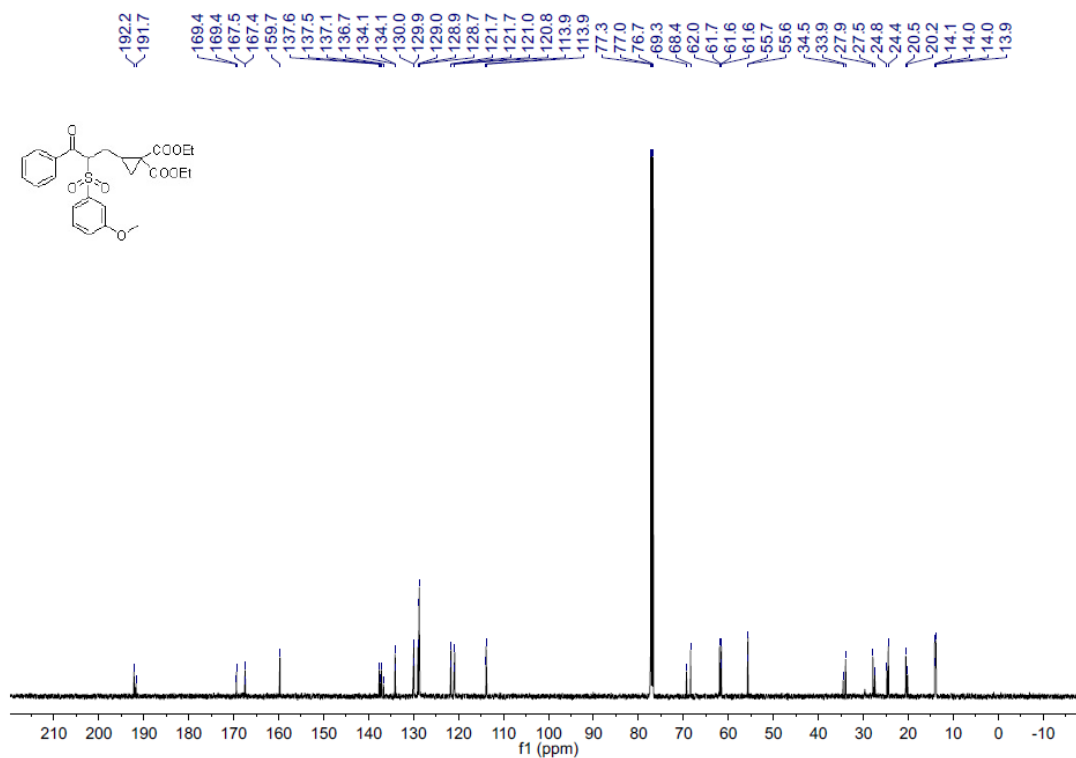
^{19}F NMR (470 MHz, CDCl_3) spectrum of compound **3d**



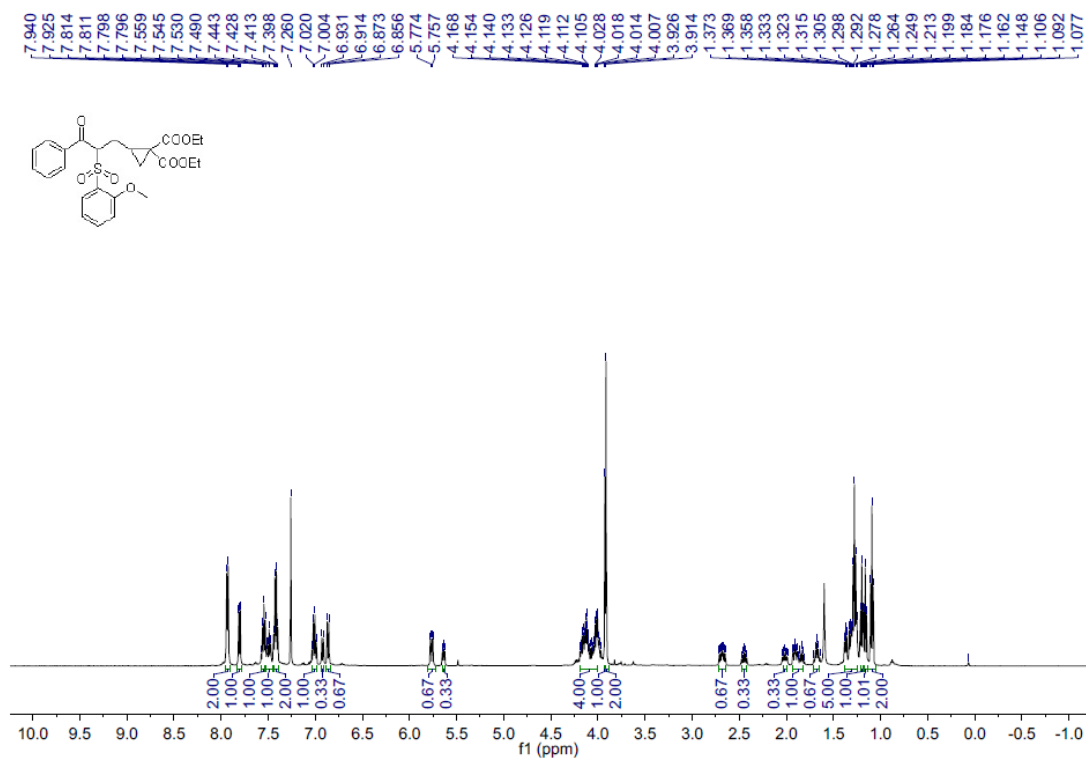
^1H NMR (500 MHz, CDCl_3) spectrum of compound **3e**



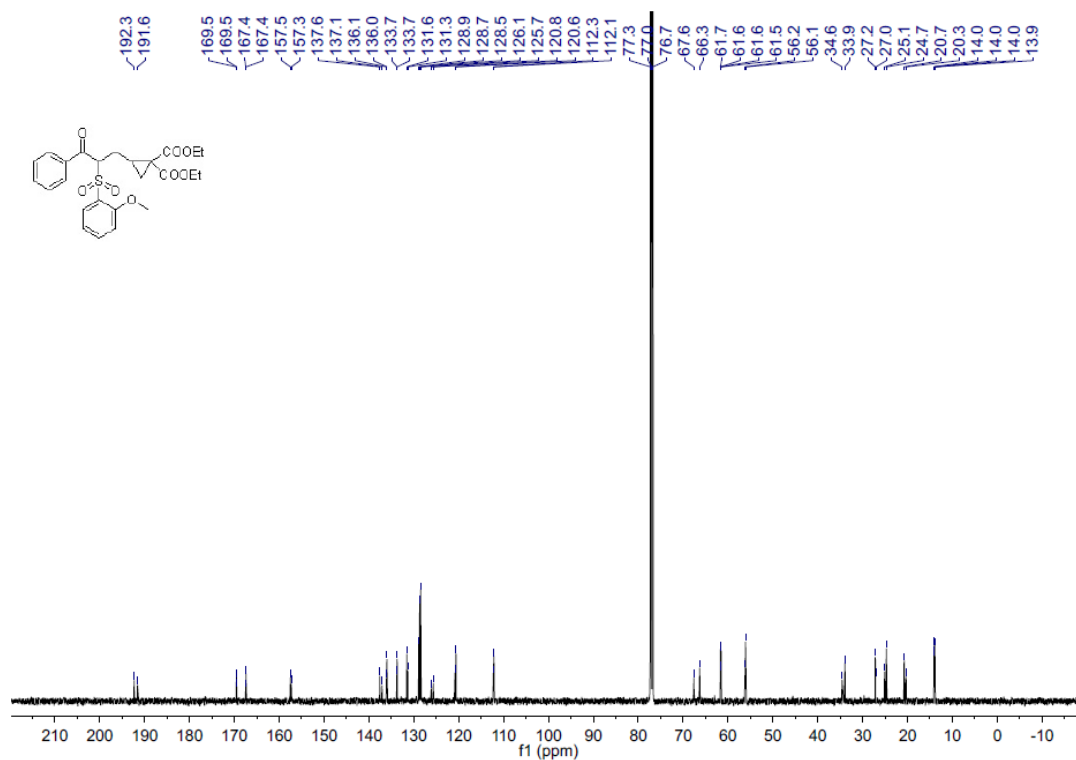
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **3e**



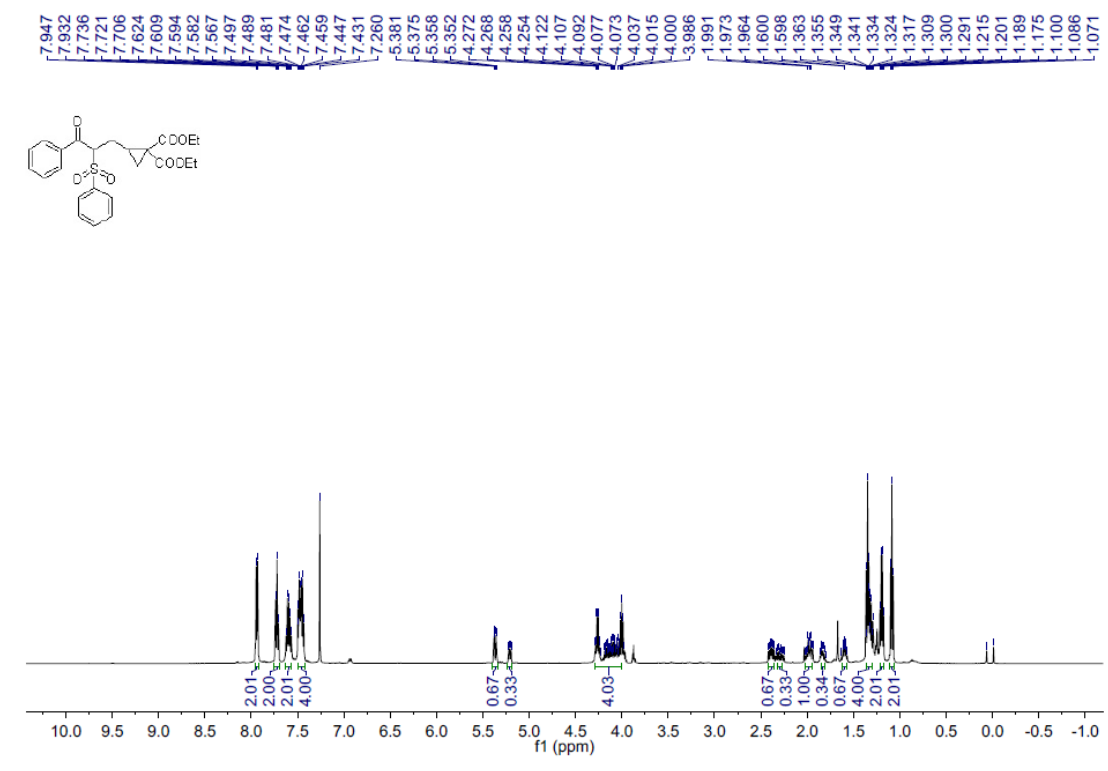
^1H NMR (500 MHz, CDCl_3) spectrum of compound **3f**



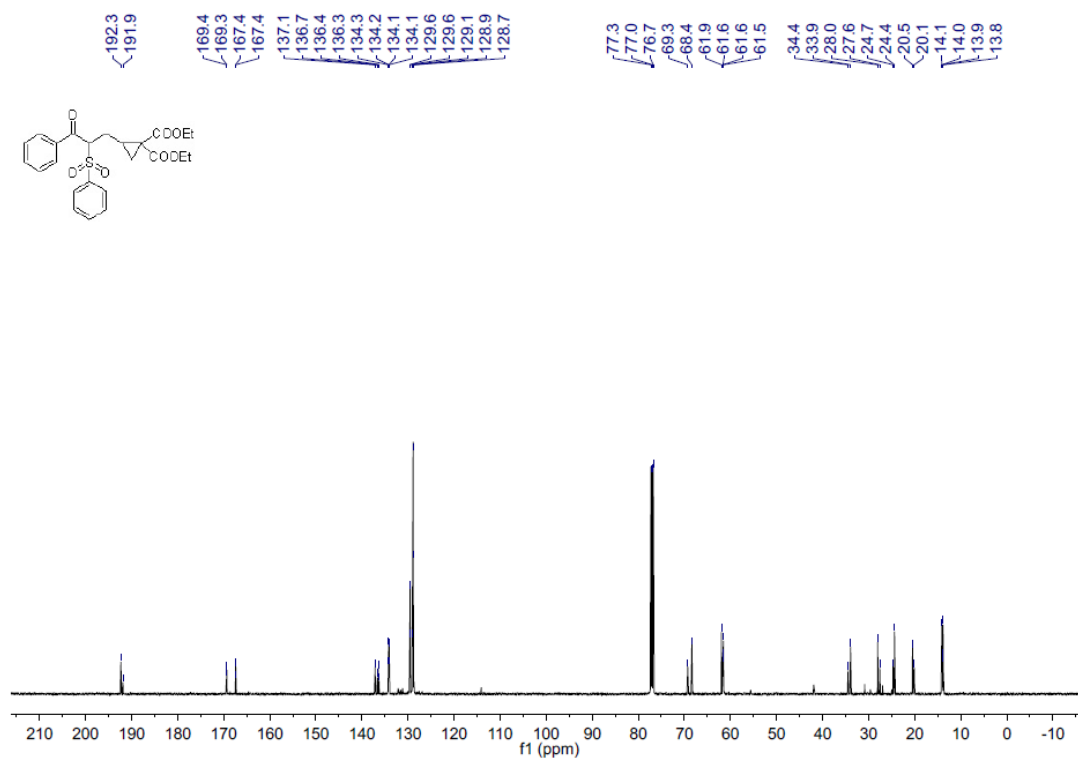
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **3f**



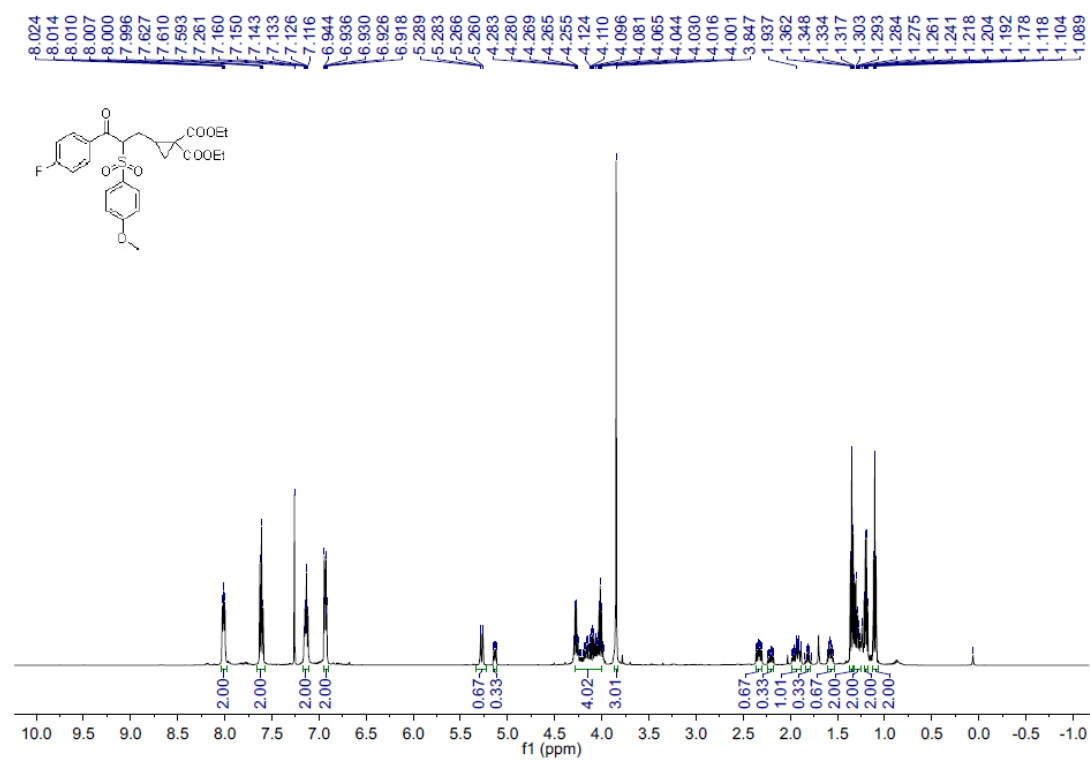
^1H NMR (500 MHz, CDCl_3) spectrum of compound **3g**



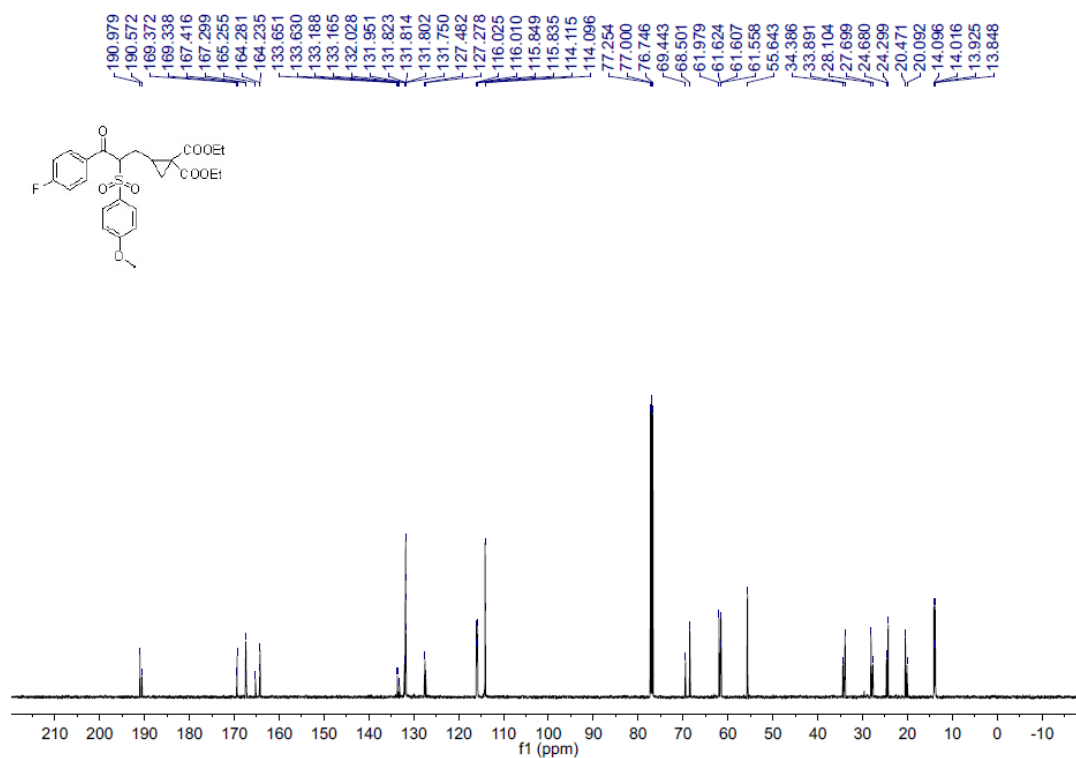
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **3g**



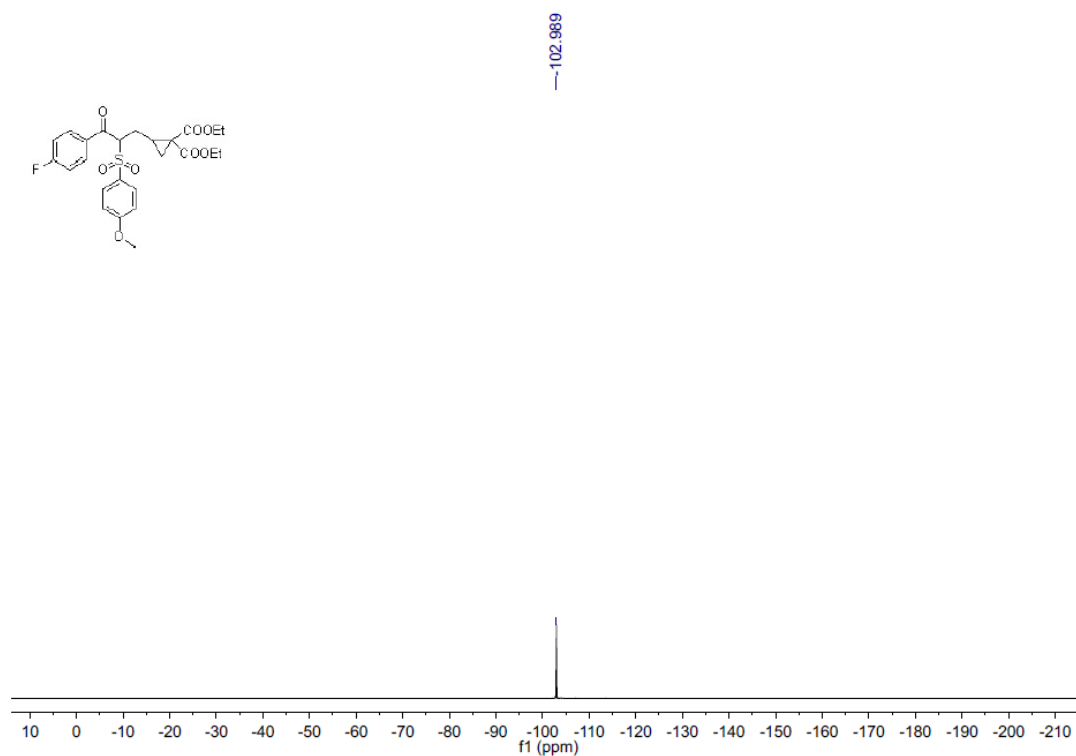
^1H NMR (500 MHz, CDCl_3) spectrum of compound **3h**



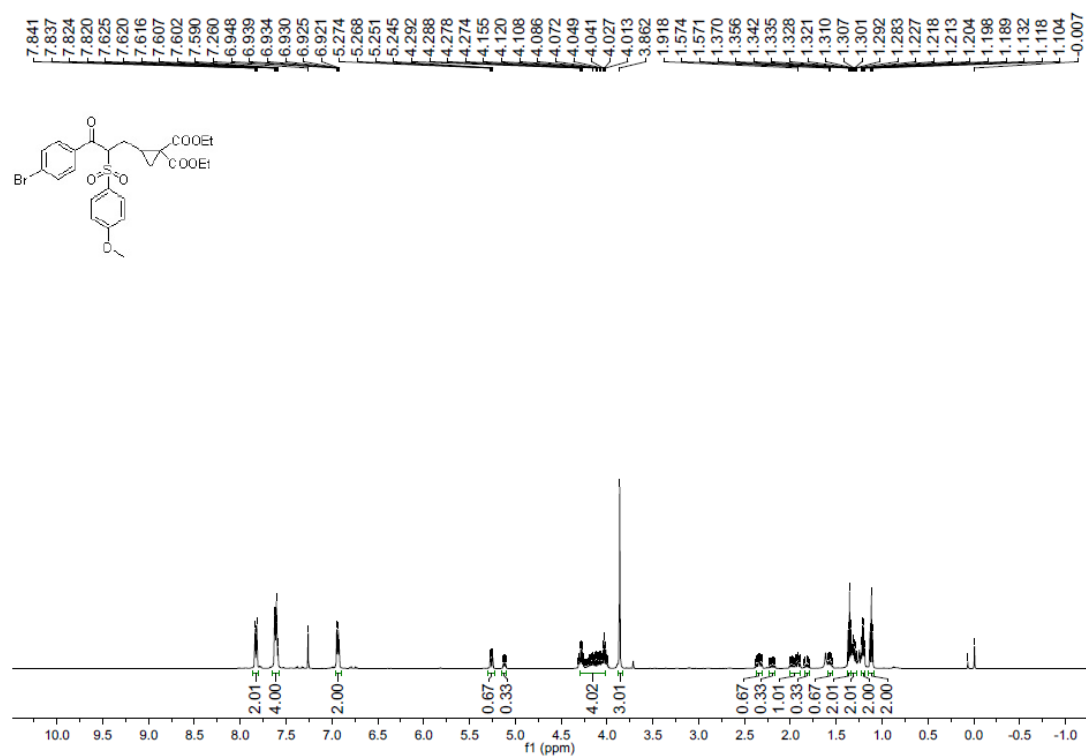
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **3h**



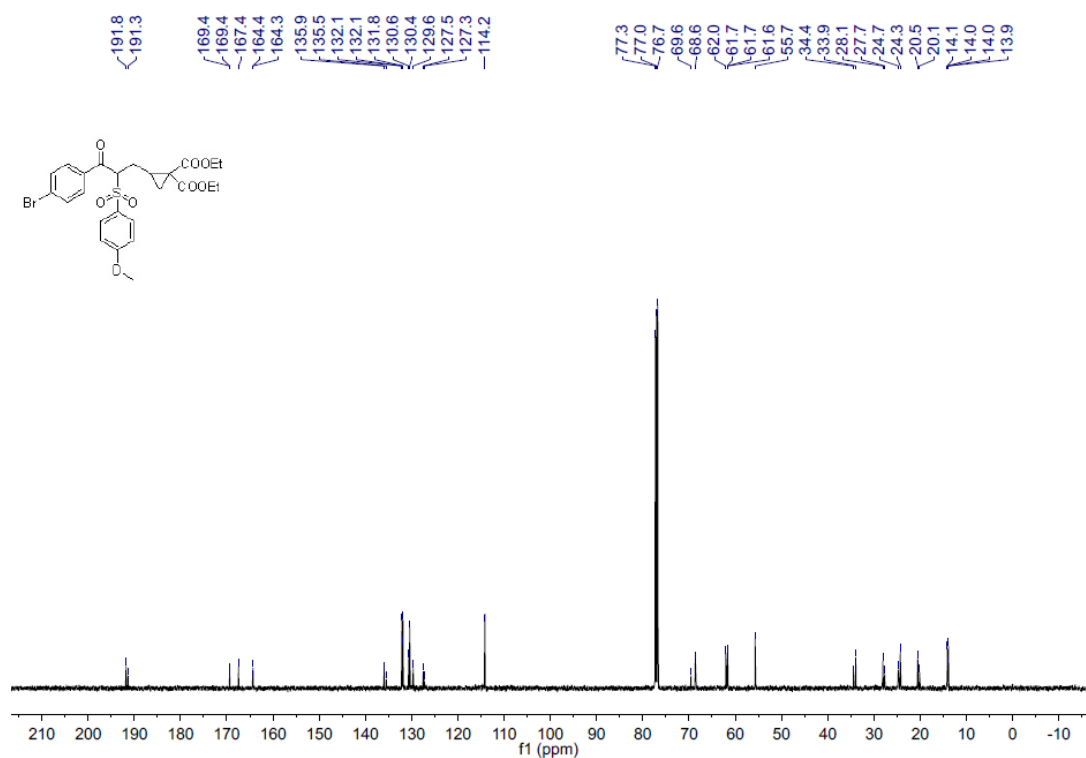
^{19}F NMR (470 MHz, CDCl_3) spectrum of compound **3h**



