Supporting Information for

Iron-Catalyzed α-C–H functionalization of π-Bonds: Cross-Dehydrogenative Coupling and Mechanistic Insights

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Contents

General Information:

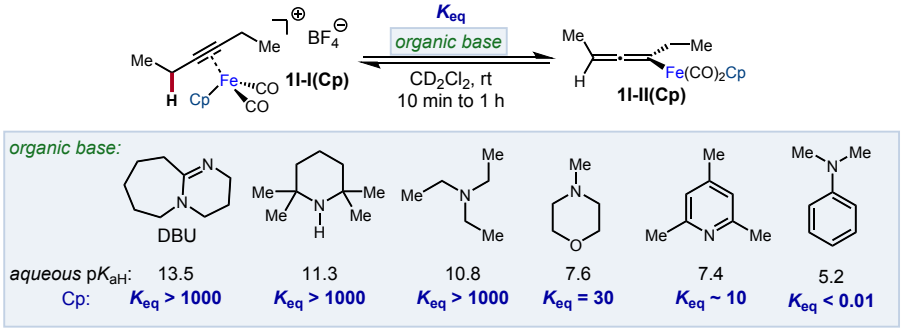
General Reagent Information: Anhydrous tetrahydrofuran, 1,2-dichloroethane, and trifluorotoluene were purchased from Acros (AcroSeal packaging), Sigma Aldrich (Sure/Seal packaging), and Frontier Scientific (J&KSeal packaging), respectively, and were transferred into an argon-filled glovebox and used as received. Other dry solvents were obtained by distillation and storage over 4Å molecular sieves. Triphenylcarbenium tetrafluoroborate ($Ph_3C^+BF_4^-$) was purchased from Alfa Aesar and stored in an argon-filled glove box. All other reagents were purchased from Oakwood, Acros, Alfa Aesar, or Sigma Aldrich and used as received. Compounds were purified by flash column chromatography using SiliCycle *SiliaFlash® F60* silica gel, unless otherwise indicated.

General Analytical Information: New compounds were characterized by ^{1}H NMR, ^{13}C NMR and HRMS. Copies of the ^{1}H NMR and ^{13}C NMR spectra can be found at the end of the Supporting Information. ^{1}H and ^{13}C NMR spectra were recorded on Bruker 400 MHz or 500 MHz instruments. All ^{1}H NMR data are reported in δ units, parts per million (ppm), and were measured relative to the residual proton signal in the deuterated solvent at 7.26 ppm ($CDCl_3$) or 5.32 ppm (CD_2Cl_2). All ^{13}C NMR spectra are ^{1}H decoupled and reported in ppm relative to the solvent signal at 77.16 ppm ($CDCl_3$) or 53.84 ppm (CD_2Cl_2). Thin-layer chromatography (TLC) was performed on Silicycle 250 μm (analytical) or 1000 μm (preparative) silica gel plates. Compounds were visualized by irradiation with UV light, or by staining with iodine/silica gel, potassium permanganate, or phosphomolybdic acid (PMA). Yields refer to isolated compounds, unless otherwise indicated. High resolution mass spectra were recorded on a Thermo Scientific Q-Exactive mass spectrometer.

NMR yield was determined by using 1,1,2,2-tetrachloroethane as internal standard for ^{1}H spectroscopy and using $CDCl_3$ as internal standard for ^{2}H spectroscopy.

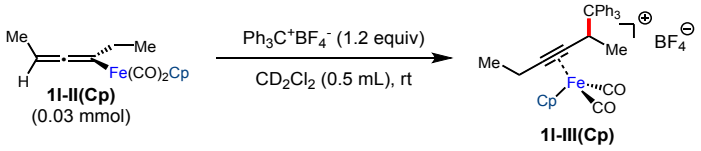
Mechanistic experiments

Stoichiometric experiments for Cp-based complexes.

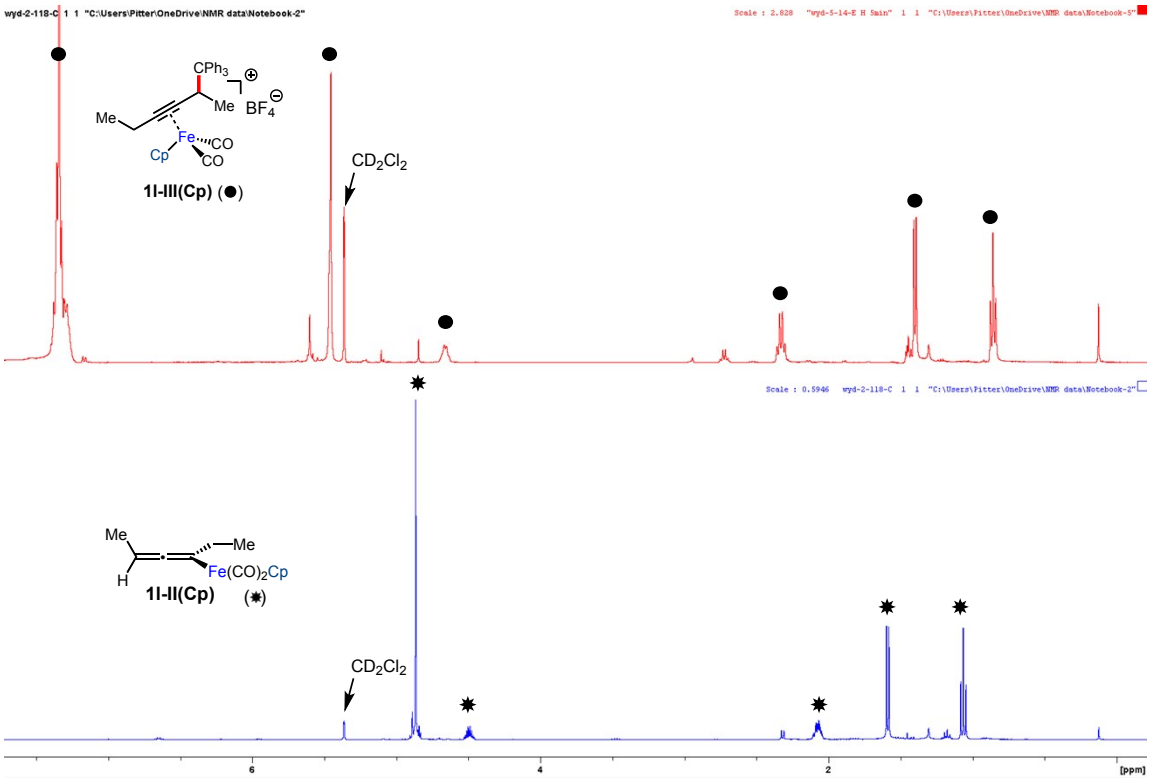


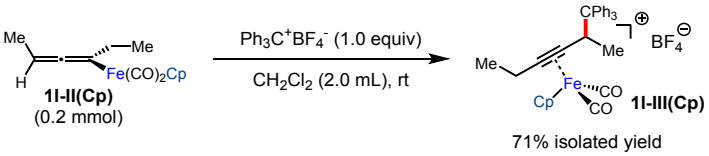
In an argon-filled glovebox, an NMR tube was charged with **1l-I(Cp)** (0.029 mmol, 10 mg) and CD_2Cl_2 (0.5 mL), then the base (1.0 equiv) was added into the above solution. The NMR tube was capped and removed from the glovebox and shaken by hand. ^{1}H NMR spectra were then recorded after 5 min, 10 min, 20 min, 30 min, 1 h, and 2 h. The position of the equilibrium was determined by integration of the Cp signal of the cationic and neutral Fp-alkyne complexes. Equilibrium was established within 1 h for all bases examined.

Note: The K_{eq} of *sym*-collidine is not determined because **1l-I(Cp)** was transformed to the by-product for which we have so far not been able to assign a definitive structure.



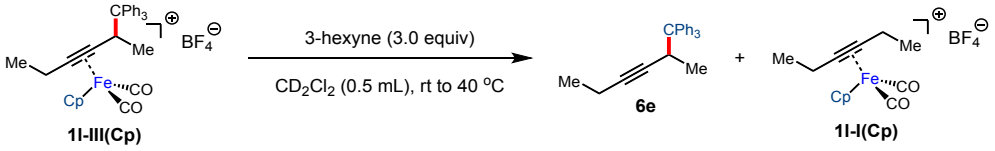
In an argon-filled glovebox, **1l-II(Cp)**, prepared from **1l-I(Cp)** (approx. 0.03 mmol), was dissolved in CD_2Cl_2 (0.5 mL) and added to an NMR tube charged with $Ph_3C^+BF_4^-$ (12 mg, 1.2 equiv). The NMR tube was capped and removed from the glovebox and shaken by hand. ^{1}H NMR spectra were acquired after 5 min.





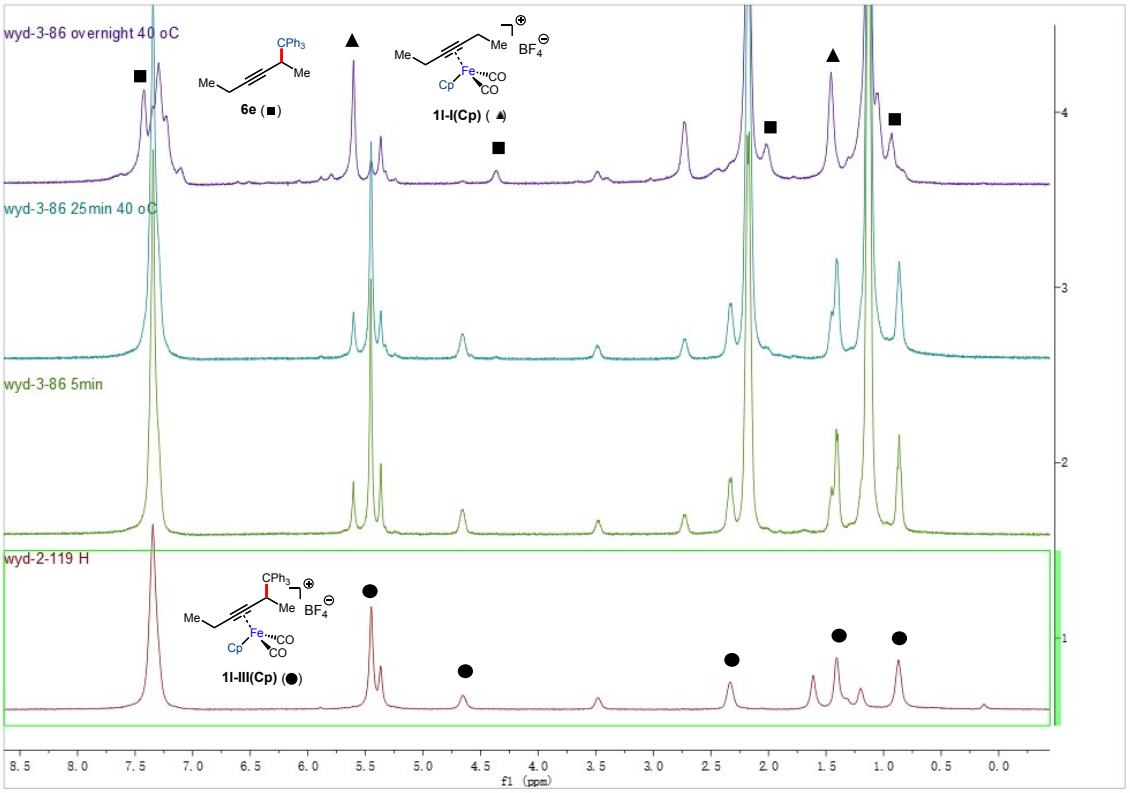
71% isolated yield

The Fe complex **1l-III (Cp)** is stable to collect for ^{1}H NMR and HRMS. To a solution of **1l-II(Cp)** prepared from above (approx. 0.2 mmol) in dry CH_2Cl_2 (2 mL) was added $Ph_3C^+BF_4^-$ (66 mg, 0.2 mmol, 1.0 equiv.) as a solid in the argon-filled glove box. The mixture was stirred under N_2 at room temperature for 1.5 h. Then dry diethyl ether (5 mL) was added to the above solution. The desired product precipitated as an orange solid, which was collected on a frit, and further dried *in vacuo* (84 mg, 71% yield).

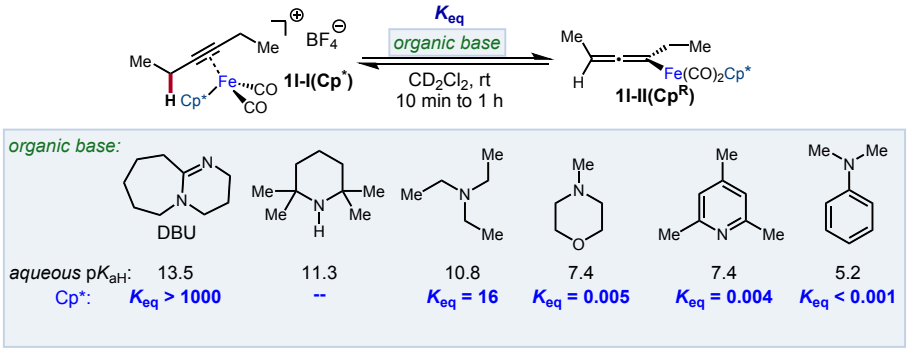


In an argon-filled glovebox, **1l-III(Cp)** (0.012 mmol, 7 mg) was added to an NMR tube and dissolved in CD_2Cl_2 (0.5 mL). Then 3-hexyne (0.036 mmol, 4 µL, 3 equiv) was added to the above solution. The NMR tube was capped and removed from the glovebox and shaken by hand. A ^{1}H NMR spectrum was acquired after 5 min. Then the NMR tube was placed in an oil bath, preheated to 40 °C, and another spectrum was acquired after 25 min, and then after 12 h. The alkyne exchange with Cp as the supporting ligand was still incomplete (80% conversion) after 12 hours. **1l-III(Cp)**/**6e** or **1l-III(Cp)**/ **1l-I(Cp)** = 1:4.

An experiment in which 3-hexyne was added directly to an NMR tube containing *in situ* generated **1l-III(Cp)** showed largely identical results (80% NMR yield of **6e** after 12 h at 40 °C). The allenyl complex **1l-II(Cp)** was observed after addition of Et_3N.



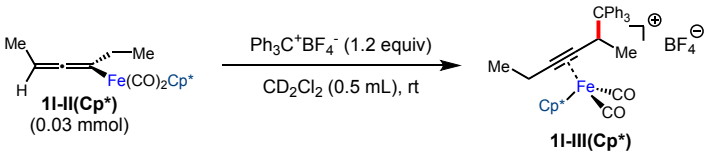
Stoichiometric experiments for Cp*-based complexes.



In an argon-filled glovebox, an NMR tube was charged with **1l-I(Cp*)** (0.024 mmol, 10 mg) and CD_2Cl_2 (0.5 mL), then the base (1.0 equiv) was added into the above solution. The NMR tube was capped and removed from the glovebox and shaken by hand. ^1H NMR spectra were then recorded after 5 min, 10 min, 20 min, 30 min, 1 h, and 2h. The

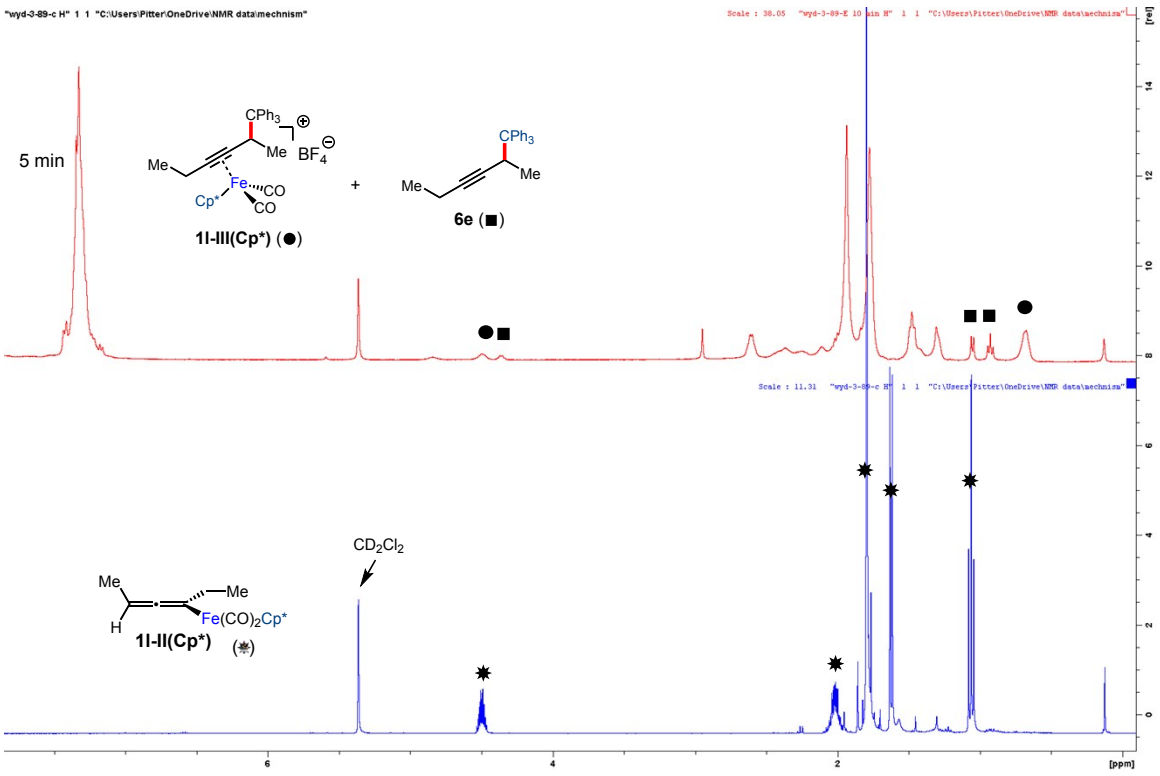
position of the equilibrium was determined by integration of the Cp signal of the cationic and neutral Fp-alkyne complexes. Equilibrium was established within 2h for all bases examined, except in the case of TMPH.

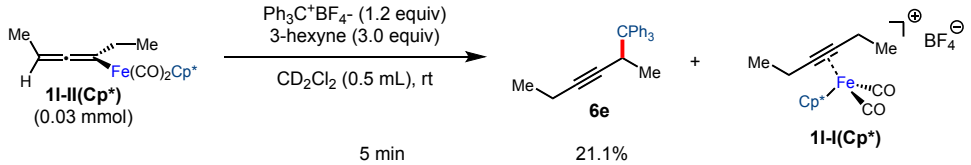
NOTE: The equilibrium constant for the deprotonation of **1l-I(Cp*)** by TMPH could not be determined, as equilibrium was not reached even after 24 h and the NMR sample had started to undergo decomposition.



In an argon-filled glovebox, **1l-II(Cp*)** (0.03 mmol) was dissolved in CD_2Cl_2 (0.5 mL) and added to an NMR tube containing $Ph_3C^+BF_4^-$ (12 mg, 1.2 equiv). The NMR tube was removed from the glovebox and shaken by hand. ^1H NMR spectra were acquired in 5 min. It showed the reaction was done determined by comparing the position of CH_3 groups in spectrum.

NOTE: The Fe complex **1l-III(Cp*)** is not stable and will decompose to the product **6e** slowly.





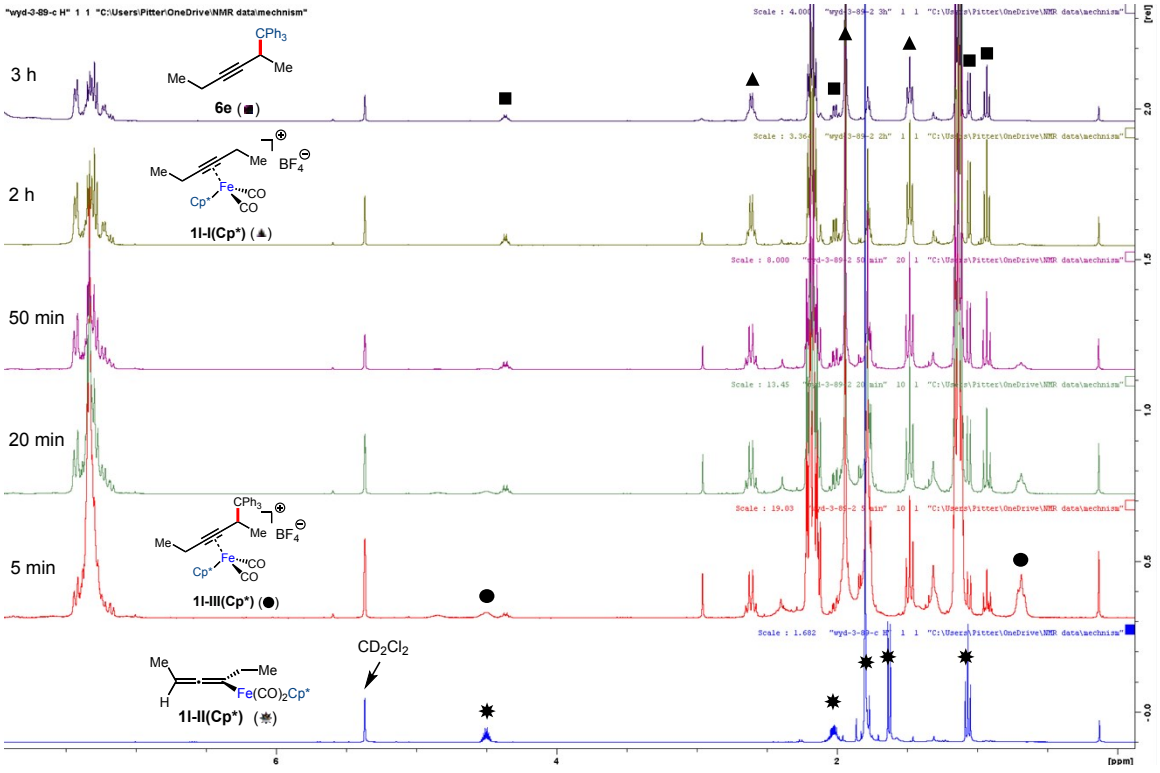
5 min	21.1%
20 min	42.7%
50 min	62.5%
2 h	83.3%
3 h	100%

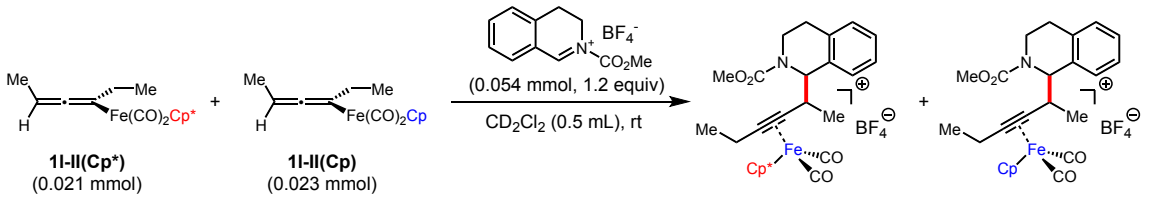
Because of the instability of Fe complex **1l-III(Cp*)**, we conduct the alkyne-exchange experiments in one pot. The procedure is as following:

In an argon-filled glovebox, **1l-II(Cp*)** (0.03 mmol) was dissolved in CD_2Cl_2 (0.5 mL) and added to an NMR tube containing $Ph_3C^+BF_4^-$ (12 mg, 1.2 equiv) and 3-hexyne (10 μL, 0.09 mmol, 3.0 equiv). The NMR tube was removed from the glovebox and shaken by hand. ^1H NMR spectra were acquired after 5 min, 20 min, 50 min, 2 h and 3 h.

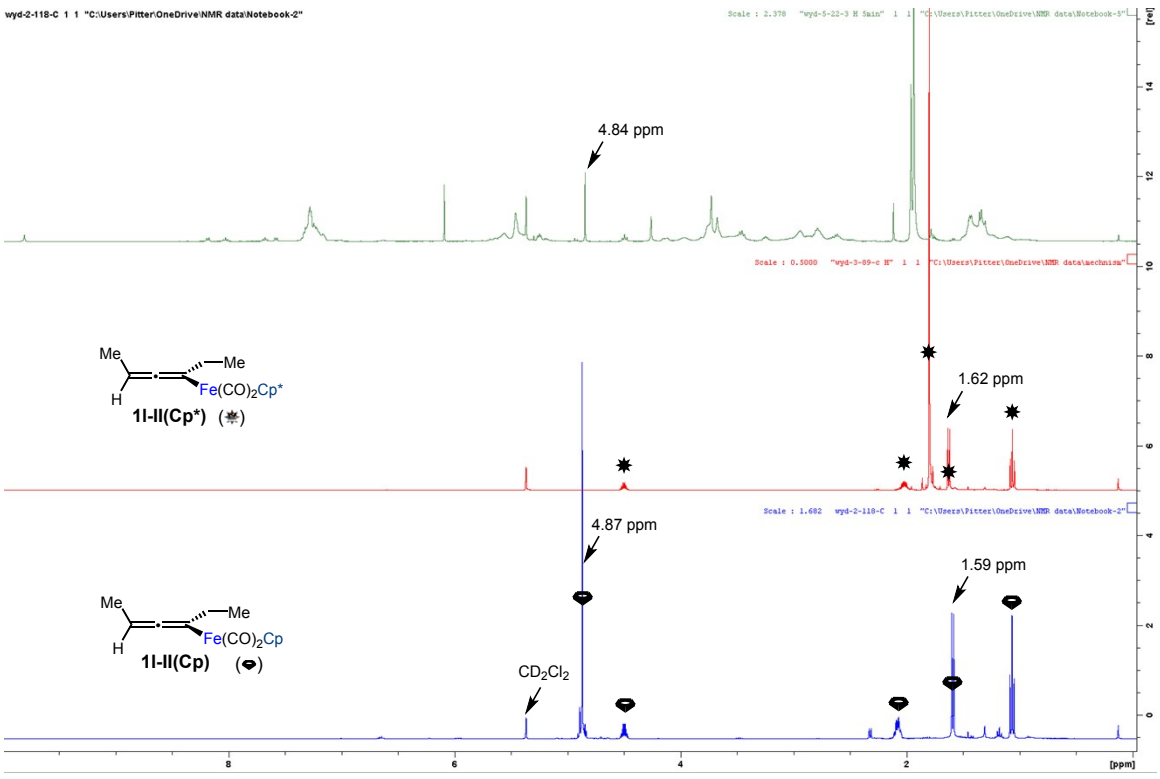
In contrast to the sluggish alkyne exchange for **1l-III(Cp)** (incomplete even after 12 h at 40 °C), the exchange of 3-hexyne with **1l-III(Cp*)** took place readily at room temperature, and was complete after 3 h.

These observations are consistent with rapid irreversible functionalization and rate-determining deprotonation observed for the optimized catalytic system.

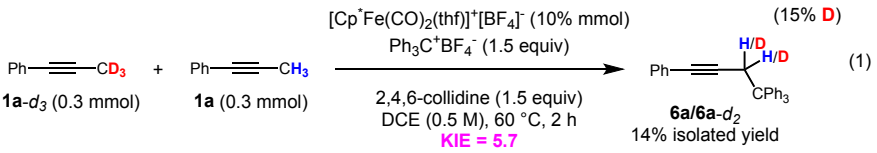




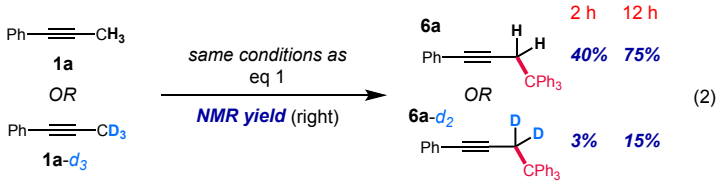
In an argon-filled glovebox, **1l-II(Cp*)** (0.021 mmol) and **1l-II(Cp)** (0.023 mmol) was dissolved in CD_2Cl_2 (0.5 mL) and added to an NMR tube containing $Ph_3C^+BF_4^-$ (15 mg, 0.054 mmol, 1.2 equiv). The NMR tube was removed from the glovebox and shaken by hand. ^1H NMR spectra were acquired in 5 min and 10 min. The CH_3 groups of **1l-II(Cp*)** and **1l-II(Cp)** are well-resolved as doublets in the ^1H spectra. The spectrum of product is complicated because of the presence of rotamers and diastereomers. Thus, we are unable to determine the NMR yield of the products based on the spectrum. However, after 5 min, **1l-II(Cp*)** could no longer be observed, while a trace of **1l-II(Cp)** remained. After 10 min, **1l-II(Cp)** could no longer be observed.



Kinetic isotope effect experiments

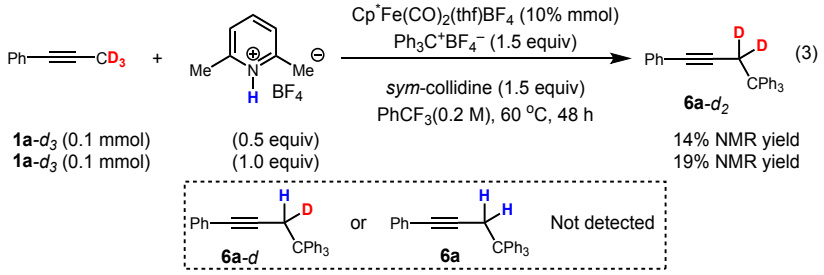


In an argon-filled glovebox, Cp*Fe(CO)$_2$(thf)$^+$BF$_4^-$ (24.4 mg, 10 mol%), Ph$_3$C$^+$BF$_4^-$ (300 mg, 0.9 mmol, 1.5 equiv), dry 1,2-dichloroethane (1.2 mL), **1a** (39 µL, 0.3 mmol, 1.0 equiv), **1a**-d_3 (36 mg, 0.3 mmol, 1.0 equiv) and 2,4,6-collidine (120 µL, 0.9 mmol, 1.5 equiv) were added to a flame-dried screw-cap reaction tube (13 mm × 100 mm, Fisherbrand, part # 14-959-35C) in rapid succession. The reaction tube was capped with a Teflon/silicone septum (Thermo/National part # C4015-66A) screw cap and removed from the glovebox. The reaction tube was then placed in an oil bath, preheated to 60 °C, where it was stirred for 2 h. The crude mixture was concentrated *in vacuo* and purified by flash column chromatography to provide a mixture of **6a** and **6a**-d_2 (20% NMR yield, 85:15 ratio, 31 mg, 14% yield).

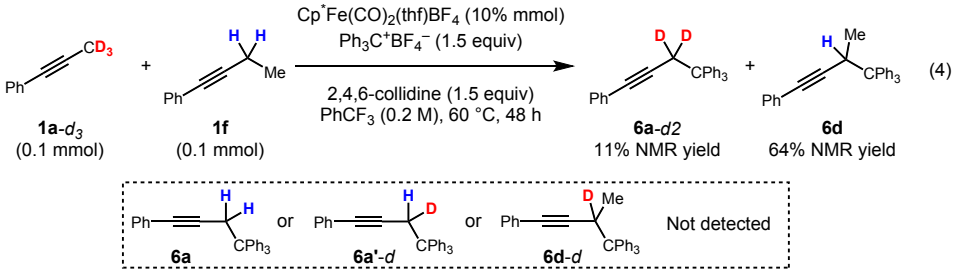


In an argon-filled glovebox, Cp*Fe(CO)$_2$(thf)$^+$BF$_4^-$ (10 mol %, 12.2 mg), Ph$_3$C$^+$BF$_4^-$ (150 mg, 0.45 mmol, 1.5 equiv), dry 1,2-dichloroethane (0.6 mL), **1a** (39 µL, 0.3 mmol, 1.0 equiv) or **1a**-d_3 (36 mg, 0.3 mmol, 1.0 equiv) and 2,4,6-collidine (60 µL, 0.45 mmol, 1.5 equiv) were added to a flame-dried screw-cap reaction tube (13 mm × 100 mm, Fisherbrand, part # 14-959-35C) in rapid succession. The reaction tube was capped with a Teflon/silicone septum (Thermo/National part # C4015-66A) screw cap and removed from the glovebox. The reaction tube was then placed in an oil bath, preheated to 60 °C, where it was stirred for 2 h or 12 h. The NMR yield was then determined by ^{1}H and ^{2}H NMR spectroscopy. We note that for **1a**-d_3, the reaction was sluggish and low isolated yield was obtained after 48 h. This is due to formation of the by-product of **1a**-d_3 and deactivation of the catalyst. Hence, absolute rate constant measurements could not be made. Nevertheless, the observation of a primary kinetic isotope effect by competition and by parallel experiments indicates that deprotonation is the turnover-limiting step of the catalytic cycle.

Isotope crossover/exchange experiments



In an argon-filled glovebox, Cp*Fe(CO)$_2$(THF)$^+$BF$_4^-$ (4.1 mg, 10 mol%), Ph$_3$C$^+$BF$_4^-$ (50 mg, 0.15 mmol, 1.5 equiv), dry trifluorotoluene(0.5 mL), **1a**-d_3 (0.1 mmol, 1.0 equiv) 2,6-lutidinium tetrafluoroborate (0.5 equiv or 1.0 equiv) and 2,4,6-collidine (20 µL, 0.15 mmol, 1.5 equiv) were added in rapid succession. The reaction tube was capped and removed from the glovebox. The reaction tube was then placed in an oil bath, preheated to 60 °C, where it was stirred for 48 h. The crude product was then analyzed by ^{1}H and ^{2}H NMR spectroscopy. In both cases, only **6a**-d_2 was observed, while **6a**-d and **6a** were not detected. *Note*: PhCF$_3$ was instead of 1,2-dichloroethane as the solvent in this experiment because signals for **6a** and DCE both appear around 3.7 ppm.

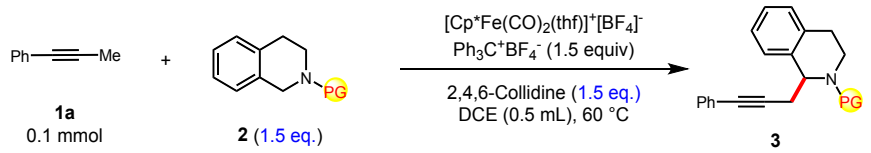


In an argon-filled glovebox, Cp*Fe(CO)$_2$(thf)$^+$BF$_4^-$ (8.2 mg, 10 mol %), Ph$_3$C$^+$BF$_4^-$ (100 mg, 0.3 mmol, 1.5 equiv.), dry trifluorotoluene (1.0 mL), **1a**-d_3 (0.1 mmol, 1.0 equiv), **1f** (0.1 mmol, 1.0 equiv), and 2,4,6-collidine (40 µL, 0.3 mmol, 1.5 equiv) were added to a screw-cap reaction tube in rapid succession. The reaction tube was capped and removed from the glovebox. The reaction tube was then placed in an oil bath, preheated to 60 °C, where it was stirred for 48 h. After cooling down to room temperature, the internal standard $CDCl_3$ was used for ^{2}H spectroscopy and the internal standard 1,1,2,2-tetrachloroethane was used for ^{1}H spectroscopy. None of the crossover products could be observed by ^{1}H or ^{2}H NMR spectroscopy. *Note*: PhCF$_3$ was instead of 1,2-dichloroethane as the solvent in this experiment because signals for **6a** and DCE both appear around 3.7 ppm.

The above two control experiments indicate that the deprotonation of alkyne is an irreversible process, consistent with the previous result that deprotonation is the turnover-limiting step.

Optimization of reaction conditions

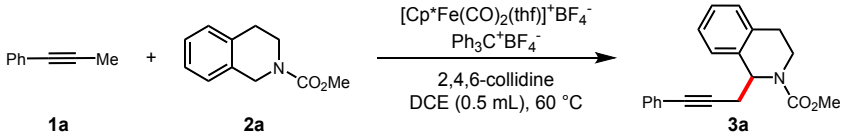
1. Optimization of the protecting group.



Entry	PG	NMR Yield (%) [b]
1	CO_2Me	55
2	CO_2Bn	40
3	Fmoc	43
4	Piv	31
5	$P(O)(OEt)_2$	No Product
6	Boc	No Product
7	$CF_3C(O)$	No Product

[a] All reactions were carried out with **1a** (0.1 mmol), **2** (1.5 equiv), $Ph_3C^+BF_4^-$ (1.5 equiv), 2,4,6-collidine (1.5 equiv), $Cp^*Fe(CO)_2(thf)BF_4$ (30 mol%) and dry DCE (0.5 mL) at 60 °C. [b] NMR yield was determined by using 1,1,2,2-tetrachloroethane as internal standard.

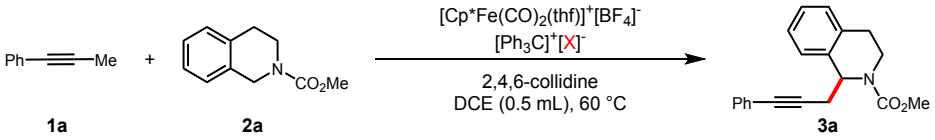
2. Optimization of reagent ratio.



Entry	**1a**: **2a**: Ph_3CBF_4: Collidine	NMR Yield (%) [b]
1	1.0:1.5:1.5:1.8	55
2	1.0:1.8:1.8:1.5	No Product
3	1.0:2.0:2.0:2.5	47
4	1.0:1.5:1.7:1.8	62
5	1.0:1.5:1.5:2.5	47
6	2.0:1.0:1.0:1.0	8
7	2.0:1.0:1.0:1.2	29

[a] All reactions were carried out with **1a** (0.1 mmol), **2a** (x equiv), $Ph_3C^+BF_4^-$ (x equiv), 2,4,6-collidine (x equiv), $Cp^*Fe(CO)_2(thf)BF_4$ (30 mol%) and dry DCE (0.5 mL) at 60 ˚C. [b] NMR yield was determined by using 1,1,2,2-tetrachloroethane as internal standard.

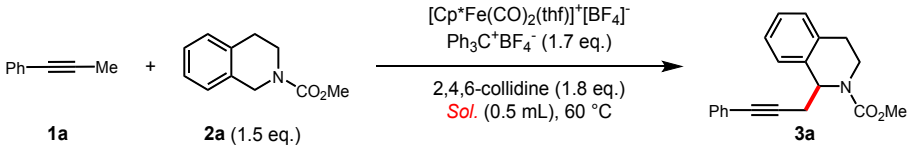
3. Optimization of the anion.



Entry	$Ph_3C^+X^-$	NMR Yield (%) [b]
1	$Ph_3C^+BF_4^-$	62
2	$Ph_3C^+ClO_4^-$	17
3	$Ph_3CCl + GaCl_3$	No Product
4	$Ph_3C^+PF_6^-$	trace
5	$Ph_3C^+SbCl_6^-$	No Product

[a] All reactions were carried out with **1a** (0.1 mmol), **2a** (1.5 equiv), $Ph_3C^+X^-$ (1.7 equiv), 2,4,6-collidine (1.8 equiv), Cp*Fe(CO)$_2$(thf)BF_4 (30 mol%) and dry DCE (0.5 mL) at 60 ˚C. [b] NMR yield was determined by using 1,1,2,2-tetrachloroethane as internal standard.

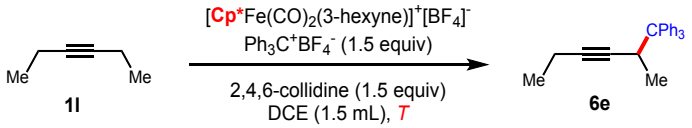
4. Optimization of solvent and catalyst loading.



Entry	Loading of [Fe] (%)	Solvents	NMR Yield (%) [b]
1	30	DCE	62
2	30	$CHCl_3$	60
3	30	$C_6H_4F_2$	70
4	30	PhCl	76
5	30	*t*-BuOMe	No Product
6	30	$PhCF_3$	83
7	20	$PhCF_3$	77
8	10	$PhCF_3$	56

[a] All reactions were carried out with **1a** (0.1 mmol), **2a** (1.5 equiv), $Ph_3C^+BF_4^-$ (1.7 equiv), 2,4,6-collidine (1.8 equiv), Cp*Fe(CO)$_2$(thf)BF_4 (x mol%) and dry solvent (0.5 mL) at 60 ˚C. [b] NMR yield was determined by using 1,1,2,2-tetrachloroethane as internal standard.

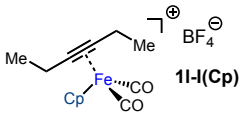
5. Optimization of the temperature.[a]



Entry	*T* (°C)	NMR Yield (%) [b]
1	25	12
2	40	28
3	60	43
4	80	38

[a] All reactions were carried out with **1l** (0.1 mmol), $Ph_3C^+BF_4^-$ (1.5 equiv), 2,4,6-collidine (1.5 equiv), $Cp^*Fe(CO)_2(3\text{-hexyne})^+BF_4^-$ (10 mol%) and dry DCE (0.5 mL). [b] NMR yield was determined by using 1,1,2,2-tetrachloroethane as internal standard.

Characterization data for iron complexes.



1l-I(Cp)

Dicarbonylcyclopentadienyliron(II)-alkyne complex **1l-I(Cp)** was synthesized according to a modification of a literature procedure[1].

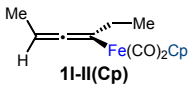
Under N_2, a solution of 3-hexyne (1.71 mL, 15 mmol, 3.0 equiv) in dry toluene (50 mL) was added to a 100 mL flame-dried round-bottom flask, charged with $CpFe(CO)_2I$ (1.52 g, 5 mmol, 1.0 equiv), $AgBF_4$ (1.0 g, ~5 mmol, 1.0 equiv), and a stir bar. The obtained suspension was stirred at 50 °C for 2-3 h. Subsequently, the reaction mixture was allowed to cool to room temperature and filtered through a pad of Celite. The filter cake was rinsed with CH_2Cl_2 until the filtrate turned colorless. The desired iron complex **1l-I(Cp)** was collected as yellow solid (1.68 g, 98%) upon concentration *in vacuo*.

^{1}H NMR (500 MHz, $CDCl_3$) δ 5.64 (br s, 5H), 2.71 (br s, 4H), 1.39 (br s, 6H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 207.9, 89.3, 50.3, 20.1, 15.2.

^{19}F NMR (470 MHz, $CDCl_3$) δ -151.3.

HRMS (ESI) calcd for $C_{13}H_{15}O_2Fe$ $[M–BF_4^-]^+$: 259.0416, found: 259.0424.



1l-II(Cp)

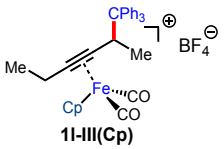
To a solution of **1l-I(Cp)** (69 mg, 0.2 mmol) in dry CH_2Cl_2 (2 mL) was added triethylamine (28 μL, 1.0 equiv.) under N_2. The mixture was stirred at room temperature

for 0.5 h. The solvent was removed under reduced pressure, and the residue was extracted with dry ether and filtered through Celite to remove the insoluble triethylammonium tetrafluoroborate. The filtrate was concentrated to give the crude product **1l-II(Cp)** as a yellow oil. The major peaks of the ^{1}H NMR spectrum could be assigned,[2] but an analytically pure sample of **1l-II(Cp)** could not be obtained.

^{1}H NMR (400 MHz, CD_2Cl_2) δ 4.80 (s, 5H), 4.50-4.35 (m, 1H), 2.10-1.90 (m, 2H), 1.52 (d, J = 6.4 Hz, 3H), 1.00 (t, J = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, CD_2Cl_2) δ 216.5, 216.3, 199.5, 85.8, 84.6, 75.4, 36.1, 15.1, 15.0.

HRMS (ESI) calcd for $C_{13}H_{15}O_2Fe$ $[M+H]^+$: 259.0416, found: 259.0419.

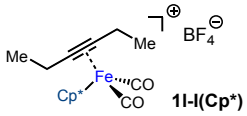


1l-III(Cp)

To a solution of **1l-II(Cp)** prepared from above (approx. 0.2 mmol) in dry CH_2Cl_2 (2 mL) was added $Ph_3C^+BF_4^-$ (66 mg, 0.2 mmol, 1.0 equiv.) as a solid in the argon-filled glove box. The mixture was stirred under N_2 at room temperature for 1 h. Then dry ether (5 mL) was added to the above solution. The desired product precipitated as an orange solid, which was collected on a frit, and further dried *in vacuo* (84 mg, 71% yield).

^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.35 (s, 15H), 5,45 (s, 5H), 4.65 (s, 1H), 2.33 (s, 2H), 1.41 (s, 3H), 0.87 (s, 3H).

HRMS (ESI) calcd for $C_{32}H_{29}O_2Fe$ $[M–BF_4^-]^+$: 501.1511, found: 501.1501.



1l-I(Cp*)

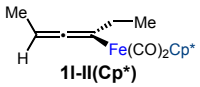
Dicarbonylpentamethylcyclopentadienyliron(II)-alkyne complex **1l-I(Cp*)** was synthesized according to a modification of a literature procedure[1].

Under N_2, a solution of 3-hexyne (0.7 mL, 6 mmol, 3.0 equiv) in dry toluene (20 mL) was added to a 50 mL flame-dried round-bottom flask, charged with $Cp^*Fe(CO)_2I$ (748 mg, 2 mmol), $AgBF_4$ (400 mg, ~2 mmol), and a stir bar. The obtained suspension was stirred at 50 °C for 2-3 h. Subsequently, the reaction mixture was allowed to cool to room temperature and filtered through a pad of Celite. The filter cake was rinsed with CH_2Cl_2 until the filtrate turned colorless. The desired iron complex **1l-I(Cp*)** was collected as brown solid (776 mg, 93%) upon concentration *in vacuo*.

^{1}H NMR (500 MHz, $CDCl_3$) δ 2.56 (q, J = 7.0 Hz, 4H), 1.89 (s, 15H), 1.42 (t, J = 7.0 Hz, 6H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 211.1, 102.4, 60.3, 18.8, 16.1, 9.5.

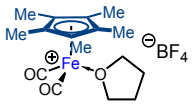
^{19}F NMR (470 MHz, $CDCl_3$) δ –153.4.



1l-II(Cp*)

To a solution of **1l-I(Cp*)** (12.5 mg, 0.03 mmol) in dry CH_2Cl_2 (0.5 mL) was added triethylamine (4 μL, 1.0 equiv.) under N_2. The mixture was stirred at room temperature for 0.5 h. The solvent was removed under reduced pressure, and the residue was extracted with dry ether, filtered through Celite to remove the insoluble triethylammonium tetrafluoroborate. The filtrate was concentrated to give the crude product **1l-II(Cp*)** as a yellow oil.

^{1}H NMR (400 MHz, CD_2Cl_2) δ 4.56-4.43 (m, 1H), 2.10-1.97 (m, 2H), 1.80 (s, 15H), 1.63 (d, J = 6.8 Hz, 3H), 1.06 (t, J = 7.2 Hz, 3H).



Fp*(thf)BF$_4$

The precatalyst was synthesized according to a modification of literature procedures.[3,4]

A mixture of (η^5-C_5Me_5)$Fe(CO)_2I$ (1.53 g, 4 mmol) and $AgBF_4$ (800 mg, ~4.0 mmol) was stirred in THF (22 mL) overnight under N_2. The solvent was removed *in vacuo*, the residue was extracted with CH_2Cl_2, and the solution was filtered through Celite to remove AgI, and the filter cake was washed with CH_2Cl_2 until the filtrate became colorless. The filtrate was concentrated to 10 mL and diethyl ether (Et_2O) was added to precipitate the product as a red crystalline solid. After cooling the mixture at –20 °C for 2 h, the crystals were collected, washed with Et_2O, and dried *in vacuo* to give red crystals **Fp*(thf)BF$_4$** (1.47 g, 91% yield).

^{1}H NMR (400 MHz, CD_2Cl_2) δ 3.42 (t, J = 6.4 Hz, 4H), 1.88-1.82 (m, 4H), 1.77 (s, 15H).

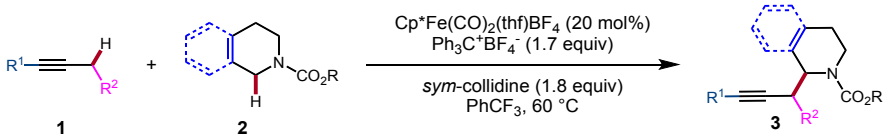
^{13}C NMR (100 MHz, CD_2Cl_2) δ 210.8, 98.5, 80.8, 26.2, 9.4.

^{19}F NMR (376 MHz, CD_2Cl_2) δ -152.1.

The spectroscopic data were identical in all respects to those previously reported.[4]

HRMS (ESI) calcd for $C_{14}H_{18}O_2NFe$ [M – C_4H_8O + C_2H_3N – BF_4^-]$^+$: 288.0681, found: 288.0683.

General procedure A for propargylic C-H functionalization of alkynes.

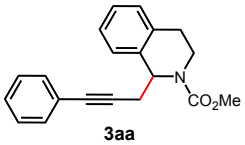


1 **2** **3**

A reaction tube (13 mm × 100 mm, Fisherbrand, part # 14-959-35C) equipped with a

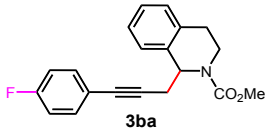
magnetic stir bar was capped with a Teflon/silicone septum (Thermo/National part # C4015-66A) screw cap and flame dried under vacuum. The reaction tube was cooled under argon and transferred into a nitrogen-filled glovebox. In the glovebox, the suspension of carbamate (**2**, 0.45 mmol, 1.5 equiv) and $Ph_3C^+BF_4^-$ (168 mg, 0.51 mmol, 1.7 equiv) in dry trifluorotoluene (1 mL) was stirred at room temperature for 3 hours to generate the iminium salt. Then $[Cp^*Fe(CO)_2(thf)]^+[BF_4]^-$ (20 mol%, 24.6 mg), alkyne **1** (0.3 mmol, 1.0 equiv), trifluorotoluene (0.5 mL) and *sym*-collidine (72 µL, 0.54 mmol, 1.8 equiv) were added in rapid succession. The reaction tube was capped and removed from the glovebox. The reaction tube was placed in an oil bath, preheated to 60 °C, where it was stirred for 48 h. After completion of the reaction, the reaction mixture was cooled to room temperature. The crude mixture was concentrated in vacuo and purified by flash column chromatography to provide the desired product **3**.

Characterization Data for Products 3:



3aa

Methyl 1-(3-phenylprop-2-yn-1-yl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (3aa): Prepared following general procedure A, using prop-1-yn-1-ylbenzene (**1a**, 39 µL, 0.3 mmol, 1.0 equiv) and methyl 3,4-dihydroisoquinoline-2(1H)-carboxylate (**2a**, 86.1 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **3aa** as a colorless oil (66 mg, 72% yield).
^{1}H NMR (500 MHz, $CDCl_3$, rotamers seen) δ 7.31-7.21 (m, 2H), 7.21-7.15 (m, 4H), 7.15-7.09 (m, 2H), 7.09-7.01 (m, 1H), 5.33 (t, *J* = 6.0 Hz, 0.5H), 5.26 (t, *J* = 6.5 Hz, 0.5H), 4.20-4.10 (m, 0.5H), 3.94-3.81 (m, 0.5H), 3.67 (s, 1.5H), 3.66 (s, 1.5H), 3.61-3.49 (m, 0.5H), 3.46-3.34 (m, 0.5H), 2.97-2.68 (m, 4H).
^{13}C NMR (125 MHz, $CDCl_3$, rotamers seen) δ 156.0, 135.7, 135.5, 134.42, 134.37, 131.4, 129.0, 128.5, 128.2, 128.1, 127.8, 127.6, 127.4, 127.1, 127.0, 126.2, 126.1, 123.6, 123.4, 86.8, 86.5, 82.9, 53.7, 52.73, 52.67, 39.3, 38.2, 28.7, 28.4, 27.5, 27.2.
HRMS (ASAP) calcd for $C_{20}H_{20}O_2N$ $[M+H]^+$: 306.1494, found: 306.1480.



3ba

Methyl 1-(3-(4-fluorophenyl)prop-2-yn-1-yl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (3ba): Prepared following general procedure A, using 1-fluoro-4-(prop-1-

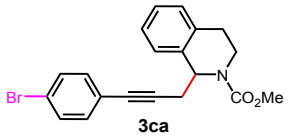
yn-1-yl)benzene (**1b**, 40.2 mg, 0.3 mmol, 1.0 equiv) and methyl 3,4-dihydroisoquinoline-2(1H)-carboxylate (**2a**, 86.1 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **3ba** as a colorless oil (64 mg, 66% yield).

^{1}H NMR (500 MHz, $CDCl_3$, rotamers seen) δ 7.38-7.18 (m, 5H), 7.18-7.10 (m, 1H), 7.02-6.96 (q, *J* = 8.5 Hz, 2H), 5.41 (t, *J* = 6.0 Hz, 0.5H), 5.33 (t, *J* = 6.5 Hz, 0.5H), 4.30-4.18 (m, 0.5H), 4.05-3.91 (m, 0.5H), 3.75 (s, 1.5H), 3.74 (s, 1.5H), 3.66-3.56 (m, 0.5H), 3.52-3.40 (m, 0.5H), 3.05-2.73 (m, 4H).

^{19}F NMR (470 MHz, $CDCl_3$, rotamers seen) δ -111.6, -111.9.

^{13}C NMR (125 MHz, $CDCl_3$, rotamers seen) δ 163.2, 163.1, 161.2, 161.1, 156.1, 135.6, 135.5, 134.4, 134.3, 133.3, 133.2, 131.4, 129.0, 128.5, 127.3, 127.1, 127.0, 126.2, 126.1, 119.7, 119.5, 115.5, 115.4, 115.3, 115.2, 86.5, 86.1, 81.83, 81.77, 53.7, 53.6, 52.73, 52.68, 39.2, 38.2, 28.7, 28.4, 27.5, 27.1 (the ^{13}C spectrum is too complicated to distinguish the C-F coupling);

HRMS (ESI) calcd for $C_{20}H_{19}O_2NF$ $[M+H]^+$: 324.1394, found: 324.1408.



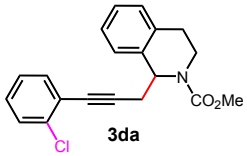
3ca

Methyl 1-(3-(4-bromophenyl)prop-2-yn-1-yl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (3ca): Prepared following general procedure A, using 1-bromo-4-(prop-1-yn-1-yl)benzene (**1c**, 58.2 mg, 0.3 mmol, 1.0 equiv) and methyl 3,4-dihydroisoquinoline-2(1H)-carboxylate (**2a**, 86.1 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **3ca** as a brown oil (65 mg, 56% yield).

^{1}H NMR (500 MHz, $CDCl_3$, rotamers seen) δ 7.48-7.36 (m, 2H), 7.32-7.08 (m, 6H), 5.44 (t, *J* = 6.0 Hz, 0.5H), 5.36 (t, *J* = 6.0 Hz, 0.5H), 4.32-4.20 (m, 0.5H), 4.03-3.93 (m, 0.5H), 3.78 (s, 1.5H), 3.76 (s, 1.5H), 3.68-3.56 (m, 0.5H), 3.54-3.40 (m, 0.5H), 3.07-2.75 (m, 4H).

^{13}C NMR (125 MHz, $CDCl_3$, rotamers seen) δ 156.1, 135.6, 135.5, 134.5, 134.4, 133.0, 131.5, 131.4, 129.1, 128.6, 127.4, 127.2, 127.12, 127.08, 126.3, 126.2, 122.6, 122.4, 122.0, 121.9, 88.2, 87.9, 82.0, 81.9, 53.7, 53.6, 52.81, 52.77, 39.3, 38.2, 28.7, 28.4, 27.6, 27.3.

HRMS (ESI) calcd for $C_{20}H_{19}O_2NBr$ $[M+H]^+$: 384.0594, found: 384.0612.



3da

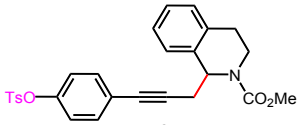
Methyl 1-(3-(2-chlorophenyl)prop-2-yn-1-yl)-3,4-dihydroisoquinoline-2(1H)-

carboxylate (3da): Prepared following general procedure A, using 1-chloro-2-(prop-1-yn-1-yl)benzene (**1d**, 45 mg, 0.3 mmol, 1.0 equiv) and methyl 3,4-dihydroisoquinoline-2(1H)-carboxylate (**2a**, 86.1 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **3da** as a colorless oil (65 mg, 64% yield).

^{1}H NMR (500 MHz, $CDCl_3$, rotamers seen) δ 7.45-7.27 (m, 3H), 7.25-7.10 (m, 5H), 5.44 (t, J = 5.5 Hz, 0.5H), 5.37 (t, J = 6.0 Hz, 0.5H), 4.33-4.19 (m, 0.5H), 4.06-3.93 (m, 0.5H), 3.754 (s, 1.5H), 3.749 (s, 1.5H), 3.70-3.64 (m, 0.5H), 3.56-3.43 (m, 0.5H), 3.12-2.75 (m, 4H).

^{13}C NMR (125 MHz, $CDCl_3$, rotamers seen) δ 156.0, 135.6, 135.5, 135.4, 134.5, 134.4, 133.4, 129.1, 129.02, 128.99, 128.8, 128.64, 128.58, 127.4, 127.1, 127.0, 126.3, 126.23, 126.17, 123.4, 123.3, 92.3, 92.0, 79.7, 53.5, 52.73, 52.67, 39.2, 38.2, 28.7, 28.4, 27.8, 27.3.

