

SUPPROTING INFORMATION

Carbonylative Coupling of Simple Alkanes and Alkenes Enabled by Organic Photoredox Catalysis

Ling Chen, Jing Hou,* Ming Zheng, Le-Wu Zhan, Wan-Ying Tang,* Bin-dong Li*
College of Chemical Engineering, Nanjing University of Science and Technology, Nanjing
210094, China.

E-mail: libindong@njjust.edu.cn; chmhouj@njjust.edu.cn

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1. General Information

Chemicals and solvents were purchased from commercial suppliers and used as received. ^1H NMR, ^{13}C NMR spectra were recorded on a Bruker AVANCEIII-500 (500 MHz) spectrometer. Chemical shifts were calibrated using residual undeuterated solvent as an internal reference (CDCl_3 : 7.26 ppm ^1H NMR, 77.16 ppm ^{13}C NMR). Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (doublet of doublet), brs (broad singlet). All high resolution mass spectra (HRMS) were obtained on a AB Sciex TripieTOF 4600 spectrometer. Column chromatography was carried out over 200-300 mesh silica gel. Blue LED (40 W, $\lambda_{\text{max}} = 440$ nm) purchased from Kessil was used for blue light irradiation. Alkenes (**1b**¹, **1c**², **1d**², **1e**², **1f**³, **1g**¹, **1h**¹, **1i**⁴, **1j**⁵, **1k**⁶, **1l**², **1m**¹, **1n**³, **1o**⁴, **1p**¹, **1q**⁷, **1r**⁸) and *N*-alkoxyazinium salts (**A**⁹, **B**¹⁰, **C**¹⁰, **D**¹⁰) were all prepared following reported literature protocols.

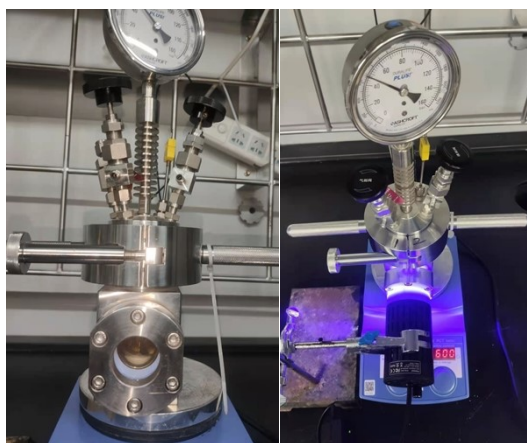


Figure S1. Devices for the photocatalytic reactions

2. General procedures

General procedure for carbonylation of alkanes: a 5 mL vial equipped with a magnetic stir bar was added 4CzIPN (3.2 mg, 0.004 mmol, 2 mol%), 4-cyano-1-isopropoxy-pyridin-1-ium trifluoromethanesulfonate (93.6 mg, 0.3 mmol, 1.5 equiv). Subsequently, corresponding alkene (0.2 mmol, 1.0 equiv), cyclohexane (1.5 mL) and 1,2-dichloromethane (1.5 mL) was added through injection port. The vials were placed into an autoclave with one inserted quartz-glass windows. After the autoclave was flushed three times, it was filled with 20 atm of CO and then irradiated with blue LED (40 W, $\lambda_{\text{max}} = 440$ nm) at rt for 12 h. Upon completion of the reaction, the pressure was carefully released and the solvent was removed under reduced pressure. The products were purified by flash chromatography (hexanes/EtOAc = 50/1).

3. Complementary Reaction Optimization Data

Table S1 Photocat. evaluation^a

Table S1 Photocatalytic reaction

Reaction scheme showing the photocatalytic reaction of 1a (1-phenylprop-1-ene) and 2a (cyclohexane) with CO (50 bar) to form 3a (1-(cyclohexylidene)-2-phenylprop-1-ene). The reaction conditions are: Photocat. (2 mol%), A (1.5 equiv), DCE (2.0 mL), Blue LED, rt, 12 h.

entry	Photocat.	Yield (%) ^b
1	4CzIPN	55
2	EosinY	13
3	Ru(bpy) ₃ Cl ₂	16
4	Ru(bpy) ₃ [PF ₆] ₂	16
5	Ir(ppy) ₂ (dtbpy)PF ₆	33
6	Ir(ppy) ₃	28
7	[Ir(dF(CF ₃)ppy) ₂](dtbbpy)]PF ₆	37

^aStandard conditions: **1a** (0.2 mmol), **A** (1.5 equiv), Photocat. (2 mol%), **2a** (1 mL), DCE (2 mL) and CO (50 bar) at rt under LED irradiation, 12 h. ^bIsolated yields.

Table S2 Screening of solvent^a

Reaction scheme for the synthesis of **3a** from **1a** and **2a**:

1a + **2a** + CO (50 bar) $\xrightarrow[\text{Blue LED, rt, 12 h}]{\text{4CzIPN (2 mol\%), A (1.5 equiv), Solvent (2.0 mL)}}$ **3a**

Chemical structures: **1a** (1-phenylprop-1-ene), **2a** (cyclohexane), **3a** (1-(cyclohexylidene)-2-phenylprop-1-ene).

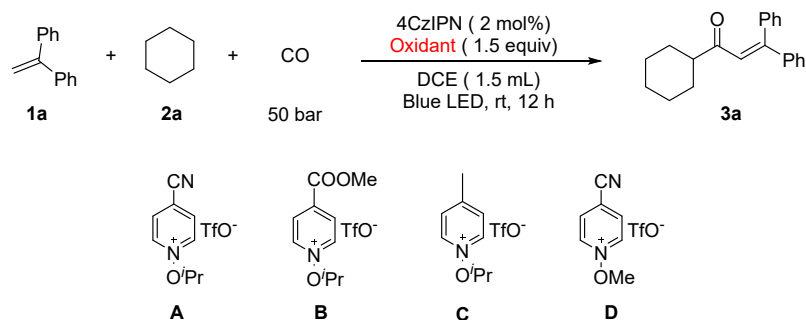
entry	Solvent	Yield (%) ^b
1	EA	24
2	MeCN	10
3	Acetone	30
4	DCE	55
5	DMSO	3
6	DCM	50
7	CHCl ₃	14

^aStandard conditions: **1a** (0.2 mmol), **A** (1.5 equiv), 4CzIPN (2 mol%), **2a** (1 mL), Solvent (2 mL) and CO (50 bar) at rt under LED irradiation, 12 h. ^bIsolated yields.

Table S3 Screening of the solvent ratio^a

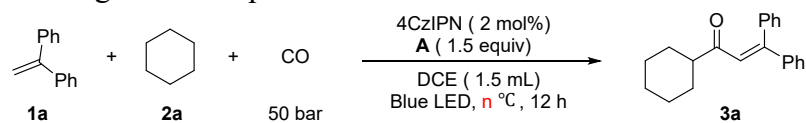
entry	2a (n mL)	DCE (m mL)	Yield (%) ^b
1	1	3	38
2	0.5	2.5	21
3	1	2	55
4	1.5	1.5	71
5	2	1	54

^aStandard conditions: **1a** (0.2 mmol), **A** (1.5 equiv), 4CzIPN (2 mol%), **2a** (n mL), DCE (m mL) and CO (50 bar) at rt under LED irradiation, 12 h. ^bIsolated yields.

Table S4 Screening of the oxidant^a

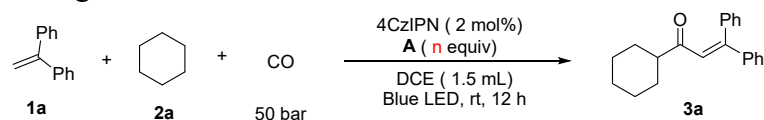
entry	Oxidant	Yield (%) ^b
1	A	71
2	B	47
3	C	32
4	D	53
5	K ₂ S ₂ O ₈	NR
6	(NH ₄) ₂ S ₂ O ₈	NR

^aStandard conditions: **1a** (0.2 mmol), Oxidant (1.5 equiv), 4CzIPN (2 mol%), **2a** (1.5 mL), DCE (1.5 mL) and CO (50 bar) at rt under LED irradiation, 12 h. ^bIsolated yields. NR= no reaction.

Table S5 Screening of the temperature^a

entry	Temperature (°C)	Yield (%) ^b
1	rt	71
2	50	67
3	70	65

^aStandard conditions: **1a** (0.2 mmol), **A** (n equiv), 4CzIPN (2 mol%), **2a** (1.5 mL), DCE (1.5 mL) and CO (50 bar) at n °C under LED irradiation, 12 h. ^bIsolated yields.

Table S6 Screening of the amount of **A**^a

entry	A (n equiv)	Yield (%) ^b
1	1.0	69
2	1.5	71
3	2.0	60

^aStandard conditions: **1a** (0.2 mmol), **A** (n equiv), 4CzIPN (2 mol%), **2a** (1.5 mL), DCE (1.5 mL) and CO (50 bar) at rt under LED irradiation, 12 h. ^bIsolated yields.

Table S7 Screening of the concentration^a

<chem>c1ccccc1C=C(c2ccccc2)c3ccccc3</chem> (1a) + <chem>C1CCCCC1</chem> (2a) + CO			$\xrightarrow[\text{DCE (1.5 mL), Blue LED, rt, 12 h}]{\text{4CzIPN (2 mol\%), A (1.5 equiv)}}$	<chem>c1ccccc1C(=O)C=C(c2ccccc2)c3ccccc3</chem> (3a)
	50 bar			
entry	1a (mmol)	Yield (%) ^b		
1	0.1	69		
2	0.2	71		
3	0.3	60		

^aStandard conditions: **1a** (n mmol), **A** (1.5 equiv), 4CzIPN (2 mol%), **2a** (1.5 mL), DCE (1.5 mL) and CO (50 bar) at rt under LED irradiation, 12 h. ^bIsolated yields.

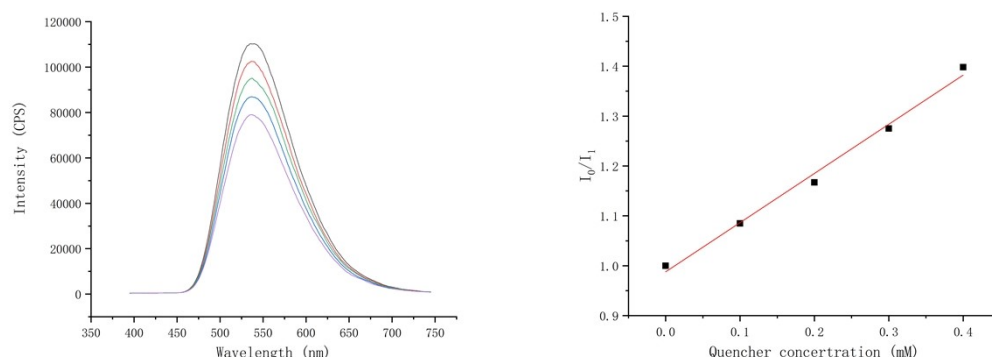
Table S8 Screening of the CO pressure^a

<chem>c1ccccc1C=C(c2ccccc2)c3ccccc3</chem> (1a) + <chem>C1CCCCC1</chem> (2a) + CO			$\xrightarrow[\text{DCE (1.5 mL), Blue LED, rt, 12 h}]{\text{4CzIPN (2 mol\%), A (1.5 equiv)}}$	<chem>c1ccccc1C(=O)C=C(c2ccccc2)c3ccccc3</chem> (3a)
	n bar			
entry	CO pressure (bar)	Yield (%) ^b		
1	10	55		
2	20	72		
3	50	71		

^aStandard conditions: **1a** (0.2 mmol), **A** (1.5 equiv), 4CzIPN (2 mol%), **2a** (1.5 mL), DCE (1.5 mL) and CO (n bar) at rt under LED irradiation, 12 h. ^bIsolated yields.

4. Mechanistic Studies

In a typical experiment, a solution of 4CzIPN in anhydrous DCE (0.125 mM) was added with an appropriate amount of quencher in a quartz cuvette. Then the emission of the sample was collected. The emission intensity at 538 nm was collected with excited wavelength of 380 nm.

**Figure S2.** Luminescence quenching of 4CzIPN by **A**

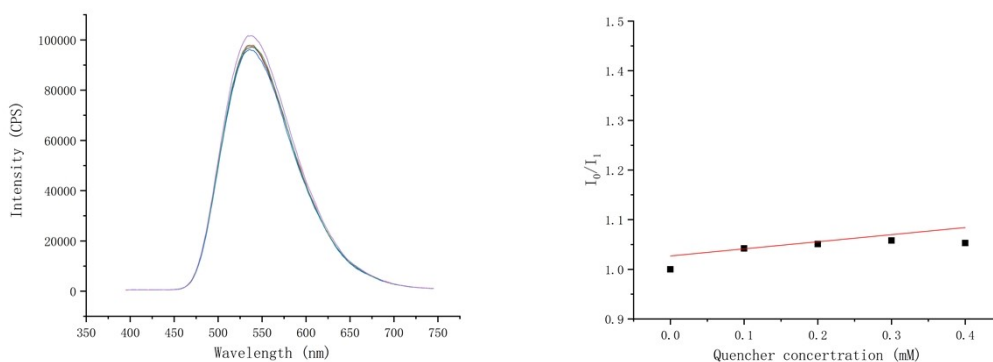
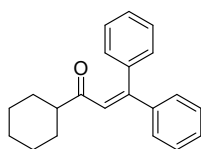
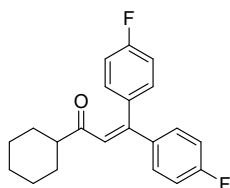


Figure S3. Luminescence quenching of 4CzIPN by **2a**

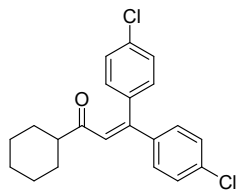
5. Analytical Data of the Products



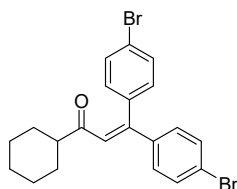
1-cyclohexyl-3,3-diphenylprop-2-en-1-one (**3a**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a yellow oil, 41.8 mg, 72% yield. ^1H NMR (500 MHz, CDCl_3) δ 7.40 – 7.27 (m, 8H), 7.22 – 7.14 (m, 2H), 6.62 (s, 1H), 2.25 (tt, J = 11.6, 3.5 Hz, 1H), 1.84 – 1.68 (m, 4H), 1.62 (d, J = 10.7 Hz, 1H), 1.35 – 1.27 (m, 2H), 1.19 – 1.08 (m, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 204.9, 153.5, 141.3, 139.2, 129.4, 129.3, 128.4, 128.4, 128.4, 128.2, 125.5, 50.9, 28.8, 25.9, 25.8. The data consistent with previously reported literature.¹¹



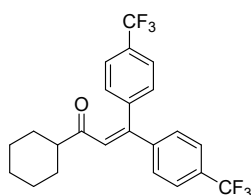
1-cyclohexyl-3,3-bis(4-fluorophenyl)prop-2-en-1-one (**3b**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a yellow oil, 45.8 mg, 70% yield. ^1H NMR (500 MHz, CDCl_3) δ 7.28 – 7.24 (m, 2H), 7.17 – 7.11 (m, 2H), 7.04 (dt, J = 20.5, 8.6 Hz, 4H), 6.59 (s, 1H), 2.31 (tt, J = 11.5, 3.4 Hz, 1H), 1.86 – 1.72 (m, 4H), 1.68 – 1.59 (m, 1H), 1.36 – 1.30 (m, 2H), 1.22 – 1.15 (m, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 204.1, 163.5 (d, J = 250.5 Hz), 162.8 (d, J = 248.3 Hz), 151.7, 137.4 (d, J = 3.5 Hz), 134.8 (d, J = 3.3 Hz), 131.3 (d, J = 8.3 Hz), 130.3 (d, J = 8.4 Hz), 125.0, 115.5 (d, J = 21.7 Hz), 115.3 (d, J = 21.5 Hz), 51.2, 28.7, 25.8, 25.8. ^{19}F NMR (470 MHz, CDCl_3) δ -111.47, -112.81. HRMS ESI $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{21}\text{H}_{21}\text{F}_2\text{O}$ 327.1555, found 327.1546.



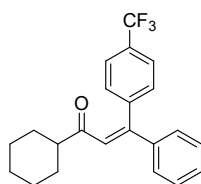
3,3-bis(4-chlorophenyl)-1-cyclohexylprop-2-en-1-one (**3c**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a yellow oil, 49.5 mg, 69% yield. ^1H NMR (500 MHz, CDCl_3) δ 7.32 (dd, $J = 21.1, 8.5$ Hz, 4H), 7.20 (d, $J = 8.6$ Hz, 2H), 7.09 (d, $J = 8.4$ Hz, 2H), 6.63 (s, 1H), 2.34 (tt, $J = 11.4, 3.4$ Hz, 1H), 1.85 – 1.73 (m, 4H), 1.68 – 1.61 (m, 1H), 1.36 – 1.27 (m, 2H), 1.25 – 1.15 (m, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 203.8, 151.4, 139.4, 137.1, 135.6, 134.6, 130.7, 129.7, 128.8, 128.5, 125.4, 51.3, 28.6, 25.8, 25.7. HRMS ESI $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{21}\text{H}_{21}\text{Cl}_2\text{O}$ 359.0964, found 359.0963.



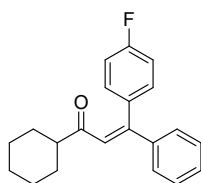
3,3-bis(4-bromophenyl)-1-cyclohexylprop-2-en-1-one (**3d**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a colorless oil, 53.6 mg, 60% yield. ^1H NMR (500 MHz, CDCl_3) δ 7.48 (dd, $J = 20.2, 8.5$ Hz, 4H), 7.13 (d, $J = 8.5$ Hz, 2H), 7.02 (d, $J = 8.4$ Hz, 2H), 6.64 (s, 1H), 2.34 (tt, $J = 11.4, 3.4$ Hz, 1H), 1.88 – 1.73 (m, 4H), 1.68 – 1.61 (m, 1H), 1.35 – 1.29 (m, 2H), 1.25 – 1.16 (m, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 203.8, 151.5, 139.8, 137.5, 131.7, 131.5, 131.0, 129.9, 125.4, 124.0, 122.8, 51.3, 28.6, 25.8, 25.7. HRMS ESI $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{21}\text{H}_{21}\text{Br}_2\text{O}$ 446.9954, found 446.9960.



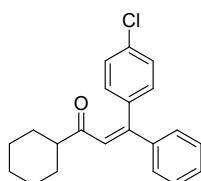
1-cyclohexyl-3,3-bis(4-(trifluoromethyl)phenyl)prop-2-en-1-one (**3e**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a yellow oil, 54.8 mg, 64% yield. ^1H NMR (500 MHz, CDCl_3) δ 7.65 (t, $J = 9.0$ Hz, 2H), 7.57 (s, 1H), 7.50 (dt, $J = 26.5, 7.8$ Hz, 2H), 7.43 – 7.34 (m, 3H), 6.74 (s, 1H), 2.36 (tt, $J = 11.4, 3.4$ Hz, 1H), 1.90 – 1.73 (m, 4H), 1.68 – 1.60 (m, 1H), 1.38 – 1.27 (m, 2H), 1.27 – 1.16 (m, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 203.46, 150.47, 141.45, 139.12, 132.71, 131.73, 131.23 (q, $J = 32.8$ Hz), 130.85 (q, $J = 32.8$ Hz), 129.22, 128.85, 127.17, 126.19 (q, $J = 3.7$ Hz), 126.03 (q, $J = 3.8$ Hz), 125.40 (q, $J = 3.4$ Hz), 123.93 (q, $J = 272.3$ Hz), 123.81 (q, $J = 272.6$ Hz), 124.62 (q, $J = 3.8$ Hz), 51.28, 28.48, 25.78, 25.67. ^{19}F NMR (470 MHz, Chloroform- d) δ -62.60, -62.68. HRMS ESI $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{23}\text{H}_{21}\text{F}_6\text{O}$ 427.1491, found 427.1493.