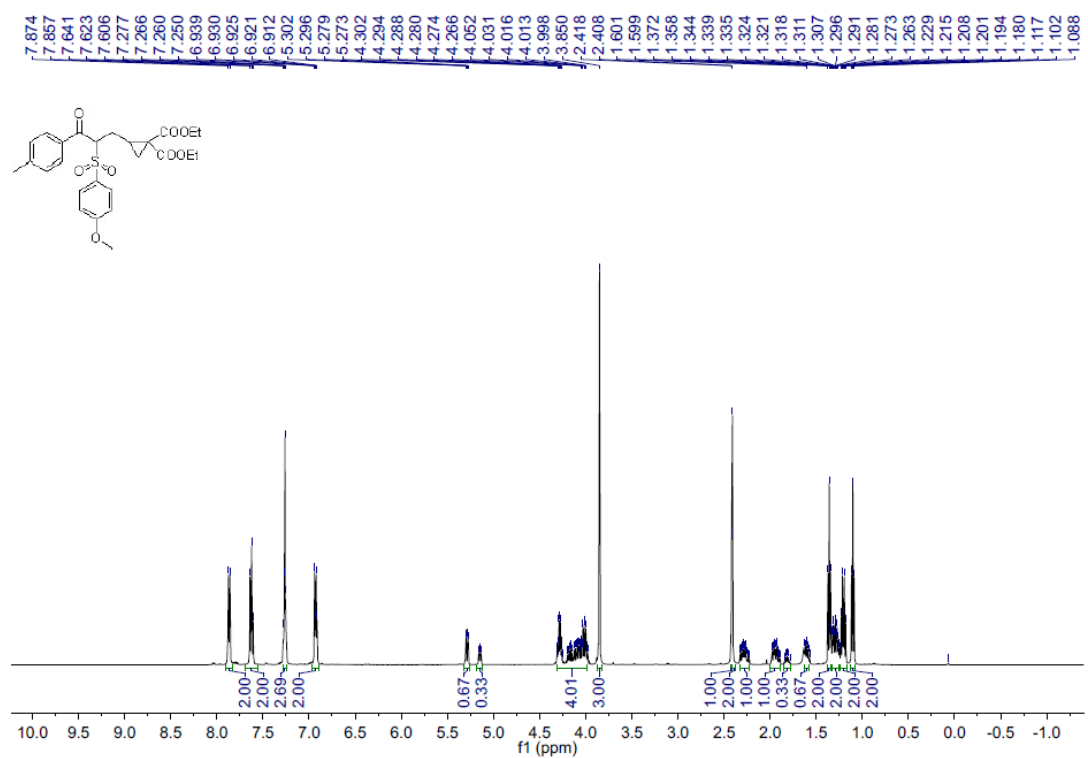
^1H NMR (500 MHz, CDCl_3) spectrum of compound **3i**



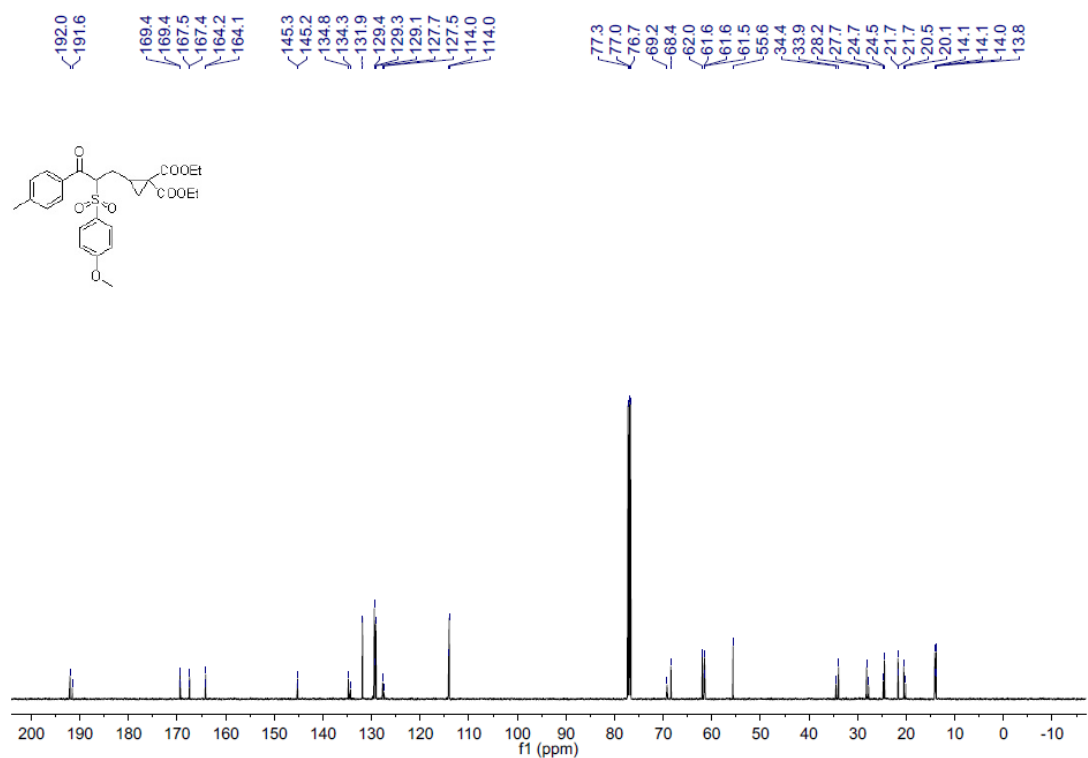
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **3i**



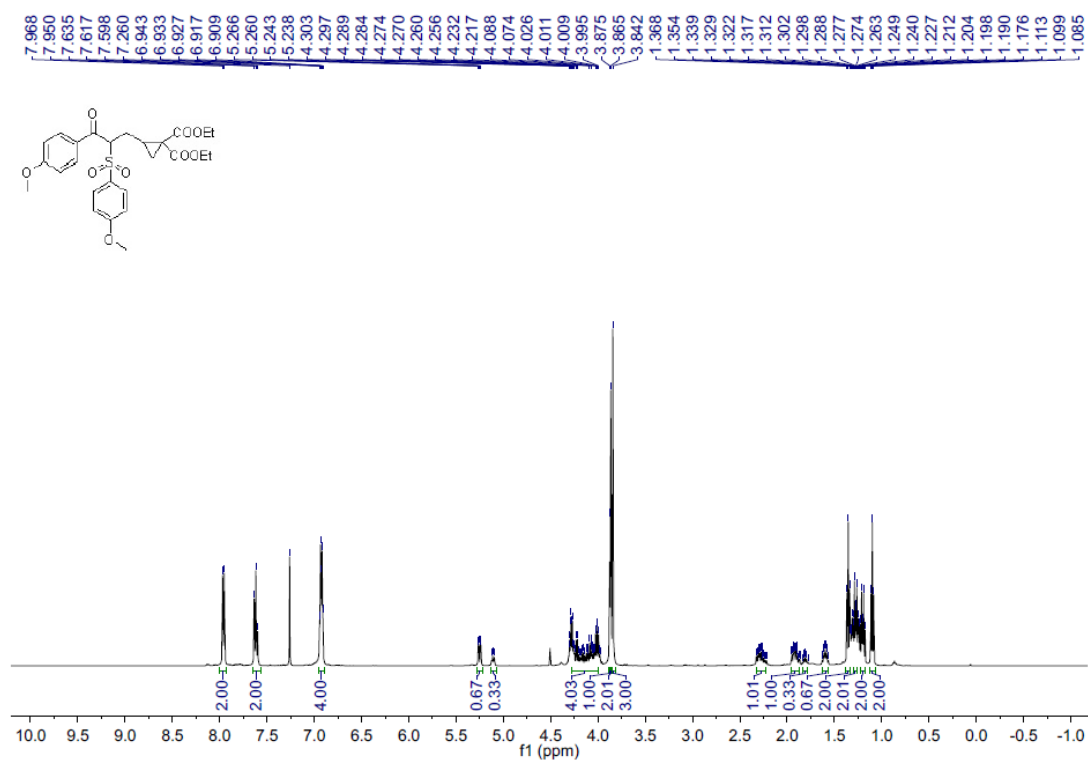
^1H NMR (500 MHz, CDCl_3) spectrum of compound **3j**



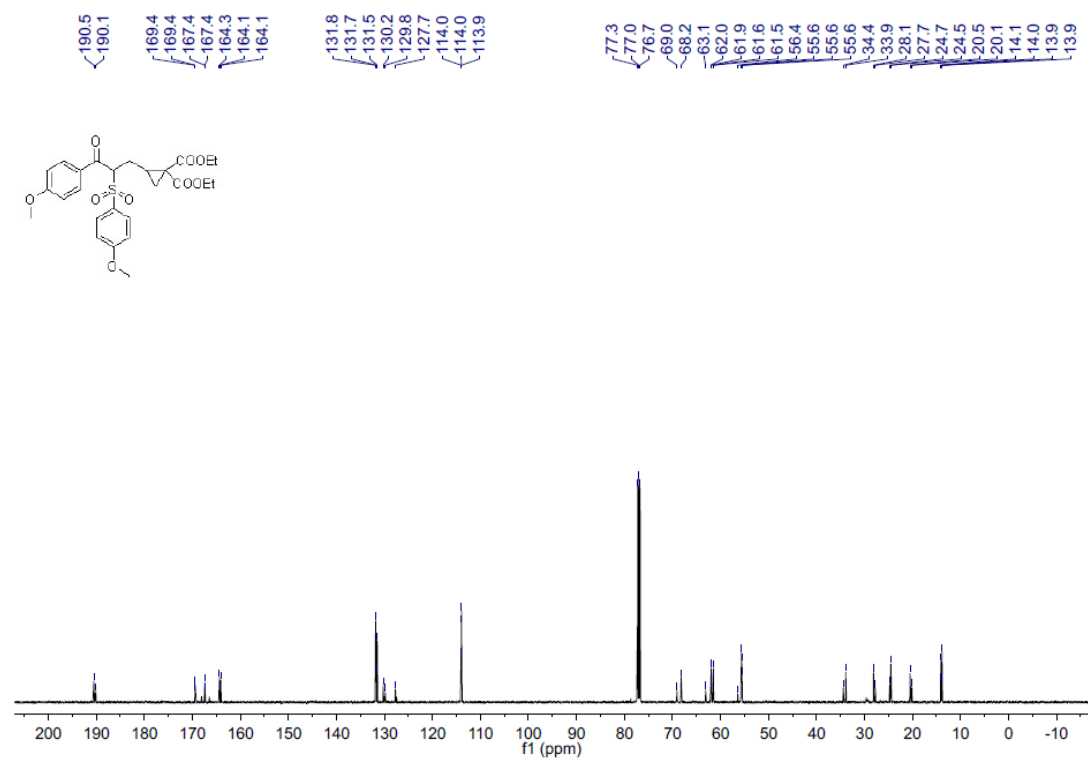
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **3j**



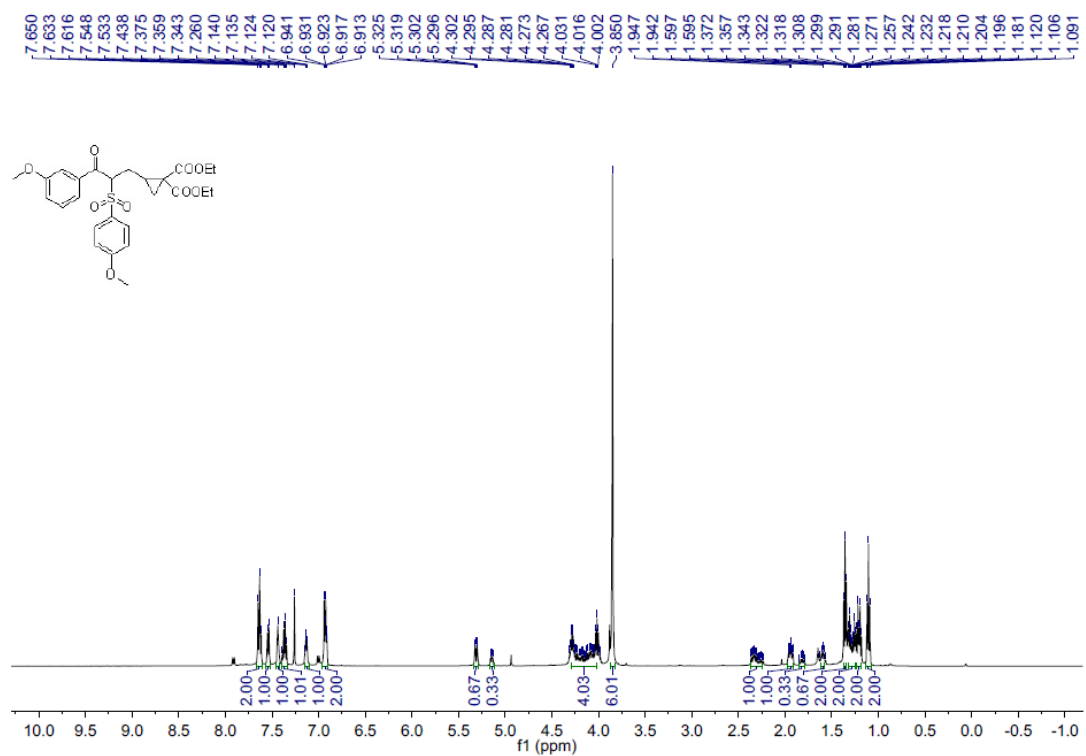
^1H NMR (500 MHz, CDCl_3) spectrum of compound **3k**



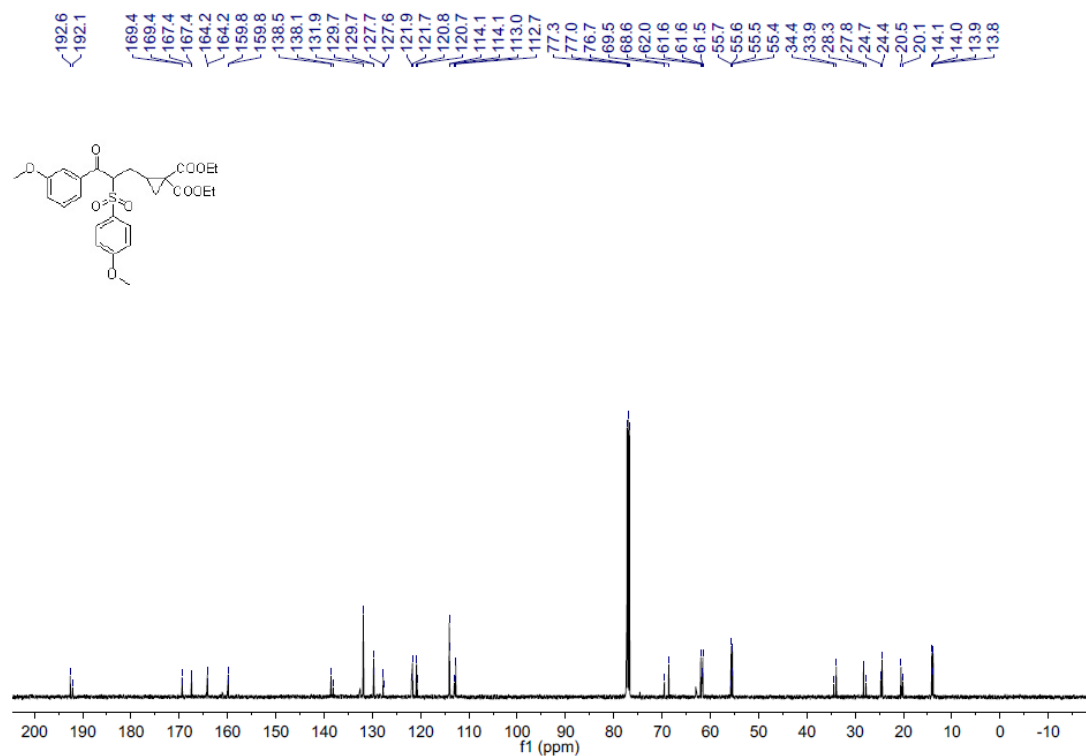
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **3k**



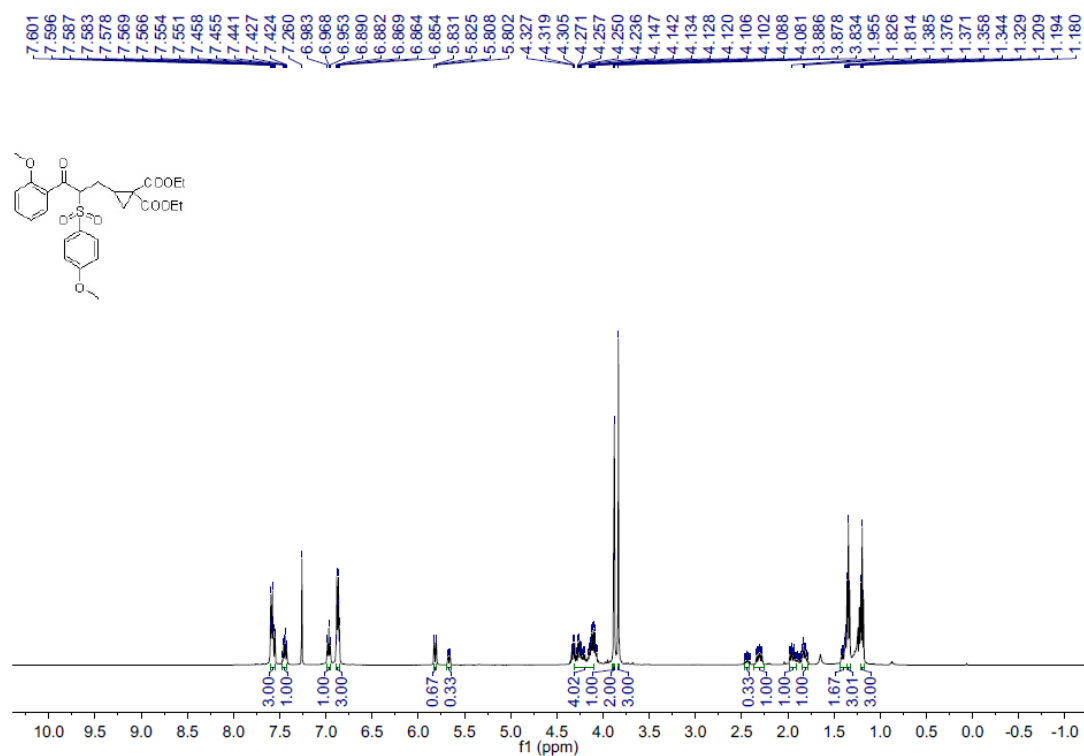
^1H NMR (500 MHz, CDCl_3) spectrum of compound **31**



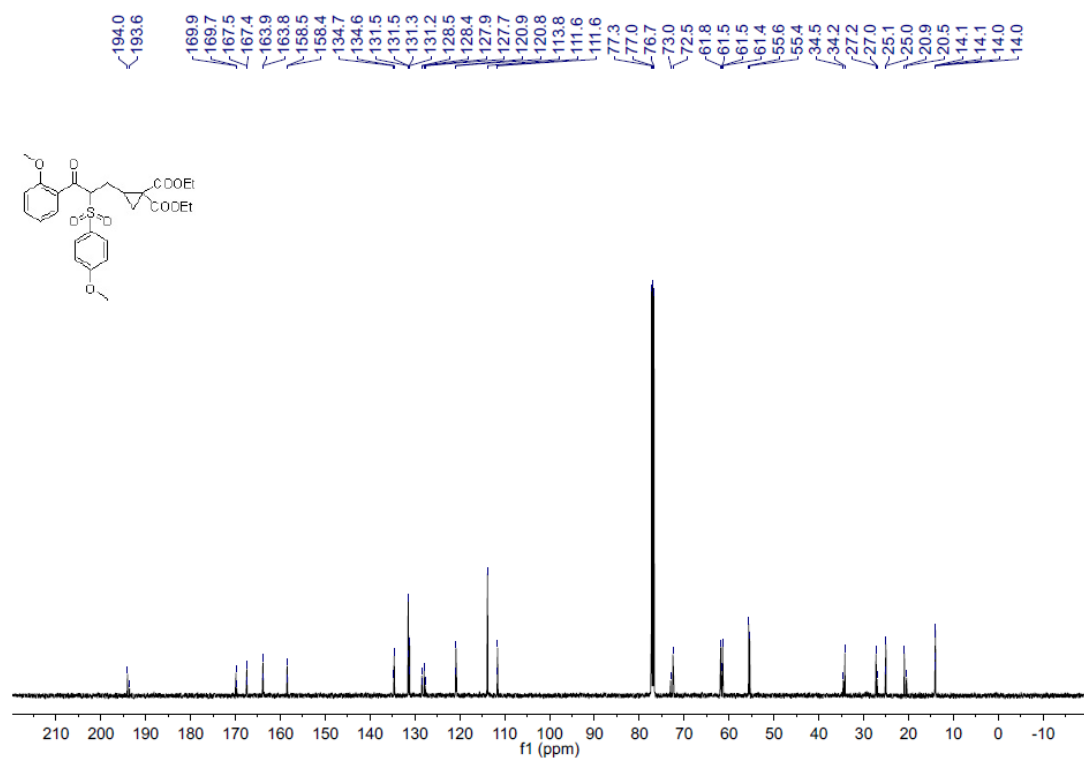
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **31**



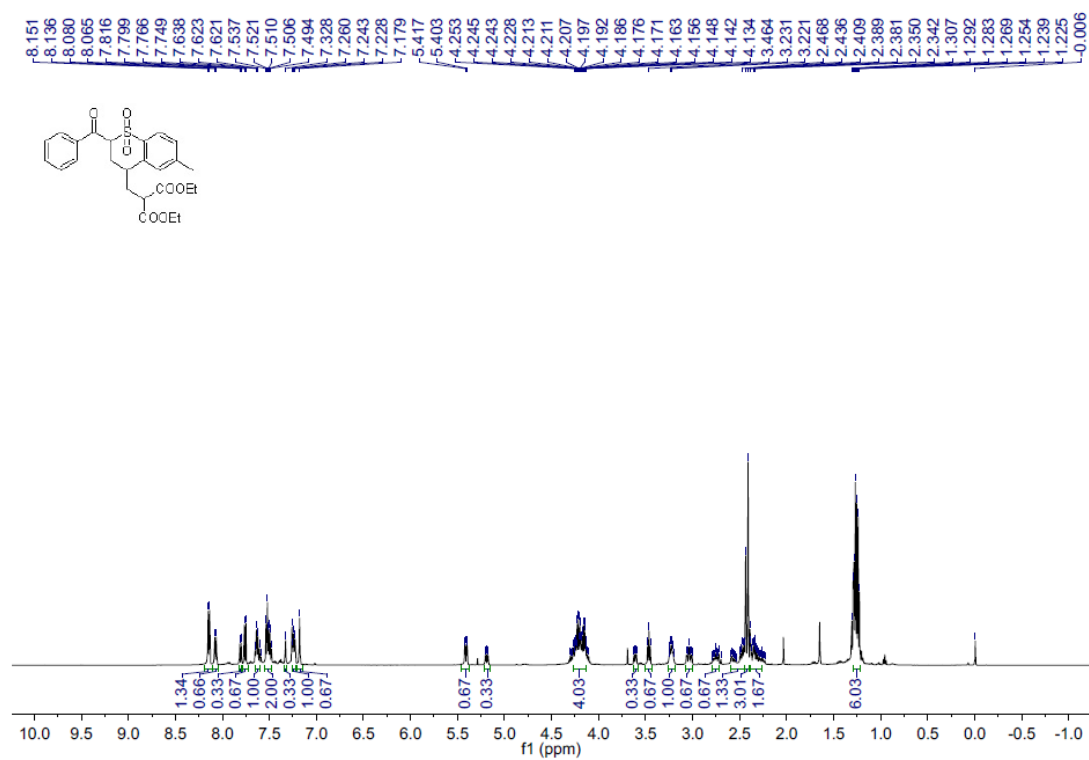
^1H NMR (500 MHz, CDCl_3) spectrum of compound **3m**



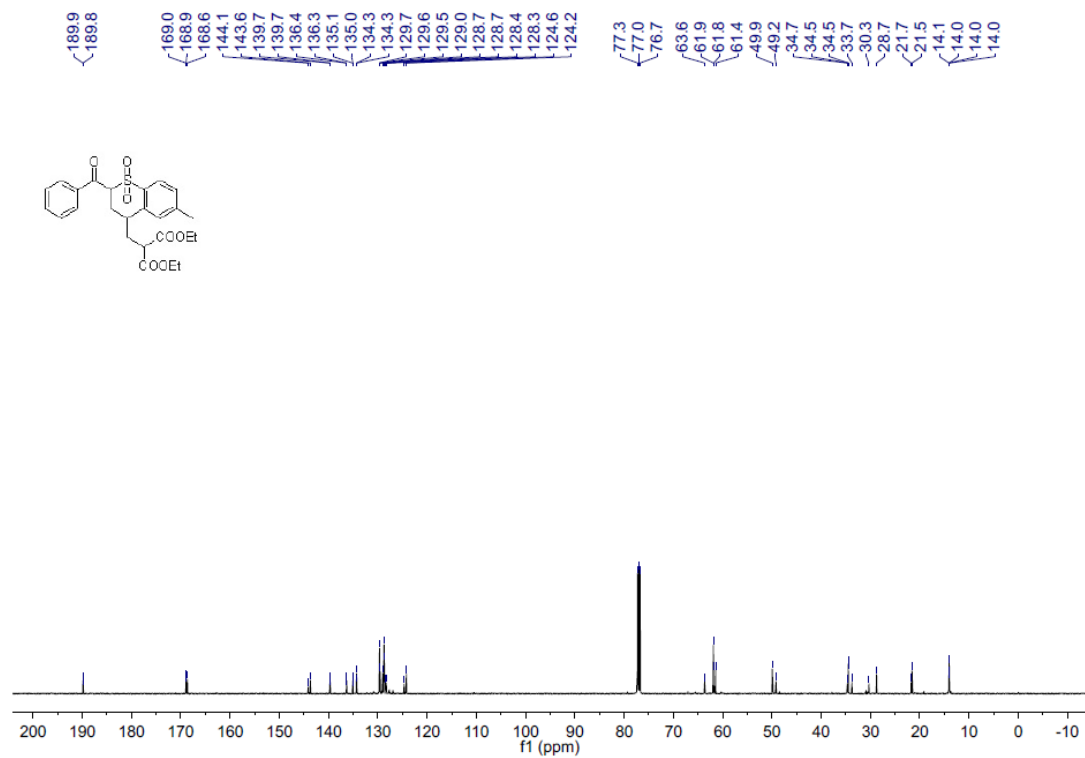
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **3m**



