Supporting Information

Copper-Catalyzed Hydroaminocarbonylation of Benzylidenecyclopropanes: Synthesis of γ,δ-Unsaturated Amides

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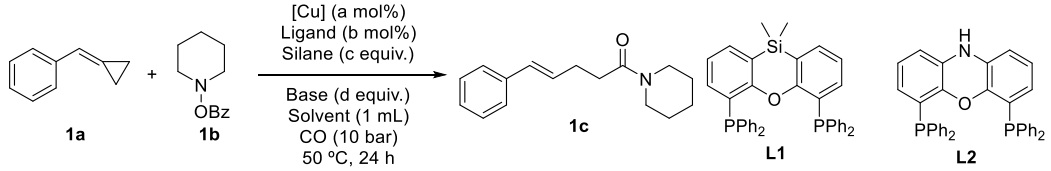
1. General information

Unless otherwise noted, all commercial reagents were ordered from Sigma-Aldrich, TCI, ABCR, or Across. All solvents (anhydrous and under inert atmosphere) were collected by M BRAUN from the solvent purification system and used under standard Schlenk techniques. Benzylidenecyclopropanes (BCPs)[1] and hydroxyamine derivatives[2,3] were synthesized based on known literatures. Column chromatography was performed on silica gel (200-300 meshes) using *n*-pentane (bp. 36.1 °C), dichloromethane and ethyl acetate as eluents. All NMR spectra were recorded at ambient temperature using Bruker Advance 300 NMR, Bruker ARX 400 NMR spectrometers. Multiplets were assigned as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad), coupling constant (Hz), and integration. All ^{13}C NMR spectra were broad-band ^{1}H decoupled. Gas chromatography (GC) analyses were performed on an Agilent HP-7890A instrument with a FID detector and HP-5 capillary column (polydimethylsiloxane with 5% phenyl groups, 30 m, 0.32 mm i.d. 0.25 µm film thickness) using argon as carrier gas. High resolution mass spectra (HRMS) were recorded on an Agilent 6210 system. Because of the high toxicity of carbon monoxide, all reactions should be performed in an autoclave. The laboratory should be well-equipped with a CO detector and alarm system.

2. Optimization of Reaction Conditions

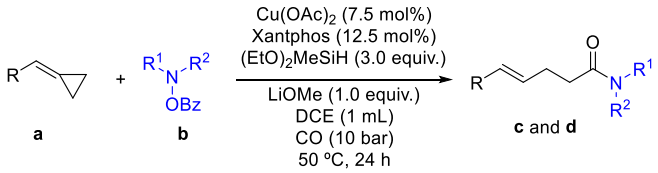


[Cu] (a mol%)	Ligand (b mol%)	Silane (c equiv.)	Base (d equiv.)	Solvent (1 mL)	T (°C)	Yield (%)
CuCl (10)	Xantphos (10)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	Cycloheptane	50	24
CuCl (10)	Xantphos (10)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	Toluene	50	15
CuCl (10)	Xantphos (10)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	THF	50	10
CuCl (10)	Xantphos (10)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCM	50	13
CuCl (10)	Xantphos (10)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	MeCN	50	13
CuCl (10)	Xantphos (10)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	1,4-Dioxane	50	22
CuCl (10)	Xantphos (10)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DMAc	50	trace
CuCl (10)	Xantphos (10)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DMSO	50	0
CuCl (10)	Xantphos (10)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	43
CuCl (10)	Xantphos (10)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	*n*-Hexane	50	14
CuCl (10)	Xantphos (10)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	PhF	50	22
CuCl (10)	Xantphos (10)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	PhCl	50	26
CuCl (10)	Xantphos (10)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	$PhCF_3$	50	18
CuCl (5)	Xantphos (5)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	32
CuCl (7.5)	Xantphos (7.5)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	54
CuCl (10)	Xantphos (10)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	43
CuCl (12.5)	Xantphos (12.5)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	42
CuCl (15)	Xantphos (15)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	48
CuCl (20)	Xantphos (50)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	38
CuCl (7.5)	Xantphos (10)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	43
CuCl (7.5)	Xantphos (12.5)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	57
CuCl (7.5)	Xantphos (15)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	59
CuCl (7.5)	Xantphos (17.5)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	53
CuCl (7.5)	DPPP (15)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	0
CuCl (7.5)	BINAP (15)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	0
CuCl (7.5)	DPPB (15)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	0
CuCl (7.5)	DPPE (15)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	0
CuCl (7.5)	DPPBz (15)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	0
CuCl (7.5)	DPPF (15)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	28
CuCl (7.5)	Xantphos (15)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	57
CuCl (7.5)	**L1** (15)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	27
CuCl (7.5)	**L2** (15)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	0
$CuCl_2$ (7.5)	Xantphos (15)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	34
CuCN (7.5)	Xantphos (15)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	0
$Cu(OAc)_2$ (7.5)	Xantphos (15)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	72
CuI (7.5)	Xantphos (15)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	29
$Cu(OTf)_2$ (7.5)	Xantphos (15)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	49
$Cu(acac)_2$ (7.5)	Xantphos (15)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	55
$CuSO_4$ (7.5)	Xantphos (15)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	11
CuCl (7.5)	Xantphos (15)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	57
$Cu(OAc)_2$ (7.5)	Xantphos (15)	Et_2SiH_2 (3)	NaO^{t}Bu (3)	DCE	50	0
$Cu(OAc)_2$ (7.5)	Xantphos (15)	Et_2SiH_2 (3)	KO^{t}Bu (3)	DCE	50	0
$Cu(OAc)_2$ (7.5)	Xantphos (15)	Et_2SiH_2 (3)	NaOEt (3)	DCE	50	49
$Cu(OAc)_2$ (7.5)	Xantphos (15)	Et_2SiH_2 (3)	NaOMe (3)	DCE	50	34
$Cu(OAc)_2$ (7.5)	Xantphos (15)	Et_2SiH_2 (3)	LiOMe (3)	DCE	50	79
$Cu(OAc)_2$ (7.5)	Xantphos (15)	Et_2SiH_2 (3)	NaOPh (3)	DCE	50	0
$Cu(OAc)_2$ (7.5)	Xantphos (15)	Et_2SiH_2 (3)	KOMe (3)	DCE	50	0
$Cu(OAc)_2$ (7.5)	Xantphos (15)	Et_2SiH_2 (3)	LiO^{t}Bu (3)	DCE	50	72

[Cu] (a mol%)	Ligand (b mol%)	Silane (c equiv.)	Base (d equiv.)	Solvent (1 mL)	T (ºC)	Yield (%)
$Cu(OAc)_2$ (7.5)	Xantphos (15)	Et_3SiH (3)	LiOMe (3)	DCE	50	0
$Cu(OAc)_2$ (7.5)	Xantphos (15)	$(EtO)_3SiH$ (3)	LiOMe (3)	DCE	50	0
$Cu(OAc)_2$ (7.5)	Xantphos (15)	$(EtO)_2MeSiH$ (3)	LiOMe (3)	DCE	50	83
$Cu(OAc)_2$ (7.5)	Xantphos (15)	Ph_2SiH_2 (3)	LiOMe (3)	DCE	50	80
$Cu(OAc)_2$ (7.5)	Xantphos (15)	$PhSiH_3$ (3)	LiOMe (3)	DCE	50	23
$Cu(OAc)_2$ (7.5)	Xantphos (15)	PhMeSiH (3)	LiOMe (3)	DCE	50	0
$Cu(OAc)_2$ (7.5)	Xantphos (15)	$(TMS)_3SiH$ (3)	LiOMe (3)	DCE	50	0
$Cu(OAc)_2$ (7.5)	Xantphos (15)	$(MeO)_2MeSiH$ (3)	LiOMe (3)	DCE	50	78
$Cu(OAc)_2$ (7.5)	Xantphos (15)	Et_2SiH_2 (3)	LiOMe (3)	DCE	50	79
$Cu(OAc)_2$ (7.5)	Xantphos (15)	$PhMeSiH_2$ (3)	LiOMe (3)	DCE	50	77
$Cu(OAc)_2$ (7.5)	Xantphos (15)	$(EtO)_2MeSiH$ (3)	LiOMe (1)	DCE	50	86
$Cu(OAc)_2$ (7.5)	Xantphos (15)	$(EtO)_2MeSiH$ (3)	LiO^{t}Bu (2)	DCE	50	73
$Cu(OAc)_2$ (7.5)	Xantphos (15)	$(EtO)_2MeSiH$ (3)	LiO^{t}Bu (2.5)	DCE	50	66
$Cu(OAc)_2$ (7.5)	Xantphos (15)	$(EtO)_2MeSiH$ (3)	LiO^{t}Bu (3)	DCE	50	83
$Cu(OAc)_2$ (7.5)	Xantphos (15)	$(EtO)_2MeSiH$ (3)	LiO^{t}Bu (3.5)	DCE	50	76
$Cu(OAc)_2$ (7.5)	Xantphos (15)	$(EtO)_2MeSiH$ (1.5)	LiOMe (1)	DCE	50	60
$Cu(OAc)_2$ (7.5)	Xantphos (15)	$(EtO)_2MeSiH$ (2.0)	LiOMe (1)	DCE	50	60
$Cu(OAc)_2$ (7.5)	Xantphos (15)	$(EtO)_2MeSiH$ (2.5)	LiOMe (1)	DCE	50	81
$Cu(OAc)_2$ (7.5)	Xantphos (15)	$(EtO)_2MeSiH$ (3.0)	LiOMe (1)	DCE	50	86
$Cu(OAc)_2$ (7.5)	Xantphos (15)	$(EtO)_2MeSiH$ (4.0)	LiOMe (1)	DCE	50	74
$Cu(OAc)_2$ (7.5)	Xantphos (15)	$(EtO)_2MeSiH$ (3.0)	LiOMe (1)	DCE	40	72
$Cu(OAc)_2$ (7.5)	Xantphos (15)	$(EtO)_2MeSiH$ (3.0)	LiOMe (1)	DCE	50	86
$Cu(OAc)_2$ (7.5)	Xantphos (15)	$(EtO)_2MeSiH$ (3.0)	LiOMe (1)	DCE	60	62
$Cu(OAc)_2$ (7.5)	Xantphos (15)	$(EtO)_2MeSiH$ (3.0)	LiOMe (1)	DCE	70	60

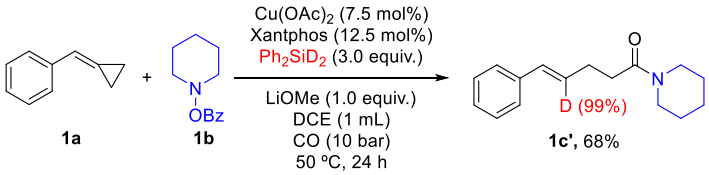
3. Synthetic Procedure

3.1 General procedure



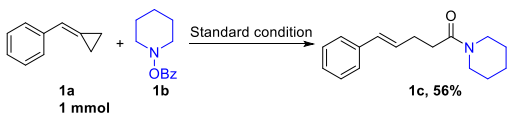
An oven dried 4 mL screw-cap vial equipped with a septum and a stirring bar was charged with $Cu(OAc)_2$ (7.5 mol%), DPPP (12.5 mol%), **b** (1.5 equiv.) and LiOMe (1.0 equiv.). The vial was sealed, connected to atmosphere with a small cannula, evacuated, and backfilled with argon three times. Then DCE (1.0 mL) was added *via* syringe. To this suspension, BCPs **a** (0.2 mmol) and $(EtO)_2MeSiH_2$ (3.0 equiv.) were added *via* Hamilton® syringe. The vial was placed on an alloy plate and transferred into a 300 mL stovetop autoclave (4560 series from Parr instrument company®). The autoclave was flushed one time with nitrogen (<10 bar) and three times with CO (<10 bar). The autoclave was then charged with CO (10 bar). The autoclave was then placed into an aluminum block on a magnetic stirrer. The reaction mixture was stirred (500 rpm) for 24 h at 50 °C. Then it was cooled to room temperature and the pressure was released carefully. The reaction mixture was added water (1 mL) and extracted by DCE (1 mL x 2). The organic phase was dried by Na_2SO_4 and removed under vacuum. The reaction purified by gradient column chromatography to yield products **c** or **d**.

3.2 Isotope labeling experiment.



1c' Pentane/ethyl acetate = 8/3, colorless oil (33.2 mg, 68% yield).
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.28 – 7.10 (m, 5H), 6.35 (s, 1H), 3.59 – 3.28 (m, 4H), 2.50 – 2.38 (m, 4H), 1.60 – 1.44 (m, 6H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 170.5, 137.5, 130.5, 128.5, 127.0, 126.0, 46.8, 42.8, 33.0, 28.7, 26.5, 25.7, 24.6.

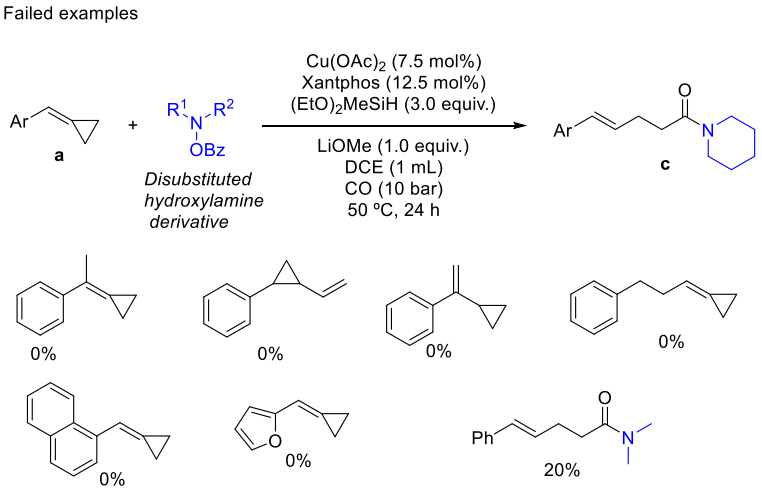
3.3 1 mmol scale reaction



An oven dried 12 mL screw-cap vial equipped with a septum and a stirring bar was charged with $Cu(OAc)_2$ (7.5 mol%), DPPP (12.5 mol%), **1b** (1.5 equiv.) and LiOMe (1.0 equiv.). The vial was sealed, connected to atmosphere with a small cannula, evacuated, and backfilled with argon three times. Then DCE (5.0 mL) was added *via* syringe. To this suspension, BCPs **1a** (1 mmol) and $(EtO)_2MeSiH_2$ (3.0 equiv.) were added *via* Hamilton® syringe. The vial was placed on an alloy plate and transferred into a 300 mL

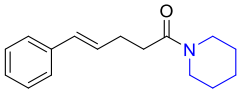
stovetop autoclave (4560 series from Parr instrument company®). The autoclave was flushed one time with nitrogen (<10 bar) and three times with CO (<10 bar). The autoclave was then charged with CO (10 bar). The autoclave was then placed into an aluminum block on a magnetic stirrer. The reaction mixture was stirred (500 rpm) for 24 h at 50 °C. Then it was cooled to room temperature and the pressure was released carefully. The reaction mixture was added water (3 mL) and extracted by DCE (3 mL x 2). The organic phase was dried by Na_2SO_4 and removed under vacuum. The reaction purified by gradient column chromatography (Pentane/ethyl acetate = 8/3) to yield product 1**c** in 56% yield (136.1 mg).

3.4 Failed example



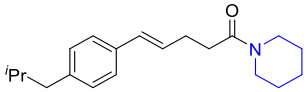
4 Analytic data of the Products

(*E*)-5-Phenyl-1-(piperidin-1-yl)pent-4-en-1-one (1c)



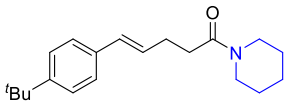
Pentane/ethyl acetate = 8/3, colorless oil (37.9 mg, 78% yield). **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.21 (dd, *J* = 8.3, 1.3 Hz, 2H), 7.18 – 7.14 (m, 2H), 7.09 – 7.05 (m, 1H), 6.31 (d, *J* = 15.9 Hz, 1H), 6.14 (dt, *J* = 15.8, 6.6 Hz, 1H), 3.43 – 3.30 (m, 4H), 2.46 – 2.34 (m, 4H), 1.55 – 1.42 (m, 6H). **^{13}C NMR (101 MHz, CDCl$_3$)** δ 170.5, 137.5, 130.6, 129.6, 128.5, 127.0, 126.0, 46.6, 42.7, 33.1, 28.9, 26.5, 25.7, 24.6. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{16}H_{22}NO^+$ ([M+H]$^+$) 244.1696, found 244.1701.

(*E*)-5-(4-Isobutylphenyl)-1-(piperidin-1-yl)pent-4-en-1-one (2c)



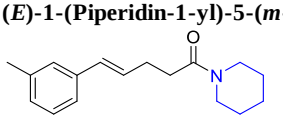
Pentane/ethyl acetate = 8/3, colorless oil (43.1 mg, 72% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.18 (d, *J* = 7.9 Hz, 2H), 6.99 (d, *J* = 8.1 Hz, 2H), 6.34 (d, *J* = 15.9 Hz, 1H), 6.15 (dt, *J* = 15.8, 6.6 Hz, 1H), 3.49 (t, *J* = 5.0 Hz, 2H), 3.33 (t, *J* = 5.0 Hz, 2H), 2.50 – 2.34 (m, 6H), 1.82 – 1.72 (m, 1H), 1.60 – 1.43 (m, 6H), 0.83 (s, 3H), 0.80 (s, 3H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 170.5, 140.7, 135.0, 130.5, 129.3, 128.6, 125.8, 46.7, 45.1, 42.7, 33.2, 31.5, 30.2, 28.9, 26.5, 25.6, 24.6, 22.4. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{20}H_{30}NO^+$ ([M+H]$^+$) 300.2322, found 300.2325.

(*E*)-5-(4-(*tert*-Butyl)phenyl)-1-(piperidin-1-yl)pent-4-en-1-one (3c)



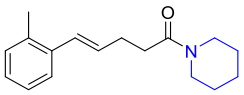
Pentane/ethyl acetate = 8/3, colorless oil (73.6 mg, 73% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.24 – 7.17 (m, 4H), 6.34 (d, *J* = 15.9 Hz, 1H), 6.15 (dt, *J* = 15.8, 6.6 Hz, 1H), 3.42 (d, *J* = 25.9 Hz, 4H), 2.53 – 2.28 (m, 4H), 1.60 – 1.43 (m, 6H), 1.23 (s, 9H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 170.5, 150.0, 134.7, 130.3, 128.7, 125.7, 125.4, 34.5, 33.2, 31.3, 28.8, 24.6. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{20}H_{30}NO^+$ ([M+H]$^+$) 300.2322, found 300.2327.

(*E*)-1-(Piperidin-1-yl)-5-(*m*-tolyl)pent-4-en-1-one (4c)



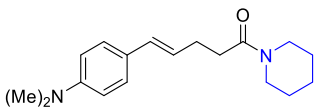
Pentane/ethyl acetate = 8/3, colorless oil (34.4 mg, 67% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.19 – 7.09 (m, 3H), 7.04 – 6.97 (m, 1H), 6.40 (d, *J* = 15.9 Hz, 1H), 6.25 (dt, *J* = 15.8, 6.4 Hz, 1H), 3.49 (s, 4H), 2.51 (dtd, *J* = 12.2, 6.3, 1.9 Hz, 4H), 2.33 (s, 3H), 1.66 – 1.51 (m, 6H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 170.5, 138.0, 137.4, 130.6, 129.3, 128.3, 127.8, 126.7, 123.1, 46.6, 42.8, 33.1, 28.8, 24.5, 21.4. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{17}H_{24}NO^+$ ([M+H]$^+$) 258.1852, found 258.1854.

(*E*)-1-(Piperidin-1-yl)-5-(*o*-tolyl)pent-4-en-1-one (5c)



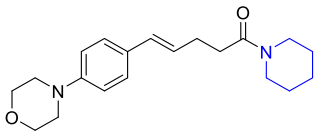
Pentane/ethyl acetate = 8/3, colorless oil (30.3 mg, 59% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.43 – 7.38 (m, 1H), 7.16 – 7.09 (m, 3H), 6.63 (d, *J* = 15.7 Hz, 1H), 6.13 (dt, *J* = 15.7, 6.7 Hz, 1H), 3.49 (d, *J* = 35.8 Hz, 4H), 2.60 – 2.46 (m, 4H), 2.32 (s, 3H), 1.61 (ddd, *J* = 19.8, 12.2, 4.1 Hz, 6H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 170.4, 136.6, 134.9, 130.8, 130.1, 128.4, 126.9, 126.0, 125.4, 46.6, 42.7, 33.2, 29.1, 26.5, 25.6, 24.5, 19.8. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{17}H_{24}NO^+$ ([M+H]$^+$) 258.1852, found 258.1856.

(*E*)-5-(4-(Dimethylamino)phenyl)-1-(piperidin-1-yl)pent-4-en-1-one (6c)



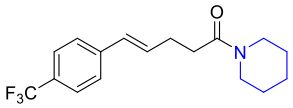
Pentane/ethyl acetate = 1/1, colorless oil (37.1 mg, 65% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.23 (d, *J* = 8.7 Hz, 2H), 6.68 (d, *J* = 8.7 Hz, 2H), 6.34 (d, *J* = 15.8 Hz, 1H), 6.05 (dt, *J* = 15.8, 6.6 Hz, 1H), 3.56 (t, *J* = 6.0 Hz, 2H), 3.40 (t, *J* = 6.0 Hz, 2H), 2.94 (s, 6H), 2.54 – 2.42 (m, 4H), 1.67 – 1.51 (m, 6H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 170.6, 149.6, 130.2, 126.8, 125.3, 112.6, 46.6, 42.6, 40.6, 33.4, 28.9, 26.5, 25.5, 24.5. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{18}H_{27}N_2O^+$ ([M+H]$^+$) 287.2118, found 287.2123.

(*E*)-5-(4-Morpholinophenyl)-1-(piperidin-1-yl)pent-4-en-1-one (7c)



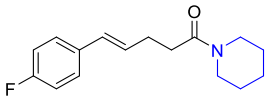
Pentane/ethyl acetate = 1/1, white solid (38.7 mg, 59% yield). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.26 (d, *J* = 8.7 Hz, 2H), 6.84 (d, *J* = 8.5 Hz, 2H), 6.36 (d, *J* = 15.9 Hz, 1H), 6.12 (dt, *J* = 15.8, 6.6 Hz, 1H), 3.91 – 3.78 (m, 4H), 3.62 – 3.50 (m, 2H), 3.46 – 3.33 (m, 2H), 3.24 – 3.10 (m, 4H), 2.62 – 2.33 (m, 4H), 1.68 – 1.50 (m, 6H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 170.5, 129.9, 127.0, 126.8, 115.6, 66.8, 49.3, 46.6, 42.6, 33.3, 31.5, 30.1, 28.8, 26.5, 25.6, 24.5. **HRMS** (ESI-TOF): *m*/*z* calcd. for $C_{20}H_{29}N_2O_2^+$ ($[M+H]^+$) 329.2224, found 329.2229.

(*E*)-1-(Piperidin-1-yl)-5-(4-(trifluoromethyl)phenyl)pent-4-en-1-one (8c)



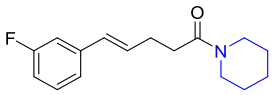
Pentane/ethyl acetate = 2/1, white solid (40.4 mg, 65% yield). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.52 (d, *J* = 8.2 Hz, 2H), 7.42 (d, *J* = 8.2 Hz, 2H), 6.51 – 6.32 (m, 2H), 3.49 (s, 4H), 2.55 (m, 4H), 1.67 – 1.53 (m, 6H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 170.2, 141.0, 132.5, 129.4, 128.7, 126.2, 125.4 (q, *J* = 3.8 Hz), 124.2 (q, *J* = 271.7 Hz), 32.7, 28.8, 24.5. **^{19}F NMR (282 MHz, $CDCl_3$)** δ -62.40. **HRMS** (ESI-TOF): *m*/*z* calcd. for $C_{17}H_{21}F_3NO^+$ ($[M+H]^+$) 312.1570, found 312.1575.

(*E*)-5-(4-Fluorophenyl)-1-(piperidin-1-yl)pent-4-en-1-one (9c)



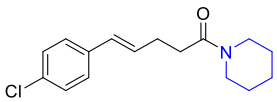
Pentane/ethyl acetate = 8/3, colorless oil (35.5 mg, 68% yield). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.33 – 7.16 (m, 2H), 6.98 – 6.76 (m, 2H), 6.32 (d, *J* = 15.9 Hz, 1H), 6.11 (dt, *J* = 15.8, 6.4 Hz, 1H), 3.41 (d, *J* = 41.5 Hz, 4H), 2.52 – 2.38 (m, 4H), 1.60 – 1.44 (m, 6H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 170.4, 162.0 (d, *J* = 245.7 Hz), 133.7 (d, *J* = 3.2 Hz), 129.4, 129.4 (d, *J* = 2.1 Hz), 127.4 (d, *J* = 7.9 Hz), 115.3 (d, *J* = 21.5 Hz), 46.7, 42.8, 33.0, 28.7, 26.5, 25.6, 24.6. **^{19}F NMR (282 MHz, $CDCl_3$)** δ -115.48. **HRMS** (ESI-TOF): *m*/*z* calcd. for $C_{16}H_{21}FNO^+$ ($[M+H]^+$) 262.1602, found 262.1602.

(*E*)-5-(3-Fluorophenyl)-1-(piperidin-1-yl)pent-4-en-1-one (10c)



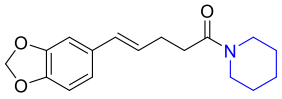
Pentane/ethyl acetate = 8/3, colorless oil (37.6 mg, 72% yield). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.26 – 7.20 (m, 1H), 7.10 – 7.00 (m, 2H), 6.87 (tdd, *J* = 8.5, 2.6, 1.0 Hz, 1H), 6.39 (d, *J* = 16.0 Hz, 1H), 6.27 (dt, *J* = 15.8, 6.2 Hz, 1H), 3.47 (s, 4H), 2.58 – 2.44 (m, 4H), 1.60 (m, 6H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 170.2, 163.1 (d, *J* = 244.8 Hz), 140.0 (d, *J* = 7.8 Hz), 131.1, 129.9 (d, *J* = 8.5 Hz), 129.5 (d, *J* = 2.6 Hz), 121.9 (d, *J* = 2.7 Hz), 113.8 (d, *J* = 21.4 Hz), 112.4 (d, *J* = 21.7 Hz), 46.4, 42.8, 32.8, 28.6, 24.5. **^{19}F NMR (282 MHz, $CDCl_3$)** δ -113.83. **HRMS** (ESI-TOF): *m*/*z* calcd. for $C_{16}H_{21}FNO^+$ ($[M+H]^+$) 262.1602, found 262.1608.

(*E*)-5-(4-Chlorophenyl)-1-(piperidin-1-yl)pent-4-en-1-one (11c)



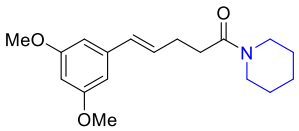
Pentane/ethyl acetate = 2/1, white solid (31.6 mg, 57% yield). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.18 (s, 4H), 6.31 (d, *J* = 15.9, 1H), 6.17 (dt, *J* = 15.8, 6.4 Hz, 1H), 3.41 (d, *J* = 46.3 Hz, 4H), 2.51 – 2.36 (m, 4H), 1.60 – 1.44 (m, 6H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 170.2, 136.0, 132.5, 130.3, 129.3, 128.5, 127.2, 46.6, 42.7, 32.8, 28.7, 26.5, 25.5, 24.5. **HRMS** (ESI-TOF): *m*/*z* calcd. for $C_{16}H_{21}ClNO^+$ ($[M+H]^+$) 278.1306, found 278.1312.

***(E)*-5-(Benzo[*d*][1,3]dioxol-5-yl)-1-(piperidin-1-yl)pent-4-en-1-one (12c)**



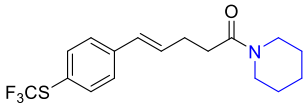
Pentane/ethyl acetate = 1/1, white solid (40.2 mg, 70% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 6.89 (d, *J* = 1.5 Hz, 1H), 6.77 – 6.70 (m, 2H), 6.34 (d, *J* = 15.8 Hz, 1H), 6.14 – 6.04 (m, 1H), 5.93 (s, 2H), 3.48 (s, 4H), 2.54 – 2.44 (m, 4H), 1.66 – 1.52 (m, 6H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 170.5, 147.9, 146.7, 132.0, 130.1, 127.8, 120.4, 108.2, 105.4, 100.9, 33.1, 28.7, 24.5. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{17}H_{22}NO_3^+$ ([M+H]$^+$) 288.1594, found 288.1604.

(*E*)-5-(3,5-Dimethoxyphenyl)-1-(piperidin-1-yl)pent-4-en-1-one (13c)



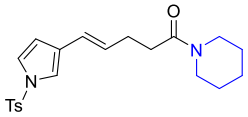
Pentane/ethyl acetate = 1/1, colorless oil (40.6 mg, 67% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 6.51 (d, *J* = 2.3 Hz, 2H), 6.41 – 6.34 (m, 2H), 6.27 (dt, *J* = 15.8, 6.2 Hz, 1H), 3.80 (s, 6H), 3.53 (d, *J* = 17.3 Hz, 4H), 2.61 – 2.48 (m, 4H), 1.68 – 1.54 (m, 6H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 170.3, 160.8, 139.5, 130.5, 130.1, 104.0, 99.3, 55.2, 46.7, 42.8, 32.8, 28.7, 24.5. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{18}H_{26}NO_3^+$ ([M+H]$^+$) 304.1907, found 304.1917.

(*E*)-1-(Piperidin-1-yl)-5-(4-((trifluoromethyl)thio)phenyl)pent-4-en-1-one (14c)



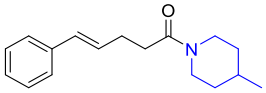
Pentane/ethyl acetate = 2/1, colorless oil (34 mg, 35% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.56 (d, *J* = 8.3 Hz, 2H), 7.39 – 7.35 (m, 2H), 6.48 – 6.30 (m, 2H), 3.49 (s, 4H), 2.60 – 2.46 (m, 4H), 1.67 – 1.52 (m, 6H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 170.2, 140.2, 136.5, 132.4, 129.6 (q, *J* = 307.9 Hz), 129.4, 126.9, 32.7, 28.8, 24.5. **^{19}F NMR (282 MHz, CDCl$_3$)** δ -42.98. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{17}H_{21}F_3NOS^+$ ([M+H]$^+$) 344.1290, found 344.1295.

(*E*)-1-(Piperidin-1-yl)-5-(1-tosyl-1*H*-pyrrol-3-yl)pent-4-en-1-one (15c)



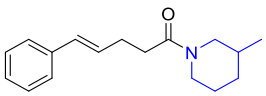
Pentane/ethyl acetate = 1/1, brown oil (42.5 mg, 55% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.50 (d, *J* = 8.4 Hz, 2H), 7.13 – 7.07 (m, 4H), 6.62 (d, *J* = 15.8 Hz, 1H), 6.03 (td, *J* = 3.4, 0.5 Hz, 1H), 5.83 (dt, *J* = 15.8, 6.6 Hz, 1H), 3.43 – 3.38 (m, 2H), 3.28 – 3.19 (m, 2H), 2.36 – 2.27 (m, 4H), 2.23 (s, 3H), 1.48 – 1.35 (m, 6H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 170.1, 144.8, 136.0, 133.7, 131.5, 129.8, 126.9, 122.4, 119.1, 112.1, 111.0, 46.5, 42.7, 32.6, 28.5, 26.5, 25.5, 24.5, 21.6. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{21}H_{27}N_2O_3S^+$ ([M+H]$^+$) 387.1737, found 387.1736.

(*E*)-1-(4-Methylpiperidin-1-yl)-5-phenylpent-4-en-1-one (16c)



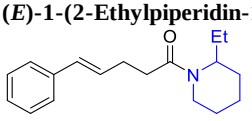
Pentane/ethyl acetate = 8/3, colorless oil (45.7 mg, 89% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.20 – 7.09 (m, 4H), 7.05 – 6.99 (m, 1H), 6.26 (d, *J* = 15.9 Hz, 1H), 6.10 (dt, *J* = 15.8, 6.5 Hz, 1H), 4.43 (ddd, *J* = 10.8, 4.0, 2.0 Hz, 1H), 3.79 – 3.46 (m, 1H), 2.81 (td, *J* = 13.2, 2.5 Hz, 1H), 2.43 – 2.28 (m, 5H), 1.53 – 1.39 (m, 3H), 0.98 – 0.82 (m, 2H), 0.76 (d, *J* = 6.4 Hz, 3H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 170.4, 137.5, 130.6, 129.6, 128.5, 127.0, 126.0, 46.0, 42.1, 34.7, 33.8, 33.1, 31.1, 28.9, 21.7. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{17}H_{24}NO^+$ ([M+H]$^+$) 258.1852, found 258.1855.

(*E*)-1-(3-Methylpiperidin-1-yl)-5-phenylpent-4-en-1-one (17c)



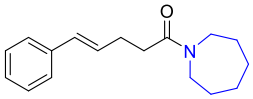
Pentane/ethyl acetate = 1/1, colorless oil (37.1 mg, 72% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.22 (dd, *J* = 8.4, 1.5 Hz, 2H), 7.16 (tt, *J* = 6.2, 1.4 Hz, 2H), 7.11 – 7.03 (m, 1H), 6.31 (d, *J* = 15.9 Hz, 1H), 6.14 (dt, *J* = 15.8, 6.5 Hz, 1H), 4.33 (d, *J* = 8.3 Hz, 1H), 3.67 – 3.51 (m, 1H), 3.05 – 2.56 (m, 1H), 2.57 – 2.33 (m, 5H), 1.72 – 1.25 (m, 5H), 0.78 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 170.4, 137.5, 130.6, 129.6, 128.5, 127.0, 126.0, 53.2, 49.2, 33.1, 33.1, 28.8, 26.0, 24.8, 19.0. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{17}H_{24}NO^+$ ([M+H]$^+$) 258.1852, found 258.1860.

(*E*)-1-(2-Ethylpiperidin-1-yl)-5-phenylpent-4-en-1-one (18c)



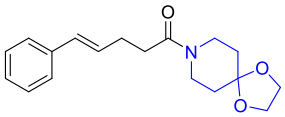
Pentane/ethyl acetate = 4/1, colorless oil (22.2 mg, 41% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.17 – 7.06 (m, 4H), 7.03 – 6.96 (m, 1H), 6.24 (d, *J* = 15.9 Hz, 1H), 6.07 (dt, *J* = 15.9, 6.3 Hz, 1H), 4.70 – 4.28 (m, 1H), 3.68 – 3.43 (m, 1H), 2.33 (dt, *J* = 12.5, 6.1 Hz, 5H), 1.54 – 1.15 (m, 8H), 0.72 – 0.62 (m, 3H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 170.9, 137.5, 130.6, 129.6, 128.5, 127.0, 126.0, 54.5, 49.5, 29.0,26.5, 26.4, 23.0, 22.3, 19.0, 10.7. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{18}H_{26}NO^+$ ([M+H]$^+$) 272.2009, found 272.2010.

(*E*)-1-(Azepan-1-yl)-5-phenylpent-4-en-1-one (19c)



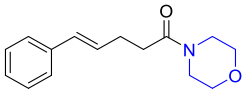
Pentane/ethyl acetate = 8/3, colorless oil (34.9 mg, 68% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.37 – 7.27 (m, 4H), 7.24 – 7.18 (m, 1H), 6.46 (d, *J* = 15.9 Hz, 1H), 6.29 (dt, *J* = 15.8, 6.5 Hz, 1H), 3.56 (t, *J* = 6.0 Hz, 2H), 3.45 (t, *J* = 6.0 Hz, 2H), 2.70 – 2.46 (m, 4H), 1.78 – 1.69 (m, 4H), 1.57 (dt, *J* = 5.9, 2.7 Hz, 4H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 171.7, 137.5, 130.5, 129.6, 128.4, 126.9, 125.9, 47.8, 45.9, 32.9, 29.1, 28.7, 27.6, 27.0, 26.8. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{17}H_{24}NO^+$ ([M+H]$^+$) 258.1852, found 258.1858.

(*E*)-5-Phenyl-1-(1,4-dioxa-8-azaspiro[4.5]decan-8-yl)pent-4-en-1-one (20c)



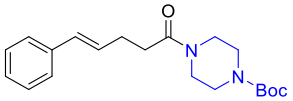
Pentane/ethyl acetate = 3/2, white solid (51.2 mg, 85% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.27 – 7.18 (m, 4H), 7.13 – 7.10 (m, 1H), 6.35 (d, *J* = 15.9 Hz, 1H), 6.18 (dt, *J* = 15.8, 6.5 Hz, 1H), 3.88 (s, 4H), 3.55 (d, *J* = 48.4 Hz, 4H), 2.56 – 2.40 (m, 4H), 1.63 – 1.57 (m, 4H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 170.3, 137.3, 130.6, 129.2, 128.4, 126.9, 125.9, 106.8, 64.3, 43.4, 39.7, 35.5, 34.7, 32.9, 28.6. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{18}H_{24}NO_3^+$ ([M+H]$^+$) 302.1751, found 302.1758.

(*E*)-1-Morpholino-5-phenylpent-4-en-1-one (21c)



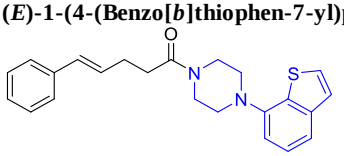
Pentane/ethyl acetate = 1/1, colorless oil (41.2 mg, 84% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.37 – 7.28 (m, 4H), 7.24 – 7.19 (m, 1H), 6.46 (d, *J* = 15.9 Hz, 1H), 6.27 (dt, *J* = 15.8, 6.5 Hz, 1H), 3.69 – 3.67 (m, 8H), 2.63 – 2.47 (m, 4H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 170.9, 137.4, 130.9, 129.1, 128.5, 127.2, 126.0, 66.9, 66.7, 46.0, 42.0, 32.8, 28.6. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{15}H_{20}NO_2^+$ ([M+H]$^+$) 246.1489, found 246.1490.

***tert*-Butyl (*E*)-4-(5-phenylpent-4-enoyl)piperazine-1-carboxylate (22c)**



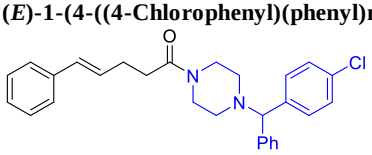
Pentane/ethyl acetate = 2/1, yellow solid (57.8 mg, 84% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.36 – 7.27 (m, 4H), 7.24 – 7.17 (m, 1H), 6.45 (d, *J* = 14.6 Hz, 1H), 6.26 (dt, *J* = 15.8, 6.5 Hz, 1H), 3.62 – 3.62 (m, 8H), 2.66 – 2.48 (m, 4H), 1.48 (s, 9H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 170.8, 154.6, 137.4, 130.9, 129.1, 128.5, 127.1, 126.0, 80.3, 45.3, 41.4, 33.0, 28.6, 28.4. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{20}H_{29}N_2O_3^+$ ([M+H]$^+$) 345.2173, found 345.2186.

(*E*)-1-(4-(Benzo[*b*]thiophen-7-yl)piperazin-1-yl)-5-phenylpent-4-en-1-one (23c)



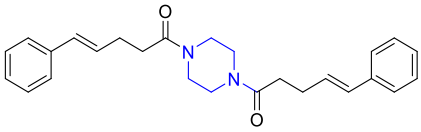
Pentane/ethyl acetate = 2/1, colorless oil (57.9 mg, 77% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.63 (d, *J* = 8.0 Hz, 1H), 7.46 (s, 2H), 7.41 – 7.24 (m, 6H), 6.87 (dd, *J* = 7.7, 0.7 Hz, 1H), 6.51 (d, *J* = 15.9 Hz, 1H), 6.33 (dt, *J* = 15.8, 6.5 Hz, 1H), 3.97 – 3.86 (m, 2H), 3.79 – 3.67 (m, 2H), 3.18 – 3.05 (m, 4H), 2.69 – 2.54 (m, 4H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 170.9, 147.7, 141.3, 137.5, 134.2, 130.9, 129.3, 128.6, 127.2, 126.1, 125.6, 125.0, 121.5, 117.8, 112.6, 52.3, 52.3, 46.0, 42.0, 33.0, 28.8. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{23}H_{25}N_2OS^+$ ([M+H]$^+$) 377.1682, found 377.1684.

(*E*)-1-(4-((4-Chlorophenyl)(phenyl)methyl)piperazin-1-yl)-5-phenylpent-4-en-1-one (24c)



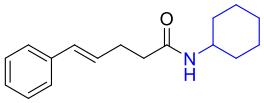
Pentane/ethyl acetate = 8/3, colorless oil (71.0 mg, 80% yield). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.19 – 7.00 (m, 14H), 6.24 (d, *J* = 15.9 Hz, 1H), 6.06 (dt, *J* = 15.8, 6.6 Hz, 1H), 3.99 (s, 1H), 3.51 – 3.38 (m, 2H), 3.35 – 3.22 (m, 2H), 2.40 – 2.24 (m, 4H), 2.20 – 2.08 (m, 4H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 170.6, 141.6, 140.8, 137.5, 132.8, 130.8, 129.3, 129.1, 128.8, 128.8, 128.5, 127.8, 127.4, 127.1, 126.1, 75.3, 52.0, 51.6, 45.7, 41.8, 32.9, 28.8. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{28}H_{30}ClN_2O^+$ ($[M+H]^+$) 445.2041, found 445.2054.

(4*E*,4'*E*)-1,1'-(Piperazine-1,4-diyl)bis(5-phenylpent-4-en-1-one) (25c)



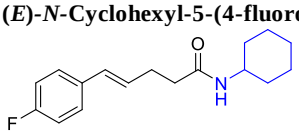
Ethyl acetate, white solid (18.1 mg, 45% yield). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.28 – 7.18 (m, 8H), 7.18 – 7.09 (m, 2H), 6.37 (d, *J* = 15.9 Hz, 2H), 6.18 (dt, *J* = 15.8, 6.6 Hz, 2H), 3.57 (s, 4H), 3.41 (s, 4H), 2.55 – 2.34 (m, 8H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 171.1, 137.3, 131.0, 128.9, 128.6, 127.2, 126.0, 45.3, 41.6, 33.0, 28.5. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{26}H_{31}N_2O_2^+$ ($[M+H]^+$) 403.2380, found 403.2375.

(*E*)-*N*-Cyclohexyl-5-phenylpent-4-enamide (1d)



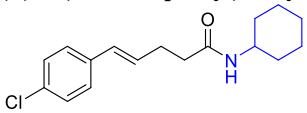
Ethyl acetate, white solid (37 mg, 72% yield). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.32 (dd, *J* = 7.8, 1.2 Hz, 4H), 7.22 (dt, *J* = 3.7, 2.3 Hz, 1H), 6.46 (d, *J* = 15.9 Hz, 1H), 6.23 (dt, *J* = 15.8, 6.8 Hz, 1H), 5.42 (d, *J* = 5.5 Hz, 1H), 3.85 – 3.77 (m, 1H), 2.61 – 2.54 (m, 2H), 2.36 – 2.31 (m, 2H), 1.92 (dd, *J* = 12.3, 3.6 Hz, 2H), 1.69 (q, *J* = 4.3 Hz, 3H), 1.45 – 1.36 (m, 2H), 1.18 – 1.09 (m, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 171.2, 137.4, 131.0, 128.9, 128.5, 127.1, 126.0, 48.1, 36.6, 33.2, 29.2, 25.5, 24.8. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{17}H_{24}NO^+$ ($[M+H]^+$) 258.1852, found 258.1860.

