

Supporting Information

Photo-induced C(sp²)–H/C(sp²)–H Cross-Coupling of Alkenes: Direct Synthesis of 1,3-Dienes

Qi Xu, Baihui Zheng, Xiaoxuan Zhou, Ling Pan, Qun Liu, Yifei Li*

Jilin Province Key Laboratory of Organic Functional Molecules, Design & Synthesis, Department of Chemistry, Northeast Normal University, Changchun 130024, China

E-Mail: liyf640@nenu.edu.cn

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1. General Information

Unless otherwise noted, all reagents were purchased from commercial suppliers without further purification. The photocatalysts Mes-Acr⁺ClO₄⁻, Mes-Acr-Ph⁺BF₄⁻ and Mes-Acr-Ph⁺Cl⁻ were purchased from Aldrich. The cobalt complexes Co(dmgH)₂PyCl was purchased from Aldrich. Anhydrous dichloroethane (DCE) and acetonitrile (MeCN) were purchased from Energy Chemical.

Reactions were monitored by thin-layer chromatography (TLC) carried out on 0.25 mm Tsingdao silica gel plates (GF-254) using UV light as visualizing agent. Tsingdao silica gel (60, particle size 0.040-0.063 mm) was used for flash column chromatography.

NMR spectra were recorded on a Brüker Advance 600 (¹H: 600 MHz, ¹³C: 151 MHz and 126 MHz) at ambient temperature. Data were reported as chemical shifts in ppm relative to TMS (0.00 ppm) for ¹H NMR and CDCl₃ (77.2 ppm) for ¹³C NMR. The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. Infrared spectroscopy (IR) were obtained by Fourier Transform Infrared Spectrometer (FT-IR). High-resolution mass spectra (HRMS) were obtained using a Bruker micro TOF II focus spectrometer (ESI).

The source of the blue LED using Wattecs Parallel Light Reactor WP-TEC-1020HC (Blue LED Light source, every tube hole groove, 5 W). UV-vis absorption analysis using Varian; Stern-Volmer fluorescence quenching experiments using spectrofluorometer (Edinburgh FS5) and Cyclic Voltammetry experiments using CH Instruments (CHI 660E).

Instruments of Photochemical Reactions



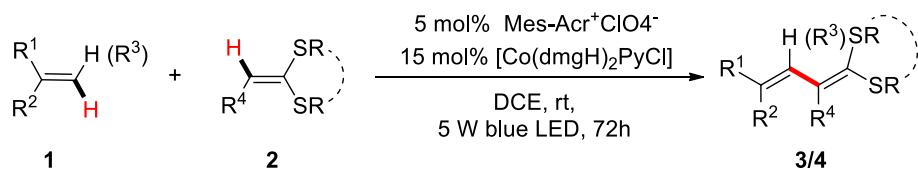
Figure S1. Blue LED Light source, every tube hole groove, 5 W

2. Experimental Procedure

2.1 Preparation of Catalysts and Substrates

α -Ketene dithioacetal **2a–2n**, **2p** and α -cyanoketene dithioacetal **2o** are known compounds according to reported procedures.^[1] The cobalt complexes $[\text{Co}(\text{dmgH})_2\text{Py}_2]\text{PF}_6$ were prepared according to the reported procedures.^[2]

2.2 Typical Procedures for C(sp²)–H/C(sp²)–H cross-coupling



Typical synthetic procedure (with **3a** as an example): To a 15 mL flask equipped with a stir-bar was added α -ketene dithioacetal **2a** (80 mg, 0.5 mmol), Mes-Acr⁺ClO₄⁻ (10 mg, 0.025 mmol), Co(dmgH)₂PyCl (30 mg, 0.075 mmol) and 2 mL DCE. Then styrene **1a** (189 μ L, 1.5 mmol) was added with stirring. The reaction mixture was stirred for 72 h at Wattecs Parallel Light Reactor (Blue LED Light source, every tube hole groove, 5 W) at ambient temperature. After the completion of the reaction as indicated by TLC, the mixture was quenched with saturated NaHCO₃ (20 mL), extracted with DCE (20 mL \times 3), washed with brine (20 mL), and dried over anhydrous Na₂SO₄. The solution was concentrated by rotary evaporation, then the residue was purified by silica gel flash column chromatography (PE/EA = 4/1) to afford product **3a** (yellow solid, 114 mg, 87%).

Experimental phenomenon:

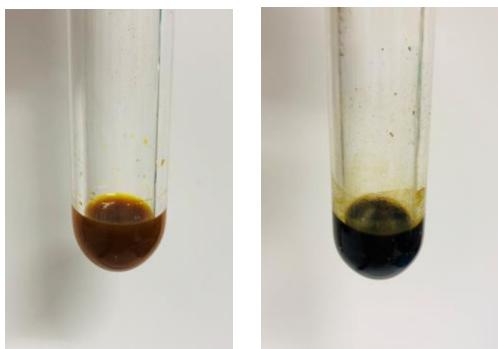


Figure S2. Typical reaction mixture before (light brown) and after (dark brown) 5 W blue LED irradiation 72 h.

2.3 Procedure for gram-scale synthesis of **3a**

Condition 1: To a 15 mL quartz tube with a stir-bar was added α -ketene dithioacetal **2a** (1.12 g, 7 mmol), Mes-Acr $^+$ ClO $_4^-$ (144 mg, 0.35 mmol), Co(dmgH) $_2$ PyCl (424 mg, 1.05 mmol) and DCE (10 mL). Then styrene **1a** (1.9 mL, 15 mmol) was added with stirring. The reaction mixture was stirred for 72 h at Wattecs Parallel Light Reactor (Blue LED Light source, 5 W) at ambient temperature. After the completion of the reaction as indicated by TLC, the mixture was quenched with saturated NaHCO $_3$ (20 mL), extracted with DCE (20 mL \times 3), washed with brine (20 mL), and dried over anhydrous Na $_2$ SO $_4$. The solution was concentrated by rotary evaporation, then the residue was purified by silica gel flash column chromatography (PE/EA = 4/1) to afford product **3a** (yellow solid, 1.3g, 71%).

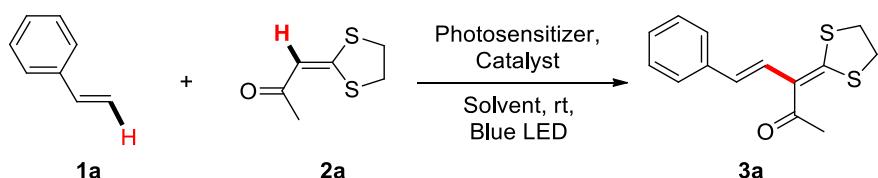
Condition 2: To a 200 mL wide-mouth reagent bottle was added α -ketene dithioacetal **2a** (1.12 g, 7mmol), Mes-Acr $^+$ ClO $_4^-$ (144 mg, 0.35 mmol), Co(dmgH) $_2$ PyCl (424 mg, 1.05 mmol) and DCE (70 mL). Then styrene **1a** (1.9 mL, 15 mmol) was added. The reaction mixture was exposed to sunlight for 4 days without stir at ambient temperature. After the completion of the reaction as indicated by TLC, the mixture was quenched with saturated NaHCO $_3$ (20 mL), extracted with DCE (20 mL \times 3), washed with brine (20 mL), and dried over anhydrous Na $_2$ SO $_4$. The solution was concentrated by rotary evaporation, then the residue was purified by silica gel flash column chromatography (PE/EA = 4/1) to afford product **3a** (yellow solid, 1.2g, 67%).

Phenomenon of gram-scale synthesis of condition 2:



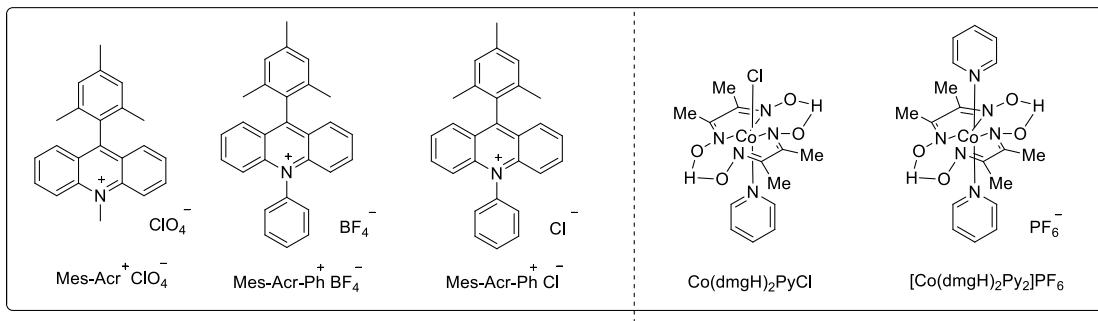
Figure S3. Gram-scale mixture before (light brown) and after (dark brown) under sunlight 4 days.

3. Optimization of Photocatalytic Reactions^{a,b}



Entry	1a:2a	Photosensitizer (mol%)	Catalyst (mol%)	Solvent	Blue LEDs	Yield (%)
1	2:1	3 mmol% Mes-Acr ⁺ ClO ₄ ⁻	8 mmol% Co(dmgH) ₂ PyCl	DCE	3 W	74%
2	0:1	3 mmol% Mes-Acr ⁺ ClO ₄ ⁻	8 mmol% Co(dmgH) ₂ PyCl	DCE	3 W	nr
3	1:0	3 mmol% Mes-Acr ⁺ ClO ₄ ⁻	8 mmol% Co(dmgH) ₂ PyCl	DCE	3 W	nr
4	2:1	3 mmol% Mes-Acr ⁺ ClO ₄ ⁻	8 mmol% Co(dmgH) ₂ PyCl	DCE	Black	nr
5	2:1	3 mmol% Mes-Acr ⁺ ClO ₄ ⁻	None	DCE	3 W	nr
6	2:1	None	8 mmol% Co(dmgH) ₂ PyCl	DCE	3 W	nr
7	2:1	5 mmol% Mes-Acr ⁺ ClO ₄ ⁻	20 mmol% PhSSPh	DCE	3 W	nr
8	2:1	5 mmol% Mes-Acr ⁺ ClO ₄ ⁻	20 mmol% Cu(OAc) ₂	DCE	3 W	nr
9	2:1	5 mmol% Mes-Acr ⁺ ClO ₄ ⁻	8 mmol% [Co(dmgH) ₂ Py] ₂ PF ₆	DCE	3 W	61 %
10	2:1	3 mmol% Mes-Acr-Ph ⁺ BF ₄ ⁻	8 mmol% Co(dmgH) ₂ PyCl	DCE	3 W	68%
11	2:1	3 mmol% Mes-Acr-Ph ⁺ Cl ⁻	8 mmol% Co(dmgH) ₂ PyCl	DCE	3 W	67%
12 ^c	2:1	3 mmol% Ir(ppy) ₃	8 mmol% Co(dmgH) ₂ PyCl	DCE	3 W	nr
13 ^c	2:1	3 mmol% Ru(bpy) ₃ Cl ₂ ·6H ₂ O	8 mmol% Co(dmgH) ₂ PyCl	DCE	3 W	nr
14	2:1	3 mmol% Mes-Acr ⁺ ClO ₄ ⁻	8 mmol% Co(dmgH) ₂ PyCl	DCE	5 W	73 %

15	1.5:1	3 mmol% Mes-Acr ⁺ ClO ₄ ⁻	8 mmol% Co(dmgH) ₂ PyCl	DCE	5 W	73 %
16	3:1	3 mmol% Mes-Acr ⁺ ClO ₄ ⁻	8 mmol% Co(dmgH) ₂ PyCl	DCE	5 W	80%
17	3:1	3 mmol% Mes-Acr ⁺ ClO ₄ ⁻	8 mmol% Co(dmgH) ₂ PyCl	MeCN	5 W	64 %
18	3:1	3 mmol% Mes-Acr ⁺ ClO ₄ ⁻	8 mmol% Co(dmgH) ₂ PyCl	DCM	5 W	78 %
19	3:1	5 mmol% Mes-Acr⁺ClO₄⁻	15 mmol% Co(dmgH)₂PyCl	DCE	5 W	87%
20 ^c	3:1	5 mmol% Mes-Acr ⁺ ClO ₄ ⁻	8 mmol% Co(dmgH) ₂ PyCl	DCE	5 W	67%
21	1.5:1	5 mmol% Mes-Acr ⁺ ClO ₄ ⁻	15 mmol% Co(dmgH) ₂ PyCl	DCE	5 W	78%



^a Styrene **1a** (0.5 mmol), α -acetyl dithioacetal **2a** (x mmol), photosensitizer (x mol%), catalyst (x mol %), and solvent (2 mL) were stirred in a tube irradiated using 5 W blue LEDs at rt. ^b Isolated Yields. ^c Under N₂ protection.

4. Mechanistic studies

4.1 UV-vis spectrum

UV-vis absorption of a solution of styrene **1a** (0.5 mmol), α -acetyl dithioacetal **2a** (0.5 mmol), Mes-Acr⁺ClO₄⁻ (0.025mmol, 5 mol %, 0.25 mM), Co(dmgH)₂PyCl (0.075 mmol, 15 mol %, 0.75 mM) in 100 mL DCE with 5 W blue LED.

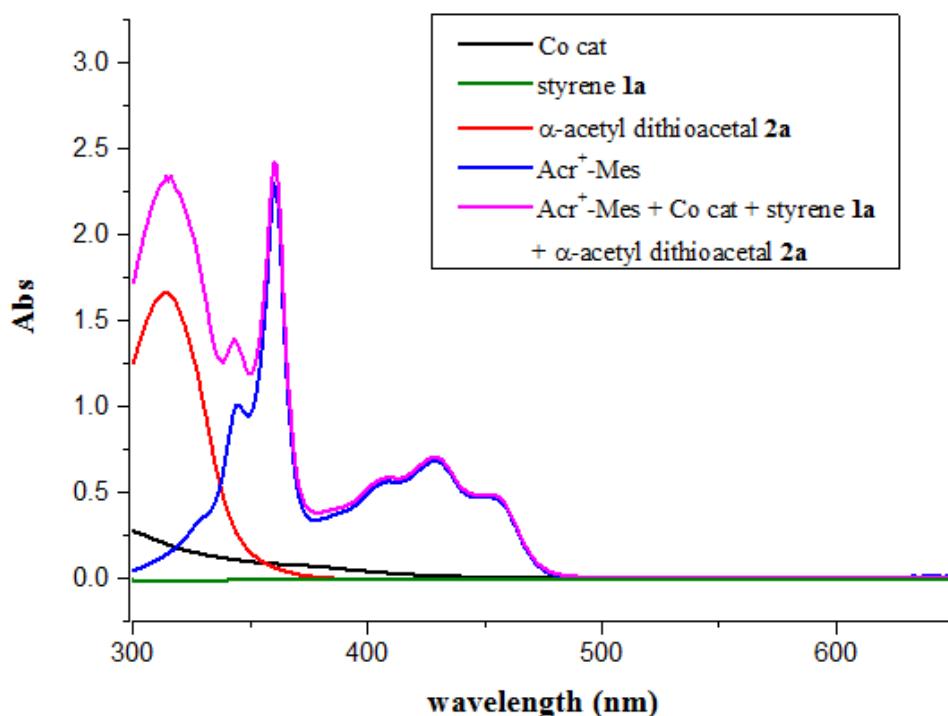


Figure S4. UV-vis spectrum of styrene **1a**, α -acetyl dithioacetal **2a**, Mes-Acr⁺ClO₄⁻ and Co(dmgH)₂PyCl

4.2 Emission Quenching Experiments

Emission intensities were recorded using spectrofluorometer (Edinburgh FS5) at ambient temperature. All Mes-Acr⁺ClO₄⁻ solutions were excited at 450 nm and the emission intensity at 507 nm was observed.^[3] Firstly, the emission spectrum of a 5×10^{-5} M solution of Mes-Acr⁺ClO₄⁻ in DCM was collected. Then, appropriate amount of quencher was added to the measured solution and the emission spectrum of the sample was collected.

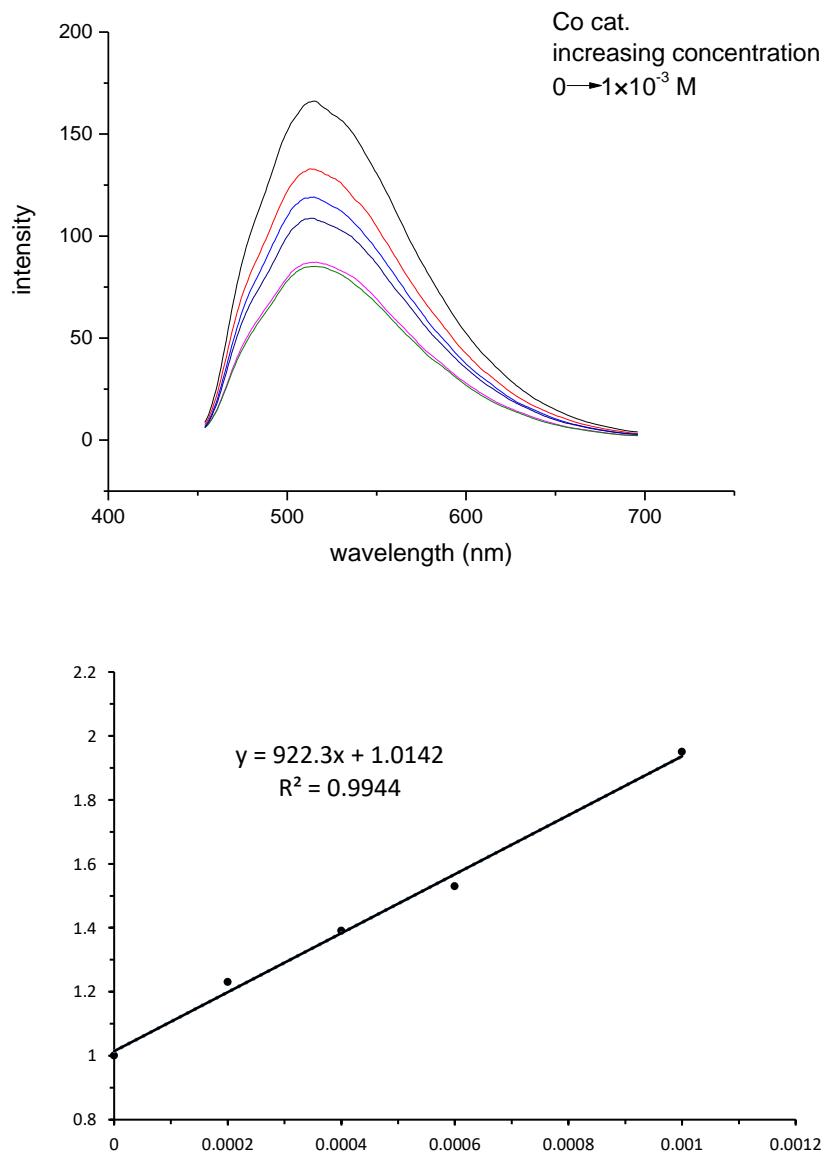


Figure S5. Mes-Acr⁺ClO₄⁻ Emission Quenching by Co(dmgH)₂PyCl catalyst

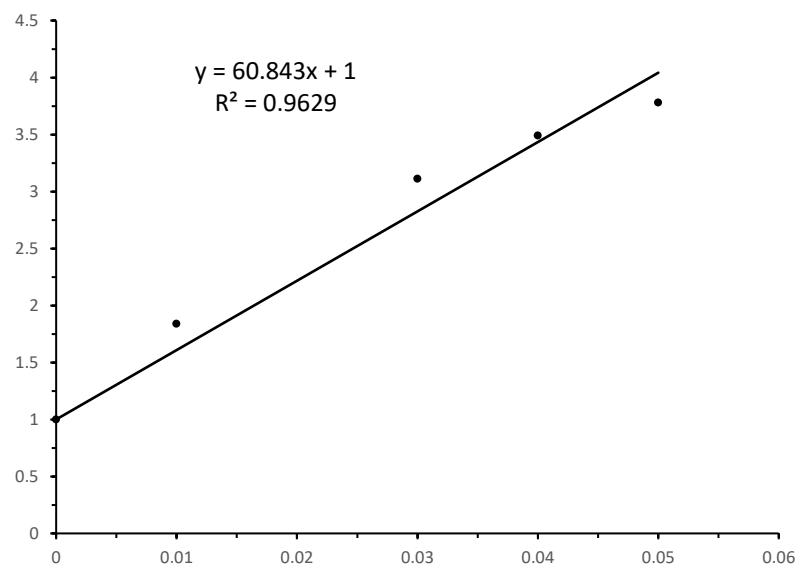
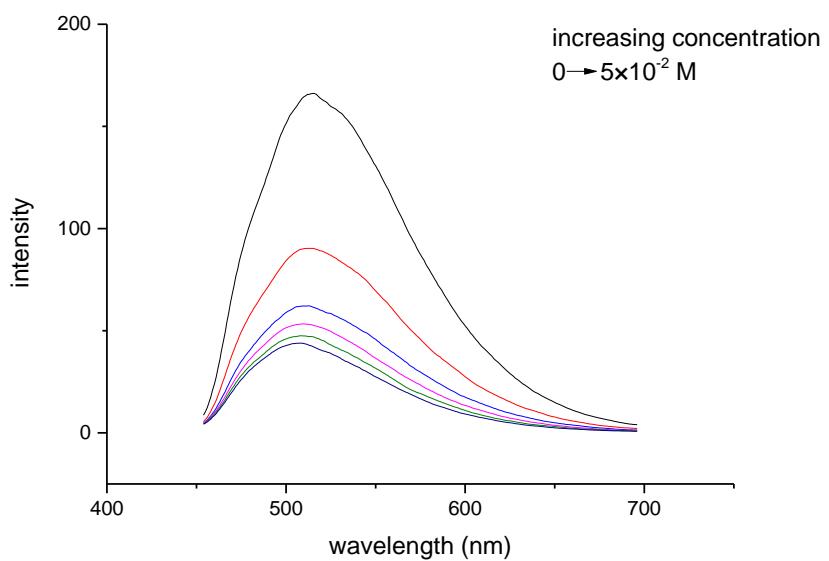


Figure S6. Mes-Acr⁺ClO₄⁻ Emission Quenching by α -ketene dithioacetal **2a**

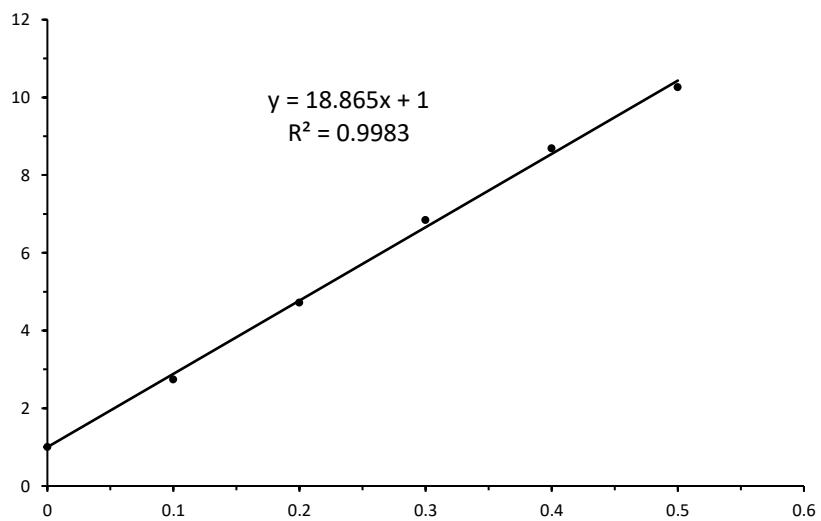
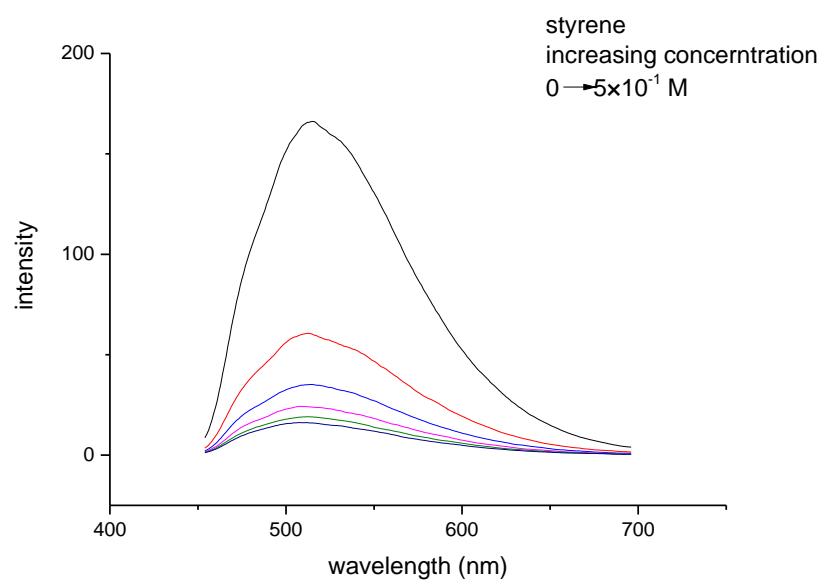


Figure S7. Mes-Acr⁺ClO₄⁻ Emission Quenching by styrene **1a**

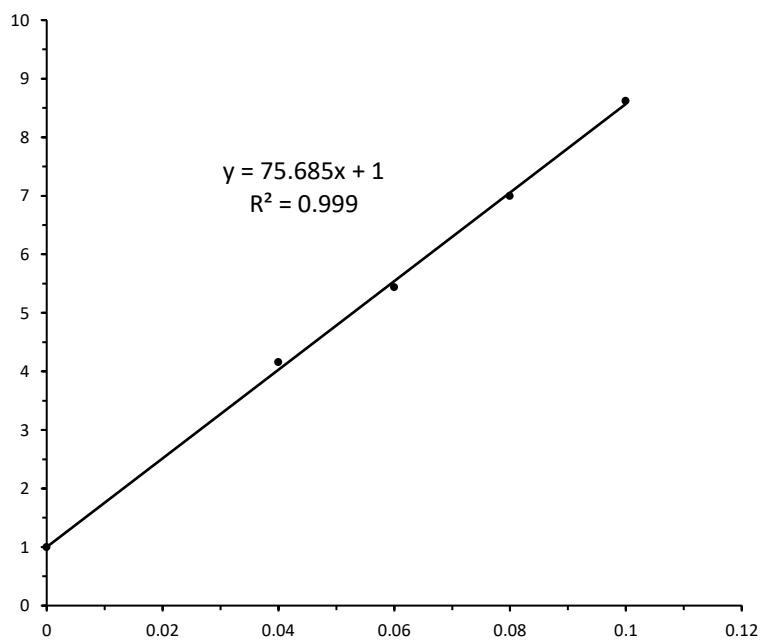
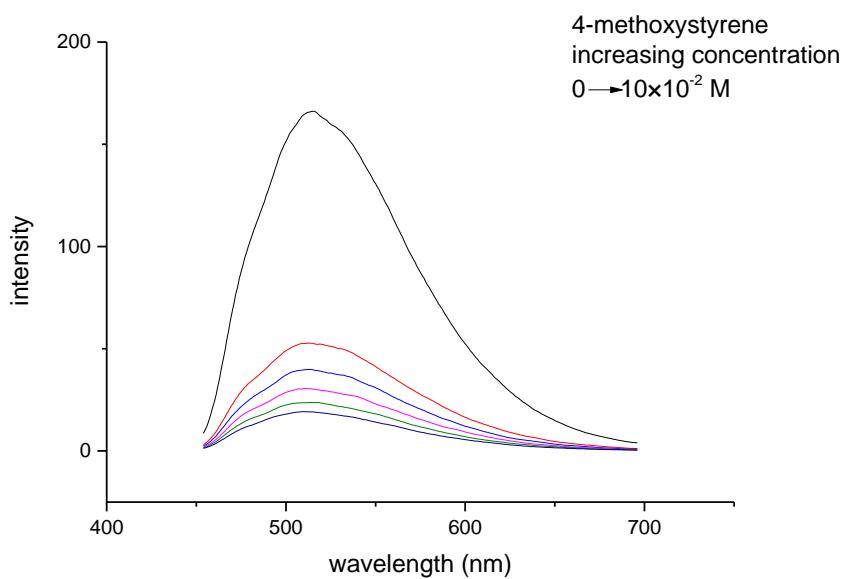


Figure S8. Mes-Acr⁺ClO₄⁻ Emission Quenching by 4-methoxystyrene **1m**

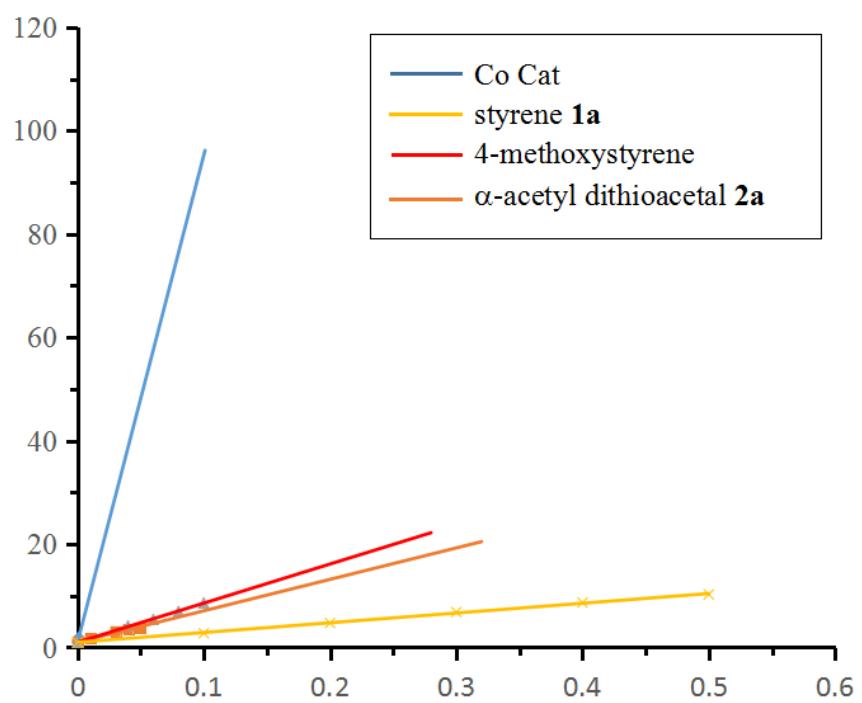


Figure S9. Emission-quenching experiments of $\text{Mes}-\text{Acr}^+\text{ClO}_4^-$ with $\text{Co}(\text{dmgH})_2\text{PyCl}$, α -ketene dithioacetal **2a**, styrene **1a** and 4-methoxystyrene **1m**

4.3 Cyclic Voltammetry (CV) Experiments

For the electrochemical measurements a three-electrode system connected to an electrochemical station was used. The reference electrode, Ag/AgCl in 0.1 M KCl. A glassy carbon electrode was used as working electrode. And a Pt wire was used as counter electrode. All electrochemical measurements were performed in degassed DMF under dry N₂ atmosphere.

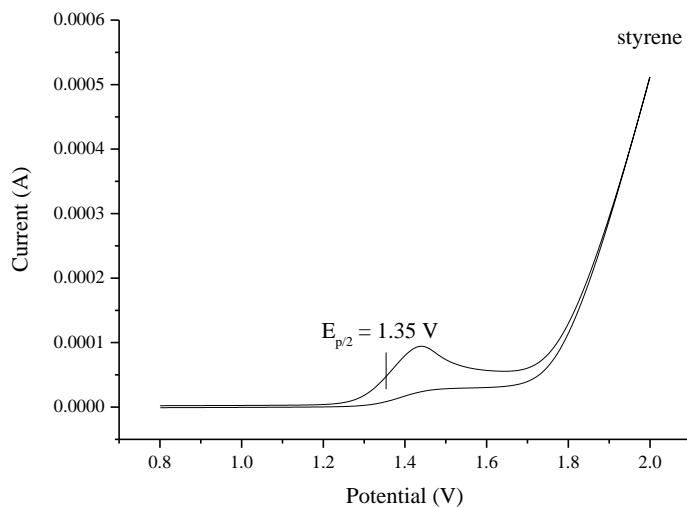


Figure S10. CV spectra of 3 mM styrene **1a** in 0.1 M NBu₄PF₆ in degassed DMF with scan rate 100 mV/s. The oxidation potential $E_{p/2}^{1\text{a}+\bullet/1\text{a}}$ (+1.54 V vs. SCE) was converted from internal standard SCE.

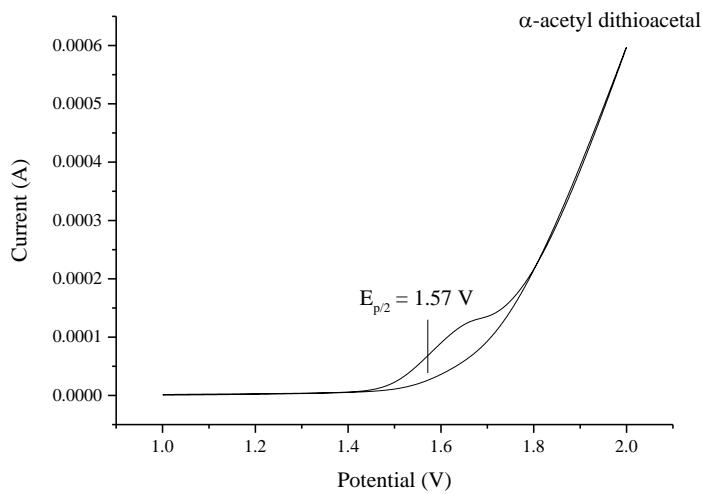


Figure S11. CV spectra of 3 mM α -acetyl dithioacetal **2a** in 0.1 M NBu₄PF₆ in degassed DMF with scan rate 100 mV/s. The oxidation potential $E_{p/2}^{2\text{a}+\bullet/2\text{a}}$ (+1.76 V vs. SCE) was converted from internal standard SCE.

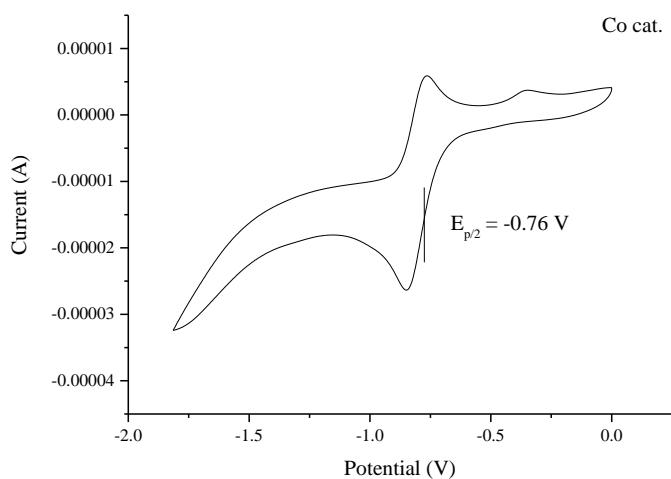


Figure S12. CV spectra of 1 mM $\text{Co}(\text{dmgH})_2\text{PyCl}$ in 0.1 M NBu_4PF_6 in degassed DMF with scan rate 100 mV/s. The oxidation potential $E_{p/2}^{\text{Co(III)/Co(II)}}$ (-0.57 V vs. SCE) was converted from internal standard SCE.