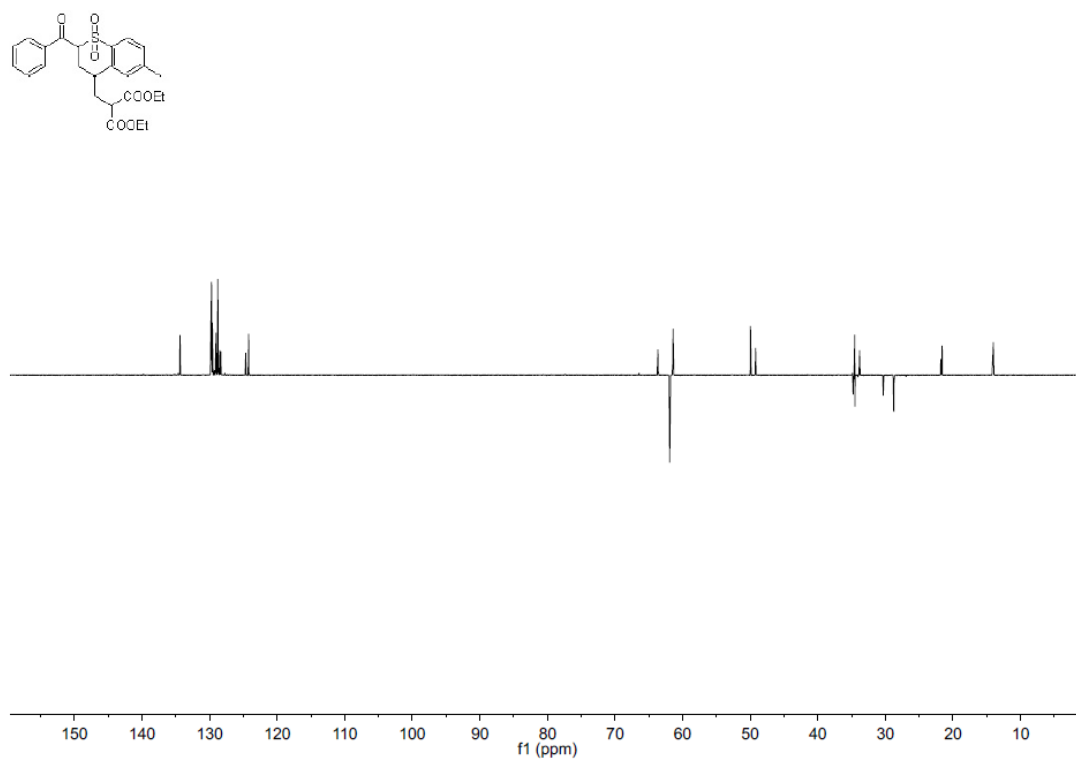
^1H NMR (500 MHz, CDCl_3) spectrum of compound **4a**



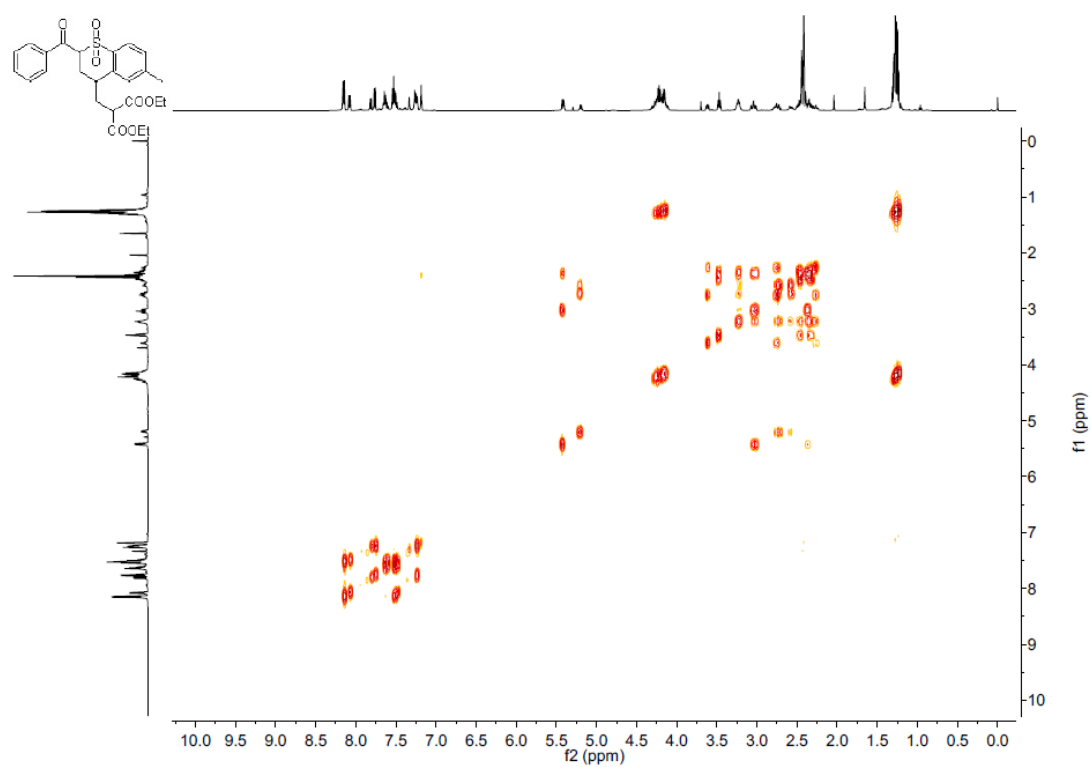
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **4a**



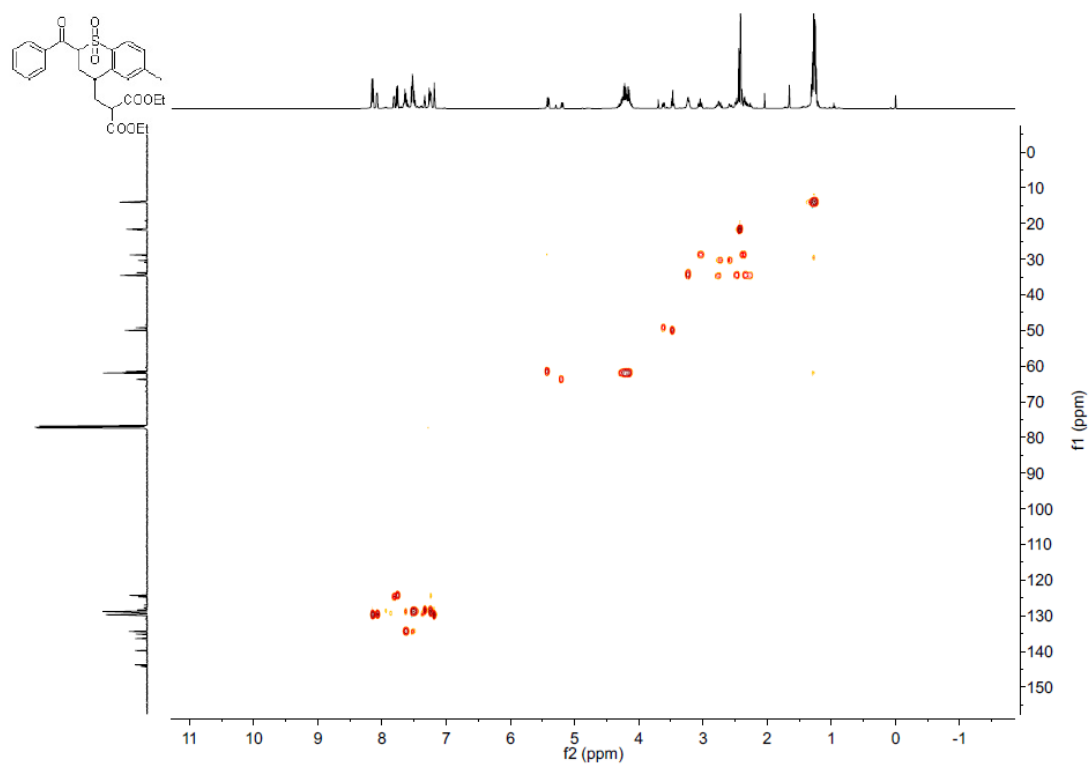
DEPT 135 NMR (CDCl₃) spectrum of compound **4a**



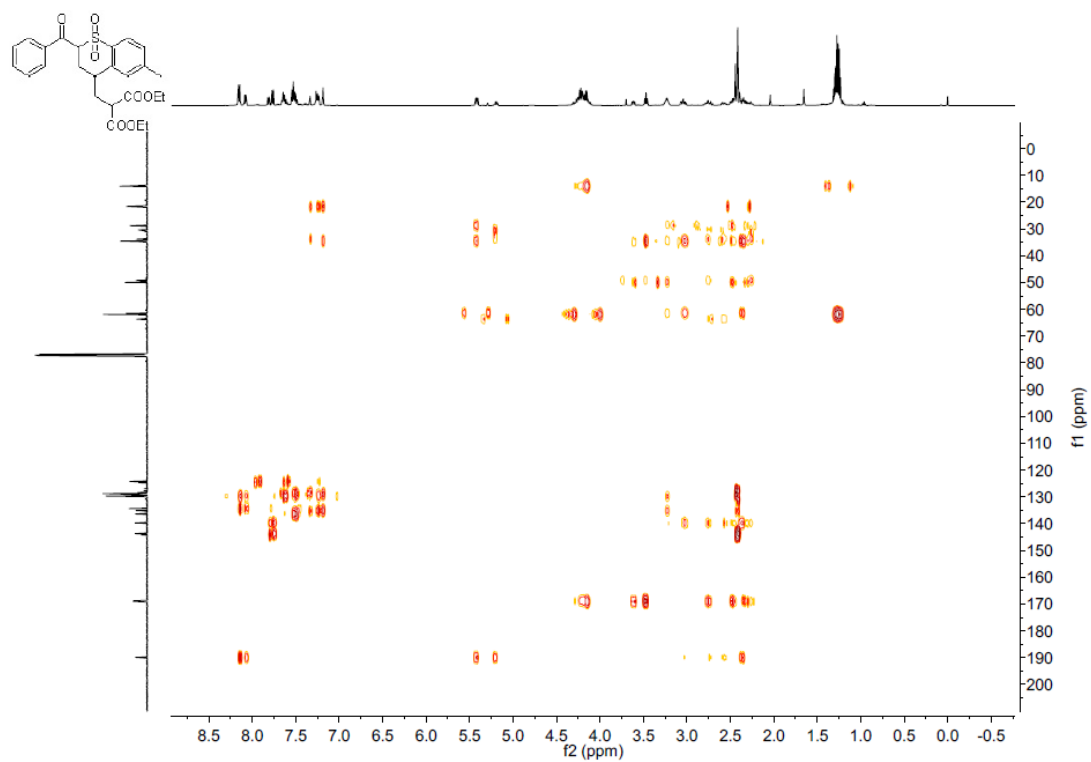
¹H-¹H COSY NMR (CDCl₃) spectrum of compound **4a**



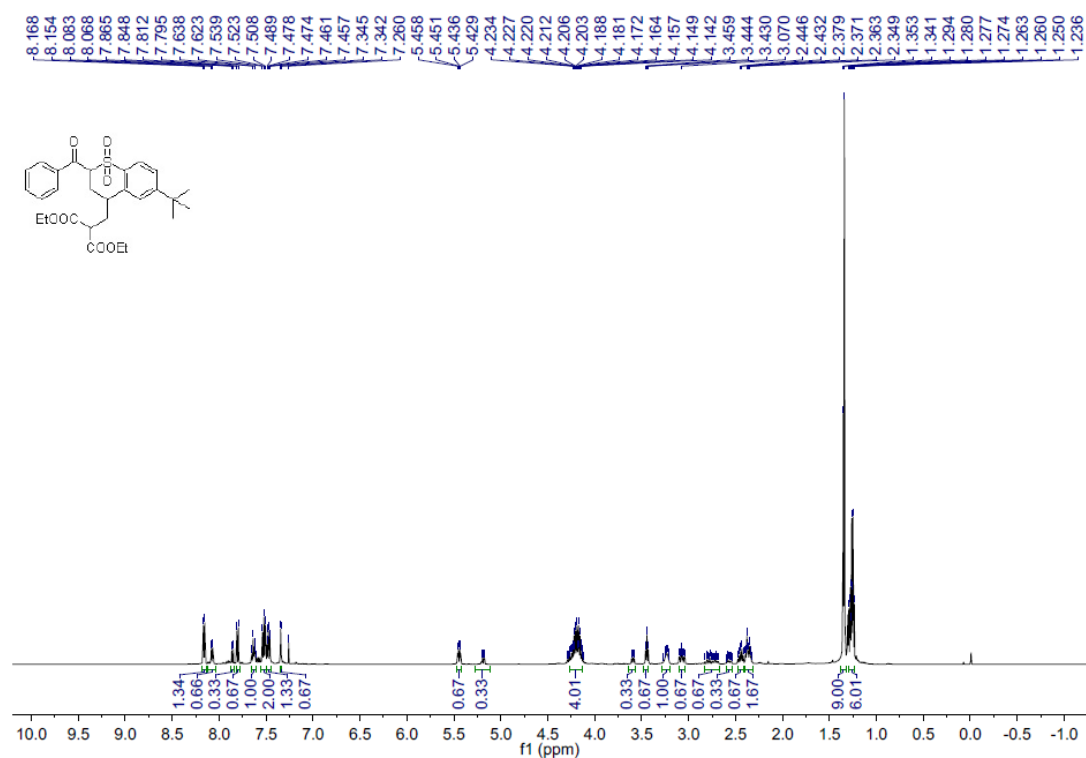
HSQC NMR (CDCl₃) spectrum of compound **4a**



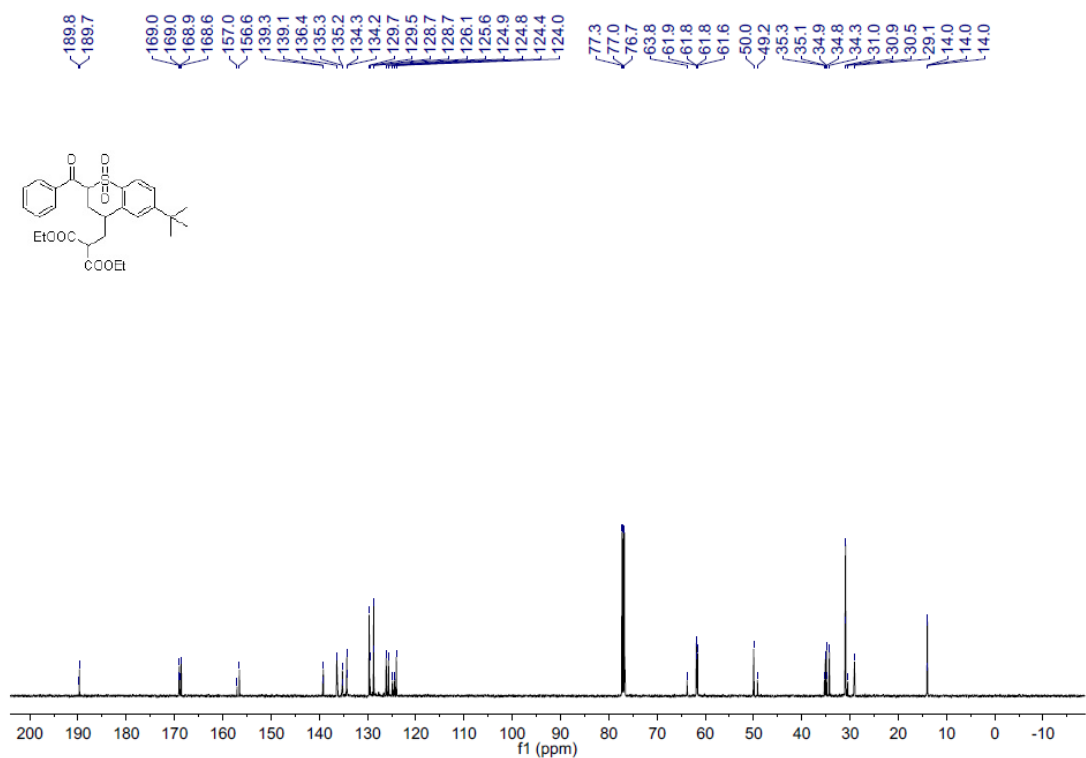
HMBC NMR (CDCl₃) spectrum of compound **4a**



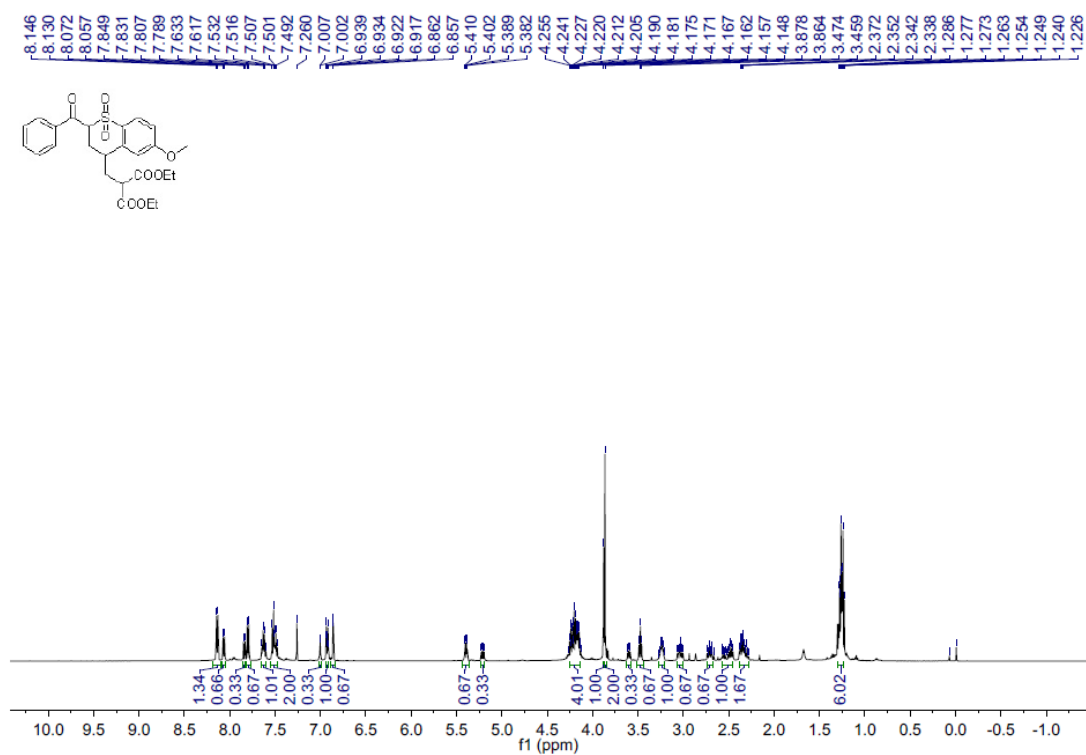
^1H NMR (500 MHz, CDCl_3) spectrum of compound **4b**



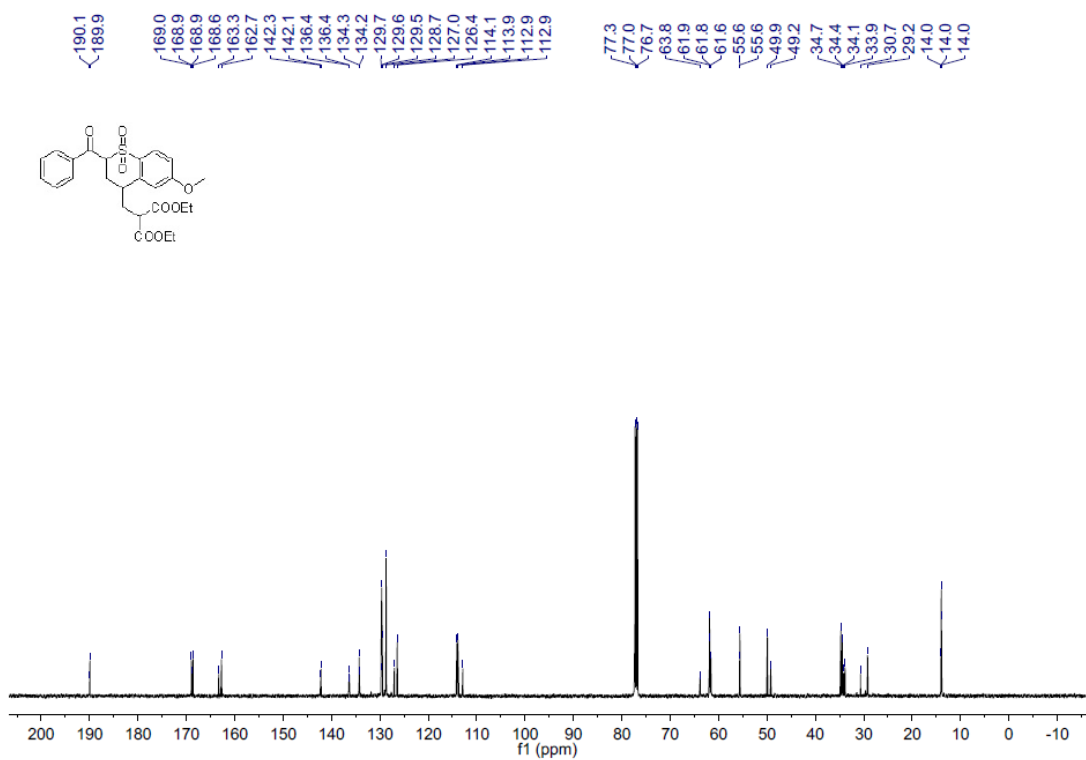
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **4b**



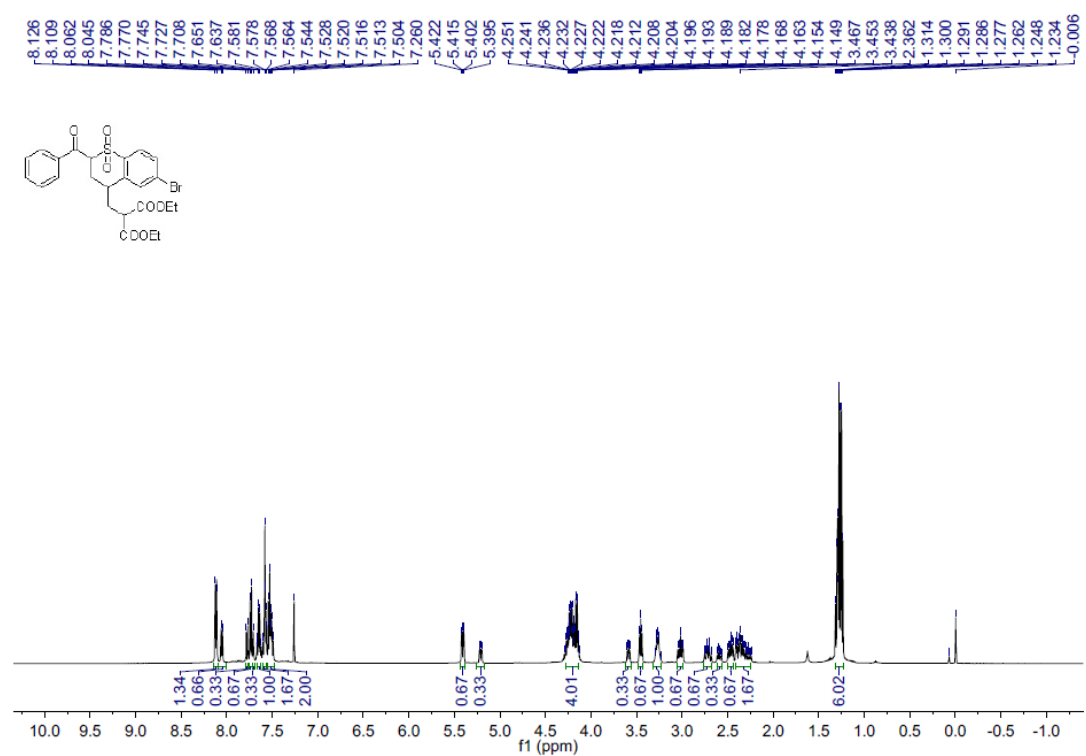
^1H NMR (500 MHz, CDCl_3) spectrum of compound **4c**



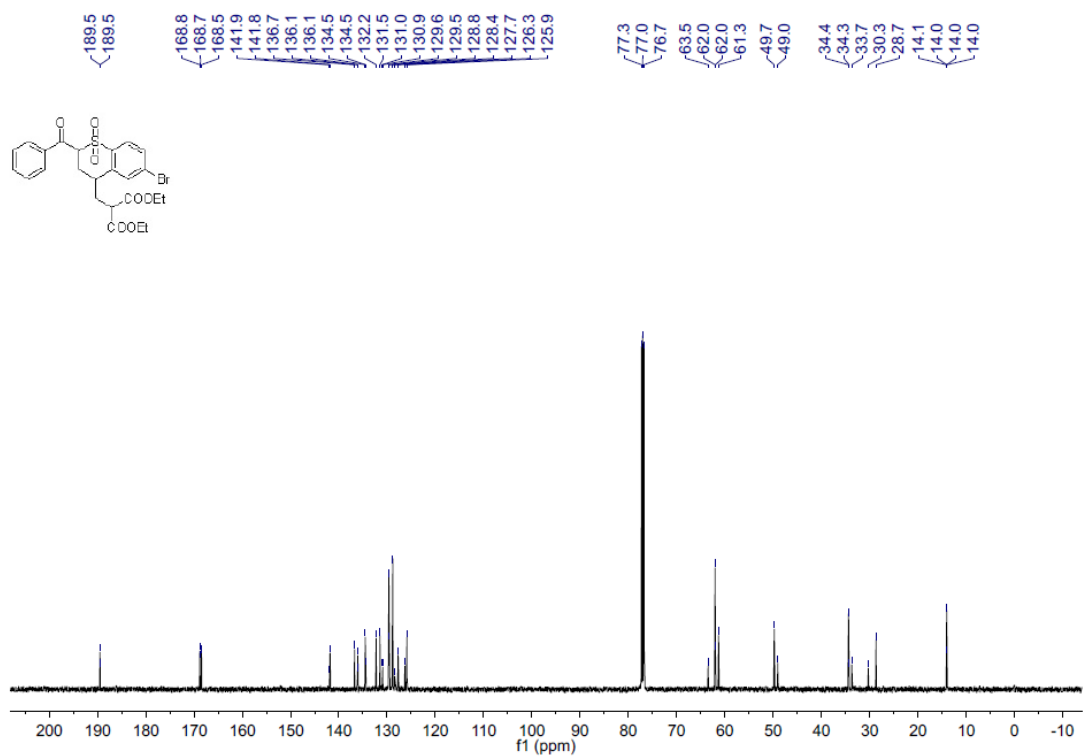
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **4c**



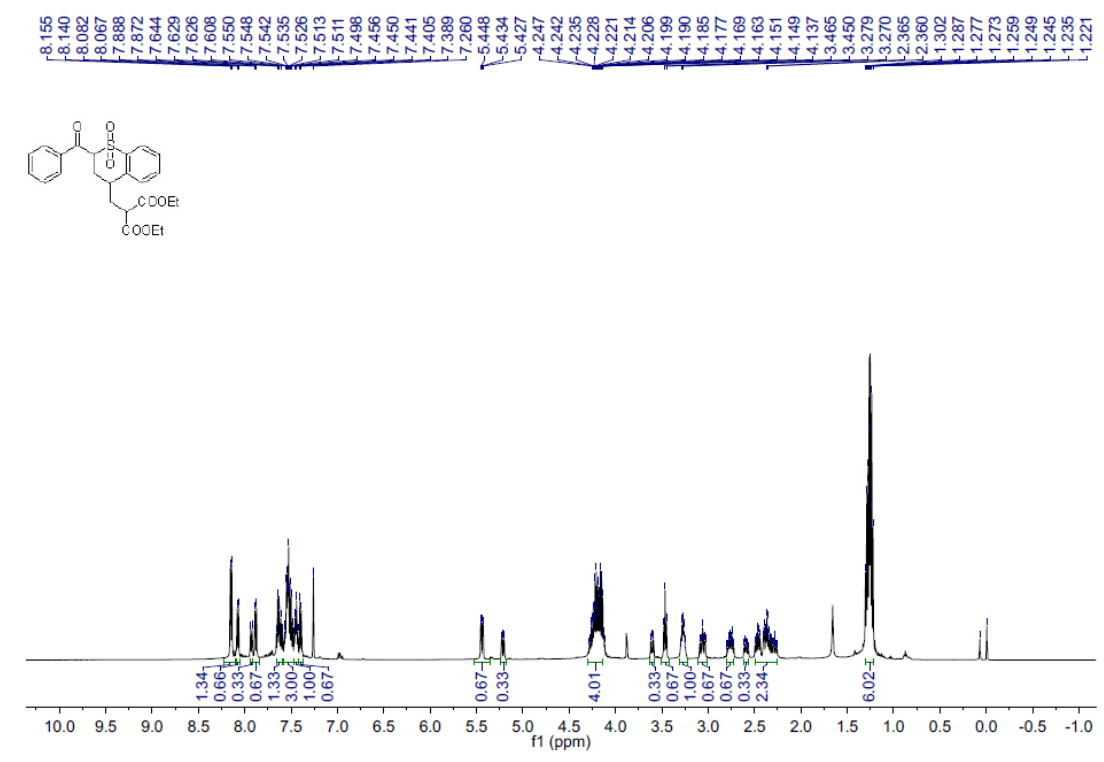
^1H NMR (500 MHz, CDCl_3) spectrum of compound **4d**



