Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Deaminative Carbonylative Coupling of Alkylamines with Styrenes under Transition-Metal-Free Conditions

Fengqian Zhao, Chong-Liang Li, and Xiao-Feng Wu*

Leibniz-Institut für Katalyse an der Universität Rostock e. V., Albert-Einstein-Straße 29a, 18059 Rostock, Germany
Phone: (+49) -0381-1281-343; E-Mail: xiao-feng.wu@catalysis.de

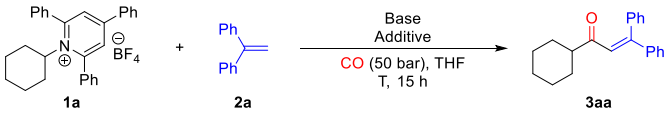
Table of Contents

1. General Information

Unless otherwise noted, materials were purchased from commercial suppliers and used without further purification. Flash column chromatography was performed using 200-300 mesh silica gel. ^{1}H NMR spectra were recorded on 300 or 400 MHz spectrophotometers. Chemical shifts are reported in ppm relative to tetramethylsilane (TMS) with the solvent resonance employed as the internal standard ($CDCl_3$: δ = 7.26 ppm, DMSO-*d6*: δ = 2.50 ppm). ^{13}C NMR was recorded at 75 MHz or 101 MHz: chemical shifts are reported in ppm from tetramethylsilane (TMS) with the solvent resonance as the internal standard ($CDCl_3$: δ = 77.00 ppm, DMSO-*d6*: δ = 39.52 ppm). Electron impact (EI) mass spectra were recorded on AMD 402 mass spectrometer (70 eV). High resolution mass spectra (HR-MS) were recorded on Agilent 6210. The data were given as mass units per charge (m/z). Gas chromatography analysis was performed on an Agilent HP-5890 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.

2. Optimization studies

Table S1 Optimization of the reaction conditions

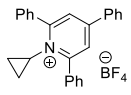


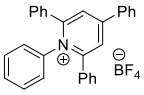
Entry	2a (equiv)	Base (equiv)	Additive (equiv)	T (°C)	3aa (%)[a]
1	2.0	DBU (2.0)	/	100	54
2	2.0	DBN (2.0)	/	100	53
3	2.0	TBD (2.0)	/	100	54
4	2.0	DABCO (2.0)	/	100	1
5	2.0	DIPEA (2.0)	/	100	2
6	2.0	DBU (4.0)	/	100	62
7	2.0	DBU (2.0)	Cs_2CO_3 (1.0)	100	58
8	2.0	DBU (2.0)	K_2CO_3 (1.0)	100	58
9	2.0	DBU (2.0)	KOH (1.0)	100	55
10	2.0	DBU (2.0)	K_2HPO_4 (1.0)	100	41
11	2.0	DBU (2.0)	*t*-BuOK (1.0)	100	64
12	2.0	DBU (2.0)	*t*-BuONa (1.0)	100	50
13	2.0	DBU (2.0)	*t*-BuOLi (1.0)	100	66
14	2.0	DBU (2.0)	NaOMe (1.0)	100	57

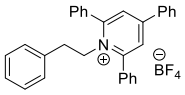
15	2.0	DBU (2.0)	LiOMe (1.0)	100	68
16	2.0	DBU (2.0)	LiOH (1.0)	100	59
17	2.0	DBU (2.0)	Li_2CO_3 (1.0)	100	40
18	2.0	/	LiOMe (1.0)	100	2
19	1.0	DBU (2.0)	LiOMe (1.0)	100	48
20	1.5	DBU (2.0)	LiOMe (1.0)	100	60
21	3.0	DBU (2.0)	LiOMe (1.0)	100	58
22	2.0	DBU (2.0)	LiOMe (1.0)	80	70
23	2.0	DBU (2.0)	LiOMe (1.0)	70	59
24[b]	2.0	DBU (2.0)	LiOMe (1.0)	80	76 (74)[c]

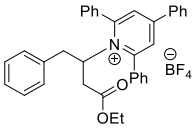
Reaction conditions: **1a** (0.1 mmol), **2a** (0.2 mmol), base, additive, THF, CO (50 bar), 15 h. [a] Determined by GC using hexadecane as the internal standard. [b] THF (2.0 mL). [c] isolated yield.

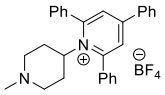
Unsuccessful Katritzky salts:



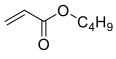


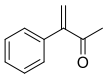


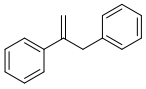




Unsuccessful styrenes:



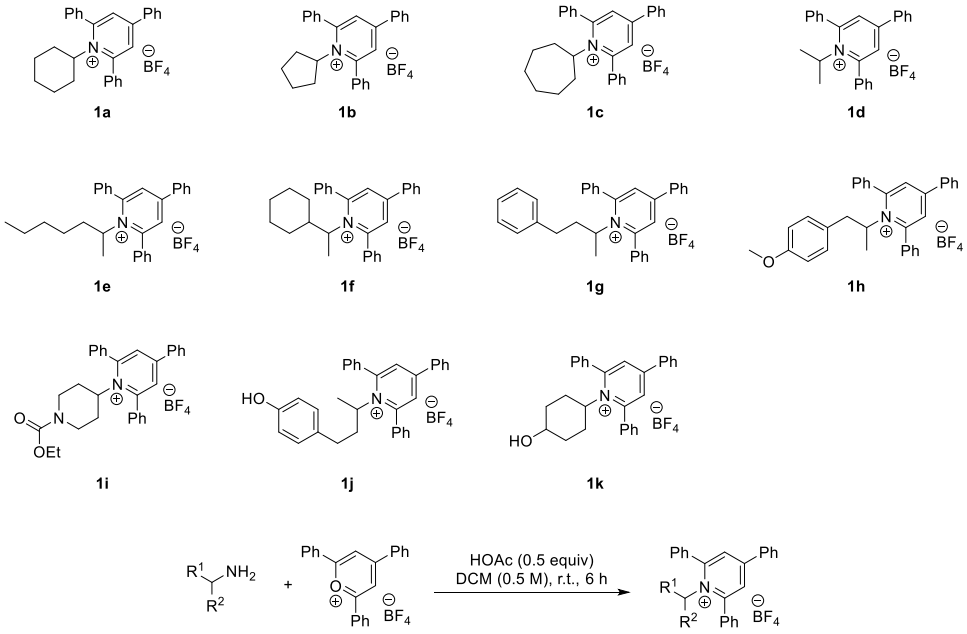




3. General procedure for the synthesis of various substrates

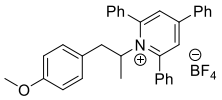
3.1 General procedure for synthesize Katritzky salts

Katritzky salts **1a-1g**, **1k** were all synthesized as described previously.[1]



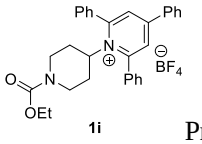
1h, **1i** and **1j** were prepared following a procedure by Glorus et al.[1d] A flask was charged with amine (1.2 equiv), DCM (0.5 M) and acetic acid (0.5 equiv), the mixtures was stirred at room temperature for 15-30 mins. Subsequently, 2,4,6-Triphenylpyrylium tetrafluoroborate (1.0 equiv) was added and stirred at room temperature for 6 h. If precipitation occurred during the reaction, the solid was collected by filtration and washed with EtOH and Et_2O. If no precipitation occurred, Et_2O was added and the crude mixture was stirred for 1 h. The resulting solid was collected by filtration and washed with Et_2O. If precipitation did still not take place, the solvent was removed under reduced pressure and the crude product was purified by column chromatography (DCM: acetone gradient).

1-(1-(4-Methoxyphenyl)propan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (1h):



Prepared in accordance to the general procedure to afford **1h** (263 mg, 48% yield) as white solid. **^{1}H NMR** (300 MHz, DMSO) δ 8.38 (s, 2H), 8.28 – 8.18 (m, 2H), 7.86 – 7.56 (m, 13H), 6.71 (d, *J* = 8.7 Hz, 2H), 6.57 (d, *J* = 8.7 Hz, 2H), 5.05 – 4.89 (m, 1H), 3.66 (s, 3H), 3.15 (dd, *J* = 13.5, 6.0 Hz, 1H), 2.53 (dd, *J* = 13.8, 8.4 Hz, 1H), 1.27 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR** (75 MHz, DMSO) δ 158.8, 157.9, 153.9, 134.3, 133.3, 133.0, 131.4, 130.1, 123.0, 129.2, 128.6, 114.5, 68.7, 55.5, 41.7, 21.4. **HRMS (ESI)**, m/z: $[M\text{-}BF_4]^+$ calculated for $C_{33}H_{30}NO$: 456.2332, found $[M\text{-}BF_4]^+$: 456.2324.

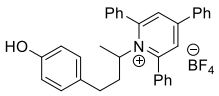
1-(1-(Ethoxycarbonyl)piperidin-4-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (1i):



1i

Prepared in accordance to the general procedure to afford **1i** (220 mg, 40% yield) as white solid. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.78 – 7.69 (m, 6H), 7.69 – 7.63 (m, 2H), 7.61 – 7.45 (m, 7H), 7.40 (t, *J* = 7.5 Hz, 2H), 4.75 (t, *J* = 12.0 Hz, 1H), 3.94 (q, *J* = 7.2 Hz, 4H), 2.15 – 2.03 (m, 4H), 1.65 – 1.60 (m, 2H), 1.11 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 157.0, 155.3, 155.0, 133.9, 133.7, 131.9, 131.0, 129.5, 129.3, 128.9, 128.2, 128.1, 69.6, 61.4, 44.1, 32.4, 14.5. **HRMS (ESI)**, m/z: $[M\text{-}BF_4]^+$ calculated for $C_{31}H_{31}N_2O_2$: 463.2390, found$[M\text{-}BF_4]^+$: 463.2388.

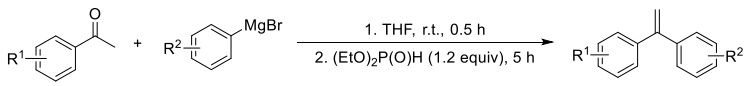
1-(1-(4-Methoxyphenyl)propan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (1j):



1j

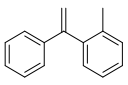
Prepared in accordance to the general procedure to afford **1j** (120 mg, 22% yield) as yellow solid. **^{1}H NMR** (300 MHz, DMSO) δ 9.23 (s, 1H), 8.37 (s, 2H), 8.27 – 8.22 (m, 2H), 7.78 – 7.56 (m, 13H), 6.73 (d, *J* = 8.7 Hz, 2H), 6.58 (d, *J* = 8.4 Hz, 2H), 4.84 – 4.69 (m, 1H), 2.23 – 2.13 (m, 1H), 2.02 – 1.91 (m, 2H), 1.66 – 1.52 (m, 1H), 1.39 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR** (75 MHz, DMSO) δ 157.2, 155.7, 153.3, 133.9, 132.9, 132.4, 130.8, 129.6, 129.4, 128.9, 128.7, 115.3, 66.0, 37.5, 30.8, 21.9. **HRMS (ESI)**, m/z: $[M\text{-}BF_4]^+$ calculated for $C_{33}H_{30}NO_2$: 456.2332, found$[M\text{-}BF_4]^+$: 456.2330.

3.2 General procedure for synthesize styrenes



General procedure A:[2] The Grignard solution in THF (4.0 mmol) was added dropwise to a solution of acetophenone (2.0 mmol, 1.0 equiv) in dry THF (10 mL) under a nitrogen atmosphere at room temperature. The reaction mixture was stirred for 0.5 h and the diethyl phosphite (2.4 mmol) was added. The mixture was stirred for 5 h. Then, the reaction mixture was added water and extracted with diethyl ether. The combined organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure. The crude residue was purified by flash chromatography using *n*-Pentane.

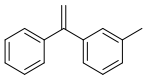
1-Methyl-2-(1-phenylvinyl)benzene (2b):[3]



2b

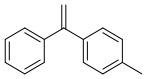
Prepared in accordance to the general procedure A to afford **2b** (217 mg, 56% yield) as colorless oil. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.33 – 7.30 (m, 5H), 7.28 – 7.20 (m, 4H), 5.81 (dd, *J* = 1.5, 0.6 Hz, 1H), 5.24 (dd, *J* = 1.5, 0.6 Hz, 1H), 2.09 (d, *J* = 0.6 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 149.4, 141.6, 140.6, 136.1, 130.0, 130.0, 128.3, 127.5, 127.5, 126.5, 125.6, 114.8, 20.1. **GC-MS** (EI, 70 eV): m/z (%) = 115 (20), 179 (100), 194 (20).

1-Methyl-3-(1-phenylvinyl)benzene (2c):[4]



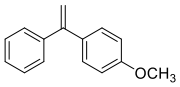
2c Prepared in accordance to the general procedure A to afford **2c** (208 mg, 54% yield) as colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40 – 7.34 (m, 5H), 7.29 – 7.24 (m, 1H), 7.21 – 7.16 (m, 3H), 5.49 (s, 2H), 2.39 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 150.1, 141.6, 141.5, 137.7, 128.9, 128.4, 128.2, 128.1, 128.0, 127.6, 125.4, 114.1, 21.4. **GC-MS** (EI, 70 eV): m/z (%) = 179 (100), 194 (80).

1-Methyl-4-(1-phenylvinyl)benzene (2d):[5]



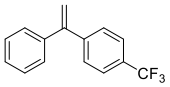
2d Prepared in accordance to the general procedure A to afford **2d** (259 mg, 67% yield) as colorless oil. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.27 – 7.22 (m, 5H), 7.18 – 7.13 (m, 2H), 7.07 – 7.04 (m, 2H), 5.35 (d, *J* = 1.5 Hz, 1H), 5.32 (d, *J* = 1.5 Hz, 1H), 2.29 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 149.9, 141.7, 138.6, 137.5, 128.8, 128.3, 128.1, 128.1, 127.6, 113.6, 21.2. **GC-MS** (EI, 70 eV): m/z (%) = 179 (100), 197(80).

1-Methoxy-4-(1-phenylvinyl)benzene (2e):[3]



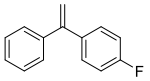
2e Prepared in accordance to the general procedure A to afford **2e** (340.6 mg, 81% yield) as white solid. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.28 – 7.17 (m, 7H), 6.81 – 6.76 (m, 2H), 5.32 (d, *J* = 1.5 Hz, 1H), 5.28 (d, *J* = 1.5 Hz, 1H), 3.75 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 159.3, 149.5, 141.8, 134.0, 129.4, 128.3, 128.1, 127.6, 113.5, 112.9, 55.3. **GC-MS** (EI, 70 eV): m/z (%) = 152 (50), 165 (75), 195 (80), 210 (100).

1-(1-Phenylvinyl)-4-(trifluoromethyl)benzene (2g):[3]



2g Prepared in accordance to the general procedure A to afford **2g** (352.1 mg, 74% yield) as colorless oil. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.61 – 7.58 (m, 2H), 7.47 – 7.44 (m, 2H), 7.37 – 7.29 (m, 5H), 5.57 (d, *J* = 0.9 Hz, 1H), 5.52 (d, *J* = 0.9 Hz, 1H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 148.9, 145.1, 140.6, 128.6, 128.4, 128.1, 128.1, 125.2 (q, *J* = 3.8 Hz), 124.2 (q, *J* = 272.2 Hz), 115.9. **^{19}F NMR** (282 MHz, $CDCl_3$) δ -62.5. **GC-MS** (EI, 70 eV): m/z (%) = 179 (100), 233 (40), 248 (100).

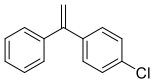
1-Fluoro-4-(1-phenylvinyl)benzene (2h):[6]



2h Prepared in accordance to the general procedure A to afford **2h** (277.5 mg, 70% yield) as colorless oil. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.35 – 7.28 (m, 7H), 7.06 – 6.98 (m, 2H), 5.44 (d, *J* = 1.2 Hz, 1H), 5.42 (d, *J* = 1.2 Hz,

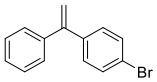
1H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 162.5 (d, *J* = 244.5 Hz), 149.0, 141.3, 137.5, 129.9 (d, *J* = 7.5 Hz), 128.2, 128.2, 127.8, 115.0 (d, *J* = 21.0Hz), 114.2 (d, *J* = 1.5 Hz). **^{19}F NMR** (282 MHz, $CDCl_3$) δ -114.8. **GC-MS** (EI, 70 eV): m/z (%) = 177 (40), 183 (100), 198 (100).

1-Chloro-4-(1-phenylvinyl)benzene (2i):[5]



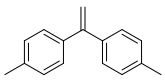
2i Prepared in accordance to the general procedure A to afford **2i** (283.4 mg, 66% yield) as colorless oil. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.38 – 7.34 (m, 5H), 7.34 – 7.27 (m, 4H), 5.50 (d, *J* = 1.2 Hz, 1H), 5.48 (d, *J* = 1.2 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 149.0, 141.0, 139.9, 133.6, 129.5, 128.3, 128.2, 128.2, 127.9, 114.7. **GC-MS** (EI, 70 eV): m/z (%) = 178 (100), 199 (20), 214 (60).

1-Bromo-4-(1-phenylvinyl)benzene (2j):[6]



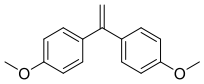
2j Prepared in accordance to the general procedure A to afford **2j** (283.6 mg, 55% yield) as colorless oil. **^{1}H NMR** (300 MHz, $CDCl_3$)) δ 7.48 (dt, *J* = 8.7, 2.4 Hz, 2H), 7.37 – 7.30 (m, 5H), 7.23 (dt, *J* = 8.7, 2.4 Hz, 2H), 5.49 (d, *J* = 1.2 Hz, 1H), 5.47 (d, *J* = 1.2 Hz, 1H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 149.0, 140.9, 140.4, 131.3, 129.9, 128.3, 128.2, 127.9, 121.8, 114.7. **GC-MS** (EI, 70 eV): m/z (%) = 178 (100), 258 (50).

4,4'-(Ethene-1,1-diyl)bis(methylbenzene) (2l):[3]



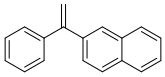
2l Prepared in accordance to the general procedure A to afford **2l** (299.5 mg, 72% yield) as white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.24 (dt, *J* = 8.0, 2.0 Hz, 4H), 7.14 (m, 4H), 5.39 (s, 2H), 2.37 (s, 6H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 149.7, 138.8, 137.4, 128.8, 128.2, 113.0, 21.2. **GC-MS** (EI, 70 eV): m/z (%) = 115 (30), 178 (80), 193 (100), 208 (100).

4,4'-(Ethene-1,1-diyl)bis(methoxybenzene) (2m):[4]



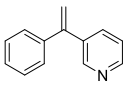
2m Prepared in accordance to the general procedure A to afford **2m** (398.0 mg, 62% yield) as white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.30 (dt, *J* = 10.8, 2.8 Hz, 4H), 6.89 (dt, *J* = 10.4, 2.8 Hz, 4H), 5.32 (s, 2H), 3.84 (s, 6H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 159.3, 148.9, 134.3, 129.4, 113.4, 111.6, 55.2. **GC-MS** (EI, 70 eV): m/z (%) = 153 (40), 165 (50), 182 (30), 209 (40), 225 (80), 240 (100).

2-(1-Phenylvinyl)naphthalene (2p):[7]



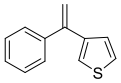
2p Prepared in accordance to the general procedure A to afford **2p** (320.0 mg, 35% yield) as colorless oil. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.90 – 7.79 (m, 4H), 7.54 – 7.46 (m, 3H), 7.45 – 7.35 (m, 5H), 5.62 (dd, *J* = 0.9, 0.6 Hz, 1H), 5.58 (dd, *J* = 0.9, 0.6 Hz, 1H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 150.0, 141.5, 138.9, 133.3, 133.0, 128.4, 128.2, 128.2, 127.8, 127.7, 127.6, 127.3, 126.4, 126.1, 126.0, 114.8. **GC-MS** (EI, 70 eV): m/z (%) = 215 (70), 230 (100).

3-(1-Phenylvinyl)thiophene (2r):[8]

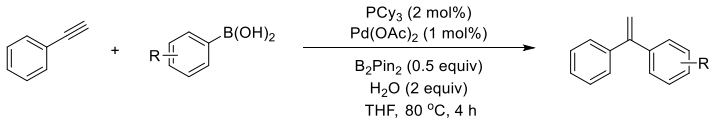


2r Prepared in accordance to the general procedure A to afford **2r** (80 mg, 25% yield) as yellow oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.61 (d, *J* = 28.8 Hz, 2H), 7.62 (dt, *J* = 8.0, 2.0 Hz, 1H), 7.60 – 7.20 (m, 6H), 5.57 (d, *J* = 0.8 Hz, 1H), 5.50 (d, *J* = 0.8 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 149.0, 148.6, 146.8, 140.2, 137.2, 135.7, 128.4, 128.2, 128.0, 123.1. **GC-MS** (EI, 70 eV): m/z (%) = 152 (30), 166 (30), 180 (100), 181 (70).

3-(1-Phenylvinyl)thiophene (2s):[9]

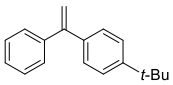


2s Prepared in accordance to the general procedure A to afford **2s** (189.0 mg, 50% yield) as yellow oil. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.43 – 7.32 (m, 5H), 7.31 (dd, *J* = 4.8, 3.0 Hz, 1H), 7.19 (dd, *J* = 6.8, 1.5 Hz, 1H), 7.13 (dd, *J* = 3.0, 1.2 Hz, 1H), 5.54 (d, *J* = 1.2 Hz, 1H), 5.34 (d, *J* = 1.2 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.6, 142.5, 141.5, 128.2, 128.1, 127.8, 127.2, 125.4, 123.3, 113.4. **GC-MS** (EI, 70 eV): m/z (%) = 152 (30), 171 (60), 186 (100).



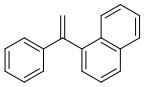
General procedure B:[10] A 10 mL round bottomed flask equipped with a magnetic stir bar was charged with alkyne (2.0 mmol), B_2Pin_2 (1.0 mmol, 0.5 equiv), arylboronic acid (2.0 mmol, 1.0 equiv), PCy_3 (2 mol%), $Pd(OAc)_2$ (1 mol%), H_2O (4.0 mmol, 2.0 equiv) and THF (8 mL). The mixture was stirred for 4 h at 80 °C. After the completion of the reaction, the reaction mixture was concentrated under reduced pressure. The crude residue was purified by flash chromatography using *n*-Pentane.

1-(*tert*-Butyl)-4-(1-phenylvinyl)benzene (2f):[5]

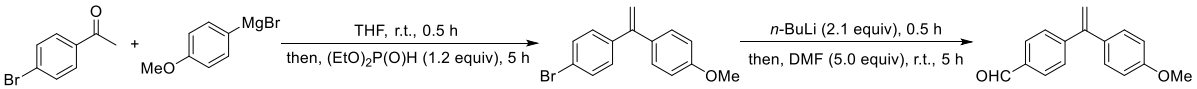


2f Prepared in accordance to the general procedure B to afford **2f** (168.5 mg, 71% yield) as colorless oil. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.38 – 7.26 (m, 9H), 5.46 (d, *J* = 1.5 Hz, 1H), 5.41 (d, *J* = 1.2 Hz, 1H), 1.34 (s, 9H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 150.7, 149.8, 141.7, 138.4, 128.3, 128.1, 127.8, 127.6, 125.0, 113.7, 34.6, 31.3. **GC-MS** (EI, 70 eV): m/z (%) = 103 (50), 221 (100), 236 (50).

1-(1-Phenylvinyl)naphthalene (2q):[3]



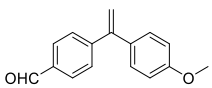
2q Prepared in accordance to the general procedure A to afford **2q** (504.4 mg, 55% yield) as white solid. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.1 Hz, 2H), 7.84 (d, *J* = 8.2 Hz, 1H), 7.56 (t, *J* = 7.2 Hz, 1H), 7.52 – 7.46 (m, 2H), 7.42 – 7.35 (m, 3H), 7.35 – 7.29 (m, 3H), 6.05 (d, *J* = 1.8 Hz, 1H), 5.46 (d, *J* = 1.5 Hz, 1H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 148.4, 141.0, 139.8, 133.7, 131.8, 128.4, 128.2, 127.9, 127.7, 127.2, 126.6, 126.4, 125.8, 125.6, 125.4, 116.2. **GC-MS** (EI, 70 eV): m/z (%) = 152 (50), 229 (100), 230 (80).



General procedure C: The Grignard solution in THF (10.0 mmol) was added dropwise to a solution of 1-(4-bromophenyl)ethan-1-one (5.0 mmol) in dry THF (20 mL) under a nitrogen atmosphere at room temperature. The reaction mixture was stirred for 0.5 h and the diethyl phosphite (6.0 mmol) was added. The mixture was stirred for 5 h. Then, the reaction mixture was added water and extracted with diethyl ether. The combined organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure. 1-bromo-4-(1-(4-methoxyphenyl)vinyl)benzene was obtained by flash chromatography using *n*-Pentane/Ethyl acetate.

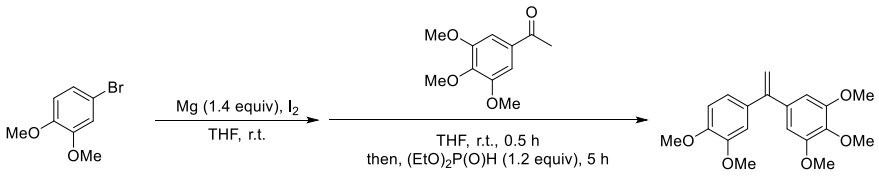
1-bromo-4-(1-(4-methoxyphenyl)vinyl)benze (3.0 mmol) was dissolved in THF (12 mL) under a nitrogen atmosphere and cooled down to -78 °C. The *n*-BuLi (6.3 mmol, 2.1 equiv) was added dropwise over 10 min. The reaction mixture was stirred for 2 h at -78 °C before DMF (15.0 mmol) was added at once. The solution was allowed to room temperature slowly and stirred for 5 h. After the completion of the reaction, the mixture was added water and extracted with diethyl ether. The combined organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure. The crude residue was purified by flash chromatography using *n*-Pentane/Ethyl acetate to afford **2n**.

4-(1-(4-Methoxyphenyl)vinyl)benzaldehyde (2n)



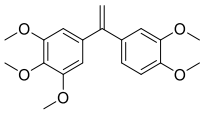
2n Prepared in accordance to the general procedure C to afford **2n** (364.1 mg, 51% yield) as white solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.92 (s, 1H), 7.74 (dt, *J* = 8.0, 1.6 Hz, 2H), 7.40 (dt, *J* = 8.0, 1.6 Hz, 2H), 7.14

(dt, *J* = 8.8, 2.0 Hz, 2H), 6.78 (dt, *J* = 8.8, 2.0 Hz, 2H), 5.42 (d, *J* = 1.2 Hz, 1H), 5.36 (d, *J* = 1.2 Hz, 1H), 3.72 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 191.8, 159.5, 148.6, 147.9, 135.5, 132.9, 129.6, 129.3, 128.8, 115.0, 113.7, 55.2. **HRMS** (ESI) calcd for $C_{16}H_{14}O_2$ $[M+H]^+$: 239.1072, Found: 239.1073.



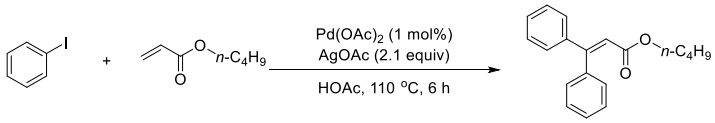
General procedure D: To an oven dried 25 mL schlenk tube equipped with a magnetic stir bar, I_2 and activated magnesium turnings (7 mmol, 1.4 equiv) was added. The equipment was sealed with rubber septum, evacuated, and back filled with nitrogen. To the funnel was added the solution of 4-bromo-1,2-dimethoxybenzene (5.0 mmol, 1.0 equiv) in THF (5 ml) by syringe. The above solution was added dropwise at room temperature within 30 mins. After reaction at room temperature for 2 h, the Grignard solution in THF (5.0 mmol) was added dropwise to a solution of 1-(3,4,5-trimethoxyphenyl)ethan-1-one (2.5 mmol) in dry THF (10 mL) under a nitrogen atmosphere at room temperature. The reaction mixture was stirred for 0.5 h and the diethyl phosphite (3.0 mmol) was added. The mixture was stirred for 5 h. Then, the reaction mixture was added water and extracted with diethyl ether. The combined organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure. The crude residue was purified by flash chromatography using *n*-Pentane/Ethyl acetate to afford **2o**.

5-(1-(3,4-Dimethoxyphenyl)vinyl)-1,2,3-trimethoxybenzene (2o):



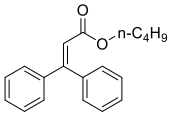
2o

Prepared in accordance to the general procedure D to afford **2o** (780.0 mg, 47% yield) as colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.93 – 6.91 (m, 1H) 6.89 (d, *J* = 2.0 Hz, 1H), 6.84 (d, *J* = 8.4 Hz, 1H), 6.57 (s, 2H), 5.38 (d, *J* = 1.2 Hz, 1H), 5.35 (d, *J* = 2.0 Hz, 1H), 3.91 (s, 3H), 3.88 (s, 3H), 3.85 (s, 3H), 3.82 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 152.8, 149.7, 148.9, 148.5, 137.8, 137.3, 134.0, 121.0, 112.7, 111.4, 110.7, 105.6, 60.9, 56.1, 55.9, 55.9. **HRMS** (ESI) calcd for $C_{19}H_{22}O_5$ $[M+H]^+$: 331.1545, Found: 331.1549.



General procedure E:[11] A 10 mL round bottomed flask equipped with a magnetic stir bar was charged with butyl acrylate (2.0 mmol), iodobenzene (3.1 mmol), $Pd(OAc)_2$ (1 mol%), AgOAc (4.2 mmol) and acetic acid (6 mL). The mixture was stirred for 6 h at 110°C. After the completion of the reaction, the reaction mixture was diluted with water and extracted with ethyl acetate. The combined organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure. The crude residue was purified by flash chromatography using *n*-Pentane/Ethyl acetate to afford **2t**.

3-(1-Phenylvinyl)thiophene (2t):[11]



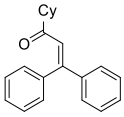
2t

Prepared in accordance to the general procedure E to afford **2t** (100 mg, 24% yield) as colorless oil. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.44 – 7.40 (m, 3H), 7.40 – 7.32 (m, 5H), 7.29 – 7.22 (m, 2H), 6.41 (s, 1H), 4.04 (t, *J* = 6.6 Hz, 2H), 1.55 – 1.44 (m, 2H), 1.33 – 1.21 (m, 2H), 0.90 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 166.2, 156.2, 140.8, 139.0, 129.3, 129.0, 128.3, 128.2, 128.0, 127.8, 117.5, 64.0, 30.4, 19.0, 13.6. **GC-MS** (EI, 70 eV): m/z (%) = 178 (100), 207 (80), 223 (50), 280 (30).

4. General procedure for the synthesis of *α*, *β*-unsaturated ketones

A 4 mL screw-cap vial was charged with Katritzky salts (0.1 mmol), LiOMe (0.1 mmol) and an oven-dried stirring bar. The vial was closed by Teflon septum and phenolic cap and connected with atmosphere with a needle. After flashed the vials with argon and vacuum three times, styrenes (0.2 mmol), DBU (0.2 mmol) and dry THF (2 mL) were injected by syringe. The vial was fixed in an alloy plate and put into Paar 4560 series autoclave (500 mL) under argon atmosphere. At room temperature, the autoclave was flushed with carbon monoxide for three times and 50 bar of carbon monoxide was charged. The autoclave was reacted at 80 °C for 15 h. Afterwards, the pressure was carefully released. After removal of solvent under reduced pressure, pure product was obtained by column chromatography.

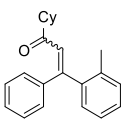
1-Cyclohexyl-3,3-diphenylprop-2-en-1-one (3aa):



3aa

Light cyan oil (21.5 mg, 74% yield), R_f = 0.4 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.39 – 7.29 (m, 8H), 7.21 – 7.16 (m, 2H), 6.63 (s, 1H), 2.25 (tt, *J* = 11.2, 3.2 Hz, 1H), 1.83 – 1.70 (m, 4H), 1.63 – 1.59 (m, 1H), 1.39 – 1.23 (m, 3H), 1.16 – 1.11 (m, 2H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 204.8, 153.4, 141.2, 139.2, 129.4, 129.2, 128.4, 128.3, 128.3, 128.1, 125.4, 50.8, 28.7, 25.8, 25.7. **HRMS** (ESI) calcd for $C_{21}H_{22}O$ $[M+H]^+$: 290.1671, Found: 290.1672.

1-Cyclohexyl-3-phenyl-3-(*o*-tolyl)prop-2-en-1-one (3ab):

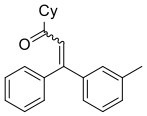


3ab

Light cyan oil (20.1 mg, 66% yield, *E*/*Z* isomer: 85:15 based on NMR), R_f = 0.4 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.35 – 7.28 (m, 5H), 7.28 – 7.14 (m, 3H), 7.06 – 7.02 (m, 1H), 6.81 (s, 0.85H),

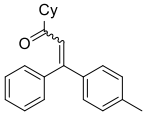
6.25 (s, 0.15H), 2.31 – 2.22 (m, 1H), 2.08 (d, J = 0.9 Hz, 0.45H), 2.06 (s, 2.55H), 1.82 – 1.60 (m, 5H), 1.39 – 1.19 (m, 3H), 1.18 – 1.11 (m, 2H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 203.9, 152.9, 139.8, 138.7, 135.6, 130.6, 130.0, 129.7, 129.3, 129.2, 128.7, 128.5, 128.3, 128.1, 127.9, 127.4, 125.7, 125.6, 125.2, 51.0, 50.7, 28.7, 28.3, 25.8, 25.7, 20.4, 19.6. **HRMS** (ESI) calcd for $C_{22}H_{24}O$ $[M+H]^+$: 305.1905, Found: 305.1901.

1-Cyclohexyl-3-phenyl-3-(*m*-tolyl)prop-2-en-1-one (3ac):



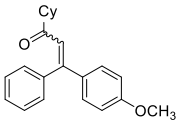
3ac Light cyan oil (19.7 mg, 65% yield, *E*/*Z* isomer: 50:50 based on NMR), R_f = 0.4 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.39 – 7.29 (m, 4H), 7.28 – 7.15 (m, 3H), 7.14 – 7.06 (m, 1H), 7.01 – 6.97 (m, 1H), 6.61 (s, 0.5H), 6.59 (s, 0.5H), 2.35 (s, 1.5H), 2.33 (s, 1.5H), 2.28 – 2.19 (m, 1H), 1.83 – 1.68 (m, 4H), 1.63 – 1.57 (m, 1H), 1.39 – 1.24 (m, 3H), 1.16 – 1.09 (m, 2H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 205.2, 204.9, 153.7, 153.5, 141.35, 141.31, 139.3, 139.1, 138.0, 137.7, 130.0, 123.0, 129.3, 129.13, 129.14, 128.9, 128.4, 128.3, 128.21, 128.22, 128.01, 128.03, 126.5, 125.7, 125.6, 125.3, 50.8, 50.6, 28.8, 28.7, 25.8, 25.7, 21.4. **HRMS** (ESI) calcd for $C_{22}H_{24}O$ $[M+H]^+$: 305.1905, Found: 305.1905.

1-Cyclohexyl-3-phenyl-3-(*p*-tolyl)prop-2-en-1-one (3ad):



3ad Light cyan oil (24.9 mg, 82% yield, *E*/*Z* isomer: 57:43 based on NMR), R_f = 0.4 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.40 – 7.29 (m, 4H), 7.22 – 7.14 (m, 4H), 7.12 – 7.05 (m, 1H), 6.61 (s, 0.57H), 6.58 (s, 0.43H), 2.39 (s, 1.3H), 2.36 (s, 1.7H), 2.34 – 2.18 (m, 1H), 1.85 – 1.69 (m, 4H), 1.63 – 1.59 (m, 1H), 1.38 – 1.26 (m, 3H), 1.16 – 1.10 (m, 2H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 204.9, 204.8, 153.8, 153.6, 141.6, 139.5, 139.4, 138.4, 138.3, 136.2, 129.4, 129.3, 129.1, 129.0, 128.8, 128.5, 128.31, 128.34, 128.2, 128.1, 125.1, 124.6, 50.80, 50.82, 28.7, 25.8, 25.7, 21.3, 21.2. **HRMS** (ESI) calcd for $C_{22}H_{24}O$ $[M+H]^+$: 305.1905, Found: 305.1906.

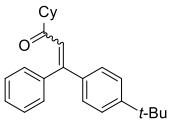
1-Cyclohexyl-3-(4-methoxyphenyl)-3-phenylprop-2-en-1-one (3ae):



3ae Light cyan oil (23.3 mg, 75% yield, *E*/*Z* isomer: 50:50 based on NMR), R_f = 0.2 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.28 – 7.16 (m, 4H), 7.14 – 7.11 (m, 1H), 7.07 – 6.98 (m, 2H), 6.80 – 6.70 (m, 2H), 6.46 (s, 0.5H), 6.41 (s, 0.5H), 3.72 (s, 1.5H), 3.69 (s, 1.5H), 2.21 – 2.07 (m, 1H), 1.72 – 1.57 (m, 4H), 1.52 – 1.46 (m, 1H), 1.23 – 1.13 (m, 3H), 1.05 – 0.98 (m, 2H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 204.9, 204.6, 160.6, 159.9, 153.6, 153.4, 141.9, 139.4, 133.6, 131.2, 131.1, 129.8, 129.3, 129.1, 128.6, 128.21, 128.23, 128.1, 124.9, 123.5,

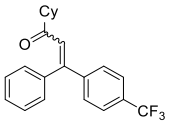
113.7, 113.4, 55.3, 55.2, 50.9, 50.7, 28.8, 25.80, 25.81. **HRMS** (ESI) calcd for $C_{22}H_{24}O_2$ $[M+H]^+$: 321.1855, Found: 321.1855.

