

Electronic Supplementary Information

HFIP-mediated 2-aza-Cope rearrangement: metal-free synthesis of α -substituted homoallylamines at ambient temperature

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2 General information

Nuclear Magnetic Resonance (NMR) spectra were recorded on a Bruker Model Avance 400 Fourier transform NMR spectrometer. The chemical shifts (δ) are reported in parts per million (ppm) with respect to the downfield of tetramethylsilane (TMS, served as internal standard; $\delta = 0.00$ ppm) in deuterated chloroform (CDCl_3) unless noted otherwise. All NMR measurements were carried out at room temperature unless otherwise stated. All coupling constants (J) are reported in Hertz (Hz). The following abbreviations are used to describe peak splitting patterns when appropriate: s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, dd = doublet of doublets, dt = doublet of triplets, ddd = doublet of doublet of doublets, ddt = doublet of doublet of triplets, m = multiplet, and br s = broad singlet.

High-resolution mass spectrometry (HRMS) samples were prepared by dissolving 0.1–3.0 mg of compound in acetonitrile and further diluting to a concentration of 10^{-5} – 10^{-6} M with 50% acetonitrile/50% H_2O . The samples were injected in the MS, using a CapLC system (Waters) and a nanoelectrospray source operated in positive ion mode at a potential of 1.5 or 1.7 kV. The eluent used was 30% A (0.1% formic acid in H_2O) and 70% B (0.1% formic acid in $\text{MeCN}/\text{H}_2\text{O}$ -95/5) at a flow rate of 6.0 mL/min. Samples were injected with an interval of 3 min. Before analysis, 2.0 mL of a 0.025% H_3PO_4 solution ($\text{MeOH}/\text{H}_2\text{O}$ -50/50) or 10.0 mL of 10^{-6} M deoxyadenosine solution ($\text{MeOH}/\text{H}_2\text{O}$ -50/50) was injected as a lock mass. Positive-ion mode accurate mass spectra were acquired using a Q-TOF instrument.

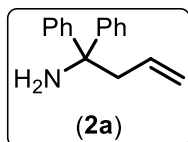
Melting points were measured on a BÜCHI B-545 capillary melting point apparatus.

All aldehydes used were purchased from commercial sources and used as received. The starting materials, 1,1-diphenylbut-3-en-1-amine (**2a**) and 2-methyl-1,1-diphenylbut-3-en-1-amine (**2b**) were prepared using known literature procedures (see Section 3.1 for detailed procedures). 4Å molecular sieves (powder) were purchased from Sigma-Aldrich and activated prior to use by drying in a vacuum oven at 200 °C for 24 h. 1,1,1,3,3,3-Hexafluoro-2-propanol (HFIP) was purchased from Fluorochem and used as received. All reactions were carried out in oven-dried vials.

3 Experimental procedures and compound characterization data

3.1 Synthesis of starting materials

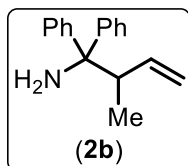
3.1.1 Synthesis of 1,1-Diphenylbut-3-en-1-amine (2a) [KG-0700]



This compound was prepared according to a literature procedure.¹ An oven-dried, 100 mL two-necked round-bottomed flask was charged with a Teflon-coated magnetic stir bar and benzophenone imine (6.0 mL, 35.76 mmol, 1.0 equiv.). The flask was equipped with a pressure equalizing addition funnel and connected to a reflux condenser which had been evacuated and refilled with argon three times. Then, 40 mL of dry 2-methyl tetrahydrofuran (2-MeTHF) was added and the flask cooled to 0 °C. Allylmagnesium chloride (46 mL at 1.7 M in THF, 79 mmol, 2.2 equiv.) was added into the additional funnel and then added dropwise to the stirring benzophenone imine solution over the course of 60 minutes at 0 °C. Subsequently, the reaction mixture was stirred up to room temperature and stirred for 18 hours, then it was cooled to 0 °C and quenched by the dropwise addition of ice cold solution of 1N aqueous NaOH, passed through a small plug of celite and concentrated in vacuo to remove solvent. Then, the resulting residue was dissolved in 30% aqueous AcOH at room temperature. The mixture was stirred at room temperature for 1 hour, methyl *tert*-butyl ether (MTBE) (15 mL) added, then partitioned in between MTBE and aqueous layer (two times). The aqueous layer was then neutralized with 2N aqueous NaOH and extracted with ethyl acetate. The ethyl acetate fractions were combined, dried using MgSO₄ and concentrated under vacuum affording the pure desired 1,1-diphenylbut-3-en-1-amine **2a** (6.4 g, 80%).

Pale yellow liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.39 – 7.37 (m, 4H), 7.30 – 7.27 (m, 4H), 7.21 – 7.17 (m, 2H), 5.53 (ddt, *J* = 17.2, 10.1, 7.1 Hz, 1H), 5.19 – 5.06 (m, 2H), 3.01 (d, *J* = 7.1 Hz, 2H), 1.80 (br s, 2H) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 148.2 (C), 134.2 (CH), 128.1 (CH), 126.6 (CH), 126.4 (CH), 119.1 (CH₂), 60.3 (C), 47.6 (CH₂) ppm. The spectroscopic data are in accordance with the literature.¹⁻³

3.1.2 Synthesis of 2-Methyl-1,1-diphenylbut-3-en-1-amine (2b) [KG-0790]



This compound was prepared according to a literature procedure.² An oven-dried, 100 mL two-necked round-bottomed flask was charged with a Teflon-coated magnetic stir bar and benzophenone imine (1.5 mL, 8.94 mmol, 1.0 equiv.). The flask was equipped with a pressure equalizing addition funnel and connected to a reflux condenser which had been evacuated and refilled with argon three times. Then, 15 mL of dry 2-MeTHF was added and the flask cooled to 0 °C. 2-Butenylmagnesium chloride (39.5 mL at 0.5 M in THF, 19.67 mmol, 2.2 equiv.) was added into the additional funnel and then added dropwise to the stirring

benzophenone imine solution over the course of 60 minutes at 0 °C. Subsequently, the reaction mixture was stirred up to room temperature and stirred for 18 hours, then it was cooled to 0 °C and quenched by the dropwise addition of ice cold solution of 1N aqueous NaOH, passed through a small plug of celite and concentrated in vacuo to remove solvent. Then, the resulting residue was dissolved in 30% aqueous AcOH at room temperature. The mixture was stirred at room temperature for 1 hour, MTBE (10 mL) added, then partitioned in between MTBE and aqueous layer (two times). The aqueous layer was then neutralized with 2N aqueous NaOH and extracted with ethyl acetate. The ethyl acetate fractions were combined, dried using MgSO₄ and concentrated under vacuum affording the pure desired 2-methyl-1,1-diphenylbut-3-en-1-amine **2b** (1.5 g, 71%).

Pale yellow liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.52 – 7.49 (m, 4H), 7.30 – 7.24 (m, 4H), 7.19 – 7.12 (m, 2H), 5.73 (ddd, *J* = 17.2, 10.6, 6.3 Hz, 1H), 5.11 – 5.05 (m, 2H), 3.51 (q, *J* = 6.6 Hz, 1H), 1.71 (br s, 2H), 1.00 (d, *J* = 6.8 Hz, 3H) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 147.6 (C), 146.9 (C), 139.9 (CH), 128.1 (CH), 128.0 (CH), 126.7 (CH), 126.6 (CH), 126.1 (CH), 126.0 (CH), 116.3 (CH₂), 63.1 (C), 44.4 (CH), 14.2 (CH₃) ppm. The spectroscopic data are in accordance with the literature.²⁻³

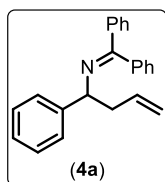
3.2 Synthesis of α -substituted homoallylamines (4a-4z and 4aa-4ao)

3.2.1 General experimental procedure

In an oven-dried 10 mL vial was charged with a Teflon-coated magnetic stir bar, 1,1-diphenylbut-3-en-1-amine (**2a** or **2b**) (0.5 mmol, 1.0 equiv.), aldehyde **1** (0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and 1,1,1,3,3,3-hexafluoro-2-propanol (HFIP) (0.5 mL, 9.5 equiv.). The vial was capped under air. The reaction mixture was then stirred for the indicated time (see manuscript Scheme 3) at room temperature. After the reaction time, the reaction mixture was filtered (to remove 4Å molecular sieves) and the solvent was removed under reduced pressure to afford pure α -substituted homoallylamine **4**.

Note : Aqueous work-up required for products **4u**, **4v**, **4w** and **4x** only. After the reaction time, the reaction mixture was filtered (to remove 4Å molecular sieves), the solvent was removed under reduced pressure, then the reaction mixture was diluted with 10 mL EtOAc (or DCM) and washed with 10 mL of 0.5 N aqueous NaOH solution. The aqueous layer was extracted with EtOAc (or DCM) (5, 5, 5 mL). Combined organic layers were dried using MgSO₄, filtered and concentrated under reduced pressure to afford pure α -substituted homoallylamine (**4u-4x**).

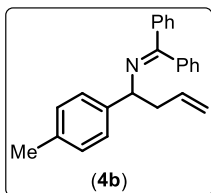
3.2.2 Characterization data of α -substituted homoallylamines (4a-4z and 4aa-4ao)



1,1-Diphenyl-*N*-(1-phenylbut-3-en-1-yl)methanimine (4a) [KG-0743/0768]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), benzaldehyde (**1a**, 53 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 3

hours at room temperature. 1,1-Diphenyl-*N*-(1-phenylbut-3-en-1-yl)methanimine (**4a**) was obtained in 99% (154 mg) yield. The spectroscopic data are in accordance with the literature.^{3,6,7}

White solid; **mp**: 86–88 °C; **¹H NMR** (400 MHz, CDCl₃): δ 7.68 – 7.65 (m, 2H), 7.42 – 7.38 (m, 3H), 7.36 – 7.24 (m, 7H), 7.21 – 7.16 (m, 1H), 7.07 – 7.04 (m, 2H), 5.65 (ddt, J = 17.2, 10.1, 7.1 Hz, 1H), 4.95 (ddd, J = 10.1, 9.2, 1.5 Hz, 2H), 4.43 (dd, J = 7.8, 5.5 Hz, 1H), 2.68 (dt, J = 15.0, 7.6 Hz, 1H, diastereotopic allyl-CH₂), 2.62 – 2.53 (m, 1H, diastereotopic allyl-CH₂) ppm; **¹³C NMR** (101 MHz, CDCl₃): δ 166.6 (C=N), 144.5 (C), 140.1 (C), 137.1 (C), 135.8 (CH), 130.7 (CH), 129.8 (CH), 128.6(CH), 128.27 (CH), 128.26 (CH), 128.0 (CH), 127.9 (CH), 127.1 (CH), 126.7 (CH), 116.7 (CH₂), 66.5 (CH), 43.9 (CH₂) ppm; **HRMS** (ESI) m/z calculated for C₂₃H₂₂N [M+H]⁺ : 312.1747; found 312.1756.

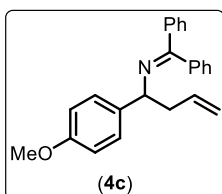


***N*-[1-(4-methylphenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (4b) [KG-**

0750]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (2a, 112 mg, 0.5 mmol, 1.0 equiv.), *p*-tolualdehyde (1b, 60 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture

was vigorously stirred for 6 hours at room temperature. *N*-[1-(4-methylphenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (4b) was obtained in 99% (160 mg) yield. The spectroscopic data are in accordance with the literature.^{3,7}

Colourless liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.67 – 7.63 (m, 2H), 7.41 – 7.38 (m, 3H), 7.36 – 7.28 (m, 3H), 7.20 (d, *J* = 8.0 Hz, 2H), 7.09 – 7.04 (m, 4H), 5.64 (ddt, *J* = 17.2, 10.1, 7.1 Hz, 1H), 4.99 – 4.92 (m, 2H), 4.40 (dd, *J* = 7.8, 5.5 Hz, 1H), 2.67 (dt, *J* = 14.8, 7.6 Hz, 1H, diastereotopic allyl-CH₂), 2.59 – 2.52 (m, 1H, diastereotopic allyl-CH₂), 2.31 (s, 3H) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 166.4 (C=N), 141.5 (C), 140.1 (C), 137.2 (C), 136.2 (C), 135.9 (CH), 130.1 (CH), 129.8 (CH), 129.0 (CH), 128.6 (CH), 128.3 (CH), 128.2 (CH), 127.9 (CH), 127.0 (CH), 116.5 (CH₂), 66.2 (CH), 43.9 (CH₂), 21.1 (CH₃) ppm; HRMS (ESI) *m/z* calculated for C₂₄H₂₄N [M+H]⁺ : 326.1903; found 326.1912.

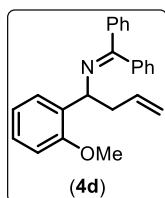


***N*-[1-(4-methoxyphenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (4c) [KG-**

0707]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (2a, 112 mg, 0.5 mmol, 1.0 equiv.), *p*-anisaldehyde (1c, 68 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture

was vigorously stirred for 18 hours at room temperature. *N*-[1-(4-methoxyphenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (4c) was obtained in 94% (160 mg) yield. The spectroscopic data are in accordance with the literature.^{3,6}

Brown liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.66 – 7.63 (m, 2H), 7.42 – 7.37 (m, 3H), 7.35 – 7.26 (m, 3H), 7.24 – 7.21 (m, 2H), 7.07 – 7.04 (m, 2H), 6.83 (d, *J* = 8.7 Hz, 2H), 5.64 (ddt, *J* = 17.2, 10.2, 7.1 Hz, 1H), 4.99 – 4.92 (m, 2H), 4.38 (dd, *J* = 7.7, 5.7 Hz, 1H), 3.76 (s, 3H), 2.66 (dt, *J* = 14.9, 7.5 Hz, 1H, diastereotopic allyl-CH₂), 2.58 – 2.51 (m, 1H, diastereotopic allyl-CH₂) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 166.4 (C=N), 158.4 (C), 140.1 (C), 137.2 (C), 136.7 (C), 135.8 (CH), 129.8 (CH), 128.6 (CH), 128.3 (CH), 128.2 (CH), 128.1 (CH), 127.94 (CH), 127.89 (CH), 116.6 (CH₂), 113.7 (CH), 65.9 (CH), 55.2 (CH₃), 43.9 (CH₂) ppm; HRMS (ESI) *m/z* calculated for C₂₄H₂₄NO [M+H]⁺ : 342.1852; found 342.1846.

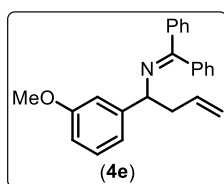


***N*-[1-(2-methoxyphenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (4d) [KG-**

0708]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), *o*-anisaldehyde (**1d**, 68 mg, 0.5 mmol, 1.0 equiv.), 4Å

molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 18 hours at room temperature. *N*-[1-(2-methoxyphenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (**4d**) was obtained in 95% (163 mg) yield. The spectroscopic data are in accordance with the literature.³

Brown liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.68 – 7.66 (m, 3H), 7.36 – 7.27 (m, 6H), 7.16 (td, *J* = 8.1, 1.7 Hz, 1H), 7.00 (dd, *J* = 6.4, 3.1 Hz, 2H), 6.93 (t, *J* = 7.5 Hz, 1H), 6.77 (d, *J* = 8.1 Hz, 1H), 5.71 (ddt, *J* = 17.2, 10.2, 7.0 Hz, 1H), 5.01 – 4.88 (m, 3H), 3.60 (s, 3H), 2.67 – 2.54 (m, 2H, overlapping diastereotopic allyl-CH₂) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 166.9 (C=N), 155.9 (C), 140.4 (C), 137.4 (C), 136.4 (CH), 133.4 (C), 129.7 (CH), 128.6 (CH), 128.3 (CH), 128.1 (CH), 128.0 (CH), 127.9 (CH), 127.8 (CH), 127.2 (CH), 120.7 (CH), 116.1 (CH₂), 110.4 (CH), 58.8 (CH₃), 55.2 (CH), 42.6 (CH₂) ppm; HRMS (ESI) *m/z* calculated for C₂₄H₂₄NO [M+H]⁺: 342.1852; found 342.1859.

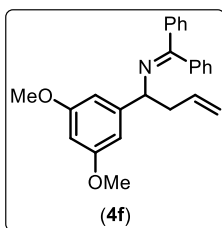


***N*-[1-(3-methoxyphenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (4e) [KG-**

0709]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), *m*-anisaldehyde (**1e**, 68 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture

was vigorously stirred for 18 hours at room temperature. *N*-[1-(3-methoxyphenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (**4e**) was obtained in 98% (167 mg) yield. New compound according to a Sci-finder search.

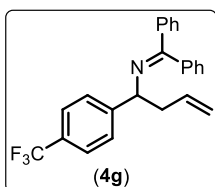
Brown liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.67 – 7.65 (m, 2H), 7.41 – 7.39 (m, 3H), 7.36 – 7.28 (m, 3H), 7.22 – 7.17 (m, 1H), 7.07 – 7.05 (m, 2H), 6.92 – 6.87 (m, 2H), 6.75 (dd, *J* = 8.2, 2.5 Hz, 1H), 5.65 (ddt, *J* = 17.2, 10.2, 7.1 Hz, 1H), 5.01 – 4.93 (m, 2H), 4.40 (dd, *J* = 7.8, 5.5 Hz, 1H), 3.77 (s, 3H), 2.68 (dt, *J* = 14.3, 7.6 Hz, 1H, diastereotopic allyl-CH₂), 2.57 (dt, *J* = 12.7, 6.1 Hz, 1H, diastereotopic allyl-CH₂) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 166.9 (C=N), 159.6 (C), 146.1 (C), 140.0 (C), 137.1 (C), 135.7 (CH), 129.9 (CH), 129.2 (CH), 128.6 (CH), 128.3 (CH), 128.0 (CH), 127.9 (CH), 126.6 (CH), 119.5 (CH), 116.7 (C), 112.9 (CH), 112.0 (CH), 66.4 (CH₂), 55.2 (CH), 43.9 (CH₂) ppm; HRMS (ESI) *m/z* calculated for C₂₄H₂₄NO [M+H]⁺: 342.1852; found 342.1862.



***N*-[1-(3,5-dimethoxyphenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (4f)**

[KG-0751]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), 3,5-dimethoxybenzaldehyde (**1f**, 83 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 6 hours at room temperature. *N*-[1-(3,5-dimethoxyphenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (**4f**) was obtained in 80% (149 mg) yield. The spectroscopic data are in accordance with the literature.^{3,7}

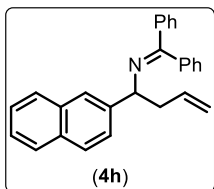
Colourless liquid; **¹H NMR** (400 MHz, CDCl₃): δ 7.66 (dd, *J* = 8.1, 1.4 Hz, 2H), 7.43 – 7.38 (m, 3H), 7.37 – 7.28 (m, 3H), 7.07 (dd, *J* = 6.5, 2.9 Hz, 2H), 6.50 (d, *J* = 2.3 Hz, 2H), 6.33 (t, *J* = 2.3 Hz, 1H), 5.65 (ddt, *J* = 17.2, 10.2, 7.1 Hz, 1H), 5.01 – 4.93 (m, 2H), 4.35 (dd, *J* = 7.9, 5.4 Hz, 1H), 3.76 (s, 6H), 2.67 (dt, *J* = 15.1, 7.6 Hz, 1H, diastereotopic allyl-CH₂), 2.59 – 2.53 (m, 1H, diastereotopic allyl-CH₂) ppm; **¹³C NMR** (101 MHz, CDCl₃): δ 166.8 (C=N), 160.7 (C), 147.0 (C), 140.0 (C), 137.1 (C), 135.7 (CH), 129.9 (CH), 128.6 (CH), 128.3 (CH), 127.96 (CH), 127.95 (CH), 116.7 (CH₂), 105.2 (CH), 98.7 (CH), 66.6 (CH), 55.3 (CH₃), 43.9 (CH₂) ppm; **HRMS** (ESI) *m/z* calculated for C₂₅H₂₆NO₂ [M+H]⁺: 372.1958; found 372.1951.



1,1-Diphenyl-*N*-{1-[4-(trifluoromethyl)phenyl]but-3-en-1-yl}methanimine (4g)

[KG-0739]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), *p*-(trifluoromethyl)benzaldehyde (**1g**, 87 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 6 hours at room temperature. 1,1-Diphenyl-*N*-{1-[4-(trifluoromethyl)phenyl]but-3-en-1-yl}methanimine (**4g**) was obtained in 96% (182 mg) yield. The spectroscopic data are in accordance with the literature.^{1,3}

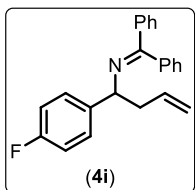
Brown liquid; **¹H NMR** (400 MHz, CDCl₃): δ 7.68 – 7.66 (m, 2H), 7.54 (d, *J* = 8.2 Hz, 2H), 7.45 – 7.41 (m, 5H), 7.39 – 7.30 (m, 3H), 7.03 (dd, *J* = 6.5, 2.9 Hz, 2H), 5.68 – 5.58 (m, 1H), 4.99 – 4.95 (m, 2H), 4.48 (dd, *J* = 7.5, 5.7 Hz, 1H), 2.67 (dt, *J* = 14.9, 7.5 Hz, 1H, diastereotopic allyl-CH₂), 2.59 – 2.53 (m, 1H, diastereotopic allyl-CH₂) ppm; **¹³C NMR** (101 MHz, CDCl₃): δ 167.5 (C=N), 148.5 (C), 139.8 (C), 136.9 (C), 135.0 (CH), 130.1 (CH), 129.2 (CH), 128.6 (CH), 128.5 (CH), 128.4 (CH), 128.0 (CH), 127.8 (CH), 127.5 (C), 125.2 (q, *J* = 3.8 Hz), 124.3 (q, *J* = 270 Hz, CF₃), 117.2 (CH₂), 66.1 (CH), 43.8 (CH₂) ppm; **¹⁹F NMR** (376 MHz, CDCl₃): -62.32 ppm **HRMS** (ESI) *m/z* calculated for C₂₄H₂₁NF₃ [M+H]⁺: 380.1621; found 380.1632.



***N*-[1-(naphthalen-2-yl)but-3-en-1-yl]-1,1-diphenylmethanimine (4h) [KG-0763]:**

The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), 2-naphthaldehyde (**1h**, 78 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 6 hours at room temperature. *N*-[1-(naphthalen-2-yl)but-3-en-1-yl]-1,1-diphenylmethanimine (**4h**) was obtained in 80% (145 mg) yield. The spectroscopic data are in accordance with the literature.¹

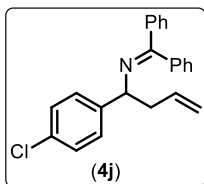
Colourless liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.79 – 7.75 (m, 3H), 7.71 – 7.66 (m, 3H), 7.54 (dd, *J* = 8.5, 0.9 Hz, 1H), 7.43 – 7.29 (m, 8H), 7.06 (dd, *J* = 6.3, 2.7 Hz, 2H), 5.68 (ddt, *J* = 17.2, 10.1, 7.1 Hz, 1H), 5.02 – 4.94 (m, 2H), 4.60 (dd, *J* = 7.6, 5.7 Hz, 1H), 2.77 (dt, *J* = 14.7, 7.5 Hz, 1H, diastereotopic allyl-CH₂), 2.70 – 2.64 (m, diastereotopic allyl-CH₂) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 167.1 (C=N), 142.0 (C), 140.1 (C), 137.1 (C), 135.7 (CH), 133.5 (C), 132.7 (C), 129.9 (CH), 129.1 (CH), 128.6 (CH), 128.3 (CH), 128.0 (CH), 127.9 (CH), 127.8 (CH), 127.6 (CH), 125.8 (CH), 125.7 (CH), 125.5 (CH), 125.4 (CH), 122.8 (CH), 116.8 (CH₂), 66.7 (CH), 43.9 (CH₂) ppm; HRMS (ESI) *m/z* calculated for C₂₀H₂₄N [M+H]⁺ : 362.1903; found 362.1914.



***N*-[1-(4-fluorophenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (4i) [KG-0752]:**

The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), *p*-fluorobenzaldehyde (**1i**, 62 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 6 hours at room temperature. *N*-[1-(4-fluorophenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (**4i**) was obtained in 98% (161 mg) yield. The spectroscopic data are in accordance with the literature.^{3,7}

White solid; *mp*: 54 – 56 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.67 – 7.64 (m, 2H), 7.43 – 7.41 (m, 3H), 7.39 – 7.29 (m, 3H), 7.28 – 7.24 (m, 2H), 7.05 – 7.03 (m, 2H), 6.99 – 6.94 (m, 2H), 5.62 (ddt, *J* = 17.3, 10.2, 7.1 Hz, 1H), 4.98 – 4.93 (m, 2H), 4.40 (dd, *J* = 7.6, 5.7 Hz, 1H), 2.64 (dt, *J* = 14.8, 7.5 Hz, 1H, diastereotopic allyl-CH₂), 2.57 – 2.50 (m, 1H, diastereotopic allyl-CH₂) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 166.8 (C=N), 161.7 (d, *J* = 244 Hz, C-F), 140.2 (d, *J* = 3.1 Hz, C), 139.9 (C), 137.0 (C), 135.5 (CH), 130.0 (CH), 128.6 (CH), 128.58 (CH), 128.5 (CH), 128.4 (CH), 128.0 (CH), 127.8 (CH), 116.9 (CH₂), 115.0 (d, *J* = 21 Hz, CH), 65.7 (CH), 44.0 (CH₂) ppm; ¹⁹F NMR (376 MHz, CDCl₃): -116.3 ppm; HRMS (ESI) *m/z* calculated for C₂₃H₂₁FN [M+H]⁺ : 330.1653; found 330.1665.

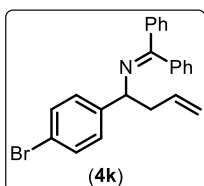


***N*-[1-(4-chlorophenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (4j) [KG-**

0738]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), *p*-chlorobenzaldehyde (**1j**, 70 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture

was vigorously stirred for 6 hours at room temperature. *N*-[1-(4-chlorophenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (**4j**) was obtained in 98% (169 mg) yield. The spectroscopic data are in accordance with the literature.³

White solid; **mp**: 58 – 60 °C; **¹H NMR** (400 MHz, CDCl₃): δ 7.66 (d, *J* = 7.1 Hz, 2H), 7.41 – 7.39 (m, 3H), 7.37 – 7.29 (m, 3H), 7.26 – 7.20 (m, 4H), 7.03 (d, *J* = 3.2 Hz, 2H), 5.67 – 5.57 (m, 1H), 4.98 – 4.93 (m, 2H), 4.39 (dd, *J* = 7.6, 5.7 Hz, 1H), 2.64 (dt, *J* = 14.7, 7.5 Hz, 1H, diastereotopic allylCH₂), 2.57 – 2.50 (m, 1H, diastereotopic allyl-CH₂) ppm; **¹³C NMR** (101 MHz, CDCl₃): δ 167.1 (C=N), 143.0 (C), 139.8 (C), 137.0 (C), 135.3 (CH), 132.3 (C), 130.0 (CH), 129.5 (CH), 128.6 (CH), 128.5 (CH), 128.4 (CH), 128.37 (CH), 128.0 (CH), 127.8 (CH), 117.0 (CH₂), 65.8 (CH), 43.9 (CH₂) ppm; **HRMS** (ESI) *m/z* calculated for C₂₃H₂₁NCl [M+H]⁺ : 346.1357; found 346.1360.

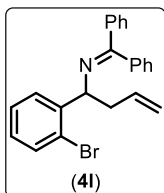


***N*-[1-(4-bromophenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (4k) [KG-**

0713]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), *p*-bromobenzaldehyde (**1k**, 93 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture

was vigorously stirred for 6 hours at room temperature. *N*-[1-(4-bromophenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (**4k**) was obtained in 99% (193 mg) yield. The spectroscopic data are in accordance with the literature.³

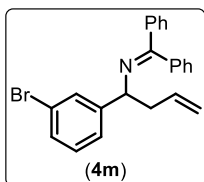
Brown liquid; **¹H NMR** (400 MHz, CDCl₃): δ 7.65 (dd, *J* = 8.2, 1.4 Hz, 2H), 7.41 – 7.38 (m, 5H), 7.36 – 7.28 (m, 3H), 7.18 (d, *J* = 8.4 Hz, 2H), 7.04 – 7.02 (m, 2H), 5.62 (ddt, *J* = 17.4, 10.3, 7.1 Hz, 1H), 4.98 – 4.93 (m, 2H), 4.38 (dd, *J* = 7.6, 5.7 Hz, 1H), 2.63 (dt, *J* = 14.0, 7.5 Hz, 1H, diastereotopic allyl-CH₂), 2.57 – 2.48 (m, 1H, diastereotopic allyl-CH₂) ppm; **¹³C NMR** (101 MHz, CDCl₃): δ 167.1 (C=N), 143.5 (C), 139.8 (C), 136.9 (C), 135.2 (CH), 131.3 (CH), 130.0 (CH), 128.9 (CH), 128.6 (CH), 128.4 (CH), 128.3 (CH), 128.0 (CH), 127.7 (CH), 120.4 (C), 117.0 (CH₂), 65.9 (CH), 43.8 (CH₂) ppm; **HRMS** (ESI) *m/z* calculated for C₂₃H₂₁NBr [M+H]⁺ : 390.0852; found 390.0867.



***N*-[1-(2-bromophenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (4l) [KG-0714]:**

The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv), *o*-bromobenzaldehyde (**1l**, 93 mg, 0.5 mmol, 1.0 equiv), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 6 hours at room temperature. *N*-[1-(2-bromophenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (**4l**) was obtained in 99% (193 mg) yield. New compound according to a Sci-finder search.

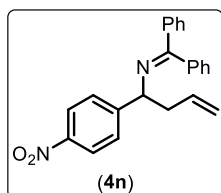
Brown liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.82 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.70 (d, *J* = 7.8 Hz, 2H), 7.44 (d, *J* = 8.0 Hz, 1H), 7.40 – 7.27 (m, 7H), 7.06 – 7.02 (m, 1H), 7.00 (dd, *J* = 6.4, 2.7 Hz, 2H), 5.71 (ddt, *J* = 17.2, 10.1, 7.1 Hz, 1H), 5.00 – 4.90 (m, 3H), 2.63 – 2.52 (m, 2H, overlapping diastereotopic allyl-CH₂) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 167.9 (C=N), 143.9 (C), 140.0 (C), 137.0 (C), 135.4 (CH), 132.4 (CH), 130.0 (CH), 129.8 (CH), 128.7 (CH), 128.4 (CH), 128.3 (CH), 128.0 (CH), 127.9 (CH), 127.8 (CH), 127.5 (CH), 122.4 (C), 116.8 (CH₂), 64.6 (CH), 42.9 (CH₂) ppm; HRMS (ESI) *m/z* calculated for C₂₃H₂₁OBr [M+H]⁺: 390.0852; found 390.0834.



***N*-[1-(3-bromophenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (4m) [KG-0715]:**

The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), *m*-bromobenzaldehyde (**1m**, 93 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 6 hours at room temperature. *N*-[1-(3-bromophenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (**4m**) was obtained in 99% (193 mg) yield. The spectroscopic data are in accordance with the literature.¹

White solid; *mp*: 95-97 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.66 (d, *J* = 7.1 Hz, 2H), 7.48 – 7.31 (m, 8H), 7.24 – 7.21 (m, 1H), 7.14 (t, *J* = 7.7 Hz, 1H), 7.04 (d, *J* = 3.3 Hz, 2H), 5.67 – 5.56 (m, 1H), 4.99 – 4.94 (m, 2H), 4.40 – 4.37 (m, 1H), 2.64 (dt, *J* = 14.6, 7.5 Hz, 1H), 2.56 – 2.50 (m, 1H) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 167.3 (C), 146.9 (C), 139.8 (C), 136.9 (C), 135.2 (CH), 130.2 (CH), 130.1 (CH), 129.84 (CH), 129.83 (CH), 128.6 (CH), 128.41 (CH), 128.40 (CH), 128.0 (CH), 127.8 (CH), 125.8 (CH), 122.4 (C), 117.1 (CH₂), 66.0 (CH), 43.9 (CH₂) ppm; HRMS (ESI) *m/z* calculated for C₂₃H₂₁OBr [M+H]⁺: 390.0852; found 390.0864.

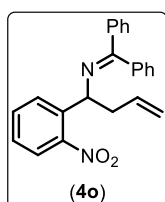


***N*-[1-(4-nitrophenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (4n) [KG-**

0710]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), *p*-nitrobenzaldehyde (**1n**, 76 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction

mixture was vigorously stirred for 6 hours at room temperature. *N*-[1-(4-nitrophenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (**4n**) was obtained in 94% (167 mg) yield. The spectroscopic data are in accordance with the literature.^{3,7}

Brown liquid; ¹H NMR (400 MHz, CDCl₃): δ 8.14 (d, *J* = 8.8 Hz, 2H), 7.69 – 7.66 (m, 2H), 7.48 (d, *J* = 8.7 Hz, 2H), 7.44 – 7.39 (m, 3H), 7.39 – 7.31 (m, 3H), 7.03 – 7.01 (m, 2H), 5.68 – 5.58 (m, 1H), 4.99 – 4.94 (m, 2H), 4.54 (dd, *J* = 7.4, 5.8 Hz, 1H), 2.66 (dt, *J* = 13.9, 7.4 Hz, 1H, diastereotopic allyl-CH₂), 2.59 – 2.53 (m, 1H, diastereotopic allyl-CH₂) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 168.1 (C=N), 152.0 (C), 146.9 (C), 139.5 (C), 136.7 (C), 134.5 (CH), 130.3 (CH), 128.63 (CH), 128.61 (CH), 128.5 (CH), 128.1 (CH), 128.0 (CH), 127.6 (CH), 123.6 (CH), 117.6 (CH₂), 65.9 (CH), 43.8 (CH₂) ppm; HRMS (ESI) *m/z* calculated for C₂₃H₂₁N₂O₂ [M+H]⁺ : 357.1598; found 357.1608.

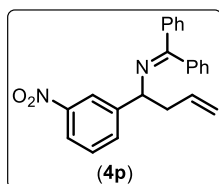


***N*-[1-(2-nitrophenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (4o) [KG-0711]:**

The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), *o*-nitrobenzaldehyde (**1o**, 76 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously

stirred for 12 hours at room temperature. *N*-[1-(2-nitrophenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (**4o**) was obtained in 95% (170 mg) yield. New compound according to a Sci-finder search.

Brown liquid; ¹H NMR (400 MHz, CDCl₃): δ 8.07 (d, *J* = 7.9 Hz, 1H), 7.71 – 7.67 (m, 3H), 7.56 (t, *J* = 7.6 Hz, 1H), 7.40 – 7.29 (m, 7H), 6.91 (dd, *J* = 7.4, 1.6 Hz, 2H), 5.71 (ddt, *J* = 17.3, 10.2, 7.2 Hz, 1H), 5.01 – 4.96 (m, 3H), 2.74 – 2.62 (m, 2H, overlapping diastereotopic allyl-CH₂) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 168.8 (C=N), 148.4 (C), 139.6 (C), 139.4 (C), 136.7 (C), 134.9 (CH), 132.7 (CH), 130.2 (CH), 130.0 (CH), 128.6 (CH), 128.5 (CH), 128.3 (CH), 128.0 (CH), 127.3 (CH), 127.2 (CH), 123.7 (CH), 117.4 (CH₂), 60.6 (CH), 43.6 (CH₂) ppm; HRMS (ESI) *m/z* calculated for C₂₃H₂₁N₂O₂ [M+H]⁺ : 357.1598; found 357.1603.

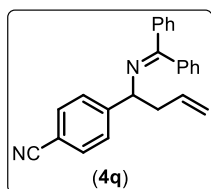


***N*-[1-(3-nitrophenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (4p) [KG-**

0712]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), *m*-nitrobenzaldehyde (**1p**, 76 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg), HFIP (0.5 mL). The reaction mixture

was vigorously stirred for 6 hours at room temperature. *N*-[1-(3-nitrophenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (**4p**) was obtained in 98% (175 mg) yield. The spectroscopic data are in accordance with the literature.⁷

Brown liquid; ¹H NMR (400 MHz, CDCl₃): δ 8.18 (d, *J* = 1.7 Hz, 1H), 8.08 – 8.04 (m, 1H), 7.69 – 7.66 (m, 3H), 7.47 – 7.43 (m, 4H), 7.40 – 7.31 (m, 3H), 7.05 – 7.03 (m, 2H), 5.63 (ddt, *J* = 16.1, 11.5, 7.2 Hz, 1H), 4.99 – 4.94 (m, 2H), 4.56 – 4.53 (m, 1H), 2.70 – 2.63 (m, 1H, diastereotopic allyl-CH₂), 2.61 – 2.56 (m, 1H, diastereotopic allyl-CH₂) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 167.9 (C=N), 148.3 (C), 146.5 (C), 139.5 (C), 136.7 (C), 134.5 (CH), 133.5 (CH), 130.3 (CH), 129.1 (CH), 128.7 (CH), 128.6 (CH), 128.57 (CH), 128.1 (CH), 127.6 (CH), 122.3 (CH), 121.9 (CH), 117.7 (CH₂), 65.7 (CH), 43.8 (CH₂) ppm; HRMS (ESI) *m/z* calculated for C₂₃H₂₁N₂O₂ [M+H]⁺ : 357.1598; found 357.1596.

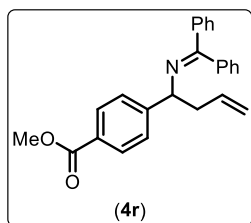


4{1-[(Diphenylmethylidene)amino]but-3-en-1-yl}benzonitrile (4q) [KG-0742]:

The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), 4-cyanobenzaldehyde (**1q**, 66 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg), HFIP (0.5 mL). The reaction mixture was

vigorously stirred for 6 hours at room temperature. 4{1-[(Diphenylmethylidene)amino]but-3-en-1-yl}benzonitrile (**4q**) was obtained in 94% (159 mg) yield. The spectroscopic data are in accordance with the literature.^{3,6}

Colourless liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.67 – 7.64 (m, 2H), 7.57 (d, *J* = 8.2 Hz, 2H), 7.43 – 7.31 (m, 8H), 7.02 (dd, *J* = 6.4, 2.8 Hz, 2H), 5.61 (ddt, *J* = 15.9, 11.5, 7.2 Hz, 1H), 4.98 – 4.94 (m, 2H), 4.47 (dd, *J* = 7.3, 5.9 Hz, 1H), 2.64 (dt, *J* = 14.7, 7.4 Hz, 1H, diastereotopic allyl-CH₂), 2.57 – 2.50 (m, 1H, diastereotopic allyl-CH₂) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 167.9 (C=N), 149.9 (C), 139.6 (C), 136.7 (C), 134.7 (CH), 132.2 (CH), 130.3 (CH), 128.6 (CH), 128.6 (CH), 128.5 (CH), 128.1 (CH), 128.0 (CH), 127.7 (CH), 119.0 (C), 117.5 (CH₂), 110.6 (C), 66.1 (CH), 43.8 (CH₂) ppm; HRMS (ESI) *m/z* calculated for C₂₄H₂₁N₂ [M+H]⁺ : 337.1699; found 337.1707.

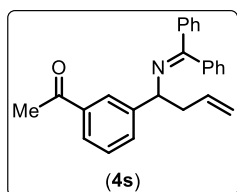


Methyl 4-{1-[(diphenylmethylidene)amino]but-3-en-1-yl}benzoate (4r)

[KG-0718]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), methyl 4-formylbenzoate (**1r**, 82 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 6 hours at room temperature. Methyl

4-{1-[(diphenylmethylidene)amino]but-3-en-1-yl}benzoate (**4r**) was obtained in 99% (183 mg) yield. The spectroscopic data are in accordance with the literature.^{3,7}

Colourless liquid; **¹H NMR** (400 MHz, CDCl₃): δ 7.97 (d, *J* = 8.3 Hz, 2H), 7.67 (dd, *J* = 8.2, 1.4 Hz, 2H), 7.42 – 7.30 (m, 8H), 7.02 (dd, *J* = 6.5, 2.9 Hz, 2H), 5.64 (ddt, *J* = 17.3, 10.3, 7.1 Hz, 1H), 4.98 – 4.94 (m, 2H), 4.48 (dd, *J* = 7.6, 5.6 Hz, 1H), 3.89 (s, 3H), 2.67 (dt, *J* = 14.8, 7.5 Hz, 1H, diastereotopic allyl-CH₂), 2.60 – 2.53 (m, 1H, diastereotopic allyl-CH₂) ppm; **¹³C NMR** (101 MHz, CDCl₃): δ 167.4 (C=N), 167.1 (C), 149.8 (C), 139.8 (C), 136.9 (C), 135.2 (CH), 130.1 (CH), 129.7 (CH), 128.64 (C), 128.64 (CH), 128.42 (CH), 128.39 (CH), 128.0 (CH), 127.8 (CH), 127.2 (CH), 117.1 (CH₂), 66.3 (CH), 52.0 (CH₃), 43.8 (CH₂) ppm; **HRMS** (ESI) *m/z* calculated for C₂₅H₂₄NO₂ [M+H]⁺ : 370.1802; found 370.1813.

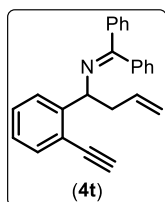


1-(3-{1-[(diphenylmethylidene)amino]but-3-en-1-yl}phenyl)ethan-1-one (4s)

[KG-0717]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), 3-formylacetophenone (**1s**, 74 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The

reaction mixture was vigorously stirred for 12 hours at room temperature. 1-(3-{1-[(diphenylmethylidene)amino]but-3-en-1-yl}phenyl)ethan-1-one (**4s**) was obtained in 98% (173 mg) yield. New compound according to a Sci-finder search.

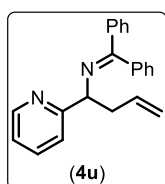
Colourless liquid; **¹H NMR** (400 MHz, CDCl₃): δ 7.89 (d, *J* = 8.2 Hz, 1H), 7.67 (d, *J* = 6.9 Hz, 2H), 7.49 – 7.30 (m, 8H), 7.03 (d, *J* = 3.5 Hz, 2H), 5.69 – 5.59 (m, 1H), 4.99 – 4.95 (m, 2H), 4.49 (dd, *J* = 7.1, 5.9 Hz, 1H), 2.73 – 2.60 (m, 2H, overlapping diastereotopic allyl-CH₂), 2.57 (s, 3H) ppm; **¹³C NMR** (101 MHz, CDCl₃): δ 197.8 (C=O), 167.5 (C=N), 150.0 (C), 139.8 (C), 136.9 (C), 135.8 (C), 135.1 (CH), 132.4 (CH), 130.1 (CH), 128.6 (CH), 128.5 (CH), 128.45 (CH), 128.41 (CH), 128.3 (CH), 128.0 (CH), 127.7 (CH), 127.3 (CH), 117.2 (CH₂), 66.3 (CH), 43.8 (CH₂), 26.6 (CH₃) ppm; **HRMS** (ESI) *m/z* calculated for C₂₅H₂₄NO [M+H]⁺ : 354.1852; found 354.1846.



***N*-[1-(2-ethynylphenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (**4t**) [KG-0704]:**

The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), 2-ethynylbenzaldehyde (**1t**, 65 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 6 hours at room temperature. *N*-[1-(2-ethynylphenyl)but-3-en-1-yl]-1,1-diphenylmethanimine (**4t**) was obtained in 93% (156 mg) yield. New compound according to a Sci-finder search.

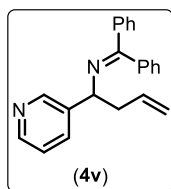
Black liquid; **¹H NMR** (400 MHz, CDCl₃): δ 7.81 (d, *J* = 7.7 Hz, 1H), 7.69 (d, *J* = 6.7 Hz, 2H), 7.40 – 7.30 (m, 8H), 7.15 (t, *J* = 7.2 Hz, 1H), 7.03 (dd, *J* = 6.4, 2.9 Hz, 2H), 5.69 (ddt, *J* = 17.2, 10.1, 7.1 Hz, 1H), 5.09 – 5.06 (m, 1H), 4.98 – 4.92 (m, 2H), 2.88 (s, 1H), 2.67 – 2.56 (m, 2H, overlapping diastereotopic allyl-CH₂) ppm; **¹³C NMR** (101 MHz, CDCl₃): δ 167.4 (C=N), 147.3 (C), 140.2 (C), 137.0 (C), 135.7 (C), 132.5 (CH), 129.9 (CH), 129.0 (CH), 128.7 (CH), 128.3 (CH), 128.1 (CH), 128.0 (CH), 127.99 (CH), 127.7 (CH), 126.3 (CH), 119.9 (CH₂), 116.6 (CH₂), 81.4 (CH), 81.0 (CH), 63.5 (CH), 43.6 (CH₂) ppm; **HRMS** (ESI) *m/z* calculated for C₂₅H₂₂N [M+H]⁺: 336.1747; found 336.1755.



1,1-Diphenyl-*N*-[1-(pyridin-2-yl)but-3-en-1-yl]methanimine (4u**) [KG-0701]:**

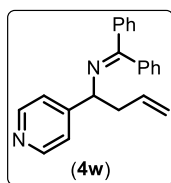
The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), 2-pyridinecarboxaldehyde (**1u**, 54 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 18 hours at room temperature. 1,1-Diphenyl-*N*-[1-(pyridin-2-yl)but-3-en-1-yl]methanimine (**4u**) was obtained in 94% (147 mg) yield after aqueous work-up. The spectroscopic data are in accordance with the literature.³

Brown liquid; **¹H NMR** (400 MHz, CDCl₃): δ 8.51 (d, *J* = 4.3 Hz, 1H), 7.70 (d, *J* = 7.3 Hz, 2H), 7.63 (t, *J* = 7.4 Hz, 1H), 7.52 (d, *J* = 7.8 Hz, 1H), 7.40 – 7.30 (m, 6H), 7.15 – 7.09 (m, 1H), 7.07 (d, *J* = 3.7 Hz, 2H), 5.74 – 5.64 (m, 1H), 5.00 – 4.93 (m, 2H), 4.69 (dd, *J* = 7.6, 5.1 Hz, 1H), 2.80 – 2.63 (m, 2H, overlapping diastereotopic allyl-CH₂) ppm; **¹³C NMR** (101 MHz, CDCl₃): δ 168.1 (C=N), 163.4 (C), 148.9 (CH), 140.0 (C), 136.8 (C), 136.4 (CH), 135.5 (CH), 130.0 (CH), 128.7 (CH), 128.4 (CH), 128.3 (CH), 128.0 (CH), 127.9 (CH), 121.9 (CH), 121.8 (CH), 116.9 (CH₂), 67.8 (CH), 42.6 (CH₂) ppm; **HRMS** (ESI) *m/z* calculated for C₂₂H₂₁N₂ [M+H]⁺: 313.1699; found 313.1710.



1,1-Diphenyl-*N*-[1-(pyridin-3-yl)but-3-en-1-yl]methanimine (4v) [KG-0753]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), 3-pyridinecarboxaldehyde (**1v**, 54 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 18 hours at room temperature. 1,1-Diphenyl-*N*-[1-(pyridin-3-yl)but-3-en-1-yl]methanimine (**4v**) was obtained in 80% (125 mg) yield after aqueous work-up. The spectroscopic data are in accordance with the literature.^{3,6}

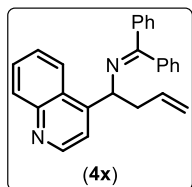
Pale yellow solid; **mp**: 80 – 82 °C; **¹H NMR** (400 MHz, CDCl₃): δ 8.47 (d, *J* = 4.1 Hz, 1H), 8.42 (s, 1H), 7.74 (d, *J* = 7.9 Hz, 1H), 7.66 (d, *J* = 7.1 Hz, 2H), 7.44 – 7.42 (m, 3H), 7.40 – 7.31 (m, 3H), 7.25 – 7.22 (m, 1H), 7.04 (dd, *J* = 6.3, 2.7 Hz, 2H), 5.69 – 5.58 (m, 1H), 4.99 – 4.96 (m, 2H), 4.48 – 4.45 (m, 1H), 2.68 (dt, *J* = 14.6, 7.4 Hz, 1H, diastereotopic allyl-CH₂), 2.60 – 2.54 (m, 1H, diastereotopic allyl-CH₂) ppm; **¹³C NMR** (101 MHz, CDCl₃): δ 167.7 (C=N), 148.9 (CH), 148.3 (CH), 139.8 (C), 139.7 (C), 136.8 (C), 134.9 (CH), 134.8 (CH), 130.1 (CH), 128.6 (CH), 128.5 (CH), 128.0 (CH), 127.7 (CH), 123.4 (CH), 117.4 (CH₂), 64.1 (CH), 43.6 (CH₂) ppm; **HRMS** (ESI) *m/z* calculated for C₂₂H₂₁N₂ [M+H]⁺ : 313.1699; found 313.1684.



1,1-Diphenyl-*N*-[1-(pyridin-4-yl)but-3-en-1-yl]methanimine (4w) [KG-0732]:

The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), 4-pyridinecarboxaldehyde (**1w**, 54 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 18 hours at room temperature. 1,1-Diphenyl-*N*-[1-(pyridin-4-yl)but-3-en-1-yl]methanimine (**4w**) was obtained in 99% (155 mg) yield after aqueous work-up. The spectroscopic data are in accordance with the literature.³

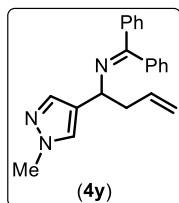
Brown solid; **mp**: 93 – 95 °C; **¹H NMR** (400 MHz, CDCl₃): δ 8.51 (d, *J* = 5.5 Hz, 2H), 7.68 – 7.66 (m, 2H), 7.43 – 7.32 (m, 6H), 7.24 (d, *J* = 5.5 Hz, 2H), 7.03 (dd, *J* = 6.4, 2.8 Hz, 2H), 5.67 – 5.58 (m, 1H), 4.99 – 4.95 (m, 2H), 4.41 (dd, *J* = 7.4, 5.7 Hz, 1H), 2.65 (dt, *J* = 14.7, 7.5 Hz, 1H, diastereotopic allyl-CH₂), 2.58 – 2.52 (m, 1H, diastereotopic allyl-CH₂) ppm; **¹³C NMR** (101 MHz, CDCl₃): δ 168.1 (C=N), 153.2 (C), 149.7 (CH), 139.6 (C), 136.7 (C), 134.7 (CH), 130.3 (CH), 128.6 (CH), 128.55 (CH), 128.49 (CH), 128.1 (CH), 127.7 (CH), 122.4 (CH), 117.5 (CH₂), 65.4 (CH), 43.4 (CH₂) ppm; **HRMS** (ESI) *m/z* calculated for C₂₂H₂₁N₂ [M+H]⁺ : 313.1699; found 313.1712.



1,1-Diphenyl-*N*-[1-(quinolin-4-yl)but-3-en-1-yl]methanimine (4x) [KG-0733]:

The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), 4-quinolinecarboxaldehyde (**1x**, 79 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 18 hours at room temperature. 1,1-Diphenyl-*N*-[1-(quinolin-4-yl)but-3-en-1-yl]methanimine (**4x**) was obtained in 92% (167 mg) yield after aqueous work-up. The spectroscopic data are in accordance with the literature.³

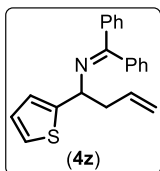
Colourless liquid; ¹H NMR (400 MHz, CDCl₃): δ 8.85 (d, *J* = 4.5 Hz, 1H), 8.11 (d, *J* = 8.4 Hz, 1H), 7.82 (d, *J* = 8.5 Hz, 1H), 7.73 – 7.70 (m, 3H), 7.68 – 7.64 (m, 1H), 7.45 – 7.32 (m, 7H), 6.96 (d, *J* = 7.0 Hz, 2H), 5.71 (ddt, *J* = 17.2, 10.1, 7.1 Hz, 1H), 5.21 (dd, *J* = 7.9, 4.8 Hz, 1H), 5.00 – 4.95 (m, 2H), 2.85 – 2.77 (m, 1H, diastereotopic allyl-CH₂), 2.73 – 2.66 (m, 1H, diastereotopic allyl-CH₂) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 168.3 (C=N), 150.5 (C), 150.3 (CH), 148.4 (C), 139.6 (C), 136.7 (CH), 135.0 (CH), 130.3 (CH), 130.2 (CH), 128.9 (CH), 128.7 (CH), 128.6 (CH), 128.5 (CH), 128.1 (CH), 127.8 (CH), 126.2 (CH), 125.9 (C), 123.4 (CH), 119.8 (CH), 117.3 (CH₂), 62.1 (CH), 43.3 (CH₂) ppm; HRMS (ESI) *m/z* calculated for C₂₆H₂₃N₂ [M+H]⁺ : 363.1856; found 363.1859.



***N*-[1-(1-methyl-1*H*-pyrazol-4-yl)but-3-en-1-yl]-1,1-diphenylmethanimine (4y) [KG-0734]:**

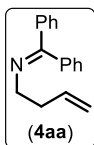
The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), 1-methyl-1*H*-pyrazole-4-carboxaldehyde (**1y**, 79 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 6 hours at room temperature. *N*-[1-(1-methyl-1*H*-pyrazol-4-yl)but-3-en-1-yl]-1,1-diphenylmethanimine (**4y**) was obtained in 95% (150 mg) yield. The spectroscopic data are in accordance with the literature.³

White solid; *mp*: 78 – 81 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.63 (dd, *J* = 8.2, 1.4 Hz, 2H), 7.44 – 7.41 (m, 3H), 7.38 – 7.25 (m, 5H), 7.11 (dd, *J* = 6.4, 3.1 Hz, 2H), 5.69 (ddt, *J* = 17.2, 10.2, 7.1 Hz, 1H), 5.03 – 4.97 (m, 2H), 4.45 (dd, *J* = 7.6, 5.5 Hz, 1H), 3.85 (s, 3H), 2.65 – 2.51 (m, 2H, overlapping diastereotopic allyl-CH₂) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 166.9 (C=N), 140.0 (C), 137.4 (CH), 137.0 (C), 135.6 (CH), 134.5 (CH), 129.9 (CH), 128.6 (CH), 128.4 (CH), 128.37 (CH), 128.0 (CH), 127.8 (CH), 124.9 (C), 116.9 (CH₂), 58.2 (CH), 43.2 (CH₂), 38.9 (CH₃) ppm; HRMS (ESI) *m/z* calculated for C₂₁H₂₂N₃ [M+H]⁺ : 316.1808; found 316.1804.



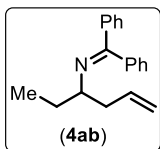
1,1-Diphenyl-*N*-[1-(thiophen-2-yl)but-3-en-1-yl]methanimine (4z) [KG-0756]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), thiophenecarboxaldehyde (**1z**, 56 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 18 hours at room temperature. 1,1-Diphenyl-*N*-[1-(thiophen-2-yl)but-3-en-1-yl]methanimine (**4z**) was obtained in 94% (149 mg) yield. New compound according to a Sci-finder search.

Colourless liquid; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.67 (dd, $J = 5.2, 3.2$ Hz, 2H), 7.44 – 7.41 (m, 3H), 7.39 – 7.29 (m, 3H), 7.18 (dd, $J = 5.1, 1.1$ Hz, 1H), 7.13 (dd, $J = 6.4, 3.1$ Hz, 2H), 6.92 (dd, $J = 5.0, 3.5$ Hz, 1H), 6.78 (d, $J = 3.4$ Hz, 1H), 5.68 (ddt, $J = 17.3, 10.2, 7.1$ Hz, 1H), 5.04 – 4.97 (m, 2H), 4.73 (dd, $J = 7.3, 5.9$ Hz, 1H), 2.77 – 2.59 (m, 2H, overlapping diastereotopic allyl- CH_2) ppm; $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ 167.4 (C=N), 148.0 (C), 139.7 (C), 136.6 (C), 135.1 (CH), 130.1 (CH), 128.7 (CH), 128.44 (CH), 128.40 (CH), 128.0 (CH), 127.8 (CH), 126.2 (CH), 123.8 (CH), 122.7 (CH), 117.2 (CH_2), 62.3 (CH), 44.2 (CH_2) ppm; **HRMS** (ESI) m/z calculated for $\text{C}_{21}\text{H}_{20}\text{NS}$ $[\text{M}+\text{H}]^+$: 318.1311; found 318.1317.



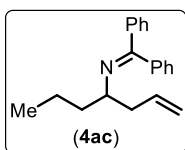
***N*-(but-3-en-1-yl)-1,1-diphenylmethanimine (4aa)** [KG-0727]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), paraformaldehyde (**1aa**, 18 mg, 0.6 mmol, 1.2 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 6 hours at room temperature. *N*-(but-3-en-1-yl)-1,1-diphenylmethanimine (**4aa**) was obtained in 99% (116 mg) yield. The spectroscopic data are in accordance with the literature.³

Colourless liquid; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.62 – 7.57 (m, 2H), 7.46 – 7.28 (m, 6H), 7.15 (dd, $J = 7.8, 1.5$ Hz, 2H), 5.82 (ddt, $J = 17.0, 10.2, 6.8$ Hz, 1H), 5.07 – 4.96 (m, 2H), 3.44 (t, $J = 7.2$ Hz, 2H), 2.44 (q, $J = 7.0$ Hz, 2H) ppm; $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ 168.2 (C=N), 140.0 (C), 137.0 (C), 136.8 (CH), 129.8 (CH), 128.4 (CH), 128.3 (CH), 128.29 (CH), 128.0 (CH), 127.8 (CH), 115.7 (CH_2), 53.4 (CH_2), 35.6 (CH_2) ppm; **HRMS** (ESI) m/z calculated for $\text{C}_{17}\text{H}_{18}\text{N}$ $[\text{M}+\text{H}]^+$: 236.1434; found 236.1432.



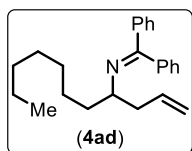
***N*-(hex-5-en-3-yl)-1,1-diphenylmethanimine (4ab)** [KG-0735]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), propionaldehyde (**1ab**, 35 mg, 0.6 mmol, 1.2 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 2 hours at room temperature. *N*-(hex-5-en-3-yl)-1,1-diphenylmethanimine (**4ab**) was obtained in 99% (130 mg) yield. The spectroscopic data are in accordance with the literature.³

Brown liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.60 (dd, *J* = 8.0, 1.5 Hz, 2H), 7.44 – 7.28 (m, 6H), 7.14 (dd, *J* = 7.7, 1.5 Hz, 2H), 5.70 (ddt, *J* = 17.3, 10.1, 7.3 Hz, 1H), 5.02 – 4.95 (m, 2H), 3.28 (q, *J* = 6.3 Hz, 1H), 2.31 (t, *J* = 6.7 Hz, 2H), 1.64 – 1.57 (m, 2H), 0.80 (t, *J* = 7.4 Hz, 3H) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 166.7 (C=N), 140.4 (C), 137.7 (C), 136.3 (CH), 130.1 (CH), 129.6 (CH), 128.4 (CH), 128.2 (CH), 128.1 (CH), 128.0 (CH), 116.3 (CH₂), 63.2 (CH), 41.0 (CH₂), 29.0 (CH₂), 11.0 (CH₃) ppm; HRMS (ESI) *m/z* calculated for C₁₉H₂₂N [M+H]⁺ : 264.1747; found 264.1741.



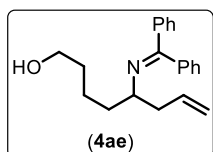
***N*-(hept-1-en-4-yl)-1,1-diphenylmethanimine (4ac)** [KG-0722]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), butyraldehyde (**1ac**, 36 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 2 hours at room temperature. *N*-(hept-1-en-4-yl)-1,1-diphenylmethanimine (**4ac**) was obtained in 99% (137 mg) yield. The spectroscopic data are in accordance with the literature.³

Colourless liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.59 (dd, *J* = 8.0, 1.5 Hz, 2H), 7.43 – 7.28 (m, 6H), 7.13 (dd, *J* = 7.8, 1.5 Hz, 2H), 5.70 (ddt, *J* = 17.3, 10.1, 7.2 Hz, 1H), 5.02 – 4.96 (m, 2H), 3.39 – 3.33 (m, 1H), 2.31 (t, *J* = 6.4 Hz, 2H), 1.65 – 1.47 (m, 2H), 1.36 – 1.25 (m, 1H), 1.23 – 1.07 (m, 1H), 0.81 (t, *J* = 7.3 Hz, 3H) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 166.5 (C=N), 140.4 (C), 137.6 (C), 136.3 (CH), 130.1 (CH), 129.6 (CH), 128.4 (CH), 128.2 (CH), 128.1 (CH), 128.0 (CH), 116.3 (CH₂), 61.7 (CH), 41.4 (CH₂), 38.5 (CH₂), 19.8 (CH₂), 14.2 (CH₃) ppm; HRMS (ESI) *m/z* calculated for C₂₀H₂₄N [M+H]⁺ : 278.1903; found 278.1900.



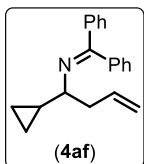
1,1-Diphenyl-*N*-(undec-1-en-4-yl)methanimine (4ad) [KG-0736]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), *n*-octylaldehyde (**1ad**, 64 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 6 hours at room temperature. 1,1-Diphenyl-*N*-(undec-1-en-4-yl)methanimine (**4ad**) was obtained in 99% (165 mg) yield. New compound according to a Sci-finder search.

Colourless liquid; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.61 – 7.58 (m, 2H), 7.44 – 7.36 (m, 3H), 7.35 – 7.26 (m, 3H), 7.13 (dd, J = 7.8, 1.6 Hz, 2H), 5.70 (ddt, J = 17.3, 10.1, 7.2 Hz, 1H), 5.02 – 4.95 (m, 2H), 3.38 – 3.31 (m, 1H), 2.31 (t, J = 6.6 Hz, 1H), 1.63 – 1.48 (m, 2H), 1.34 – 1.09 (m, 10H), 0.86 (t, J = 7.0 Hz, 3H) ppm; $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ 166.5 (C=N), 140.4 (C), 137.6 (C), 136.3 (CH), 129.6 (CH), 128.5 (CH), 128.4 (CH), 128.2 (CH), 128.1 (CH), 128.0 (CH), 116.3 (CH_2), 61.9 (CH), 41.3 (CH_2), 36.2 (CH_2), 31.9 (CH_2), 29.7 (CH_2), 29.3 (CH_2), 26.5 (CH_2), 22.7 (CH_2), 14.1 (CH_3) ppm; **HRMS** (ESI) m/z calculated for $\text{C}_{24}\text{H}_{32}\text{N}$ $[\text{M}+\text{H}]^+$: 334.2529; found 334.2523.



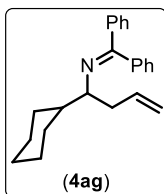
5-[(diphenylmethylidene)amino]oct-7-en-1-ol (4ae) [KG-0731]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), 5-hydroxypentanal (**1ae**, 51 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 2 hours at room temperature. 5-((diphenylmethylene)amino)oct-7-en-1-ol (**4ae**) was obtained in 97% (149 mg) yield. The spectroscopic data are in accordance with the literature.³

Colourless liquid; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.59 – 7.56 (m, 2H), 7.44 – 7.28 (m, 6H), 7.15 – 7.12 (m, 2H), 5.68 (ddt, J = 17.3, 10.1, 7.2 Hz, 1H), 5.02 – 4.96 (m, 2H), 3.56 (t, J = 6.6 Hz, 2H), 3.37 (dtd, J = 8.1, 6.3, 4.7 Hz, 1H), 2.30 (t, J = 6.8 Hz, 2H), 2.19 (br s, 1H), 1.69 – 1.52 (m, 2H), 1.50 – 1.43 (m, 2H), 1.38 – 1.29 (m, 1H), 1.27 – 1.16 (m, 1H) ppm; $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ 167.1 (C=N), 140.2 (C), 137.5 (C), 136.0 (CH), 129.8 (CH), 128.4 (CH), 128.3 (CH), 128.1 (CH), 128.0 (CH), 127.9 (CH), 116.5 (CH_2), 62.6 (CH_2), 61.8 (CH), 41.2 (CH_2), 35.7 (CH_2), 32.8 (CH_2), 22.6 (CH_2) ppm; **HRMS** (ESI) m/z calculated for $\text{C}_{21}\text{H}_{26}\text{NO}$ $[\text{M}+\text{H}]^+$: 308.2009; found 308.2003.



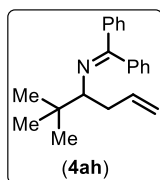
***N*-(1-cyclopropylbut-3-en-1-yl)-1,1-diphenylmethanimine (4af)** [KG-0730]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), cyclopropanecarboxaldehyde (**1af**, 42 mg, 0.6 mmol, 1.2 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 6 hours at room temperature. *N*-(1-cyclopropylbut-3-en-1-yl)-1,1-diphenylmethanimine (**4af**) was obtained in 96% (132 mg) yield. The spectroscopic data are in accordance with the literature.³

Colourless liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.60 (dd, *J* = 8.1, 1.5 Hz, 2H), 7.42 – 7.28 (m, 6H), 7.12 (dd, *J* = 7.7, 1.6 Hz, 2H), 5.75 (ddt, *J* = 17.3, 10.1, 7.3 Hz, 1H), 5.04 – 4.95 (m, 2H), 2.78 (dd, *J* = 13.6, 6.8 Hz, 1H), 2.50 – 2.40 (m, 2H), 1.18 – 1.09 (m, 1H), 0.46 – 0.34 (m, 2H), 0.15 – 0.03 (m, 2H) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 166.2 (C=N), 140.3 (C), 137.4 (C), 136.3 (CH), 129.7 (CH), 128.5 (CH), 128.2 (CH), 128.1 (CH), 128.0 (CH), 127.9 (CH), 116.2 (CH₂), 65.9 (CH), 41.6 (CH₂), 17.0 (CH), 2.7 (CH₂), 2.6 (CH₂) ppm; HRMS (ESI) *m/z* calculated for C₂₀H₂₄N [M+H]⁺ : 278.1903; found 278.1901.



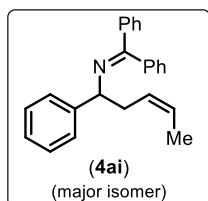
***N*-(1-cyclohexylbut-3-en-1-yl)-1,1-diphenylmethanimine (4ag)** [KG-0724]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), cyclohexanecarboxaldehyde (**1ag**, 56 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 6 hours at room temperature. *N*-(1-cyclohexylbut-3-en-1-yl)-1,1-diphenylmethanimine (**4ag**) was obtained in 98% (156 mg) yield. The spectroscopic data are in accordance with the literature.³

Colourless liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.59 (dd, *J* = 8.0, 1.6 Hz, 2H), 7.43 – 7.27 (m, 6H), 7.14 – 7.12 (m, 2H), 5.71 (ddt, *J* = 17.3, 10.1, 7.3 Hz, 1H), 5.02 – 4.95 (m, 2H), 3.18 – 3.13 (m, 1H), 2.41 – 2.29 (m, 2H), 1.79 – 1.53 (m, 5H), 1.26 – 1.01 (m, 5H), 0.95 – 0.85 (m, 1H) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 166.2 (C=N), 140.5 (C), 137.6 (C), 136.7 (CH), 129.5 (CH), 128.4 (CH), 128.3 (CH), 128.1 (CH), 128.0 (CH), 127.9 (CH), 116.2 (CH₂), 66.5 (CH), 42.9 (CH), 38.3 (CH₂), 30.2 (CH₂), 29.3 (CH₂), 26.7 (CH₂), 26.6 (CH₂) ppm; HRMS (ESI) *m/z* calculated for C₂₃H₂₈N [M+H]⁺ : 318.2216; found 318.2224.



***N*-(2,2-dimethylhex-5-en-3-yl)-1,1-diphenylmethanimine (4ah)** [KG-0723]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), pivalaldehyde (**1ah**, 43 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 2 hours at room temperature. *N*-(2,2-dimethylhex-5-en-3-yl)-1,1-diphenylmethanimine (**4ah**) was obtained in 99% (144 mg) yield. The spectroscopic data are in accordance with the literature.³

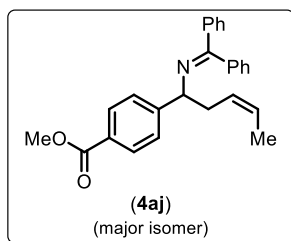
Colourless liquid; **¹H NMR** (400 MHz, CDCl₃): δ 7.60 (dd, *J* = 8.0, 1.6 Hz, 2H), 7.40 – 7.27 (m, 6H), 7.16 (dd, *J* = 7.6, 1.8 Hz, 2H), 5.62 (ddt, *J* = 17.3, 10.1, 7.3 Hz, 1H), 4.99 – 4.93 (m, 2H), 3.12 – 3.05 (m, 1H), 2.40 – 2.37 (m, 2H), 0.91 (s, 9H) ppm; **¹³C NMR** (101 MHz, CDCl₃): δ 165.6 (C=N), 140.7 (C), 138.1 (CH), 137.4 (C), 129.4 (CH), 128.8 (CH), 128.4 (CH), 127.9 (CH), 127.85 (CH), 127.81 (CH), 116.0 (CH₂), 69.9 (CH), 36.1 (CH₂), 35.5 (C), 27.0 (CH₃) ppm; **HRMS** (ESI) *m/z* calculated for C₂₁H₂₆N [M+H]⁺ : 292.2060; found 292.2050.



(*Z*)-1,1-diphenyl-*N*-(1-phenylpent-3-en-1-yl)methanimine (4ai) [KG-0741]:

The general procedure was applied using 2-methyl-1,1-diphenylbut-3-en-1-amine (**2b**, 119 mg, 0.5 mmol, 1.0 equiv.), benzaldehyde (**1a**, 54 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 6 hours at room temperature. (*Z*)-1,1-diphenyl-*N*-(1-phenylpent-3-en-1-yl)methanimine (**4ai**) was obtained in 99% (161 mg) yield. The spectroscopic data are in accordance with the literature.³

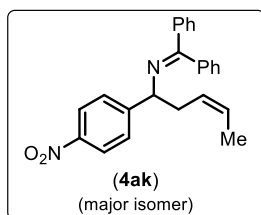
Colourless liquid; **¹H NMR** (400 MHz, CDCl₃): δ 7.67 (dd, *J* = 8.0, 1.5 Hz, 2H), 7.42 – 7.40 (m, 3H), 7.37 – 7.18 (m, 8H), 7.08 – 7.04 (m, 2H), 5.48 – 5.33 (m, 1H), 5.27 – 5.21 (m, 1H), 4.39 (dd, *J* = 7.6, 5.9 Hz, 1H), 2.71 – 2.47 (m, 2H, overlapping diastereotopic allyl-CH₂) 1.50 (d, *J* = 6.6 Hz, 3H) ppm; **¹³C NMR** (101 MHz, CDCl₃): δ 166.4 (C=N), 144.7 (C), 140.1 (C), 137.2 (C), 132.4 (CH), 130.1 (CH), 129.8 (CH), 128.6 (CH), 128.3 (CH), 128.2 (CH), 128.1 (CH), 128.0 (CH), 127.9 (CH), 127.2 (CH), 127.1 (CH), 126.7 (CH), 125.7 (CH), 66.6 (CH), 36.9 (CH₂), 12.9 (CH₃) ppm; **HRMS** (ESI) *m/z* calculated for C₂₄H₂₄N [M+H]⁺ : 326.1903; found 326.1902.



Methyl-(Z)-4-(1-((diphenylmethylene)amino)pent-3-en-1-yl)benzoate (4aj) [KG-0747]: The general procedure was applied using 2-methyl-1,1-diphenylbut-3-en-1-amine (**2b**, 119 mg, 0.5 mmol, 1.0 equiv.), methyl 4-formylbenzoate (**1r**, 82 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 6

hours at room temperature. Methyl-(Z)-4-(1-((diphenylmethylene)amino)pent-3-en-1-yl)benzoate (**4aj**) was obtained in 99% (190 mg) yield. The spectroscopic data are in accordance with the literature.³

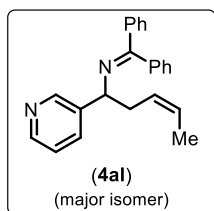
Colourless liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.96 (d, *J* = 8.3 Hz, 2H), 7.68 – 7.66 (m, 2H), 7.42 – 7.30 (m, 8H), 7.05 – 7.01 (m, 2H), 5.48 – 5.34 (m, 1H), 5.28 – 5.19 (m, 1H), 4.43 (dd, *J* = 11.1, 6.4 Hz, 1H), 3.89 (s, 3H), 2.70 – 2.41 (m, 2H, overlapping diastereotopic allyl-CH₂), 1.47 (d, *J* = 6.8 Hz, 3H) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 167.2 (C=N), 150.0 (C), 139.8 (C), 136.9 (C), 130.0 (CH), 129.6 (CH), 128.6 (CH), 128.58 (C), 128.38 (CH), 128.36 (CH), 128.0 (CH), 127.8 (CH), 127.76 (C), 127.2 (CH), 126.5 (CH), 126.2 (CH), 66.4 (CH), 52.0 (CH₃), 36.8 (CH₂), 12.9 (CH₃) ppm; HRMS (ESI) *m/z* calculated for C₂₆H₂₆NO₂ [M+H]⁺: 384.1958; found 384.1954.



(Z)-N-(1-(4-nitrophenyl)pent-3-en-1-yl)-1,1-diphenylmethanimine (4ak) [KG-0766]: The general procedure was applied using 2-methyl-1,1-diphenylbut-3-en-1-amine (**2b**, 119 mg, 0.5 mmol, 1.0 equiv.), *p*-nitrobenzaldehyde (**1n**, 76 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 6

hours at room temperature. (Z)-N-(1-(4-nitrophenyl)pent-3-en-1-yl)-1,1-diphenylmethanimine (**4ak**) was obtained in 96% (178 mg) yield. New compound according to a Sci-finder search.

Colourless liquid; ¹H NMR (400 MHz, CDCl₃): δ 8.15 (d, *J* = 8.7 Hz, 2H), 7.68 – 7.66 (m, 2H), 7.51 – 7.48 (m, 2H), 7.44 – 7.42 (m, 3H), 7.41 – 7.32 (m, 3H), 7.05 – 7.01 (m, 2H), 5.51 – 5.32 (m, 1H), 5.25 – 5.18 (m, 1H), 4.49 (dd, *J* = 15.1, 8.3 Hz, 1H), 2.68 – 2.45 (m, 2H, overlapping diastereotopic allyl-CH₂), 1.46 (dd, *J* = 6.8, 0.8 Hz, 3H) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 167.8 (C=N), 152.2 (C), 146.9 (C), 139.5 (C), 136.7 (C), 130.3 (CH), 128.6 (CH), 128.55 (CH), 128.52 (CH), 128.1 (CH), 128.0 (CH), 127.6 (CH), 126.7 (CH), 125.9 (CH), 123.5 (CH), 66.0 (CH), 36.9 (CH₂), 12.9 (CH₃) ppm; HRMS (ESI) *m/z* calculated for C₂₄H₂₃N₂O₂ [M+H]⁺: 371.1754; found 371.1741.

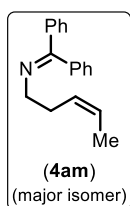


(Z)-1,1-diphenyl-N-(1-(pyridin-3-yl)pent-3-en-1-yl)methanimine (4al) [KG-

0767]: The general procedure was applied using 2-methyl-1,1-diphenylbut-3-en-1-amine (**2b**, 119 mg, 0.5 mmol, 1.0 equiv.), 3-pyridinecarboxaldehyde (**1v**, 54 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The

reaction mixture was vigorously stirred for 18 hours at room temperature. (Z)-1,1-diphenyl-N-(1-(pyridin-3-yl)pent-3-en-1-yl)methanimine (**4al**) was obtained in 92% (150 mg) yield. New compound according to a Sci-finder search.

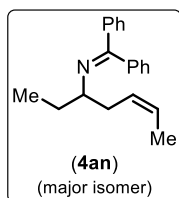
Brown liquid; ¹H NMR (400 MHz, CDCl₃): δ 8.47 – 8.42 (m, 2H), 7.78 – 7.76 (m, 1H), 7.67 – 7.65 (m, 2H), 7.45 – 7.43 (m, 3H), 7.38 – 7.32 (m, 3H), 7.25 – 7.21 (m, 1H), 7.07 – 7.05 (m, 2H), 5.50 – 5.38 (m, 1H), 5.26 – 5.19 (m, 1H), 4.45 – 4.39 (m, 1H), 2.69 – 2.45 (m, 2H, overlapping diastereotopic allyl-CH₂), 1.48 – 1.46 (m, 3H) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 167.4 (C=N), 149.0 (CH), 148.2 (CH), 139.9 (C), 139.7 (C), 136.8 (C), 134.9 (CH), 130.1 (CH), 128.6 (CH), 128.5 (CH), 128.46 (CH), 128.0 (CH), 127.7 (CH), 126.5 (CH), 126.2 (CH), 123.4 (CH), 64.2 (CH), 36.7 (CH₂), 12.9 (CH₃) ppm; HRMS (ESI) *m/z* calculated for C₂₃H₂₃N₂ [M+H]⁺ : 327.1856; found 327.1861.



(Z)-N-(pent-3-en-1-yl)-1,1-diphenylmethanimine (4am) [KG-0748]: The general procedure was applied using 2-methyl-1,1-diphenylbut-3-en-1-amine (**2b**, 119 mg, 0.5 mmol, 1.0 equiv), paraformaldehyde (**1aa**, 18 mg, 0.6 mmol, 1.2 equiv), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 2

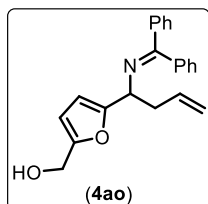
hours at room temperature. (Z)-N-(pent-3-en-1-yl)-1,1-diphenylmethanimine (**4am**) was obtained in 98% (122 mg) yield. The spectroscopic data are in accordance with the literature.³

Brown liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.60 (dd, *J* = 8.1, 1.4 Hz, 2H), 7.46 – 7.40 (m, 3H), 7.36 – 7.29 (m, 3H), 7.16 (dd, *J* = 7.8, 1.5 Hz, 2H), 5.52 – 5.44 (m, 1H), 5.43 – 5.35 (m, 1H), 3.41 (t, *J* = 7.3 Hz, 2H), 2.43 (q, *J* = 7.2 Hz, 2H), 1.59 (dd, *J* = 6.6, 0.8 Hz, 3H) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 168.1 (C=N), 140.0 (C), 137.0 (C), 129.8 (CH), 128.4 (CH), 128.3 (CH), 128.2 (CH), 128.16 (CH), 128.0 (CH), 127.9 (CH), 125.2 (CH), 53.7 (CH₂), 28.9 (CH₂), 12.9 (CH₃) ppm; HRMS (ESI) *m/z* calculated for C₁₈H₂₀N [M+H]⁺ : 250.1590; found 250.1600.



(*E*)-*N*-(pent-3-en-1-yl)-1,1-diphenylmethanimine (4an) [KG-0749]: The general procedure was applied using 2-methyl-1,1-diphenylbut-3-en-1-amine (**2b**, 119 mg, 0.5 mmol, 1.0 equiv.), propionaldehyde (**1ab**, 35 mg, 0.6 mmol, 1.2 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The reaction mixture was vigorously stirred for 6 hours at room temperature. (*Z*)-*N*-(pent-3-en-1-yl)-1,1-diphenylmethanimine was obtained in 96% (133 mg) yield. The spectroscopic data are in accordance with the literature.³

Brown liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.60 (dd, *J* = 8.0, 1.5 Hz, 2H), 7.44 – 7.36 (m, 3H), 7.34 – 7.28 (m, 3H), 7.14 (dd, *J* = 7.8, 1.6 Hz, 2H), 5.51 – 5.37 (m, 1H), 5.35 – 5.27 (m, 1H), 3.29 – 3.18 (m, 1H), 2.30 (t, *J* = 6.5 Hz, 2H), 1.65 – 1.55 (m, 5H), 0.83 – 0.78 (m, 3H) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 166.5 (C=N), 140.4 (C), 137.7 (C), 129.6 (CH), 128.4 (CH), 128.2 (CH), 128.1 (CH), 128.0 (CH), 127.9 (CH), 127.7 (CH), 125.3 (CH), 63.5 (CH), 33.9 (CH₂), 29.0 (CH₂), 13.0 (CH₃), 11.1 (CH₃) ppm; HRMS (ESI) *m/z* calculated for C₂₀H₂₄N [M+H]⁺ : 278.1903; found 278.1894.

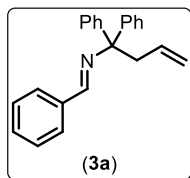


(5-{1-[(Diphenylmethylidene)amino]but-3-en-1-yl}furan-2-yl)methanol (4ao) [KG-0721]: The general procedure was applied using 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), 5-hydroxymethylfurfural (**1ao**, 63 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and HFIP (0.5 mL). The

reaction mixture was vigorously stirred for 6 hours at room temperature. (5-{1-[(Diphenylmethylidene)amino]but-3-en-1-yl}furan-2-yl)methanol (**4ao**) was obtained in 88% (133 mg) yield. New compound according to a Sci-finder search.

Pale yellow liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.67 (dd, *J* = 5.2, 3.2 Hz, 2H), 7.44 – 7.41 (m, 3H), 7.39 – 7.29 (m, 3H), 7.18 (dd, *J* = 5.1, 1.1 Hz, 2H), 6.92 (dd, *J* = 5.0, 3.5 Hz, 1H), 6.78 (d, *J* = 3.4 Hz, 1H), 5.68 (ddt, *J* = 17.3, 10.2, 7.1 Hz, 1H), 5.04 – 4.97 (m, 2H), 4.73 (dd, *J* = 7.3, 5.9 Hz, 1H), 4.52 (s, 2H), 2.77 – 2.59 (m, 2H, overlapping diastereotopic allyl-CH₂), 2.05 (br s, 1H) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 169.0 (C=N), 156.2 (C), 153.1 (C), 139.9 (C), 136.7 (C), 135.0 (CH), 130.1 (CH), 128.8 (CH), 128.5 (CH), 128.4 (CH), 128.03 (CH), 128.02 (CH), 117.2 (CH₂), 108.4 (CH), 106.5 (CH), 60.4 (CH), 57.6 (CH₂), 39.8 (CH₂) ppm; HRMS (ESI) *m/z* calculated for C₂₂H₂₂NO₂ [M+H]⁺ : 332.1645; found 332.1646.

3.3 Synthesis of aldimine 3a



(*E*)-*N*-(1,1-diphenylbut-3-en-1-yl)-1-phenylmethanimine (3a) [KG-0768]: In an oven-dried 10 mL vial was charged with a Teflon-coated magnetic stir bar, 1,1-diphenylbut-3-en-1-amine (**2a**, 112 mg, 0.5 mmol, 1.0 equiv.), benzaldehyde (**1a**, 53 mg, 0.5 mmol, 1.0 equiv.), 4Å molecular sieves (200 mg) and 2,2,2-trifluoroethanol

(TFE) (0.5 mL). The vial was capped under air. The reaction mixture was then stirred for 12 h at room temperature. After the reaction time, the reaction mixture was filtered (to remove 4Å molecular sieves) and the solvent was removed under reduced pressure to afford pure (*E*)-*N*-(1,1-diphenylbut-3-en-1-yl)-1-phenylmethanimine (**3a**) in 99% (154 mg) yield.

Colourless liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.82 (s, 1H), 7.77 (dd, *J* = 6.6, 2.9 Hz, 2H), 7.39 – 7.34 (m, 7H), 7.29 (t, *J* = 7.6 Hz, 4H), 7.21 (t, *J* = 7.1 Hz, 2H), 5.80 (ddt, *J* = 17.2, 10.5, 6.8 Hz, 1H), 4.96 – 4.91 (m, 2H), 3.13 (d, *J* = 6.8 Hz, 2H) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 159.8 (CH), 146.4 (C), 137.0 (C), 134.6 (CH), 130.6 (CH), 128.6 (CH), 128.5 (CH), 128.4 (CH), 128.0 (CH), 126.6 (CH), 117.4 (CH₂), 72.1 (C), 46.8 (CH₂) ppm; HRMS (ESI) *m/z* calculated for C₂₃H₂₂N [M+H]⁺ : 312.1747; found 312.1760.

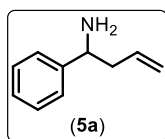
3.4 Gram-scale synthesis of 4a

In an oven-dried 25 mL vial, 1,1-diphenylbut-3-en-1-amine (**2a**) (1.117 g, 5 mmol, 1.0 equiv.), benzaldehyde (**1a**) (0.531 g, 5 mmol, 1.0 equiv.), 4Å molecular sieves (2 g), and HFIP (5 mL) were added successively and the vial was capped under air. The reaction mixture was then vigorously stirred for 12 h at room temperature. After the reaction time, the reaction mixture was filtered and HFIP was distilled off under reduced pressure at room temperature with a Kugelrohr distillation apparatus to obtain pure 1,1-diphenyl-*N*-(1-phenylbut-3-en-1-yl) methanimine (**4a**) in 91% (1.42 mg) yield and HFIP (4.1 mL) was recovered.

3.5 Hydrolysis of the ketimine and recovery of benzophenone

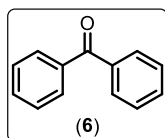
This general procedure was adapted from a literature procedure.⁴ 1,1-diphenyl-*N*-(1-phenylbut-3-en-1-yl)methanimine (**4a**, 1.417 g, 4.55 mmol) was dissolved in 2-MeTHF (20 mL), followed by the addition of 2N aqueous HCl solution (10.0 mL). The mixture was stirred at room temperature for 5 h and monitored by TLC. Upon completion, 2-MeTHF was removed by rotary evaporation and another 10 mL of H₂O was added. The mixture was washed with EtOAc (4 x 5 mL) and the combined organic phase was extracted with H₂O (5 mL) which was then washed with EtOAc (1.0 mL). The EtOAc fractions were combined, dried using MgSO₄ and concentrated under vacuum affording the pure benzophenone (0.746 g, 90 %). The combined aqueous phase was neutralized with 3N aqueous NaOH

and extracted with ethyl acetate, the organic fractions were combined, dried using MgSO_4 and concentrated under vacuum affording the pure 1-phenylbut-3-en-1-amine (0.596 g, 89 %).



1-phenylbut-3-en-1-amine (5a) [KG-0743-h]: The general procedure 3.3 was applied using 1,1-diphenyl-*N*-(1-phenylbut-3-en-1-yl)methanimine (**4a**, 1.417 g, 4.55 mmol) after aqueous work-up, 1-phenylbut-3-en-1-amine was obtained in 89% (0.596 g) yield.