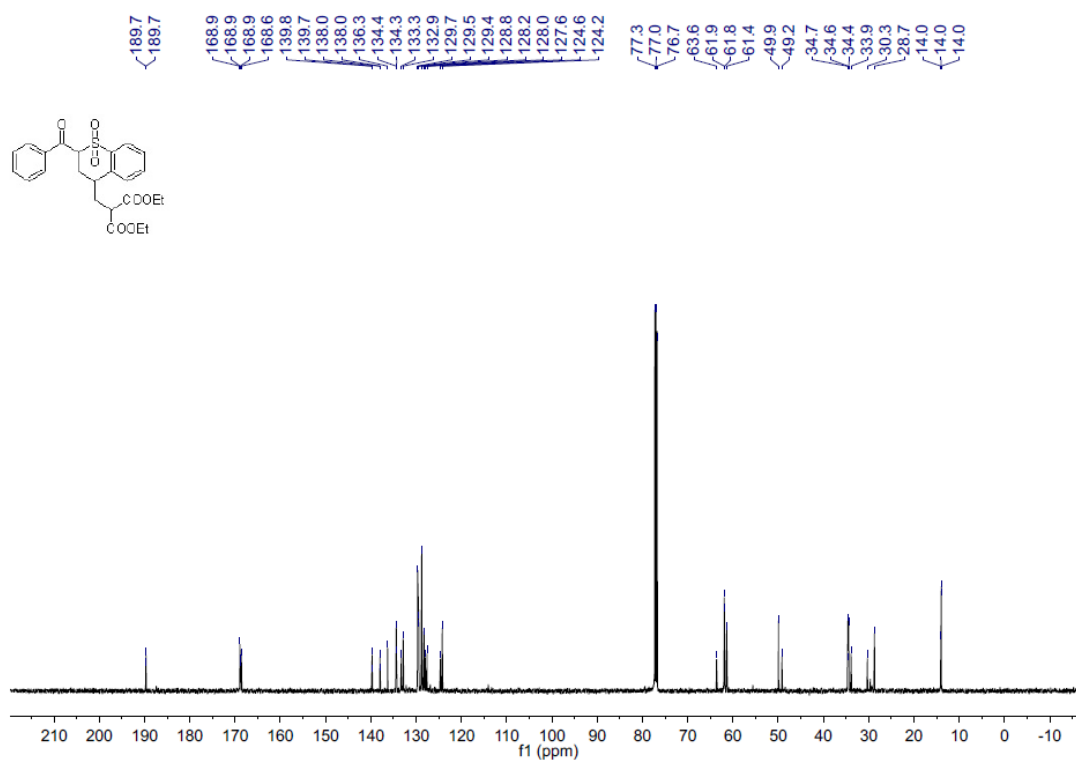
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **4d**



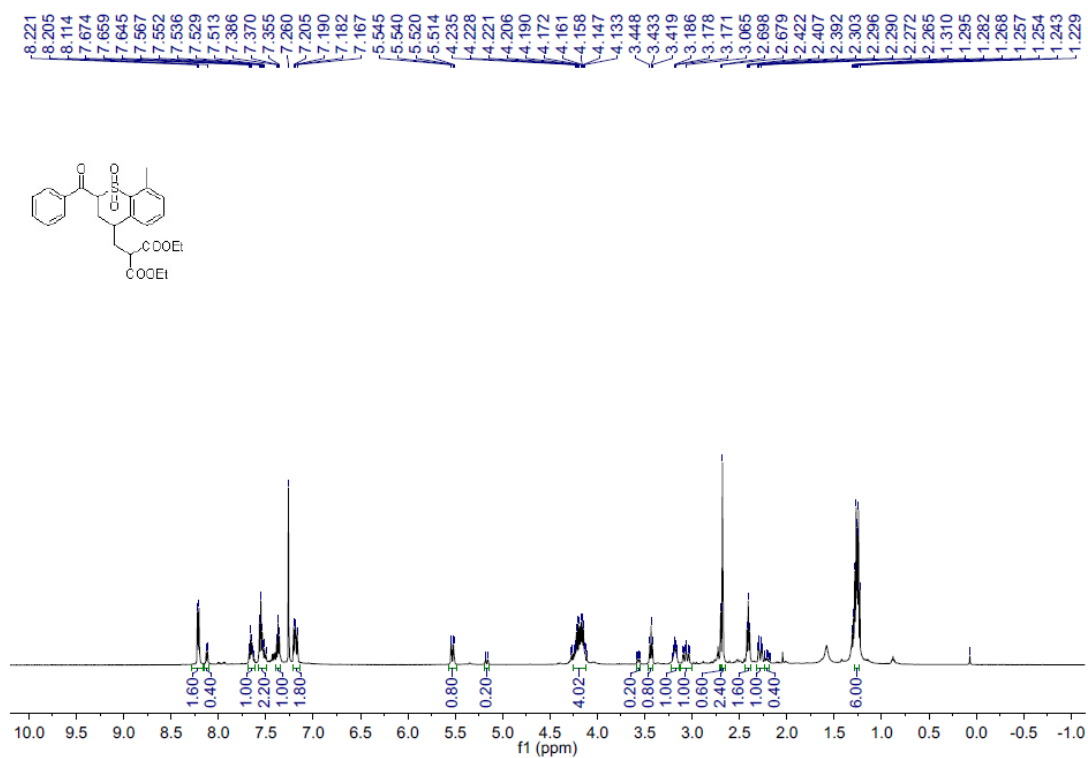
^1H NMR (500 MHz, CDCl_3) spectrum of compound **4e**



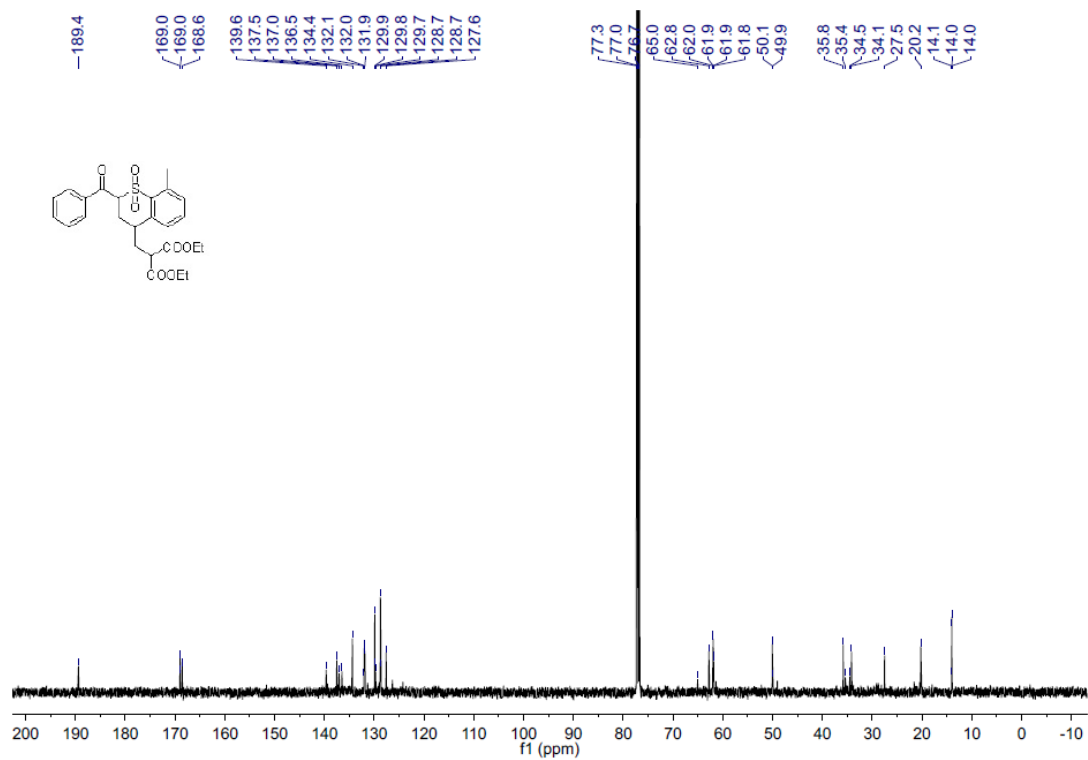
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **4e**



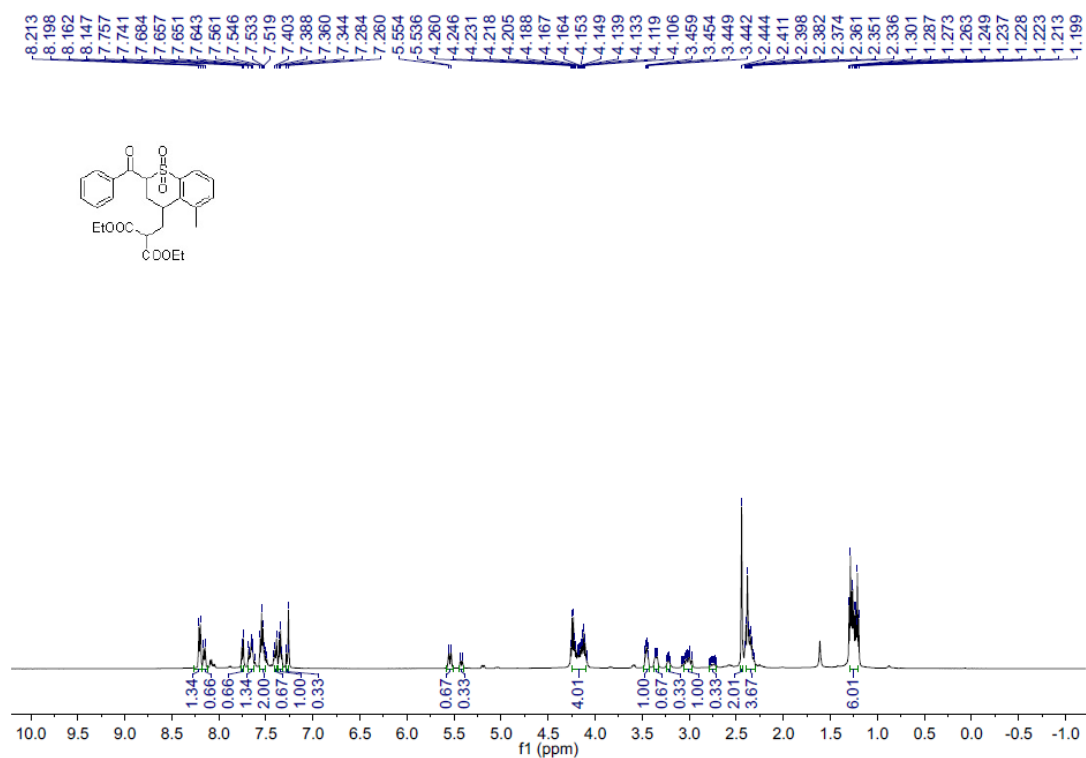
^1H NMR (500 MHz, CDCl_3) spectrum of compound **4f**



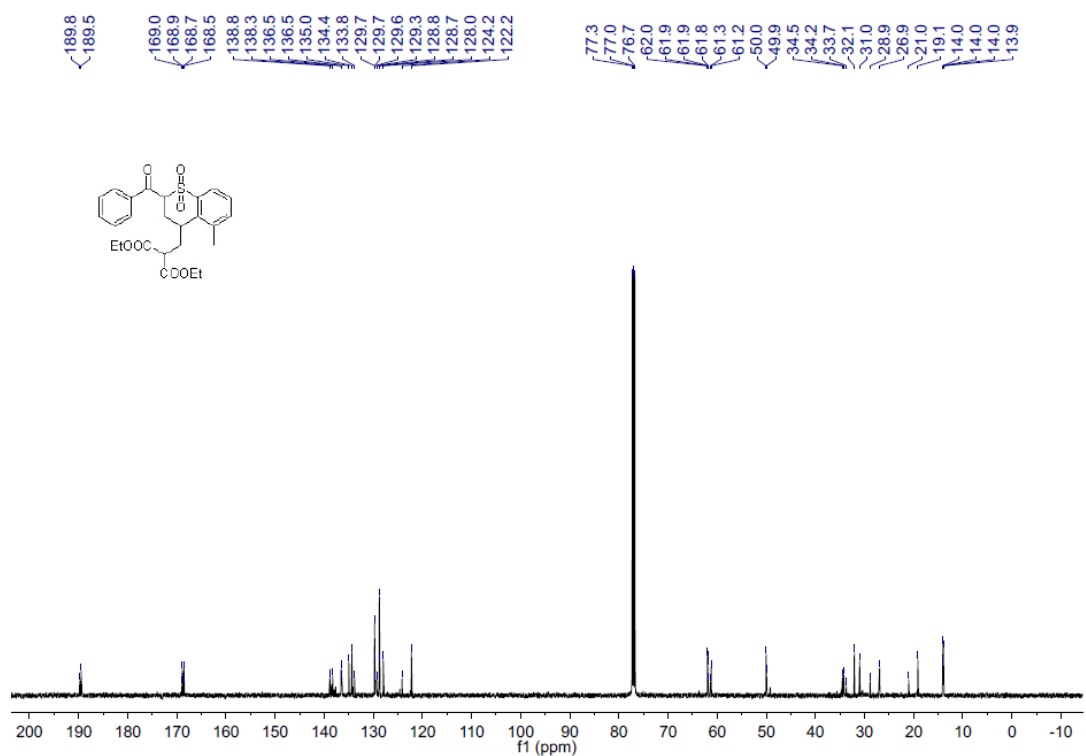
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **4f**



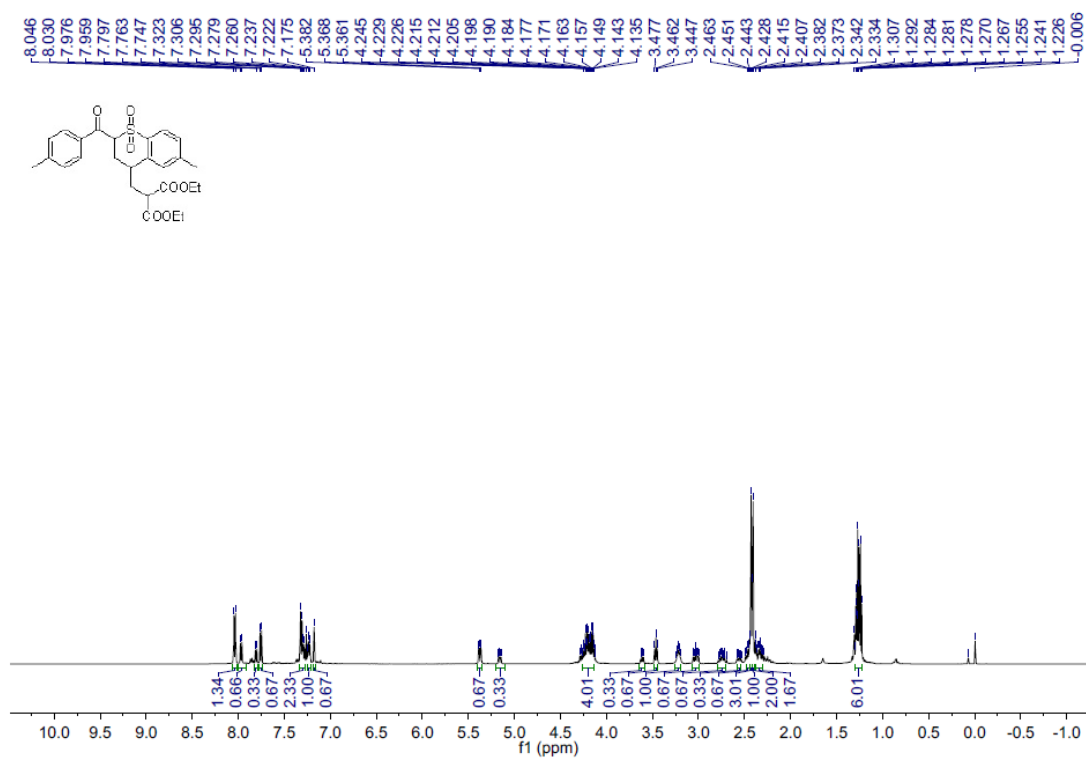
^1H NMR (500 MHz, CDCl_3) spectrum of compound **4g**



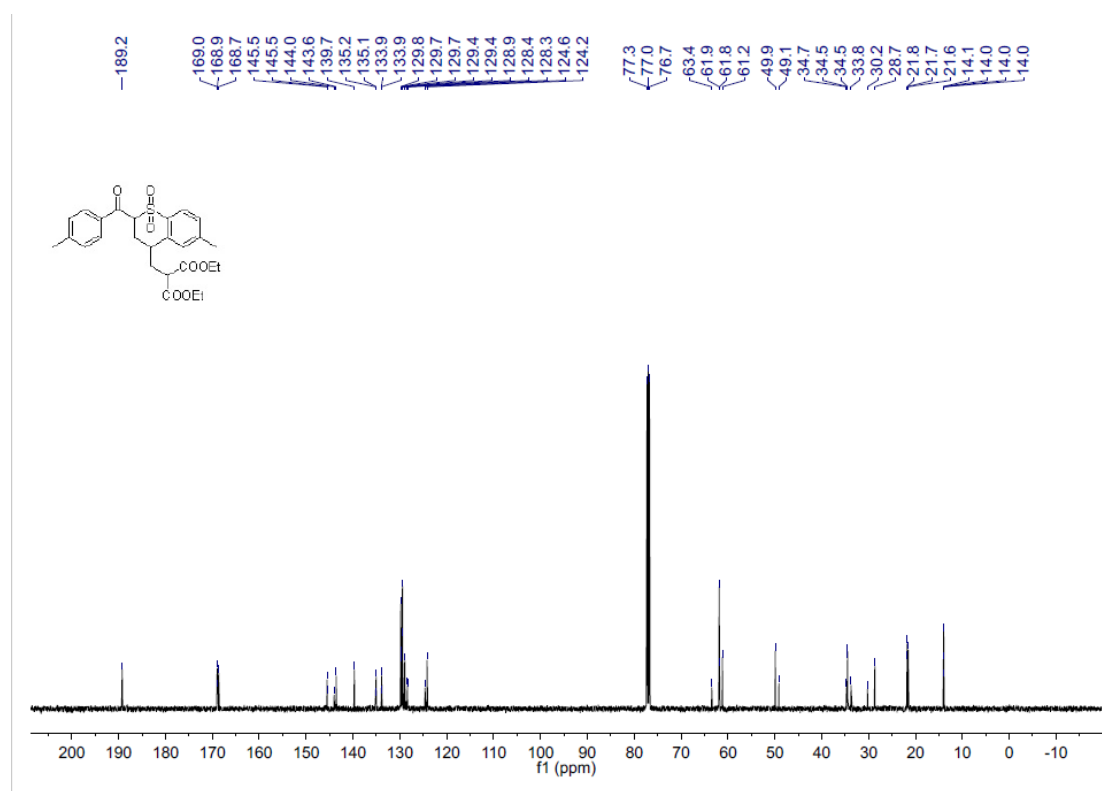
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **4g**



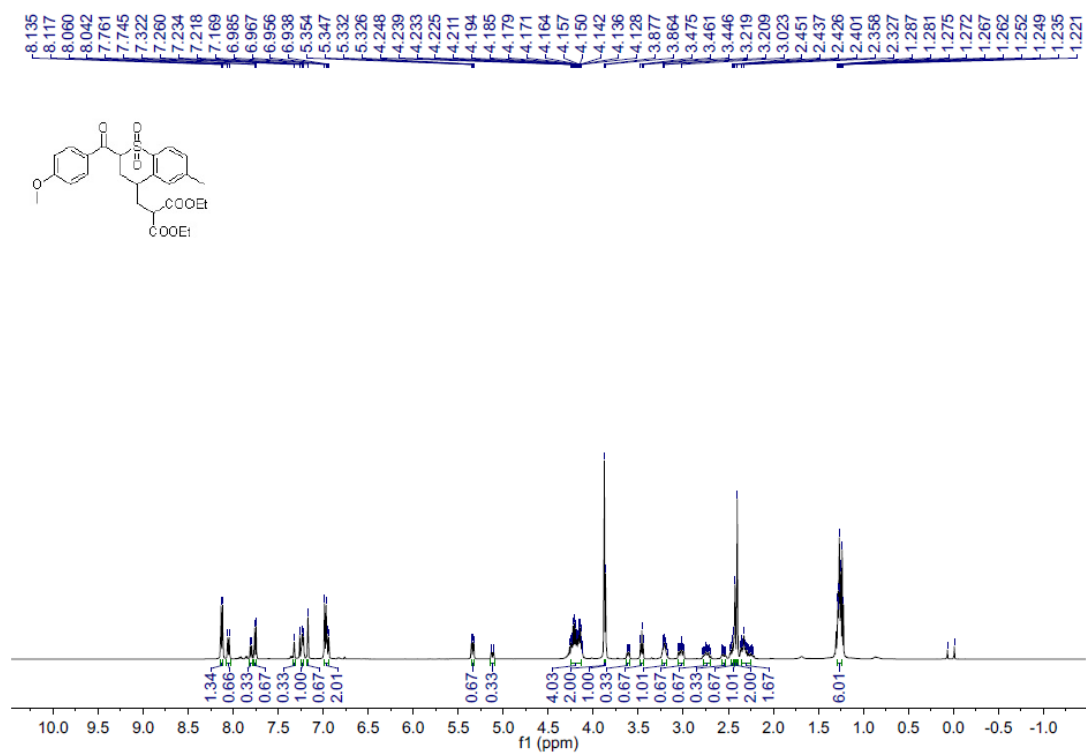
^1H NMR (500 MHz, CDCl_3) spectrum of compound **4h**



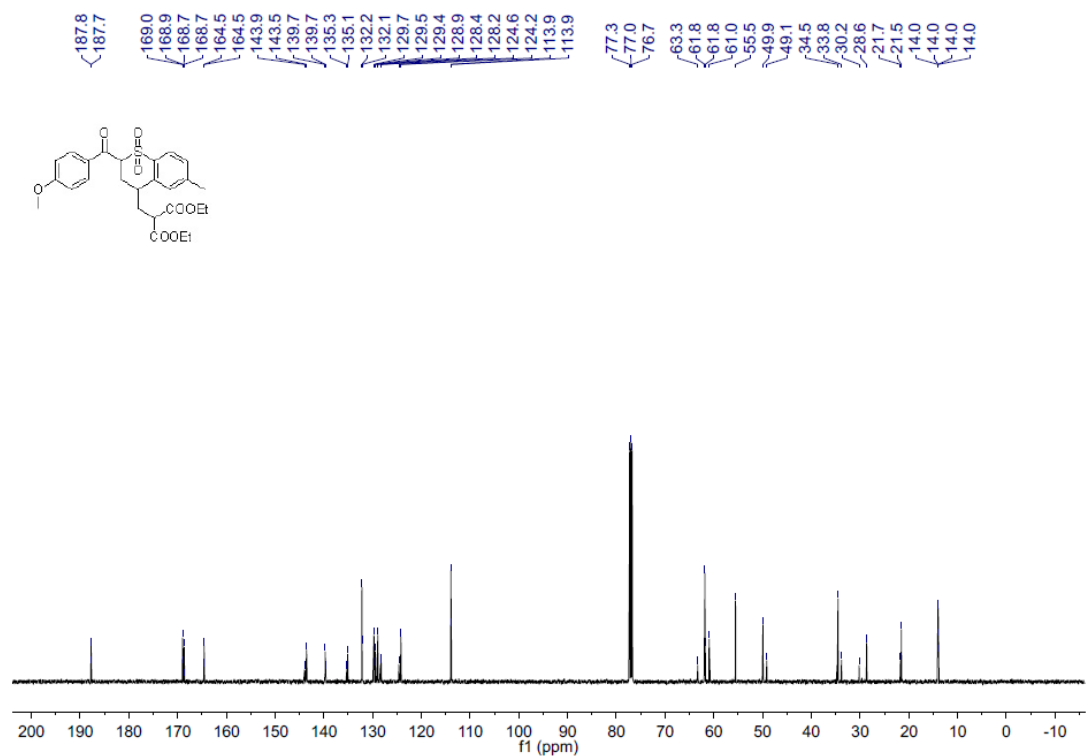
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **4h**



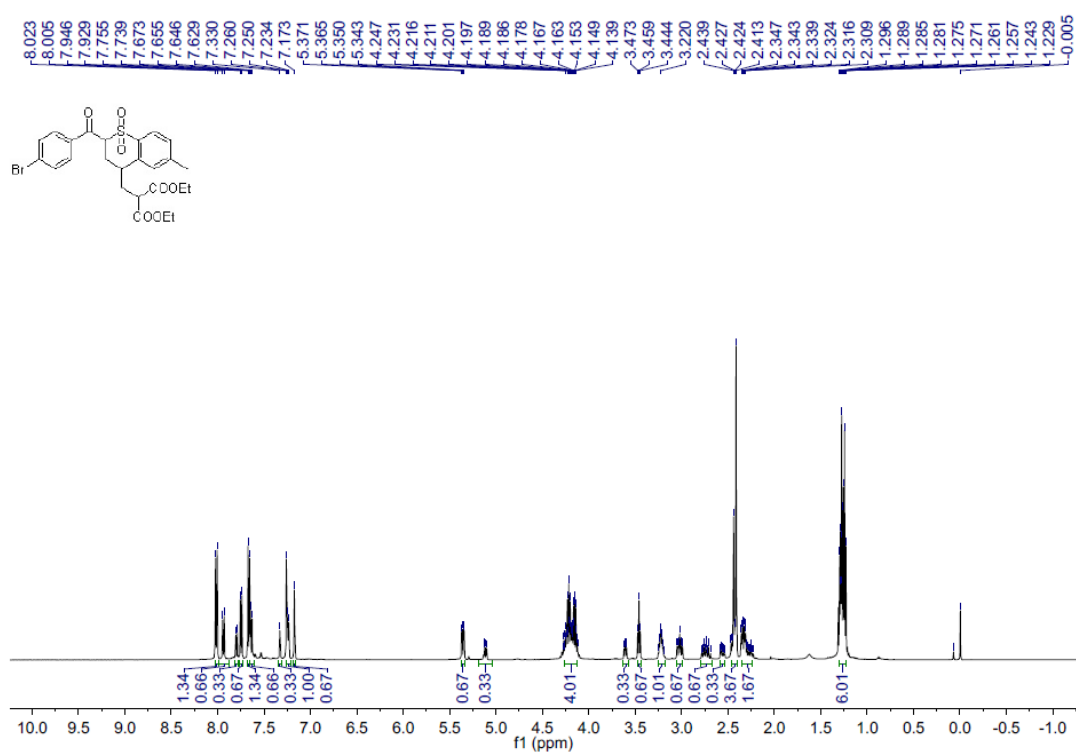
^1H NMR (500 MHz, CDCl_3) spectrum of compound **4i**