3-(4-(*tert*-Butyl)phenyl)-1-cyclohexyl-3-phenylprop-2-en-1-one (3af):



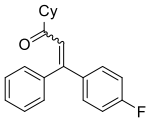
3af Light cyan oil (24.8 mg, 72% yield, *E*/*Z* isomer: 58:42 based on NMR), R_f = 0.4 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.43 – 7.33 (m, 6H), 7.29 – 7.27 (m, 1H), 7.22 – 7.19 (m, 1H), 7.17 – 7.11 (m, 1H), 6.66 (s, 0.58H), 6.58 (s, 0.42H), 2.30 – 2.15 (m, 1H), 1.88 – 1.53 (m, 6H), 1.37 (s, 3.5H), 1.34 (s, 5.5H), 1.31 – 1.27 (m, 1H), 1.20 – 1.02 (m, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 205.5, 204.7, 153.6, 153.5, 152.7, 151.6, 141.5, 139.3, 138.2, 136.0, 129.4, 129.3, 129.1, 128.5, 128.3, 128.2, 128.11, 128.13, 125.7, 125.3, 125.0, 124.6, 50.8, 50.6, 34.7, 34.6, 31.3, 31.2, 28.9, 28.7, 25.80, 25.81, 25.82, 25.7. **HRMS** (ESI) calcd for $C_{25}H_{30}O$ $[M+H]^+$: 347.2375, Found: 347.2368.

1-Cyclohexyl-3-phenyl-3-(4-(trifluoromethyl)phenyl)prop-2-en-1-one (3ag):



3ag Yellow solid (26.4 mg, 74% yield, *E*/*Z* isomer: 50:50 based on NMR), R_f = 0.3 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.65 – 7.56 (m, 2H), 7.43 – 7.25 (m, 6H), 7.20 – 7.13 (m, 1H), 6.76 (s, 0.5H), 6.63 (s, 0.5H), 2.38 (tt, *J* = 11.1, 3.0 Hz, 0.5H), 2.26 (tt, *J* = 11.4, 3.3 Hz, 0.5H), 1.89 – 1.74 (m, 4H), 1.68 – 1.59 (m, 1H), 1.37 – 1.23 (m, 3H), 1.21 – 1.11 (m, 2H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 204.8, 203.4, 152.6, 151.5, 144.8, 143.0, 140.4, 138.3, 131.1, 130.7, 130.2, 129.8, 129.6, 129.5, 129.3, 128.7, 128.6, 128.5, 128.3, 128.2, 127.1, 125.9, 125.3 (q, *J* = 3.8 Hz), 125.2, 125.1 (q, *J* = 3.8 Hz), 122.3, 51.4, 50.8, 28.6, 28.5, 25.8, 25.70, 25.71. **^{19}F NMR** (282 MHz, $CDCl_3$) δ -62.5, -62.7. **HRMS** (ESI) calcd for $C_{22}H_{21}F_3O$ $[M+H]^+$: 359.1623, Found: 359.1624.

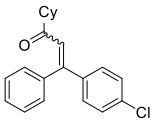
1-Cyclohexyl-3-(4-fluorophenyl)-3-phenylprop-2-en-1-one (3ah):



3ah Light cyan oil (21.6 mg, 70% yield, *E*/*Z* isomer: 50:50 based on NMR), R_f = 0.3 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.40 – 7.25 (m, 5H), 7.19 – 7.13 (m, 2H), 7.10 – 6.97 (m, 2H), 6.64 (s, 0.5H), 6.57 (s, 0.5H), 2.39 – 2.18 (m, 1H), 1.87 – 1.69 (m, 4H), 1.66 – 1.59 (m, 1H), 1.37 – 1.23 (m, 3H), 1.19 – 1.10 (m, 2H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 204.7, 204.2, 165.0, 164.4, 161.7, 161.1, 152.8, 152.4, 141.2, 139.0, 137.3, 135.0, 134.9, 131.3, 131.2, 130.3, 130.2, 129.4, 129.3, 128.5, 128.40, 128.41, 128.2, 125.21, 125.22, 115.5, 115.20, 115.21,

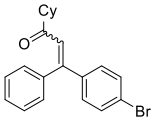
115.0, 51.1, 50.8, 28.7, 28.6, 25.80, 25.81, 25.7. **^{19}F NMR** (282 MHz, $CDCl_3$) δ -111.9, -113.2. **HRMS** (ESI) calcd for $C_{21}H_{21}FO$ $[M+H]^+$: 309.1655, Found: 309.1653.

3-(4-Chlorophenyl)-1-cyclohexyl-3-phenylprop-2-en-1-one (3ai):



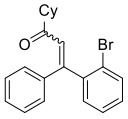
3ai Light cyan oil (22.5 mg, 69% yield, *E*/*Z* isomer: 50:50 based on NMR), R_f = 0.4 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.33 – 7.23 (m, 4H), 7.25 – 7.11 (m, 3H), 7.11 – 7.00 (m, 2H), 6.58 (s, 0.5H), 6.51 (s, 0.5H), 2.27 (tt, *J* = 11.6, 3.6 Hz, 0.5H), 2.16 (tt, *J* = 11.6, 3.6 Hz, 0.5H), 1.82 – 1.61 (m, 4H), 1.60 – 1.50 (m, 1H), 1.26 – 1.16 (m, 2H), 1.16 – 0.99 (m, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 204.7, 203.9, 152.7, 152.1, 140.9, 139.7, 138.7, 137.5, 135.3, 134.2, 130.7, 129.6, 129.5, 129.3, 128.50, 128.51, 128.4, 128.31, 128.32, 128.2, 125.6, 125.1, 51.2, 50.8, 28.63, 28.61, 25.7. **HRMS** (ESI) calcd for $C_{21}H_{21}OCl$ $[M+H]^+$: 325.1359, Found: 325.1359.

3-(4-Bromophenyl)-1-cyclohexyl-3-phenylprop-2-en-1-one (3aj):



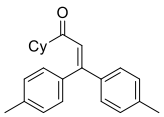
3aj Light cyan oil (25.7 mg, 70% yield, *E*/*Z* isomer: 50:50 based on NMR), R_f = 0.4 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.37 – 7.29 (m, 2H), 7.25 – 7.16 (m, 3H), 7.15 – 7.11 (m, 1H), 7.04 – 6.99 (m, 2H), 6.93 – 6.88 (m, 1H), 6.52 (s, 0.5H), 6.45 (s, 0.5H), 2.21 (tt, *J* = 12.0, 3.3 Hz, 0.5H), 2.09 (tt, *J* = 11.4, 3.3 Hz, 0.5H), 1.74 – 1.55 (m, 4H), 1.52 – 1.44 (m, 1H), 1.20 – 1.09 (m, 3H), 1.06 – 0.96 (m, 2H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 204.7, 203.8, 152.7, 152.1, 140.8, 140.2, 138.6, 138.0, 131.5, 131.3, 131.0, 129.9, 129.5, 129.3, 128.5, 128.4, 128.3, 128.2, 125.7, 125.0, 123.6, 122.5, 51.2, 50.8, 28.62, 28.61, 25.83, 25.81, 25.7, 25.6. **HRMS** (ESI) calcd for $C_{21}H_{21}OBr$ $[M+H]^+$: 369.0854, Found: 369.0849.

3-(2-Bromophenyl)-1-cyclohexyl-3-phenylprop-2-en-1-one (3ak):



3ak Light cyan oil (30.0 mg, 81% yield, *E*/*Z* isomer: 89:11 based on NMR), R_f = 0.3 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.56 – 7.53 (m, 1H), 7.28 – 7.24 (m, 5H), 7.21 – 7.13 (m, 2H), 7.08 – 7.05 (m, 1H), 6.79 (s, 0.89H), 6.23 (s, 0.11H), 2.31 (tt, *J* = 11.1, 3.3 Hz, 0.91H), 2.23 – 2.17 (m, 0.14H), 1.83 – 1.67 (m, 4H), 1.59 – 1.50 (m, 1H), 1.24 – 1.17 (m, 2H), 1.15 – 1.03 (m, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 202.7, 151.8, 140.3, 138.9, 132.8, 130.3, 129.5, 129.1, 128.6, 127.5, 127.2, 125.1, 122.3, 51.2, 28.63, 28.62, 28.2, 25.82, 25.81, 25.7. **HRMS** (ESI) calcd for $C_{21}H_{21}OBr$ $[M+H]^+$: 369.0854, Found: 369.0858.

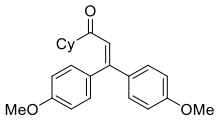
1-Cyclohexyl-3,3-di-*p*-tolylprop-2-en-1-one (3al):



3al

White solid (22.9 mg, 72% yield), R_f = 0.3 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.22 – 7.11 (m, 6H), 7.09 – 7.04 (m, 2H), 6.57 (s, 1H), 2.39 (s, 3H), 2.36 (s, 3H), 2.27 (tt, *J* = 11.4, 3.0 Hz, 1H), 1.85 – 1.69 (m, 4H), 1.65 – 1.57 (m, 1H), 1.40 – 1.21 (m, 3H), 1.17 – 1.11 (m, 2H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 204.8, 153.9, 139.4, 138.7, 138.1, 136.3, 129.4, 129.0, 128.7, 128.4, 124.3, 50.8, 28.8, 25.82, 25.81, 21.3, 21.2. **HRMS** (ESI) calcd for $C_{23}H_{26}O$ $[M+H]^+$: 318.1984, Found: 318.1982.

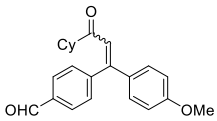
1-Cyclohexyl-3,3-bis(4-methoxyphenyl)prop-2-en-1-one (3am):



3am

Light cyan solid (21.7 mg, 62% yield), R_f = 0.3 (*n*-Pentane/EtOAc = 20/1). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.17 – 7.12 (m, 2H), 7.04 – 6.99 (m, 2H), 6.83 – 6.71 (m, 4H), 6.40 (s, 1H), 3.74 (s, 3H), 3.72 (s, 3H), 2.18 (tt, *J* = 11.4, 3.3 Hz, 1H), 1.75 – 1.59 (m, 4H), 1.54 – 1.46 (m, 1H), 1.27 – 1.16 (m, 3H), 1.10 – 0.99 (m, 2H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 204.7, 160.6, 159.8, 153.6, 134.2, 131.5, 131.1, 130.1, 123.1, 113.6, 113.4, 55.3, 55.2, 50.8, 28.8, 25.82, 25.81. **HRMS** (ESI) calcd for $C_{23}H_{26}O_3$ $[M+H]^+$: 351.1960, Found: 351.1955.

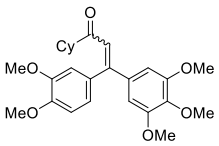
4-(3-Cyclohexyl-1-(4-methoxyphenyl)-3-oxoprop-1-en-1-yl)benzaldehyde (3an):



3an

Yellow oil (23.5 mg, 67% yield, *E*/*Z* isomer: 57:43 based on NMR), R_f = 0.4 (*n*-Pentane/EtOAc = 20/1). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 10.04 (s, 0.57H), 10.03 (s, 0.43H), 7.91 – 7.82 (m, 2H), 7.48 – 7.43 (m, 1H), 7.34 – 7.30 (m, 1H), 7.23 – 7.19 (m, 1H), 7.13 – 7.08 (m, 1H), 6.92 – 6.83 (m, 2H), 6.72 (s, 0.57H), 6.58 (s, 0.43H), 3.84 (s, 1.13H), 3.82 (s, 1.85H), 2.34 – 2.25 (m, 1H), 1.93 – 1.70 (m, 4H), 1.69 – 1.59 (m, 1H), 1.29 – 1.20 (m, 3H), 1.17 – 1.14 (m, 2H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 204.9, 203.1, 191.9, 191.6, 161.0, 160.1, 152.7, 151.6, 147.8, 146.4, 136.4, 135.6, 132.4, 131.1, 130.3, 129.7, 129.6, 129.5, 129.2, 128.7, 126.9, 122.9, 114.0, 113.7, 55.4, 55.3, 51.4, 50.9, 28.7, 28.5, 25.82, 25.81, 25.7. **HRMS** (ESI) calcd for $C_{23}H_{24}O_3$ $[M+H]^+$: 349.1804, Found: 349.1806.

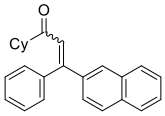
1-Cyclohexyl-3-(3,4-dimethoxyphenyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one (3ao):



3ao

Light cyan oil (40.0 mg, 91% yield, *E*/*Z* isomer: 55:45 based on NMR), R_f = 0.3 (*n*-Pentane/EtOAc = 5/1). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 6.88 – 6.81 (m, 2H), 6.79 – 6.74 (m, 0.6H), 6.50 (s, 0.4H), 6.48 (s, 0.88H), 6.48 (s, 0.55H), 6.46 (s, 0.45H), 6.40 (s, 1.11H), 3.91 (s, 1.38H), 3.89 (s, 1.38H), 3.88 (s, 1.68H), 3.86 (s, 1.66H), 3.83 (s, 1.69H), 3.80 (s, 1.31H), 3.78 (s, 3H), 3.78 (s, 3H), 2.30 – 2.14 (m, 1H), 1.82 – 1.66 (m, 4H), 1.69 – 1.58 (m, 1H), 1.32 – 1.23 (m, 2H), 1.17 – 1.00 (m, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 205.5, 205.3, 153.2, 152.92, 152.90, 152.8, 150.3, 149.4, 148.7, 148.4, 139.2, 138.2, 137.0, 134.7, 133.4, 131.3, 124.9, 124.3, 122.8, 122.0, 112.8, 111.0, 110.6, 110.5, 106.9, 106.0, 61.0, 60.9, 56.12, 56.11, 55.92, 55.91, 55.90, 55.8, 50.5, 50.4, 29.0, 28.9, 25.8, 25.7. **HRMS** (ESI) calcd for $C_{26}H_{32}O_6$ $[M+H]^+$: 441.2277, Found: 441.2273.

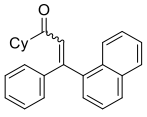
1-Cyclohexyl-3-(naphthalen-2-yl)-3-phenylprop-2-en-1-one (3ap):



3ap

Light cyan oil (27.9 mg, 82% yield, *E*/*Z* isomer: 50:50 based on NMR), R_f = 0.3 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.81 – 7.66 (m, 3H), 7.62 – 7.57 (m, 1H), 7.44 – 7.39 (m, 2H), 7.39 – 7.30 (m, 2H), 7.29 – 7.24 (m, 2H), 7.24 – 7.13 (m, 2H), 6.68 (s, 0.5H), 6.64 (s, 0.5H), 2.27 – 2.16 (m, 1H), 1.78 – 1.58 (m, 4H), 1.56 – 1.47 (m, 1H), 1.36 – 1.14 (m, 3H), 1.08 – 0.99 (m, 2H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 204.9, 204.7, 153.6, 153.4, 141.4, 139.2, 138.5, 136.7, 133.6, 133.1, 133.0, 129.5, 129.3, 128.7, 128.6, 128.42, 128.41, 128.21, 128.20, 128.0, 127.7, 127.62, 127.61, 127.4, 126.9, 126.41, 126.40, 126.2, 125.9, 125.6, 125.3, 51.0, 50.8, 28.72, 28.71, 25.82, 25.81, 25.7, 25.6. **HRMS** (ESI) calcd for $C_{25}H_{24}O$ $[M+H]^+$: 341.1905, Found: 341.1901.

1-Cyclohexyl-3-(naphthalen-1-yl)-3-phenylprop-2-en-1-one (3aq):

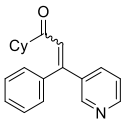


3aq

Light cyan oil (23.1 mg, 68% yield, *E*/*Z* isomer: 77:23 based on NMR), R_f = 0.3 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.91 – 7.87 (m, 2.26H), 7.68 – 7.65 (m, 0.74H), 7.54 – 7.48 (m, 1H), 7.47 – 7.27 (m, 8H), 7.00 (d, *J* = 0.8 Hz, 0.77H), 6.47 (d, *J* = 0.8 Hz, 0.23H), 2.36 (tt, *J* = 11.6, 3.6 Hz, 0.23H), 2.13 (tt, *J* = 11.6, 2.8 Hz, 0.77H), 1.96 – 1.74 (m, 1H), 1.68 – 1.49 (m, 4H), 1.23 – 1.19 (m, 2H), 1.11 – 0.78 (m, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 205.7, 204.1, 152.0, 151.6, 140.4, 140.1, 139.6, 136.9, 133.8, 133.5, 131.6, 131.2, 129.4, 129.3, 129.0, 128.8, 128.62, 128.61, 128.4, 128.3, 128.1, 127.51, 127.50, 127.3, 127.0, 126.6, 126.4, 126.3, 125.92, 125.91,

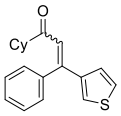
125.7, 125.6, 125.2, 125.1, 51.0, 50.5, 28.9, 28.7, 28.2, 25.8, 25.7, 25.6, 25.5. **HRMS** (ESI) calcd for $C_{25}H_{24}O$ $[M+H]^+$: 341.1905, Found: 341.1901.

1-Cyclohexyl-3-phenyl-3-(pyridin-3-yl)prop-2-en-1-one (3ar):



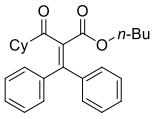
3ar Light cyan oil (17.9 mg, 61% yield, *E*/*Z* isomer: 50:50 based on NMR), R_f = 0.2 (*n*-Pentane/EtOAc = 10/1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.54 (s, 1.3H), 8.34 (s, 0.6H), 7.51 – 7.44 (m, 1H), 7.35 – 7.27 (m, 3H), 7.27 – 7.17 (m, 2H), 7.12 – 7.07 (m, 1H), 6.71 (s, 0.5H), 6.55 (s, 0.5H), 2.33 (tt, *J* = 11.2, 3.2 Hz, 0.5H), 2.19 (tt, *J* = 11.2, 3.2 Hz, 0.5H), 1.85 – 1.77 (m, 1H), 1.76 – 1.63 (m, 3H), 1.61 – 1.52 (m, 1H), 1.26 – 1.18 (m, 2H), 1.16 – 1.00 (m, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 204.5, 203.2, 150.4, 150.0, 149.3, 148.9, 148.8, 140.4, 138.0, 137.2, 135.7, 129.7, 129.3, 128.8, 128.6, 128.4, 128.2, 126.6, 125.7, 51.4, 50.8, 28.6, 28.4, 25.8, 25.7, 25.6. **HRMS** (ESI) calcd for $C_{20}H_{21}NO$ $[M+H]^+$: 292.1701, Found: 292.1702.

1-Cyclohexyl-3-phenyl-3-(thiophen-3-yl)prop-2-en-1-one (3as):



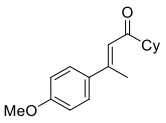
3as Light cyan oil (23.9 mg, 81% yield, *E*/*Z* isomer: 85:15 based on NMR), R_f = 0.2 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.36 – 7.33 (m, 2H), 7.32 – 7.26 (m, 2H), 7.24 – 7.22 (m, 1H), 7.20 – 7.15 (m, 2H), 6.98 (dd, *J* = 3.0, 1.5 Hz, 0.85H), 6.92 (dd, *J* = 4.8, 1.5 Hz, 0.15H), 6.62 (s, 0.85H), 6.48 (s, 0.15H), 2.32 – 2.26 (m, 0.15H), 2.21 – 2.12 (m, 0.9H), 1.76 – 1.64 (m, 4H), 1.56 – 1.51 (m, 1H), 1.29 – 1.12 (m, 2H), 1.13 – 0.99 (m, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 205.0, 204.5, 147.8, 143.1, 141.5, 139.1, 129.2, 128.7, 128.32, 128.31, 128.11, 128.10, 127.7, 126.8, 126.2, 125.8, 125.0, 123.5, 50.9, 50.7, 28.8, 28.7, 25.82, 25.81, 25.80, 25.7. **HRMS** (ESI) calcd for $C_{19}H_{20}OS$ $[M+H]^+$: 297.1313, Found: 297.1312.

1-Cycloheptyl-3,3-diphenylprop-2-en-1-one (3at):



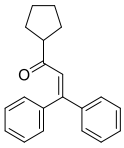
3at Colorless oil (27.0 mg, 69% yield), R_f = 0.2 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.41 – 7.29 (m, 6H), 7.24 – 7.11 (m, 4H), 3.97 (t, *J* = 6.6 Hz, 2H), 1.94 (tt, *J* = 11.4, 3.0 Hz, 1H), 1.74 – 1.62 (m, 4H), 1.57 – 1.46 (m, 1H), 1.39 – 1.26 (m, 4H), 1.18 – 1.04 (m, 3H), 0.98 – 0.87 (m, 2H), 0.80 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 207.0, 167.3, 151.7, 140.3, 139.6, 134.3, 130.0, 129.5, 129.2, 129.0, 128.4, 128.2, 65.0, 50.8, 30.1, 28.8, 25.7, 25.6, 18.8, 13.6. **HRMS** (ESI) calcd for $C_{26}H_{30}O_3$ $[M+H]^+$: 391.2273, Found: 391.2270.

1-Cyclohexyl-3-(4-methoxyphenyl)but-2-en-1-one (3au):



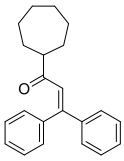
3au Light cyan oil (11.0 mg, 43% yield), R_f = 0.3 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.47 (dt, *J* = 9.2, 2.4 Hz, 2H), 6.90 (dt, *J* = 8.8, 2.4 Hz, 1H), 6.53 (q, *J* = 1.2 Hz, 1H), 3.84 (s, 3H), 2.52 (d, *J* = 1.2 Hz, 3H), 2.43 (tt, *J* = 11.2, 3.2 Hz, 1H), 1.94 – 1.85 (m, 2H), 1.85 – 1.77 (m, 2H), 1.70 – 1.66 (m, 1H), 1.41 – 1.23 (m, 5H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 204.5, 160.4, 153.6, 134.9, 127.9, 122.0, 113.9, 55.4, 52.2, 28.8, 26.0, 25.9, 18.2. **HRMS** (ESI) calcd for $C_{17}H_{22}O_2$ $[M+H]^+$: 259.1698, Found: 259.1700.

1-Cycloheptyl-3,3-diphenylprop-2-en-1-one (3ba):



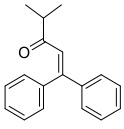
3ba Light cyan oil (20.4 mg, 74% yield), R_f = 0.4 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.44 – 7.29 (m, 8H), 7.24 – 7.15 (m, 2H), 6.65 (s, 1H), 2.91 – 2.76 (m, 1H), 1.82 – 1.48 (m, 8H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 204.2, 153.3, 141.3, 139.1, 129.4, 129.2, 128.4, 128.32, 128.31, 128.1, 125.8, 51.7, 29.4, 26.1. **HRMS** (ESI) calcd for $C_{20}H_{20}O$ $[M+H]^+$: 277.1592, Found: 277.1593.

1-Cycloheptyl-3,3-diphenylprop-2-en-1-one (3ca):



3ca Light cyan oil (10.7 mg, 35% yield), R_f = 0.3 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40 – 7.28 (m, 8H), 7.21 – 7.15 (m, 2H), 6.60 (s, 1H), 2.43 (tt, *J* = 10.0, 4.0 Hz, 1H), 1,85 – 1,78 (m, 2H), 1.70 – 1.62 (m, 2H), 1.58 – 1.49 (m, 2H), 1.49 – 1.42 (m, 4H), 1.34 – 1.26 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 205.4, 153.3, 141.3, 139.2, 129.4, 129.2, 128.4, 128.3, 128.2, 125.8, 52.0, 30.2, 28.2, 26.8. **HRMS** (ESI) calcd for $C_{22}H_{24}O$ $[M+H]^+$: 305.1905, Found: 305.1905.

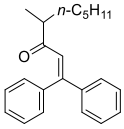
4-Methyl-1,1-diphenylpent-1-en-3-one (3da):



3da Light cyan oil (14.6 mg, 58% yield), R_f = 0.5 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.39 – 7.29 (m, 8H), 7.21 – 7.16 (m, 2H), 6.64 (s, 1H), 2.61 – 2.52 (m, 1H), 1.06 (d, *J* = 6.9 Hz, 6H). **^{13}C NMR** (75

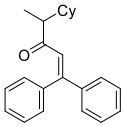
MHz, $CDCl_3$) δ 205.4, 153.6, 141.3, 139.1, 129.4, 129.2, 128.41, 128.40, 128.1, 125.0, 40.9, 18.4. **HRMS** (ESI) calcd for $C_{18}H_{18}O$ $[M+H]^+$: 251.1436, Found: 251.1433.

4-Methyl-1,1-diphenylnon-1-en-3-one (3ea):



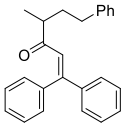
3ea Light cyan oil (23.1 mg, 69% yield), R_f = 0.4 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.39 – 7.28 (m, 8H), 7.19 – 7.16 (m, 2H), 6.62 (s, 1H), 2.49 – 2.41 (m, 1H), 1.69 – 1.59 (m, 1H), 1.30 – 1.16 (m, 7H), 1.02 (d, *J* = 7.2 Hz, 3H), 0.87 (t, *J* = 6.8 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 205.5, 153.5, 141.3, 139.1, 129.4, 129.2, 128.41, 128.40, 128.1, 125.5, 46.2, 33.3, 31.8, 27.0, 22.5, 16.3, 14.0. **HRMS** (ESI) calcd for $C_{22}H_{26}O$ $[M+H]^+$: 307.2062, Found: 307.2062.

4-Cyclohexyl-1,1-diphenylpent-1-en-3-one (3fa):



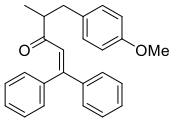
3fa Yellow oil (19.6 mg, 62% yield), R_f = 0.3 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.39 – 7.30 (m, 8H), 7.20 – 7.16 (m, 2H), 6.62 (s, 1H), 2.37 – 2.28 (m, 1H), 1.78 – 1.52 (m, 7H), 1.28 – 1.05 (m, 4H), 0.96 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 205.2, 153.3, 141.4, 139.2, 129.4, 129.2, 128.41, 128.40, 128.3, 128.1, 125.9, 51.9, 40.5, 31.8, 29.3, 26.5, 26.4, 12.9. **HRMS** (ESI) calcd for $C_{23}H_{26}O$ $[M+H]^+$: 319.2062, Found: 319.2062.

4-Methyl-1,1,6-triphenylhex-1-en-3-one (3ga):



3ga Yellow oil (30.4 mg, 89% yield), R_f = 0.3 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.31 – 7.23 (m, 6H), 7.23 – 7.16 (m, 4H), 7.12 – 7.04 (m, 5H), 6.52 (s, 1H), 2.49 – 2.37 (m, 3H), 1.97 – 1.84 (m, 1H), 1.58 – 1.48 (m, 1H), 1.00 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 204.8, 154.0, 141.9, 141.2, 139.0, 129.4, 129.3, 128.42, 128.41, 128.40, 128.3, 128.1, 125.8, 125.3, 45.6, 34.8, 33.5, 16.4. **HRMS** (ESI) calcd for $C_{25}H_{24}O$ $[M+H]^+$: 341.1905, Found: 341.1910.

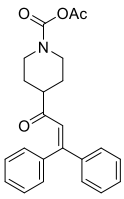
5-(4-Methoxyphenyl)-4-methyl-1,1-diphenylpent-1-en-3-one (3ha):



3ha

Light cyan liquid (14.3 mg, 40% yield), R_f = 0.2 (*n*-Pentane/EtOAc = 50/1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.41 – 7.28 (m, 6H), 7.20 – 7.12 (m, 4H), 6.97 – 6.94 (m, 2H), 6.82 – 6.79 (m, 2H), 6.45 (s, 1H), 3.79 (s, 3H), 2.84 (dd, *J* = 13.6, 6.8 Hz, 1H), 2.74 – 2.68 (m, 1H), 2.49 (dd, *J* = 13.2, 7.6 Hz, 1H), 1.00 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 205.5, 158.0, 153.4, 141.2, 139.1, 131.9, 123.0, 129.5, 129.2, 128.51, 128.50, 128.3, 128.2, 126.1, 113.7, 55.2, 48.1, 38.7, 16.5. **HRMS** (ESI) calcd for $C_{25}H_{24}O_2$ $[M+H]^+$: 357.1855, Found: 357.1853.

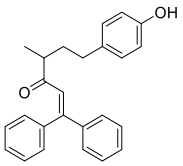
Acetic 4-(3,3-diphenylacryloyl)piperidine-1-carboxylic anhydride (3ia):



3ia

Light cyan liquid (27.0 mg, 74% yield), R_f = 0.4 (*n*-Pentane/EtOAc = 10/1). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.40 – 7.28 (m, 8H), 7.20 – 7.16 (m, 2H), 6.61 (s, 1H), 4.17 – 4.12 (m, 2H), 4.09 (t, *J* = 7.2 Hz, 2H), 2.71 – 2.58 (m, 2H), 2.36 (tt, *J* = 11.1, 3.6 Hz, 1H), 1.75 – 1.69 (m, 2H), 1.58 – 1.45 (m, 2H), 1.23 (t, *J* = 6.9 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 202.7, 155.4, 154.5, 140.8, 139.0, 129.5, 129.3, 128.6, 128.41, 128.40, 128.2, 124.7, 61.2, 48.2, 43.3, 27.6, 14.6. **HRMS** (ESI) calcd for $C_{23}H_{25}NO_3$ $[M+H]^+$: 364.1913, Found: 364.1912.

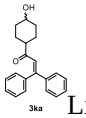
6-(4-Hydroxyphenyl)-4-methyl-1,1-diphenylhex-1-en-3-one (3ja):



3ja

Light cyan liquid (16.6 mg, 47% yield), R_f = 0.4 (*n*-Pentane/EtOAc = 5/1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.39 – 7.28 (m, 8H), 7.20 – 7.14 (m, 2H), 6.98 (d, *J* = 8.4 Hz, 2H), 6.71 (dt, *J* = 8.4, 2.4 Hz, 2H), 6.61 (d, *J* = 0.8 Hz, 1H), 2.54 – 2.40 (m, 3H), 2.00 – 1.90 (m, 1H), 1.61 – 1.52 (m, 1H), 1.09 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 205.6, 205.5, 154.3, 153.9, 153.9, 141.2, 139.0, 133.9, 133.8, 129.41, 129.40, 128.52, 128.51, 128.4, 128.2, 125.3, 115.3, 45.6, 35.2, 32.6, 16.5. **HRMS** (ESI) calcd for $C_{25}H_{24}O_2$ $[M+H]^+$: 357.1855, Found: 357.1858.

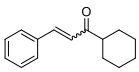
1-(4-Hydroxycyclohexyl)-3,3-diphenylprop-2-en-1-one (3ka):



Light cyan liquid (18.7 mg, 61% yield).

Major isomer: R_f = 0.2 (*n*-Pentane/EtOAc = 2/1), **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40 – 7.28 (m, 8H), 7.20 – 7.16 (m, 2H), 6.61 (s, 1H), 3.53 (tt, *J* = 8.1, 3.3 Hz, 1H), 2.19 (tt, *J* = 8.4, 3.6 Hz, 1H), 2.02 – 1.93 (m, 2H), 1.88 – 1.79 (m, 2H), 1.44 – 1.34 (m, 2H), 1.14 – 1.04 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) 204.3, 154.0, 141.0, 139.0, 129.4, 129.3, 128.5, 128.41, 128.40, 128.2, 125.3, 70.0, 49.4, 34.7, 26.9. **HRMS** (ESI) calcd for $C_{21}H_{22}O_2$ $[M+H]^+$: 307.1698, Found: 307.1697.

Minor isomer: R_f = 0.3 (*n*-Pentane/EtOAc = 2/1), **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.40 – 7.28 (m, 8H), 7.21 – 7.16 (m, 2H), 6.64 (s, 1H), 3.90 (tt, *J* = 4.5, 3.0 Hz, 1H), 2.29 (tt, *J* = 9.9, 3.6 Hz, 1H), 1.89 – 1.67 (m, 4H), 1.63 – 1.54 (m, 2H), 1.51 – 1.40 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 204.0, 153.7, 141.2, 139.1, 129.4, 129.3, 128.42, 128.41, 128.1, 125.0, 66.4, 49.1, 32.1, 22.9. **HRMS** (ESI) calcd for $C_{21}H_{22}O_2$ $[M+H]^+$: 307.1698, Found: 307.1700.

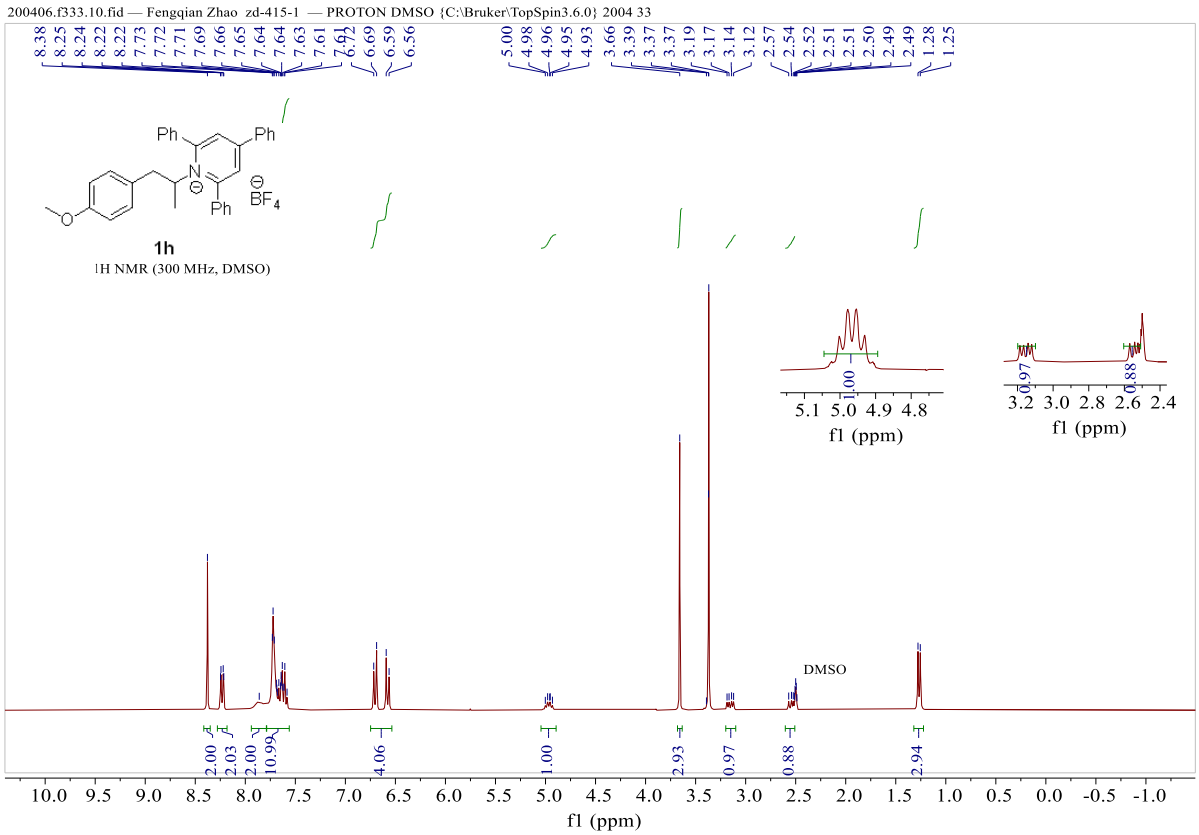


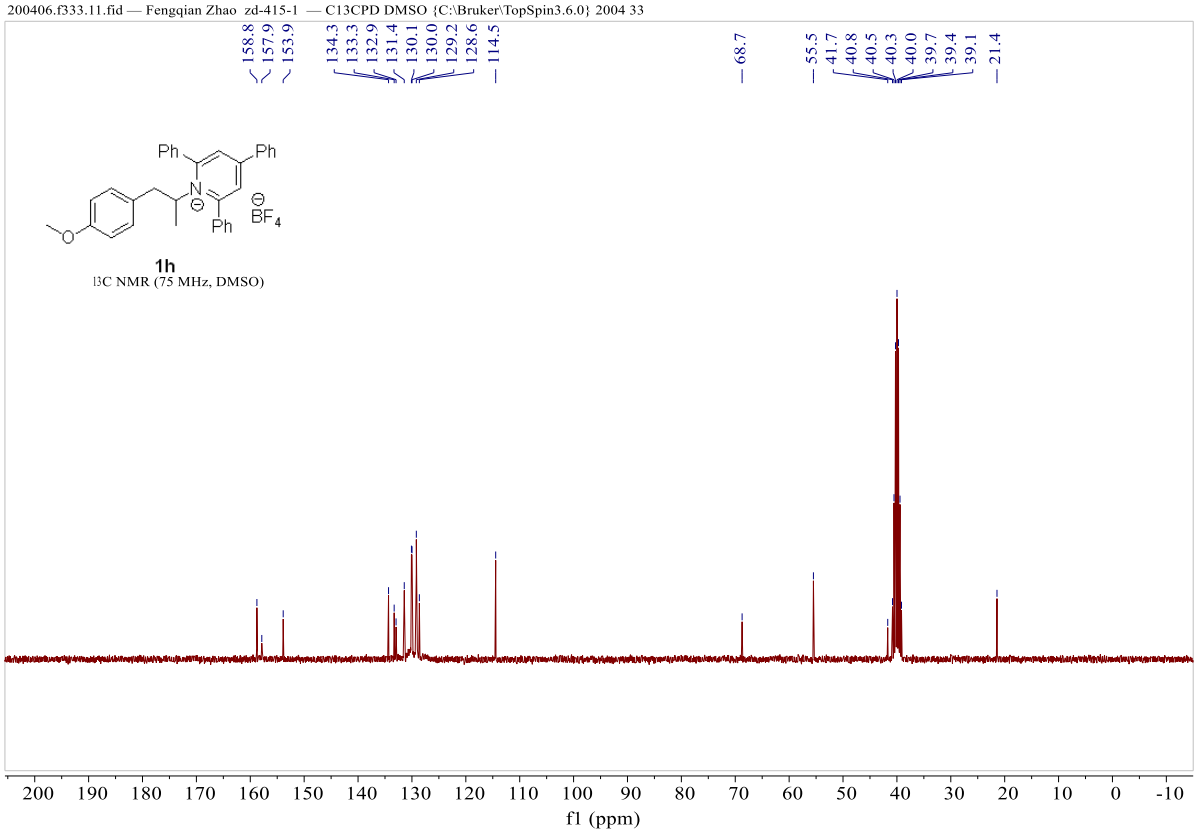
GC-MS (EI, 70 eV): m/z (%) = 77 (20), 103 (40), 131 (100), 214 (15).