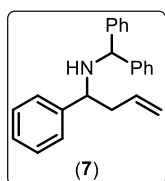
Pale yellow liquid; ^1H NMR (400 MHz, CDCl_3): δ 7.35 – 7.30 (m, 4H), 7.27 – 7.22 (m, 1H), 5.81 – 5.68 (m, 1H), 5.14 – 5.05 (m, 2H), 3.99 (dd, J = 8.0, 5.4 Hz, 1H), 2.51 – 2.40 (m, 1H, diastereotopic allyl- CH_2), 2.41 – 2.32 (m, 1H, diastereotopic allyl- CH_2), 1.88 (br s, 2H) ppm; ^{13}C NMR (101 MHz, CDCl_3): δ 145.8 (C), 135.4 (CH), 128.4 (CH), 127.0 (CH), 126.3 (CH), 117.6 (CH_2), 55.4 (CH), 44.2 (CH_2) ppm. The spectroscopic data for **5a** is in agreement with literature.⁸



Benzophenone (6) [KG-0743-h]: The general procedure 3.3 was applied using 1,1-diphenyl-*N*-(1-phenylbut-3-en-1-yl)methanimine (**4a**, 1.417 g, 4.55 mmol) after aqueous work-up, benzophenone was obtained in 90% (0.746 g) yield.

White solid; *mp*: 48–50 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.80 – 7.78 (m, 4H), 7.57 (t, J = 7.4 Hz, 2H), 7.46 (t, J = 7.7 Hz, 4H) ppm; ^{13}C NMR (101 MHz, CDCl_3): δ 196.7 (C=O), 137.6 (C), 132.4 (CH), 130.0 (CH), 128.3 (CH) ppm. HRMS (ESI) m/z calculated for $\text{C}_{13}\text{H}_{11}\text{O}$ $[\text{M}+\text{H}]^+$: 183.0804; found 183.0800. The spectroscopic data for **6** is in agreement with literature.⁹

3.6 Synthesis of *N*-benzhydryl-protected homoallylamine **7**



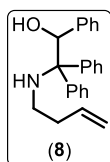
***N*-benzhydryl-1-phenylbut-3-en-1-amine (7)** [KG-0779]: This experimental procedure was adapted from a literature procedure.¹ In an oven-dried 10 mL vial, 1,1-diphenylbut-3-en-1-amine (**2a**) (0.5 mmol, 1.0 equiv.), benzaldehyde (**1a**) (0.5 mmol, 1.0 equiv.), 4 Å molecular sieves (200 mg) and HFIP (1.0 mL) were added successively

and the vial was capped under air. The reaction mixture was then stirred for 6 hours at room temperature. After 6 hours, sodium cyanoborohydride (126 mg, 4 equiv.) was added to the reaction mixture. Then, the reaction vial was flushed with argon in 5 minutes and then the vial was sealed with a septum. The reaction vial was stirred at room temperature under argon for 6 hours. After the reaction time, the reaction mixture was filtered and the solvent was removed under reduced pressure. Then the reaction mixture was diluted with dichloromethane and washed with 1N aqueous NaOH solution. The aqueous layer was extracted with dichloromethane. Combined dichloromethane layers were dried and

concentrated in vacuo. The crude residue was purified by an automated flash chromatography system on silica gel using *n*-heptane/EtOAc gradient (from 100% *n*-heptane to 10% EtOAc in 25 minutes, 25 mL/min.). *N*-benzhydryl-1-phenylbut-3-en-1-amine (**7**) was obtained in 95% (149 mg) yield. New compound according to a Sci-finder search.

Colorless liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.34 – 7.29 (m, 4H), 7.27 – 7.20 (m, 10H), 7.17 – 7.13 (m, 1H), 5.69 (ddt, *J* = 17.2, 10.1, 7.1 Hz, 1H), 5.06 – 5.00 (m, 2H), 4.61 (s, 1H), 3.59 – 3.56 (m, 1H), 2.48 – 2.36 (m, 2H), 2.06 (br s, 1H) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 144.7 (C), 143.9 (C), 143.4 (C), 135.6 (CH), 128.4 (CH), 128.39 (CH), 128.2 (CH), 127.8 (CH), 127.4 (CH), 127.3 (CH), 127.0 (CH), 126.9 (CH), 126.7 (CH), 117.3 (CH₂), 63.3 (CH), 59.3 (C), 43.0 (CH₂) ppm. HRMS (ESI) *m/z* calculated for C₂₃H₂₄N [M+H]⁺ : 314.1903; found 314.1907.

3.7 Synthesis of α-amino alcohol **8**

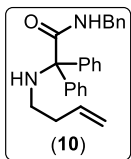


2-(But-3-en-1-ylamino)-1,2,2-triphenylethan-1-ol (8**)** [KG-0769]: This experimental procedure was adapted from a literature procedure.¹⁰ In an oven-dried 4 mL Wheaton vial, *N*-(but-3-en-1-yl)-1,1-diphenylmethanimine (**4aa**) (24 mg, 0.1 mmol, 1.0 equiv.), [Ir(dtbbpy)(ppy)₂][PF₆] (0.5 mg, 0.5 mol%), methyldicyclohexylamine (39 mg, 0.2 mmol,

2 equiv.), benzaldehyde (**1a**) (13 mg, 0.120 mmol, 1.2 equiv.) and anhydrous MeCN (1 mL) were added successively. The reaction vial was flushed with argon in 5 minutes, a magnetic stirring bar was added and then the vial was sealed with a septum. The reaction mixture was placed under a 19 W blue LED light source and stirred at ambient temperature (25-30 °C) for 20 h. After 20 h, the vial was opened to air and the volatile materials were removed using a rotary evaporator under reduced pressure. The crude residue was purified by an automated flash chromatography system on silica gel using *n*-heptane/EtOAc gradient (from 100% *n*-heptane to 10% EtOAc in 25 minutes, 25 mL/min). 2-(but-3-en-1-ylamino)-1,2,2-triphenylethan-1-ol (**8**) was obtained in 68% (23 mg) yield. New compound according to a Sci-finder search.

White solid; *mp*: 59-61 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.37 (dd, *J* = 7.6, 2.1 Hz, 2H), 7.30 – 7.25 (m, 3H), 7.21 – 7.08 (m, 6H), 7.02 (t, *J* = 7.5 Hz, 2H), 6.77 (d, *J* = 7.4 Hz, 2H), 5.69 (ddt, *J* = 17.1, 10.1, 6.9 Hz, 1H), 5.58 (s, 1H), 5.08 – 4.98 (m, 2H), 3.74 (br s, 1H), 2.41 – 2.30 (m, 2H), 2.17 (q, *J* = 6.6 Hz, 2H), 1.78 (br s, 1H) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 142.2 (C), 141.7 (C), 140.2 (C), 136.4 (CH), 129.9 (CH), 129.4 (CH), 128.2 (CH), 127.4 (CH), 127.3 (CH), 127.2 (CH), 127.0 (CH), 126.9 (CH), 126.8 (CH), 116.7 (CH₂), 76.9 (CH), 70.7 (C), 42.0 (CH₂), 34.8 (CH₂) ppm. HRMS (ESI) *m/z* calculated for C₂₄H₂₆NO [M+H]⁺ : 344.2009; found 344.2014.

3.8 Synthesis of α -amino amide **10**

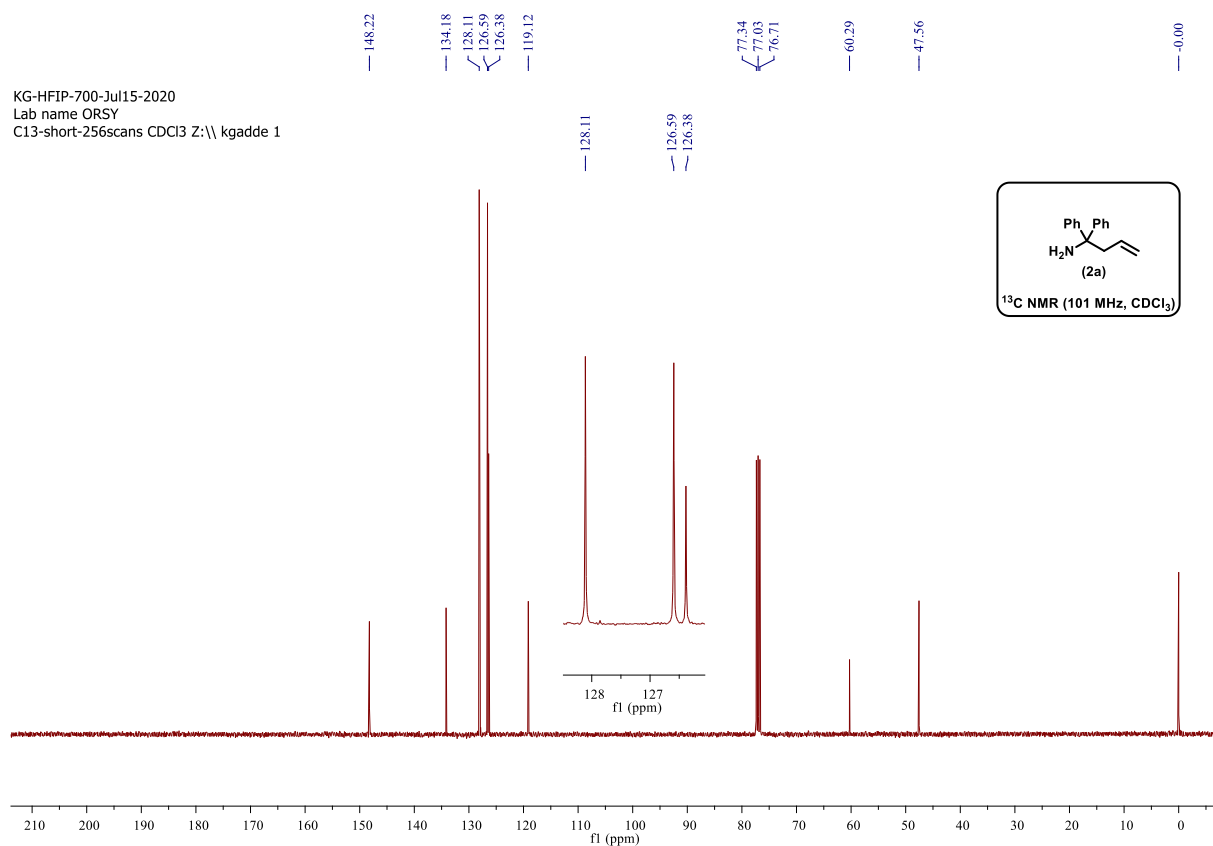
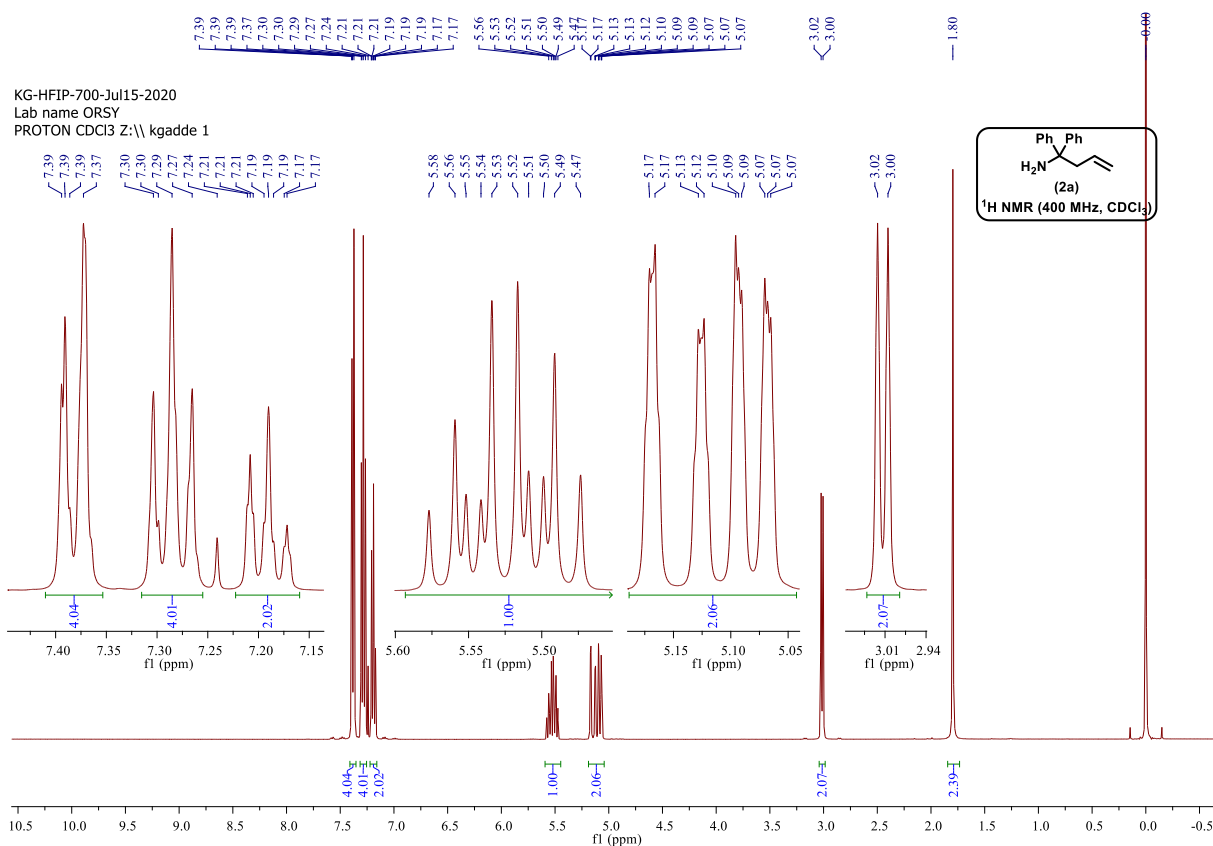


***N*-benzyl-2-(but-3-en-1-ylamino)-2,2-diphenylacetamide (**10**)** [KG-0776]: This experimental procedure was adapted from a literature procedure.¹¹ In an oven-dried 4 mL Wheaton vial, *N*-(but-3-en-1-yl)-1,1-diphenylmethanimine (**4aa**) (24 mg, 0.1 mmol, 1.0 equiv.), [Ir(dtbbpy)(ppy)₂][PF₆] (1 mg, 1.0 mol%), methyldicyclohexylamine (39 mg, 0.2 mmol, 2 equiv.), benzyl isocyanate (**9**) (27 mg, 0.2 mmol, 2 equiv.) and anhydrous MeCN (1 mL, 0.1 M) were added successively. The reaction vial was flushed with argon in 5 minutes, a magnetic stirring bar was added and then the vial was sealed with a septum. The reaction mixture was placed under a 19 W blue LED light source and stirred at ambient temperature (~30 °C) for 20 h. After 20 h, the vial was opened to air and the volatile materials were removed using a rotary evaporator under reduced pressure. The crude residue was purified by an automated flash chromatography system on silica gel using *n*-heptane/EtOAc gradient (from 100% *n*-heptane to 10% EtOAc in 25 minutes, 25 mL/min.). *N*-benzyl-2-(but-3-en-1-ylamino)-2,2-diphenylacetamide (**10**) was obtained in 54 % (20 mg) yield. Colorless liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.35 – 7.28 (m, 8H), 7.26 – 7.21 (m, 5H), 7.18 – 7.16 (m, 2H), 6.53 (s, 1H), 5.48 (ddt, *J* = 17.2, 10.2, 7.0 Hz, 1H), 4.86 – 4.73 (m, 2H), 4.71 (t, *J* = 5.3 Hz, 1H), 4.43 (d, *J* = 5.5 Hz, 2H), 3.34 – 3.30 (m, 2H), 1.84 – 1.78 (m, 2H) ppm; ¹³C NMR (101 MHz, CDCl₃): δ 157.9 (C), 139.9 (C), 139.5 (C), 135.1 (CH), 128.8 (CH), 128.6 (CH), 128.5 (CH), 127.6 (CH), 127.4 (CH), 127.1 (CH), 116.6 (CH₂), 63.3 (CH), 45.4 (CH₂), 45.0 (CH₂), 33.6 (CH₂) ppm. HRMS (ESI) *m/z* calculated for C₂₅H₂₇N₂O [M+H]⁺ : 371.2118; found 371.2131.

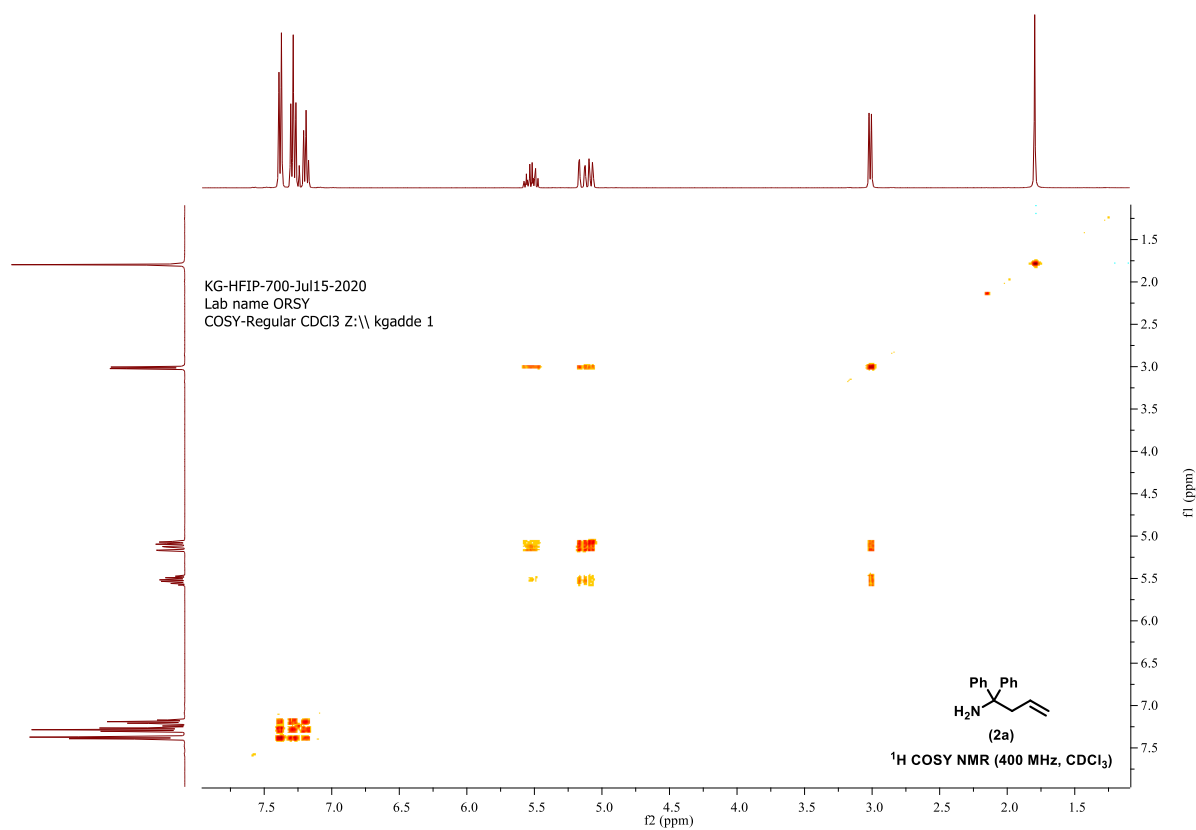
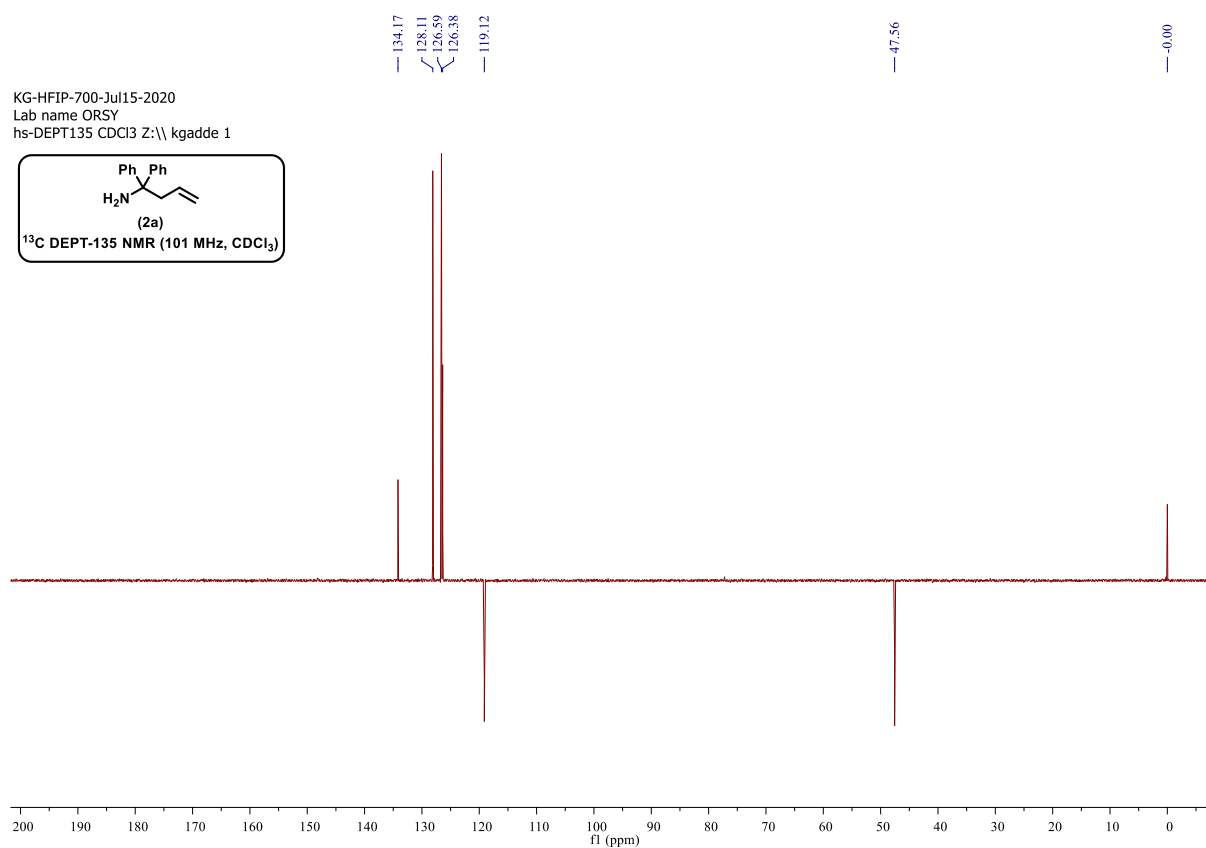
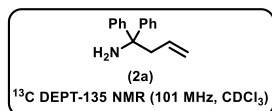
4 References

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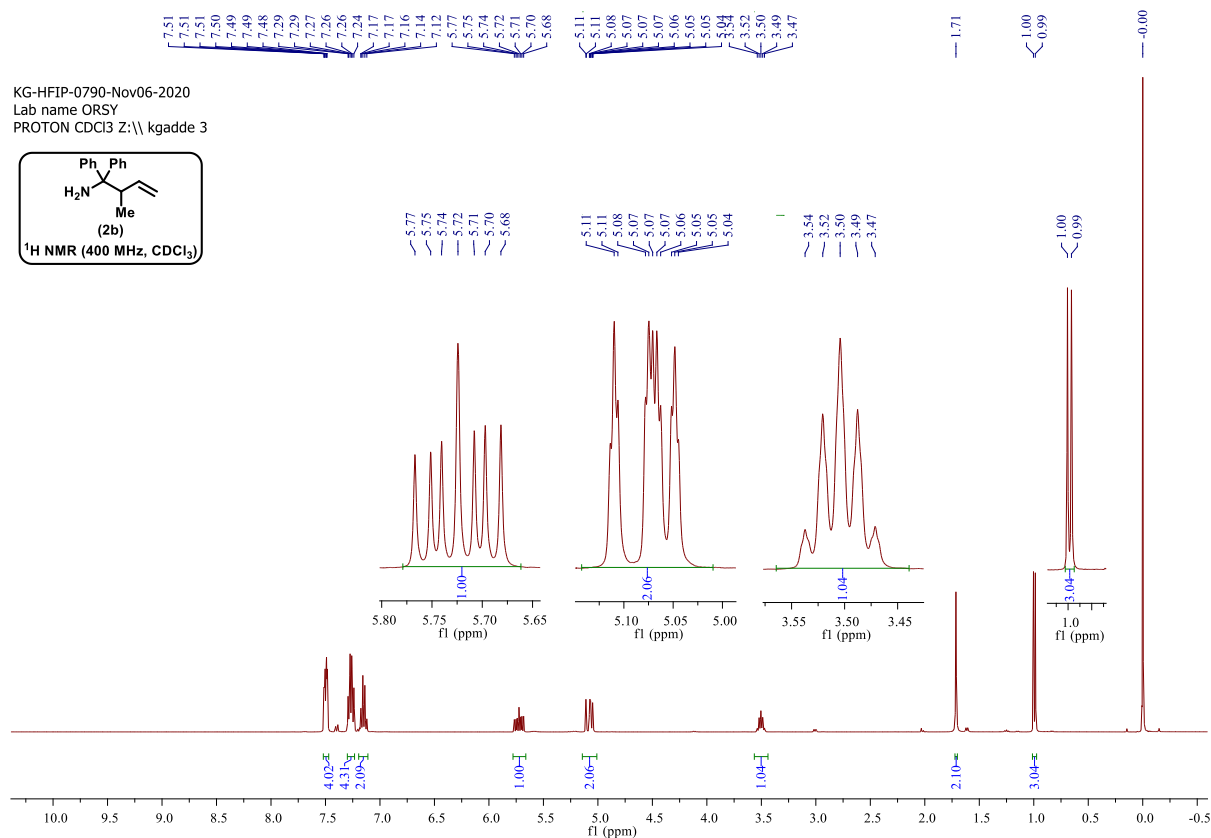
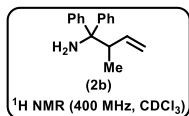
5 Copies of NMR spectra



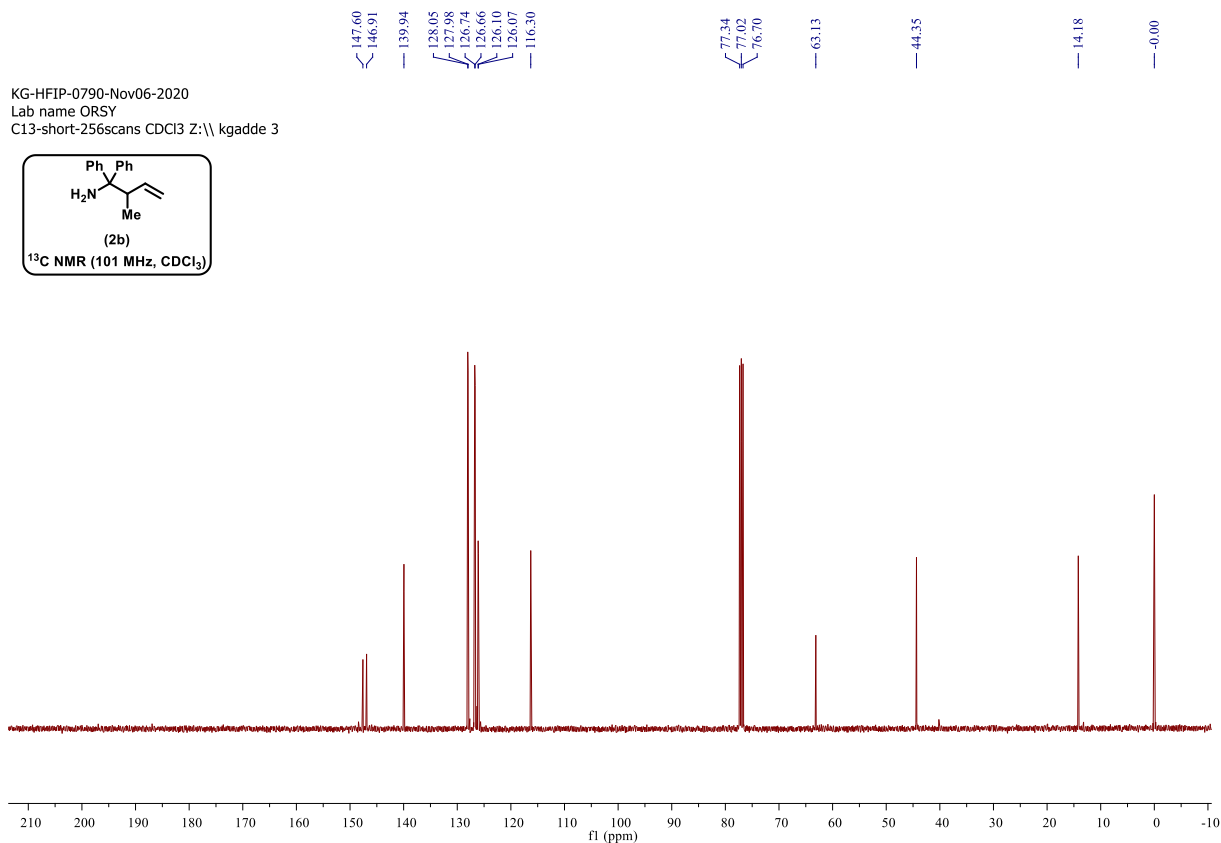
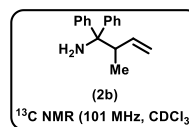
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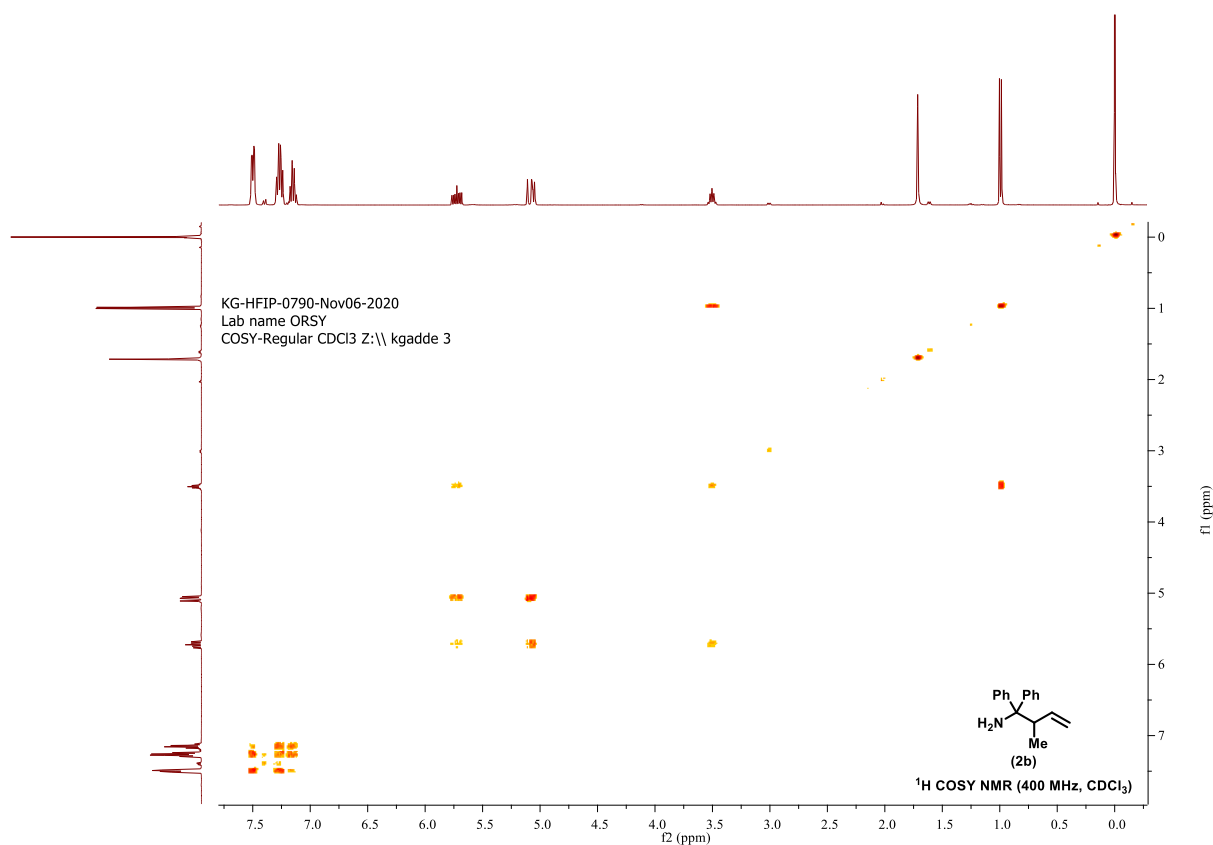
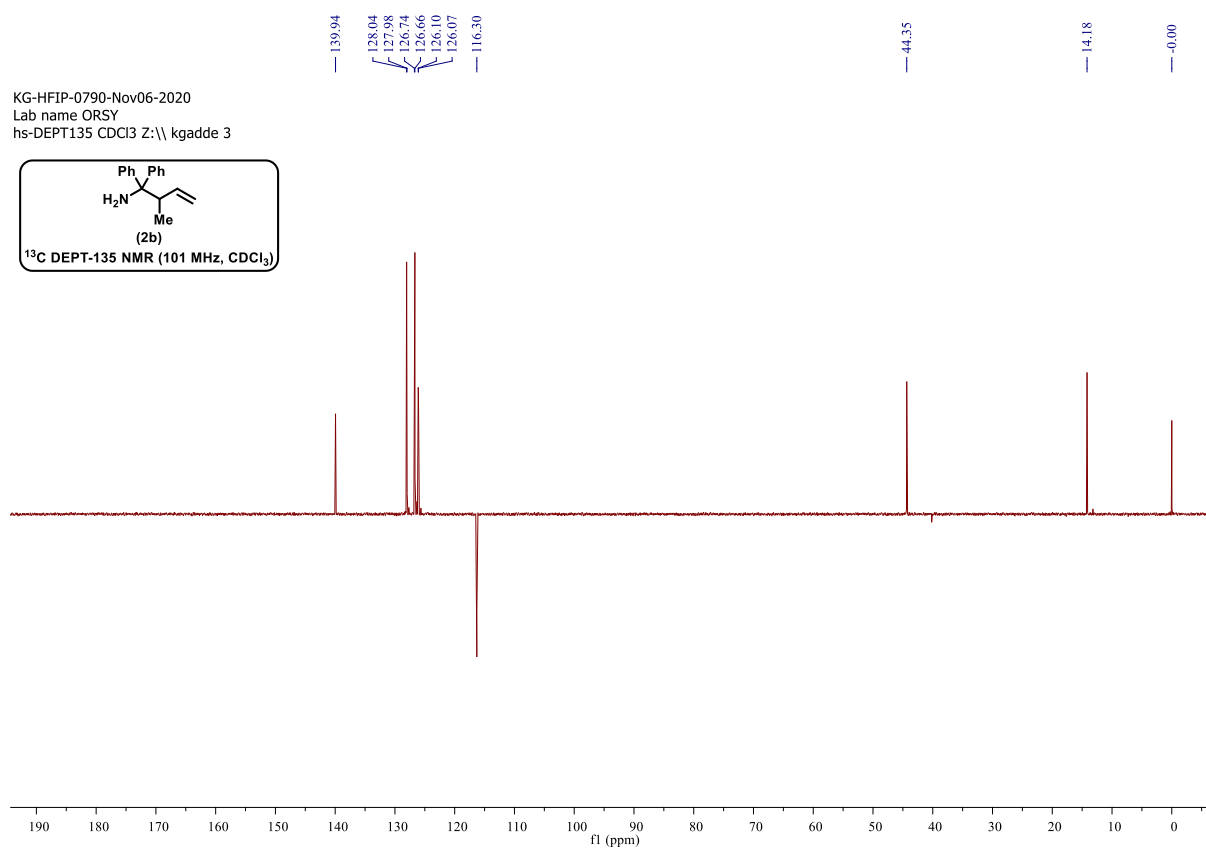
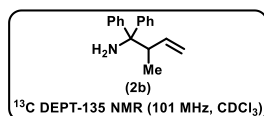
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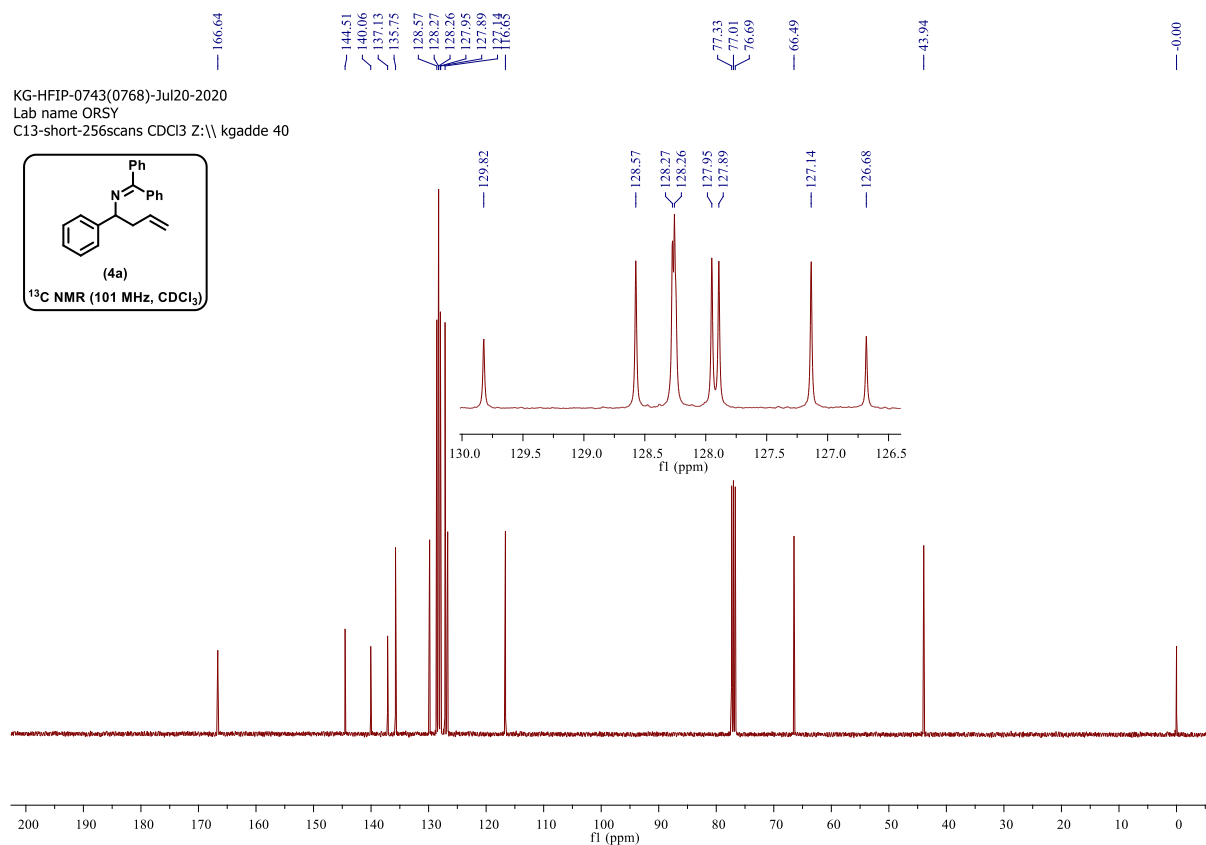
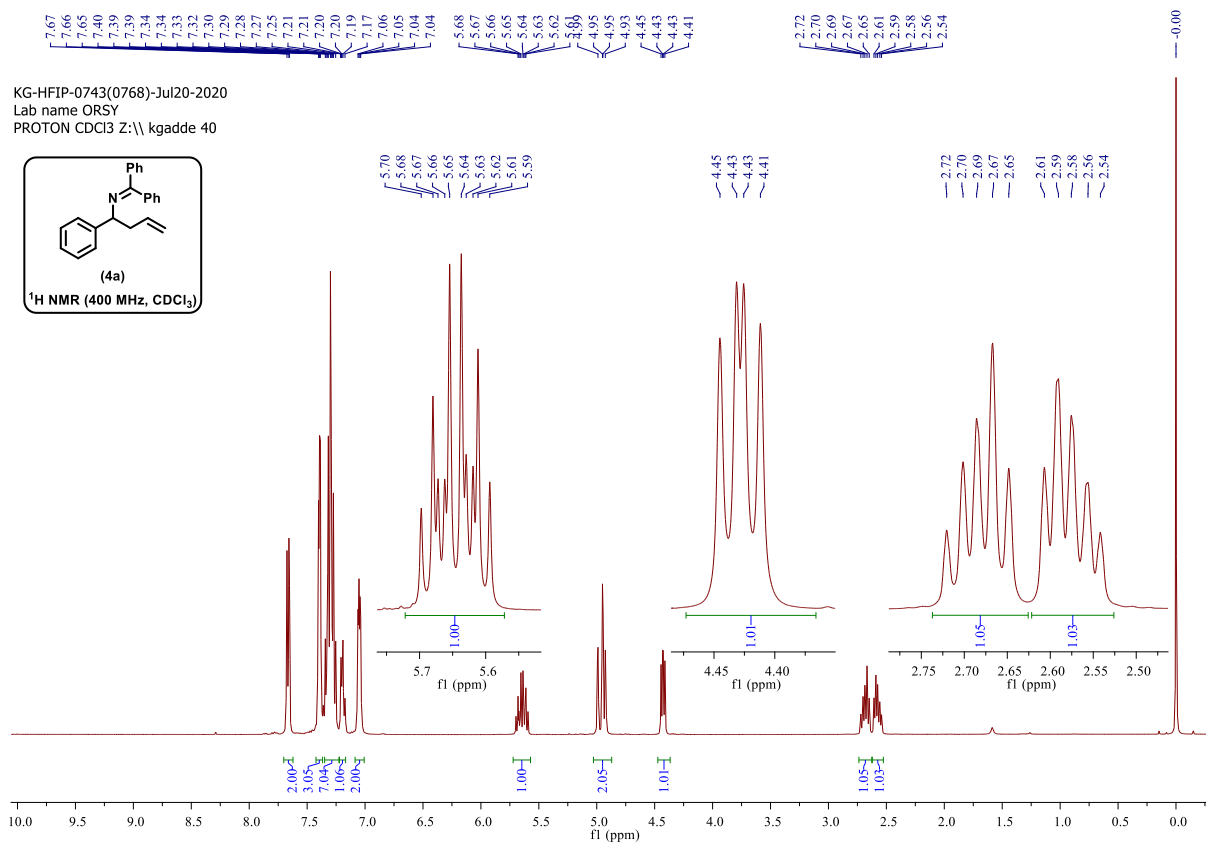


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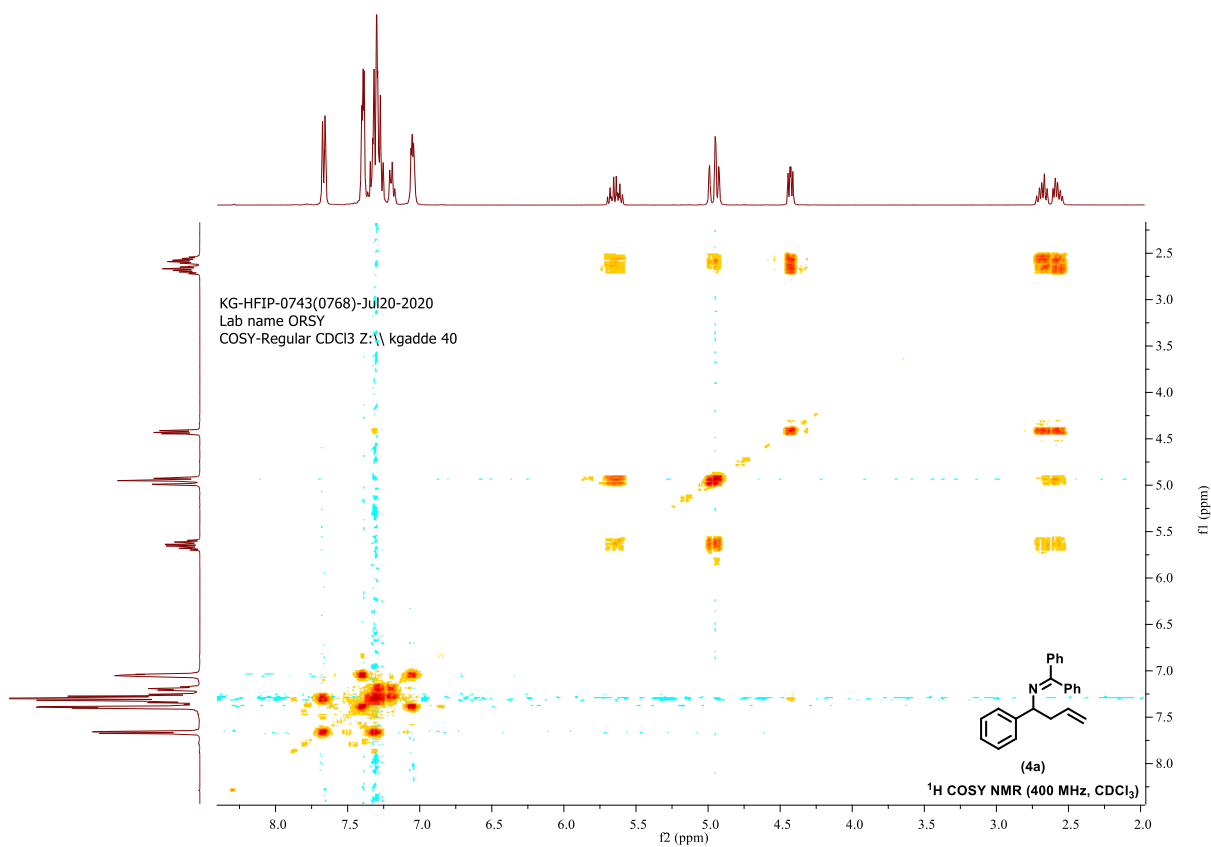
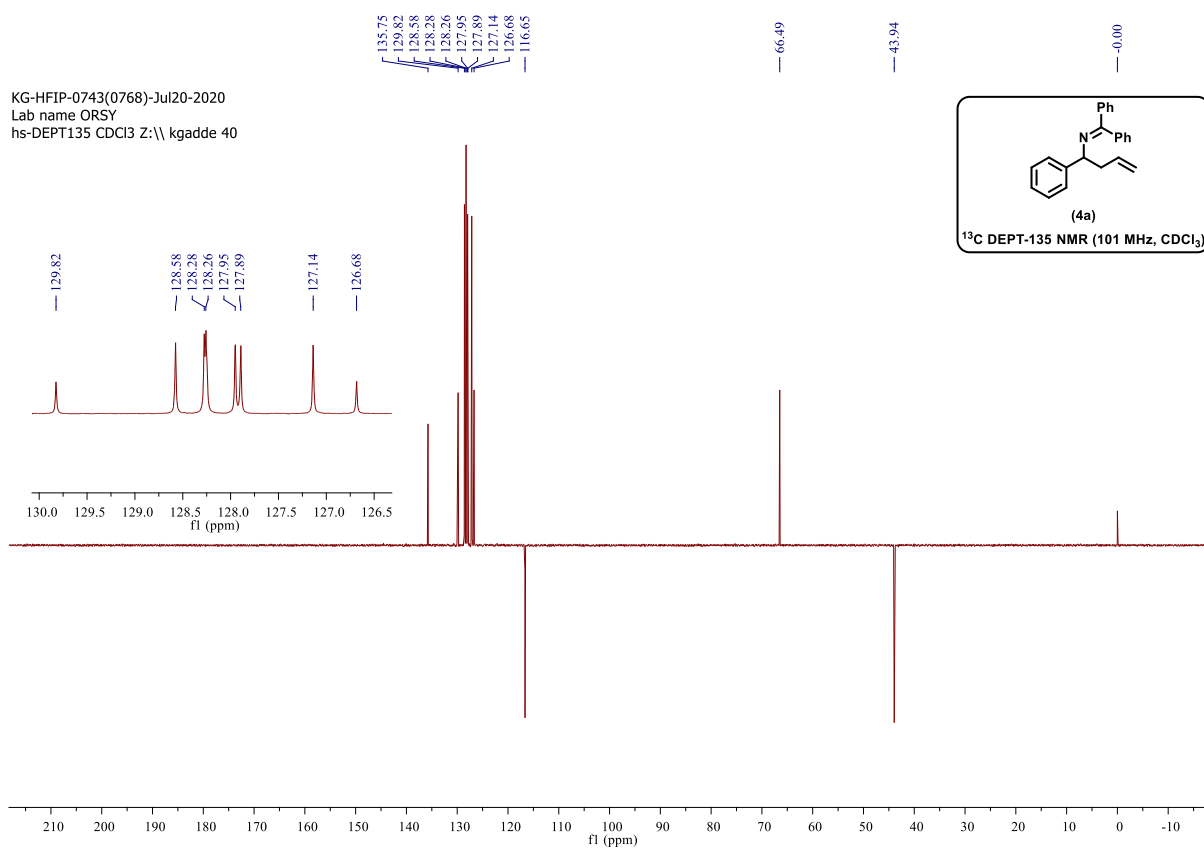


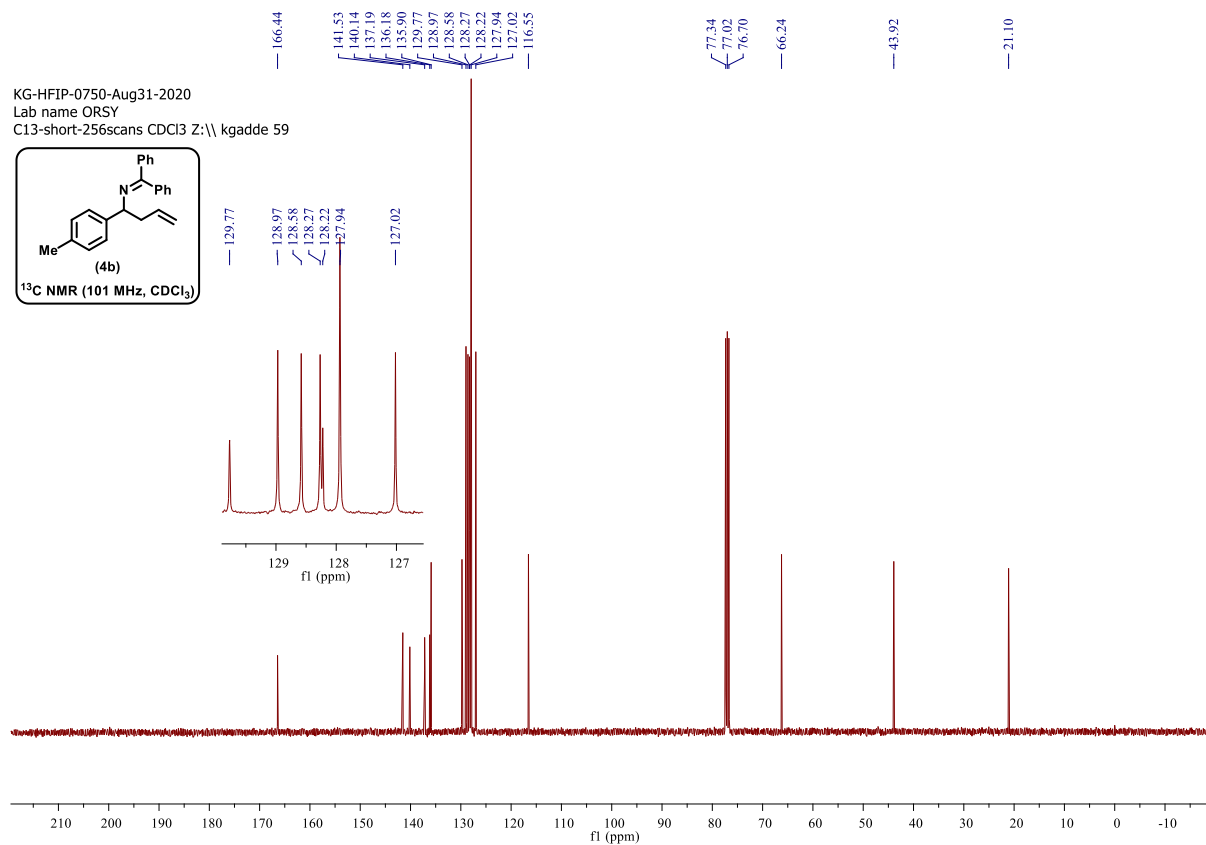
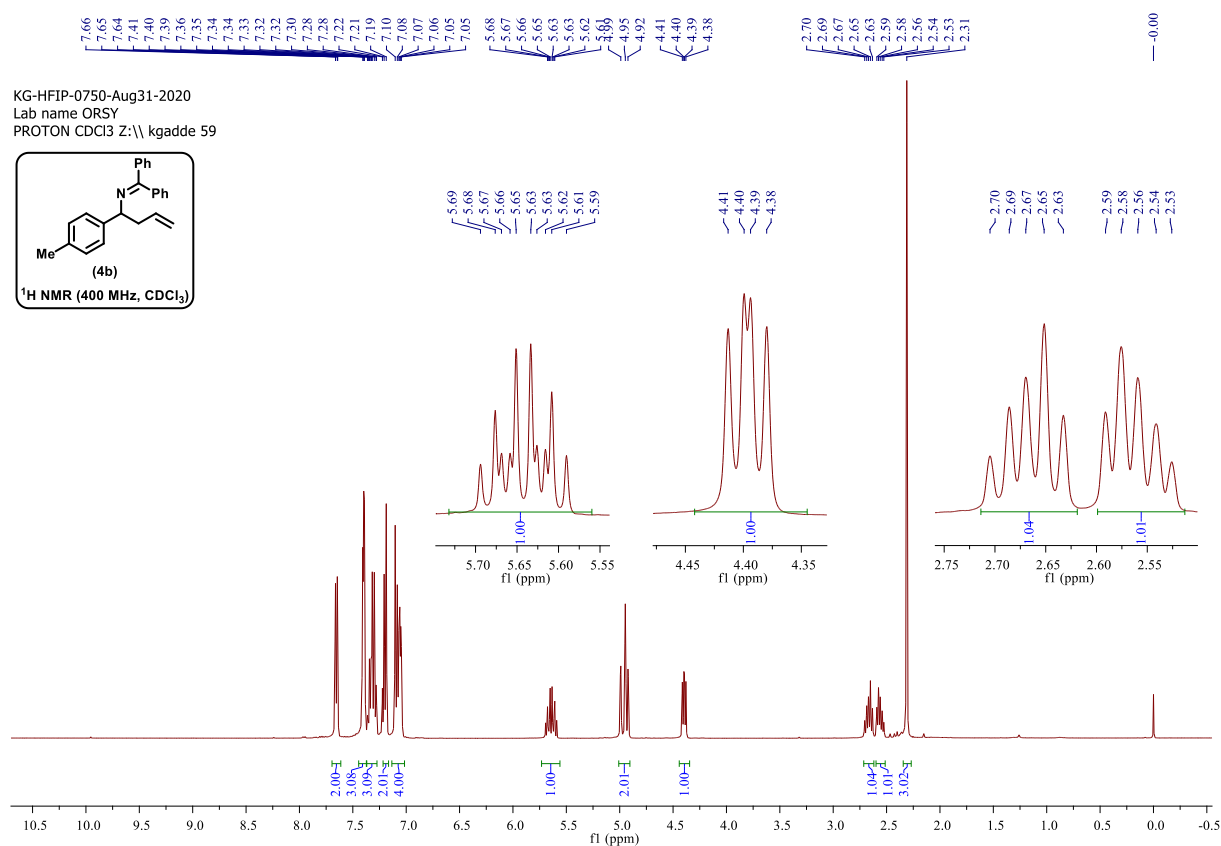
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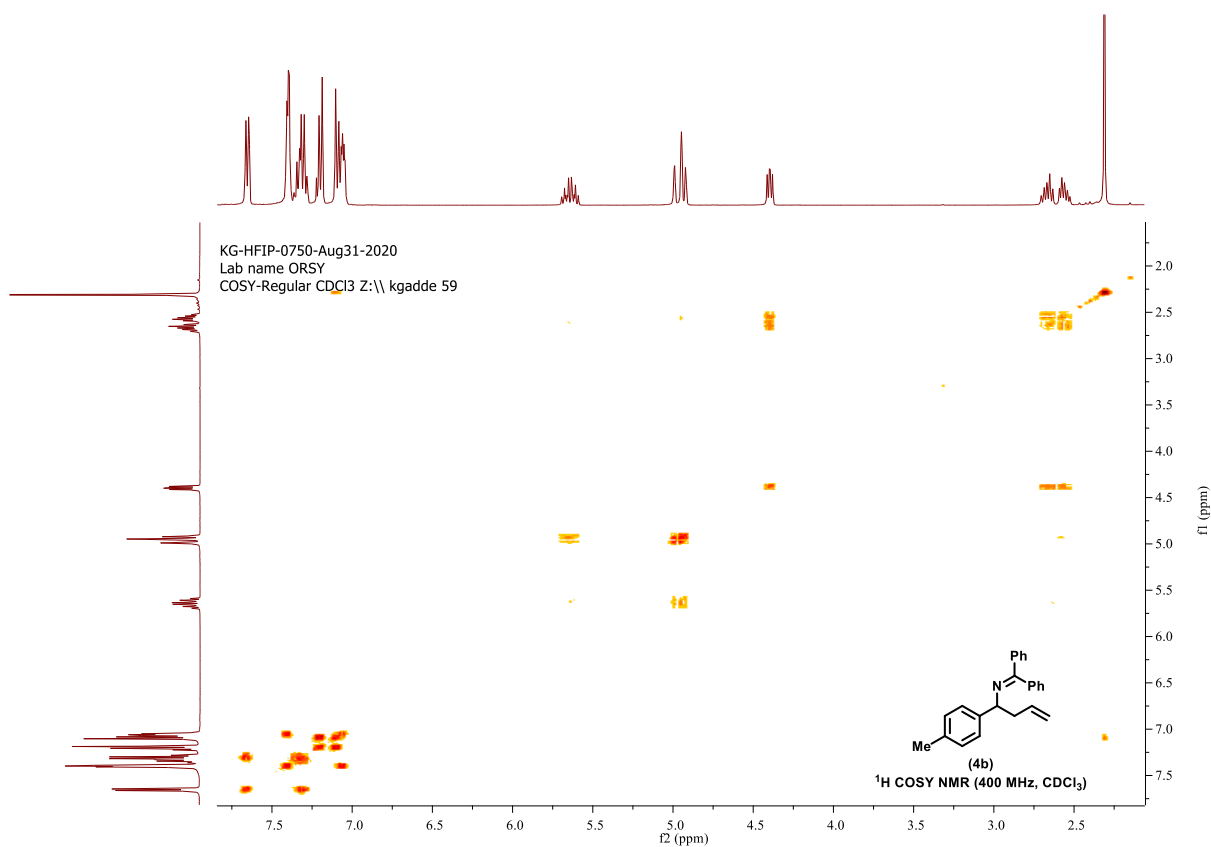
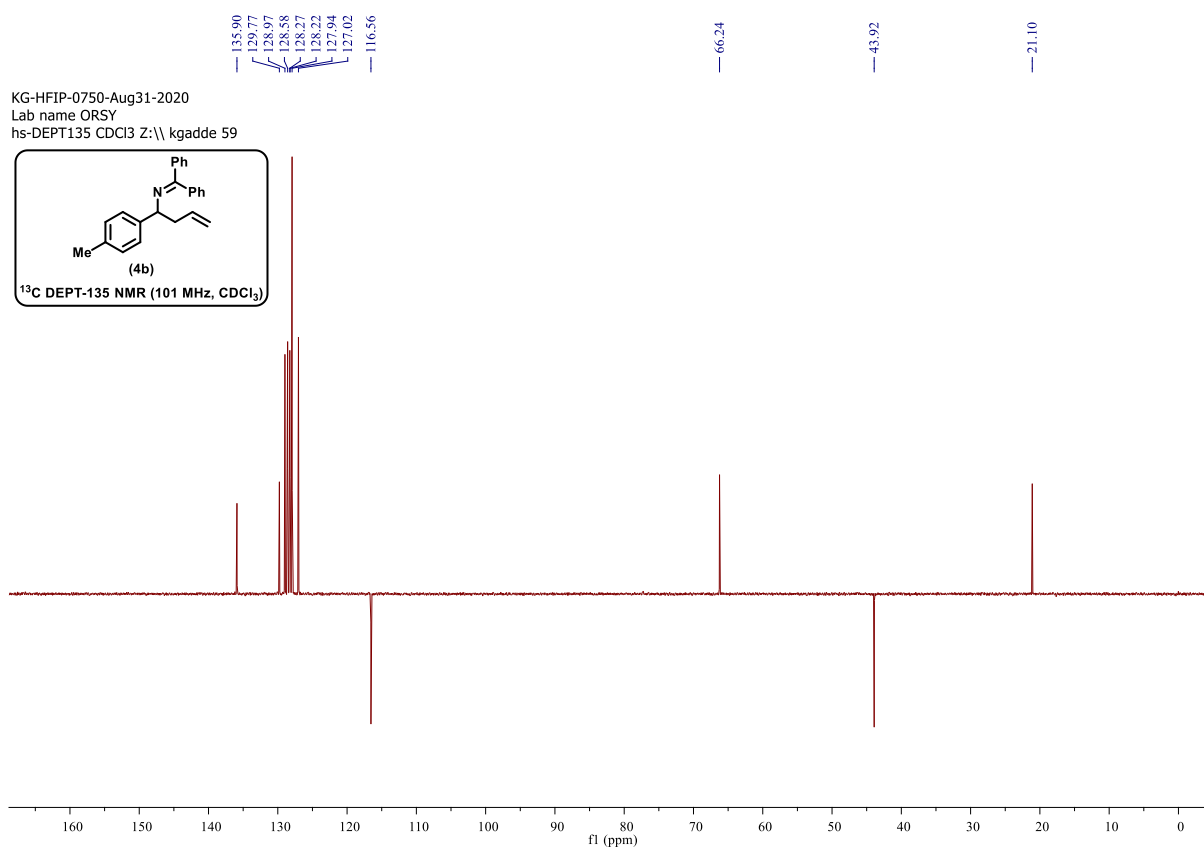
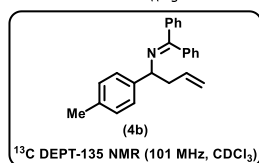


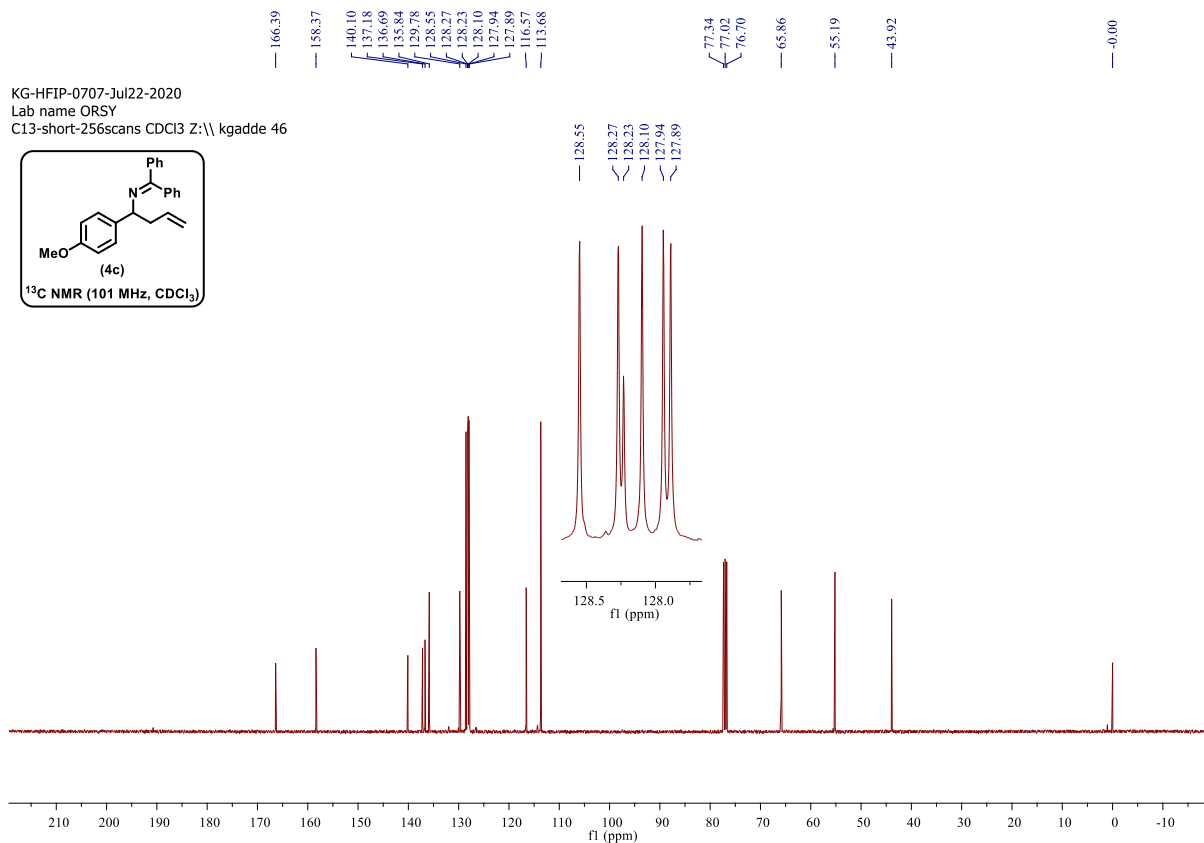
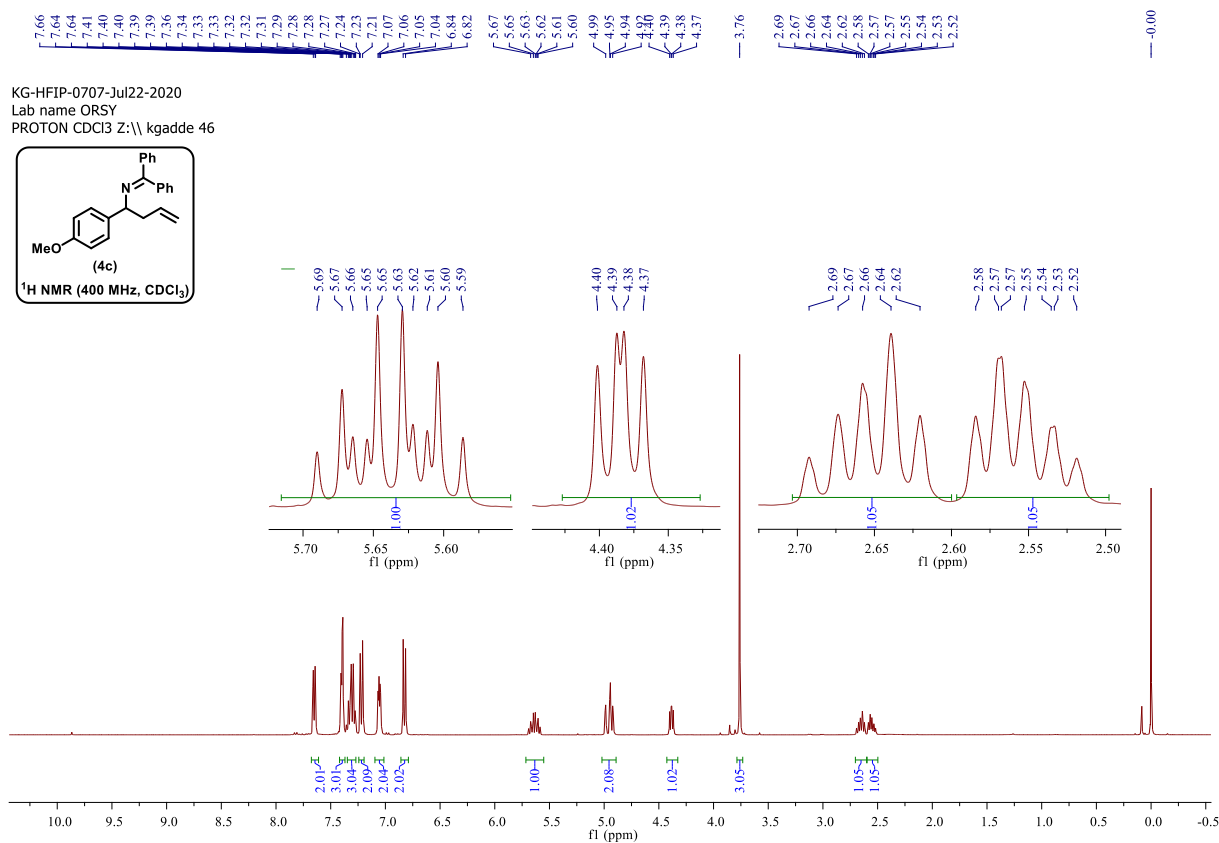
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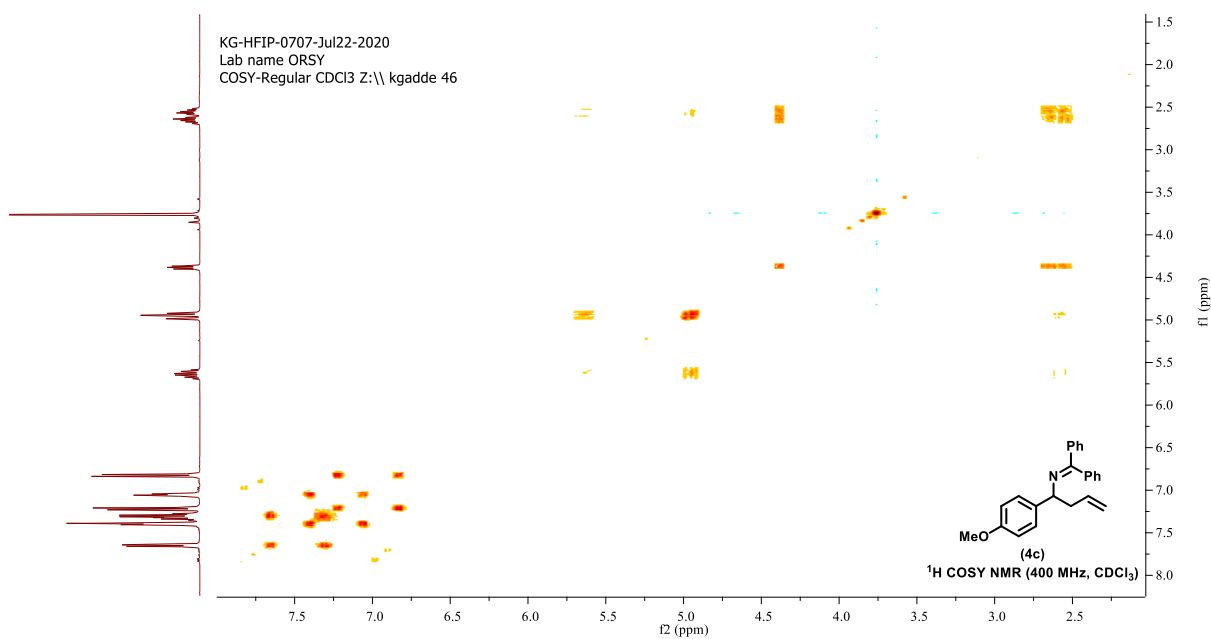
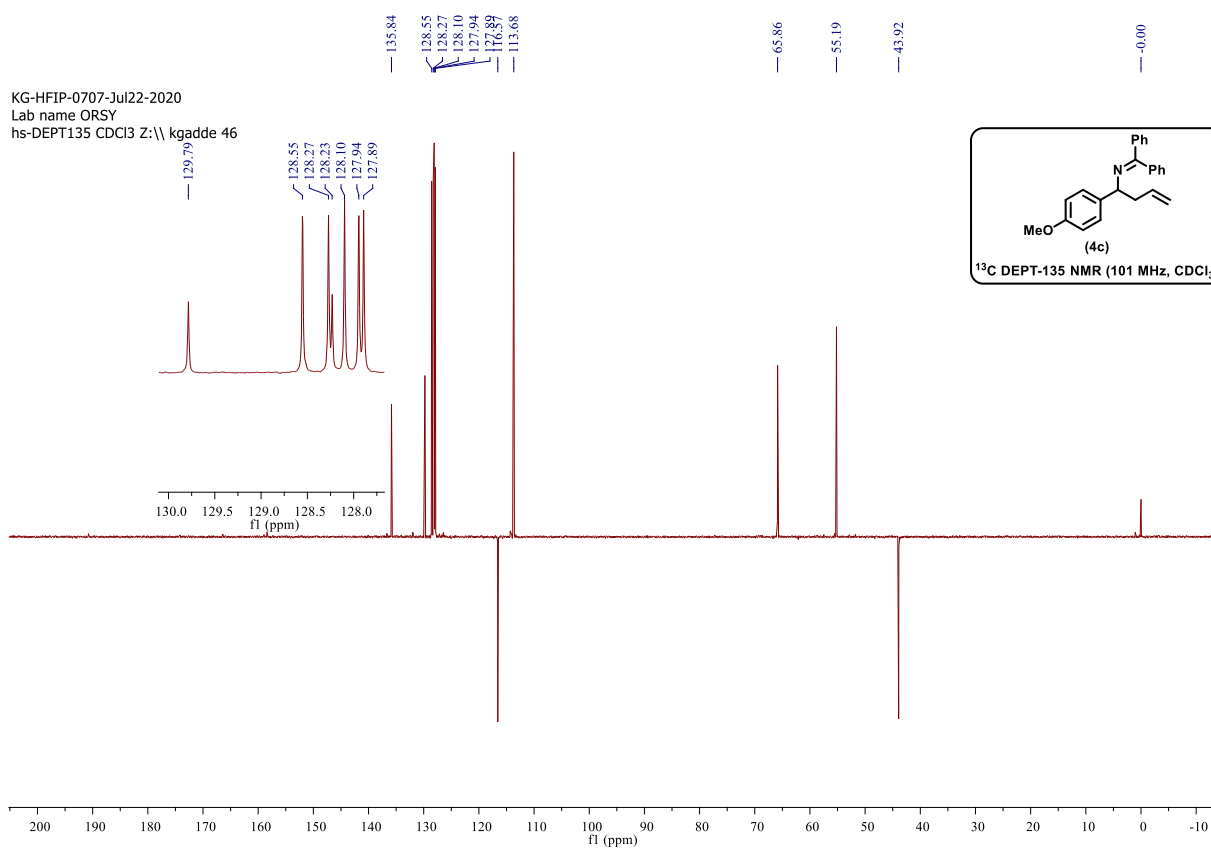


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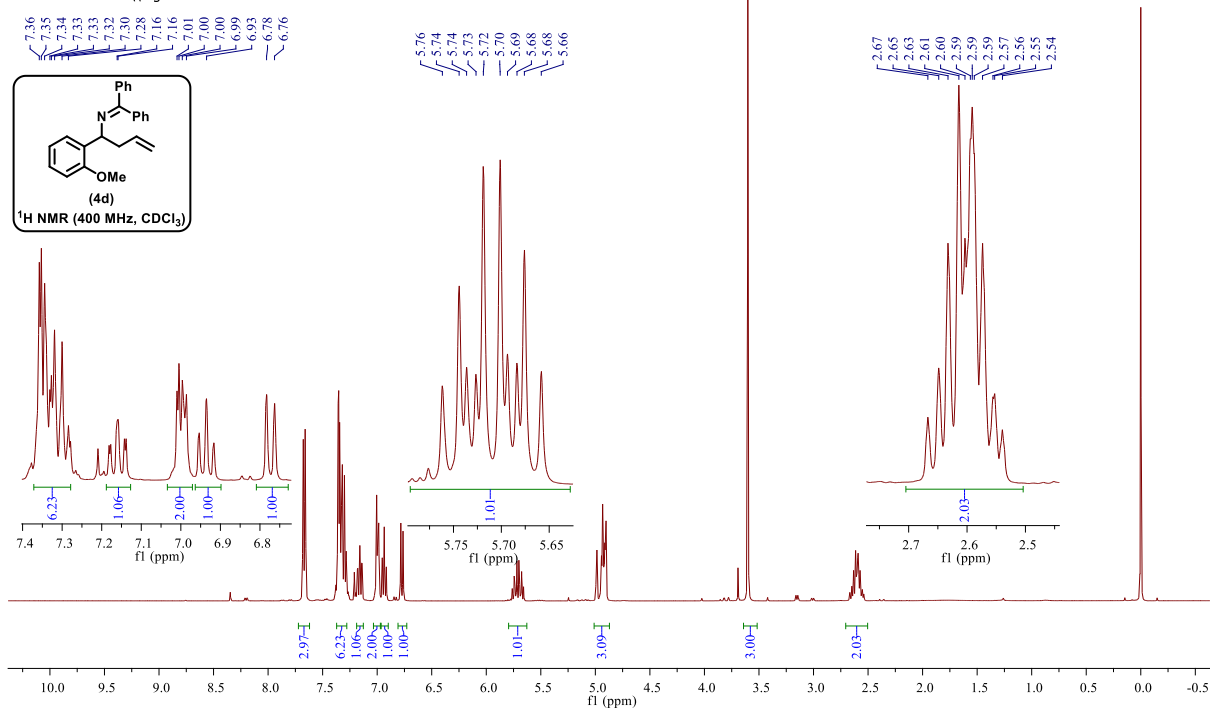




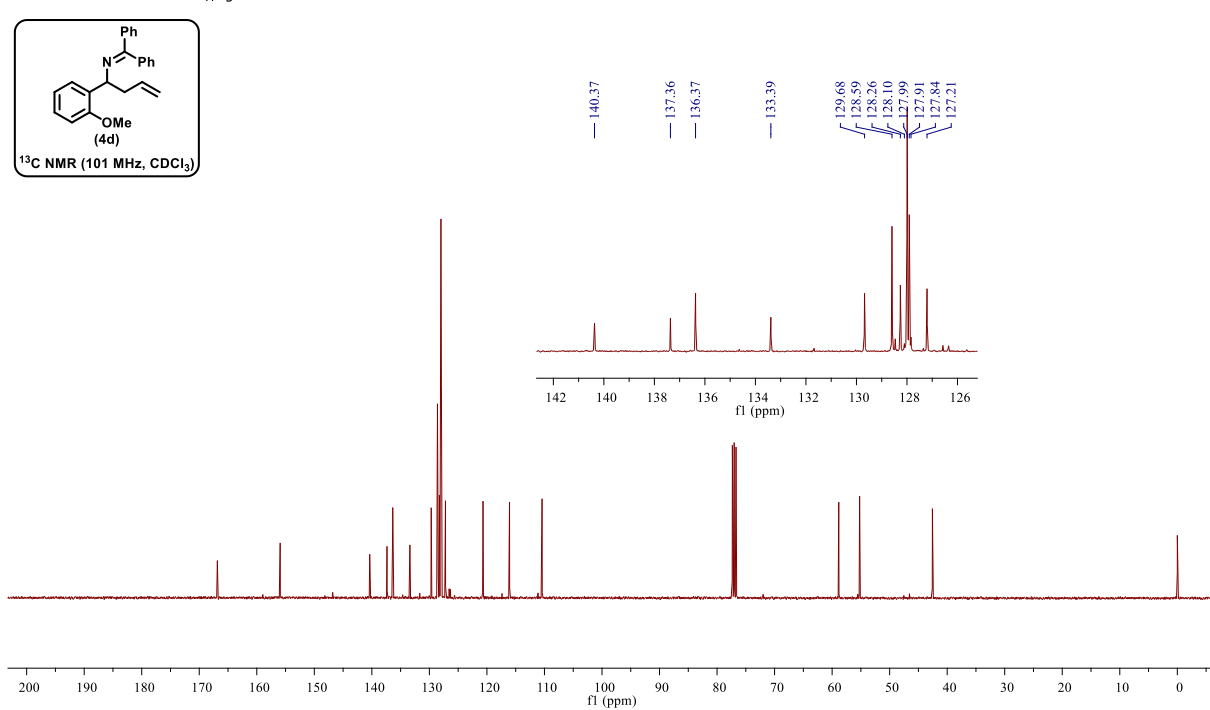
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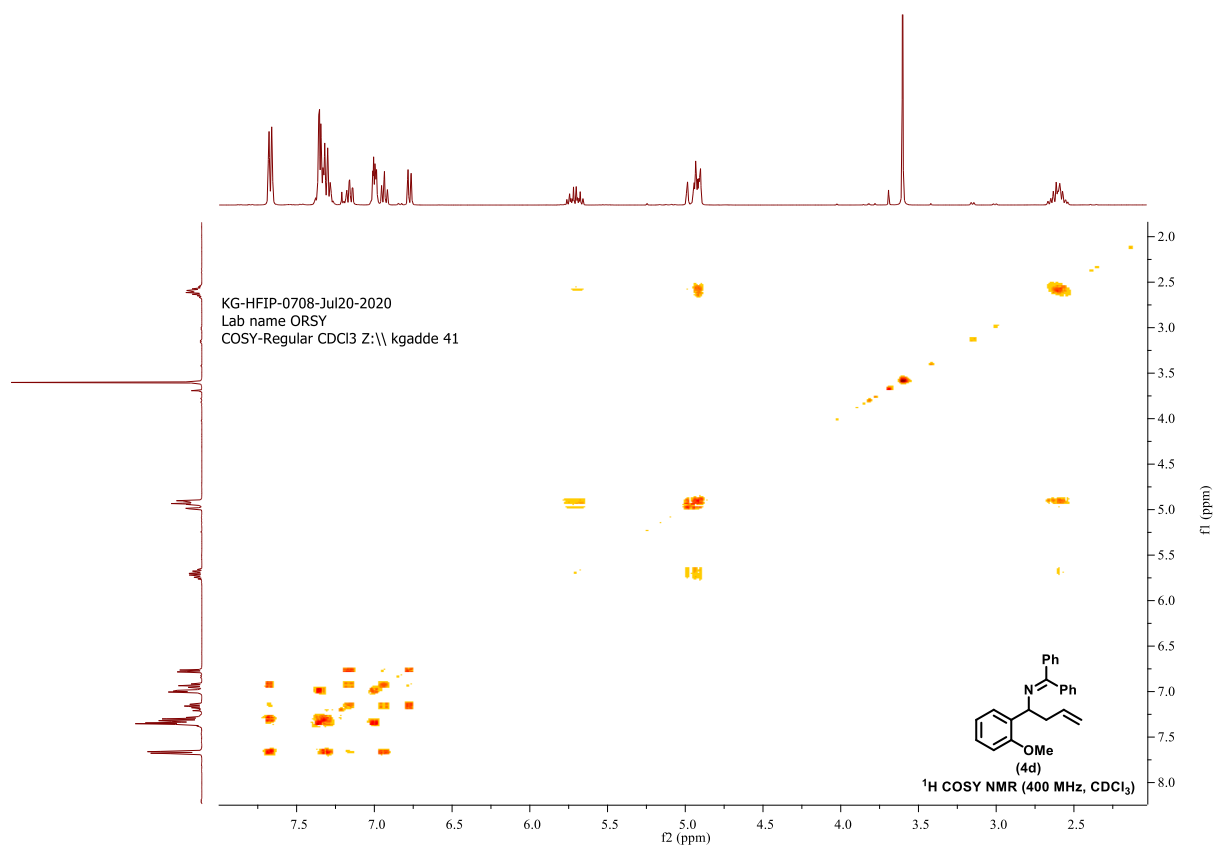
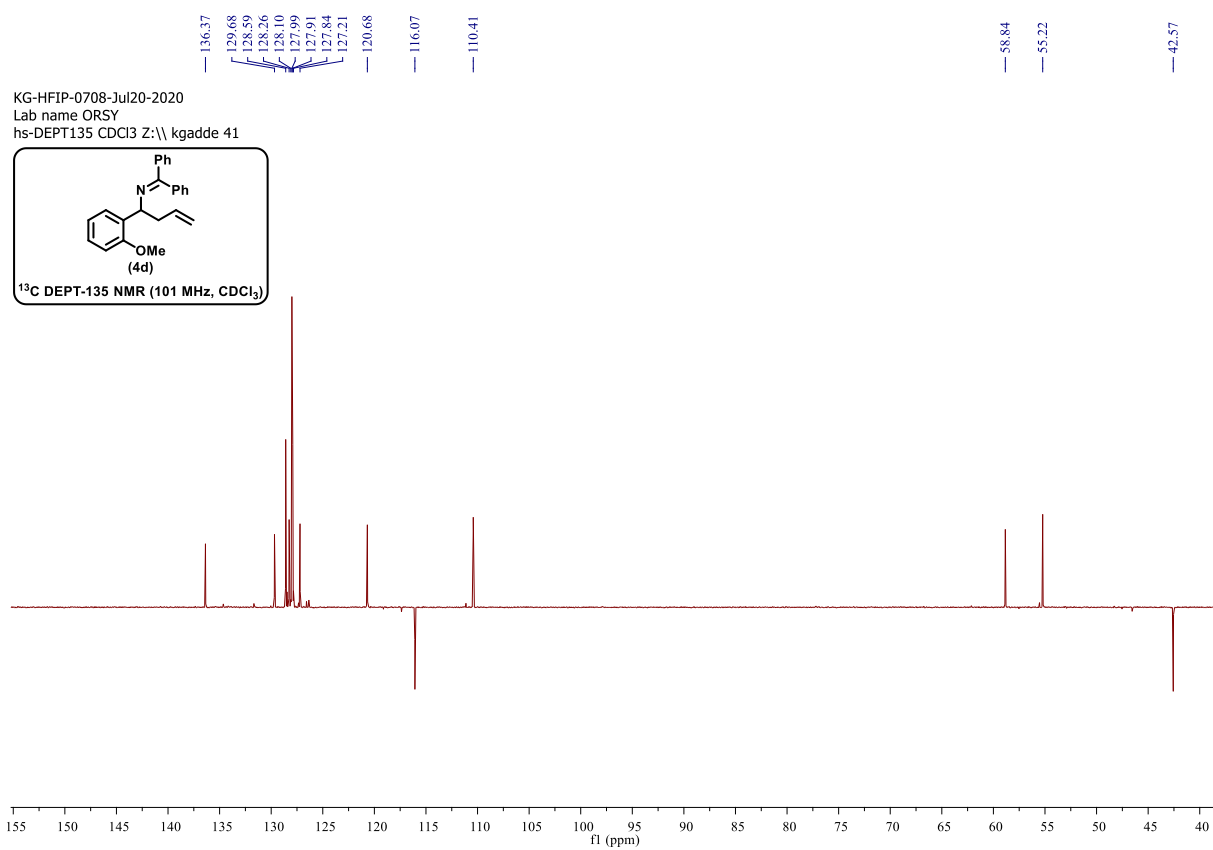