1-cyclohexyl-3-phenyl-3-(4-(trifluoromethyl)phenyl)prop-2-en-1-one (**3f**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a yellow oil, 47.0 mg, 66% yield. ^1H NMR (500 MHz, CDCl_3) δ 7.61 (dd, J = 24.7, 7.9 Hz, 2H), 7.43 – 7.32 (m, 4H), 7.31 – 7.27 (m, 2H), 7.20 – 7.13 (m, 1H), 6.76 (s, 0.64H), 6.64 (s, 0.36H), 2.38 (tt, J = 11.4, 3.5 Hz, 0.68H), 2.26 (tt, J = 11.7, 3.5 Hz, 0.42H), 1.90 – 1.73 (m, 4H), 1.68 – 1.60 (m, 1H), 1.35 – 1.23 (m, 3H), 1.22 – 1.12 (m, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 204.8, 203.4, 152.6, 151.6, 144.9, 143.1, 140.5, 138.4, 131.1, 130.8, 130.2, 130.0, 129.7, 129.6, 129.4, 128.7, 128.7, 128.6, 128.4, 128.3, 127.2, 125.4 (q, J = 3.7 Hz), 125.3, 125.1 (q, J = 3.6 Hz), 125.0, 123.1, 51.4, 50.9, 28.7, 28.6, 25.9, 25.8, 25.7. ^{19}F NMR (470 MHz, CDCl_3) δ -62.49, -62.66. The data consistent with previously reported literature.¹¹

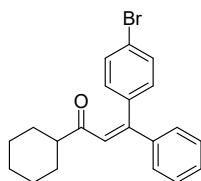


1-cyclohexyl-3-phenyl-3-(4-fluorophenyl)prop-2-en-1-one (**3g**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a yellow oil, 47.9 mg, 78% yield (*E*:*Z* isomer: 55:45 based on crude product's NMR). ^1H NMR (500 MHz, CDCl_3) δ 7.41 – 7.32 (m, 3H), 7.30 – 7.26 (m, 2H), 7.20 – 7.13 (m, 2H), 7.09 – 6.98 (m, 2H), 6.64 (s, 0.17H), 6.57 (s, 0.83H), 2.32 (tt, J = 11.5, 3.4 Hz, 0.18H), 2.23 (tt, J = 11.6, 3.4 Hz, 0.84H), 1.84 – 1.69 (m, 4H), 1.64 – 1.59 (m, 1H), 1.34 – 1.25 (m, 2H), 1.19 – 1.07 (m, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 204.7, 204.3, 163.4 (d, J = 249.5 Hz), 162.8 (d, J = 247.6 Hz), 152.8, 152.4, 141.3, 139.0, 137.4 (d, J = 3.4 Hz), 135.0 (d, J = 3.2 Hz), 131.3 (d, J = 8.1 Hz), 130.3 (d, J = 8.5 Hz), 129.4 (d, J = 10.9 Hz), 128.5 (d, J = 9.7 Hz), 128.3, 125.3, 115.4 (d, J = 29.0 Hz), 115.2 (d, J = 29.2 Hz), 51.2, 50.8, 28.7 (d, J = 9.0 Hz), 25.8 (d, J = 9.3 Hz). ^{19}F NMR (470 MHz, CDCl_3) δ -111.87, -113.17. The data consistent with previously reported literature.¹¹

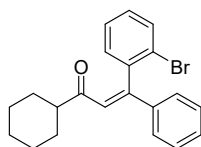


1-cyclohexyl-3-phenyl-3-(4-chlorophenyl)prop-2-en-1-one (**3h**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the

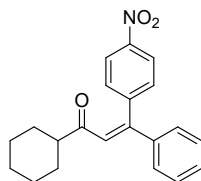
product as a yellow oil, 50.0 mg, 77% yield (*E:Z* isomer: 54:46 based on crude product's NMR). ¹H NMR (500 MHz, CDCl₃) δ 7.41 – 7.31 (m, 4H), 7.31 – 7.26 (m, 2H), 7.23 (d, *J* = 8.6 Hz, 1H), 7.19 – 7.08 (m, 2H), 6.63 (d, *J* = 37.1 Hz, 1H), 2.29 (dt, *J* = 57.7, 11.5, 3.4 Hz, 1H), 1.86 – 1.70 (m, 4H), 1.67 – 1.59 (m, 1H), 1.36 – 1.26 (m, 2H), 1.19 – 1.10 (m, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 204.7, 203.9, 152.7, 152.1, 141.0, 139.8, 138.7, 137.6, 135.3, 134.3, 130.8, 129.7, 129.5, 129.4, 128.6, 128.6, 128.5, 128.4, 128.3, 125.7, 125.2, 51.3, 50.8, 28.7, 28.6, 25.9, 25.8, 25.8. The data consistent with previously reported literature.¹¹



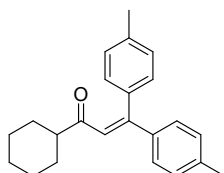
(4-bromophenyl)-1-cyclohexyl-3-phenylprop-2-en-1-one (**3i**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a yellow oil, 56.0 mg, 76% yield (*E:Z* isomer: 53:47 based on crude product's NMR). ¹H NMR (500 MHz, CDCl₃) δ 7.48 (dd, *J* = 22.7, 8.5 Hz, 2H), 7.38 (dd, *J* = 5.1, 2.2 Hz, 2H), 7.34 – 7.26 (m, 1H), 7.18 – 7.03 (m, 4H), 6.67 (s, 0.27H), 6.59 (s, 0.73H), 2.35 (tt, *J* = 11.4, 3.4 Hz, 0.30H), 2.23 (tt, *J* = 11.6, 3.3 Hz, 0.78H), 1.87 – 1.70 (m, 4H), 1.67 – 1.60 (m, 1H), 1.37 – 1.24 (m, 3H), 1.20 – 1.10 (m, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 204.8, 203.9, 152.8, 152.2, 140.9, 140.2, 138.7, 138.1, 131.6, 131.3, 131.1, 123.0, 129.6, 129.4, 128.6, 128.5, 128.4, 128.3, 125.8, 125.1, 123.7, 122.5, 51.3, 50.8, 28.7, 28.6, 25.9, 25.9, 25.8. The data consistent with previously reported literature.¹¹



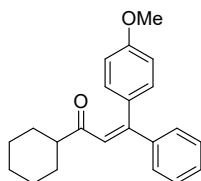
3-(2-bromophenyl)-1-cyclohexyl-3-phenylprop-2-en-1-one (**3j**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a yellow oil, 29.3 mg, 40% yield (*E:Z* isomer: 88:12 based on crude product's NMR). ¹H NMR (500 MHz, CDCl₃) δ 7.63 (d, *J* = 8.0 Hz, 1H), 7.35 (d, *J* = 8.5 Hz, 6H), 7.26 – 7.22 (m, 1H), 7.15 (d, *J* = 7.9 Hz, 1H), 6.88 (s, 0.95H), 6.31 (s, 0.05H), 2.39 (tt, *J* = 11.4, 3.4 Hz, 0.96H), 2.30 (tt, *J* = 11.6, 3.5 Hz, 0.05H), 1.93 – 1.72 (m, 4H), 1.66 – 1.60 (m, 1H), 1.38 – 1.24 (m, 3H), 1.23 – 1.14 (m, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 202.8, 151.9, 140.4, 139.0, 132.8, 130.4, 129.5, 129.2, 128.6, 127.6, 127.3, 125.1, 122.4, 51.3, 51.0, 29.8, 28.7, 28.3, 25.9, 25.9, 25.8. The data consistent with previously reported literature.¹¹



1-cyclohexyl-3-(4-nitrophenyl)-3-phenylprop-2-en-1-one (**3k**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a yellow oil, 20.6 mg, 31% yield (*E:Z* isomer: 52:48 based on crude product's NMR). ¹H NMR (500 MHz, CDCl₃) δ 8.15 (dd, *J* = 29.5, 8.3 Hz, 2H), 7.37 (dd, *J* = 23.6, 7.5 Hz, 4H), 7.29 (dd, *J* = 16.9, 8.1 Hz, 1H), 7.22 – 7.07 (m, 2H), 6.76(s,0.27H), 6.61 (s, 0.73H), 2.38 (tt, *J* = 11.1, 3.5 Hz, 0.28H), 2.20 (tt, *J* = 11.7, 3.5 Hz, 0.77H), 1.86 – 1.64 (m, 4H), 1.64 – 1.55 (m, 1H), 1.29 – 1.20 (m, 3H), 1.11 – 1.02 (m, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 204.7, 202.9, 150.4, 148.0, 147.6, 146.6, 139.8, 137.9, 130.0, 130.0, 129.3, 129.2, 129.0, 128.8, 128.6, 128.3, 128.2, 125.1, 123.7, 123.4, 51.6, 50.9, 28.6, 28.4, 25.8, 25.8, 25.7, 25.7. HRMS ESI [M + H]⁺ Calcd for C₂₁H₂₂NO₃ 336.1594, found 336.1597.

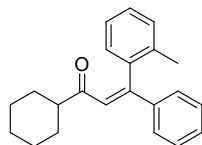


1-cyclohexyl-3,3-di-p-tolylprop-2-en-1-one (**3l**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a yellow oil, 26.3 mg, 41% yield. ¹H NMR (500 MHz, CDCl₃) δ 7.22 – 7.10 (m, 6H), 7.06 (d, *J* = 7.8 Hz, 2H), 6.56 (s, 1H), 2.38 (s, 3H), 2.36 (s, 3H), 2.26 (tt, *J* = 11.7, 3.4 Hz, 1H), 1.83 – 1.69 (m, 4H), 1.63 – 1.58 (m, 1H), 1.36 – 1.29 (m, 3H), 1.18 – 1.07 (m, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 204.9, 154.0, 139.5, 138.8, 138.2, 136.4, 129.5, 129.1, 128.8, 128.5, 124.3, 50.9, 28.8, 25.9, 25.8, 21.4, 21.3. The data consistent with previously reported literature.¹¹

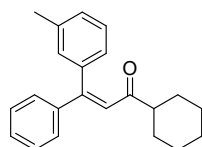


1-cyclohexyl-3-(4-methoxyphenyl)-3-phenylprop-2-en-1-one (**3m**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a yellow oil, 22.5 mg, 35% yield (*E:Z* isomer: 63:37 based on crude product's NMR). ¹H NMR (500 MHz, CDCl₃) δ 7.40 – 7.29 (m, 3H), 7.27 – 7.22 (m, 2H), 7.20 – 7.09 (m, 2H), 6.87 (dd, *J* = 23.4, 8.4 Hz, 2H), 6.56 (d, *J* = 25.2 Hz, 1H), 3.83 (d, *J* = 11.6 Hz, 3H), 2.36 – 2.17 (m, 1H), 1.84 – 1.69 (m, 4H), 1.64 – 1.59 (m, 1H), 1.34 – 1.24 (m, 3H), 1.15 – 1.10 (m, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 204.6, 160.7, 153.5, 139.5, 133.6, 131.2, 129.9, 129.4, 128.7, 128.3, 128.2, 128.1, 125.0,

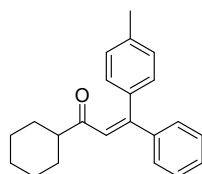
123.6, 113.8, 113.5, 55.4, 55.3, 50.9, 50.8, 29.7, 28.8, 25.9, 25.8. The data consistent with previously reported literature.¹¹



cyclohexyl-3-phenyl-3-(o-tolyl)prop-2-en-1-one (**3n**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a yellow oil, 40.0 mg, 66% yield (*E:Z* isomer: 83:17 based on crude product's NMR). ¹H NMR (500 MHz, CDCl₃) δ 7.34 – 7.28 (m, 5H), 7.26 – 7.15 (m, 3H), 7.04 (dd, *J* = 7.4, 1.4 Hz, 1H), 6.80 (s, 0.76H), 6.25 (s, 0.24H), 2.33 – 2.20(m, 1H), 2.07 (d, *J* = 11.5 Hz, 3H), 1.88 – 1.68 (m, 4H), 1.64 – 1.60 (m, 1H), 1.42 – 1.24 (m, 3H), 1.16 – 1.10 (m, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 204.0, 153.0, 139.9, 138.8, 135.7, 130.7, 130.1, 129.7, 129.3, 129.2, 128.8, 128.6, 128.6, 128.3, 128.2, 128.0, 128.0, 127.5, 125.7, 125.7, 125.2, 51.1, 50.7, 29.7, 28.8, 25.9, 25.9, 25.8, 20.4, 19.7. The data consistent with previously reported literature.¹¹

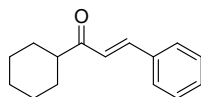


1-cyclohexyl-3-phenyl-3-(m-tolyl)prop-2-en-1-one (**3o**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a yellow oil, 45.4 mg, 75% yield (*E:Z* isomer: 50:50 based on crude product's NMR). ¹H NMR (500 MHz, CDCl₃) δ 7.39 – 7.29 (m, 4H), 7.25 – 7.15 (m, 3H), 7.13 – 7.05 (m, 1H), 6.98 (d, *J* = 7.2 Hz, 1H), 6.59 (d, *J* = 8.2 Hz, 1H), 2.34 (d, *J* = 6.9 Hz, 3H), 2.23 (tdd, *J* = 11.6, 6.4, 3.2 Hz, 1H), 1.82 – 1.69 (m, 4H), 1.64 – 1.58 (m, 1H), 1.36 – 1.24 (m, 3H), 1.15 – 1.08 (m, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 205.2, 204.9, 153.7, 153.6, 141.4, 141.3, 139.3, 139.2, 138.0, 137.8, 130.1, 130.0, 129.4, 129.2, 129.1, 129.0, 128.4, 128.4, 128.3, 128.3, 128.1, 128.0, 126.6, 125.7, 125.6, 125.4, 50.8, 50.7, 28.8, 28.8, 25.9, 25.8, 21.4. The data consistent with previously reported literature.¹¹

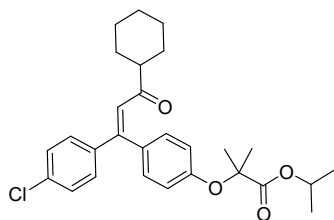


cyclohexyl-3-phenyl-3-(p-tolyl)prop-2-en-1-one (**3p**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a yellow oil, 36.2 mg, 60% yield (*E:Z* isomer: 58:42 based on crude product's NMR). ¹H NMR (500 MHz, CDCl₃) δ 7.43 – 7.29 (m, 4H), 7.20 – 7.16 (m, 3H), 7.14 – 7.06 (m, 2H), 6.60 (d, *J* = 14.0 Hz, 1H), 2.38 (d, *J* = 15.1 Hz, 3H), 2.27 (dt, *J* = 23.6, 11.5,

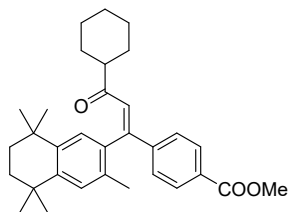
3.5 Hz, 1H), 1.84 – 1.70 (m, 4H), 1.65 – 1.59 (m, 1H), 1.36 – 1.27 (m, 2H), 1.15 – 1.10 (m, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 204.8, 153.6, 141.6, 139.5, 139.4, 138.5, 138.3, 136.2, 129.5, 129.4, 129.2, 129.1, 128.8, 128.5, 128.4, 128.3, 128.3, 128.1, 125.2, 124.6, 50.9, 50.8, 28.8, 28.8, 25.9, 25.8, 21.4, 21.3. The data consistent with previously reported literature.¹¹



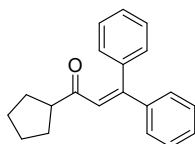
(*E*)-1-cyclohexyl-3-phenylprop-2-en-1-one (**3q**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a yellow oil, 11.0 mg, 26% yield (*E*:*Z* isomer: 92:8 based on crude product's NMR). ¹H NMR (500 MHz, CDCl₃) δ 7.64 – 7.50 (m, 3H), 7.42 – 7.34 (m, 3H), 6.82 (d, *J* = 16.0 Hz, 1H), 2.66 (tt, *J* = 11.4, 3.4 Hz, 1H), 1.95 – 1.80 (m, 4H), 1.75 – 1.67 (m, 1H), 1.48 – 1.37 (m, 2H), 1.37 – 1.24 (m, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 203.2, 142.2, 134.8, 130.3, 128.9, 128.3, 124.8, 49.5, 28.8, 25.9, 25.8. The data consistent with previously reported literature.¹²



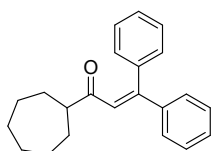
isopropyl 2-(4-(1-(4-chlorophenyl)-3-cyclohexyl-3-oxoprop-1-en-1-yl)phenoxy)-2-methylpropanoate (**3r**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a yellow oil, 46.9 mg, 50% yield (*E*:*Z* isomer: 61:39 based on crude product's NMR). ¹H NMR (500 MHz, CDCl₃) δ 7.31 (dd, *J* = 23.4, 8.2 Hz, 2H), 7.17 (dd, *J* = 19.6, 8.6 Hz, 2H), 7.06 (dd, *J* = 22.4, 8.2 Hz, 2H), 6.79 (dd, *J* = 29.4, 8.6 Hz, 2H), 6.61 (s, 0.62H), 6.45 (s, 0.38H), 5.07 (tq, *J* = 12.5, 6.2 Hz, 1H), 2.32 (tt, *J* = 11.5, 3.4 Hz, 0.58H), 2.20 (tt, *J* = 11.8, 3.2 Hz, 0.4H), 1.86 – 1.67 (m, 4H), 1.61 (d, *J* = 8.9 Hz, 7H), 1.36 – 1.24 (m, 3H), 1.22 (dd, *J* = 11.3, 6.2 Hz, 6H), 1.19 – 1.15 (m, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 205.5, 203.7, 173.6, 173.3, 157.0, 156.2, 152.4, 151.5, 150.1, 142.9, 140.2, 137.8, 135.2, 134.2, 134.0, 131.8, 130.8, 130.7, 129.8, 129.4, 128.5, 128.3, 125.8, 123.4, 118.2, 118.1, 79.2, 79.2, 69.2, 69.1, 51.3, 50.6, 28.9, 28.7, 25.9, 25.8, 25.8, 25.8, 25.4, 25.4, 21.6. HRMS ESI [*M* + *H*]⁺ Calcd for C₂₈H₃₄ClO₄ 469.2140, found 469.2140.



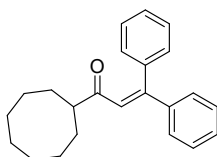
methyl -4-(3-cyclohexyl-3-oxo-1-(3,5,5,8,8-pentamethyl-5,6,7,8-tetrahydronaphthalen-2-yl)prop-1-en-1-yl)benzoate (**3s**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a yellow oil, 38.9 mg, 41% yield (*E:Z* isomer: 66:34 based on crude product's NMR). ¹H NMR (500 MHz, CDCl₃) δ 7.97 (t, *J* = 8.8 Hz, 2H), 7.31 (dd, *J* = 69.5, 8.2 Hz, 2H), 7.15 – 6.95 (m, 2H), 6.67 (s, 0.72H), 6.35 (s, 0.28H), 3.90 (d, *J* = 3.0 Hz, 3H), 2.34 (tt, *J* = 11.7, 3.6 Hz, 0.33H), 2.00 (dt, *J* = 11.4, 3.0 Hz, 0.71H), 1.96 (d, *J* = 6.7 Hz, 3H), 1.92 – 1.72 (m, 2H), 1.71 – 1.49 (m, 8H), 1.33 – 1.18 (m, 16H). ¹³C NMR (126 MHz, CDCl₃) δ 206.1, 204.8, 166.8, 166.7, 153.6, 151.3, 145.5, 145.5, 144.6, 144.3, 142.5, 142.5, 138.8, 134.8, 132.8, 132.7, 130.4, 129.8, 129.6, 129.2, 129.2, 128.8, 128.8, 128.4, 128.3, 128.0, 127.9, 127.4, 52.2, 52.1, 51.3, 49.4, 35.2, 35.1, 35.1, 34.1, 34.1, 33.9, 33.9, 31.9, 31.8, 29.7, 28.7, 25.8, 25.8, 25.7, 20.3, 19.5. HRMS ESI [*M* + *H*]⁺ Calcd for C₃₃H₄₁O₃ 473.3050, found 473.3043.



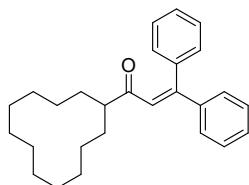
1-cyclopentyl-3,3-diphenylprop-2-en-1-one (**4a**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a yellow oil, 31.8 mg, 58% yield. ¹H NMR (500 MHz, CDCl₃) δ 7.43 – 7.28 (m, 8H), 7.23 – 7.14 (m, 2H), 6.64 (s, 1H), 2.83 (p, *J* = 7.9 Hz, 1H), 1.80 – 1.67 (m, 4H), 1.62 (td, *J* = 10.5, 9.8, 4.5 Hz, 2H), 1.54 – 1.48 (m, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 204.2, 153.3, 141.4, 139.2, 129.5, 129.3, 128.4, 128.4, 128.3, 128.1, 125.9, 51.8, 29.4, 26.2. HRMS ESI [*M* + *H*]⁺ Calcd for C₂₀H₂₁O 277.1587, found 277.1583.



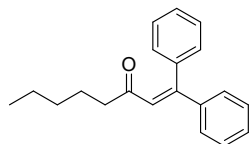
1-cycloheptyl-3,3-diphenylprop-2-en-1-one (**4b**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a yellow oil, 33.1 mg, 54% yield. ¹H NMR (500 MHz, CDCl₃) δ 7.41 – 7.28 (m, 8H), 7.18 (dd, *J* = 6.6, 3.0 Hz, 2H), 6.61 (s, 1H), 2.43 (tt, *J* = 9.8, 3.9 Hz, 1H), 1.82 (ddt, *J* = 14.2, 7.2, 3.8 Hz, 2H), 1.71 – 1.63 (m, 2H), 1.53 (dtd, *J* = 13.7, 10.0, 3.5 Hz, 2H), 1.46 (p, *J* = 2.9 Hz, 4H), 1.33 – 1.26 (m, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 205.5, 153.4, 141.3, 139.2, 129.4, 129.2, 128.4, 128.4, 128.2, 125.8, 52.0, 30.2, 28.2, 26.8. HRMS ESI [*M* + *H*]⁺ Calcd for C₂₂H₂₅O 305.1900, found 305.1894.



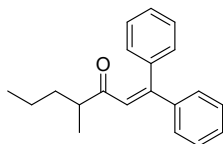
1-cyclooctyl-3,3-diphenylprop-2-en-1-one (**4c**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a yellow oil, 26.1 mg, 41% yield. ^1H NMR (500 MHz, CDCl_3) δ 7.41 – 7.35 (m, 4H), 7.36 – 7.28 (m, 5H), 7.21 – 7.17 (m, 2H), 6.59 (s, 1H), 2.49 (tt, J = 9.4, 3.5 Hz, 1H), 1.76 (ddt, J = 14.9, 7.7, 3.6 Hz, 2H), 1.67 – 1.59 (m, 3H), 1.56 – 1.49 (m, 2H), 1.42 (dq, J = 8.1, 3.7 Hz, 3H), 1.37 – 1.26 (m, 4H). ^{13}C NMR (126 MHz, CDCl_3) δ 206.0, 153.1, 141.3, 139.3, 129.5, 129.2, 128.4, 128.4, 128.3, 126.1, 50.1, 28.7, 26.4, 26.4, 25.6. HRMS ESI $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{23}\text{H}_{27}\text{O}$ 319.2056, found 319.2053.