HRMS (ESI) calcd for $C_{20}H_{19}O_2NCl$ $[M+H]^+$: 340.1099, found: 340.1109.



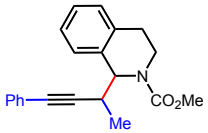
3ea

Methyl 1-(3-(4-(tosyloxy)phenyl)prop-2-yn-1-yl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (3ea): Prepared following general procedure A, using 4-(prop-1-yn-1-yl)phenyl 4-methylbenzenesulfonate (**1e**, 86 mg, 0.3 mmol, 1.0 equiv) and methyl 3,4-dihydroisoquinoline-2(1H)-carboxylate (**2a**, 86.1 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with 30% ethyl acetate in hexanes to provide the product **3ea** as a colorless oil (79 mg, 55% yield).

^{1}H NMR (500 MHz, $CDCl_3$, rotamers seen) δ 7.71 (d, J = 8.0 Hz, 2H), 7.32 (d, J = 8.0 Hz, 2H), 7.30-7.13 (m, 6H), 6.91 (t, J = 8.5 Hz, 2H), 5.42 (t, J = 6.5 Hz, 0.5H), 5.37 (t, J = 6.5 Hz, 0.5H), 4.31-4.18 (m, 0.5H), 4.03-3.93 (m, 0.5H), 3.76 (s, 1.5H), 3.75 (s, 1.5H), 3.67-3.55 (m, 0.5H), 3.53-3.41 (m, 0.5H), 3.07-2.80 (m, 4H), 2.47 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$, rotamers seen) δ 156.0, 148.9, 148.8, 145.5, 135.6, 135.4, 134.41, 134.35, 132.7, 132.2, 129.8, 129.0, 128.6, 128.5, 127.3, 127.2, 127.1, 127.0, 126.22, 126.18, 122.4, 122.3, 88.1, 87.7, 81.6, 53.7, 53.6, 52.7, 39.2, 38.2, 28.7, 28.4, 27.5, 27.2, 21.7.

HRMS (ESI) calcd for $C_{27}H_{26}O_5NS$ $[M+H]^+$: 476.1526, found: 476.1535.



3fa

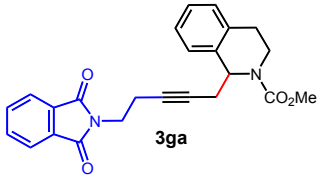
Methyl 1-(4-phenylbut-3-yn-2-yl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (3fa): Prepared following general procedure A, using but-1-yn-1-ylbenzene (**1f**, 42 μL, 0.3

mmol, 1.0 equiv) and methyl 3,4-dihydroisoquinoline-2(1H)-carboxylate (**2a**, 86.1 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **3fa** as a colorless oil (64 mg, 67% yield, 1:1 d.r.). The d.r. value was detected by crude NMR. The diastereoisomer could not be separated by column.

^{1}H NMR (500 MHz, $CDCl_3$, rotamers and diastereoisomer seen) δ 7.42-7.00 (m, 9H), 5.24 (d, *J* = 5.5 Hz, 0.25H), 5.11-5.02 (m, 0.5 H), 4.97 (d, *J* = 8.0 Hz, 0.25H), 4.18-4.08 (m, 0.25H), 4.06-3.98 (m, 0.25H), 3.98-3.91 (m, 0.25H), 3.90-3.81 (m, 0.25H), 3.81-3.71 (m, 0.25H), 3.73-3.61 (m, 0.25H), 3.66 (s, 1.5H), 3.65 (s, 0.75H), 3.64 (s, 0.75H), 3.59-3.51 (m, 0.25H), 3.42-3.33 (m, 0.25H), 3.25-2.65 (m, 3H), 1.38-1.22 (m, 3H).

^{13}C NMR (125 MHz, $CDCl_3$, rotamers and diastereoisomer seen) δ 156.8, 156.5, 156.4, 135.7, 135.4, 135.2, 134.8, 134.64, 134.56, 134.4, 131.4, 129.0, 128.9, 128.72, 128.67, 128.3, 128.2, 128.1, 128.0, 127., 127.83, 127.7, 127.6, 127.4, 127.3, 127.23, 127.18, 126.9, 126.1, 125.9, 125.5, 125.4, 92.1, 92.0, 91.5, 83.8, 83.5, 83.3, 83.2, 59.4, 58.9, 58.2, 57.9, 52.8, 52.7, 39.8, 39.5, 39.0, 38.8, 33.9, 33.2, 28.4, 28.2, 28.0, 27.8, 18.8, 18.6, 18.3, 18.1.

HRMS (ESI) calcd for $C_{21}H_{22}NO_2$ $[M+H]^+$: 320.1645, found: 320.1653.



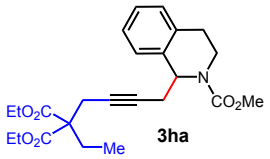
3ga

Methyl 1-(5-(1,3-dioxoisoindolin-2-yl)pent-2-yn-1-yl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (3ga): Prepared following general procedure A, using 2-(pent-3-yn-1-yl)isoindoline-1,3-dione (**1g**, 64 mg, 0.3 mmol, 1.0 equiv) and methyl 3,4-dihydroisoquinoline-2(1H)-carboxylate (**2a**, 86.1 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with 20% ethyl acetate in hexane to provide the product **3ga** as a colorless oil (74 mg, 61% yield).

^{1}H NMR (400 MHz, $CDCl_3$, rotamers seen) δ 7.91-7.77 (m, 2H), 7.77-7.62 (m, 2H), 7.20-7.00 (m, 4H), 5.22 (t, *J* = 6.0 Hz, 0.5H), 5.12 (t, *J* = 6.0 Hz, 0.5H), 4.16-4.02 (m, 0.5H), 3.91-3.82 (m, 0.5H), 3.81-3.73 (m, 2H), 3.71 (s, 1.5H), 3.70 (s, 1.5H), 3.54-3.40 (m, 0.5H), 3.40-3.25 (m, 0.5H), 3.05-2.73 (m, 2H), 2.73-2.40 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$, rotamers seen) δ 167.9, 155.9, 135.7, 135.5, 134.23, 134.16, 133.9, 132.0, 128.8, 128.4, 127.2, 127.0, 126.9, 126.8, 126.0, 125.9, 123.2, 78.7, 78.6, 78.5, 78.4, 53.55, 53.49, 52.6, 38.9, 38.1, 36.8, 28.6, 28.2, 26. 8, 26.3, 18.6.

HRMS (ESI) calcd for $C_{24}H_{23}O_4N_2$ $[M+H]^+$: 403.1652, found: 403.1655.



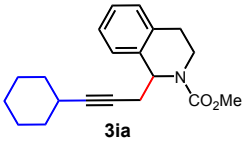
3ha

Diethyl 2-ethyl-2-(4-(2-(methoxycarbonyl)-1,2,3,4-tetrahydroisoquinolin-1-yl) but-2-yn-1-yl) malonate (3ha): Prepared following general procedure A, using diethyl 2-(but-2-yn-1-yl)-2-ethylmalonate (**1h**, 72 mg, 0.3 mmol, 1.0 equiv) and methyl 3,4-dihydroisoquinoline-2(1H)-carboxylate (**2a**, 86.1 mg, 0.45 mmol, 1.5 equiv). The crude residue was diluted with CH_2Cl_2 and washed with 1N HCl to remove the *sym*-collidine. The extracts were combined and concentrated, purified by flash column chromatography, eluting with 20% ethyl acetate in hexane to provide the product **3ha** as a colorless oil (77 mg, 60% yield).

^{1}H NMR (500 MHz, $CDCl_3$, rotamers seen) δ 7.22-7.14 (m, 3H),7.14-7.06 (m, 1H), 5.22 (t, J = 6.5 Hz, 0.5H), 5.13 (t, J = 7.0 Hz, 0.5H), 4.22-4.04 (m, 4.5H), 4.00-3.87 (m, 0.5H), 3.73 (s, 3H), 3.60-3.47 (m, 0.5H), 3.45-3.31 (m, 0.5H), 3.00-2.50 (m, 6H), 2.00-1.80 (m, 2H), 1.29-1.10 (m, 6H), 0.83-0.65 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, rotamers seen) δ 170.4, 155.8, 135.6, 135.5, 134.4, 134.3, 128.9, 128.5, 127.2, 127.0, 126.9, 126.7, 126.1, 126.0, 79.5, 79.4, 77.20, 77.17, 61.2, 57.3, 53.45, 53.38, 52.61, 52.58, 39.1, 38.2, 28.7, 28.3, 26.9, 26.4, 24.7, 22.4, 14.0, 8.2.

HRMS (ESI) calcd for $C_{24}H_{32}O_6N$ $[M+H]^+$: 430.2224, found: 430.2217.



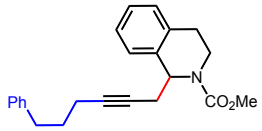
3ia

Methyl 1-(3-cyclohexylprop-2-yn-1-yl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (3ia): Prepared following general procedure A, using prop-1-yn-1-ylcyclohexane (**1i**, 36.6 mg, 0.3 mmol, 1.0 equiv) and methyl 3,4-dihydroisoquinoline-2(1H)-carboxylate (**2a**, 86.1 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **3ia** as a colorless oil (66 mg, 60% yield).

^{1}H NMR (500 MHz, $CDCl_3$, rotamers seen) δ 7.25-7.08 (m, 4H), 5.28 (t, J = 6.0 Hz, 0.5H), 5.19 (t, J = 6.5 Hz, 0.5H), 4.25-4.15 (m, 0.5H), 4.00-3.90 (m, 0.5H), 3.75 (s, 3H), 3.63-3.53 (m, 0.5H), 3.47-3.35 (m, 0.5H), 3.00-2.55 (m, 4H), 2.28 (s, 1H), 1.80-1.55 (m, 4H), 1.55-1.40 (m, 1H), 1.40-1.13 (m, 5H).

^{13}C NMR (125 MHz, $CDCl_3$, rotamers seen) δ 156.0, 155.9, 136.0, 135.8, 134.3, 134.2, 128.9, 128.4, 127.4, 127.1, 126.9, 126.8, 126.0, 125.9, 87.1, 87.0, 76.5, 76.2, 53.8, 52.6, 39.0, 38.0, 32.8, 32.7, 29.7, 29.1, 29.0, 28.7, 28.4, 26.9, 26.5, 25.9, 24.8, 24.7.

HRMS (ESI) calcd for $C_{20}H_{26}O_2N$ $[M+H]^+$: 312.1958, found: 312.1960.



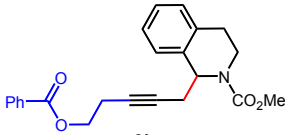
3ja

Methyl 1-(6-phenylhex-2-yn-1-yl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (3ja): Prepared following general procedure A, using hex-4-yn-1-ylbenzene (**1j**, 47.4 mg, 0.3 mmol, 1.0 equiv) and methyl 3,4-dihydroisoquinoline-2(1H)-carboxylate (**2a**, 86.1 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **3ja** as a colorless oil (65 mg, 62% yield, 10:1 r.r.).

^{1}H NMR (500 MHz, $CDCl_3$, rotamers seen) δ 7.50-7.00 (m, 9H), 5.35 (t, *J* = 6.0 Hz, 0.5H), 5.25 (t, *J* = 6.0 Hz, 0.5H), 4.33-4.16 (m, 0.5H), 4.10-3.94 (m, 0.5H), 3.77 (s, 1.5H), 3.76 (s, 1.5H), 3.69-3.57 (m, 0.5H), 3.57-3.41 (m, 0.5H), 3.05-2.88 (m, 1H), 2.88-2.69 (m, 3H), 2.69-2.59 (m, 2H), 2.25-2.07 (m, 2H), 1.85-1.65 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$, rotamers seen) δ 156.01, 155.96, 141.7, 141.6, 135.9, 135.7, 134.4, 134.3, 128.9, 128.52, 128.46, 128.2, 127.3, 127.05, 126.95, 126.8, 126.1, 126.0, 125.8, 125.7, 82.4, 82.2, 53.8, 52.6, 39.1, 38.2, 34.7, 34.6, 30.3, 28.7, 28.4, 26.9, 26.5, 18.1.

HRMS (ASAP) calcd for $C_{23}H_{26}NO_2$ $[M+H]^+$: 348.1964, found: 348.1974.



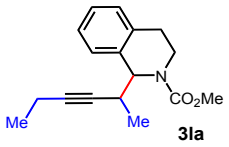
3ka

Methyl 1-(5-(benzoyloxy)pent-2-yn-1-yl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (3ka): Prepared following general procedure A, using pent-3-yn-1-yl benzoate (**1k**, 56.4 mg, 0.3 mmol, 1.0 equiv) and methyl 3,4-dihydroisoquinoline-2(1H)-carboxylate (**2a**, 86.1 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **3ka** as a colorless oil (58 mg, 51% yield, 4.3:1 r.r.).

^{1}H NMR (500 MHz, $CDCl_3$, rotamers seen) δ 8.04 (d, *J* = 10.0 Hz, 2H), 7.61-7.53 (m, 1H), 7.44 (t, *J* = 9.5 Hz, 2H), 7.25-7.02 (m, 4H), 5.28 (t, *J* = 7.5 Hz, 0.5H), 5.19 (t, *J* = 7.5 Hz, 0.5H), 4.40-4.25 (m, 2H), 4.21-4.08 (m, 0.5H), 3.97-3.84 (m, 0.5H), 3.75 (s, 1.5H), 3.74 (s, 1.5H), 3.62-3.50 (m, 0.5H), 3.48-3.36 (m, 0.5H), 2.98-2.63 (m, 4H), 2.63-2.51 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$, rotamers seen) δ 166.3, 156.0, 135.7, 135.6, 134.4, 134.3, 133.0, 130.1, 129.7, 128.9, 128.5, 128.4, 127.3, 127.1, 126.9, 126.14, 126.09, 78.6, 78.5, 78.3, 78.2, 63.1, 53.7, 52.7, 39.2, 38.3, 28.7, 28.4, 26.9, 26.5, 19.4.

HRMS (ESI) calcd for $C_{23}H_{24}O_4N$ $[M+H]^+$: 378.1700, found: 378.1701.



3la

Methyl 1-(hex-3-yn-2-yl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (3la): Prepared following general procedure A, using hex-3-yne (**1l**, 33 μL, 0.3 mmol, 1.0 equiv) and methyl 3,4-dihydroisoquinoline-2(1H)-carboxylate (**2a**, 86.1 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **3la** as a colorless oil (57 mg, 70% yield, 1:1 d.r.). The d.r. value was detected by crude NMR.

One isomer:

^{1}H NMR (500 MHz, $CDCl_3$, rotamers seen) δ 7.42 (d, *J* = 6.5 Hz, 0.5H), 7.38 (*J* = 7.0 Hz, 0.5H), 7.24-7.06 (m, 3H), 5.01 (d, *J* = 8.5 Hz, 0.5H), 4.91 (d, *J* = 9.0 Hz, 0.5H), 4.12-4.02 (m, 0.5H), 3.80-3.70 (m, 0.5H), 3.73 (s, 3H), 3.60-3.52 (m, 0.5H), 3.43-3.34 (m, 0.5H), 3.06-2.76 (m, 3H), 2.19-2.06 (m, 2H), 1.26 (d, *J* = 6.5 Hz, 1.5H), 1.22 (d, *J* = 6.5 Hz, 1.5H), 1.09 (t, *J* = 7.5 Hz, 1.5H), 1.05 (t, *J* = 7.5 Hz, 1.5H).

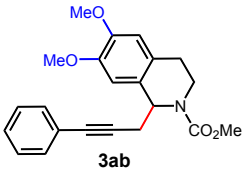
^{13}C NMR (125 MHz, $CDCl_3$, rotamers seen) δ 156.7, 156.3, 135.51, 135.49, 134.4, 134.3, 129.0, 128.8, 128.6, 128.1, 127.2, 127.1, 125.2, 125.1, 85.0, 84.7, 81.5, 59.4, 58.9, 52.7, 52.6, 39.6, 38.6, 32.4, 28.1, 27.7, 19.1, 18.8, 13.8, 12.4.

HRMS (ESI) calcd for $C_{17}H_{22}NO_2$ $[M+H]^+$: 272.1645, found: 272.1648.

Another isomer:

^{1}H NMR (500 MHz, $CDCl_3$, rotamers seen) δ 7.23-7.06 (m, 4H), 5.17 (d, *J* = 6.0 Hz, 0.5H), 5.00 (d, *J* = 6.0 Hz, 0.5H), 4.22-4.12 (m, 0.5H), 4.04-3.95 (m, 0.5H), 3.95-3.85 (m, 0.5H), 3.85-3.75 (m, 0.5H), 3.72 (s, 3H), 3.09-2.74 (m, 3H), 2.10-1.96 (m, 2H), 1.26 (d, *J* = 7.0 Hz, 1.5H), 1.23 (d, *J* = 7.0 Hz, 1.5H), 1.03-0.92 (m, 3H).

^{13}C NMR (125 MHz, $CDCl_3$, rotamers seen) δ 156.7, 157.5, 136.0, 135.7, 134.8, 134.5, 128.9, 128.6, 127.4, 127.3, 126.9, 126.7, 125.9, 125.8, 84.6, 84.4, 81.3, 81.1, 58.2, 57.9, 52.7, 52.5, 39.3, 38.8, 33.14, 33.11, 28.4, 27.9, 18.64, 18.59, 13.93, 13.90, 12.4.



3ab

Methyl 6,7-dimethoxy-1-(3-phenylprop-2-yn-1-yl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (3ab): Prepared following general procedure A, using prop-1-yn-1-ylbenzene (**1a**, 39 μL, 0.3 mmol, 1.0 equiv) and methyl 6,7-dimethoxy-3,4-dihydroisoquinoline-2(1H)-carboxylate (**2b**, 113 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with 5% ethyl acetate in CH_2Cl_2 to provide the product **3ab** as a white solid (81 mg, 74% yield).

Scale-up to 5 mmol:

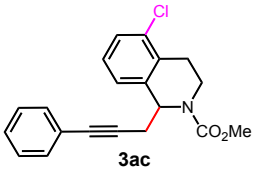
In an argon-filled glovebox, to the 50 mL flame-dry flask, methyl 6,7-dimethoxy-3,4-dihydroisoquinoline-2(1H)-carboxylate (**2b**, 1883 mg, 7.5 mmol, 1.5 equiv) and $Ph_3C^+BF_4^-$ (2800 mg, 8.5 mmol, 1.7 equiv) was stirred in dry trifluorotoluene (15 mL) at room temperature for 3 hours to generate the iminium salt. Then $Cp^*Fe(CO)_2(thf)^+BF_4^-$ (205 mg, 10 mol%), prop-1-yn-1-ylbenzene **1a** (650 µL, 5 mmol, 1.0 equiv), trifluorotoluene (5 mL) and *sym*-collidine (1.2 mL, 9.0 mmol, 1.8 equiv) were added in rapid succession. The reaction tube was capped and removed from the glovebox. The reaction tube was placed in an oil bath, preheated to 60 °C, where it was stirred for 48 h. After completion of the reaction, the reaction mixture was cooled to room temperature. The crude mixture was concentrated in vacuo and purified by flash column chromatography to provide the desired product **3ab** as a pale-yellow solid (1.008 g, 55% yield)

m.p. = 50-51 °C.

^{1}H NMR (500 MHz, $CDCl_3$, rotamers seen) δ 7.40-7.31 (m, 2H), 7.31-7.23 (m, 3H), 6.84 (d, *J* = 25 Hz, 1H), 6.64 (s, 1H), 5.35 (t, *J* = 6.0 Hz, 0.5H), 5.28 (t, *J* = 6.5 Hz, 0.5H), 4.31-4.20 (m, 0.5H), 4.04-3.94 (m, 0.5H), 3.88 (s, 3H), 3.81 (s, 1.5H), 3.79 (s, 1.5H), 3.76 (s, 3H), 3.64-3.52 (m, 0.5H), 3.50-3.35 (m, 0.5H), 3.05-2.80 (m, 3H), 2.80-2.67 (m, 1H).

^{13}C NMR (125 MHz, $CDCl_3$, rotamers seen) δ 156.0, 148.1, 148.0, 147.4, 131.5, 128.3, 128.2, 127.9, 127.8, 127.7, 127.5, 126.5, 126.3, 123.6, 123.4, 111.5, 111.2, 110.5, 110.2, 87.1, 86.8, 83.1, 55.95, 55.91, 53.4, 52.8, 52.7, 39.2, 38.2, 28.3, 28.0, 27.5, 27.1.

HRMS (ESI) calcd for $C_{22}H_{24}O_4N$ $[M+H]^+$: 366.1700, found: 366.1707.



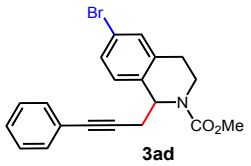
3ac

Methyl 5-chloro-1-(3-phenylprop-2-yn-1-yl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (3ac): Prepared following general procedure A, using prop-1-yn-1-ylbenzene (**1a**, 39 µL, 0.3 mmol, 1.0 equiv) and methyl 5-chloro-3,4-dihydroisoquinoline-2(1H)-carboxylate (**2c**, 101 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **3ac** as a light-yellow oil (75 mg, 74% yield).

^{1}H NMR (500 MHz, $CDCl_3$, rotamers seen) δ 7.42-7.26 (m, 6H), 7.26-7.14 (m, 2H), 5.47 (t, *J* = 6.0 Hz, 0.5H), 5.37 (t, *J* = 6.5 Hz, 0.5H), 4.44-4.32 (m, 0.5H), 4.20-4.04 (m, 0.5H), 3.78 (s, 1.5H), 3.77 (s, 1.5H), 3.65-3.55 (m, 0.5H), 3.50-3.38 (m, 0.5H), 3.05-2.80 (m, 4H).

^{13}C NMR (125 MHz, $CDCl_3$, rotamers seen) δ 155.9, 137.9, 137.8, 134.5, 134.1, 132.6, 132.5, 131.5, 128.3, 128.2, 128.0, 127.9, 127.0, 126.9, 125.9, 125.6, 123.5, 123.3, 86.3, 86.0, 83.3, 53.4, 53.3, 52.9, 38.2, 37.2, 27.4, 27.0, 26.5, 26.3.

HRMS (ESI) calcd for $C_{20}H_{19}O_2NCl$ $[M+H]^+$: 340.1099, found: 340.1111.



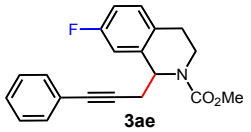
3ad

Methyl 6-bromo-1-(3-phenylprop-2-yn-1-yl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (3ad): Prepared following general procedure A, using prop-1-yn-1-ylbenzene (**1a**, 39 μL, 0.3 mmol, 1.0 equiv) and methyl 6-bromo-3,4-dihydroisoquinoline-2(1H)-carboxylate (**2d**, 122 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **3ad** as a colorless oil (84 mg, 73% yield).

^{1}H NMR (500 MHz, $CDCl_3$, rotamers seen) δ 7.42-7.25 (m, 7H), 7.24-7.11 (m, 1H), 5.38 (t, J = 6.0 Hz, 0.5H), 5.30 (t, J = 6.0 Hz, 0.5H), 4.30-4.18 (m, 0.5H), 4.04-3.90 (m, 0.5H), 3.78 (s, 3H), 3.69-3.58 (m, 0.5H), 3.54-3.42 (m, 0.5H), 3.05-2.75 (m, 4H).

^{13}C NMR (125 MHz, $CDCl_3$, rotamers seen) δ 156.0, 136.83, 136.76, 134.7, 134.5, 131.8, 131.8, 131.5, 131.4, 129.34, 129.29, 129.1, 128.8, 128.3, 128.2, 128.0, 127.9, 123.5, 123.3, 120.9, 120.8, 86.3, 86.1, 83.3, 53.4, 52.9, 52.8, 38.9, 37.9, 28.6, 28.3, 27.5, 27.1.

HRMS (ESI) calcd for $C_{20}H_{19}O_2NBr$ $[M+H]^+$: 384.0594, found: 384.0602.



3ae

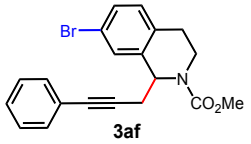
Methyl 7-fluoro-1-(3-phenylprop-2-yn-1-yl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (3ae): Prepared following general procedure A, using prop-1-yn-1-ylbenzene (**1a**, 39 μL, 0.3 mmol, 1.0 equiv) and methyl 7-fluoro-3,4-dihydroisoquinoline-2(1H)-carboxylate (**2e**, 94 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **3ae** as a colorless oil (73 mg, 75% yield).

^{1}H NMR (500 MHz, $CDCl_3$, rotamers seen) δ 7.43-7.20 (m, 5H), 7.19-7.10 (m, 1H), 7.10-6.99 (m, 1H), 6.99-6.89 (m, 1H), 5.40 (t, J = 6.0 Hz, 0.5H), 5.32 (t, J = 6.0 Hz, 0.5H), 4.33-4.19 (m, 0.5H), 4.05-3.93 (m, 0.5H), 3.78 (s, 3H), 3.69-3.56 (m, 0.5H), 3.54-3.41 (m, 0.5H), 3.07-2.74 (m, 4H).

^{19}F NMR (470 MHz, $CDCl_3$, rotamers seen) δ -116.23, -116.16.

^{13}C NMR (125 MHz, $CDCl_3$, rotamers seen) δ 162.1, 162.0, 160.15, 160.09, 156.0, 137.5, 137.4, 137.3, 137.2, 131.5, 130.44, 130.38, 130.1, 130.0, 128.3, 128.2, 128.0, 127.8, 123.5, 123.3, 114.4, 114.3, 114.1, 113.95, 113.86, 113.7, 86.4, 86.1, 83.32, 83.27, 53.60, 53.58, 52.9, 52.8, 39.3, 38.3, 28.1, 27.8, 27.5, 27.0. (the ^{13}C spectrum is too complicated to distinguish the C-F coupling)

HRMS (ESI) calcd for $C_{20}H_{19}O_2NF$ $[M+H]^+$: 324.1394, found: 324.1408.



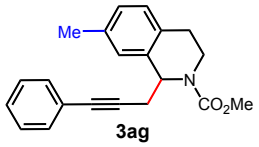
3af

Methyl 7-bromo-1-(3-phenylprop-2-yn-1-yl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (3af): Prepared following general procedure A, using prop-1-yn-1-ylbenzene (**1a**, 39 µL, 0.3 mmol, 1.0 equiv) and methyl 7-bromo-3,4-dihydroisoquinoline-2(1H)-carboxylate (**2f**, 122 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **3af** as a colorless oil (80 mg, 69% yield).

^{1}H NMR (500 MHz, $CDCl_3$, rotamers seen) δ 7.50 (d, *J* = 16.5 Hz, 1H), 7.43-7.33 (m, 3H),7.33-7.25 (m, 3H), 7.09-7.00 (m, 1H), 5.38 (t, *J* = 5.5 Hz, 0.5H), 5.30 (t, *J* = 6.5 Hz, 0.5H), 4.33-4.17 (m, 0.5H), 4.04-3.93 (m, 0.5H), 3.78 (s, 3H), 3.69-3.56 (m, 0.5H), 3.53-3.41 (m, 0.5H), 3.05-2.75 (m, 4H).

^{13}C NMR (125 MHz, $CDCl_3$, rotamers seen) δ 156.0, 137.8, 137.6, 133.5, 131.5, 130.6, 130.4, 130.3, 130.2, 128.3, 128.2, 128.0, 127.9, 123.4, 123.3, 119.7, 119.6, 86.3, 86.0, 83.5, 53.3, 52.9, 52.8, 39.1, 38.1, 28.3, 28.0, 27.5, 27.1.

HRMS (ESI) calcd for $C_{20}H_{19}O_2NBr$ $[M+H]^+$: 384.0593, found: 384.0602.



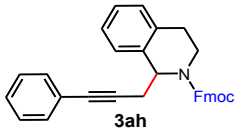
3ag

Methyl 7-methyl-1-(3-phenylprop-2-yn-1-yl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (3ag): Prepared following general procedure A, using prop-1-yn-1-ylbenzene (**1a**, 26 µL, 0.2 mmol, 1.0 equiv) and methyl 7-methyl-3,4-dihydroisoquinoline-2(1H)-carboxylate (**2g**, 61 mg, 0.3 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **3ag** as a colorless oil (43 mg, 67% yield).

^{1}H NMR (500 MHz, $CDCl_3$, rotamers seen) δ 7.49-7.20 (m, 5H), 7.19-6.95 (m, 3H), 5.39 (s, 0.5H), 5.33 (s, 0.5H), 4.34-4.20 (m, 0.5H), 4.05-3.92 (m, 0.5H), 3.77 (s, 3H), 3.70-3.55 (m, 0.5H), 3.54-3.40 (m, 0.5H), 3.10-2.70 (m, 4H), 2.35 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$, rotamers seen) δ 156.1, 135.64, 135.61, 135.5, 135.4, 131.4, 131.3, 128.8, 128.3, 128.2, 128.1, 128.0, 127.9 127.83, 127.78, 127.7, 123.7, 123.5, 87.0, 86.6, 82.9, 53.7, 52.7, 52.6, 39.4, 38.3, 28.3, 28.0, 27.5, 27.2, 21.1.

HRMS (ESI) calcd for $C_{21}H_{22}O_2N$ $[M+H]^+$: 320.1645, found: 320.1658.



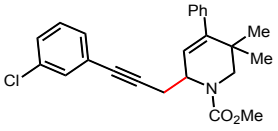
3ah

Methyl 7-methyl-1-(3-phenylprop-2-yn-1-yl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (3ah): Prepared following general procedure A, using prop-1-yn-1-ylbenzene (**1a**, 39 µL, 0.3 mmol, 1.0 equiv) and (9H-fluoren-9-yl) methyl 3,4-dihydroisoquinoline-2(1H)-carboxylate (**2h**, 159 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **3ah** as a colorless oil (78 mg, 55% yield).

^{1}H NMR (500 MHz, $CDCl_3$, rotamers seen) δ 7.85-7.70 (m, 2H), 7.70-7.50 (m, 2H), 7.47-7.40 (m, 2H), 7.40-7.23 (m, 10H), 7.23-7.16 (m, 1H), 5.46 (s, 0.5H), 5.22 (s, 0.5H), 4.64-4.42 (m, 2H), 4.36-4.24 (m, 1H), 4.23-4.14 (m, 0.5H), 4.06-3.94 (m, 0.5H), 3.78-3.66 (m, 0.5H), 3.60-3.48 (m, 0.5H), 3.06-2.74 (m, 4H).

^{13}C NMR (125 MHz, $CDCl_3$, rotamers seen) δ 155.4, 144.1, 144.0, 143.9, 141.33, 141.31, 135.6, 135.4, 134.4, 134.3, 131.5, 128.9, 128.5, 128.2, 128.1, 127.8, 127.64, 127.57, 127.4, 127.22, 127.19, 127.1, 127.0, 126.3, 126.1, 125.0, 124.9, 123.6, 123.5, 119.94, 119.90, 86.7, 86.5, 83.1, 82.9, 67.5, 67.4, 53.9, 53.8, 47.3, 39.4, 38.6, 28.7, 28.3, 27.6, 27.2.

HRMS (ESI) calcd for $C_{33}H_{28}O_2N$ $[M+H]^+$: 470.2115, found: 470.2126.



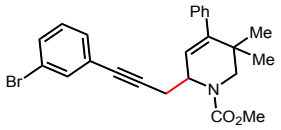
3mi

Methyl 6-(3-(3-chlorophenyl)prop-2-yn-1-yl)-3,3-dimethyl-4-phenyl-3,6-dihydropyridine-1(2H)-carboxylate (3mi): Prepared following general procedure A, using 1-chloro-3-(prop-1-yn-1-yl)benzene (**1m**, 45 mg, 0.3 mmol, 1.0 equiv) and methyl 3,3-dimethyl-4-phenyl-3,6-dihydropyridine-1(2H)-carboxylate (**2i**, 110 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **3mi** as a colorless oil (72 mg, 61% yield).

^{1}H NMR (400 MHz, $CDCl_3$, rotamers seen) δ 7.42-7.37 (m, 1H), 7.37-7.25 (m, 5H), 7.25-7.16 (m, 3H), 5.66 (s, 1H), 4.83 (s, 0.5H), 4.73 (s, 0.5H), 4.10-3.96 (m, 0.5H), 3.75-3.85 (m, 0.5H), 3.81 (s, 3H), 3.18 (d, J = 12.4 Hz, 0.5H), 3.05 (d, J = 12.8 Hz, 0.5H), 2.80 (d, J = 12.0 Hz, 2H), 1.26 (s, 3H), 0.91 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, rotamers seen) δ 156.2, 156.1, 148.3, 147.9, 141.0, 134.1, 131.4, 129.6, 129.5, 128.5, 128.1, 127.8, 126.9, 125.4, 125.3, 124.6, 124.1, 88.3, 87.9, 81.5, 52.8, 52.2, 51.5, 35.5, 25.8, 24.9, 24.7, 24.3.

HRMS (ESI) calcd for $C_{24}H_{25}O_2NCl$ $[M+H]^+$: 394.1574, found: 394.1579.



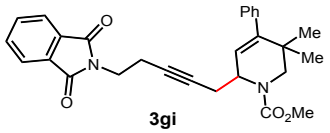
3ni

Methyl 6-(3-(3-bromophenyl)prop-2-yn-1-yl)-3,3-dimethyl-4-phenyl-3,6-dihydropyridine-1(2H)-carboxylate (3ni): Prepared following general procedure A, using 1-bromo-3-(prop-1-yn-1-yl)benzene (**1n**, 59 mg, 0.3 mmol, 1.0 equiv) and methyl 3,3-dimethyl-4-phenyl-3,6-dihydropyridine-1(2H)-carboxylate (**4i**, 110 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **3ni** as a colorless oil (85 mg, 65% yield).

^{1}H NMR (500 MHz, $CDCl_3$, rotamers seen) δ 7.58-7.52 (m, 1H), 7.43 (d, *J* = 8.0 Hz, 1H), 7.37-7.29 (m, 4H), 7.25-7.19 (m, 2H), 7.16 (t, *J* = 8.0 Hz, 1H), 5.66 (s, 0.5H), 5.65 (s, 0.5H), 4.83 (s, 0.5H), 4.72 (s, 0.5H), 4.03 (d, *J* = 13.0 Hz, 0.5H), 3.84 (d, *J* = 13.0 Hz, 0.5H), 3.81 (s, 3H), 3.18 (d, *J* = 13.0 Hz, 0.5H), 3.04 (d, *J* = 12.5 Hz, 0.5H), 2.90-2.68 (m, 2H), 1.26 (s, 1.5H), 1.24 (s, 1.5H), 0.91 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$, rotamers seen) δ 156.2, 156.0, 148.3, 147.9, 141.0, 134.3, 131.0, 130.1, 129.7, 128.5, 127.8, 126.9, 125.5, 124.6, 124.1, 122.1, 88.5, 88.1, 81.4, 52.9, 52.7, 52.2, 51.4, 35.5, 25.7, 24.9, 24.7, 24.3.

HRMS (ESI) calcd for $C_{24}H_{25}O_2NBr$ $[M+H]^+$: 438.1078, found: 438.1063.



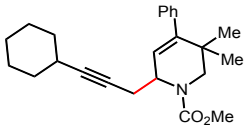
3gi

Methyl 6-(5-(1,3-dioxoisoindolin-2-yl)pent-2-yn-1-yl)-3,3-dimethyl-4-phenyl-3,6-dihydropyridine-1(2H)-carboxylate (3gi): Prepared following general procedure A, using 2-(pent-3-yn-1-yl)isoindoline-1,3-dione (**1g**, 64 mg, 0.3 mmol, 1.0 equiv) and methyl 3,3-dimethyl-4-phenyl-3,6-dihydropyridine-1(2H)-carboxylate (**2i**, 110 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with 20% ethyl acetate in hexane to provide the product **3gi** as a pale-yellow oil (81 mg, 59% yield).

^{1}H NMR (400 MHz, $CDCl_3$, rotamers seen) δ 7.84-7.74 (m, 2H), 7.74-7.64 (m, 2H), 7.36-7.26 (m, 3H), 7.22-7.12 (m, 2H), 5.56 (s, 1H), 4.64 (s, 0.5H), 4.52 (s, 0.5H), 3.98-3.66 (m, 6H), 3.02 (d, *J* = 11.6 Hz, 0.5H), 2.87 (d, *J* = 12.4 Hz, 0.5H), 2.66-2.65 (m, 2H), 2.54-2.32 (m, 2H), 1.19 (s, 3H), 0.86 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, rotamers seen) δ 168.0, 156.2, 155.9, 147.7, 147.4, 141.0, 133.9, 132.0, 128.6, 127.7, 126.8, 124.8, 124.2, 123.3, 78.5, 52.7, 52.0, 51.6, 36.9, 35.4, 25.7, 24.7, 24.1, 23.5, 18.8;

HRMS (ESI) calcd for $C_{28}H_{29}O_4N_2$ $[M+H]^+$: 457.2127, found: 457.2130.



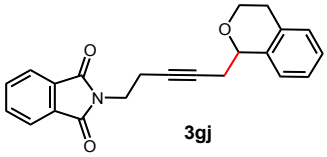
3ii

Methyl 6-(3-cyclohexylprop-2-yn-1-yl)-3,3-dimethyl-4-phenyl-3,6-dihydropyridine-1(2H)-carboxylate (3ii): Prepared following general procedure A, using prop-1-yn-1-ylcyclohexane (**1i**, 36.6 mg, 0.3 mmol, 1.0 equiv) and methyl 3,3-dimethyl-4-phenyl-3,6-dihydropyridine-1(2H)-carboxylate (**2i**, 110 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **3ii** as a colorless oil (62 mg, 57% yield).

^{1}H NMR (500 MHz, $CDCl_3$, rotamers seen) δ 7.45-7.10 (m, 5H), 5.62 (s, 1H), 4.68 (s, 0.5H), 4.57 (s, 0.5H), 3.97 (d, *J* = 12.5 Hz, 0.5H), 3.87-3.65 (m, 3.5H), 3.19 (d, *J* = 12.0 Hz, 0.5H), 3.05 (d, *J* = 12.0 Hz, 0.5H), 2.70-2.45 (m, 2H), 2.40-2.25 (m, 1H), 1.85-1.65 (m, 4H), 1.57-1.45 (m, 1H), 1.45-1.35 (m, 2H), 1.35-1.15 (m, 6H), 0.90 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$, rotamers seen) δ 156.3, 155.9, 147.7, 147.2, 141.3, 128.5, 127.7, 126.8, 125.2, 124.7, 87.1, 86.9, 76.5, 76.2, 52.7, 52.6, 52.3, 51.7, 51.6, 35.3, 33.0, 29.2, 25.9, 24.9, 24.7, 24.2, 23.6.

HRMS (ESI) calcd for $C_{24}H_{32}O_2N$ $[M+H]^+$: 366.2428, found: 366.2439.



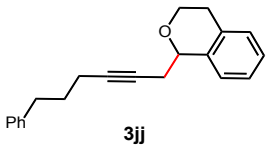
3gj

2-(5-(Isochroman-1-yl)pent-3-yn-1-yl)isoindoline-1,3-dione (3gj): Prepared following general procedure A, using 2-(pent-3-yn-1-yl)isoindoline-1,3-dione (**1g**, 64 mg, 0.3 mmol, 1.0 equiv) and isochroman (**2j**, 60.3 mg, 0.45 mmol, 1.5 equiv) in $CHCl_3$ (0.2 M). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **3gj** as a colorless oil (53 mg, 51% yield, > 20:1 r.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.90-7.80 (m, 2H), 7.75-7.65 (m, 2H), 7.17-7.03 (m, 4H), 4.85-4.75 (m, 1H), 4.12-4.05 (m, 1H), 3.82 (t, *J* = 7.5 Hz, 2H), 3.77-3.68 (m, 1H), 3.00-2.89 (m, 1H), 2.76-2.54 (m, 5H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 168.0, 136.9, 134.0, 133.9, 132.1, 128.8, 126.6, 126.1, 124.8, 123.3, 79.2, 77.7, 74.6, 63.3, 37.0, 28.9, 26.8, 18.9.

HRMS (ESI) calcd for $C_{22}H_{20}O_3N$ $[M+H]^+$: 346.1438, found: 346.1454.



3jj

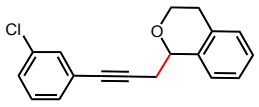
1-(6-Phenylhex-2-yn-1-yl)isochromane (3jj): Prepared following general procedure A, using hex-4-yn-1-ylbenzene (**1j**, 47 mg, 0.3 mmol, 1.0 equiv) and isochroman (**2j**, 60.3

mg, 0.45 mmol, 1.5 equiv) in $CHCl_3$ (0.2 M). The crude residue was purified by flash column chromatography, eluting with 33% DCM in hexanes to provide the product **3jj** as a colorless oil (39 mg, 45% yield, 15:1 r.r.).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.22-7.15 (m, 2H), 7.15-6.98 (m, 7H), 4.86-4.78 (m, 1H), 4.18-4.08 (m, 1H), 4.80-4.69 (m, 1H), 3.00-2.87 (m, 1H), 2.78-2.58 (m, 3H), 2.55 (t, *J* = 7.5 Hz, 2H), 2.13-2.03 (m, 2H), 1.73-1.62 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 141.9, 137.1, 134.2, 128.9, 128.6, 128.3, 126.6, 126.2, 125.8, 125.0, 81.6, 77.4, 74.9, 63.5, 34.6, 30.5, 29.0, 26.8, 18.3.

HRMS (ESI) calcd for $C_{21}H_{23}O$ $[M+H]^+$: 291.1743, found: 291.1778.



3mj

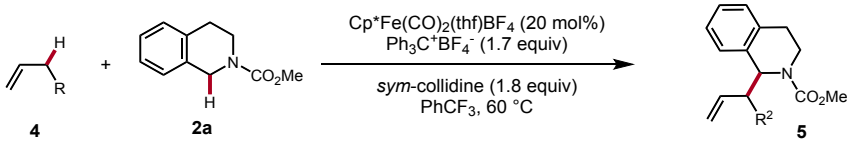
1-(3-(3-Chlorophenyl)prop-2-yn-1-yl)isochromane (3mj): Prepared following general procedure C, using 1-chloro-3-(prop-1-yn-1-yl)benzene (**1m**, 45 mg, 0.3 mmol, 1.0 equiv) and isochroman (**2j**, 60.3 mg, 0.45 mmol, 1.5 equiv) in $CHCl_3$ (0.2 M). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **3mj** as a colorless oil (34 mg, 40% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 (s, 1H), 7.30-7.14 (m, 7H), 5.04 (t, *J* = 5.0 Hz, 1H), 4.28-4.18 (m, 1H), 3.92-3.82 (m, 1H), 3.10-3.00 (m, 2H), 3.00-2.91 (m, 1H), 2.80-2.70 (m, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 136.7, 134.2, 133.9, 131.6, 129.8, 129.3, 128.9, 128.0, 126.8, 126.3, 125.4, 124.9, 88.5, 80.9, 74.5, 63.4, 29.0, 27.6.

HRMS (ESI) calcd for $C_{18}H_{16}OCl$ $[M+H]^+$: 283.0884, found: 283.0905.

General procedure B for propargylic C-H functionalization of alkenes.

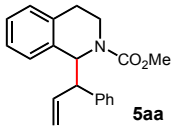


4 **2a** **5**

A reaction tube (13 mm × 100 mm, Fisherbrand, part # 14-959-35C) equipped with a magnetic stir bar was capped with a Teflon/silicone septum (Thermo/National part # C4015-66A) screw cap and flame dried under vacuum. The reaction tube was cooled under argon and transferred into a nitrogen-filled glovebox. In the glovebox, the suspension of carbamate (**2a**, 0.45 mmol, 1.5 equiv) and $Ph_3C^+BF_4^-$ (168 mg, 0.51 mmol, 1.7 equiv) in dry trifluorotoluene (1 mL) was stirred at room temperature for 3 hours to generate the iminium salt. Then $Cp^*Fe(CO)_2(thf)^+BF_4^-$ (20 mol%, 24.6 mg), alkene **4** (0.3 mmol, 1.0 equiv), trifluorotoluene (0.5 mL) and *sym*-collidine (72 µL, 0.54 mmol,

1.8 equiv) were added in rapid succession. The reaction tube was capped and removed from the glovebox. The reaction tube was placed in an oil bath, preheated to 60 °C, where it was stirred for 48 h. After completion of the reaction, the reaction mixture was cooled to room temperature. The crude mixture was concentrated in vacuo and purified by flash column chromatography to provide the desired product **5**.

Characterization Data for Products 5:



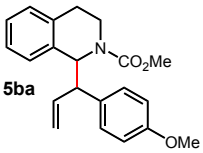
5aa

Methyl 1-(1-phenylallyl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (5aa): Prepared following general procedure B, using prop-1-yn-1-ylbenzene (**4a**, 39 μL, 0.3 mmol, 1.0 equiv) and methyl 3,4-dihydroisoquinoline-2(1H)-carboxylate (**2a**, 86.1 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **5aa** as a colorless oil (86 mg, 93% yield, 1:1 d.r.). The diastereoisomer could not be separated by column.

^{1}H NMR (500 MHz, $CDCl_3$, rotamers and diastereoisomer seen) δ 7.40-6.65 (m, 8.5H), 6.27-5.90 (m, 1.5H), 5.43-5.15 (m, 1H), 5.08-4.76 (m, 2H), 4.03- 3.30 (m, 6H), 2.93-2.45 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$, rotamers and diastereoisomer seen) δ 156.8, 156.4, 156.3, 155.9, 141.7, 141.5, 141.4, 141.3, 139.2, 138.4, 138.2, 138.1, 135.7, 135.3, 135.1, 135.0, 134.6, 134.4, 134.2, 134.1, 128.81, 128.75, 128.71, 128.67, 128.62, 128.58, 128.5, 128.4, 128.30, 128.28, 128.26, 128.23, 128.19, 127.9, 127.7, 127.2, 127.0, 126.91, 126.89, 126.78, 126.75, 126.5, 125.29, 125.26, 125.16, 125.11, 117.63, 117.56, 116.4, 115.9, 60.0, 59.73, 59.70, 59.6, 57.9, 57.6, 56.7, 56.5, 52.75, 52.67, 52.6, 52.4, 39.54, 39.46, 39.0, 38.7, 28.2, 27.9, 27.8, 27.7.

HRMS (ESI) calcd for $C_{20}H_{22}O_2N$ $[M+H]^+$: 308.1645, found: 308.1636.

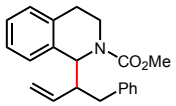


5ba

Methyl 1-(1-(4-methoxyphenyl)allyl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (5ba): Prepared following general procedure B, using 1-allyl-4-methoxybenzene (**4b**, 45 mg, 0.3 mmol, 1.0 equiv) and methyl 3,4-dihydroisoquinoline-2(1H)-carboxylate (**2a**, 86.1 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **5ba** as a colorless oil (85 mg, 84% yield, 1.3:1 d.r.). The diastereoisomer could be separated by column.

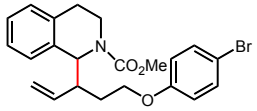
One isomer:

^{1}H NMR (500 MHz, $CDCl_3$, rotamers seen) δ 7.23-7.02 (m, 2H), 7.00 (t, *J* = 8.0 Hz, 2H), 6.93-6.85 (m, 1H), 6.85-6.73 (m, 2H), 6.35 (d, *J* = 8.0 Hz, 0.5H), 6.30 (d, *J* = 8.0 Hz, 0.5H), 6.27-6.05 (m, 1H), 5.39 (d, *J* = 9.0 Hz, 0.5H), 5.25 (d, *J* = 9.0 Hz, 0.5H), 5.15-4.97 (m, 2H), 4.10-3.67 (m, 7H), 3.58 (t, *J* = 9.0 Hz, 1H), 3.55-3.36 (m, 1H), 3.00-2.75 (m, 1H), 2.74-2.55 (m, 1H).
^{13}C NMR (125 MHz, $CDCl_3$, rotamers seen) δ 158.5, 158.4, 139.5, 138.7, 135.7, 135.4, 134.2, 134.1, 133.7, 133.5, 129.71, 129.66, 128.7, 128.2, 128.0, 127.8, 126.95, 126.86, 125.3, 125.2, 116.0, 115.5, 113.97, 113.89, 60.0, 59.8, 56.9, 56.7, 55.27, 55.25, 52.73, 52.68, 39.4, 38.7, 27.9, 27.7.
Another isomer:
^{1}H NMR (500 MHz, $CDCl_3$, rotamers seen) δ 7.24-7.00 (m, 5H), 6.95-6.76 (m, 3H), 6.15-5.95 (m, 1H), 5.41 (d, *J* = 7.0 Hz, 0.5H), 5.26 (d, *J* = 7.5 Hz, 0.5H), 5.05 (t, *J* = 9.0 Hz, 1H), 4.98 (d, *J* = 17.0 Hz, 0.5H), 4.91 (d, *J* = 17.0 Hz, 0.5H), 3.98-3.76 (m, 4H), 3.76-3.54 (m, 4H), 3.50-3.34 (m, 1H), 2.98-2.76 (m, 2H).
^{13}C NMR (125 MHz, $CDCl_3$, rotamers seen) δ 158.2, 158.1, 156.4, 155.9, 138.4, 138.3, 135.1, 135.0, 134.5, 134.4, 133.5, 133.2, 129.1, 129.0, 128.7, 128.6, 128.5, 128.3, 127.0, 126.8, 125.2, 125.0, 117.2, 117.1, 113.63, 113.59, 59.7, 59.6, 55.8, 55.5, 55.1, 52.5, 52.4, 39.5, 38.9, 28.1, 27.7.
HRMS (ESI) calcd for $C_{21}H_{24}O_3N$ $[M+H]^+$: 338.1750, found: 338.1741.



5ca

Methyl 1-(1-phenylbut-3-en-2-yl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (5ca): Prepared following general procedure B, using but-3-en-1-ylbenzene (**4c**, 45 μL, 0.3 mmol, 1.0 equiv) and methyl 3,4-dihydroisoquinoline-2(1H)-carboxylate (**2a**, 86.1 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **5ca** as a colorless oil (34 mg, 35% yield, 1.2:1 d.r.). The diastereoisomer could not be separated by column.
^{1}H NMR (500 MHz, $CDCl_3$, rotamers and diastereoisomer seen) δ 7.28-7.00 (m, 9H), 5.80-5.48 (m, 1H), 5.28-4.98 (m, 1H), 4.93-4.82 (m, 1H), 4.72-4.50 (m, 1H), 4.10-3.66 (m, 4H), 3.66-3.46 (m, 1H), 3.08-2.50 (m, 5H).
^{13}C NMR (125 MHz, $CDCl_3$, rotamers and diastereoisomer seen) δ 156.96, 156.91, 156.48, 146.46, 140.42, 140.30, 140.11, 140.07, 138.62, 138.47, 138.30, 138.09, 136.70, 136.24, 135.80, 135.36, 134.80, 134.71, 134.58, 134.53, 129.24, 129.19, 129.14, 128.99, 128.70, 128.55, 128.31, 128.20, 128.09, 128.03, 127.69, 127.12, 127.09, 126.99, 125.96, 125.90, 125.84, 125.77, 125.42, 125.31, 117.94, 117.88, 117.45, 117.16, 59.28, 58.86, 58.68, 58.57, 53.35, 52.96, 52.74, 52.69, 52.66, 52.52, 52.35, 40.31, 39.85, 39.68, 38.52, 38.38, 38.36, 38.26, 28.16, 27.98, 27.89, 27.68.
HRMS (ESI) calcd for $C_{21}H_{24}O_2N$ $[M+H]^+$: 322.1802, found: 322.1800.



5da

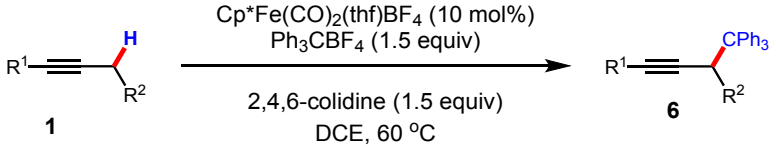
Methyl 1-(5-(4-bromophenoxy)pent-1-en-3-yl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (5da): Prepared following general procedure B, using 1-bromo-4-(pent-4-en-1-yloxy)benzene (**4d**, 72 mg, 0.3 mmol, 1.0 equiv) and methyl 3,4-dihydroisoquinoline-2(1H)-carboxylate (**2a**, 86.1 mg, 0.45 mmol, 1.5 equiv). The crude residue was purified by flash column chromatography, eluting with pure DCM to provide the product **5da** as a colorless oil (43 mg, 33% yield, 1.2:1 d.r.). The diastereoisomer could not be separated by column.

^{1}H NMR (500 MHz, $CDCl_3$, rotamers and diastereoisomer seen) δ 7.40-7.27 (m, 2H), 7.23-7.03 (m, 4H), 6.80-6.65 (m, 2H), 5.81-5.50 (m, 1H), 5.20-4.75 (m, 3H), 4.10-3.75 (m, 3H), 3.75-3.67 (m, 3H), 3.63-3.43 (m, 1H), 3.00-2.80 (m, 2H), 2.77-2.63 (m, 1H), 2.20-2.00 (m, 1H), 1.87-1.70 (m, 1H).

^{13}C NMR (125 MHz, $CDCl_3$, rotamers and diastereoisomer seen) δ 158.06, 158.01, 156.96, 156.90, 156.40, 156.35, 138.54, 138.46, 138.31, 137.95, 136.25, 135.84, 135.75, 135.35, 134.72, 134.54, 134.39, 132.20, 132.18, 132.16, 129.00, 128.79, 128.64, 128.59, 128.30, 127.97, 127.73, 127.22, 127.10, 127.02, 126.50, 126.33, 126.00, 125.84, 125.38, 125.25, 118.40, 118.31, 117.78, 117.27, 116.40, 116.35, 116.29, 112.81, 112.73, 112.68, 112.66, 66.26, 65.99, 65.83, 58.73, 58.53, 58.50, 58.37, 52.78, 52.74, 52.69, 52.57, 47.53, 47.30, 47.28, 47.08, 40.15, 40.02, 39.63, 39.37, 30.95, 30.81, 30.49, 28.08, 27.93, 27.78, 27.57.

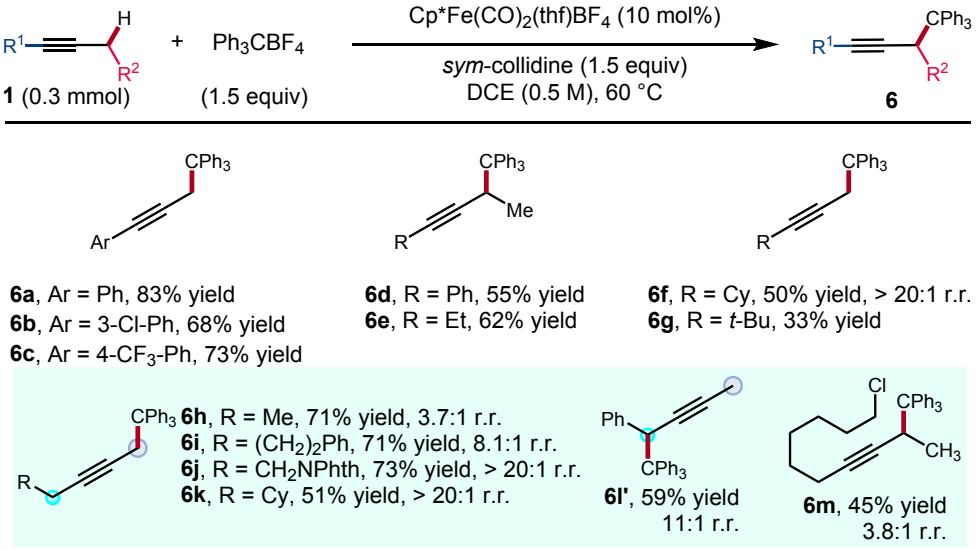
HRMS (ESI) calcd for $C_{22}H_{25}O_3NBr$ $[M+H]^+$: 433.1012, found: 433.1017.

General procedure C for propargylic C-H functionalization of alkynes.

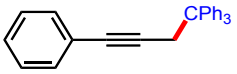


A reaction tube (13 mm × 100 mm, Fisherbrand, part # 14-959-35C) equipped with a magnetic stir bar was capped with a Teflon/silicone septum (Thermo/National part # C4015-66A) screw cap and flame dried under vacuum. The reaction tube was cooled under argon and transferred into an argon-filled glovebox. In the glovebox, $[Cp^*Fe(CO)_2(thf)]^+[BF_4]^-$ (12.2 mg, 10 mol%), $Ph_3C^+BF_4^-$ (150 mg, 0.45 mmol, 1.5 equiv.), dry 1,2-dichloroethane (0.6 mL), alkyne **1** (0.3 mmol, 1.0 equiv) and 2,4,6-

collidine (60 µL, 0.45 mmol, 1.5 equiv) were added in rapid succession. The reaction tube was capped and removed from the glovebox. The reaction tube was placed in an oil bath, preheated to 60 °C, where it was stirred for 48 h. After completion of the reaction, the reaction mixture was cooled to room temperature. The crude mixture was concentrated *in vacuo* and purified by flash column chromatography to provide the desired product.



