

Supporting Information for

Cu-Catalyzed Stereoconvergent and Enantioselective C–S Cross-Coupling of Alkenyl Halides with Sulfenamides via Alkenyl Radicals

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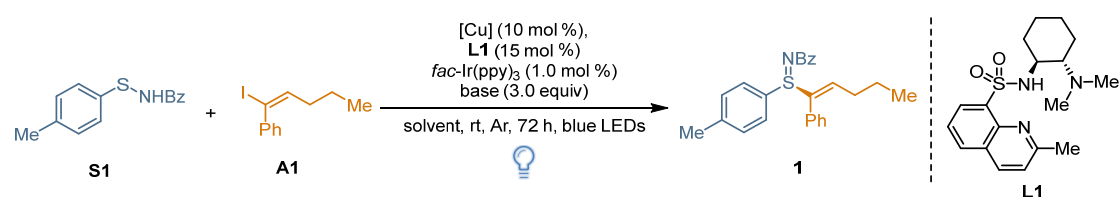
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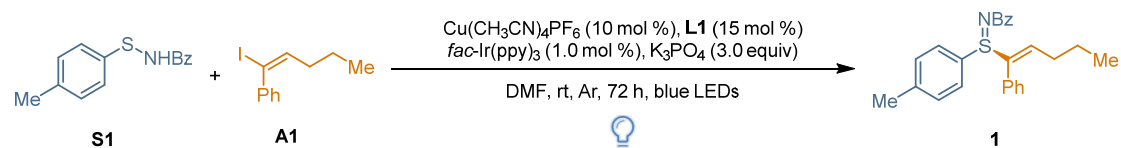
1. Supplementary tables for experiments

Table S1. Reaction condition optimization with sulfenamide **S1** and alkenyl iodide **A1**^a



Entry	[Cu]	L*	Base	Solvent	Yield (%)	Ee (%)
1	Cu(CH ₃ CN) ₄ PF ₆	L1	K ₃ PO ₄	DCM	21	94
2	Cu(CH ₃ CN) ₄ PF ₆	L1	K ₃ PO ₄	CH ₃ CN	10	93
3	Cu(CH ₃ CN) ₄ PF ₆	L1	K ₃ PO ₄	PhCF ₃	15	94
4	CuI	L1	K ₃ PO ₄	DMF	76	94
5	CuBr	L1	K ₃ PO ₄	DMF	72	94
6	CuTc	L1	K ₃ PO ₄	DMF	70	94
7	Cu(OAc) ₂	L1	K ₃ PO ₄	DMF	63	94
8	Cu(CH ₃ CN) ₄ PF ₆	L1	Li ₃ PO ₄	DMF	trace	—
9	Cu(CH ₃ CN) ₄ PF ₆	L1	Cs ₂ CO ₃	DMF	trace	—

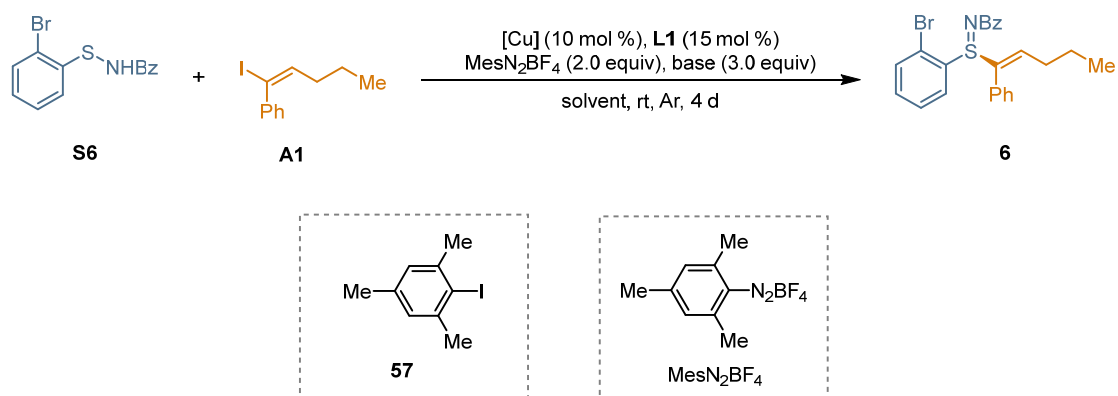
^aReaction conditions: **S1** (0.20 mmol, 1.0 equiv), **A1** (1.5 equiv), [Cu] (10 mol %), **L1** (15 mol %), *fac*-Ir(ppy)₃ (1.0 mol %) and base (3.0 equiv) in solvent (2.0 mL) stirred at rt and blue LEDs irradiation for 72 h under argon. Yield was based on ¹H NMR analysis of the crude product using 1,3,5-trimethoxybenzene as an internal standard. Ee values were based on chiral HPLC analysis. *fac*-Ir(ppy)₃, tris(2-phenylpyridine)iridium (CAS: 94928-86-6).

Table S2. Control experiments of standard conditions for **S1** and **A1**^a

Entry	<i>fac</i> - Ir(ppy) ₃	[Cu]	L1	K ₃ PO ₄	Blue LEDs	Yield (%)	Ee (%)
1	√	√	√	√	√	80	94
2	×	√	√	√	√	0	—
3	√	×	√	√	√	0	—
4	√	√	×	√	√	8	0
5	√	√	√	×	√	0	—
6	√	√	√	√	×	0	—

^aReaction conditions: **S1** (0.20 mmol, 1.0 equiv), **A1** (1.5 equiv), Cu(CH₃CN)₄PF₆ (10 mol %), **L1** (15 mol %), *fac*-Ir(ppy)₃ (1.0 mol %), and K₃PO₄ (3.0 equiv) in DMF (2.0 mL) stirred at rt and blue LEDs irradiation for 72 h under argon. Yield was based on ¹H NMR analysis of the crude product using 1,3,5-trimethoxybenzene as an internal standard. Ee values were based on chiral HPLC analysis.

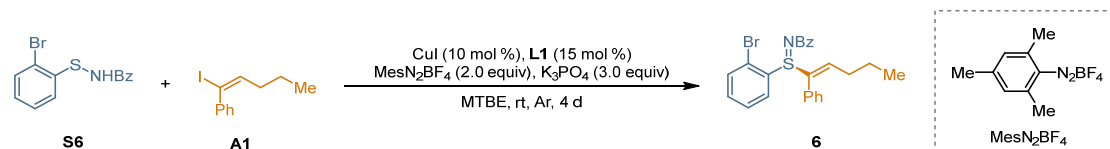
Table S3. Screening of reaction conditions with diazonium salts for **S6** and **A1**^a



Entry	[Cu]	Base	Solvent	Yield (%)	Ee (%)
1	Cu(CH ₃ CN) ₄ PF ₆	K ₃ PO ₄	DMF	0	—
2	Cu(CH ₃ CN) ₄ PF ₆	K ₃ PO ₄	DCM	trace	—
3	Cu(CH ₃ CN) ₄ PF ₆	K ₃ PO ₄	EA	10	86
4	Cu(CH ₃ CN) ₄ PF ₆	K ₃ PO ₄	THF	18	90
5	Cu(CH ₃ CN) ₄ PF ₆	K ₃ PO ₄	MTBE	33	90
6	CuTc	K ₃ PO ₄	MTBE	28	90
7	CuBr	K ₃ PO ₄	MTBE	38	90
8 ^b	CuI	K ₃ PO ₄	MTBE	53	90
9	CuI	K ₃ PO ₄	MTBE	50	90
10	CuI	Cs ₂ CO ₃	MTBE	39	90

^aReaction conditions: **S6** (0.20 mmol, 1.0 equiv), **A1** (1.5 equiv), [Cu] (10 mol %), **L1** (15 mol %), MesN₂BF₄ (2.0 equiv) and base (3.0 equiv) in solvent (4.0 mL) stirred at rt for 4 days under argon. Yield was based on ¹H NMR analysis of the crude product using 1,3,5-trimethoxybenzene as an internal standard. Ee values were based on chiral HPLC analysis. ^bGenerated 84% yield of by-product **57**.

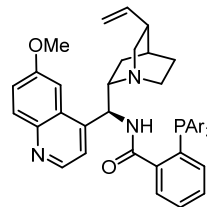
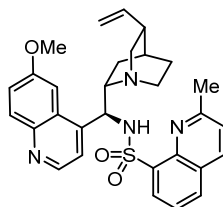
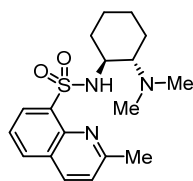
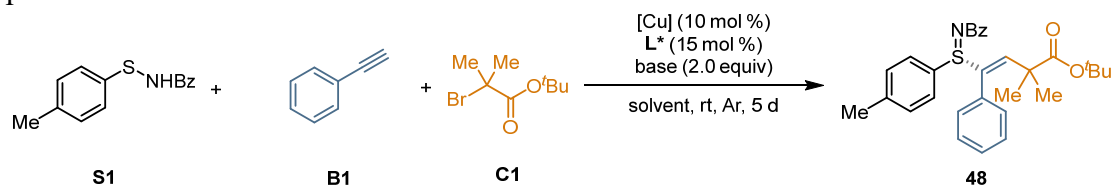
Table S4. Control experiments of standard conditions with diazonium salts for **S6** and **A1**^a



Entry	CuI	L1	MesN ₂ BF ₄	K ₃ PO ₄	Yield (%)	Ee (%)
1	✓	✓	✓	✓	53	90
2	✗	✓	✓	✓	0	—
3	✓	✗	✓	✓	0	—
4	✓	✓	✗	✓	0	—
5	✓	✓	✓	✗	trace	—

^aReaction conditions: **S6** (0.20 mmol, 1.0 equiv), **A1** (1.5 equiv), CuI (10 mol %), **L1** (15 mol %), MesN₂BF₄ (2.0 equiv) and K₃PO₄ (3.0 equiv) in MTBE (4.0 mL) stirred at rt for 4 days under argon. Yield was based on ¹H NMR analysis of the crude product using 1,3,5-trimethoxybenzene as an internal standard. Ee values were based on chiral HPLC analysis.

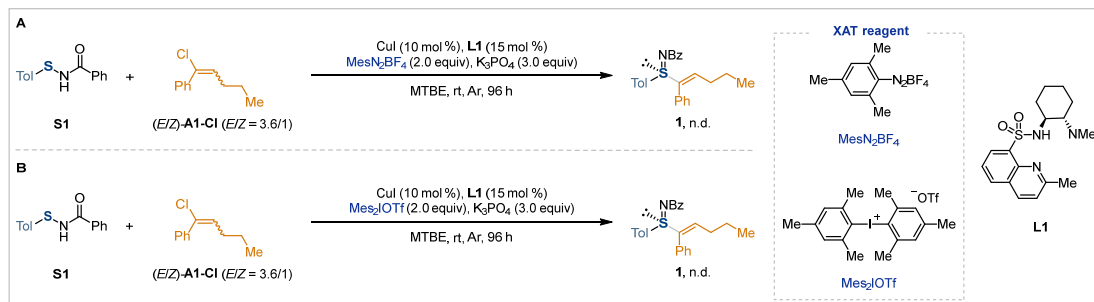
Table S5. Reaction condition optimization with sulfenamide **S1**, alkyne **B1** and radical precursor **C1**^a



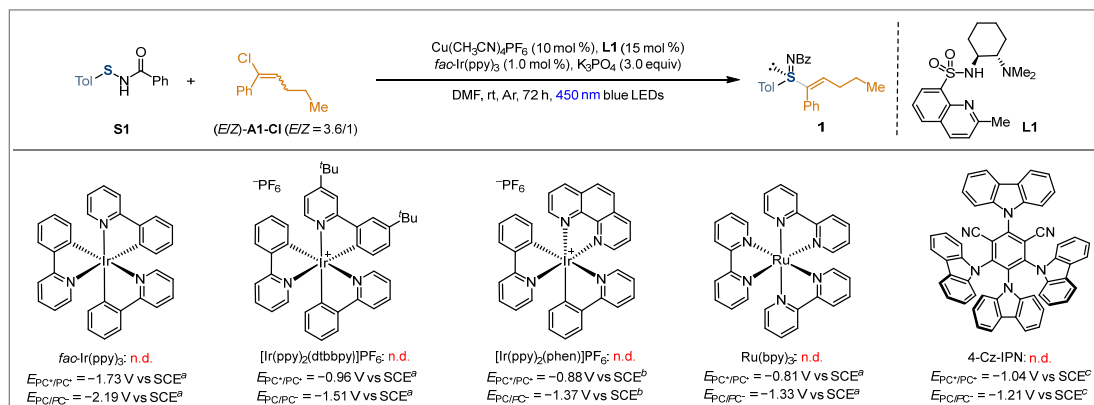
Entry	[Cu]	L*	Base	Solvent	Yield (%)	Ee (%)
1	CuI	L1	Cs ₂ CO ₃	1,4-dioxane	17	58
2	CuI	L5	Cs ₂ CO ₃	1,4-dioxane	13	69
3	CuI	L6	Cs ₂ CO ₃	1,4-dioxane	53	90
4	CuI	L6	Cs ₂ CO ₃	THF	17	5
5	CuI	L6	Cs ₂ CO ₃	toluene	25	10
6	CuI	L6	Cs ₂ CO ₃	DCM	36	15
7	Cu(CH ₃ CN) ₄ PF ₆	L6	Cs ₂ CO ₃	1,4-dioxane	21	44
8	CuTc	L6	Cs ₂ CO ₃	1,4-dioxane	33	86
9	CuI	L6	Na ₂ CO ₃	1,4-dioxane	0	—
10	CuI	L6	K ₃ PO ₄	1,4-dioxane	30	84

^aReaction conditions: **S1** (0.10 mmol, 1.0 equiv), **B1** (2.0 equiv), **C1** (3.0 equiv), [Cu] (10 mol %), **L*** (15 mol %) and base (2.0 equiv) in solvent (1.0 mL) stirred at rt for 5 days. Yield was based on ¹H NMR analysis of the crude product using 1,3,5-trimethoxybenzene as an internal standard. Ee values were based on chiral HPLC analysis.

Scheme S1. Experimental Results of Alkenyl Chloride (*E/Z*)-A1-Cl with Halogen Atom Transfer Reagents

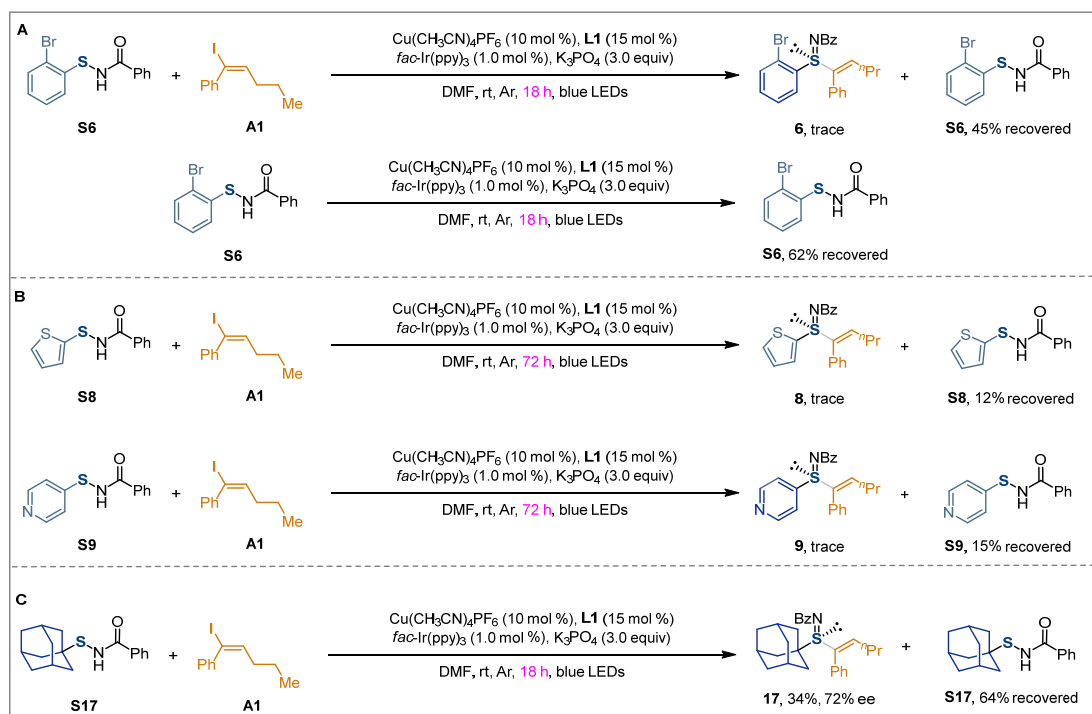


Scheme S2. Screening of Photocatalysts for the Coupling Reaction with Alkenyl Chloride (*E/Z*)-A1-Cl

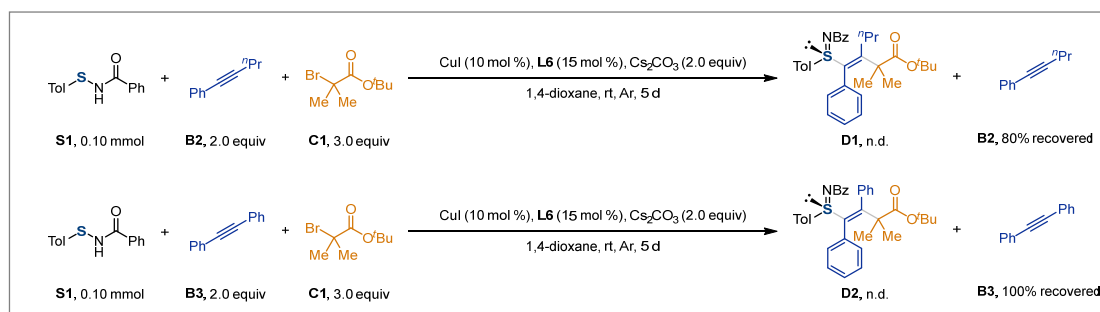


^{a-c}Redox potentials of photocatalysts, including excited-state oxidation potentials (PC*/PC⁺) and ground-state reduction potentials (PC/PC⁻), were obtained from the literature: ^a*J. Photochem. Photobiol. C* **2022**, 50, 100488; ^b*ChemCatChem* **2025**, 17, e00964; ^c*ACS Catal.* **2016**, 6, 873. N.d., not detected.

Scheme S3. Control experiments with sulfenamides S6, S8, S9, and S17



Scheme S4. The Results of Radical 1,2-Carbosulfilimination of Internal Alkynes



2. Supplementary figure for experiments

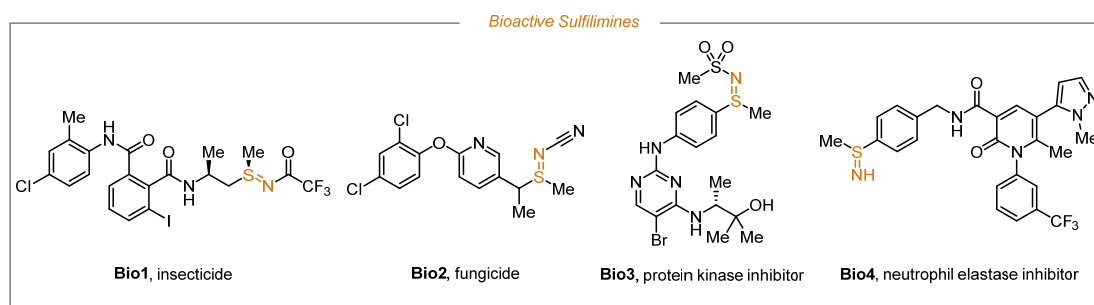


Figure S1. Representative bioactive sulfilimines in medicinal chemistry and crop protection.

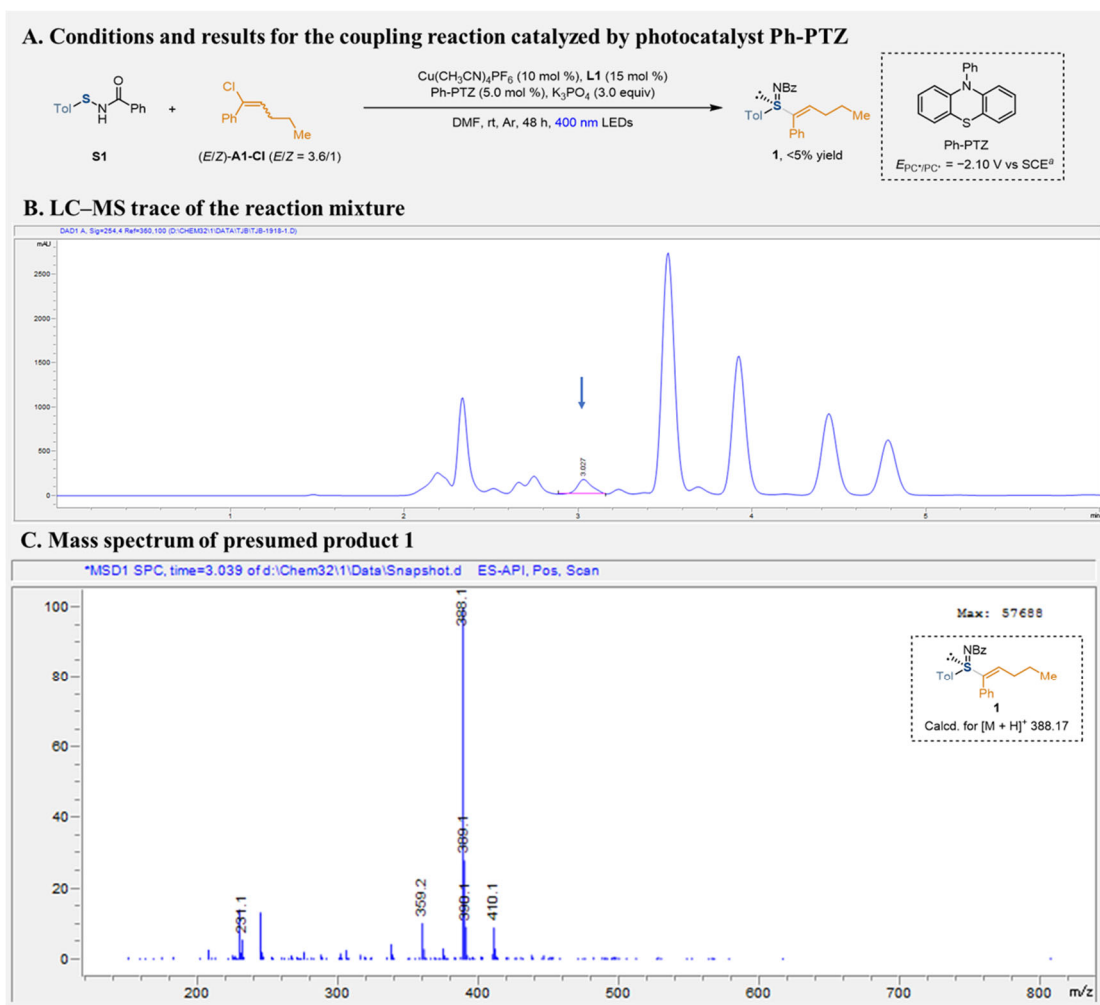


Figure S2. Conditions and results for the coupling reaction of alkenyl chloride (*E/Z*)-A1-Cl with the photocatalyst Ph-PTZ. LC-MS, liquid chromatography-mass spectrometry. ^aExcited-state oxidation potential (PC^{*}/PC⁺) of Ph-PTZ was obtained from the literature: *J. Am. Chem. Soc.* **2014**, *136*, 16096.

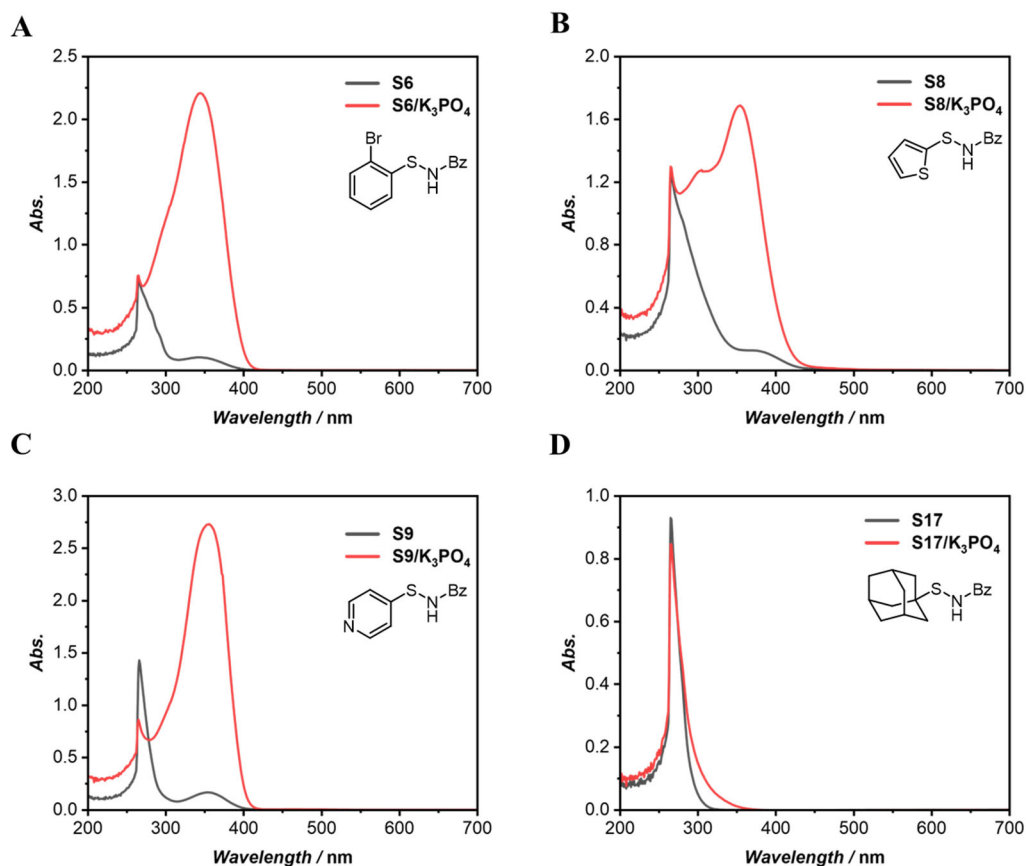


Figure S3. UV-Vis absorption spectra of sulfenamides under base-free and base-added conditions. Spectra were recorded on a Shimadzu UV-2600i spectrometer using a 10 mm path-length cuvette. Gray line: sulfenamide (2.0 mg) was dissolved in DMF (0.20 mL), and an appropriate aliquot was diluted with DMF to give a 2.0 mL, 250 μ M solution. Red line: sulfenamide (2.0 mg) and K_3PO_4 (20.0 mg) were mixed with DMF (0.20 mL) and stirred at rt for 1.5 h; the resulting mixture was filtered to remove undissolved K_3PO_4 and then an appropriate aliquot of the filtered mixture was diluted with DMF to give a 2.0 mL, 250 μ M solution. (A) S6; (B) S8; (C) S9; (D) S17.

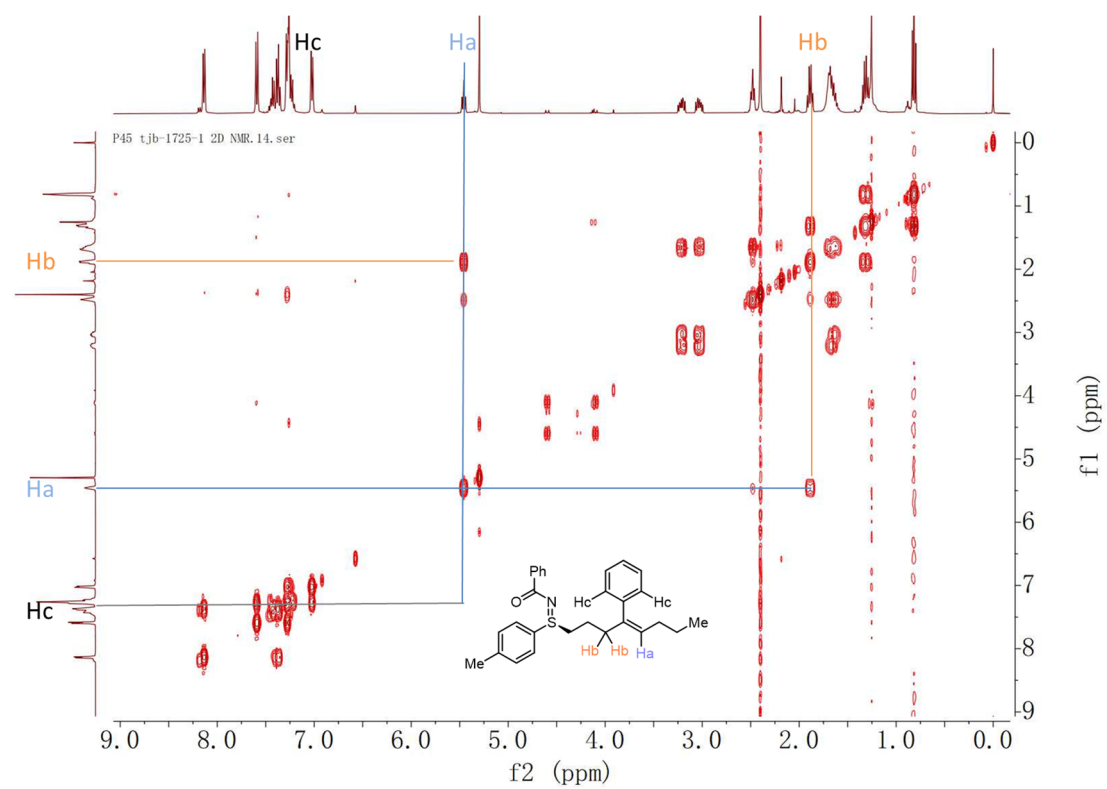


Figure S4. NOESY spectrum of compound **47**.

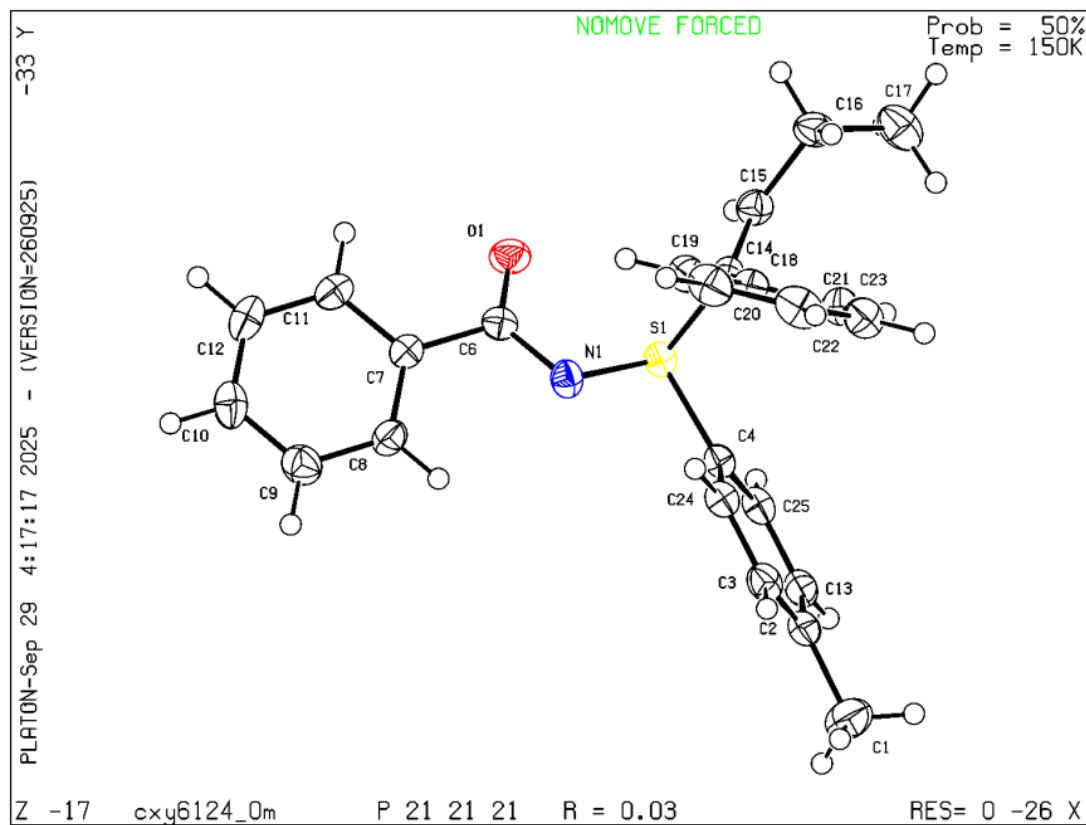
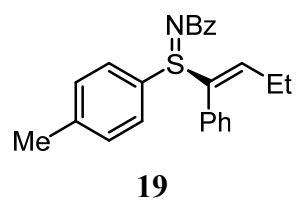


Figure S5. The X-ray structure of **19** (CCDC 2504953, 50% probability ellipsoids).

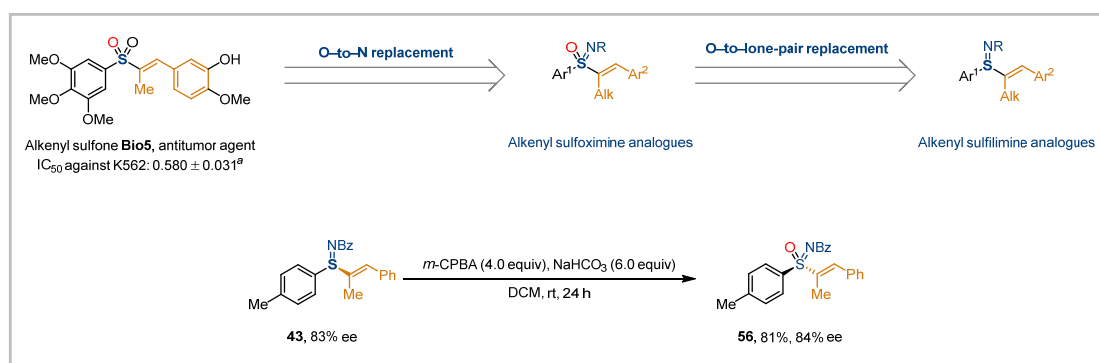


Figure S6. Synthesis of sulfilimine **43** and sulfoximine **56** as S(IV) and S(VI) analogues of the bioactive vinyl sulfone **Bio5**. ^aThe IC₅₀ value of compound **Bio5** against the human cancer K562 cell line was obtained from the literature (*Eur. J. Med. Chem.* **2018**, *157*, 1068).

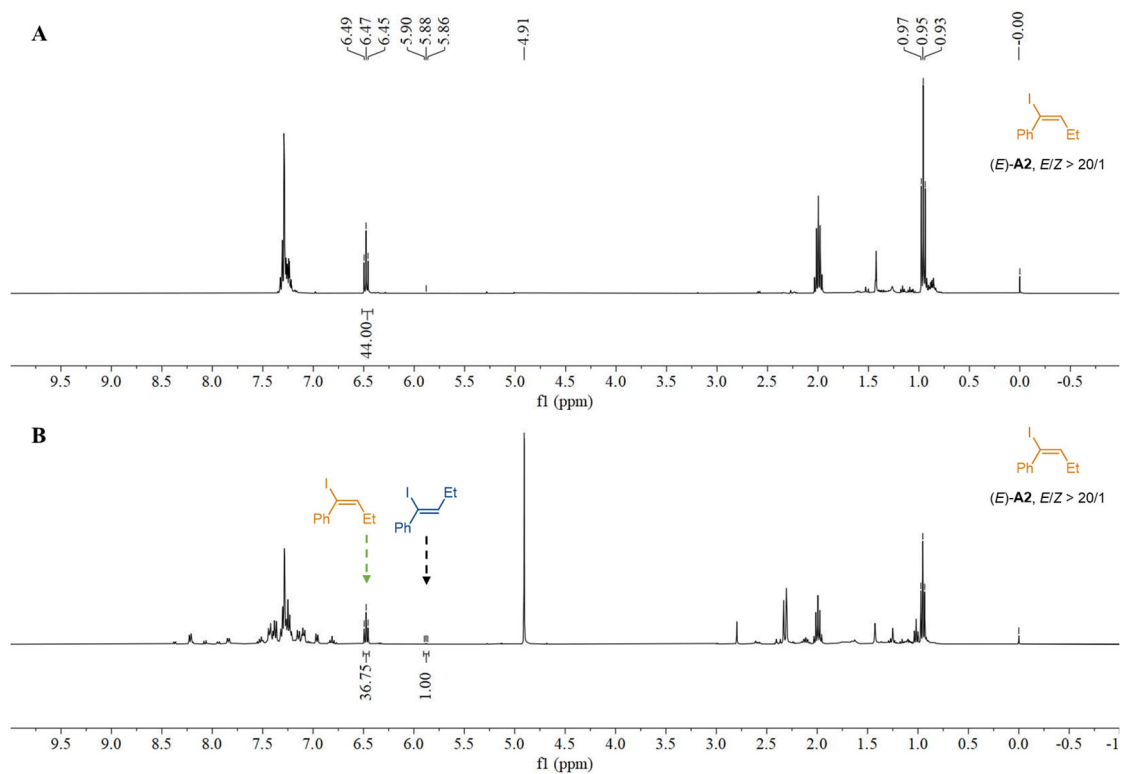


Figure S7. Control experiments of alkenyl iodide (*E*)-**A2** with photoinduced. **(A)** ^1H NMR spectrum of alkenyl iodide (*E*)-**A2**; **(B)** The *E/Z* ratio of the recovered **A2** were based on ^1H NMR analysis of the crude reaction mixture using CH_2Br_2 as an internal standard.

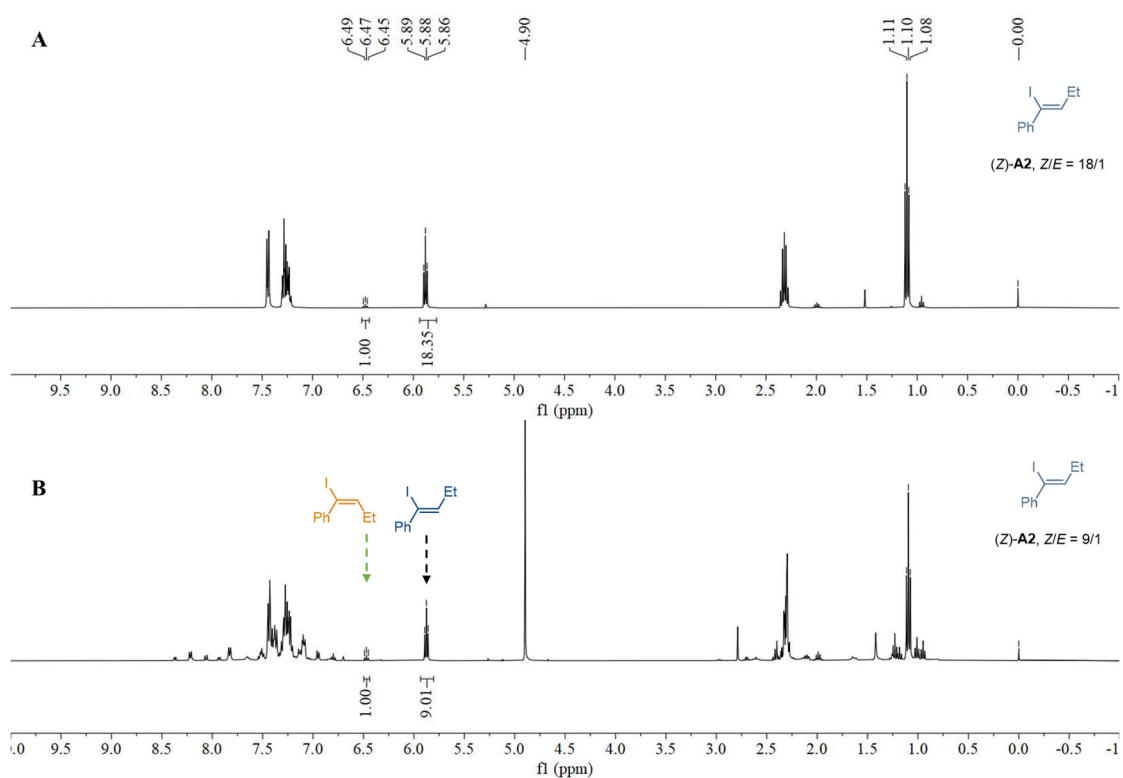


Figure S8. Control experiments of alkenyl iodide (Z)-A2 with photoinduced. **(A)** ^1H NMR spectrum of alkenyl iodide (Z)-A2; **(B)** The *E/Z* ratio of the recovered A2 were based on ^1H NMR analysis of the crude reaction mixture using CH_2Br_2 as an internal standard.

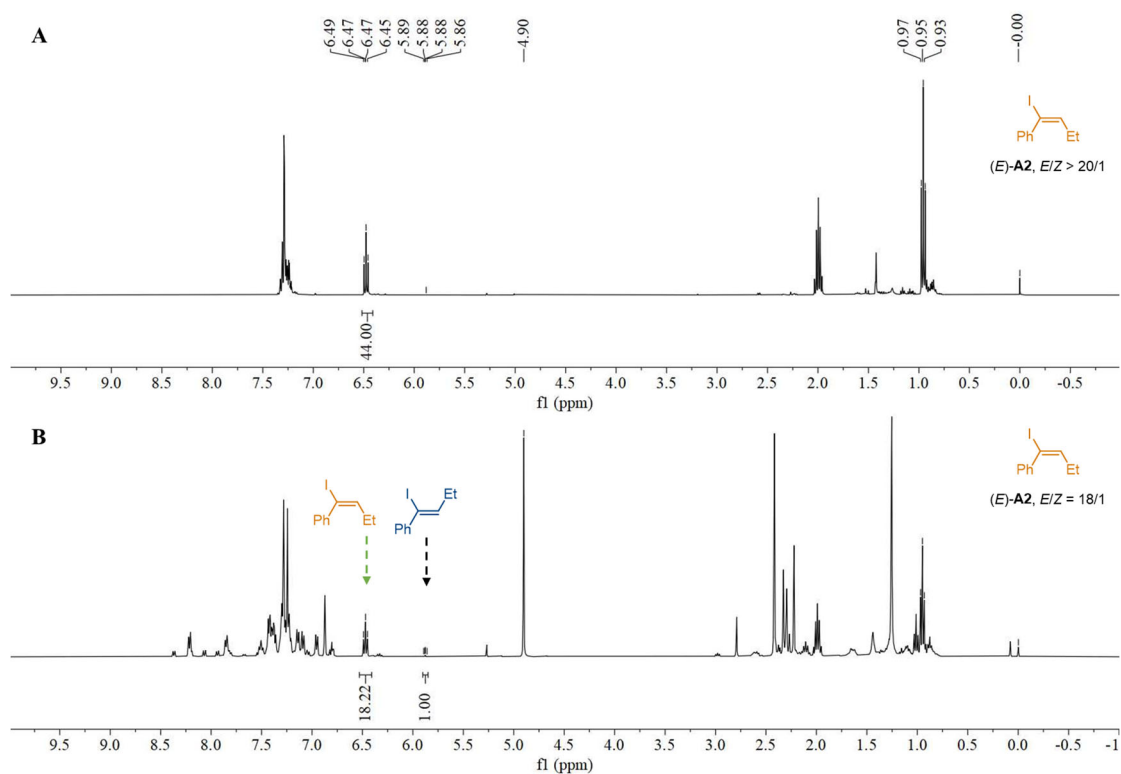


Figure S9. Control experiments of alkenyl iodide (*E*)-A2 with diazonium salts. **(A)** ^1H NMR spectrum of alkenyl iodide (*E*)-A2; **(B)** The *E/Z* ratio of the recovered A2 were based on ^1H NMR analysis of the crude reaction mixture using CH_2Br_2 as an internal standard.

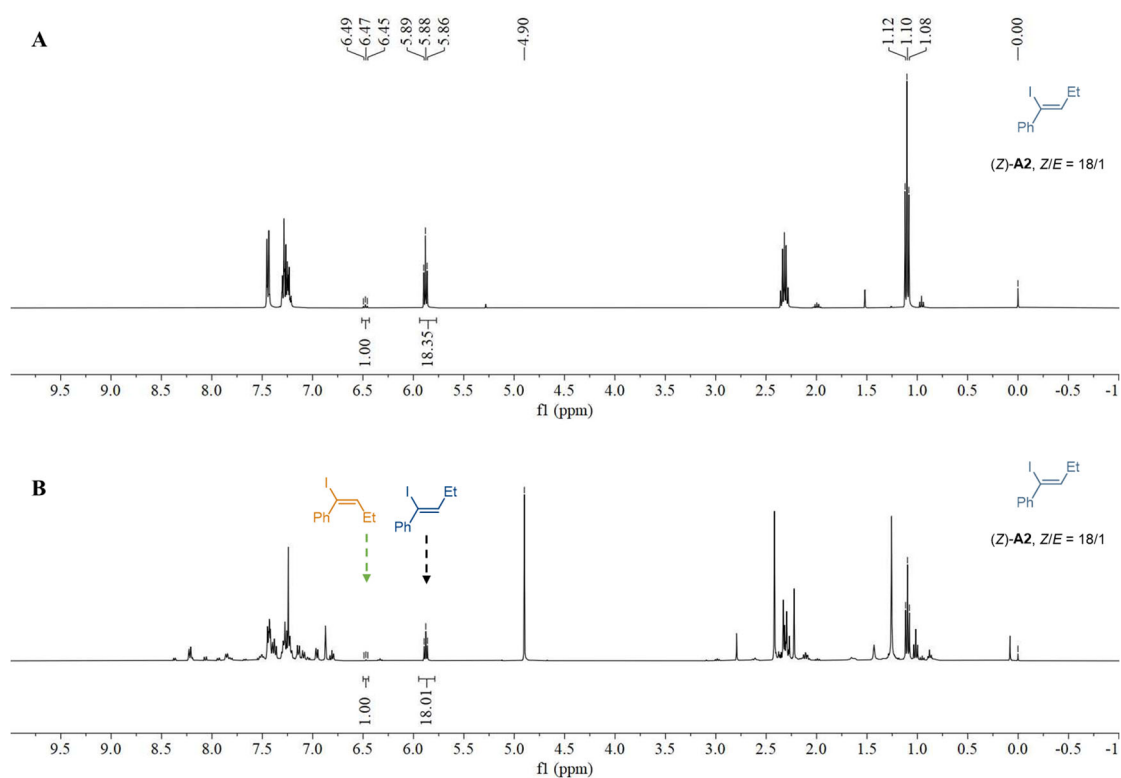


Figure S10. Control experiments of alkenyl iodide (Z)-A2 with diazonium salts. **(A)** ^1H NMR spectrum of alkenyl iodide (Z)-A2; **(B)** The *E/Z* ratio of the recovered A2 were based on ^1H NMR analysis of the crude reaction mixture using CH_2Br_2 as an internal standard.

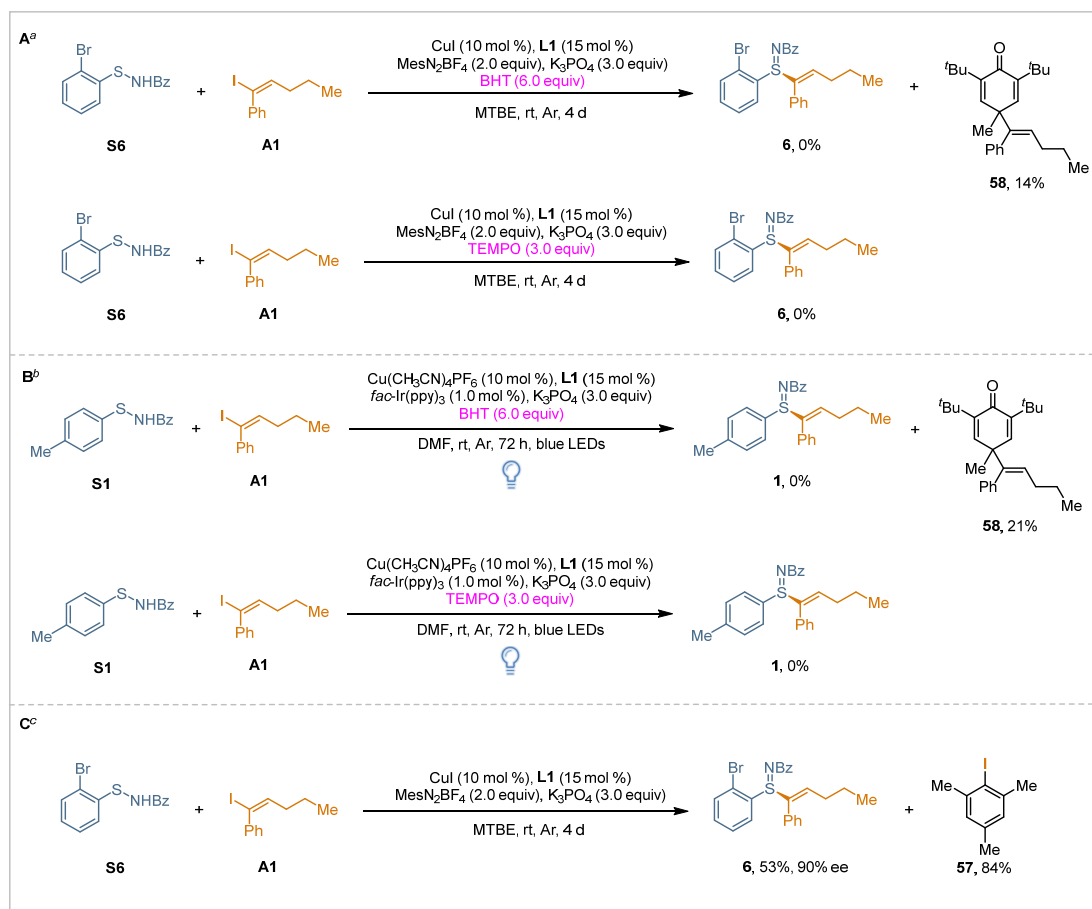


Figure S11. Radical trapping experiments for vinyl radical in chiral **S(IV)**. ^aConditions: **S6** (0.20 mmol), **A1** (1.5 equiv), CuI (10 mol %), **L1** (15 mol %), BHT (6.0 equiv) or TEMPO (3.0 equiv), MesN₂BF₄ (2.0 equiv) and K₃PO₄ (3.0 equiv) in MTBE (4.0 mL) at rt for 4 days under argon. ^bConditions: **S1** (0.20 mmol), **A1** (1.5 equiv), Cu(CH₃CN)₄PF₆ (10 mol %), **L1** (15 mol %), BHT (6.0 equiv) or TEMPO (3.0 equiv), *fac*-Ir(ppy)₃ (1.0 mol %), and K₃PO₄ (3.0 equiv) in DMF (2.0 mL) stirred at rt and blue LEDs irradiation for 72 h under argon. ^cConditions: **S6** (0.20 mmol), **A1** (1.5 equiv), CuI (10 mol %), **L1** (15 mol %), MesN₂BF₄ (2.0 equiv) and K₃PO₄ (3.0 equiv) in MTBE (4.0 mL) at rt for 4 days under argon. Yields were isolated. BHT, butylated hydroxytoluene; TEMPO, 2,2,6,6-tetramethylpiperidinoxy; Mes, 2,4,6-trimethylphenyl.

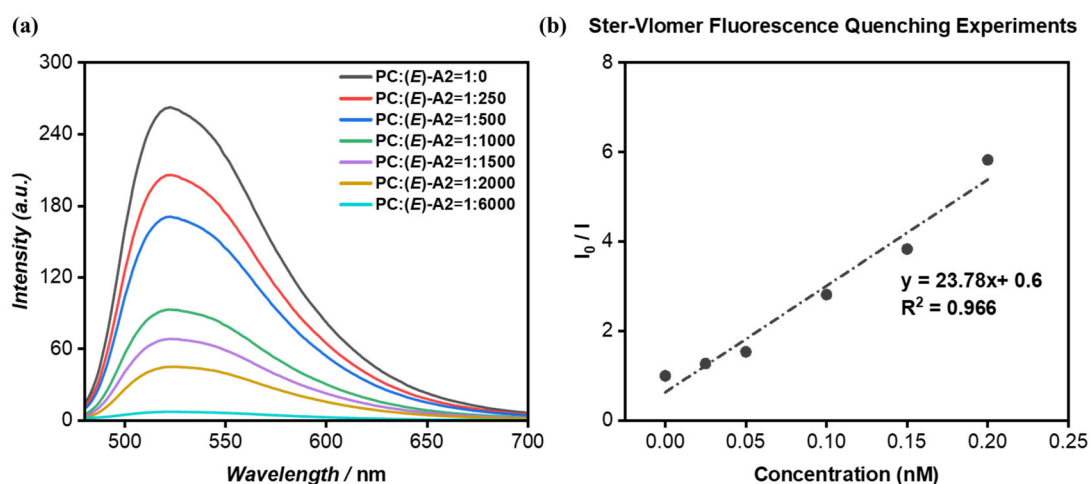


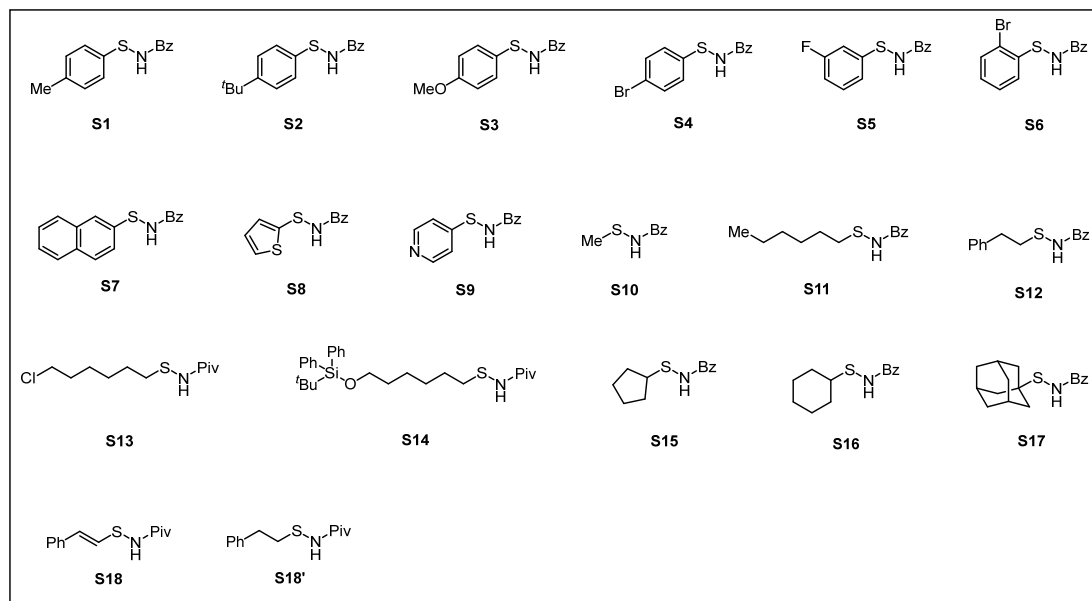
Figure S12. Stern-Volmer Fluorescence Quenching Experiments. Stern-Volmer plot for the emission quenching of *fac*-Ir(ppy)₃ by various concentrations of (*E*)-A2. Emission intensities were recorded using Edinburgh Instruments FLS1000 photoluminescence spectrometer for all experiments. All *fac*-Ir(ppy)₃ solutions were excited at 375 nm and the emission intensity was collected at 528 nm. The solution of *fac*-Ir(ppy)₃ in a mixture solvent of DMF (1.0×10^{-4} M) was added the appropriate amount of (*E*)-A2 in a screw-top 1.0 cm quartz cuvette.

3. General information

Most of reactions were carried out under argon atmosphere using Schlenk techniques. Reagents were purchased at the highest commercial quality and used without further purification, unless otherwise stated. Cu-TMEDA and K₃PO₄ were purchased from Bide Pharmatech Ltd., which was directly used without further treatment. Other reagents were purchased from J&K, Energy, etc. Anhydrous PhCF₃ were purchased from J&K. Analytical thin layer chromatography (TLC) was performed on precoated silica gel 60 GF254 plates. Flash column chromatography was performed using Tsingdao silica gel (60, particle size 0.040–0.063 mm). As the eluent, the petroleum ether (PE), hexane, ethyl acetate (EtOAc), dichloromethane (DCM) and methanol were purchased from Shanghai Titan Scientific Co. Ltd without further purification. The Blue LED lamp is purchased from Taobao (Figure S9). Visualization on TLC was achieved by use of UV light (254 nm), iodine or basic KMnO₄ indicator. NMR spectra were recorded on Bruker DRX-400 and DPX-600 spectrometers at 400 or 600 MHz for ¹H NMR and 100 or 151 MHz for ¹³C NMR, respectively, in CDCl₃ with tetramethylsilane (TMS) as internal standard. The chemical shifts were expressed in ppm and coupling constants were given in Hz. Data for ¹H NMR are recorded as follows: chemical shift (ppm), multiplicity (s, singlet; d, doublet; dd = doublet of doublets; dt = doublet of triplet; ddd = doublet of doublet of doublets; t, triplet; q, quartet; m, multiplet), coupling constant (Hz), integration. Data for ¹³C NMR are reported in terms of chemical shift (δ, ppm), multiplicity (d, doublet; q, quarter), coupling constant (Hz), integration. High-resolution mass spectroscopy (HRMS) was obtained on Thermo Scientific Q Exactive mass spectrometer using ESI ion source. Enantiomeric excess (ee) was determined using SHIMADZU LC-20AD with SPD-20AV detector. Column conditions were reported in the experimental section below. X-ray diffraction was measured on a 'Bruker APEX-II CCD' diffractometer with Cu–Kα radiation.

4. General procedure for the synthesis of the substrates

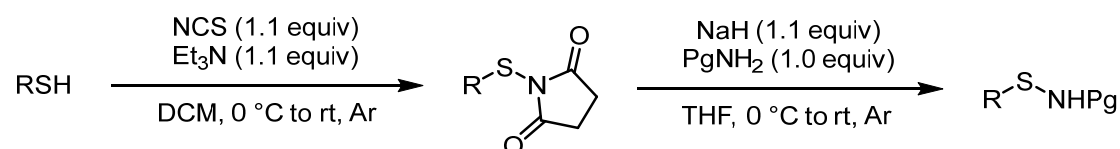
The synthesis of sulfenamides:



S1-S8, S11-S17 and S18' were synthesized according to the **General procedure 1**. All the characterization data are consistent with those in the reported literature.¹

S9 and S10 were synthesized according to the **General procedure 2**. All the characterization data are consistent with those in the reported literature.²

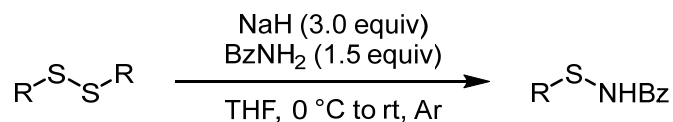
S18 was synthesized according to the **General procedure 3**. All the characterization data are consistent with those in the reported literature.³



General procedure 1:

According to the literature reported procedure.⁴ Under argon atmosphere and ice bath, to a solution of thiol (10.0 mmol, 1.0 equiv) in anhydrous DCM (0.2 M, 50.0 mL) was slowly added *N*-Chlorosuccinimide (NCS) (11.0 mmol, 1.1 equiv). Then, the mixture was stirred at rt for 0.5 h. After that, triethylamine (11.0 mmol, 1.1 equiv) was slowly added into the mixture under an ice bath, and then the reaction mixture was stirred at rt overnight. Upon completion (monitored by TLC), the reaction was quenched with a saturated aqueous solution of NaHCO₃ and extracted with EtOAc. The organic layers were collected, washed with brine, dried over anhydrous Na₂SO₄, filtered, and concentrated. The crude product was directly used in next step without further purification. Under argon atmosphere and ice bath, to a solution of amide (1.0 equiv) in anhydrous THF (0.33 M) was slowly added sodium hydride (60% in mineral oil, 1.1 equiv). Then, the mixture was stirred at rt for 0.5 h. After that, the intermediate product (1.0 equiv) was slowly added into the mixture under an ice bath, and then the reaction mixture was stirred at rt overnight. Upon completion (monitored by TLC), the reaction

was quenched with a saturated aqueous solution of NH_4Cl , and extracted with EtOAc. The organic layers were collected, washed with brine, dried over anhydrous Na_2SO_4 , filtered, and concentrated. The crude mixture was purified by column chromatography on silica gel to afford the desired product.



General procedure 2:

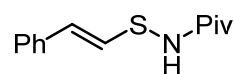
According to the literature reported procedure.⁵ Under argon atmosphere and ice bath, to a solution of benzamide (1.82 g, 15.0 mmol, 1.5 equiv) in anhydrous THF (100.0 mL) was slowly added sodium hydride (60% in mineral oil, 1.20 g, 30.0 mmol, 3.0 equiv). Then, the mixture was stirred at rt for 0.5 h. After that, disulfide (10.0 mmol, 1.0 equiv) was slowly added into the mixture under an ice bath, and then the reaction mixture was stirred at rt overnight. Upon completion (monitored by TLC), the reaction was quenched with a saturated aqueous solution of NH_4Cl and extracted with EtOAc. The organic layers were collected, washed with brine, dried over anhydrous Na_2SO_4 , filtered, and concentrated. The crude mixture was purified by column chromatography on silica gel to afford the desired product.

General procedure 3:



According to the literature reported procedure with slight modification.³ To a flame-dried 100 mL Schlenk tube equipped with a magnetic stir bar was charged with CuI (380.9 mg, 2.0 mmol, 40 mol %), sulfenamide **S18'** (1.18 g, 5.0 mmol, 1.0 equiv), (*E*)-(2-iodovinyl)benzene (2.30 g, 10.0 mmol, 2.0 equiv) and Na_2CO_3 (1.06 g, 10.0 mmol, 2.0 equiv). The tube was evacuated and backfilled with argon three times, followed by the addition of anhydrous DMSO (50 mL). The reaction mixture was stirred at 80 °C for 48 h. After cooling to rt, the reaction mixture was filtered through a pad of celite, which was washed with ethyl acetate. The filtrate was transferred to a separatory funnel and the organic layer was washed with H_2O and then with saturated aqueous NaCl. The organic layer was dried over sodium sulfate and concentrated. The residue was purified by column chromatography on silica gel to afford the desired product **S18**.

(*E*)-*N*-(Styrylthio)pivalamide (**S18**)



The reaction mixture was purified by column chromatography on silica gel (petroleum ether) to afford product **S18** as a white solid (0.72 g, 61% yield).

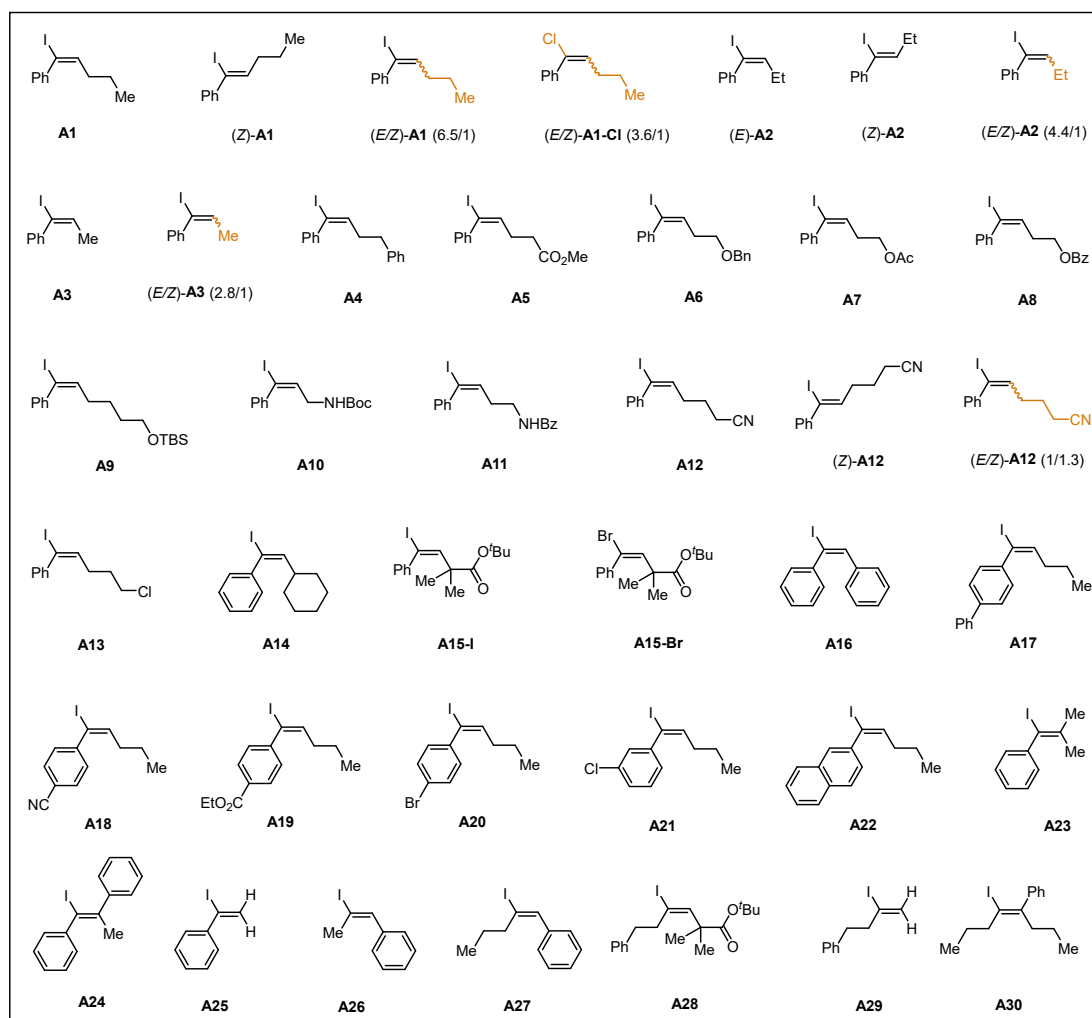
^1H NMR (400 MHz, CDCl_3) δ 7.39 – 7.23 (m, 4H), 7.23 – 7.17 (m, 1H), 6.75 (s, 1H),

6.68 (d, $J = 15.4$ Hz, 1H), 6.40 (d, $J = 15.4$ Hz, 1H), 1.30 (s, 9H).

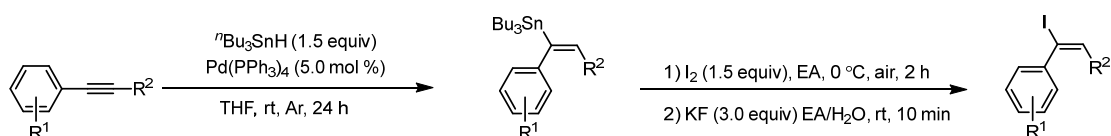
^{13}C NMR (100 MHz, CDCl_3) δ 179.7, 136.0, 128.7, 127.6, 126.5, 126.2, 126.0, 40.1, 27.9.

HRMS (ESI) m/z calcd. for $\text{C}_{13}\text{H}_{18}\text{NOS}$ $[\text{M} + \text{H}]^+$ 236.1104, found 236.1104.

The synthesis of alkenyl iodide:



The synthesis procedure for A1, (E)-A2, A3-A14 and A16-A22:

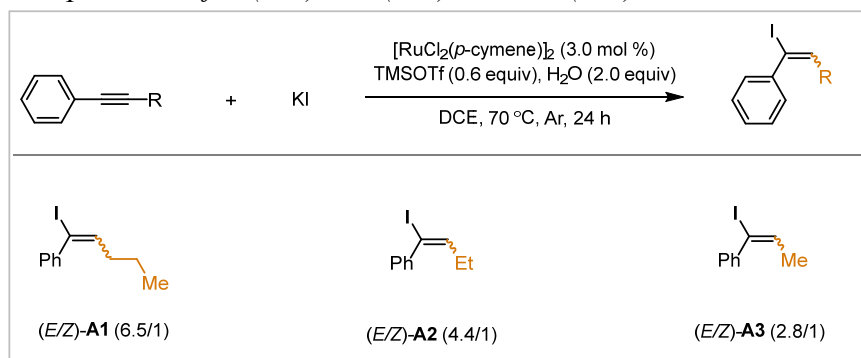


According to the literature reported procedure with modification.⁶ The analytic data of **A1**, (*E*)-**A2**, **A3**, **A13**, **A14** and **A16** is in accordance with literature data.

ⁿBu₃SnH (2.02 mL, 7.5 mmol, 1.5 equiv) was added to a solution of internal alkyne (5.0 mmol, 1.0 equiv) and Pd(PPh₃)₄ (288.9 mg, 0.25 mmol, 0 mol %) in dry THF (10.0 mL) at rt. The mixture was stirred under argon at that temperature for 24 h and then solvent was removed under vacuum. To a solution of the crude vinyl stannane in ethyl acetate (20.0 mL) at 0 °C was added I₂ (1.91 g, 7.5 mmol, 1.5 equiv) in portions. After 2 h at rt, saturated aqueous Na₂S₂O₃ solution was added. The organic layer was separated. The resulting organic layer was added KF (0.87 g, 15.0 mmol, 3.0 equiv) and H₂O (10.0 mL) and then stirred at rt until the by-product ⁿBu₃SnI was completely precipitated into ⁿBu₃SnF. After filtration of the solid, the organic layer was concentrated under reduced pressure. The residue was purified by flash silica gel column chromatography to afford

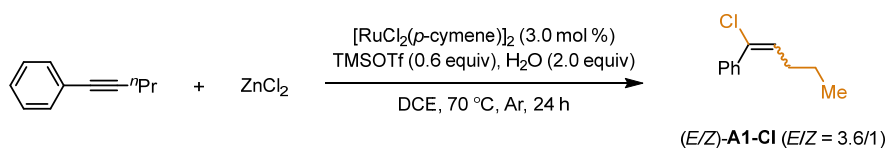
the desired product.

The synthesis procedure for (E/Z)-A1, (E/Z)-A2, and (E/Z)-A3 mixture:



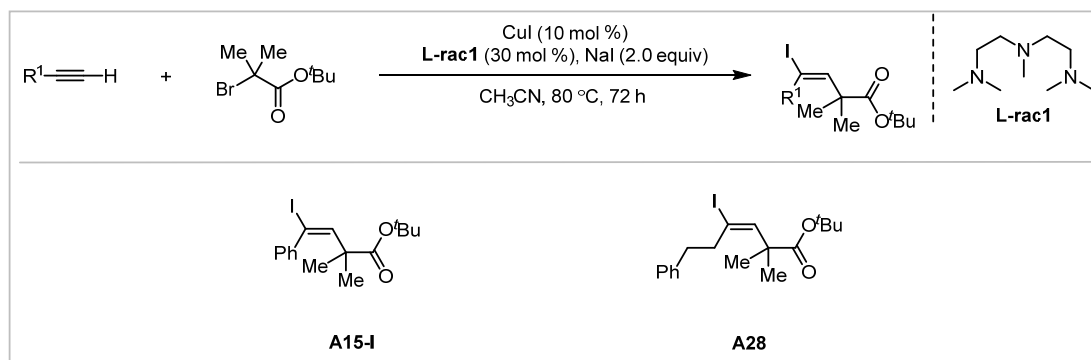
According to the literature reported procedure.⁷ A solution of internal alkyne (10.0 mmol, 1.0 equiv), KI (1.99 g, 12.0 mmol, 1.2 equiv), [RuCl₂(p-cymene)]₂ (184.0 mg, 0.3 mmol, 3.0 mol %), TMSOTf (1.08 mL, 0.6 mmol, 0.6 equiv) and H₂O (0.36 mL, 20.0 mmol, 2.0 equiv) in DCE (30.0 mL) were stirred at 70 °C for 24 h under argon. After completion of the reaction, 30.0 mL of water added, and the mixture was extracted three times with ethyl acetate. The combined organic layer was washed with brine, dried over anhydrous Na₂SO₄ and concentrated. The residue was purified by column chromatography on silica gel to afford the desired product.

The synthesis procedure for (E/Z)-A1-Cl mixture:



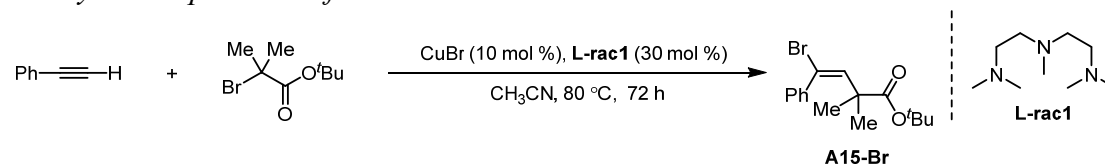
According to the literature reported procedure.⁷ A solution of pent-1-yn-1-ylbenzene (1.44 g, 10.0 mmol, 1.0 equiv), ZnCl₂ (0.82 g, 6.0 mmol, 0.6 equiv), [RuCl₂(p-cymene)]₂ (184.0 mg, 0.3 mmol, 3.0 mol %), TMSOTf (1.08 mL, 0.6 mmol, 0.6 equiv) and H₂O (0.36 mL, 20.0 mmol, 2.0 equiv) in DCE (30.0 mL) were stirred at 70 °C for 24 h under argon. After completion of the reaction, 30.0 mL of water added, and the mixture was extracted three times with ethyl acetate. The combined organic layer was washed with brine, dried over anhydrous Na₂SO₄ and concentrated. The residue was purified by column chromatography on silica gel to afford the desired product.

The synthesis procedure for A15-I and A28:



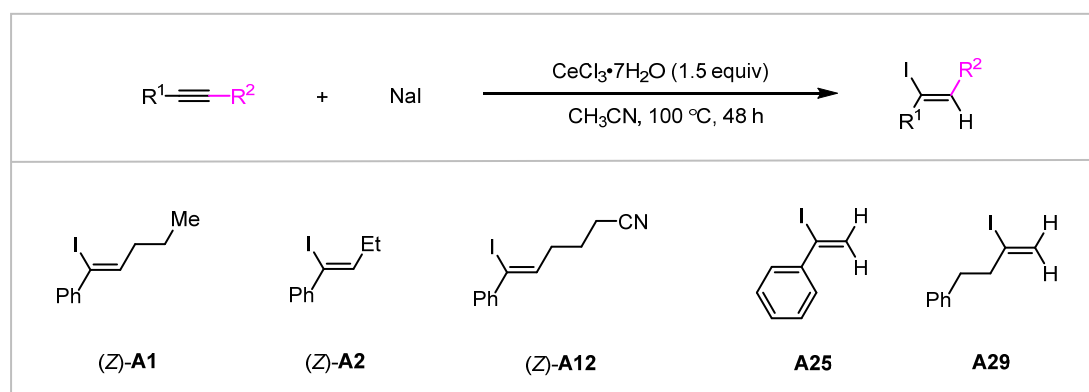
According to the literature reported procedure.⁸ Under an argon atmosphere, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with alkyne (12.0 mmol, 1.2 equiv), *tert*-butyl 2-bromo-2-methylpropanoate (1.86 mL, 10.0 mmol, 1.0 equiv), CuI (190.4 mg, 1.0 mmol, 10 mol %), **L-rac1** (0.52 g, 3.0 mmol, 30 mol %) and NaI (3.0 g, 20.0 mmol, 2.0 equiv) in CH₃CN (30.0 mL) and stirred at 80 °C for 72 h under argon. Upon completion, the precipitate was filtered off and washed by DCM and twice with saturated aqueous Na₂S₂O₃ solution, saturated aqueous NH₄Cl solution. The organic phase was dried with anhydrous Na₂SO₄, filtered, and concentrated under reduced pressure and the residue was purified by flash silica gel column chromatography to afford the desired product.

The synthesis procedure for A15-Br:



According to the literature reported procedure.⁸ Under an argon atmosphere, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with ethynylbenzene (1.09 mL, 10.0 mmol, 1.0 equiv), *tert*-butyl 2-bromo-2-methylpropanoate (2.24 mL, 12.0 mmol, 1.2 equiv), CuBr (141.8 mg, 1.0 mmol, 10 mol %) and **L-rac1** (0.52 g, 3.0 mmol, 30 mol %) in CH₃CN (30.0 mL) and stirred at 80 °C for 72 h under argon. Upon completion, the precipitate was filtered off and washed by DCM and saturated aqueous NH₄Cl solution. The organic phase was dried with anhydrous Na₂SO₄, filtered, and concentrated under reduced pressure and the residue was purified by flash silica gel column chromatography to afford the product.

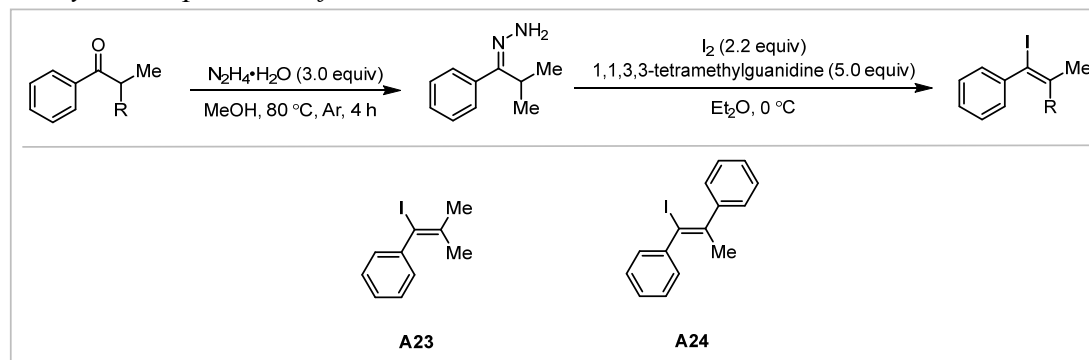
The synthesis procedure for (Z)-A1, (Z)-A2, (Z)-A12, A25 and A29:



According to the literature reported procedure.⁹ The analytic data of (Z)-A2, A25 and A29 is in accordance with literature data.

In a 100 mL round-bottom flask, the alkyne (10.0 mmol, 1.0 equiv), NaI (4.49 g, 30.0 mmol, 3.0 equiv) and $CeCl_3 \cdot 7H_2O$ (5.59 g, 15.0 mmol, 1.5 equiv) was dissolved in CH_3CN (50.0 mL). Then the resulting mixture was reflux for 48 h. The reaction mixture was diluted with ethyl acetate and treated with 1.0 M HCl. The solution was extracted with ethyl acetate. The organic layer was washed with saturated aqueous $NaHCO_3$ solution and saturated aqueous $Na_2S_2O_3$ solution and dried over anhydrous Na_2SO_4 . The organic layer was concentrated under reduced pressure and the residue was purified by flash silica gel column chromatography to afford the desired product.

The synthesis procedure for A23 and A24:



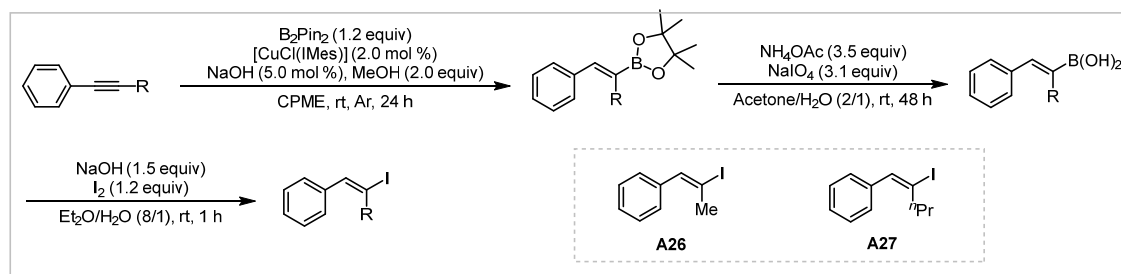
According to the literature reported procedure.^{10,11} The analytic data of A23 is in accordance with literature data.

To a solution of aryl ketone (10.0 mmol, 1.0 equiv) in methanol (10 mL) was added hydrazine hydrate (1.5 mL, 30.0 mmol, 3.0 equiv) and the mixture stirred at 80 °C for 4 h in a sealed vial. The solvent was removed under reduced pressure and the residue diluted with water and DCM. The mixture was separated, the aqueous layer extracted with DCM and the combined organic extracts were dried over anhydrous Na_2SO_4 , filtered and evaporated under reduced pressure to provide the crude hydrazone without further purification.

A flame-dried flask with stir bar was charged with iodine (5.58 g, 22.0 mmol, 2.2 equiv). Anhydrous Et_2O (15.0 mL) was added and the flask was cooled at 0 °C and 1,1,3,3-tetramethylguanidine (6.3 mL, 50.0 mmol, 5.0 equiv) was added dropwise via syringe

as a solution in anhydrous Et₂O (15.0 mL). The reaction was allowed to stir at 0 °C for 10 min after the crude hydrazone (10.0 mmol, 1.0 equiv) was added dropwise via syringe as a solution in anhydrous Et₂O (8.0 mL). Following complete addition of the hydrazone solution, the reaction was left to stir at 0 °C for 30 min. The reaction was then quenched with 1 M HCl and diluted with Et₂O. The aqueous phase was removed and the organic phase was washed twice with saturated aqueous Na₂S₂O₃ solution, once with 1 M HCl, and once with brine. The organic phase was dried with anhydrous Na₂SO₄, filtered and concentrated under reduced pressure and the residue was purified by flash silica gel column chromatography to afford the desired product.

The synthesis procedure for A26 and A27:



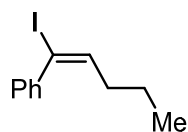
According to the literature reported procedure.¹² Under an argon atmosphere, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with internal alkyne (20.0 mmol, 1.0 equiv), $[\text{CuCl}(\text{IMes})]$ (161.8 mg, 0.4 mmol, 2.0 mol %), NaOH (40.0 mg, 1.0 mmol, 0 mol %), and bis-(pinacolato)diboron (6.10 g, 24 mmol, 1.2 equiv), in CPME (50.0 mL), followed by the addition of MeOH (1.28 g, 40.0 mmol, 2.0 equiv) and stirred at rt for 18 h under argon. Upon completion, quenched with H_2O , extracted with DCM, dried with anhydrous Na_2SO_4 , filtered and concentrated. The residue was purified by silica gel column chromatography to afford the vinyl boronate.

A 250-mL round-bottomed flask equipped with a magnetic stir bar was charged with vinyl boronate (2.44 g, 10.0 mmol, 1.0 equiv), NH_4OAc (2.73 g, 35.0 mmol, 3.5 equiv), NaIO_4 (6.63 g, 31.0 mmol, 3.1 equiv), acetone (80.0 mL) and H_2O (40.0 mL). The resulting reaction mixture was stirred for 48 h at rt and extracted with ethyl acetate. The combined organic phase was dried over Na_2SO_4 . After removal of the solvent, the desired vinyl boric acid was obtained as a crude reaction mixture and the was used next step without further purification.

A 100-mL round-bottomed flask equipped with a magnetic stir bar was charged with the crude vinyl boric acid (1.61 g, 10.0 mmol, 1.0 equiv), NaOH (0.6 g, 15.0 mmol, 1.5 equiv), Et_2O (20.0 mL) and H_2O (5.0 mL) and I_2 (3.04 g, 12.0 mmol, 1.2 equiv) in Et_2O (20.0 mL). The resulting reaction mixture was stirred for 1h at rt and quenched with saturated aqueous $\text{Na}_2\text{S}_2\text{O}_3$ solution and the resulting aqueous phase was extracted with Et_2O . The combined organic phase was dried over Na_2SO_4 . After removal of the solvent, the resulting crude mixture was purified by silica gel column chromatography to afford the vinyl iodide.

A30 was synthesized according to the reported literature and the characterization data is consistent with those in the reported literature.¹³

(E)-(1-Iodopent-1-en-1-yl)benzene (A1)

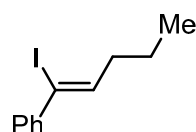


The reaction mixture was purified by column chromatography on silica gel (petroleum ether) to afford product **A1** as a colorless oil (1.08 g, 80% yield).

^1H NMR (400 MHz, CDCl_3) δ 7.43 – 7.14 (m, 5H), 6.48 (t, J = 7.6 Hz, 1H), 1.97 (q, J = 7.5 Hz, 2H), 1.44 – 1.32 (m, 2H), 0.85 (t, J = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 143.7, 142.0, 128.9, 128.3, 128.0, 94.8, 34.3, 22.5, 13.7.

(Z)-(1-Iodopent-1-en-1-yl)benzene ((Z)-A1)

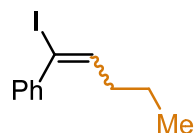


The reaction mixture was purified by column chromatography on silica gel (petroleum ether) to afford product (Z)-**A1** as a colorless oil (1.66 g, 61% yield)

^1H NMR (400 MHz, CDCl_3) δ 7.49 – 7.39 (m, 2H), 7.35 – 7.20 (m, 3H), 5.89 (t, J = 6.7 Hz, 1H), 2.35 – 2.22 (m, 2H), 1.60 – 1.47 (m, 2H), 1.00 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 143.4, 139.1, 128.7, 128.3, 128.2, 105.1, 39.9, 21.8, 14.0.

(E/Z)-(1-Iodopent-1-en-1-yl)benzene ((E/Z)-A1)

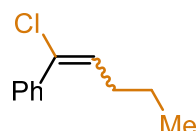


The reaction mixture was purified by column chromatography on silica gel (petroleum ether) to afford product (E/Z)-**A1** as a colorless oil (1.77 g, 65% yield, E/Z = 6.5/1).

^1H NMR of (400 MHz, CDCl_3) δ 7.37 – 7.08 (m, 5H), 6.48 (t, J = 7.6 Hz, 1H), 1.96 (q, J = 7.5 Hz, 2H), 1.43 – 1.31 (m, 2H), 0.85 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 143.7, 142.0, 139.0, 128.9, 128.7, 128.3, 128.0, 94.8, 39.8, 34.3, 22.5, 21.8, 14.0, 13.7.

(E/Z)-(1-Chloropent-1-en-1-yl)benzene ((E/Z)-A1-Cl)



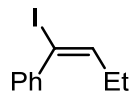
The reaction mixture was purified by column chromatography on silica gel (petroleum ether) to afford product (E/Z)-**A1-Cl** as a colorless oil (1.26 g, 70% yield, E/Z = 3.6/1).

^1H NMR of major product (400 MHz, CDCl_3) δ 7.46 – 7.30 (m, 6H), 5.99 (t, J = 7.8 Hz, 1H), 2.16 – 2.03 (m, 2H), 1.50 – 1.37 (m, 2H), 0.91 (t, J = 7.4 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 138.6, 137.5, 132.9, 130.5, 128.9, 128.5, 128.4, 128.3(1), 128.2(8), 128.1, 126.5, 31.9, 31.8, 22.9, 22.1, 14.0, 13.8.

HRMS (ESI) *m/z* calcd. for C₁₁H₁₄Cl [M + H]⁺ 181.0779, found 181.0778.

(*E*)-(1-Iodobut-1-en-1-yl)benzene ((*E*)-A2)

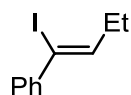


The reaction mixture was purified by column chromatography on silica gel (petroleum ether) to afford product (*E*)-A2 as a yellow oil (1.06 g, 82% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.41 – 7.10 (m, 5H), 6.47 (t, *J* = 7.7 Hz, 1H), 1.99 (p, *J* = 7.6 Hz, 2H), 0.95 (t, *J* = 7.5 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 145.1, 141.8, 128.8, 128.2, 128.0, 94.5, 25.8, 13.9.

(*Z*)-(1-Iodobut-1-en-1-yl)benzene ((*Z*)-A2)

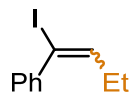


The reaction mixture was purified by column chromatography on silica gel (petroleum ether) to afford product (*Z*)-A2 as a yellow oil (1.29 g, 50% yield, *Z/E* = >20/1).

¹H NMR (400 MHz, CDCl₃) δ 7.49 – 7.40 (m, 2H), 7.34 – 7.21 (m, 3H), 5.88 (t, *J* = 6.7 Hz, 1H), 2.41 – 2.24 (m, 2H), 1.10 (t, *J* = 7.6 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 143.3, 140.5, 128.7, 128.3, 128.2, 104.4, 31.4, 12.9.

(*E/Z*)-(1-Iodobut-1-en-1-yl)benzene ((*E/Z*)-A2)

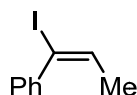


The reaction mixture was purified by column chromatography on silica gel (petroleum ether) to afford product (*E/Z*)-A2 as a yellow oil ((1.55 g, 60% yield, *E/Z* = 4.4/1).

¹H NMR (400 MHz, CDCl₃) δ 7.50 – 7.40 (m, 0.5H), 7.35 – 7.17 (m, 6H), 6.48 (t, *J* = 7.8 Hz, 1H), 5.88 (t, *J* = 6.7 Hz, 0.2H), 2.32 (p, *J* = 7.4 Hz, 0.5H), 2.00 (p, *J* = 7.5 Hz, 2H), 1.10 (t, *J* = 7.6 Hz, 0.7H), 0.95 (t, *J* = 7.5 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 145.1, 141.9, 140.5, 128.8, 128.7, 128.26, 128.1, 94.5, 31.4, 25.8, 13.9, 12.9.

(*E*)-(1-Iodoprop-1-en-1-yl)benzene (A3)

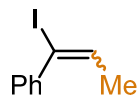


The reaction mixture was purified by column chromatography on silica gel (petroleum ether) to afford product A3 as a colorless oil (0.93 g, 76% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.36 – 7.22 (m, 5H), 6.55 (q, *J* = 7.1 Hz, 1H), 1.62 (d, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 141.6, 137.9, 129.0, 128.3, 128.1, 95.3, 77.4, 18.0.

(*E/Z*)-(1-Iodoprop-1-en-1-yl)benzene ((*E/Z*)-A3)

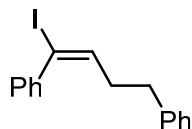


The reaction mixture was purified by column chromatography on silica gel (petroleum ether) to afford product (*E/Z*)-A3 as a colorless oil (1.26 g, 60% yield, *E/Z* = 2.8:1).

^1H NMR of major product (400 MHz, CDCl_3) δ 7.38 – 7.14 (m, 7H), 6.55 (q, J = 7.1 Hz, 1H), 1.62 (d, J = 7.2 Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 143.4, 141.5, 137.9, 133.7, 129.0, 128.6, 128.3, 128.2, 128.1, 106.8, 95.3, 23.7, 18.0.

(*E*)-(1-Iodobut-1-ene-1,4-diyl)dibenzene (A4)

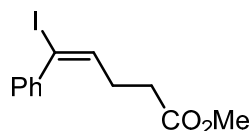


The reaction mixture was purified by column chromatography on silica gel (petroleum ether) to afford product A4 as a colorless oil (1.57 g, 94% yield).

^1H NMR (400 MHz, CDCl_3) δ 7.31 – 7.23 (m, 4H), 7.22 – 7.11 (m, 4H), 7.10 – 7.04 (m, 2H), 6.50 (t, J = 7.6 Hz, 1H), 2.66 (t, J = 7.6 Hz, 2H), 2.28 (q, J = 7.6 Hz, 2H).

^{13}C NMR (100 MHz, CDCl_3) δ 142.4, 141.8, 141.0, 128.7, 128.6, 128.5, 128.3, 128.1, 126.2, 95.7, 35.4, 34.1.

Methyl (*E*)-5-iodo-5-phenylpent-4-enoate (A5)



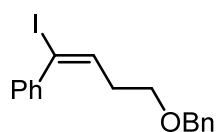
The reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 30:1) to afford product A5 as a colorless oil (0.95 g, 61% yield).

^1H NMR (400 MHz, CDCl_3) δ 7.41 – 7.17 (m, 5H), 6.46 (t, J = 7.3 Hz, 1H), 3.65 (s, 3H), 2.55 – 2.15 (m, 4H).

^{13}C NMR (100 MHz, CDCl_3) δ 172.8, 141.5, 140.9, 128.6, 128.4, 128.3, 96.5, 51.8, 33.2, 27.4.

HRMS (ESI) m/z calcd. for $\text{C}_{12}\text{H}_{13}\text{O}_2\text{NaI}$ $[\text{M} + \text{Na}]^+$ 338.9852, found 338.9852.

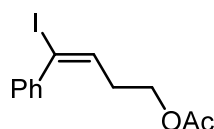
(*E*)-(4-(Benzyloxy)-1-iodobut-1-en-1-yl)benzene (A6)



The reaction mixture was purified by column chromatography on silica gel (petroleum

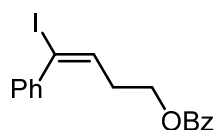
ether/ethyl acetate = 40:1) to afford product **A6** as a colorless oil (1.15 g, 63% yield).
¹H NMR (400 MHz, CDCl₃) δ 7.42 – 7.16 (m, 10H), 6.55 (t, *J* = 7.6 Hz, 1H), 4.46 (s, 2H), 3.45 (t, *J* = 6.5 Hz, 2H), 2.30 (q, *J* = 6.7 Hz, 2H).
¹³C NMR (100 MHz, CDCl₃) δ 141.7, 139.8, 138.3, 128.8, 128.5, 128.3, 128.2, 127.7(4), 127.7(2), 96.7, 73.0, 68.8, 32.7.
HRMS (ESI) *m/z* calcd. for C₁₇H₁₇ONaI [M + Na]⁺ 387.0216, found 387.0213.

(*E*)-4-Iodo-4-phenylbut-3-en-1-yl acetate (A7)



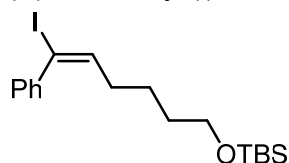
The reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 40:1) to afford product **A7** as a colorless oil (1.11 g, 70% yield).
¹H NMR (400 MHz, CDCl₃) δ 7.45 – 7.12 (m, 5H), 6.48 (t, *J* = 7.6 Hz, 1H), 4.03 (t, *J* = 6.6 Hz, 2H), 2.45 – 2.21 (m, 2H), 2.03 (s, 3H).
¹³C NMR (100 MHz, CDCl₃) δ 170.9, 141.4, 138.3, 128.5, 128.3(3), 128.2(9), 97.6, 62.8, 31.4, 21.0.
HRMS (ESI) *m/z* calcd. for C₁₂H₁₃O₂NaI [M + Na]⁺ 338.9852, found 338.9852.

(*E*)-4-Iodo-4-phenylbut-3-en-1-yl benzoate (A8)



The reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 60:1) to afford product **A8** as a colorless oil (1.42 g, 75% yield).
¹H NMR (400 MHz, CDCl₃) δ 8.02 (d, *J* = 6.9 Hz, 2H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.7 Hz, 2H), 7.36 – 7.19 (m, 5H), 6.58 (t, *J* = 7.6 Hz, 1H), 4.30 (t, *J* = 6.5 Hz, 2H), 2.46 (q, *J* = 6.8 Hz, 2H).
¹³C NMR (100 MHz, CDCl₃) δ 166.5, 141.6, 138.5, 133.2, 130.2, 129.7, 128.6, 128.5(2), 128.4(6), 128.4, 97.8, 63.4, 31.6.
HRMS (ESI) *m/z* calcd. for C₁₇H₁₅O₂NaI [M + Na]⁺ 401.0009, found 401.0009.

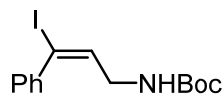
(*E*)-tert-Butyl((6-iodo-6-phenylhex-5-en-1-yl)oxy)dimethylsilane (A9)



The reaction mixture was purified by column chromatography on silica gel (petroleum ether) to afford product **A9** as a colorless oil (1.24 g, 60% yield).
¹H NMR (400 MHz, CDCl₃) δ 7.35 – 7.11 (m, 5H), 6.44 (t, *J* = 7.6 Hz, 1H), 3.49 (t, *J* = 5.8 Hz, 2H), 1.97 (q, *J* = 7.1 Hz, 2H), 1.30 – 1.46 (m, 4H), 0.84 (s, 9H), –0.02 (s, 6H).
¹³C NMR (100 MHz, CDCl₃) δ 143.6, 141.9, 128.8, 128.3, 128.0, 94.9, 62.8, 32.2, 32.0,

30.4, 26.1, 25.5, 18.4, -5.18.

***tert*-Butyl (*E*)-(3-iodo-3-phenylallyl)carbamate (**A10**)**



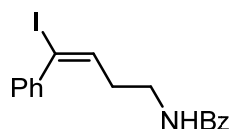
The reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 30:1) to afford product **A10** as a white solid (1.27 g, 71% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.38 – 7.19 (m, 5H), 6.52 (t, *J* = 6.9 Hz, 1H), 4.58 (s, 1H), 3.69 – 3.54 (m, 2H), 1.43 (s, 9H).

¹³C NMR (100 MHz, CDCl₃) δ 155.6, 141.0, 139.4, 128.7, 128.6, 128.5, 98.4, 79.8, 41.1, 28.5.

HRMS (ESI) *m/z* calcd. for C₁₄H₁₉NO₂I [M + H]⁺ 360.0455, found 360.0454.

(*E*)-*N*-(4-Iodo-4-phenylbut-3-en-1-yl)benzamide (A11**)**



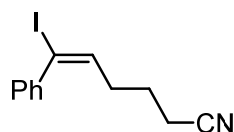
The reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 5:1) to afford product **A11** as a white solid (1.25 g, 66% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.71 (d, *J* = 7.0 Hz, 2H), 7.55 – 7.37 (m, 3H), 7.35 – 7.15 (m, 5H), 6.52 (t, *J* = 7.7 Hz, 1H), 6.18 (s, 1H), 3.46 (q, *J* = 6.6 Hz, 2H), 2.33 (q, *J* = 7.1 Hz, 2H).

¹³C NMR (100 MHz, CDCl₃) δ 167.6, 141.5, 139.6, 134.6, 131.7, 128.7, 128.6, 128.5, 128.4, 127.0, 97.6, 39.1, 32.3.

HRMS (ESI) *m/z* calcd. for C₁₇H₁₇NOI [M + H]⁺ 378.0349, found 378.0348.

(*E*)-6-Iodo-6-phenylhex-5-enenitrile (A12**)**



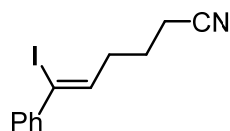
The reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 60:1) to afford product **A12** as a colorless oil (0.74 g, 50% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.39 – 7.20 (m, 5H), 6.42 (t, *J* = 7.6 Hz, 1H), 2.27 (t, *J* = 7.2 Hz, 2H), 2.14 (q, *J* = 7.5 Hz, 2H), 1.71 (p, *J* = 7.3 Hz, 2H).

¹³C NMR (100 MHz, CDCl₃) δ 141.4, 140.4, 128.5, 119.2, 97.2, 30.9, 24.9, 16.6.

HRMS (ESI) *m/z* calcd. for C₁₂H₁₃NI [M + H]⁺ 298.0087, found 298.0087.

(*Z*)-6-Iodo-6-phenylhex-5-enenitrile ((*Z*)-A12**)**



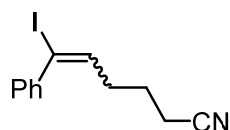
The reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 60:1) to afford product (Z)-**A12** as a colorless oil (1.81 g, 61% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.50 – 7.37 (m, 2H), 7.36 – 7.17 (m, 3H), 5.88 (t, *J* = 6.9 Hz, 1H), 2.53 – 2.32 (m, 2H), 1.94 – 1.84 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 142.8, 135.9, 128.6, 128.4, 119.5, 107.5, 36.6, 24.3, 16.8.

HRMS (ESI) *m/z* calcd. for C₁₂H₁₃NI [M + H]⁺ 298.0087, found 298.0087.

(*E/Z*)-6-Iodo-6-phenylhex-5-enenitrile ((*E/Z*)-A12**)**



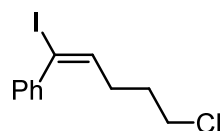
The *E/Z* mixed alkenyl iodide **A12** was obtained by mixing the (*E*)-**A12** and (*Z*)-**A12** alkenyl iodide, and the *E/Z* ratio was determined by ¹H NMR (*E/Z* = 1/1.3).

¹H NMR (400 MHz, CDCl₃) δ 7.53 – 7.37 (m, 2H), 7.38 – 7.19 (m, 7H), 6.42 (t, *J* = 7.6 Hz, 1H), 2.52 – 2.34 (m, 3H), 2.26 (t, *J* = 7.2 Hz, 2H), 2.13 (q, *J* = 7.5 Hz, 2H), 1.94 – 1.84 (m, 1H), 1.76 – 1.64 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 142.8, 141.4, 140.4, 135.9, 131.7, 128.6, 128.5, 128.4, 119.5, 119.2, 107.5, 97.2, 36.6, 30.9, 24.9, 24.4, 16.8, 16.5.

HRMS (ESI) *m/z* calcd. for C₁₂H₁₃NI [M + H]⁺ 298.0087, found 298.0086.

(*E*)-(5-Chloro-1-iodopent-1-en-1-yl)benzene (A13**)**

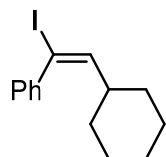


The reaction mixture was purified by column chromatography on silica gel (petroleum ether) to afford product **A13** as a colorless oil (0.95 g, 62% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.42 – 7.20 (m, 5H), 6.45 (t, *J* = 7.6 Hz, 1H), 3.46 (t, *J* = 6.6 Hz, 2H), 2.15 (q, *J* = 7.5 Hz, 2H), 1.92 – 1.77 (m, 2H).

¹³C NMR (100 MHz, CDCl₃) δ 141.6, 141.5, 128.7, 128.4, 128.3, 96.3, 44.0, 32.0, 29.5.

(*E*)-(2-Cyclohexyl-1-iodovinyl)benzene (A14**)**



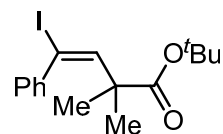
The reaction mixture was purified by column chromatography on silica gel (petroleum ether) to afford product **A14** as a colorless oil (0.95 g, 61% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.36 – 7.19 (m, 5H), 6.31 (d, *J* = 10.2 Hz, 1H), 2.09 – 1.95 (m, 1H), 1.73 – 1.50 (m, 5H), 1.20 – 1.01 (m, 5H).

¹³C NMR (100 MHz, CDCl₃) δ 149.2, 142.3, 128.6, 128.3, 128.0, 93.7, 41.3, 32.8, 25.8,

25.5.

***tert*-Butyl (*E*)-4-iodo-2,2-dimethyl-4-phenylbut-3-enoate (**A15-I**)**

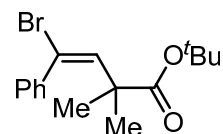


The reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 30:1) to afford product **A15-I** as a colorless oil (3.43 g, 92% yield). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.34 – 7.14 (m, 5H), 6.59 (s, 1H), 1.38 (s, 9H), 1.09 (s, 6H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 174.7, 146.2, 143.4, 128.3, 128.1(9), 128.1(5), 96.6, 80.8, 48.3, 28.0, 26.7.

HRMS (ESI) m/z calcd. for $\text{C}_{16}\text{H}_{22}\text{O}_2\text{I}$ $[\text{M} + \text{H}]^+$ 373.0659, found 373.0657.

***tert*-Butyl (*E*)-4-bromo-2,2-dimethyl-4-phenylbut-3-enoate (**A15-Br**)**



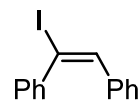
The reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 30:1) to afford product **A15-Br** as a colorless oil (3.06 g, 94% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.34 – 7.27 (m, 5H), 6.35 (s, 1H), 1.36 (s, 9H), 1.11 (s, 6H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 174.7, 139.9, 138.0, 128.9, 128.8, 128.2, 121.9, 80.8, 46.5, 27.9, 26.8.

HRMS (ESI) m/z calcd. for $\text{C}_{16}\text{H}_{22}\text{O}_2\text{Br}$ $[\text{M} + \text{H}]^+$ 325.0798, found 325.0796.

(*E*)-(1-Iodobut-1-ene-1,4-diyl)dibenzene (A16**)**

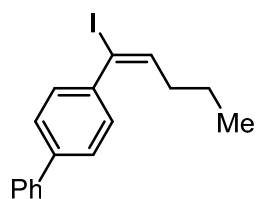


The reaction mixture was purified by column chromatography on silica gel (petroleum ether) to afford product **A16** as a yellow oil (1.37 g, 90% yield).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.42 (s, 1H), 7.36 – 7.20 (m, 5H), 7.16 – 7.05 (m, 3H), 6.95 – 6.85 (m, 2H).

$^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 143.1, 141.4, 137.3, 128.9, 128.8(1), 128.7(9), 128.5, 128.3, 127.7, 98.5.

(*E*)-4-(1-Iodopent-1-en-1-yl)-1,1'-biphenyl (A17**)**

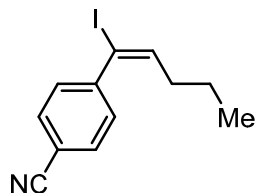


The reaction mixture was purified by column chromatography on silica gel (petroleum ether) to afford product **A17** as a colorless oil (1.41 g, 81% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.64 – 7.58 (m, 2H), 7.58 – 7.51 (m, 2H), 7.45 (t, *J* = 7.6 Hz, 2H), 7.40 – 7.32 (m, 3H), 6.52 (t, *J* = 7.7 Hz, 1H), 2.04 (q, *J* = 7.5 Hz, 2H), 1.42 (h, *J* = 7.4 Hz, 2H), 0.89 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 143.9, 140.9, 140.8, 140.6, 129.4, 129.0, 127.7, 127.2, 126.9, 94.6, 34.4, 22.6, 13.7.

(*E*)-4-(1-Iodopent-1-en-1-yl)benzonitrile (A18)



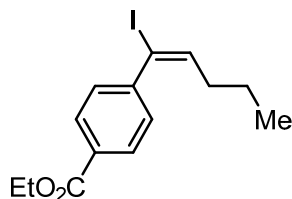
The reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 60:1) to afford product **A18** as a colorless oil (1.04 g, 70% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.61 (d, *J* = 8.3 Hz, 2H), 7.36 (d, *J* = 8.4 Hz, 2H), 6.54 (t, *J* = 7.7 Hz, 1H), 1.93 (q, *J* = 7.5 Hz, 2H), 1.43 – 1.32 (m, 2H), 0.84 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 146.4, 145.3, 132.1, 129.6, 118.5, 111.6, 91.3, 34.3, 22.3, 13.6.

HRMS (ESI) *m/z* calcd. for C₁₂H₁₃NI [M + H]⁺ 298.0087, found 298.0085.

Ethyl (*E*)-4-(1-iodopent-1-en-1-yl)benzoate (A19)



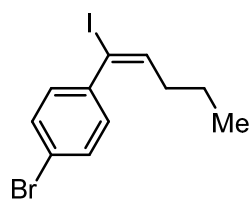
The reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 100:1) to afford product **A19** as a colorless oil (1.41 g, 82% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.99 (d, *J* = 8.3 Hz, 2H), 7.33 (d, *J* = 8.4 Hz, 2H), 6.52 (t, *J* = 7.7 Hz, 1H), 4.37 (q, *J* = 7.2 Hz, 2H), 1.94 (q, *J* = 7.5 Hz, 2H), 1.46 – 1.28 (m, 5H), 0.84 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 166.2, 146.3, 144.5, 129.9, 129.5, 128.9, 92.9, 61.2, 34.3, 22.4, 14.5, 13.7.

HRMS (ESI) *m/z* calcd. for C₁₄H₁₇NNaI [M + Na]⁺ 367.0165, found 367.0165.

(*E*)-1-Bromo-4-(1-iodopent-1-en-1-yl)benzene (A20)



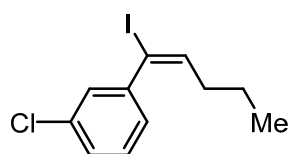
The reaction mixture was purified by column chromatography on silica gel (petroleum ether) to afford product **A20** as a colorless oil (0.91 g, 52% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.51 – 7.39 (m, 2H), 7.20 – 7.10 (m, 2H), 6.55 – 6.43 (m, 1H), 2.08 – 1.83 (m, 2H), 1.47 – 1.28 (m, 2H), 0.89 – 0.78 (m, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 144.4, 140.9, 131.5, 130.5, 122.1, 93.1, 34.4, 22.5, 13.8.

HRMS (ESI) *m/z* calcd. for C₁₁H₁₃BrI [M + H]⁺ 350.9240, found 350.9240.

(*E*)-1-Chloro-3-(1-iodopent-1-en-1-yl)benzene (**A21**)



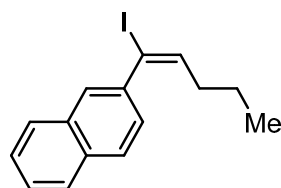
The reaction mixture was purified by column chromatography on silica gel (petroleum ether) to afford product **A21** as a colorless oil (0.97 g, 63% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.33 – 7.19 (m, 3H), 7.15 (d, *J* = 6.7 Hz, 1H), 6.49 (t, *J* = 7.7 Hz, 1H), 1.95 (q, *J* = 7.5 Hz, 2H), 1.38 (h, *J* = 7.4 Hz, 2H), 0.86 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 144.6, 143.6, 133.9, 129.5, 128.9, 128.2, 127.1, 92.3, 34.3, 22.4, 13.7.

HRMS (ESI) *m/z* calcd. for C₁₁H₁₃ClI [M + H]⁺ 306.9745, found 306.9744.

(*E*)-2-(1-Iodopent-1-en-1-yl)naphthalene (**A22**)

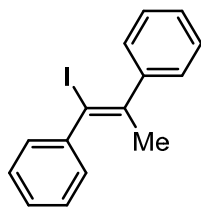


The reaction mixture was purified by column chromatography on silica gel (petroleum ether) to afford product **A22** as a colorless oil (1.14 g, 71% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.89 – 7.78 (m, 3H), 7.75 (s, 1H), 7.57 – 7.47 (m, 2H), 7.44 (dd, *J* = 8.5, 1.8 Hz, 1H), 6.59 (t, *J* = 7.7 Hz, 1H), 2.05 (q, *J* = 7.5 Hz, 2H), 1.43 (h, *J* = 7.4 Hz, 2H), 0.88 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 144.0, 139.3, 132.8(8), 132.8(6), 128.2, 128.0, 127.8, 127.5, 127.0, 126.6, 126.5, 95.0, 34.4, 22.5, 13.7.

(*Z*)-(1-Iodoprop-1-ene-1,2-diyl)dibenzene (**A24**)

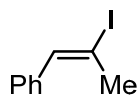


The reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 100:1) to afford product **A24** as a colorless oil (0.73 g, 23% yield). The *Z* configuration of **A24** was confirmed according to the two-dimensional NMR spectroscopy (2D NOESY, HSQC, and HMBC) analysis and the reported literature synthetic mechanism.¹⁴

¹H NMR (400 MHz, CDCl₃) δ 7.24 – 6.78 (m, 10H), 2.49 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 144.8, 144.6, 141.0, 130.1, 128.7, 128.0, 127.8, 127.2, 126.8, 100.2, 32.6.

(*E*)-(2-Iodoprop-1-en-1-yl)benzene (A26)

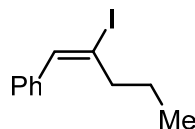


The reaction mixture was purified by column chromatography on silica gel (petroleum ether) to afford product **A26** as a colorless oil (2.22 g, 91% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.44 – 7.31 (m, 2H), 7.30 – 7.23 (m, 2H), 7.23 – 7.17 (m, 2H), 2.62 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 140.9, 137.6, 128.5, 128.4, 127.3, 98.7, 29.6.

(*E*)-(2-Iodopent-1-en-1-yl)benzene (A27)



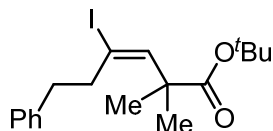
The reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 80:1) to afford product **A27** as a colorless oil (2.01 g, 73% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.38 – 7.21 (m, 4H), 7.17 (d, *J* = 6.4 Hz, 2H), 2.70 – 2.25 (m, 2H), 1.63 (h, *J* = 7.4 Hz, 2H), 0.92 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 140.9, 137.8, 128.5, 128.2, 127.3, 109.4, 41.2, 23.5, 13.1.

HRMS (ESI) *m/z* calcd. for C₂₀H₂₃NO [M + H]⁺ 273.0135, found 273.0132.

***tert*-Butyl (*E*)-4-iodo-2,2-dimethyl-6-phenylhex-3-enoate (A28)**



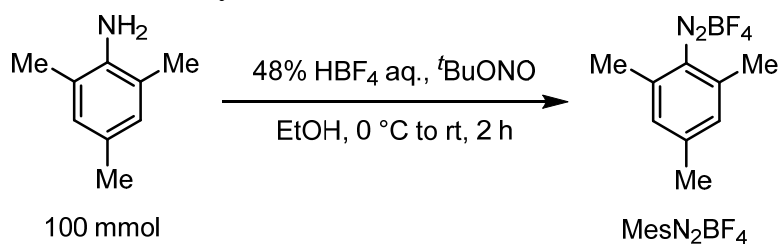
The reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 40:1) to afford product **A28** as a colorless oil (0.48 g, 12% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.33 – 7.24 (m, 2H), 7.24 – 7.13 (m, 3H), 6.28 (s, 1H), 2.89 – 2.75 (m, 2H), 2.59 – 2.48 (m, 2H), 1.41 (s, 9H), 1.22 (s, 6H).

¹³C NMR (100 MHz, CDCl₃) δ 175.3, 146.1, 140.5, 128.7, 128.6, 126.3, 106.5, 80.9, 46.9, 42.0, 36.3, 28.0, 27.2.

HRMS (ESI) *m/z* calcd. for C₁₈H₂₆O₂I [M + H]⁺ 401.0972, found 401.0972.

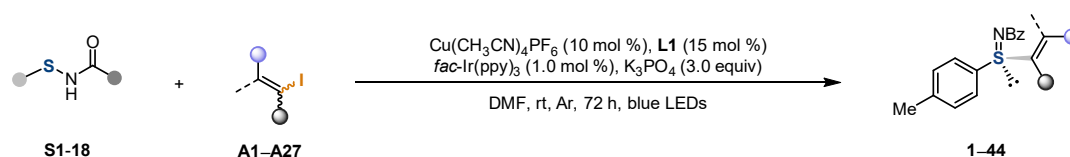
General procedure for the synthesis of diazonium salt¹⁵



In a 500 mL round-bottom flask, 2,4,6-trimethylaniline (13.5 g, 100 mmol, 1.0 equiv) was dissolved in a mixture of ethanol (200.0 mL) and an aqueous solution of HBF₄ (48%, 25.0 mL), followed by dropwise addition of ^tBuONO (27.0 mL) at 0 °C. Stirred at rt for 2 h, diethyl ether (200 mL) was added to precipitate the 2,4,6-trimethylbenzenediazonium tetrafluoroborate. After filtration and washing with diethyl ether (3×100 mL), the product was dried in vacuo for 10.0 minutes and stored in refrigerator under argon atmosphere.

5. General procedure for the synthesis of Chiral S-vinyl sulfilimines

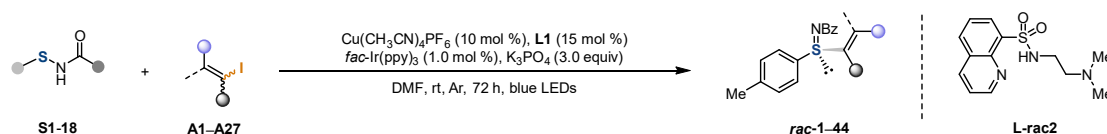
General procedure A:



Under an argon atmosphere, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with sulfenamide **S** (0.20 mmol, 1.0 equiv), alkenyl iodide **A** (0.3 mmol, 1.5 equiv), $\text{Cu}(\text{CH}_3\text{CN})_4\text{PF}_6$ (7.45 mg, 0.02 mmol, 10 mol %), **L1** (10.5 mg, 0.03 mmol, 15 mol %), *fac*- $\text{Ir}(\text{ppy})_3$ (1.30 mg, 1.0 mol %), and K_3PO_4 (127.4 mg, 0.6 mmol, 3.0 equiv) in DMF (2.0 mL) stirred at rt and blue LEDs irradiation for 72 h under argon. Upon completion, the reaction mixture was extracted three times with Et_2O and organic phase was evaporated. The residue was purified by column chromatography on silica gel to afford the desired product.



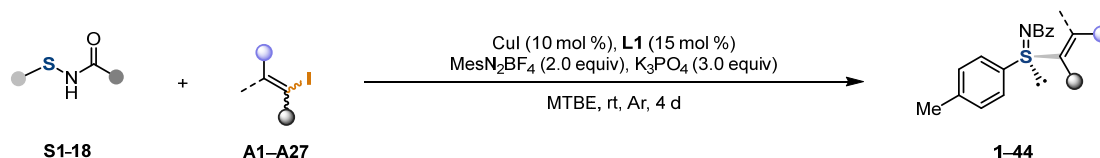
Figure S13. Assembling of setup for photochemical reaction.



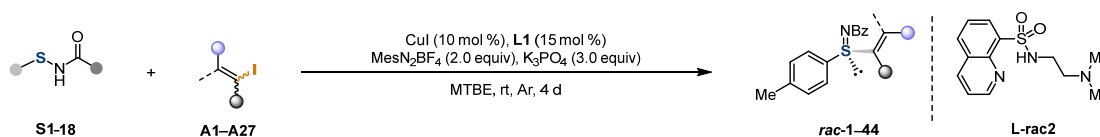
Under an argon atmosphere, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with sulfenamide **S** (0.20 mmol, 1.0 equiv), alkenyl iodide **A** (0.3 mmol, 1.5 equiv), $\text{Cu}(\text{CH}_3\text{CN})_4\text{PF}_6$ (7.45 mg, 0.02 mmol, 10 mol %), **L**-

rac2 (8.38 mg, 0.03 mmol, 15 mol %), *fac*-Ir(ppy)₃ (1.30 mg, 1.0 mol %), and K₃PO₄ (127.4 mg, 0.6 mol, 3.0 equiv) in DMF (2.0 mL) stirred at rt and blue LEDs irradiation for 72 h under argon. Upon completion, the reaction mixture was extracted three times with Et₂O and organic phase was evaporated. The residue was purified by column chromatography on silica gel to afford the desired product.

General procedure B:

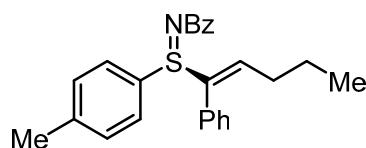


Under an argon atmosphere, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with sulfenamide **S** (0.20 mmol, 1.0 equiv), alkenyl iodide **A** (0.3 mmol, 1.5 equiv), CuI (3.80 mg, 0.02 mmol, 10 mol %), **L1** (10.5 mg, 0.03 mmol, 15 mol %), MesN₂BF₄ (93.6 mg, 0.40 mmol, 2.0 equiv) and K₃PO₄ (127.4 mg, 0.6 mol, 3.0 equiv) in MTBE (4.0 mL) stirred at rt for 4 days under argon. Upon completion, the precipitate was filtered off and washed by DCM. The filtrate was evaporated and the residue was purified by column chromatography on silica gel to afford the desired product.



Under an argon atmosphere, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with sulfenamide **S** (0.20 mmol, 1.0 equiv), alkenyl iodide **A** (0.3 mmol, 1.5 equiv), CuI (3.80 mg, 0.02 mmol, 10 mol %), **L-rac2** (8.38 mg, 0.03 mmol, 15 mol %), MesN₂BF₄ (93.6 mg, 0.40 mmol, 2.0 equiv) and K₃PO₄ (127.4 mg, 0.6 mol, 3.0 equiv) in MTBE (4.0 mL) stirred at rt for 4 days under argon. Upon completion, the precipitate was filtered off and washed by DCM. The filtrate was evaporated and the residue was purified by column chromatography on silica gel to afford the desired product.

***N*-((*R*)-((*E*)-1-Phenylpent-1-en-1-yl)(*p*-tolyl)- λ^4 -sulfaneylidene)benzamide (1)**



According to **General procedure A** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A1** (81.7 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **1** as a yellow oil (from **A1**, 62.0 mg, 80% yield, 94% ee; from (*E/Z*)-**A1** mixture, 60.5 mg, 78% yield, 93% ee)

¹H NMR (400 MHz, CDCl₃) δ 8.22 (d, *J* = 6.7 Hz, 2H), 7.49 – 7.34 (m, 5H), 7.34 – 7.21 (m, 3H), 7.16 (d, *J* = 8.1 Hz, 2H), 6.97 (d, *J* = 6.6 Hz, 2H), 6.83 (t, *J* = 7.6 Hz, 1H), 2.35 (s, 3H), 2.14 – 2.02 (m, 2H), 1.47 (h, *J* = 7.4 Hz, 2H), 0.87 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 176.3, 142.5, 140.1, 137.1, 136.7, 131.3, 130.8, 130.5, 130.3, 130.0, 129.0, 128.9, 128.2(8), 128.2(6), 127.9, 31.4, 22.2, 21.6, 13.9.

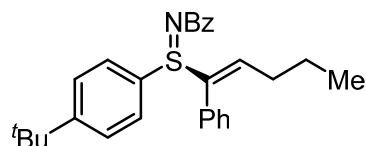
HRMS (ESI) *m/z* calcd. for C₂₅H₂₆NOS [M + H]⁺ 388.1730, found 388.1728.

HPLC analysis of **1** from (*E*)-**A1**: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (major) = 16.51 min, *t_R* (minor) = 18.22 min.

HPLC analysis of **1** from (*E/Z*)-**A1** mixture: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (major) = 16.50 min, *t_R* (minor) = 18.22 min.

HPLC analysis of **1** from (*Z*)-**A1**: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (major) = 18.65 min, *t_R* (minor) = 20.12 min.

***N*-((*R*)-(4-(*tert*-Butyl)phenyl)((*E*)-1-phenylpent-1-en-1-yl)- λ^4 -sulfaneylidene)benzamide (2)**



According to **General procedure A** with sulfenamide **S2** (57.1 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A1** (81.7 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 4:1) to afford product **2** as a yellow oil (65.3 mg, 76% yield, 94% ee).

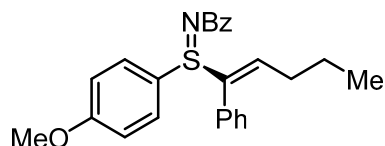
¹H NMR (400 MHz, CDCl₃) δ 8.23 (d, *J* = 6.7 Hz, 2H), 7.57 – 7.15 (m, 10H), 6.96 (d, *J* = 7.0 Hz, 2H), 6.84 (t, *J* = 7.6 Hz, 1H), 2.16 – 2.02 (m, 2H), 1.52 – 1.40 (m, 2H), 1.28 (s, 9H), 0.87 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 176.3, 155.5, 140.0, 137.1, 136.6, 131.3, 130.7, 130.4, 130.1, 128.9(3), 128.8(6), 128.2, 128.1, 127.8, 126.4, 35.1, 31.4, 31.2, 22.2, 13.8.

HRMS (ESI) *m/z* calcd. for C₂₈H₃₂NOS [M + H]⁺ 430.2199, found 430.2198.

HPLC analysis: Chiralpak IG (*n*-hexane/*i*-PrOH = 85/15, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (minor) = 47.82 min, *t_R* (major) = 64.41 min.

***N*-((*R*)-(4-Methoxyphenyl)((*E*)-1-phenylpent-1-en-1-yl)- λ^4 -sulfaneylidene)benzamide (3)**



According to **General procedure A** with sulfenamide **S3** (51.9 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A1** (81.7 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 2:1) to afford product **3** as a yellow oil (75.9 mg, 94% yield, 91% ee).

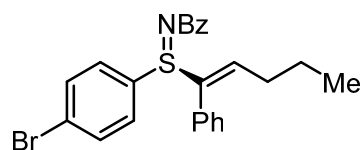
¹H NMR (400 MHz, CDCl₃) δ 8.22 (d, *J* = 6.8 Hz, 2H), 7.49 (d, *J* = 8.8 Hz, 2H), 7.47 – 7.34 (m, 3H), 7.32 – 7.20 (m, 3H), 6.97 (d, *J* = 8.6 Hz, 2H), 6.91 – 6.77 (m, 3H), 3.78 (s, 3H), 2.18 – 2.00 (m, 2H), 1.47 (h, *J* = 7.3 Hz, 2H), 0.86 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 176.2, 162.4, 139.6, 137.1, 136.8, 131.3, 130.7, 130.4, 130.2, 128.9, 128.9, 128.3, 127.9, 124.2, 114.8, 55.6, 31.4, 22.2, 13.8.

HRMS (ESI) *m/z* calcd. for C₂₅H₂₆NO₂S [M + H]⁺ 404.1679, found 404.1679.

HPLC analysis: Chiralpak IC (*n*-hexane/*i*-PrOH = 70/30, flow rate 1.0 mL/min, λ = 254 nm), *t_R* (minor) = 20.41 min, *t_R* (major) = 24.98 min.

***N*-((*R*)-(4-Bromophenyl))((*E*)-1-phenylpent-1-en-1-yl)-λ⁴-sulfaneylidenebenzamide (**4**)**



According to **General procedure A** with sulfenamide **S4** (61.7 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A1** (81.7 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **4** as a yellow oil (58.8 mg, 65% yield, 92% ee).

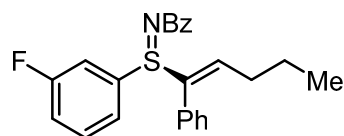
¹H NMR (400 MHz, CDCl₃) δ 8.21 (d, *J* = 6.8 Hz, 2H), 7.56 – 7.36 (m, 7H), 7.35 – 7.21 (m, 3H), 6.96 (d, *J* = 6.7 Hz, 2H), 6.88 (t, *J* = 7.7 Hz, 1H), 2.18 – 2.00 (m, 2H), 1.47 (h, *J* = 7.3 Hz, 2H), 0.87 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 176.3, 141.3, 136.7, 136.3, 132.9, 132.5, 131.0, 130.8, 130.4, 129.6, 129.2, 128.9, 128.4, 127.9, 126.5, 31.5, 22.1, 13.8.

HRMS (ESI) *m/z* calcd. for C₂₄H₂₃NOSBr [M + H]⁺ 452.0678, found 452.0676.

HPLC analysis: Chiralpak IC (*n*-hexane/*i*-PrOH = 70/30, flow rate 0.5 mL/min, λ = 220 nm), *t_R* (minor) = 23.60 min, *t_R* (major) = 29.32 min.

***N*-((*R*)-(3-Fluorophenyl))((*E*)-1-phenylpent-1-en-1-yl)-λ⁴-sulfaneylidenebenzamide (**5**)**



According to **General procedure A** with sulfenamide **S5** (49.5 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A1** (81.7 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was

purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **5** as a yellow oil (43.1 mg, 55% yield, 93% ee).

¹H NMR (400 MHz, CDCl₃) δ 8.23 (d, *J* = 6.8 Hz, 2H), 7.51 – 7.37 (m, 3H), 7.37 – 7.23 (m, 6H), 7.17 – 7.08 (m, 1H), 6.96 (d, *J* = 6.8 Hz, 2H), 6.89 (t, *J* = 7.7 Hz, 1H), 2.16 – 2.03 (m, 2H), 1.48 (h, *J* = 7.3 Hz, 2H), 0.87 (t, *J* = 7.4 Hz, 3H).