(*E*)-*N*-Cyclohexyl-5-(4-fluorophenyl)pent-4-enamide (2d)



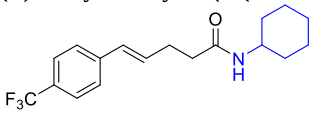
Pentane/ethyl acetate = 2/1, white solid (35.7 mg, 65% yield). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.29 – 7.25 (m, 2H), 7.00 – 6.93 (m, 2H), 6.39 (d, *J* = 15.8 Hz, 1H), 6.11 (dt, *J* = 15.8, 6.9 Hz, 1H), 5.41 (d, *J* = 4.5 Hz, 1H), 3.83 – 3.70 (m, 1H), 2.57 – 2.49 (m, 2H), 2.33 – 2.27 (m, 2H), 1.98 – 1.86 (m, 2H), 1.71 – 1.59 (m, 3H), 1.29 – 1.21 (m, 2H), 1.16 – 1.02 (m, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 171.1, 133.56 (d, *J* = 3.4 Hz), 162.04 (d, *J* = 246.0 Hz), 128.60 (d, *J* = 2.0 Hz), 127.45 (d, *J* = 8.0 Hz), 127.4, 115.37 (d, *J* = 21.6 Hz), 48.2, 36.5, 33.2, 29.1, 25.5, 24.8. **^{19}F NMR (282 MHz, $CDCl_3$)** δ -115.31. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{17}H_{23}FNO^+$ ($[M+H]^+$) 276.1758, found 276.1761.

(*E*)-5-(4-Chlorophenyl)-*N*-cyclohexylpent-4-enamide (3d)



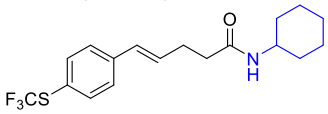
Pentane/ethyl acetate = 2/1, white solid (44.8 mg, 77% yield). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.23 (s, 4H), 6.37 (dt, *J* = 15.8, 1.3 Hz, 1H), 6.17 (dt, *J* = 15.8, 6.8 Hz, 1H), 5.47 (d, *J* = 6.5 Hz, 1H), 3.83 – 3.71 (m, 1H), 2.58 – 2.47 (m, 2H), 2.34 – 2.23 (m, 2H), 1.92 – 1.82 (m, 2H), 1.71 – 1.56 (m, 3H), 1.41 – 1.32 (m, 2H), 1.18 – 1.02 (m, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 171.0, 135.9, 132.7, 129.8, 129.6, 128.6, 127.2, 48.2, 36.4, 33.2, 29.1, 25.5, 24.9. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{17}H_{23}ClNO^+$ ($[M+H]^+$) 292.1463, found 292.1471.

(*E*)-*N*-Cyclohexyl-5-(4-(trifluoromethyl)phenyl)pent-4-enamide (4d)



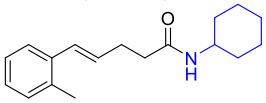
Pentane/ethyl acetate = 1/1, white solid (40.3 mg, 62% yield). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.45 (d, *J* = 8.2 Hz, 2H), 7.33 (d, *J* = 8.1 Hz, 2H), 6.39 (d, *J* = 15.9 Hz, 1H), 6.24 (dt, *J* = 15.8, 6.6 Hz, 1H), 5.42 (d, *J* = 6.1 Hz, 1H), 3.77 – 3.63 (m 1H), 2.51 (q, *J* = 7.3 Hz, 2H), 2.26 (t, *J* = 7.4 Hz, 2H), 1.82 (dd, *J* = 12.4, 3.4 Hz, 2H), 1.65 – 1.50 (m, 3H), 1.37 – 1.17 (m, 2H), 1.10 – 0.98 (m, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 171.9, 140.8, 131.7, 129.7, 128.9 (q, *J* = 32.5 Hz), 126.1, 125.4 (q, *J* = 3.8 Hz), 48.2, 36.2, 33.2, 29.0, 25.4, 24.8. **^{19}F NMR (282 MHz, $CDCl_3$)** δ -62.43. **HRMS** (ESI-TOF): *m/z* calcd. for $C_{18}H_{23}F_3NO^+$ ($[M+H]^+$) 326.1726, found 326.1733.

(*E*)-*N*-Cyclohexyl-5-(4-((trifluoromethyl)thio)phenyl)pent-4-enamide (5d)



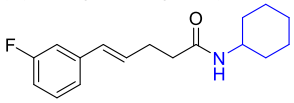
Pentane/ethyl acetate = 1/1, white solid (44.9 mg, 63% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.55 (d, *J* = 8.3 Hz, 2H), 7.37 – 7.32 (m, 2H), 6.43 (d, *J* = 15.9 Hz, 1H), 6.30 (dt, *J* = 15.8, 6.6 Hz, 1H), 5.53 (d, *J* = 6.9 Hz, 1H), 3.83 – 3.70 (m, 1H), 2.61 – 2.52 (m, 2H), 2.32 (t, *J* = 7.4 Hz, 2H), 1.92 – 1.84 (m, 2H), 1.80 – 1.43 (m, 4H), 1.37 – 1.28 (m, 2H), 1.15 – 1.08 (m, 2H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 170.9, 140.1, 136.6, 131.7, 129.7, 129.6 (q, *J* = 301.2 Hz), 126.9, 122.4 (q, *J* = 2.26 Hz), 48.3, 36.2, 33.2, 29.1, 25.5, 24.8. **^{19}F NMR (282 MHz, CDCl$_3$)** δ -42.95. **HRMS** (ESI-TOF): *m*/*z* calcd. for $C_{18}H_{23}F_3NOS^+$ ([M+H]$^+$) 358.1447, found 358.1455.

(*E*)-*N*-Cyclohexyl-5-(*o*-tolyl)pent-4-enamide (6d)



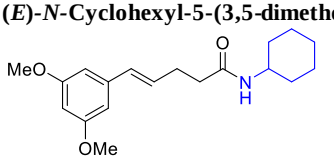
Pentane/ethyl acetate = 2/1, white solid (35.7 mg, 66% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.40 – 7.35 (m, 1H), 7.16 – 7.08 (m, 3H), 6.63 (d, *J* = 15.7 Hz, 1H), 6.07 (dt, *J* = 15.7, 6.9 Hz, 1H), 5.42 (d, *J* = 6.8 Hz, 1H), 3.85 – 3.72 (m, 1H), 2.63 – 2.51 (m, 2H), 2.32 (s, 5H), 1.95 – 1.85 (m, 2H), 1.73 – 1.59 (m, 3H), 1.43 – 1.33 (m, 2H), 1.19 – 0.95 (m, 3H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 171.2, 136.5, 135.0, 130.2, 130.2, 128.8, 127.1, 126.0, 125.5, 48.2, 36.8, 33.3, 29.5, 25.5, 24.9, 19.8. **HRMS** (ESI-TOF): *m*/*z* calcd. for $C_{18}H_{26}NO^+$ ([M+H]$^+$) 272.2009, found 272.2015.

(*E*)-*N*-Cyclohexyl-5-(3-fluorophenyl)pent-4-enamide (7d)



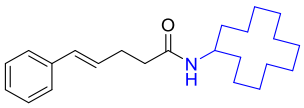
Pentane/ethyl acetate = 2/1, white solid (33.5 mg, 61% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.25 – 7.19 (m, 1H), 7.08 – 6.98 (m, 2H), 6.88 (tdd, *J* = 8.5, 2.6, 1.0 Hz, 1H), 6.39 (d, *J* = 16.1 Hz, 1H), 6.29 – 6.09 (m, 1H), 5.46 (d, *J* = 6.8 Hz, 1H), 3.84 – 3.70 (m, 1H), 2.58 – 2.50 (m, 2H), 2.30 (t, *J* = 7.5 Hz, 2H), 1.89 (dd, *J* = 12.4, 3.5 Hz, 2H), 1.71 – 1.56 (m, 3H), 1.42 – 1.32 (m, 2H), 1.17 – 1.00 (m, 3H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 171.0, 163.1 (d, *J* = 245.0 Hz), 139.8 (d, *J* = 7.9 Hz), 130.4, 130.0 (d, *J* = 1.9 Hz), 129.9 (d, *J* = 3.9 Hz), 121.9 (d, *J* = 2.7 Hz), 113.9 (d, *J* = 21.4 Hz), 112.4 (d, *J* = 21.7 Hz), 48.2, 36.4, 33.2, 29.0, 25.5, 24.9. **^{19}F NMR (282 MHz, CDCl$_3$)** δ -113.76. **HRMS** (ESI-TOF): *m*/*z* calcd. for $C_{17}H_{23}FNO^+$ ([M+H]$^+$) 276.1758, found 276.1760.

(*E*)-*N*-Cyclohexyl-5-(3,5-dimethoxyphenyl)pent-4-enamide (8d)



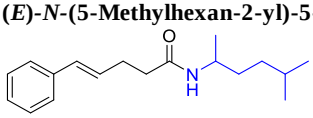
Pentane/ethyl acetate = 1/1, white solid (34.9 mg, 55% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 6.56 – 6.43 (m, 2H), 6.42 – 6.27 (m, 2H), 6.18 (dt, *J* = 15.8, 6.8 Hz, 1H), 5.48 (d, *J* = 7.7 Hz, 1H), 3.77 (s, 6H), 3.79 – 3.73 (m, 1H), 2.59 – 2.42 (m, 2H), 2.40 – 2.06 (m, 2H), 1.97 – 1.79 (m, 2H), 1.71 – 1.55 (m, 3H), 1.38 – 1.28 (m, 2H), 1.20 – 0.99 (m, 3H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 171.2, 160.9, 139.5, 131.0, 129.5, 104.1, 99.5, 55.3, 48.2, 36.5, 33.2, 29.0, 25.5, 24.9. **HRMS** (ESI-TOF): *m*/*z* calcd. for $C_{19}H_{28}NO_3^+$ ([M+H]$^+$) 318.2064, found 318.2068.

(*E*)-*N*-Cyclododecyl-5-phenylpent-4-enamide (9d)



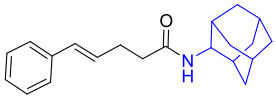
Pentane/ethyl acetate = 4/1, white solid (46.4 mg, 68% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.27 – 7.18 (m, 4H), 7.16 – 7.04 (m, 1H), 6.36 (d, *J* = 15.8 Hz, 1H), 6.13 (dt, *J* = 15.8, 6.8 Hz, 1H), 5.26 (d, *J* = 8.3 Hz, 1H), 4.04 – 3.96 (m, 1H), 2.52 – 2.42 (m, 2H), 2.23 (t, *J* = 7.4 Hz, 2H), 1.38 – 1.18 (m, 22H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 171.3, 137.4, 131.0, 128.9, 128.5, 127.1, 126.0, 46.1, 36.6, 30.3, 29.1, 24.0, 23.8, 23.5, 23.3, 21.4. **HRMS** (ESI-TOF): *m*/*z* calcd. for $C_{23}H_{36}NO^+$ ([M+H]$^+$) 342.2791, found 342.2800.

(*E*)-*N*-(5-Methylhexan-2-yl)-5-phenylpent-4-enamide (10d)



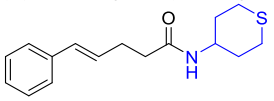
Pentane/ethyl acetate = 3/1, white solid (43.7 mg, 69% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.14 – 7.08 (m, 4H), 7.04 – 7.01 (m, 1H), 6.26 (d, *J* = 15.8 Hz, 1H), 6.04 (dt, *J* = 15.8, 6.8 Hz, 1H), 5.28 (s, 1H), 3.84 – 3.75 (m, 1H), 2.43 – 2.33 (m, 2H), 2.19 – 2.09 (m, 2H), 1.37 – 1.16 (m, 4H), 1.02 – 0.99 (m, 1H), 0.93 (d, *J* = 6.6 Hz, 3H), 0.67 (d, *J* = 6.6 Hz, 6H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 171.4, 137.4, 131.0, 128.9, 128.5, 127.1, 126.0, 45.4, 36.6, 35.1, 34.8, 29.2, 28.0, 22.6, 21.1. **HRMS** (ESI-TOF): *m*/*z* calcd. for $C_{18}H_{28}NO^+$ ([M+H]$^+$) 274.2165, found 274.2167.

(*E*)-*N*-((1*R*,2*S*,5*S*)-Adamantan-2-yl)-5-phenylpent-4-enamide (11d)



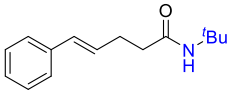
Pentane/ethyl acetate = 2/1, colorless oil (50.6 mg, 82% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.12 – 7.04 (m, 4H), 7.01 – 6.97 (m, 1H), 6.24 (d, *J* = 15.8 Hz, 1H), 6.01 (dt, *J* = 15.8, 6.8 Hz, 1H), 5.73 (d, *J* = 7.5 Hz, 1H), 3.86 (d, *J* = 8.0 Hz, 1H), 2.35 (q, *J* = 7.1 Hz, 2H), 2.20 – 2.14 (m, 2H), 1.68 – 1.38 (m, 14H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 171.3, 137.3, 131.1, 128.9, 128.5, 127.2, 126.0, 53.2, 37.5, 37.1, 36.6, 31.9, 31.9, 29.3, 27.2, 27.1, 26.9. **HRMS** (ESI-TOF): *m*/*z* calcd. for $C_{21}H_{28}NO^+$ ([M+H]$^+$) 310.2165, found 310.2169.

(*E*)-5-Phenyl-*N*-(tetrahydro-2*H*-thiopyran-4-yl)pent-4-enamide (12d)



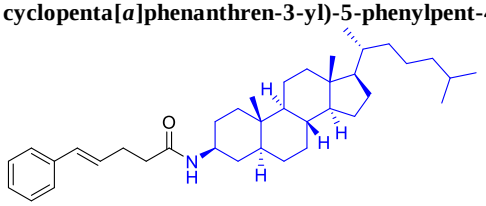
Pentane/ethyl acetate = 3/2, white solid (22.6 mg, 41% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.24 – 7.14 (m, 5H), 6.36 (d, *J* = 15.8 Hz, 1H), 6.12 (dt, *J* = 15.8, 6.8 Hz, 1H), 5.46 (d, *J* = 8.0 Hz, 1H), 3.80 – 3.68 (m, 1H), 2.67 – 2.43 (m, 6H), 2.28 – 2.21 (m, 2H), 2.11 – 2.03 (m, 2H), 1.52 – 1.39 (m, 2H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 171.2, 137.3, 131.1, 128.6, 128.6, 127.2, 126.0, 47.4, 36.5, 34.0, 29.1, 27.9. **HRMS** (ESI-TOF): *m*/*z* calcd. for $C_{26}H_{22}NOS^+$ ([M+H]$^+$) 276.1417, found 276.1416.

(*E*)-*N*-(*tert*-Butyl)-5-phenylpent-4-enamide (13d)



Pentane/ethyl acetate = 4/1, white solid (31.4 mg, 68% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.27 – 7.18 (m, 4H), 7.17 – 7.03 (m, 1H), 6.35 (d, *J* = 15.8 Hz, 1H), 6.12 (dt, *J* = 15.8, 6.9 Hz, 1H), 5.29 (s, 1H), 2.50 – 2.39 (m, 2H), 2.17 (t, *J* = 7.4 Hz, 2H), 1.26 (s, 9H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 171.4, 137.4, 130.9, 128.9, 128.5, 127.0, 125.9, 51.1, 37.2, 29.0, 28.8. **HRMS** (ESI-TOF): *m*/*z* calcd. for $C_{15}H_{22}NO^+$ ([M+H]$^+$) 232.1696, found 232.1705.

(*E*)-*N*-((3*S*,5*S*,8*R*,9*S*,10*S*,13*R*,14*S*,17*R*)-10,13-dimethyl-17-((*R*)-6-methylheptan-2-yl)hexadecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl)-5-phenylpent-4-enamide (14d)



Pentane/ethyl acetate = 2/1, white solid (38.2 mg, 35% yield). **^{1}H NMR (300 MHz, CDCl$_3$)** δ 7.26 – 7.18 (m, 4H), 7.15 – 7.08 (m, 1H), 6.35 (d, *J* = 15.8 Hz, 1H), 6.13 (dt, *J* = 15.8, 6.8 Hz, 1H), 5.37 (d, *J* = 8.1 Hz, 1H), 3.94 – 3.48 (m, 1H), 2.57 – 2.41 (m, 2H), 2.23 (t, *J* = 7.3 Hz, 2H), 1.88 (dt, *J* = 12.2, 3.1 Hz, 1H), 1.72 (dd, *J* = 9.2, 3.5 Hz, 2H), 1.66 – 1.35 (m, 7H), 1.27 – 0.91 (m, 21H), 0.82 (d, *J* = 6.5 Hz, 3H), 0.80 (d, *J* = 1.4 Hz, 3H), 0.78 (d, *J* = 1.3 Hz, 3H), 0.68 (s, 3H), 0.57 (s, 3H). **^{13}C NMR (75 MHz, CDCl$_3$)** δ 171.3, 137.4, 131.0, 128.8, 128.5, 127.1, 126.0, 56.4, 56.3, 54.2, 49.0, 45.3, 42.6, 40.0, 39.5, 37.4, 36.6, 36.2, 35.8, 35.5, 35.4, 32.0, 29.2, 28.9, 28.6, 28.3, 28.0, 24.2, 23.9, 22.8, 22.6, 21.1, 18.7, 12.2, 12.1. **HRMS** (ESI-TOF): *m*/*z* calcd. for $C_{38}H_{60}NO^+$ ([M+H]$^+$) 546.4669, found 546.4681.

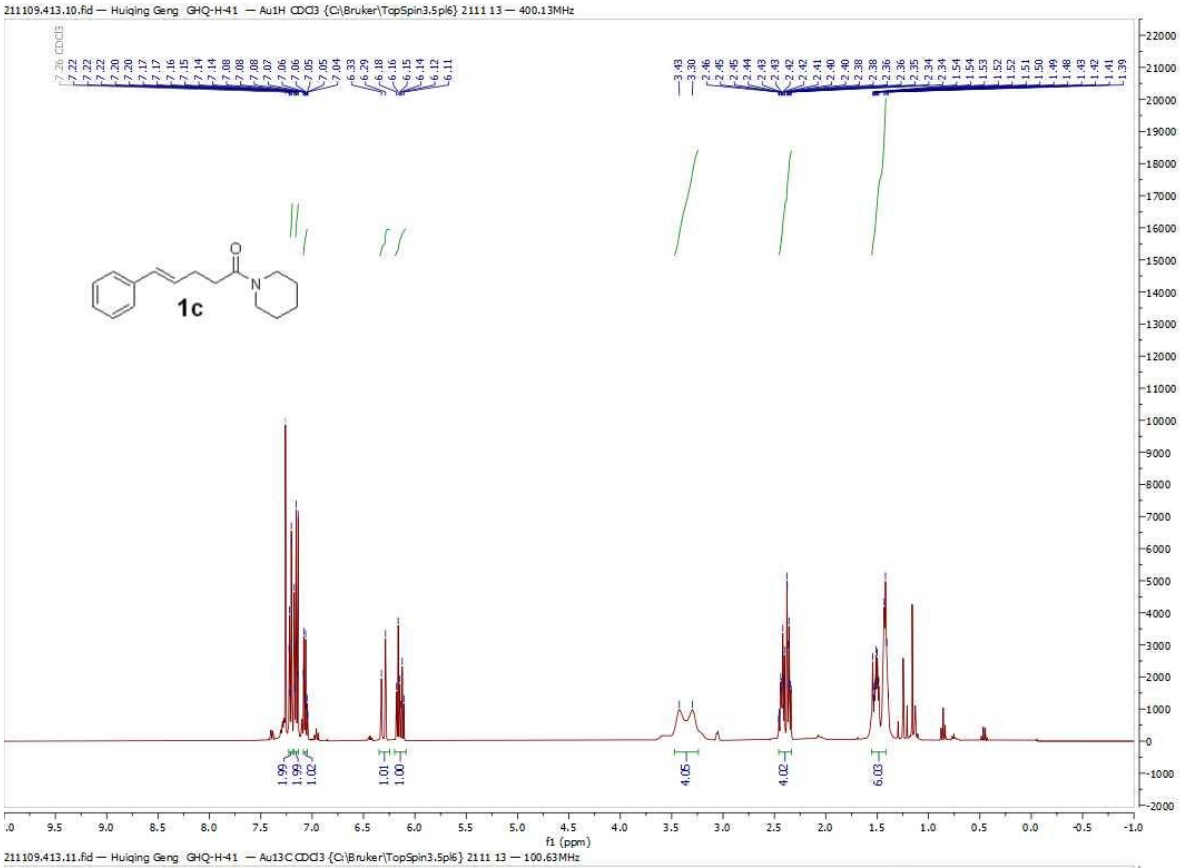
5. References

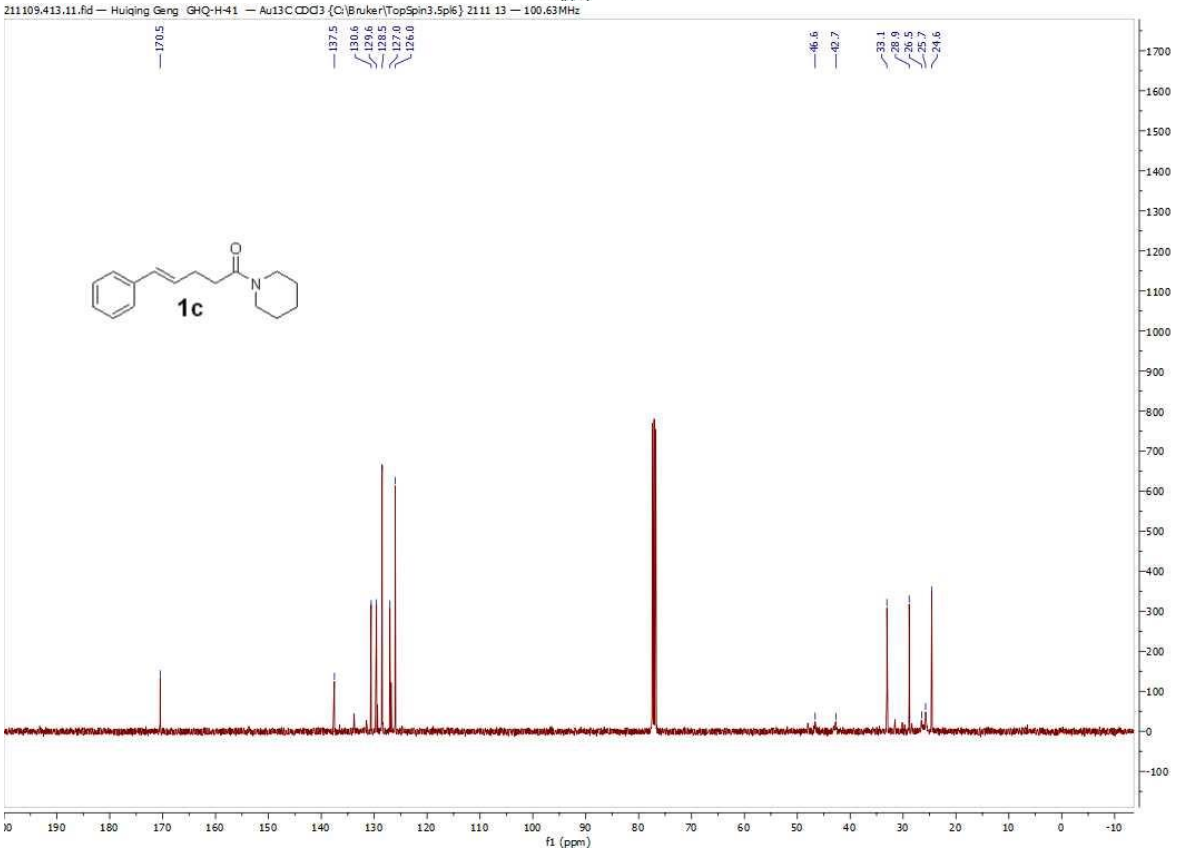
[1] Medina, J. M.; Kang, T.; Erbay, T. G.; Shao, H.; Gallego, G. M.; Yang, S.; Tran-Dube M.; Richardson P. F.; Derosa J.; Helsel, R. T.; Patman R. L.; Wang F.; Ashcroft C, P.; Braganza J. F.; McAlpine I.; Liu P.; Engle K. M. Cu-Catalyzed Hydroboration of Benzylidenecyclopropanes: Reaction Optimization, (Hetero) Aryl Scope, and Origins of Pathway Selectivity. *ACS Catal.* **2019**, *9*, 11130-11136.

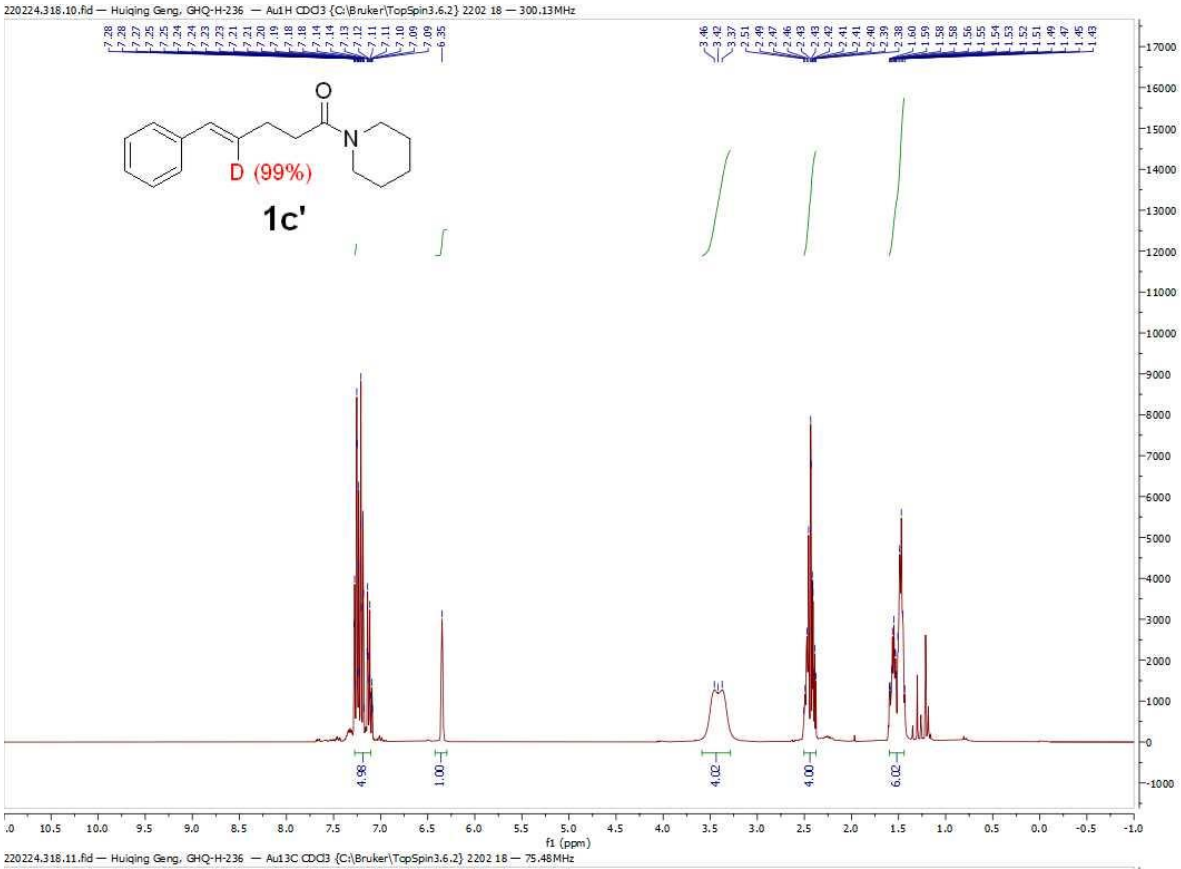
[2] Yuan, Y.; Wu, F.-P.; Schünemann, C.; Holz, J.; Kamer, P. C.; Wu, X.-F. Copper-Catalyzed Carbonylative Hydroamidation of Styrenes to Branched Amides. *Angew. Chem. Int. Ed*. **2020**, *59*, 22441-22445.

[3] Yuan, Y.; Zhao, F.; Wu, X.-F. Copper-Catalyzed Enantioselective Carbonylation toward α-Chiral Secondary Amides. *Chem. Sci.*, **2021**, *12*, 12676-12681.