Characterization data for products 6:



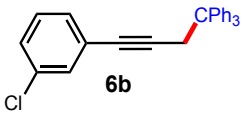
6a

But-3-yne-1,1,1,4-tetrayltetrabenzene (6a): Prepared following general procedure C using prop-1-yn-1-ylbenzene (**1a**, 39 µL, 0.3 mmol, 1.0 equiv). The reaction mixture was quenched after 48 h, and the crude residue was purified by flash column chromatography, eluting with a gradient of 5 to 6.3% DCM in hexanes to provide the title compound **6a** as a white solid (86 mg, 80% yield). **m.p.** = 101–102 °C.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.22-7.16 (m, 12H), 7.16-7.10 (m, 3H), 7.10-7.04 (m, 3H), 7.01-6.92 (m, 2H), 3.61 (s, 2H).

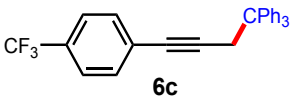
^{13}C NMR (125 MHz, $CDCl_3$) δ 146.8, 131.3, 129.3, 128.0, 127.8, 127.5, 126.3, 123.8, 88.6, 84.3, 56.6, 33.5.

HRMS (ASAP) calcd for $C_{28}H_{23}$ $[M+H]^+$: 359.1800, found: 359.1812.



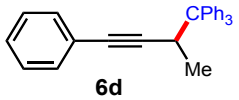
6b

(4-(3-chlorophenyl) but-3-yne-1,1,1-triyl) tribenzene (6b): Prepared following general procedure C using 1-chloro-3-(prop-1-yn-1-yl) benzene (**1m**, 45 mg, 0.3 mmol, 1.0 equiv). The reaction mixture was quenched after 48 h, and the crude residue was purified by flash column chromatography, eluting with a gradient of 5 to 6.3% DCM in hexanes to provide the title compound **6b** as a white solid (80 mg, 68% yield). **m.p.** = 99–100 °C.
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50-7.22 (m, 15H), 7.21-7.16 (m, 1H), 7.15-7.08 (m, 1H), 7.05 (s, 1H), 6.98-6.91 (m, 1H), 3.72 (s, 2H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 146.7, 133.8, 131.2, 129.4, 129.2, 127.83, 127.77, 126.4, 125.4, 90.1, 83.1, 56.6, 33.5.
HRMS (ASAP) calcd for $C_{28}H_{22}Cl$ $[M+H]^+$: 393.1404, found: 393.1418.



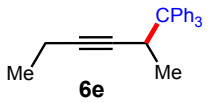
6c

(4-(4-(trifluoromethyl)phenyl)but-3-yne-1,1,1-triyl)tribenzene (6c): Prepared following general procedure C using 1-(prop-1-yn-1-yl)-4-(trifluoromethyl)benzene (**1o**, 55.2 mg, 0.3 mmol, 1.0 equiv). The reaction mixture was quenched after 48 h, and the crude residue was purified by flash column chromatography, eluting with a gradient of 5 to 6.3% DCM in hexanes to provide the title compound **6c** as a white solid (93 mg, 73% yield). **m.p.** = 67–68 °C.
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.47 (d, J = 8.0 Hz, 2H), 7.38-7.26 (m, 15H), 7.20-7.14 (m, 2H), 3.76 (s, 2H).
^{19}F NMR (470 MHz, $CDCl_3$, rotamers seen) δ –62.8.
^{13}C NMR (125 MHz, $CDCl_3$) δ 146.6, 131.5, 129.2, 128.9 (q, $J_{C–F}$ = 145 Hz), 127.9, 126.9 (q, $J_{C–F}$ = 150 Hz), 126.4, 124.9 (q, $J_{C–F}$ = 3.8 Hz), 124.0 (q, $J_{C–F}$ = 262.5 Hz), 91.5, 83.3, 56.6, 33.6.
HRMS (ASAP) calcd for $C_{29}H_{22}CF_3$ $[M+H]^+$: 427.1674, found: 427.1681.



6d

(2-methylbut-3-yne-1,1,1,4-tetrayl) tetrabenzene (6d): Prepared following general procedure C using but-1-yn-1-ylbenzene (**1f**, 42 µL, 0.3 mmol, 1.0 equiv). The reaction mixture was quenched after 48 h, and the crude residue was purified by flash column chromatography, eluting with a gradient of 5 to 6.3% DCM in hexanes to provide the title compound **6d** as a white solid (61 mg, 55% yield). **m.p.** = 91–92 °C.
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.47 (d, J = 8.0 Hz, 6H), 7.30 (t, J = 7.5 Hz, 6H), 7.25-7.18 (m, 6H), 7.17-7.11 (m, 2H), 4.56 (q, J = 7.0 Hz, 1H), 1.22 (d, J = 7.0 Hz, 3H).
^{13}C NMR (125 MHz, $CDCl_3$) δ 145.2, 131.3, 130.0, 128.1, 127.44, 127.37, 126.0, 124.0, 93.3, 84.4, 61.0, 33.3, 18.2.
HRMS (ASAP) calcd for $C_{29}H_{25}$ $[M+H]^+$: 373.1956, found: 373.1977.



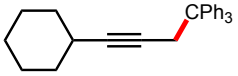
6e

(2-methylhex-3-yne-1,1,1-triyl) tribenzene (6e): Prepared following general procedure C using 3-hexyne (**1l**, 33 µL, 0.3 mmol, 1.0 equiv). The reaction mixture was quenched after 48 h, and the crude residue was purified by flash column chromatography (Hexanes) to provide the title compound **6e** as a colorless liquid (61 mg, 62% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50-7.36 (m, 6H), 7.34-7.25 (m, 6H), 7.25-7.16 (m, 3H), 4.33 (qt, J = 6.8, 2.0 Hz, 1H), 2.02 (qd, J = 7.6, 2.0 Hz, 2H), 1.11 (d, J = 6.8 Hz, 3H), 0.93 (t, J = 7.6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 145.3, 130.0, 127.3, 125.8, 85.6, 82.5, 60.8, 32.4, 18.5, 13.9, 12.5.

HRMS (ESI) calcd for $C_{25}H_{25}$ $[M+H]^+$: 325.1951, found: 325.1963.



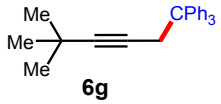
6f

(4-cyclohexylbut-3-yne-1,1,1-triyl)tribenzene (6f): Prepared following general procedure C using prop-1-yn-1-ylcyclohexane (**1i**, 36.6 mg, 0.3 mmol, 1.0 equiv). The reaction mixture was quenched after 48 h, and the crude residue was purified by flash column chromatography, eluting with a gradient of 5 to 6.3% DCM in hexanes to provide the title compound **6f** as a colorless liquid (55 mg, 50% yield, >20:1 r.r.).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38-7.19 (m, 15H), 3.50 (d, J = 2.0 Hz, 2H), 2.24 (s, 1H), 1.55-1.33 (m, 5H), 1.27-1.08 (m, 5H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 147.1, 129.4, 127.7, 126.1, 88.3, 78.7, 56.4, 32.9, 32.3, 28.7, 26.0, 24.3.

HRMS (ASAP) calcd for $C_{28}H_{29}$ $[M+H]^+$: 365.2269, found: 365.2289.



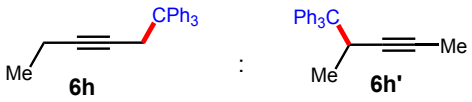
6g

(5,5-dimethylhex-3-yne-1,1,1-triyl) tribenzene (6g): Prepared following general procedure C using 4,4-dimethylpent-2-yne (**1r**, 40 µL, 0.3 mmol, 1.0 equiv). The reaction mixture was quenched after 48 h, and the crude residue was purified by flash column chromatography, eluting with a gradient of 5 to 6.3% DCM in hexanes to provide the title compound **6g** as a colorless liquid (34 mg, 33% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31-7.20 (m, 15H), 3.44 (s, 2H), 0.96 (s, 9H).

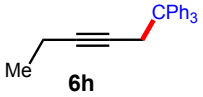
^{13}C NMR (125 MHz, $CDCl_3$) δ 147.1, 129.4, 127.6, 126.1, 100.0, 92.9, 56.5, 32.8, 30.7, 27.2.

HRMS (ASAP) calcd for $C_{26}H_{27}$ $[M+H]^+$: 339.2113, found: 339.2131.



6h : **6h'** = 3.7 : 1

Prepared following general procedure C using pent-2-yne (**1p**, 29 µL, 0.3 mmol, 1.0 equiv). The reaction mixture was quenched after 48 h, and the crude residue was purified by flash column chromatography, eluting with a gradient of 5 to 6.3% DCM in hexanes to provide the title compound **6h** and **6h'** as a colorless oil (66 mg, 71% yield, 3.7:1 r.r.). The regioisomeric ratio was determined by ^{1}H NMR of the crude material.



hex-3-yne-1,1,1-triyltribenzene (6h):

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38-7.18 (m, 15H), 3.49 (t, *J* = 2.4 Hz, 2H), 2.00 (qt, *J* = 7.6, 2.4 Hz, 2H), 0.90 (t, *J* = 7.6 Hz, 3H).

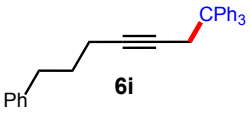
^{13}C NMR (100 MHz, $CDCl_3$) δ 147.1, 129.4, 127.7, 126.1, 85.6, 77.9, 56.4, 32.8, 13.9, 12.4.

HRMS (ESI) calcd for $C_{24}H_{22}$ $[M+H]^+$: 311.1794, found: 311.1804.

(2-methylpent-3-yne-1,1,1-triyl)tribenzene (6h'):

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45-7.35 (m, 5H), 7.30-7.12 (m, 10H), 4.37-4.20 (m, 1H), 1.70-1.55 (m, 3H), 1.08 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 145.5, 129.9, 127.3, 125.8, 81.9, 79.6, 60.8, 32.3, 18.5, 3.6.

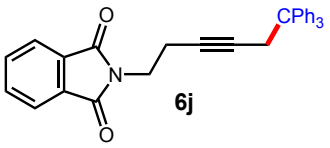


Hept-3-yne-1,1,1,7-tetrayltetrabenzene (6i): Prepared following general procedure C using hex-4-yn-1-ylbenzene (**1j**, 47 mg, 0.3 mmol, 1.0 equiv). The reaction mixture was quenched after 48 h, and the crude residue was purified by flash column chromatography, eluting with a gradient of 5 to 6.3% DCM in hexanes to provide the title compound **6i** as a colorless oil (85 mg, 71% yield, 8.1:1 r.r.). The regioisomeric ratio was determined by ^{1}H NMR of the crude material.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-7.24 (m, 18H), 7.08-7.03 (m, 2H), 3.55 (t, *J* = 2.4 Hz, 2H), 2.44 (t, *J* = 7.6 Hz, 2H), 2.07-1.98 (m, 2H), 1.63-1.53 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 147.1, 141.9, 129.4, 128.6, 128.2, 127.8, 126.2, 125.7, 83.6, 79.1, 56.4, 34.3, 32.9, 30.2, 18.0.

HRMS (ESI) calcd for $C_{31}H_{29}$ $[M+H]^+$: 401.2264, found: 401.2278.



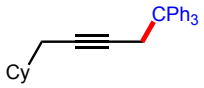
6j

2-(6,6,6-triphenylhex-3-yn-1-yl) isoindoline-1,3-dione (6j): Prepared following general procedure C using 2-(pent-3-yn-1-yl)isoindoline-1,3-dione (**1g**, 64 mg, 0.3 mmol, 1.0 equiv). The reaction mixture was quenched after 48 h, and the crude residue was purified by flash column chromatography, eluting with a gradient of 6 to 12% ethyl acetate in hexanes to provide the title compound **6j** as a light-yellow solid (100 mg, 73% yield, >20:1 r.r.). The regioisomeric ratio was determined by ^{1}H NMR of the crude material. **m.p.** = 158-159 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90-7.80 (m, 2H), 7.78-7.68 (m, 2H), 7.38-7.08 (m, 15H), 3.58 (t, J = 7.6 Hz, 2H), 3.45 (t, J = 2.4 Hz, 2H), 2.48-2.32 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 167.9, 146.8, 133.9, 132.1, 129.2, 127.7, 126.2, 123.3, 80.5, 79.8, 56.2, 36.8, 32.8, 18.6.

HRMS (ESI) calcd for $C_{32}H_{26}O_2N$ $[M+H]^+$: 456.1958, found: 456.1955.



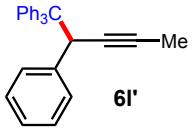
6k

(5-cyclohexylpent-3-yne-1,1,1-triyl)tribenzene (6k): Prepared following general procedure C using but-2-yn-1-ylcyclohexane (**1q**, 40.8 mg, 0.3 mmol, 1.0 equiv). The reaction mixture was quenched after 48 h, and the crude residue was purified by flash column chromatography, eluting with a gradient of 5 to 6.3% DCM in hexanes to provide the title compound **6k** as a colorless oil (58 mg, 51% yield, >20:1 r.r.).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25-7.05 (m, 15H), 3.39 (t, J = 2.4 Hz, 2H), 1.82-1.72 (m, 2H), 1.54-1.44 (m, 3H), 1.38-1.28 (m, 2H), 1.12-0.88 (m, 4H), 0.70-0.54 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 147.1, 129.3, 127.7, 126.1, 82.9, 79.2, 56.3, 37.4, 32.9, 32.3, 26.6, 26.2.

HRMS (ESI) calcd for $C_{29}H_{31}$ $[M+H]^+$: 379.2420, found: 379.2432.



6l'

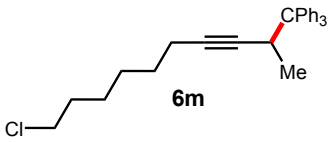
Pent-3-yne-1,1,1,2-tetrayltetrabenzene (6l'): Prepared following general procedure A using but-2-yn-1-ylbenzene (**1s**, 39 mg, 0.3 mmol, 1.0 equiv). The reaction mixture was quenched after 48 h, and the crude residue was purified by flash column chromatography, eluting with a gradient of 5 to 6.3% DCM in hexanes to provide the title compound **6l'** as

a white solid (66 mg**,** 59% yield, 1:11 r.r.). The regioisomeric ratio was determined by ^{1}H NMR of the crude material. **m.p.** = 173-174 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50-7.33 (m, 6H), 7.28-7.15 (m, 9H), 7.15-7.08 (m, 1 H), 7.07-6.95 (m, 2H), 6.93-6.75 (m, 2H), 5.49 (s, 1H), 1.73 (d, J = 1.6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 144.7, 138.8, 131.3, 130.8, 127.0, 126.8, 126.7, 125.9, 82.4, 80.4, 63.6, 45.1, 3.7.

HRMS (ESI) calcd for $C_{29}H_{25}$ $[M+H]^+$: 373.1951, found: 373.1959.



6m

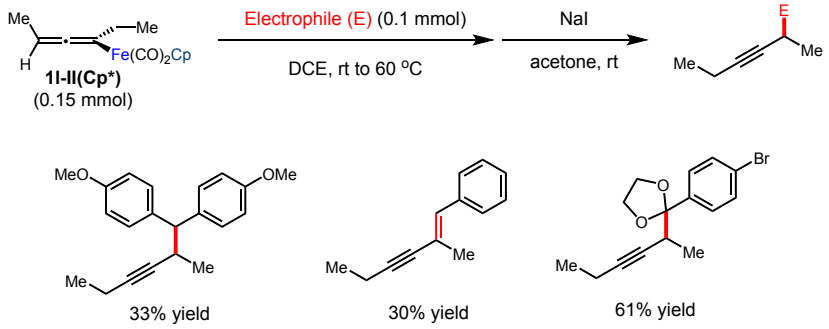
(10-chloro-2-methyldec-3-yne-1,1,1-triyl) tribenzene (6m): Prepared following general procedure C using 10-chlorodec-3-yne (**1t**, 57 µL, 0.3 mmol, 1.0 equiv). The reaction mixture was quenched after 48 h, and the crude residue was purified by flash column chromatography, eluting with a gradient of 5 to 6.3% DCM in hexanes to provide the title compound **6m** as a colorless oil (56 mg, 45% yield, 3.8:1 r.r.). The regioisomeric ratio was determined by ^{1}H NMR of the crude material.

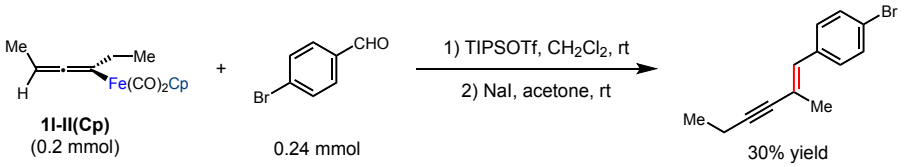
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.48-7.38 (m, 6H), 7.33-7.28 (m, 6H), 7.25-7.19 (m, 3H), 4.42-4.29 (m, 1H), 3.59-3.49 (m, 2H), 2.05 (td, J = 7.0, 2.0 Hz, 2H), 1.78-1.67 (m, 2H), 1.40-1.26 (m, 4H), 1.22-1.14 (m, 2H), 1.11 (d, J = 6.5 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 146.2, 130.2, 127.3, 125.8, 83.9, 83.3, 60.7, 45.0, 32.5, 28.4, 27.7, 26.4, 18.6, 18.5.

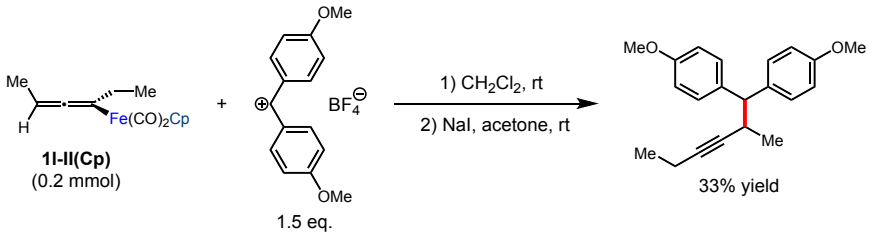
HRMS (ASAP) calcd for $C_{29}H_{32}Cl$ $[M+H]^+$: 415.2193, found: 415.2212.

Additional stoichiometric functionalization experiments

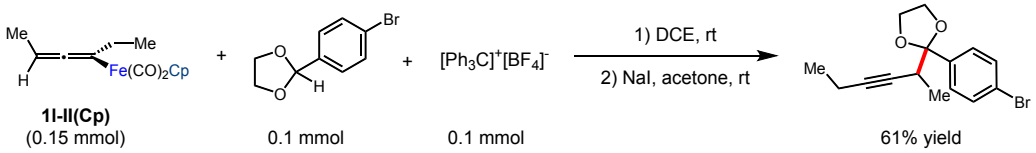




A reaction tube equipped with a magnetic stir bar was capped with a Teflon/silicone septum screw cap and flame dried under vacuum. The solution of **1l-II(Cp)** (approx. 0.2 mmol) in CH_2Cl_2 (2 mL) was added dropwise to a stirred solution of 4-bromobenzaldehyde (40 mg, 0.24 mmol, 1.2 equiv) and triisopropylsilyl trifluoromethanesulfonate (TIPSOTf, 64 µL, 0.24 mmol, 1.2 equiv) in CH_2Cl_2 (2 mL). The reaction mixture was stirred at rt for 1 h. Then acetone (2 mL) and sodium iodide (30 mg, 0.2 mmol) were added in succession, and stirred at rt for another 10 min. The crude mixture was concentrated *in vacuo* and purified by preparative thin-layer chromatography to provide the desired product.



A reaction tube equipped with a magnetic stir bar was capped with a Teflon/silicone septum screw cap and flame dried under vacuum. The solution of **1l-II(Cp)** (approx. 0.2 mmol) in CH_2Cl_2 (2 mL) was added dropwise to a stirred solution of 4,4'-bis(dimethoxy)benzhydrilium tetrafluoroborate (68 mg, 0.3 mmol, 1.5 equiv) in CH_2Cl_2 (1 mL) at rt for 1 h. Acetone (2 mL) and sodium iodide (30 mg, 0.2 mmol) were then added in succession, and stirred at rt for another 10 min. The crude mixture was concentrated *in vacuo* and purified by preparative thin-layer chromatography to provide the desired product.

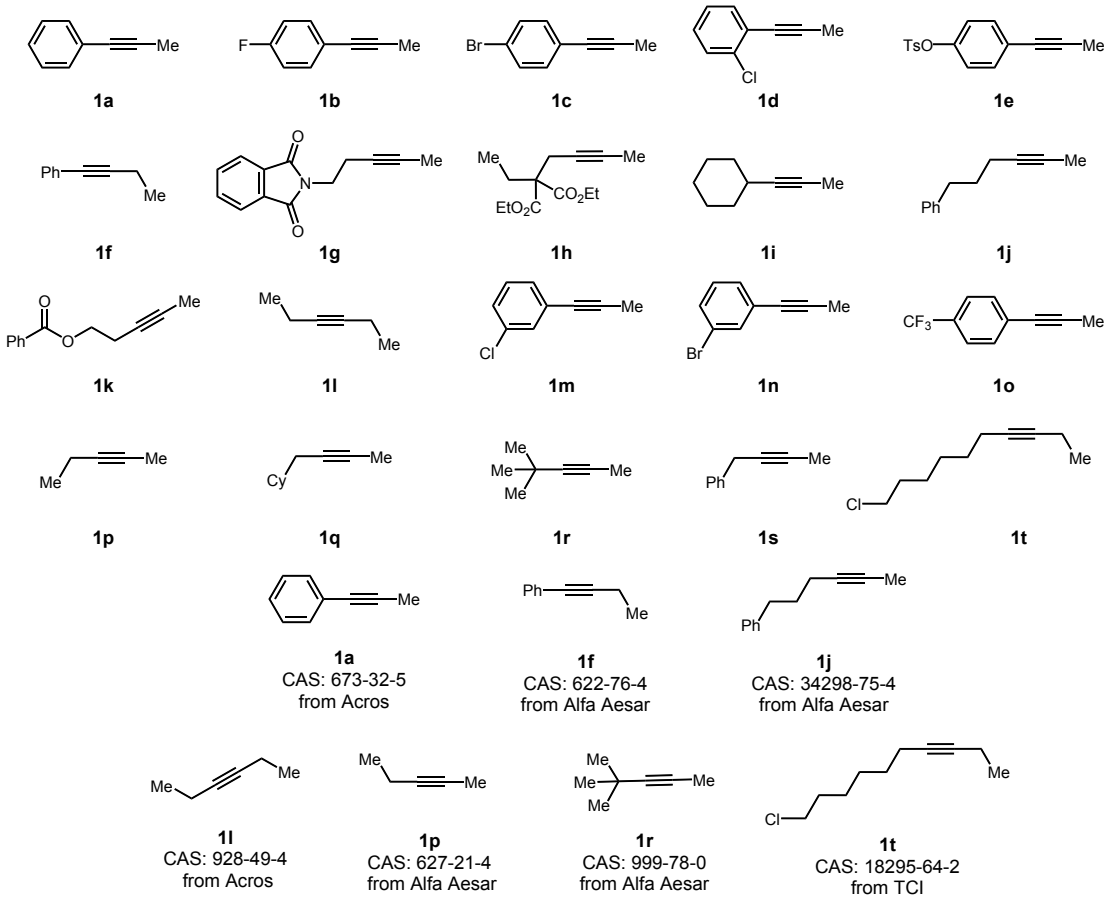


A reaction tube equipped with a magnetic stir bar was capped with a Teflon/silicone septum screw cap and flame dried under vacuum. The reaction tube was cooled under argon and transferred into a nitrogen-filled glovebox. In the glovebox, the suspension of 2-(4-bromophenyl)-1,3-dioxolane (23 mg, 0.1 mmol) and $Ph_3C^+BF_4^-$ (33 mg, 0.1 mmol) in dry 1,2-dichloroethane (1 mL) was stirred at room temperature for 3 hours. Then the solution of **1l-II(Cp)** (approx. 0.15 mmol) in 1,2-dichloroethane (1 mL) was added dropwise at rt, and the reaction mixture was stirred at 60 °C for 2 h. Subsequently, acetone (2 mL) and sodium iodide (30 mg, 0.2 mmol) were added in succession, and

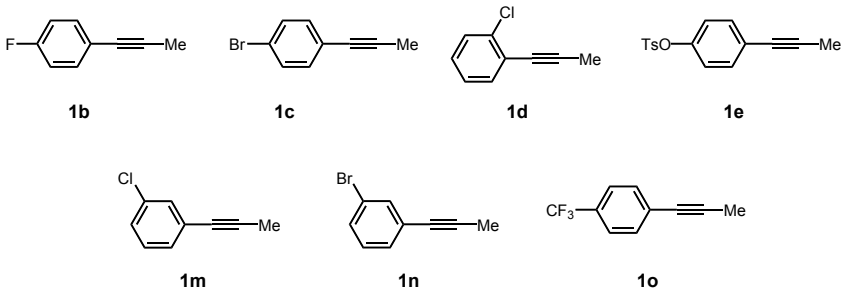
stirred at rt for another 10 min. The crude mixture was concentrated *in vacuo* and purified by preparative thin-layer chromatography to provide the desired product.

Synthesis of substrates and characterization data for new compounds:

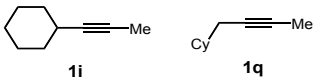
The alkyne substrates are list as following:



The alkynes **1a**, **1f**, **1j, 1l**, **1p**, **1r**, **1s** are commercial, and used directly without purification.

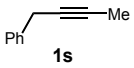


The alkynes were synthesized according to a known literature procedure and has been previously characterized.[5]



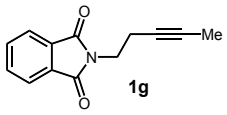
1i **1q**

The alkynes were synthesized according to a known literature procedure and has been previously characterized.[6]



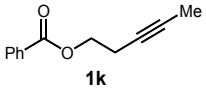
1s

The alkyne was synthesized according to a known literature procedure and has been previously characterized.[7]



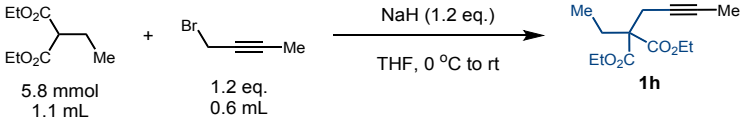
1g

The alkyne was synthesized according to a known literature procedure and has been previously characterized.[8]



1k

The alkyne was synthesized according to a known literature procedure and has been previously characterized.[9]

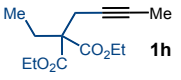


5.8 mmol
1.1 mL

1.2 eq.
0.6 mL

1h

The diethyl 2-ethylmalonate (1.1 mL, 5.8 mmol) was added to the suspension of NaH (278 mg, 1.2 equiv) in THF (12 mL) at 0 °C. The mixture was stirred at room temperature for 0.5 h. Then 1-bromobut-2-yne (0.6 mL, 1.2 equiv) was added to the above suspension dropwise, stirred at room temperature for another 1 h. After completion, quenched with saturated NH_4Cl solution, extracted with ethyl acetate for three times. The combined organic solution was washed with brine, dried (Na_2SO_4), filtered, and concentrated under reduced pressure. Purification by column chromatography (10% - 20% ethyl acetate in hexanes) afforded the product as colorless liquid (1.27 g, 91% yield).



1h

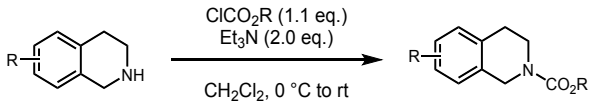
Diethyl 2-(but-2-yn-1-yl)-2-ethylmalonate (1h).

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.25-4.12 (m, 4H), 2.77-2.68 (m, 2H), 2.12-2.00 (m, 2H), 1.78-1.68 (m, 3H), 1.30-1.17 (m, 6H), 0.88-0.77 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 170.6, 78.3, 73.5, 61.2, 57.5, 24.9, 22.4, 14.0, 8.3, 3.4.

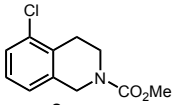
HRMS (ESI) calcd for $C_{13}H_{21}O_4$ $[M+H]^+$: 241.1434, found: 241.1439.

Synthesis of carbamates.



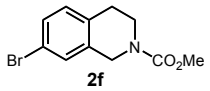
The *N*-carbamate starting materials **2a-2g** were synthesized according to the corresponding literatures[10] and has been previously characterized.

The chloroformate (1.1 equiv) was added dropwise to the solution of tetrahydroisoquinolines (1.0 equiv) and Et_3N (2.0 equiv) in CH_2Cl_2 (0.25 M) at 0 °C. The mixture was stirred at room temperature for 2 h. After completion, quenched with saturated NH_4Cl solution, extracted with CH_2Cl_2 for three times. The combined organic solution was washed with brine, dried (Na_2SO_4), filtered, and concentrated under reduced pressure. Purification by column chromatography (10% - 20% ethyl acetate in hexanes) afforded the product.



2c

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.14 (d, *J* = 8.0 Hz, 1H), 7.03 (t, *J* = 8.0 Hz, 1H), 6.92 (s, 1H), 4.52 (s, 2H), 3.66 (s, 3H), 3.62 (s, 2H), 2.78 (t, *J* = 5.5 Hz, 2H);
^{13}C NMR (125 MHz, $CDCl_3$) δ 155.9, 135.6, 134.3, 132.5, 127.2, 127.1, 124.7, 52.7, 45.7, 41.1, 26.6;



2f

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25-7.05 (m, 2H), 6.91 (d, *J* = 8.0 Hz, 1H), 4.49 (s, 2H), 3.66 (s, 3H), 3.58 (s, 2H), 2.69 (t, *J* = 4.8 Hz, 2H);
^{13}C NMR (100 MHz, $CDCl_3$) δ 156.0, 135.3, 133.5, 130.4, 129.6, 129.1, 119.8, 52.8, 45.3, 41.4, 28.3;

Reference

1. Reger, D. L.; Coleman, C. J. and McElligott, P. J. *J. Organomet. Chem.*, **1979**, *171*, 73.
2. Akita, M.; Kakuta, S.; Sugimoto, S.; Terada, M.; Tanaka, M.; Moro-oka, Y. *Organometallics*, **2001**, *20*, 2736.
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4. Akita, H.; Terada, M.; Tanaka, M.; Morooka, Y. *J. Organomet. Chem.*, **1996**, 510, 255.
5. Suero, M. G.; Bayle, E. D.; Collins, B. S. L.; Gaunt, M. J. *J. Am. Chem. Soc.* **2013**, *135*, 5332.
6. Mulzer, M.; Coates, G. W. *J. Org. Chem.,* **2014**, *79*, 11851.
7. Tan, E. H. P.; Lloyd-Jones, G. C.; Harvey, J. N.; Lennox, A. J. J.; Mills, B. M. *Angew. Chem. Int. Ed.* **2011**, *50*, 9602.
8. Beck, T. M.; Breit, B. *Eur. J. Org. Chem.* **2016**, 5839.
9. Dipl.-Chem, B. H.; Wu, X.; Brandhorst, K.; Priv.-Doz. J. G.; Daniliuc, C. G.; Jones, P. G.; Tamm, M. *Chem. Eur. J.* **2010**, *16*, 8868.
10. Strokker, G. E. *Tetrahedron Lett*. **1996**, *37*, 5453.

X-ray structures for products 3ab and 6d.

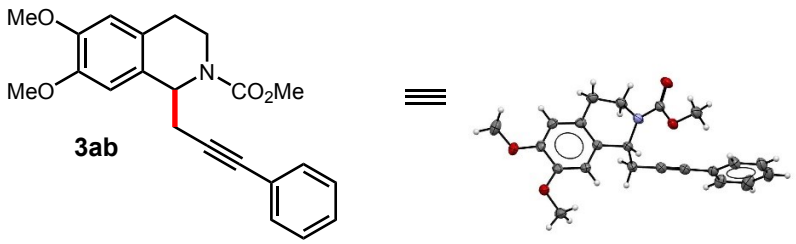


Figure S2. Molecular structure of 3ab (CCDC 1909840)

Datablock: wyd2_282

Bond precision: C-C = 0.0024 A Wavelength=1.54178

Cell: a=17.0265(10) b=14.1187(8) c=8.9890(5)
alpha=90 beta=118.511(2) gamma=90
Temperature: 150 K

	Calculated	Reported
Volume	1898.82(19)	1898.82(19)
Space group	C c	C c
Hall group	C -2yc	C -2yc
Moiety formula	C22 H23 N O4	?
Sum formula	C22 H23 N O4	C22 H23 N O4
Mr	365.41	365.41
Dx,g cm-3	1.278	1.278
Z	4	4
Mu (mm-1)	0.712	0.712
F000	776.0	776.0
F000'	778.40	
h,k,lmax	20,17,10	20,17,10
Nref	3606[1807]	3528
Tmin,Tmax	0.855,0.918	0.840,0.920
Tmin'	0.855	

Correction method= # Reported T Limits: Tmin=0.840 Tmax=0.920
AbsCorr = MULTI-SCAN

Data completeness= 1.95/0.98 Theta(max)= 70.057

R(reflections)= 0.0257(3509) wR2(reflections)= 0.0773(3528)

S = 1.074 Npar= 247

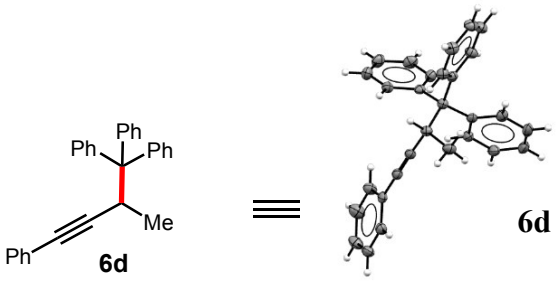


Figure S1. Molecular structure of 6d (CCDC 1909839)

Datablock: Yidong1_a

Bond precision: C-C = 0.0019 A Wavelength=1.54178

Cell: a=9.6092(4) b=24.7359(10) c=9.7874(4)
alpha=90 beta=116.350(2) gamma=90
Temperature: 150 K

	Calculated	Reported
Volume	2084.68(15)	2084.67(15)
Space group	P 21/c	P 21/c
Hall group	: -P 2ybc	-P 2ybc
Moiety formula	C29 H24	?
Sum formula	C29 H24	C29 H24
Mr	372.48	372.48
Dx,g cm-3	1.187	1.187
Z	4	4
Mu (mm-1)	0.503	0.503
F000	792.0	792.0
F000'	794.00	
h,k,lmax	11,29,11	11,29,11
Nref	3820	3689
Tmin,Tmax	0.947,0.980	0.920,0.980
Tmin'	0.913	

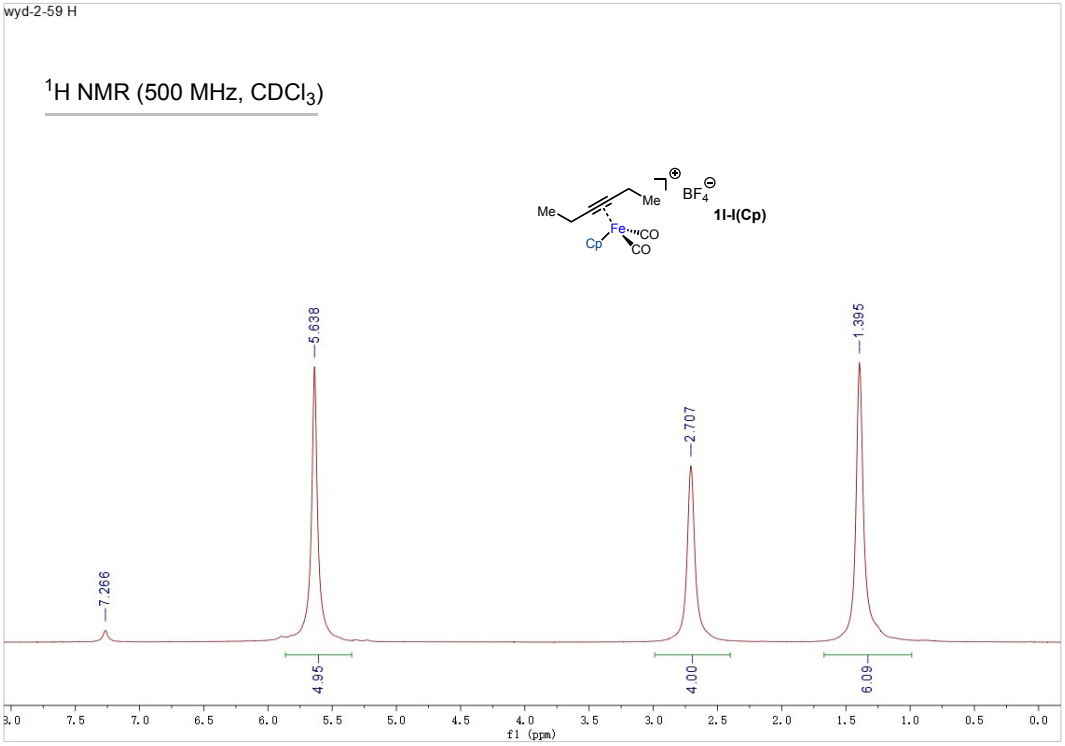
Correction method= # Reported T Limits: Tmin=0.920 Tmax=0.980
AbsCorr = MULTI-SCAN

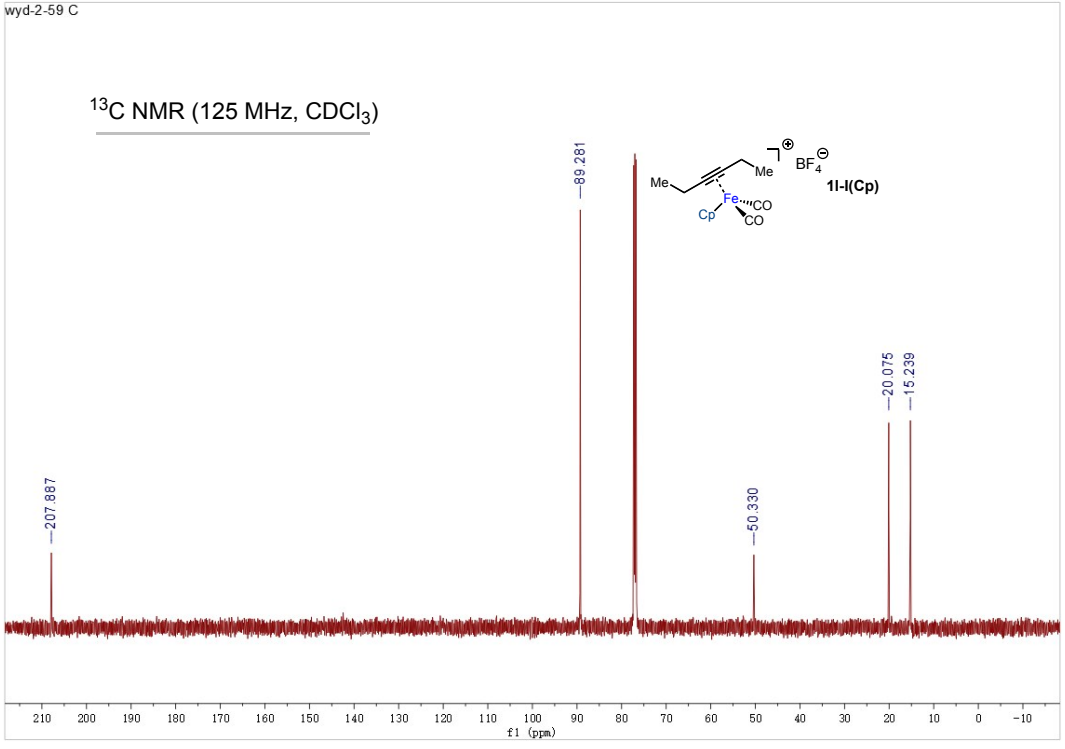
Data completeness= 0.966 Theta(max)= 68.240

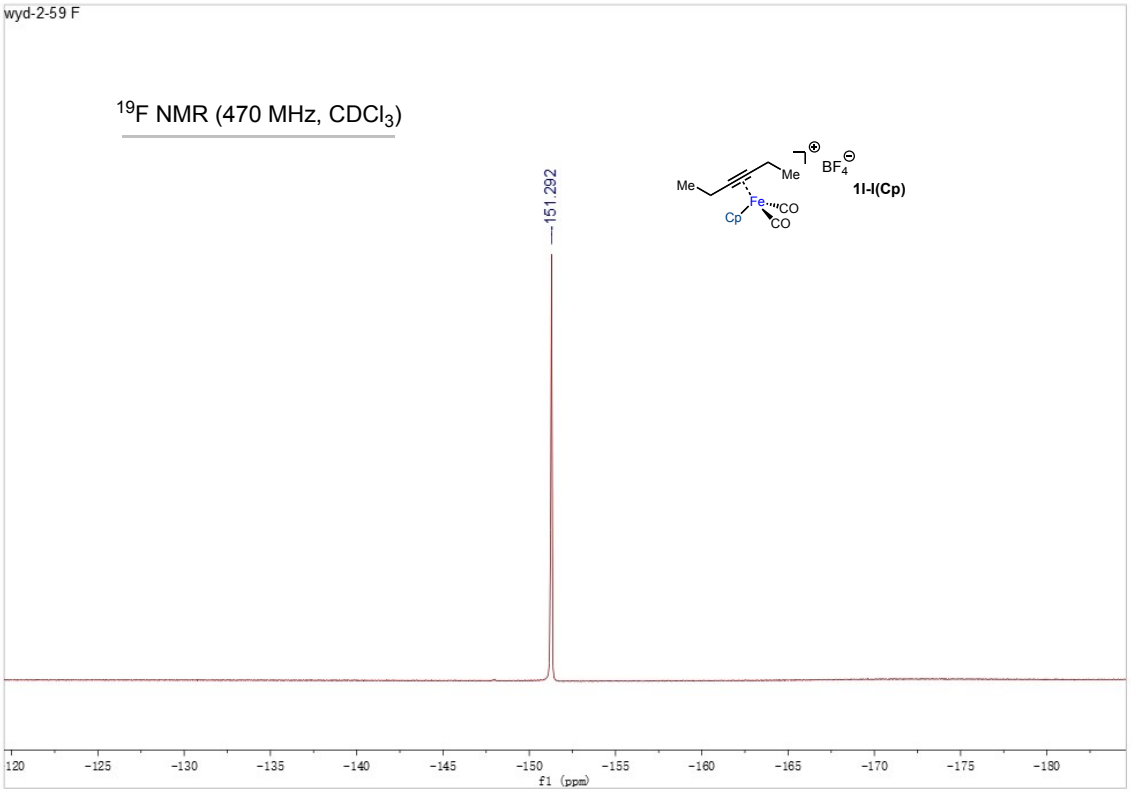
R(reflections)= 0.0366(3093) wR2(reflections)= 0.1116(3689)

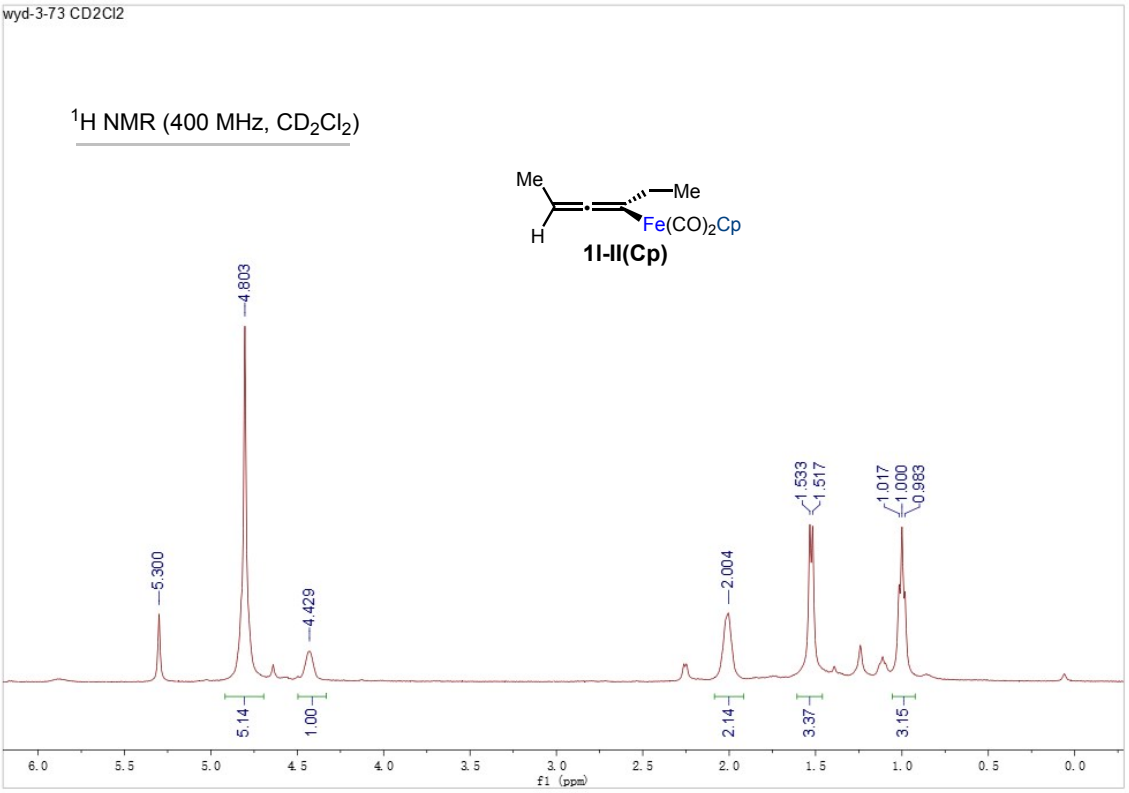
S = 1.093 Npar= 263

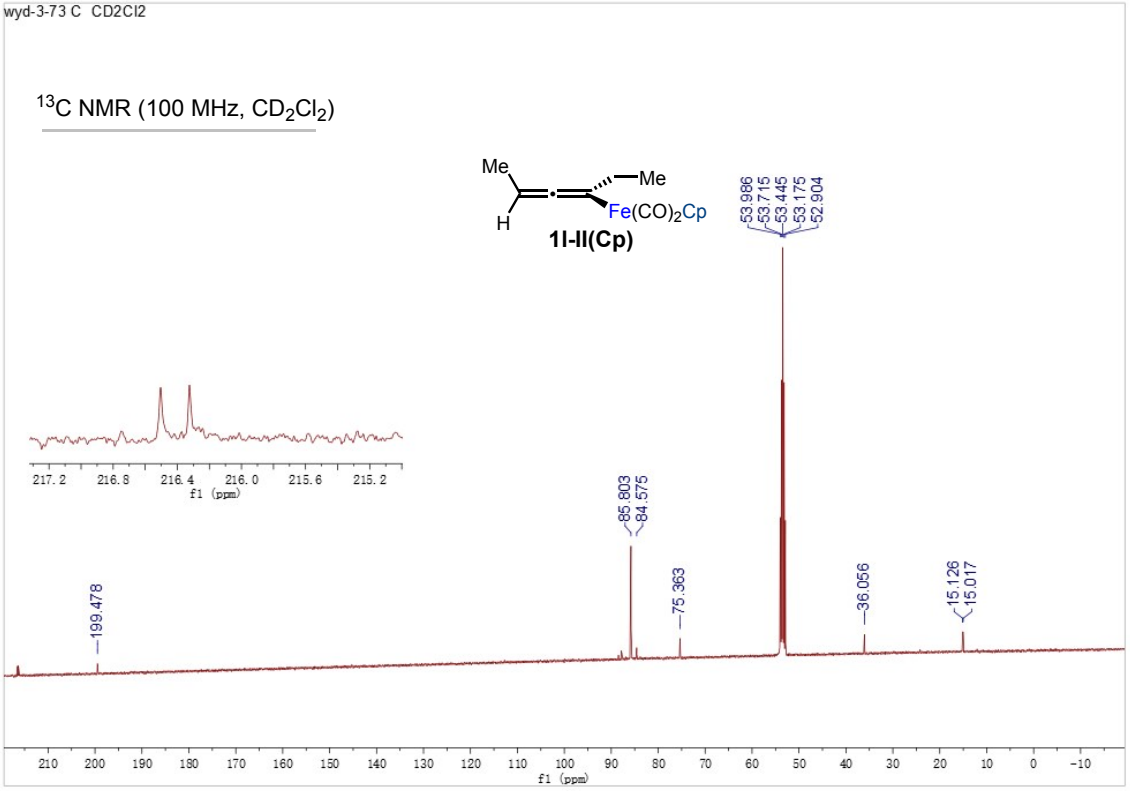
Copies of ^{1}H and ^{13}C NMR spectra for catalyst, products and substrates.