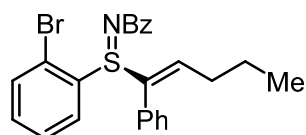
¹³C NMR (100 MHz, CDCl₃) δ 176.5, 162.7 (d, *J* = 251.6 Hz), 141.6, 136.6 (d, *J* = 25.6 Hz), 135.9 (d, *J* = 7.0 Hz), 131.0, 130.8, 130.7 (d, *J* = 3.3 Hz), 130.4, 129.2, 129.0, 128.4, 127.9, 119.0 (d, *J* = 21.5 Hz), 115.1 (d, *J* = 24.6 Hz), 31.5, 22.1, 13.8.

¹⁹F NMR (376 MHz, CDCl₃) δ –109.43.

HRMS (ESI) *m/z* calcd. for C₂₄H₂₃NOSF [M + H]⁺ 392.1479, found 392.1477.

HPLC analysis: Chiralpak IC (*n*-hexane/*i*-PrOH = 85/15, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (minor) = 36.43 min, *t_R* (major) = 42.44 min.

***N*-((*S*)-(2-Bromophenyl)((*E*)-1-phenylpent-1-en-1-yl)-λ⁴-sulfaneylidene)benzamide (**6**)**



According to **General procedure B** with sulfenamide **S6** (61.7 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A1** (81.7 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 4:1) to afford product **6** as a colorless oil (48.0 mg, 53% yield, 90% ee).

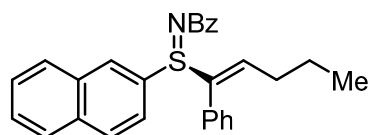
¹H NMR (400 MHz, CDCl₃) δ 8.22 (d, *J* = 6.9 Hz, 2H), 7.58 (d, *J* = 7.8 Hz, 1H), 7.51 – 7.34 (m, 4H), 7.31 – 7.13 (m, 5H), 7.01 (t, *J* = 7.6 Hz, 1H), 6.91 (d, *J* = 7.0 Hz, 2H), 2.16 – 1.98 (m, 2H), 1.49 (h, *J* = 7.2 Hz, 2H), 0.89 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 176.2, 146.0, 136.9, 134.4, 133.4, 133.2, 132.8, 130.9, 130.6, 130.3, 130.1, 128.9(4), 128.9(1), 128.1, 127.9(1), 127.8(9), 123.2, 32.0, 22.0, 13.9.

HRMS (ESI) *m/z* calcd. for C₂₄H₂₃NOSBr [M + H]⁺ 452.0678, found 452.0677.

HPLC analysis: Chiralpak IC (*n*-hexane/*i*-PrOH = 70/30, flow rate 1.0 mL/min, λ = 254 nm), *t_R* (minor) = 10.47 min, *t_R* (major) = 13.13 min.

***N*-((*R*)-Naphthalen-2-yl)((*E*)-1-phenylpent-1-en-1-yl)-λ⁴-sulfaneylidene)benzamide (**7**)**



According to **General procedure B** with sulfenamide **S7** (55.9 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A1** (81.7 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 4:1) to afford product **7** as a colorless oil (66.1 mg, 78% yield, 95% ee).

¹H NMR (400 MHz, CDCl₃) δ 8.28 (d, *J* = 6.4 Hz, 2H), 8.04 (s, 1H), 7.84 (d, *J* = 8.7

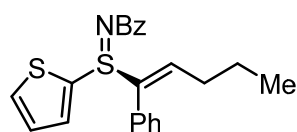
Hz, 1H), 7.81 (d, J = 8.0 Hz, 1H), 7.75 (d, J = 8.0 Hz, 1H), 7.63 (dd, J = 8.7, 1.8 Hz, 1H), 7.58 – 7.36 (m, 5H), 7.28 – 7.10 (m, 3H), 7.07 – 6.88 (m, 3H), 2.18 – 2.04 (m, 2H), 1.48 (h, J = 7.4 Hz, 2H), 0.87 (t, J = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 176.4, 140.5, 137.0, 136.3, 134.6, 132.6, 131.1, 130.8, 130.3, 129.6(0), 129.5(7), 128.9(4), 128.9(2), 128.7, 128.3, 128.2, 127.9(4), 127.8(6), 127.2, 123.1, 31.4, 22.2, 13.8.

HRMS (ESI) m/z calcd. for $\text{C}_{28}\text{H}_{26}\text{NOS}$ $[\text{M} + \text{H}]^+$ 424.1729, found 424.1729.

HPLC analysis: Chiralpak OD-H (n -hexane/ i -PrOH = 85/15, flow rate 0.5 mL/min, λ = 254 nm), t_{R} (minor) = 10.32 min, t_{R} (major) = 10.95 min.

***N*-((*S*)-((*E*)-1-Phenylpent-1-en-1-yl)(thiophen-2-yl)- λ^4 -sulfaneylidene)benzamide (8)**



According to **General procedure B** with sulfenamide **S8** (47.1 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A1** (81.7 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 4:1) to afford product **8** as a colorless oil (45.6 mg, 60% yield, 90% ee).

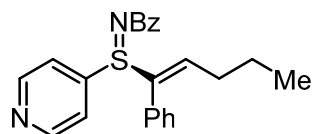
^1H NMR (400 MHz, CDCl_3) δ 8.22 (d, J = 6.8 Hz, 2H), 7.56 (d, J = 5.1 Hz, 1H), 7.49 – 7.36 (m, 3H), 7.36 – 7.21 (m, 4H), 7.06 (d, J = 6.5 Hz, 2H), 6.96 (dd, J = 5.1, 3.8 Hz, 1H), 6.80 (t, J = 7.7 Hz, 1H), 2.18 – 2.04 (m, 2H), 1.46 (h, J = 7.4 Hz, 2H), 0.86 (t, J = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 139.4, 137.7, 136.5, 134.1, 132.9, 132.4, 131.0, 130.9, 130.3, 129.1, 129.0, 128.4, 127.9, 127.3.

HRMS (ESI) m/z calcd. for $\text{C}_{22}\text{H}_{22}\text{NOS}_2$ $[\text{M} + \text{H}]^+$ 380.1137, found 380.1137.

HPLC analysis: Chiralpak OD-H (n -hexane/ i -PrOH = 90/10, flow rate 0.5 mL/min, λ = 254 nm), t_{R} (major) = 11.91 min, t_{R} (minor) = 13.55 min.

***N*-((*R*)-((*E*)-1-Phenylpent-1-en-1-yl)(pyridin-4-yl)- λ^4 -sulfaneylidene)benzamide (9)**



According to **General procedure B** with sulfenamide **S9** (46.1 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A1** (81.7 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 1:2) to afford product **9** as a colorless oil (63.0 mg, 84% yield, 94% ee).

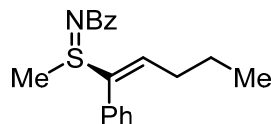
^1H NMR (400 MHz, CDCl_3) δ 8.63 (d, J = 5.4 Hz, 2H), 8.23 (d, J = 6.9 Hz, 2H), 7.58 – 7.39 (m, 5H), 7.38 – 7.22 (m, 3H), 7.04 – 6.89 (m, 3H), 2.18 – 2.08 (m, 2H), 1.50 (h, J = 7.4 Hz, 2H), 0.89 (t, J = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 176.7, 150.6, 144.2, 143.3, 136.4, 135.8, 131.2, 130.4, 129.5, 129.0, 128.5, 128.0, 127.5, 121.7, 31.7, 22.2, 13.9.

HRMS (ESI) m/z calcd. for $C_{23}H_{23}N_2OS$ $[M + H]^+$ 375.1526, found 375.1525.

HPLC analysis: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 85/15, flow rate 0.5 mL/min, λ = 254 nm), t_R (minor) = 15.43 min, t_R (major) = 21.30 min.

***N*-((*R*)-Methyl((*E*)-1-phenylpent-1-en-1-yl)- λ^4 -sulfaneylidene)benzamide (10)**



According to **General procedure A** with sulfenamide **S10** (33.5 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A1** (81.7 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 2:1) to afford product **10** as a yellow oil (30.0 mg, 48% yield, 75% ee).

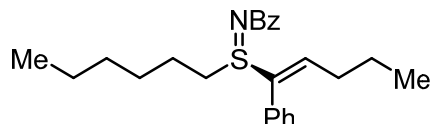
¹H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, J = 6.9 Hz, 2H), 7.51 – 7.28 (m, 8H), 6.64 (t, J = 7.7 Hz, 1H), 2.51 (s, 3H), 2.19 – 2.05 (m, 2H), 1.45 (h, J = 7.3 Hz, 2H), 0.86 (t, J = 7.4 Hz, 3H).

¹³C NMR (100 MHz, $CDCl_3$) δ 176.9, 140.6, 136.8, 134.7, 131.1, 130.8, 130.1, 129.4, 128.9, 128.7, 127.9, 31.4, 30.5, 22.2, 13.8.

HRMS (ESI) m/z calcd. for $C_{19}H_{22}NOS$ $[M + H]^+$ 312.1417, found 312.1415.

HPLC analysis: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 85/15, flow rate 0.5 mL/min, λ = 254 nm), t_R (major) = 17.36 min, t_R (minor) = 20.01 min.

***N*-((*R*)-Hexyl((*E*)-1-phenylpent-1-en-1-yl)- λ^4 -sulfaneylidene)benzamide (11)**



According to **General procedure A** with sulfenamide **S11** (47.5 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A1** (81.7 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 4:1) to afford product **11** as a yellow oil (61.8 mg, 81% yield, 90% ee).

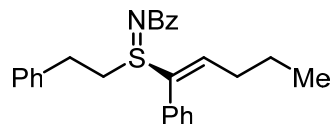
¹H NMR (400 MHz, $CDCl_3$) δ 8.16 (d, J = 6.8 Hz, 2H), 7.53 – 7.29 (m, 8H), 6.57 (t, J = 7.7 Hz, 1H), 2.87 – 2.60 (m, 2H), 2.23 – 2.04 (m, 2H), 1.68 (p, J = 7.6 Hz, 2H), 1.57 – 1.13 (m, 8H), 0.95 – 0.74 (m, 6H).

¹³C NMR (100 MHz, $CDCl_3$) δ 177.0, 141.0, 137.2, 133.3, 131.5, 130.7, 130.0, 129.3, 128.9, 128.8, 127.9, 44.5, 31.4, 31.3, 28.1, 23.2, 22.4, 22.3, 14.0, 13.8.

HRMS (ESI) m/z calcd. for $C_{24}H_{32}NOS$ $[M + H]^+$ 382.2199, found 382.2197.

HPLC analysis: Chiralpak IC (*n*-hexane/*i*-PrOH = 85/15, flow rate 0.5 mL/min, λ = 254 nm), t_R (major) = 31.57 min, t_R (minor) = 37.86 min.

***N*-((*R*)-Phenethyl((*E*)-1-phenylpent-1-en-1-yl)- λ^4 -sulfaneylidene)benzamide (12)**



According to **General procedure A** with sulfenamide **S12** (51.5 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A1** (81.7 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **12** as a yellow oil (48.2 mg, 60% yield, 92% ee).

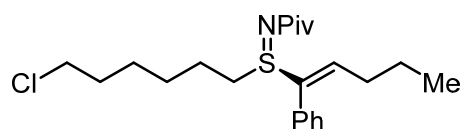
¹H NMR (400 MHz, CDCl₃) δ 8.22 (d, *J* = 6.9 Hz, 2H), 7.54 – 7.17 (m, 11H), 7.10 (d, *J* = 6.7 Hz, 2H), 6.60 (t, *J* = 7.7 Hz, 1H), 3.06 – 2.93 (m, 4H), 1.54 – 1.38 (m, 2H), 1.47 (h, *J* = 7.3 Hz, 2H), 0.88 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 177.0, 141.1, 138.2, 137.0, 132.8, 131.5, 130.8, 129.9, 129.4, 129.0, 128.9, 128.6, 127.9, 127.0, 45.4, 31.4, 29.3, 22.3, 13.9.

HRMS (ESI) *m/z* calcd. for C₂₆H₂₈NOS [M + H]⁺ 402.1886, found 402.1886.

HPLC analysis: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 85/15, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (minor) = 13.67 min, *t_R* (major) = 21.27 min.

***N*-((*R*)-(6-Chlorohexyl)((*E*)-1-phenylpent-1-en-1-yl)-λ⁴-sulfaneylidene)pivalamide (13)**



According to **General procedure B** with sulfenamide **S13** (50.4 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A1** (81.7 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **13** as a colorless oil (67.2 mg, 85% yield, 94% ee).

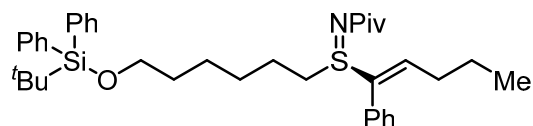
¹H NMR (400 MHz, CDCl₃) δ 7.49 – 7.38 (m, 3H), 7.34 – 7.23 (m, 2H), 6.43 (t, *J* = 7.6 Hz, 1H), 3.49 (t, *J* = 6.6 Hz, 2H), 2.78 – 2.51 (m, 2H), 2.20 – 2.07 (m, 2H), 1.80 – 1.54 (m, 4H), 1.51 – 1.30 (m, 6H), 1.25 (s, 9H), 0.88 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 190.7, 139.5, 133.7, 131.9, 129.9, 129.2, 128.9, 44.9, 44.2, 40.3, 32.2, 31.2, 28.8, 27.7, 26.4, 23.2, 22.3, 13.8.

HRMS (ESI) *m/z* calcd. for C₂₂H₃₅NOSCl [M + H]⁺ 396.2122, found 396.2121.

HPLC analysis: Chiralpak IC (*n*-hexane/*i*-PrOH = 70/30, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (major) = 10.63 min, *t_R* (minor) = 15.93 min.

***N*-((*R*)-(6-((*Tert*-butyldiphenylsilyl)oxy)hexyl)((*E*)-1-phenylpent-1-en-1-yl)-λ⁴-sulfaneylidene)pivalamide (14)**



According to **General procedure B** with sulfenamide **S14** (94.3 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A1** (81.7 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **14** as a colorless oil (103.4 mg, 84% yield, 94% ee).

¹H NMR (400 MHz, CDCl₃) δ 7.70 – 7.54 (m, 4H), 7.45 – 7.33 (m, 9H), 7.31 – 7.26 (m, 2H), 6.42 (t, *J* = 7.6 Hz, 1H), 3.61 (t, *J* = 6.4 Hz, 2H), 2.68 – 2.50 (m, 2H), 2.19 –

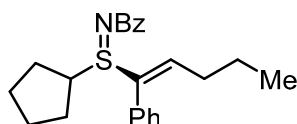
2.07 (m, 2H), 1.66 – 1.52 (m, 2H), 1.52 – 1.37 (m, 4H), 1.35 – 1.26 (m, 4H), 1.26 (s, 9H), 1.03 (s, 9H), 0.88 (t, $J = 7.4$ Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 190.6, 139.4, 135.6, 134.1, 133.9, 131.9, 129.9, 129.6, 129.1, 128.8, 127.7, 63.7, 44.3, 40.3, 32.3, 31.2, 28.8, 28.1, 27.0, 25.4, 23.3, 22.3, 19.3, 13.8.

HRMS (ESI) m/z calcd. for $\text{C}_{38}\text{H}_{54}\text{NSiO}_2\text{S}$ $[\text{M} + \text{H}]^+$ 616.3639, found 616.3635.

HPLC analysis: Chiralpak IC (n -hexane/ i -PrOH = 85/15, flow rate 0.5 mL/min, $\lambda = 254$ nm), t_R (major) = 10.80 min, t_R (minor) = 13.27 min.

***N*-((*R*)-((*E*)-1,4-Diphenylbut-1-en-1-yl)(*p*-tolyl)- λ^4 -sulfaneylidene)benzamide (**15**)**



According to **General procedure A** with sulfenamide **S15** (44.3 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A1** (81.7 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 4:1) to afford product **15** as a yellow oil (51.2 mg, 70% yield, 90% ee).

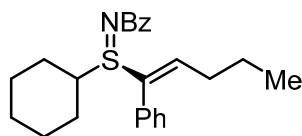
^1H NMR (400 MHz, CDCl_3) δ 8.18 (d, $J = 6.7$ Hz, 2H), 7.49 – 7.27 (m, 8H), 6.53 (t, $J = 7.7$ Hz, 1H), 3.22 – 3.05 (m, 1H), 2.22 – 2.06 (m, 3H), 1.93 – 1.68 (m, 5H), 1.67 – 1.52 (m, 2H), 1.45 (h, $J = 7.4$ Hz, 2H), 0.86 (t, $J = 7.4$ Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 177.3, 141.4, 137.5, 133.7, 132.0, 130.6, 129.9, 129.1, 128.9, 128.8, 127.8, 53.6, 31.3, 28.5, 27.2, 26.3, 25.8, 22.3, 13.9.

HRMS (ESI) m/z calcd. for $\text{C}_{23}\text{H}_{28}\text{NOS}$ $[\text{M} + \text{H}]^+$ 366.1866, found 366.1884.

HPLC analysis: Chiralpak IG (n -hexane/ i -PrOH = 70/30, flow rate 1.0 mL/min, $\lambda = 254$ nm), t_R (major) = 11.40 min, t_R (minor) = 12.58 min.

***N*-((*R*)-Cyclohexyl((*E*)-1-phenylpent-1-en-1-yl)- λ^4 -sulfaneylidene)benzamide (**16**)**



According to **General procedure A** with sulfenamide **S16** (47.1 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A1** (81.7 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **16** as a yellow oil (62.3 mg, 82% yield, 92% ee).

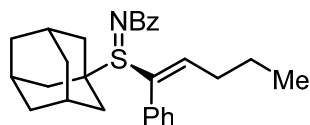
^1H NMR (400 MHz, CDCl_3) δ 8.19 (d, $J = 6.6$ Hz, 2H), 7.55 – 7.30 (m, 8H), 6.50 (t, $J = 7.7$ Hz, 1H), 2.73 – 2.59 (m, 1H), 2.37 – 1.73 (m, 7H), 1.70 – 1.31 (m, 5H), 1.31 – 1.08 (m, 3H), 0.86 (t, $J = 7.4$ Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 177.1, 142.0, 137.5, 132.1, 131.6, 130.6, 129.9, 129.1, 128.8(8), 128.8(5), 127.8, 53.2, 31.4, 27.9, 25.6, 25.4, 22.4, 13.9.

HRMS (ESI) m/z calcd. for $\text{C}_{24}\text{H}_{30}\text{NOS}$ $[\text{M} + \text{H}]^+$ 380.2043, found 380.2039.

HPLC analysis: Chiralpak IH (n -hexane/ i -PrOH = 85/15, flow rate 0.5 mL/min, $\lambda = 254$ nm), t_R (minor) = 9.00 min, t_R (major) = 15.72 min.

***N*-((*R*)-((3*S*,5*S*,7*S*)-Adamantan-1-yl)((*E*)-1-phenylpent-1-en-1-yl)- λ^4 -sulfaneylidene)benzamide (17)**



According to **General procedure A** with sulfenamide **S17** (57.5 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A1** (81.7 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 4:1) to afford product **17** as a yellow oil (61.3 mg, 71% yield, 72% ee).

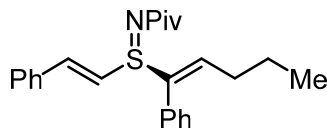
¹H NMR (400 MHz, CDCl₃) δ 8.25 (d, J = 6.3 Hz, 2H), 7.54 (d, J = 7.1 Hz, 2H), 7.47 – 7.30 (m, 6H), 6.41 (t, J = 7.7 Hz, 1H), 2.33 – 2.12 (m, 2H), 2.11 – 2.00 (m, 3H), 1.93 – 1.78 (m, 6H), 1.64 (q, J = 12.4 Hz, 6H), 1.43 (h, J = 7.3 Hz, 2H), 0.84 (t, J = 7.3 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 176.4, 140.6, 137.7, 135.1, 130.4, 120.0, 129.7, 128.8, 128.7, 128.7, 127.7, 58.2, 37.2, 36.1, 31.1, 29.3, 22.6, 13.8.

HRMS (ESI) m/z calcd. for C₂₈H₃₄NOS [M + H]⁺ 432.2356, found 432.2353.

HPLC analysis: Chiralpak IH (*n*-hexane/*i*-PrOH = 85/15, flow rate 0.5 mL/min, λ = 254 nm), t_R (minor) = 9.30 min, t_R (major) = 11.76 min.

***N*-((*R*)-((*E*)-1-Phenylpent-1-en-1-yl)((*E*)-styryl)- λ^4 -sulfaneylidene)pivalamide (18)**



According to **General procedure A** with sulfenamide **S18** (47.1 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A1** (81.7 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 2:1) to afford product **18** as a yellow oil (57.7 mg, 76% yield, 75% ee).

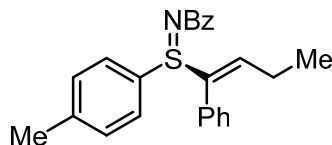
¹H NMR (400 MHz, CDCl₃) δ 7.46 – 7.25 (m, 10H), 7.06 (d, J = 15.5 Hz, 1H), 6.53 (t, J = 7.6 Hz, 1H), 6.47 (d, J = 15.5 Hz, 1H), 2.18 – 2.06 (m, 2H), 1.52 – 1.38 (m, 2H), 1.28 (s, 9H), 0.88 (t, J = 7.4 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 190.3, 141.3, 138.4, 135.8, 133.9, 131.8, 130.3, 130.2, 129.1, 129.0, 128.6, 127.8, 121.2, 77.4, 40.4, 31.3, 28.8, 22.3, 13.8.

HRMS (ESI) m/z calcd. for C₂₄H₃₀NOS [M + H]⁺ 380.2043, found 380.2039.

HPLC analysis: Chiralpak IG (*n*-hexane/*i*-PrOH = 85/15, flow rate 0.5 mL/min, λ = 270 nm), t_R (major) = 24.57 min, t_R (minor) = 26.72 min.

***N*-((*R*)-((*E*)-1-phenylbut-1-en-1-yl)(*p*-tolyl)- λ^4 -sulfaneylidene)benzamide (19)**



According to **General procedure A** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A2** (77.5 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **19** as a yellow oil (from (*E*)-**A2**, 52.3 mg, 70% yield, 93% ee; from (*Z*)-**A2**, 59.7 mg, 80% yield, 92% ee, from (*E/Z*)-**A2** mixture, 55.5 mg, 73% yield, 93% ee).

¹H NMR (400 MHz, CDCl₃) δ 8.23 (d, *J* = 6.7 Hz, 2H), 7.49 – 7.35 (m, 5H), 7.32 – 7.20 (m, 3H), 7.16 (d, *J* = 8.0 Hz, 2H), 6.97 (d, *J* = 6.7 Hz, 2H), 6.83 (t, *J* = 7.6 Hz, 1H), 2.34 (s, 3H), 2.17 – 2.06 (m, 2H), 1.03 (t, *J* = 7.5 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 176.3, 142.4, 141.4, 137.1, 136.1, 131.2, 130.8, 130.4, 130.2, 130.0, 128.9(4), 128.9(2), 128.3, 128.2, 127.9, 23.0, 21.6, 13.6.

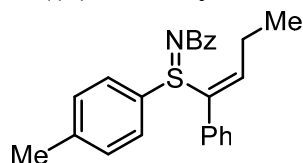
HRMS (ESI) *m/z* calcd. for C₂₄H₂₄NOS [M + H]⁺ 374.1573, found 374.1570.

HPLC analysis of **19** from (*E*)-**A2**: Chiralpak IH (*n*-hexane/*i*-PrOH = 70/30, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (minor) = 8.87 min, *t_R* (major) = 22.17 min.

HPLC analysis of **19** from (*Z*)-**A2**: Chiralpak IH (*n*-hexane/*i*-PrOH = 70/30, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (minor) = 8.87 min, *t_R* (major) = 22.33 min.

HPLC analysis of **19** from (*E/Z*)-**A2** mixture: Chiralpak IH (*n*-hexane/*i*-PrOH = 70/30, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (minor) = 8.94 min, *t_R* (major) = 22.71 min.

N-((*Z*)-1-Phenylbut-1-en-1-yl)(*p*-tolyl)-λ⁴-sulfaneylidene)benzamide (**19'**)



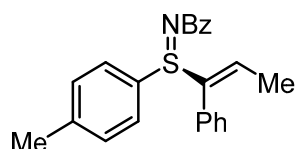
According to **General procedure B** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide (*Z*)-**A2** (77.5 mg, 0.30 mmol, 1.5 equiv) and **L-rac2** as ligand, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **19'** as a yellow oil (3.7 mg, 5.0% yield).

¹H NMR (400 MHz, CDCl₃) δ 8.21 (d, *J* = 6.8 Hz, 2H), 7.49 – 7.34 (m, 5H), 7.25 – 7.20 (m, 1H), 7.20 – 7.10 (m, 4H), 7.07 – 7.00 (m, 2H), 6.35 (t, *J* = 7.6 Hz, 1H), 3.07 – 2.92 (m, 2H), 2.34 (s, 3H), 1.29 (t, *J* = 7.5 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 176.5, 147.1, 142.0, 137.1, 136.6, 133.8, 130.7, 130.2, 130.0, 129.0, 128.9, 128.6, 127.9, 127.6, 23.3, 21.5, 14.0

HRMS (ESI) *m/z* calcd. for C₂₄H₂₄NOS [M + H]⁺ 374.1573, found 374.1572.

N-((*R*)-((*E*)-1-Phenylprop-1-en-1-yl)(*p*-tolyl)-λ⁴-sulfaneylidene)benzamide (**20**)



According to **General procedure A** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A3** (73.2 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **20** as a yellow oil (from **A3**, 53.2 mg, 74% yield, 91% ee; from (*E/Z*)-

A3 mixture, 48.9 mg, 68% yield, 90% ee).

¹H NMR (400 MHz, CDCl₃) δ 8.23 (d, *J* = 6.8 Hz, 2H), 7.51 – 7.34 (m, 5H), 7.33 – 7.21 (m, 3H), 7.16 (d, *J* = 7.9 Hz, 2H), 7.02 – 6.96 (m, 2H), 6.90 (q, *J* = 7.1 Hz, 1H), 2.34 (s, 3H), 1.78 (d, *J* = 7.0 Hz, 3H).

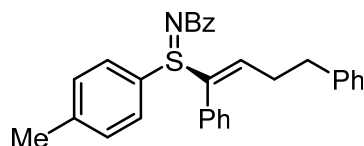
¹³C NMR (100 MHz, CDCl₃) δ 176.3, 142.5, 137.2, 137.0, 134.9, 131.1, 130.8, 130.4, 130.0, 129.0, 128.4, 128.3, 127.9, 21.6, 15.4.

HRMS (ESI) *m/z* calcd. for C₂₃H₂₂NOS [M + H]⁺ 360.1417, found 360.1413.

HPLC analysis of **20** from **A3**: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (major) = 26.71 min, *t_R* (minor) = 30.03 min.

HPLC analysis of **20** from (*E/Z*)-**A3** mixture: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (major) = 26.40 min, *t_R* (minor) = 29.25 min.

N-((*R*)-((*E*)-1,4-Diphenylbut-1-en-1-yl)(*p*-tolyl)-λ⁴-sulfaneylidene)benzamide (**21**)



According to **General procedure A** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A4** (100.3 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 4:1) to afford product **21** as a yellow oil (66.6 mg, 74% yield, 91% ee).

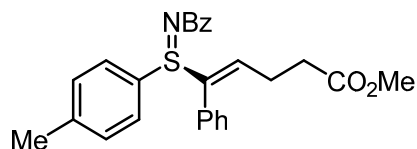
¹H NMR (400 MHz, CDCl₃) δ 8.22 (d, *J* = 8.3 Hz, 2H), 7.50 – 7.31 (m, 5H), 7.27 – 7.04 (m, 10H), 6.83 (t, *J* = 7.6 Hz, 1H), 6.74 (d, *J* = 7.0 Hz, 2H), 2.79 – 2.69 (m, 2H), 2.41 (q, *J* = 7.4 Hz, 2H), 2.33 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 176.2, 142.4, 140.5, 138.3, 137.1, 137.0, 131.0, 130.8, 130.2, 130.0, 129.9, 129.0, 128.9, 128.8, 128.5, 128.3, 128.2, 127.8, 126.2, 35.0, 31.2, 21.5.

HRMS (ESI) *m/z* calcd. for C₃₀H₂₈NOS [M + H]⁺ 450.1886, found 450.1884.

HPLC analysis: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 85/15, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (minor) = 15.21 min, *t_R* (major) = 17.34 min.

Methyl (*E*)-5-((*R*)-*N*-benzoyl-*S*-(*p*-tolyl)sulfinimidoyl)-5-phenylpent-4-enoate (**22**)

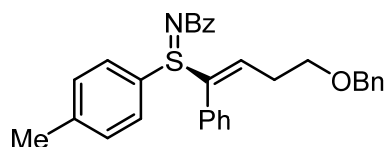


According to **General procedure A** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A5** (94.8 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 1:1) to afford product **22** as a yellow oil (54.4 mg, 63% yield, 94% ee).

¹H NMR (400 MHz, CDCl₃) δ 8.22 (d, *J* = 6.8 Hz, 2H), 7.49 – 7.35 (m, 5H), 7.34 – 7.22 (m, 3H), 7.17 (d, *J* = 8.1 Hz, 2H), 6.99 (d, *J* = 6.6 Hz, 2H), 6.85 – 6.76 (m, 1H), 3.63 (s, 3H), 2.47 – 2.39 (m, 4H), 2.35 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 176.3, 172.7, 142.7, 138.0, 137.0(1), 136.9(6), 130.9(3), 130.8(5), 130.3, 130.1, 129.9, 129.2, 129.0, 128.5, 128.4, 127.9, 51.9, 33.0, 24.8, 21.6.
HRMS (ESI) m/z calcd. for $\text{C}_{26}\text{H}_{26}\text{NO}_3\text{S}$ $[\text{M} + \text{H}]^+$ 432.1628, found 432.1626.
HPLC analysis: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 85/15, flow rate 0.5 mL/min, λ = 254 nm), t_R (major) = 23.92 min, t_R (minor) = 30.47 min.

***N*-((*R*)-((*E*)-4-(Benzyloxy)-1-phenylbut-1-en-1-yl)(*p*-tolyl)- λ^4 -sulfaneylidene)benzamide (**23**)**



According to **General procedure A** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A6** (109.3 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 2:1) to afford product **23** as a yellow oil (48.9 mg, 51% yield, 93% ee).

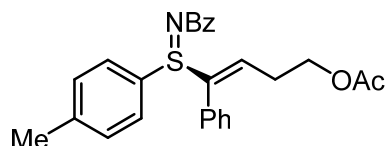
^1H NMR (400 MHz, CDCl_3) δ 8.23 (d, J = 6.8 Hz, 2H), 7.52 – 7.34 (m, 6H), 7.34 – 7.19 (m, 7H), 7.10 (d, J = 8.0 Hz, 2H), 7.03 (d, J = 6.8 Hz, 2H), 6.91 (t, J = 7.5 Hz, 1H), 4.47 (s, 2H), 3.62 – 3.49 (m, 2H), 2.57 – 2.33 (m, 2H), 2.32 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 176.3, 142.5, 138.2, 137.8, 137.0, 136.3, 131.1, 130.8, 130.4, 130.0, 129.0(1), 128.9(8), 128.5, 128.4, 128.3, 127.9, 127.7(3), 127.7(0), 73.1, 68.6, 30.2, 21.6.

HRMS (ESI) m/z calcd. for $\text{C}_{31}\text{H}_{30}\text{NO}_2\text{S}$ $[\text{M} + \text{H}]^+$ 480.1992, found 480.1991.

HPLC analysis: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 85/15, flow rate 0.5 mL/min, λ = 254 nm), t_R (major) = 24.17 min, t_R (minor) = 35.14 min.

***(E)*-4-((*R*)-*N*-Benzoyl-*S*-(*p*-tolyl)sulfinimidoyl)-4-phenylbut-3-en-1-yl acetate (**24**)**



According to **General procedure B** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A7** (94.9 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 1:1) to afford product **24** as a yellow oil (69.9 mg, 81% yield, 93% ee).

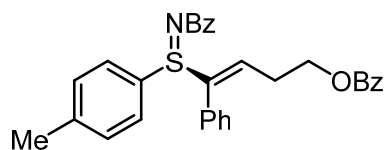
^1H NMR (400 MHz, CDCl_3) δ 8.22 (d, J = 6.8 Hz, 2H), 7.51 – 7.35 (m, 5H), 7.33 – 7.23 (m, 3H), 7.17 (d, J = 8.1 Hz, 2H), 6.99 (d, J = 6.7 Hz, 2H), 6.83 (t, J = 7.6 Hz, 1H), 4.13 (t, J = 6.5 Hz, 2H), 2.62 – 2.36 (m, 2H), 2.35 (s, 3H), 2.00 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 176.4, 170.9, 142.7, 139.3, 136.9, 134.2, 130.9, 130.2, 130.1, 129.9, 129.3, 129.0, 128.5, 128.4, 127.9, 62.6, 28.9, 21.6, 20.9.

HRMS (ESI) m/z calcd. for $\text{C}_{26}\text{H}_{26}\text{NO}_3\text{S}$ $[\text{M} + \text{H}]^+$ 432.1628, found 432.1627.

HPLC analysis: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 85/15, flow rate 0.5 mL/min, λ = 254 nm), t_R (major) = 24.55 min, t_R (minor) = 35.92 min.

(*E*)-4-((*R*)-*N*-Benzoyl-*S*-(*p*-tolyl)sulfinimidoyl)-4-phenylbut-3-en-1-yl benzoate (25**)**



According to **General procedure B** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A8** (113.5 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 1:1) to afford product **25** as a yellow oil (70.1 mg, 71% yield, 90% ee).

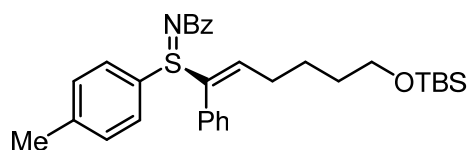
¹H NMR (400 MHz, CDCl₃) δ 8.22 (d, *J* = 8.1 Hz, 2H), 7.98 (d, *J* = 7.3 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.48 – 7.33 (m, 7H), 7.33 – 7.17 (m, 3H), 7.08 (d, *J* = 8.0 Hz, 2H), 6.98 (d, *J* = 7.6 Hz, 2H), 6.94 (d, *J* = 7.5 Hz, 1H), 4.48 – 4.30 (m, 2H), 2.69 – 2.49 (m, 2H), 2.31 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 176.3, 166.3, 142.6, 139.2, 136.8, 134.2, 133.1, 130.7(9), 130.7(7), 130.2, 130.0, 129.9, 129.7, 129.6, 129.2, 128.9, 128.4(3), 128.4(2), 128.3, 127.8, 63.1, 29.1, 21.5.

HRMS (ESI) *m/z* calcd. for C₃₁H₂₈NO₃S [M + H]⁺ 494.1784, found 494.1781.

HPLC analysis: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 85/15, flow rate 0.5 mL/min, λ = 254 nm), *t*_R (major) = 20.69 min, *t*_R (minor) = 24.12 min.

***N*-((*R*)-((*E*)-6-((*tert*-Butyldimethylsilyl)oxy)-1-phenylhex-1-en-1-yl)(*p*-tolyl)-λ⁴-sulfaneylidene)benzamide (**26**)**



According to **General procedure A** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A9** (124.9 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 4:1) to afford product **26** as a yellow oil (85.1 mg, 80% yield, 91% ee).

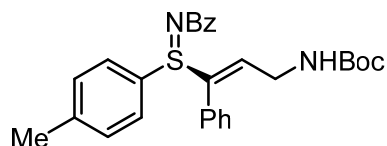
¹H NMR (400 MHz, CDCl₃) δ 8.24 (d, *J* = 5.2 Hz, 2H), 7.52 – 7.37 (m, 5H), 7.35 – 7.22 (m, 3H), 7.18 (d, *J* = 8.1 Hz, 2H), 6.98 (d, *J* = 6.7 Hz, 2H), 6.86 (t, *J* = 7.6 Hz, 1H), 3.55 (t, *J* = 5.8 Hz, 2H), 2.37 (s, 3H), 2.20 – 2.08 (m, 2H), 1.64 – 1.39 (m, 4H), 0.88 (s, 9H), 0.02 (s, 6H).

¹³C NMR (100 MHz, CDCl₃) δ 176.3, 142.4, 140.0, 137.1, 136.7, 131.3, 130.8, 130.4, 130.3, 130.0, 129.0, 128.9, 128.2(8), 128.2(6), 127.9, 62.7, 32.3, 29.2, 26.1, 25.3, 21.6, 18.4, –5.2.

HRMS (ESI) *m/z* calcd. for C₃₂H₄₂NSiO₂S [M + H]⁺ 532.2700, found 532.2701.

HPLC analysis: Chiralpak IC (*n*-hexane/*i*-PrOH = 70/30, flow rate 1.0 mL/min, λ = 254 nm), *t*_R (minor) = 10.30 min, *t*_R (major) = 11.57 min.

***tert*-Butyl((*E*)-3-((*R*)-*N*-benzoyl-*S*-(*p*-tolyl)sulfinimidoyl)-3-phenylallyl)carbamate (**27**)**



According to **General procedure A** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A10** (107.8 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 2:1) to afford product **27** as a yellow oil (66.5 mg, 70% yield, 90% ee).

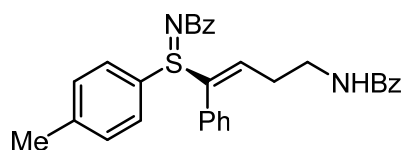
¹H NMR (400 MHz, CDCl₃) δ 8.22 (d, *J* = 8.0 Hz, 2H), 7.53 – 7.34 (m, 5H), 7.35 – 7.21 (m, 3H), 7.16 (d, *J* = 8.0 Hz, 2H), 7.03 (d, *J* = 6.9 Hz, 2H), 6.76 (t, *J* = 6.4 Hz, 1H), 4.92 (s, 1H), 4.01 – 3.66 (m, 2H), 2.34 (s, 3H), 1.40 (s, 9H).

¹³C NMR (100 MHz, CDCl₃) δ 176.5, 155.7, 142.8, 138.4, 136.7, 135.0, 130.9, 130.5, 130.2, 130.0, 129.4, 129.0, 128.6, 128.5, 127.9, 79.9, 39.4, 28.4, 21.6.

HRMS (ESI) *m/z* calcd. for C₂₈H₃₁N₂O₃S [M + H]⁺ 475.2050, found 475.2051.

HPLC analysis: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 85/15, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (major) = 14.15 min, *t_R* (minor) = 19.23 min.

***N*-((*R*)-((*E*)-4-Benzamido-1-phenylbut-1-en-1-yl)(*p*-tolyl)-λ⁴-sulfanylidene)benzamide (28)**



According to **General procedure A** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A11** (113.2 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 1:1) to afford product **28** as a yellow oil (66.1 mg, 67% yield, 94% ee).

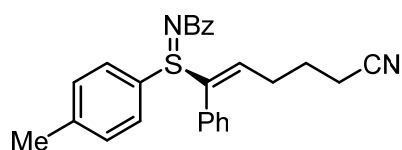
¹H NMR (400 MHz, CDCl₃) δ 8.20 (d, *J* = 6.9 Hz, 2H), 7.82 (d, *J* = 6.9 Hz, 2H), 7.53 – 7.36 (m, 6H), 7.29 (d, *J* = 7.6 Hz, 2H), 7.24 – 7.12 (m, 4H), 7.08 (t, *J* = 7.7 Hz, 2H), 6.83 (d, *J* = 6.7 Hz, 2H), 6.78 (t, *J* = 8.0 Hz, 1H), 3.57 (q, *J* = 6.1 Hz, 2H), 2.54 – 2.36 (m, 2H), 2.34 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 176.8, 167.5, 142.9, 138.5, 138.0, 137.0, 134.2, 131.4, 131.0, 130.4, 130.2, 129.2, 128.9, 128.5(3), 128.4(7), 128.4, 128.1, 127.3, 38.6, 29.9, 21.6.

HRMS (ESI) *m/z* calcd. for C₃₁H₂₉N₂O₂S [M + H]⁺ 493.1944, found 493.1944.

HPLC analysis: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 90/10, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (minor) = 37.62 min, *t_R* (major) = 43.38 min.

***N*-((*R*)-((*E*)-5-Cyano-1-phenylpent-1-en-1-yl)(*p*-tolyl)-λ⁴-sulfanylidene)benzamide (29)**



According to **General procedure A** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A12** (89.2 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 2:1) to afford product **29** as a colorless oil (from **A12**, 51.2 mg, 62% yield, 92% ee; from (*Z*)-**A12**, 47.8 mg, 58% yield, 96% ee; from (*E/Z*)-**A12** mixture, 49.5 mg, 60% yield, 95% ee).

¹H NMR (400 MHz, CDCl₃) δ 8.22 (d, *J* = 6.8 Hz, 2H), 7.47 – 7.35 (m, 5H), 7.35 – 7.24 (m, 3H), 7.17 (d, *J* = 8.0 Hz, 2H), 6.97 (d, *J* = 6.8 Hz, 2H), 6.70 (t, *J* = 7.5 Hz, 1H), 2.35 (s, 3H), 2.32 – 2.23 (m, 4H), 1.83 – 1.73 (m, 2H).

¹³C NMR (100 MHz, CDCl₃) δ 176.4, 142.8, 138.9, 136.8, 135.8, 131.0, 130.7, 130.2, 129.4, 128.9, 128.7, 128.6, 128.4, 128.0, 127.5, 119.1, 28.1, 24.7, 21.6, 16.6.

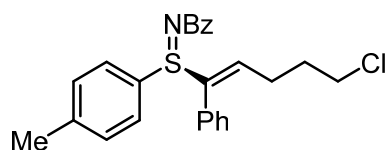
HRMS (ESI) *m/z* calcd. for C₂₆H₂₅N₂OS [M + H]⁺ 413.1682, found 413.1680.

HPLC analysis of **29** from **A12**: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 85/15, flow rate 0.5 mL/min, λ = 254 nm), *t*_R (major) = 23.39 min, *t*_R (minor) = 28.61 min.

HPLC analysis of **29** from (*Z*)-**A12**: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 85/15, flow rate 0.5 mL/min, λ = 254 nm), *t*_R (major) = 28.25 min, *t*_R (minor) = 34.80 min.

HPLC analysis of **29** from (*E/Z*)-**A12** mixture: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 85/15, flow rate 0.5 mL/min, λ = 254 nm), *t*_R (major) = 28.31 min, *t*_R (minor) = 34.54 min.

***N*-((*R*)-((*E*)-5-Chloro-1-phenylpent-1-en-1-yl)(*p*-tolyl)-λ⁴-sulfaneylidene)benzamide (**30**)**



According to **General procedure A** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A13** (92.0 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **30** as a colorless oil (51.5 mg, 61% yield, 92% ee).

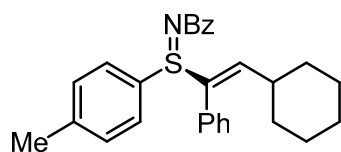
¹H NMR (400 MHz, CDCl₃) δ 8.24 (d, *J* = 6.8 Hz, 2H), 7.52 – 7.38 (m, 5H), 7.36 – 7.24 (m, 3H), 7.19 (d, *J* = 8.1 Hz, 2H), 7.00 (d, *J* = 6.6 Hz, 2H), 6.80 (t, *J* = 7.6 Hz, 1H), 3.49 (t, *J* = 6.4 Hz, 2H), 2.37 (s, 3H), 2.36 – 2.26 (m, 2H), 1.92 (p, *J* = 6.9 Hz, 2H).

¹³C NMR (100 MHz, CDCl₃) δ 176.4, 142.7, 137.9, 137.3, 136.7, 131.0, 130.9, 130.3, 130.1, 129.1, 128.9, 128.4(2), 128.3(6), 127.9, 44.1, 31.7, 26.8, 21.6.

HRMS (ESI) *m/z* calcd. for C₂₅H₂₅NOSCl [M + H]⁺ 422.1340, found 422.1338.

HPLC analysis: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 85/15, flow rate 0.5 mL/min, λ = 254 nm), *t*_R (major) = 16.54 min, *t*_R (minor) = 19.46 min.

***N*-((*R*)-((*E*)-2-Cyclohexyl-1-phenylvinyl)(*p*-tolyl)-λ⁴-sulfaneylidene)benzamide (**31**)**



According to **General procedure A** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A14** (93.7 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **31** as a yellow oil (72.7 mg, 85% yield, 92% ee).

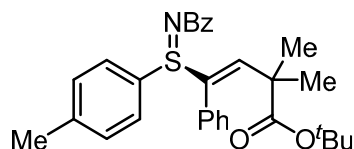
¹H NMR (400 MHz, CDCl₃) δ 8.24 (d, *J* = 6.7 Hz, 2H), 7.50 – 7.36 (m, 5H), 7.35 – 7.22 (m, 3H), 7.17 (d, *J* = 8.0 Hz, 2H), 6.98 (d, *J* = 6.8 Hz, 2H), 6.70 (d, *J* = 10.2 Hz, 1H), 2.35 (s, 3H), 2.23 – 2.04 (m, 1H), 1.80 – 1.54 (m, 4H), 1.52 – 0.91 (m, 6H).

¹³C NMR (100 MHz, CDCl₃) δ 176.2, 145.1, 142.3, 137.2, 135.0, 131.3, 130.7, 130.3(7), 130.3(5), 130.0, 128.9, 128.8, 128.2, 128.1, 127.8, 38.5, 32.4, 32.3, 25.6, 25.2(2), 25.1(9), 21.5.

HRMS (ESI) *m/z* calcd. for C₂₈H₃₀NOS [M + H]⁺ 428.2043, found 428.2041.

HPLC analysis: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 98/2, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (major) = 47.98 min, *t_R* (minor) = 54.14 min.

***tert*-Butyl (E)-4-((R)-N-benzoyl-S-(*p*-tolyl)sulfinimidoyl)-2,2-dimethyl-4-phenylbut-3-enoate (**32**)**



According to **General procedure A** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A15-I** (111.7 mg, 0.30 mmol, 1.5 equiv) or alkenyl bromide **A15-Br** (97.6 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **32** as a yellow oil (from **A15-I**: 68.3 mg, 70% yield, 96% ee; from **A15-Br**: 59.5 mg, 61% yield, 92% ee).

¹H NMR (400 MHz, CDCl₃) δ 8.20 (d, *J* = 6.8 Hz, 2H), 7.48 – 7.41 (m, 3H), 7.38 (dd, *J* = 8.1, 6.4 Hz, 2H), 7.32 – 7.26 (m, 1H), 7.19 (dd, *J* = 7.8, 5.4 Hz, 4H), 6.95 (s, 1H), 6.87 (d, *J* = 6.8 Hz, 2H), 2.37 (s, 3H), 1.38 (s, 9H), 1.15 (s, 3H), 1.10 (s, 3H).

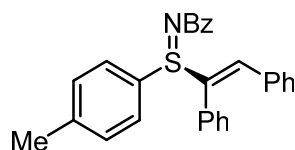
¹³C NMR (100 MHz, CDCl₃) δ 176.3, 174.5, 142.6, 142.0, 137.5, 137.1, 130.9(3), 130.9(0), 130.8, 130.0(3), 130.0(1), 129.2, 129.0, 128.5, 127.9, 127.8, 81.3, 45.8, 29.8, 28.0, 26.4, 26.3, 21.6.

HRMS (ESI) *m/z* calcd. for C₃₀H₃₄NO₃S [M + H]⁺ 488.2254, found 488.2252.

HPLC analysis of **32** from **A5-I**: Chiralpak IH (*n*-hexane/*i*-PrOH = 80/20, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (minor) = 7.98 min, *t_R* (major) = 13.44 min.

HPLC analysis of **32** from **A5-Br**: Chiralpak IH (*n*-hexane/*i*-PrOH = 80/20, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (minor) = 7.99 min, *t_R* (major) = 13.43 min.

***N*-((R)-((E)-1,2-Diphenylvinyl)(*p*-tolyl)-λ⁴-sulfaneylidene)benzamide (**33**)**



According to **General procedure A** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0

equiv), alkenyl iodide **A16** (91.9 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **33** as a yellow oil (60.7 mg, 72% yield, 88% ee).

According to **General procedure B**, the product **33** was afforded in 80% yield and 95% ee.

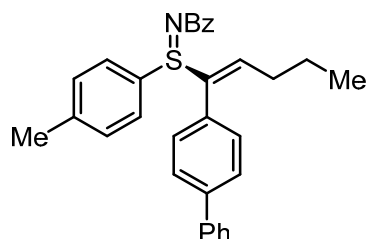
¹H NMR (400 MHz, CDCl₃) δ 8.25 (d, *J* = 6.7 Hz, 2H), 7.62 (s, 1H), 7.52 (d, *J* = 8.3 Hz, 2H), 7.49 – 7.37 (m, 3H), 7.37 – 7.30 (m, 1H), 7.30 – 7.23 (m, 2H), 7.22 – 7.11 (m, 5H), 7.10 – 7.05 (m, 2H), 7.00 (d, *J* = 6.8 Hz, 2H), 2.35 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 176.4, 142.7, 137.0, 136.7, 135.7, 133.6, 131.8, 130.9, 130.4, 130.1, 129.8, 129.4, 129.3, 129.0, 128.9, 128.5(4), 128.4(5), 127.9, 21.6.

HRMS (ESI) *m/z* calcd. for C₂₈H₂₄NOS [M + H]⁺ 422.1573, found 422.1569.

HPLC analysis: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 90/10, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (major) = 28.78 min, *t_R* (minor) = 32.53 min.

***N*-((*R*)-((*E*)-1-([1,1'-Biphenyl]-4-yl)pent-1-en-1-yl)(*p*-tolyl)-λ⁴-sulfaneylidene)benzamide (**34**)**



According to **General procedure A** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A17** (104.5 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **34** as a colorless oil (57.5 mg, 62% yield, 90% ee).

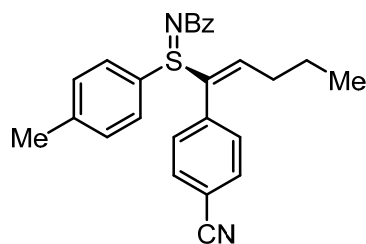
¹H NMR (400 MHz, CDCl₃) δ 8.25 (d, *J* = 6.6 Hz, 2H), 7.58 (d, *J* = 7.4 Hz, 2H), 7.55 – 7.30 (m, 10H), 7.18 (d, *J* = 8.0 Hz, 2H), 7.05 (d, *J* = 8.2 Hz, 2H), 6.87 (t, *J* = 7.6 Hz, 1H), 2.35 (s, 3H), 2.20 – 2.08 (m, 2H), 1.50 (h, *J* = 7.3 Hz, 2H), 0.90 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 176.3, 142.5, 141.5, 140.3, 140.2, 137.1, 136.5, 130.9, 130.8, 130.3, 130.2, 130.1, 128.9(6), 128.9(4), 128.3, 127.9, 127.8, 127.1, 126.8, 31.5, 22.3, 21.6, 13.9.

HRMS (ESI) *m/z* calcd. for C₃₁H₃₀NOS [M + H]⁺ 464.2043, found 464.2040.

HPLC analysis: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (major) = 24.83 min, *t_R* (minor) = 27.58 min.

***N*-((*R*)-((*E*)-1-(4-Cyanophenyl)pent-1-en-1-yl)(*p*-tolyl)-λ⁴-sulfaneylidene)benzamide (**35**)**



According to **General procedure A** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A18** (89.2 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **35** as a colorless oil (39.6 mg, 48% yield, 92% ee).

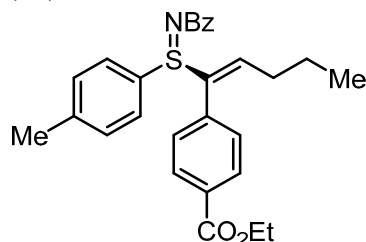
¹H NMR (400 MHz, CDCl₃) δ 8.18 (d, *J* = 7.2 Hz, 2H), 7.53 (d, *J* = 8.2 Hz, 2H), 7.45 (d, *J* = 8.3 Hz, 3H), 7.40 (t, *J* = 7.3 Hz, 2H), 7.21 (d, *J* = 8.1 Hz, 2H), 7.04 (d, *J* = 8.2 Hz, 2H), 6.92 (t, *J* = 7.7 Hz, 1H), 2.38 (s, 3H), 2.12 – 1.96 (m, 2H), 1.48 (h, *J* = 7.3 Hz, 2H), 0.87 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 176.5, 143.0, 142.9, 136.7, 136.1, 136.0, 131.9, 131.2, 131.1, 130.3, 129.3, 128.9, 127.9(8), 127.9(5), 118.4, 112.9, 31.7, 22.1, 21.6, 13.8.

HRMS (ESI) *m/z* calcd. for C₂₆H₂₅N₂OS [M + H]⁺ 413.1682, found 413.1678.

HPLC analysis: Chiralpak IH (*n*-hexane/*i*-PrOH = 70/30, flow rate 1.0 mL/min, λ = 254 nm), *t_R* (minor) = 6.63 min, *t_R* (major) = 24.37 min.

Ethyl 4-((*E*)-1-((*R*)-*N*-benzoyl-*S*-(*p*-tolyl)sulfinimidoyl)pent-1-en-1-yl)benzoate (36**)**



According to **General procedure A** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A19** (103.3 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 2:1) to afford product **36** as a colorless oil (53.3 mg, 58% yield, 92% ee).

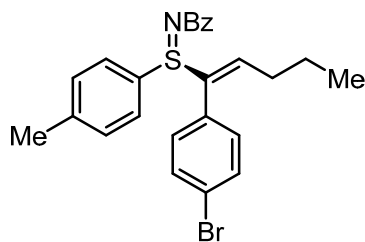
¹H NMR (400 MHz, CDCl₃) δ 8.20 (d, *J* = 6.6 Hz, 2H), 7.91 (d, *J* = 8.2 Hz, 2H), 7.48 – 7.33 (m, 5H), 7.17 (d, *J* = 8.0 Hz, 2H), 7.02 (d, *J* = 8.2 Hz, 2H), 6.87 (t, *J* = 7.7 Hz, 1H), 4.35 (q, *J* = 7.1 Hz, 2H), 2.34 (s, 3H), 2.12 – 1.99 (m, 2H), 1.46 (h, *J* = 7.3 Hz, 2H), 1.37 (t, *J* = 7.1 Hz, 3H), 0.85 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 176.4, 166.2, 142.7, 141.4, 136.9, 136.3, 135.8, 130.9, 130.8, 130.5, 130.2, 129.7, 129.3, 128.9, 128.1, 127.9, 61.29, 31.5, 22.1, 21.2, 14.4, 13.8.

HRMS (ESI) *m/z* calcd. for C₂₈H₃₀NO₃S [M + H]⁺ 460.1941, found 460.1939.

HPLC analysis: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 85/15, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (major) = 11.15 min, *t_R* (minor) = 12.16 min.

***N*-((*R*)-((*E*)-1-(4-Bromophenyl)pent-1-en-1-yl)(*p*-tolyl)-λ⁴-sulfaneylidene)benzamide (**37**)**



According to **General procedure A** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A20** (105.3 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **37** as a colorless oil (80.3 mg, 86% yield, 90% ee).

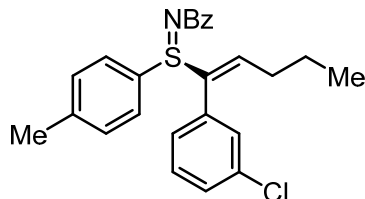
¹H NMR (400 MHz, CDCl₃) δ 8.20 (d, *J* = 6.7 Hz, 2H), 7.49 – 7.31 (m, 7H), 7.18 (d, *J* = 8.1 Hz, 2H), 6.85 (t, *J* = 7.7 Hz, 1H), 6.79 (d, *J* = 8.4 Hz, 2H), 2.34 (s, 3H), 2.10 – 1.98 (m, 2H), 1.45 (h, *J* = 7.3 Hz, 2H), 0.85 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 176.3, 142.6, 141.5, 136.9, 136.1, 132.0, 131.4, 130.8, 130.1, 129.9, 129.7, 128.9, 128.0, 127.8, 123.4, 31.4, 22.1, 21.5, 13.8.

HRMS (ESI) *m/z* calcd. for C₂₅H₂₅NOSBr [M + H]⁺ 466.0835, found 466.0834.

HPLC analysis: Chiralpak IH (*n*-hexane/*i*-PrOH = 70/30, flow rate 1.0 mL/min, λ = 254 nm), *t_R* (minor) = 4.20 min, *t_R* (major) = 14.79 min.

***N*-((*R*)-((*E*)-1-(3-Chlorophenyl)pent-1-en-1-yl)(*p*-tolyl)-λ⁴-sulfaneylidene)benzamide (**38**)**



According to **General procedure A** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A21** (92.0 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **38** as a colorless oil (55.8 mg, 66% yield, 92% ee).

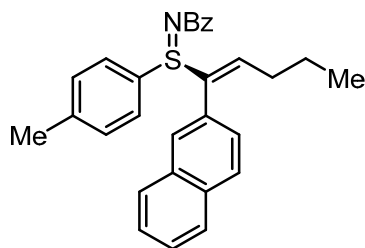
¹H NMR (400 MHz, CDCl₃) δ 8.21 (d, *J* = 6.7 Hz, 2H), 7.52 – 7.33 (m, 5H), 7.33 – 7.24 (m, 1H), 7.23 – 7.11 (m, 3H), 6.92 (t, *J* = 1.9 Hz, 1H), 6.88 – 6.77 (m, 2H), 2.35 (s, 3H), 2.12 – 2.01 (m, 2H), 1.46 (h, *J* = 7.4 Hz, 2H), 0.87 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 176.3, 142.7, 141.4, 136.9, 135.9, 134.0, 132.8, 130.8, 130.4, 130.1, 129.7, 129.4, 129.1, 128.9, 128.7, 128.1, 127.9, 31.5, 22.1, 21.5, 13.8.

HRMS (ESI) *m/z* calcd. for C₂₅H₂₅NOSCl [M + H]⁺ 422.1340, found 422.1338.

HPLC analysis: Chiralpak IH (*n*-hexane/*i*-PrOH = 70/30, flow rate 1.0 mL/min, λ = 254 nm), *t_R* (minor) = 4.25 min, *t_R* (major) = 16.75 min.

***N*-((*R*)-((*E*)-1-(Naphthalen-2-yl)pent-1-en-1-yl)(*p*-tolyl)-λ⁴-sulfaneylidene)benzamide (**39**)**



According to **General procedure B** with sulfenamide **S1** (48.6 mg, 0.20 mmol), alkenyl iodide **A22** (96.7 mg, 0.3 mmol, 1.5 equiv) and **L5** as the ligand, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **39** as a colorless oil (52.5 mg, 60% yield, 92% ee).

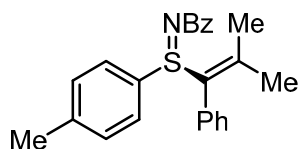
¹H NMR (400 MHz, CDCl₃) δ 8.25 (d, *J* = 6.6 Hz, 2H), 7.80 (d, *J* = 7.0 Hz, 1H), 7.75 – 7.66 (m, 2H), 7.57 – 7.33 (m, 8H), 7.14 (d, *J* = 8.1 Hz, 2H), 7.05 (dd, *J* = 8.4, 1.7 Hz, 1H), 6.91 (t, *J* = 7.7 Hz, 1H), 2.33 (s, 3H), 2.21 – 2.02 (m, 2H), 1.49 (h, *J* = 7.4 Hz, 2H), 0.87 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 176.4, 142.5, 140.2, 137.1, 136.7, 133.2, 132.8, 130.8, 130.2(2), 130.1(8), 130.1, 129.0, 128.8, 128.3(4), 128.3(2), 127.9(2), 127.8(8), 127.8, 127.7, 126.9, 126.5, 31.5, 22.3, 21.6, 13.9.

HRMS (ESI) *m/z* calcd. for C₂₉H₂₈NOS [M + H]⁺ 438.1886, found 438.1883.

HPLC analysis: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 95/5, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (major) = 22.00 min, *t_R* (minor) = 25.24 min.

(*R*)-*N*-((2-Methyl-1-phenylprop-1-en-1-yl)(*p*-tolyl)-λ⁴-sulfaneylidene)benzamide (40)



According to **General procedure A** with sulfenamide **S1** (48.6 mg, 0.20 mmol), alkenyl iodide **A23** (77.5 mg, 0.3 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **40** as a yellow oil (52.3 mg, 70% yield, 90% ee).

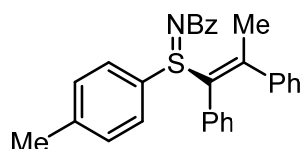
¹H NMR (400 MHz, CDCl₃) δ 8.18 (d, *J* = 6.8 Hz, 2H), 7.50 – 7.32 (m, 5H), 7.23 (t, *J* = 7.4 Hz, 1H), 7.19 – 7.05 (m, 4H), 6.65 (d, *J* = 7.6 Hz, 2H), 2.62 (s, 3H), 2.35 (s, 3H), 1.70 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 176.2, 149.0, 141.9, 137.2, 132.2, 131.6, 131.3, 130.6, 129.8, 129.3, 128.8, 128.2, 127.9, 127.8, 127.5, 23.8, 21.9, 21.5.

HRMS (ESI) *m/z* calcd. for C₂₄H₂₄NOS [M + H]⁺ 374.1573, found 374.1571.

HPLC analysis: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 85/15, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (minor) = 10.58 min, *t_R* (major) = 13.04 min.

***N*-((*R*)-((*E*)-1,2-Diphenylprop-1-en-1-yl)(*p*-tolyl)-λ⁴-sulfaneylidene)benzamide (41)**



According to **General procedure B** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A24** (96.1 mg, 0.30 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **41** as a colorless oil (58.3 mg, 66% yield, 86% ee). The *E* configuration of **41** was confirmed according to the two-dimensional NMR spectroscopy (2D NOESY, HSQC, and HMBC) analysis.

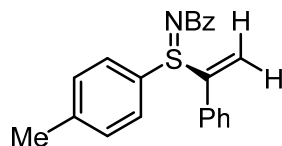
¹H NMR (400 MHz, CDCl₃) δ 8.20 (d, *J* = 6.8 Hz, 2H), 7.55 – 7.32 (m, 5H), 7.17 (d, *J* = 8.0 Hz, 2H), 7.14 – 6.99 (m, 6H), 6.94 (t, *J* = 7.6 Hz, 2H), 6.62 (d, *J* = 7.5 Hz, 2H), 2.95 (s, 3H), 2.36 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 176.5, 150.6, 142.1, 140.9, 137.2, 135.1, 132.0, 131.6, 130.7, 129.9, 129.1, 128.9, 128.1, 127.9(3), 127.9(1), 127.9, 127.7, 127.1, 22.7, 21.6.

HRMS (ESI) *m/z* calcd. for C₂₉H₂₆NOS [M + H]⁺ 436.1730, found 436.1730.

HPLC analysis: Chiralpak OD-H (*n*-hexane/*i*-PrOH = 80/20, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (minor) = 17.27 min, *t_R* (major) = 20.05 min.

***N*-((*R*)-((*E*)-1-(3-Chlorophenyl)pent-1-en-1-yl)(*p*-tolyl)-λ⁴-sulfaneylidene)benzamide (**42**)**



According to **General procedure A** with sulfenamide **S1** (48.6 mg, 0.20 mmol), alkenyl iodide **A25** (69.1 mg, 0.3 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **42** as a colorless oil (35.3 mg, 51% yield, 92% ee).

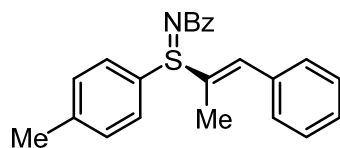
¹H NMR (400 MHz, CDCl₃) δ 8.25 (d, *J* = 6.7 Hz, 2H), 7.57 (d, *J* = 8.3 Hz, 2H), 7.49 – 7.36 (m, 5H), 7.34 – 7.25 (m, 3H), 7.17 (d, *J* = 7.9 Hz, 2H), 6.43 (s, 1H), 6.05 (s, 1H), 2.32 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 176.4, 144.8, 142.9, 136.7, 133.8, 131.0, 130.3, 129.6, 129.0, 128.8, 128.7, 128.1, 127.9, 120.8, 21.6.

HRMS (ESI) *m/z* calcd. for C₂₂H₂₀NOS [M + H]⁺ 346.1260, found 346.1254.

HPLC analysis: Chiralpak IH (*n*-hexane/*i*-PrOH = 70/30, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (minor) = 11.75 min, *t_R* (major) = 16.29 min.

***N*-((*S*)-((*E*)-1-Phenylprop-1-en-2-yl)(*p*-tolyl)-λ⁴-sulfaneylidene)benzamide (**43**)**



According to **General procedure A** with sulfenamide **S1** (48.6 mg, 0.20 mmol),

alkenyl iodide **A26** (73.2 mg, 0.3 mmol, 1.5 equiv) and the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **43** as a colorless oil (34.5 mg, 48% yield, 83% ee).

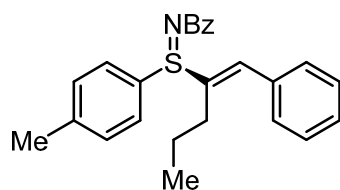
¹H NMR (400 MHz, CDCl₃) δ 8.26 (d, *J* = 5.1 Hz, 2H), 7.74 (d, *J* = 8.1 Hz, 2H), 7.60 (s, 1H), 7.50 – 7.30 (m, 10H), 2.42 (s, 3H), 2.08 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 176.6, 142.5, 137.8, 136.9, 134.4(1), 134.3(9), 130.9, 130.5, 130.2, 129.7, 129.1, 129.0, 128.8, 127.9, 127.7, 21.6, 12.2.

HRMS (ESI) *m/z* calcd. for C₂₃H₂₂NOS [M + H]⁺ 360.1417, found 360.1415.

HPLC analysis: Chiralpak IC (*n*-hexane/*i*-PrOH = 50/50, flow rate 0.6 mL/min, λ = 254 nm), *t_R* (major) = 28.98 min, *t_R* (minor) = 48.33 min.

***N*-((*S*)-((*E*)-1-Phenylpent-1-en-2-yl)(*p*-tolyl)-λ⁴-sulfaneylidene)benzamide (**44**)**



According to **General procedure B** with sulfenamide **S1** (48.6 mg, 0.20 mmol), alkenyl iodide **A27** (81.6 mg, 0.3 mmol, 1.5 equiv) and the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **44** as a colorless oil (34.1 mg, 44% yield, 94% ee).

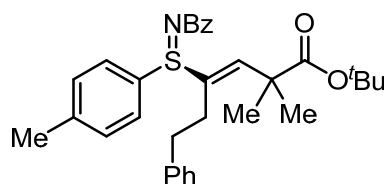
¹H NMR (400 MHz, CDCl₃) δ 8.28 (d, *J* = 6.6 Hz, 2H), 7.78 (d, *J* = 7.8 Hz, 2H), 7.54 (d, *J* = 8.1 Hz, 2H), 7.50 – 7.32 (m, 6H), 7.25 (d, *J* = 4.2 Hz, 2H), 7.14 (s, 1H), 2.58 – 2.43 (m, 1H), 2.39 (s, 3H), 2.27 – 2.02 (m, 1H), 1.64 – 1.35 (m, 2H), 0.85 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 176.6, 141.9, 139.1, 138.2, 136.9, 134.4, 130.8, 130.3, 130.1, 129.9, 129.0, 128.9, 128.8, 127.9, 127.1, 29.8, 21.8, 21.5, 13.7.

HRMS (ESI) *m/z* calcd. for C₂₅H₂₆NOS [M + H]⁺ 388.1730, found 388.1730.

HPLC analysis: Chiralpak IH (*n*-hexane/*i*-PrOH = 60/40, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (minor) = 7.74 min, *t_R* (major) = 9.83 min.

***tert*-Butyl (E)-4-((*S*)-*N*-benzoyl-*S*-(*p*-tolyl)sulfinimidoyl)-2,2-dimethyl-6-phenylhex-3-enoate (**45**)**



According to **General procedure B** with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A28** (120.0 mg, 0.30 mmol, 1.5 equiv) and **L5** as the ligand, the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 2:1) to afford product **45** as a colorless oil (41.2 mg, 40% yield, 93% ee).

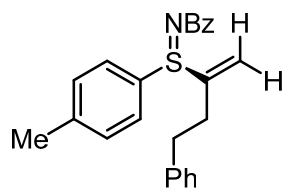
¹H NMR (400 MHz, CDCl₃) δ 8.24 (d, *J* = 6.8 Hz, 2H), 7.72 (d, *J* = 8.3 Hz, 2H), 7.50

– 7.35 (m, 3H), 7.31 (d, $J = 8.1$ Hz, 2H), 7.28 – 7.21 (m, 2H), 7.17 (t, $J = 7.3$ Hz, 1H), 7.10 (d, $J = 7.0$ Hz, 2H), 6.79 (s, 1H), 2.96 – 2.81 (m, 1H), 2.79 – 2.68 (m, 1H), 2.67 – 2.54 (m, 1H), 2.40 (s, 3H), 2.40 – 2.26 (m, 1H), 1.46 (s, 3H), 1.42 (s, 3H), 1.38 (s, 9H). ^{13}C NMR (100 MHz, CDCl_3) δ 176.4, 174.6, 143.5, 142.7, 140.8, 137.0, 136.9, 130.9(2), 130.8(8), 130.5, 129.0, 128.7, 128.3, 128.2, 127.9, 126.5, 81.5, 45.0, 35.2, 29.9, 28.0, 26.9, 26.8, 21.6.

HRMS (ESI) m/z calcd. for $\text{C}_{32}\text{H}_{38}\text{NO}_3\text{S}$ $[\text{M} + \text{H}]^+$ 516.2567, found 516.2565.

HPLC analysis: Chiralpak IH (n -hexane/ i -PrOH = 80/20, flow rate 0.5 mL/min, $\lambda = 254$ nm), t_{R} (minor) = 8.02 min, t_{R} (major) = 11.33 min.

(*S*)-*N*-((4-Phenylbut-1-en-2-yl)(*p*-tolyl)- λ^4 -sulfaneylidene)benzamide (46**)**



According to **General procedure A** with sulfenamide **S1** (48.6 mg, 0.20 mmol), alkenyl iodide **A29** (77.5 mg, 0.3 mmol, 1.5 equiv), the reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **46** as a colorless oil (26.9 mg, 36% yield, 88% ee).

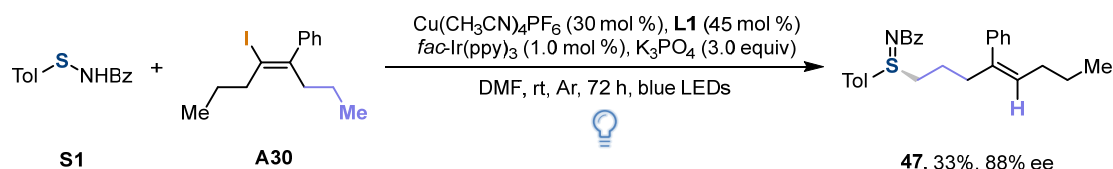
^1H NMR (400 MHz, CDCl_3) δ 8.22 (d, $J = 6.8$ Hz, 2H), 7.69 (d, $J = 8.3$ Hz, 2H), 7.49 – 7.35 (m, 3H), 7.32 (d, $J = 8.1$ Hz, 2H), 7.27 – 7.21 (m, 2H), 7.20 – 7.15 (m, 1H), 7.05 (d, $J = 6.8$ Hz, 2H), 6.24 (d, $J = 1.2$ Hz, 1H), 5.74 (d, $J = 1.4$ Hz, 1H), 2.83 – 2.74 (m, 2H), 2.67 – 2.56 (m, 1H), 2.45 – 2.34 (m, 4H).

^{13}C NMR (100 MHz, CDCl_3) δ 176.5, 144.9, 143.0, 140.2, 136.8, 130.9, 130.6, 129.8, 129.0, 128.6, 128.4, 128.2, 127.9, 126.5, 122.1, 34.0, 31.0, 21.6.

HRMS (ESI) m/z calcd. for $\text{C}_{24}\text{H}_{24}\text{NOS}$ $[\text{M} + \text{H}]^+$ 374.1573, found 374.1570.

HPLC analysis: Chiralpak IH (n -hexane/ i -PrOH = 70/30, flow rate 0.5 mL/min, $\lambda = 254$ nm), t_{R} (minor) = 13.50 min, t_{R} (major) = 26.23 min.

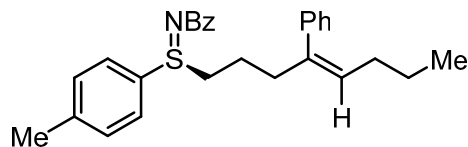
The 1,5-HAT of alkenyl iodide A30 radical experiments under standard conditions with photoinduced



According to **General procedure A**, under an argon atmosphere, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A30** (94.3 mg, 0.3 mmol, 1.5 equiv), $\text{Cu}(\text{CH}_3\text{CN})_4\text{PF}_6$ (22.4 mg, 0.06 mmol, 30 mol %), **L1** (31.5 mg, 0.09 mmol, 45 mol %), $\text{fac-Ir}(\text{ppy})_3$ (1.30 mg, 1.0 mol %), and K_3PO_4 (127.4 mg, 0.6 mol, 3.0 equiv) in DMF (2.0 mL) stirred at rt and blue LEDs irradiation for 72 h under argon.

Upon completion, the reaction mixture was extracted three times with Et₂O and organic phase was evaporated. The residue was purified by column chromatography on silica gel to afford the desired product.

***N*-((*S*)-((*Z*)-4-Phenyloct-4-en-1-yl)(*p*-tolyl)- λ^4 -sulfaneylidene)benzamide (**47**)**



The reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **47** as a yellow oil (28.4 mg, 33% yield, 88% ee).

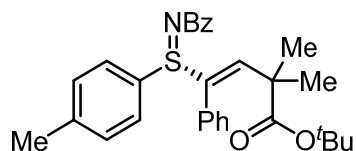
¹H NMR (400 MHz, CDCl₃) δ 8.14 (d, *J* = 6.8 Hz, 2H), 7.59 (d, *J* = 8.3 Hz, 2H), 7.48 – 7.34 (m, 3H), 7.32 – 7.17 (m, 6H), 7.06 – 6.97 (m, 2H), 5.46 (t, *J* = 7.3 Hz, 1H), 3.28 – 2.93 (m, 2H), 2.48 (t, *J* = 7.2 Hz, 2H), 2.40 (s, 3H), 1.89 (q, *J* = 7.4 Hz, 2H), 1.68 – 1.57 (m, 2H), 1.44 – 1.26 (m, 2H), 0.82 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 176.8, 142.9, 140.2, 138.6, 136.8, 131.1, 130.7, 130.6, 129.6, 128.9, 128.5, 128.3, 127.9, 127.3, 126.8, 49.1, 37.6, 31.0, 23.2, 21.6, 21.5, 13.9.

HRMS (ESI) *m/z* calcd. for C₂₈H₃₂NOS [M + H]⁺ 430.2199, found 430.2197.

HPLC analysis: Chiralpak IH (*n*-hexane/*i*-PrOH = 80/20, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (minor) = 10.98 min, *t_R* (major) = 14.89 min.

***tert*-Butyl (E)-4-((*S*)-*N*-benzoyl-*S*-(*p*-tolyl)sulfinimidoyl)-2,2-dimethyl-4-phenylbut-3-enoate (**48**)**

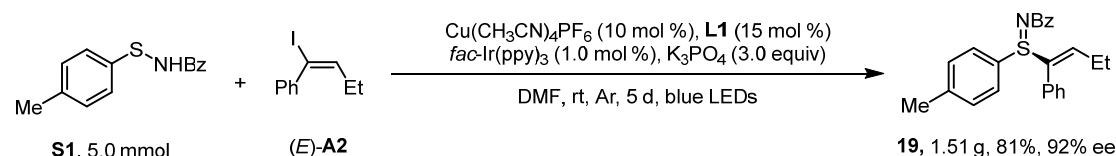


Under an argon atmosphere, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with sulfenamide **S1** (24.3 mg, 0.10 mmol, 1.0 equiv), alkyne **B1** (20.4 mg, 0.20 mmol, 2.0 equiv), *tert*-butyl 2-bromo-2-methylpropanoate **C1** (66.9 mg, 0.30 mmol, 3.0 equiv), CuI (1.90 mg, 0.01 mmol, 10 mol %), **L7** (9.96 mg, 0.015 mmol, 15 mol %) and Cs₂CO₃ (97.6 mg, 0.40 mol, 2.0 equiv) in 1,4-dioxane (1.0 mL) stirred at rt for 5 days under argon. Upon completion, the precipitate was filtered off and washed by DCM. The filtrate was evaporated and the residue was purified by column chromatography on silica gel to afford the desired product **48** as a yellow oil (25.9 mg, 53% yield, 90% ee).

HPLC analysis of **48**: Chiralpak IH (*n*-hexane/*i*-PrOH = 80/20, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (major) = 7.74 min, *t_R* (minor) = 13.39 min.

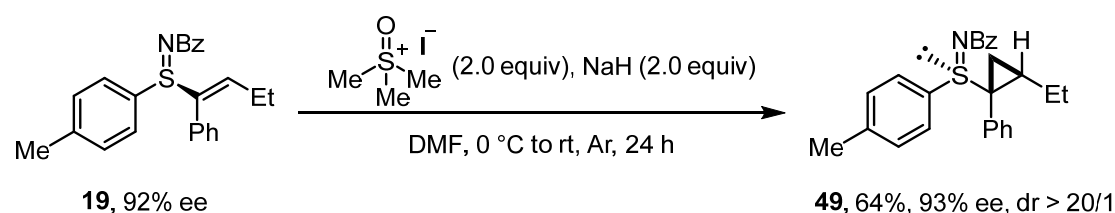
6. Transformation

Gram-scale reaction



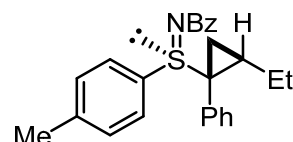
Under an argon atmosphere, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with sulfenamide **S1** (1.22 g, 5.0 mmol, 1.0 equiv), alkenyl iodide $(E)\text{-A2}$ (1.94 g, 7.5 mmol, 1.5 equiv), $\text{Cu}(\text{CH}_3\text{CN})_4\text{PF}_6$ (186.3 mg, 0.50 mmol, 10 mol %), **L1** (261.0 mg, 0.75 mmol, 15 mol %), $\text{fac-Ir}(\text{ppy})_3$ (32.8 mg, 0.05 mmol, 1.0 mol %), and K_3PO_4 (3.18 g, 15 mol, 3.0 equiv) in DMF (50.0 mL) stirred at rt and blue LEDs irradiation for 5 days under argon. Upon completion, the reaction mixture was extracted three times with Et_2O and organic phase was evaporated. The residue was purified by column chromatography on silica gel to afford the desired product **19** as a pale yellow solid (1.51 g, 81% yield, 92% ee).

Cyclopropanation of sulfimide with dimethyl sulfoxonium methylide:



According to the literature reported procedure.¹⁶ NaH (60% in oil, 16.0 mg, 0.40 mmol, 2.0 equiv) was added to a dry DMF (2.0 mL) solution of trimethylsulfoxonium iodide (44.0 mg, 0.40 mmol, 2.0 equiv) and the reaction mixture was stirred at 0 °C for 1.0 h. A dry DMF (0.50 mL) solution of **19** (74.7 mg, 0.20 mmol, 1.0 equiv) was added dropwise to the mixture at 0 °C after which the whole was stirred for 24 h at rt. After cooling to rt, the reaction mixture was extracted with DCM. The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4 , filtered, and concentrated to afford the crude product, which was purified by silica gel chromatography to afford the product **49**.

N-2-Ethyl-1-phenylcyclopropyl(*p*-tolyl)- λ^4 -sulfaneylidenebenzamide (**49**)



The product mixture was purified by silica gel column chromatography (petroleum ether/ethyl acetate = 3/1) to afford **49** as a colorless oil (49.6 mg, 64% yield, 93% ee, dr > 20/1).

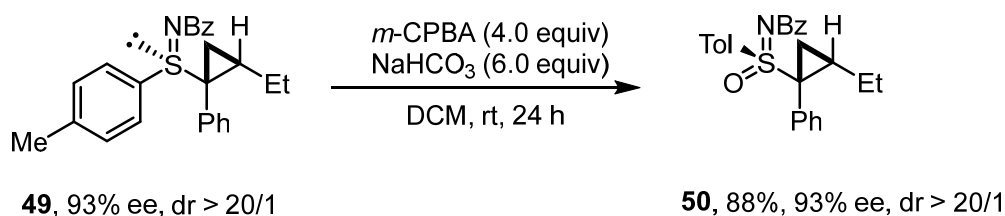
¹H NMR (400 MHz, CDCl_3) δ 8.20 (d, J = 6.7 Hz, 2H), 7.48 – 7.34 (m, 3H), 7.31 – 7.22 (m, 1H), 7.22 – 7.08 (m, 6H), 7.05 (d, J = 7.1 Hz, 2H), 2.34 (s, 3H), 2.16 – 2.04

(m, 1H), 1.96 – 1.83 (m, 1H), 1.67 – 1.56 (m, 1H), 1.47 (dd, $J = 8.4, 5.9$ Hz, 1H), 1.39 (t, $J = 6.3$ Hz, 1H), 1.21 (t, $J = 7.3$ Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 175.5, 142.4, 137.2, 134.9, 132.8, 130.6, 129.8(4), 129.7(5), 129.0, 128.6, 127.8, 127.5, 127.3, 47.1, 30.6, 23.1, 21.6, 18.2, 14.1.

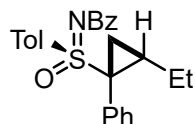
HRMS (ESI) m/z calcd. for $\text{C}_{25}\text{H}_{26}\text{NOS}$ $[\text{M} + \text{H}]^+$ 388.1730, found 388.1727.

HPLC analysis: Chiralcel IH (n -hexane/ i -PrOH = 80/20, flow rate 0.5 mL/min, $\lambda = 254$ nm), t_R (minor) = 9.24 min, t_R (major) = 11.19 min.



According to the literature reported procedure.¹⁷ To a solution of **49** (38.9 mg, 0.10 mmol, 1.0 equiv) in DCM (2.5 mL), was added 3-Chloroperoxybenzoic Acid (m -CPBA, 69.0 g, 0.40 mmol, 4.0 equiv) and NaHCO_3 (50.5 mg, 0.60 mmol, 6.0 equiv) in a 10.0 mL Schlenk tube with magnetic stir bar. Then the mixture was stirred at rt for 24 h. After the reaction finished that monitored by TLC, the reaction mixture was extracted with DCM (3×5 mL) and 2.0 M aqueous NaOH solution three times (3×10 mL). The combined organic layers were dried over anhydrous Na_2SO_4 , filtered, and concentrated to afford the crude product, which was purified by silica gel chromatography to afford the product **50** as a colorless oil.

***N*-2-Ethyl-1-phenylcyclopropyl(oxo)(*p*-tolyl)- λ^6 -sulfaneylidene)benzamide (**50**)**



The product mixture was purified by silica gel column chromatography (petroleum ether/ethyl acetate = 5/1) to afford **50** as a colorless oil (35.5 mg, 88% yield, 93% ee, dr > 20/1).

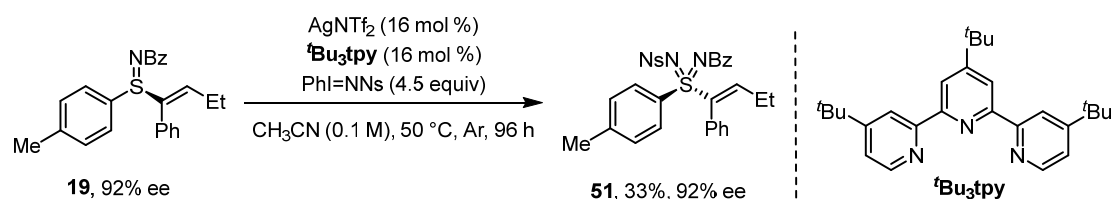
^1H NMR (400 MHz, CDCl_3) δ 8.20 (d, $J = 7.0$ Hz, 2H), 7.56 – 7.47 (m, 1H), 7.43 (t, $J = 7.4$ Hz, 2H), 7.25 – 7.10 (m, 7H), 7.08 (d, $J = 8.1$ Hz, 2H), 2.47 – 2.36 (m, 1H), 2.35 (s, 3H), 2.20 – 2.06 (m, 1H), 1.97 (dd, $J = 7.9, 5.3$ Hz, 1H), 1.83 – 1.68 (m, 1H), 1.44 (dd, $J = 9.1, 5.3$ Hz, 1H), 1.28 (t, $J = 7.4$ Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 174.1, 144.1, 136.4, 136.2, 134.3, 132.9, 132.0, 129.6, 129.5, 128.8, 128.1, 127.9, 52.7, 33.3, 21.7, 20.8, 17.4, 14.6.

HRMS (ESI) m/z calcd. for $\text{C}_{25}\text{H}_{26}\text{NO}_2\text{S}$ $[\text{M} + \text{H}]^+$ 404.1679, found 404.1679.

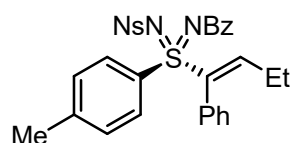
HPLC analysis: Chiralcel IC (n -hexane/ i -PrOH = 50/50, flow rate 0.6 mL/min, $\lambda = 254$ nm), t_R (minor) = 24.23 min, t_R (major) = 27.64 min.

The synthesis of sulfondiimine from sulfimide:



According to the literature reported procedure.¹⁸ To a solution of **19** (74.7 mg, 0.20 mmol, 1.0 equiv), PhI=NNs (363.6 mg, 0.90 mmol, 4.5 equiv), and AgNTf_2 (12.4 mg, 0.032 mmol, 16 mol%) in MeCN (4.0 mL) was added 4-*tert*-butyl-2,6-bis(4-*tert*-butylpyridin-2-yl)pyridine ($t\text{Bu}_3\text{tpy}$, 19.2 mg, 0.032 mmol, 16 mol%) under an argon atmosphere. The reaction mixture was heated to 50°C and stirred for 96 h. After cooling to rt, the reaction mixture was extracted with DCM three times (3×20 mL). The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4 , filtered, and concentrated to afford the crude product, which was purified by silica gel chromatography to afford the product **51**.

(*S*)-*N*-((((4-Nitrophenyl)sulfonyl)imino)(1-phenylbut-1-en-1-yl)(*p*-tolyl)- λ^6 -sulfaneylidene)benzamide (**51**)



The product mixture was purified by silica gel column chromatography (petroleum ether/ethyl acetate = 5/1) to afford **51** as a colorless oil (37.8 mg, 33% yield, 92% ee).

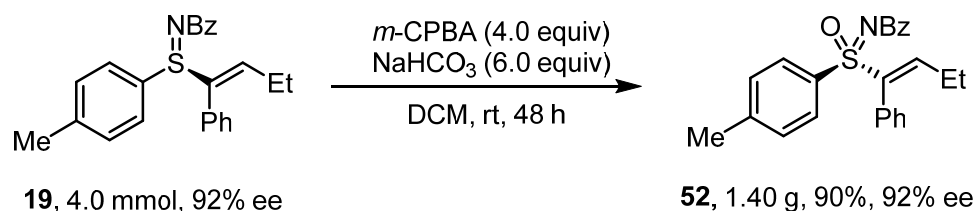
^1H NMR (400 MHz, CDCl_3) δ 7.96 (d, $J = 8.8$ Hz, 2H), 7.82 (d, $J = 6.7$ Hz, 2H), 7.80 – 7.66 (m, 5H), 7.48 (t, $J = 7.4$ Hz, 1H), 7.36 – 7.26 (m, 5H), 7.17 (t, $J = 7.7$ Hz, 2H), 6.81 (d, $J = 7.3$ Hz, 2H), 2.46 (s, 3H), 2.20 – 1.99 (m, 2H), 1.10 (t, $J = 7.5$ Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 172.2, 149.2, 148.7, 148.1, 145.6, 136.9, 134.5, 132.9, 131.7, 130.8, 130.3, 130.1, 129.2, 128.8, 128.7, 128.5, 128.1, 127.5, 123.7, 23.9, 21.8, 13.0.

HRMS (ESI) m/z calcd. for $\text{C}_{30}\text{H}_{27}\text{N}_3\text{O}_5\text{S}_2\text{Na}$ $[\text{M} + \text{Na}]^+$ 596.1284, found 596.1285.

HPLC analysis: Chiralcel IG (*n*-hexane/*i*-PrOH = 50/50, flow rate 0.6 mL/min, $\lambda = 254$ nm), t_R (minor) = 29.12 min, t_R (major) = 34.81 min.

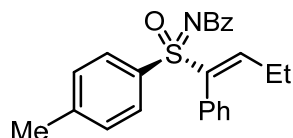
The synthesis of sulfoximine from sulfamide 19:



According to the literature reported procedure.¹⁷ To a solution of **19** (1.47 g, 4.0 mmol, 1.0 equiv) in DCM (100.0 mL), was added 3-Chloroperoxybenzoic Acid (*m*-CPBA,

2.76 g, 16.0 mmol, 4.0 equiv) and NaHCO₃ (2.02 g, 24.0 mmol, 6.0 equiv) in a 250 mL round-bottom flask with magnetic stir bar. Then the mixture was stirred at rt for 48 h. After the reaction finished that monitored by TLC, the reaction mixture was extracted with DCM (3 × 50 mL) and 2.0 M aqueous NaOH solution three times (3 × 100 mL). The combined organic layers were dried over anhydrous Na₂SO₄, filtered, and concentrated to afford the crude product, which was purified by silica gel chromatography to afford the product **52** as a colorless oil.

(S)-N-(Oxo(1-phenylbut-1-en-1-yl)(p-tolyl)-λ⁶-sulfaneylidene)benzamide (52**)**



The reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 5:1) to afford product **52** as a colorless oil (1.40 g, 90% yield, 92% ee).

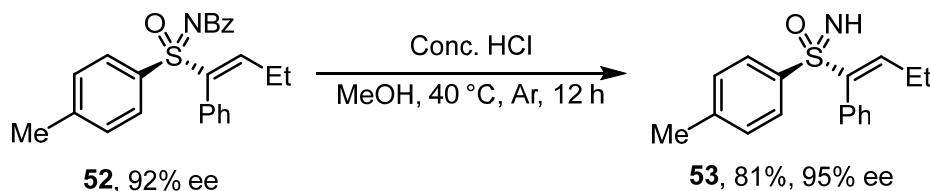
¹H NMR (400 MHz, CDCl₃) δ 8.12 (d, *J* = 6.9 Hz, 2H), 7.60 (d, *J* = 8.4 Hz, 2H), 7.54 – 7.45 (m, 1H), 7.43 – 7.34 (m, 3H), 7.33 – 7.27 (m, 1H), 7.25 – 7.16 (m, 4H), 6.91 (d, *J* = 6.8 Hz, 2H), 2.41 (s, 3H), 2.15 – 1.96 (m, 2H), 1.05 (t, *J* = 7.5 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 174.2, 145.8, 144.4, 140.1, 136.4, 134.0, 132.0, 131.1, 130.4, 129.8, 129.5, 129.2, 128.6, 128.3, 128.1, 23.2, 21.8, 13.2.

HRMS (ESI) *m/z* calcd. for C₂₄H₂₄NO₂S [M + H]⁺ 390.1522, found 390.1519.

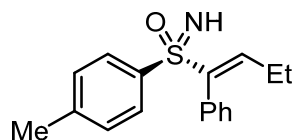
HPLC analysis: Chiralpak IH (*n*-hexane/*i*-PrOH = 80/20, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (minor) = 15.03 min, *t_R* (major) = 17.71 min.

The deprotection of sulfoximine:



According to the literature reported procedure.¹⁸ To a solution of **52** (77.9 mg, 0.20 mmol, 1.0 equiv) in MeOH (2.0 mL) was added HCl (Conc., 1.0 mL) under argon. The reaction mixture was heated to 40 °C for 12 h. After cooling to rt, the reaction mixture was quenched with sat. Na₂CO₃ aqueous solution, and extracted with Et₂O. The combined organic layers were washed with brine, dried over anhydrous Na₂SO₄, and concentrated to afford the crude product, which was purified by silica gel chromatography to afford the product **53** as a colorless oil.

(S)-Imino(1-phenylbut-1-en-1-yl)(p-tolyl)-λ⁶-sulfanone (53**)**



The reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 3:1) to afford product **53** as a colorless oil (46.3 mg, 81% yield, 95% ee).

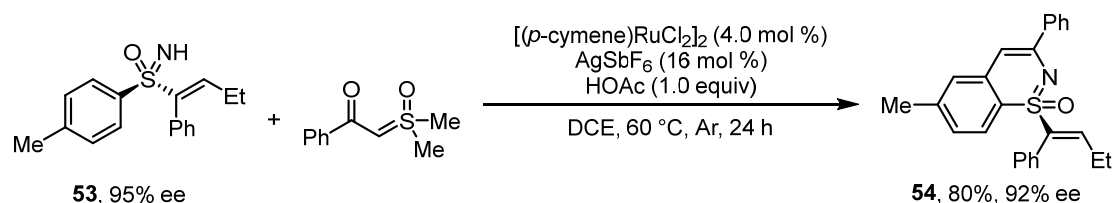
¹H NMR (400 MHz, CDCl₃) δ 7.56 (d, *J* = 8.3 Hz, 2H), 7.35 – 7.21 (m, 3H), 7.18 (d, *J* = 8.1 Hz, 2H), 7.14 (t, *J* = 7.6 Hz, 1H), 6.93 (d, *J* = 6.7 Hz, 2H), 2.39 (s, 3H), 2.23 (s, 1H), 1.98 (p, *J* = 7.5 Hz, 2H), 1.00 (t, *J* = 7.5 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 144.3, 143.5, 142.7, 137.6, 131.8, 130.7, 129.4, 128.9, 128.7, 128.2, 22.9, 21.6, 13.3.

HRMS (ESI) *m/z* calcd. for C₁₇H₂₀NOS [M + H]⁺ 286.1260, found 286.1260.

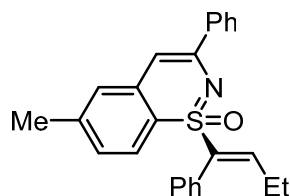
HPLC analysis: Chiralpak IH (*n*-hexane/*i*-PrOH = 70/30, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (major) = 21.49 min, *t_R* (minor) = 23.81 min.

Ru-catalyzed C-H activation-annulation with a sulfonium ylide:



According to the literature reported procedure.¹⁹ In a Schlenk tube equipped with a stir bar, **53** (28.5 mg, 0.10 mmol, 1.0 equiv), 2-(dimethyl(oxo)-sulfanylidene)-1-phenylethan-1-one (23.5 mg, 0.12 mmol, 1.2 equiv), $[(p\text{-cymene})\text{RuCl}_2]_2$ (2.45 mg, 4.0 mol %), AgSbF_6 (5.50 mg, 16 mol%) and HOAc (60.0 mg, 0.10 mol, 1.0 equiv) were dissolved in DCE (1.0 mL). The mixture was stirred at 60 °C for 24 h. The reaction mixture was evaporated under vacuum and the residue was purified by silica gel chromatography afforded the product **54** as a colorless oil.

(R)-6-Methyl-3-phenyl-1-(1-phenylbut-1-en-1-yl)-1 λ^4 -benzo[e][1,2]thiazine 1-oxide (**54**)



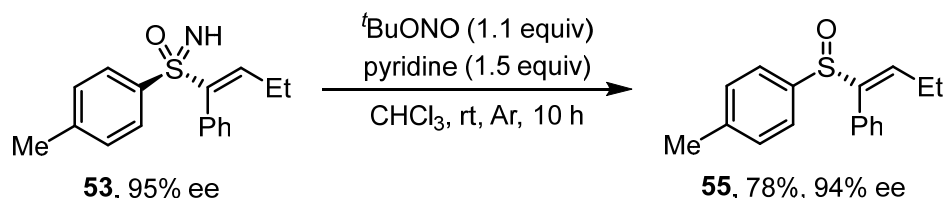
The product mixture was purified by silica gel column chromatography (petroleum ether/ethyl acetate = 10/1) to afford **54** as a colorless oil (30.9 mg, 80% yield, 92% ee). ^1H NMR (400 MHz, CDCl_3) δ 7.99 – 7.77 (m, 2H), 7.47 – 7.30 (m, 4H), 7.27 (t, J = 8.1 Hz, 1H), 7.22 – 7.10 (m, 5H), 7.04 (d, J = 8.1 Hz, 1H), 7.01 (s, 1H), 6.36 (s, 1H), 2.35 (s, 5H), 2.24 – 2.05 (m, 2H), 1.10 (t, J = 7.5 Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ 147.7, 145.2, 143.0, 140.6, 139.2, 138.1, 131.3, 131.0, 128.7, 128.6, 128.3, 128.2, 127.3, 126.7, 126.4, 125.0, 114.5, 97.3, 23.1, 21.9, 13.4.

HRMS (ESI) m/z calcd. for $\text{C}_{25}\text{H}_{24}\text{NOS}$ $[\text{M} + \text{H}]^+$ 386.1573, found 386.1568.

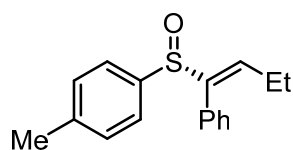
HPLC analysis: Chiralcel AD-H (n -hexane/ i -PrOH = 50/50, flow rate 0.6 mL/min, λ = 254 nm), t_R (major) = 18.75 min, t_R (minor) = 22.99 min.

The synthesis of sulfoxide:



According to the literature reported procedure.¹⁸ To a solution of **53** (57.1 mg, 0.20 mmol, 1.0 equiv) in MeCN (4.0 mL) was added $t\text{BuONO}$ (26.0 μL , 0.22 mmol, 1.1 equiv) and pyridine (25.0 μL , 0.30 mmol, 1.5 equiv) under argon. The reaction mixture was stirred at rt for 10 h. Then, the reaction mixture was concentrated. Purification by silica gel chromatography afforded the product **55** as a colorless oil.

(S)-1-Methyl-4-((1-phenylbut-1-en-1-yl)sulfinyl)benzene (55)



The product mixture was purified by silica gel column chromatography (petroleum ether/ethyl acetate = 5/1) to afford **52** as a colorless oil (42.2 mg, 78% yield, 94% ee).

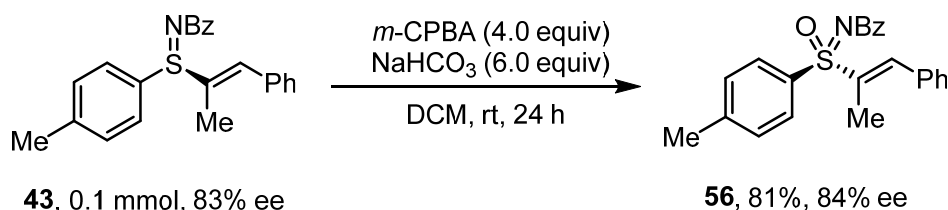
¹H NMR (400 MHz, CDCl₃) δ 7.30 – 7.14 (m, 5H), 7.10 (d, *J* = 8.1 Hz, 2H), 6.93 – 6.80 (m, 2H), 6.59 (t, *J* = 7.6 Hz, 1H), 2.32 (s, 3H), 2.15 (p, *J* = 7.5 Hz, 2H), 1.04 (t, *J* = 7.5 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 144.9, 141.3, 139.7, 135.5, 131.6, 129.8, 129.5, 128.4, 128.3, 125.2, 22.2, 21.5, 13.8.

HRMS (ESI) *m/z* calcd. for C₁₇H₁₉OS [M + H]⁺ 271.1151, found 271.1149.

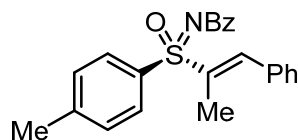
HPLC analysis: Chiralcel IG (*n*-hexane/*i*-PrOH = 70/30, flow rate 0.5 mL/min, λ = 254 nm), *t_R* (major) = 16.19 min, *t_R* (minor) = 17.08 min.

The synthesis of sulfoximine from sulfamide 43:



According to the literature reported procedure.¹⁷ To a solution of **43** (35.9 mg, 0.1 mmol, 1.0 equiv) in DCM (4.0 mL), was added 3-Chloroperoxybenzoic Acid (*m*-CPBA, 69.0 mg, 0.4 mmol, 4.0 equiv) and NaHCO₃ (50.4 mg, 0.6 mmol, 6.0 equiv) in a 25 mL round-bottom flask with magnetic stir bar. Then the mixture was stirred at rt for 24 h. After the reaction finished that monitored by TLC, the reaction mixture was extracted with DCM and 2.0 M aqueous NaOH solution three times. The combined organic layers were dried over anhydrous Na₂SO₄, filtered, and concentrated to afford the crude product, which was purified by silica gel chromatography to afford the product **56** as a colorless oil.

(R)-N-(Oxo(1-phenylprop-1-en-2-yl)(p-tolyl)-λ⁶-sulfaneylidene)benzamide (56)



The reaction mixture was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 5:1) to afford product **56** as a colorless oil (30.4 mg, 90% yield, 84% ee).

¹H NMR (400 MHz, CDCl₃) δ 8.25 (d, *J* = 7.1 Hz, 2H), 8.00 (s, 1H), 7.96 (d, *J* = 8.3 Hz, 2H), 7.62 – 7.50 (m, 1H), 7.48 – 7.30 (m, 9H), 2.45 (s, 3H), 2.15 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 173.9, 144.7, 138.4, 136.1, 136.0, 134.2, 134.0, 132.2,

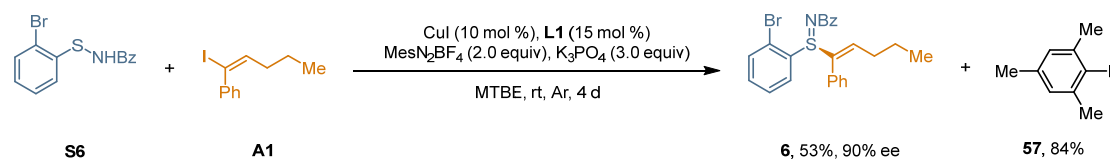
130.3, 129.9, 129.6, 129.5, 128.8, 128.3, 128.2, 21.8, 13.3.

HRMS (ESI) m/z calcd. for $C_{23}H_{22}NO_2S$ $[M + H]^+$ 376.1366, found 376.1364.

HPLC analysis: Chiralpak IH (n -hexane/ i -PrOH = 80/20, flow rate 0.5 mL/min, λ = 254 nm), t_R (minor) = 22.14 min, t_R (major) = 30.48 min.

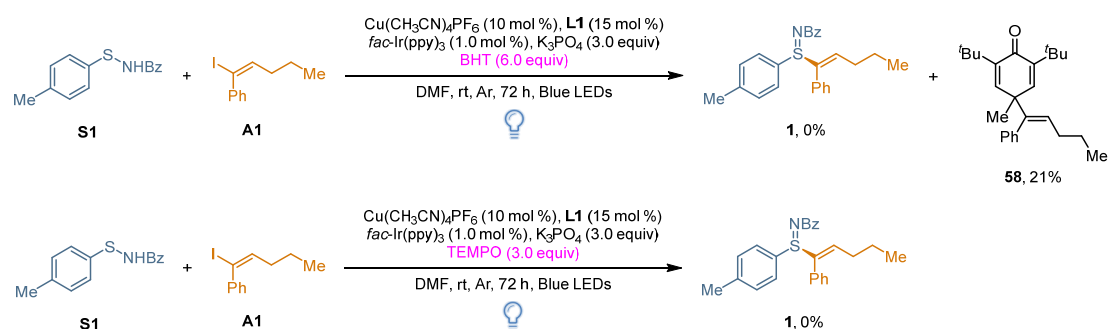
7. Mechanistic investigation

Control experiments of standard conditions with diazonium salts for S6 and A1



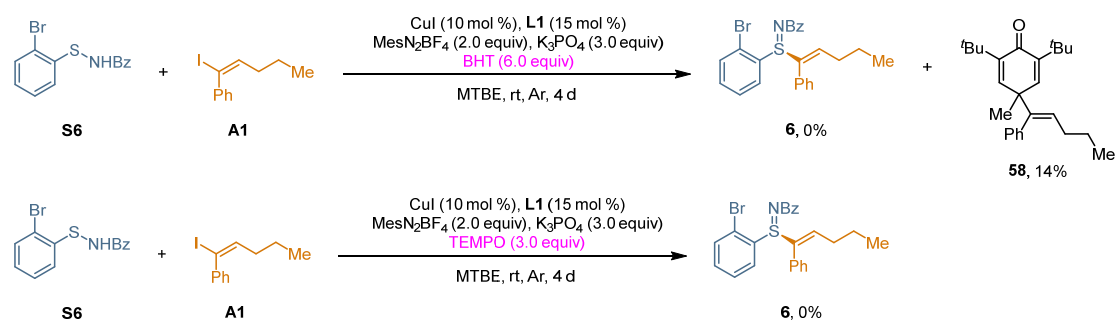
According to **General procedure B**, under an argon atmosphere, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with sulfenamide **S6** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A1** (81.7 mg, 0.3 mmol, 1.5 equiv), CuI (3.80 mg, 0.02 mmol, 10 mol %), **L1** (10.5 mg, 0.03 mmol, 15 mol %), BHT (6.0 equiv) or TEMPO (3.0 equiv), MesN₂BF₄ (93.6 mg, 0.40 mmol, 2.0 equiv) and K₃PO₄ (127.4 mg, 0.6 mol, 3.0 equiv) in MTBE (4.0 mL) stirred at rt for 4 days under argon. Upon completion, the precipitate was filtered off and washed by DCM. The filtrate was evaporated and the residue was purified by column chromatography on silica gel to afford the product.

Radical trapping experiments under standard conditions with photoinduced



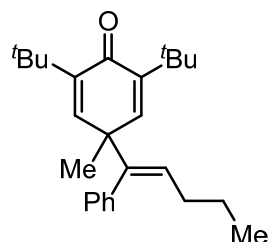
According to **General procedure A**, under an argon atmosphere, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A1** (81.7 mg, 0.3 mmol, 1.5 equiv), Cu(CH₃CN)₄PF₆ (7.45 mg, 0.02 mmol, 10 mol %), **L1** (10.5 mg, 0.03 mmol, 15 mol %), BHT (6.0 equiv) or TEMPO (3.0 equiv), *fac*-Ir(ppy)₃ (1.30 mg, 1.0 mol %), and K₃PO₄ (127.4 mg, 0.6 mol, 3.0 equiv) in DMF (2.0 mL) stirred at rt and blue LEDs irradiation for 72 h under argon. Upon completion, the reaction mixture was extracted three times with Et₂O and organic phase was evaporated. The residue was purified by column chromatography on silica gel to afford the radical trapping adduct.

Radical trapping experiments under standard conditions with diazonium salts



According to **General procedure B**, under an argon atmosphere, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with sulfenamide **S6** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide **A1** (81.7 mg, 0.3 mmol, 1.5 equiv), CuI (3.80 mg, 0.02 mmol, 10 mol %), **L1** (10.5 mg, 0.03 mmol, 15 mol %), BHT (6.0 equiv) or TEMPO (3.0 equiv), MesN₂BF₄ (93.6 mg, 0.40 mmol, 2.0 equiv) and K₃PO₄ (127.4 mg, 0.6 mol, 3.0 equiv) in MTBE (4.0 mL) stirred at rt for 4 days under argon. Upon completion, the precipitate was filtered off and washed by DCM. The filtrate was evaporated and the residue was purified by column chromatography on silica gel to afford the radical trapping adduct.

(E)-2,6-Di-*tert*-butyl-4-methyl-4-(1-phenylpent-1-en-1-yl)cyclohexa-2,5-dien-1-one (58**)**



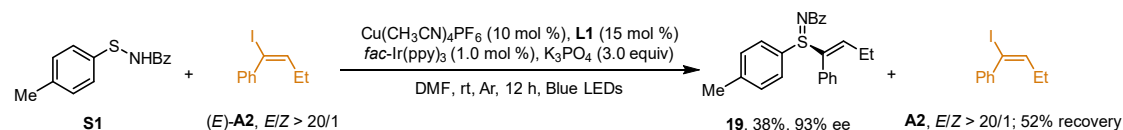
According to **General procedure A**, the product mixture was purified by silica gel column chromatography (petroleum ether/ethyl acetate = 100/1) to afford **58** as a colorless oil (15.3 mg, 21% yield).

¹H NMR (400 MHz, CDCl₃) δ 7.24 – 7.07 (m, 3H), 6.92 – 6.78 (m, 2H), 6.45 (s, 2H), 5.72 (t, *J* = 7.2 Hz, 1H), 1.73 (q, *J* = 7.3 Hz, 2H), 1.37 (s, 3H), 1.12 (s, 18H), 0.81 (t, *J* = 7.3 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ 186.2, 146.0, 145.4, 143.6, 138.9, 129.6, 128.4, 127.4, 126.8, 45.2, 34.7, 31.6, 29.4, 24.3, 23.0, 13.8.

HRMS (ESI) *m/z* calcd. for C₂₆H₃₆NaO [*M* + Na]⁺ 387.2658, found 387.3656.

Control experiments of alkenyl iodide (*E*)-A2 under standard conditions with photoinduced



According to **General procedure A**, under an argon atmosphere, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide (*E*)-**A2** (81.7 mg, 0.3 mmol, 1.5 equiv), $\text{Cu}(\text{CH}_3\text{CN})_4\text{PF}_6$ (7.45 mg, 0.02 mmol, 10 mol %), **L1** (10.5 mg, 0.03 mmol, 15 mol %), *fac*- $\text{Ir}(\text{ppy})_3$ (1.30 mg, 1.0 mol %), and K_3PO_4 (127.4 mg, 0.6 mol, 3.0 equiv) in DMF (2.0 mL) stirred at rt and blue LEDs irradiation for 12 h under argon. Then, the reaction mixture was extracted three times with Et_2O and organic phase was evaporated. The residue was purified by column chromatography on silica gel to afford product **19**.

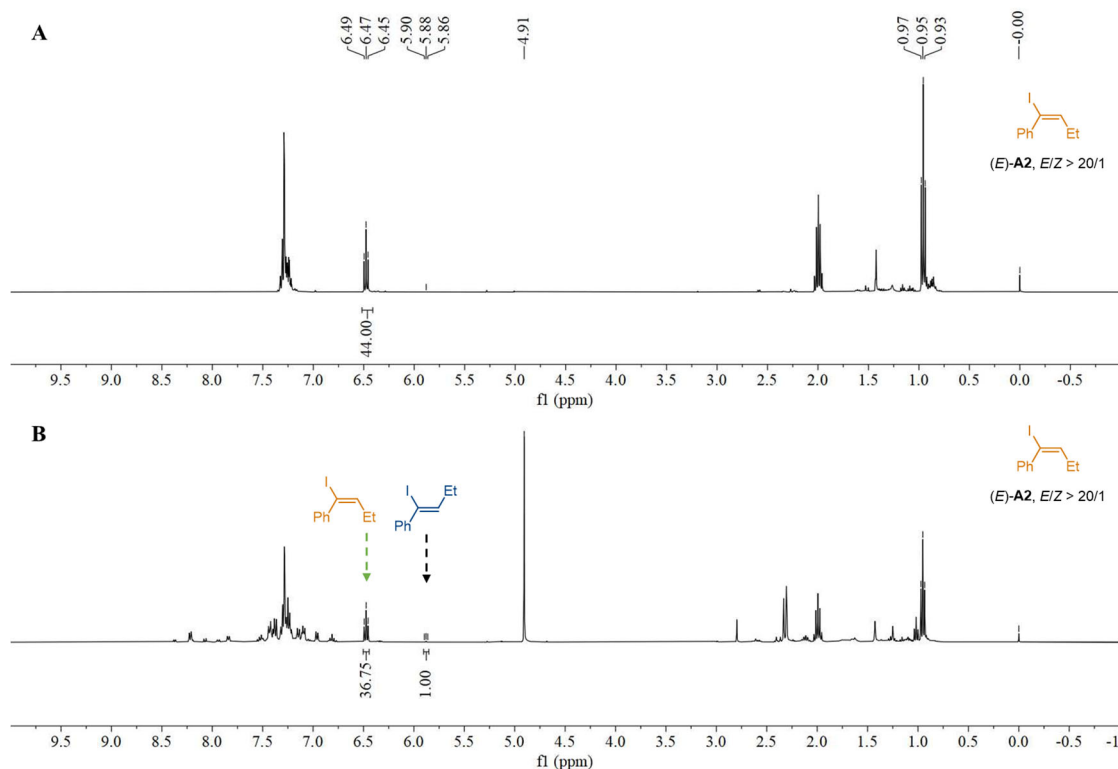
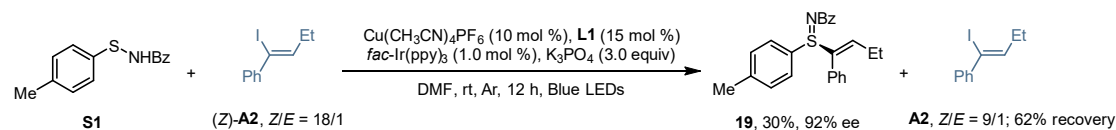


Figure S7'. Control experiments of alkenyl iodide (*E*)-**A2** with photoinduced. **(A)** ^1H NMR spectrum of alkenyl iodide (*E*)-**A2**; **(B)** The *E*/*Z* ratio of the recovered **A2** were based on ^1H NMR analysis of the crude reaction mixture using CH_2Br_2 as an internal standard.

Control experiments of alkenyl iodide (Z)-A2 under standard conditions with photoinduced



According to **General procedure A**, under an argon atmosphere, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide (Z)-**A2** (81.7 mg, 0.3 mmol, 1.5 equiv), $\text{Cu}(\text{CH}_3\text{CN})_4\text{PF}_6$ (7.45 mg, 0.02 mmol, 10 mol %), **L1** (10.5 mg, 0.03 mmol, 15 mol %), *fac*- $\text{Ir}(\text{ppy})_3$ (1.30 mg, 1.0 mol %), and K_3PO_4 (127.4 mg, 0.6 mol, 3.0 equiv) in DMF (2.0 mL) stirred at rt and blue LEDs irradiation for 12 h under argon. Then, the reaction mixture was extracted three times with Et_2O and organic phase was evaporated. The residue was purified by column chromatography on silica gel to afford product **19**.

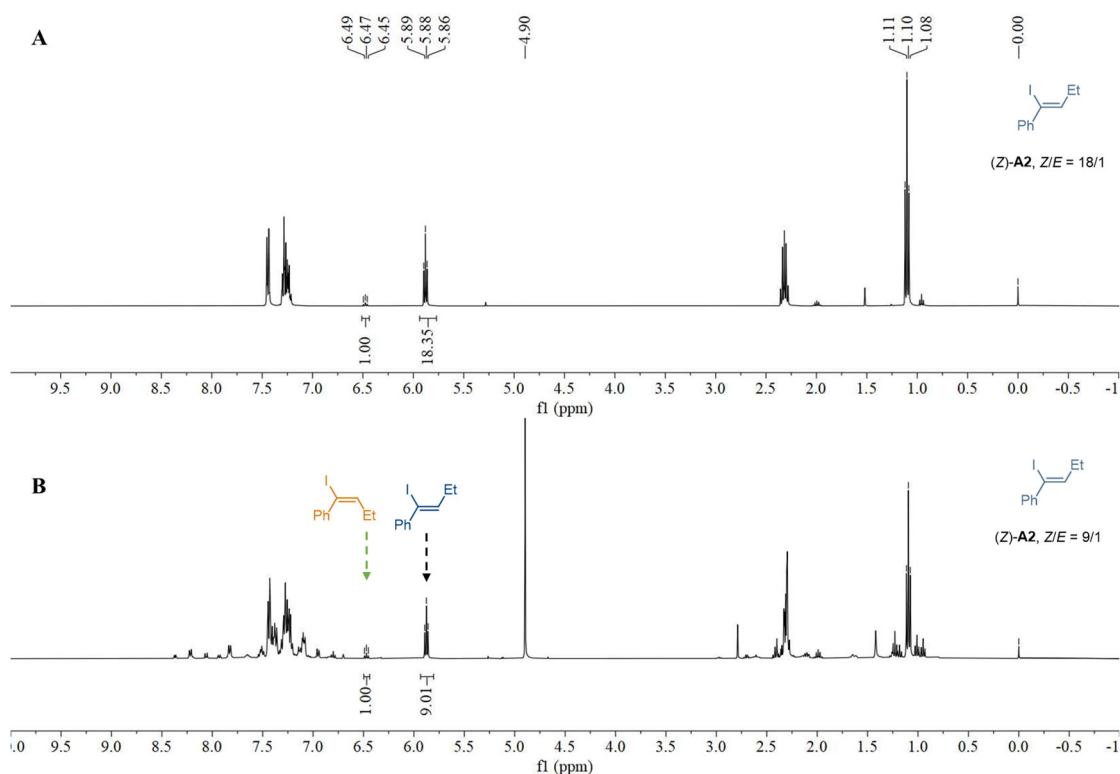
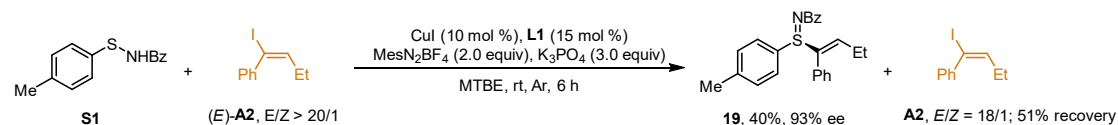


Figure S8'. Control experiments of alkenyl iodide (Z)-**A2** with photoinduced. **(A)** ^1H NMR spectrum of alkenyl iodide (Z)-**A2**; **(B)** The E/Z ratio of the recovered **A2** were based on ^1H NMR analysis of the crude reaction mixture using CH_2Br_2 as an internal standard.

Control experiments of alkenyl iodide (*E*)-A2 under standard conditions with diazonium salts



According to **General procedure B**, under an argon atmosphere, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide (*E*)-**A2** (81.7 mg, 0.3 mmol, 1.5 equiv), CuI (3.80 mg, 0.02 mmol, 10 mol %), **L1** (10.5 mg, 0.03 mmol, 15 mol %), MesN₂BF₄ (93.6 mg, 0.40 mmol, 2.0 equiv) and K₃PO₄ (127.4 mg, 0.6 mol, 3.0 equiv) in MTBE (4.0 mL) stirred at rt for 6 h under argon. Upon completion, the precipitate was filtered off and washed by DCM and the residue was purified by column chromatography on silica gel to afford product **19**.

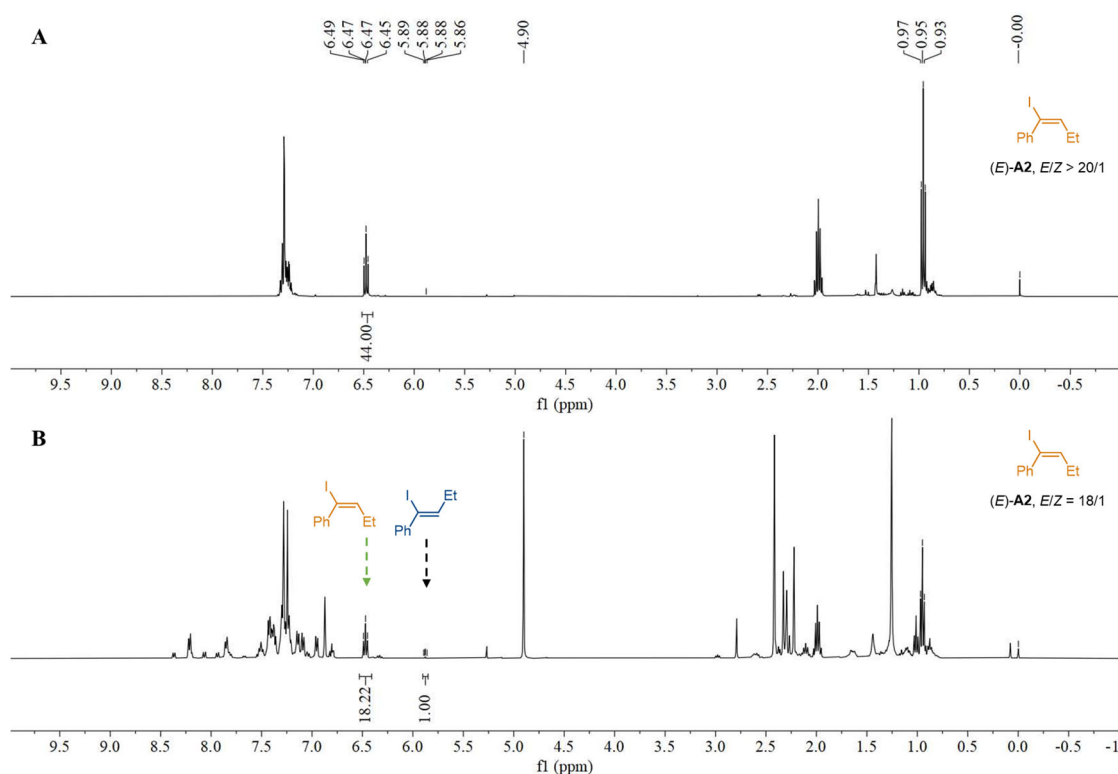
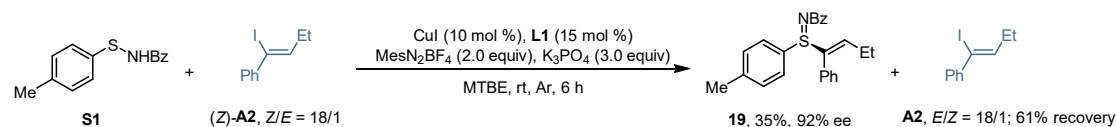


Figure S9'. Control experiments of alkenyl iodide (*E*)-**A2** with diazonium salts. **(A)** ¹H NMR spectrum of alkenyl iodide (*E*)-**A2**; **(B)** The *E/Z* ratio of the recovered **A2** were based on ¹H NMR analysis of the crude reaction mixture using CH₂Br₂ as an internal standard.

Control experiments of alkenyl iodide (Z)-A2 under standard conditions with diazonium salts



According to **General procedure B**, under an argon atmosphere, an oven-dried resealable Schlenk tube equipped with a magnetic stir bar was charged with sulfenamide **S1** (48.6 mg, 0.20 mmol, 1.0 equiv), alkenyl iodide (**Z**)-**A2** (81.7 mg, 0.3 mmol, 1.5 equiv), CuI (3.80 mg, 0.02 mmol, 10 mol %), **L1** (10.5 mg, 0.03 mmol, 15 mol %), MesN₂BF₄ (93.6 mg, 0.40 mmol, 2.0 equiv) and K₃PO₄ (127.4 mg, 0.6 mol, 3.0 equiv) in MTBE (4.0 mL) stirred at rt for 6 h under argon. Upon completion, the precipitate was filtered off and washed by DCM and the residue was purified by column chromatography on silica gel to afford product **19**.

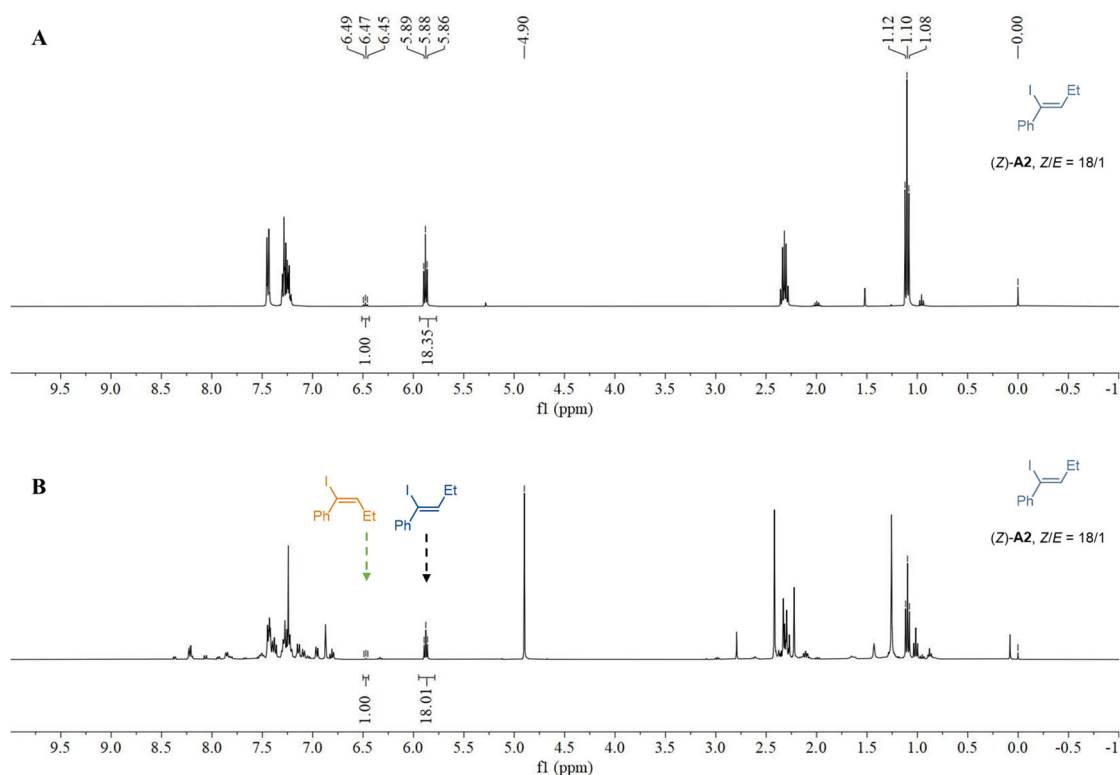


Figure S10'. Control experiments of alkenyl iodide (**Z**)-**A2** with diazonium salts. **(A)** ¹H NMR spectrum of alkenyl iodide (**Z**)-**A2**; **(B)** The *E/Z* ratio of the recovered **A2** were based on ¹H NMR analysis of the crude reaction mixture using CH₂Br₂ as an internal standard.