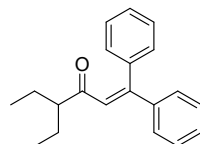
1-cyclododecyl-3,3-diphenylprop-2-en-1-one (**4d**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a yellow oil, 18.3 mg, 25% yield. ^1H NMR (500 MHz, CDCl_3) δ 7.40 – 7.27 (m, 8H), 7.20 – 7.15 (m, 2H), 6.59 (s, 1H), 2.54 (p, J = 6.4 Hz, 1H), 1.58 – 1.53 (m, 2H), 1.47 (dq, J = 13.6, 6.7 Hz, 2H), 1.32 – 1.20 (m, 14H), 1.20 – 1.16 (m, 2H), 1.10 (dt, J = 13.2, 6.6 Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 206.0, 152.6, 141.4, 139.2, 129.6, 129.2, 128.5, 128.4, 128.4, 128.3, 126.5, 47.1, 29.8, 26.4, 23.7, 23.7, 23.1, 22.8. HRMS ESI $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{27}\text{H}_{35}\text{O}$ 375.2682, found 375.2678.



4e-1

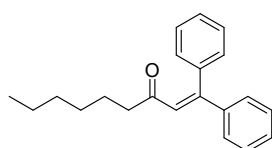


4e-2

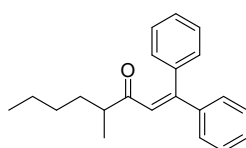


4e-3

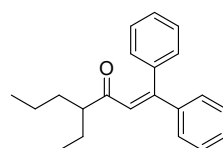
1,1-diphenyloct-1-en-3-one (**4e-1**), 4-methyl-1,1-diphenylhept-1-en-3-one (**4e-2**), 4-ethyl-1,1-diphenylhex-1-en-3-one (**4e-3**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a yellow oil, 30.0 mg, 54% total yield (Isomer: 28:61:11 based on crude product's NMR). ^1H NMR (500 MHz, CDCl_3) δ 7.40 – 7.29 (m, 8H), 7.19 (dt, J = 7.4, 3.3 Hz, 2H), 6.67 – 6.57 (m, 1H), 2.52 – 2.20 (m, 1H), 1.69 – 1.61 (m, 1H), 1.54 – 1.38 (m, 1H), 1.29 – 1.21 (m, 2H), 1.11 – 0.99 (m, 2H), 0.84 (dt, J = 14.4, 7.2 Hz, 4H). ^{13}C NMR (126 MHz, CDCl_3) δ 205.8, 204.7, 202.6, 153.6, 141.4, 141.4, 139.2, 139.2, 129.6, 129.5, 129.5, 129.3, 129.3, 128.5, 128.4, 128.4, 128.3, 128.3, 128.2, 128.1, 127.0, 125.8, 125.6, 55.2, 46.0, 43.2, 35.6, 31.4, 24.2, 20.5, 16.3, 14.1, 14.0, 11.9. HRMS ESI $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{20}\text{H}_{23}\text{O}$ 279.1743, found 279.1743.



4f-1



4f-2



4f-3

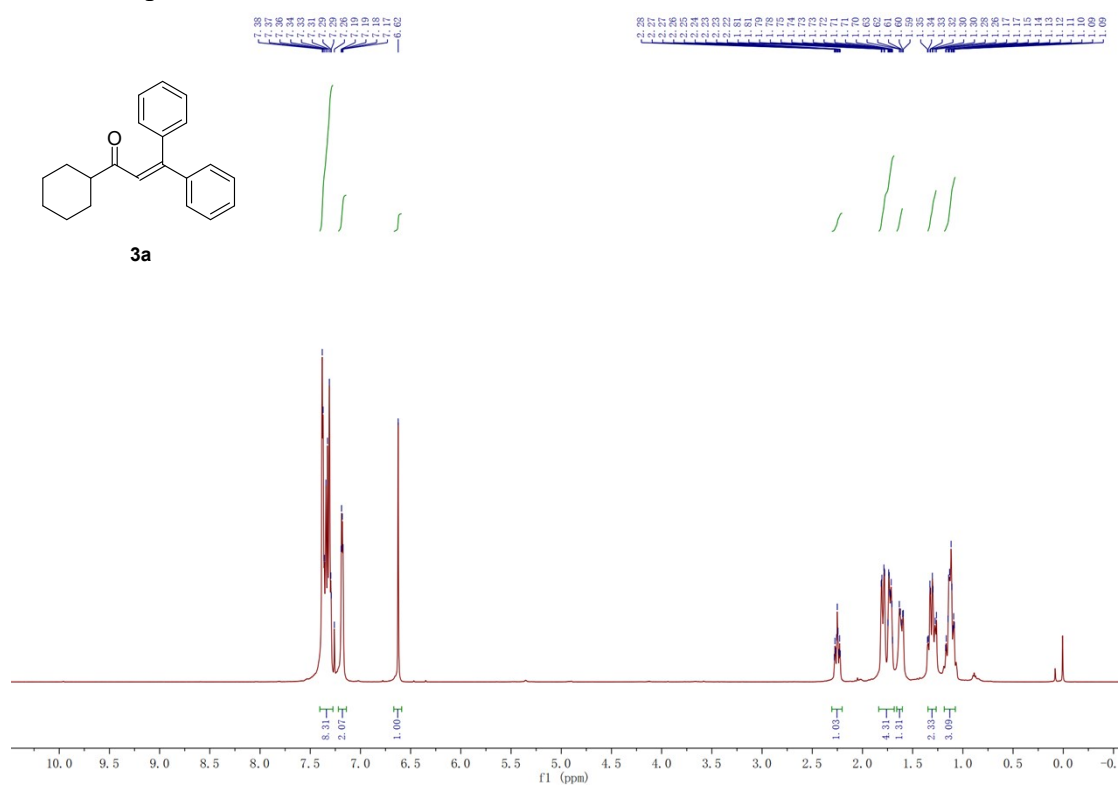
1,1-diphenylnon-1-en-3-one (**4f-1**), 4-methyl-1,1-diphenyloct-1-en-3-one (**4f-2**), 4-ethyl-1,1-diphenylhept-1-en-3-one (**4f-3**): The compound was purified by flash column chromatography (petroleum ether/EtOAc =50:1) to give the product as a yellow oil, 41.5 mg, 71% total yield (Isomer: 7:47:46 based on crude product's NMR). ¹H NMR (500 MHz, CDCl₃) δ 7.39 – 7.29 (m, 8H), 7.18 (dd, *J* = 6.6, 3.0 Hz, 2H), 6.64 (s, 0.49H), 6.62 (s, 0.49H), 6.58 (s, 0.02H), 2.45 (h, *J* = 6.9 Hz, 0.48H), 2.36 (tt, *J* = 7.6, 5.7 Hz, 0.47H), 2.23 (t, *J* = 7.5 Hz, 0.03H), 1.68 – 1.54 (m, 2H), 1.49 – 1.30 (m, 1H), 1.30 – 1.18 (m, 3H), 1.02 (d, *J* = 6.9 Hz, 1H), 0.91 – 0.79 (m, 5H). ¹³C NMR (126 MHz, CDCl₃) δ 205.5, 204.8, 153.7, 153.6, 141.4, 141.4, 139.2, 139.2, 129.5, 129.3, 129.3, 128.4, 128.4, 128.4, 128.3, 128.2, 128.1, 125.7, 125.6, 53.5, 46.2, 33.5, 33.1, 29.5, 24.6, 22.8, 20.7, 16.4, 14.3, 14.0, 11.9. HRMS ESI [*M* + *H*]⁺ Calcd for C₂₁H₂₅O 293.1900, found 293.1903.

6. Reference

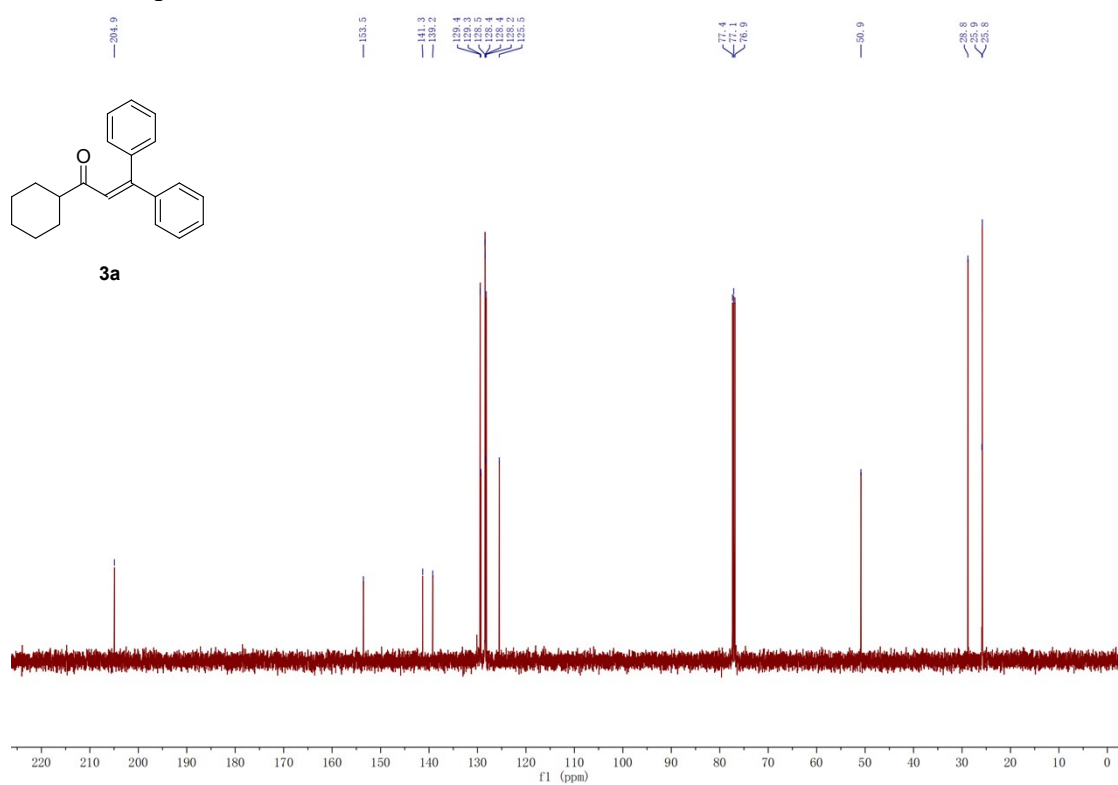
- (1) Zhang, G.; Bai, R. X.; Li, C. H.; Feng, C. G.; Lin, G. Q. *Tetrahedron* **2019**, *72*, 1658-1662.
- (2) Liu, W. Q.; Lei, T.; Zhou, S.; Yang, X. L.; Li, J.; Chen, B.; Wu, L. Z. *J. Am. Chem. Soc.* **2019**, *141*, 13941-13947.
- (3) Cheng, H. C.; Lam, T. L.; Liu, Y.; Tang, Z.; Che, C. M. *Angew. Chem. Int. Ed.* **2021**, *60*, 1383-1389.
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- (5) Pagire, S. K.; Kumagai, N.; Shibasaki, M. *Org. Lett.* **2020**, *22*, 7853-7858.
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- (10) Tang, W. Y.; Chen, L.; Zheng, Z.; Zhan, L. W.; Hou, J.; Li, B. D. *Org. Lett.* **2021**, *23*, 3939-3943.
- (11) Zhao, F.; Li, C. L.; Wu, X. F. *Chem. Commun.* **2020**, *56*, 9182-9185.
- (12) Kerr, W. J.; Mudd, R. J.; Brown, J. A. *Chem. Eur. J.* **2016**, *22*, 4738-4742.

7. NMR Spectra of New Compounds and Products

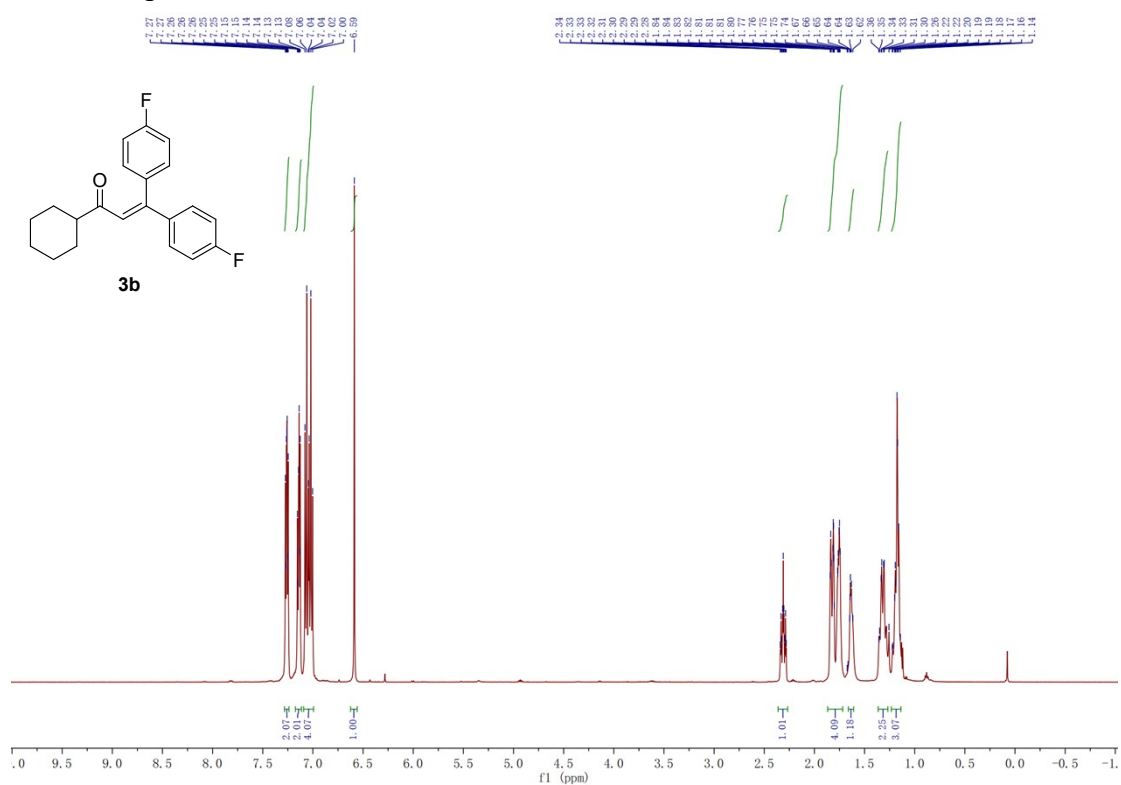
^1H NMR spectrum of **3a** in CDCl_3 at 500 MHz



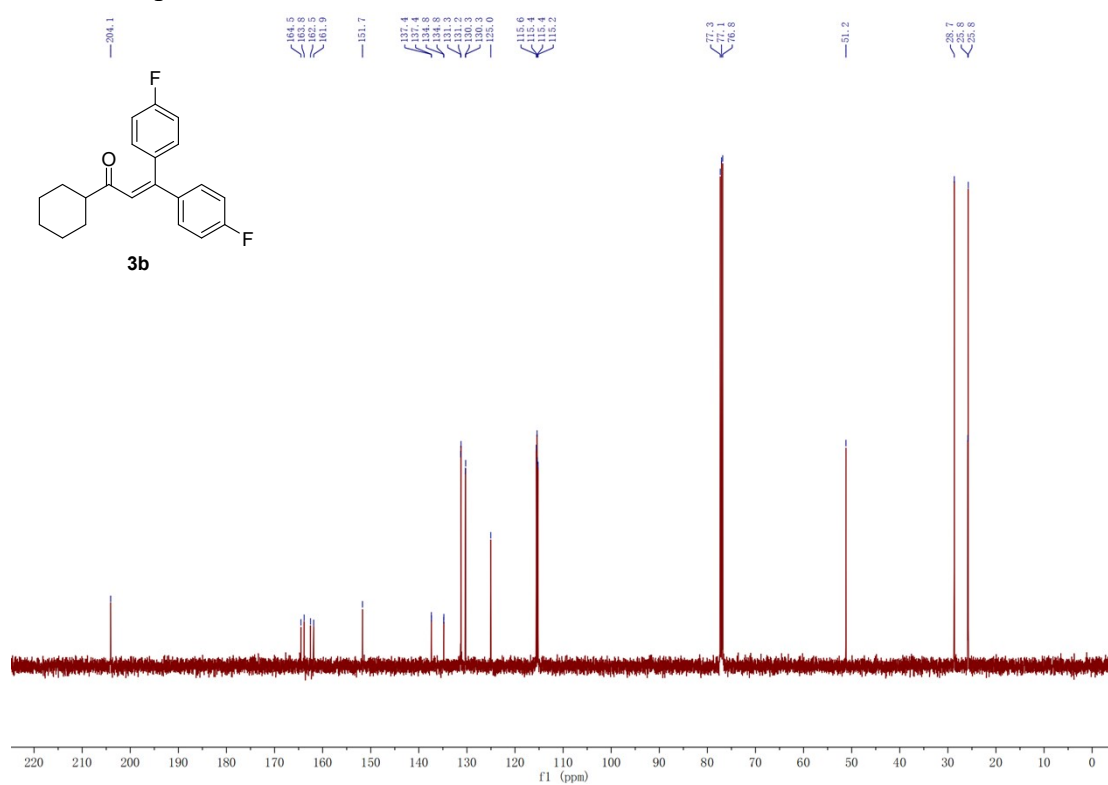
^{13}C NMR spectrum of **3a** in CDCl_3 at 126 MHz



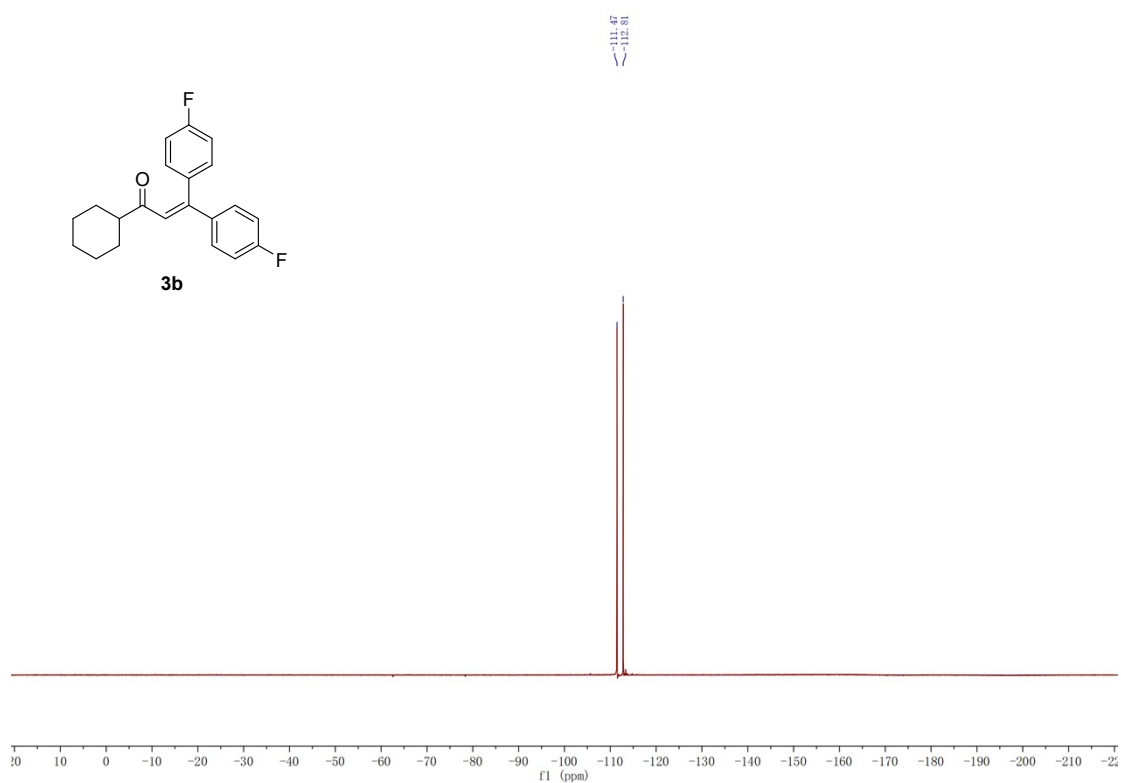
^1H NMR spectrum of **3b** in CDCl_3 at 500 MHz