6. NMR spectra



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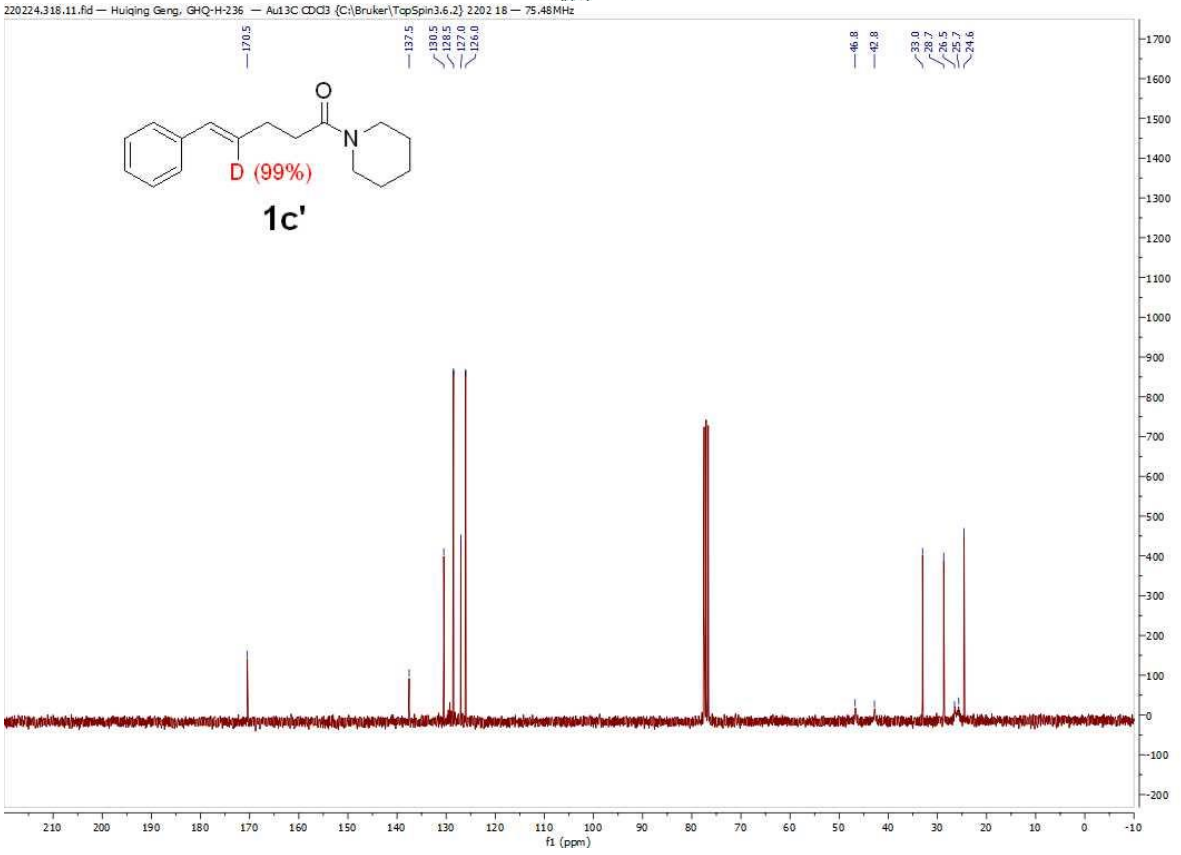
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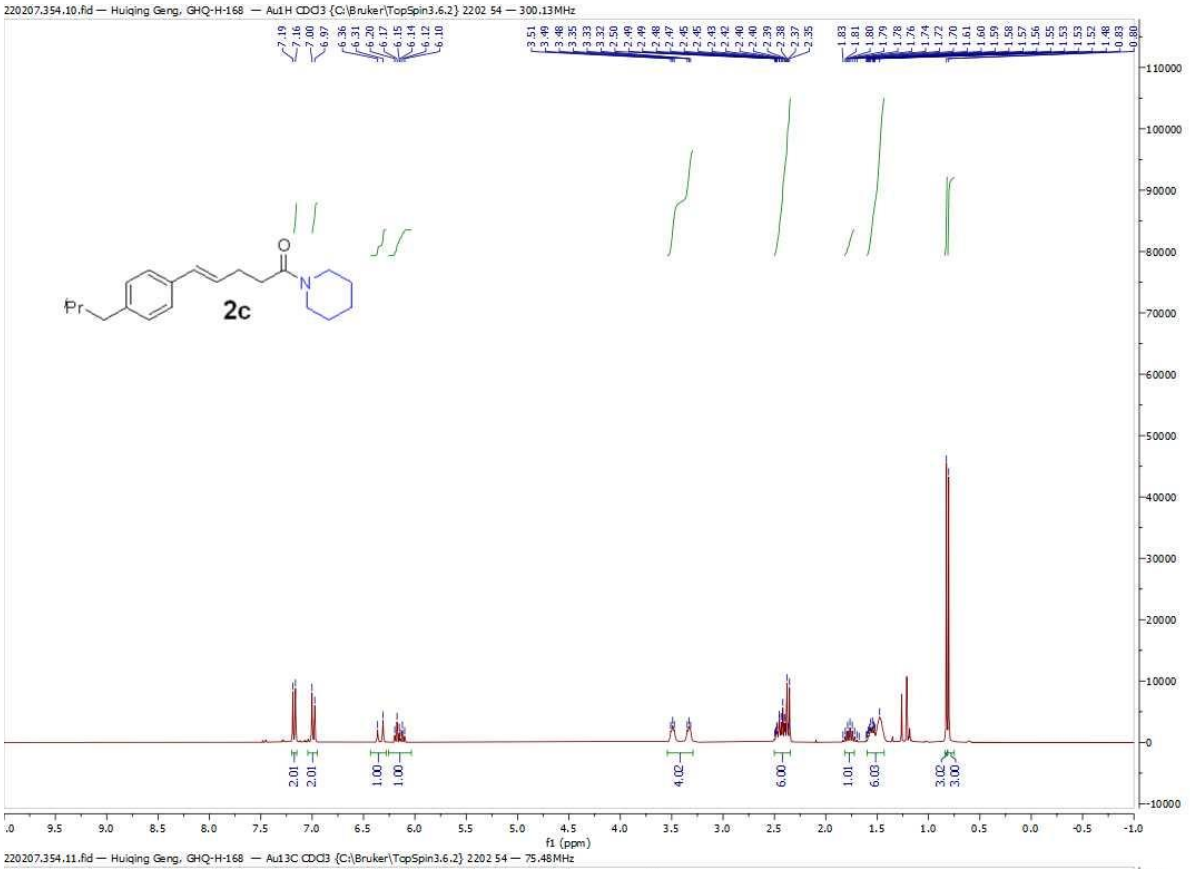
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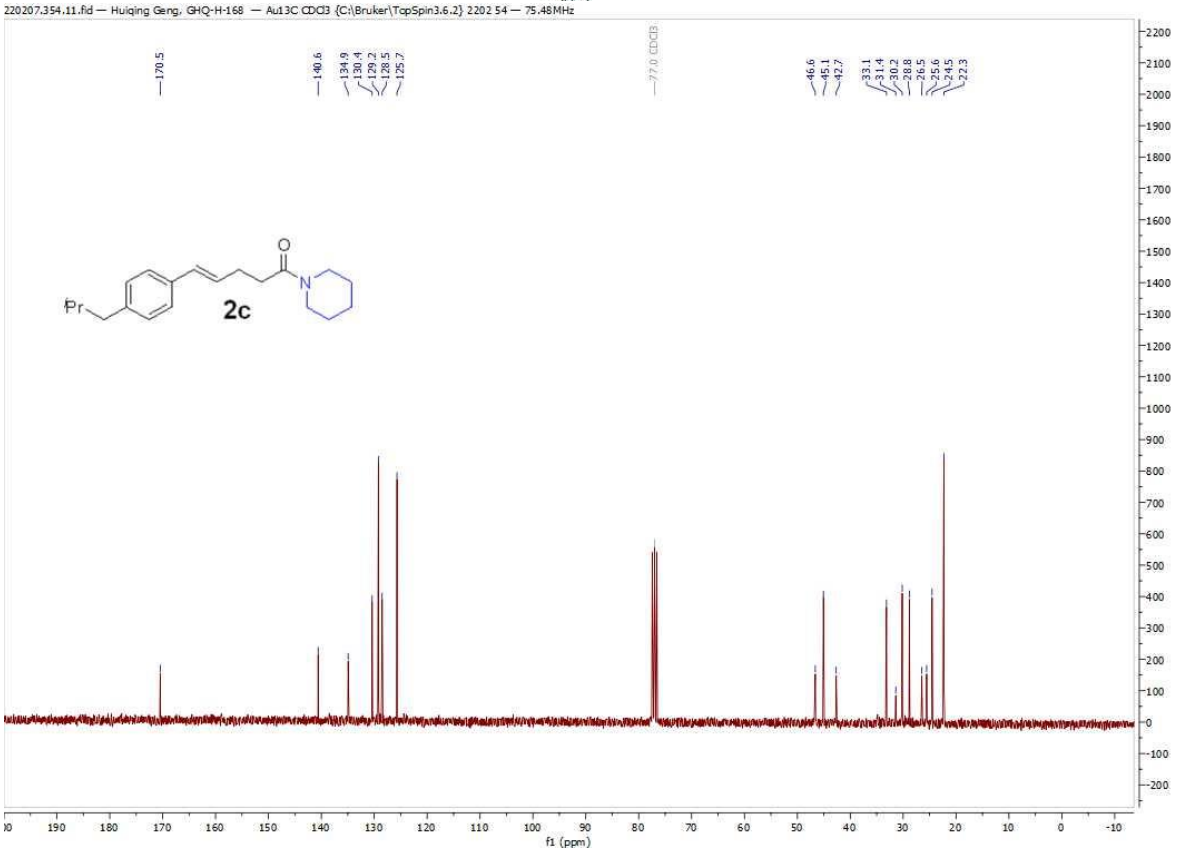
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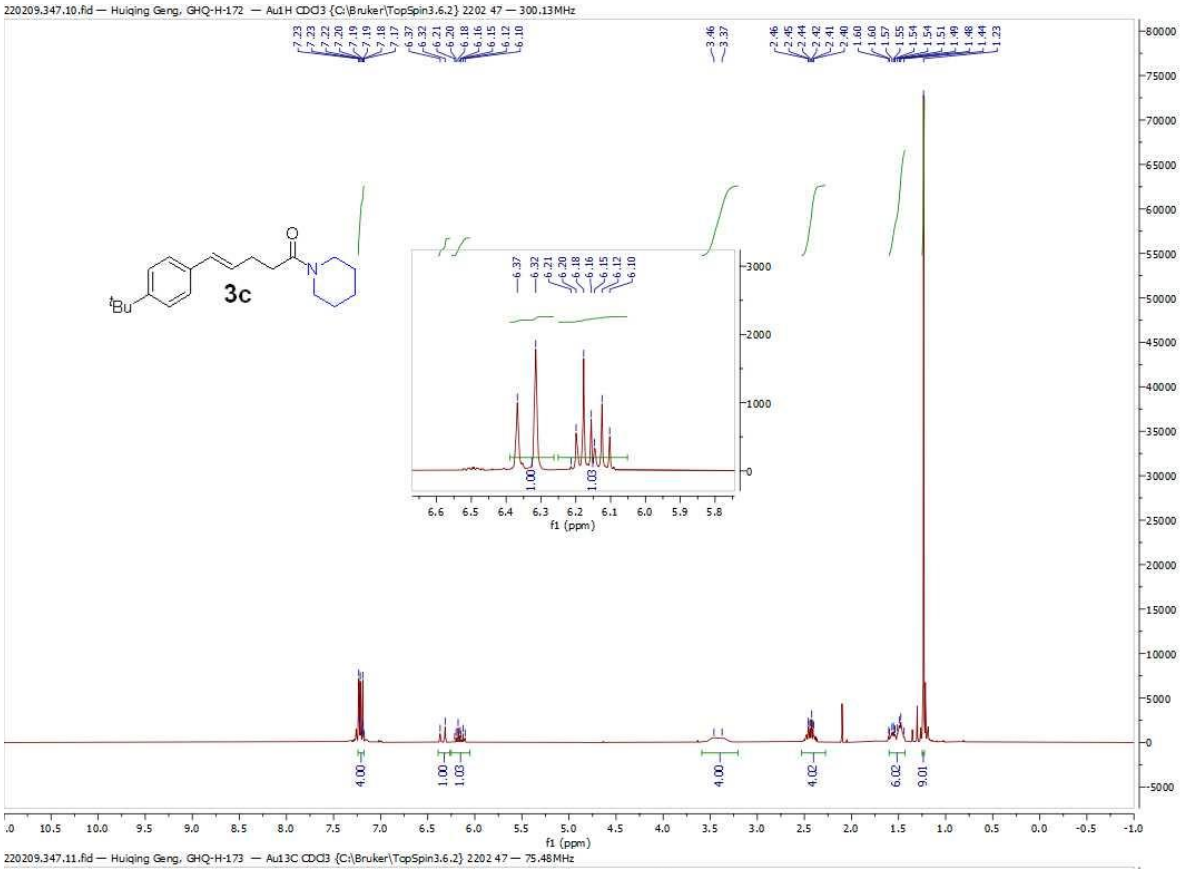
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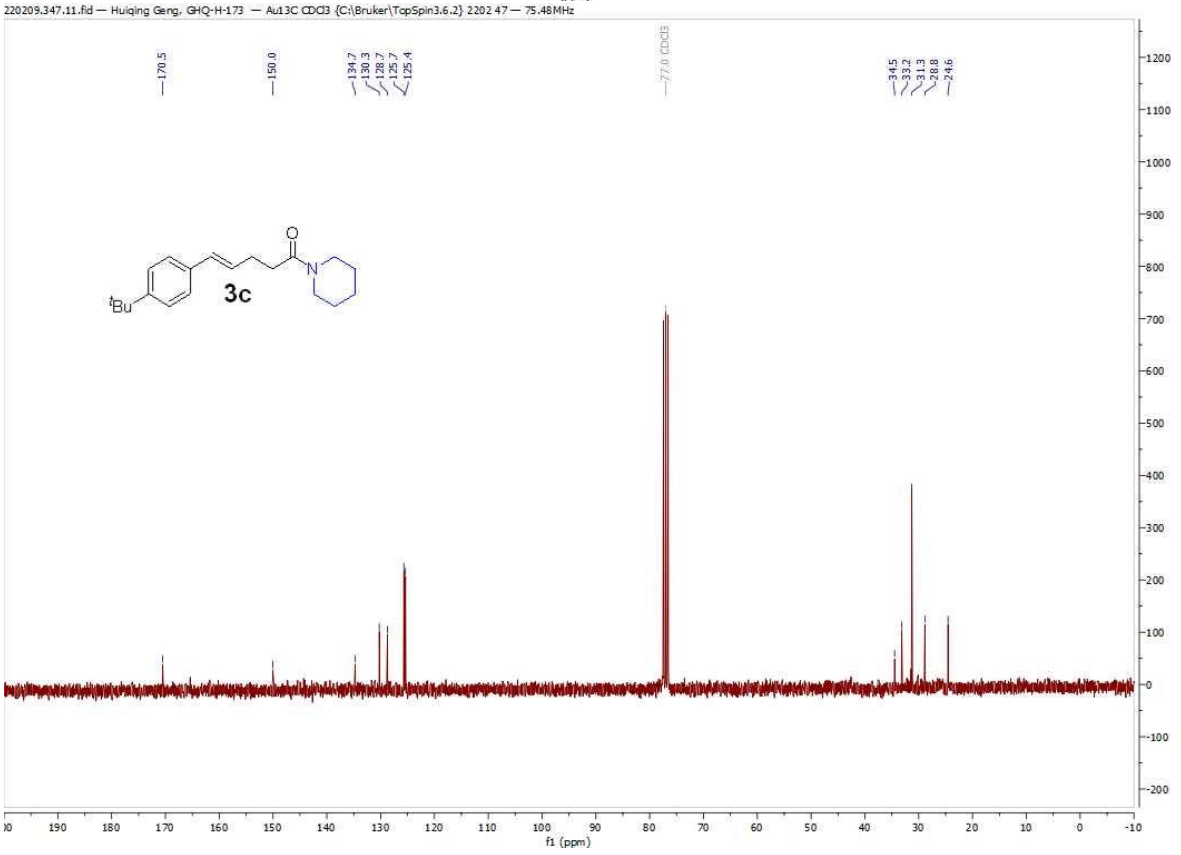
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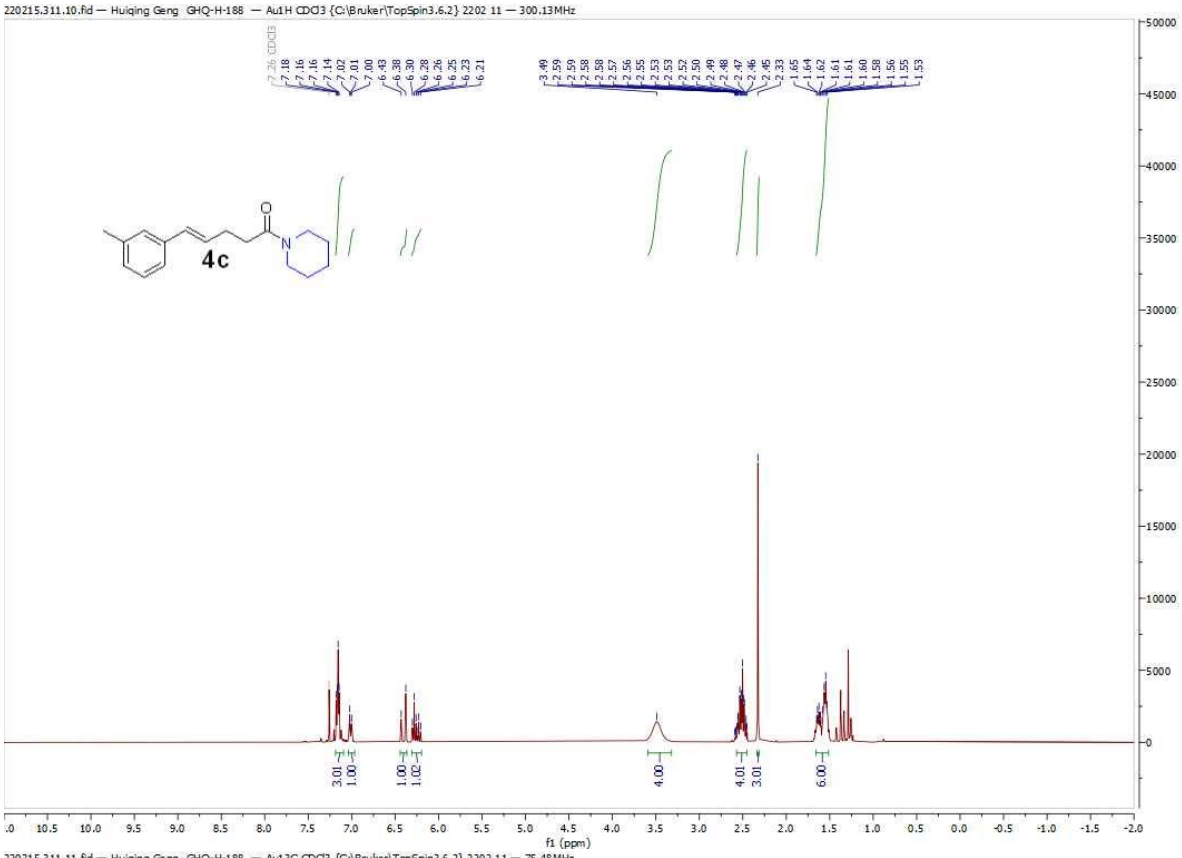
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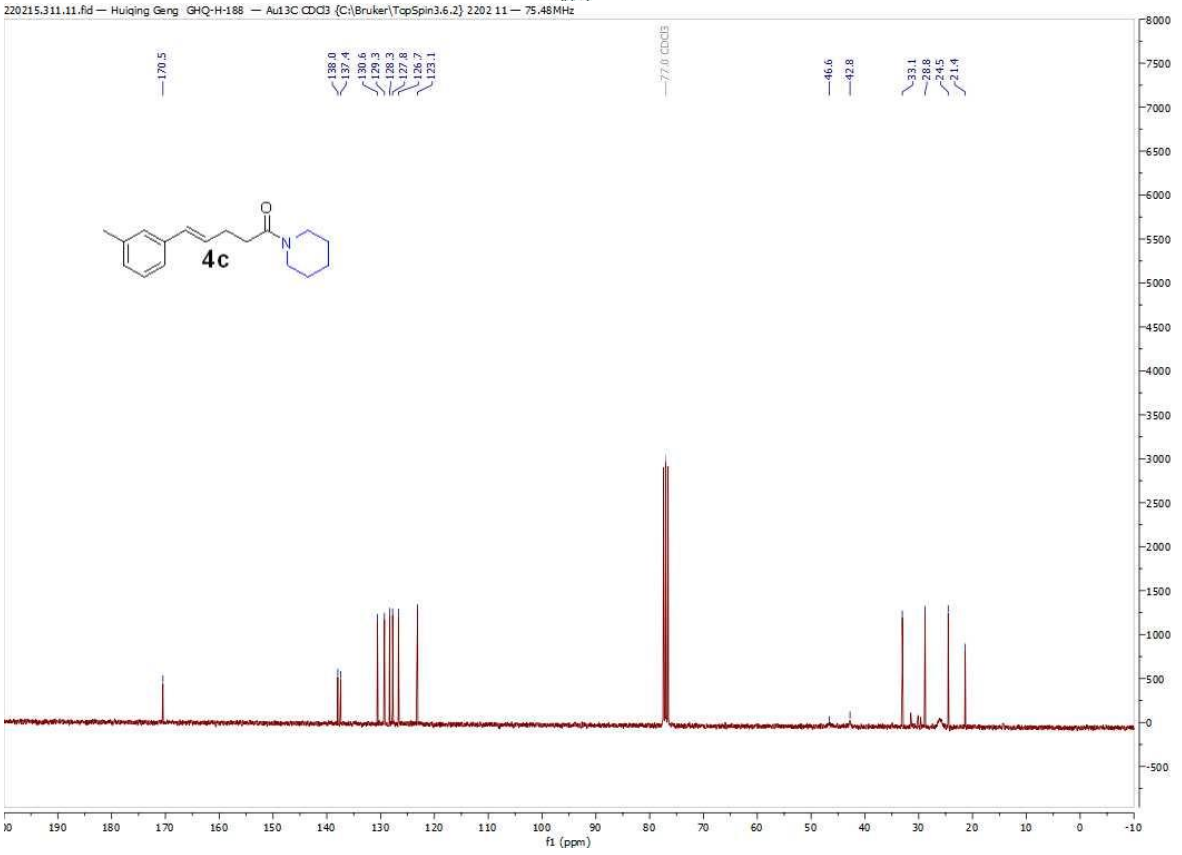
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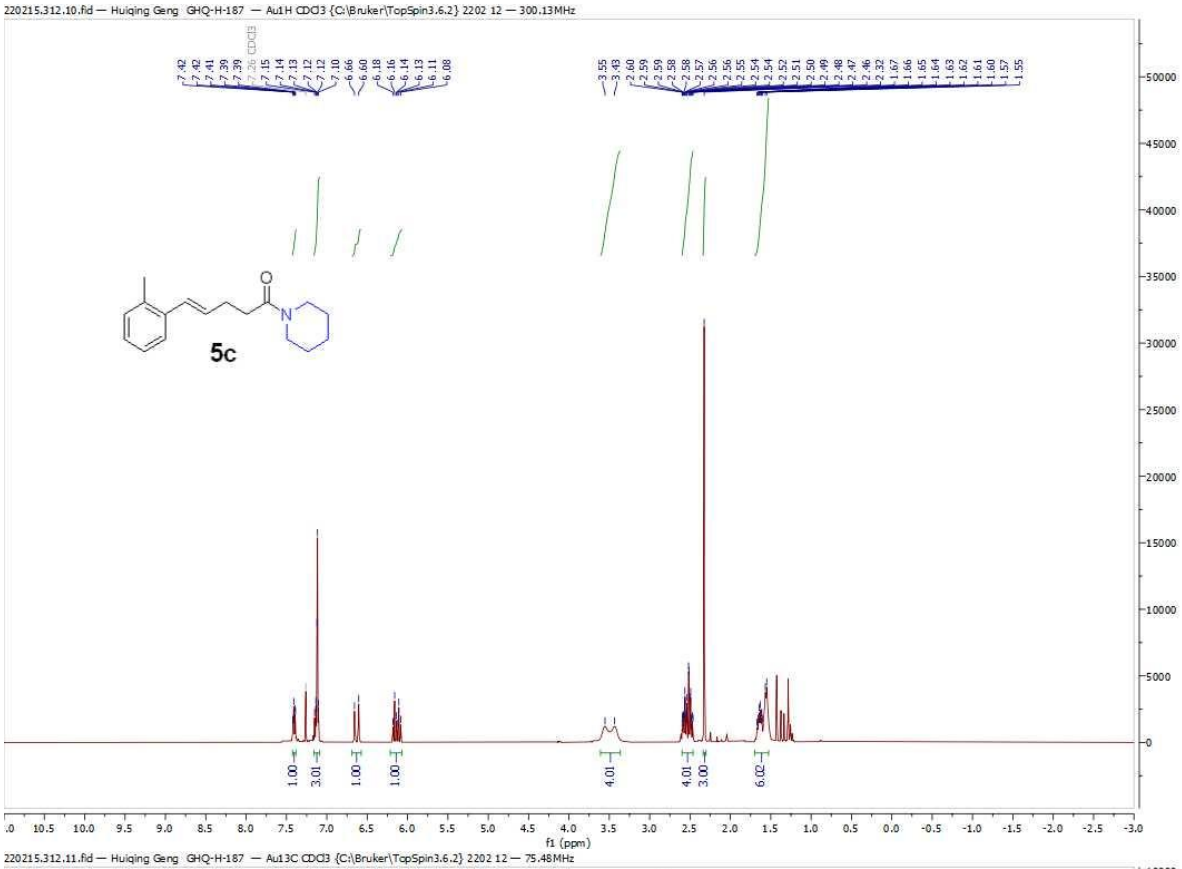
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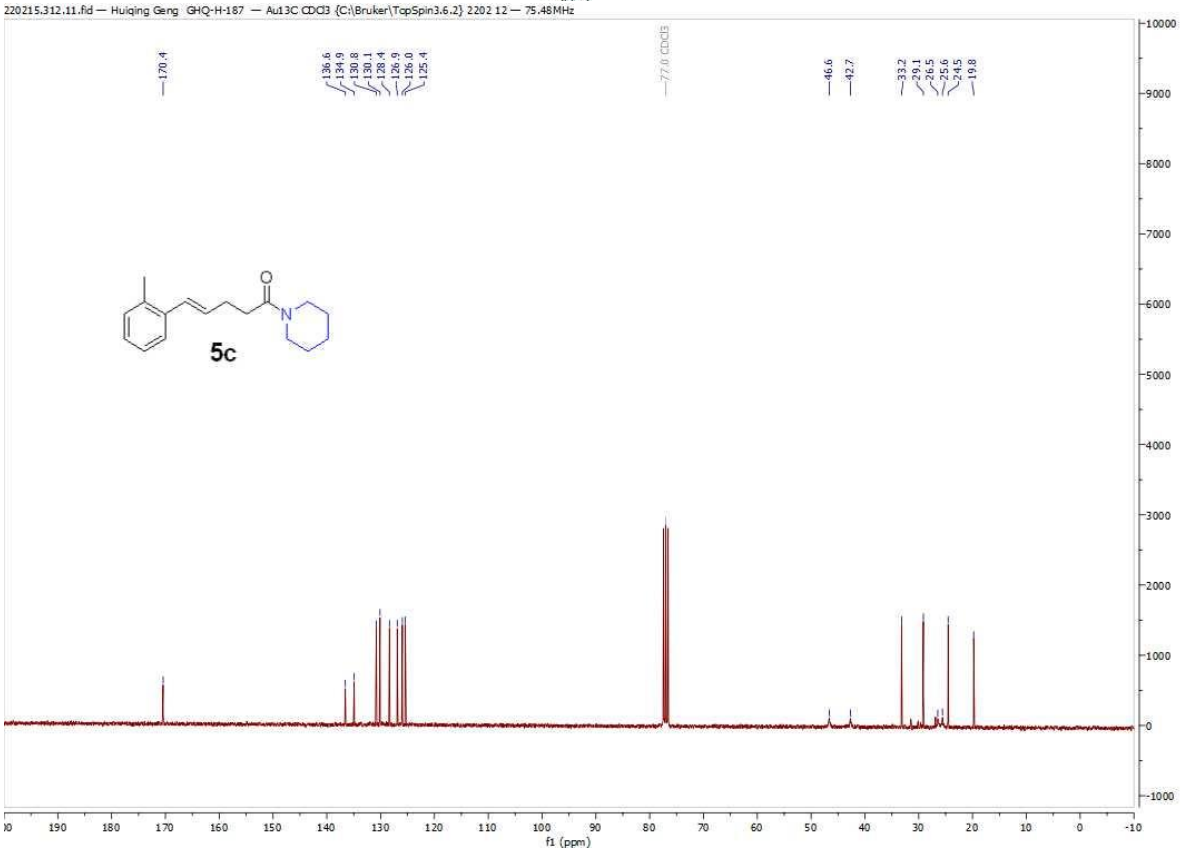
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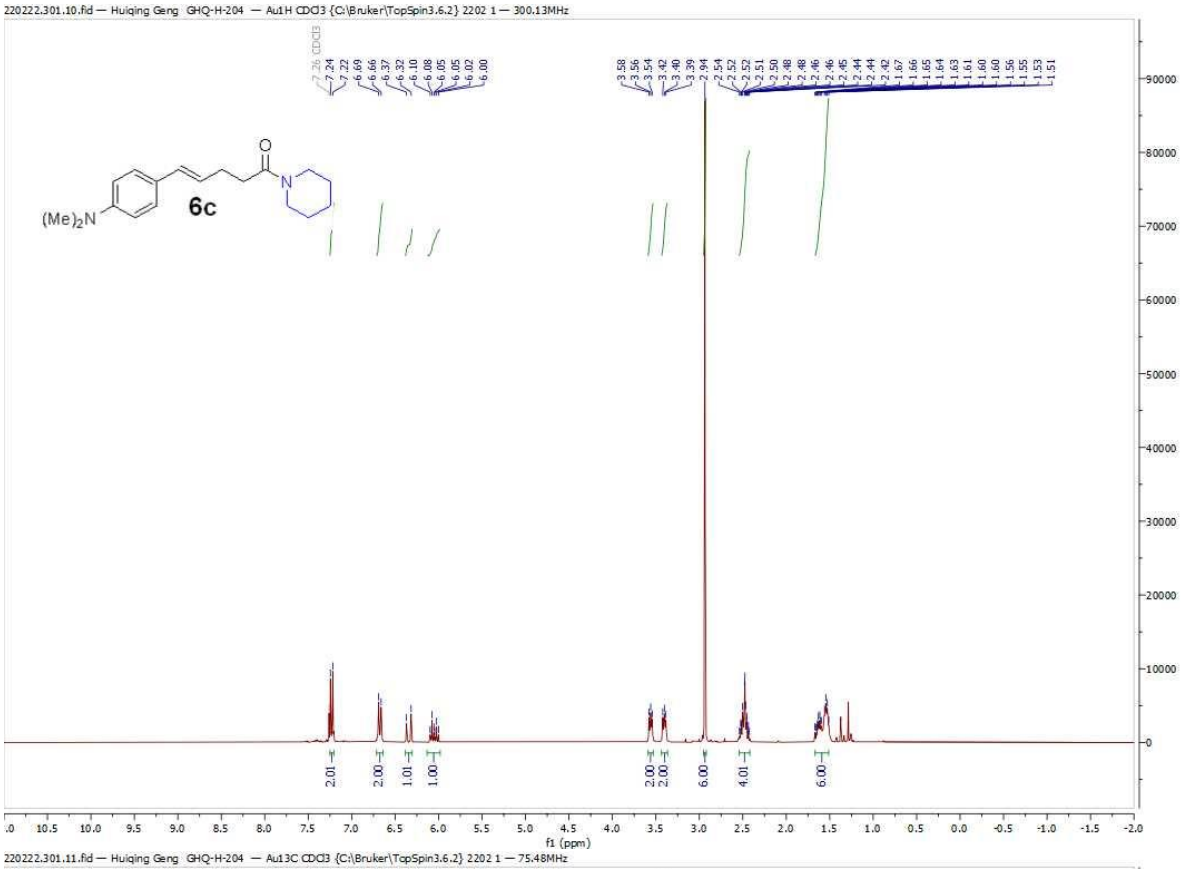
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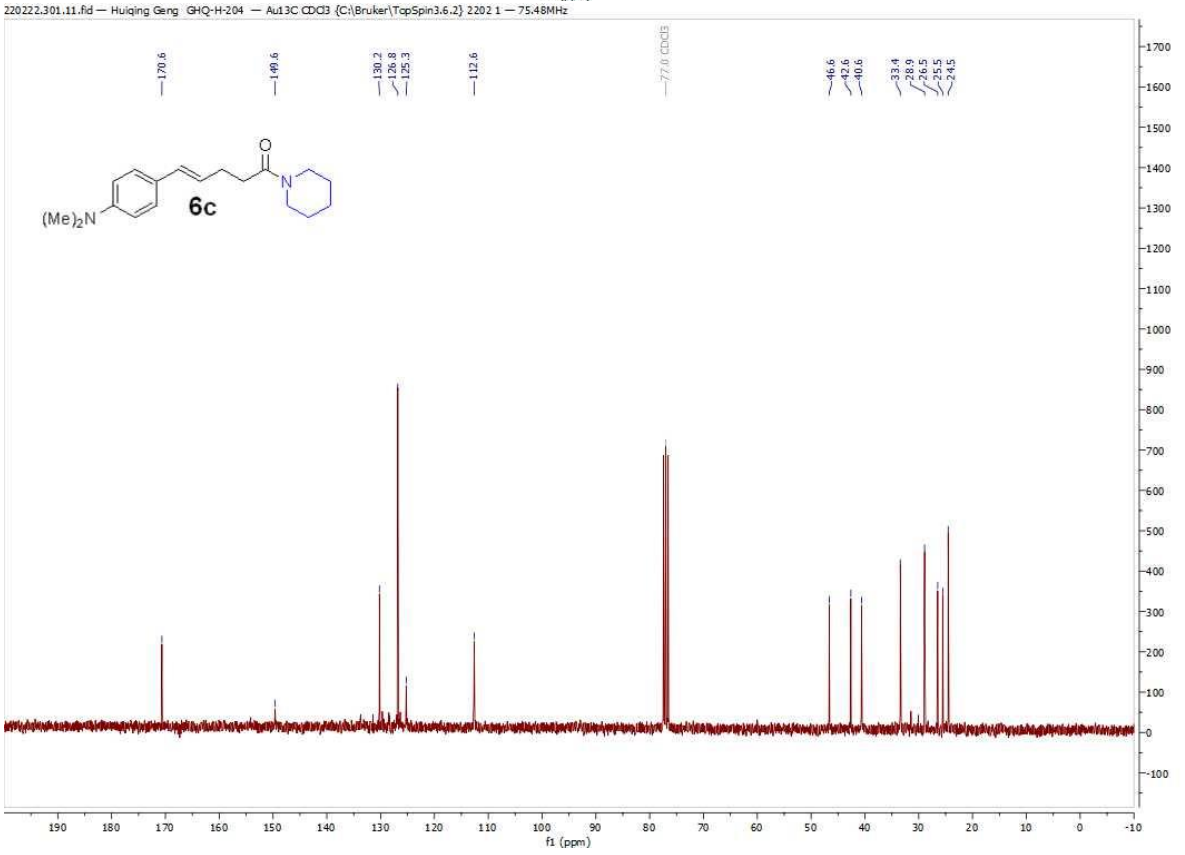
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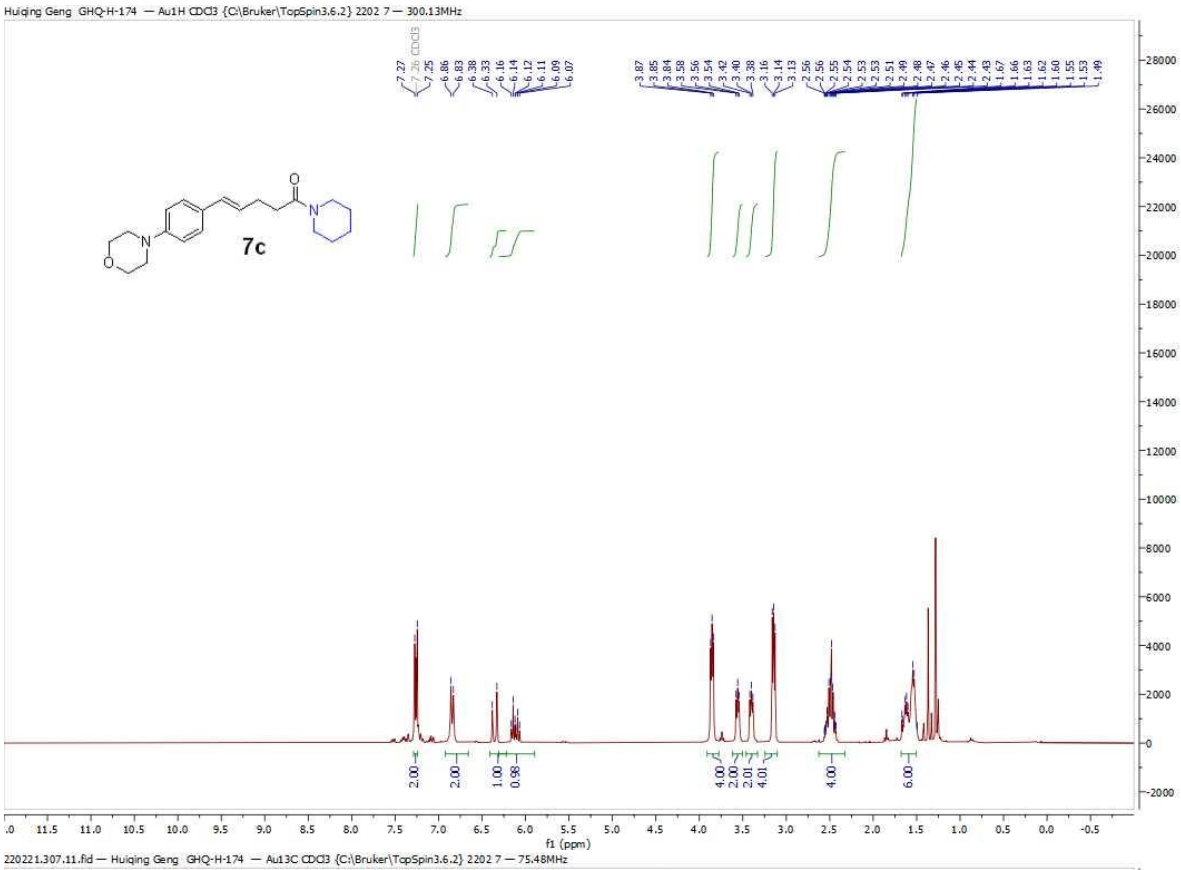
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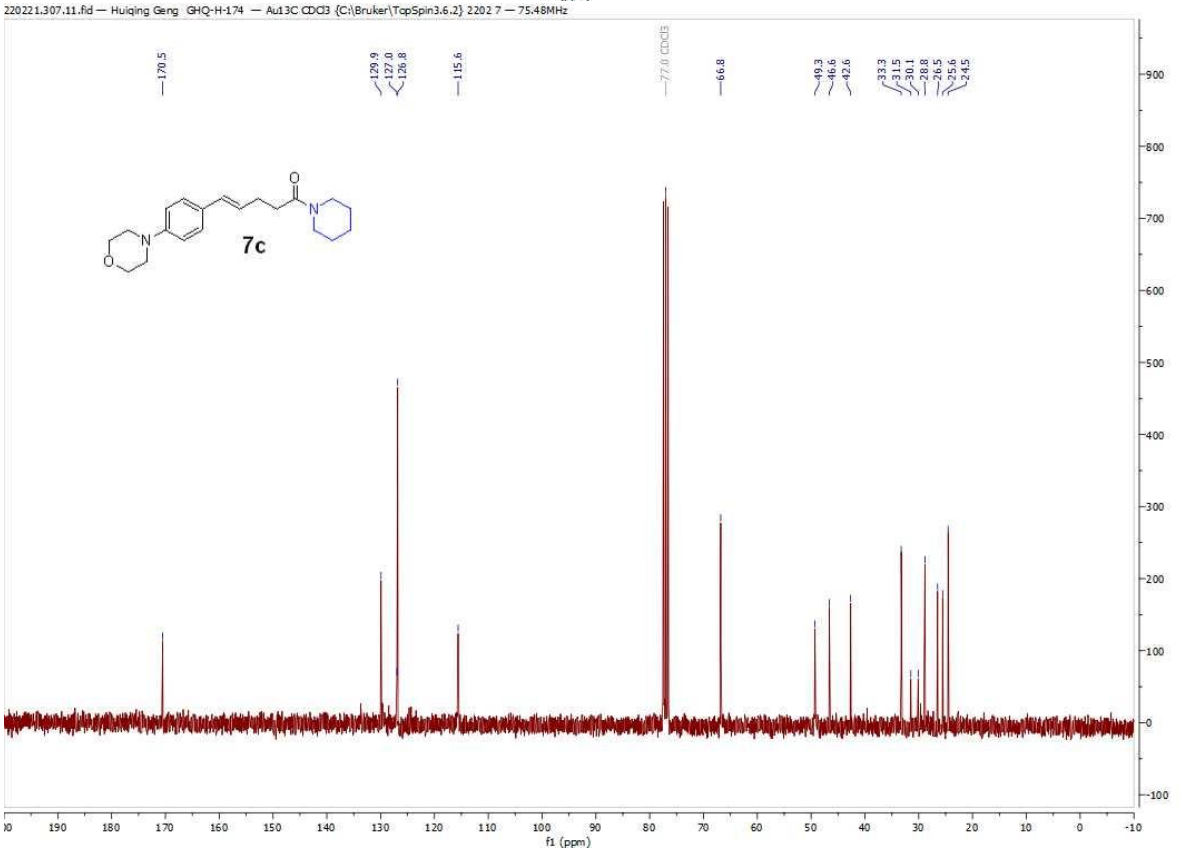
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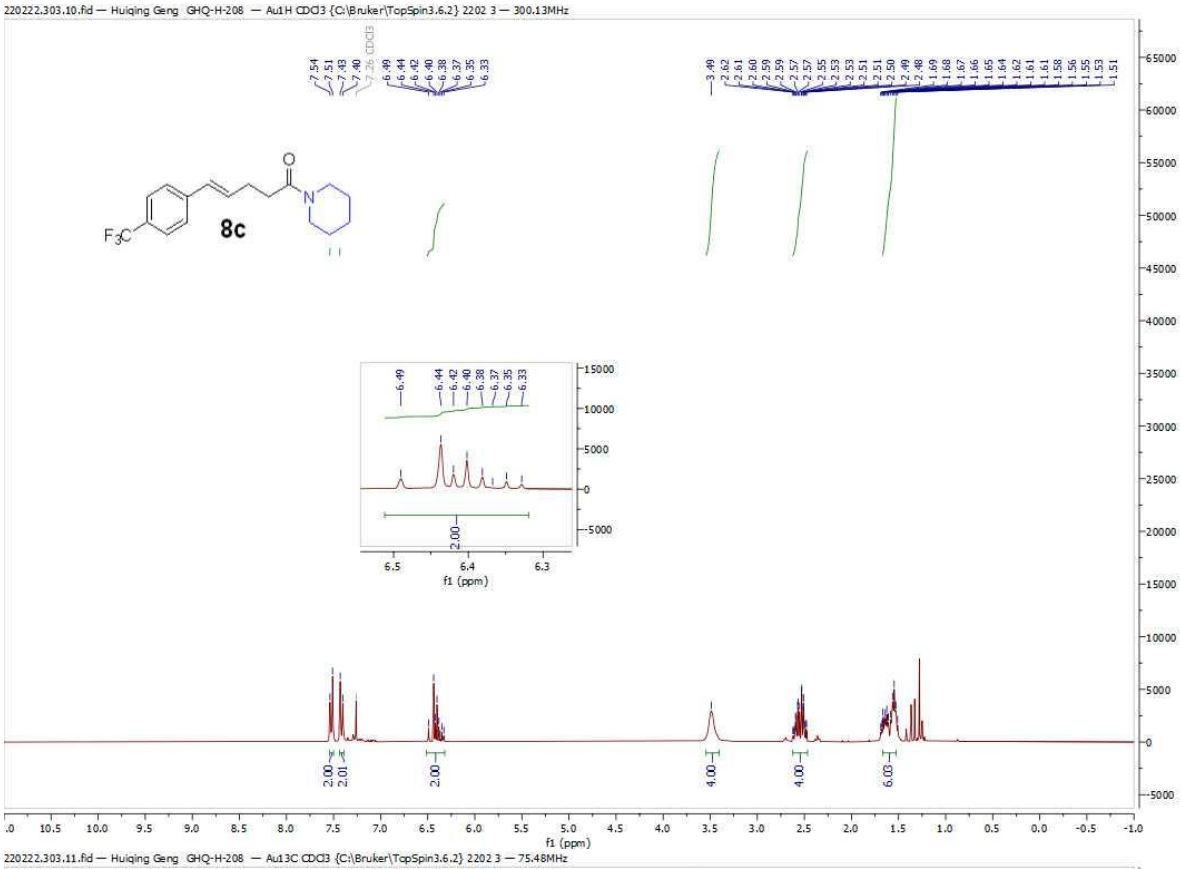
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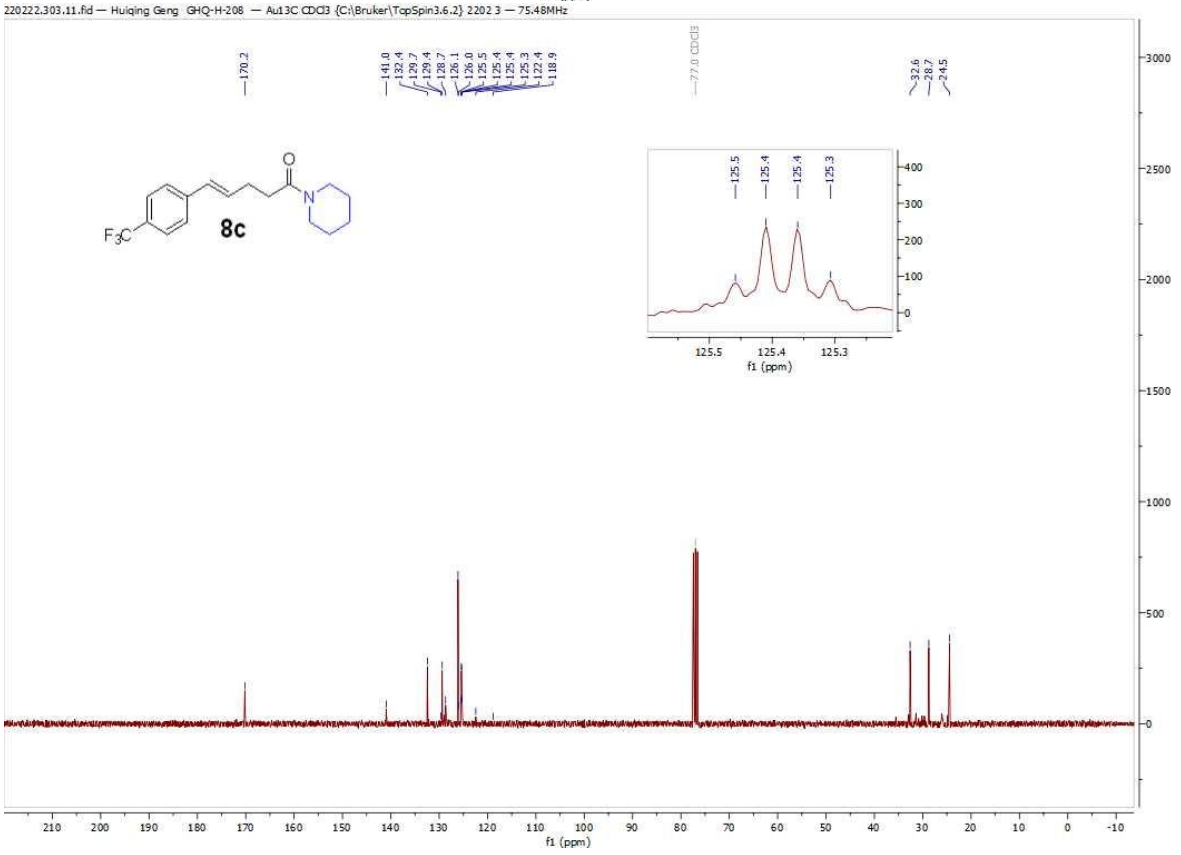
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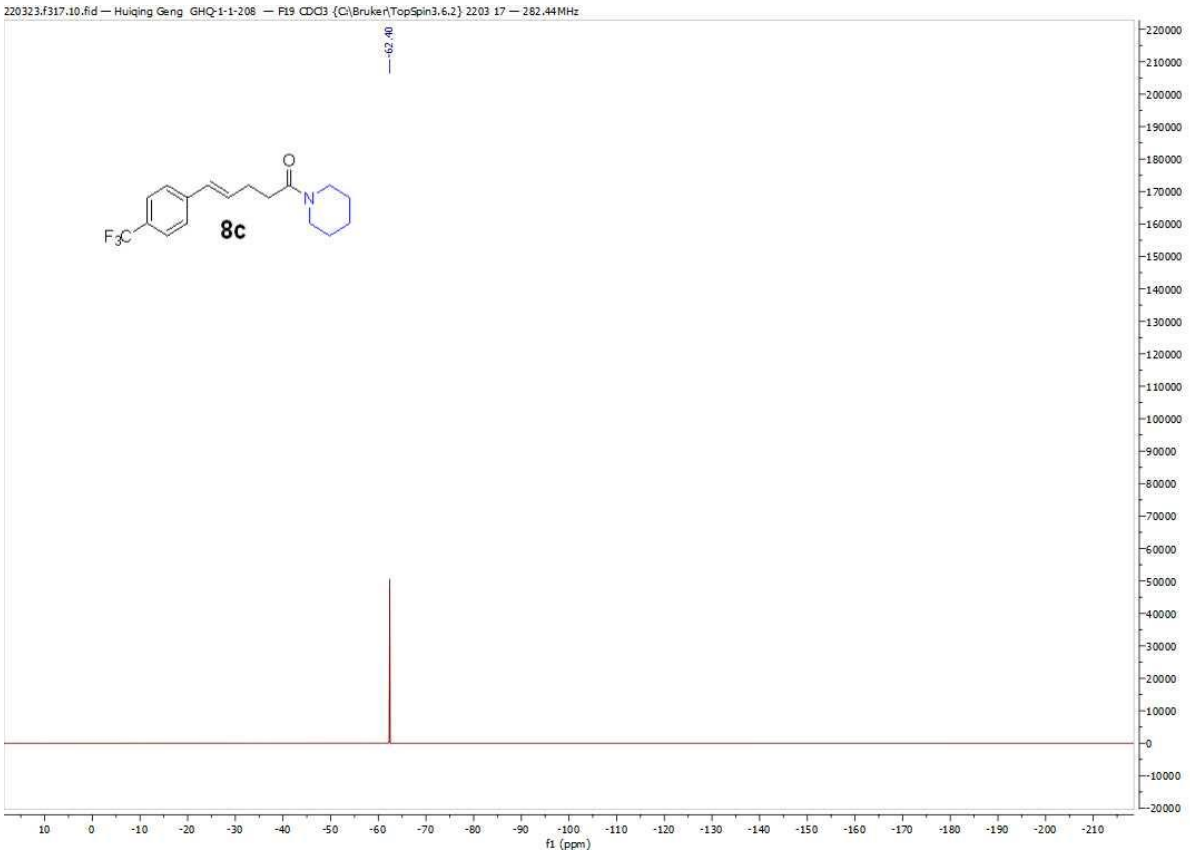
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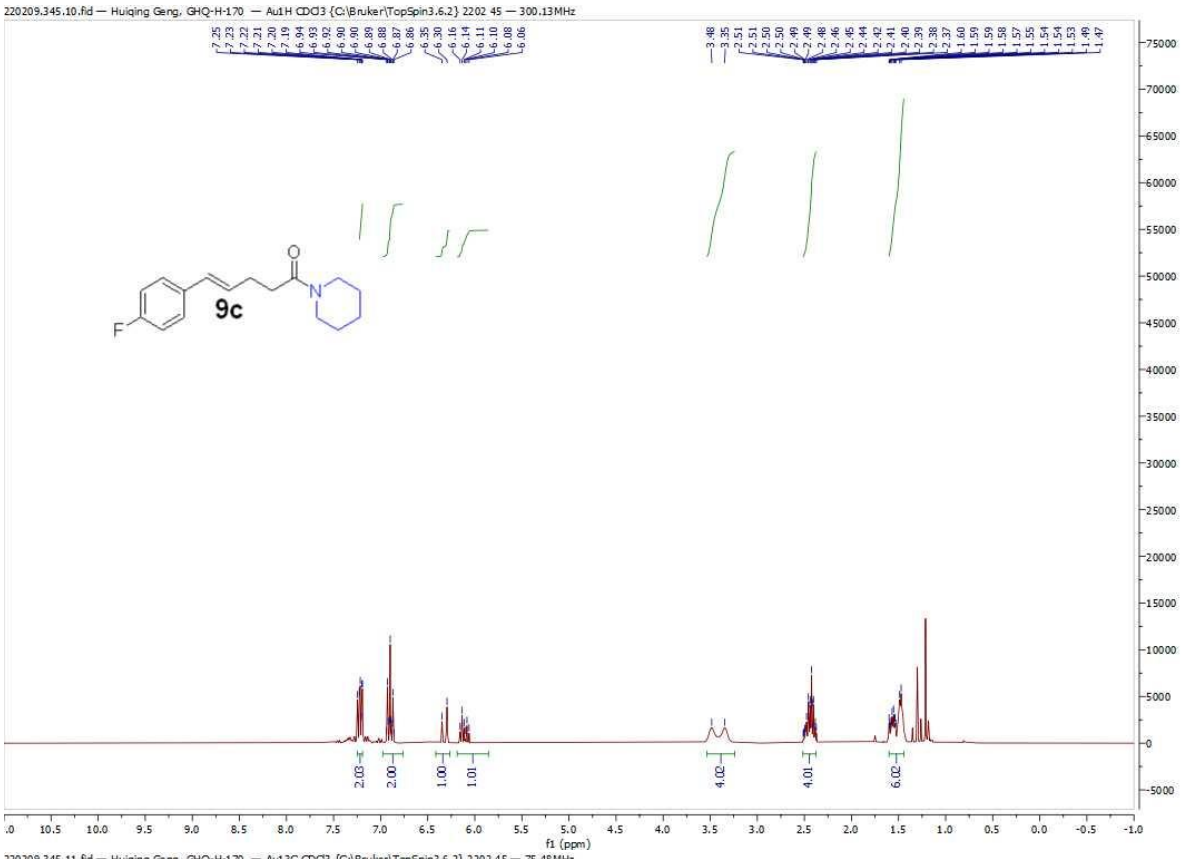