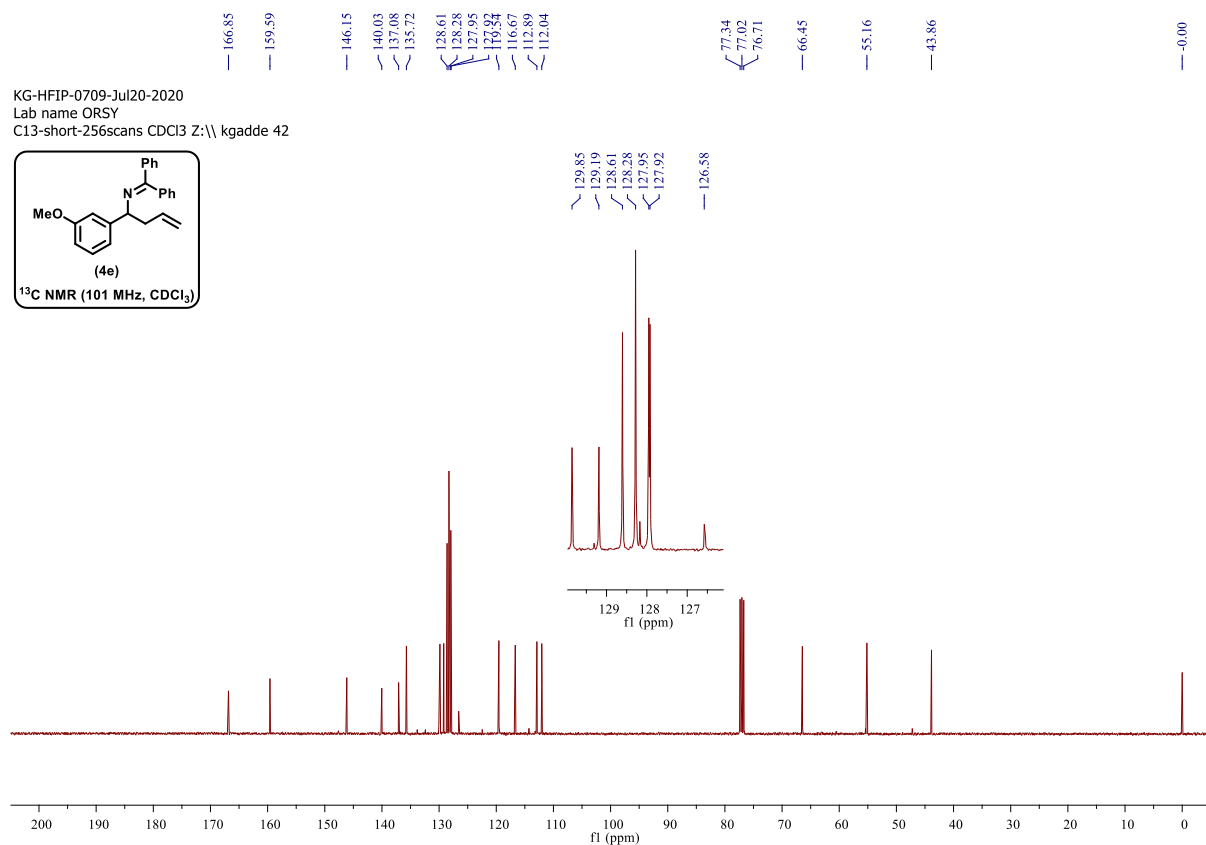
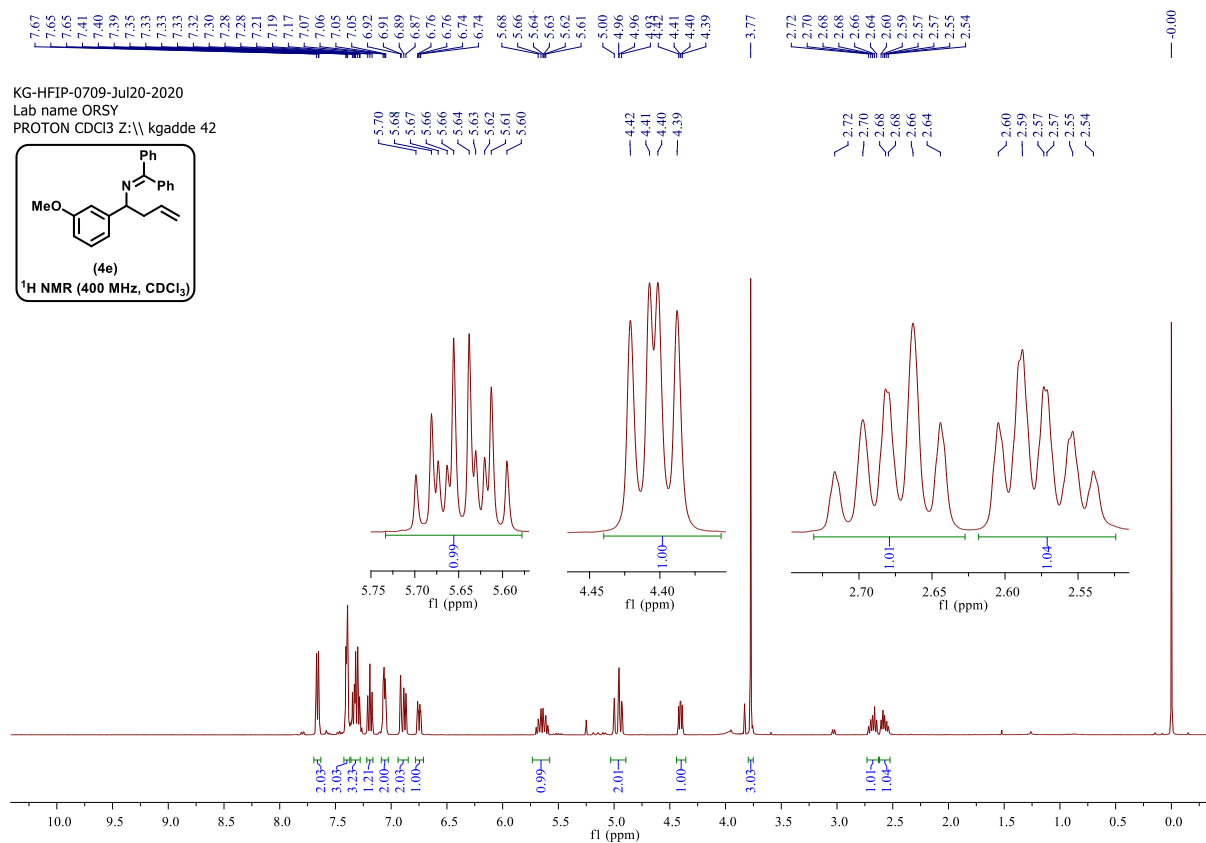
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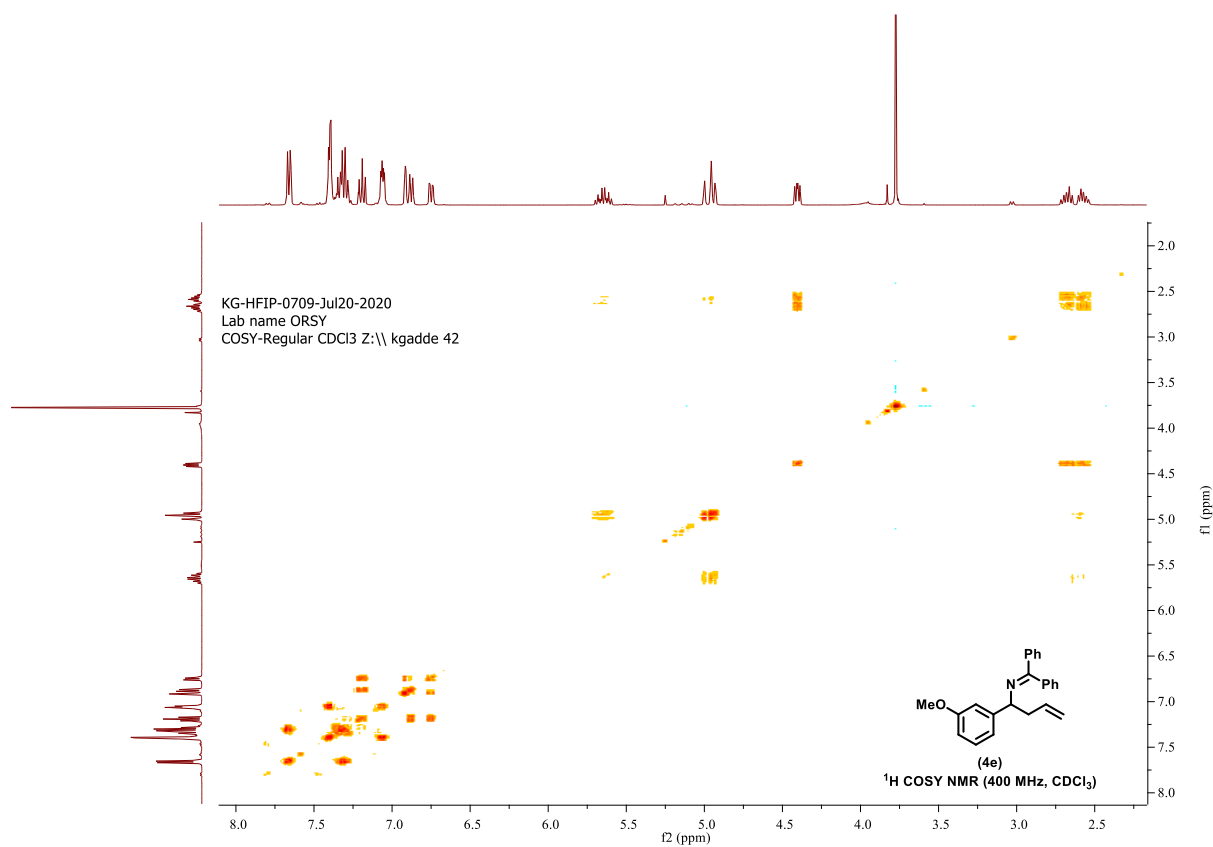
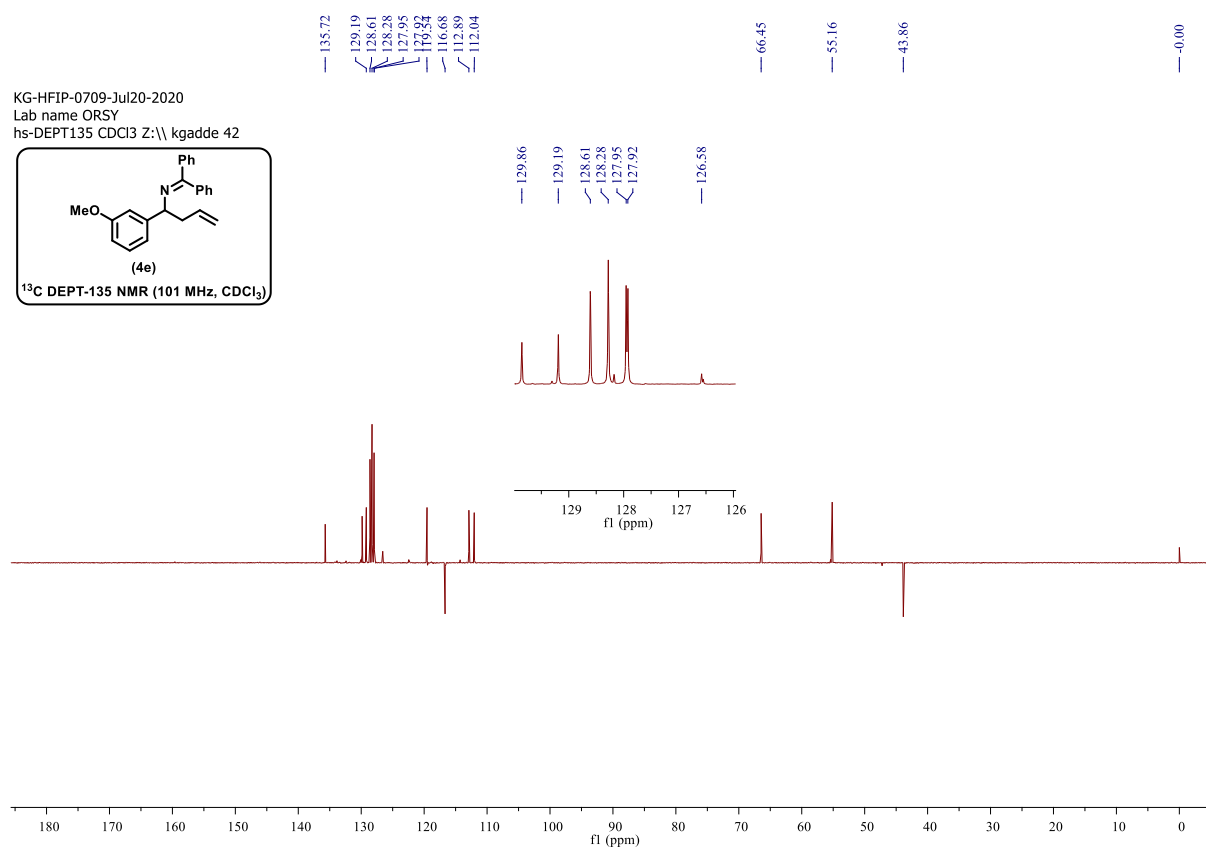
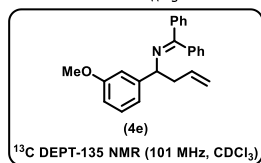
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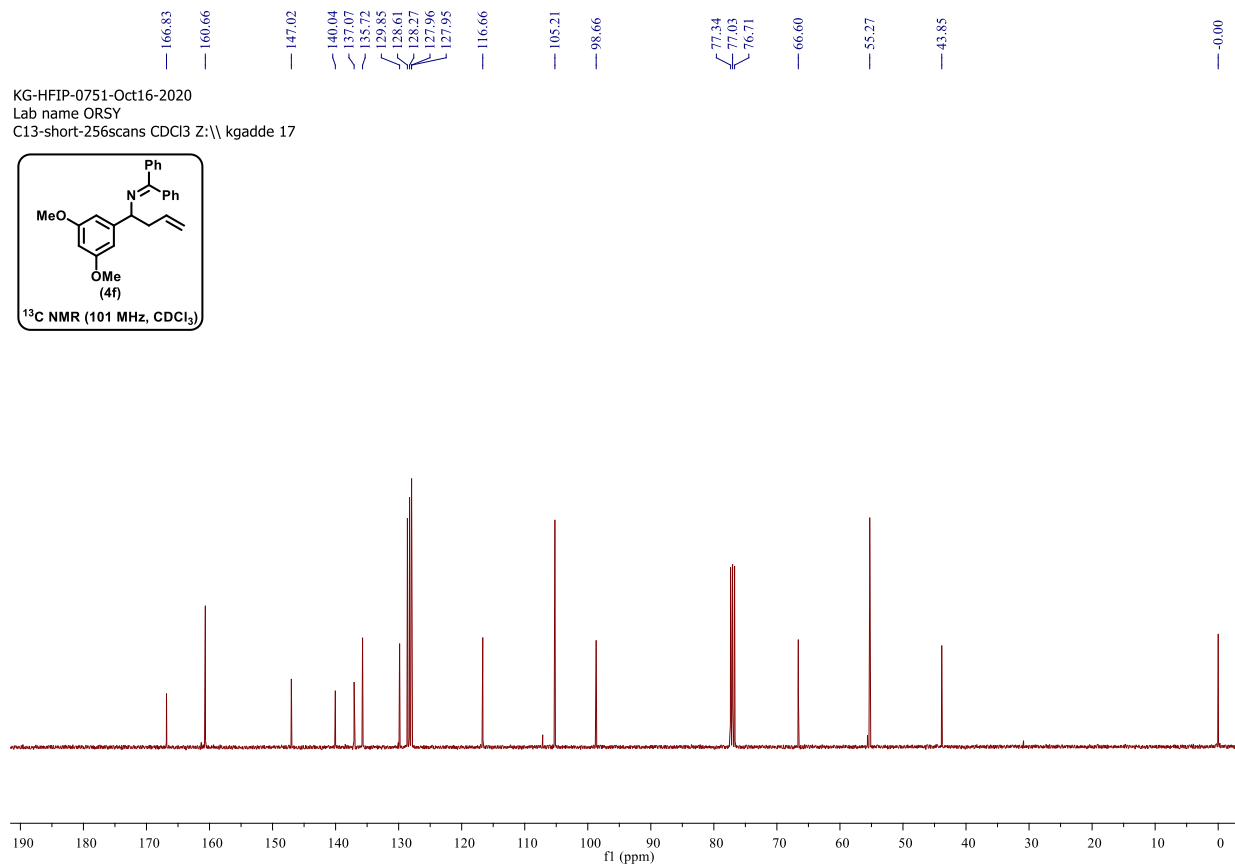
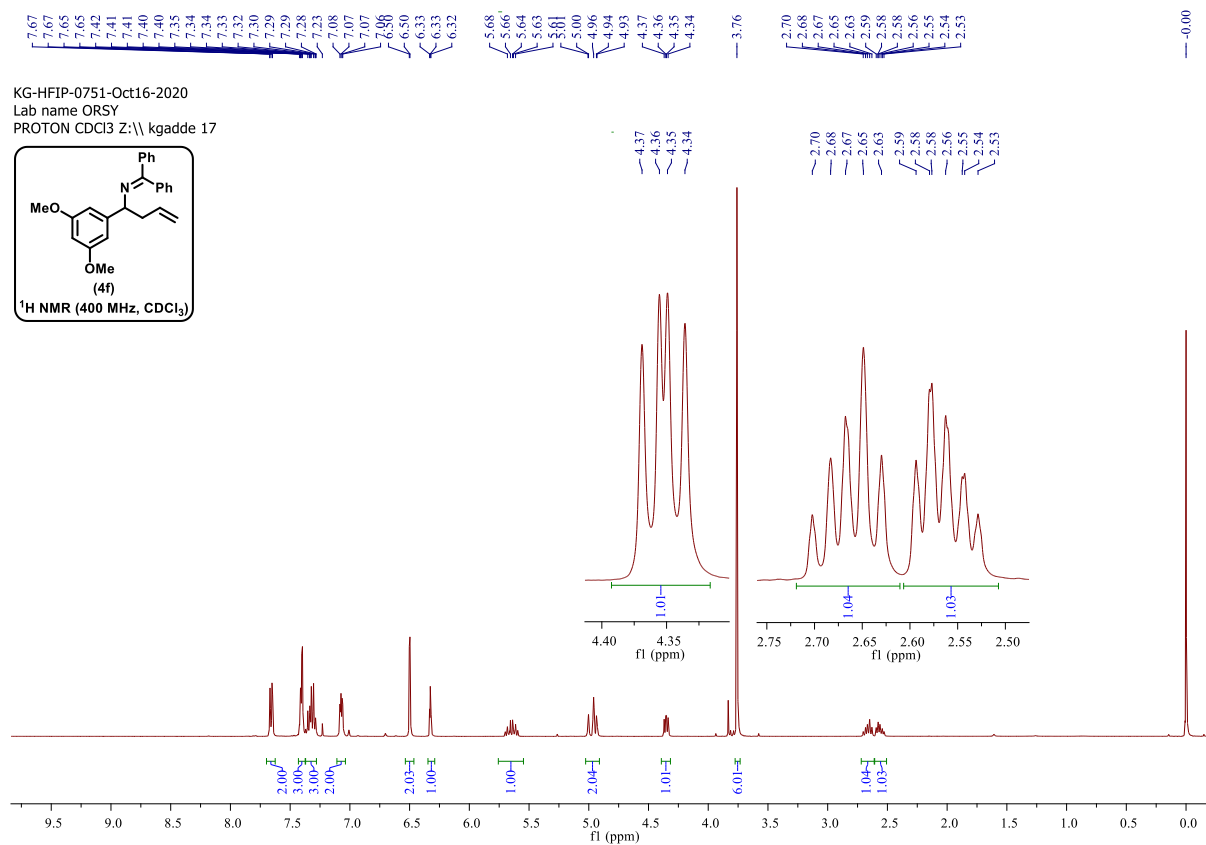




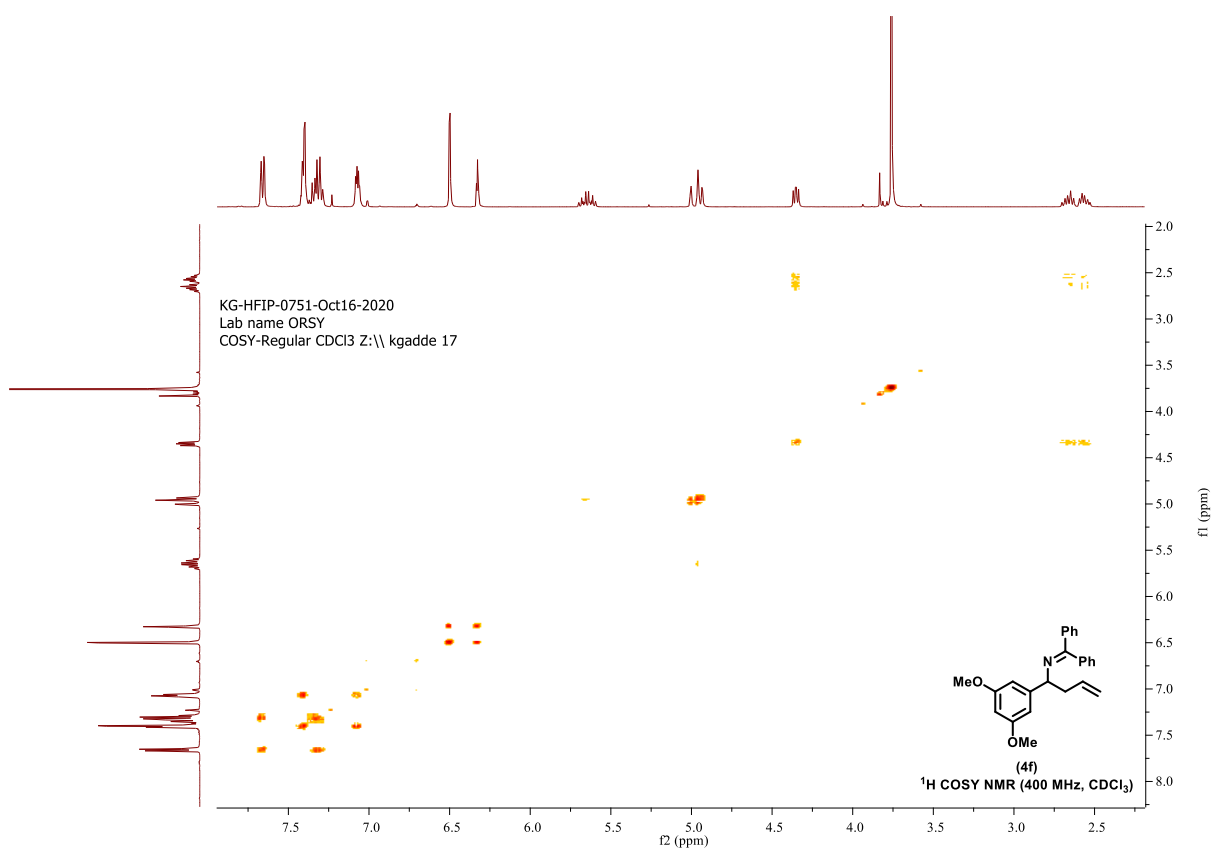
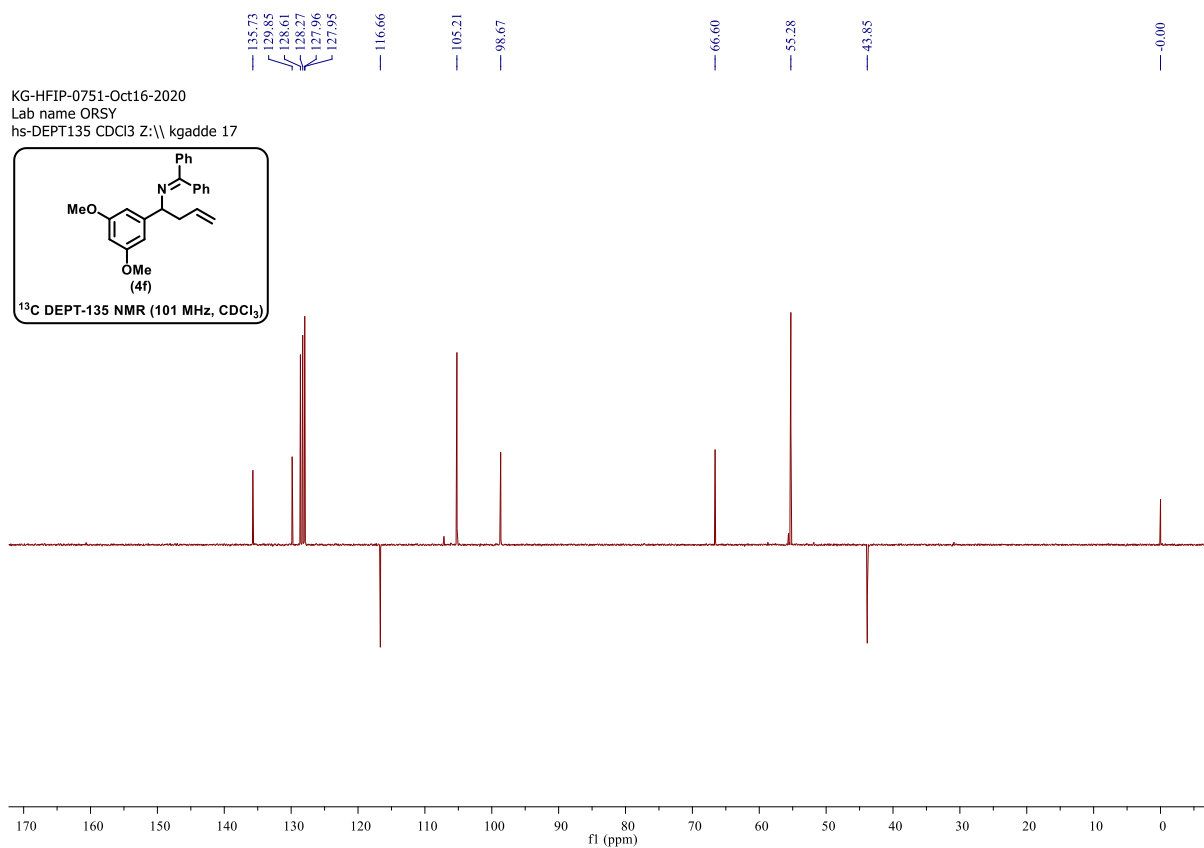
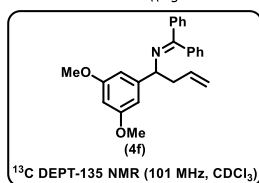


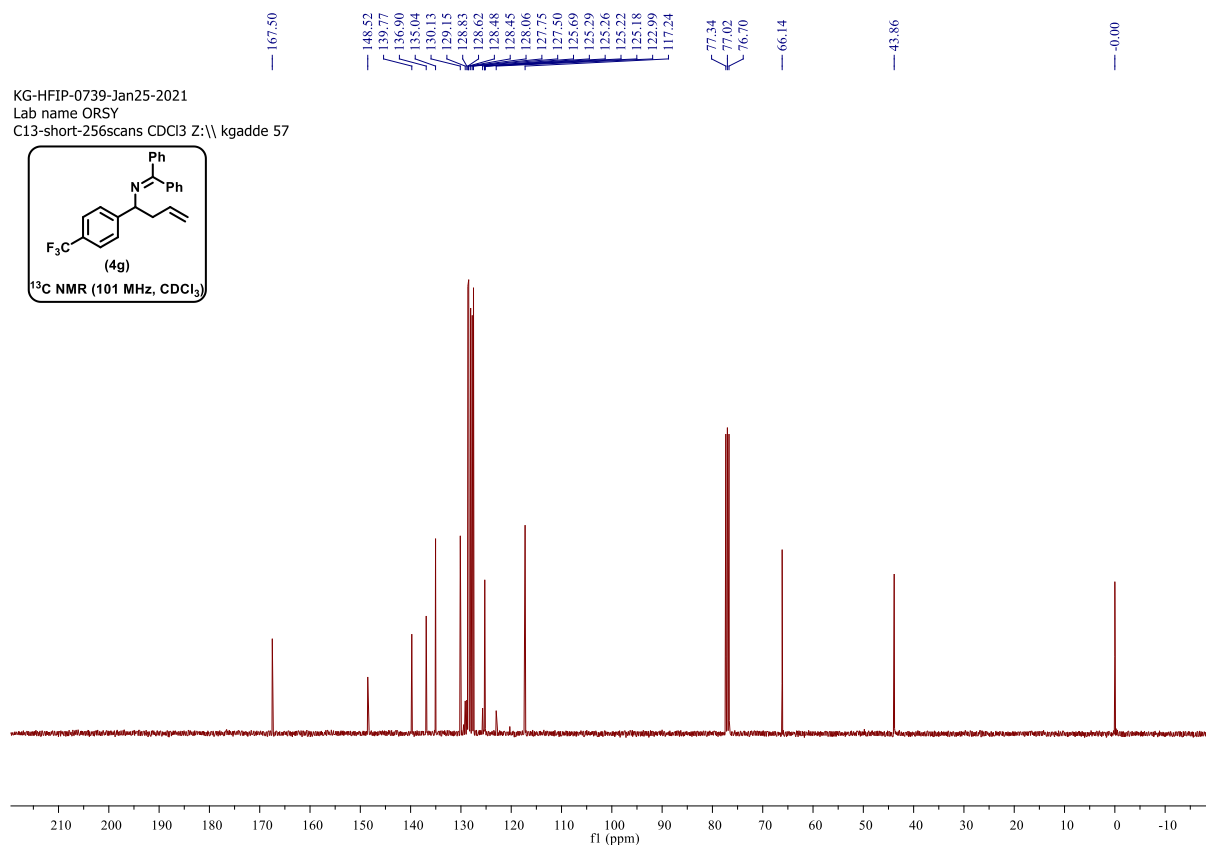
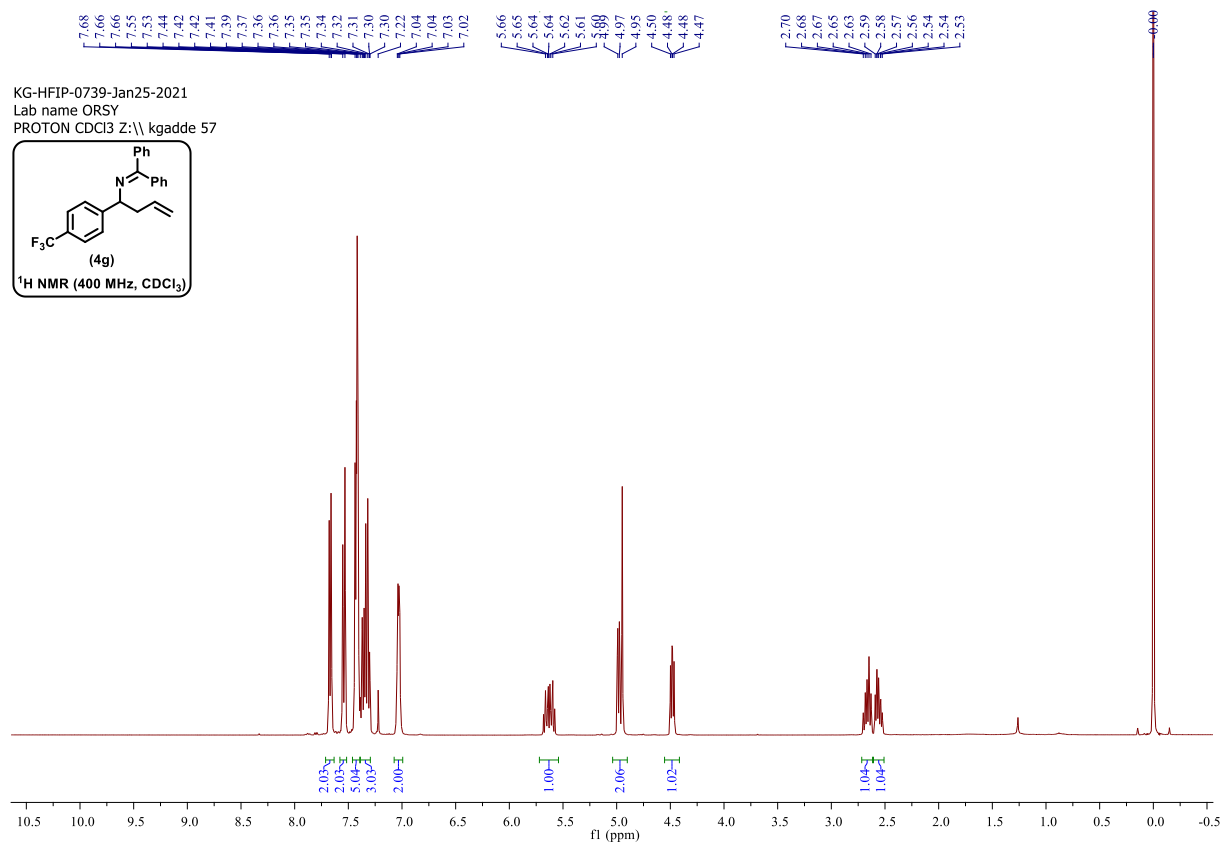

(4e)
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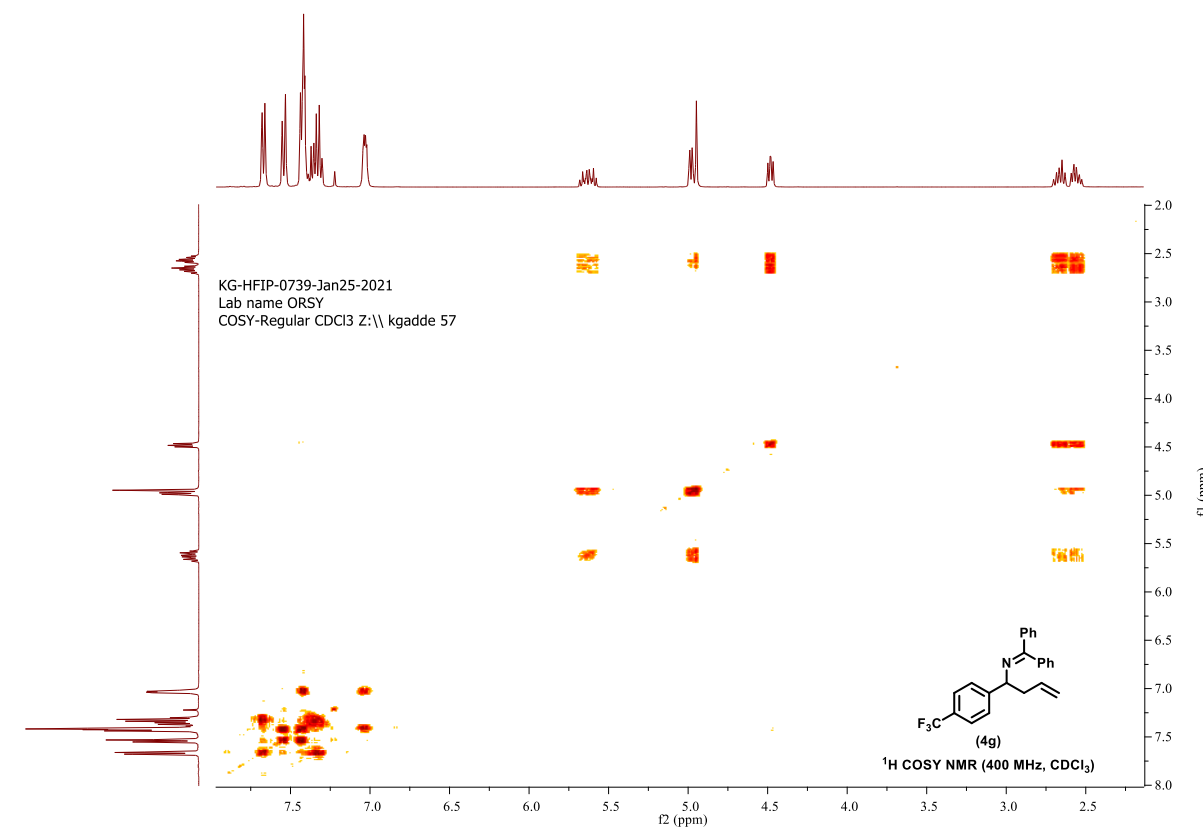


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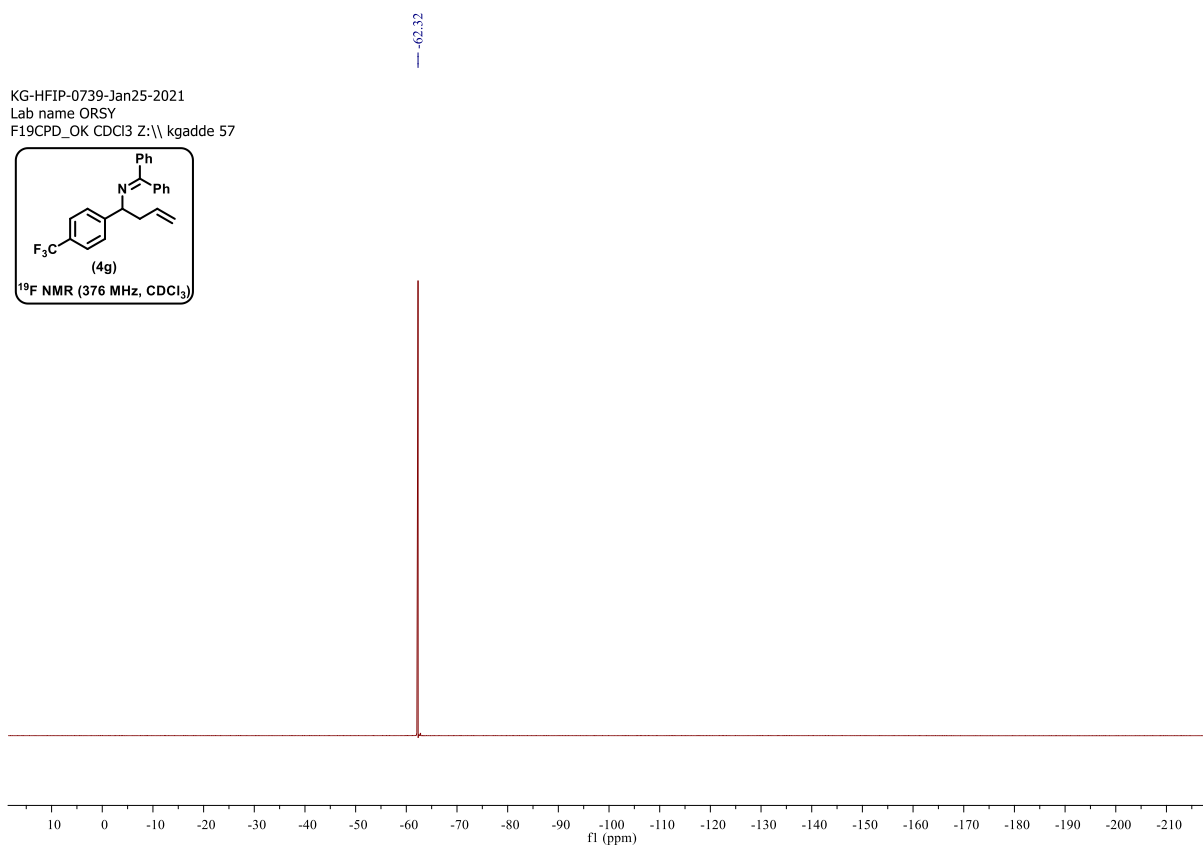
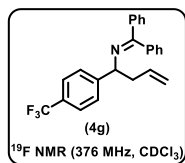


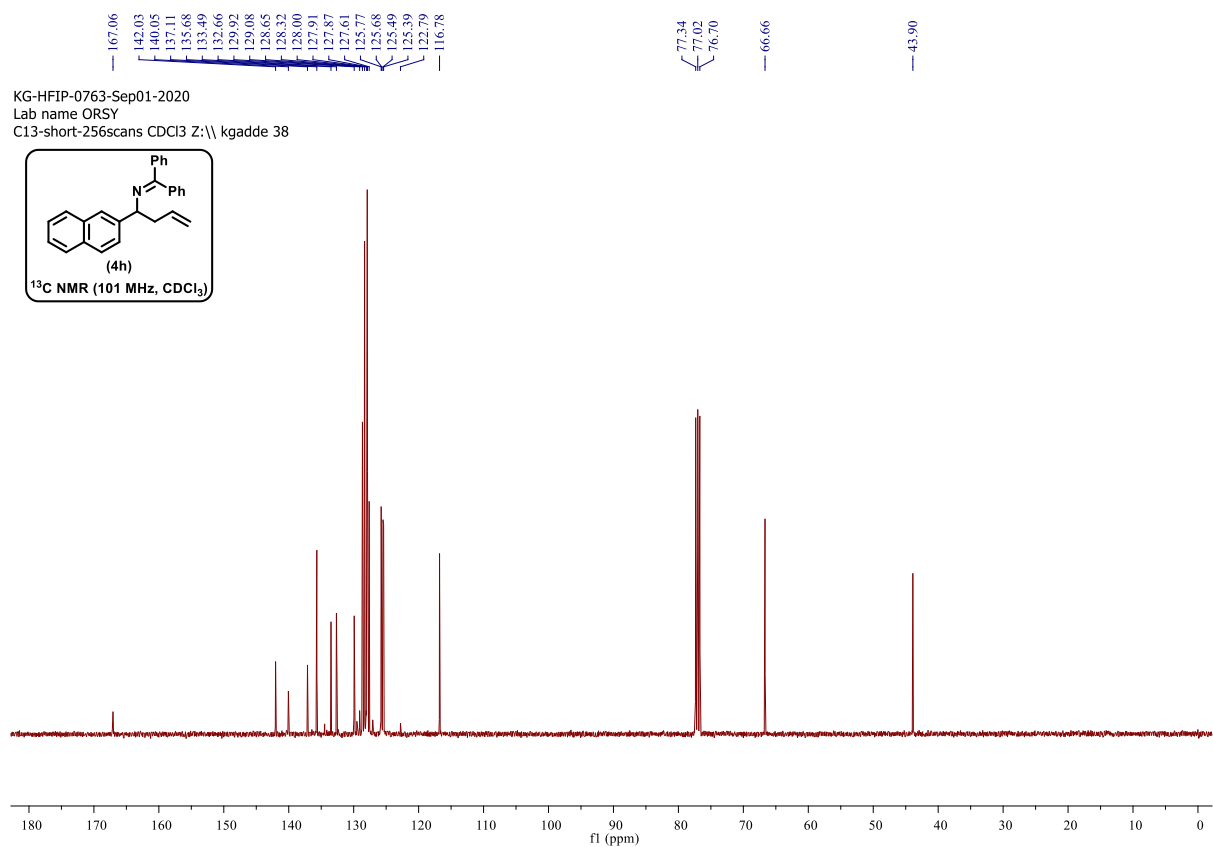
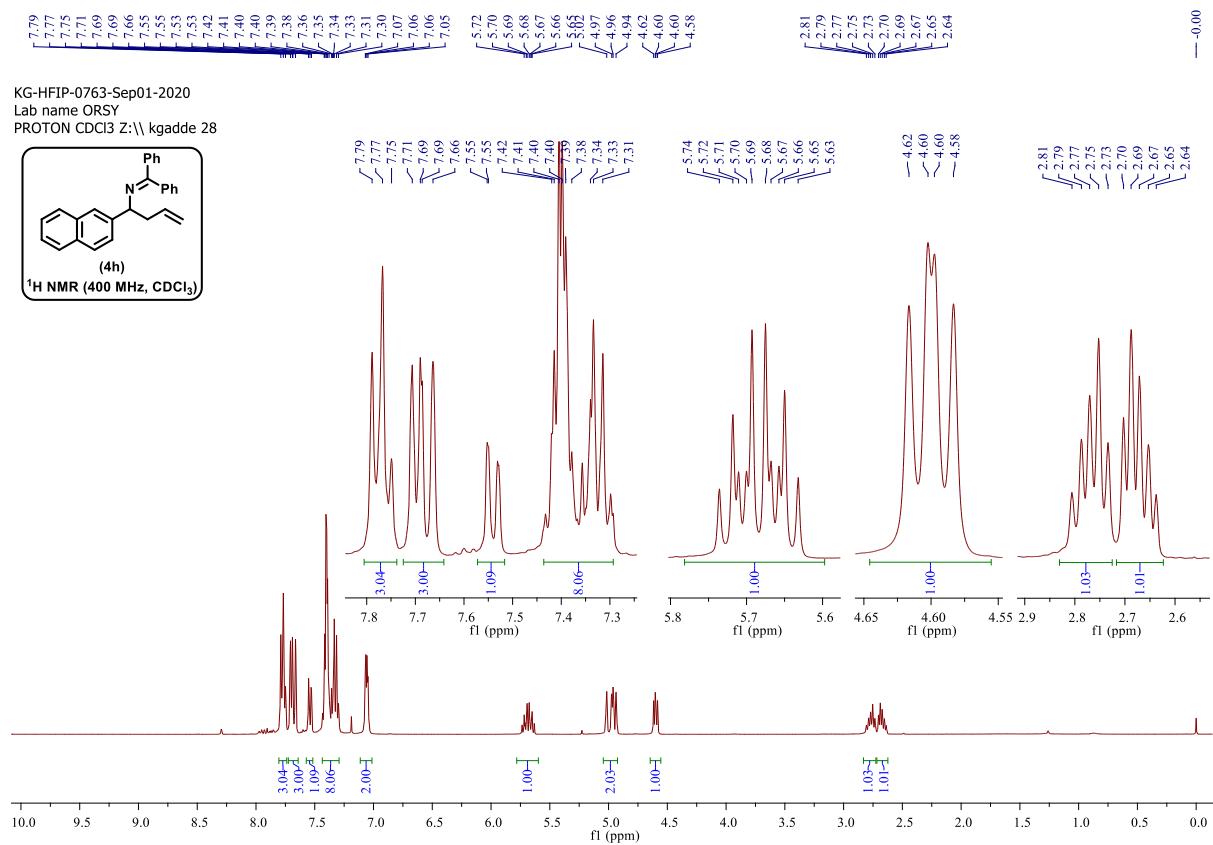


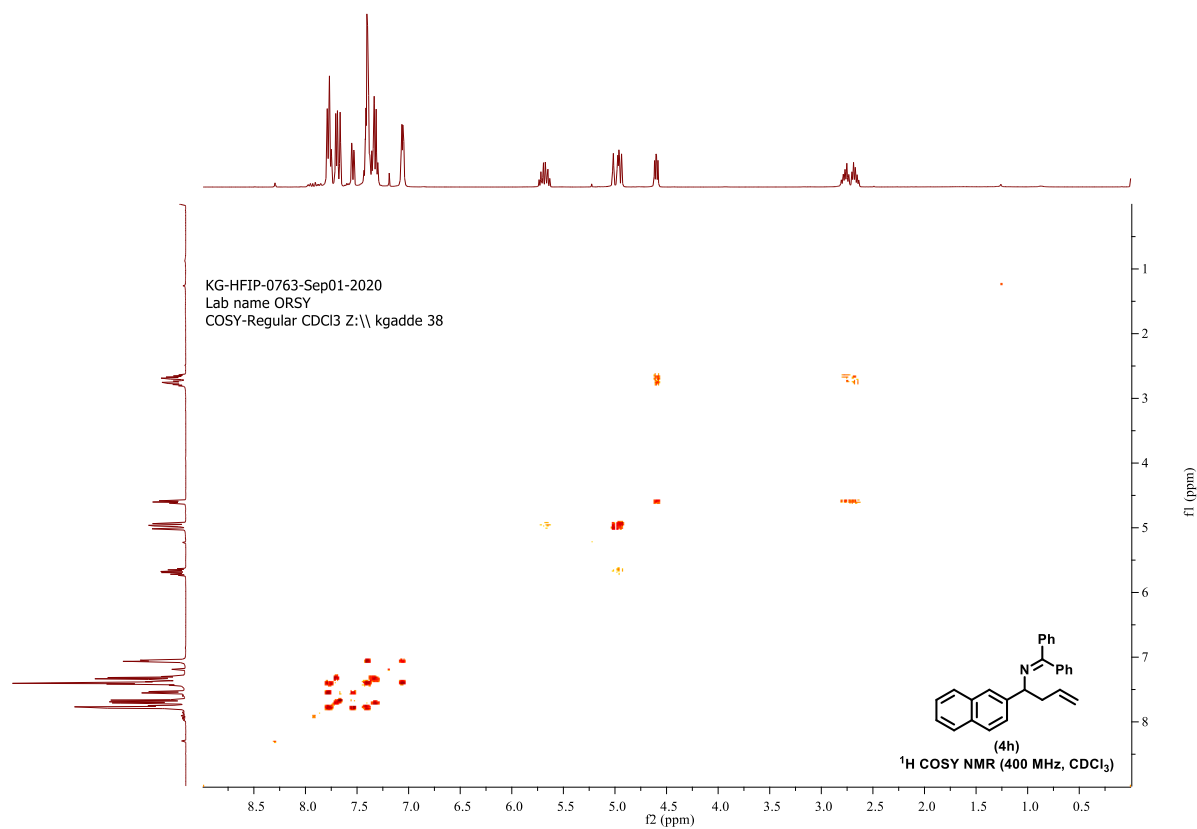
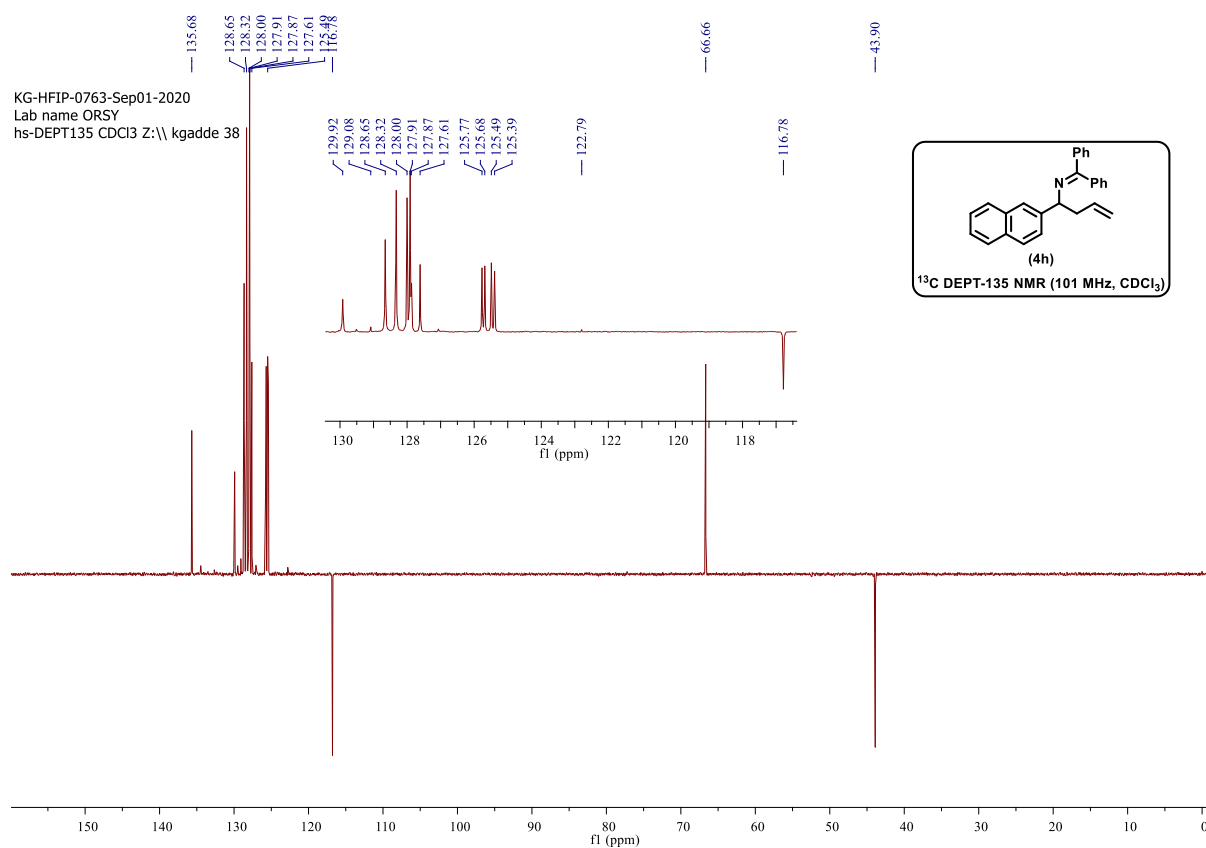

(4g)
¹³C DEPT-135 NMR (101 MHz, CDCl₃)

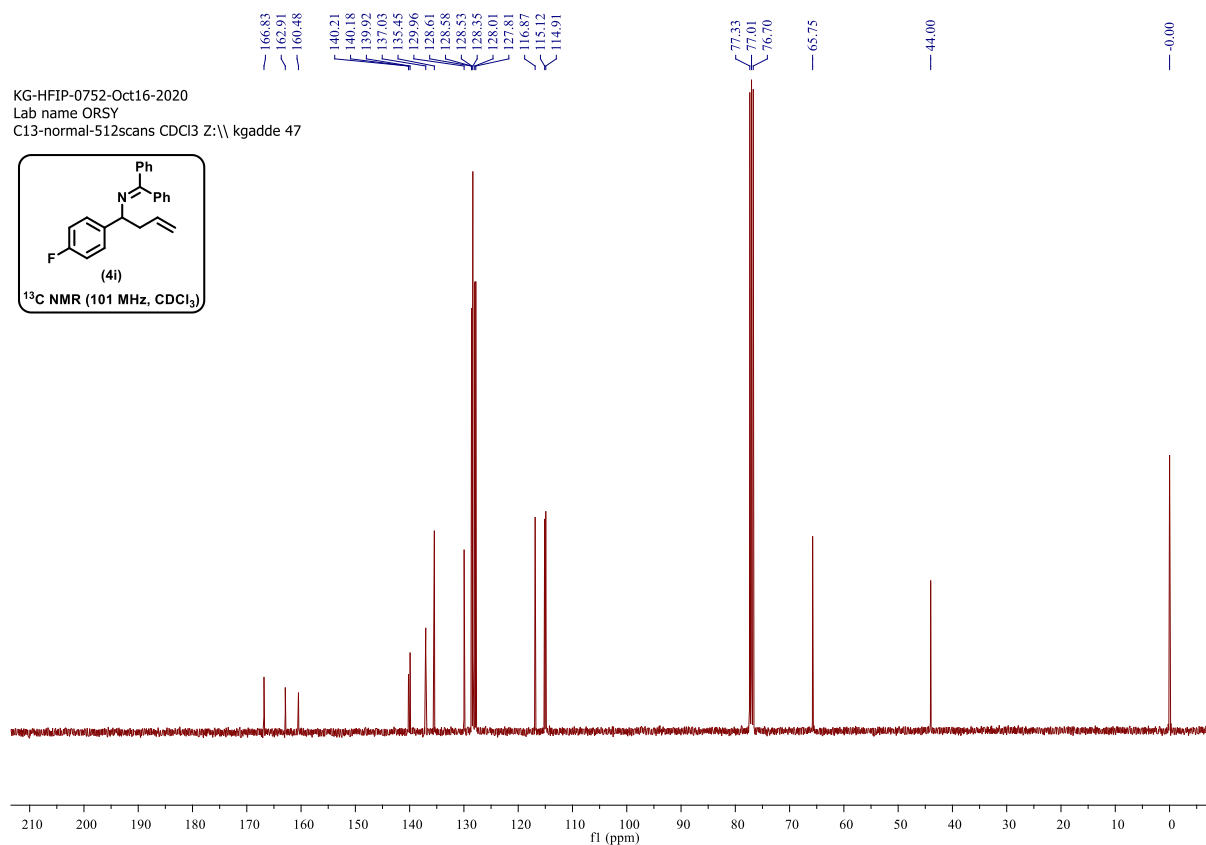
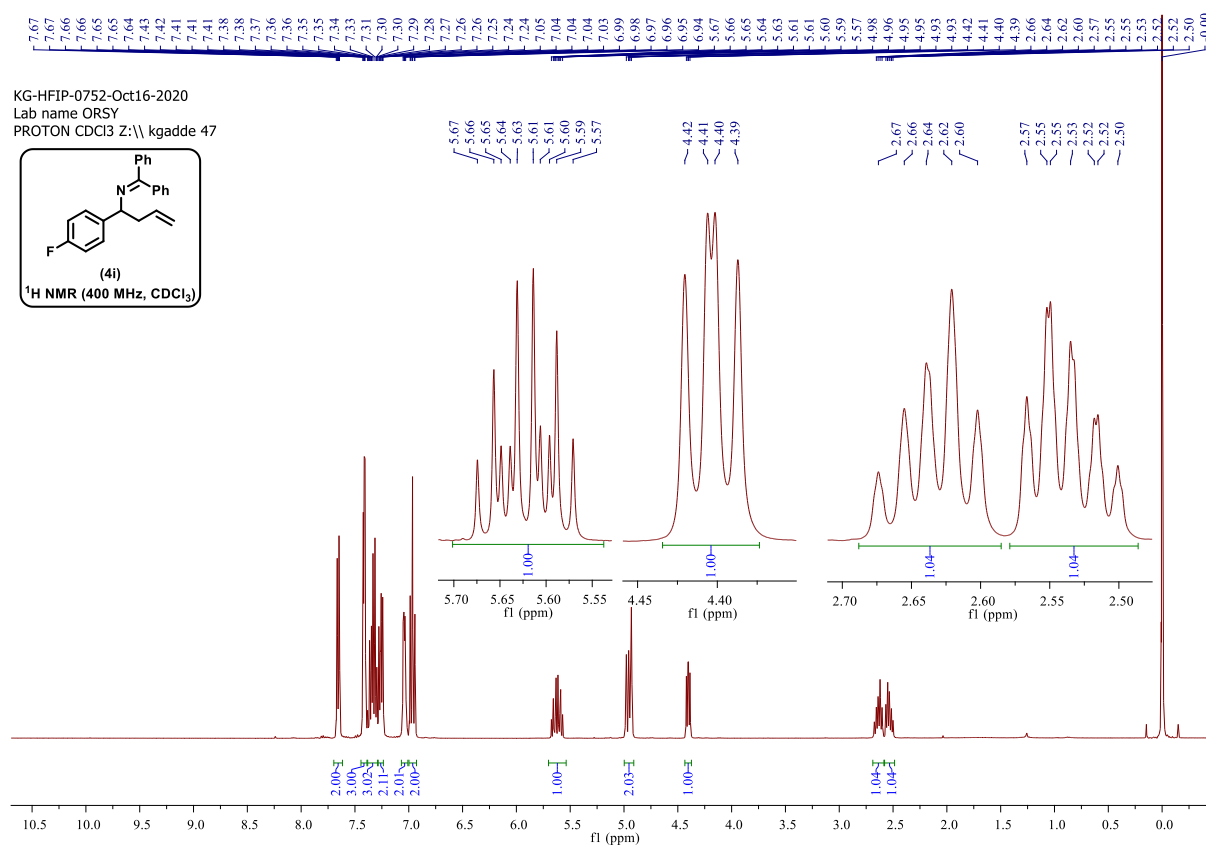


KG-HFIP-0739-Jan25-2021
Lab name ORSY
F19CPD_OK CDCl3 Z:\\ kgadde 57

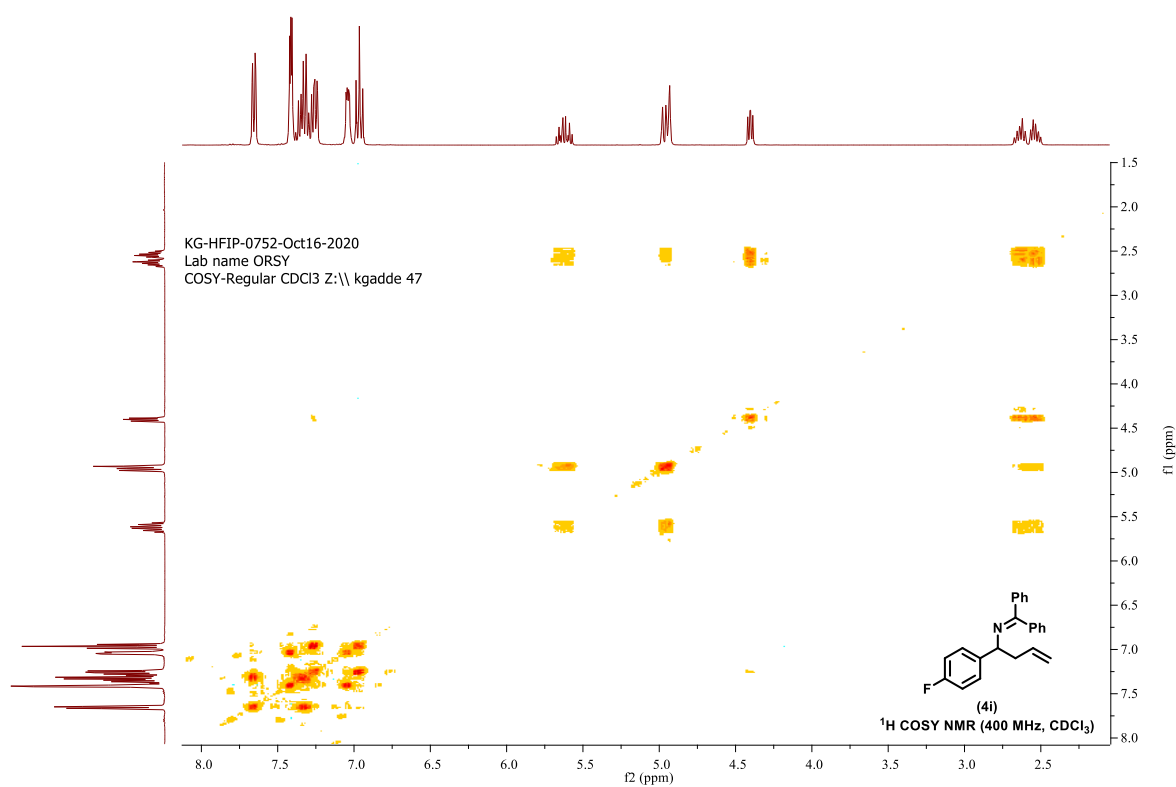
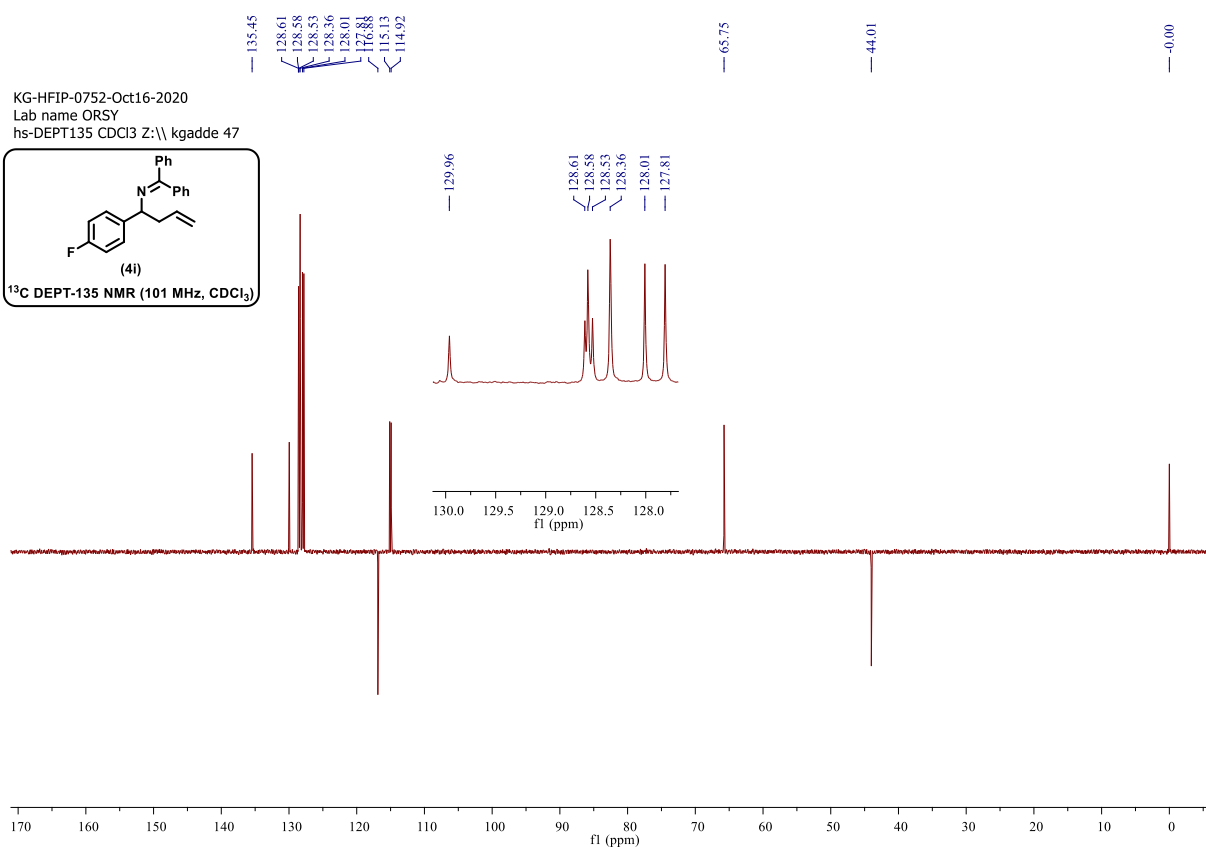
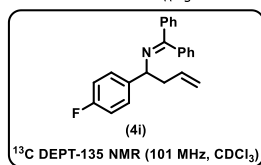




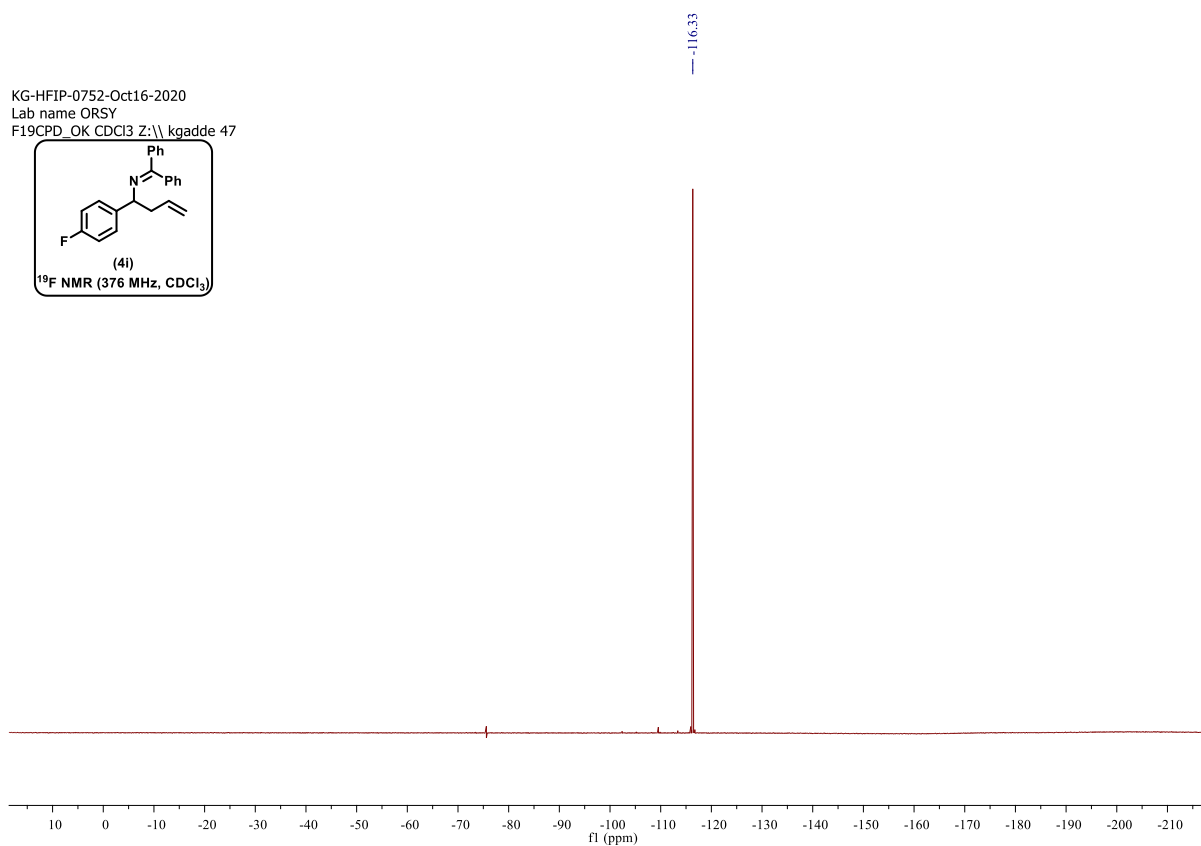
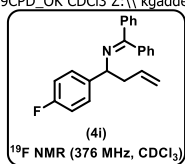




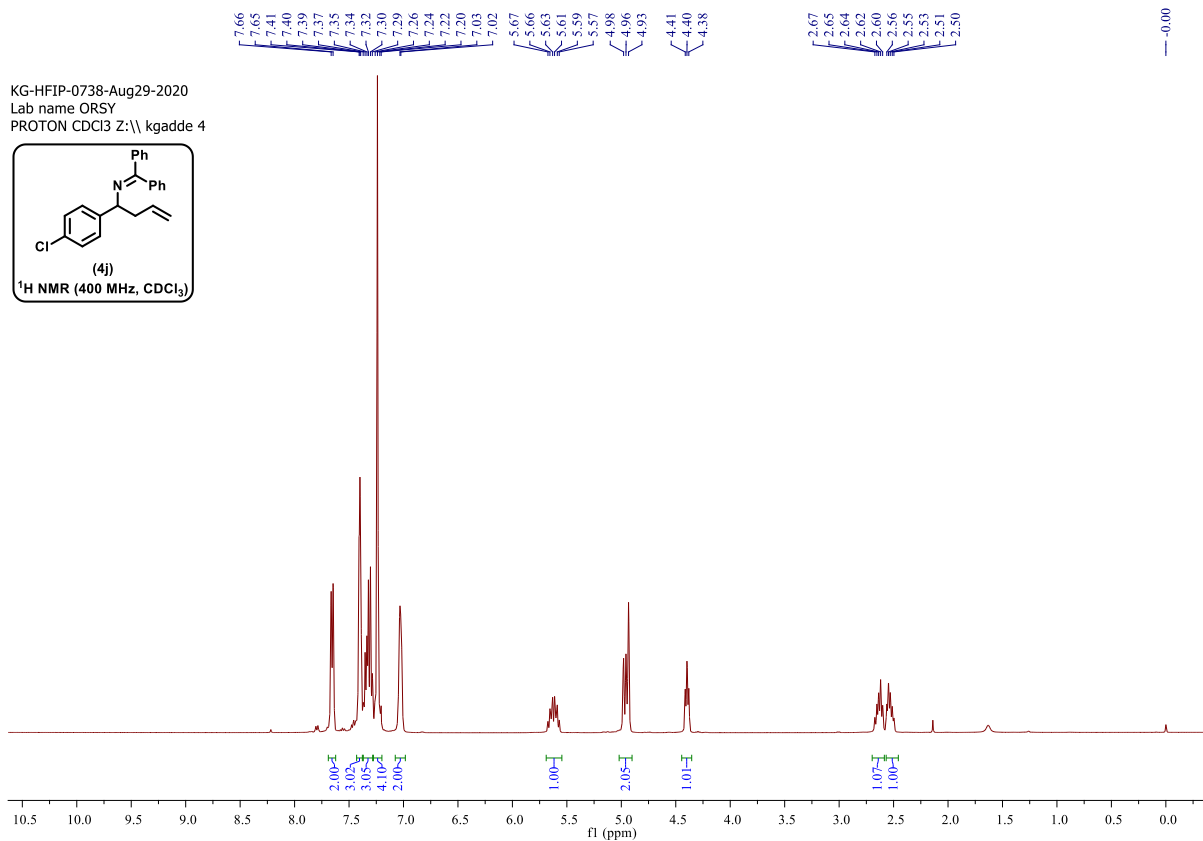
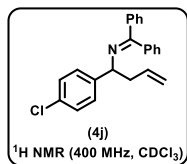
KG-HFIP-0752-Oct16-2020
 Lab name ORSY
 hs-DEPT135 CDCl3 Z:\\ kgadde 47



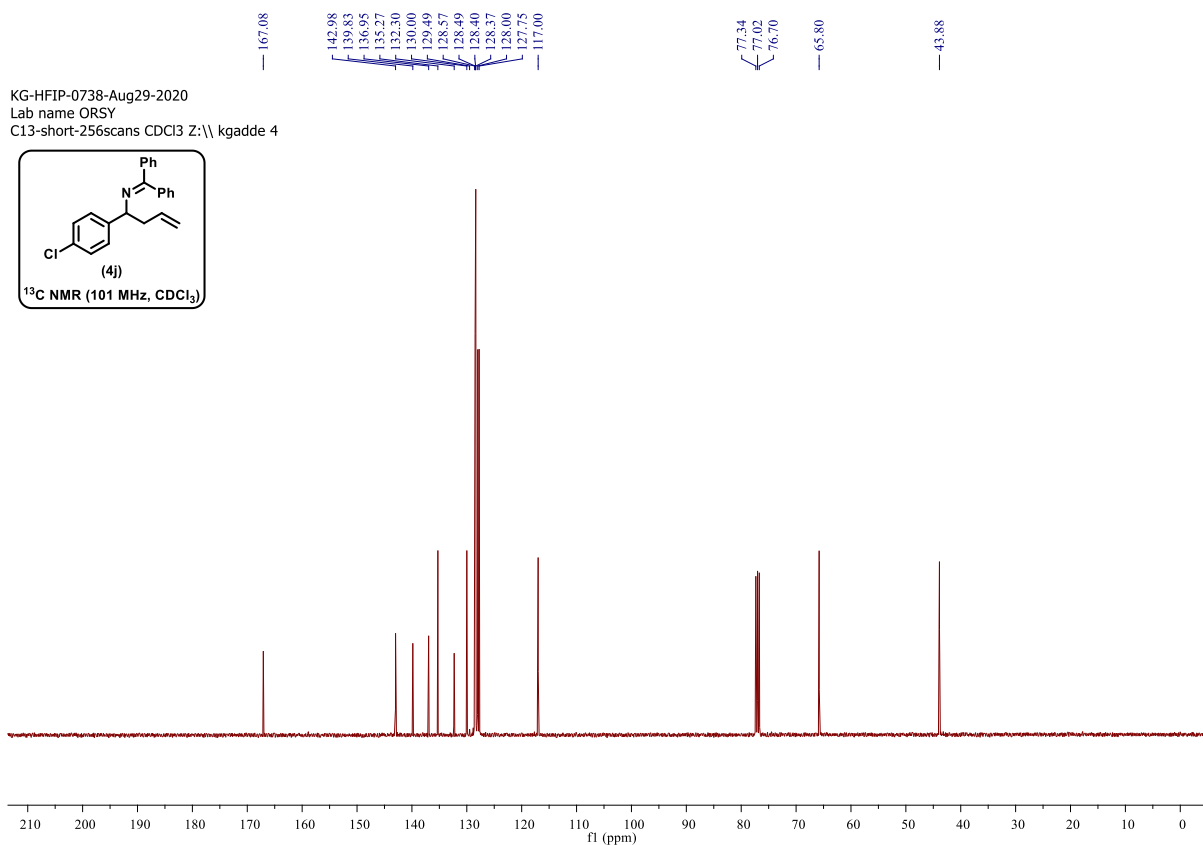
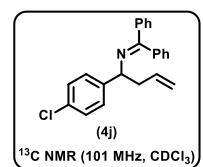
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Lab name ORSY
F19CPD_OK CDCl3 Z:\\ kgadde 47

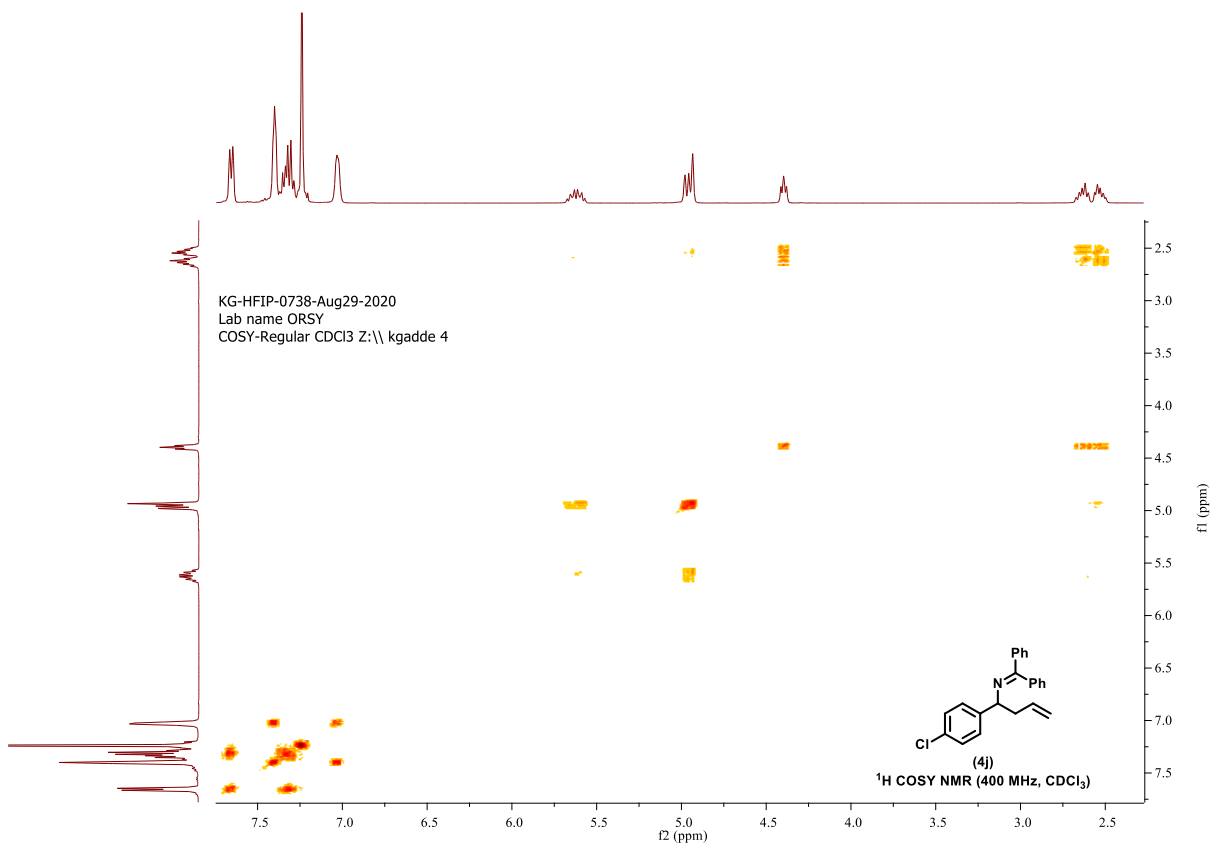
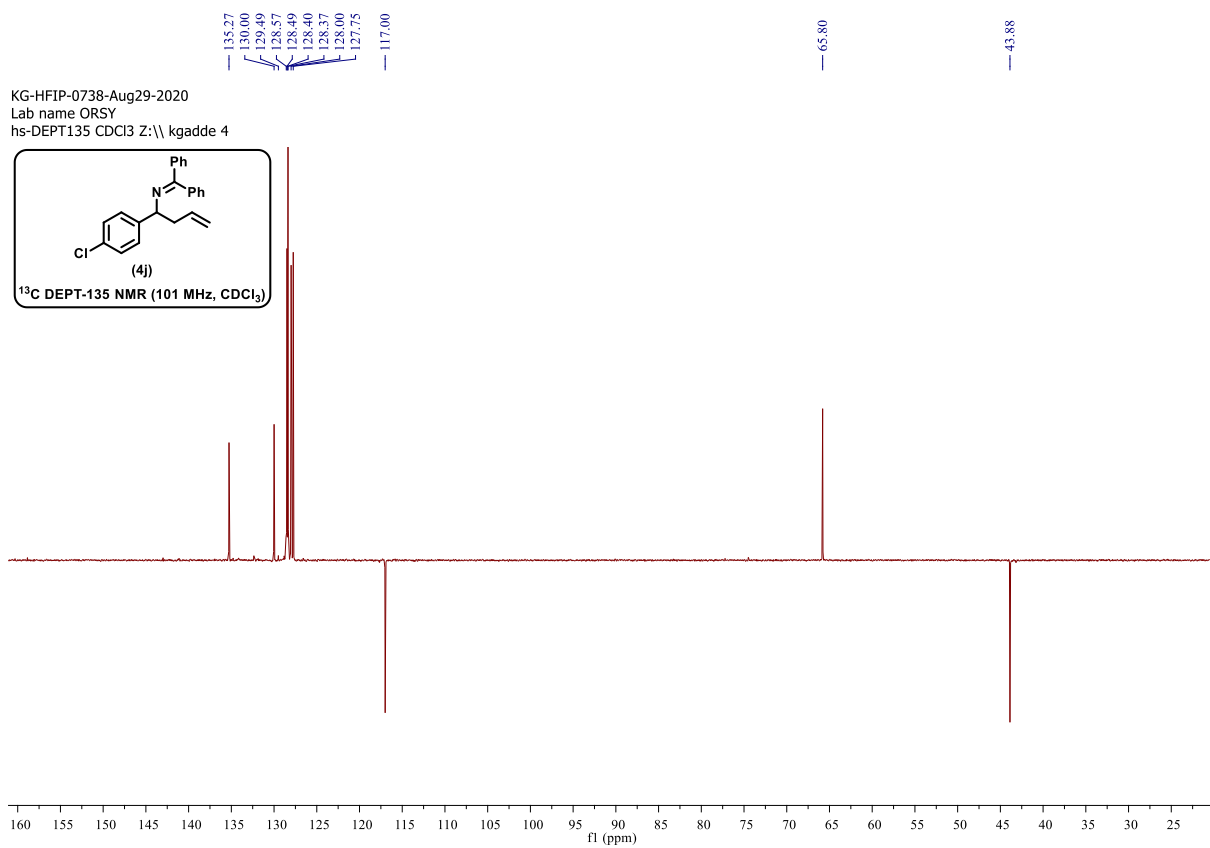


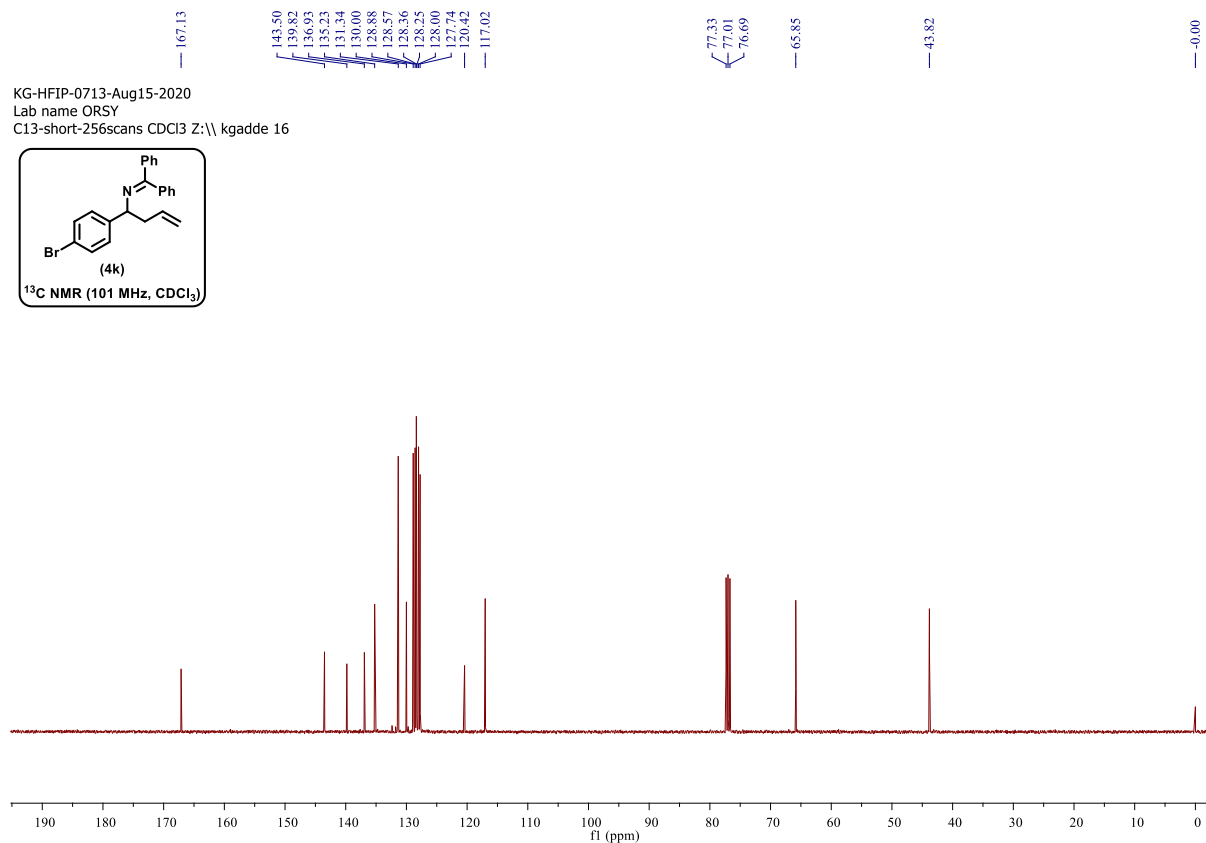
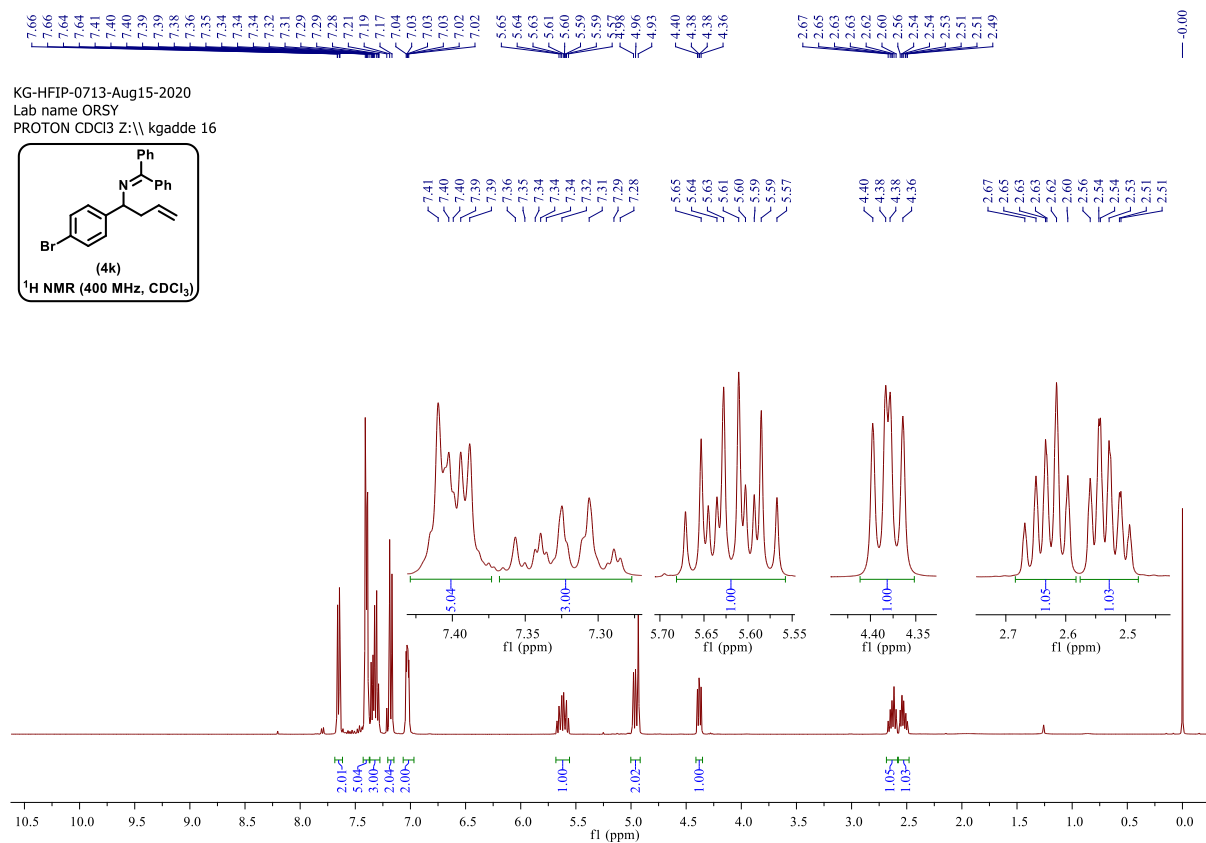
KG-HFIP-0738-Aug29-2020
Lab name ORSY
PROTON CDCl3 Z:\\ kgadde 4