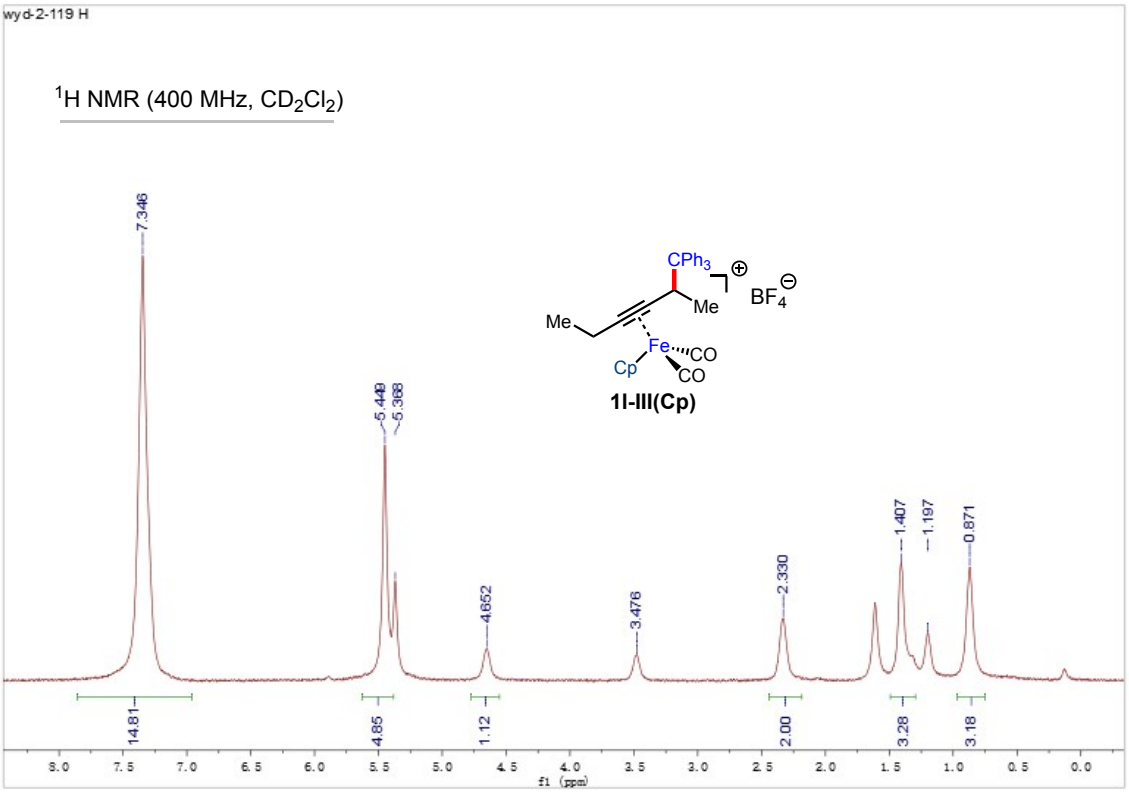





wyd-2-59 F
19F NMR (470 MHz, CDCl3)
151.292
Me
Me
BF4
Fe
Cp
CO
CO
1l-I(Cp)
120
-125
-130
-135
-140
-145
-150
-155
-160
-165
-170
-175
-180
f1 (ppm)

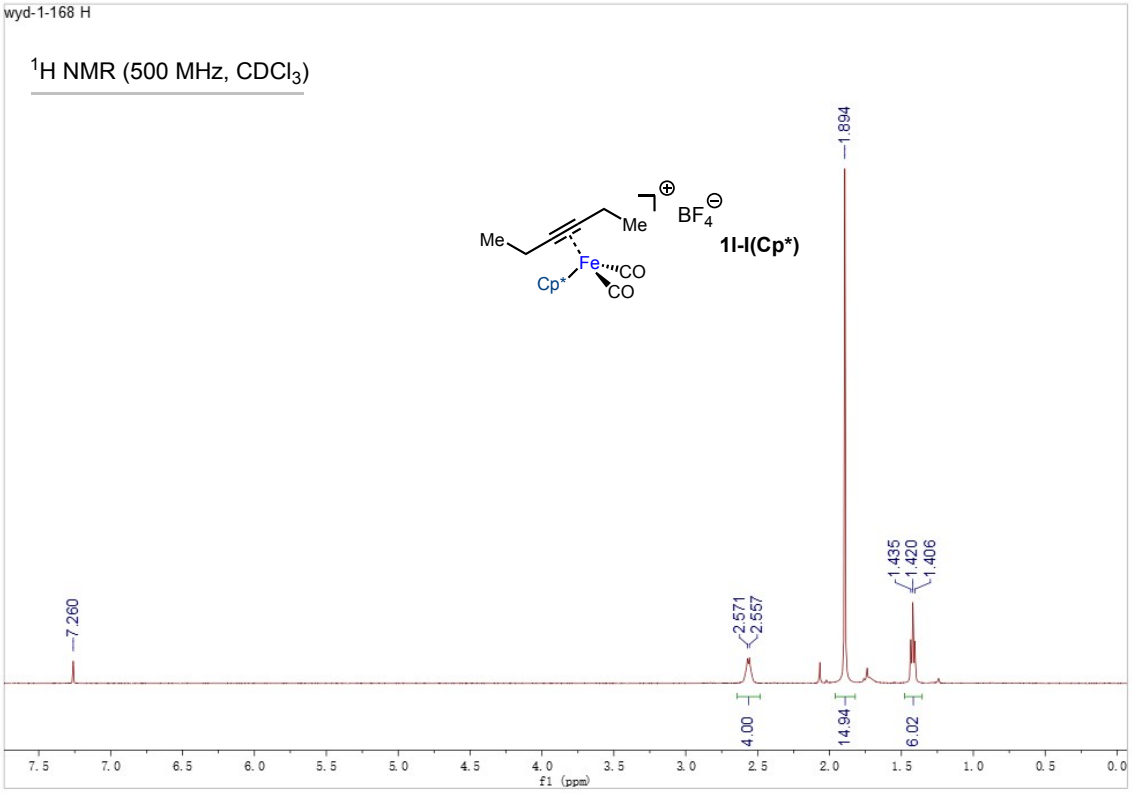

wyd-3-73 CD2Cl2
^{1}H NMR (400 MHz, CD_2Cl_2)
Me
Me
H
Fe(CO)$_2$Cp
1l-II(Cp)
4.803
5.300
4.429
2.004
1.533
1.517
1.017
1.000
0.983
5.14
1.00
2.14
3.37
3.15
f1 (ppm)


wyd-3-73 C CD2Cl2
^{13}C NMR (100 MHz, CD_2Cl_2)
Me
Me
H
Fe(CO)$_2$Cp
1l-II(Cp)
53.986
53.715
53.445
53.175
52.904
85.803
84.575
75.363
36.056
15.126
15.017
199.478
f1 (ppm)


wyd-2-119 H
1H NMR (400 MHz, CD2Cl2)
7.346
5.449
5.368
4.652
3.476
2.330
1.407
1.197
0.871
14.81
4.85
1.12
2.00
3.28
3.18
f1 (ppm)
CPh3
Me
BF4
Me
Fe
CO
CO
Cp
1l-lll(Cp)

wyd-1-168 H

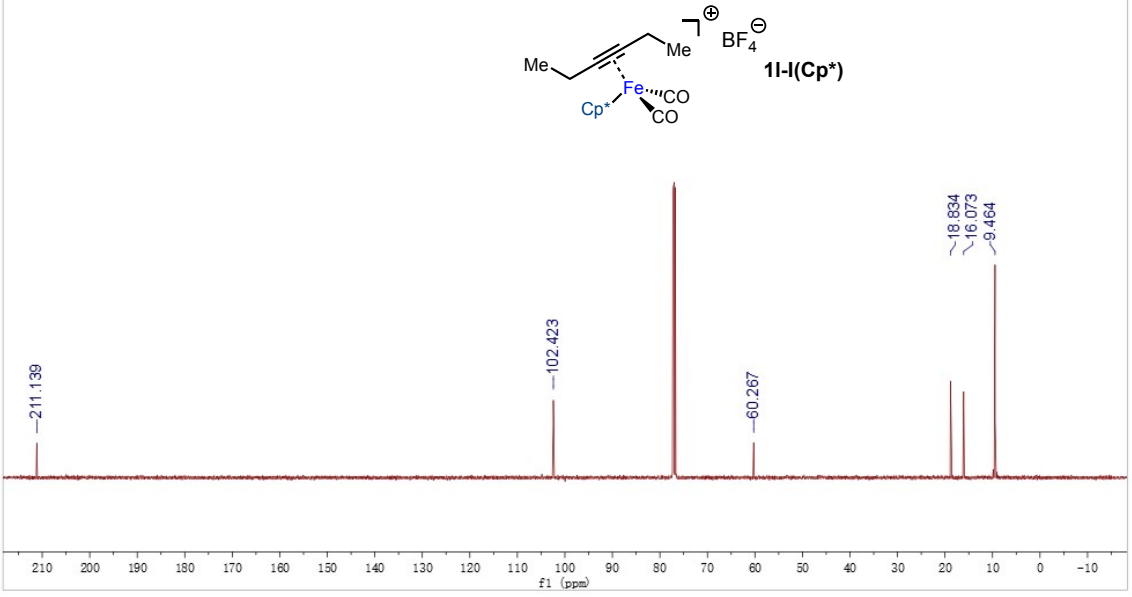
^{1}H NMR (500 MHz, $CDCl_3$)



1l-l(Cp*)

wyd-1-168 C

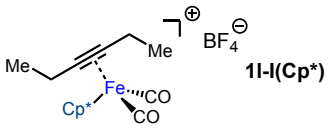
^{13}C NMR (125 MHz, $CDCl_3$)



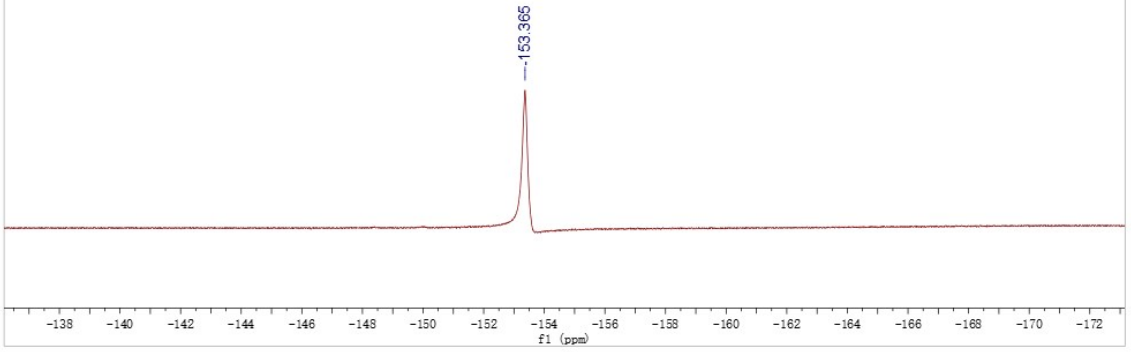
1l-l(Cp*)

wyd-1-168 F

^{19}F NMR (470 MHz, $CDCl_3$)

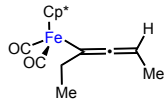


1l-I(Cp*)

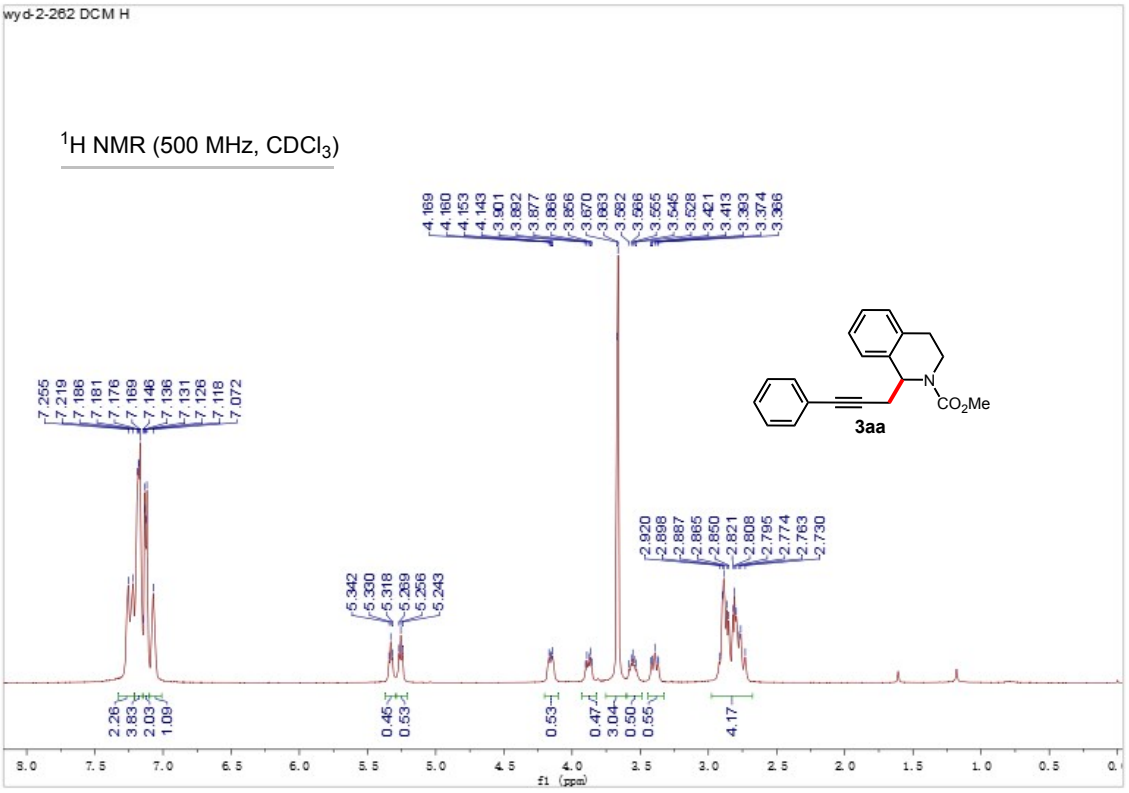


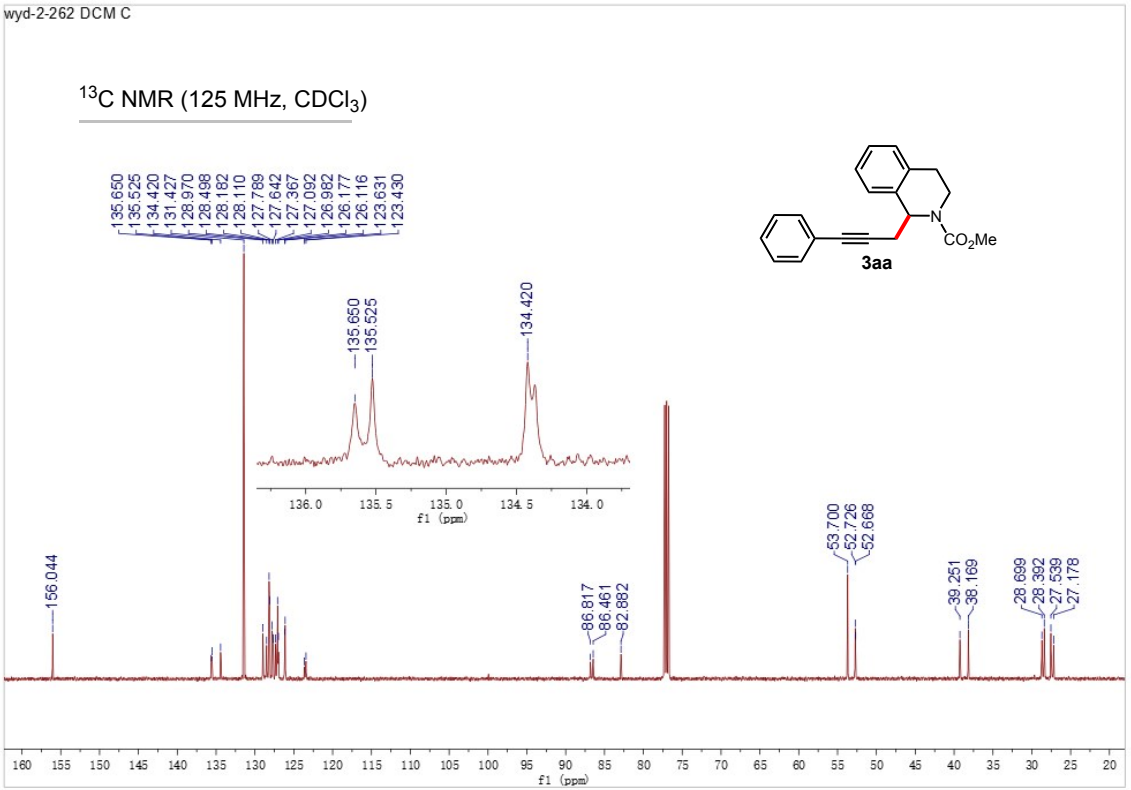
wyd-3-89-c H

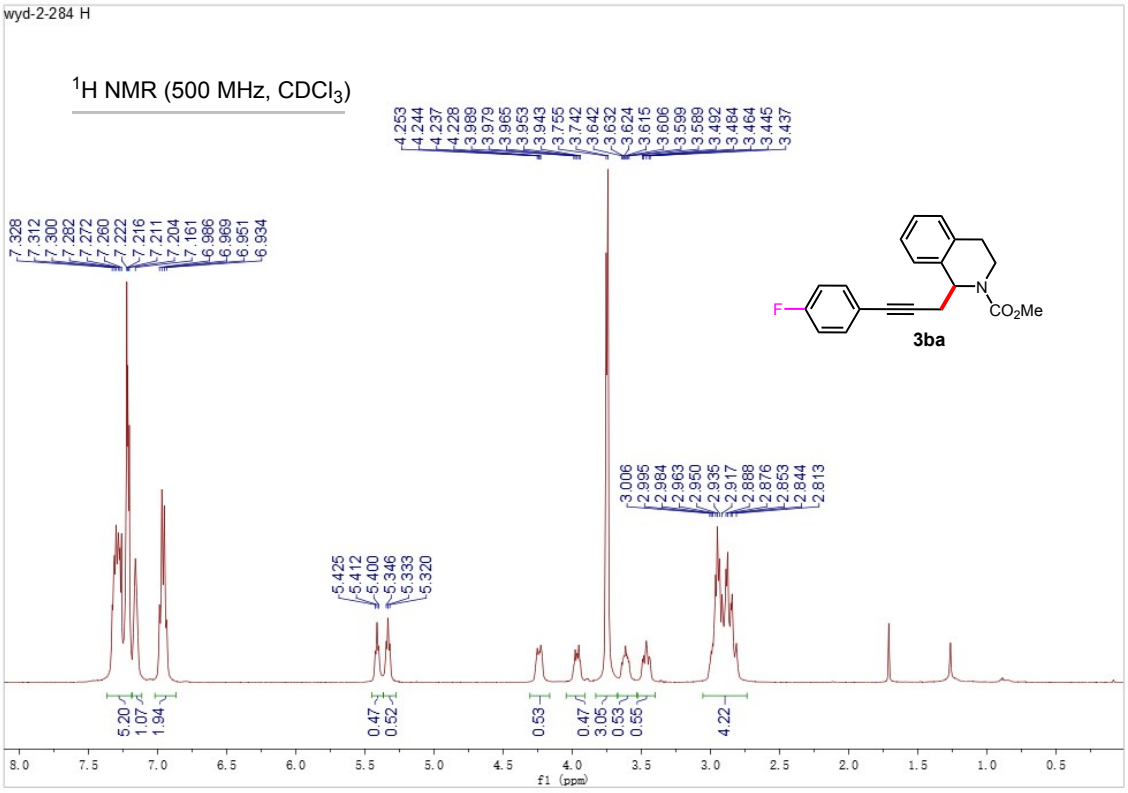
^{1}H NMR (400 MHz, CD_2Cl_2)

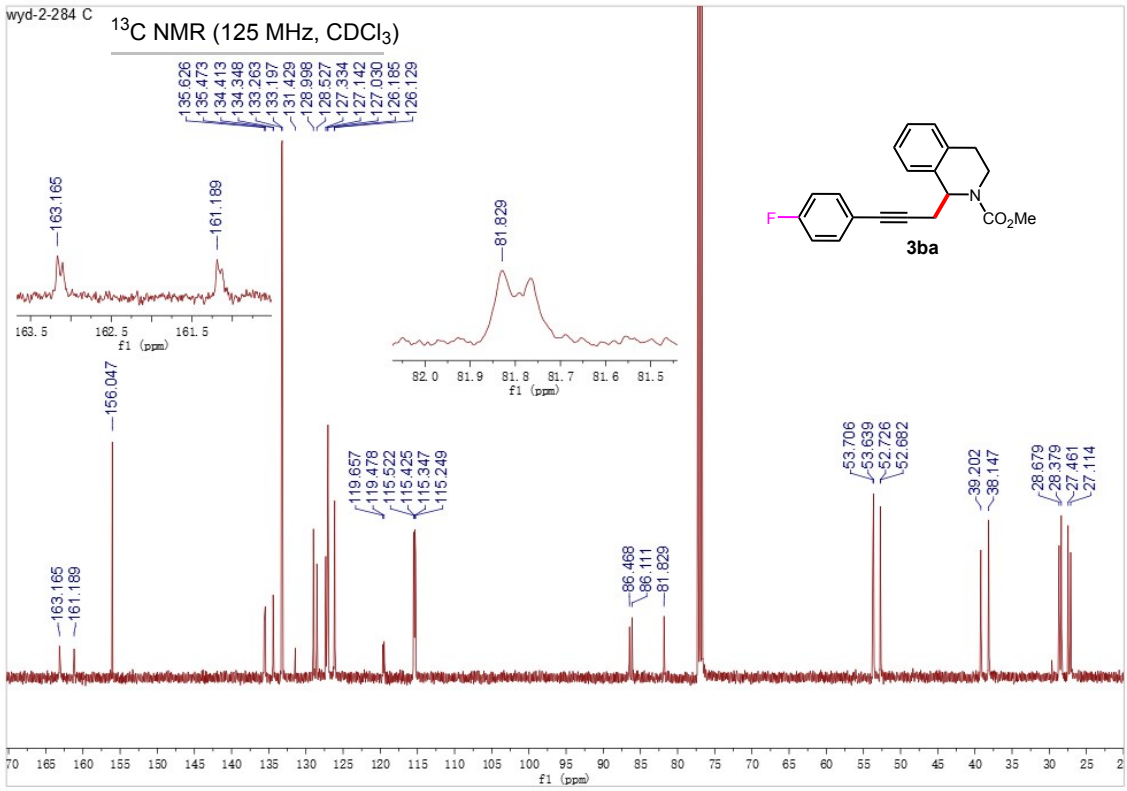


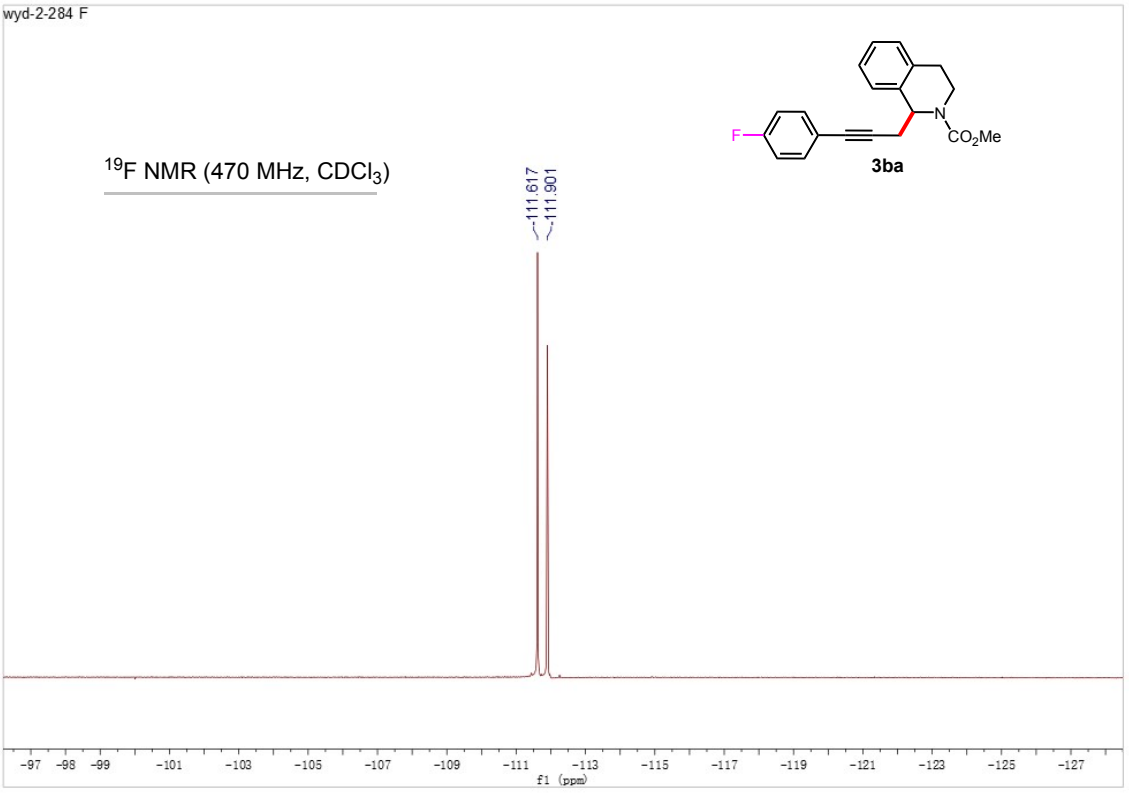
1l-II(Cp*)

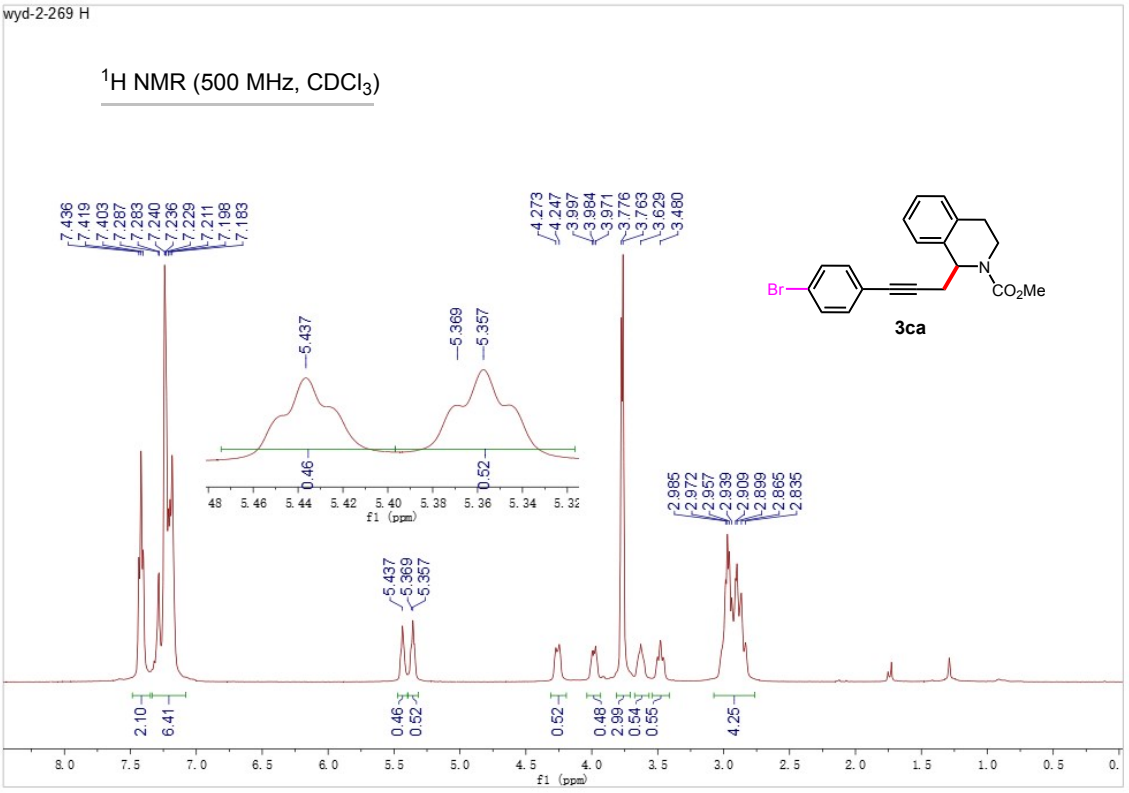

wyd-2-262 DCM H
^{1}H NMR (500 MHz, $CDCl_3$)
4.189
4.180
4.153
4.143
3.901
3.892
3.877
3.866
3.856
3.670
3.663
3.582
3.566
3.555
3.545
3.528
3.421
3.413
3.393
3.374
3.366
7.255
7.219
7.186
7.181
7.176
7.169
7.146
7.136
7.131
7.126
7.118
7.072
5.342
5.330
5.318
5.269
5.256
5.243
2.920
2.898
2.887
2.865
2.850
2.821
2.808
2.795
2.774
2.763
2.730
2.26
3.83
2.03
1.09
0.45
0.53
0.53
0.47
3.04
0.50
0.55
4.17
f1 (ppm)
N
CO_2Me
3aa

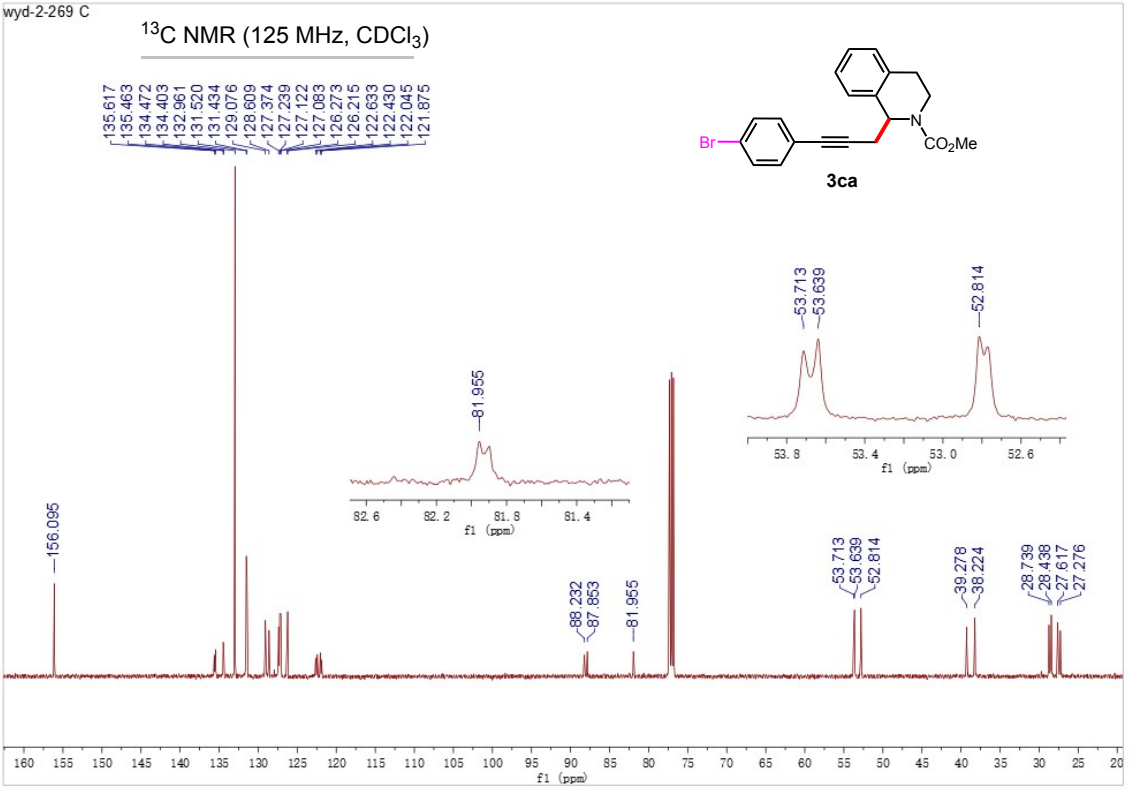

wyd-2-262 DCM C
^{13}C NMR (125 MHz, $CDCl_3$)
135.650
135.525
134.420
131.427
128.970
128.498
128.182
128.110
127.789
127.642
127.367
127.092
126.982
126.177
126.116
123.631
123.430
135.650
135.525
134.420
f1 (ppm)
156.044
86.817
86.461
82.882
53.700
52.726
52.668
39.251
38.169
28.699
28.392
27.539
27.178
f1 (ppm)
N
CO_2Me
3aa

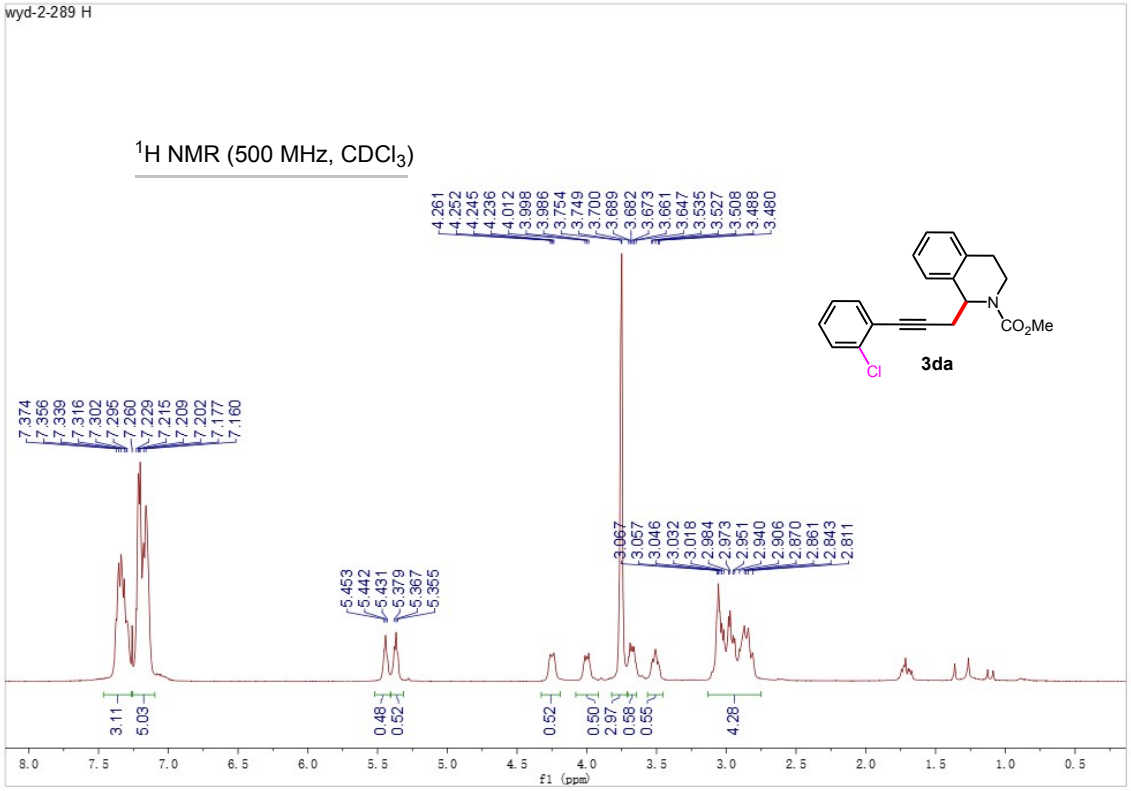

wyd-2-284 H
^{1}H NMR (500 MHz, $CDCl_3$)
7.328
7.312
7.300
7.282
7.272
7.260
7.222
7.216
7.211
7.204
7.161
6.986
6.969
6.951
6.934
4.253
4.244
4.237
4.228
3.989
3.979
3.965
3.953
3.943
3.755
3.742
3.642
3.632
3.624
3.615
3.606
3.599
3.589
3.492
3.484
3.464
3.445
3.437
5.425
5.412
5.400
5.346
5.333
5.320
3.006
2.995
2.984
2.963
2.950
2.935
2.917
2.888
2.876
2.853
2.844
2.813
5.20
1.07
1.94
0.47
0.52
0.53
0.47
3.05
0.53
0.55
4.22
F
N
CO_2Me
3ba
f1 (ppm)

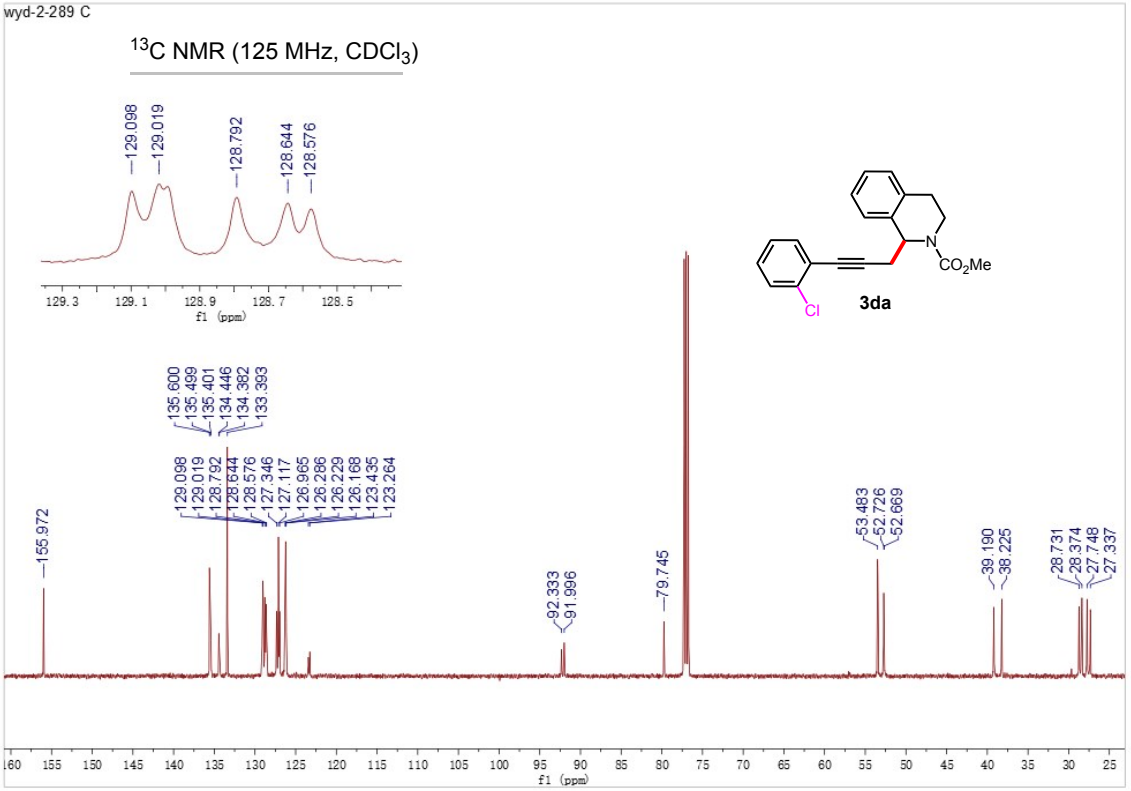

wyd-2-284 C
^{13}C NMR (125 MHz, $CDCl_3$)
135.626
135.473
134.413
134.348
133.263
133.197
131.429
128.998
128.527
127.334
127.142
127.030
126.185
126.129
163.165
161.189
81.829
156.047
119.657
119.478
115.522
115.425
115.347
115.249
86.468
86.111
81.829
53.706
53.639
52.726
52.682
39.202
38.147
28.679
28.379
27.461
27.114
F
N
CO_2Me
3ba
f1 (ppm)

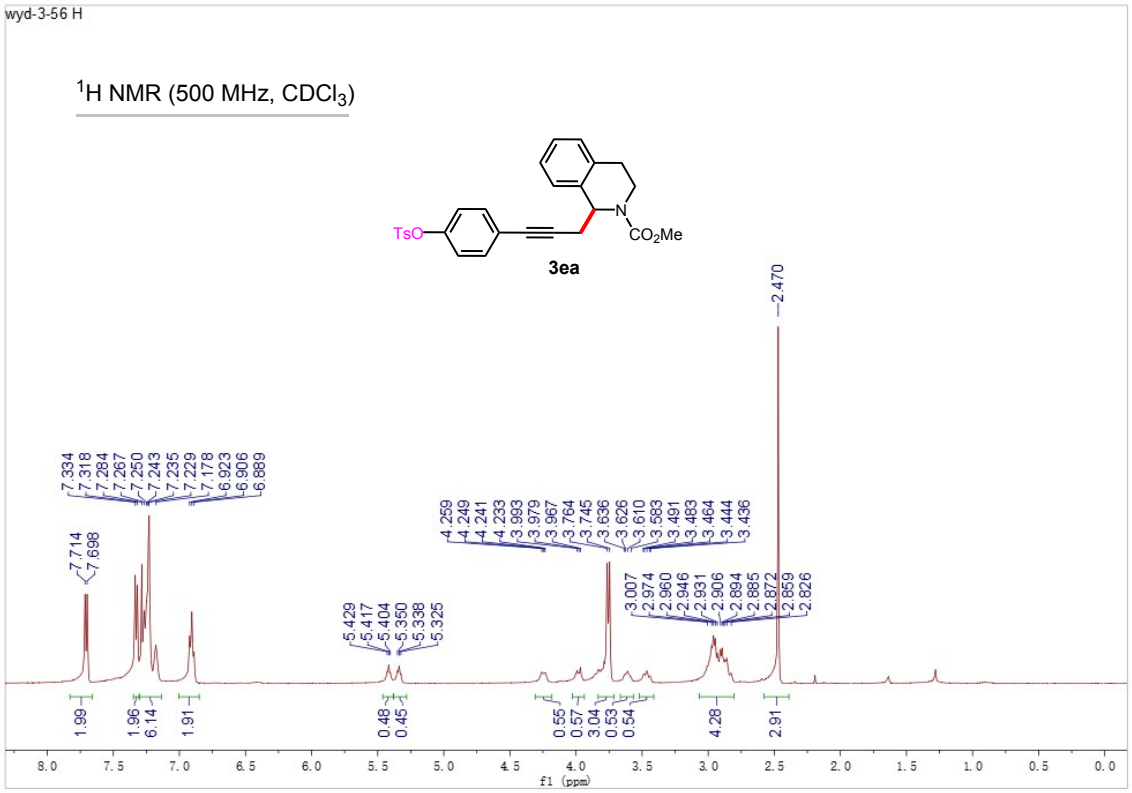

wyd-2-284 F
^{19}F NMR (470 MHz, $CDCl_3$)
-111.617
-111.901
F
N
CO_2Me
3ba
-97 -98 -99 -101 -103 -105 -107 -109 -111 -113 -115 -117 -119 -121 -123 -125 -127
f1 (ppm)

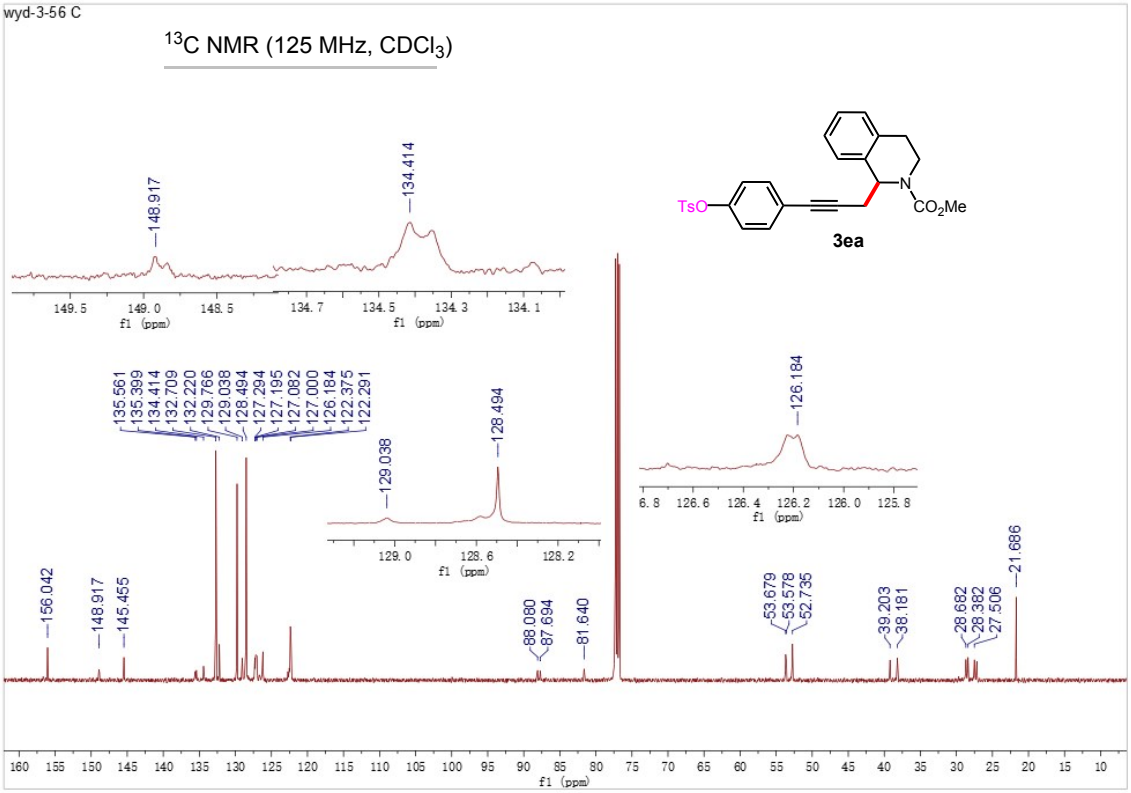

wyd-2-269 H
^{1}H NMR (500 MHz, CDCl$_3$)
3ca

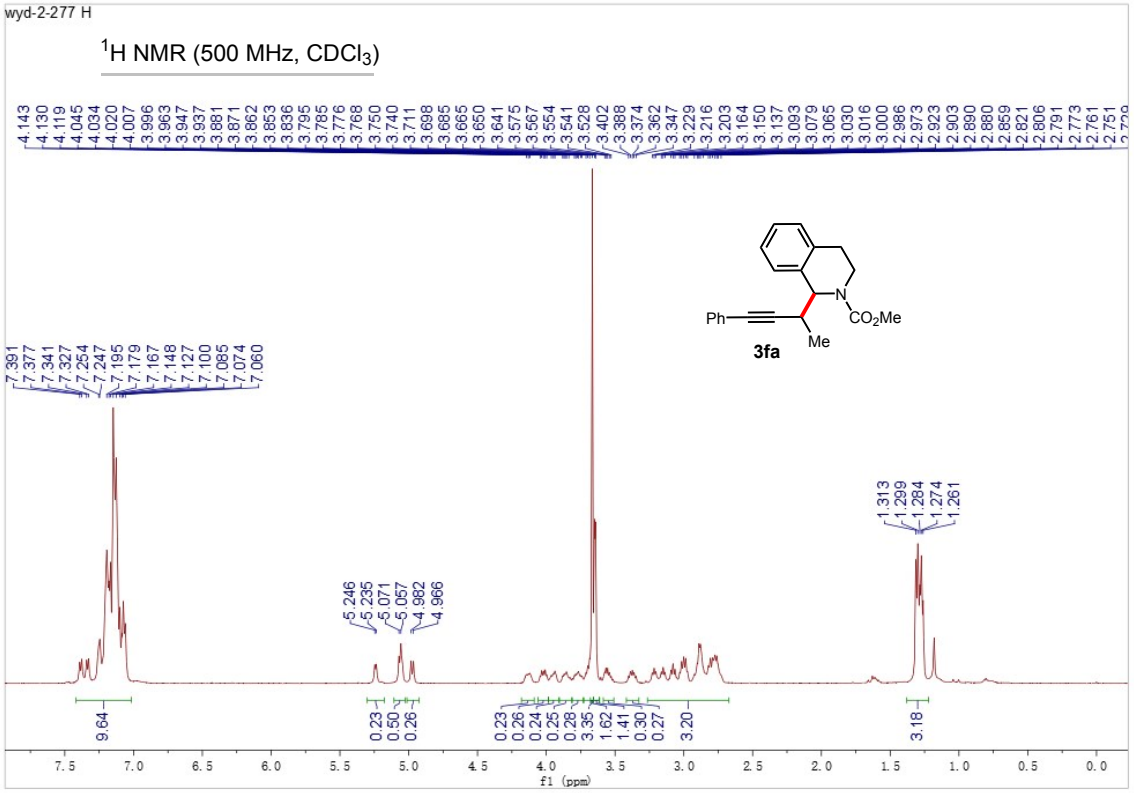

wyd-2-269 C
^{13}C NMR (125 MHz, CDCl$_3$)
3ca

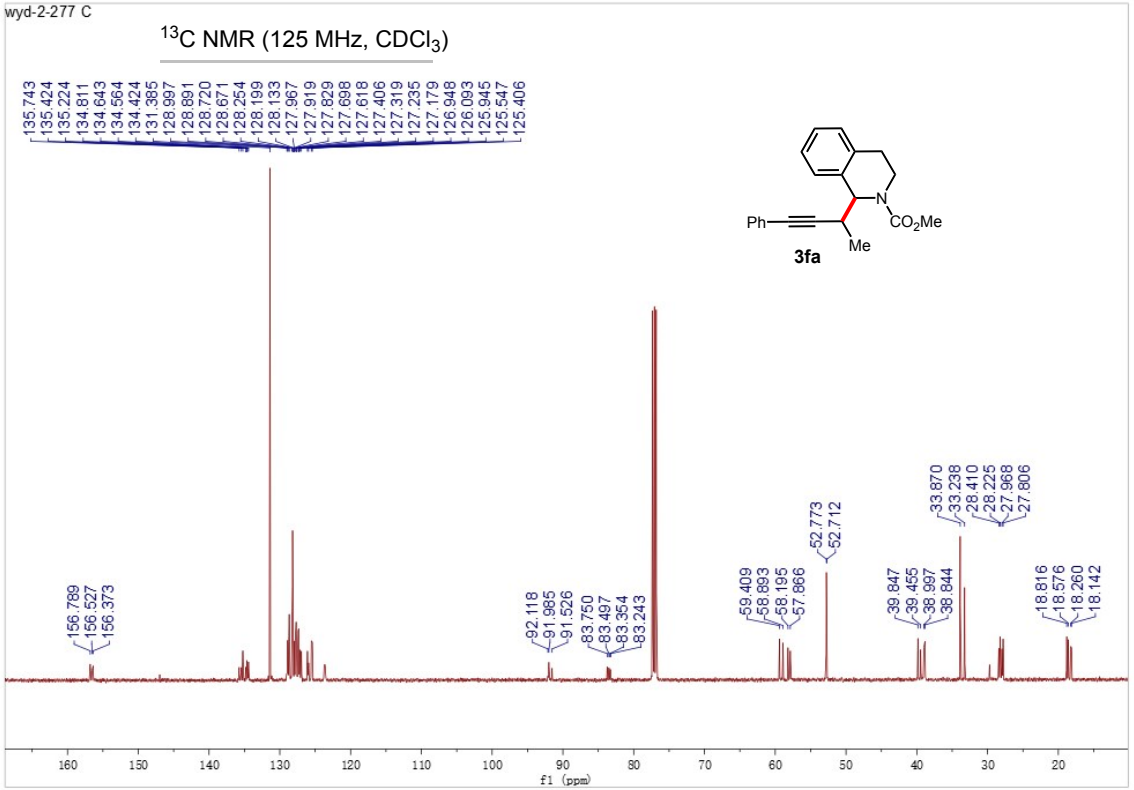

wyd-2-289 H
^{1}H NMR (500 MHz, $CDCl_3$)
4.261
4.252
4.245
4.236
4.012
3.998
3.986
3.754
3.749
3.700
3.689
3.682
3.673
3.661
3.647
3.535
3.527
3.508
3.488
3.480
7.374
7.356
7.339
7.316
7.302
7.295
7.260
7.229
7.215
7.209
7.202
7.177
7.160
5.453
5.442
5.431
5.379
5.367
5.355
3.067
3.057
3.046
3.032
3.018
2.984
2.973
2.951
2.940
2.906
2.870
2.861
2.843
2.811
3.11
5.03
0.48
0.52
0.52
0.50
2.97
0.58
0.55
4.28
f1 (ppm)
N
CO_2Me
Cl
3da

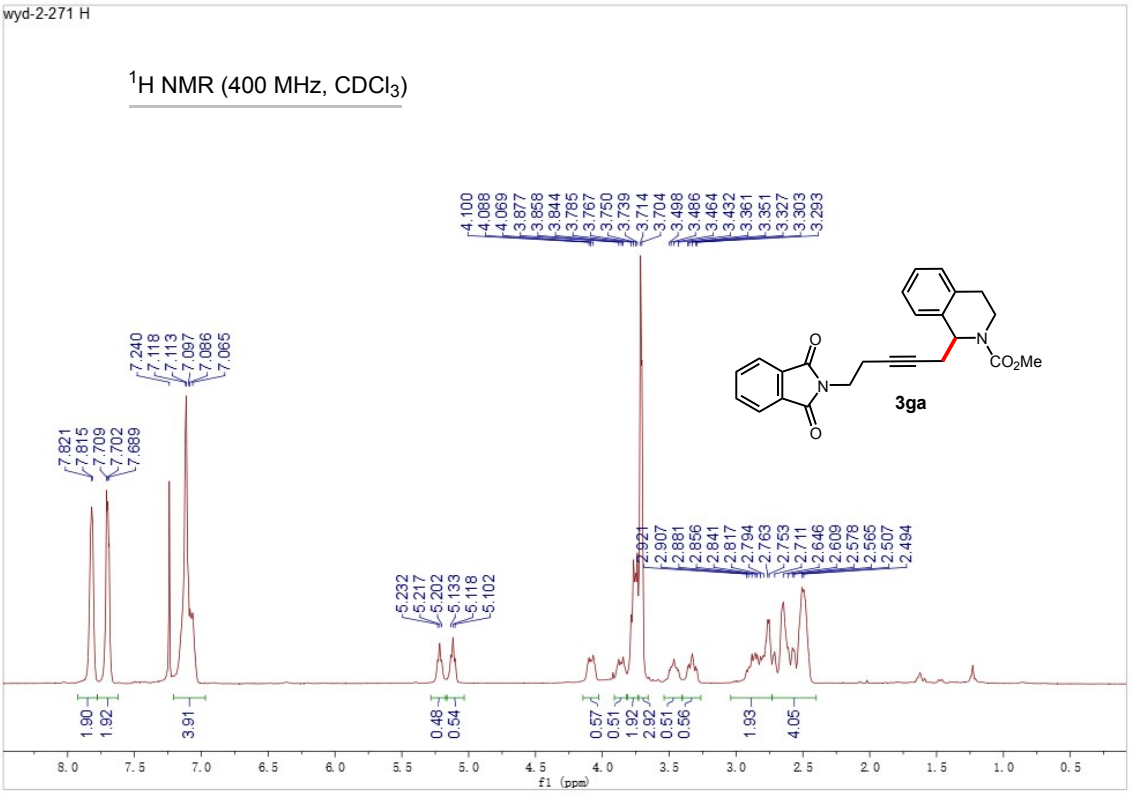

wyd-2-289 C
^{13}C NMR (125 MHz, $CDCl_3$)
129.098
129.019
128.792
128.644
128.576
f1 (ppm)
135.600
135.499
135.401
134.446
134.382
133.393
129.098
129.019
128.792
128.644
128.576
127.346
127.117
126.965
126.286
126.229
126.168
123.435
123.264
155.972
92.333
91.996
79.745
53.483
52.726
52.669
39.190
38.225
28.731
28.374
27.748
27.337
f1 (ppm)
N
CO_2Me
Cl
3da

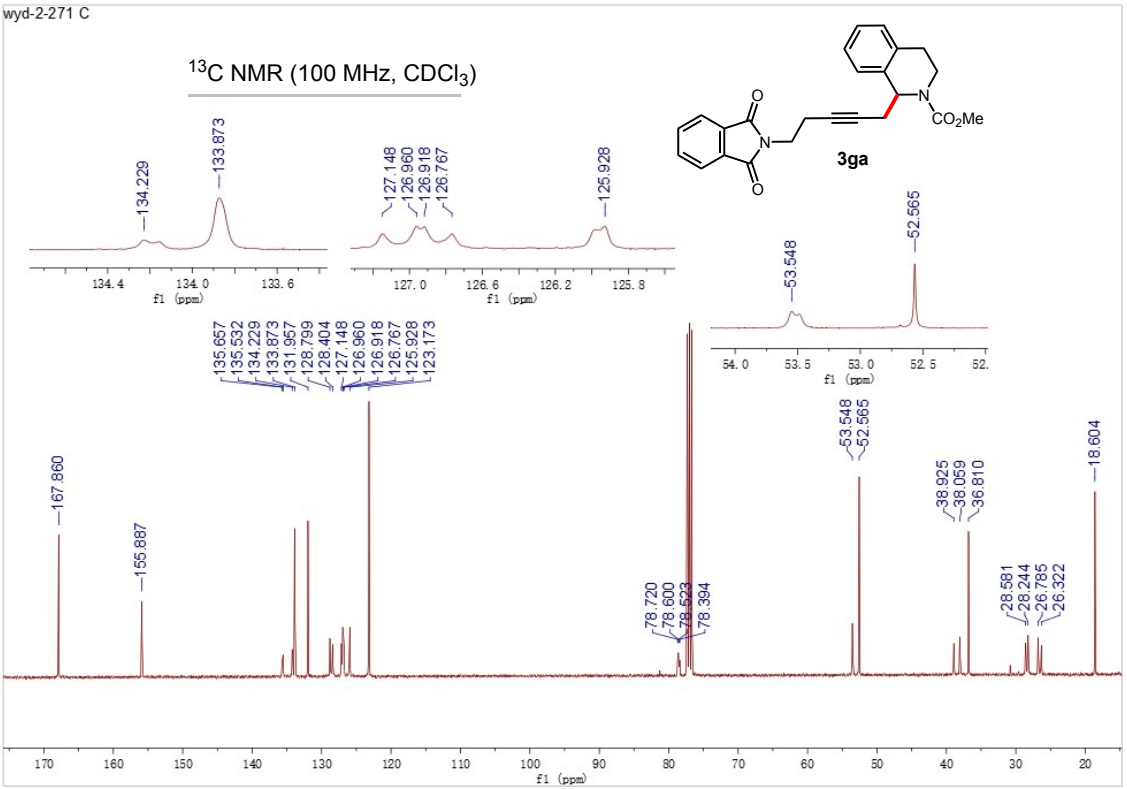

wyd-3-56 H
^{1}H NMR (500 MHz, $CDCl_3$)
TsO
N
CO_2Me
3ea
2.470
7.334
7.318
7.284
7.267
7.250
7.243
7.235
7.229
7.178
6.923
6.906
6.889
7.714
7.698
4.259
4.249
4.241
4.233
3.993
3.979
3.967
3.764
3.745
3.636
3.626
3.610
3.583
3.491
3.483
3.464
3.444
3.436
5.429
5.417
5.404
5.350
5.338
5.325
3.007
2.974
2.960
2.946
2.931
2.906
2.894
2.885
2.872
2.859
2.826
1.99
1.96
6.14
1.91
0.48
0.45
0.55
0.57
3.04
0.53
0.54
4.28
2.91
f1 (ppm)


wyd-3-56 C
^{13}C NMR (125 MHz, $CDCl_3$)
TsO
N
CO_2Me
3ea
148.917
134.414
135.561
135.399
134.414
132.709
132.220
129.766
129.038
128.494
127.294
127.195
127.082
127.000
126.184
122.375
122.291
129.038
128.494
126.184
156.042
148.917
145.455
88.080
87.694
81.640
53.679
53.578
52.735
39.203
38.181
28.682
28.382
27.506
21.686
f1 (ppm)


wyd-2-277 H
^{1}H NMR (500 MHz, $CDCl_3$)
Ph
N
CO_2Me
Me
3fa
f1 (ppm)

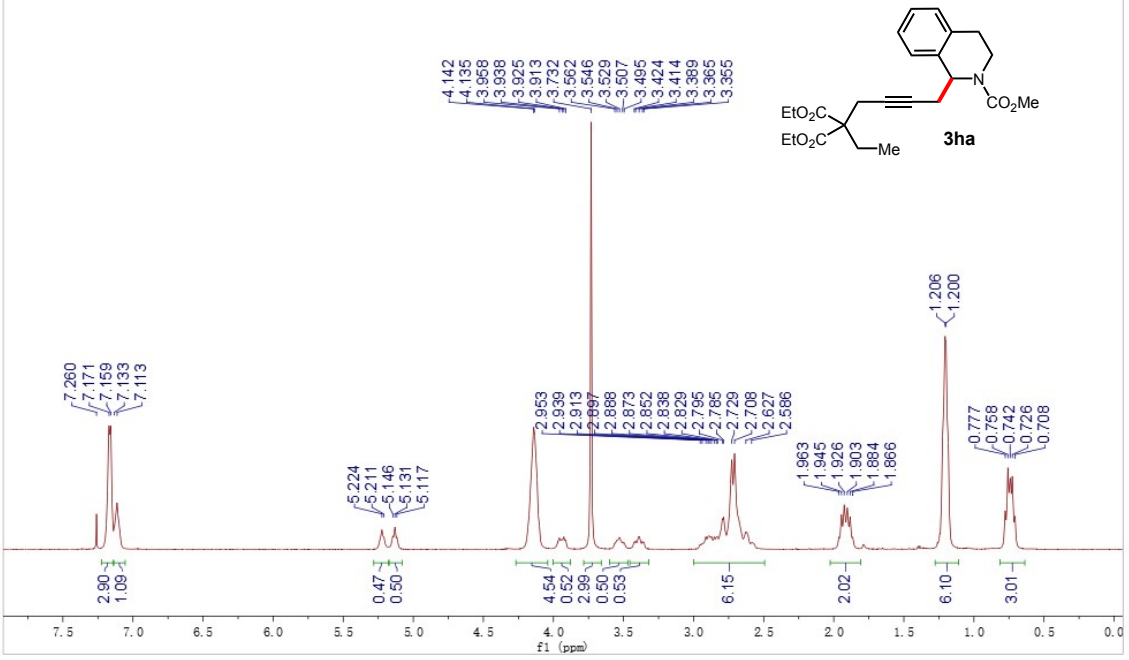

wyd-2-277 C
^{13}C NMR (125 MHz, $CDCl_3$)
Ph
N
CO_2Me
Me
3fa
f1 (ppm)


wyd-2-271 H
^{1}H NMR (400 MHz, $CDCl_3$)
3ga
f1 (ppm)


wyd-2-271 C
^{13}C NMR (100 MHz, $CDCl_3$)
3ga
f1 (ppm)

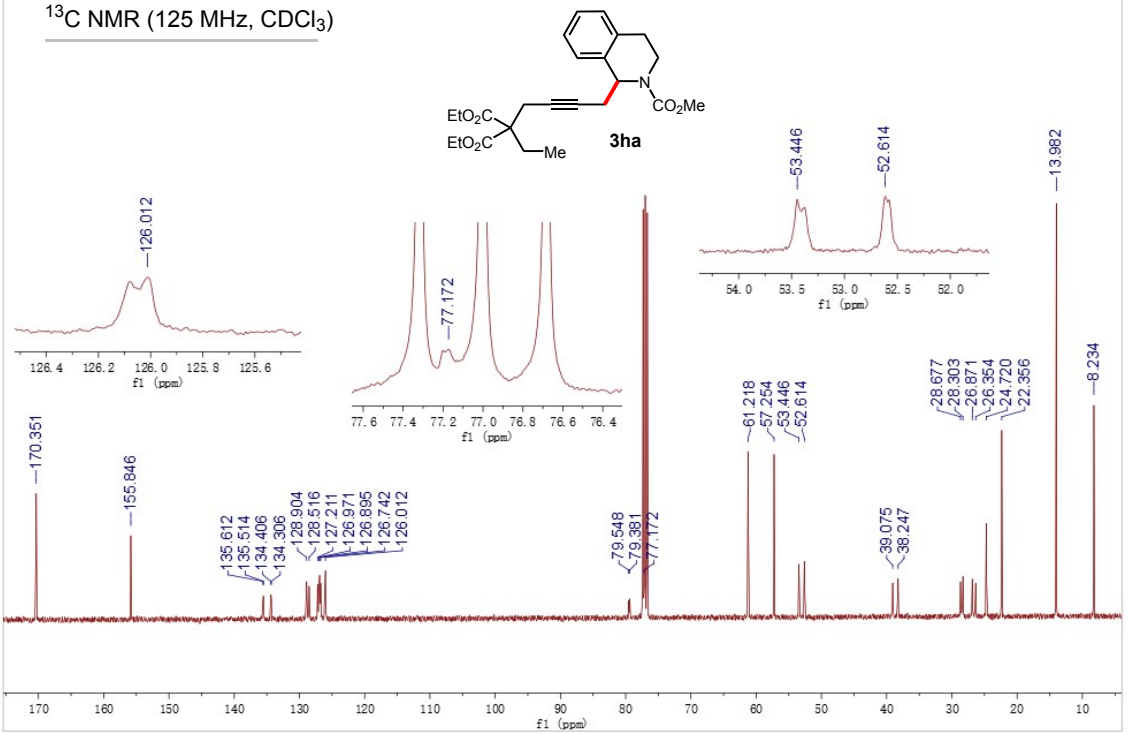
wyd-2-285 H

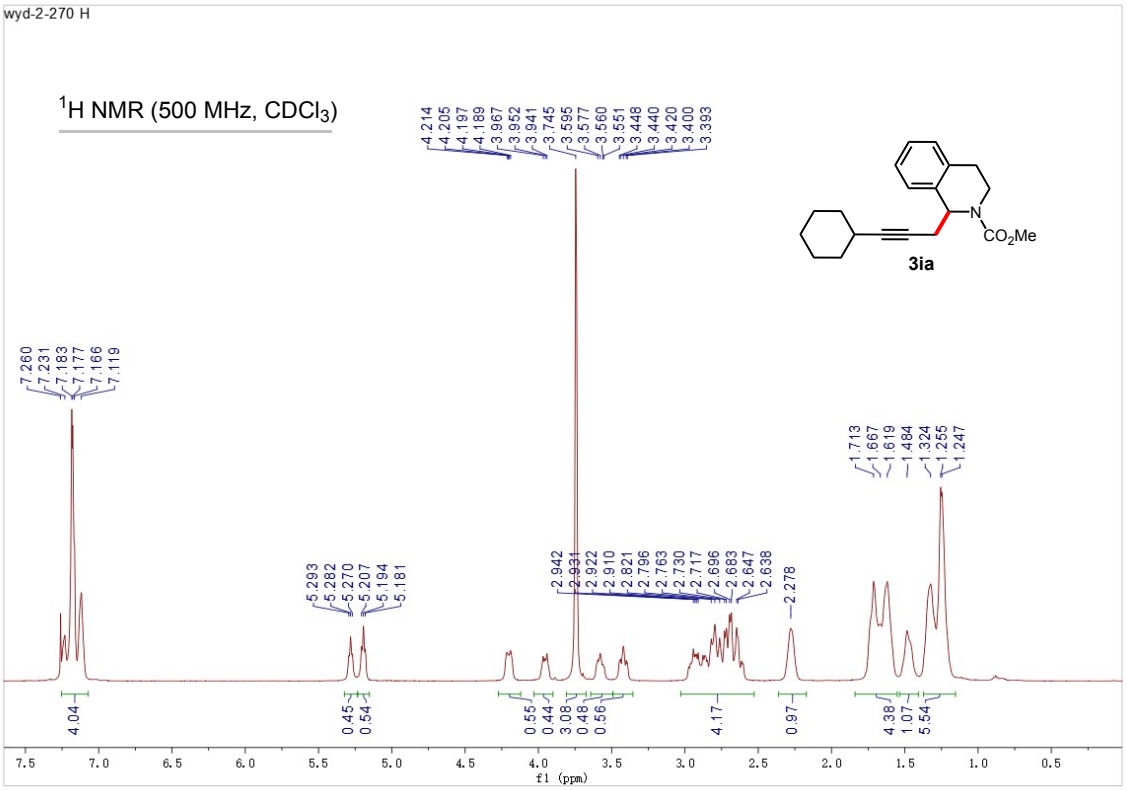
^{1}H NMR (500 MHz, $CDCl_3$)