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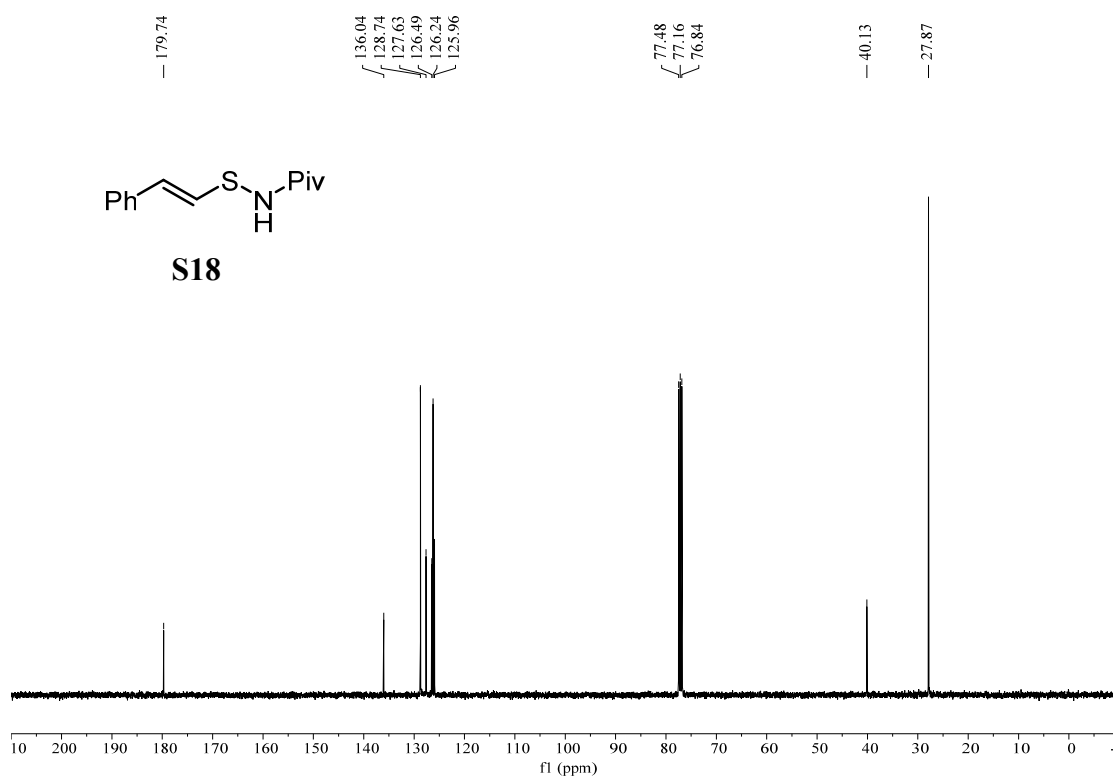
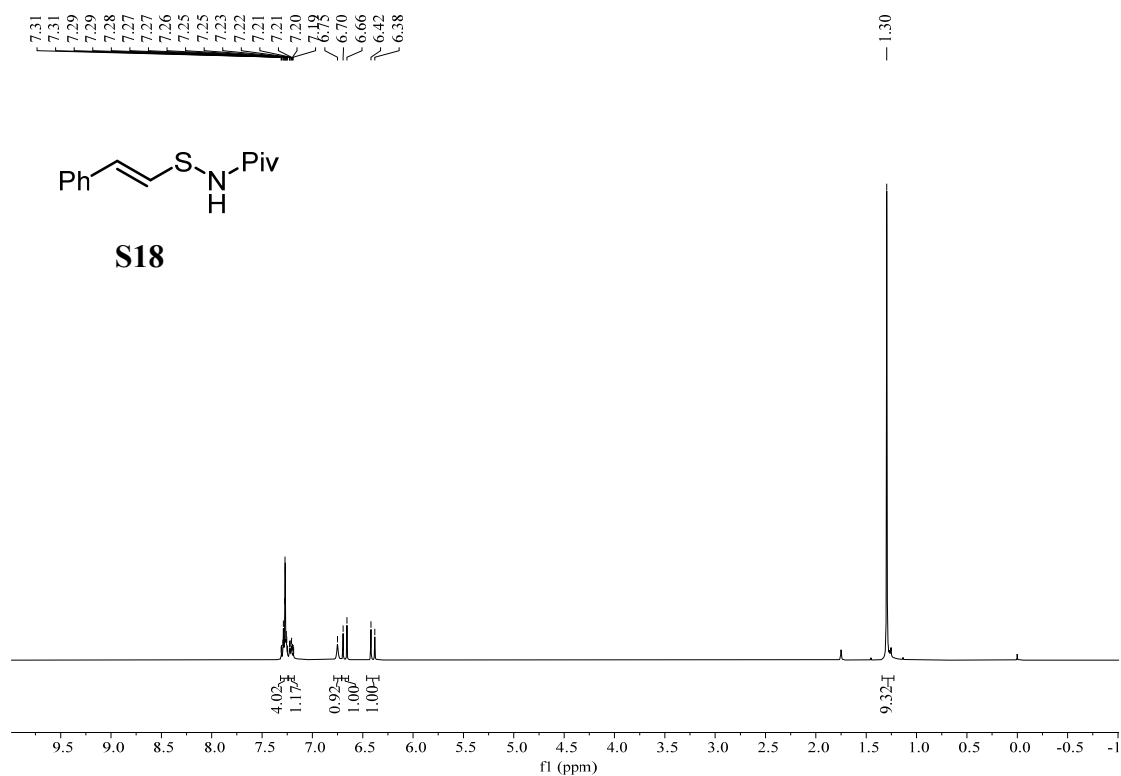
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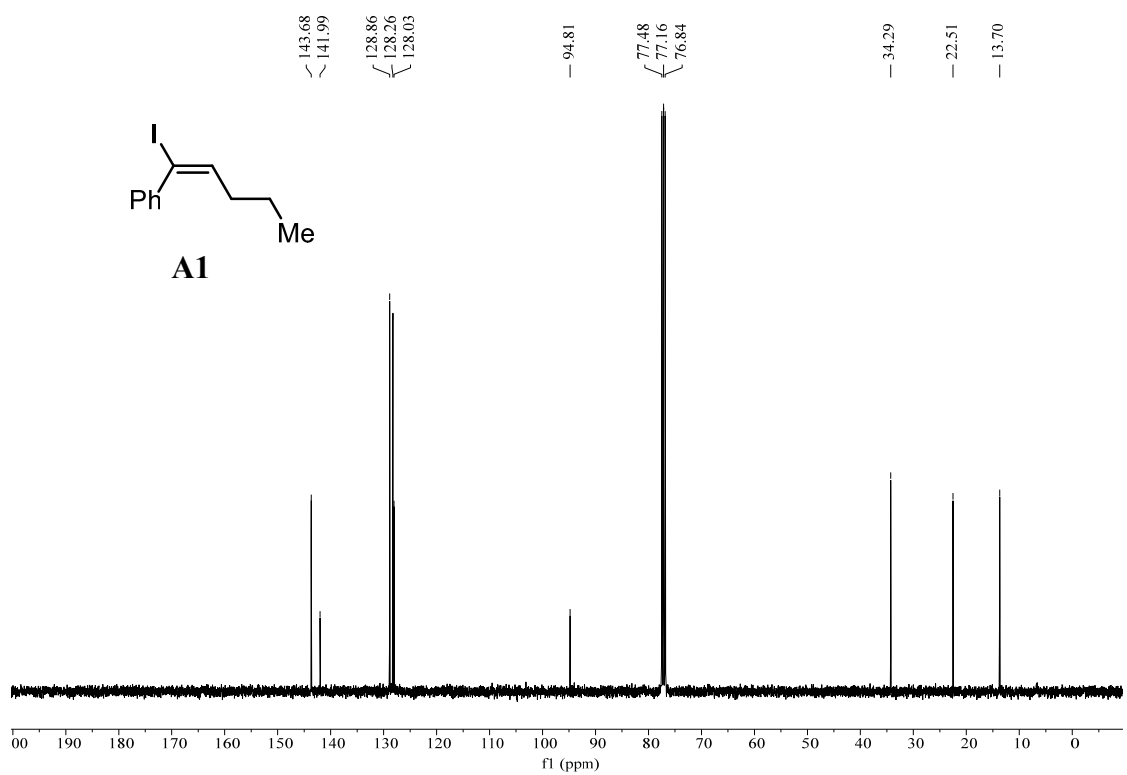
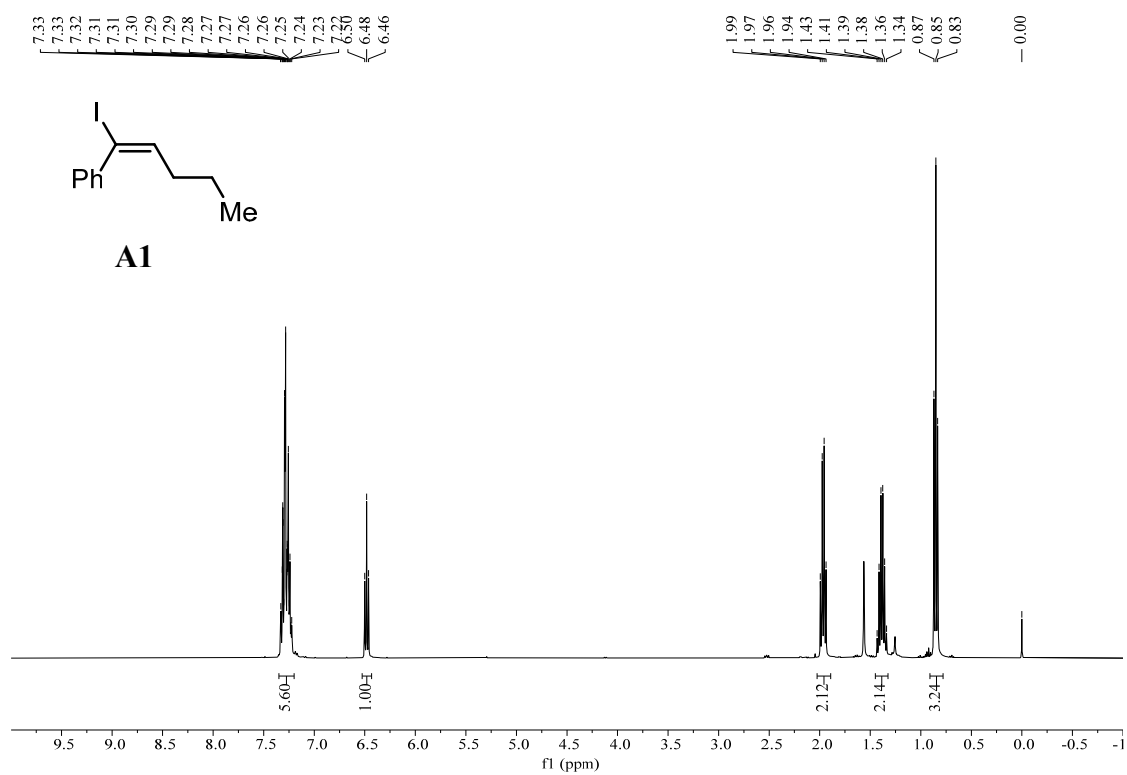
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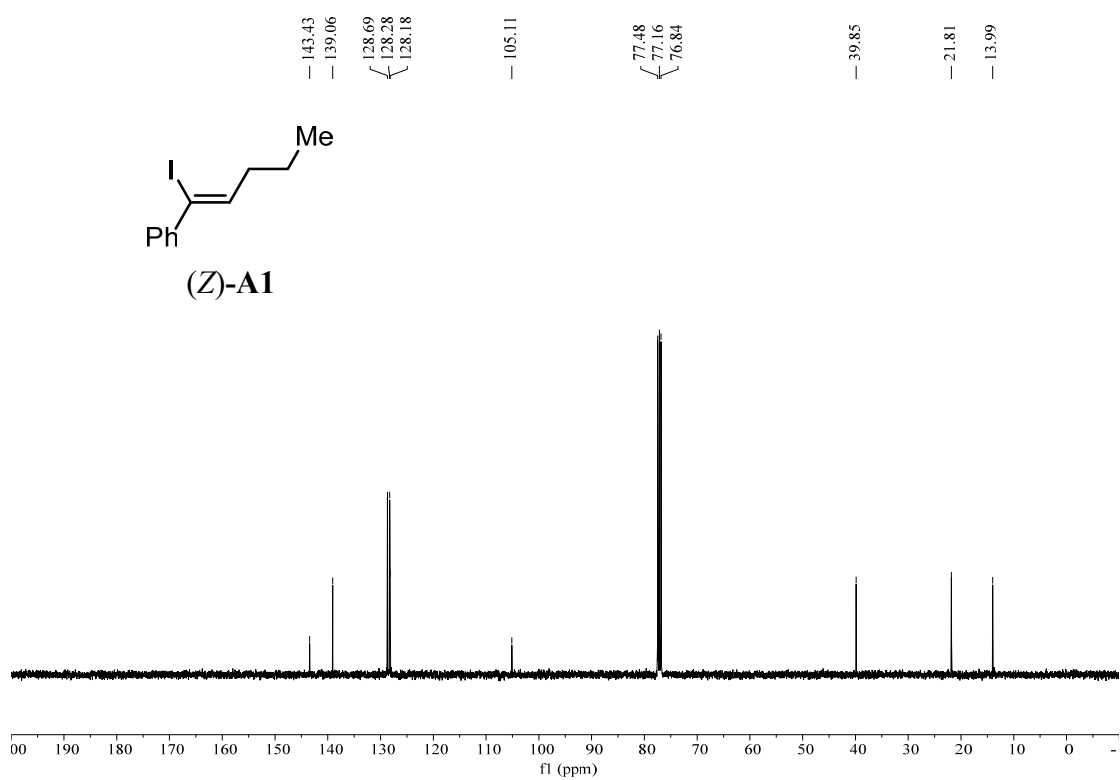
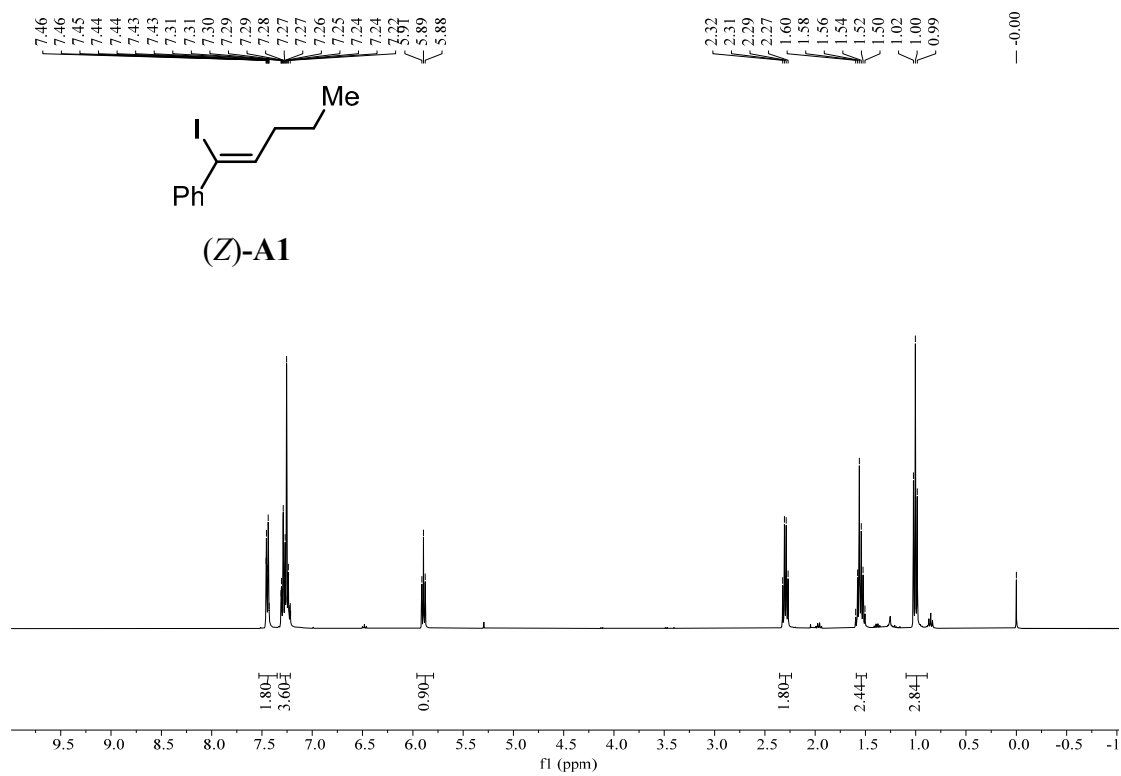
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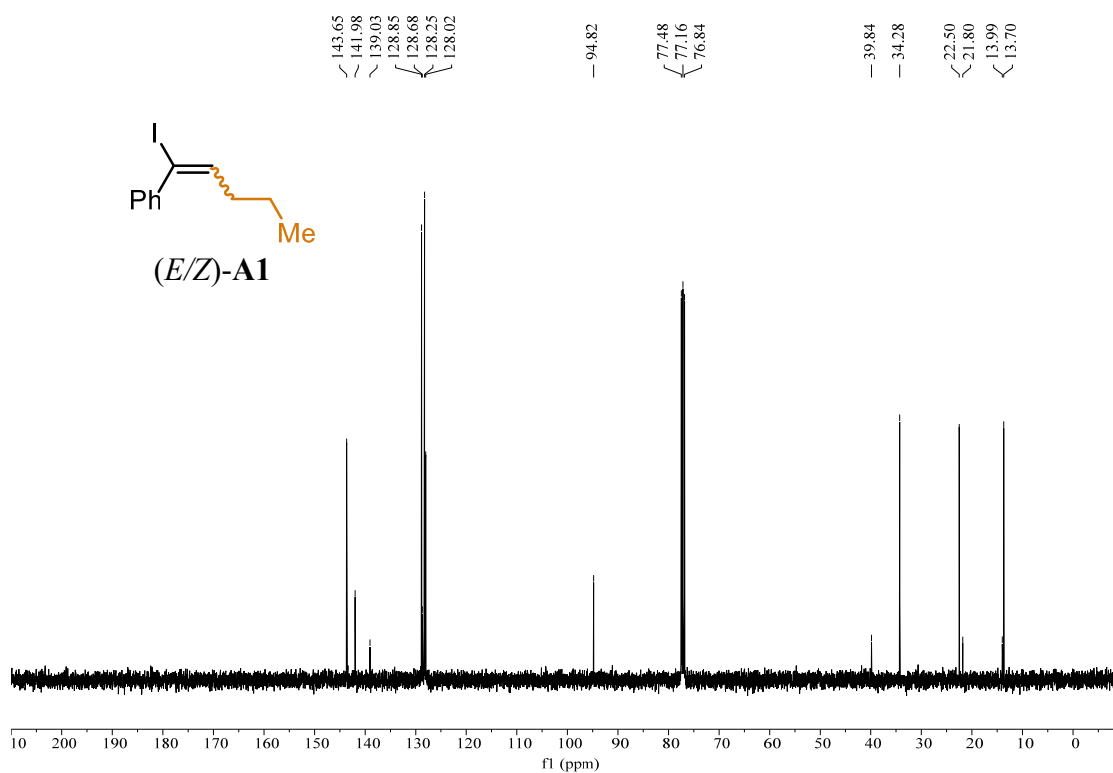
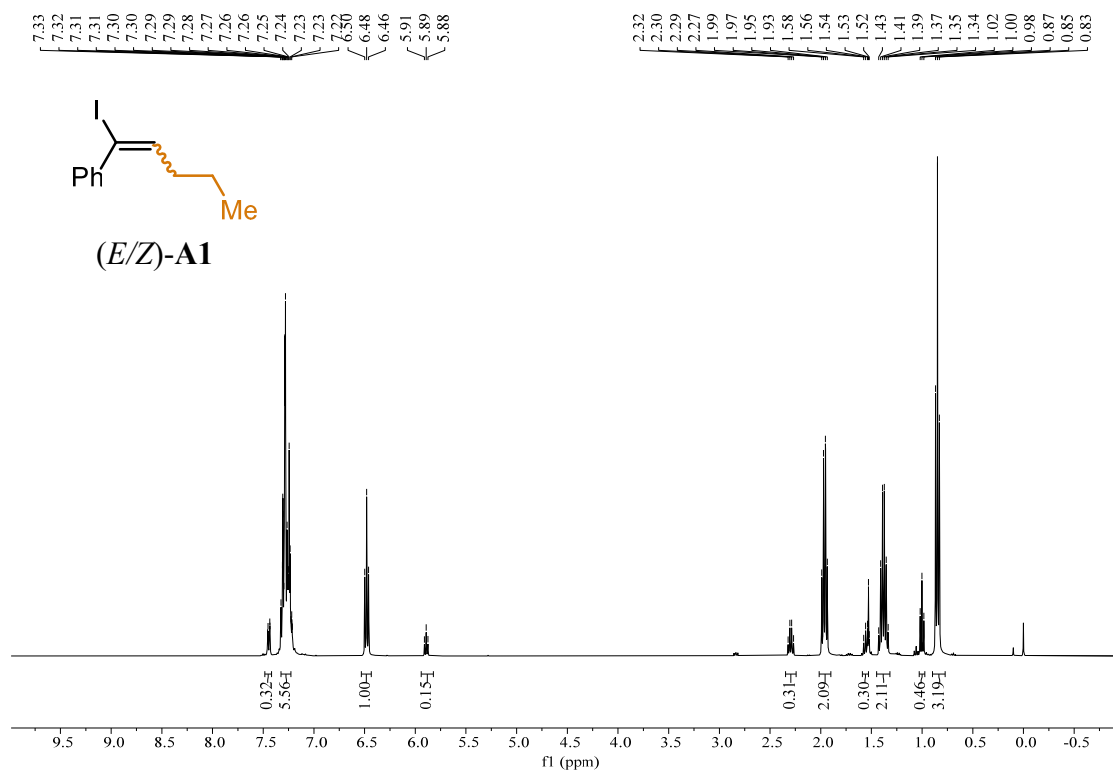
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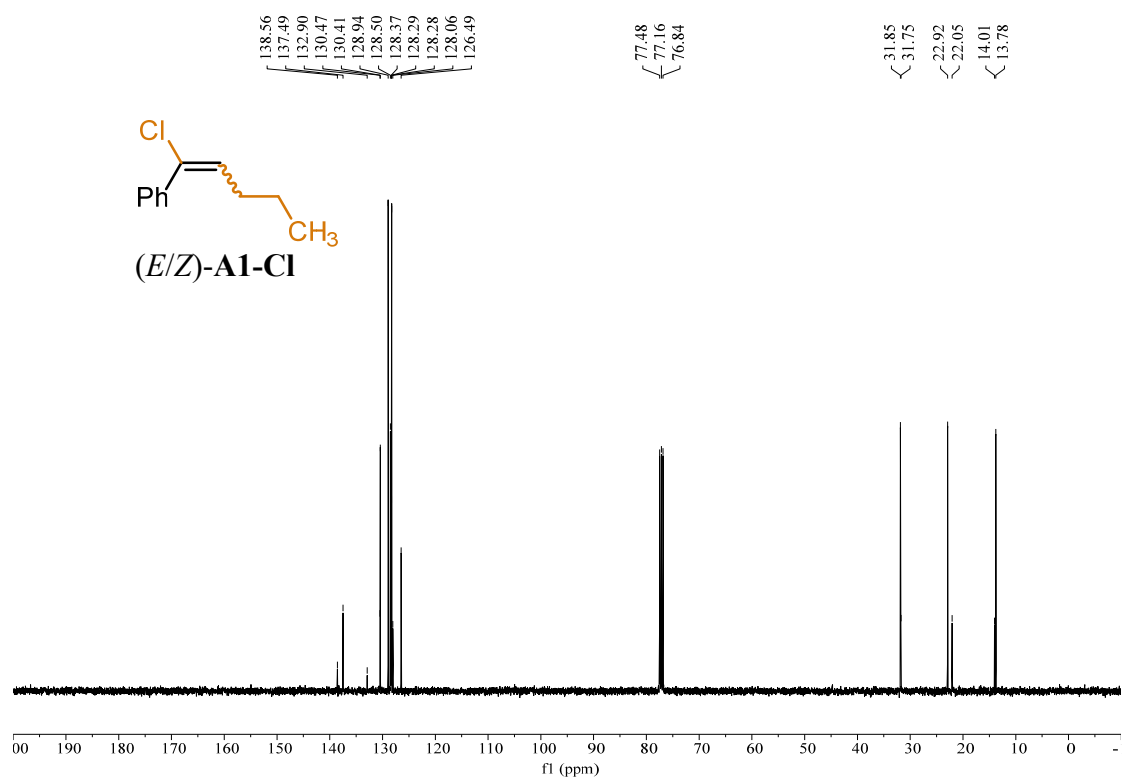
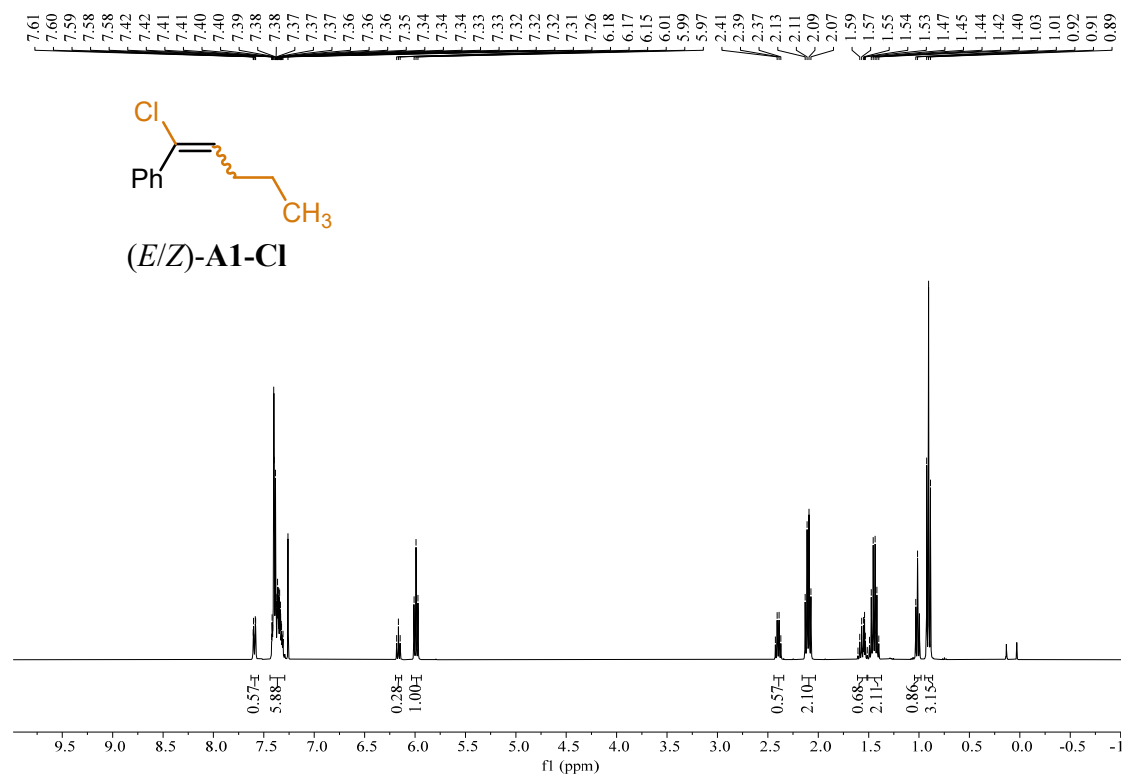
9. NMR spectra

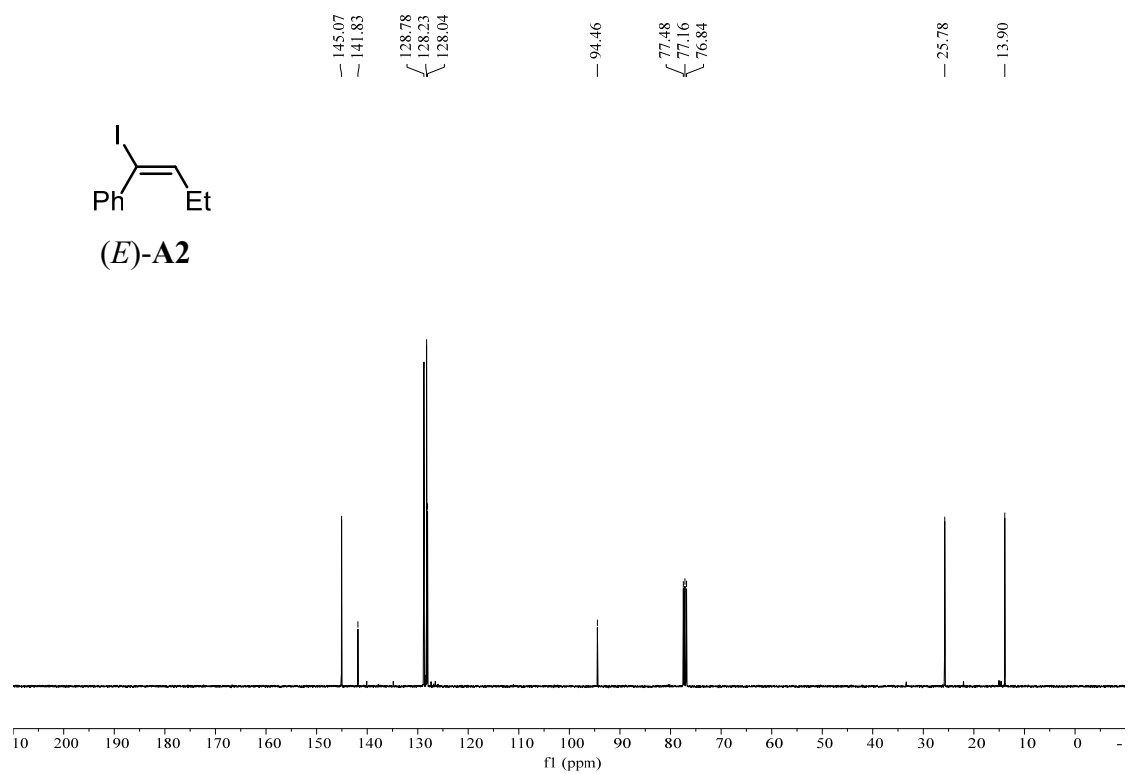
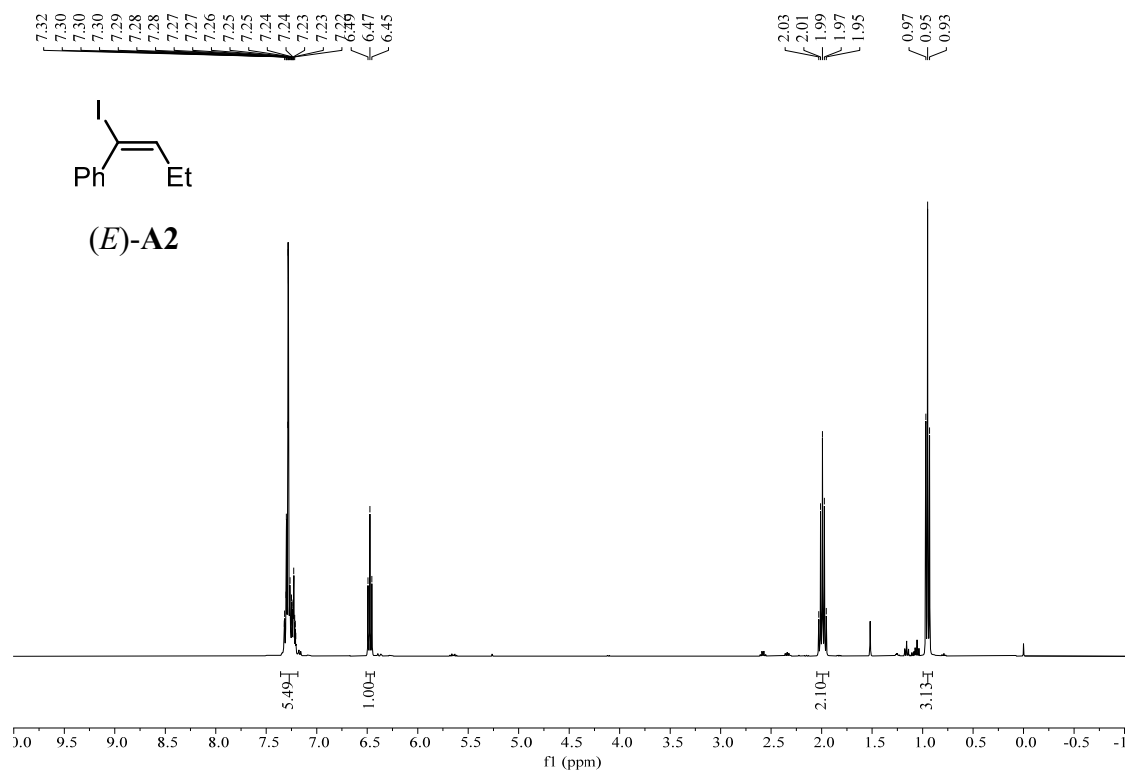


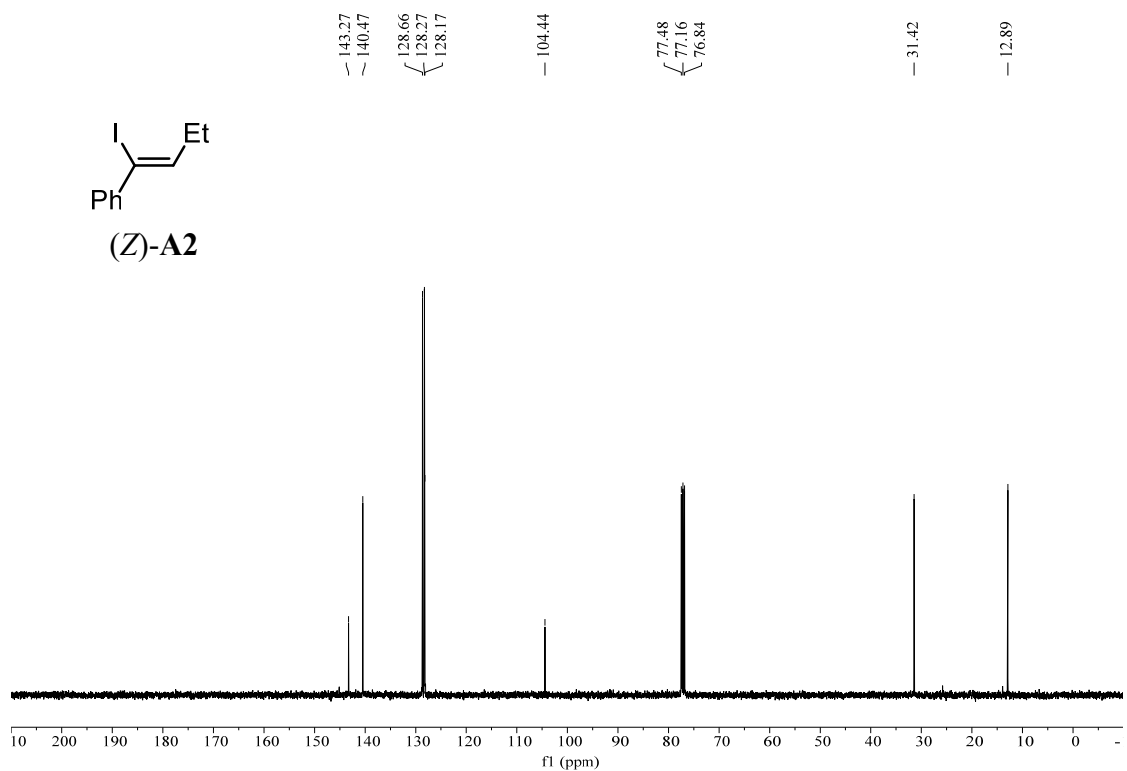
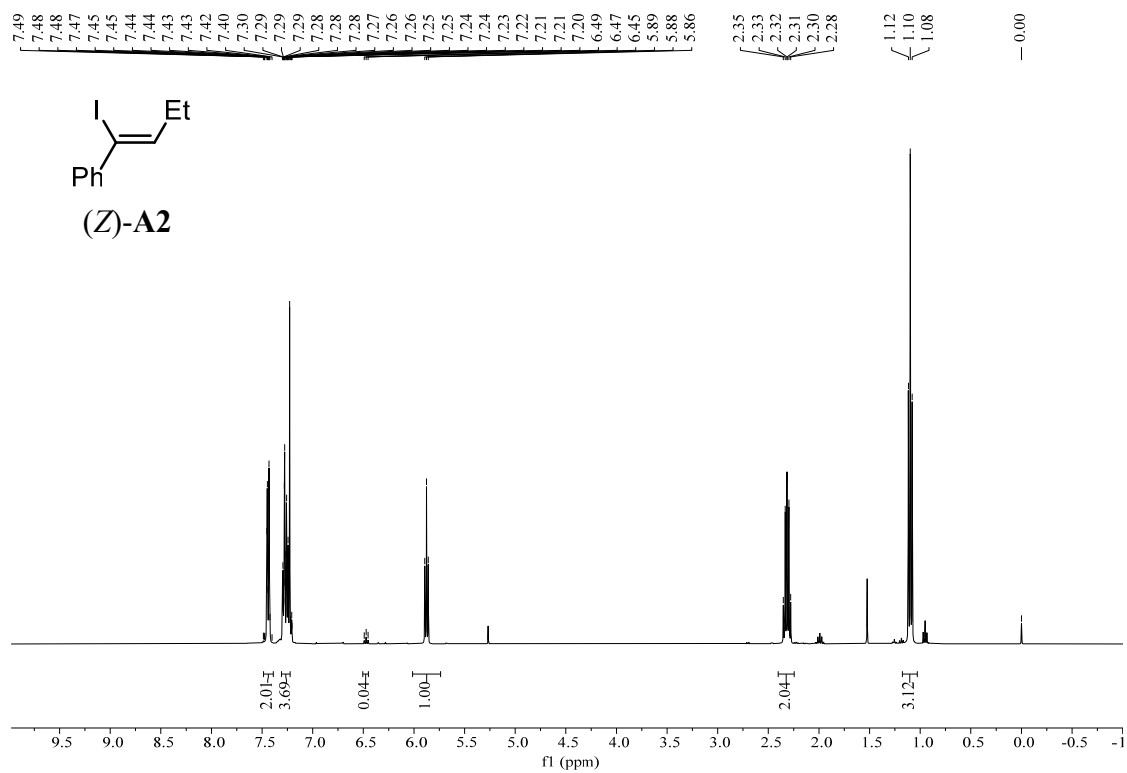


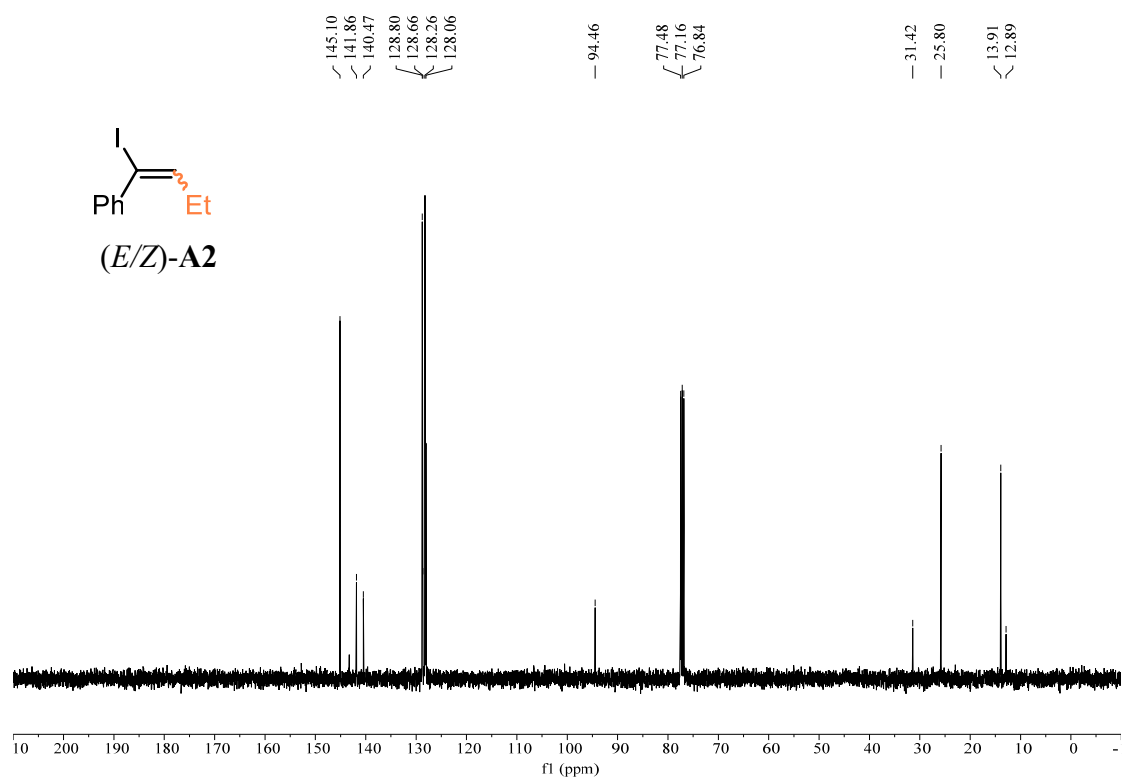
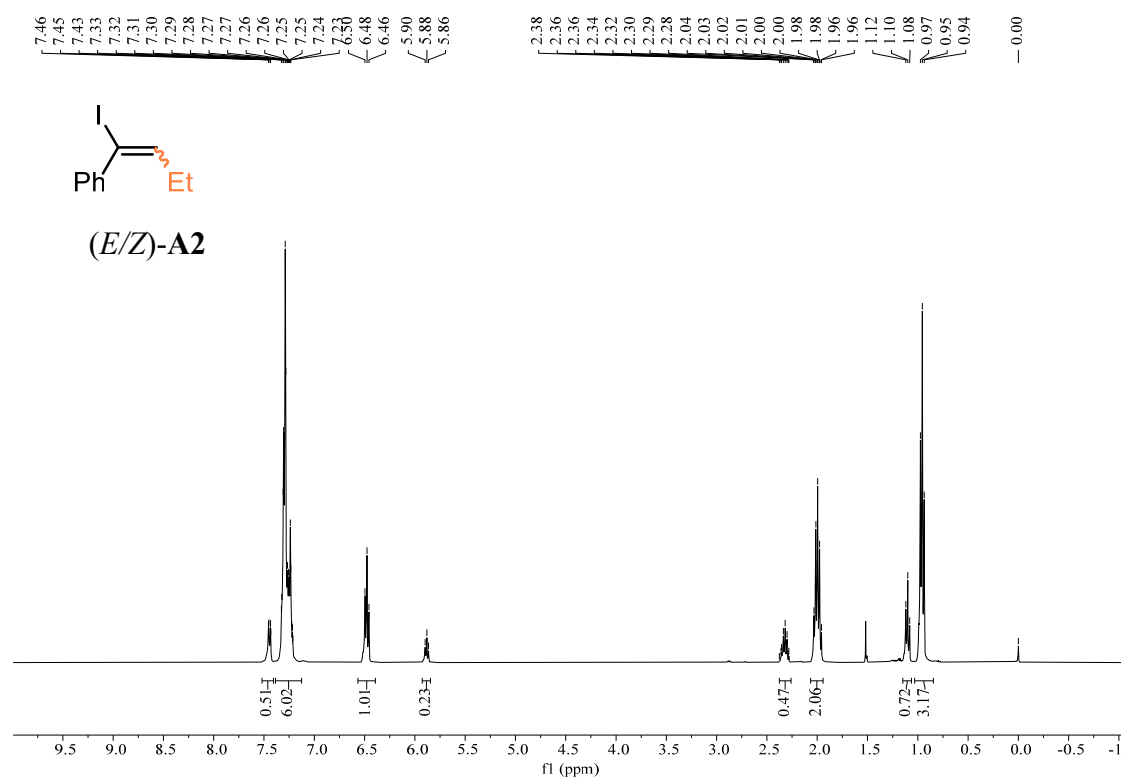


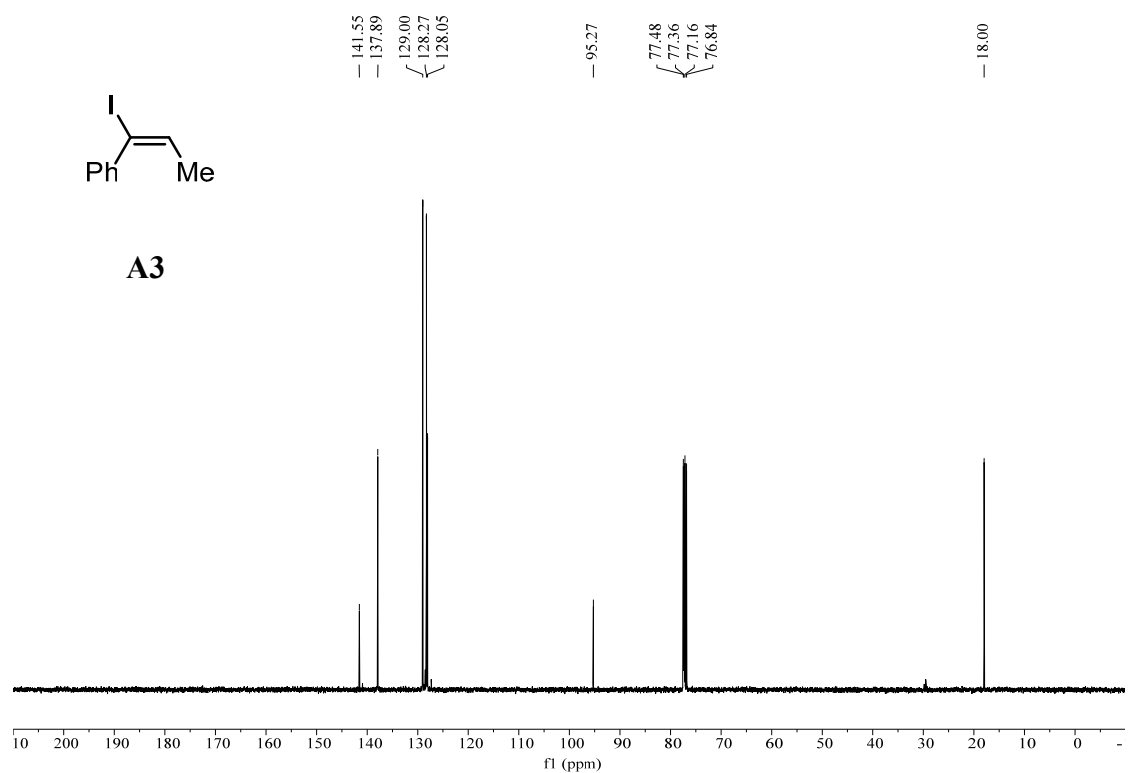
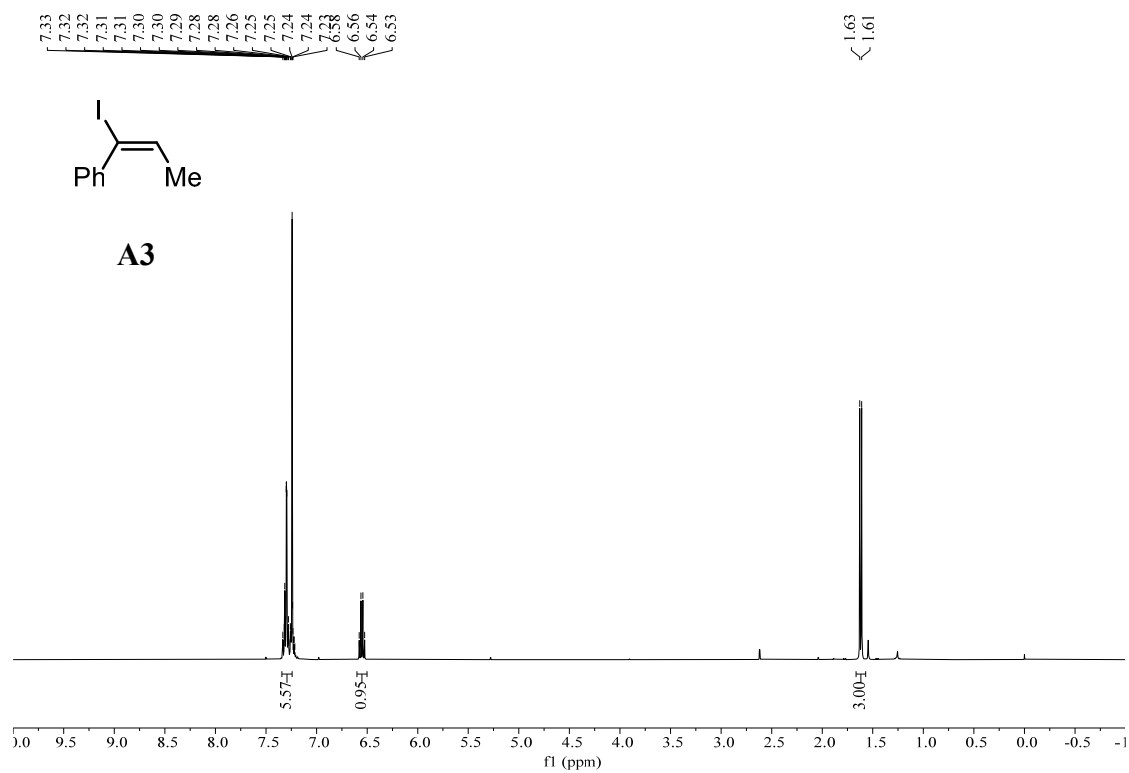


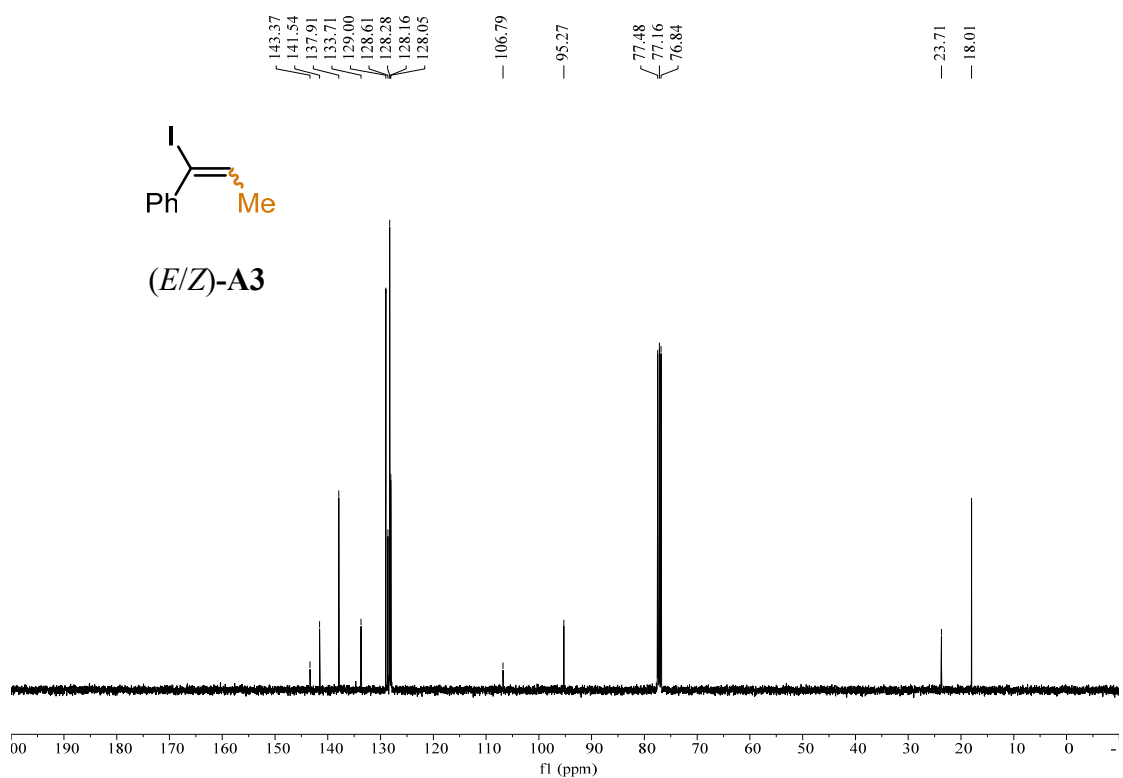
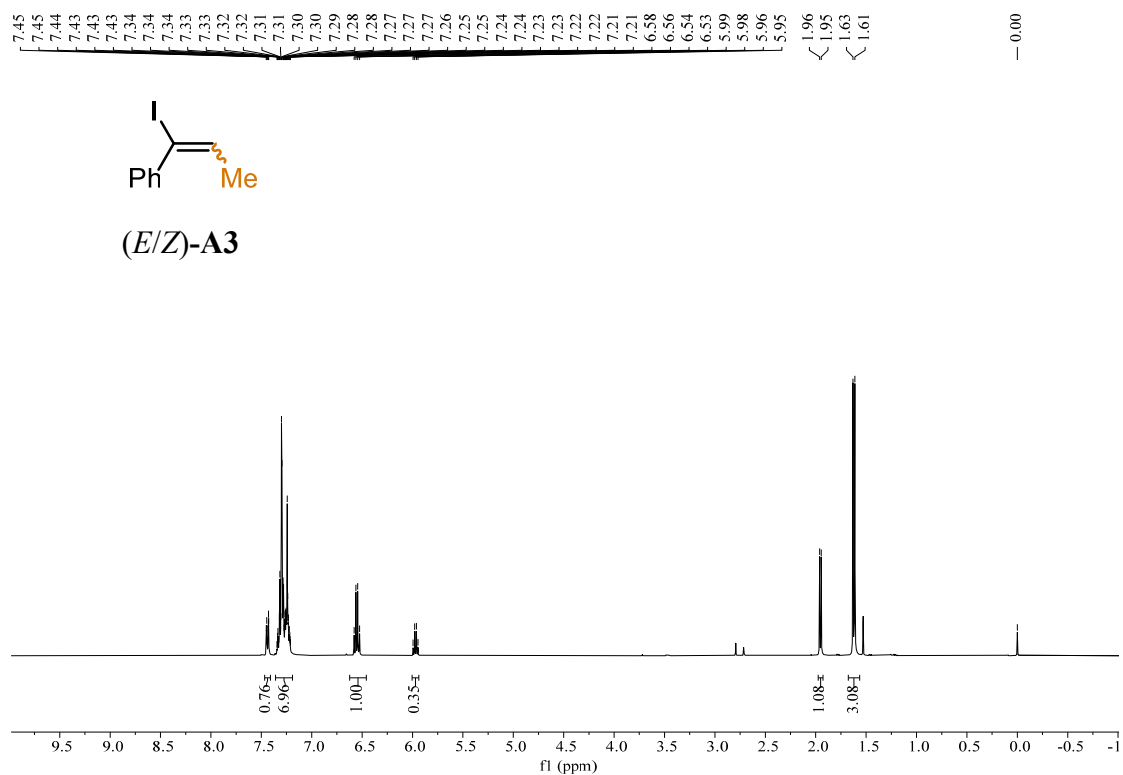


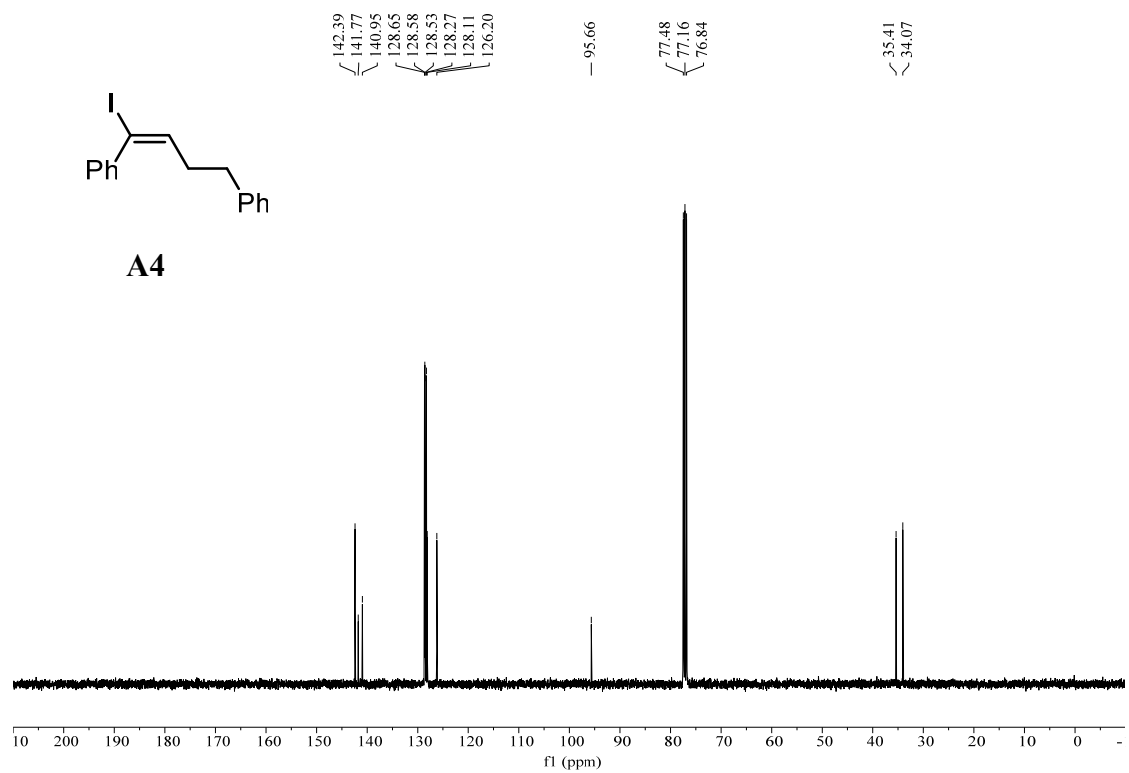
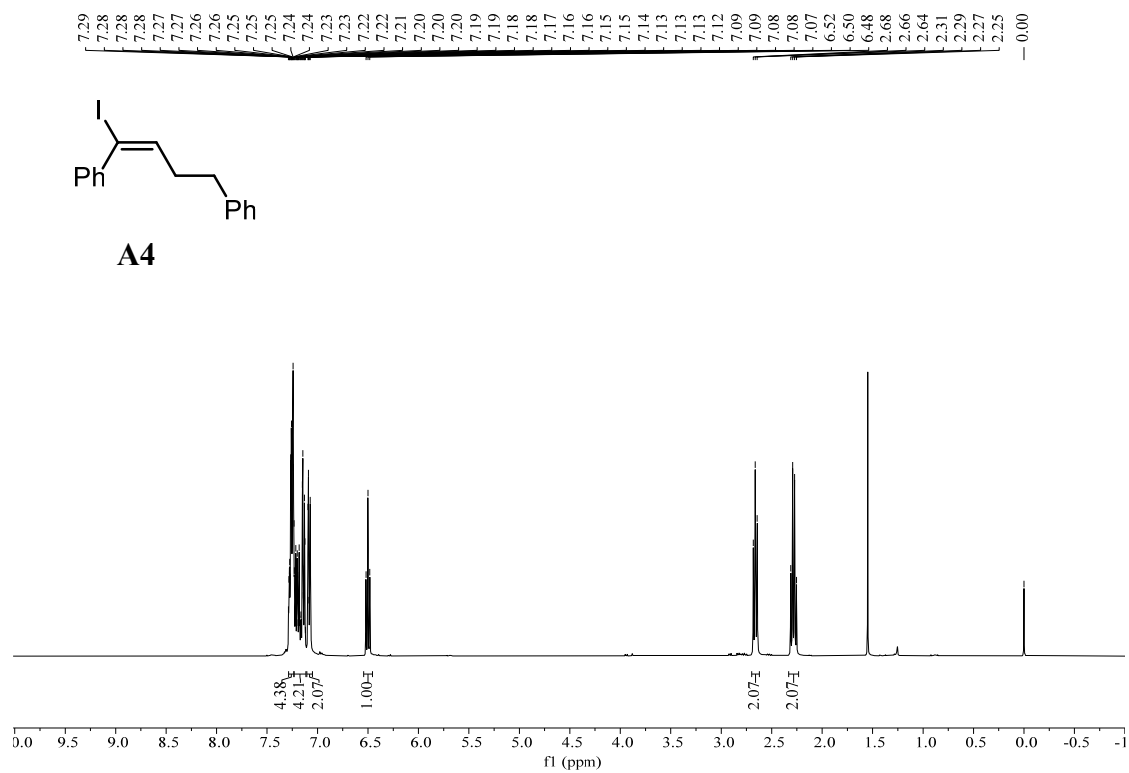


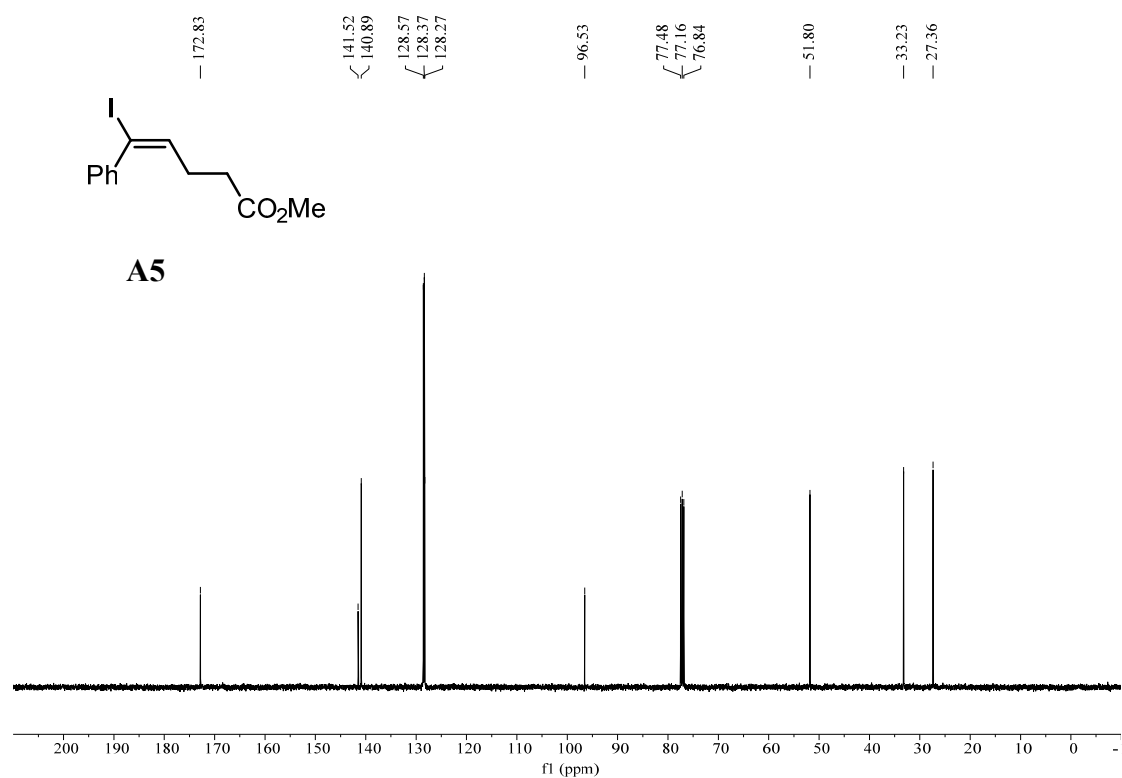
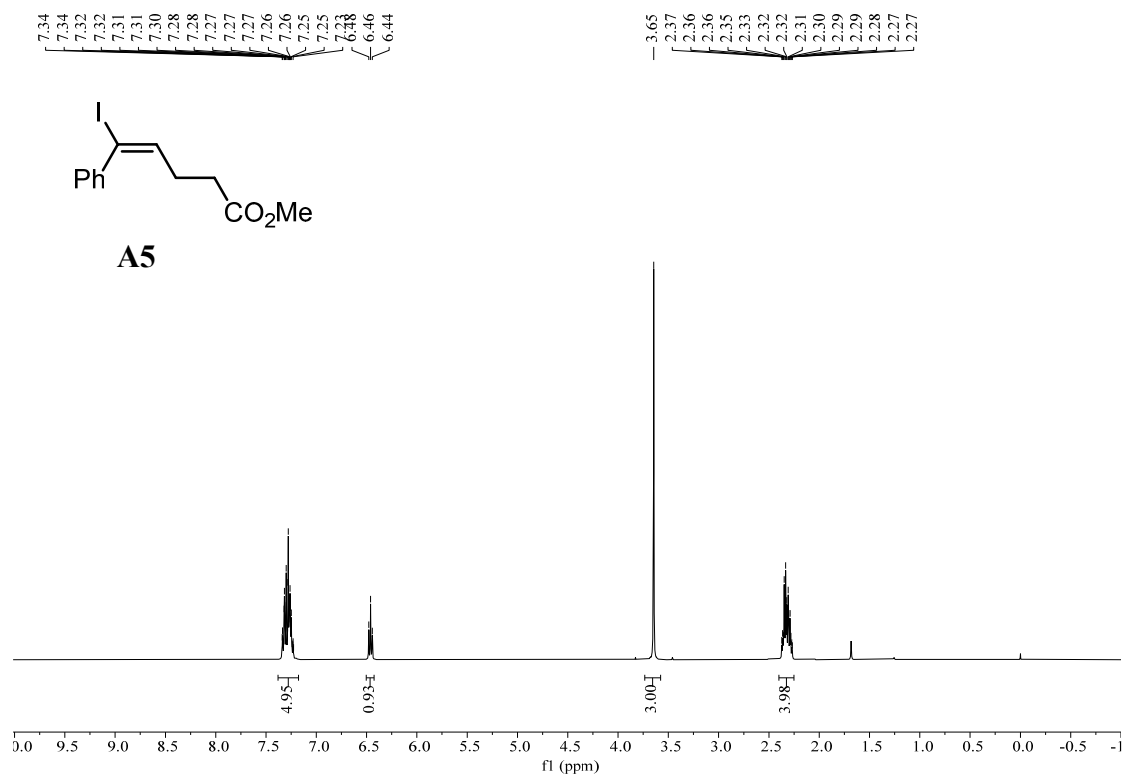


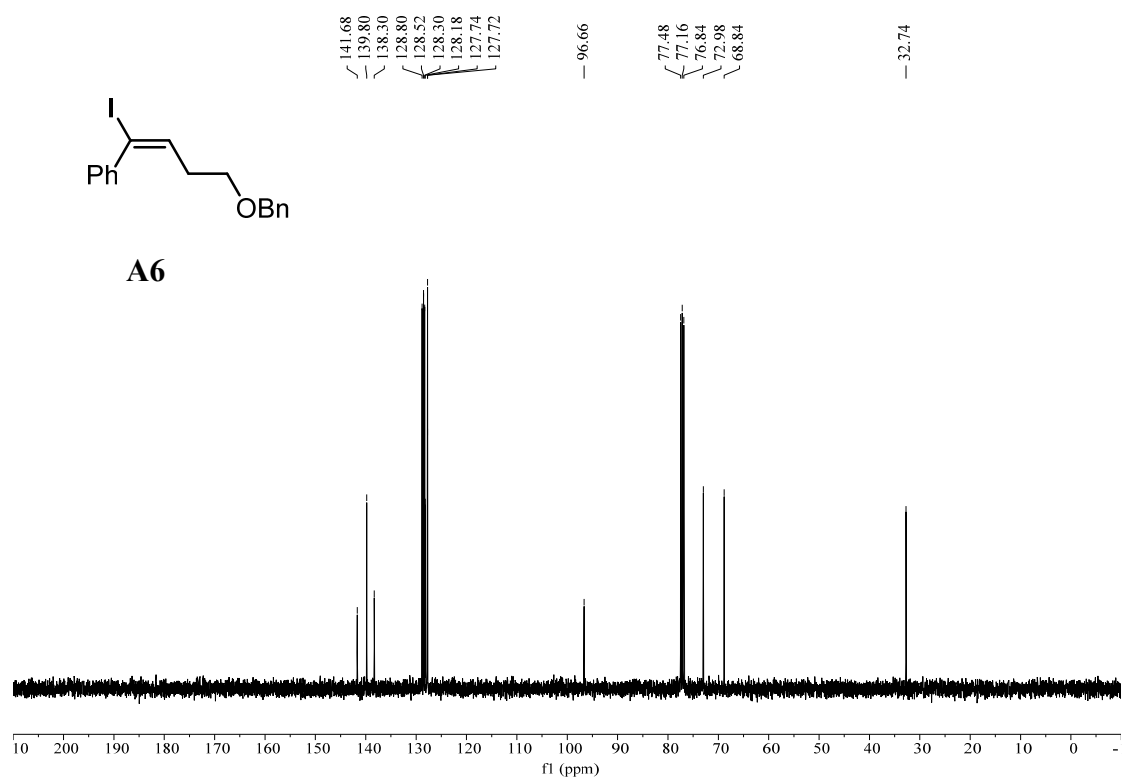
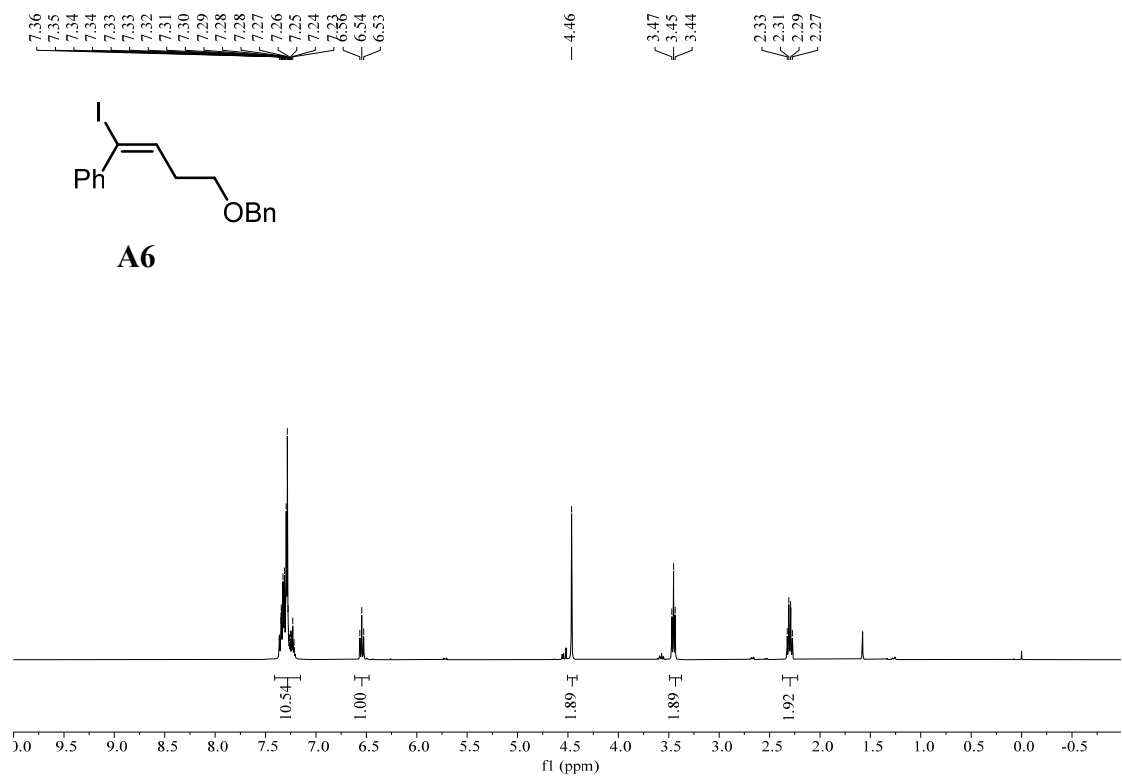


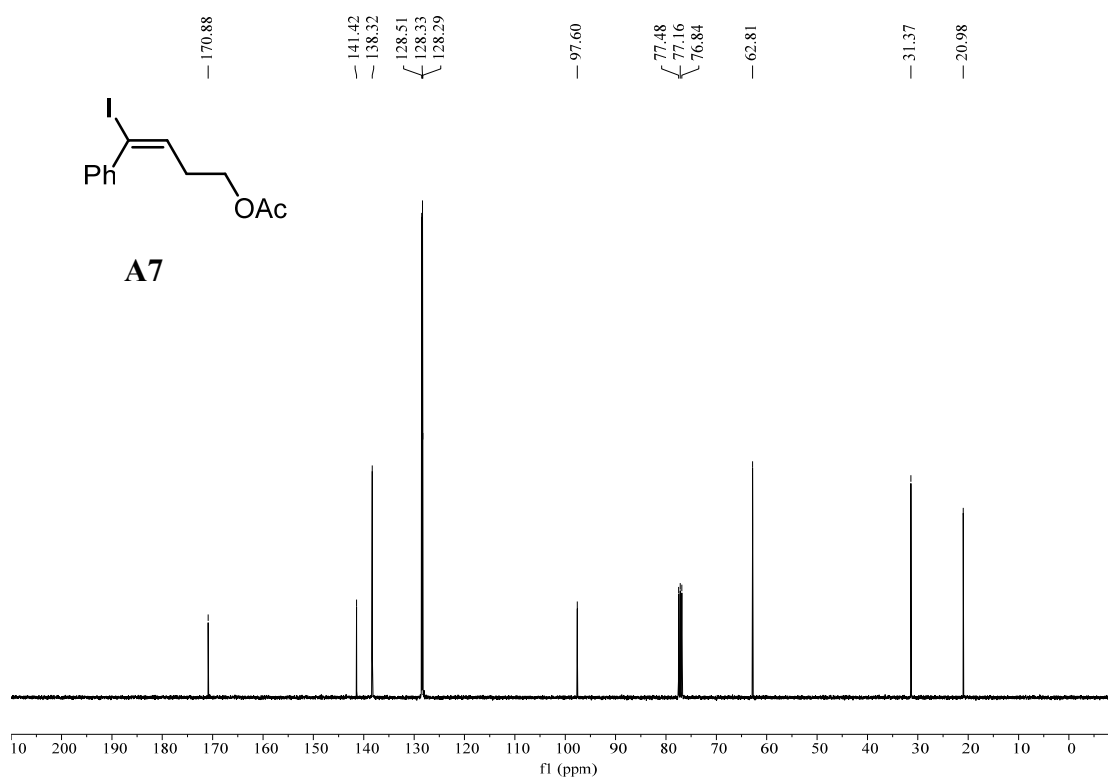
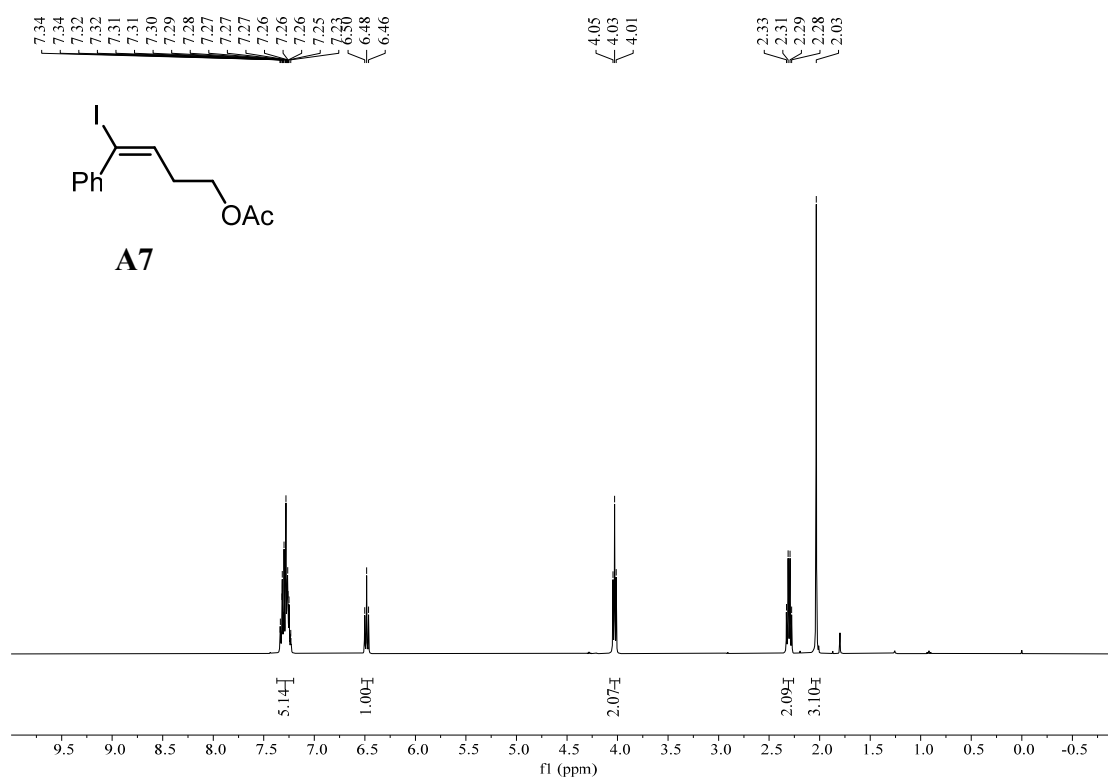


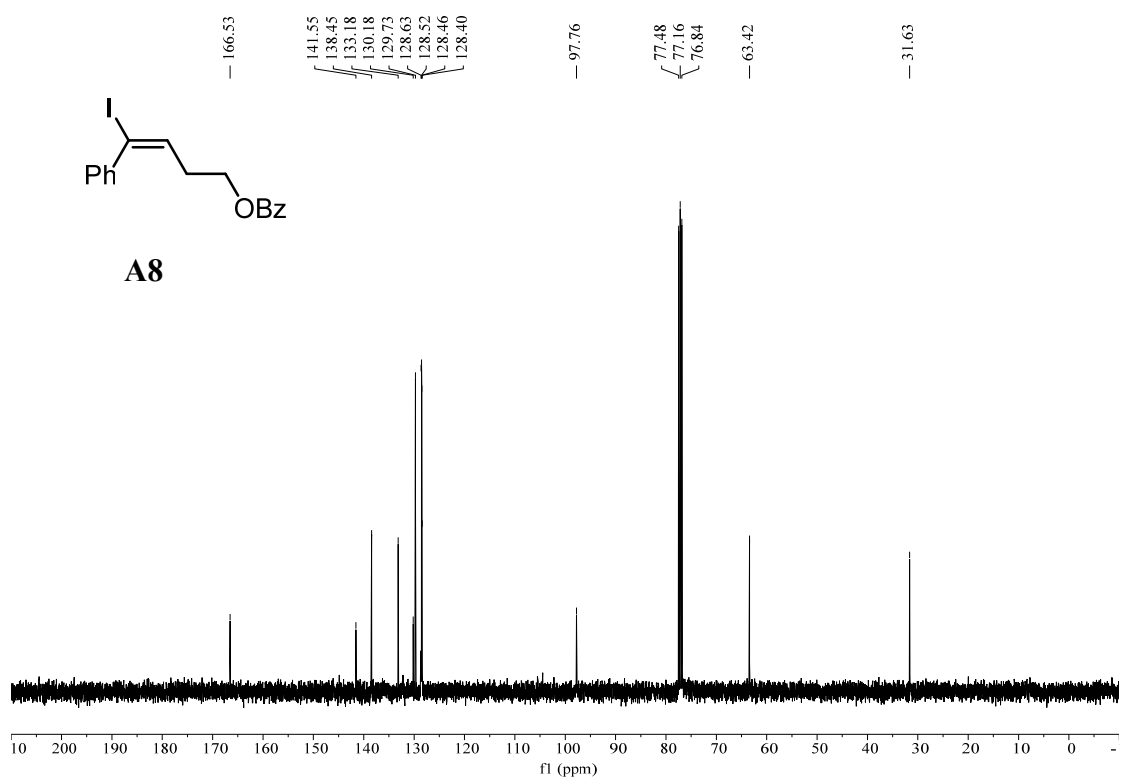
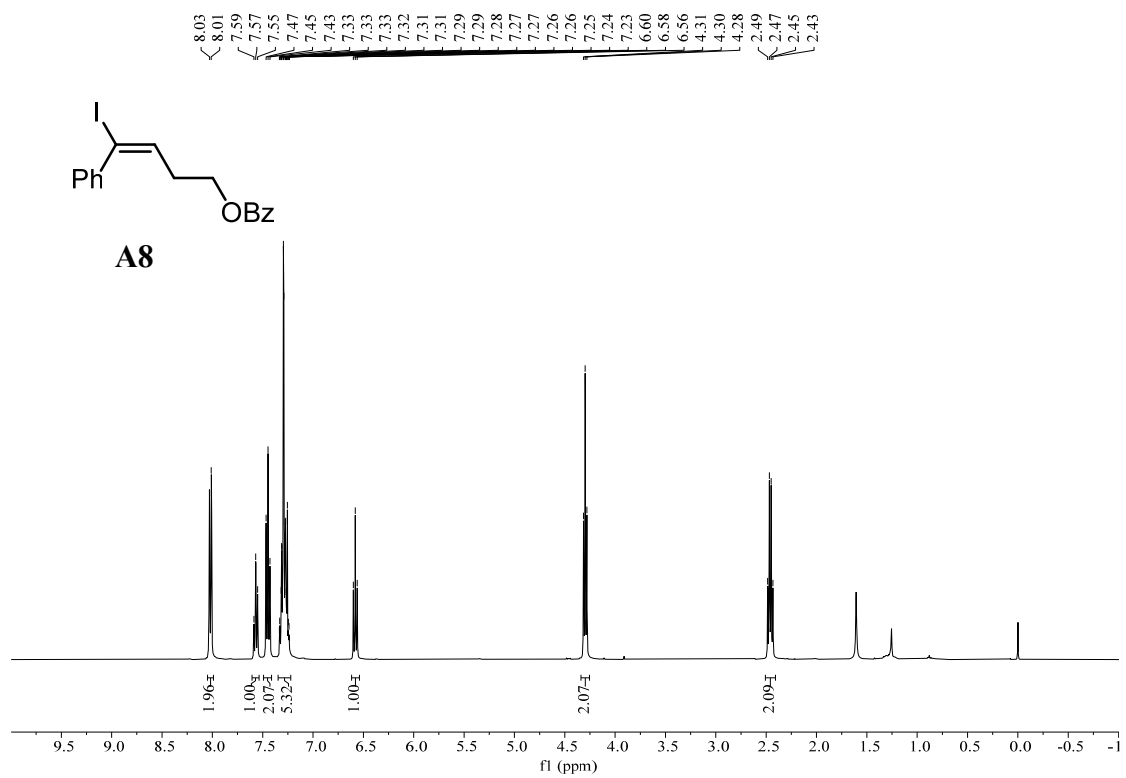


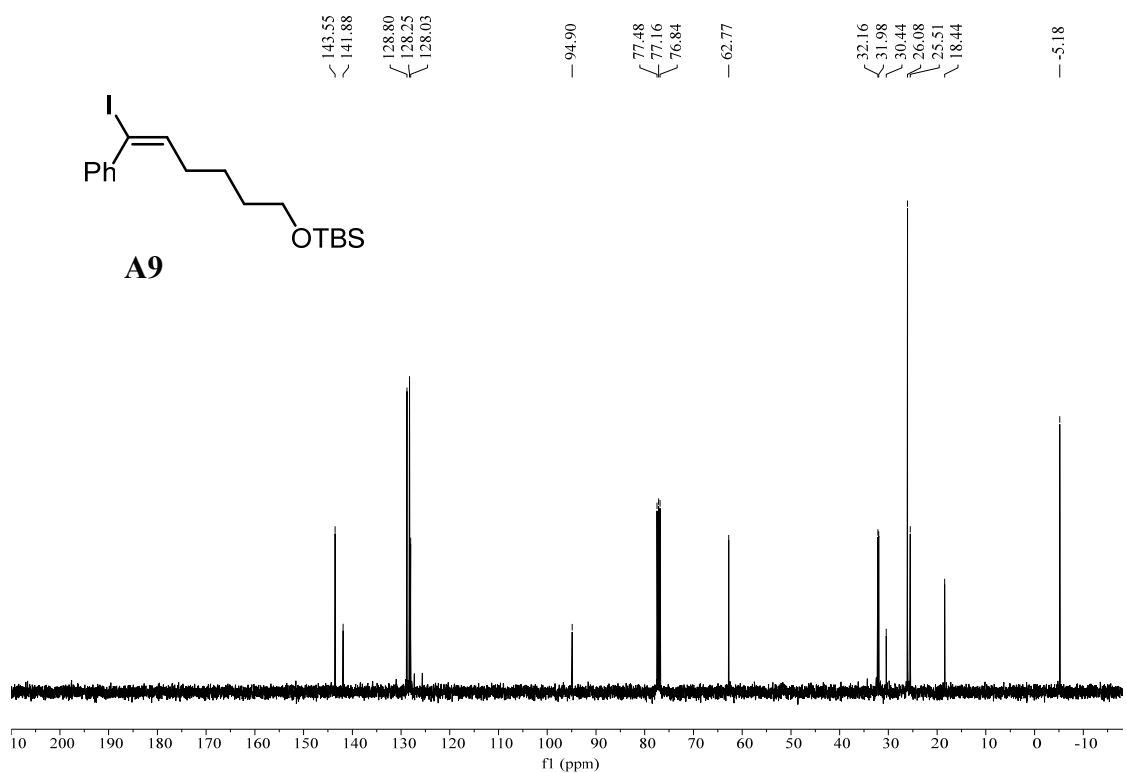
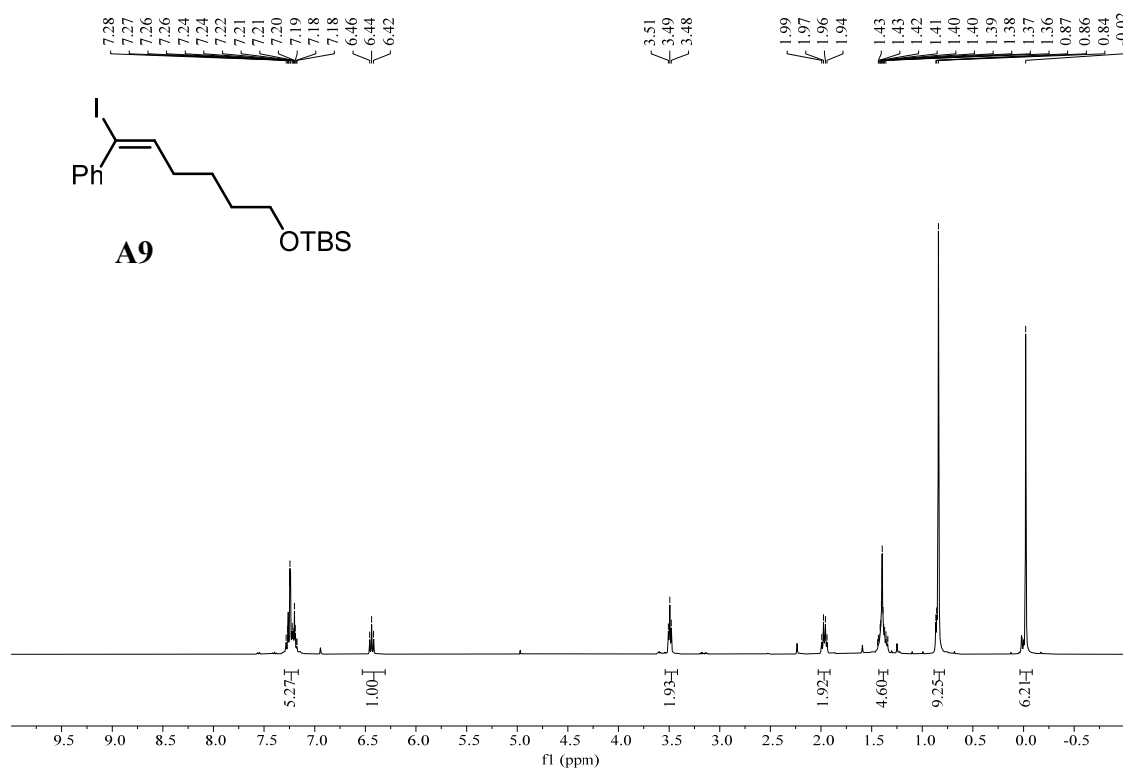


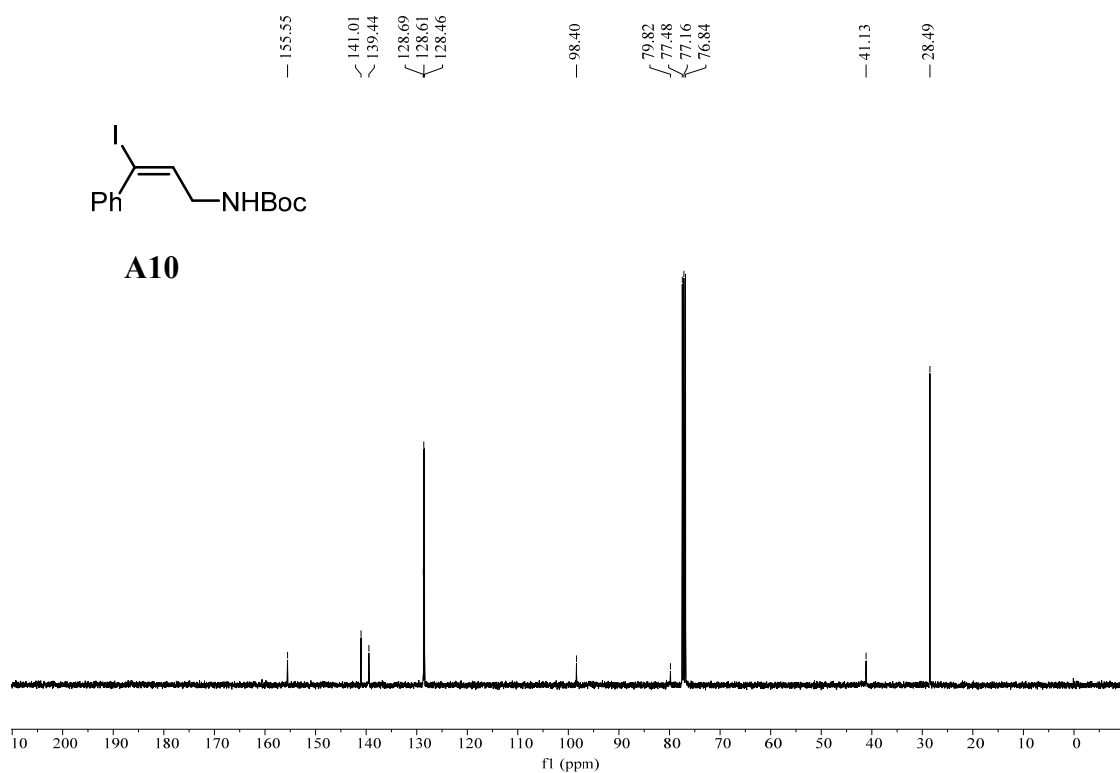
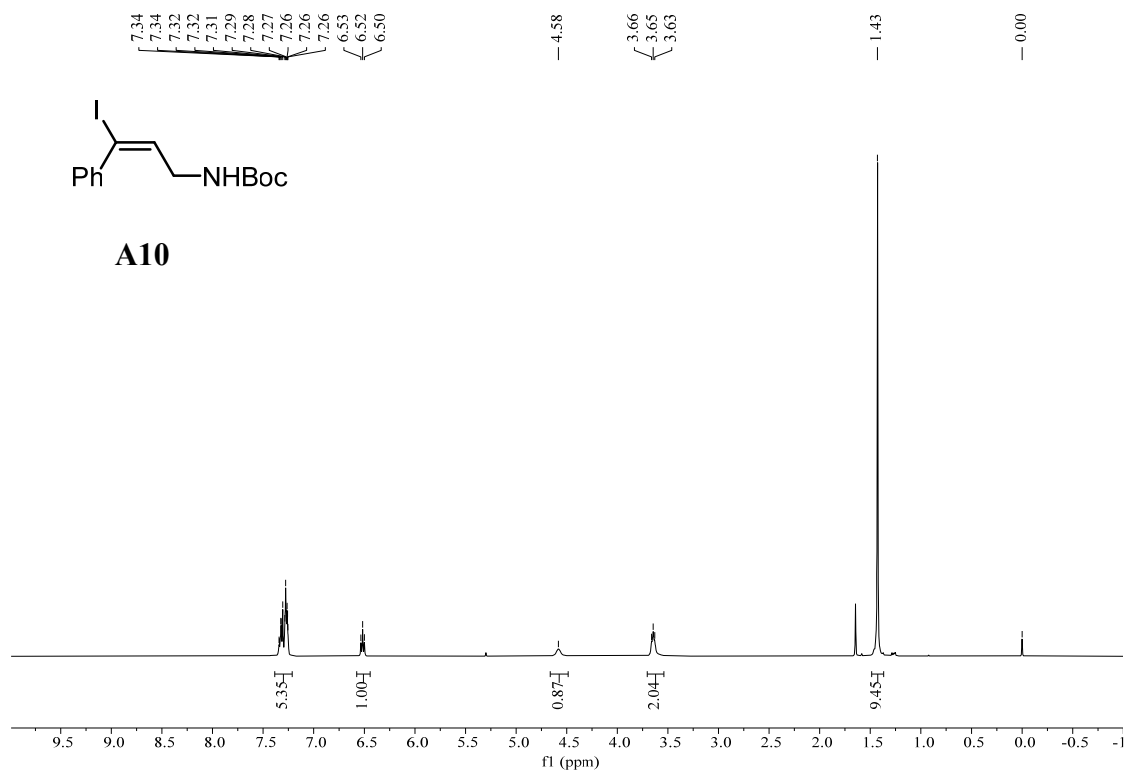


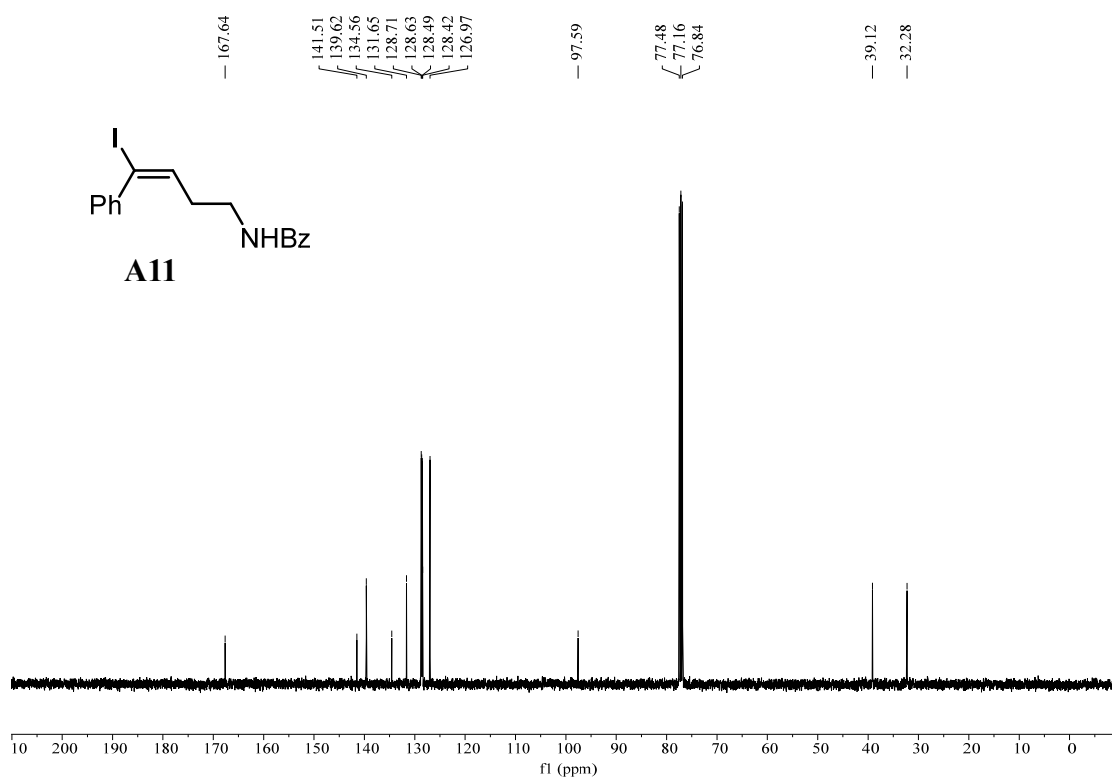
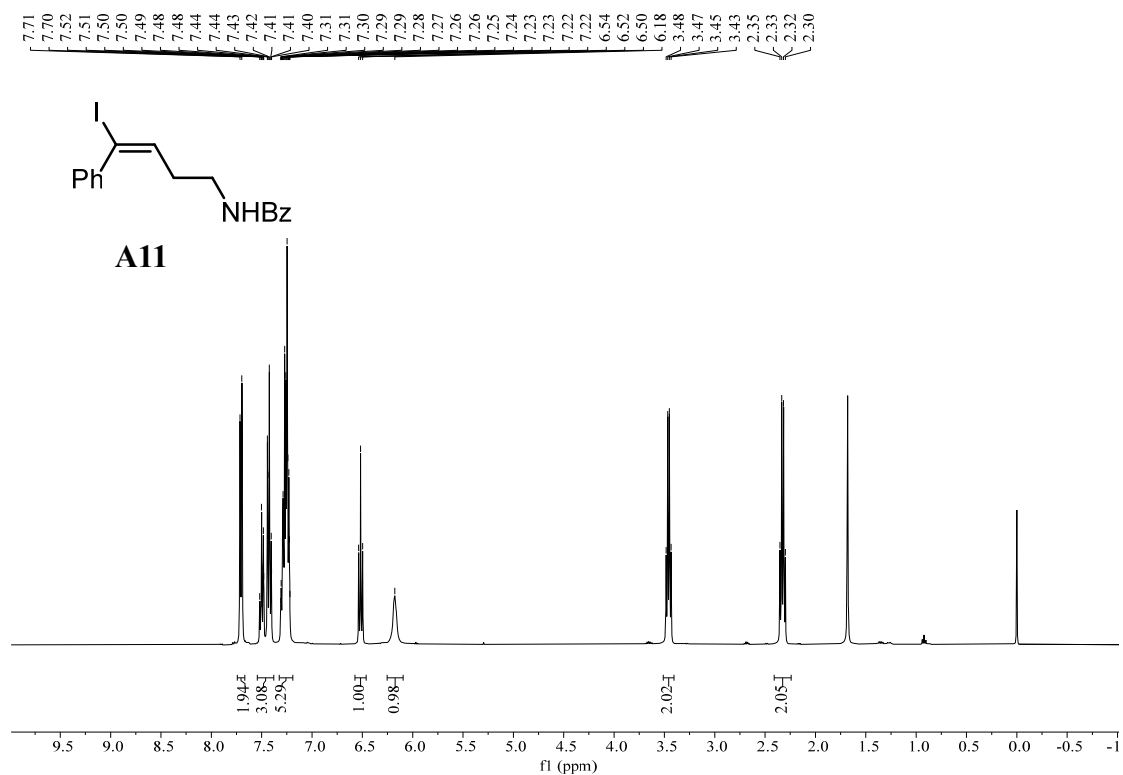


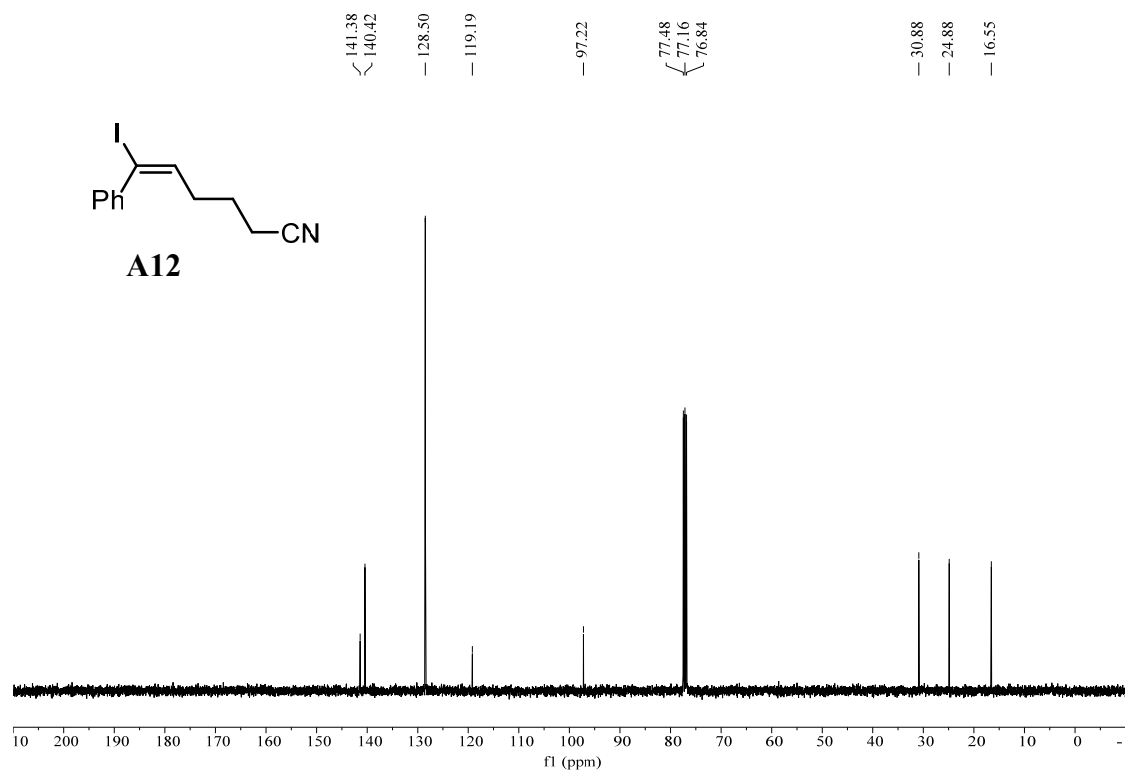
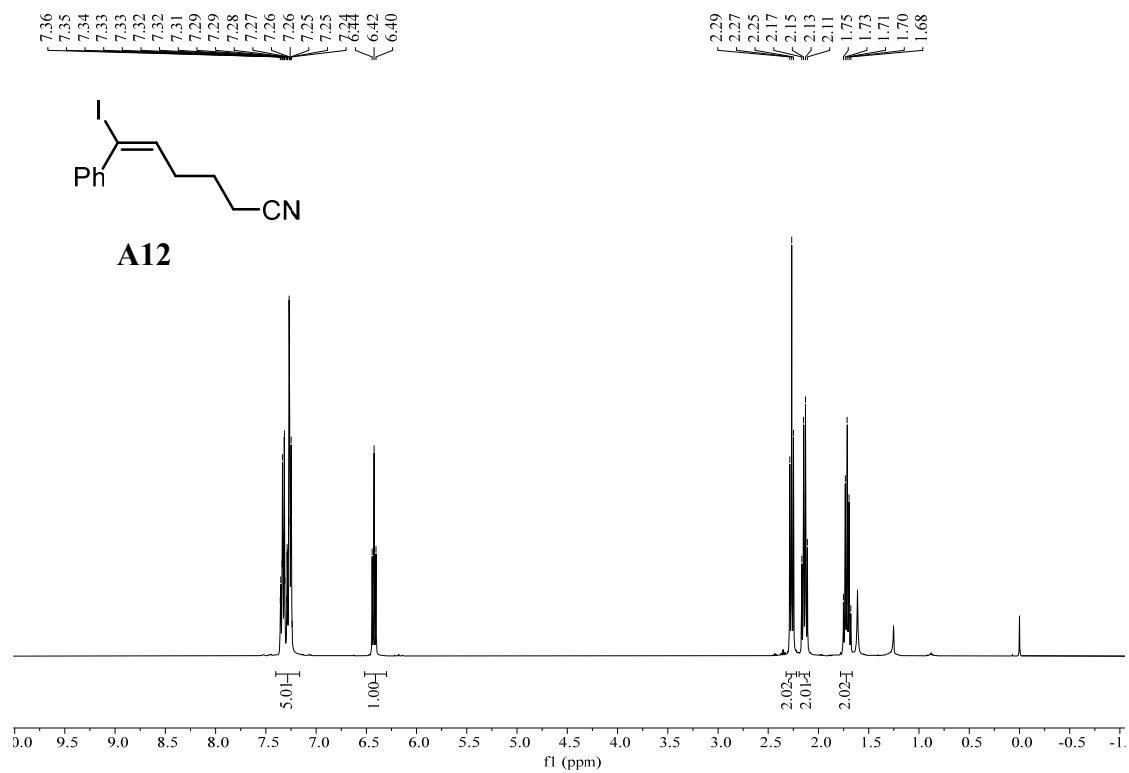


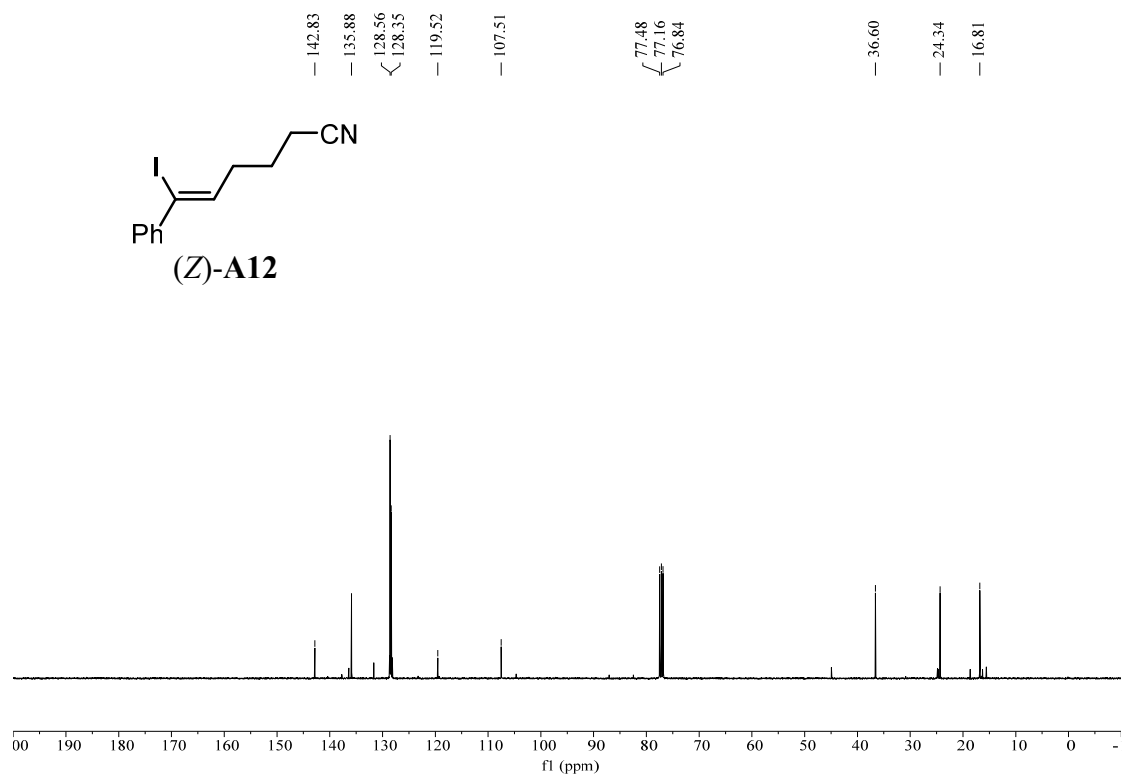
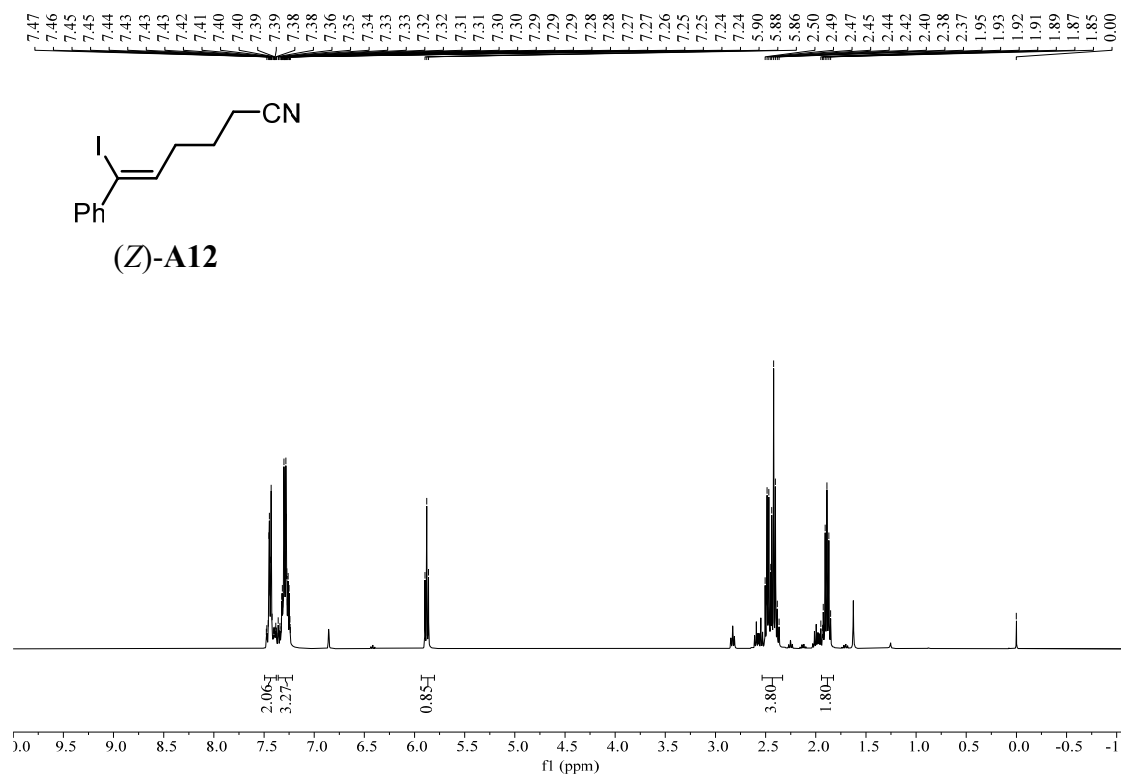


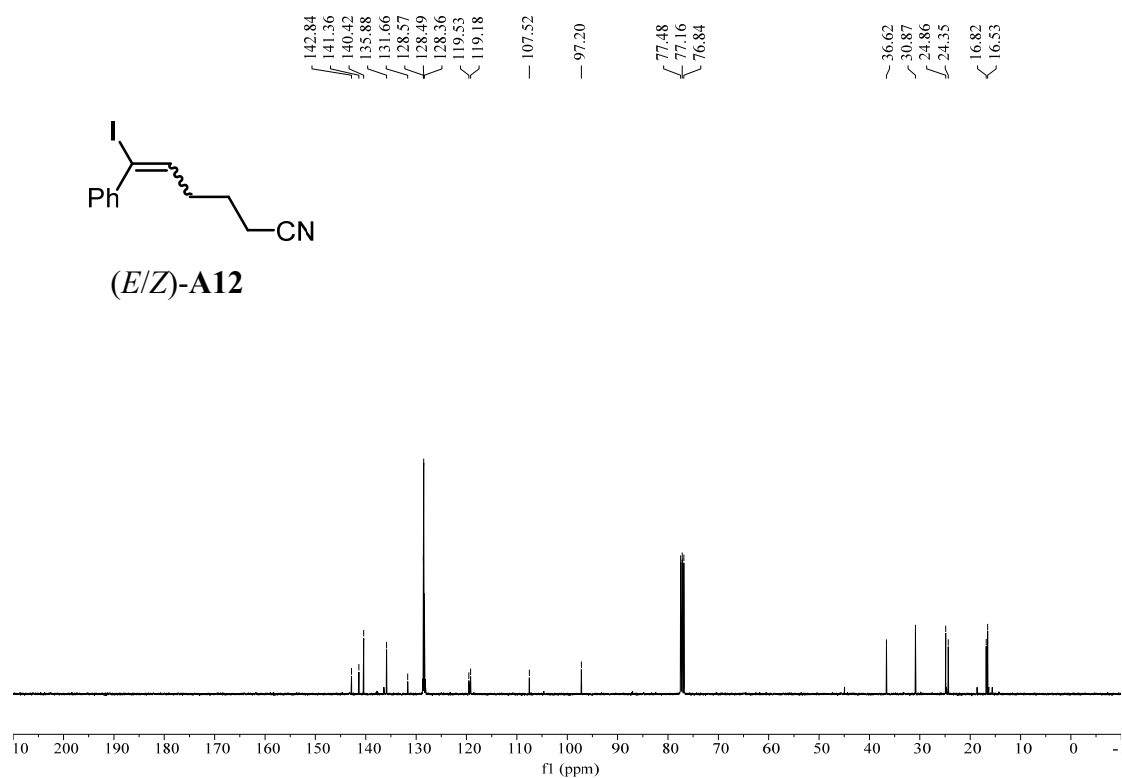
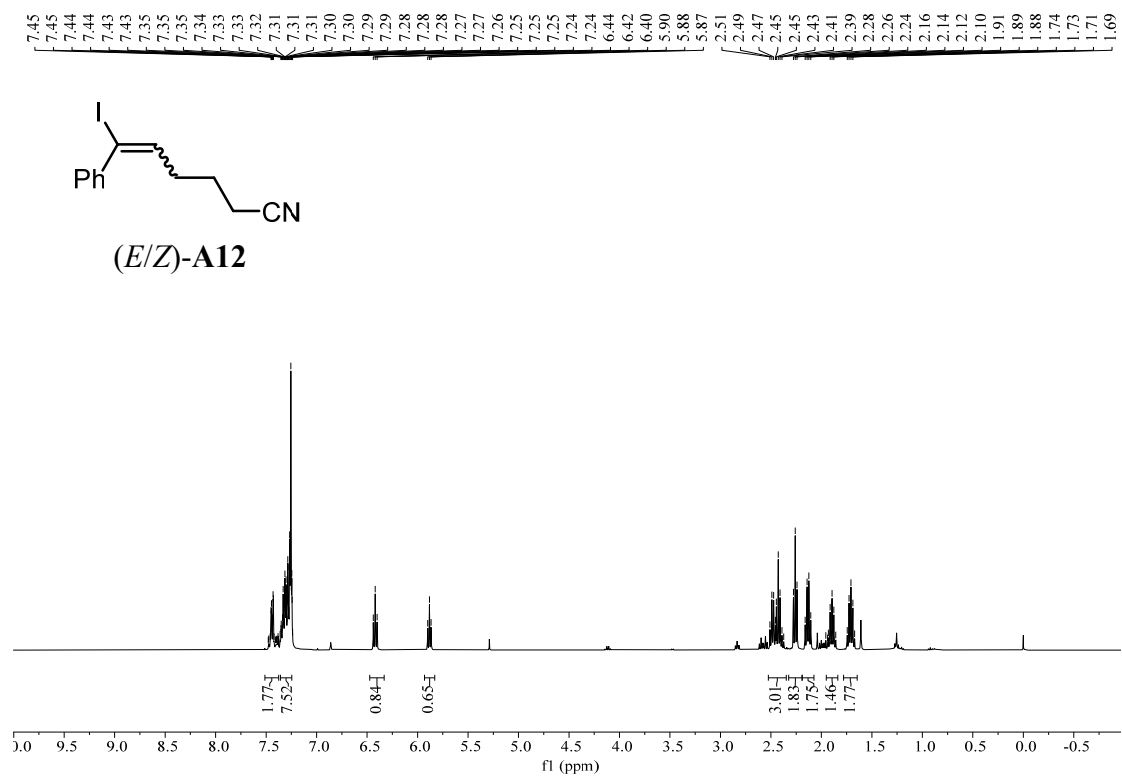


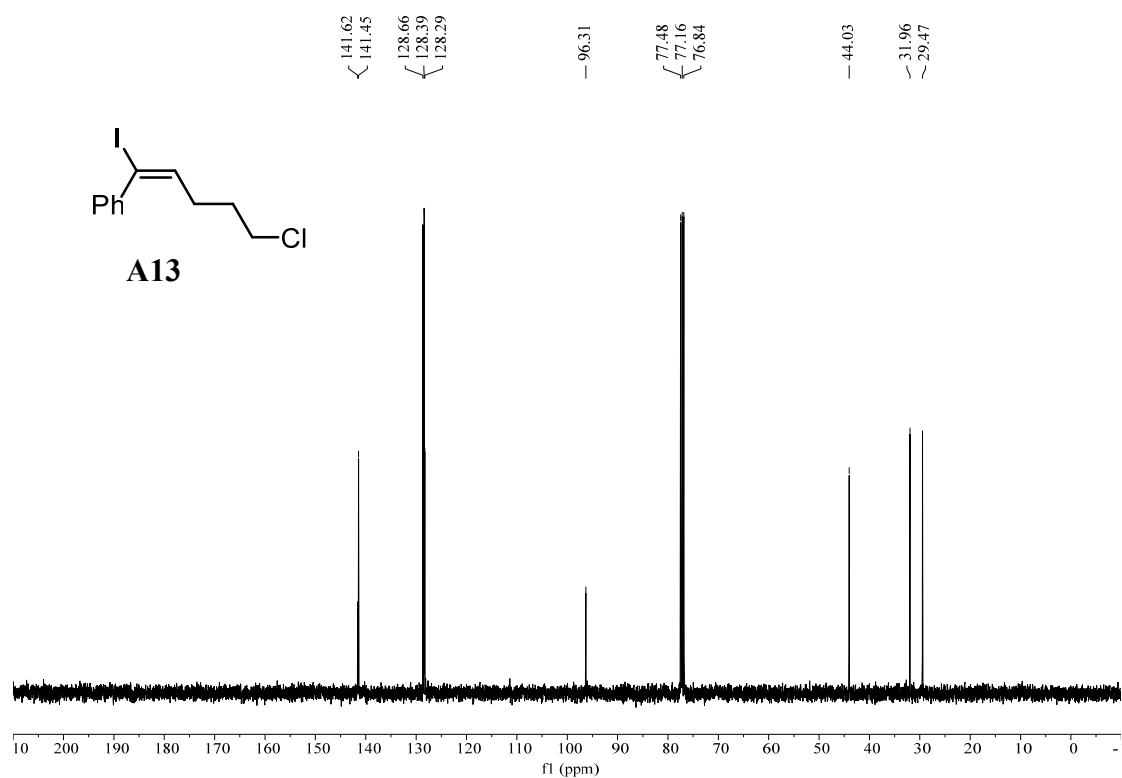
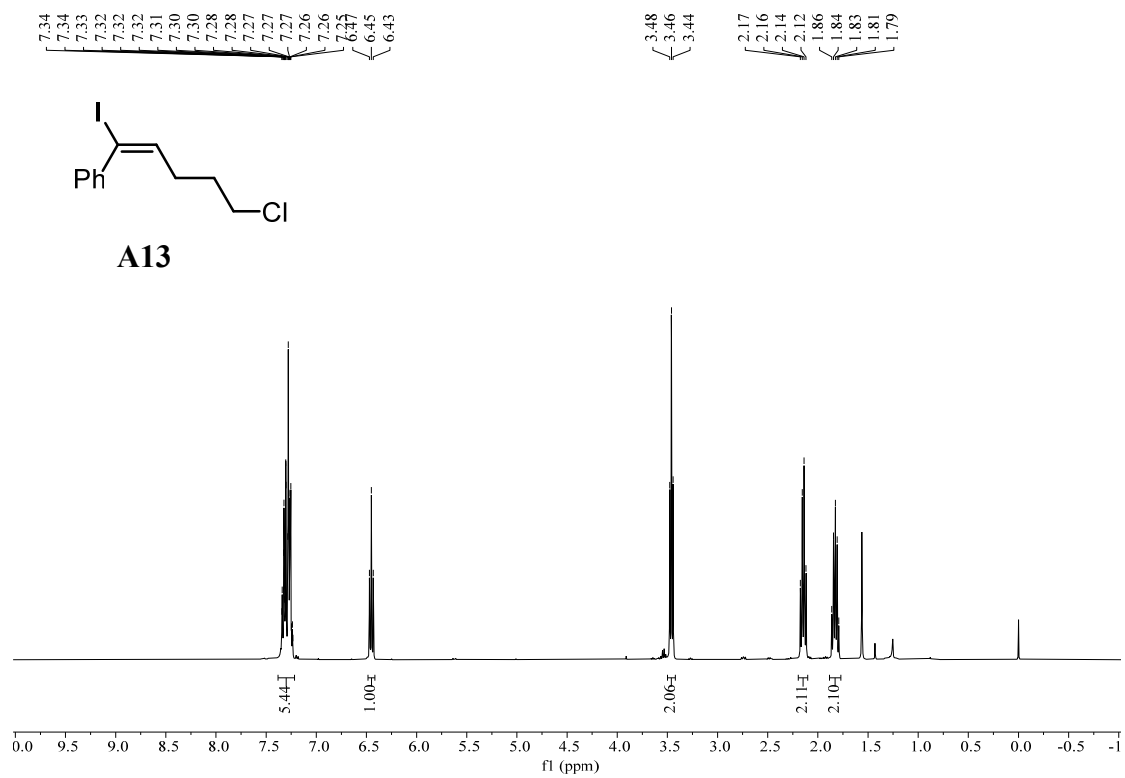


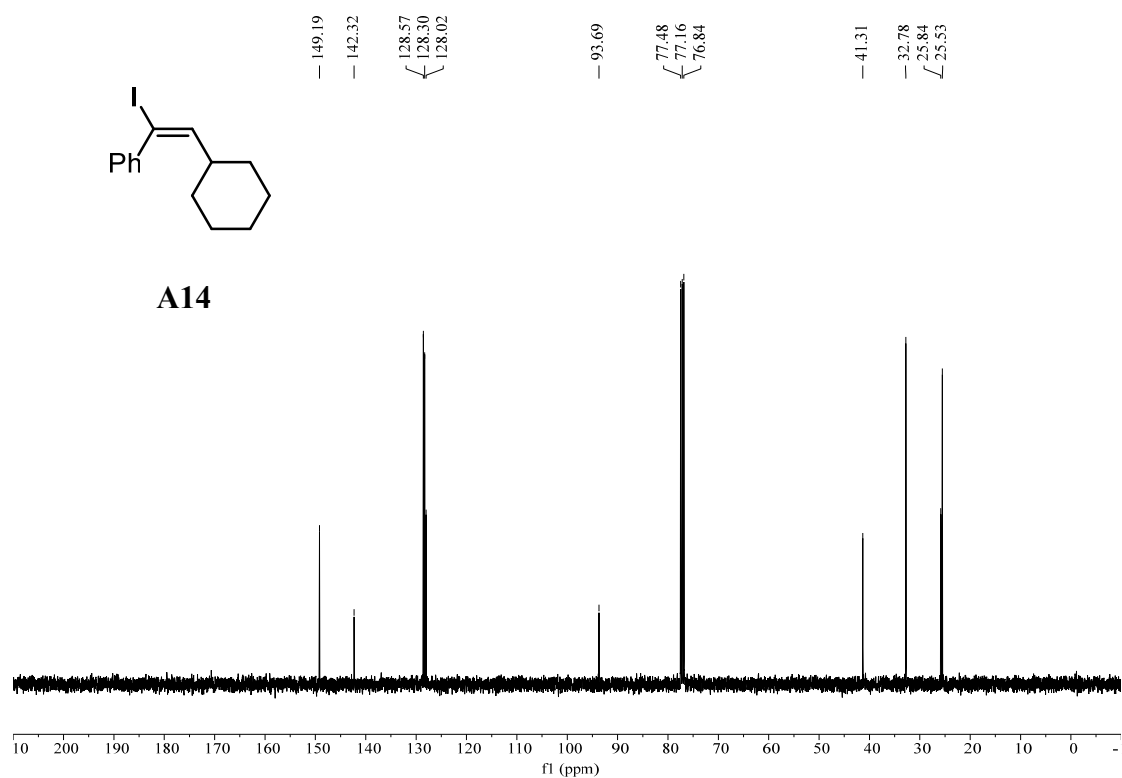
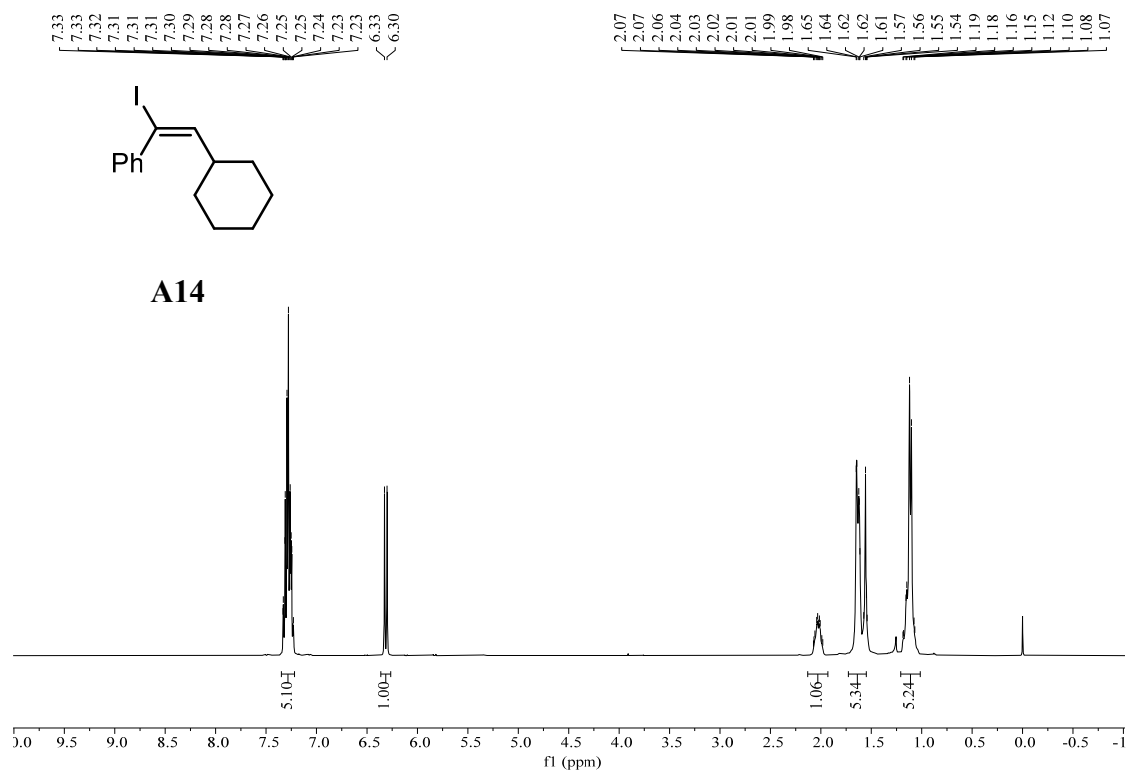