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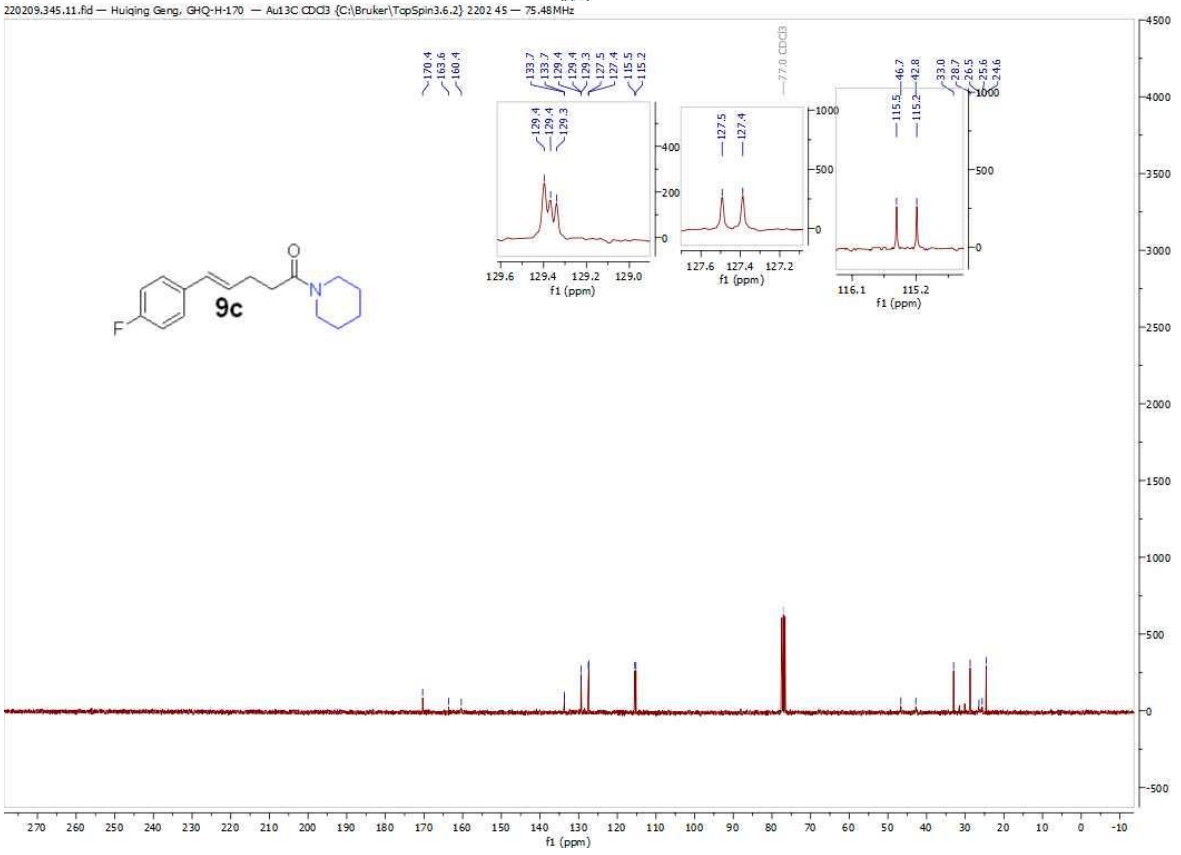
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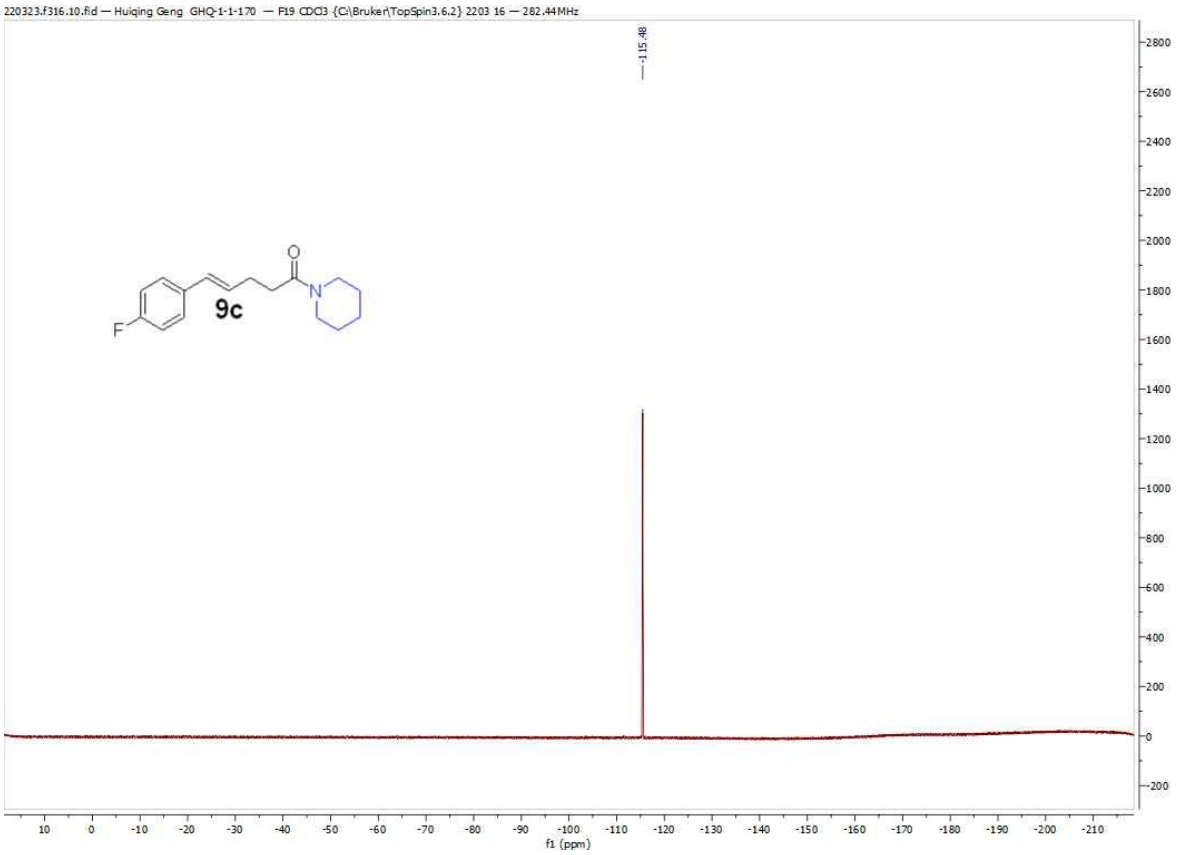
9c
f1 (ppm)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O
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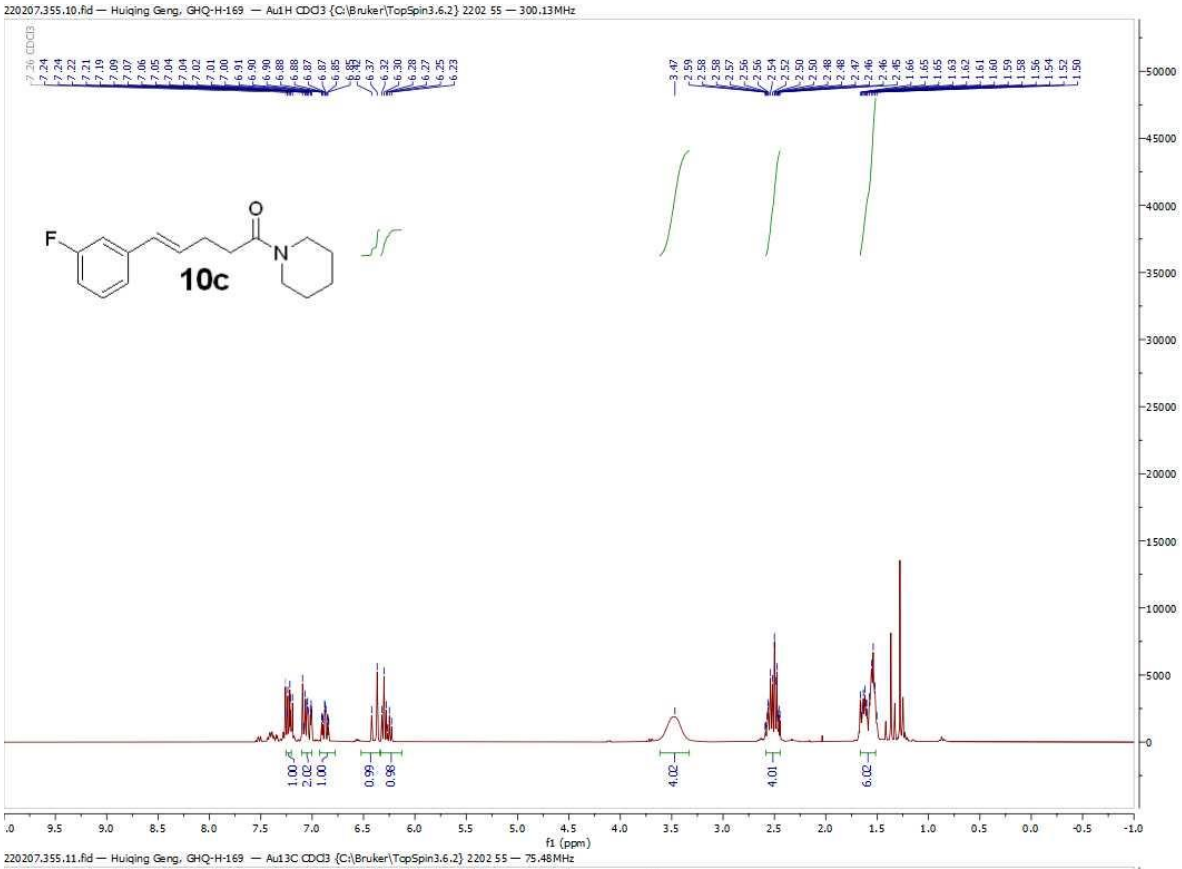
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F
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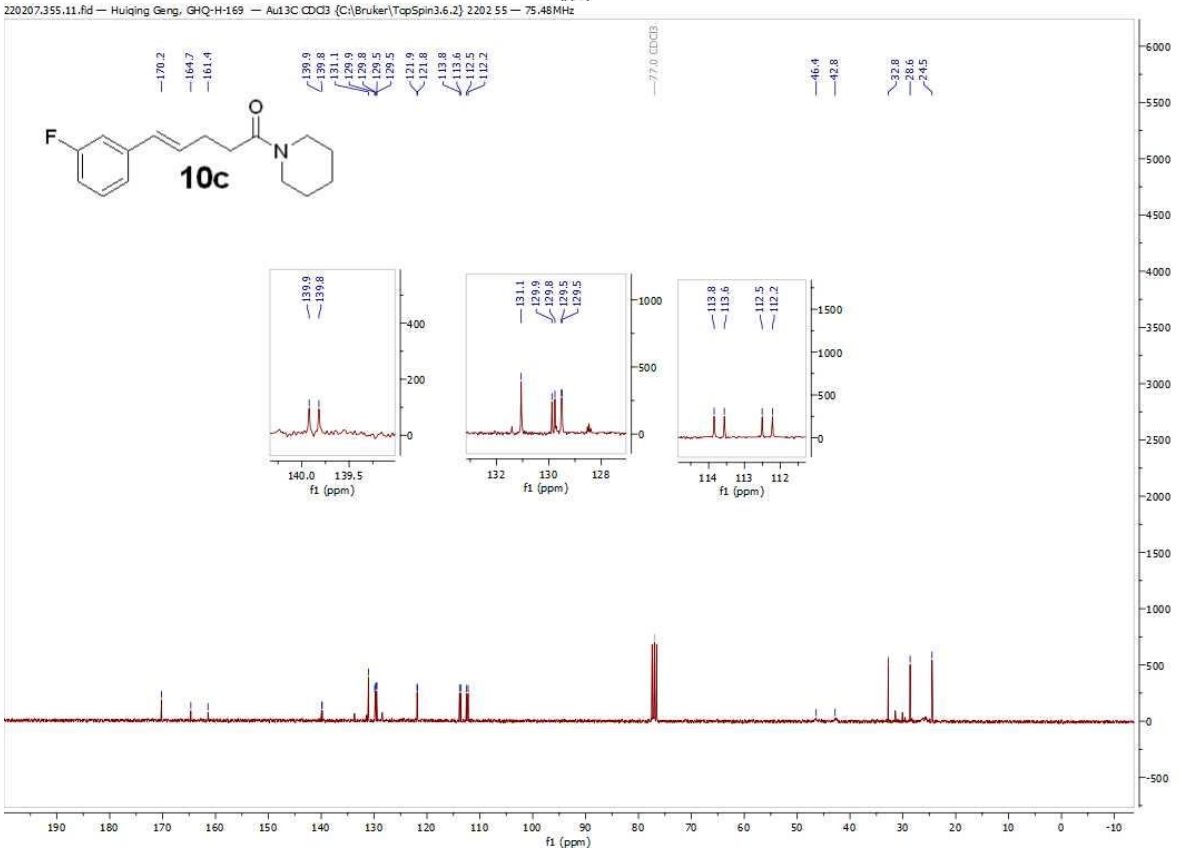
N
10c
f1 (ppm)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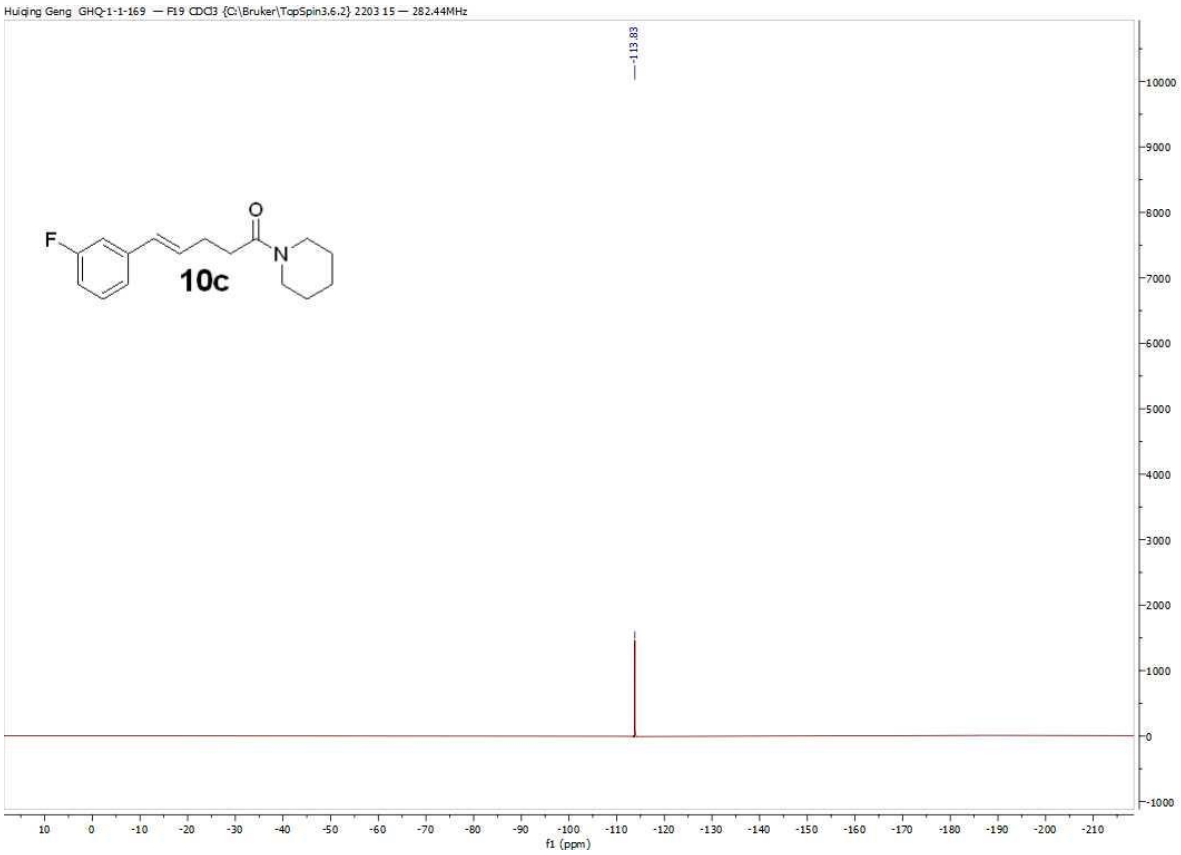
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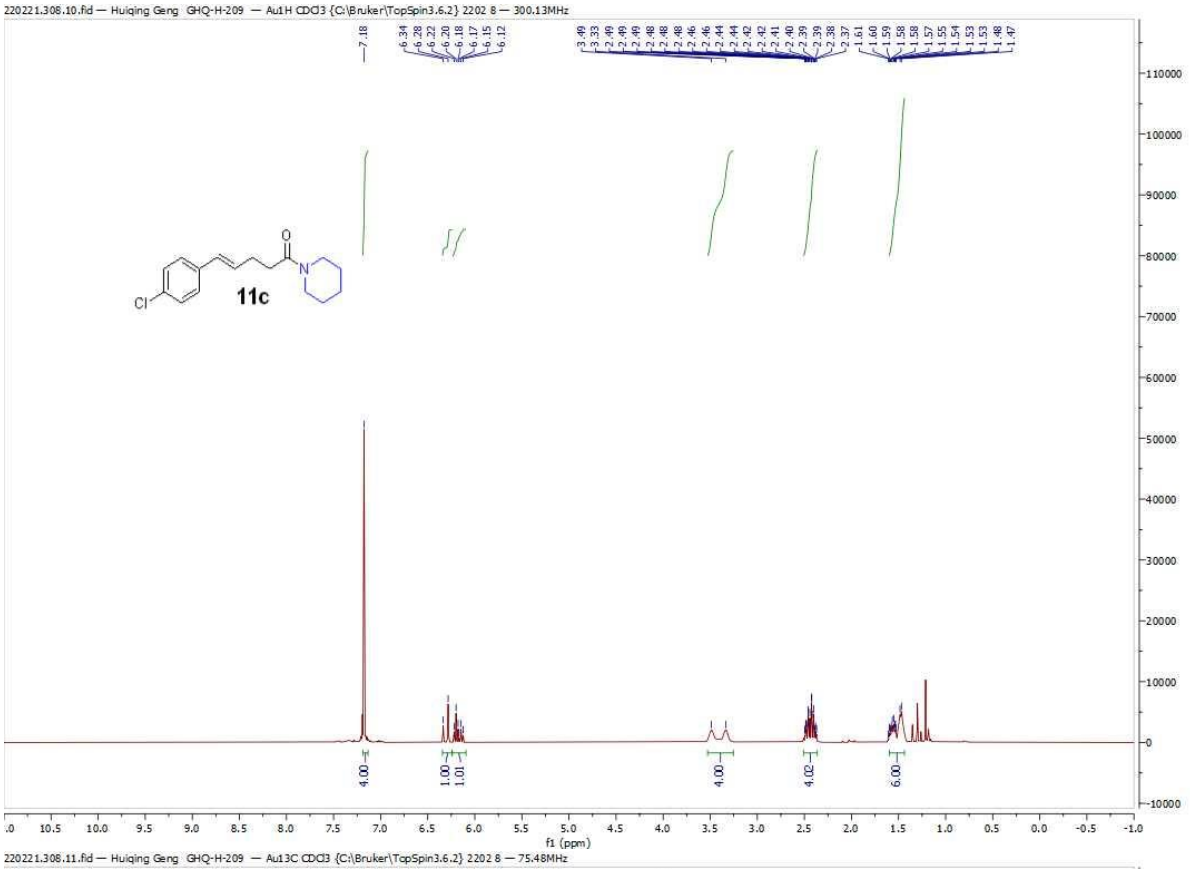
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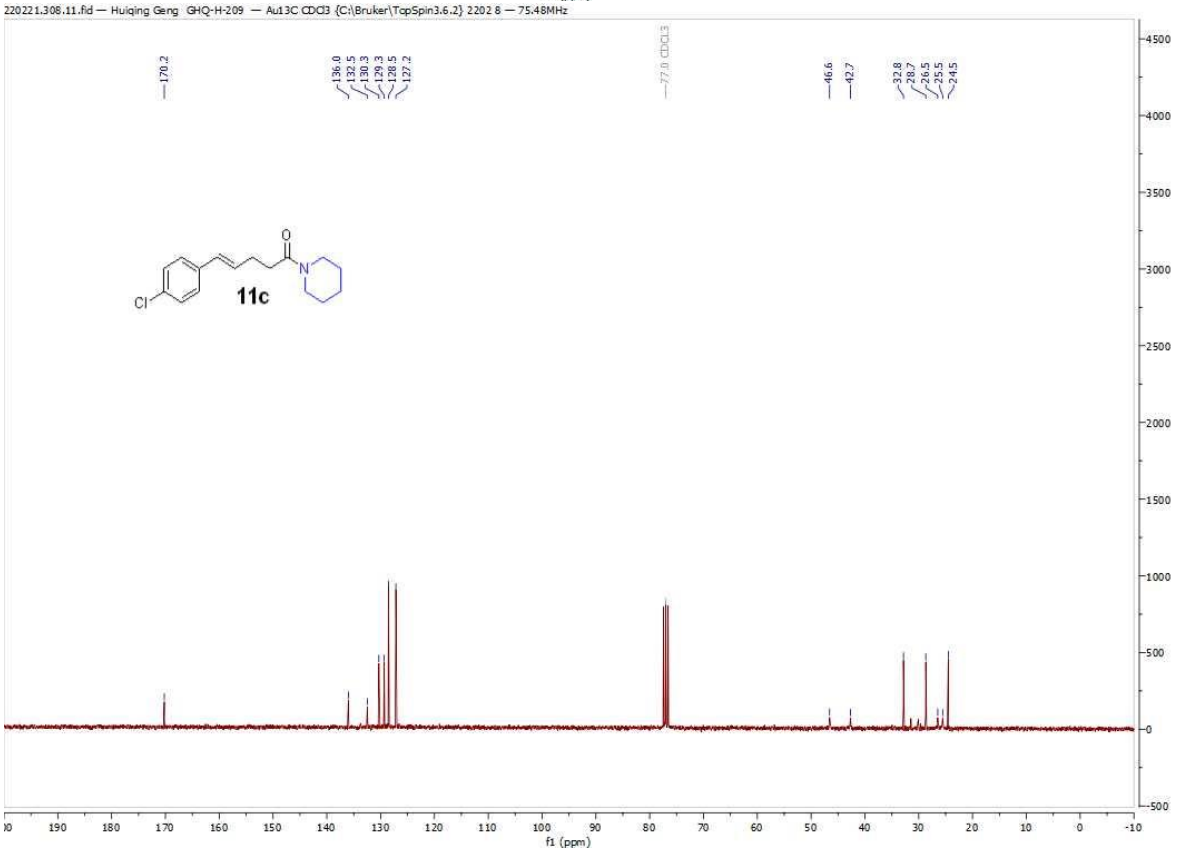
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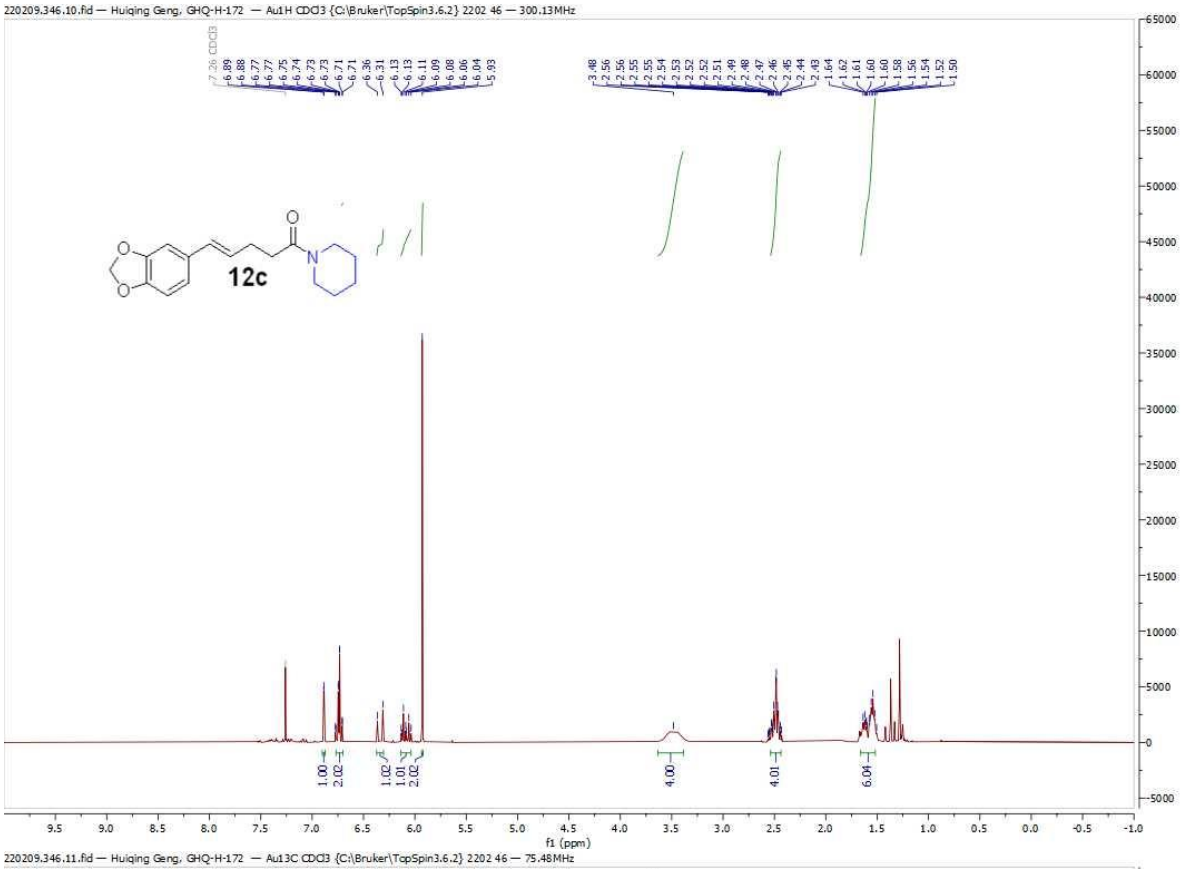
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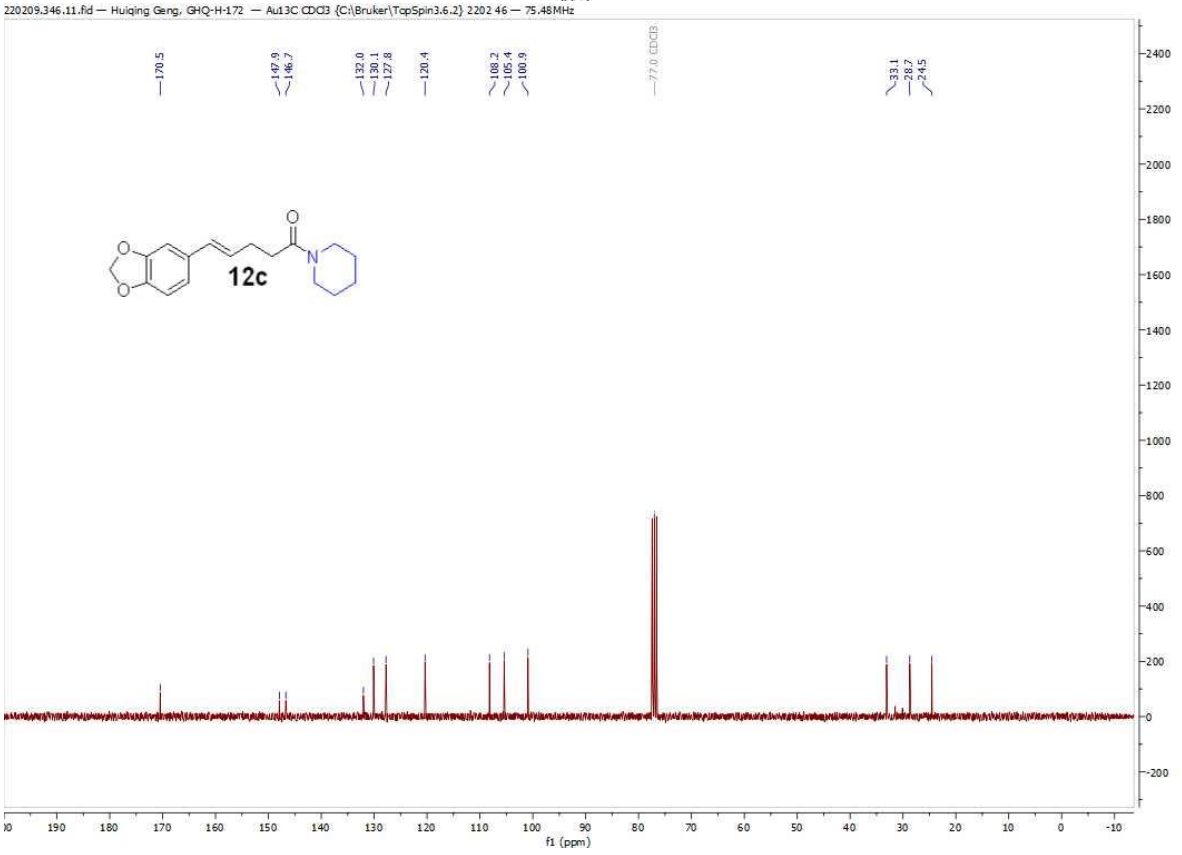
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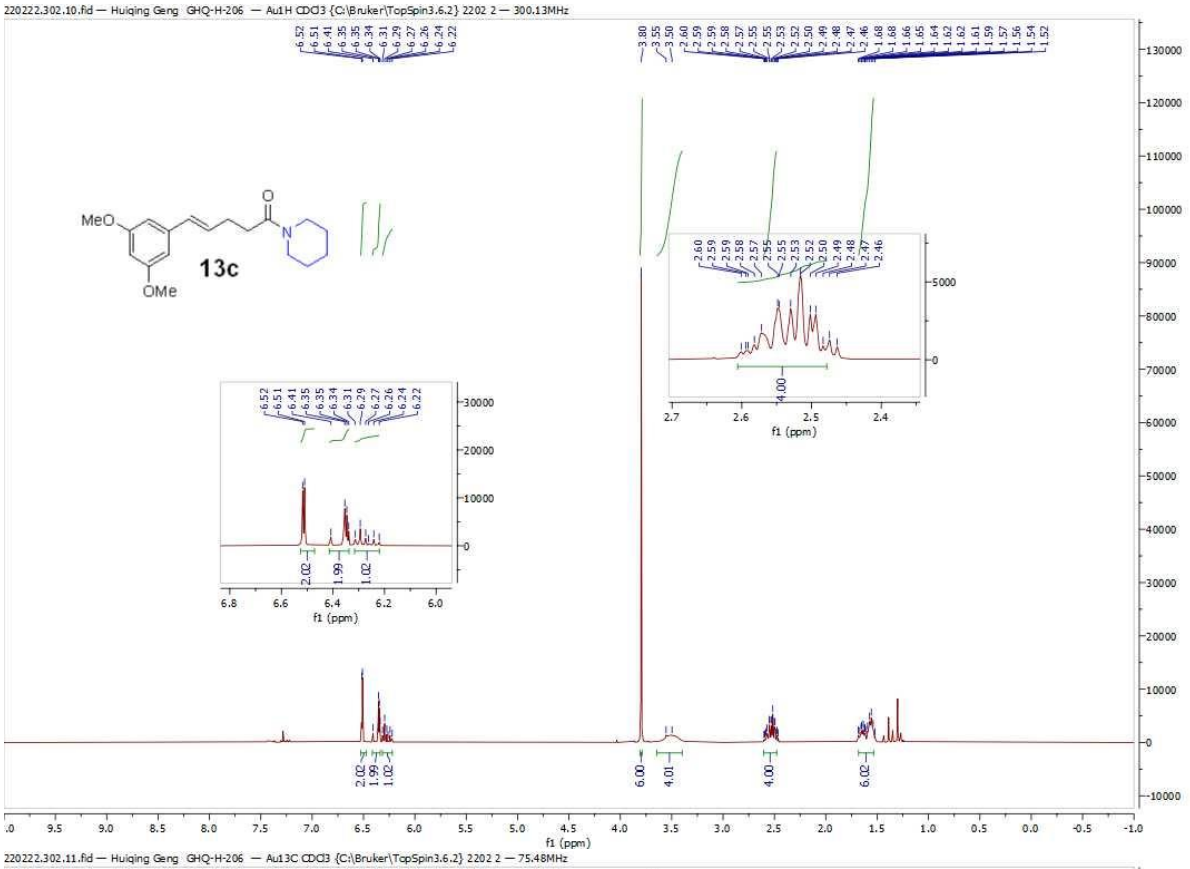
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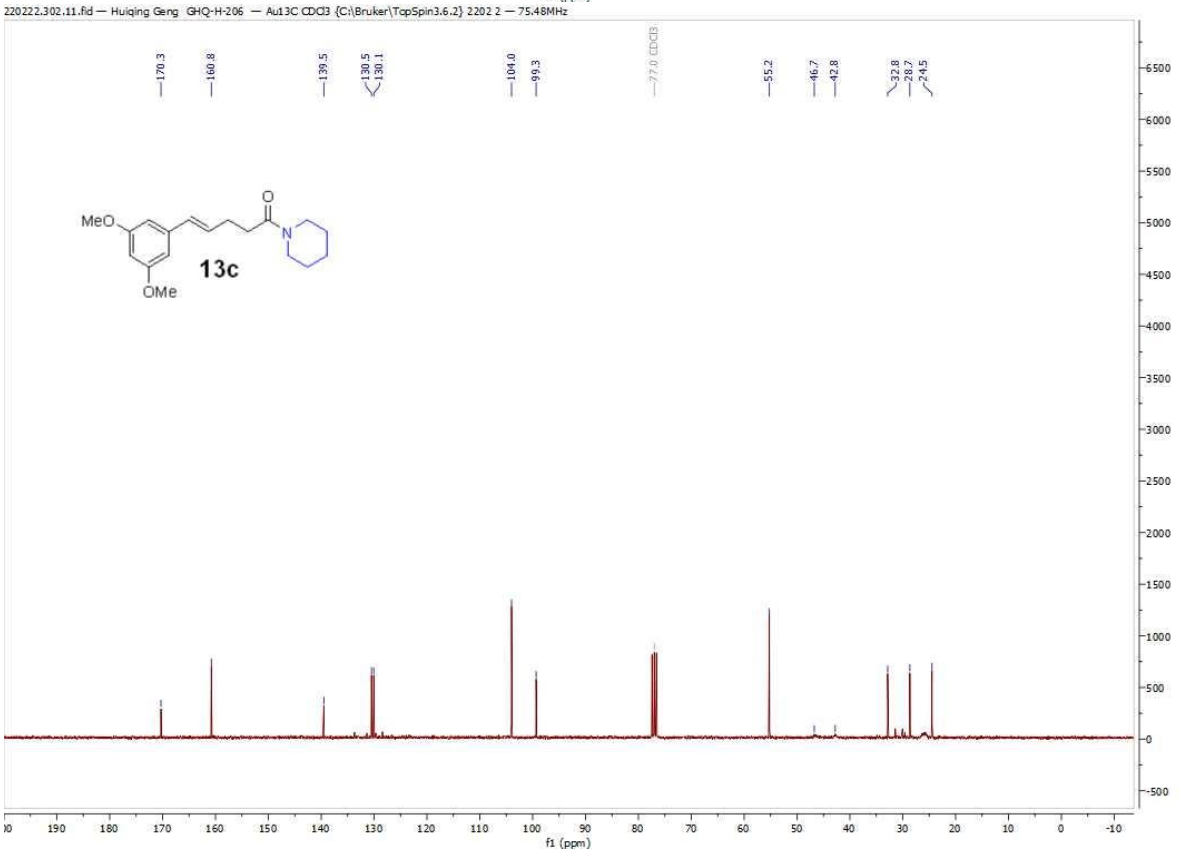
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OMe
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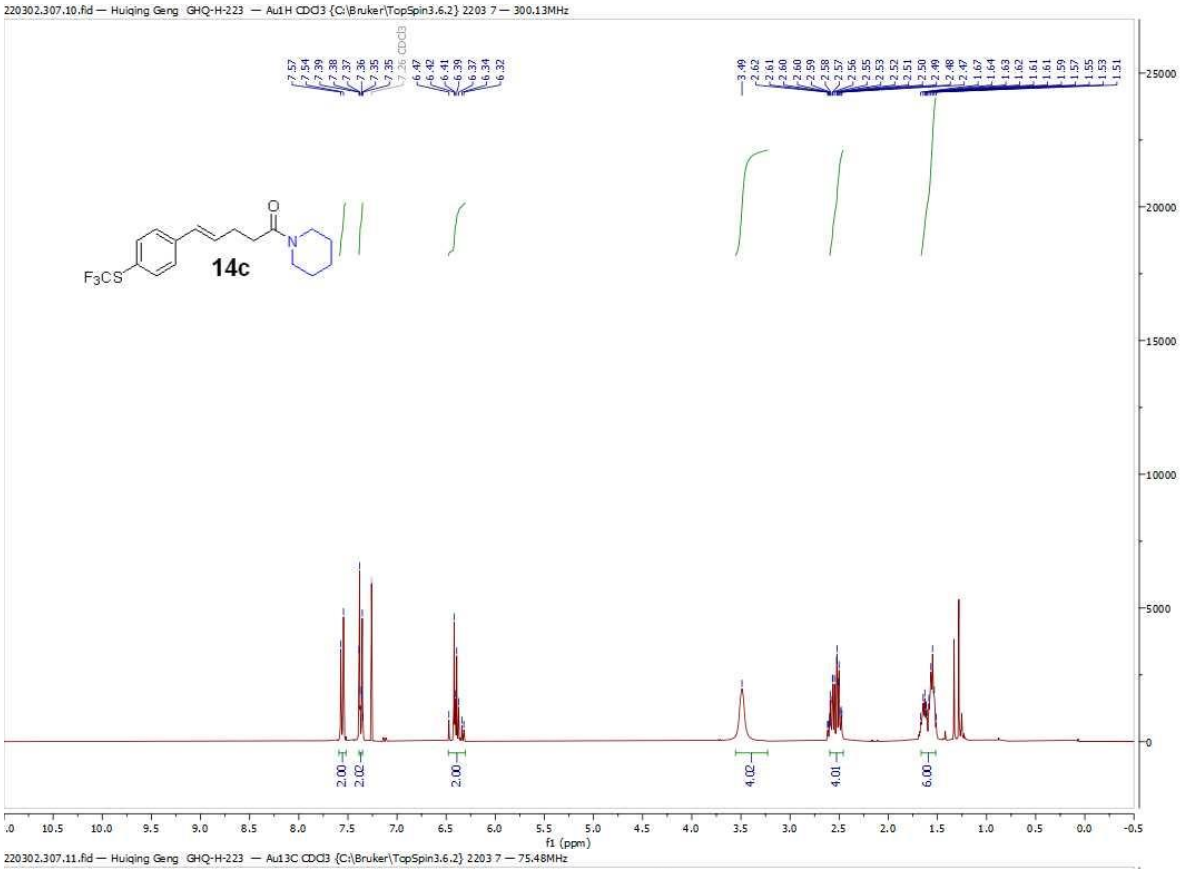
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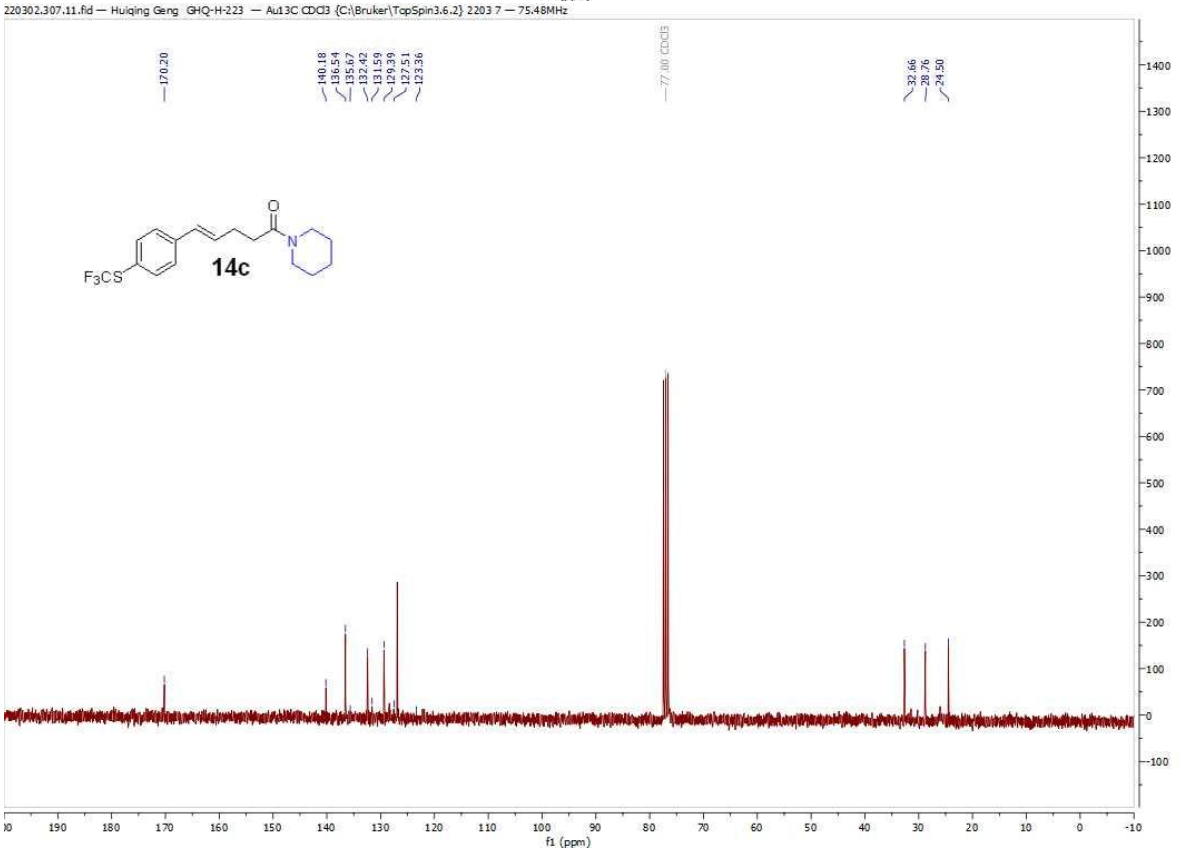
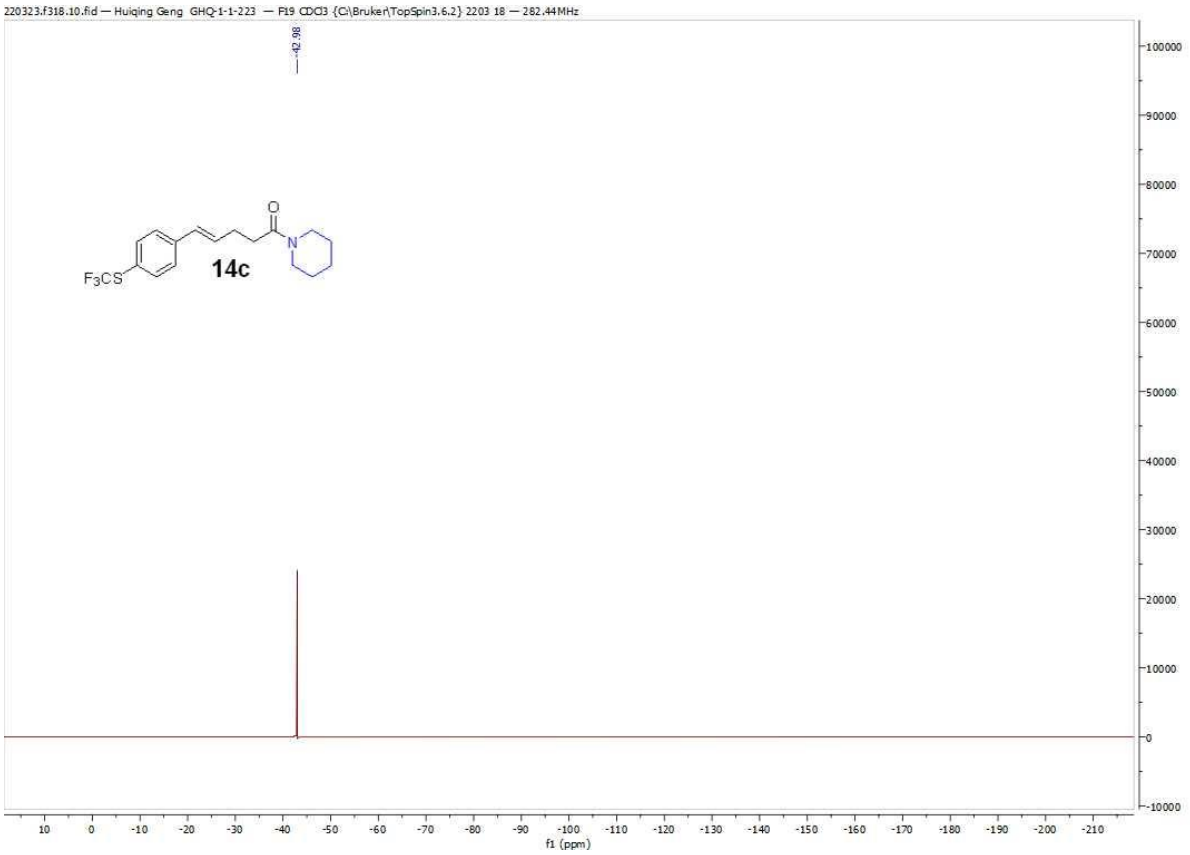
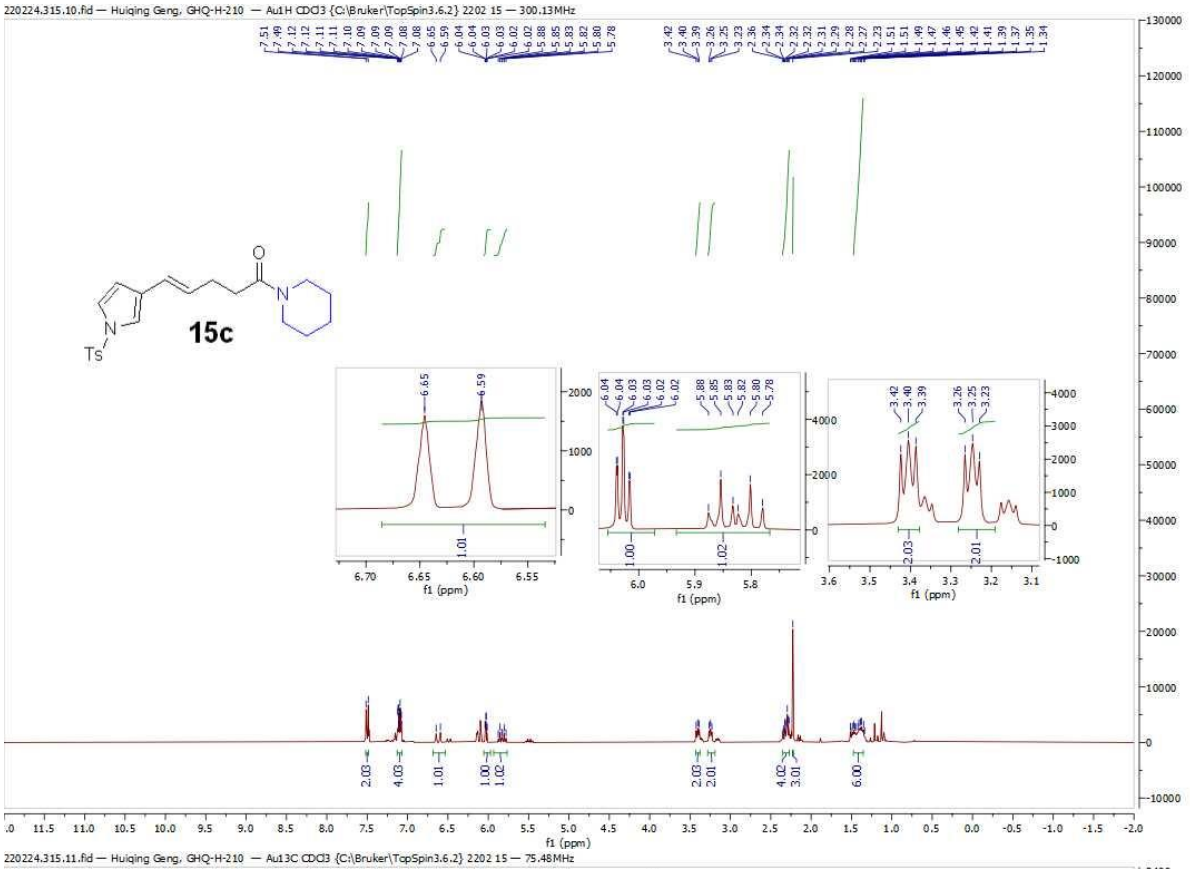
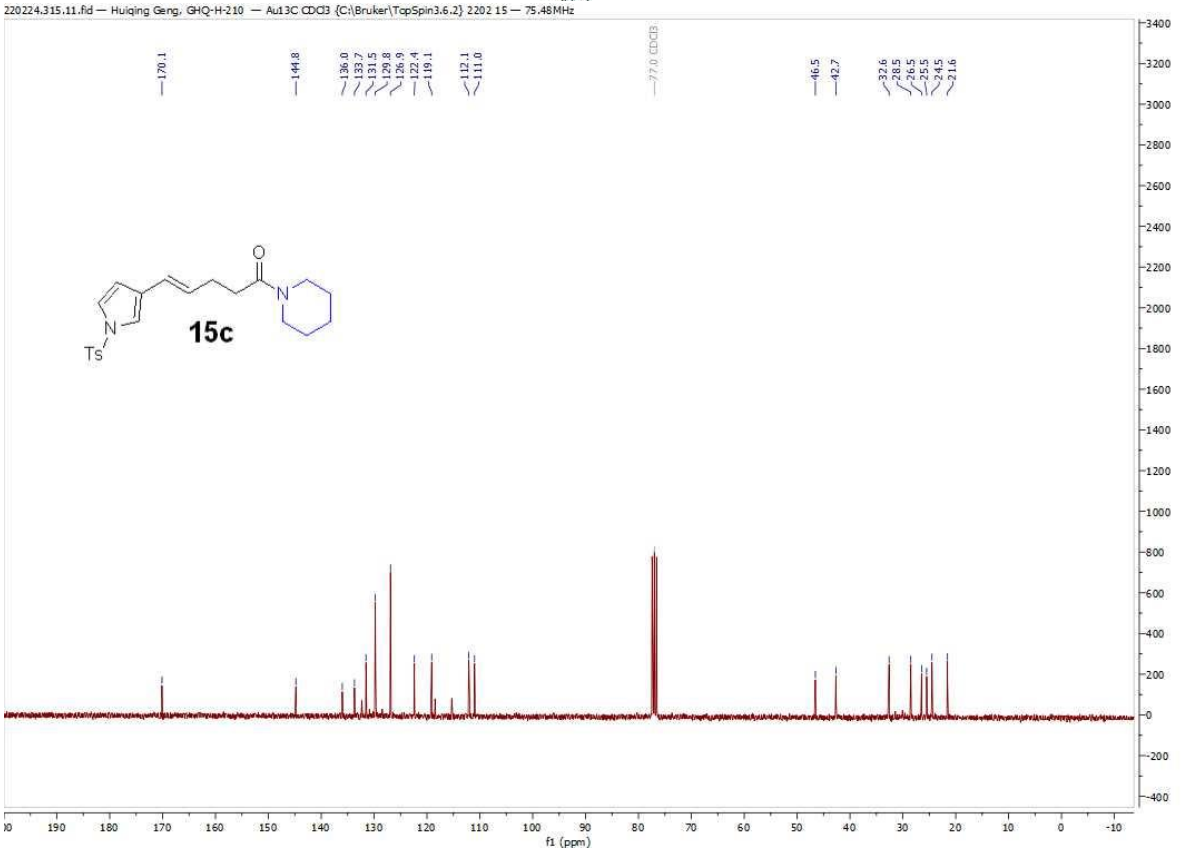
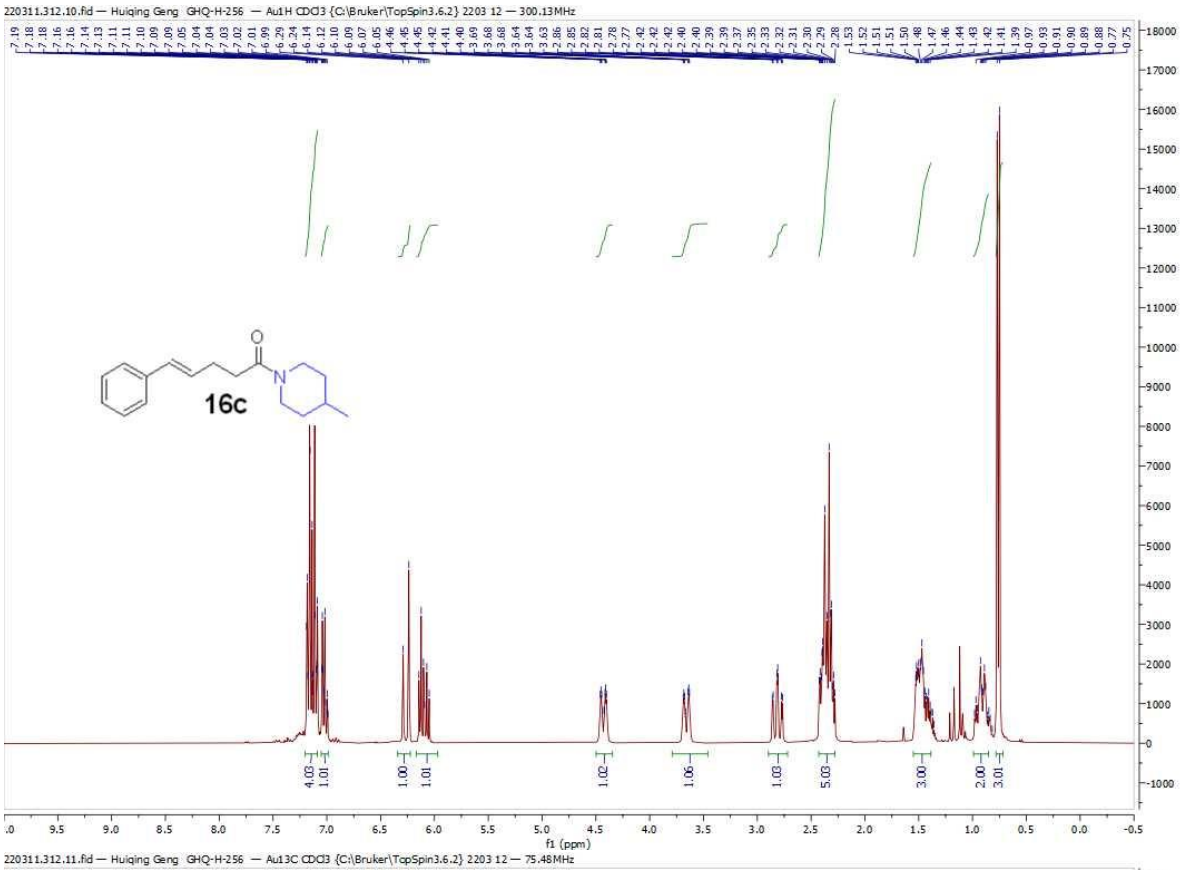
14c
f1 (ppm)