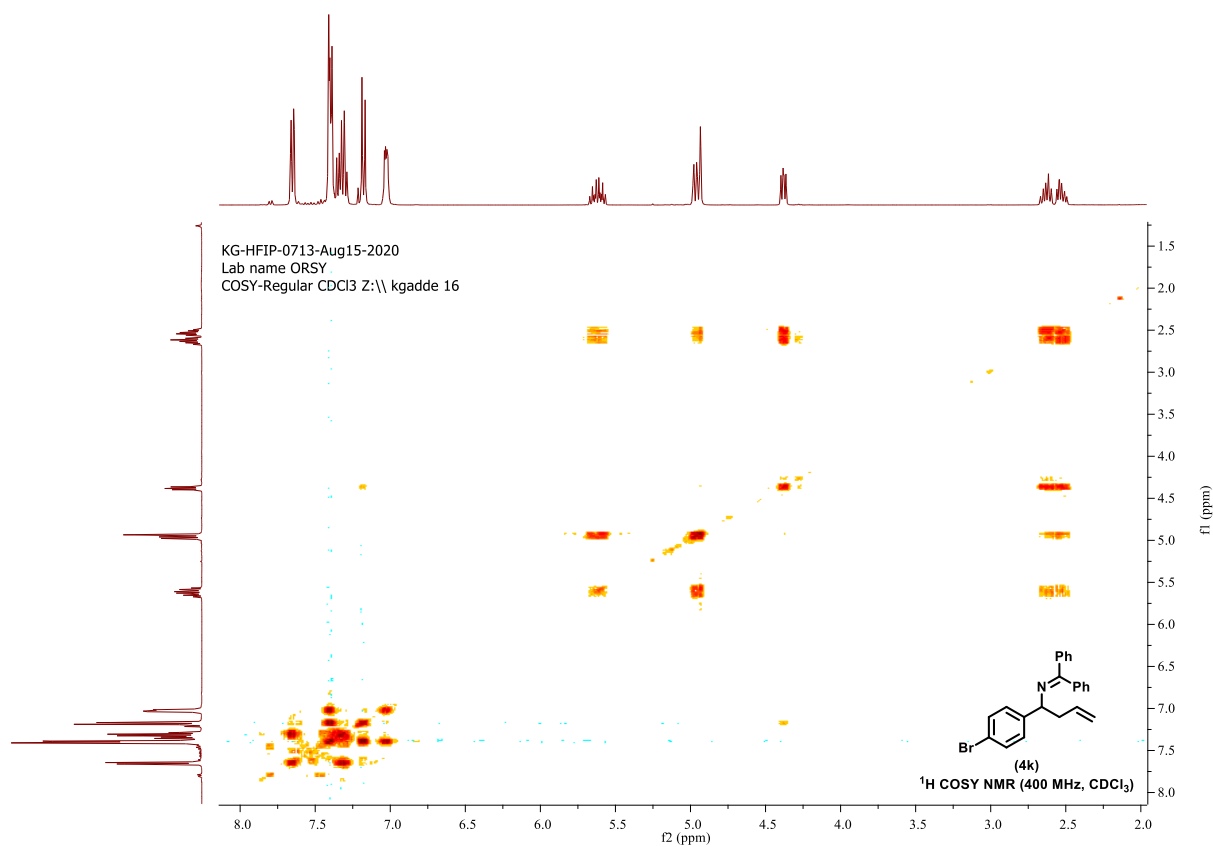
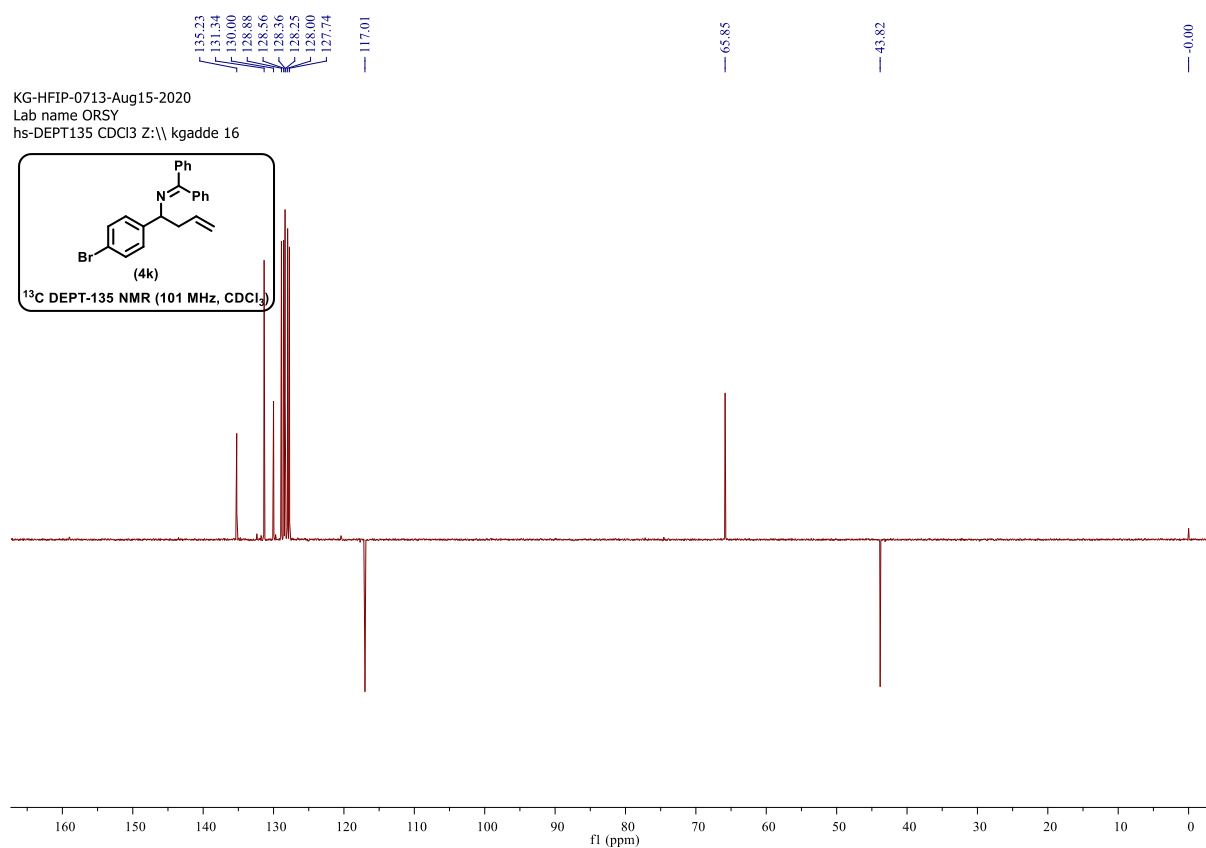


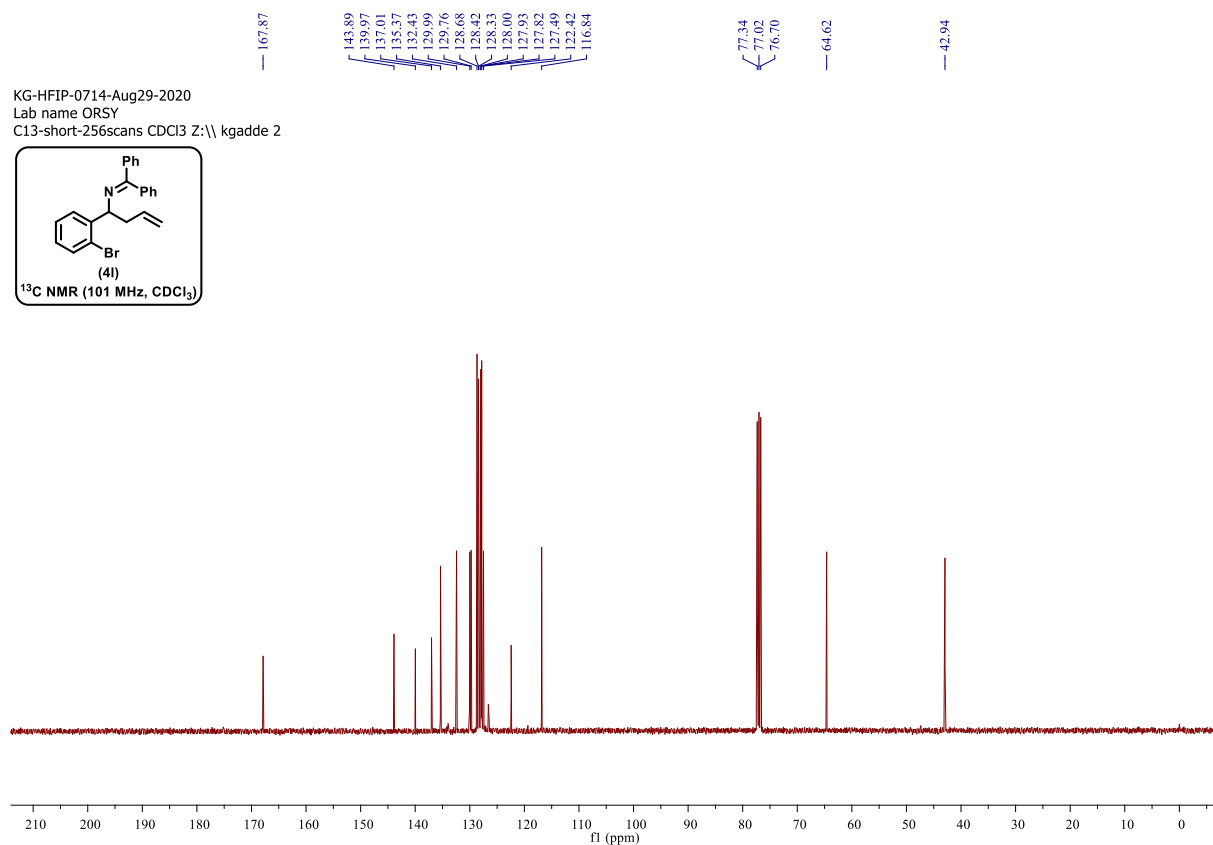
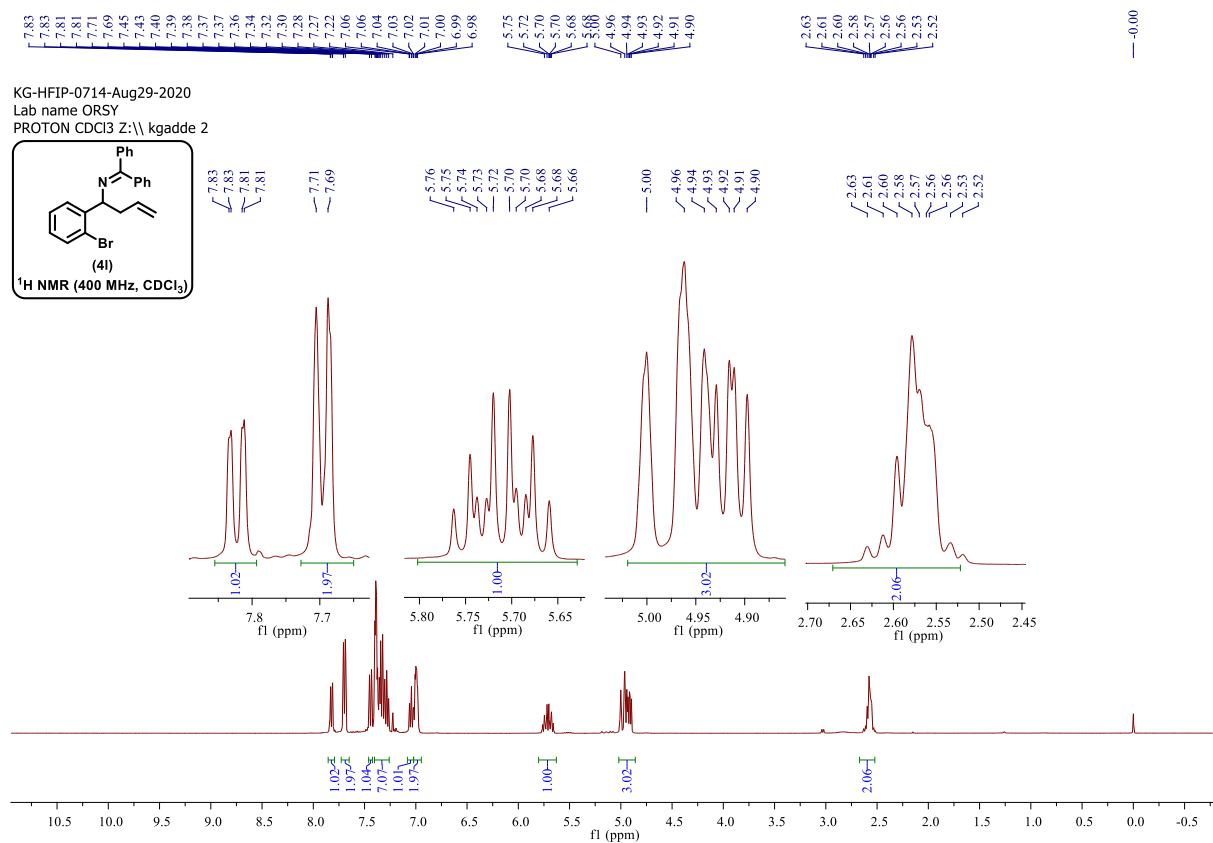
KG-HFIP-0738-Aug29-2020
Lab name ORSY
C13-short-256scans CDCl3 Z:\\ kgadde 4

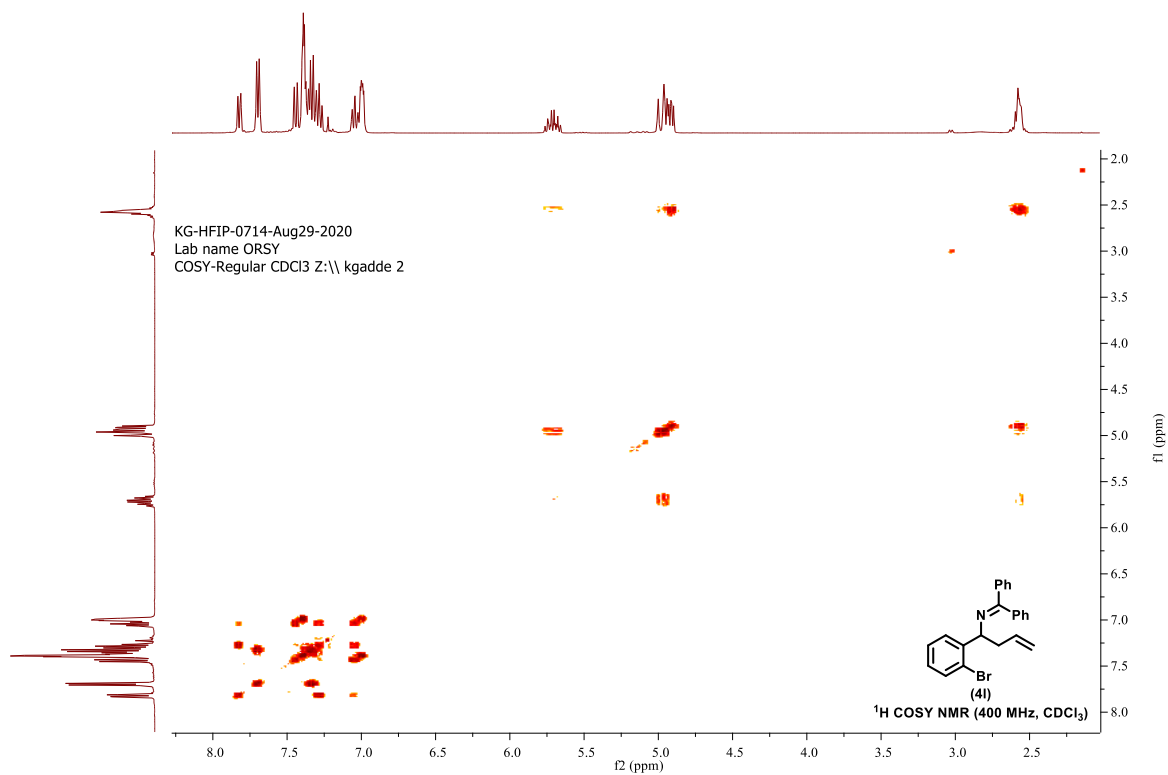
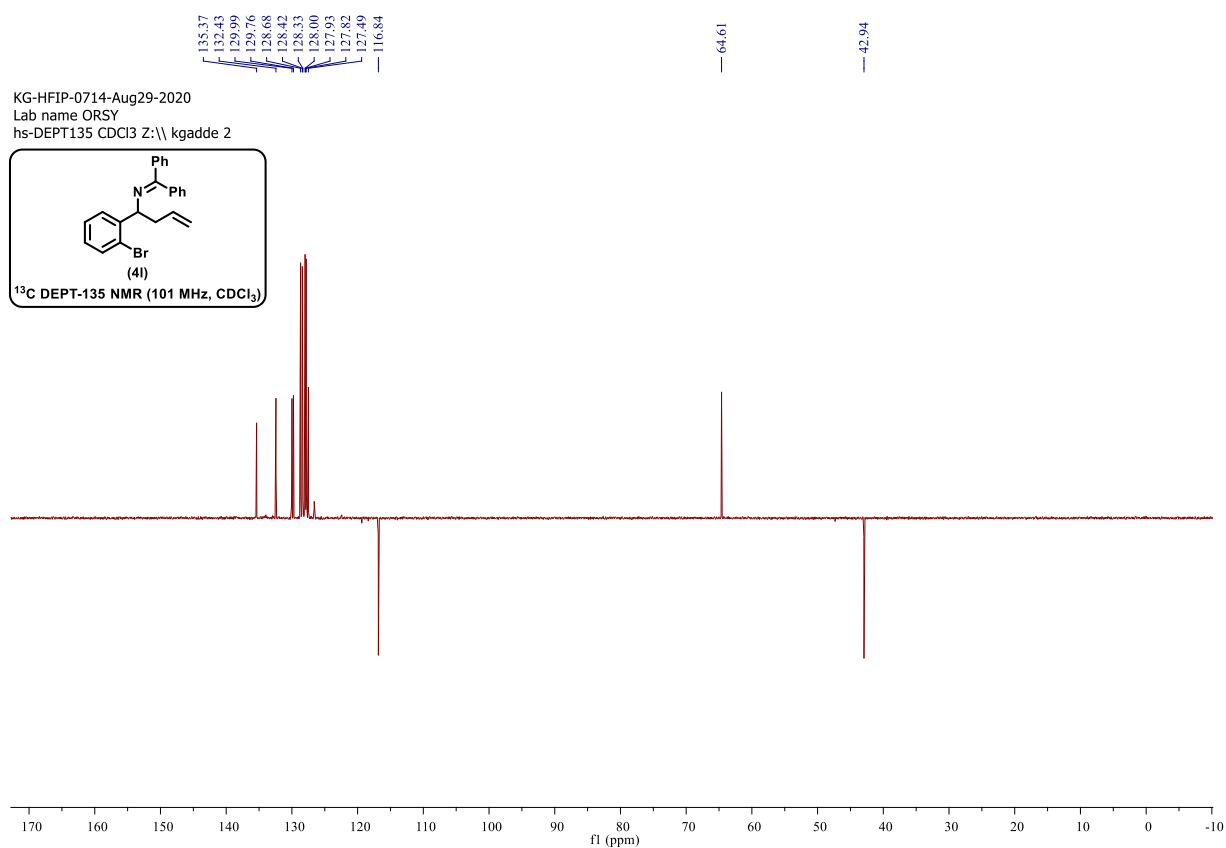


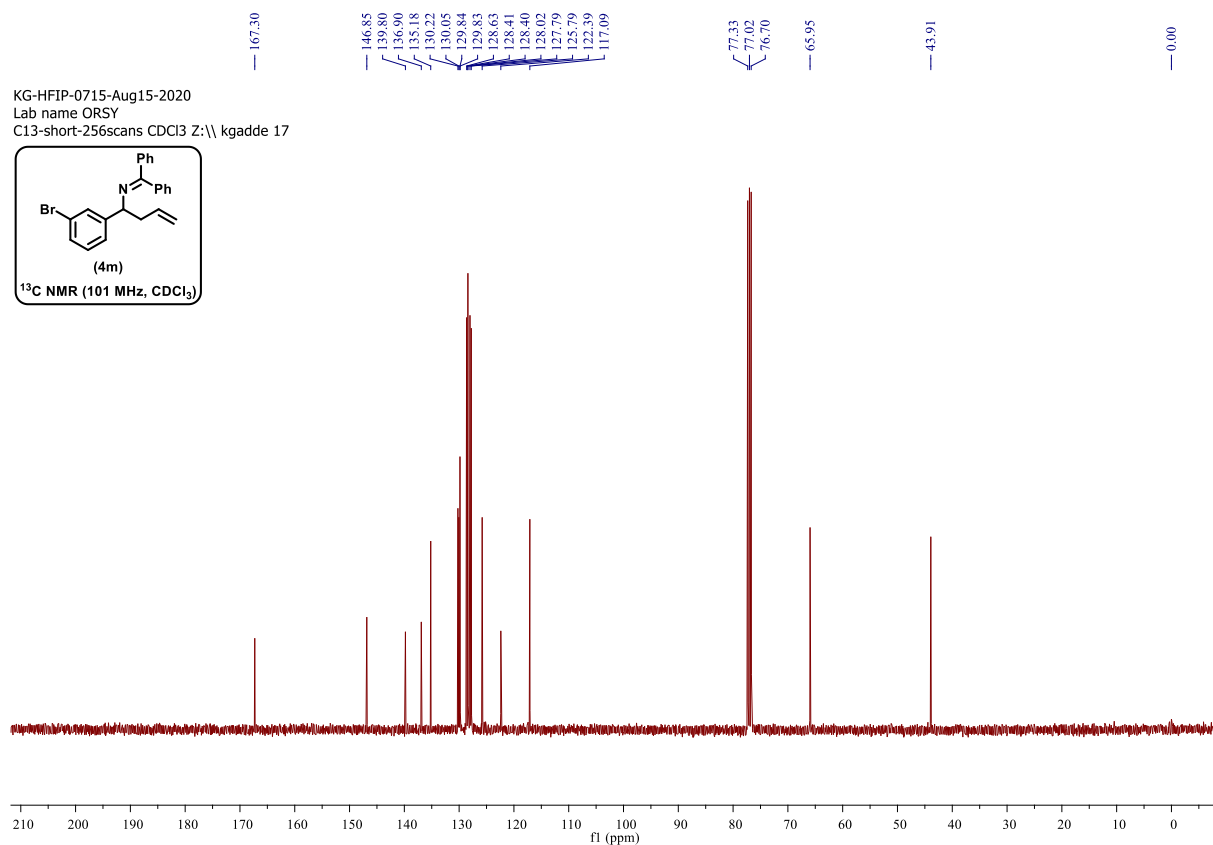
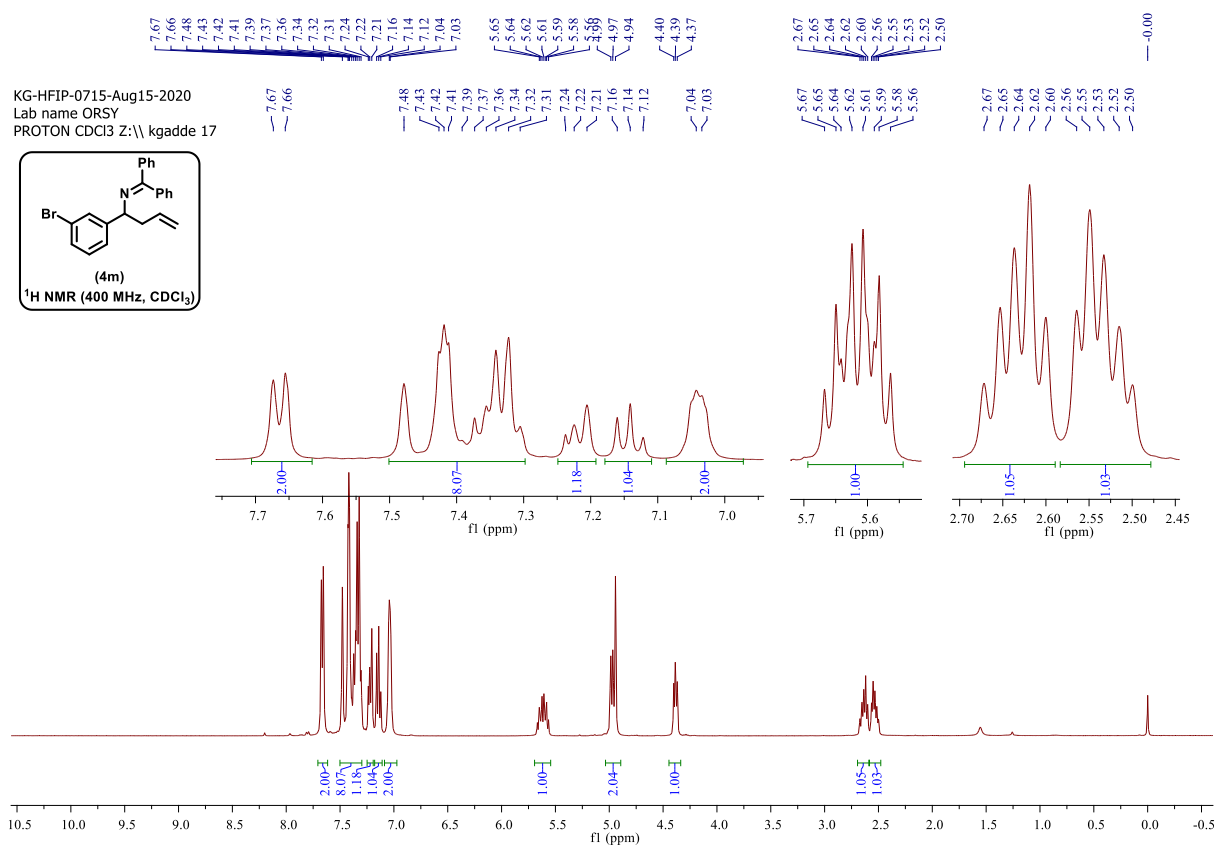


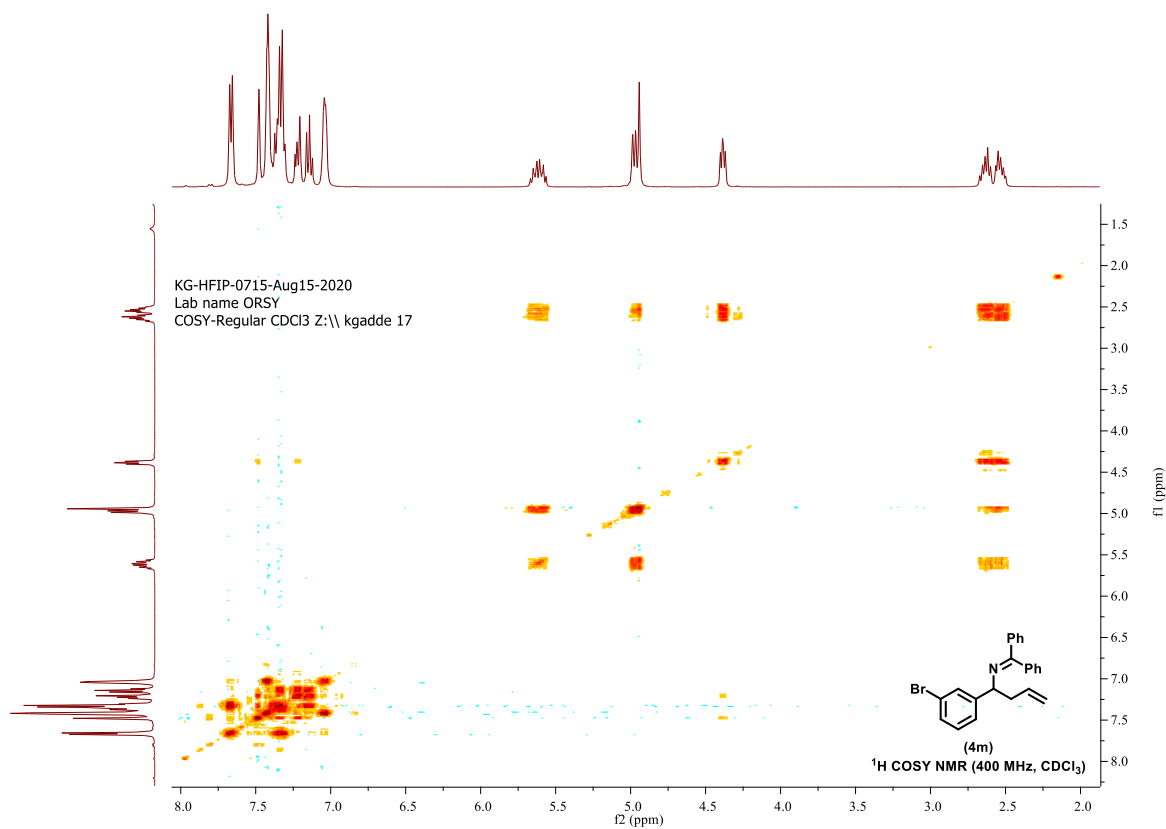
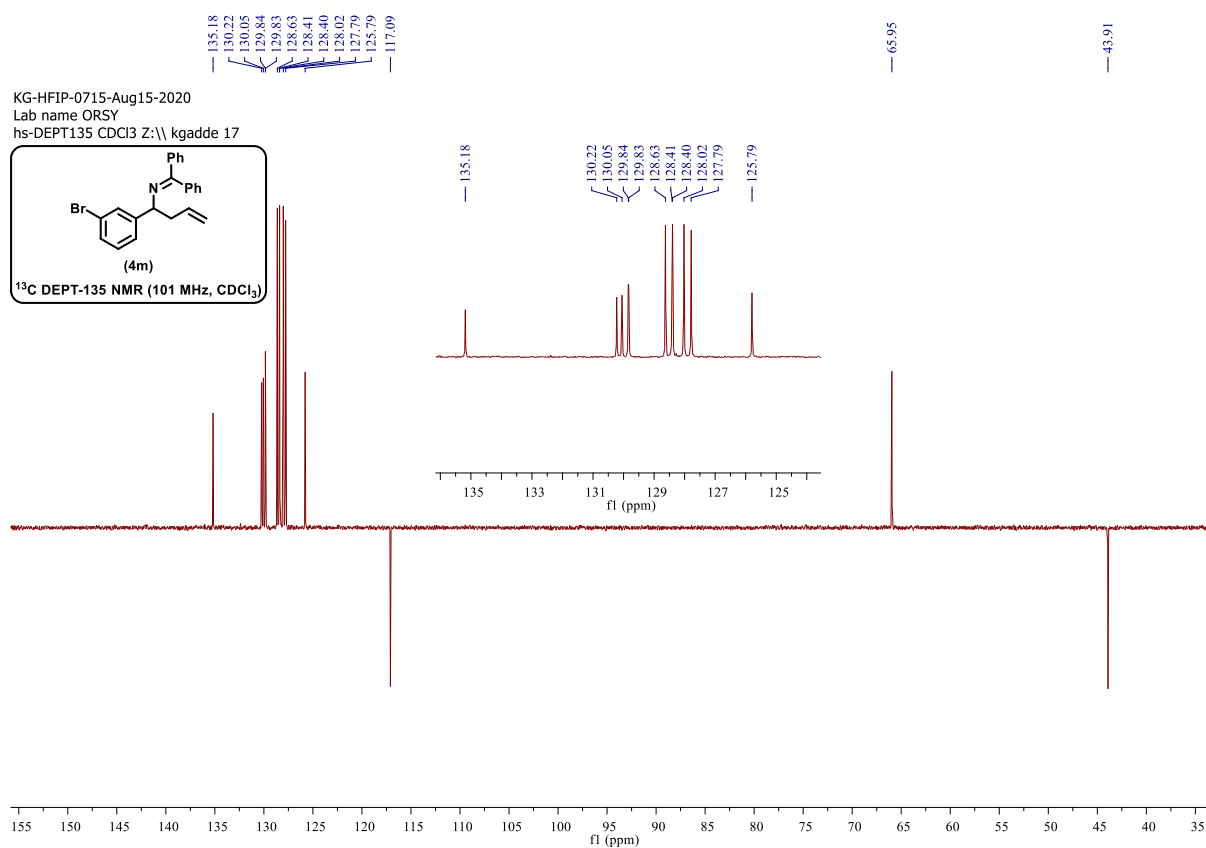




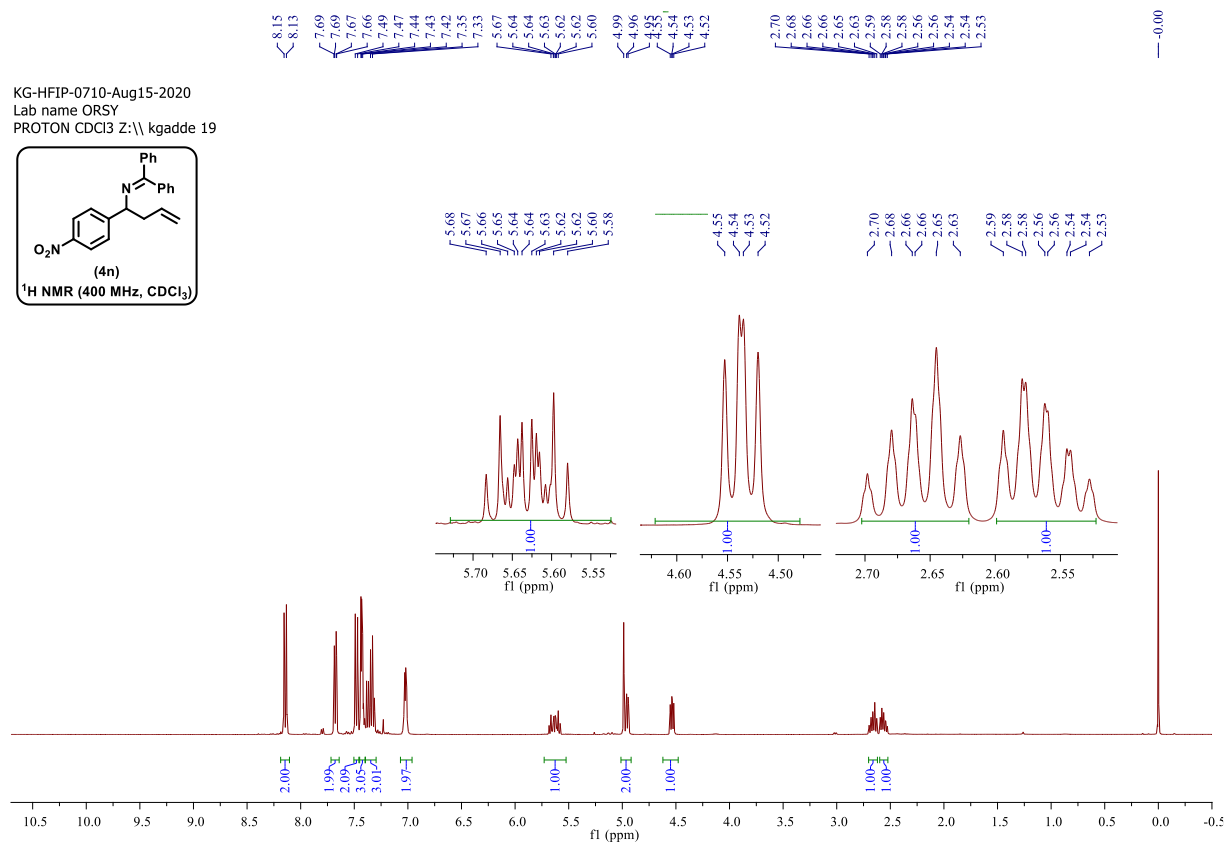
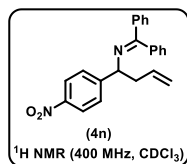




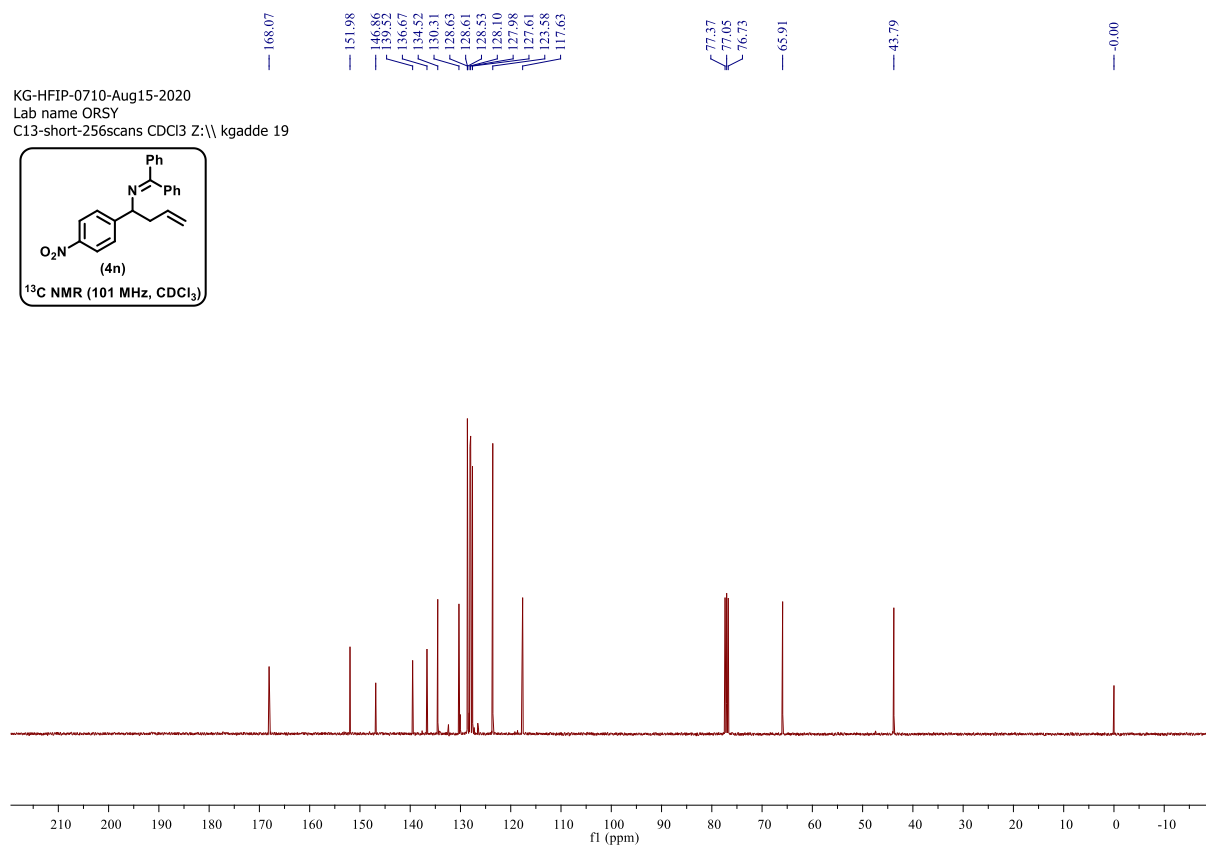
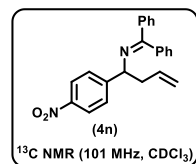




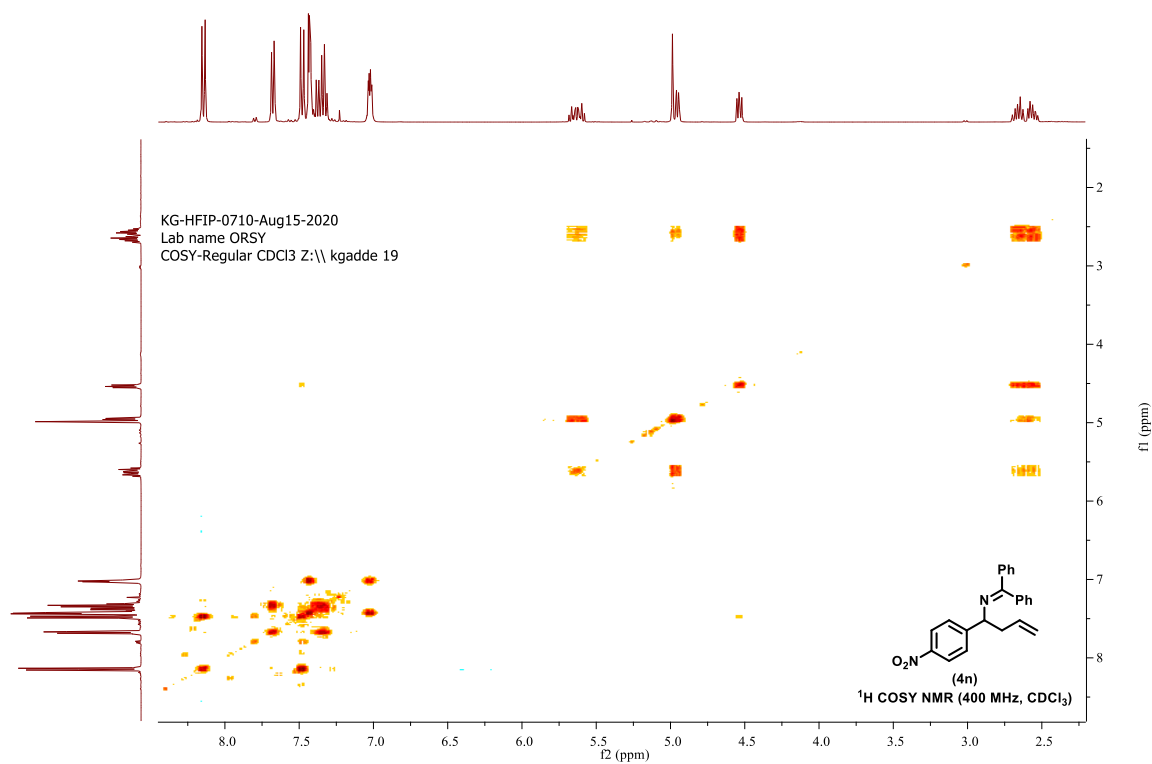
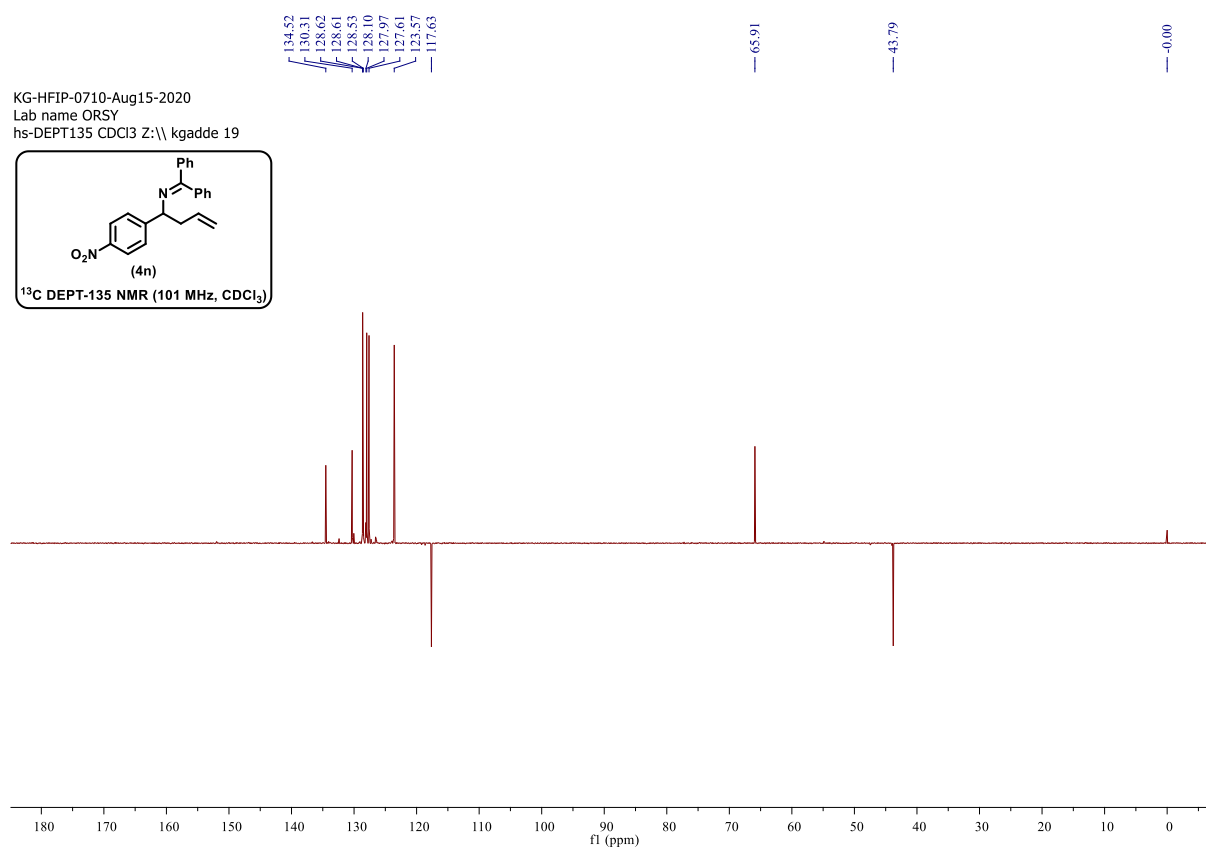
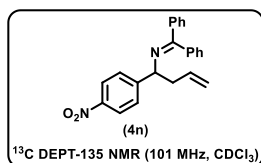
KG-HFIP-0710-Aug15-2020
Lab name ORSY
PROTON CDCl3 Z:\\ kgadde 19



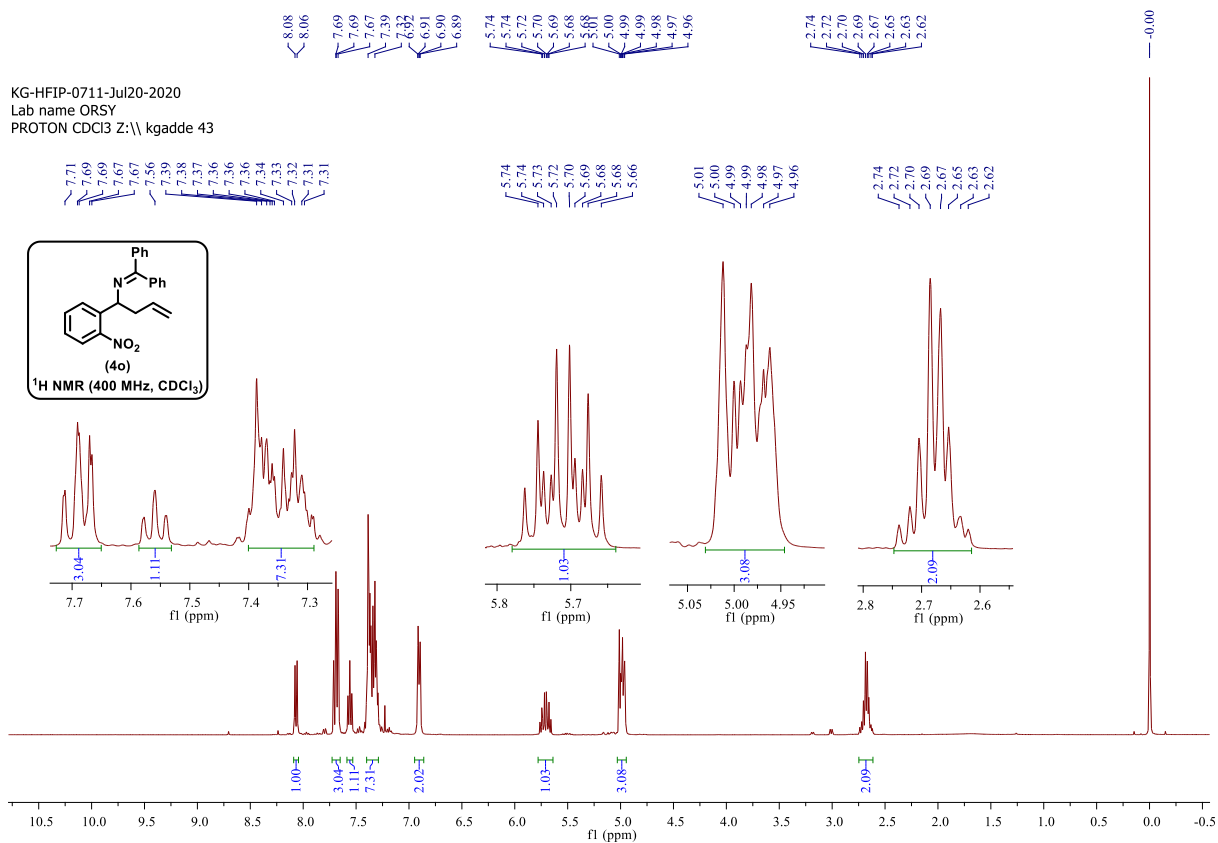
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C13-short-256scans CDCl3 Z:\\ kgadde 19



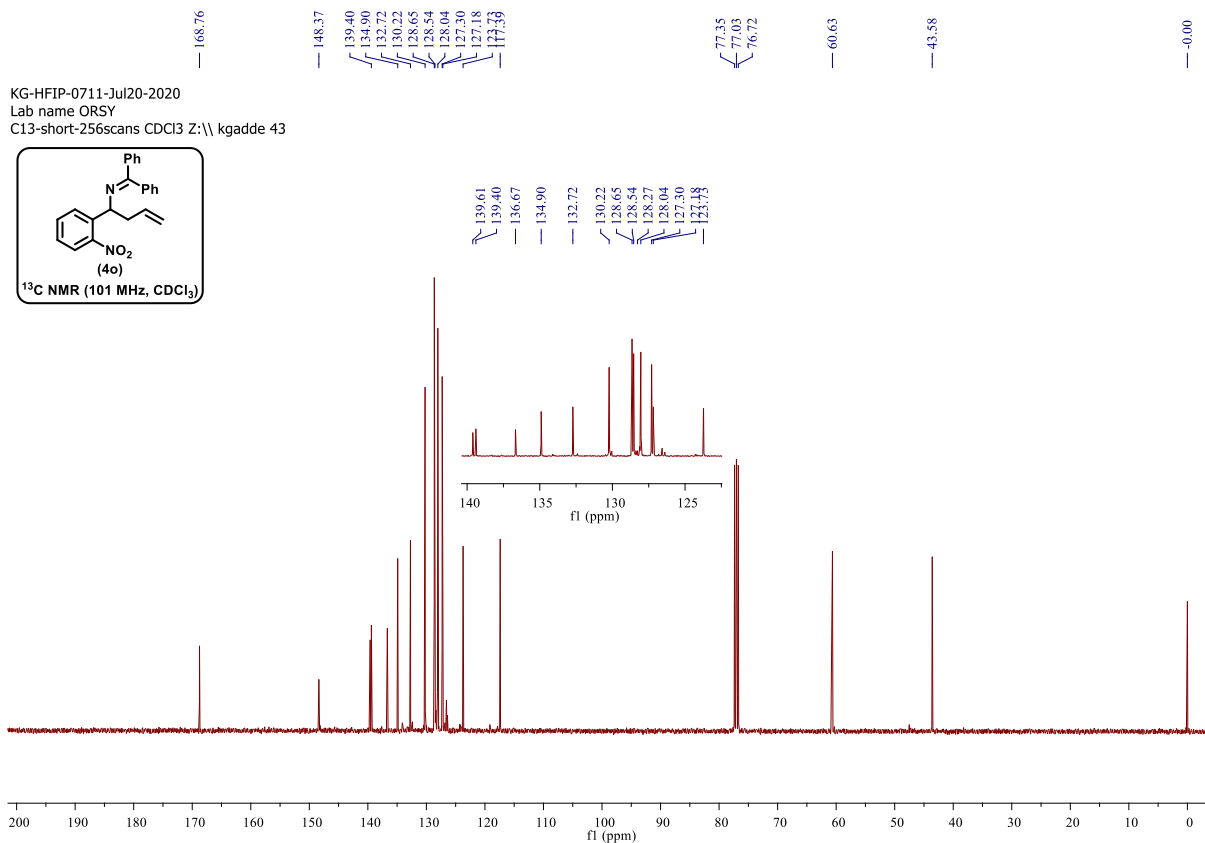
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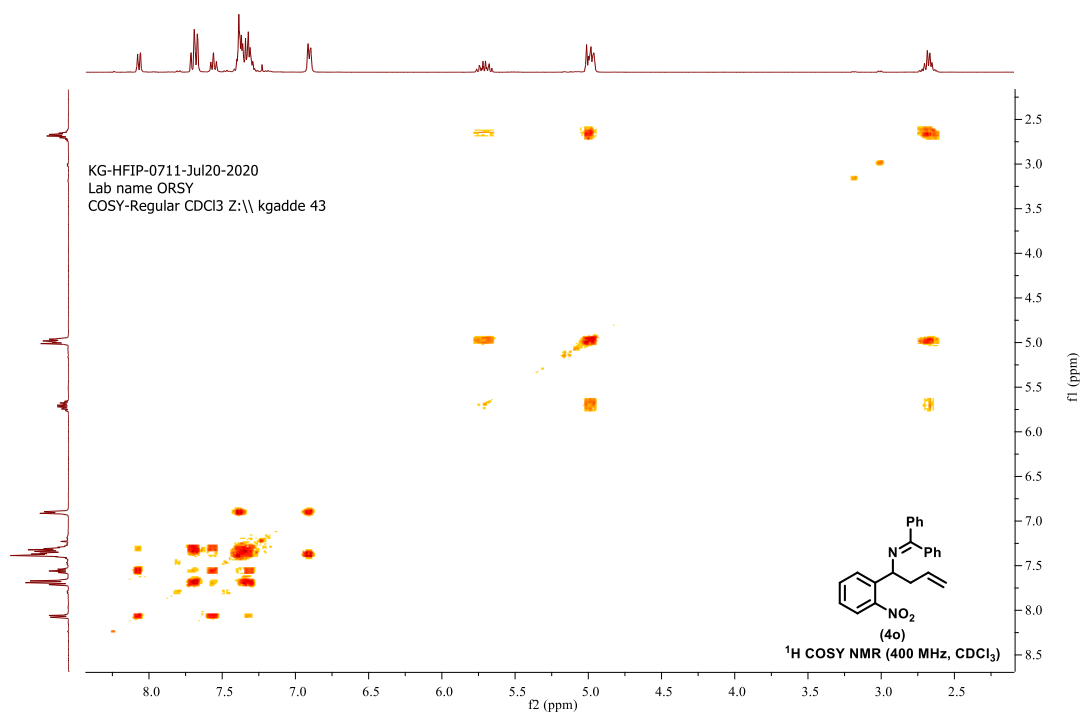
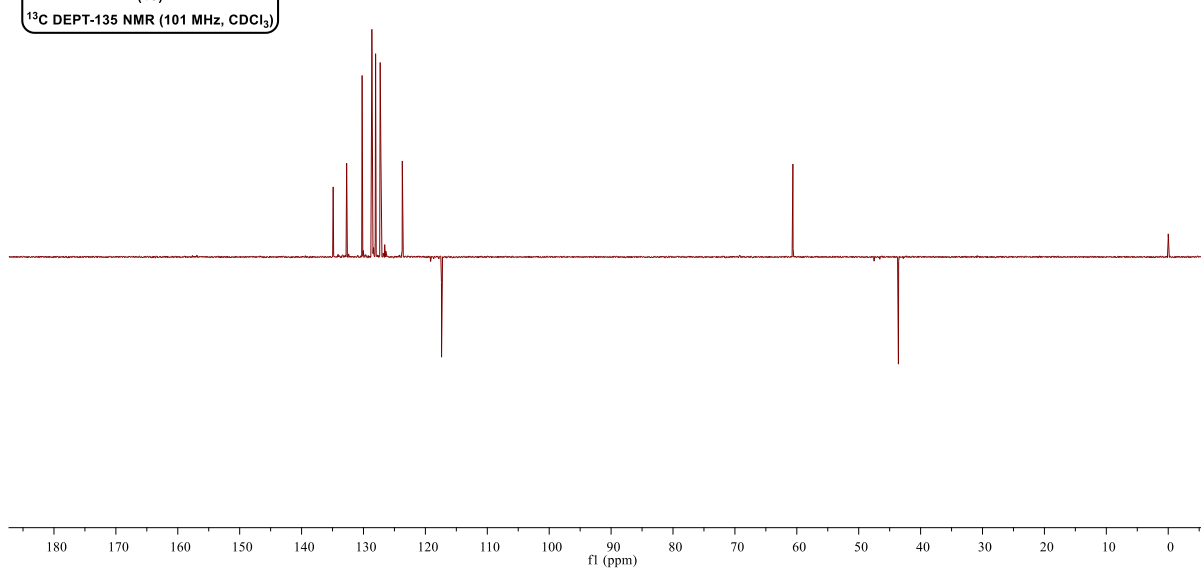
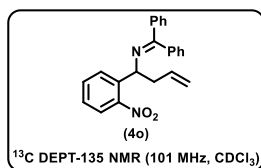
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Lab name ORSY
PROTON CDCl3 Z:\ kgadde 43

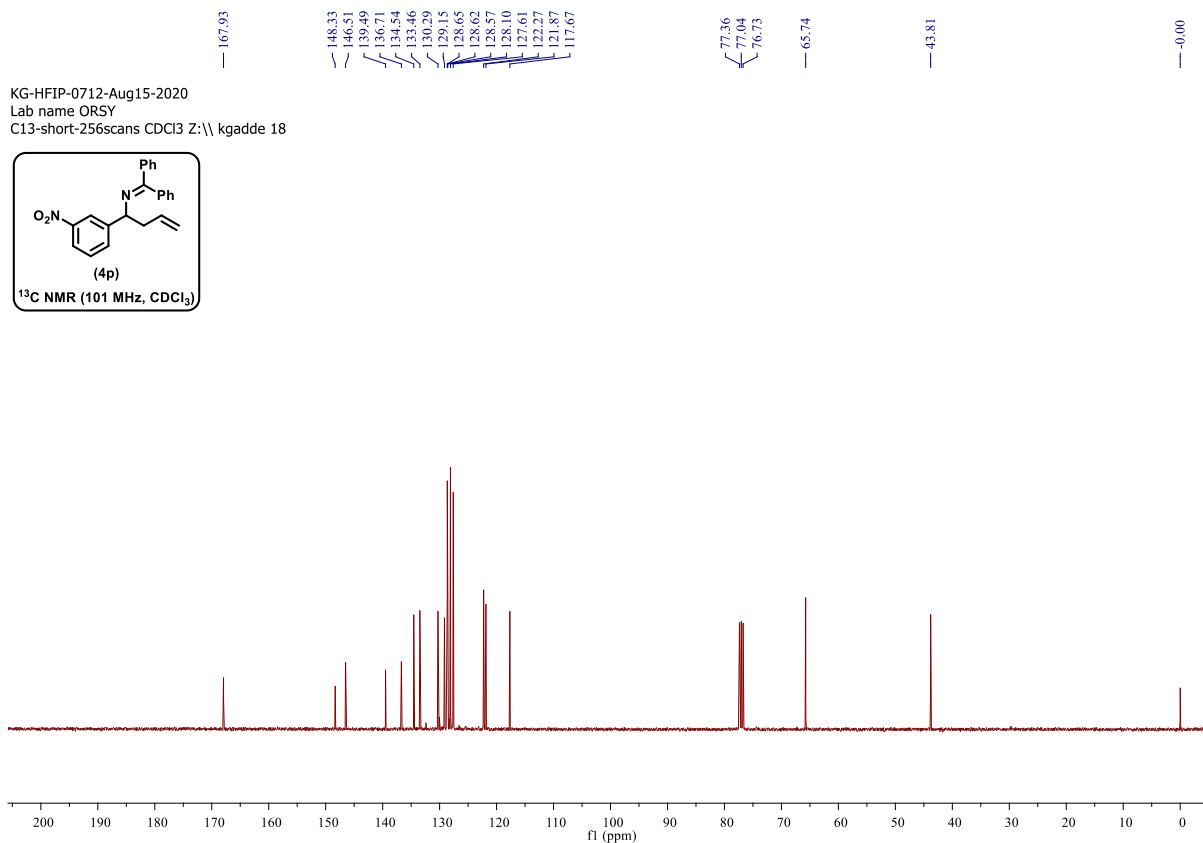
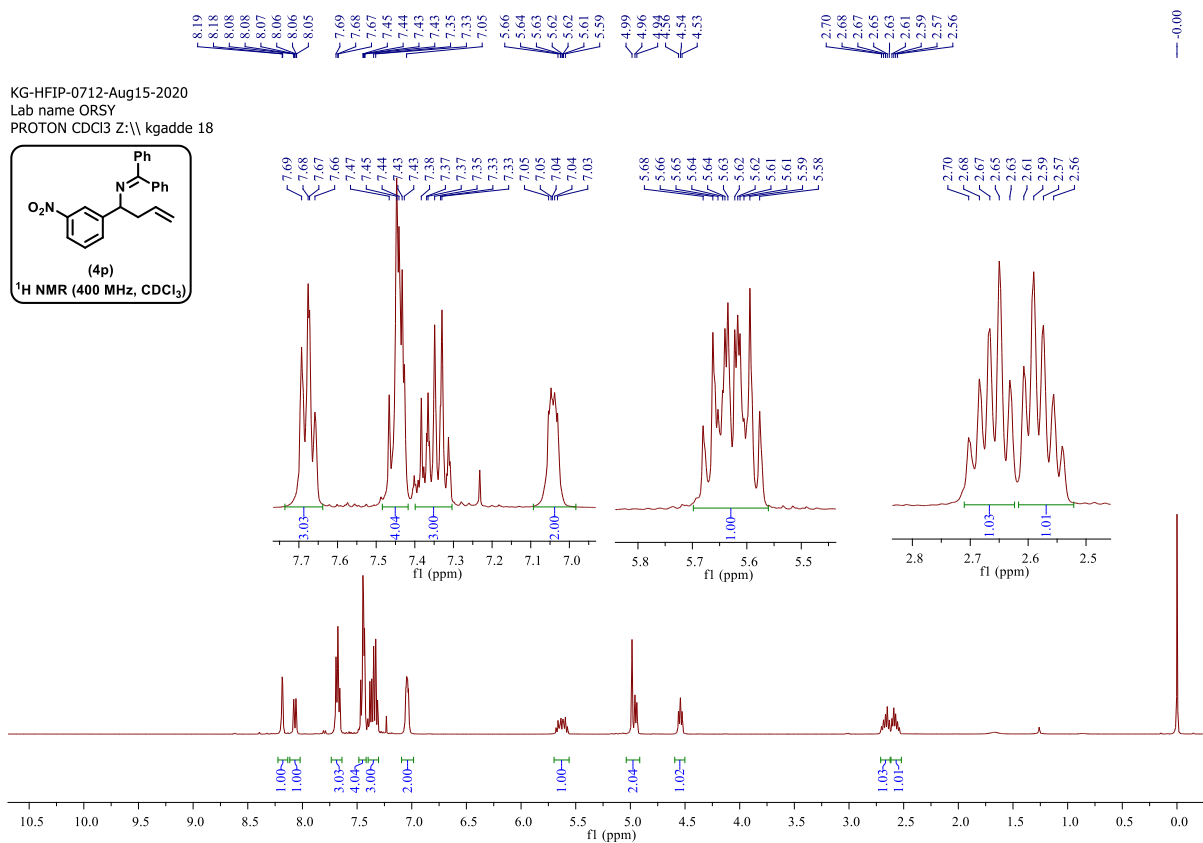


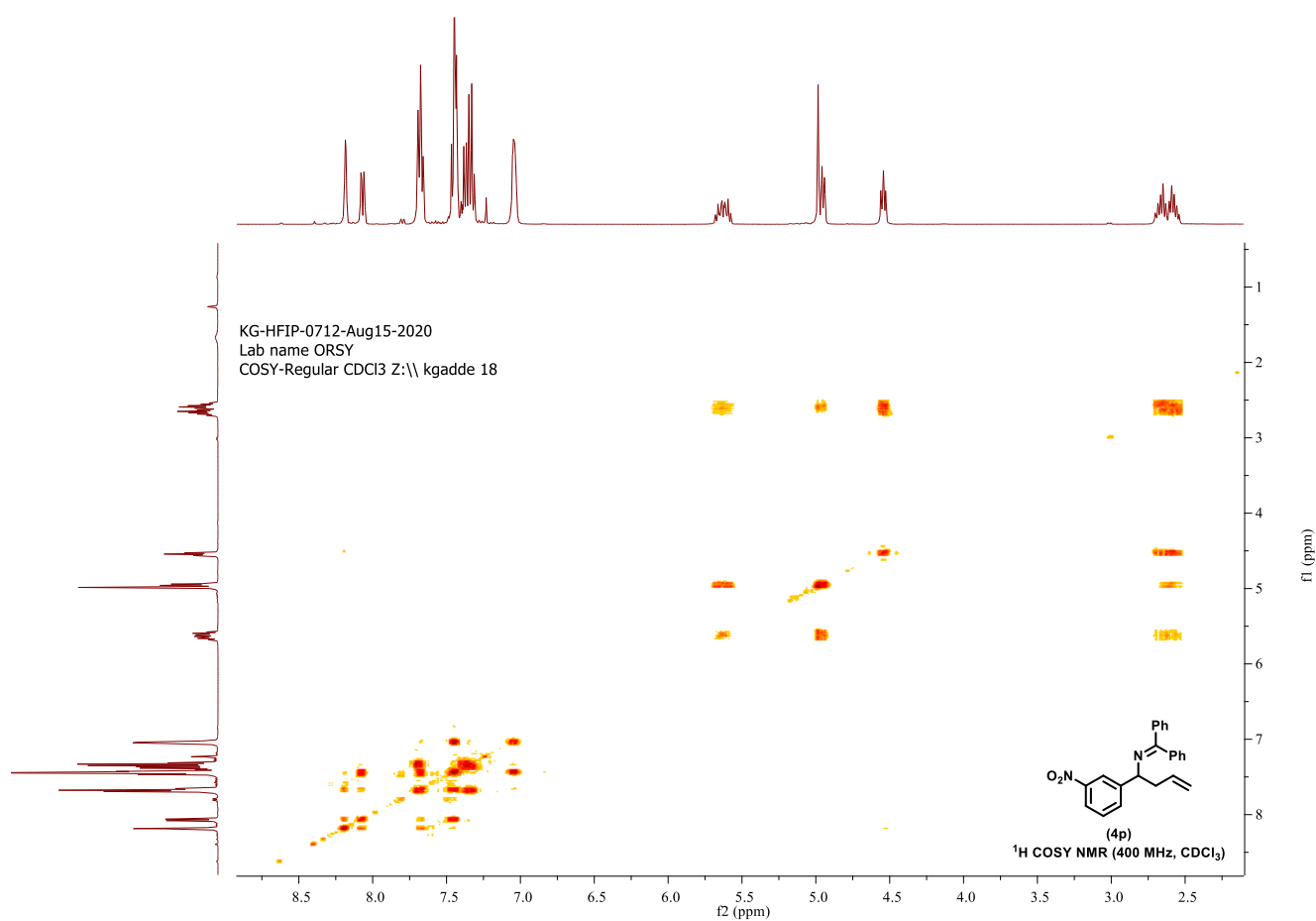
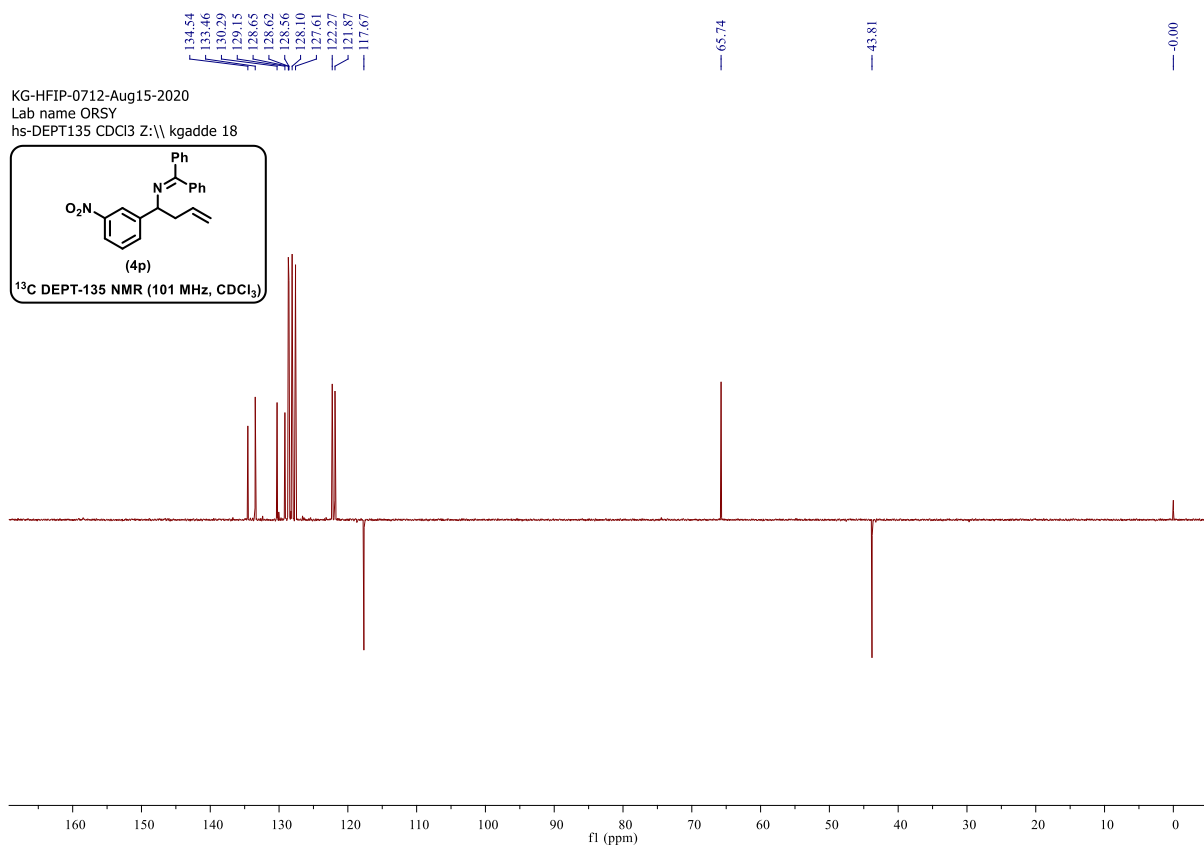
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Lab name ORSY
C13-short-256scans CDCl3 Z:\ kgadde 43



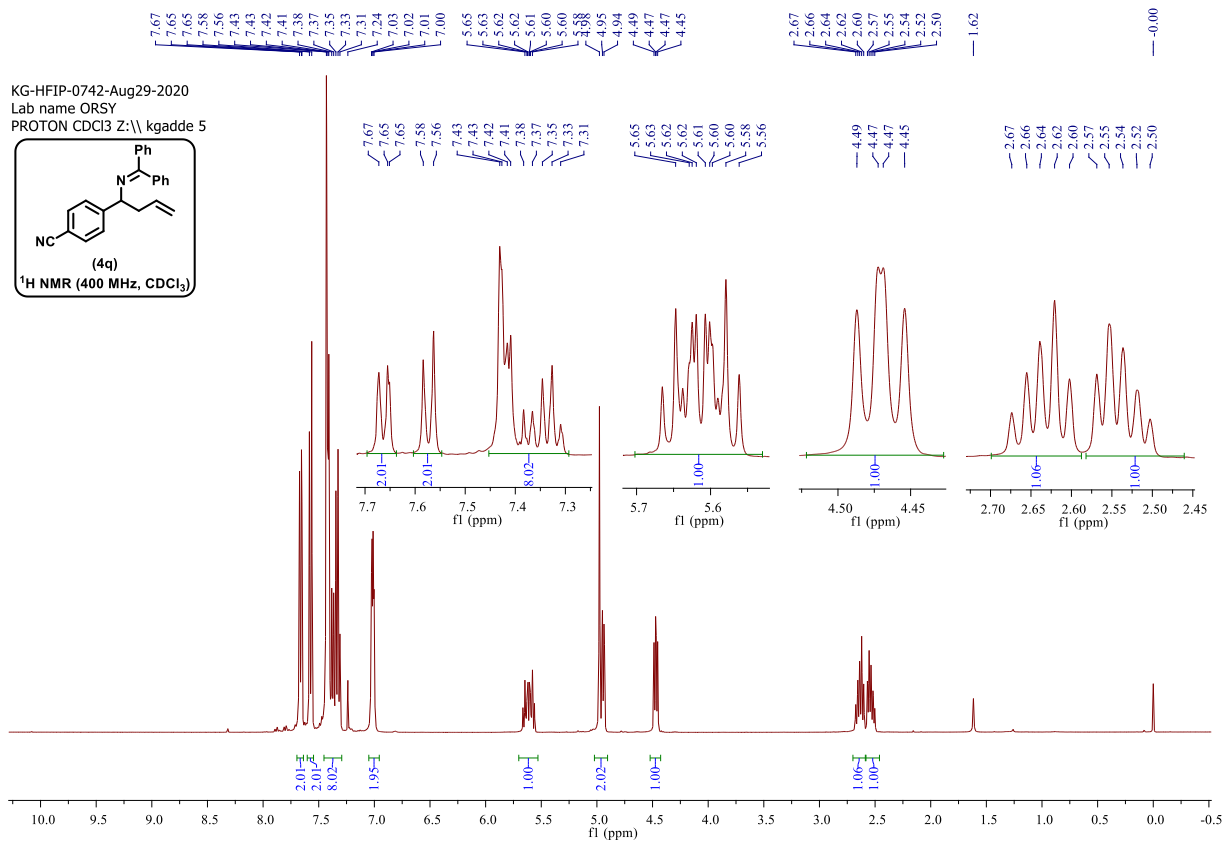
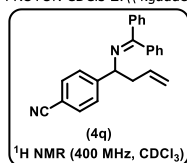
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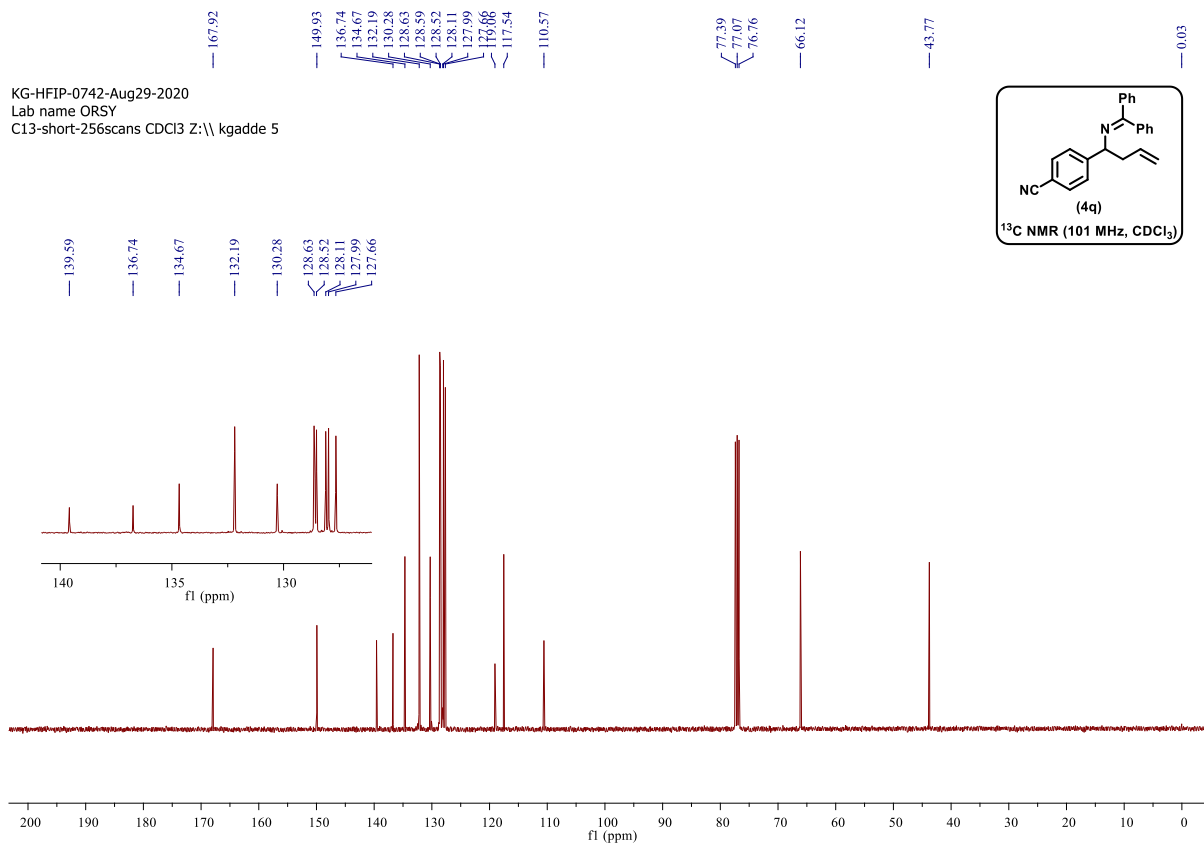
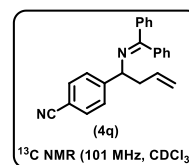




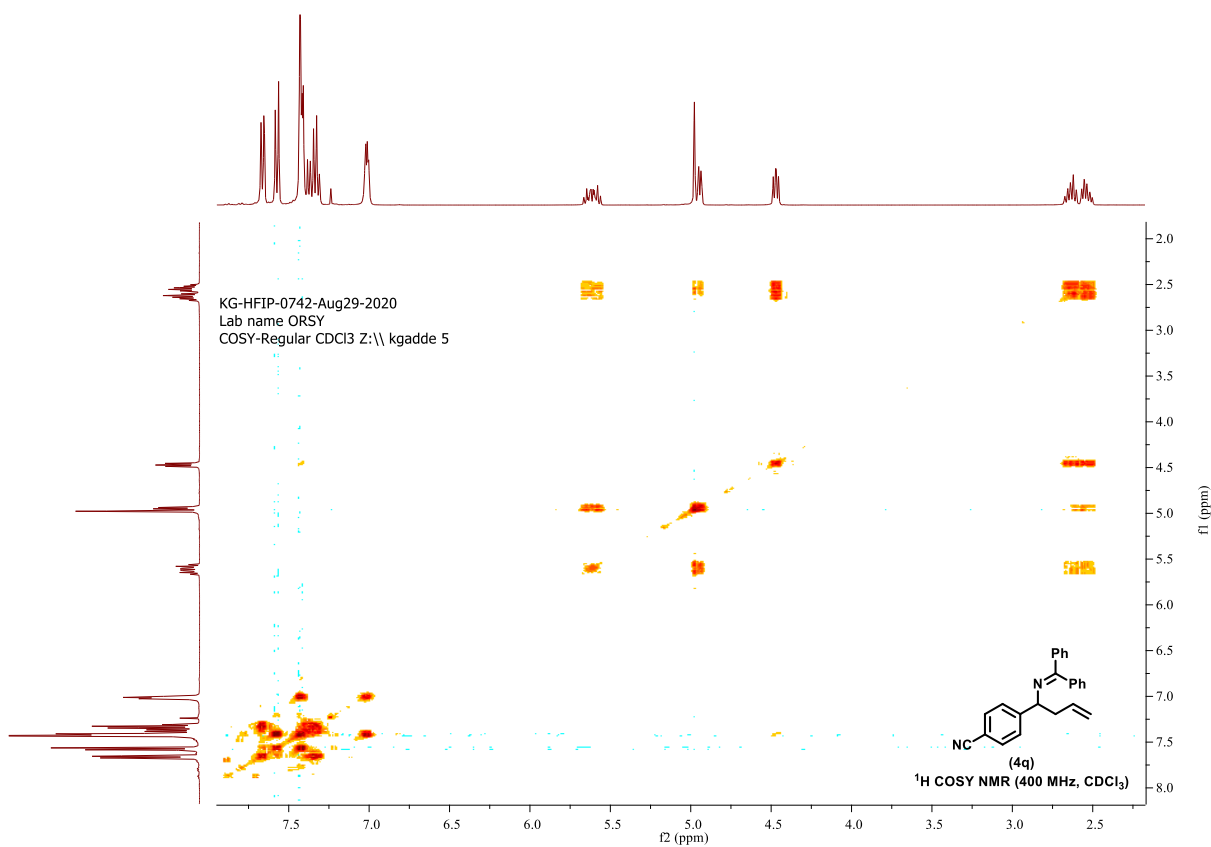
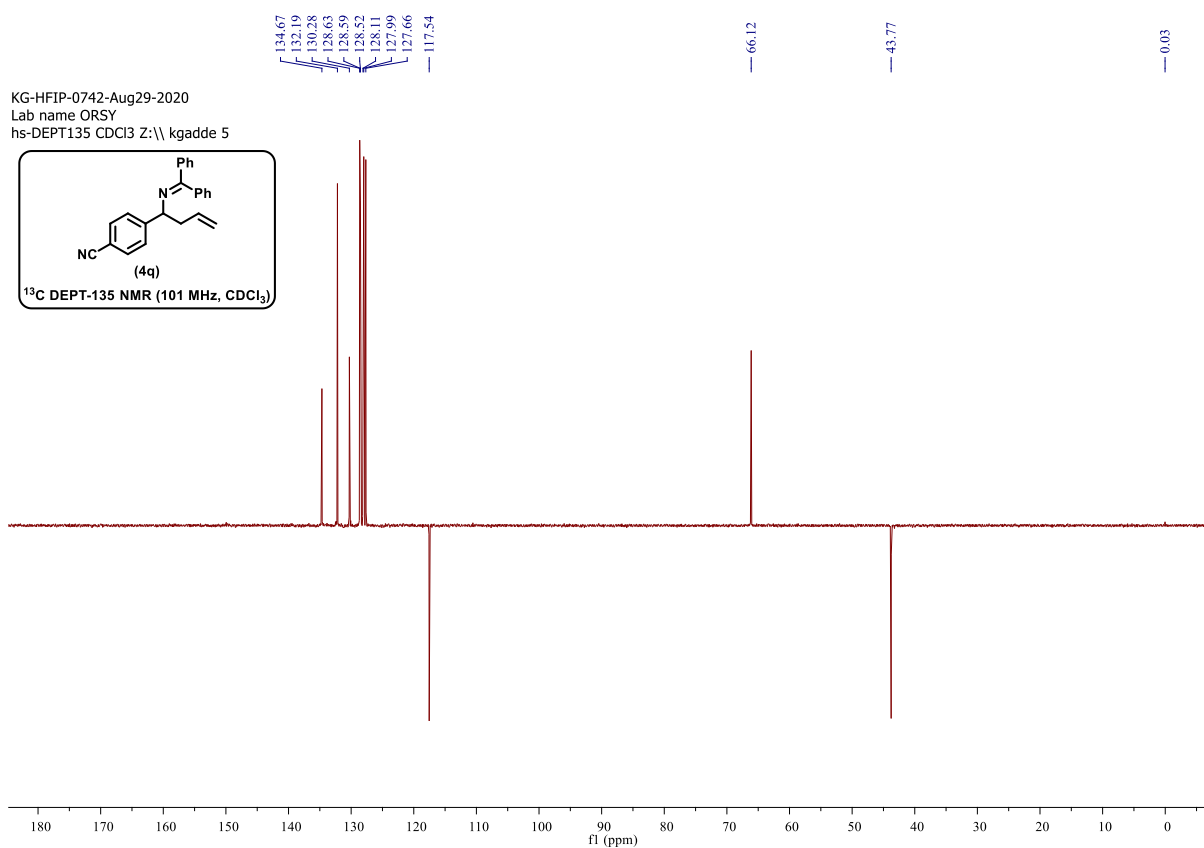
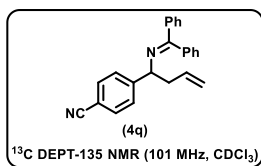
KG-HFIP-0742-Aug29-2020
Lab name ORSY
PROTON CDCl3 Z:\\ kgadde 5