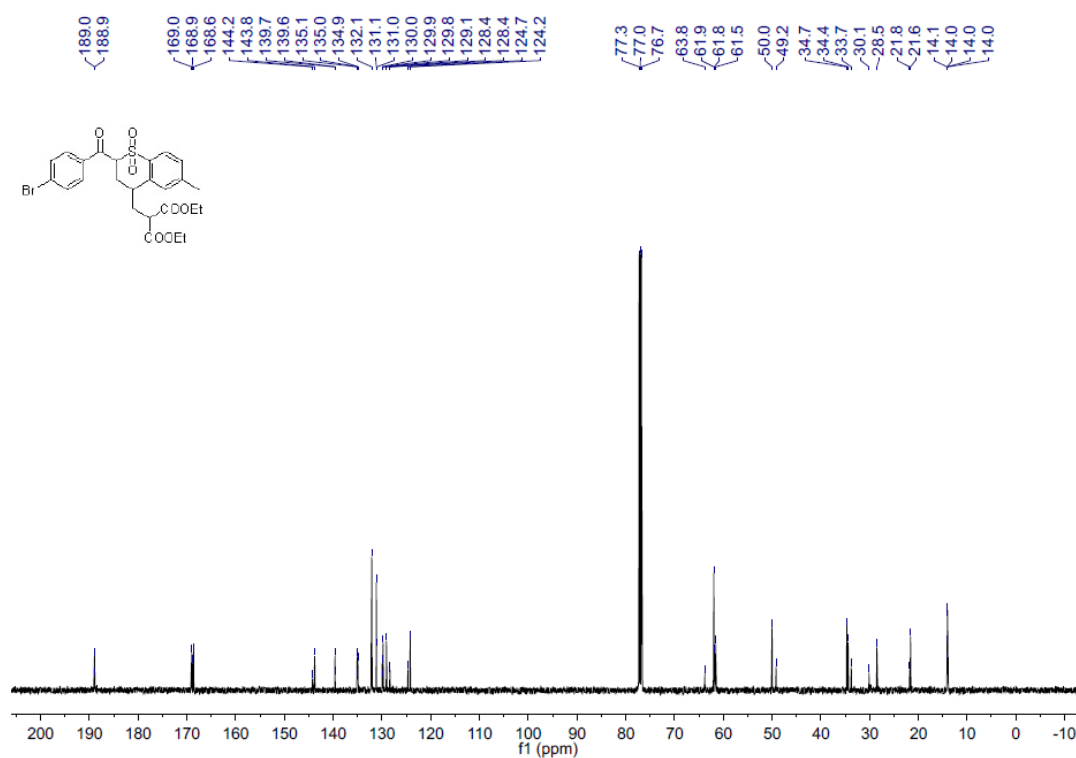
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **4i**



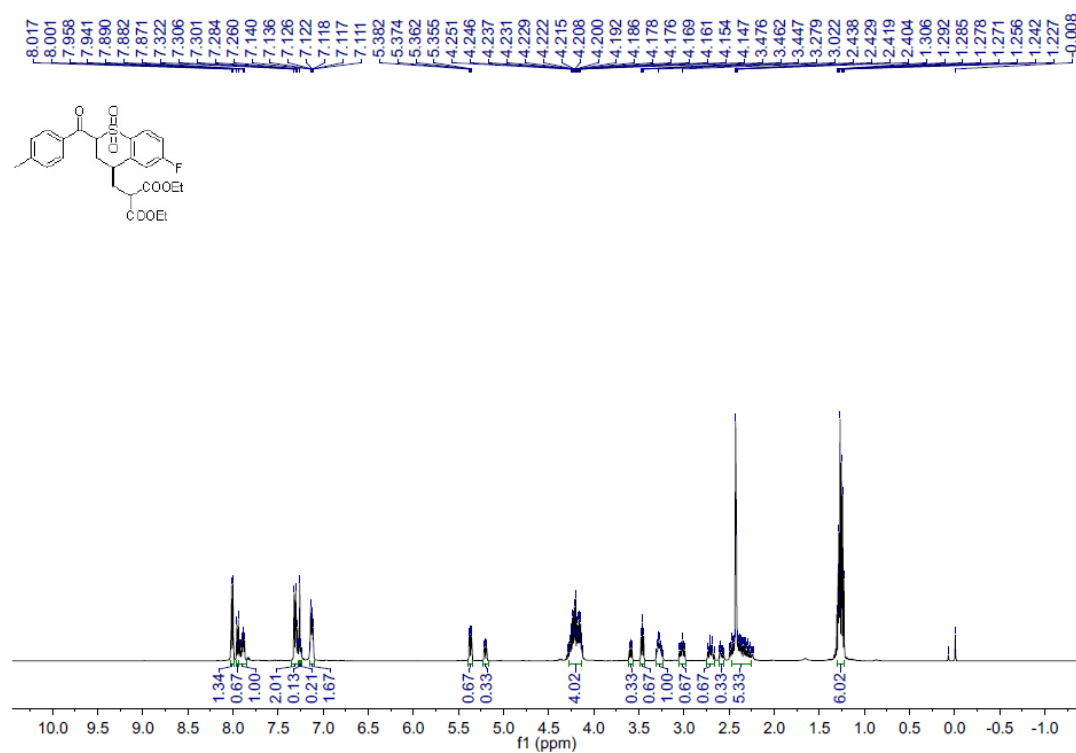
^1H NMR (500 MHz, CDCl_3) spectrum of compound **4j**



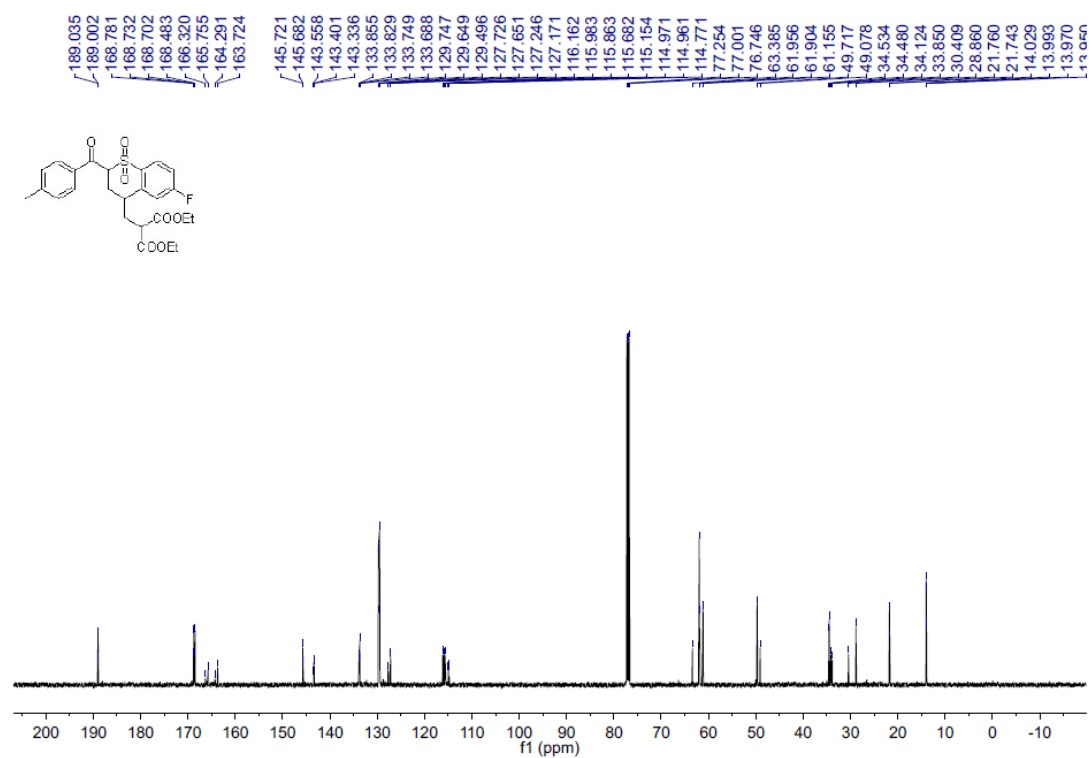
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **4j**



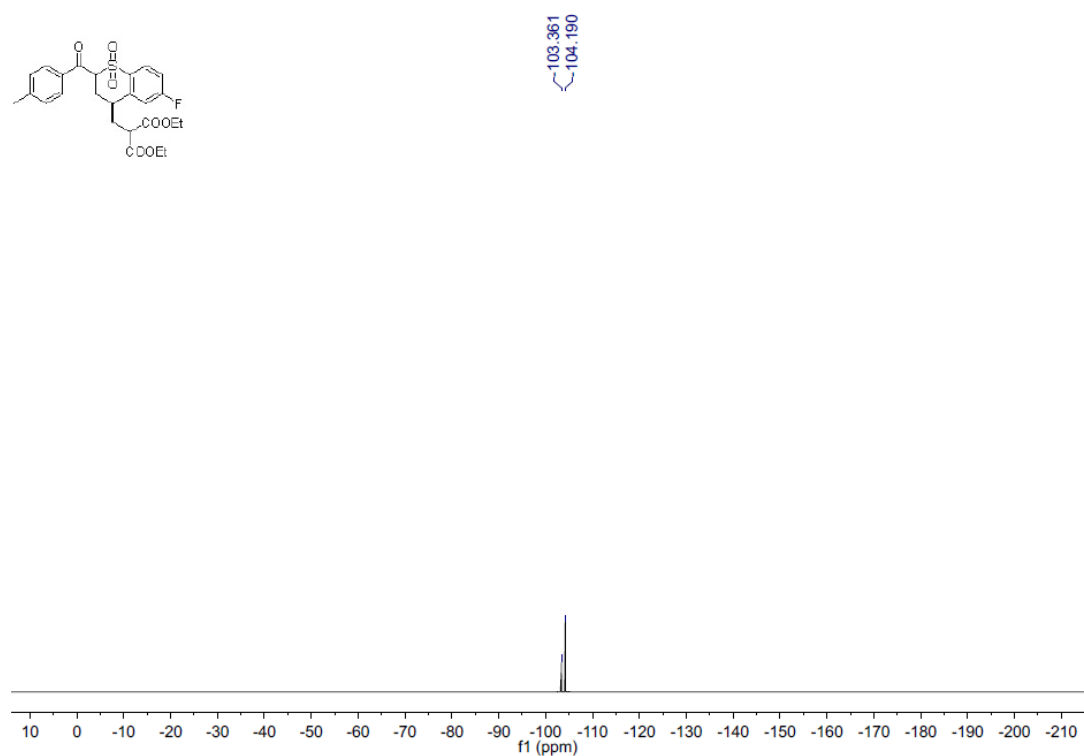
^1H NMR (500 MHz, CDCl_3) spectrum of compound **4k**



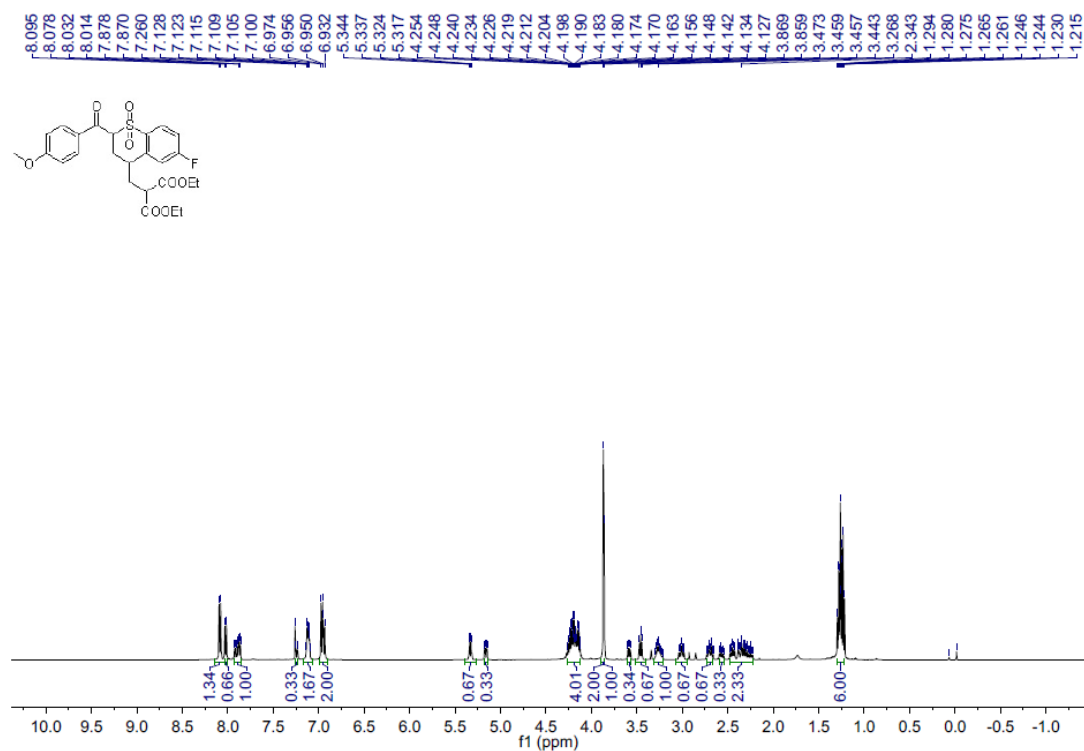
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **4k**



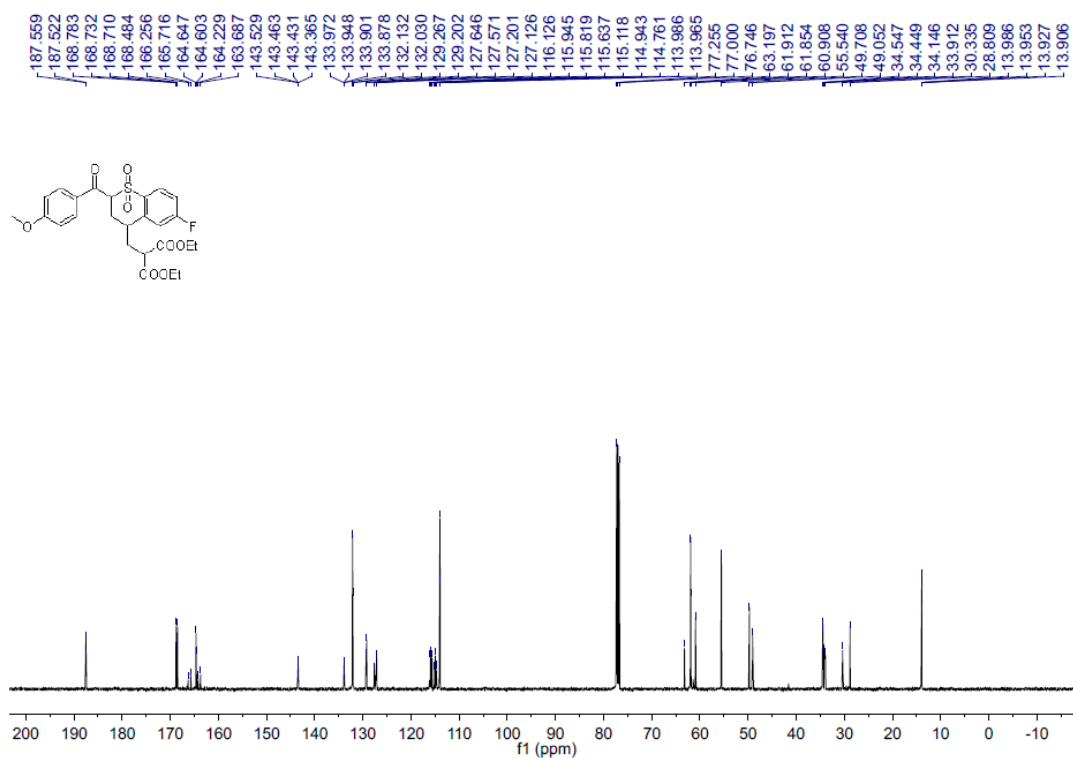
^{19}F NMR (470 MHz, CDCl_3) spectrum of compound **4k**



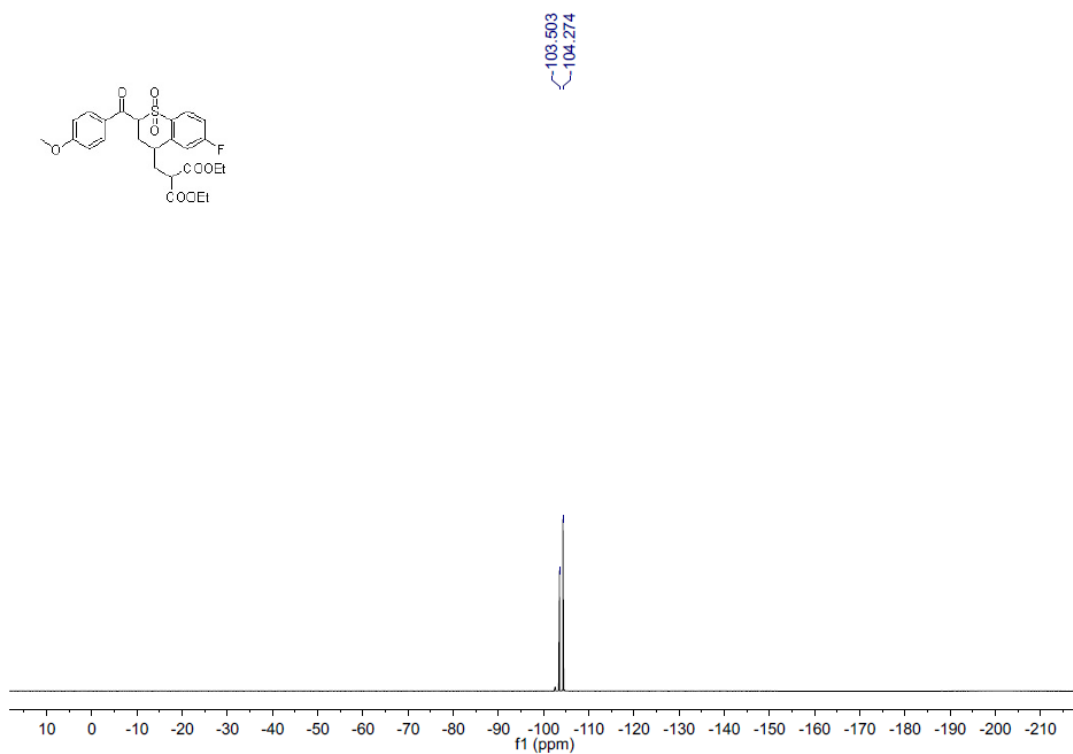
^1H NMR (500 MHz, CDCl_3) spectrum of compound **4l**



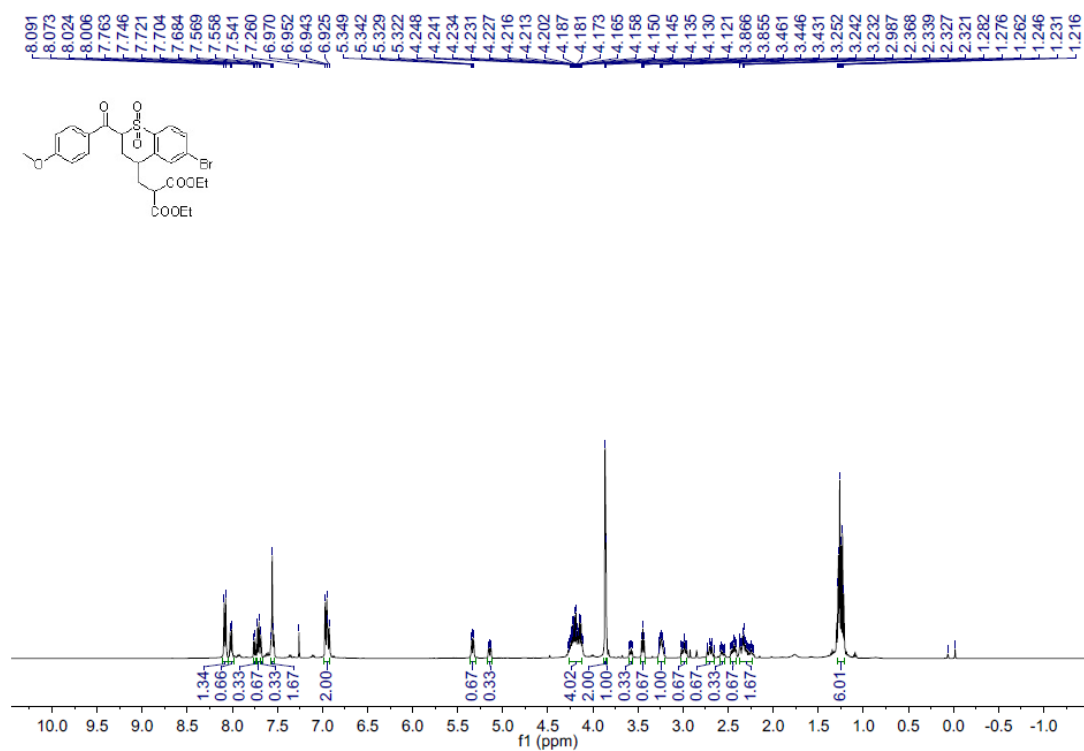
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **4l**



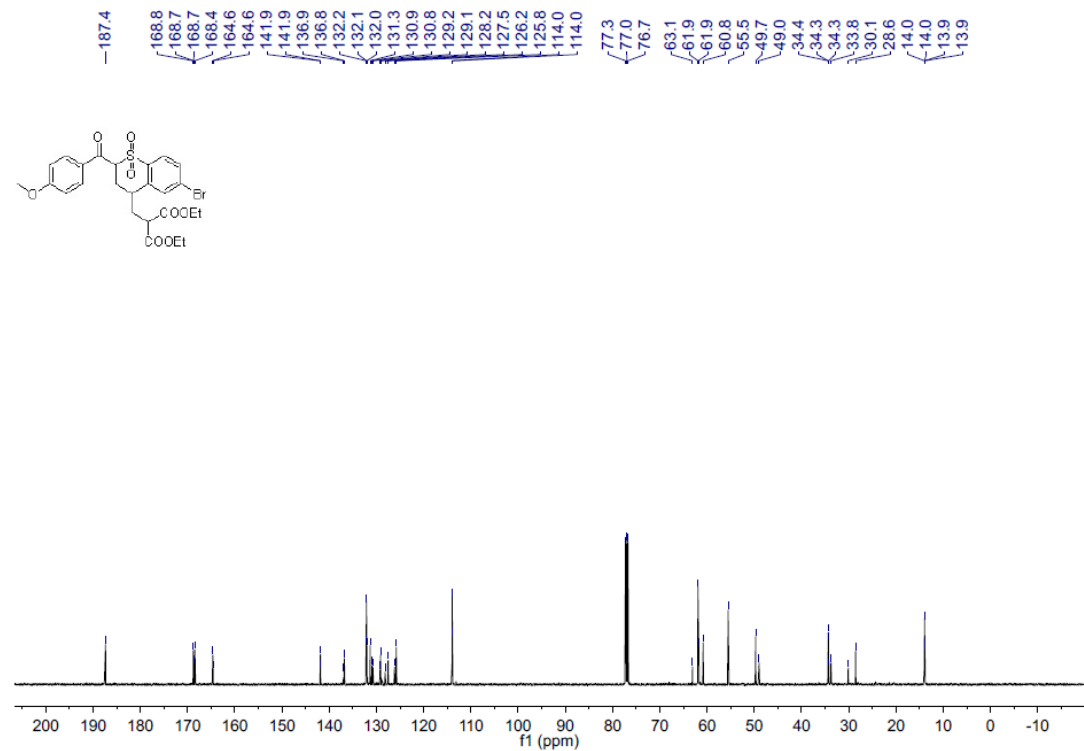
^{19}F NMR (470 MHz, CDCl_3) spectrum of compound **4l**



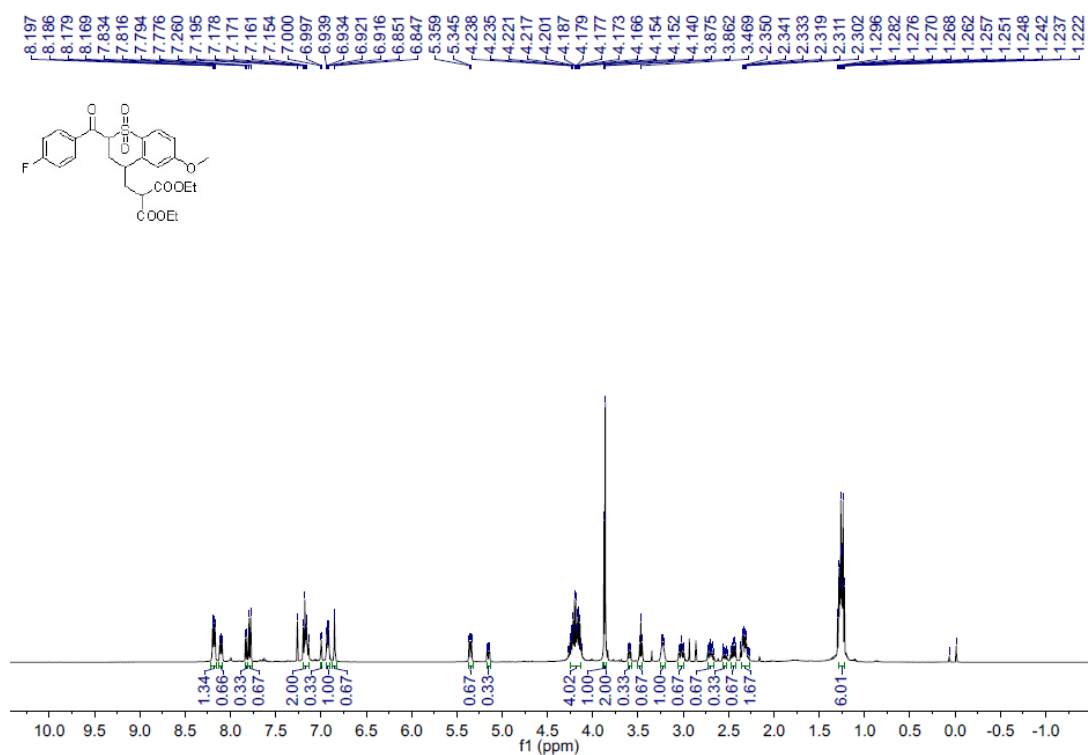
^1H NMR (500 MHz, CDCl_3) spectrum of compound **4m**