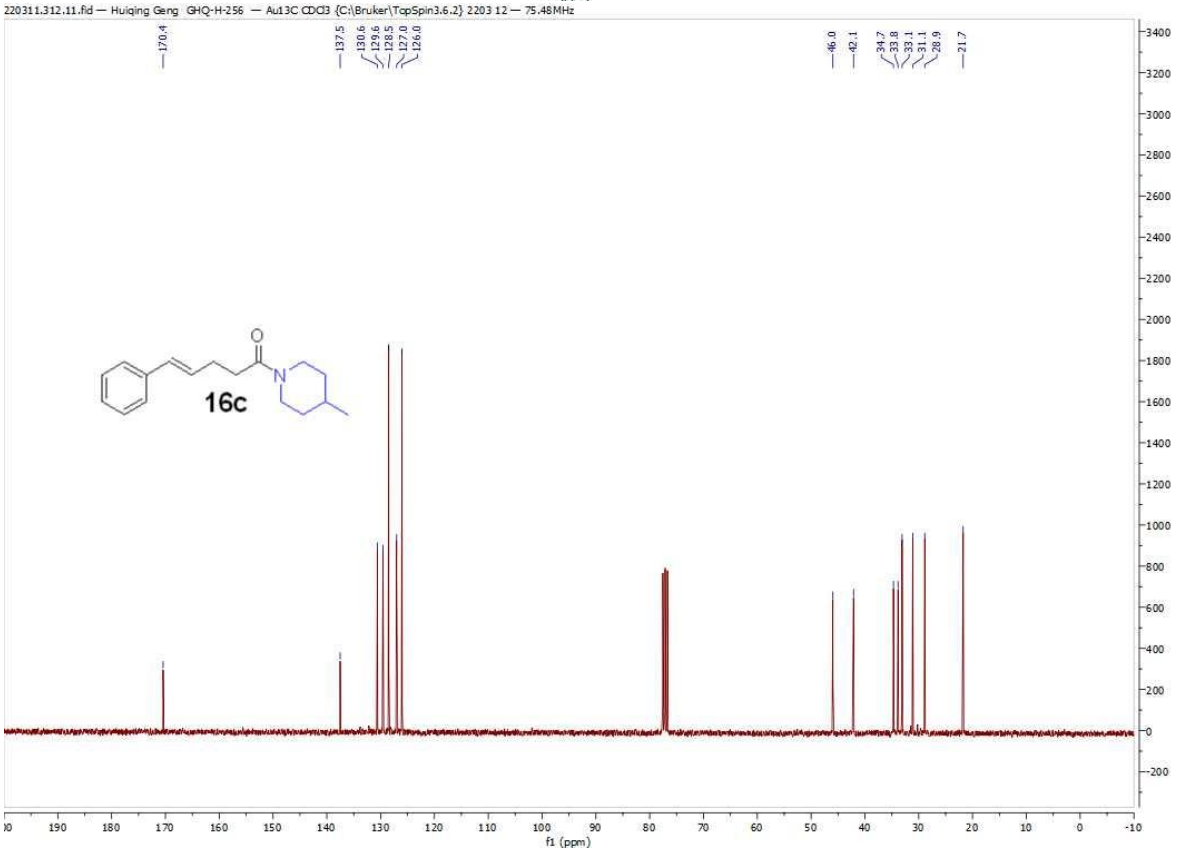

220302.307.11.fid — Huiqing Geng GHQ-H-223 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 7 — 75.48MHz
14c
f1 (ppm)

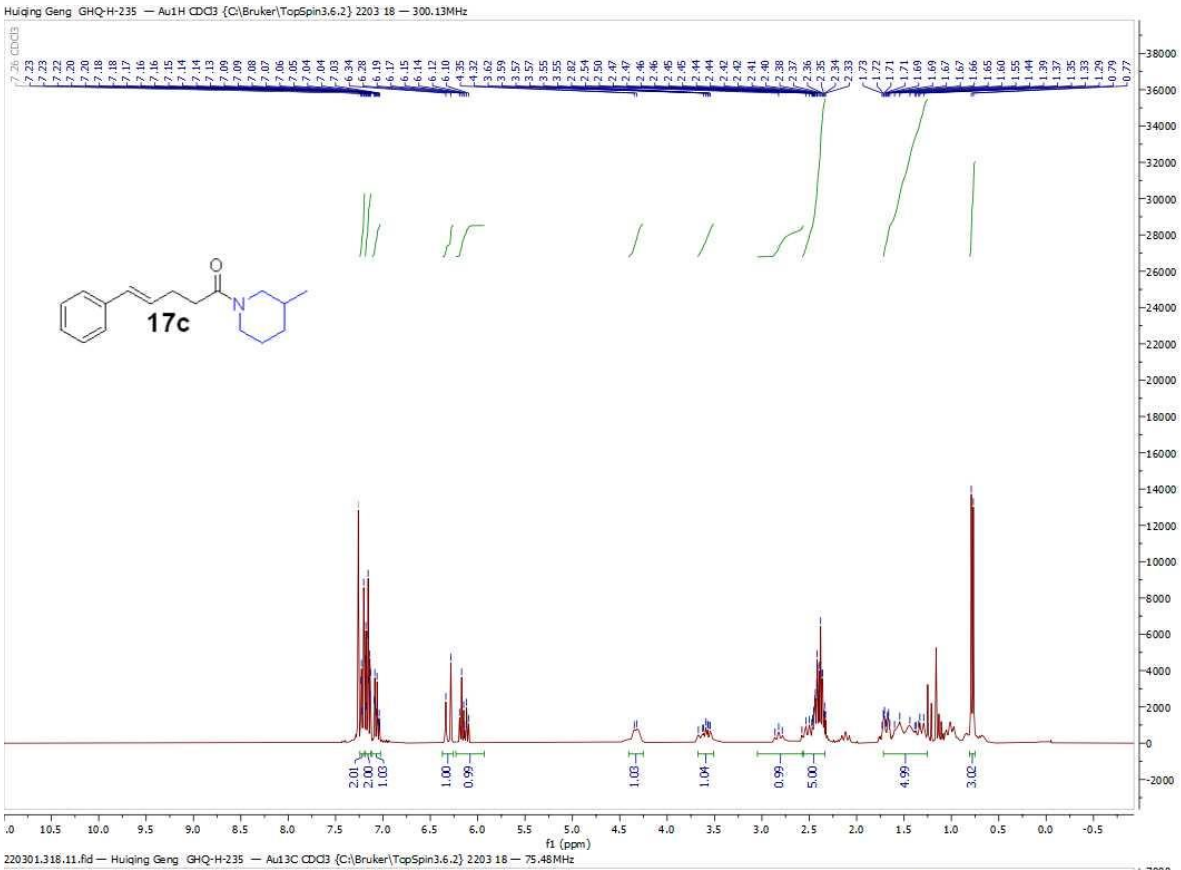

220323.f318.10.fid — Huiqing Geng GHQ-1-1-223 — F19 CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 18 — 282.44MHz
-42.98
F3CS
14c
f1 (ppm)

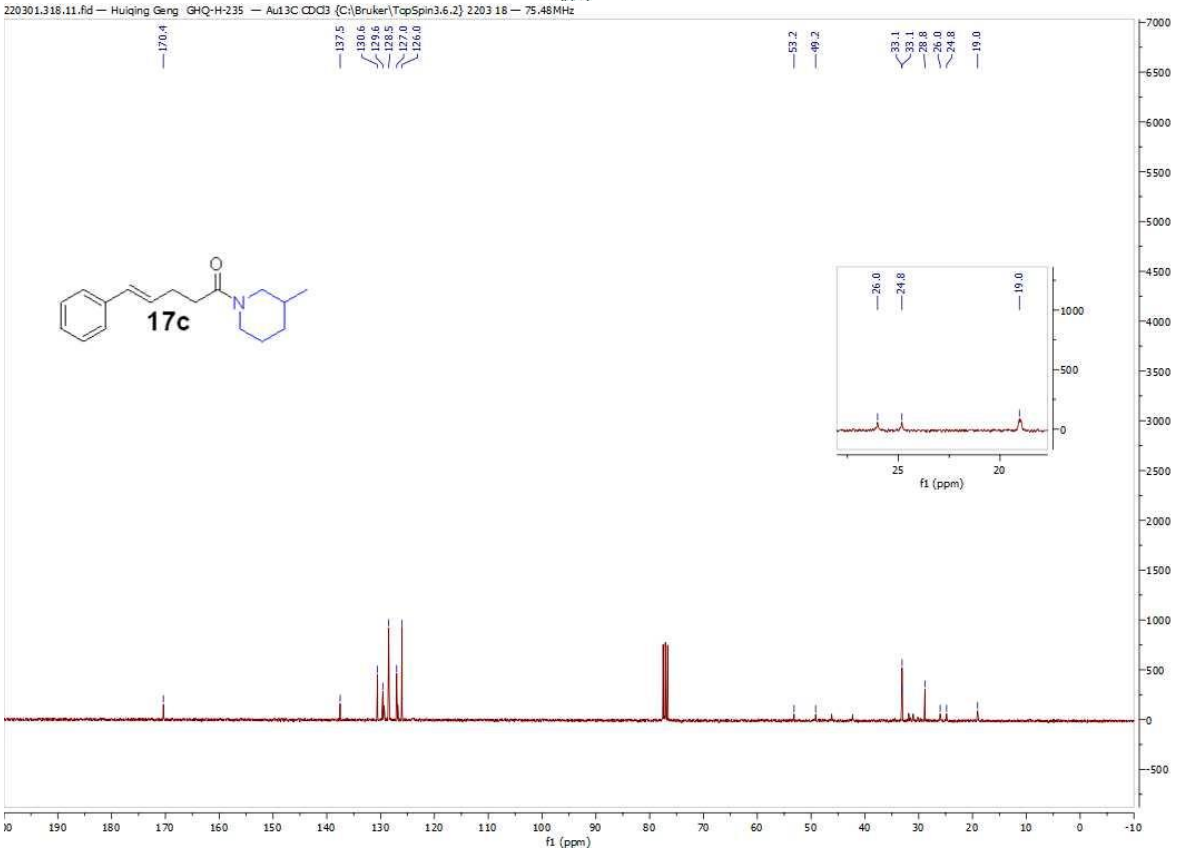

220224.315.10.fid — Huiqing Geng, GHQ-H-210 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2202 15 — 300.13MHz
15c
Ts
f1 (ppm)

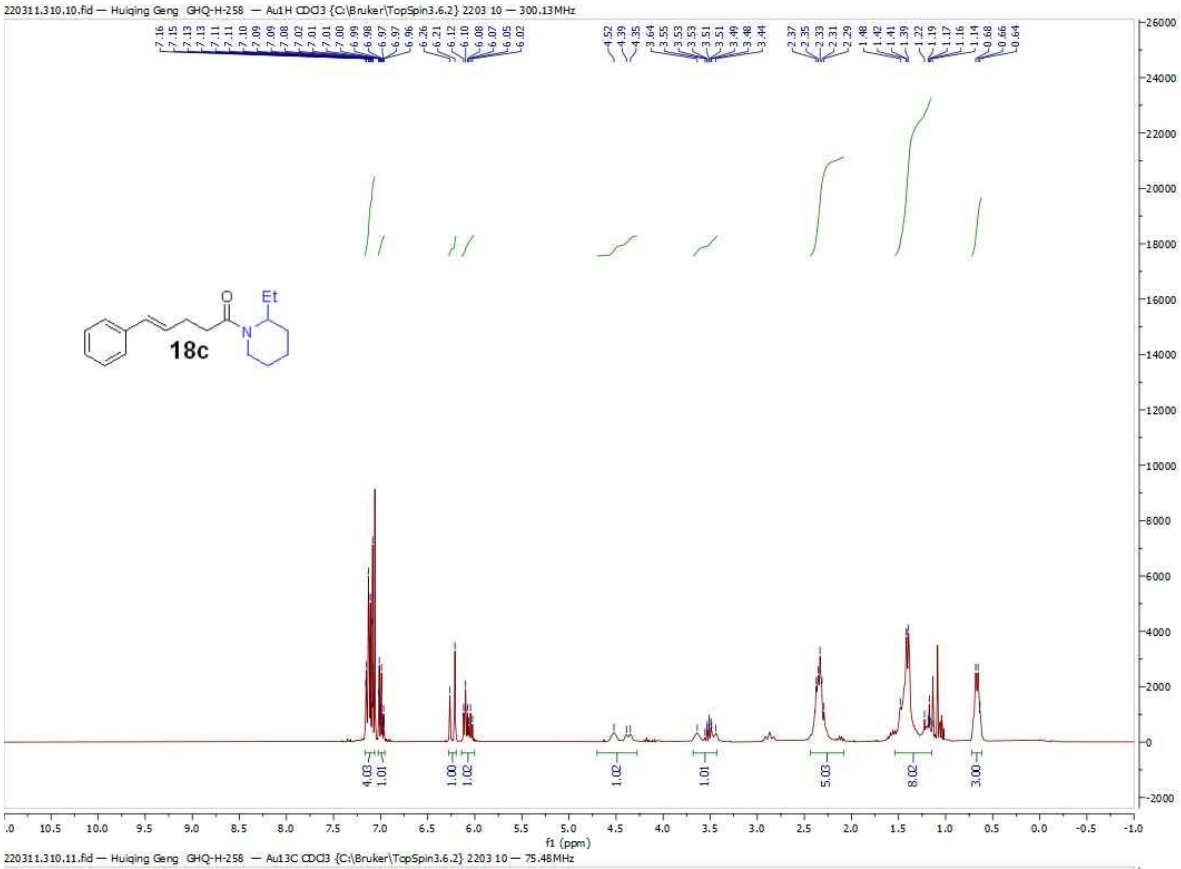

220224.315.11.fid — Huiqing Geng, GHQ-H-210 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2202 15 — 75.48MHz
15c
Ts
f1 (ppm)

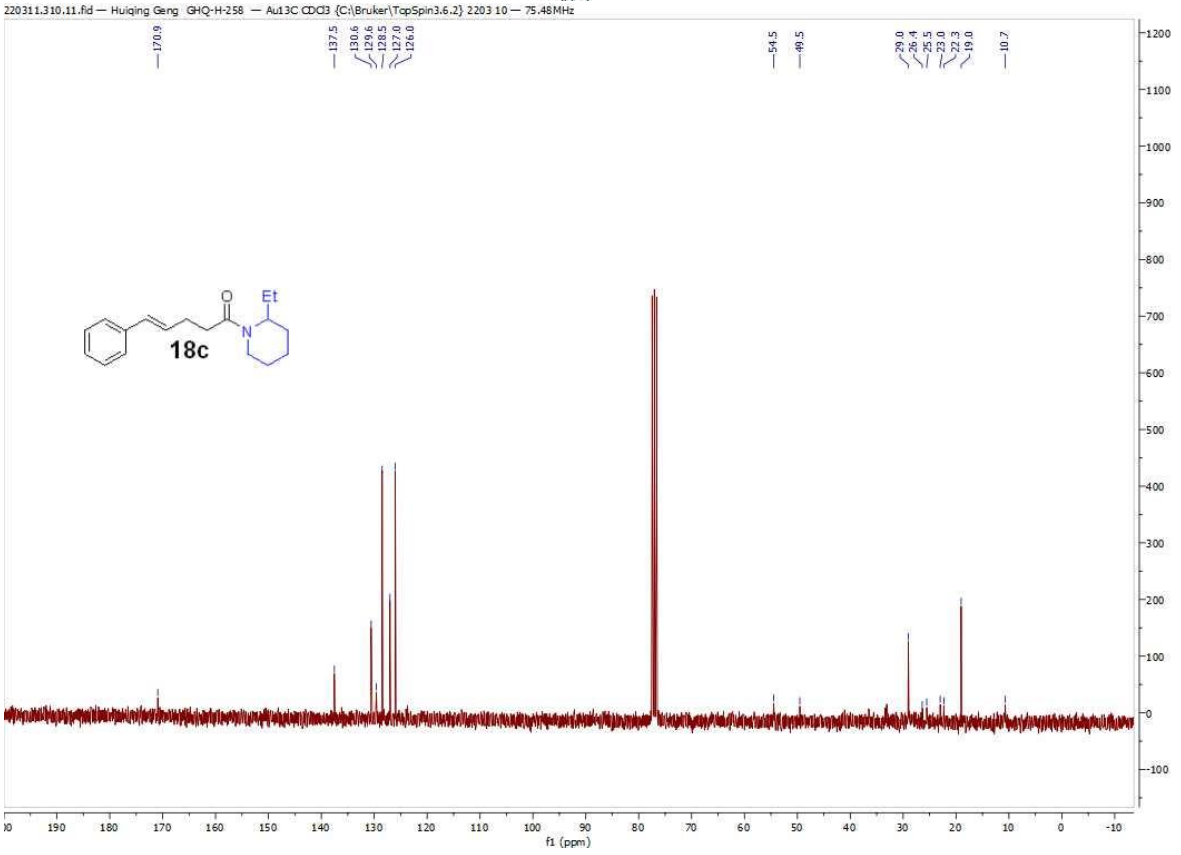

220311.312.10.fid — Huiqing Geng GHQ-H-256 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 12 — 300.13MHz
16c
f1 (ppm)

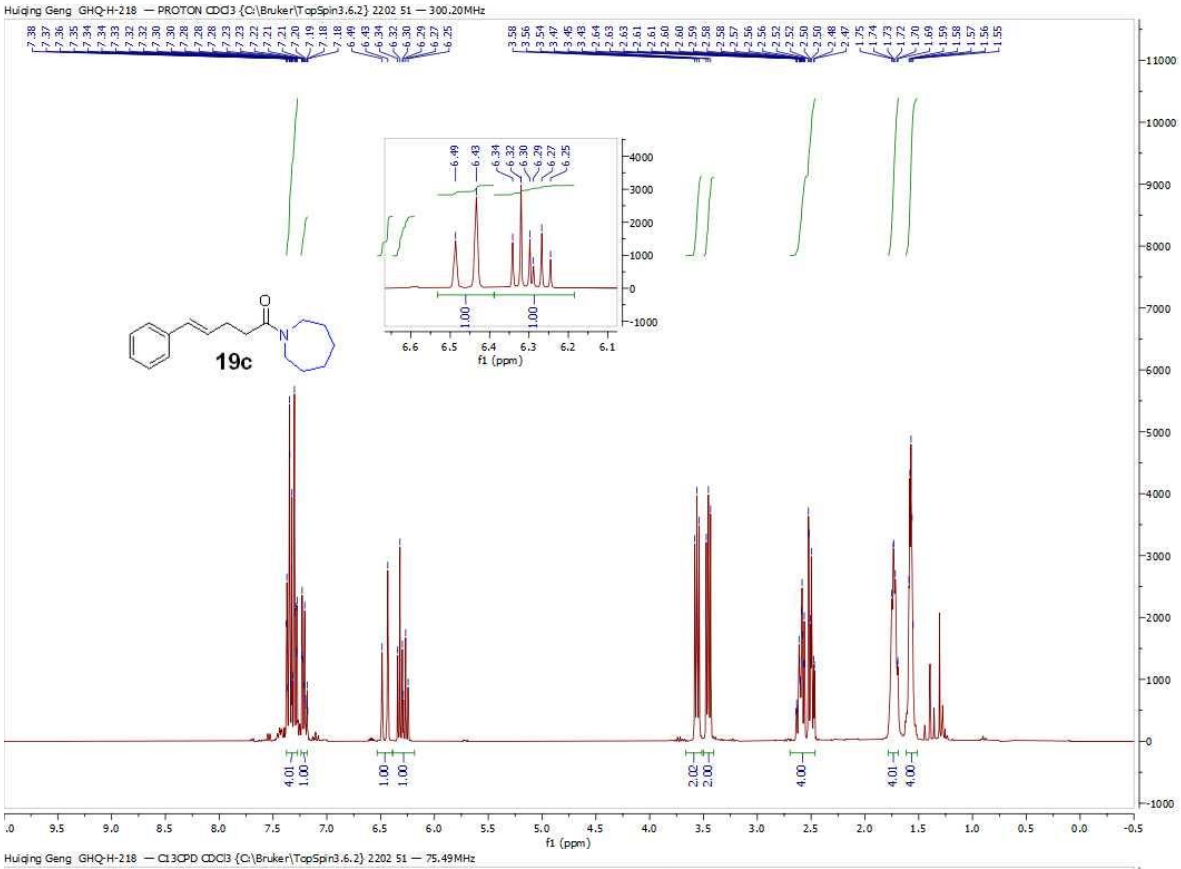

220311.312.11.fid — Huiqing Geng GHQ-H-256 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 12 — 75.48MHz
16c
f1 (ppm)

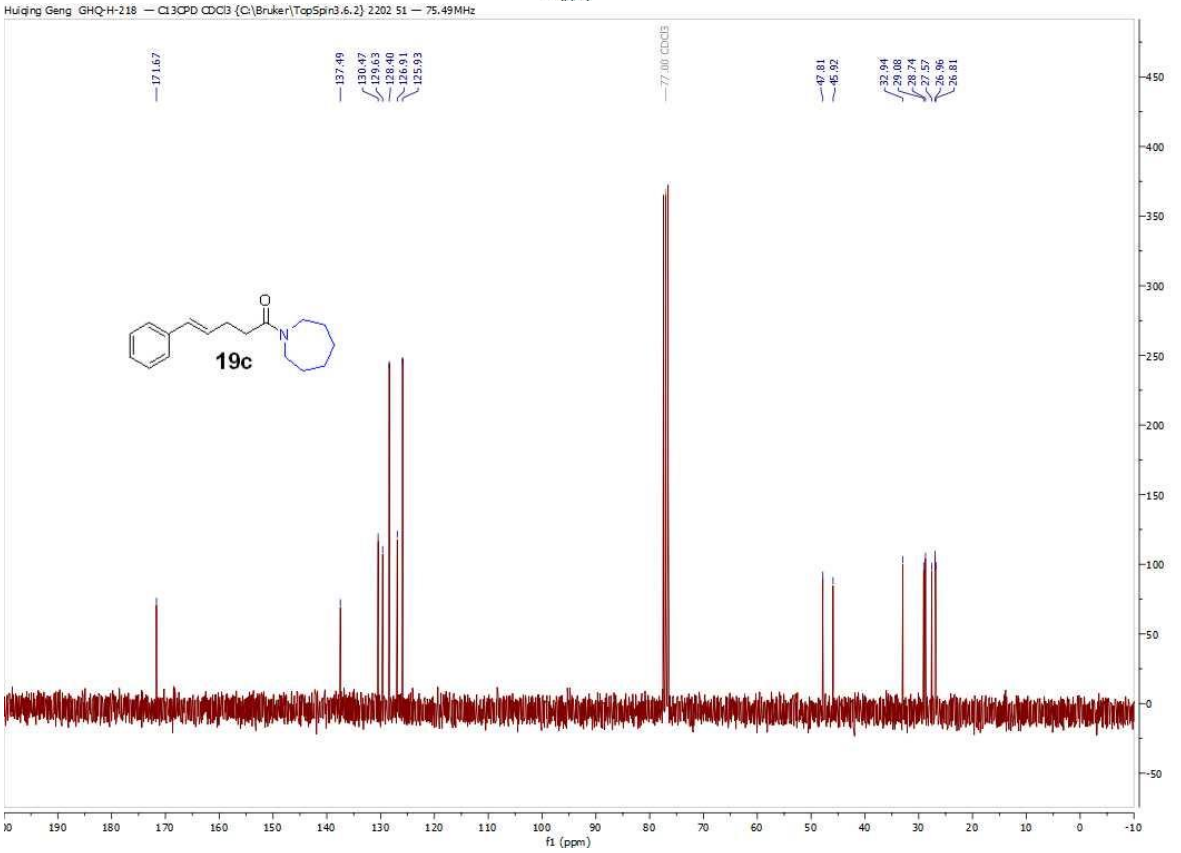

Huiqing Geng GHQ-H-235 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 18 — 300.13MHz
17c
f1 (ppm)

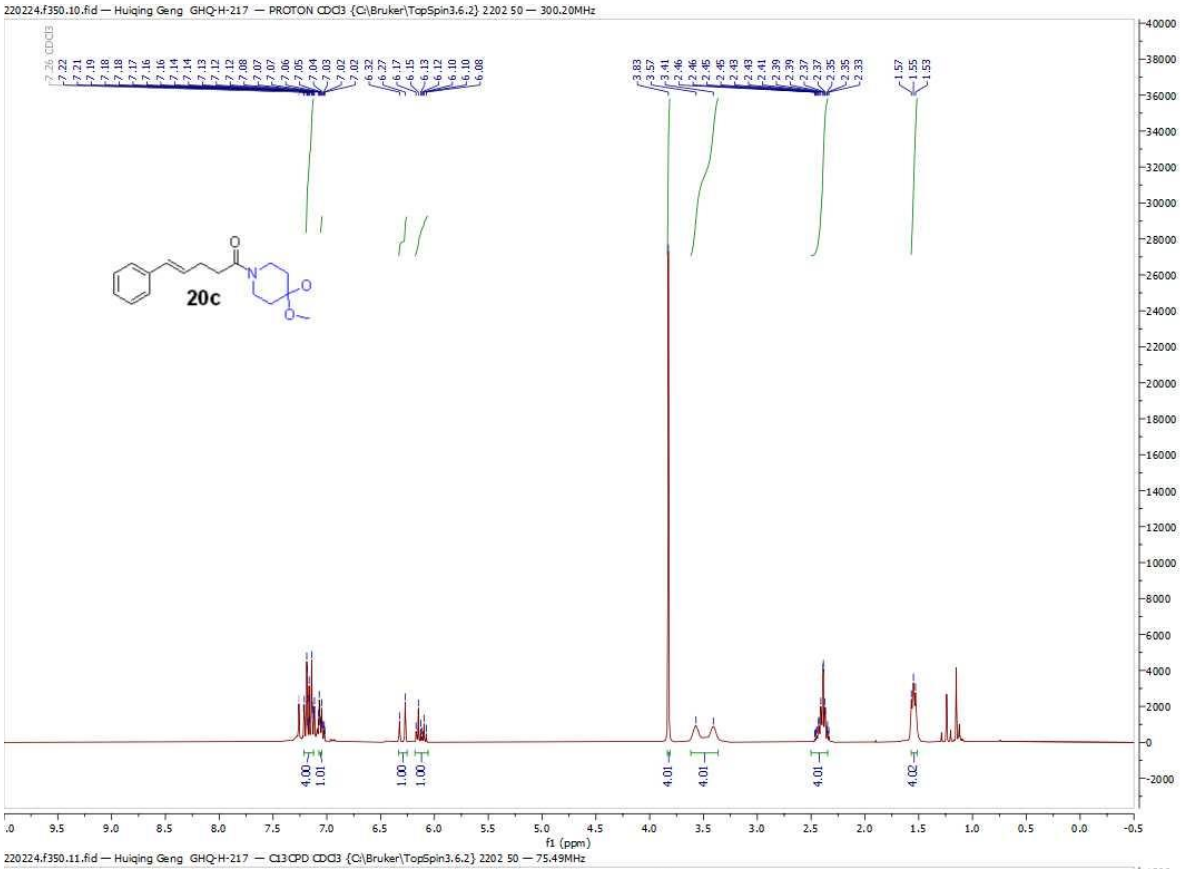

220301.318.11.fid — Huiqing Geng GHQ-H-235 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 18 — 75.48MHz
17c
f1 (ppm)

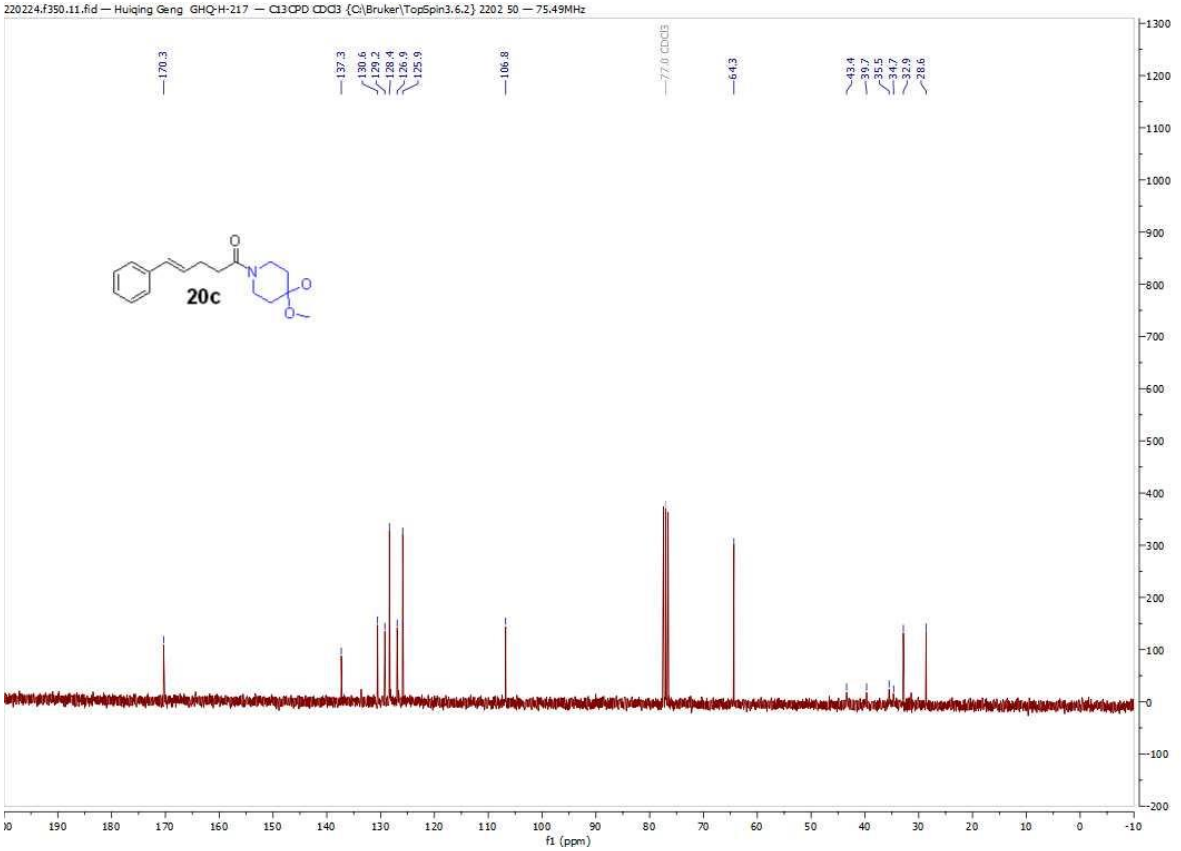

220311.310.10.fid — Huiqing Geng GHQ-H-258 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 10 — 300.13MHz
18c
f1 (ppm)

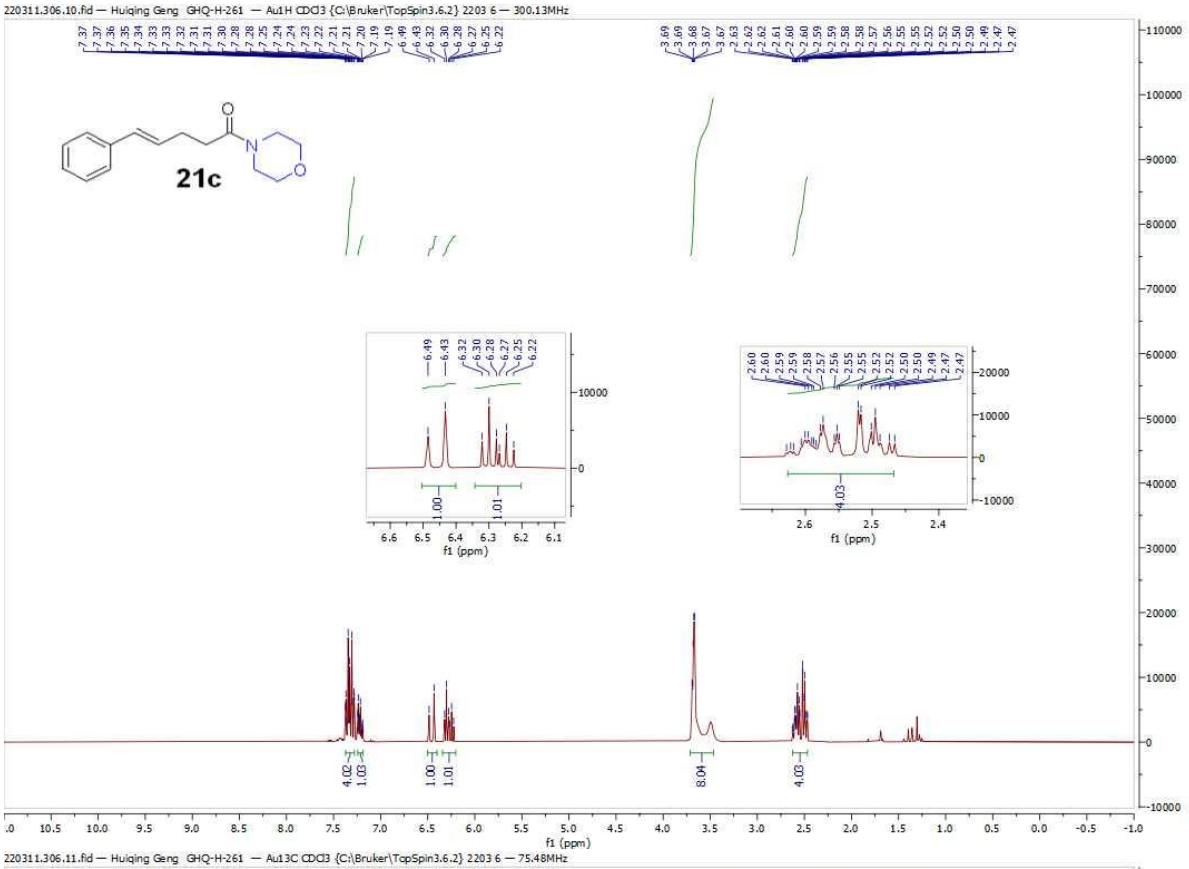

220311.310.11.fid — Huiqing Geng GHQ-H-258 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 10 — 75.48MHz
18c
f1 (ppm)

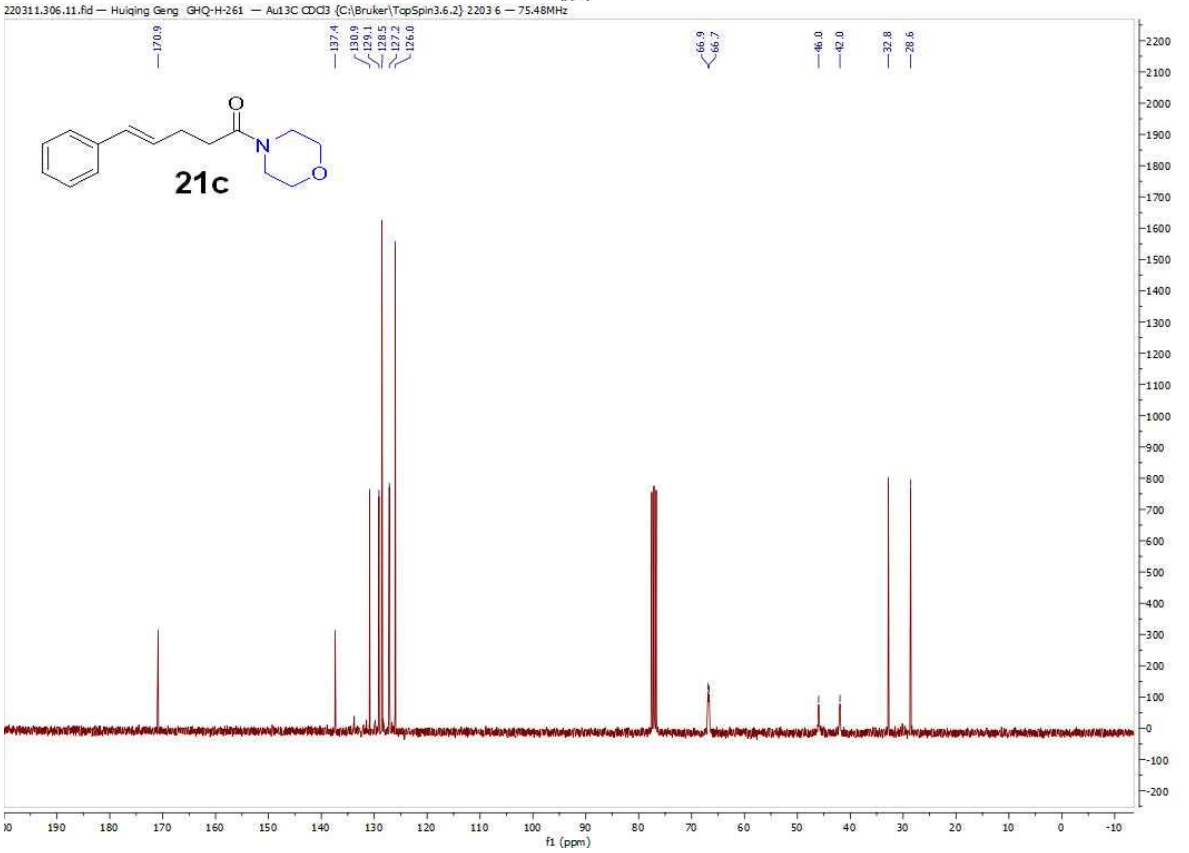

Huiqing Geng GHQ-H-218 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2202 51 — 300.20MHz
19c
f1 (ppm)