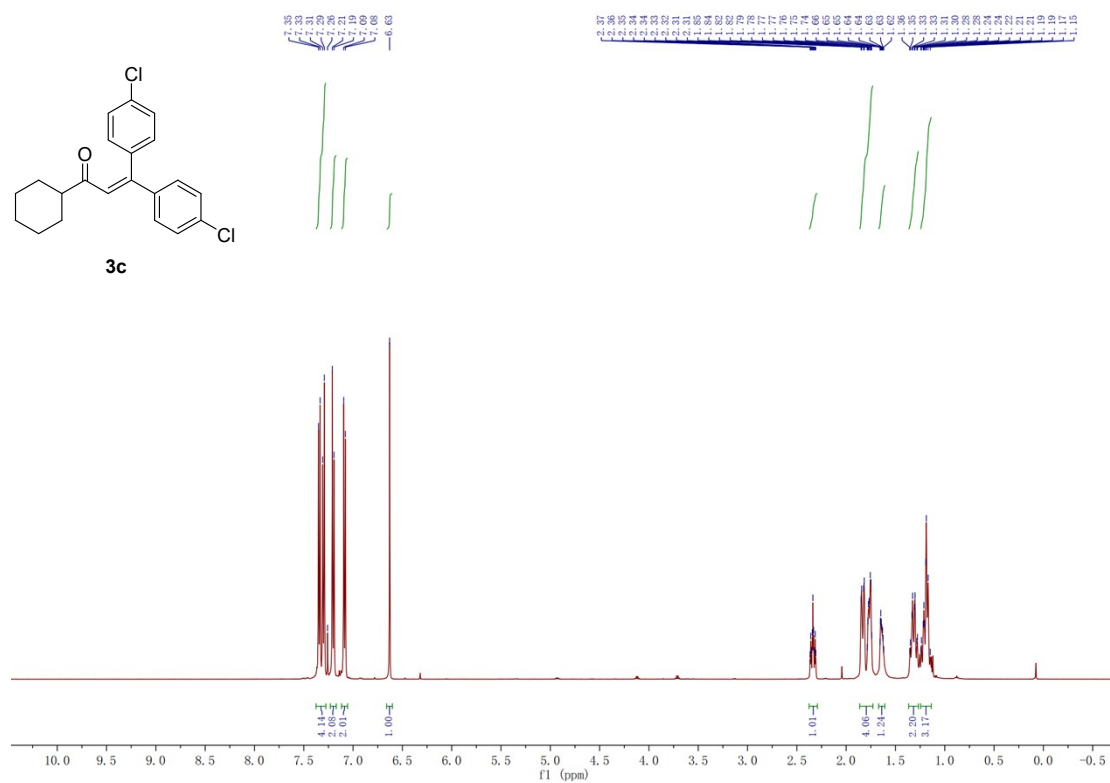
^{13}C NMR spectrum of **3b** in CDCl_3 at 126 MHz



^{19}F NMR spectrum of **3b** in CDCl_3 at 470 MHz



^1H NMR spectrum of **3c** in CDCl_3 at 500 MHz

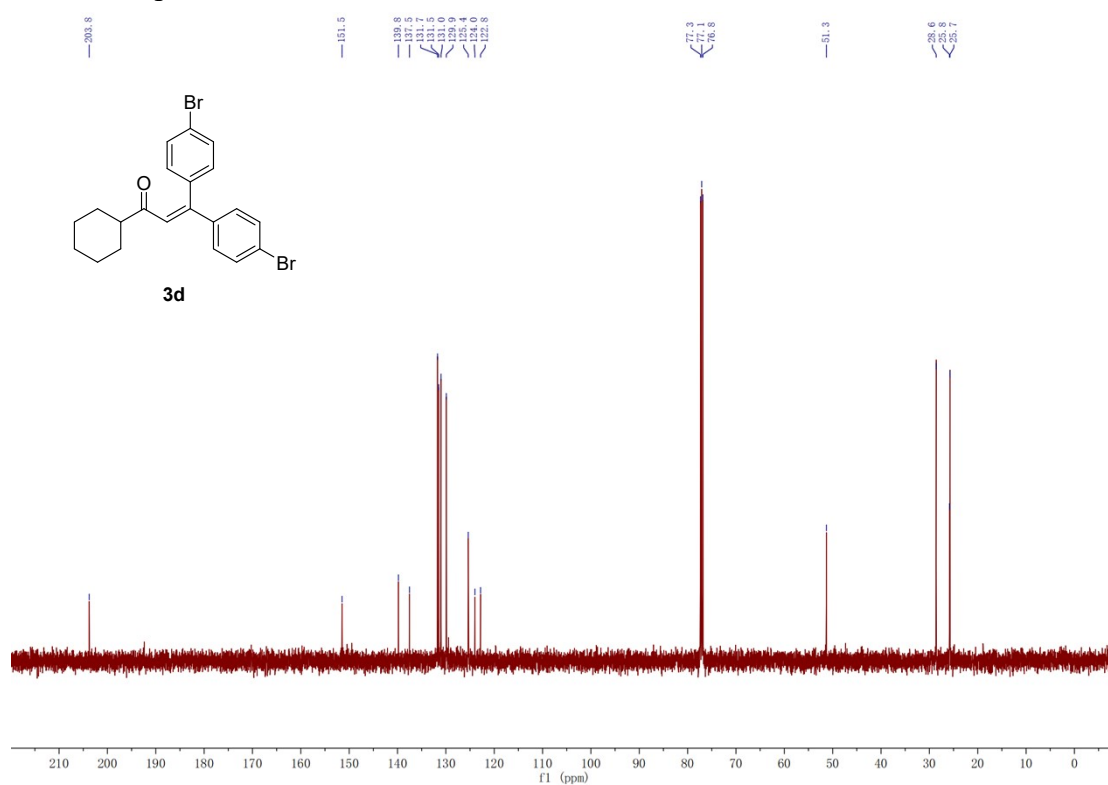


3d

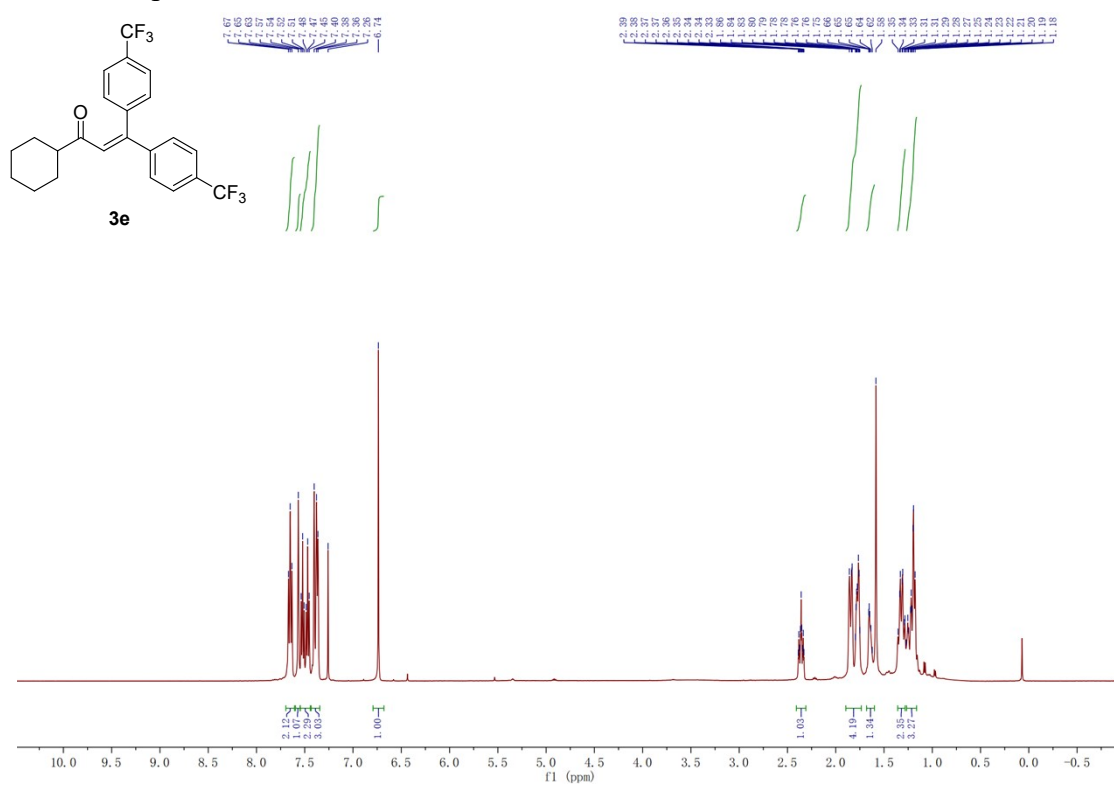
¹H NMR spectrum (CDCl₃) of compound **3d**. The spectrum shows aromatic signals between 6.5 and 7.6 ppm and aliphatic signals between 1.0 and 2.5 ppm. Integration values are provided below the peaks.

Chemical Shift (ppm)	Integration
7.50	4.07
7.25	2.06
7.15	2.05
6.60	1.00
2.45	1.02
1.85	4.14
1.75	1.11
1.45	2.22
1.35	3.12

^{13}C NMR spectrum of **3d** in CDCl_3 at 126 MHz



^1H NMR spectrum of **3e** in CDCl_3 at 500 MHz



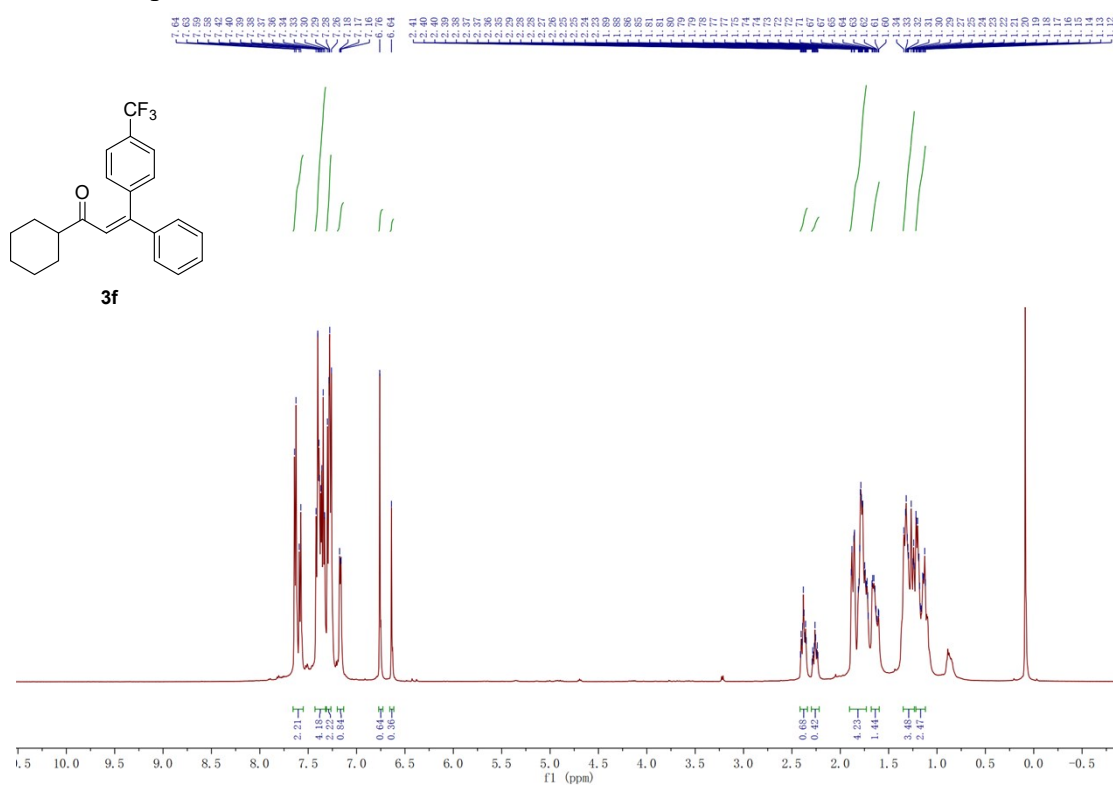
[illegible]

3e

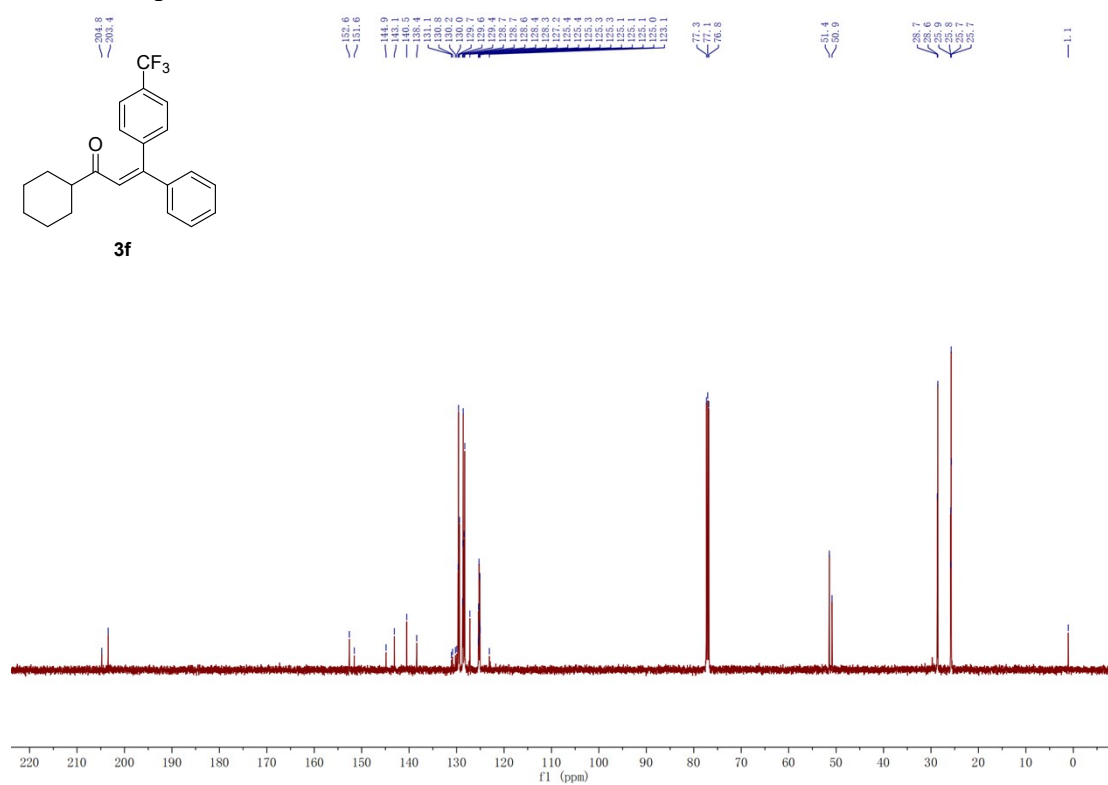
Chemical structure of **3e** is shown above the spectrum. The structure is a cyclohexylidene group attached to a propenyl chain, which is further substituted with a 4-(trifluoromethyl)phenyl group and a 4-(trifluoromethyl)phenyl group.

The spectrum displays a single sharp peak at approximately 62 ppm, labeled **f1 (ppm)** on the x-axis. The x-axis ranges from 0 to -170 ppm.

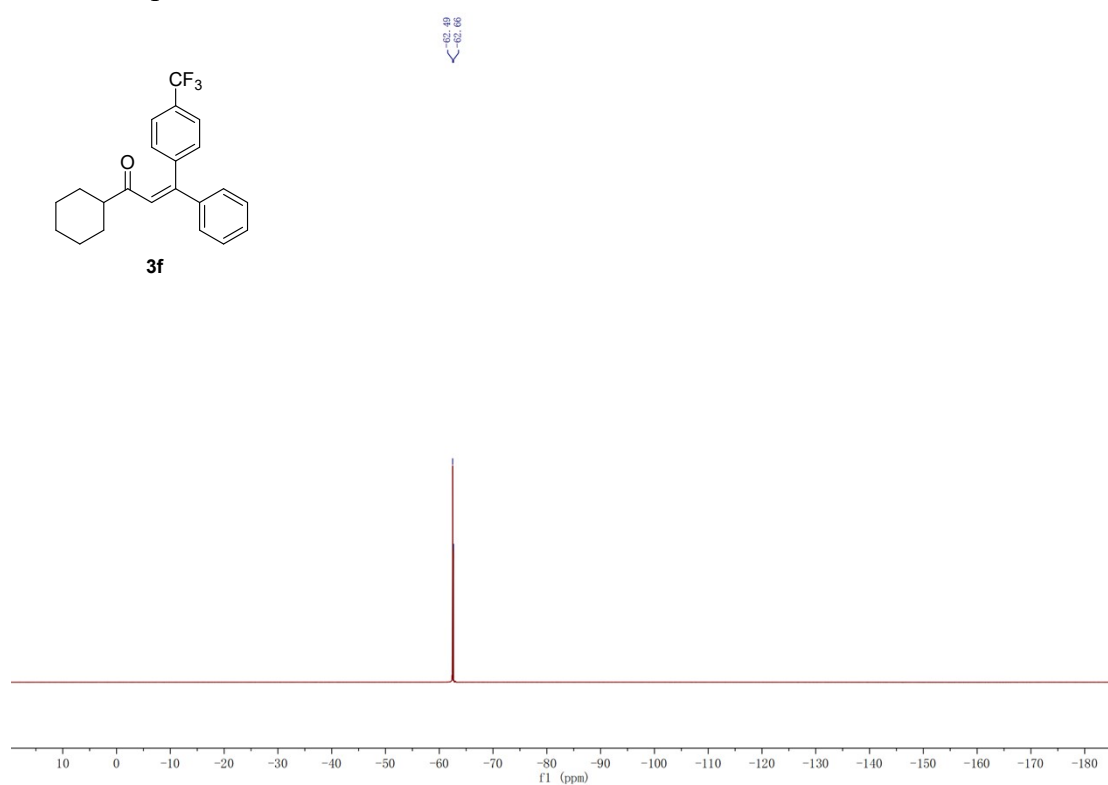
^1H NMR spectrum of **3f** in CDCl_3 at 500 MHz



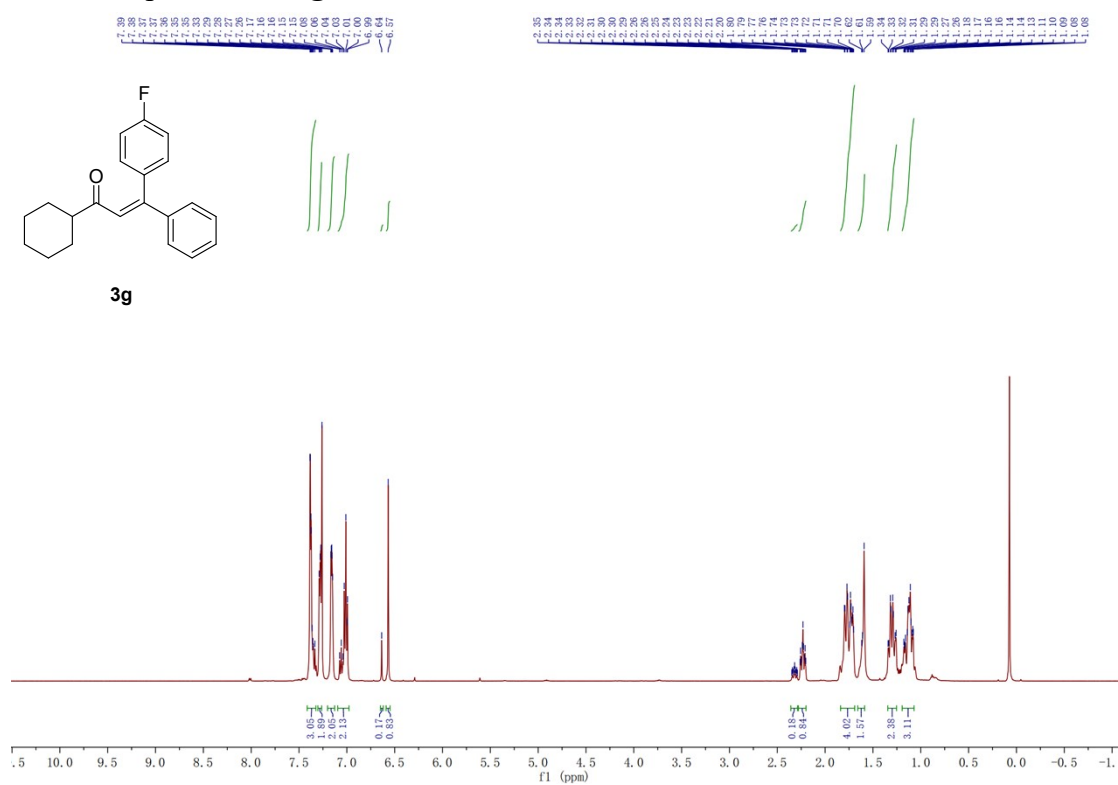
^{13}C NMR spectrum of **3f** in CDCl_3 at 126 MHz



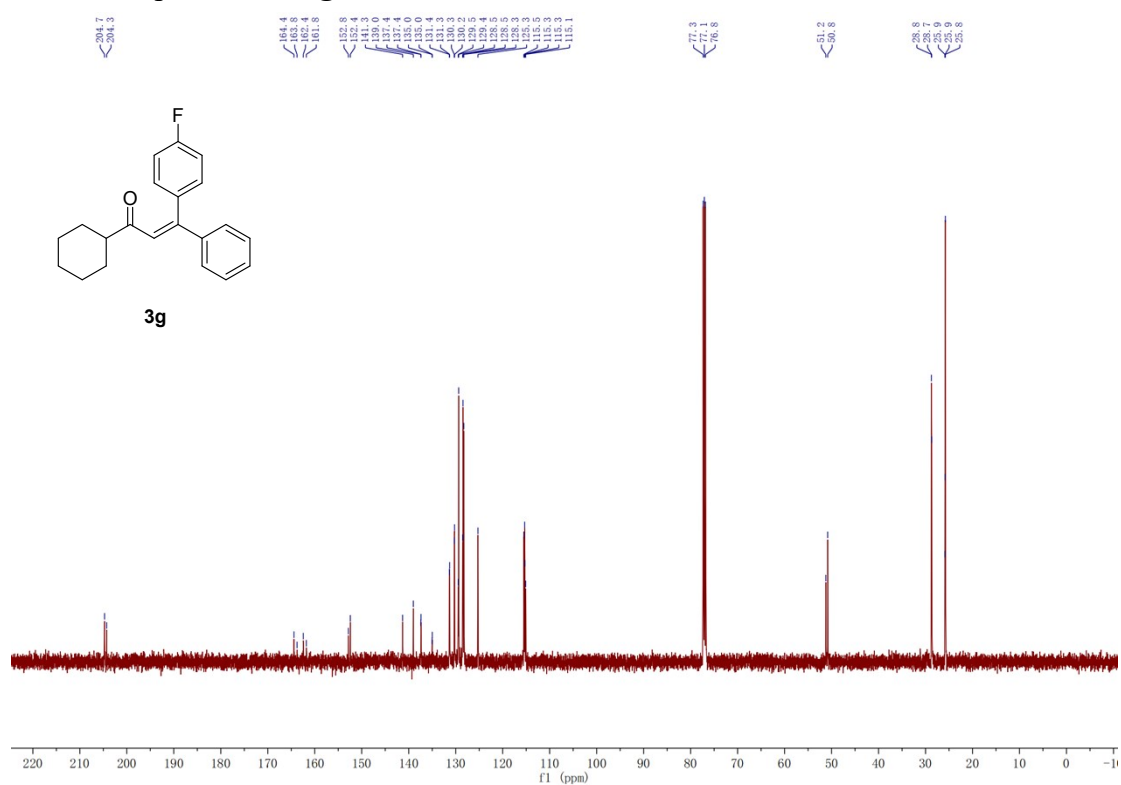
^{19}F NMR spectrum of **3f** in CDCl_3 at 470 MHz