KG-HFIP-0742-Aug29-2020
Lab name ORSY
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KG-HFIP-0742-Aug29-2020
 Lab name ORSY
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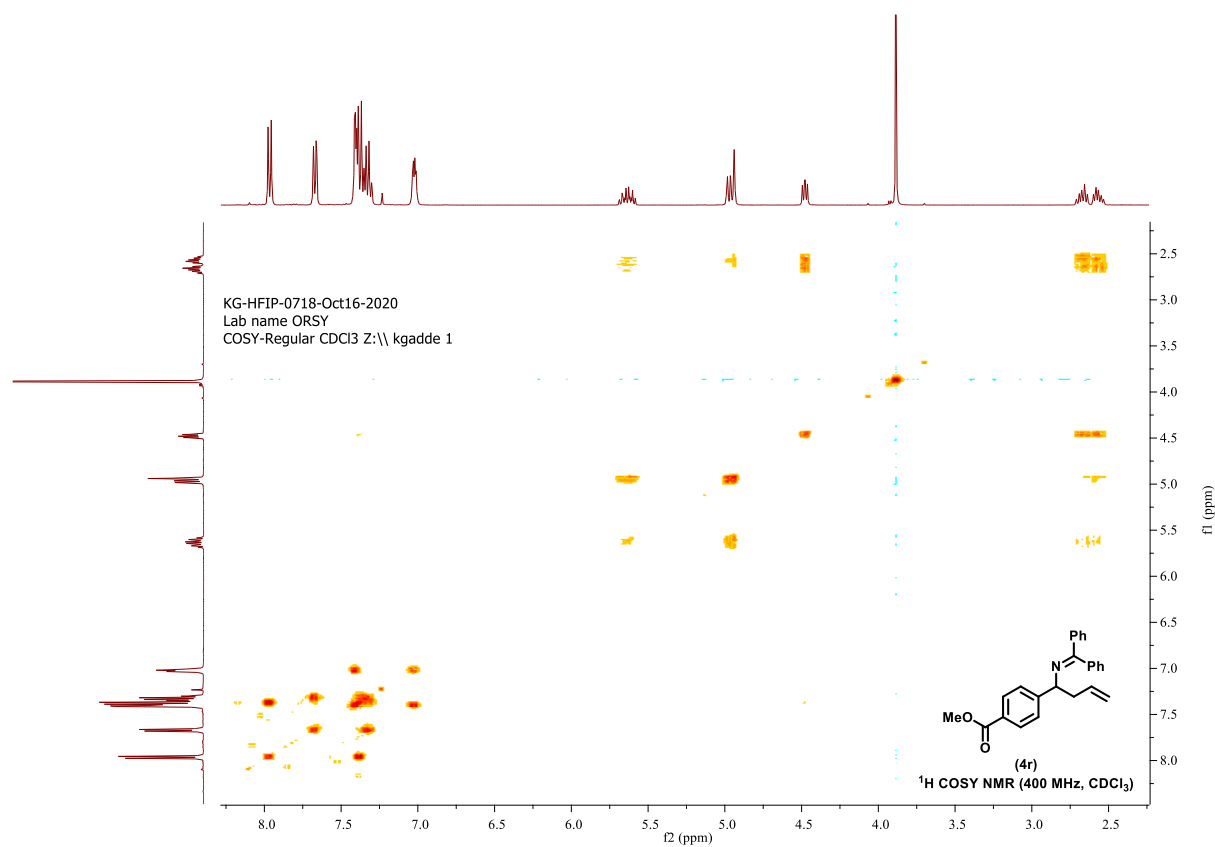
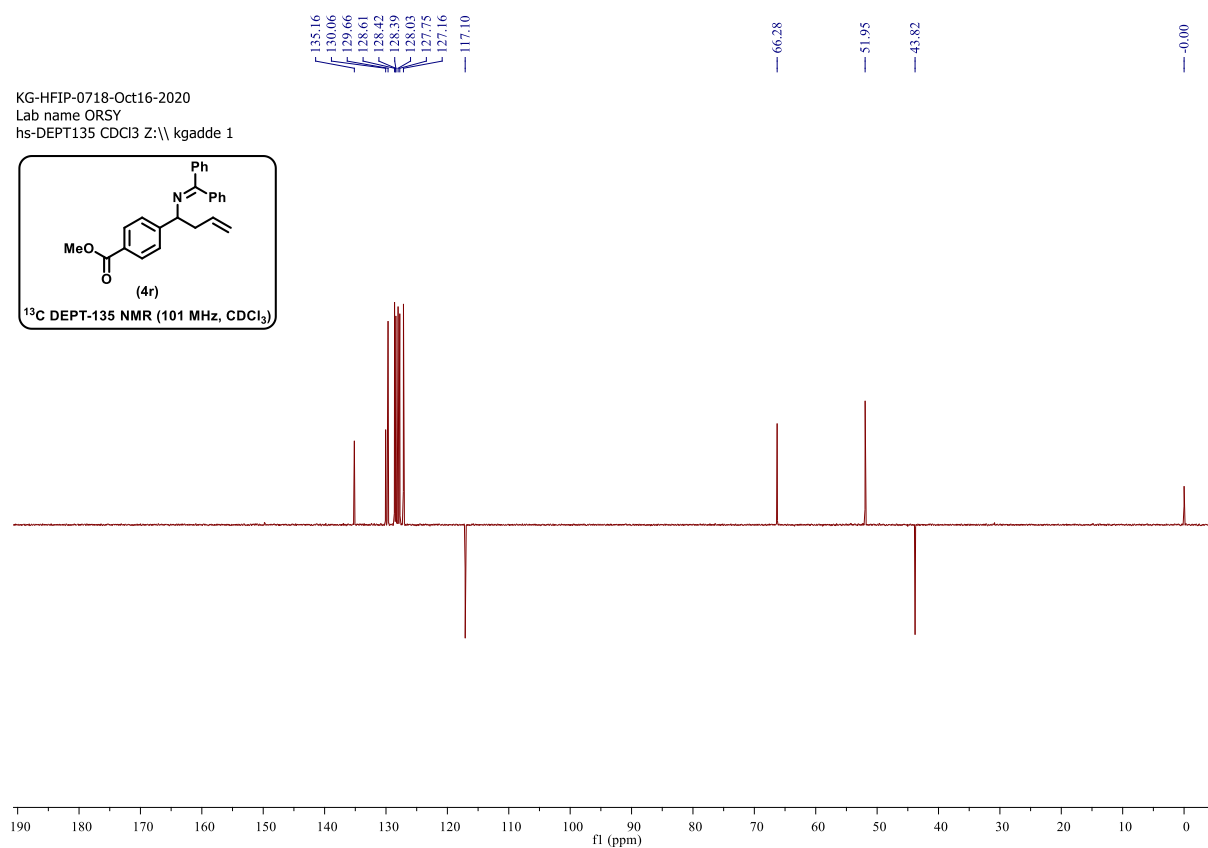
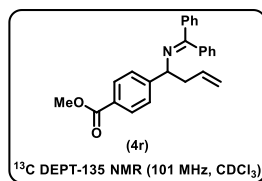

(4r)
 $^1\text{H NMR}$ (400 MHz, CDCl_3)

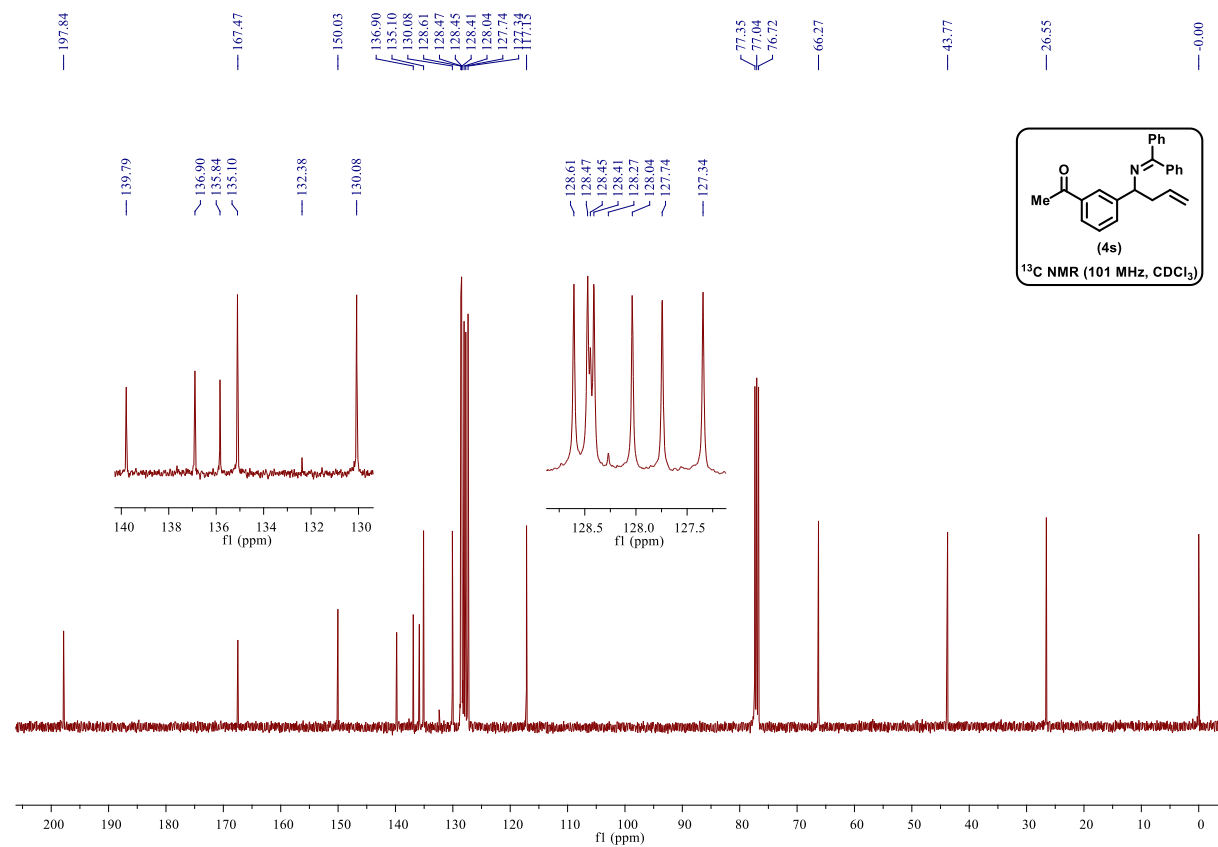
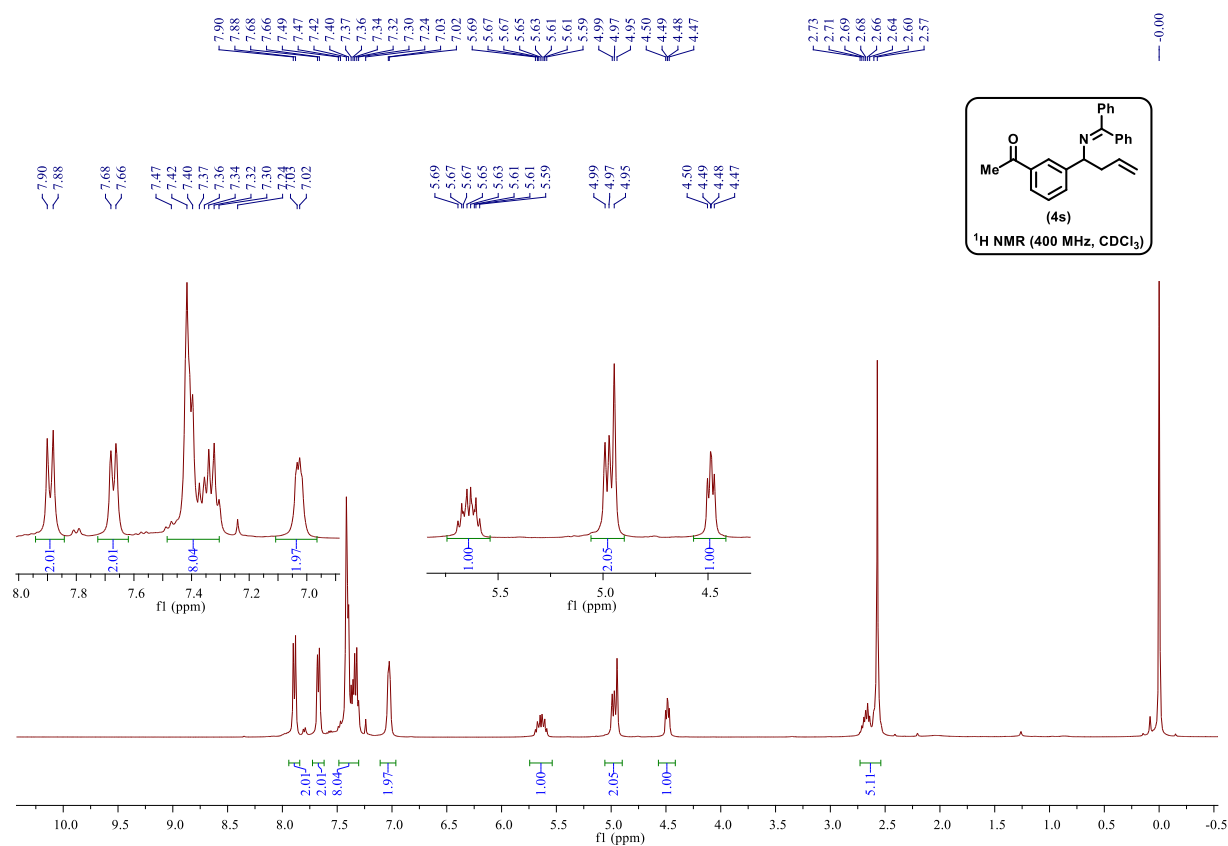


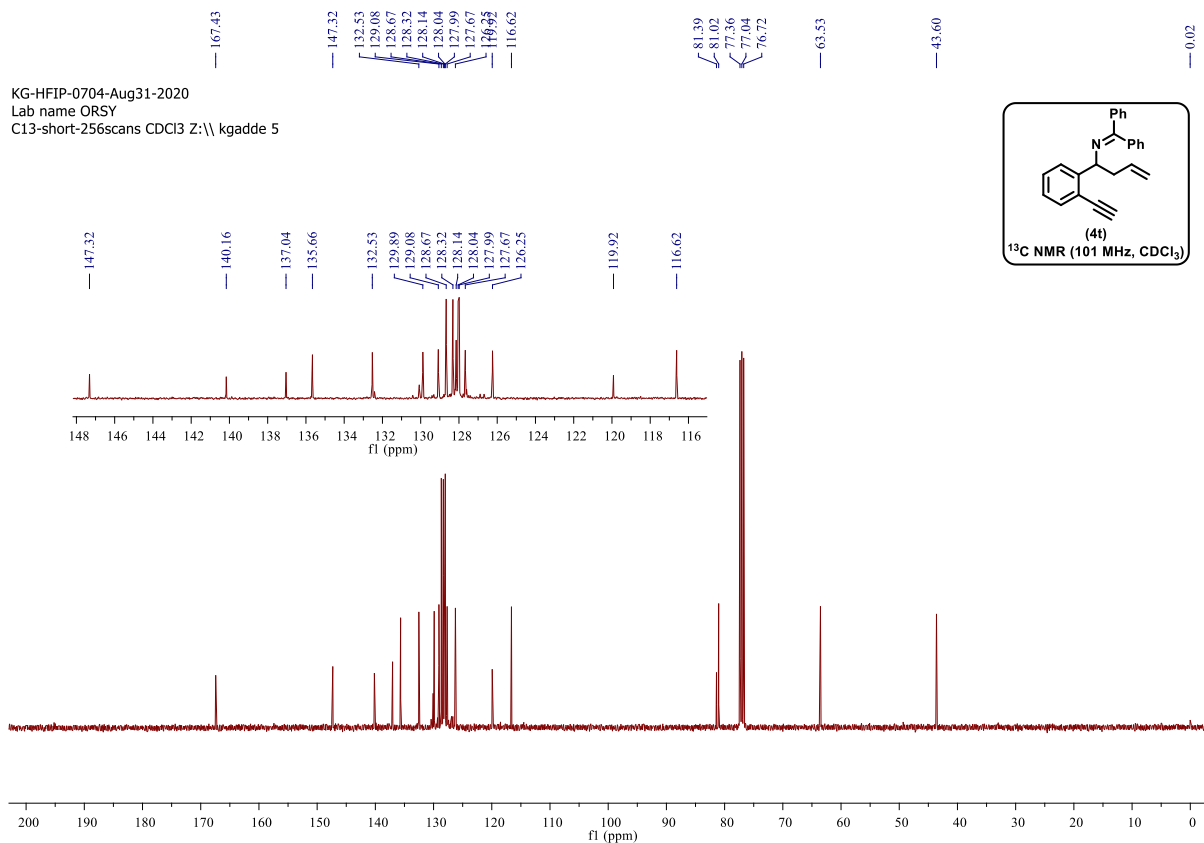
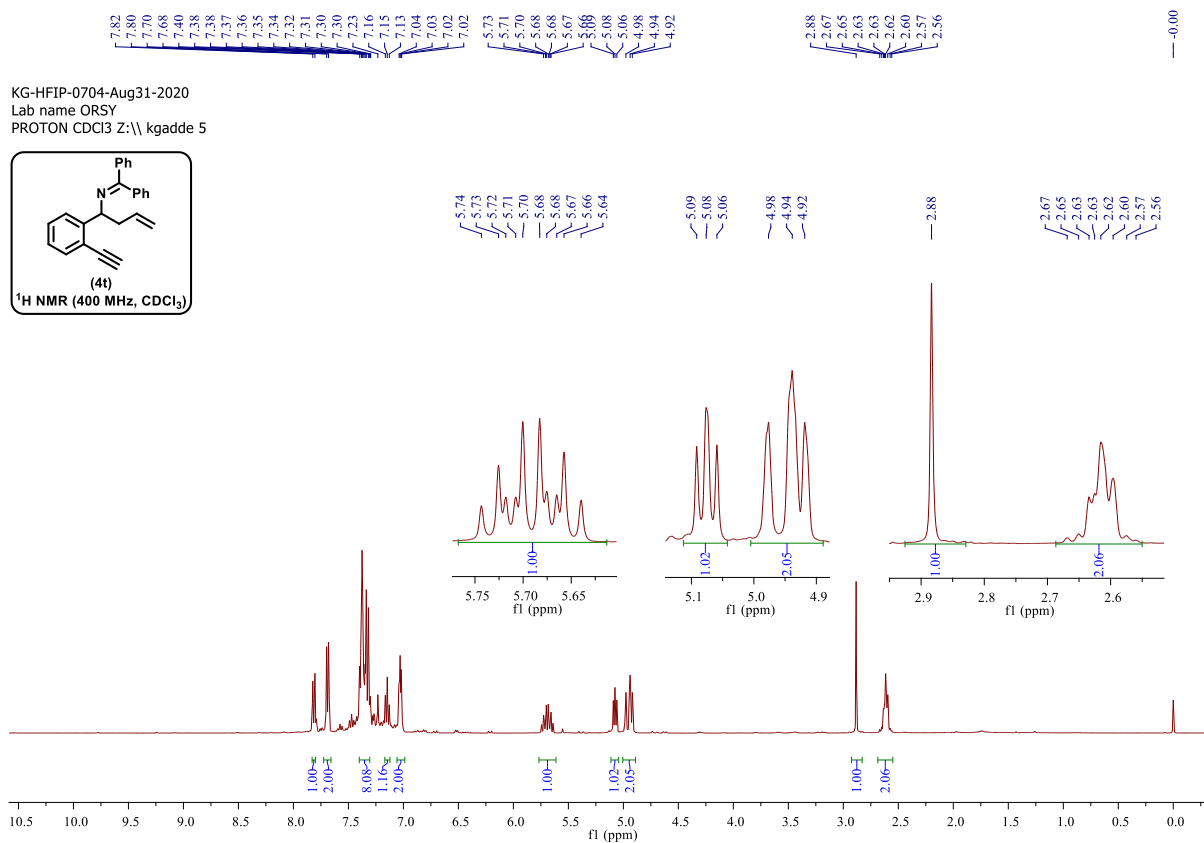

(4r)
 ^{13}C NMR (101 MHz, CDCl_3)



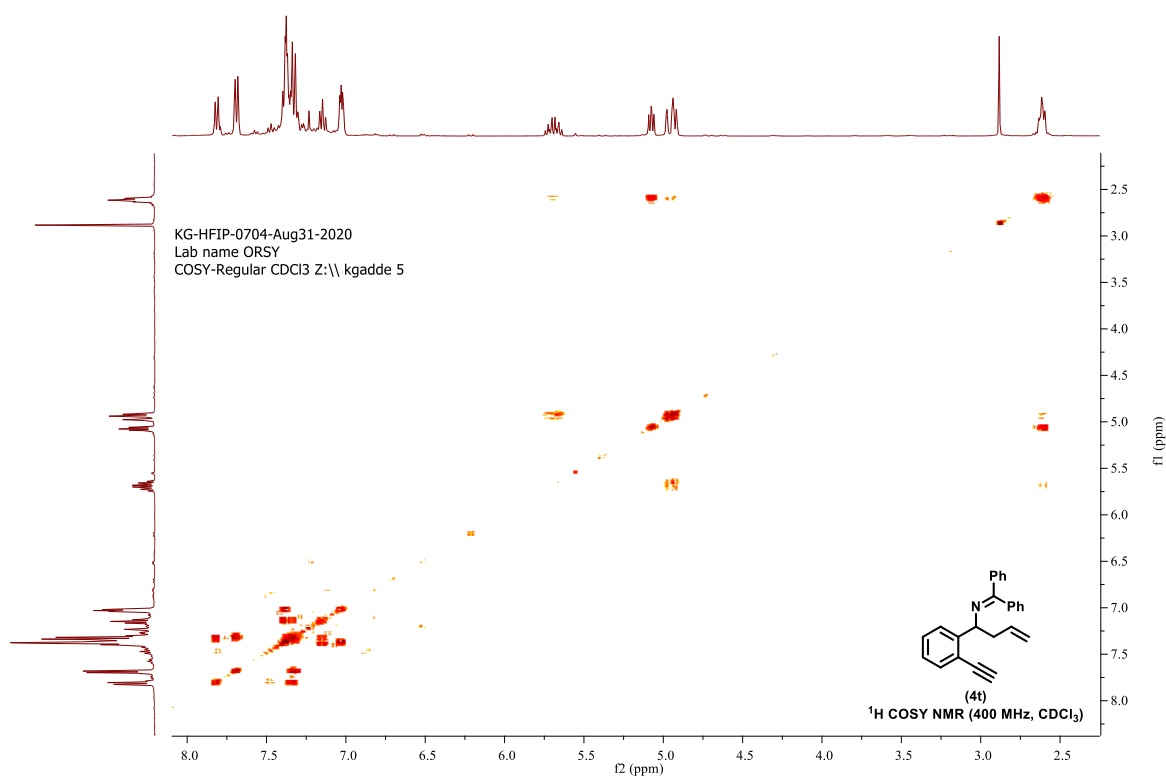
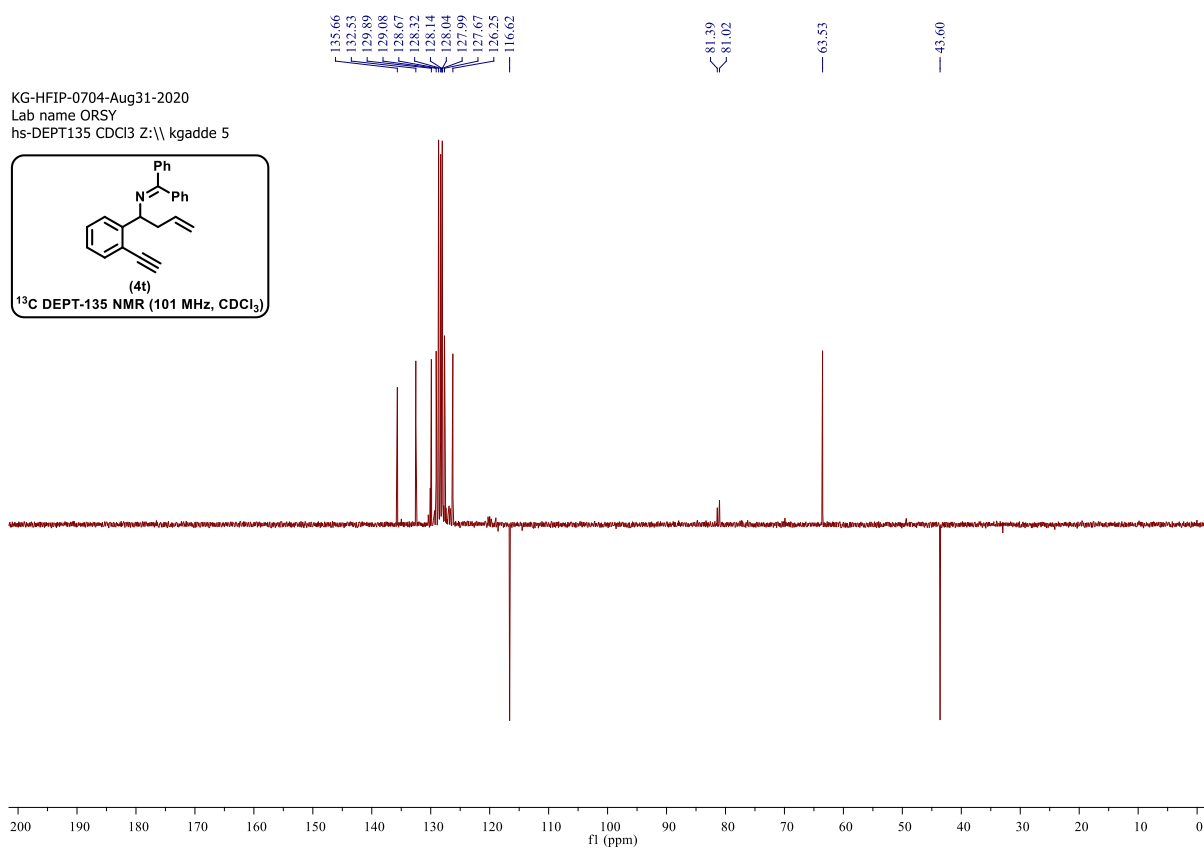
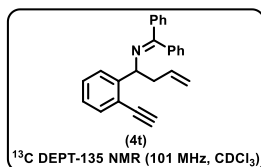
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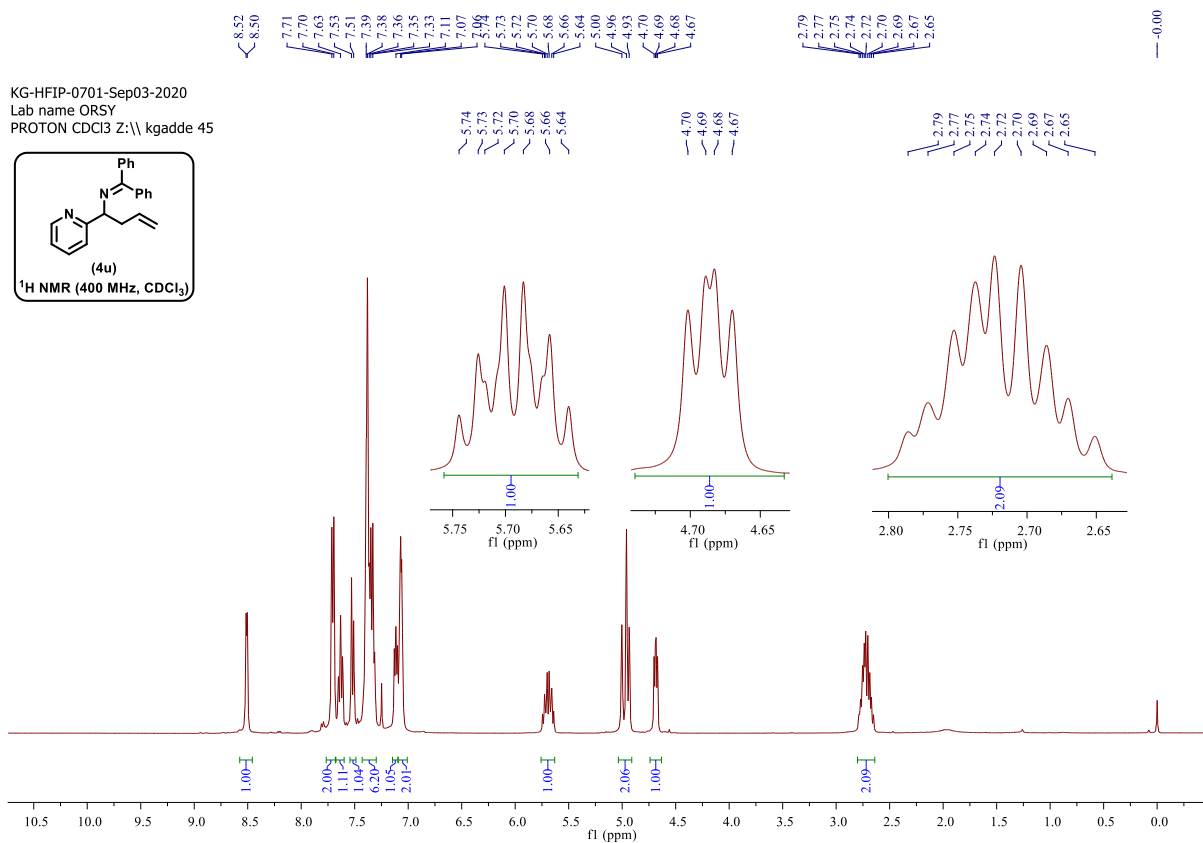
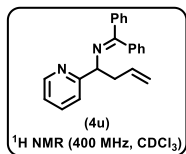




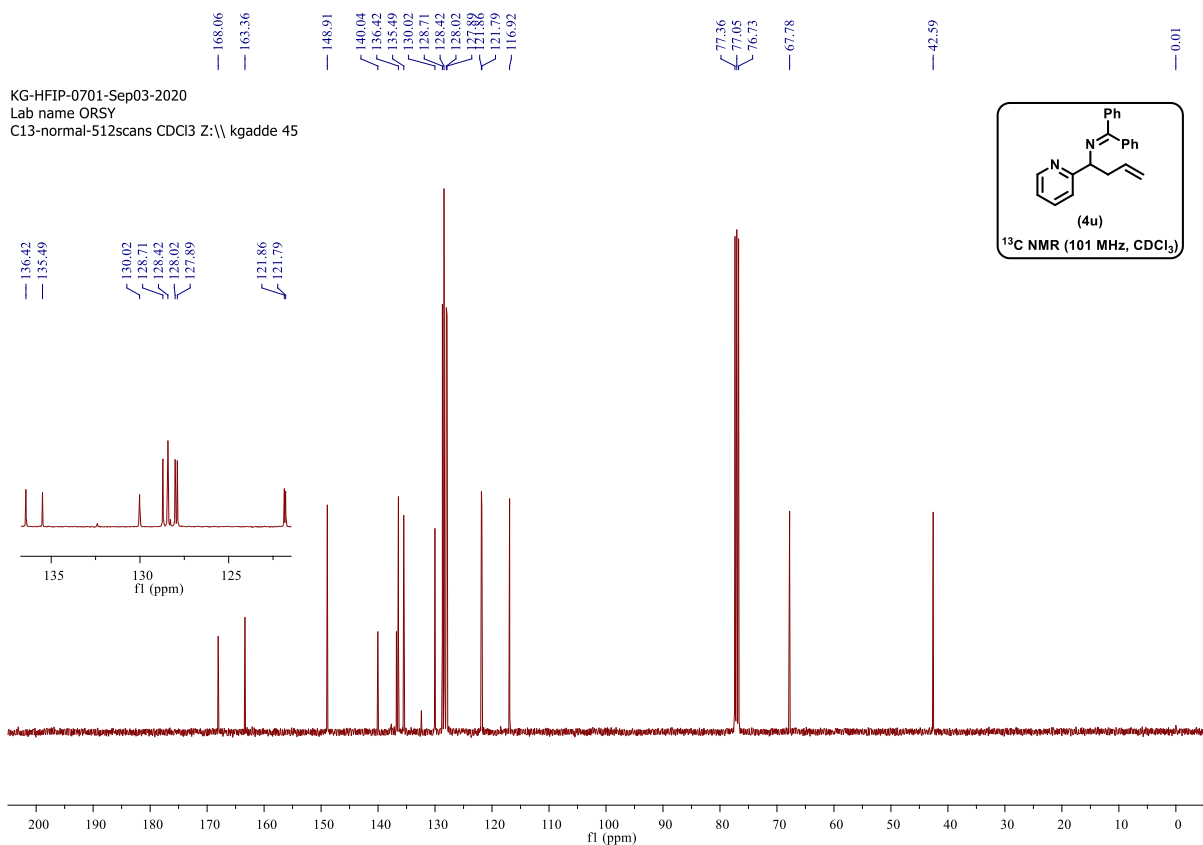
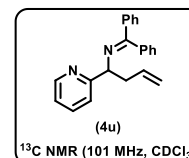
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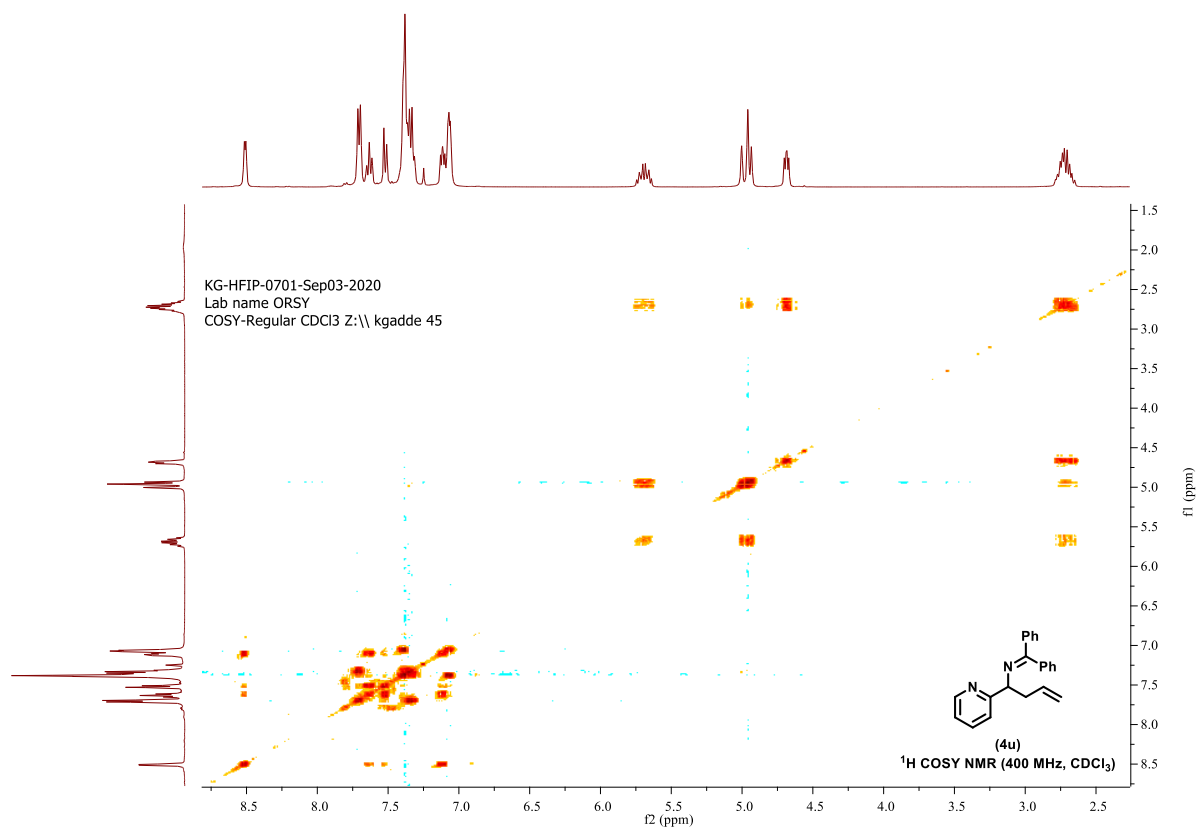
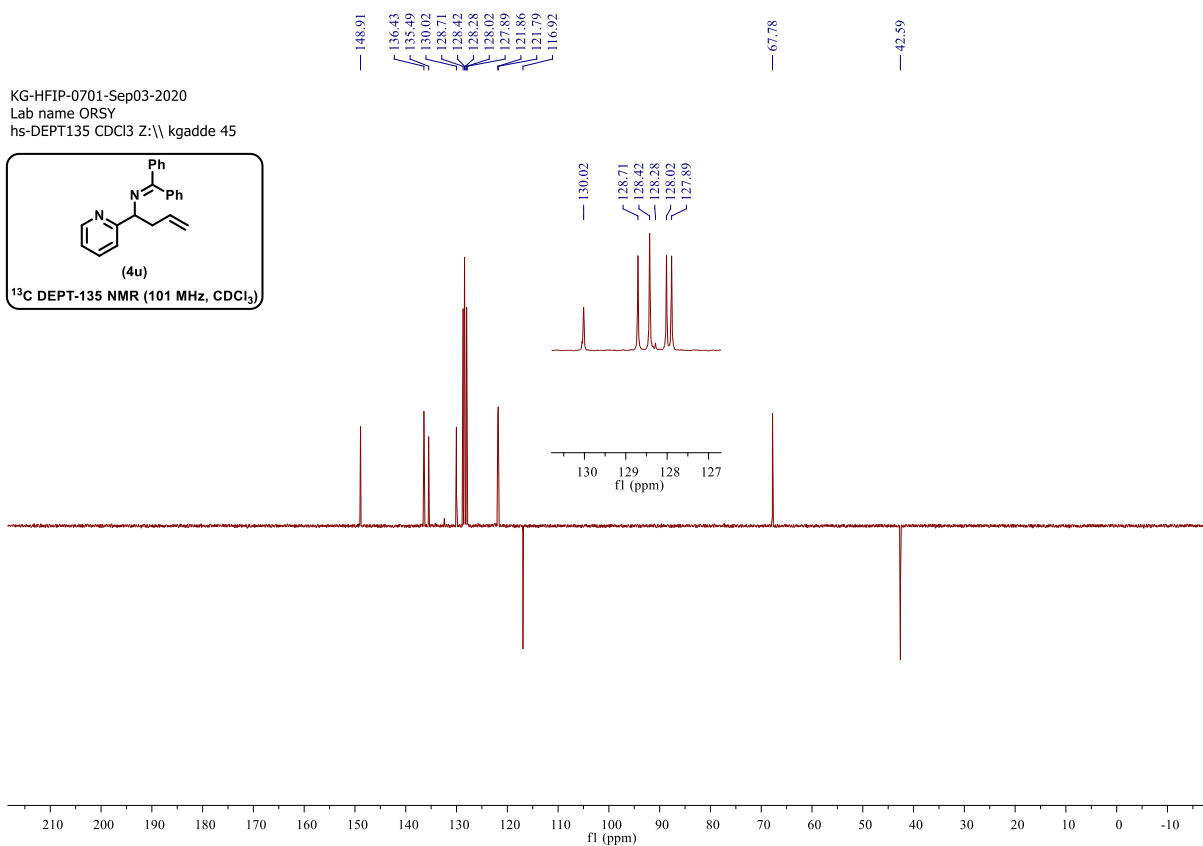
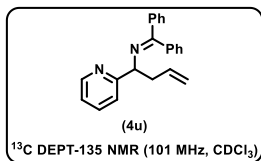
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Lab name ORSY
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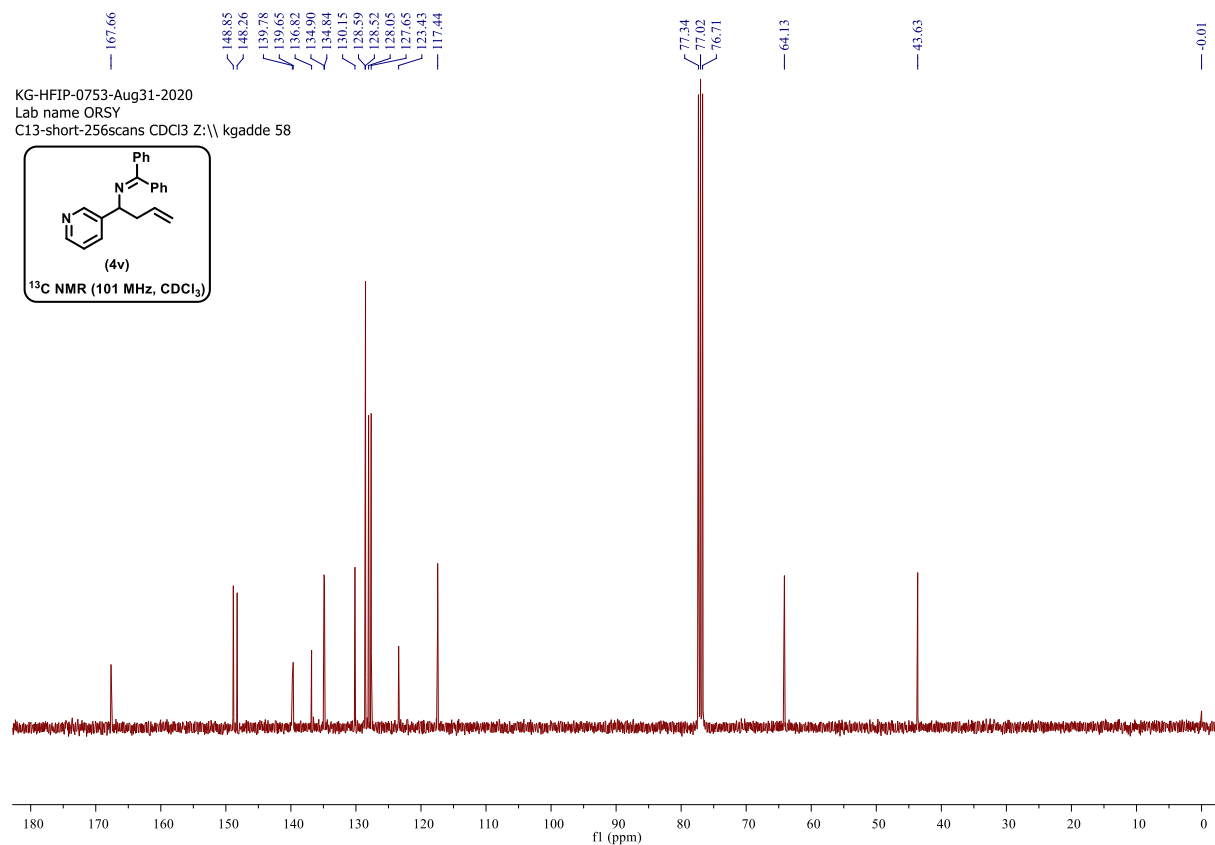
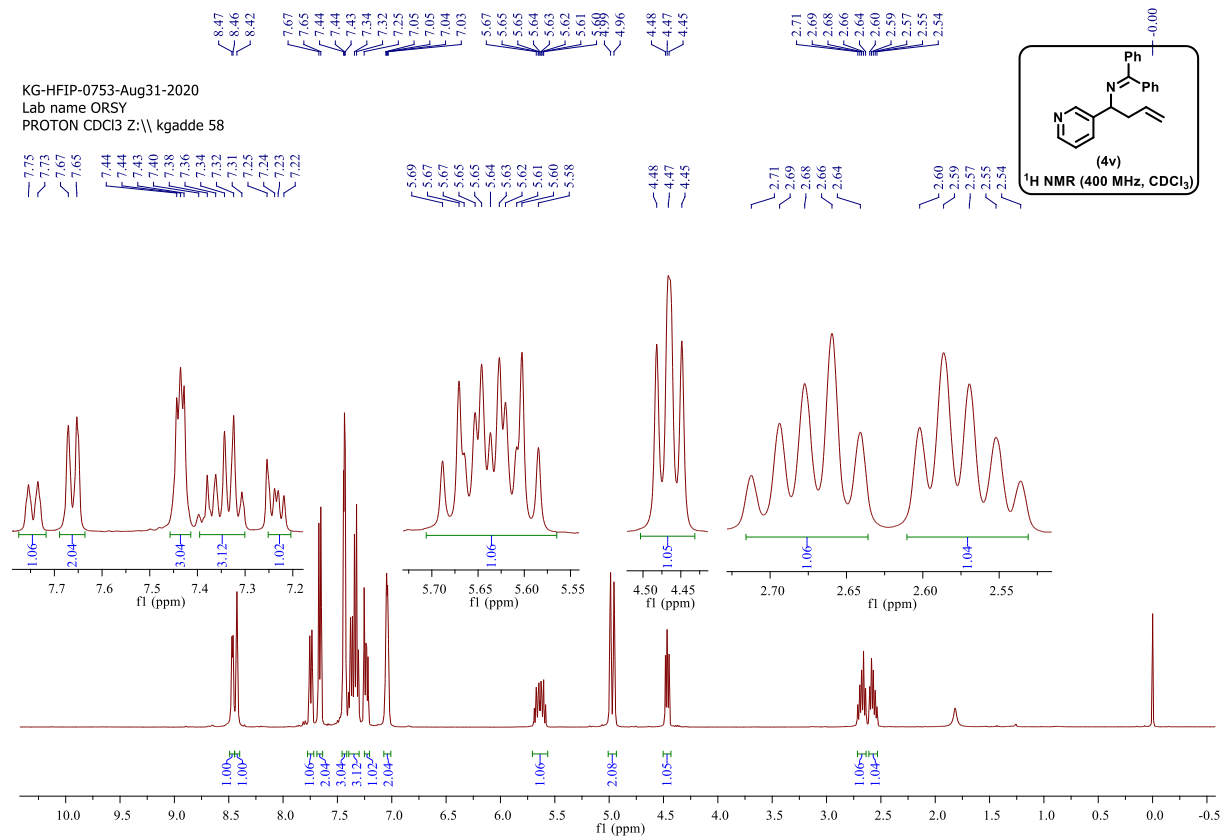


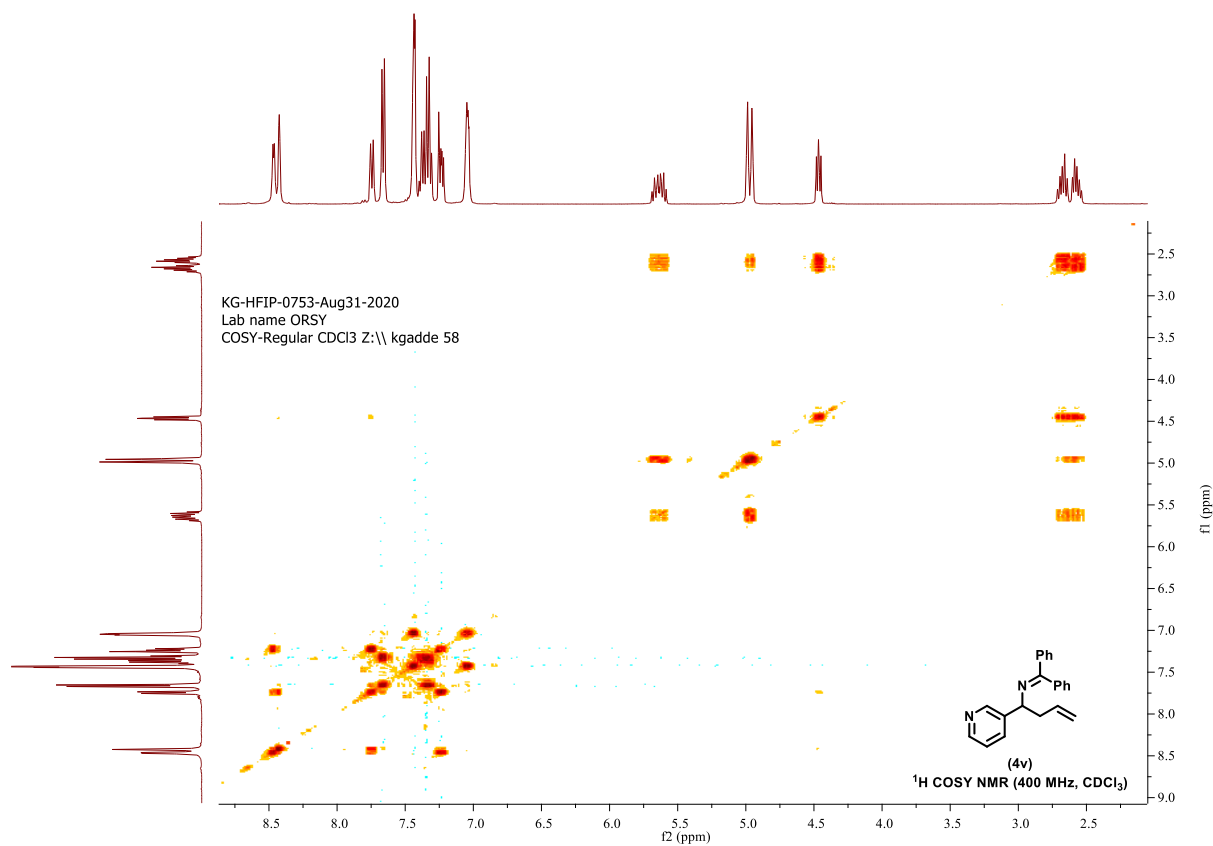
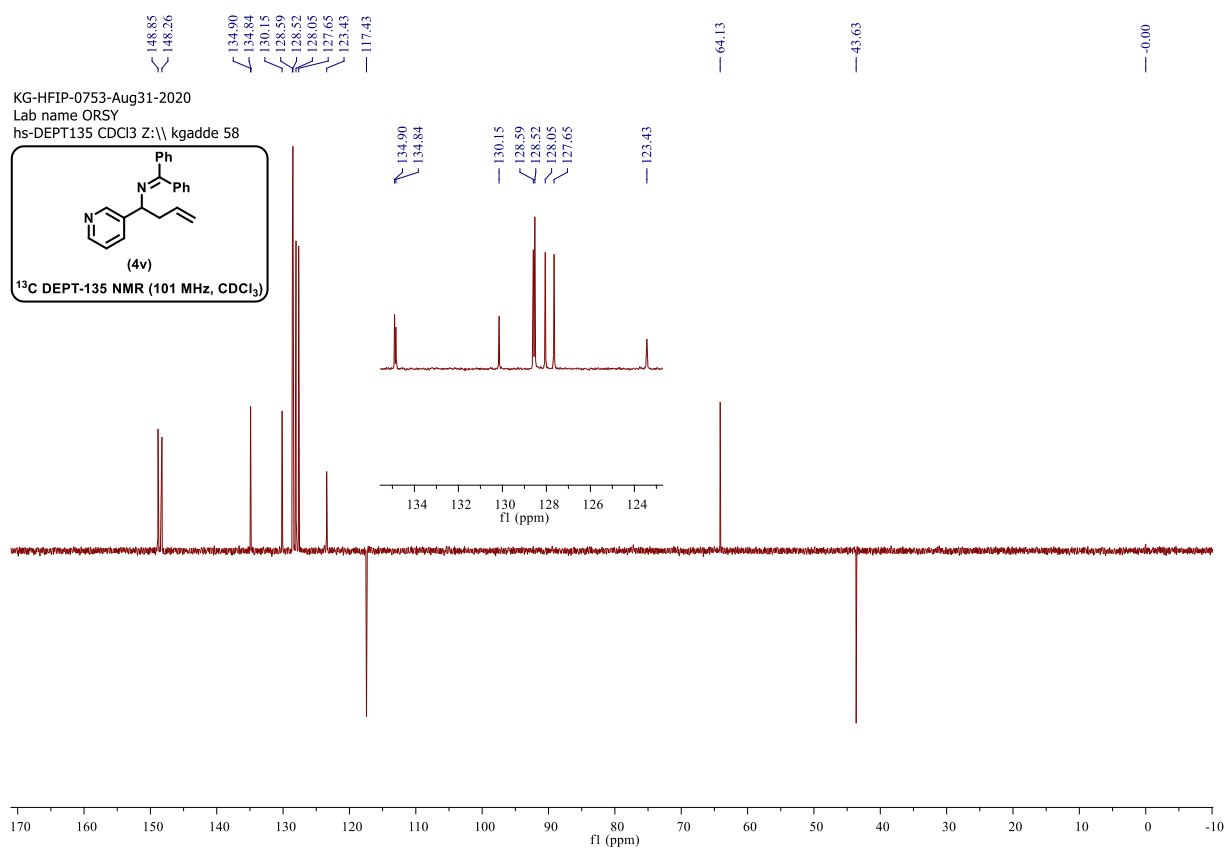
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Lab name ORSY
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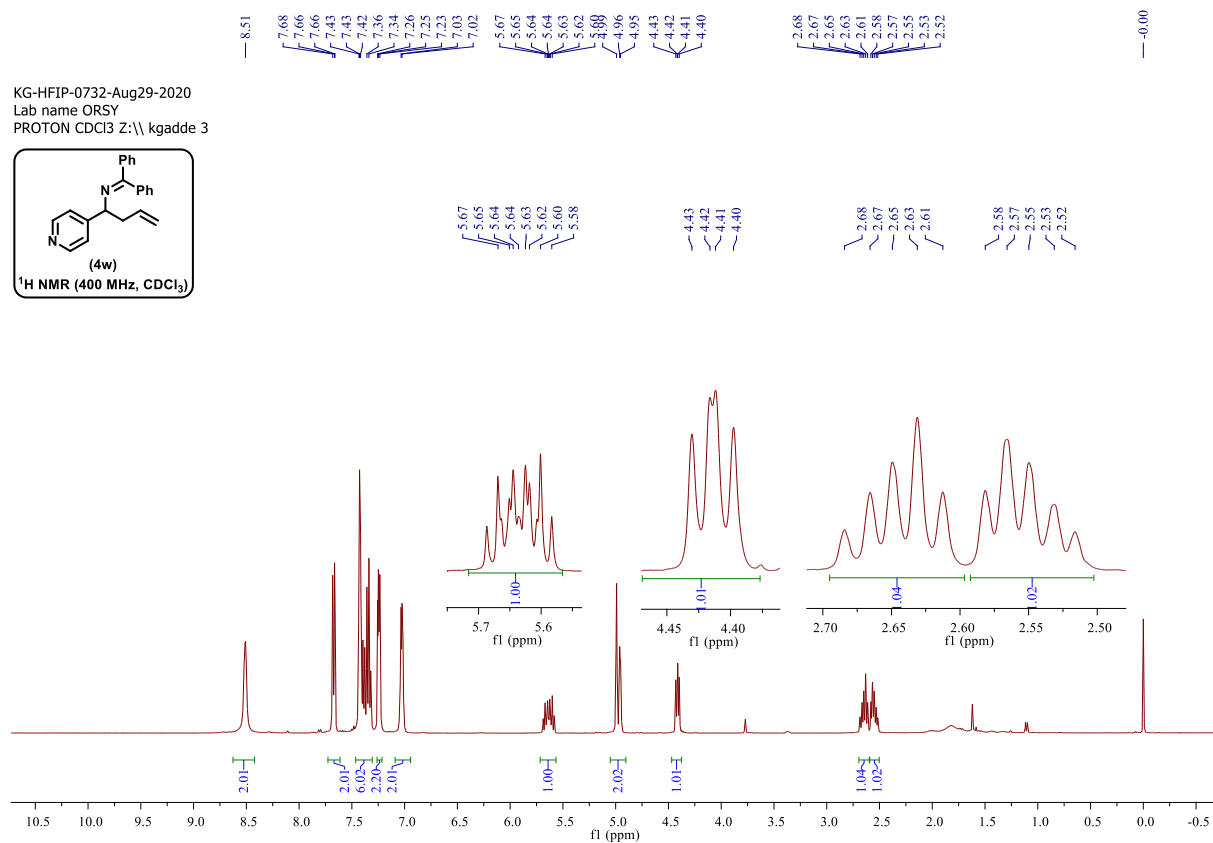
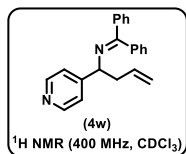
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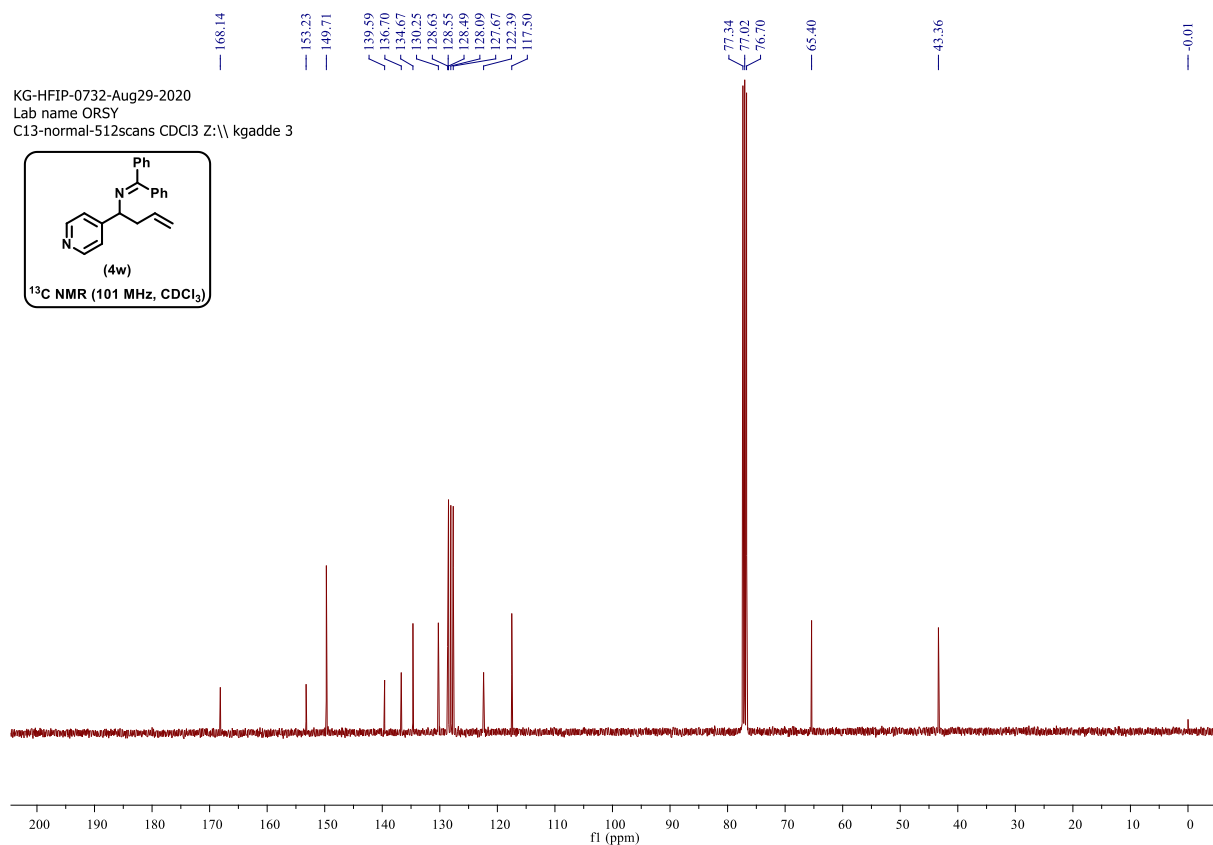
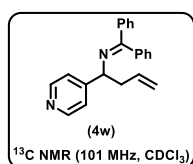




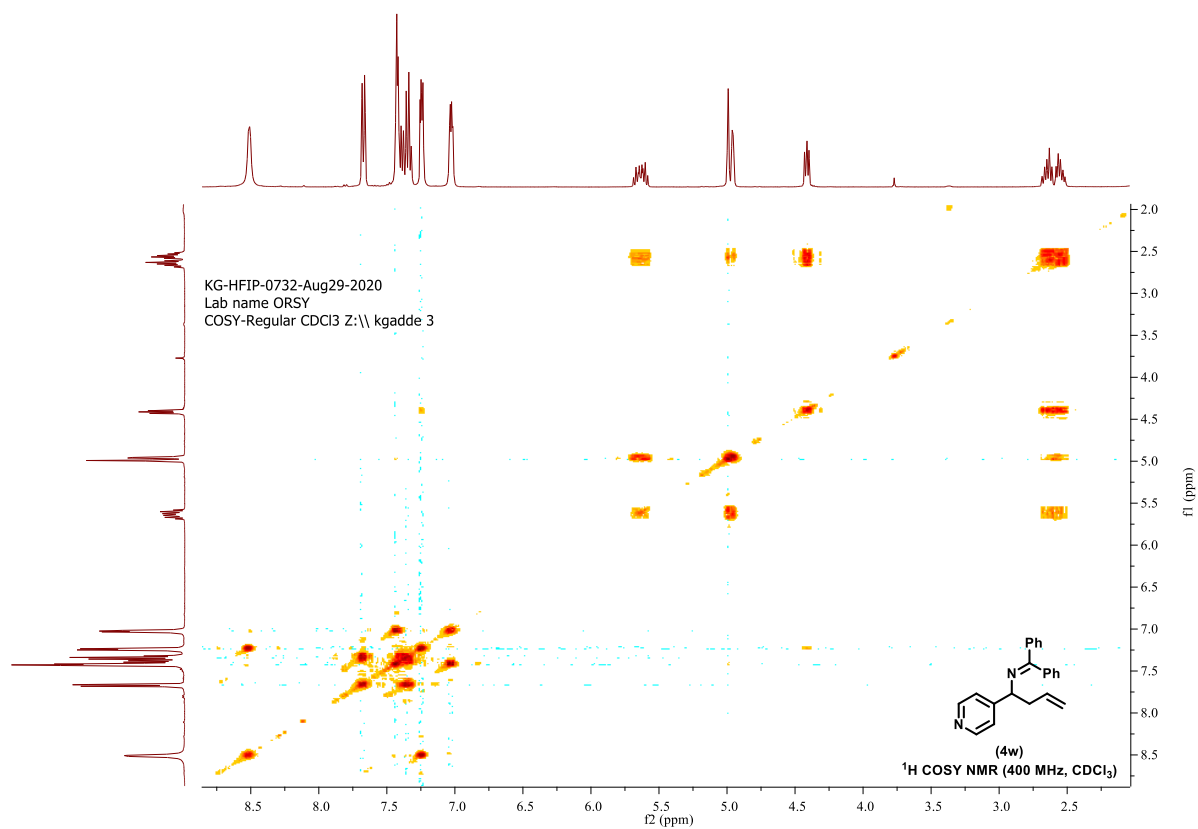
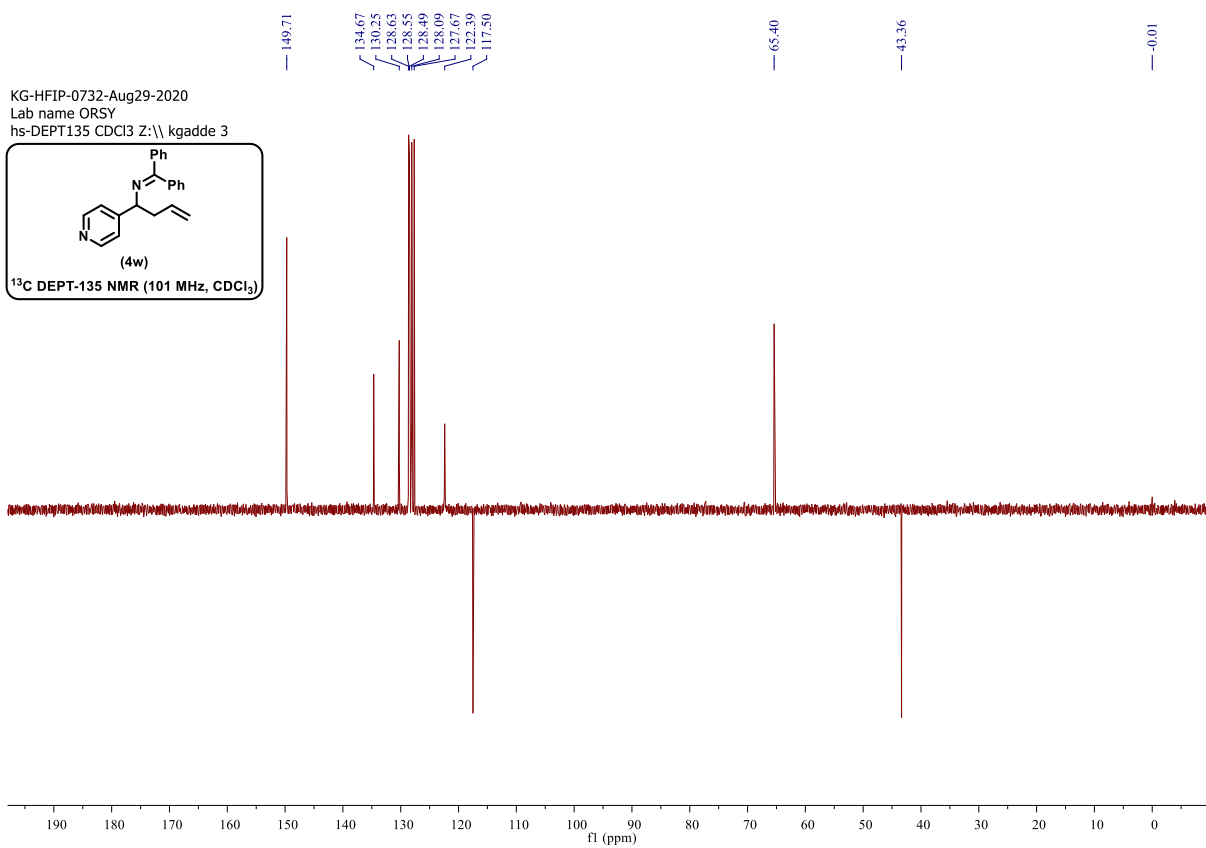
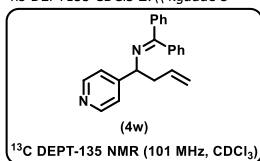
KG-HFIP-0732-Aug29-2020
Lab name ORSY
PROTON CDCl3 Z:\\\\ kgadde 3

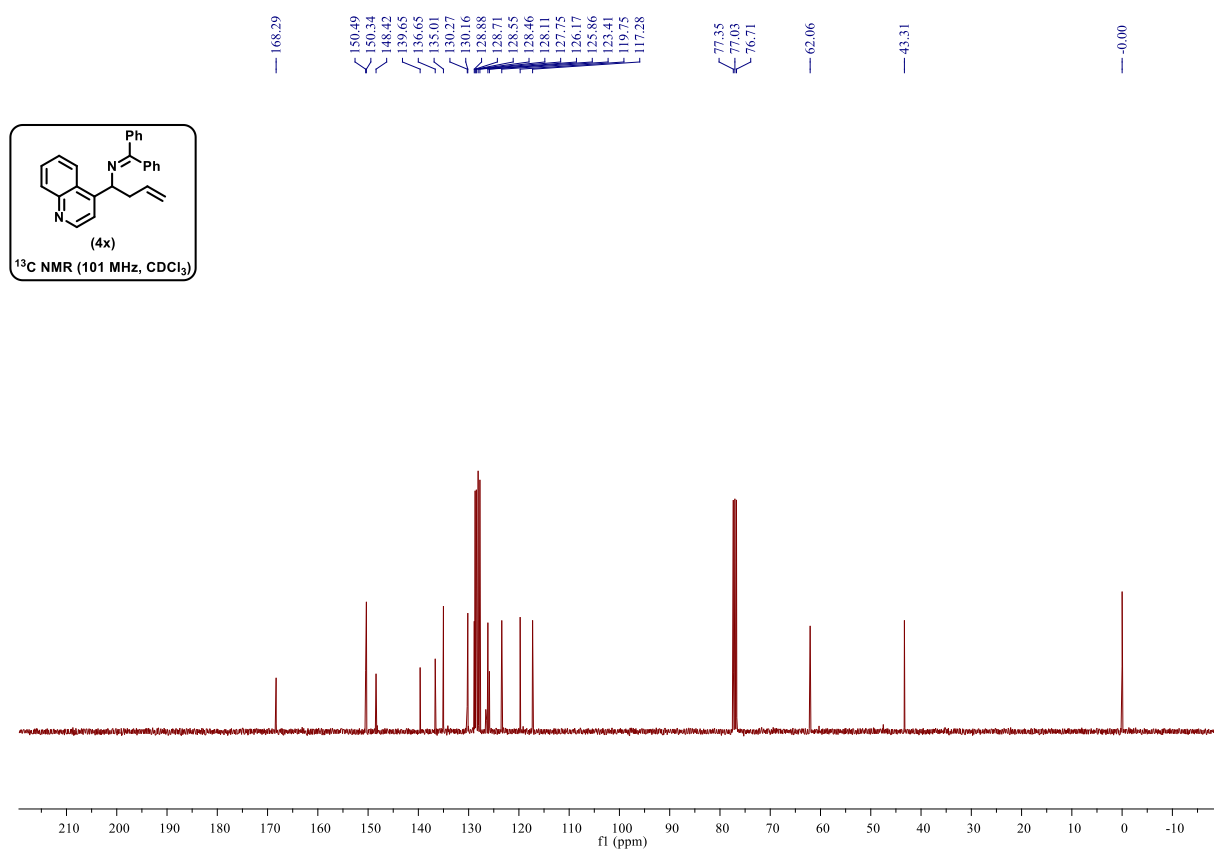
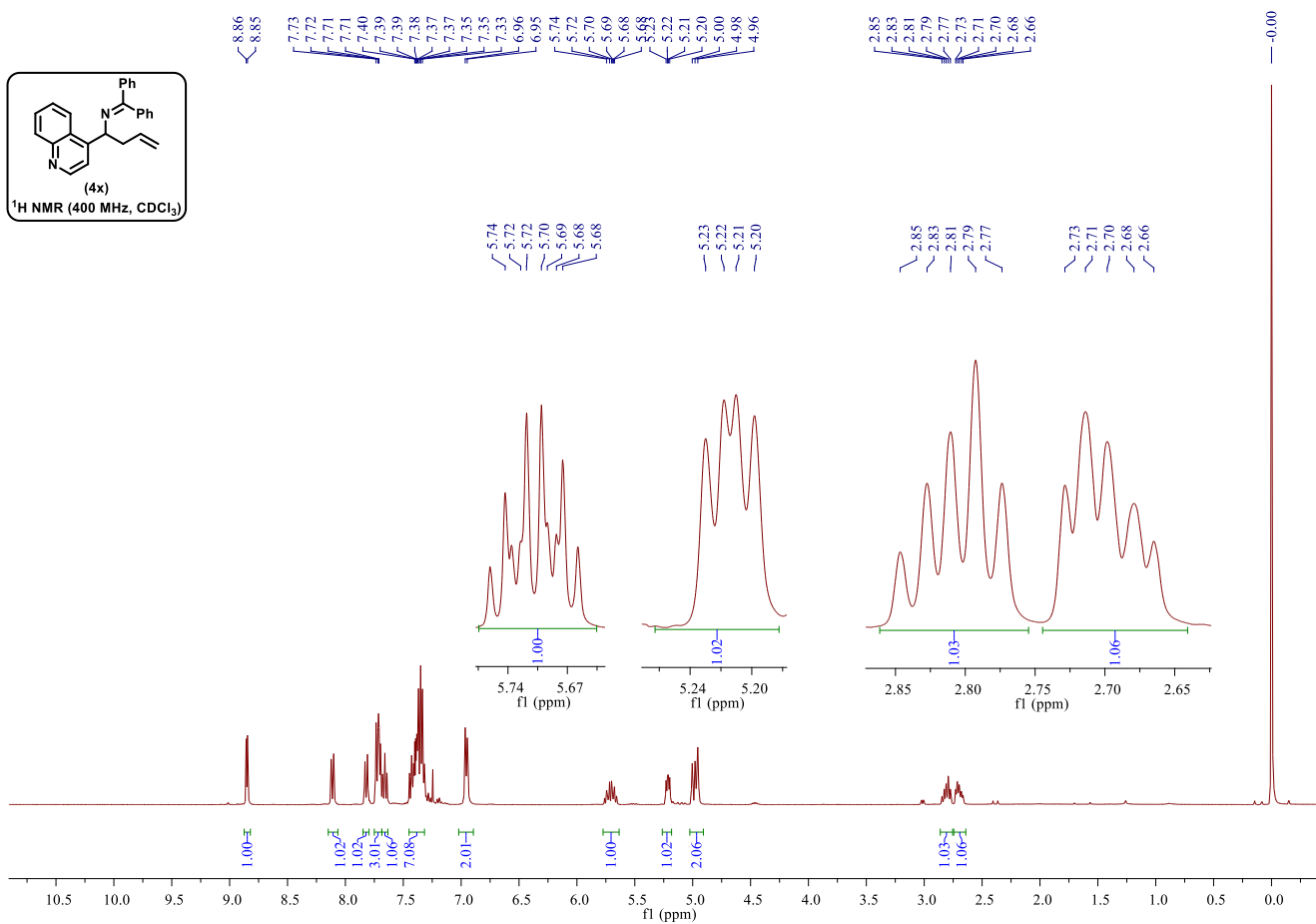


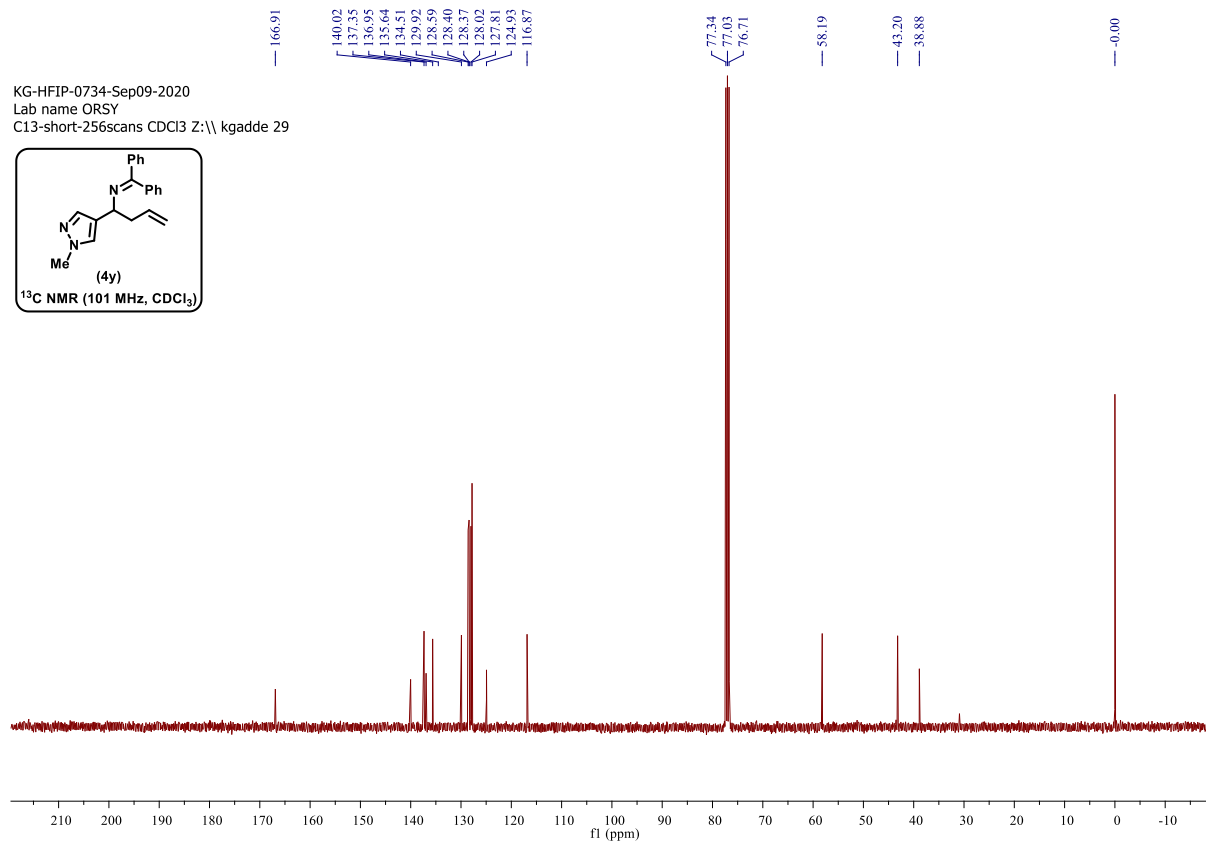
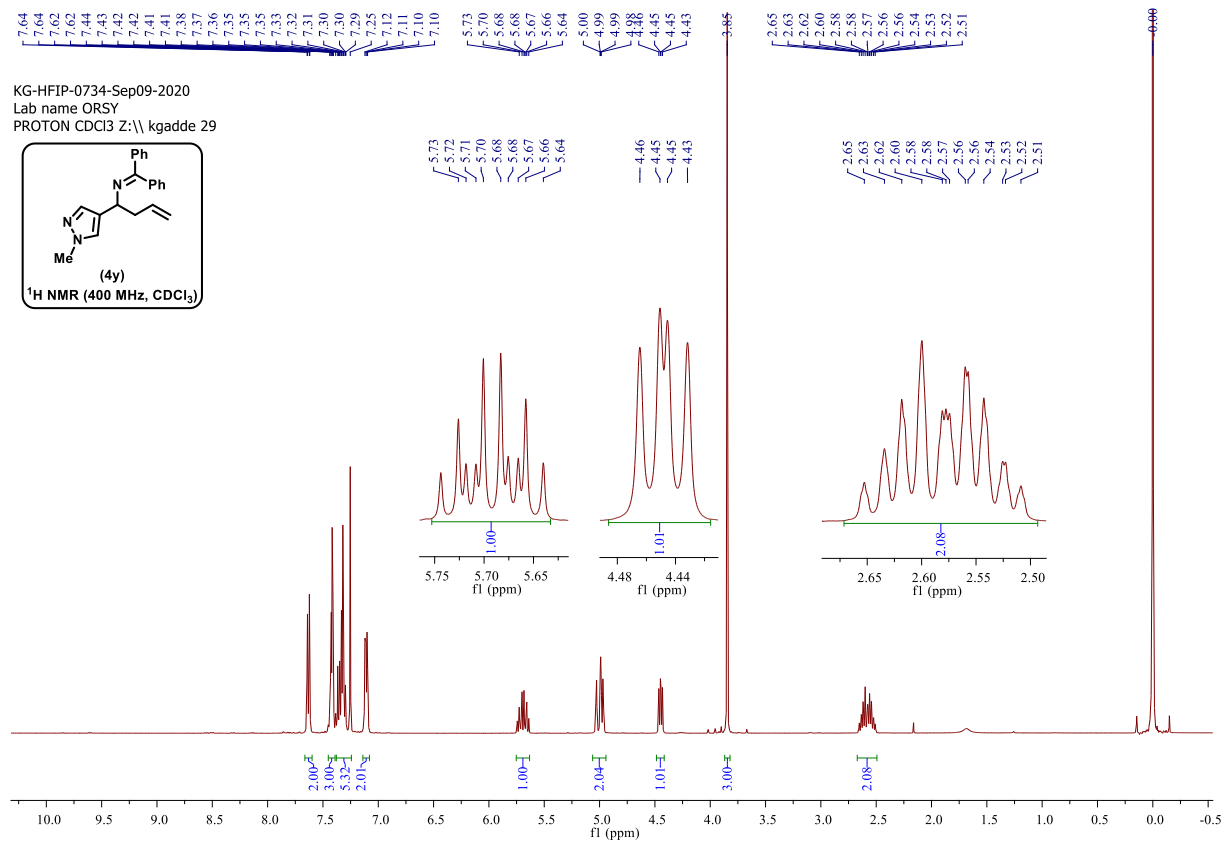
KG-HFIP-0732-Aug29-2020
Lab name ORSY
C13-normal-512scans CDCl3 Z:\\\\ kgadde 3



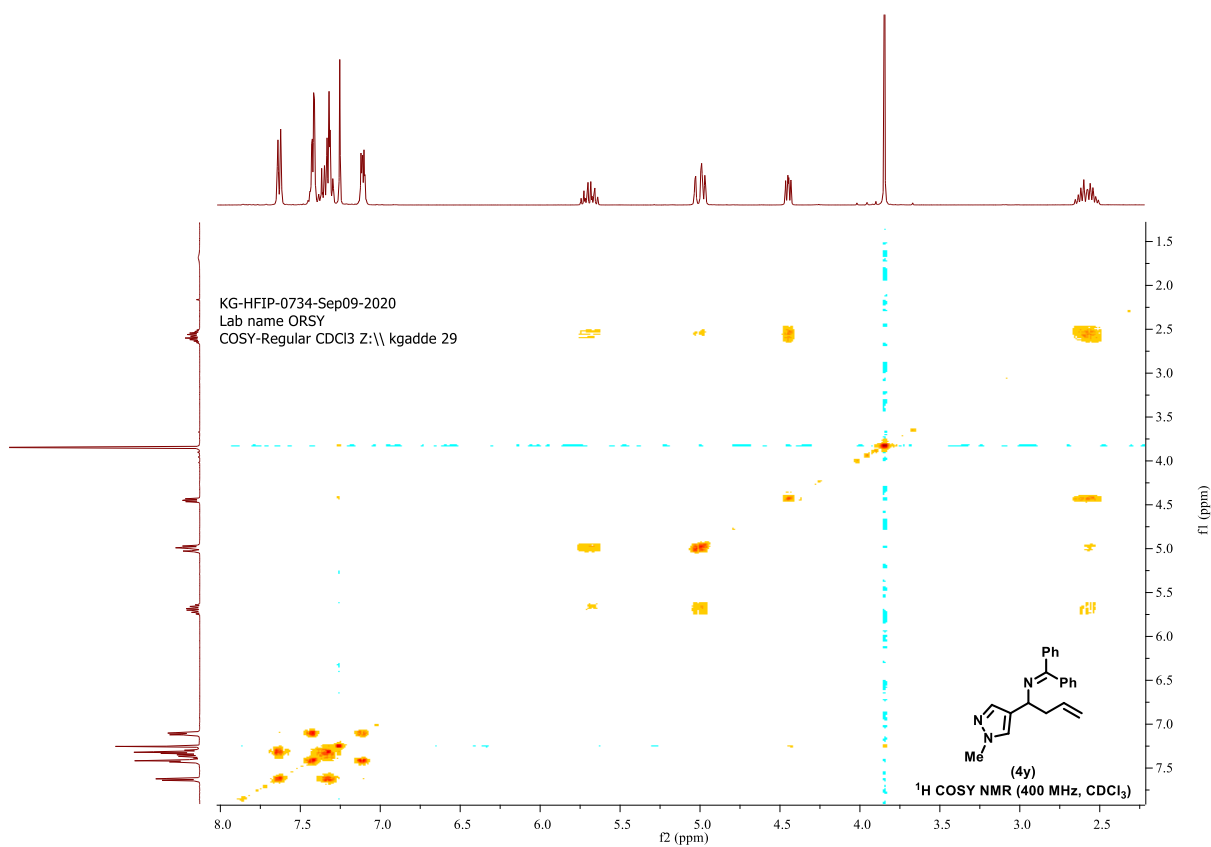
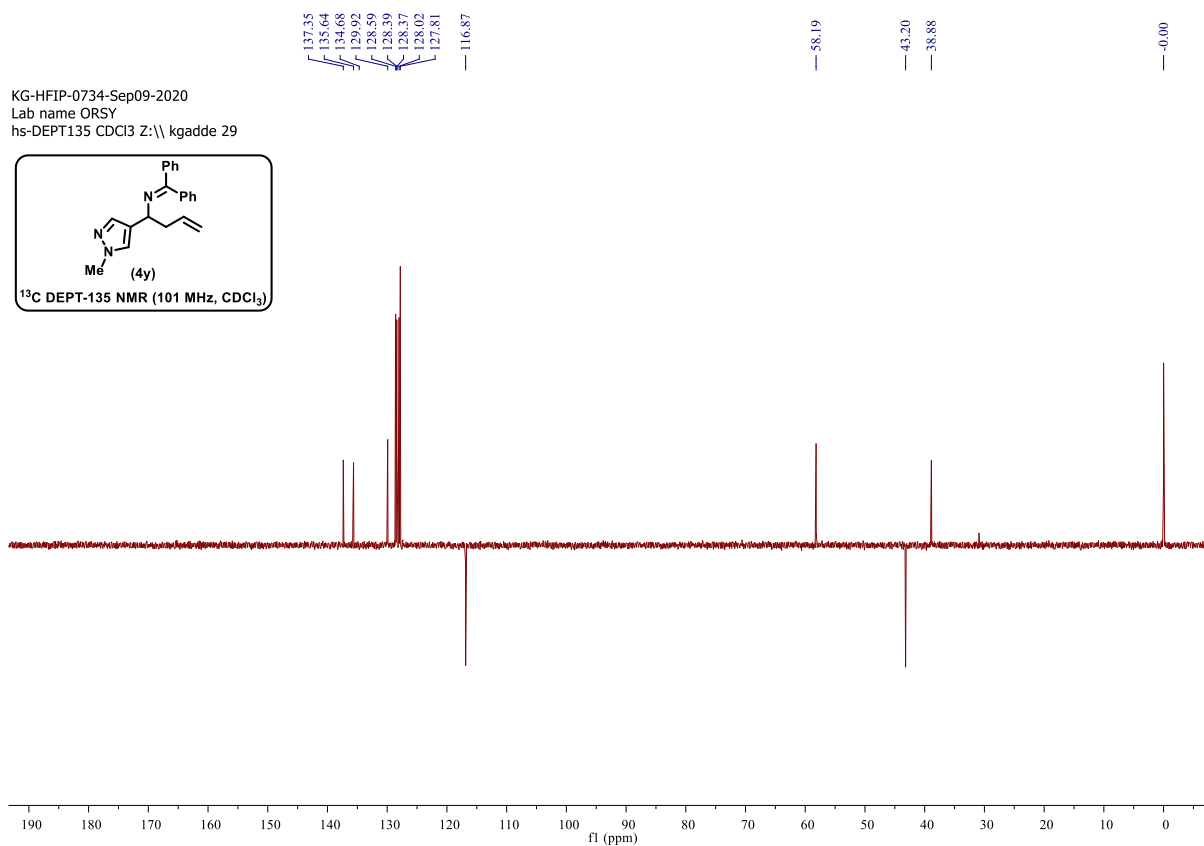
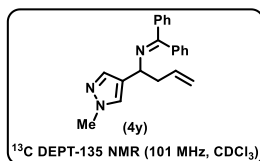
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 Lab name ORSY
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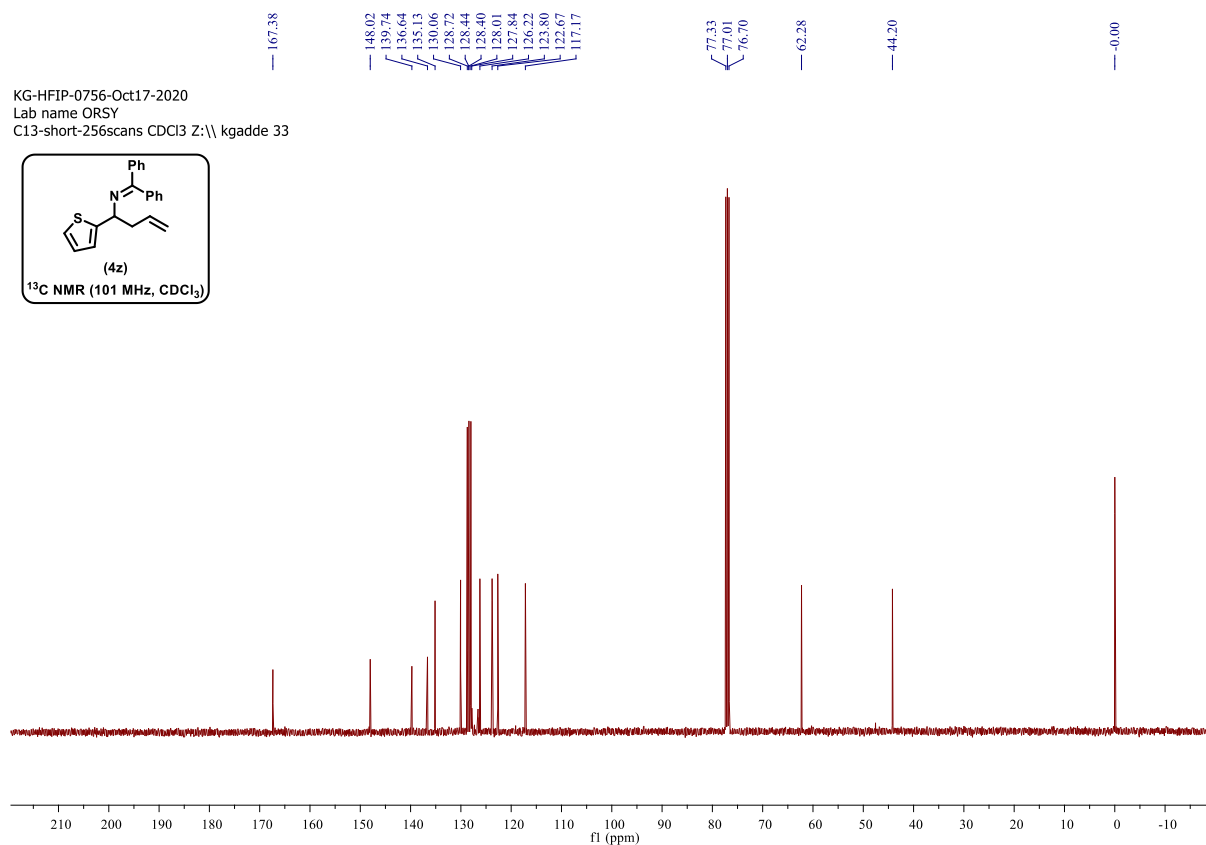
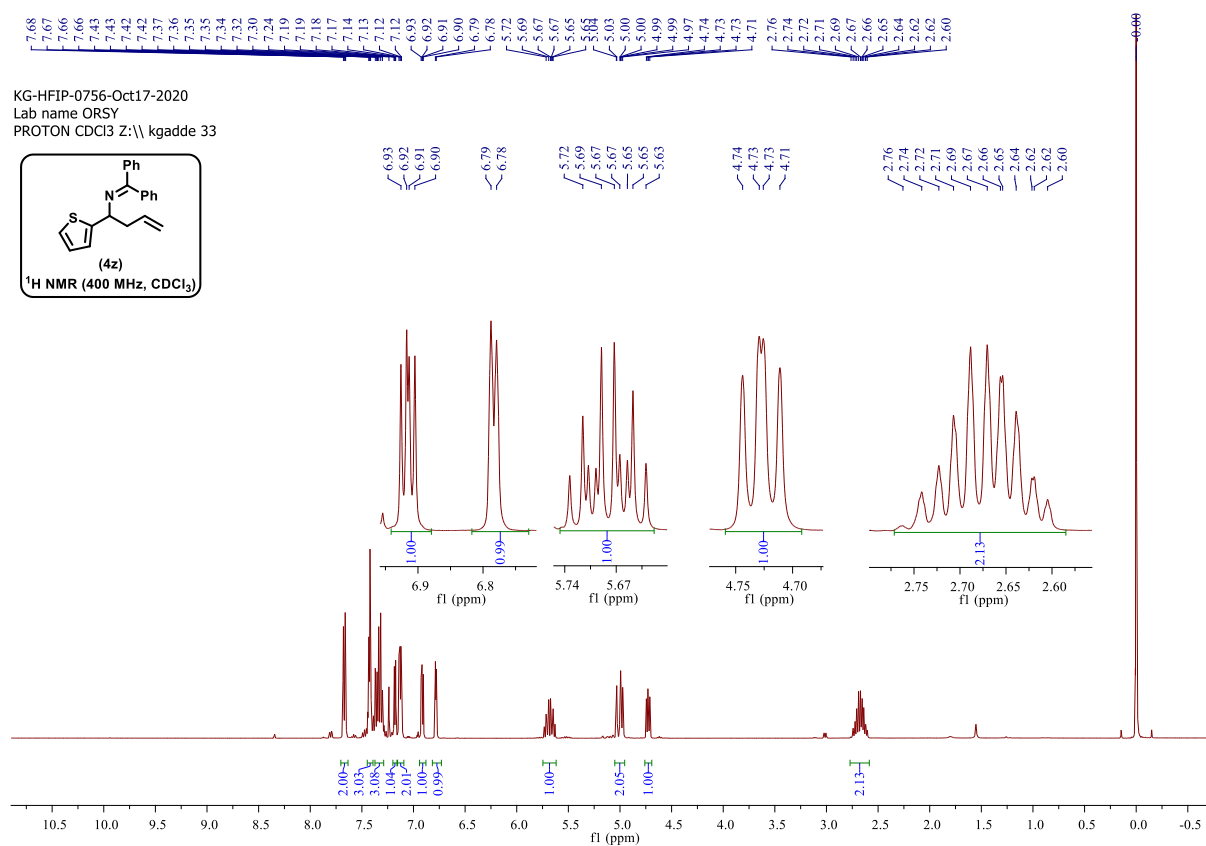