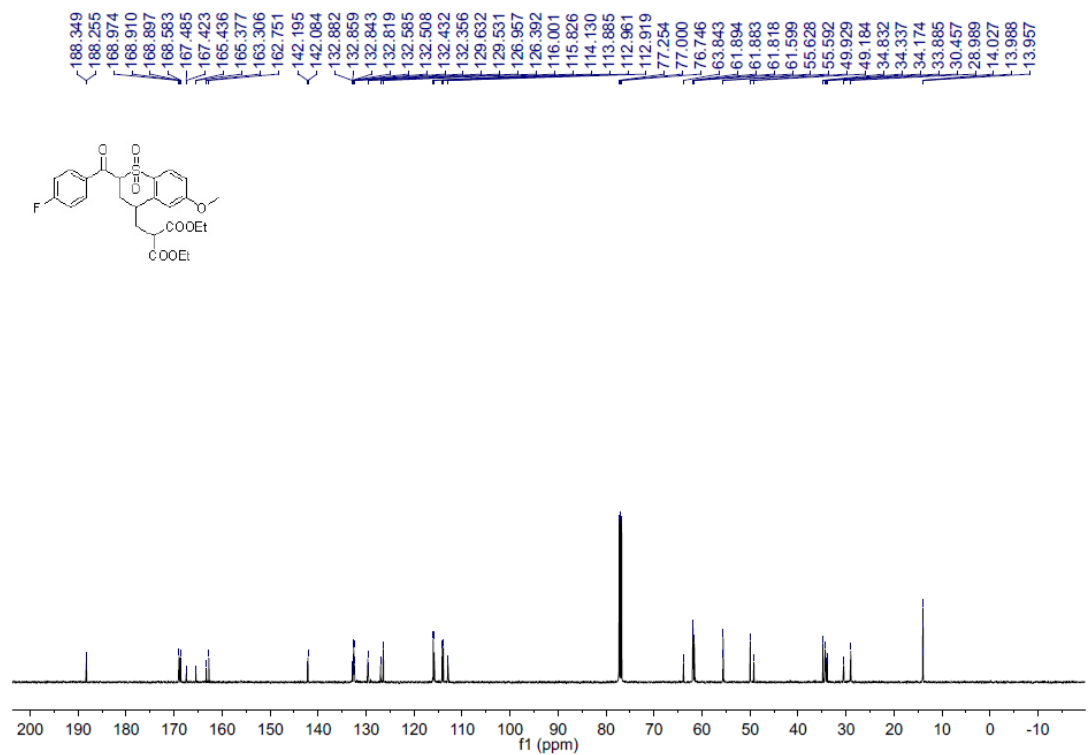
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **4m**



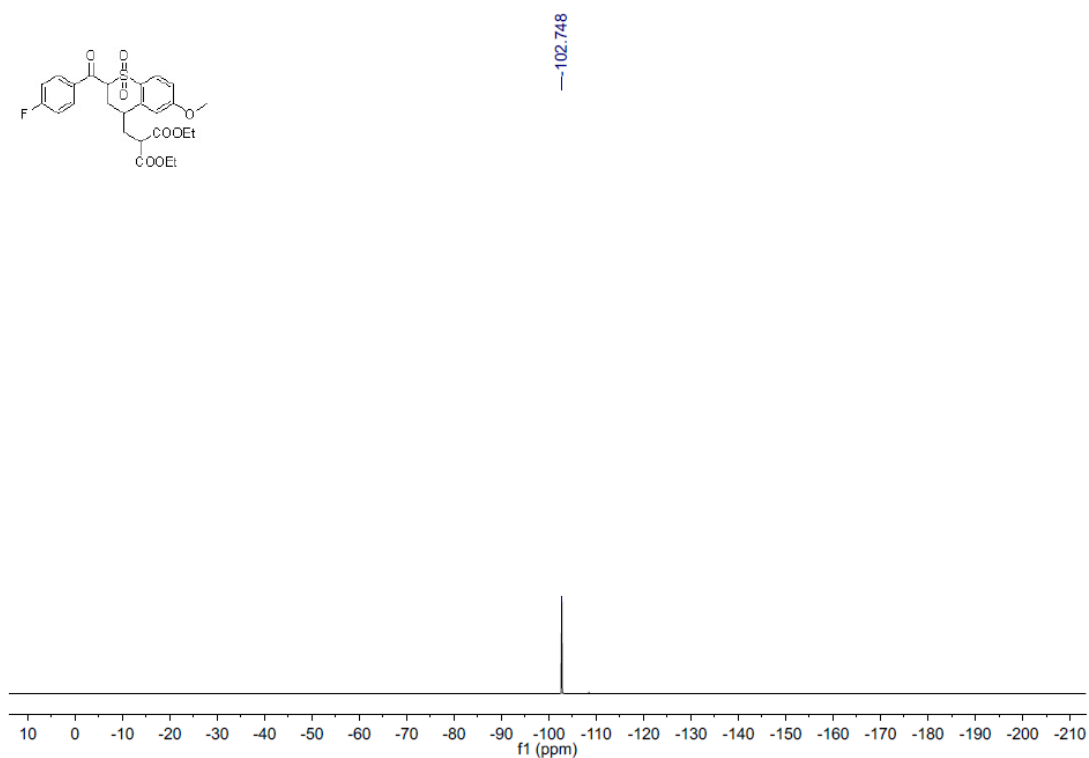
^1H NMR (500 MHz, CDCl_3) spectrum of compound **4n**



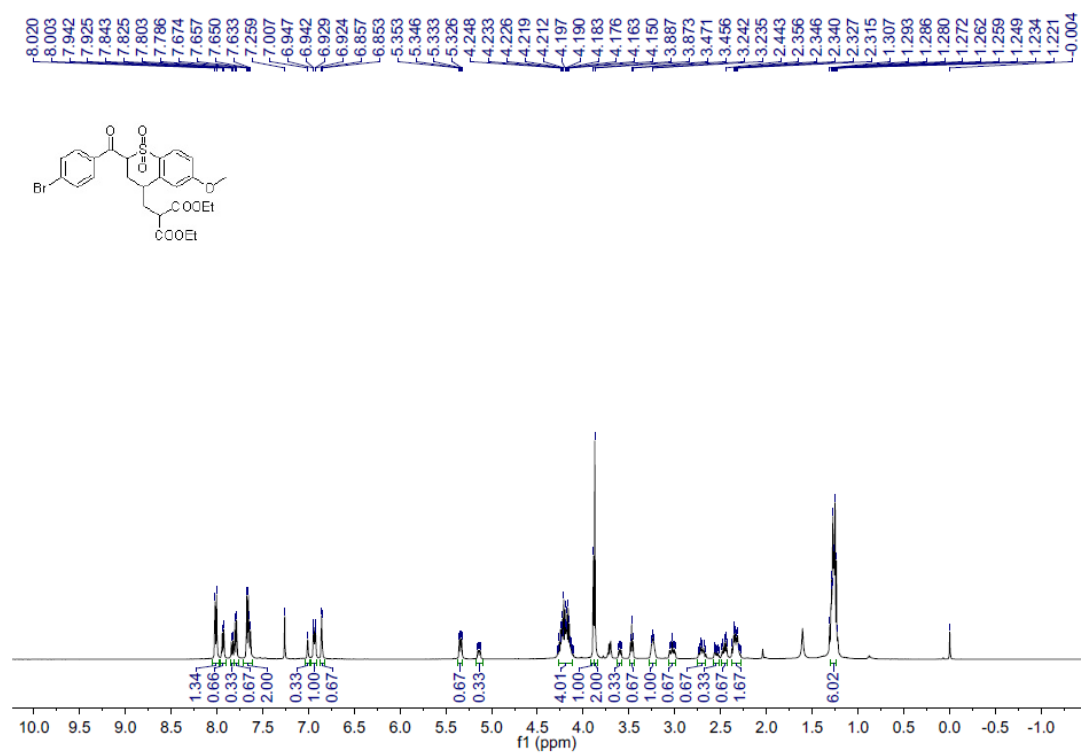
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **4n**



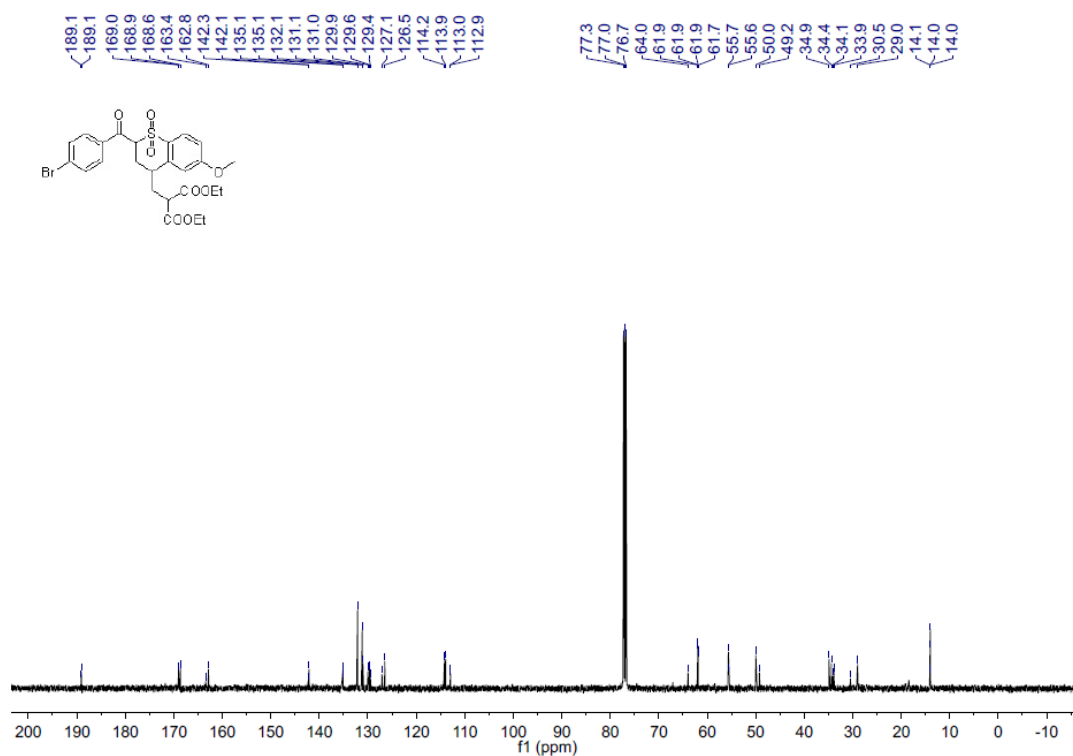
^{19}F NMR (470 MHz, CDCl_3) spectrum of compound **4n**



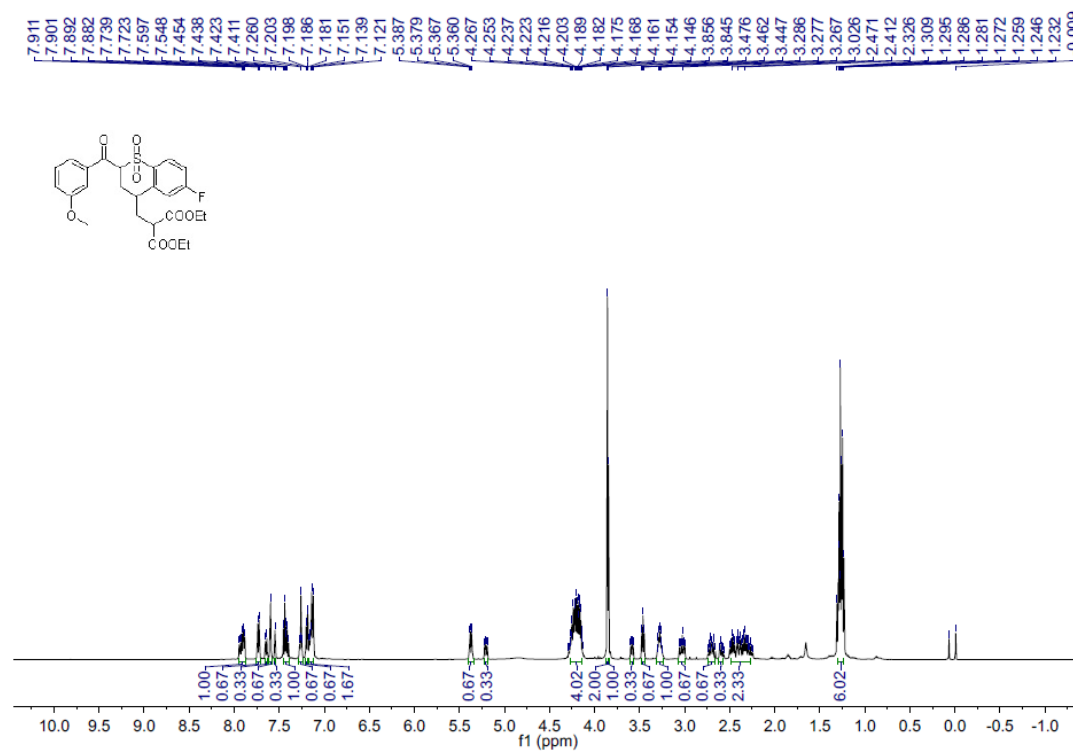
^1H NMR (500 MHz, CDCl_3) spectrum of compound **4o**



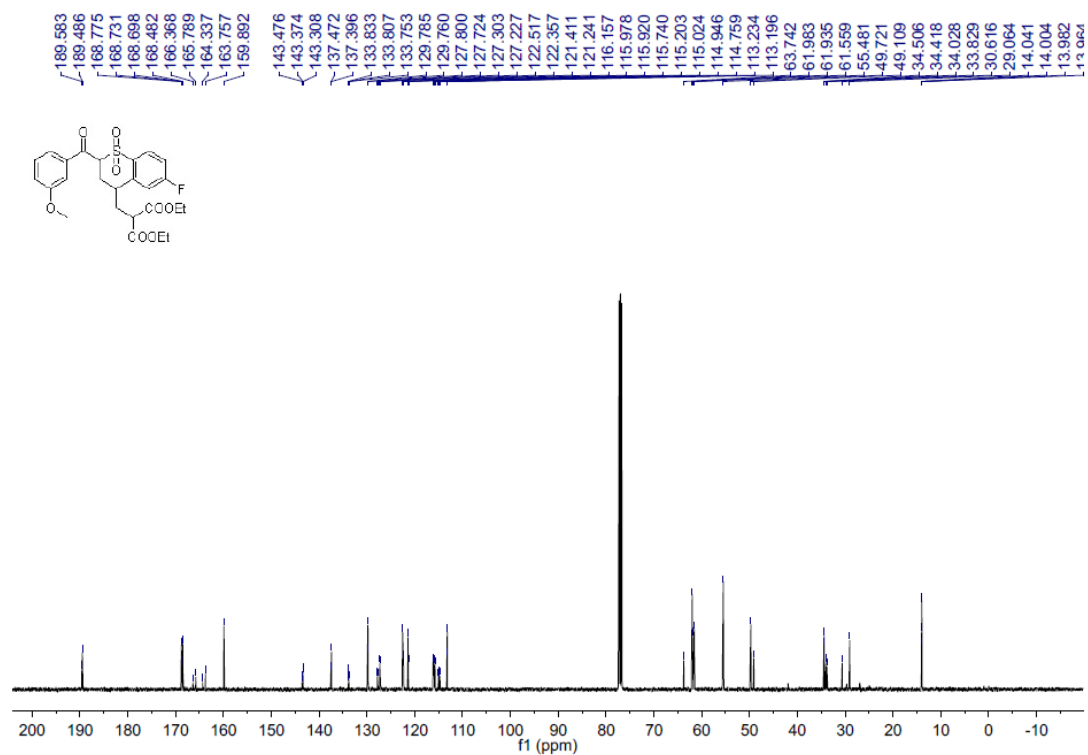
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **4o**



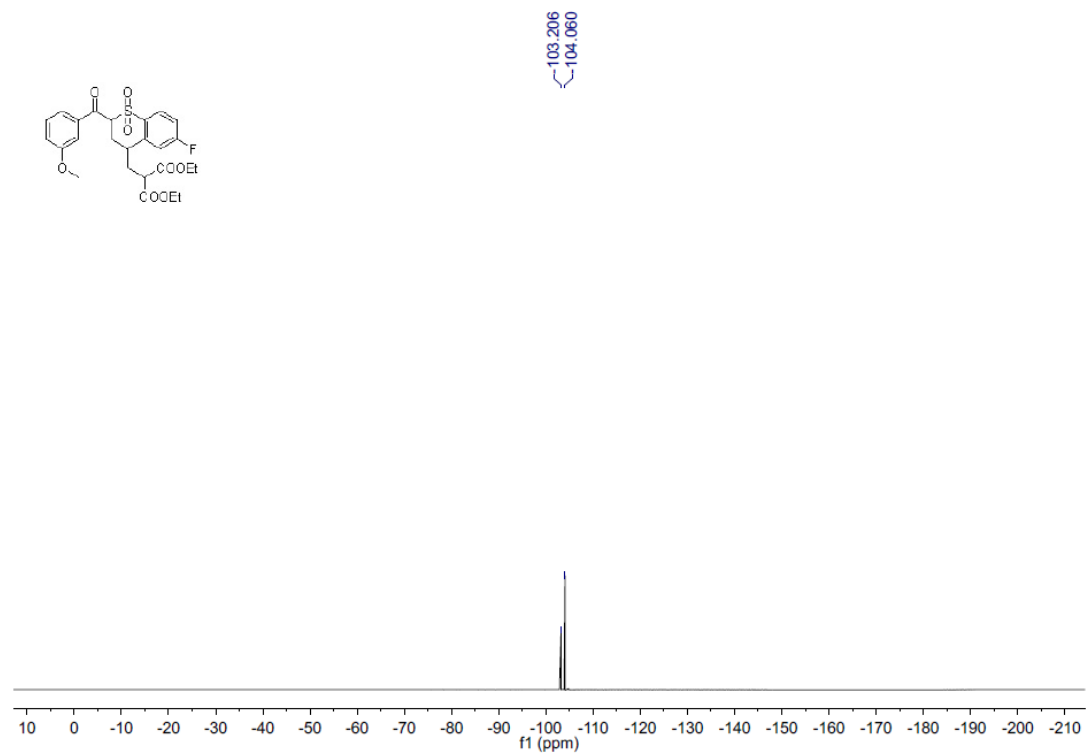
^1H NMR (500 MHz, CDCl_3) spectrum of compound **4p**



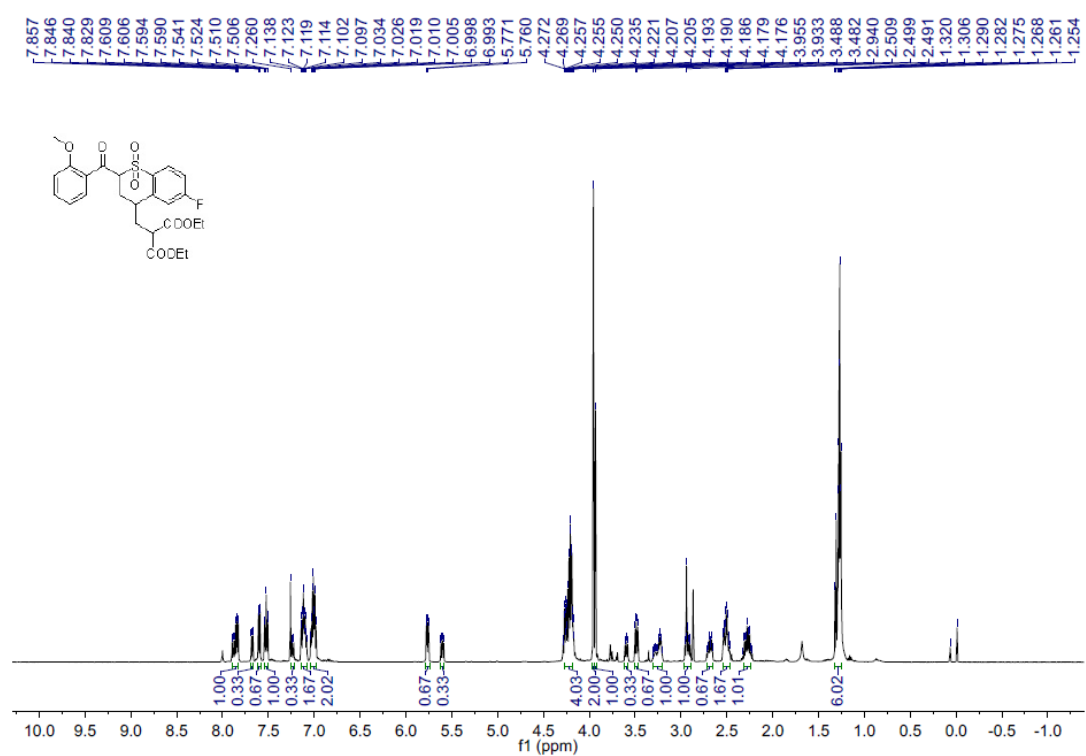
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **4p**



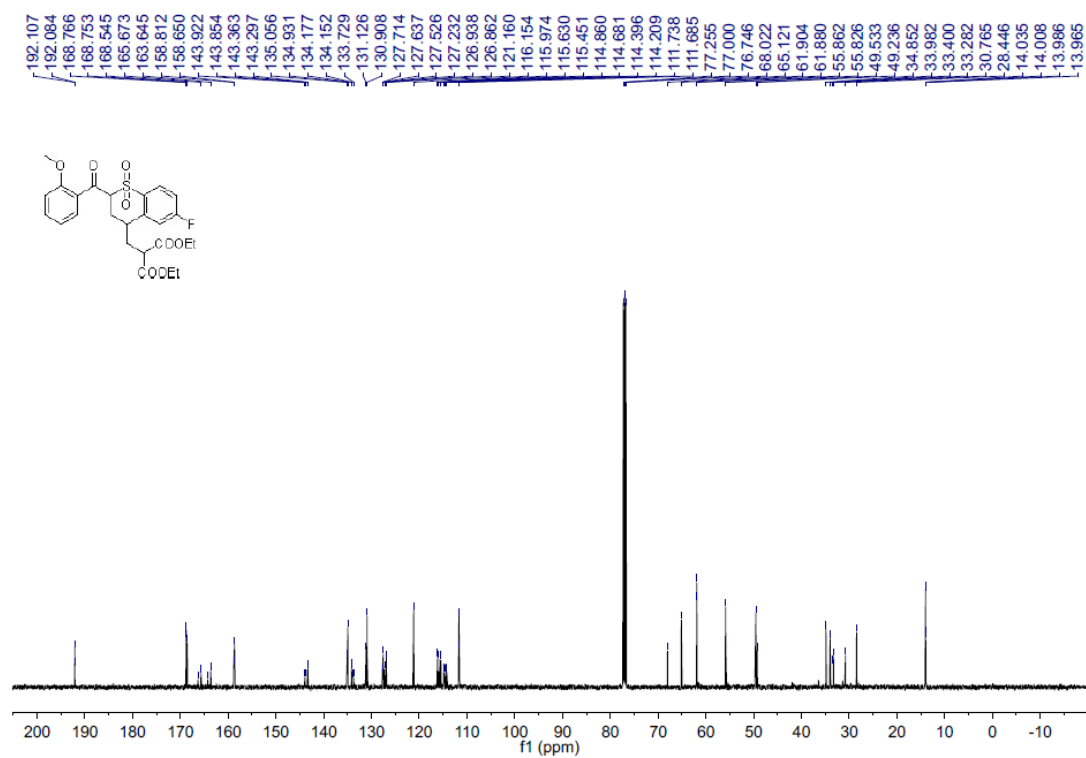
^{19}F NMR (470 MHz, CDCl_3) spectrum of compound **4p**



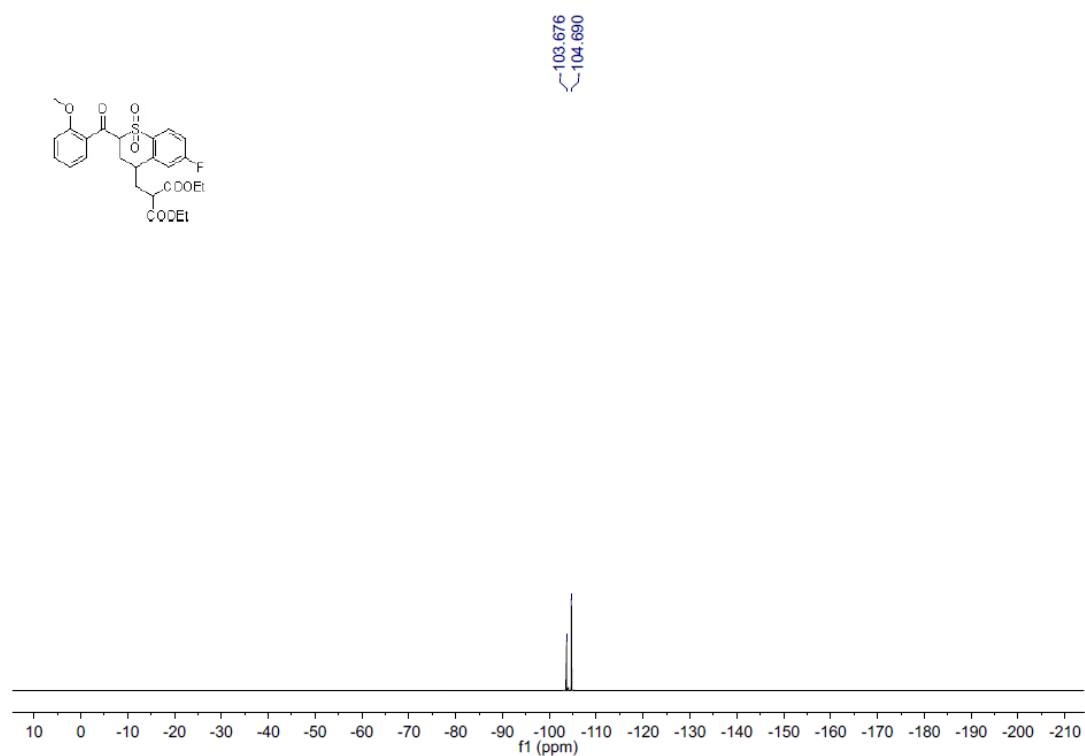
^1H NMR (500 MHz, CDCl_3) spectrum of compound **4q**