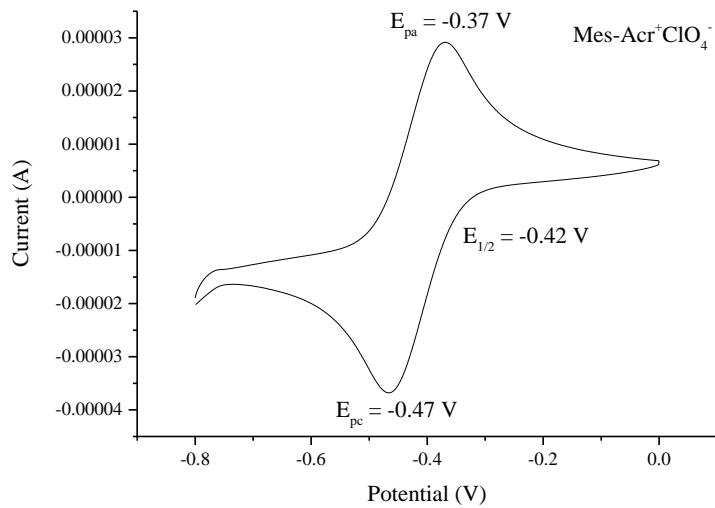


Figure S13. CV spectra of 3 mM $\text{Acr}^+\text{-Mes ClO}_4^-$ in 0.1 M NBu_4PF_6 in degassed DMF with scan rate 100 mV/s. The reduction potential $E_{1/2}^{\text{Acr}^+\text{-MesClO}_4^-/\text{Acr}^+\text{-MesClO}_4^+}$ (-0.23 V vs. SCE) was converted from internal standard SCE.

References

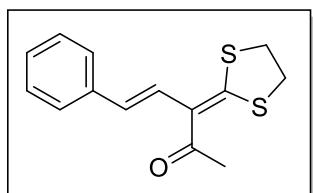
- [1] (a). Junjappa, H.; Ila, H. and Asokan, C. V. α -Oxoketene- S,S -, N,S - and N,N -acetals: Versatile intermediates in organic synthesis. *Tetrahedron*, **1990**, *46*, 5423. (b). Choi, E. B.; Youn, I. K.; Pak, C. S. The Regioselective Synthesis of Tepoxalin, 3-[5-(4-Chlorophenyl)-1-(4-methoxyphenyl)-3-pyrazolyl]-*N*-hydroxy-*N*

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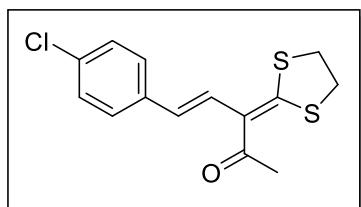
[2] Zhang, G.-T.; Lin, Y.-L.; Luo, X.; Hu, X.; Chen, C.; Lei, A. Oxidative [4+2] annulation of styrenes with alkynes under external-oxidant-free conditions. *Nat. Commun.* **2018**, 9, 1225.

[3] Tian, W.-F.; Wang, D.-P.; Wang, S.-F.; He, K.-H.; Cao, X.-P.; Li, Y. Visible-Light Photoredox-Catalyzed Iminyl Radical Formation by N–H Cleavage with Hydrogen Release and Its Application in Synthesis of Isoquinolines. *Org. Lett.*, **2018**, 20, 1421.

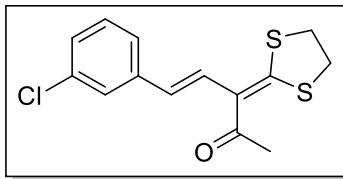
5. Analytical Data of Compounds 3a-3u, 4a-4p, 5a and 5j



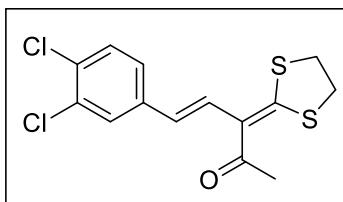
(E)-3-(1,3-Dithiolan-2-ylidene)-5-phenylpent-4-en-2-one (3a): yellow oil; $R_f = 0.36$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/8 to 1/3; 114 mg (87%); $^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.46 (d, $J = 7.2$ Hz, 2H), 7.36 (t, $J = 7.2$ Hz, 2H), 7.28 (t, $J = 7.2$ Hz, 1H), 6.98 (d, $J = 16.2$ Hz, 1H), 6.68 (d, $J = 16.2$ Hz, 1H), 3.42 (t, $J = 7.2$ Hz, 2H), 3.28 (t, $J = 6.6$ Hz, 2H), 2.33 (s, 3H). $^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 193.8, 162.7, 136.8, 134.1, 128.6 (2C), 127.9, 126.3, 126.3 (2C), 124.0, 39.2, 35.5, 28.4. **IR** (KBr): 2923, 1712, 1646, 1498, 1464, 1354, 962 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{14}\text{H}_{14}\text{NaOS}_2$ [$\text{M}+\text{Na}$] $^+$ 285.0378, found 285.0371



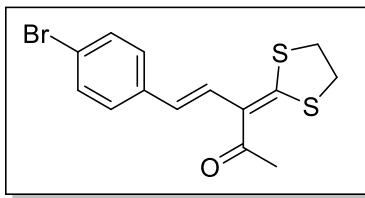
(E)-5-(4-Chlorophenyl)-3-(1,3-dithiolan-2-ylidene)pent-4-en-2-one (3b): yellow solid; $R_f = 0.34$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/8 to 1/3; 124 mg (84%); mp 84–85 °C. $^1\text{H NMR}$ (600 MHz, CDCl_3) δ 7.38 (d, $J = 9.6$ Hz, 2H), 7.31 (d, $J = 8.4$ Hz, 2H), 6.96 (d, $J = 16.2$ Hz, 1H), 6.63 (d, $J = 16.2$ Hz, 1H), 3.42 (t, $J = 6.0$ Hz, 2H), 3.29 (t, $J = 6.0$ Hz, 2H), 2.32 (s, 3H). $^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 193.6, 163.1, 135.3, 133.4, 132.6, 128.8 (2C), 127.4 (2C), 126.9, 123.7, 39.2, 35.5, 28.4. **IR** (KBr): 2982, 2925, 1715, 1630, 1490, 1464, 1420, 1355, 961, 720 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{14}\text{H}_{13}\text{ClNaOS}_2$ [$\text{M}+\text{Na}$] $^+$ 318.9989, found 318.9999.



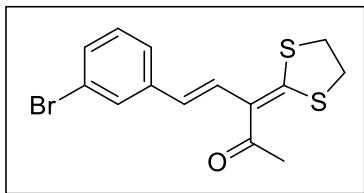
(E)-5-(3-Chlorophenyl)-3-(1,3-dithiolan-2-ylidene)pent-4-en-2-one (3c): yellow solid; $R_f = 0.37$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/8 to 1/3; 105 mg (71%); mp 88–89 °C. **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.44 (s, 1H), 7.31 (d, $J = 7.2$ Hz, 1H), 7.28 (t, $J = 7.8$ Hz, 1H), 7.24 (d, $J = 8.4$ Hz, 1H), 6.99 (d, $J = 16.2$ Hz, 1H), 6.62 (d, $J = 16.2$ Hz, 1H), 3.42 (t, $J = 6.0$ Hz, 2H), 3.30 (dd, $J = 7.8, 6.0$ Hz, 2H), 2.32 (s, 3H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 193.5, 163.4, 138.7, 134.6, 132.4, 129.8, 127.7, 127.7, 126.0, 124.5, 123.5, 39.2, 35.5, 28.4. **IR (KBr):** 2982, 2924, 1632, 1588, 1557, 1467, 1418, 1354, 990, 780 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{14}\text{H}_{13}\text{ClNaOS}_2$ $[\text{M}+\text{Na}]^+$ 318.9989, found 318.9996.



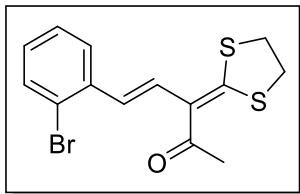
(E)-5-(3,4-Dichlorophenyl)-3-(1,3-dithiolan-2-ylidene)pent-4-en-2-one (3d): yellow oil; $R_f = 0.39$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/10 to 1/5; 117 mg (71%); **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.53 (d, $J = 2.4$ Hz, 1H), 7.42 (d, $J = 7.8$ Hz, 1H), 7.27 (dd, $J = 8.4, 2.4$ Hz, 1H), 6.98 (d, $J = 16.2$ Hz, 1H), 6.59 (d, $J = 16.2$ Hz, 1H), 3.44 (dd, $J = 7.8, 6.0$ Hz, 2H), 3.32 (dd, $J = 7.8, 6.0$ Hz, 2H), 2.33 (s, 3H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 193.5, 163.8, 137.0, 132.9, 131.5, 131.4, 130.6, 128.2, 127.9, 125.4, 123.4, 39.2, 35.6, 28.5. **IR (KBr):** 2965, 2925, 1628, 1551, 1457, 1421, 1378, 989, 786, 756 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{14}\text{H}_{13}\text{Cl}_2\text{OS}_2$ $[\text{M}+\text{H}]^+$ 330.9779, found 330.9768.



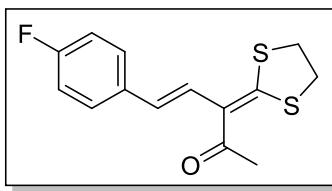
(E)-5-(4-Bromophenyl)-3-(1,3-dithiolan-2-ylidene)pent-4-en-2-one (3e): yellow solid; $R_f = 0.35$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/8 to 1/3; 143 mg (84%); mp 59–60 °C. **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.47 (d, $J = 7.8$ Hz, 2H), 7.32 (d, $J = 7.8$ Hz, 2H), 6.97 (d, $J = 16.2$ Hz, 1H), 6.61 (d, $J = 16.2$ Hz, 1H), 3.42 (t, $J = 6.6$ Hz, 2H), 3.29 (t, $J = 6.6$ Hz, 2H), 2.32 (s, 3H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 193.5, 163.1, 135.8, 132.6, 131.7 (2C), 127.7 (2C), 127.0, 123.7, 121.6, 39.2, 35.5, 28.4. **IR (KBr):** 2982, 2925, 1630, 1465, 1418, 1397, 961, 787 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{14}\text{H}_{13}\text{BrNaOS}_2$ $[\text{M}+\text{Na}]^+$ 362.9483, found 362.9462.



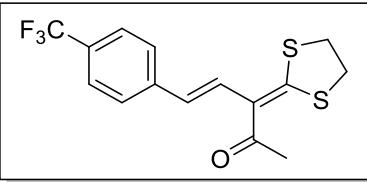
(E)-5-(3-Bromophenyl)-3-(1,3-dithiolan-2-ylidene)pent-4-en-2-one (3f): yellow solid; $R_f = 0.36$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/8 to 1/3; 134 mg (80%); mp 82–83 °C. **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.60 (s, 1H), 7.38 (d, $J = 7.8$ Hz, 1H), 7.36 (d, $J = 7.8$ Hz, 1H), 7.21 (t, $J = 7.8$ Hz, 1H), 6.98 (d, $J = 16.2$ Hz, 1H), 6.60 (d, $J = 16.2$ Hz, 1H), 3.41 (dd, $J = 7.8, 6.0$ Hz, 2H), 3.29 (dd, $J = 7.8, 6.0$ Hz, 2H), 2.32 (s, 3H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 193.4, 163.5, 139.0, 132.2, 130.5, 130.1, 128.9, 127.7, 124.8, 123.4, 122.8, 39.1, 35.5, 28.4. **IR (KBr):** 2983, 2849, 1633, 1589, 1560, 1467, 1418, 1355, 991, 781 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{14}\text{H}_{13}\text{BrNaOS}_2$ [M+Na]⁺ 362.9483, found 362.9484.



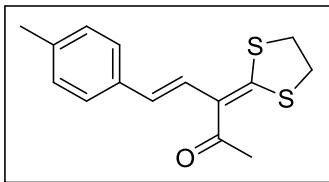
(E)-5-(2-Bromophenyl)-3-(1,3-dithiolan-2-ylidene)pent-4-en-2-one (3g): yellow solid; $R_f = 0.36$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/8 to 1/3; 143 mg (84%); mp 74–75 °C. **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.63–7.62 (m, 1H), 7.58 (d, $J = 7.8$ Hz, 1H), 7.32 (t, $J = 7.8$ Hz, 1H), 7.15–7.12 (m, 1H), 7.07 (d, $J = 16.2$ Hz, 1H), 6.93 (d, $J = 16.2$ Hz, 1H), 3.43 (t, $J = 6.6$ Hz, 2H), 3.32 (t, $J = 6.6$ Hz, 2H), 2.37 (s, 3H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 193.7, 163.6, 136.9, 133.0, 132.6, 128.9, 128.9, 127.5, 126.6, 123.9, 123.7, 39.1, 35.6, 28.6. **IR (KBr):** 2923, 2853, 1638, 1606, 1449, 1353, 991, 762 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{14}\text{H}_{13}\text{BrNaOS}_2$ [M+Na]⁺ 362.9483, found 362.9462.



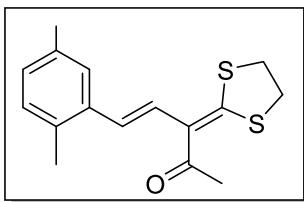
(E)-3-(1,3-Dithiolan-2-ylidene)-5-(4-fluorophenyl)pent-4-en-2-one (3h): yellow solid; $R_f = 0.31\text{Q}$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/6 to 1/2; 109 mg (78%); mp 116–117 °C. **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.43 (dd, $J = 8.4, 5.4$ Hz, 2H), 7.05 (t, $J = 8.4$ Hz, 2H), 6.89 (d, $J = 16.2$ Hz, 1H), 6.64 (d, $J = 16.2$ Hz, 1H), 3.42 (t, $J = 6.6$ Hz, 2H), 3.29 (t, $J = 7.2$ Hz, 2H), 2.32 (s, 3H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 193.7, 162.7, 162.4 ($J = 247.6$ Hz), 133.0 (d, $J = 3.3$ Hz), 132.9, 127.8 (d, $J = 8.0$ Hz, 2C), 126.11 (d, $J = 2.3$ Hz), 123.8, 115.6 (d, $J = 21.7$ Hz, 2C), 39.2, 35.4, 28.4. **IR (KBr):** 3035, 2925, 1643, 1623, 1597, 1506, 1466, 1425, 1356, 1157, 969, 771 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{14}\text{H}_{13}\text{FNaOS}_2$ [M+Na]⁺ 303.0284, found 303.0285.



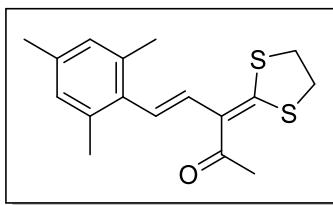
(E)-3-(1,3-Dithiolan-2-ylidene)-5-(4-(trifluoromethyl)phenyl)pent-4-en-2-one (3i): yellow oil; $R_f = 0.30$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/6 to 1/3; 144 mg (87%); **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.61 (d, $J = 8.4$ Hz, 2H), 7.55 (d, $J = 7.8$ Hz, 2H), 7.09 (d, $J = 16.2$ Hz, 1H), 6.72 (d, $J = 16.2$ Hz, 1H), 3.44 (t, $J = 7.2$ Hz, 2H), 3.32 (t, $J = 7.2$ Hz, 2H), 2.34 (s, 3H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 193.5, 163.9, 140.4, 132.3, 129.5 ($J = 22.7$ Hz), 128.8, 126.4 (2C), 125.6 (2C, $J = 3.8$ Hz), 124.1 (CF_3 , $J = 271.8$ Hz), 123.6, 39.2, 35.6, 28.4. **IR (KBr)**: 2993, 2925, 1632, 1609, 1507, 1450, 1354, 1167, 1125, 1109, 991, 825 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{15}\text{H}_{14}\text{F}_3\text{OS}_2$ [$\text{M}+\text{H}]^+$ 331.0433, found 331.0426.



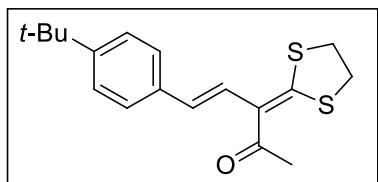
(E)-3-(1,3-Dithiolan-2-ylidene)-5-(*p*-tolyl)pent-4-en-2-one (3j): yellow solid; $R_f = 0.33$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/6 to 1/2; 109 mg (79%); mp 96–97 °C. **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.36 (d, $J = 8.4$ Hz, 2H), 7.17 (d, $J = 7.8$ Hz, 2H), 6.93 (d, $J = 16.2$ Hz, 1H), 6.65 (d, $J = 16.2$ Hz, 1H), 3.42 (dd, $J = 7.8, 6.0$ Hz, 2H), 3.27 (t, $J = 6.0$ Hz 2H), 2.36 (s, 3H), 2.32 (s, 3H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 193.9, 162.3, 137.8, 134.1, 134.0, 129.4 (2C), 126.2 (2C), 125.4, 124.1, 39.2, 35.4, 28.4, 21.2. **IR (KBr)**: 3014, 2982, 2921, 1630, 1450, 1467, 1419, 1355, 987, 821, 787 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{15}\text{H}_{16}\text{NaOS}_2$ [$\text{M}+\text{Na}]^+$ 299.0535, found 299.0533.



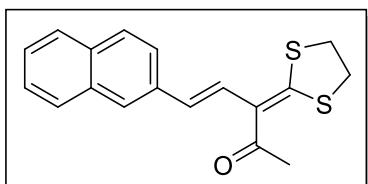
(E)-5-(2,5-Dimethylphenyl)-3-(1,3-dithiolan-2-ylidene)pent-4-en-2-one (3k): yellow solid; $R_f = 0.28$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/5 to 1/2; 115 mg (79%); mp 119–120 °C. **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.36 (s, 1H), 7.06 (d, $J = 7.8$ Hz, 1H), 7.01 (d, $J = 7.2$ Hz, 1H), 6.92 (d, $J = 15.6$ Hz, 1H), 6.83 (d, $J = 15.6$ Hz, 1H), 3.42 (t, $J = 6.6$ Hz, 2H), 3.28 (t, $J = 6.6$ Hz, 2H), 2.35 (s, 3H), 2.34 (s, 3H), 2.33 (s, 3H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 193.8, 162.4, 135.8, 135.5, 132.7, 132.6, 130.3, 128.6, 127.4, 125.8, 124.4, 39.2, 35.4, 28.4, 21.0, 19.3. **IR (KBr)**: 3039, 3010, 2917, 1639, 1619, 1558, 1541, 1493, 1447, 1352, 966, 845, 805 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{16}\text{H}_{18}\text{NaOS}_2$ [$\text{M}+\text{Na}]^+$ 313.0691, found 313.0687.



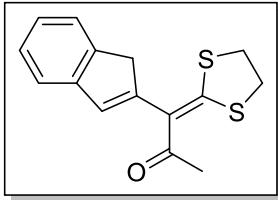
(E)-3-(1,3-Dithiolan-2-ylidene)-5-mesitylpent-4-en-2-one (3l): yellow solid; $R_f = 0.26$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/5 to 1/2; 138 mg (91%); mp 130–131 °C. **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 6.91 (s, 2H), 6.69 (d, $J = 16.8$ Hz, 1H), 6.47 (d, $J = 16.8$ Hz, 1H), 3.43 (t, $J = 6.0$ Hz, 2H), 3.30 (t, $J = 6.0$ Hz, 2H), 2.37 (s, 6H), 2.34 (s, 3H), 2.29 (s, 3H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 193.8, 162.4, 136.6, 136.1, 133.7, 133.2 (2C), 131.7, 128.8 (2C), 124.4, 39.3, 35.5, 28.7, 21.3 (2C), 20.9. **IR (KBr)**: 2947, 2916, 2855, 1638, 1566, 1449, 1375, 1349, 981, 845 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{17}\text{H}_{20}\text{NaOS}_2$ [M+Na]⁺ 327.0848, found 327.0850.



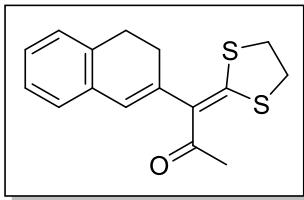
(E)-5-(4-(tert-Butyl)phenyl)-3-(1,3-dithiolan-2-ylidene)pent-4-en-2-one (3n): yellow solid; $R_f = 0.34$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/8 to 1/5; 107 mg (67%); mp 79–80 °C. **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.40 (q, $J = 8.4$ Hz, 4H), 6.95 (d, $J = 16.2$ Hz, 1H), 6.66 (d, $J = 16.2$ Hz, 1H), 3.41 (dd, $J = 7.2, 6.0$ Hz, 2H), 3.27 (dd, $J = 7.2, 5.4$ Hz, 2H), 2.31 (s, 3H), 1.33 (s, 9H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 193.9, 162.4, 151.1, 134.1, 134.0, 126.1 (2C), 125.6, 125.6 (2C), 124.1, 39.2, 35.4, 34.6, 31.2 (2C), 28.4. **IR (KBr)**: 3026, 2957, 2865, 1640, 1619, 1512, 1449, 1355, 1304, 972, 824 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{18}\text{H}_{22}\text{NaOS}_2$ [M+Na]⁺ 341.1004, found 341.1018.



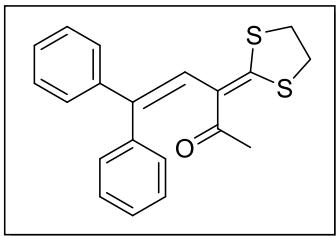
(E)-3-(1,3-Dithiolan-2-ylidene)-5-(naphthalen-2-yl)pent-4-en-2-one (3o): yellow solid; $R_f = 0.31$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/6 to 1/3; 139 mg (89%); mp 55–56 °C. **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.83–7.80 (m, 3H), 7.79 (s, 1H), 7.69 (d, $J = 8.4$ Hz, 1H), 7.50–7.42 (m, 2H), 7.11 (d, $J = 16.2$ Hz, 1H), 6.84 (d, $J = 16.2$ Hz, 1H), 3.44 (t, $J = 6.0$ Hz, 2H), 3.30 (t, $J = 6.0$ Hz, 2H), 2.37 (s, 3H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 193.9, 162.7, 134.3, 134.2, 133.6, 133.1, 128.3, 128.0, 127.7, 126.7, 126.5, 126.4, 126.0, 124.1, 123.2, 39.3, 35.5, 28.5. **IR (KBr)**: 2926, 1638, 1613, 1500, 1444, 1363, 1232, 973, 862 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{18}\text{H}_{16}\text{NaOS}_2$ [M+Na]⁺ 335.0535, found 335.0540.



1-(1,3-Dithiolan-2-ylidene)-1-(1H-inden-2-yl)propan-2-one (3q): yellow solid; $R_f = 0.24$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/4 to 1/1; 96 mg (70%); mp 114–115 °C. **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.47 (d, $J = 7.2$ Hz, 1H), 7.41 (d, $J = 7.2$ Hz, 1H), 7.30 (t, $J = 7.8$ Hz, 1H), 7.22 (t, $J = 7.2$ Hz, 1H), 6.83 (s, 1H), 3.62 (s, 2H), 3.44 (t, $J = 6.0$ Hz, 2H), 3.22 (t, $J = 6.0$ Hz, 2H), 2.14 (s, 3H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 193.0, 164.1, 146.4, 144.3, 143.4, 133.3, 126.5, 125.0, 123.6, 122.6, 121.3, 40.6, 39.5, 35.4, 28.0. **IR (KBr):** 3037, 2922, 1639, 1567, 1462, 1447, 1377, 1356, 982, 842 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{15}\text{H}_{14}\text{NaOS}_2$ $[\text{M}+\text{Na}]^+$ 397.0378, found 397.0377.



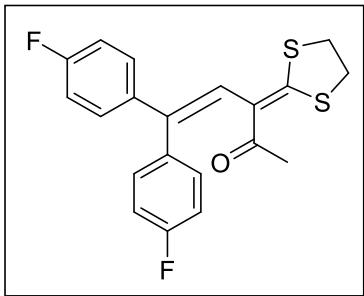
1-(3,4-Dihydronaphthalen-2-yl)-1-(1,3-dithiolan-2-ylidene)propan-2-one (3r): yellow oil; $R_f = 0.29$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/6 to 1/2; 45 mg (31%); **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.19–7.16 (m, 3H), 7.09–7.05 (m, 1H), 6.46 (d, $J = 1.8$ Hz, 1H), 3.44 (t, $J = 6.6$ Hz, 2H), 3.23 (t, $J = 6.0$ Hz, 2H), 2.98 (t, $J = 8.4$ Hz, 2H), 2.51 (t, $J = 7.8$ Hz, 2H), 2.22 (d, $J = 1.2$ Hz, 3H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 193.0, 163.2, 139.8, 134.7, 133.9, 129.5, 127.5, 127.5, 127.4, 126.6, 126.4, 39.6, 35.3, 28.6, 27.6, 27.3. **IR (KBr):** 3058, 3015, 2926, 2882, 2828, 1714, 1639, 1571, 1473, 1356, 985, 849, 829 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{16}\text{H}_{16}\text{NaOS}_2$ $[\text{M}+\text{Na}]^+$ 311.0535, found 311.0514.



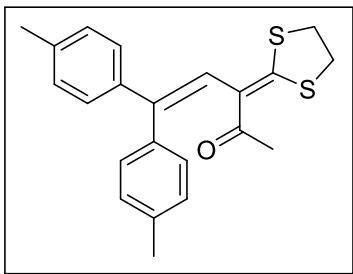
3-(1,3-Dithiolan-2-ylidene)-5,5-diphenylpent-4-en-2-one (3s): yellow oil; $R_f = 0.33$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/6 to 1/3; 144 mg (85%); **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.35–7.33 (m, 5H), 7.27–7.23 (m, 3H), 7.18–7.13 (m, 2H), 6.64 (s, 1H), 3.37 (t, $J = 6.6$ Hz, 2H), 3.26 (t, $J = 6.6$ Hz, 2H), 1.92 (s, 3H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 193.5, 162.8, 145.8, 142.5, 139.9, 129.8 (2C), 128.4 (2C), 128.2 (2C), 128.2, 128.2, 127.9, 127.9, 125.6,

123.9, 39.1, 35.5, 27.6. **IR** (KBr): 3054, 3027, 2920, 1642, 1597, 1491, 1459, 1442, 1423, 1356, 754 cm^{-1}

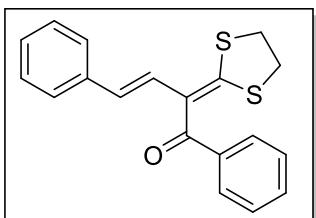
¹. **HRMS (ESI)** m/z calcd for $\text{C}_{20}\text{H}_{19}\text{OS}_2$ [$\text{M}+\text{H}]^+$ 339.0872, found 339.0872.



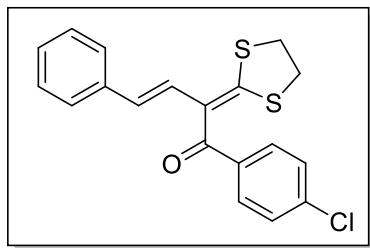
3-(1,3-Dithiolan-2-ylidene)-5,5-bis(4-fluorophenyl)pent-4-en-2-one (3t): yellow oil; $R_f = 0.27$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/5 to 1/2; 138mg (74%); **¹H NMR** (600 MHz, CDCl_3) δ 7.30 (dd, $J = 8.4, 5.4$ Hz, 2H), 7.12 (dd, $J = 8.4, 5.4$ Hz, 2H), 7.03 (t, $J = 8.4$ Hz, 2H), 6.96 (t, $J = 8.4$ Hz, 2H), 6.59 (s, 1H), 3.38 (t, $J = 6.6$ Hz, 2H), 3.27 (dd, $J = 7.2, 5.4$ Hz, 2H), 1.95 (s, 3H). **¹³C NMR** (151 MHz, CDCl_3) δ 193.2, 163.3 (d, $J = 65.8$ Hz), 163.3, 163.1 (d, $J = 66.4$ Hz), 143.7, 138.4 (d, $J = 3.3$ Hz), 135.3 (d, $J = 3.3$ Hz), 131.4 (d, $J = 8.2$ Hz, 2C), 129.9 (d, $J = 8.0$ Hz, 2C), 125.6, 123.4, 115.3 (d, $J = 21.4$ Hz, 2C), 115.1 (d, $J = 21.3$ Hz, 2C), 39.1, 35.5, 27.6. **IR** (KBr): 3056, 3026, 2927, 2854, 2205, 1664, 1597, 1537, 1514, 1492, 1446, 1356, 1152, 1090, 750 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{20}\text{H}_{17}\text{F}_2\text{OS}_2$ [$\text{M}+\text{H}]^+$ 375.0683, found 375.0686.



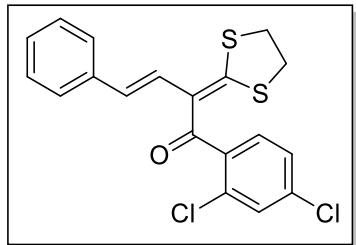
3-(1,3-Dithiolan-2-ylidene)-5,5-di-p-tolylpent-4-en-2-one (3u): yellow oil; $R_f = 0.36$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/8 to 1/4; 134 mg (73%); **¹H NMR** (600 MHz, CDCl_3) δ 7.25 (d, $J = 9.6$ Hz, 2H), 7.15 (d, $J = 9.6$ Hz, 2H), 7.04 (q, $J = 9.6$ Hz, 4H), 6.56 (s, 1H), 3.38 (dd, $J = 9.6, 7.2$ Hz, 2H), 3.27 (dd, $J = 8.4, 6.0$ Hz, 2H), 2.37 (s, 3H), 2.31 (s, 3H), 1.91 (s, 3H). **¹³C NMR** (151 MHz, CDCl_3) δ 193.9, 162.3, 145.6, 139.9, 137.7, 137.6, 137.1, 129.7 (2C), 128.9 (2C), 128.8 (2C), 128.3 (2C), 124.5, 124.3, 39.1, 35.6, 27.6, 21.2, 21.2. **IR** (KBr): 3021, 2978, 2925, 1636, 1607, 1509, 1452, 1348, 726 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{22}\text{H}_{22}\text{NaOS}_2$ [$\text{M}+\text{Na}]^+$ 389.1004, found 389.1023.



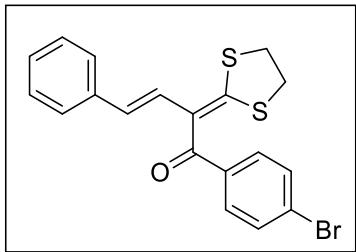
(E)-2-(1,3-Dithiolan-2-ylidene)-1,4-diphenylbut-3-en-1-one (4a): yellow solid; $R_f = 0.42$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/10 to 1/5; 144 mg (89%); mp 79–80 °C. **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.82 (d, $J = 8.4$ Hz, 2H), 7.47 (t, $J = 7.8$ Hz, 1H), 7.39 (t, $J = 7.8$ Hz, 2H), 7.30–7.23 (m, 4H), 7.21–7.15 (m, 1H), 7.02 (d, $J = 16.2$ Hz, 1H), 6.26 (d, $J = 16.2$ Hz, 1H), 3.42–3.40 (m, 2H), 3.38–3.36 (m, 2H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 193.8, 154.6, 137.7, 137.1, 132.3, 131.0, 129.6 (2C), 128.5 (2C), 128.2 (2C), 127.4, 126.5, 126.1 (2C), 124.9, 38.5, 36.8. **IR** (KBr): 3063, 3020, 2968, 2926, 1623, 1592, 1574, 1464, 954, 760 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{19}\text{H}_{16}\text{NaOS}_2$ [$\text{M}+\text{Na}]^+$ 347.0535, found 347.0527.



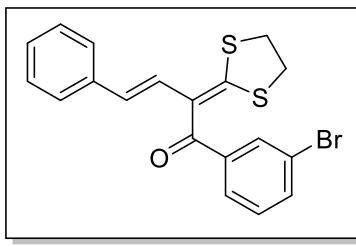
(E)-1-(4-Chlorophenyl)-2-(1,3-dithiolan-2-ylidene)-4-phenylbut-3-en-1-one (4b): yellow solid; $R_f = 0.39$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/9 to 1/5; 159 mg (89%); mp 81–82 °C. **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.76 (d, $J = 8.4$ Hz, 2H), 7.35 (d, $J = 8.4$ Hz, 2H), 7.28 (m, 3H), 7.21–7.18 (m, 1H), 7.00 (d, $J = 16.2$ Hz, 1H), 6.25 (d, $J = 16.2$ Hz, 1H), 3.42 (d, $J = 5.4$ Hz, 2H), 3.40 (d, $J = 5.4$ Hz, 2H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 192.4, 156.1, 138.6, 137.1, 136.4, 131.6, 131.2 (2C), 128.7 (2C), 128.6 (2C), 127.7, 126.4, 126.3 (2C), 124.5, 38.7, 36.9. **IR** (KBr): 3058, 3025, 2925, 2854, 1663, 1587, 1529, 1483, 1452, 980, 750 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{19}\text{H}_{16}\text{ClOS}_2$ [$\text{M}+\text{H}]^+$ 359.0326, found 359.0321.



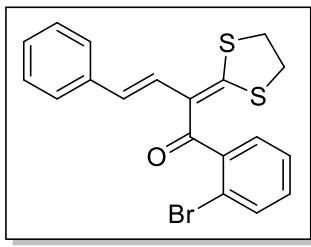
(E)-1-(2,4-Dichlorophenyl)-2-(1,3-dithiolan-2-ylidene)-4-phenylbut-3-en-1-one (4c): yellow solid; $R_f = 0.34$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/6 to 1/2; 125 mg (64%); mp 97–98 °C. **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.34 (d, $J = 1.8$ Hz, 1H), 7.30 (d, $J = 8.4$ Hz, 1H), 7.28–7.24 (m, 3H), 7.21 (d, $J = 7.2$ Hz, 3H), 6.79 (d, $J = 16.2$ Hz, 1H), 6.33 (d, $J = 16.2$ Hz, 1H), 3.49 (t, $J = 6.6$ Hz, 2H), 3.40 (dd, $J = 6.6, 4.8$ Hz, 2H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 188.7, 166.5, 138.4, 136.9, 135.7, 133.2, 131.7, 123.0, 129.6, 128.5 (2C), 127.7, 127.0, 126.2 (2C), 125.5, 111.5, 39.1, 36.1. **IR** (KBr): 3058, 3022, 2981, 2926, 1628, 1551, 1457, 1421, 989, 872, 840, 822, 756 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{19}\text{H}_{14}\text{Cl}_2\text{NaOS}_2$ [$\text{M}+\text{Na}]^+$ 414.9755, found 414.9757.



(*E*)-1-(4-Bromophenyl)-2-(1,3-dithiolan-2-ylidene)-4-phenylbut-3-en-1-one (4d): yellow solid; $R_f = 0.40$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/9 to 1/5; 197 mg (98%); mp 96–97 °C. **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.68 (d, $J = 8.4$ Hz, 2H), 7.52 (d, $J = 8.4$ Hz, 2H), 7.27–7.26 (m, 4H), 7.23–7.18 (m, 1H), 6.99 (d, $J = 16.2$ Hz, 1H), 6.25 (d, $J = 16.2$ Hz, 1H), 3.44–3.42 (m, 2H), 3.40–3.38 (m, 2H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 192.3, 156.5, 136.9, 136.7, 131.5, 131.5 (2C), 131.1 (2C), 128.6 (2C), 127.6, 127.1, 126.3, 126.1 (2C), 124.2, 38.5, 36.8. **IR** (KBr): 3057, 3025, 2925, 1663, 1583, 1532, 1477, 1451, 954, 807, 749 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{19}\text{H}_{15}\text{BrNaOS}_2$ [M+Na]⁺ 424.9640, found 424.9629.

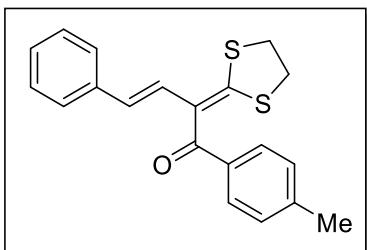


(*E*)-1-(3-Bromophenyl)-2-(1,3-dithiolan-2-ylidene)-4-phenylbut-3-en-1-one (4e): yellow oil; $R_f = 0.39$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/9 to 1/5; 197 mg (98%); **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.95 (s, 1H), 7.69 (d, $J = 7.8$ Hz, 1H), 7.58 (d, $J = 8.4$ Hz, 1H), 7.28 (d, $J = 6.0$ Hz, 4H), 7.25–7.23 (m, 1H), 7.22–7.19 (m, 1H), 6.98 (d, $J = 16.2$ Hz, 1H), 6.26 (d, $J = 16.2$ Hz, 1H), 3.45–3.39 (m, 4H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 191.7, 157.4, 139.9, 136.9, 134.9, 132.3, 131.8, 129.7, 128.6 (2C), 128.2, 127.6, 126.2, 126.2 (2C), 124.0, 122.4, 38.6, 36.8. **IR** (KBr): 3057, 3022, 2923, 2854, 1663, 1609, 1561, 1453, 994, 745, 693 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{19}\text{H}_{15}\text{BrNaOS}_2$ [M+Na]⁺ 424.9640, found 424.9637.