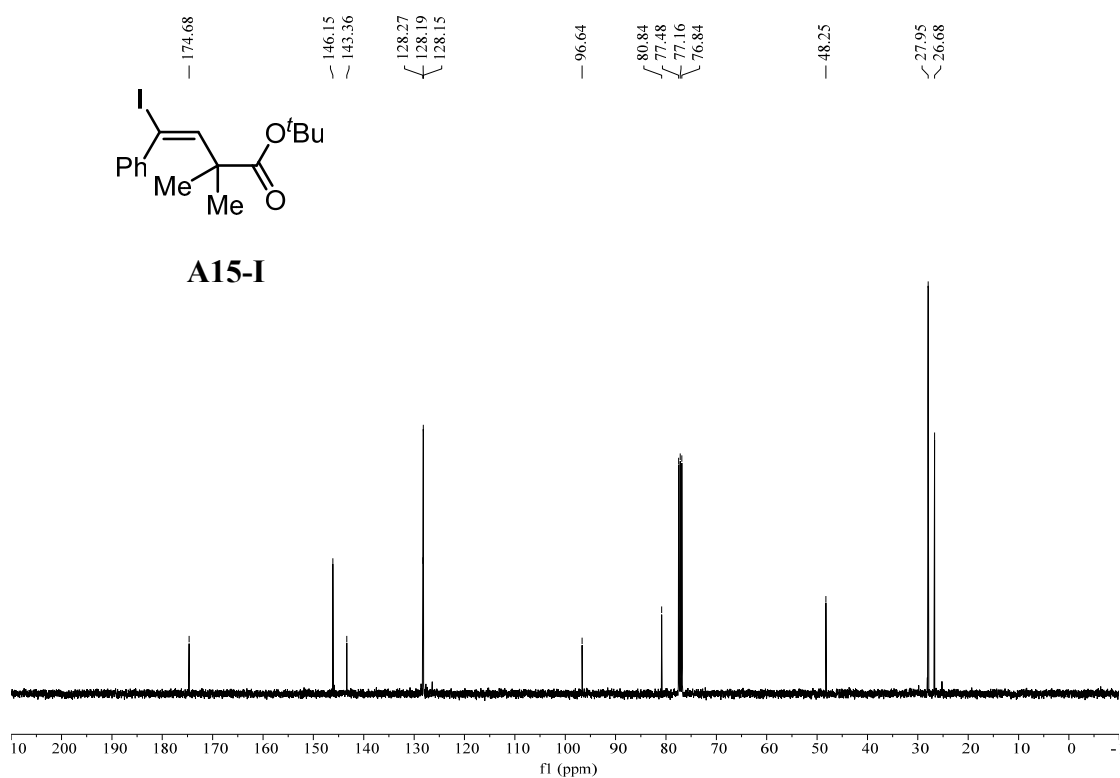
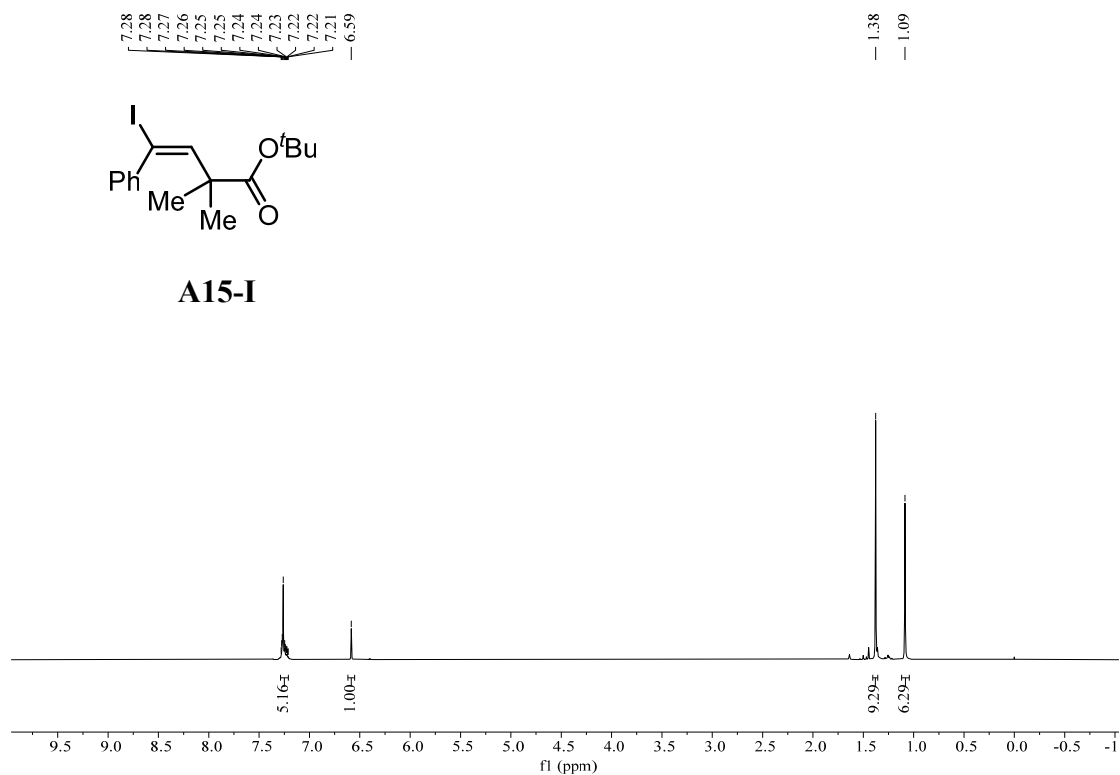


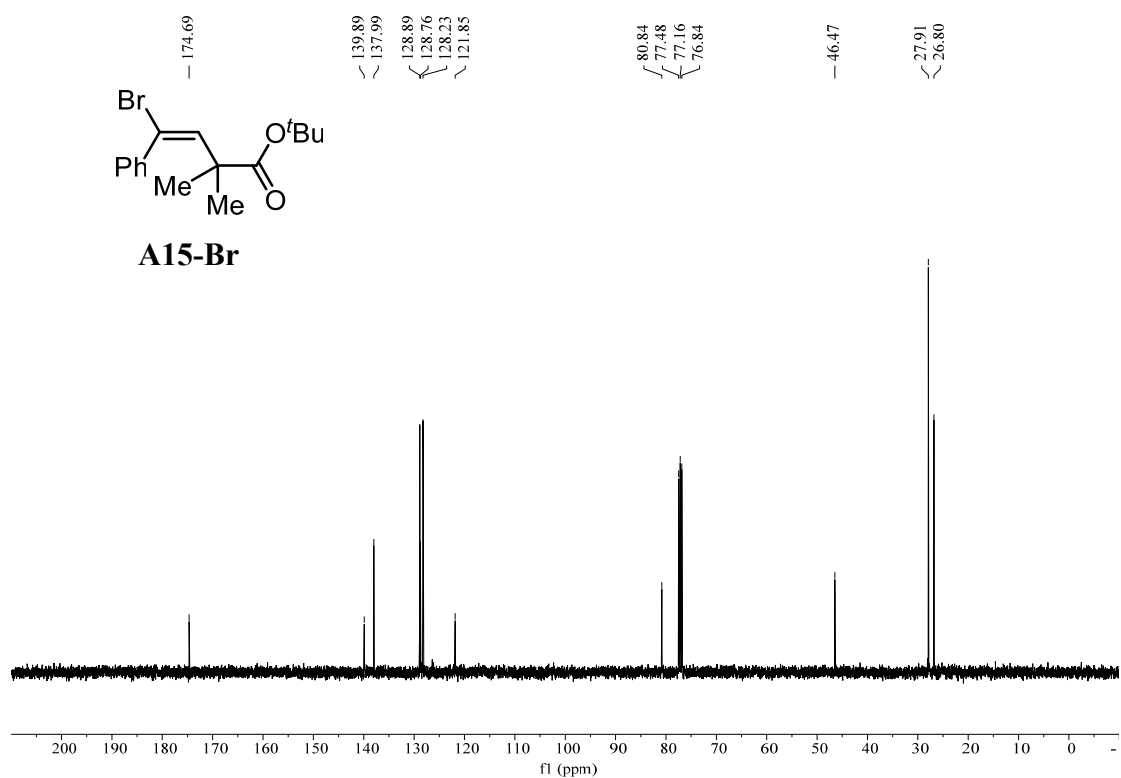
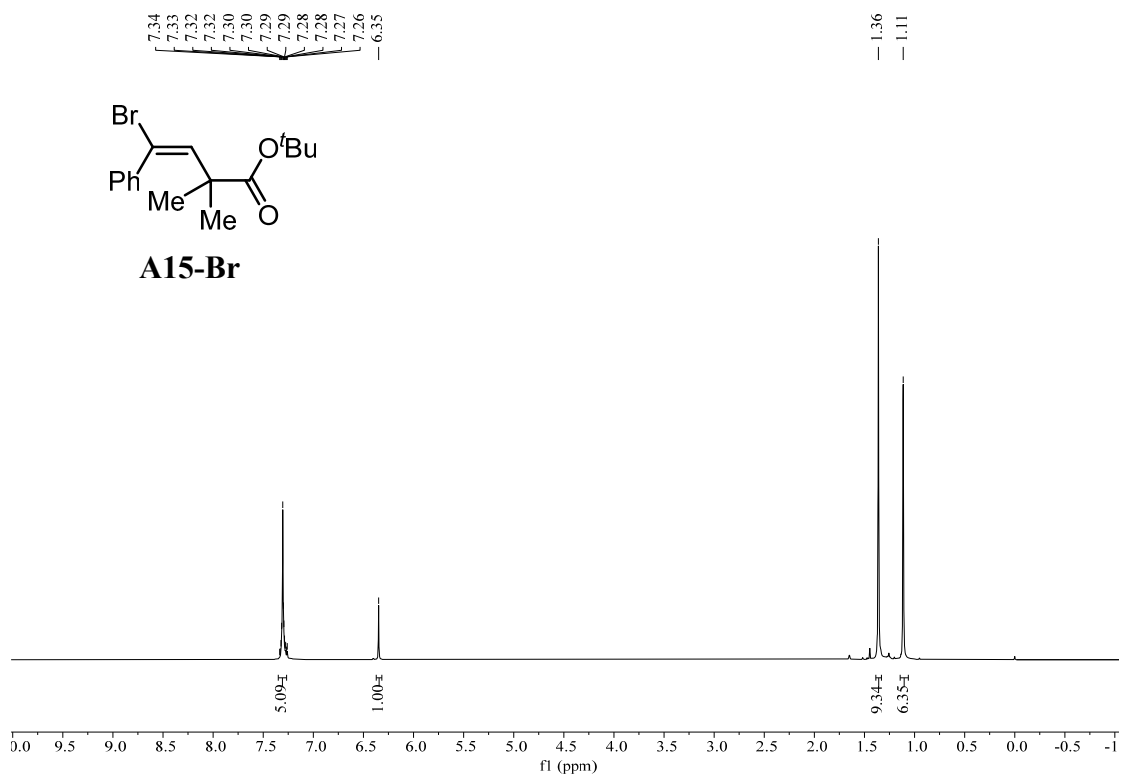


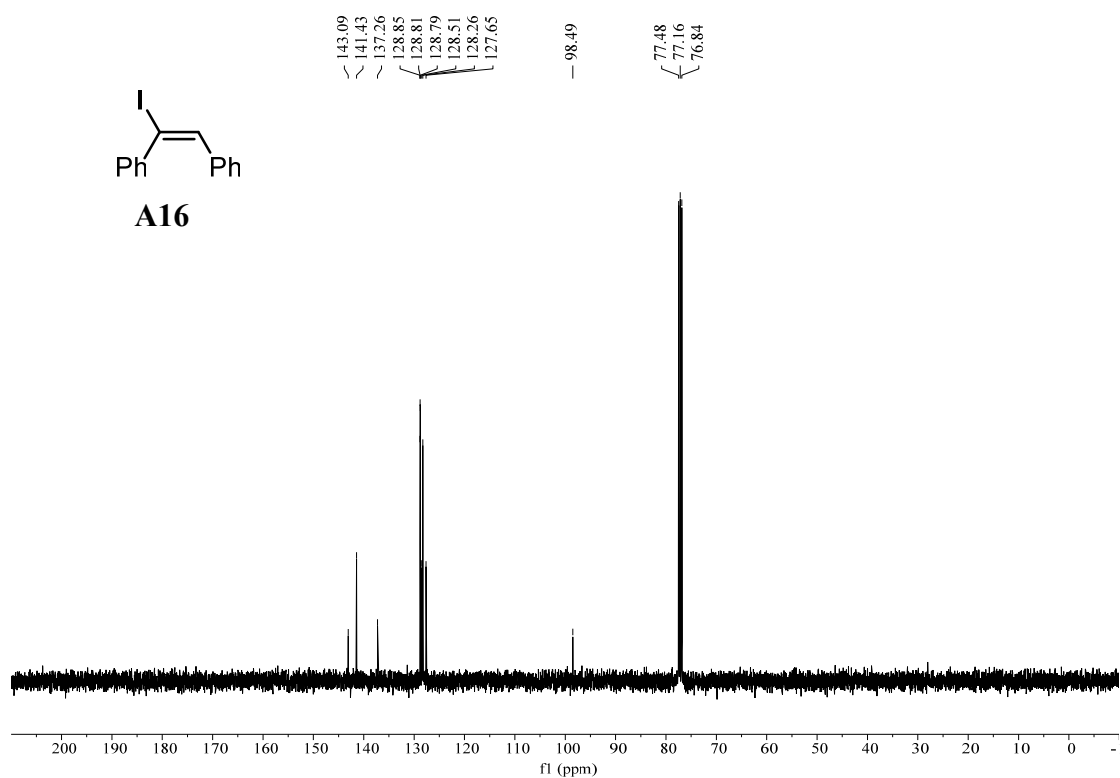
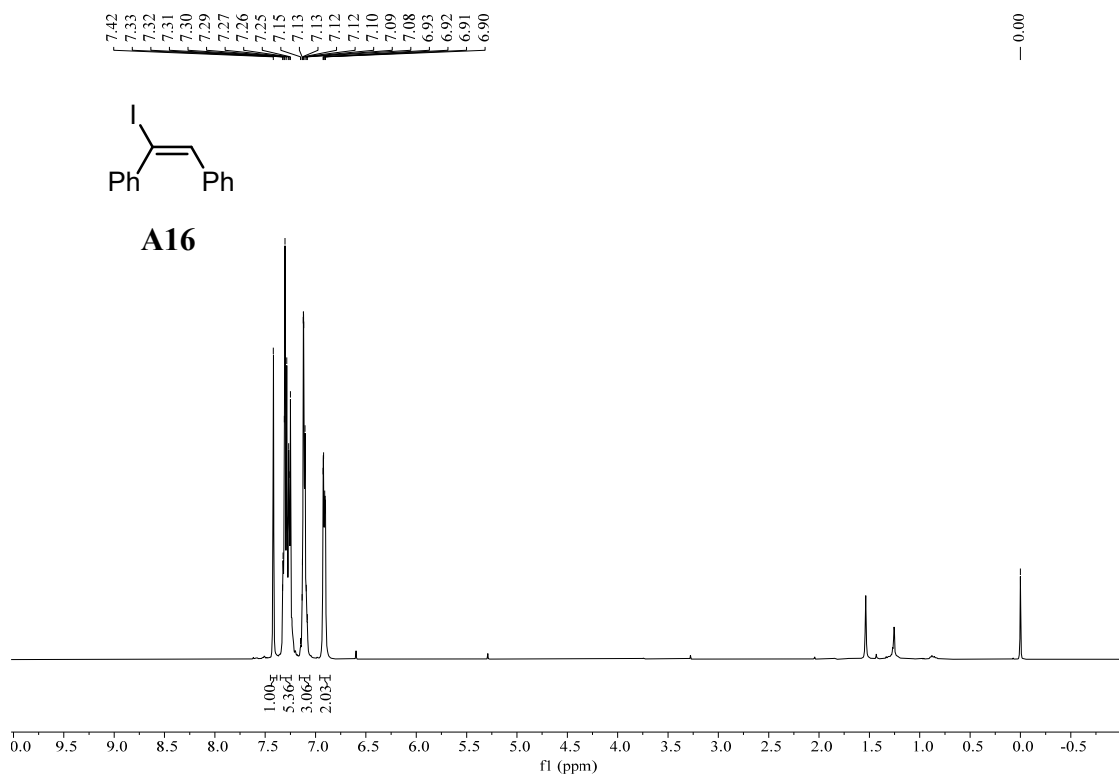


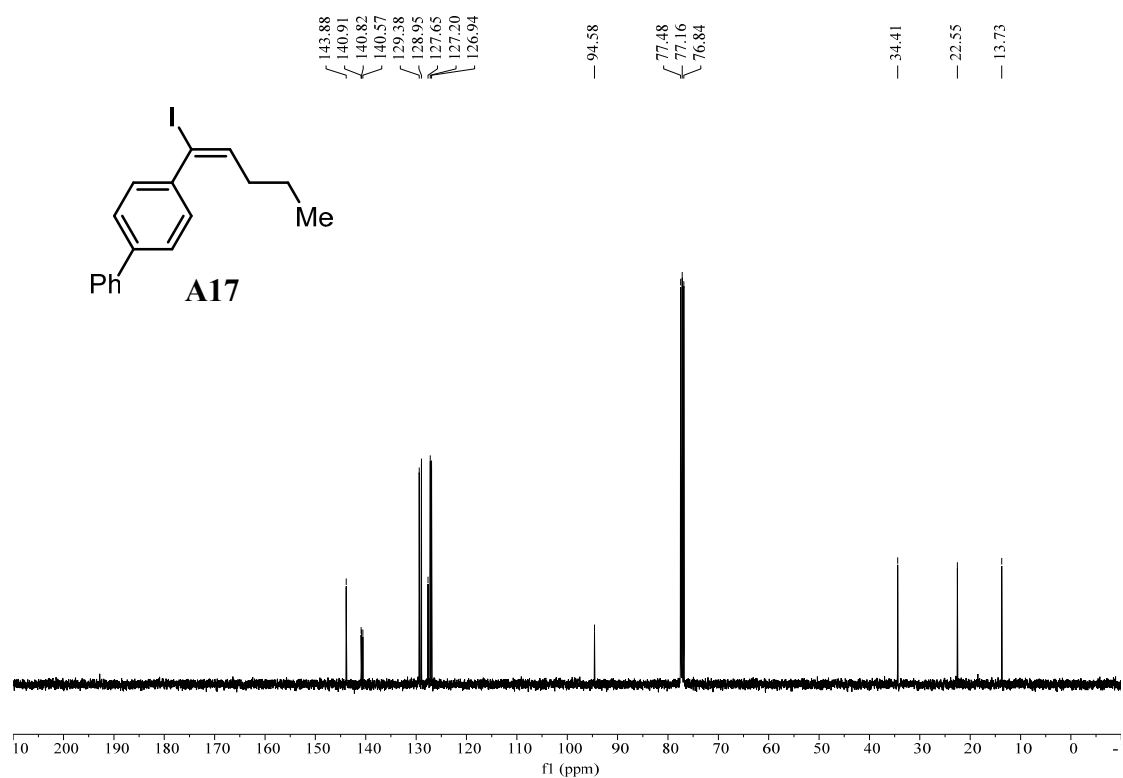
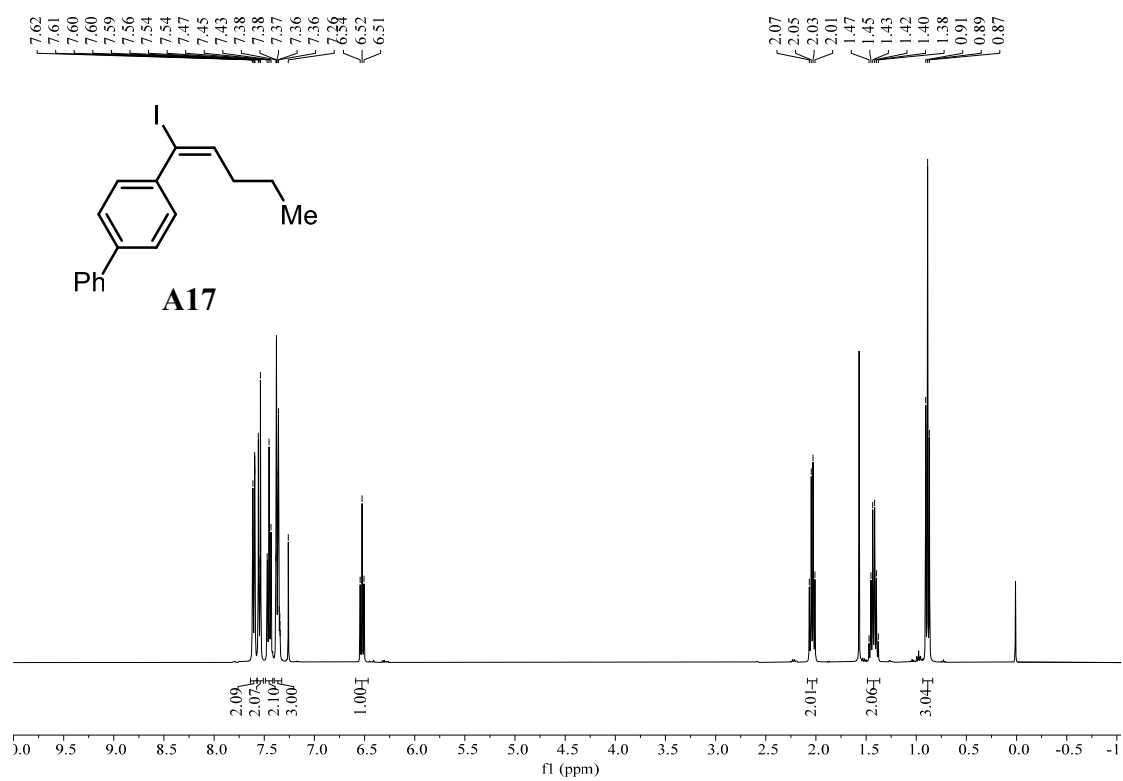


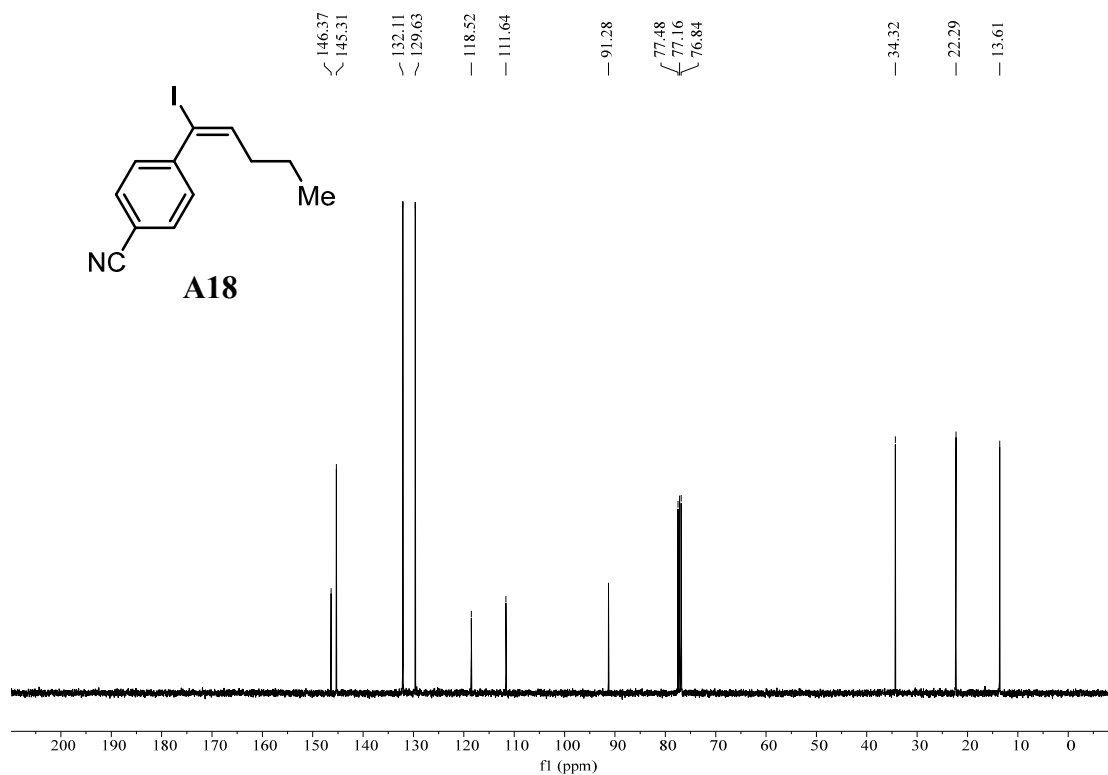
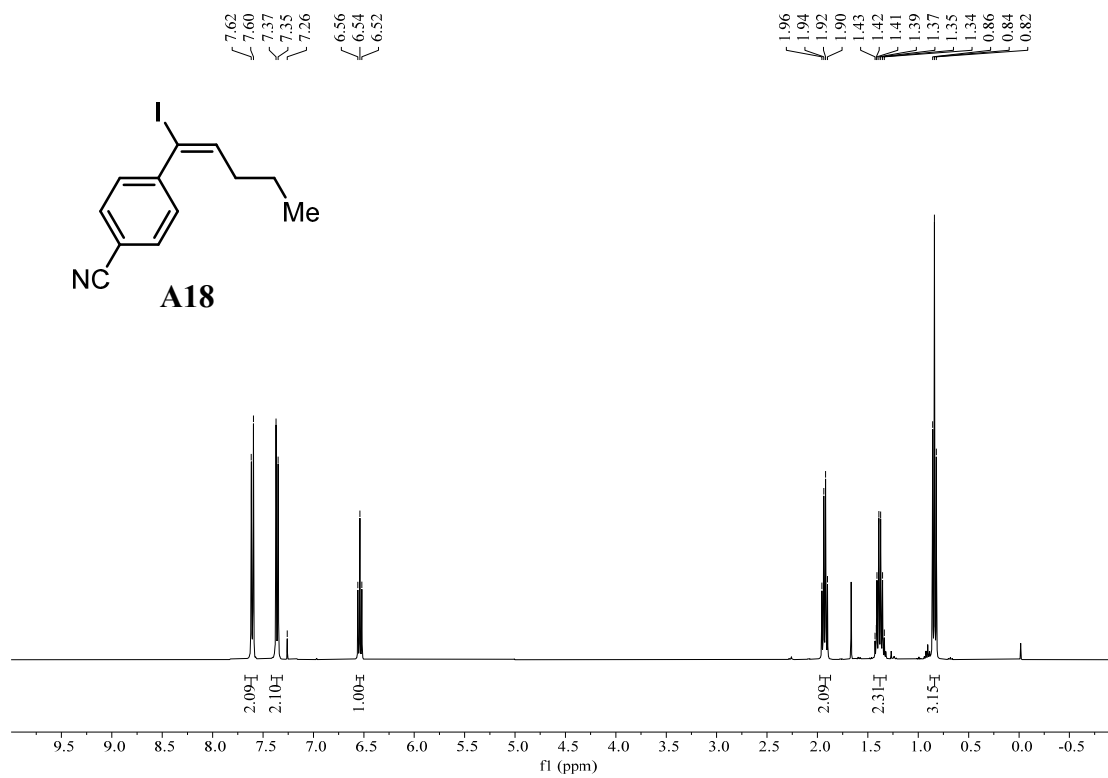


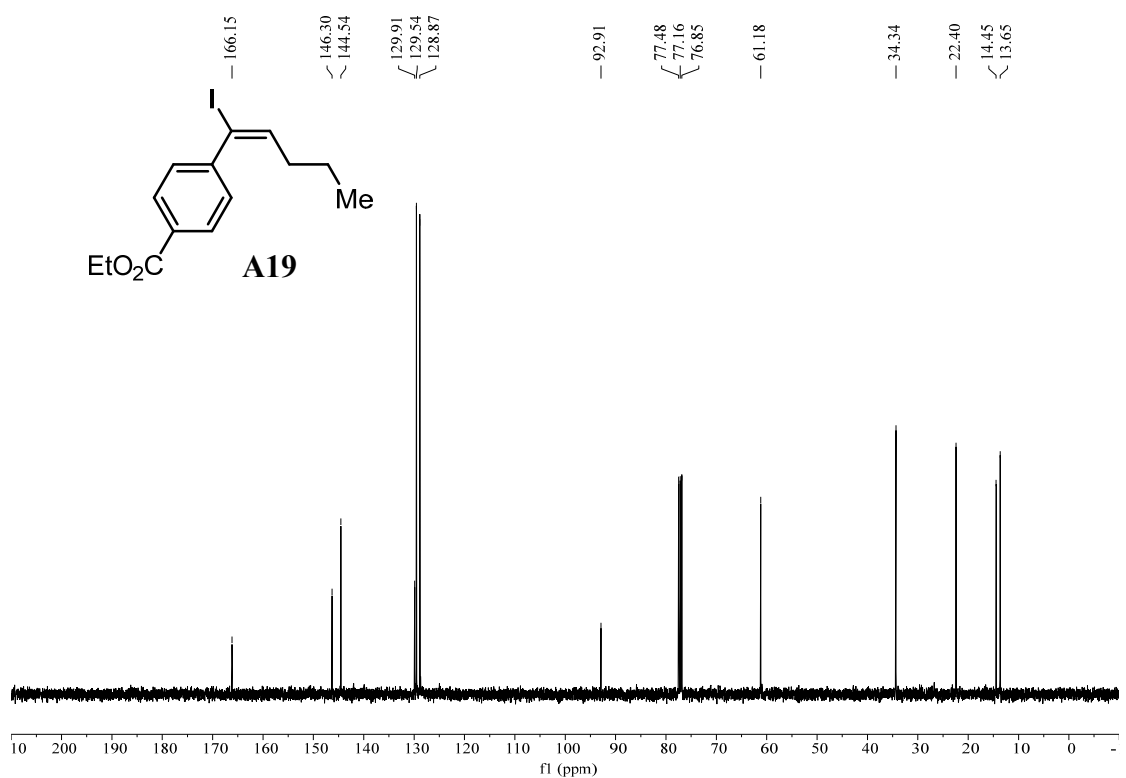
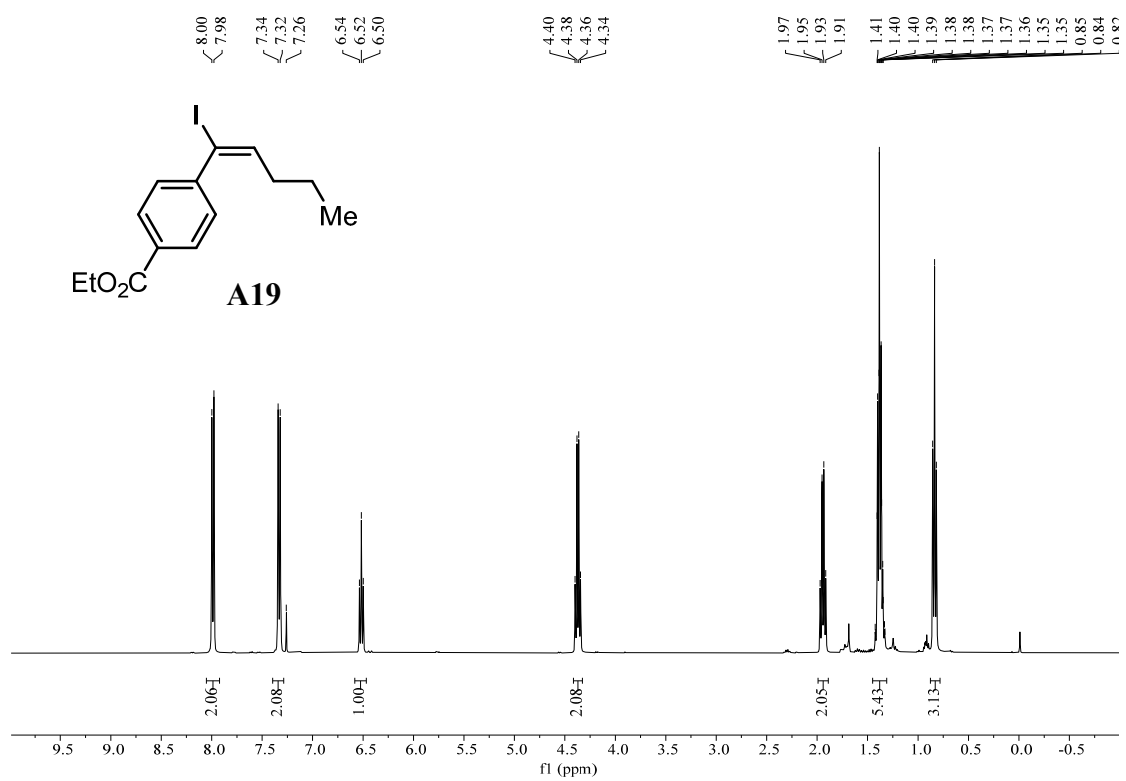


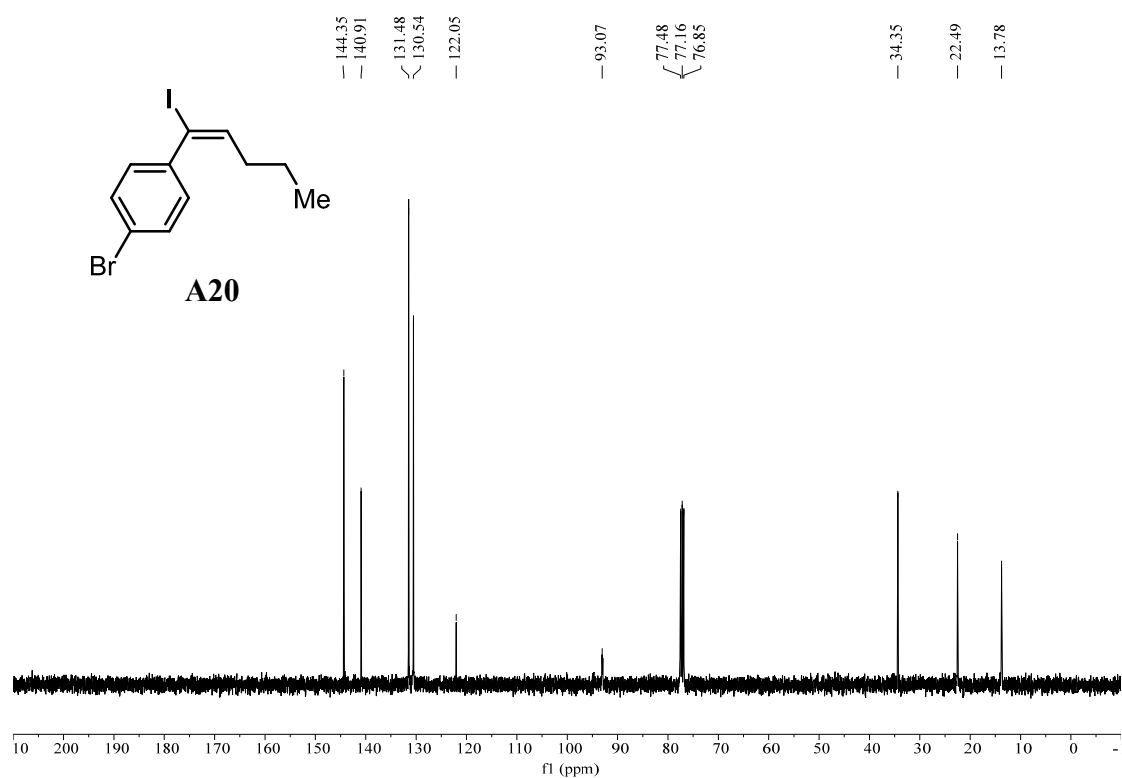
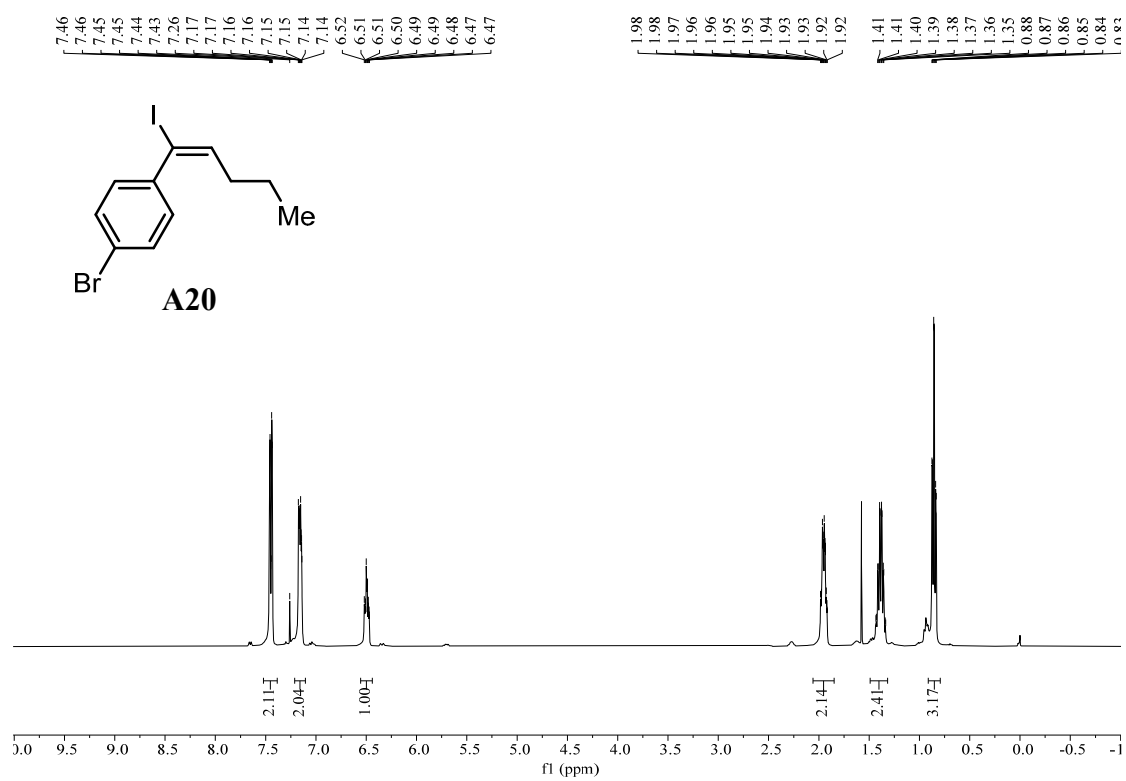


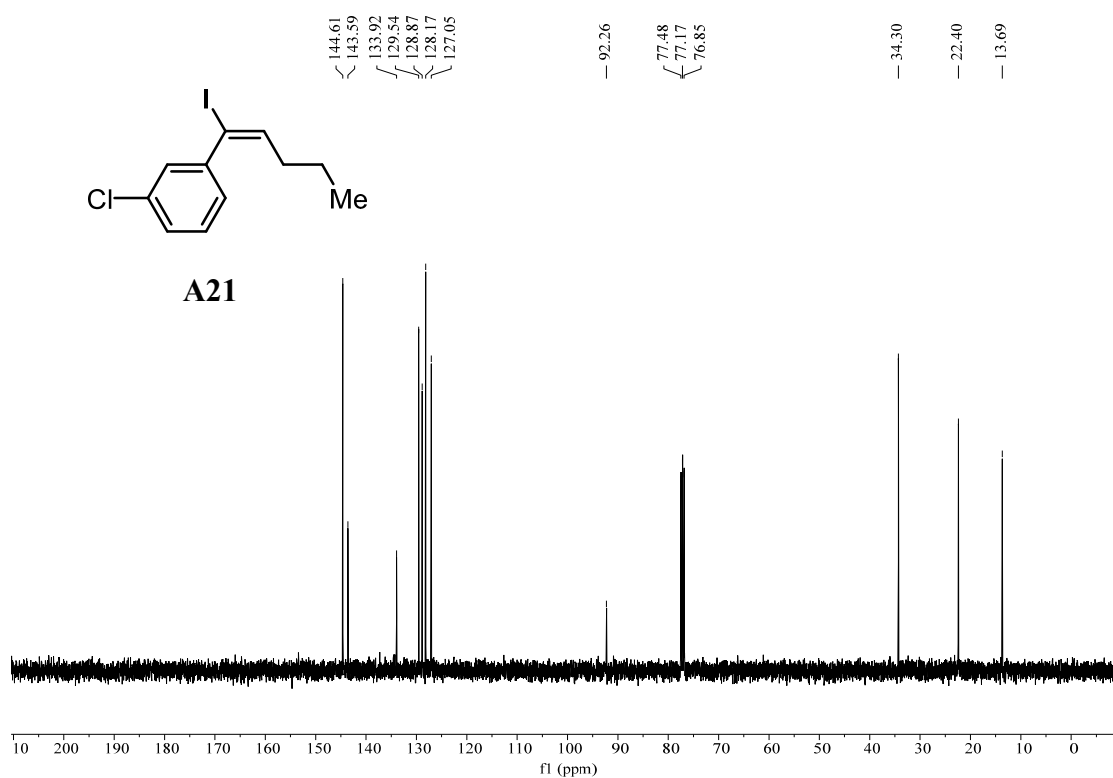
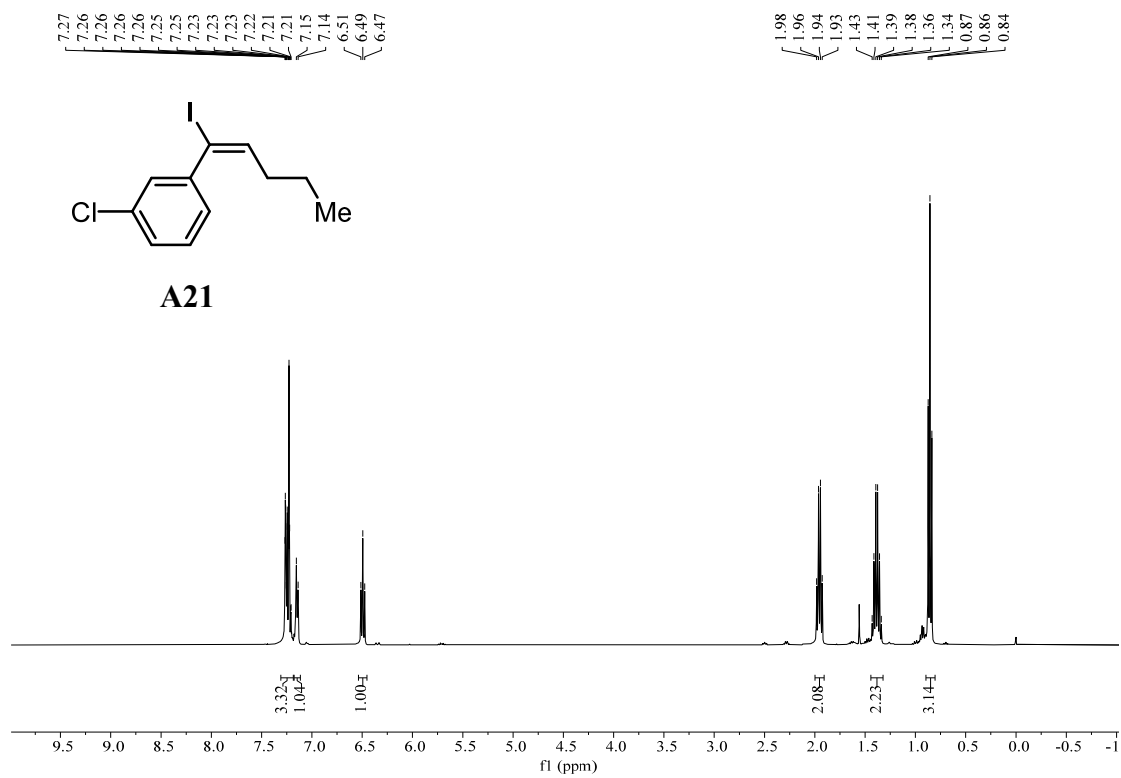


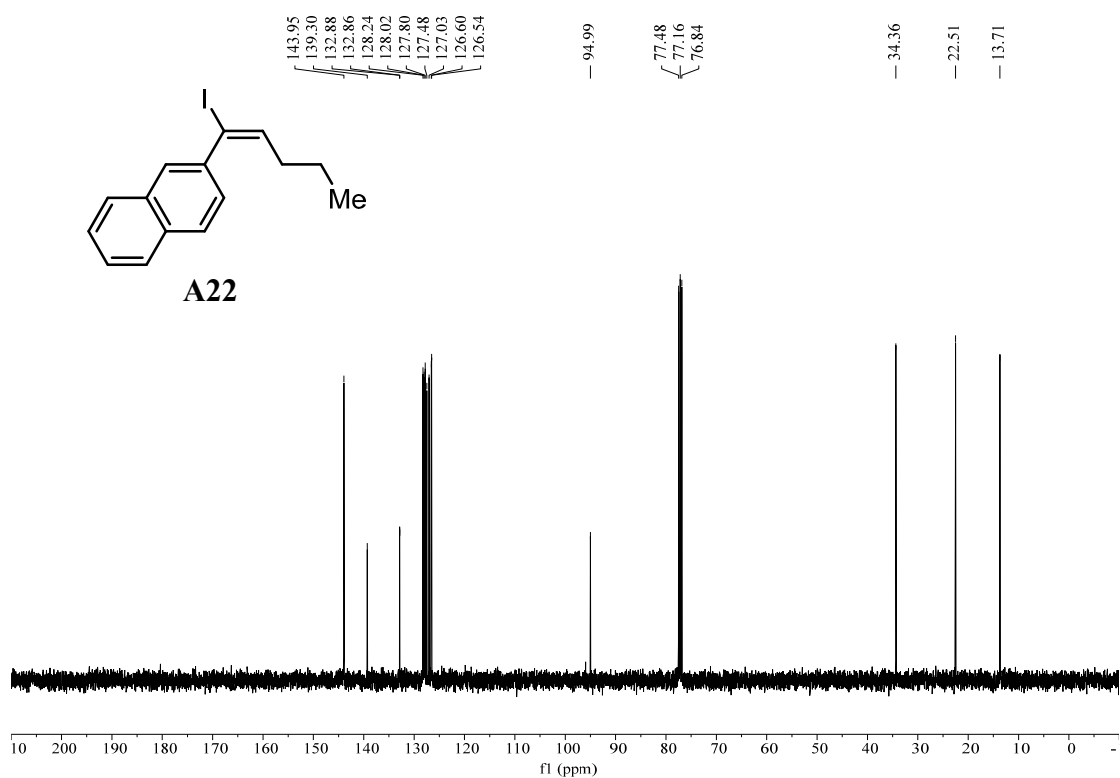
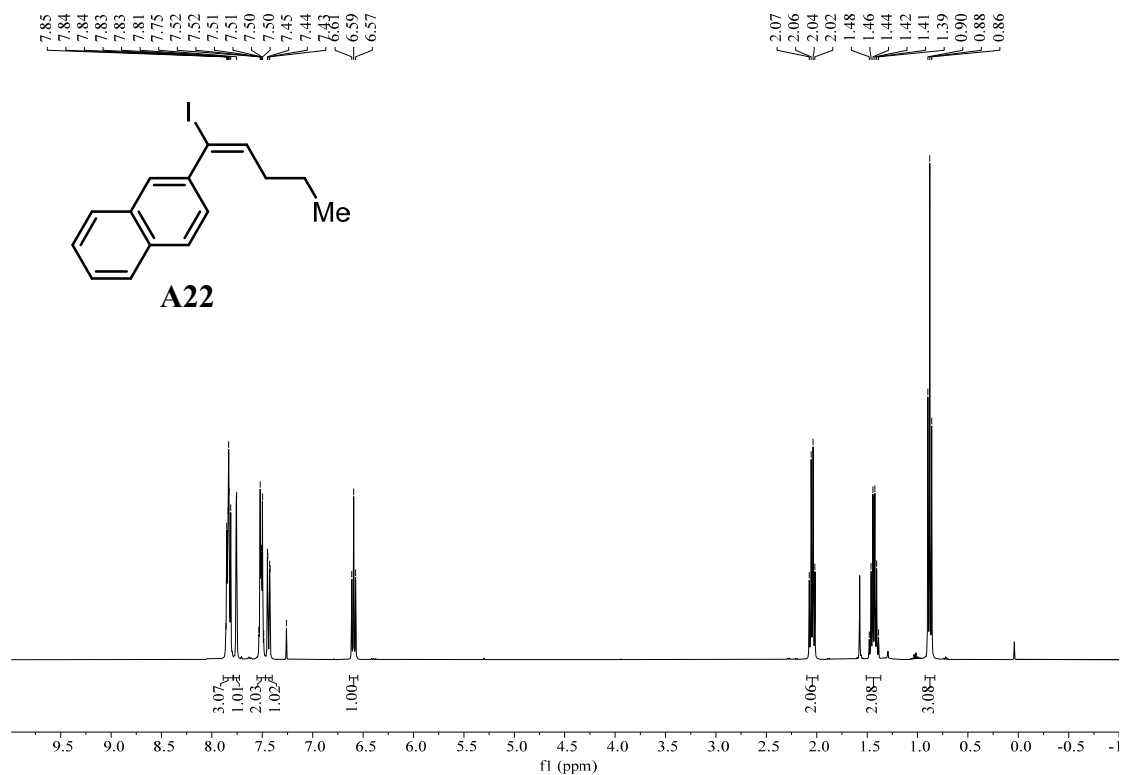


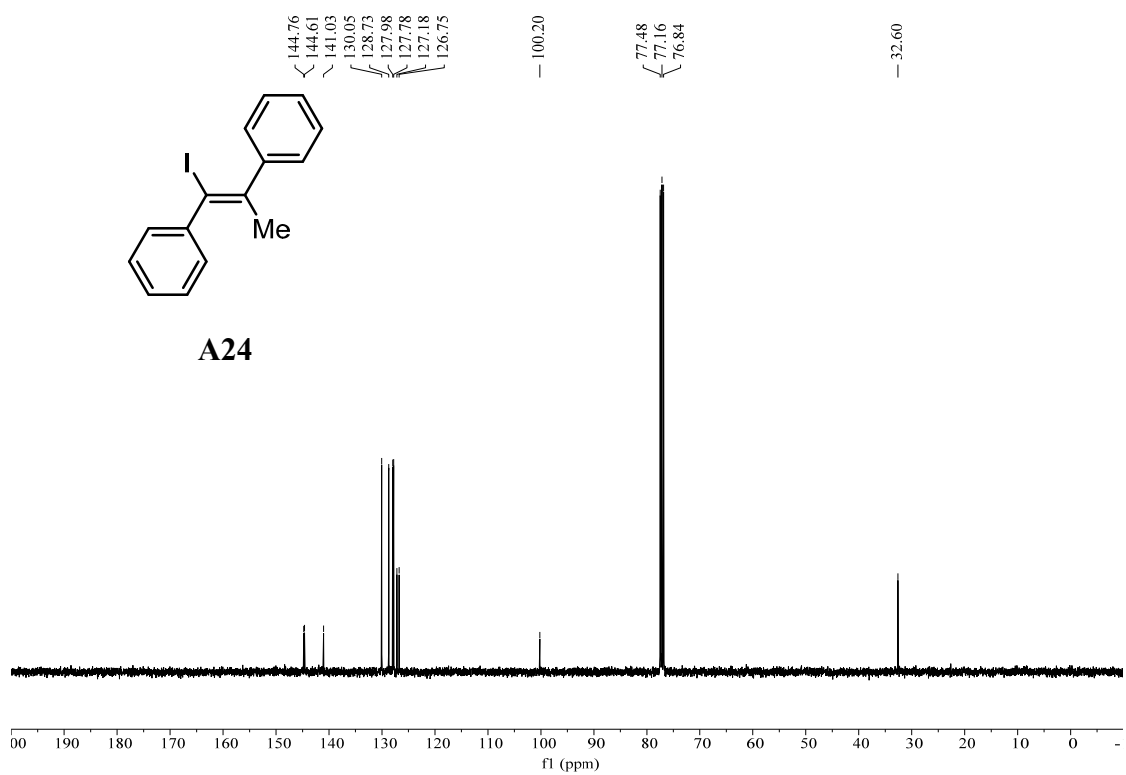
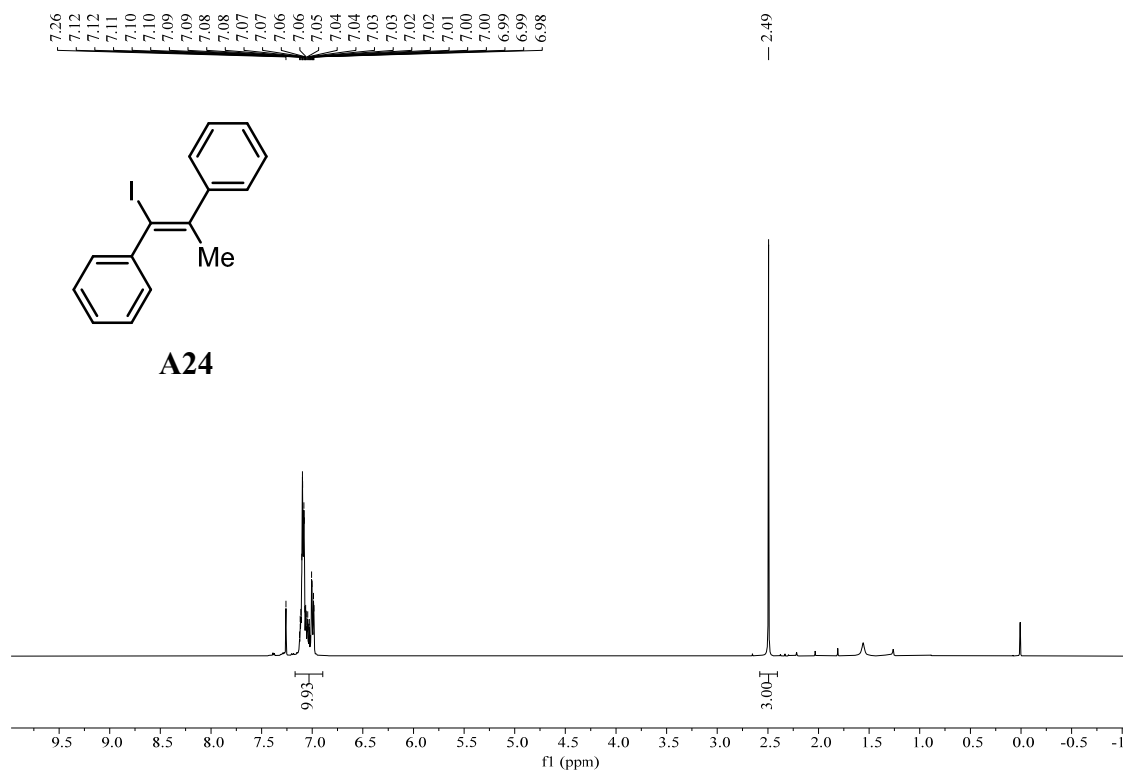


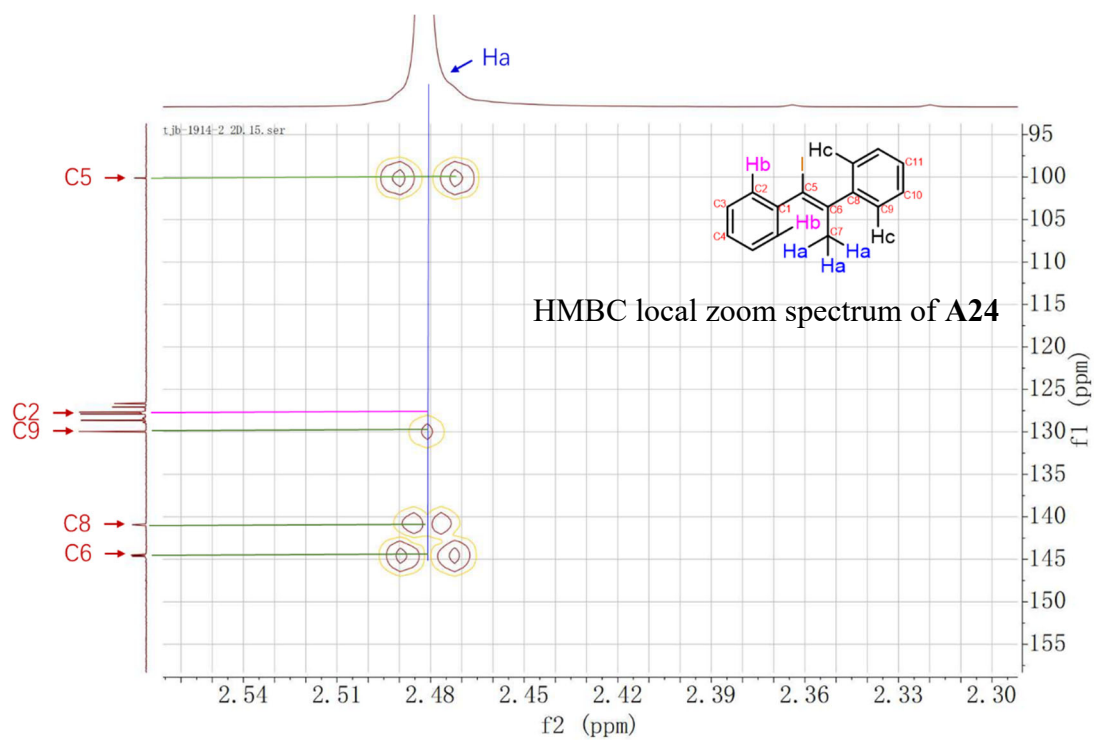
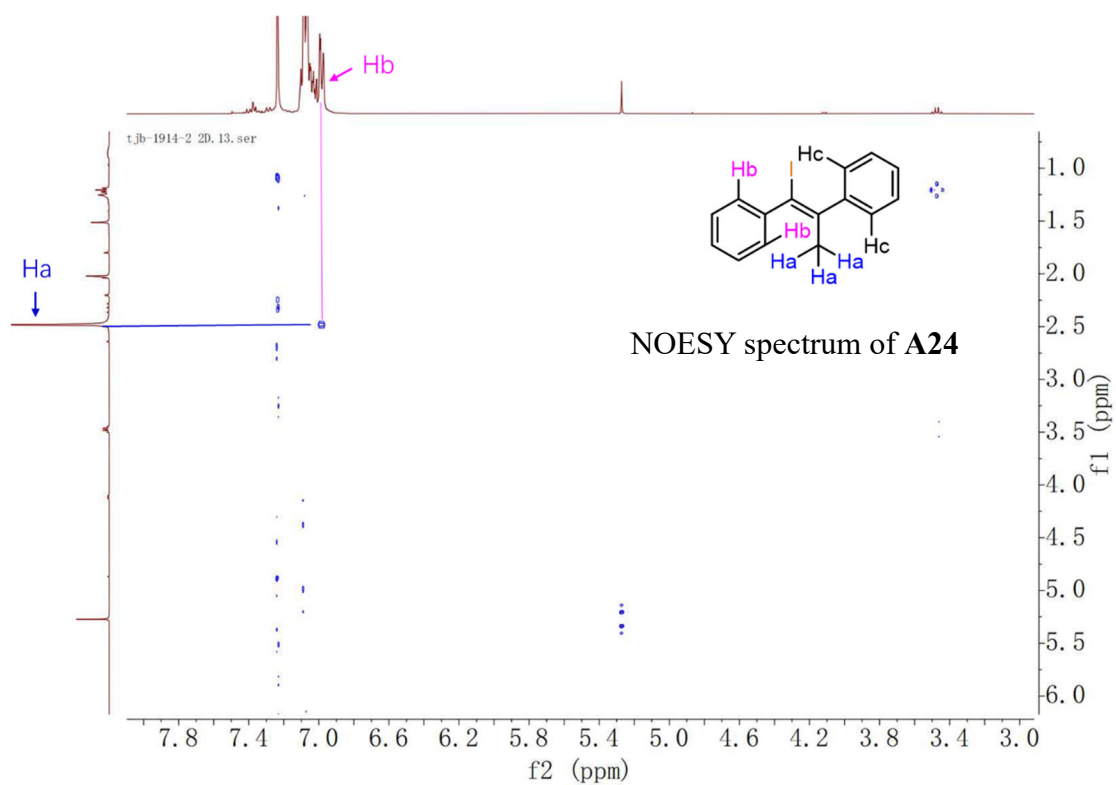


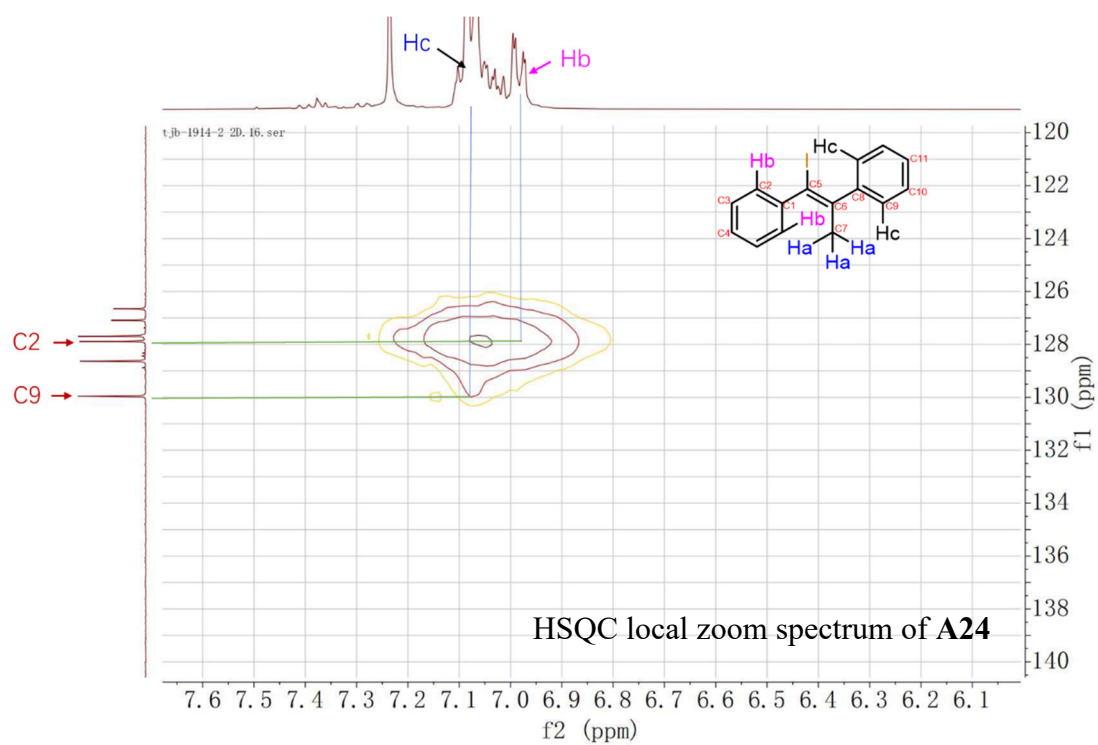


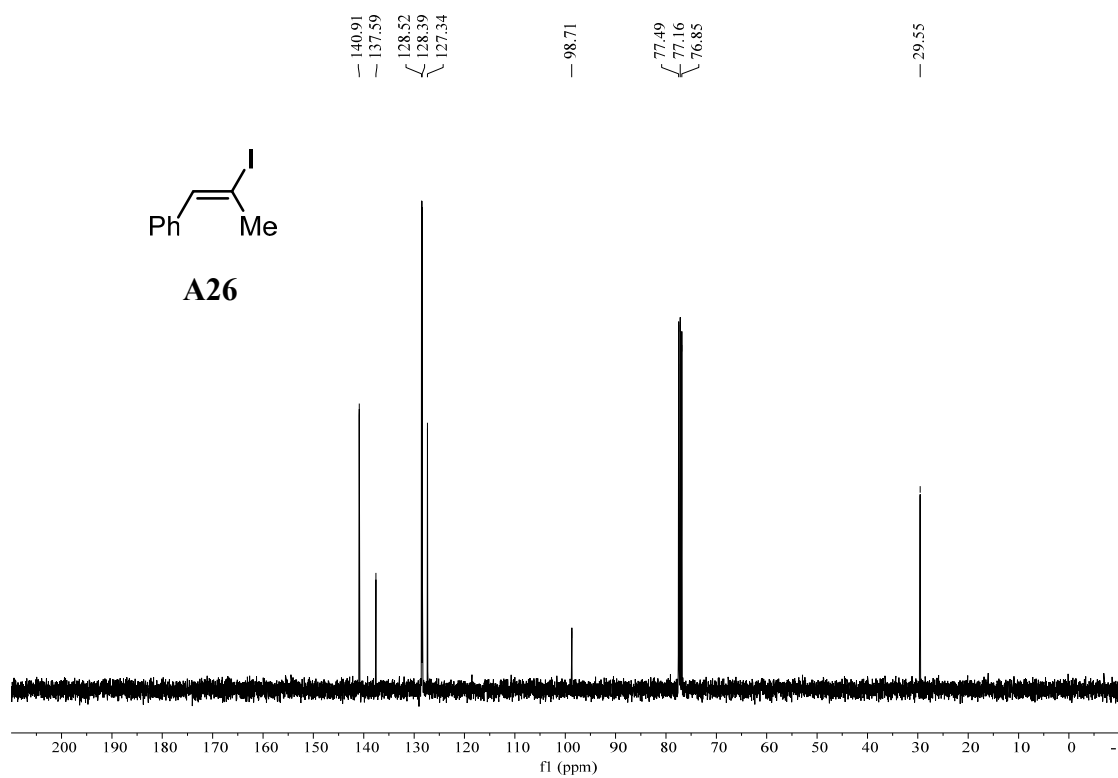
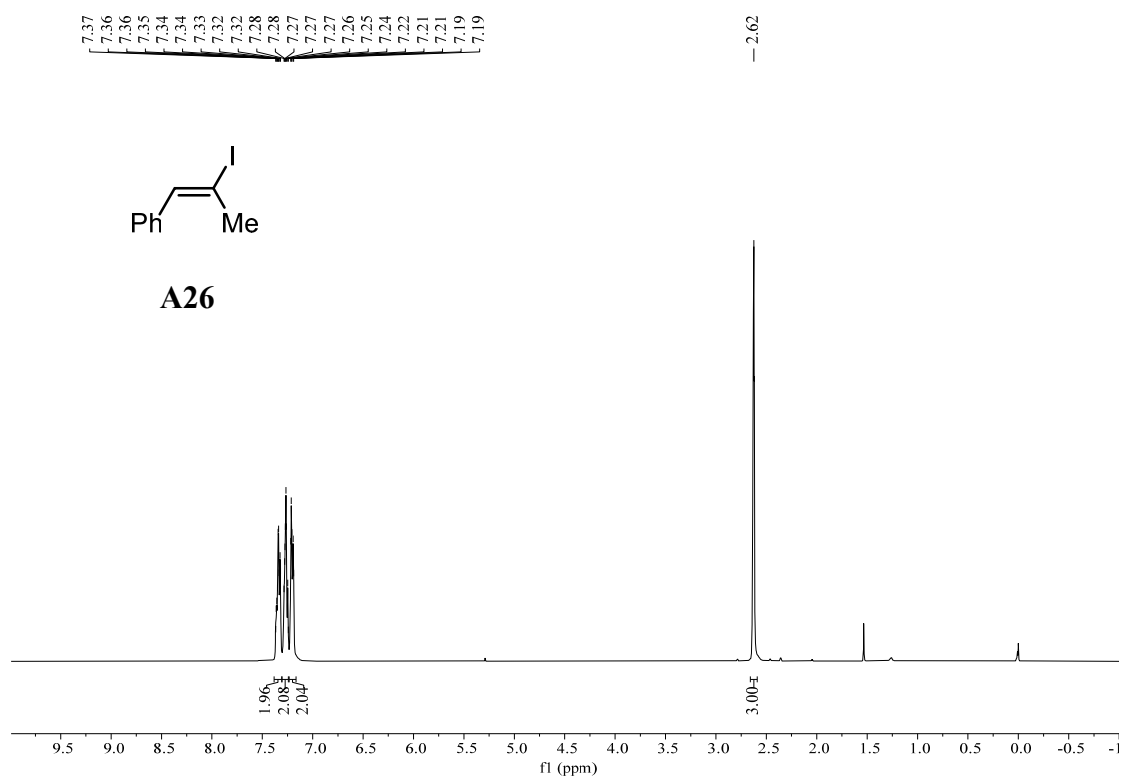


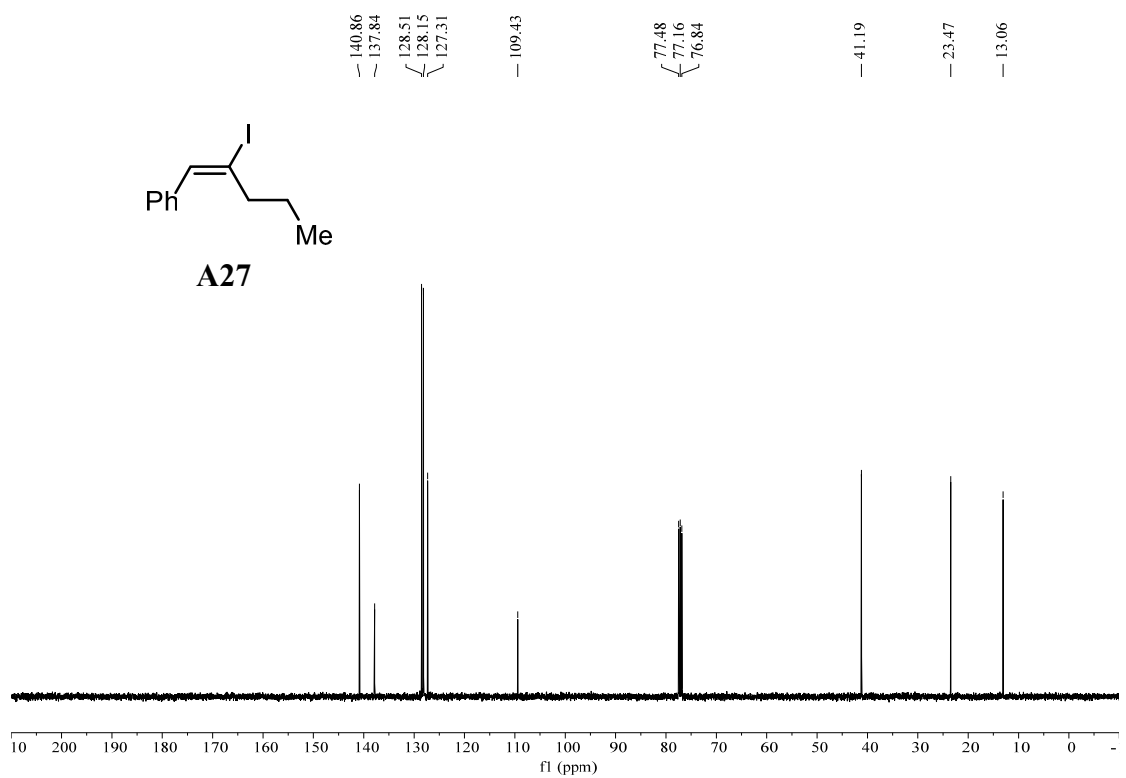
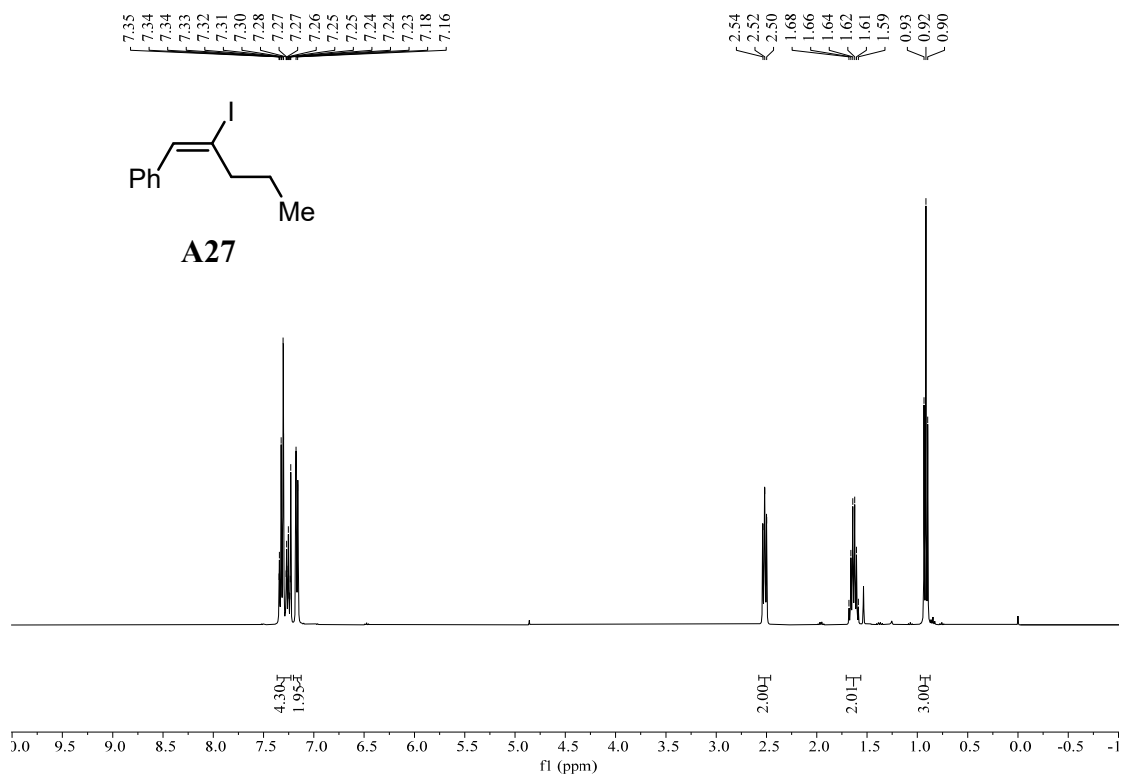


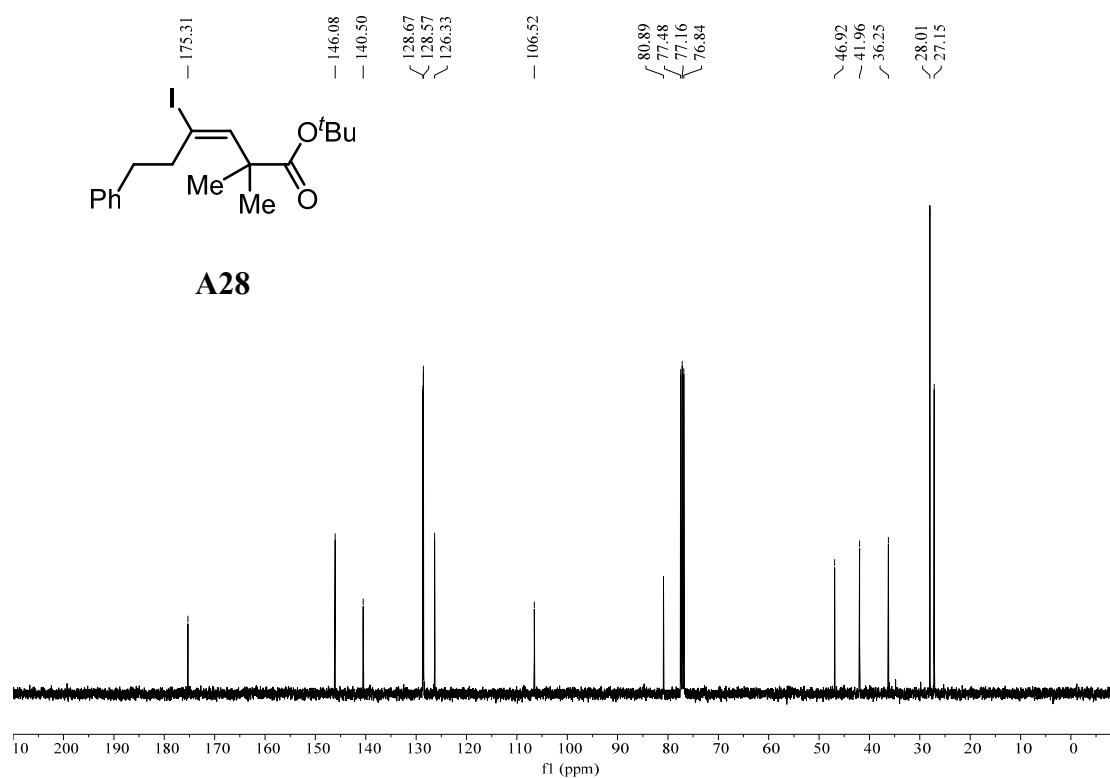
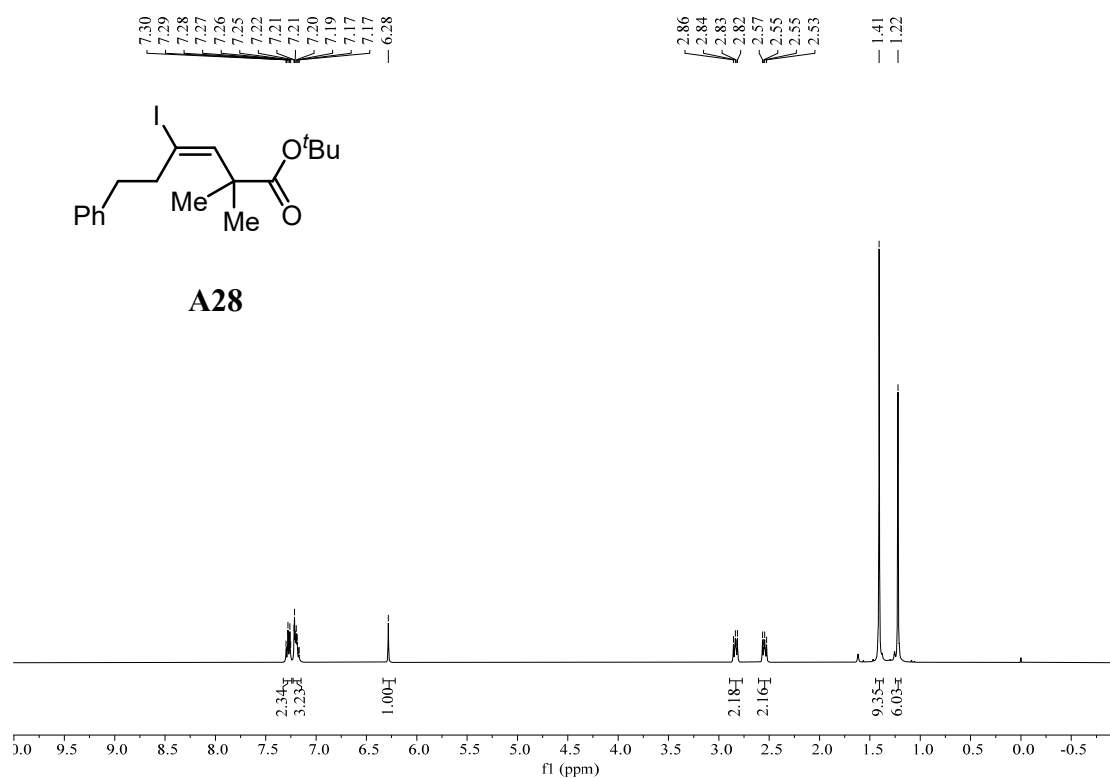


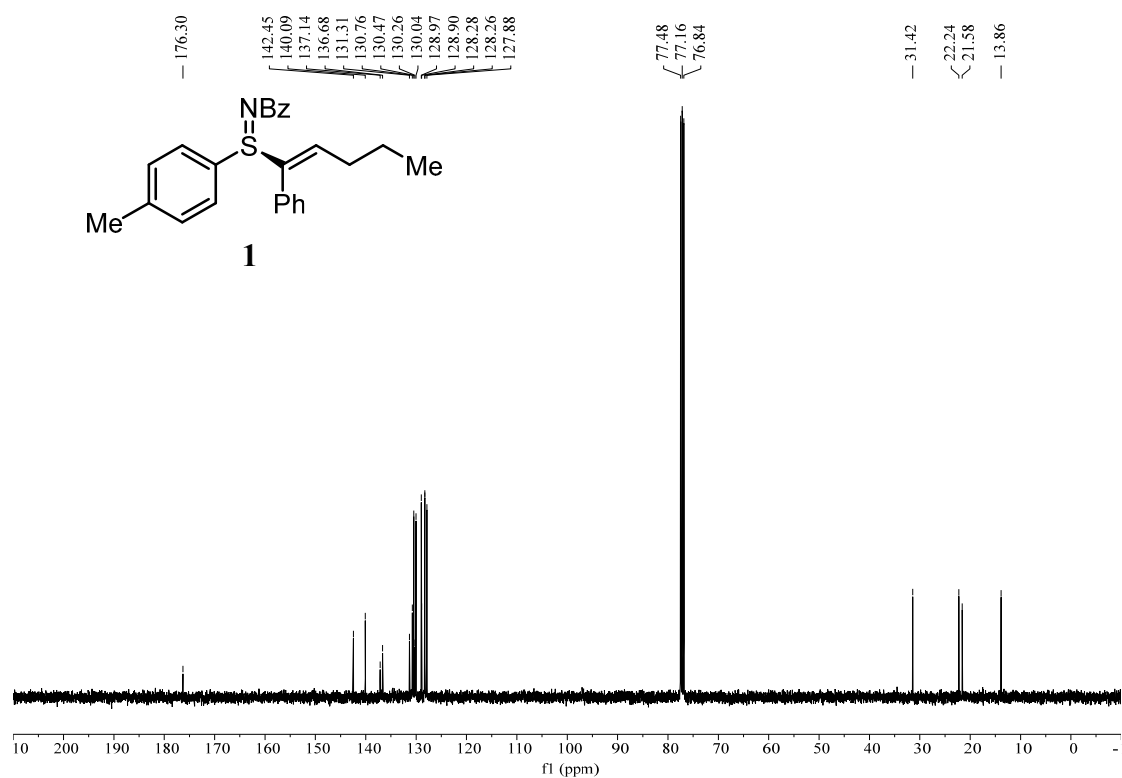
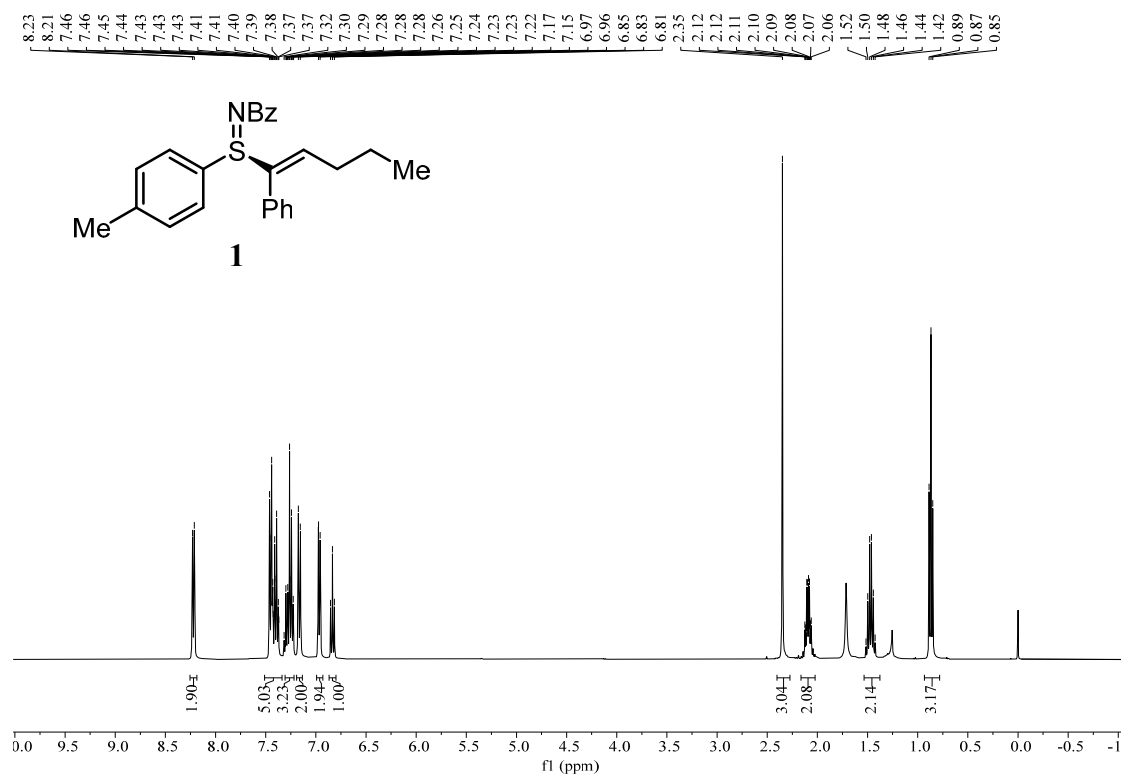


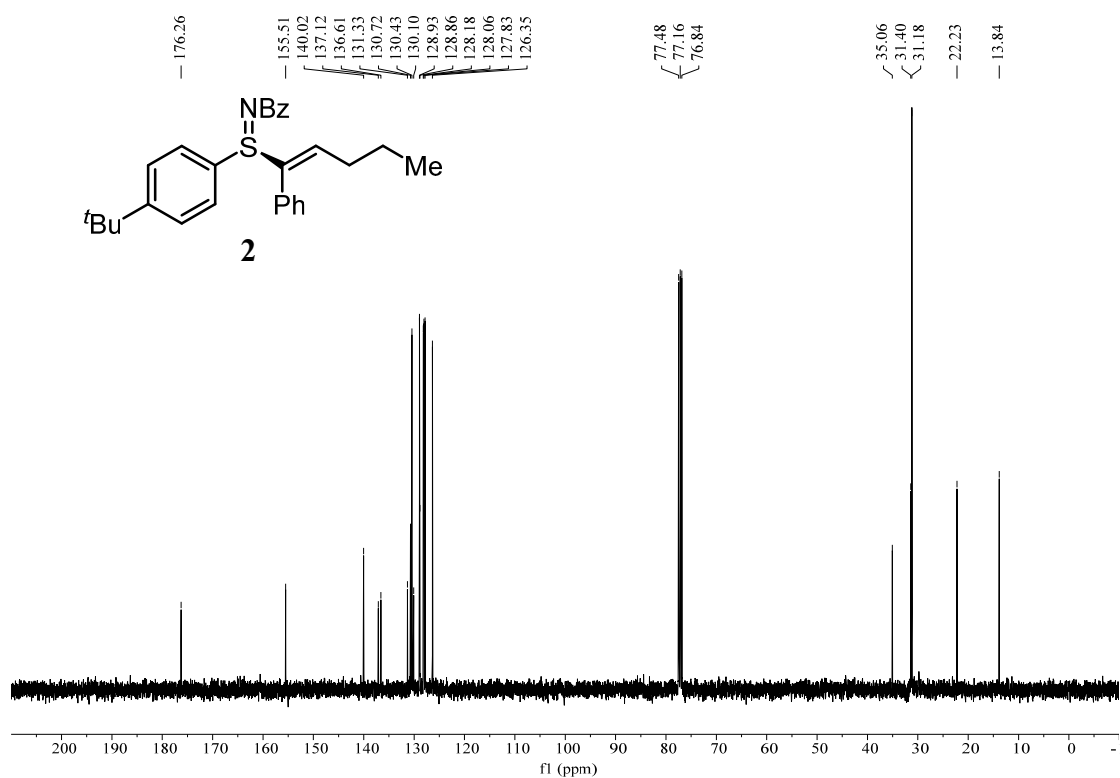
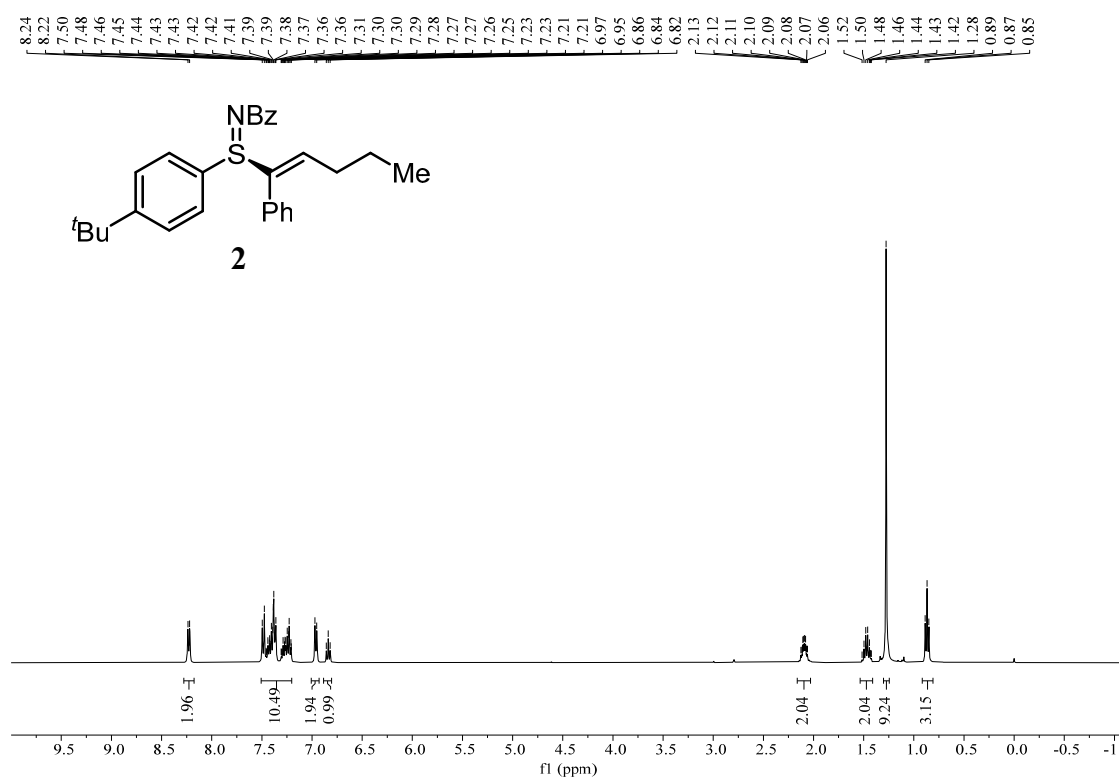


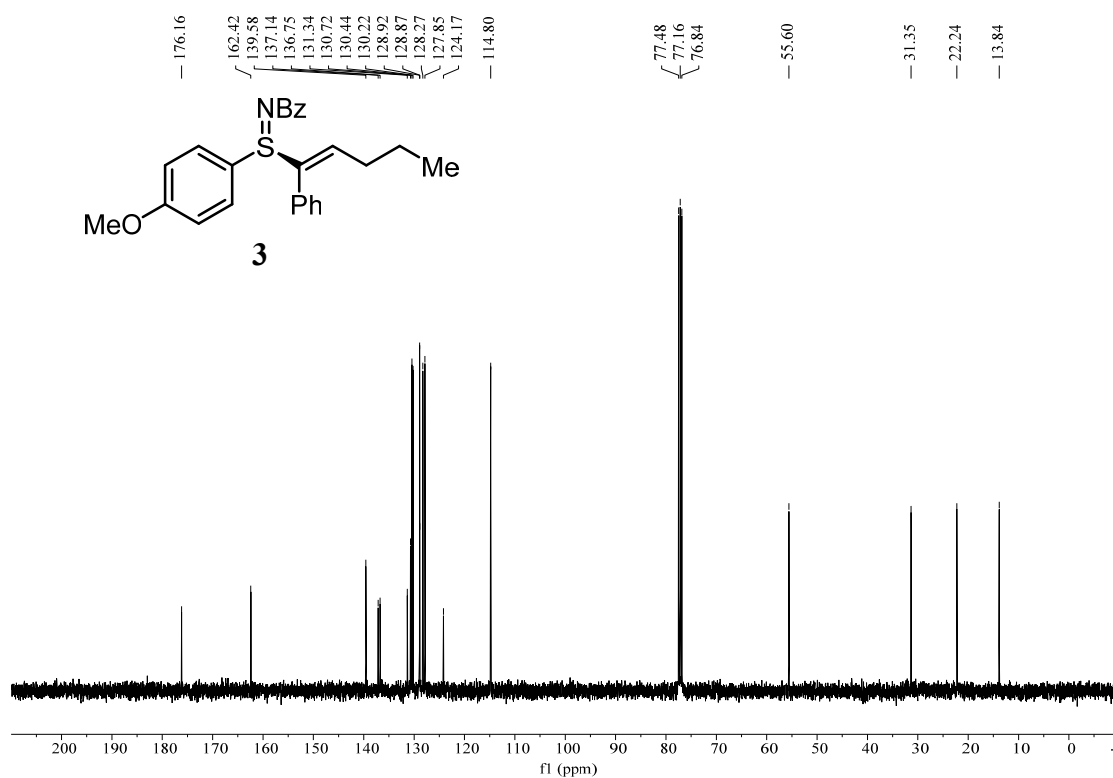
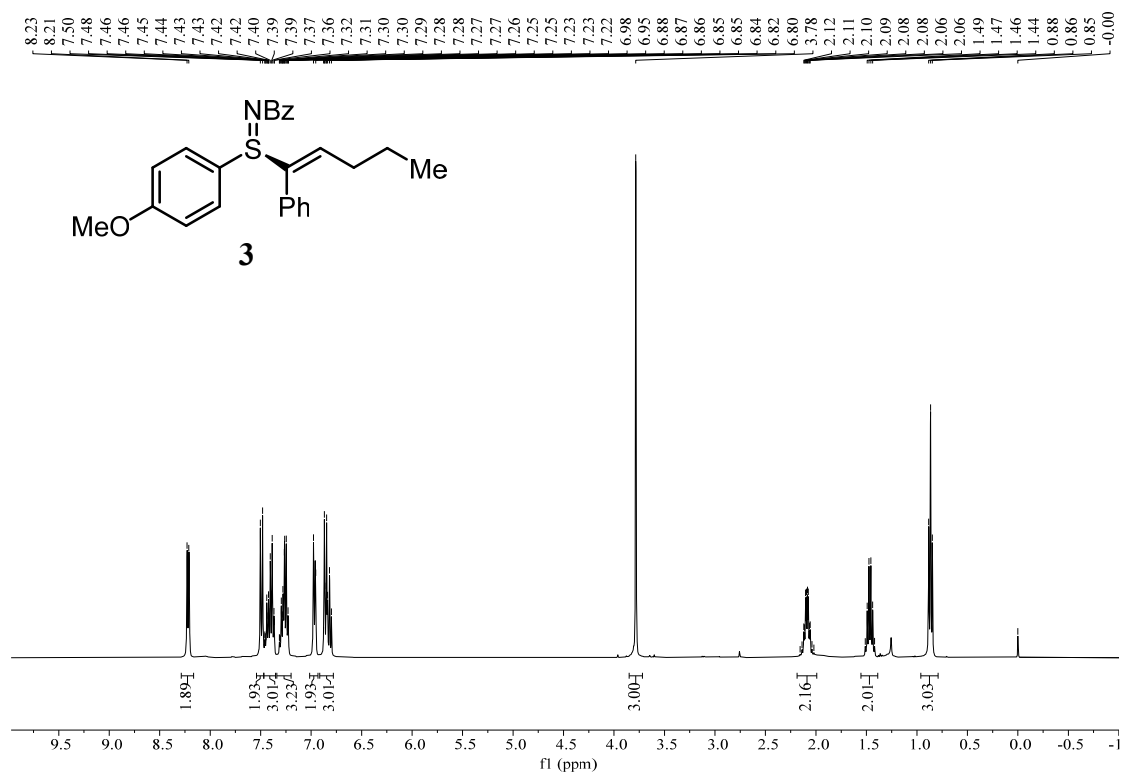


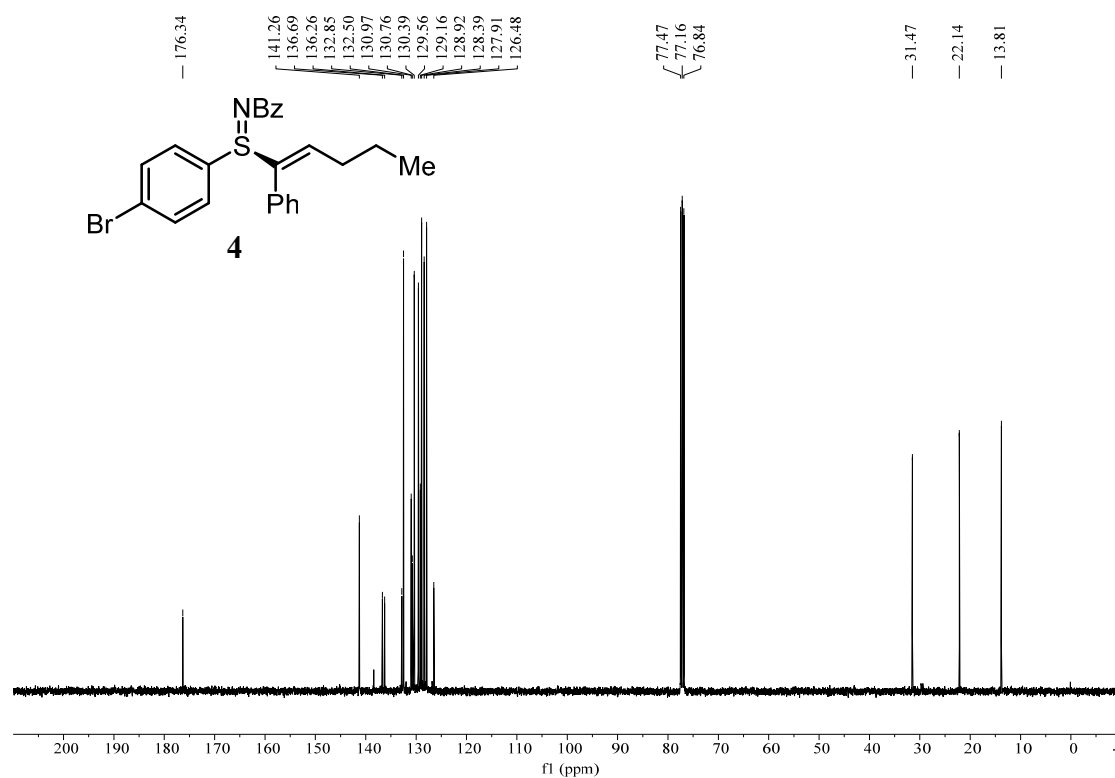
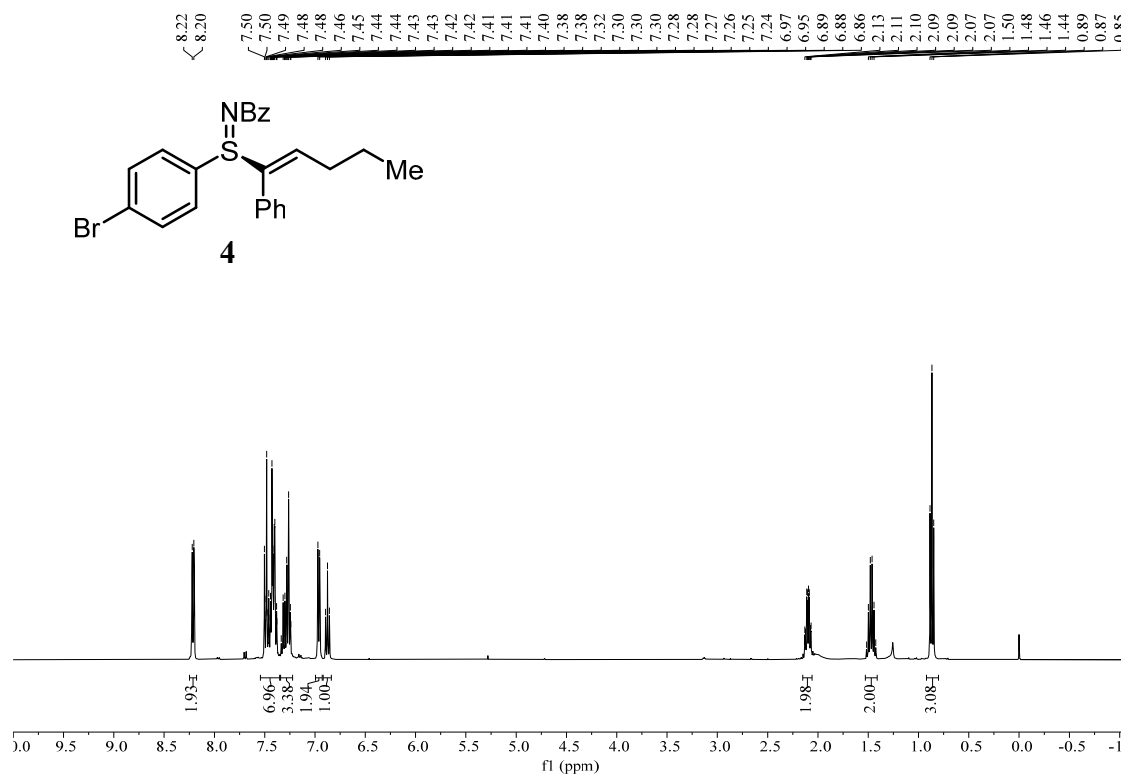


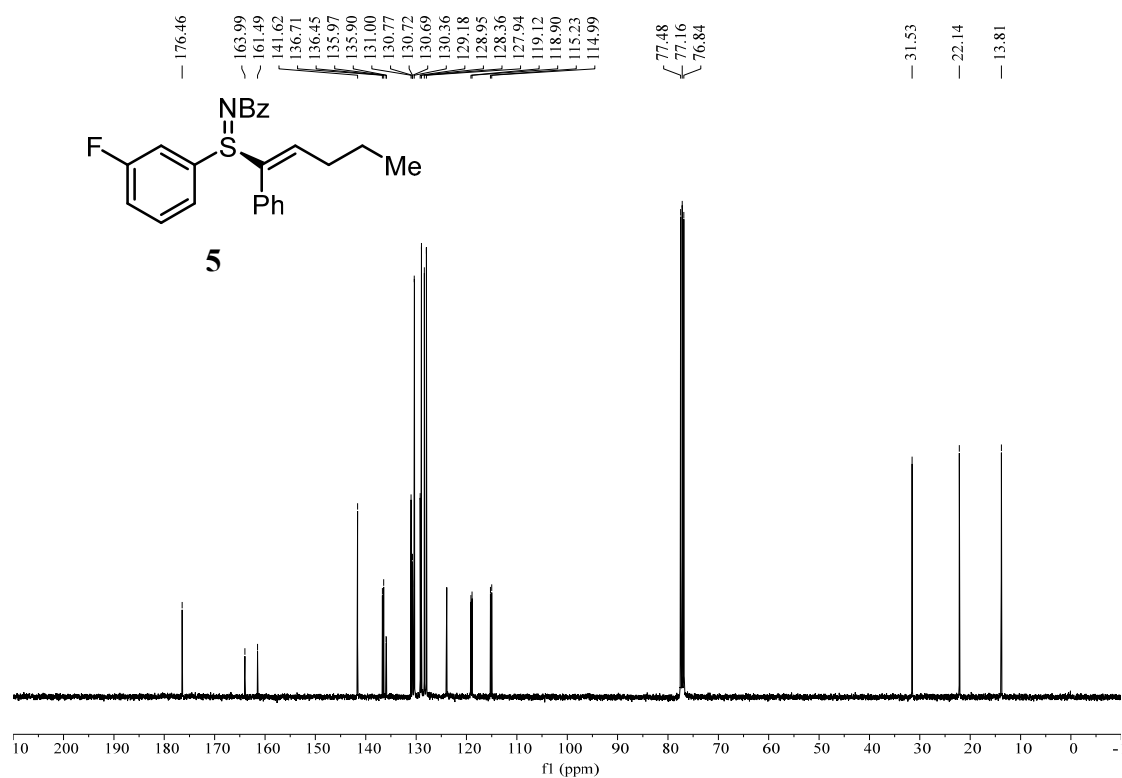
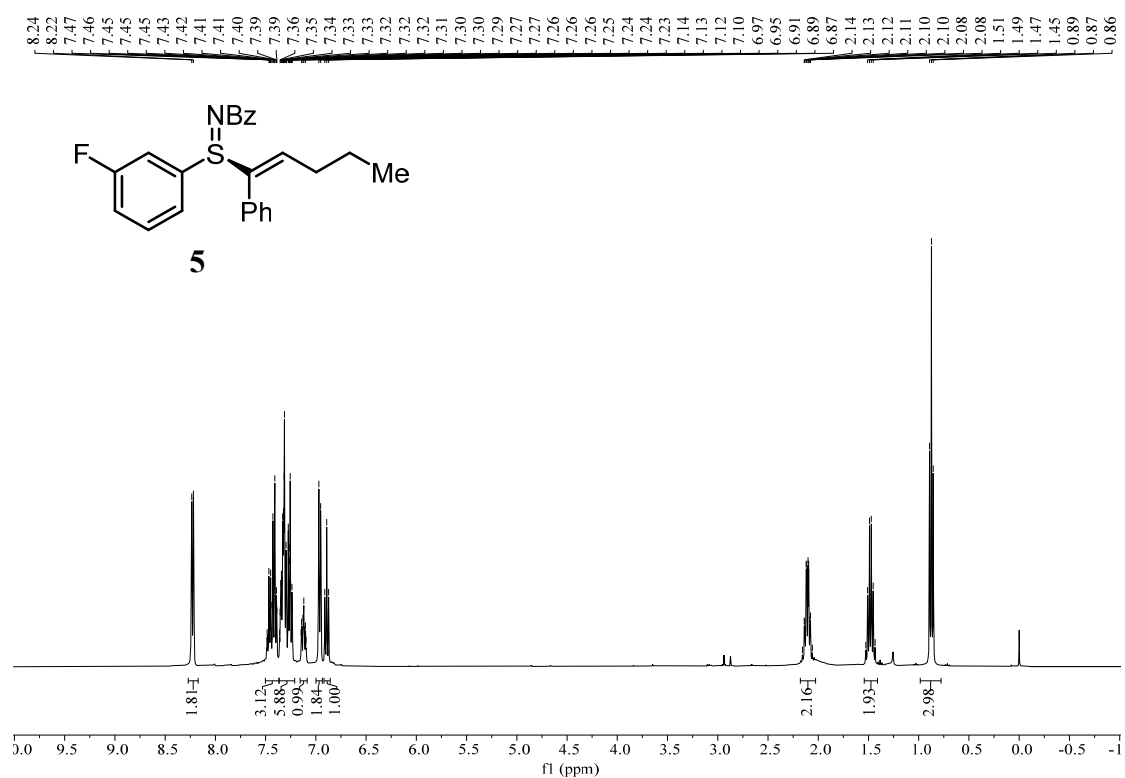


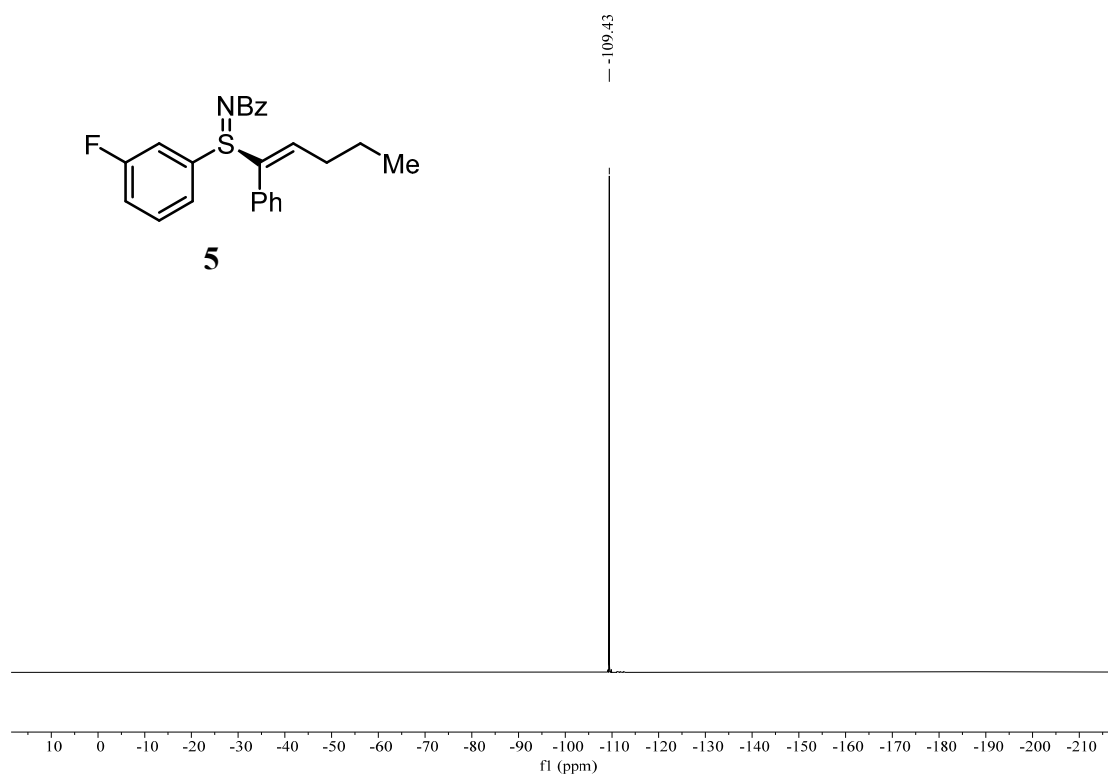


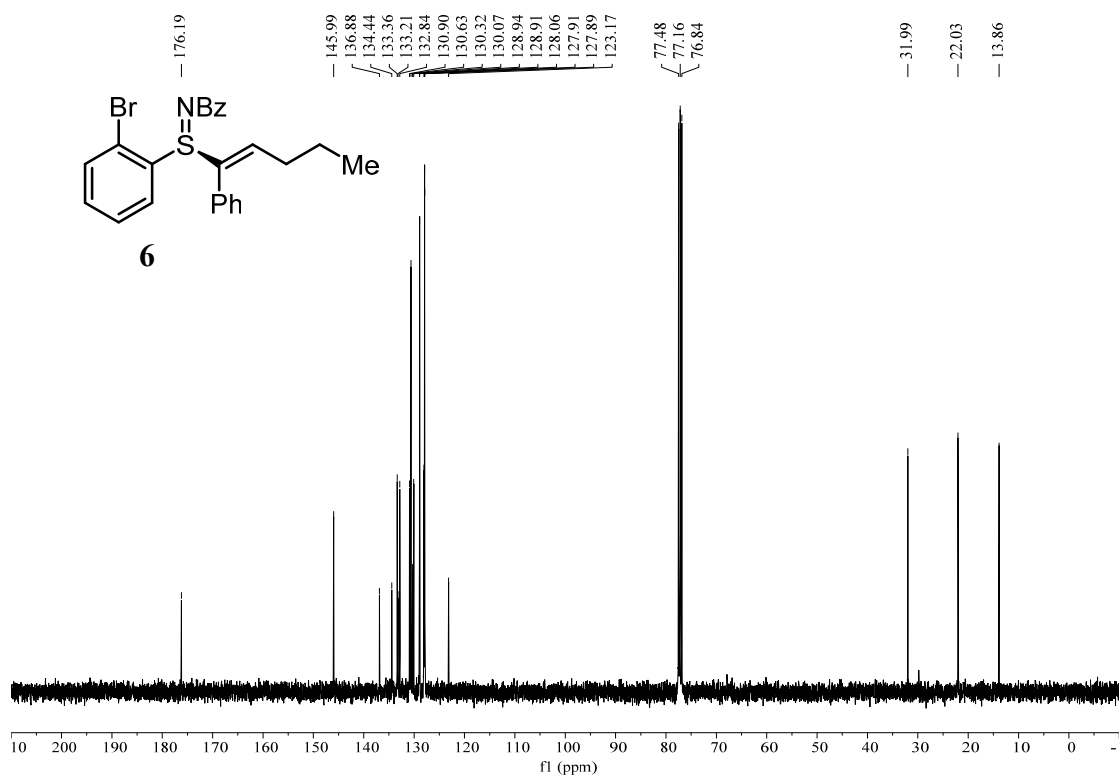
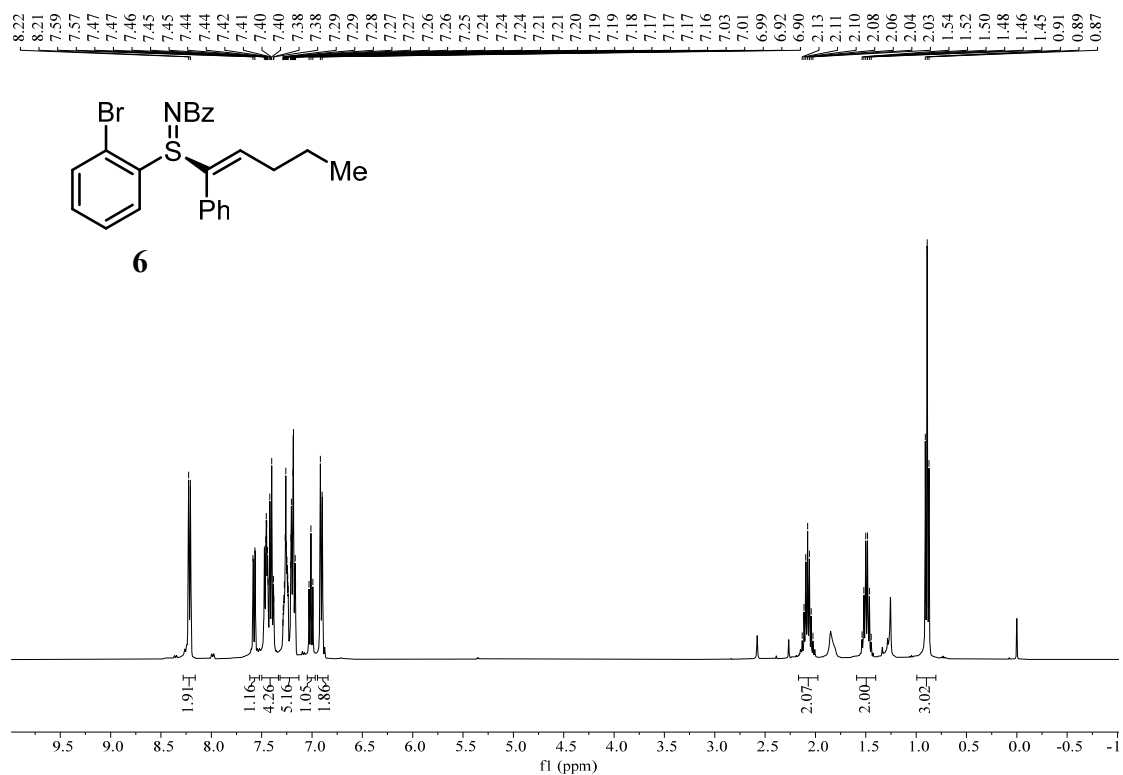


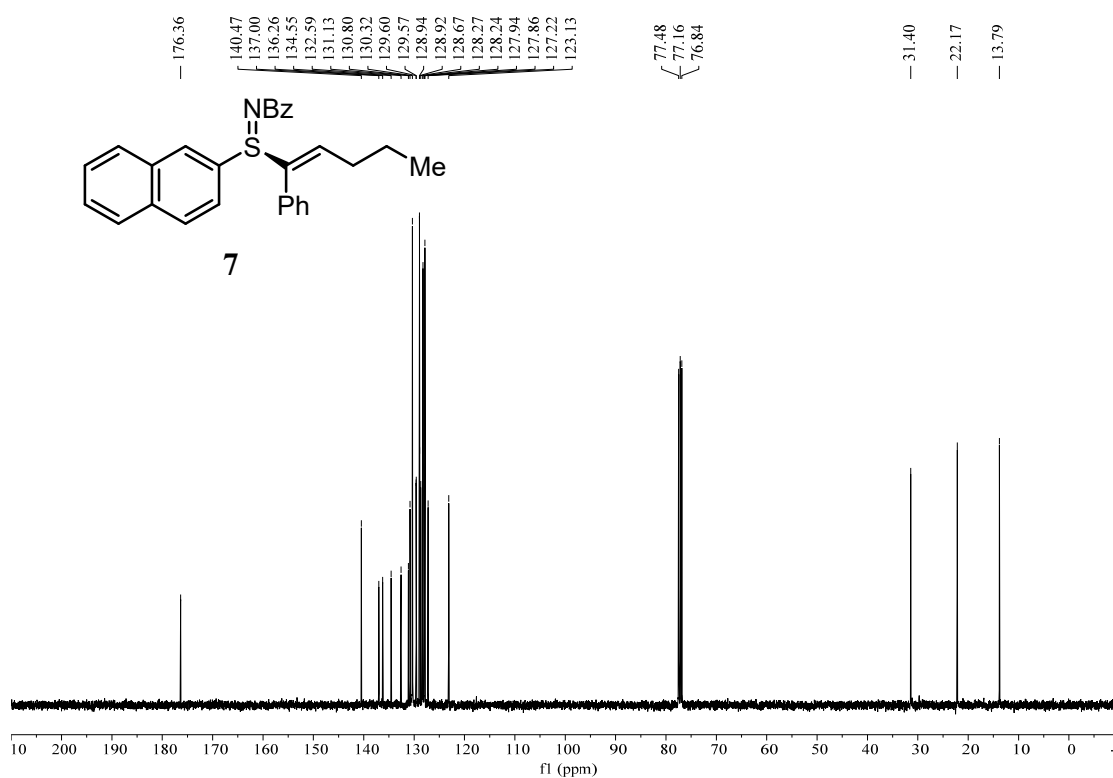
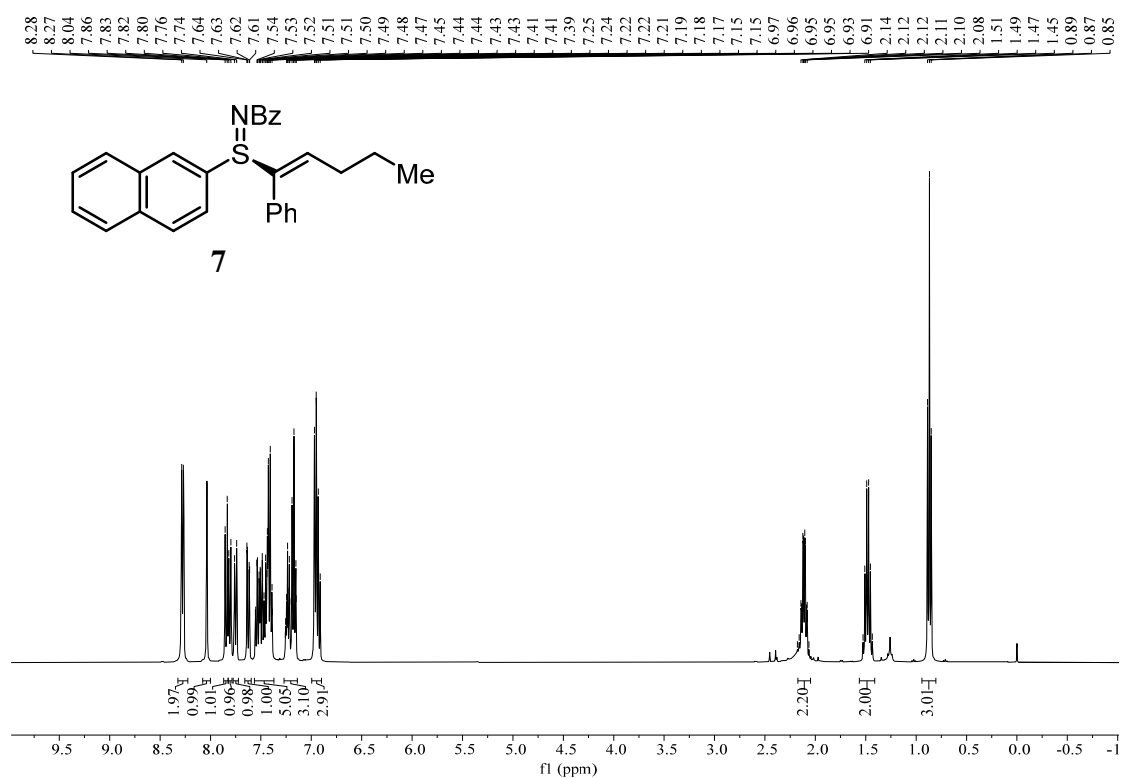


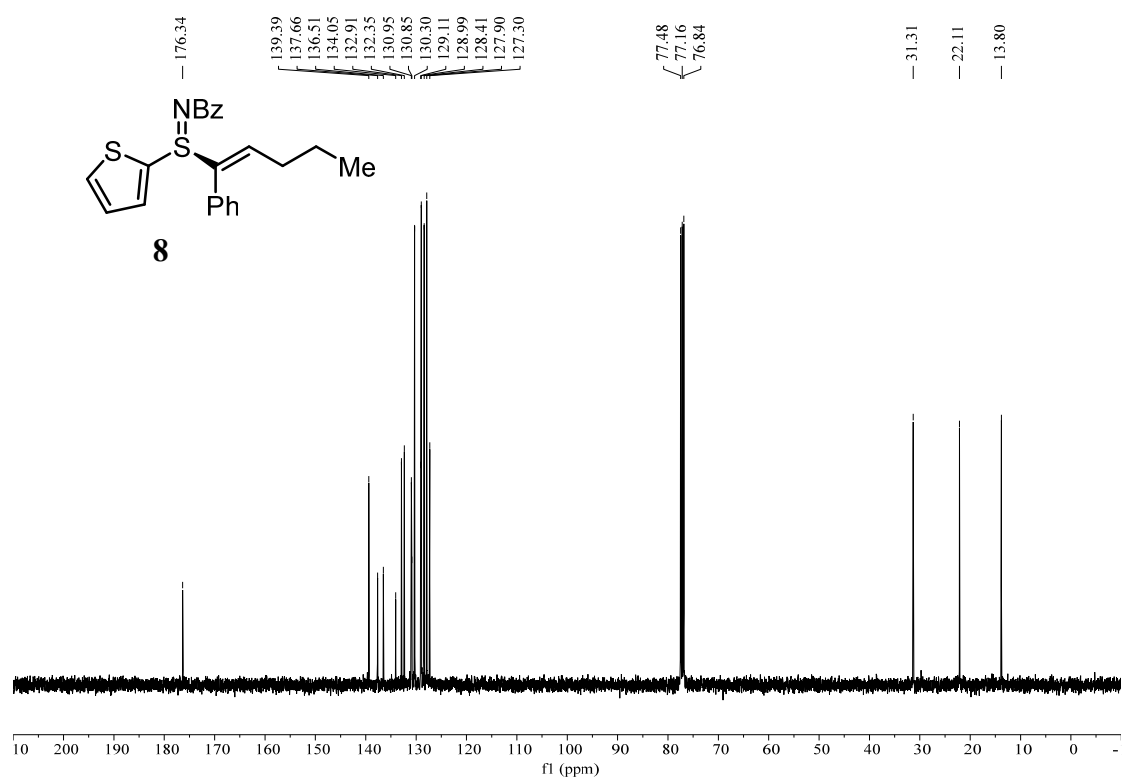
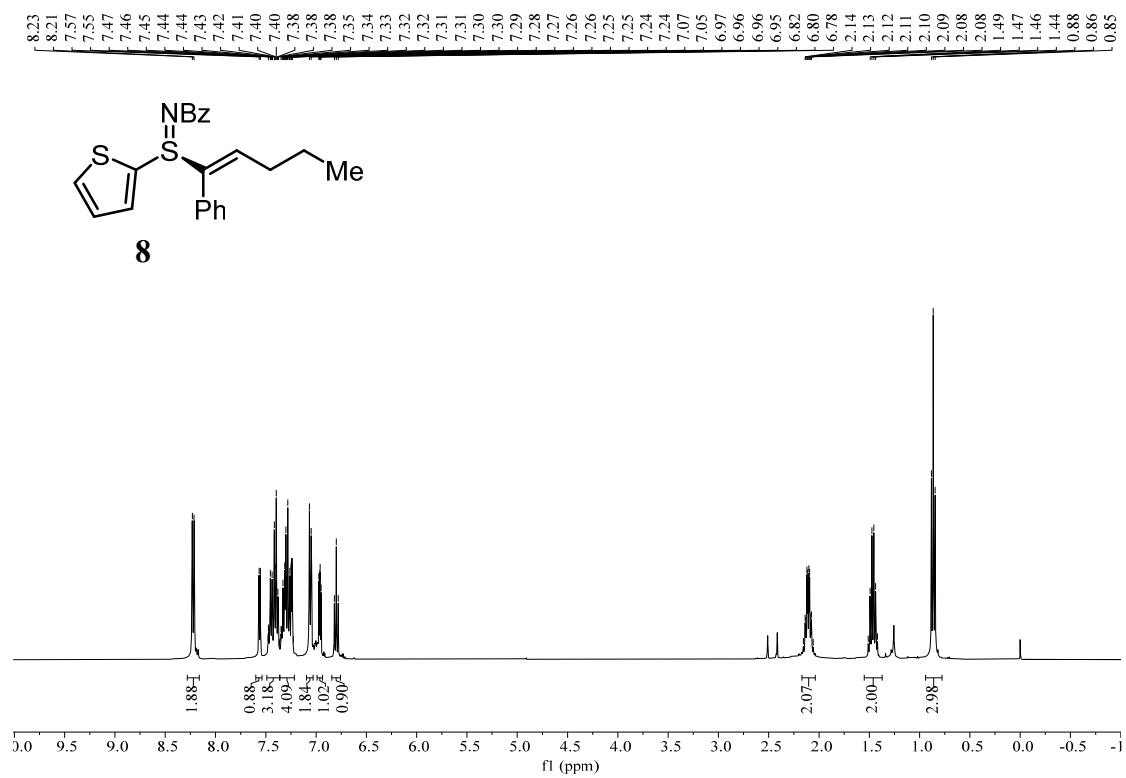