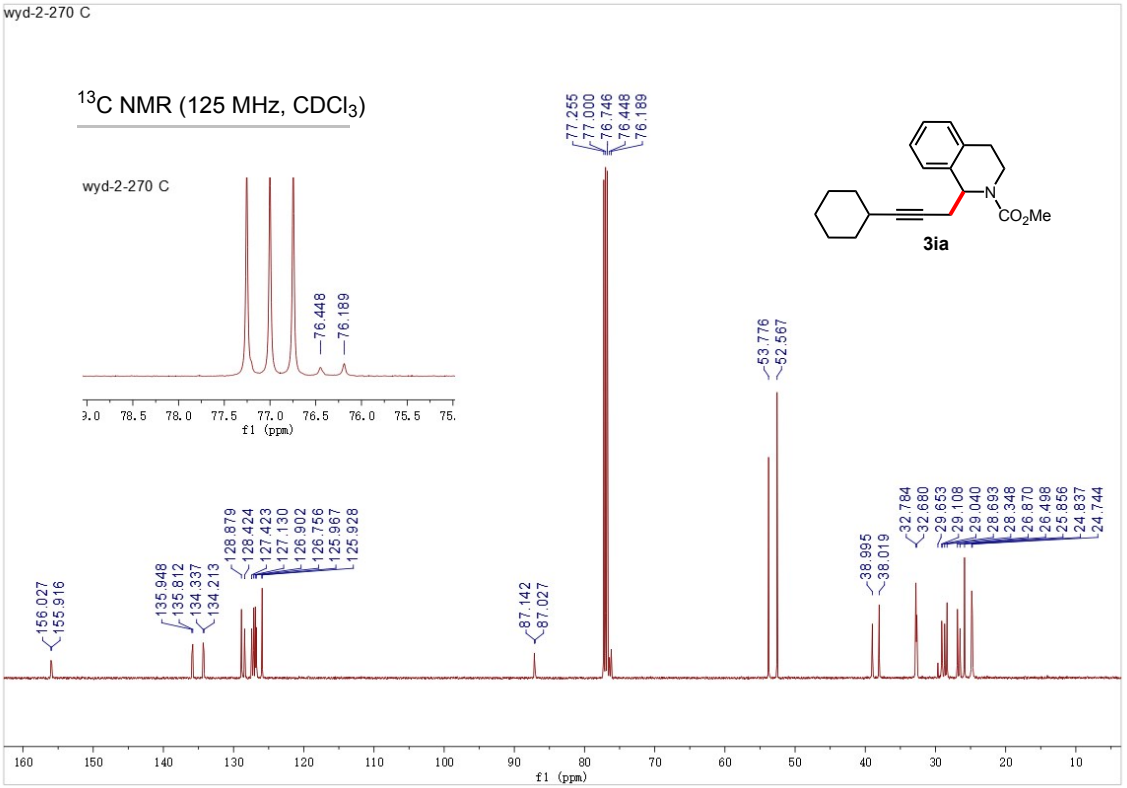


wyd-2-285 C

^{13}C NMR (125 MHz, $CDCl_3$)

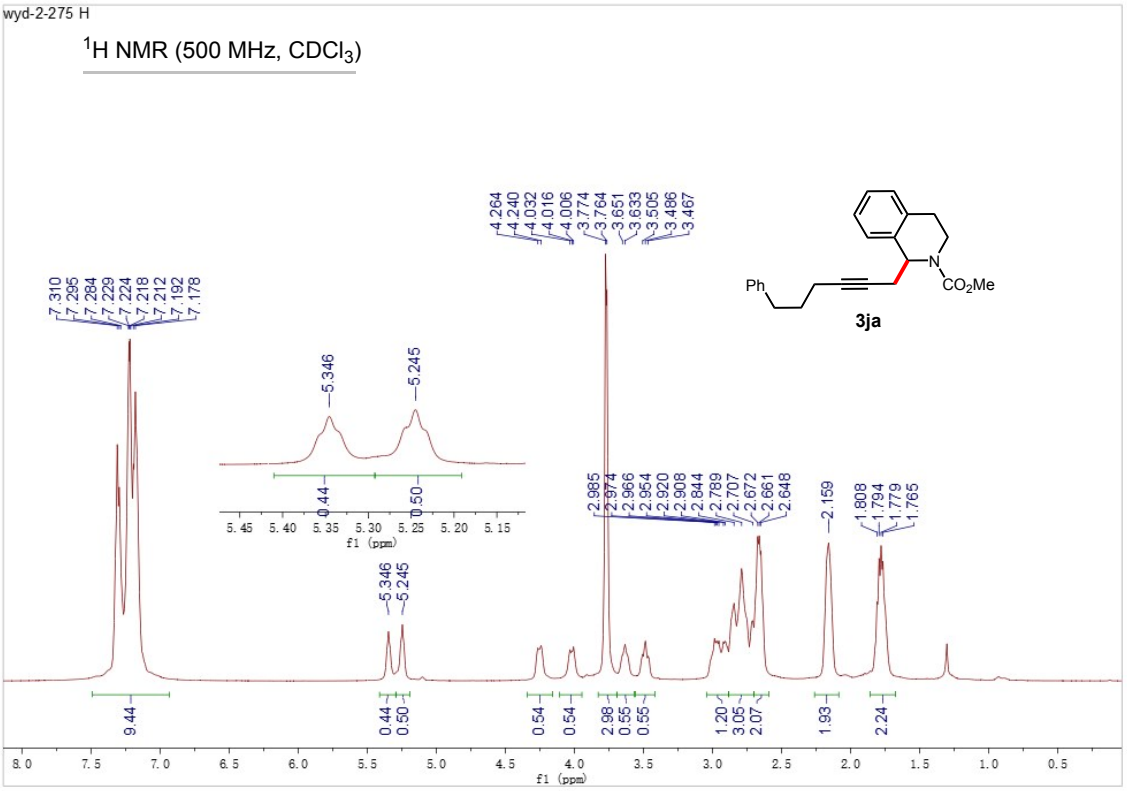



wyd-2-270 H
^{1}H NMR (500 MHz, $CDCl_3$)
3ia
f1 (ppm)


wyd-2-270 C
^{13}C NMR (125 MHz, $CDCl_3$)
wyd-2-270 C
3ia
f1 (ppm)

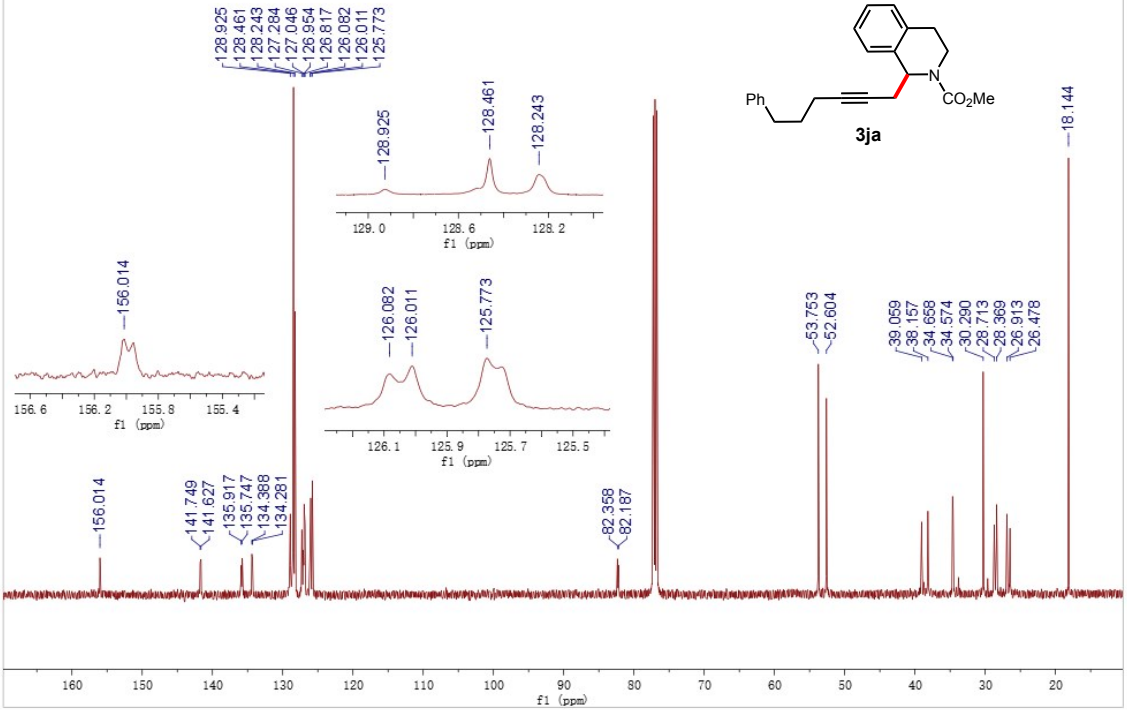
wyd-2-275 H

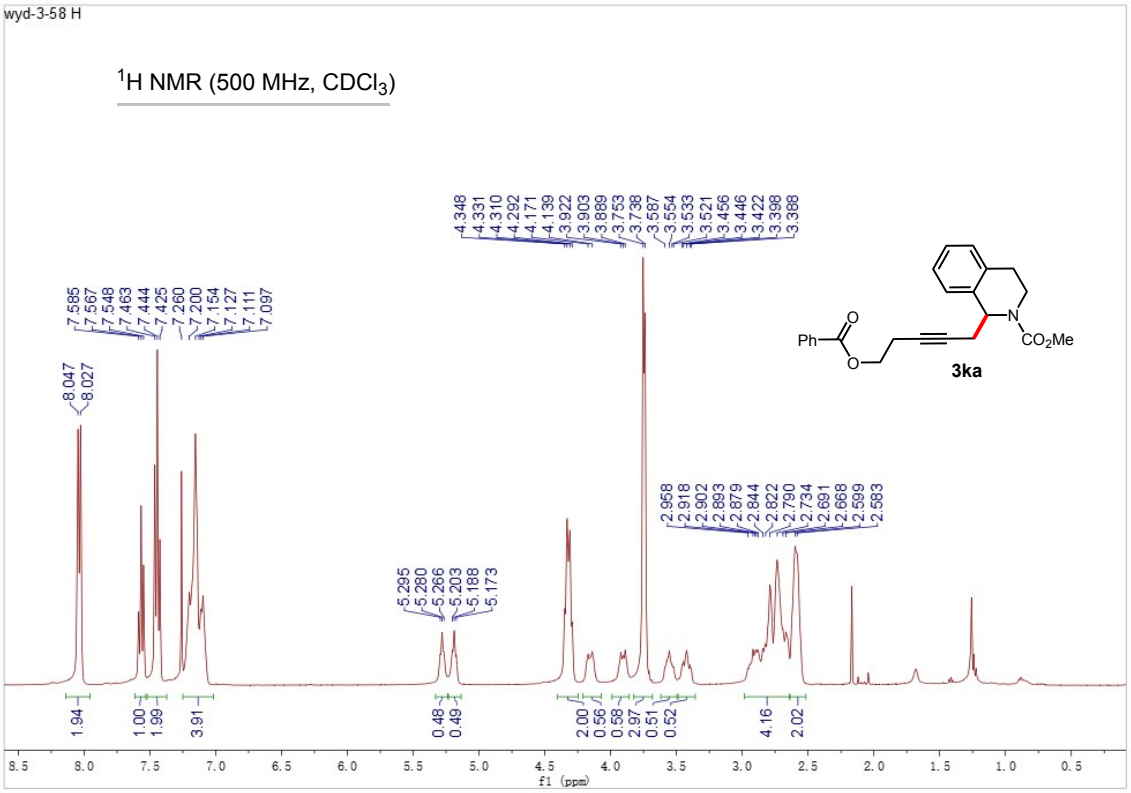
^{1}H NMR (500 MHz, $CDCl_3$)

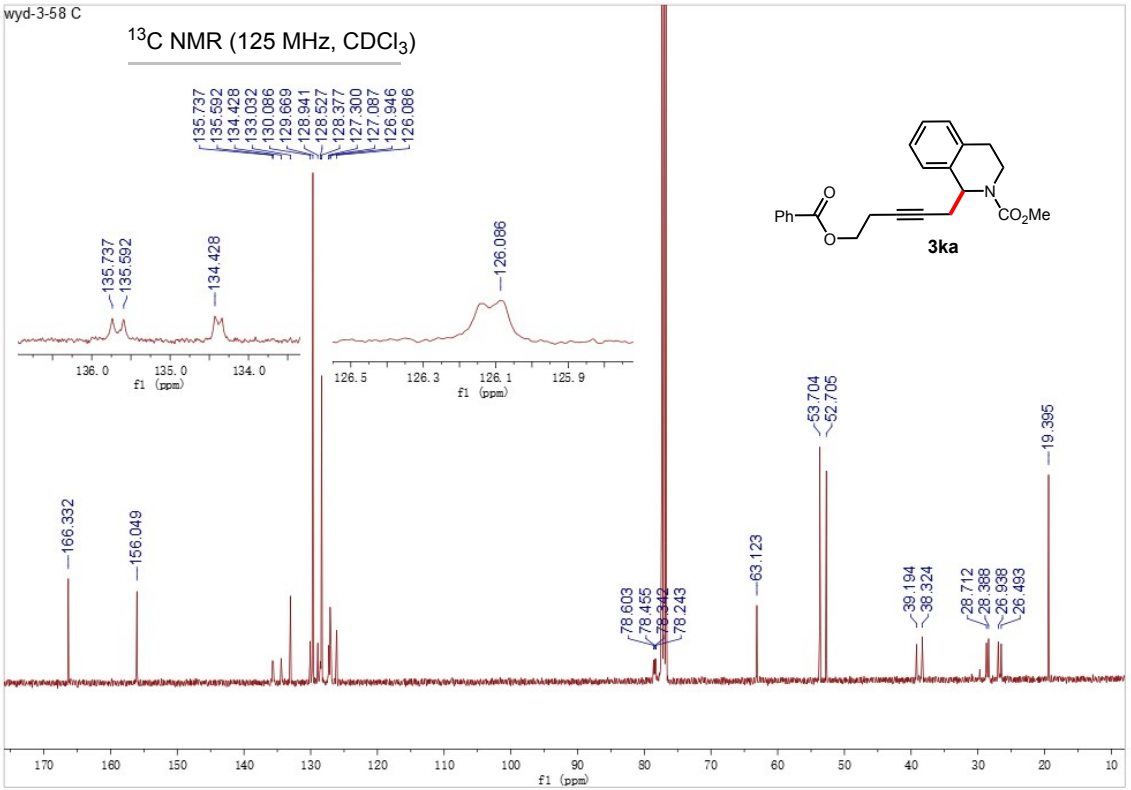


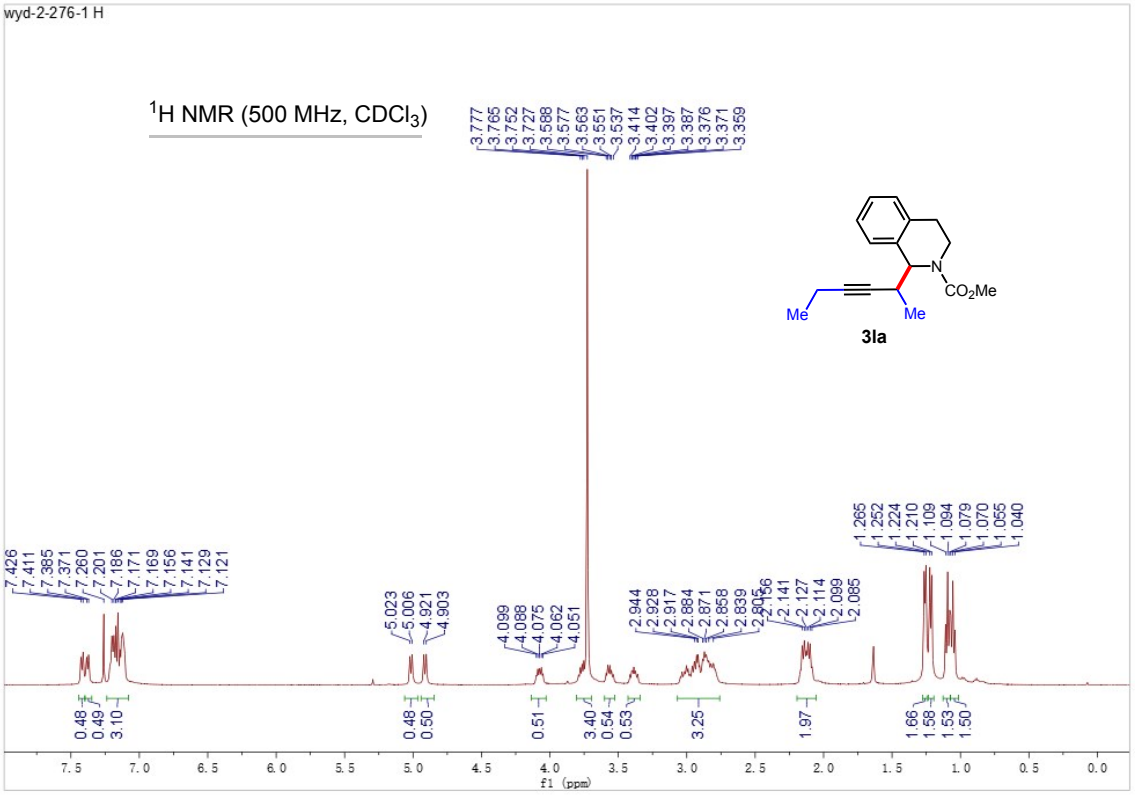
wyd-2-275 C

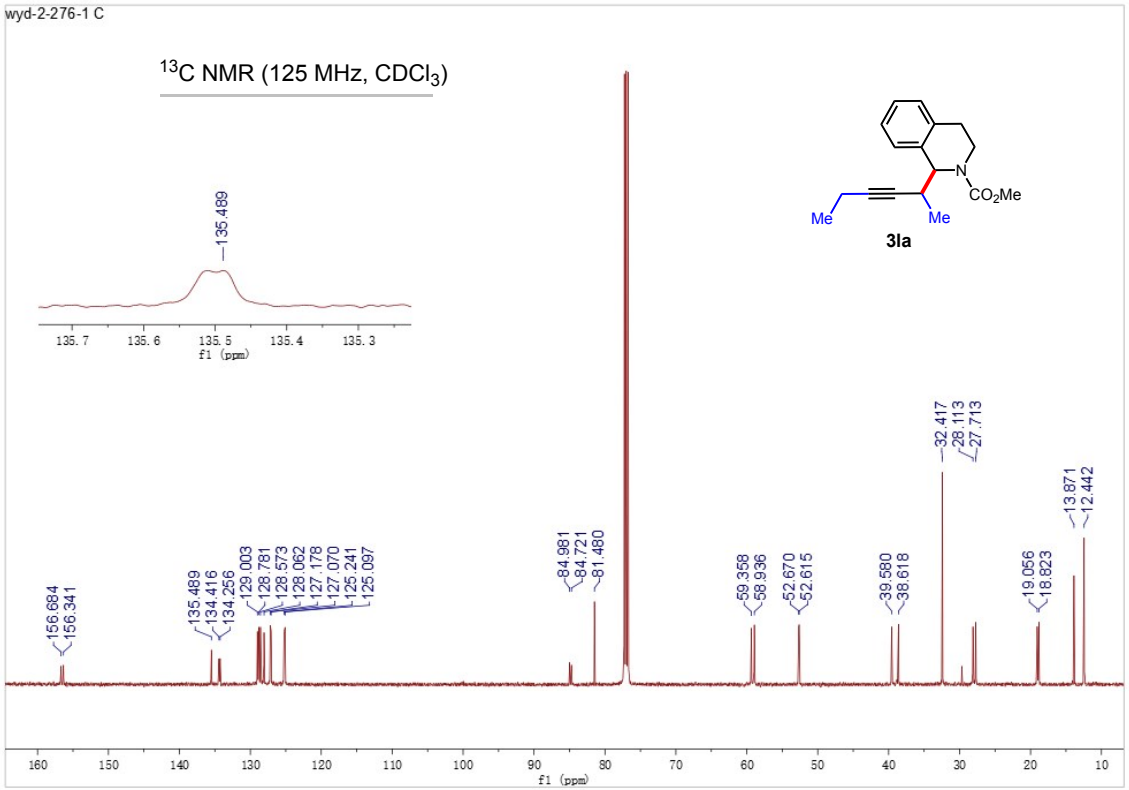
^{13}C NMR (125 MHz, $CDCl_3$)

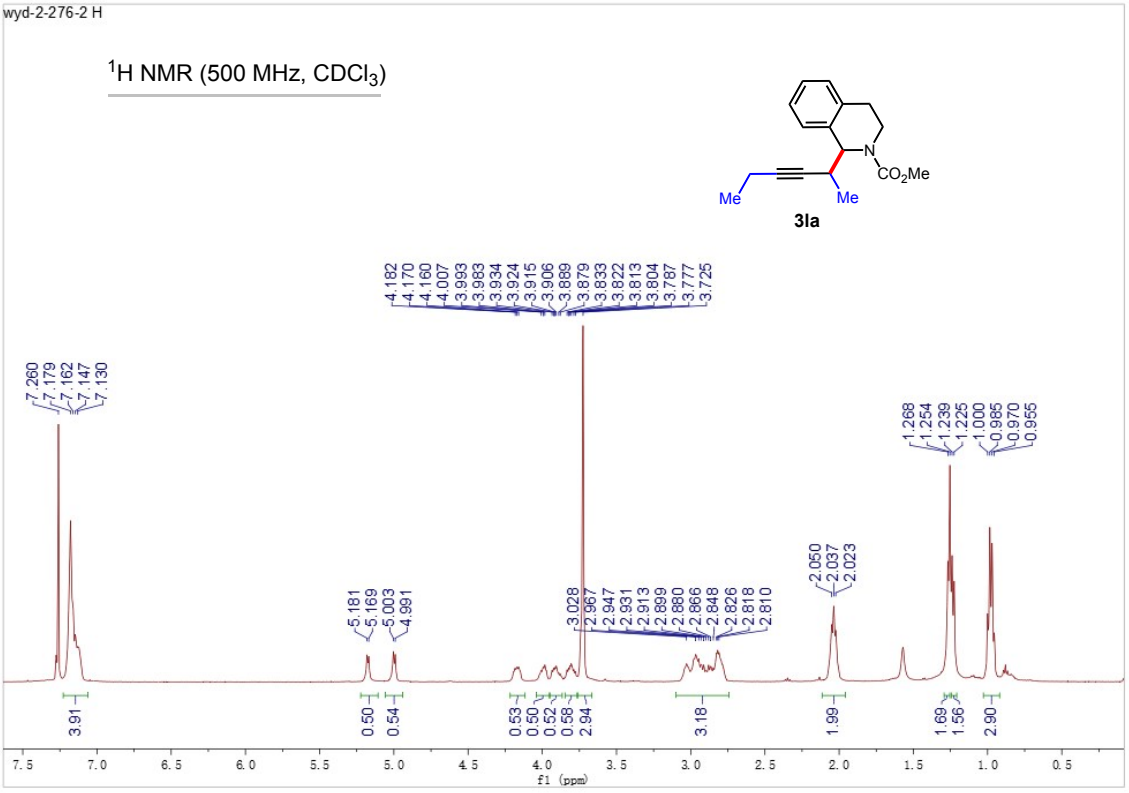


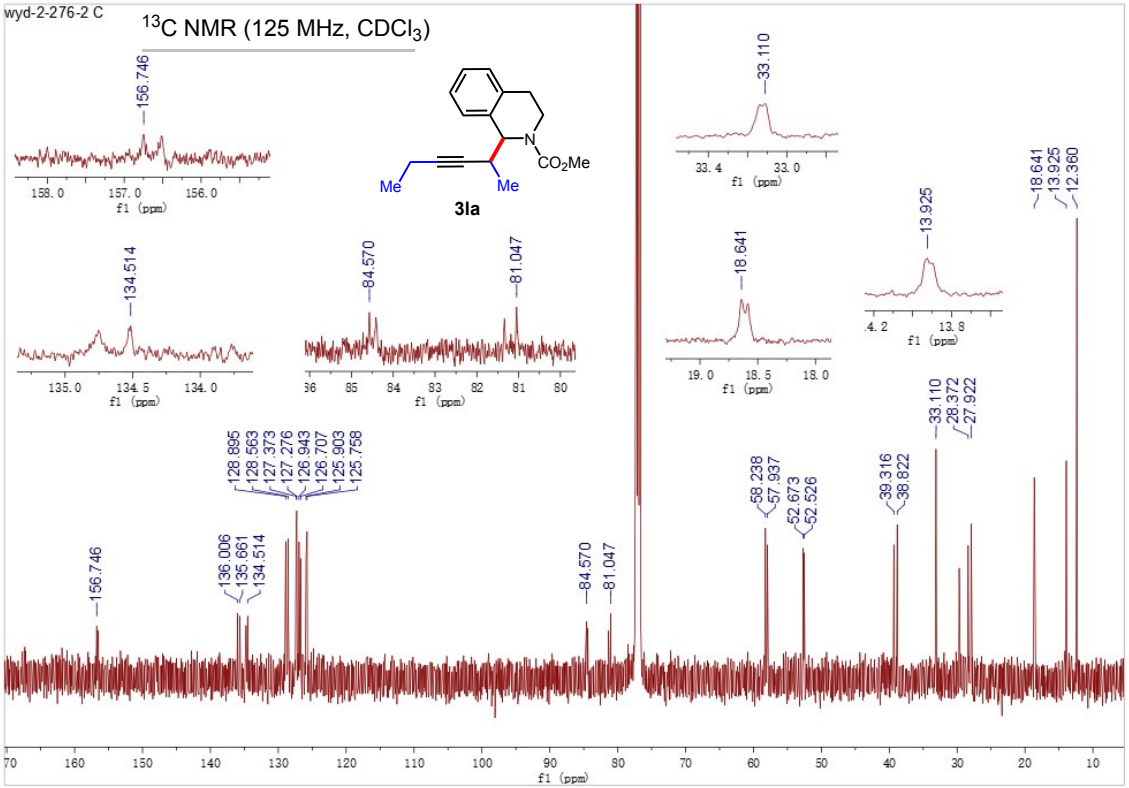

wyd-3-58 H
^{1}H NMR (500 MHz, $CDCl_3$)
3ka

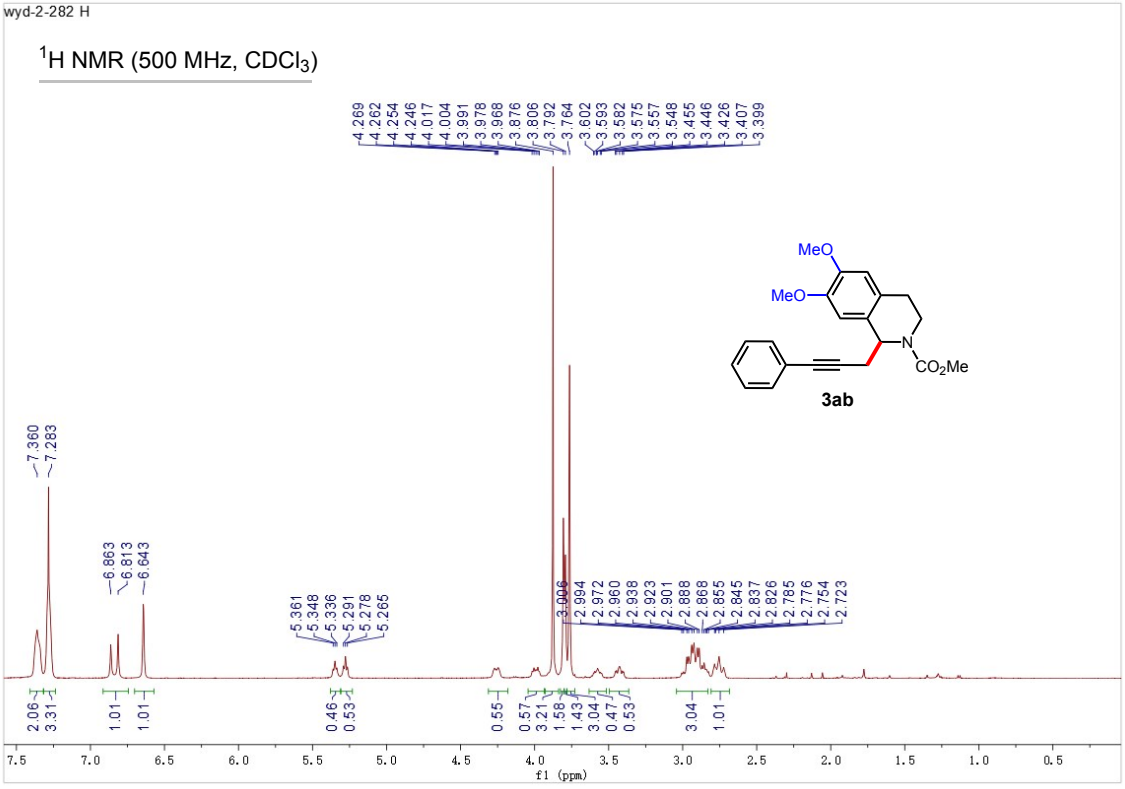

wyd-3-58 C
^{13}C NMR (125 MHz, $CDCl_3$)
3ka

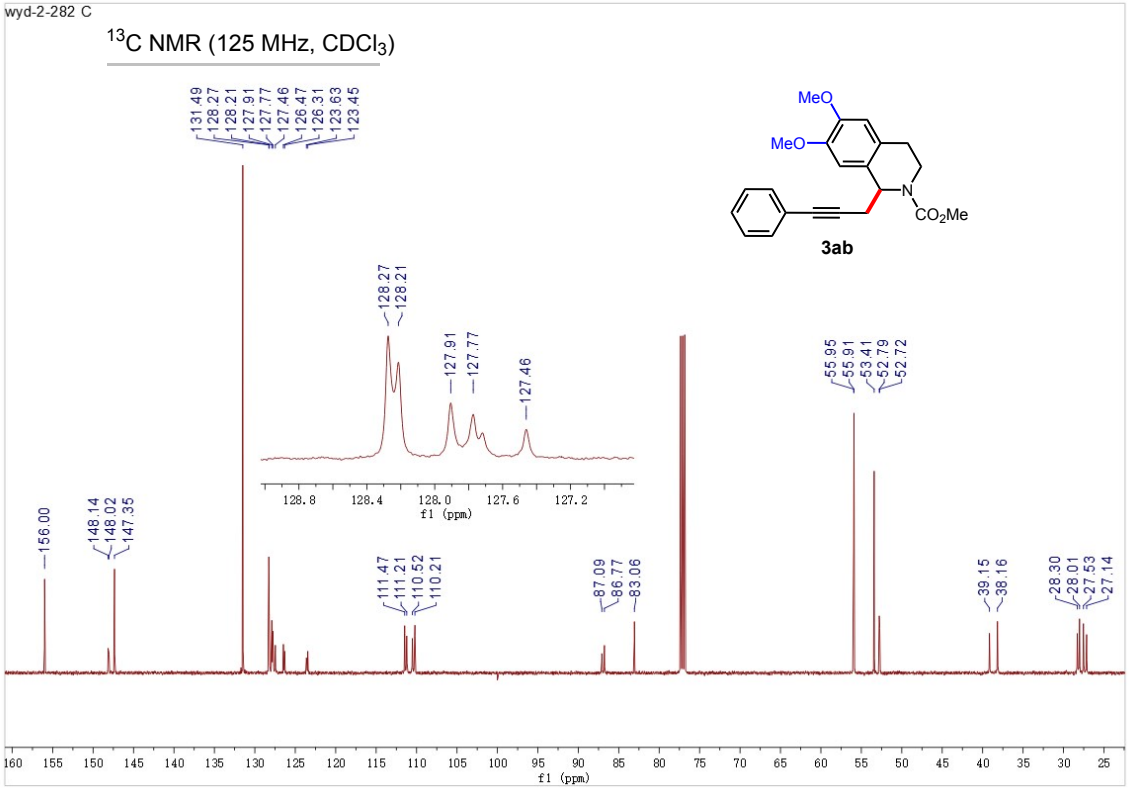

wyd-2-276-1 H
^{1}H NMR (500 MHz, $CDCl_3$)
3la
Me
Me
N
CO_2Me
f1 (ppm)

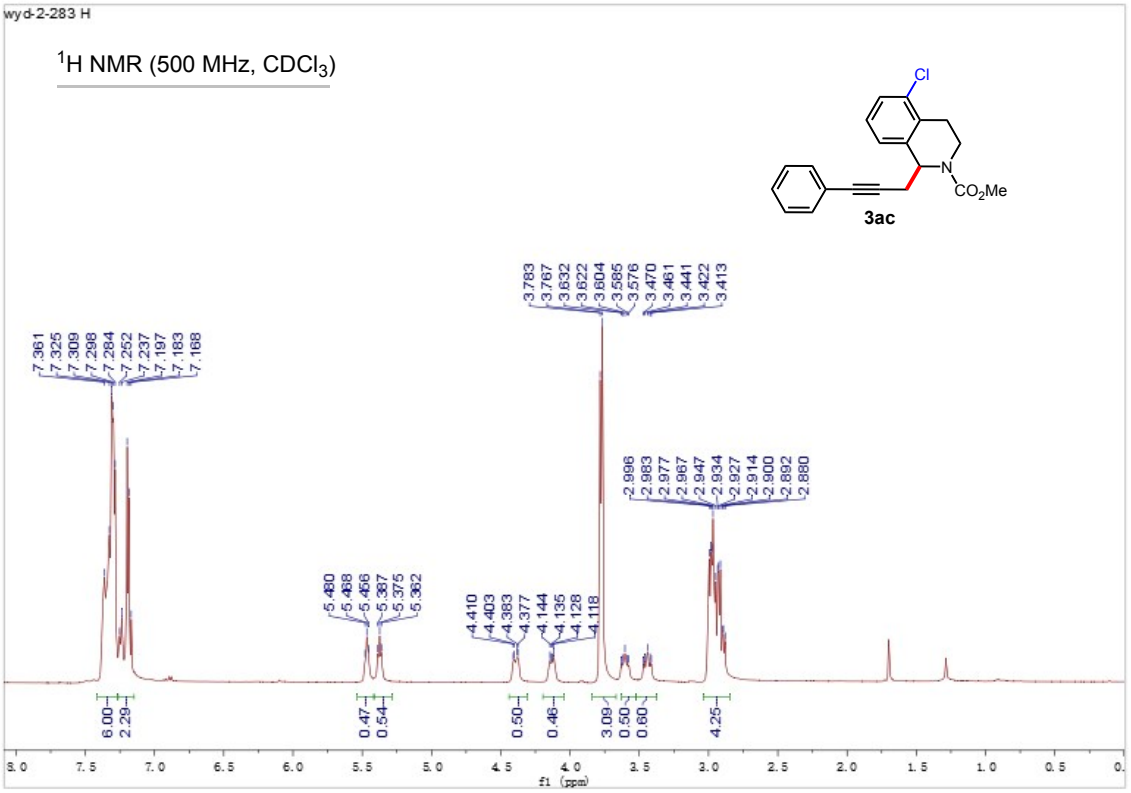

wyd-2-276-1 C
^{13}C NMR (125 MHz, $CDCl_3$)
135.489
3la
Me
Me
N
CO_2Me
f1 (ppm)

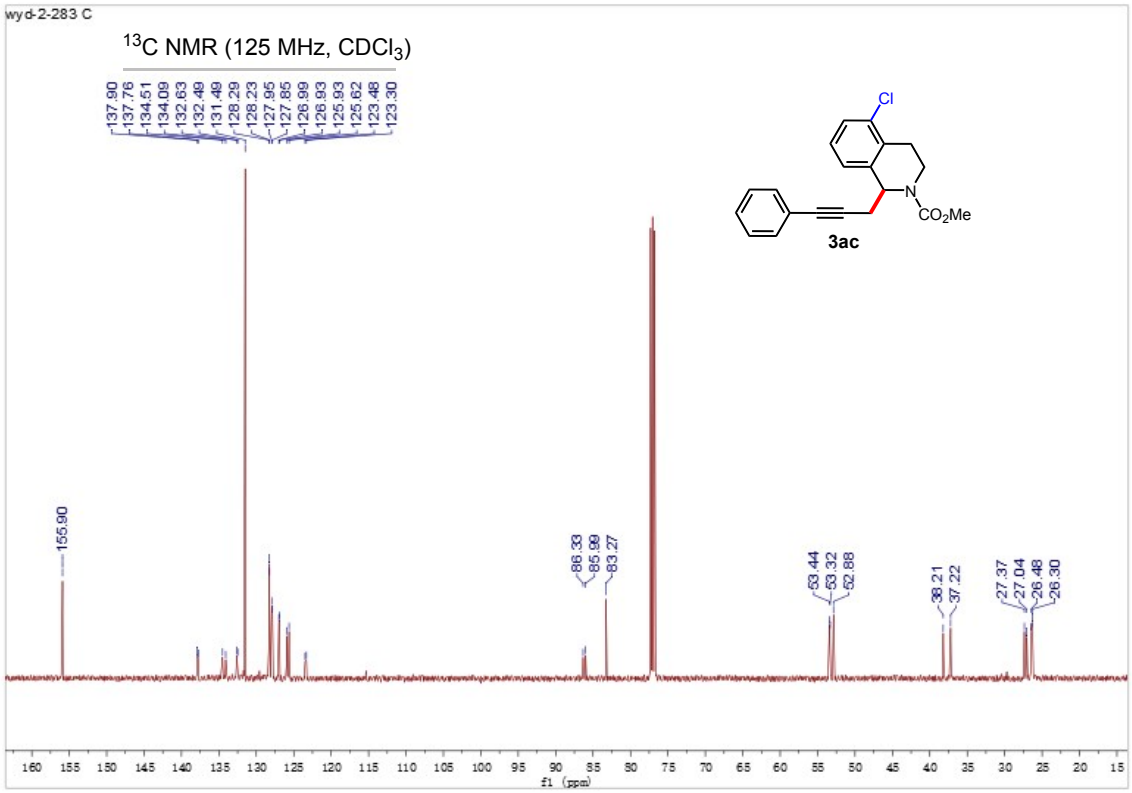

wyd-2-276-2 H
^{1}H NMR (500 MHz, $CDCl_3$)
Me
Me
N
CO_2Me
3la
7.260
7.179
7.162
7.147
7.130
4.182
4.170
4.160
4.007
3.993
3.983
3.934
3.924
3.915
3.906
3.889
3.879
3.833
3.822
3.813
3.804
3.787
3.777
3.725
5.181
5.169
5.003
4.991
3.028
2.967
2.947
2.931
2.913
2.899
2.880
2.866
2.848
2.826
2.818
2.810
2.050
2.037
2.023
1.268
1.254
1.239
1.225
1.000
0.985
0.970
0.955
3.91
0.50
0.54
0.53
0.50
0.52
0.58
2.94
3.18
1.99
1.69
1.56
2.90
f1 (ppm)

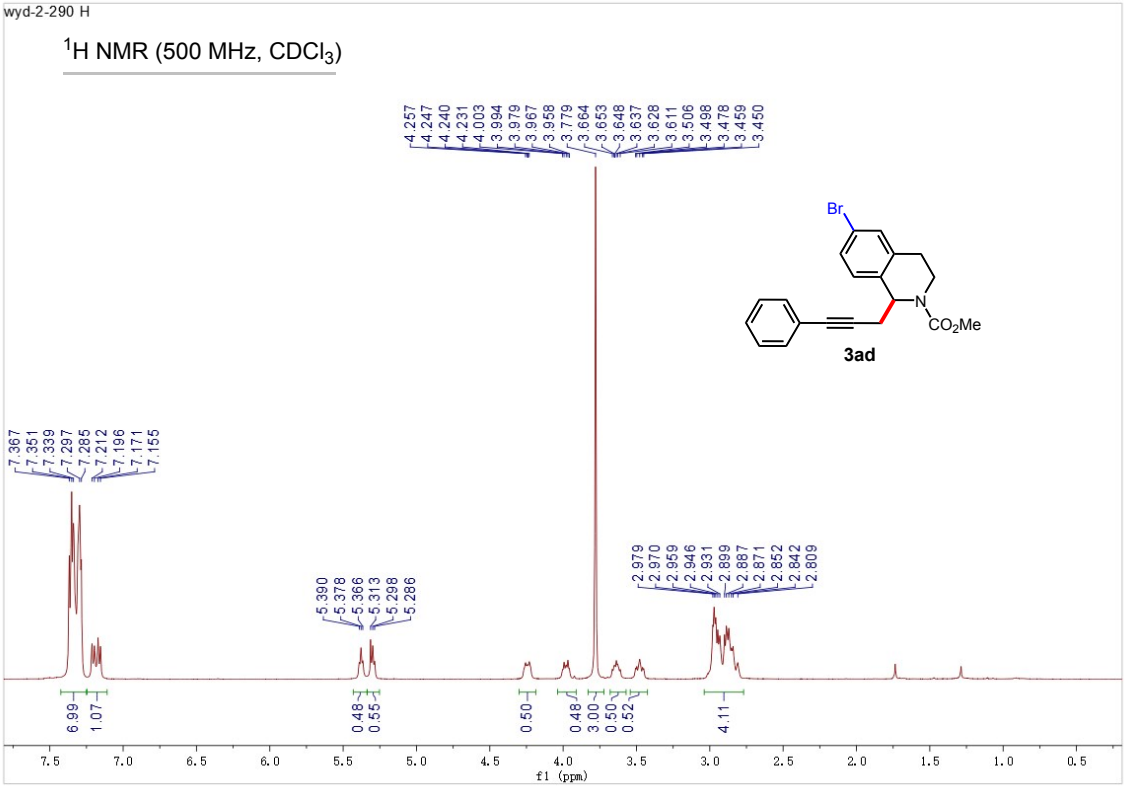

wyd-2-276-2 C
^{13}C NMR (125 MHz, $CDCl_3$)
Me
Me
N
CO_2Me
3la
156.746
134.514
84.570
81.047
33.110
18.641
13.925
128.895
128.563
127.373
127.276
126.943
126.707
125.903
125.758
136.006
135.661
134.514
58.238
57.937
52.673
52.526
39.316
38.822
33.110
28.372
27.922
18.641
13.925
12.360
f1 (ppm)

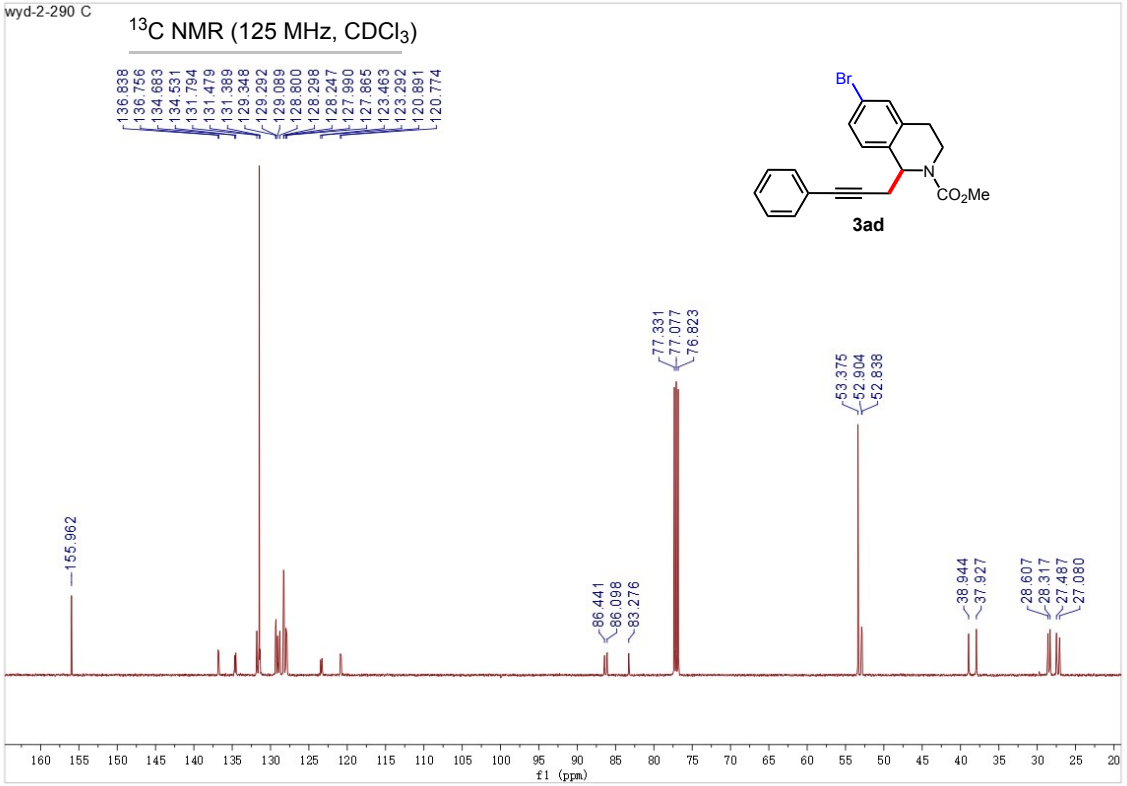

wyd-2-282 H
^{1}H NMR (500 MHz, $CDCl_3$)
MeO
MeO
N
CO_2Me
3ab
f1 (ppm)

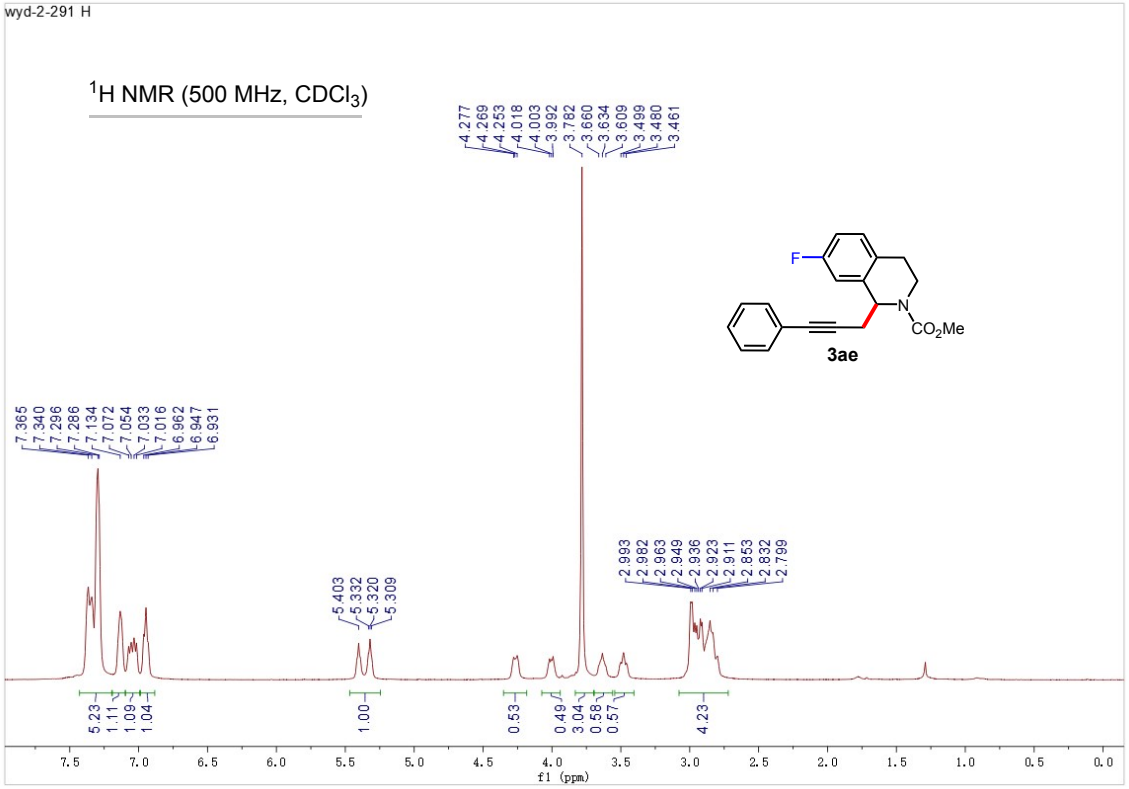

wyd-2-282 C
^{13}C NMR (125 MHz, $CDCl_3$)
MeO
MeO
N
CO_2Me
3ab
f1 (ppm)

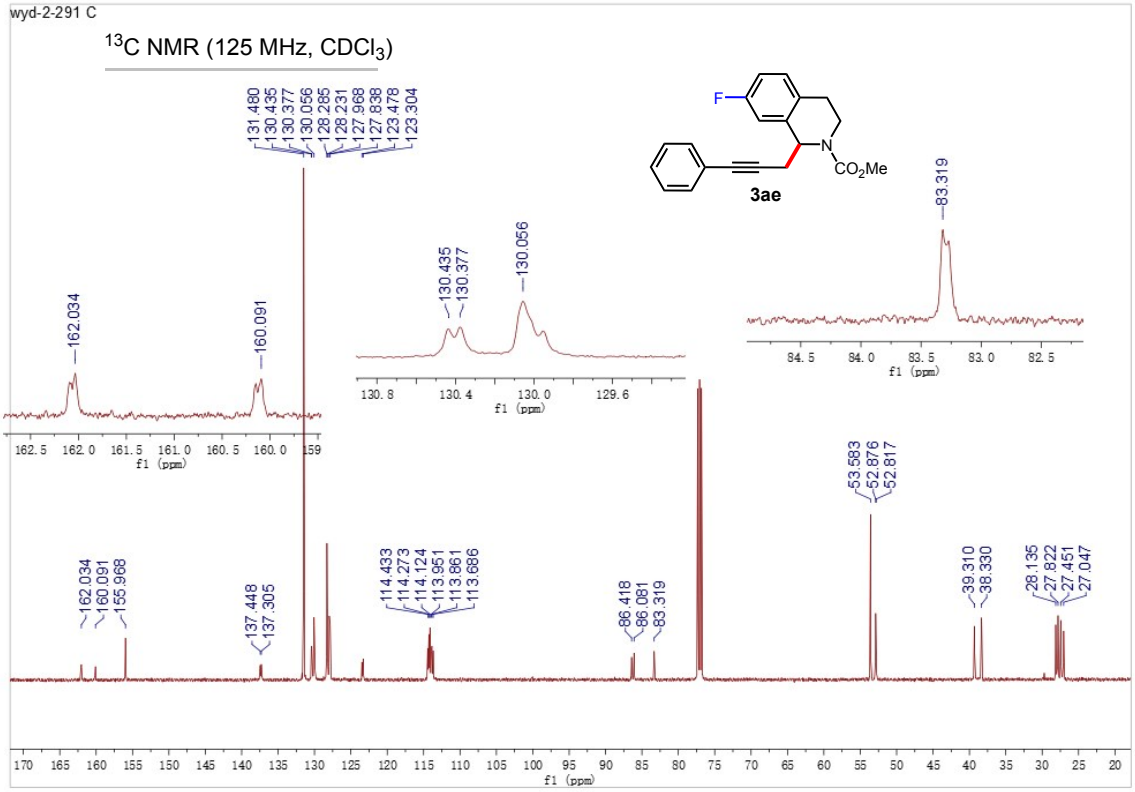

wyd-2-283 H
^{1}H NMR (500 MHz, $CDCl_3$)
Cl
N
CO_2Me
3ac
7.361
7.325
7.309
7.298
7.284
7.252
7.237
7.197
7.183
7.168
5.480
5.468
5.456
5.387
5.375
5.362
4.410
4.403
4.383
4.377
4.144
4.135
4.128
4.118
3.783
3.767
3.632
3.622
3.604
3.585
3.576
3.470
3.461
3.441
3.422
3.413
2.996
2.983
2.977
2.967
2.947
2.934
2.927
2.914
2.900
2.892
2.880
6.00
2.29
0.47
0.54
0.50
0.46
3.09
0.50
0.60
4.25
f1 (ppm)

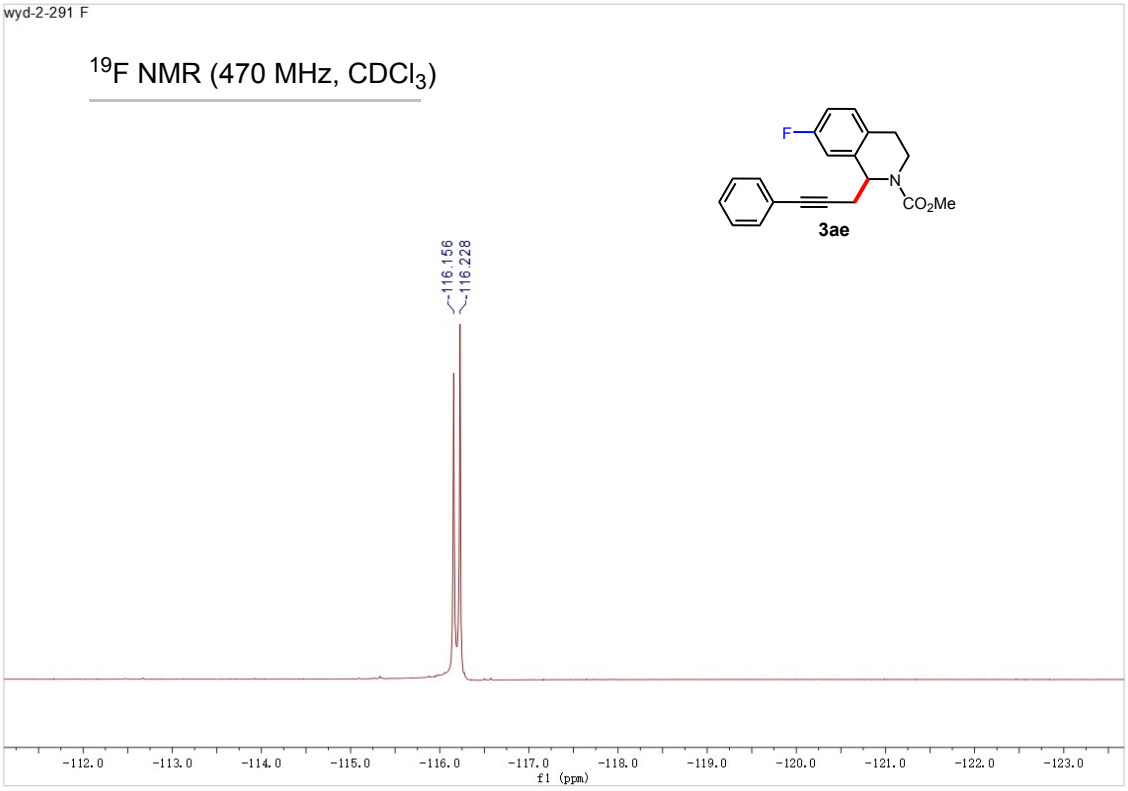

wyd-2-283 C
^{13}C NMR (125 MHz, $CDCl_3$)
Cl
N
CO_2Me
3ac
137.90
137.76
134.51
134.09
132.63
132.49
131.49
128.29
128.23
127.95
127.85
126.99
126.93
125.93
125.62
123.48
123.30
155.90
86.33
85.99
83.27
63.44
63.32
62.88
38.21
37.22
27.37
27.04
26.48
26.30
f1 (ppm)


wyd-2-290 H
^{1}H NMR (500 MHz, $CDCl_3$)
4.257
4.247
4.240
4.231
4.003
3.994
3.979
3.967
3.958
3.779
3.664
3.653
3.648
3.637
3.628
3.611
3.506
3.498
3.478
3.459
3.450
Br
N
CO_2Me
3ad
7.367
7.351
7.339
7.297
7.285
7.212
7.196
7.171
7.155
5.390
5.378
5.366
5.313
5.298
5.286
2.979
2.970
2.959
2.946
2.931
2.899
2.887
2.871
2.852
2.842
2.809
6.99
1.07
0.48
0.55
0.50
0.48
3.00
0.50
0.52
4.11
f1 (ppm)


wyd-2-290 C
^{13}C NMR (125 MHz, $CDCl_3$)
136.838
136.756
134.683
134.531
131.794
131.479
131.389
129.348
129.292
129.089
128.800
128.298
128.247
127.990
127.865
123.463
123.292
120.891
120.774
Br
N
CO_2Me
3ad
77.331
77.077
76.823
53.375
52.904
52.838
155.962
86.441
86.098
83.276
38.944
37.927
28.607
28.317
27.487
27.080
f1 (ppm)