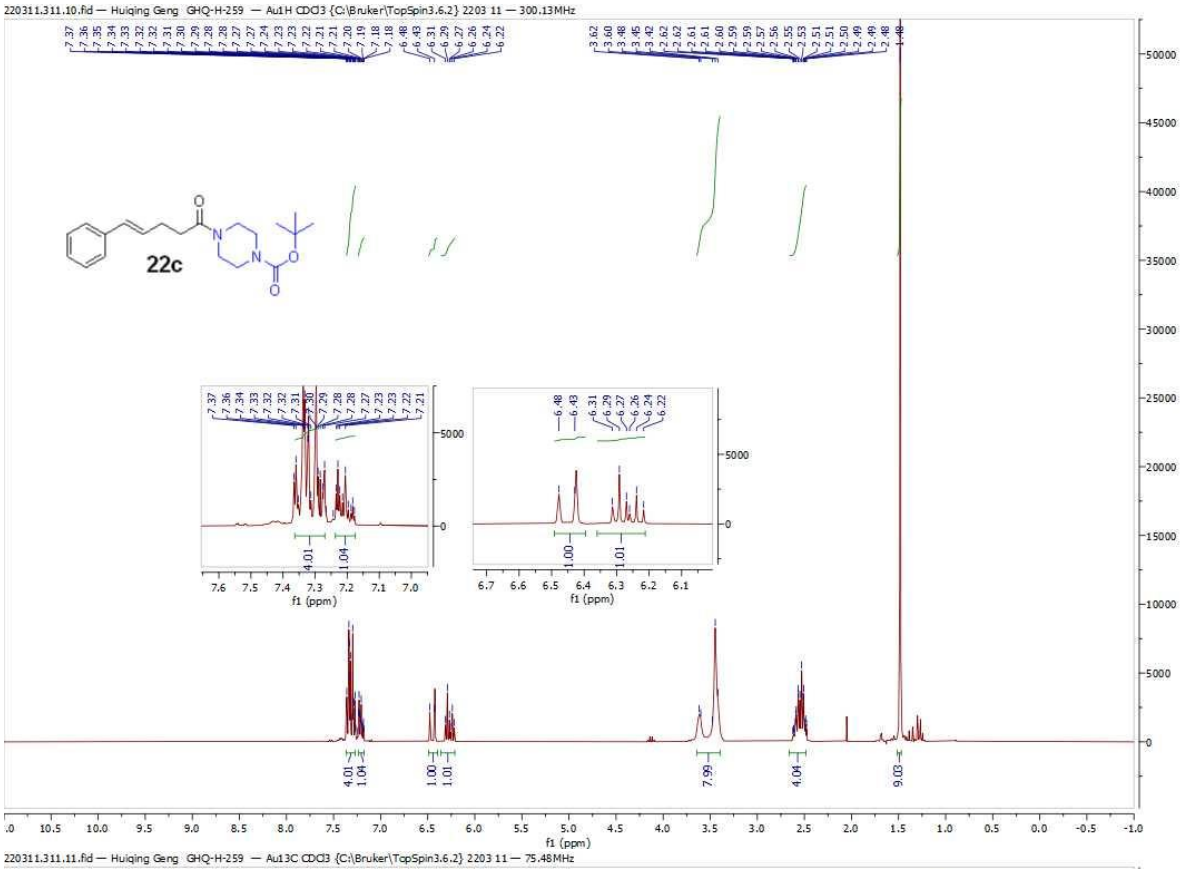

Huiqing Geng GHQ-H-218 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2202 51 — 75.49MHz
19c
f1 (ppm)

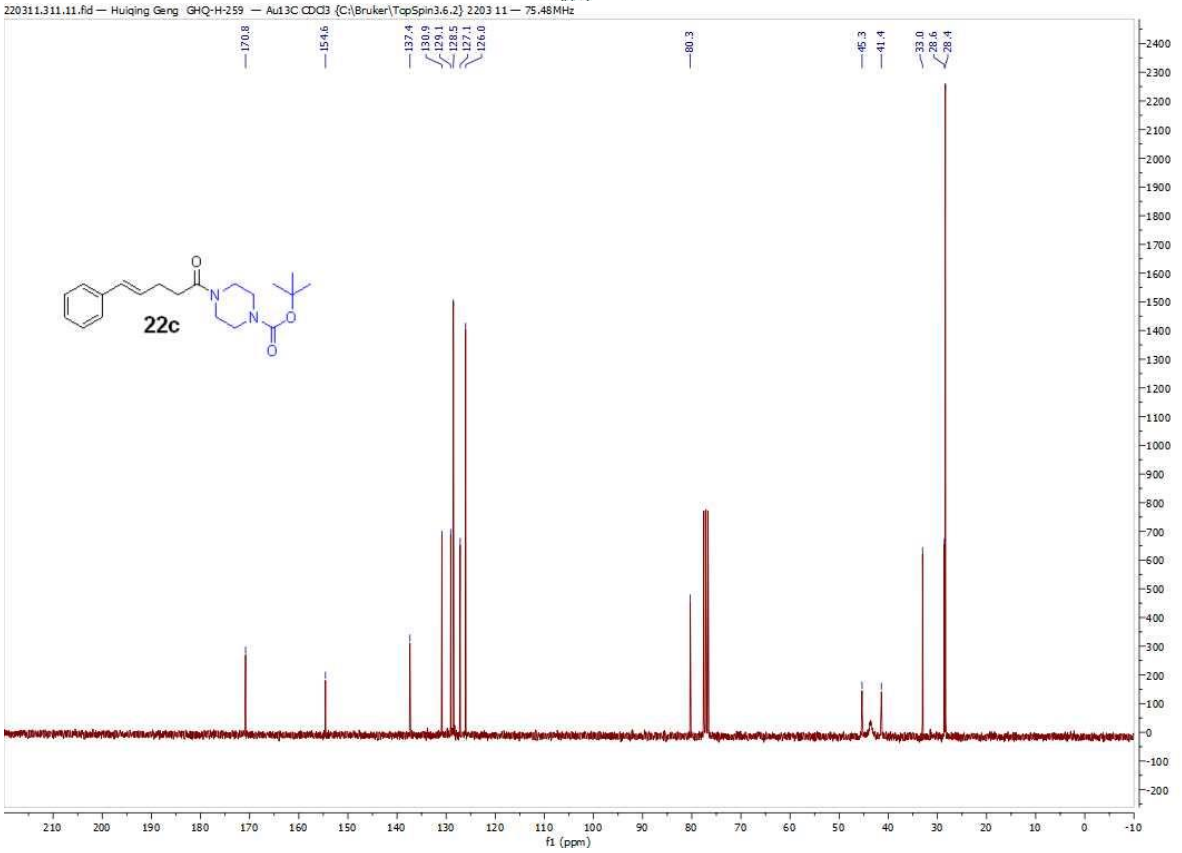

220224.f350.10.fid — Huiqing Geng GHQ-H-217 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2202 50 — 300.20MHz
20c
f1 (ppm)

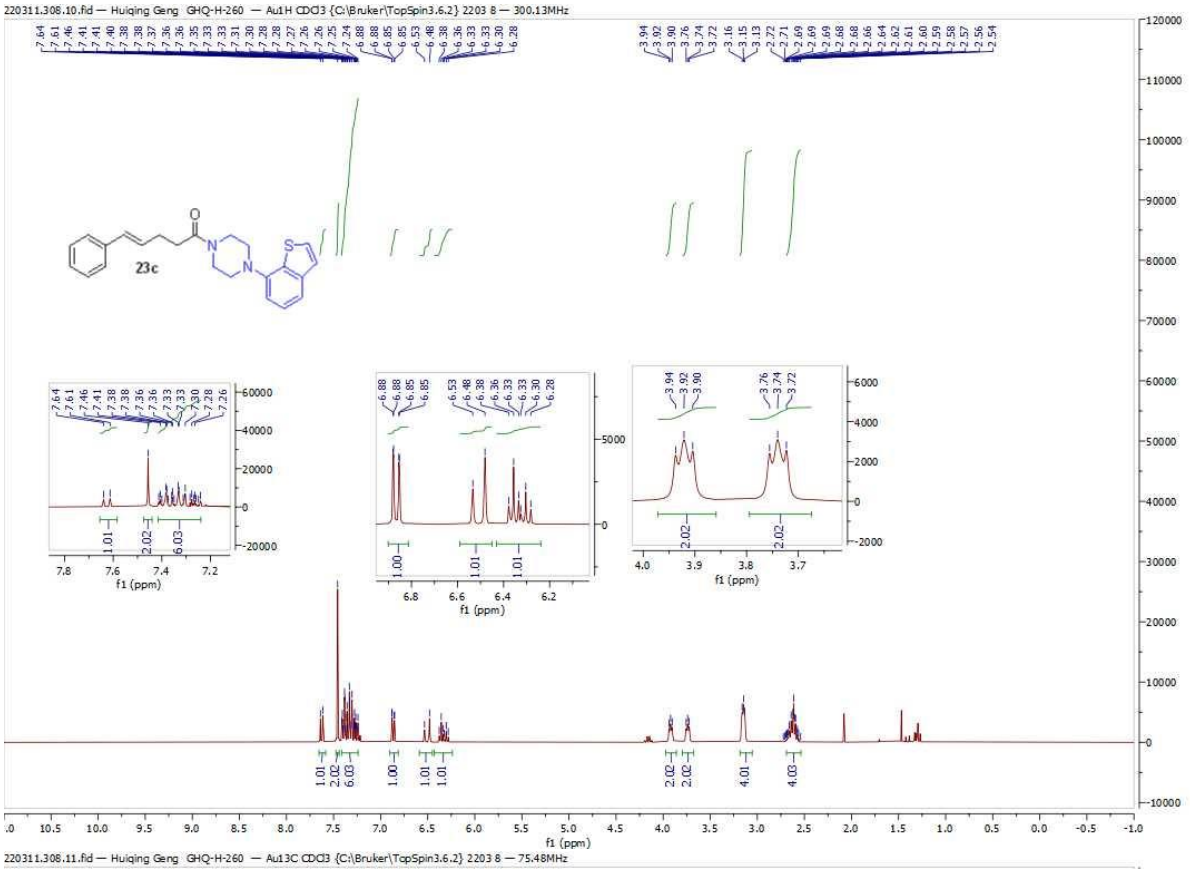

220224.f350.11.fid — Huiqing Geng GHQ-H-217 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2202 50 — 75.49MHz
20c
f1 (ppm)

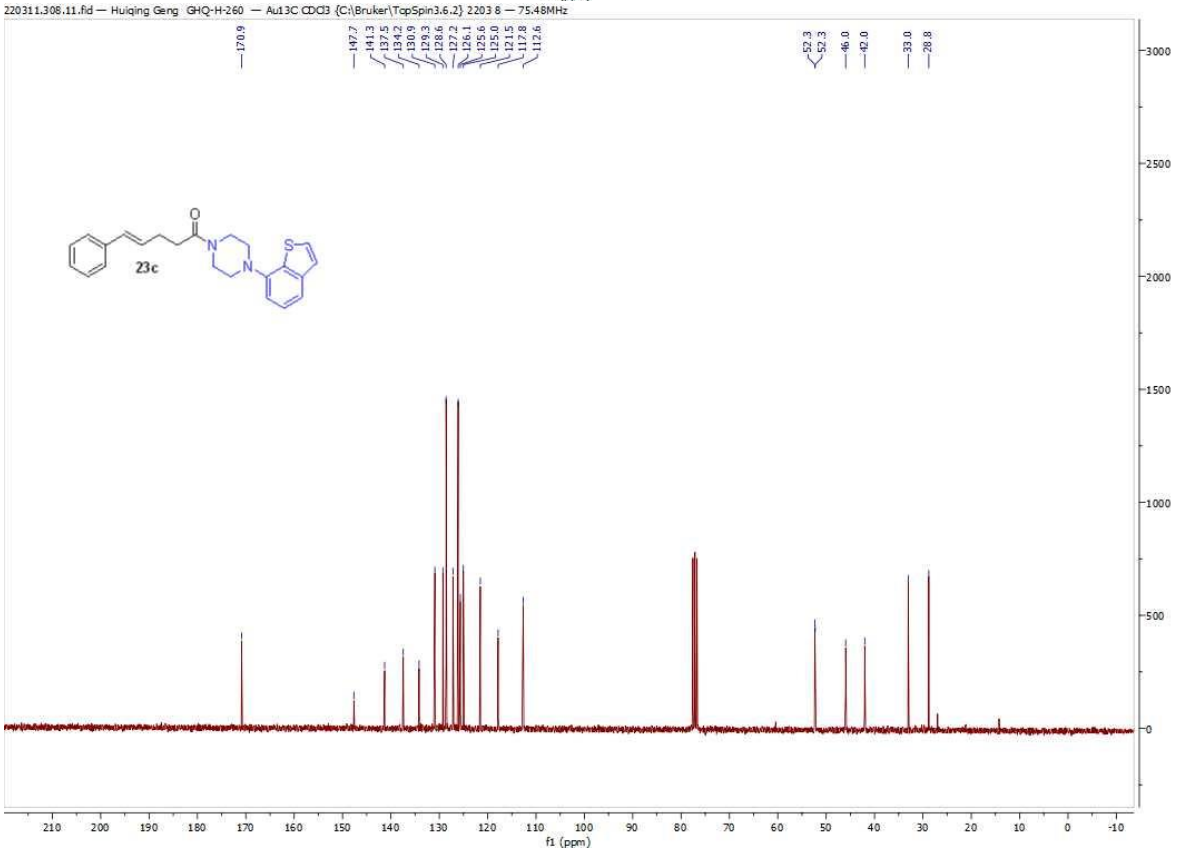

220311.306.10.fid — Huiqing Geng GHQ-H-261 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 6 — 300.13MHz
21c
f1 (ppm)

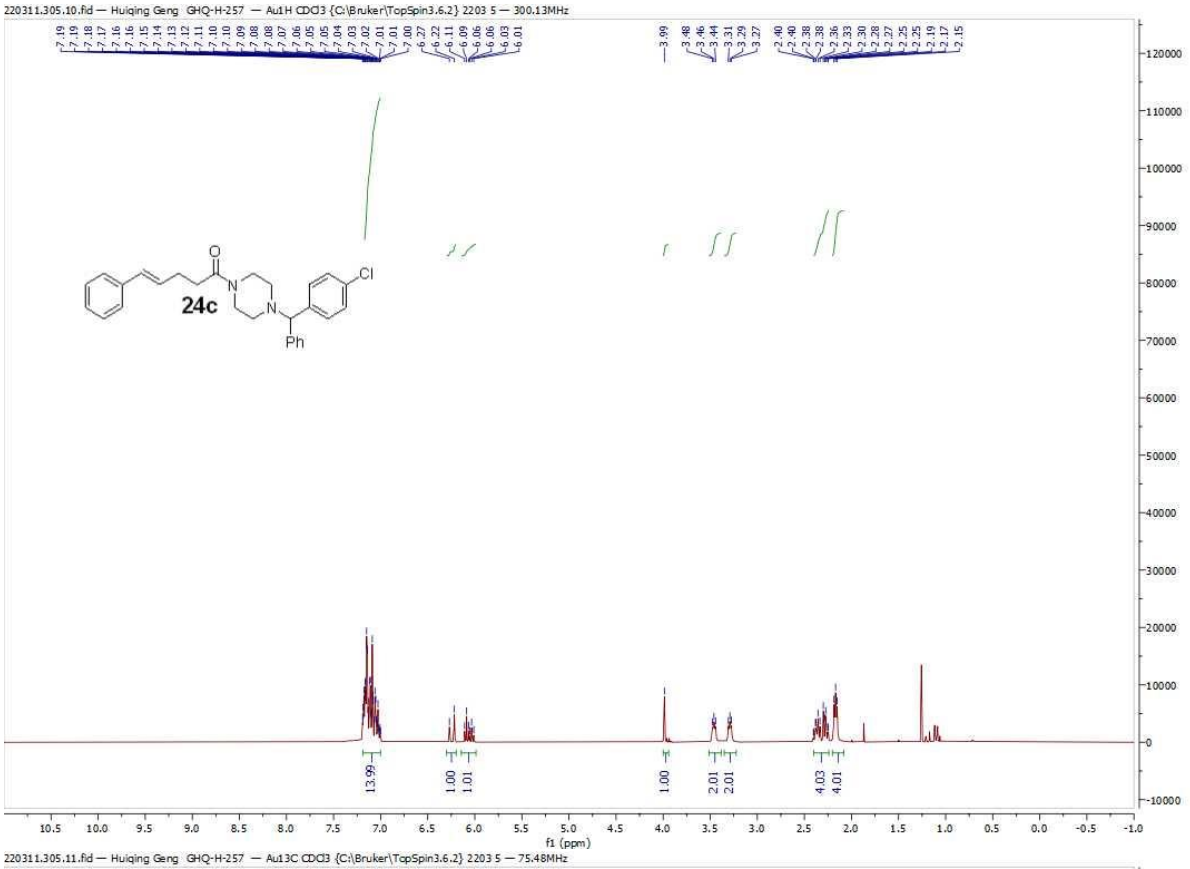

220311.306.11.fid — Huiqing Geng GHQ-H-261 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 6 — 75.48MHz
21c
f1 (ppm)

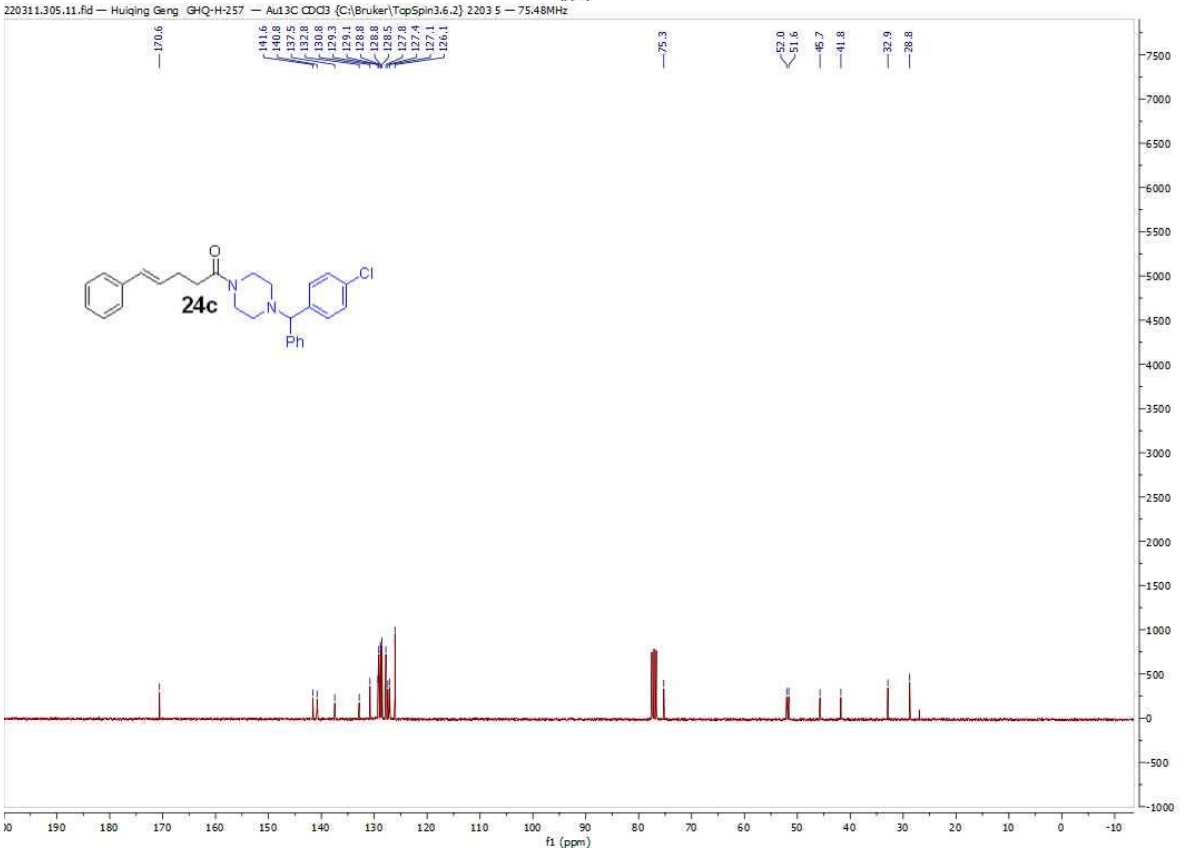

220311.311.10.fid — Huiqing Geng GHQ-H-259 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 11 — 300.13MHz
22c
f1 (ppm)

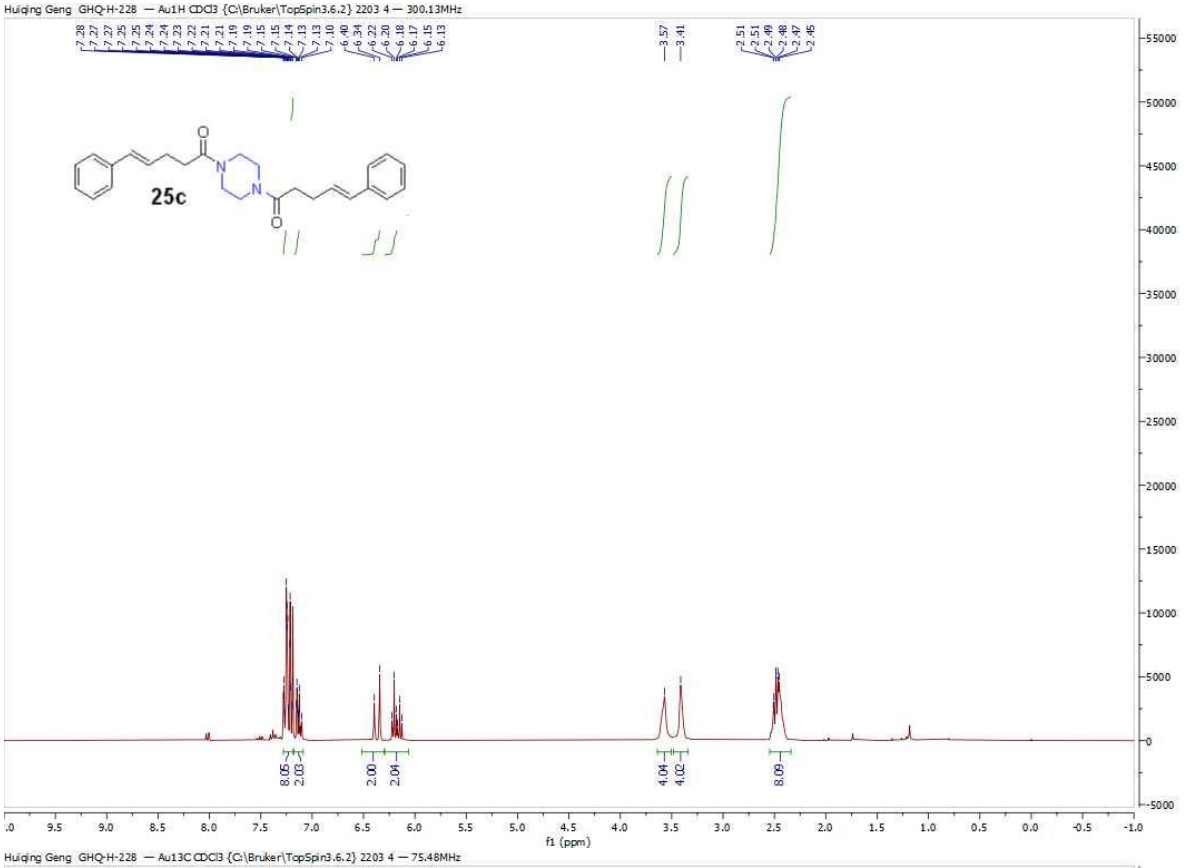

220311.311.11.fid — Huiqing Geng GHQ-H-259 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 11 — 75.48MHz
22c
f1 (ppm)

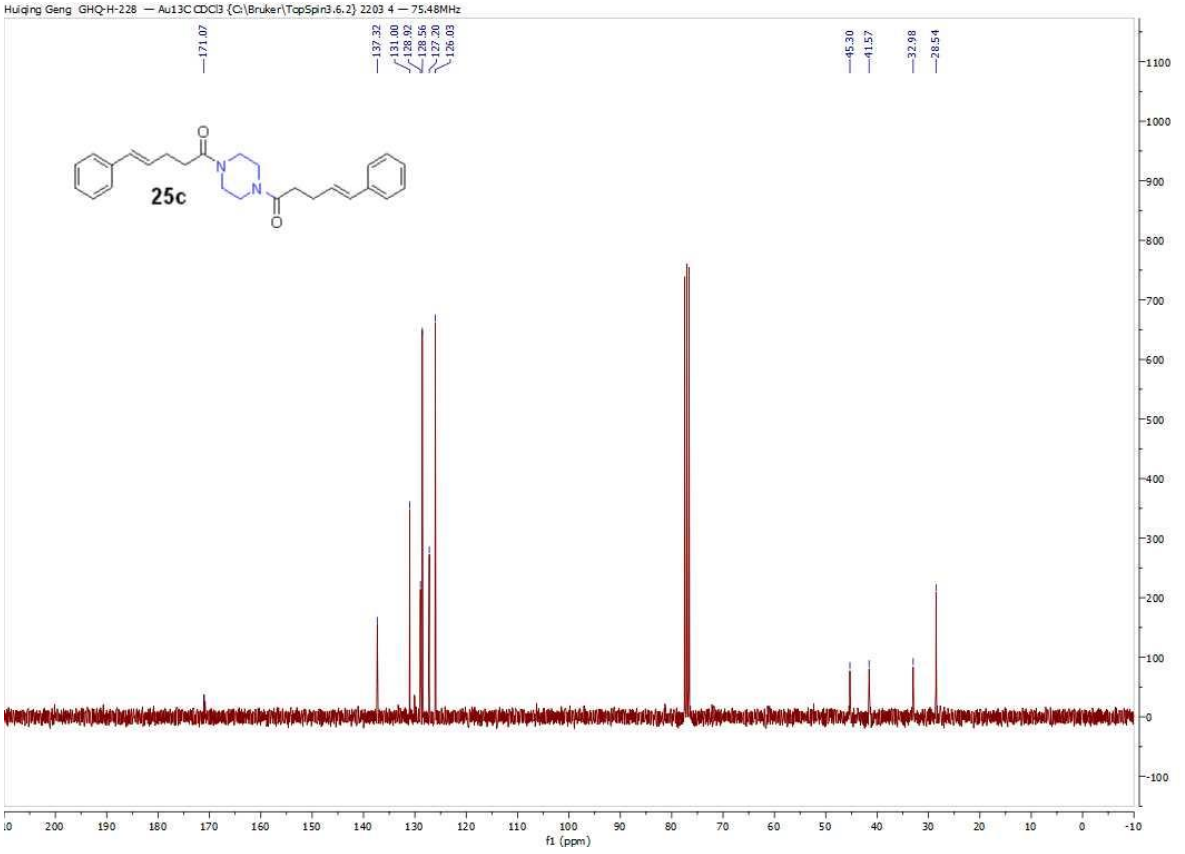

220311.308.10.fid — Huiqing Geng GHQ-H-260 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 8 — 300.13MHz
23c
f1 (ppm)

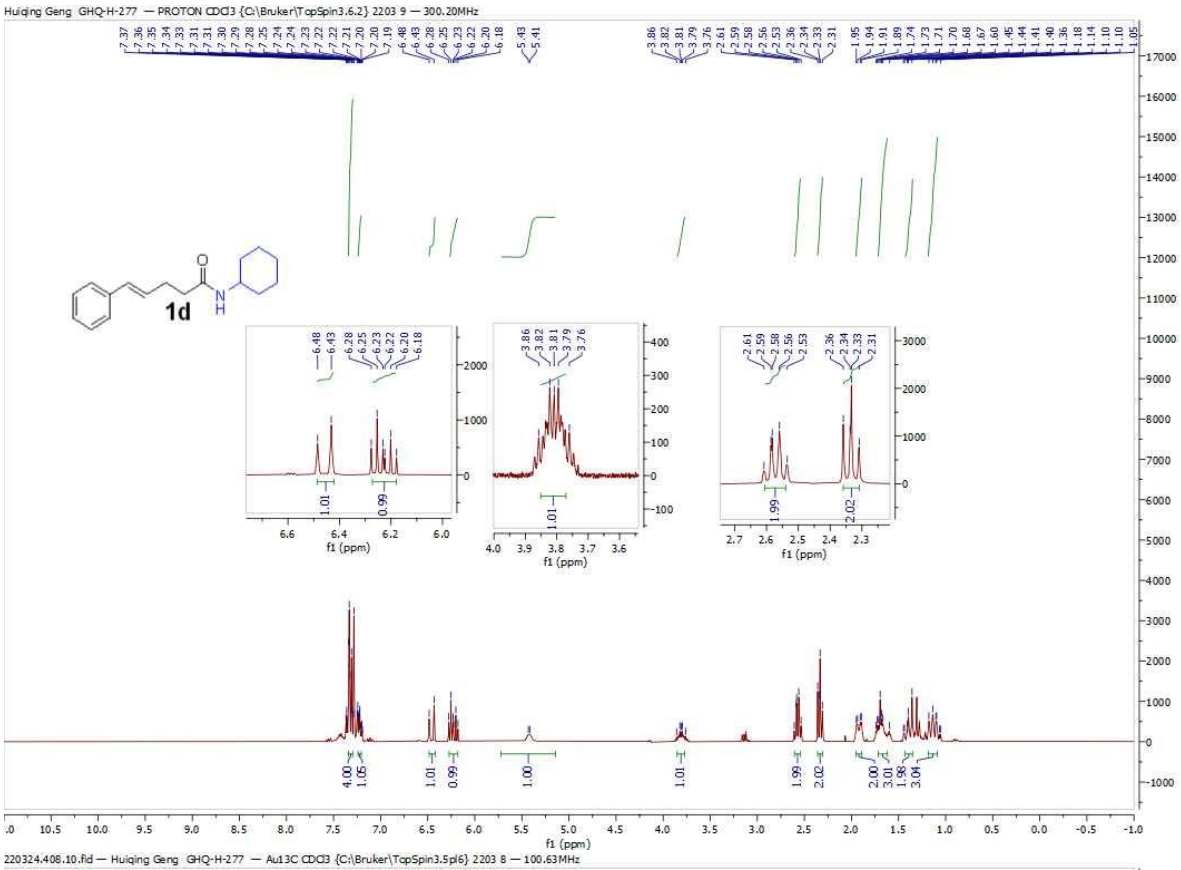

220311.308.11.fid — Huiqing Geng GHQ-H-260 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 8 — 75.48MHz
23c
f1 (ppm)

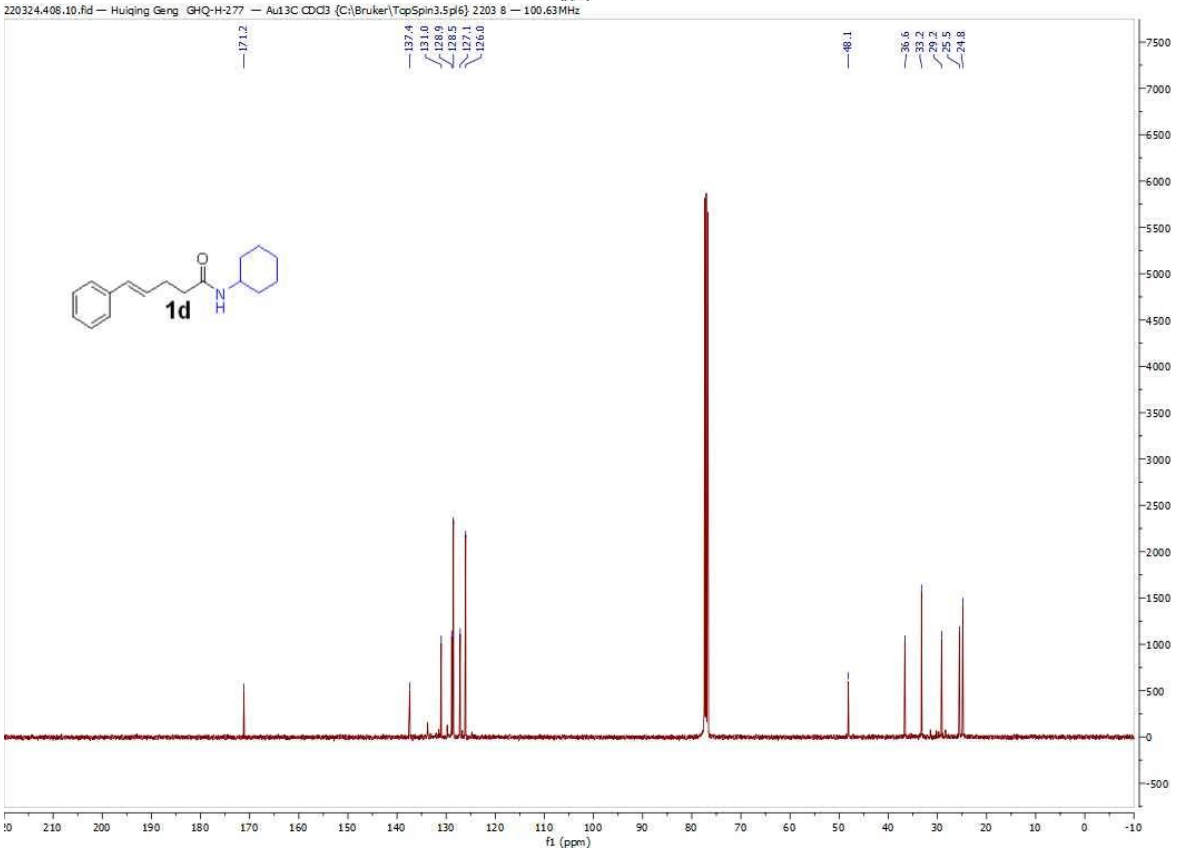

220311.305.10.fid — Huiqing Geng GHQ-H-257 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 5 — 300.13MHz
24c
Cl
Ph
f1 (ppm)

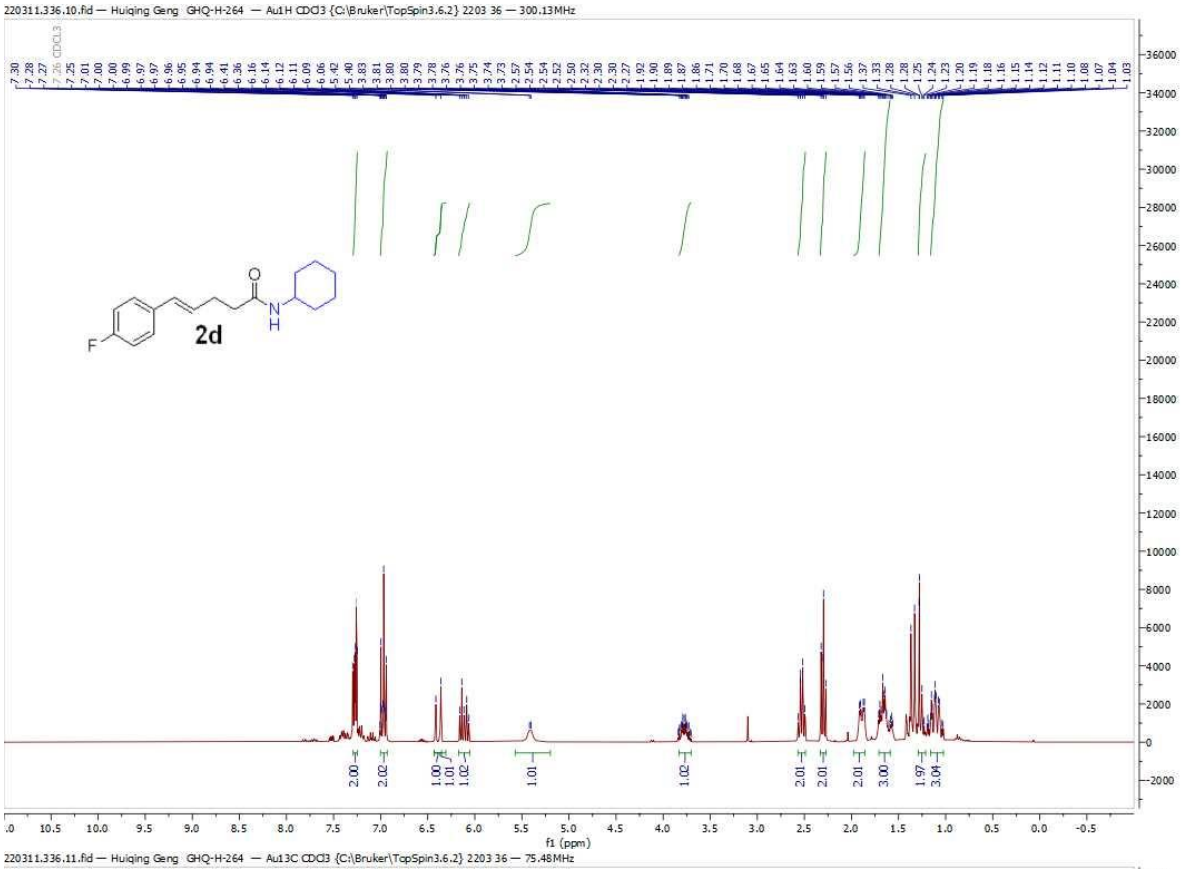

220311.305.11.fid — Huiqing Geng GHQ-H-257 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 5 — 75.48MHz
24c
Cl
Ph
f1 (ppm)

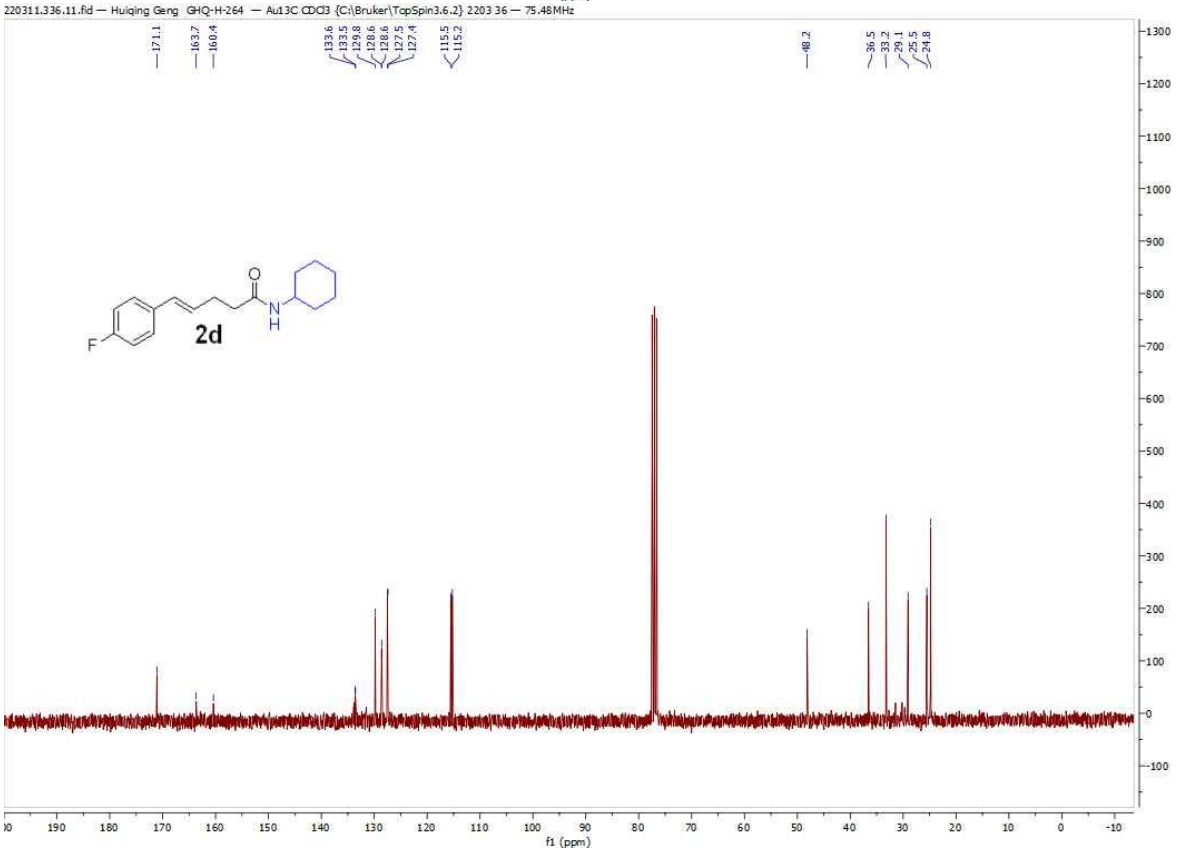

Huiqing Geng GHQ-H-228 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 4 — 300.13MHz
25c
f1 (ppm)

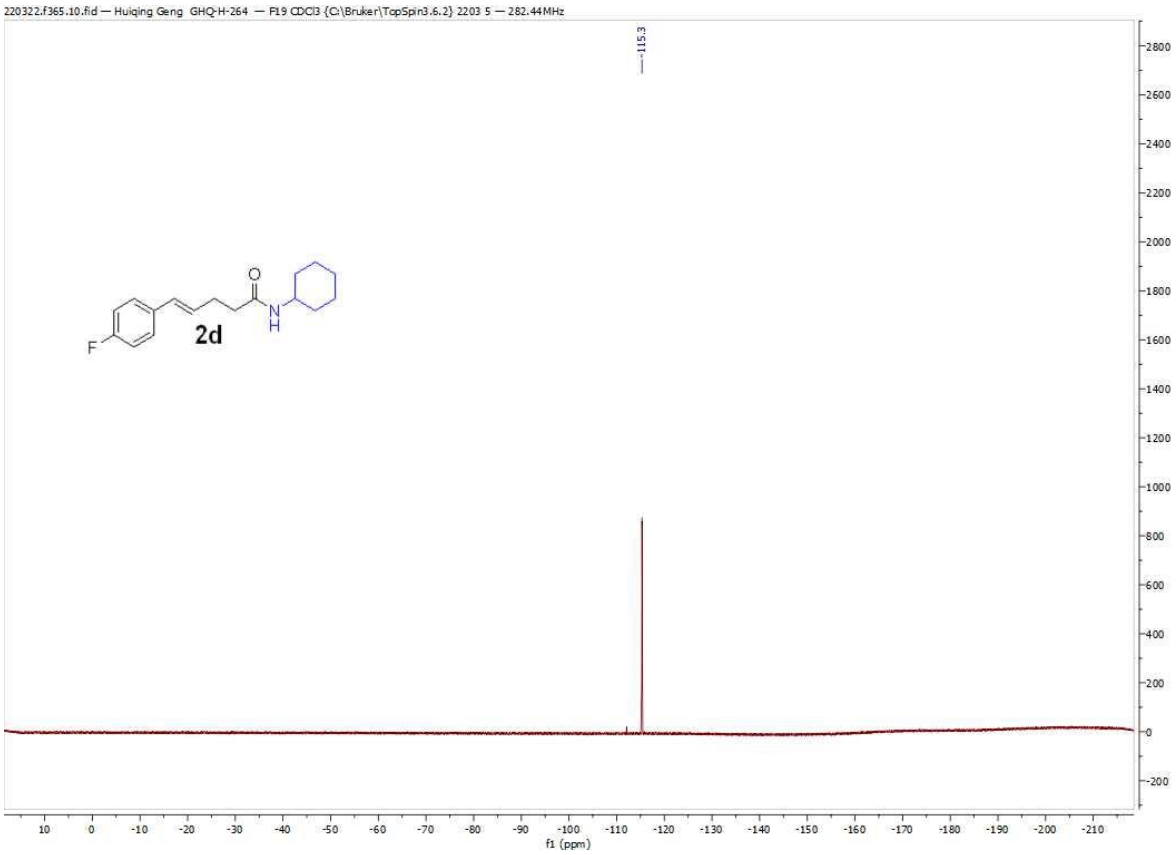

Huiqing Geng GHQ-H-228 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 4 — 75.48MHz
25c
f1 (ppm)