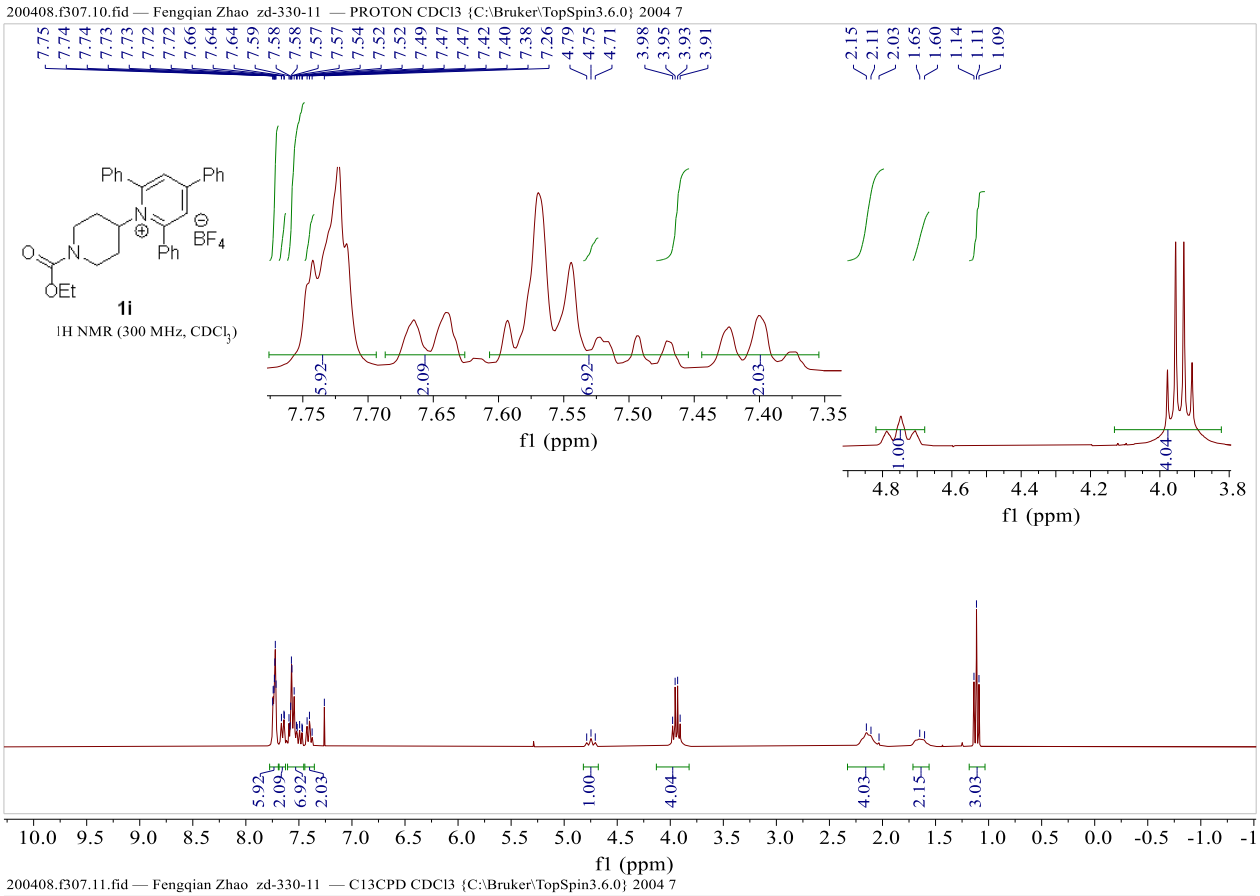
5. Reference:

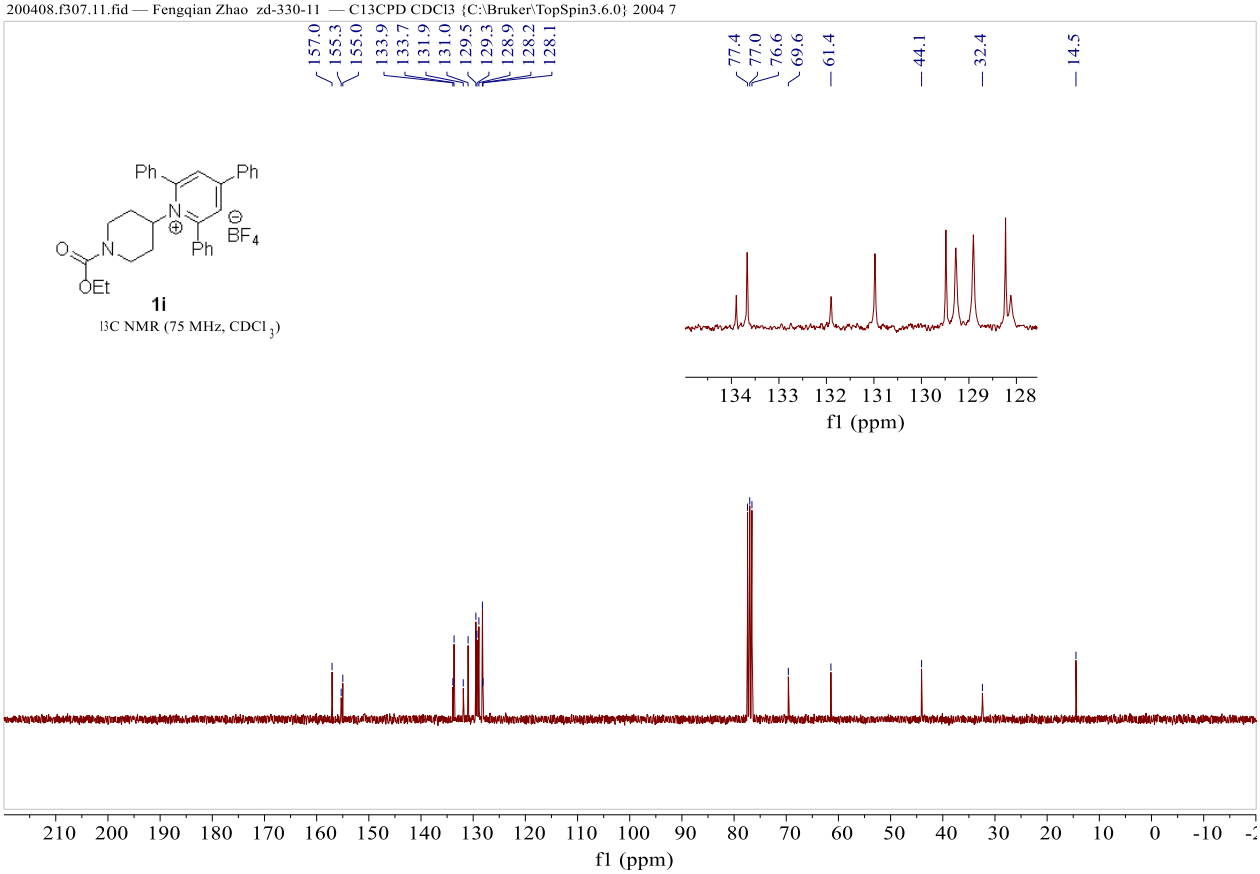
1. a) C. H. Basch, J. Liao, J. Xu, J. J. Piane and M. P. Watson, *J. Am. Chem. Soc.*, 2017, **139**, 5313; b) F. J. R. Klauck, M. J. James and F. Glorius, *Angew. Chem. Int. Ed.*, 2017, **56**, 12336; c) S. Plunkett, C. H. Basch, S. O. Santana and M. P. Watson, *J. Am. Chem. Soc.*, 2019, **141**, 2257; d) F. Sandfort, F. Strieth-Kalthoff, F. J. R. Klauck, M. J. James and F. Glorius, *Chem. Eur. J.*, 2018, **24**, 17210; d) S. A. Said and A. Fiksdahl, *Tetrahedron Asymm.*, 2001, **12**, 1947.
2. T. Wang, Y. Hu and S. Zhang, *Org. Biomol. Chem.*, 2010, **8**, 2312.
3. D. Ganapathy and G. Sekar, *Org. Lett.*, 2014, **16**, 3856.
4. H. Tan, I. Houpis, R. Liu, Y. Wang and Z. Chen, *Org. Lett.*, 2015, **17**, 3548.
5. J. C. L. Walker and M. Oestreich, *Org. Lett.*, 2018, **20**, 6411.
6. T. Vom Stein, M. Perez, R. Dobrovetsky, D. Winkelhaus, C. B. Caputo and D. W. Stephan, *Angew. Chem. Int. Ed.*, 2015, **54**, 10178.
7. Z. Cheng, W. Jin and C. Liu, *Org. Chem. Front.*, 2019, **6**, 841.
8. M. D. Ritz, A. M. Parsons, P. N. Palermo and W. D. Jones, *Polyhedron*, 2020, **180**, 114416.
9. C. Lei, Y. J. Yip and J. S. Zhou, *J. Am. Chem. Soc.*, 2017, **139**, 6086.
10. S. Rao, M. N. Joy and K. R. Prabhu, *J. Org. Chem.*, 2018, **83**, 13707.
11. D. Xu, C. Lu and W. Chen, *Tetrahedron*, 2012, **68**, 1466.

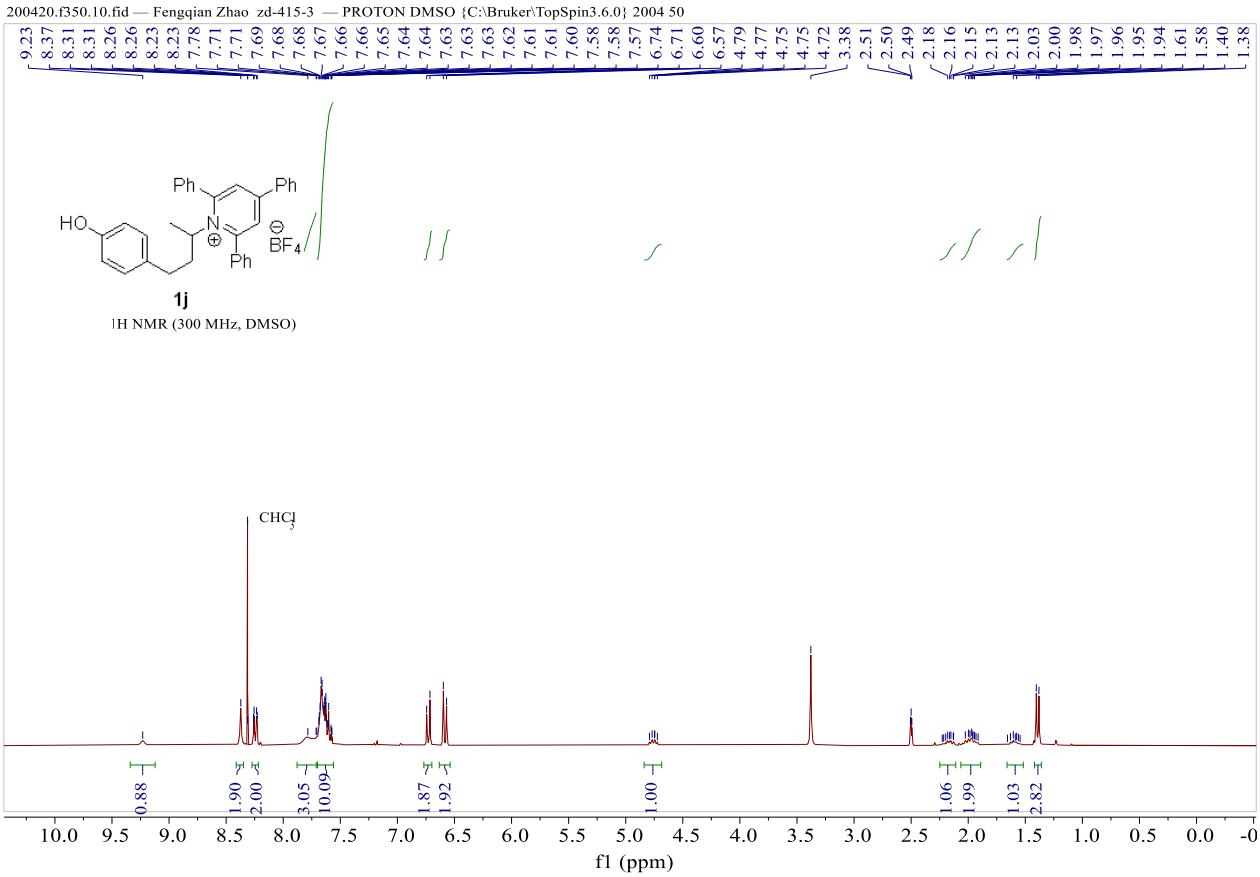
6. NMR Spectra of products: ^{1}H, ^{13}C and ^{19}F NMR

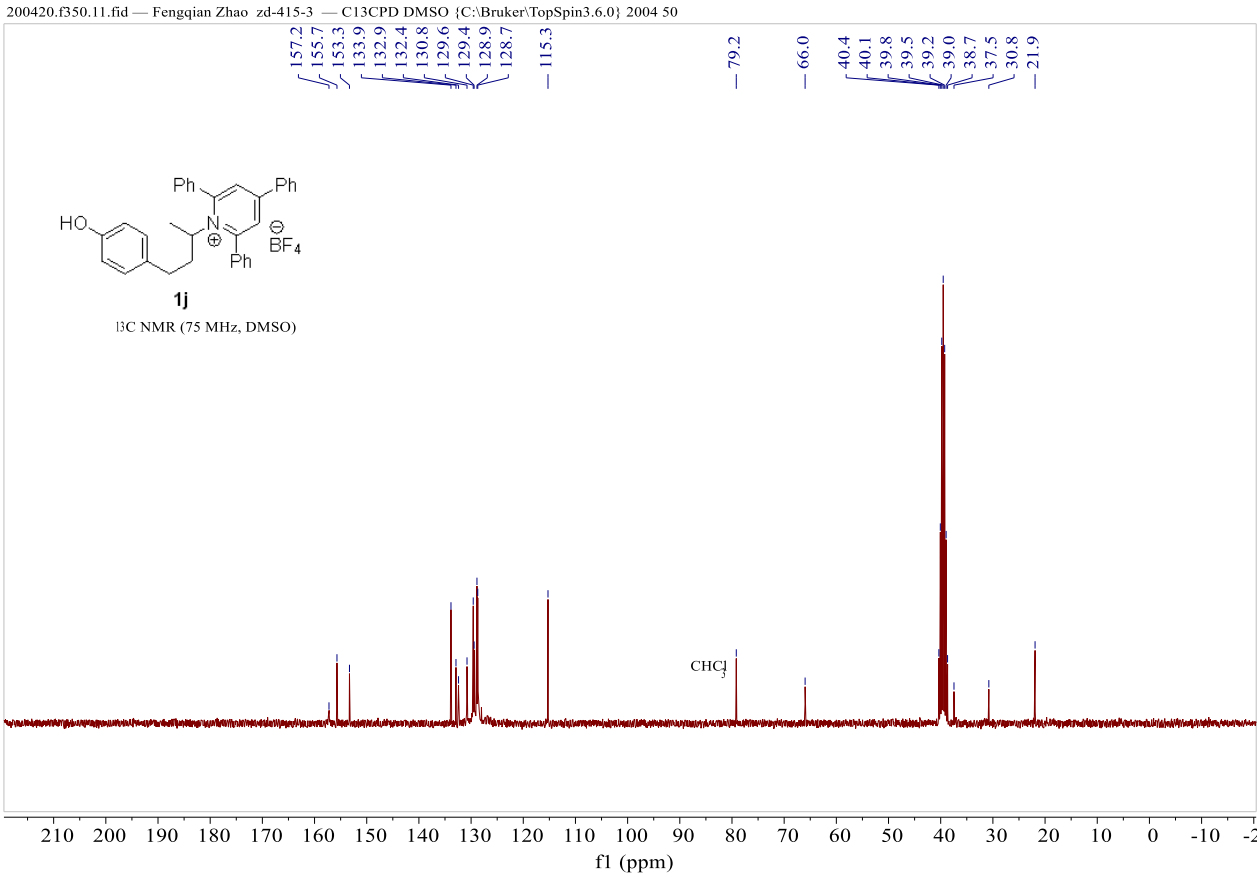


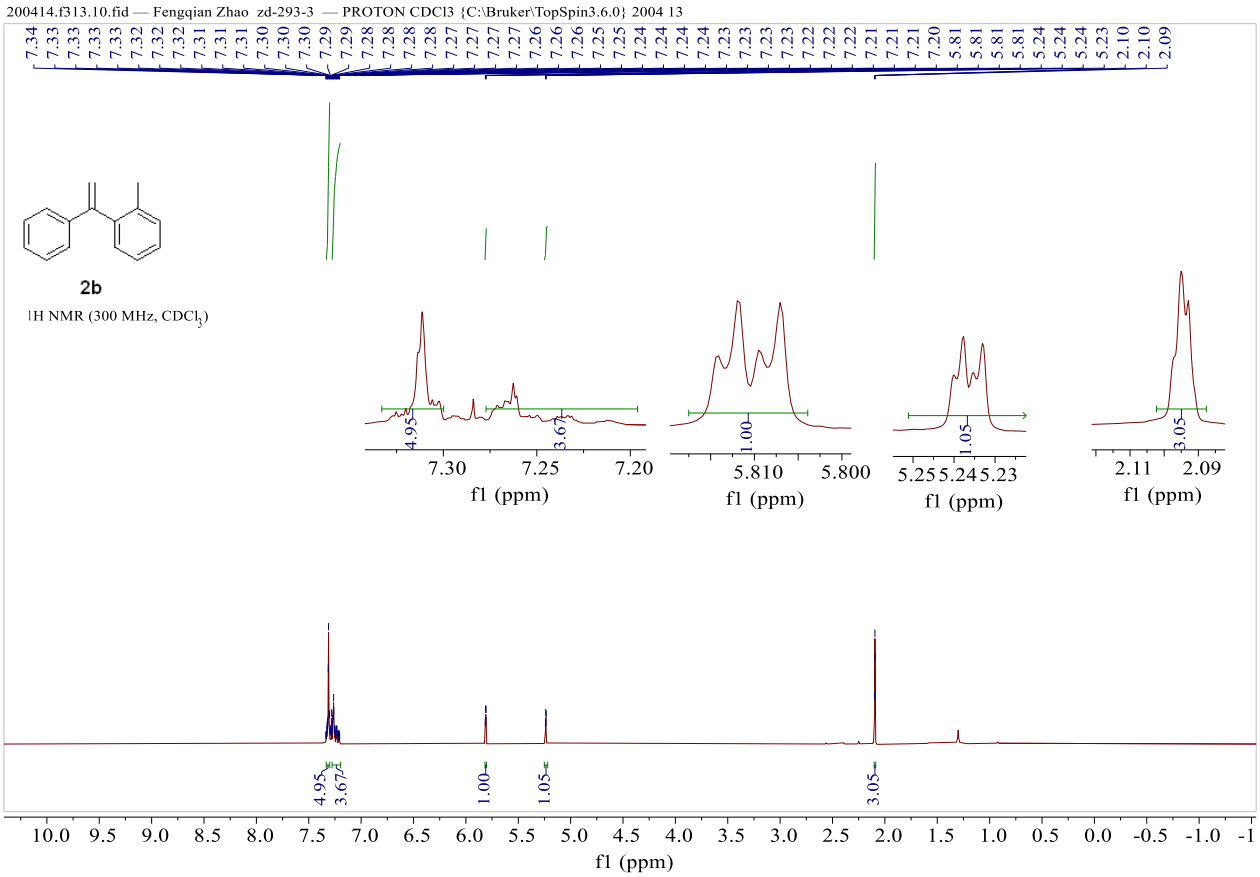


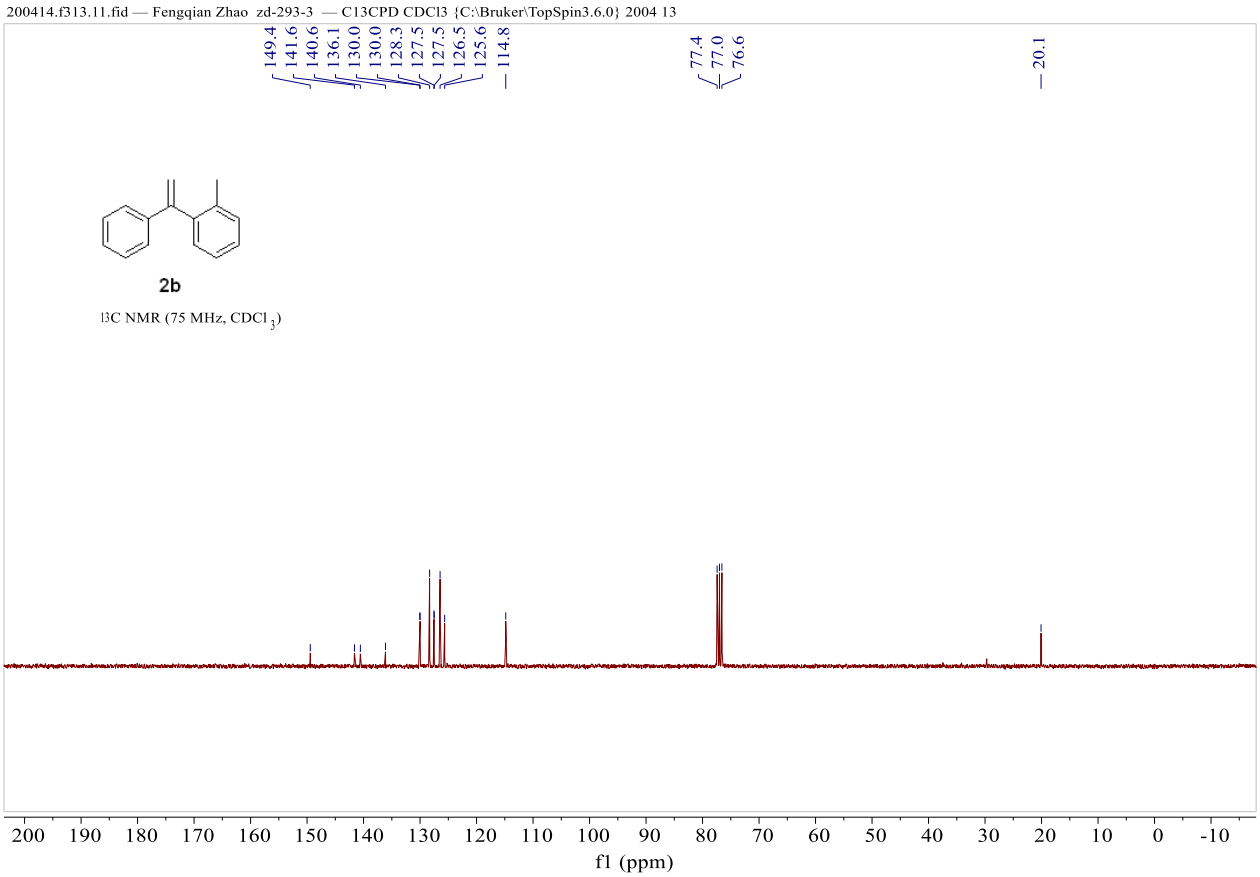

200408.f307.10.fid — Fengqian Zhao zd-330-11 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2004 7
7.75 7.74 7.74 7.73 7.73 7.72 7.72 7.66 7.64 7.64 7.59 7.58 7.58 7.57 7.57 7.54 7.52 7.52 7.49 7.47 7.47 7.42 7.40 7.38 7.26 4.79 4.75 4.71 3.98 3.95 3.93 3.91 2.15 2.11 2.03 1.65 1.60 1.14 1.11 1.09
Ph Ph N BF4 Ph O N OEt
1i
1H NMR (300 MHz, CDCl3)
5.92 2.09 6.92 2.03
7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35
f1 (ppm)
1.00 4.04
4.8 4.6 4.4 4.2 4.0 3.8
f1 (ppm)
5.92 2.09 6.92 2.03 1.00 4.04 4.03 2.15 3.03
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1
f1 (ppm)


200408.f307.11.fid — Fengqian Zhao zd-330-11 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2004 7
157.0 155.3 155.0 133.9 133.7 131.9 131.0 129.5 129.3 128.9 128.2 128.1 77.4 77.0 76.6 69.6 61.4 44.1 32.4 14.5
Ph Ph N BF4 Ph O N OEt
1i
13C NMR (75 MHz, CDCl3)
134 133 132 131 130 129 128
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -2
f1 (ppm)

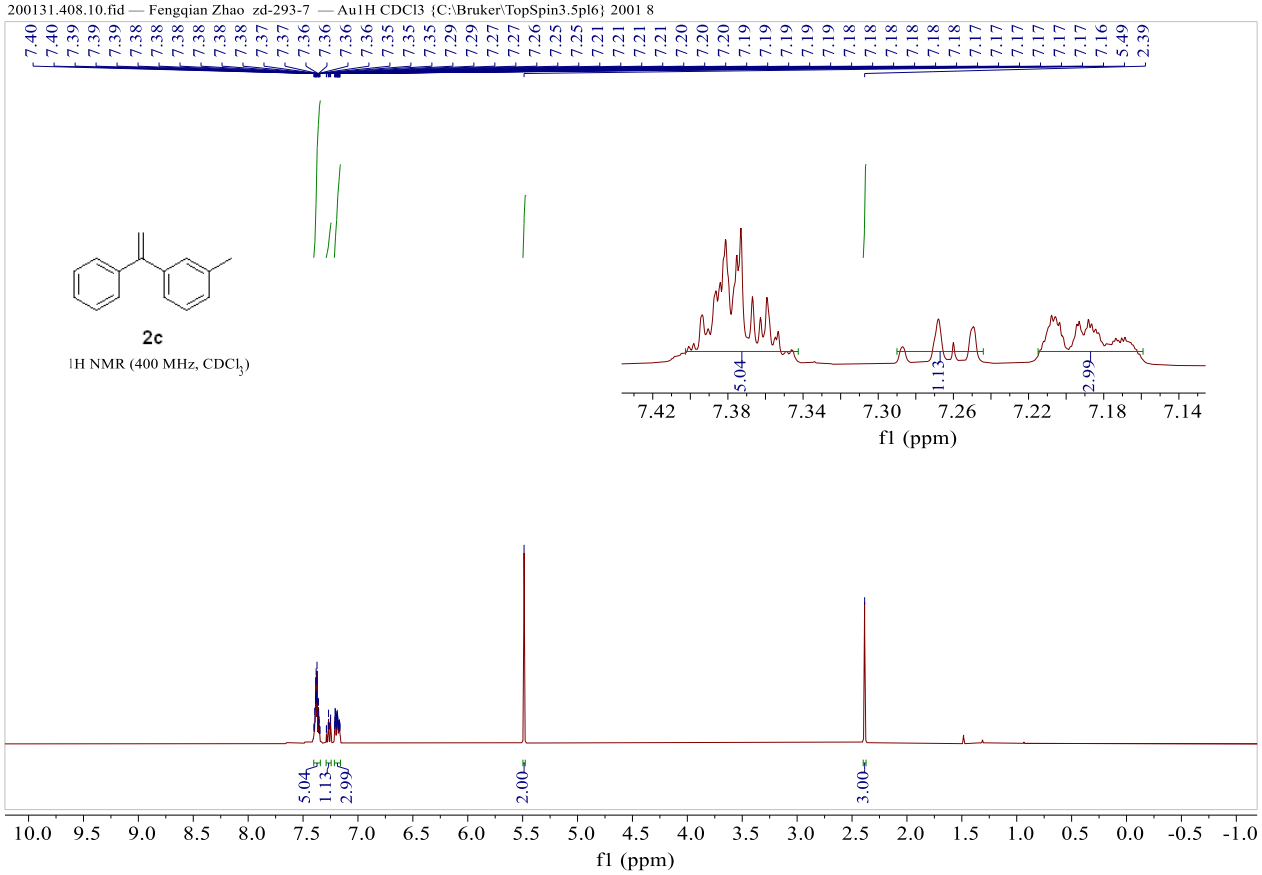

200420.f350.10.fid — Fengqian Zhao zd-415-3 — PROTON DMSO {C:\Bruker\TopSpin3.6.0} 2004 50
Ph
Ph
HO
N
BF4
Ph
1j
1H NMR (300 MHz, DMSO)
CHCl3
0.88
1.90
2.00
3.05
10.09
1.87
1.92
1.00
1.06
1.99
1.03
2.82
f1 (ppm)


200420.f350.11.fid — Fengqian Zhao zd-415-3 — C13CPD DMSO {C:\Bruker\TopSpin3.6.0} 2004 50
157.2
155.7
153.3
133.9
132.9
132.4
130.8
129.6
129.4
128.9
128.7
115.3
79.2
66.0
40.4
40.1
39.8
39.5
39.2
39.0
38.7
37.5
30.8
21.9
Ph
Ph
HO
N
BF4
Ph
1j
13C NMR (75 MHz, DMSO)
CHCl3
f1 (ppm)

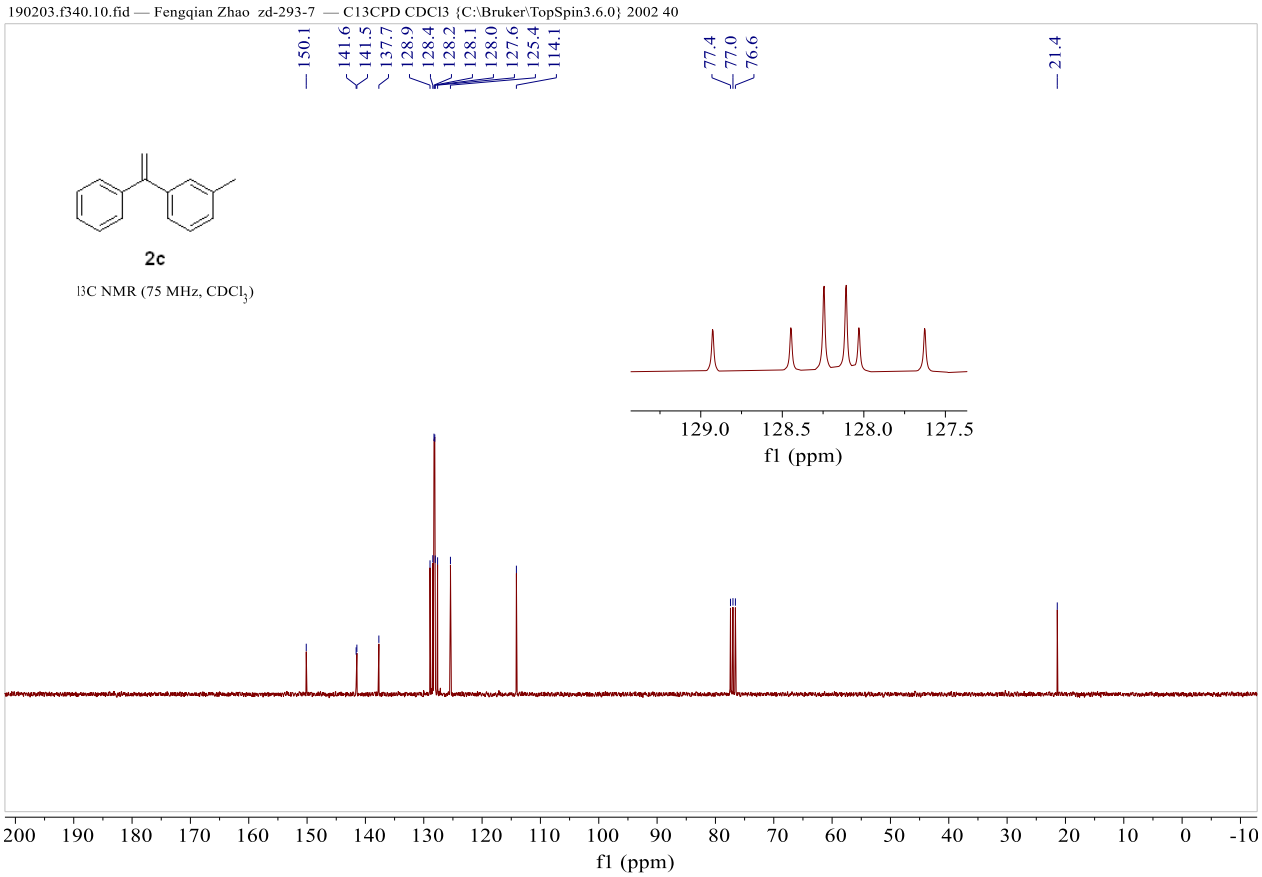

200414.f313.10.fid — Fengqian Zhao zd-293-3 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2004 13
2b
1H NMR (300 MHz, CDCl3)
f1 (ppm)


200414.f313.11.fid — Fengqian Zhao zd-293-3 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2004 13
2b
13C NMR (75 MHz, CDCl3)
f1 (ppm)

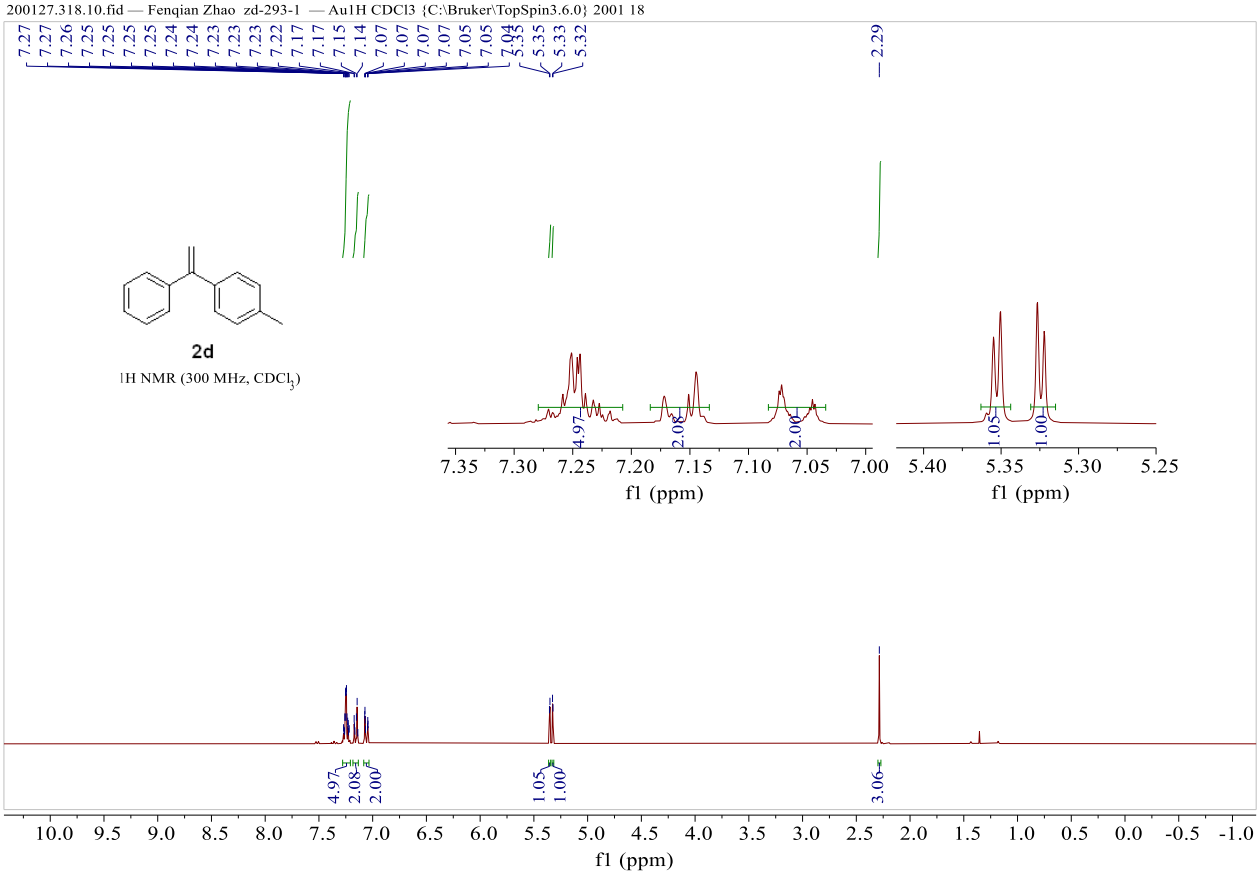
200131.408.10.fid — Fengqian Zhao zd-293-7 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2001 8