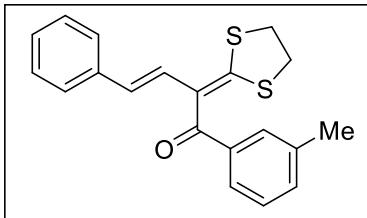


(*E*)-1-(2-Bromophenyl)-2-(1,3-dithiolan-2-ylidene)-4-phenylbut-3-en-1-one (4f): yellow oil; $R_f = 0.39$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/9 to 1/5; 183 mg (91%); **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.49 (d, $J = 7.8$ Hz, 1H), 7.29 (q, $J = 4.8$, 2H),

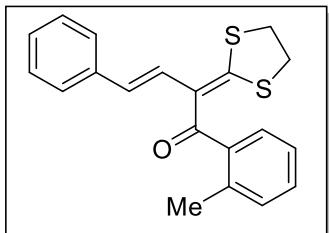
7.23–7.21 (m, 2H), 7.16–7.14 (m, 4H), 6.78 (d, J = 16.2 Hz, 1H), 6.33 (d, J = 16.2 Hz, 1H), 3.43 (dd, J = 7.8, 6.0 Hz, 2H), 3.35 (dd, J = 7.2, 5.4 Hz, 2H). ^{13}C NMR (151 MHz, CDCl_3) δ 190.4, 166.0, 141.8, 137.0, 132.8, 132.7, 130.4, 128.7, 128.4 (2C), 127.4, 127.1, 126.0 (2C), 125.7, 122.9, 119.2, 39.0, 35.9. IR (KBr): 3056, 3024, 2924, 1614, 1562, 1452, 958, 743 cm^{-1} . HRMS (ESI) m/z calcd for $\text{C}_{19}\text{H}_{15}\text{BrNaOS}_2$ [M+Na]⁺ 424.9640, found 424.9629.



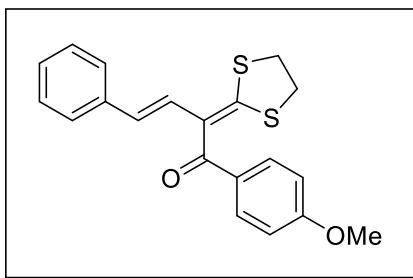
(E)-2-(1,3-Dithiolan-2-ylidene)-4-phenyl-1-(p-tolyl)but-3-en-1-one (4g): yellow solid; R_f = 0.43 (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/10 to 1/7; 139 mg (82%); mp 110–111 °C. ^1H NMR (600 MHz, CDCl_3) δ 7.77 (d, J = 8.4 Hz, 2H), 7.30 (d, J = 7.2 Hz, 2H), 7.26 (t, J = 7.2 Hz, 2H), 7.19 (dd, J = 15.6, 7.2 Hz, 3H), 7.03 (d, J = 16.2 Hz, 1H), 6.25 (d, J = 16.2 Hz, 1H), 3.43 (dd, J = 7.8, 4.8 Hz, 2H), 3.38 (dd, J = 6.6, 4.8 Hz, 2H), 2.37 (s, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 194.0, 151.9, 143.4, 137.2, 134.8, 130.5, 129.9 (2C), 129.1 (2C), 128.5 (2C), 127.4, 126.6, 126.2 (2C), 125.5, 38.5, 36.9, 21.7. IR (KBr): 3024, 2922, 2856, 1659, 1603, 1569, 1491, 1450, 1313, 952, 831, 752 cm^{-1} . HRMS (ESI) m/z calcd for $\text{C}_{20}\text{H}_{18}\text{NaOS}_2$ [M+Na]⁺ 316.0691, found 316.0705.



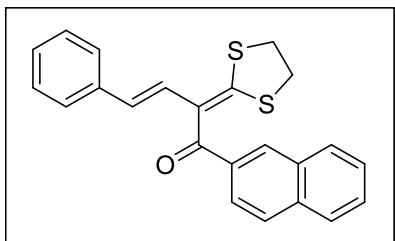
(E)-2-(1,3-Dithiolan-2-ylidene)-4-phenyl-1-(m-tolyl)but-3-en-1-one (4h): yellow solid; R_f = 0.40 (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/10 to 1/6; 142 mg (84%); mp 105–106 °C. ^1H NMR (600 MHz, CDCl_3) δ 7.67 (s, 1H), 7.61 (d, J = 7.2 Hz, 1H), 7.27 (td, J = 16.2, 7.8 Hz, 6H), 7.18 (t, J = 7.2 Hz, 1H), 7.02 (d, J = 15.6 Hz, 1H), 6.26 (d, J = 16.2 Hz, 1H), 3.46–3.40 (m, 2H), 3.39–3.37 (m, 2H), 2.36 (s, 3H). ^{13}C NMR (151 MHz, CDCl_3) δ 194.3, 153.3, 138.1, 137.7, 137.2, 133.2, 130.8, 130.0, 128.5 (2C), 128.1, 127.4, 127.0, 126.6, 126.1 (2C), 125.3, 38.5, 36.9, 21.3. IR (KBr): 3055, 3022, 2976, 2922, 1664, 1624, 1596, 1467, 1418, 991, 755, 688 cm^{-1} . HRMS (ESI) m/z calcd for $\text{C}_{20}\text{H}_{18}\text{NaOS}_2$ [M+Na]⁺ 316.0691, found 316.0689.



(*E*)-2-(1,3-Dithiolan-2-ylidene)-4-phenylbut-3-en-1-one (4i): yellow solid; $R_f = 0.41$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/10 to 1/7; 115 mg (68%); mp 59–60 °C. **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.31 (d, $J = 7.8$ Hz, 1H), 7.30–7.26 (m, 1H), 7.26–7.22 (m, 2H), 7.19–7.16 (m, 5H), 6.83 (d, $J = 16.2$ Hz, 1H), 6.33 (d, $J = 16.2$ Hz, 1H), 3.45 (dd, $J = 8.4, 4.2$ Hz, 2H), 3.41 (dd, $J = 7.2, 5.4$ Hz, 2H), 2.37 (s, 3H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 194.3, 161.5, 139.8, 137.3, 136.0, 131.9, 130.8, 129.8, 128.5 (2C), 128.2, 127.5, 126.3, 126.1 (2C), 125.4, 124.7, 38.9, 36.2, 19.8. **IR (KBr)**: 3059, 3021, 2922, 1669, 1608, 1569, 1510, 1492, 1445, 982, 748, 735 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{20}\text{H}_{18}\text{NaOS}_2$ [M+Na]⁺ 316.0691, found 316.0691.

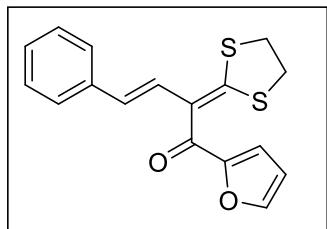


(*E*)-2-(1,3-Dithiolan-2-ylidene)-1-(4-methoxyphenyl)-4-phenylbut-3-en-1-one (4j): yellow solid; $R_f = 0.42$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/9 to 1/5; 173 mg (98%); mp 89–90 °C. **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.67 (s, 1H), 7.61 (d, $J = 7.2$ Hz, 1H), 7.31–7.25 (m, 6H), 7.19 (t, $J = 7.2$ Hz, 1H), 7.02 (d, $J = 16.2$ Hz, 1H), 6.26 (d, $J = 16.2$ Hz, 1H), 3.48–3.42 (m, 2H), 3.40 (dd, $J = 7.2, 4.8$ Hz, 2H), 2.37 (s, 3H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 194.3, 153.3, 138.1, 137.7, 137.3, 133.3, 130.8, 130.0, 128.5 (2C), 128.1, 127.4, 127.0, 126.6, 126.2 (2C), 125.3, 38.5, 36.9, 21.3. **IR (KBr)**: 3054, 3021, 2974, 2921, 1661, 1620, 1595, 1456, 1276, 1101, 958, 749 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{20}\text{H}_{18}\text{NaO}_2\text{S}_2$ [M+Na]⁺ 377.0640, found 377.0626.

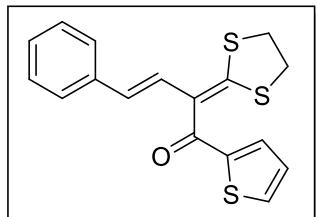


(*E*)-2-(1,3-Dithiolan-2-ylidene)-1-(naphthalen-2-yl)-4-phenylbut-3-en-1-one (4k): yellow oil; $R_f = 0.38$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/8 to 1/5; 144 mg (77%). **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 8.37 (s, 1H), 7.93 (d, $J = 8.4$ Hz, 1H), 7.90 (d, $J = 8.4$ Hz, 1H), 7.85 (d, $J = 5.6$ Hz, 1H), 7.83 (d, $J = 5.6$ Hz, 1H), 7.55 (t, $J = 7.8$ Hz, 1H), 7.49 (t, J

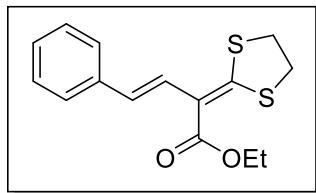
= 7.8 Hz, 1H), 7.27 (d, J = 7.8 Hz, 2H), 7.23 (t, J = 7.8 Hz, 3H), 7.16 (t, J = 7.8 Hz, 1H), 7.10 (d, J = 16.2 Hz, 1H), 6.31 (d, J = 16.2 Hz, 1H), 3.46 (t, J = 7.2 Hz, 2H), 3.39 (t, J = 6.6 Hz, 2H). **^{13}C NMR** (151 MHz, CDCl_3) δ 194.1, 153.3, 137.2, 135.3, 135.0, 132.6, 131.5, 131.0, 129.5, 128.5 (2C), 128.5, 128.2, 128.1, 127.7, 127.5, 126.6, 126.5, 126.2 (2C), 125.4, 38.5, 37.0. **IR** (KBr): 3-56, 3023, 2924, 1655, 1607, 1570, 1483, 953, 865, 822, 754 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{23}\text{H}_{18}\text{NaOS}_2$ [M+Na]⁺ 397.0691, found 397.0692.



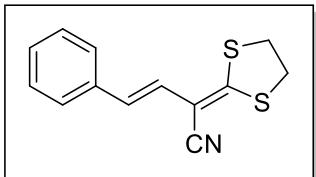
(E)-2-(1,3-Dithiolan-2-ylidene)-1-(furan-2-yl)-4-phenylbut-3-en-1-one (4l): yellow solid; R_f = 0.29 (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/6 to 1/2; 126 mg (80%); mp 80-81 °C. **^1H NMR** (600 MHz, CDCl_3) δ 7.57 (s, 1H), 7.42 (d, J = 7.8 Hz, 2H), 7.33 (t, J = 7.8 Hz, 2H), 7.25 (t, J = 7.2 Hz, 1H), 7.11 (d, J = 3.6 Hz, 1H), 7.09 (d, J = 16.8 Hz, 1H), 6.57 (d, J = 16.2 Hz, 1H), 6.44 (dd, J = 3.6, 1.8 Hz, 1H), 3.41 (dd, J = 6.6, 1.8 Hz, 2H), 3.37 (dd, J = 6.6, 1.8 Hz, 2H). **^{13}C NMR** (151 MHz, CDCl_3) δ 178.3, 159.7, 152.2, 146.3, 136.8, 132.6, 128.6 (2C), 127.7, 126.2 (2C), 126.2, 126.2, 123.0, 119.3, 111.9, 38.7, 36.2. **IR** (KBr): 3022, 2924, 1629, 1595, 1459, 1279, 1083, 968, 754 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{17}\text{H}_{14}\text{NaO}_2\text{S}_2$ [M+Na]⁺ 337.0327, found 337.0327.



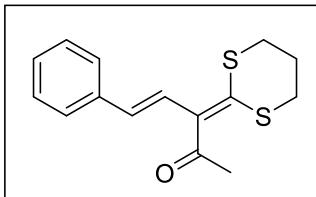
(E)-2-(1,3-Dithiolan-2-ylidene)-4-phenyl-1-(thiophen-2-yl)but-3-en-1-one (4m): yellow oil; R_f = 0.30 (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/6 to 1/2; 134 mg (81%). **^1H NMR** (600 MHz, Chloroform-*d*) δ 7.71 (d, J = 3.6 Hz, 1H), 7.59–7.55 (m, 1H), 7.40 (d, J = 7.2 Hz, 2H), 7.31 (t, J = 7.8 Hz, 2H), 7.23 (t, J = 7.2 Hz, 1H), 7.08 (d, J = 16.2 Hz, 1H), 7.04–7.00 (t, J = 4.8 Hz, 1H), 6.55 (d, J = 16.2 Hz, 1H), 3.38 (d, J = 3.0 Hz, 4H). **^{13}C NMR** (151 MHz, CDCl_3) δ 183.9, 156.8, 143.8, 136.80, 133.97, 133.60, 132.49, 128.6 (2C), 127.7, 127.7, 126.3, 126.3 (2C), 124.1, 38.9, 36.5. **IR** (KBr): 3079, 3023, 2926, 2250, 1636, 1591, 1495, 1449, 954, 753 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{17}\text{H}_{14}\text{NaOS}_3$ [M+Na]⁺ 353.0099, found 353.0103.



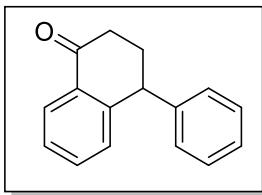
(E)-Ethyl 2-(1,3-dithiolan-2-ylidene)-4-phenylbut-3-enoate (4n): yellow oil; $R_f = 0.38$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/9 to 1/5; 105 mg (72%); **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.43 (d, $J = 7.8$ Hz, 2H), 7.32 (t, $J = 7.2$ Hz, 2H), 7.22 (t, $J = 7.2$ Hz, 1H), 6.99 (d, $J = 16.2$ Hz, 1H), 6.95 (d, $J = 16.2$ Hz, 1H), 4.30 (q, $J = 7.2$ Hz, 2H), 3.41 (t, $J = 6.0$ Hz, 2H), 3.34 (t, $J = 6.0$ Hz, 2H), 1.35 (t, $J = 7.2$ Hz, 3H). **$^{13}\text{C NMR}$** (126 MHz, CDCl_3) δ 165.9, 160.7, 137.8, 131.4, 128.6 (2C), 127.4, 126.3 (2C), 125.2, 115.9, 60.9, 38.9, 36.5, 14.4. **IR (KBr)**: 3410, 2982, 2928, 1713, 1686, 1553, 1489, 1416, 1392, 1362, 1250, 1209, 995, 769 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{15}\text{H}_{16}\text{NaO}_2\text{S}_2$ [M+Na]⁺ 315.0484, found 315.0493.



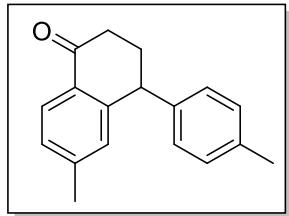
(E)-2-(1,3-Dithiolan-2-ylidene)-4-phenylbut-3-enenitrile (4o): yellow oil; $R_f = 0.24$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/5 to 1/1; 91 mg (74%); **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.41 (d, $J = 7.8$ Hz, 2H), 7.32 (t, $J = 7.8$ Hz, 2H), 7.27–7.23 (m, 1H), 6.75 (d, $J = 15.6$ Hz, 1H), 6.69 (d, $J = 16.2$ Hz, 1H), 3.61–3.58 (m, 2H), 3.57 (d, $J = 4.2$ Hz, 2H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 160.2, 136.2, 129.5, 128.7, 127.9, 126.5, 123.0, 116.4, 98.2, 39.3, 38.3. **IR (KBr)**: 3056, 3026, 2927, 2854, 2205, 1664, 1597, 1537, 1514, 1492, 1446, 982, 750 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{13}\text{H}_{11}\text{NNaOS}_2$ [M+Na]⁺ 268.0225, found 268.0229.



(E)-3-(1,3-Dithian-2-ylidene)-5-phenylpent-4-en-2-one (4p): yellow oil; $R_f = 0.21$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/5 to 1/1; 97 mg (70%); **$^1\text{H NMR}$** (600 MHz, $\text{Chloroform-}d$) δ 7.43 (d, $J = 7.2$ Hz, 2H), 7.33 (t, $J = 7.8$ Hz, 2H), 7.24 (d, $J = 7.2$ Hz, 1H), 7.08 (d, $J = 16.2$ Hz, 1H), 6.39 (d, $J = 16.2$ Hz, 1H), 3.00 (t, $J = 6.6$ Hz, 2H), 2.96 (t, $J = 6.6$ Hz, 2H), 2.41 (s, 3H), 2.21 (p, $J = 6.6$ Hz, 2H). **$^{13}\text{C NMR}$** (151 MHz, CDCl_3) δ 200.6, 142.4, 137.6, 137.0, 131.8, 128.7 (2C), 127.9, 126.5 (2C), 123.0, 30.5, 29.4, 29.3, 24.2. **IR (KBr)**: 2926, 2854, 1648, 1627, 1612, 1463, 1416, 1355, 966, 756 cm^{-1} . **HRMS (ESI)** m/z calcd for $\text{C}_{15}\text{H}_{16}\text{NaOS}_2$ [M+Na]⁺ 299.0535, found 299.0536.



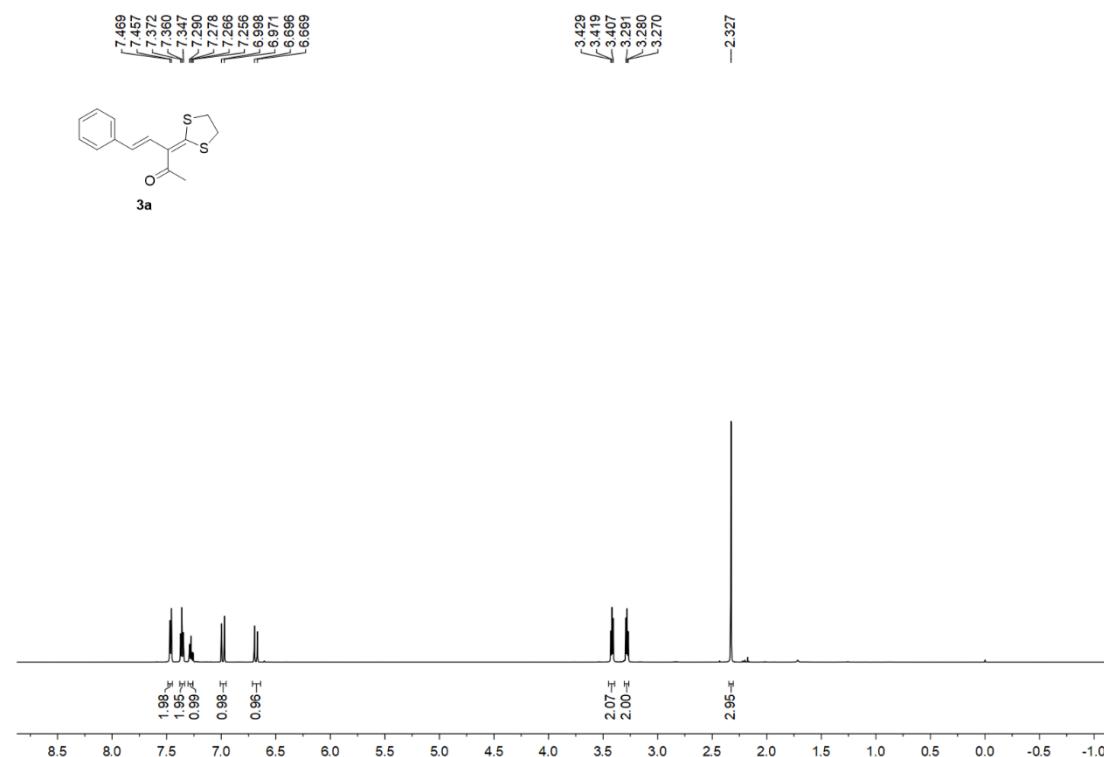
4-phenyl-3,4-dihydronaphthalen-1(2H)-one (5a): pale yellow oil; $R_f = 0.58$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/20 to 1/18; **$^1\text{H NMR}$** (600 MHz, Chloroform-*d*) δ 8.12 (d, $J = 7.8$ Hz, 1H), 7.44 (t, $J = 7.8$ Hz, 1H), 7.36 (d, $J = 7.2$ Hz, 1H), 7.33 (t, $J = 7.8$ Hz, 2H), 7.26 (d, $J = 4.2$ Hz, 1H), 7.11 (d, $J = 7.8$ Hz, 2H), 6.99 (d, $J = 7.2$ Hz, 1H), 4.31 (dd, $J = 7.8, 4.2$ Hz, 1H), 2.76–2.71 (m, 1H), 2.65–2.60 (m, 1H), 2.50–2.46 (m, 1H), 2.34–2.29 (m, 1H). **$^{13}\text{C NMR}$** (151 MHz, CDCl₃) δ 198.1, 146.3, 143.7, 133.6, 132.8, 129.5, 128.6 (2C), 128.6 (2C), 127.1, 127.0, 126.8, 45.3, 36.7, 31.8. **HRMS (ESI)** m/z calcd for C₁₆H₁₄NaO [M+Na]⁺ 245.0937, found 245.0932.



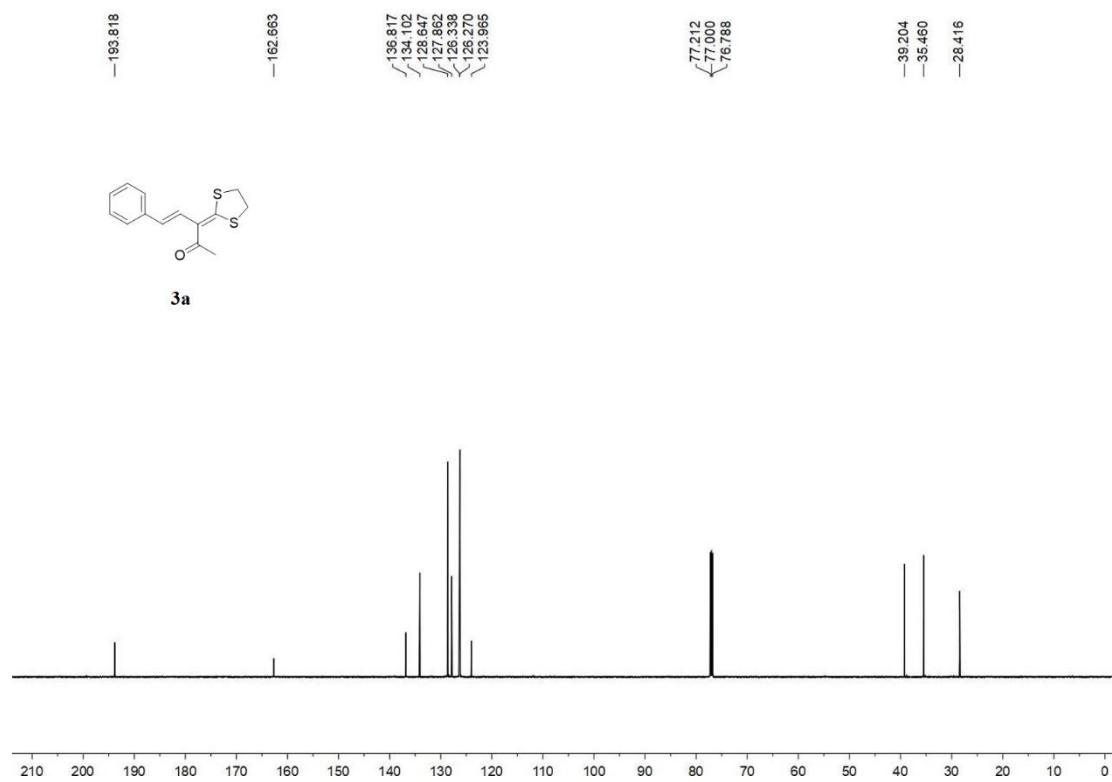
6-methyl-4-(p-tolyl)-3,4-dihydronaphthalen-1(2H)-one (5j): pale yellow oil; $R_f = 0.59$ (eluent: EtOAc/petroleum ether = 1/2); flash chromatography eluent: EtOAc/petroleum ether = 1/20 to 1/18; **$^1\text{H NMR}$** (500 MHz, Chloroform-*d*) δ 8.01 (d, $J = 9.6$ Hz, 1H), 7.17–7.11 (m, 3H), 6.99 (d, $J = 9.6$ Hz, 2H), 6.80 (s, 1H), 4.23 (dd, $J = 9.0, 4.2$ Hz, 1H), 2.72–2.66 (m, 1H), 2.60–2.54 (m, 1H), 2.49–2.41 (m, 1H), 2.35 (s, 3H), 2.29 (s, 3H), 2.33–2.21 (m, 1H). **$^{13}\text{C NMR}$** (151 MHz, CDCl₃) δ 198.0, 146.4, 144.5, 140.7, 136.3, 130.6, 129.9, 129.3 (2C), 128.4 (2C), 128.0, 127.2, 44.8, 36.4, 31.9, 21.8, 21.0. **HRMS (ESI)** m/z calcd for C₁₈H₁₈NaO [M+Na]⁺ 273.1250, found 273.1256.

6. Copies of ^1H NMR and ^{13}C NMR Spectra

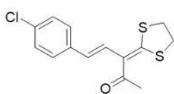
^1H spectrum (600 MHz, CDCl_3) of compound 3a



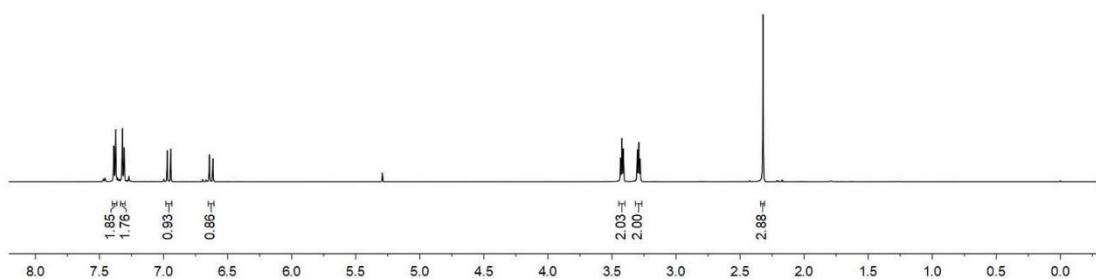
^{13}C spectrum (151 MHz, CDCl_3) of compound 3a



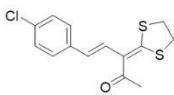
¹H spectrum (600 MHz, CDCl₃) of compound **3b**



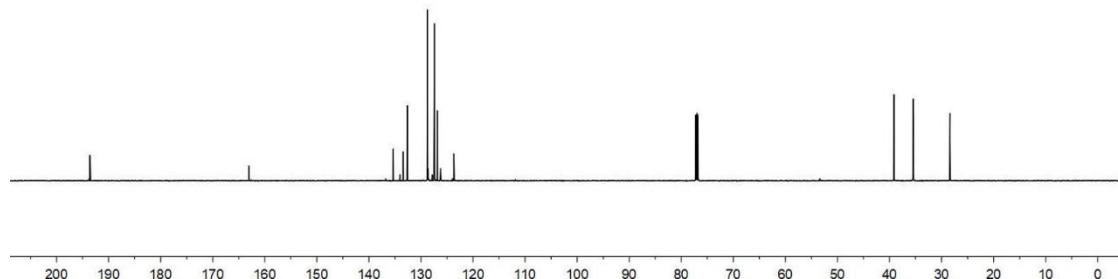
3b



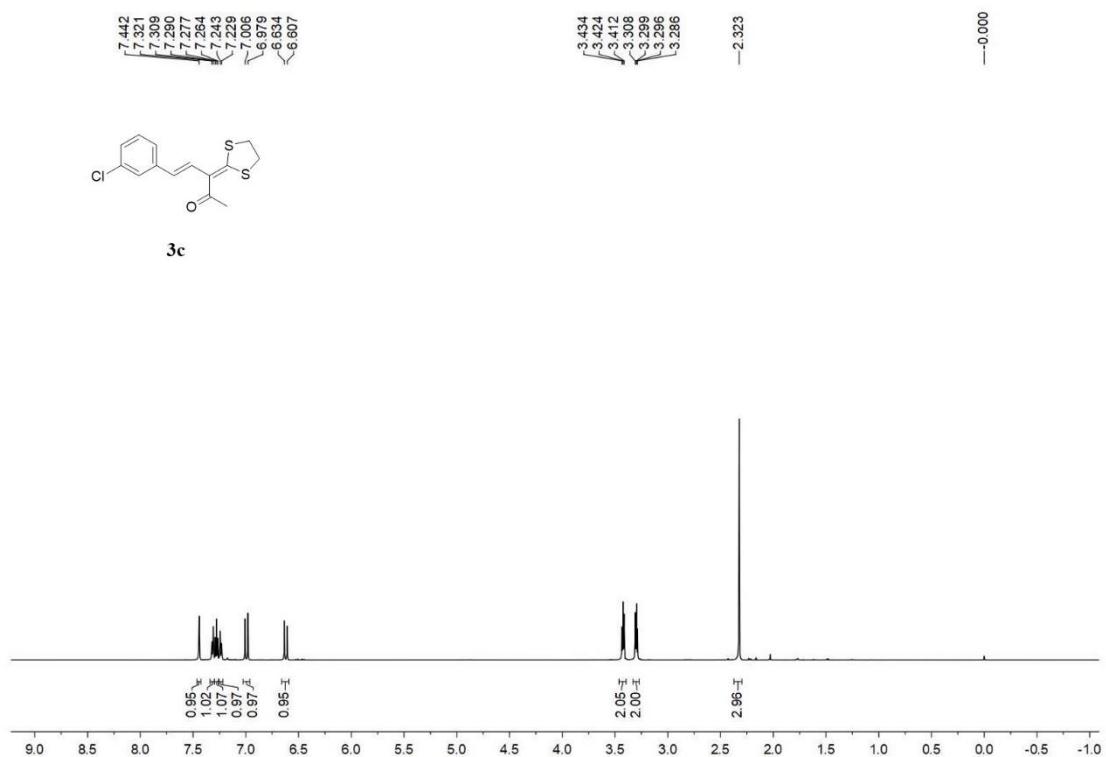
¹³C spectrum (151 MHz, CDCl₃) of compound 3b



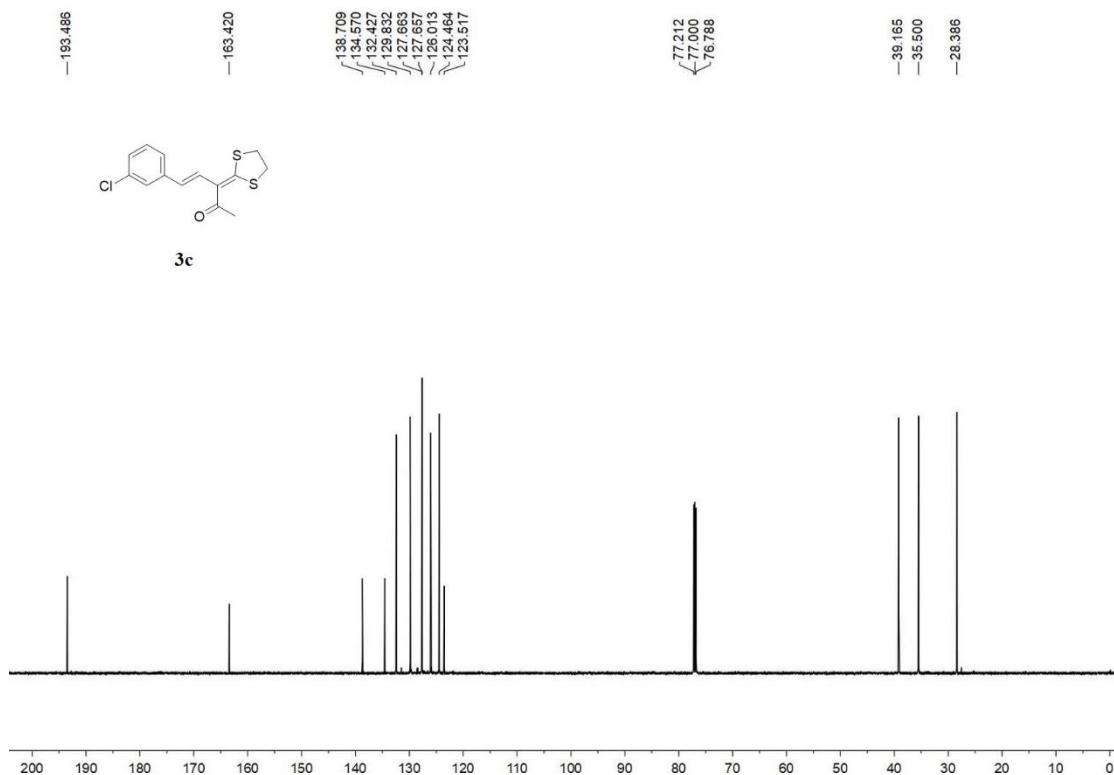
3b



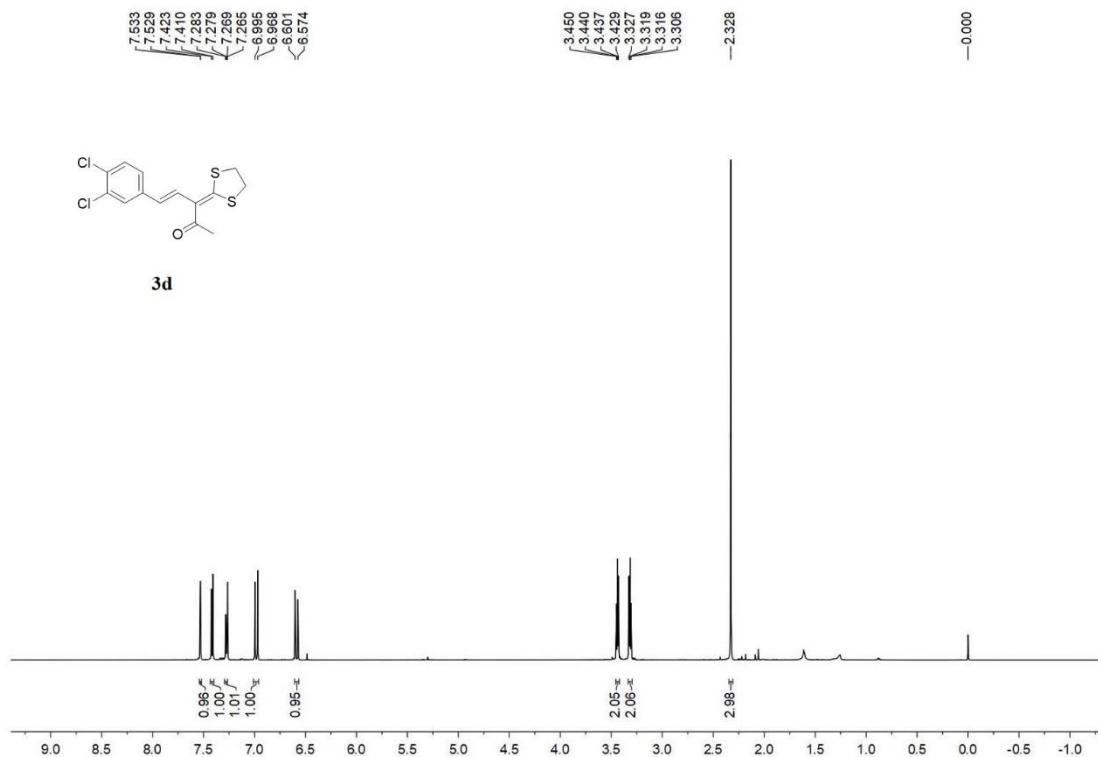
¹H spectrum (600 MHz, CDCl₃) of compound 3c