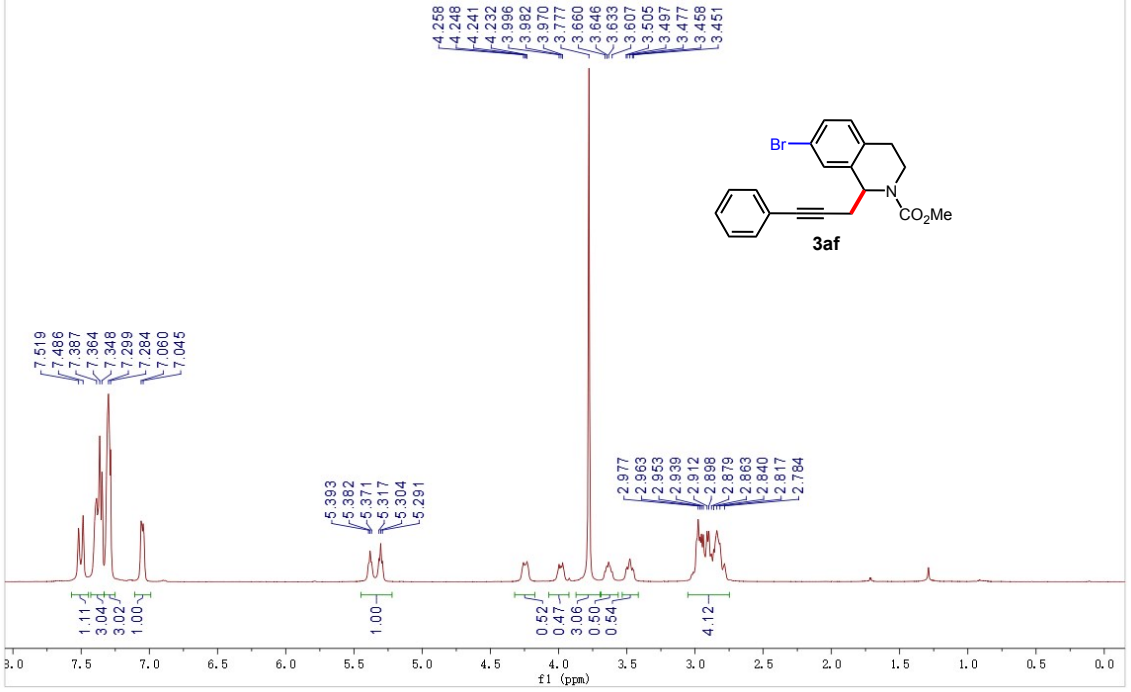

wyd-2-291 H
^{1}H NMR (500 MHz, $CDCl_3$)
F
N
CO_2Me
3ae
f1 (ppm)


wyd-2-291 C
^{13}C NMR (125 MHz, $CDCl_3$)
F
N
CO_2Me
3ae
f1 (ppm)


wyd-2-291 F
19F NMR (470 MHz, CDCl3)
F
N
CO2Me
3ae
-116.156
-116.228
-112.0
-113.0
-114.0
-115.0
-116.0
-117.0
-118.0
-119.0
-120.0
-121.0
-122.0
-123.0
f1 (ppm)

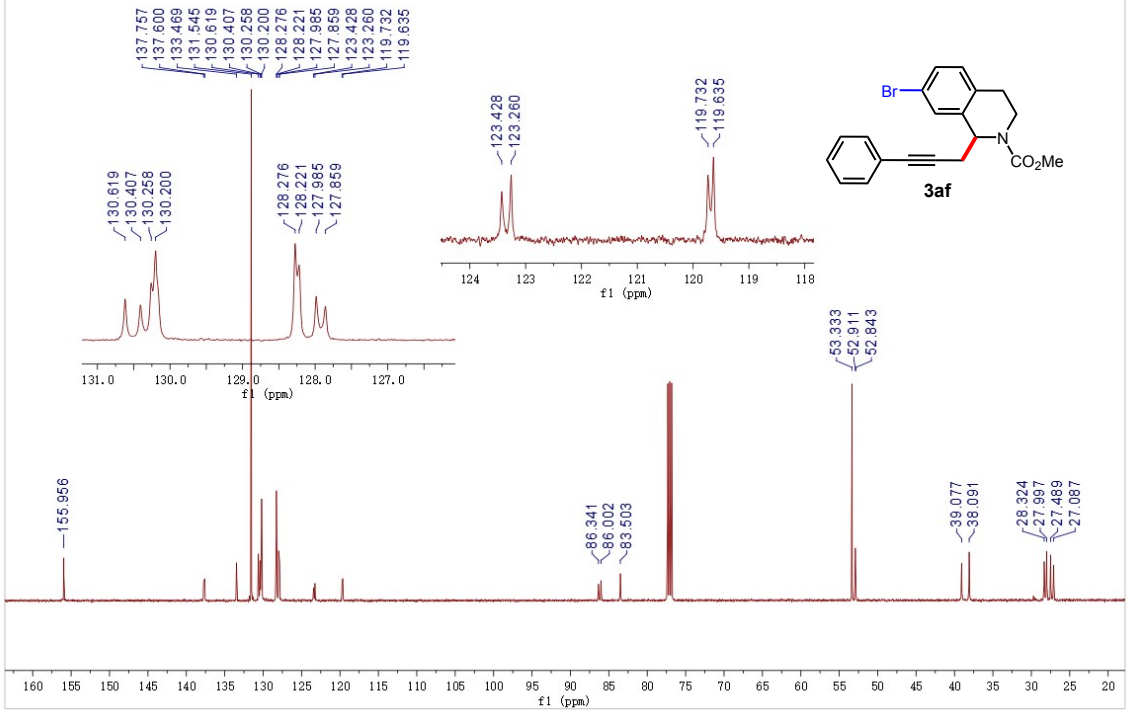
wyd-2-292 H

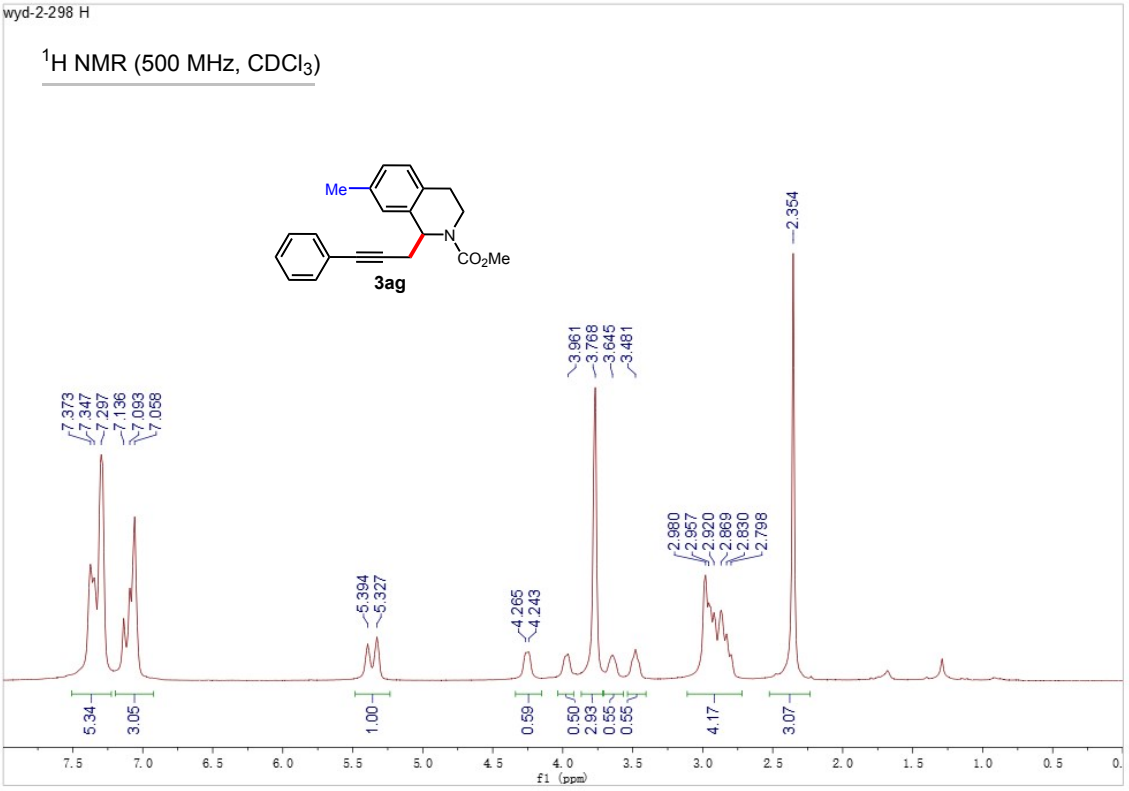
^{1}H NMR (500 MHz, $CDCl_3$)

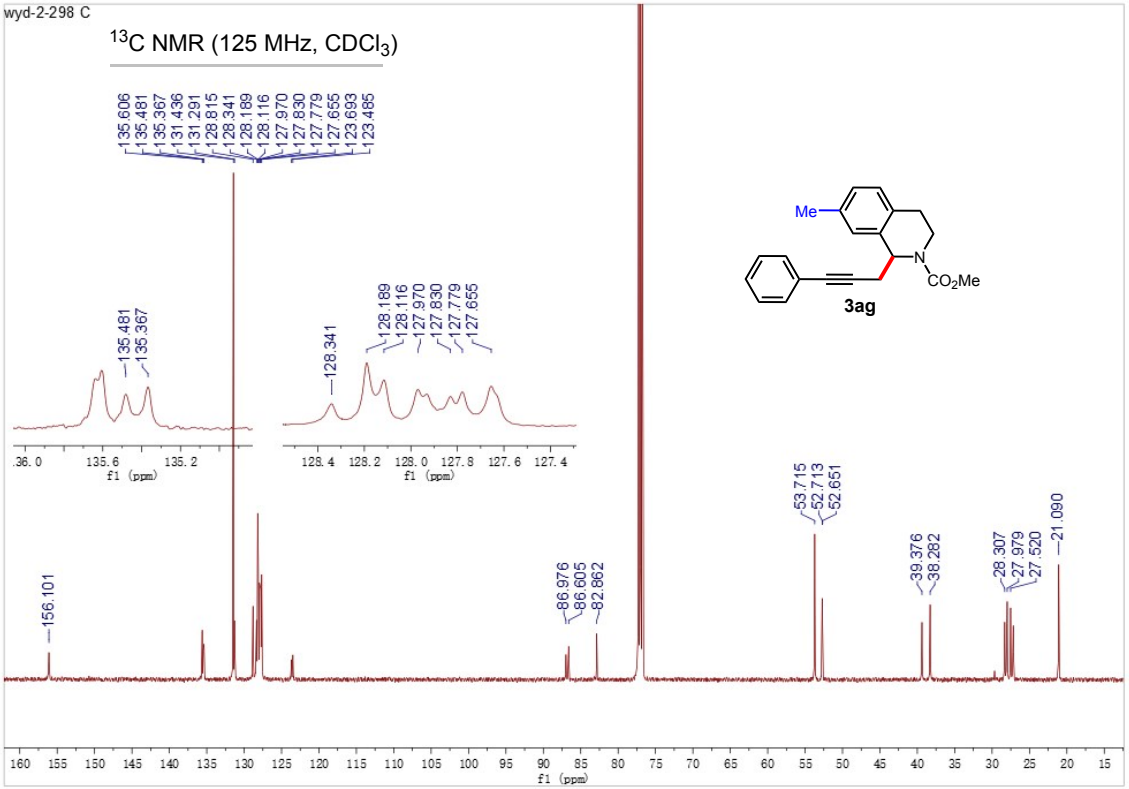


wyd-2-292 C

^{13}C NMR (125 MHz, $CDCl_3$)

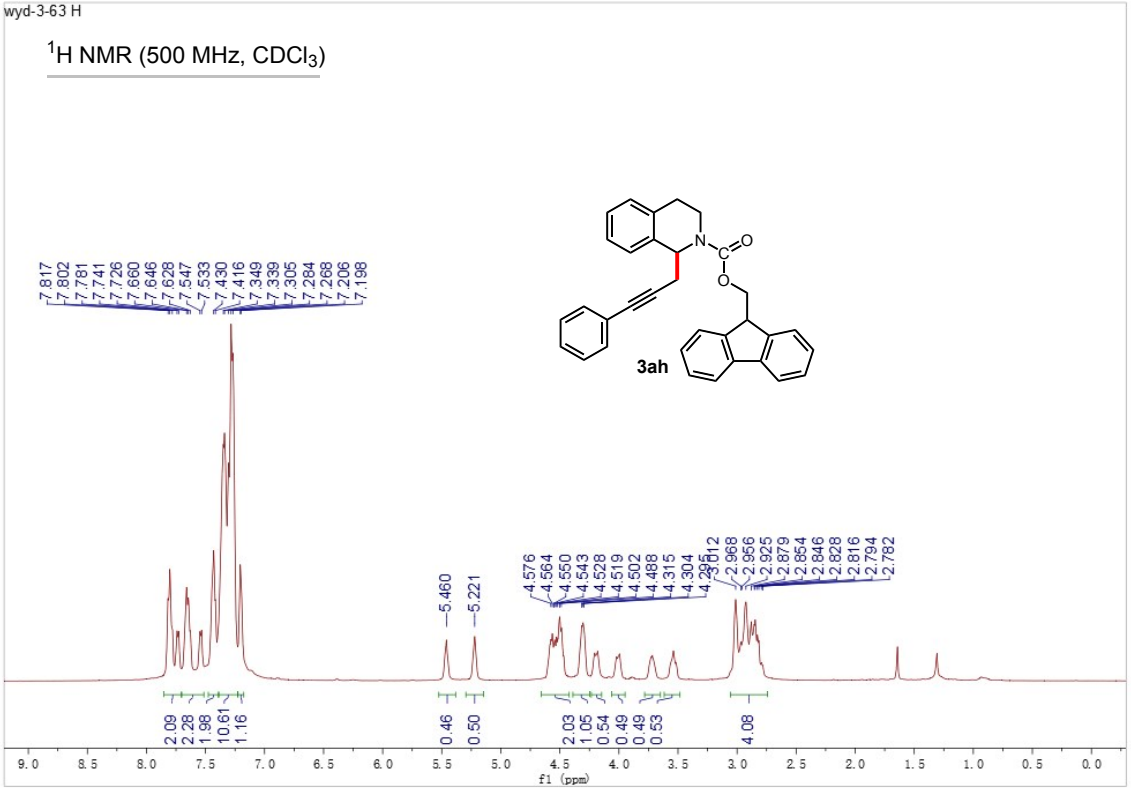



wyd-2-298 H
^{1}H NMR (500 MHz, $CDCl_3$)
Me
N
CO_2Me
3ag
7.373
7.347
7.297
7.136
7.093
7.058
5.394
5.327
4.265
4.243
3.961
3.768
3.645
3.481
2.980
2.957
2.920
2.869
2.830
2.798
2.354
5.34
3.05
1.00
0.59
0.50
2.93
0.55
0.55
4.17
3.07
f1 (ppm)


wyd-2-298 C
^{13}C NMR (125 MHz, $CDCl_3$)
135.606
135.481
135.367
131.436
131.291
128.815
128.341
128.189
128.116
127.970
127.830
127.779
127.655
123.693
123.485
Me
N
CO_2Me
3ag
156.101
86.976
86.605
82.862
53.715
52.713
52.651
39.376
38.282
28.307
27.979
27.520
21.090
f1 (ppm)

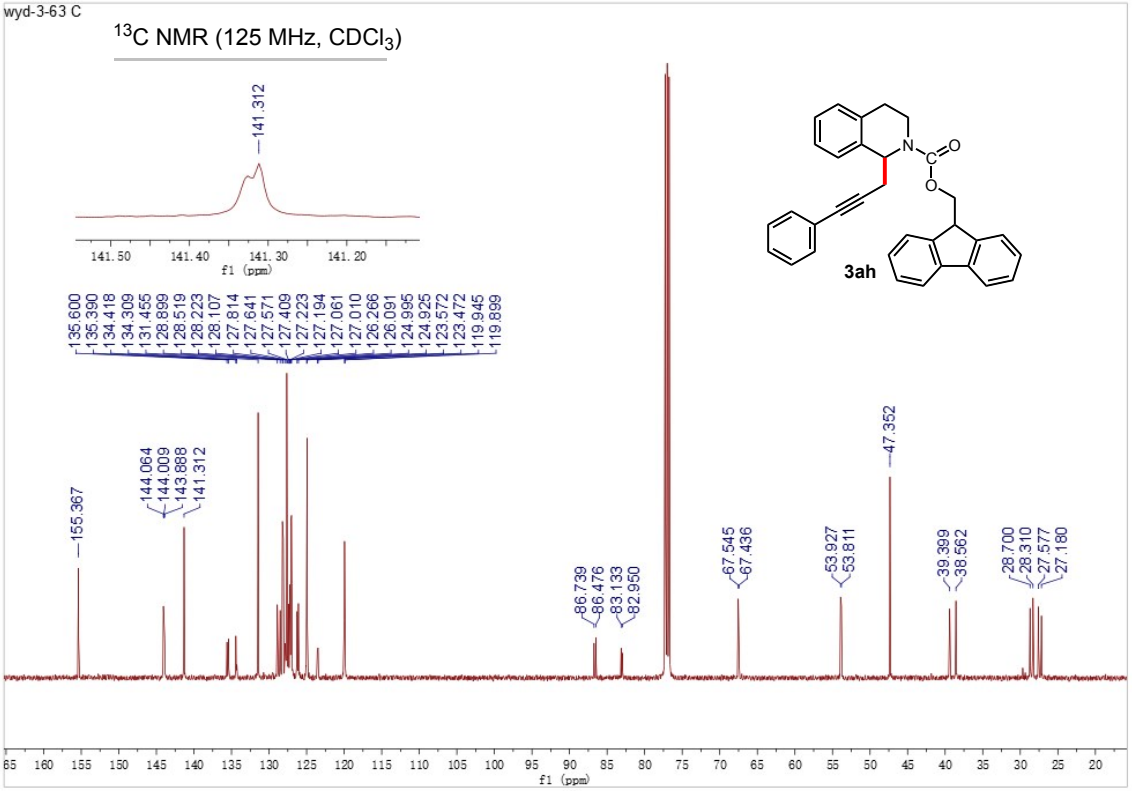
wyd-3-63 H

^{1}H NMR (500 MHz, $CDCl_3$)



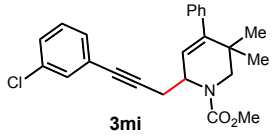
wyd-3-63 C

^{13}C NMR (125 MHz, $CDCl_3$)

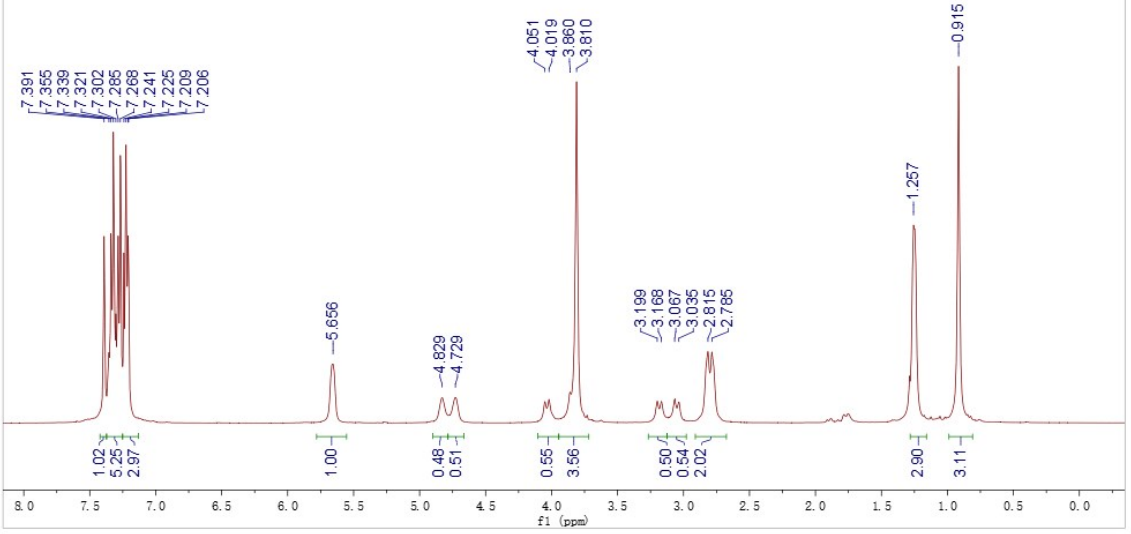


wyd-3-132 H

^{1}H NMR (400 MHz, $CDCl_3$)

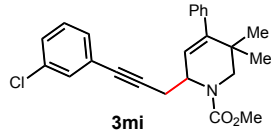


3mi



wyd-3-132 C

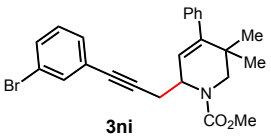
^{13}C NMR (100 MHz, $CDCl_3$)



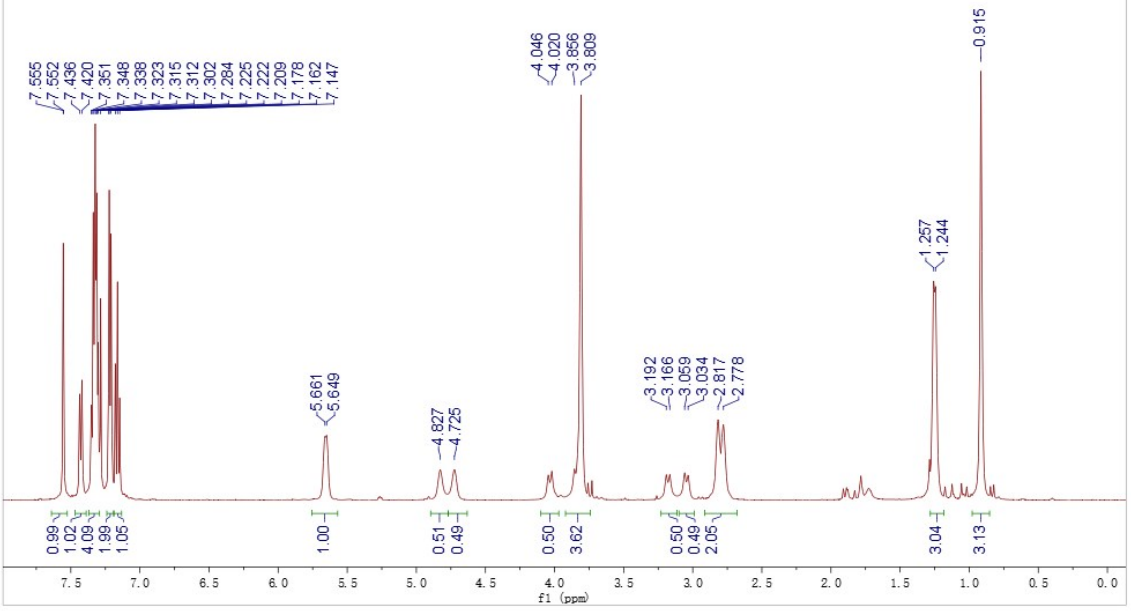
3mi

WYD-3-145 H

^{1}H NMR (500 MHz, $CDCl_3$)

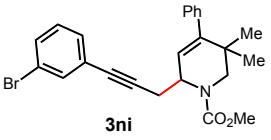


3ni

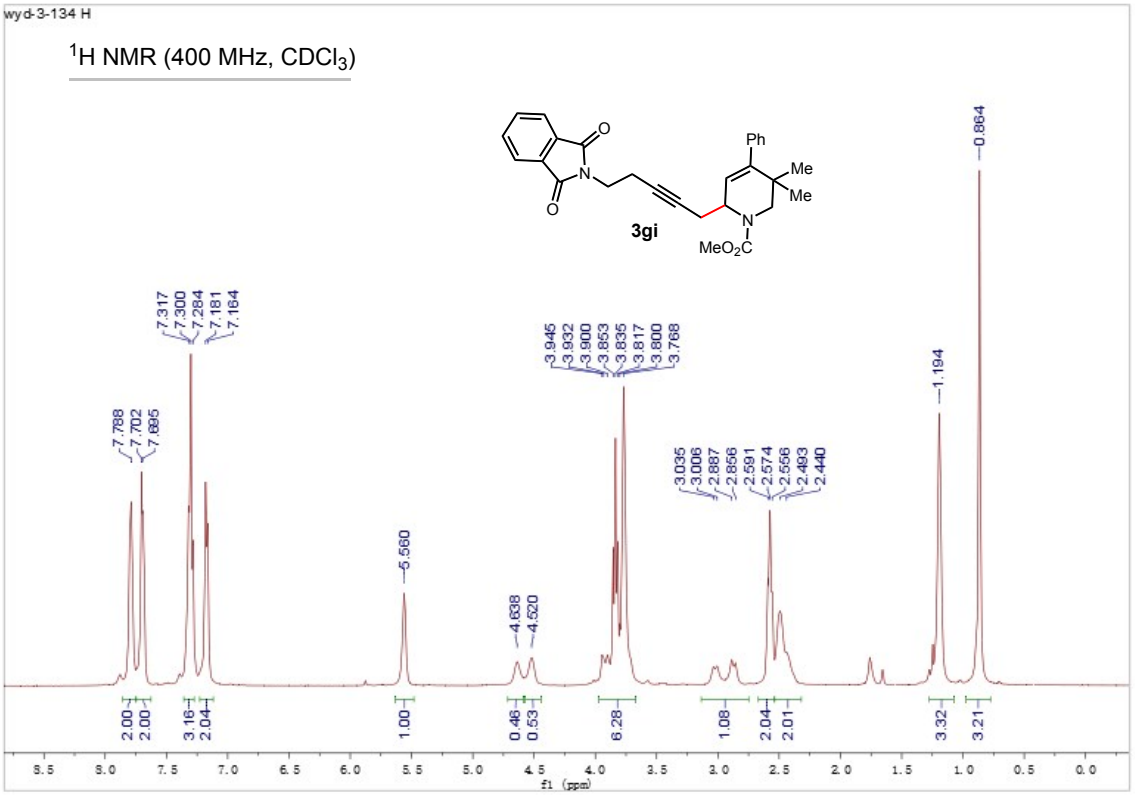


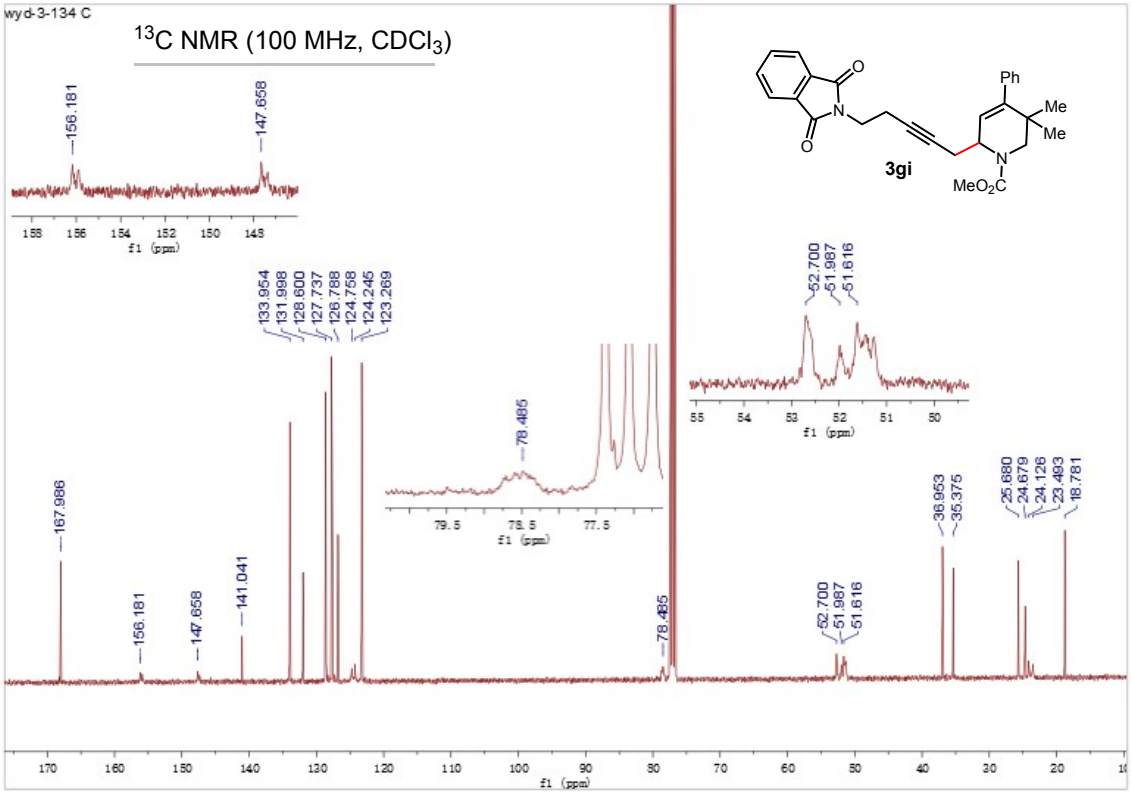
WYD-3-145 C

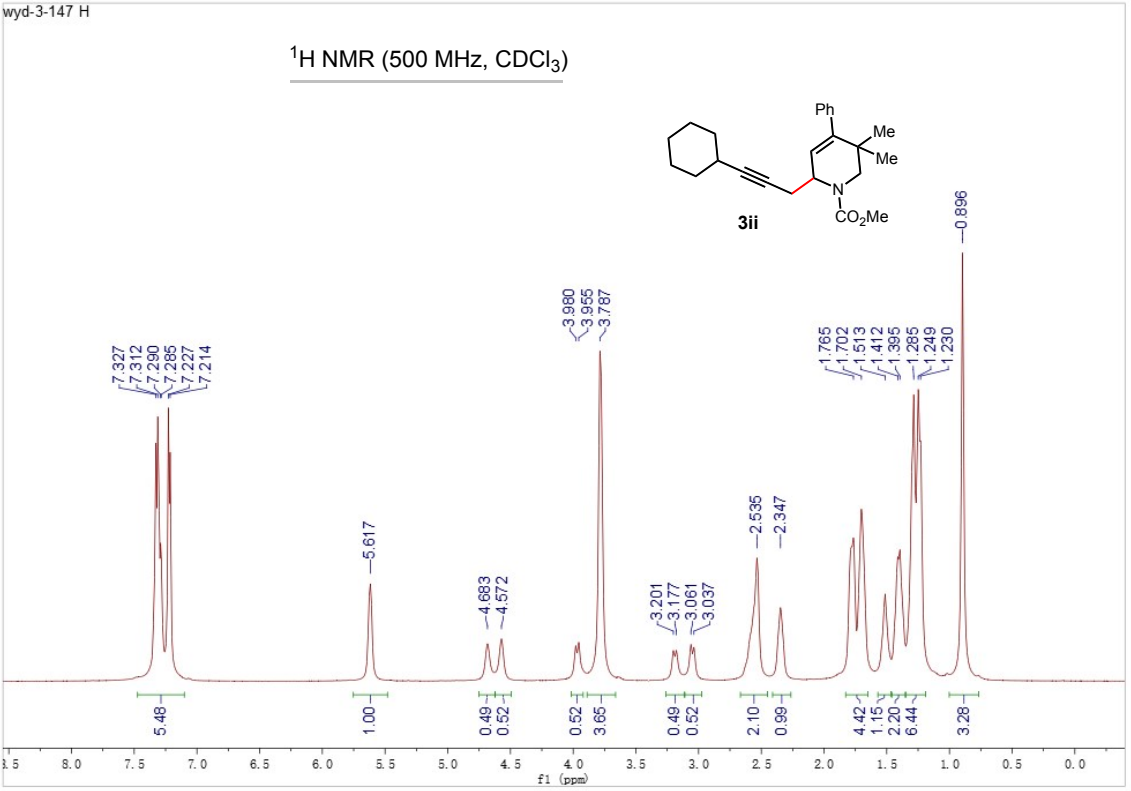
^{13}C NMR (125 MHz, $CDCl_3$)

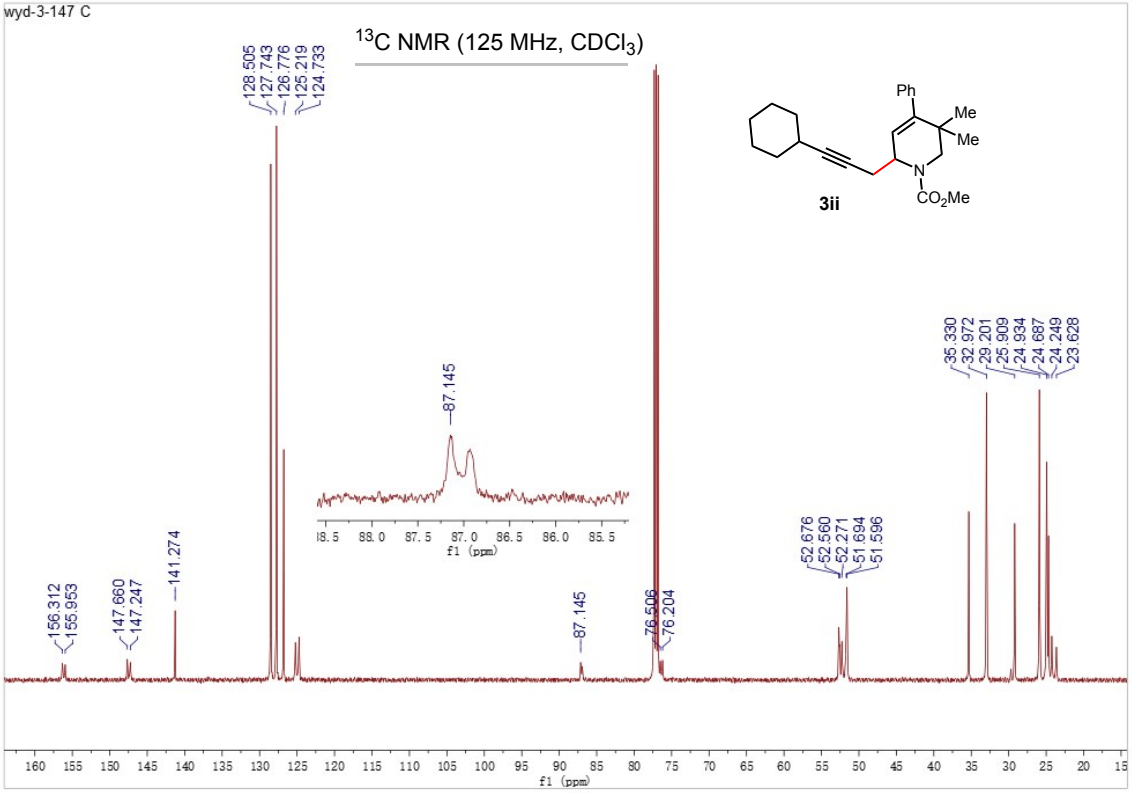


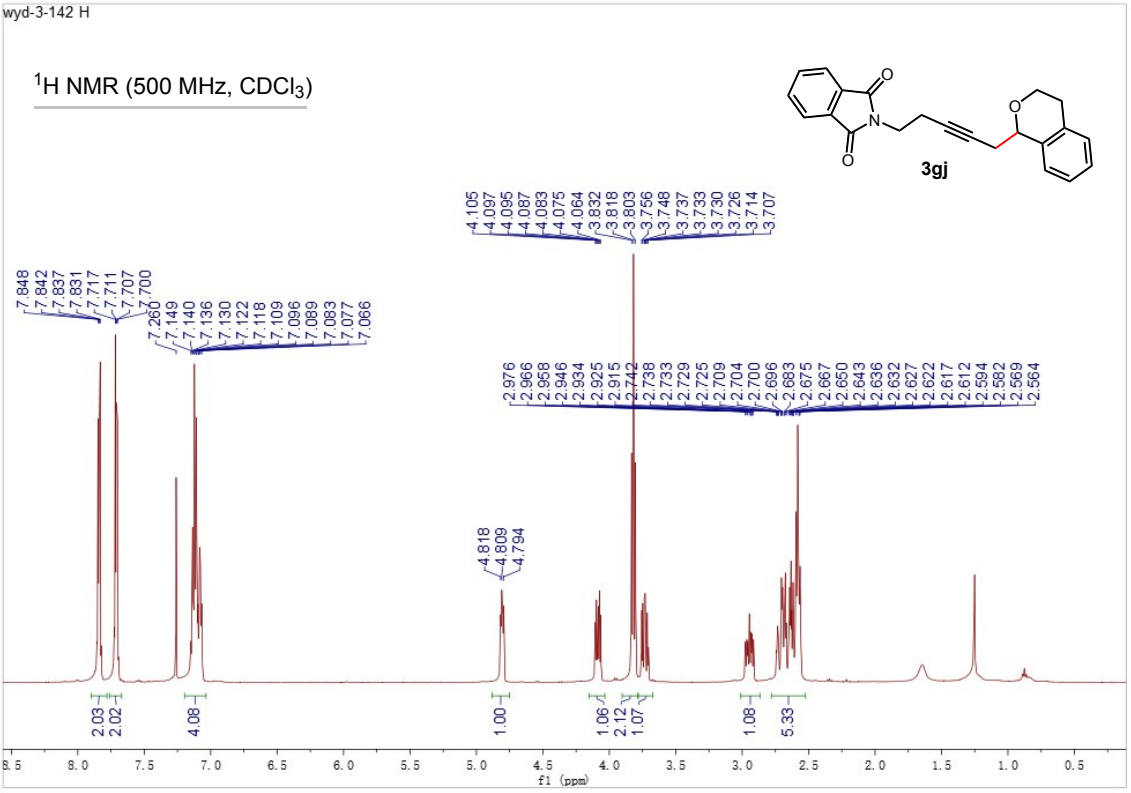
3ni

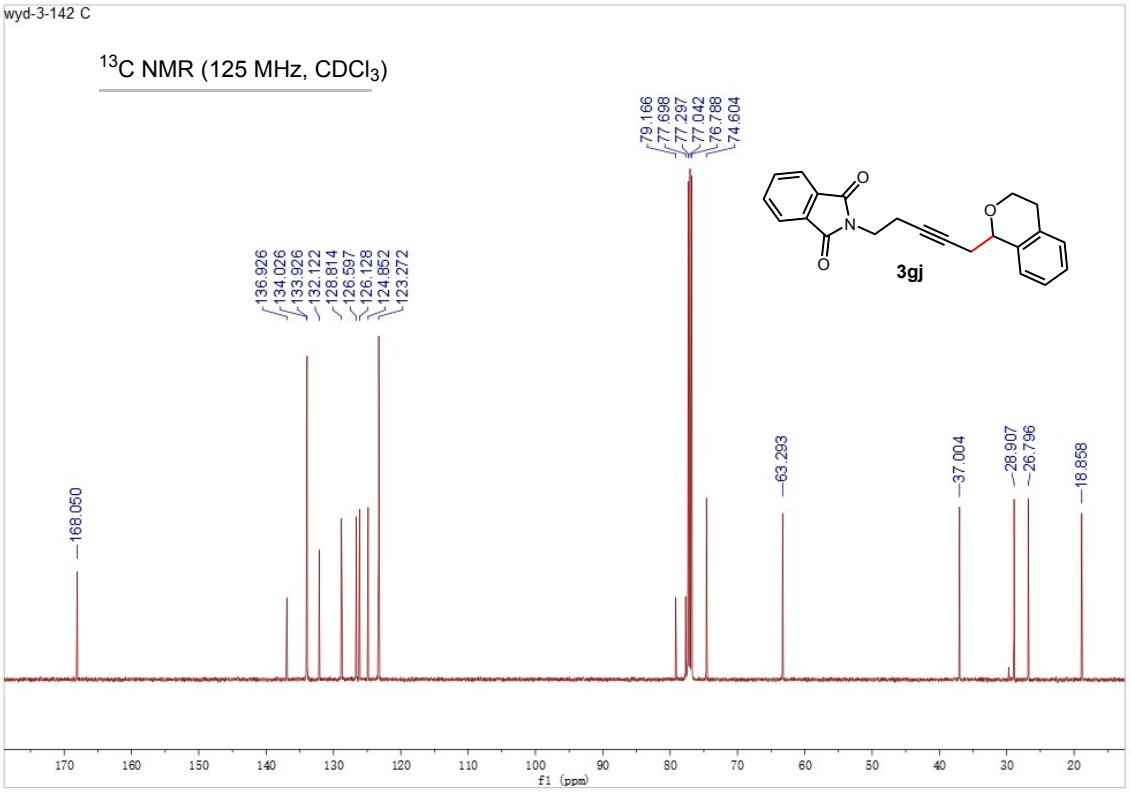

wyd-3-134 H
^{1}H NMR (400 MHz, $CDCl_3$)
3gi
Ph
Me
Me
N
MeO_2C
O
O
N
O
7.788
7.702
7.695
7.317
7.300
7.284
7.181
7.164
5.560
4.638
4.620
3.945
3.932
3.900
3.863
3.835
3.817
3.800
3.768
3.035
3.006
2.887
2.856
2.591
2.574
2.556
2.493
2.440
1.194
0.864
2.00
2.00
3.16
2.04
1.00
0.46
0.53
6.28
1.08
2.04
2.01
3.32
3.21
f1 (ppm)


wyd-3-134 C
^{13}C NMR (100 MHz, $CDCl_3$)
3gi
Ph
Me
Me
N
MeO_2C
O
O
N
O
156.181
147.658
133.954
131.998
128.600
127.737
126.788
124.758
124.245
123.269
78.495
52.700
51.987
51.616
187.986
156.181
147.658
141.041
78.495
52.700
51.987
51.616
36.953
35.375
25.680
24.679
24.128
23.493
18.781
f1 (ppm)


wyd-3-147 H
^{1}H NMR (500 MHz, $CDCl_3$)
Ph
Me
Me
N
CO_2Me
3ii
7.327
7.312
7.290
7.285
7.227
7.214
5.617
4.683
4.572
3.980
3.955
3.787
3.201
3.177
3.061
3.037
2.535
2.347
1.765
1.702
1.513
1.412
1.395
1.285
1.249
1.230
0.896
5.48
1.00
0.49
0.52
0.52
3.65
0.49
0.52
2.10
0.99
4.42
1.15
2.20
6.44
3.28
f1 (ppm)

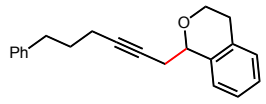

wyd-3-147 C
^{13}C NMR (125 MHz, $CDCl_3$)
Ph
Me
Me
N
CO_2Me
3ii
128.505
127.743
126.776
125.219
124.733
87.145
156.312
155.953
147.660
147.247
141.274
87.145
76.506
76.204
52.676
52.560
52.271
51.694
51.596
35.330
32.972
29.201
25.909
24.934
24.687
24.249
23.628
f1 (ppm)


wyd-3-142 H
^{1}H NMR (500 MHz, $CDCl_3$)
3gj
7.848
7.842
7.837
7.831
7.717
7.711
7.707
7.700
7.260
7.149
7.140
7.136
7.130
7.122
7.118
7.109
7.096
7.089
7.083
7.077
7.066
4.105
4.097
4.095
4.087
4.083
4.075
4.064
3.832
3.818
3.803
3.756
3.748
3.737
3.733
3.730
3.726
3.714
3.707
2.976
2.966
2.958
2.946
2.934
2.925
2.915
2.742
2.738
2.733
2.729
2.725
2.709
2.704
2.700
2.696
2.683
2.675
2.667
2.650
2.643
2.636
2.632
2.627
2.622
2.617
2.612
2.594
2.582
2.569
2.564
4.818
4.809
4.794
2.03
2.02
4.08
1.00
1.06
2.12
1.07
1.08
5.33
f1 (ppm)

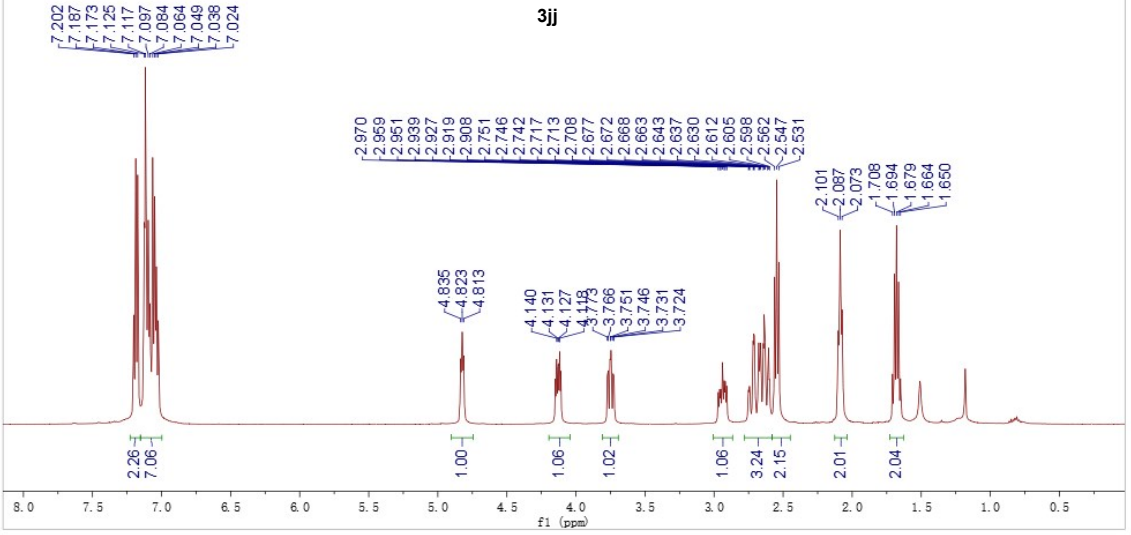

wyd-3-142 C
^{13}C NMR (125 MHz, $CDCl_3$)
3gj
79.166
77.698
77.297
77.042
76.788
74.604
136.926
134.026
133.926
132.122
128.814
126.597
126.128
124.852
123.272
168.050
63.293
37.004
28.907
26.796
18.858
f1 (ppm)

wyd-3-155 H

^{1}H NMR (500 MHz, $CDCl_3$)

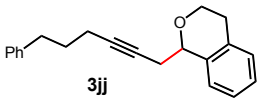


3jj

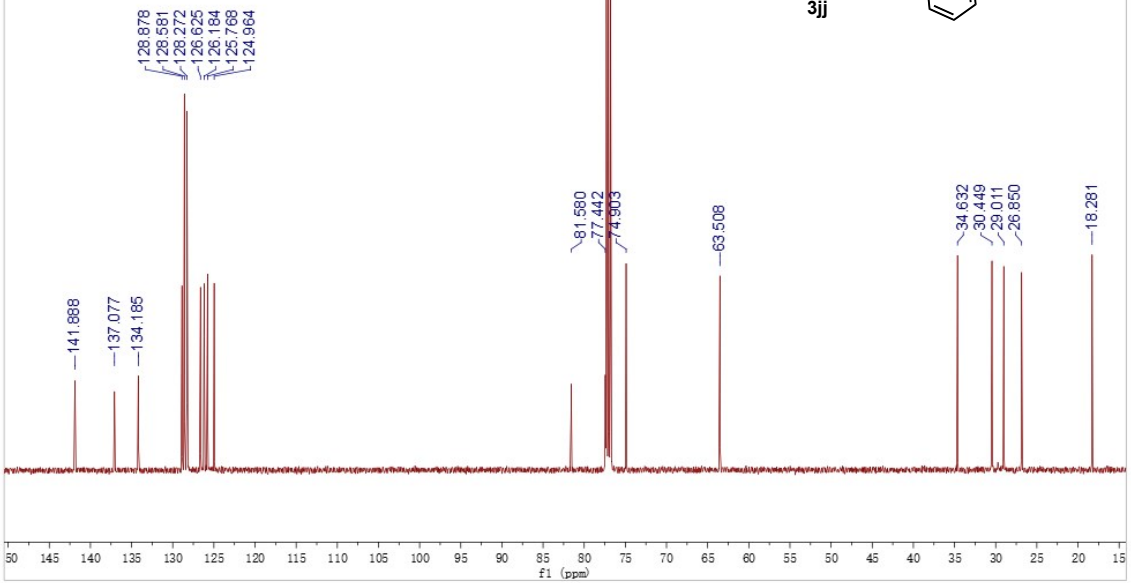


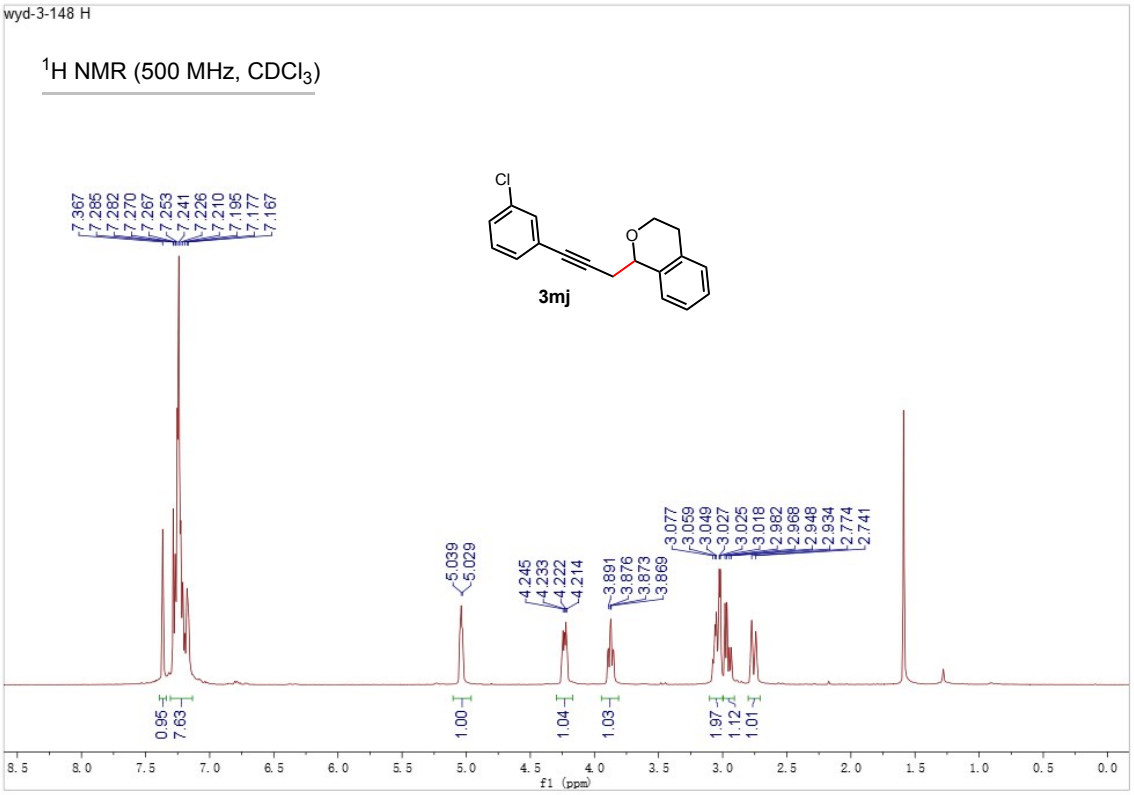
wyd-3-155 C

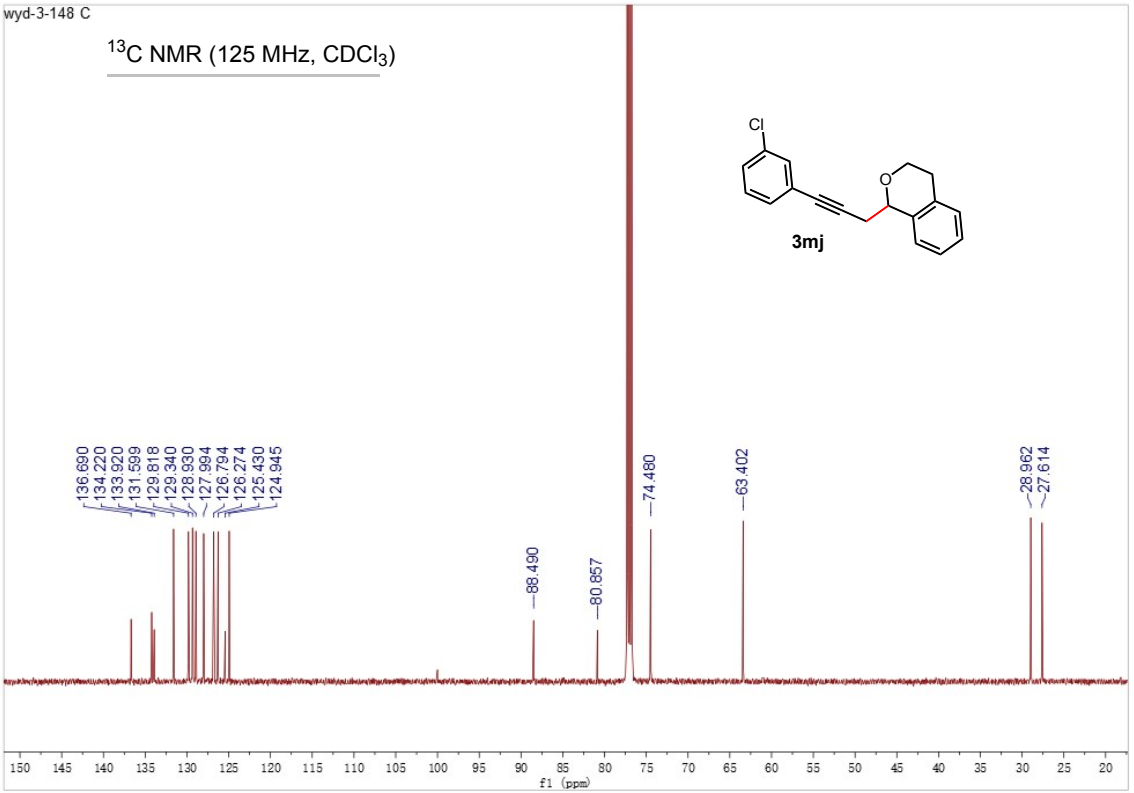
^{13}C NMR (125 MHz, $CDCl_3$)