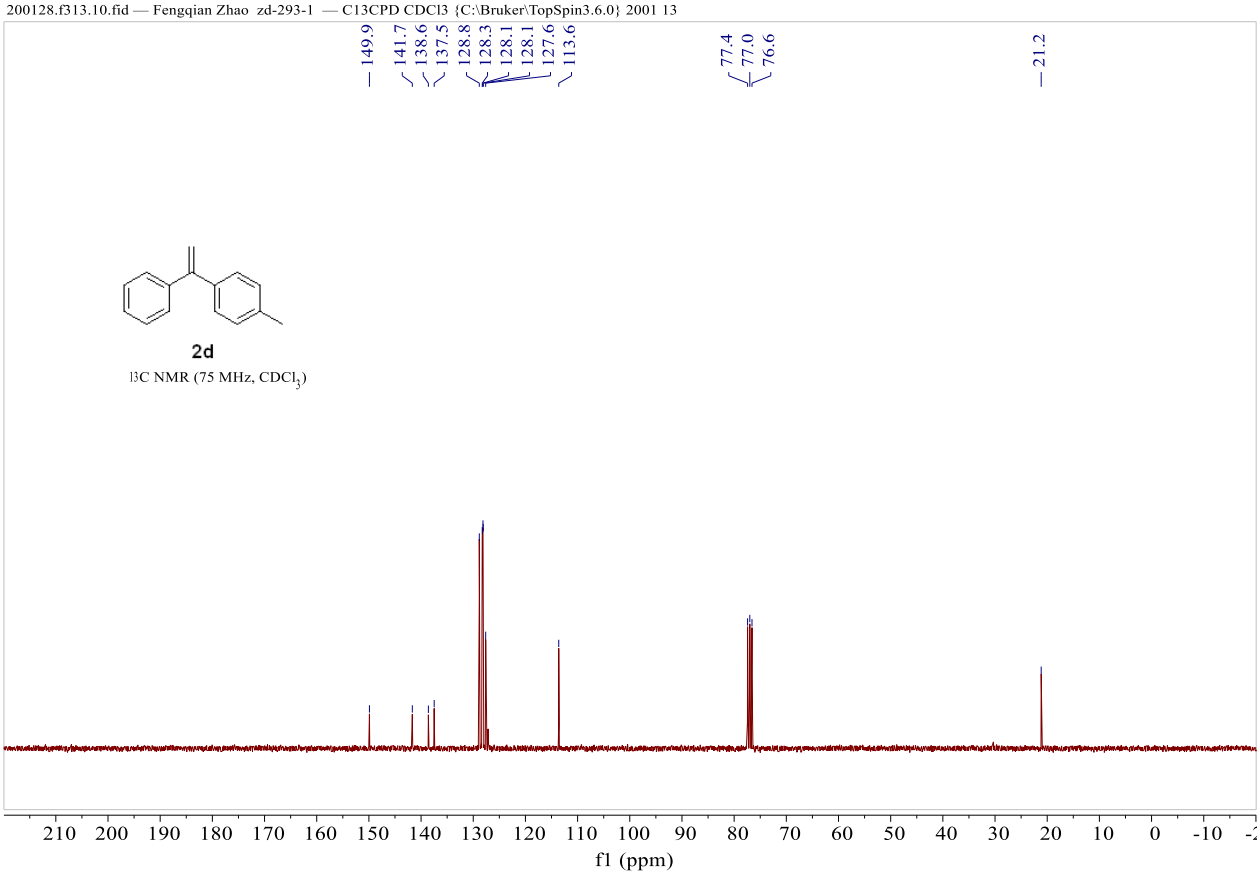
190203.f340.10.fid — Fengqian Zhao zd-293-7 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 40



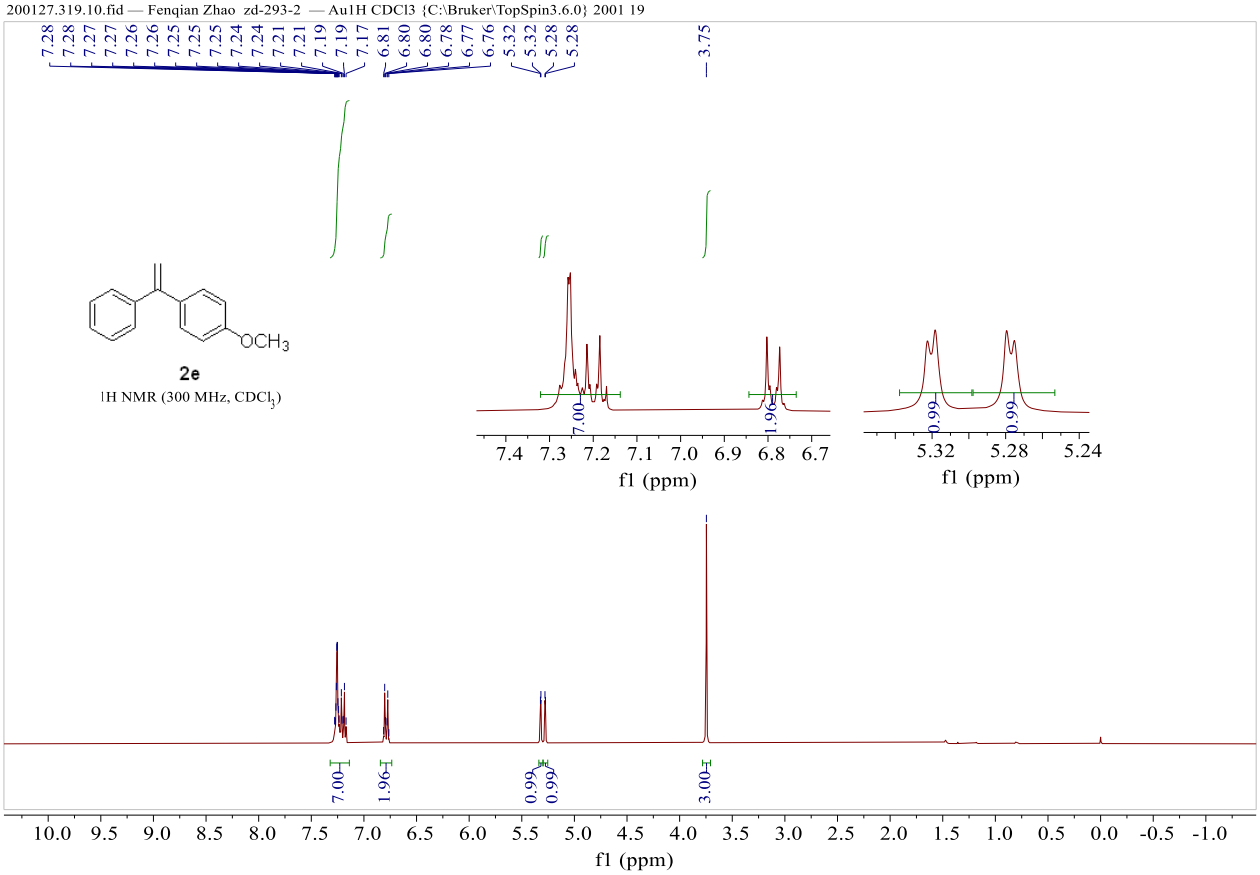
200127.318.10.fid — Fenqian Zhao zd-293-1 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.0} 2001 18



200128.f313.10.fid — Fengqian Zhao zd-293-1 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2001 13



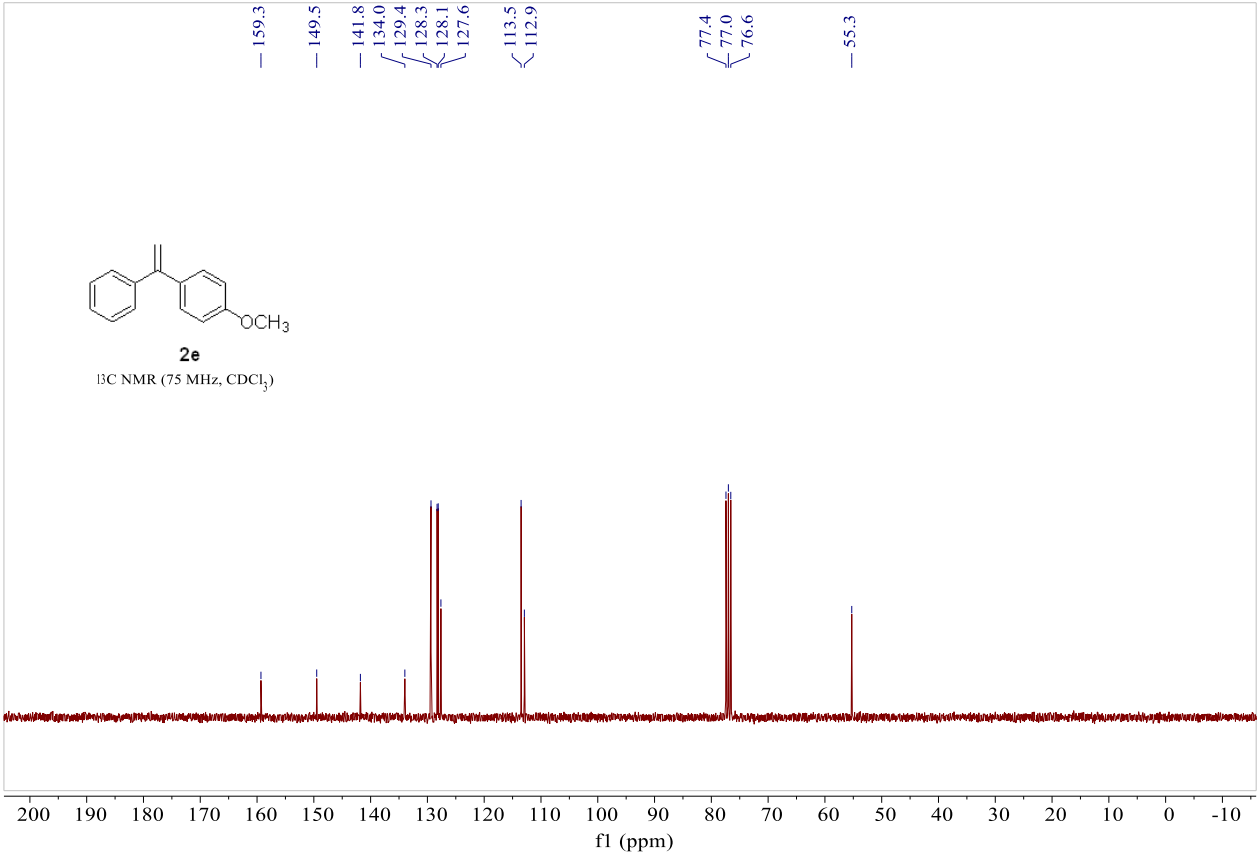
200127.319.10.fid — Fenqian Zhao zd-293-2 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.0} 2001 19



2e

^{1}H NMR (300 MHz, $CDCl_3$)

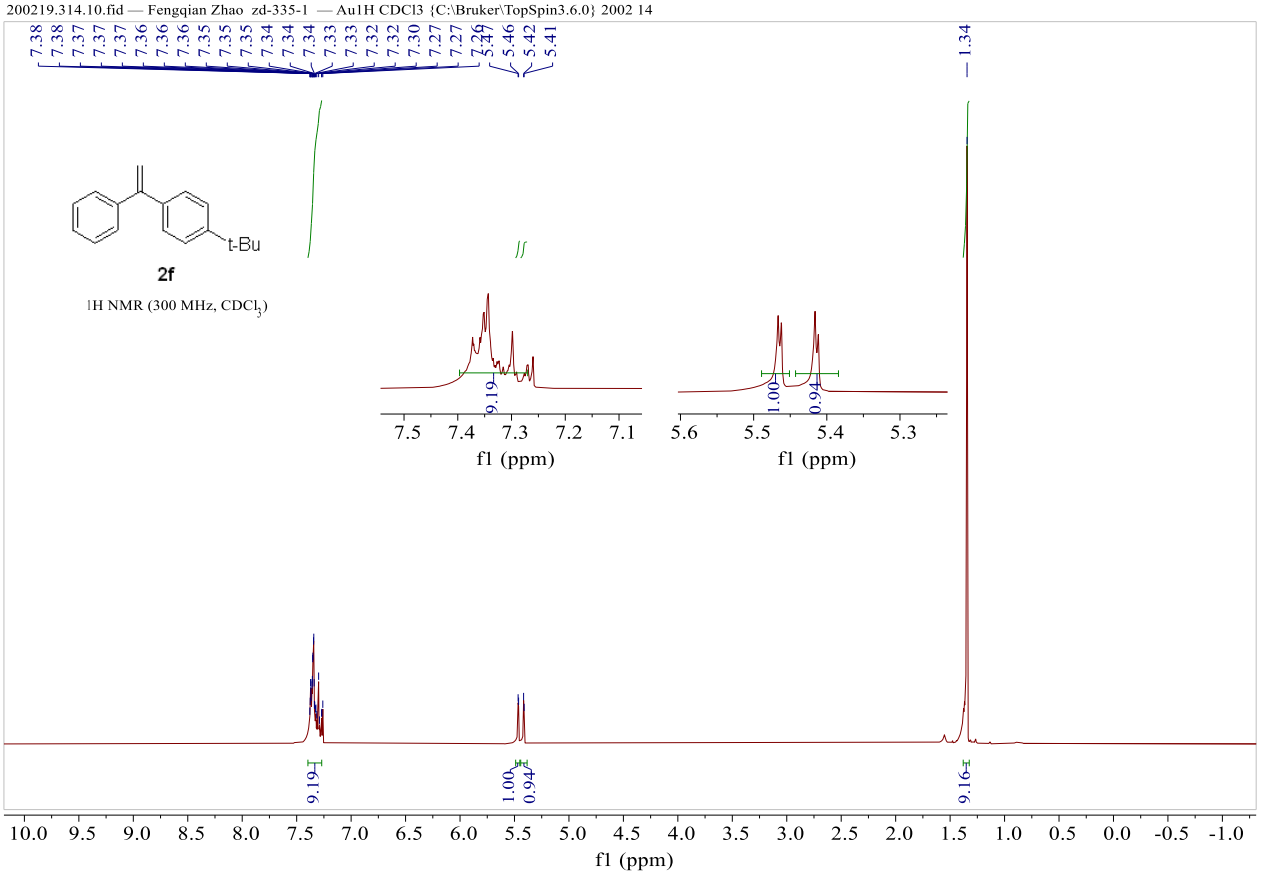
200128.f314.10.fid — Fengqian Zhao zd-293-2 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2001 14



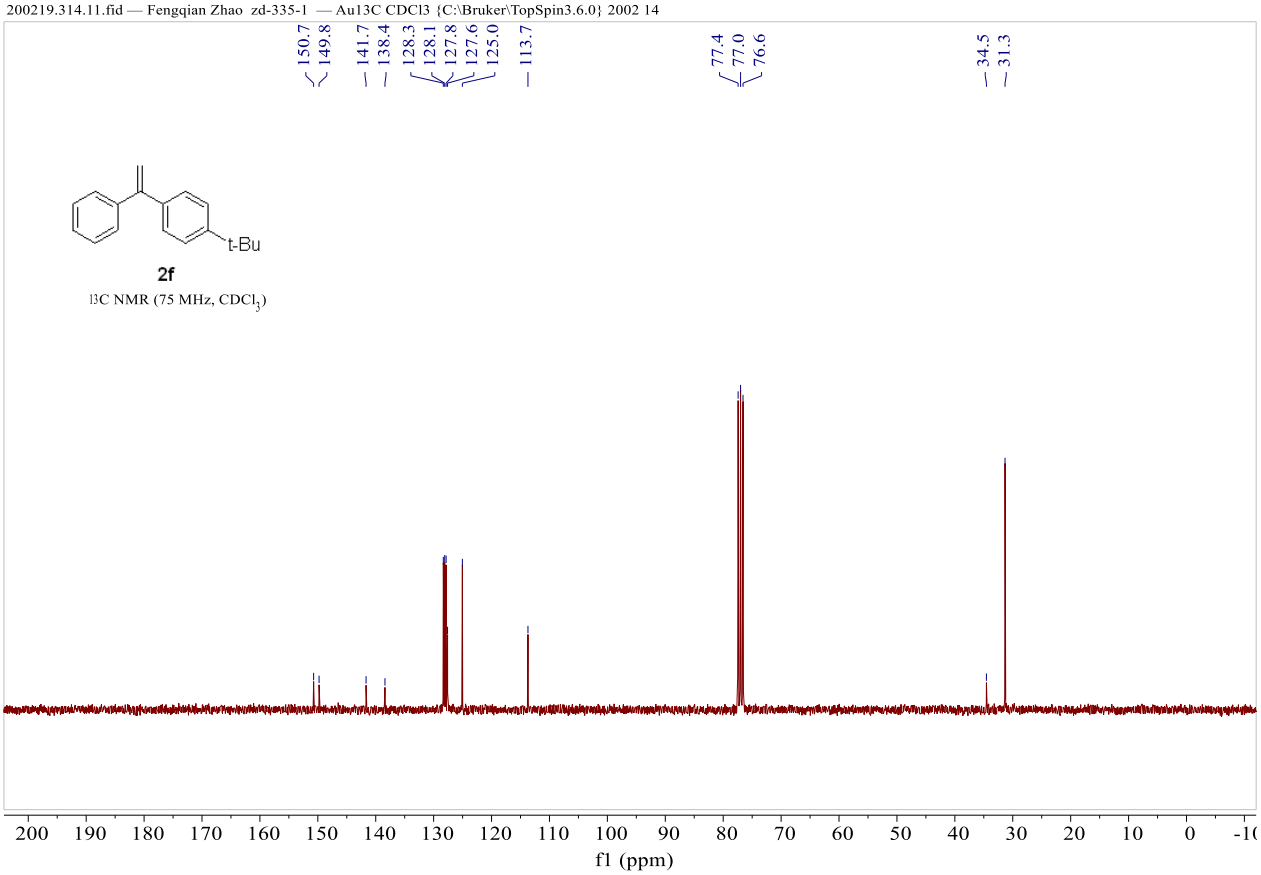
2e

^{13}C NMR (75 MHz, $CDCl_3$)

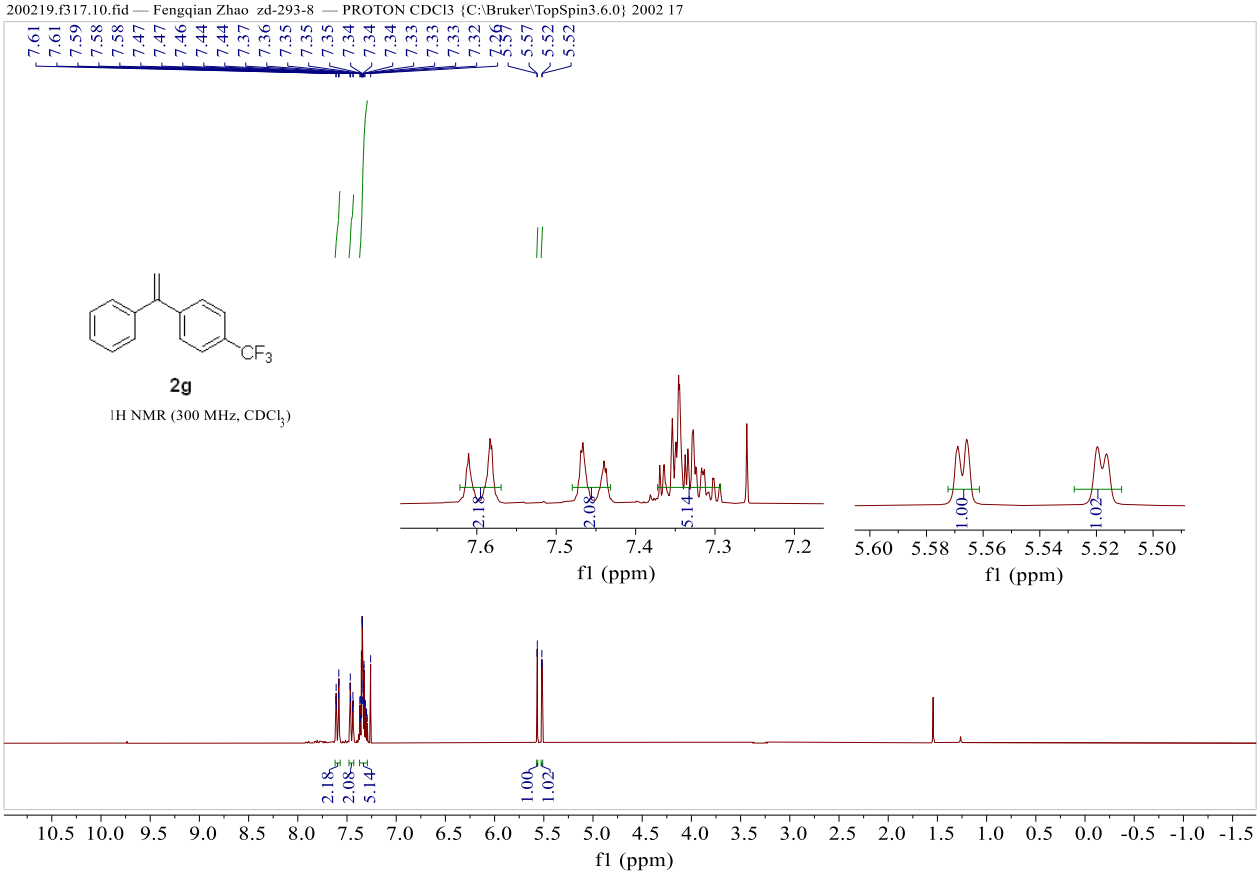
200219.314.10.fid — Fengqian Zhao zd-335-1 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 14



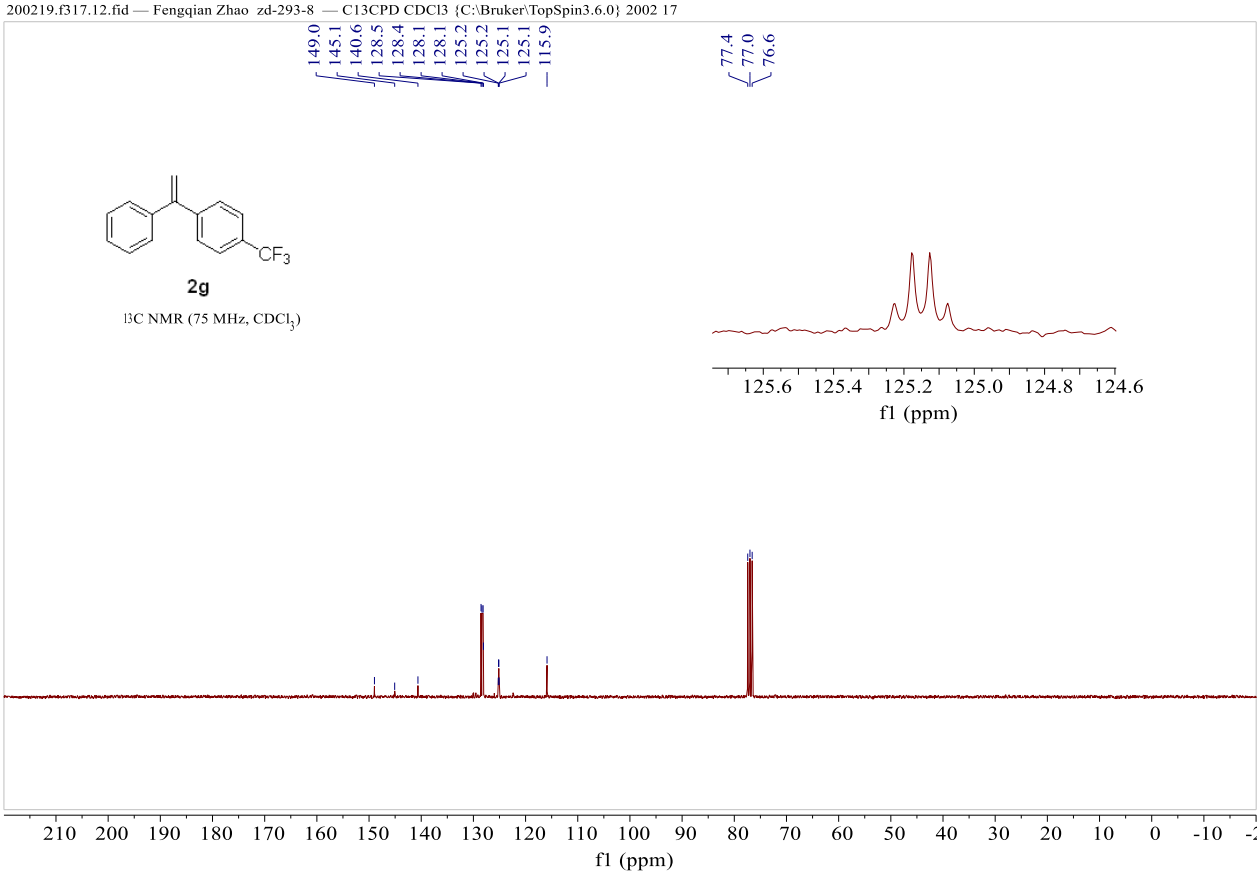
200219.314.11.fid — Fengqian Zhao zd-335-1 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 14



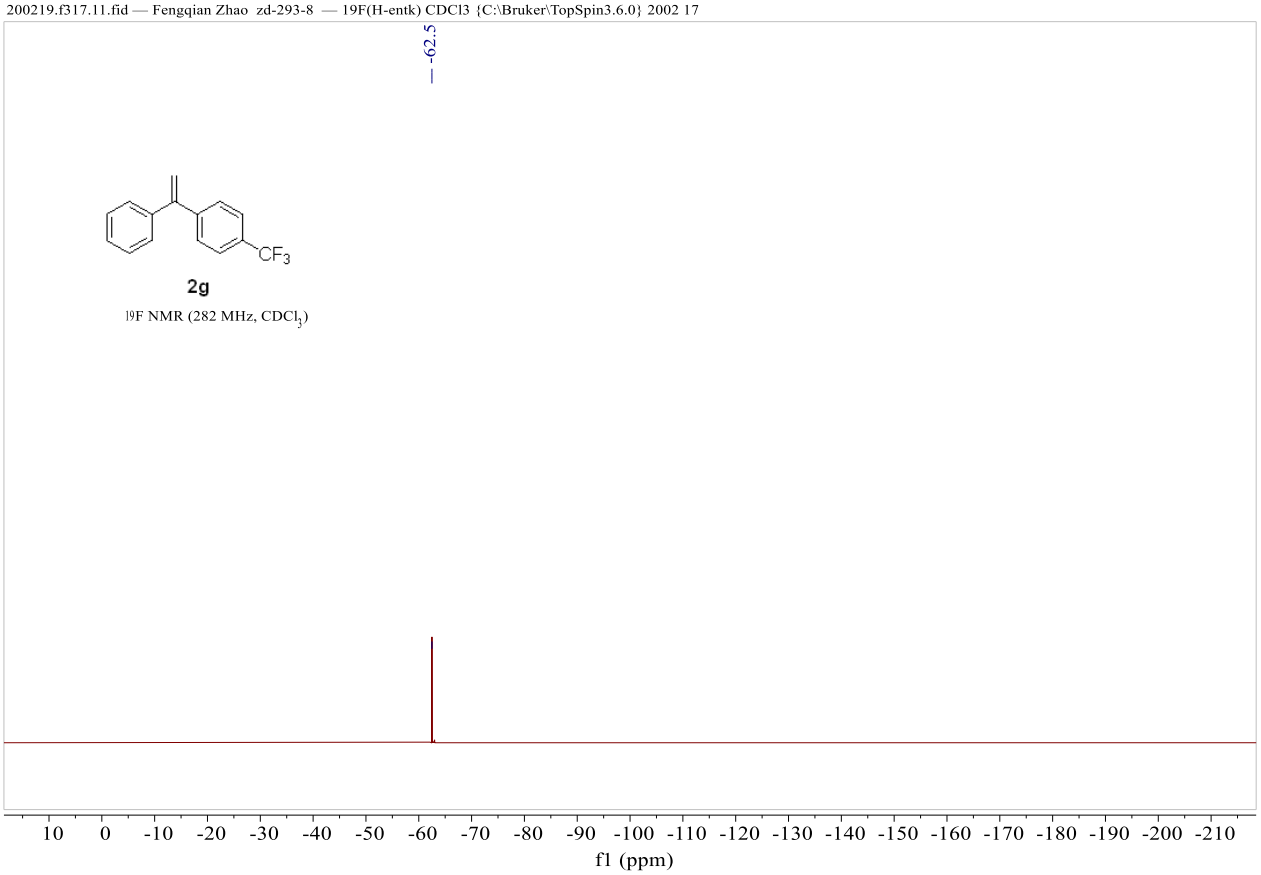
200219.f317.10.fid — Fengqian Zhao zd-293-8 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 17



200219.f317.12.fid — Fengqian Zhao zd-293-8 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 17



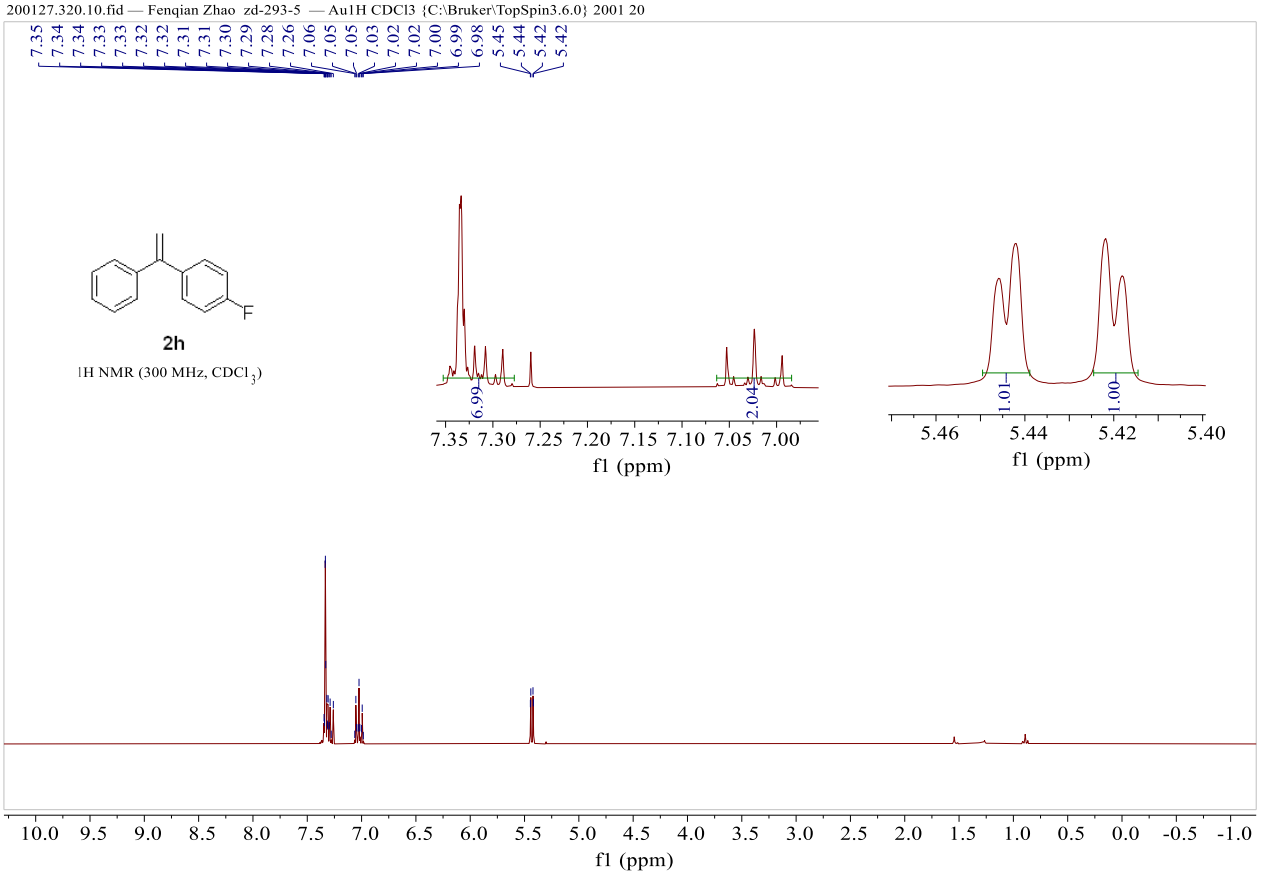
200219.f317.11.fid — Fengqian Zhao zd-293-8 — 19F(H-entk) CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 17



2g

^{19}F NMR (282 MHz, $CDCl_3$)

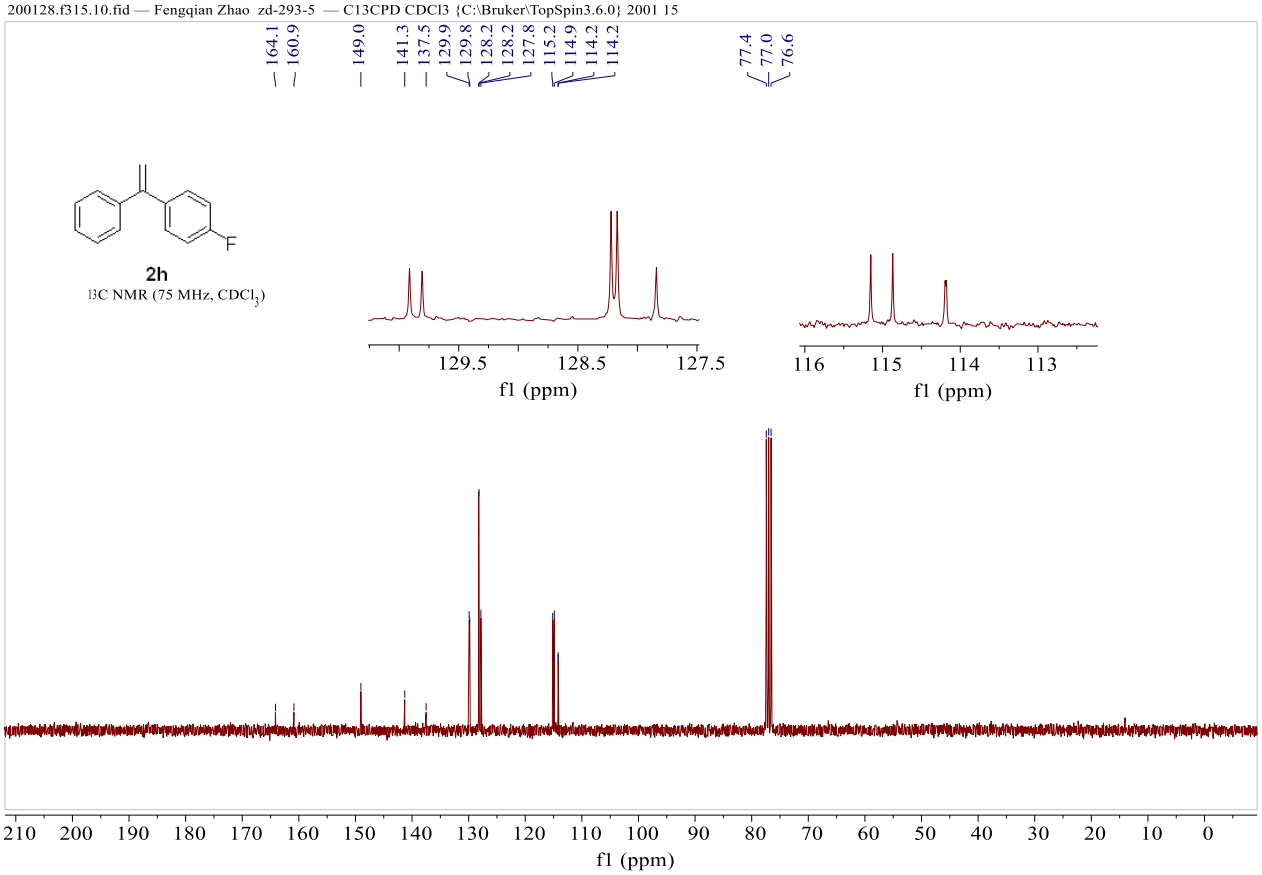
200127.320.10.fid — Fenqian Zhao zd-293-5 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.0} 2001 20