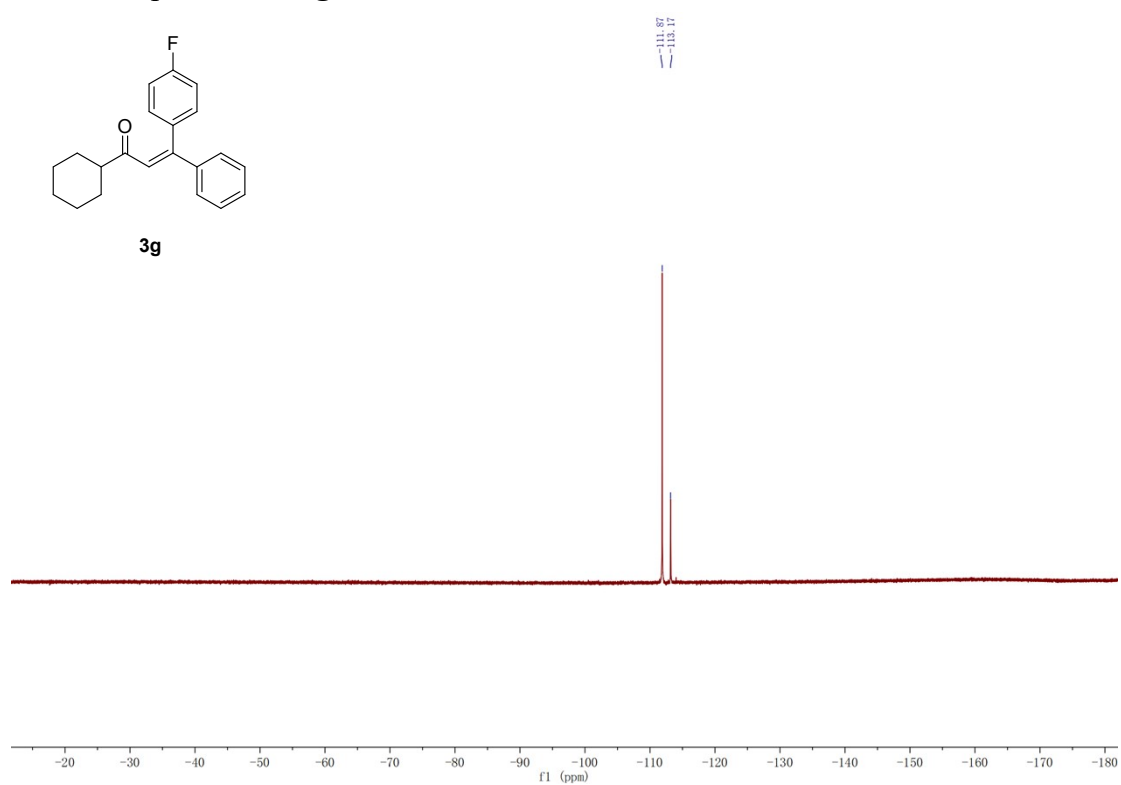
^1H NMR spectrum of **3g** in CDCl_3 at 500 MHz



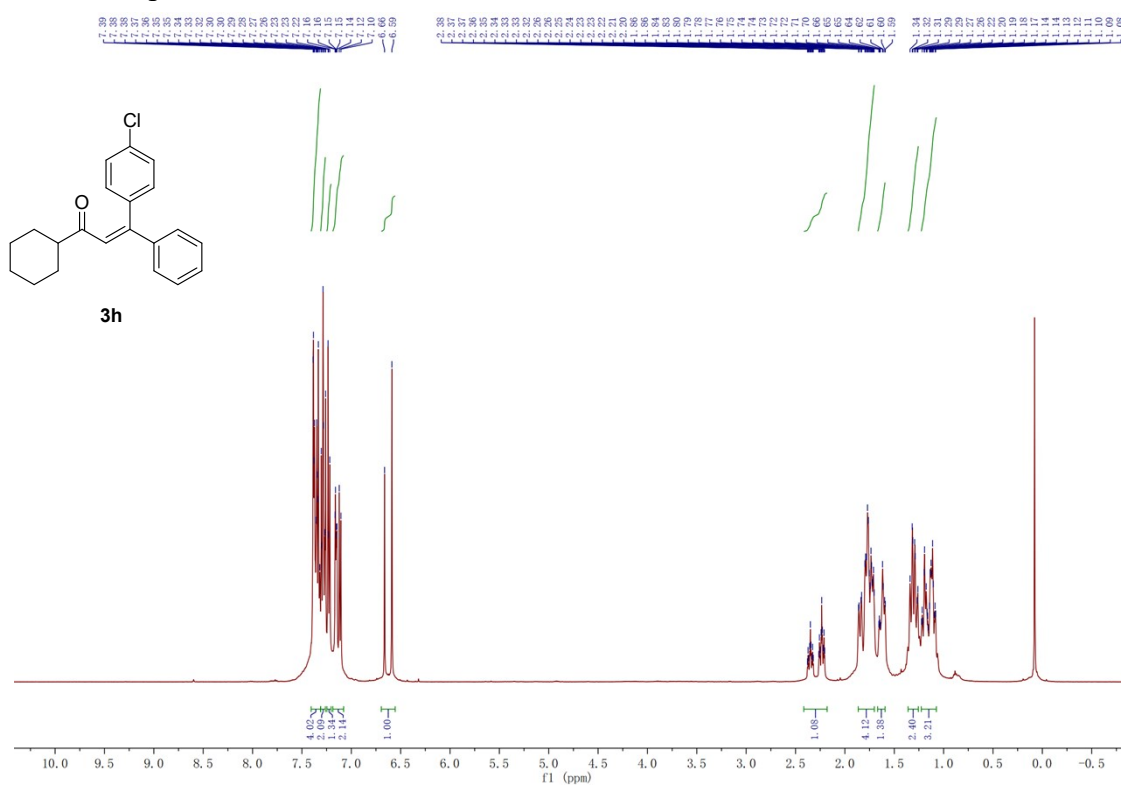
^{13}C NMR spectrum of **3g** in CDCl_3 at 126 MHz



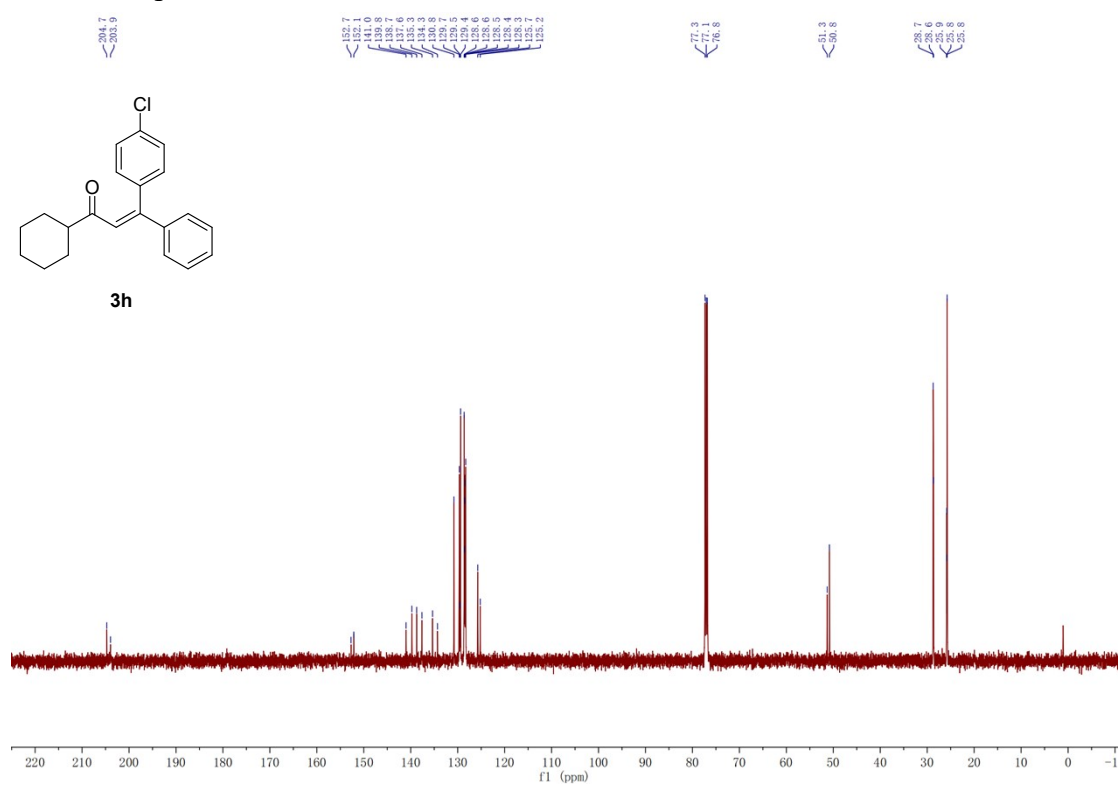
^{19}F NMR spectrum of **3g** in CDCl_3 at 470 MHz



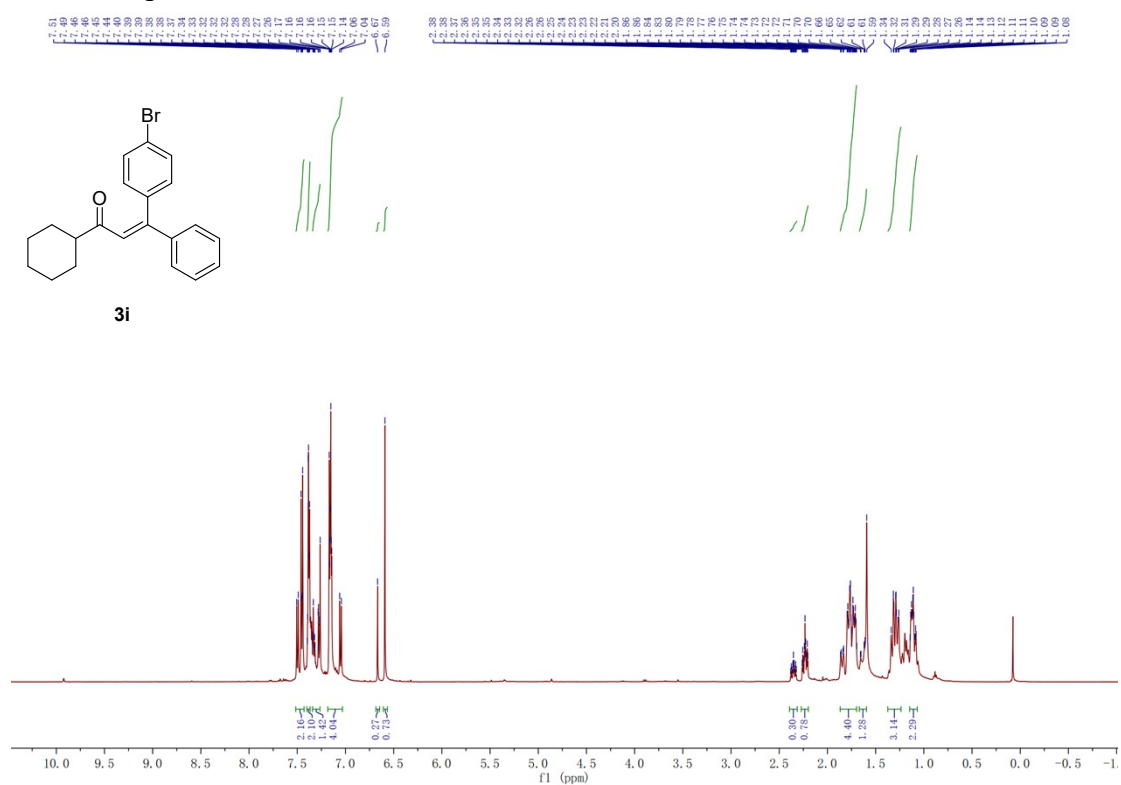
^1H NMR spectrum of **3h** in CDCl_3 at 500 MHz



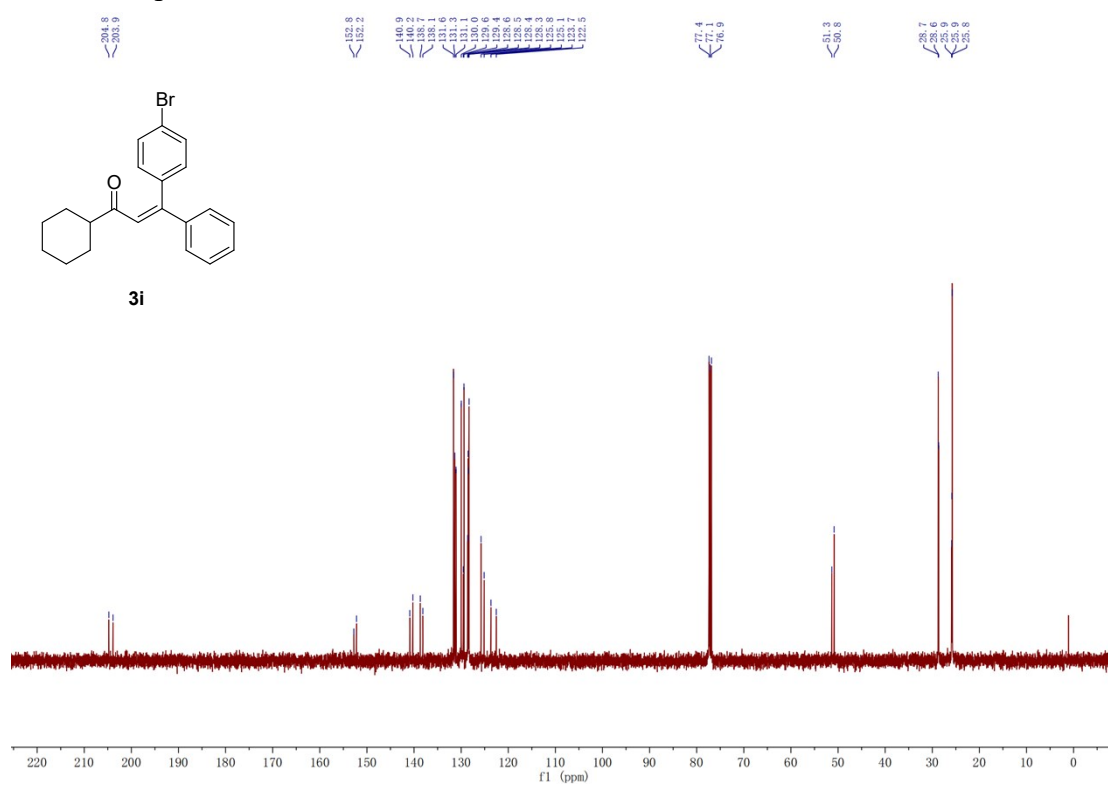
^{13}C NMR spectrum of **3h** in CDCl_3 at 126 MHz



^1H NMR spectrum of **3i** in CDCl_3 at 500 MHz



^{13}C NMR spectrum of **3i** in CDCl_3 at 126 MHz



3j

BrC1=CC=C(C=C1)/C=C(C2=CC=CC=C2)C(=O)C3CCCCC3

1.00
6.05
1.20
1.01
0.95
0.05
0.96
0.05
4.00
1.45
8.44
2.71

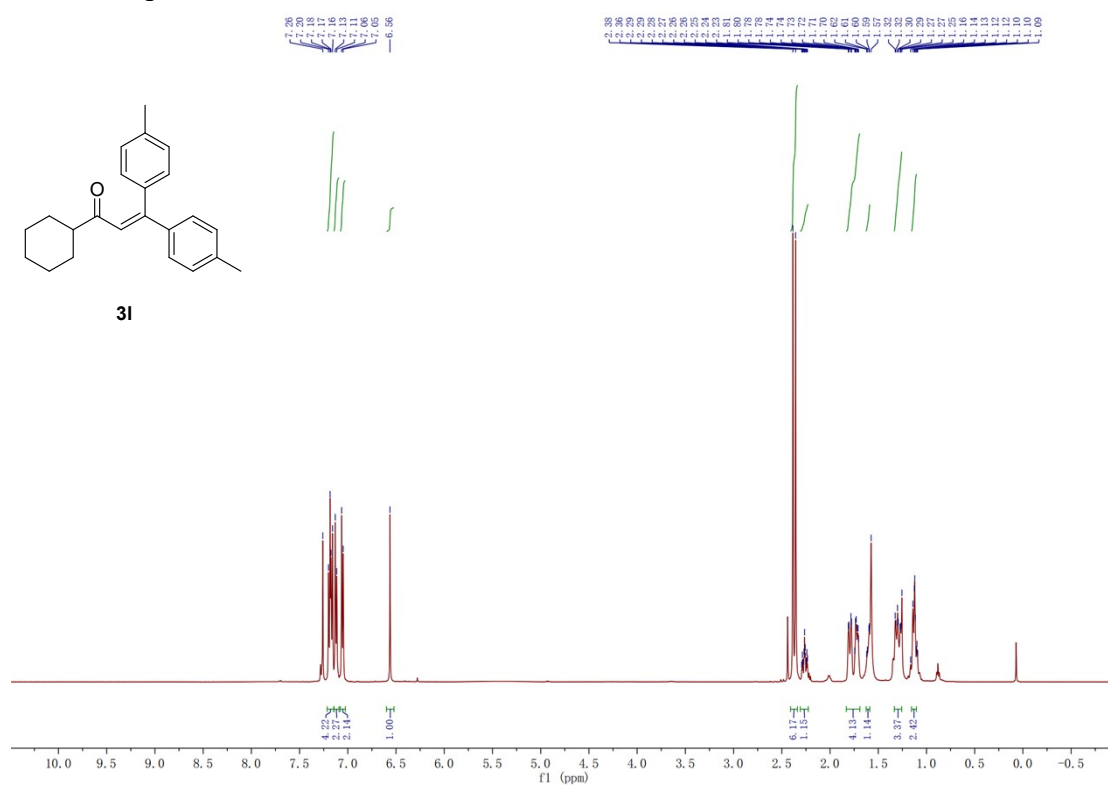
3j

Chemical structure of **3j** is shown above the spectrum. The spectrum displays peaks from 0 to 230 ppm. Key peaks are labeled with their chemical shifts: 202.8, 151.9, 148.1, 139.0, 132.8, 130.5, 129.5, 129.2, 128.6, 127.3, 125.1, 122.1, 77.1, 77.0, 76.9, 51.3, 29.0, 28.3, 27.9, 27.3, 26.6, and 0.