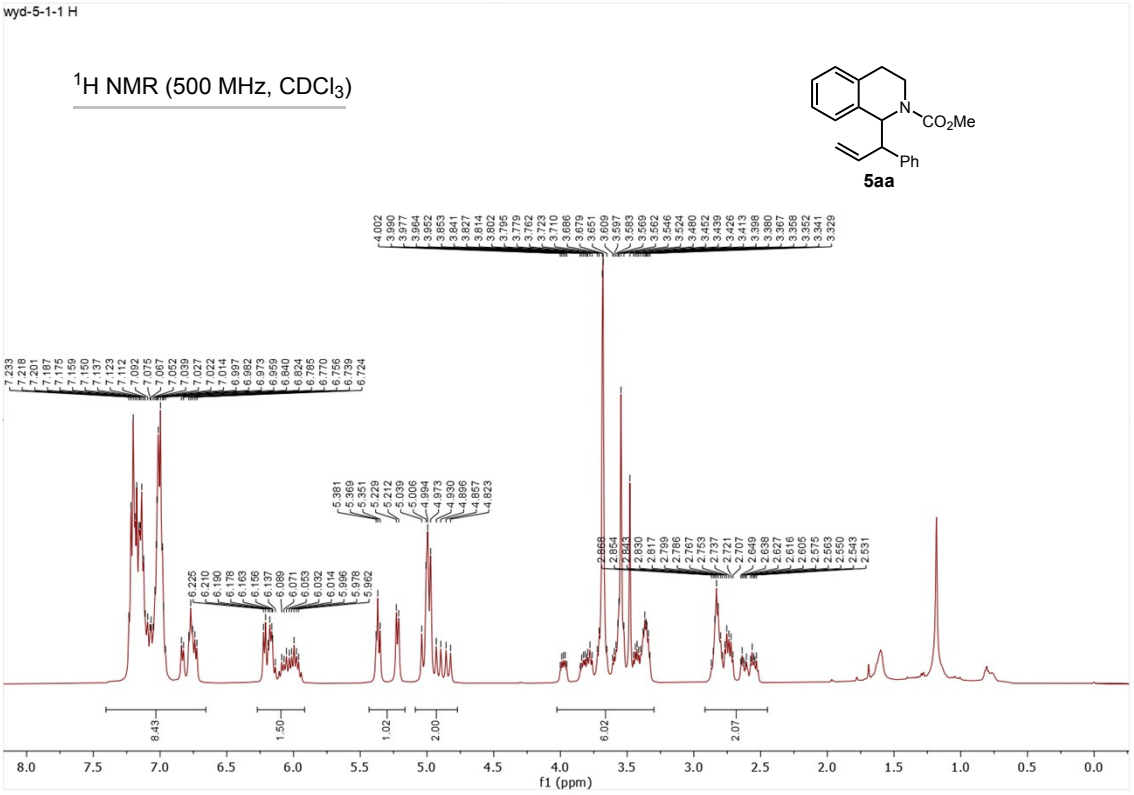


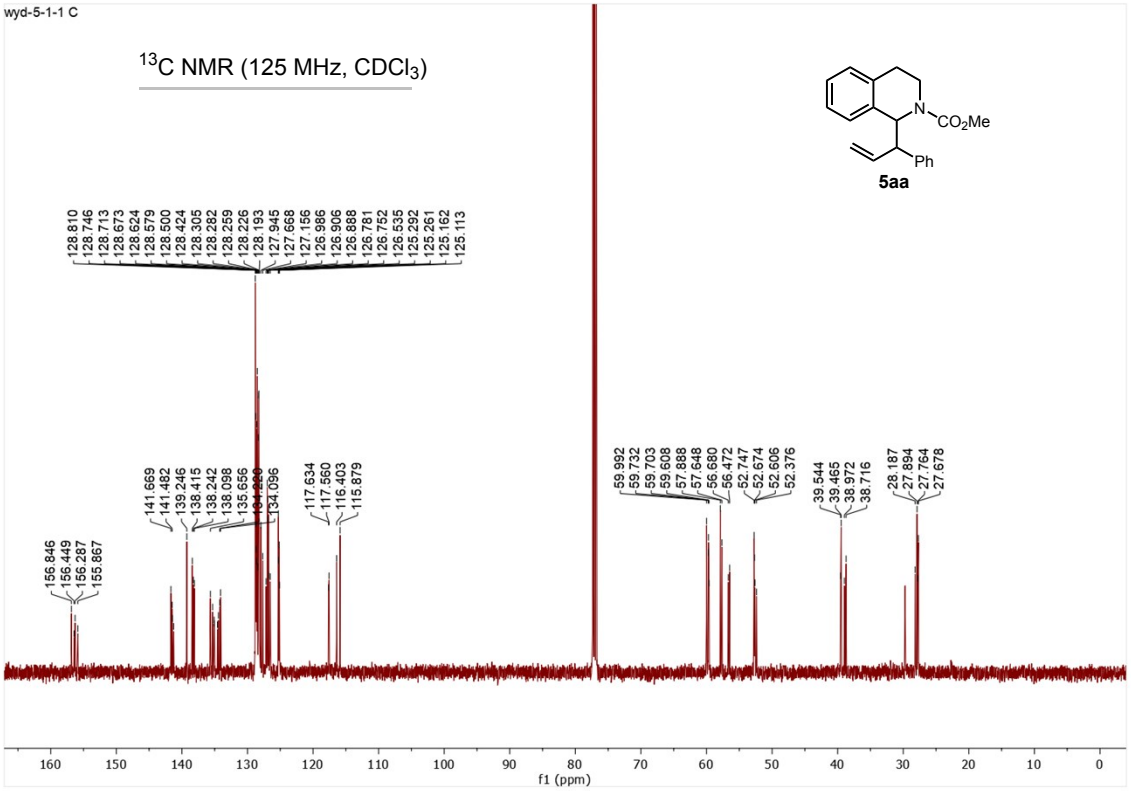
3jj




wyd-3-148 H
^{1}H NMR (500 MHz, $CDCl_3$)
7.367
7.285
7.282
7.270
7.267
7.253
7.241
7.226
7.210
7.195
7.177
7.167
Cl
O
3mj
5.039
5.029
4.245
4.233
4.222
4.214
3.891
3.876
3.873
3.869
3.077
3.059
3.049
3.027
3.025
3.018
2.982
2.968
2.948
2.934
2.774
2.741
0.95
7.63
1.00
1.04
1.03
1.97
1.12
1.01
f1 (ppm)

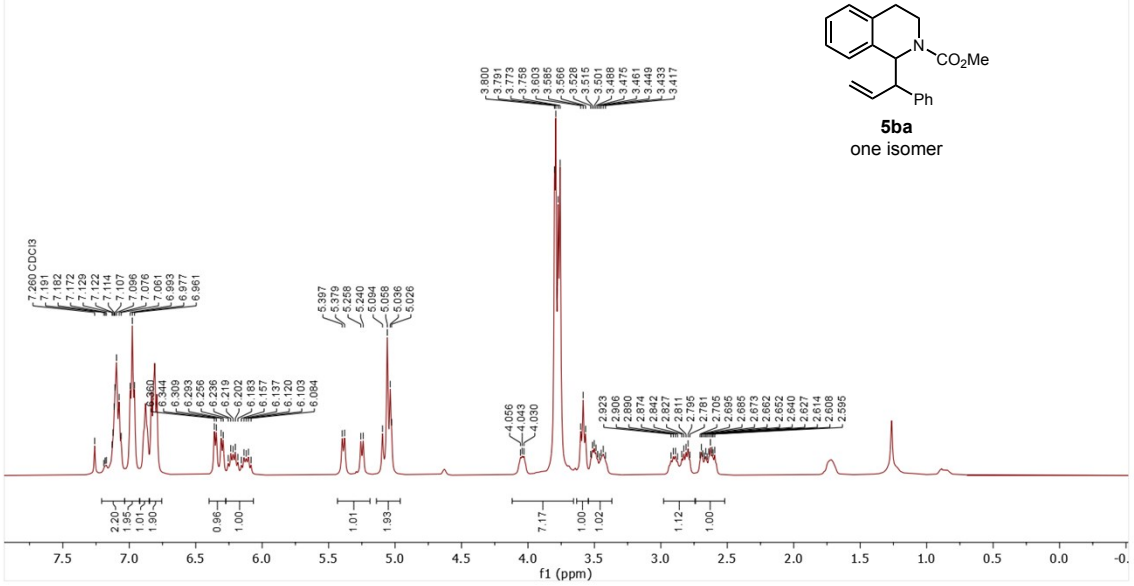

wyd-3-148 C
^{13}C NMR (125 MHz, $CDCl_3$)
Cl
O
3mj
136.690
134.220
133.920
131.599
129.818
129.340
128.930
127.994
126.794
126.274
125.430
124.945
88.490
80.857
74.480
63.402
28.962
27.614
f1 (ppm)


wyd-5-1-1 H
^{1}H NMR (500 MHz, $CDCl_3$)
N
CO_2Me
Ph
5aa
f1 (ppm)


wyd-5-1-1 C
^{13}C NMR (125 MHz, $CDCl_3$)
N
CO_2Me
Ph
5aa
f1 (ppm)

wyd-5-4-1 H

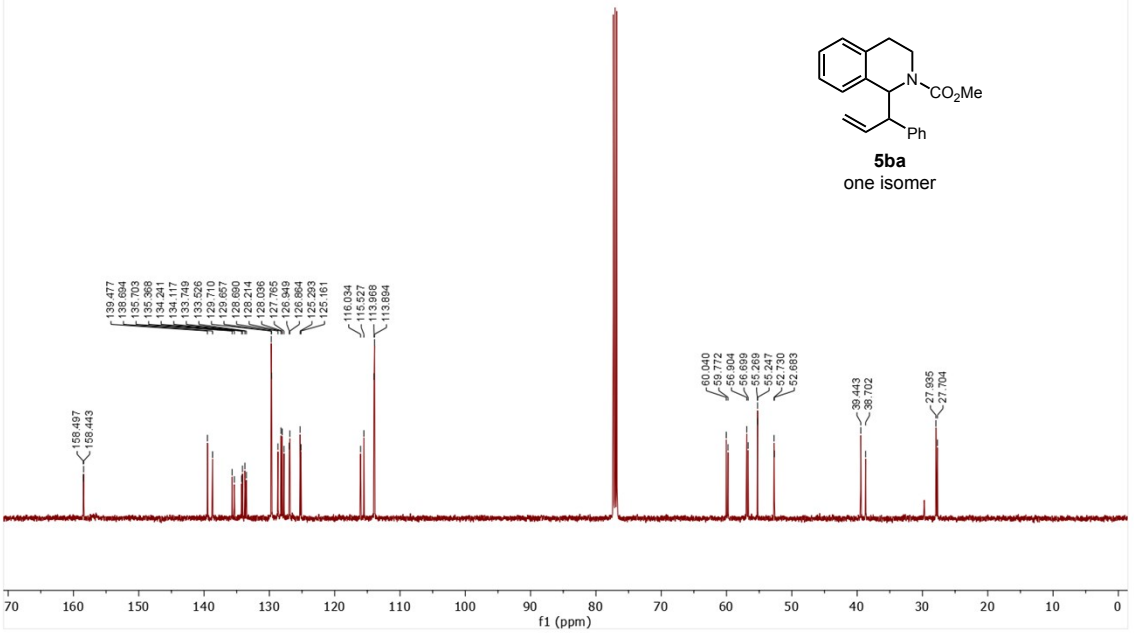
^{1}H NMR (500 MHz, $CDCl_3$)



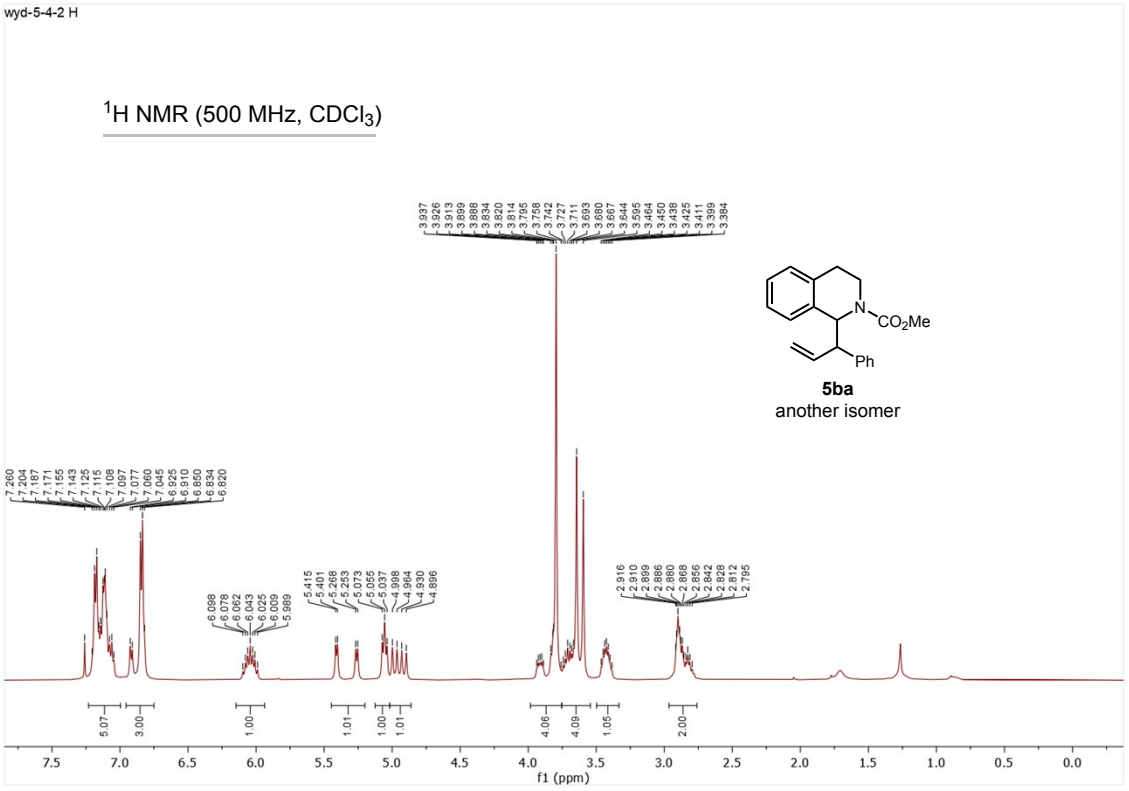
5ba
one isomer

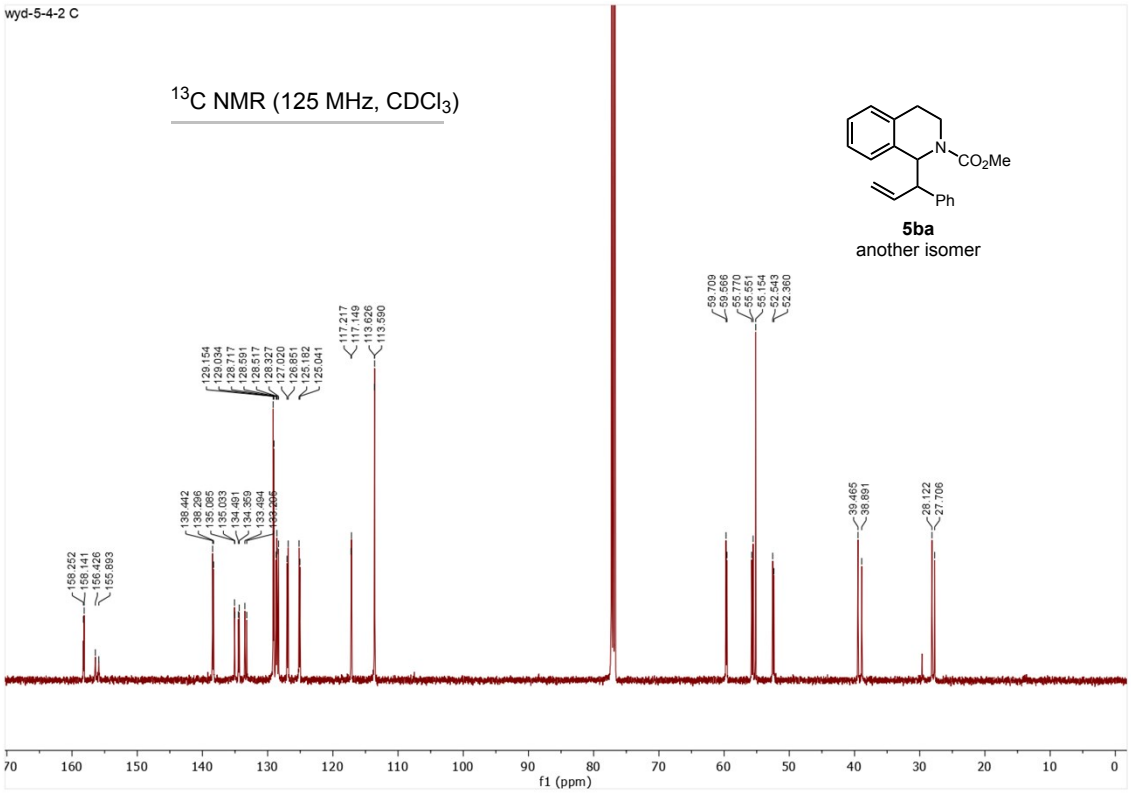
wyd-5-4-1 C

^{13}C NMR (125 MHz, $CDCl_3$)



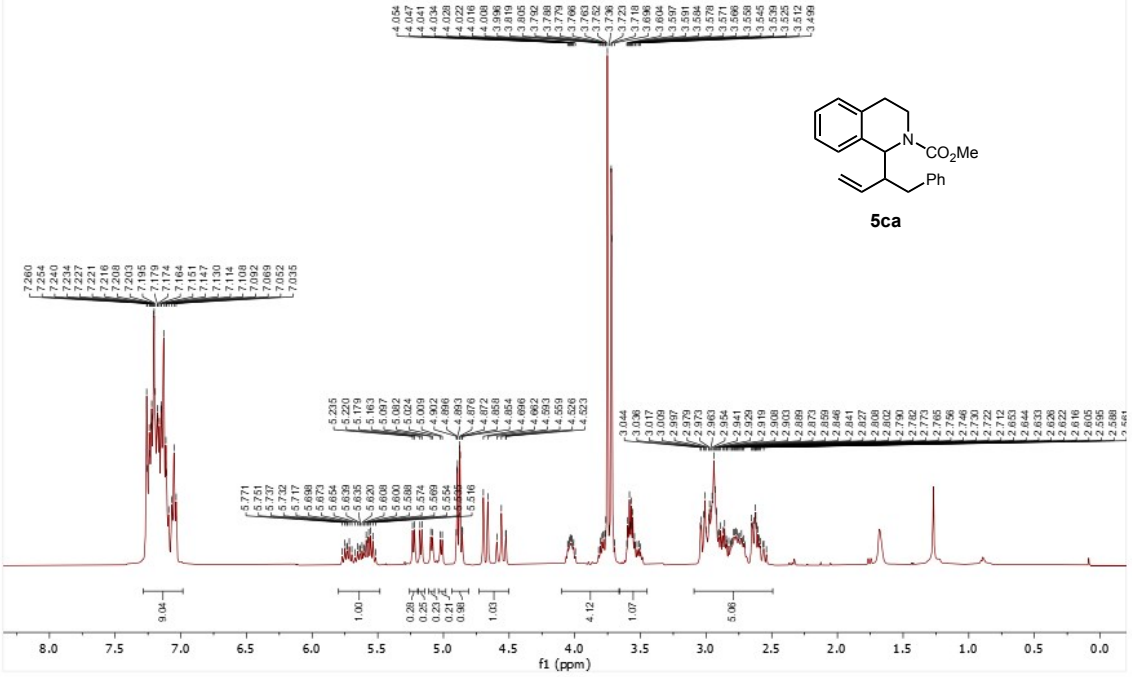
5ba
one isomer


wyd-5-4-2 H
^{1}H NMR (500 MHz, $CDCl_3$)
CO_2Me
Ph
5ba
another isomer
f1 (ppm)


wyd-5-4-2 C
^{13}C NMR (125 MHz, $CDCl_3$)
CO_2Me
Ph
5ba
another isomer
f1 (ppm)

wyd-5-12-1 H

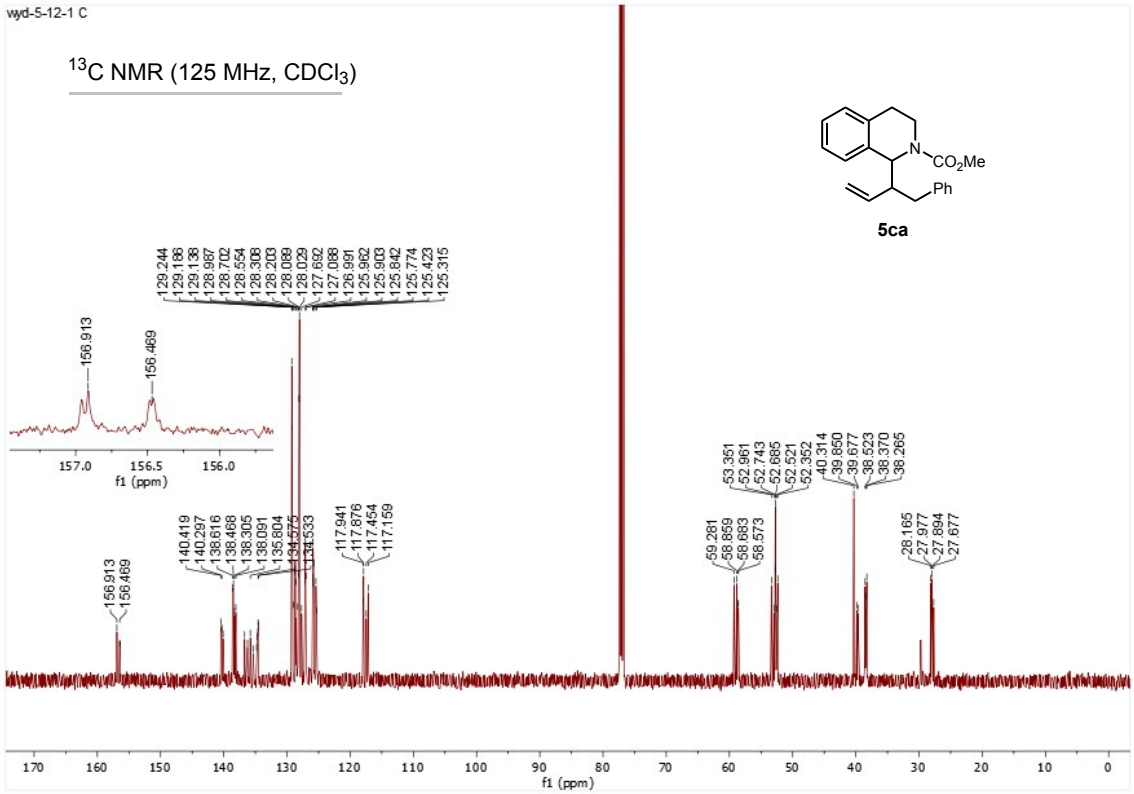
^{1}H NMR (500 MHz, $CDCl_3$)



5ca

wyd-5-12-1 C

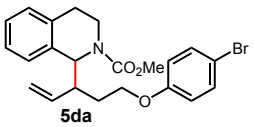
^{13}C NMR (125 MHz, $CDCl_3$)



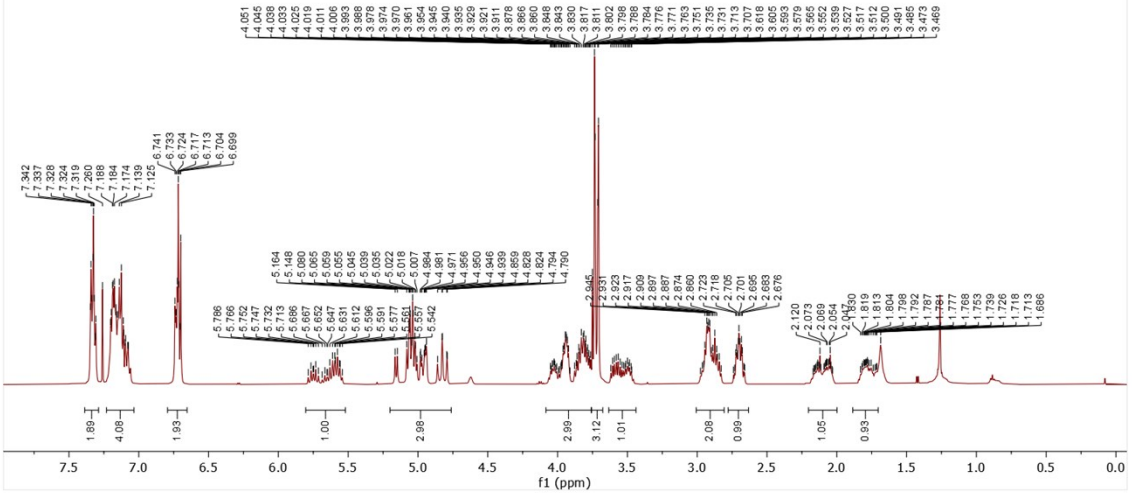
5ca

wyd-5-13-1 H

^{1}H NMR (500 MHz, $CDCl_3$)

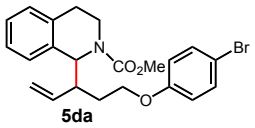


5da

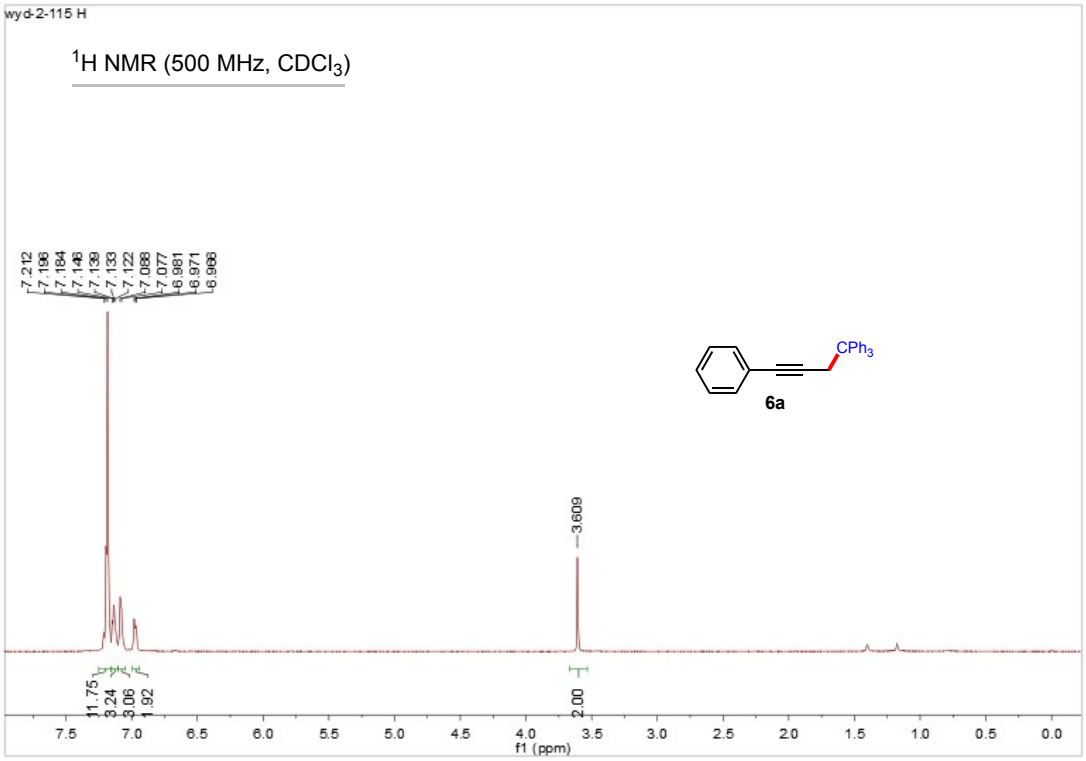


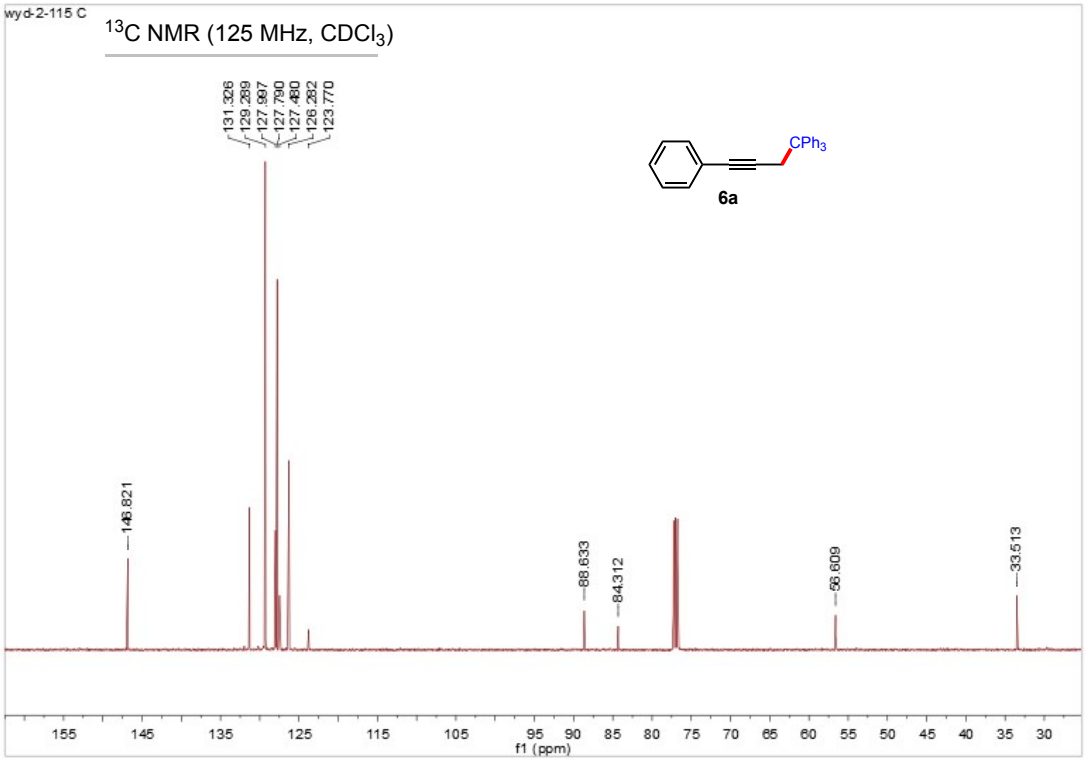
wyd-5-13-1 C

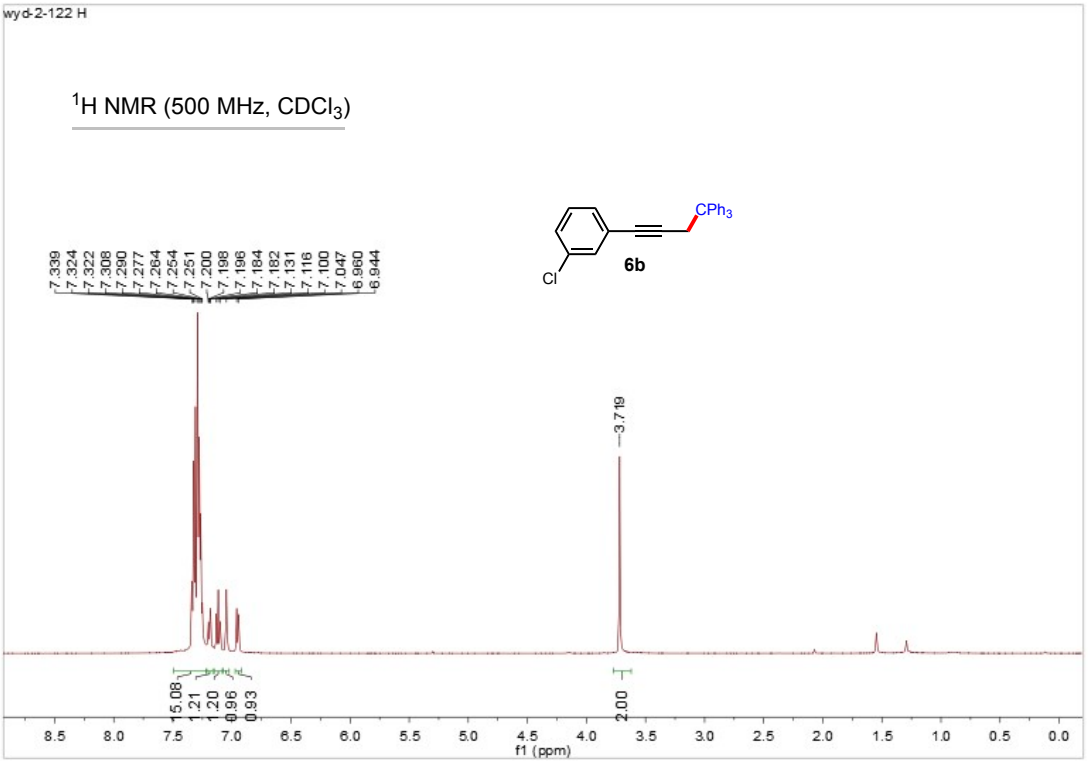
^{13}C NMR (125 MHz, $CDCl_3$)

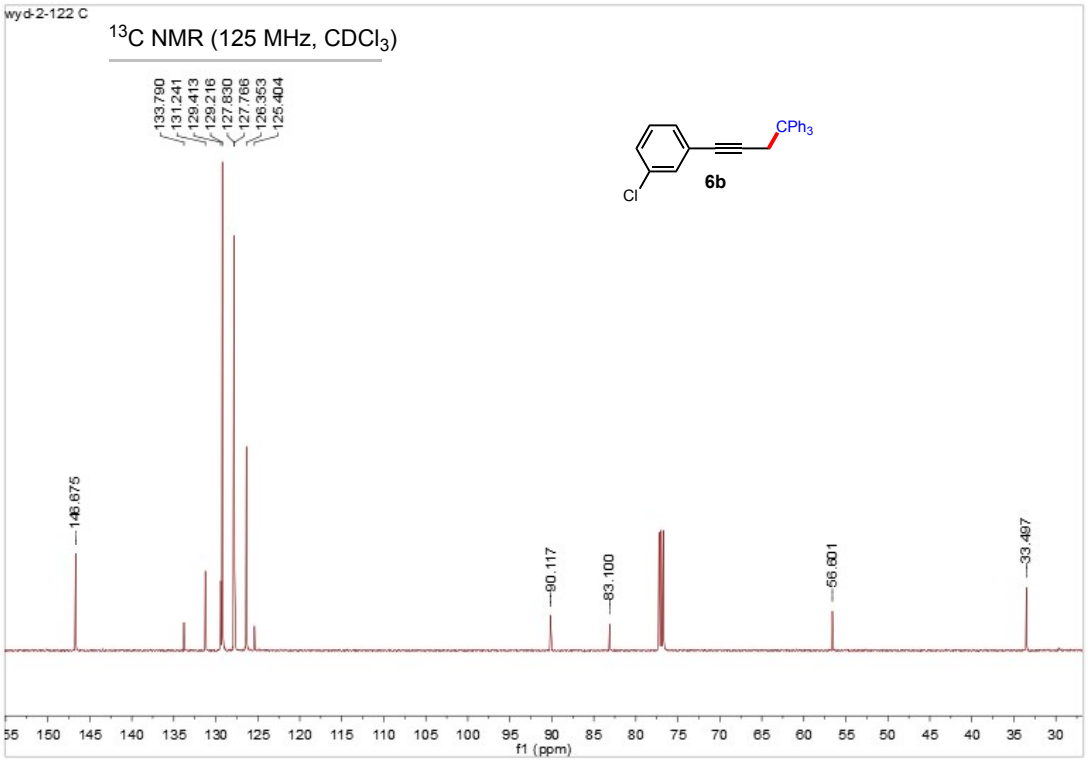


5da


wyd-2-115 H
^{1}H NMR (500 MHz, $CDCl_3$)
7.212
7.198
7.184
7.145
7.139
7.133
7.122
7.088
7.077
6.981
6.971
6.966
3.609
11.75
3.24
3.06
1.92
2.00
CPh3
6a
f1 (ppm)

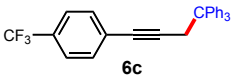

wyd-2-115 C
^{13}C NMR (125 MHz, $CDCl_3$)
131.326
129.299
127.997
127.790
127.490
126.282
123.770
146.821
88.633
84.312
56.609
33.513
CPh3
6a
f1 (ppm)


wyd-2-122 H
^{1}H NMR (500 MHz, $CDCl_3$)
CPh3
Cl
6b
7.339
7.324
7.322
7.308
7.290
7.277
7.264
7.254
7.251
7.200
7.198
7.196
7.184
7.182
7.131
7.116
7.100
7.047
6.960
6.944
3.719
15.08
1.21
1.20
0.96
0.93
2.00
f1 (ppm)

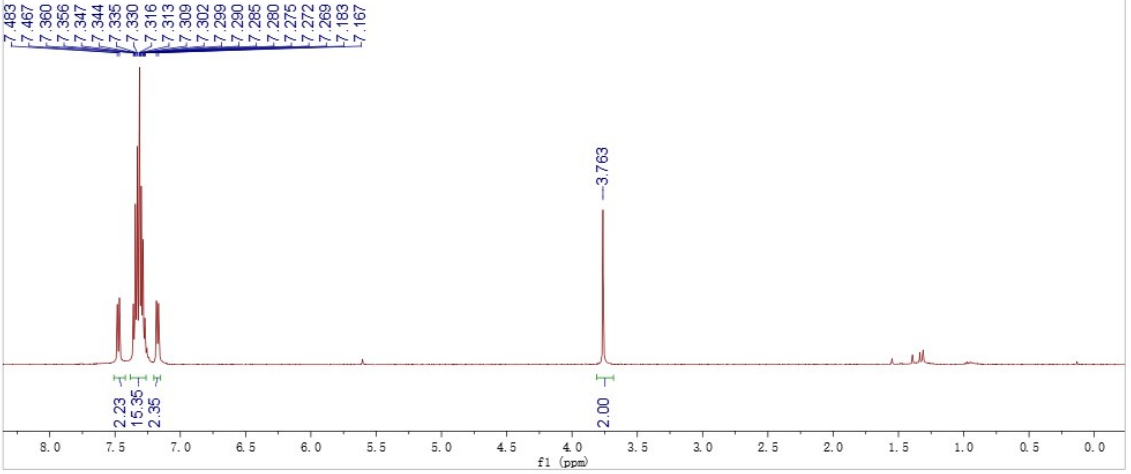

wyd-2-122 C
^{13}C NMR (125 MHz, $CDCl_3$)
CPh3
Cl
6b
133.790
131.241
129.413
129.216
127.830
127.766
126.353
125.404
146.675
90.117
83.100
56.601
33.497
f1 (ppm)

jz-1-54 H

^{1}H NMR (500 MHz, $CDCl_3$)

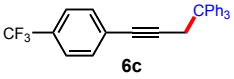


6c



JZ-1-54 C

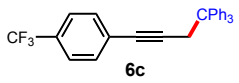
^{13}C NMR (125 MHz, $CDCl_3$)



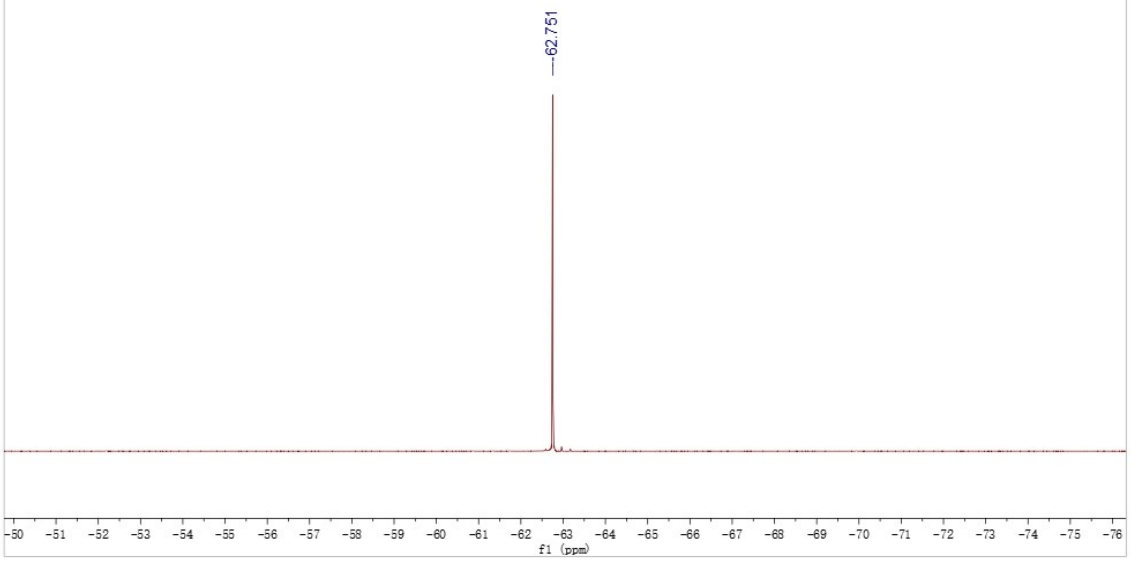
6c

JZ-1-54 F

^{19}F NMR (470 MHz, $CDCl_3$)

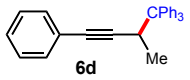


6c

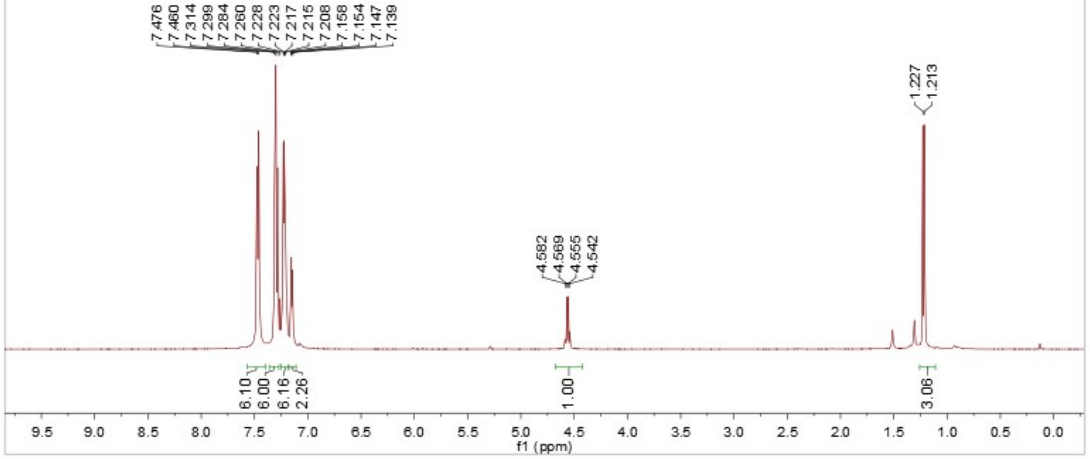


wyd-1-237 H

^{1}H NMR (500 MHz, $CDCl_3$)

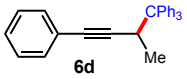


6d

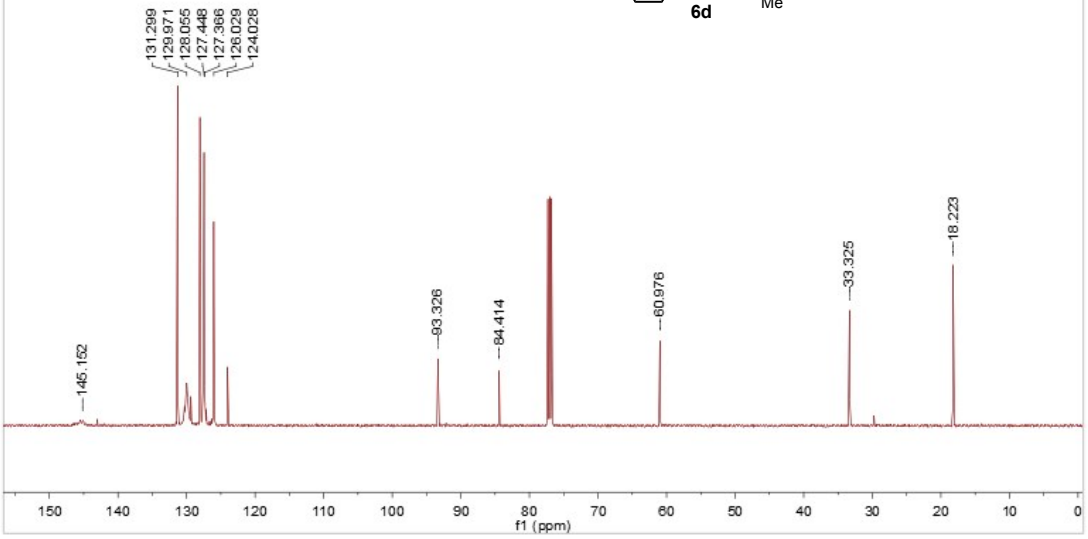


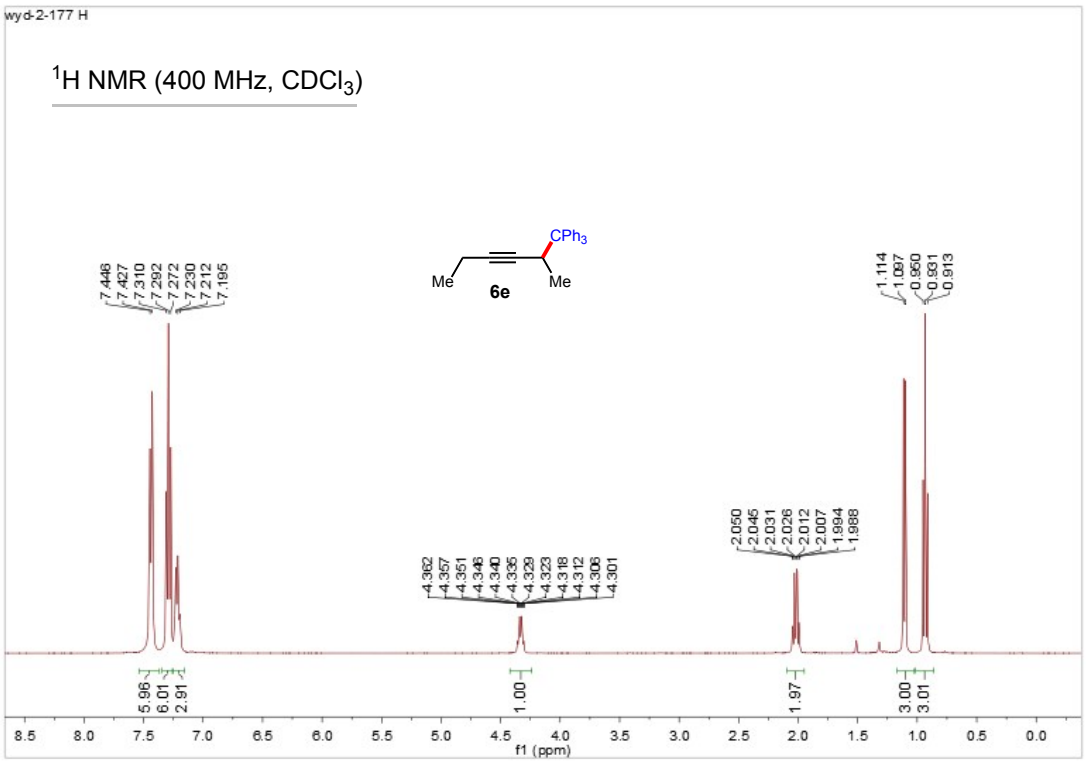
wyd-1-237 C

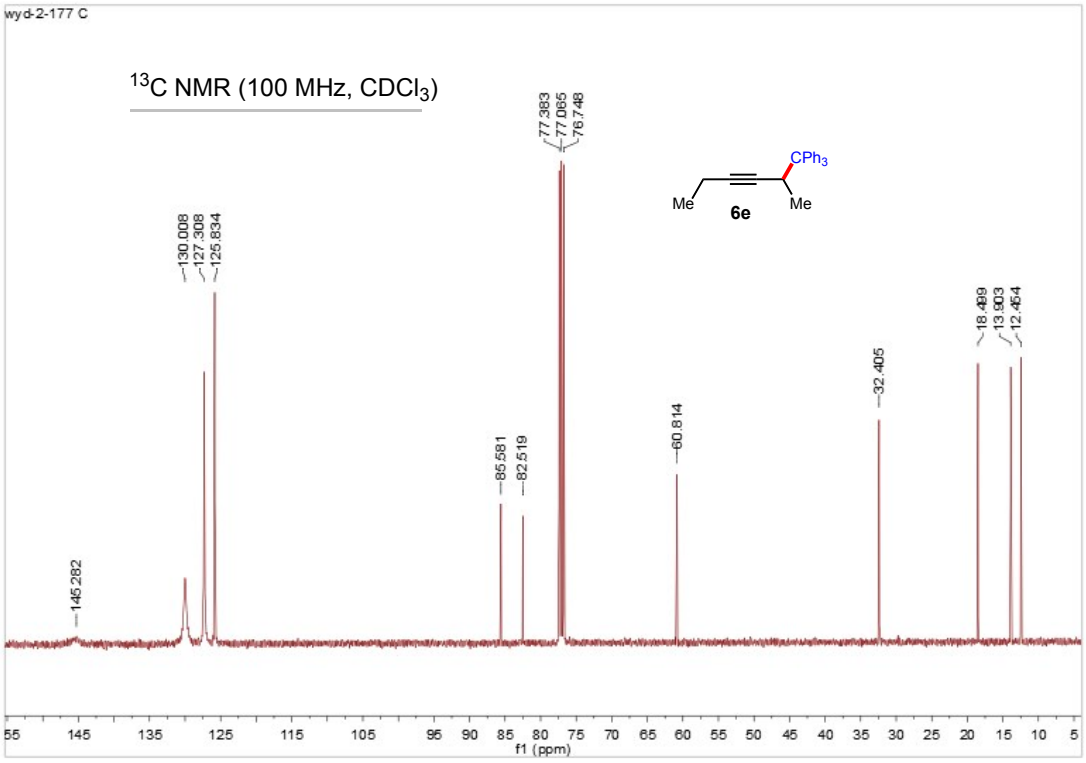
^{13}C NMR (125 MHz, $CDCl_3$)

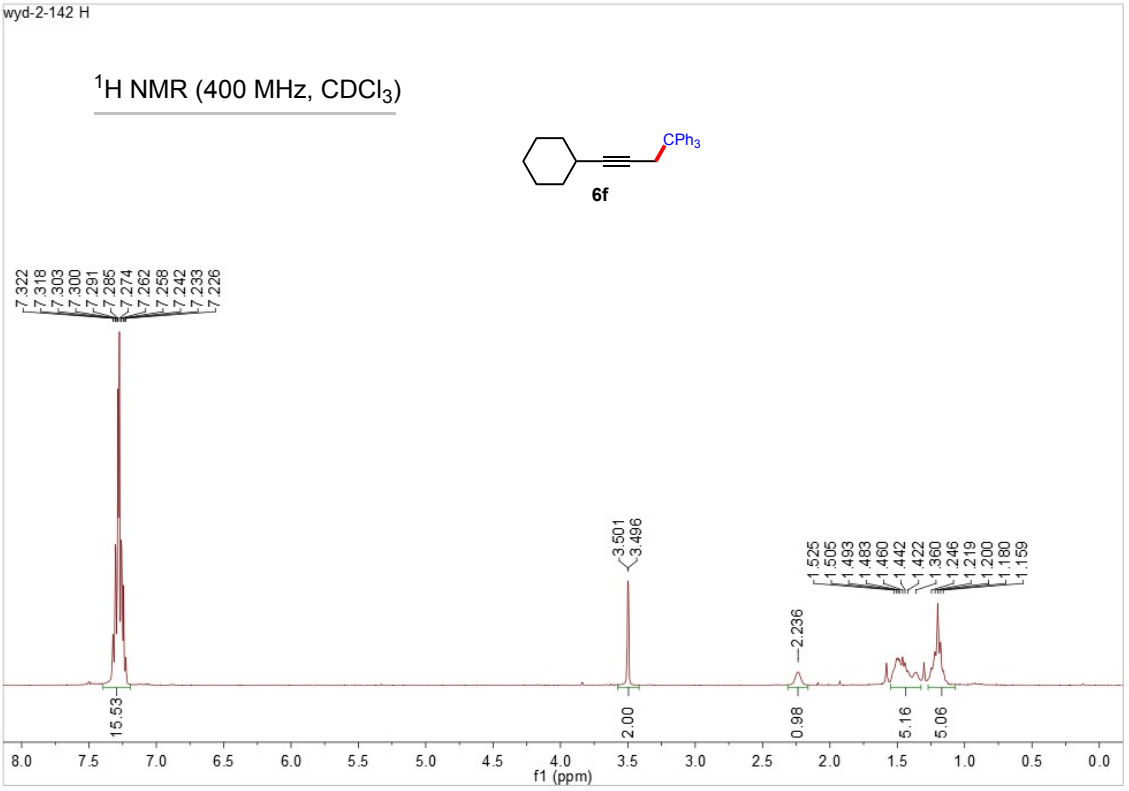


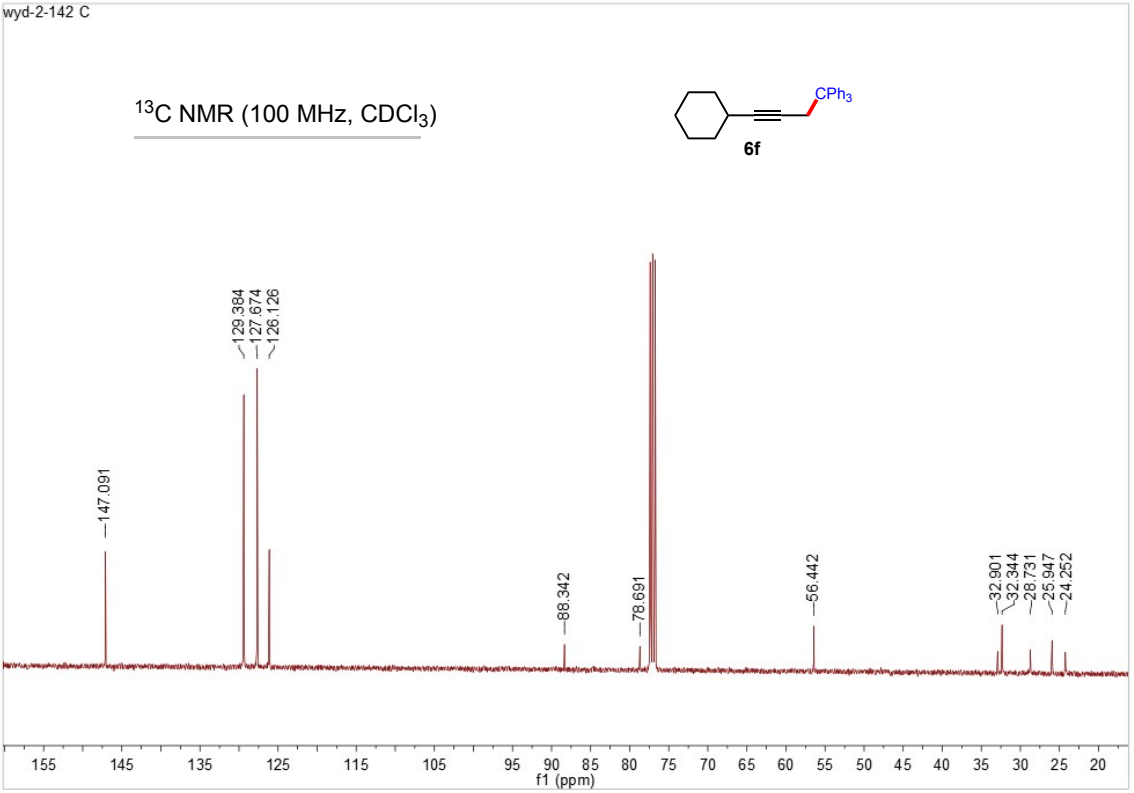
6d

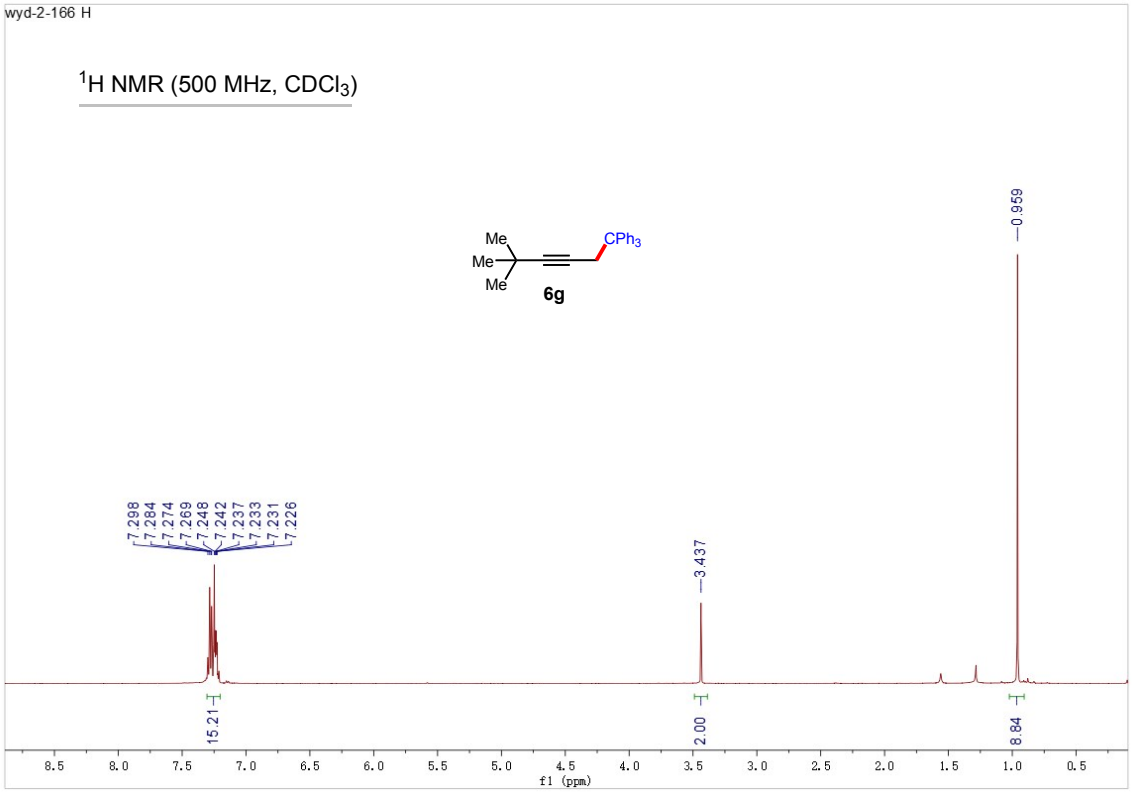


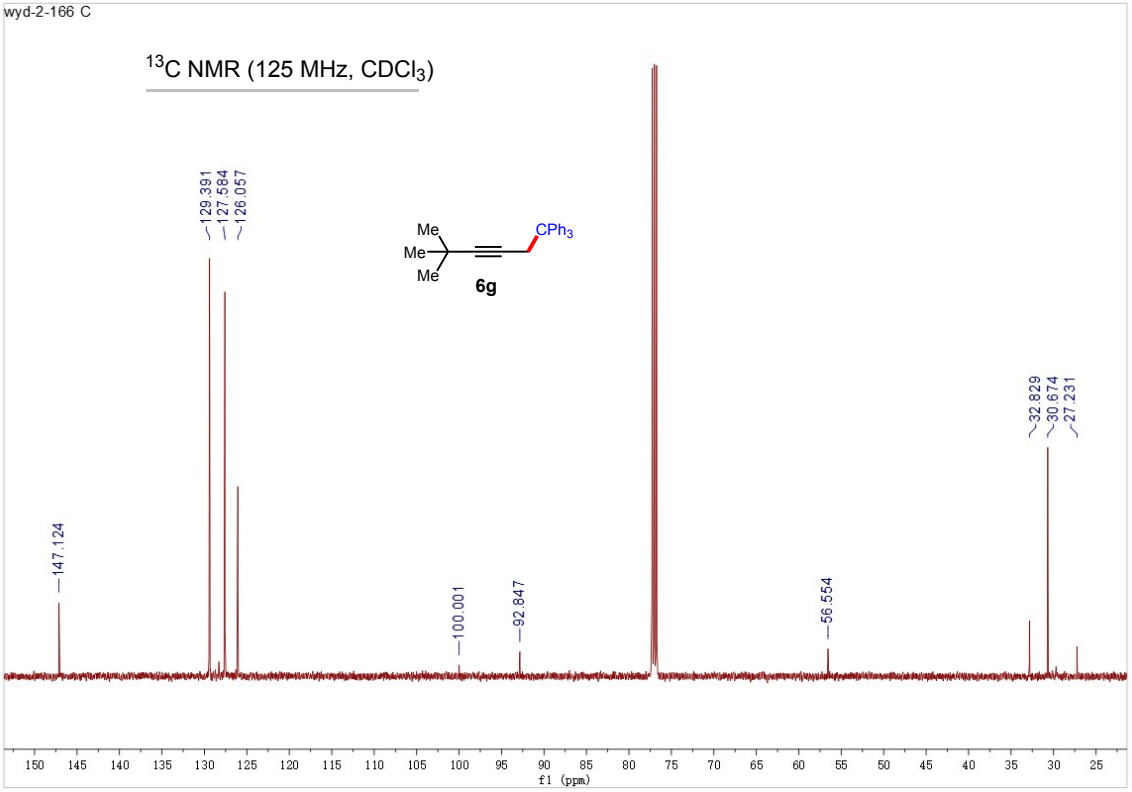

wyd-2-177 H
1H NMR (400 MHz, CDCl3)
CPh3
Me
Me
6e
7.446
7.427
7.310
7.292
7.272
7.230
7.212
7.195
4.362
4.357
4.351
4.346
4.340
4.335
4.329
4.323
4.318
4.312
4.306
4.301
2.050
2.045
2.031
2.026
2.012
2.007
1.994
1.988
1.114
1.097
0.960
0.931
0.913
5.96
6.01
2.91
1.00
1.97
3.00
3.01
f1 (ppm)


wyd-2-177 C
13C NMR (100 MHz, CDCl3)
CPh3
Me
Me
6e
77.383
77.065
76.748
130.008
127.308
125.834
145.282
85.581
82.519
60.814
32.405
18.499
13.903
12.454
f1 (ppm)


wyd-2-142 H
^{1}H NMR (400 MHz, $CDCl_3$)
CPh3
6f
7.322
7.318
7.303
7.300
7.291
7.285
7.274
7.262
7.258
7.242
7.233
7.226
3.501
3.496
2.236
1.525
1.505
1.493
1.483
1.460
1.442
1.422
1.360
1.246
1.219
1.200
1.180
1.159
15.53
2.00
0.98
5.16
5.06
f1 (ppm)