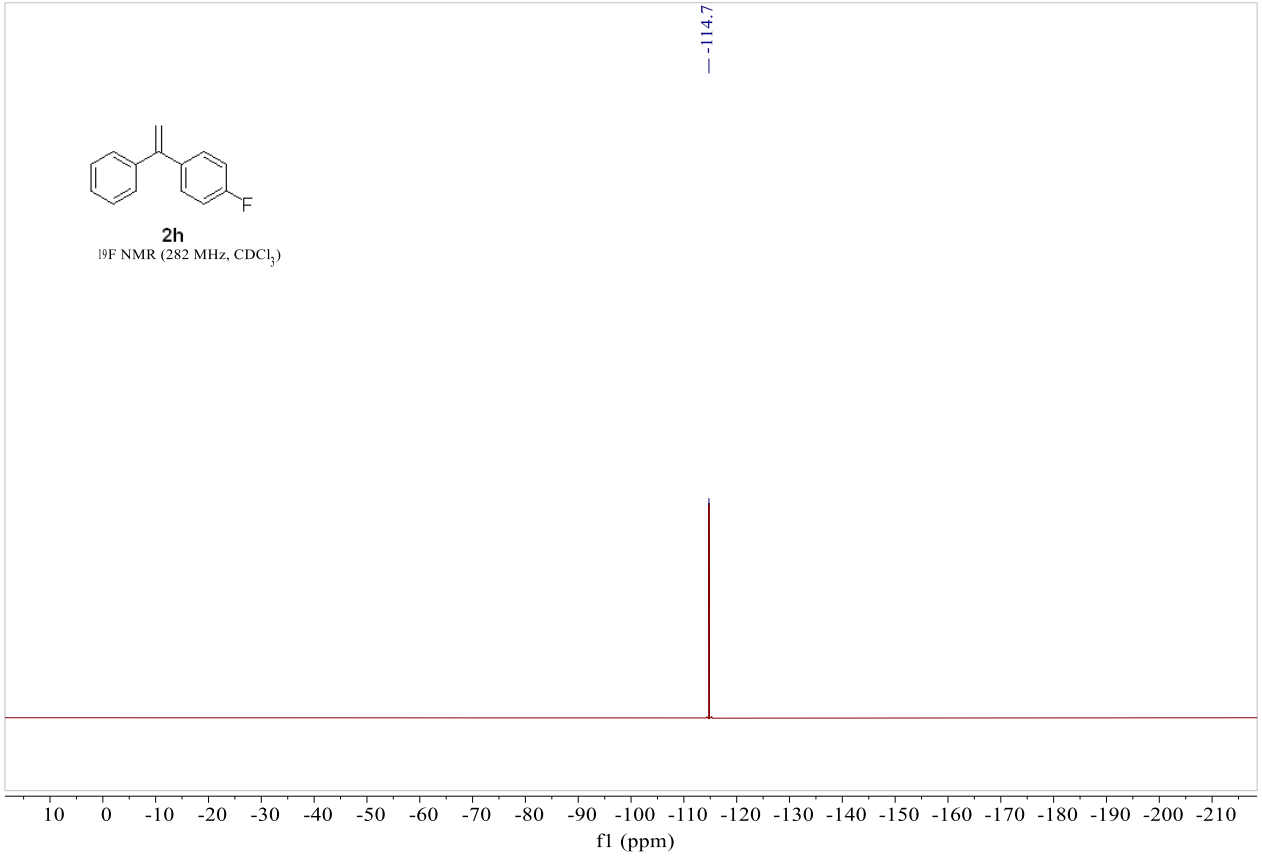
2h

^{1}H NMR (300 MHz, $CDCl_3$)

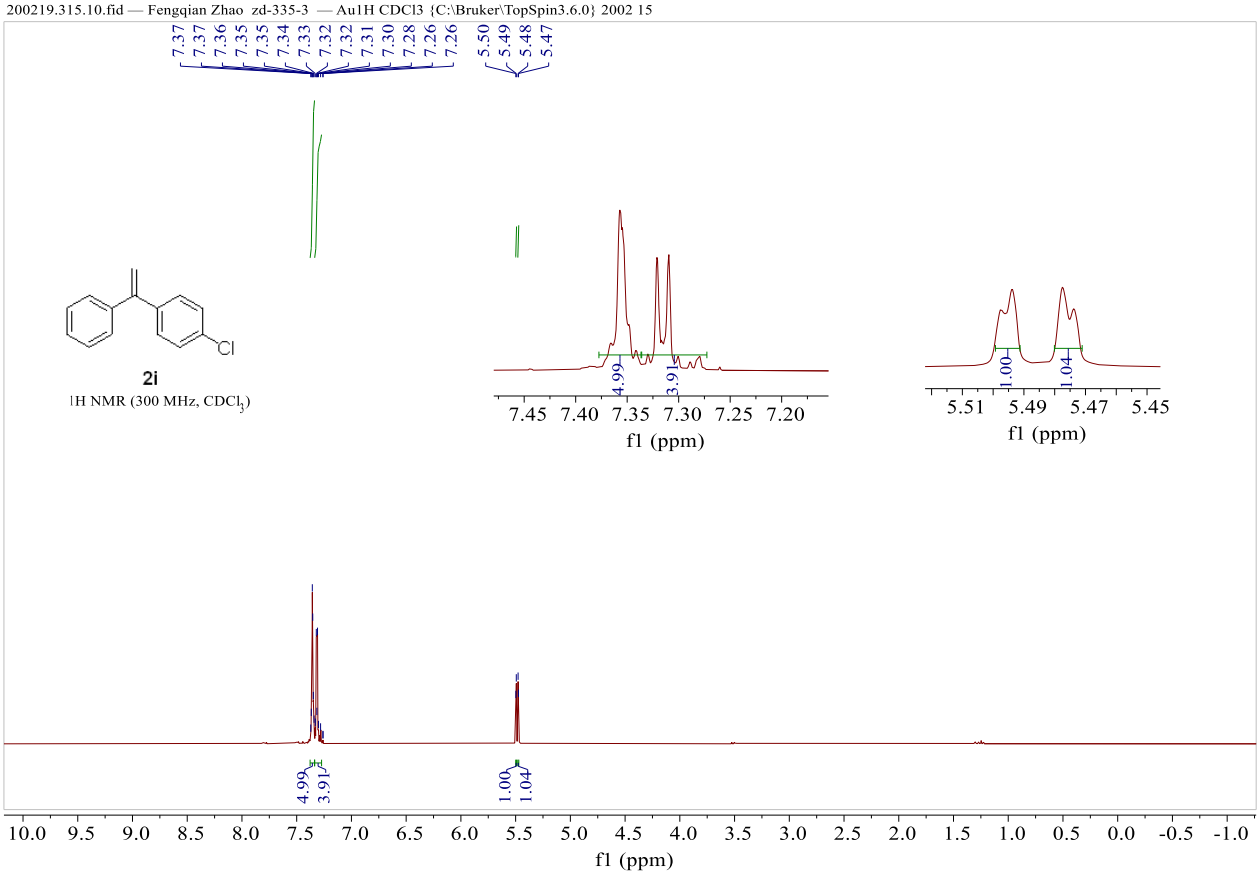
200128.f315.10.fid — Fengqian Zhao zd-293-5 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2001 15



200128.f315.11.fid — Fengqian Zhao zd-293-5 — 19F(H-entk) CDCl3 {C:\Bruker\TopSpin3.6.0} 2001 15



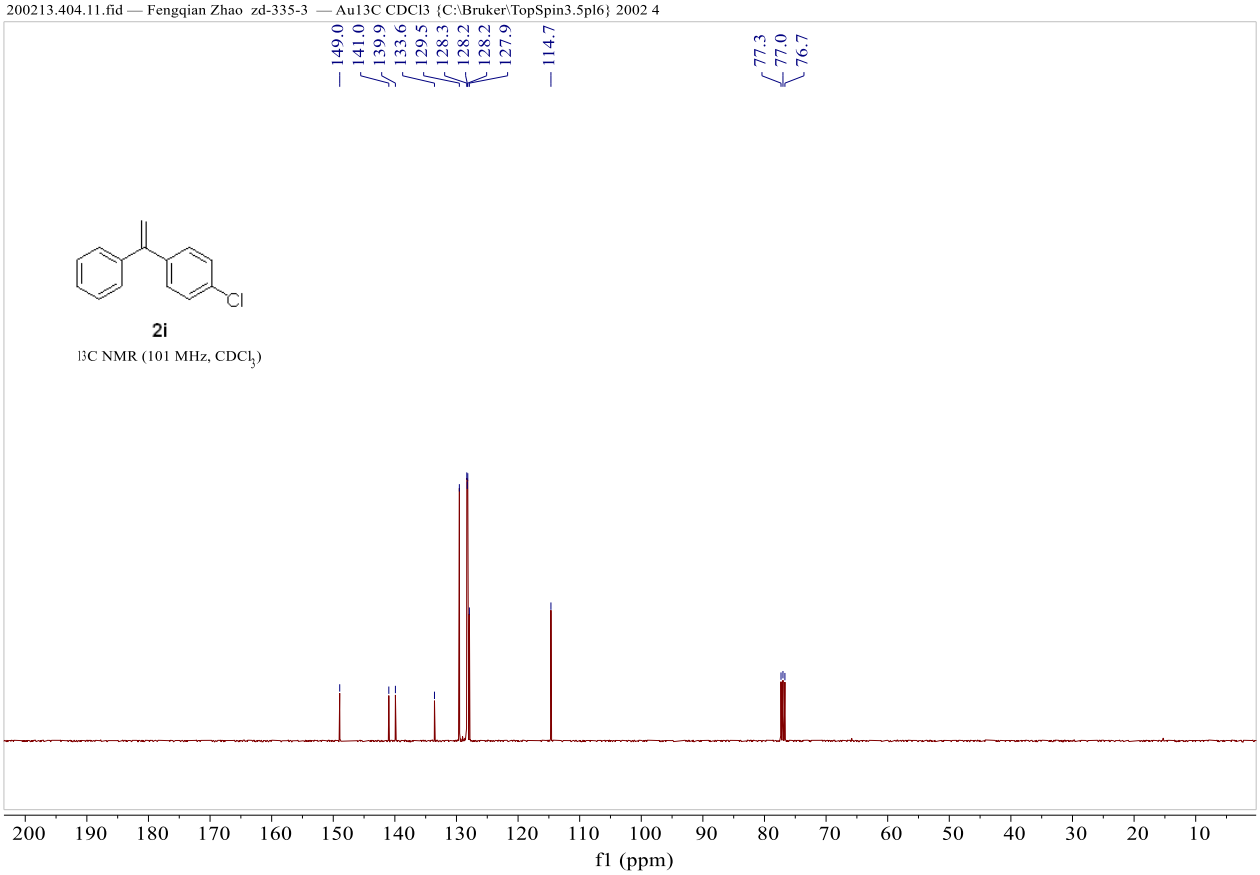
200219.315.10.fid — Fengqian Zhao zd-335-3 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 15



2i

^{1}H NMR (300 MHz, $CDCl_3$)

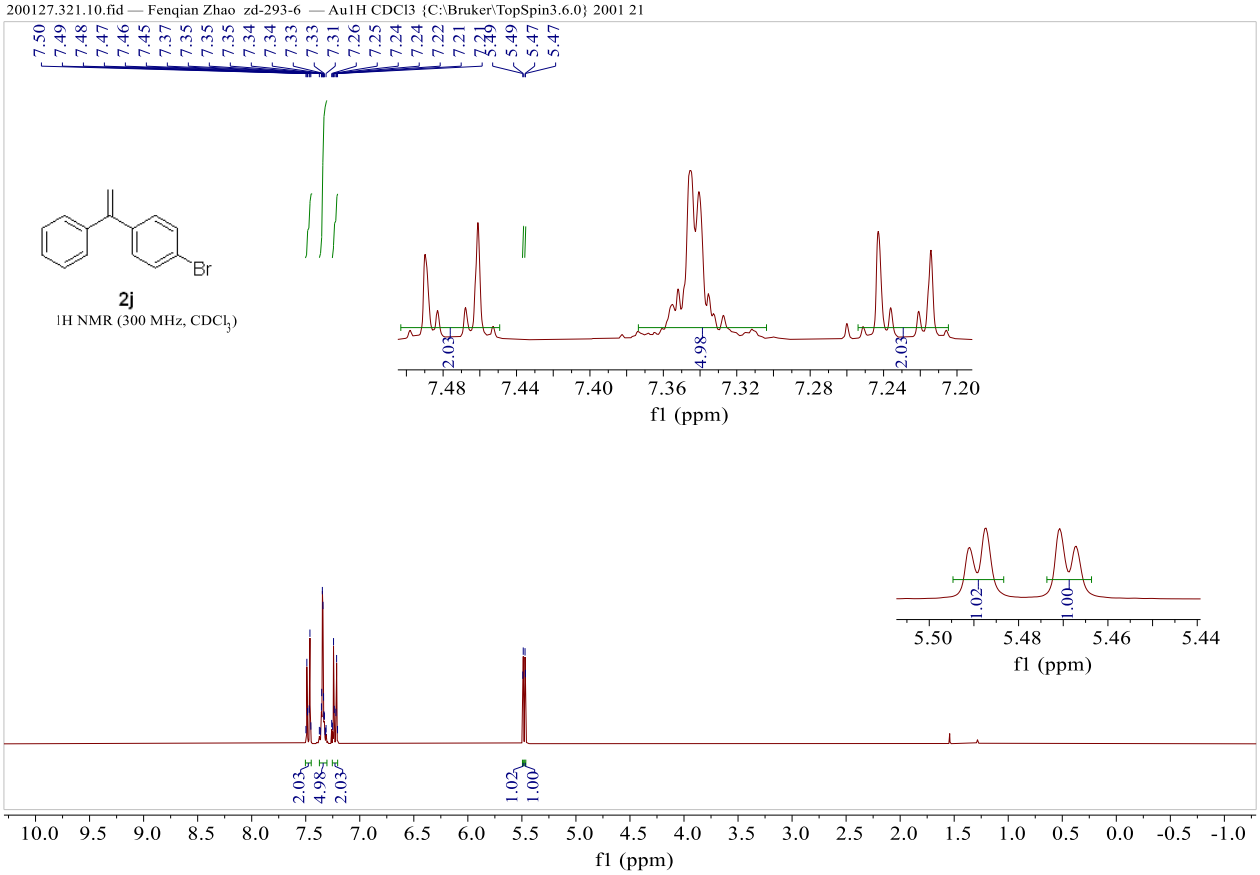
200213.404.11.fid — Fengqian Zhao zd-335-3 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2002 4



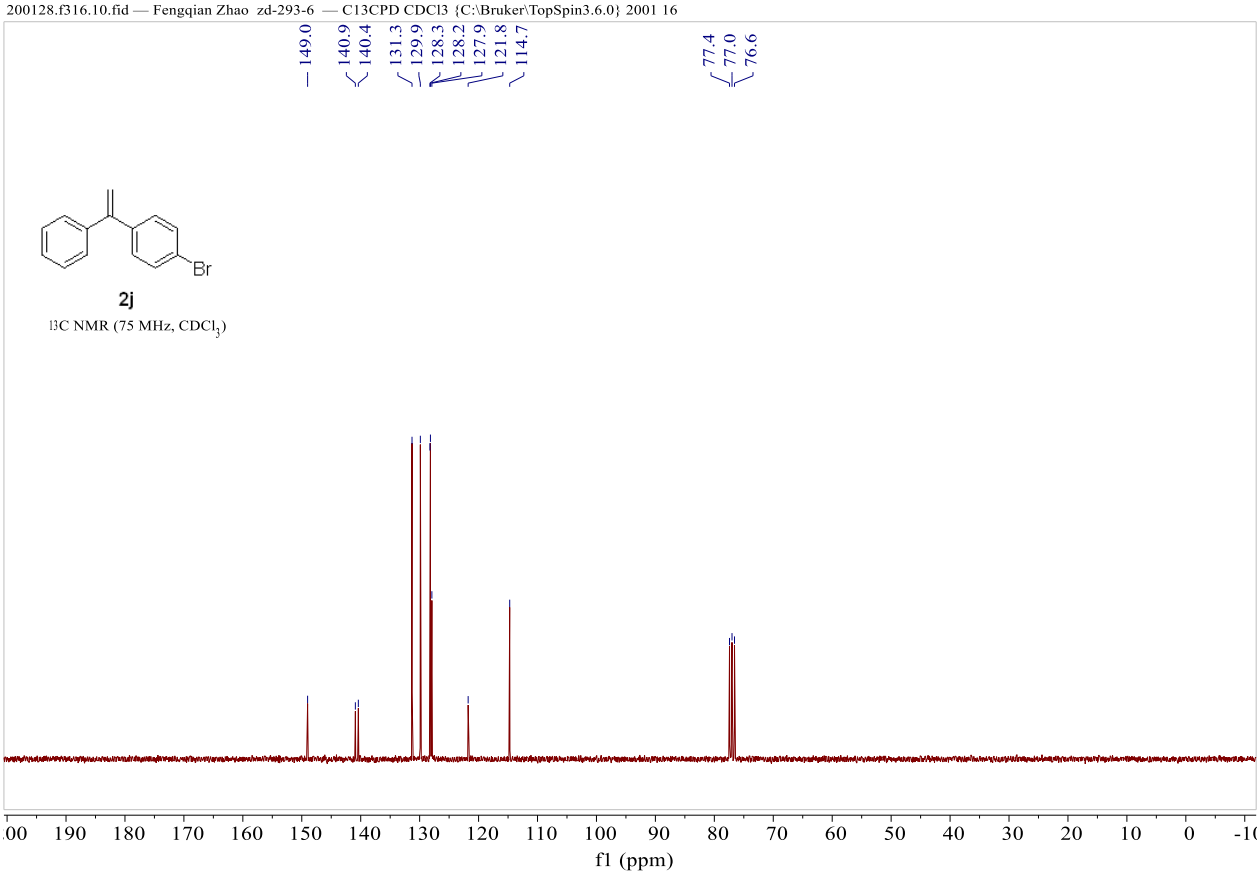
2i

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

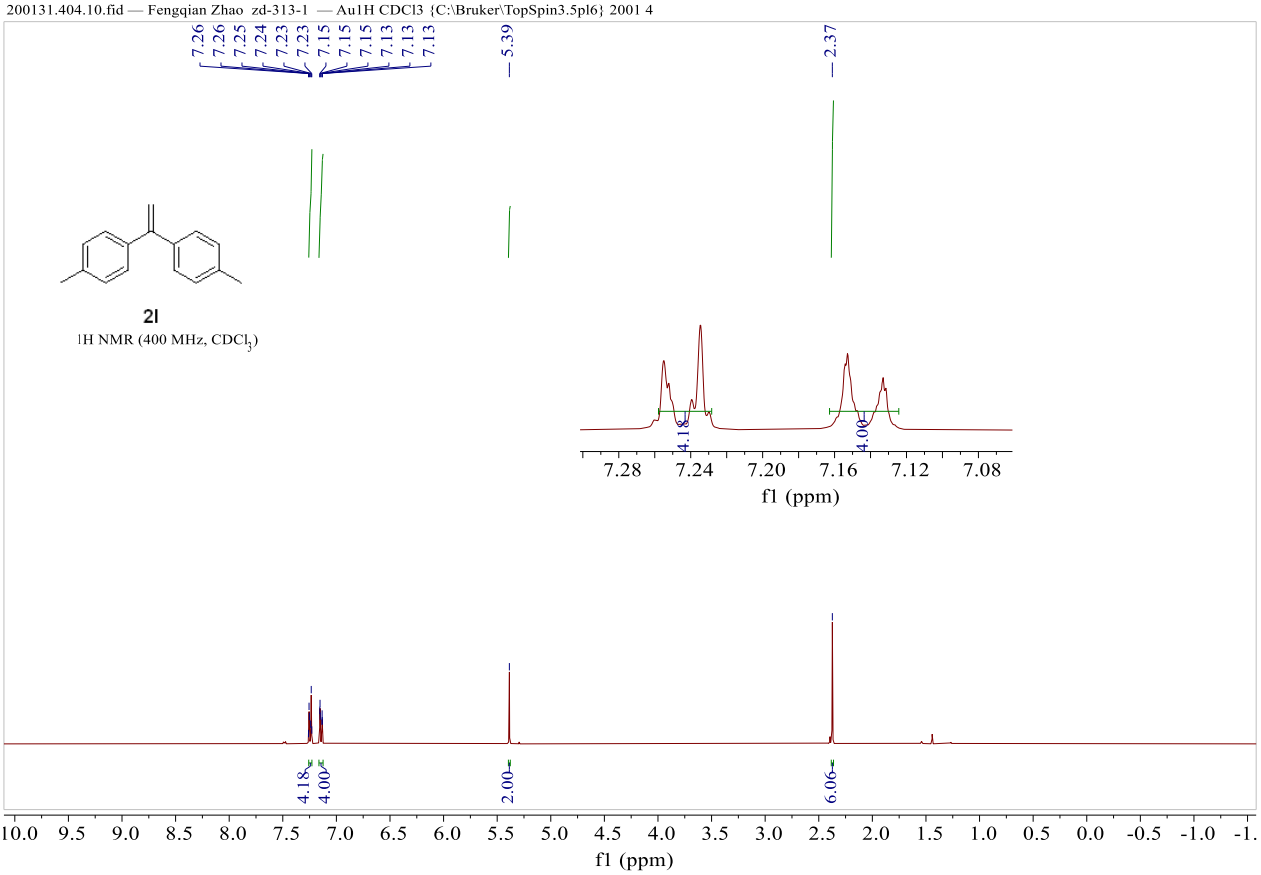
200127.321.10.fid — Fenqian Zhao zd-293-6 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.0} 2001 21



200128.f316.10.fid — Fengqian Zhao zd-293-6 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2001 16



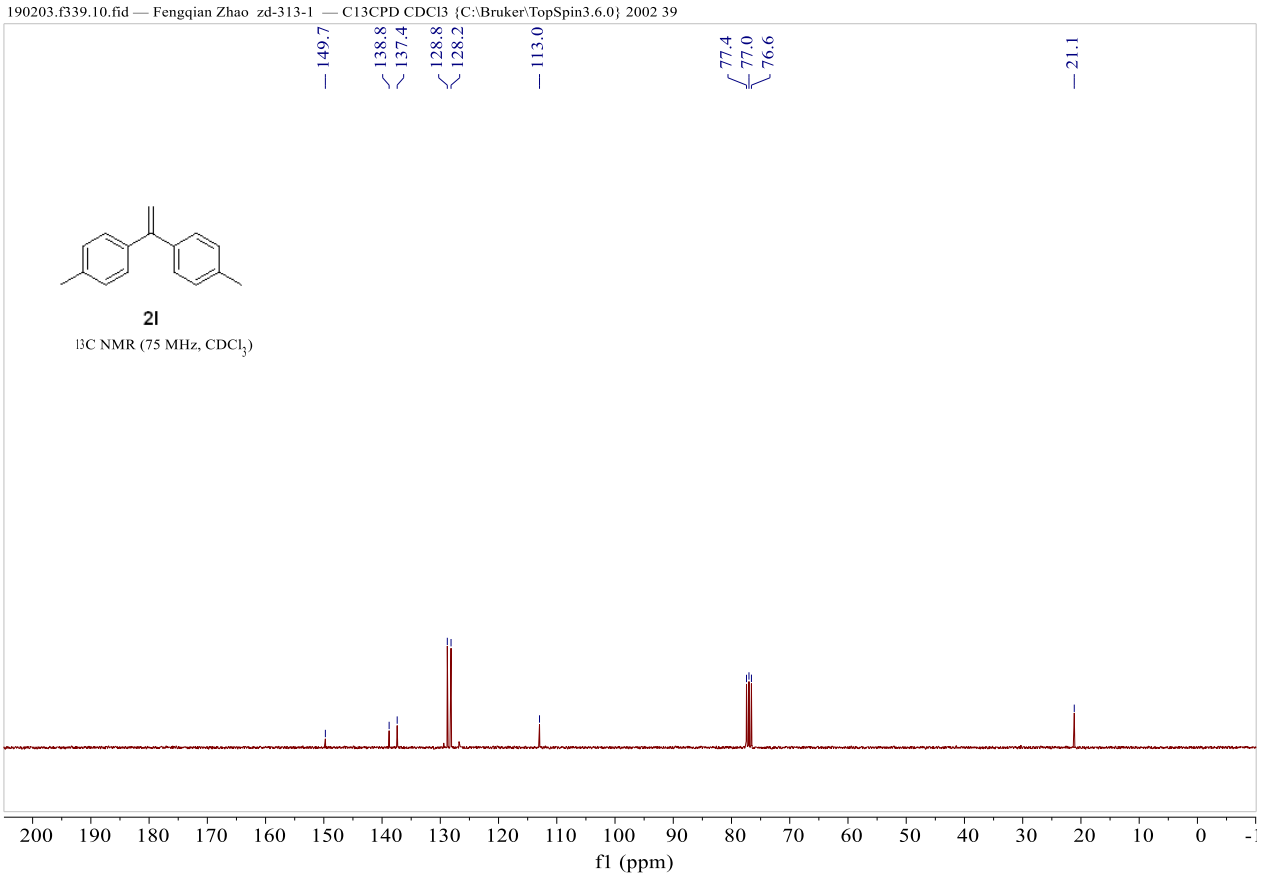
200131.404.10.fid — Fengqian Zhao zd-313-1 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2001 4



2l

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

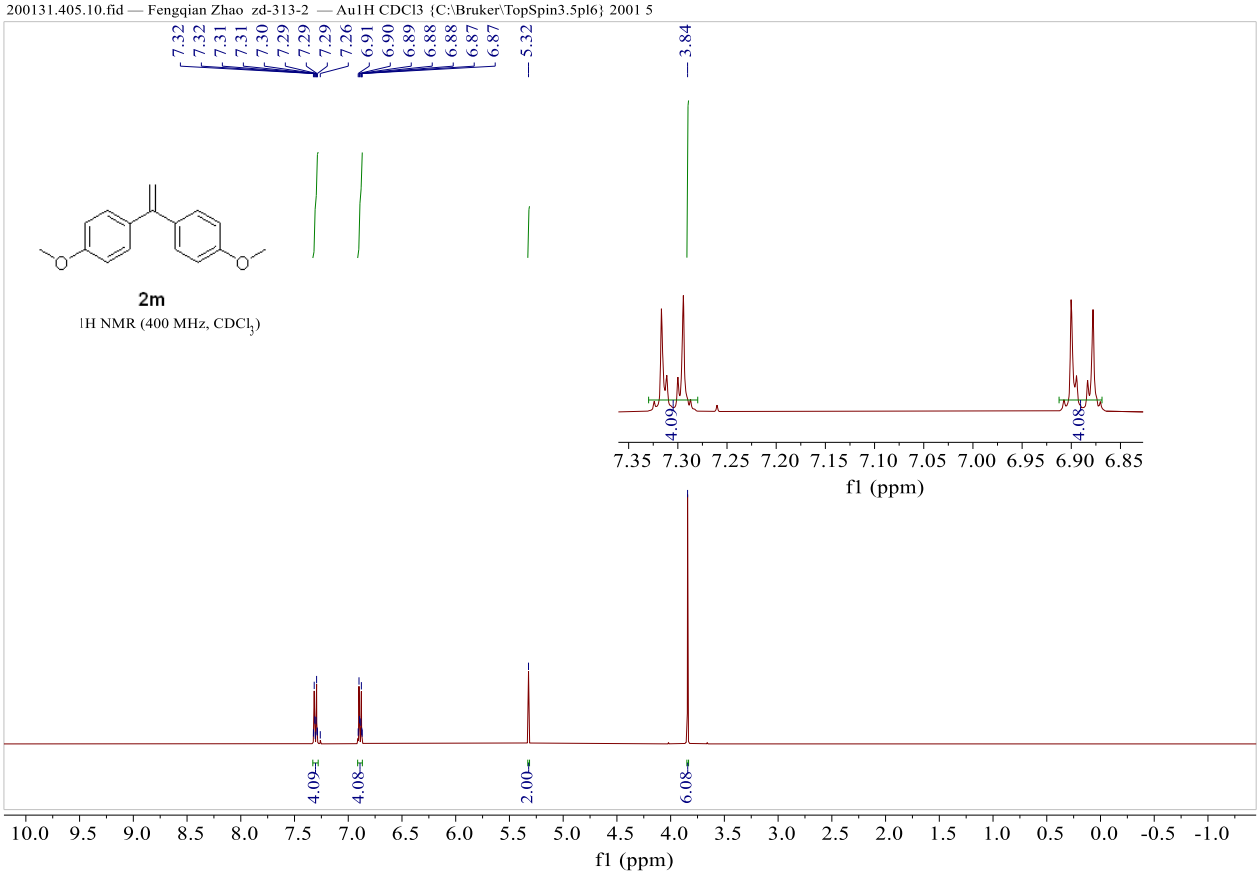
190203.f339.10.fid — Fengqian Zhao zd-313-1 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 39



2l

^{13}C NMR (75 MHz, $CDCl_3$)

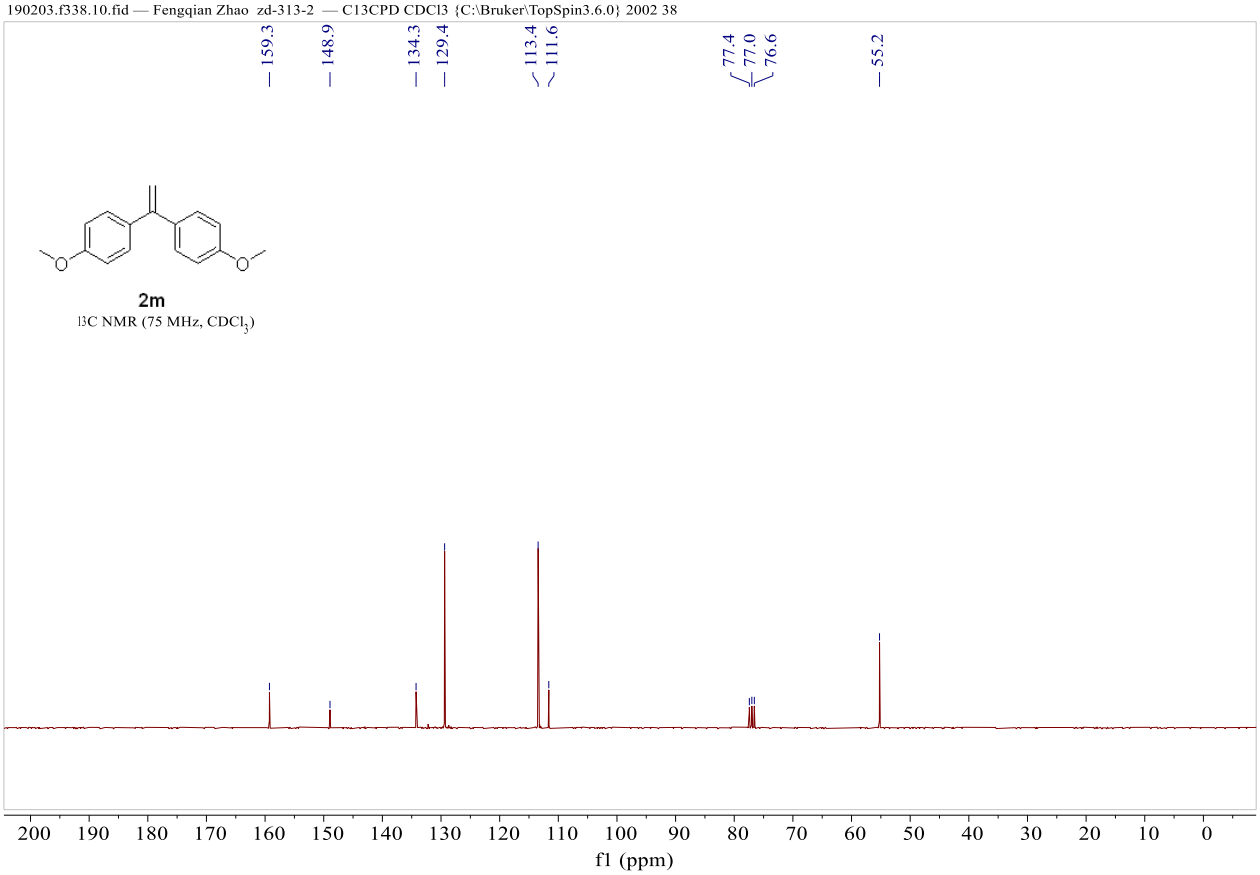
200131.405.10.fid — Fengqian Zhao zd-313-2 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2001 5



2m

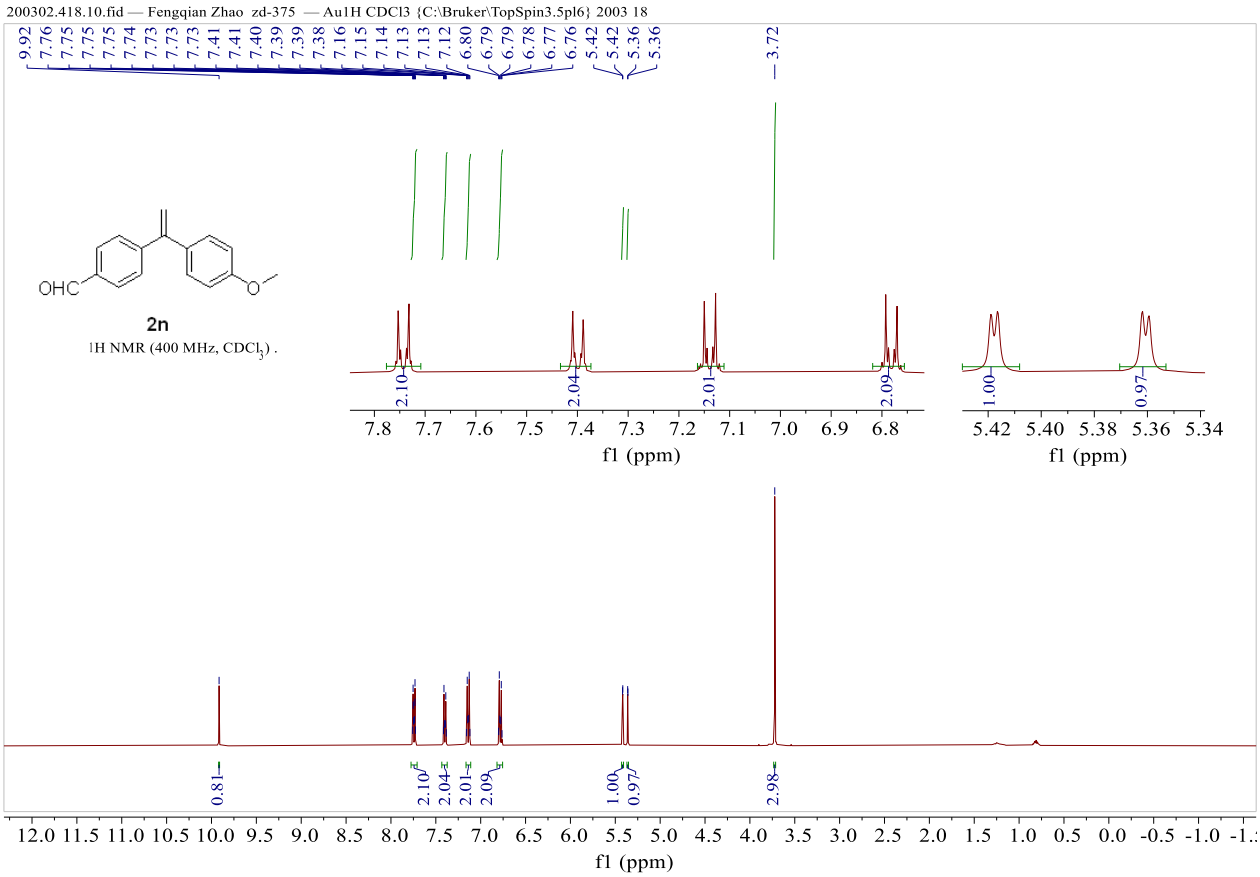
^{1}H NMR (400 MHz, $CDCl_3$)

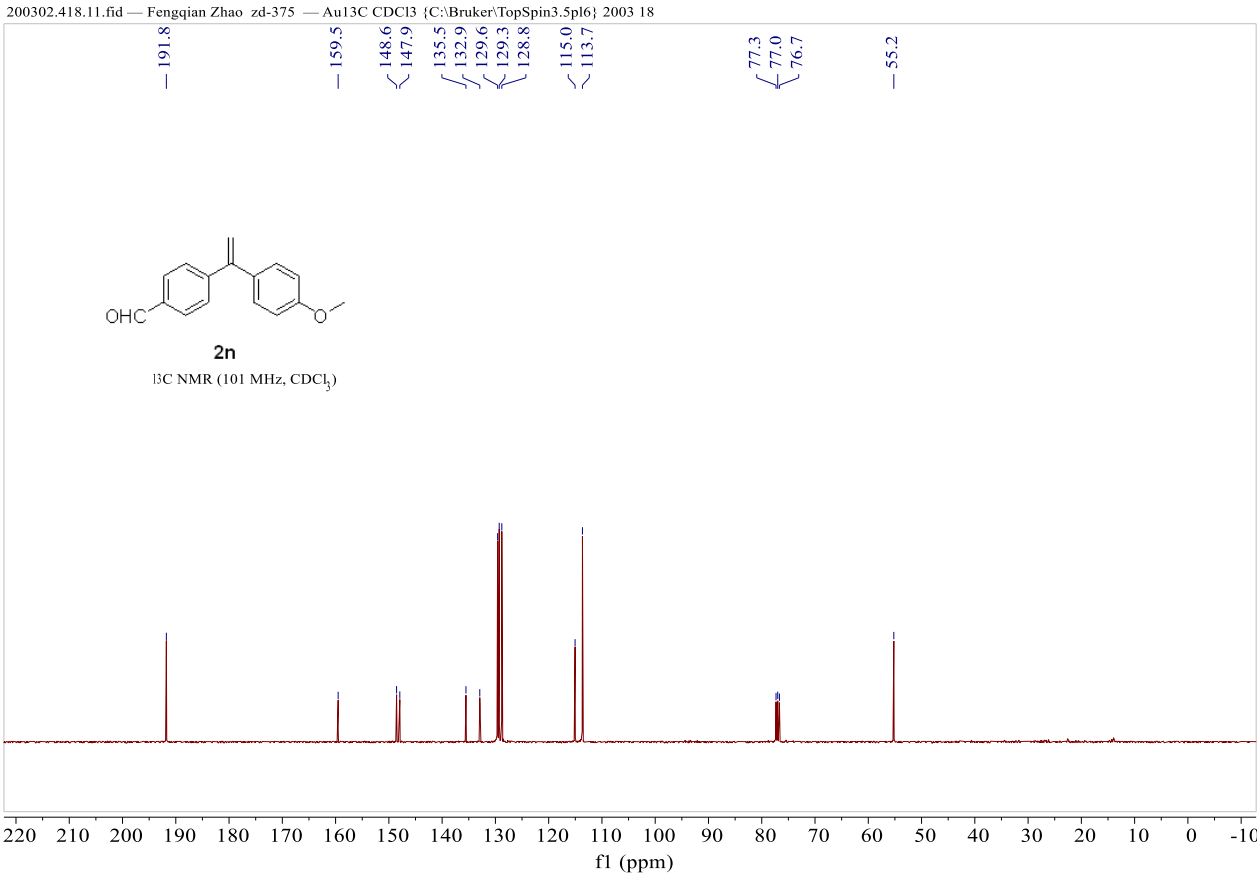
190203.f338.10.fid — Fengqian Zhao zd-313-2 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 38