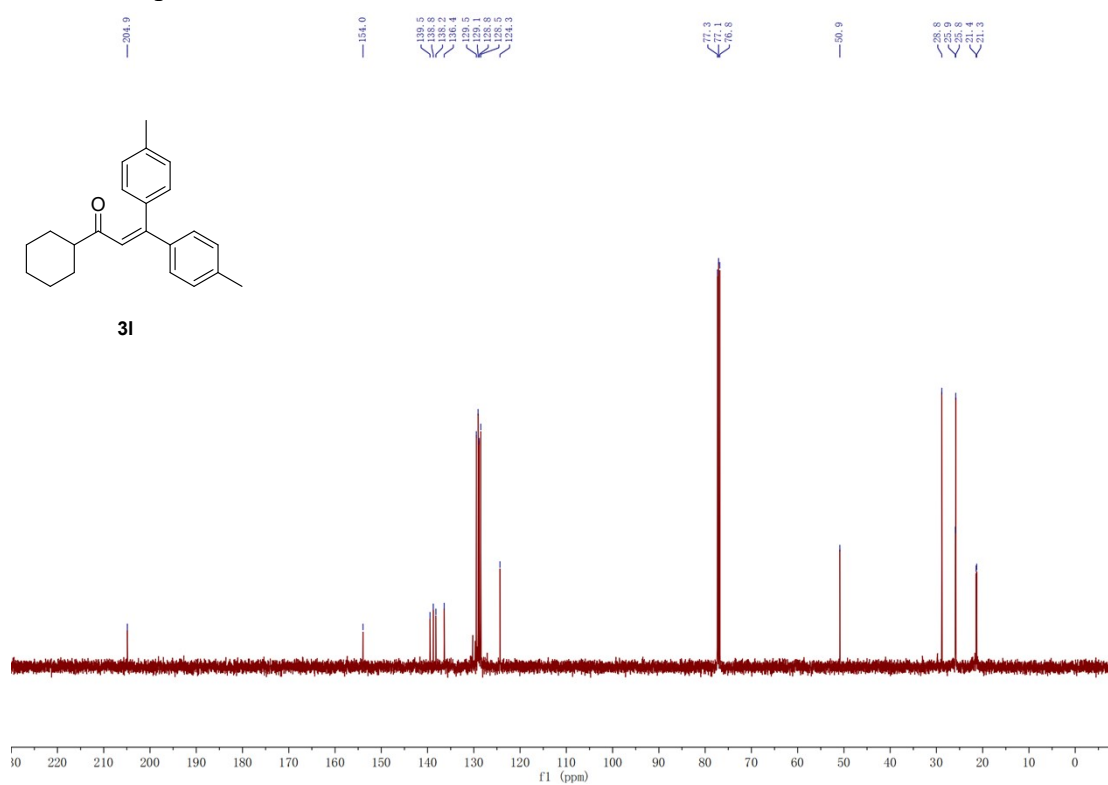
[illegible]

3k

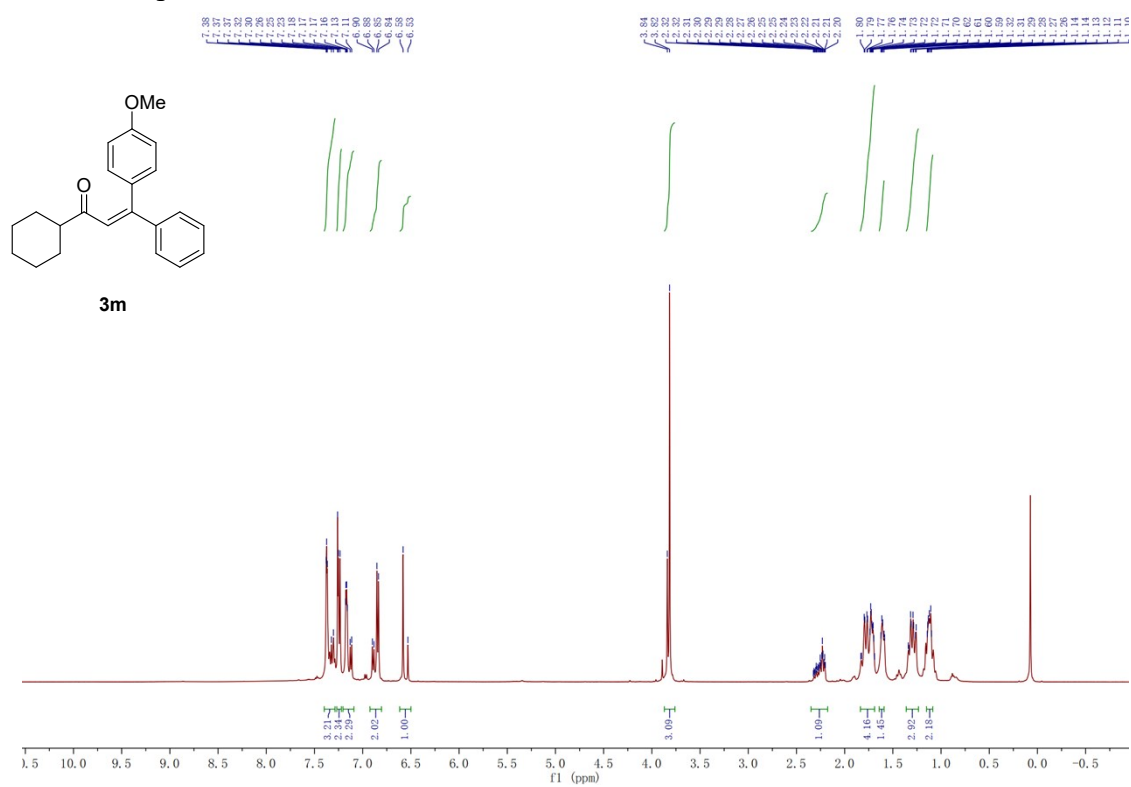
^1H NMR spectrum of **31** in CDCl_3 at 500 MHz



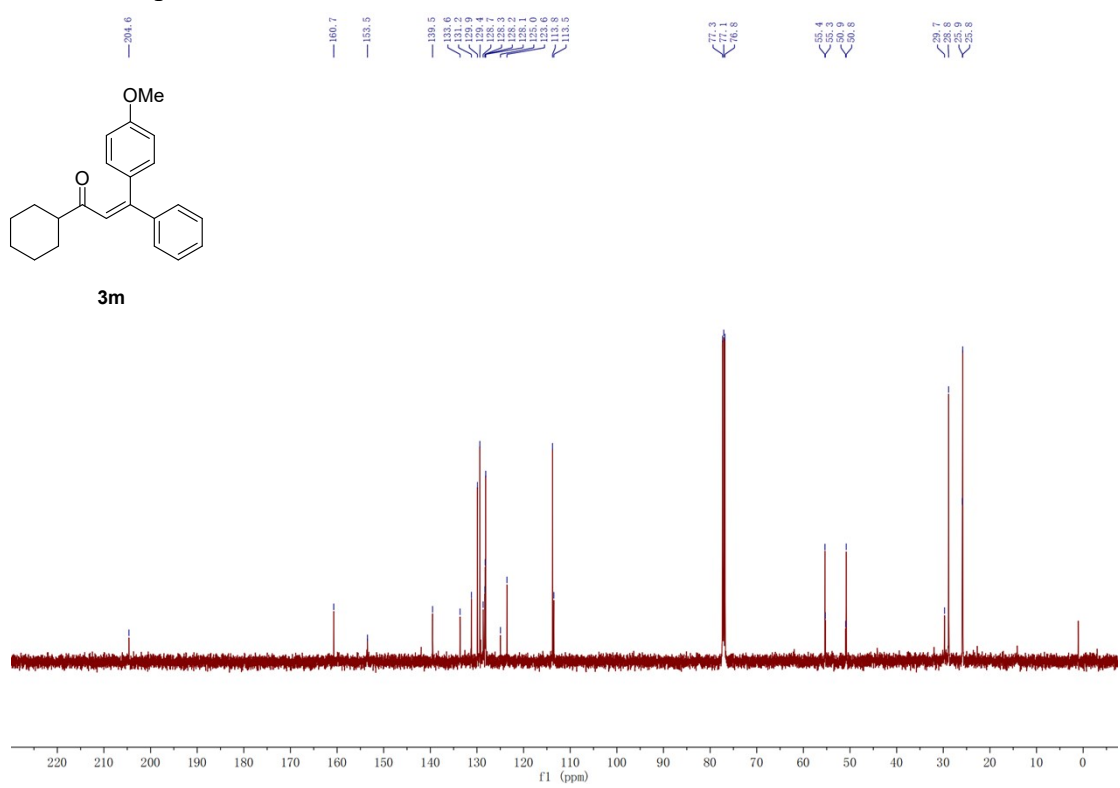
^{13}C NMR spectrum of **31** in CDCl_3 at 126 MHz



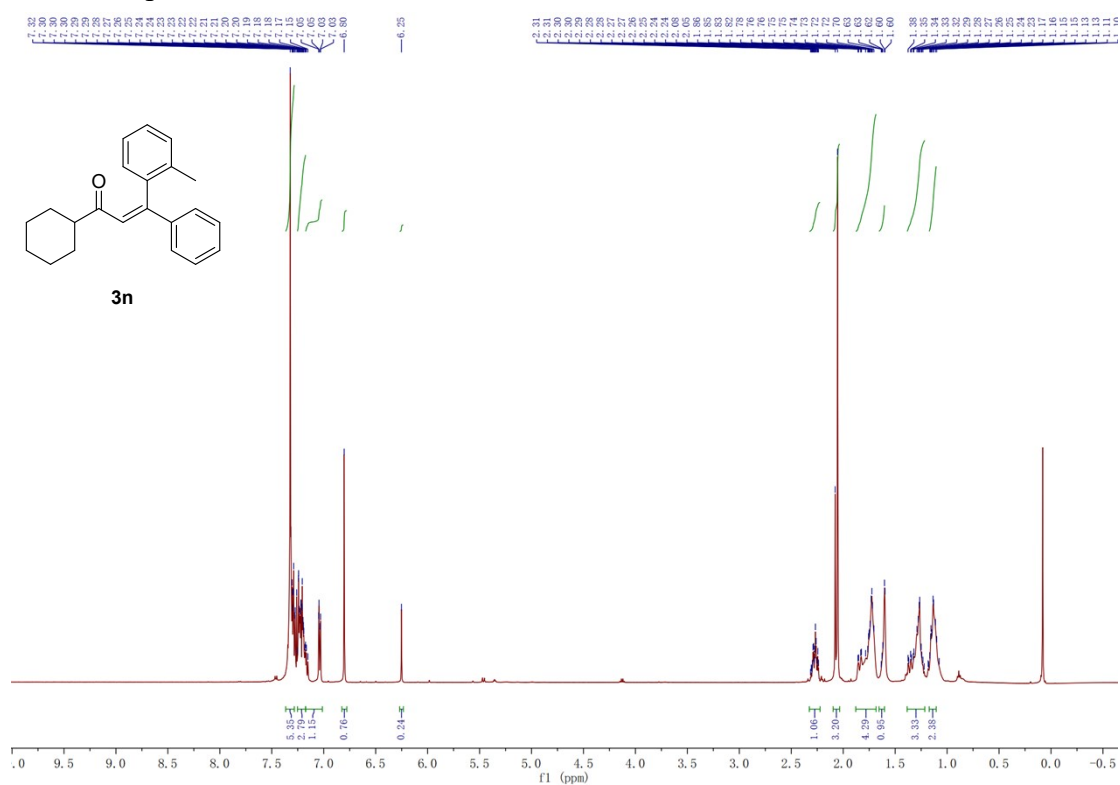
^1H NMR spectrum of **3m** in CDCl_3 at 500 MHz



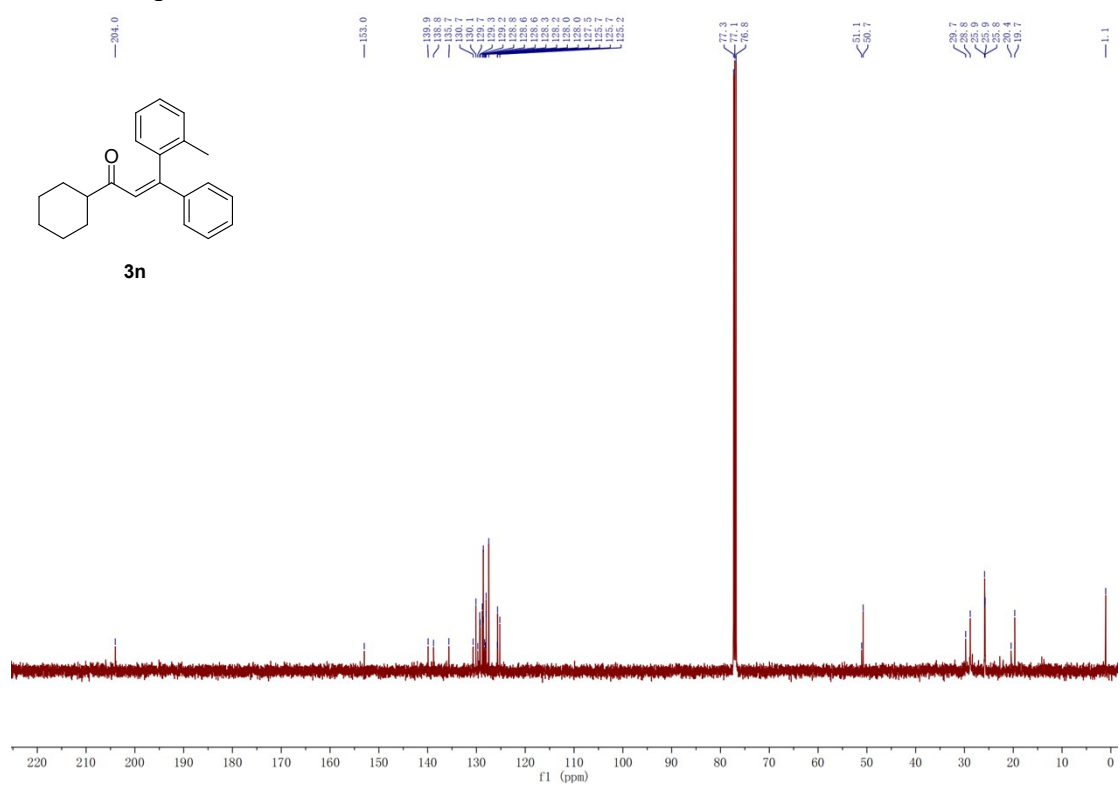
^{13}C NMR spectrum of **3m** in CDCl_3 at 126 MHz



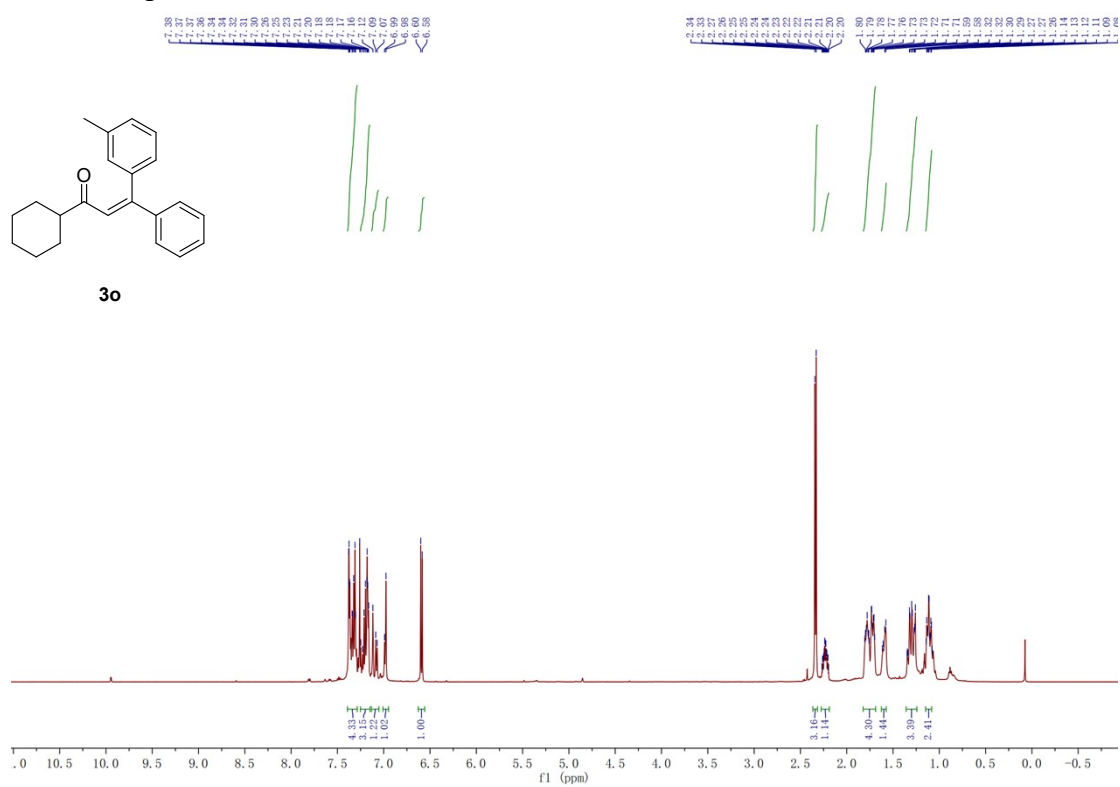
^1H NMR spectrum of **3n** in CDCl_3 at 500 MHz



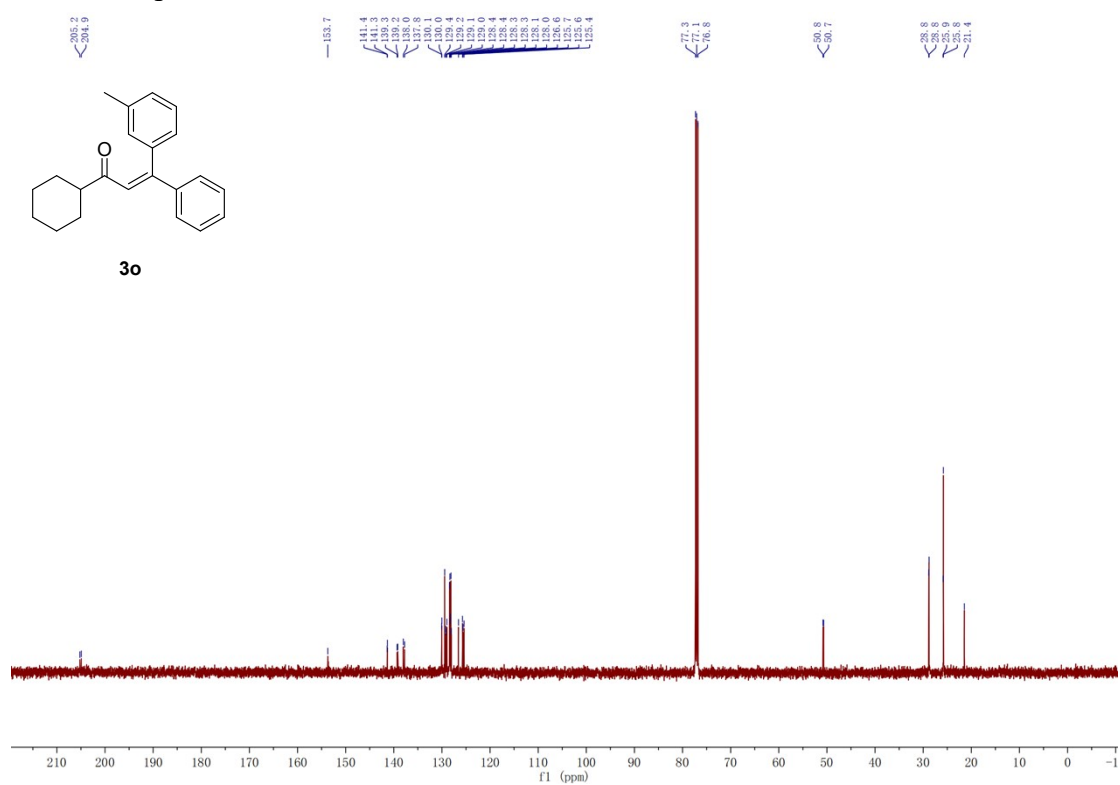
^{13}C NMR spectrum of **3n** in CDCl_3 at 126 MHz



^1H NMR spectrum of **3o** in CDCl_3 at 500 MHz



^{13}C NMR spectrum of **3o** in CDCl_3 at 126 MHz

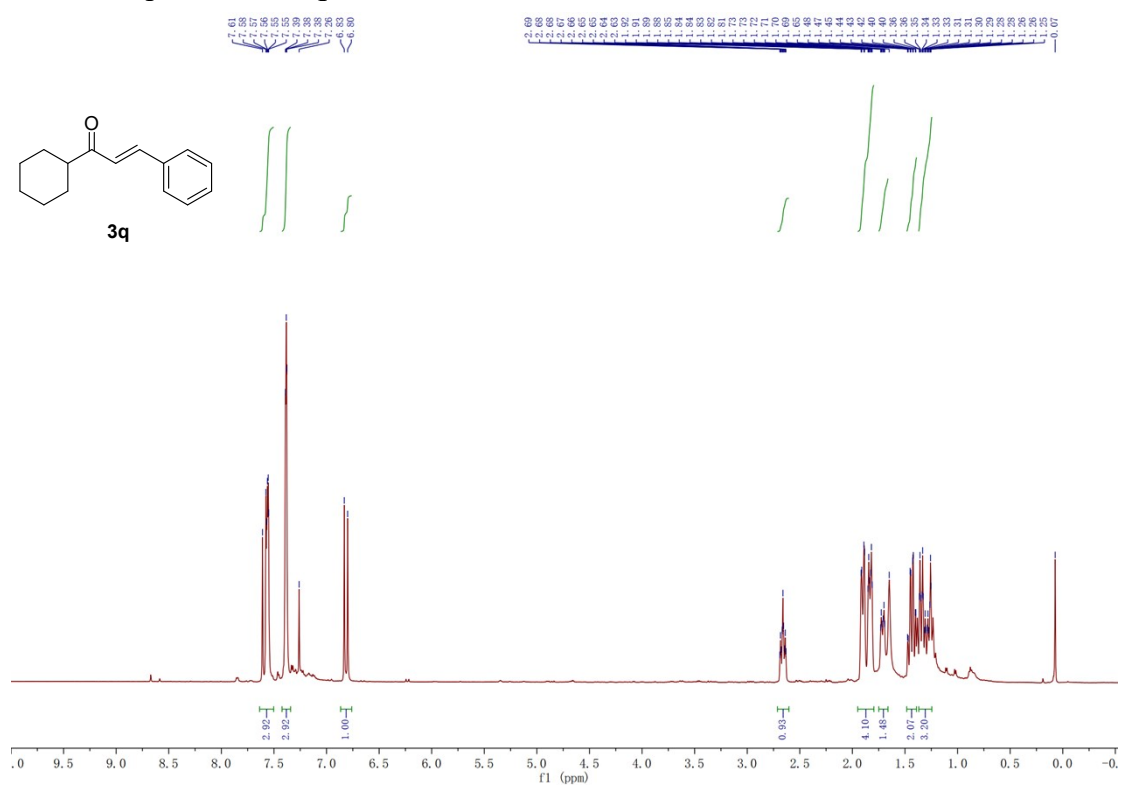


[illegible]