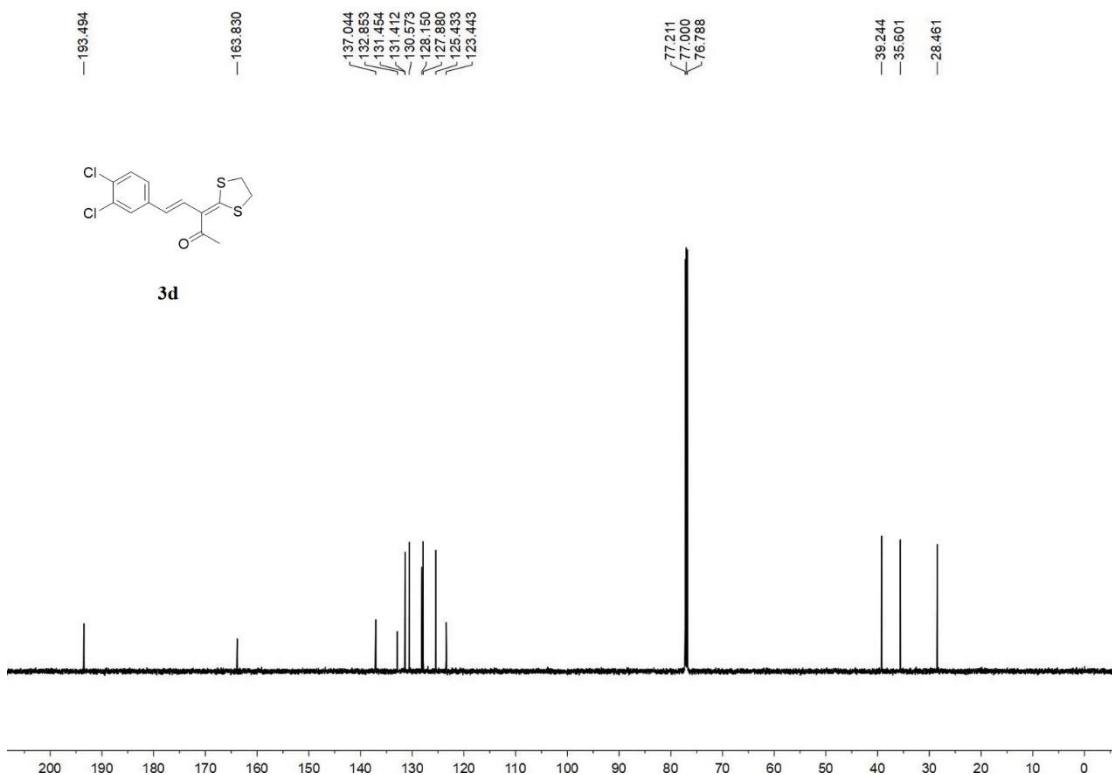
¹³C spectrum (151 MHz, CDCl₃) of compound 3c



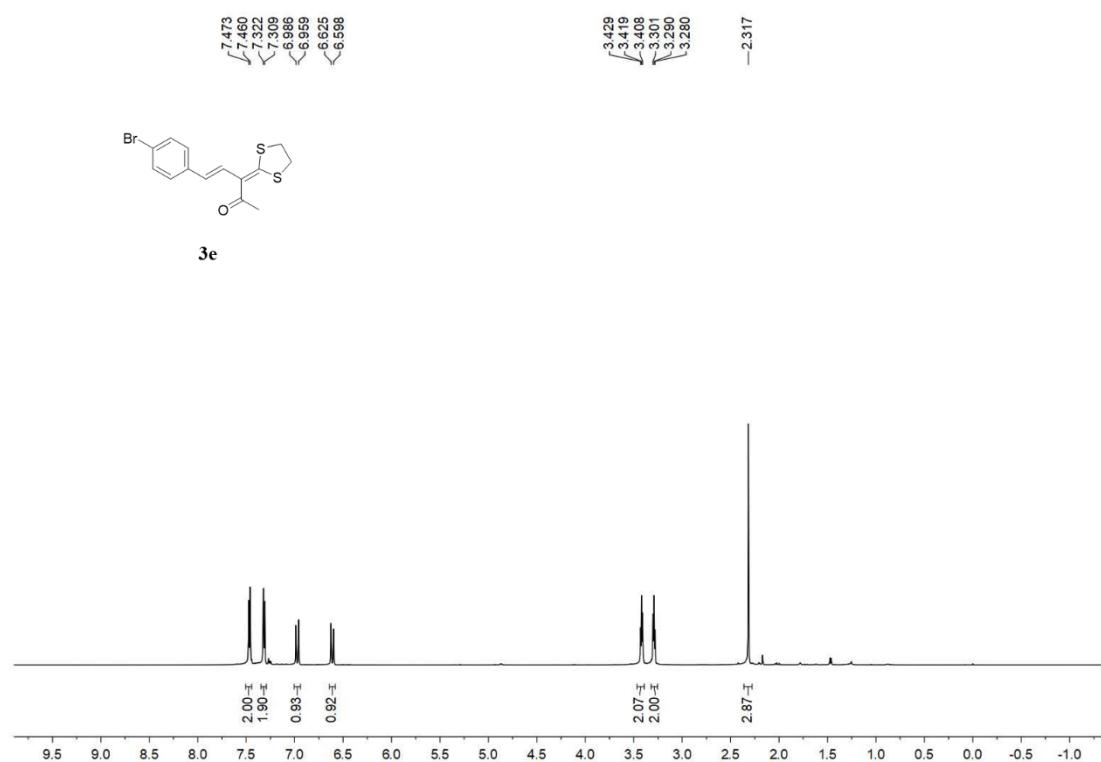
¹H spectrum (600 MHz, CDCl₃) of compound **3d**



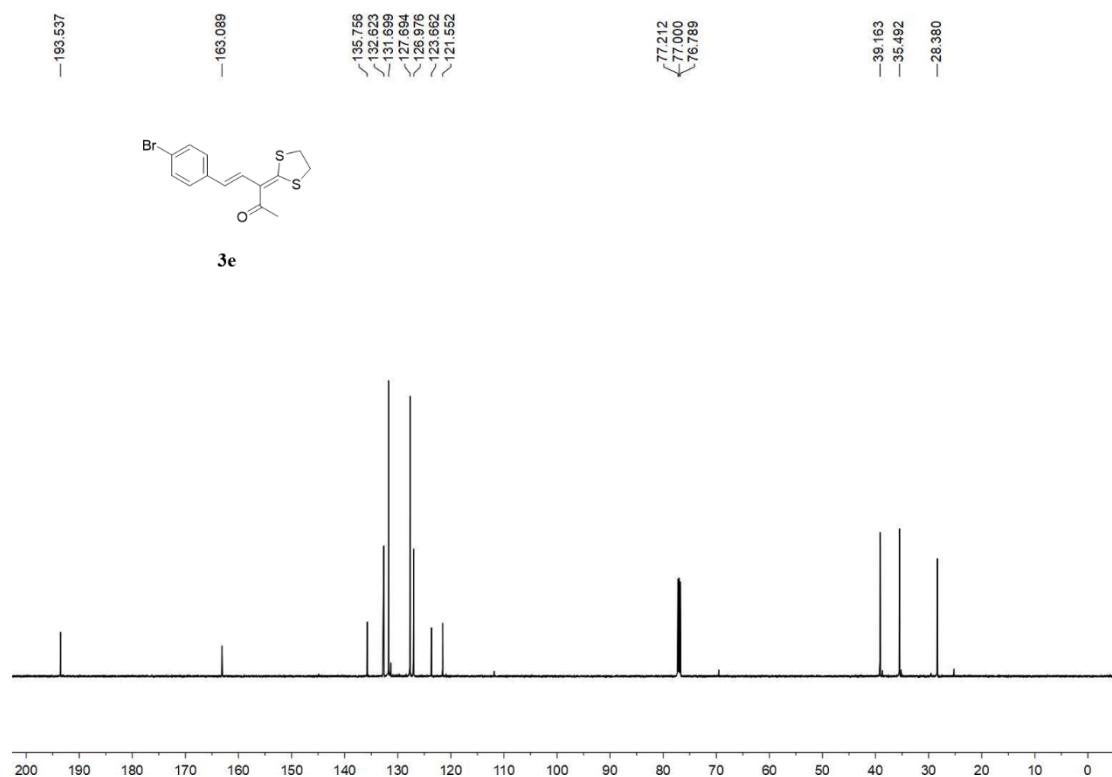
¹³C spectrum (151 MHz, CDCl₃) of compound **3d**



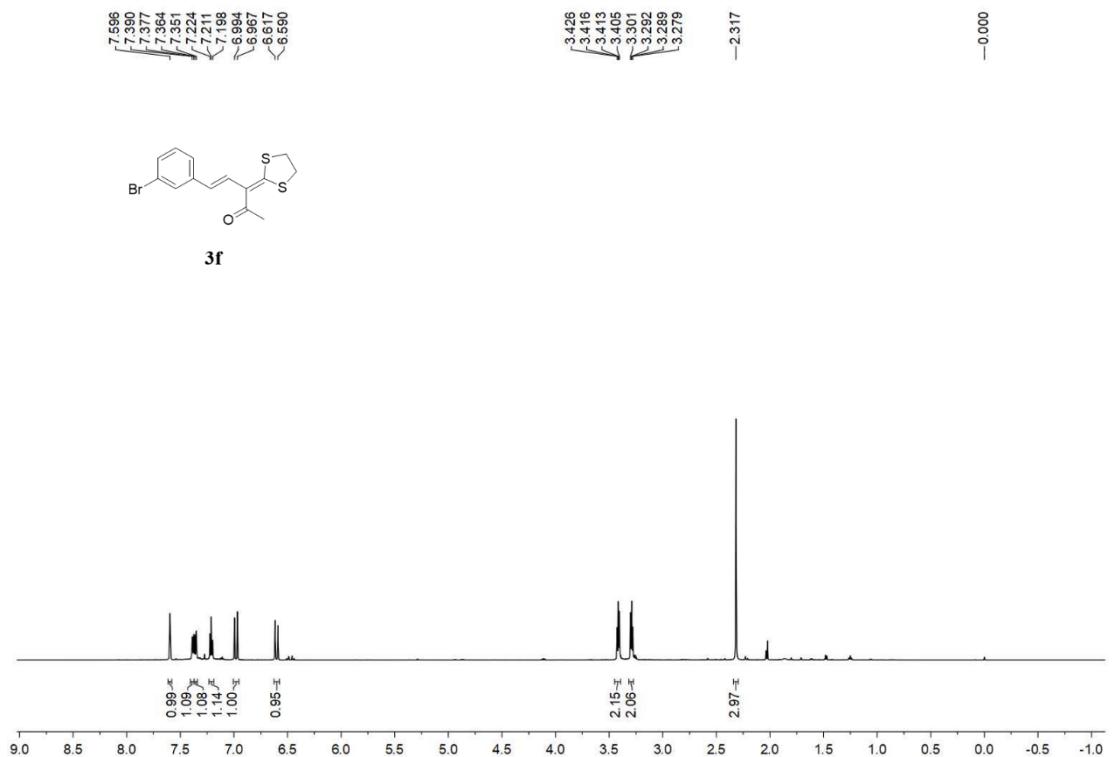
¹H spectrum (600 MHz, CDCl₃) of compound 3e



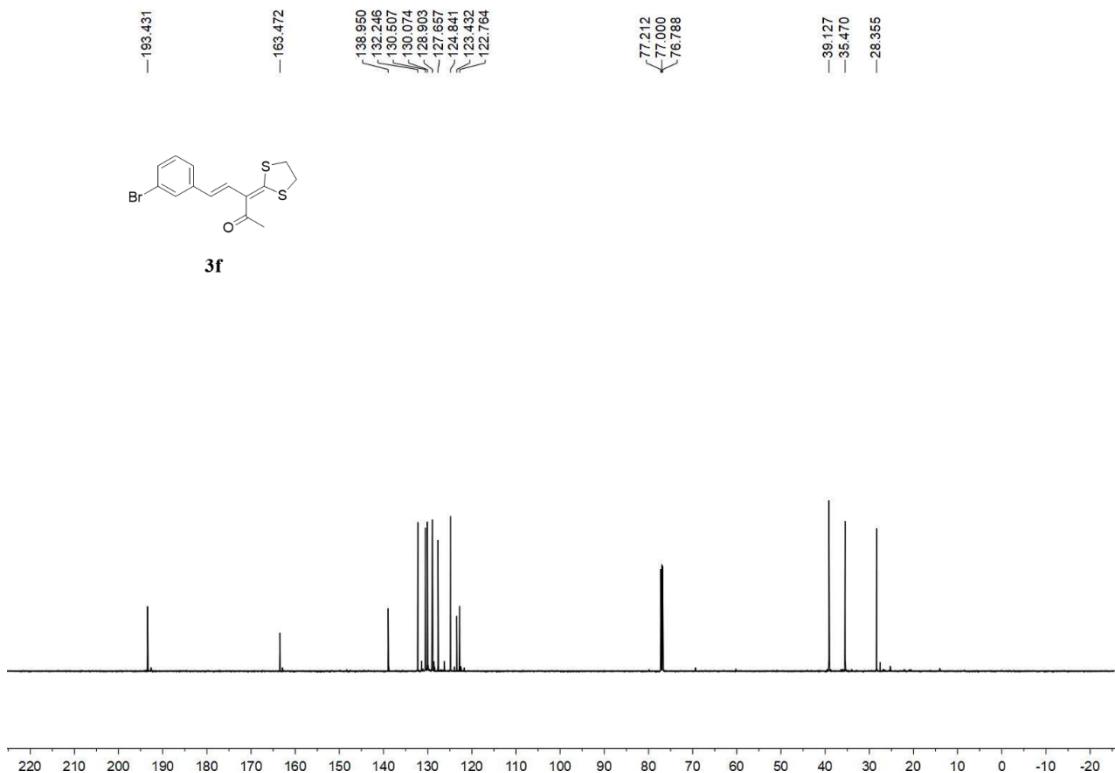
¹³C spectrum (151 MHz, CDCl₃) of compound 3e



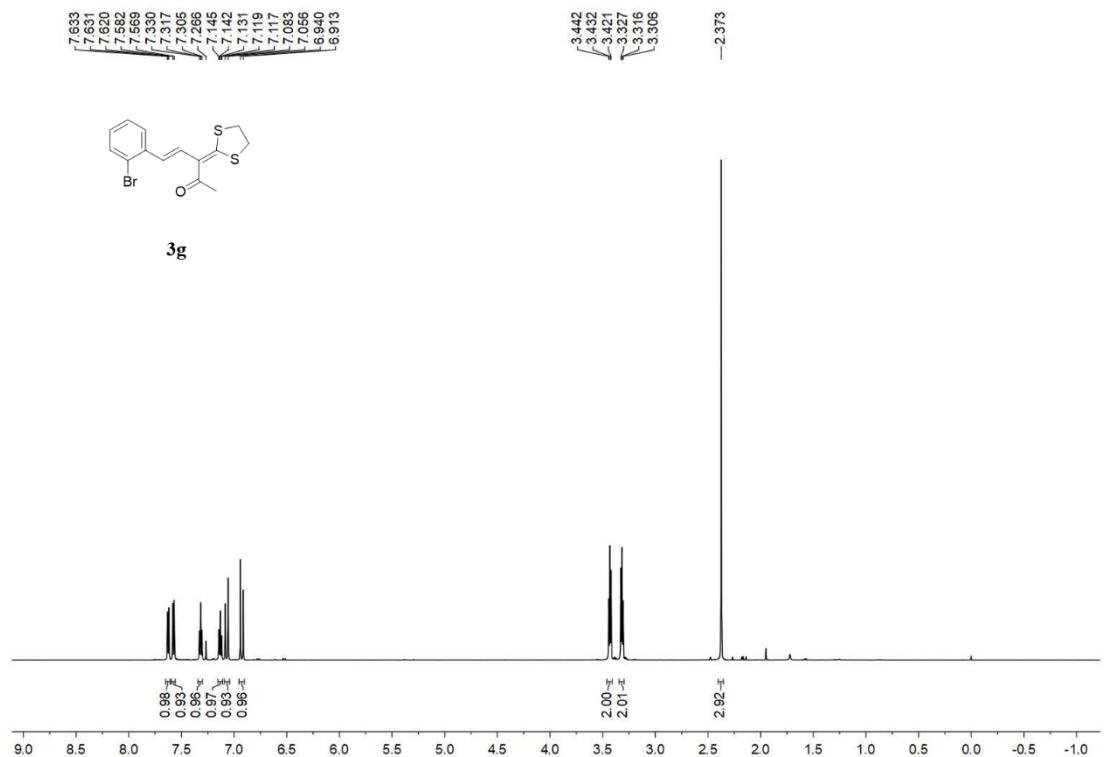
¹H spectrum (600 MHz, CDCl₃) of compound **3f**



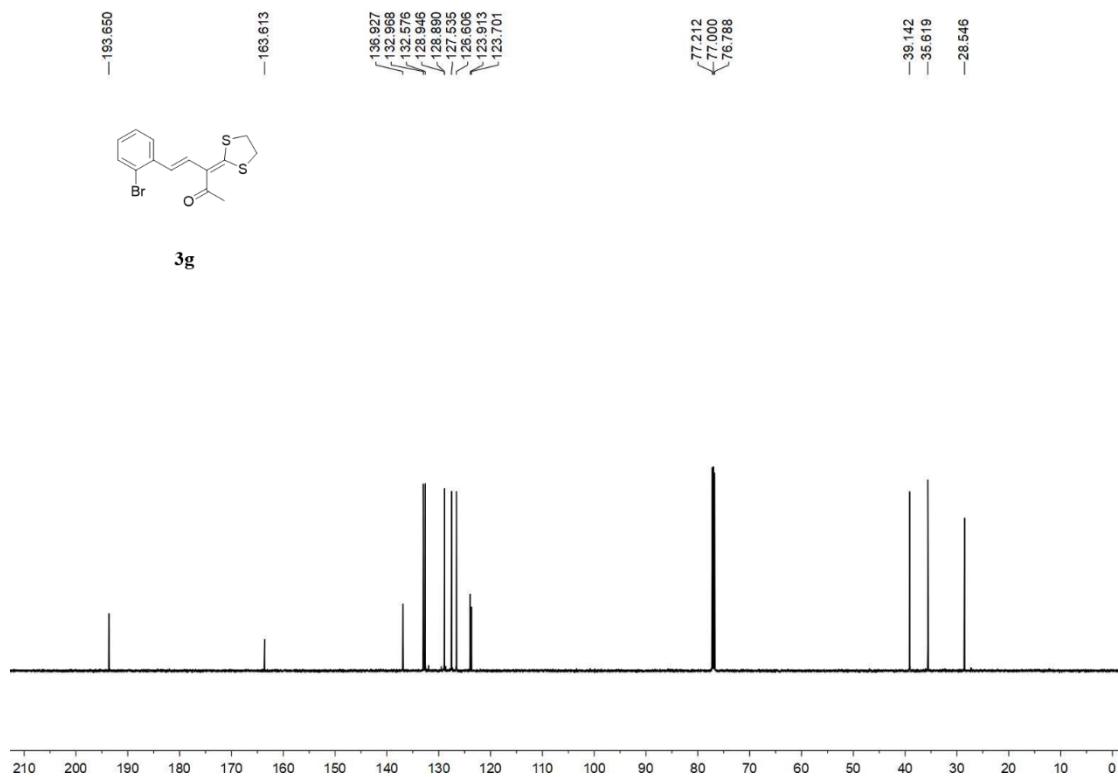
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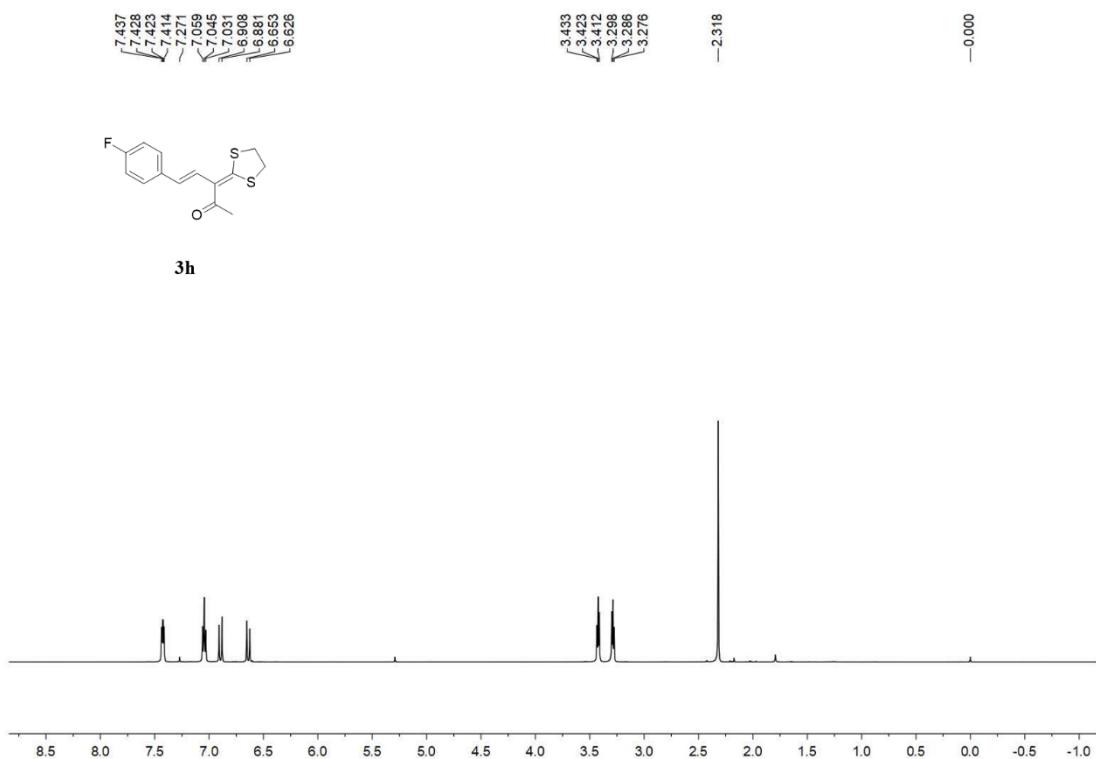
¹H spectrum (600 MHz, CDCl₃) of compound **3g**



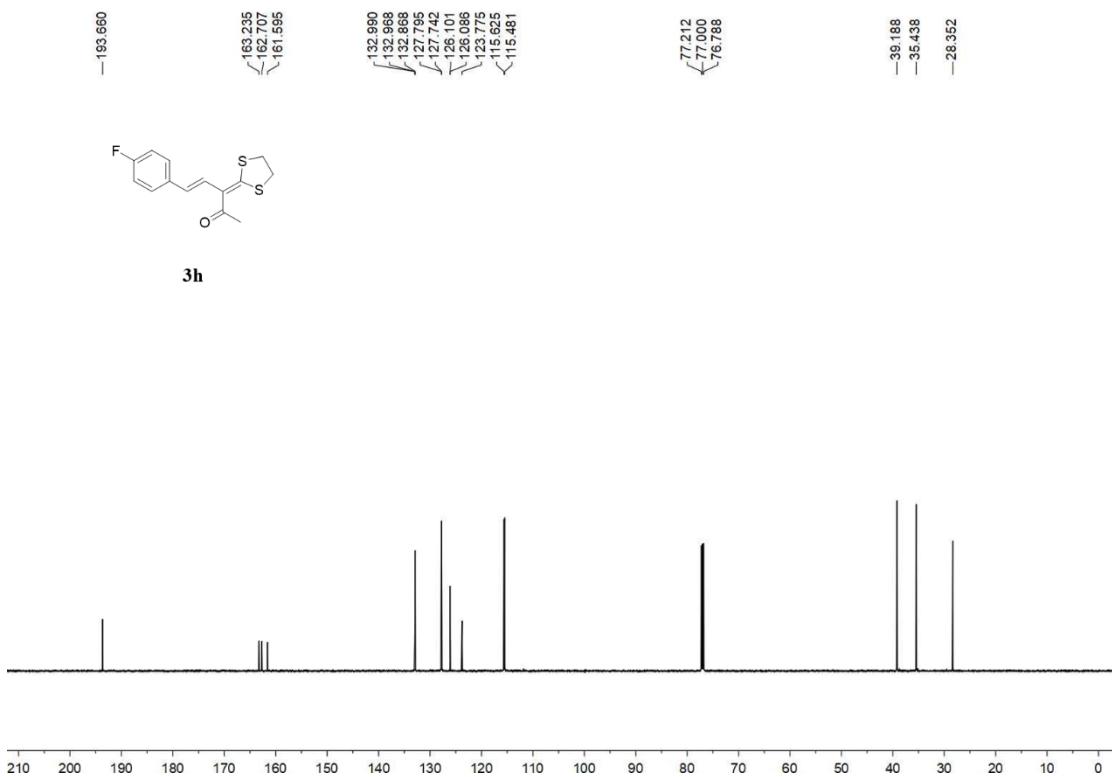
¹³C spectrum (151 MHz, CDCl₃) of compound 3g



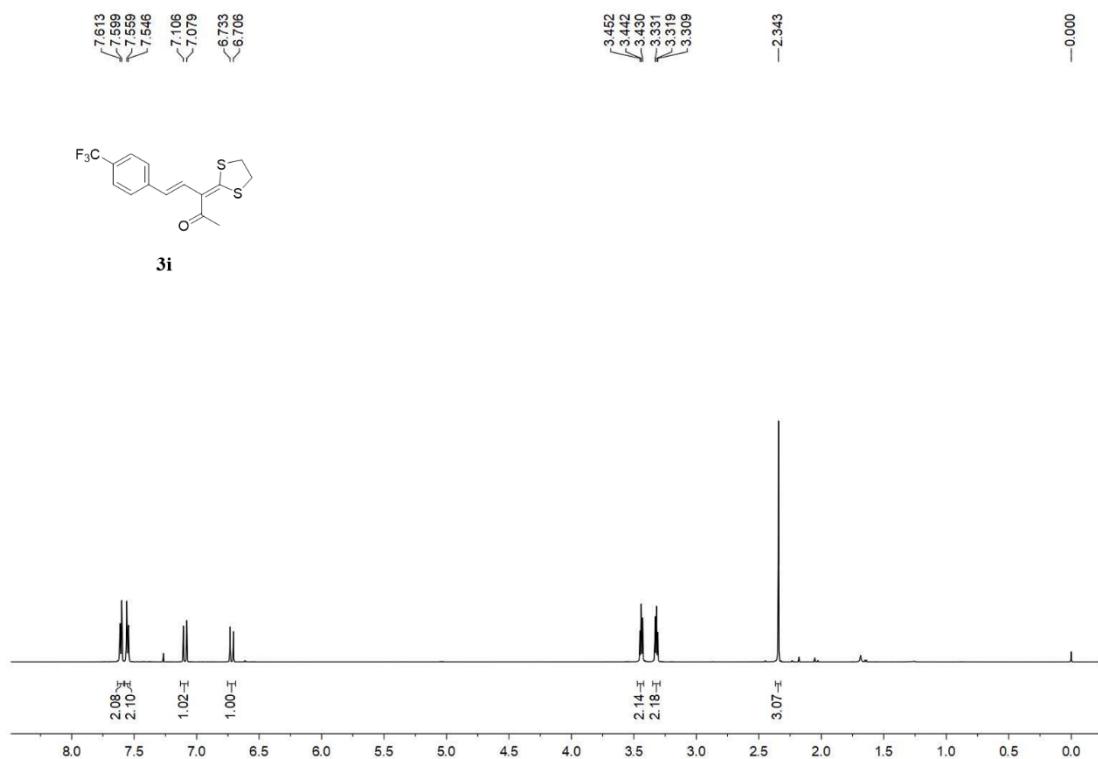
¹H spectrum (600 MHz, CDCl₃) of compound **3h**



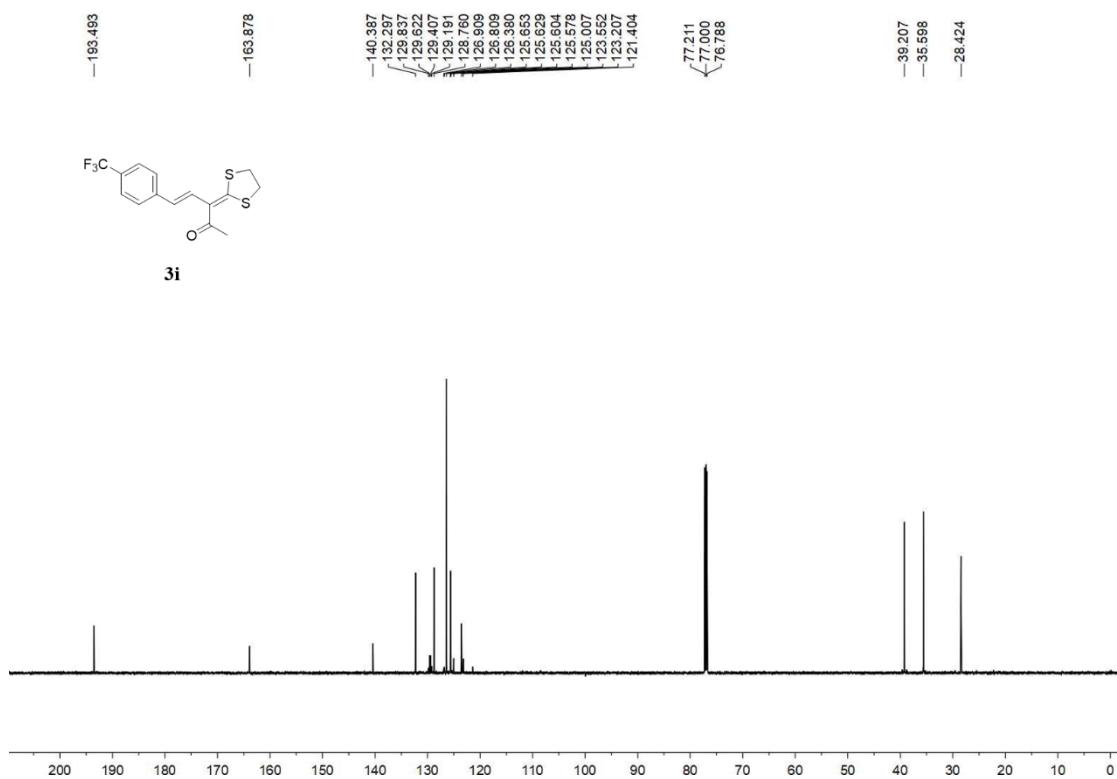
¹³C spectrum (151 MHz, CDCl₃) of compound **3h**



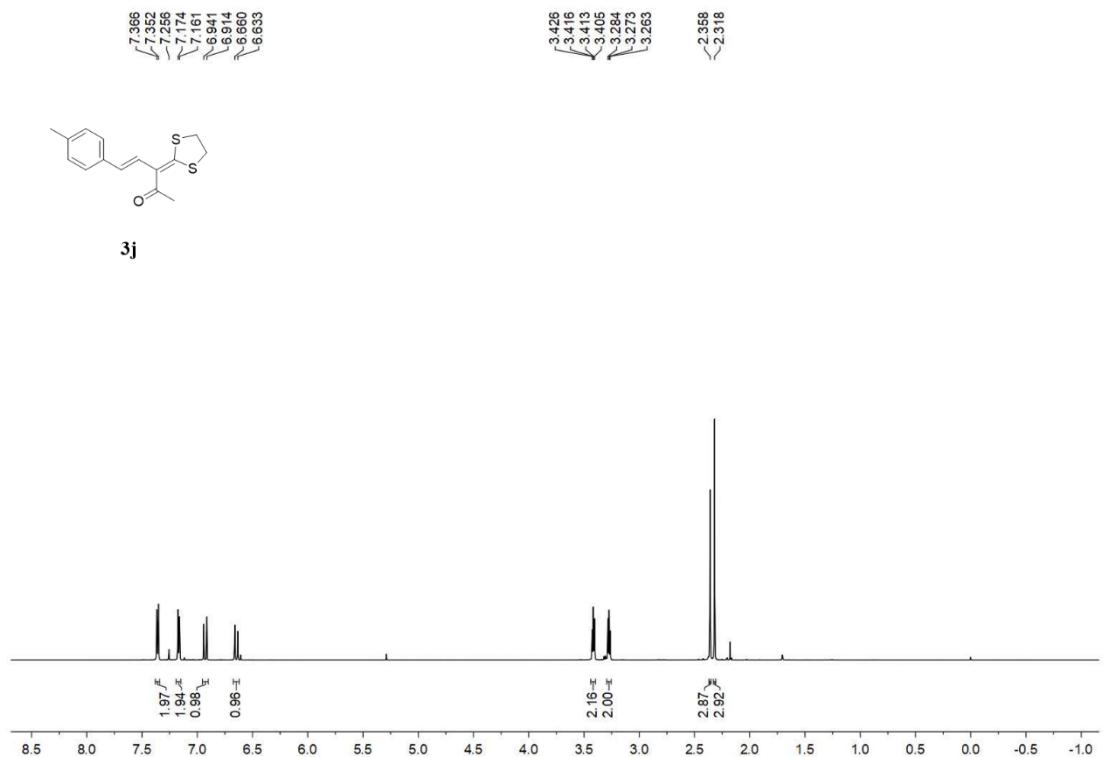
¹H spectrum (600 MHz, CDCl₃) of compound **3i**



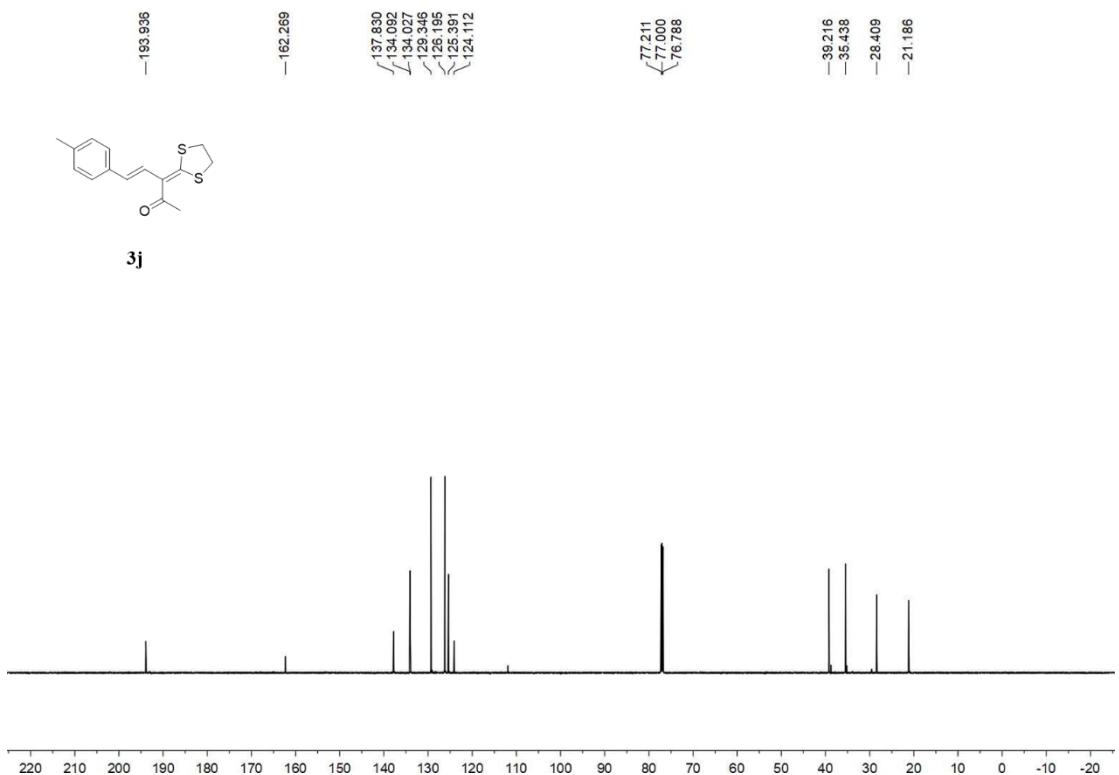
¹³C spectrum (151 MHz, CDCl₃) of compound **3i**