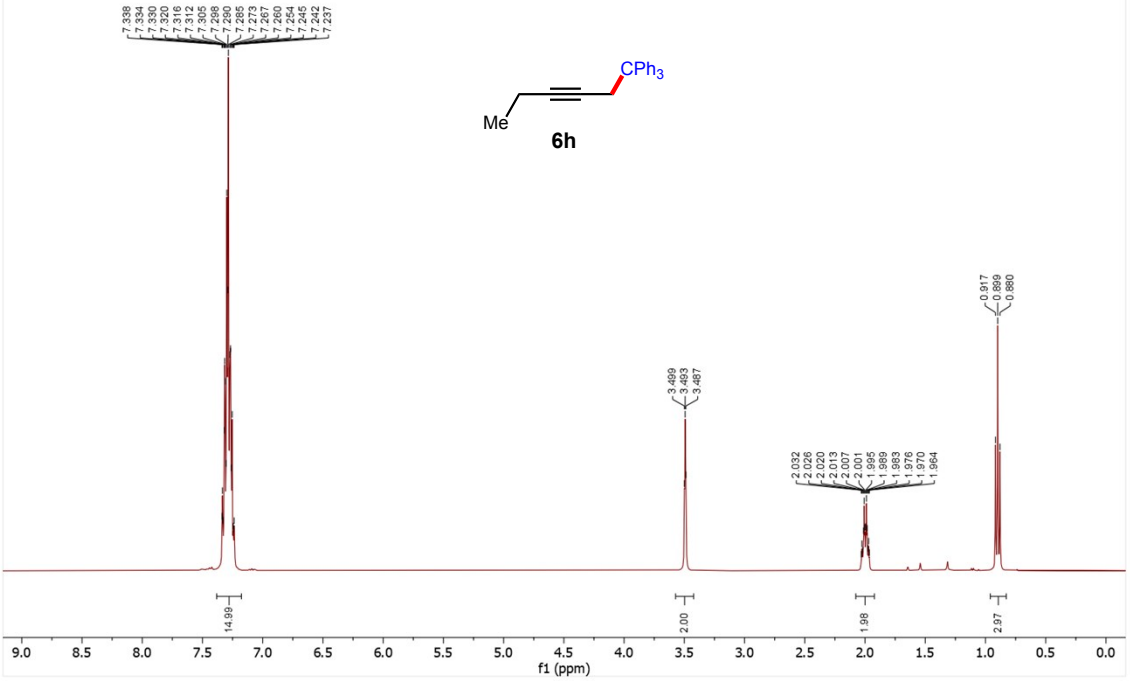

wyd-2-142 C
^{13}C NMR (100 MHz, $CDCl_3$)
CPh3
6f
147.091
129.384
127.674
126.126
88.342
78.691
56.442
32.901
32.344
28.731
25.947
24.252
f1 (ppm)


wyd-2-166 H
1H NMR (500 MHz, CDCl3)
Me
Me
Me
CPh3
6g
0.959
7.298
7.284
7.274
7.269
7.248
7.242
7.237
7.233
7.231
7.226
3.437
15.21
2.00
8.84
f1 (ppm)


wyd-2-166 C
13C NMR (125 MHz, CDCl3)
Me
Me
Me
CPh3
6g
129.391
127.584
126.057
147.124
100.001
92.847
56.554
32.829
30.674
27.231
f1 (ppm)

WYD-2-179-1 H

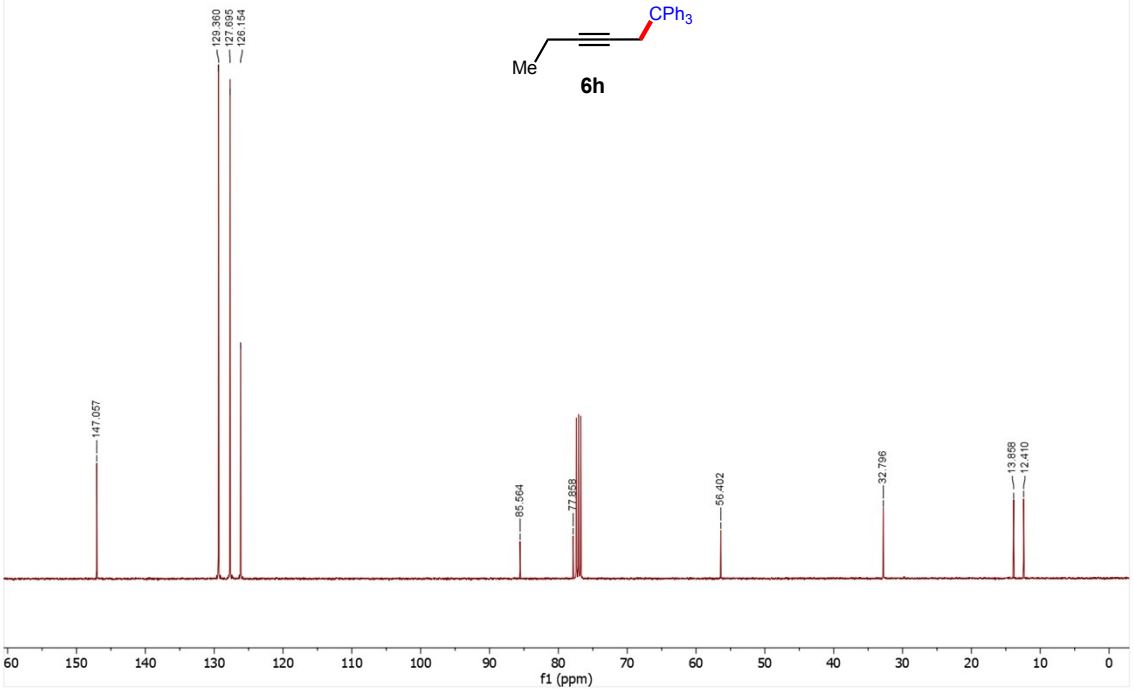
^{1}H NMR (400 MHz, $CDCl_3$)



6h

WYD-2-179-1 C

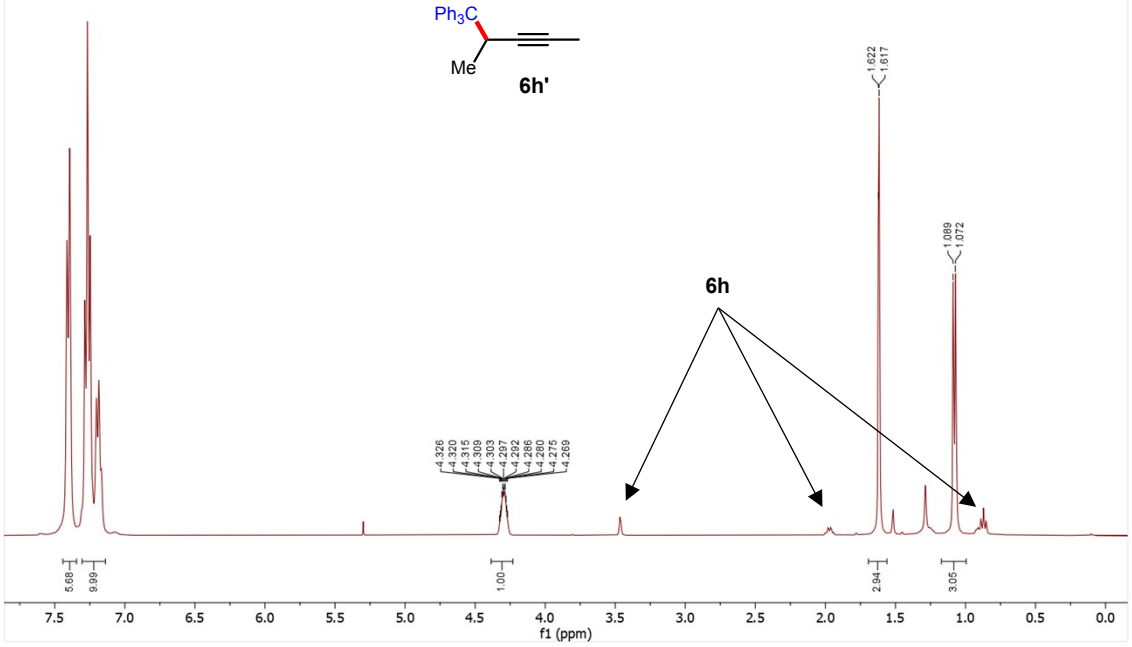
^{13}C NMR (100 MHz, $CDCl_3$)



6h

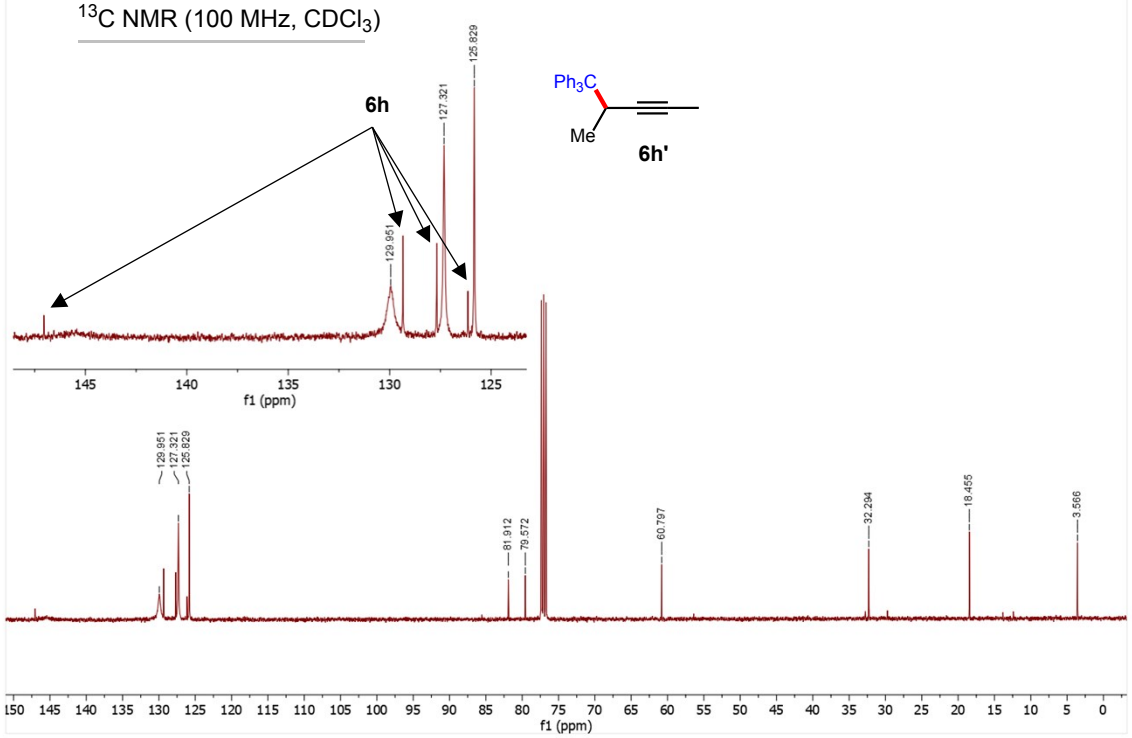
wyd-2-179-2 H

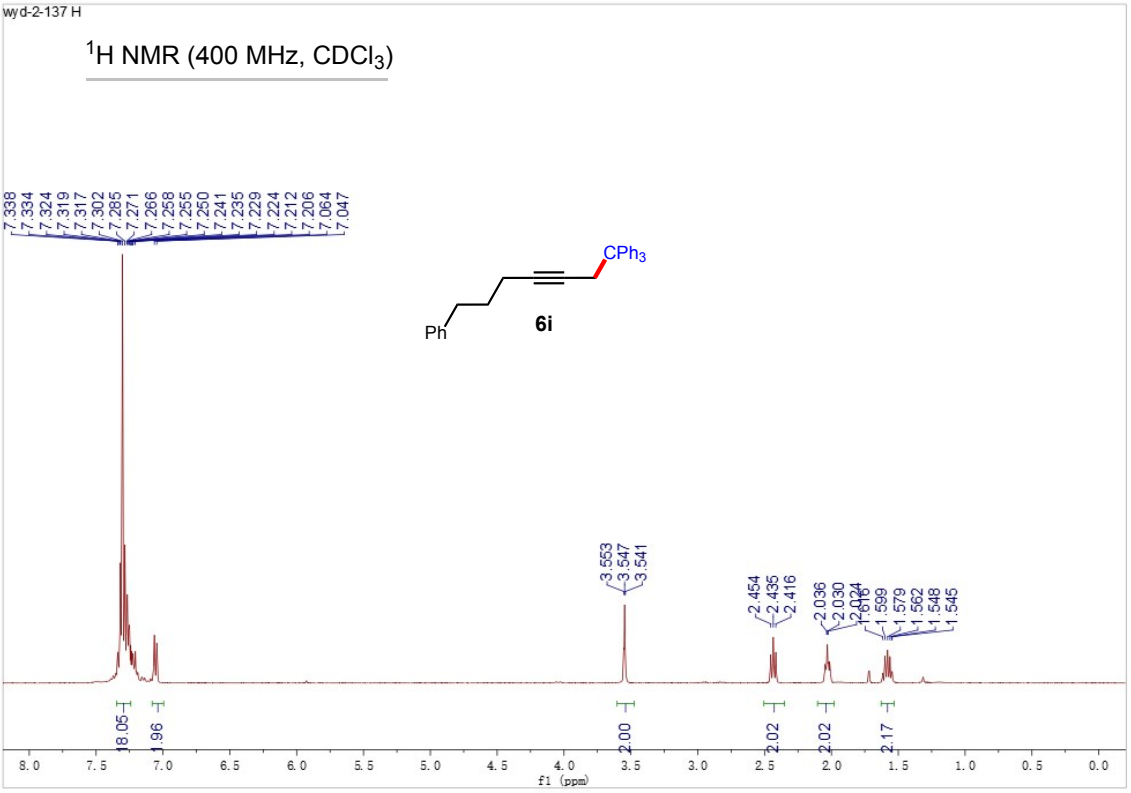
^{1}H NMR (400 MHz, $CDCl_3$)

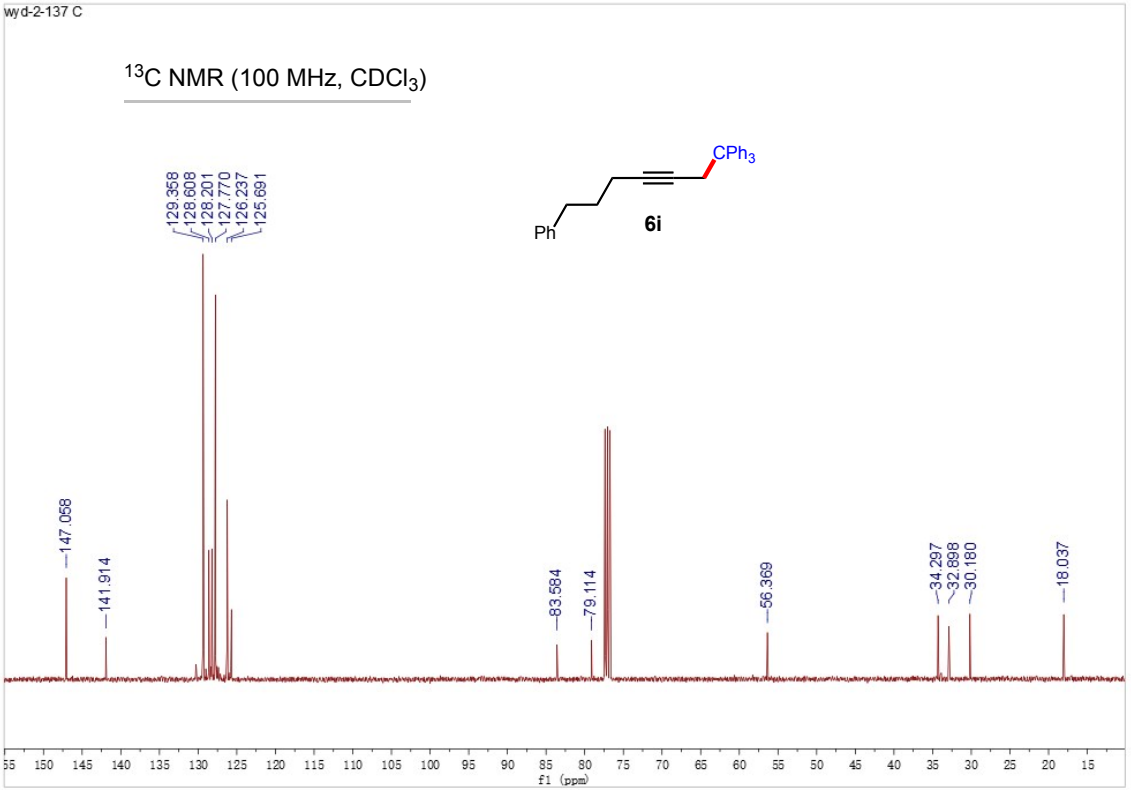


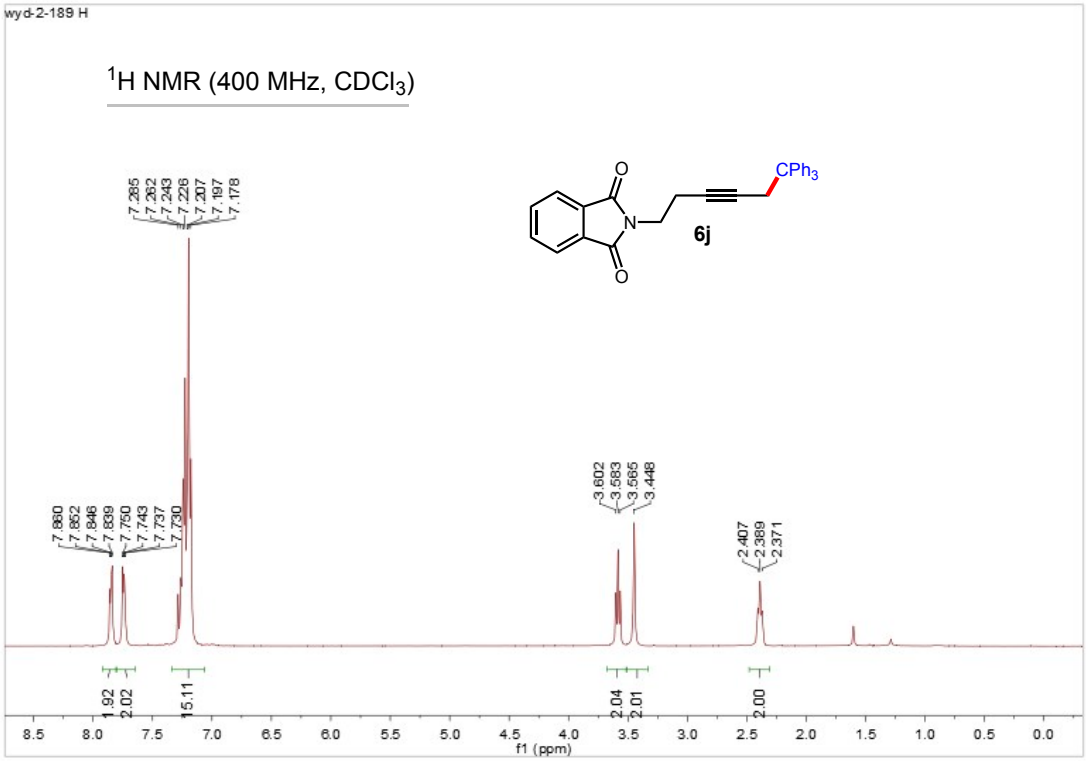
wyd-2-179-2 C

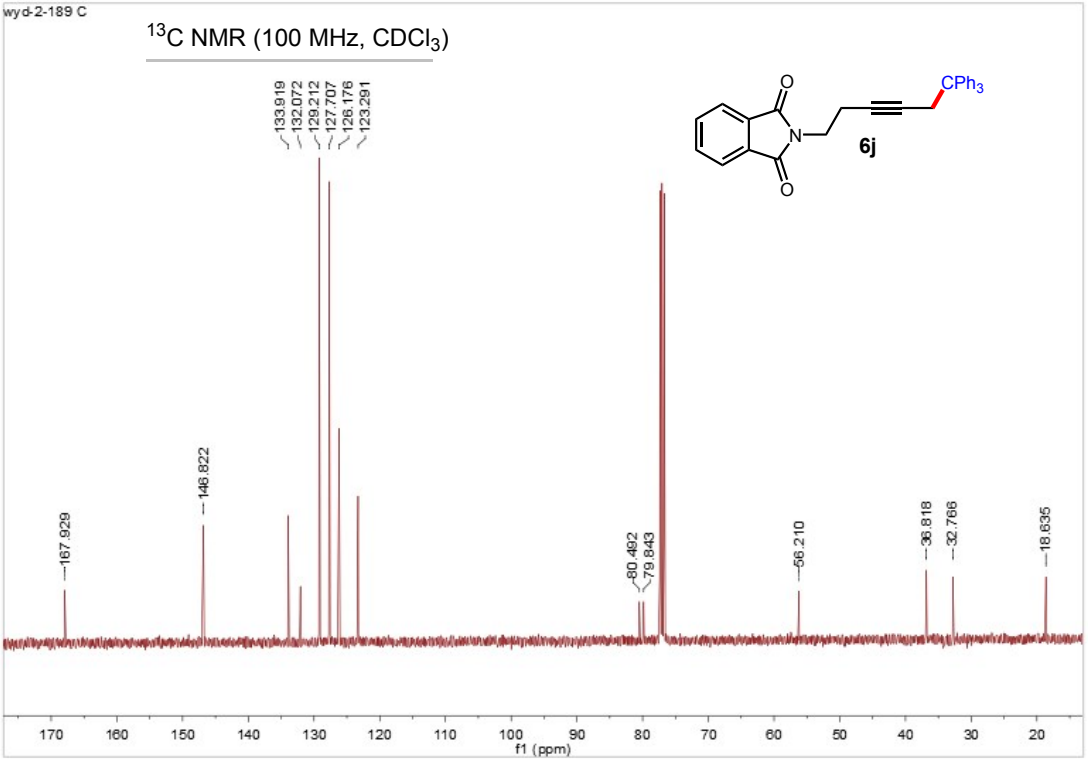
^{13}C NMR (100 MHz, $CDCl_3$)




wyd-2-137 H
1H NMR (400 MHz, CDCl3)
7.338
7.334
7.324
7.319
7.317
7.302
7.285
7.271
7.266
7.258
7.255
7.250
7.241
7.235
7.229
7.224
7.212
7.206
7.064
7.047
CPh3
Ph
6i
3.553
3.547
3.541
2.454
2.435
2.416
2.036
2.030
2.024
2.016
1.599
1.579
1.562
1.548
1.545
18.05
1.96
2.00
2.02
2.02
2.17
f1 (ppm)

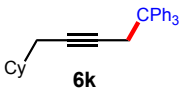

wyd-2-137 C
13C NMR (100 MHz, CDCl3)
129.358
128.608
128.201
127.770
126.237
125.691
CPh3
Ph
6i
147.058
141.914
83.584
79.114
56.369
34.297
32.898
30.180
18.037
f1 (ppm)


wyd-2-189 H
^{1}H NMR (400 MHz, $CDCl_3$)
CPh3
6j
7.885
7.862
7.846
7.839
7.750
7.743
7.737
7.730
7.285
7.262
7.243
7.228
7.207
7.197
7.178
3.602
3.583
3.565
3.448
2.407
2.389
2.371
1.92
2.02
15.11
2.04
2.01
2.00
f1 (ppm)

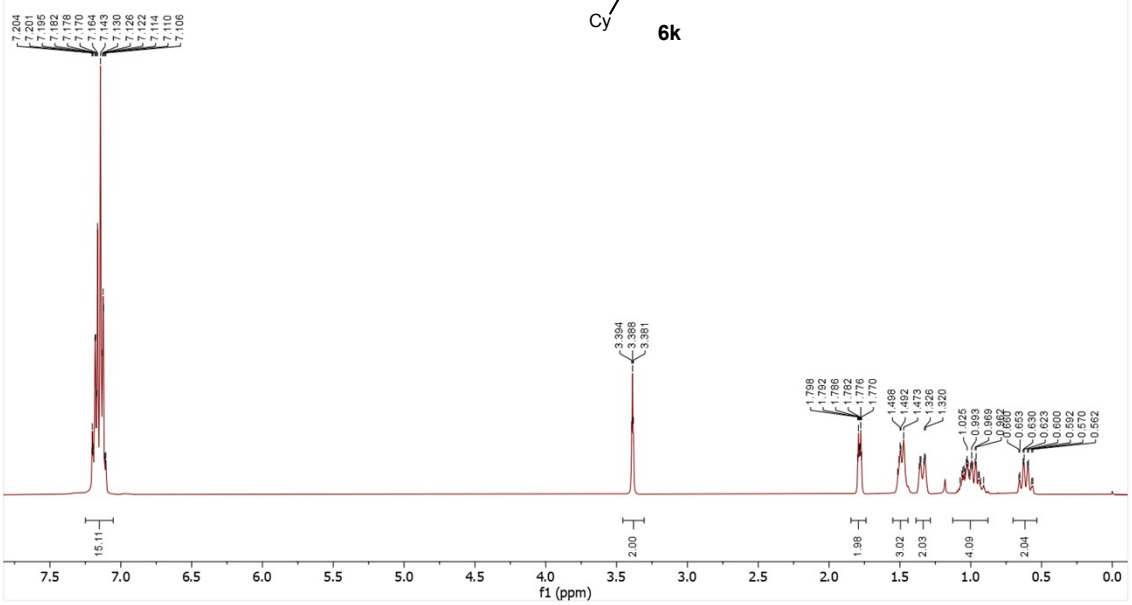

wyd-2-189 C
^{13}C NMR (100 MHz, $CDCl_3$)
CPh3
6j
133.919
132.072
129.212
127.707
126.176
123.291
167.929
146.822
80.492
79.843
56.210
36.818
32.766
18.635
f1 (ppm)

wyd-2-158 H

^{1}H NMR (400 MHz, $CDCl_3$)

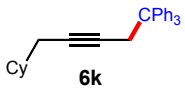


6k

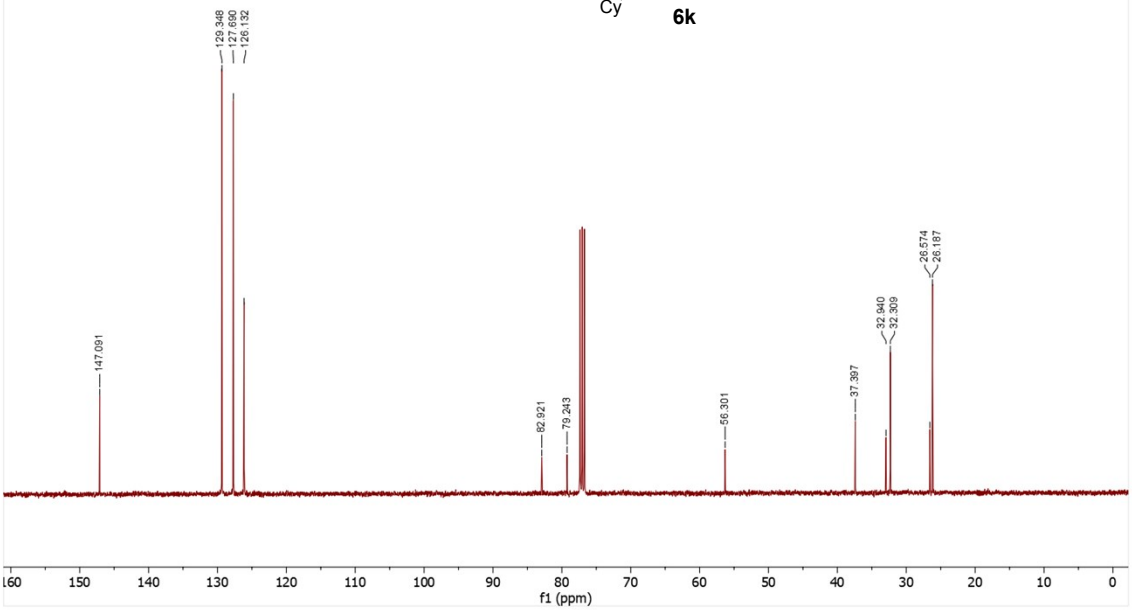


wyd-2-158 C

^{13}C NMR (100 MHz, $CDCl_3$)

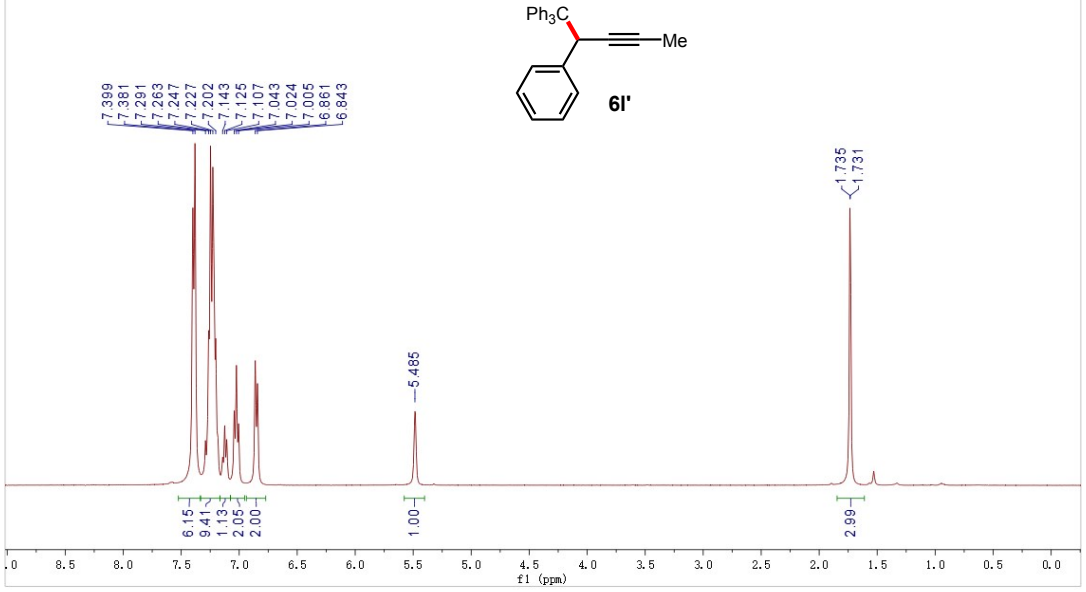


6k



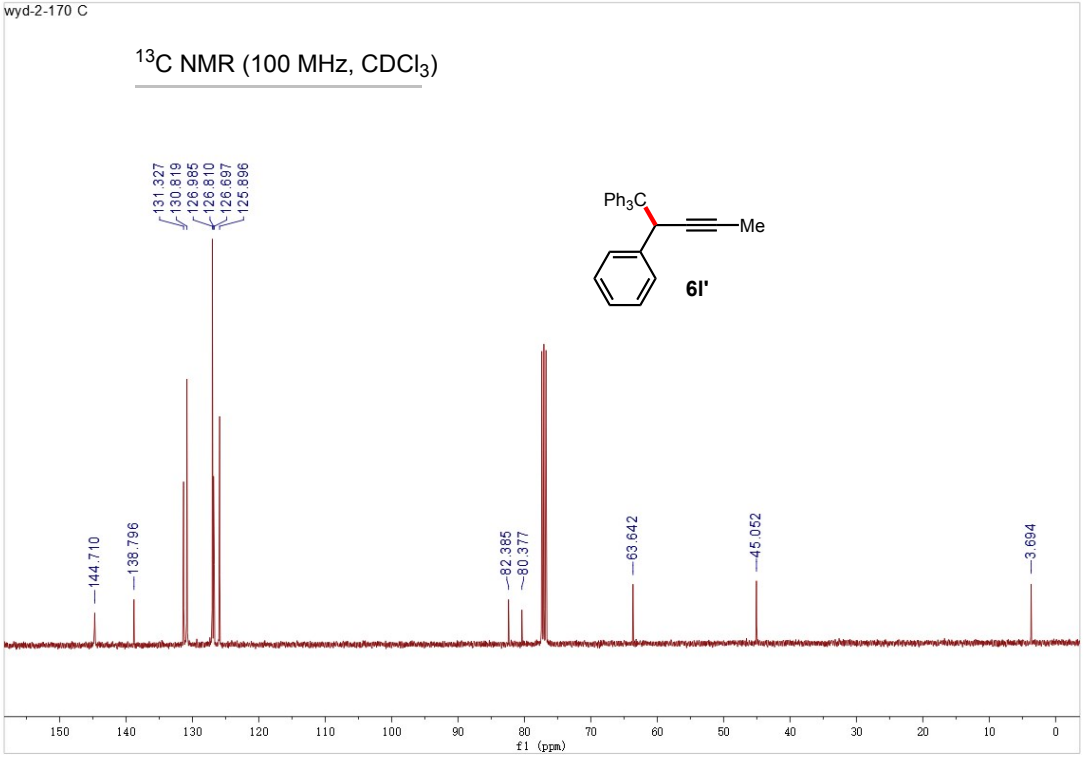
wyd-2-170 H

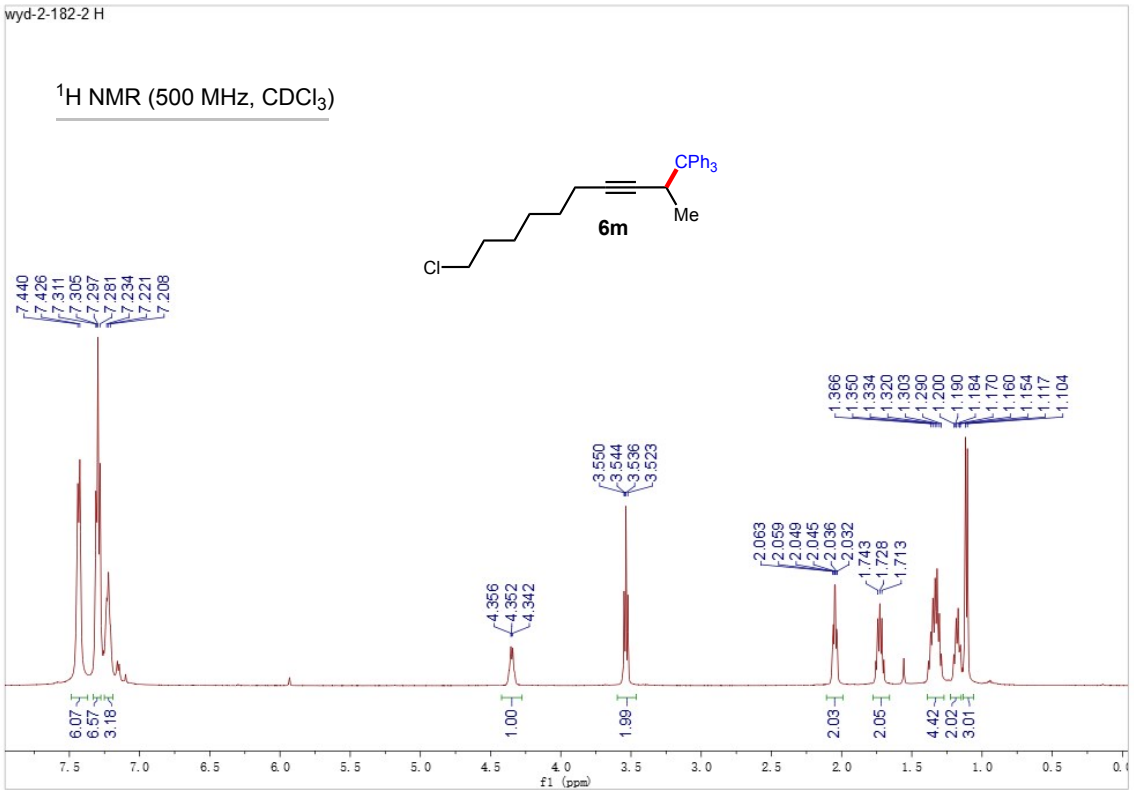
^{1}H NMR (400 MHz, $CDCl_3$)

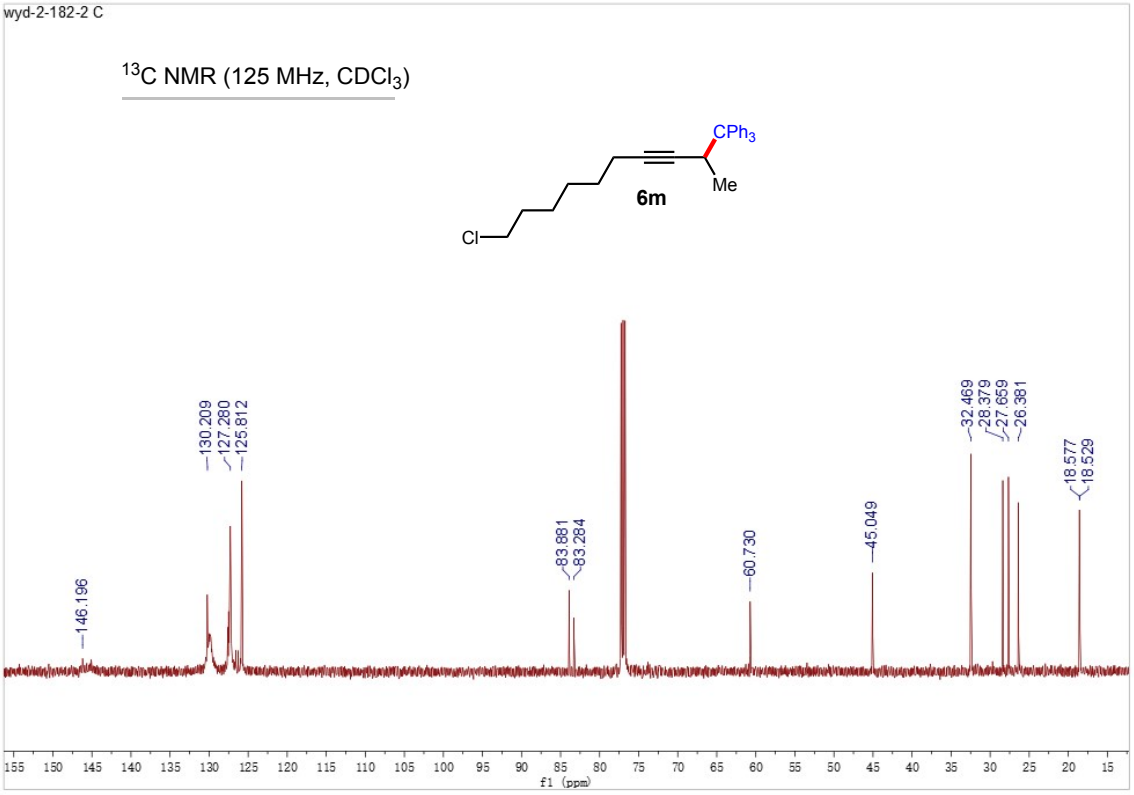


wyd-2-170 C

^{13}C NMR (100 MHz, $CDCl_3$)

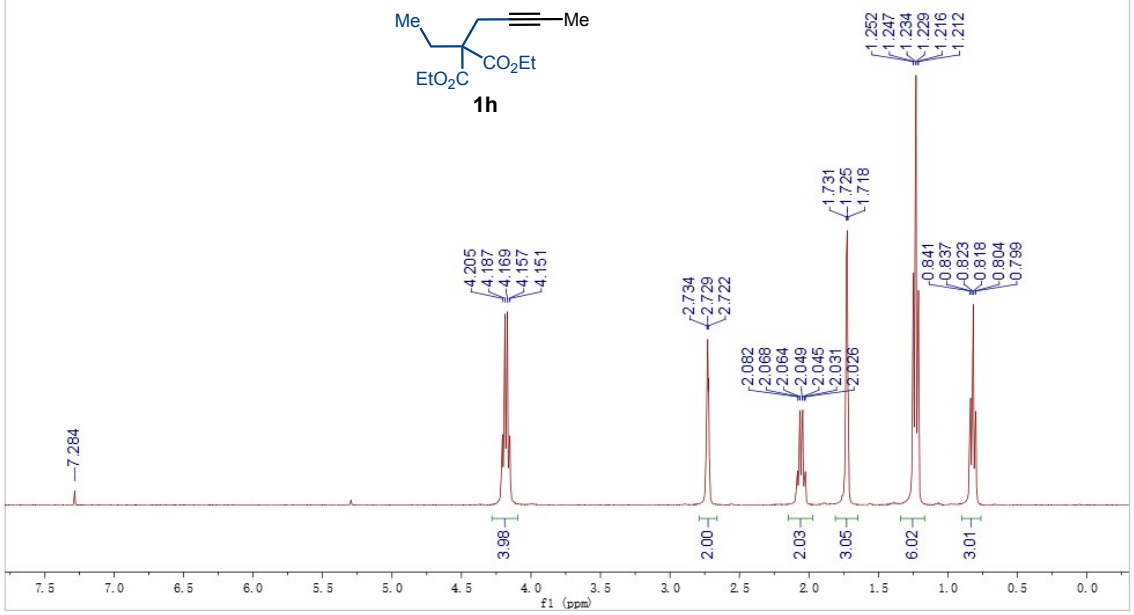



wyd-2-182-2 H
^{1}H NMR (500 MHz, $CDCl_3$)
CPh3
Me
6m
Cl
7.440
7.426
7.311
7.305
7.297
7.281
7.234
7.221
7.208
4.356
4.352
4.342
3.550
3.544
3.536
3.523
2.063
2.059
2.049
2.045
2.036
2.032
1.743
1.728
1.713
1.366
1.350
1.334
1.320
1.303
1.290
1.200
1.190
1.184
1.170
1.160
1.154
1.117
1.104
6.07
6.57
3.18
1.00
1.99
2.03
2.05
4.42
2.02
3.01
f1 (ppm)


wyd-2-182-2 C
^{13}C NMR (125 MHz, $CDCl_3$)
CPh3
Me
6m
Cl
146.196
130.209
127.280
125.812
83.881
83.284
60.730
45.049
32.469
28.379
27.659
26.381
18.577
18.529
f1 (ppm)

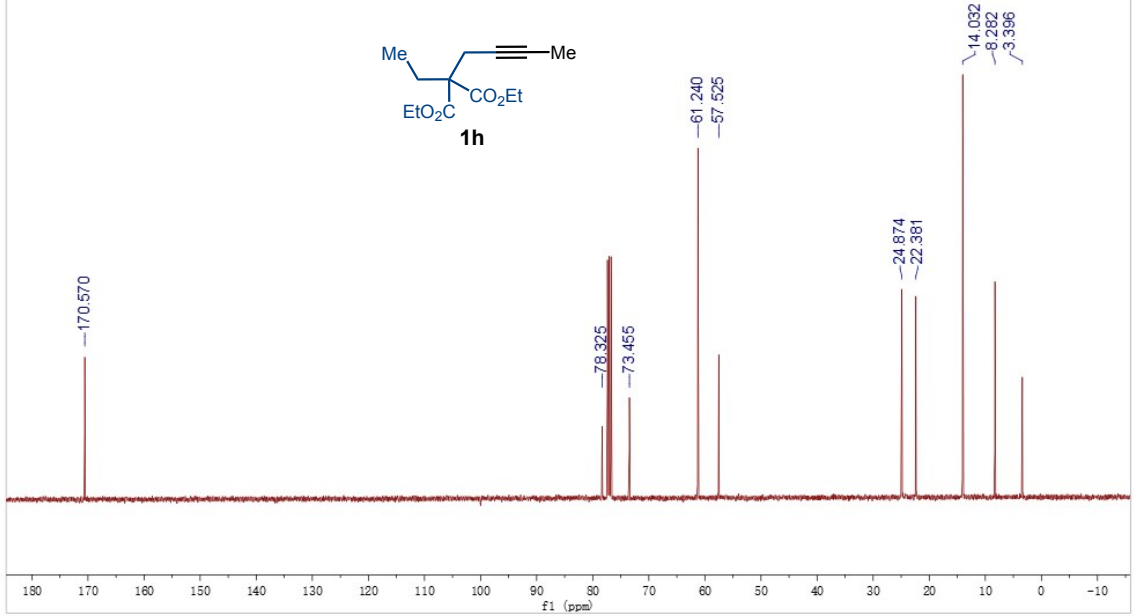
wyd-2-195 H

^{1}H NMR (400 MHz, $CDCl_3$)



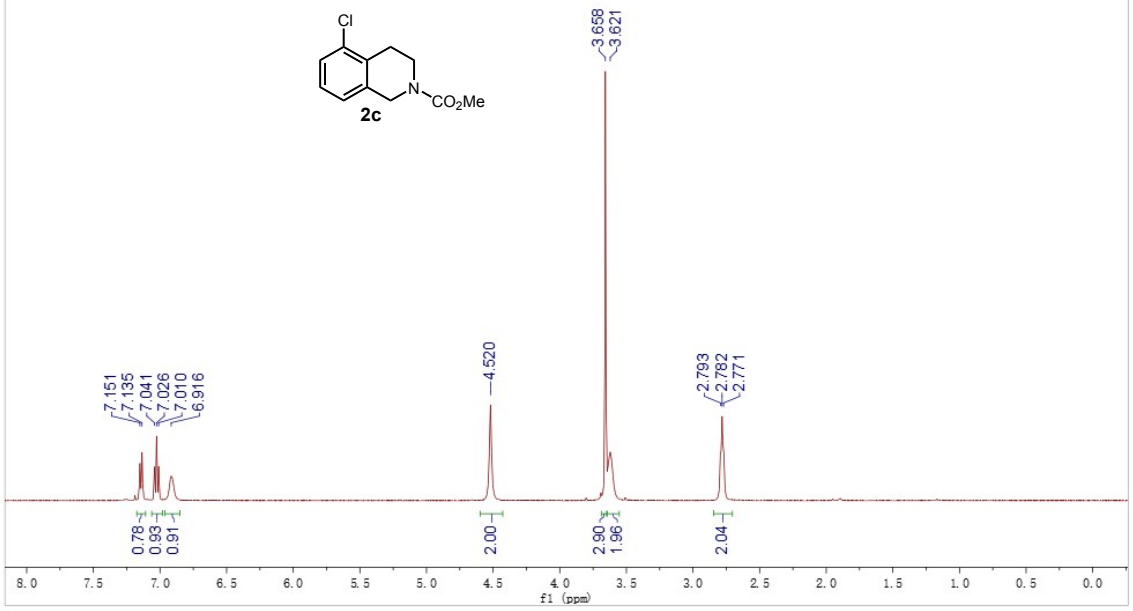
wyd-2-195 C

^{13}C NMR (100 MHz, $CDCl_3$)



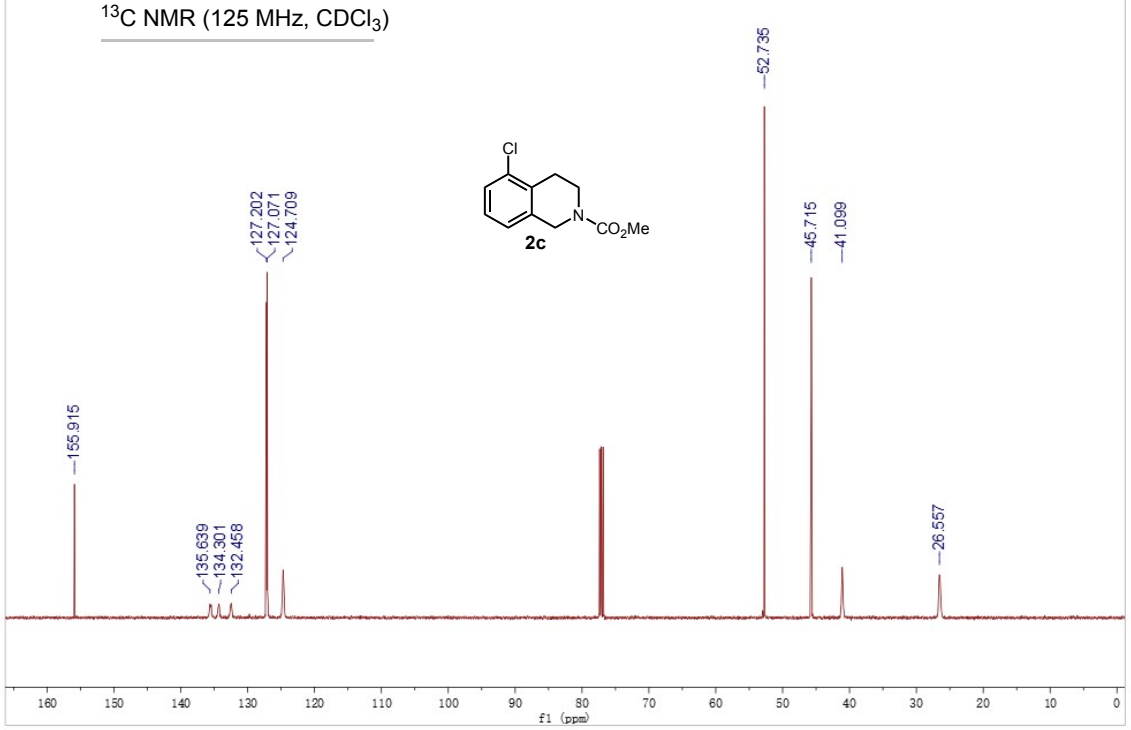
wyd-2-280 H

^{1}H NMR (500 MHz, $CDCl_3$)



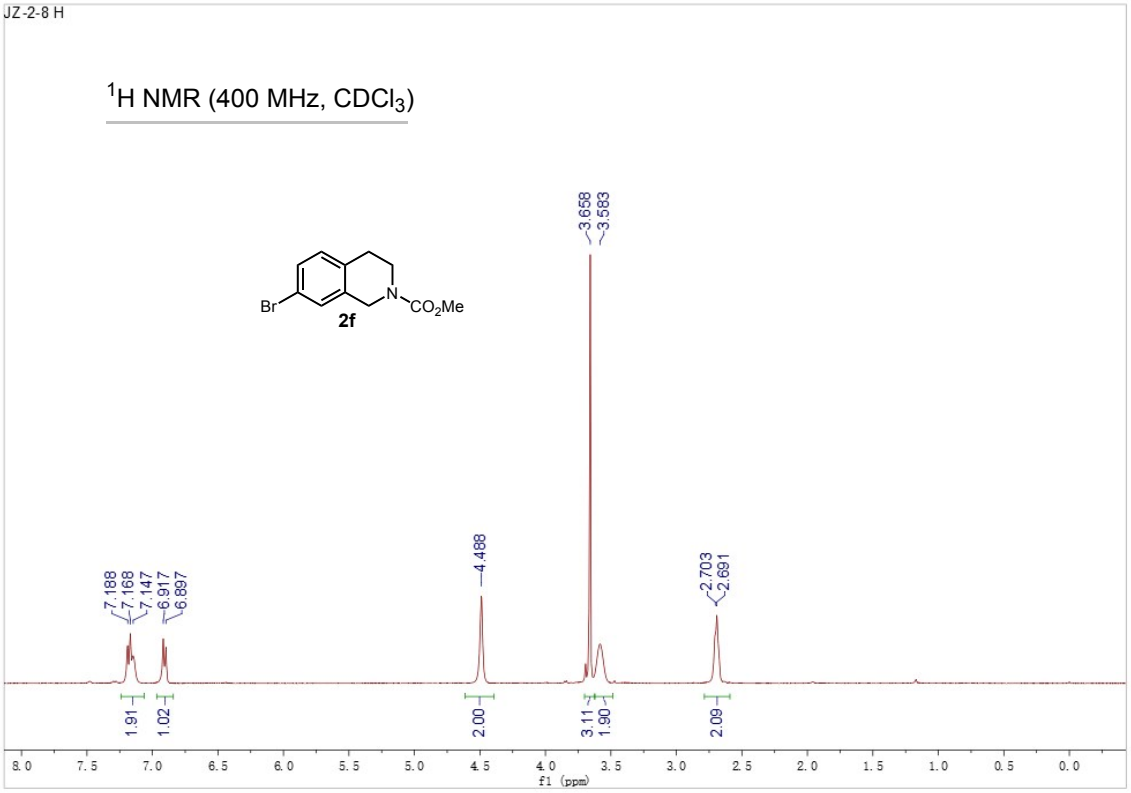
wyd-2-280 C

^{13}C NMR (125 MHz, $CDCl_3$)



JZ-2-8 H

^{1}H NMR (400 MHz, $CDCl_3$)



JZ-2-8 C

^{13}C NMR (100 MHz, $CDCl_3$)