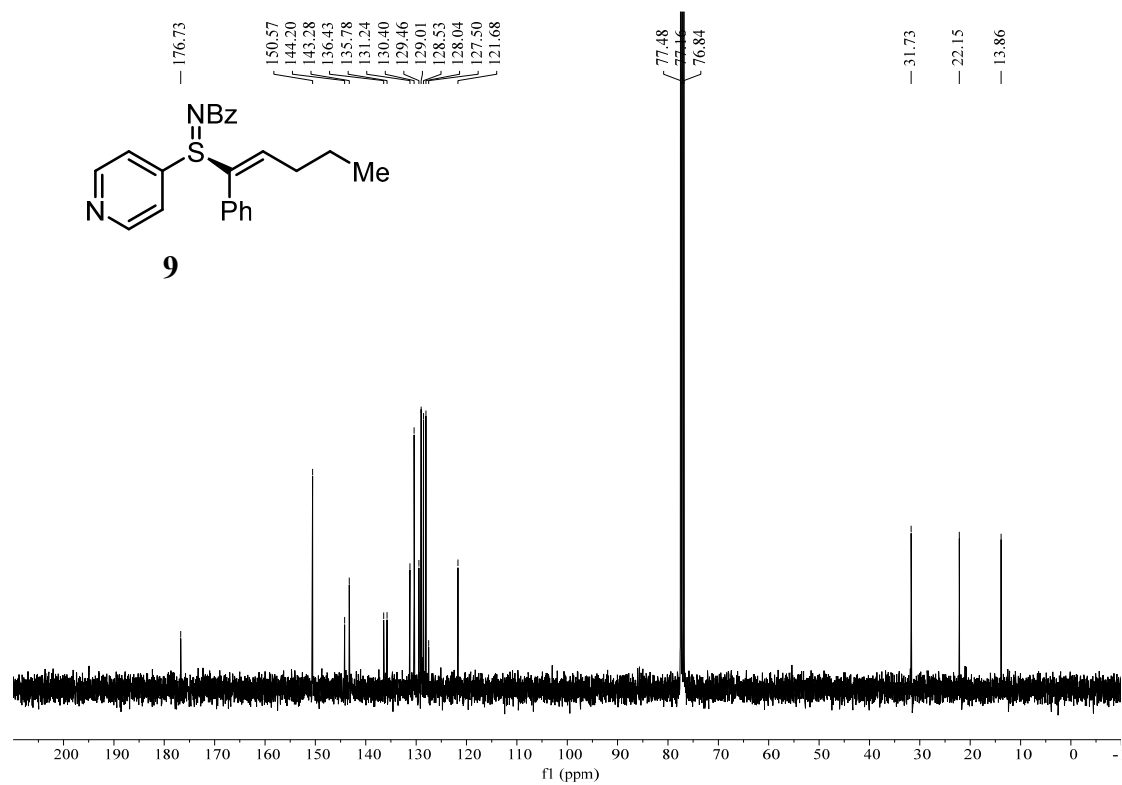
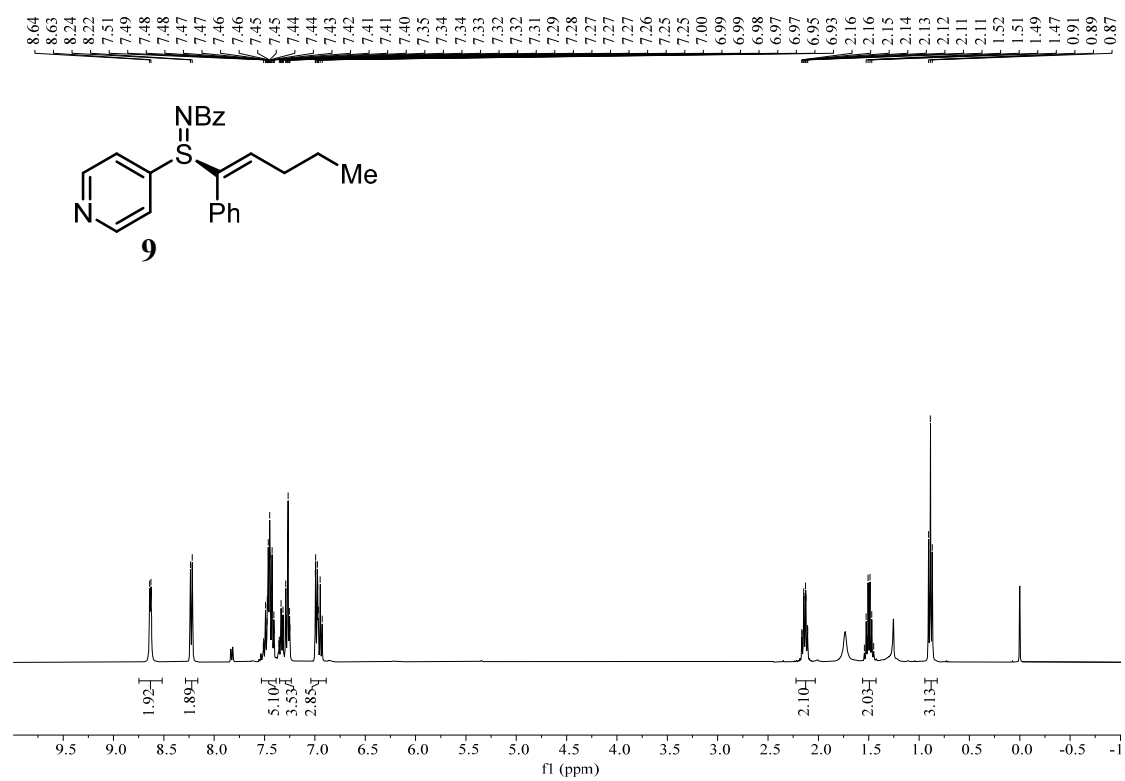


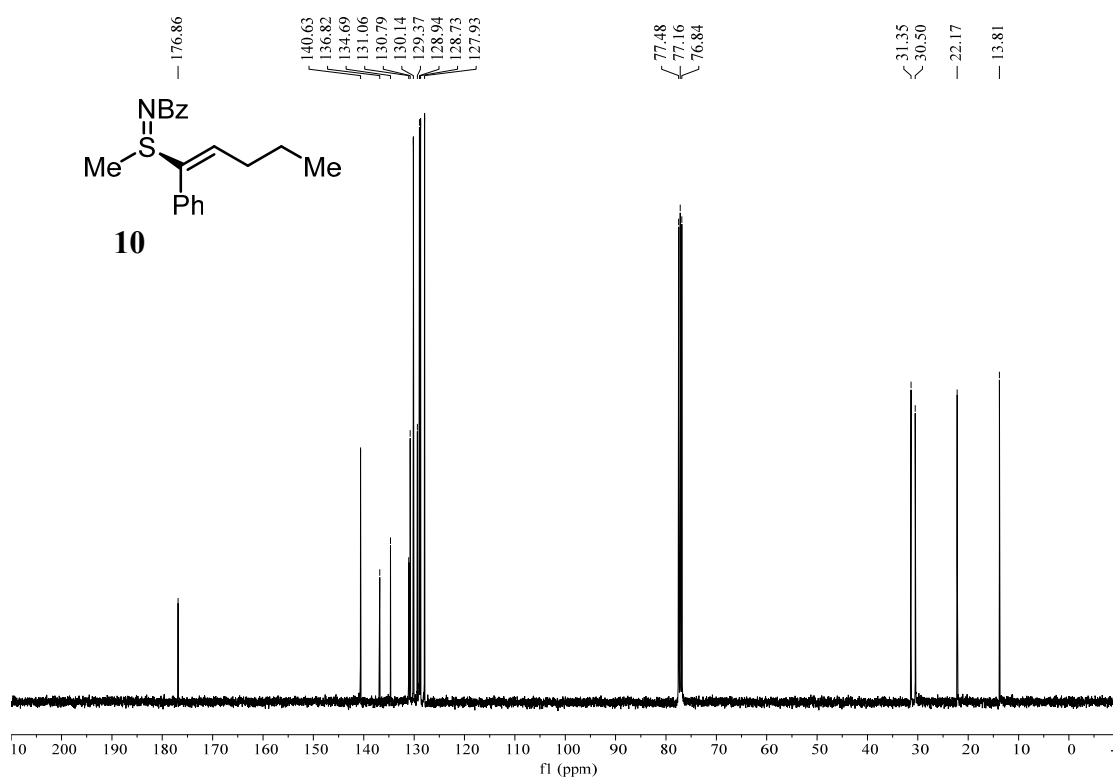
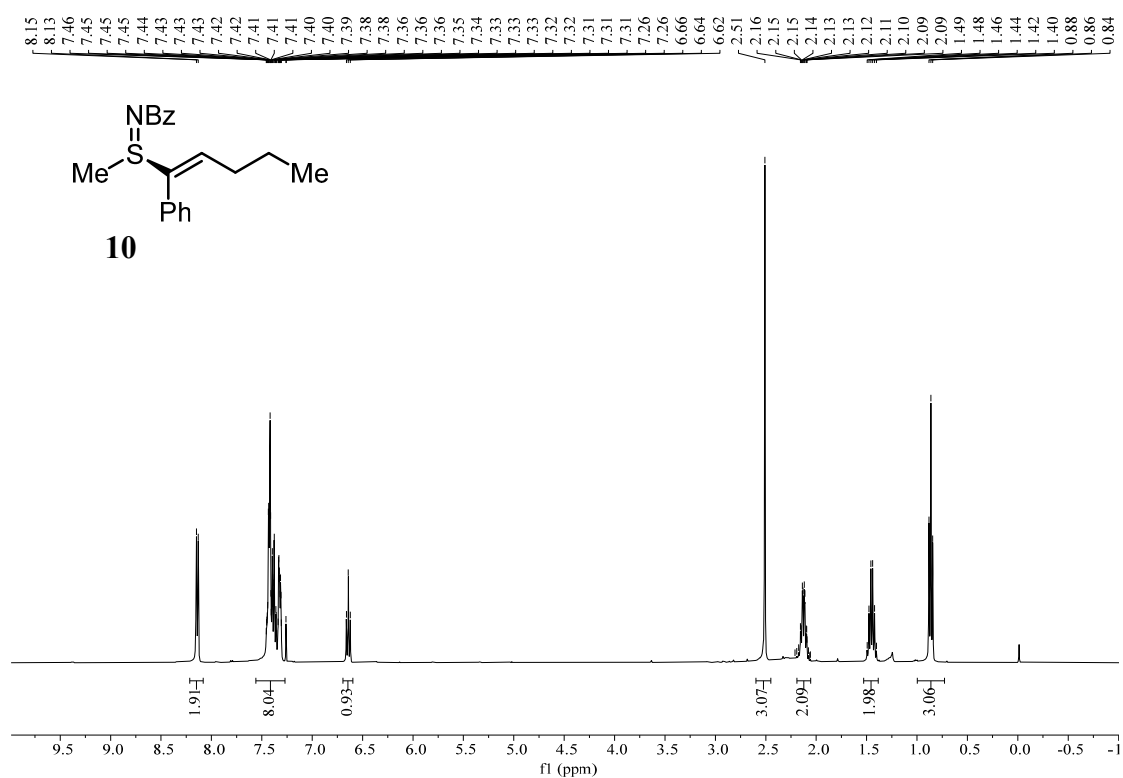


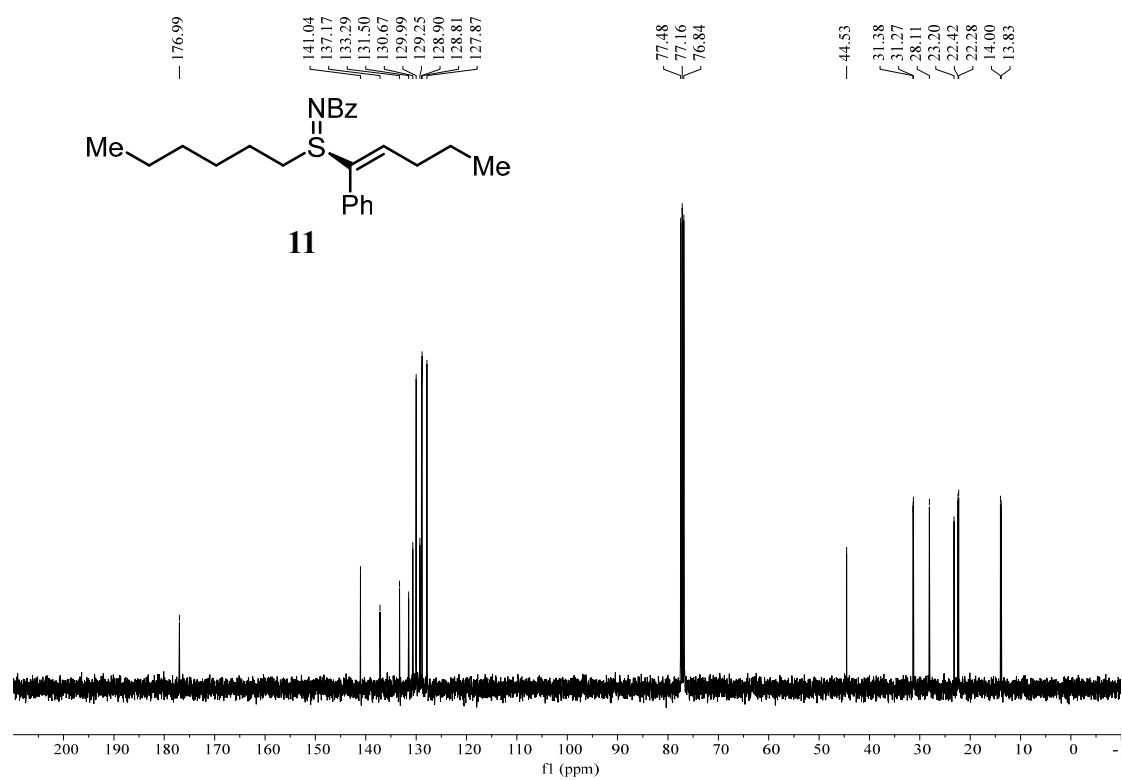
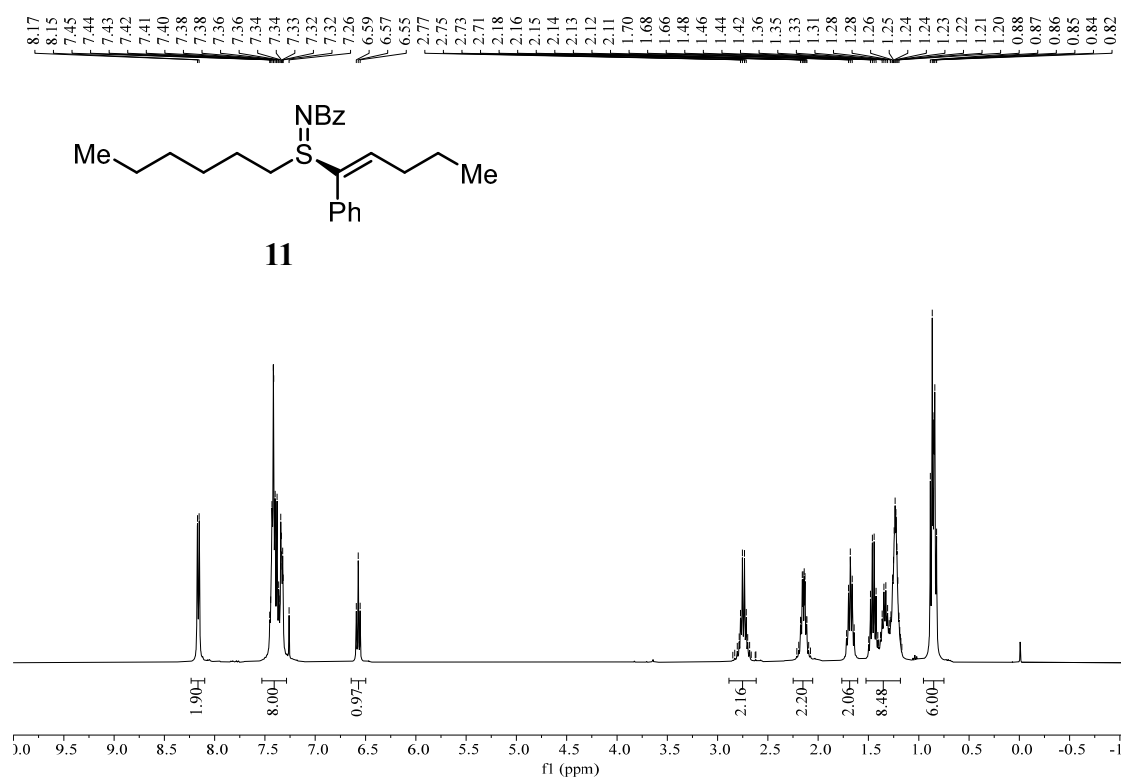


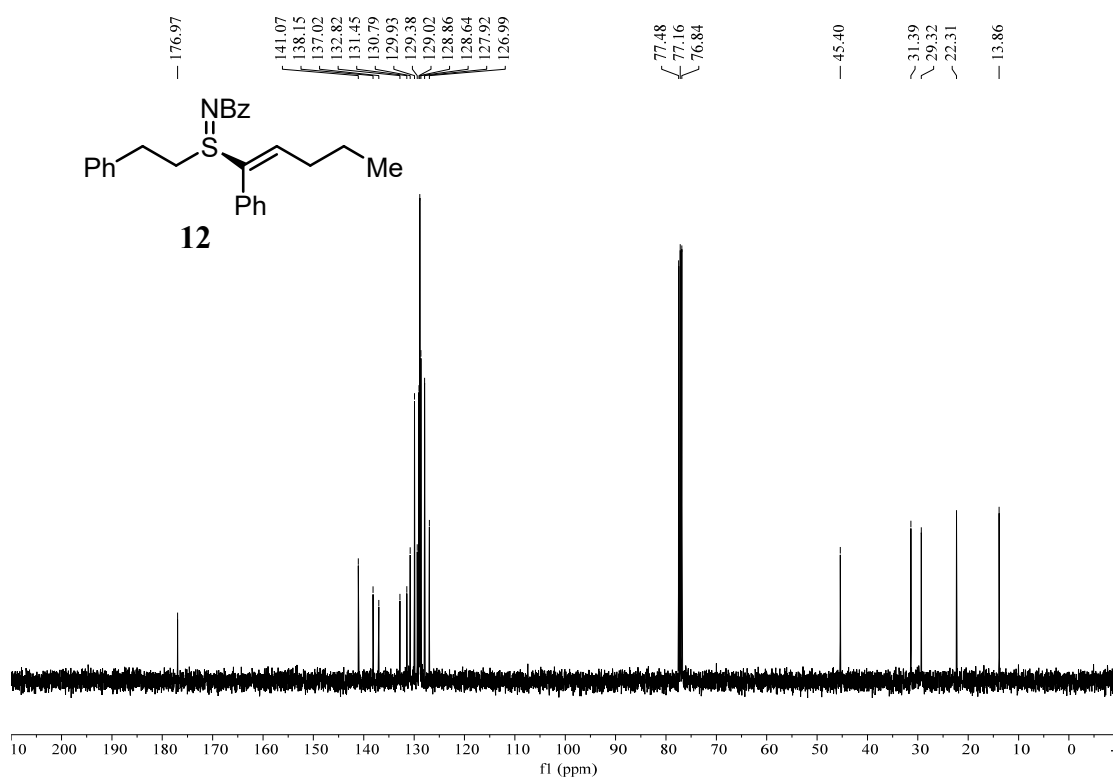
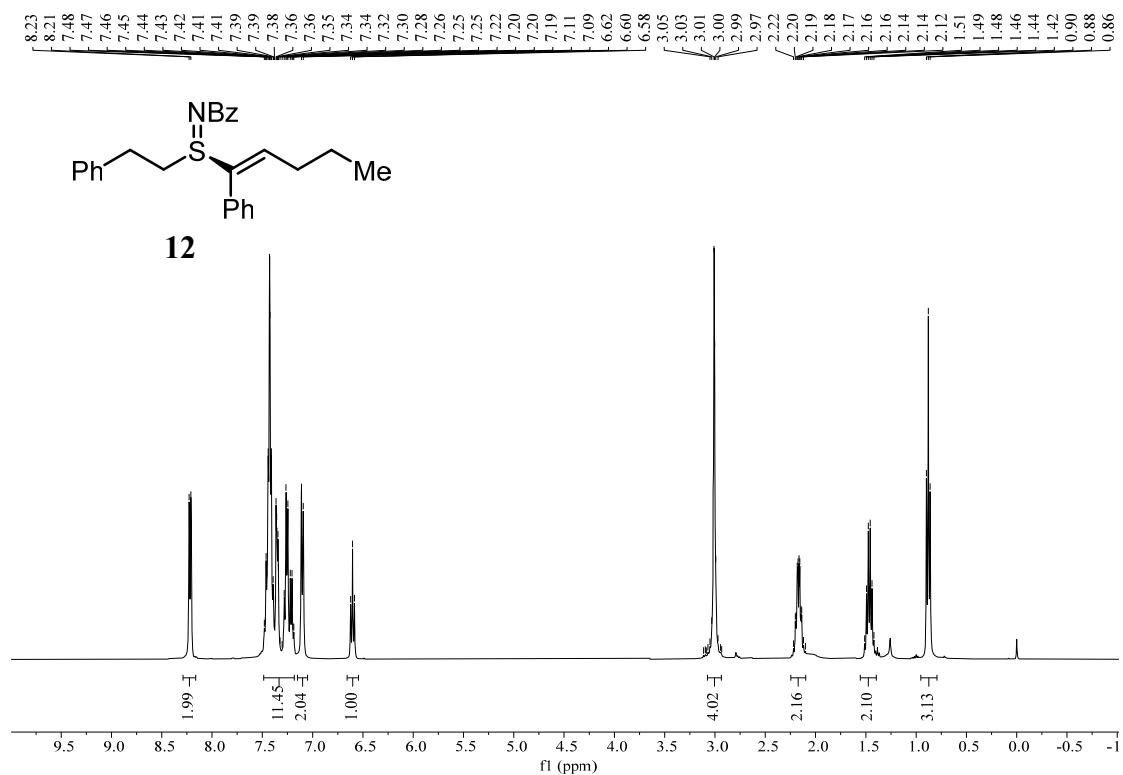


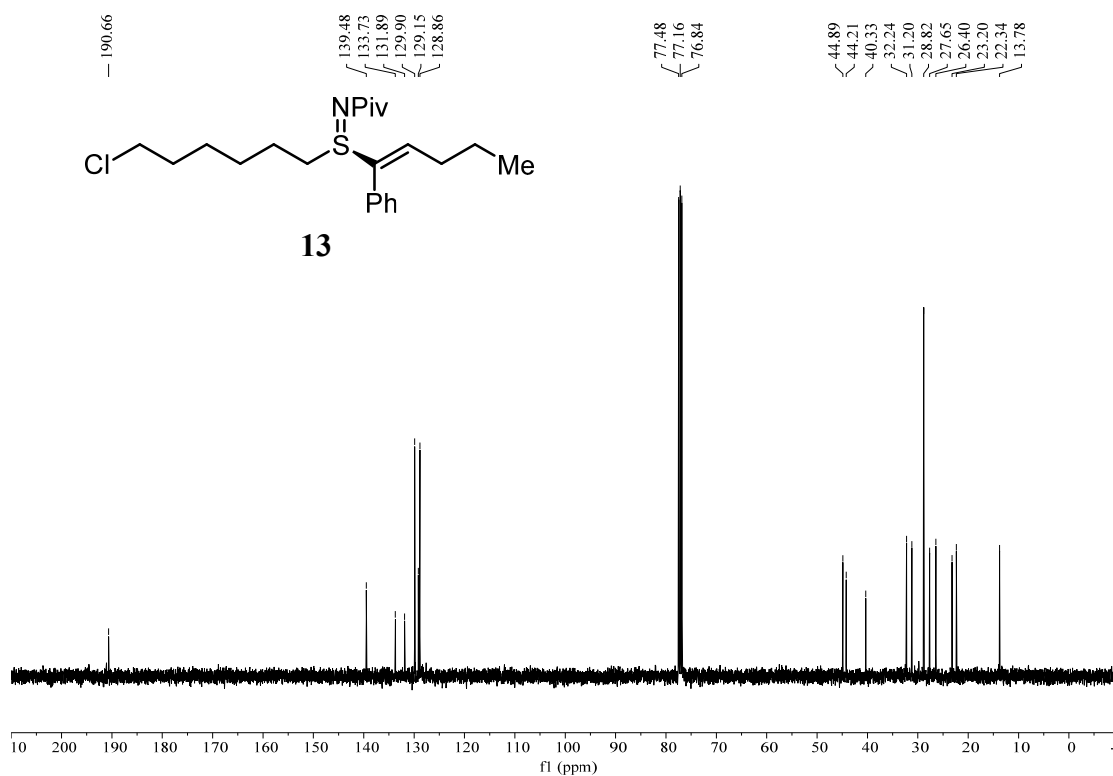
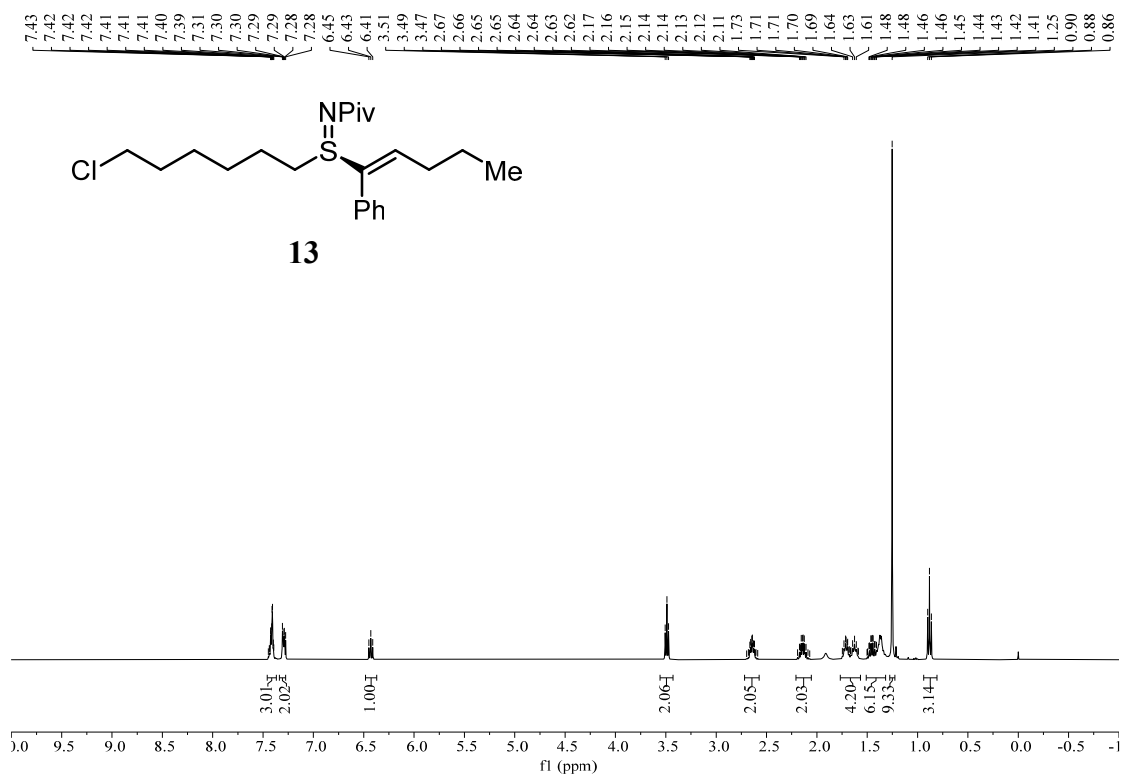


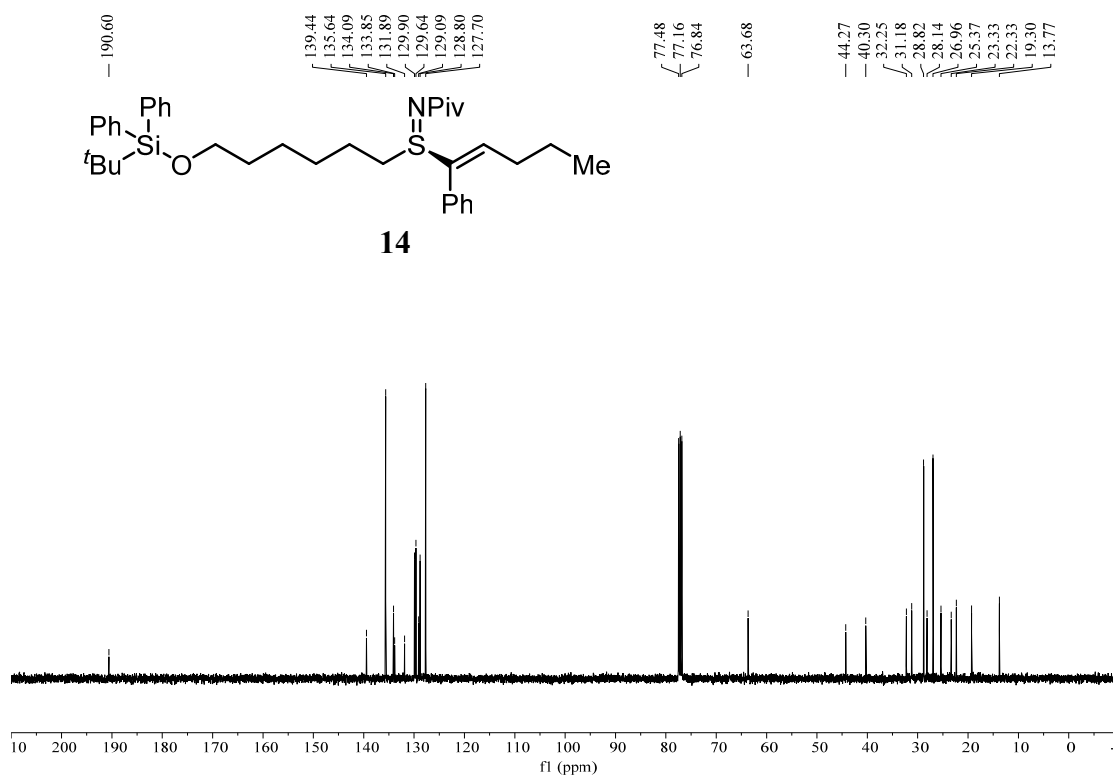
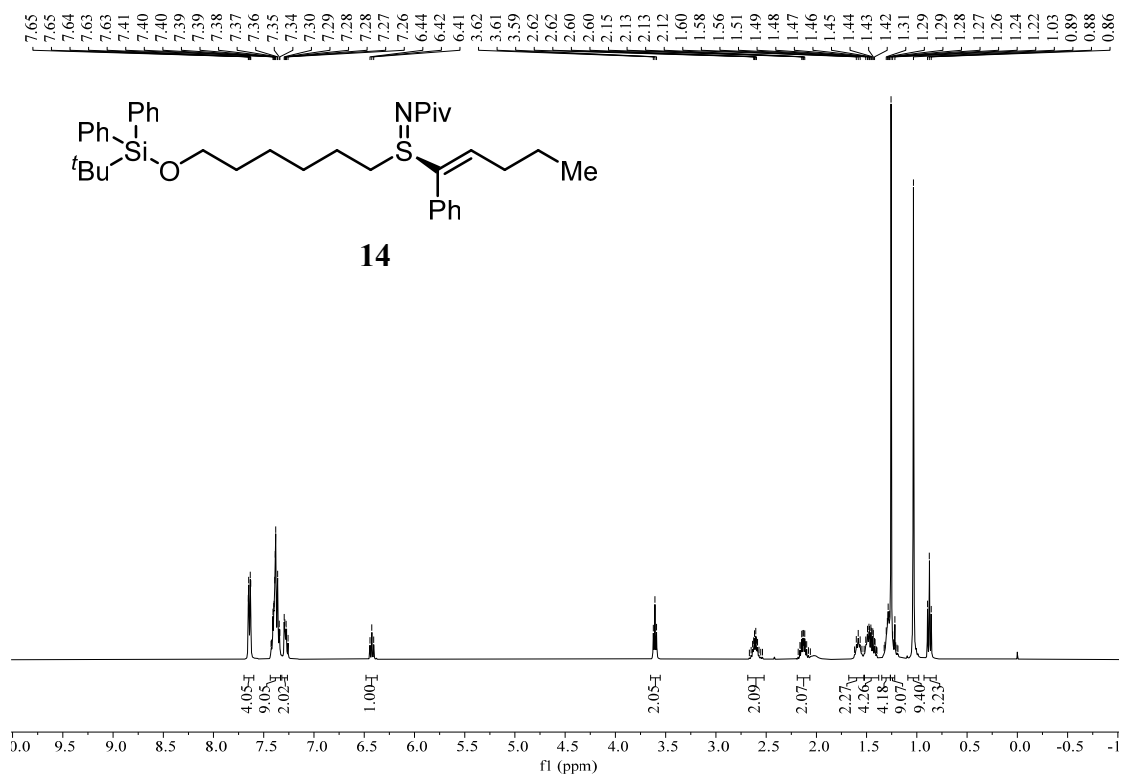


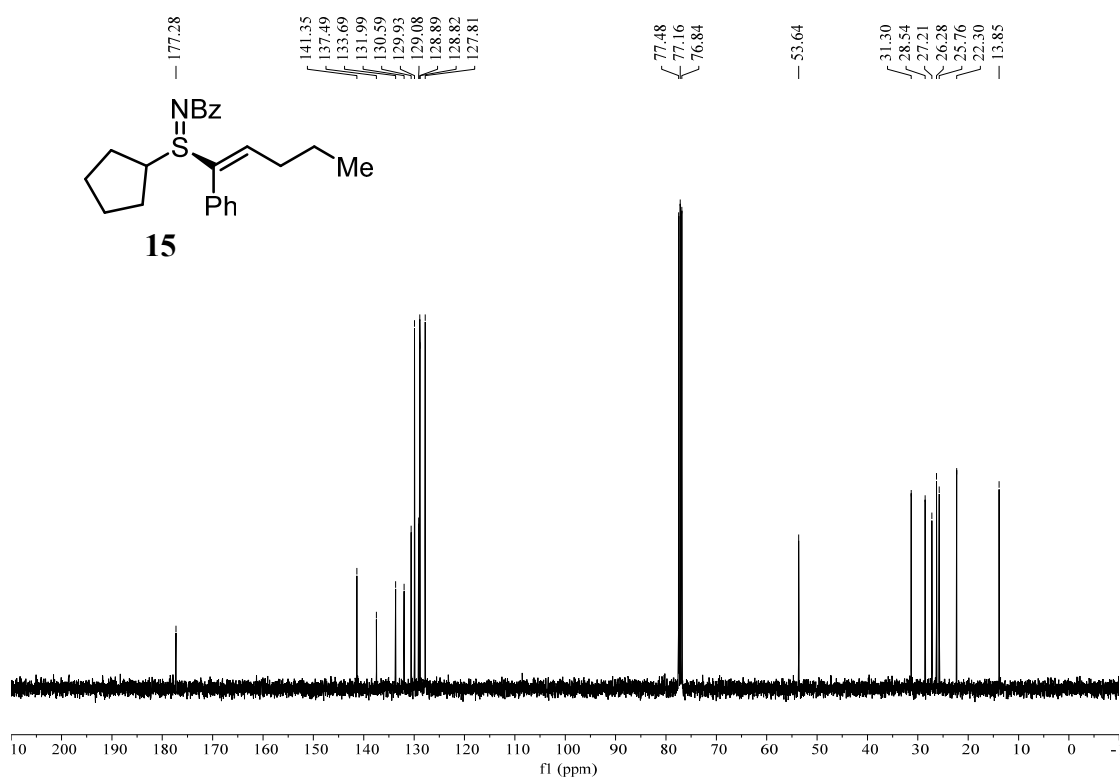
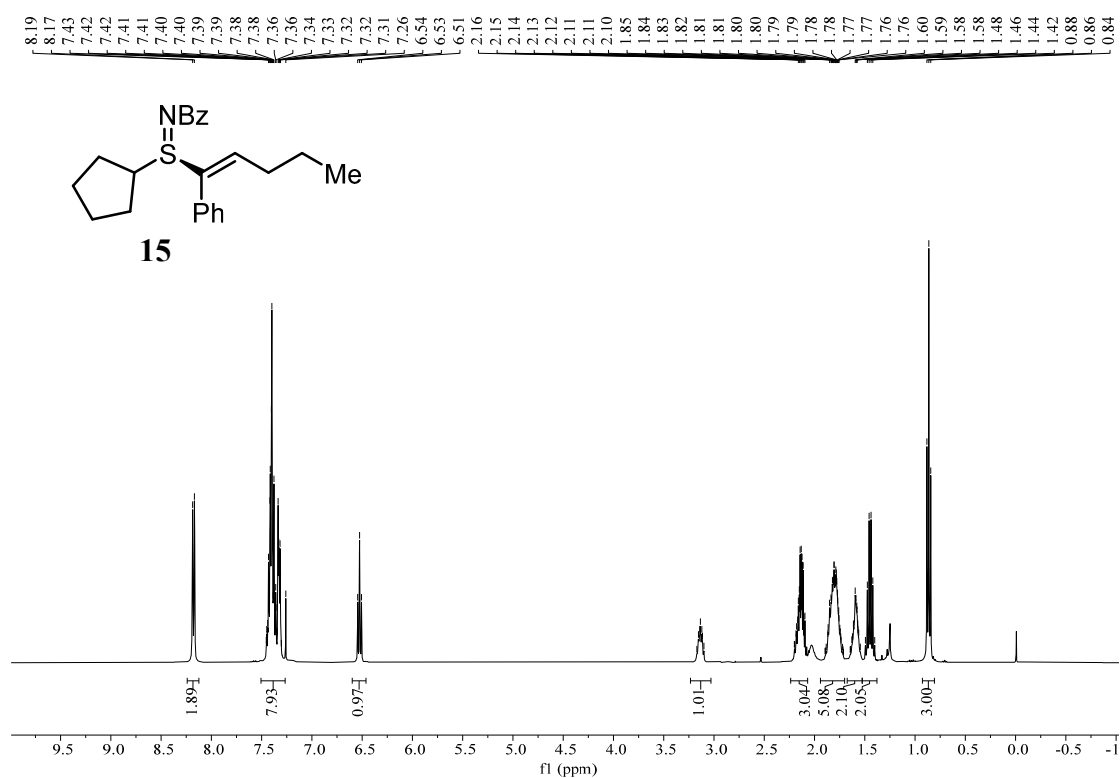


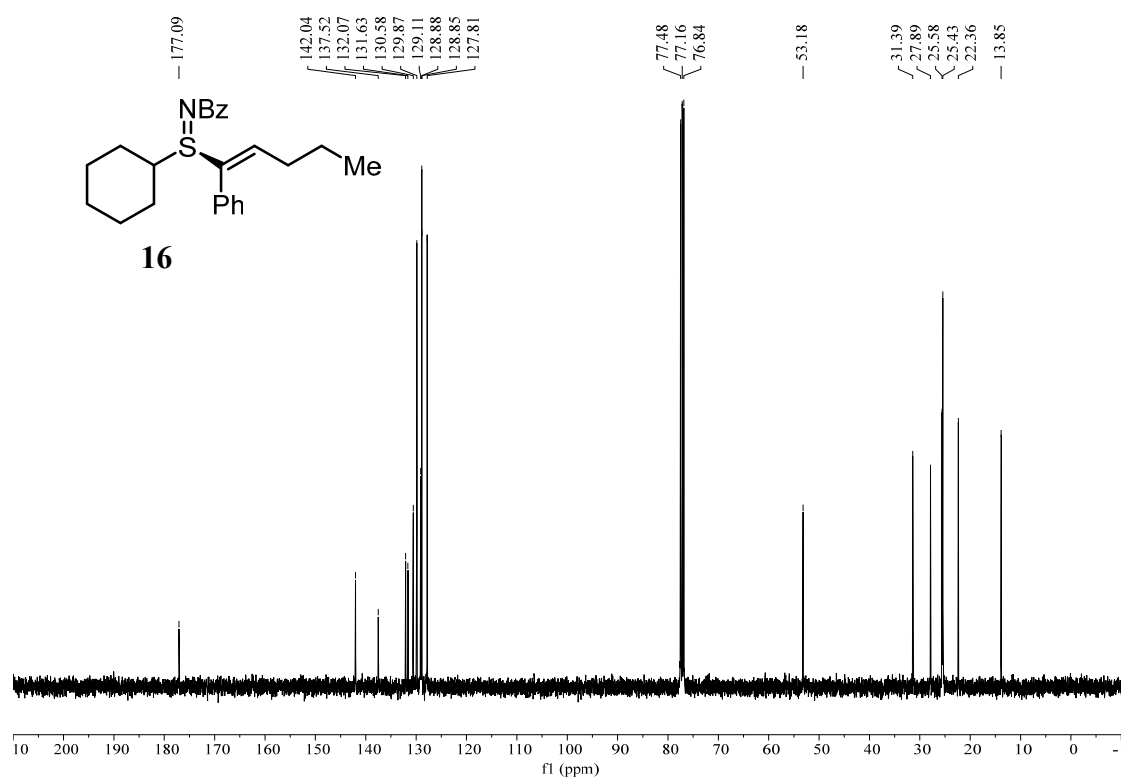
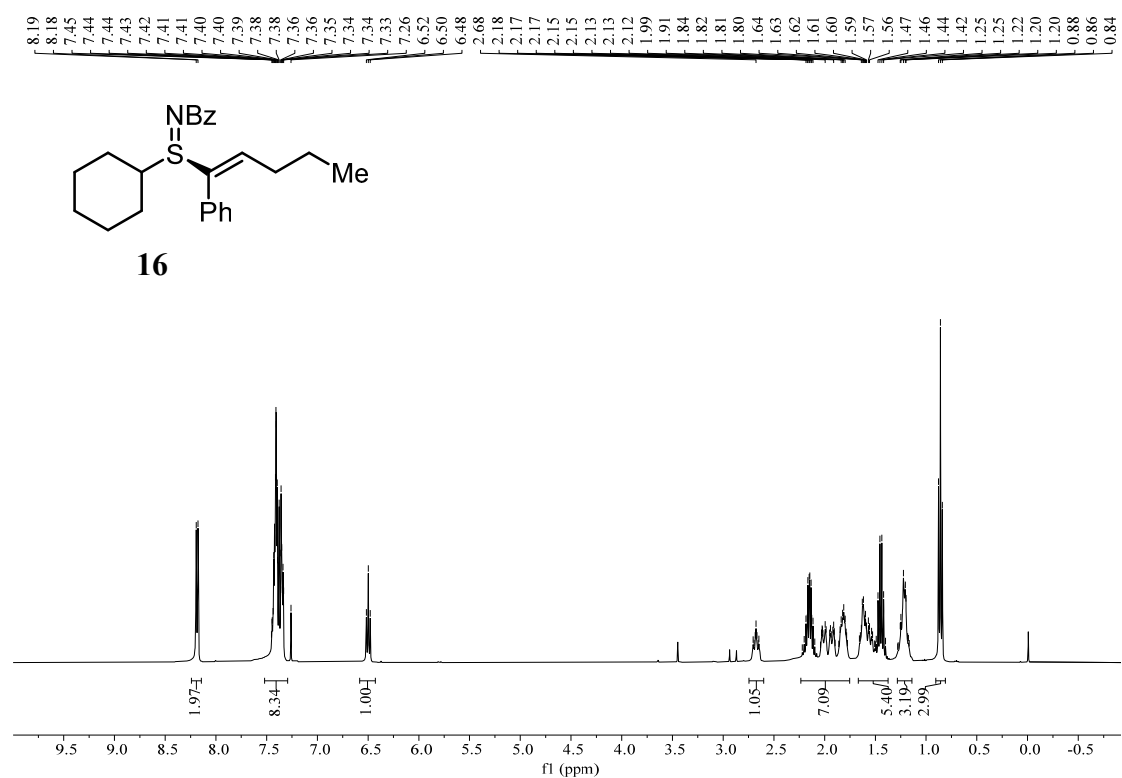


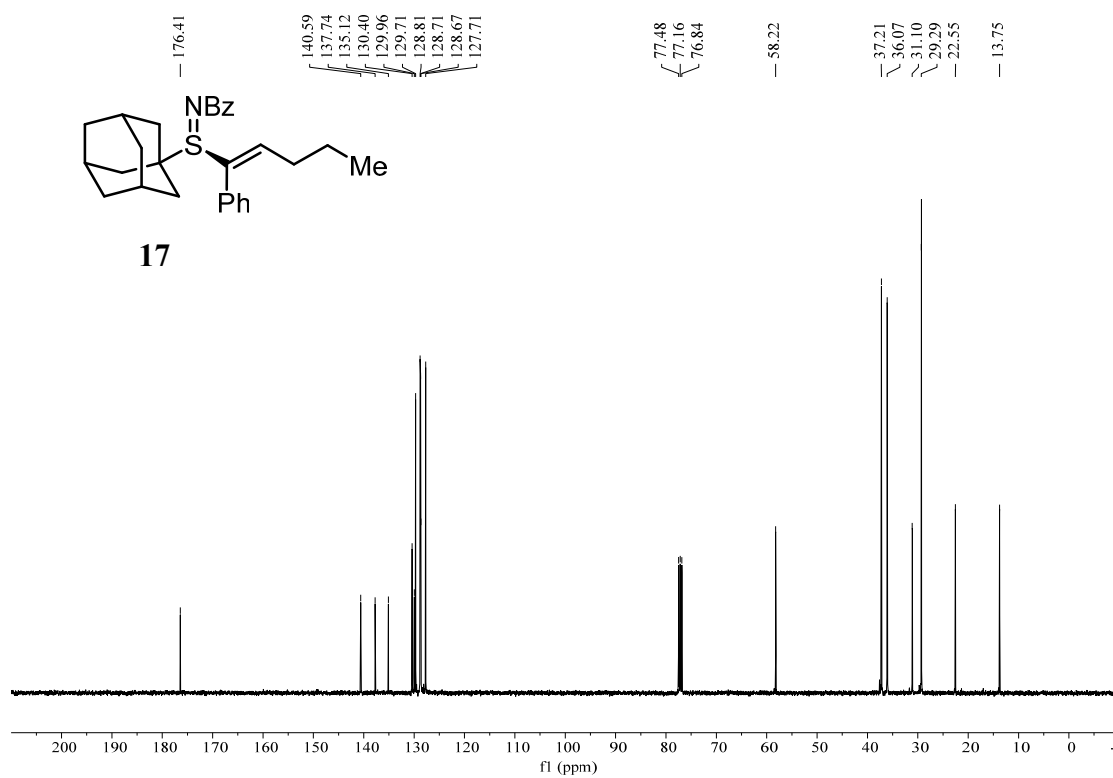
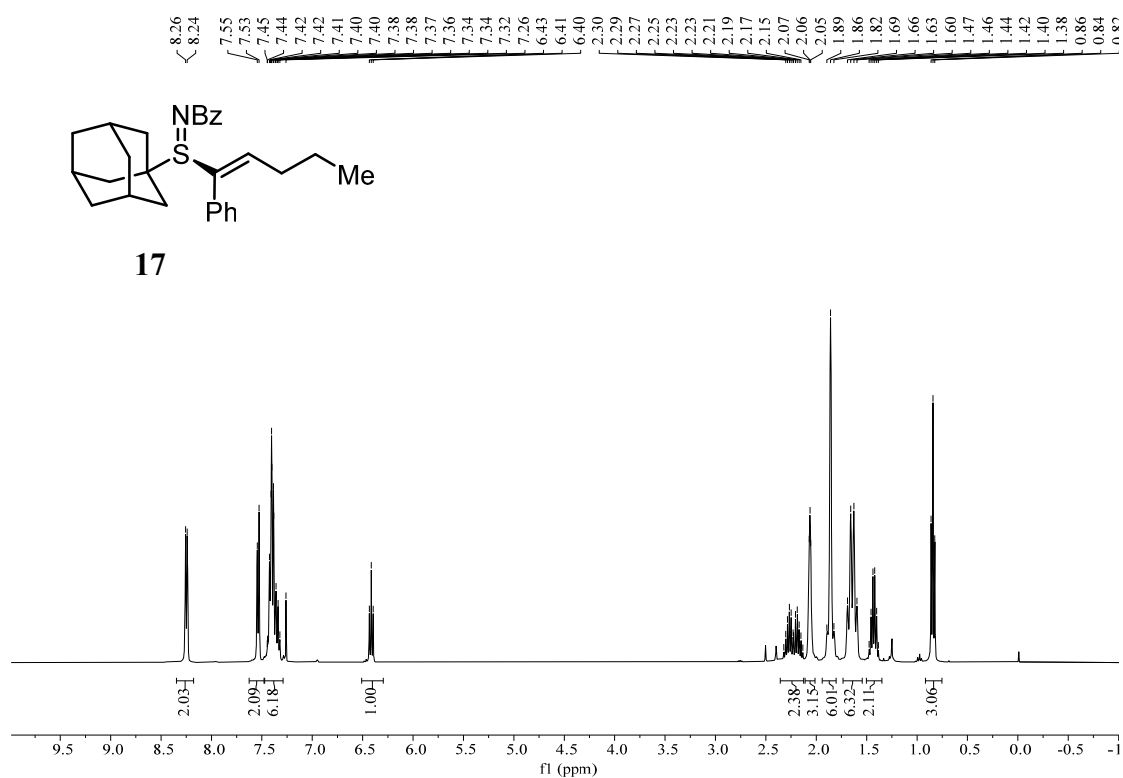


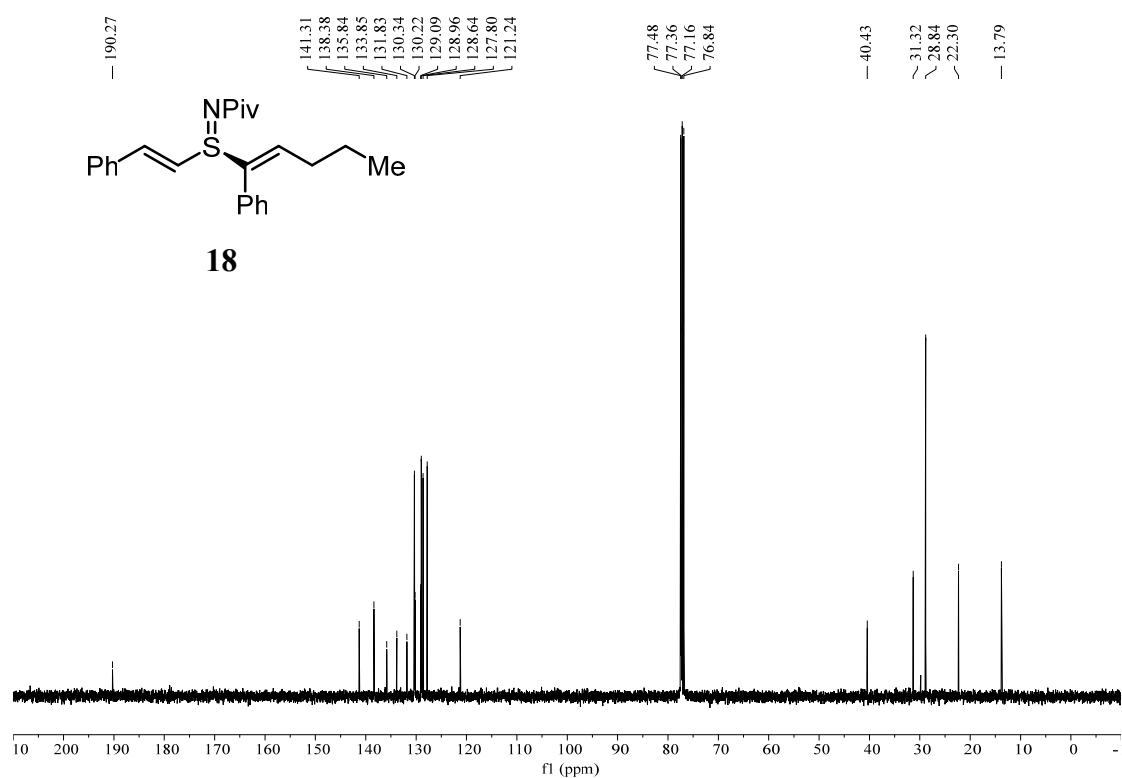
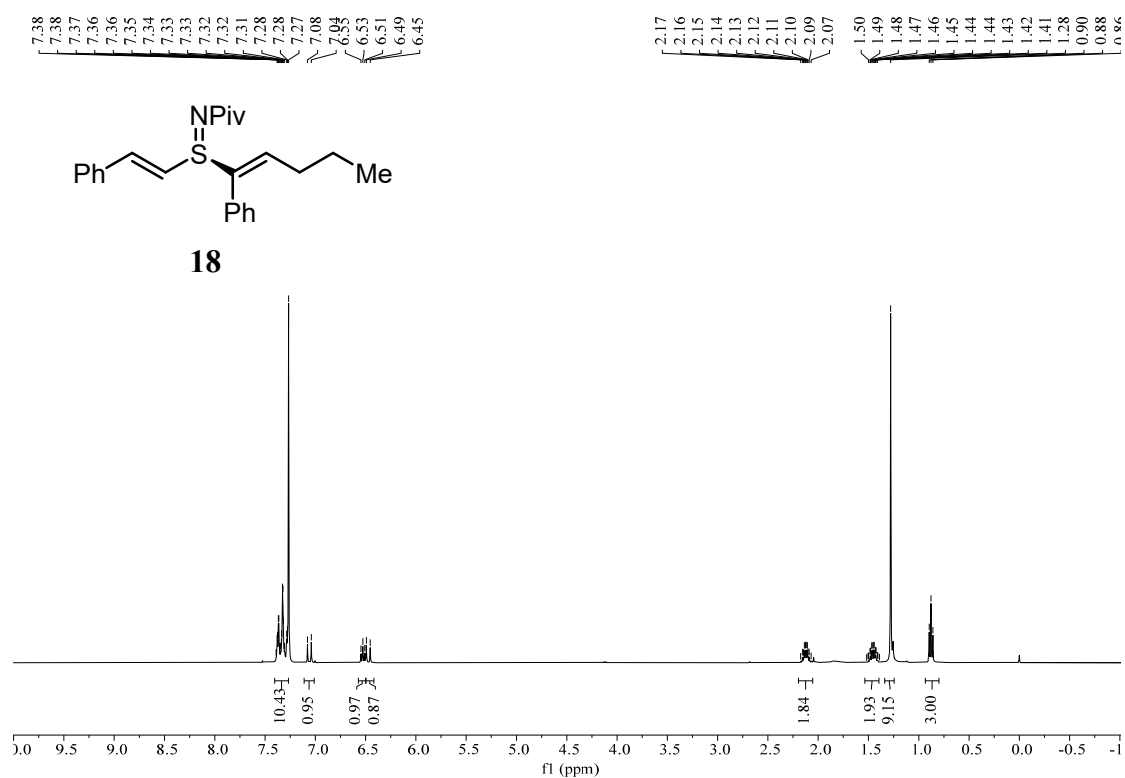


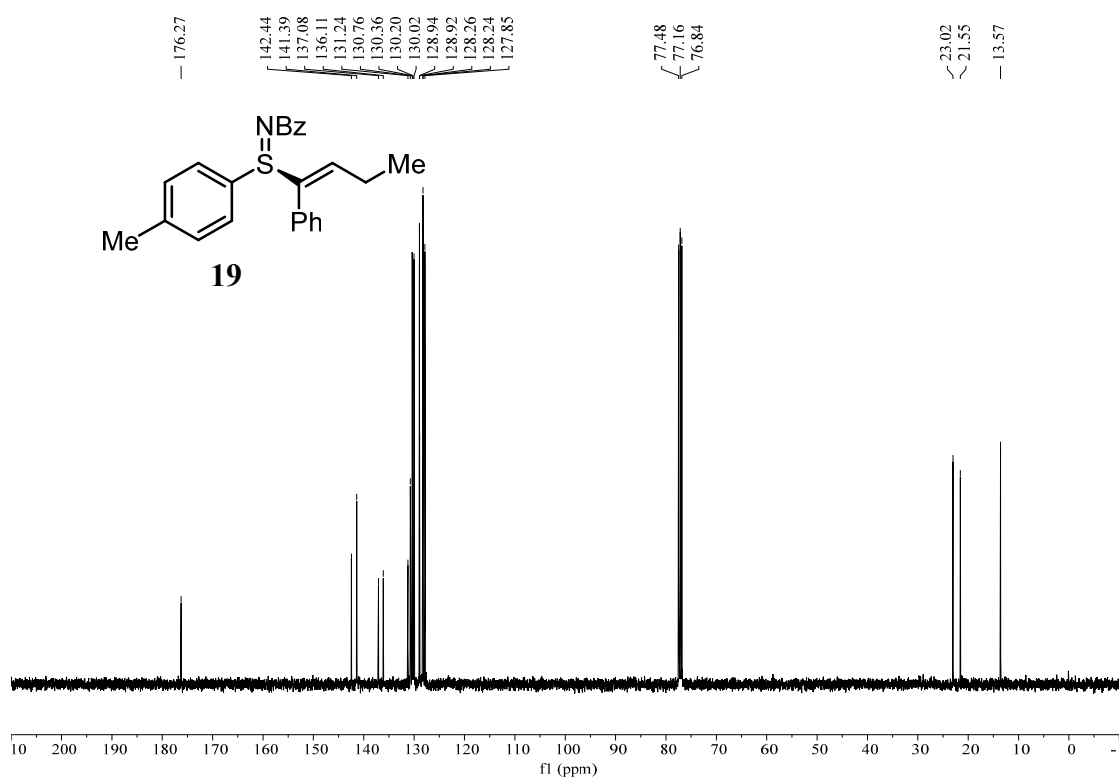
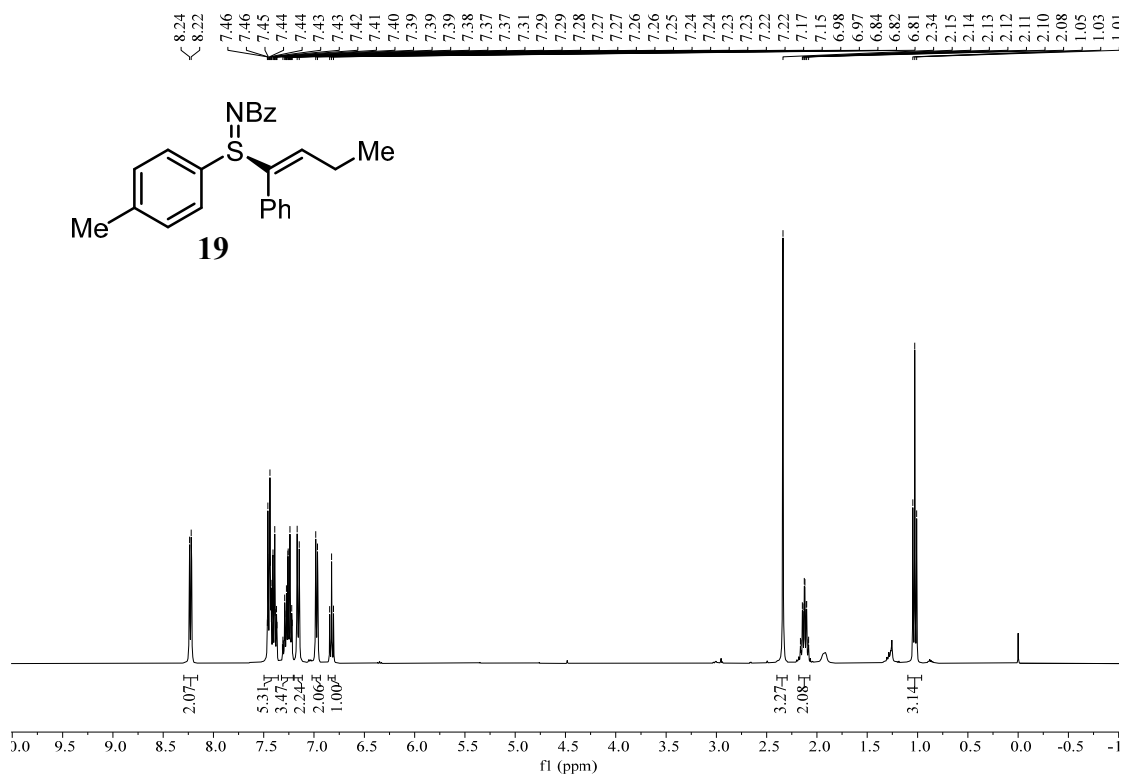


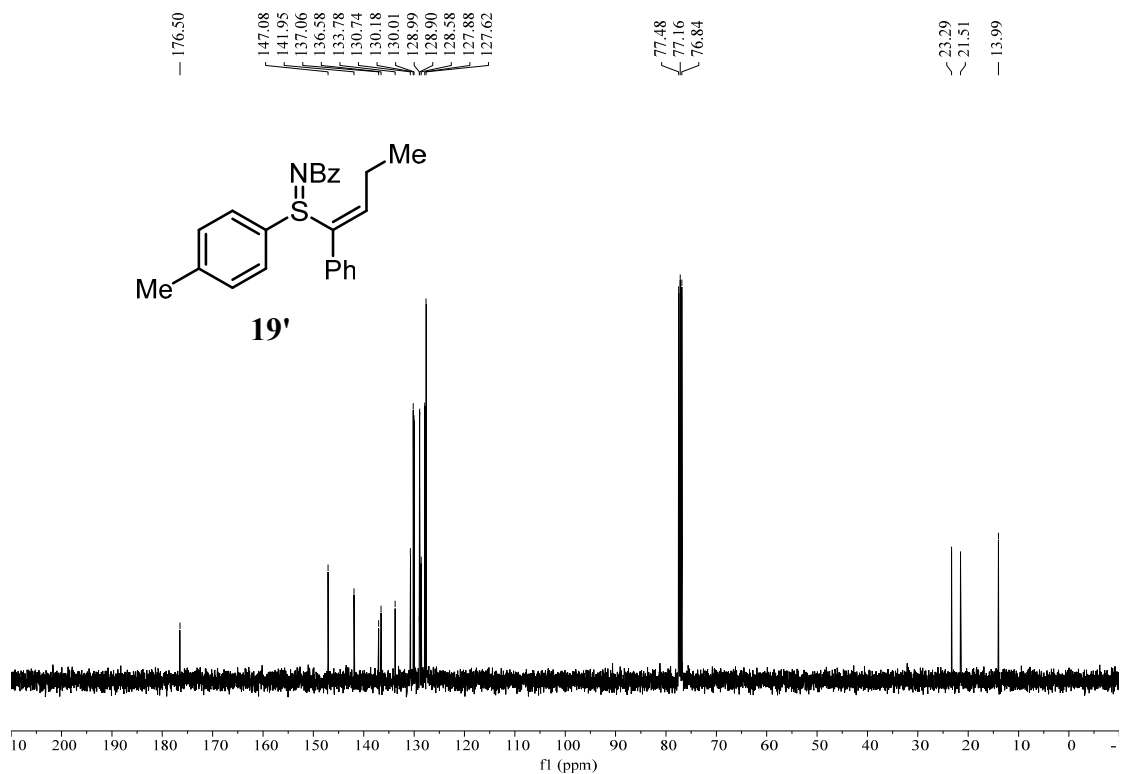
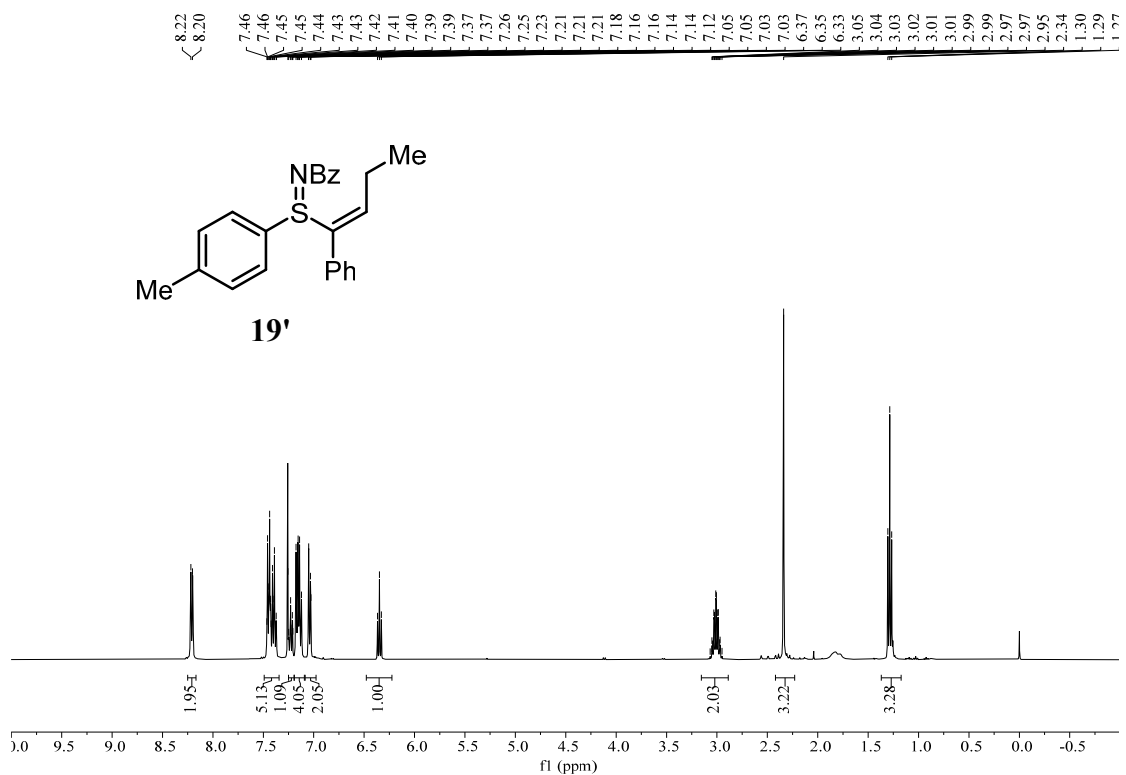


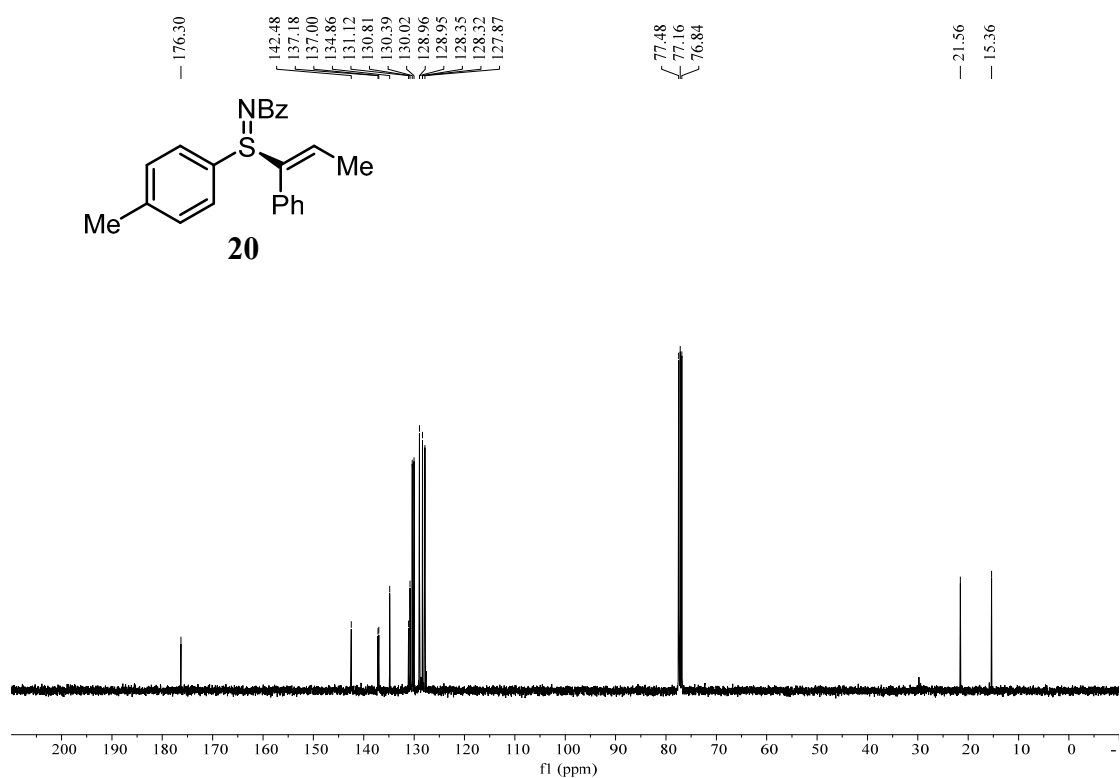
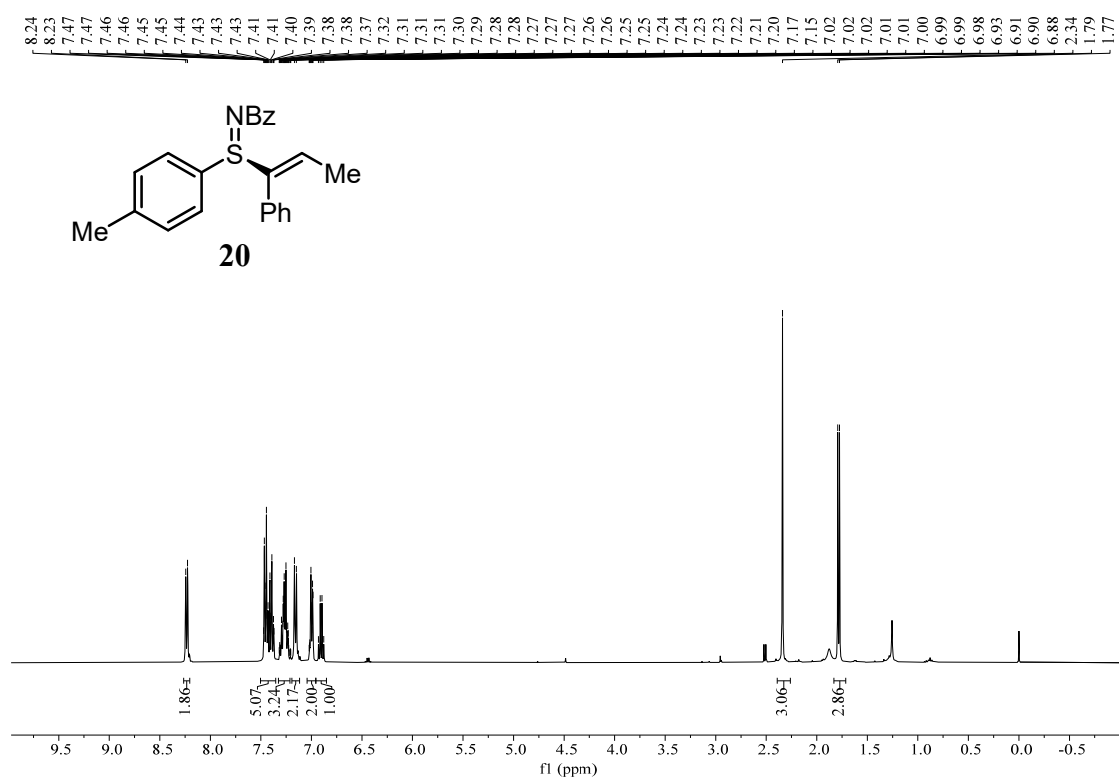


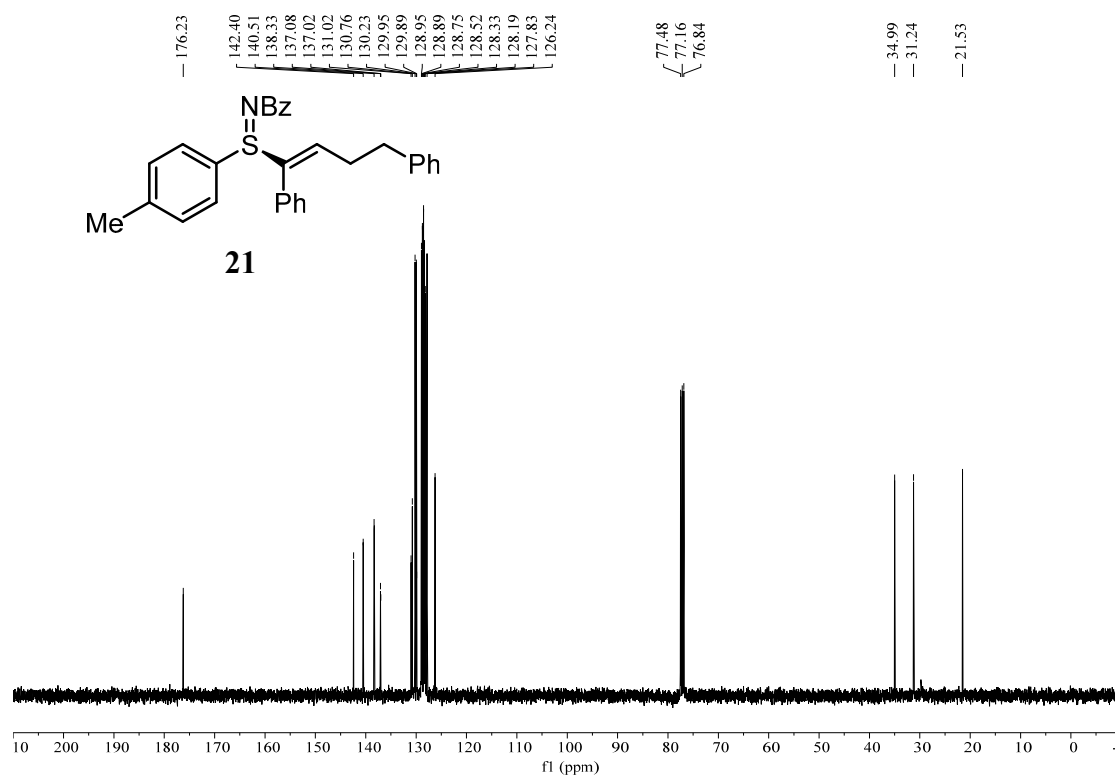
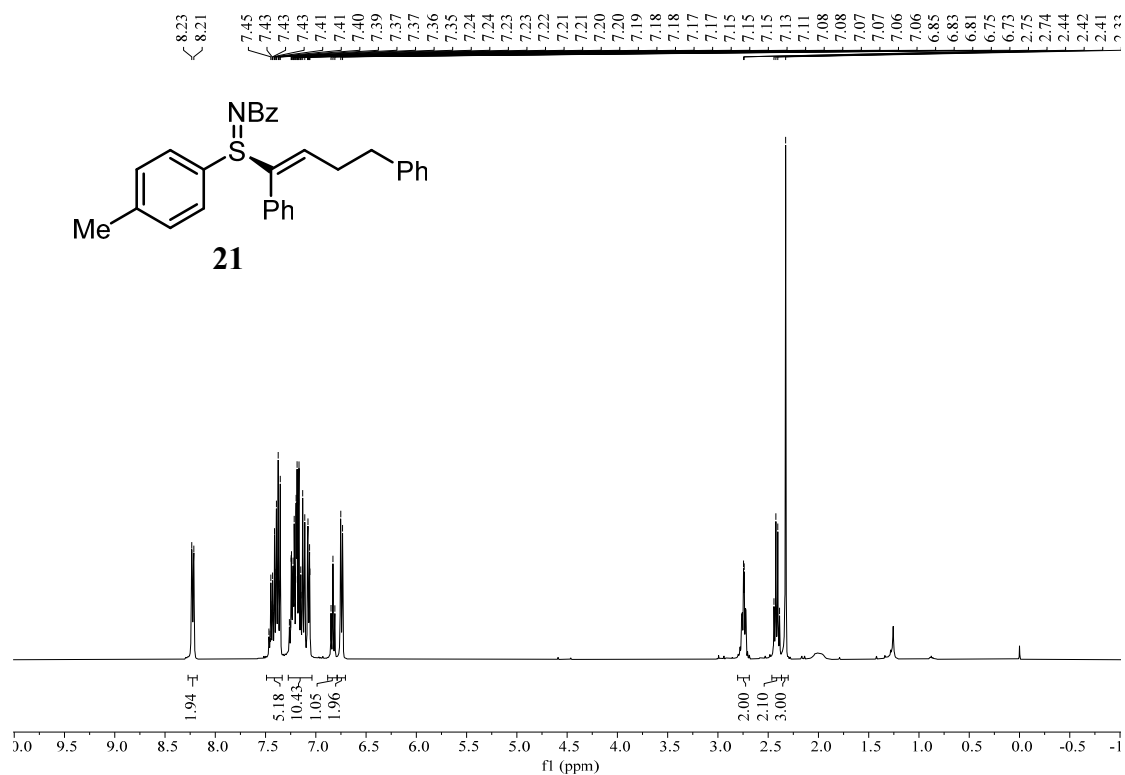


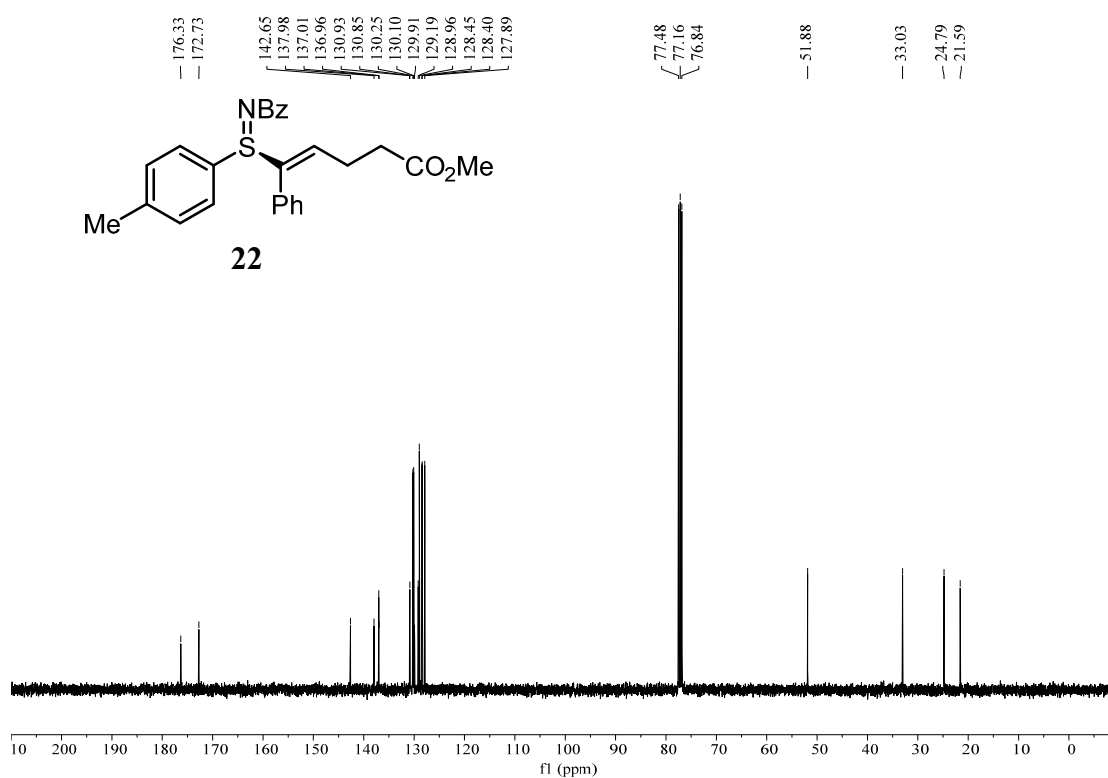
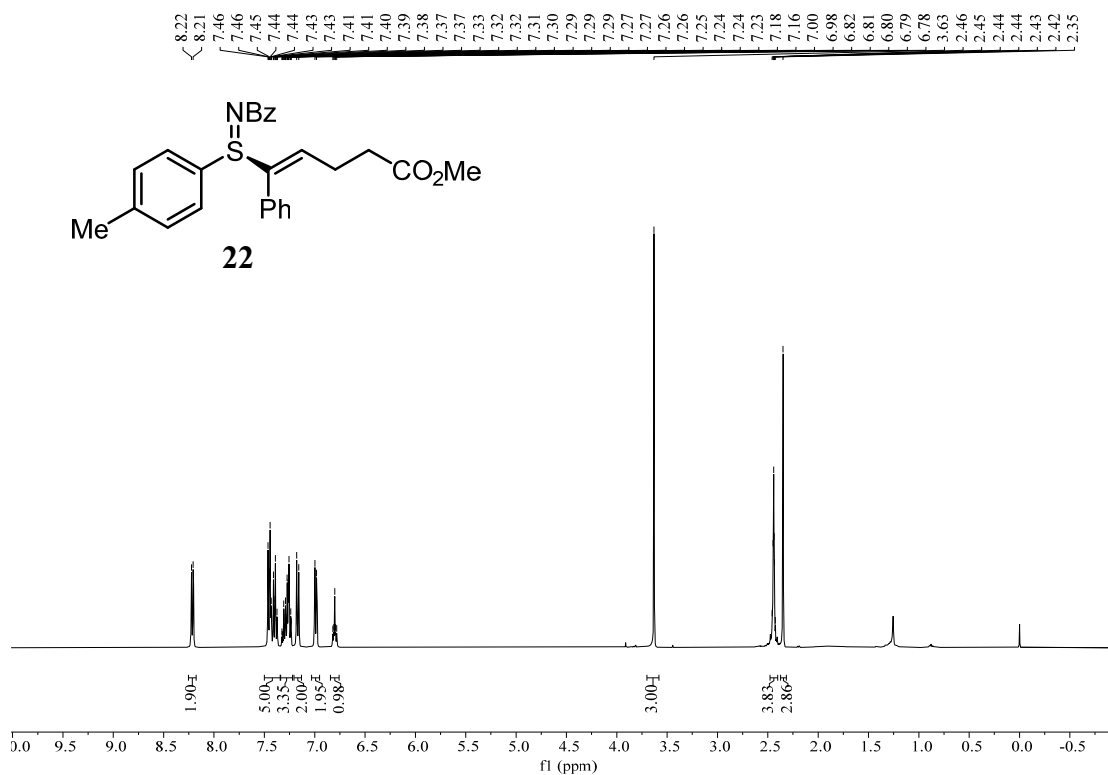


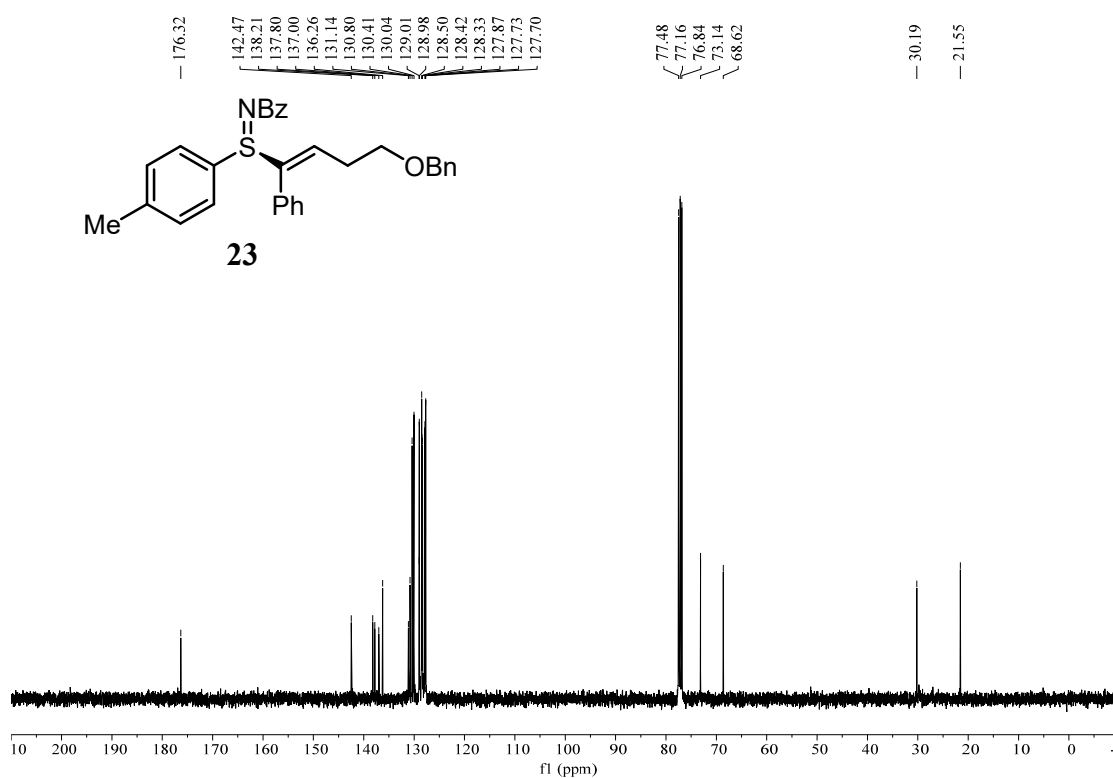
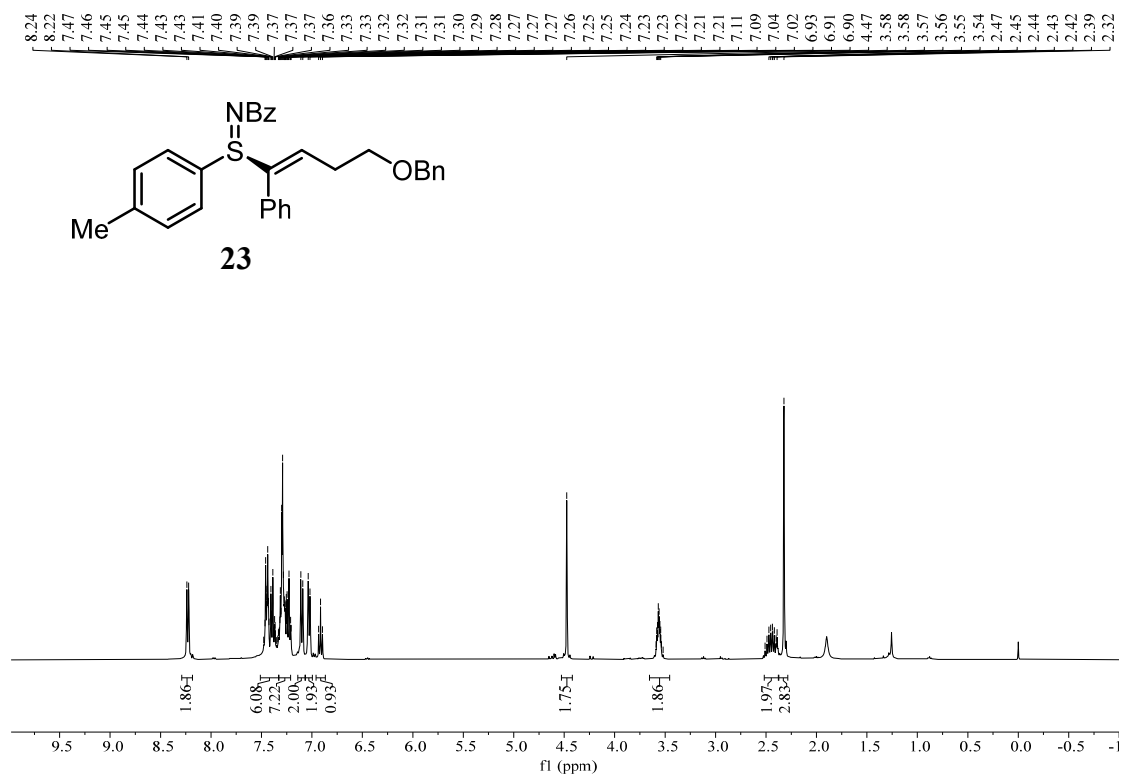


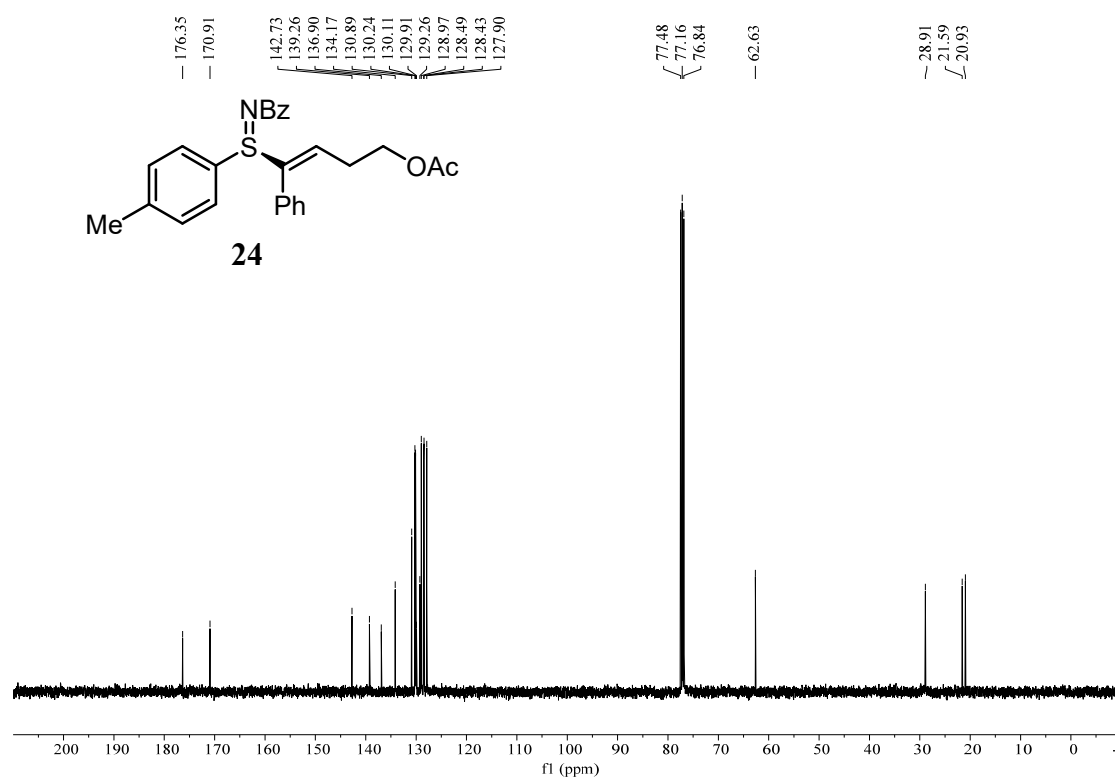
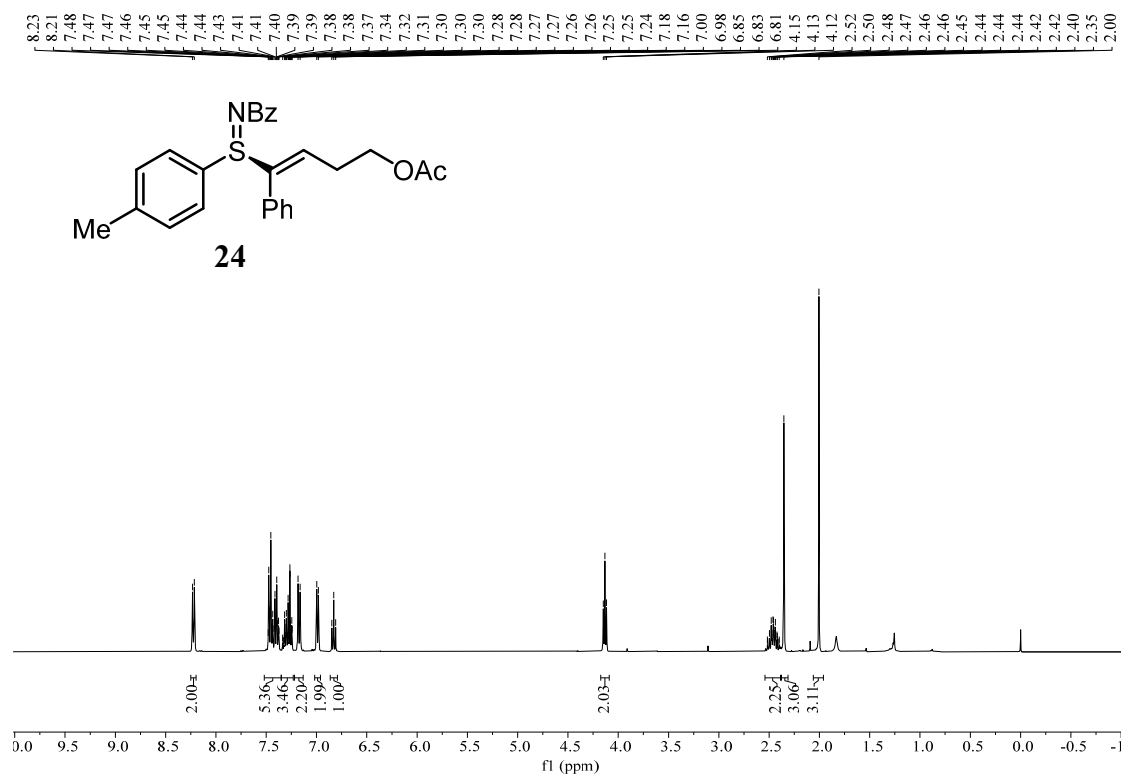


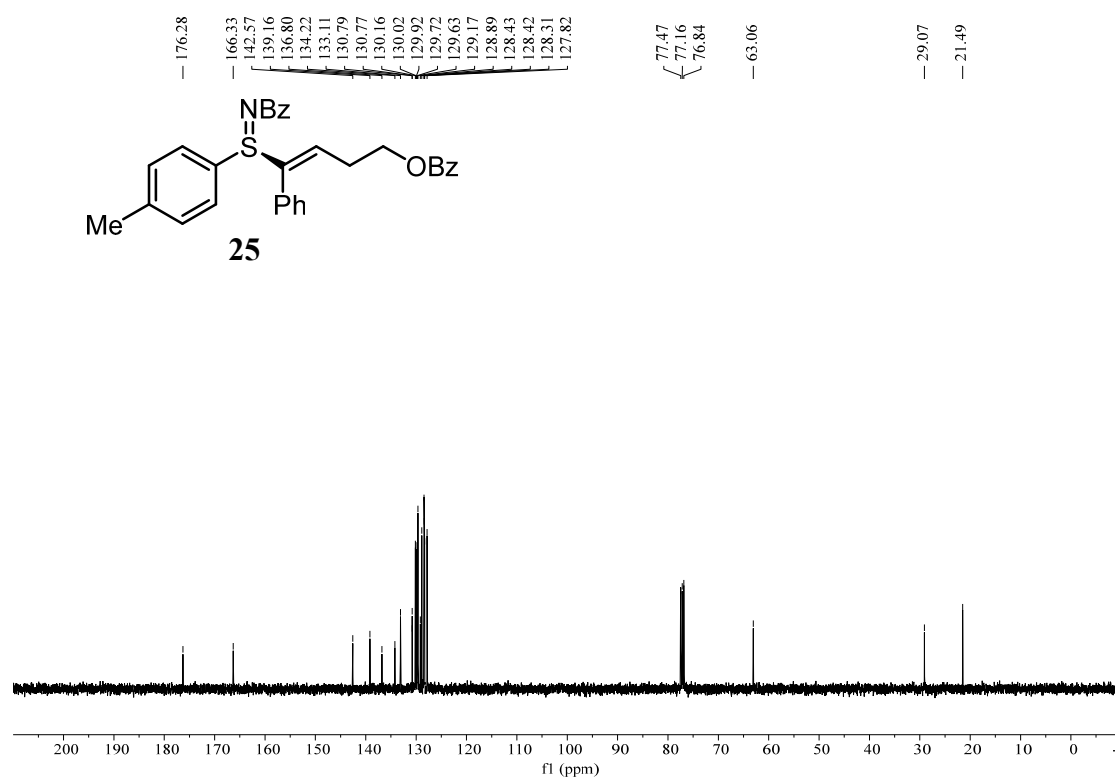
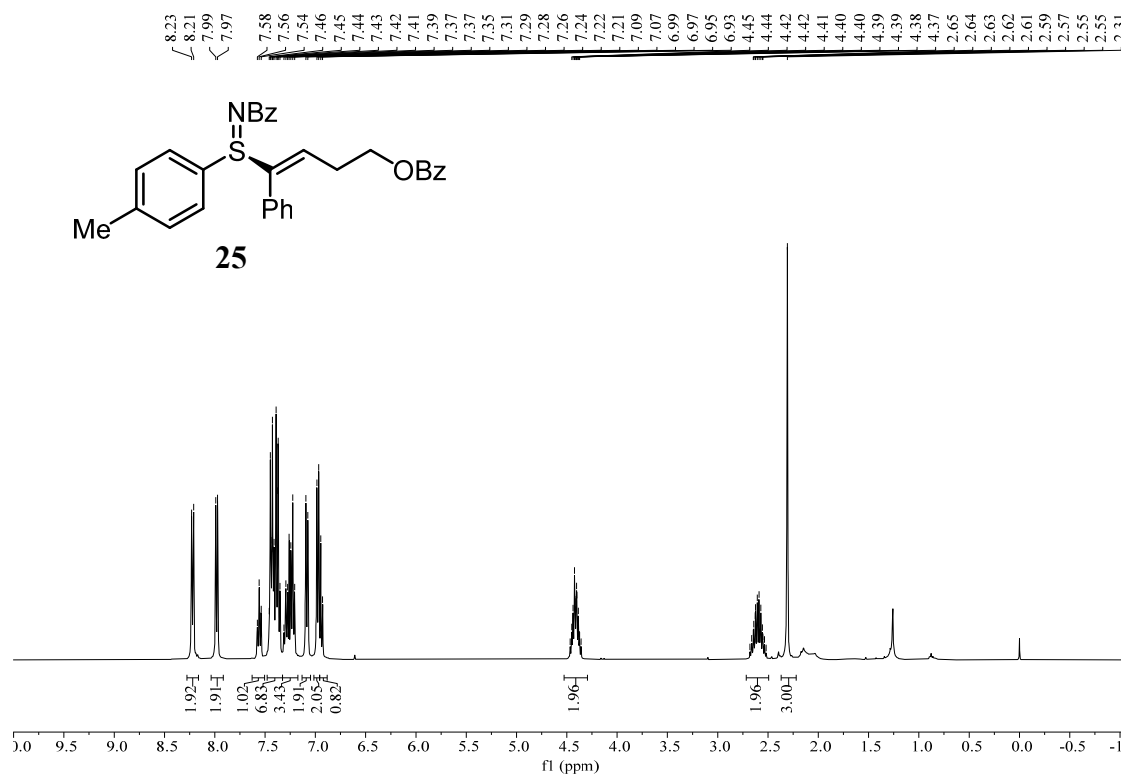


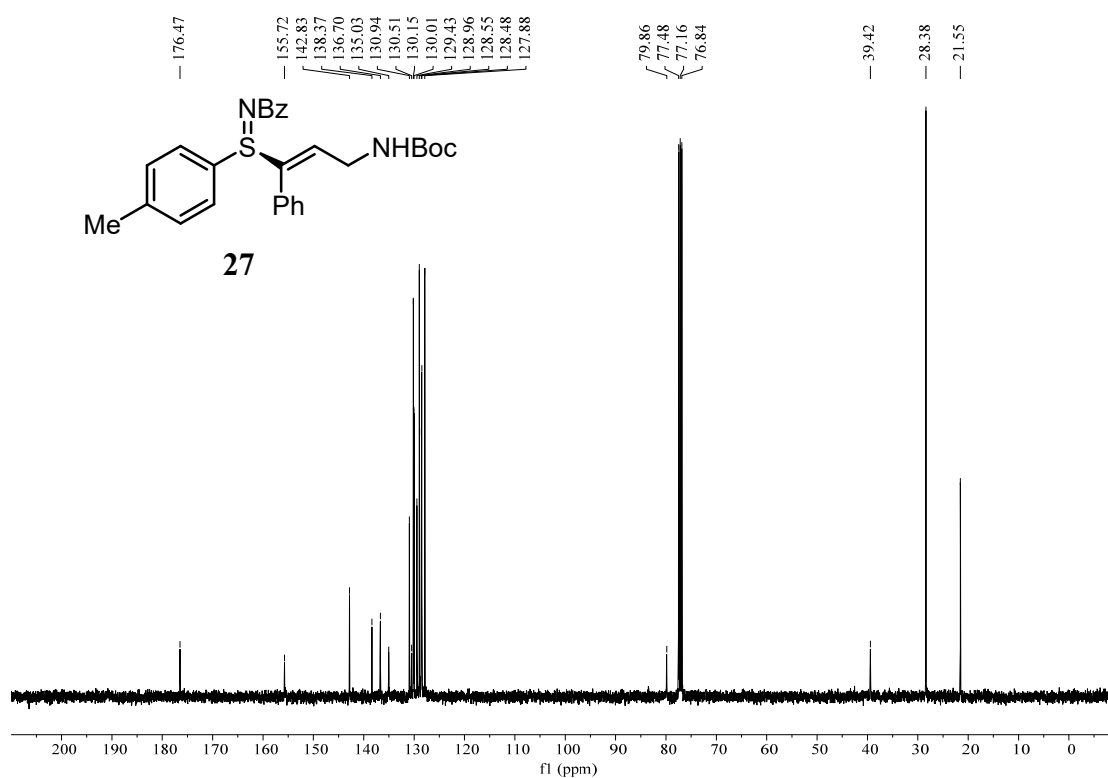
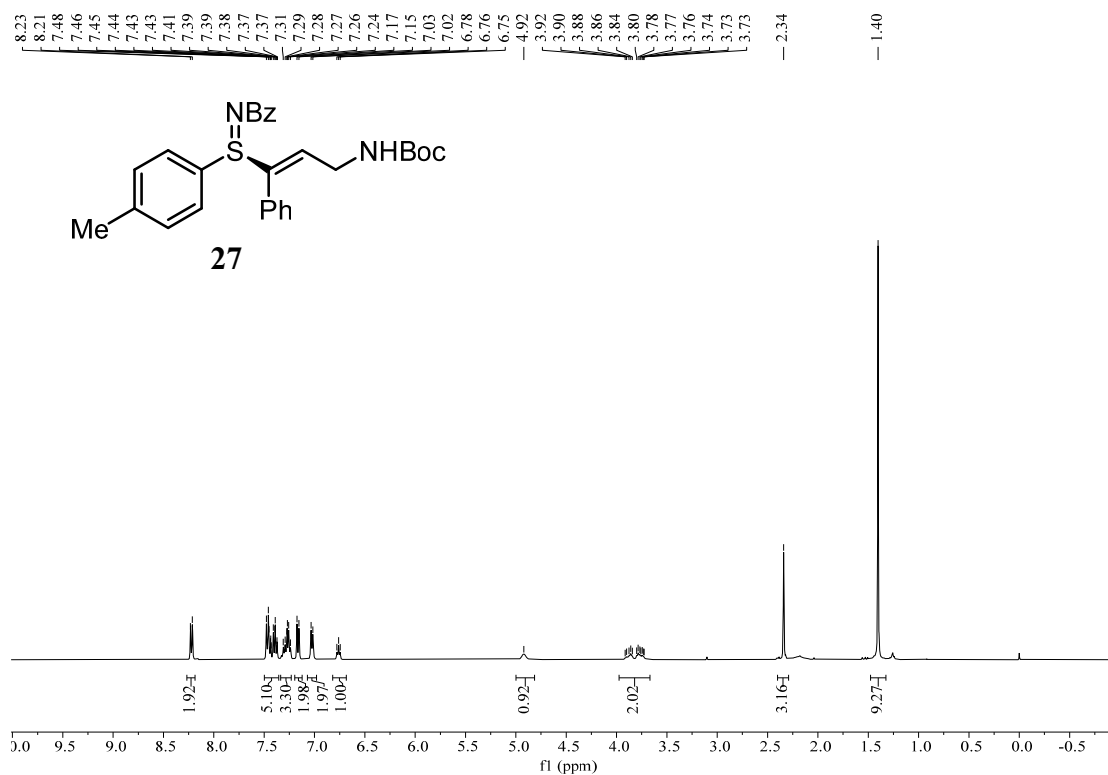


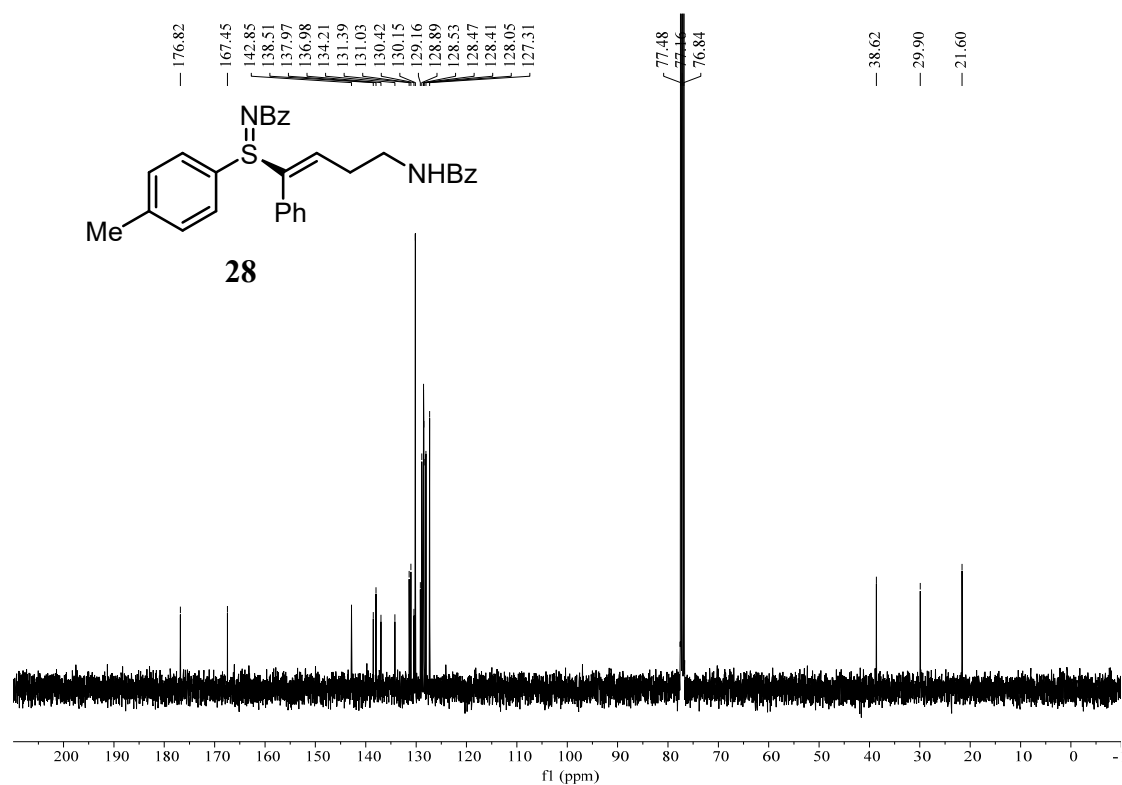
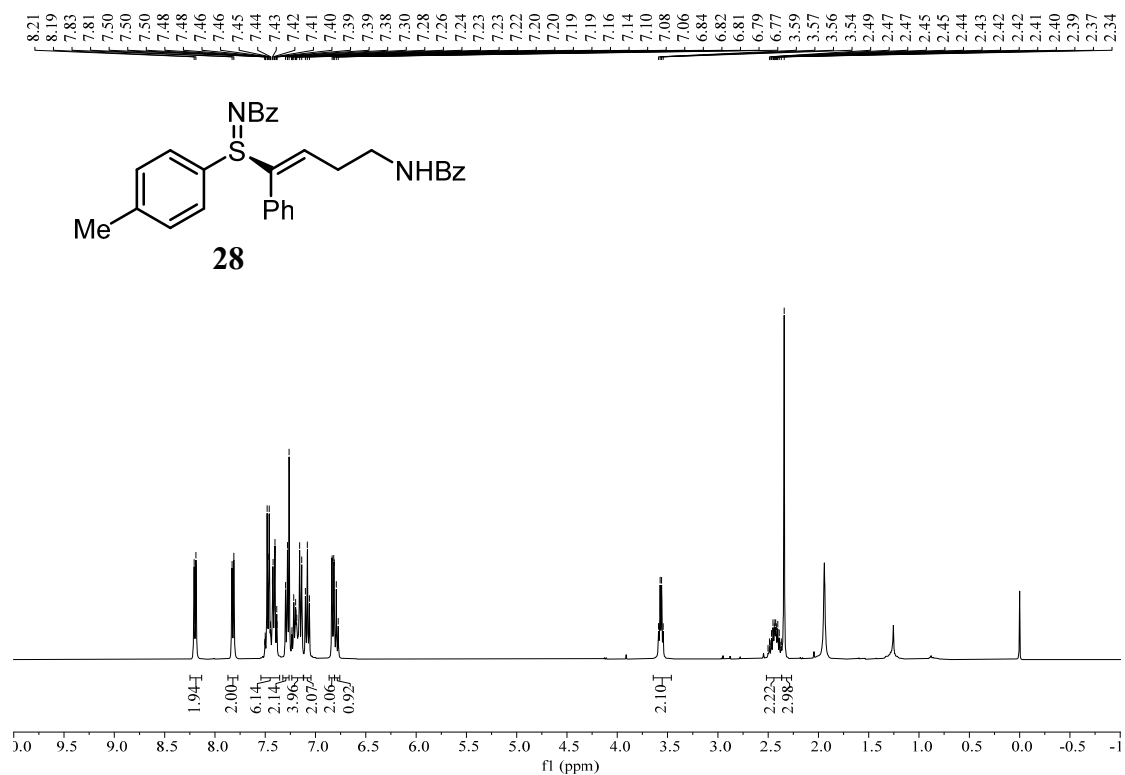


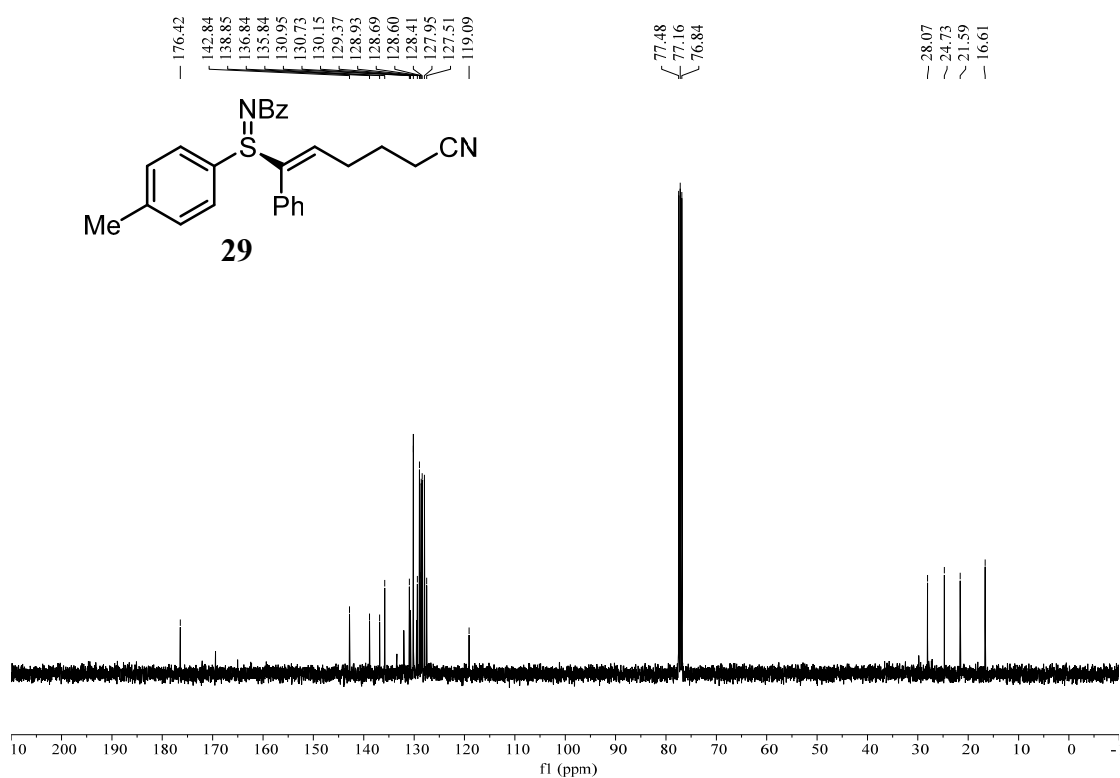


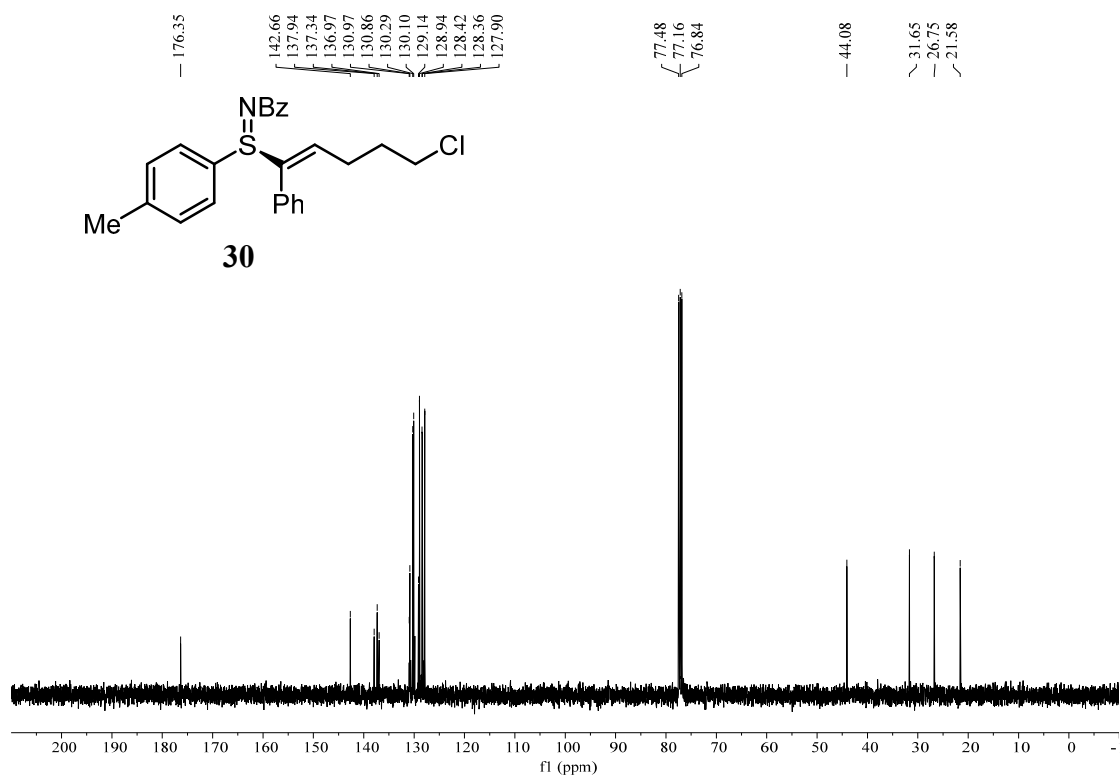
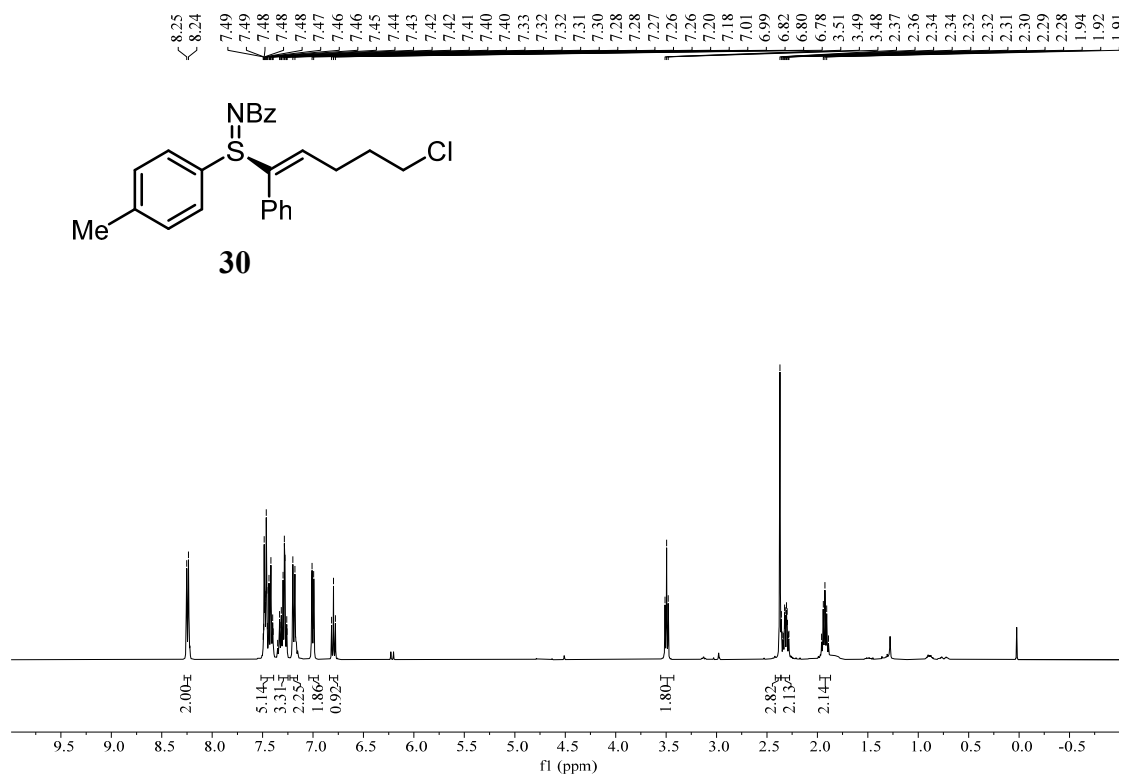


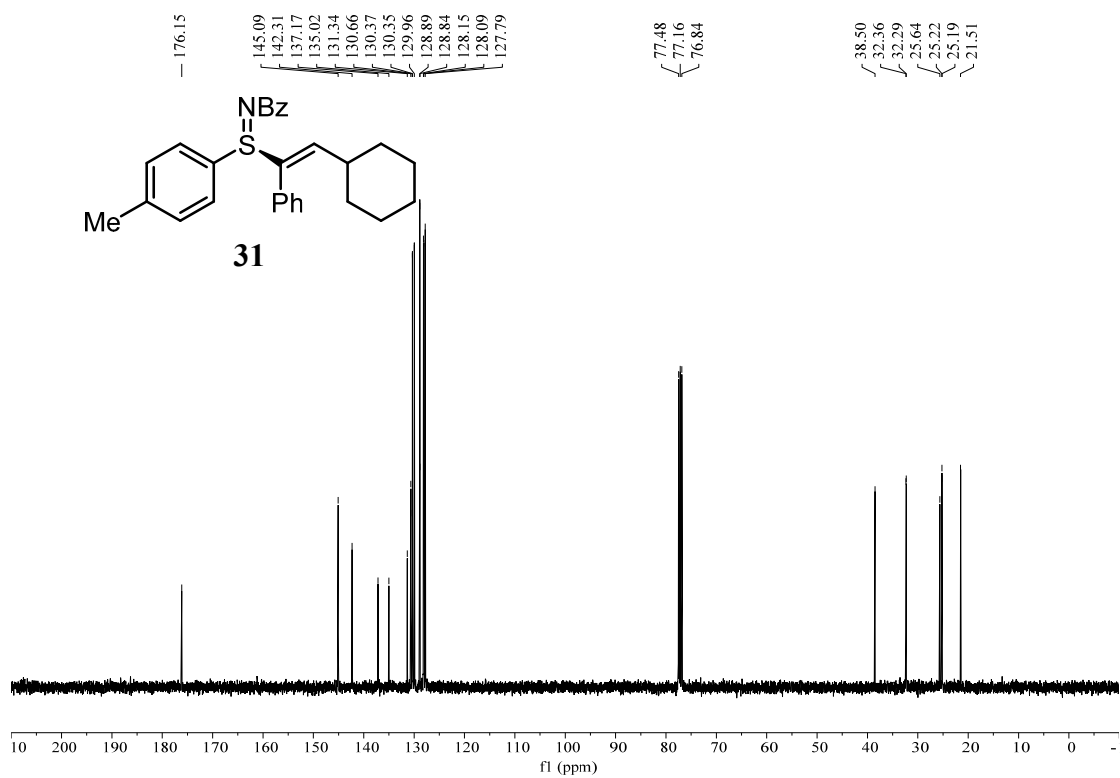
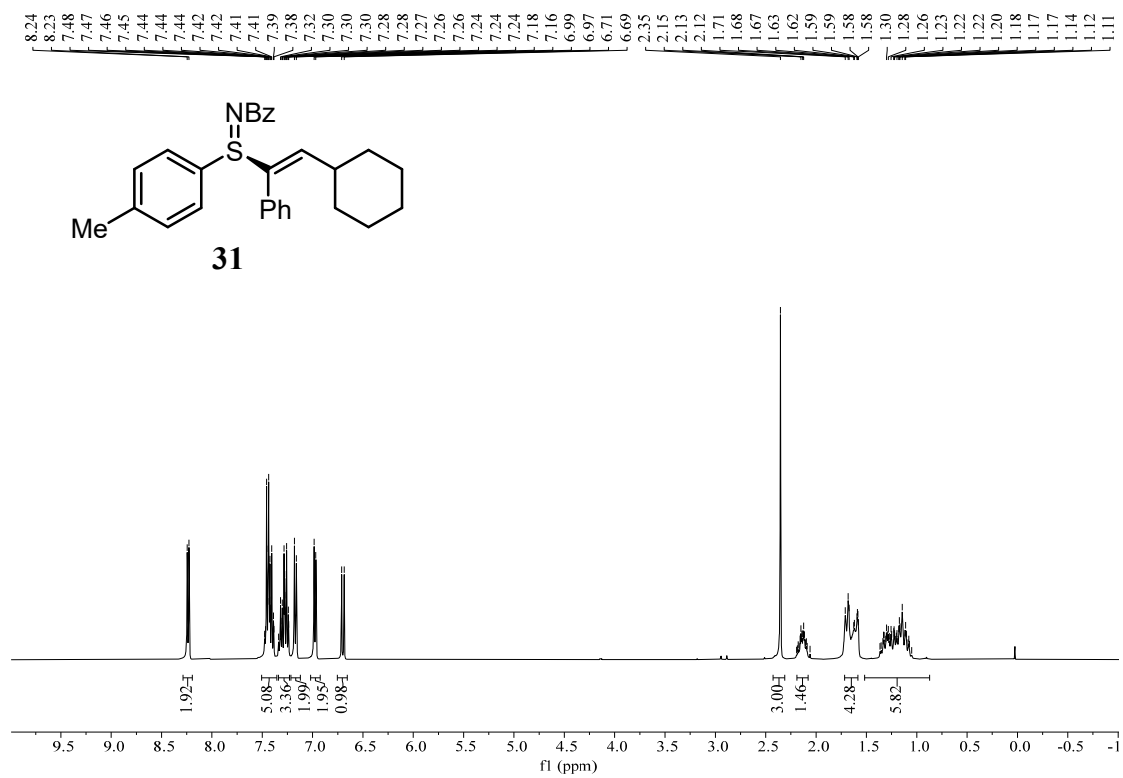


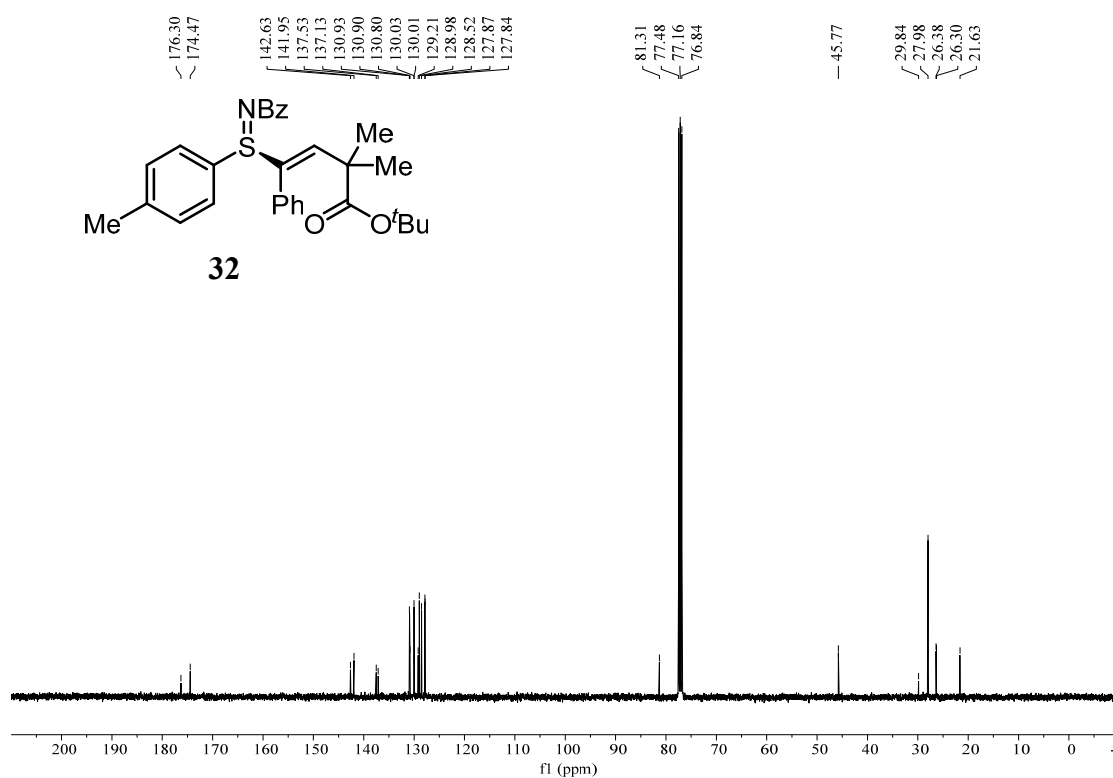
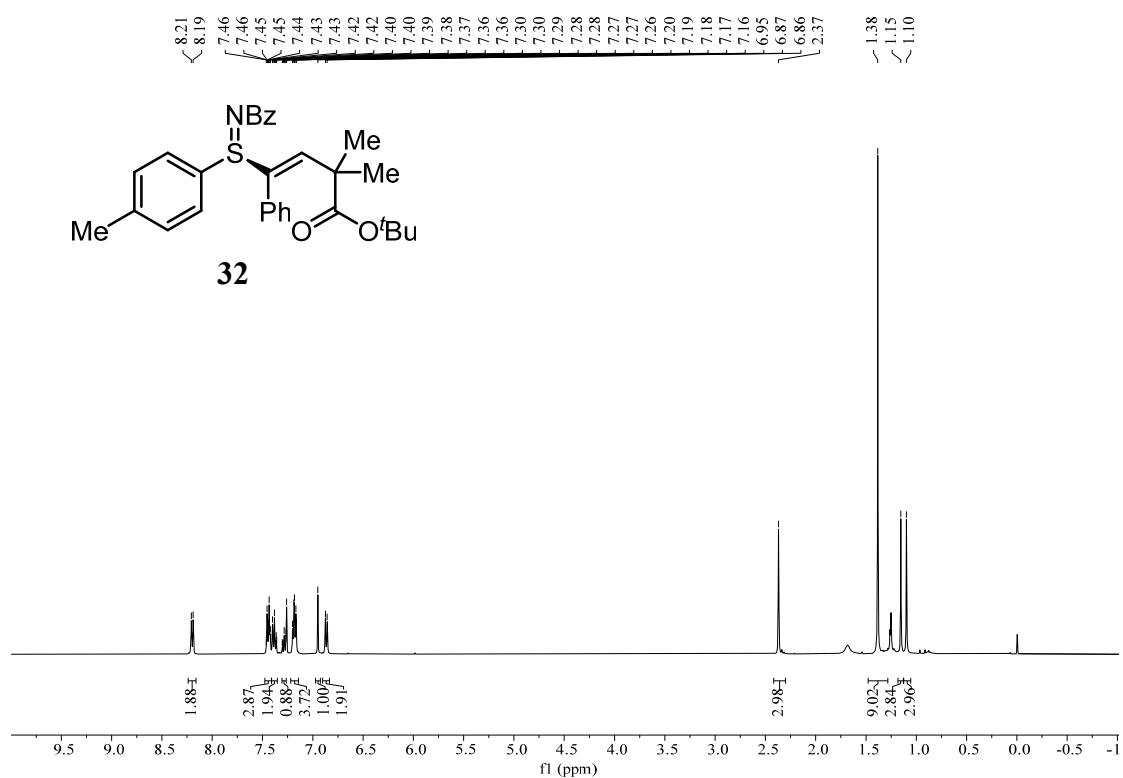