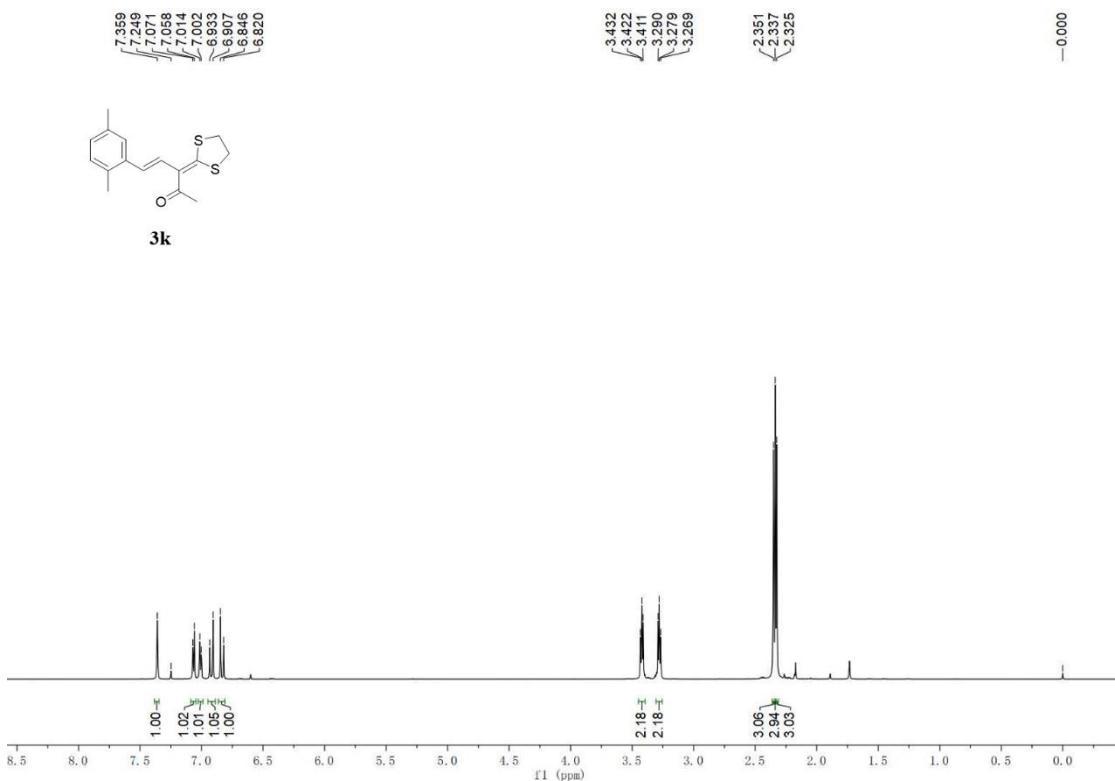
¹H spectrum (600 MHz, CDCl₃) of compound **3j**



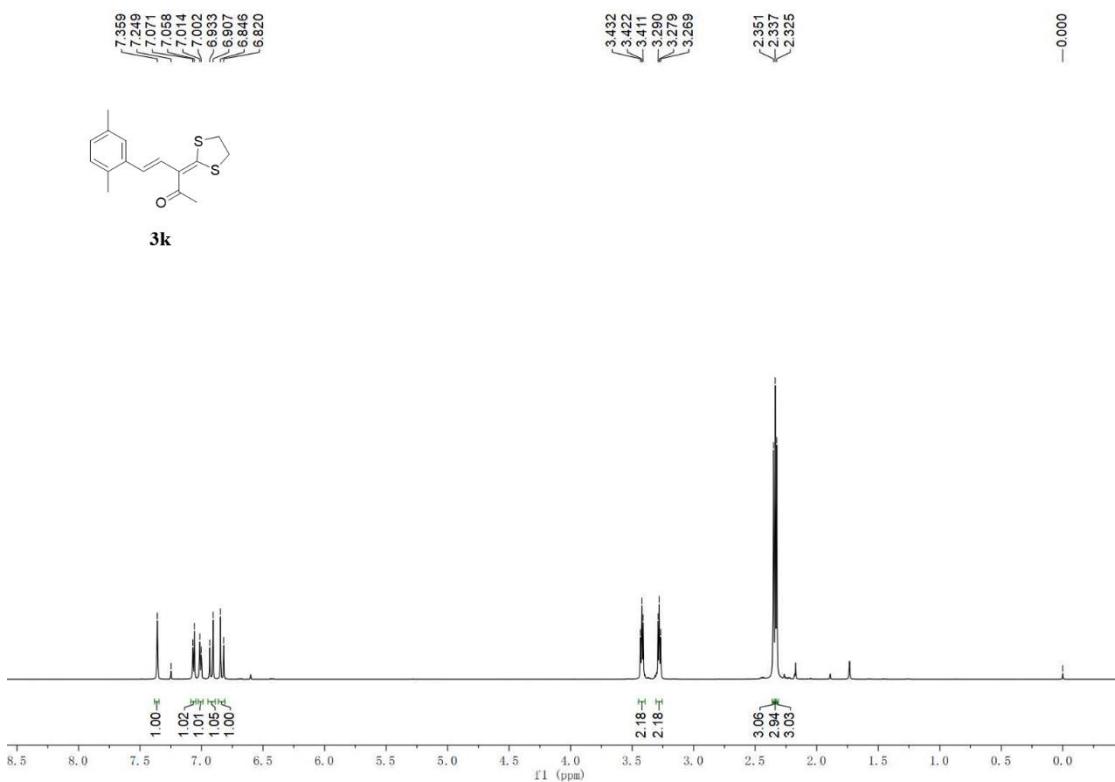
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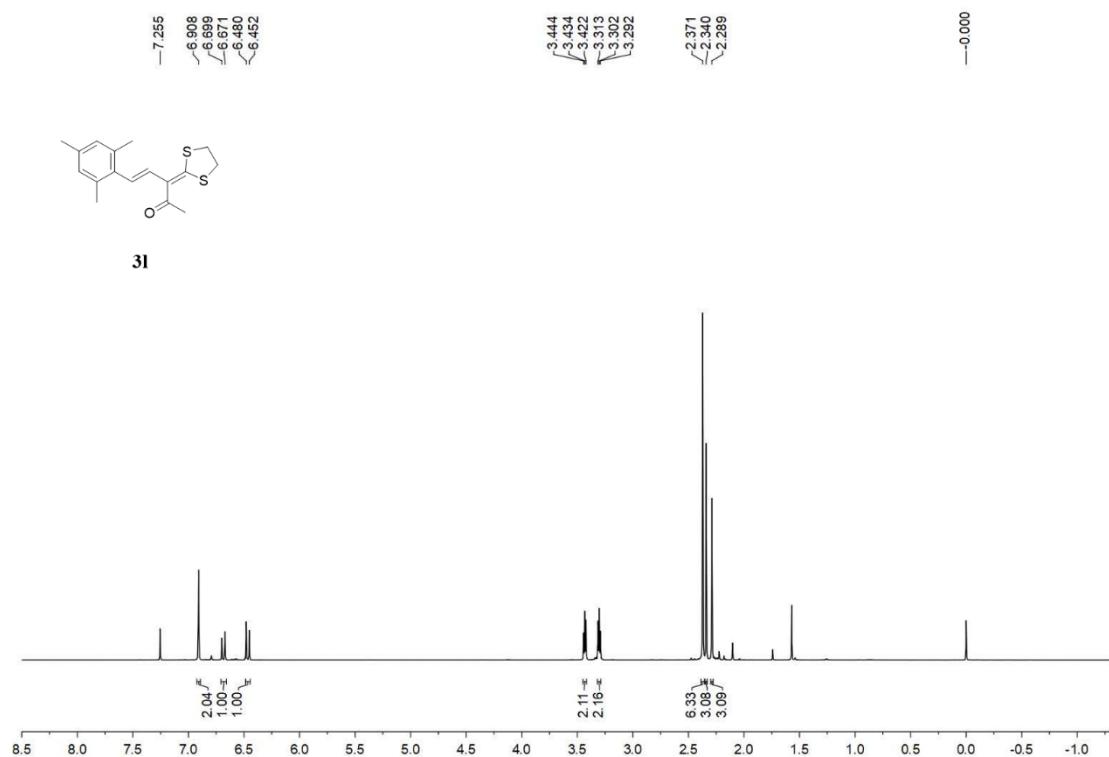
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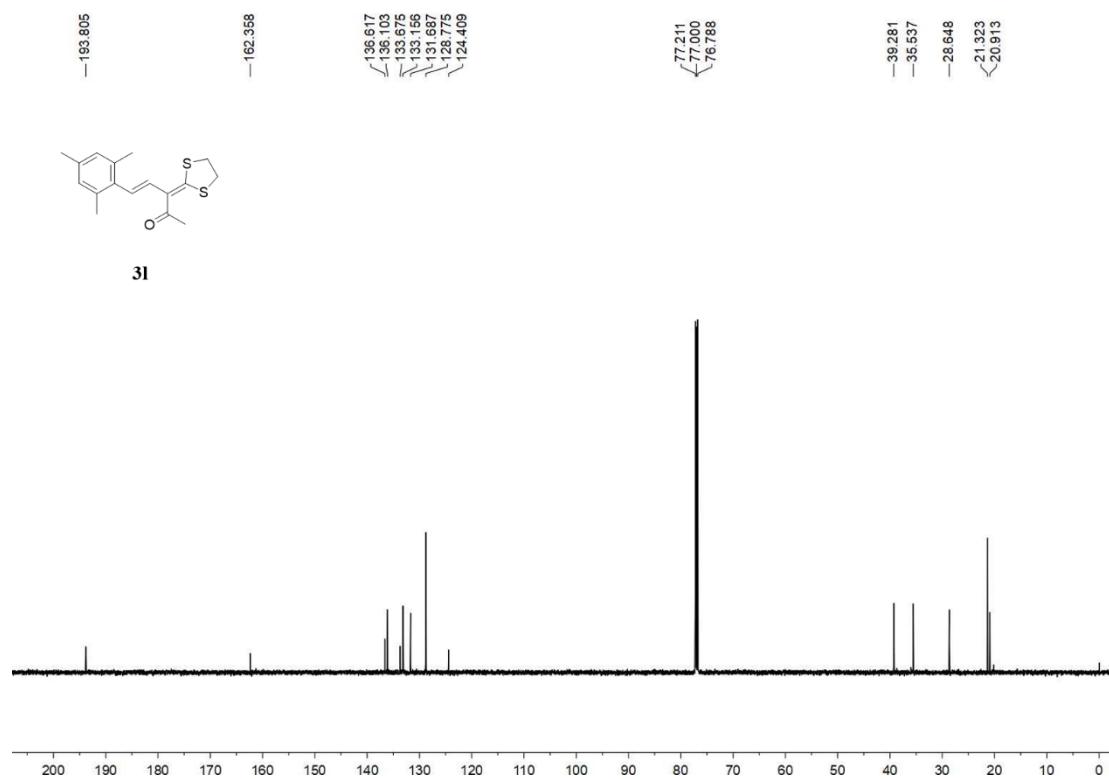
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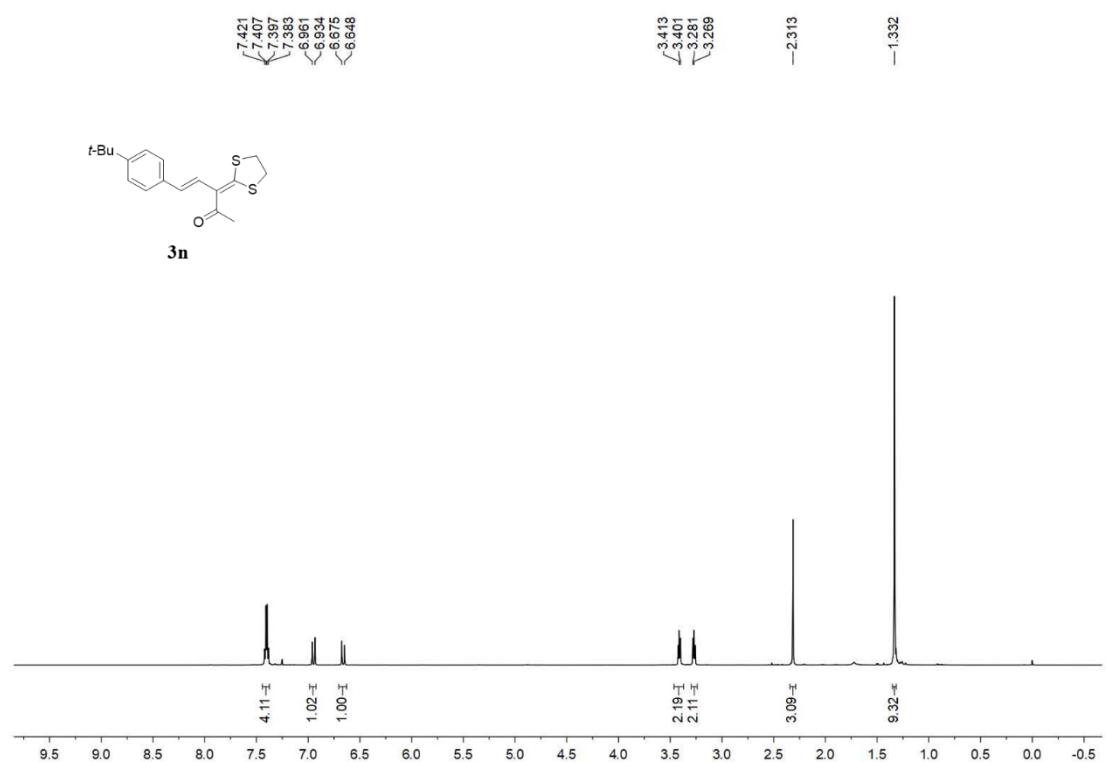
¹H spectrum (600 MHz, CDCl₃) of compound 3l



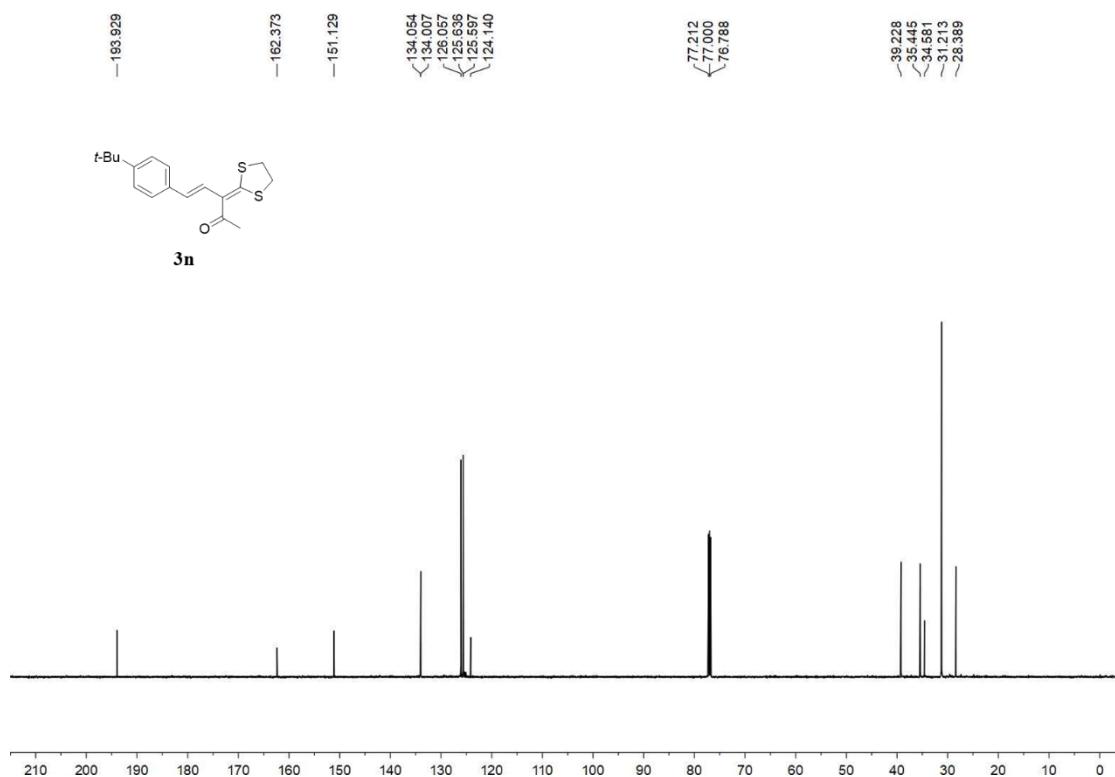
¹³C spectrum (151 MHz, CDCl₃) of compound 3l



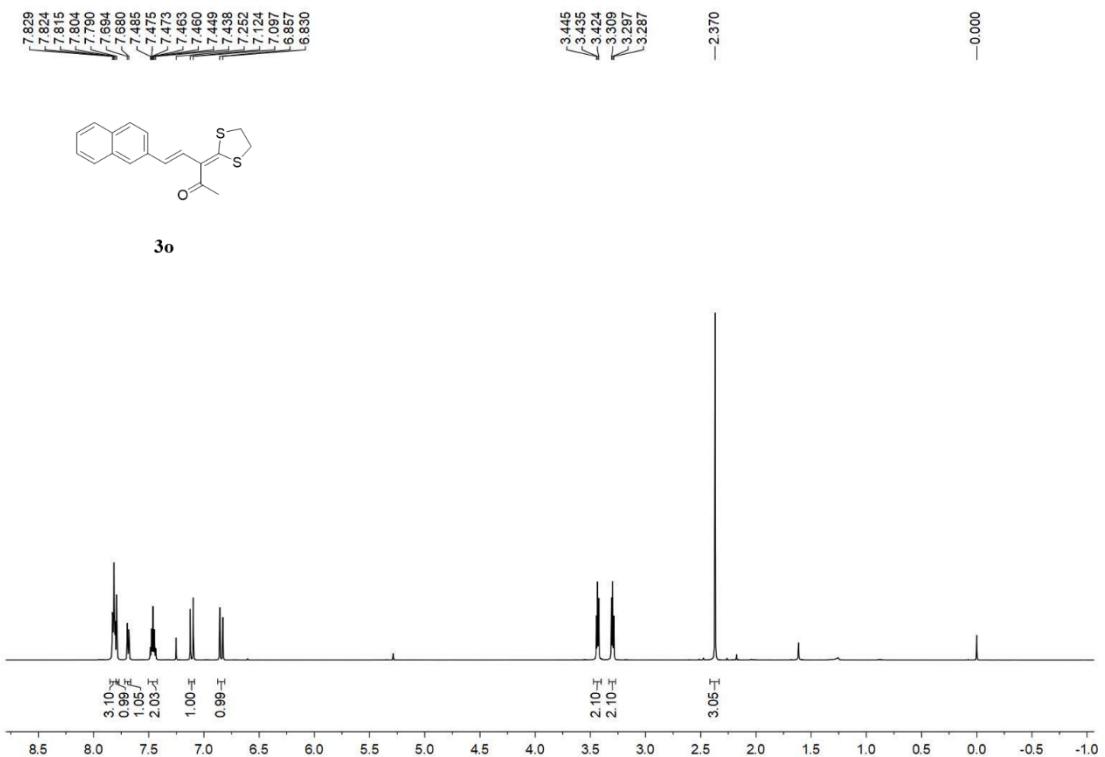
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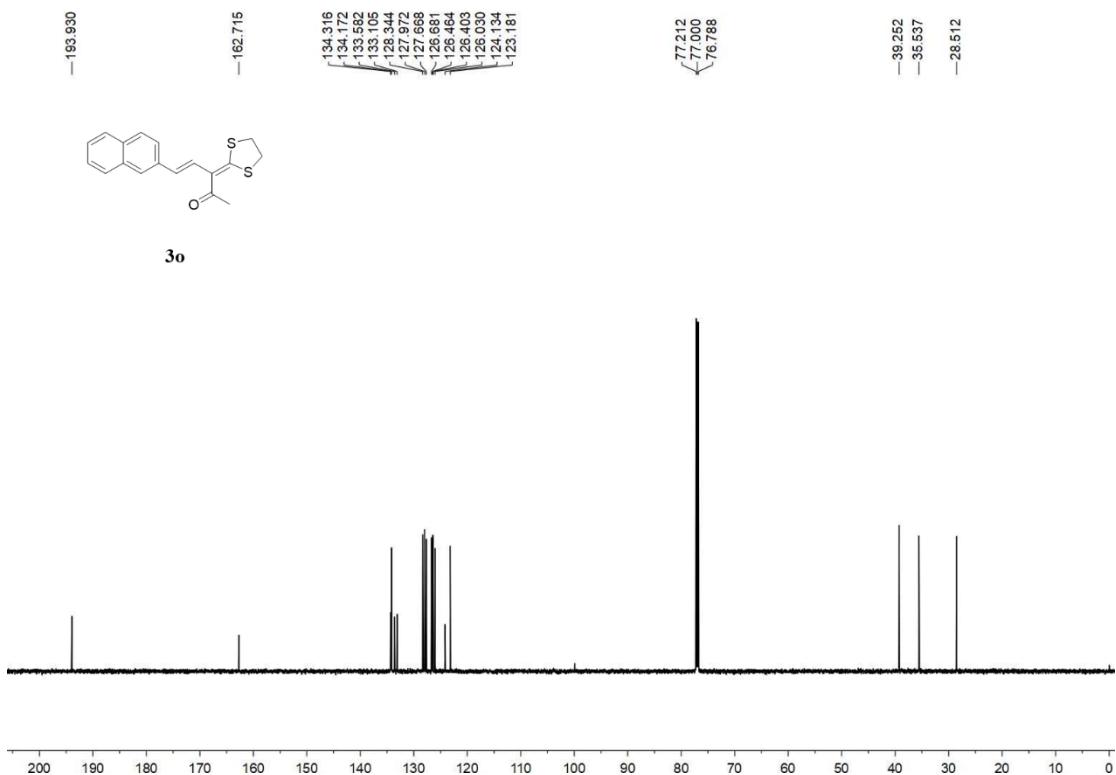
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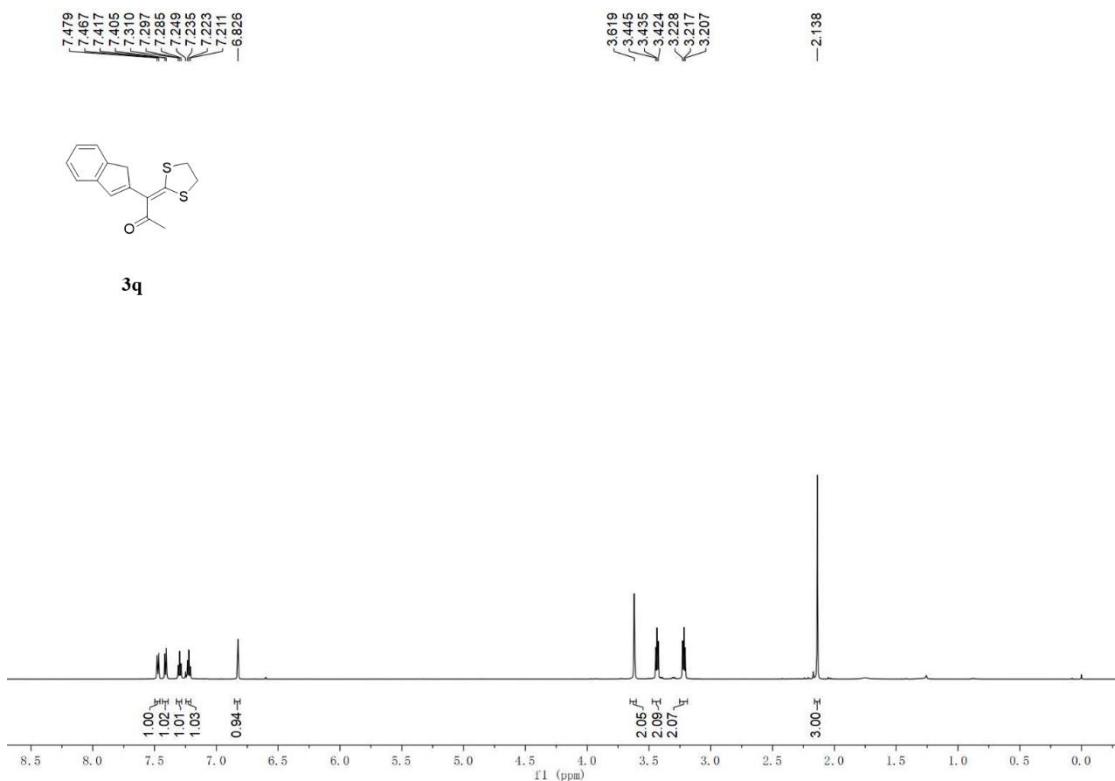
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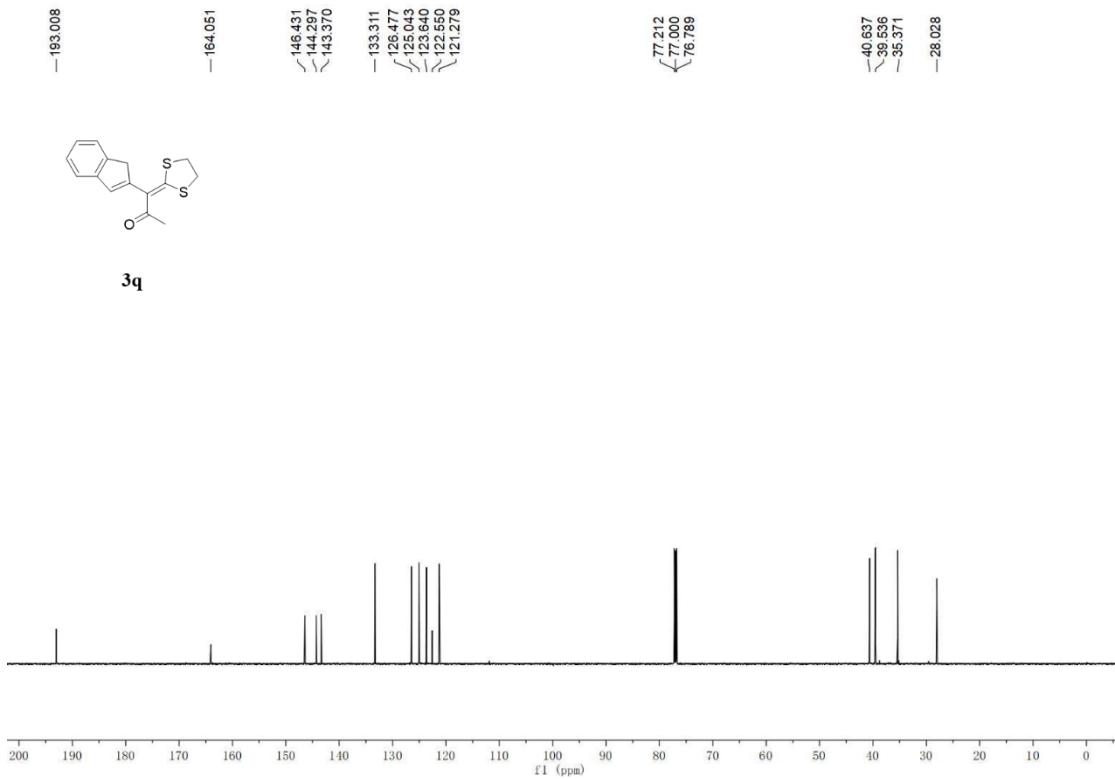
¹³C spectrum (151 MHz, CDCl₃) of compound **3o**



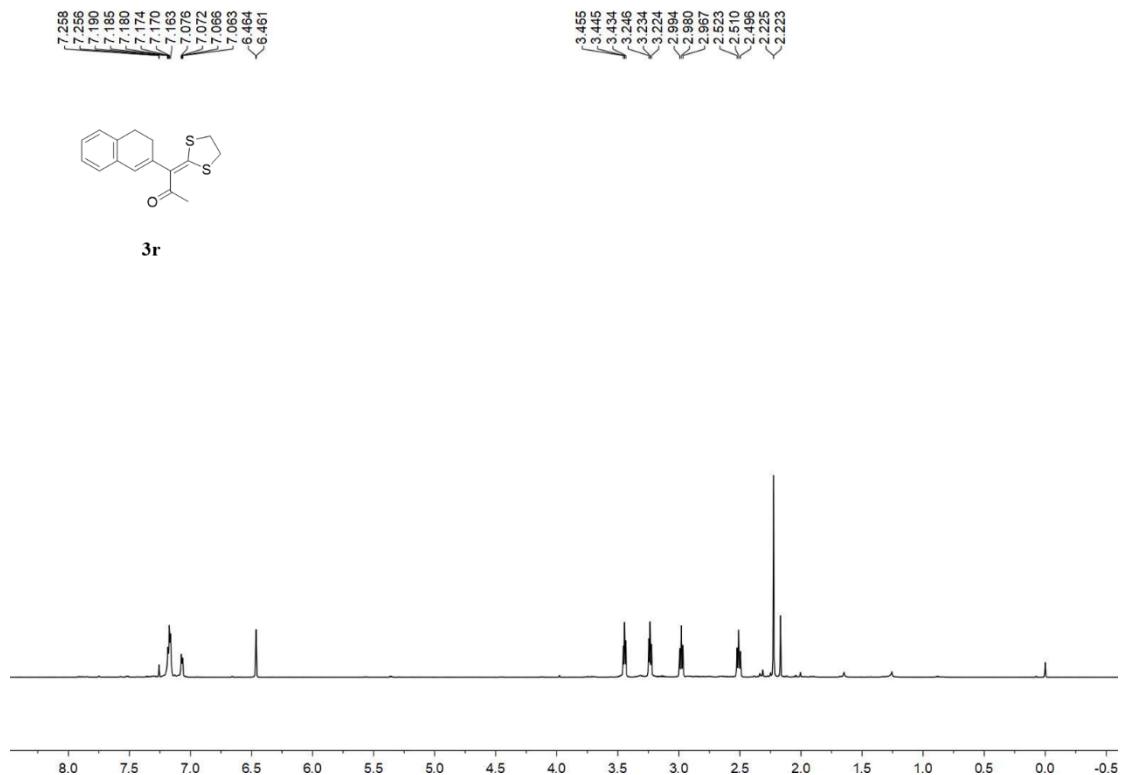
¹H spectrum (600 MHz, CDCl₃) of compound **3q**



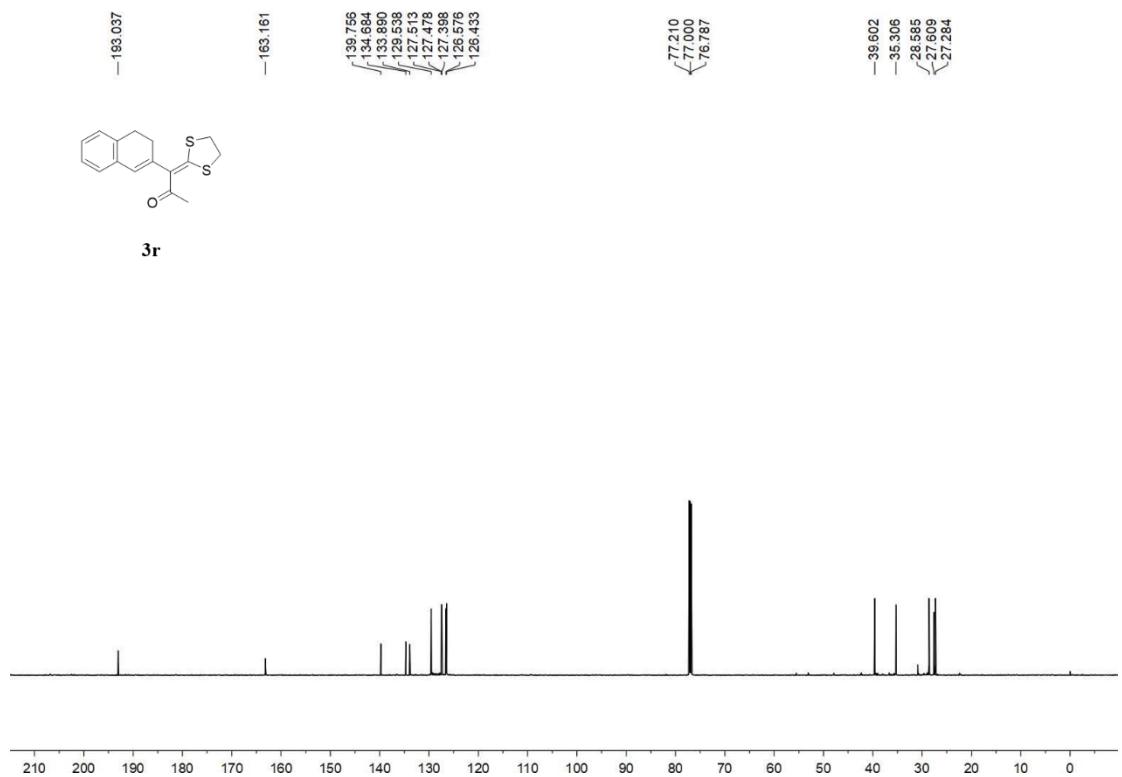
¹³C spectrum (151 MHz, CDCl₃) of compound **3q**



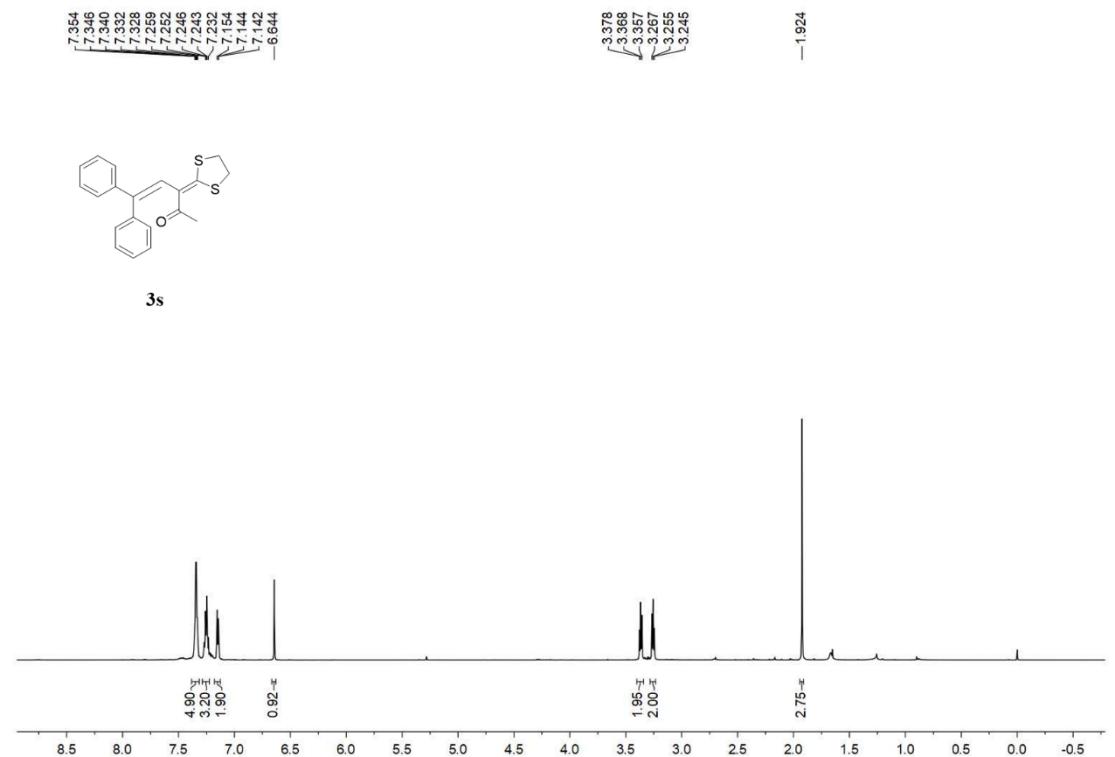
¹H spectrum (600 MHz, CDCl₃) of compound **3r**