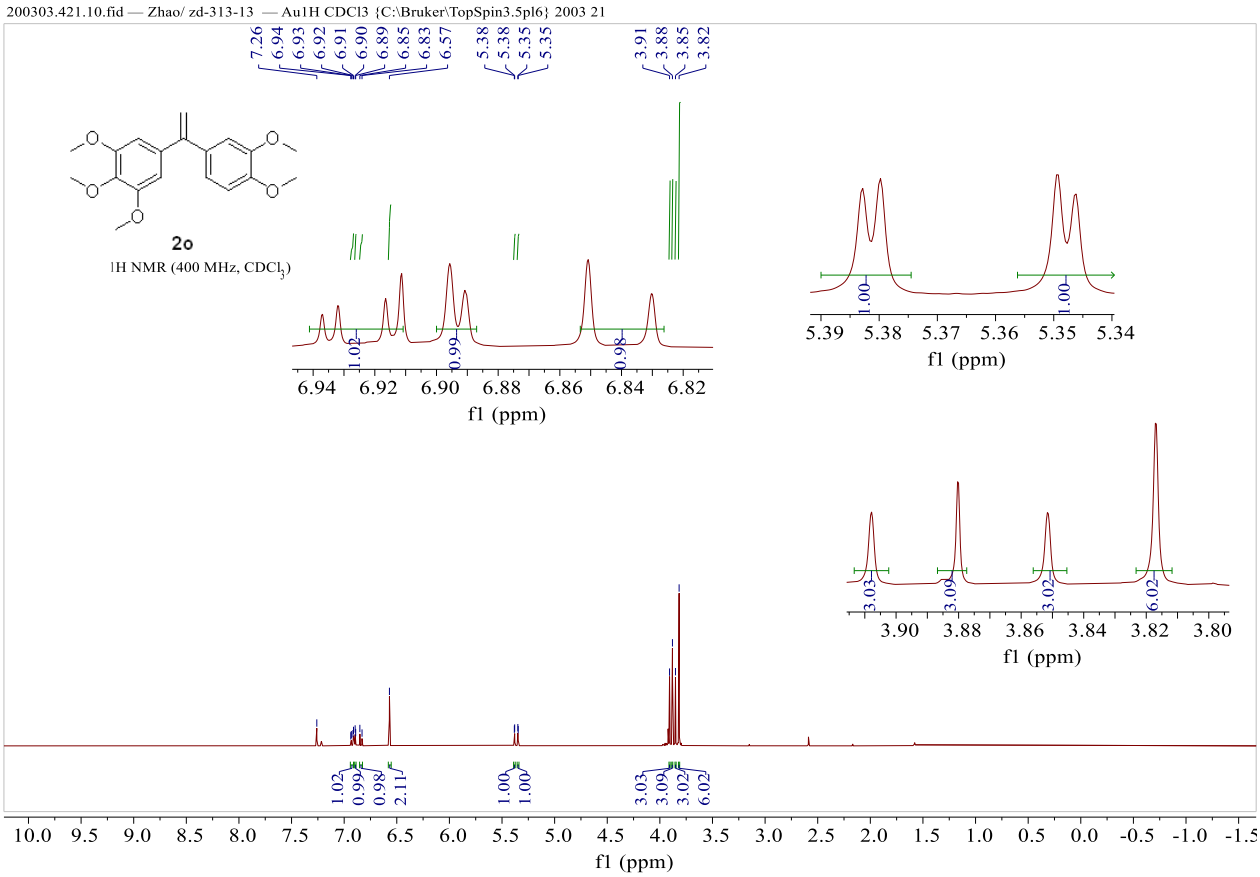


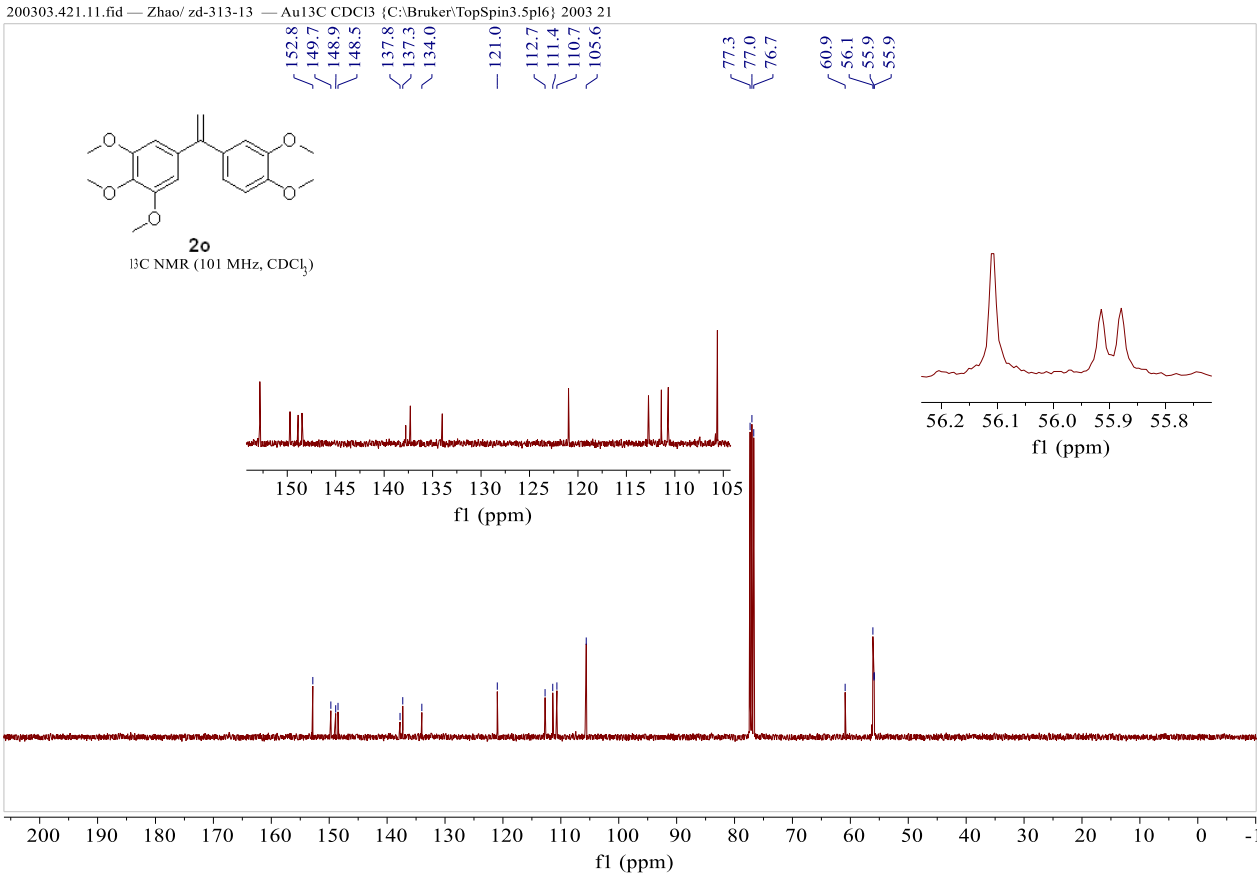
2m

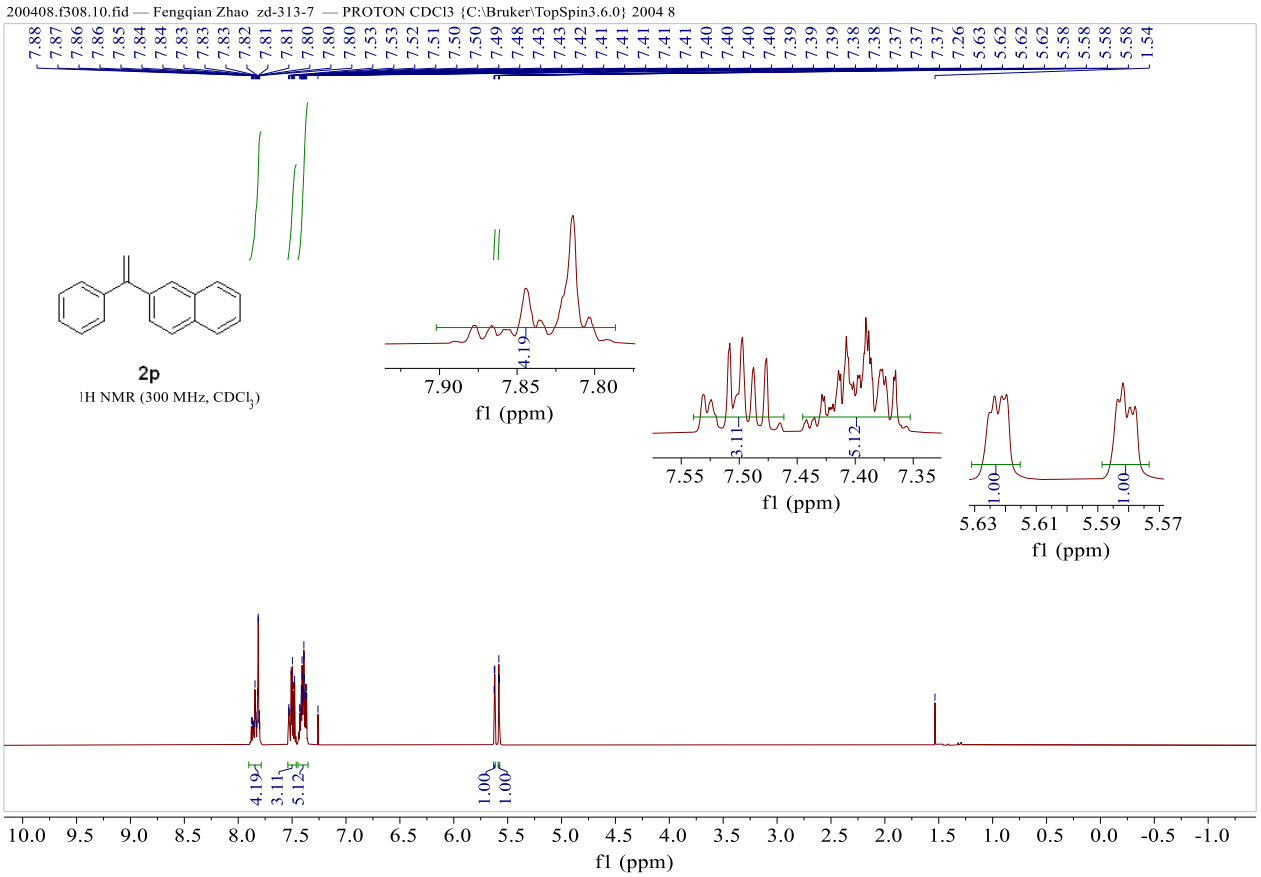
^{13}C NMR (75 MHz, $CDCl_3$)

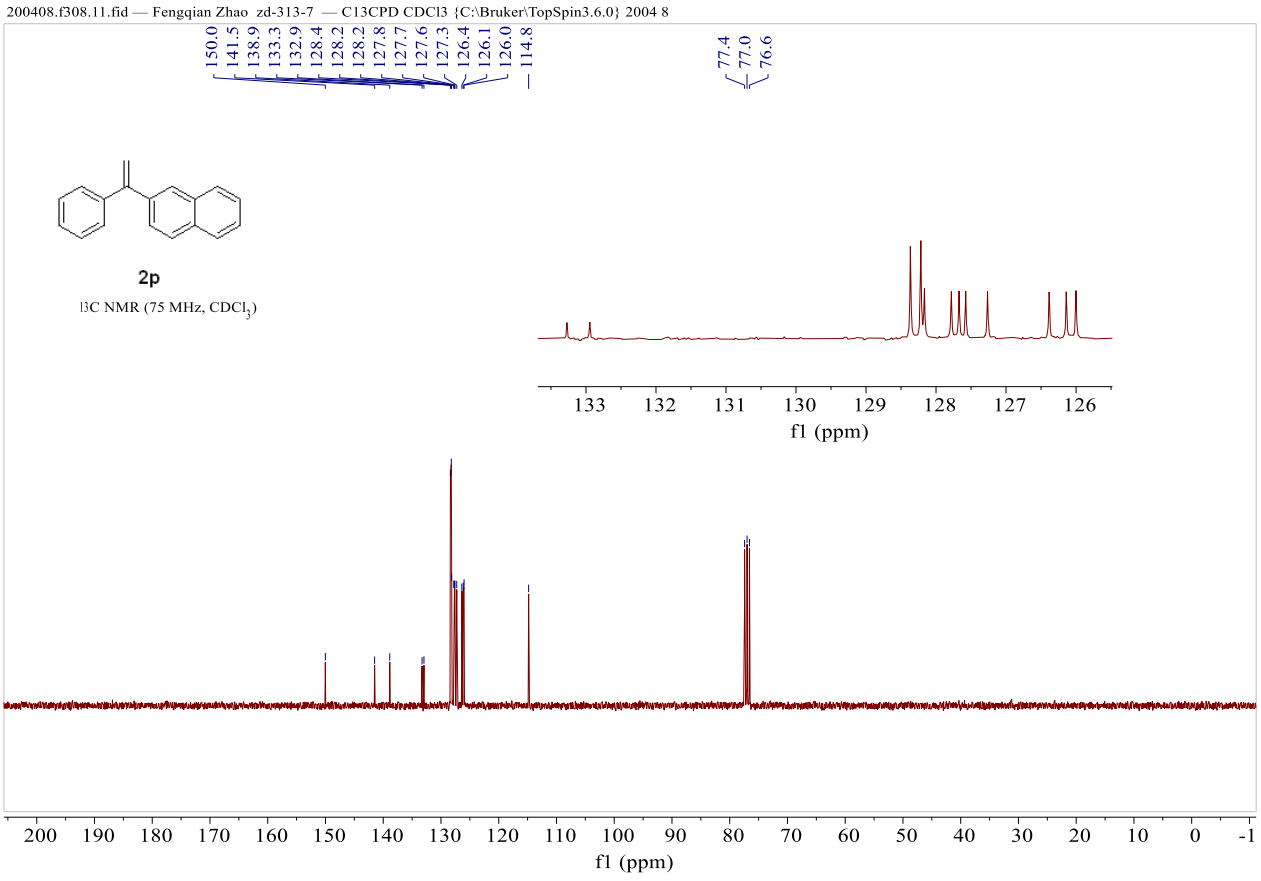

200302.418.10.fid — Fengqian Zhao zd-375 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 18
9.92 7.76 7.75 7.75 7.75 7.74 7.73 7.73 7.73 7.41 7.41 7.40 7.39 7.39 7.38 7.16 7.15 7.14 7.13 7.13 7.12 6.80 6.79 6.79 6.78 6.77 6.76 5.42 5.42 5.36 5.36 3.72
OHC
O
2n
1H NMR (400 MHz, CDCl3) .
2.10 2.04 2.01 2.09
1.00 0.97
f1 (ppm)
0.81 2.10 2.04 2.01 2.09 1.00 0.97 2.98
f1 (ppm)


200302.418.11.fid — Fengqian Zhao zd-375 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 18
191.8 159.5 148.6 147.9 135.5 132.9 129.6 129.3 128.8 115.0 113.7 77.3 77.0 76.7 55.2
OHC
O
2n
13C NMR (101 MHz, CDCl3)
f1 (ppm)

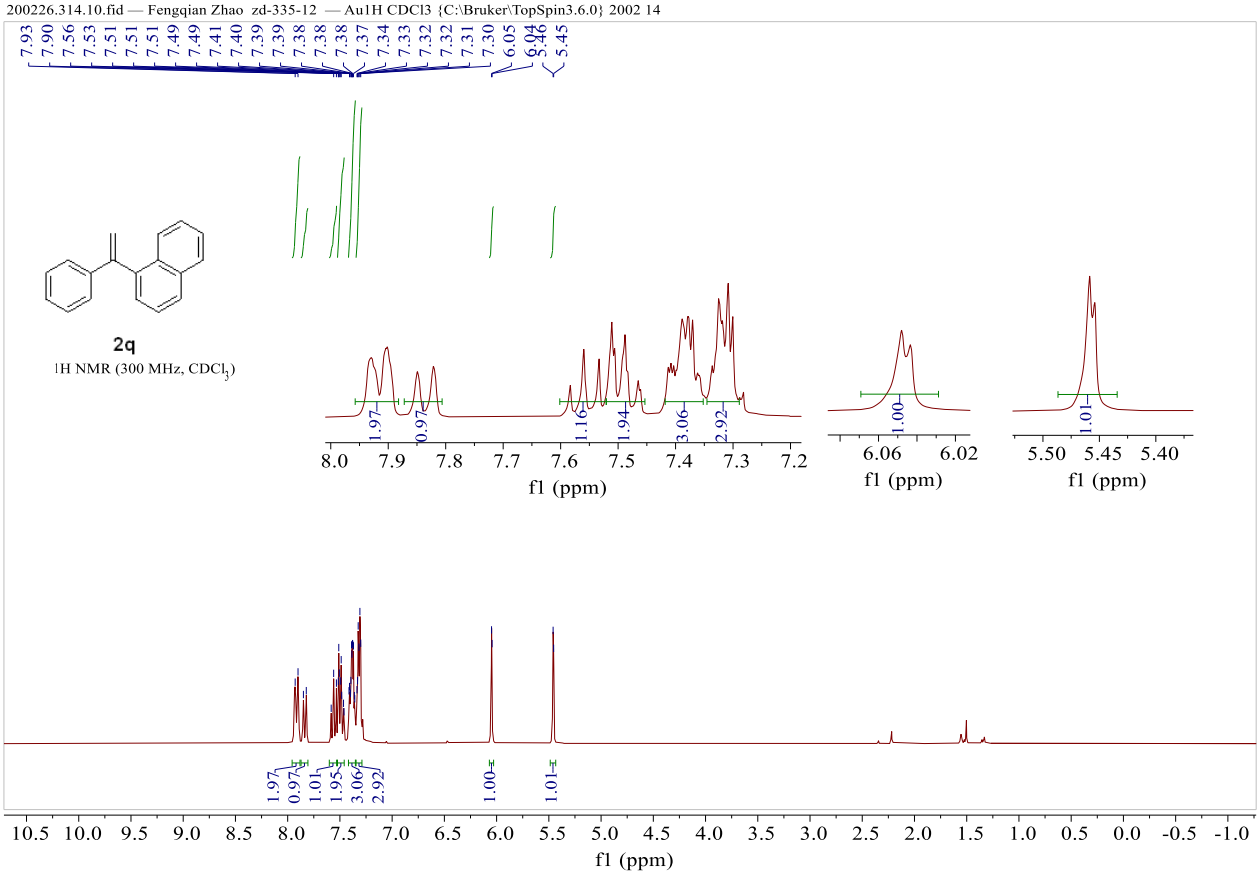

200303.421.10.fid — Zhao/ zd-313-13 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 21
7.26 6.94 6.93 6.92 6.91 6.90 6.89 6.85 6.83 6.57 5.38 5.38 5.35 5.35 3.91 3.88 3.85 3.82
2o
1H NMR (400 MHz, CDCl3)
1.02 0.99 0.98 2.11 1.00 1.00 3.03 3.09 3.02 6.02
f1 (ppm)


200303.421.11.fid — Zhao/ zd-313-13 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 21
152.8 149.7 148.9 148.5 137.8 137.3 134.0 121.0 112.7 111.4 110.7 105.6 77.3 77.0 76.7 60.9 56.1 55.9 55.9
2o
13C NMR (101 MHz, CDCl3)
f1 (ppm)

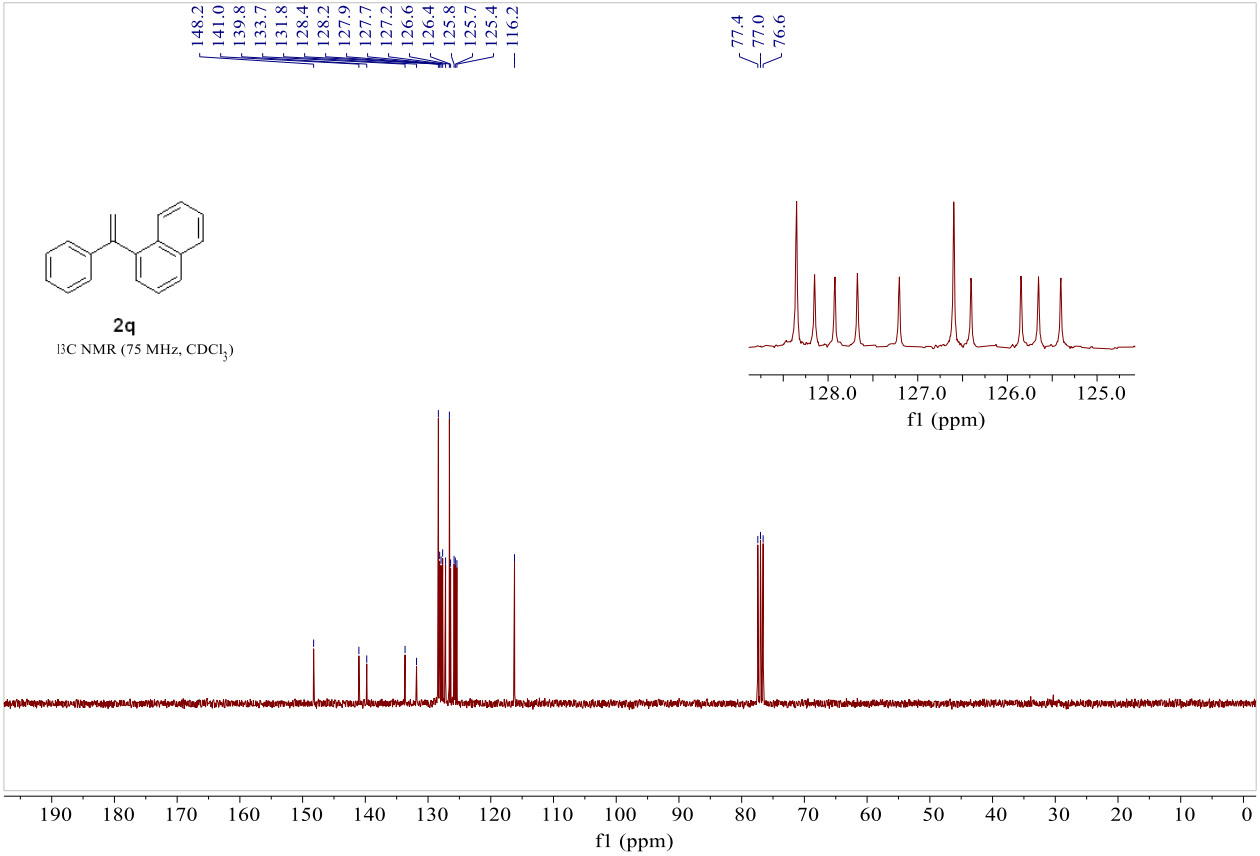

200408.f308.10.fid — Fengqian Zhao zd-313-7 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2004 8
2p
1H NMR (300 MHz, CDCl3)
f1 (ppm)

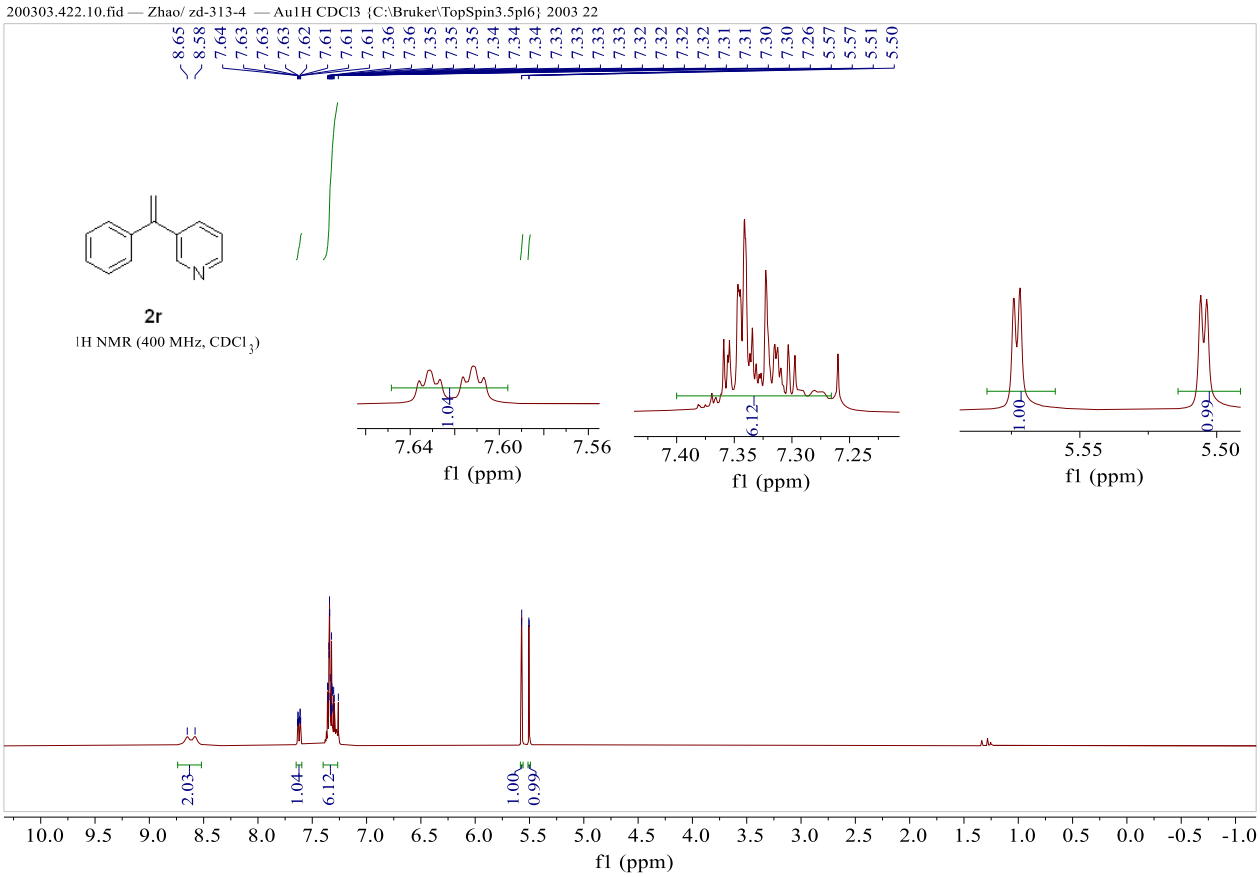

200408.f308.11.fid — Fengqian Zhao zd-313-7 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2004 8
2p
13C NMR (75 MHz, CDCl3)
f1 (ppm)

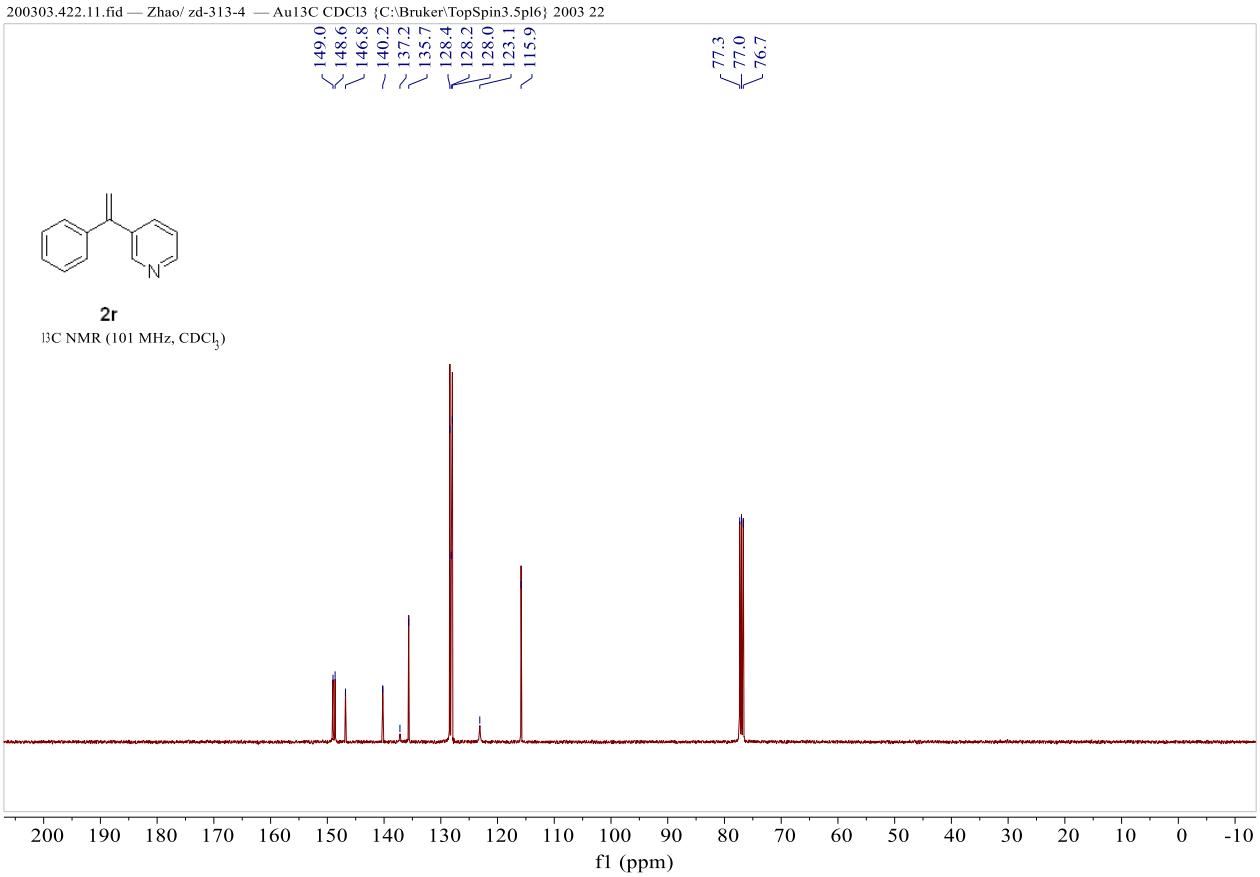
200226.314.10.fid — Fengqian Zhao zd-335-12 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 14



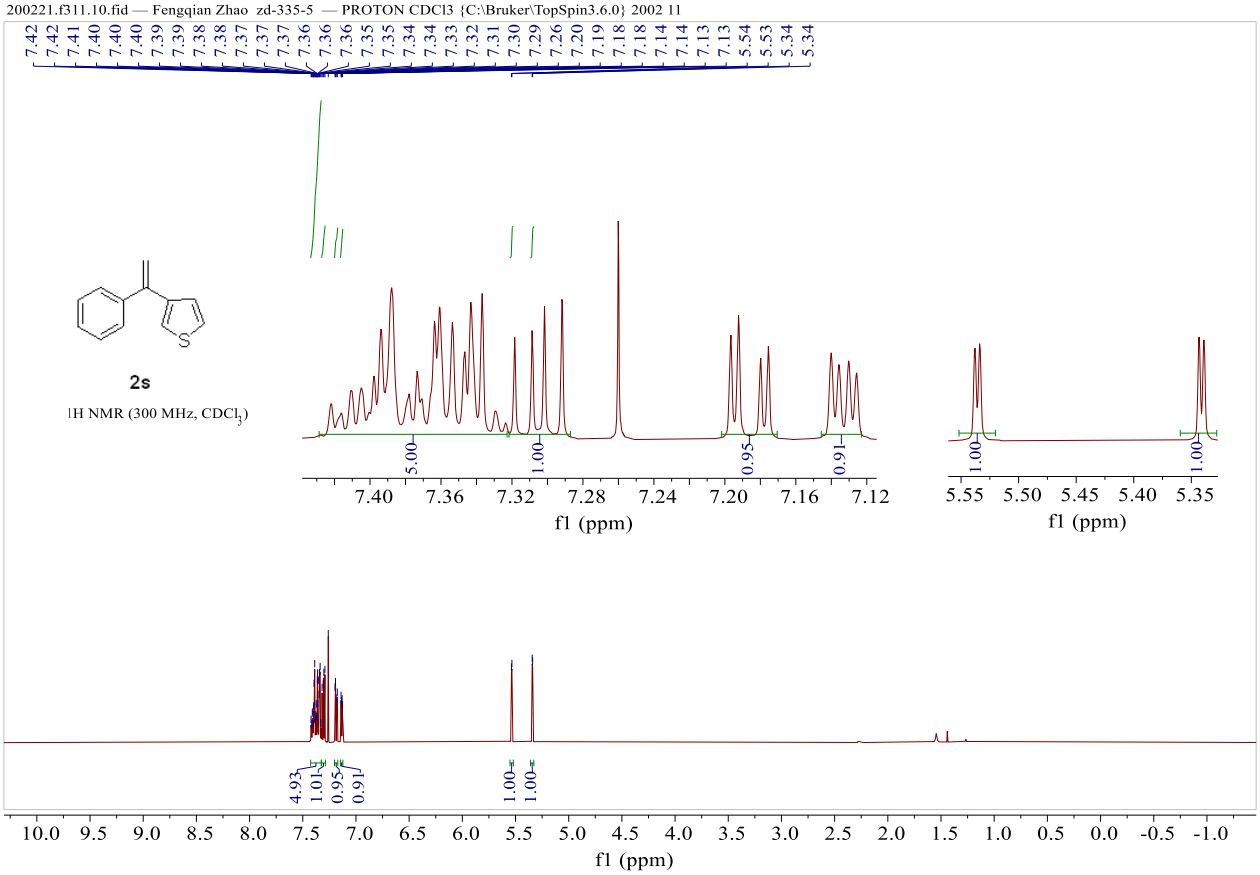
200226.314.11.fid — Fengqian Zhao zd-335-12 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 14



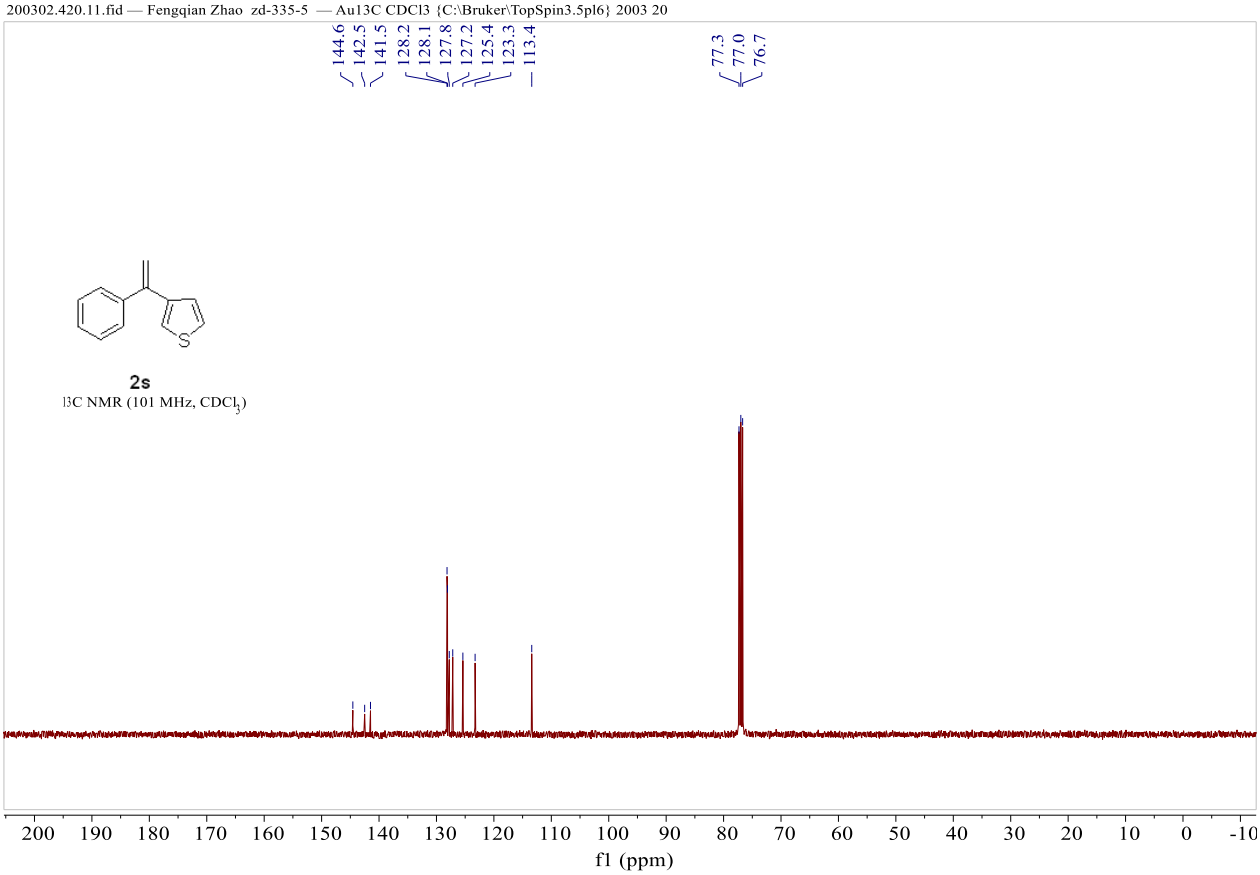

200303.422.10.fid — Zhao/ zd-313-4 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 22
2r
1H NMR (400 MHz, CDCl3)
f1 (ppm)