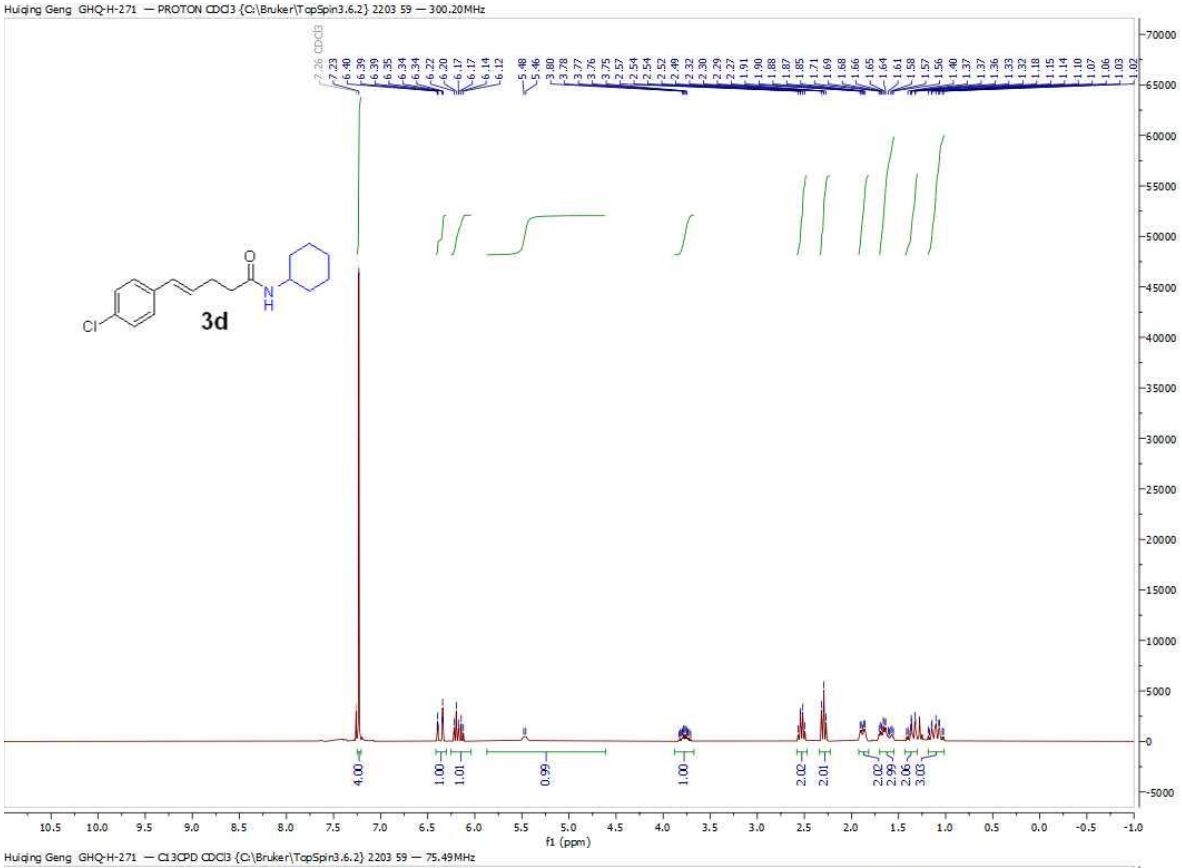

Huiqing Geng GHQ-H-277 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 9 — 300.20MHz
1d
f1 (ppm)

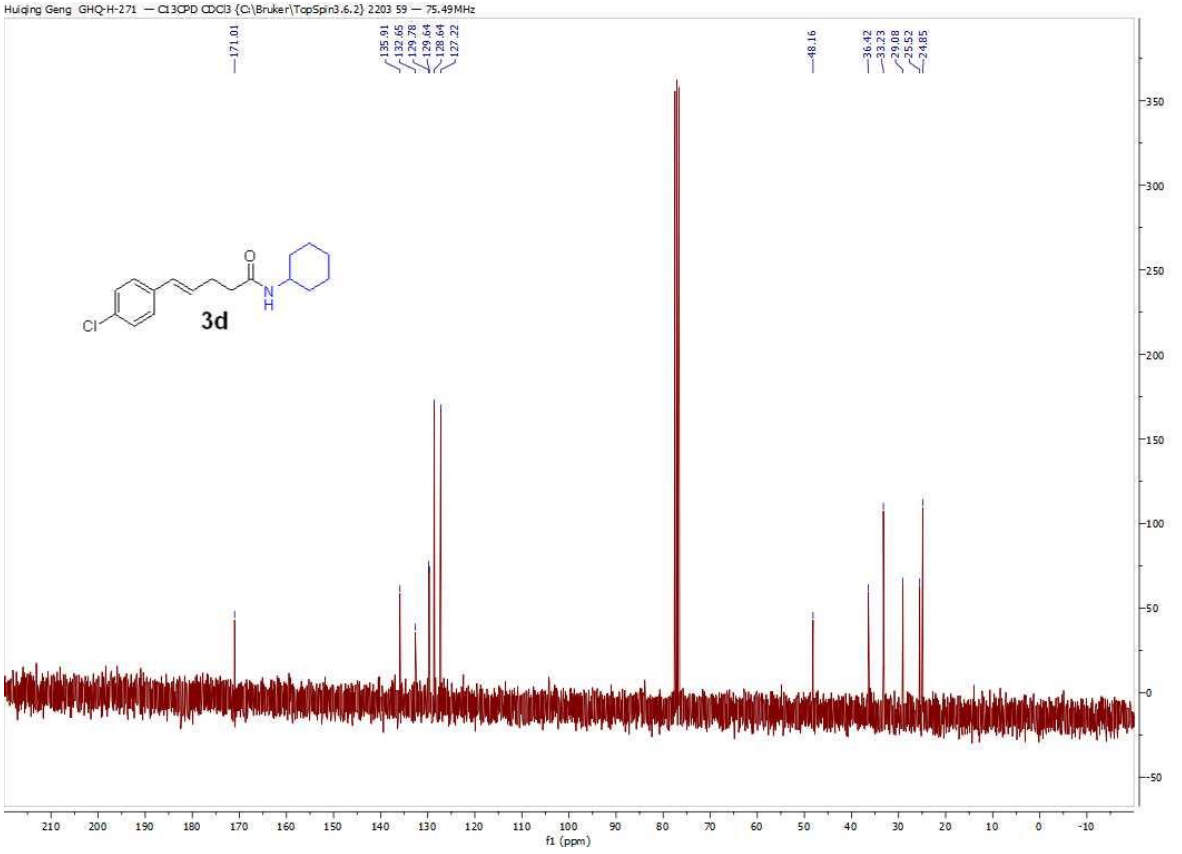

220324.408.10.fid — Huiqing Geng GHQ-H-277 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2203 8 — 100.63MHz
1d
f1 (ppm)

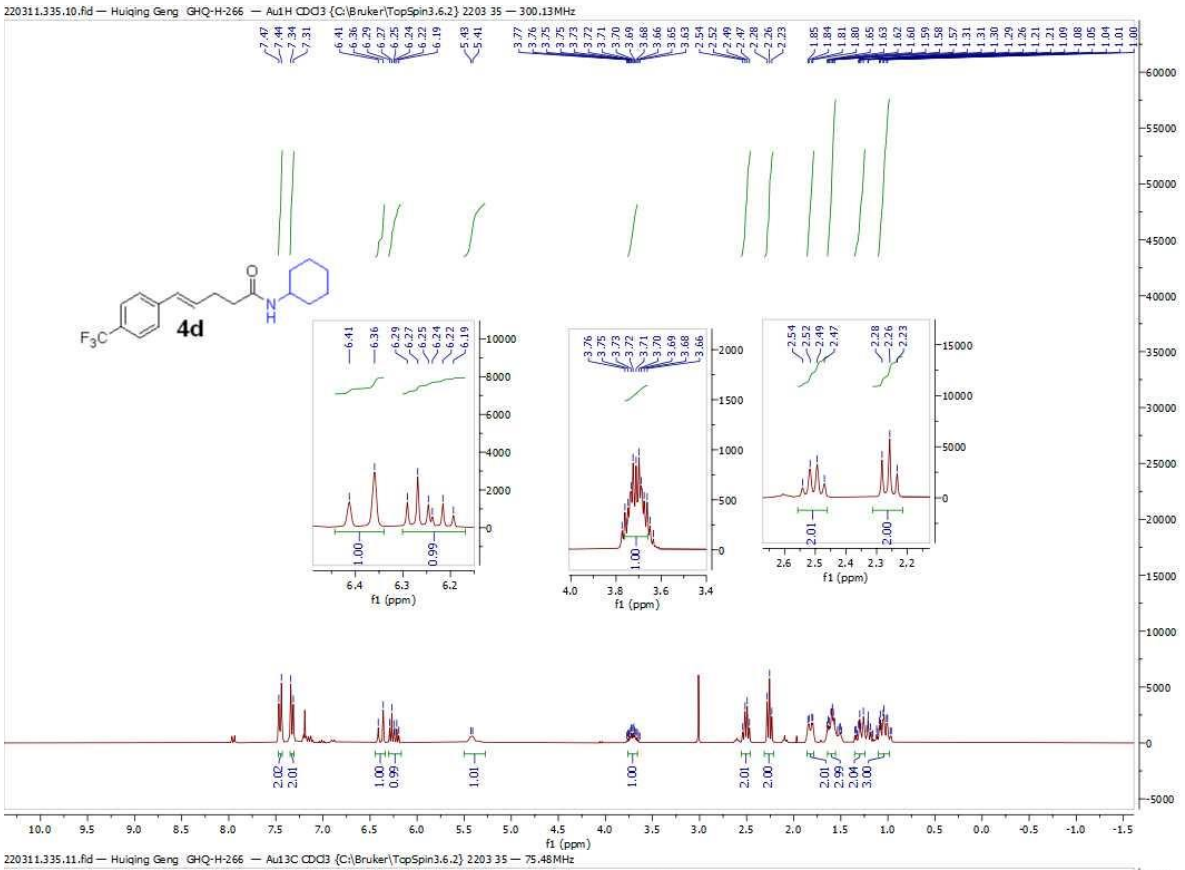

220311.336.10.fid — Huiqing Geng GHQ-H-264 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 36 — 300.13MHz
2d
f1 (ppm)

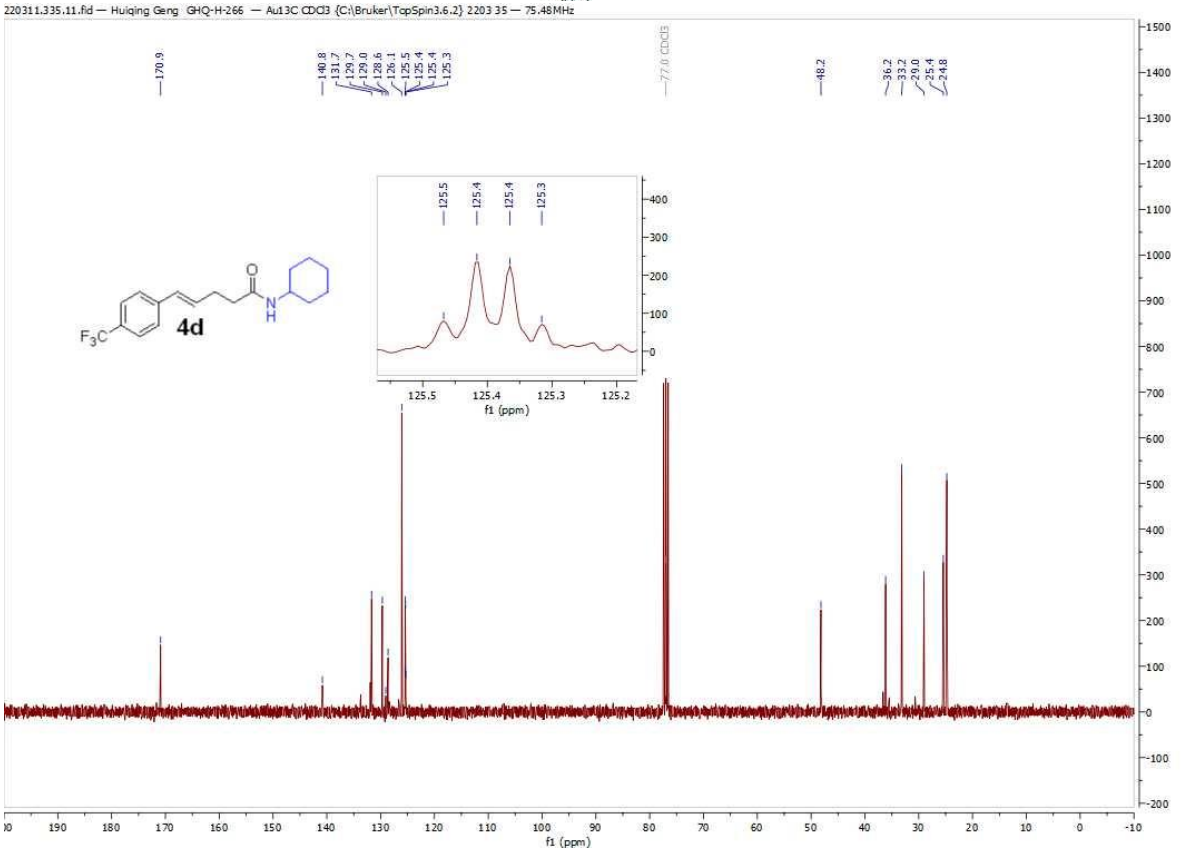

220311.336.11.fid — Huiqing Geng GHQ-H-264 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 36 — 75.48MHz
2d
f1 (ppm)

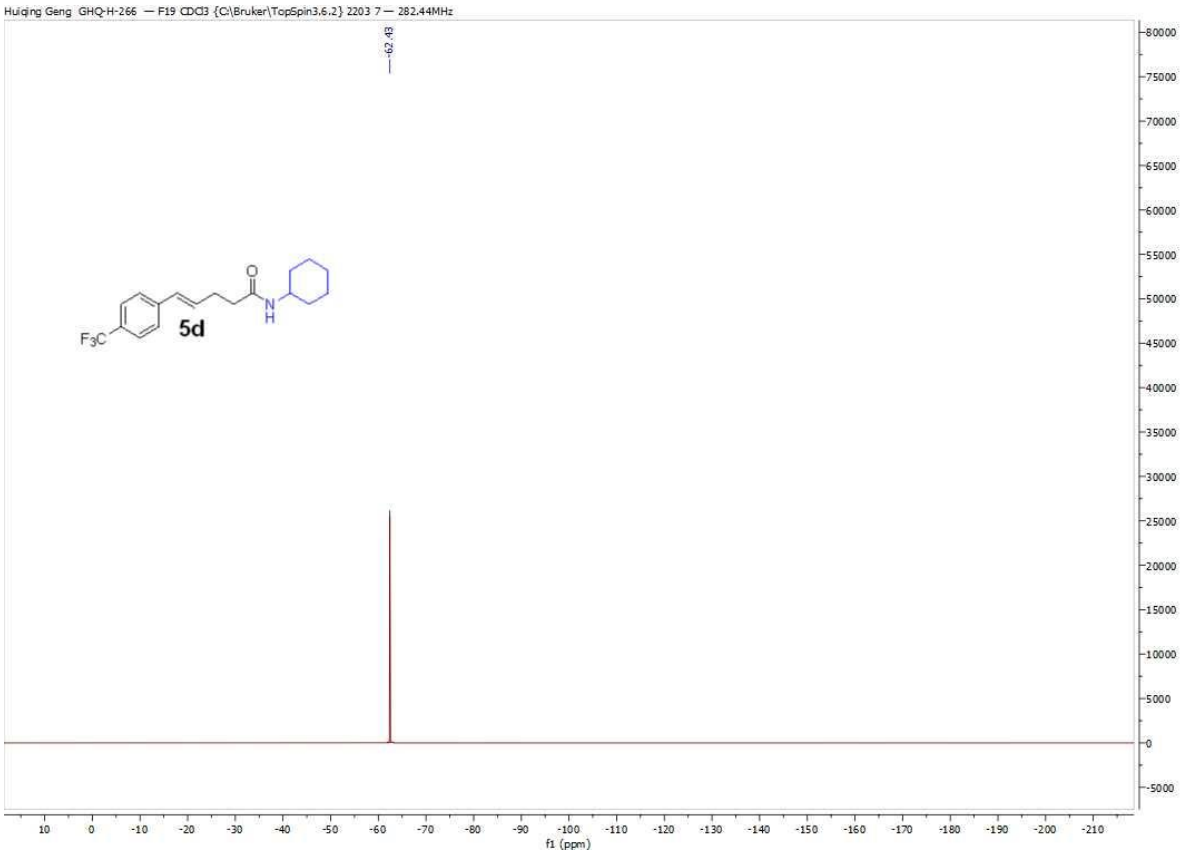

220322.f365.10.fid — Huiqing Geng GHQ-H-264 — F19 CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 5 — 282.44MHz
-115.3
2d
F
O
N
H
f1 (ppm)

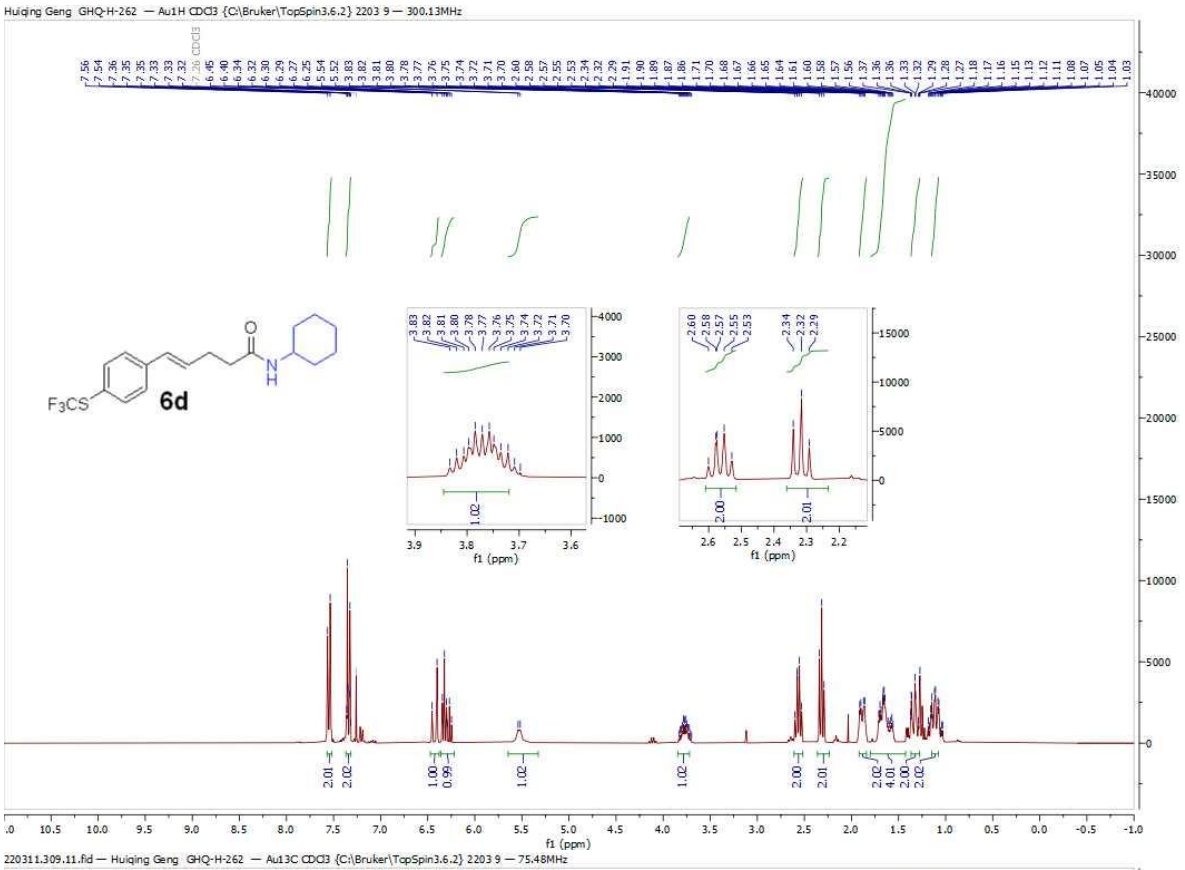

Huiqing Geng GHQ-H-271 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 59 — 300.20MHz
3d
f1 (ppm)

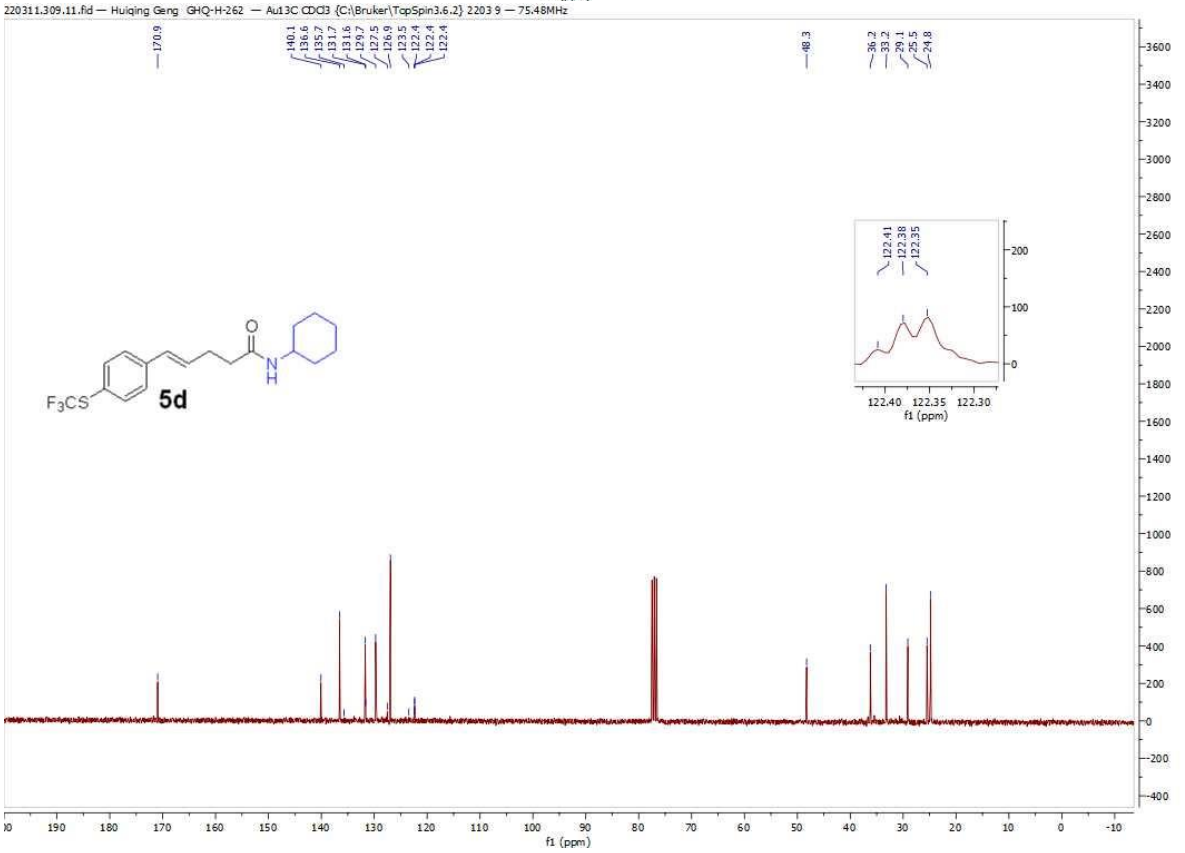

Huiqing Geng GHQ-H-271 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 59 — 75.49MHz
3d
f1 (ppm)

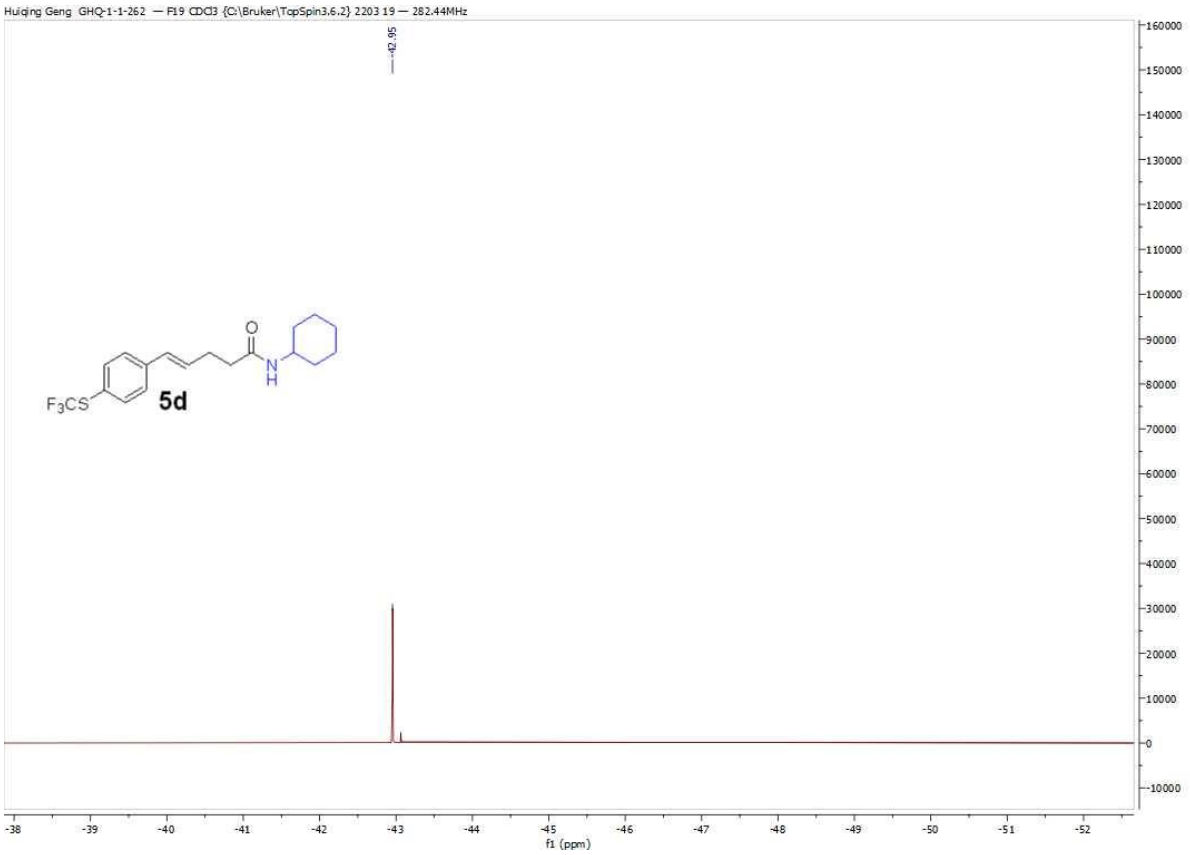

220311.335.10.fid — Huiqing Geng GHQ-H-266 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 35 — 300.13MHz
4d
f1 (ppm)

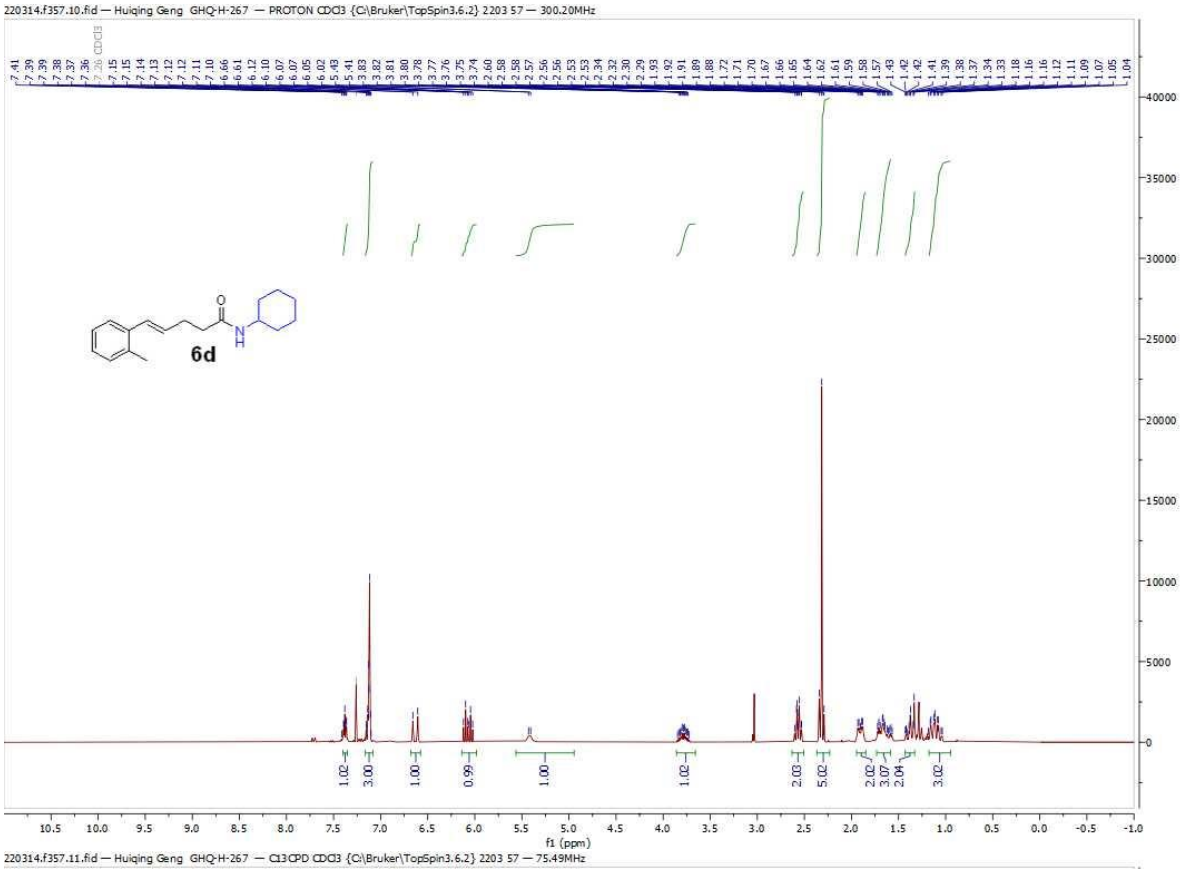

220311.335.11.fid — Huiqing Geng GHQ-H-266 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 35 — 75.48MHz
4d
f1 (ppm)

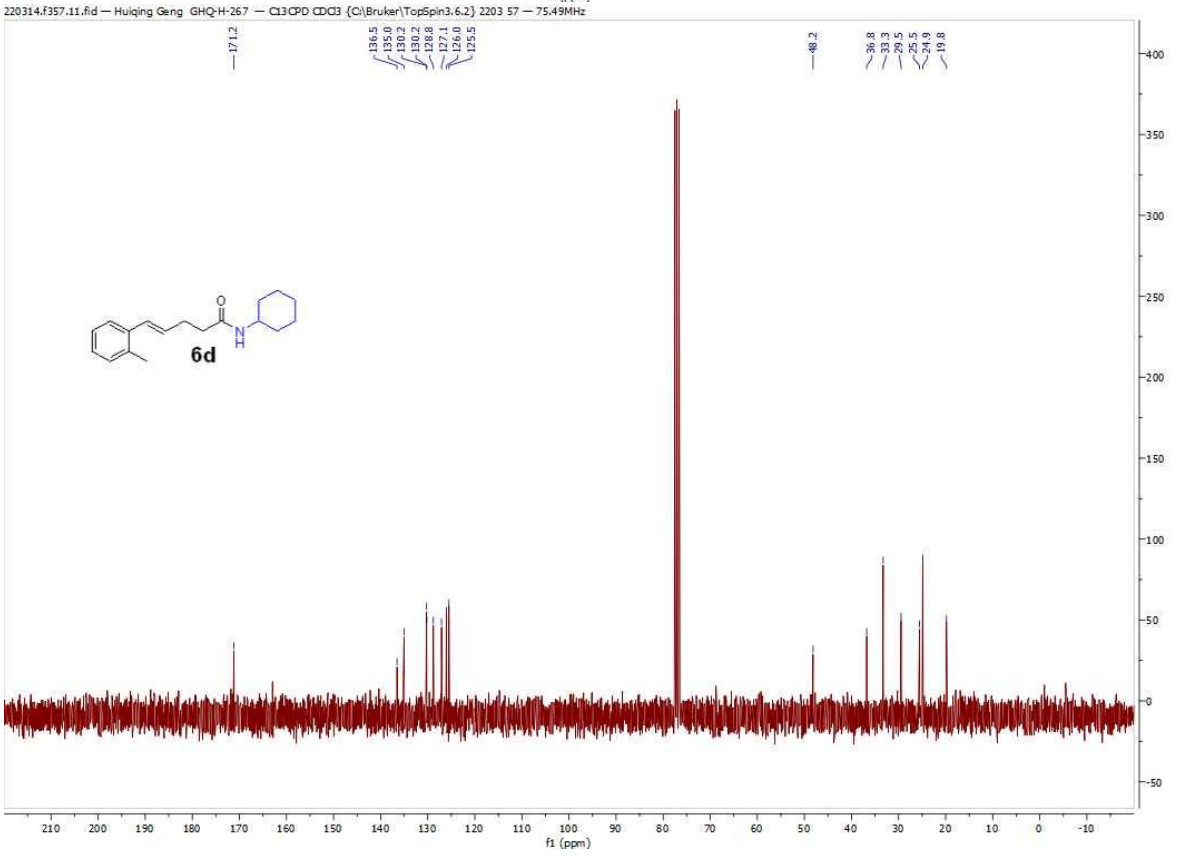

Huiqing Geng GHQ-H-266 — F19 CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 7 — 282.44MHz
-62.48
5d
F3C
O
N
H
f1 (ppm)

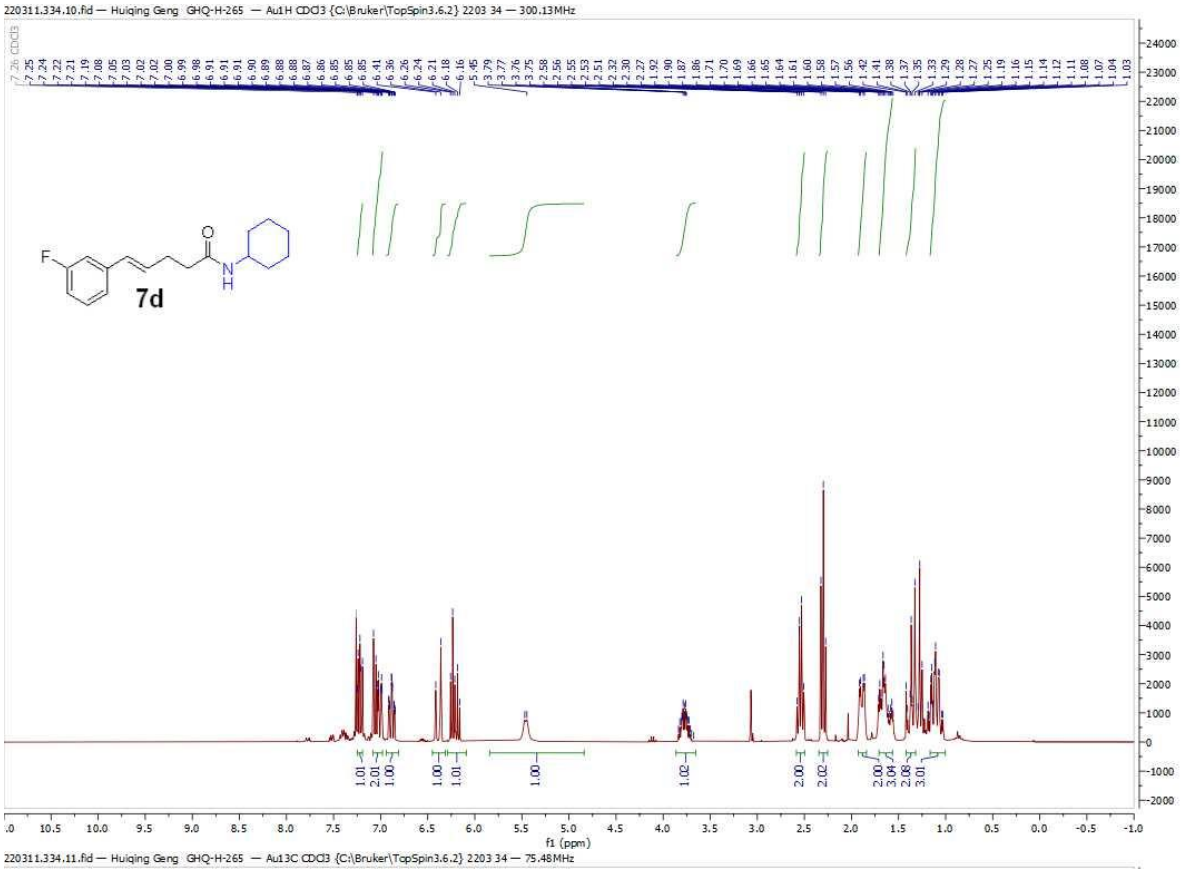

Huiqing Geng GHQ-H-262 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 9 — 300.13MHz
F3CS
6d
f1 (ppm)

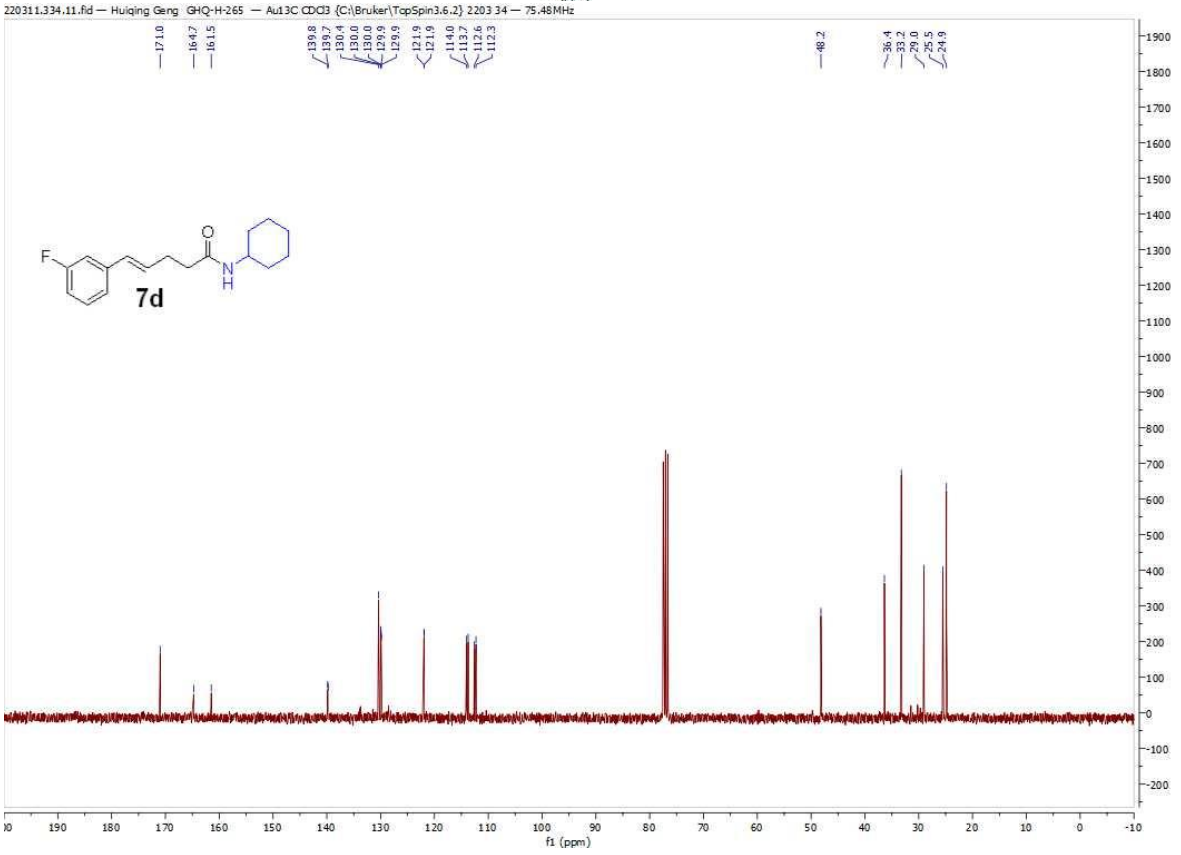

220311.309.11.fid — Huiqing Geng GHQ-H-262 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 9 — 75.48MHz
F3CS
5d
f1 (ppm)

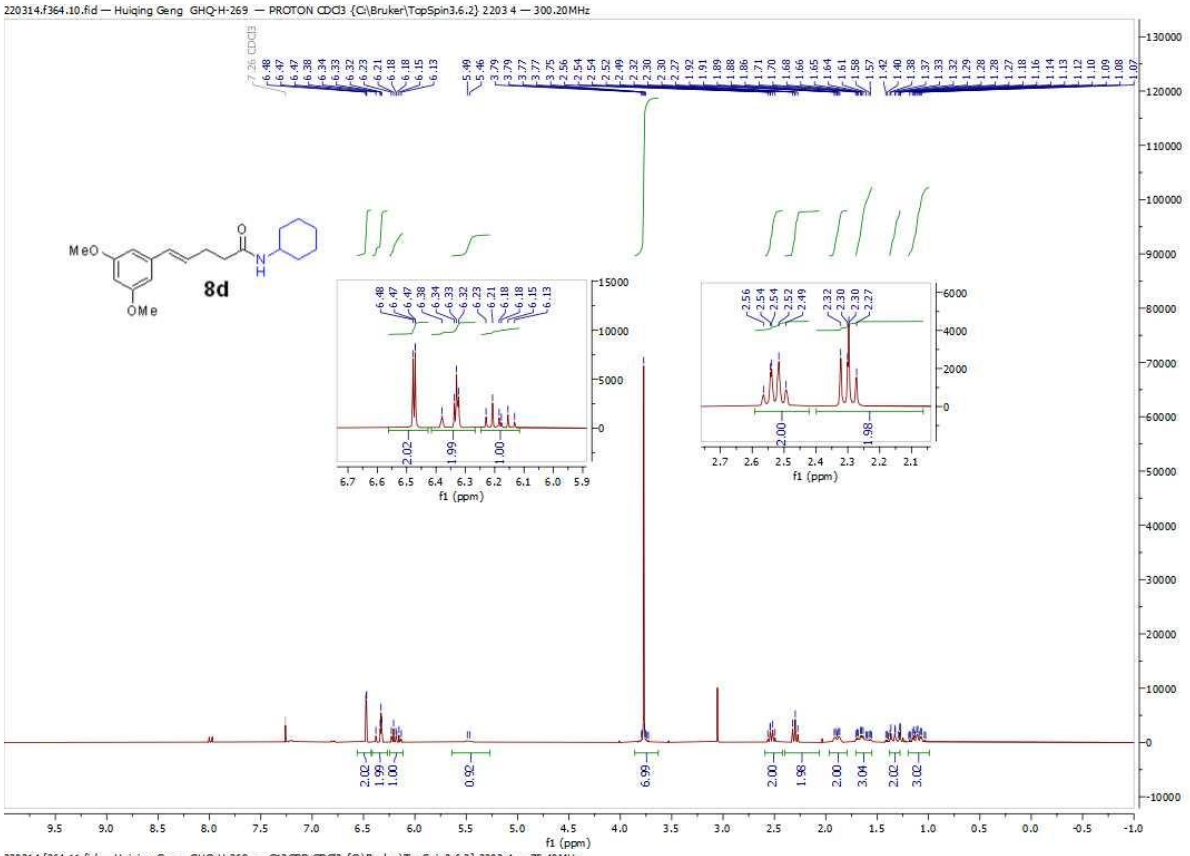

Huiqing Geng GHQ-1-1-262 — F19 CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 19 — 282.44MHz
-42.95
F3CS
5d
O
N
H
160000
150000
140000
130000
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
-38
-39
-40
-41
-42
-43
-44
-45
-46
-47
-48
-49
-50
-51
-52
f1 (ppm)