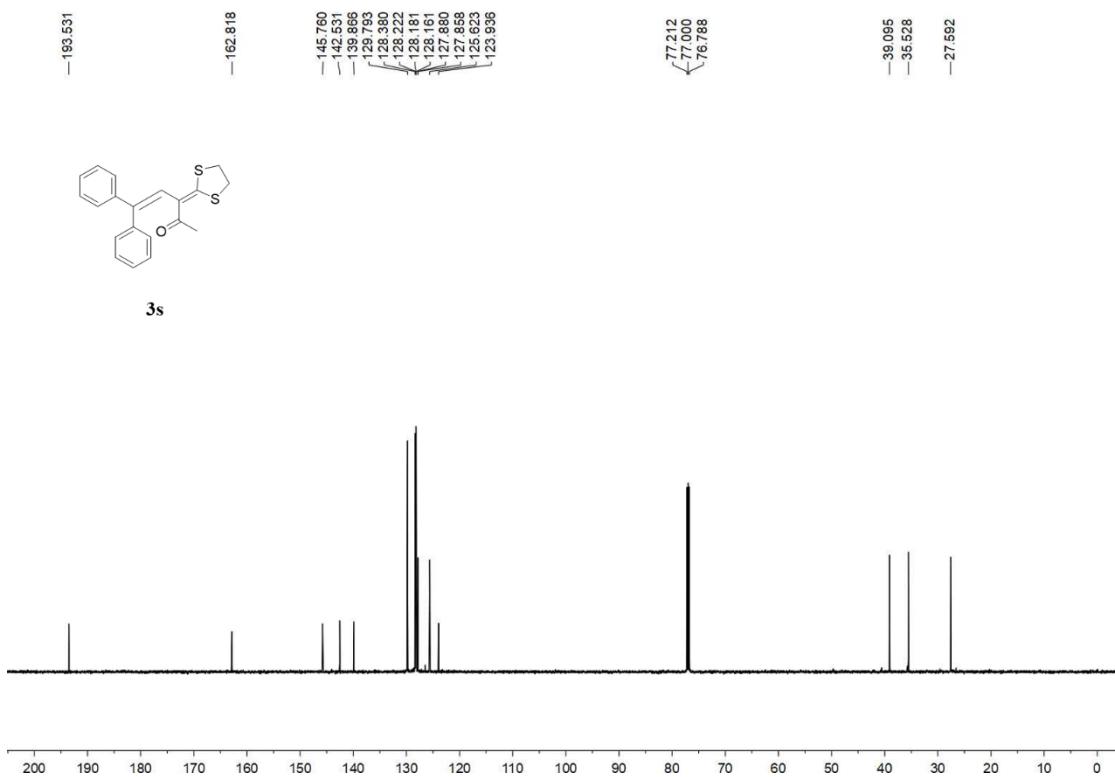
¹³C spectrum (151 MHz, CDCl₃) of compound **3r**



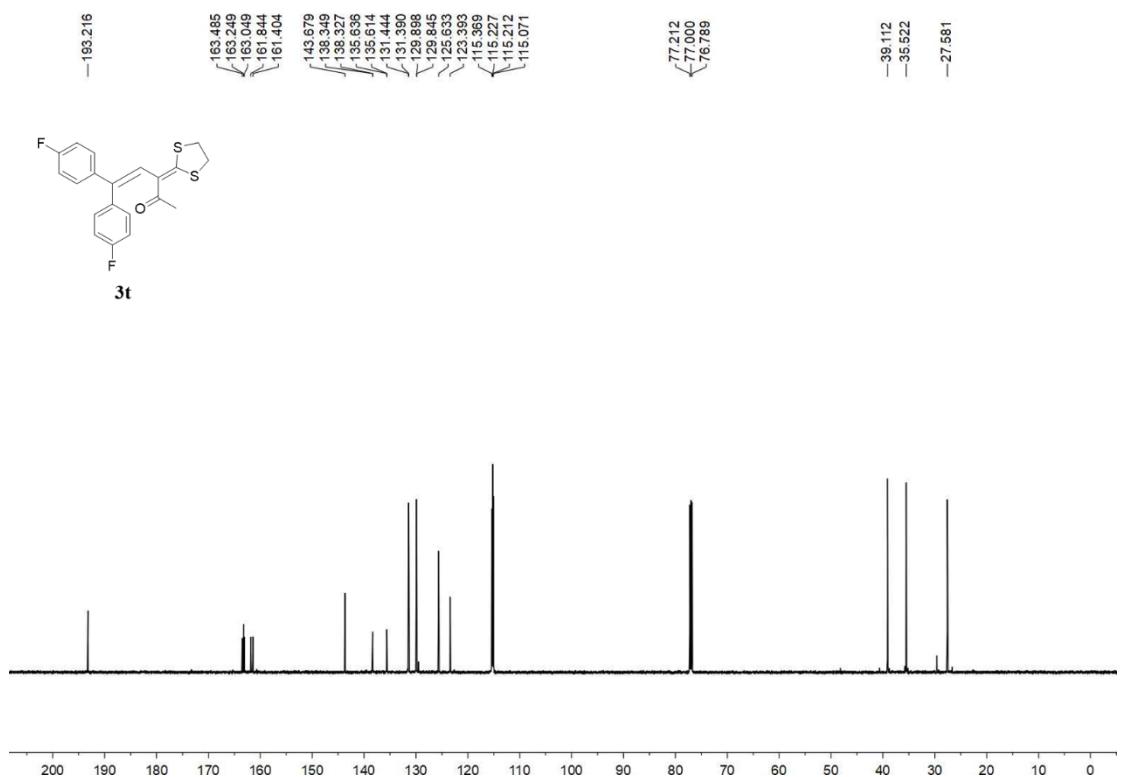
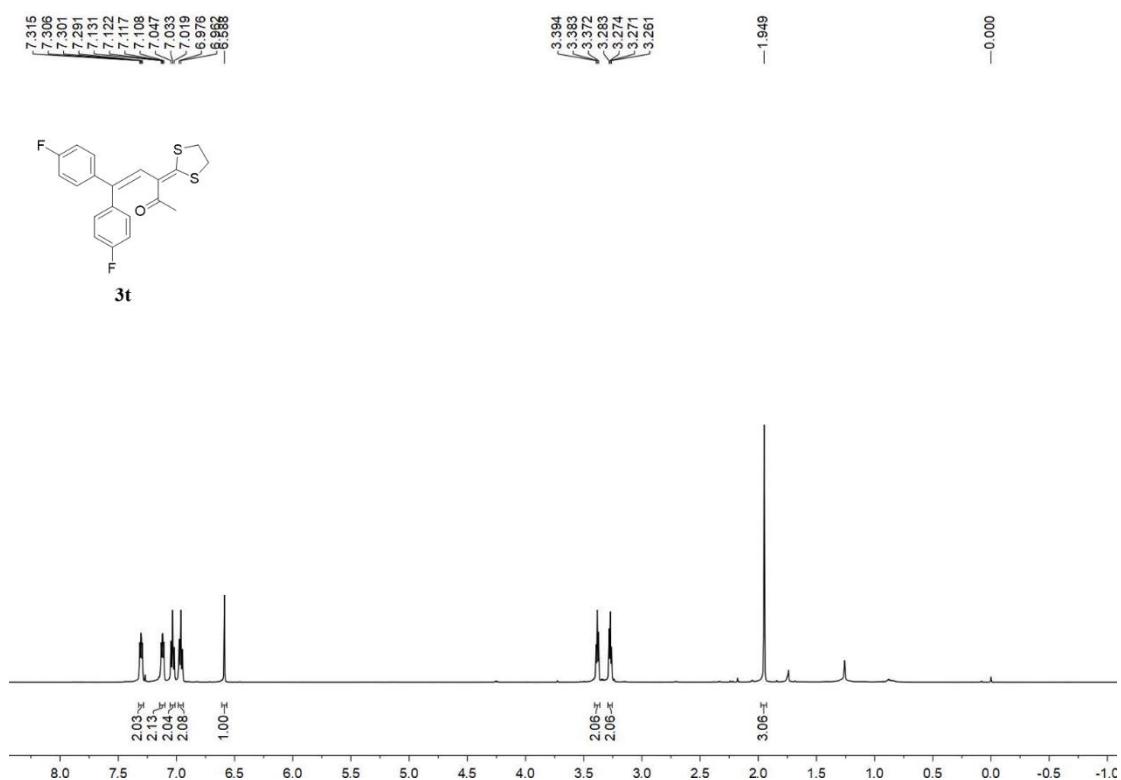
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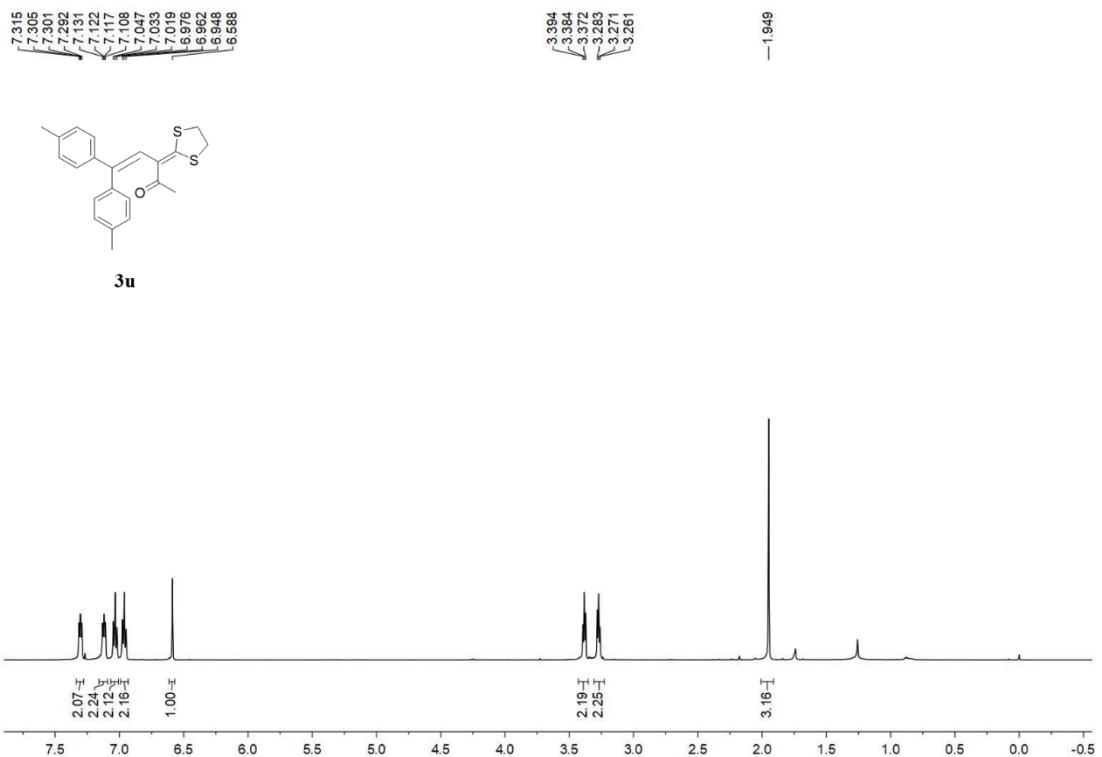
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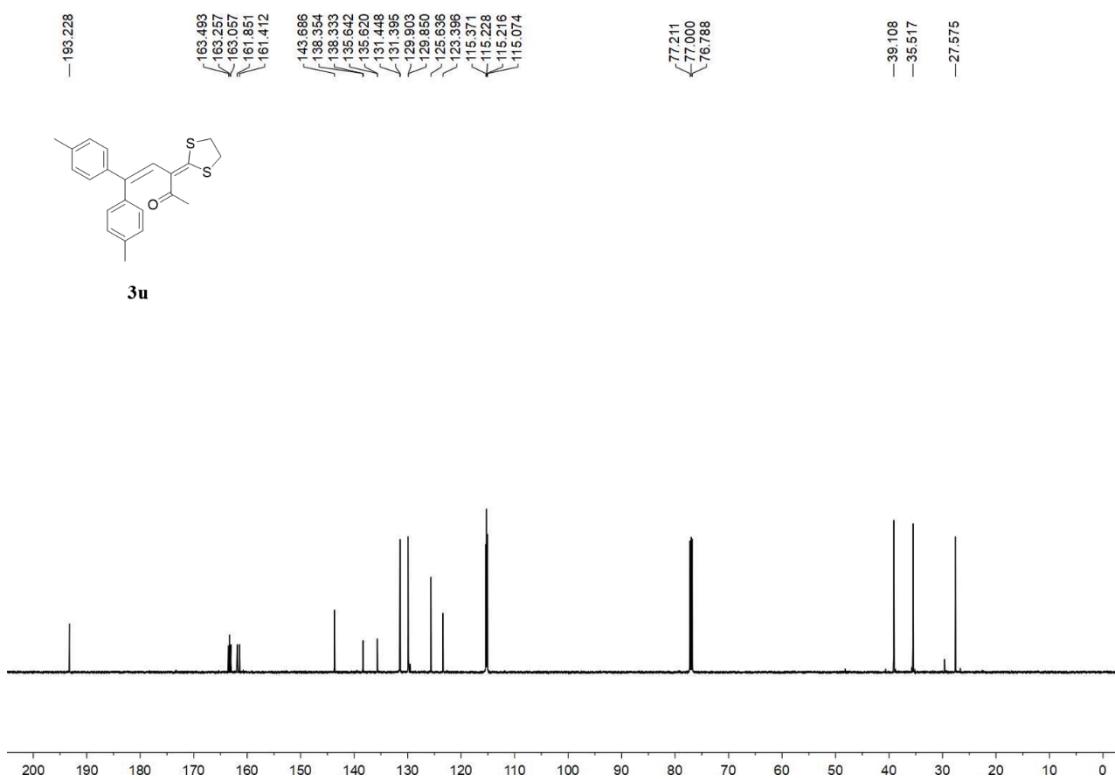
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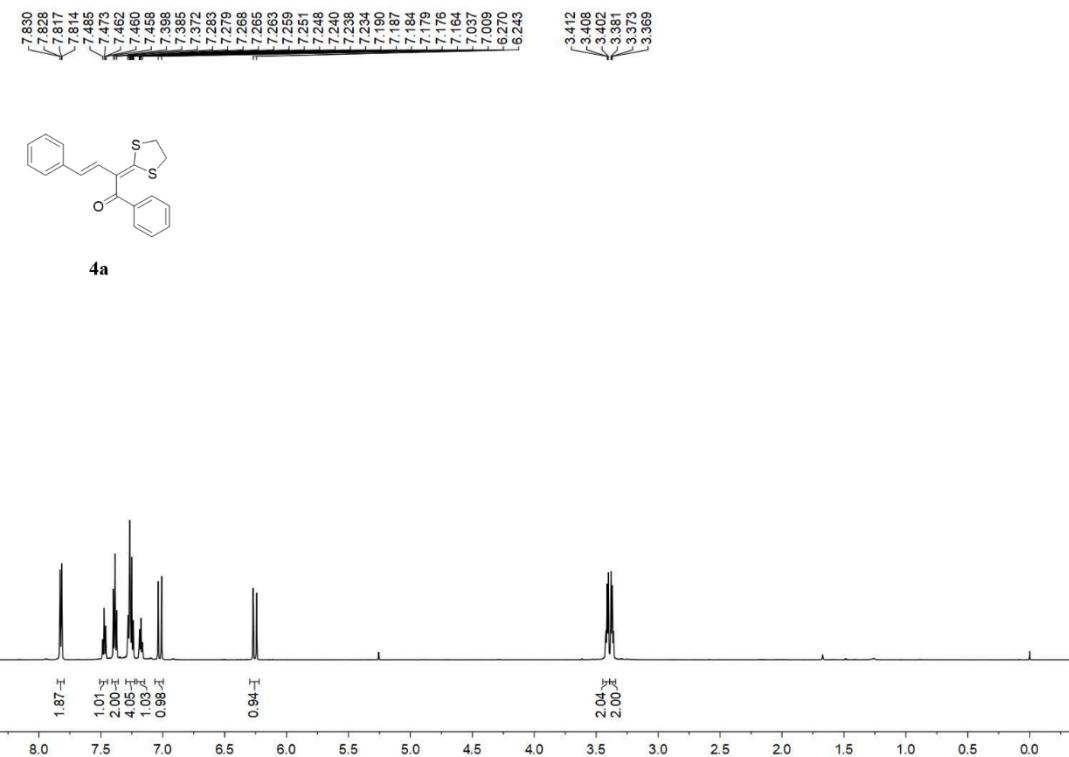
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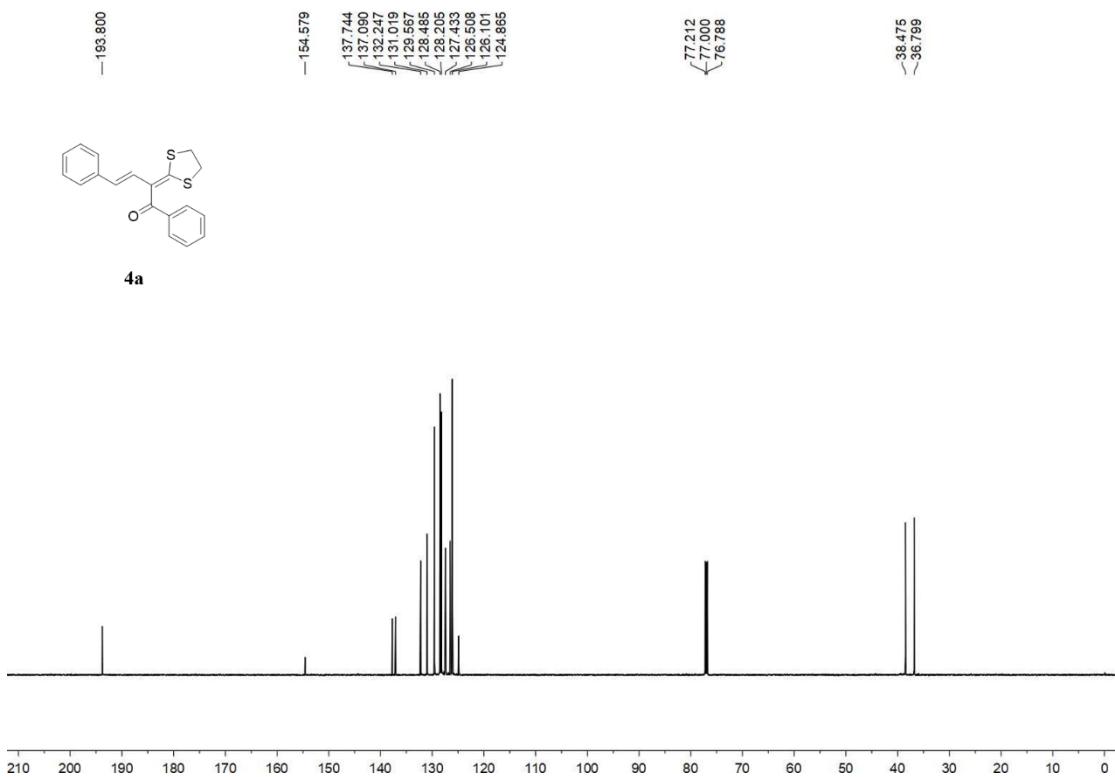
¹³C spectrum (151 MHz, CDCl₃) of compound **3u**



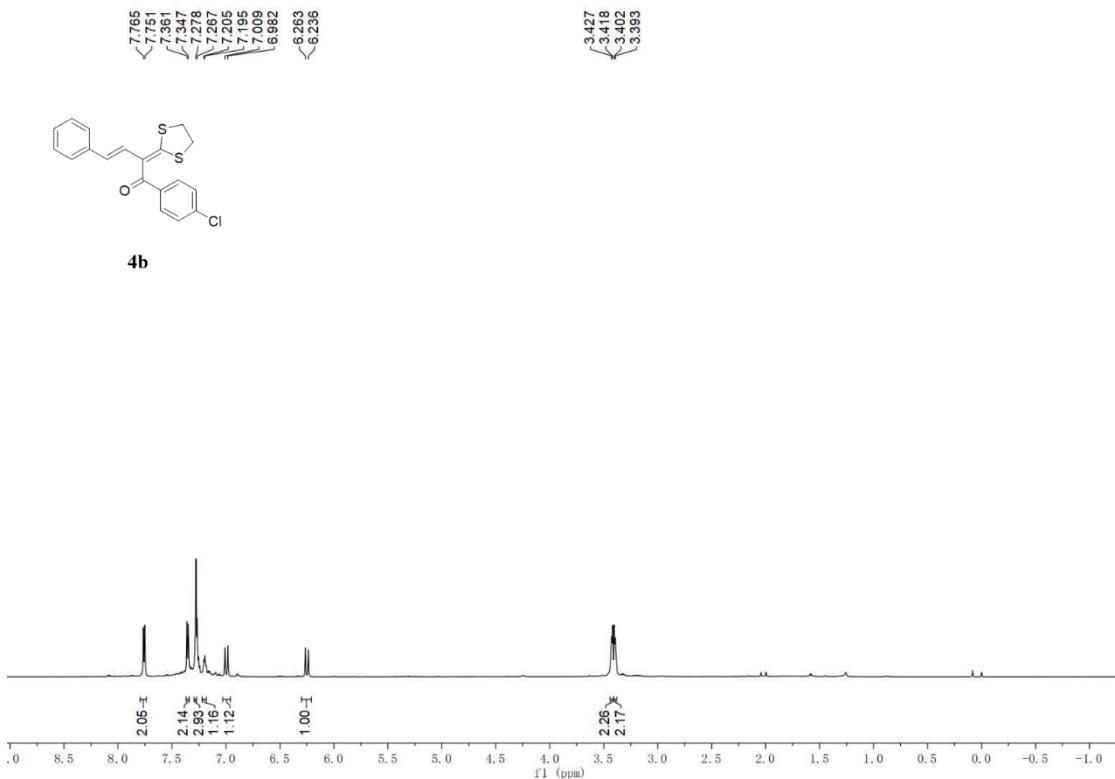
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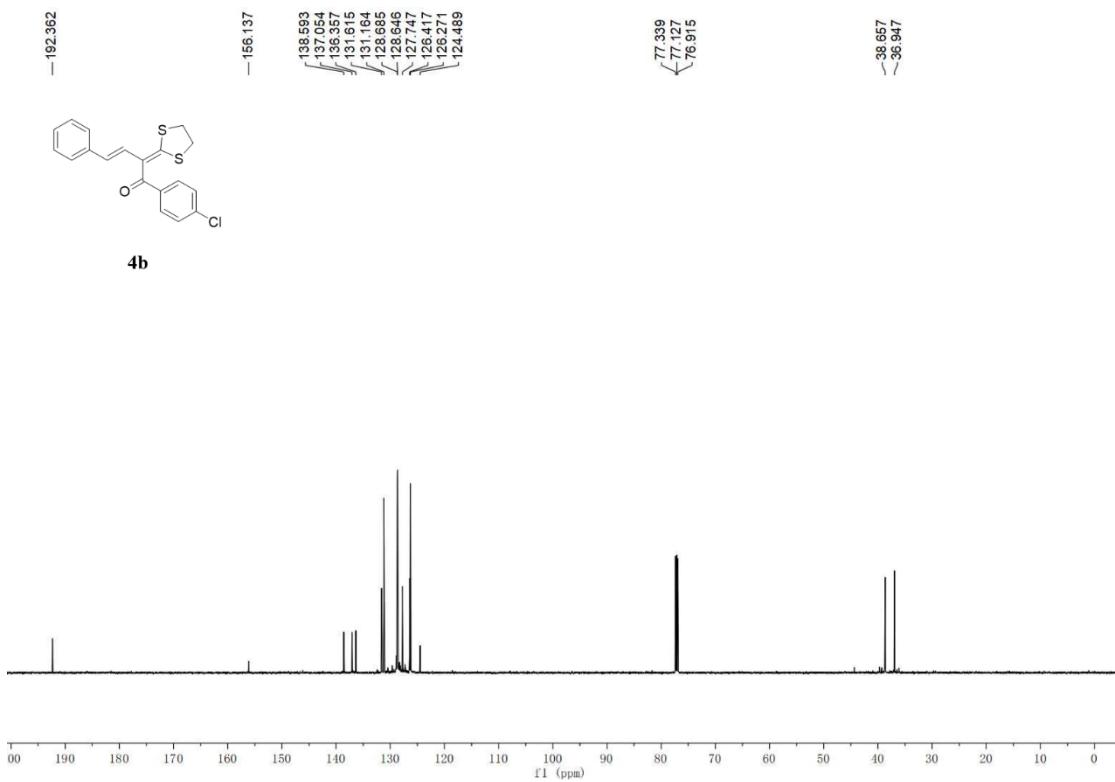
¹³C spectrum (151 MHz, CDCl₃) of compound **4a**



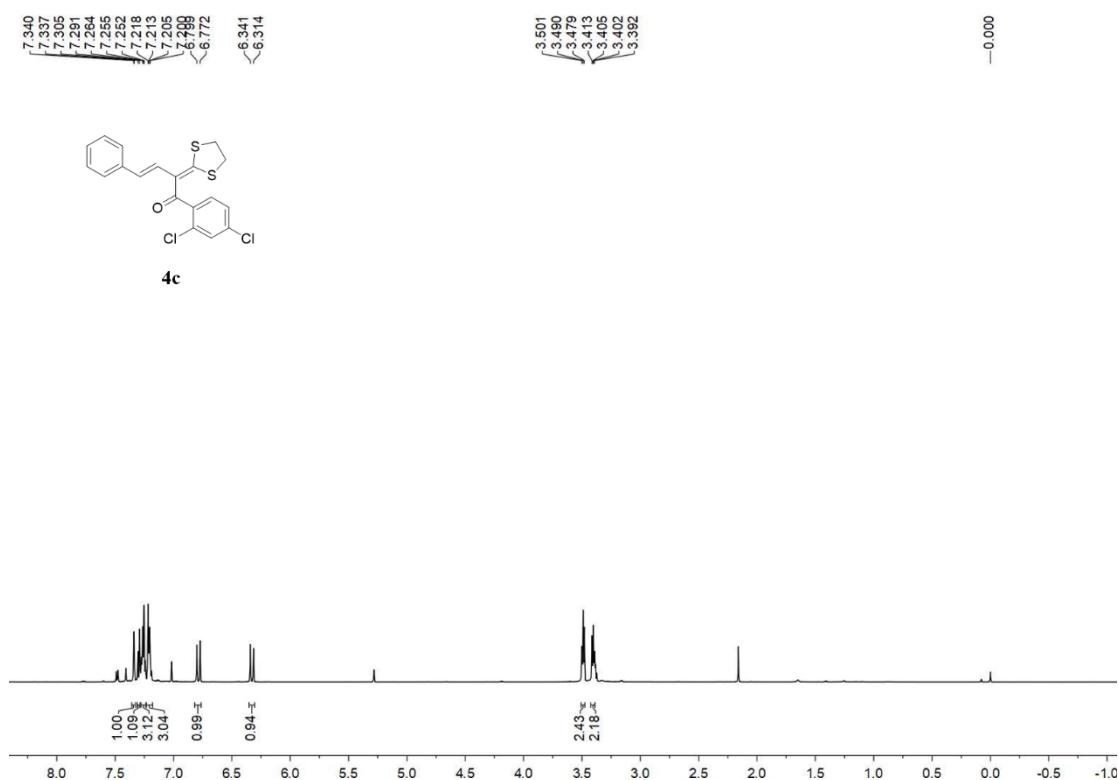
¹H spectrum (600 MHz, CDCl₃) of compound **4b**



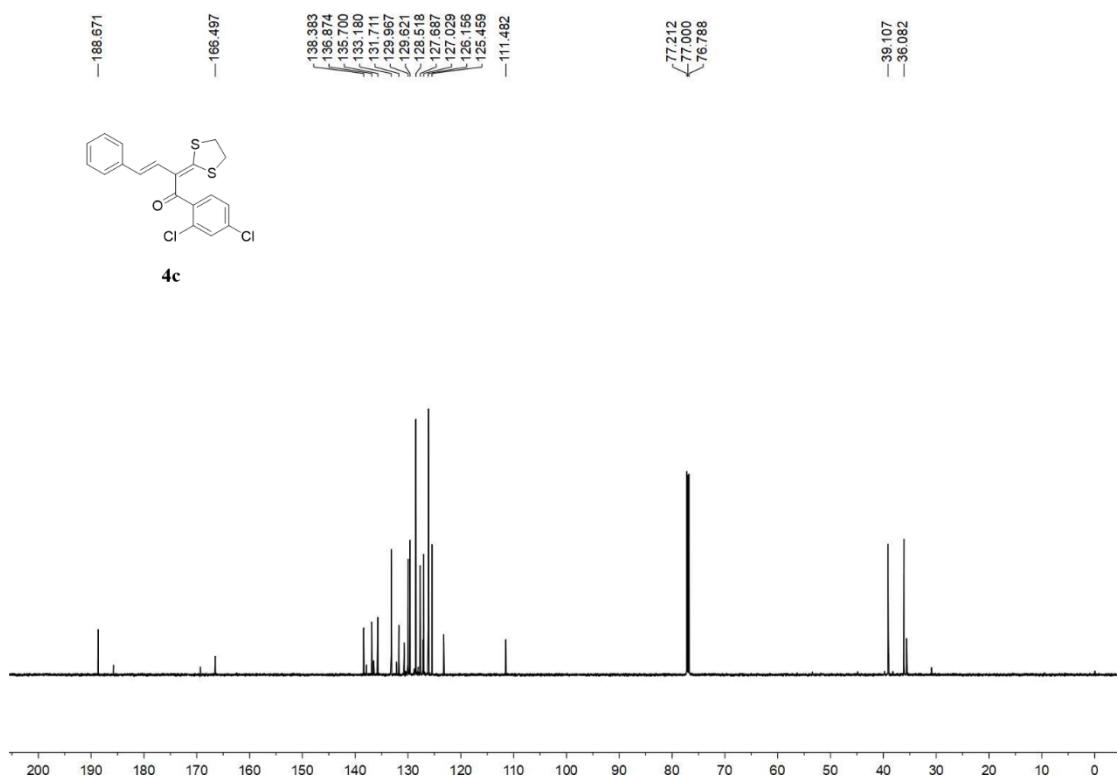
¹³C spectrum (151 MHz, CDCl₃) of compound **4b**



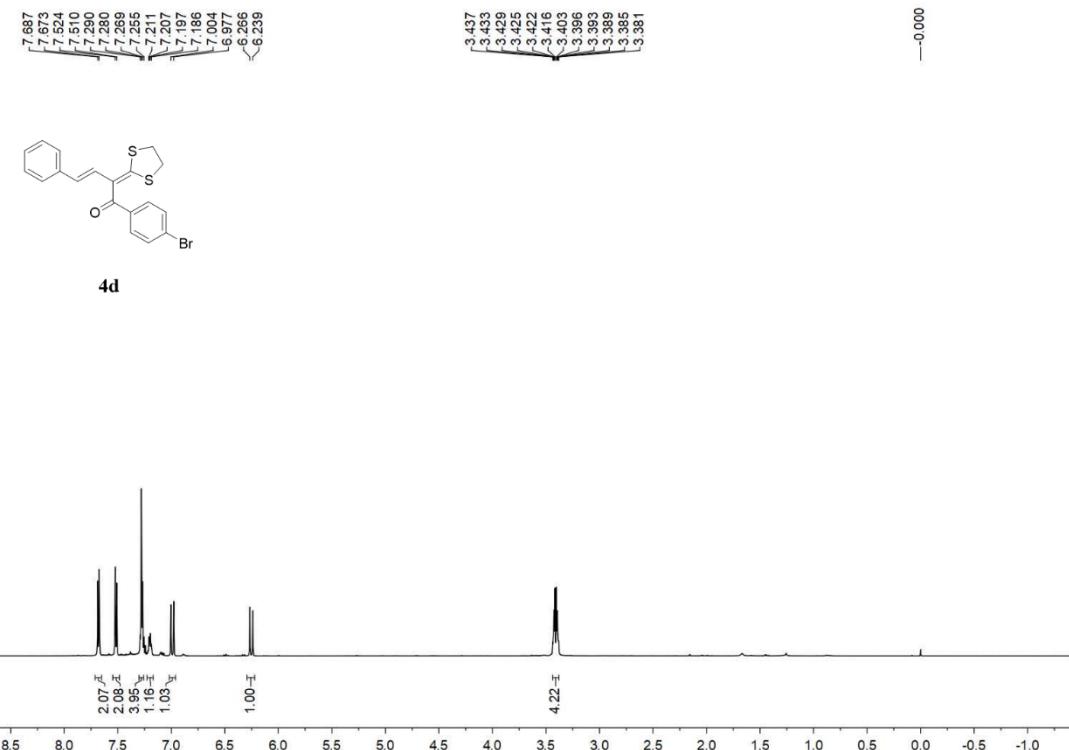
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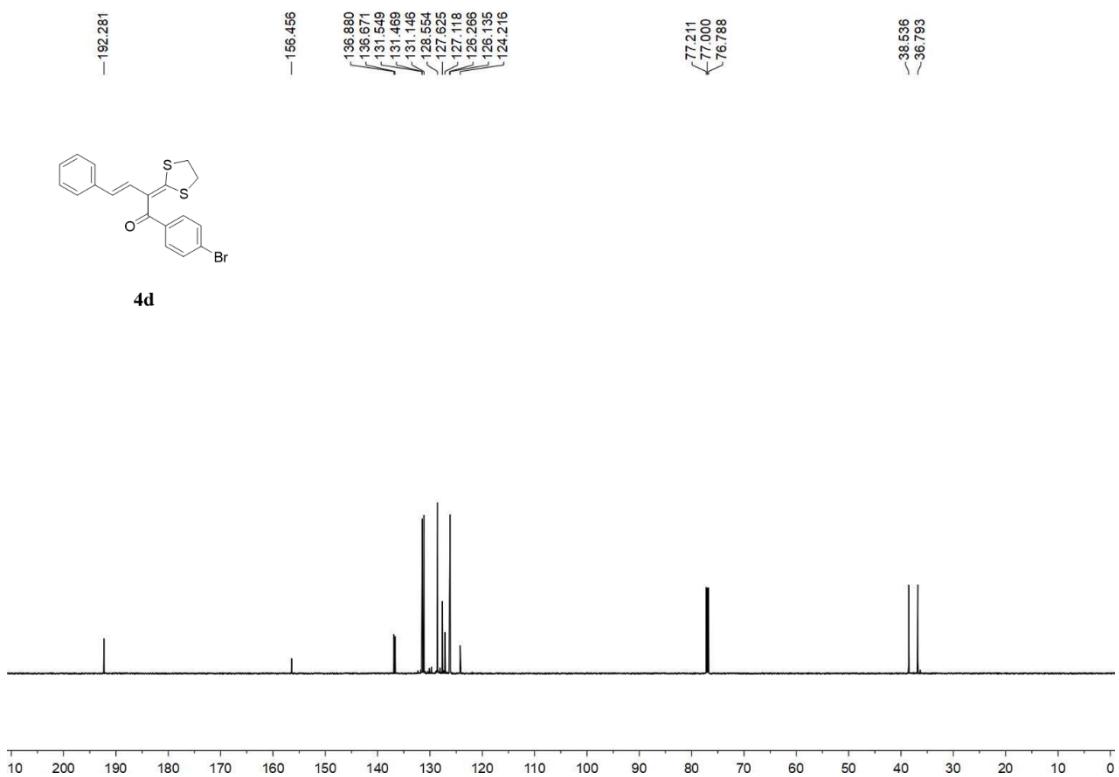
¹³C spectrum (151 MHz, CDCl₃) of compound **4c**