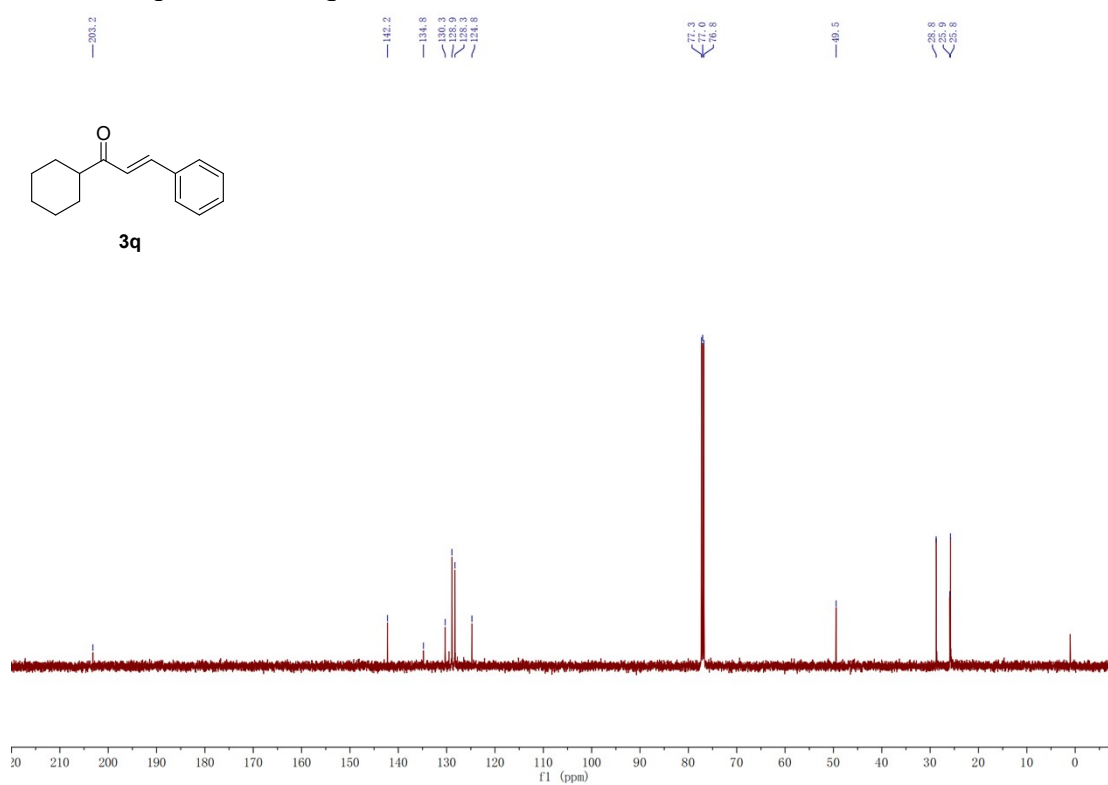
3p

¹³C NMR spectrum (ppm) of compound **3p**. The spectrum shows peaks corresponding to the chemical structure, including aromatic and aliphatic carbons. Key peaks are labeled with their chemical shifts: 204.8, 158.6, 141.6, 139.9, 138.9, 138.5, 136.2, 135.2, 133.9, 133.5, 132.9, 132.7, 132.0, 131.1, 130.9, 130.5, 129.5, 128.8, 128.4, 126.2, 125.3, 124.8, 124.2, 77.3, 77.1, 76.9, 50.9, 50.5, 29.8, 29.5, 29.2, 28.9, 28.6, 28.3, 28.0, 27.7, 27.4, 27.1, 26.8, 26.5, 26.2, 25.9, 25.6, 25.3, 25.0, 24.7, 24.4, 24.1, 23.8, 23.5, 23.2, 22.9, 22.6, 22.3, 22.0, 21.7, 21.4, 21.1, 20.8, 20.5, 20.2, 19.9, 19.6, 19.3, 19.0, 18.7, 18.4, 18.1, 17.8, 17.5, 17.2, 16.9, 16.6, 16.3, 16.0, 15.7, 15.4, 15.1, 14.8, 14.5, 14.2, 13.9, 13.6, 13.3, 13.0, 12.7, 12.4, 12.1, 11.8, 11.5, 11.2, 10.9, 10.6, 10.3, 10.0, 9.7, 9.4, 9.1, 8.8, 8.5, 8.2, 7.9, 7.6, 7.3, 7.0, 6.7, 6.4, 6.1, 5.8, 5.5, 5.2, 4.9, 4.6, 4.3, 4.0, 3.7, 3.4, 3.1, 2.8, 2.5, 2.2, 1.9, 1.6, 1.3, 1.0, 0.7, 0.4, 0.1, -0.2, -0.5, -0.8, -1.1, -1.4, -1.7, -2.0, -2.3, -2.6, -2.9, -3.2, -3.5, -3.8, -4.1, -4.4, -4.7, -5.0, -5.3, -5.6, -5.9, -6.2, -6.5, -6.8, -7.1, -7.4, -7.7, -8.0, -8.3, -8.6, -8.9, -9.2, -9.5, -9.8, -10.1.

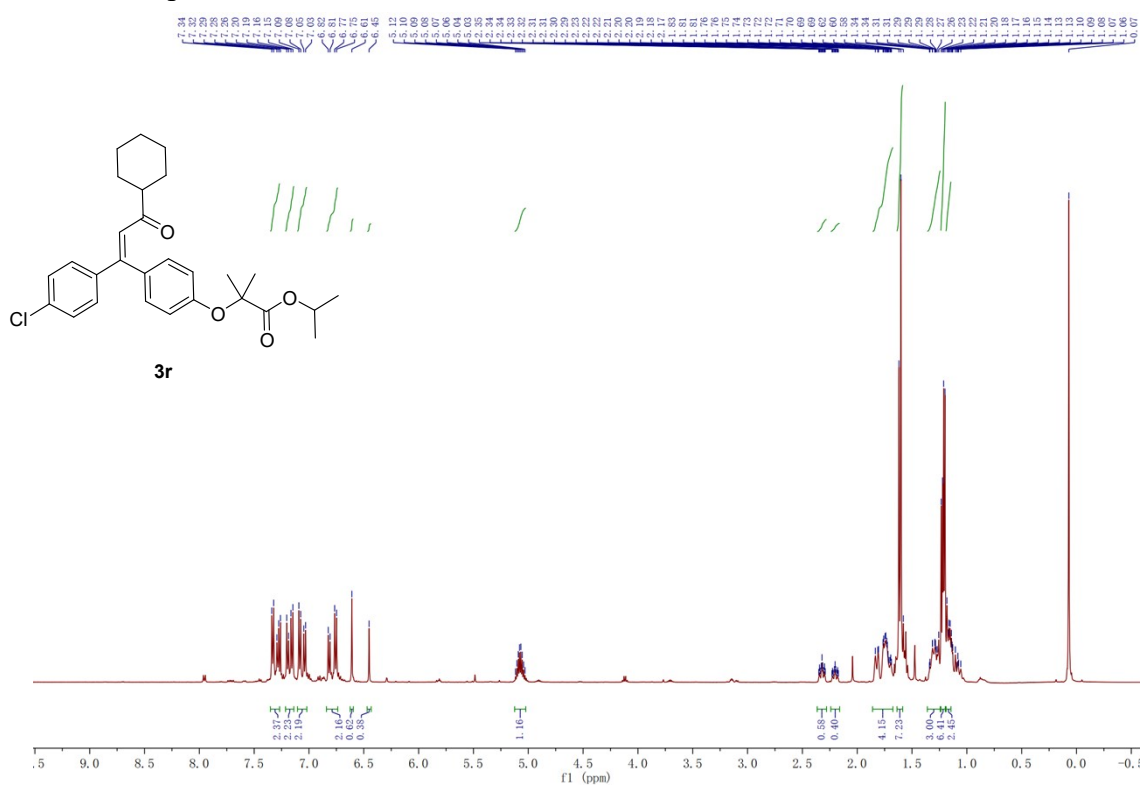
^1H NMR spectrum of **3q** in CDCl_3 at 500 MHz



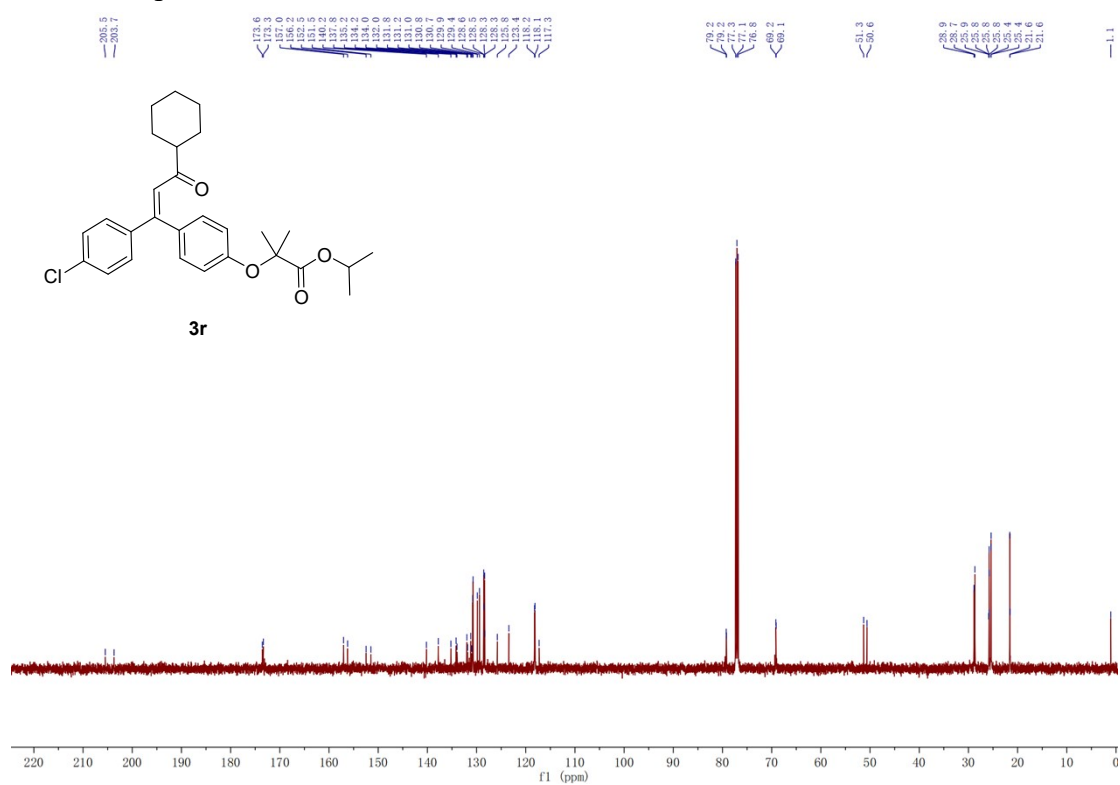
^{13}C NMR spectrum of **3q** in CDCl_3 at 126 MHz



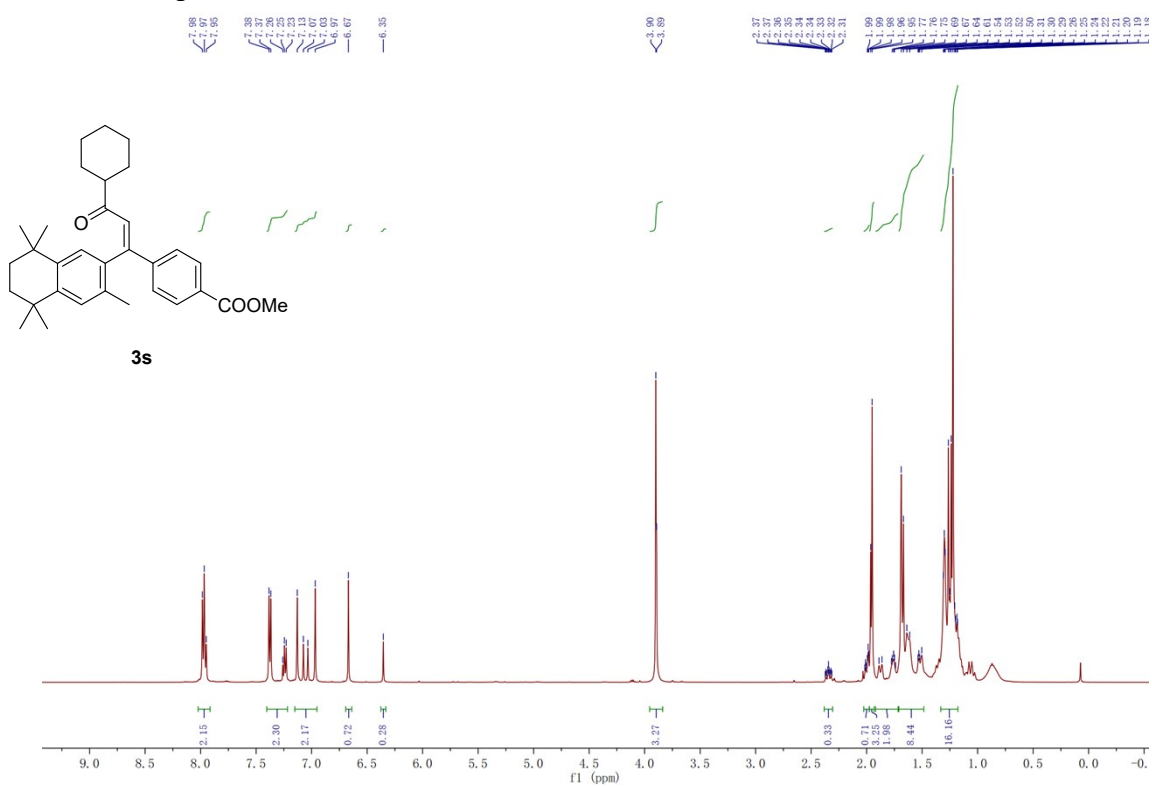
^1H NMR spectrum of **3r** in CDCl_3 at 500 MHz



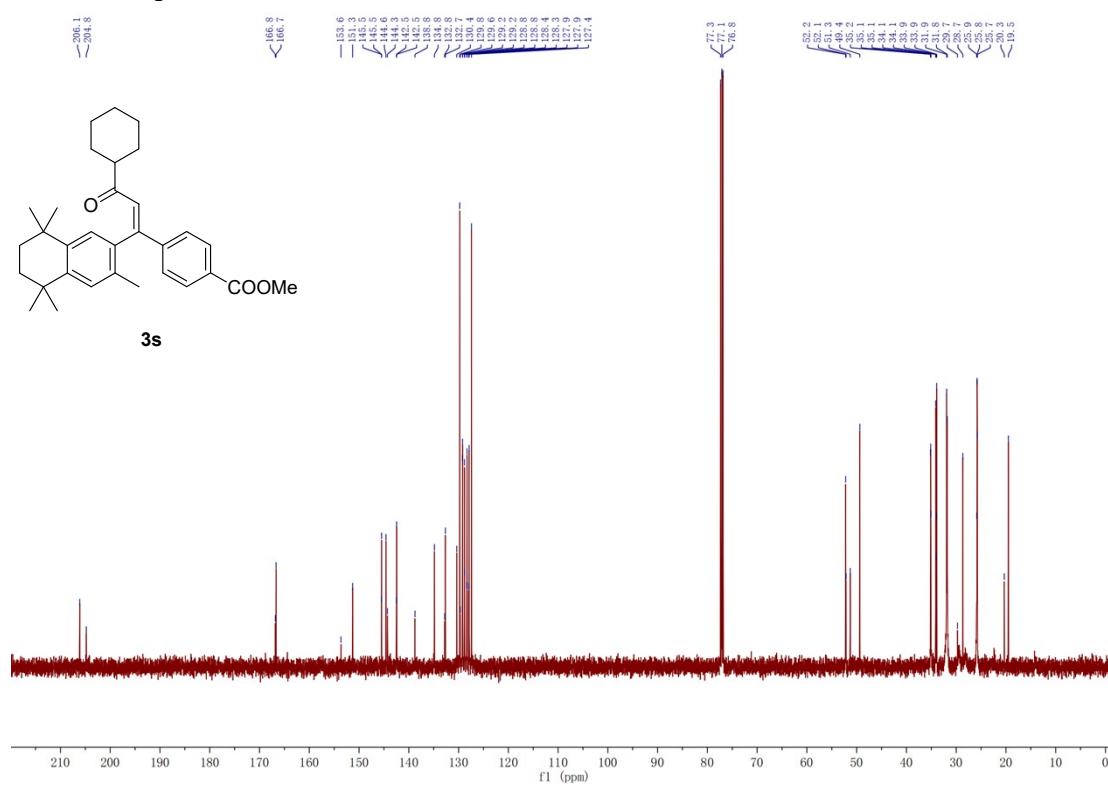
^{13}C NMR spectrum of **3r** in CDCl_3 at 126 MHz



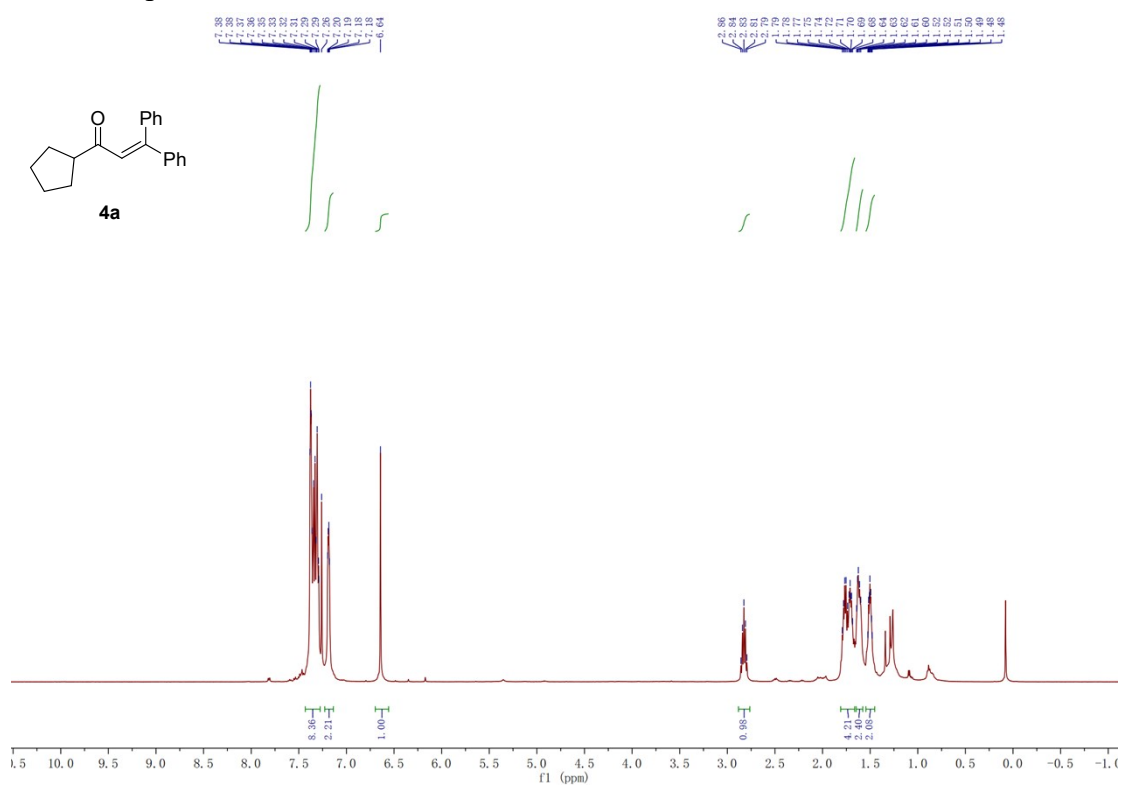
^1H NMR spectrum of **3s** in CDCl_3 at 500 MHz



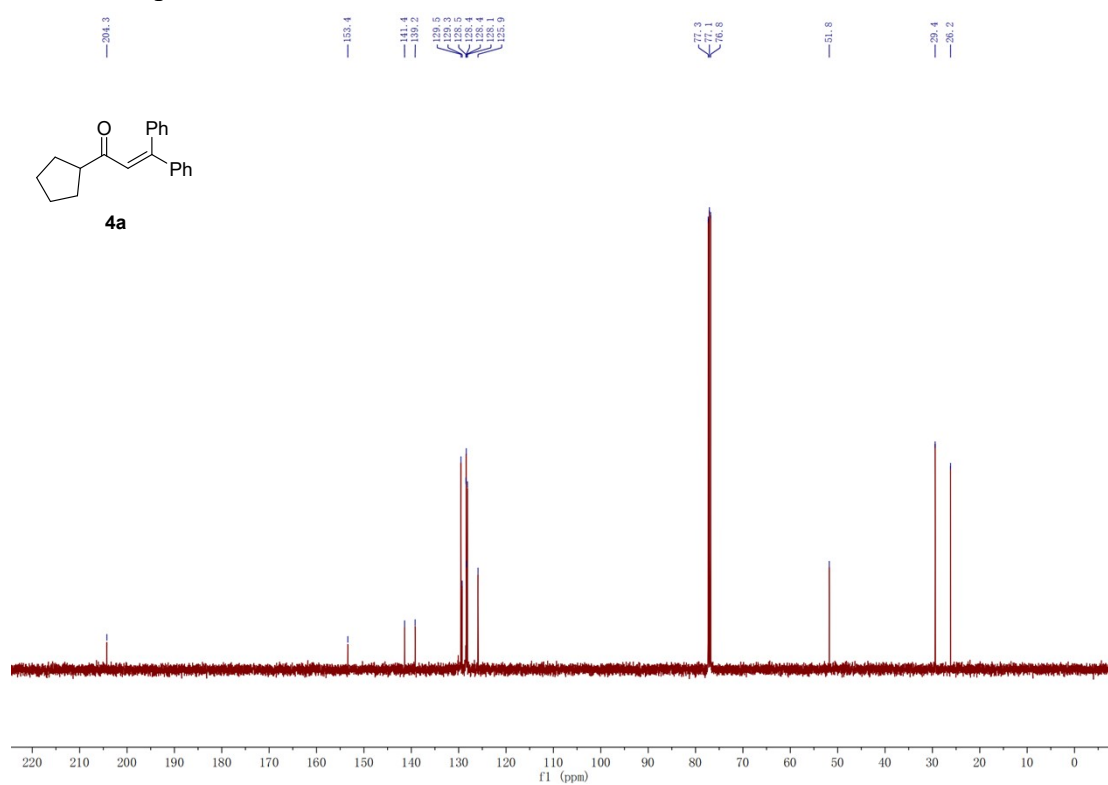
^{13}C NMR spectrum of **3s** in CDCl_3 at 126 MHz