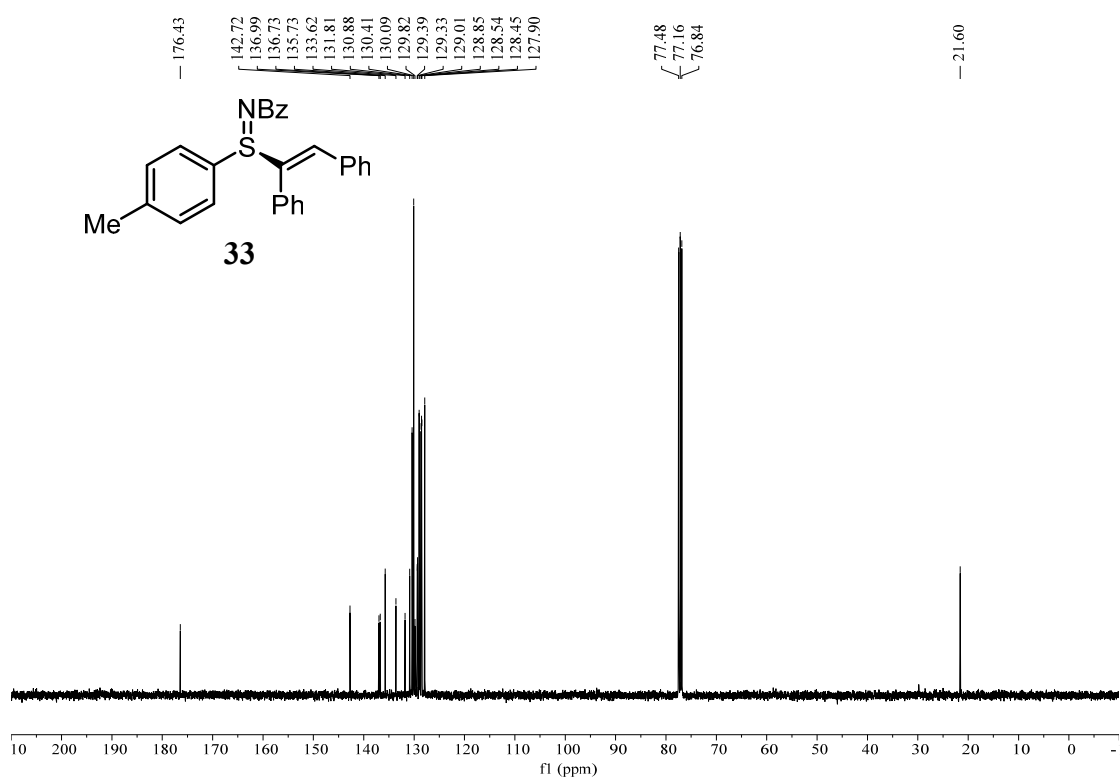
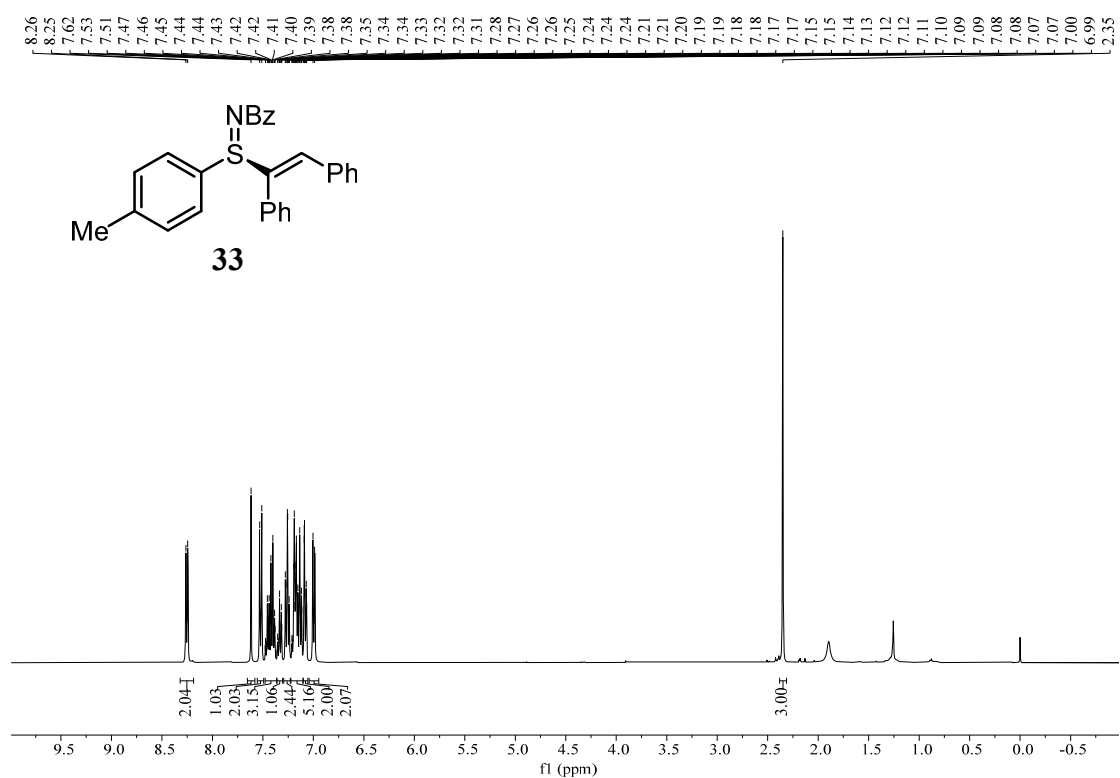


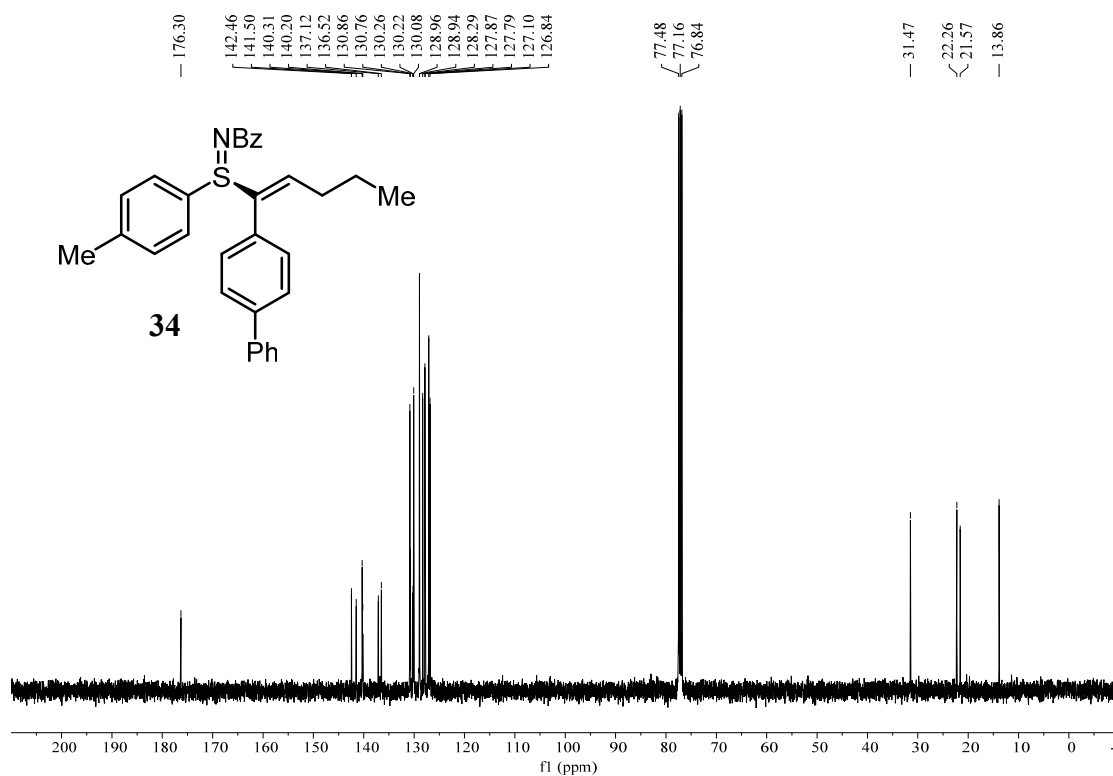
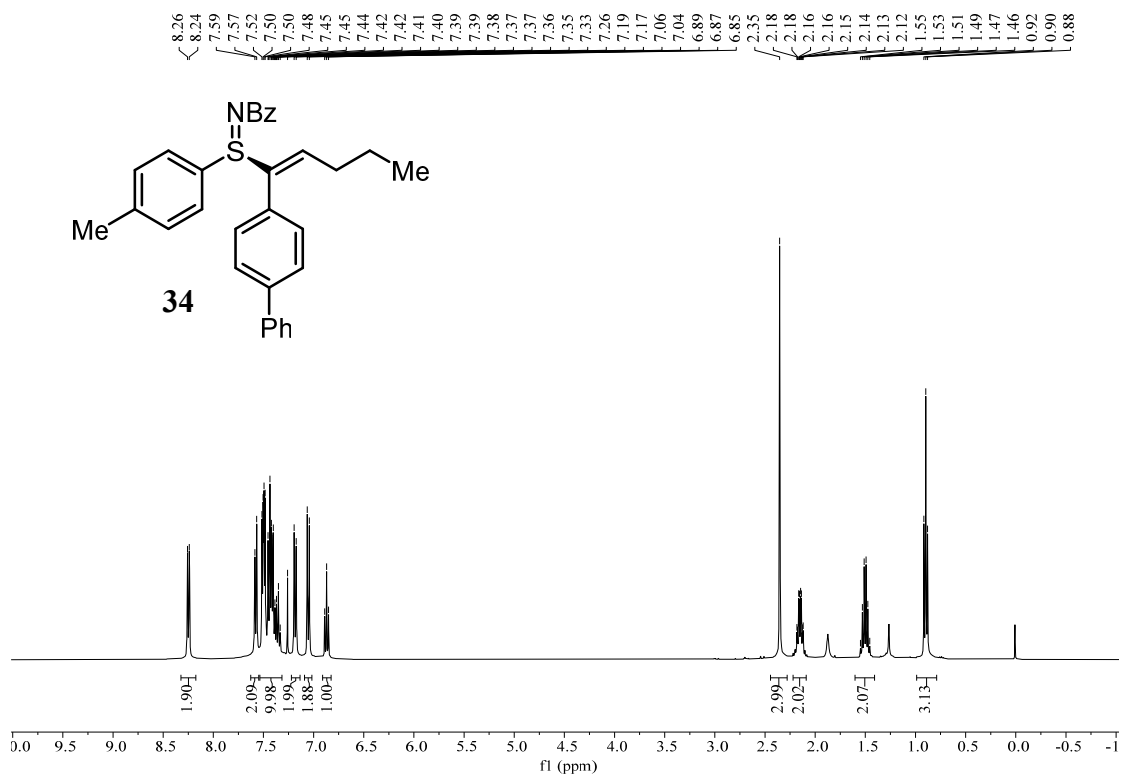


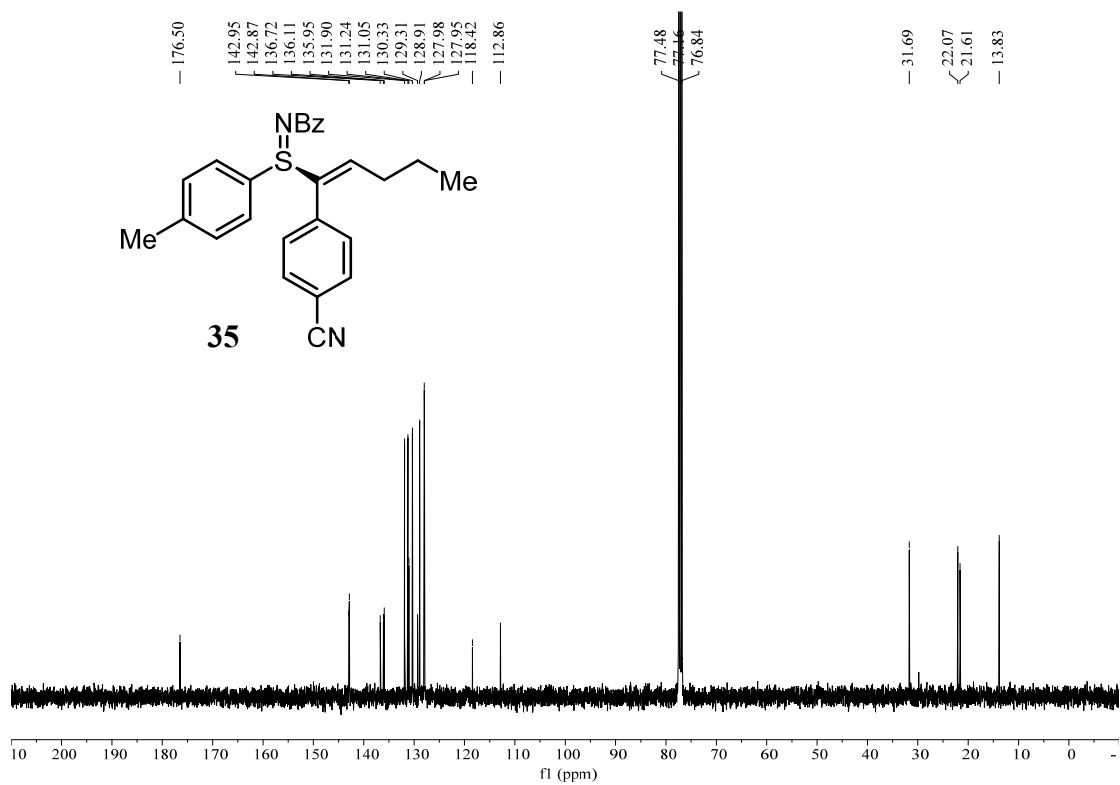
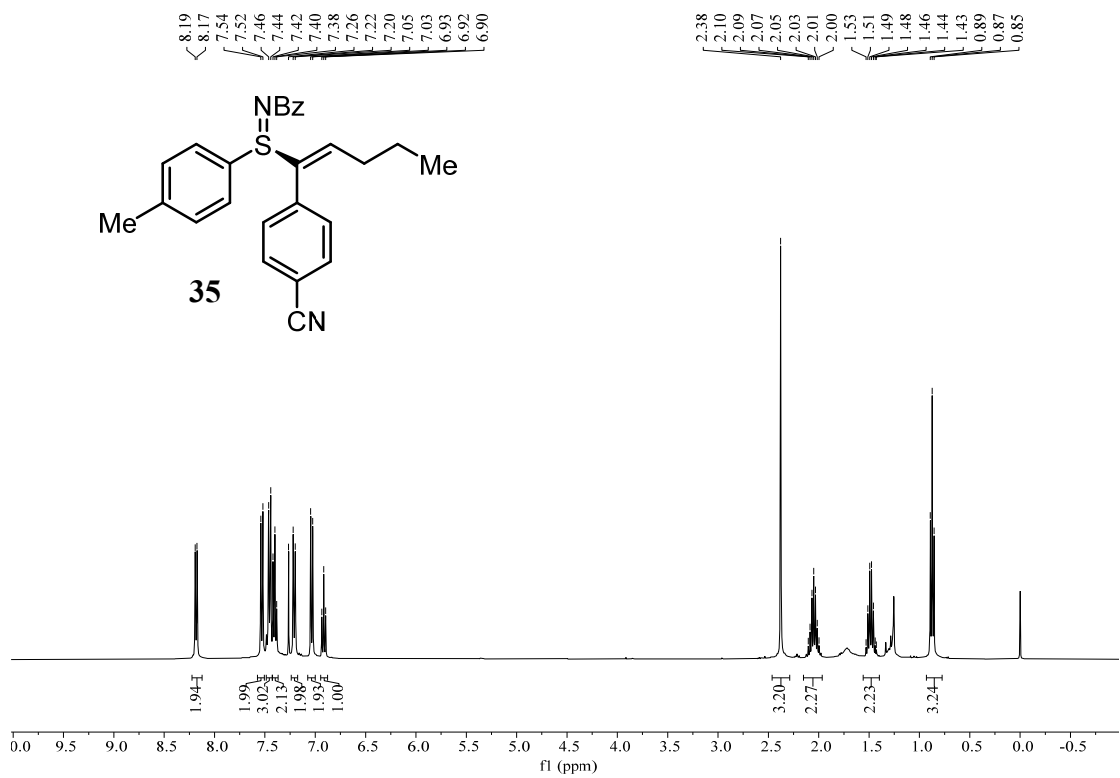


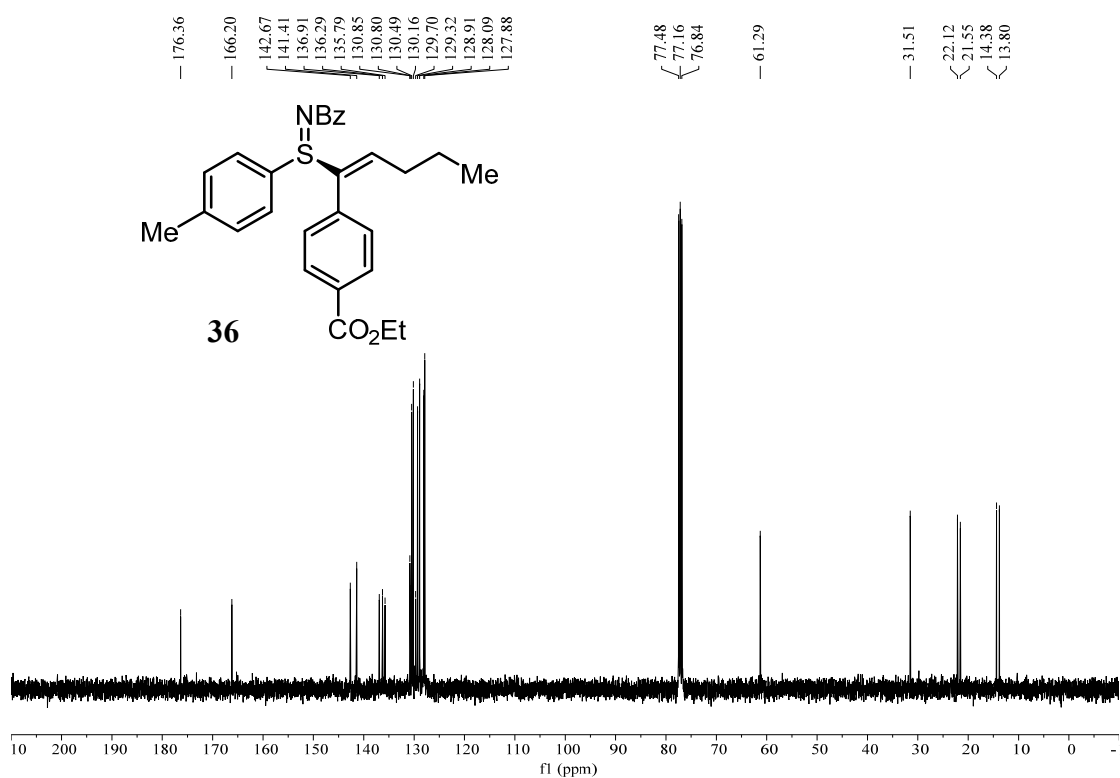
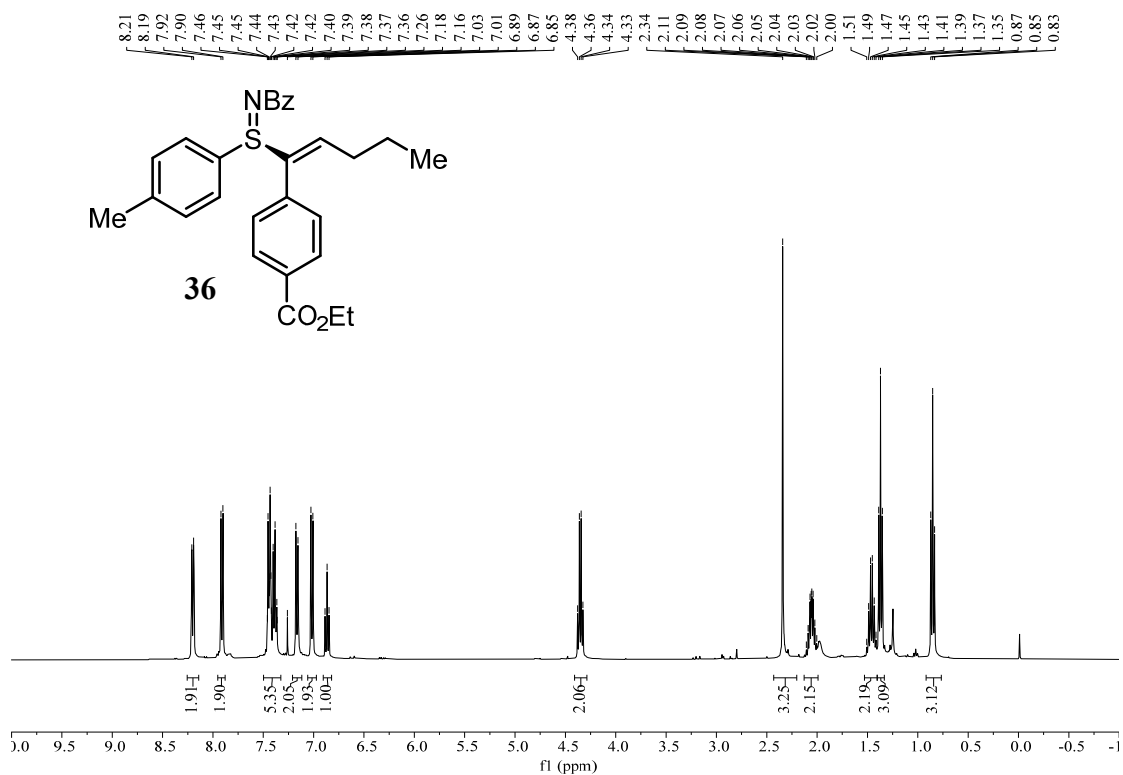


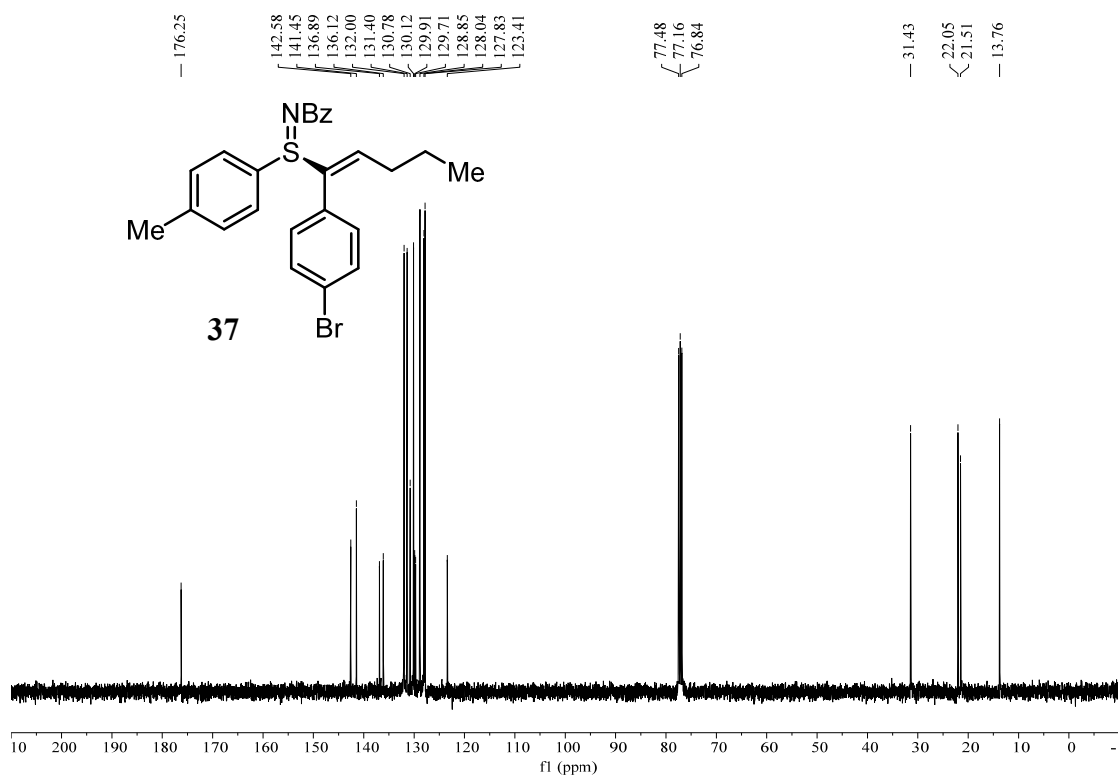
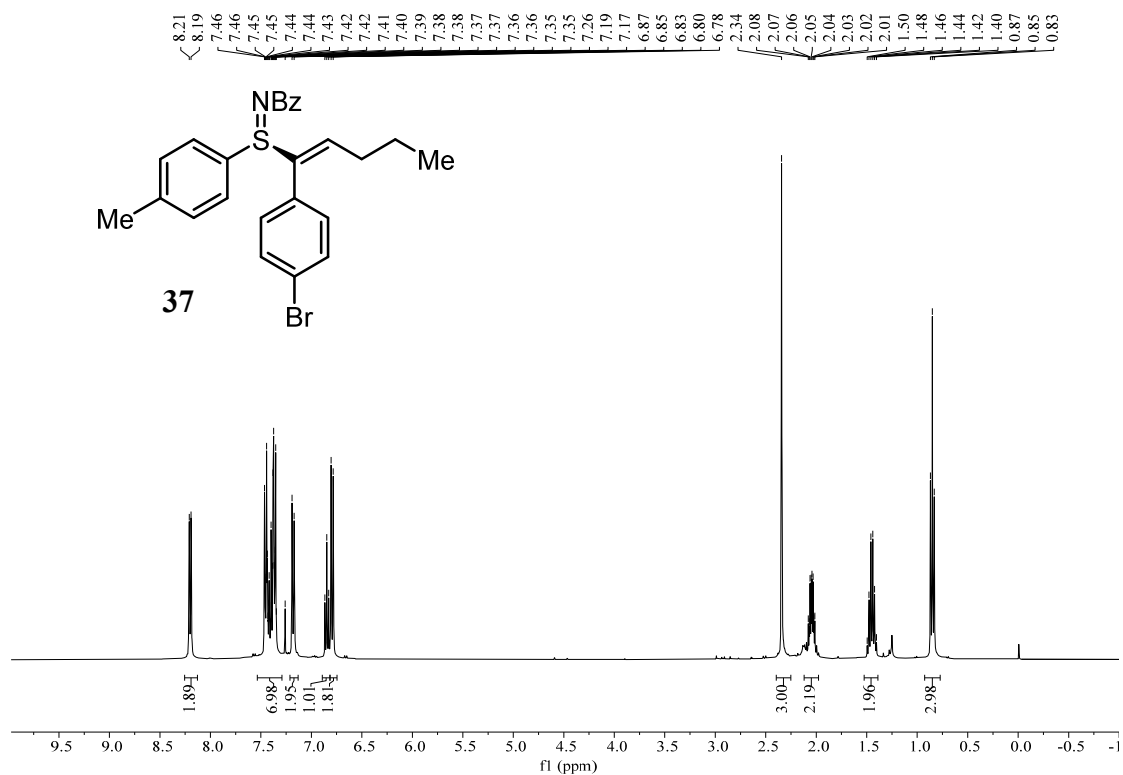


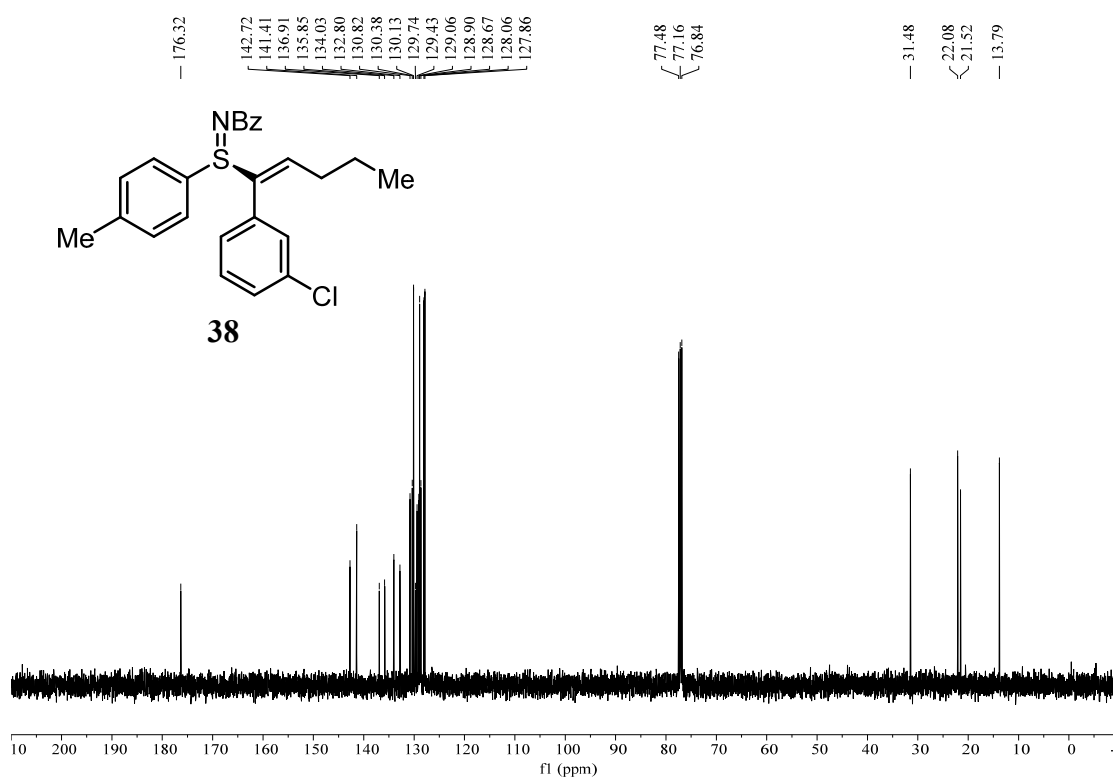
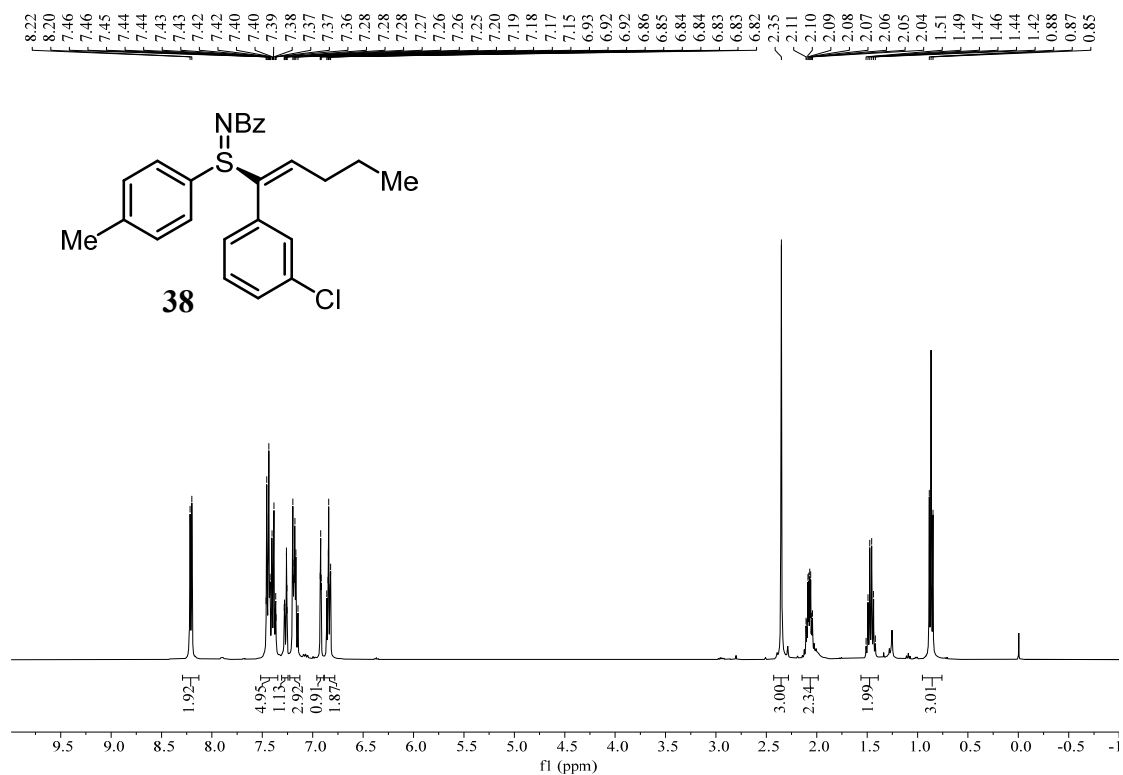


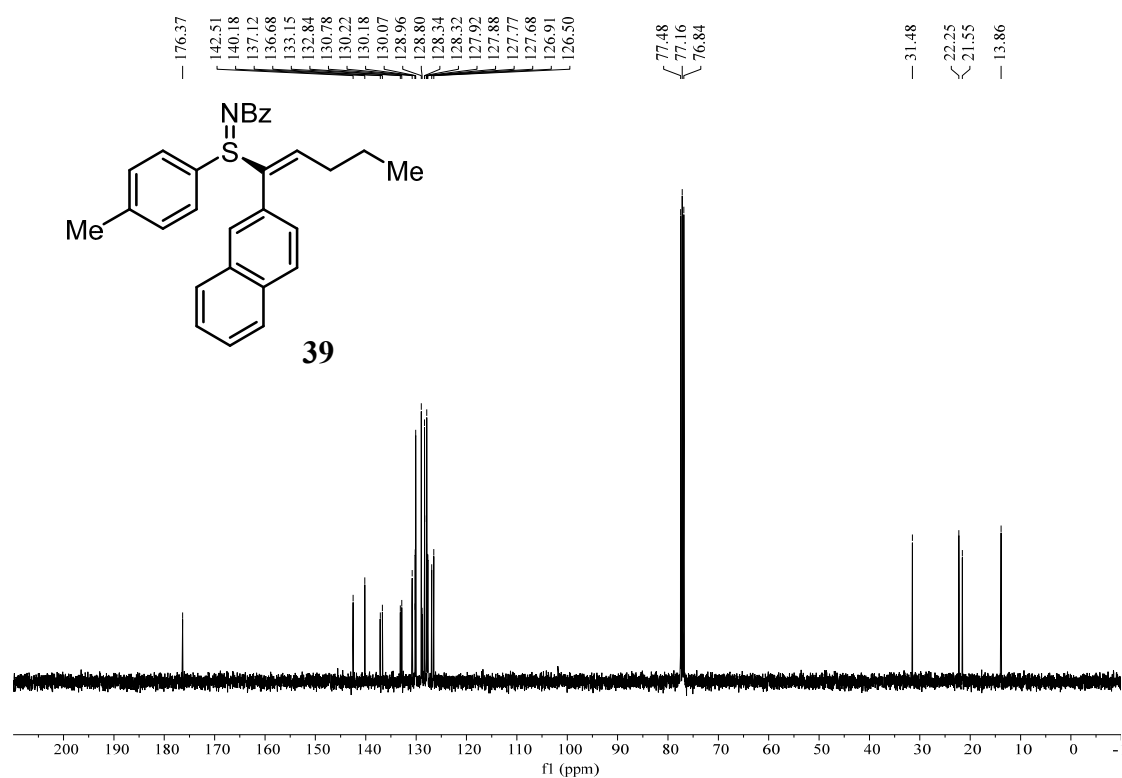
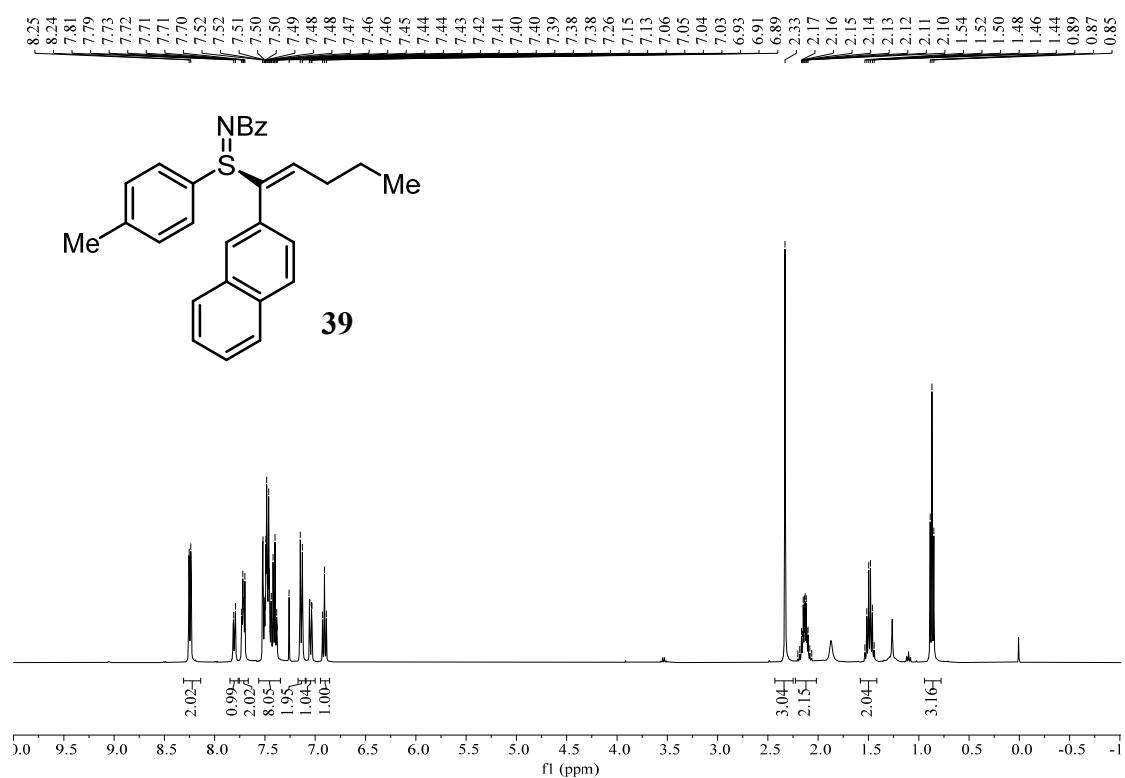


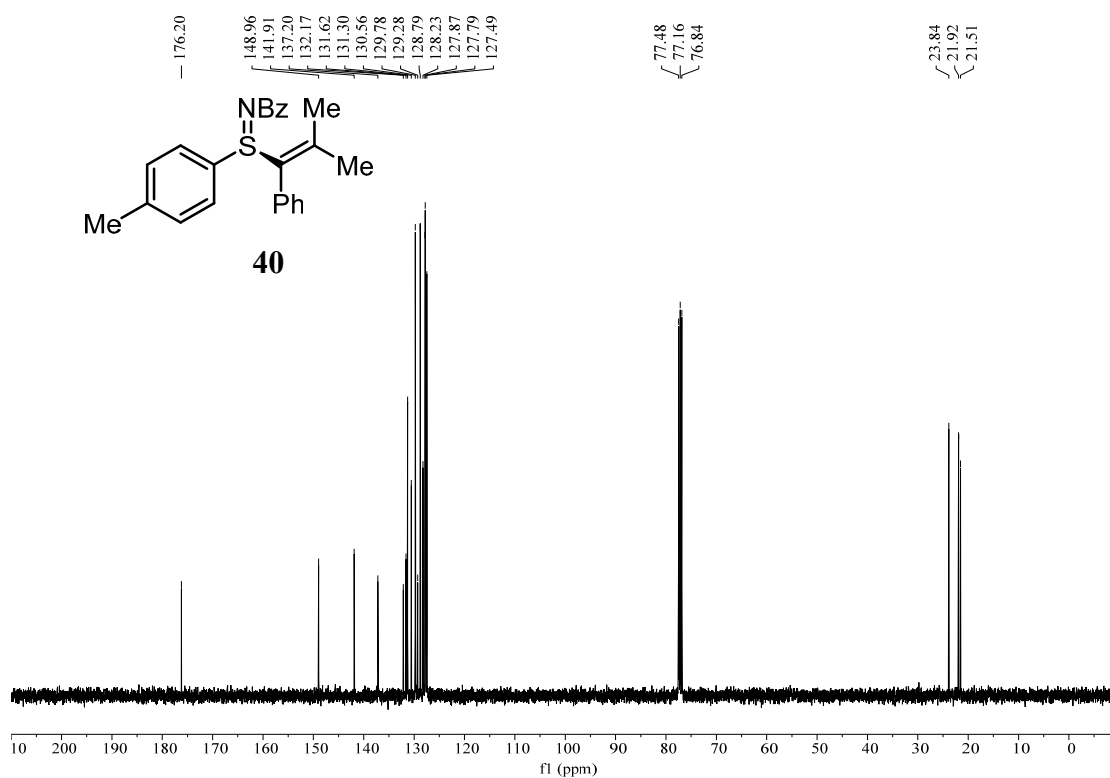
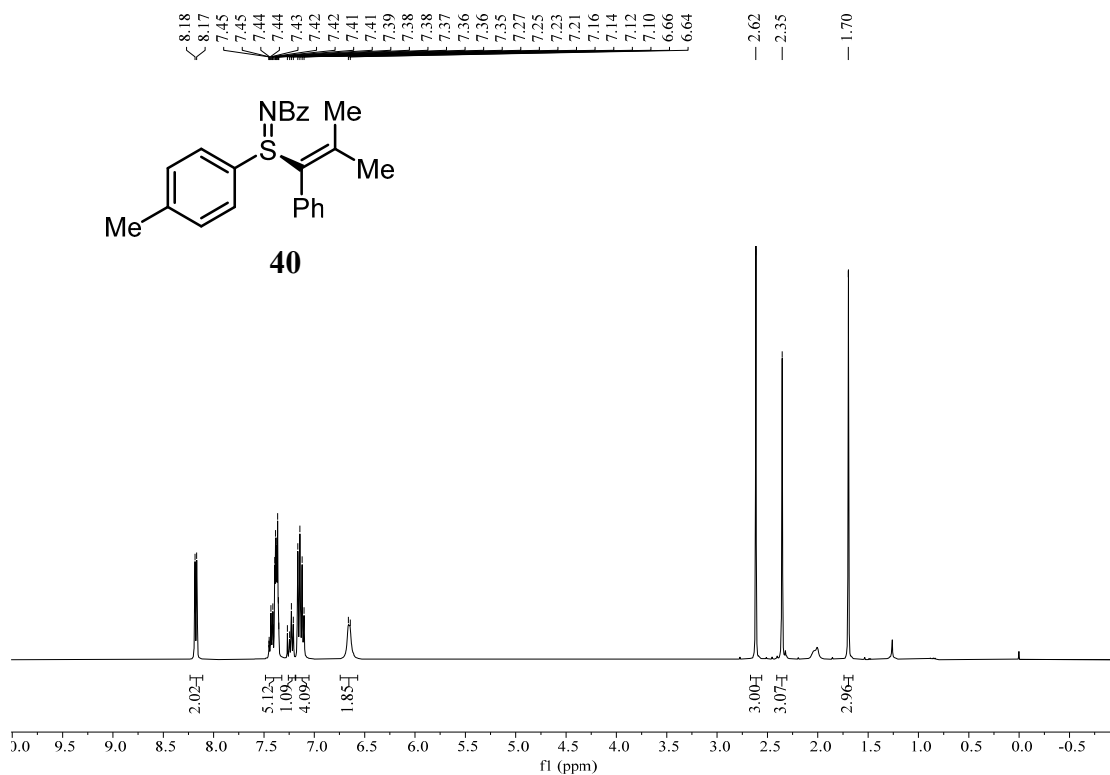


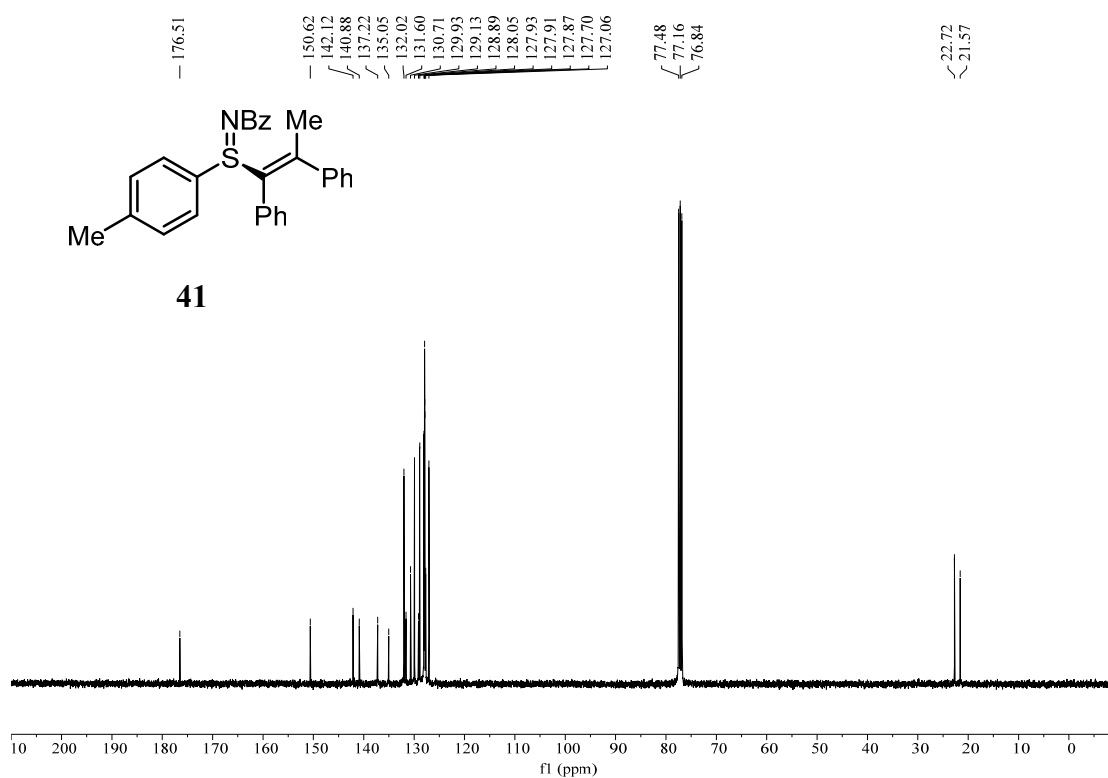
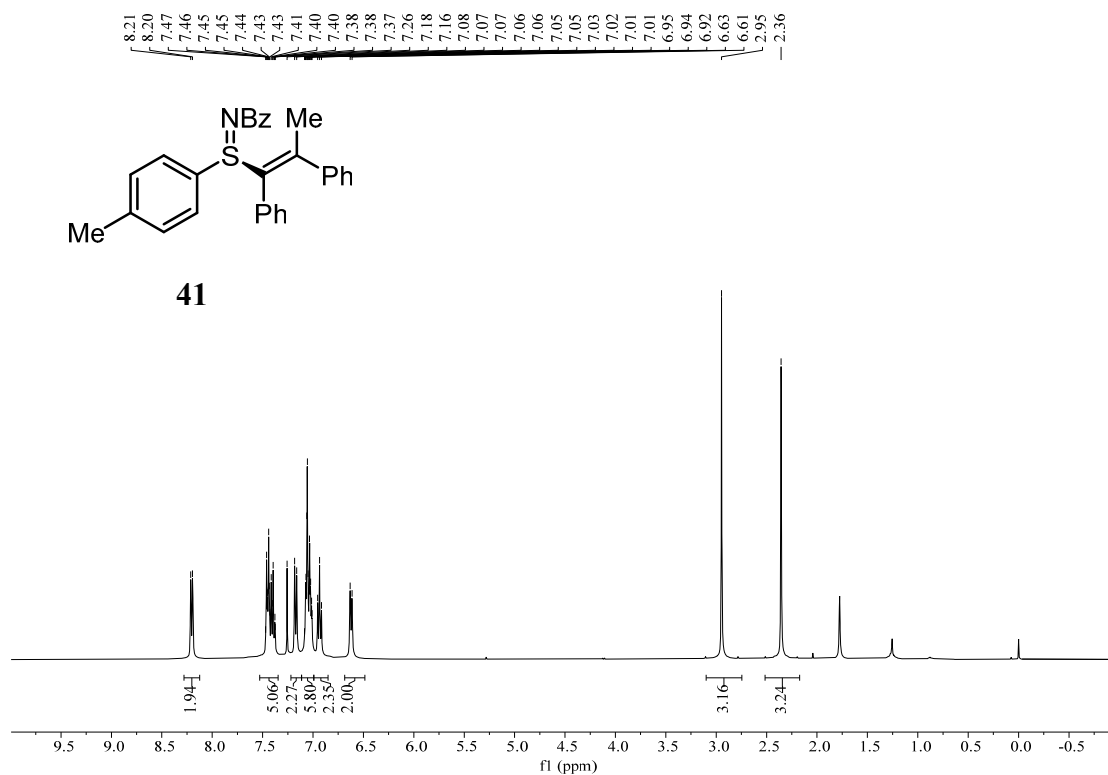


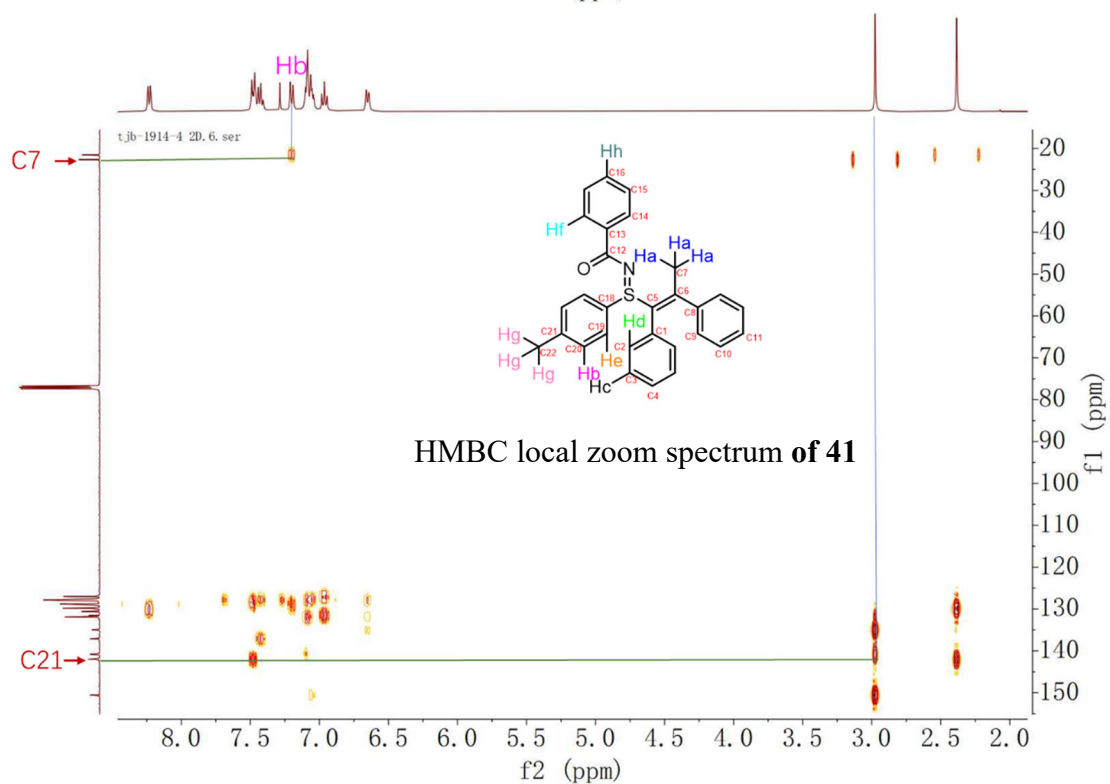
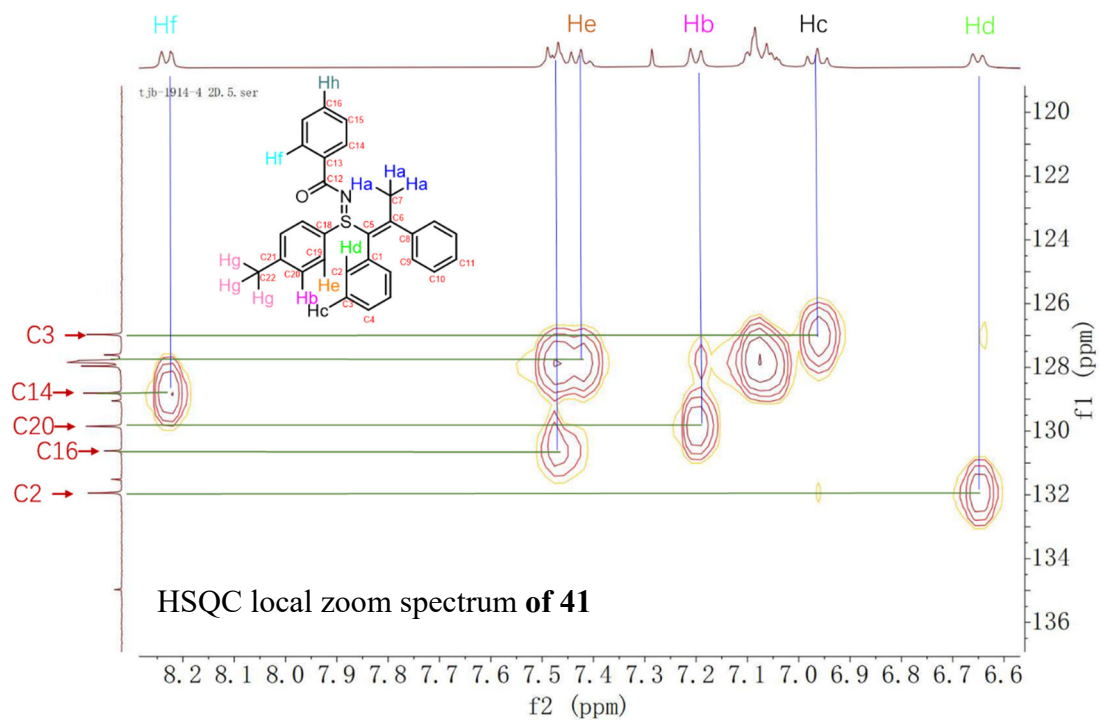


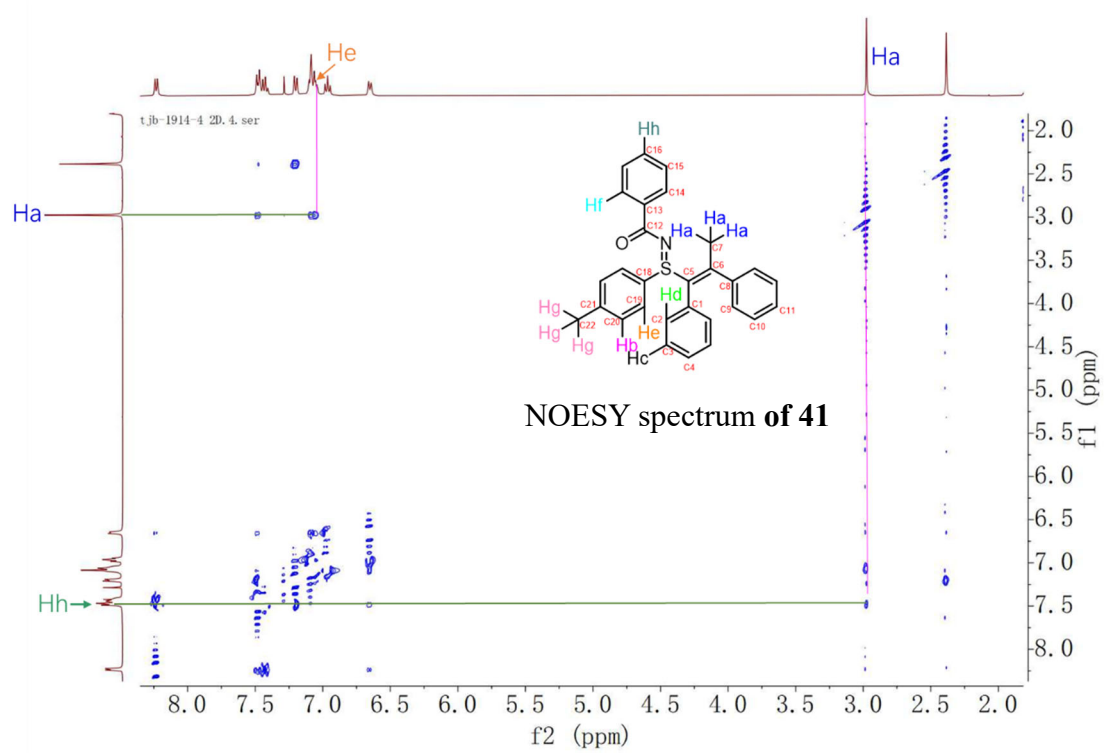


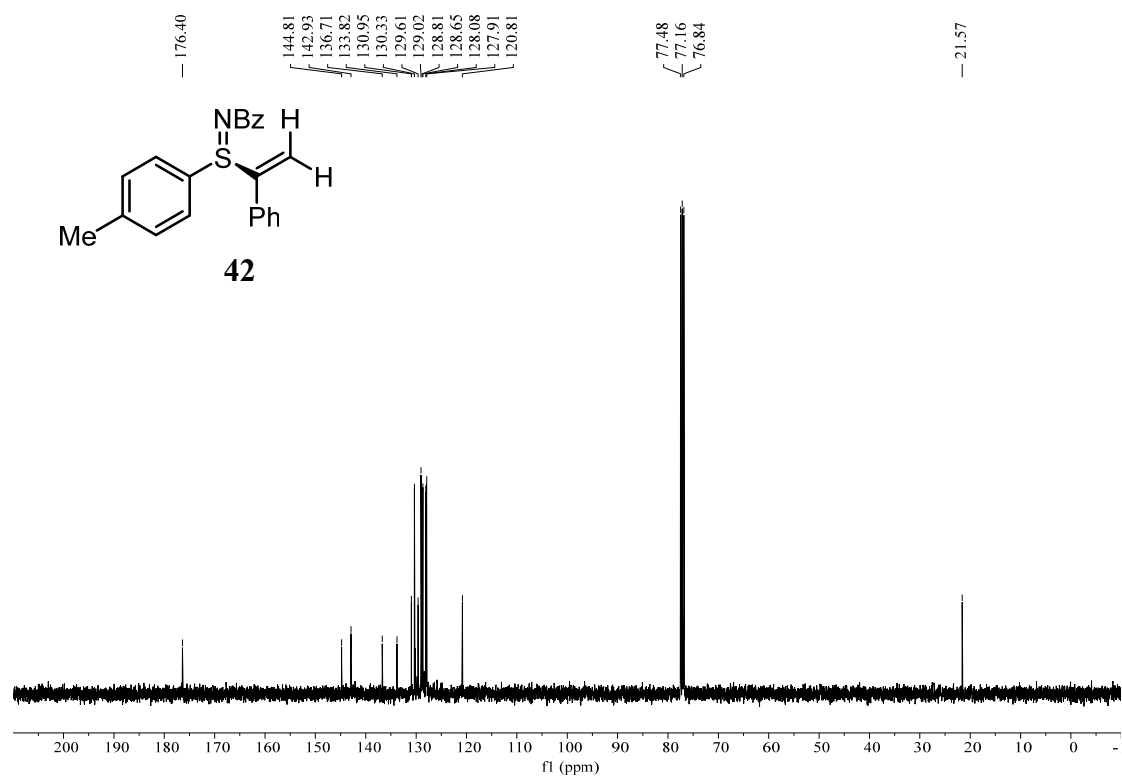
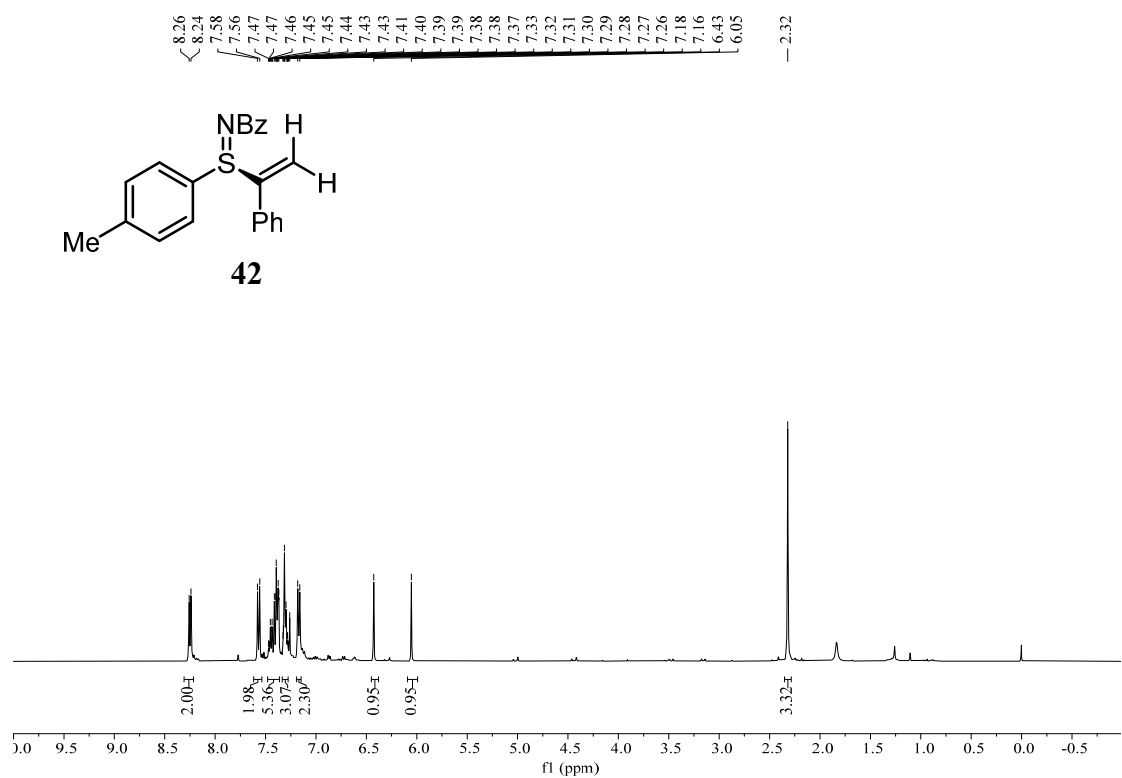


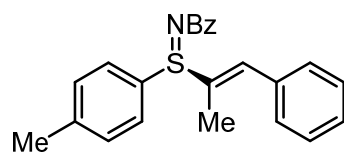




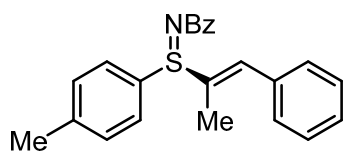
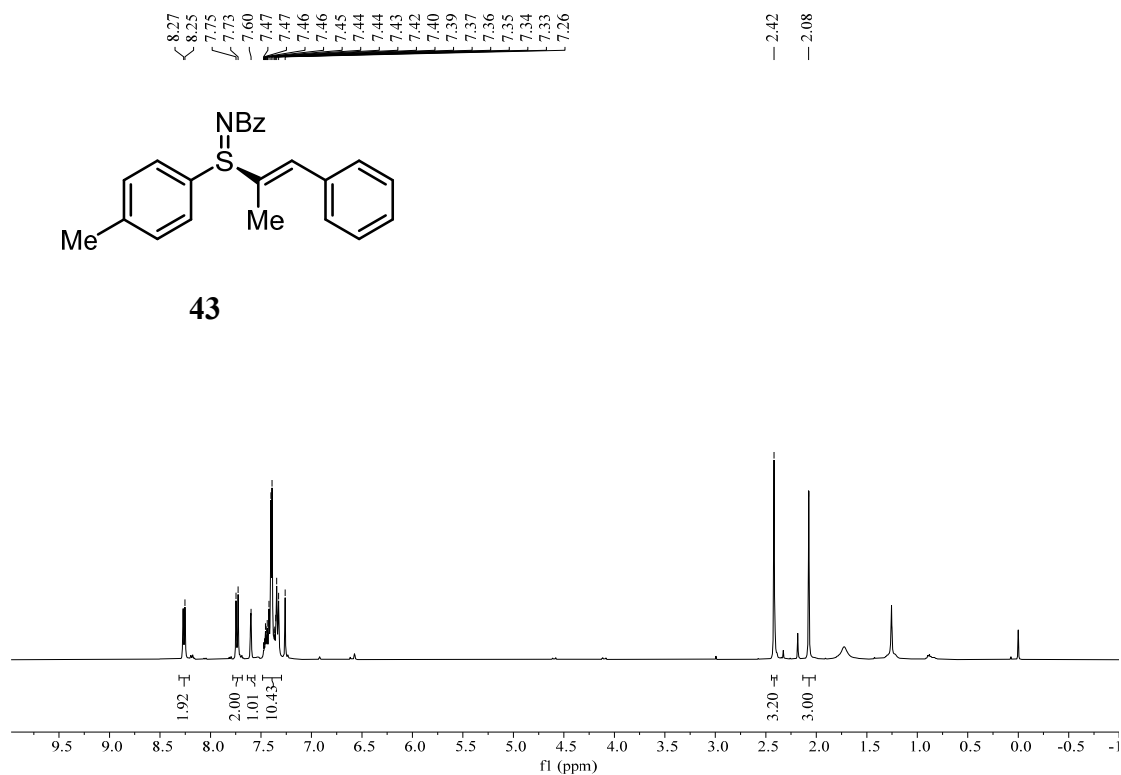




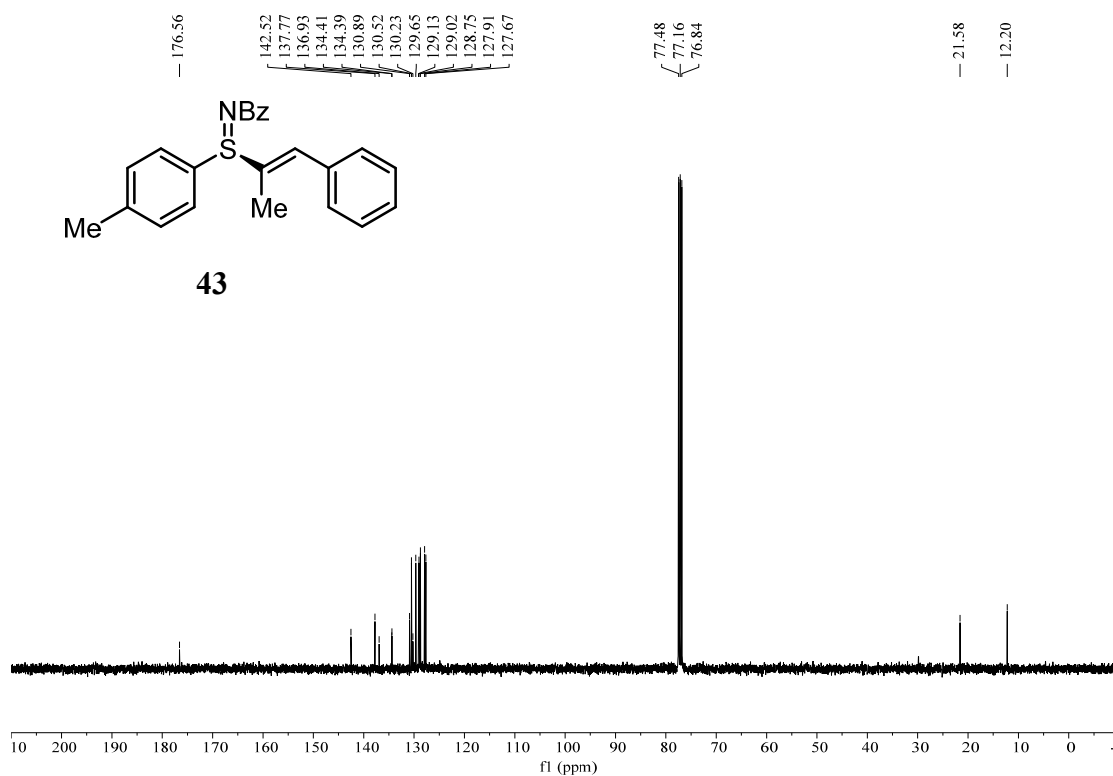


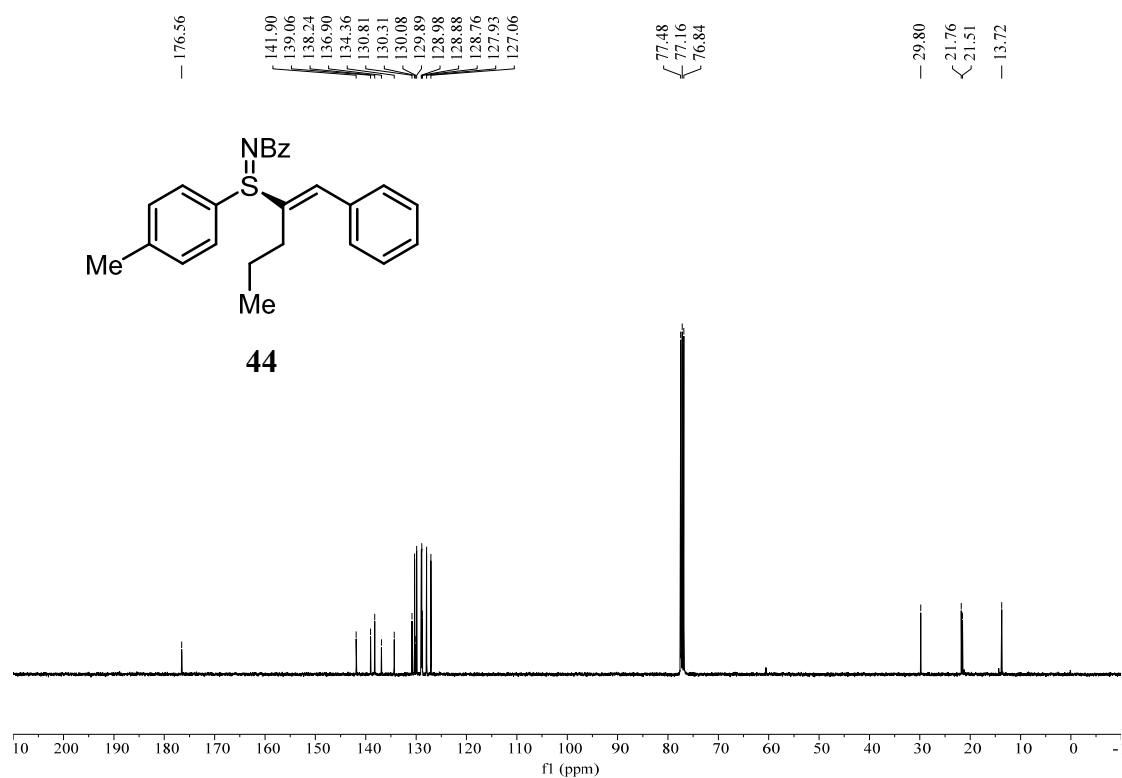
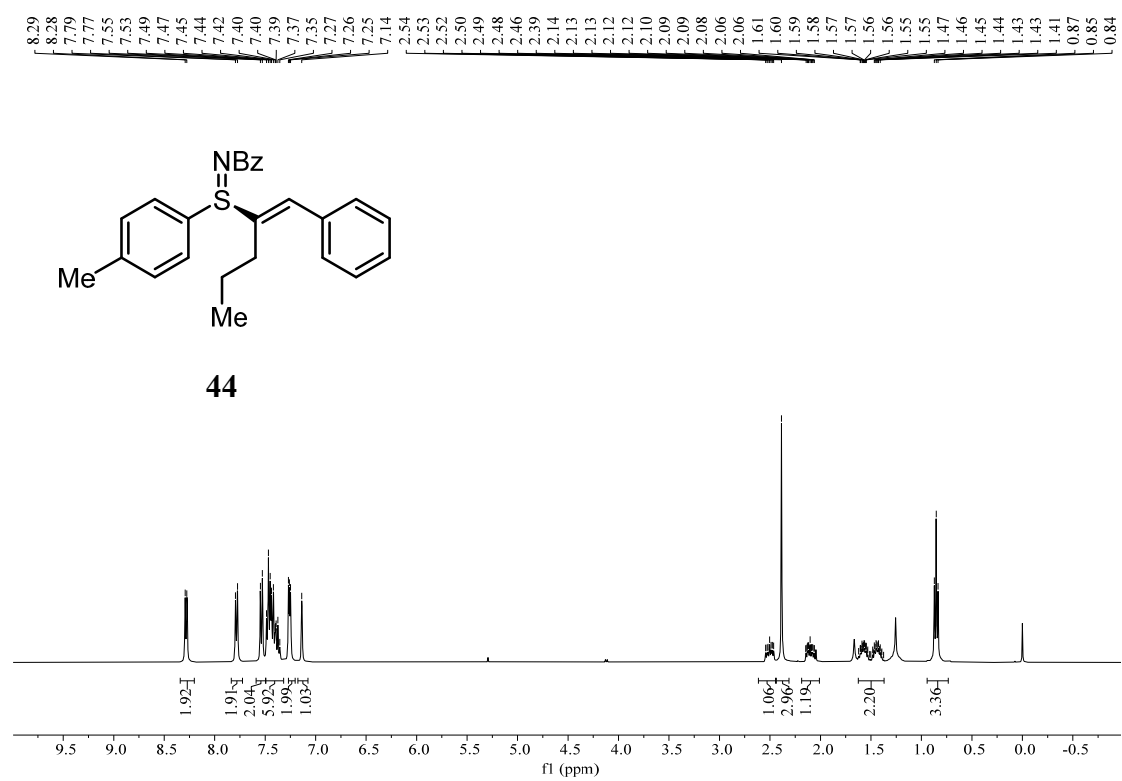


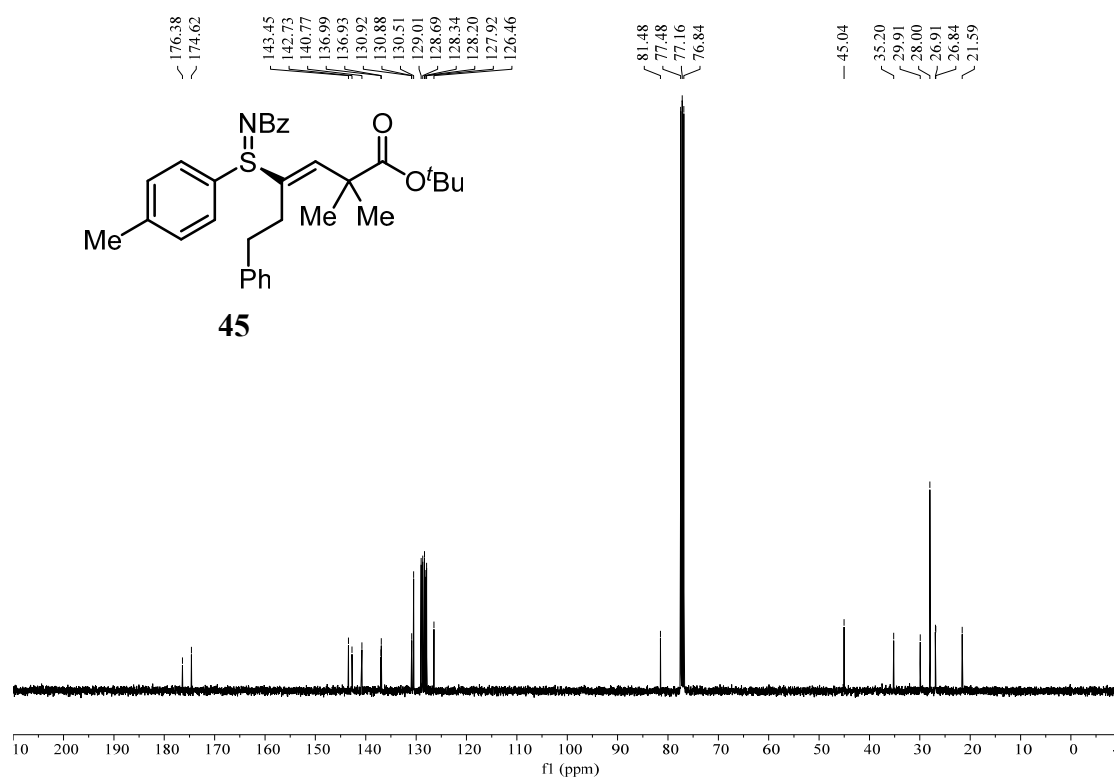
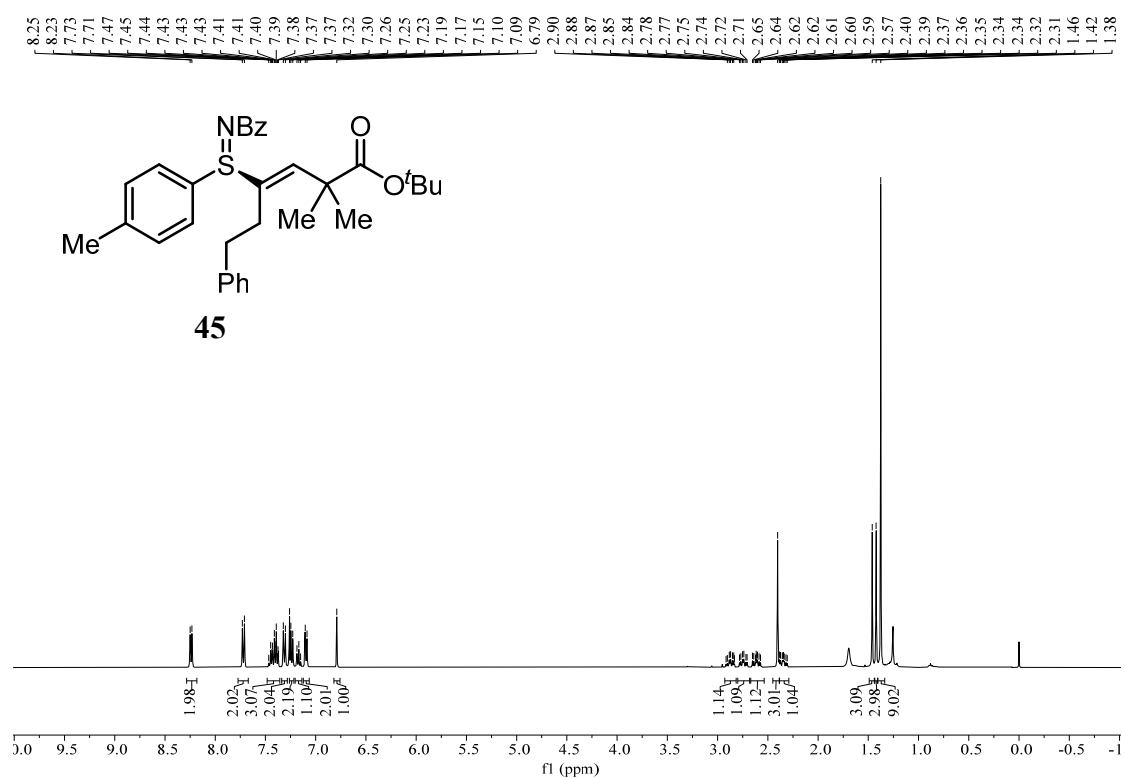
43

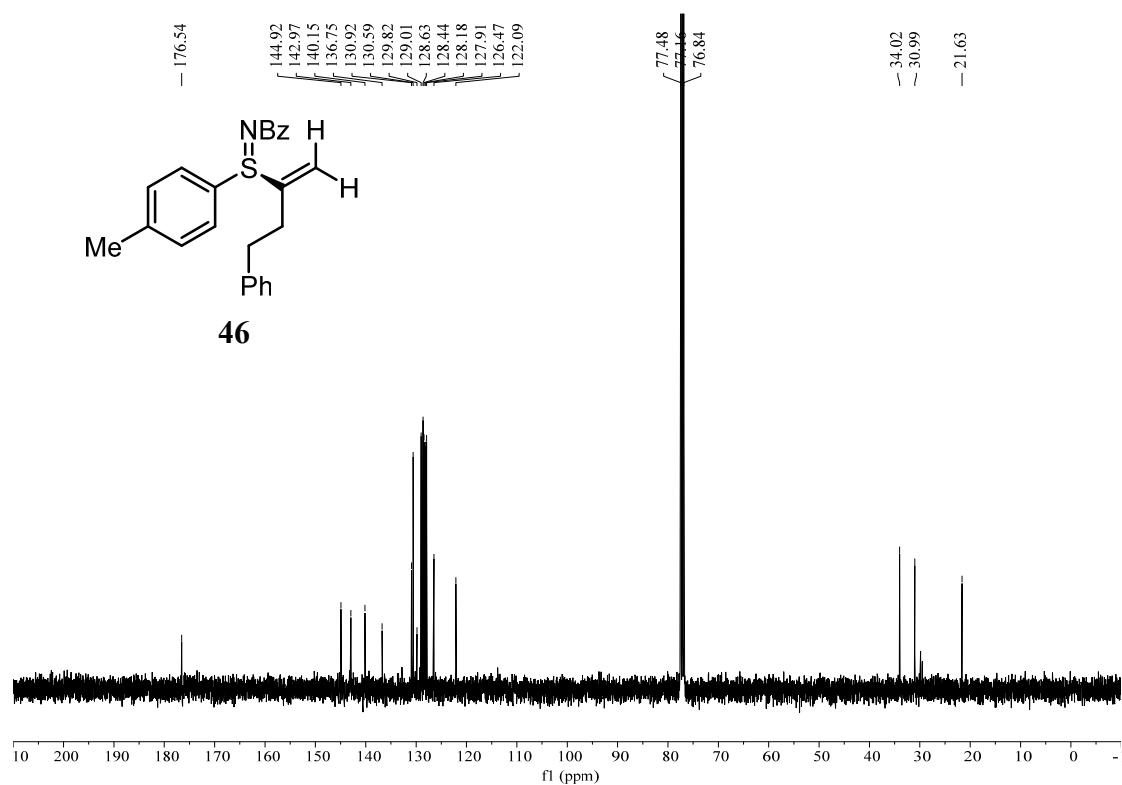
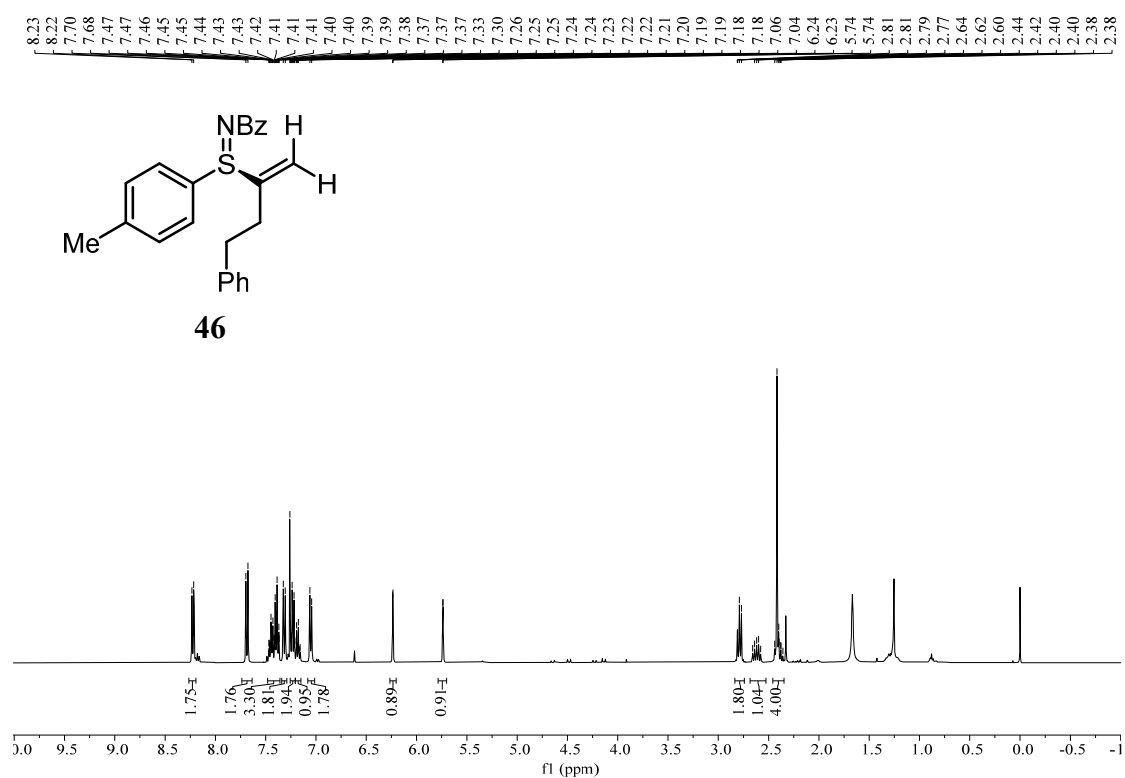


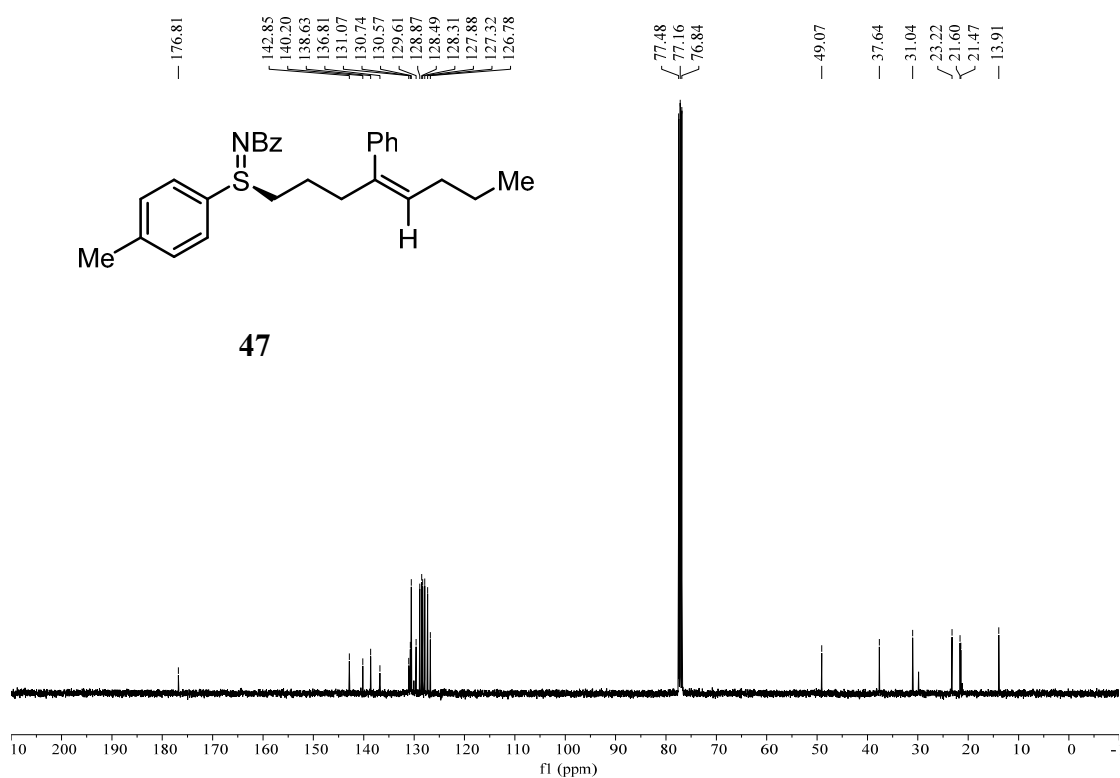
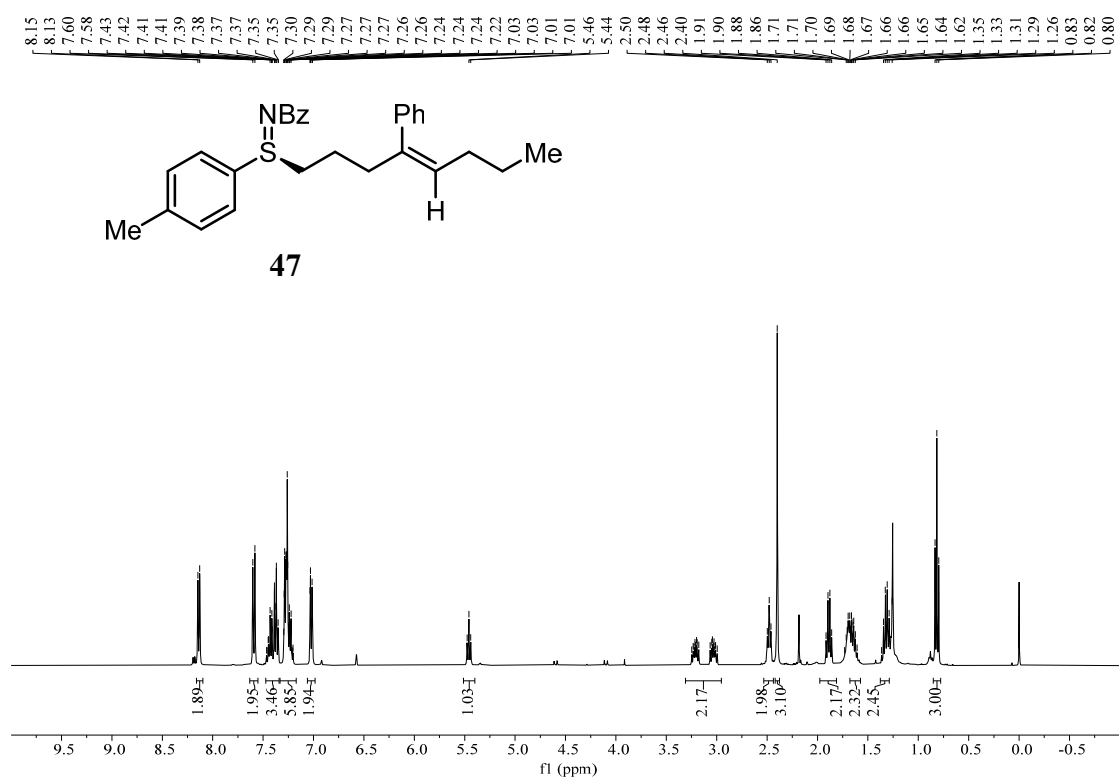
43

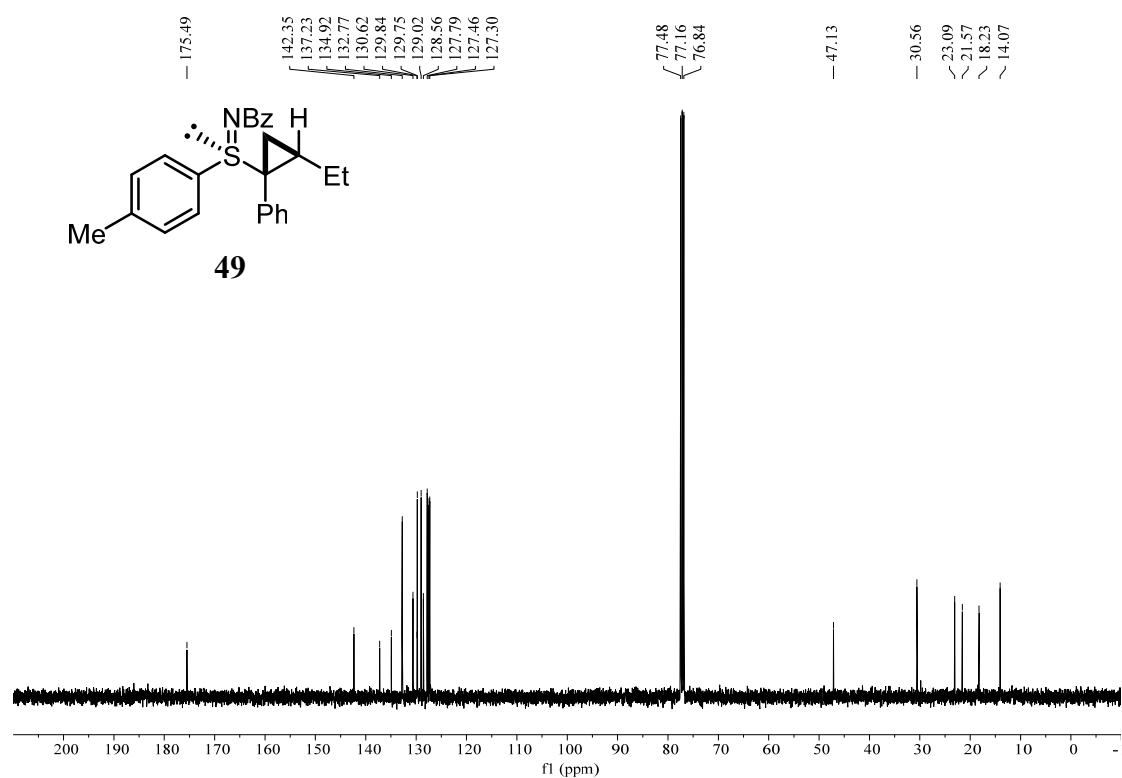
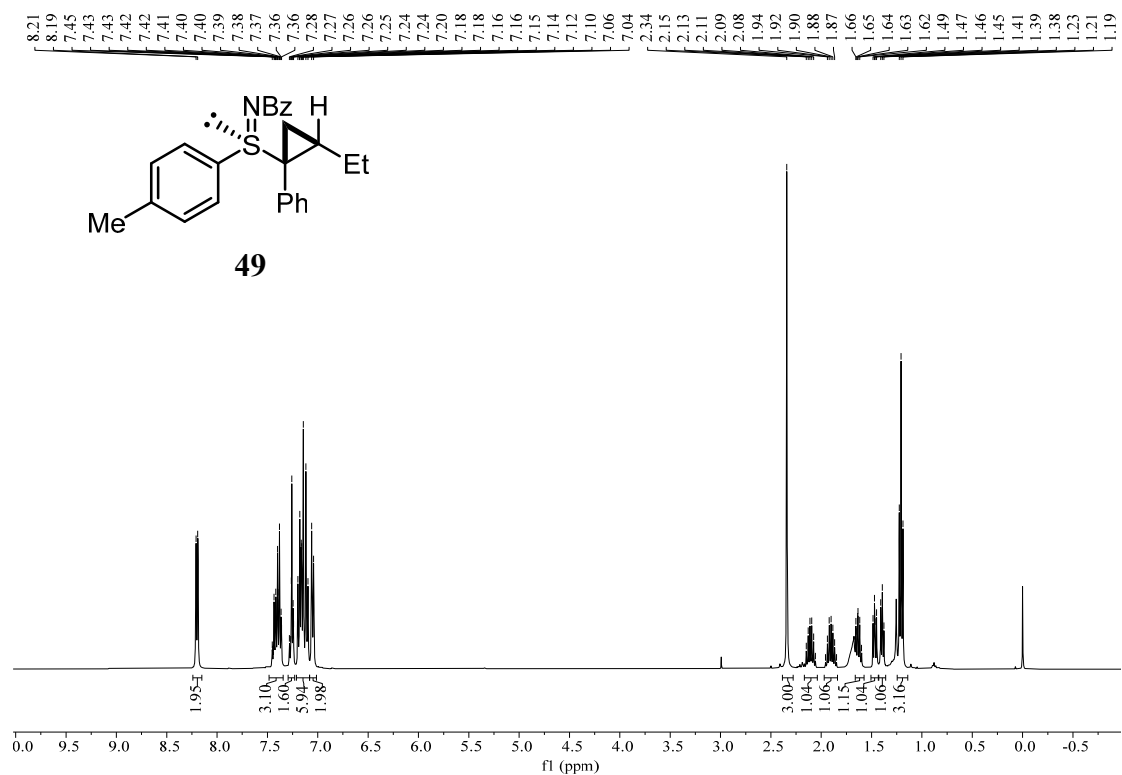


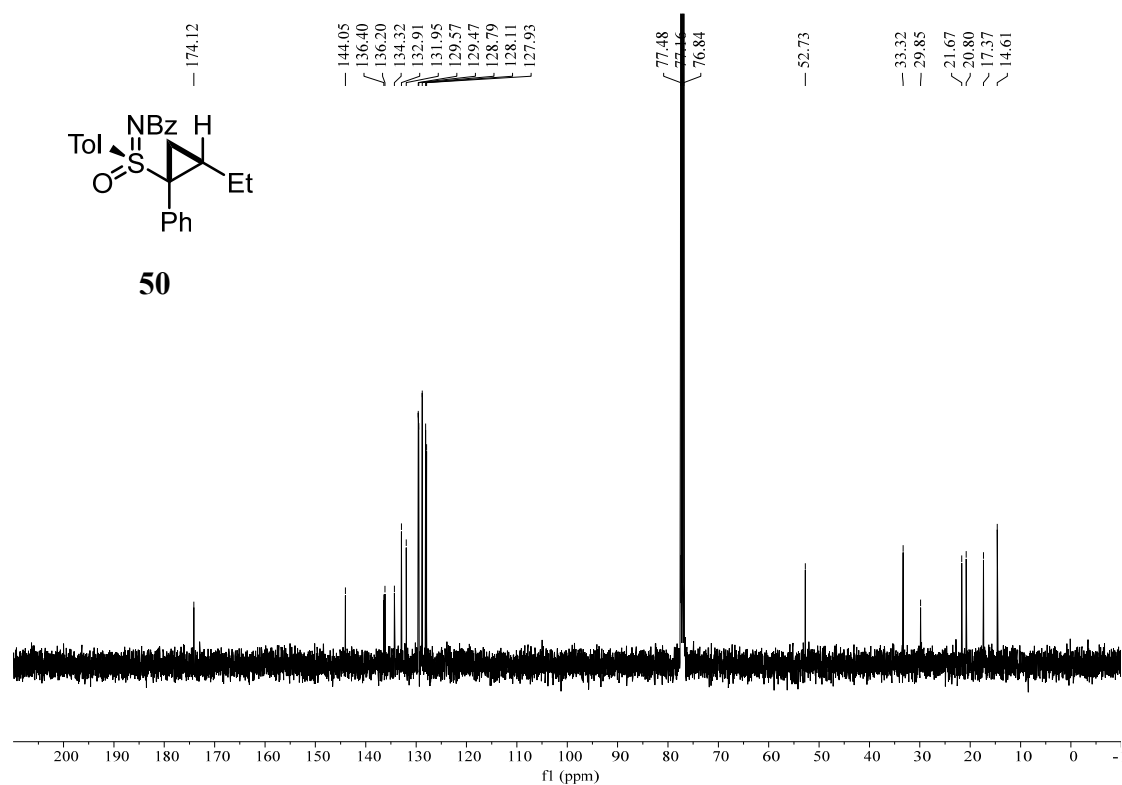
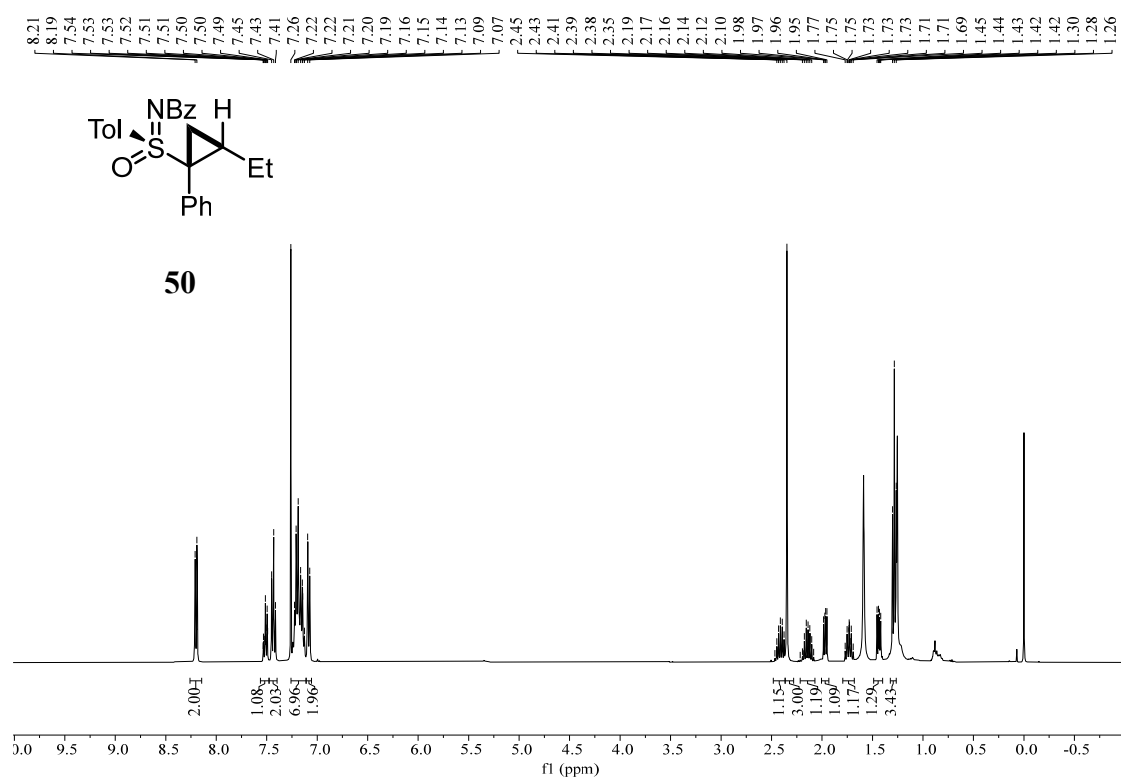


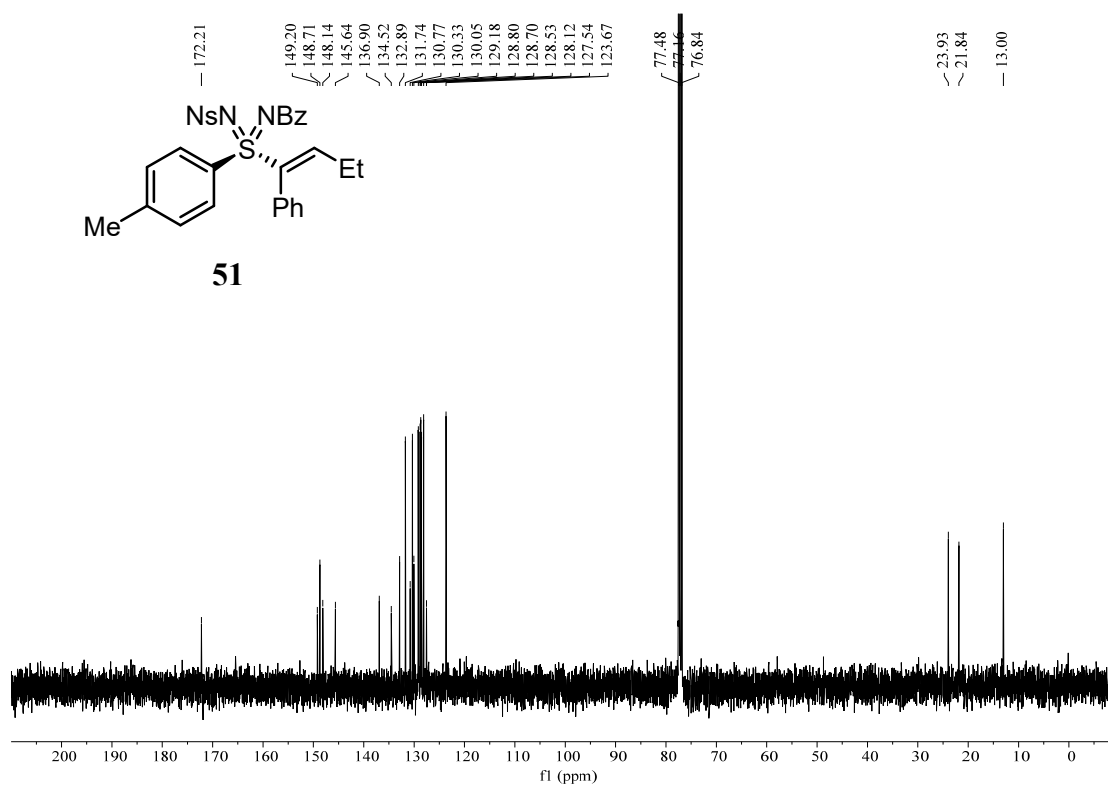
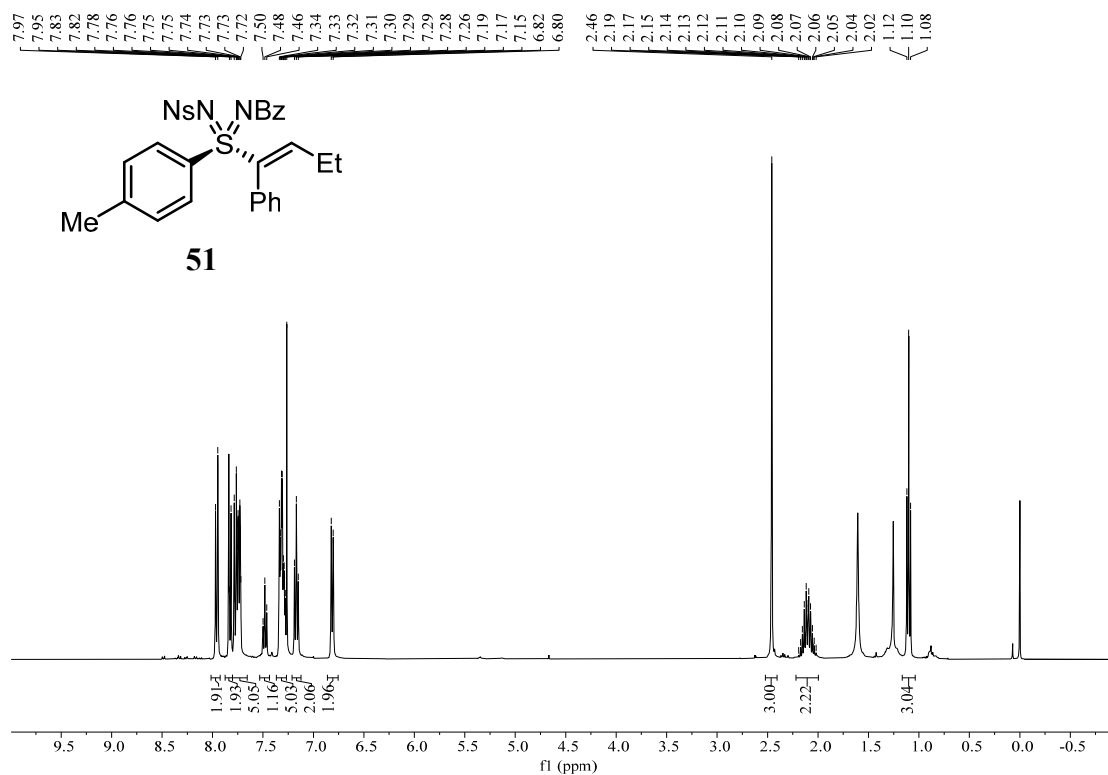


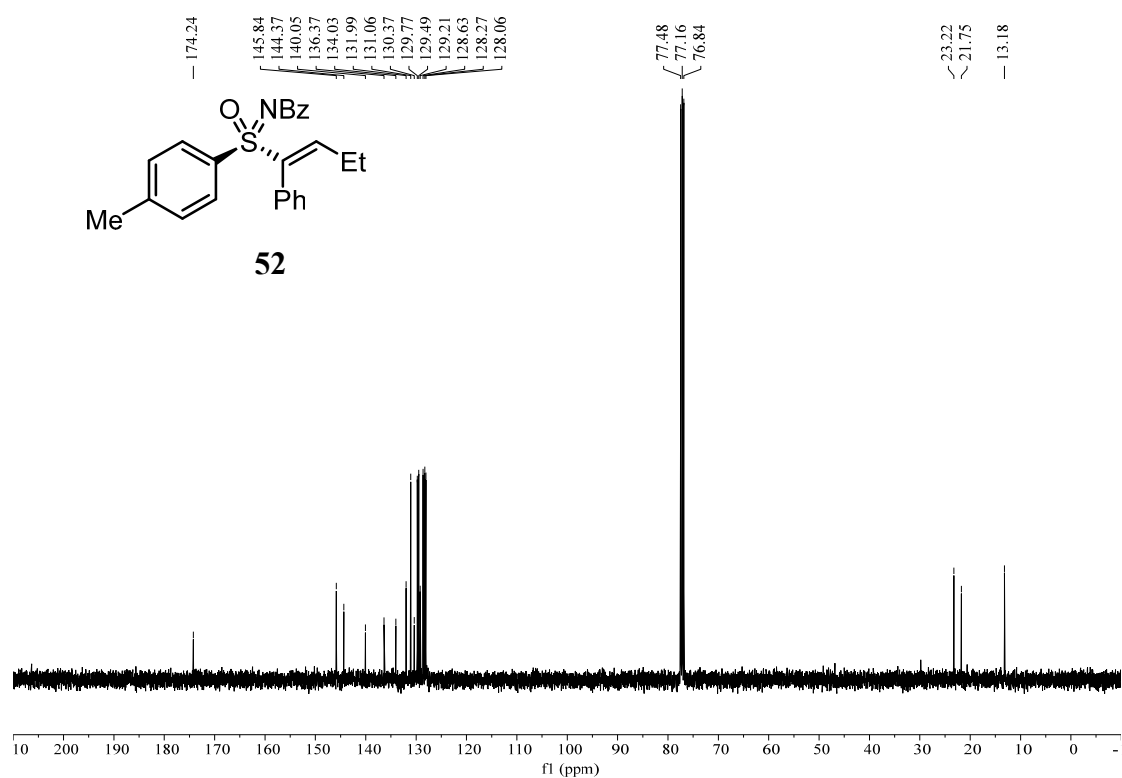
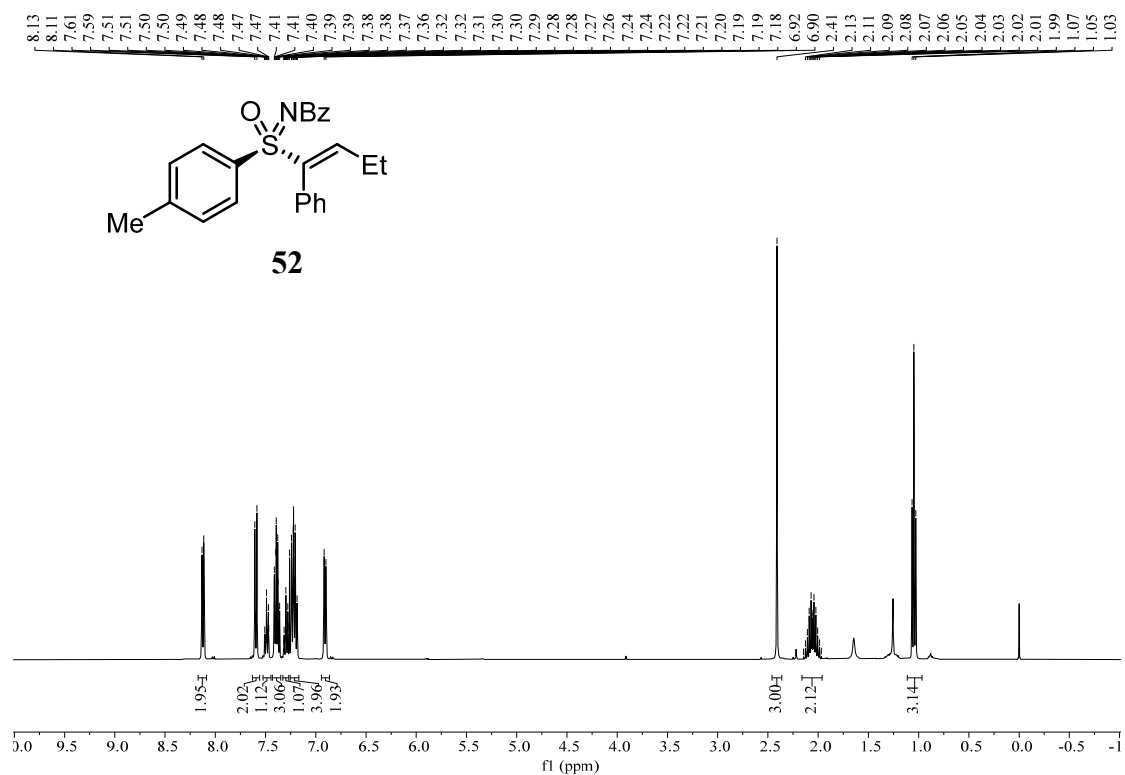


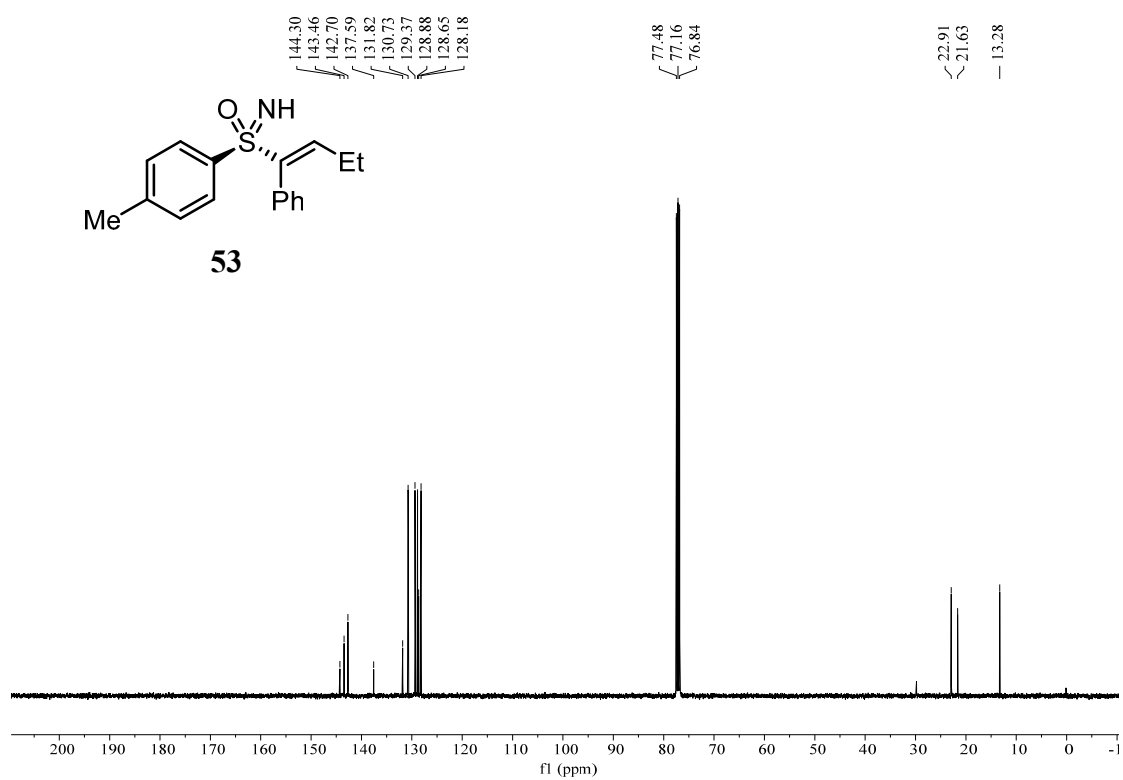
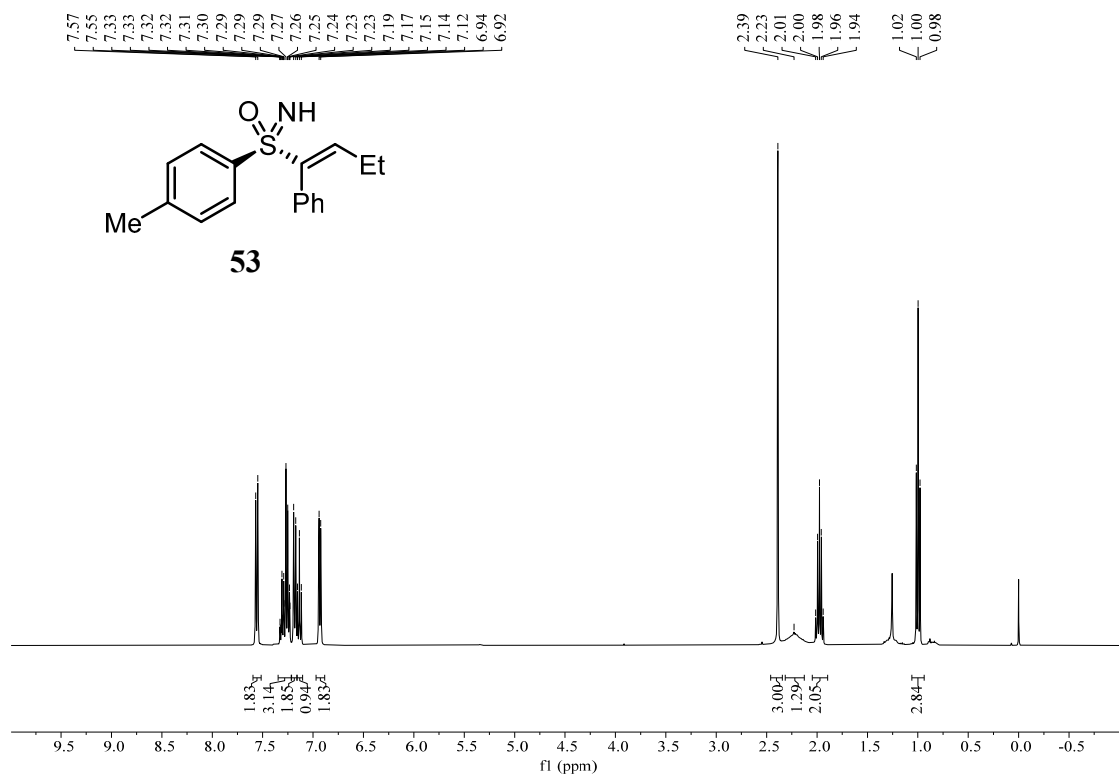


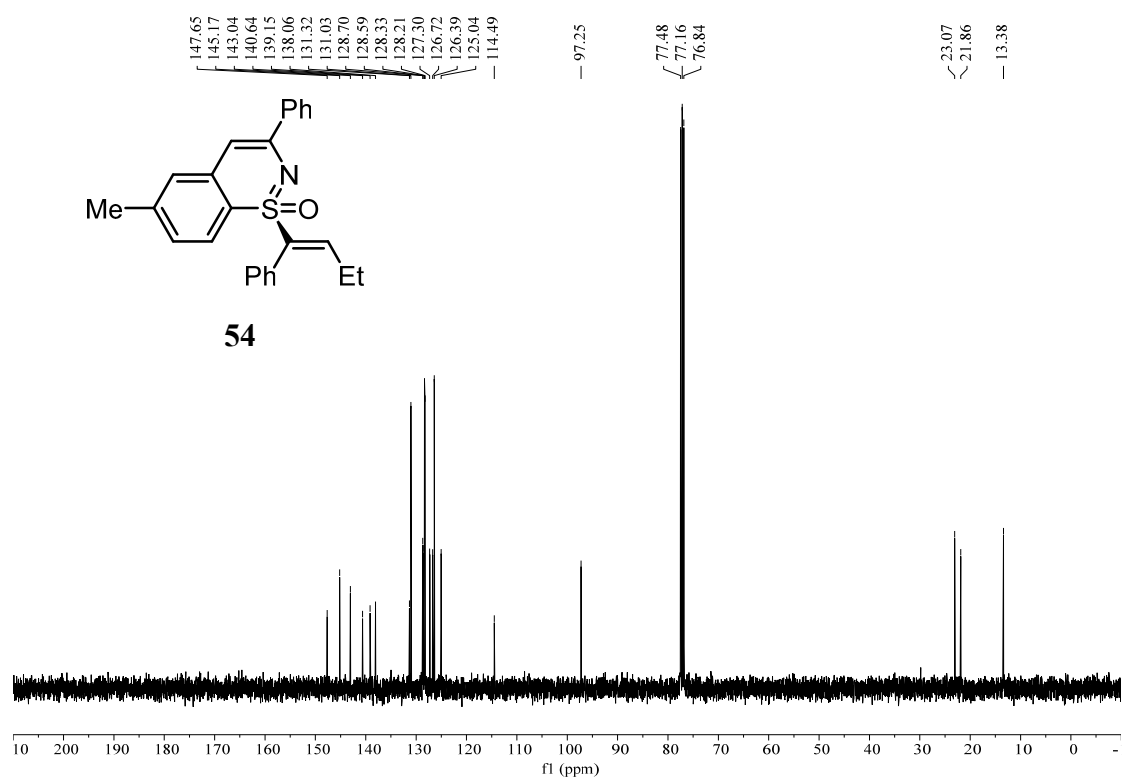
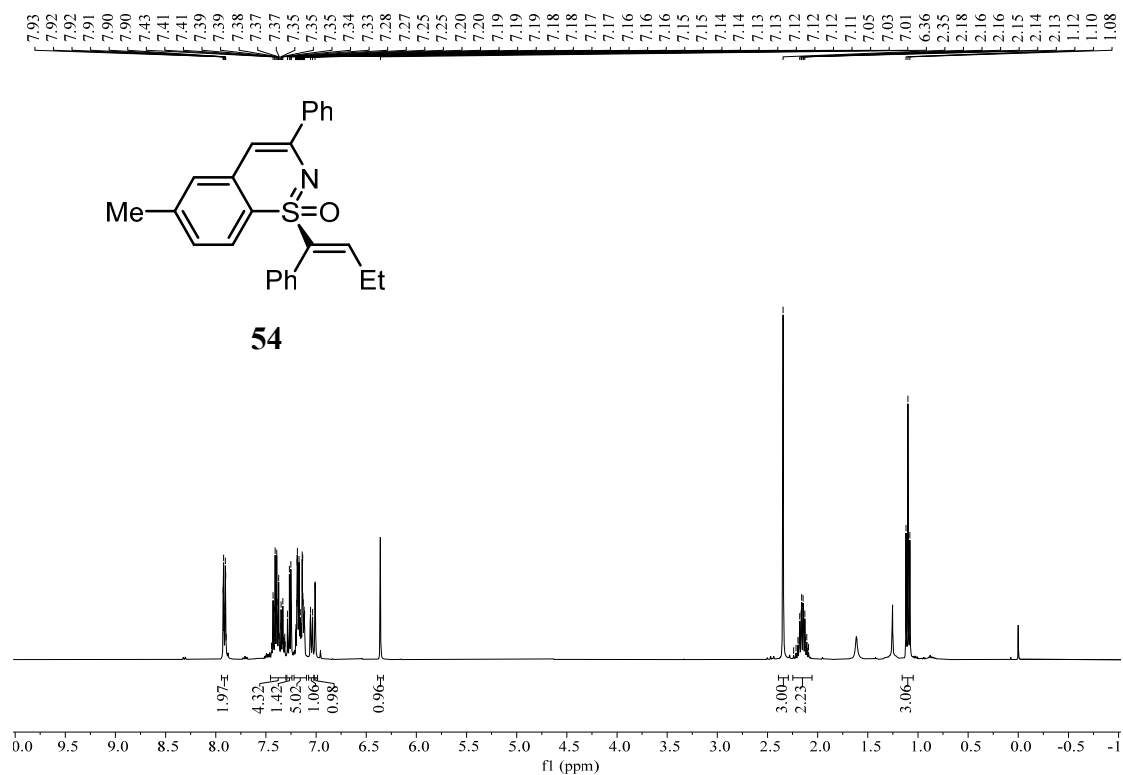


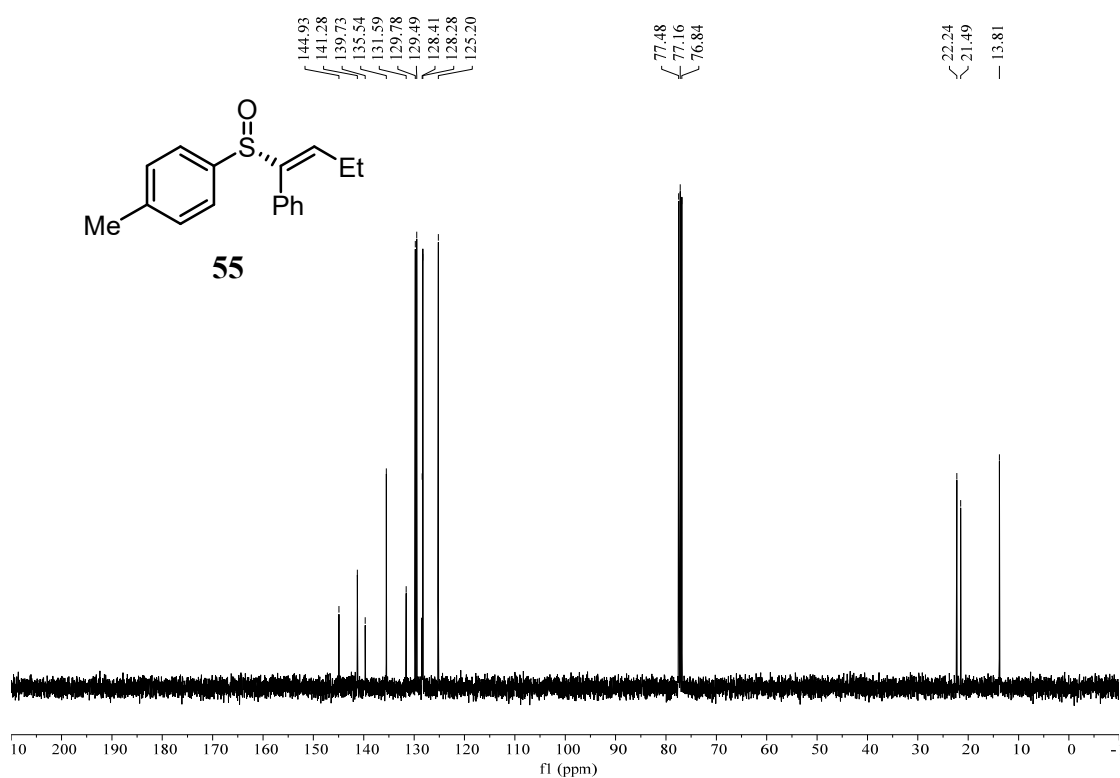
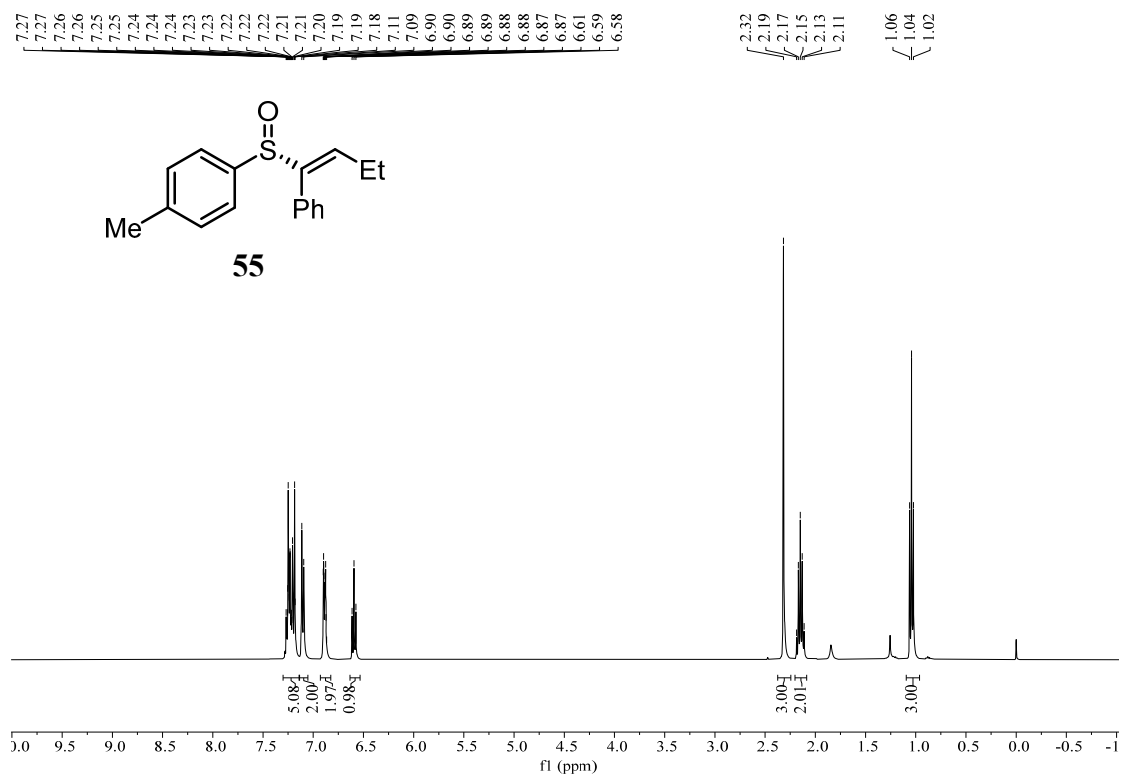