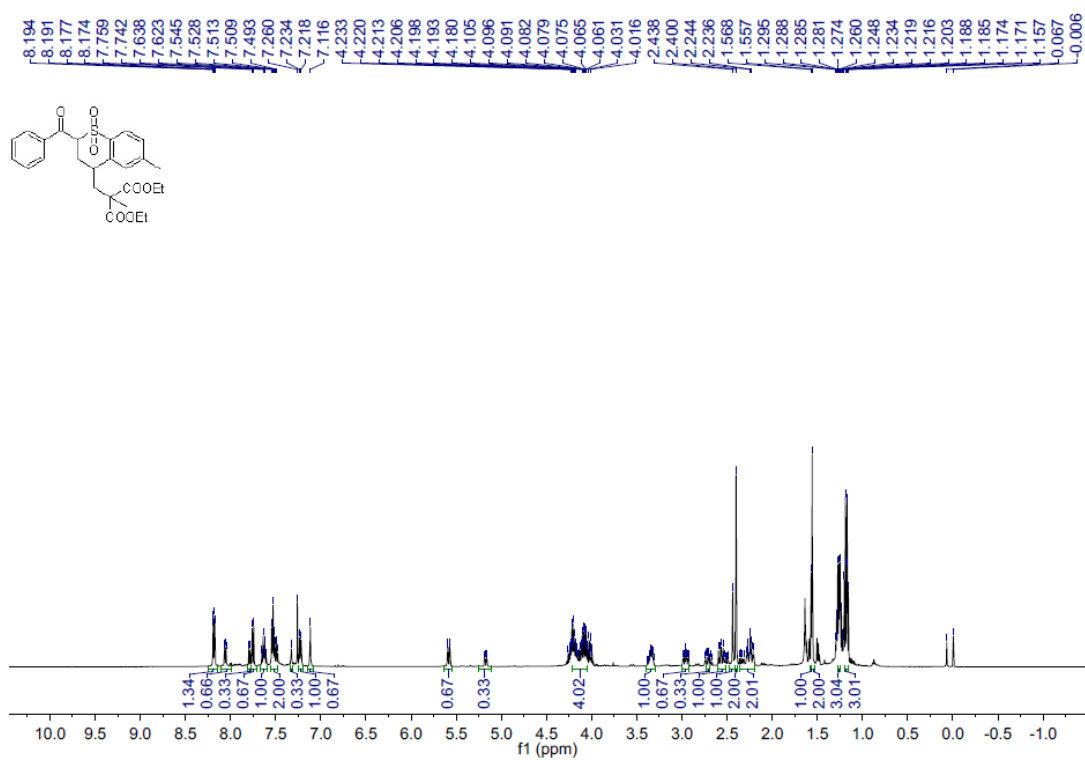
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **4q**



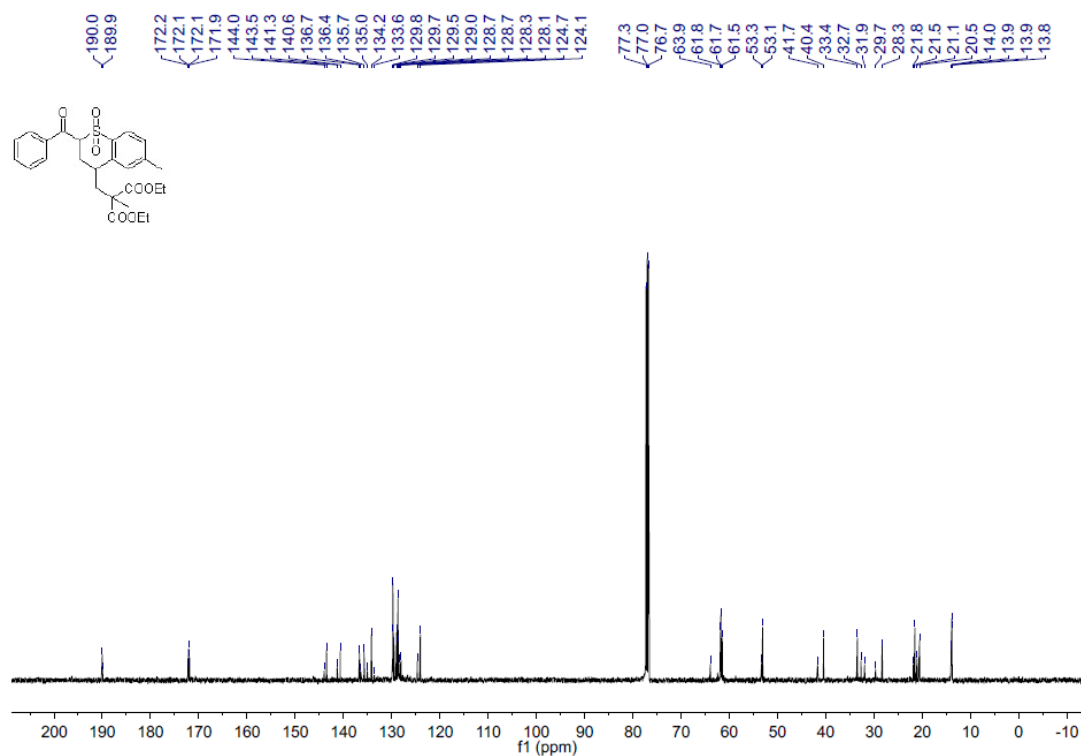
^{19}F NMR (470 MHz, CDCl_3) spectrum of compound **4q**



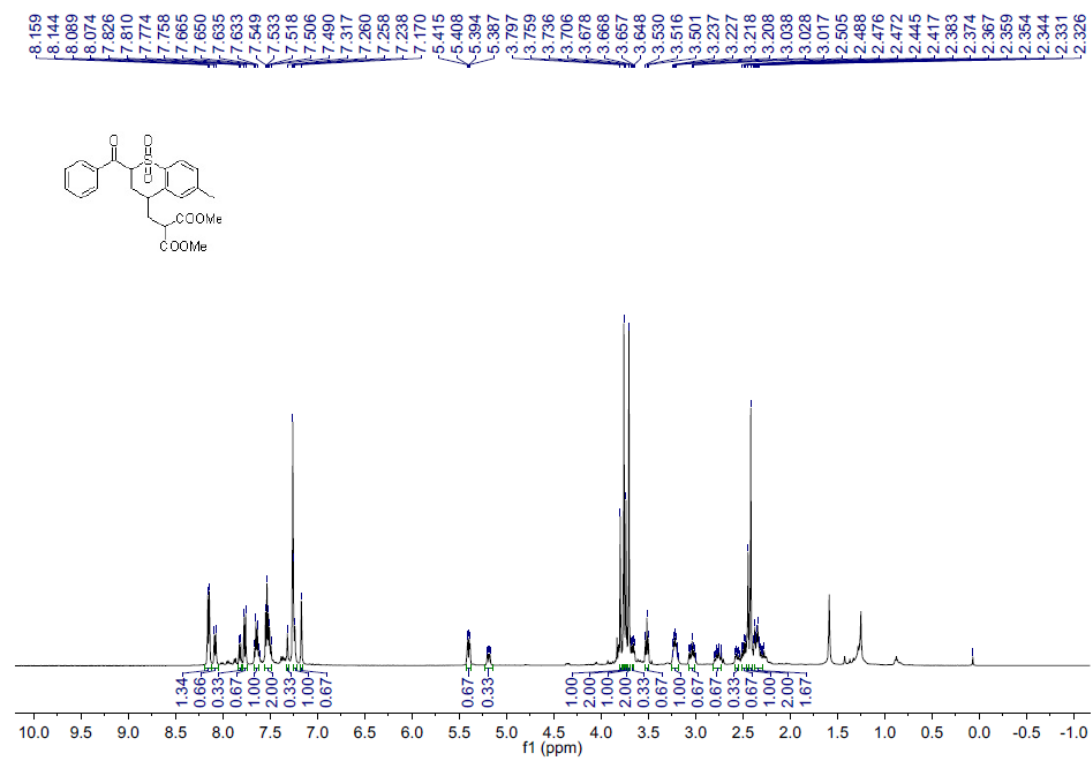
^1H NMR (500 MHz, CDCl_3) spectrum of compound **5ab**



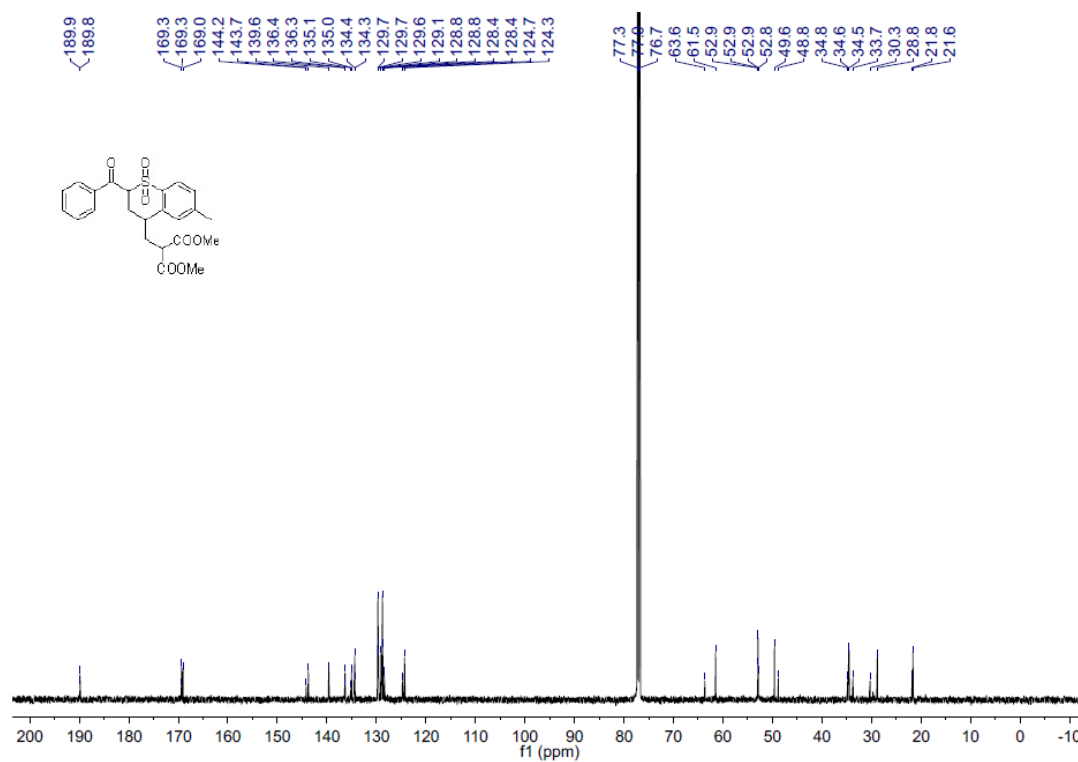
¹³C {¹H} NMR (126 MHz, CDCl₃) spectrum of compound **5ab**



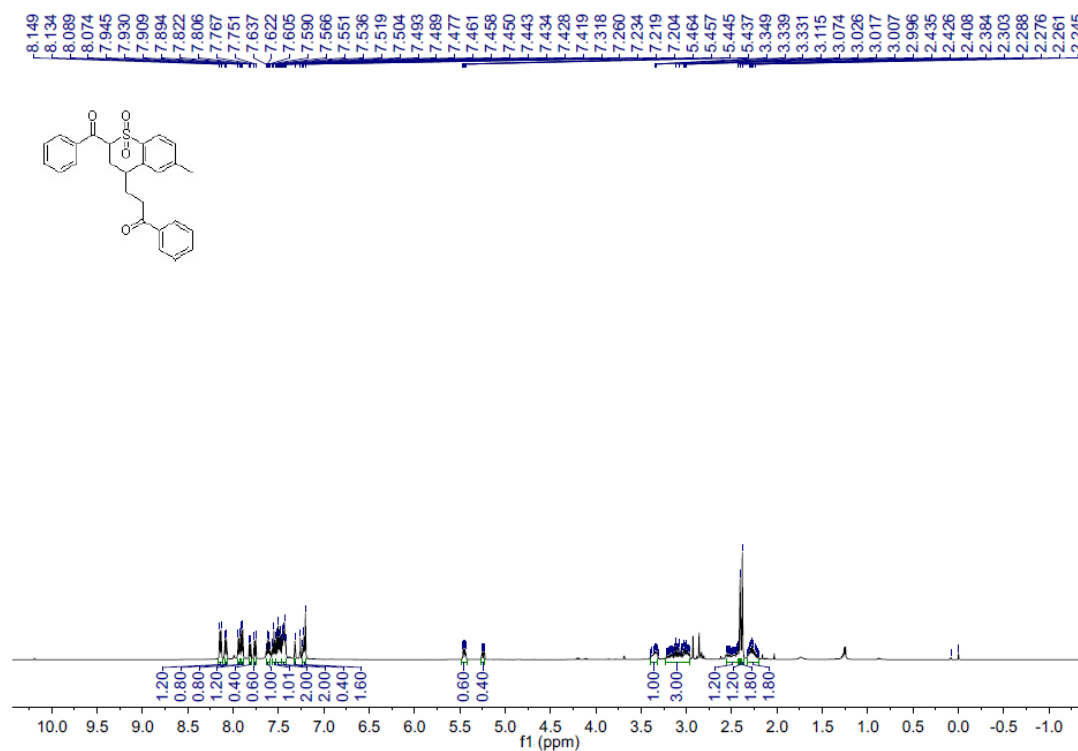
¹H NMR (500 MHz, CDCl₃) spectrum of compound **5ac**



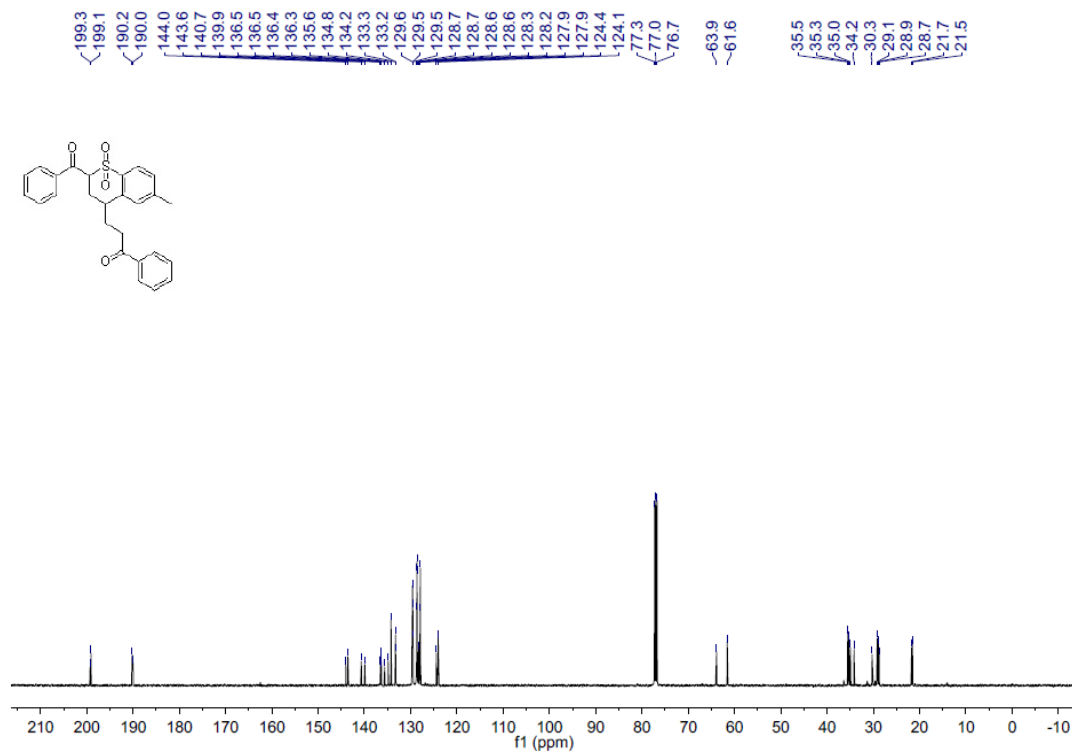
^{13}C $\{^1\text{H}\}$ NMR (126 MHz, CDCl_3) spectrum of compound **5ac**



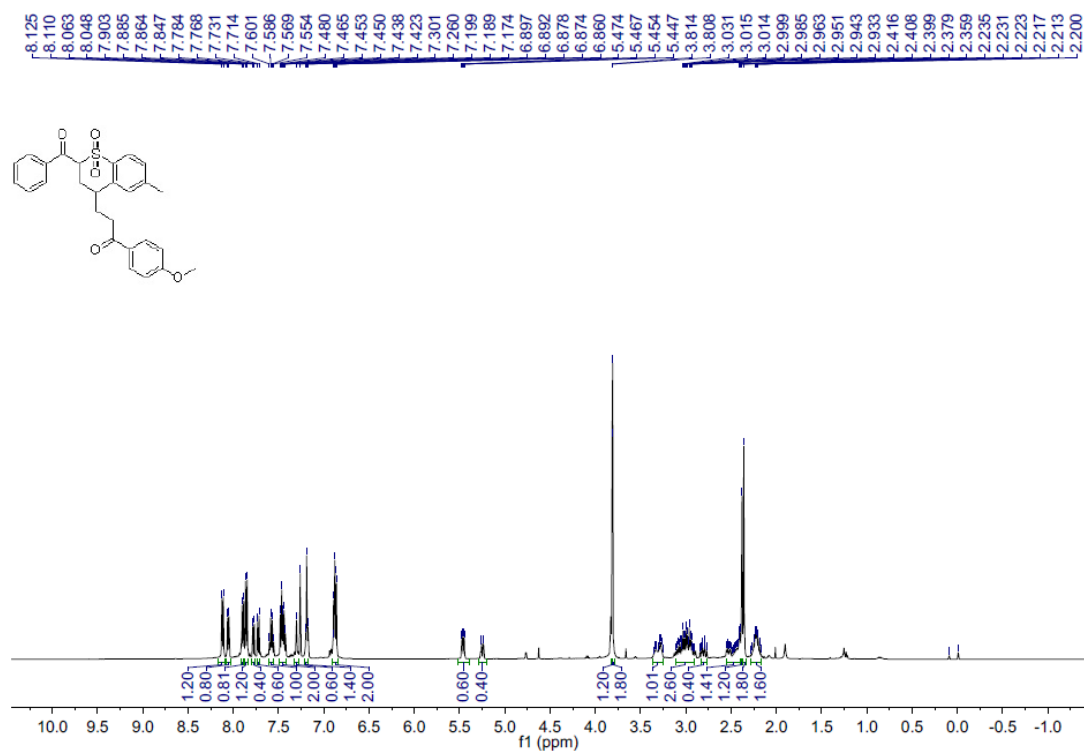
^1H NMR (500 MHz, CDCl_3) spectrum of compound **5af**



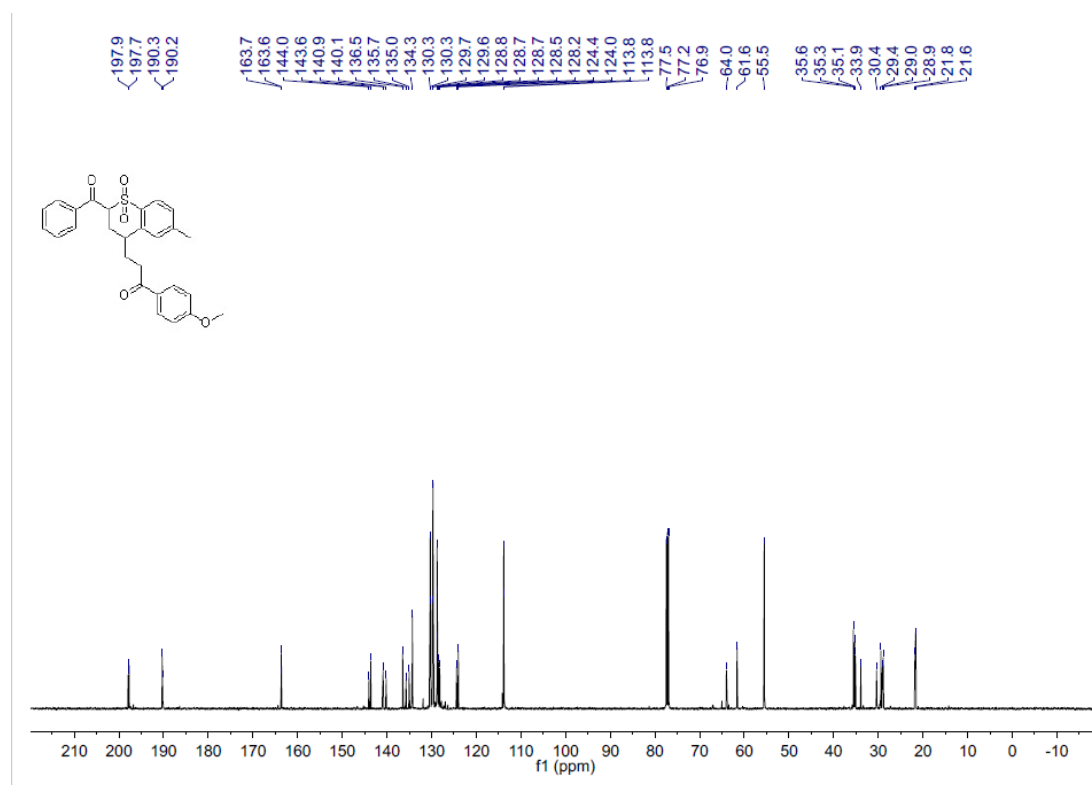
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **5af**



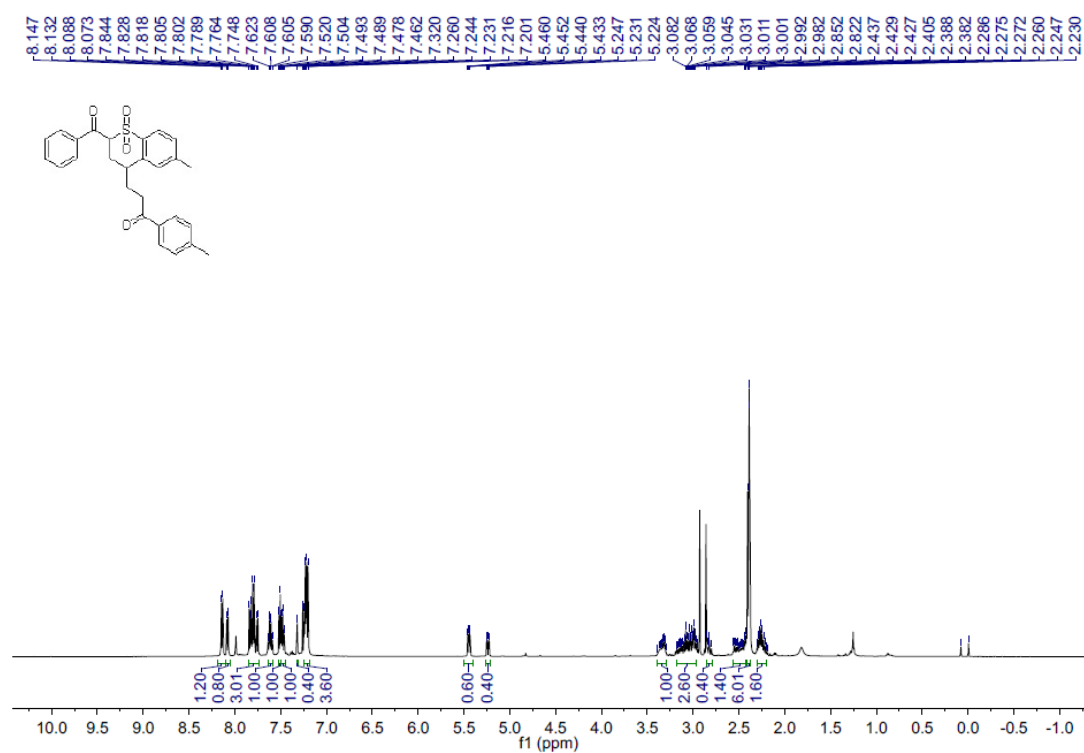
^1H NMR (500 MHz, CDCl_3) spectrum of compound **5ag**



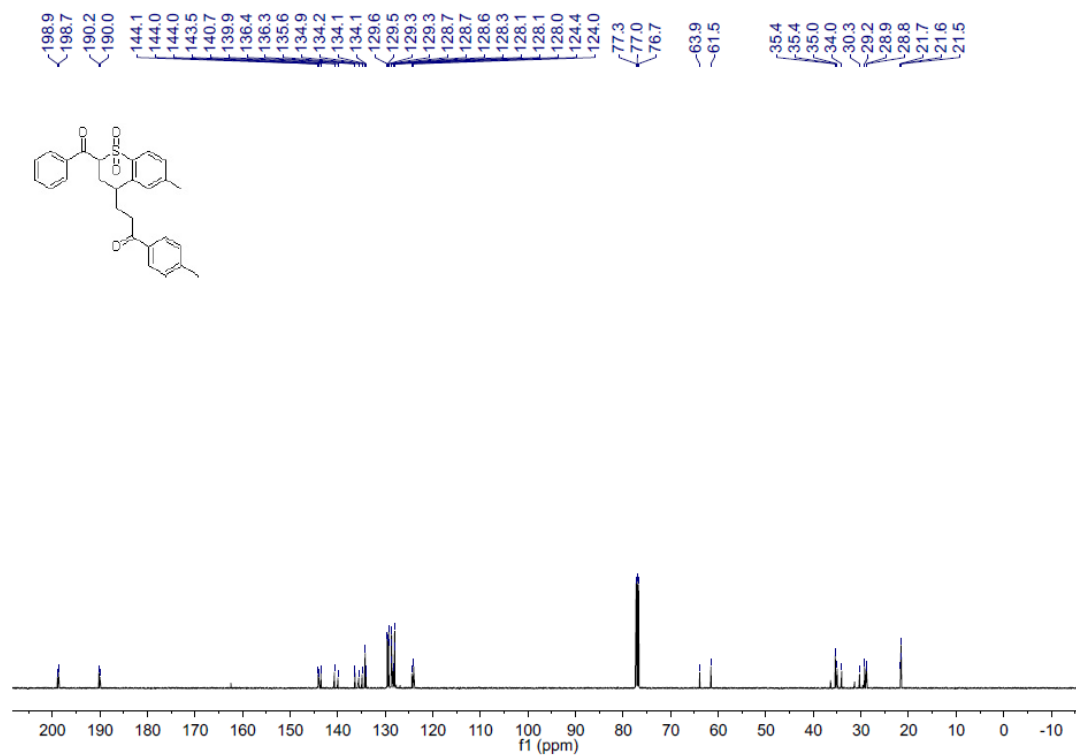
^{13}C $\{^1\text{H}\}$ NMR (126 MHz, CDCl_3) spectrum of compound **5ag**



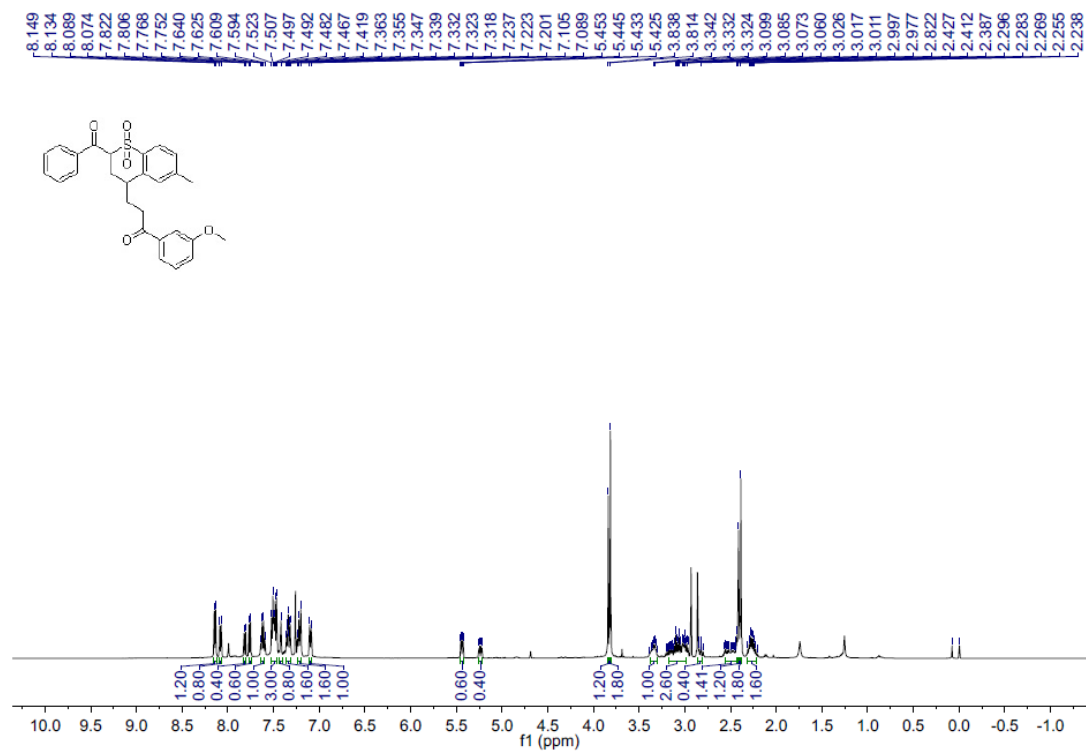
^1H NMR (500 MHz, CDCl_3) spectrum of compound **5ah**