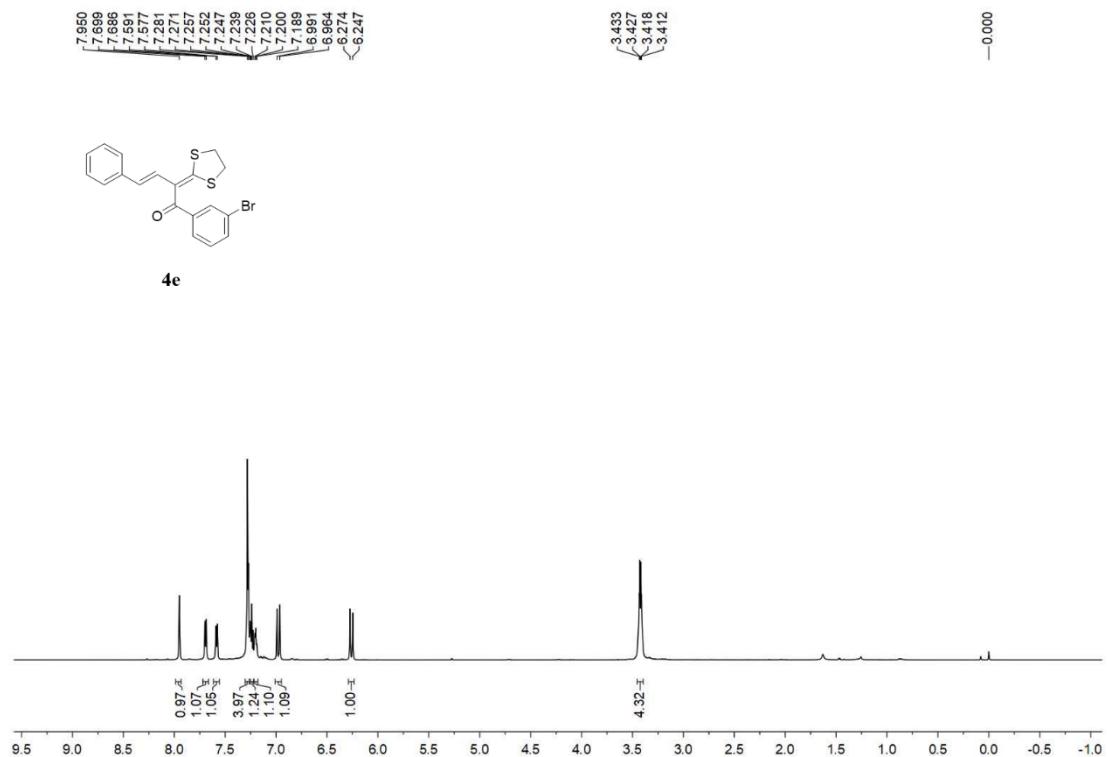
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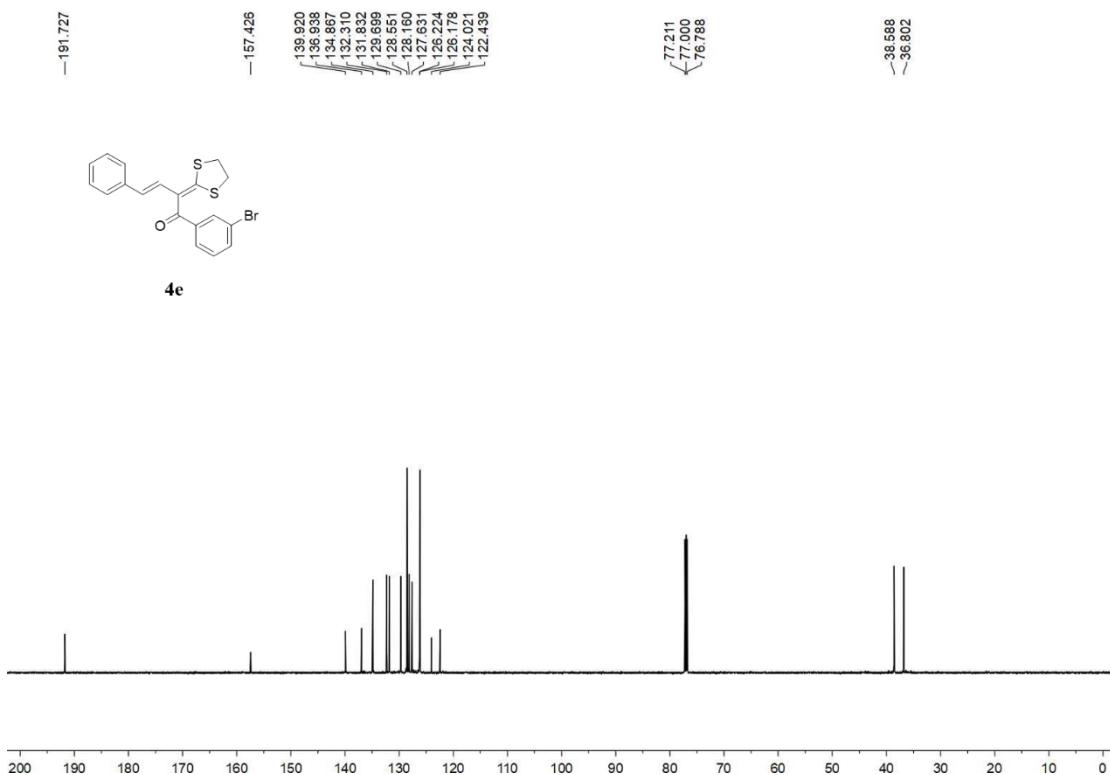
¹³C spectrum (151 MHz, CDCl₃) of compound **4d**



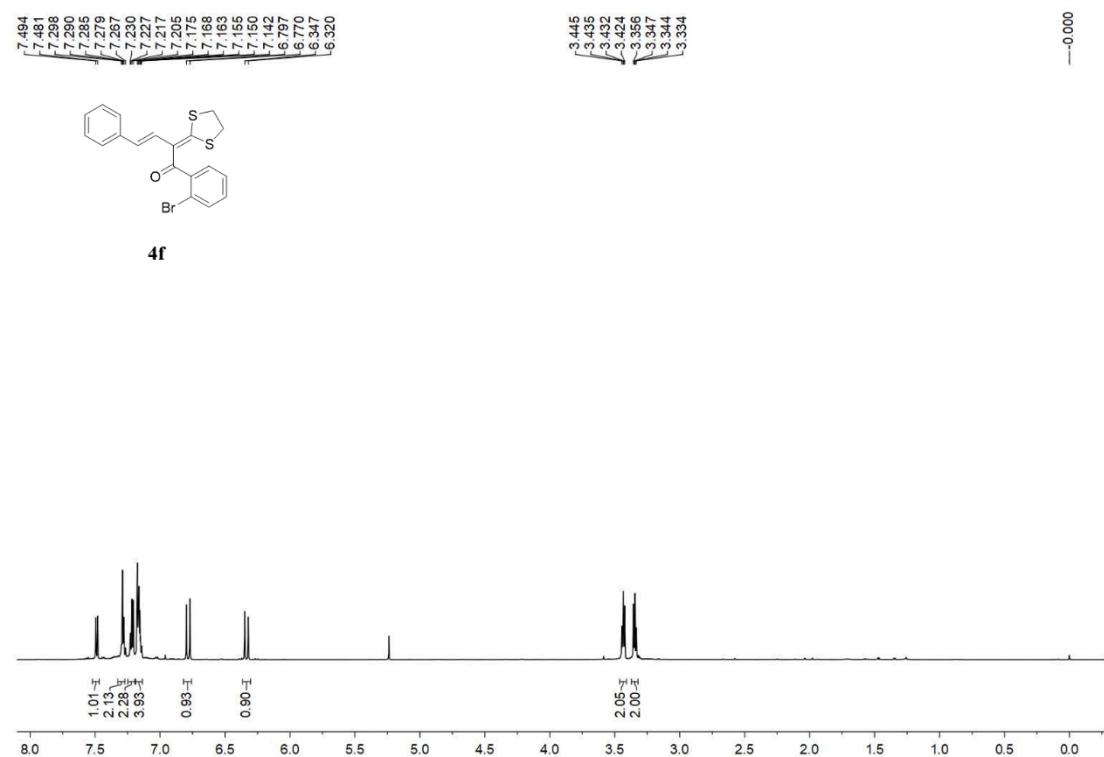
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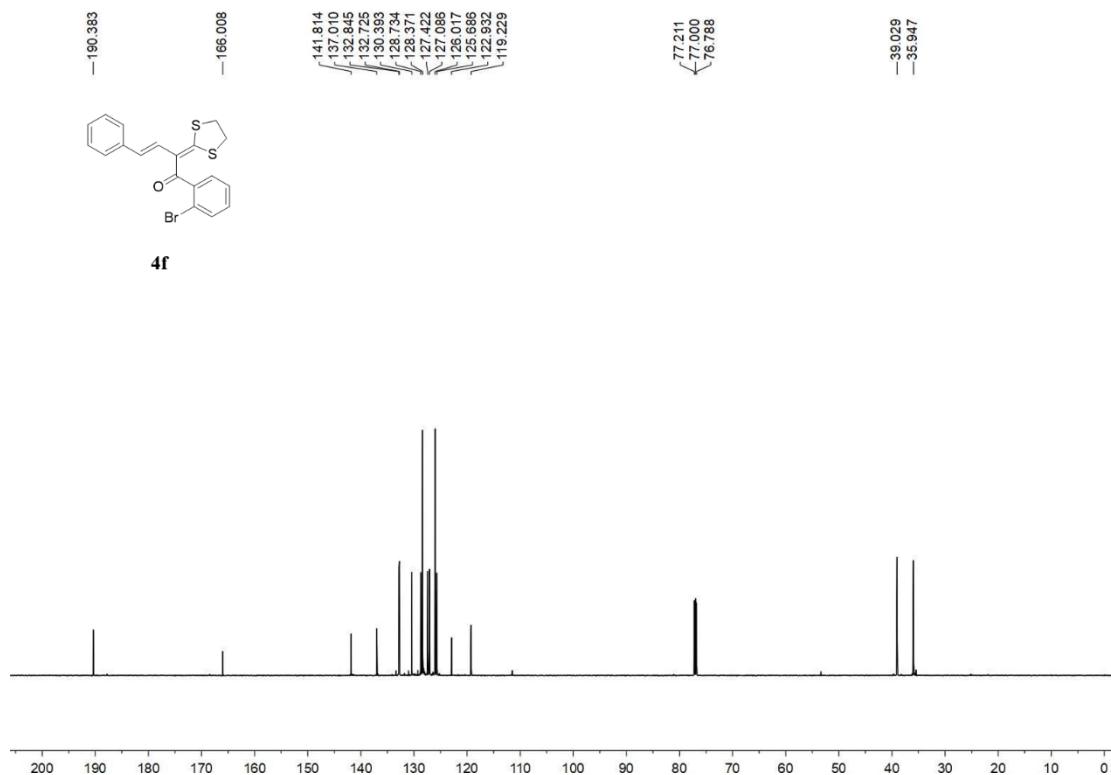
¹³C spectrum (151 MHz, CDCl₃) of compound 4e



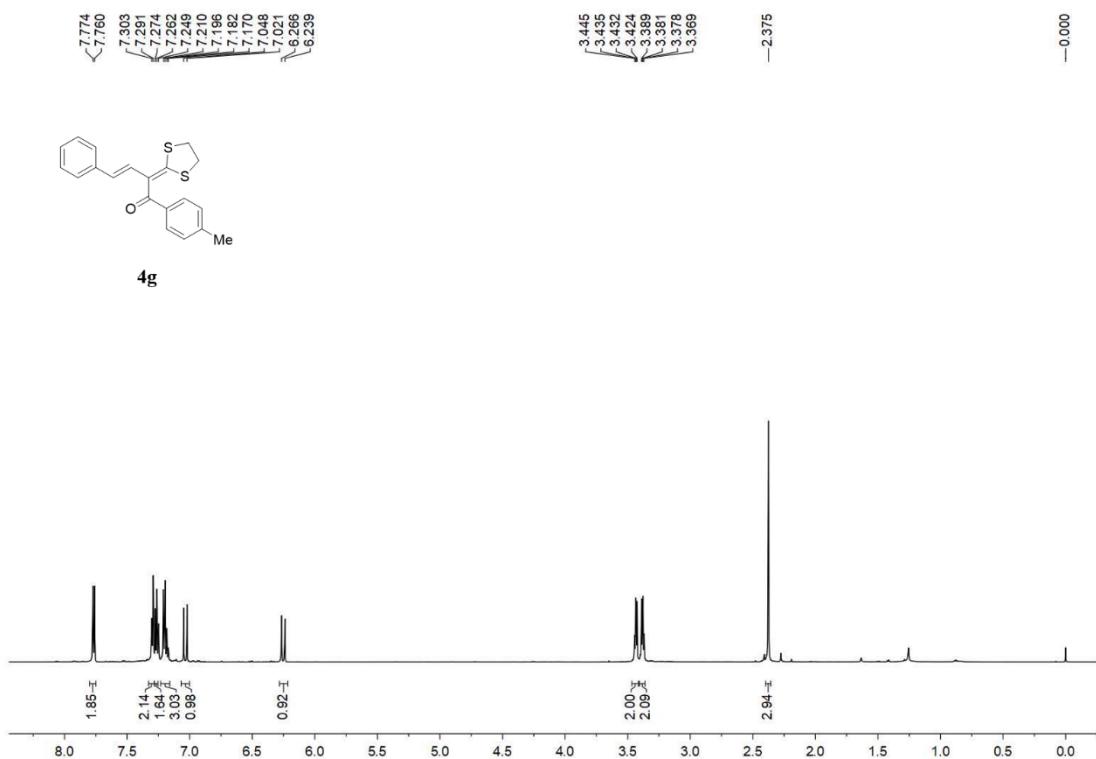
¹H spectrum (600 MHz, CDCl₃) of compound **4f**



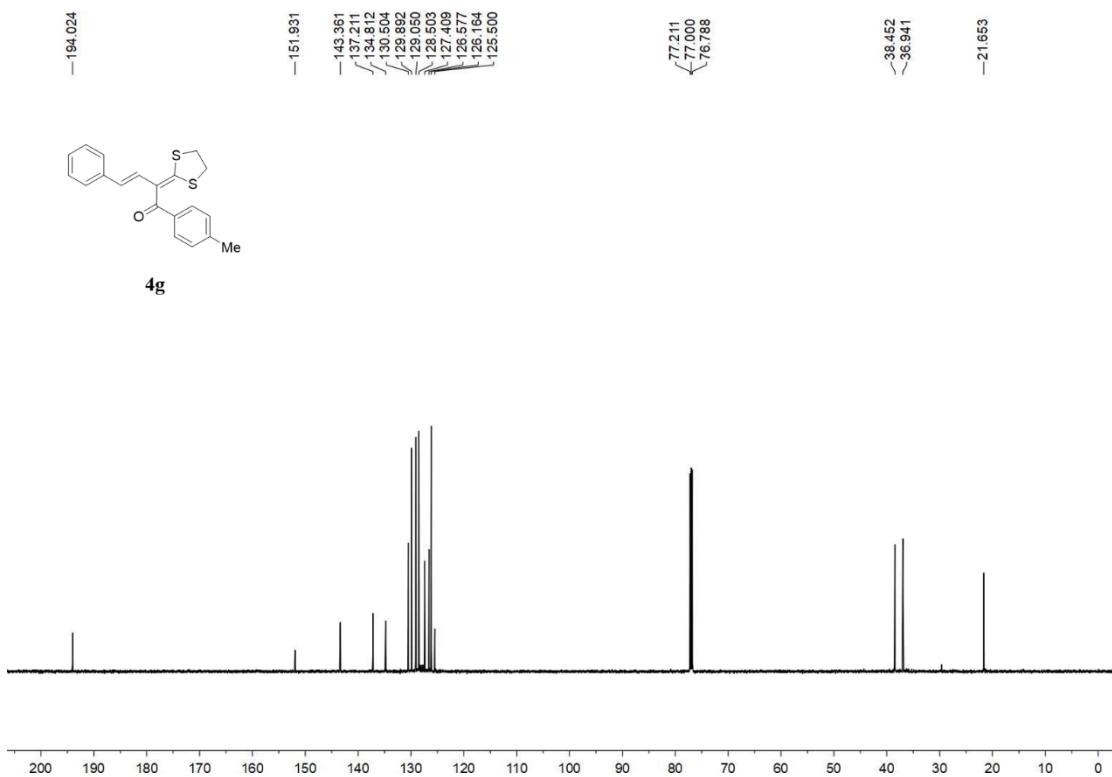
¹³C spectrum (151 MHz, CDCl₃) of compound **4f**



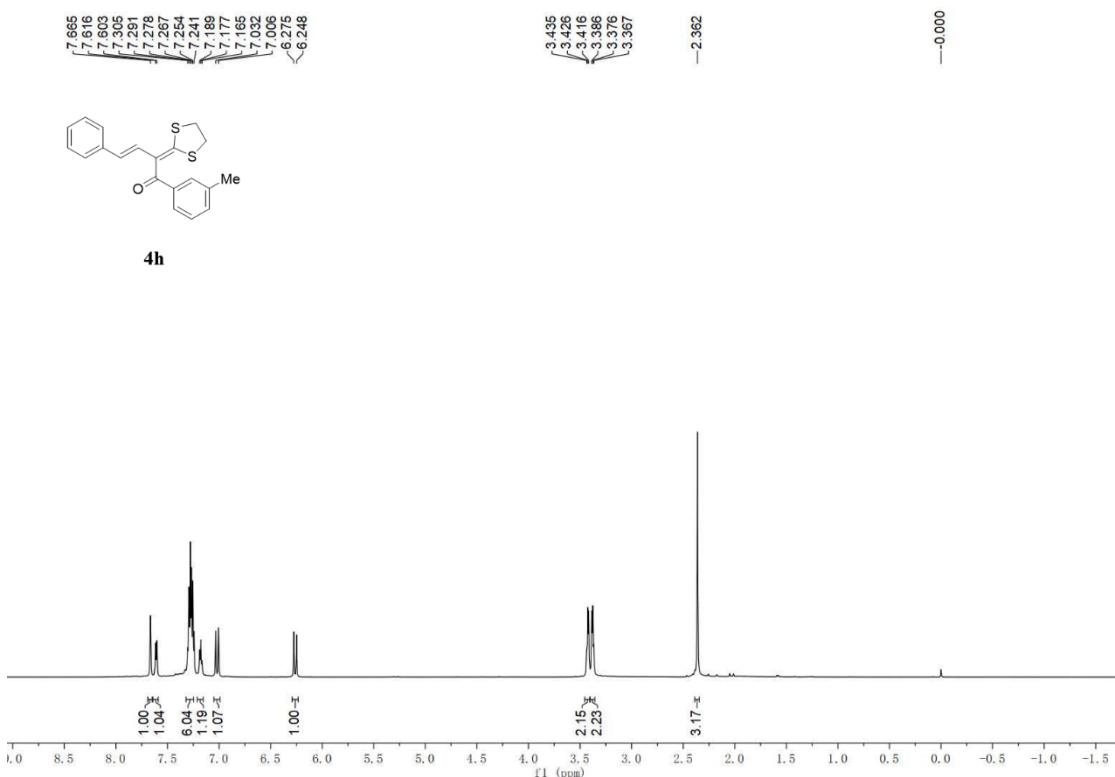
¹H spectrum (600 MHz, CDCl₃) of compound **4g**



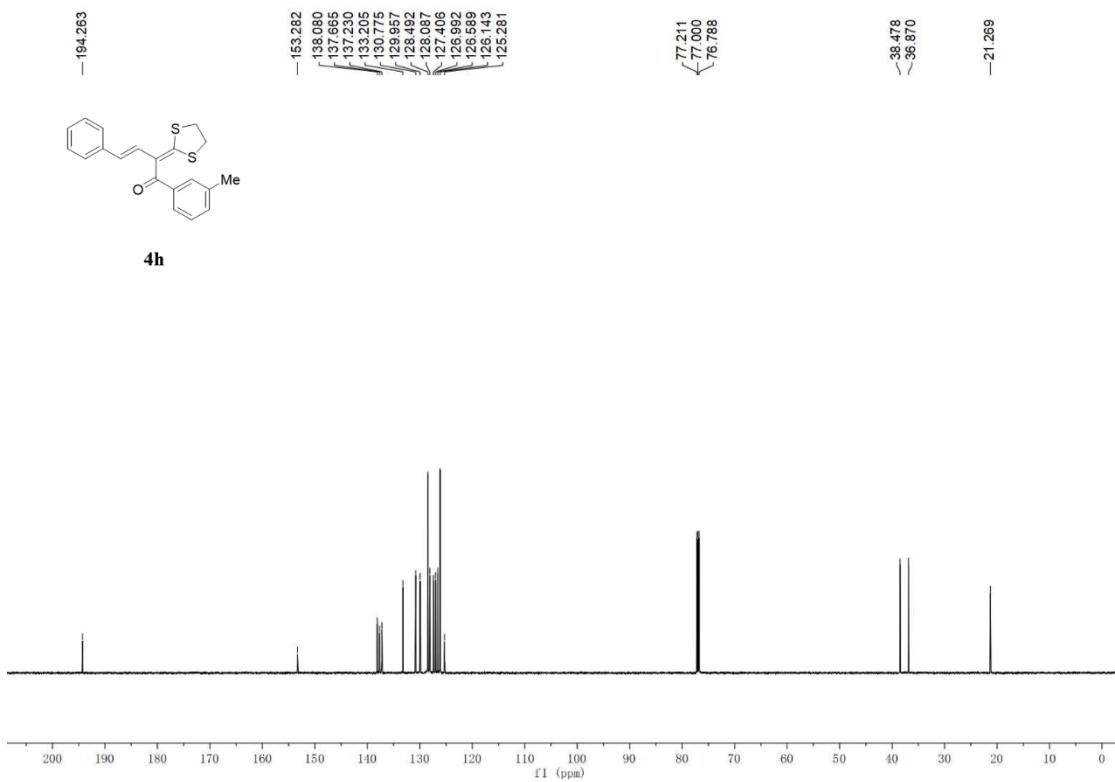
¹³C spectrum (151 MHz, CDCl₃) of compound **4g**



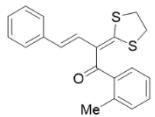
¹H spectrum (600 MHz, CDCl₃) of compound **4h**



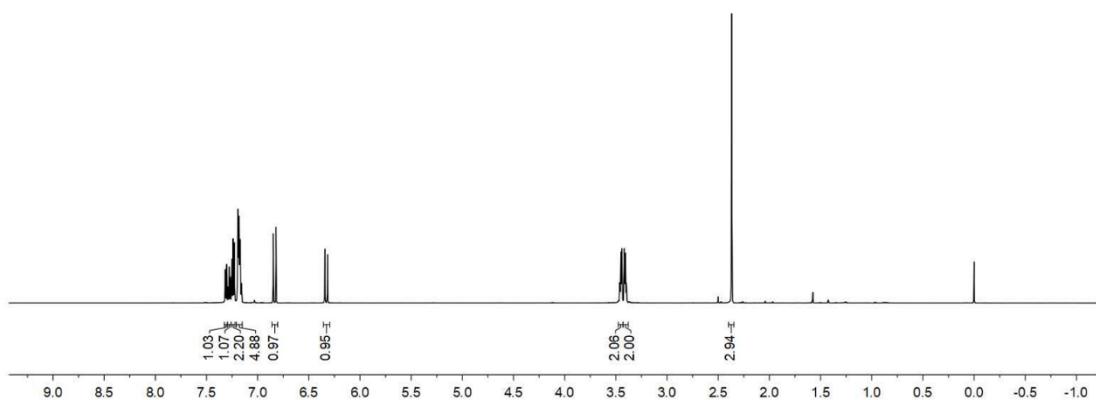
¹³C spectrum (151 MHz, CDCl₃) of compound **4h**



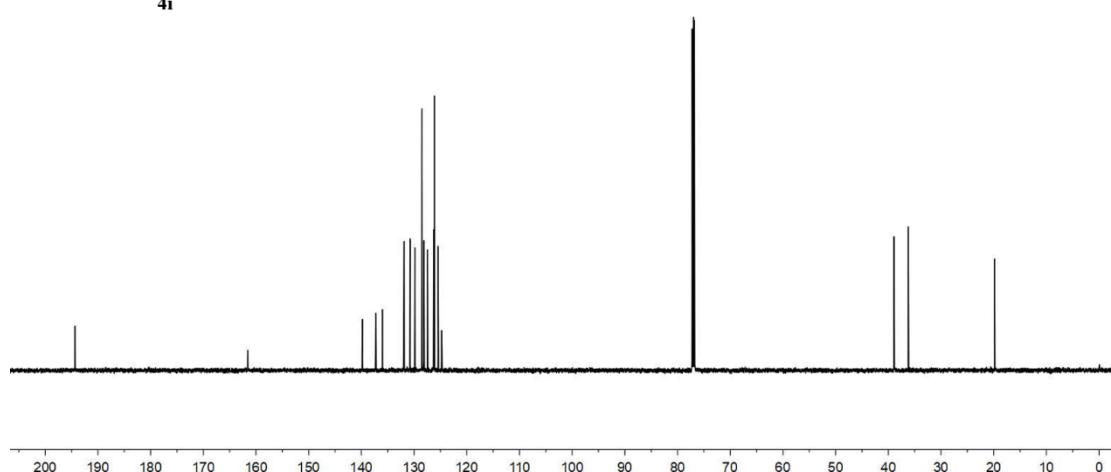
¹H spectrum (600 MHz, CDCl₃) of compound 4i



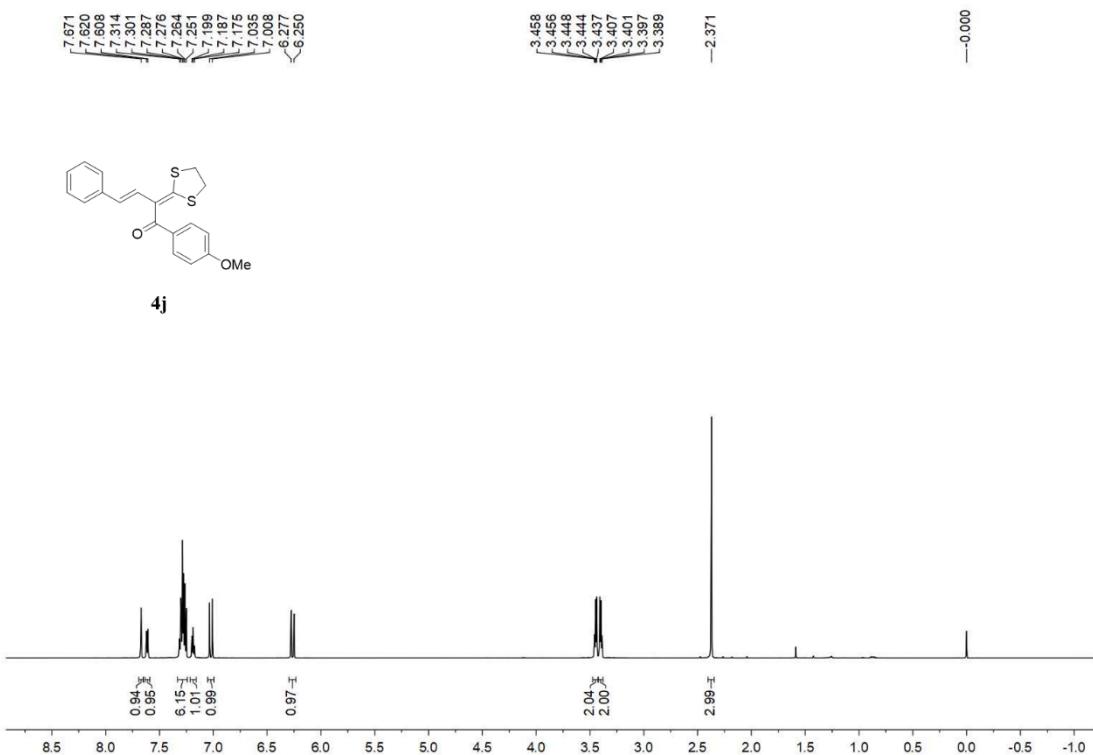
4i



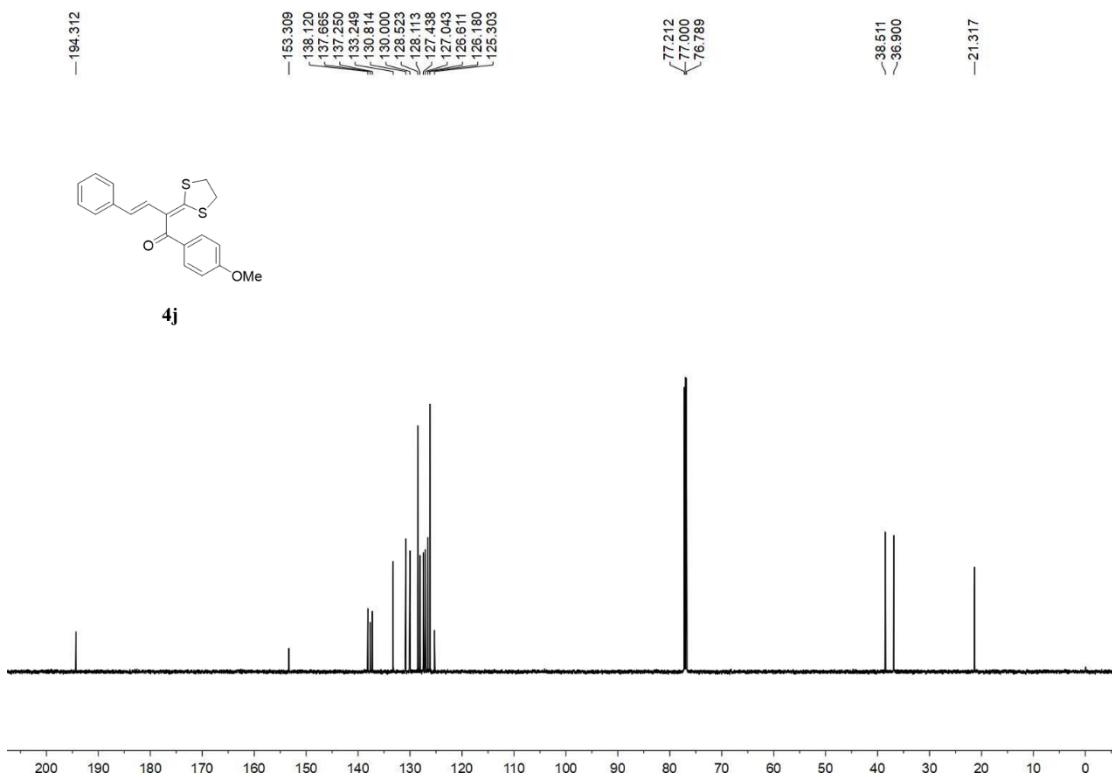
¹³C spectrum (151 MHz, CDCl₃) of compound 4i



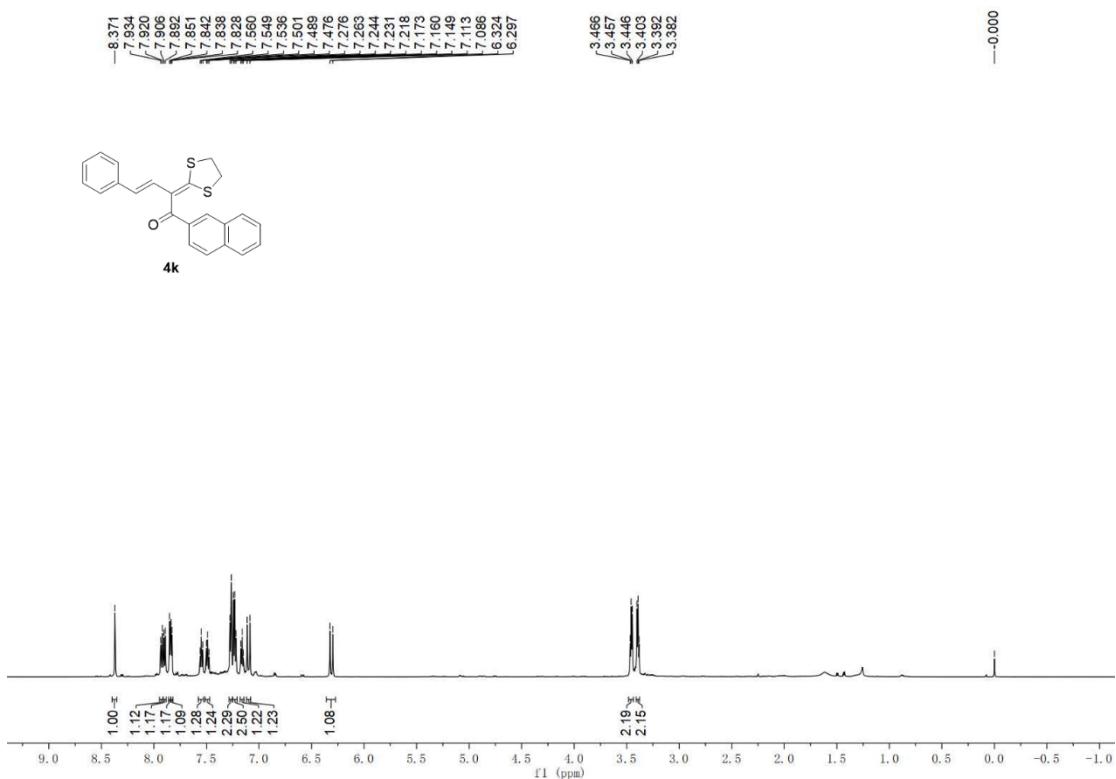
¹H spectrum (600 MHz, CDCl₃) of compound **4j**



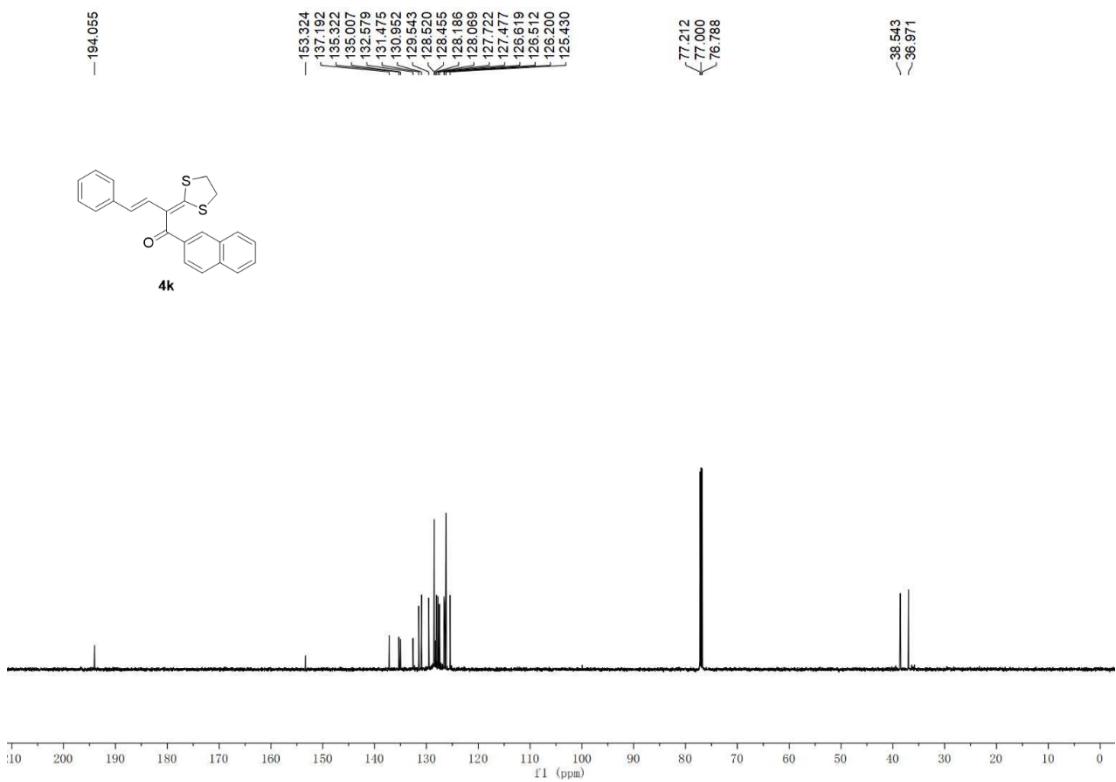
¹³C spectrum (151 MHz, CDCl₃) of compound **4j**