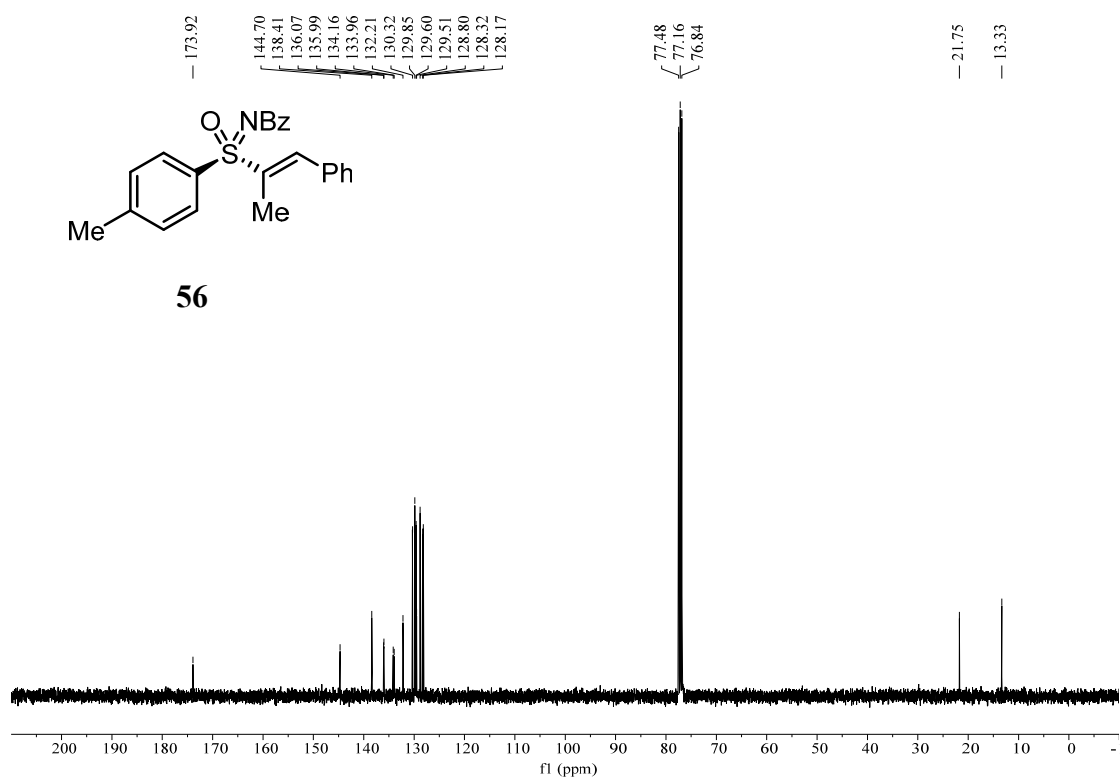
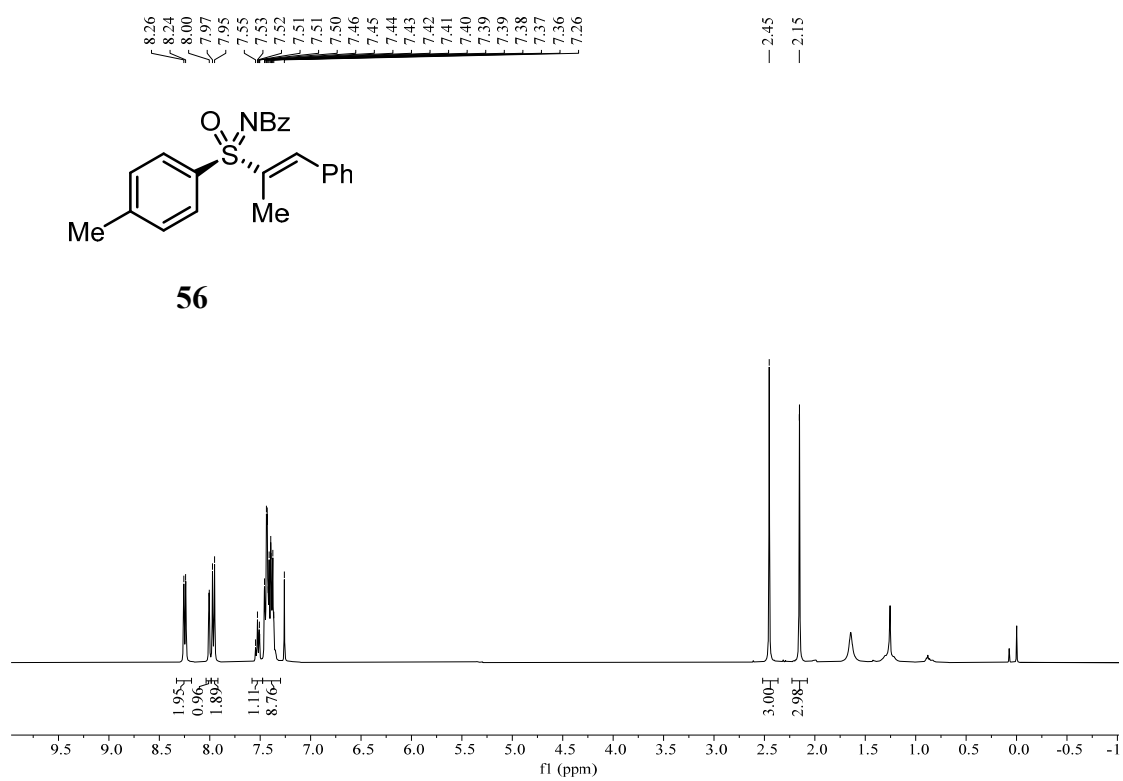


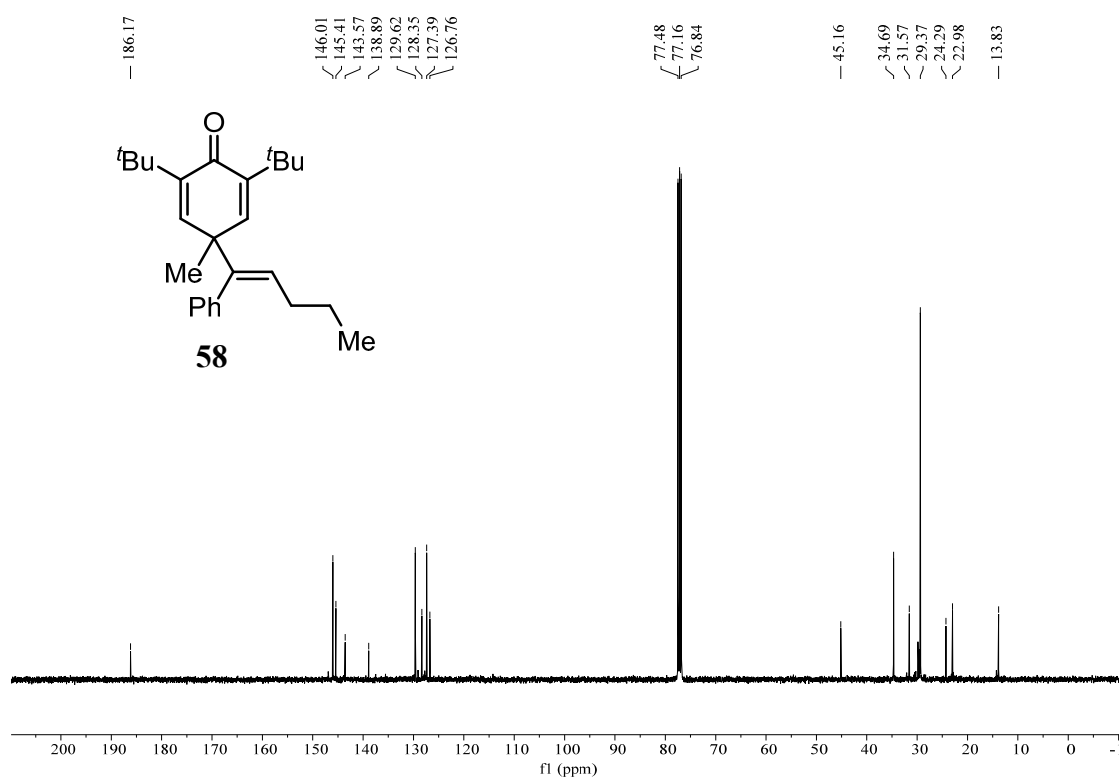
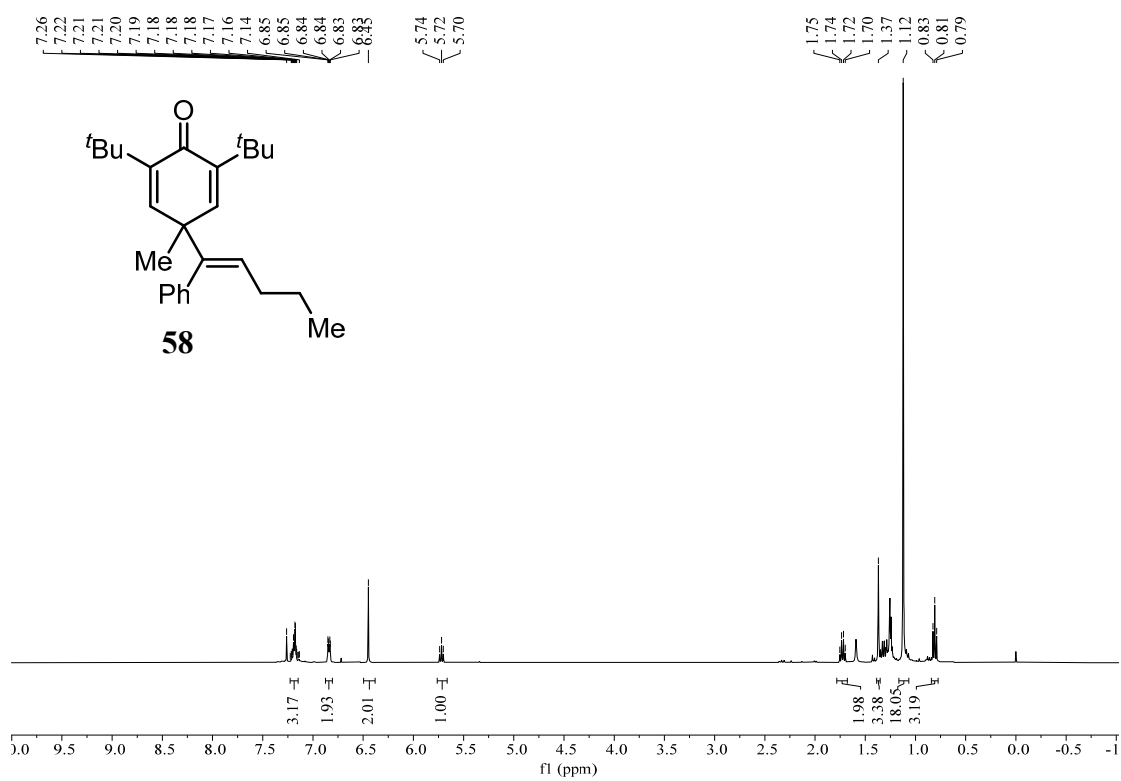




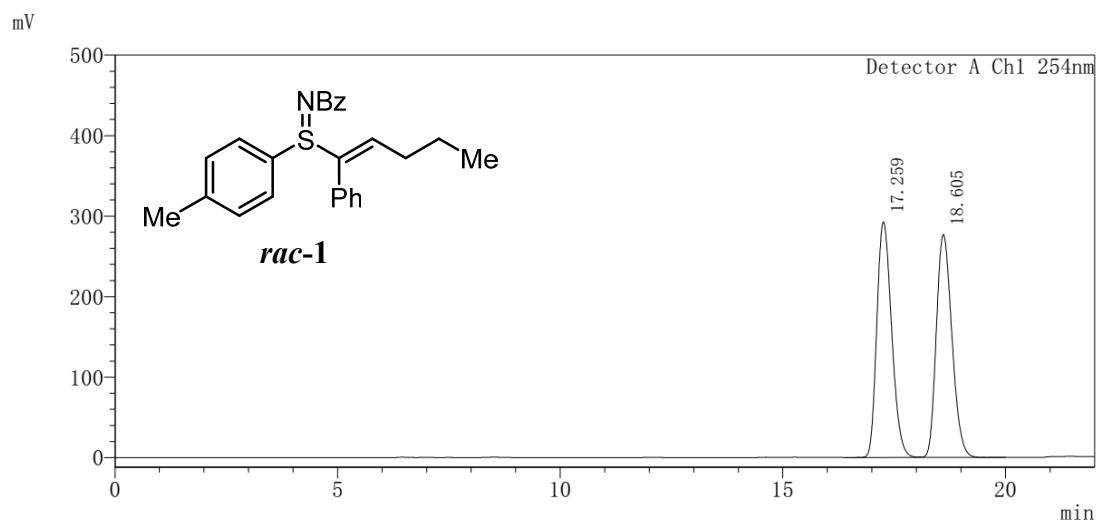








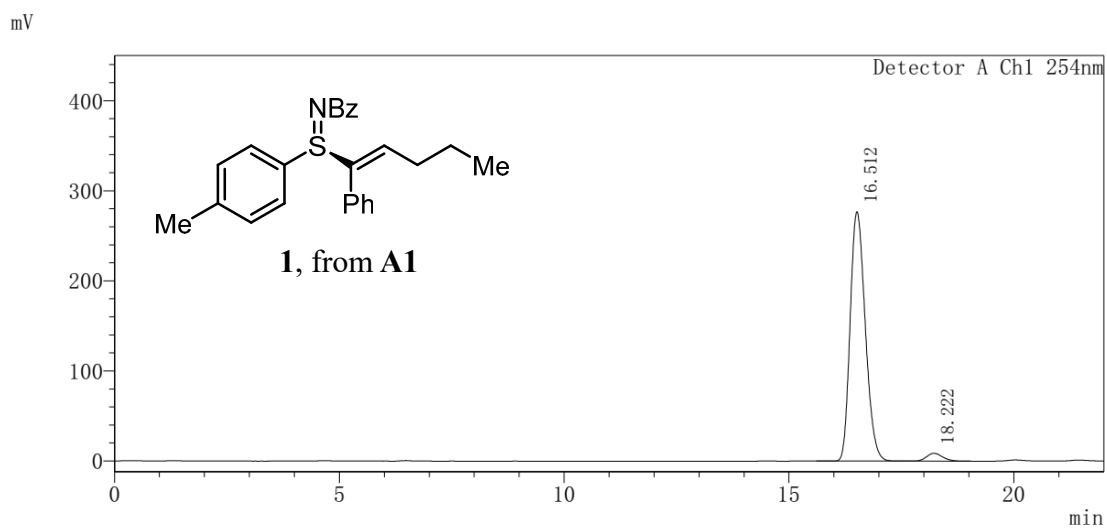
10. HPLC spectra



Peak Table

Detector A Ch1 254nm

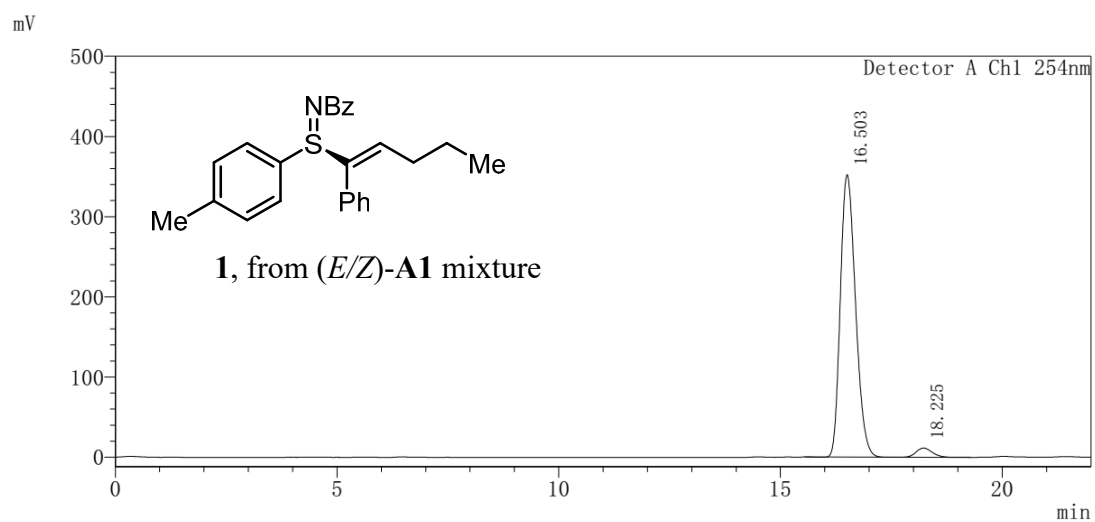
Peak#	Ret. Time	Area	Area%
1	17.259	6766430	50.140
2	18.605	6728695	49.860



Peak Table

Detector A Ch1 254nm

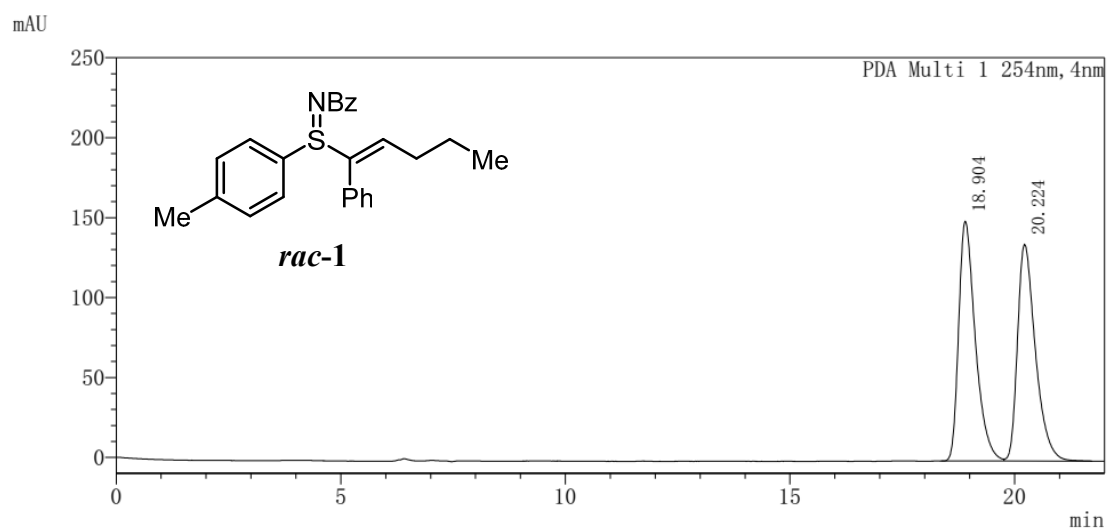
Peak#	Ret. Time	Area	Area%
1	16.512	6471579	96.743
2	18.222	217895	3.257



Peak Table

Detector A Ch1 254nm

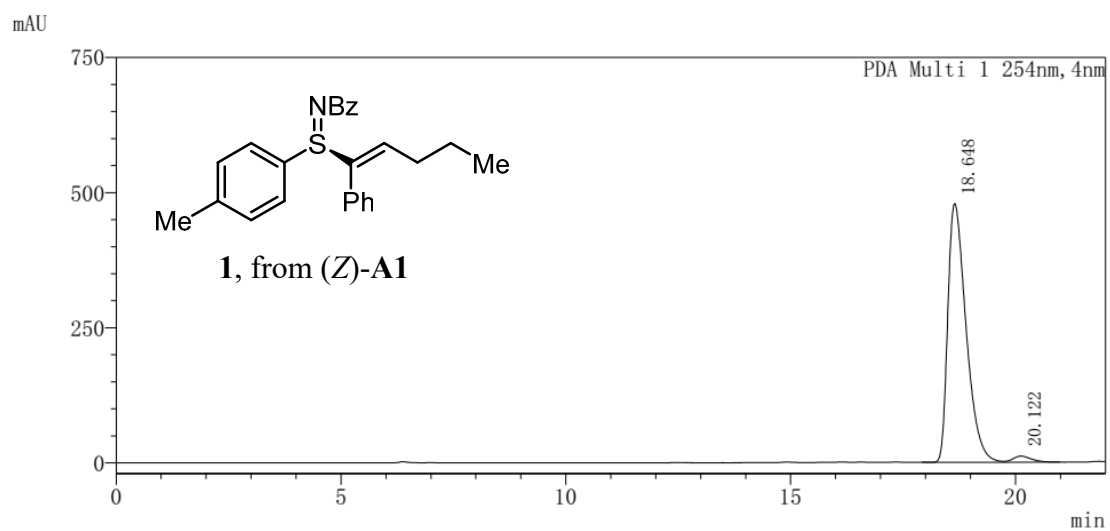
Peak#	Ret. Time	Area	Area%
1	16.503	8371993	96.604
2	18.225	294337	3.396



Peak Table

PDA Ch1 254nm

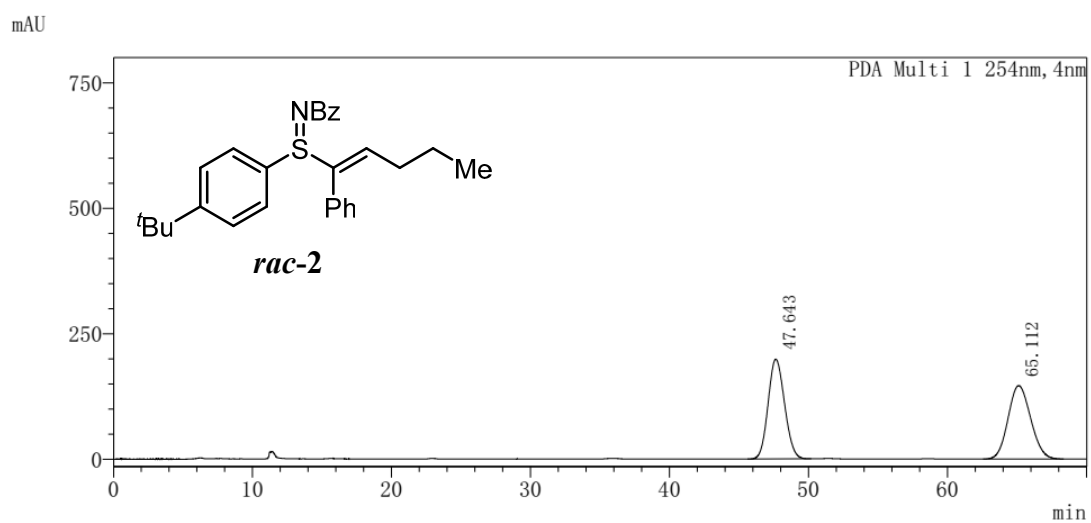
Peak#	Ret. Time	Area	Area%
1	18.904	3883048	50.686
2	20.224	3777904	49.314



Peak Table

PDA Ch1 254nm

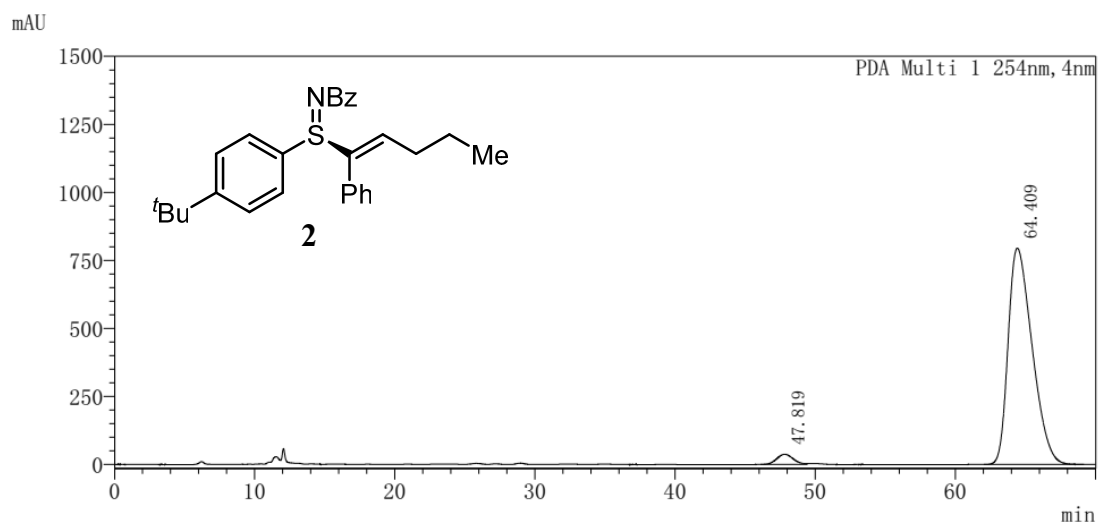
Peak#	Ret. Time	Area	Area%
1	18.648	13527052	97.619
2	20.122	329995	2.381



Peak Table

PDA Ch1 254nm

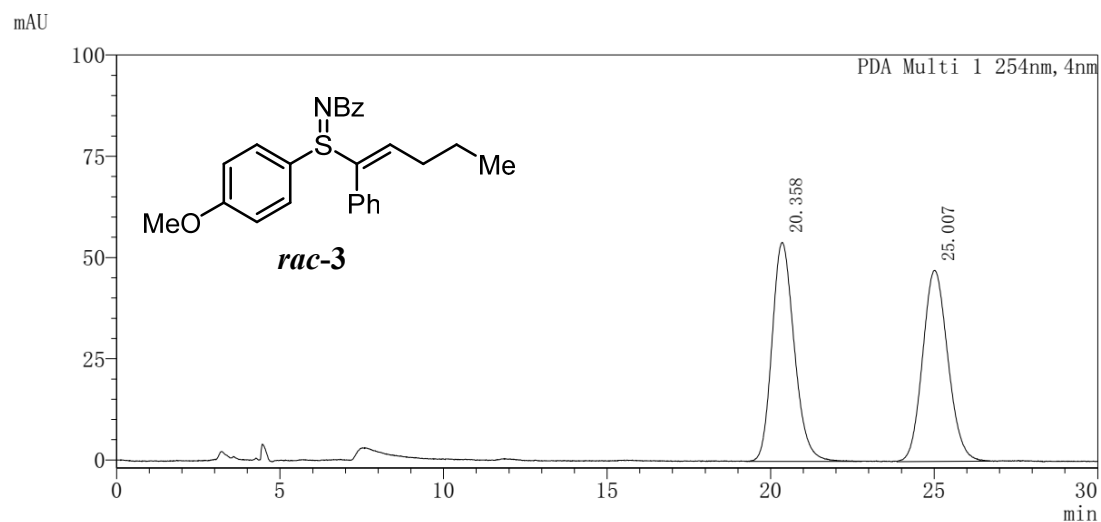
Peak#	Ret. Time	Area	Area%
1	47.643	16392606	49.807
2	65.112	16519659	50.193



Peak Table

PDA Ch1 254nm

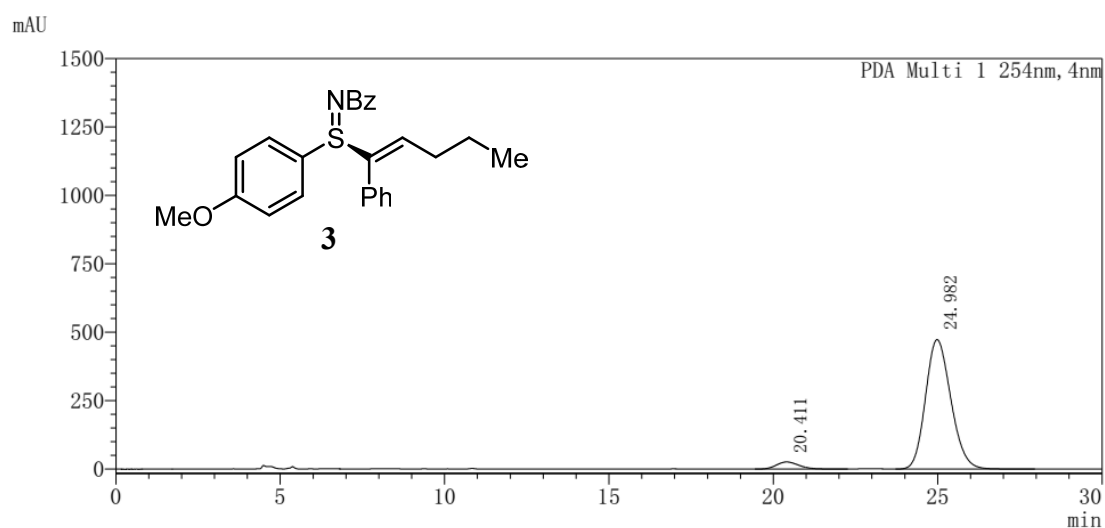
Peak#	Ret. Time	Area	Area%
1	47.819	3130038	3.205
2	64.409	94544945	96.795



Peak Table

PDA Ch1 254nm

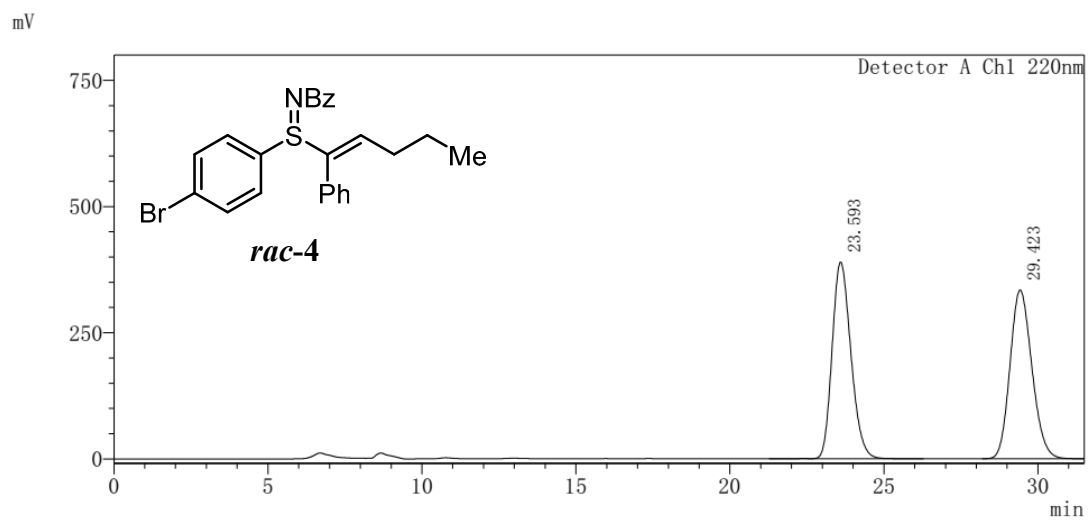
Peak#	Ret. Time	Area	Area%
1	20.358	2555097	50.209
2	25.007	2533807	49.791



Peak Table

PDA Ch1 254nm

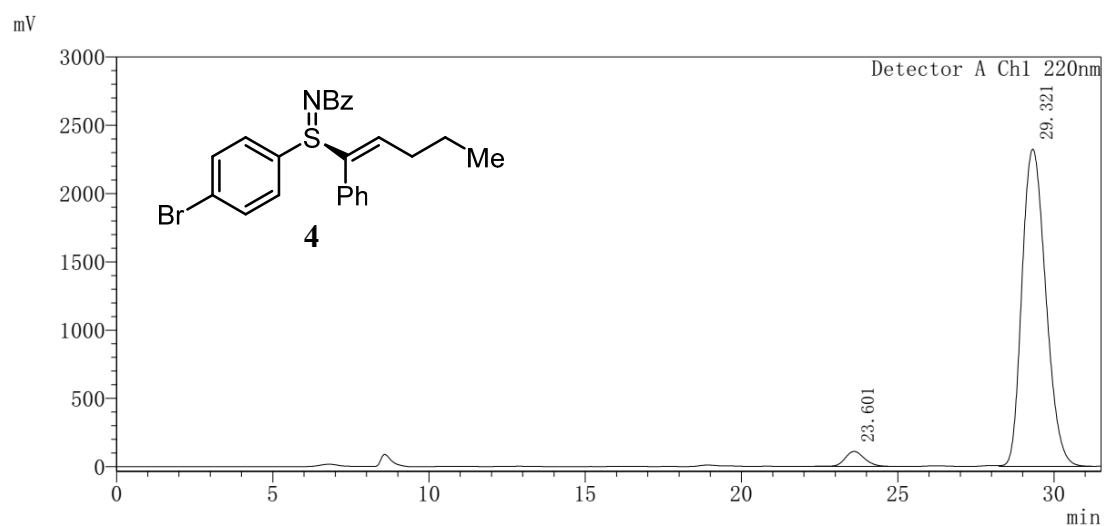
Peak#	Ret. Time	Area	Area%
1	20.411	1236451	4.652
2	24.982	25344794	95.348



Peak Table

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Area%
1	23.593	16562595	50.020
2	29.423	16549469	49.980

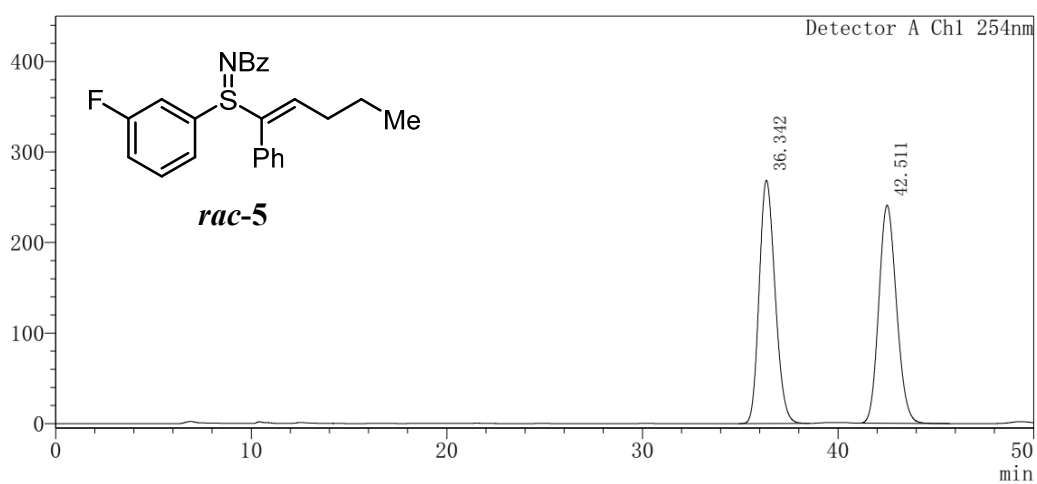


Peak Table

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Area%
1	23.601	4824836	3.771
2	29.321	123117418	96.229

mV

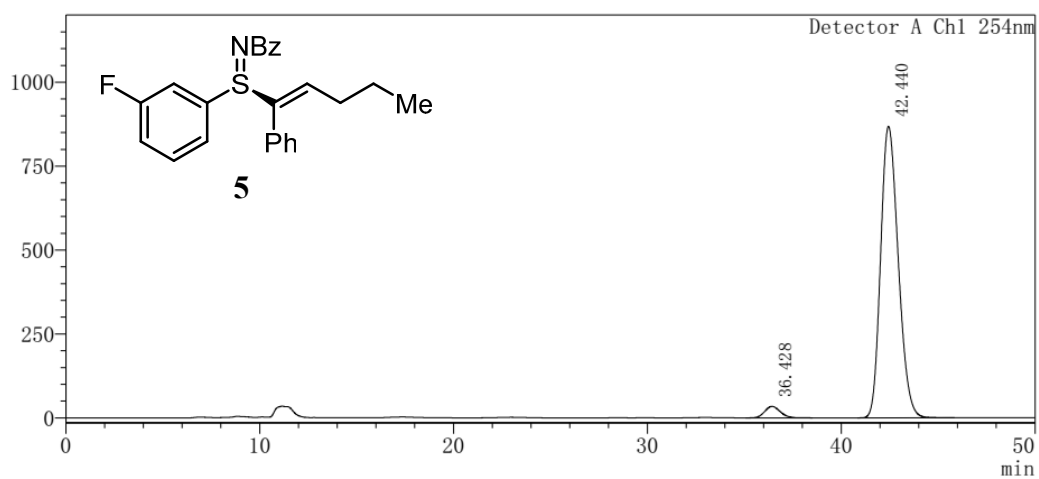


Peak Table

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	36.342	15161786	49.983
2	42.511	15172348	50.017

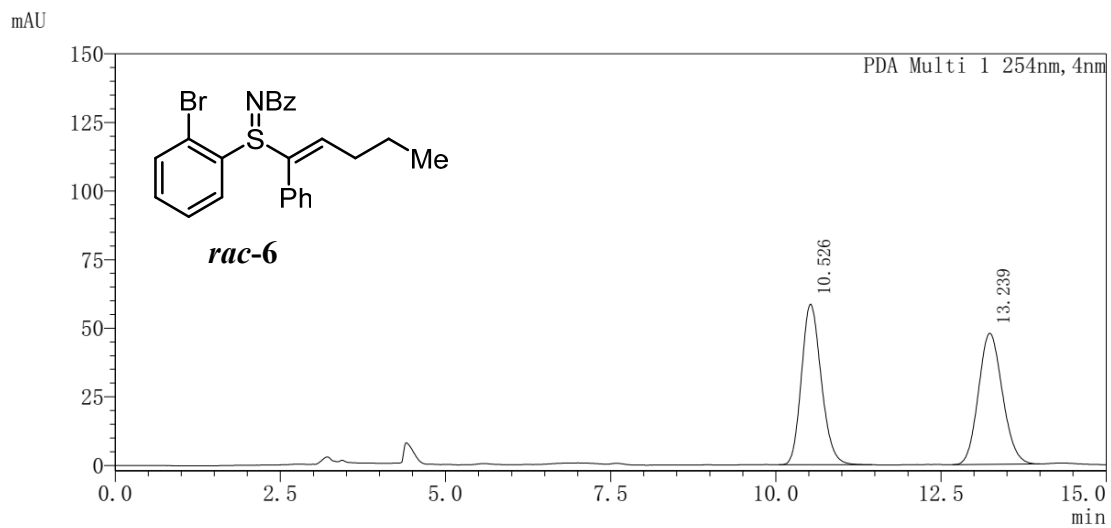
mV



Peak Table

Detector A Ch1 254nm

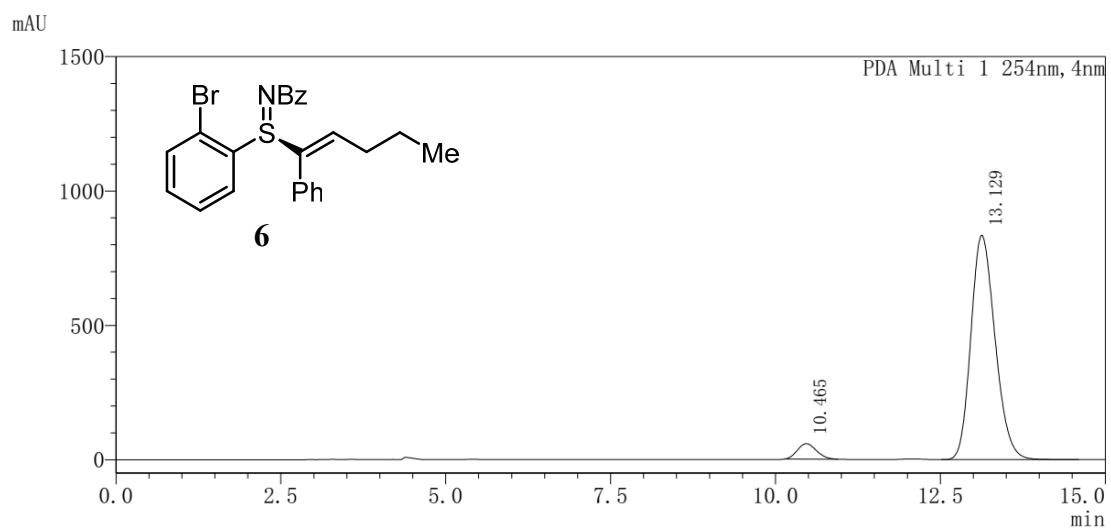
Peak#	Ret. Time	Area	Area%
1	36.428	1898712	3.347
2	42.440	54829972	96.653



Peak Table

PDA Ch1 254nm

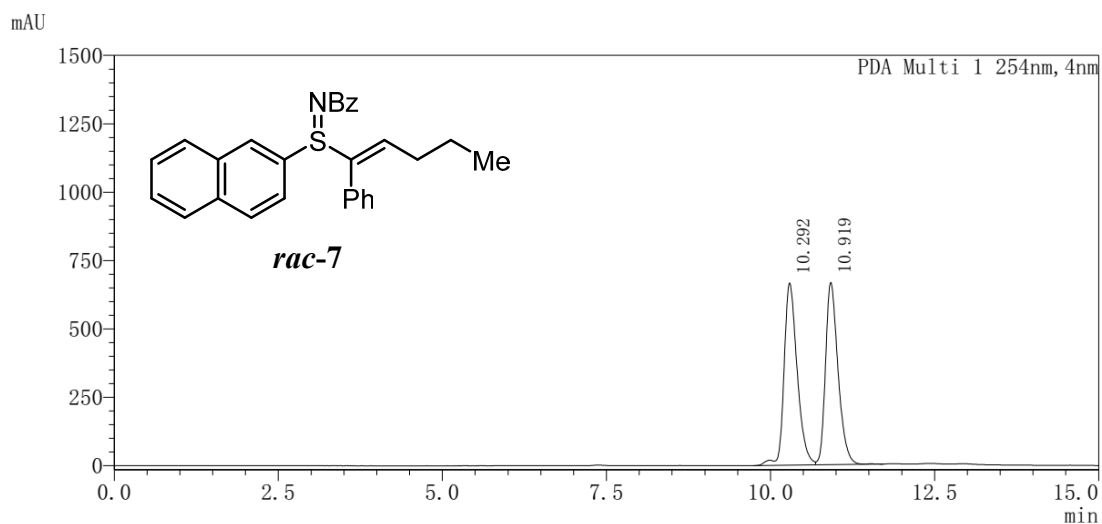
Peak#	Ret. Time	Area	Area%
1	10.526	1223948	50.313
2	13.239	1208717	49.687



Peak Table

PDA Ch1 254nm

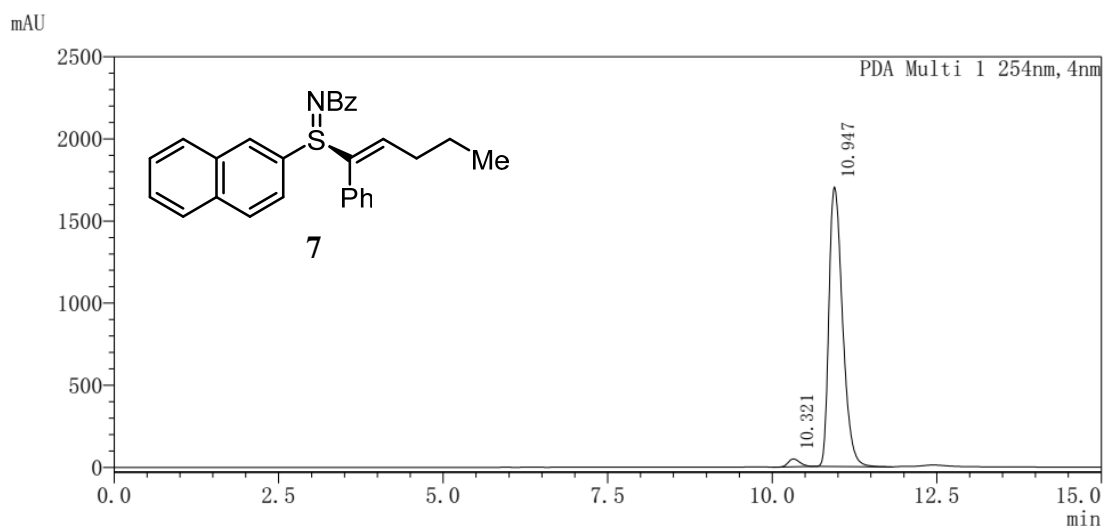
Peak#	Ret. Time	Area	Area%
1	10.465	1165640	5.185
2	13.129	21316639	94.815



Peak Table

PDA Ch1 254nm

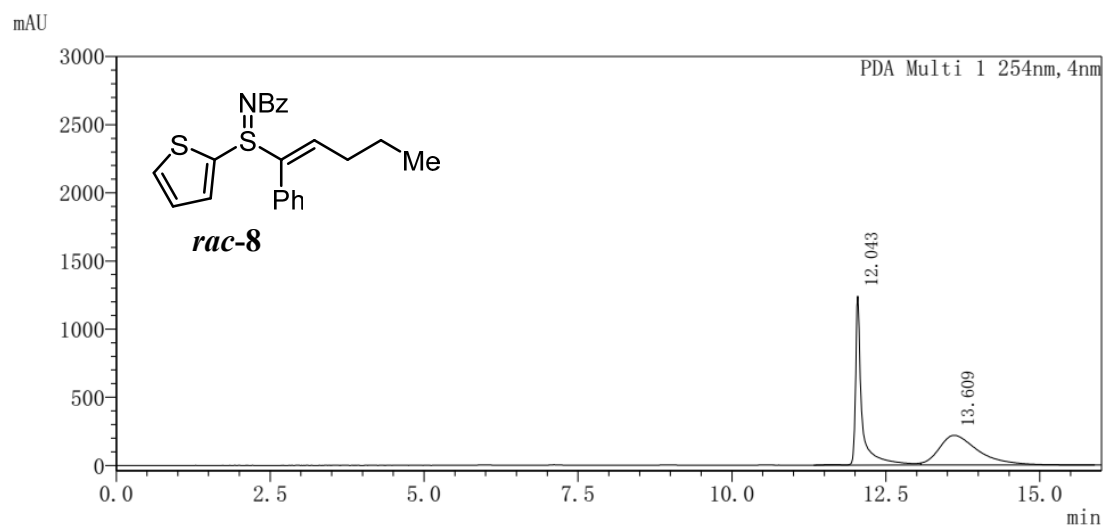
Peak#	Ret. Time	Area	Area%
1	10.292	9274278	51.135
2	10.919	8862397	48.865



Peak Table

PDA Ch1 254nm

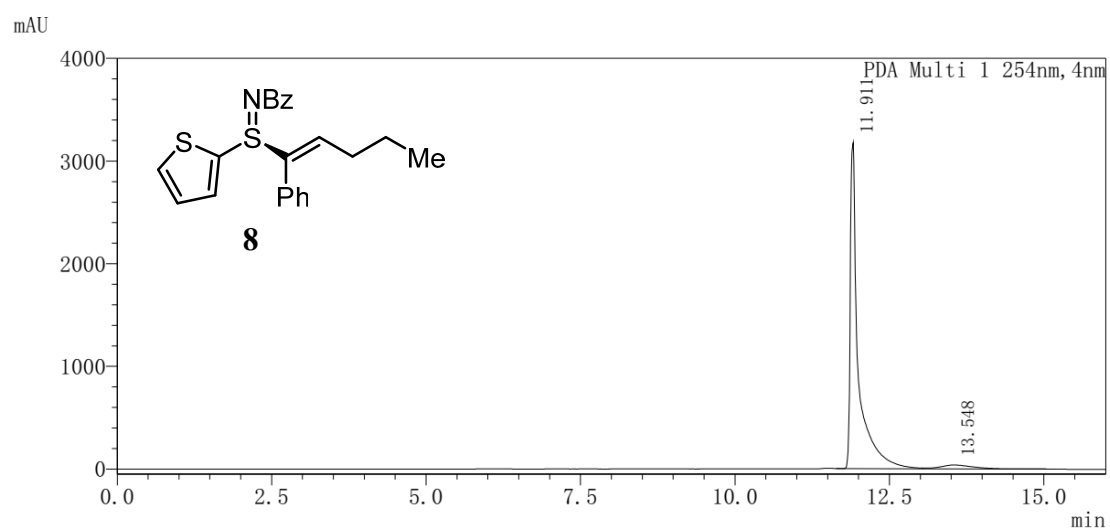
Peak#	Ret. Time	Area	Area%
1	10.321	592266	2.356
2	10.947	24547939	97.644



Peak Table

PDA Ch1 254nm

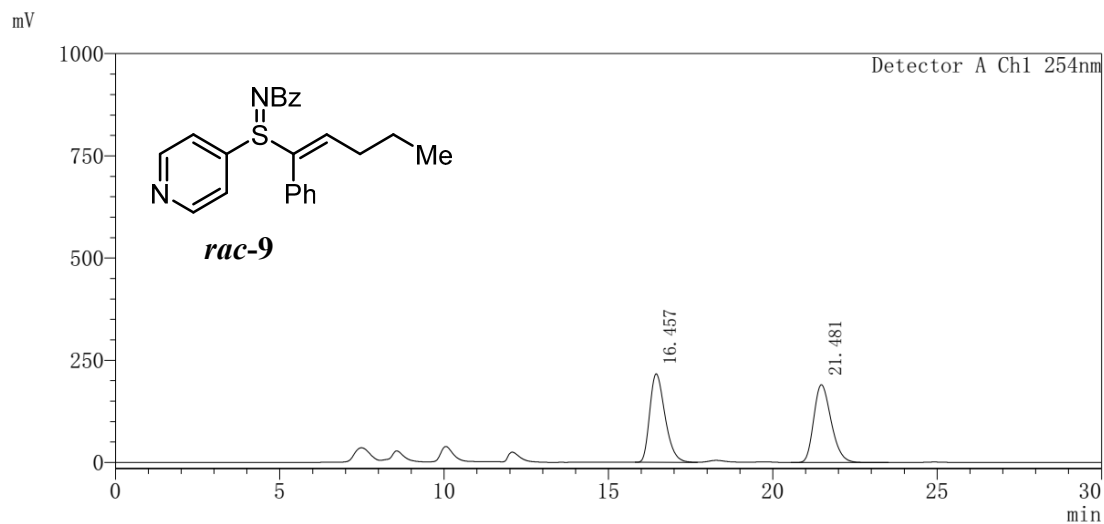
Peak#	Ret. Time	Area	Area%
1	12.043	9142317	48.496
2	13.609	9709469	51.504



Peak Table

PDA Ch1 254nm

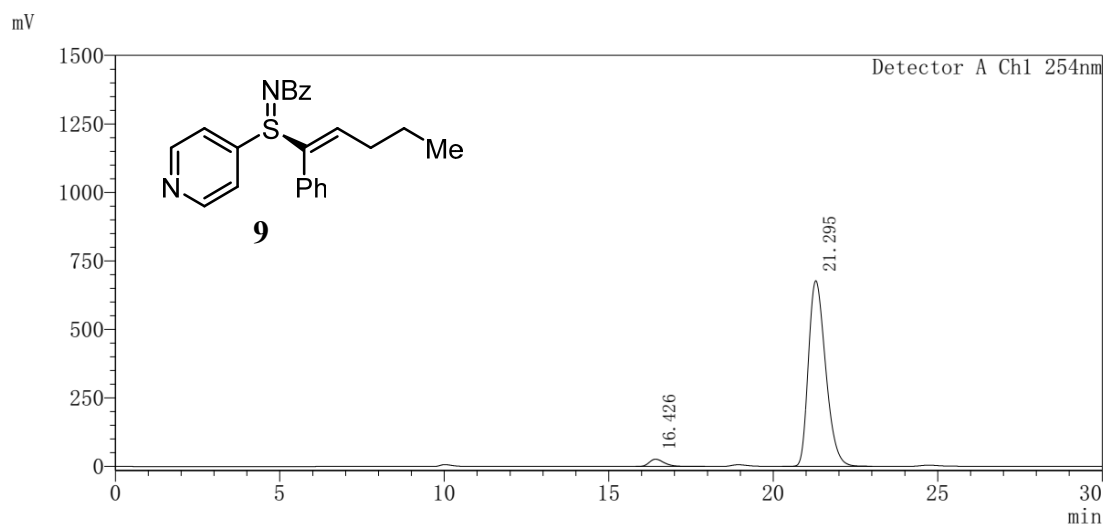
Peak#	Ret. Time	Area	Area%
1	11.911	28830790	95.149
2	13.548	1470001	4.851



Peak Table

Detector A Ch1 254nm

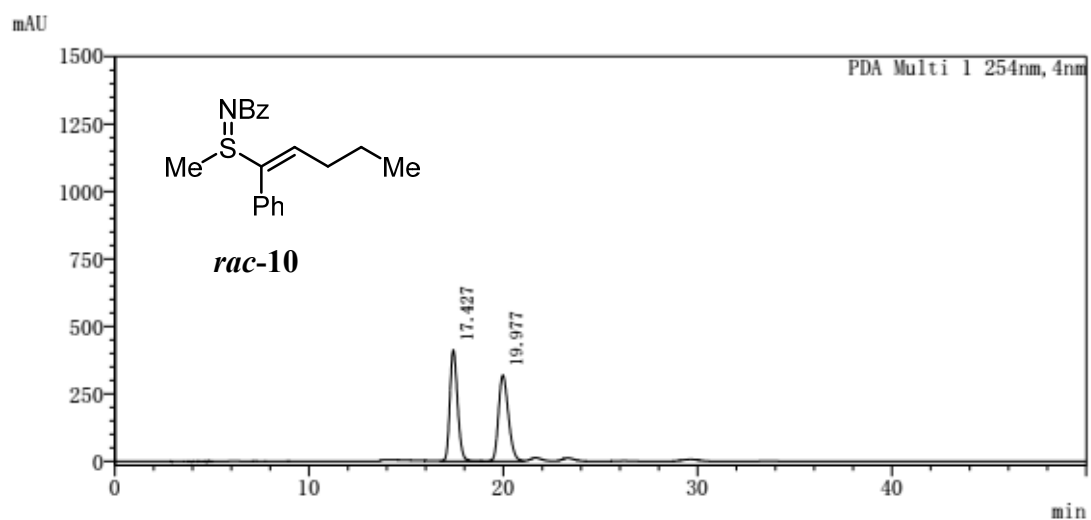
Peak#	Ret. Time	Area	Area%
1	16.457	7024768	50.039
2	21.481	7013815	49.961



Peak Table

Detector A Ch1 254nm

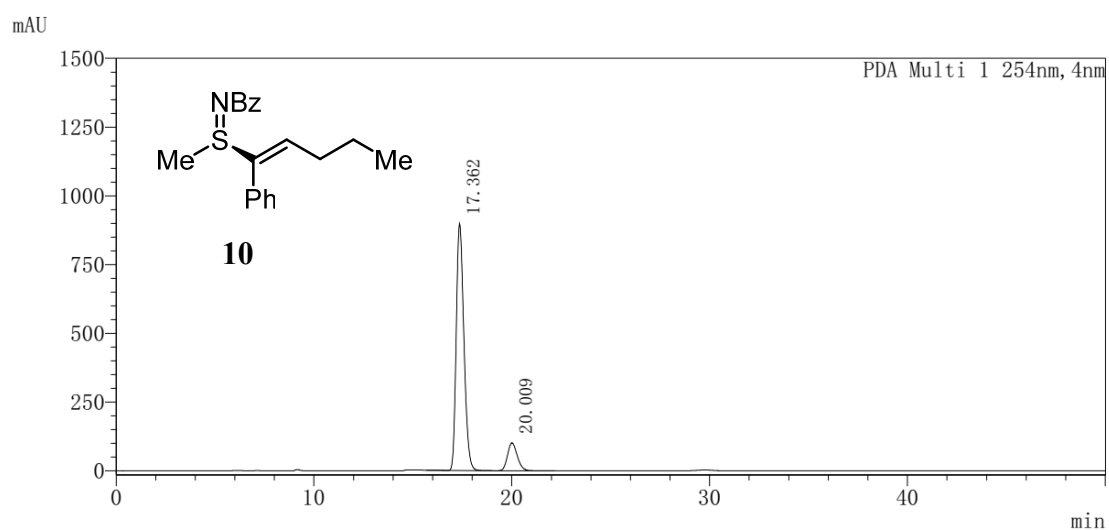
Peak#	Ret. Time	Area	Area%
1	16.426	832065	3.260
2	21.295	24689064	96.740



Peak Table

PDA Ch1 254nm

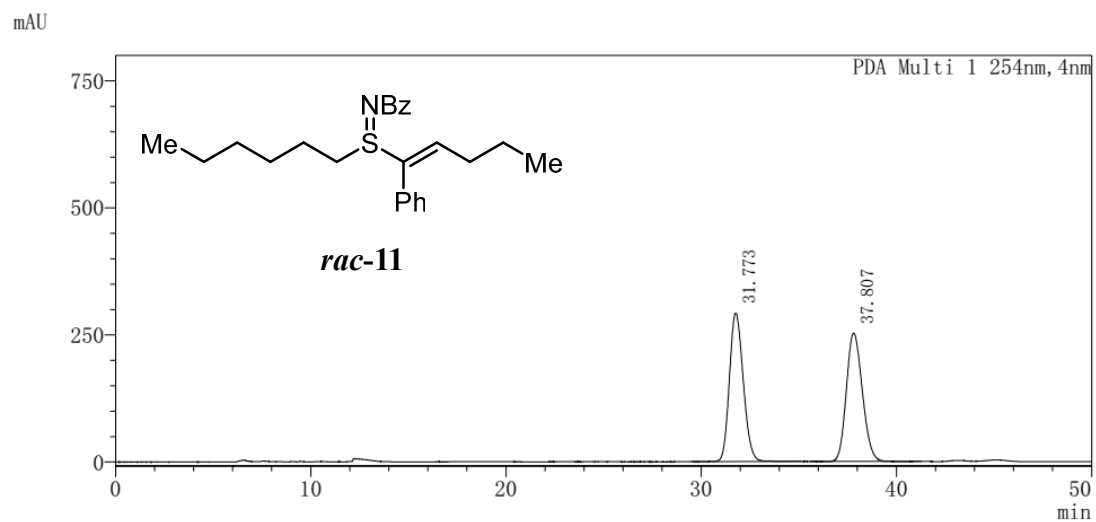
Peak#	Ret. Time	Area	Area%
1	17.427	11338931	50.167
2	19.977	11263642	49.833



Peak Table

PDA Ch1 254nm

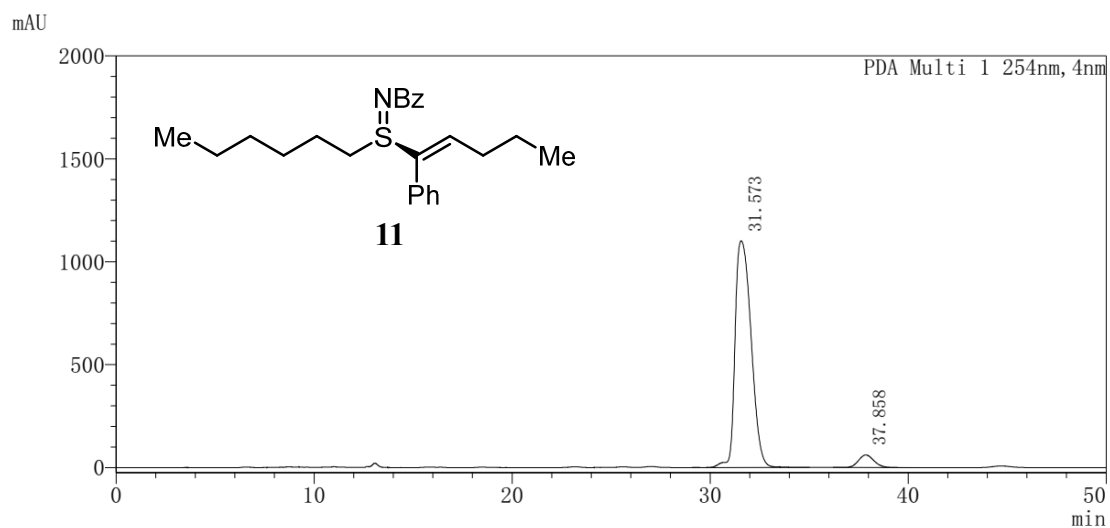
Peak#	Ret. Time	Area	Area%
1	17.362	24073833	87.494
2	20.009	3441101	12.506



Peak Table

PDA Ch1 254nm

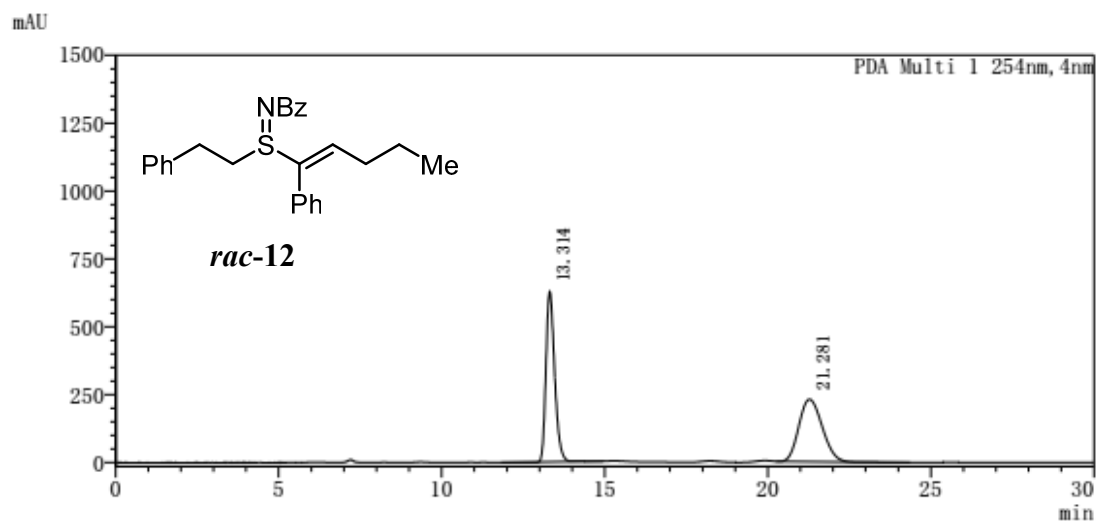
Peak#	Ret. Time	Area	Area%
1	31.773	14450419	50.235
2	37.807	14315468	49.765



Peak Table

PDA Ch1 254nm

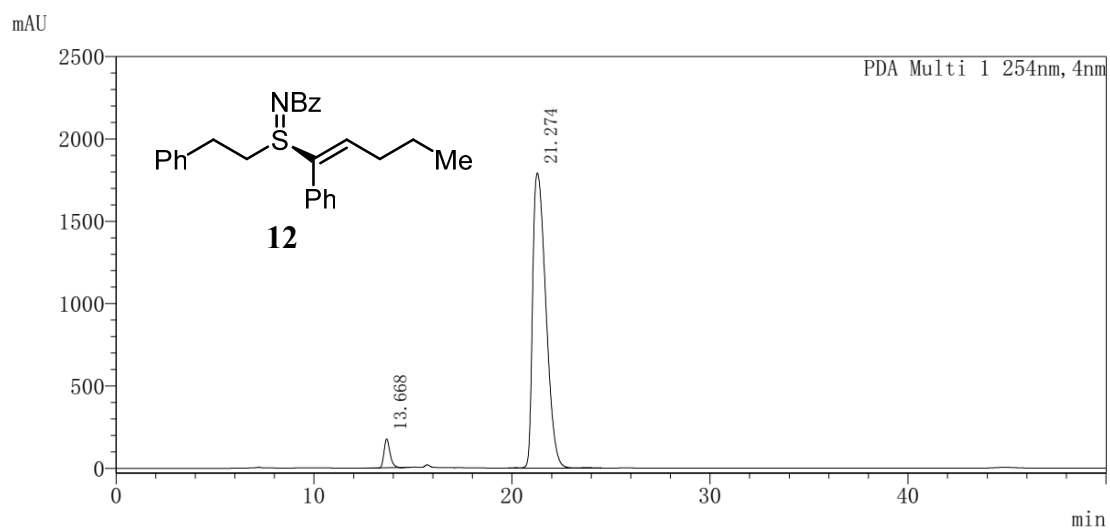
Peak#	Ret. Time	Area	Area%
1	31.573	62009766	94.997
2	37.858	3265917	5.003



Peak Table

PDA Ch1 254nm

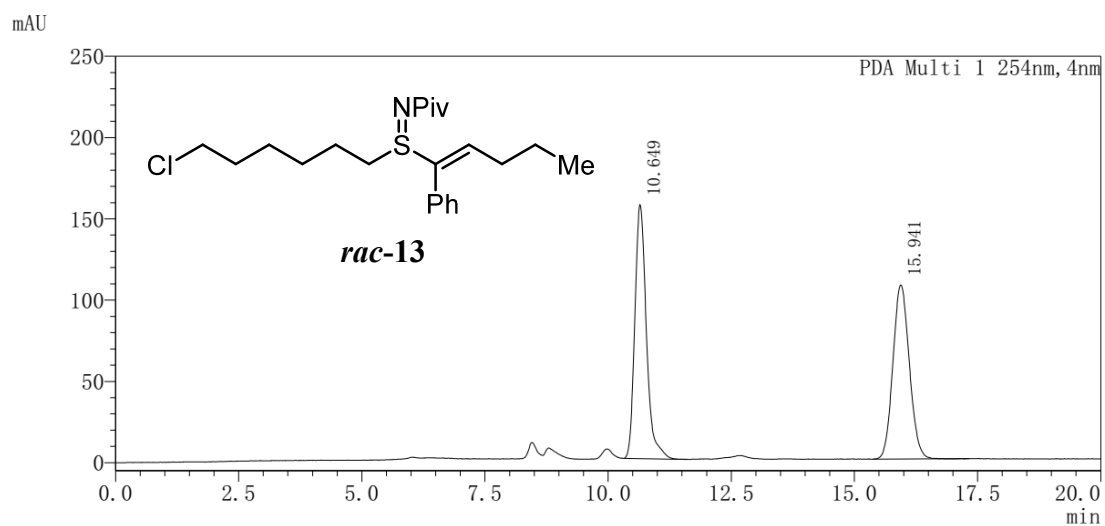
Peak#	Ret. Time	Area	Area%
1	13.314	11613905	50.531
2	21.281	11369961	49.469



Peak Table

PDA Ch1 254nm

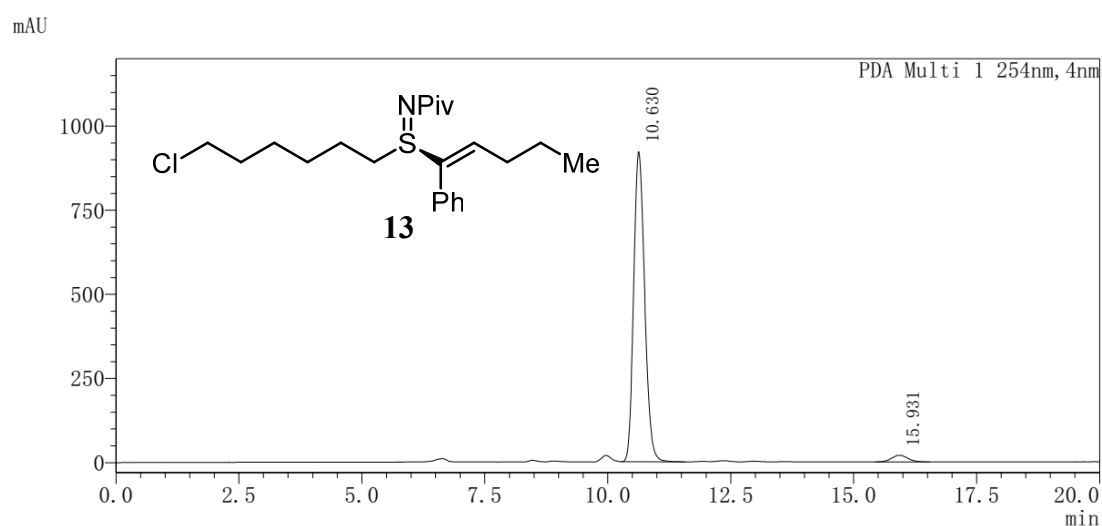
Peak#	Ret. Time	Area	Area%
1	13.668	3593777	4.134
2	21.274	83340366	95.866



Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	10.649	2551324	50.573
2	15.941	2493514	49.427

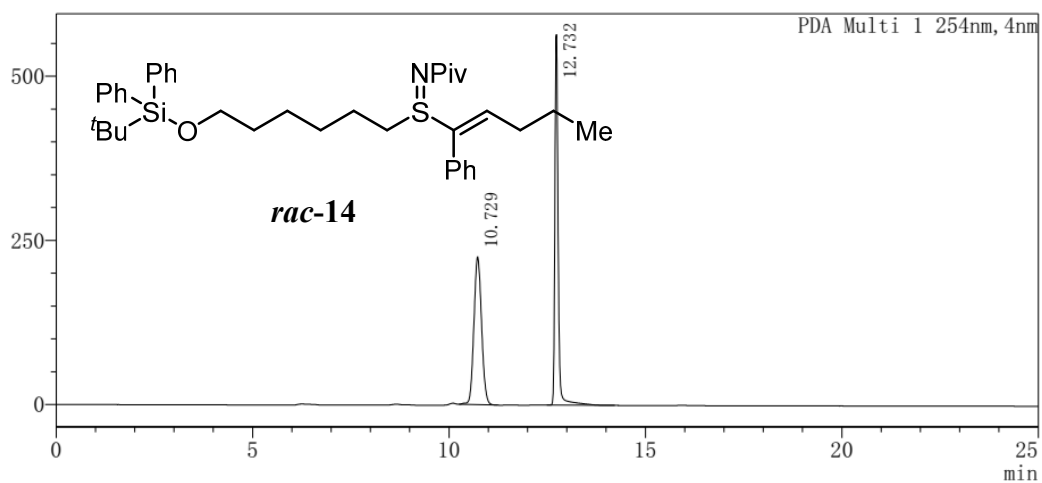


Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	10.630	14358724	96.922
2	15.931	456014	3.078

mAU

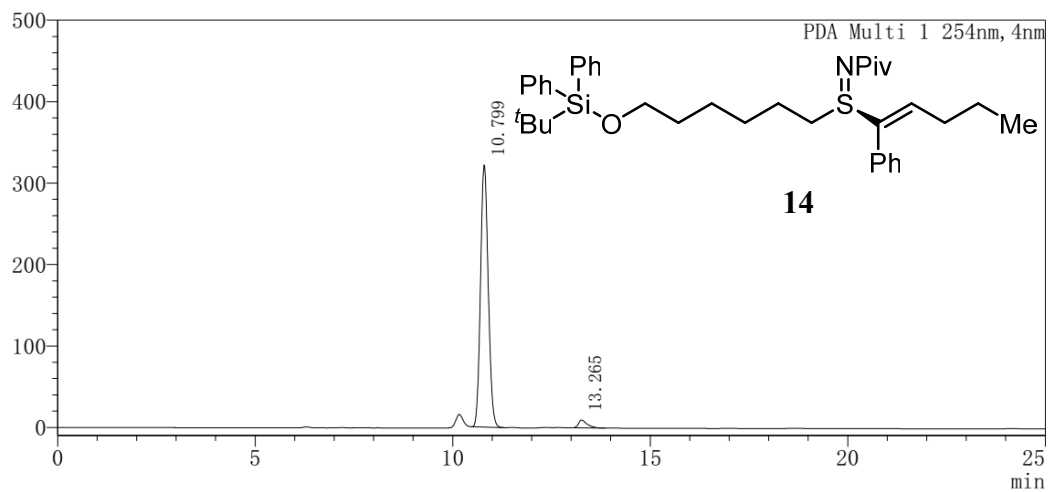


Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	10.729	3063852	48.553
2	12.732	3246478	51.447

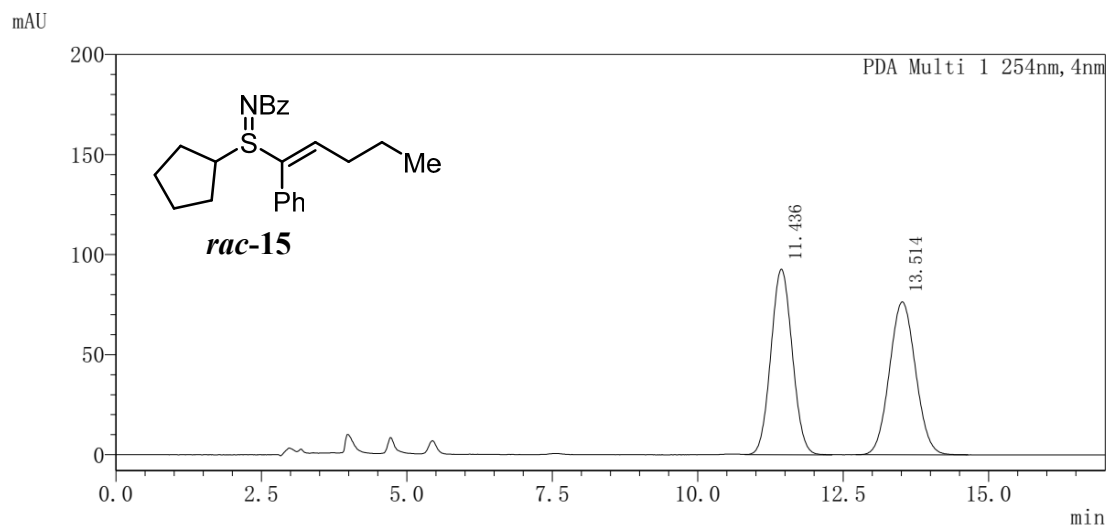
mAU



Peak Table

PDA Ch1 254nm

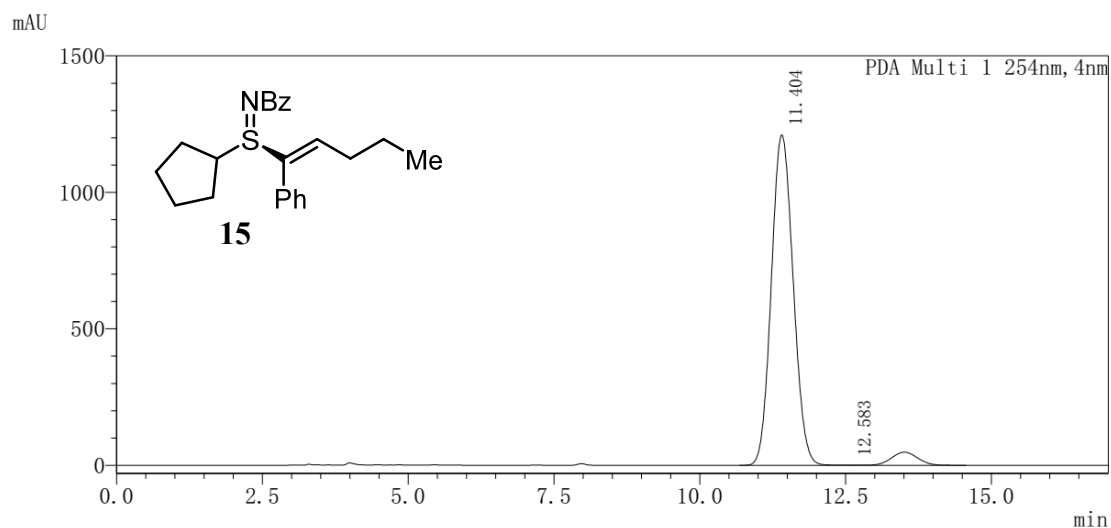
Peak#	Ret. Time	Area	Area%
1	10.799	4380107	96.840
2	13.265	142928	3.160



Peak Table

PDA Ch1 254nm

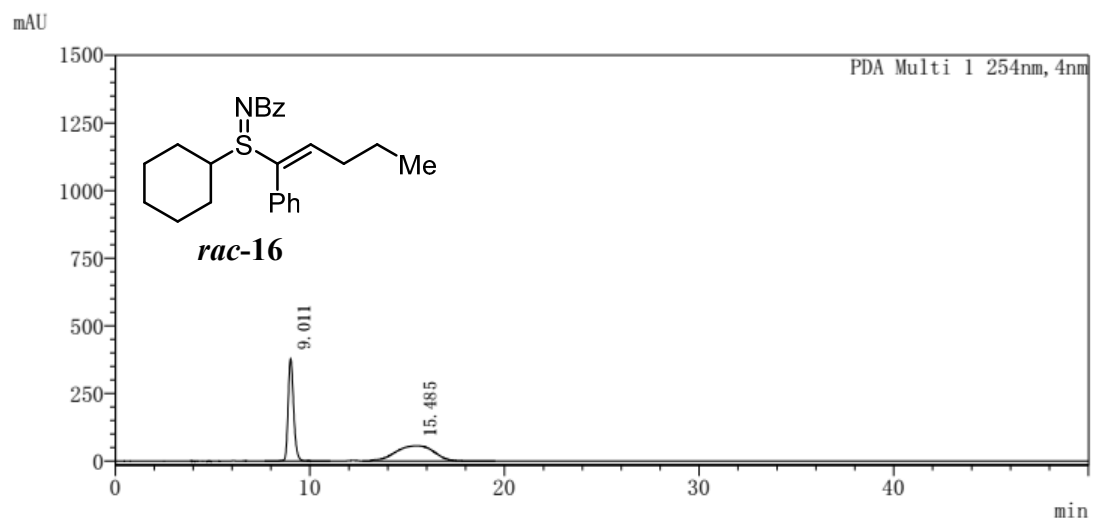
Peak#	Ret. Time	Area	Area%
1	11.436	2371509	49.777
2	13.514	2392720	50.223



Peak Table

PDA Ch1 254nm

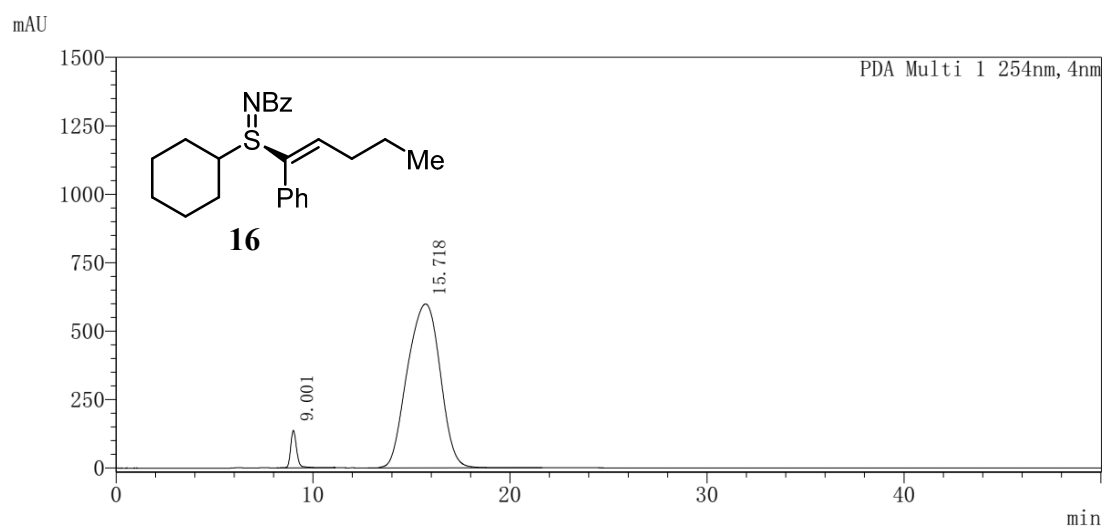
Peak#	Ret. Time	Area	Area%
1	11.404	31548527	95.385
2	12.583	1526463	4.615



Peak Table

PDA Ch1 254nm

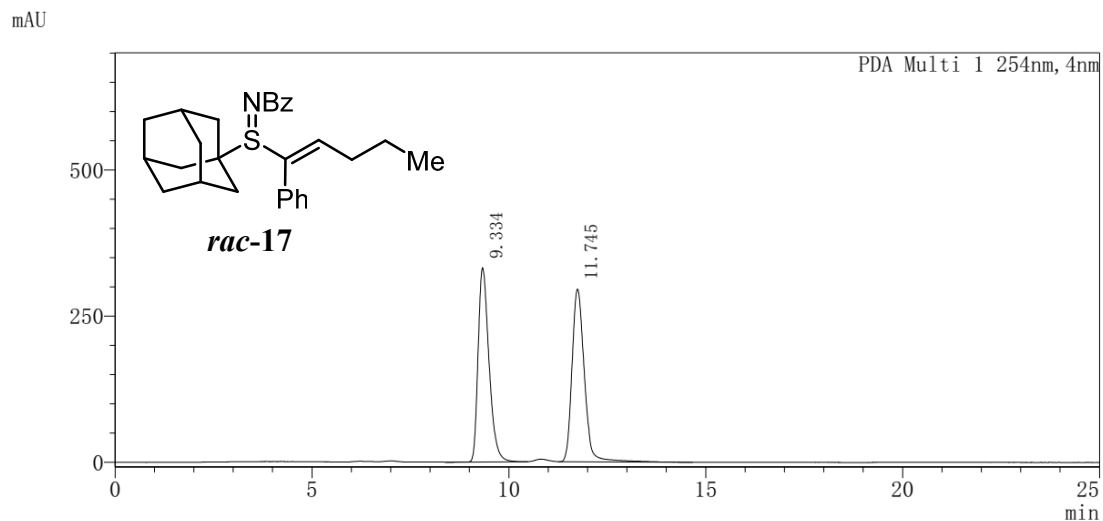
Peak#	Ret. Time	Area	Area%
1	9.011	7402913	49.033
2	15.485	7694852	50.967



Peak Table

PDA Ch1 254nm

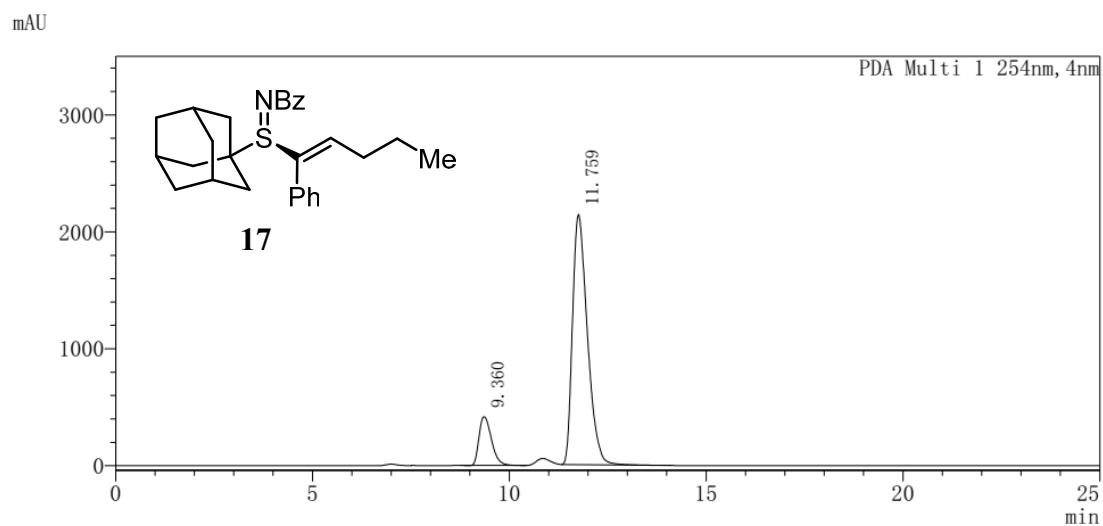
Peak#	Ret. Time	Area	Area%
1	9.001	2804850	3.880
2	15.718	69485914	96.120



Peak Table

PDA Ch1 254nm

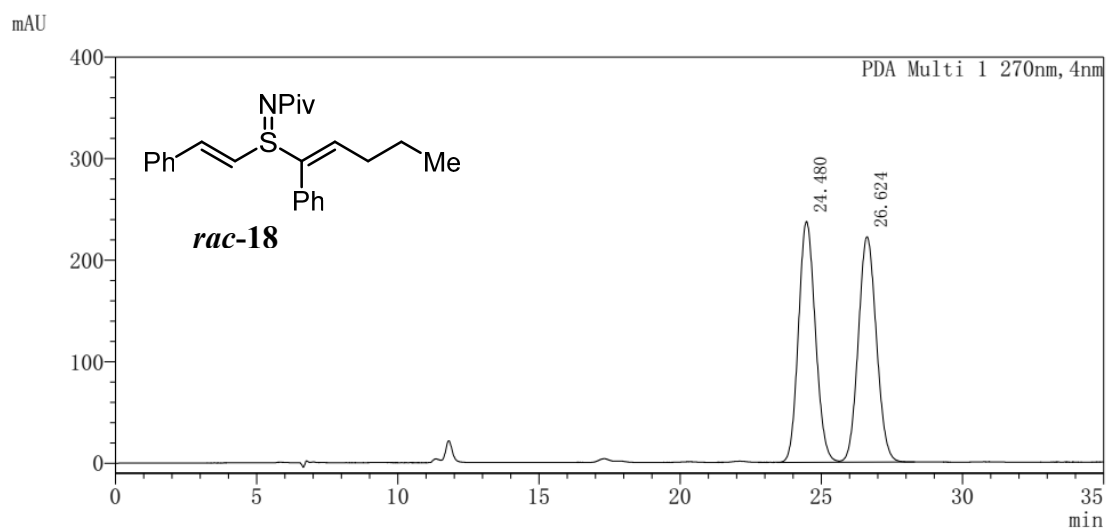
Peak#	Ret. Time	Area	Area%
1	9.334	6400589	49.851
2	11.745	6438753	50.149



Peak Table

PDA Ch1 254nm

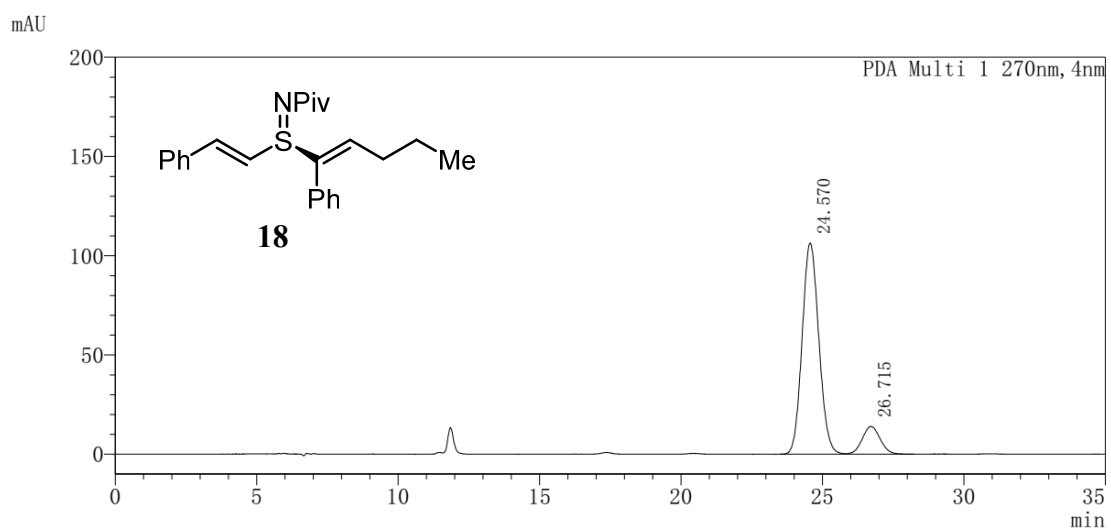
Peak#	Ret. Time	Area	Area%
1	9.360	9090541	13.915
2	11.759	56236478	86.085



Peak Table

PDA Ch1 270nm

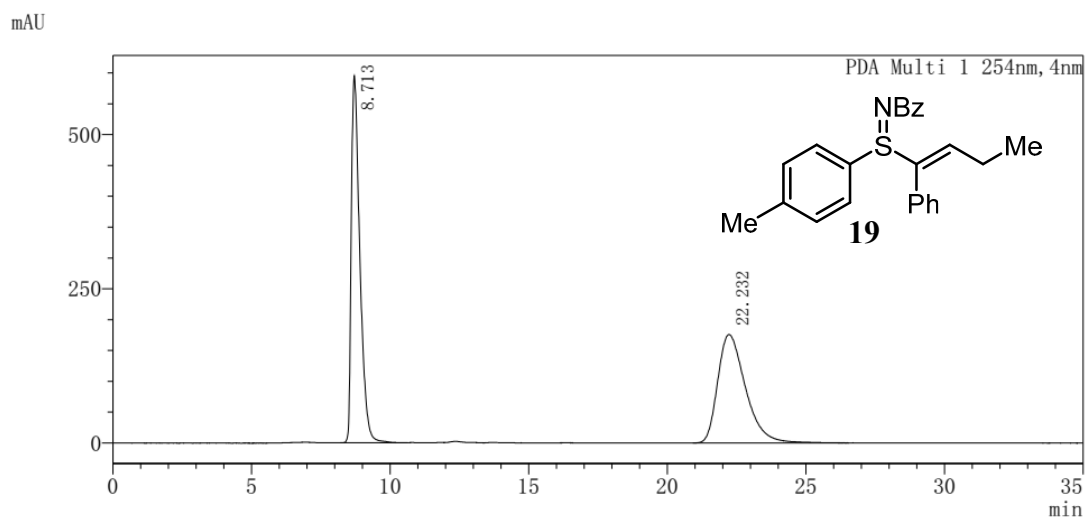
Peak#	Ret. Time	Area	Area%
1	24.480	9895987	49.939
2	26.624	9920104	50.061



Peak Table

PDA Ch1 270nm

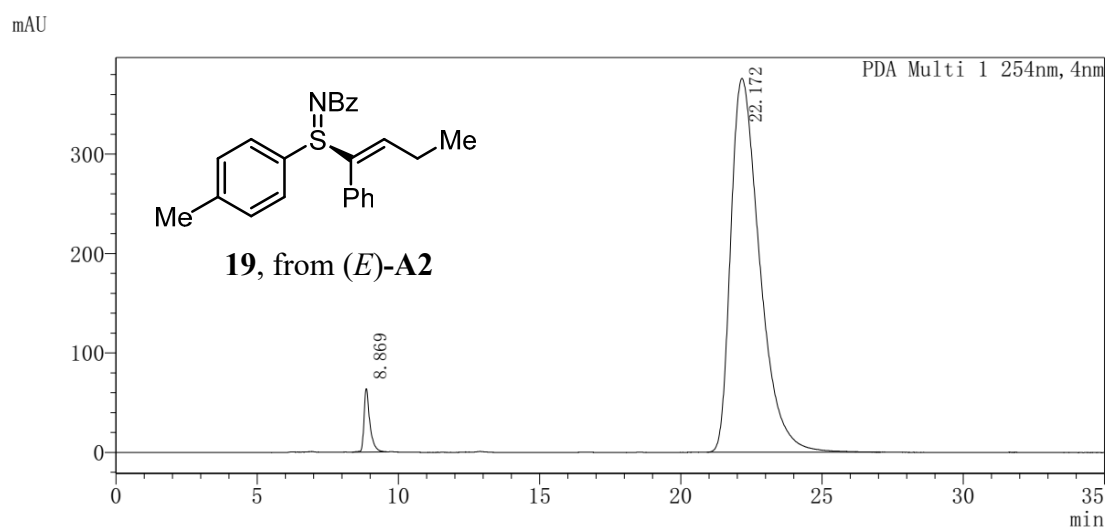
Peak#	Ret. Time	Area	Area%
1	24.570	4377027	87.659
2	26.715	616236	12.341



Peak Table

PDA Ch1 254nm

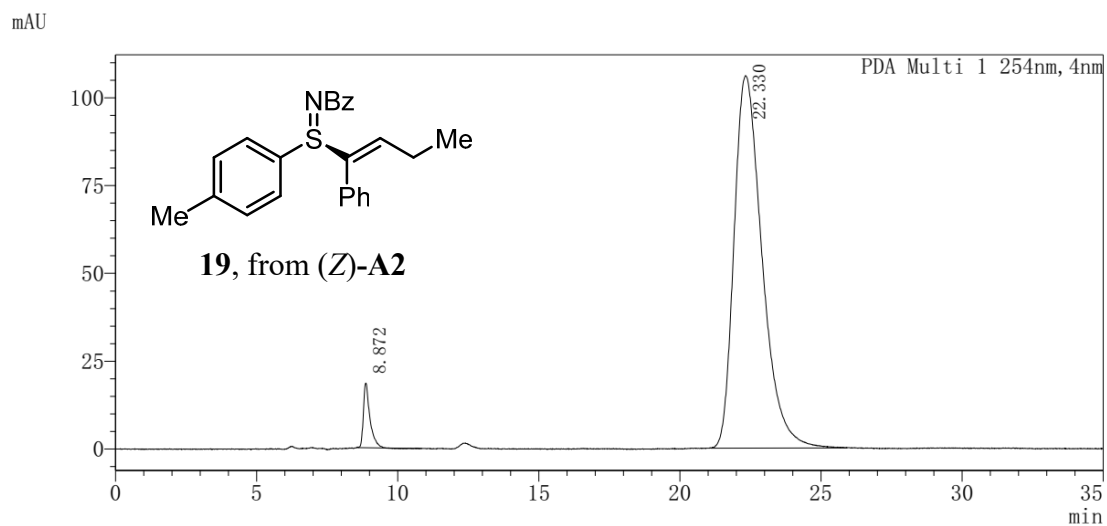
Peak#	Ret. Time	Area	Area%
1	8.713	12840895	51.094
2	22.232	12290855	48.906



Peak Table

PDA Ch1 254nm

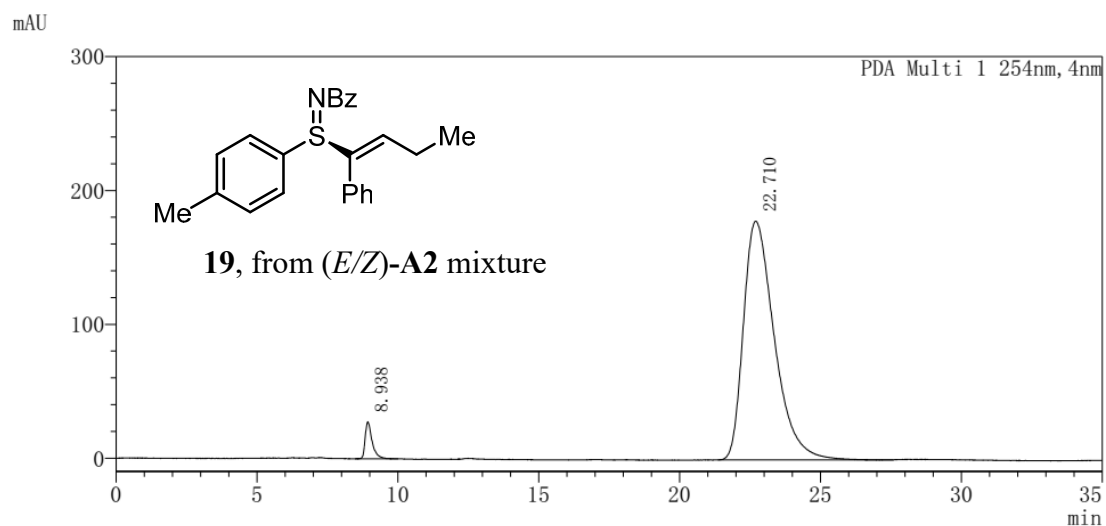
Peak#	Ret. Time	Area	Area%
1	8.869	925109	3.341
2	22.172	26766424	96.659



Peak Table

PDA Ch1 254nm

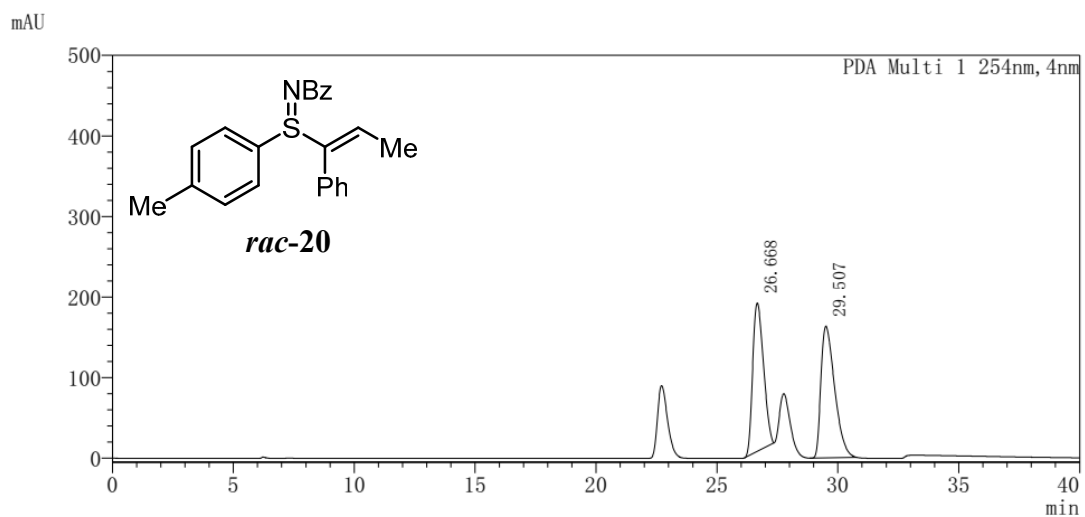
Peak#	Ret. Time	Area	Area%
1	8.872	289732	3.805
2	22.330	7325001	96.195



Peak Table

PDA Ch1 254nm

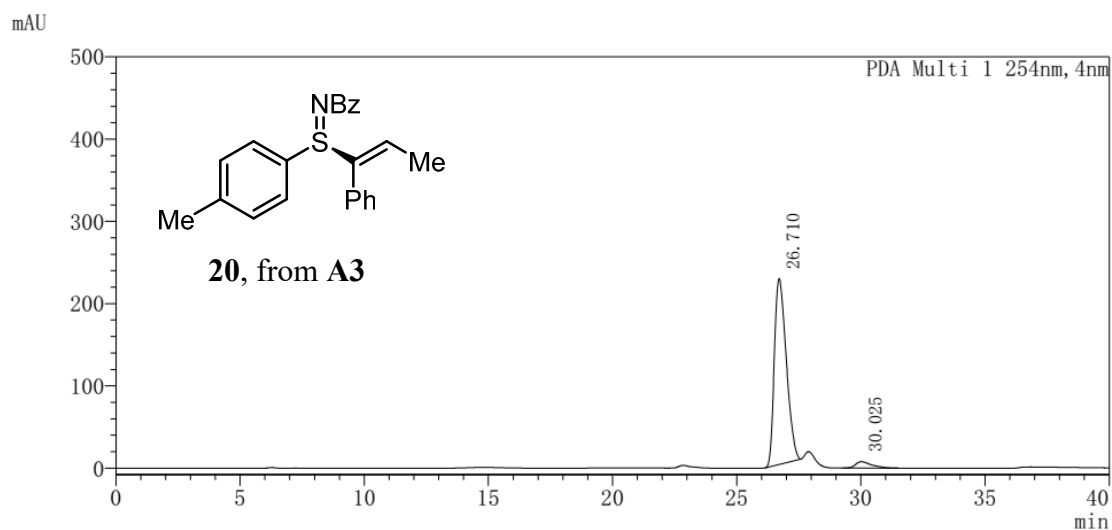
Peak#	Ret. Time	Area	Area%
1	8.938	476061	3.382
2	22.710	13601778	96.618



Peak Table

PDA Ch1 254nm

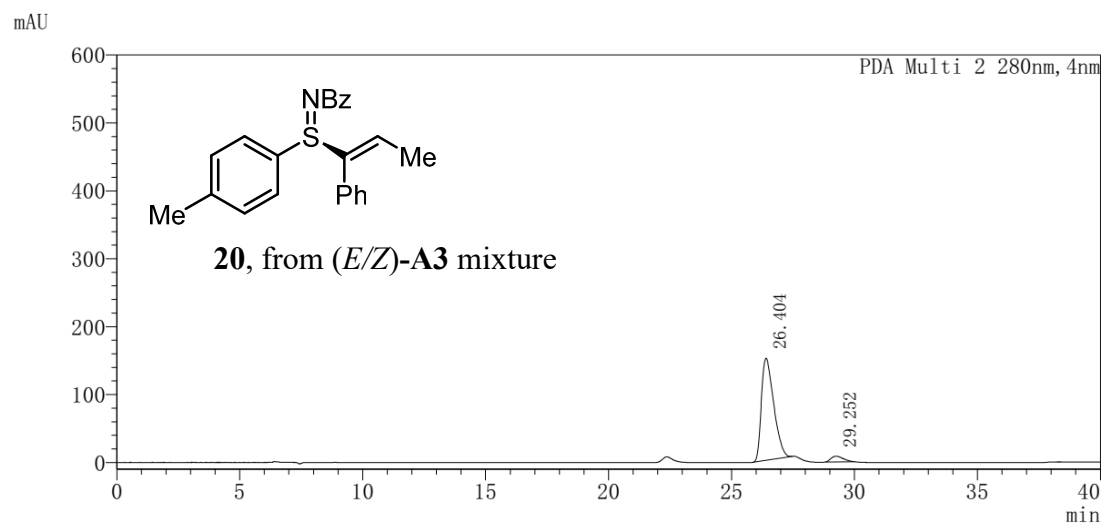
Peak#	Ret. Time	Area	Area%
1	26.668	5721407	47.000
2	29.507	6451830	53.000



Peak Table

PDA Ch1 254nm

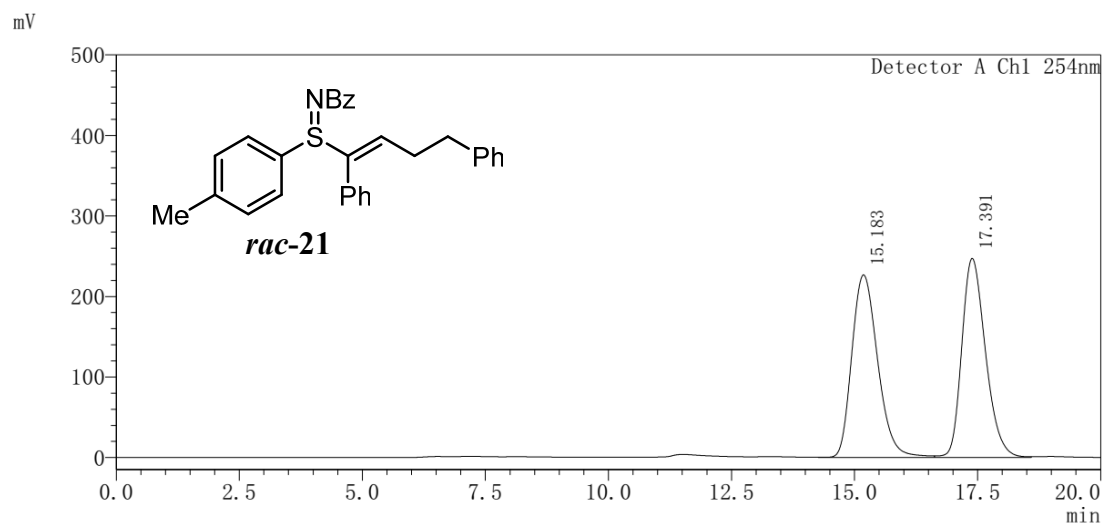
Peak#	Ret. Time	Area	Area%
1	26.710	7559671	95.544
2	30.025	352564	4.456



Peak Table

PDA Ch2 280nm

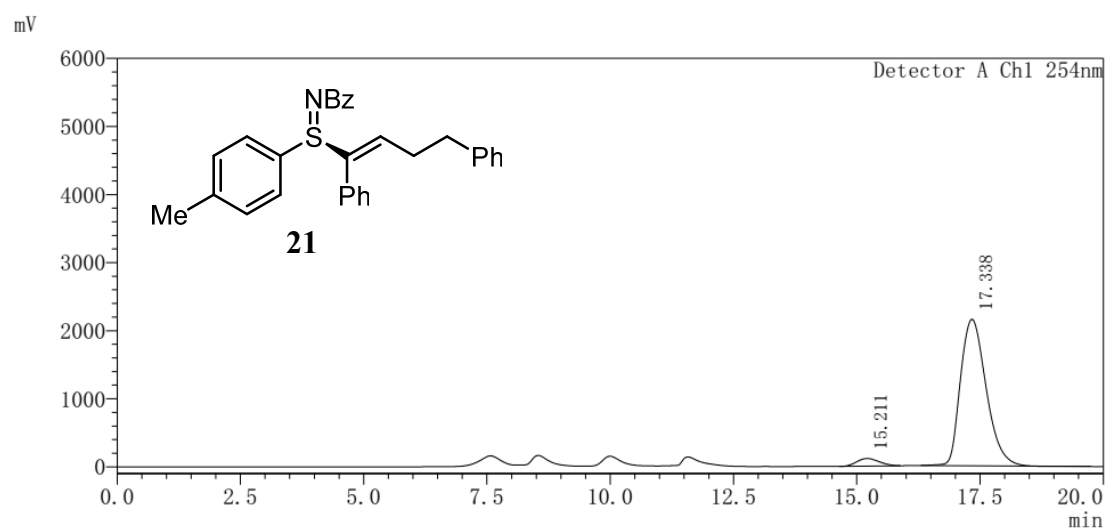
Peak#	Ret. Time	Area	Area%
1	26.404	5235256	94.805
2	29.252	286891	5.195



Peak Table

Detector A Ch1 254nm

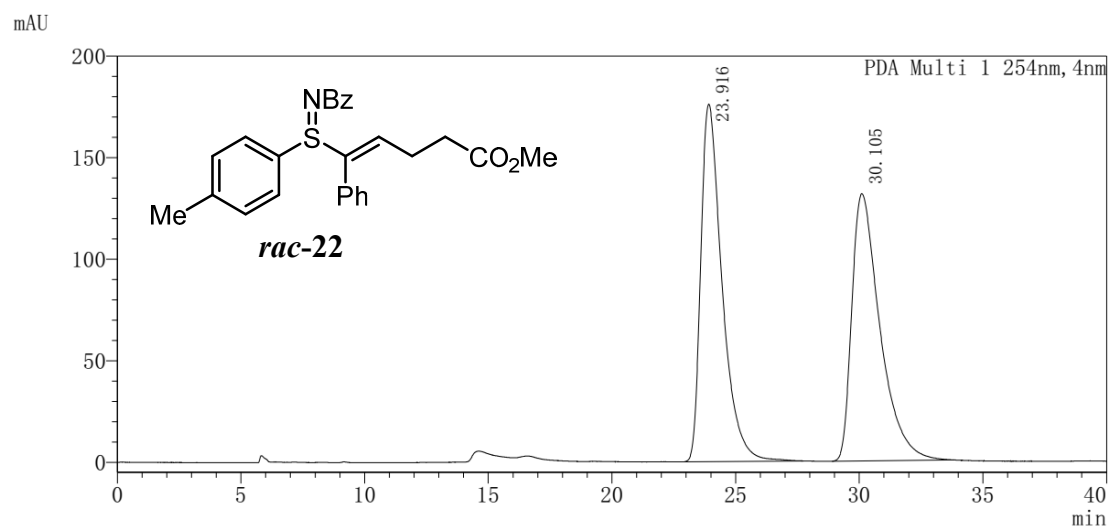
Peak#	Ret. Time	Area	Area%
1	15.183	8316591	50.044
2	17.391	8301902	49.956



Peak Table

Detector A Ch1 254nm

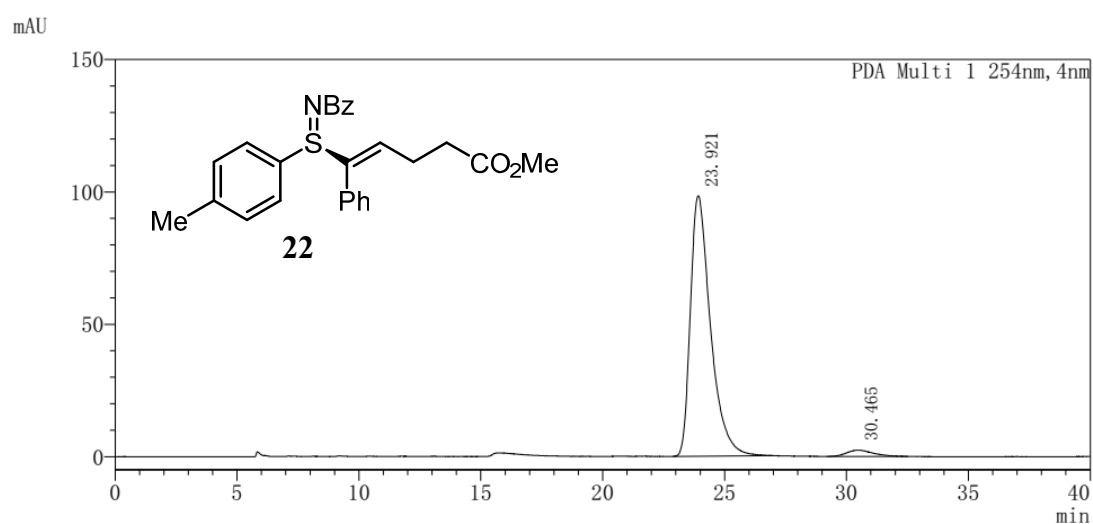
Peak#	Ret. Time	Area	Area%
1	15.211	3543799	4.342
2	17.338	78069772	95.658



Peak Table

PDA Ch1 254nm

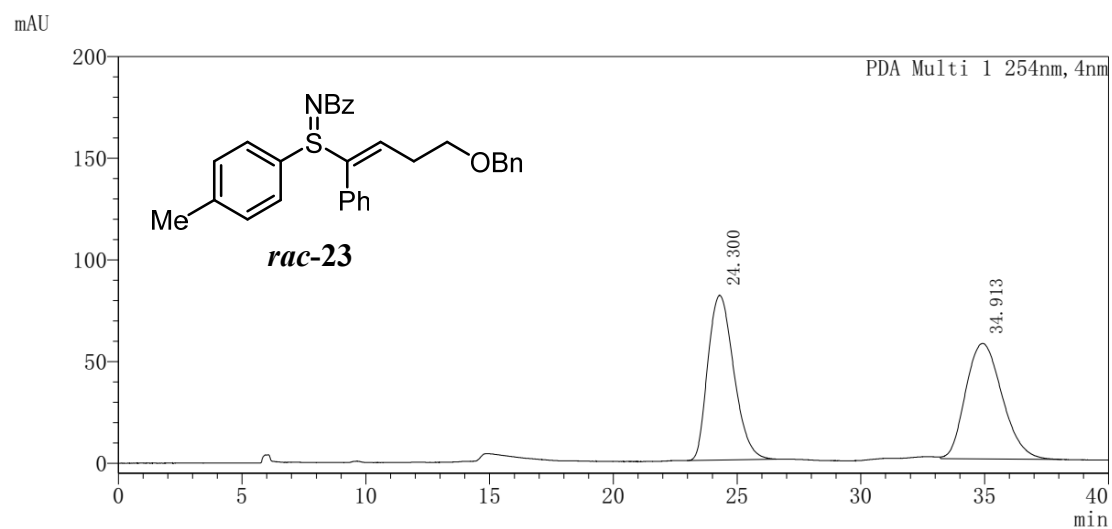
Peak#	Ret. Time	Area	Area%
1	23.916	10538963	50.246
2	30.105	10435693	49.754



Peak Table

PDA Ch1 254nm

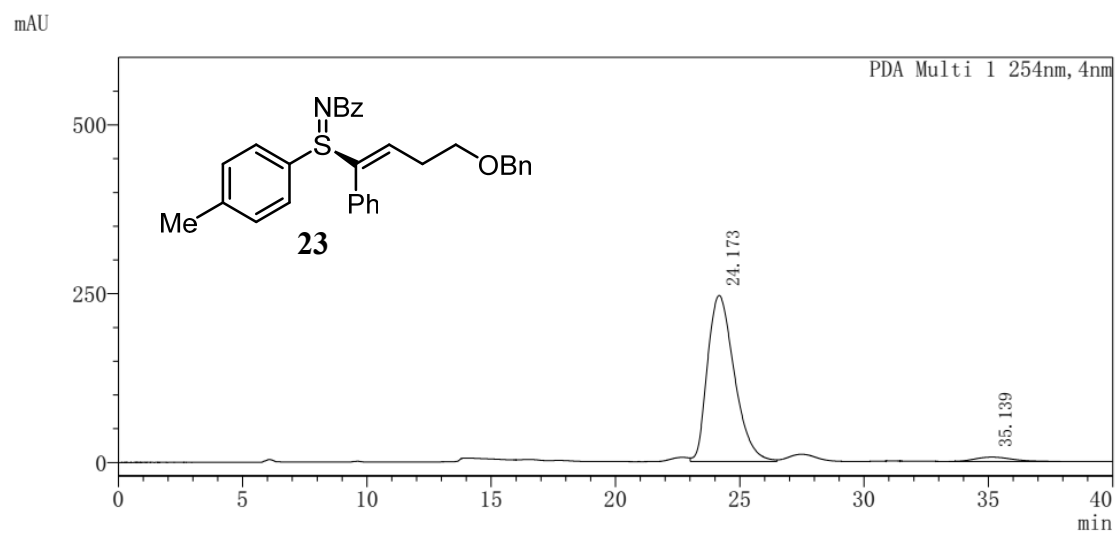
Peak#	Ret. Time	Area	Area%
1	23.921	5658254	96.922
2	30.465	179671	3.078



Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	24.300	5917872	50.345
2	34.913	5836755	49.655

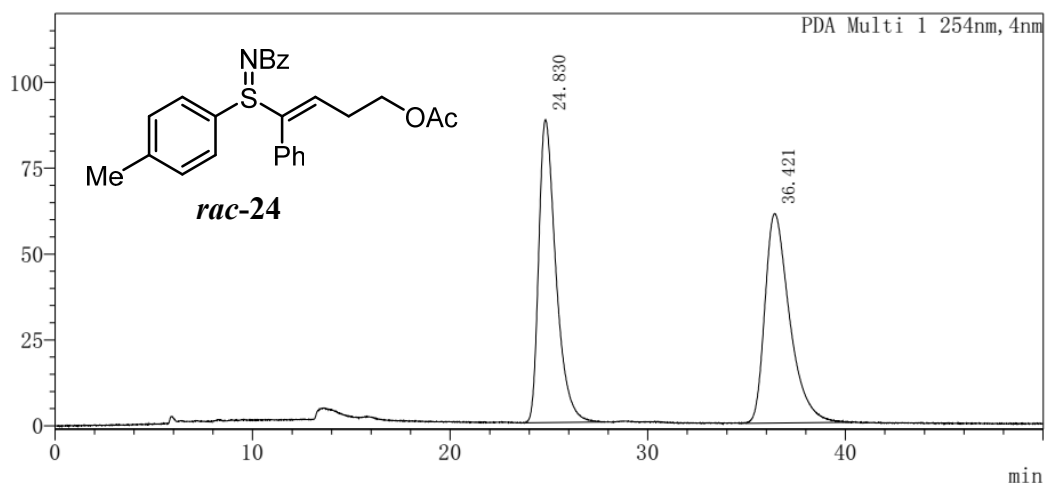


Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	24.173	18546182	96.567
2	35.139	659393	3.433

mAU

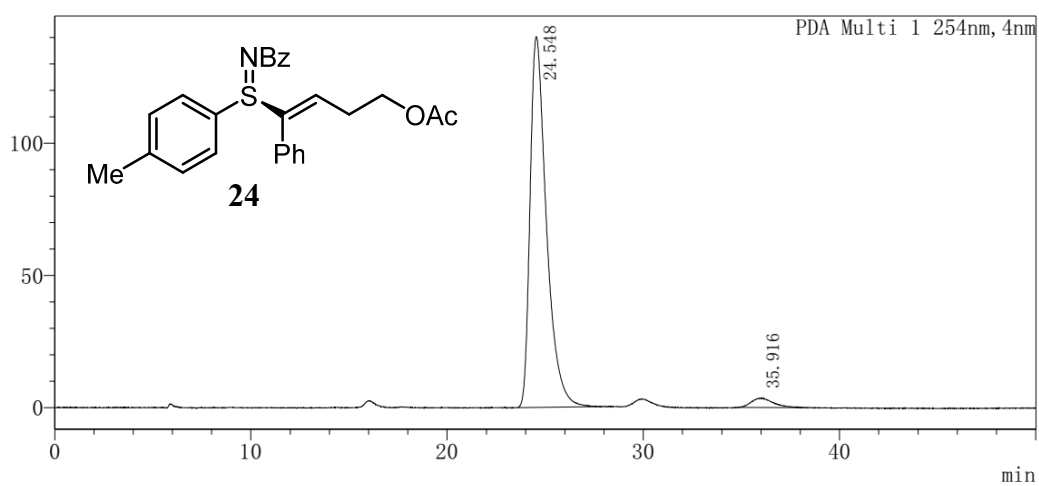


Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	24.830	5328407	50.069
2	36.421	5313787	49.931

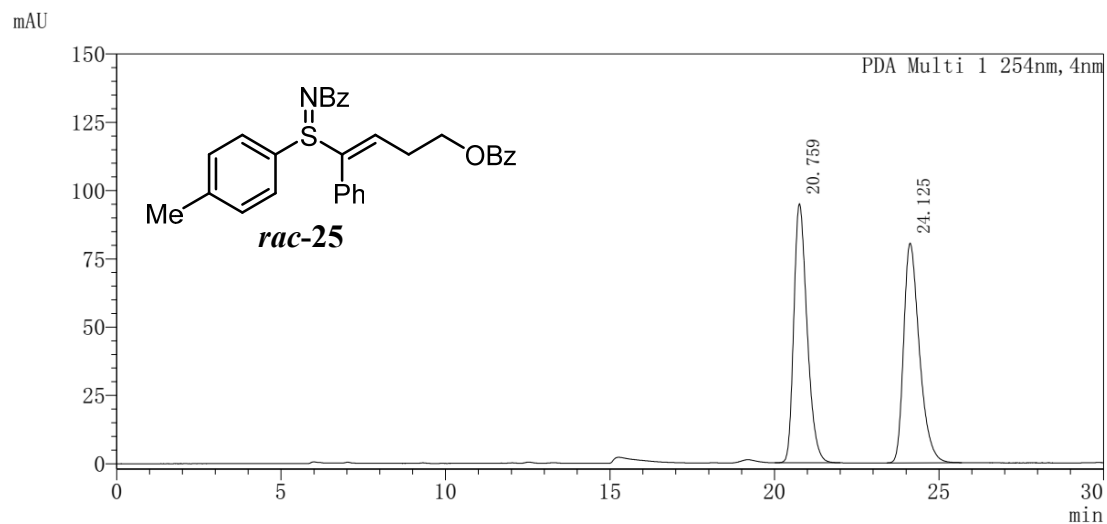
mAU



Peak Table

PDA Ch1 254nm

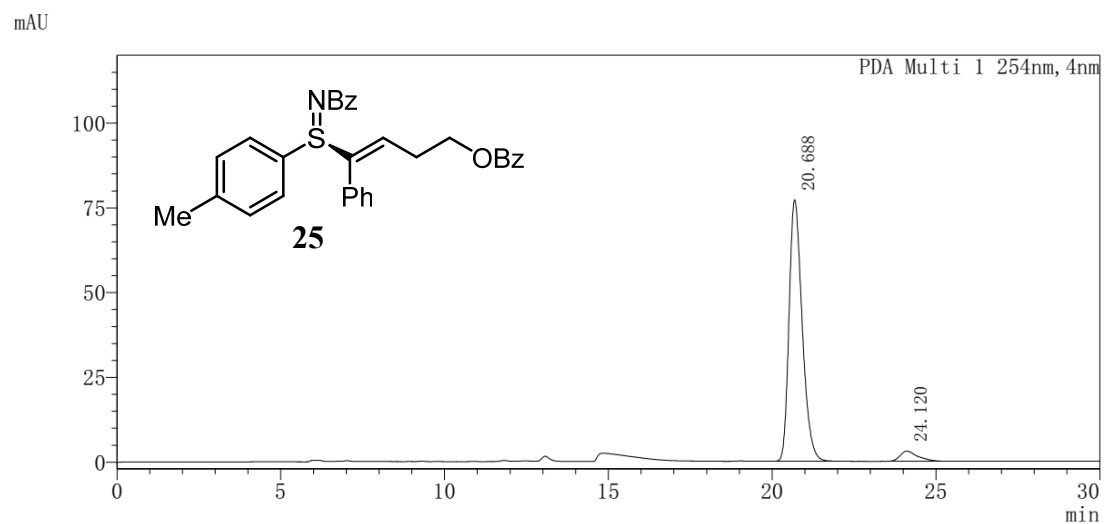
Peak#	Ret. Time	Area	Area%
1	24.548	8120586	96.677
2	35.916	279121	3.323



Peak Table

PDA Ch1 254nm

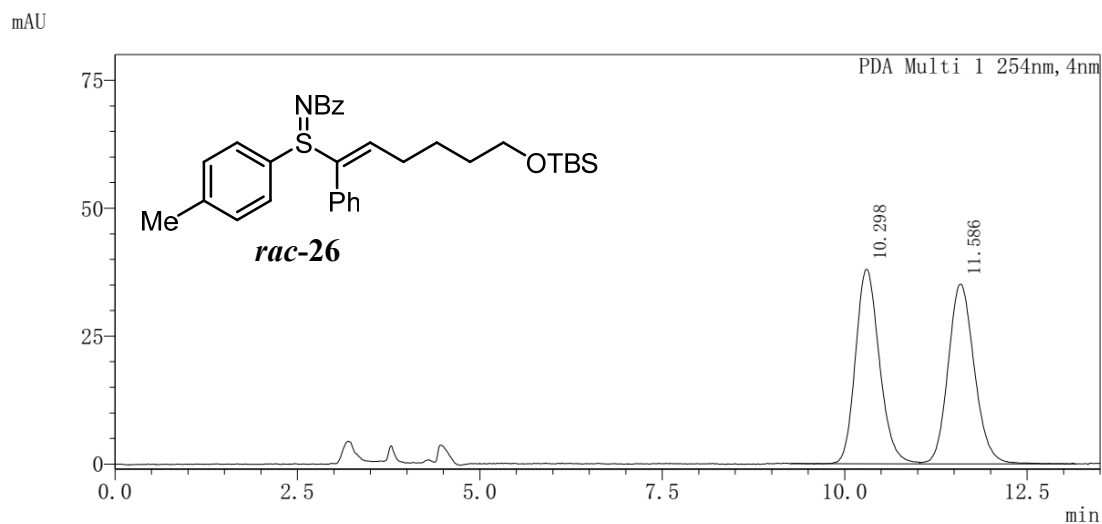
Peak#	Ret. Time	Area	Area%
1	20.759	2663076	49.857
2	24.125	2678371	50.143



Peak Table

PDA Ch1 254nm

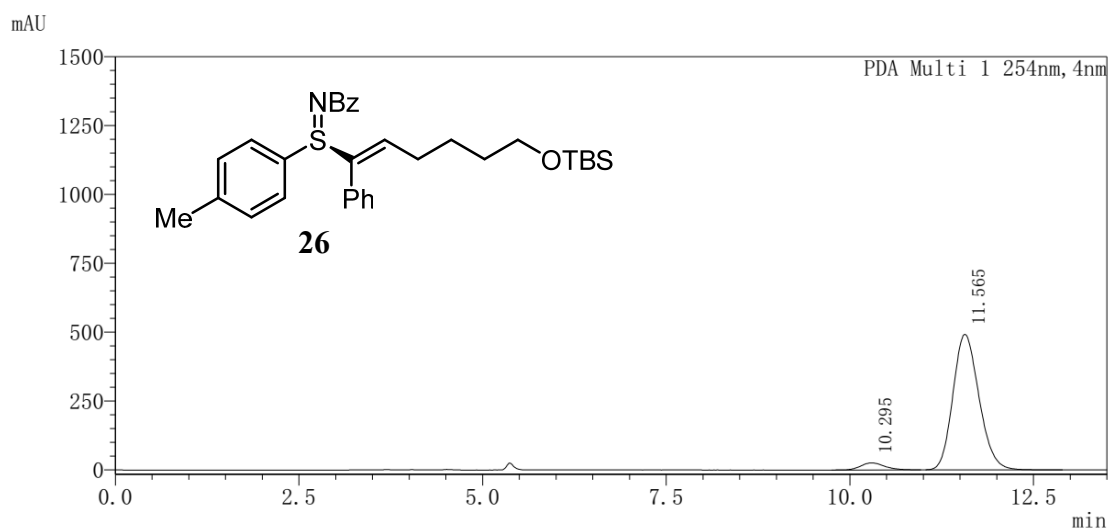
Peak#	Ret. Time	Area	Area%
1	20.688	2148200	95.120
2	24.120	110213	4.880



Peak Table

PDA Ch1 254nm

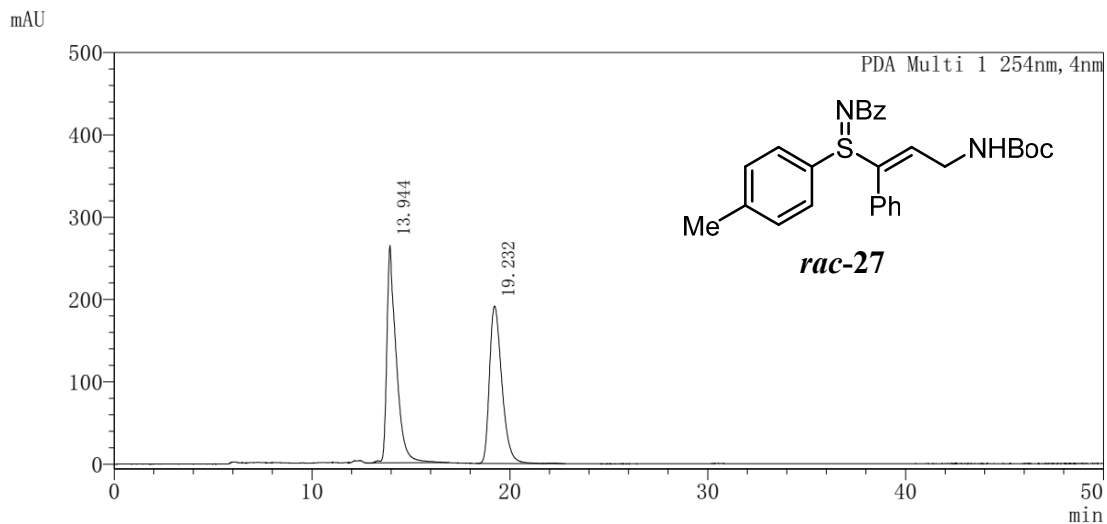
Peak#	Ret. Time	Area	Area%
1	10.298	867421	49.692
2	11.586	878171	50.308