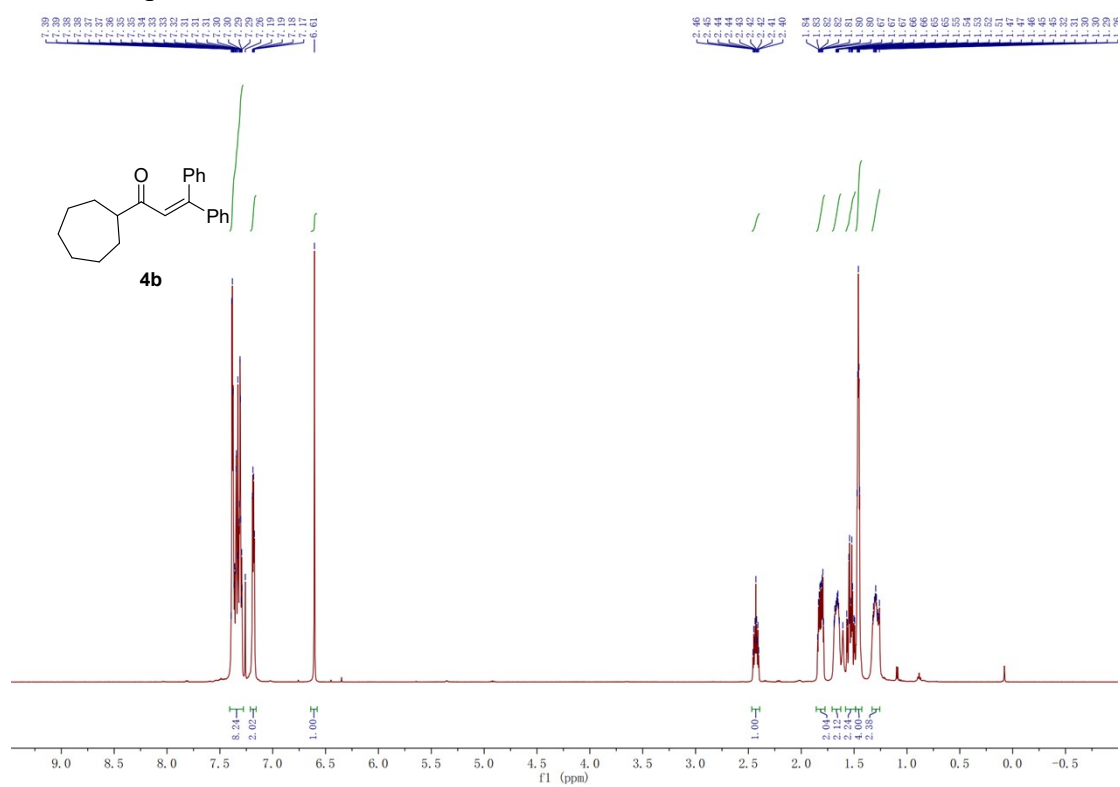
^1H NMR spectrum of **4a** in CDCl_3 at 500 MHz



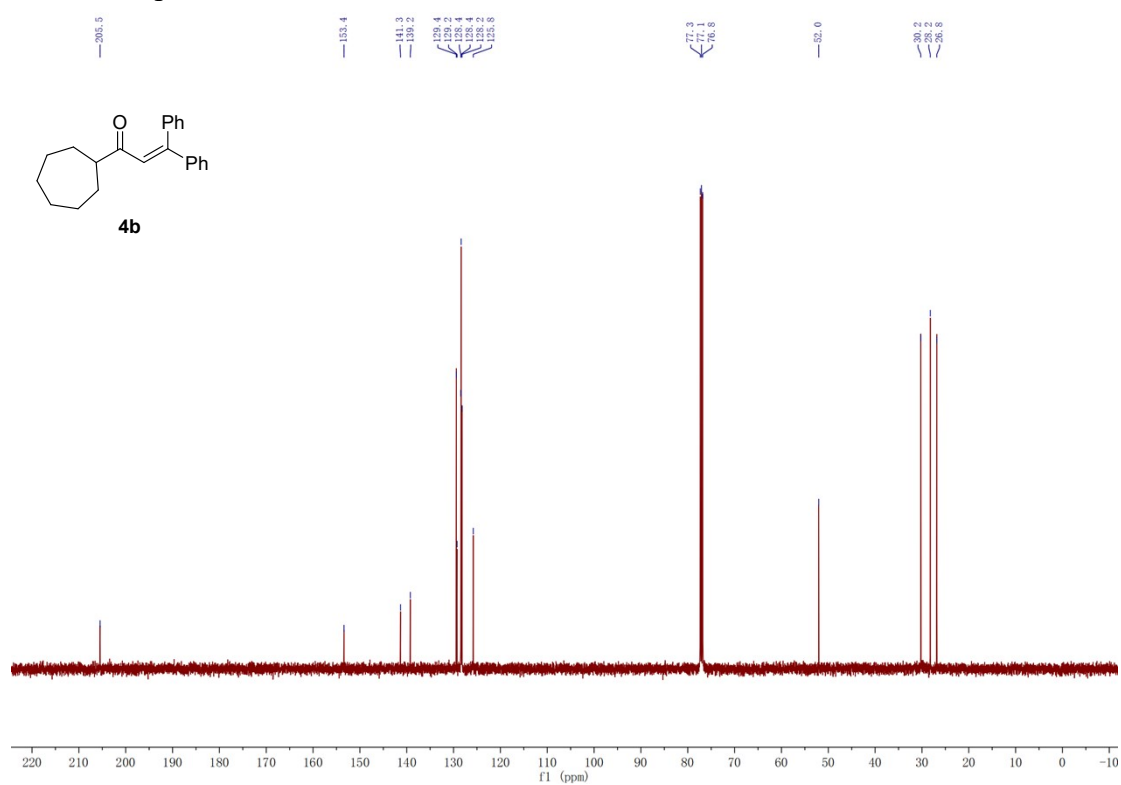
^{13}C NMR spectrum of **4a** in CDCl_3 at 126 MHz



^1H NMR spectrum of **4b** in CDCl_3 at 500 MHz



^{13}C NMR spectrum of **4b** in CDCl_3 at 126 MHz



4c

O=C1CCCCCCC1C=Cc2ccccc2

1H NMR spectrum (CDCl₃) of compound **4c**. The x-axis represents the chemical shift in ppm, ranging from -0.5 to 9.0. The spectrum shows several peaks, with integration values indicated below the baseline. The integration values are: 1.00 (aromatic region, 7.2-7.6 ppm), 1.01 (vinyl region, 6.4-6.7 ppm), and 1.00 (cyclooctanone ring region, 1.2-2.0 ppm).

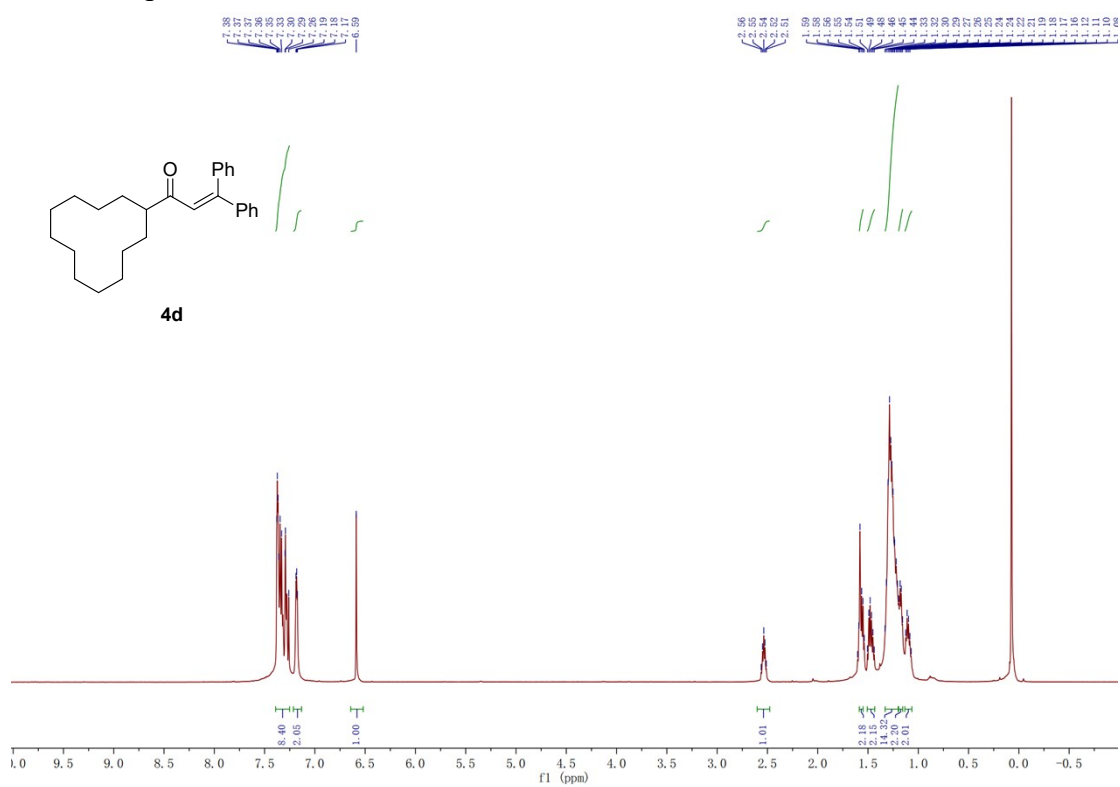
4c

O=C1CCCCCCC1/C=C/c2ccccc2

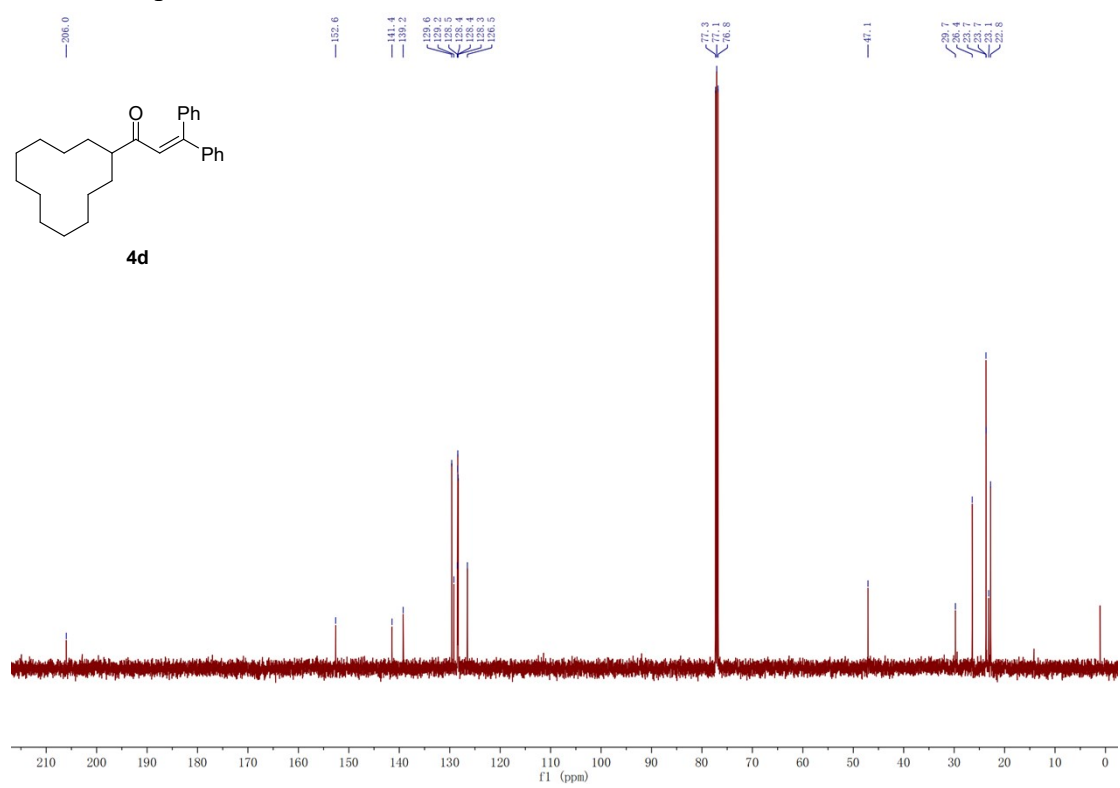
13C NMR spectrum (ppm):

- 206.0
- 153.1
- 141.3
- 139.3
- 129.5
- 129.2
- 128.2
- 128.4
- 128.3
- 128.1
- 77.3
- 77.1
- 76.8
- 50.1
- 28.7
- 28.4
- 28.6

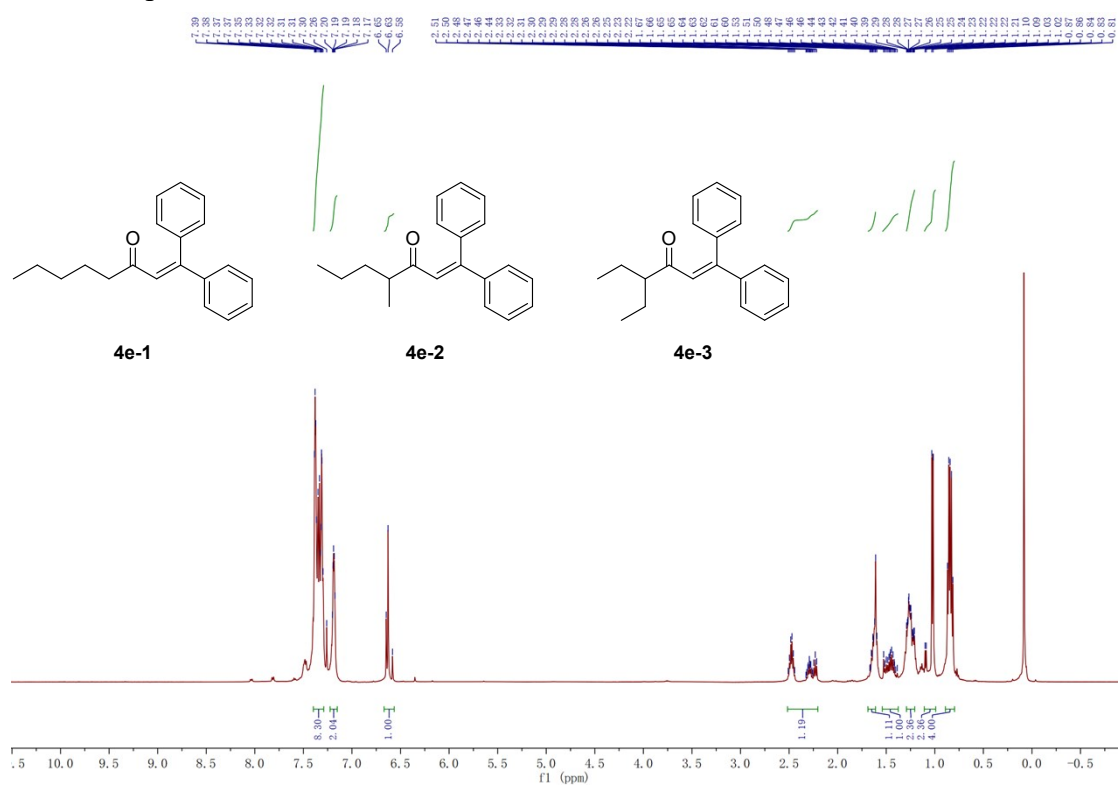
^1H NMR spectrum of **4d** in CDCl_3 at 500 MHz



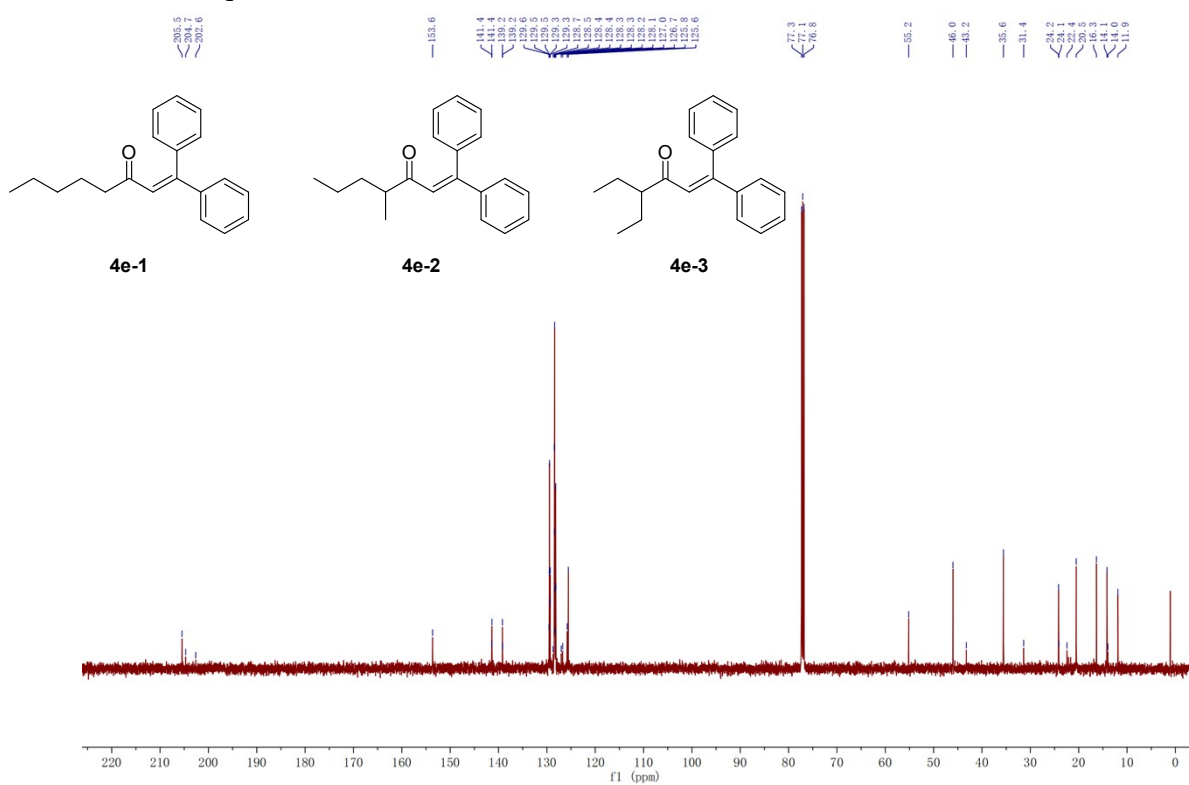
^{13}C NMR spectrum of **4d** in CDCl_3 at 126 MHz



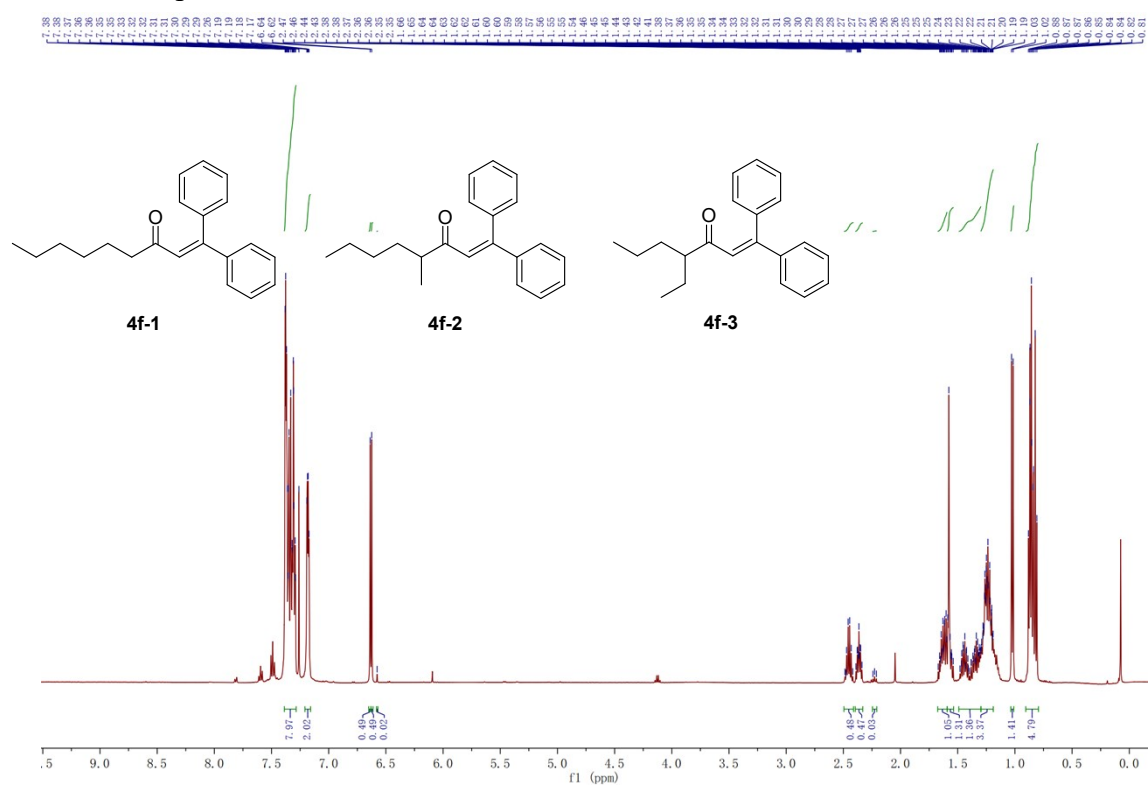
^1H NMR spectrum of **4e** in CDCl_3 at 500 MHz



^{13}C NMR spectrum of **4e** in CDCl_3 at 126 MHz



^1H NMR spectrum of **4f** in CDCl_3 at 500 MHz



^{13}C NMR spectrum of **4f** in CDCl_3 at 126 MHz