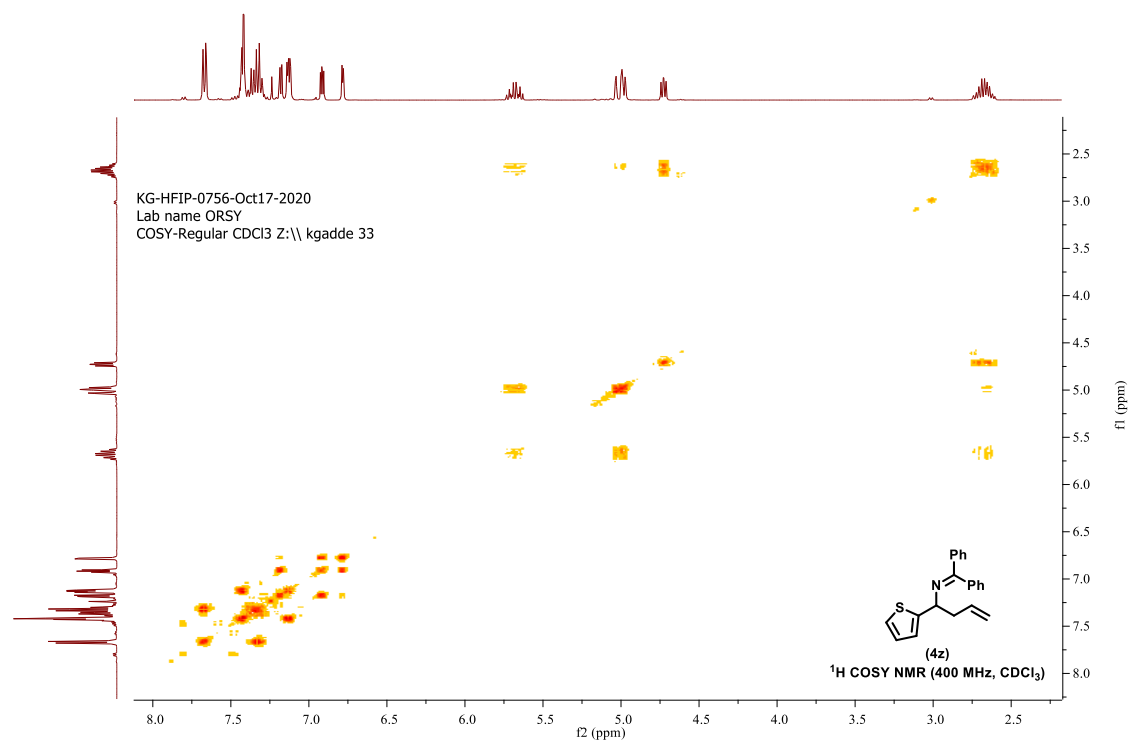
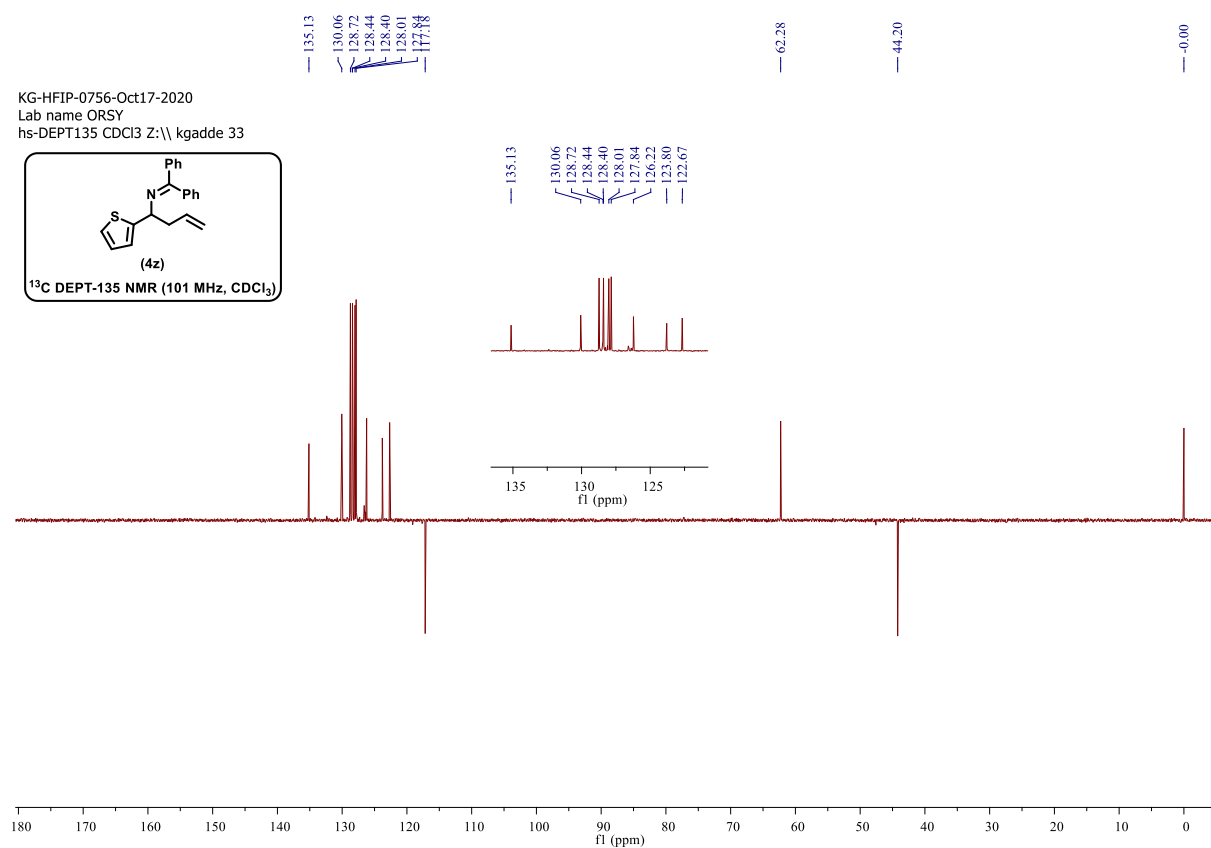
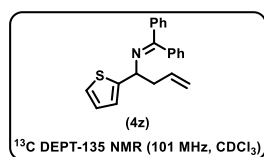


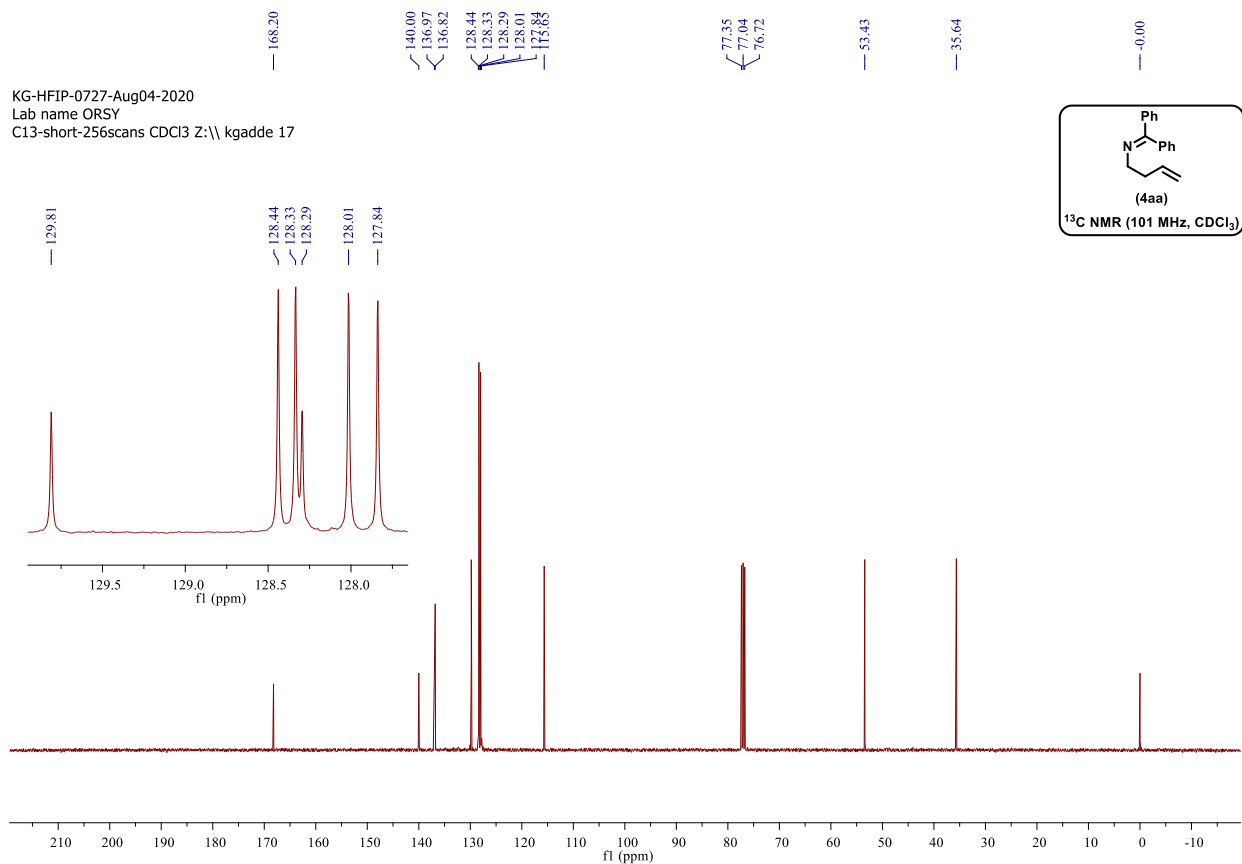
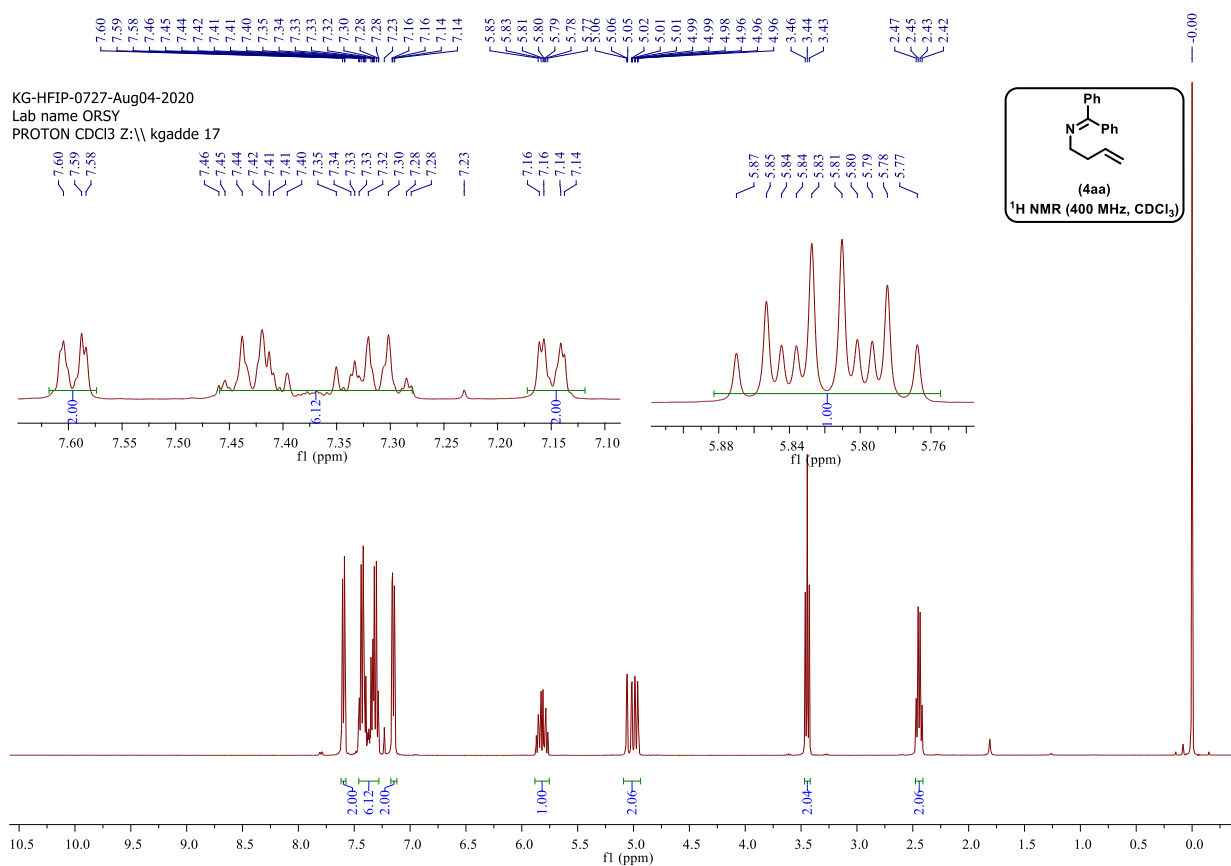
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 Lab name ORSY
 hs-DEPT135 CDCl3 Z:\\ kgadde 29



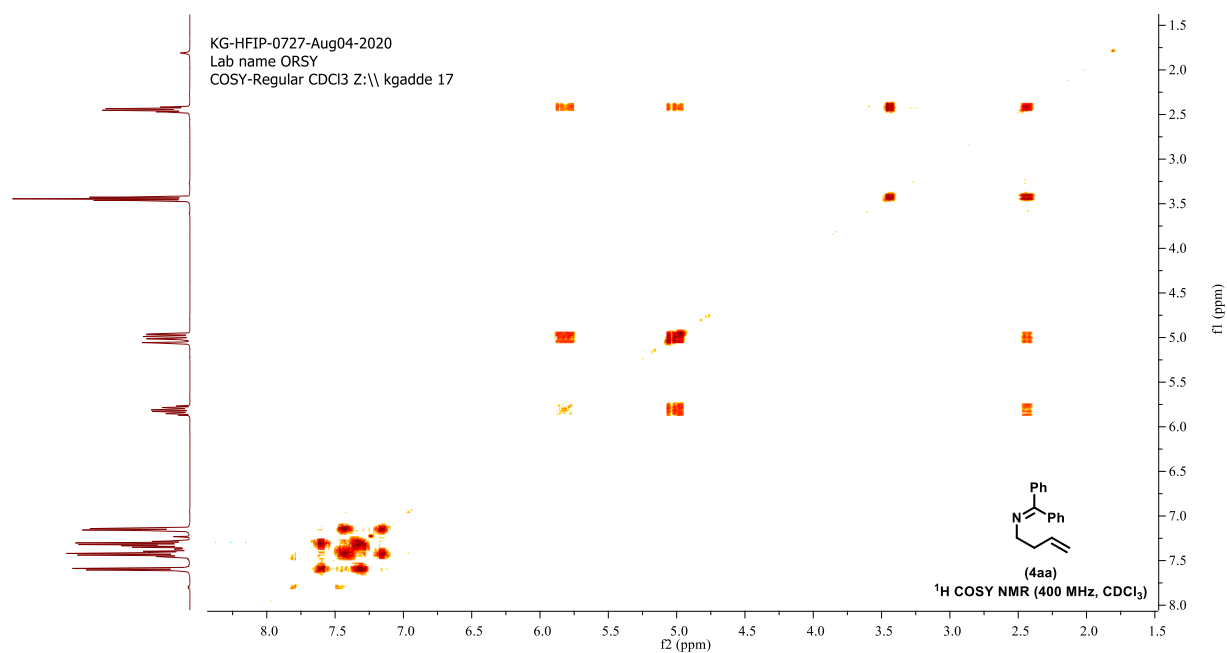
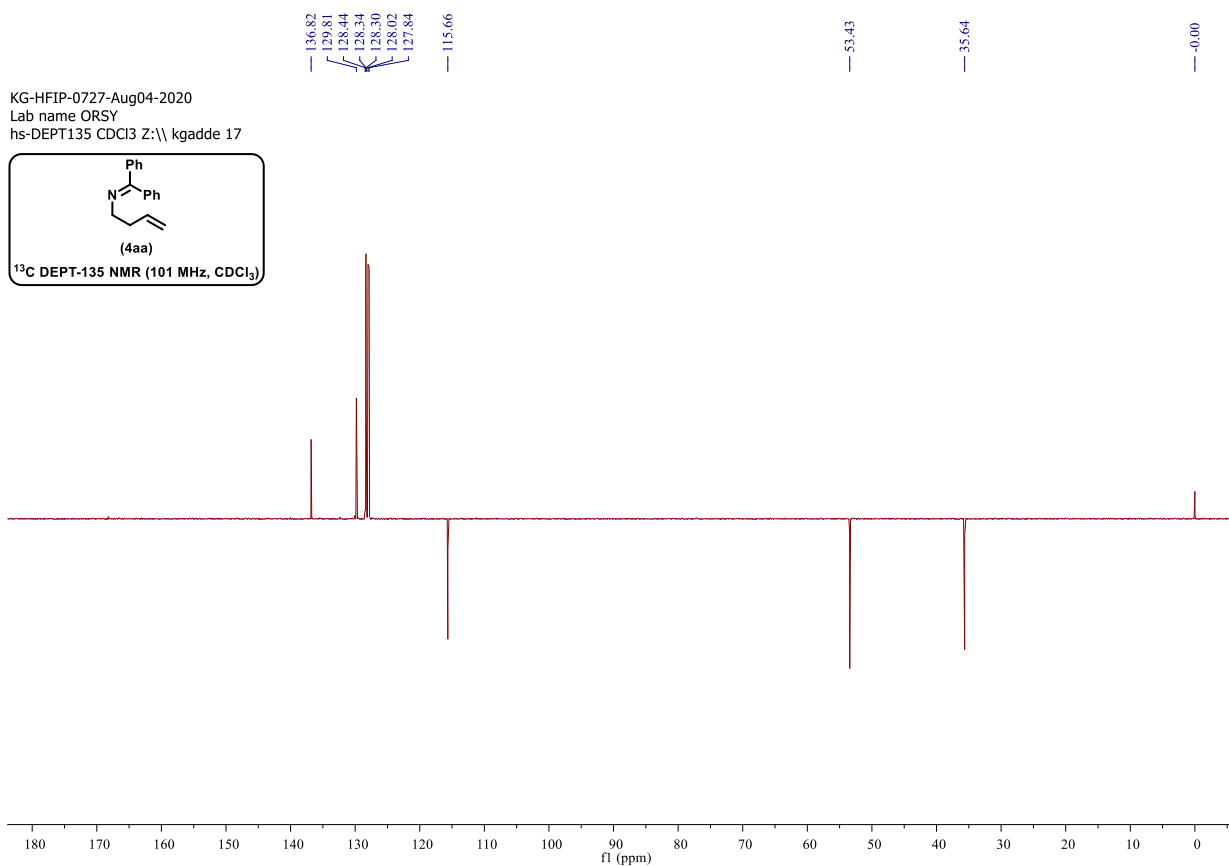
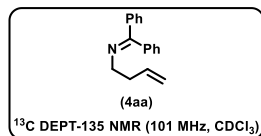


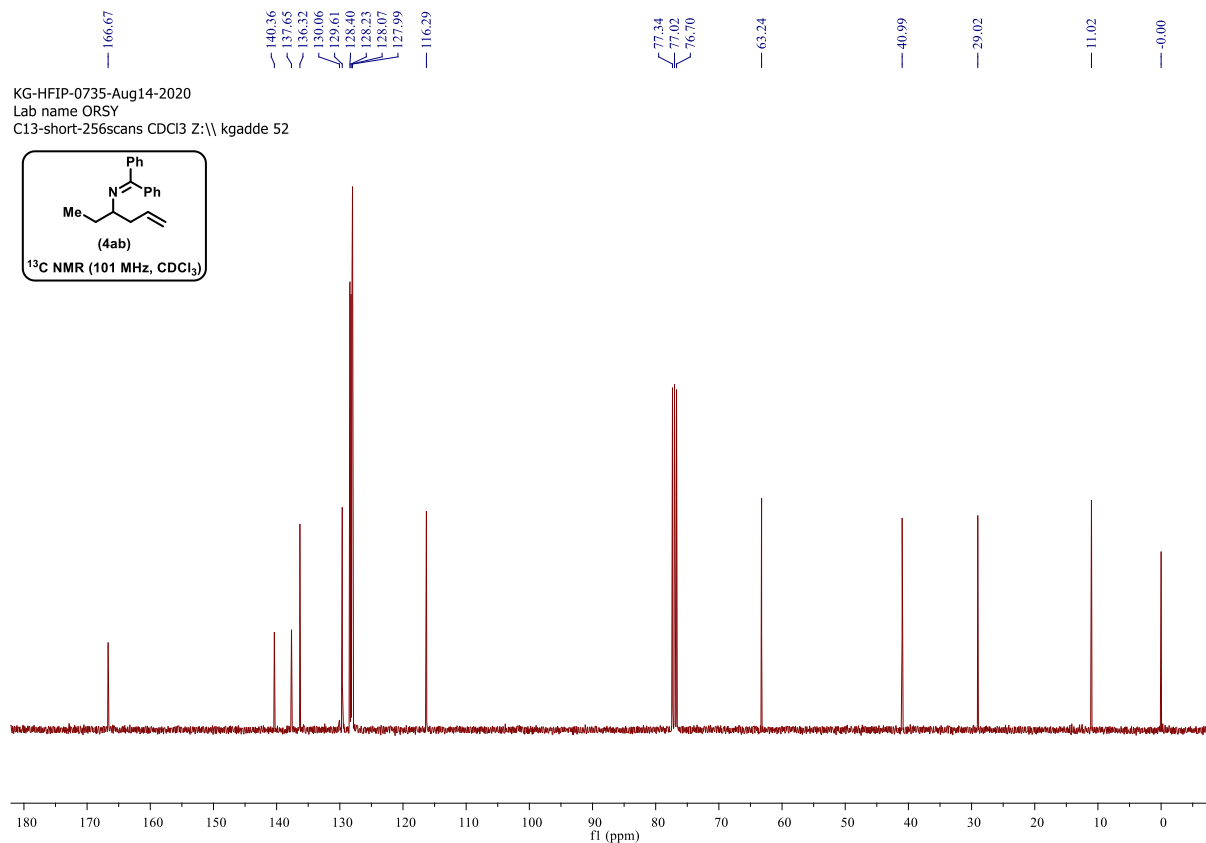
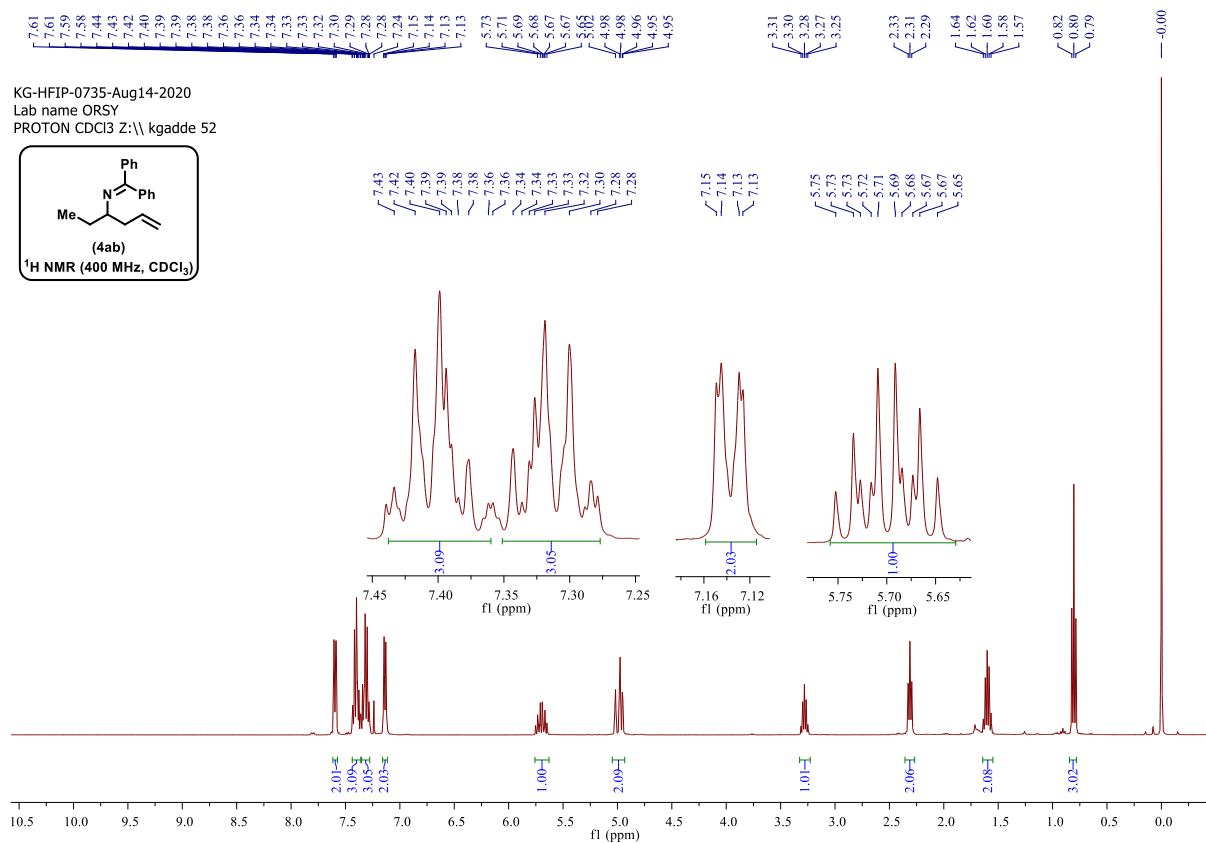
KG-HFIP-0756-Oct17-2020
 Lab name ORSY
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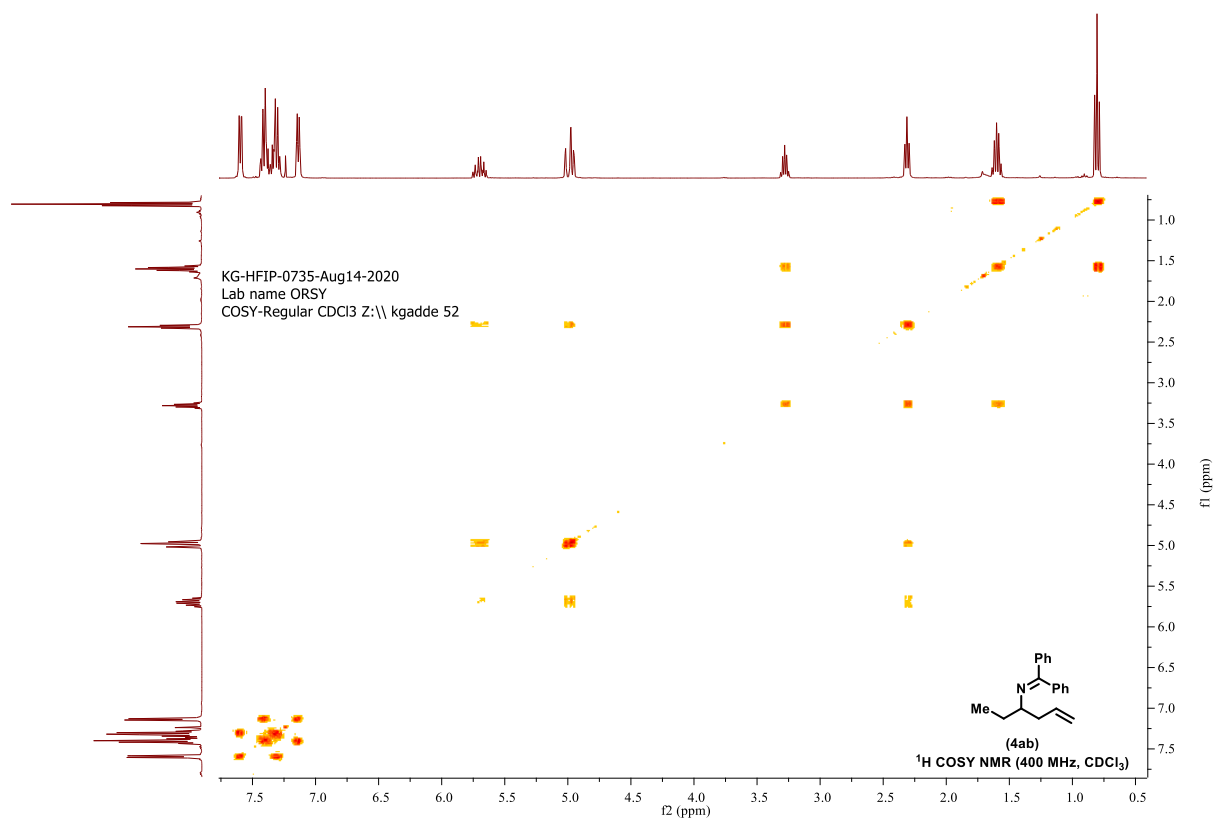
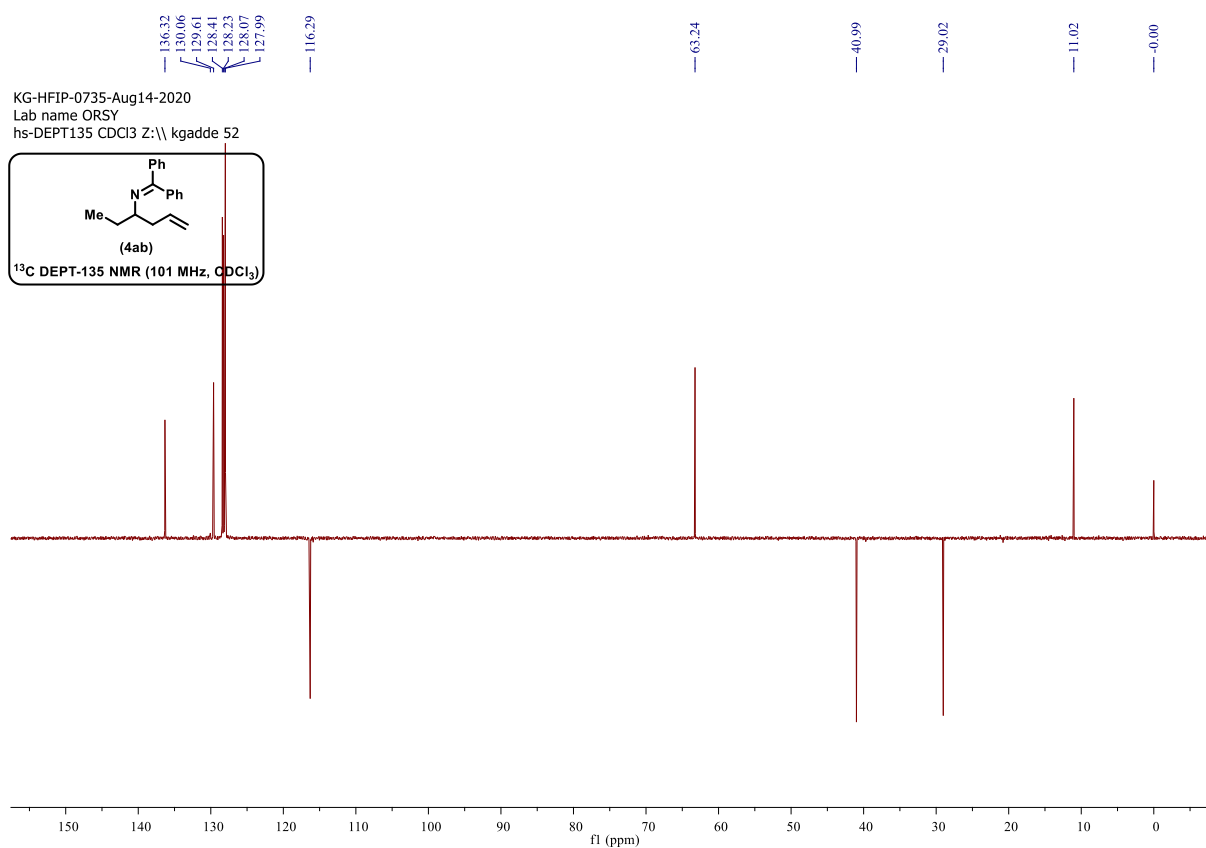


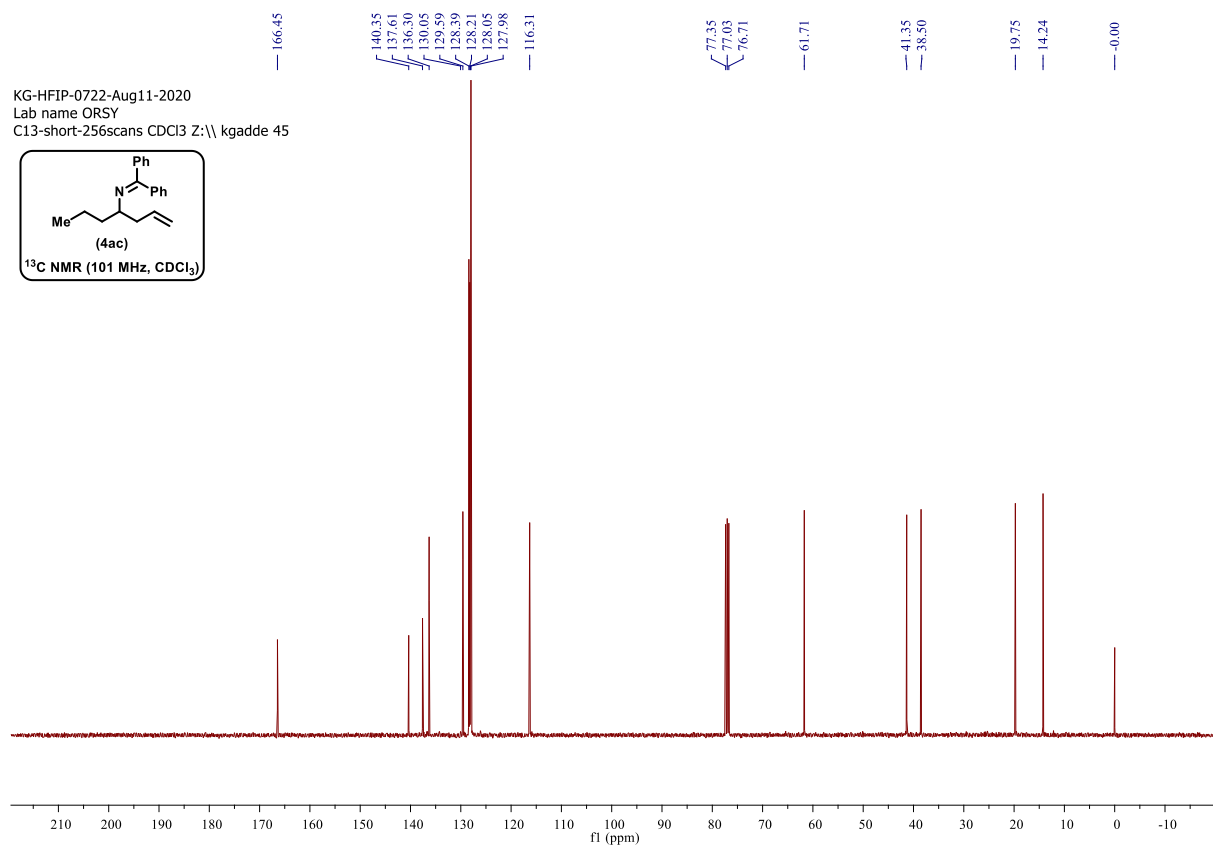
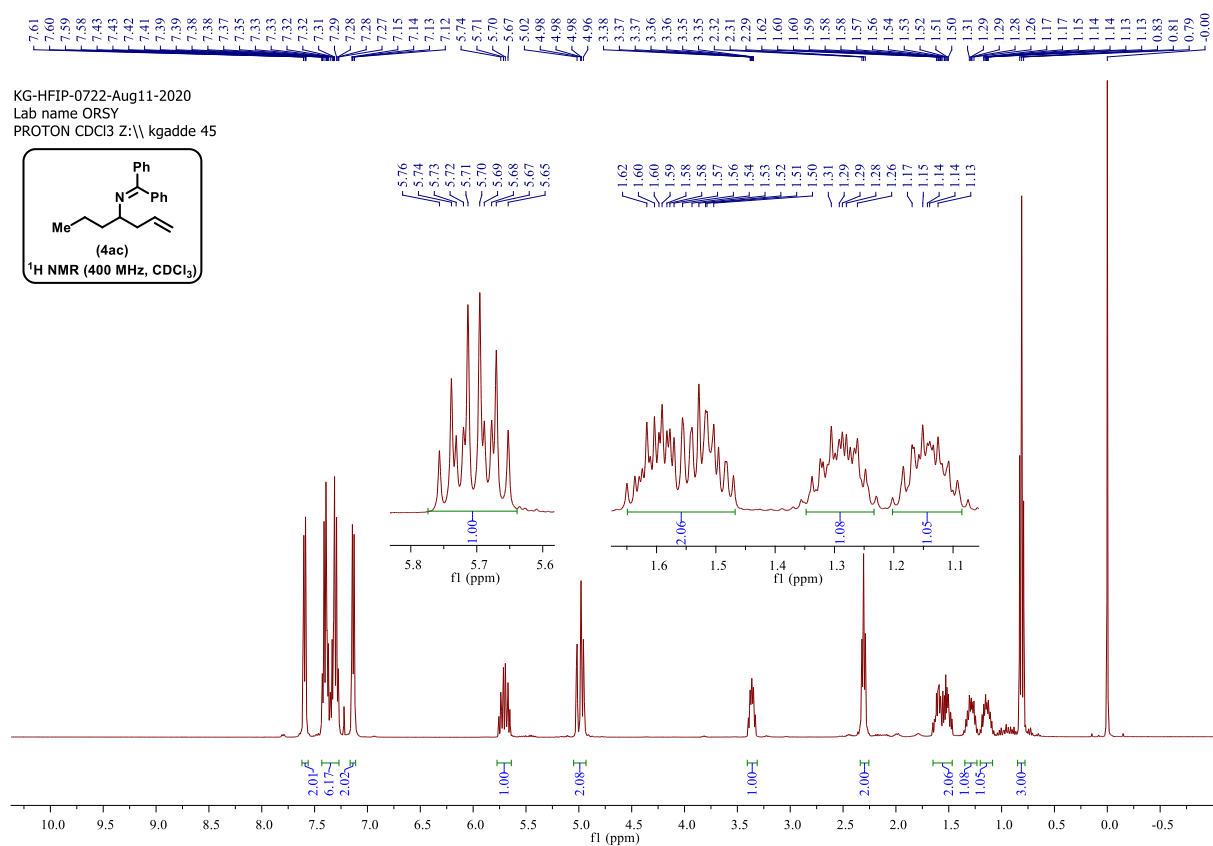


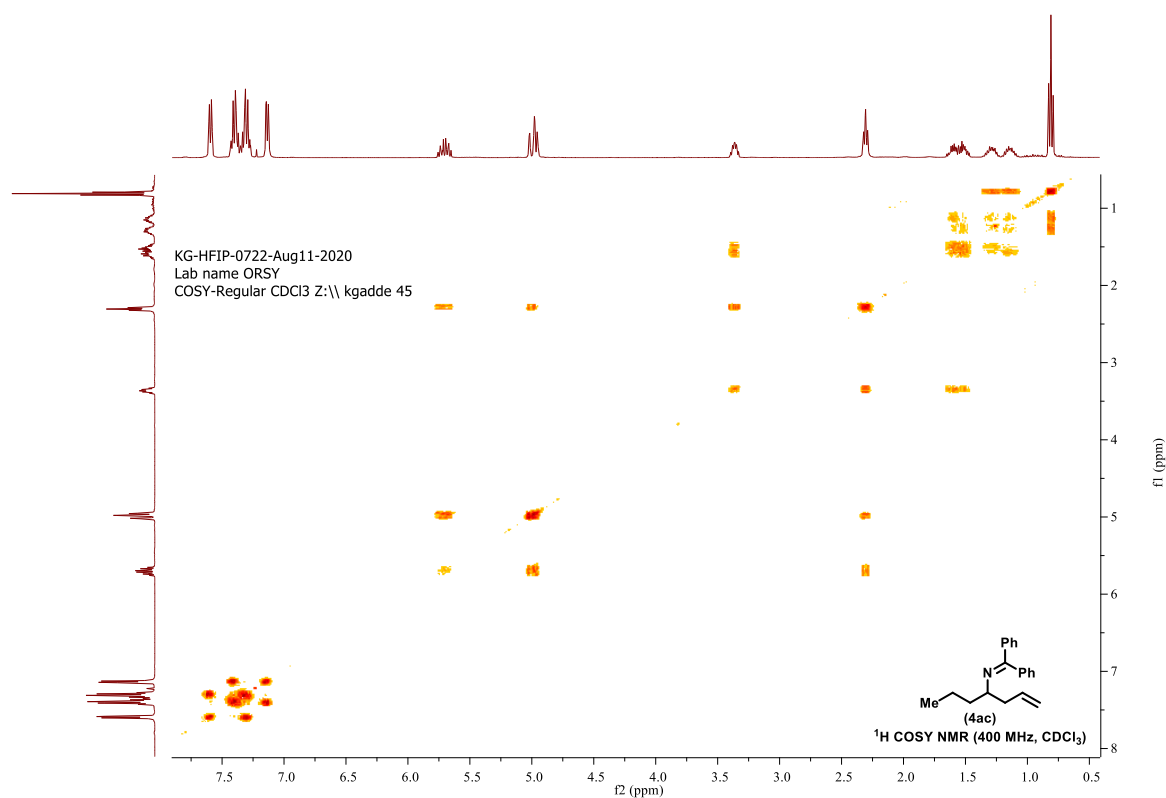
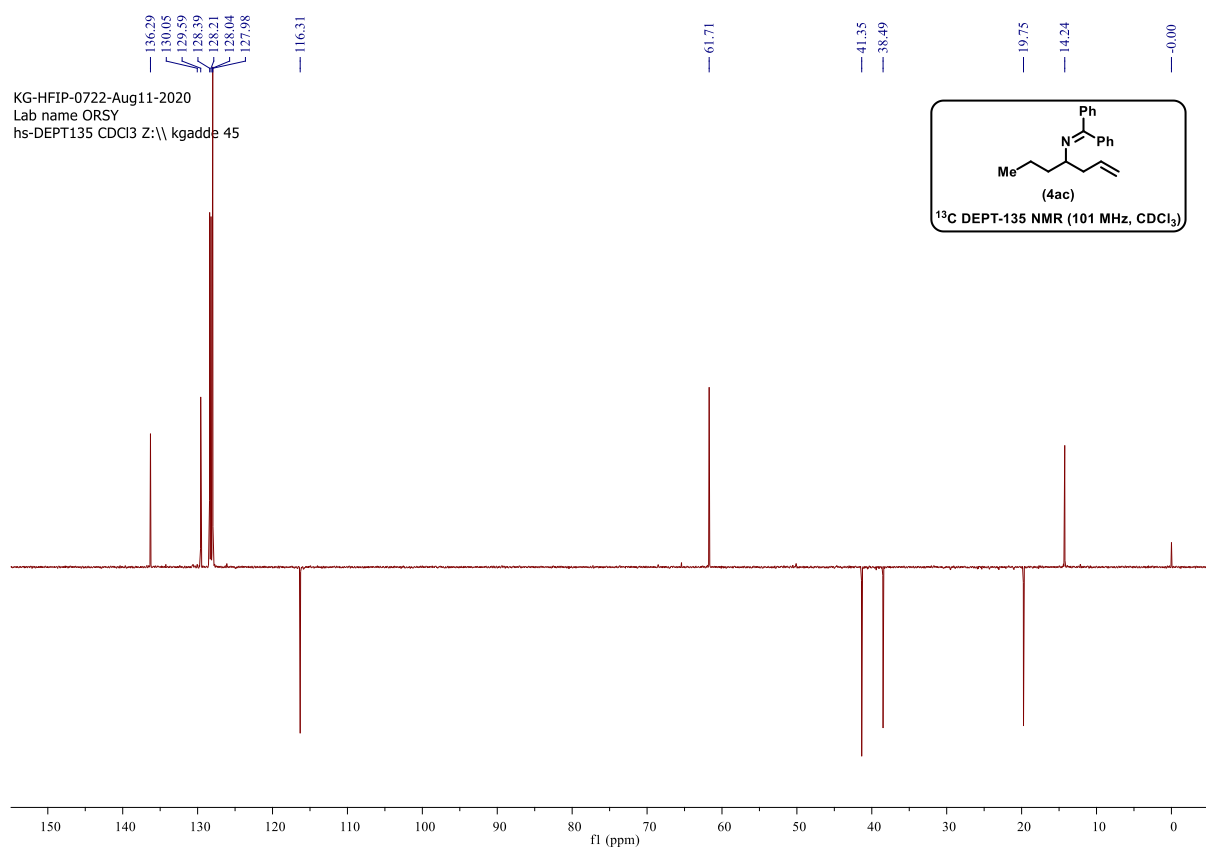
KG-HFIP-0727-Aug04-2020
 Lab name ORSY
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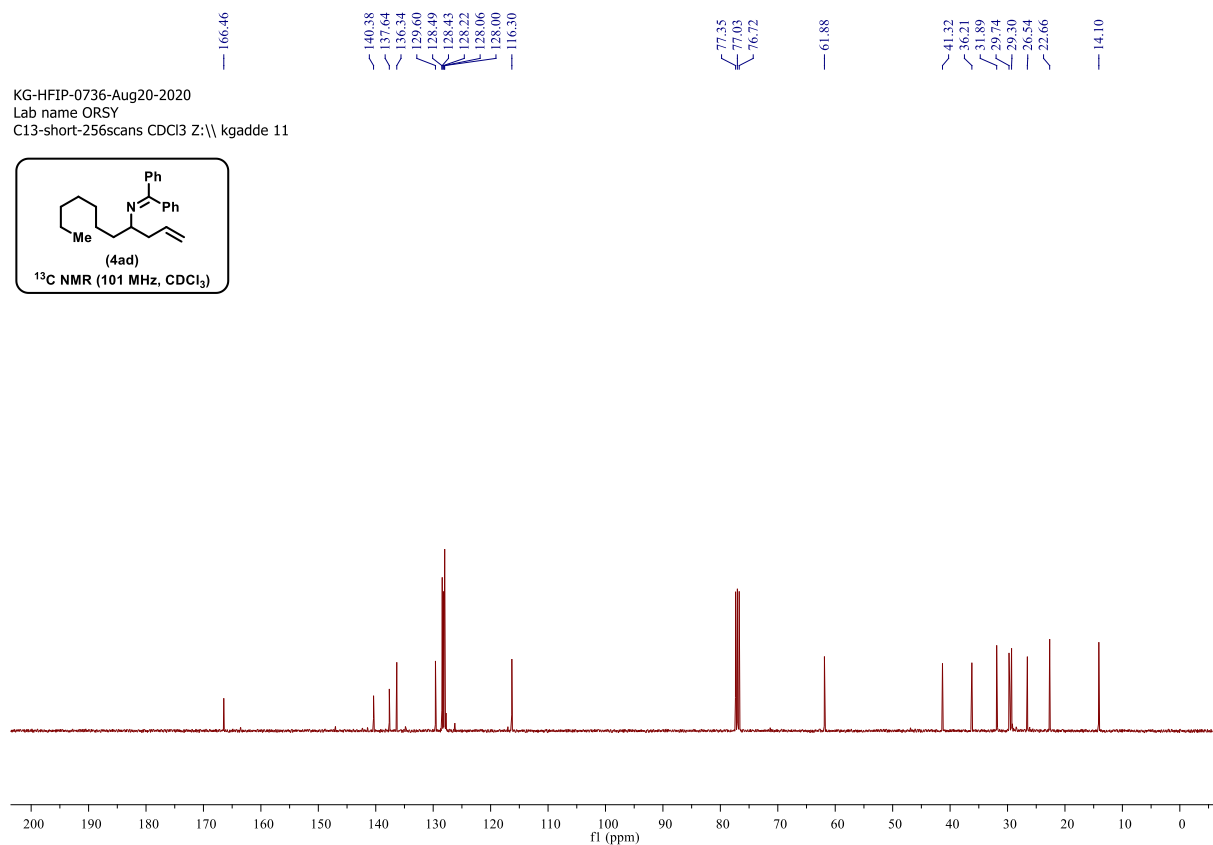
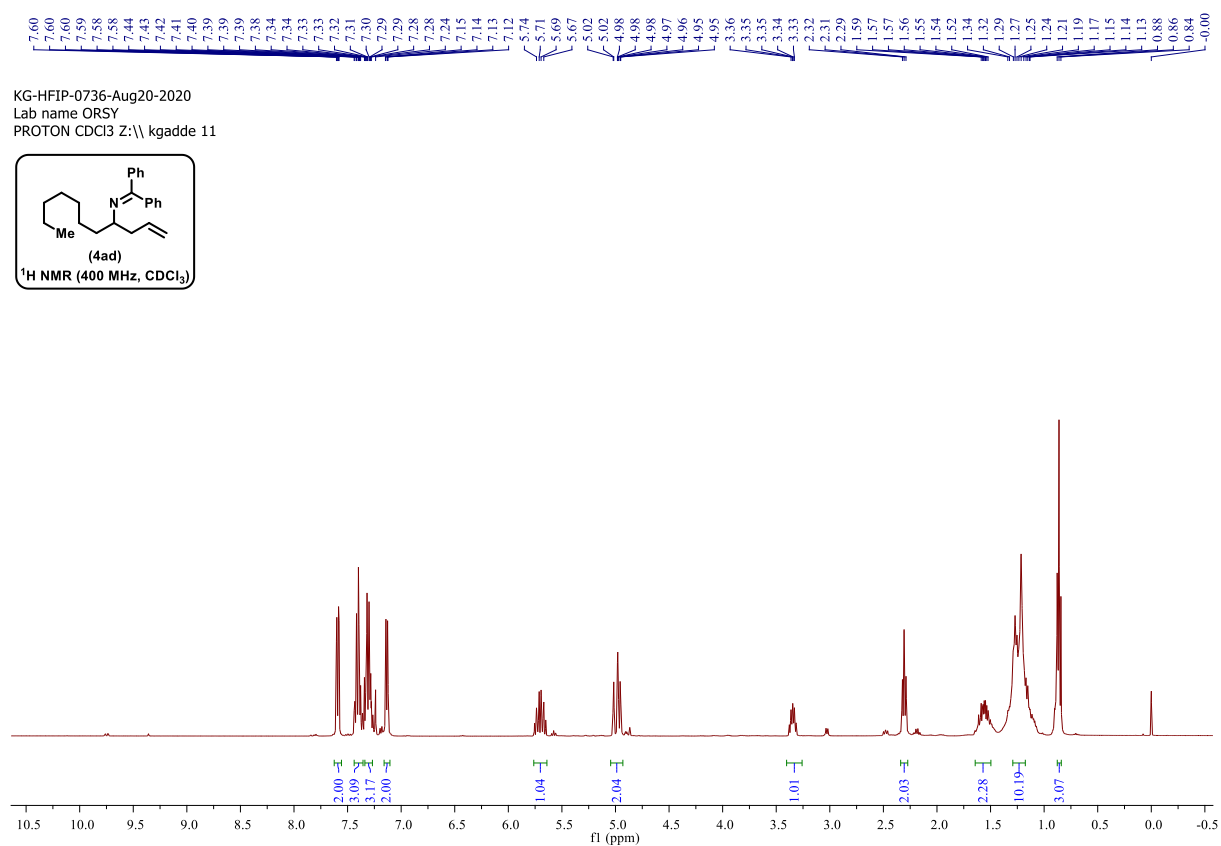


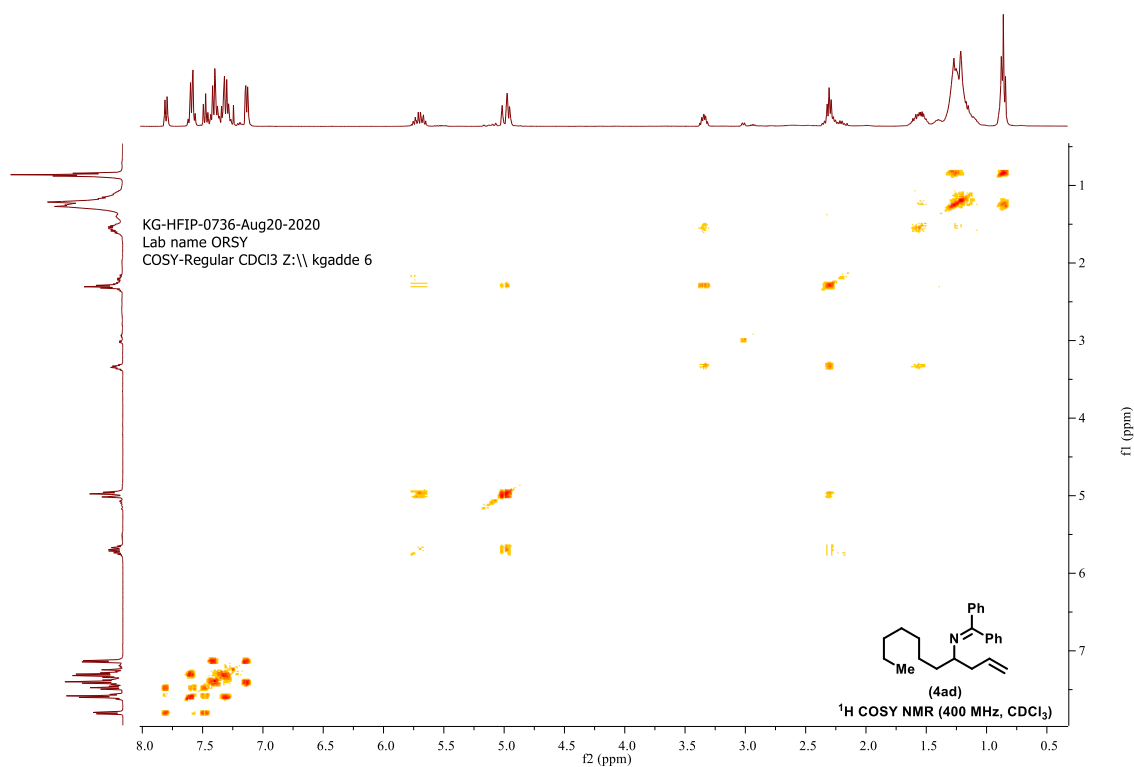
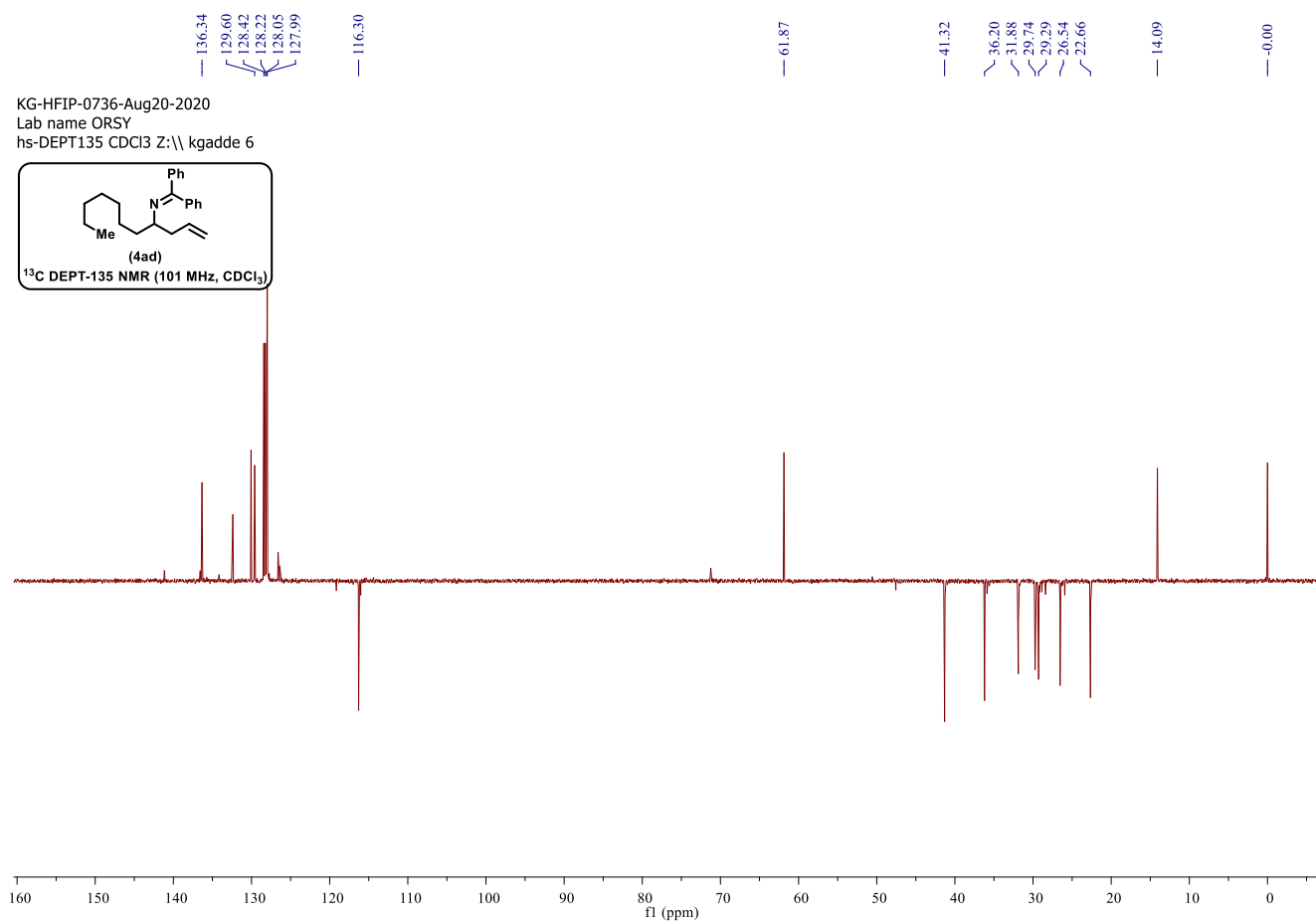


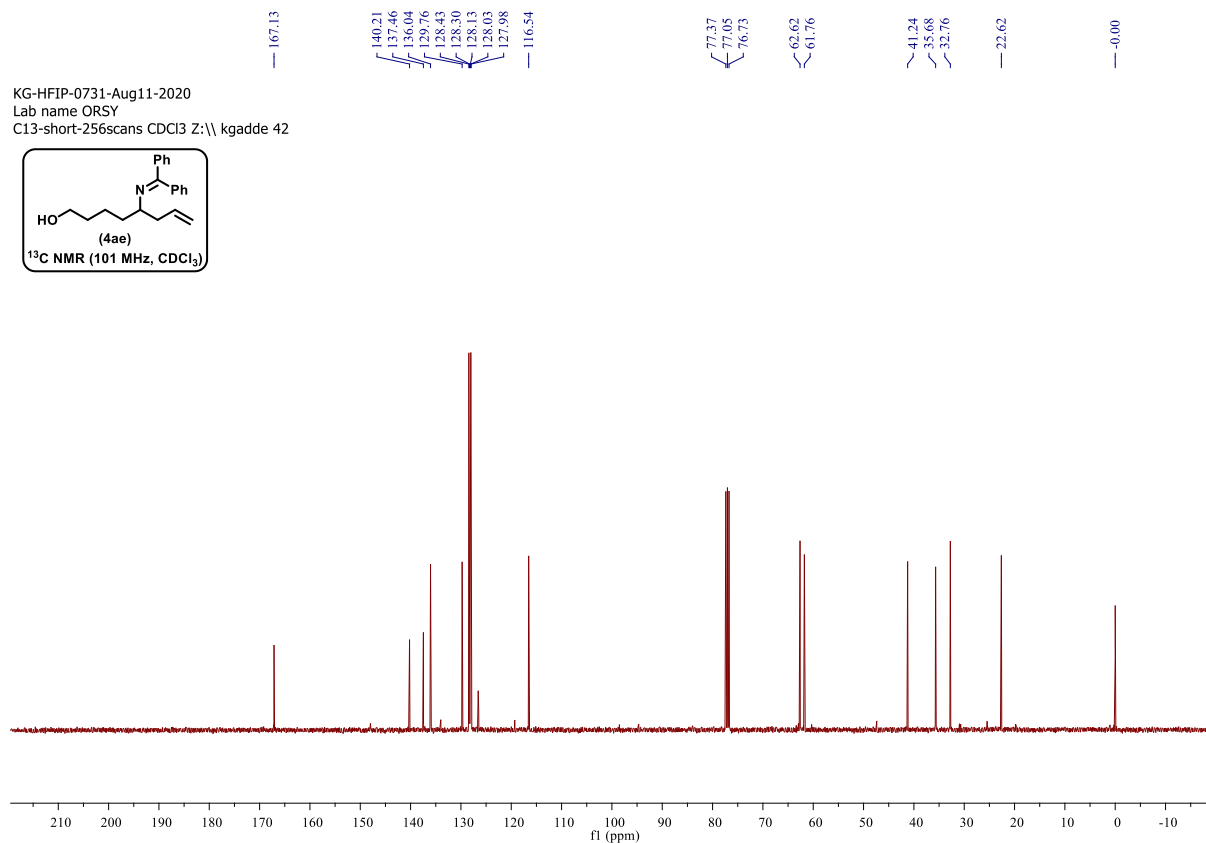
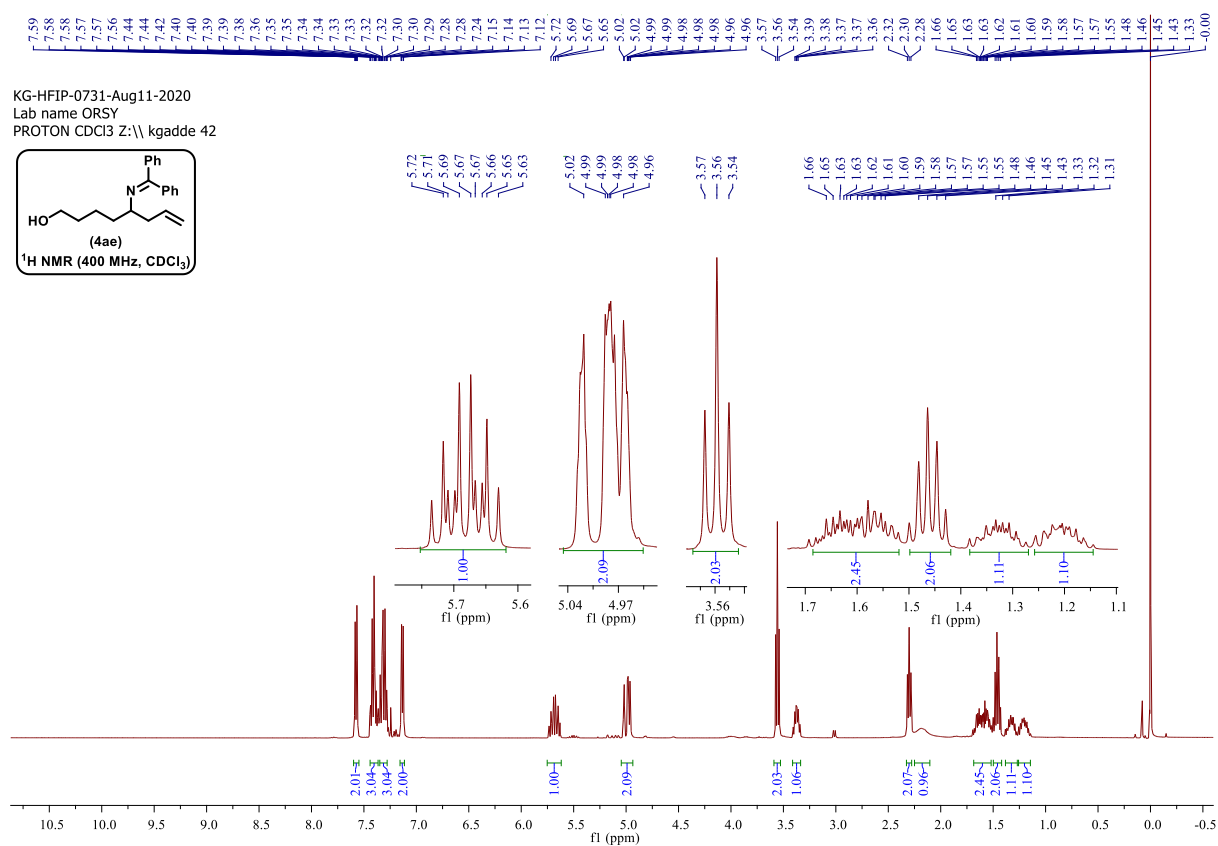


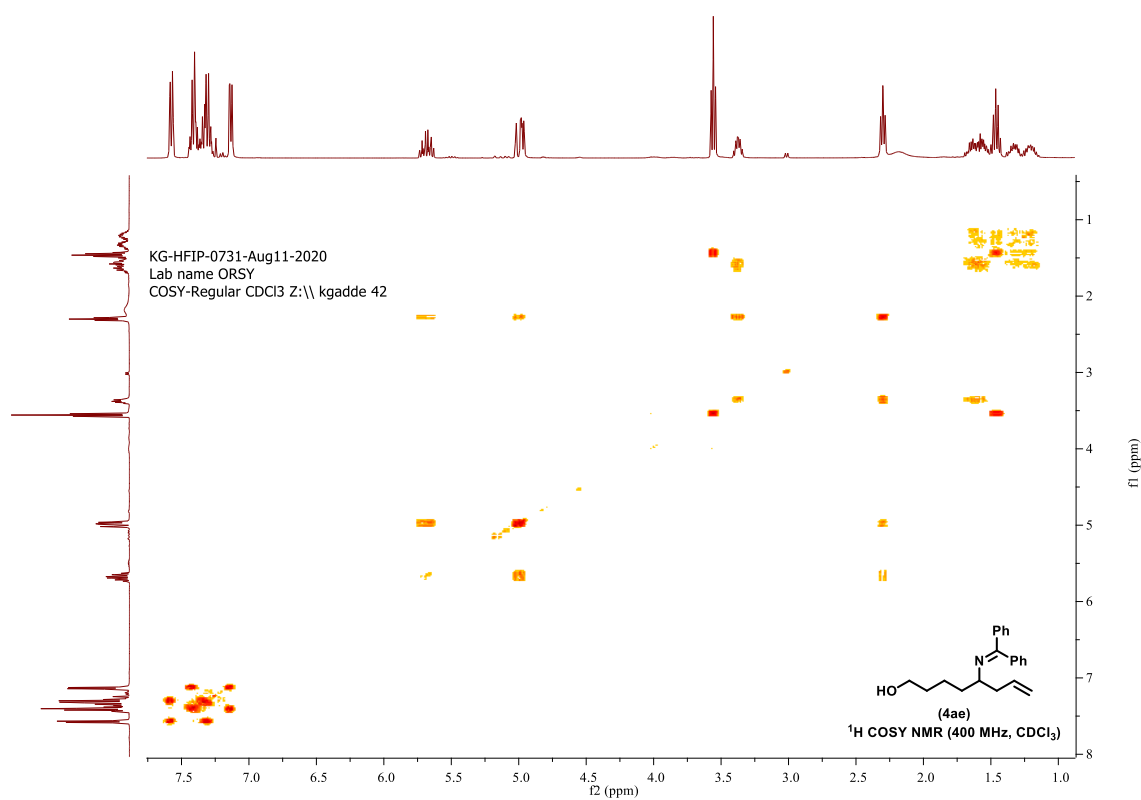
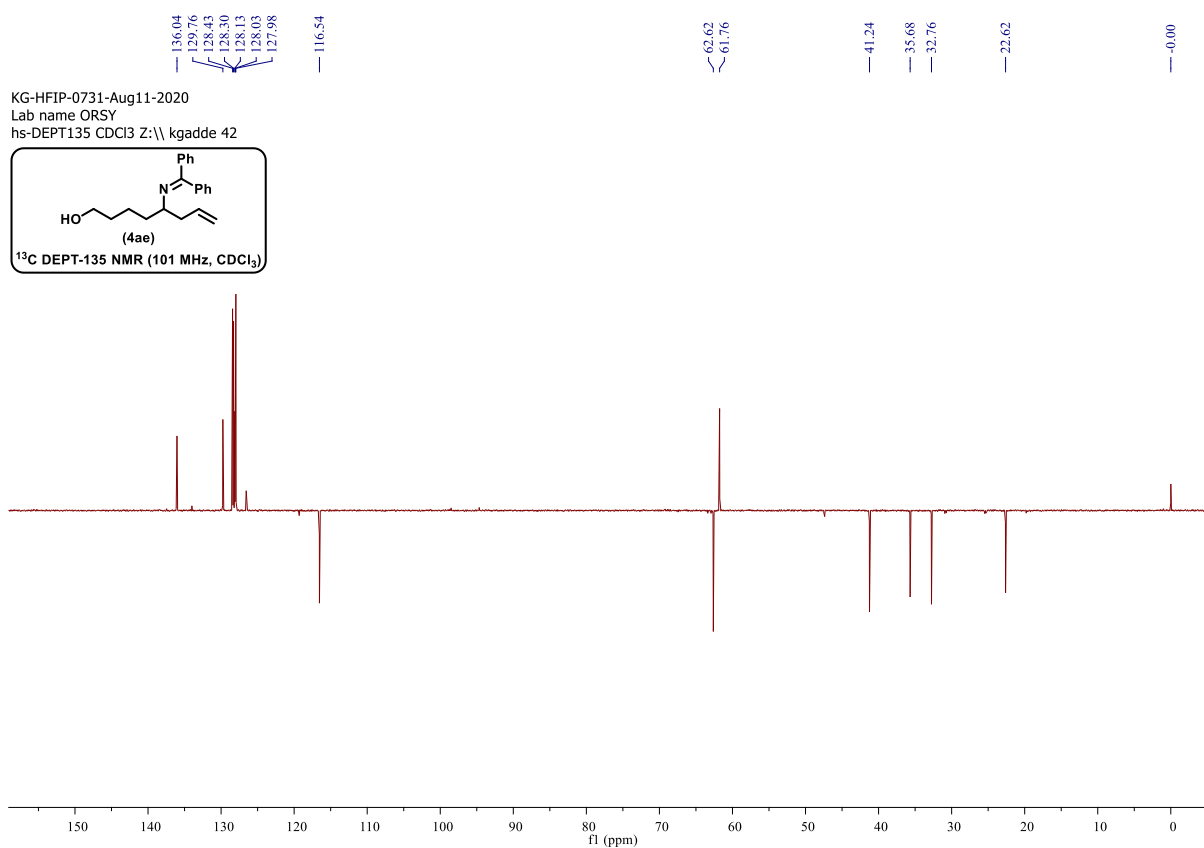


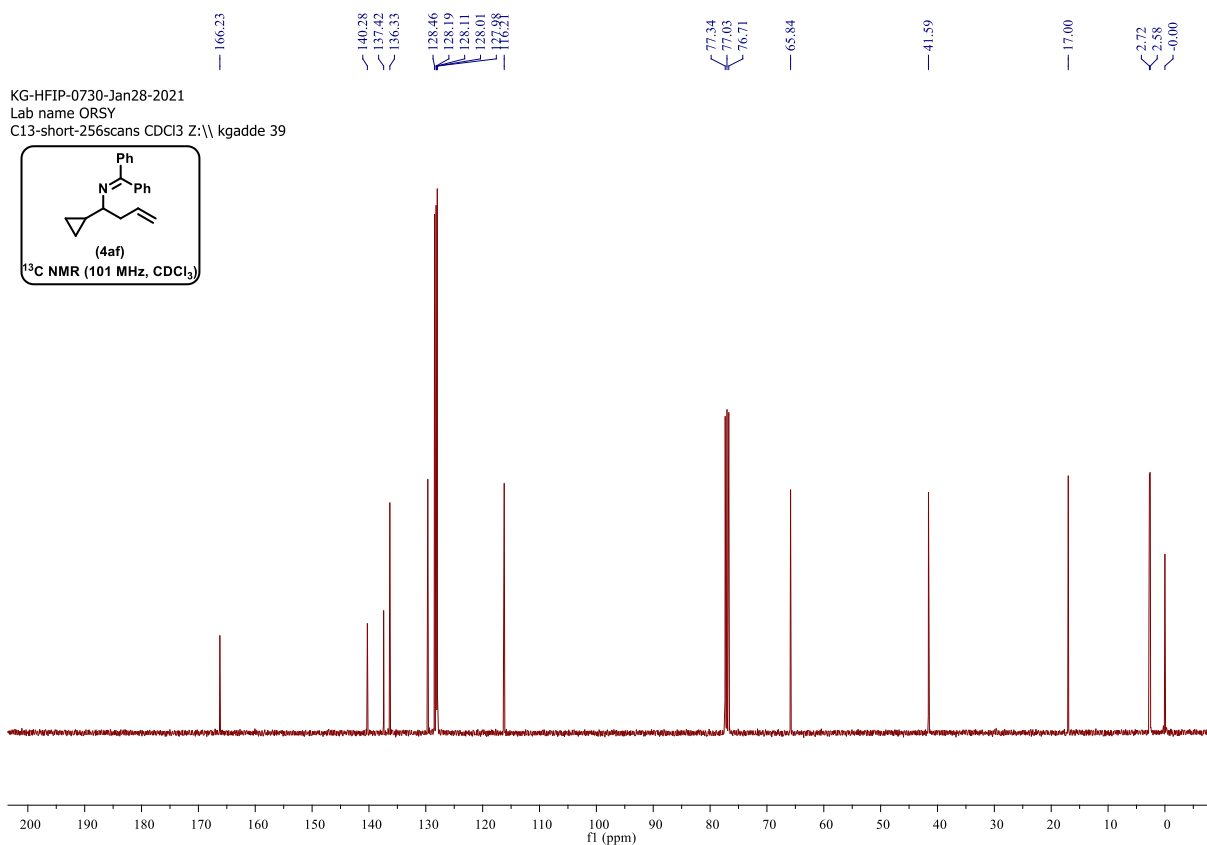
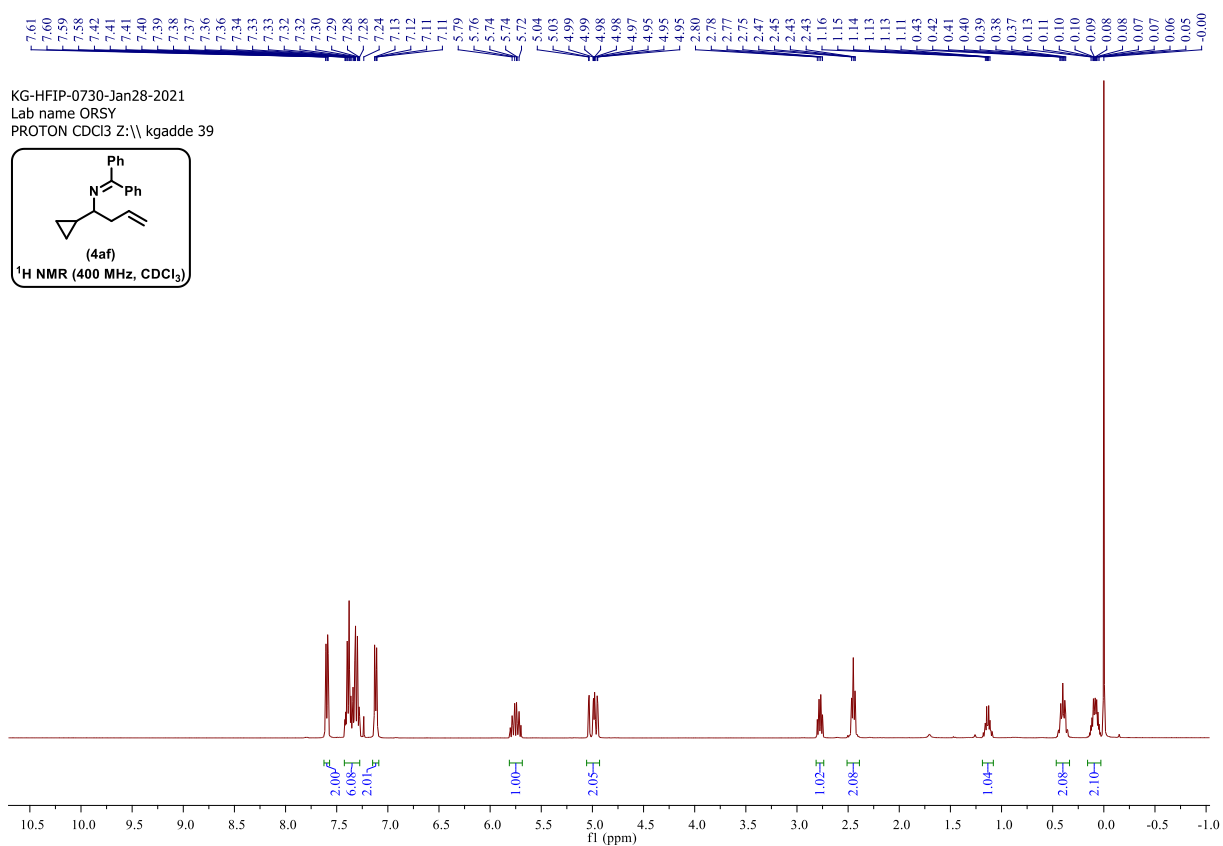