Peak Table

PDA Ch1 254nm

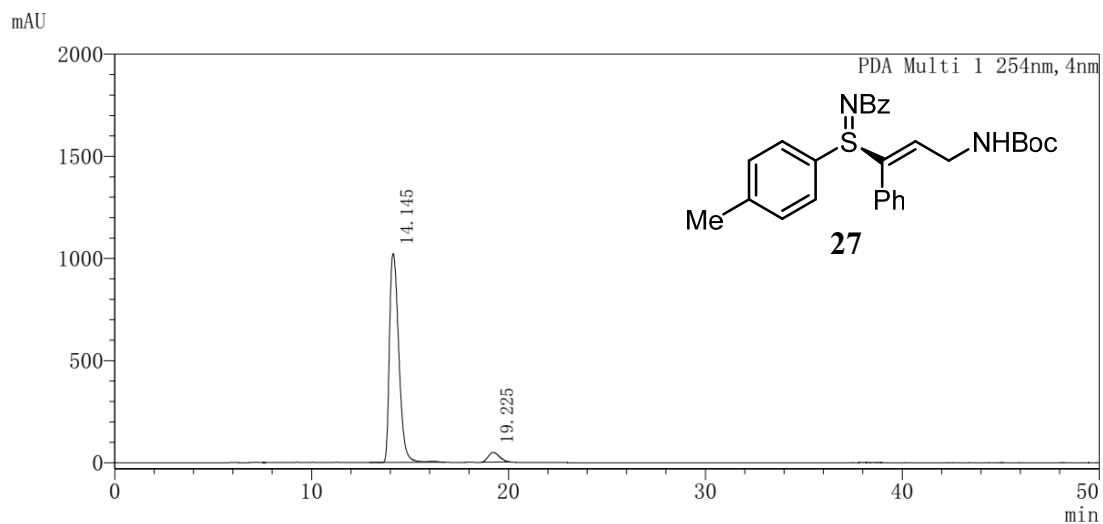
Peak#	Ret. Time	Area	Area%
1	10.295	581588	4.565
2	11.565	12158575	95.435



Peak Table

PDA Ch1 254nm

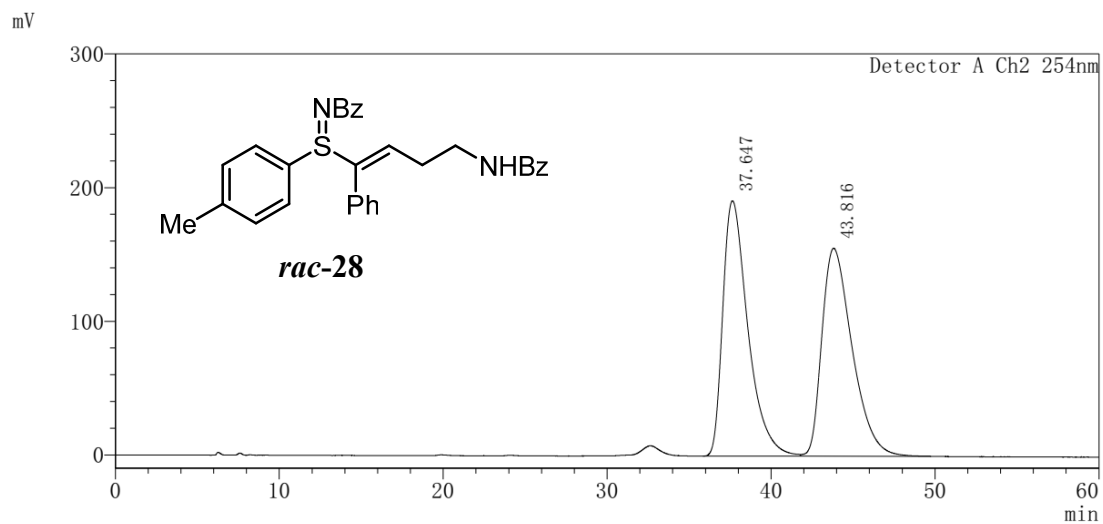
Peak#	Ret. Time	Area	Area%
1	13.944	8646868	50.939
2	19.232	8327931	49.061



Peak Table

PDA Ch1 254nm

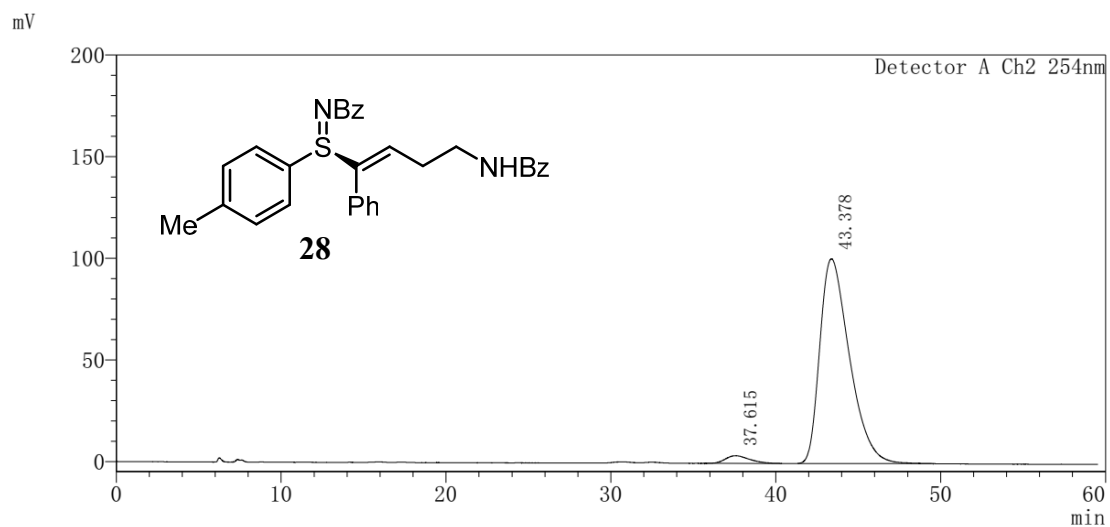
Peak#	Ret. Time	Area	Area%
1	14.145	33753695	94.745
2	19.225	1872019	5.255



Peak Table

Detector A Ch2 254nm

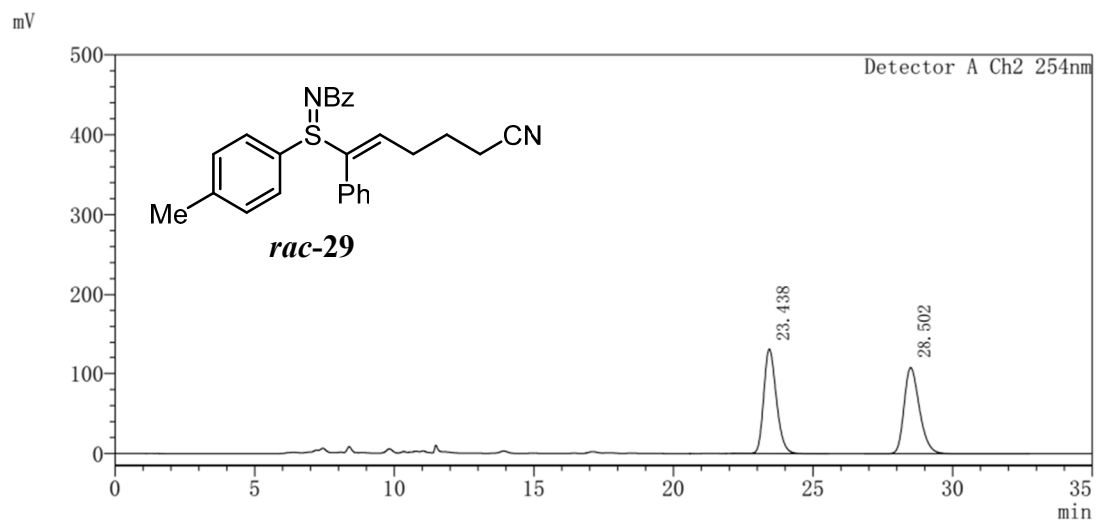
Peak#	Ret. Time	Area	Area%
1	37.647	20427788	50.539
2	43.816	19992210	49.461



Peak Table

Detector A Ch2 254nm

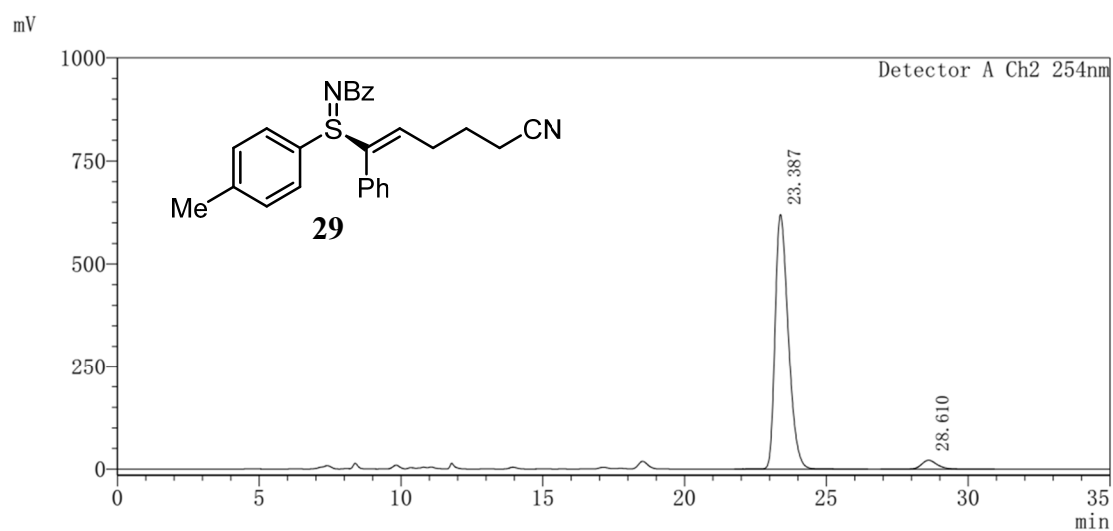
Peak#	Ret. Time	Area	Area%
1	37.615	385745	2.964
2	43.378	12626997	97.036



Peak Table

Detector A Ch2 254nm

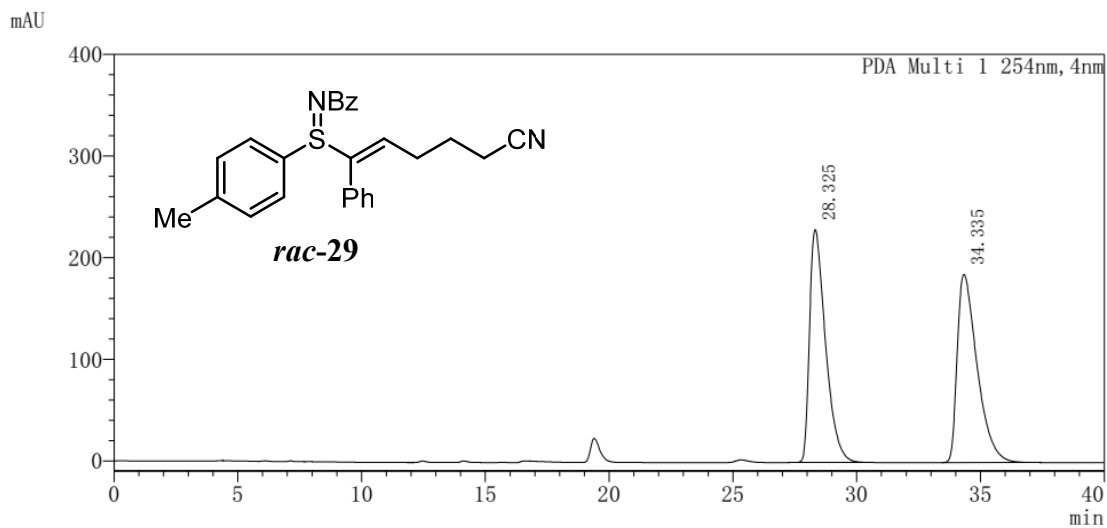
Peak#	Ret. Time	Area	Area%
1	23.438	4109484	49.949
2	28.502	4117812	50.051



Peak Table

Detector A Ch2 254nm

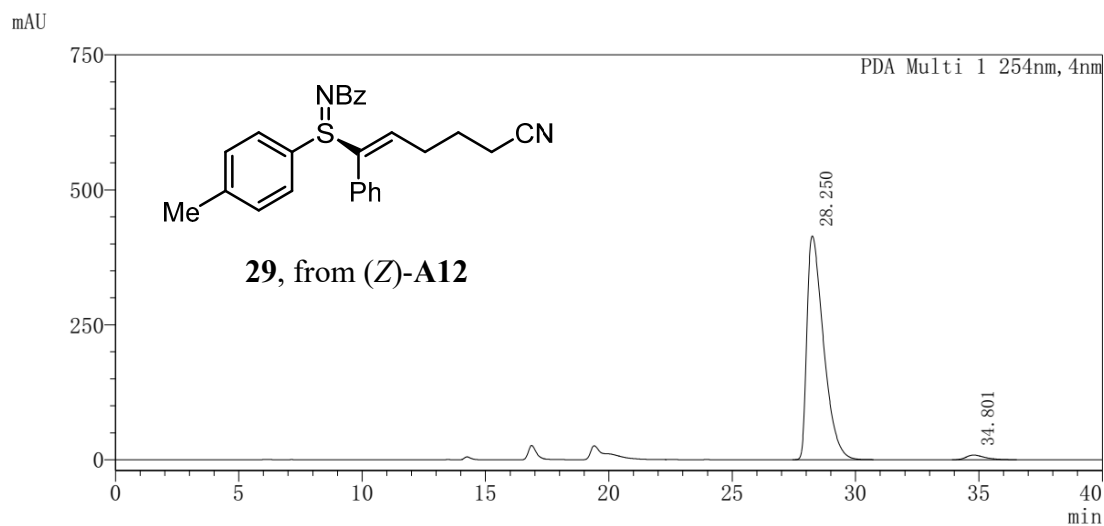
Peak#	Ret. Time	Area	Area%
1	23.387	20031954	96.129
2	28.610	806590	3.871



Peak Table

PDA Ch1 254nm

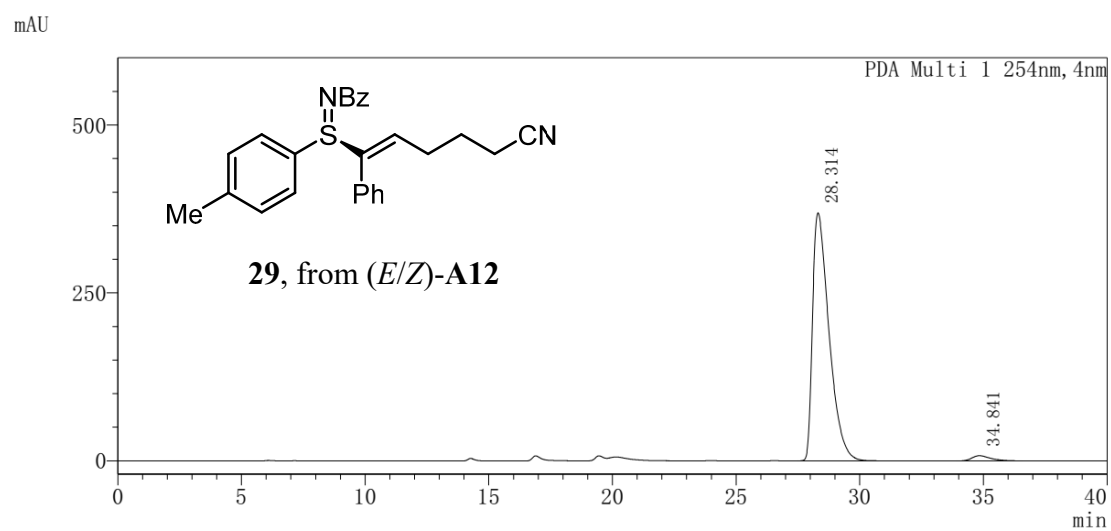
Peak#	Ret. Time	Area	Area%
1	28.325	10283617	49.872
2	34.335	10336371	50.128



Peak Table

PDA Ch1 254nm

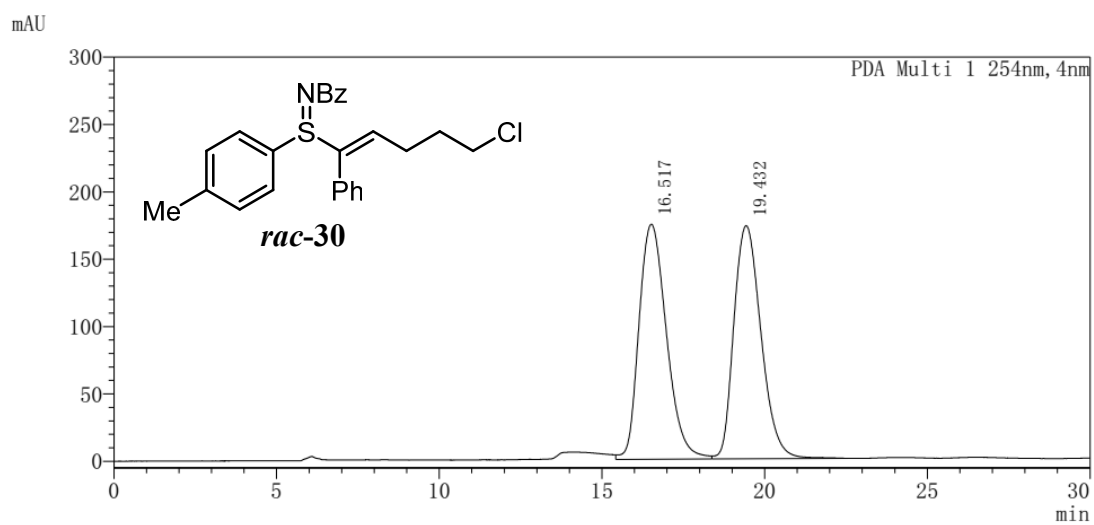
Peak#	Ret. Time	Area	Area%
1	28.250	19439443	97.711
2	34.801	455345	2.289



Peak Table

PDA Ch1 254nm

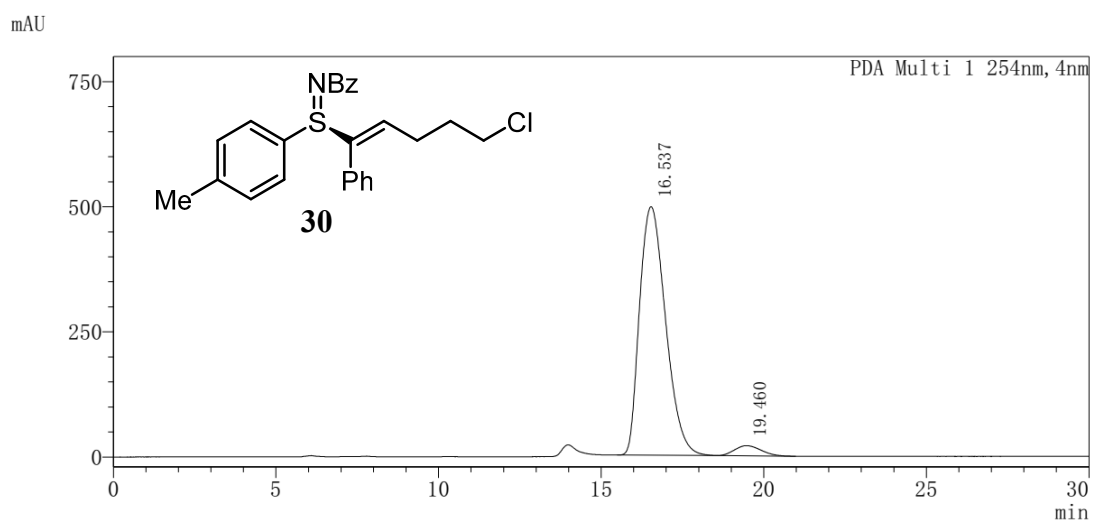
Peak#	Ret. Time	Area	Area%
1	28.314	17124821	97.682
2	34.841	406346	2.318



Peak Table

PDA Ch1 254nm

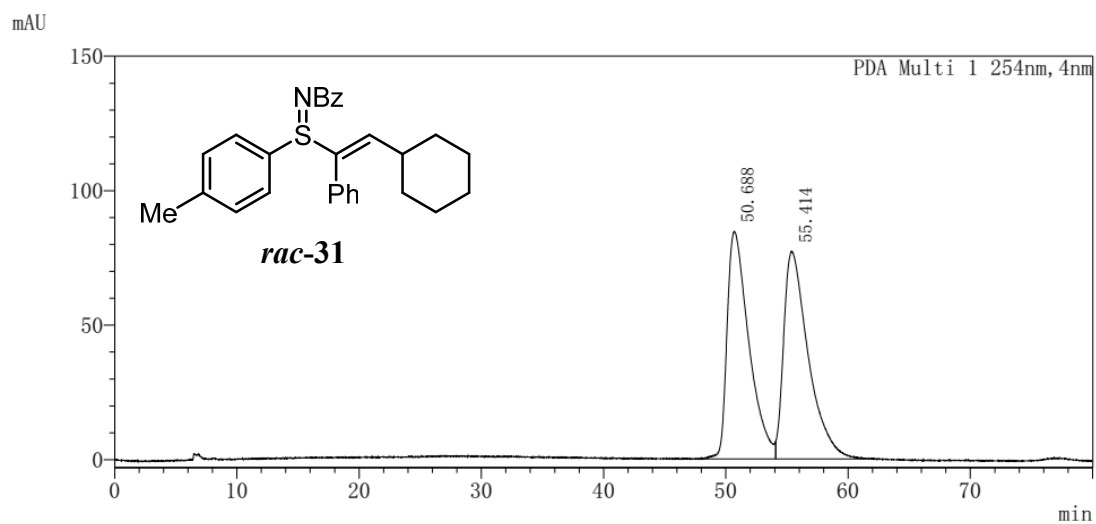
Peak#	Ret. Time	Area	Area%
1	16.517	10268236	50.499
2	19.432	10065266	49.501



Peak Table

PDA Ch1 254nm

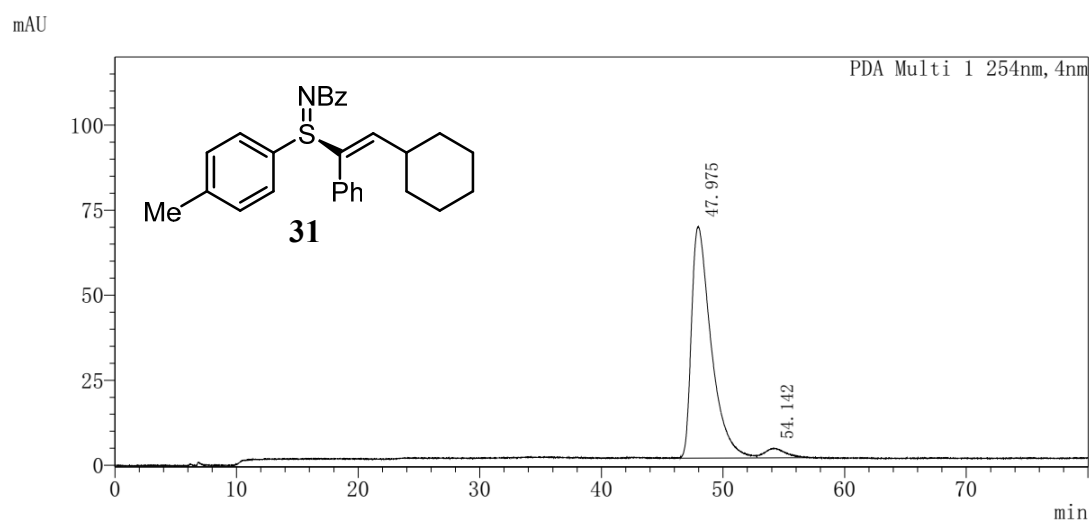
Peak#	Ret. Time	Area	Area%
1	16.537	27691213	96.013
2	19.460	1149969	3.987



Peak Table

PDA Ch1 254nm

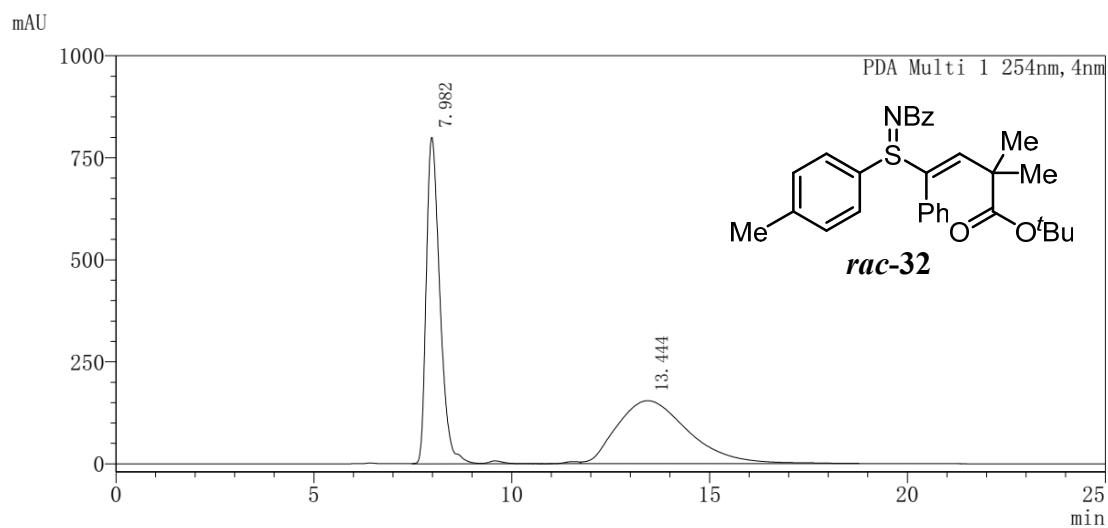
Peak#	Ret. Time	Area	Area%
1	50.688	10495093	49.269
2	55.414	10806675	50.731



Peak Table

PDA Ch1 254nm

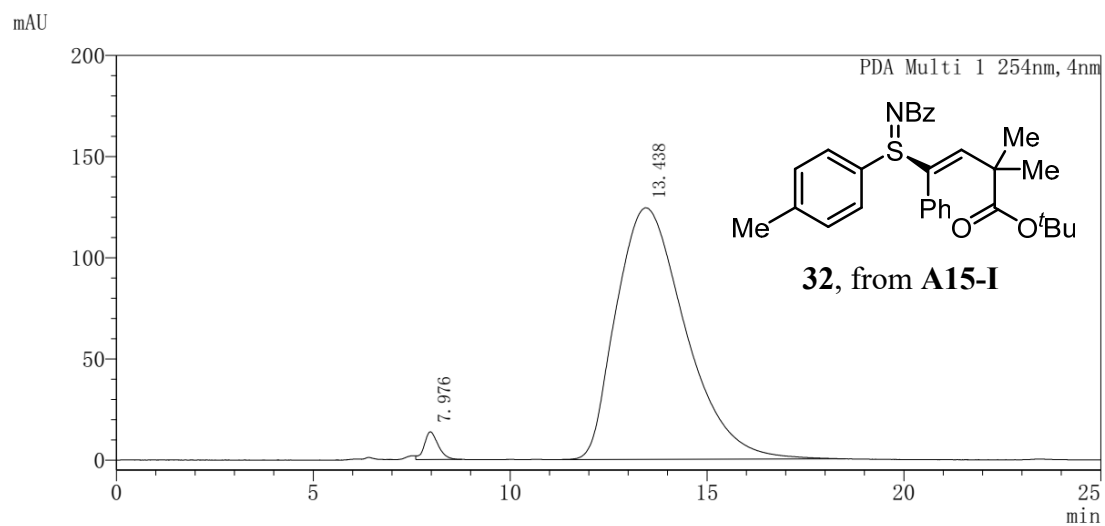
Peak#	Ret. Time	Area	Area%
1	47.975	7653155	95.733
2	54.142	341126	4.267



Peak Table

PDA Ch1 254nm

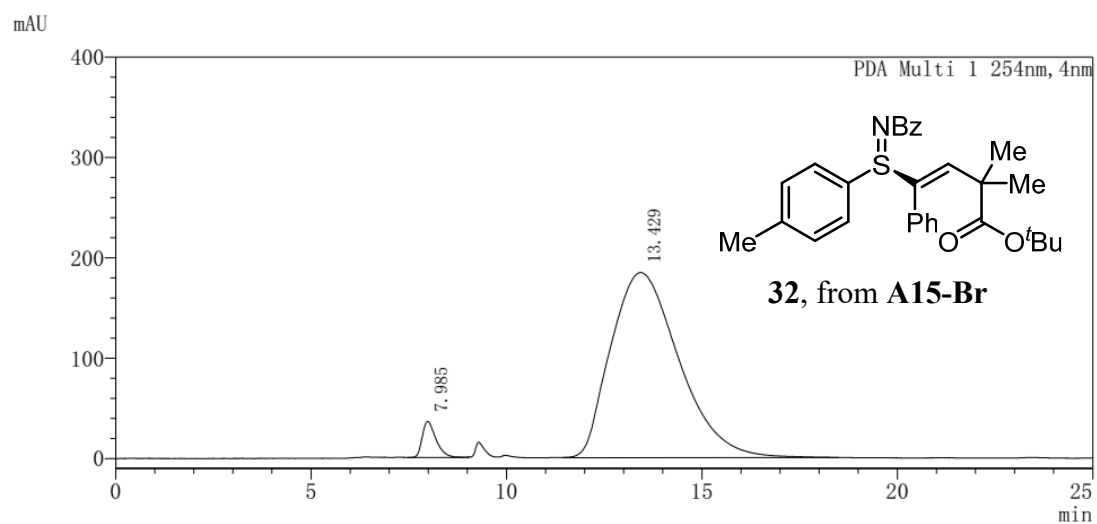
Peak#	Ret. Time	Area	Area%
1	7.982	19448916	50.178
2	13.444	19311271	49.822



Peak Table

PDA Ch1 254nm

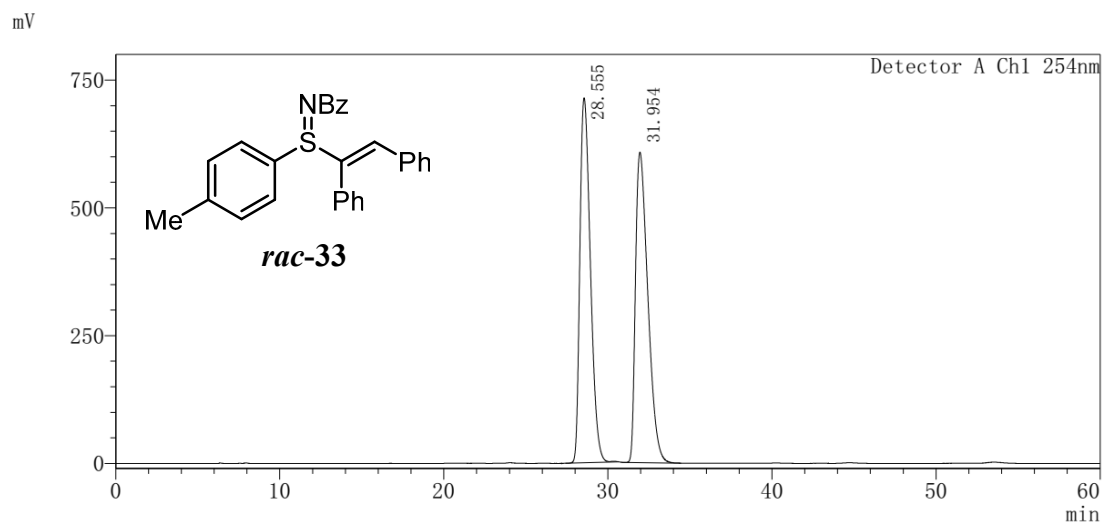
Peak#	Ret. Time	Area	Area%
1	7.976	339398	2.185
2	13.438	15194919	97.815



Peak Table

PDA Ch1 254nm

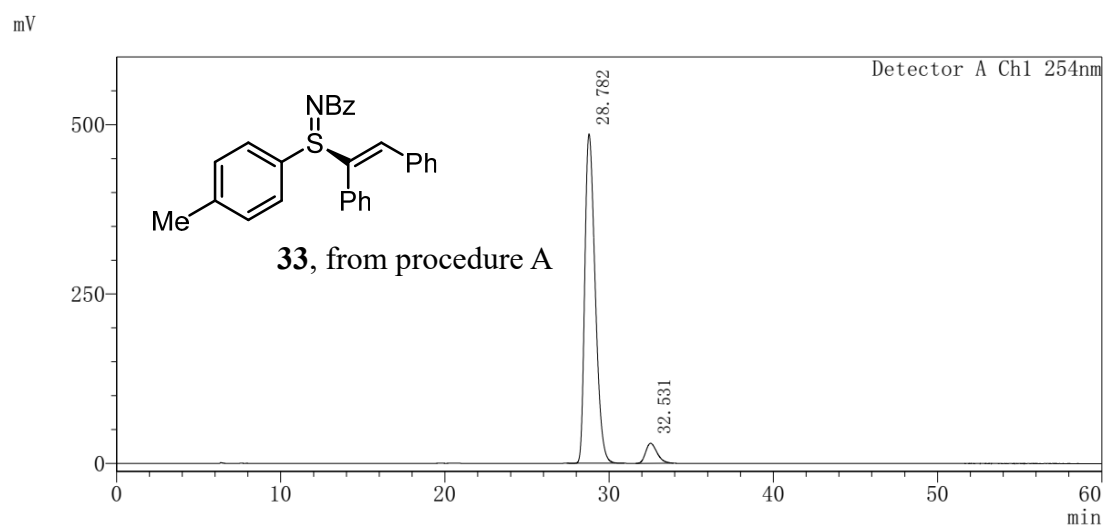
Peak#	Ret. Time	Area	Area%
1	7.985	888545	3.809
2	13.429	22437922	96.191



Peak Table

Detector A Ch1 254nm

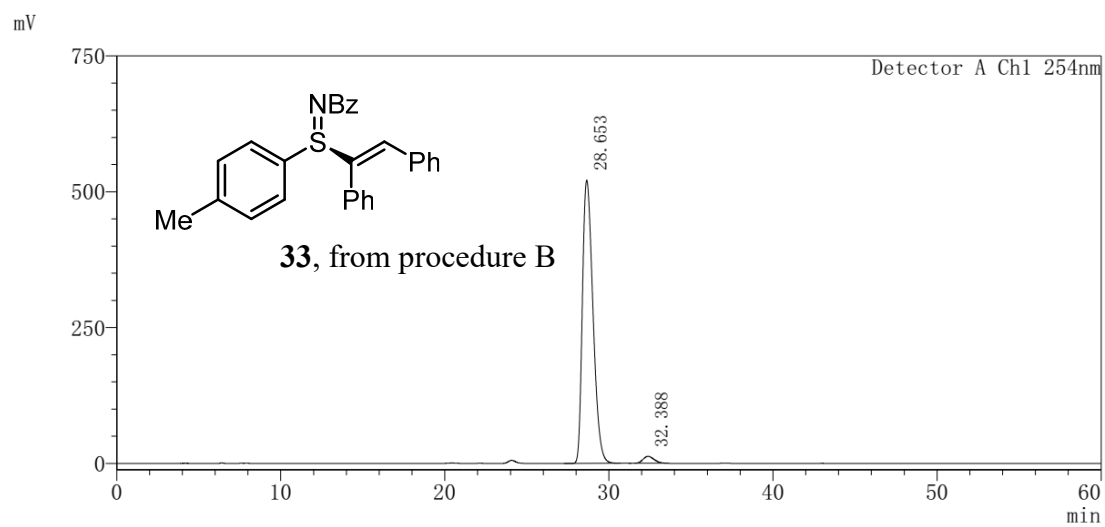
Peak#	Ret. Time	Area	Area%
1	28.555	31825249	50.027
2	31.954	31790571	49.973



Peak Table

Detector A Ch1 254nm

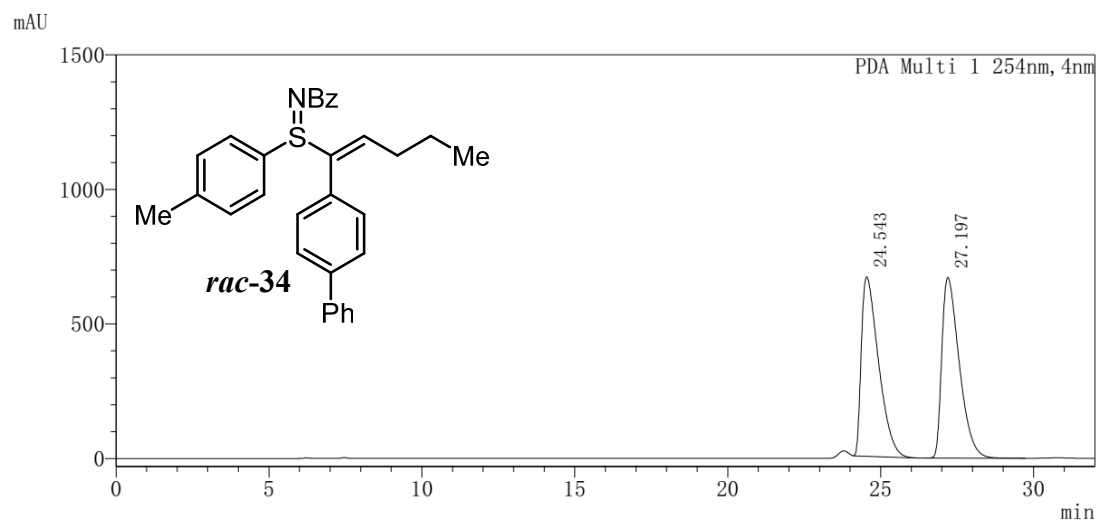
Peak#	Ret. Time	Area	Area%
1	28.782	21377155	93.774
2	32.531	1419291	6.226



Peak Table

Detector A Ch1 254nm

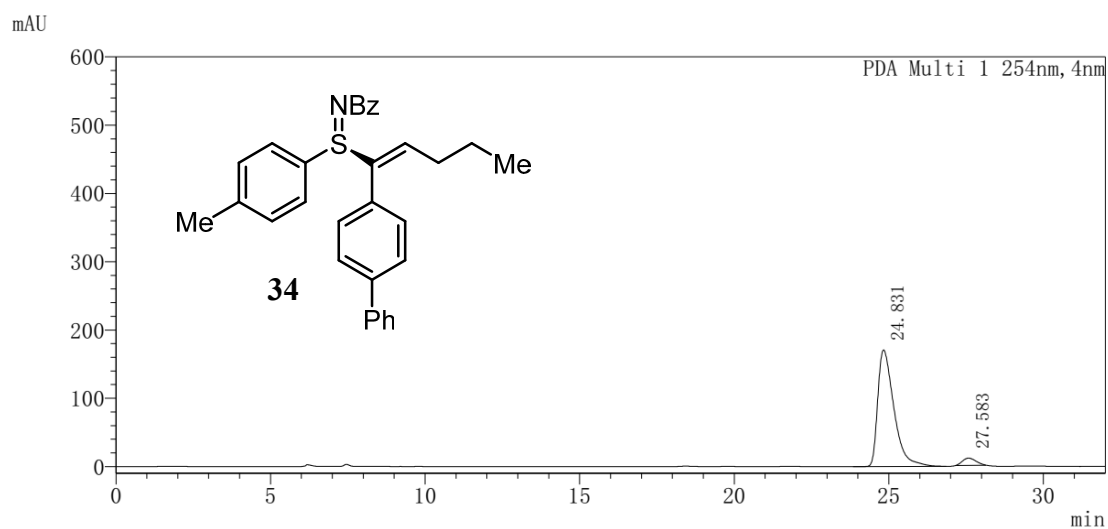
Peak#	Ret. Time	Area	Area%
1	28.653	23357441	97.452
2	32.388	610722	2.548



Peak Table

PDA Ch1 254nm

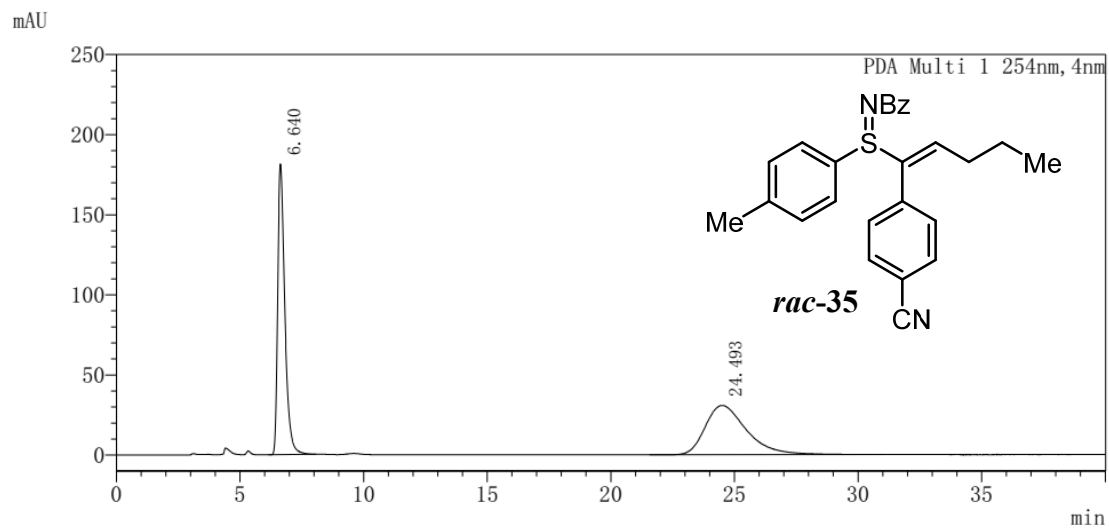
Peak#	Ret. Time	Area	Area%
1	24.543	26077804	49.462
2	27.197	26645072	50.538



Peak Table

PDA Ch1 254nm

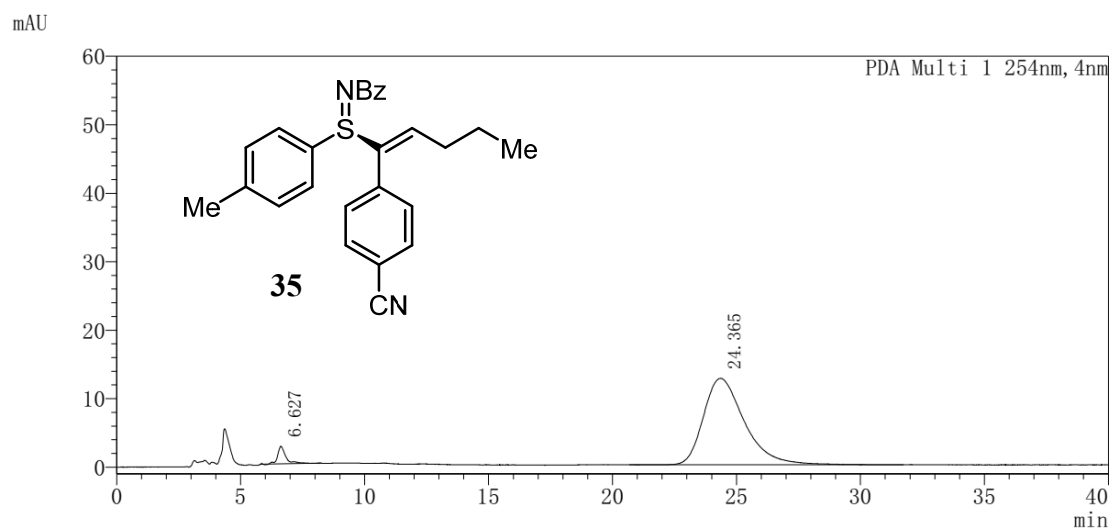
Peak#	Ret. Time	Area	Area%
1	24.831	6198405	95.149
2	27.583	316025	4.851



Peak Table

PDA Ch1 254nm

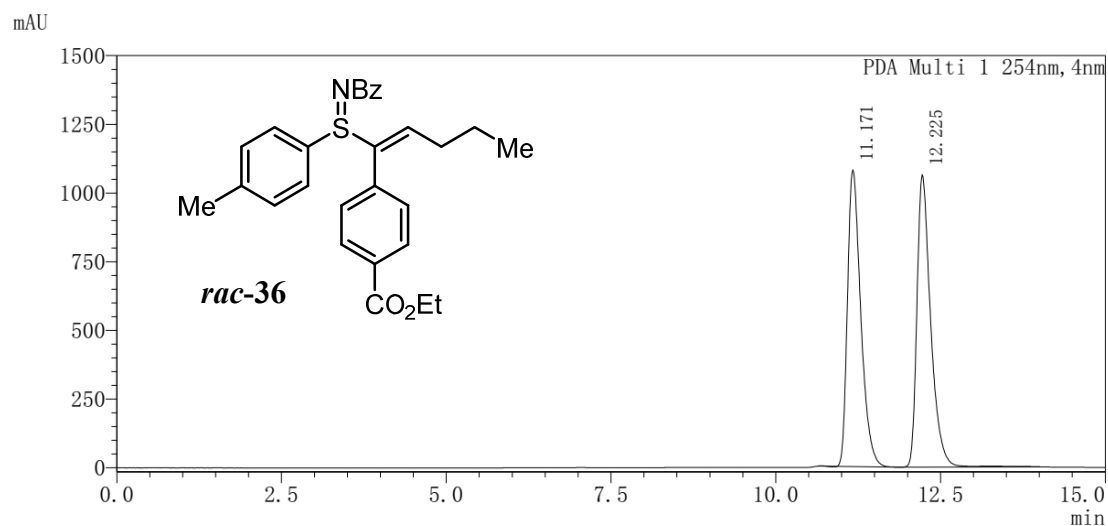
Peak#	Ret. Time	Area	Area%
1	6.640	3635530	50.395
2	24.493	3578608	49.605



Peak Table

PDA Ch1 254nm

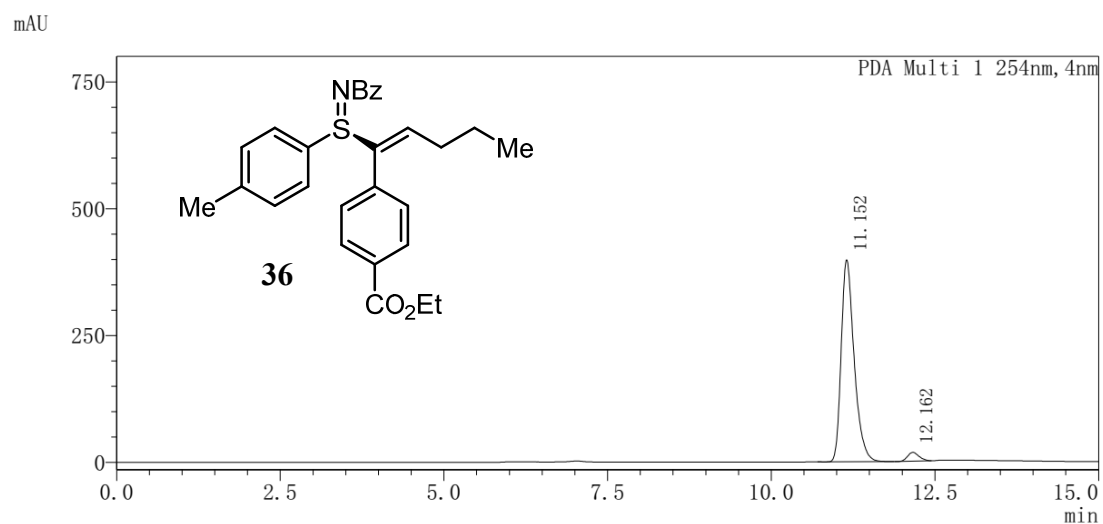
Peak#	Ret. Time	Area	Area%
1	6.627	58691	3.888
2	24.365	1450912	96.112



Peak Table

PDA Ch1 254nm

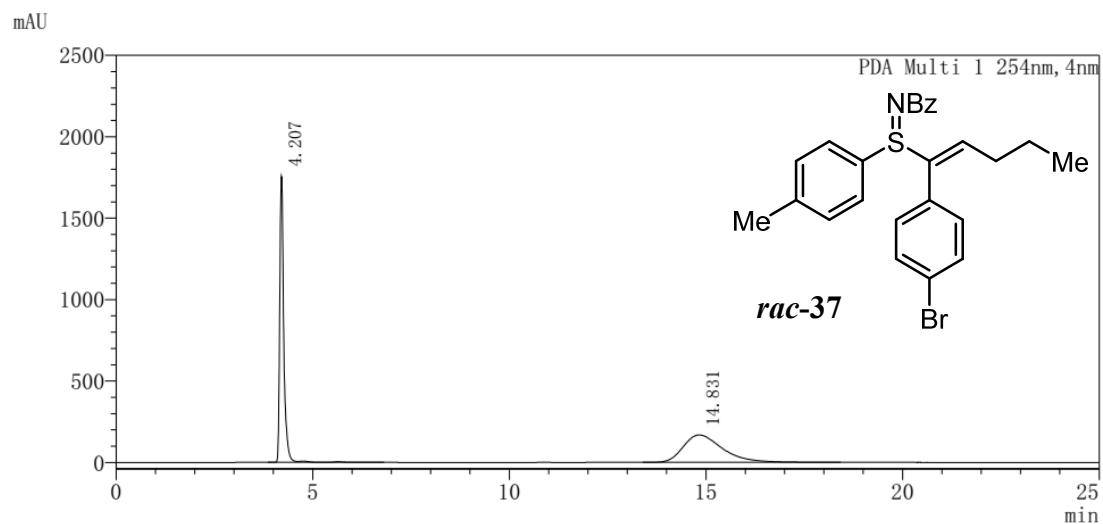
Peak#	Ret. Time	Area	Area%
1	11.171	15150033	49.422
2	12.225	15504505	50.578



Peak Table

PDA Ch1 254nm

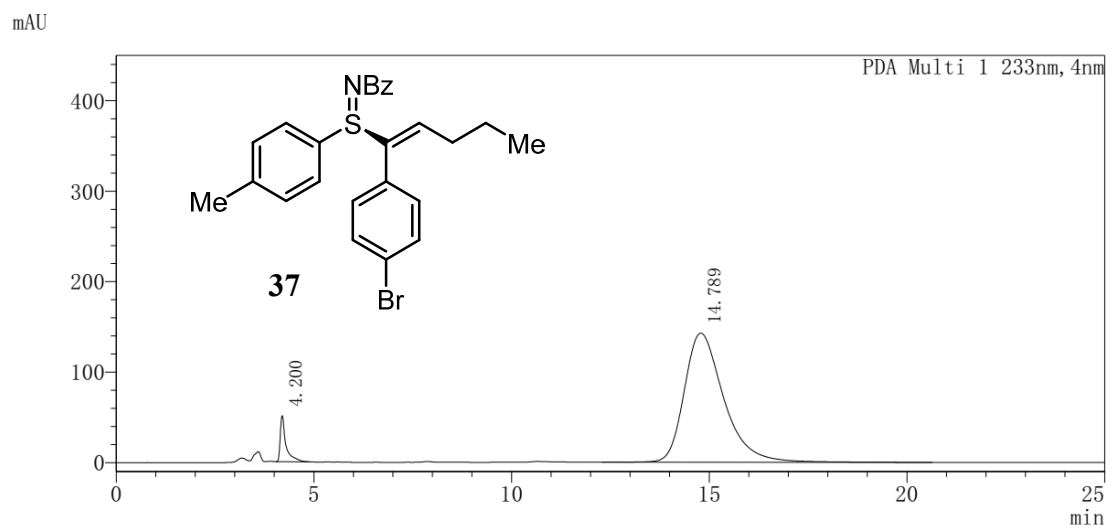
Peak#	Ret. Time	Area	Area%
1	11.152	5478007	96.213
2	12.162	215598	3.787



Peak Table

PDA Ch1 254nm

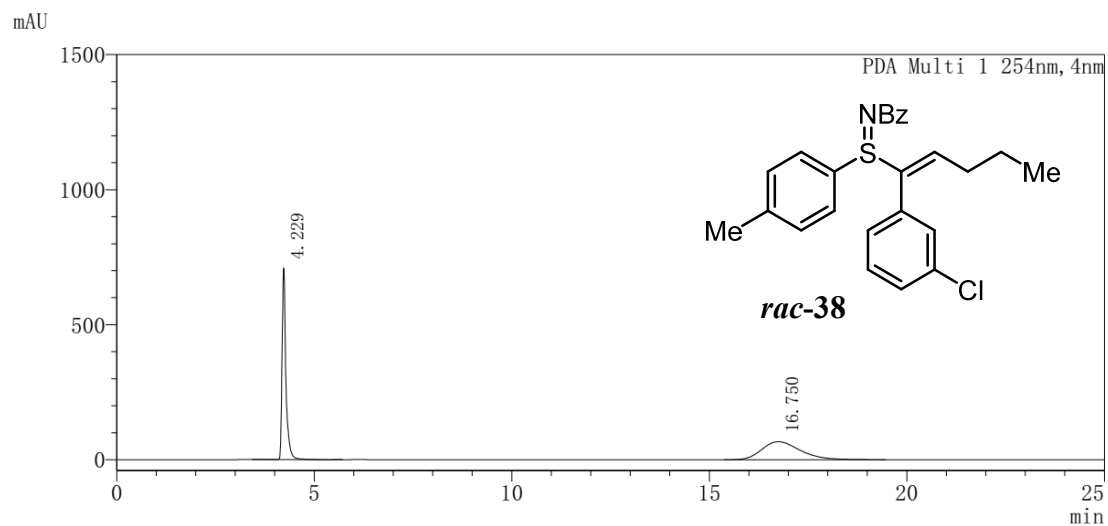
Peak#	Ret. Time	Area	Area%
1	4.207	12408549	51.274
2	14.831	11792155	48.726



Peak Table

PDA Ch1 233nm

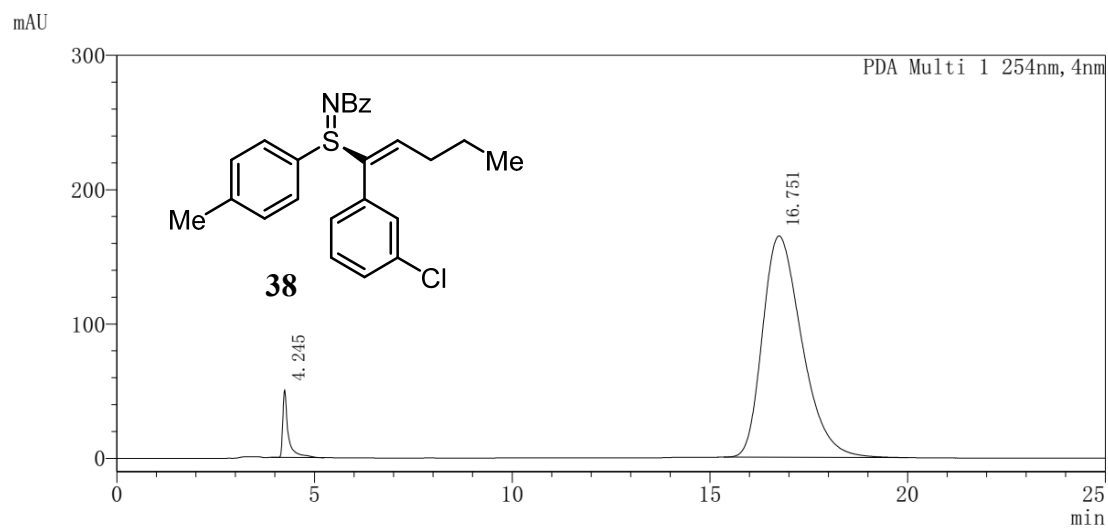
Peak#	Ret. Time	Area	Area%
1	4.200	510434	4.887
2	14.789	9934608	95.113



Peak Table

PDA Ch1 254nm

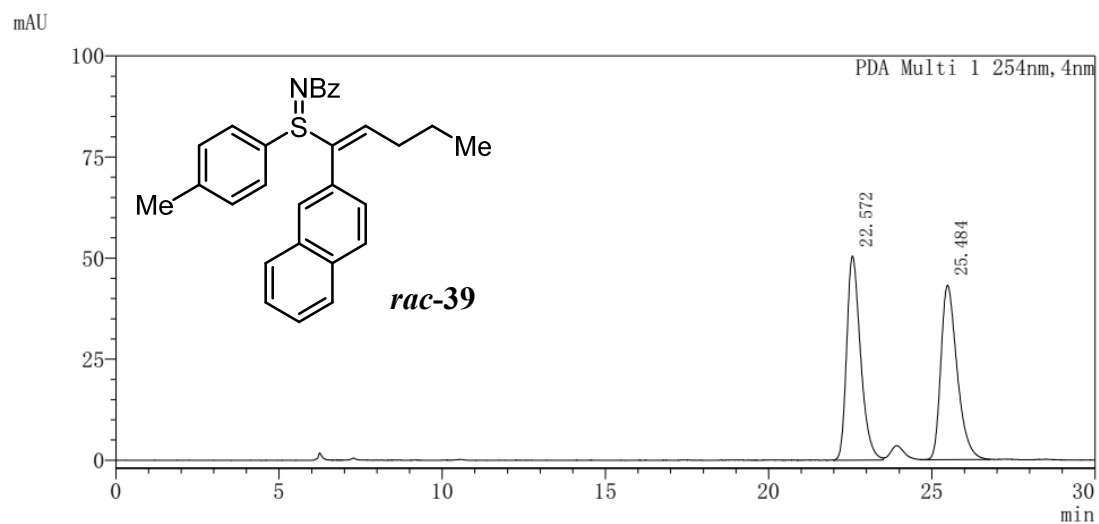
Peak#	Ret. Time	Area	Area%
1	4.229	4749557	51.253
2	16.750	4517393	48.747



Peak Table

PDA Ch1 254nm

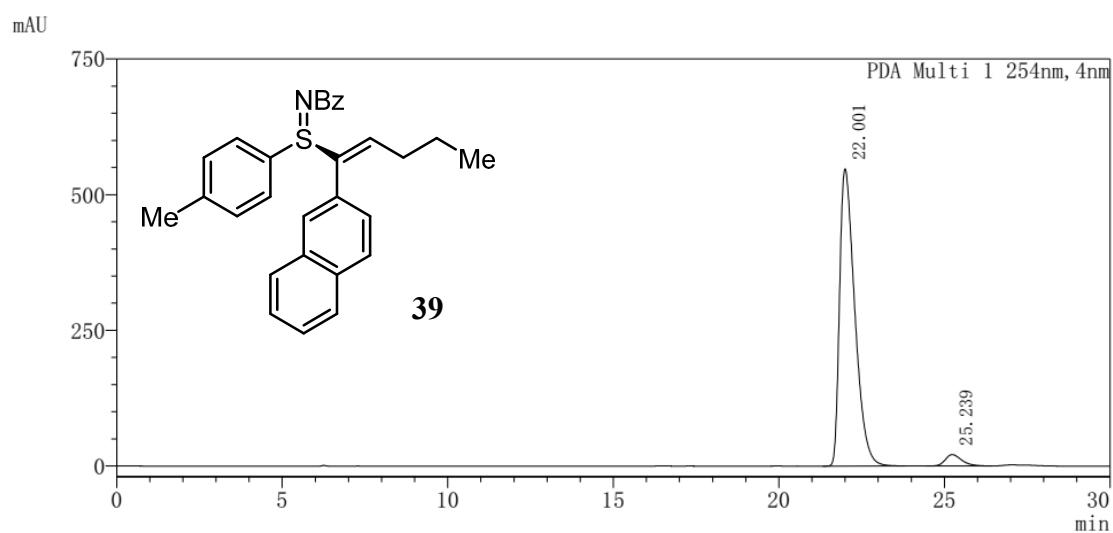
Peak#	Ret. Time	Area	Area%
1	4.245	481160	4.044
2	16.751	11417575	95.956



Peak Table

PDA Ch1 254nm

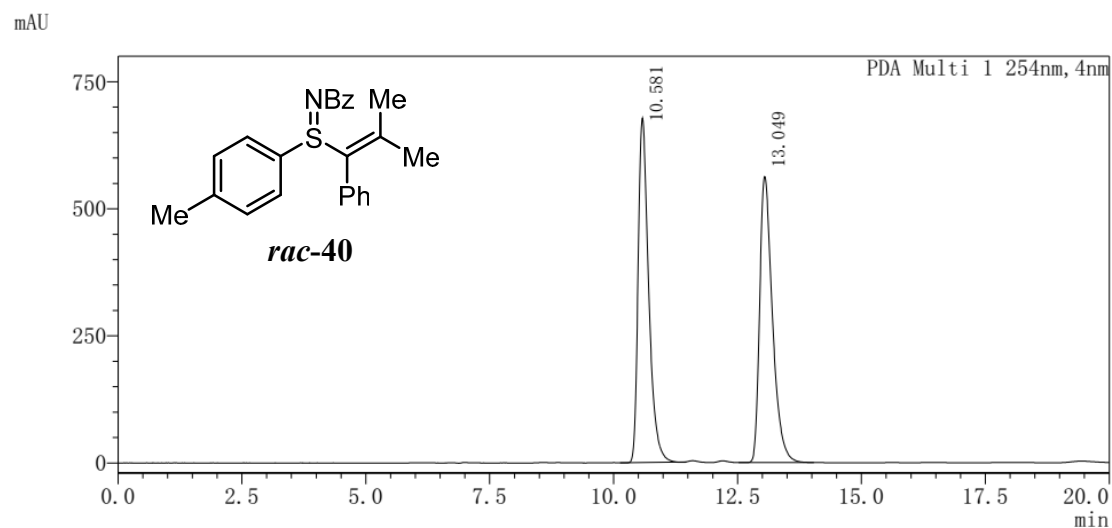
Peak#	Ret. Time	Area	Area%
1	22.572	1493786	50.012
2	25.484	1493067	49.988



Peak Table

PDA Ch1 254nm

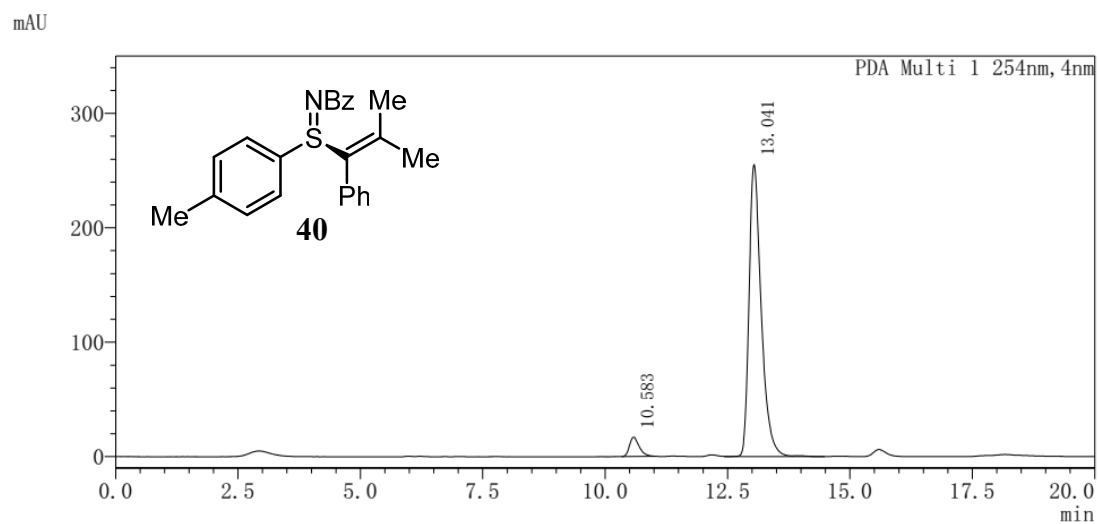
Peak#	Ret. Time	Area	Area%
1	22.001	17494208	95.905
2	25.239	746890	4.095



Peak Table

PDA Ch1 254nm

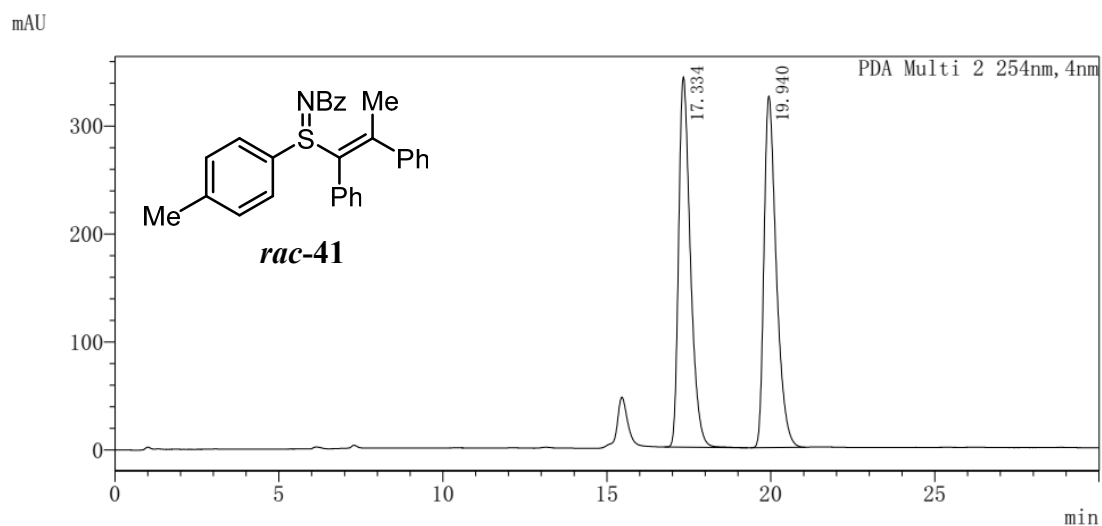
Peak#	Ret. Time	Area	Area%
1	10.581	10012844	49.823
2	13.049	10083969	50.177



Peak Table

PDA Ch1 254nm

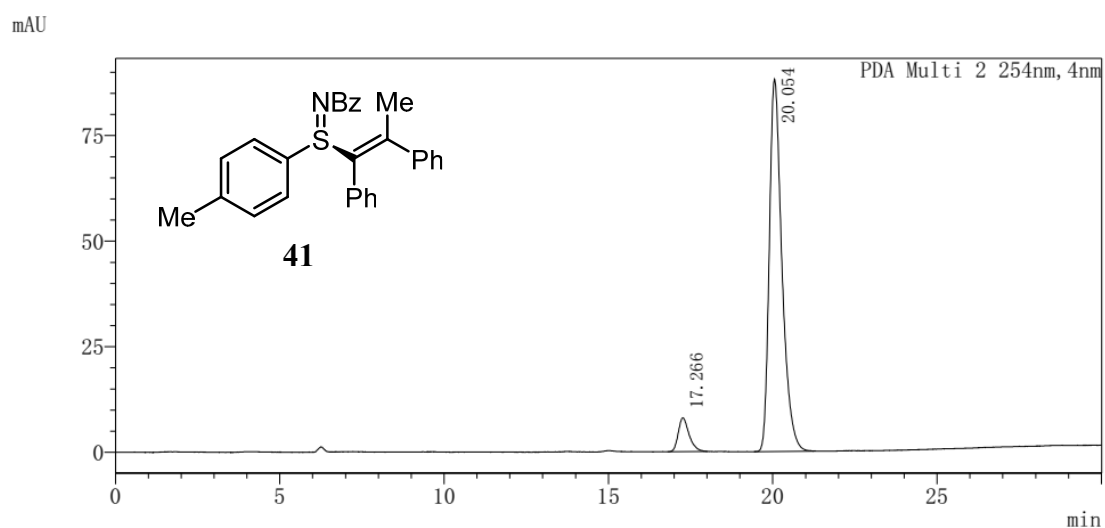
Peak#	Ret. Time	Area	Area%
1	10.583	231023	4.967
2	13.041	4420385	95.033



Peak Table

PDA Ch2 254nm

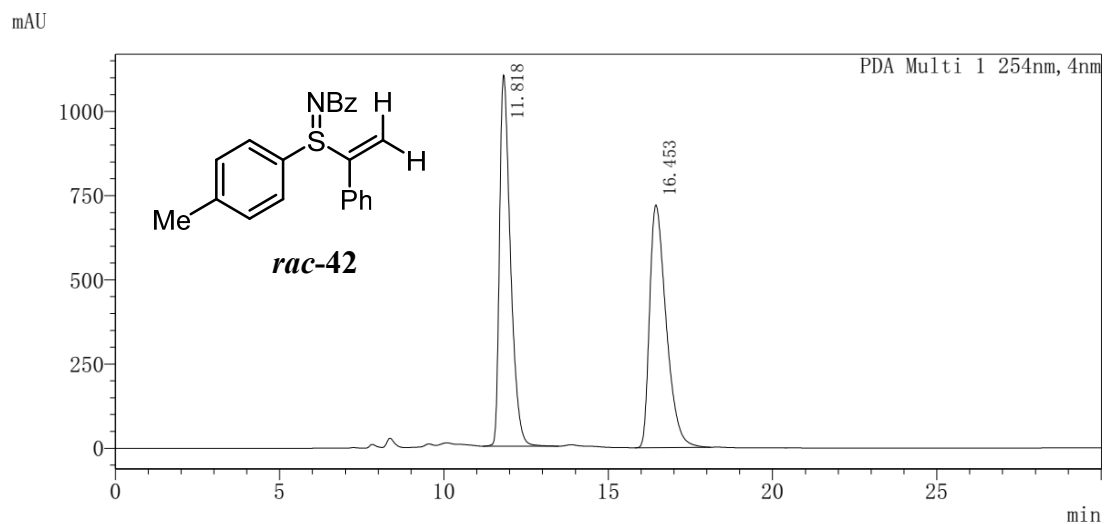
Peak#	Ret. Time	Area	Area%
1	17.334	8532775	50.066
2	19.940	8510274	49.934



Peak Table

PDA Ch2 254nm

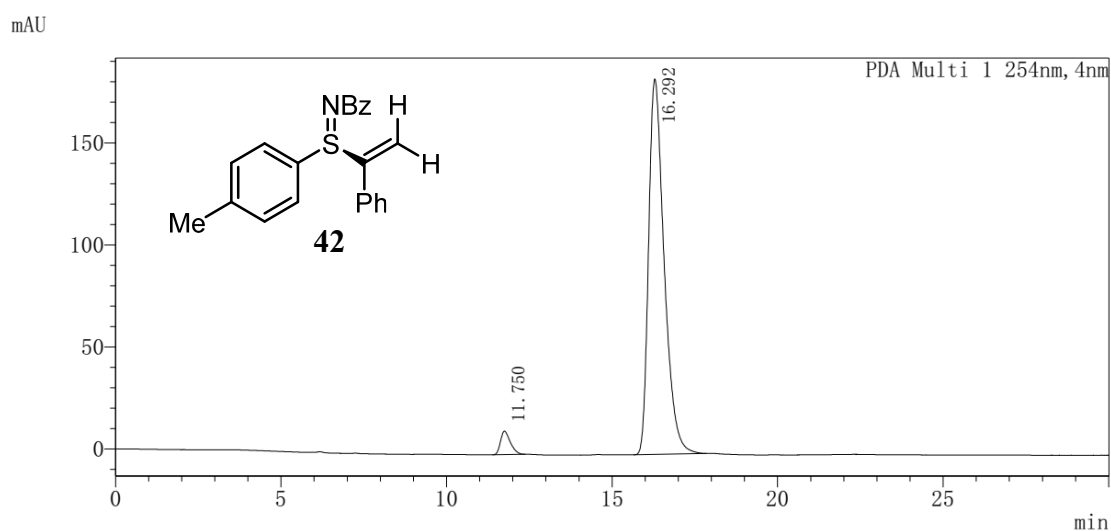
Peak#	Ret. Time	Area	Area%
1	17.266	183342	7.229
2	20.054	2352724	92.771



Peak Table

PDA Ch1 254nm

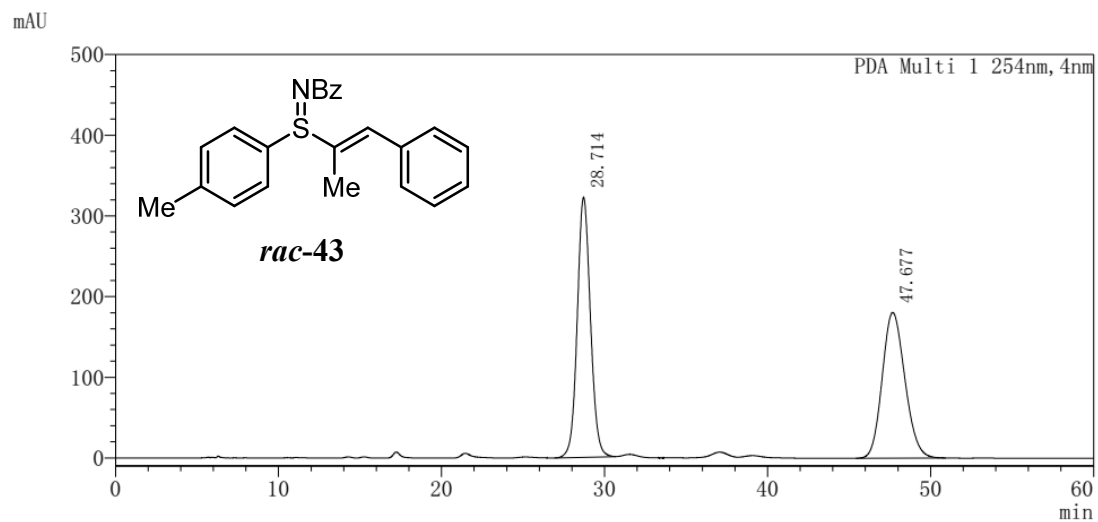
Peak#	Ret. Time	Area	Area%
1	11.818	25527832	50.235
2	16.453	25288957	49.765



Peak Table

PDA Ch1 254nm

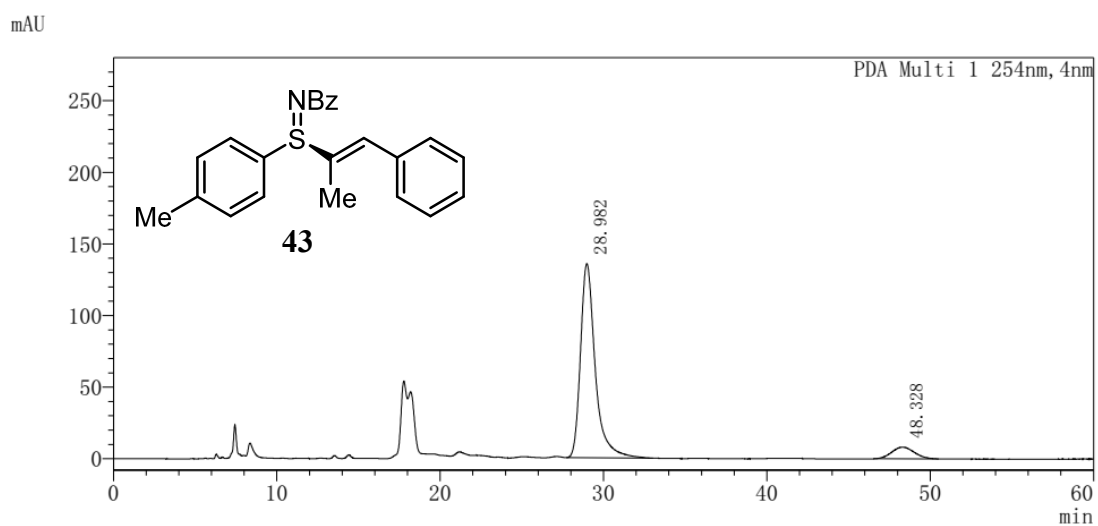
Peak#	Ret. Time	Area	Area%
1	11.750	237623	3.809
2	16.292	6000952	96.191



Peak Table

PDA Ch1 254nm

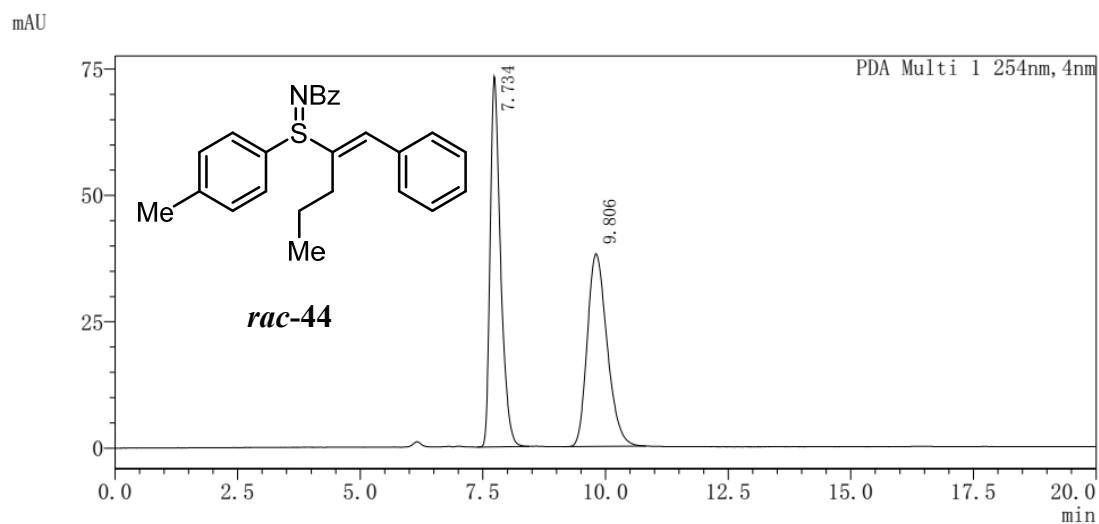
Peak#	Ret. Time	Area	Area%
1	28.714	18124538	50.949
2	47.677	17449268	49.051



Peak Table

PDA Ch1 254nm

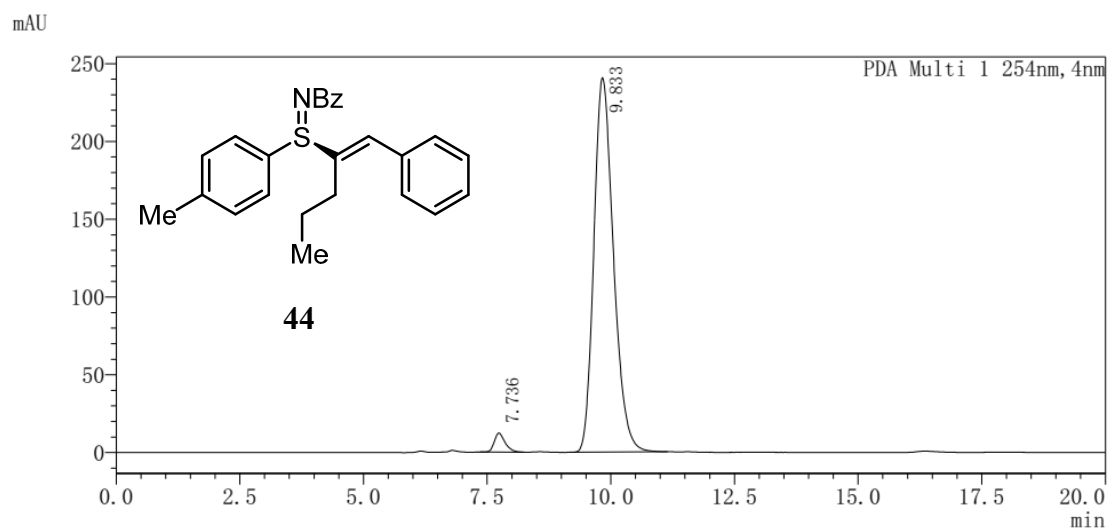
Peak#	Ret. Time	Area	Area%
1	28.982	8517830	91.548
2	48.328	786361	8.452



Peak Table

PDA Ch1 254nm

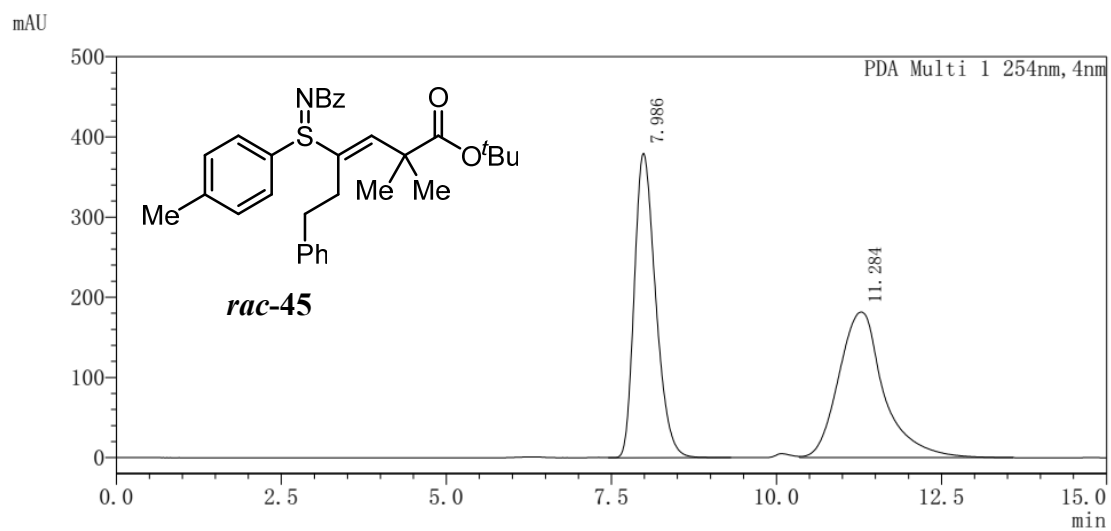
Peak#	Ret. Time	Area	Area%
1	7.734	1080242	50.139
2	9.806	1074271	49.861



Peak Table

PDA Ch1 254nm

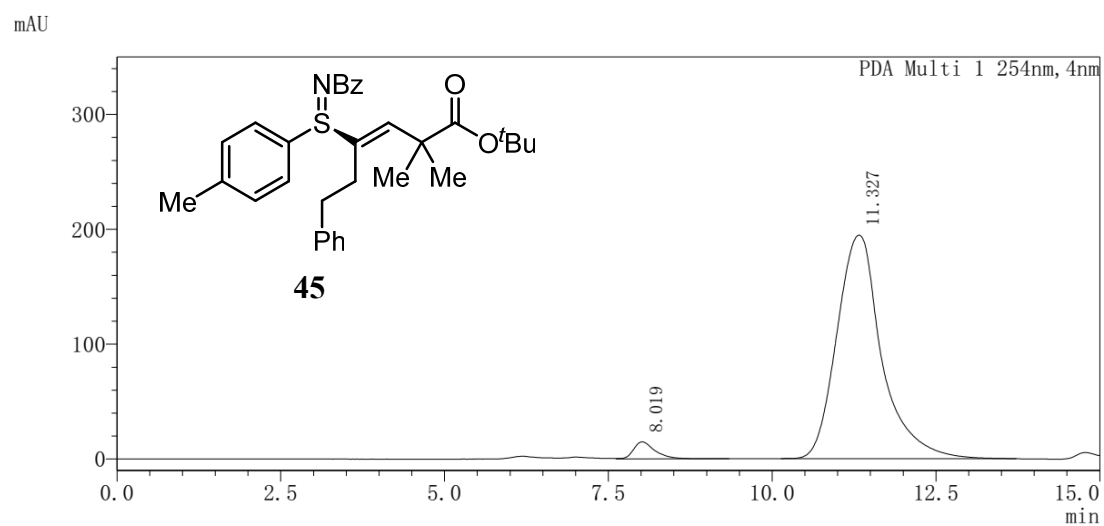
Peak#	Ret. Time	Area	Area%
1	7.736	186491	2.733
2	9.833	6636864	97.267



Peak Table

PDA Ch1 254nm

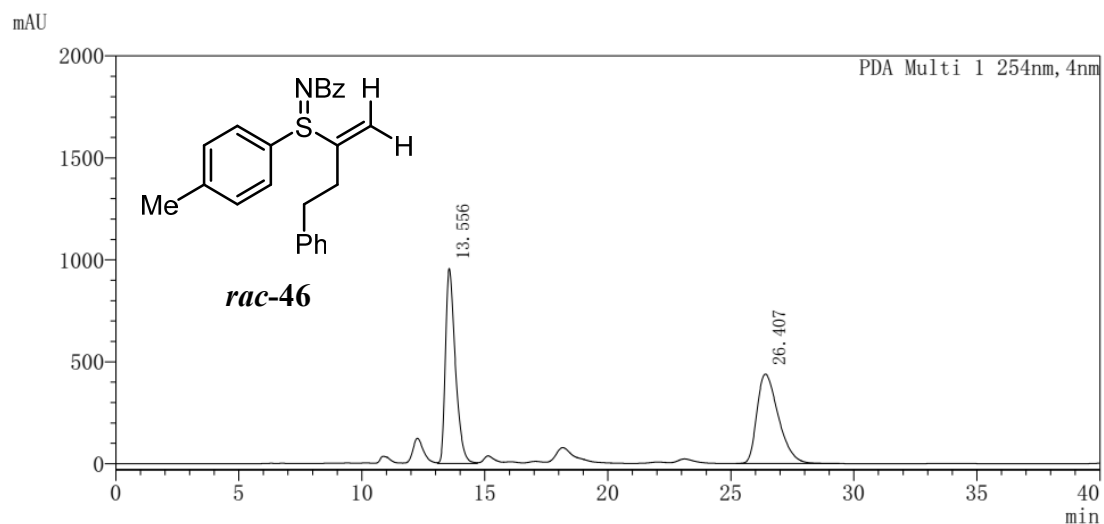
Peak#	Ret. Time	Area	Area%
1	7.986	8525655	49.695
2	11.284	8630255	50.305



Peak Table

PDA Ch1 254nm

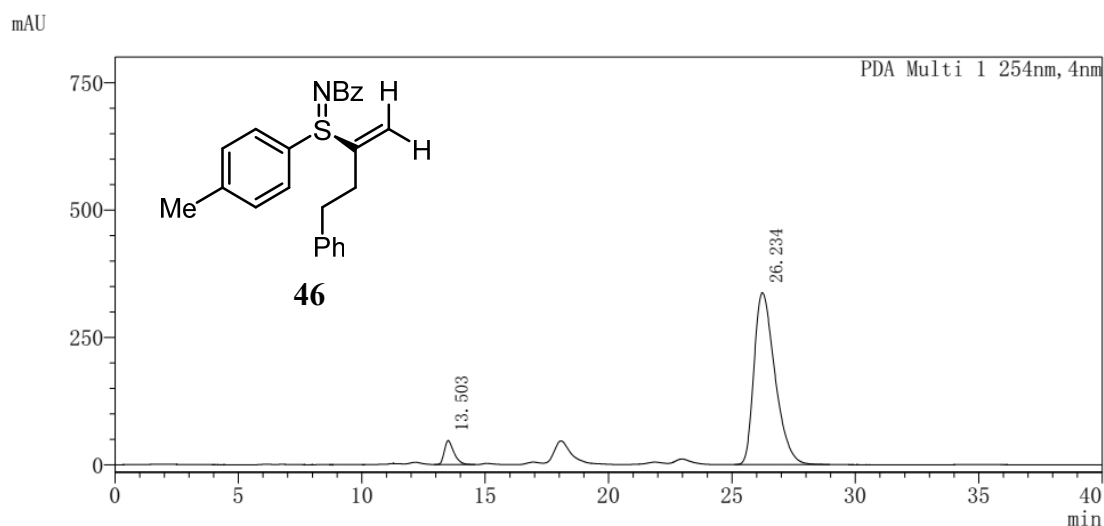
Peak#	Ret. Time	Area	Area%
1	8.019	334092	3.477
2	11.327	9274513	96.523



Peak Table

PDA Ch1 254nm

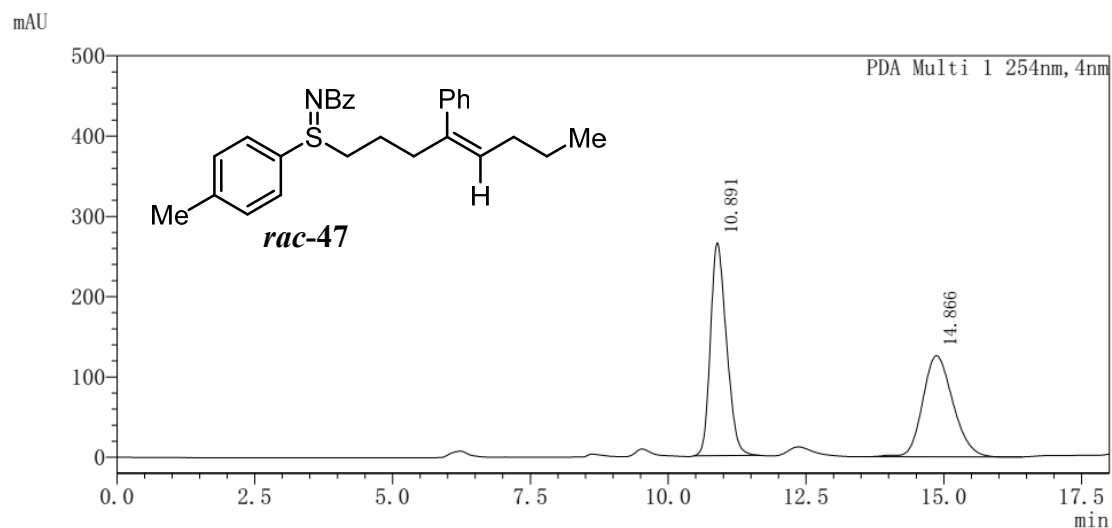
Peak#	Ret. Time	Area	Area%
1	13.556	26933352	50.228
2	26.407	26689007	49.772



Peak Table

PDA Ch1 254nm

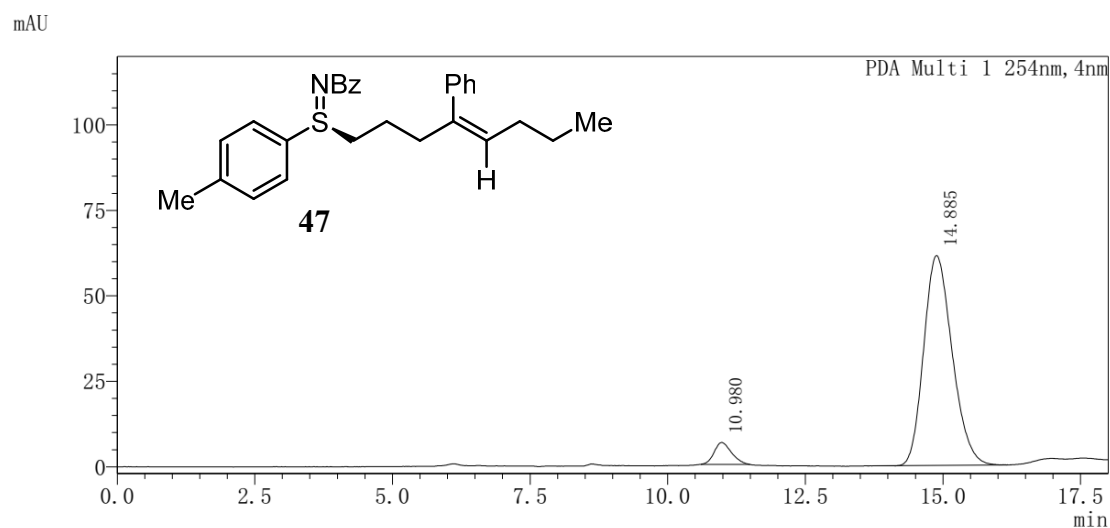
Peak#	Ret. Time	Area	Area%
1	13.503	1262256	5.959
2	26.234	19920562	94.041



Peak Table

PDA Ch1 254nm

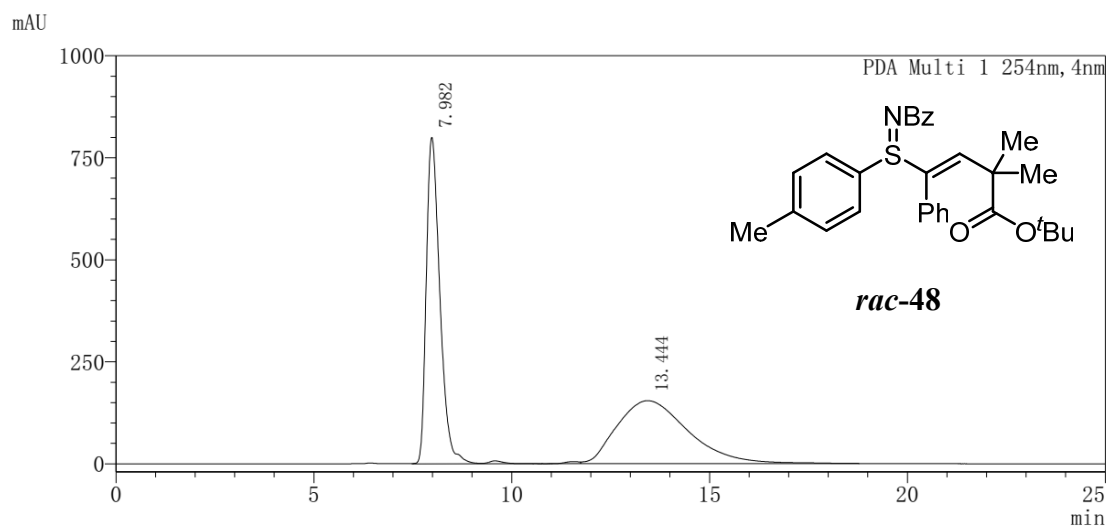
Peak#	Ret. Time	Area	Area%
1	10.891	5379600	52.899
2	14.866	4789960	47.101



Peak Table

PDA Ch1 254nm

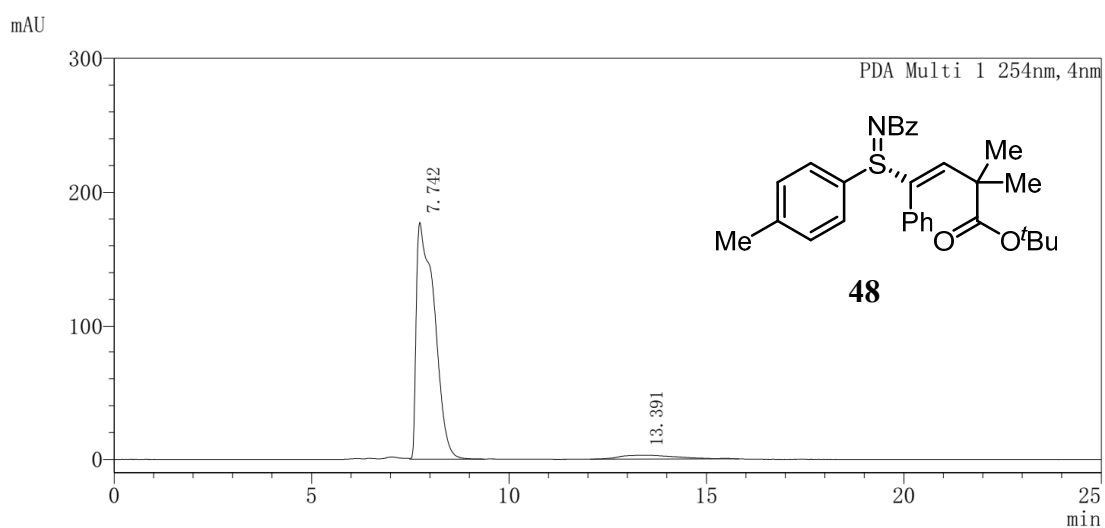
Peak#	Ret. Time	Area	Area%
1	10.980	140601	5.982
2	14.885	2209885	94.018



Peak Table

PDA Ch1 254nm

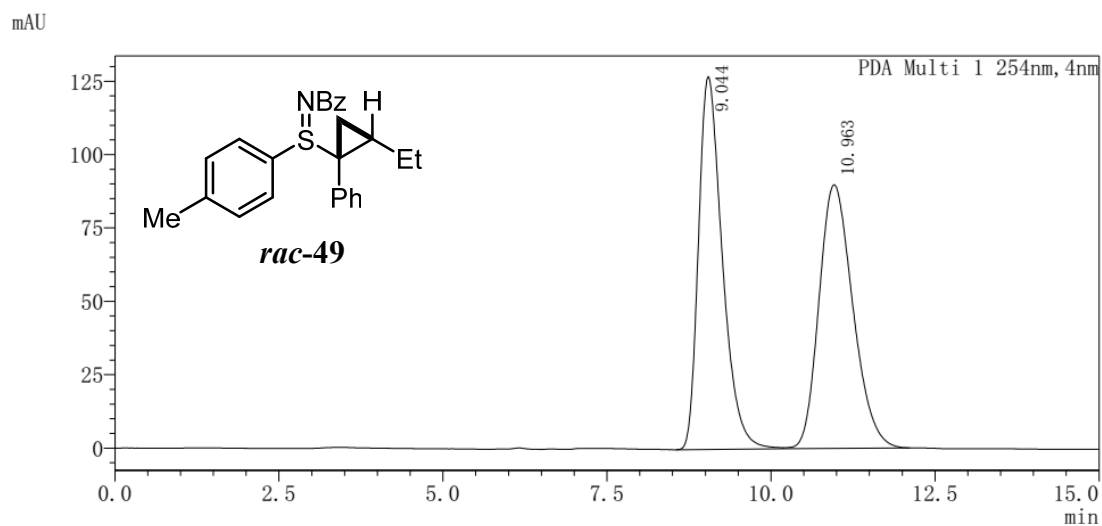
Peak#	Ret. Time	Area	Area%
1	7.982	19448916	50.178
2	13.444	19311271	49.822



Peak Table

PDA Ch1 254nm

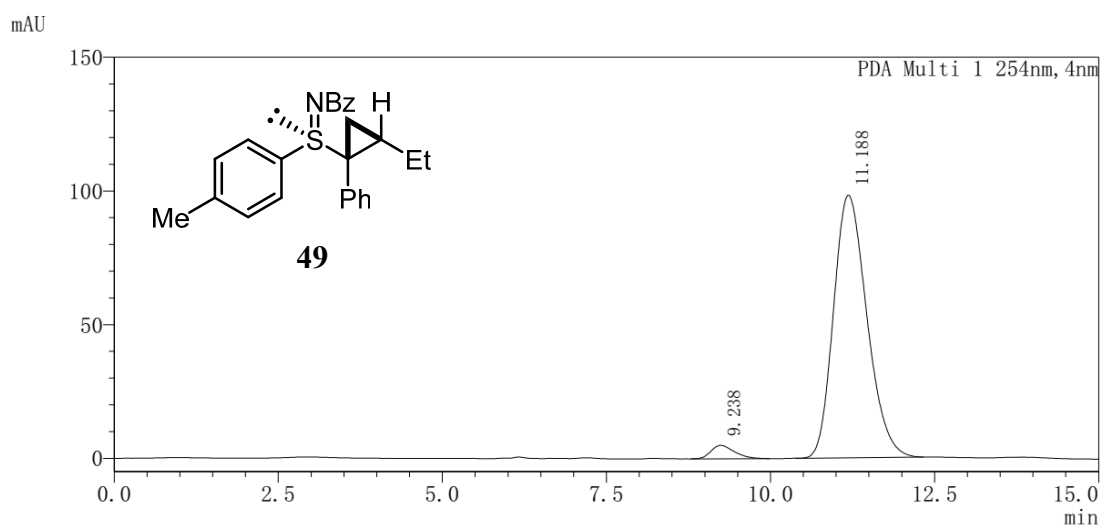
Peak#	Ret. Time	Area	Area%
1	7.742	5633509	95.054
2	13.391	293159	4.946



Peak Table

PDA Ch1 254nm

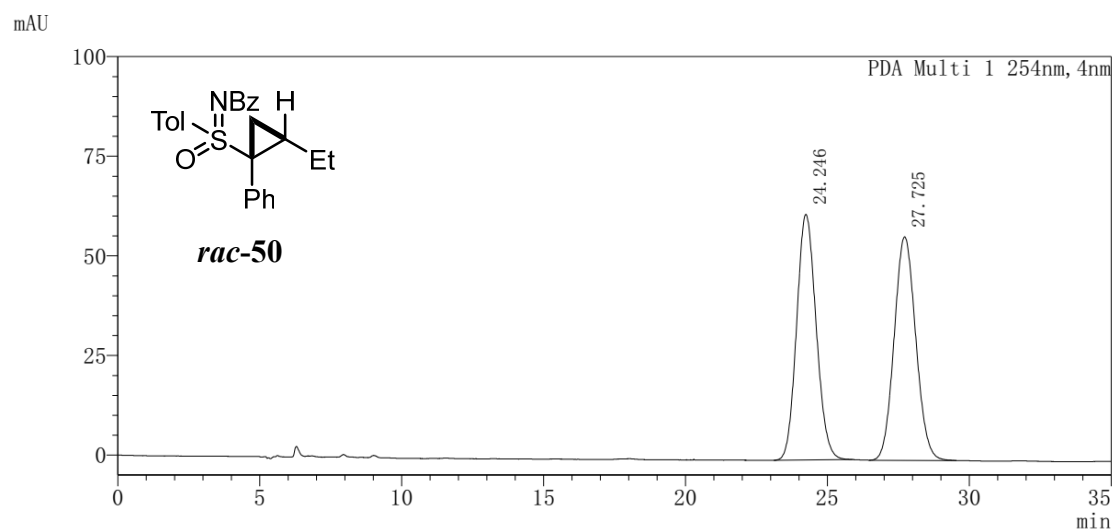
Peak#	Ret. Time	Area	Area%
1	9.044	3214387	49.890
2	10.963	3228560	50.110



Peak Table

PDA Ch1 254nm

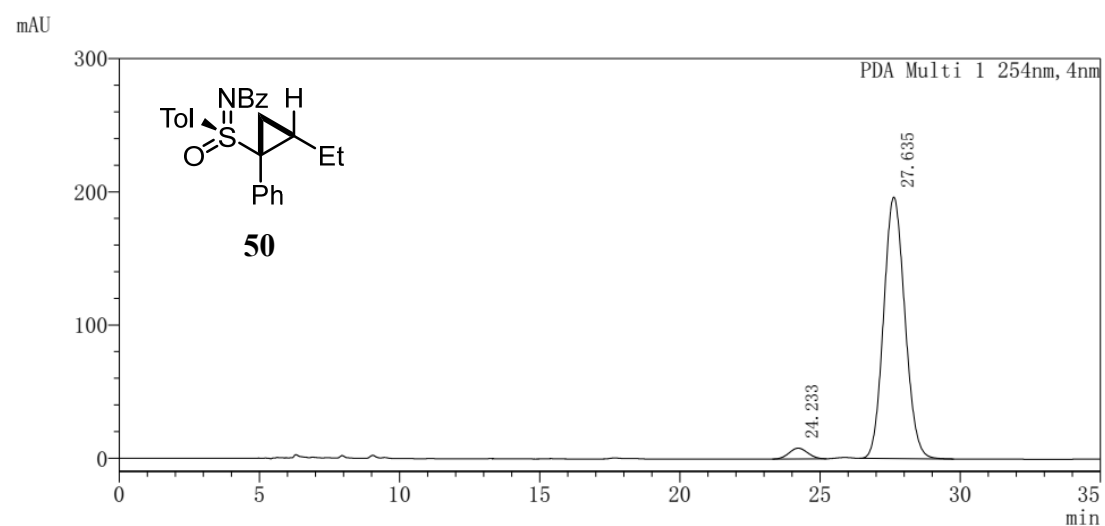
Peak#	Ret. Time	Area	Area%
1	9.238	124233	3.446
2	11.188	3480786	96.554



Peak Table

PDA Ch1 254nm

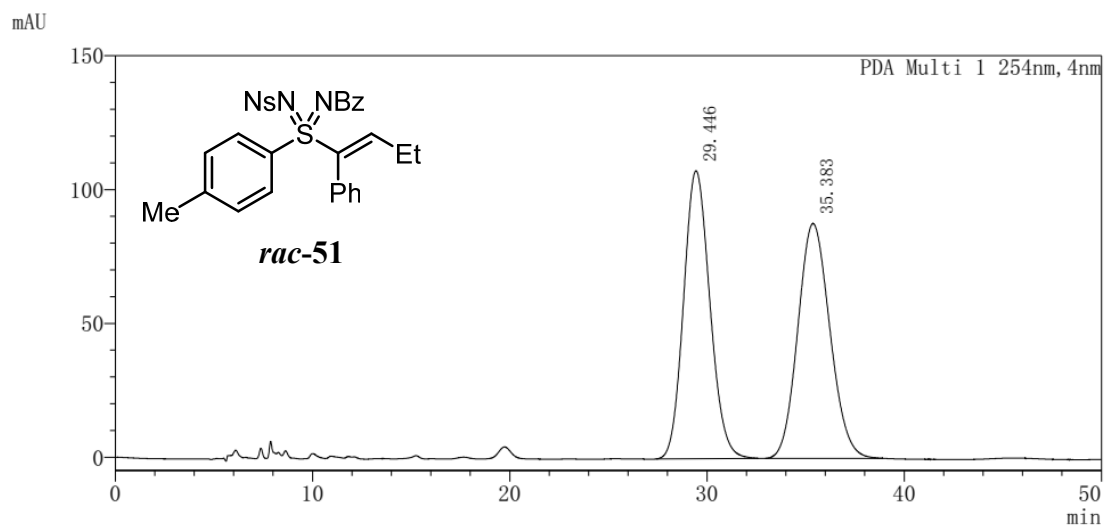
Peak#	Ret. Time	Area	Area%
1	24.246	2957241	49.924
2	27.725	2966272	50.076



Peak Table

PDA Ch1 254nm

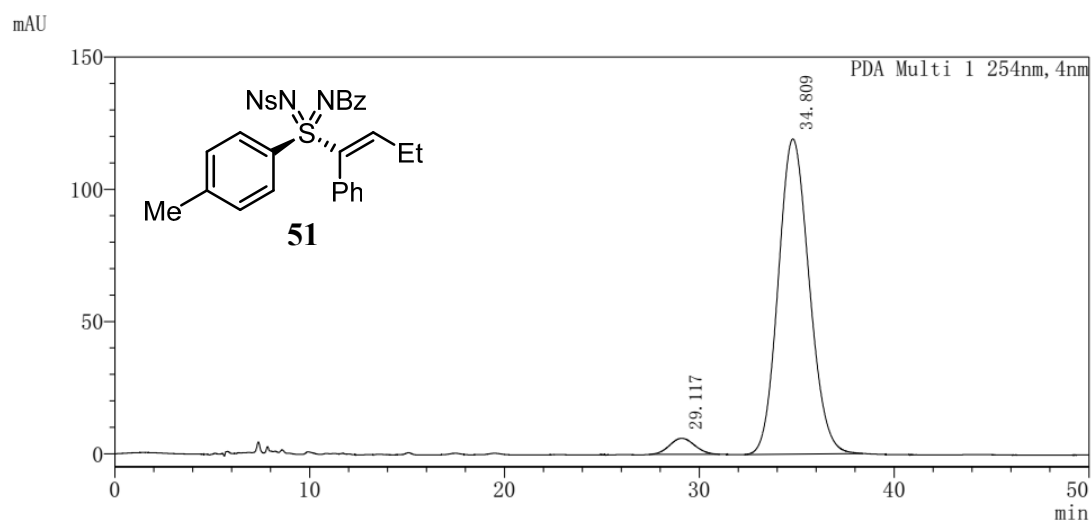
Peak#	Ret. Time	Area	Area%
1	24.233	371477	3.462
2	27.635	10359652	96.538



Peak Table

PDA Ch1 254nm

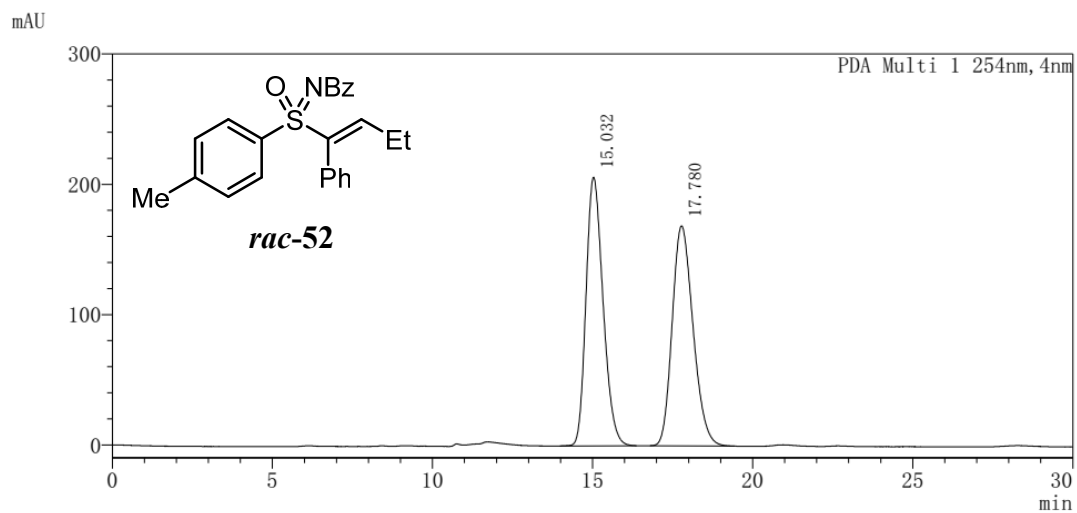
Peak#	Ret. Time	Area	Area%
1	29.446	9969949	50.115
2	35.383	9924375	49.885



Peak Table

PDA Ch1 254nm

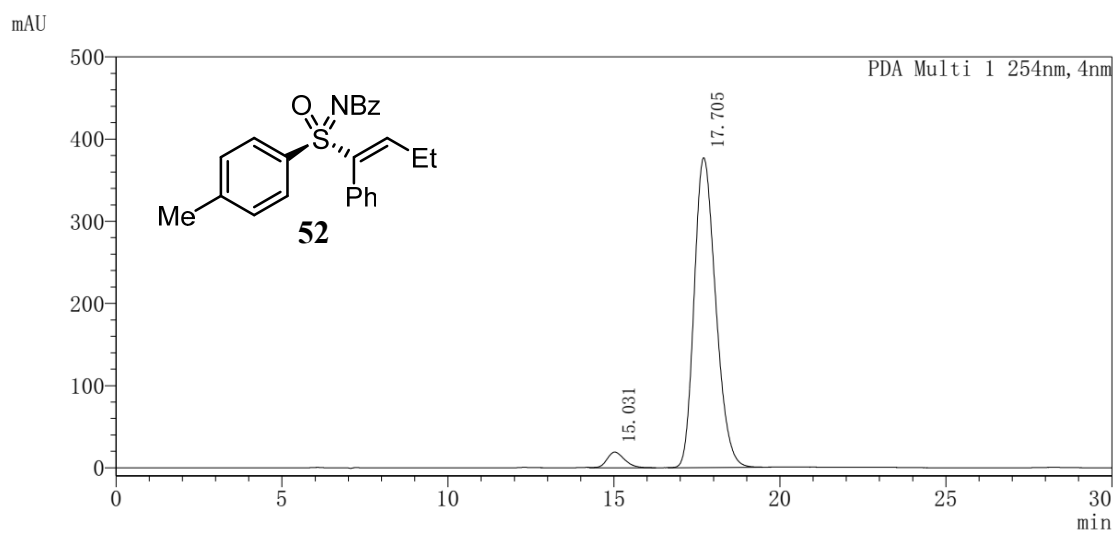
Peak#	Ret. Time	Area	Area%
1	29.117	545081	3.973
2	34.809	13175454	96.027



Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	15.032	7635877	49.892
2	17.780	7668976	50.108

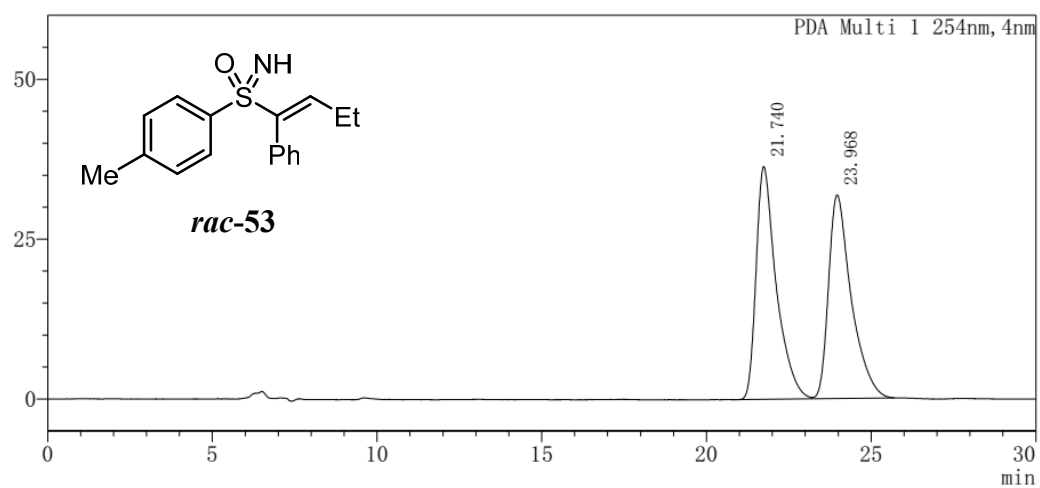


Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	15.031	697856	3.948
2	17.705	16977229	96.052

mAU

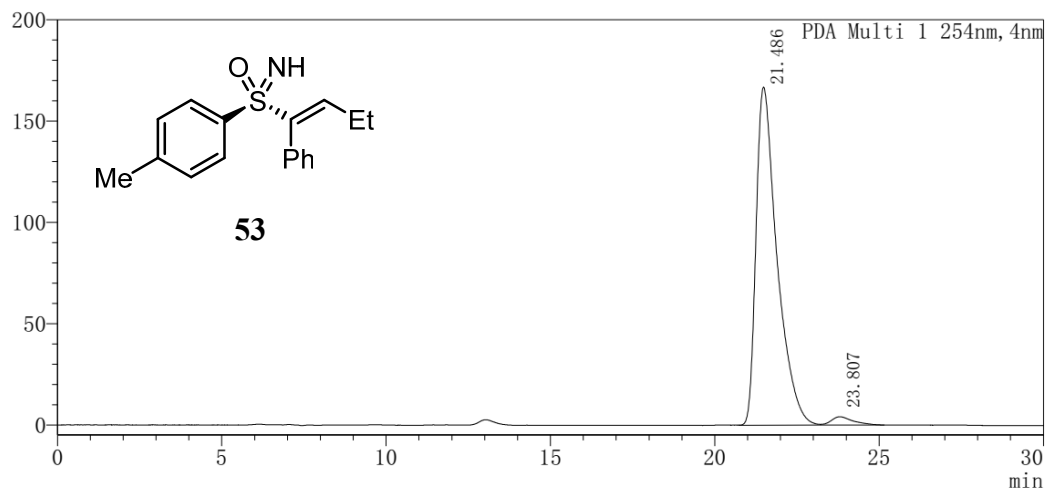


Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	21.740	1552713	50.365
2	23.968	1530181	49.635

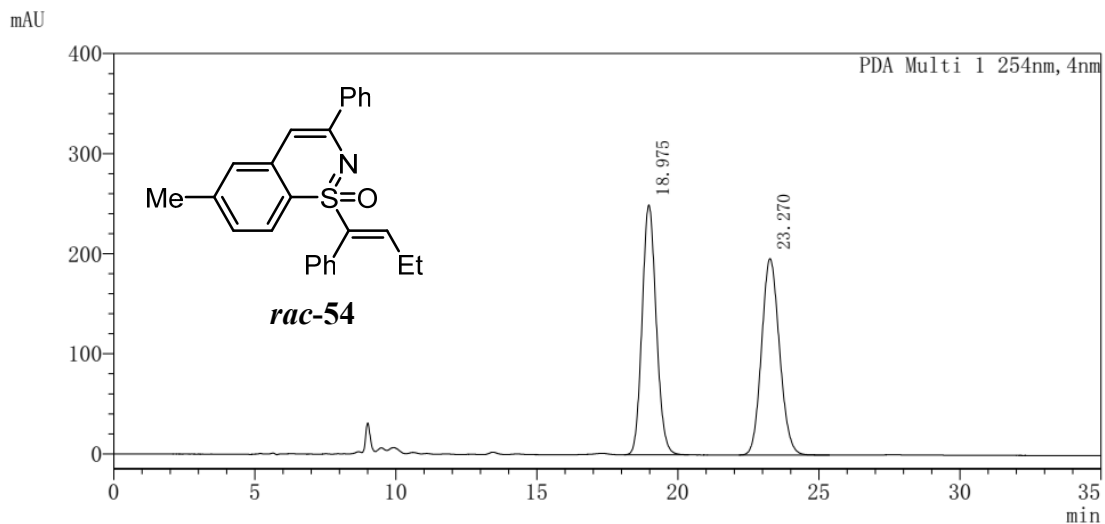
mAU



Peak Table

PDA Ch1 254nm

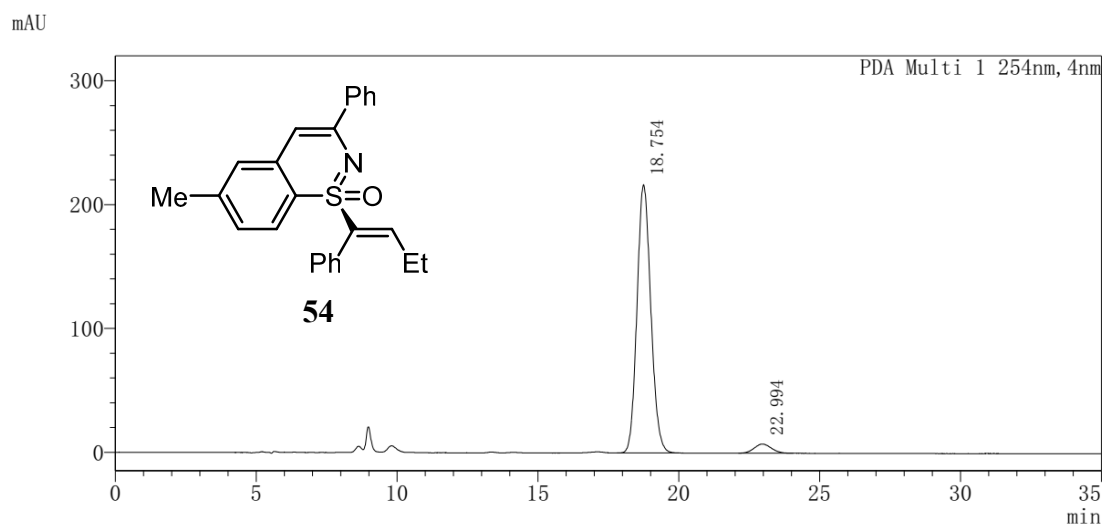
Peak#	Ret. Time	Area	Area%
1	21.486	7409132	97.486
2	23.807	191031	2.514



Peak Table

PDA Ch1 254nm

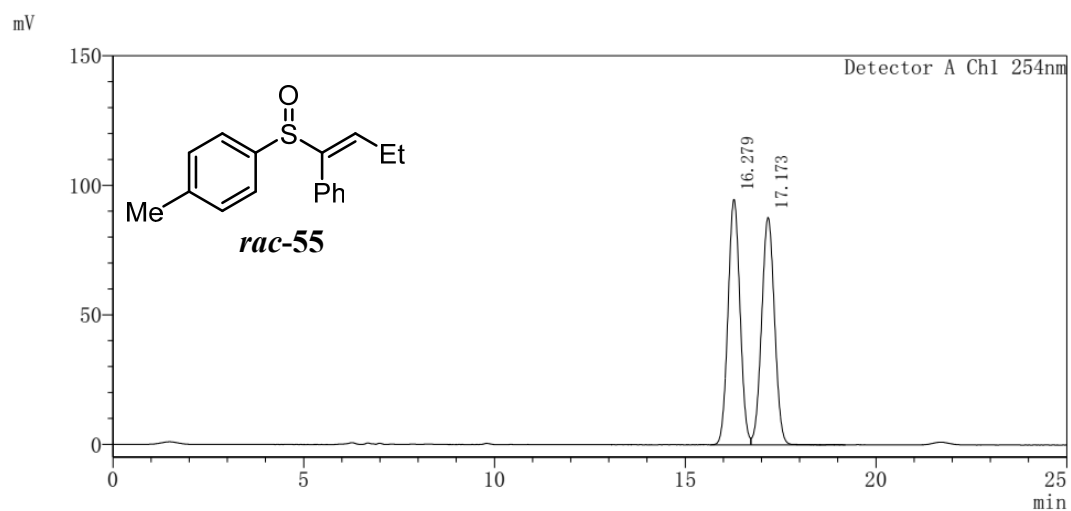
Peak#	Ret. Time	Area	Area%
1	18.975	8699546	50.034
2	23.270	8687832	49.966



Peak Table

PDA Ch1 254nm

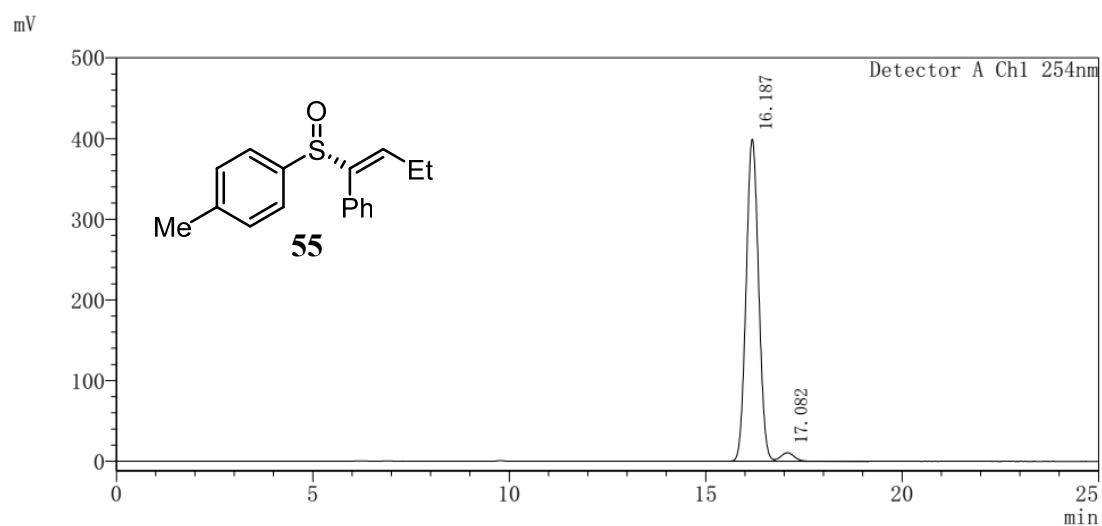
Peak#	Ret. Time	Area	Area%
1	18.754	7392799	95.892
2	22.994	316705	4.108



Peak Table

Detector A Ch1 254nm

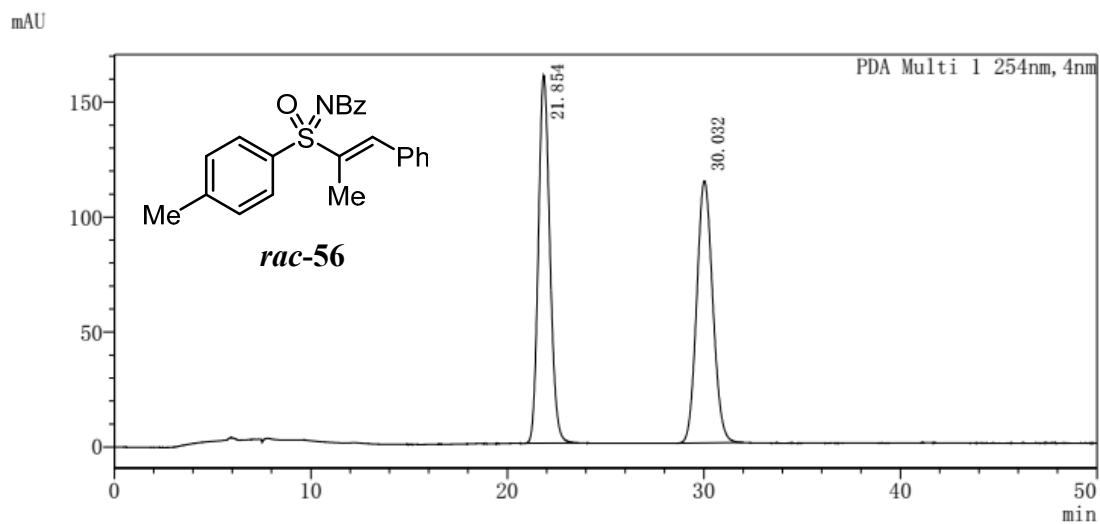
Peak#	Ret. Time	Area	Area%
1	16.279	2071307	50.017
2	17.173	2069865	49.983



Peak Table

Detector A Ch1 254nm

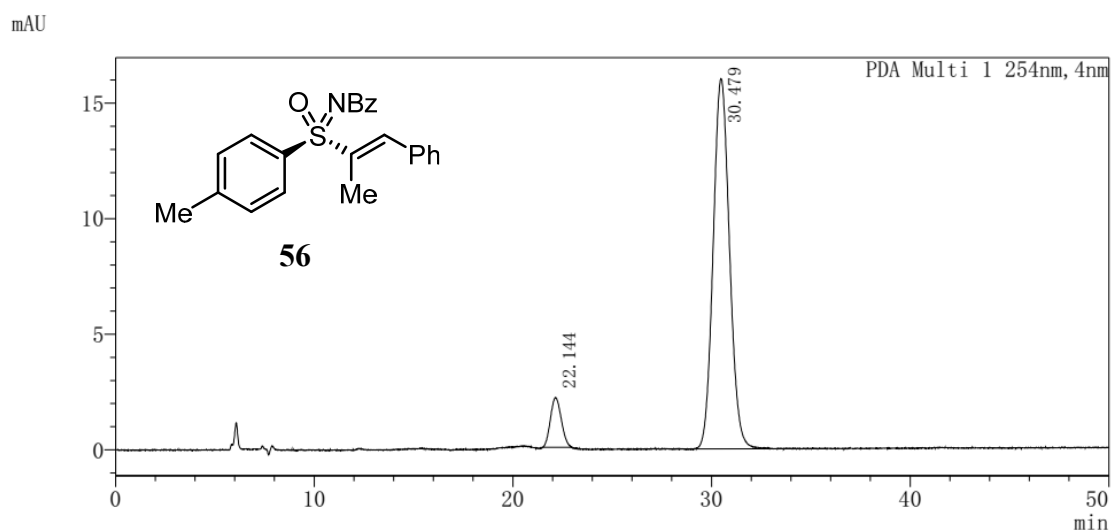
Peak#	Ret. Time	Area	Area%
1	16.187	8802695	97.036
2	17.082	268918	2.964



Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	21.854	6489602	50.097
2	30.032	6464554	49.903



Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	22.144	81942	8.194
2	30.479	918119	91.806