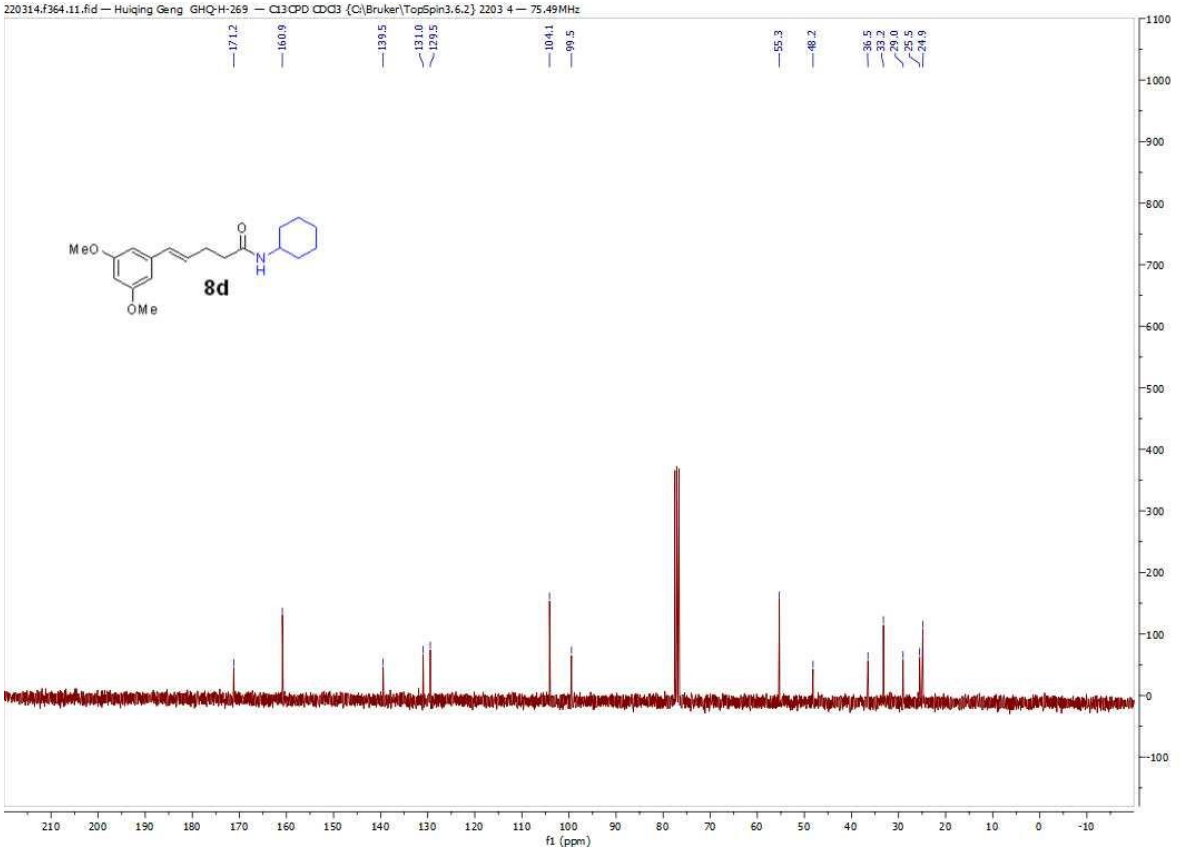

220314.f357.10.fid — Huiqing Geng GHQ-H-267 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 57 — 300.20MHz
6d
f1 (ppm)

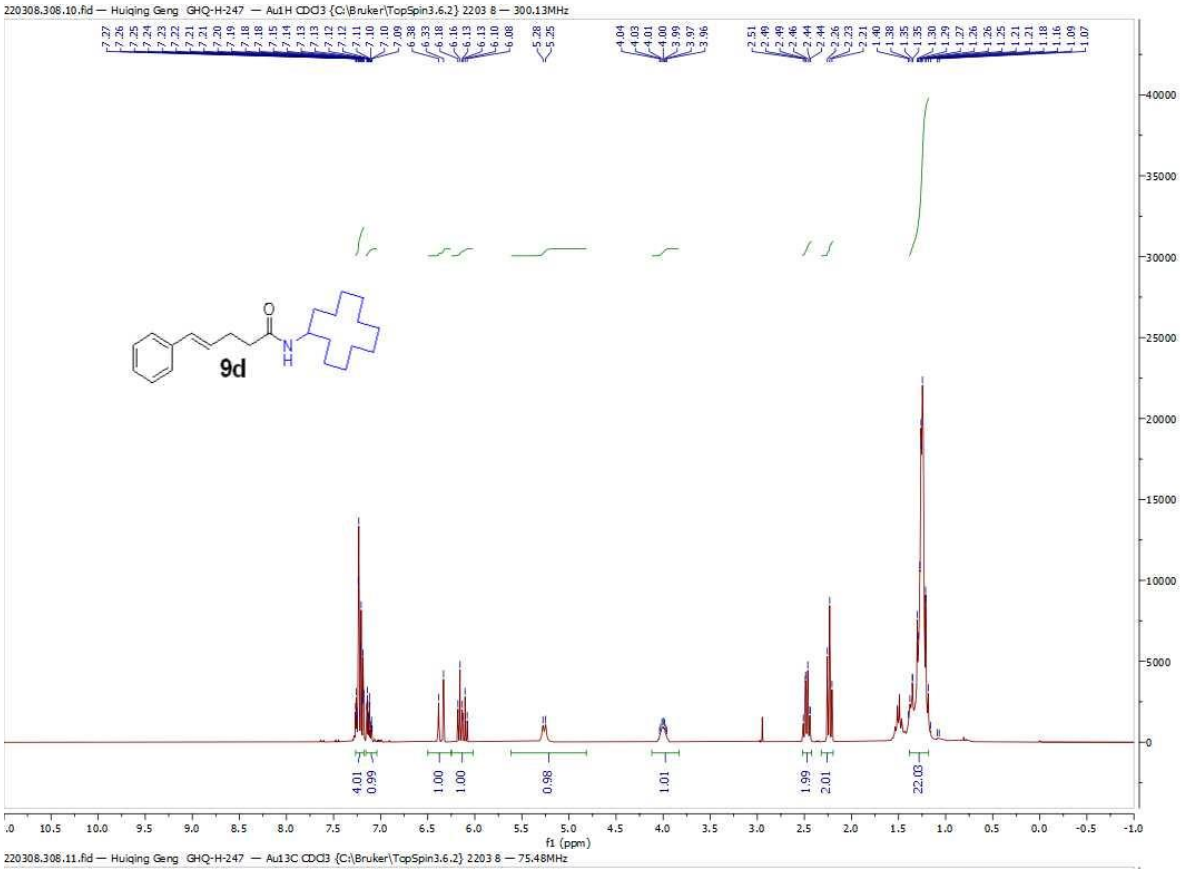

220314.f357.11.fid — Huiqing Geng GHQ-H-267 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 57 — 75.49MHz
6d
f1 (ppm)

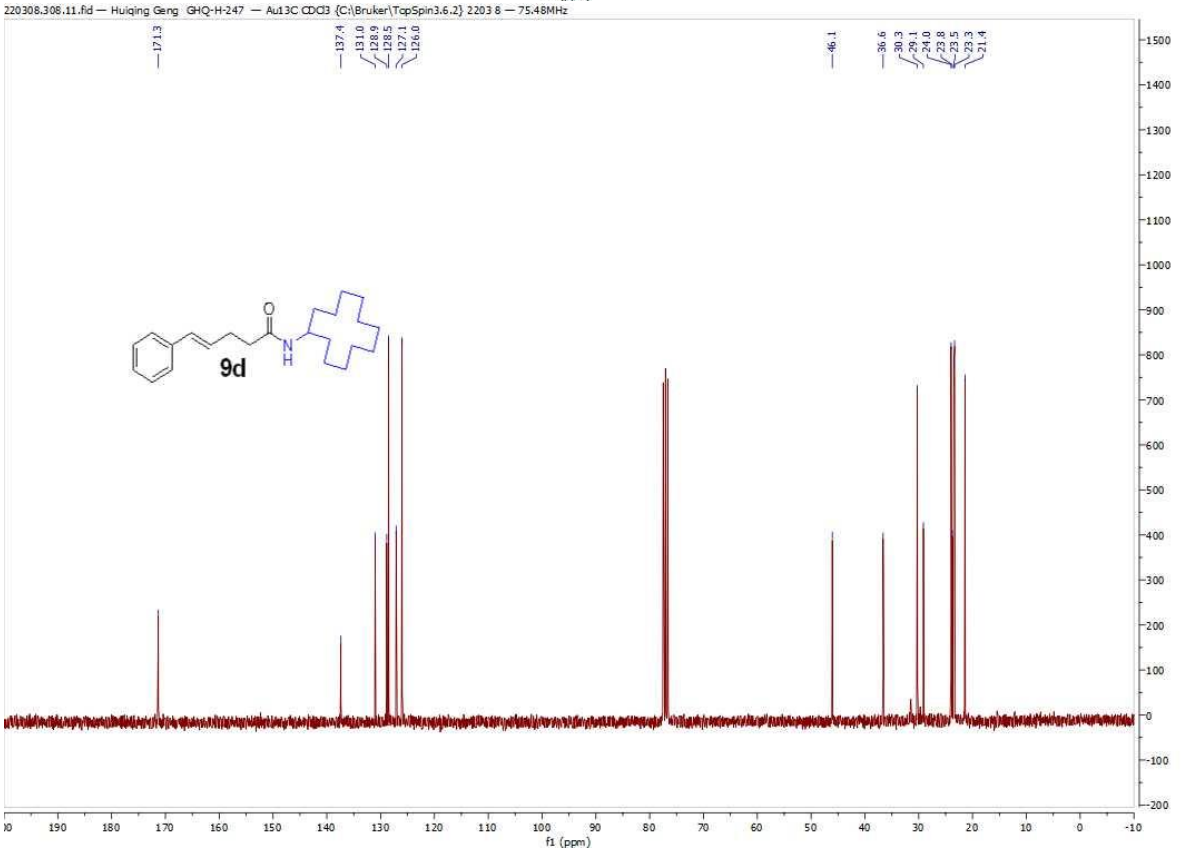

220311.334.10.fid — Huiqing Geng GHQ-H-265 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 34 — 300.13MHz
F
O
N
H
7d
f1 (ppm)

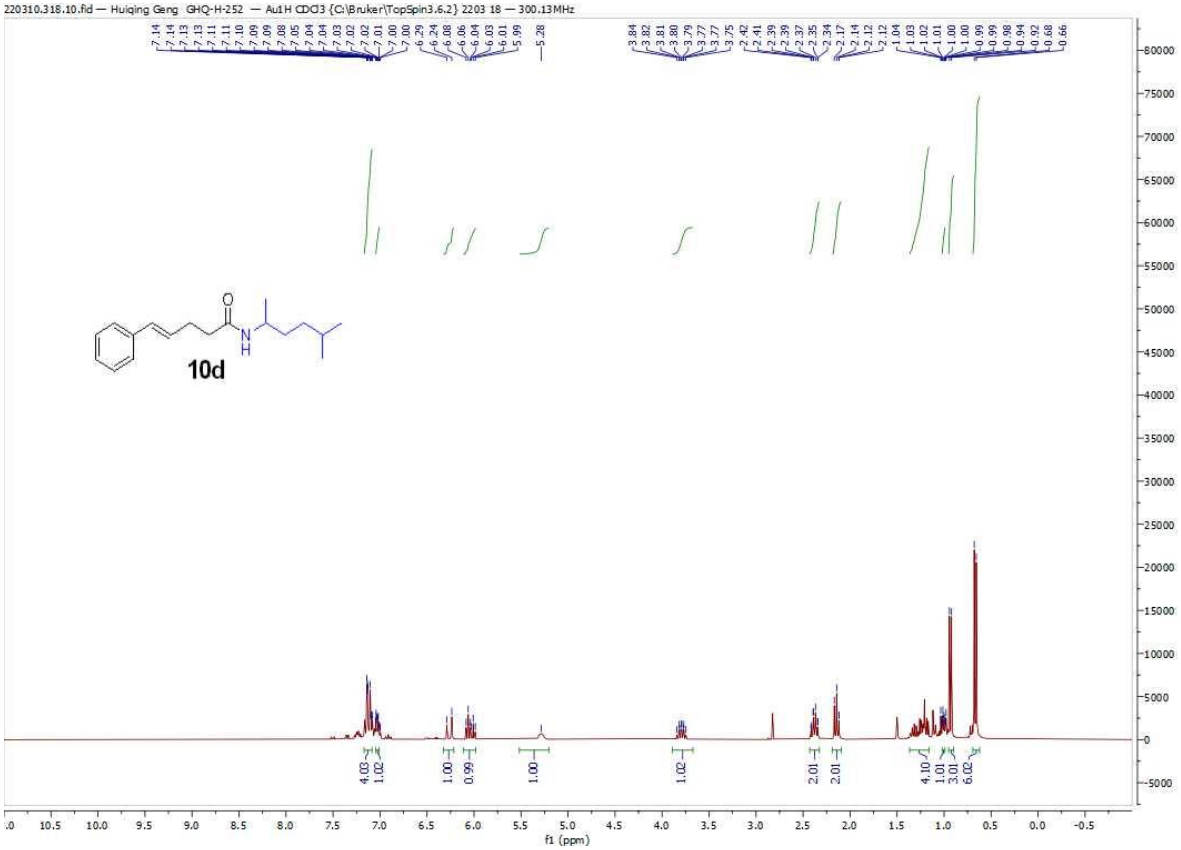

220311.334.11.fid — Huiqing Geng GHQ-H-265 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 34 — 75.48MHz
F
O
N
H
7d
f1 (ppm)

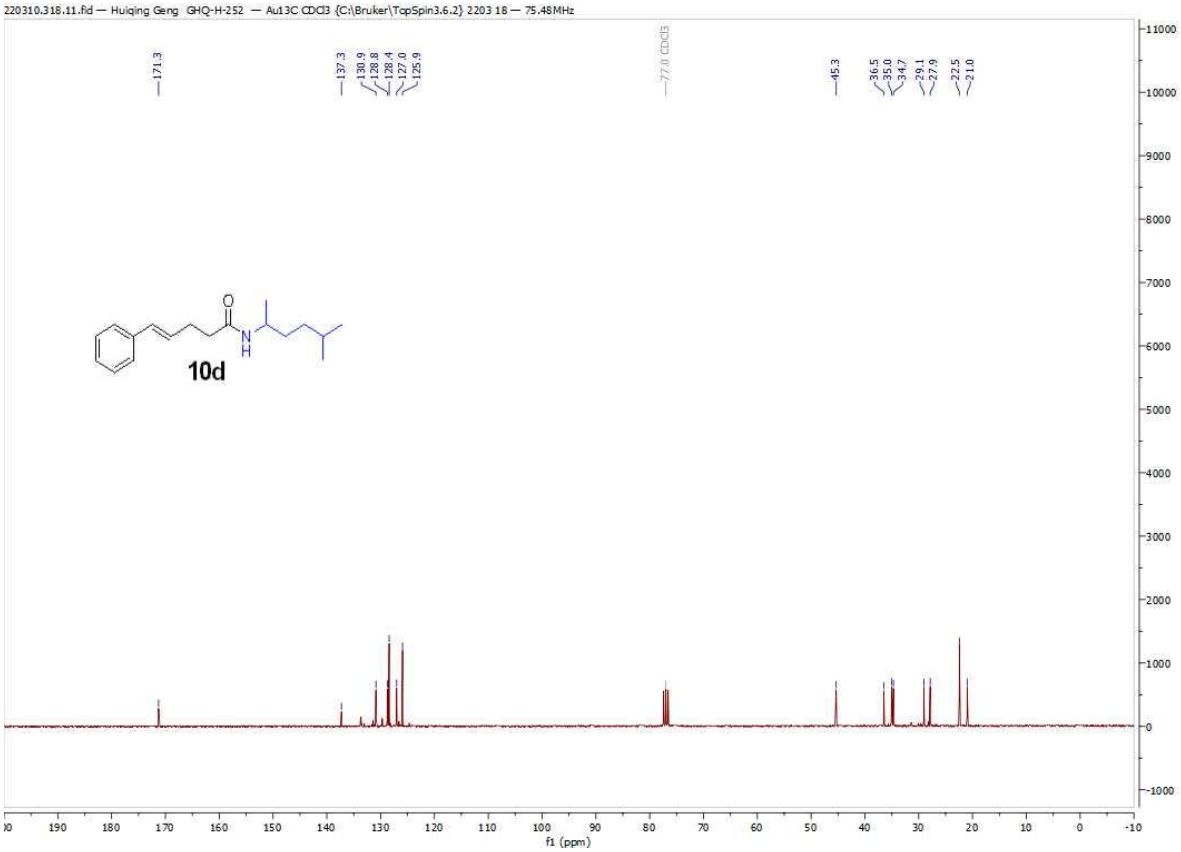

220314.f364.10.fid — Huiqing Geng GHQ-H-269 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 4 — 300.20MHz
MeO
OMe
N
H
O
8d
f1 (ppm)

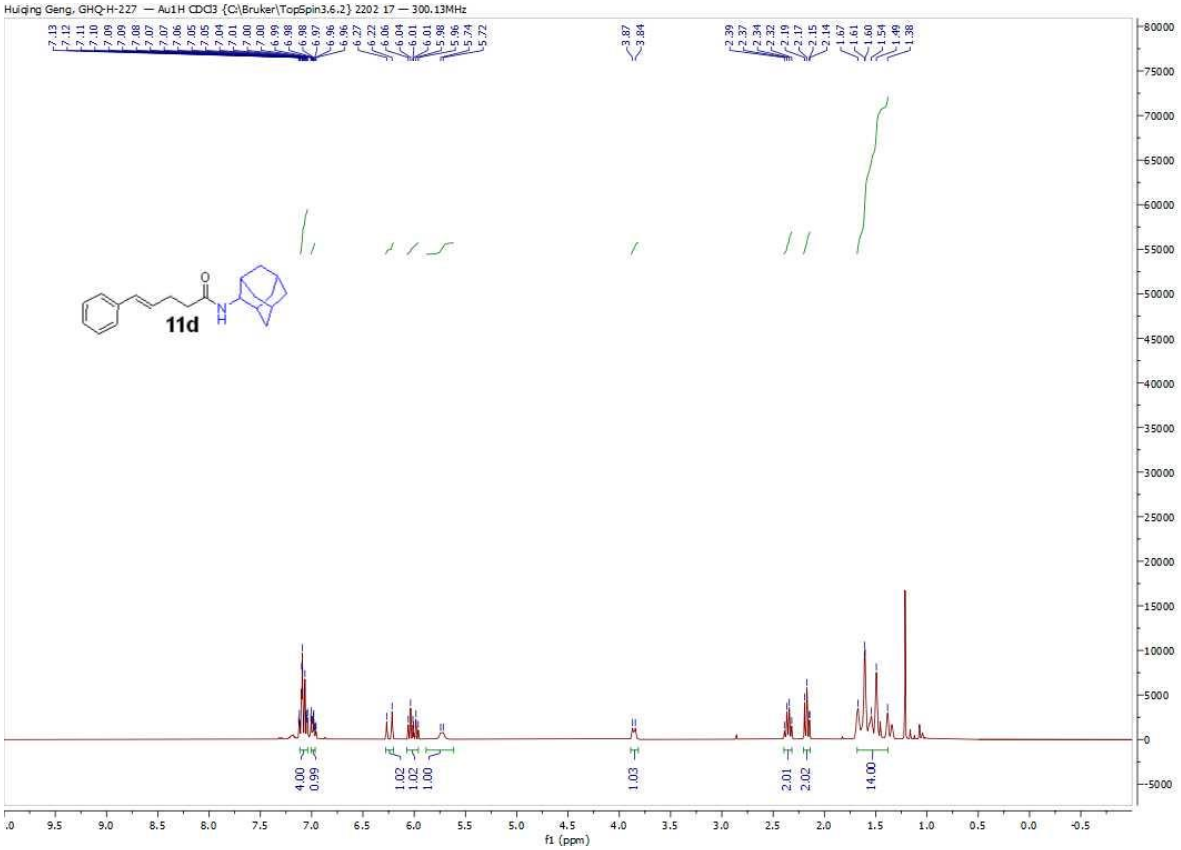

220314.f364.11.fid — Huiqing Geng GHQ-H-269 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 4 — 75.49MHz
MeO
OMe
N
H
O
8d
f1 (ppm)

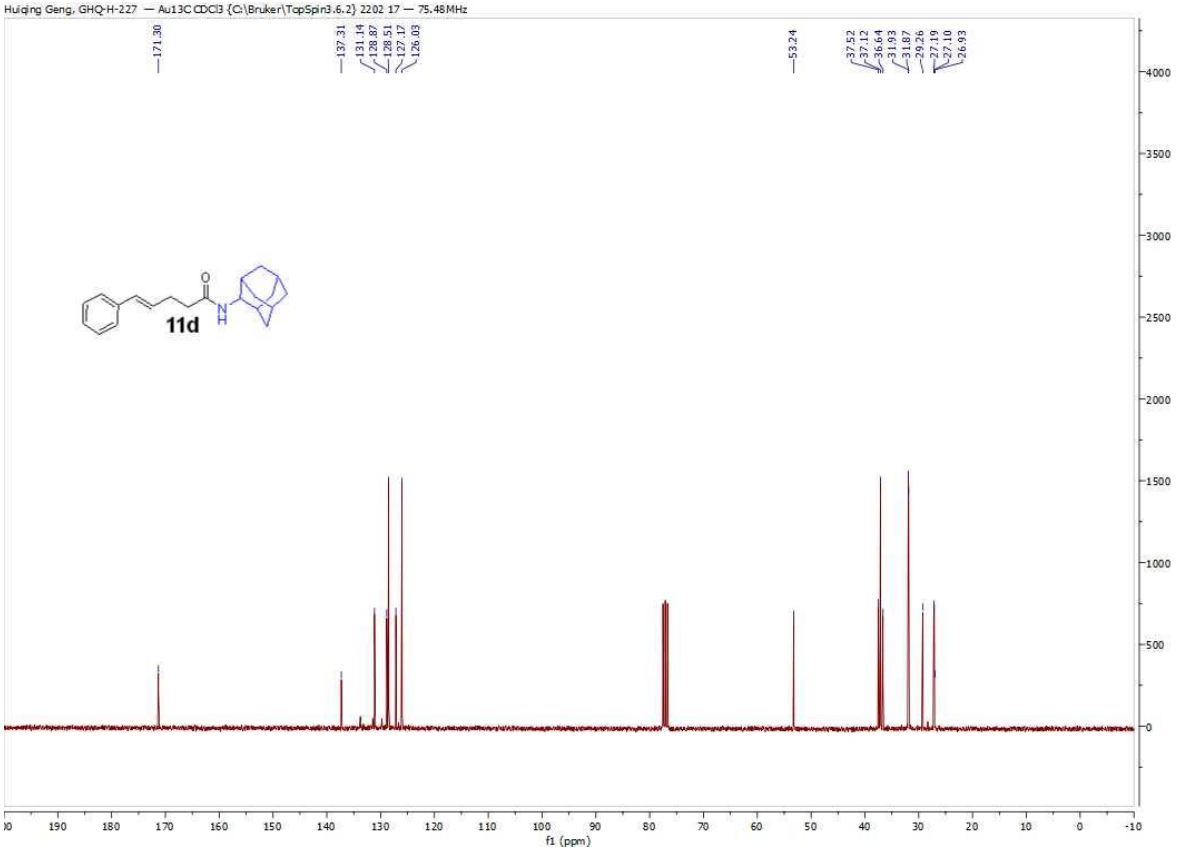

220308.308.10.fid — Huiqing Geng GHQ-H-247 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 8 — 300.13MHz
9d
f1 (ppm)

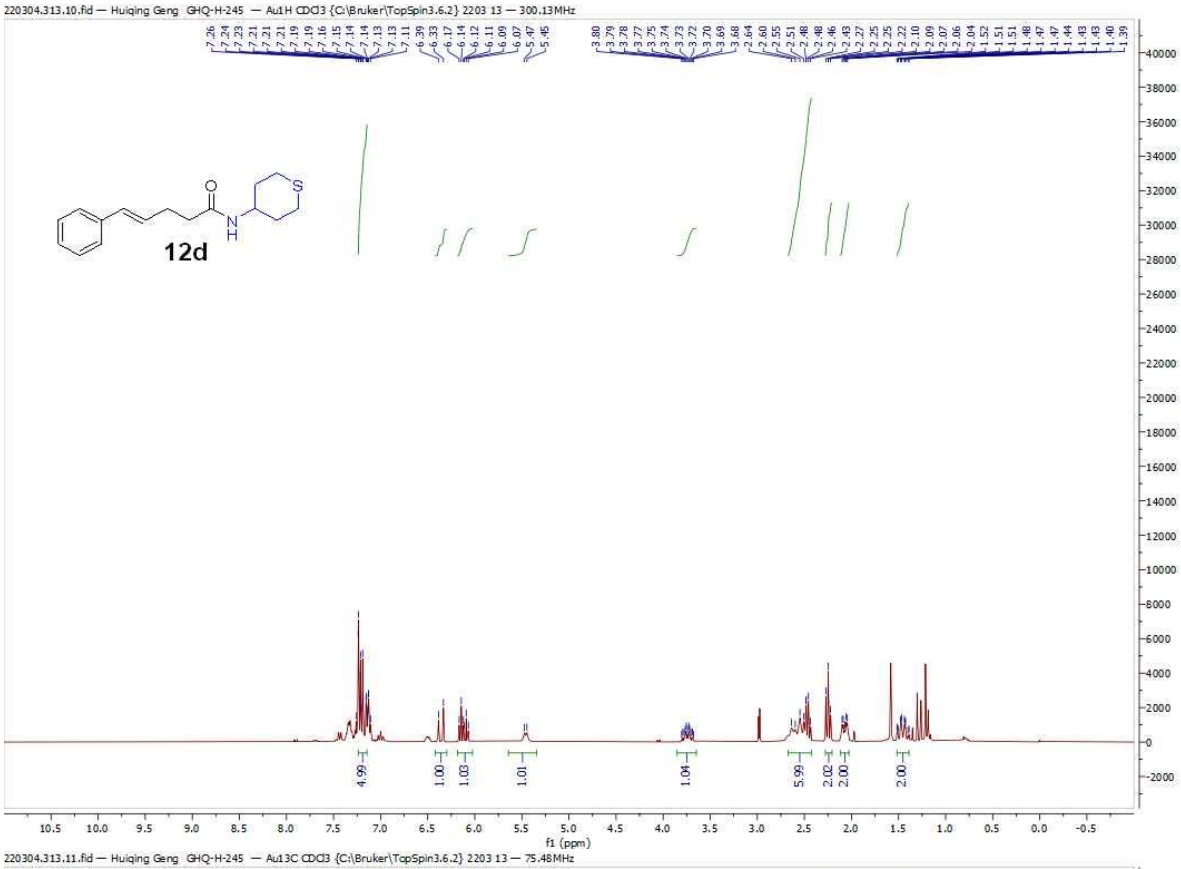

220308.308.11.fid — Huiqing Geng GHQ-H-247 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 8 — 75.48MHz
9d
f1 (ppm)

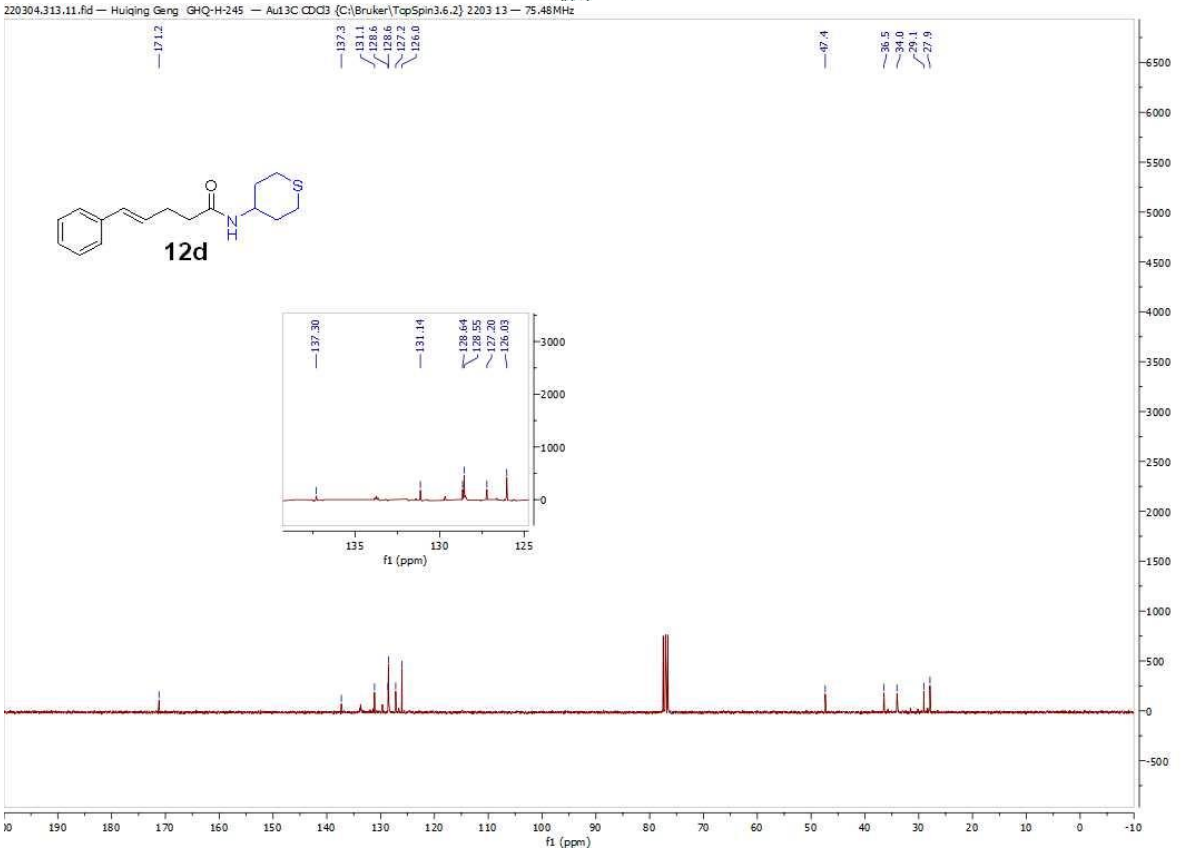

220310.318.10.fid — Huiqing Geng GHQ-H-252 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 18 — 300.13MHz
10d
f1 (ppm)

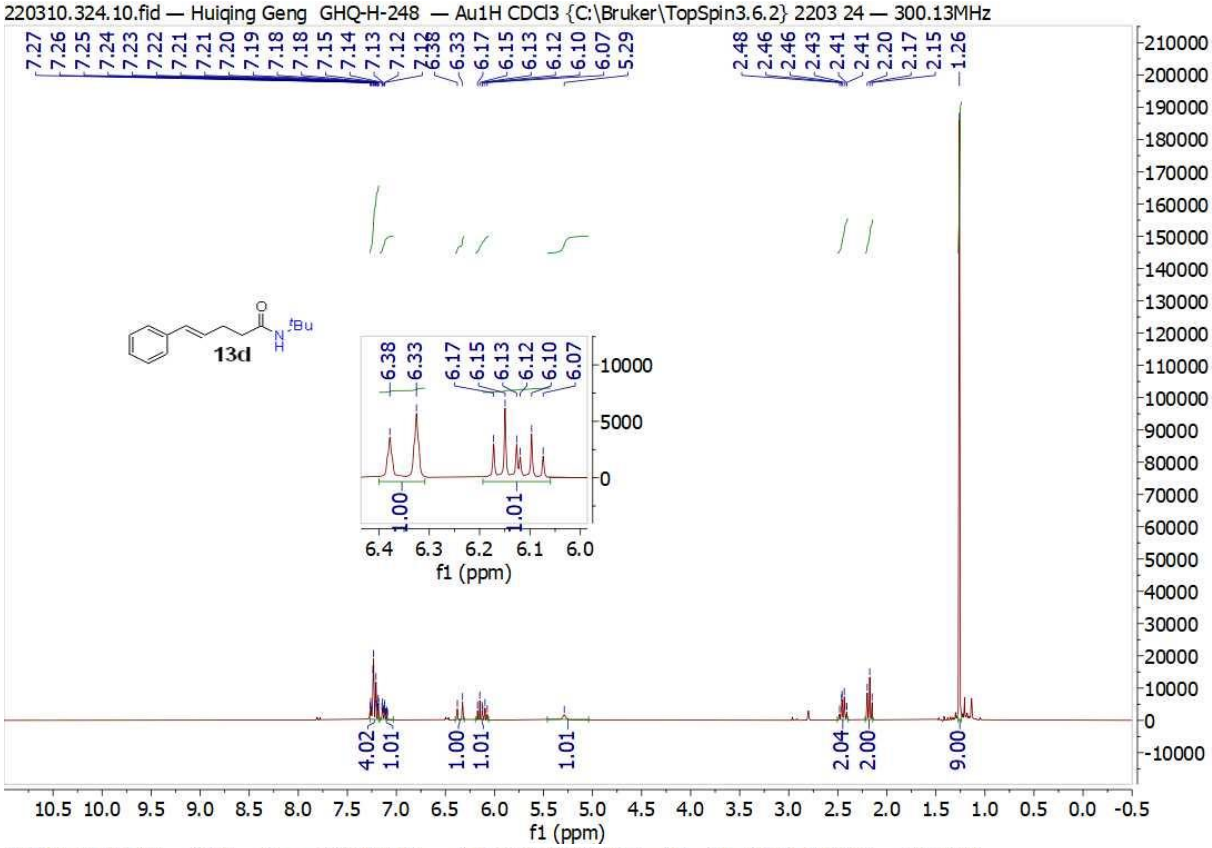

220310.318.11.fid — Huiqing Geng GHQ-H-252 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 18 — 75.48MHz
77.0 CDCl3
10d
f1 (ppm)

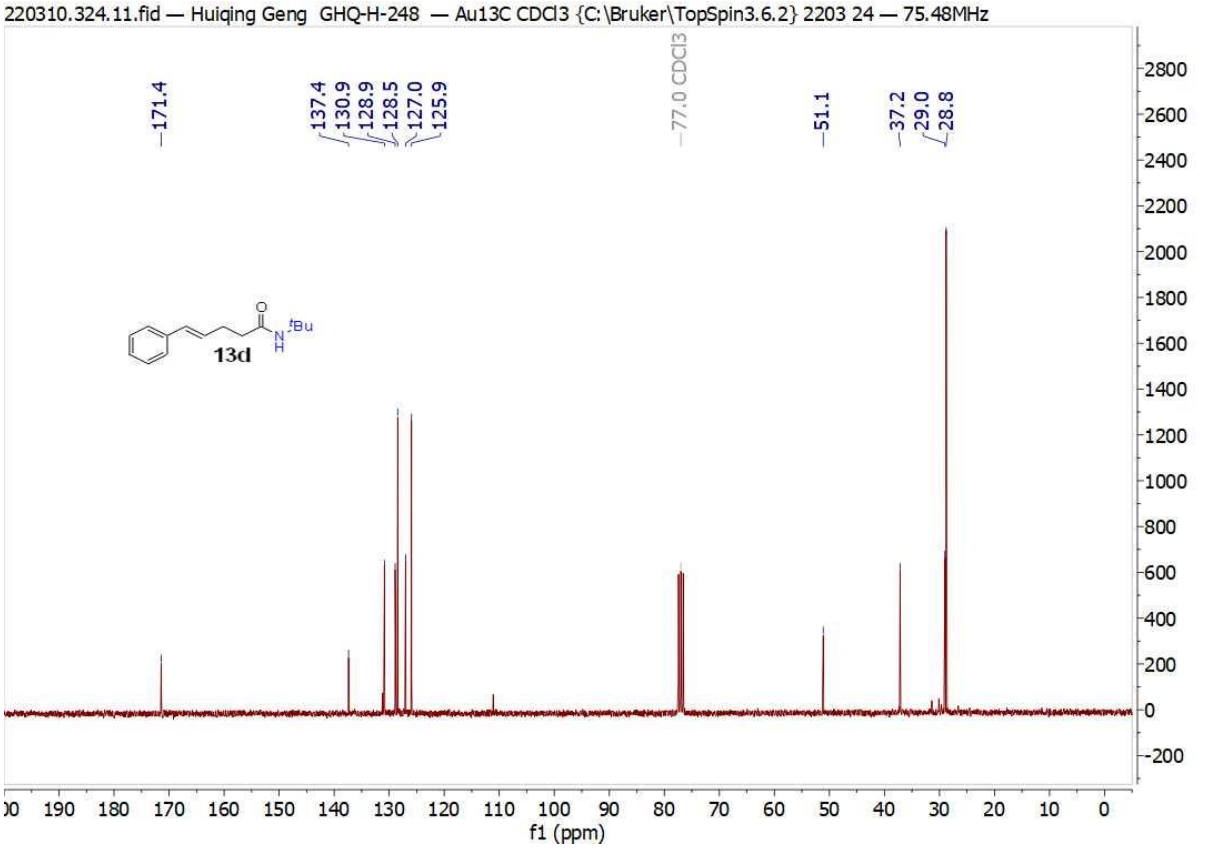

Huiqing Geng, GHQ-H-227 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2202 17 — 300.13MHz
11d
f1 (ppm)

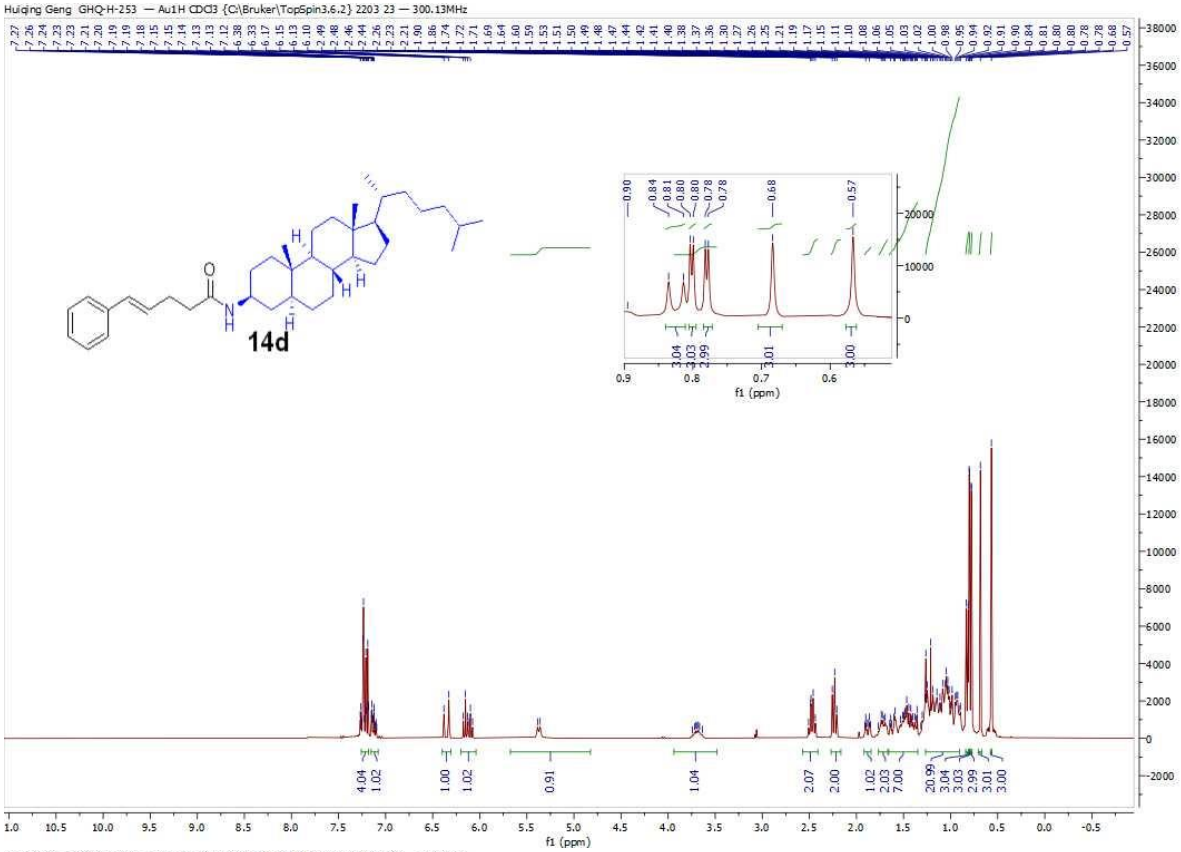

Huiqing Geng, GHQ-H-227 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2202 17 — 75.48MHz
11d
f1 (ppm)

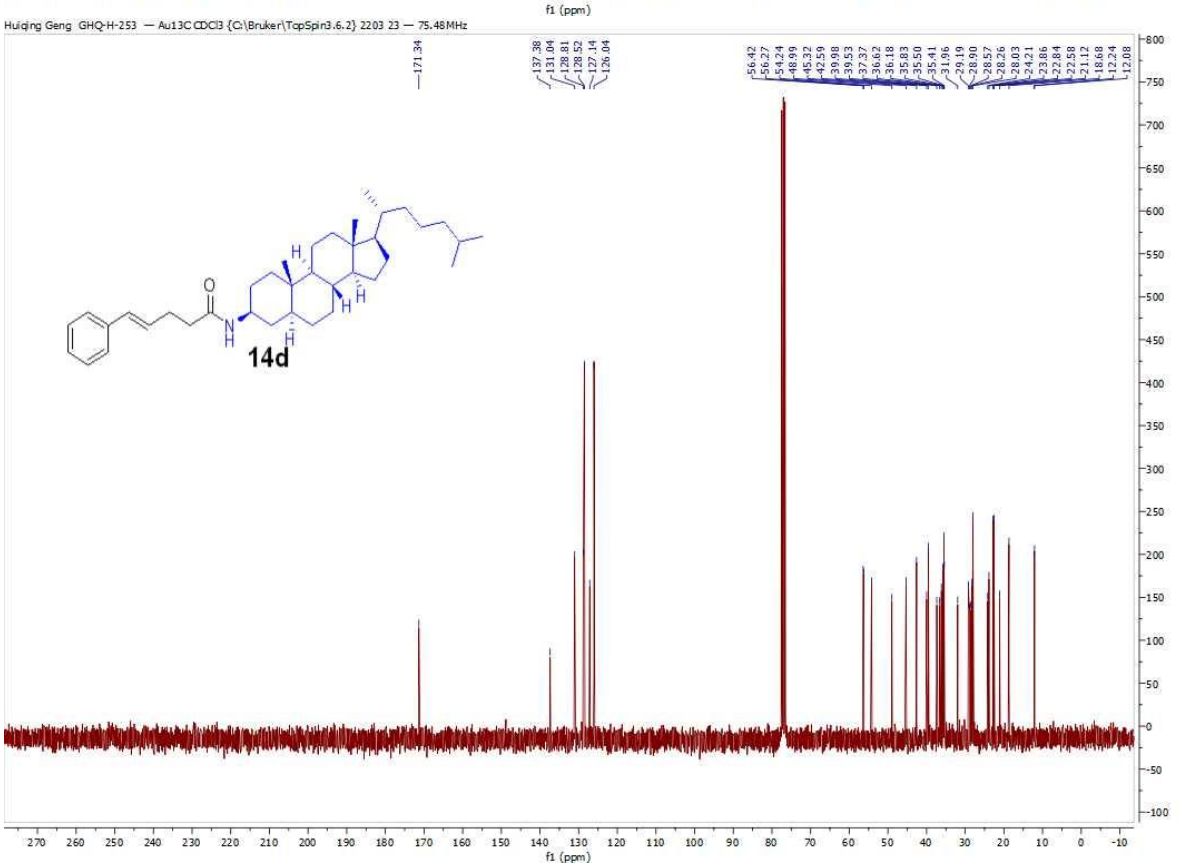

220304.313.10.fid — Huiqing Geng GHQ-H-245 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 13 — 300.13MHz
12d
f1 (ppm)


220304.313.11.fid — Huiqing Geng GHQ-H-245 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 13 — 75.48MHz
12d
f1 (ppm)


220310.324.10.fid — Huiqing Geng GHQ-H-248 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 24 — 300.13MHz
13d
f1 (ppm)


220310.324.11.fid — Huiqing Geng GHQ-H-248 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 24 — 75.48MHz
13d
f1 (ppm)


Huiqing Geng GHQ-H-253 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 23 — 300.13MHz
14d
f1 (ppm)


Huiqing Geng GHQ-H-253 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.2} 2203 23 — 75.48MHz
14d
f1 (ppm)