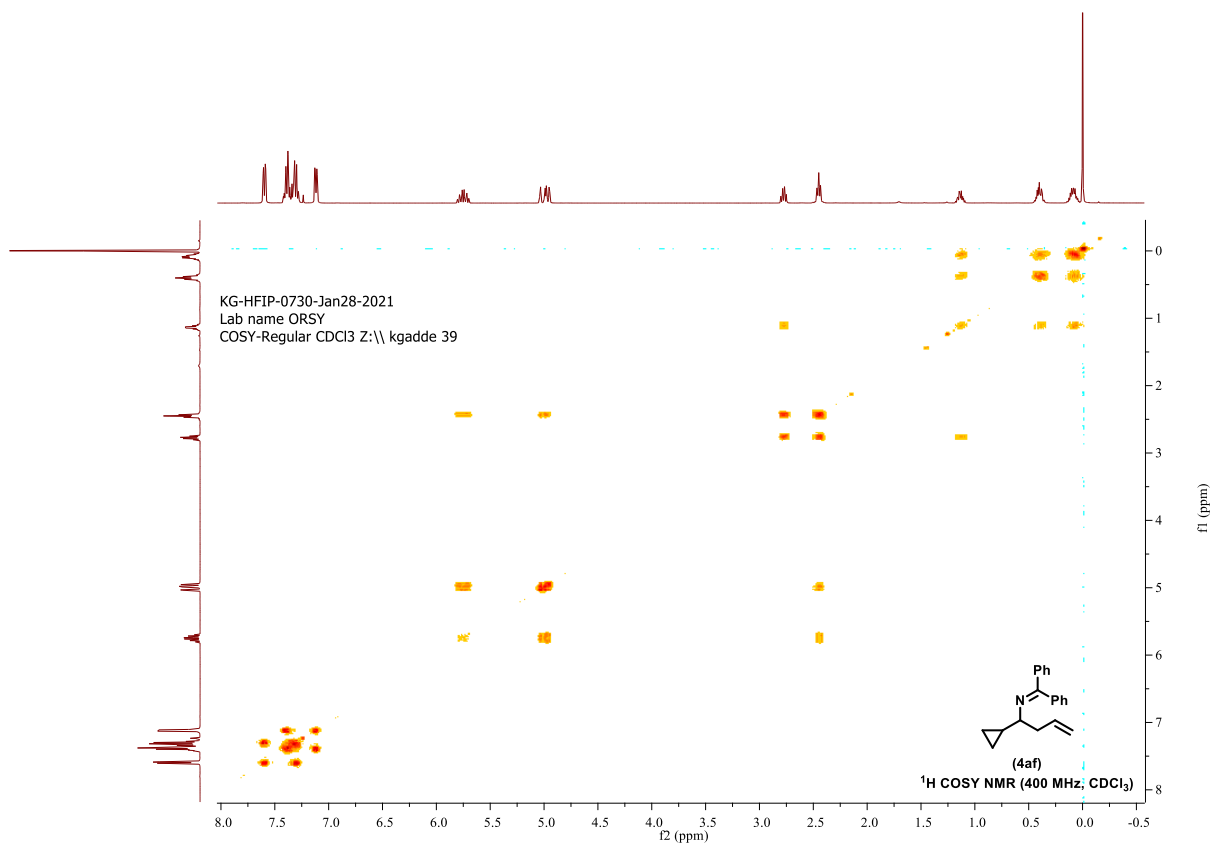
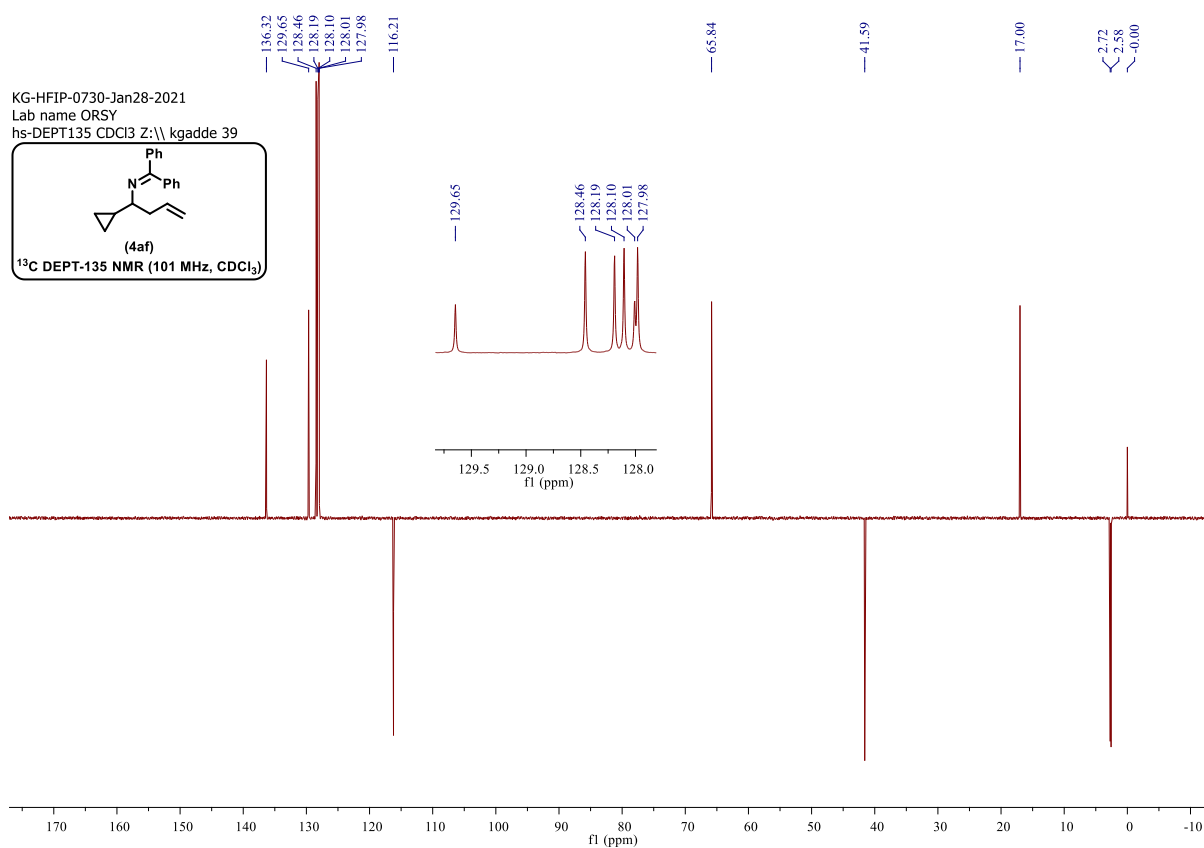


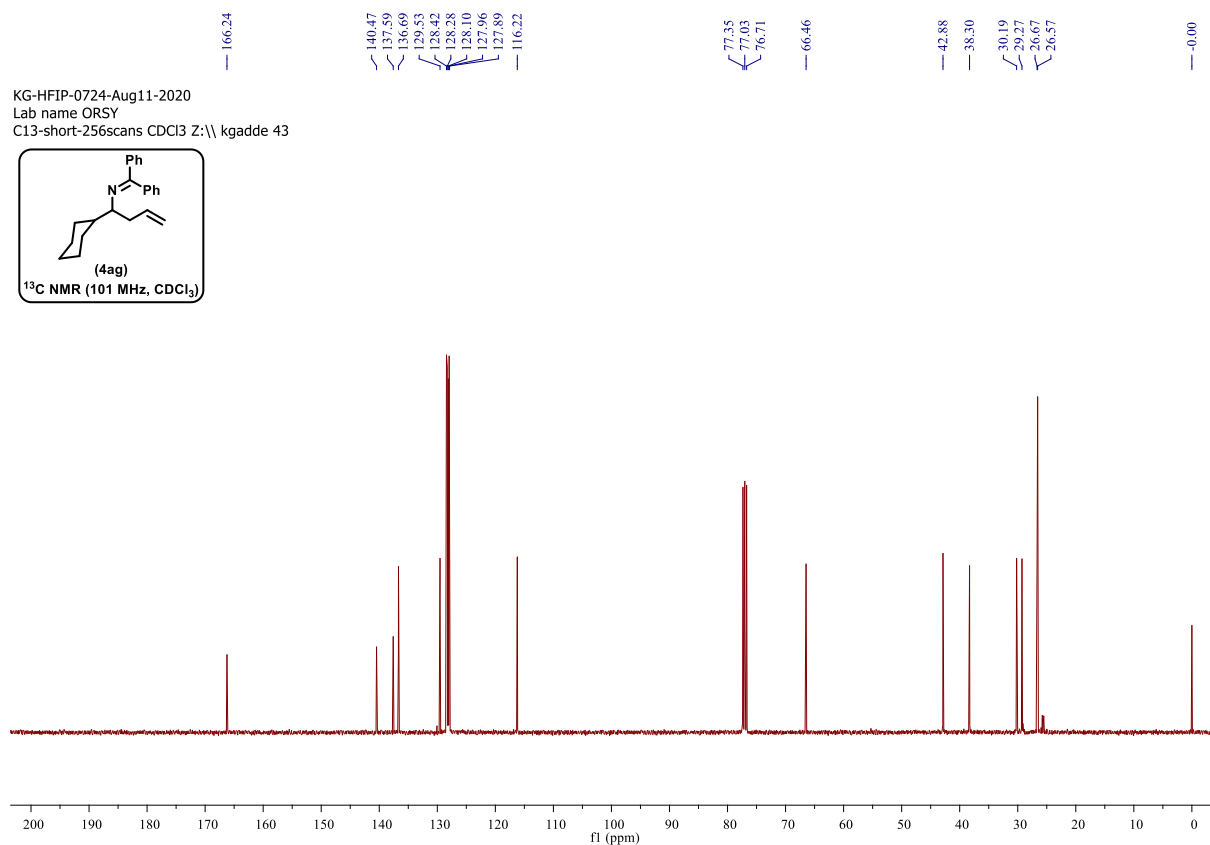
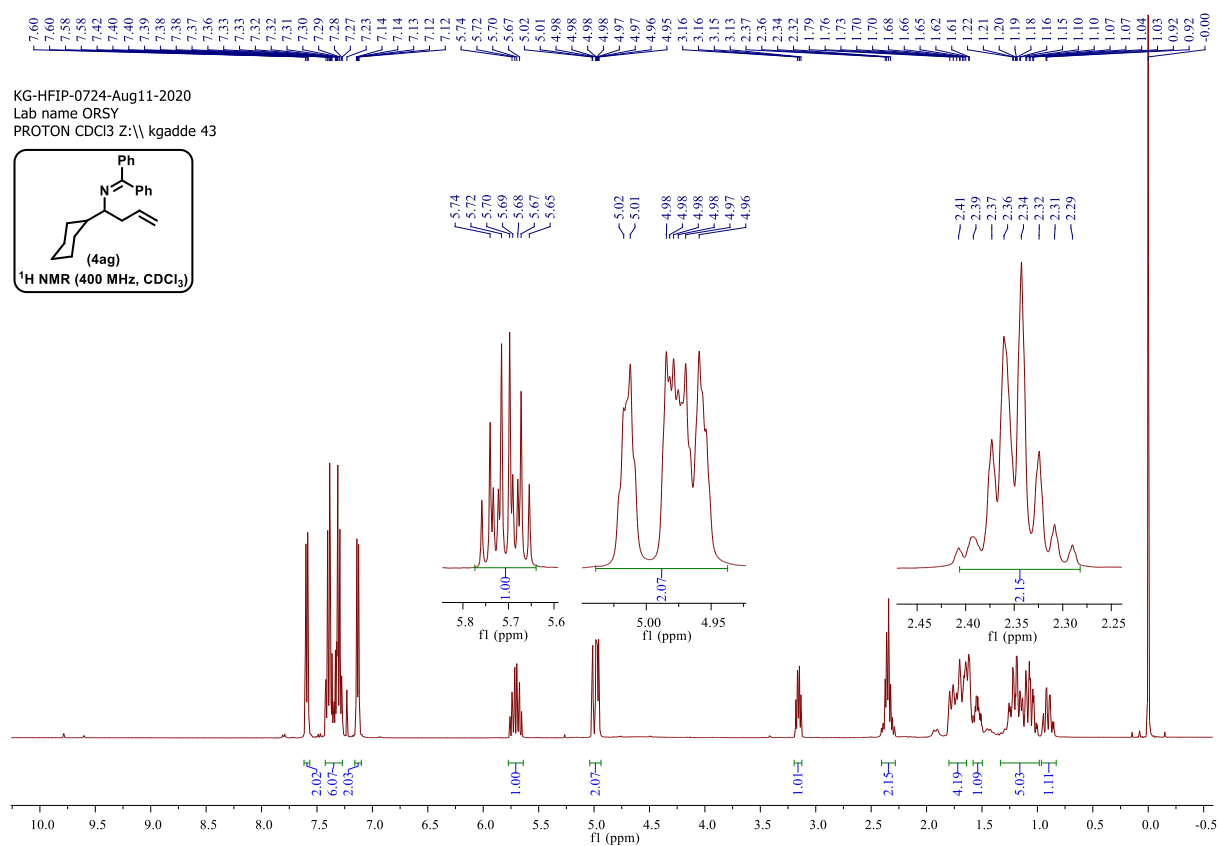




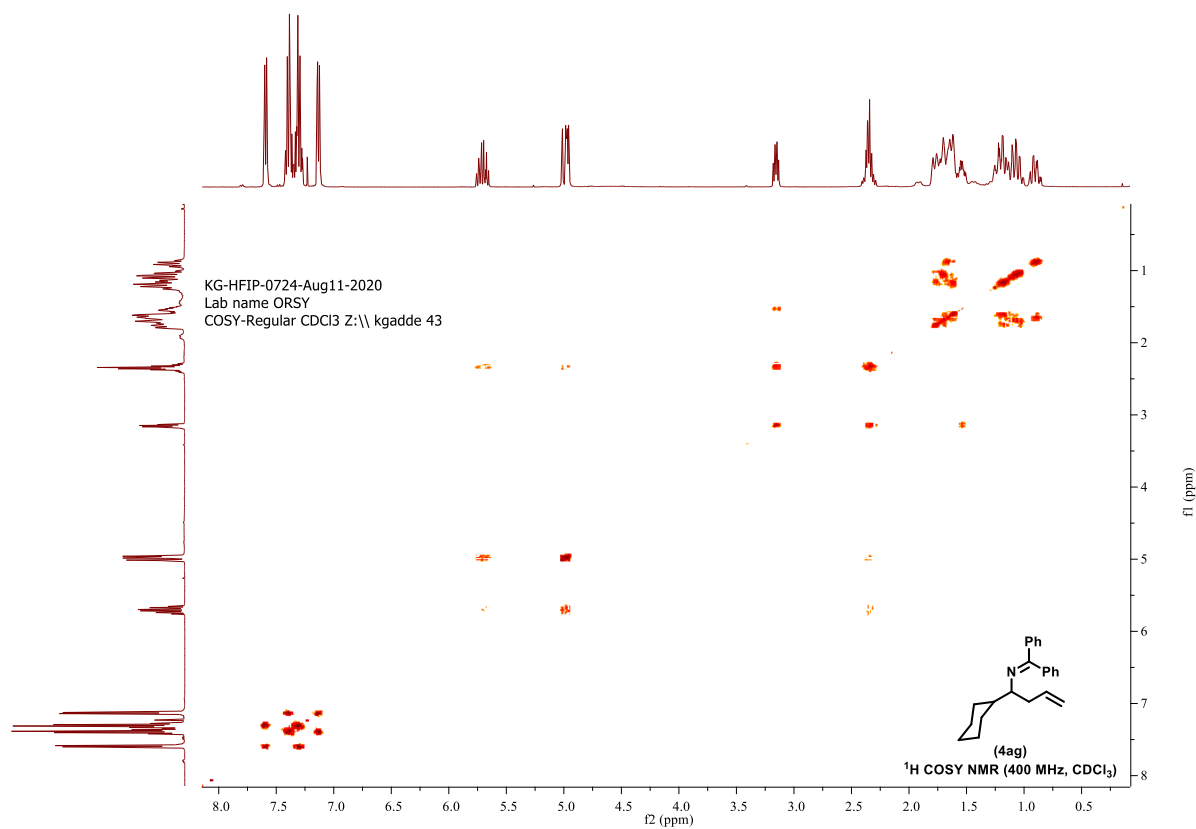
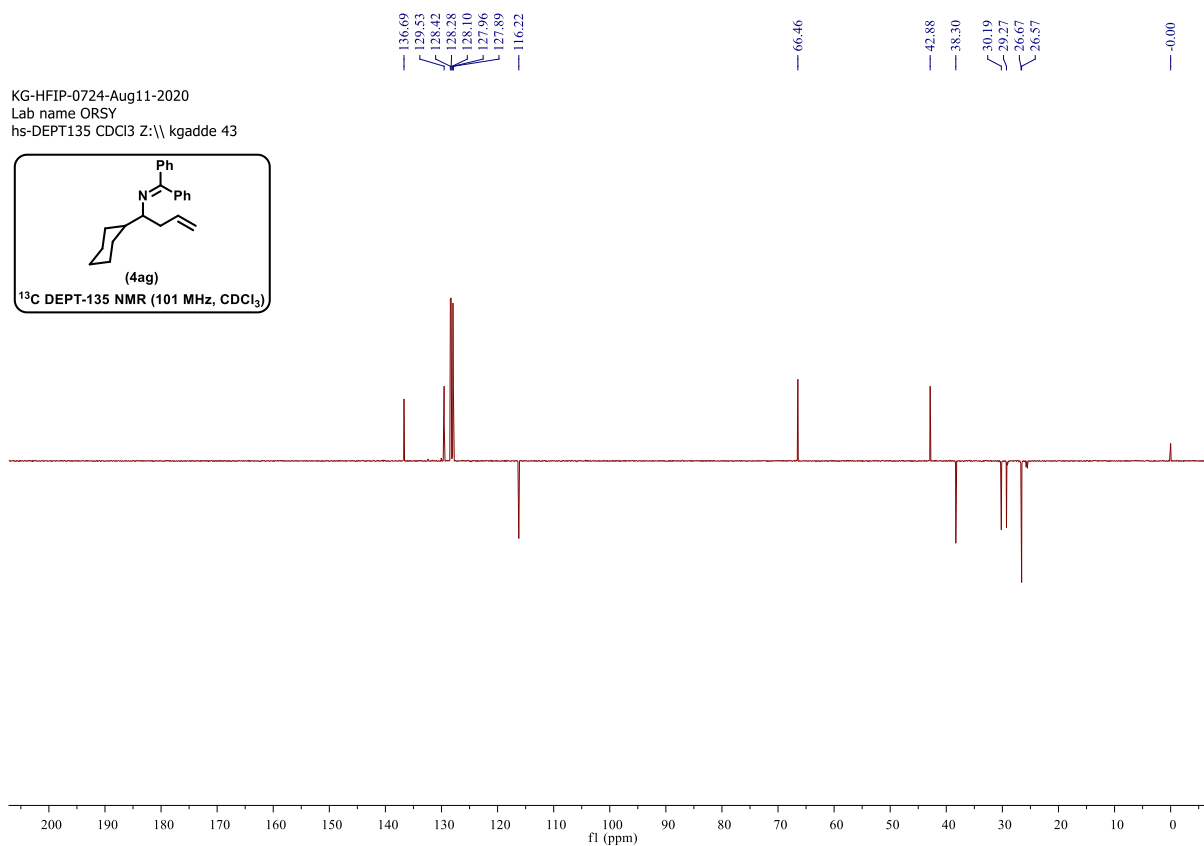
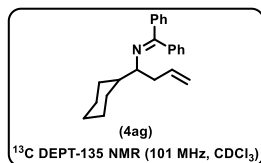


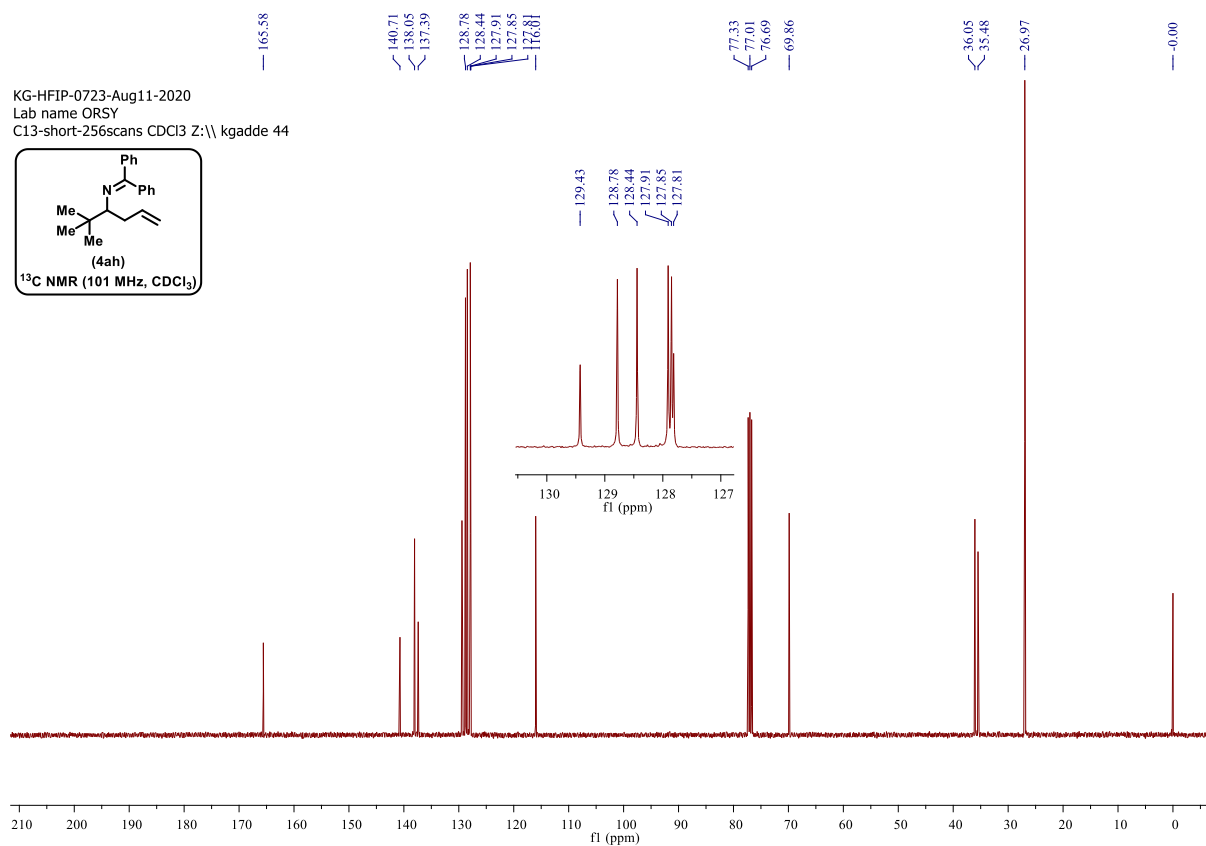
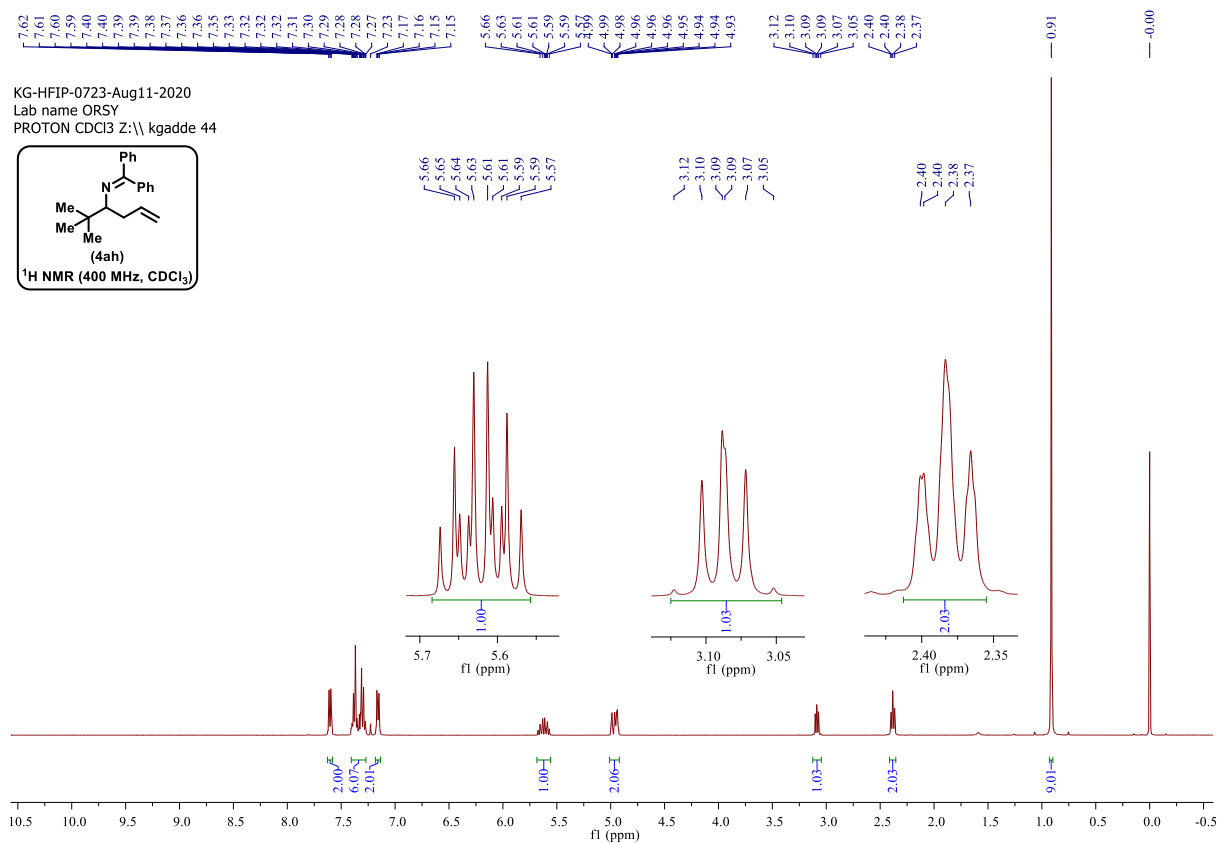


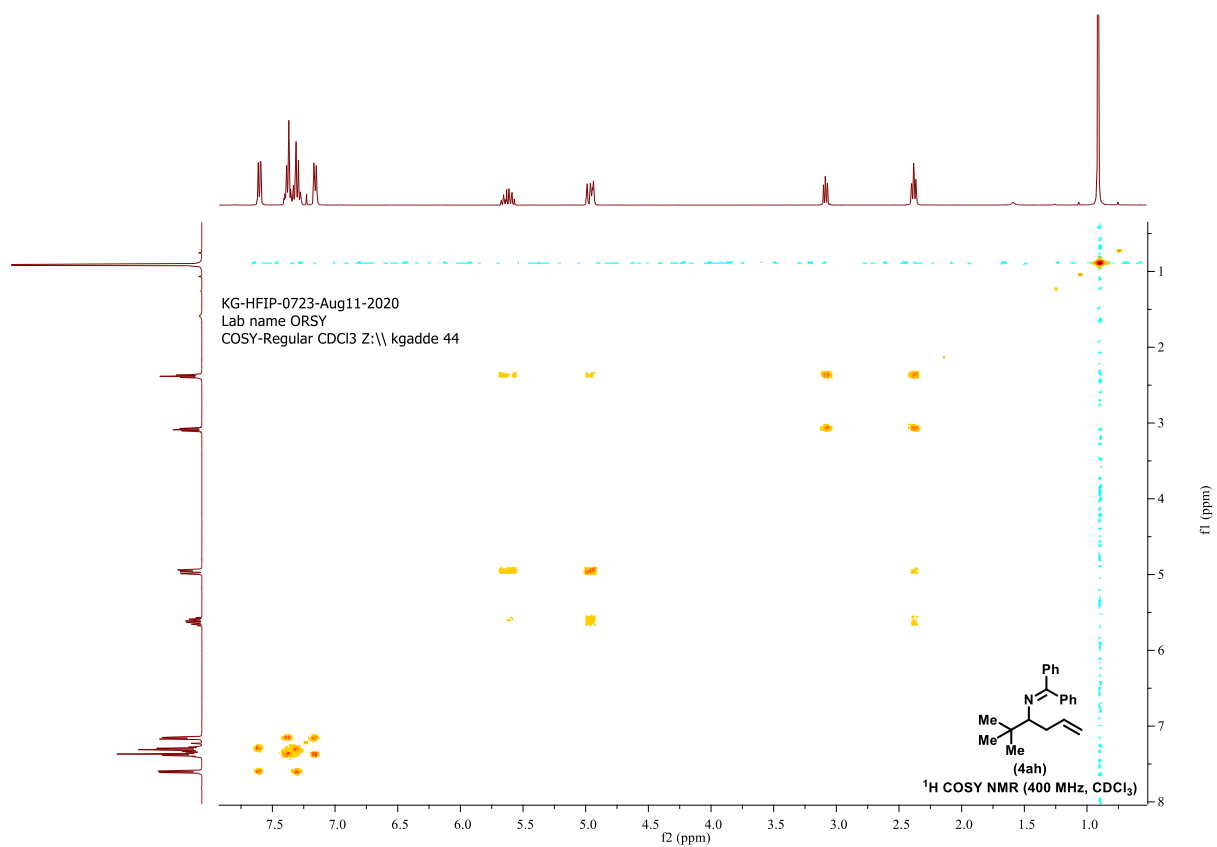
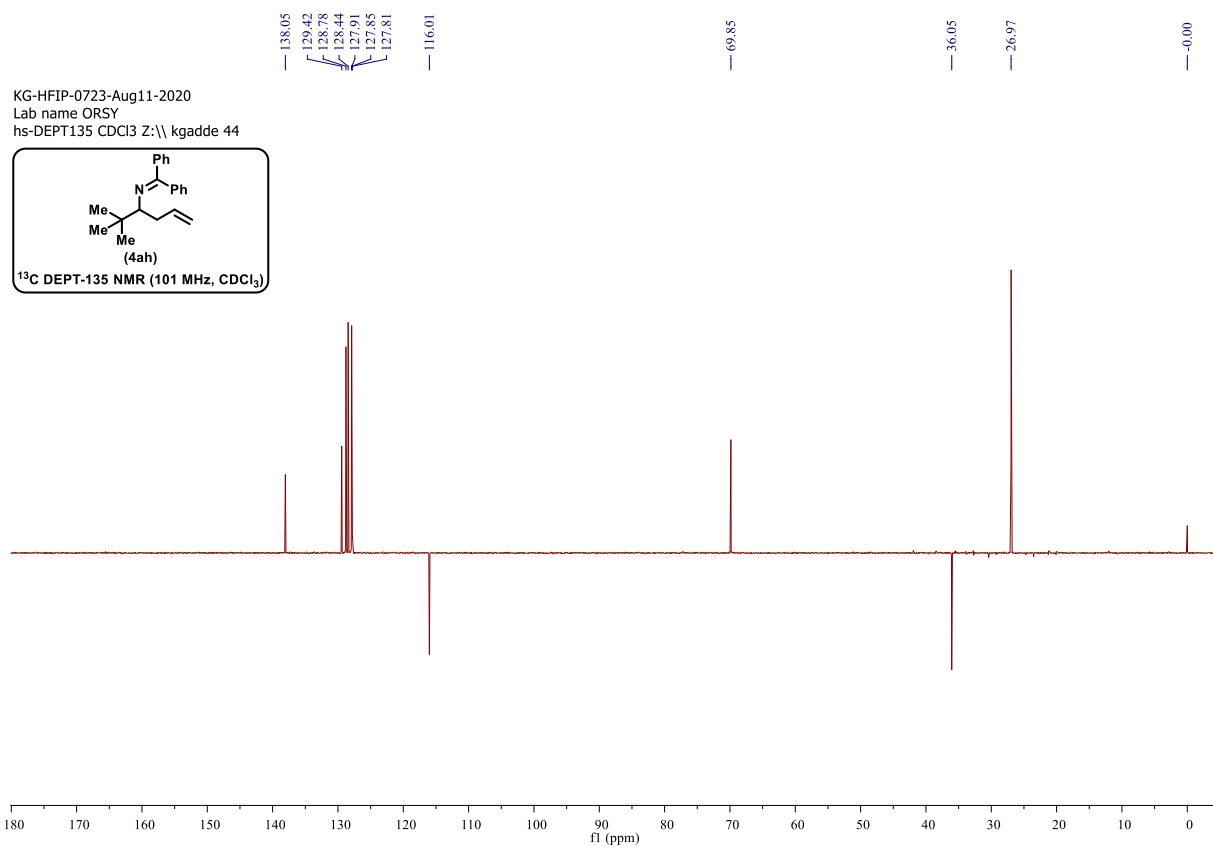


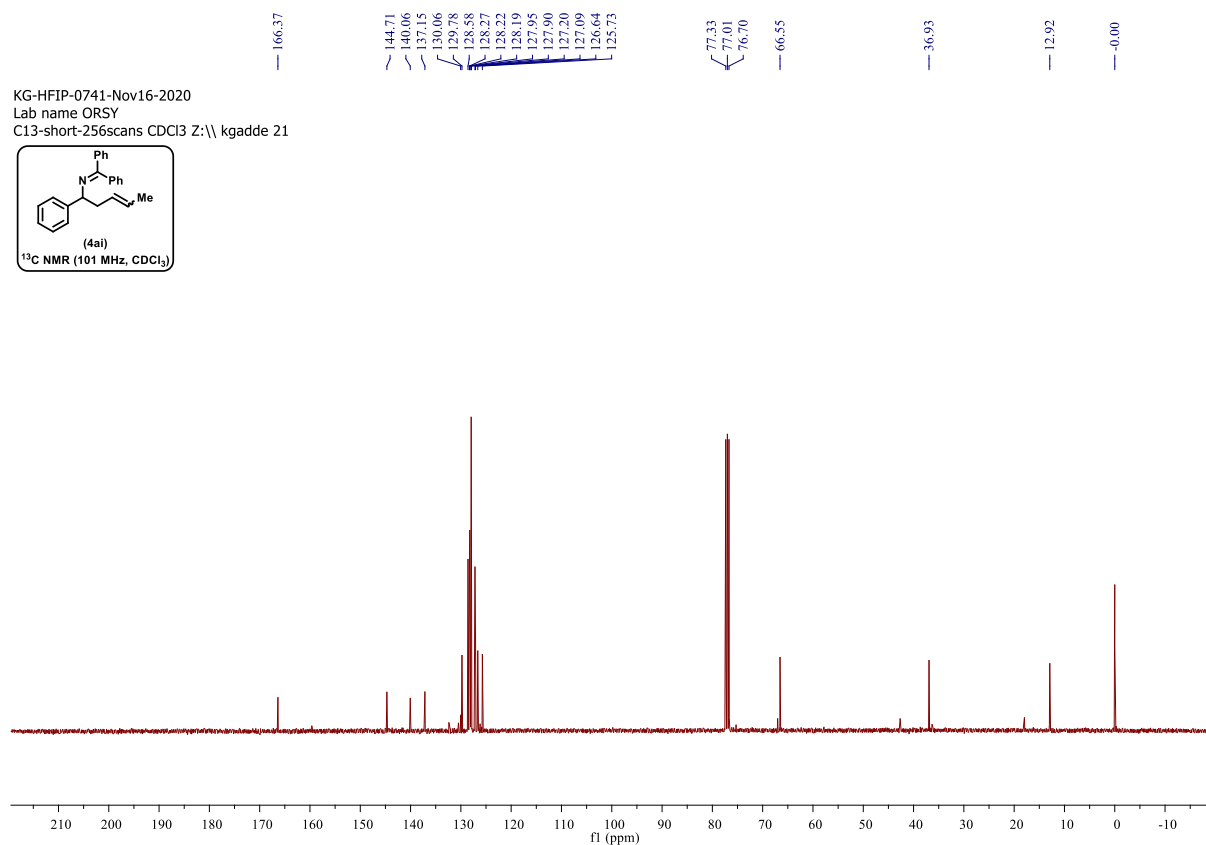
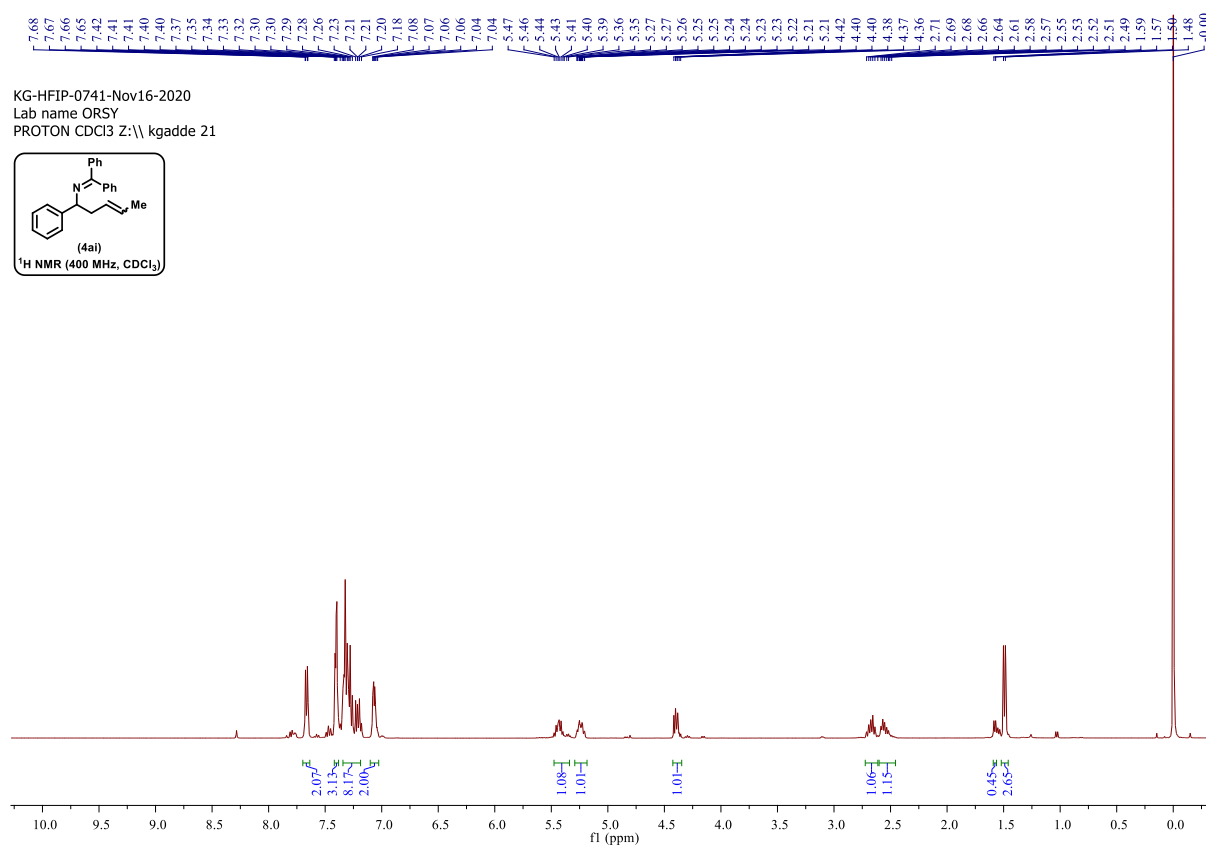


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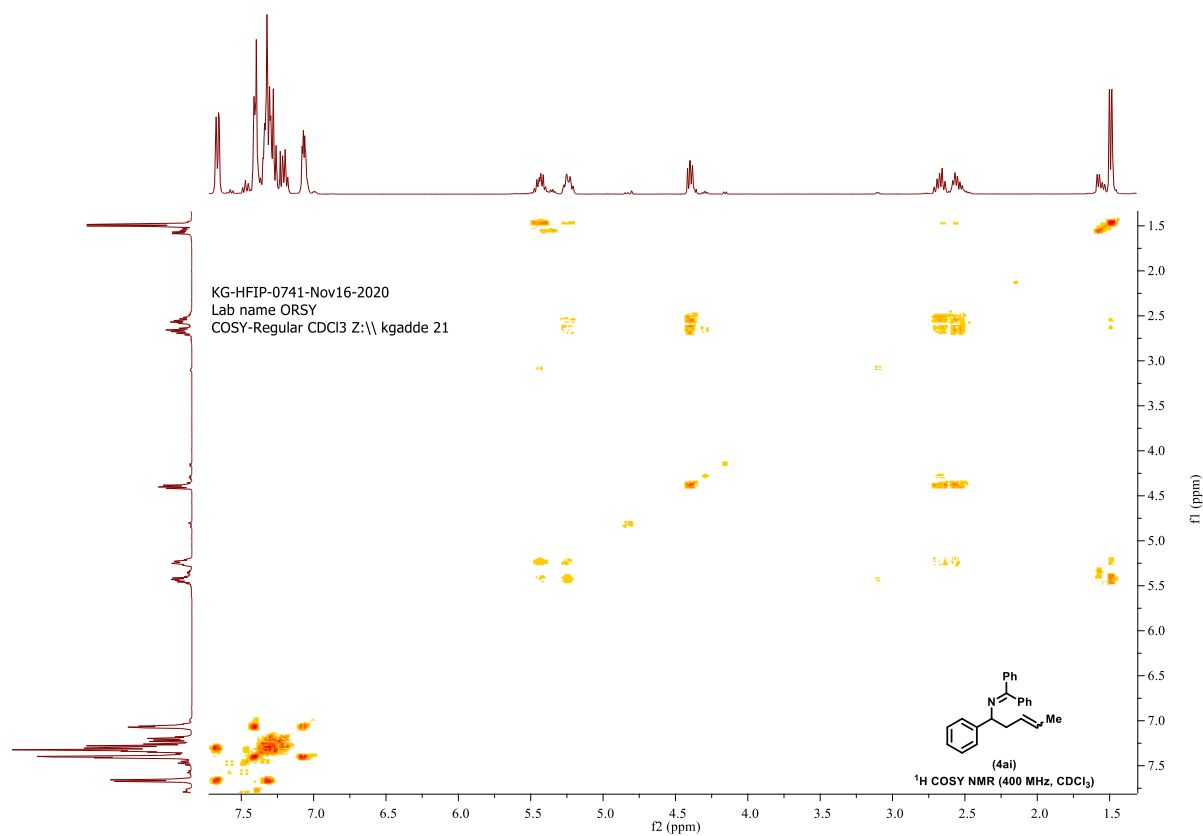
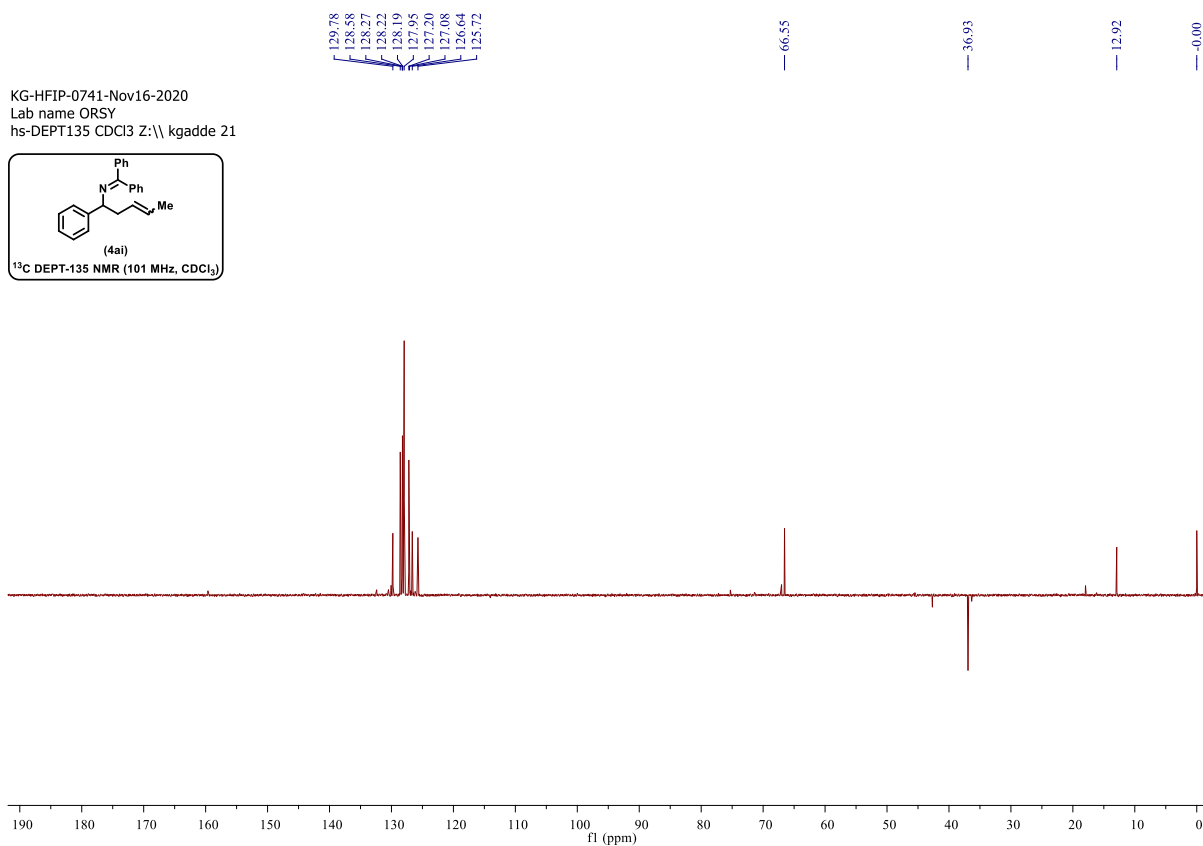
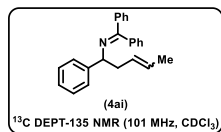


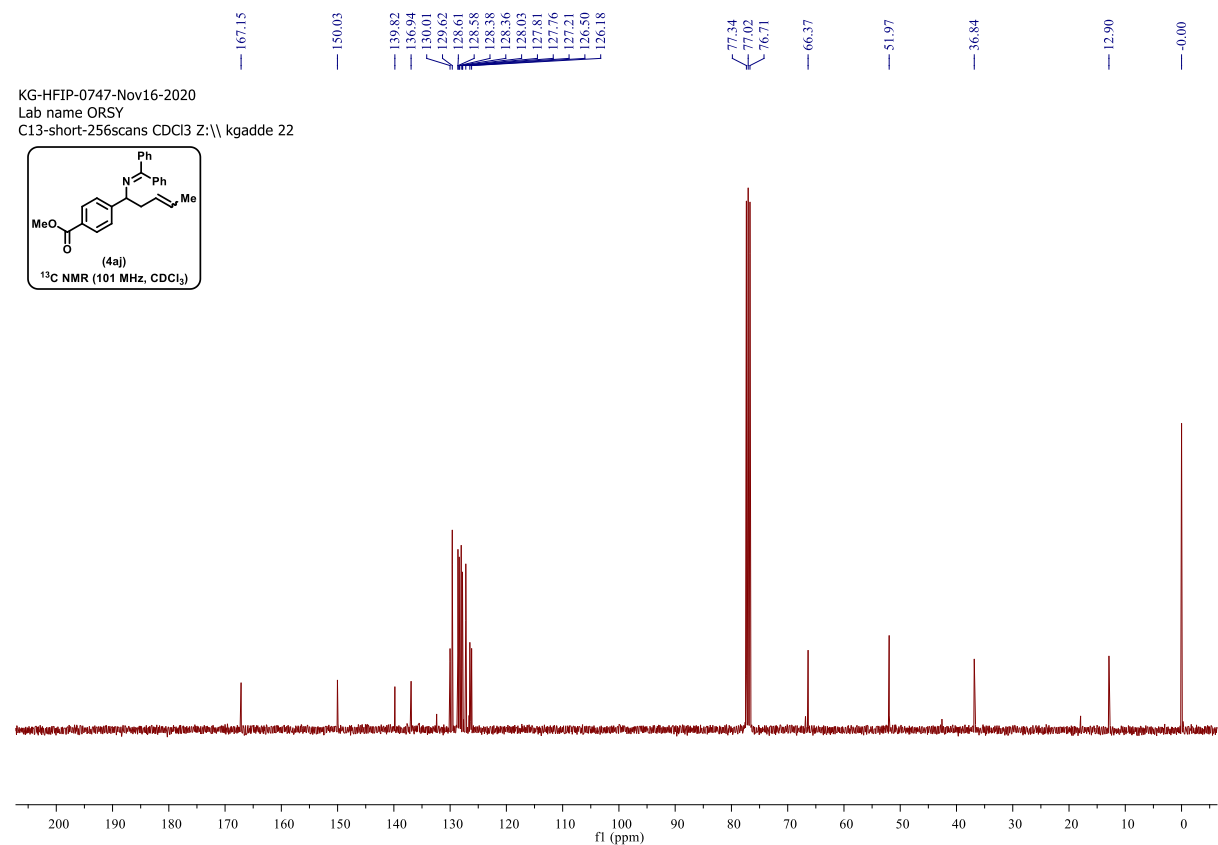
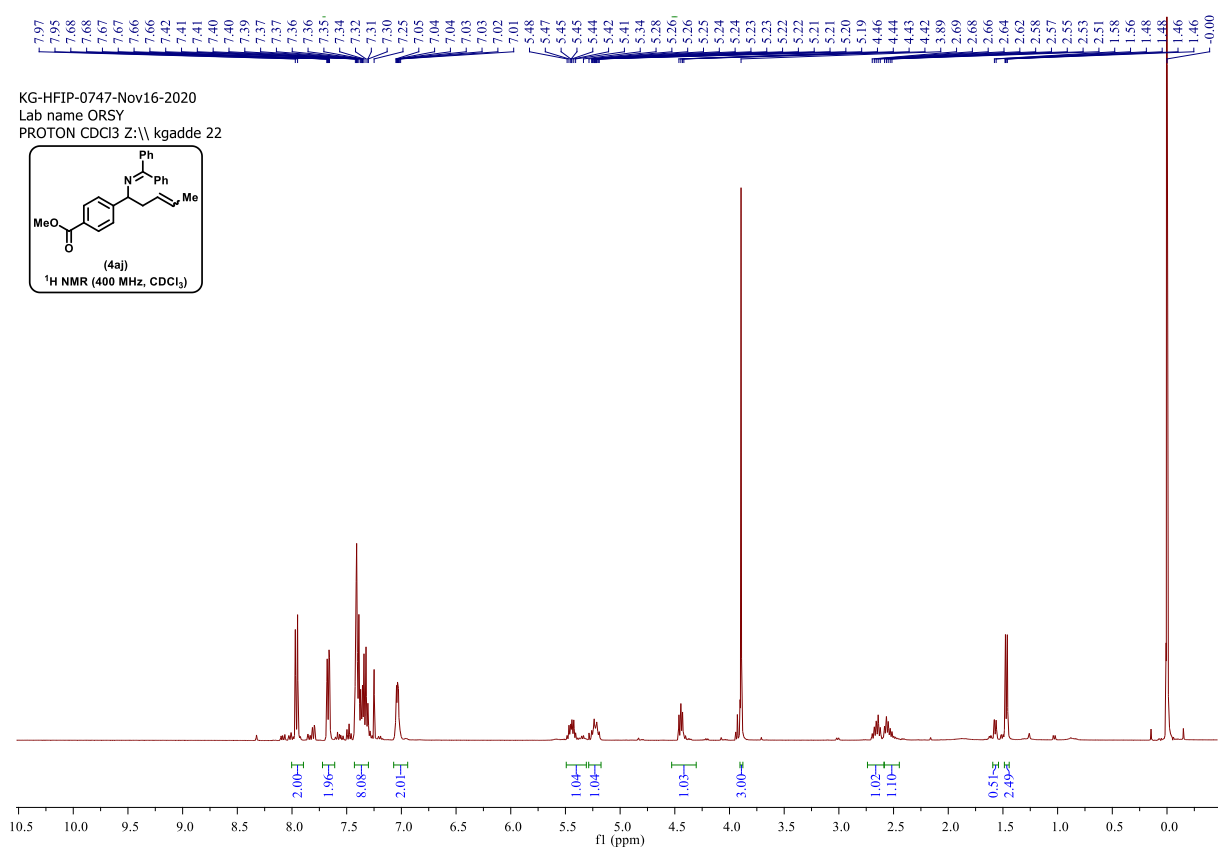




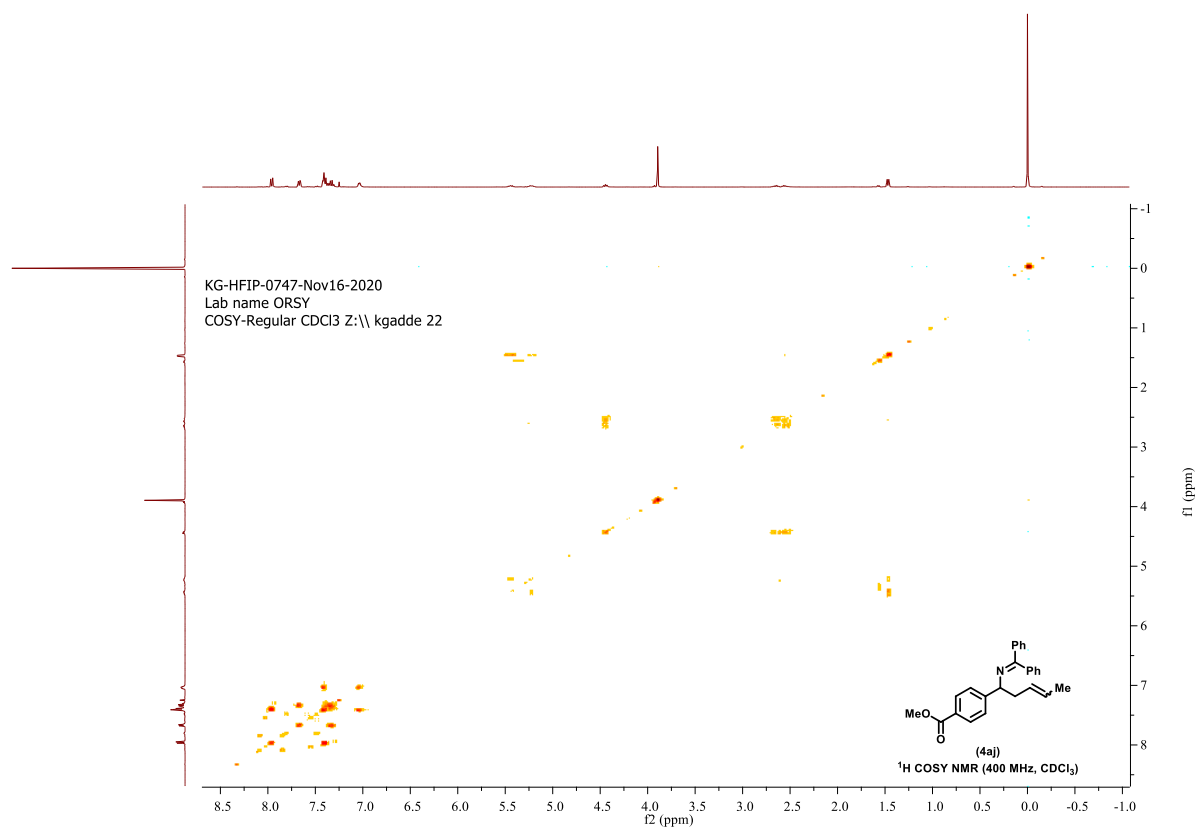
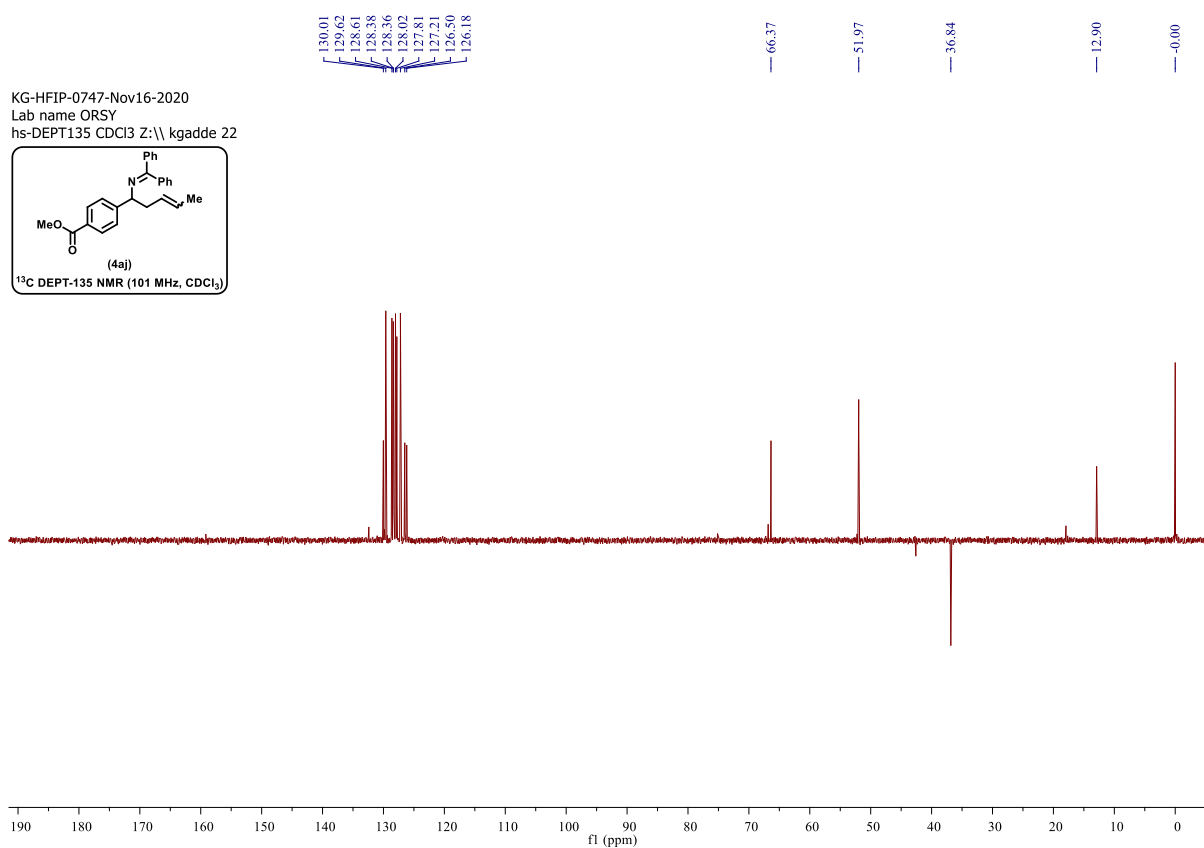
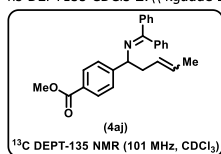


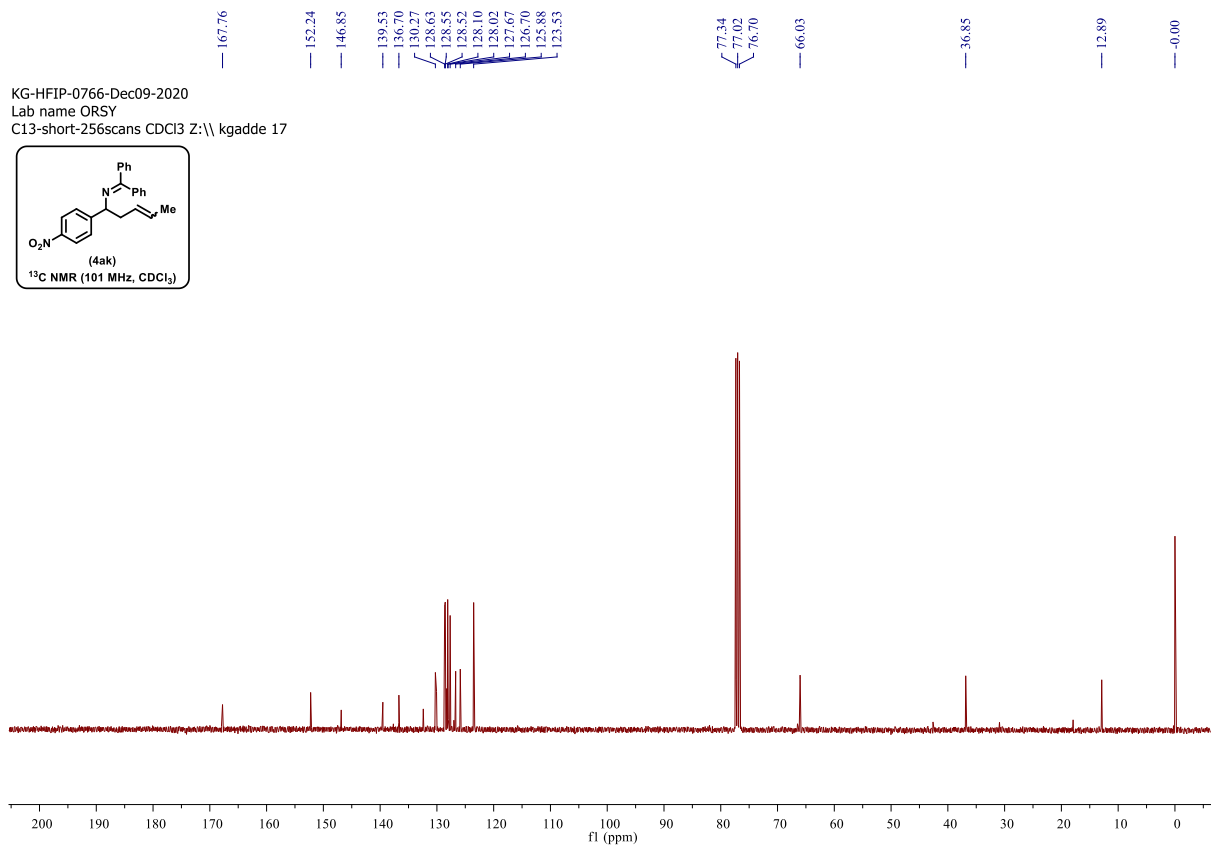
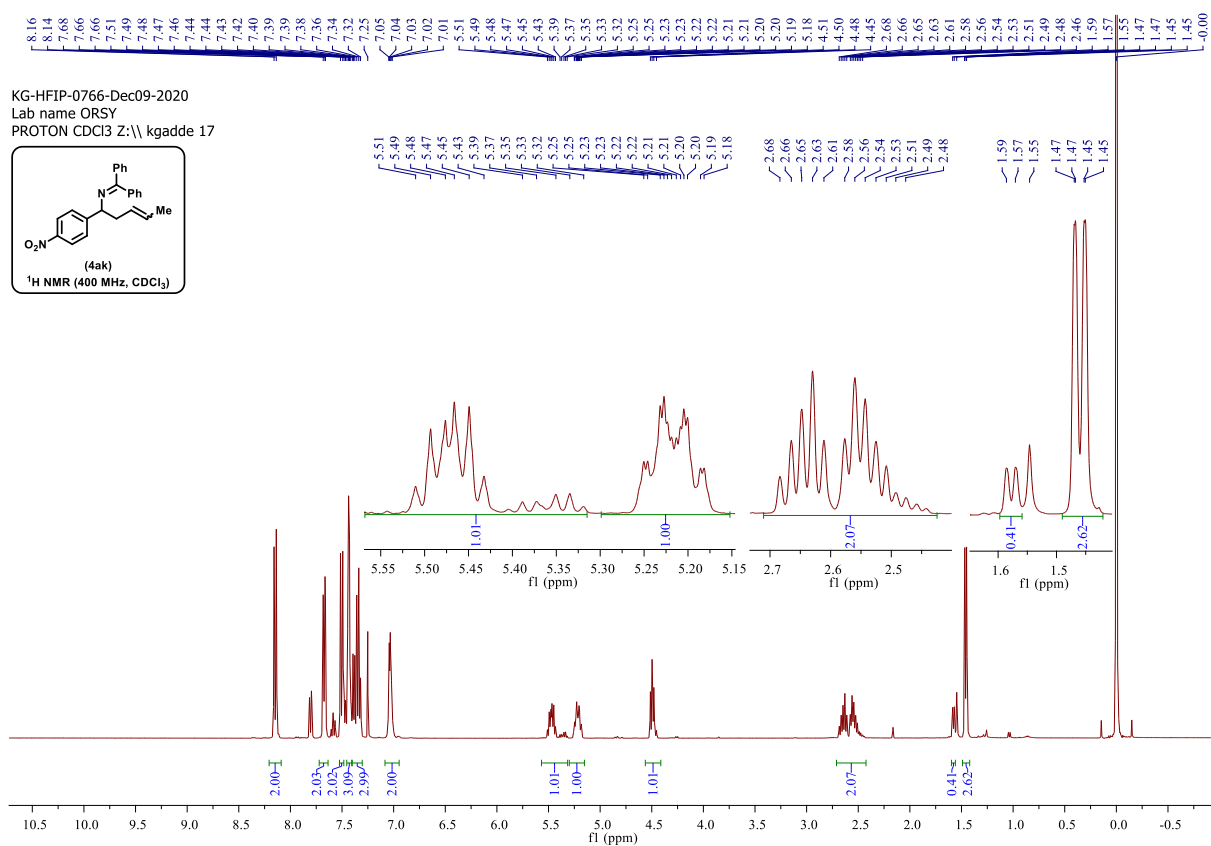
KG-HFIP-0741-Nov16-2020
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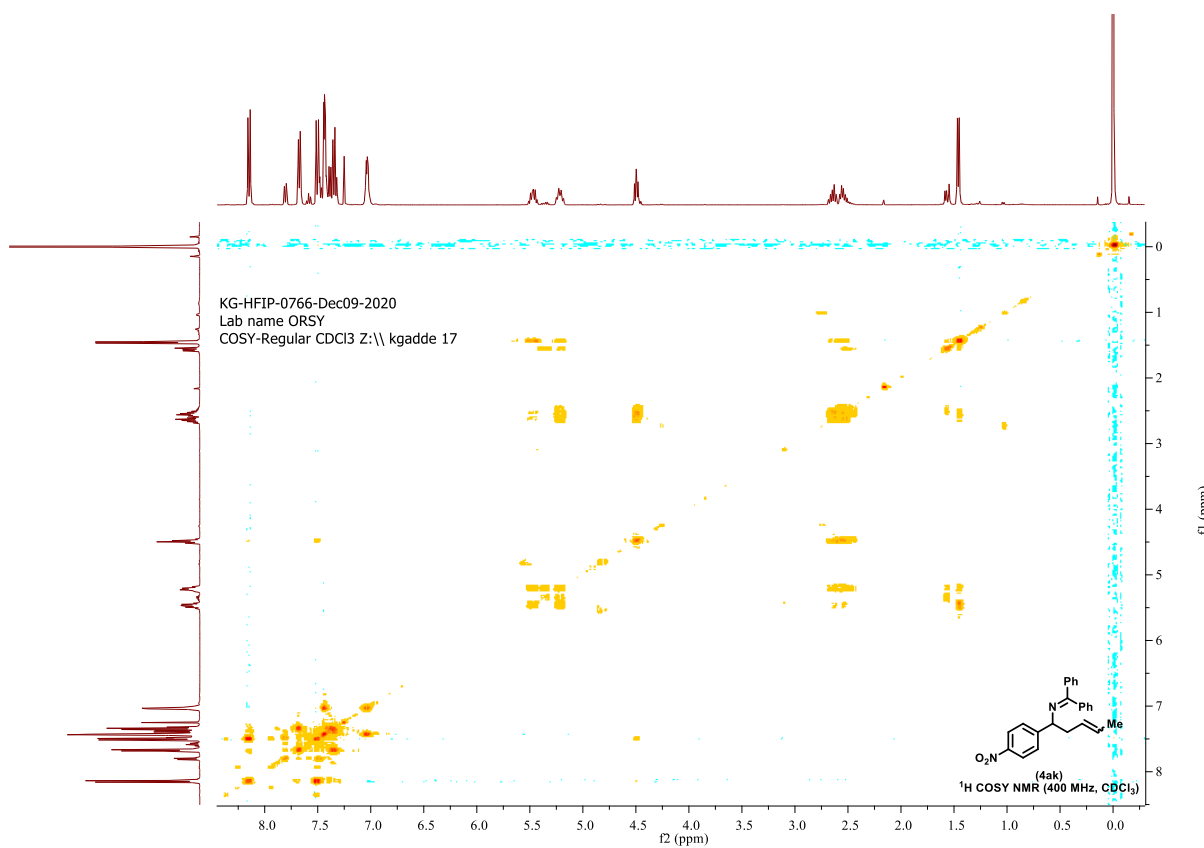
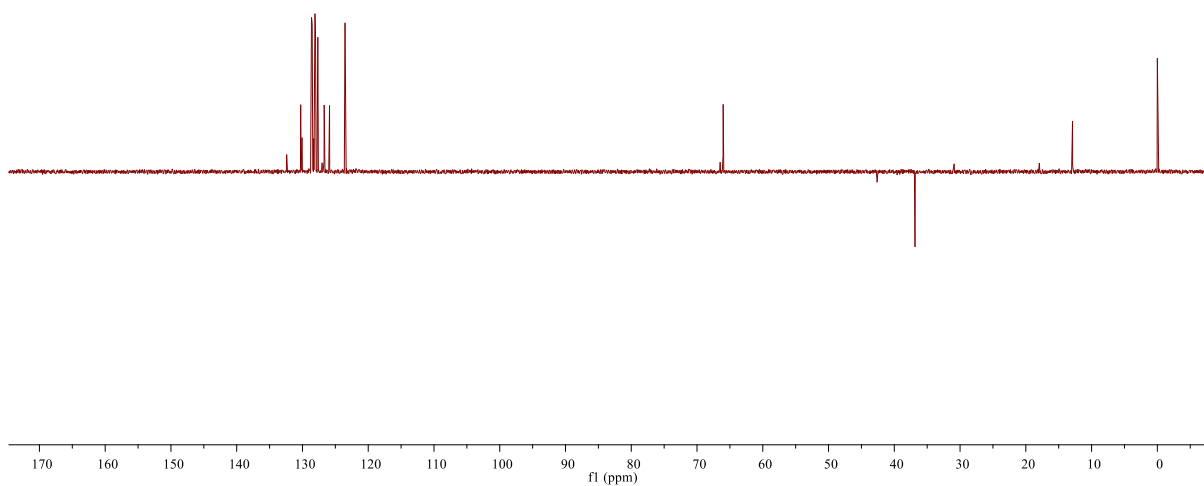
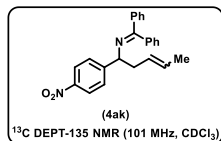


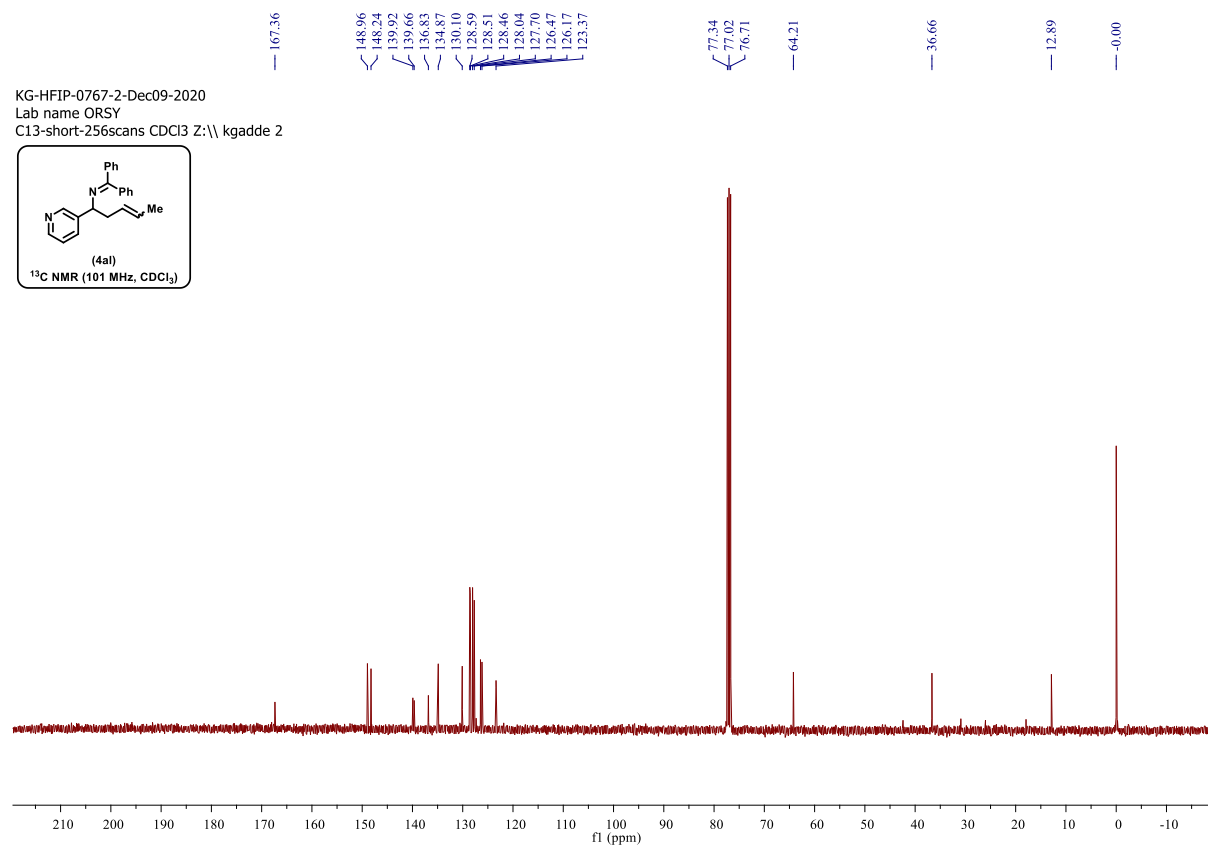
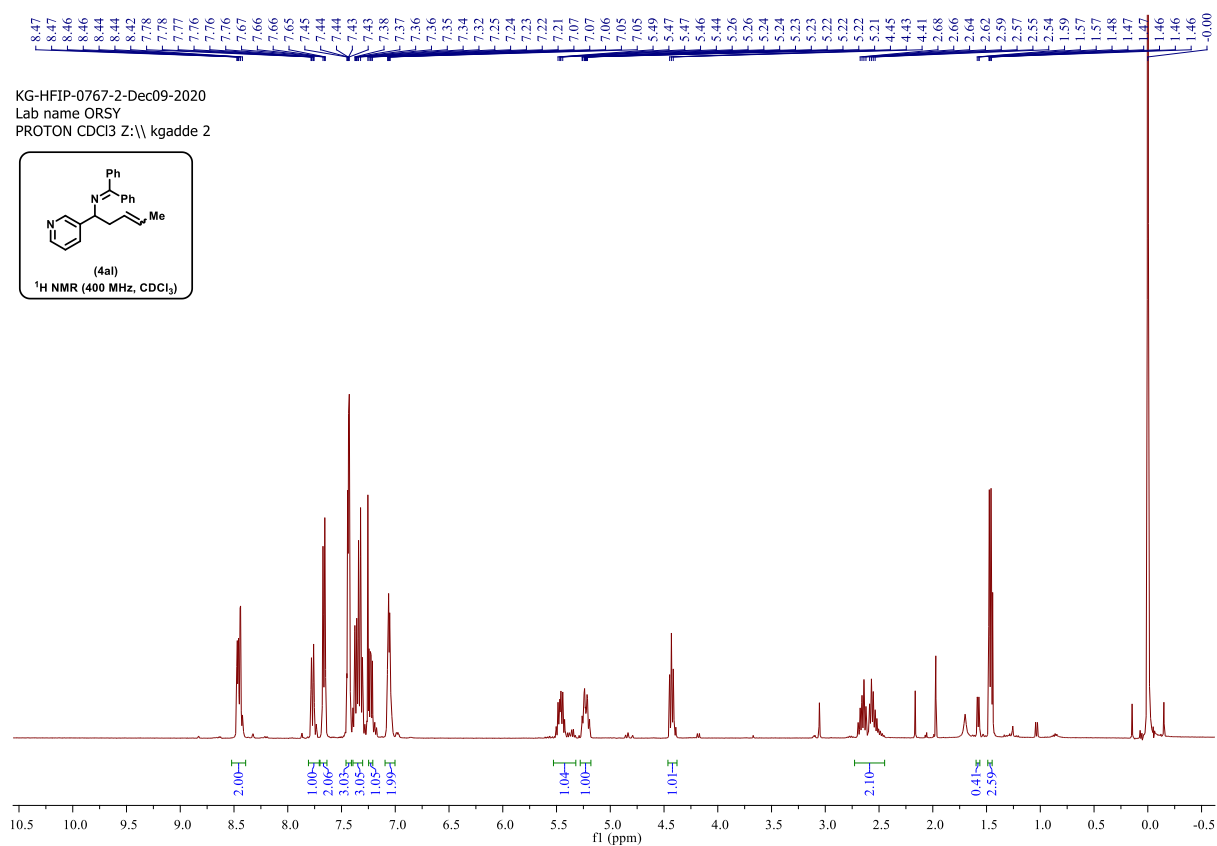
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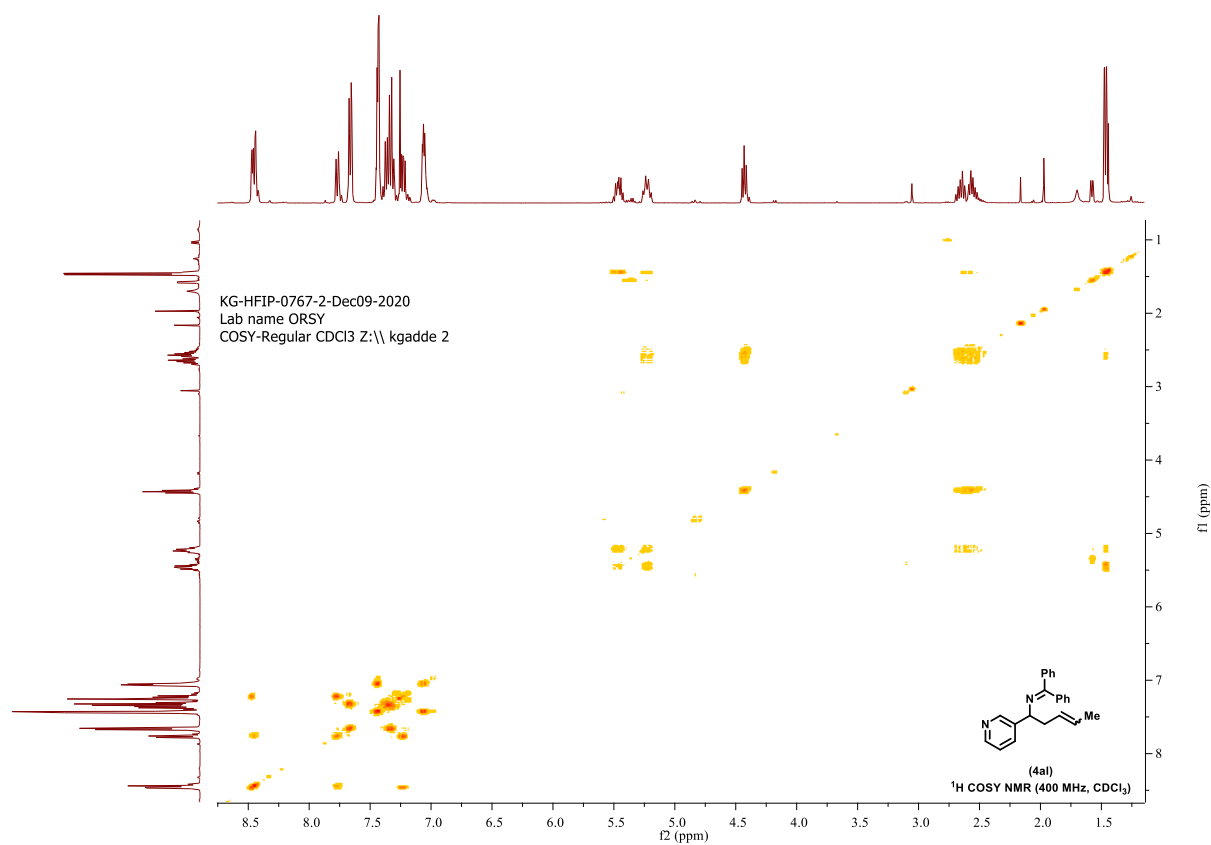
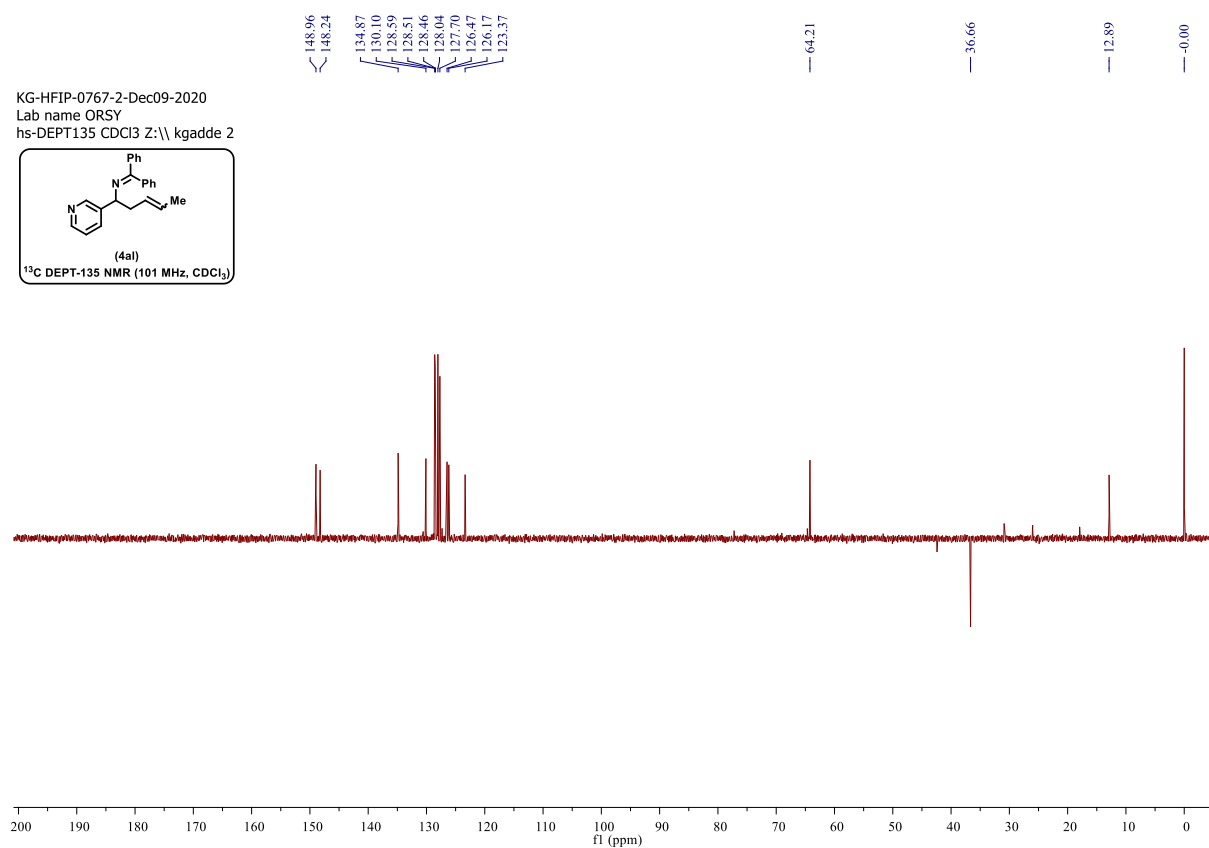
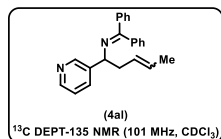


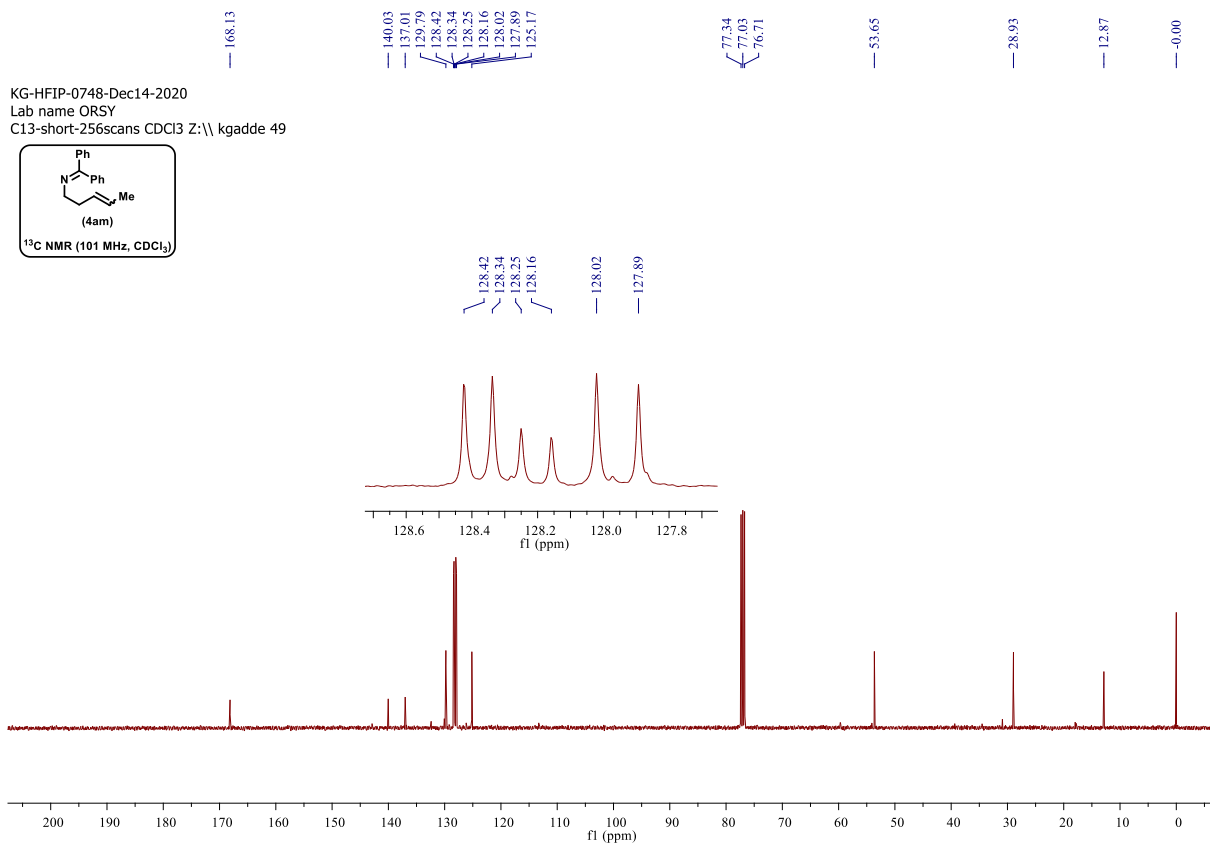
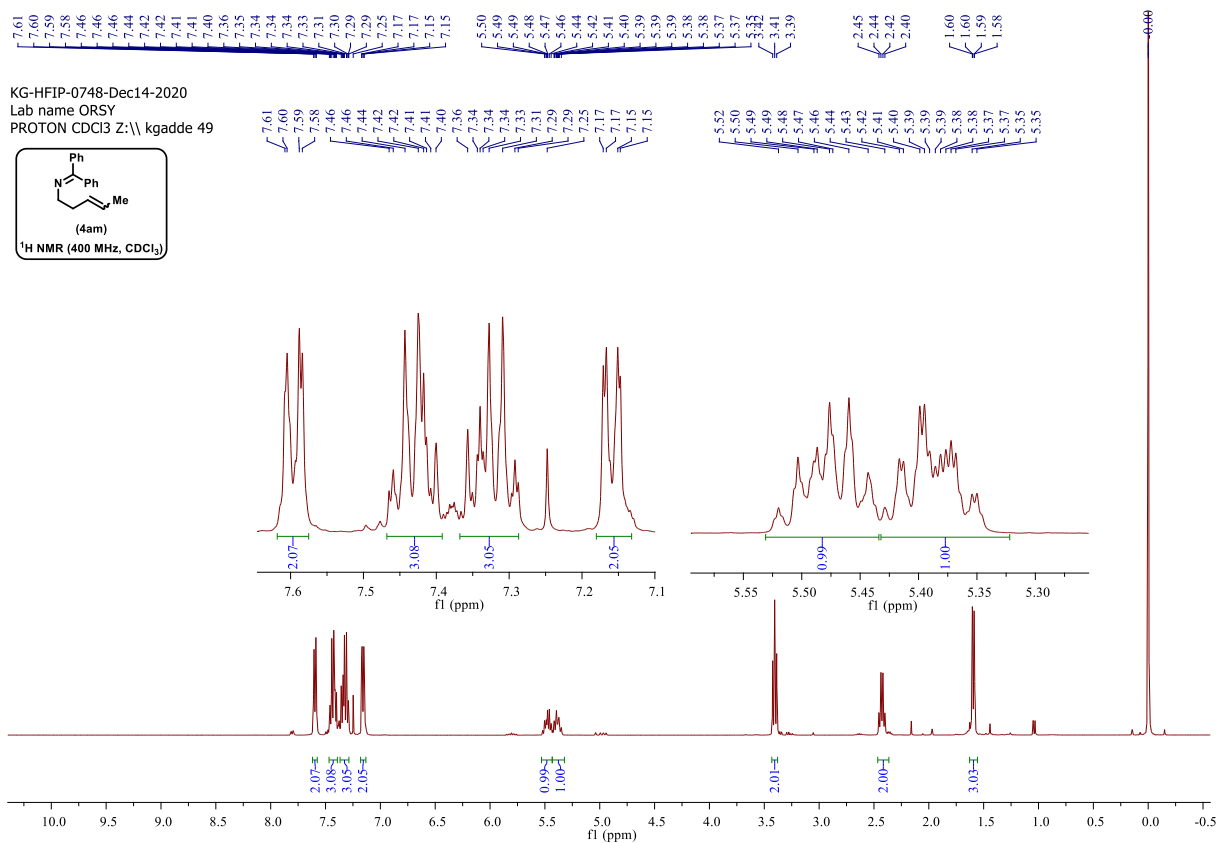
KG-HFIP-0766-Dec09-2020
 Lab name ORSY
 hs-DEPT135 CDCl3 Z:\\ kgadde 17