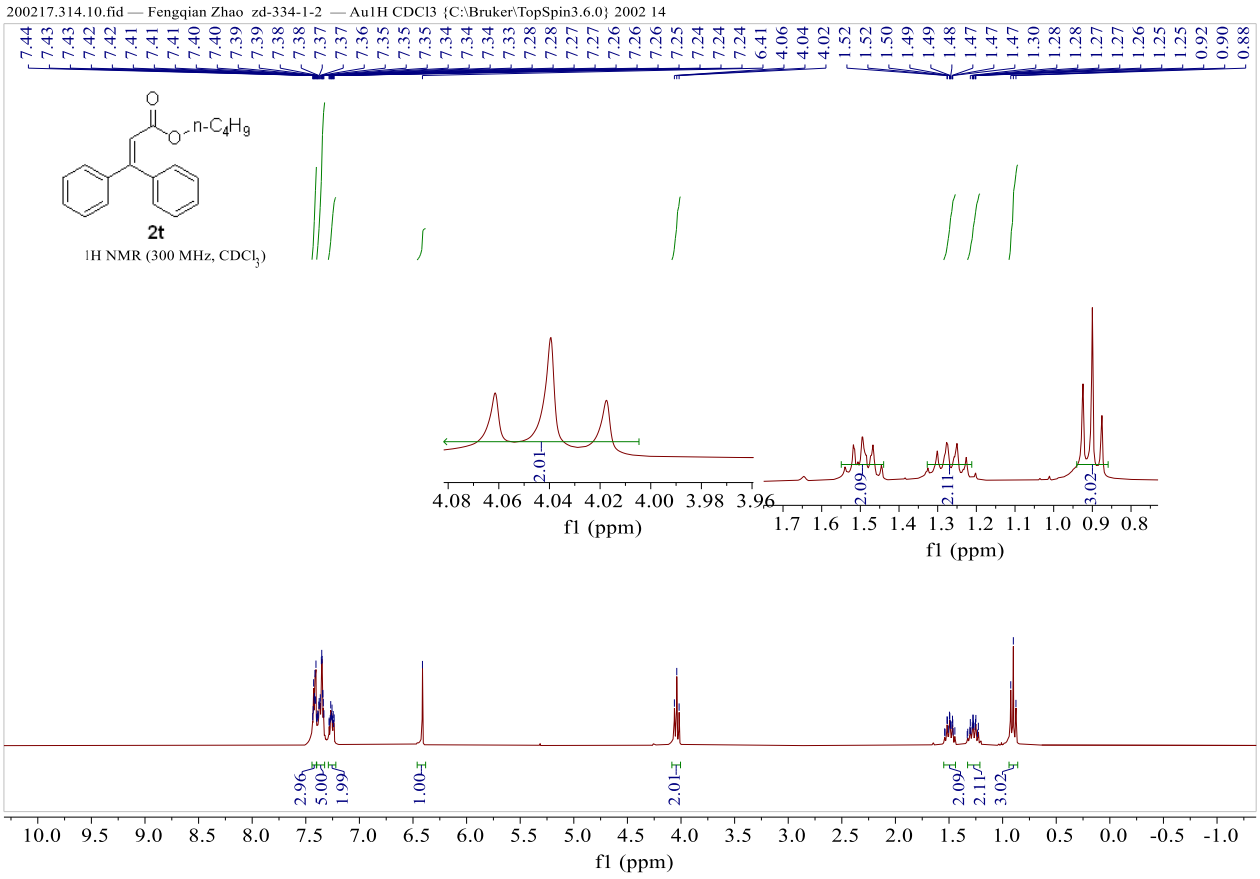

200303.422.11.fid — Zhao/ zd-313-4 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 22
2r
13C NMR (101 MHz, CDCl3)
f1 (ppm)

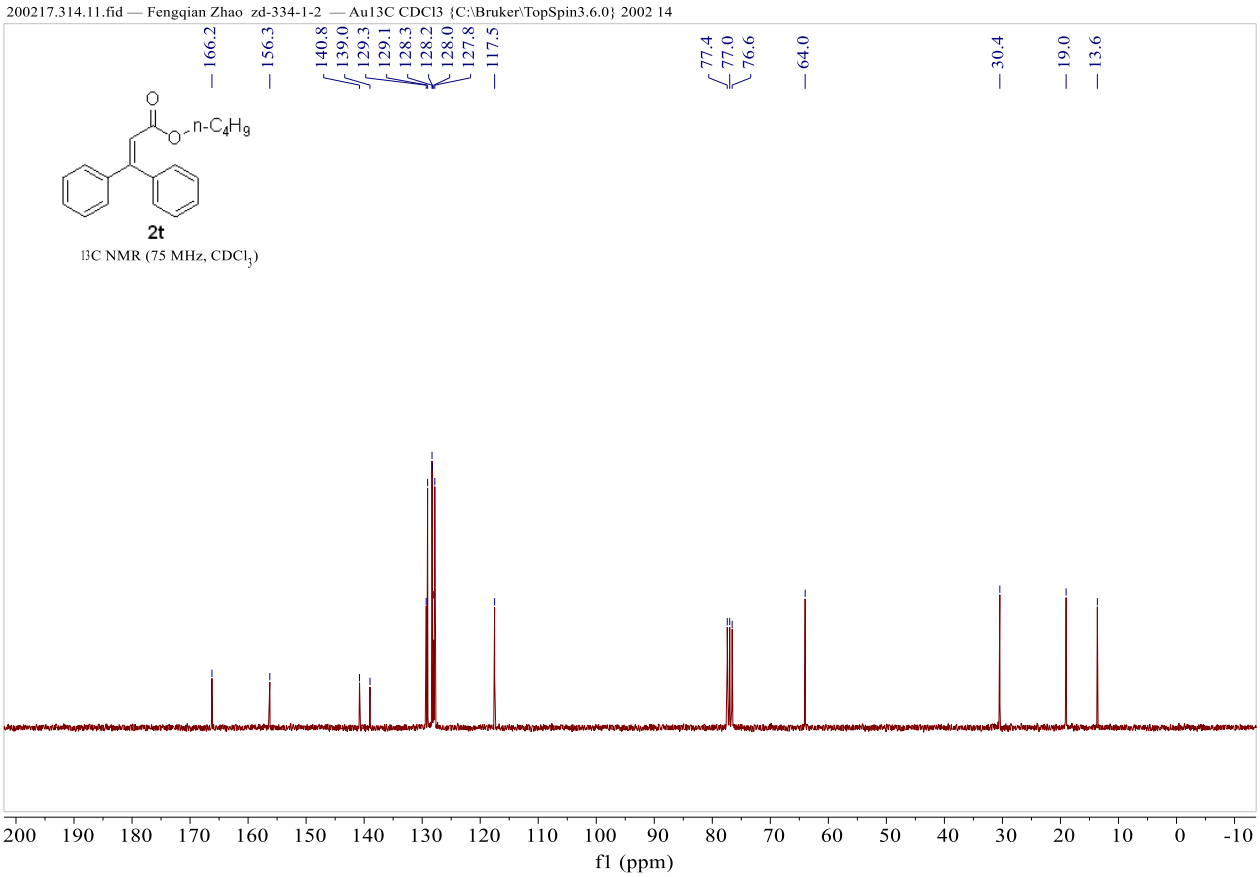
200221.f311.10.fid — Fengqian Zhao zd-335-5 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 11



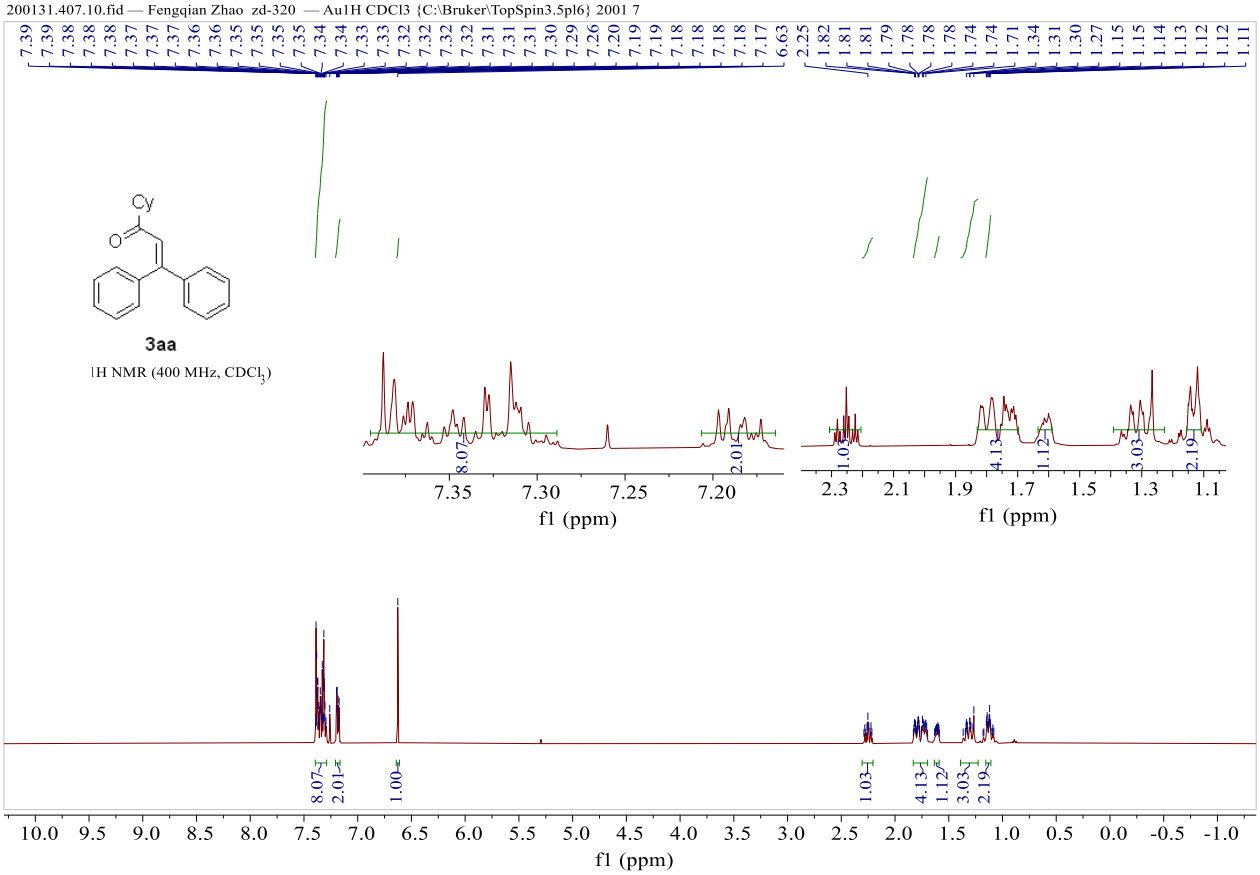
200302.420.11.fid — Fengqian Zhao zd-335-5 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 20




200217.314.10.fid — Fengqian Zhao zd-334-1-2 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 14
n-C4H9
2t
1H NMR (300 MHz, CDCl3)
f1 (ppm)


200217.314.11.fid — Fengqian Zhao zd-334-1-2 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 14
n-C4H9
2t
13C NMR (75 MHz, CDCl3)
f1 (ppm)

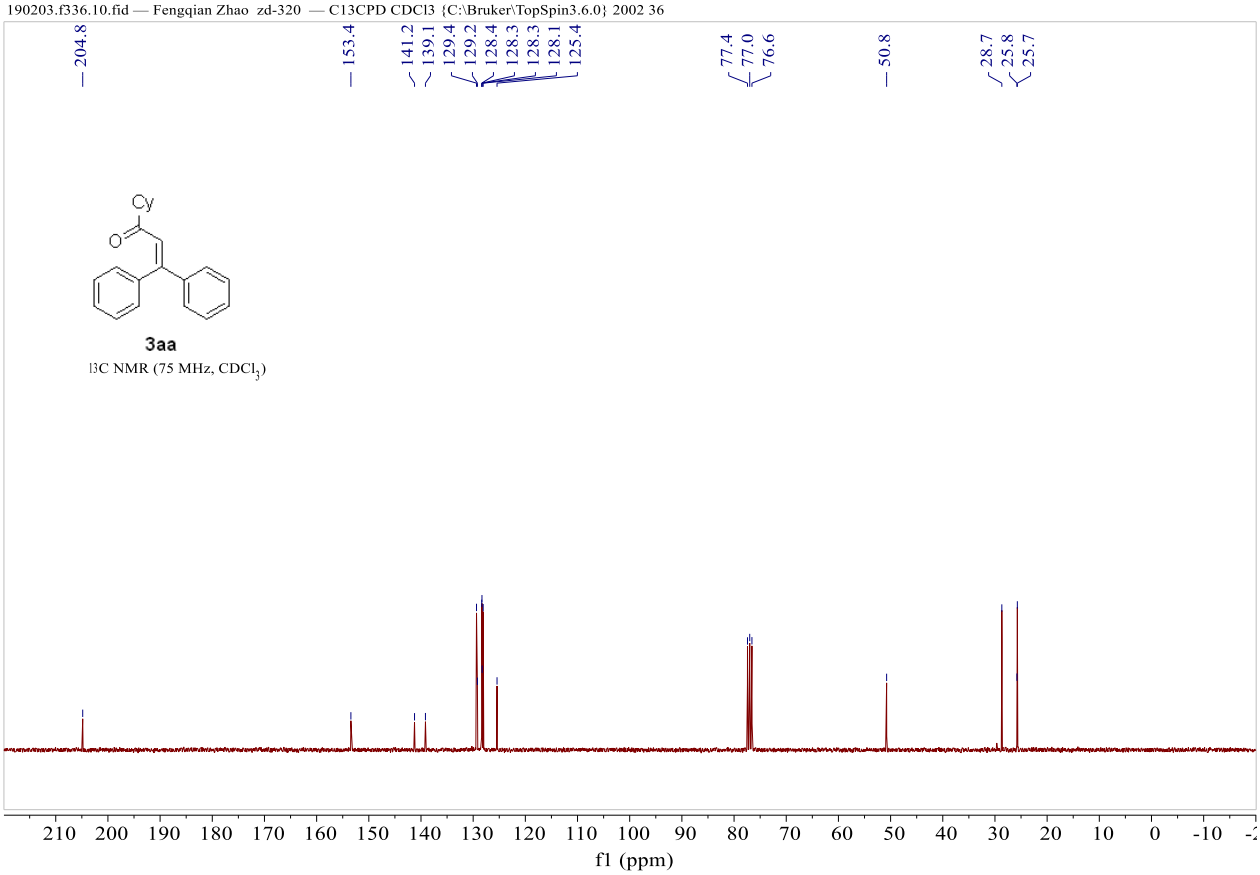
200131.407.10.fid — Fengqian Zhao zd-320 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2001 7



3aa

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

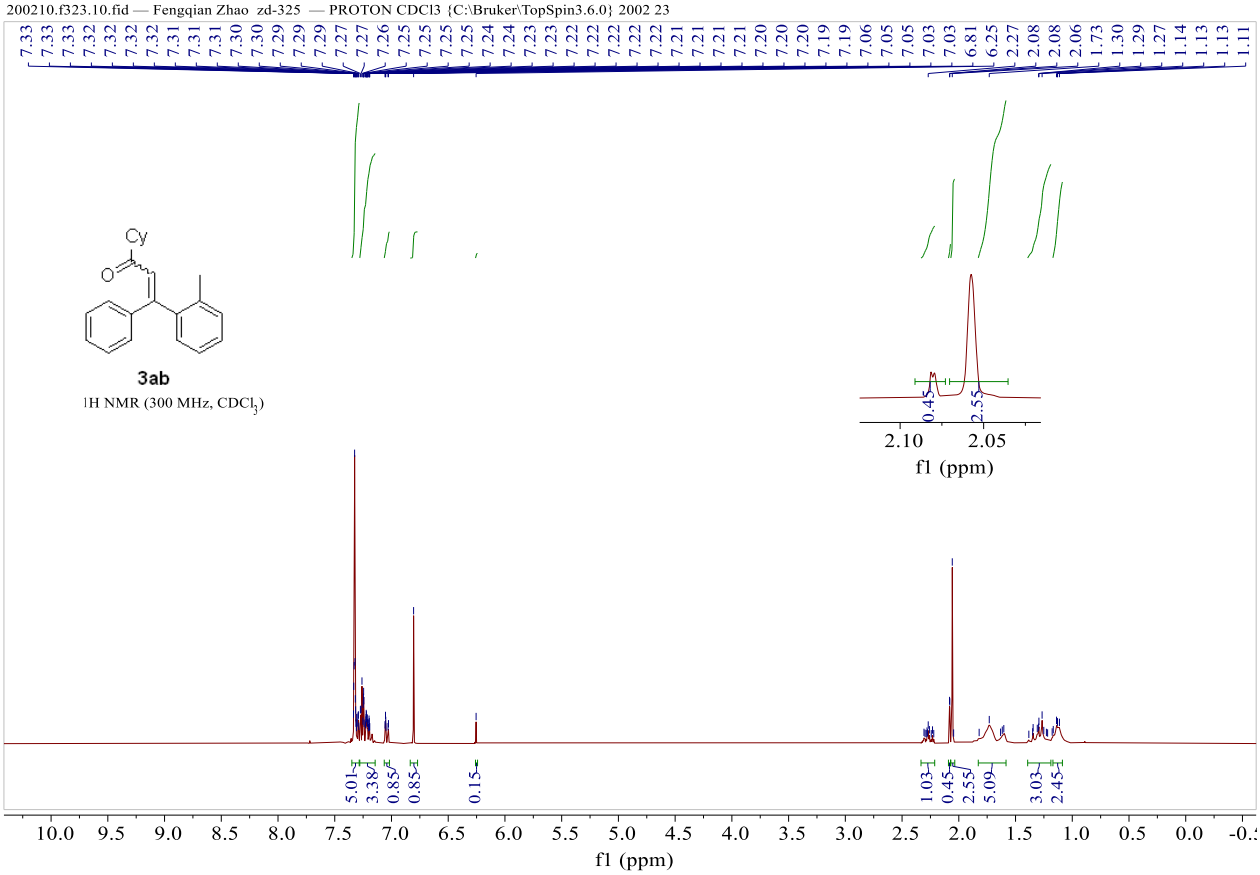
190203.f336.10.fid — Fengqian Zhao zd-320 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 36



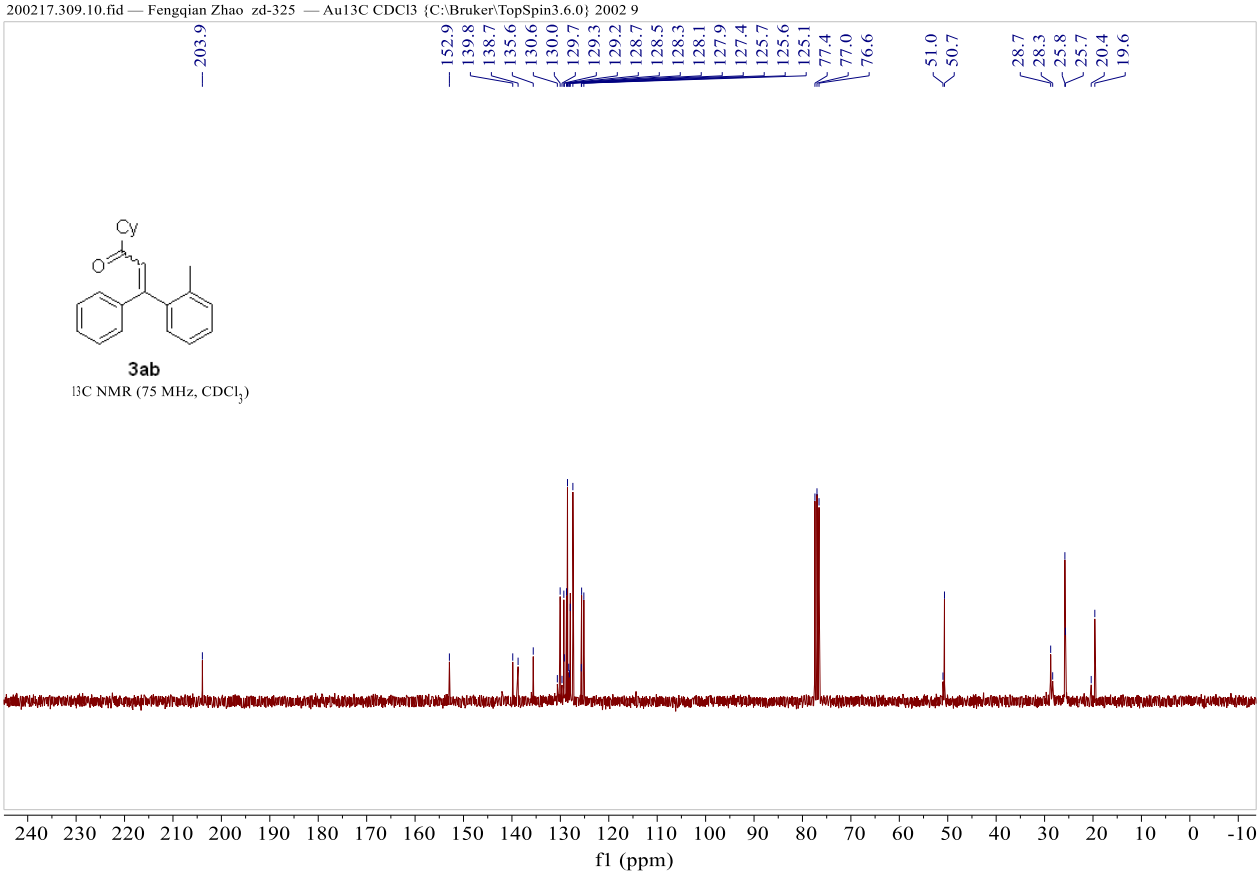
3aa

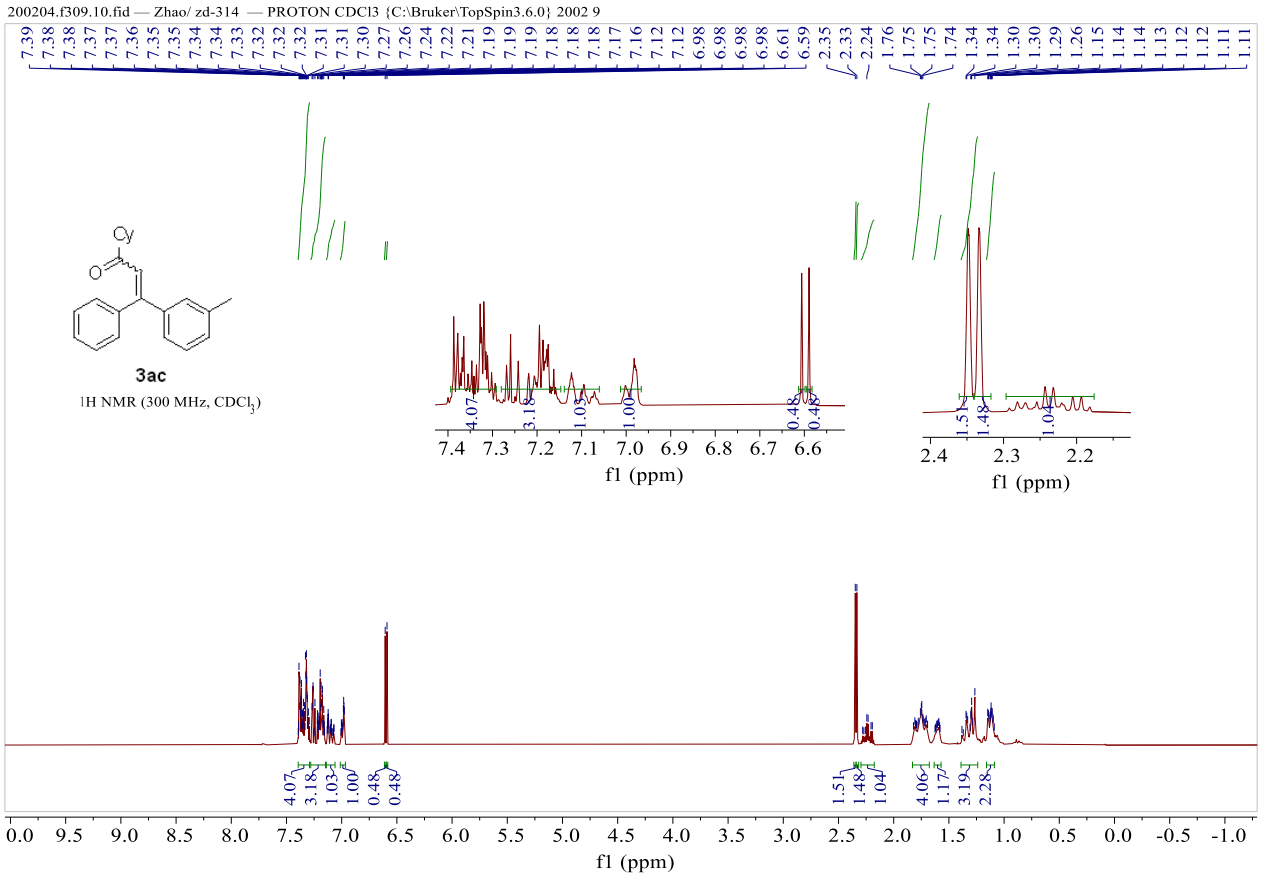
^{13}C NMR (75 MHz, $CDCl_3$)

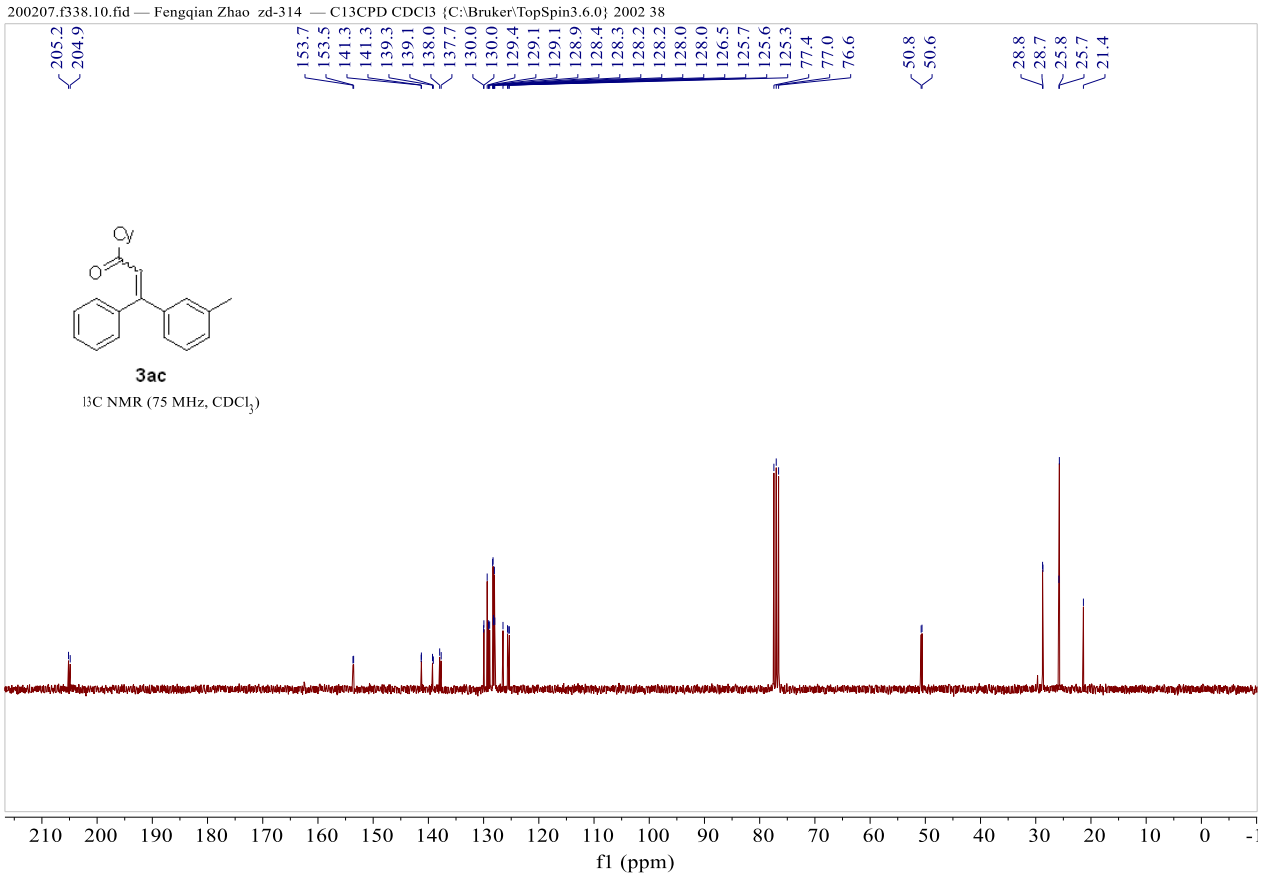
200210.f323.10.fid — Fengqian Zhao zd-325 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 23

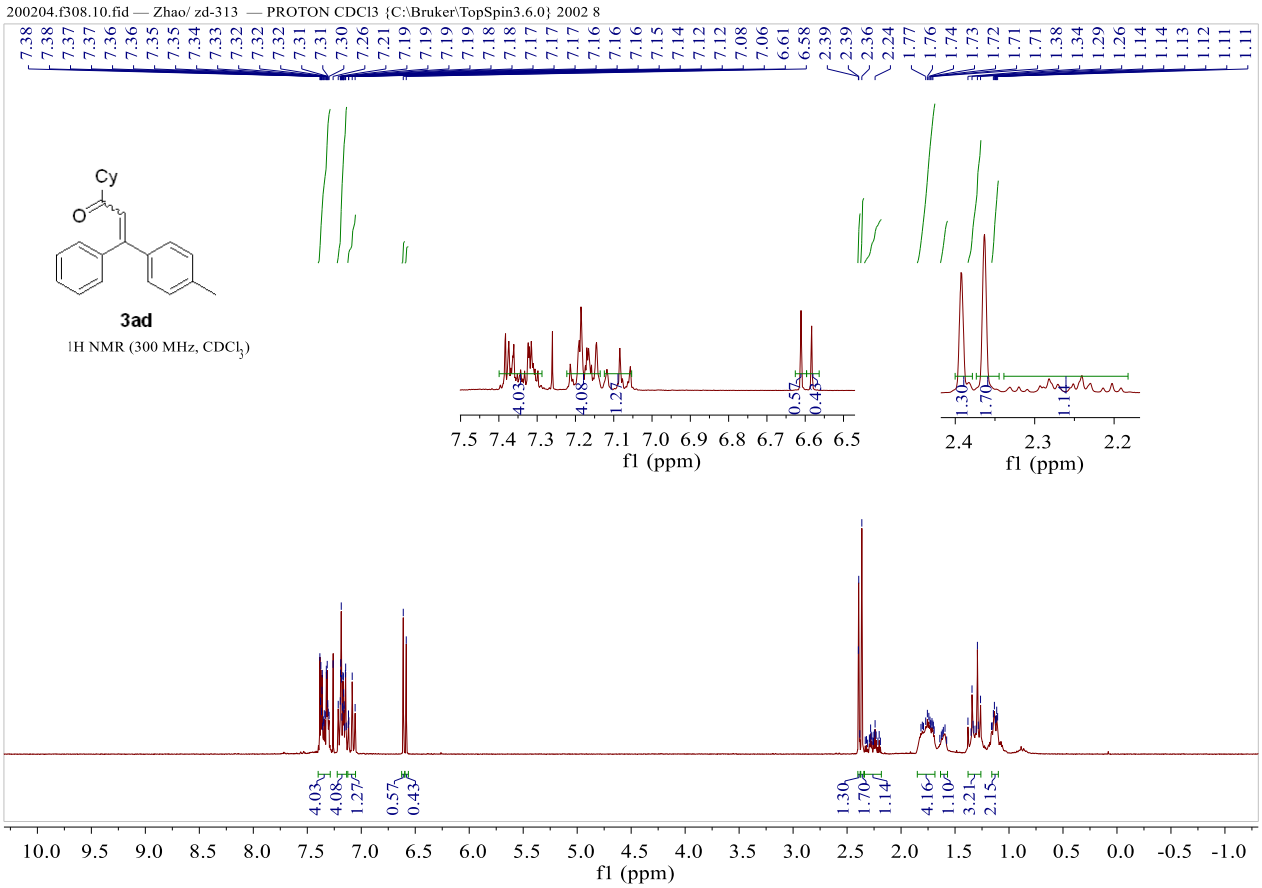


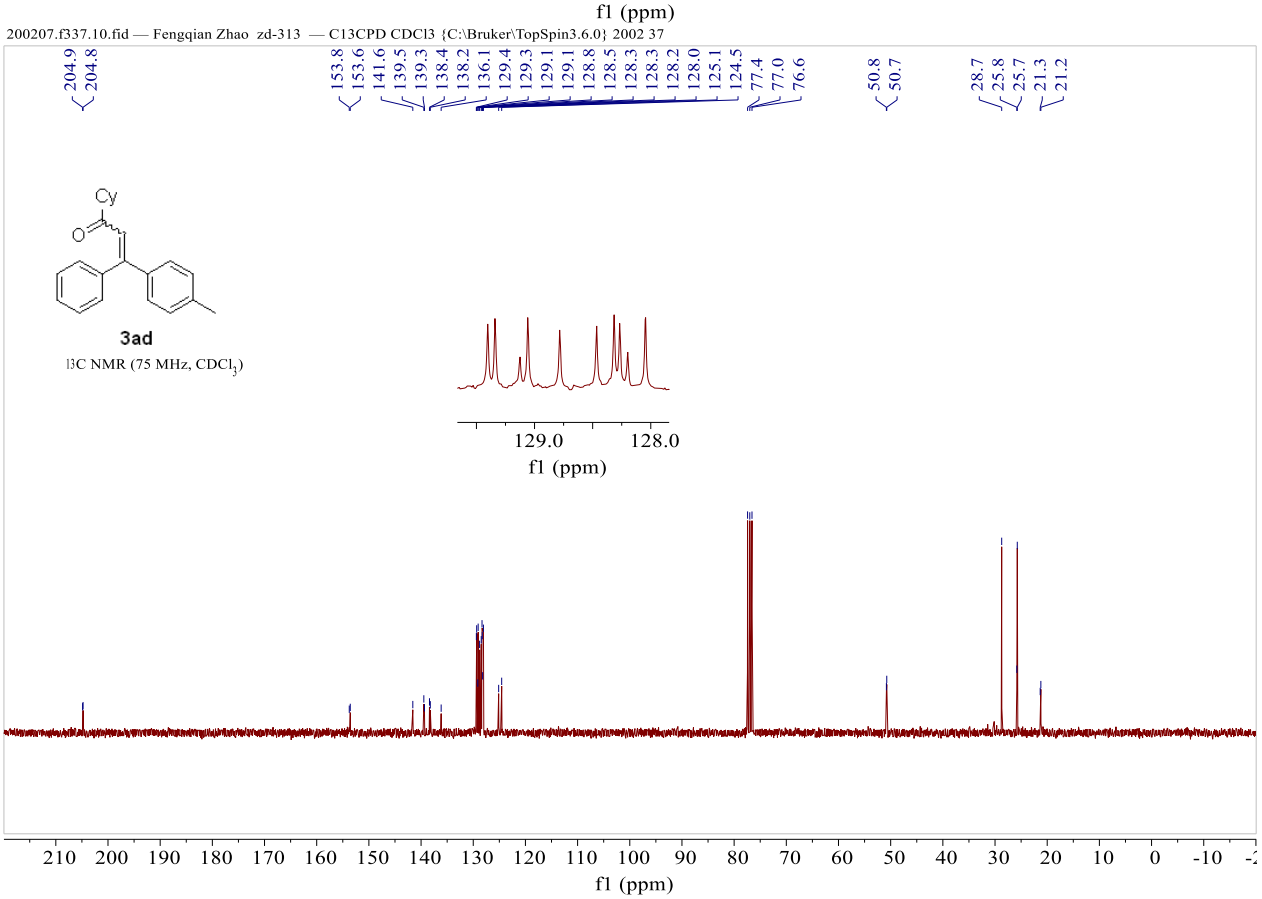
200217.309.10.fid — Fengqian Zhao zd-325 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 9