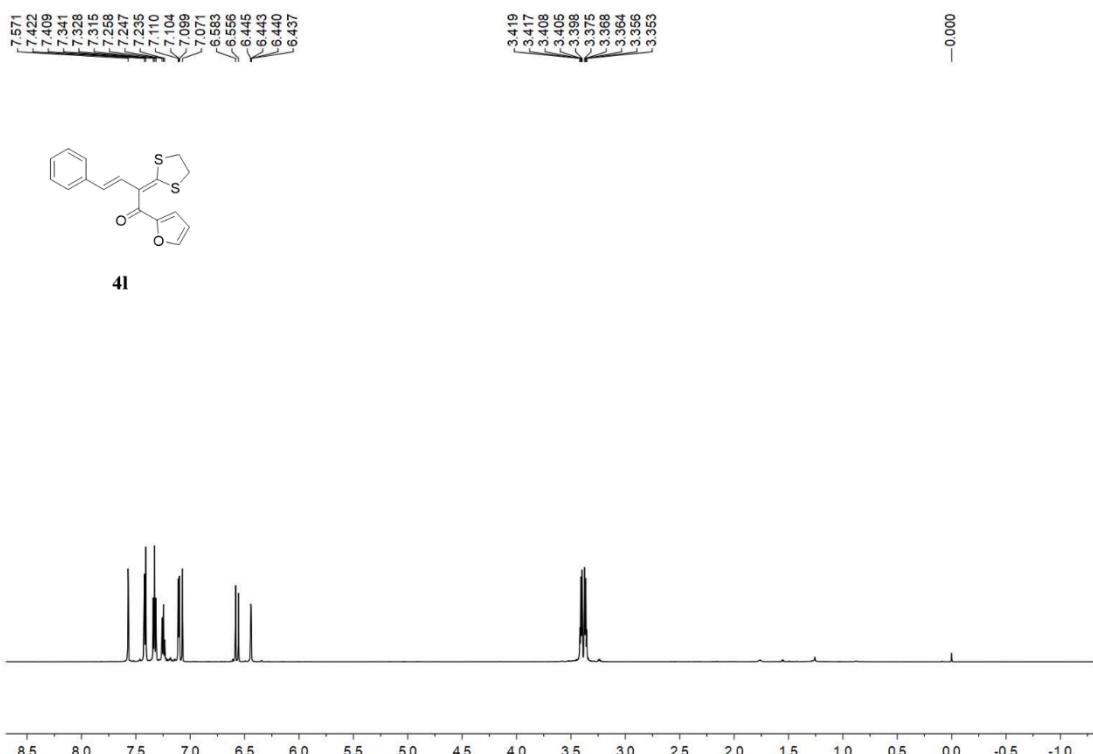
¹H spectrum (600 MHz, CDCl₃) of compound **4k**



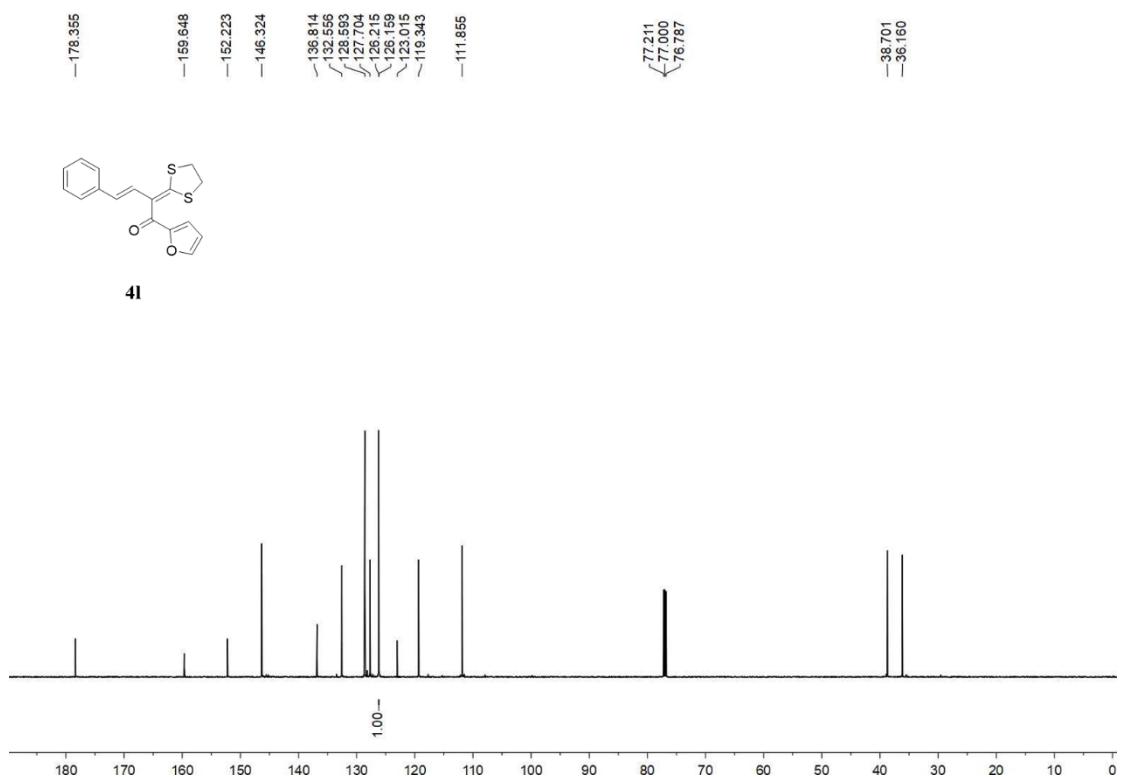
¹³C spectrum (151 MHz, CDCl₃) of compound **4k**



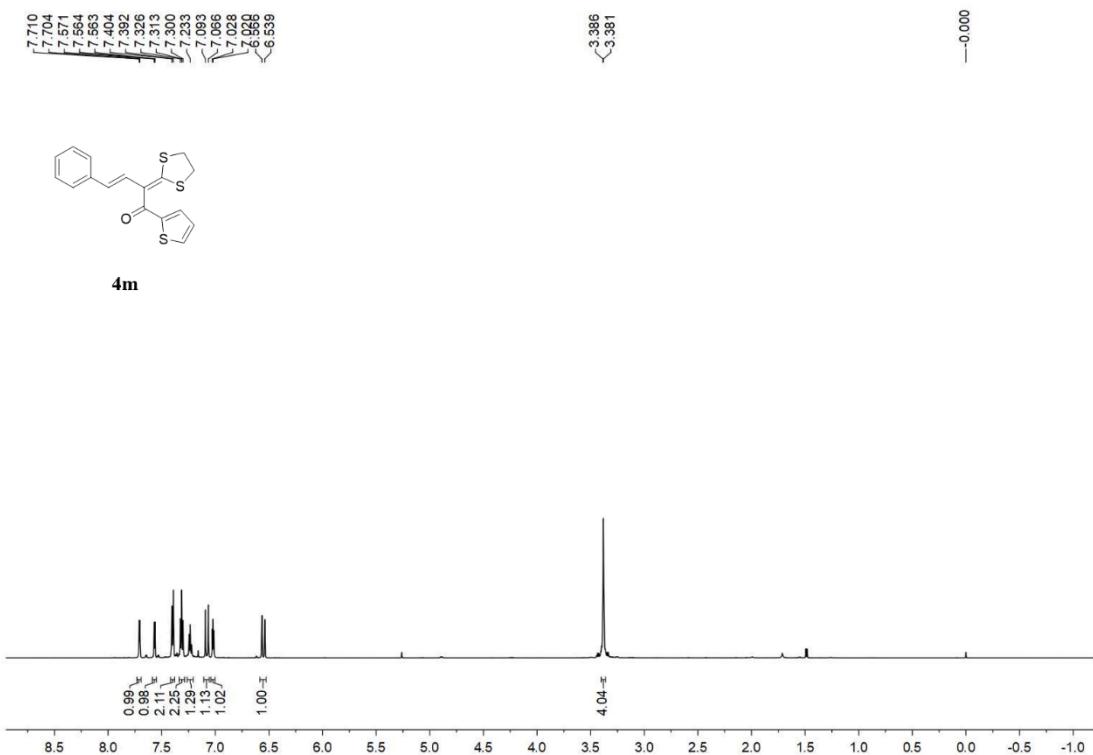
¹H spectrum (600 MHz, CDCl₃) of compound **4l**



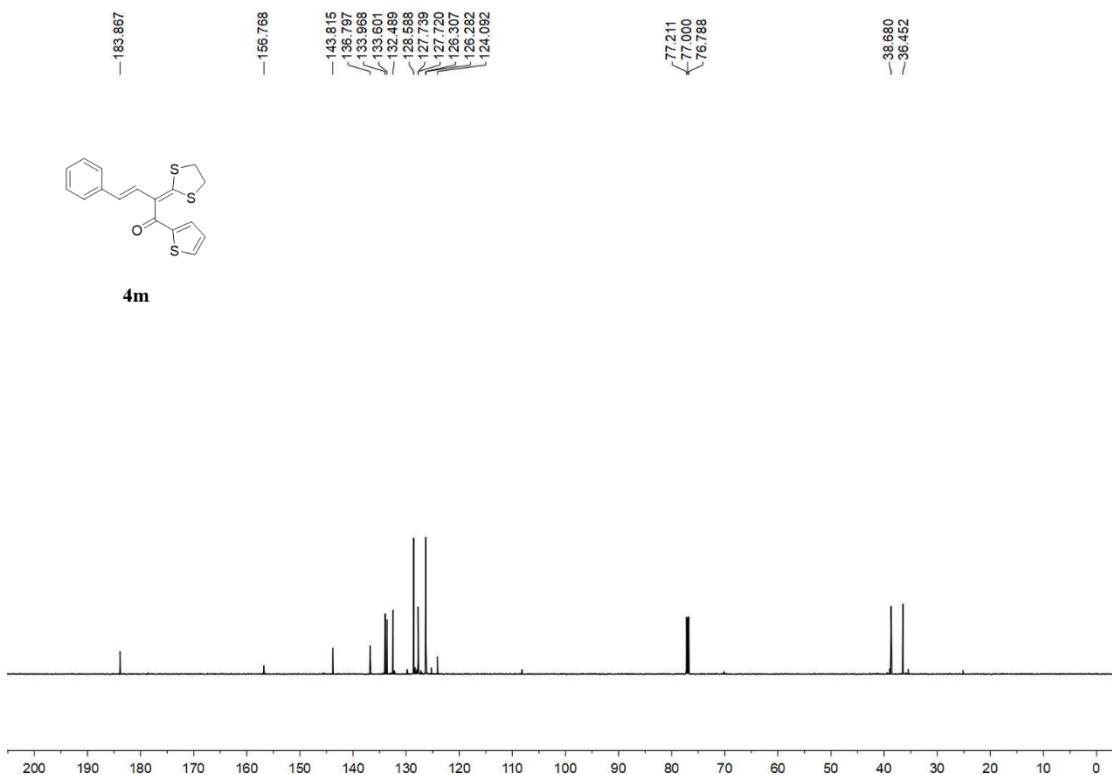
¹³C spectrum (151 MHz, CDCl₃) of compound **4l**



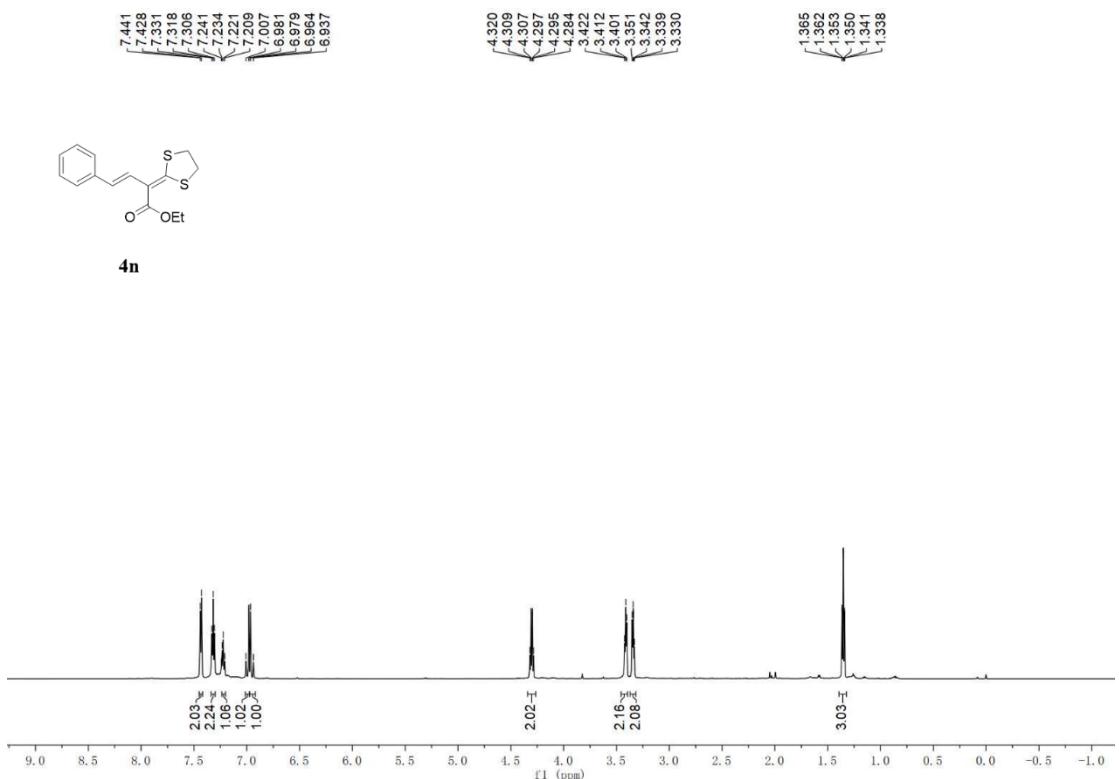
¹H spectrum (600 MHz, CDCl₃) of compound **4m**



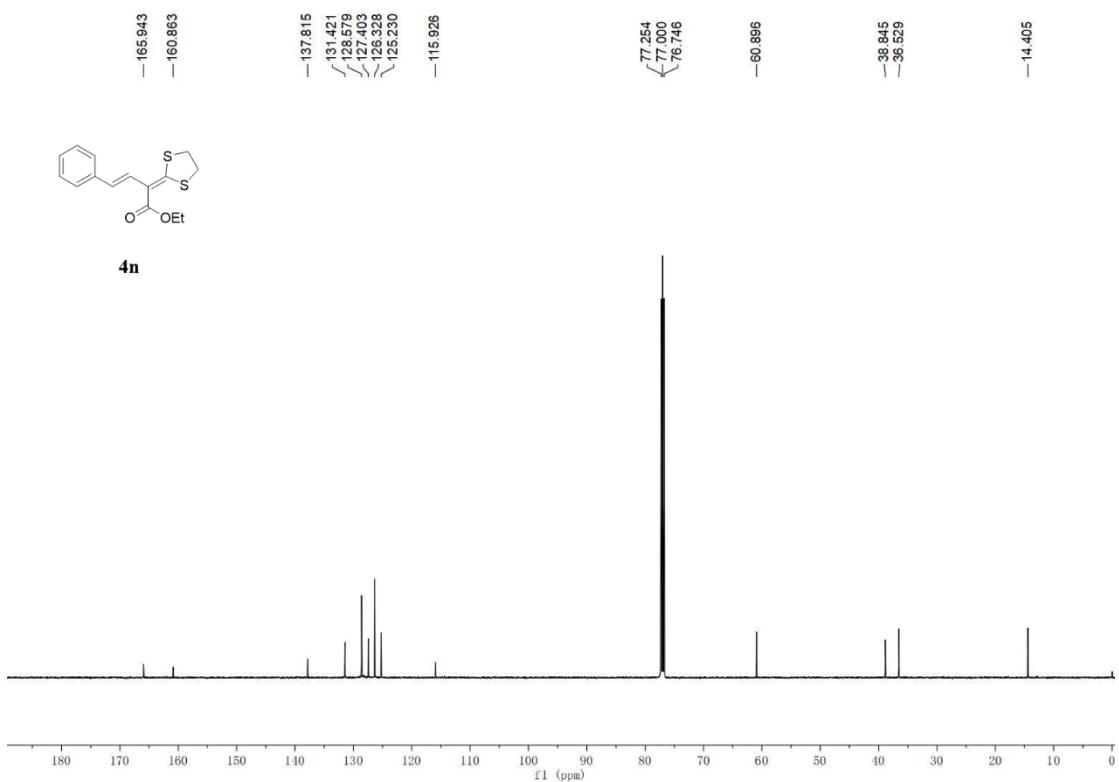
¹³C spectrum (151 MHz, CDCl₃) of compound **4m**



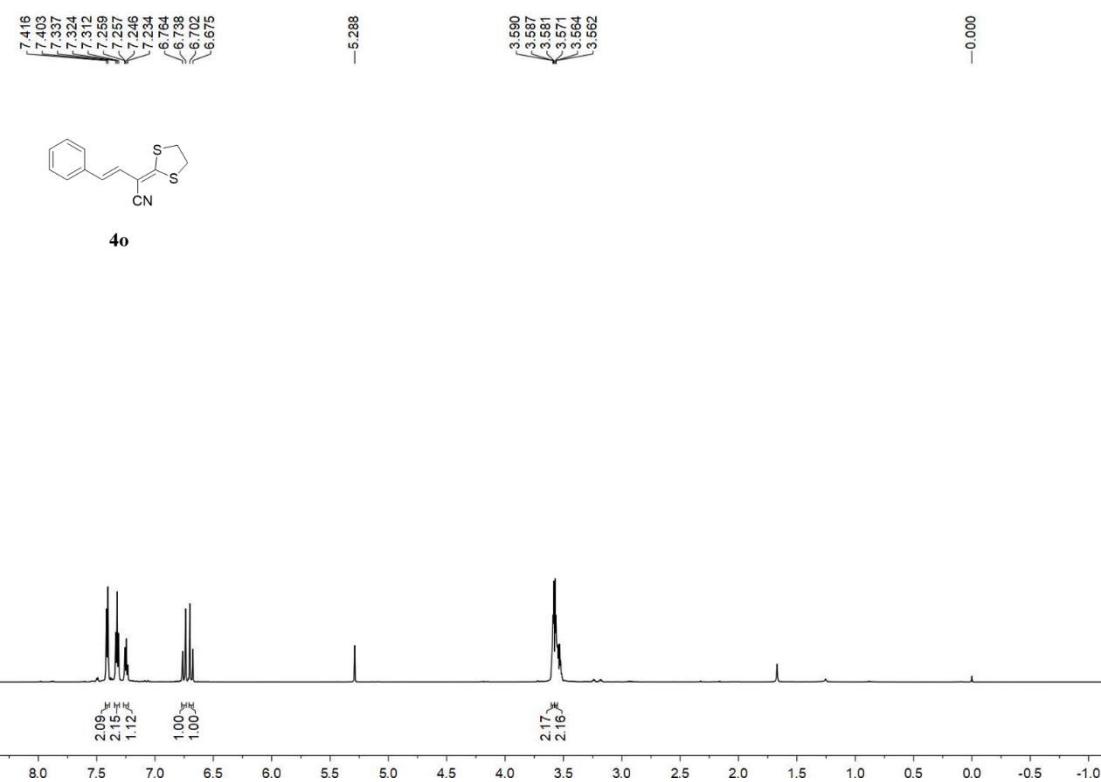
¹H spectrum (600 MHz, CDCl₃) of compound **4n**



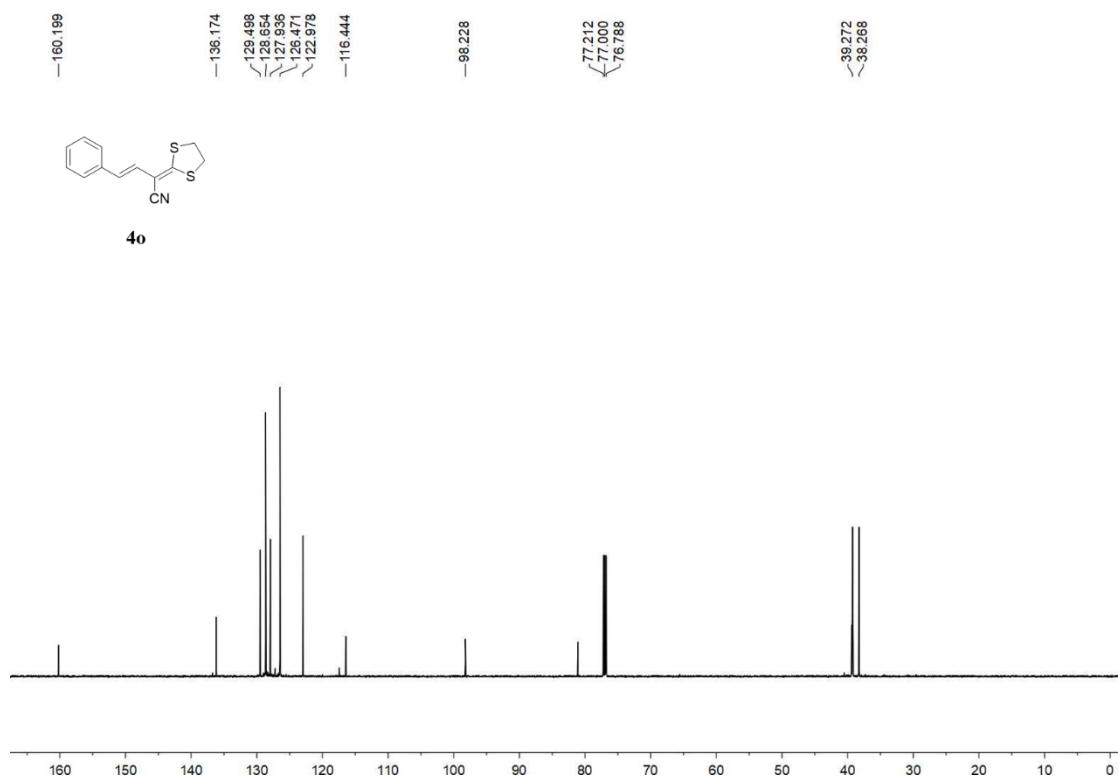
¹³C spectrum (126 MHz, CDCl₃) of compound **4n**



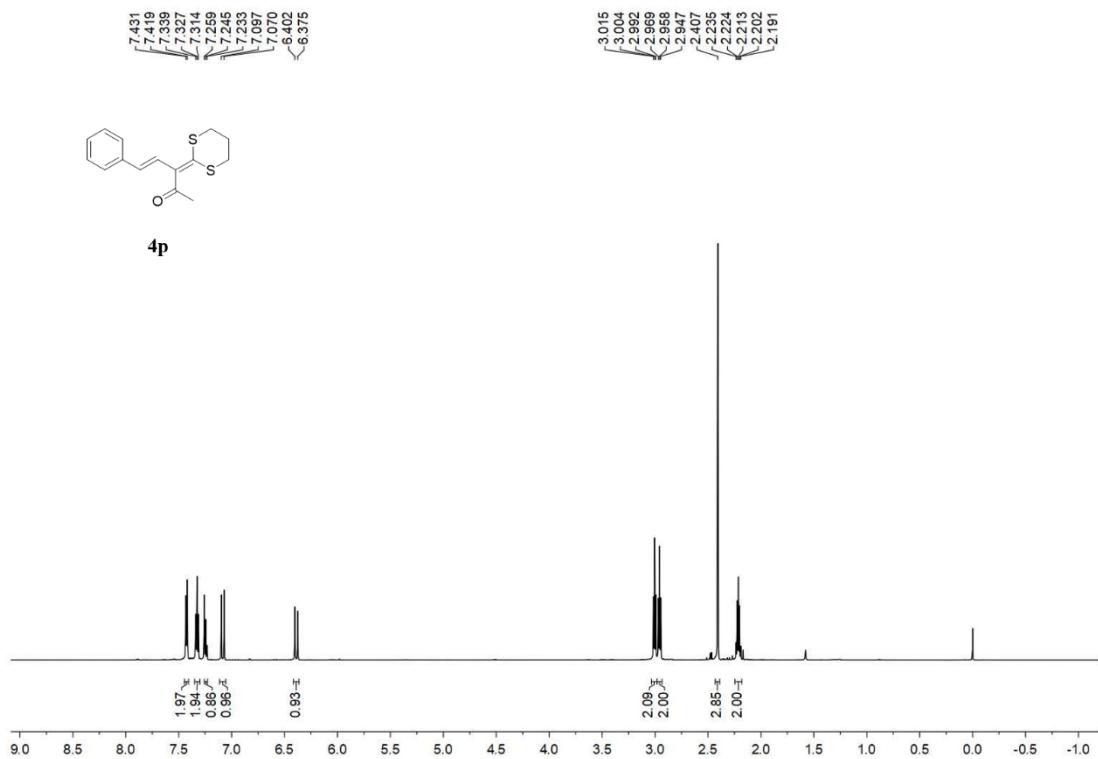
¹H spectrum (600 MHz, CDCl₃) of compound **4o**



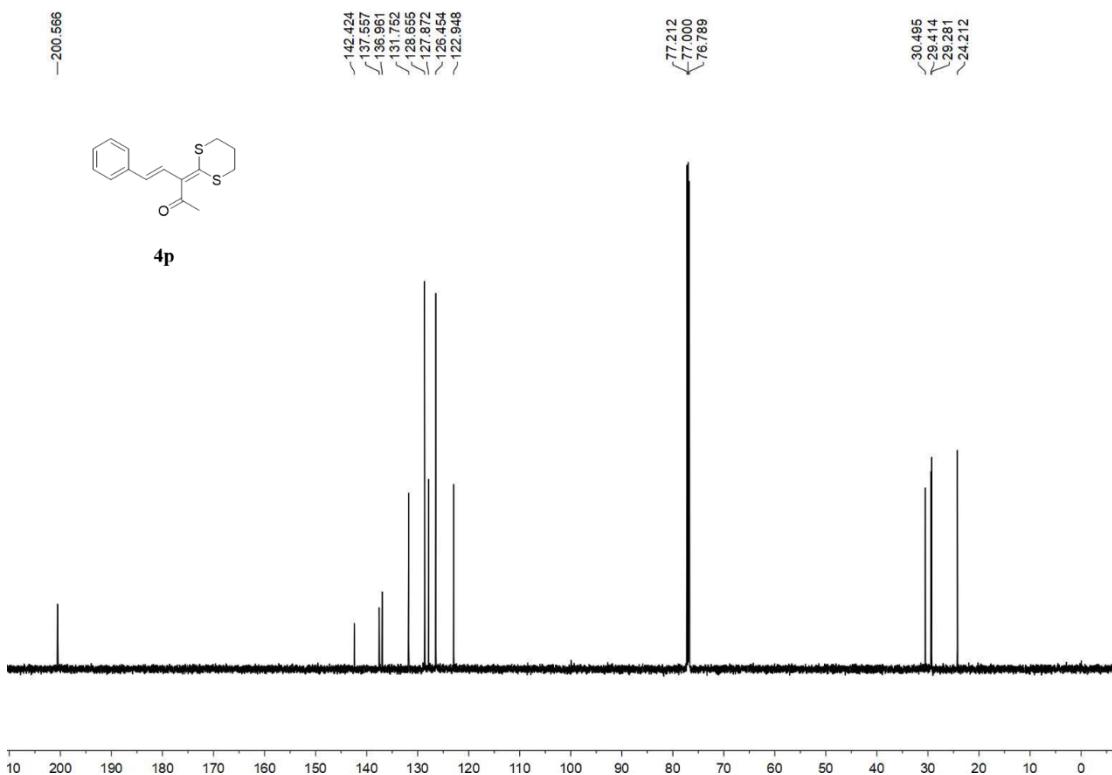
¹³C spectrum (151 MHz, CDCl₃) of compound **4o**



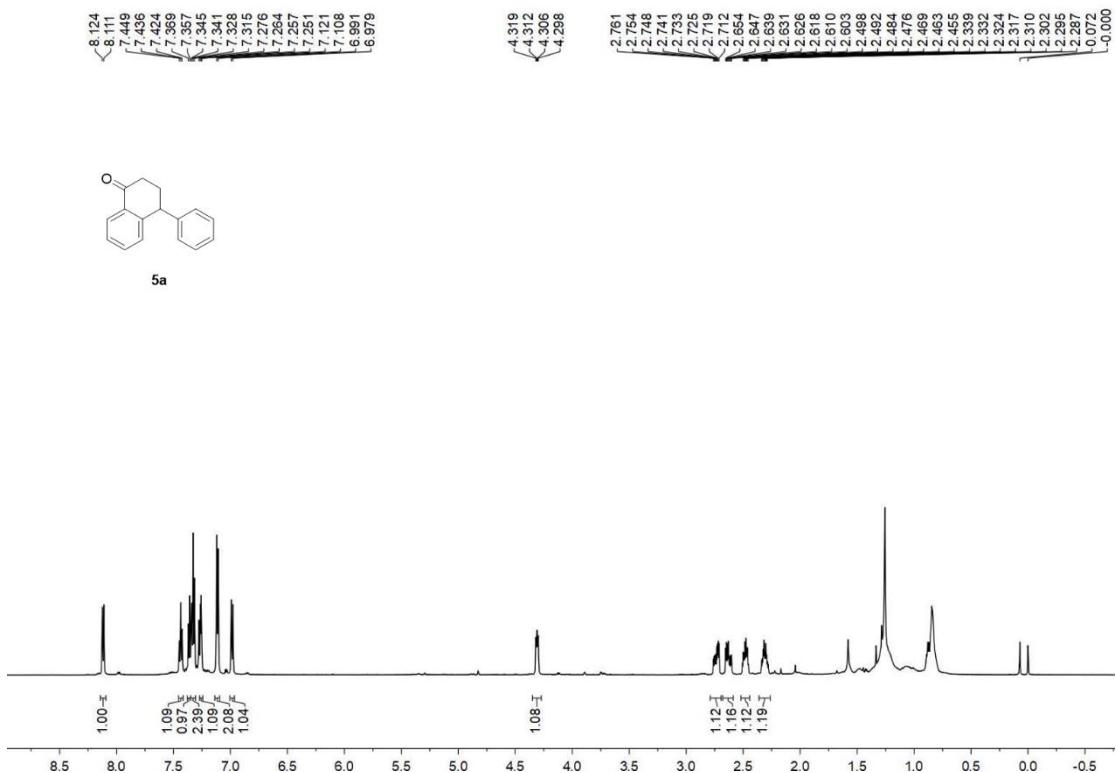
¹H spectrum (600 MHz, CDCl₃) of compound 4p



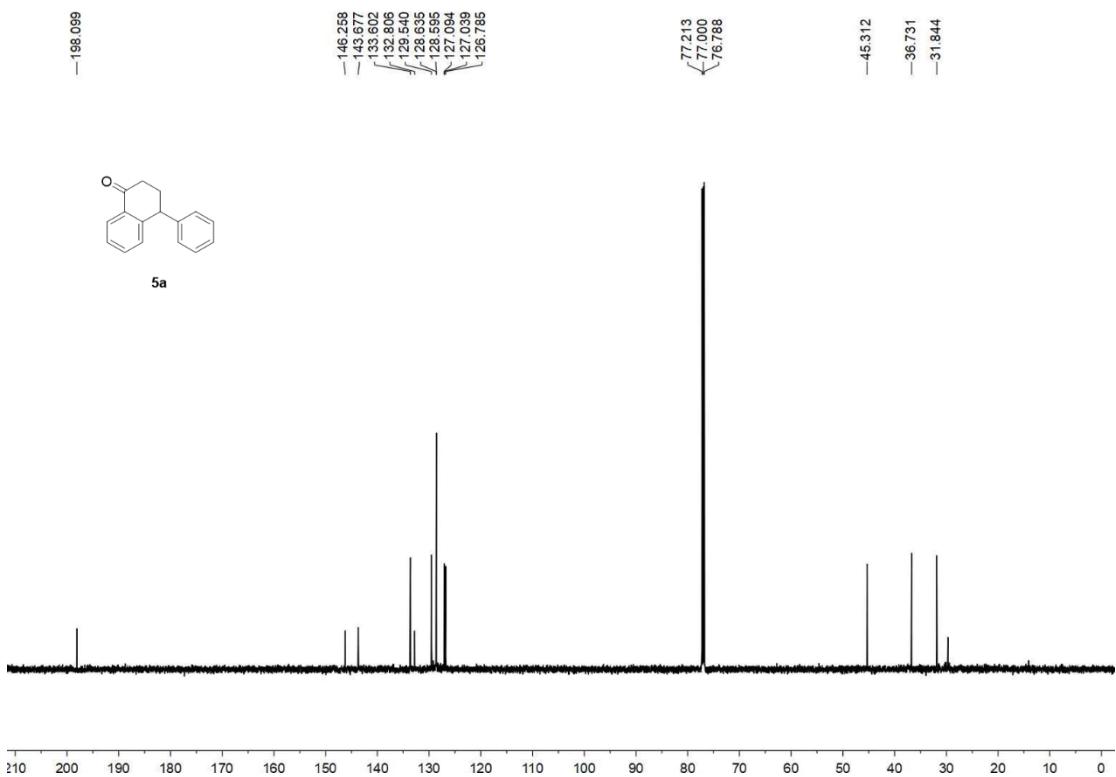
¹³C spectrum (151 MHz, CDCl₃) of compound 4p



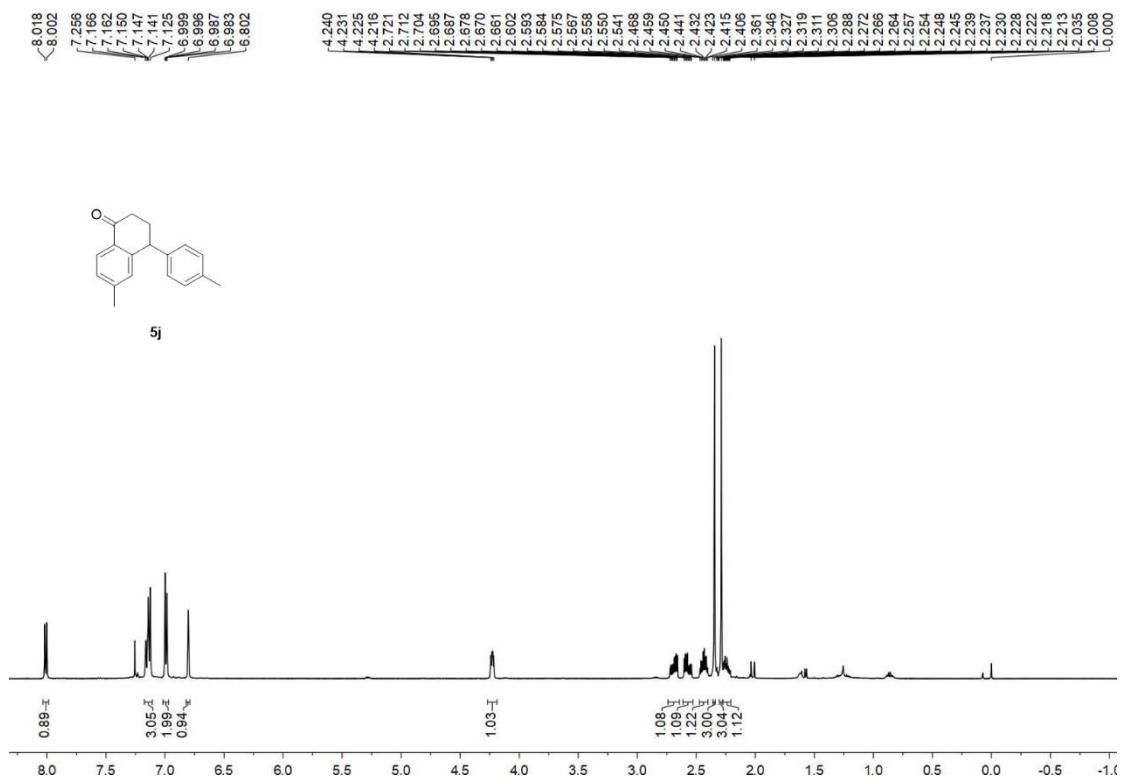
¹H spectrum (600 MHz, CDCl₃) of compound **5a**



¹³C spectrum (151 MHz, CDCl₃) of compound **5a**



¹H spectrum (600 MHz, CDCl₃) of compound **5j**



¹³C spectrum (151 MHz, CDCl₃) of compound **5j**