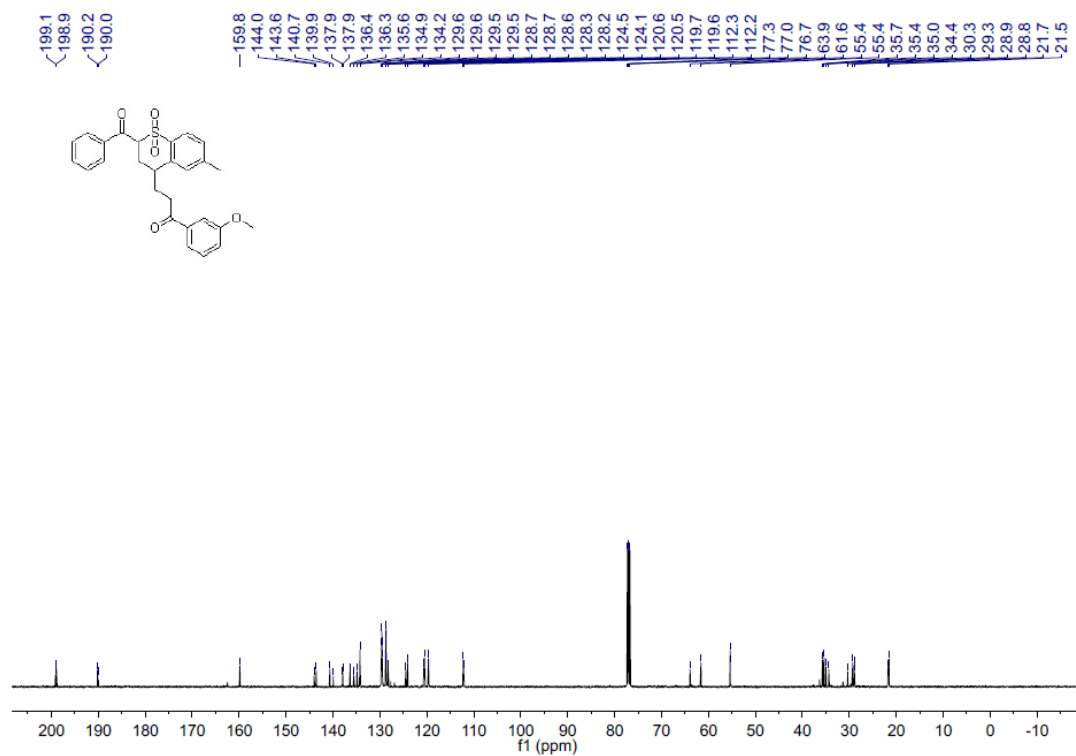
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **5ah**



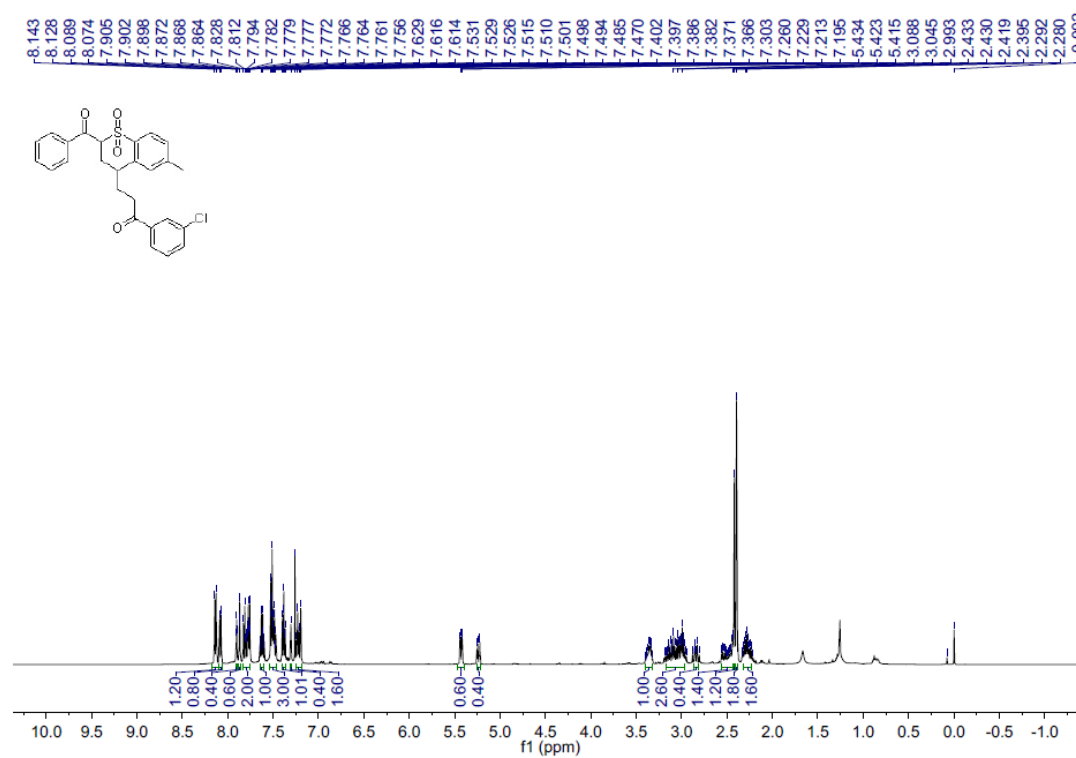
^1H NMR (500 MHz, CDCl_3) spectrum of compound **5ai**



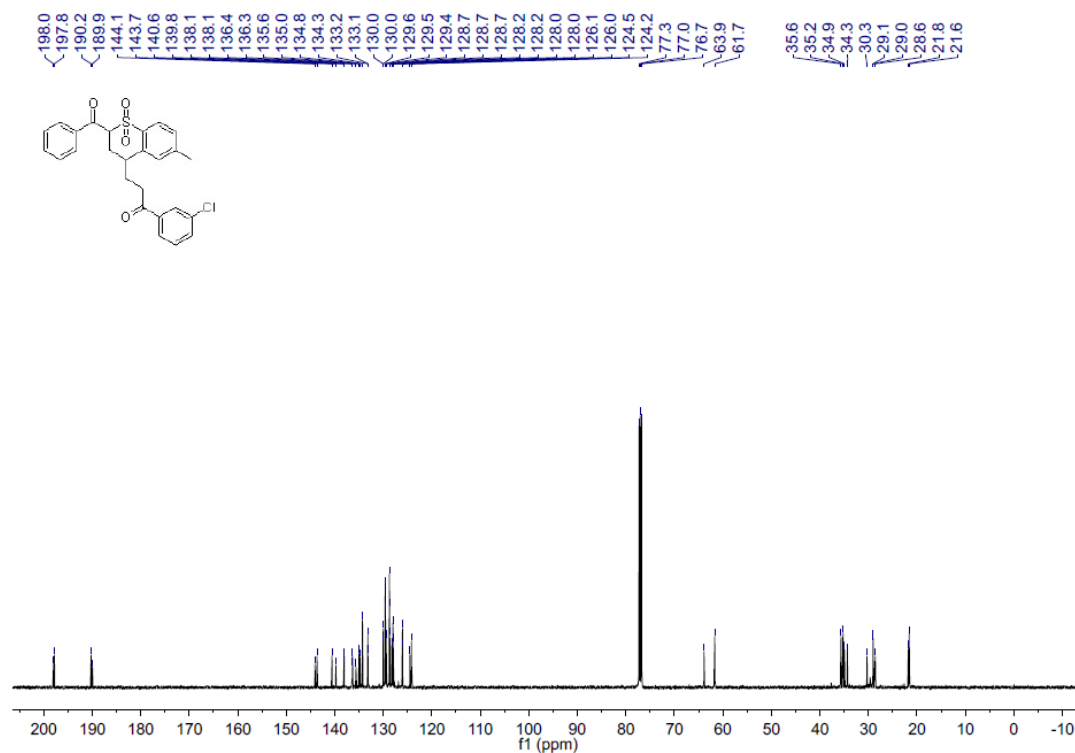
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **5ai**



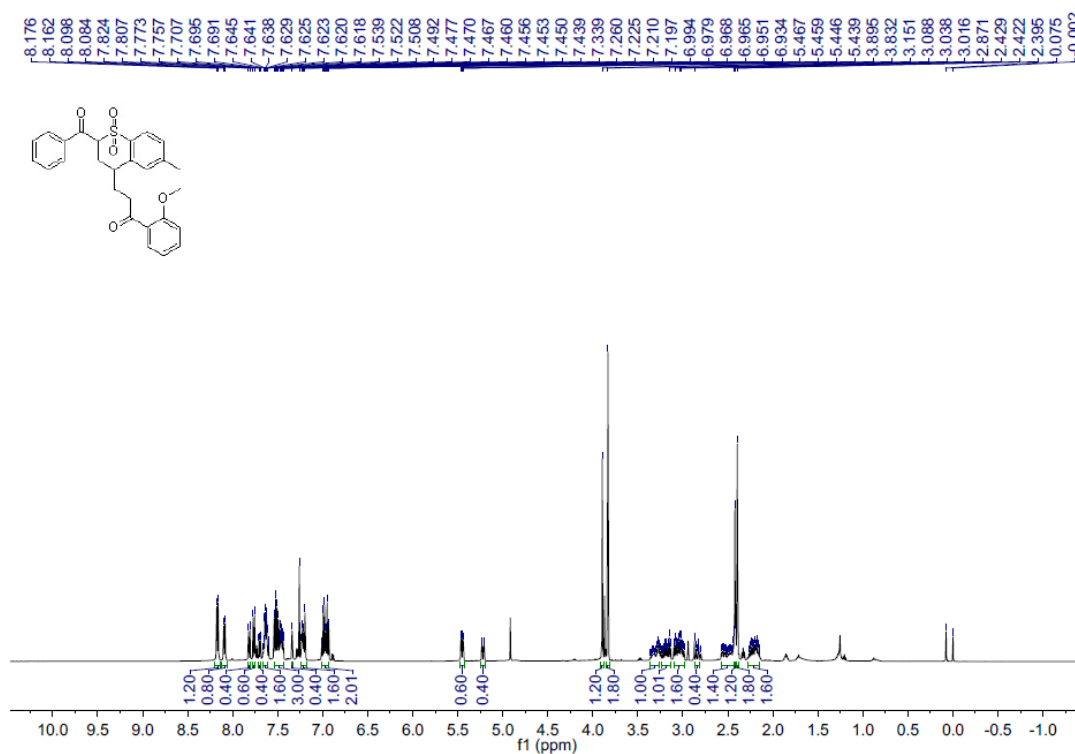
^1H NMR (500 MHz, CDCl_3) spectrum of compound **5aj**



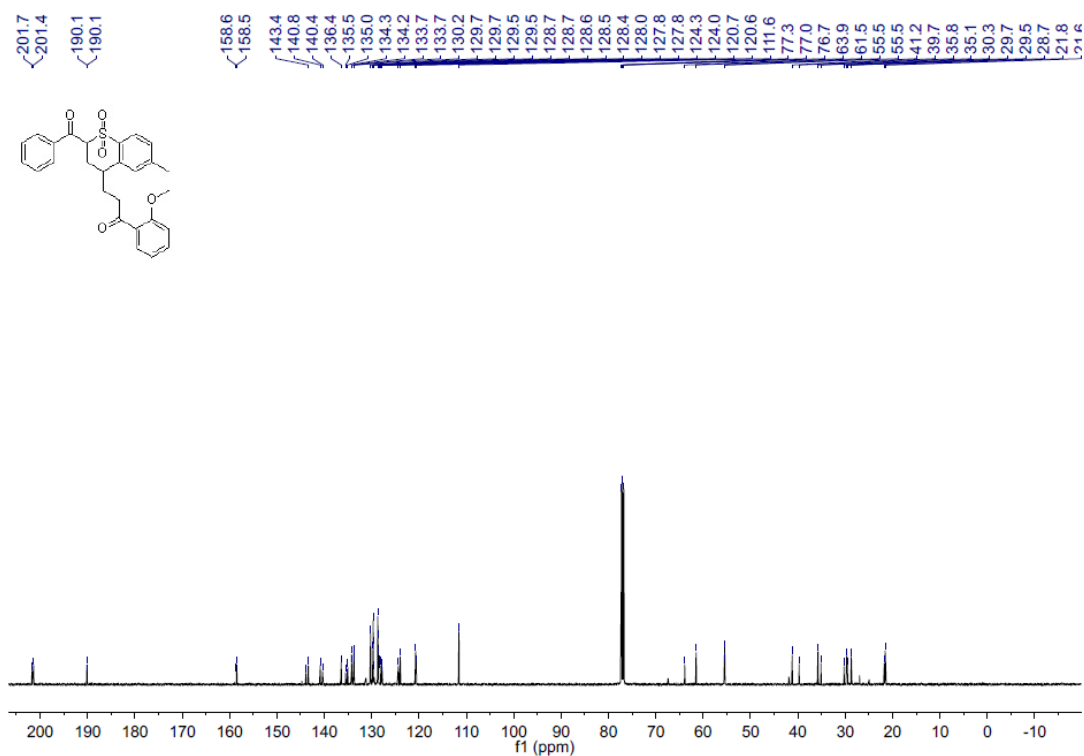
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **5aj**



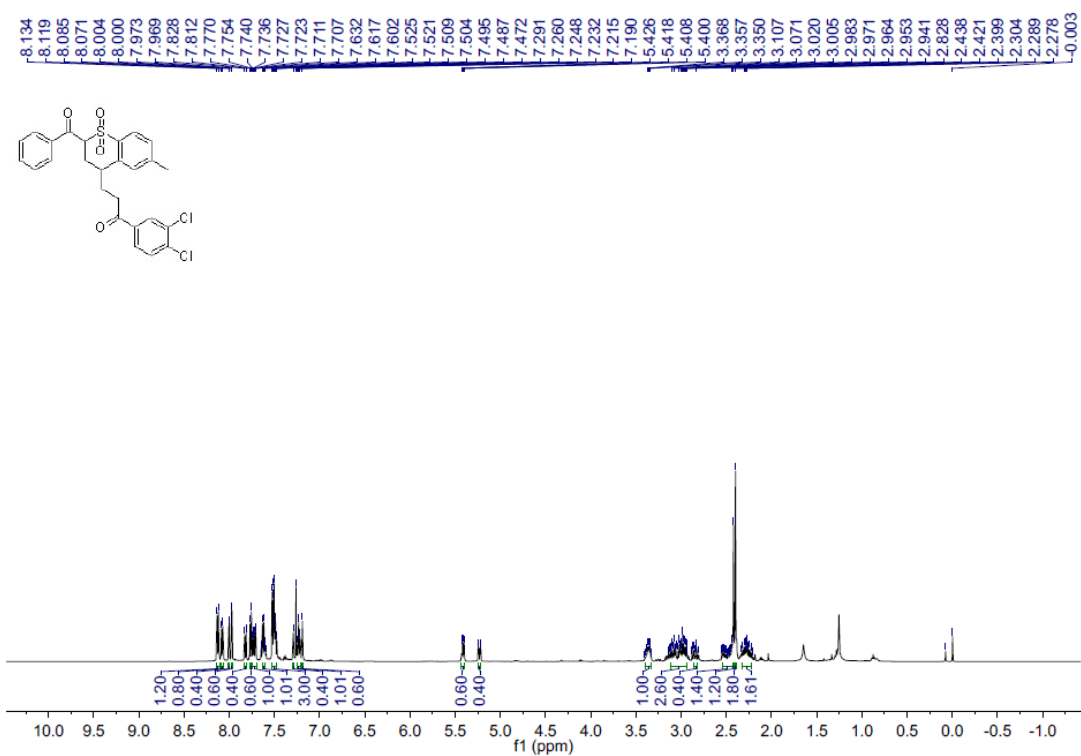
^1H NMR (500 MHz, CDCl_3) spectrum of compound **5ak**



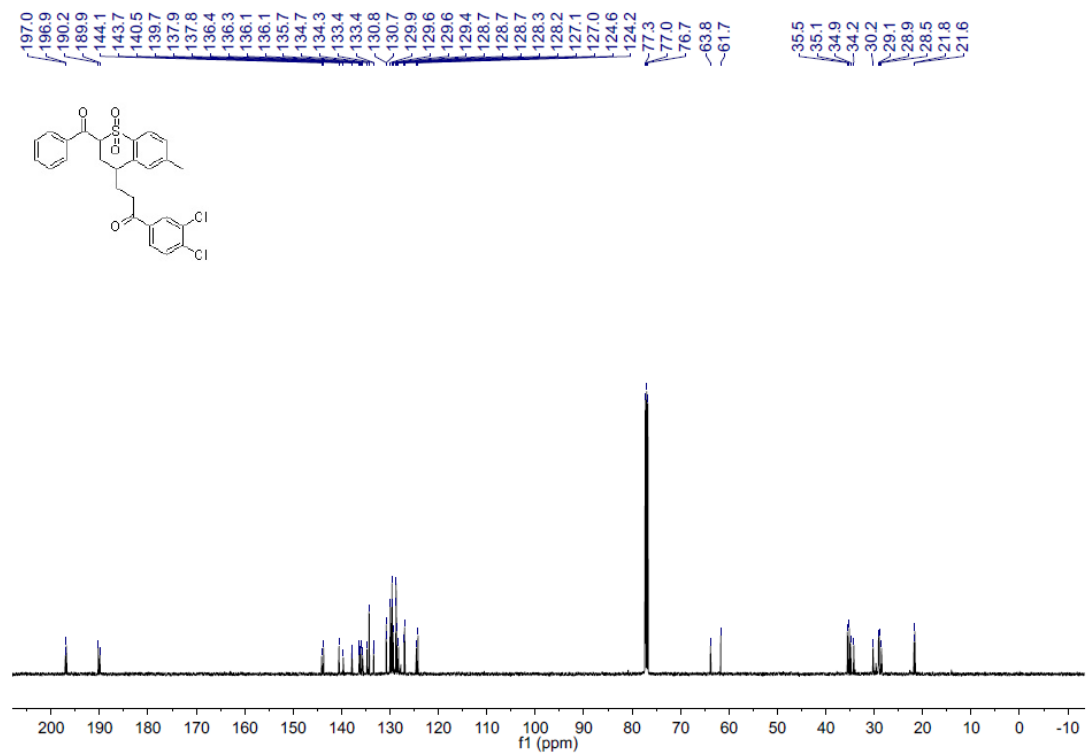
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **5ak**



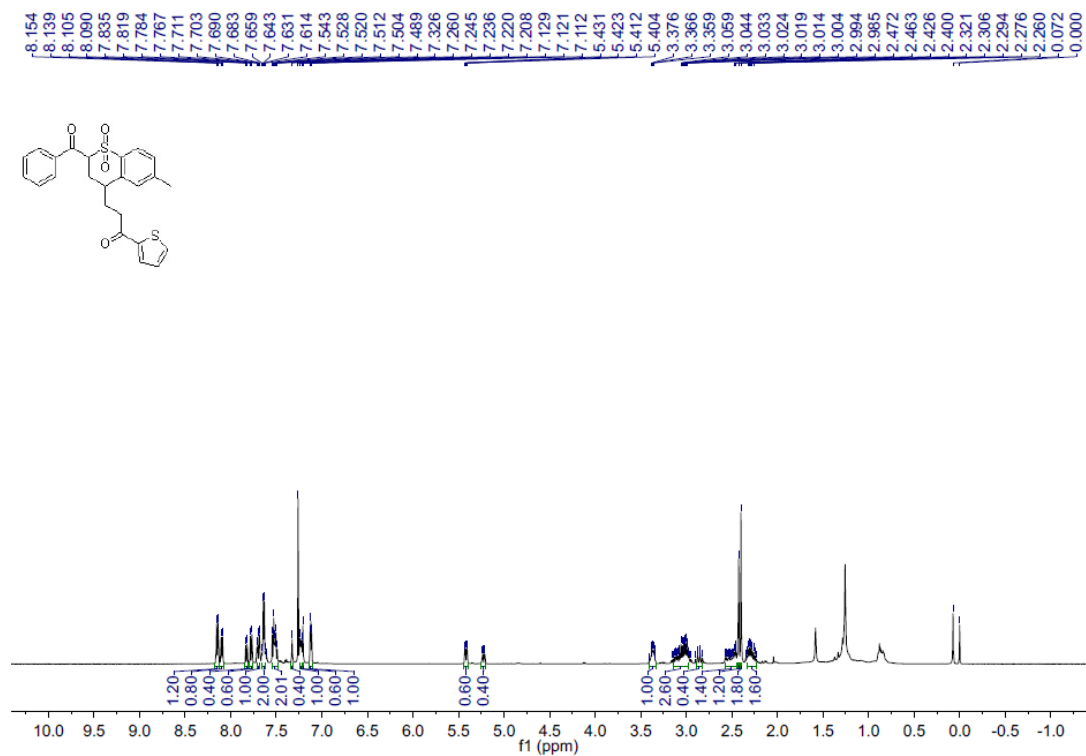
^1H NMR (500 MHz, CDCl_3) spectrum of compound **5al**



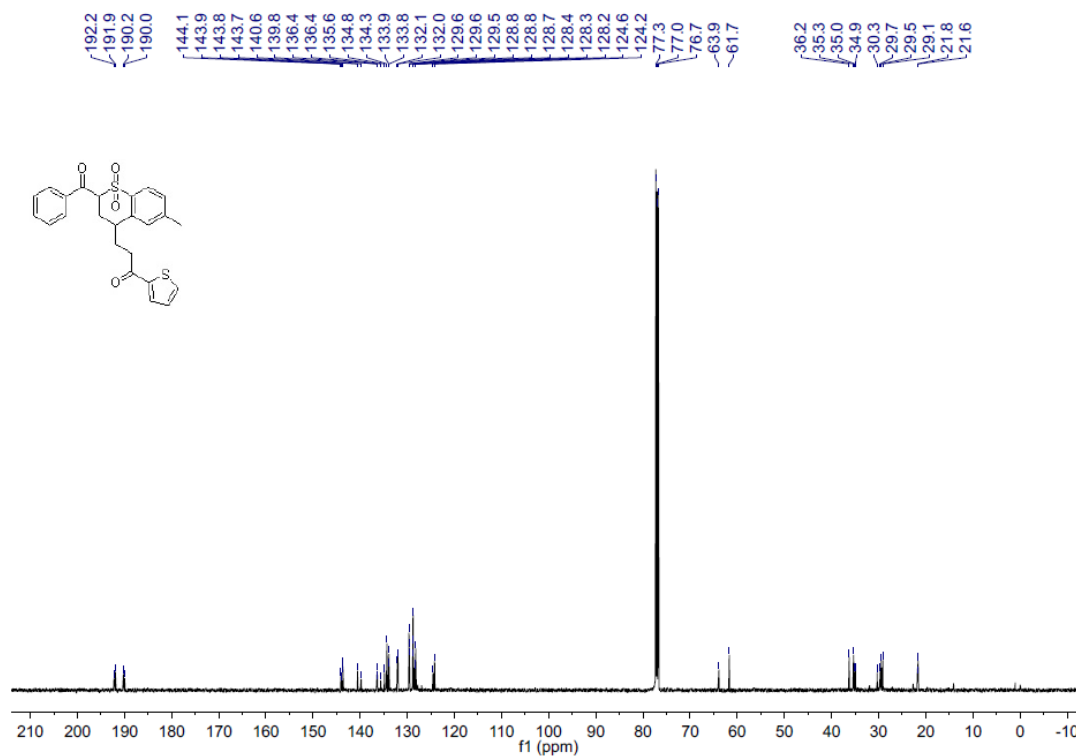
^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **5al**



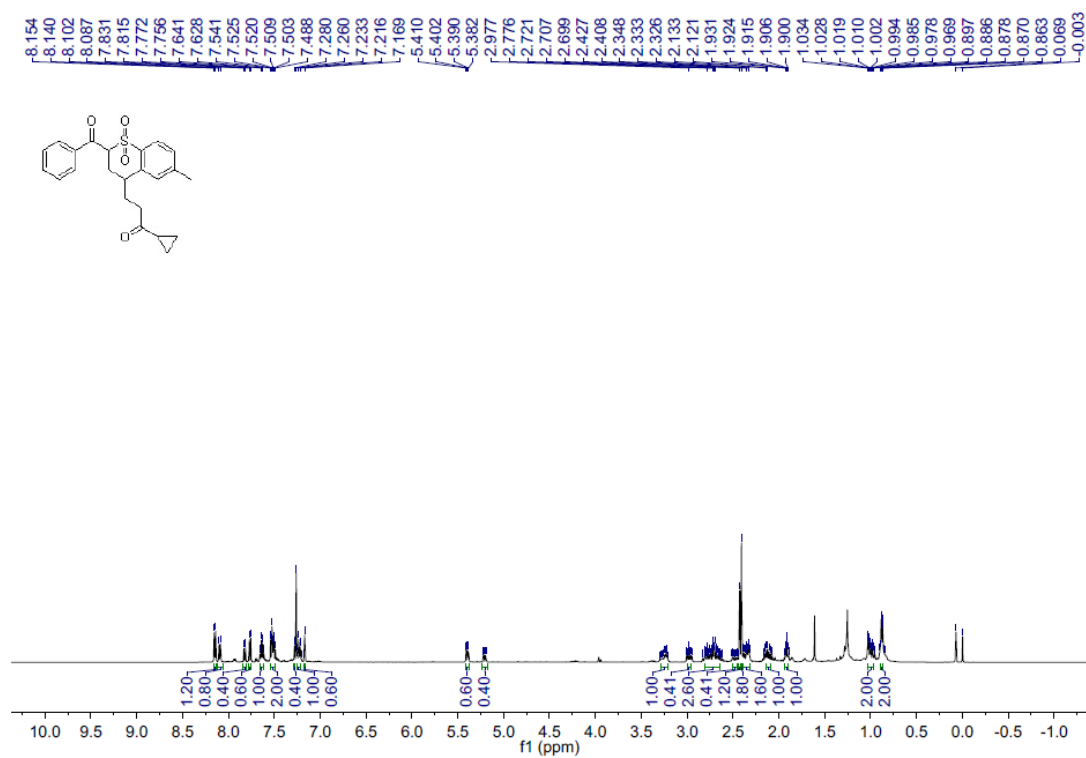
^1H NMR (500 MHz, CDCl_3) spectrum of compound **5am**



^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **5am**



^1H NMR (500 MHz, CDCl_3) spectrum of compound **5an**



^{13}C { ^1H } NMR (126 MHz, CDCl_3) spectrum of compound **5an**