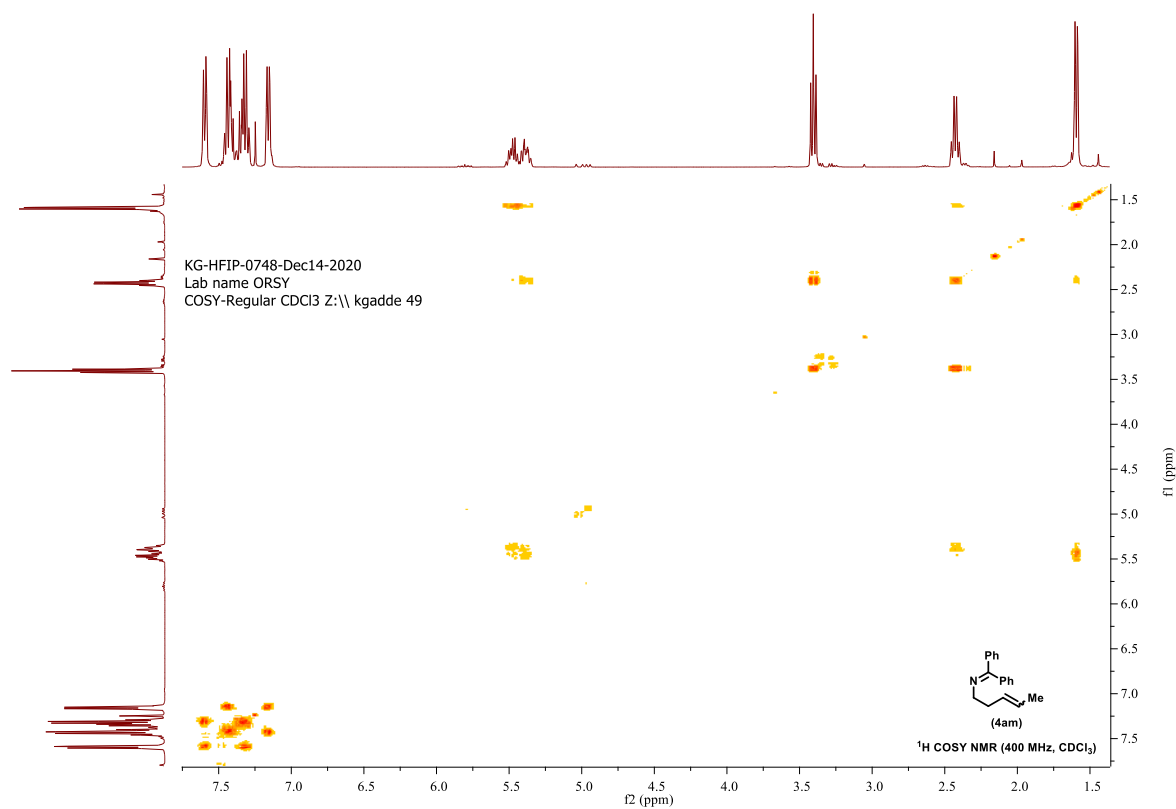
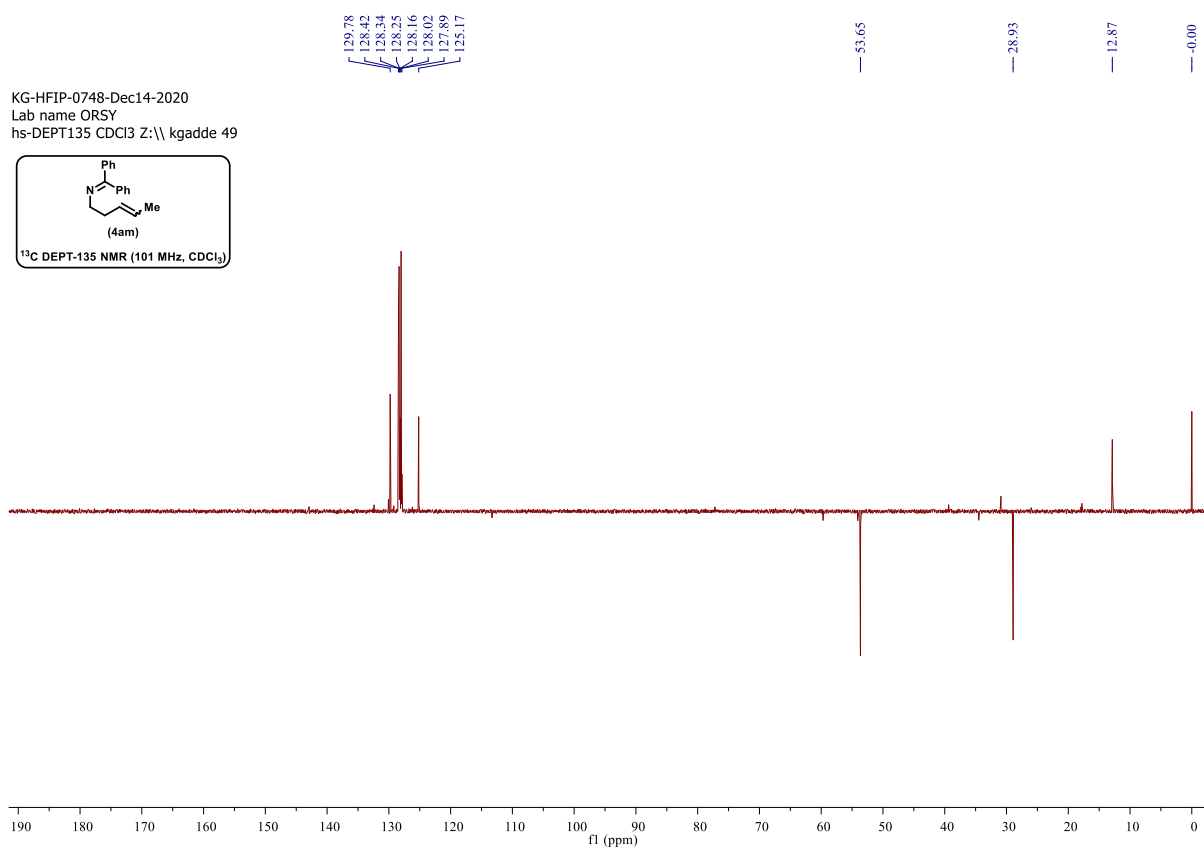
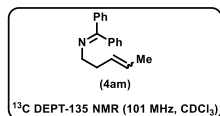


KG-HFIP-0767-2-Dec09-2020
 Lab name ORSY
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KG-HFIP-0748-Dec14-2020
 Lab name ORSY
 hs-DEPT135 CDCl₃ Z:\\ kgadde 49



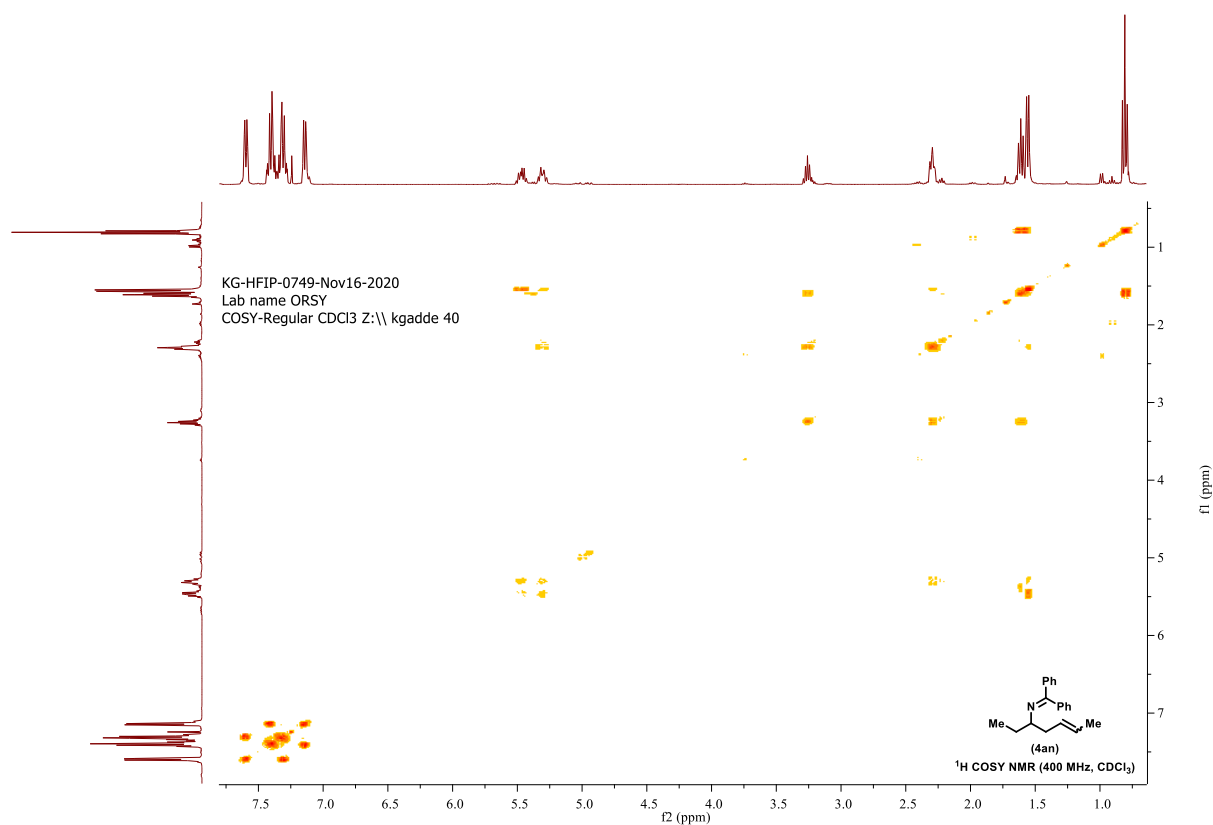
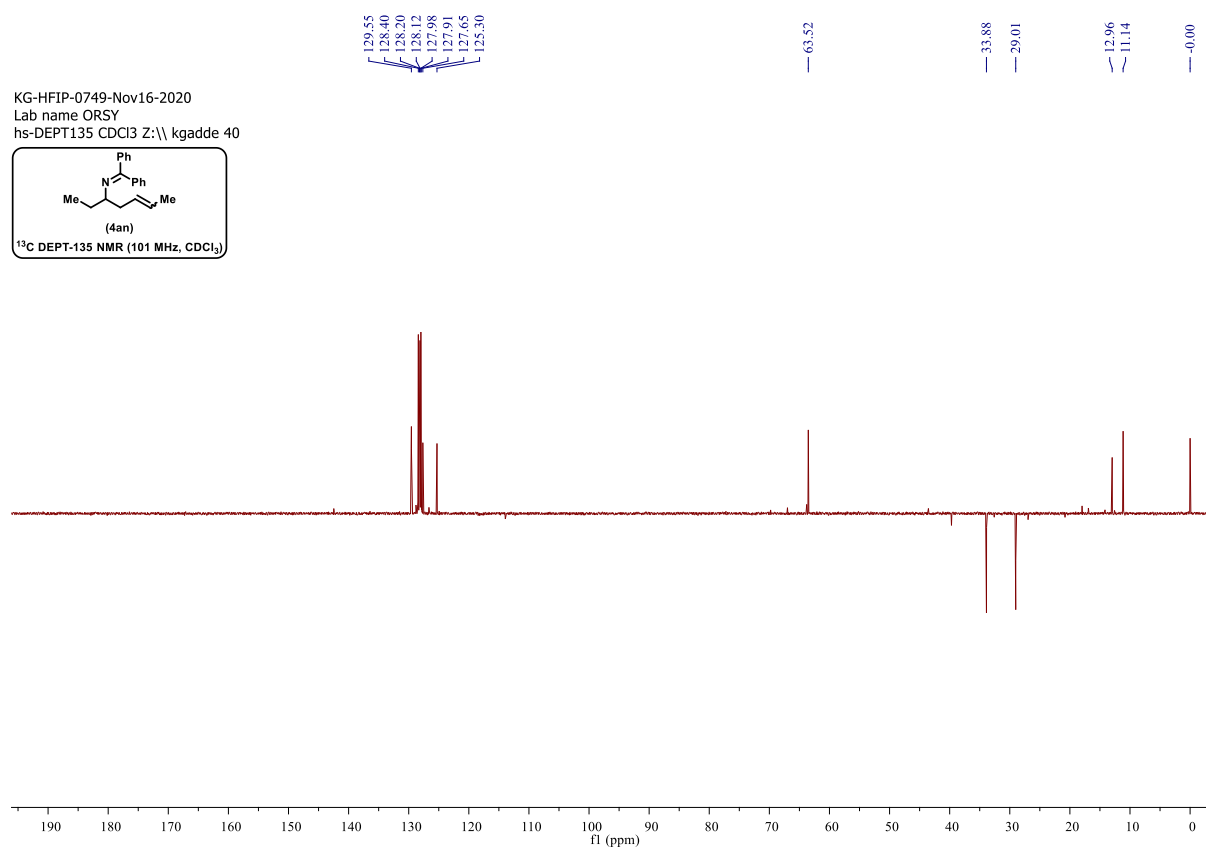
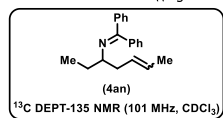
(4an)
¹H NMR (400 MHz, CDCl₃)

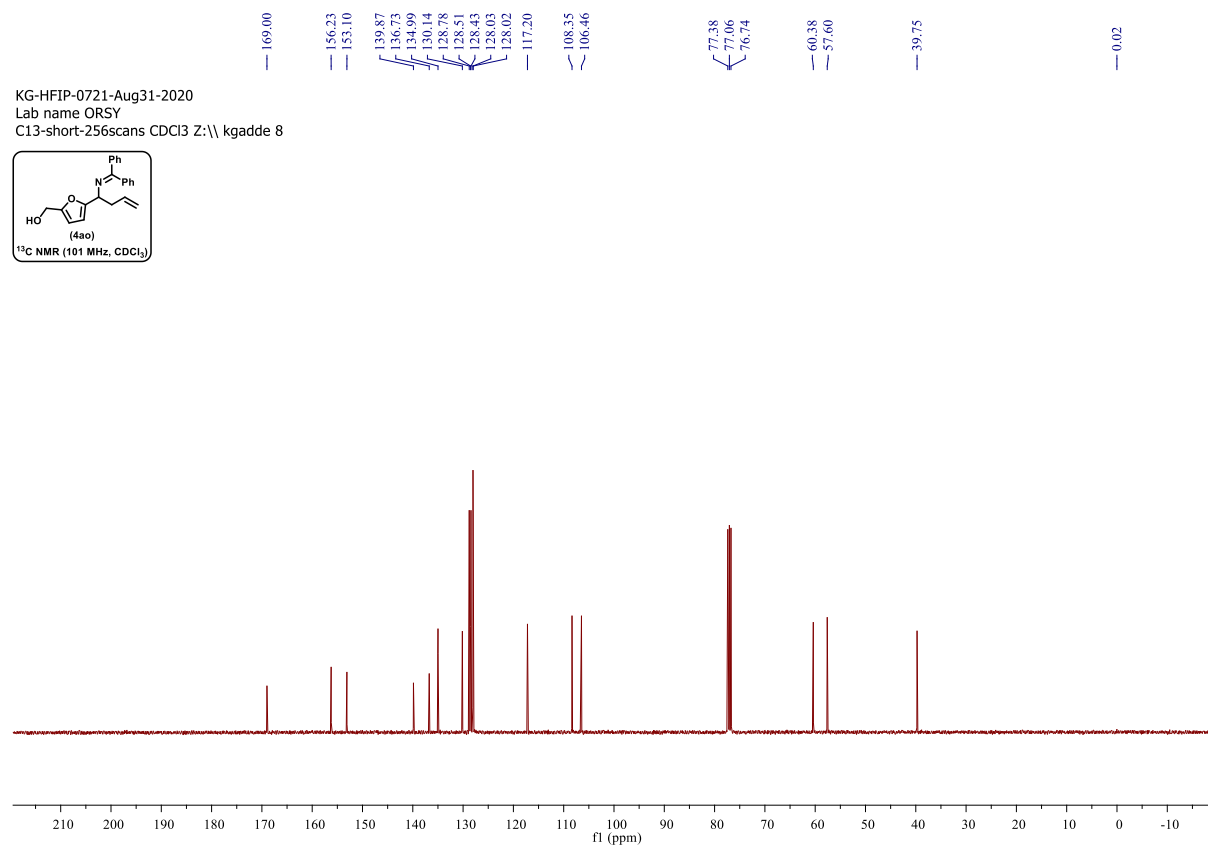
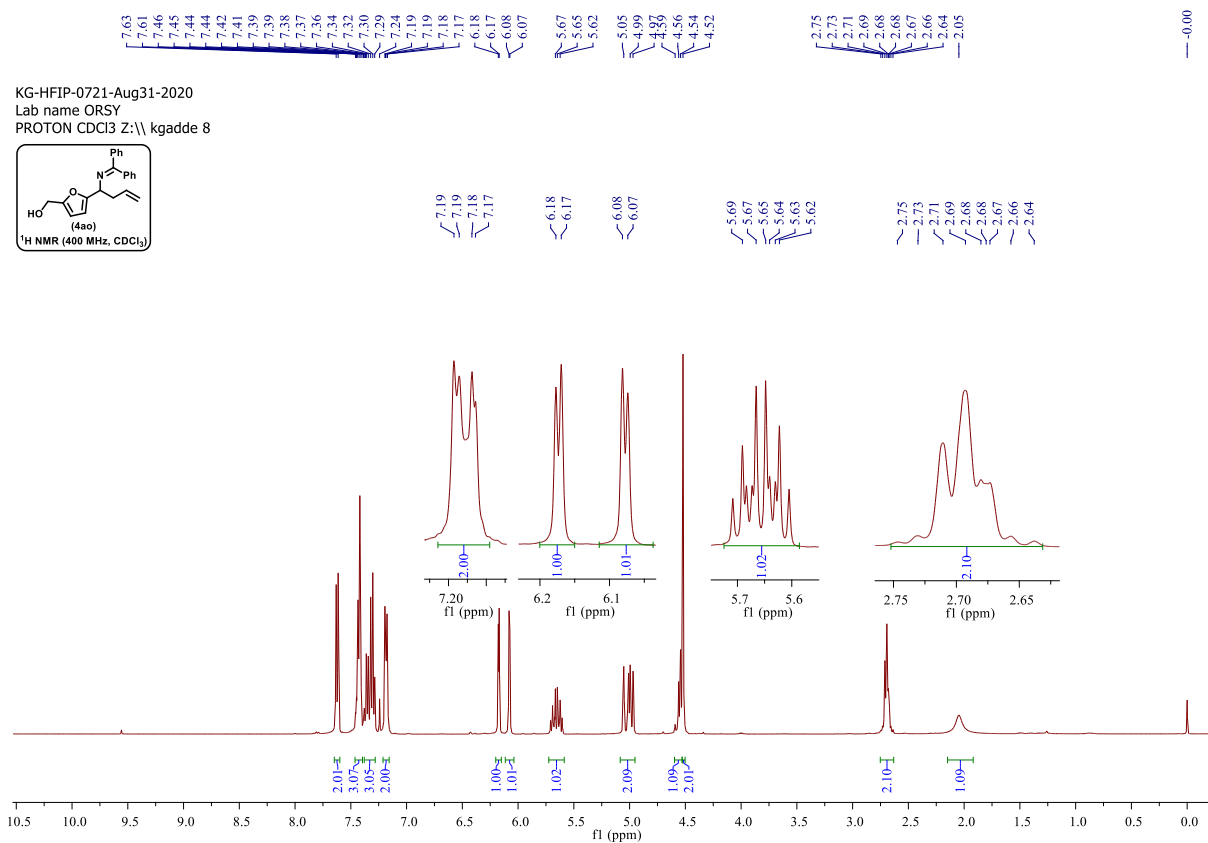



(4an)

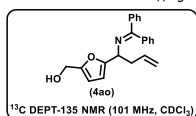


KG-HFIP-0749-Nov16-2020
Lab name ORSY
hs-DEPT135 CDCl₃ Z:\\ kgadde 40

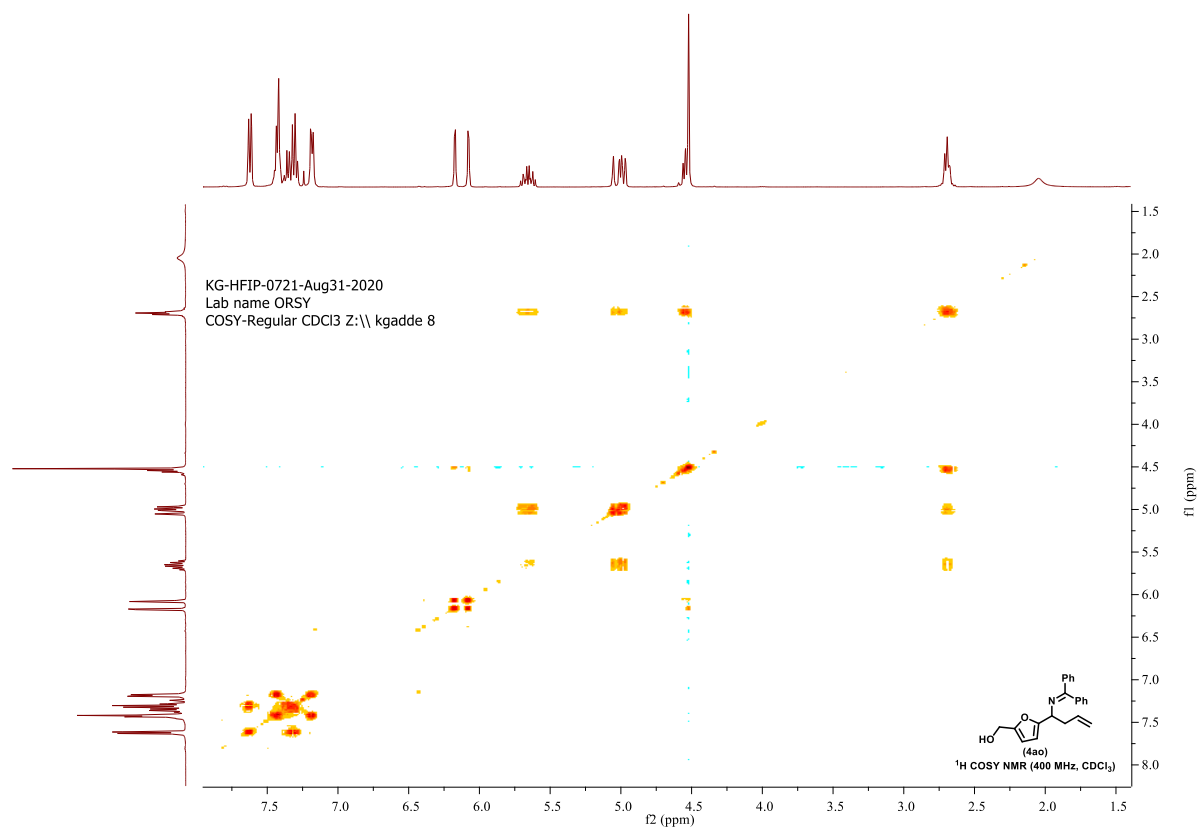
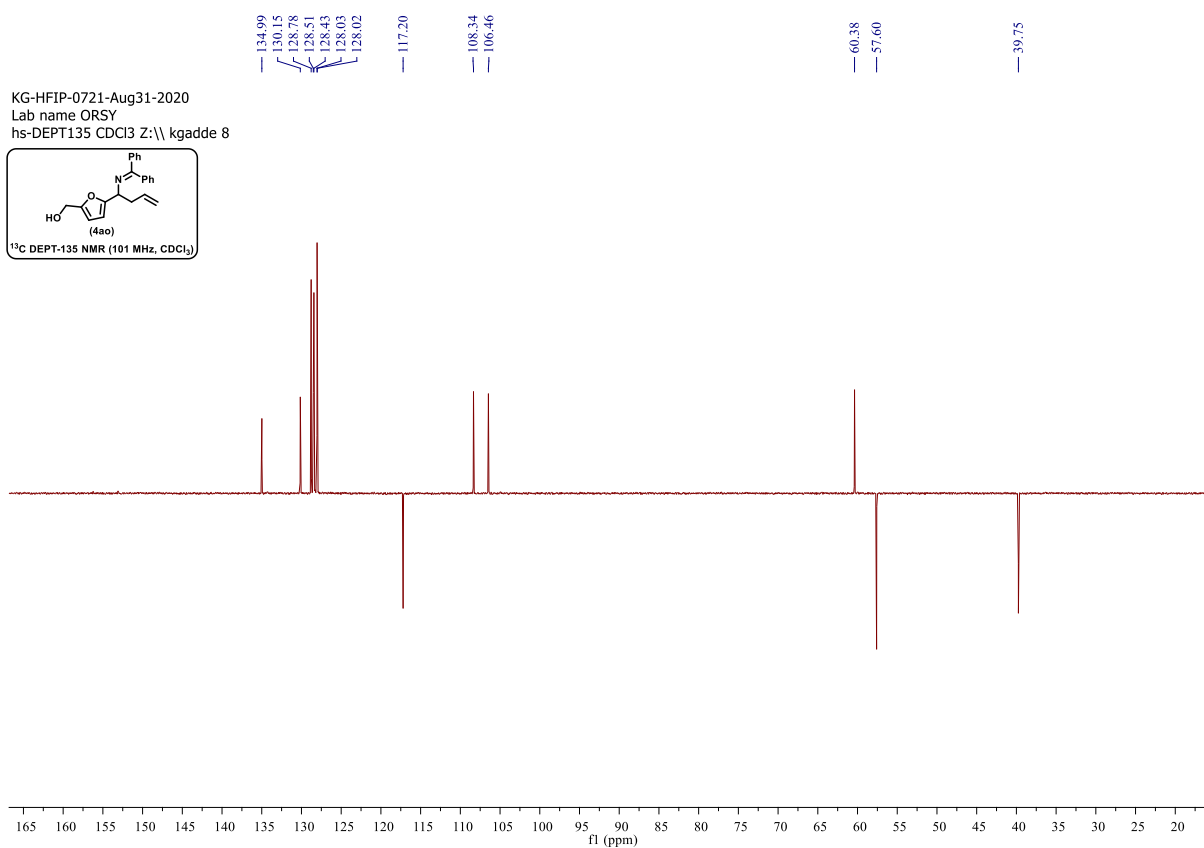


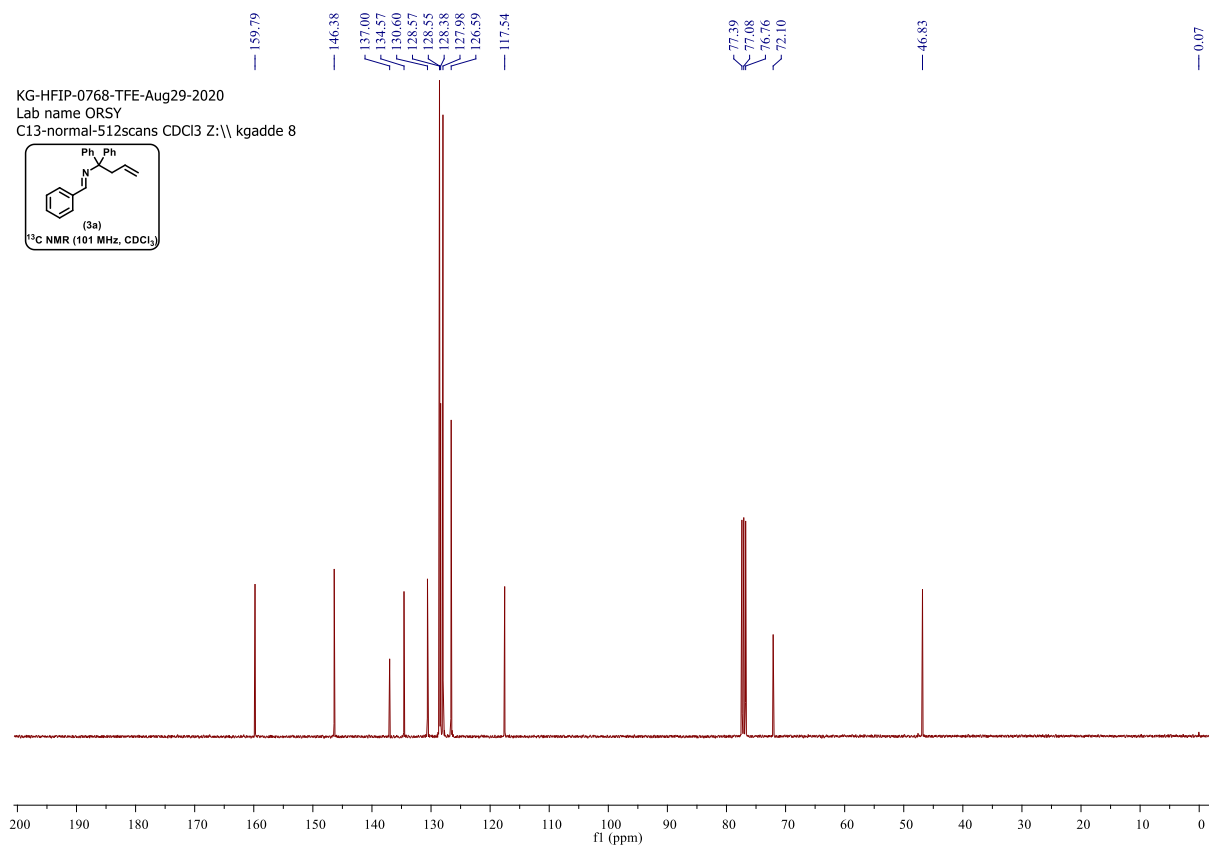
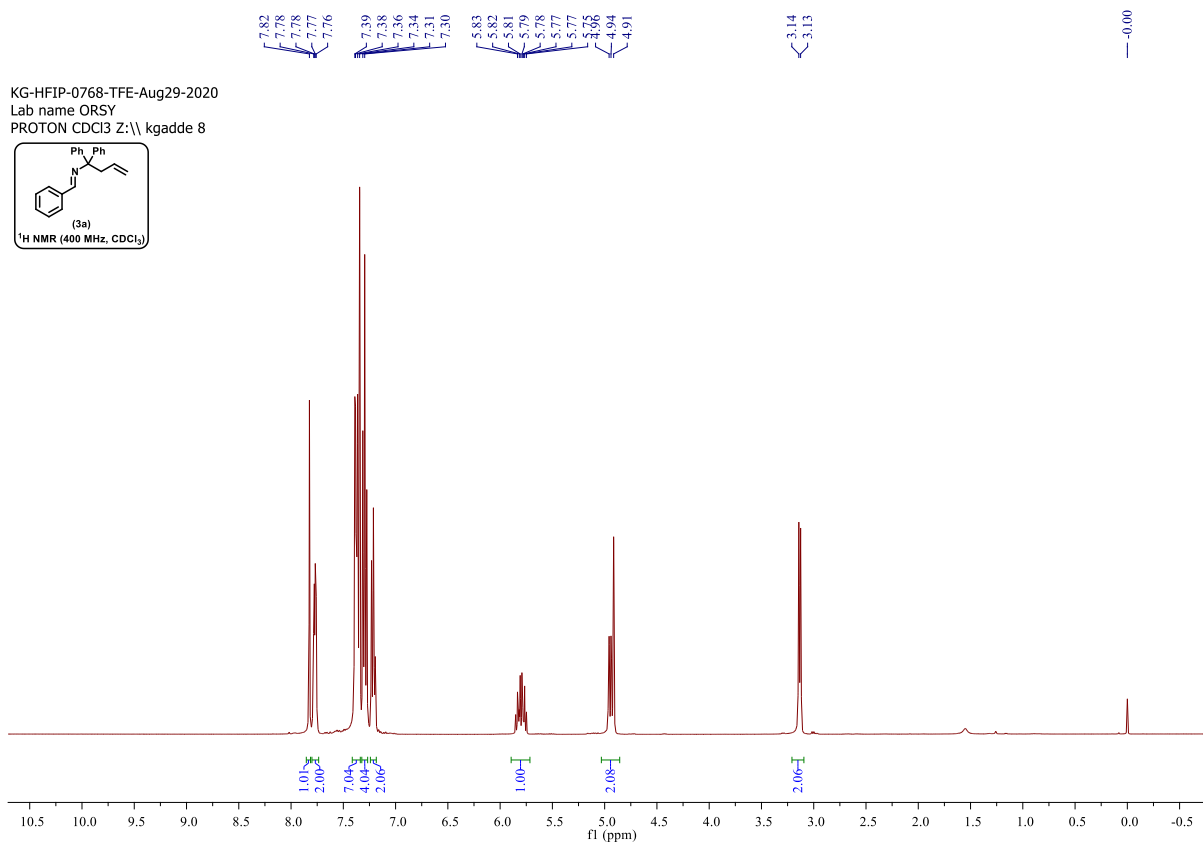


KG-HFIP-0721-Aug31-2020
 Lab name ORSY
 hs-DEPT135 CDCl₃ Z:\\ kgadde 8

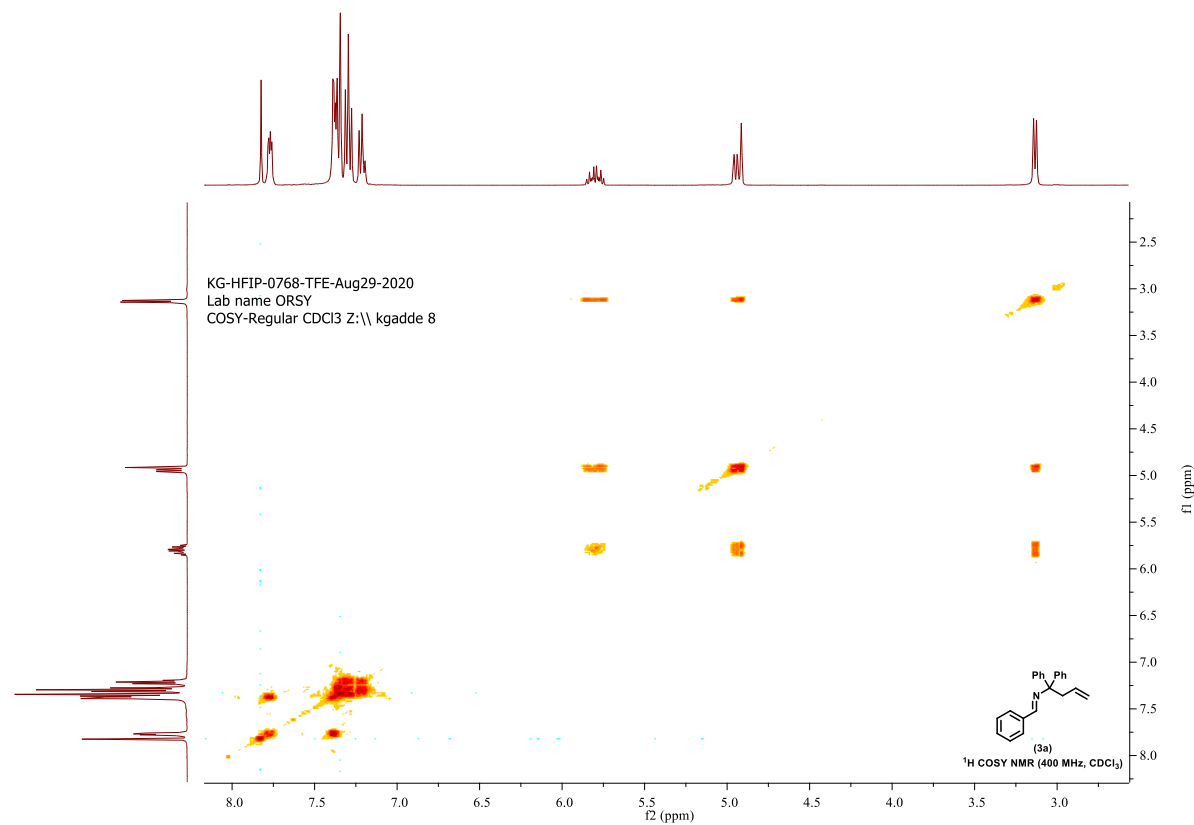
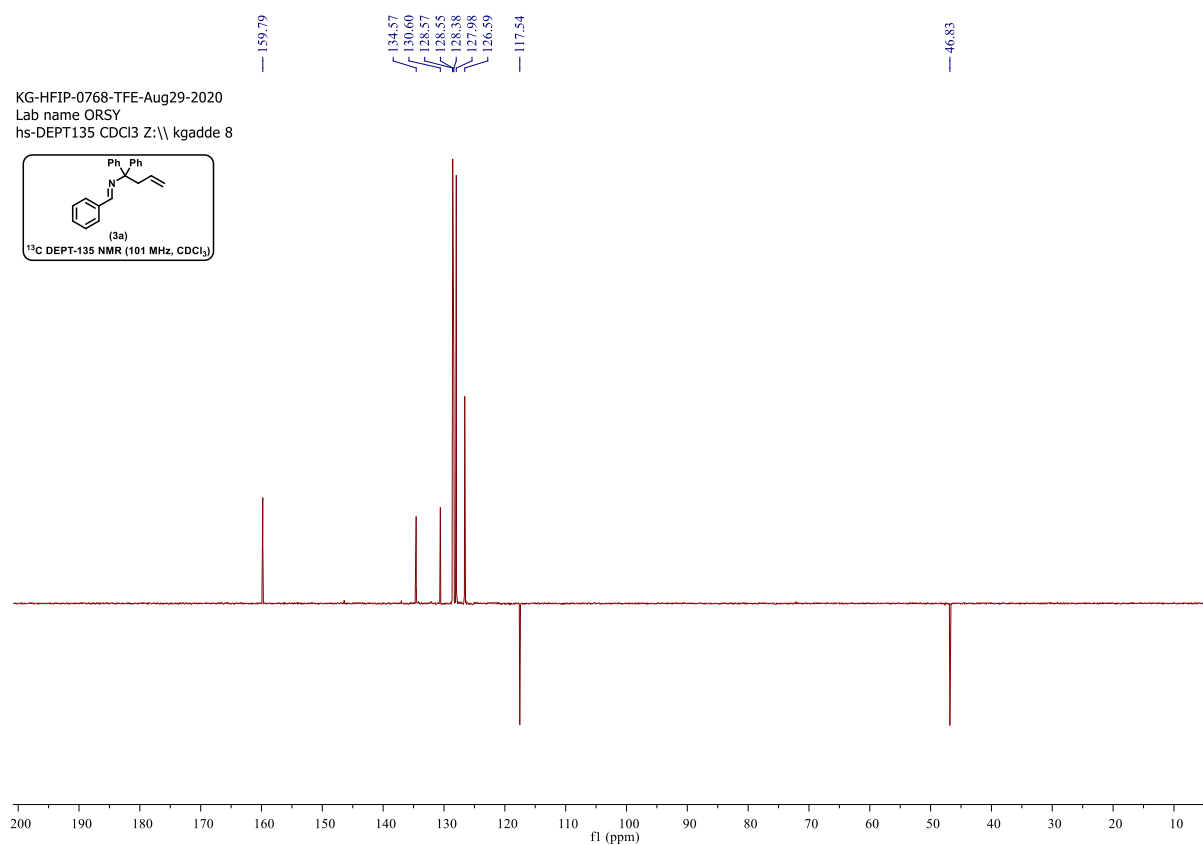
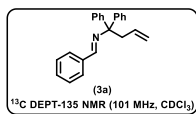


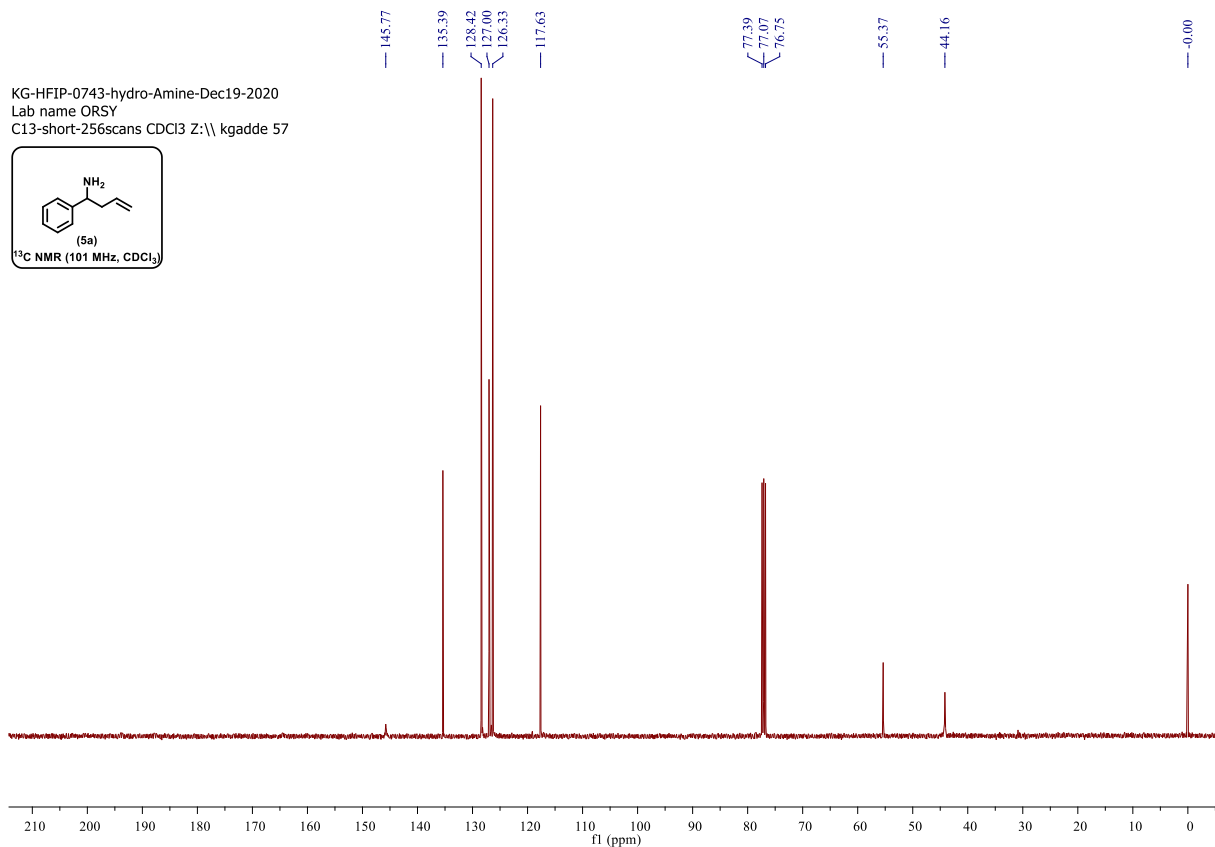
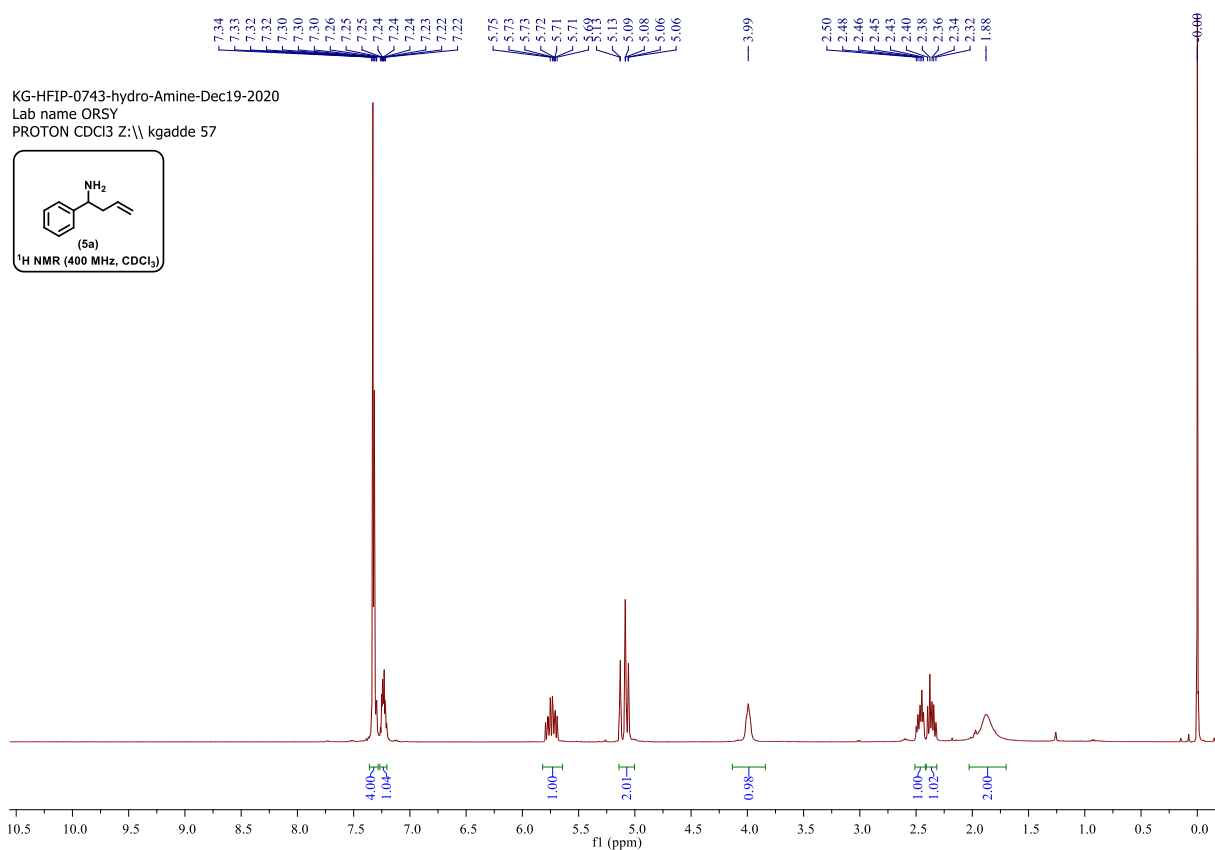
¹³C DEPT-135 NMR (101 MHz, CDCl₃)

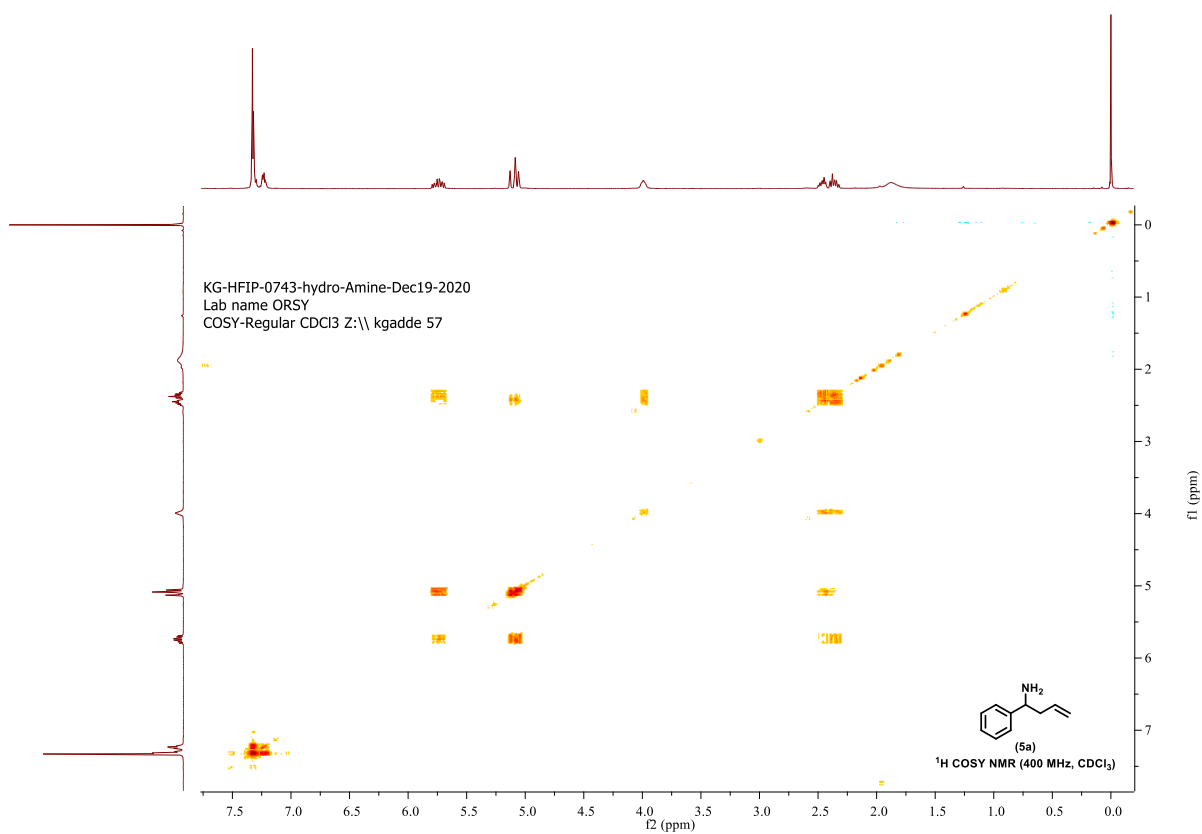
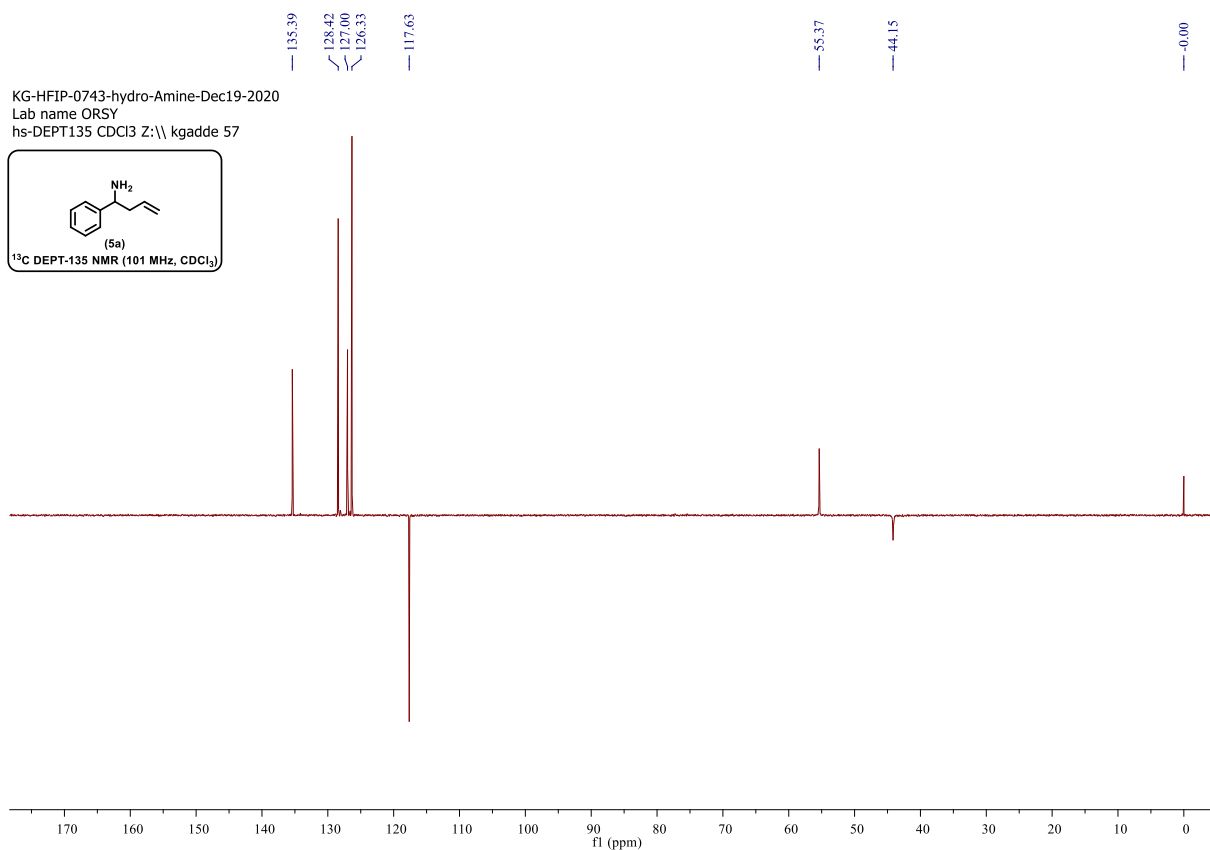


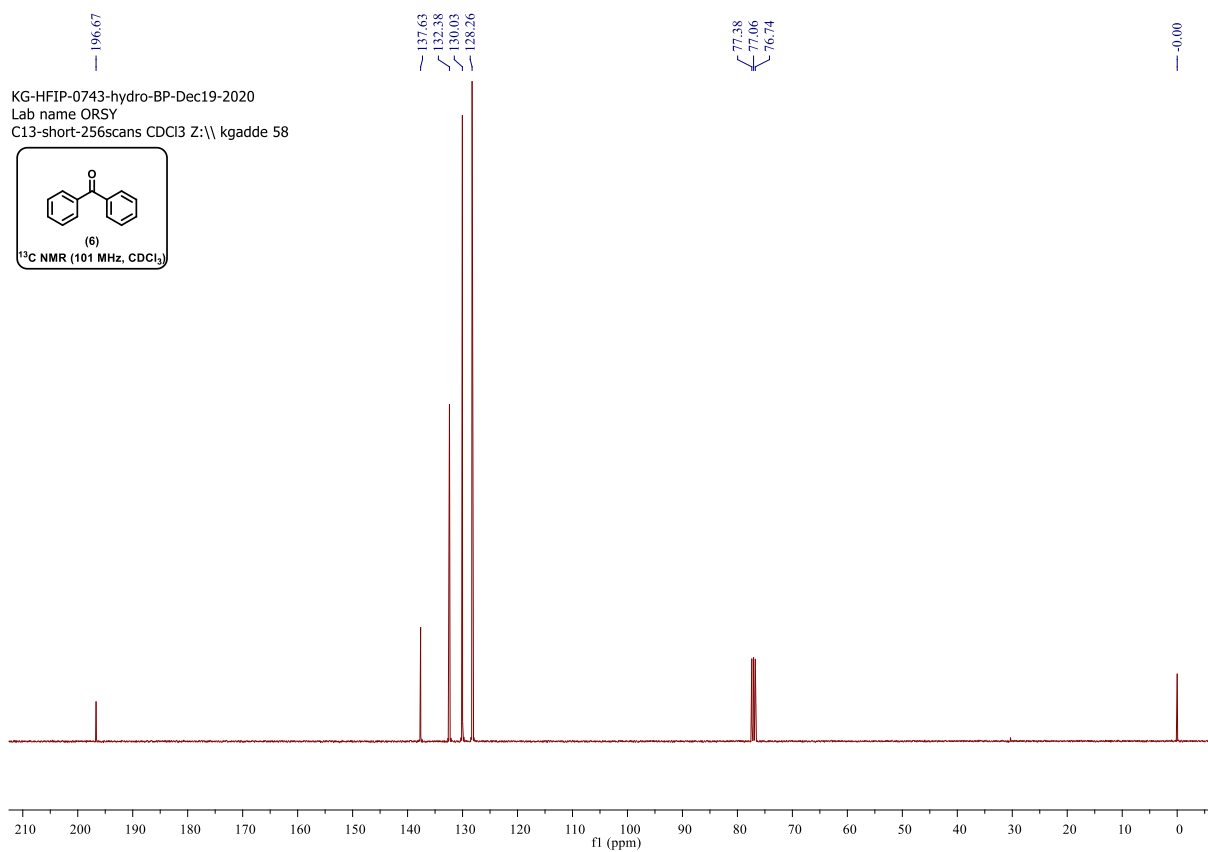
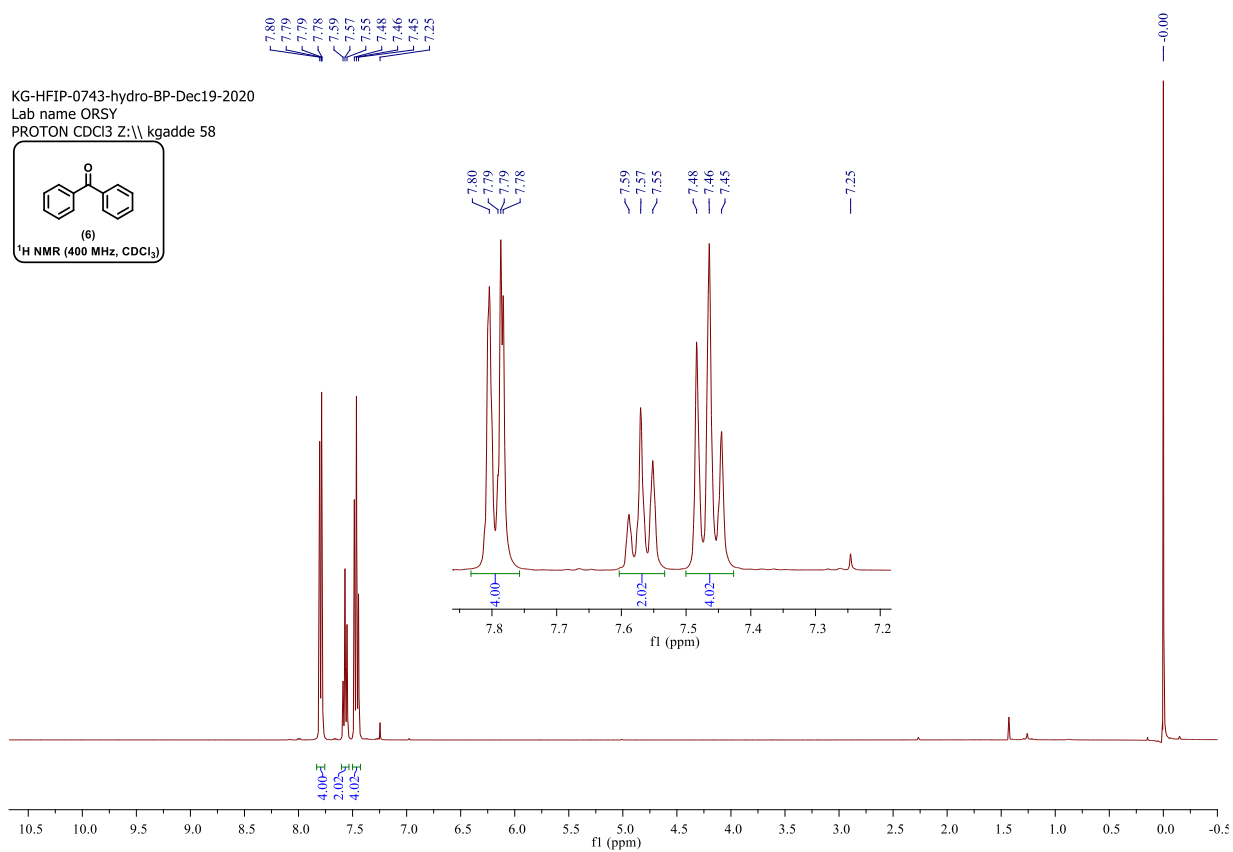


KG-HFIP-0768-TFE-Aug29-2020
 Lab name ORSY
 hs-DEPT135 CDCl₃ Z:\\ kgadde 8

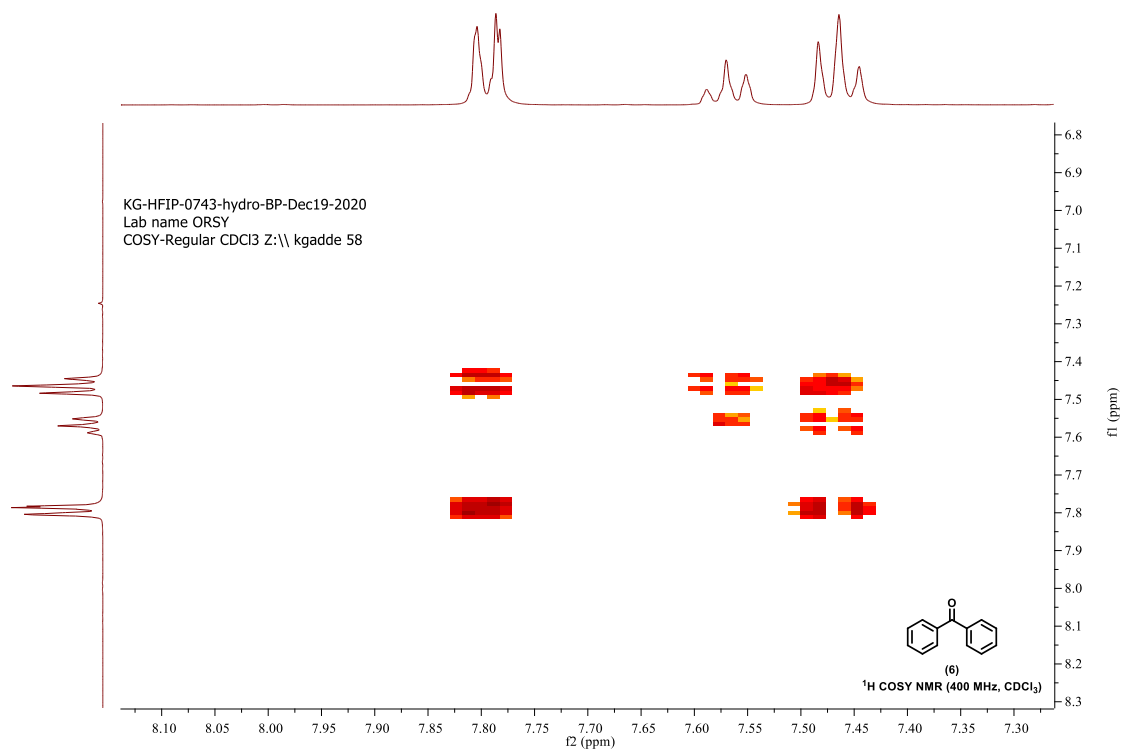
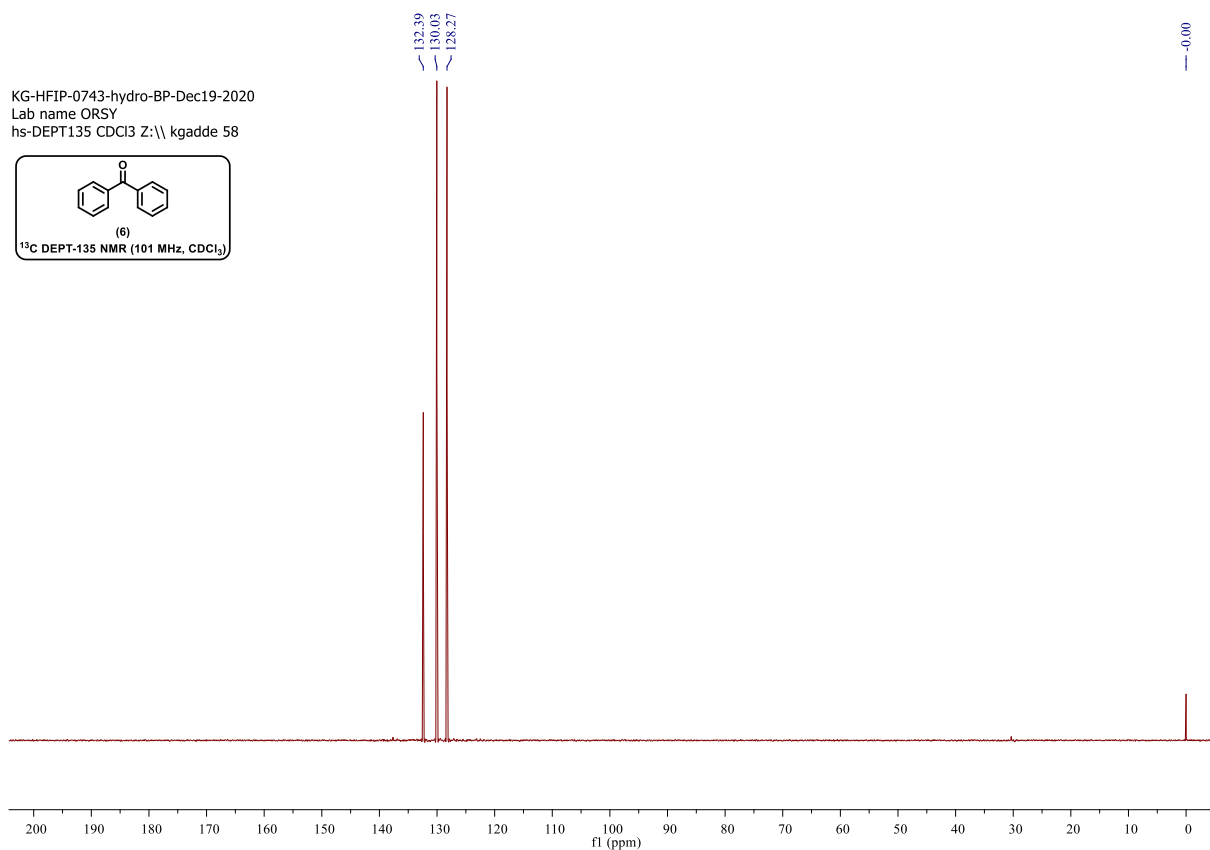
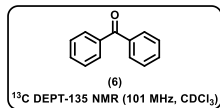


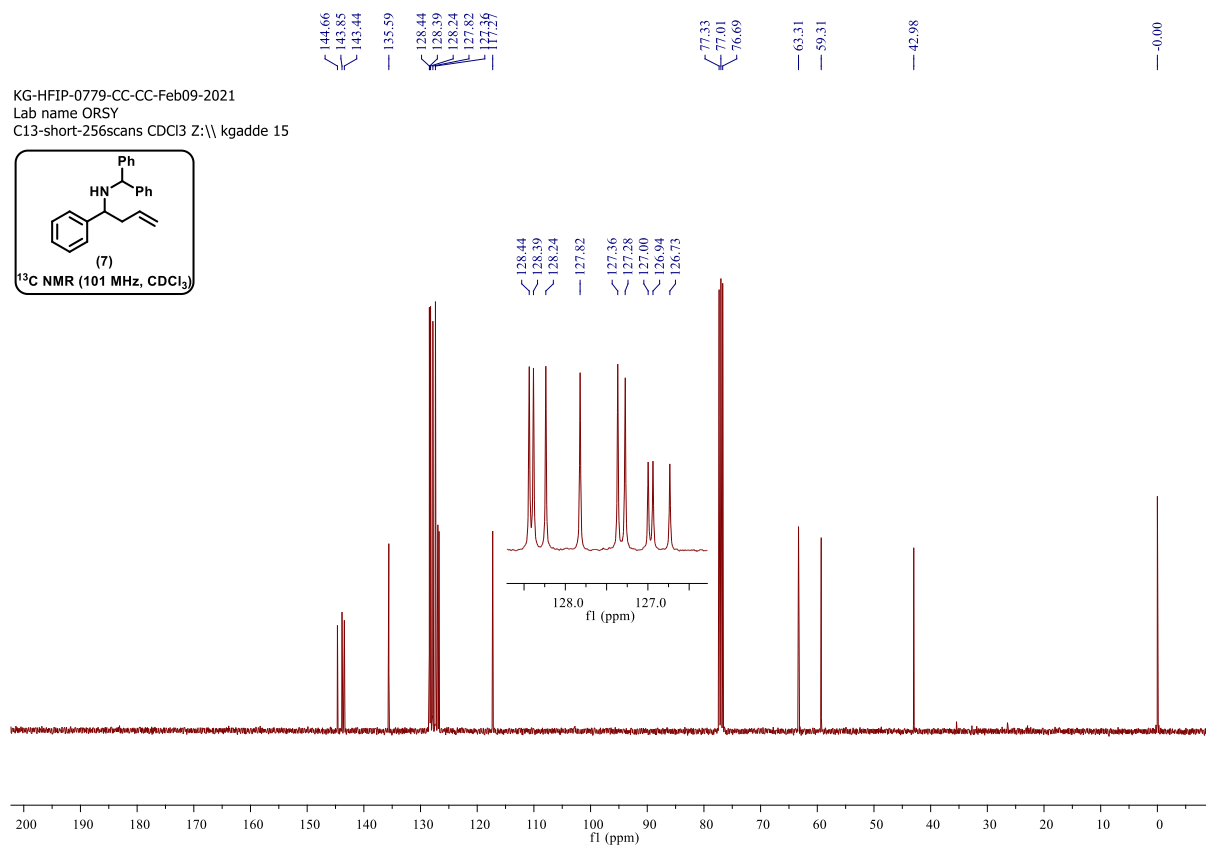
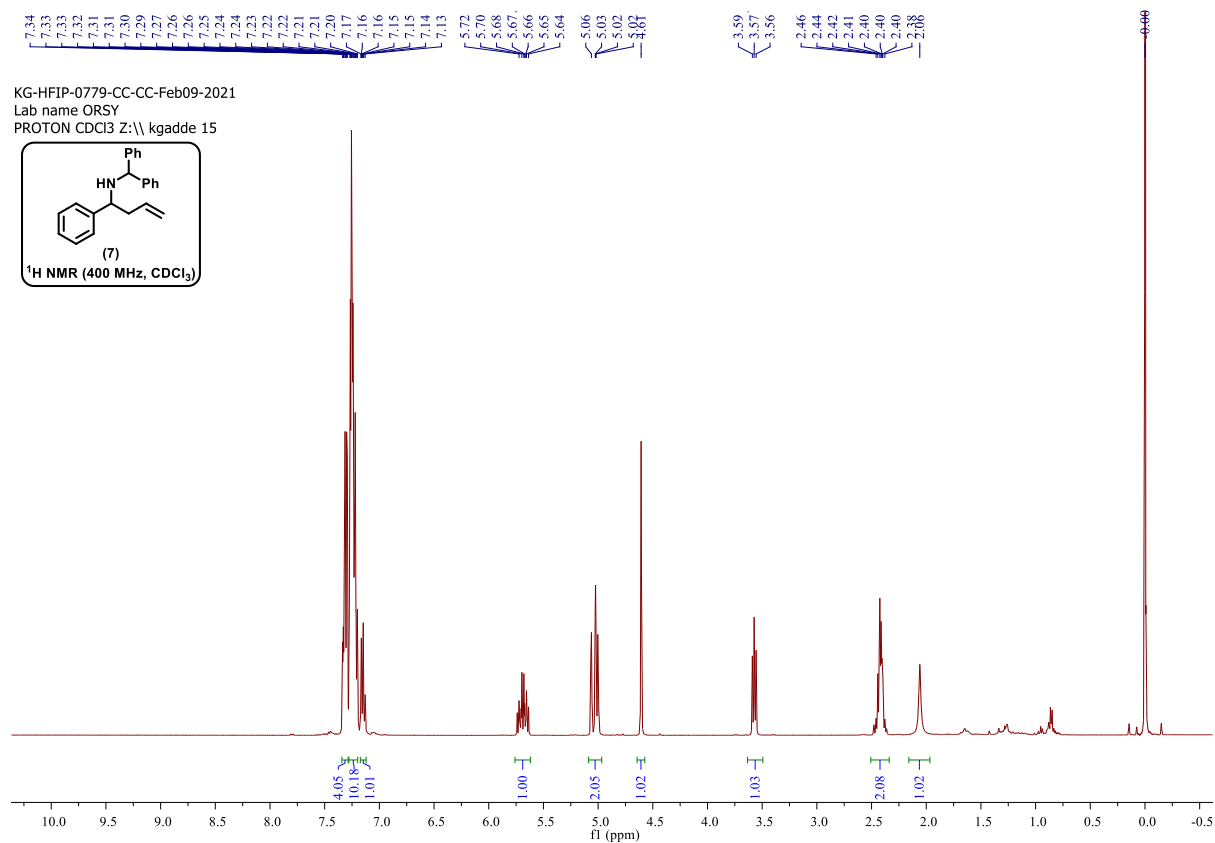




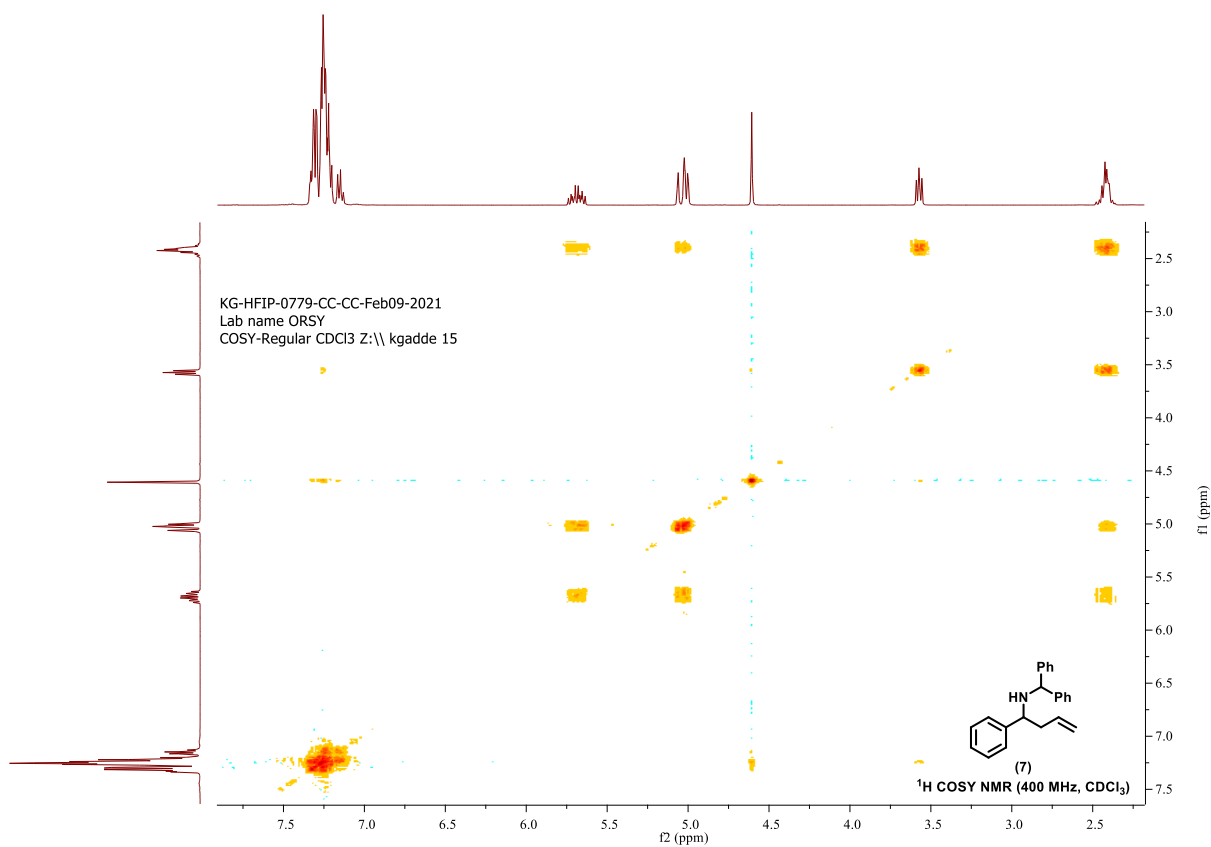
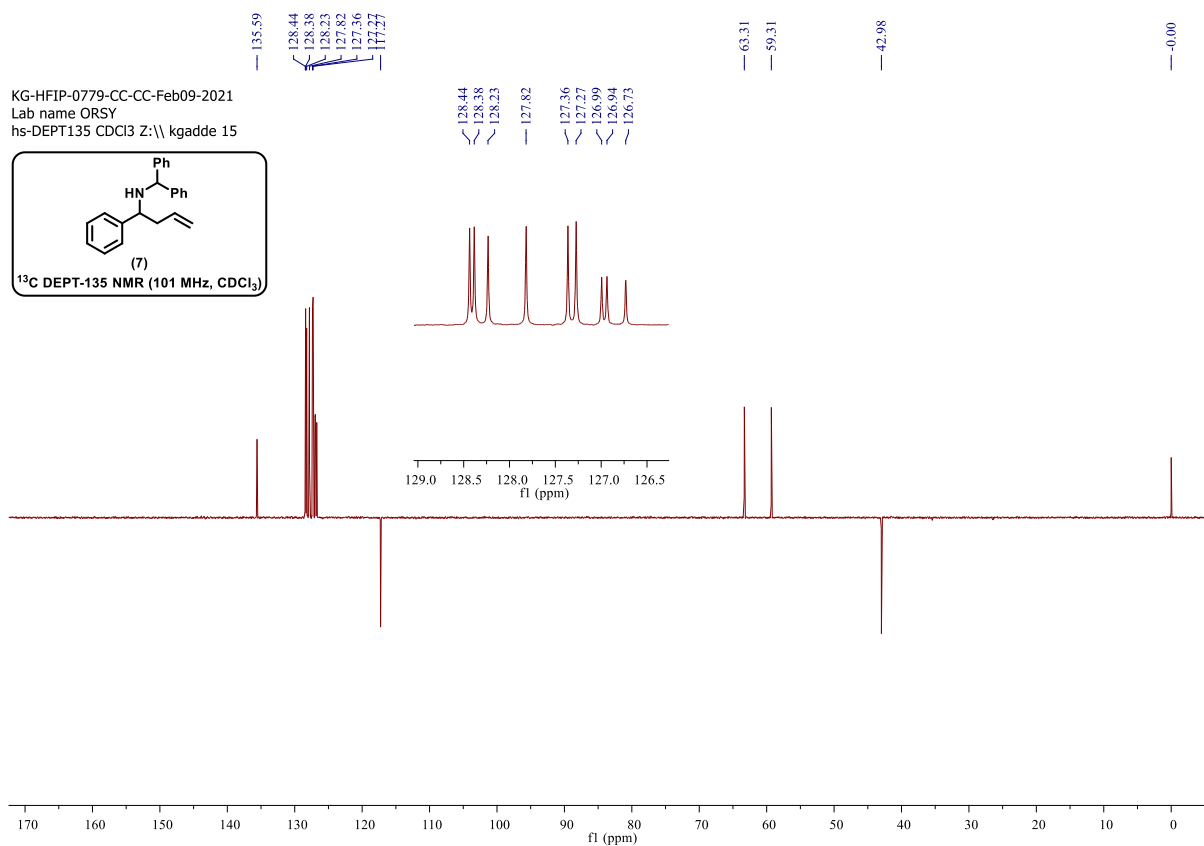
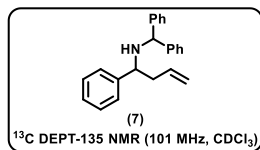


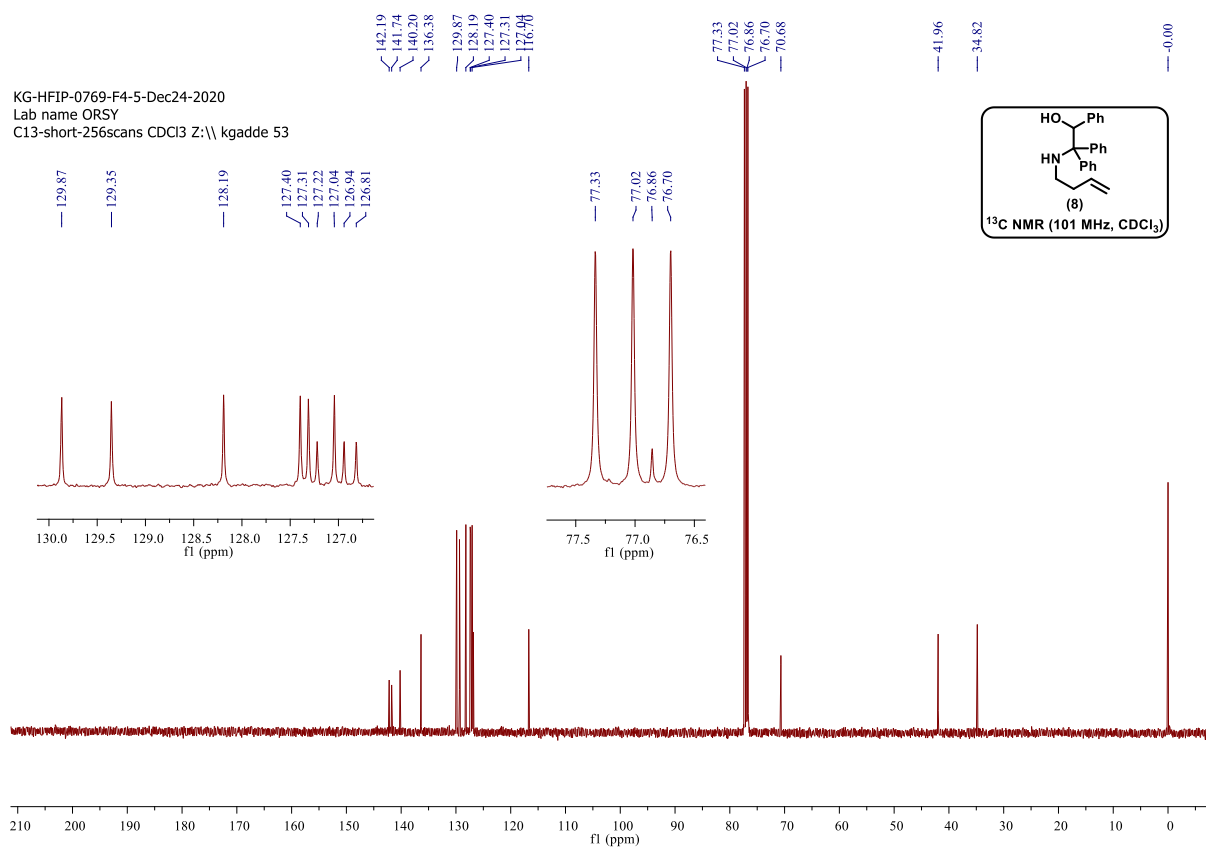
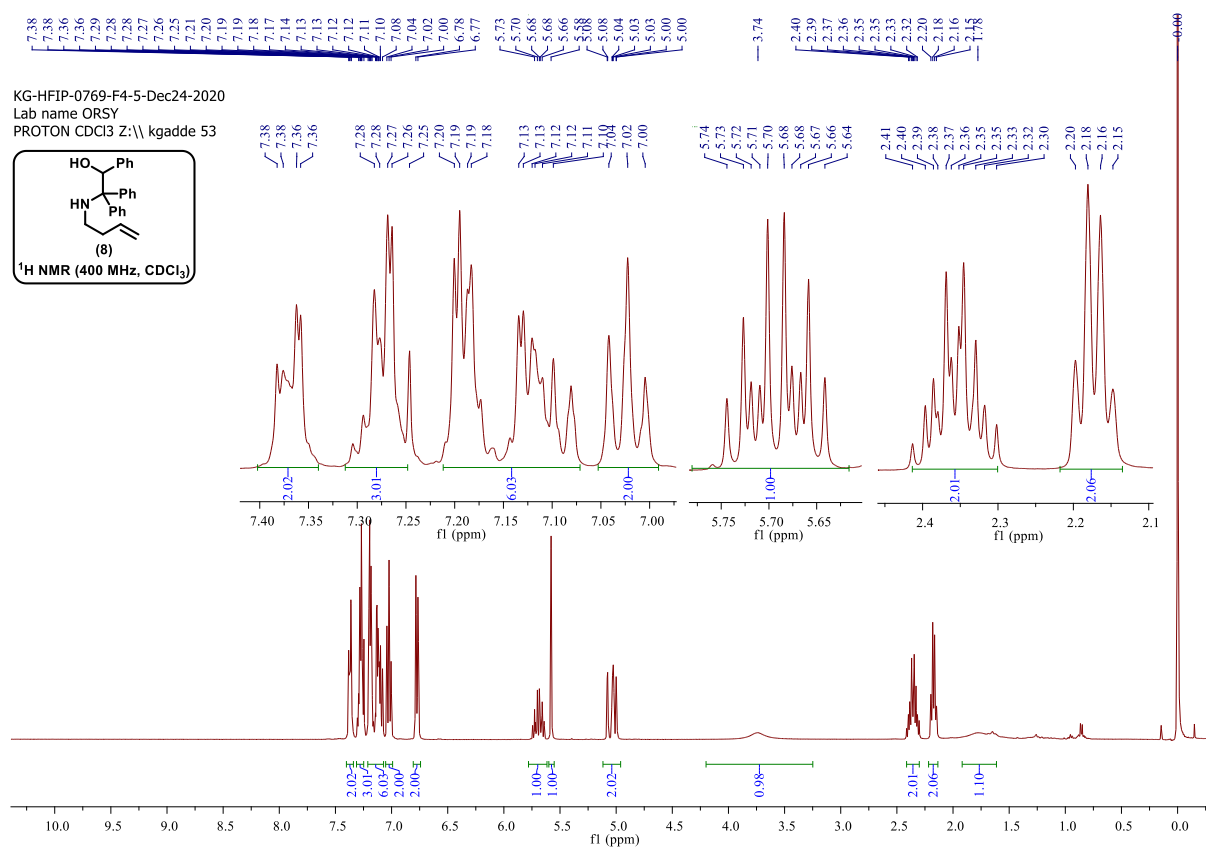
KG-HFIP-0743-hydro-BP-Dec19-2020
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 hs-DEPT135 CDCl₃ Z:\\ kgadde 58





KG-HFIP-0779-CC-CC-Feb09-2021
 Lab name ORSY
 hs-DEPT135 CDCl₃ Z:\\ kgadde 15





KG-HFIP-0769-F4-5-Dec24-2020
 Lab name ORSY
 hs-DEPT135 CDCl₃ Z:\\ kgadde 53