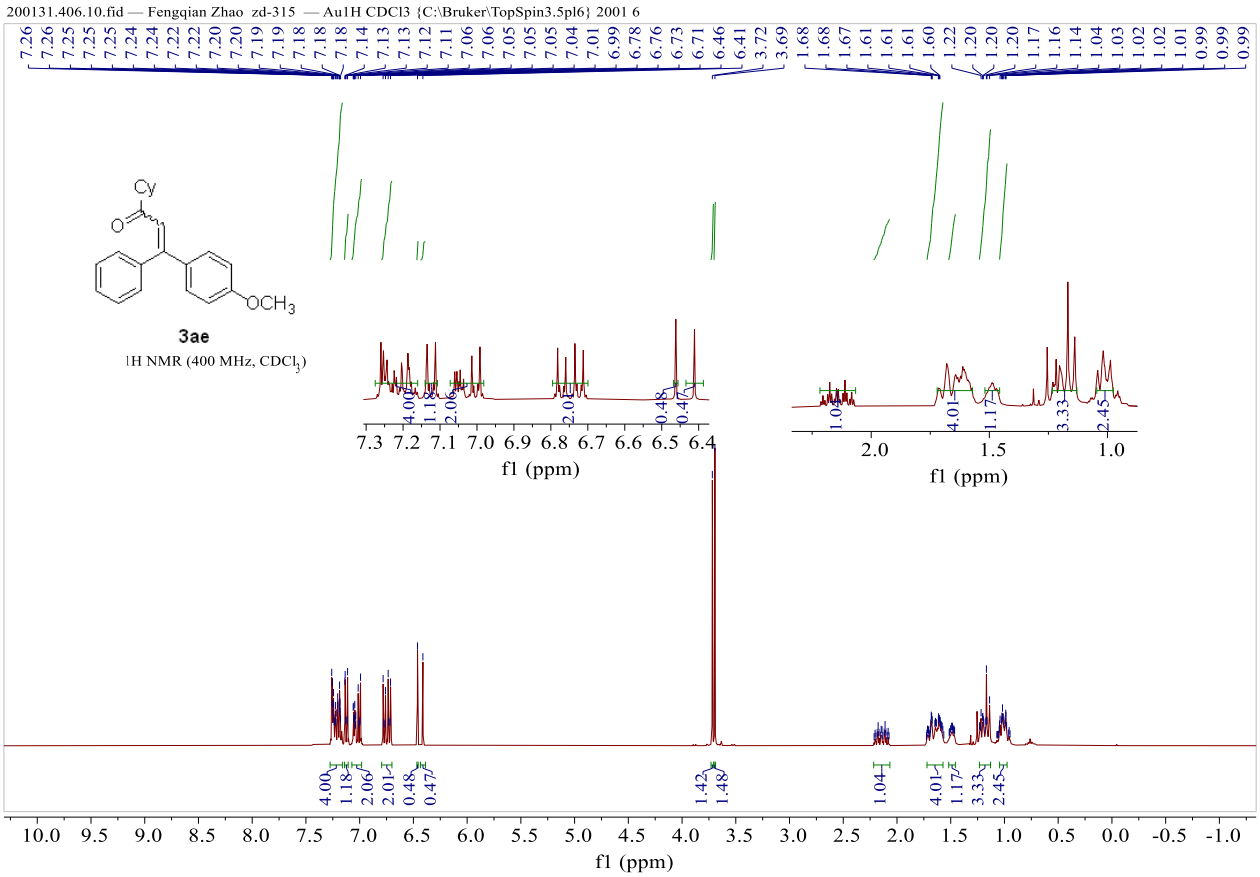


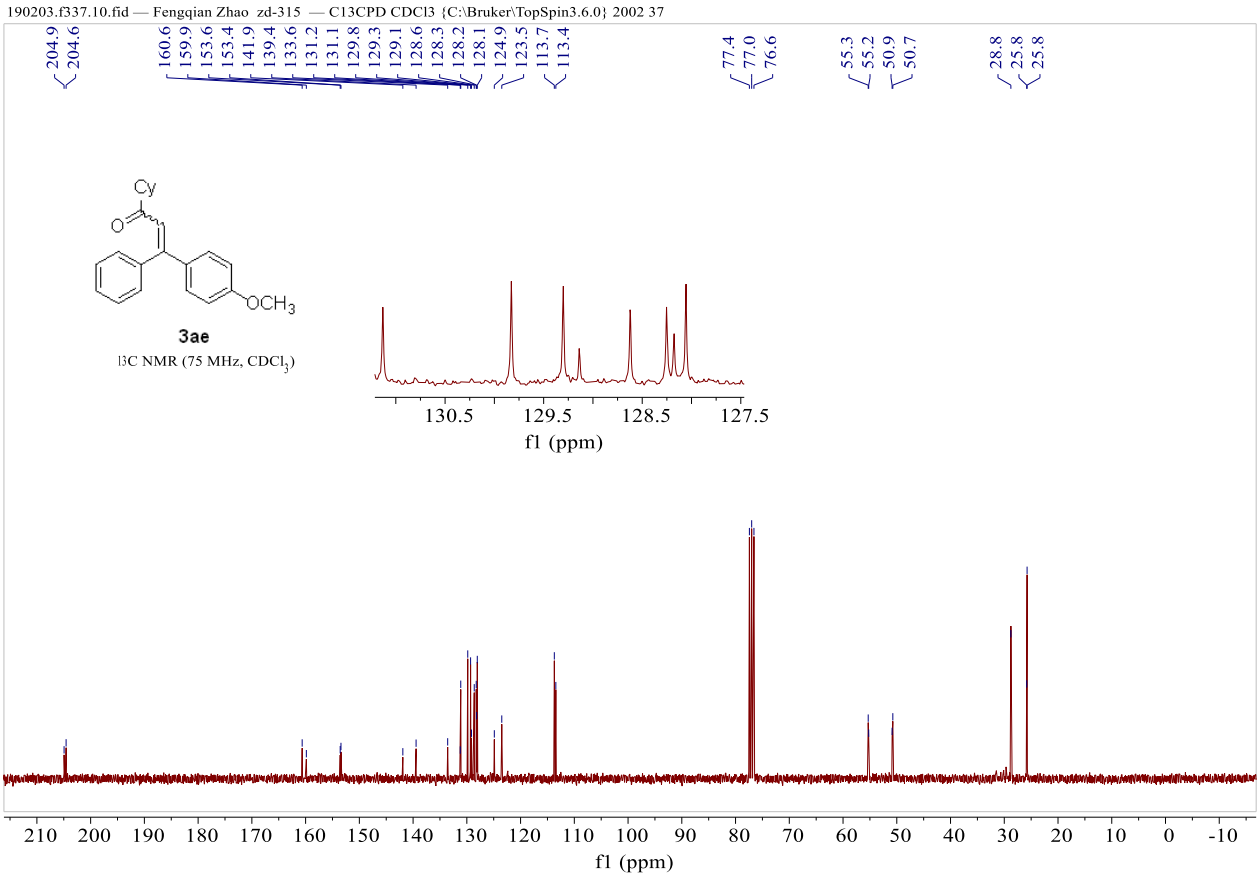

200204.f309.10.fid — Zhao/ zd-314 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 9
Cy
O
3ac
1H NMR (300 MHz, CDCl3)
f1 (ppm)

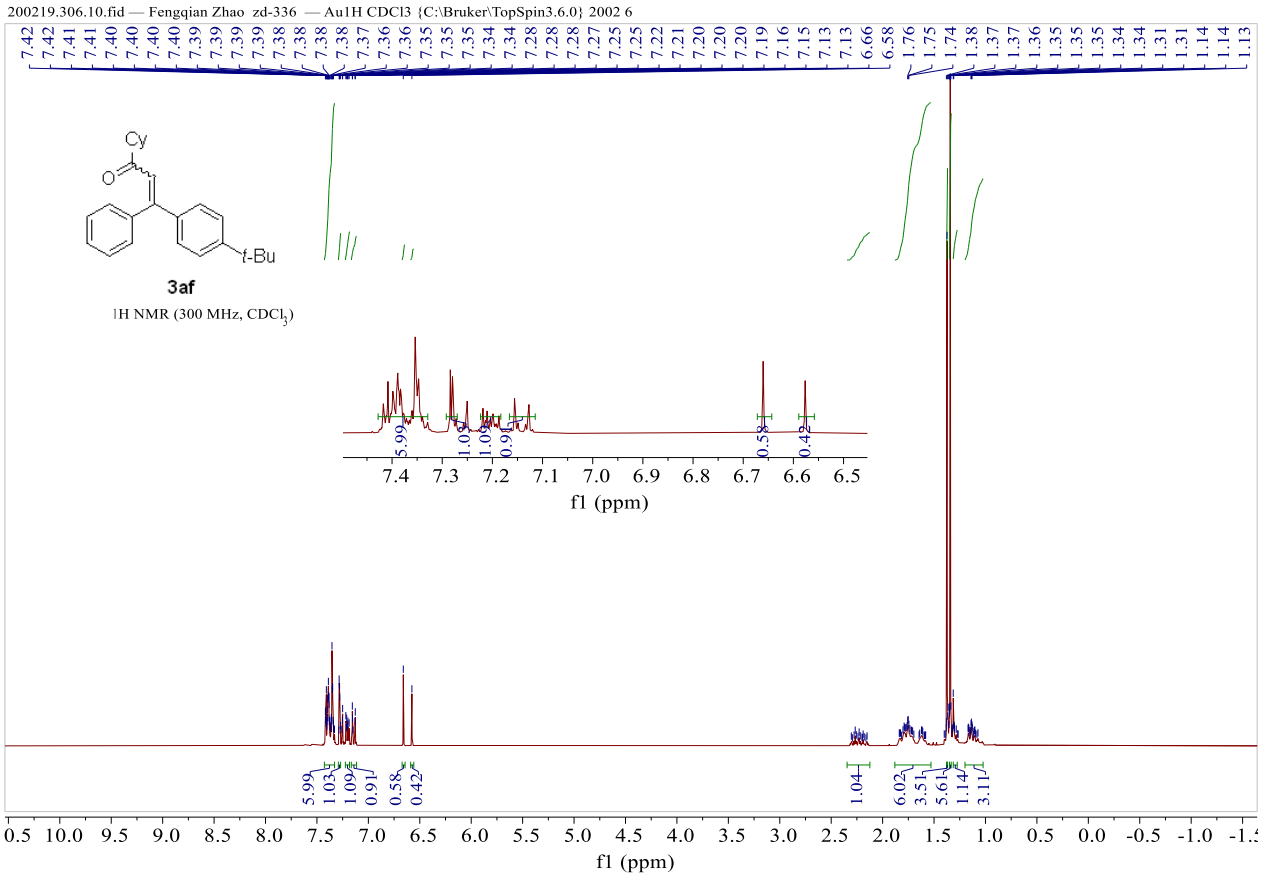

200207.f338.10.fid — Fengqian Zhao zd-314 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 38
Cy
O
3ac
13C NMR (75 MHz, CDCl3)
f1 (ppm)

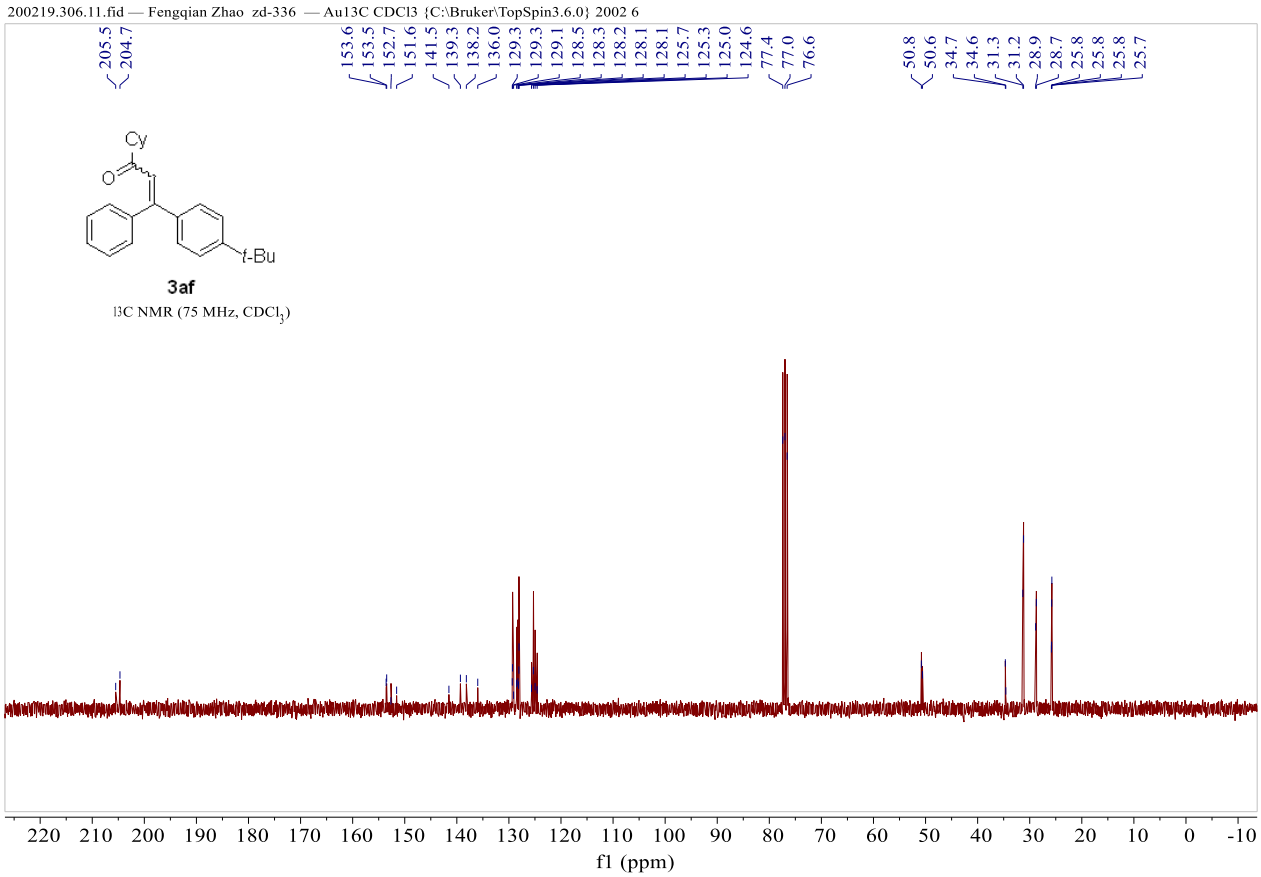

200204.f308.10.fid — Zhao/ zd-313 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 8
Cy
O
3ad
1H NMR (300 MHz, CDCl3)
f1 (ppm)

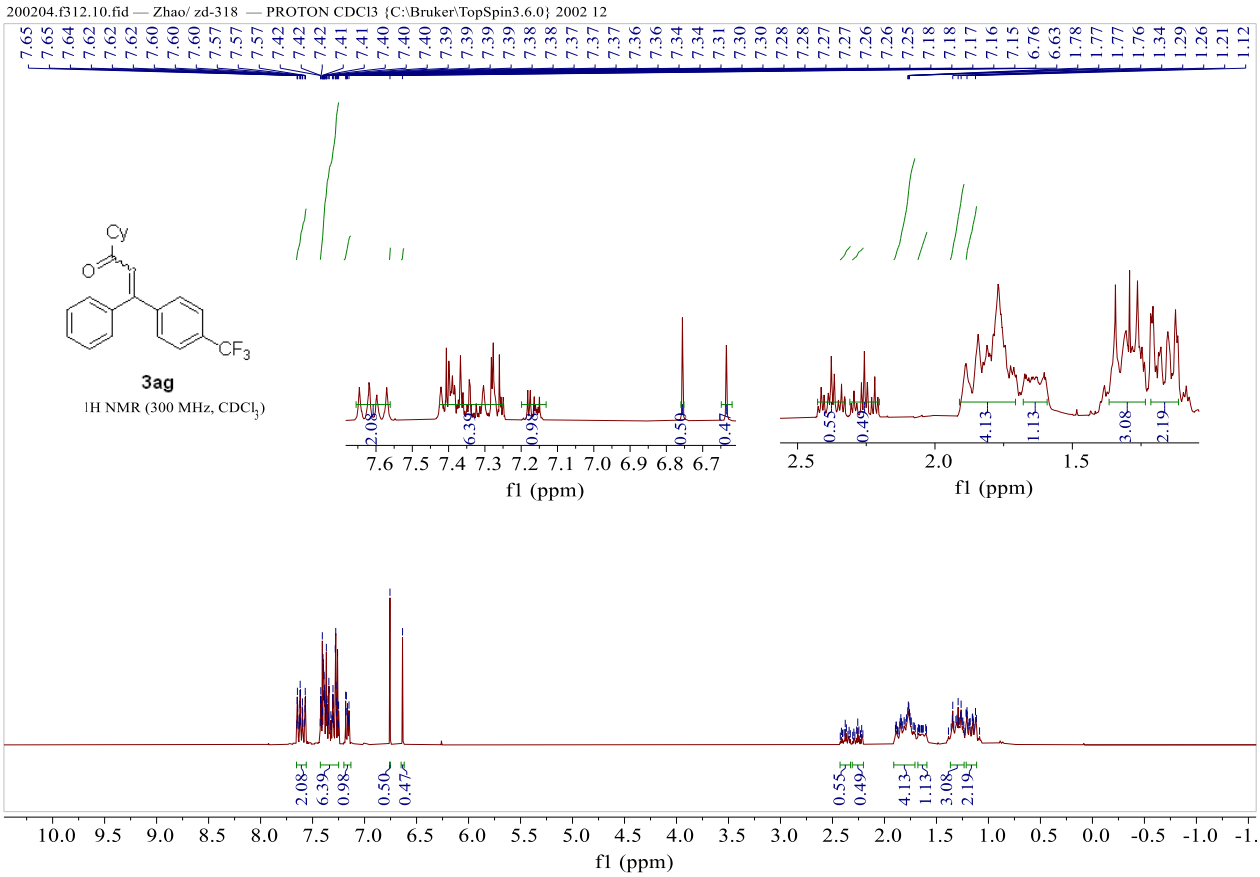

200207.f337.10.fid — Fengqian Zhao zd-313 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 37
Cy
O
3ad
13C NMR (75 MHz, CDCl3)
f1 (ppm)

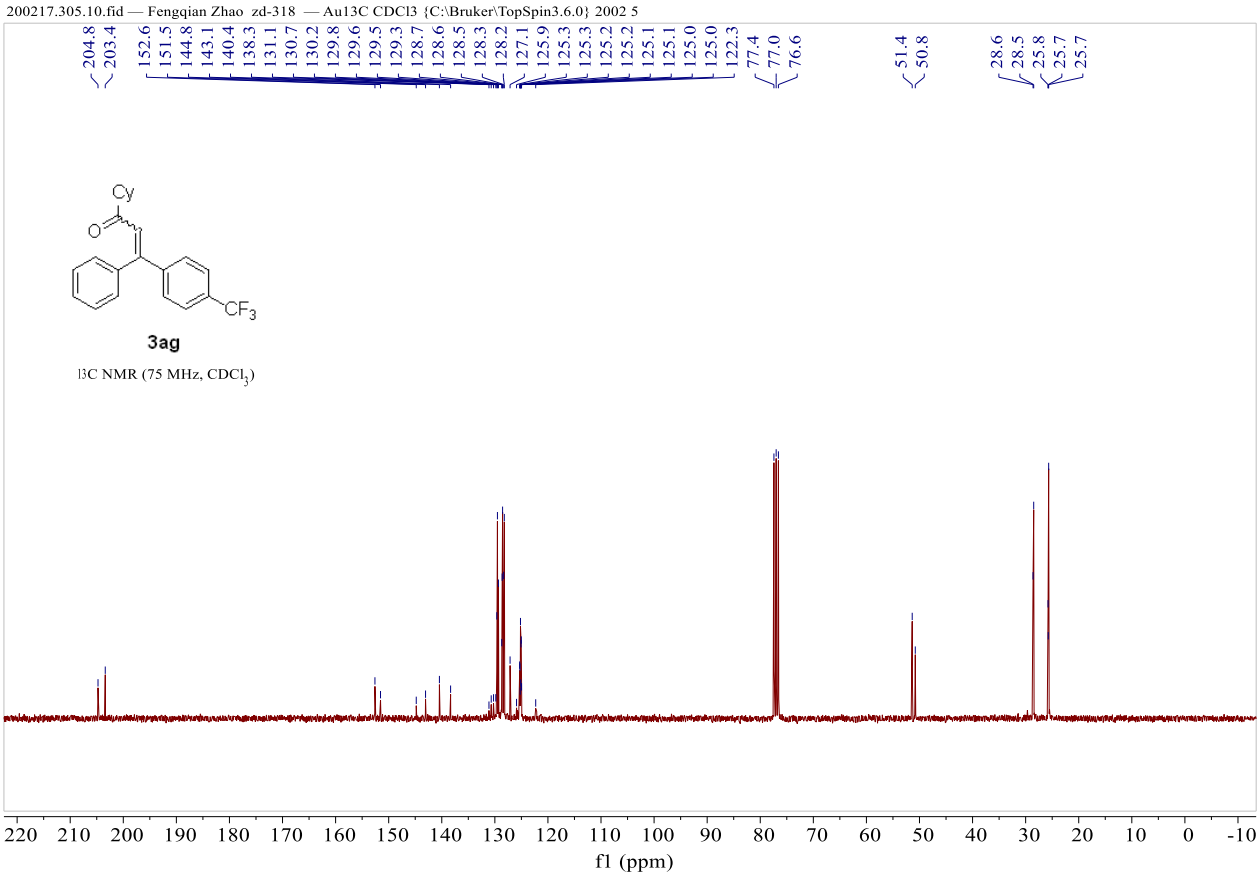

200131.406.10.fid — Fengqian Zhao zd-315 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2001 6
Cy
O
OCH3
3ae
1H NMR (400 MHz, CDCl3)
f1 (ppm)


190203.f337.10.fid — Fengqian Zhao zd-315 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 37
Cy
O
OCH3
3ae
13C NMR (75 MHz, CDCl3)
f1 (ppm)

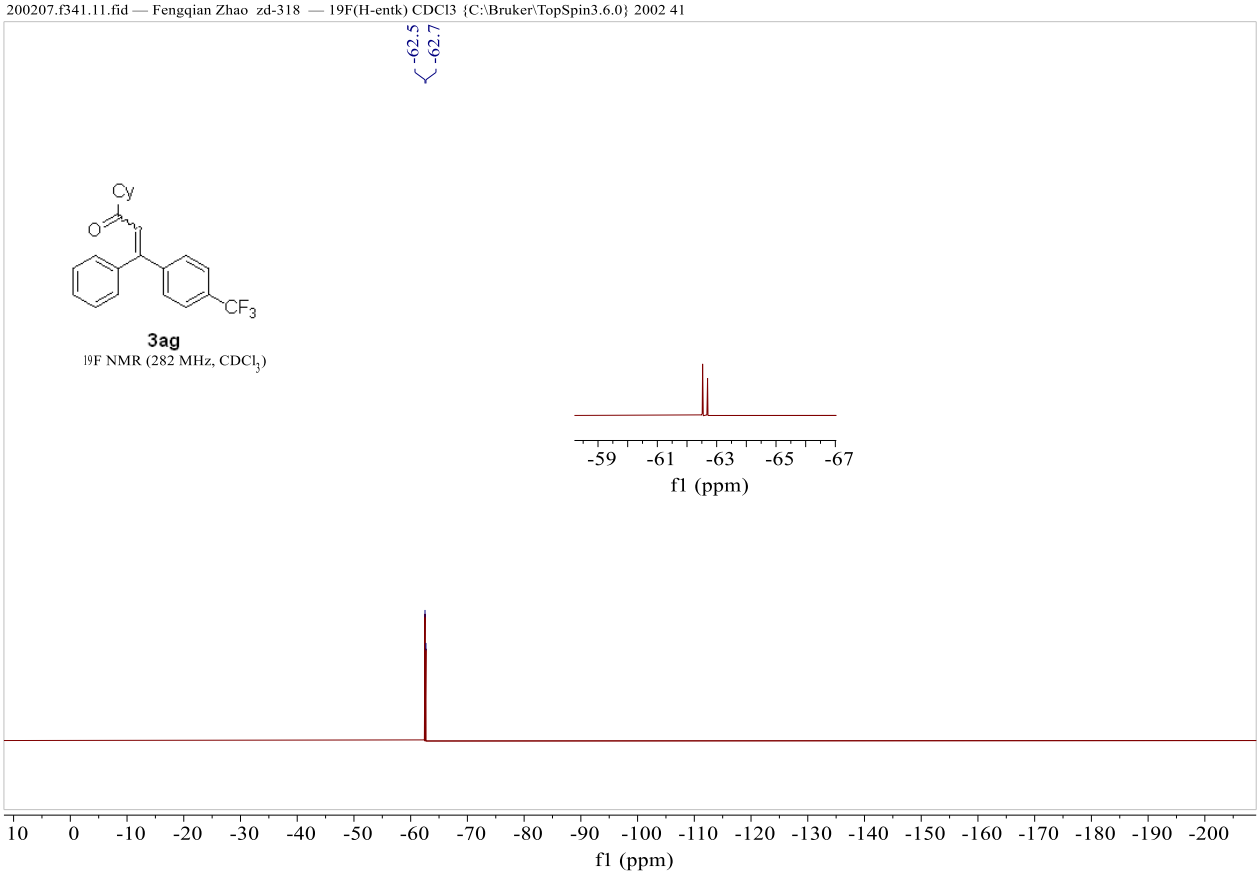

200219.306.10.fid — Fengqian Zhao zd-336 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 6
Cy
O
t-Bu
3af
1H NMR (300 MHz, CDCl3)
f1 (ppm)


200219.306.11.fid — Fengqian Zhao zd-336 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 6
Cy
O
t-Bu
3af
13C NMR (75 MHz, CDCl3)
f1 (ppm)


200204.f312.10.fid — Zhao/ zd-318 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 12
Cy
O
CF3
3ag
1H NMR (300 MHz, CDCl3)
f1 (ppm)
f1 (ppm)
f1 (ppm)


200217.305.10.fid — Fengqian Zhao zd-318 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 5
Cy
O
CF3
3ag
13C NMR (75 MHz, CDCl3)
f1 (ppm)

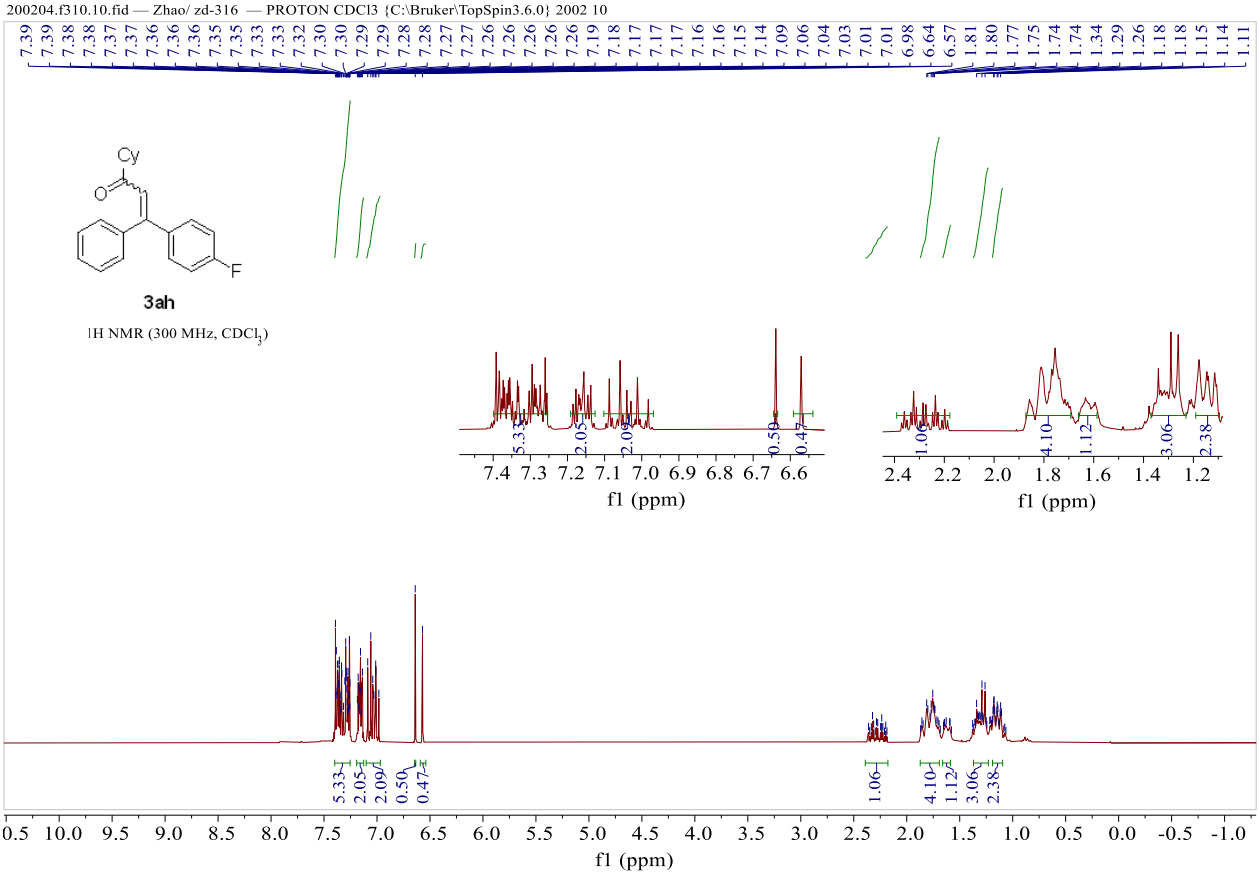
200207.f341.11.fid — Fengqian Zhao zd-318 — 19F(H-entk) CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 41



3ag

^{19}F NMR (282 MHz, $CDCl_3$)

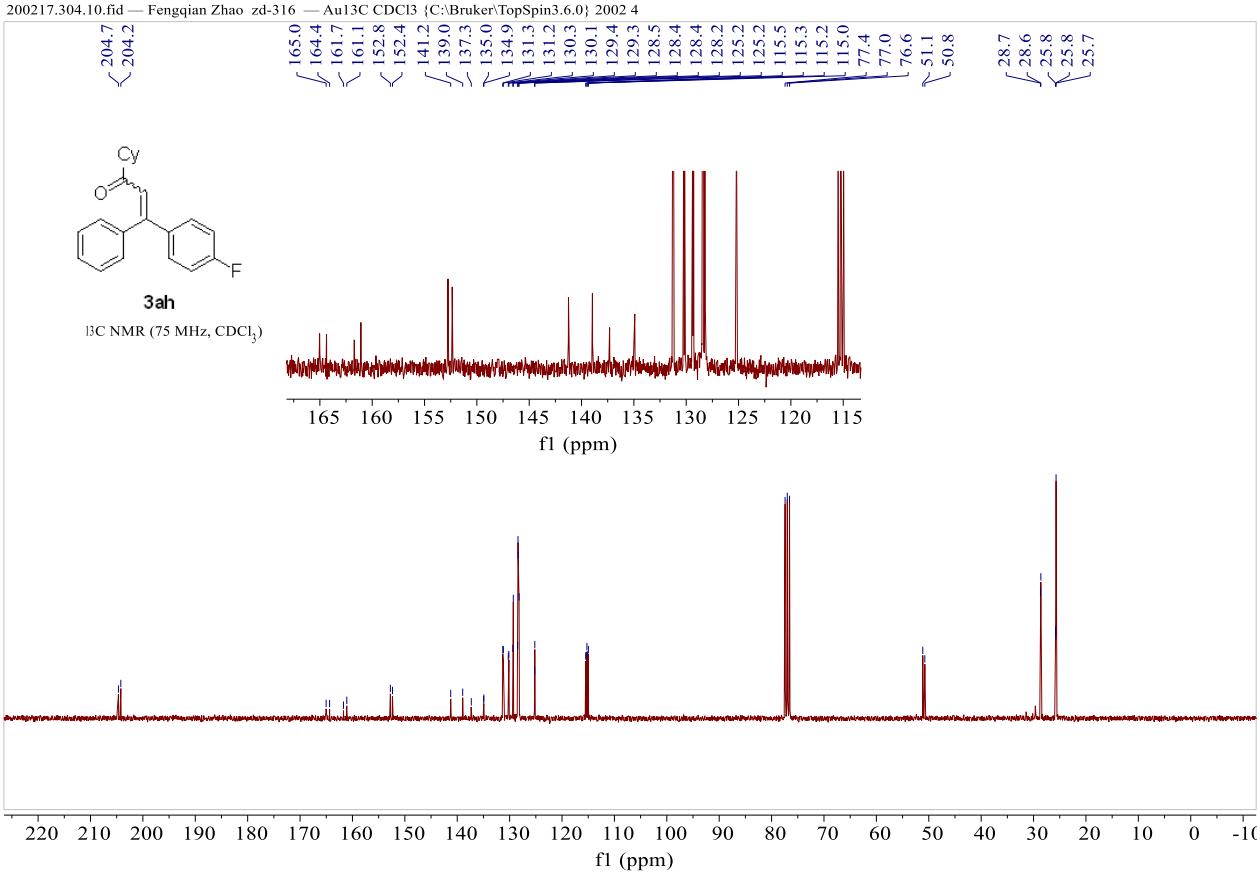
200204.f310.10.fid — Zhao/ zd-316 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 10



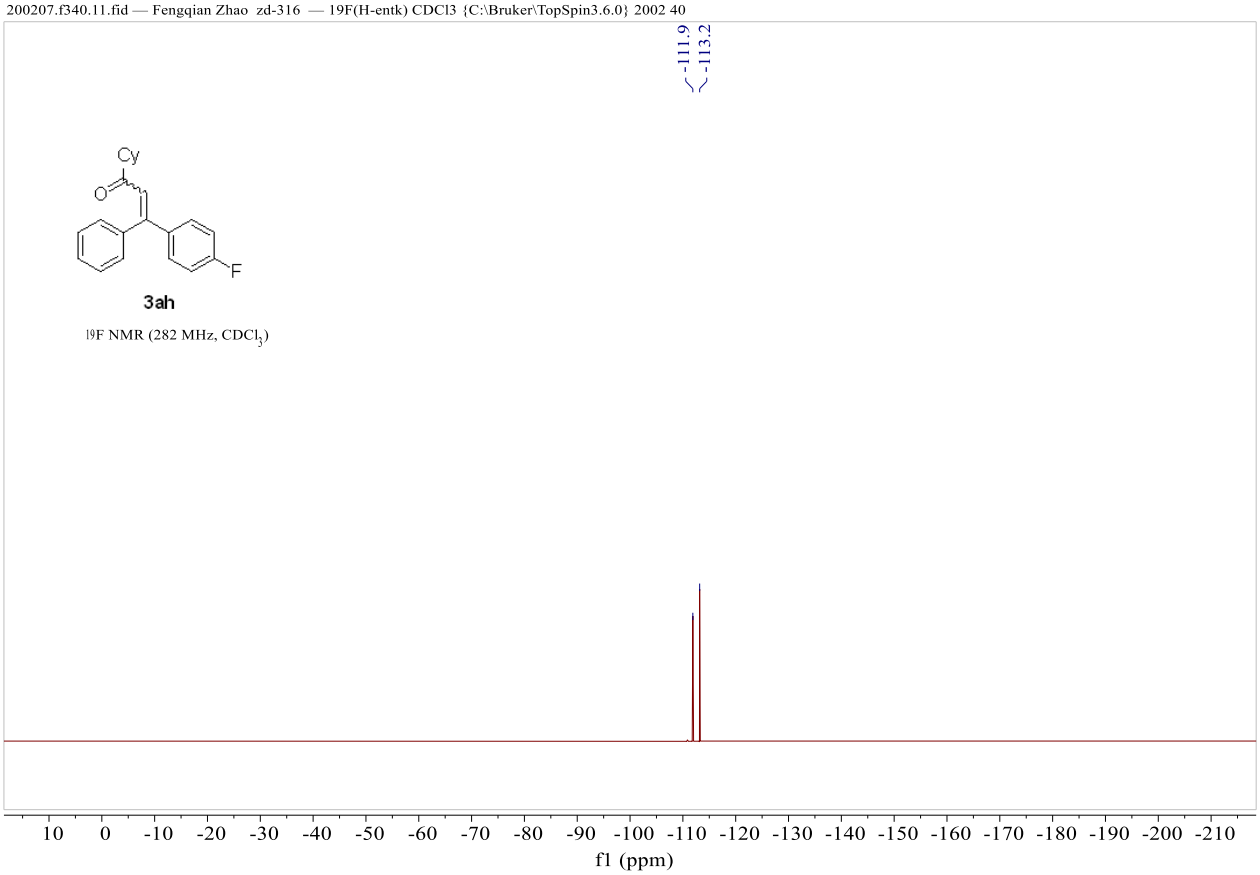
3ah

^{1}H NMR (300 MHz, $CDCl_3$)

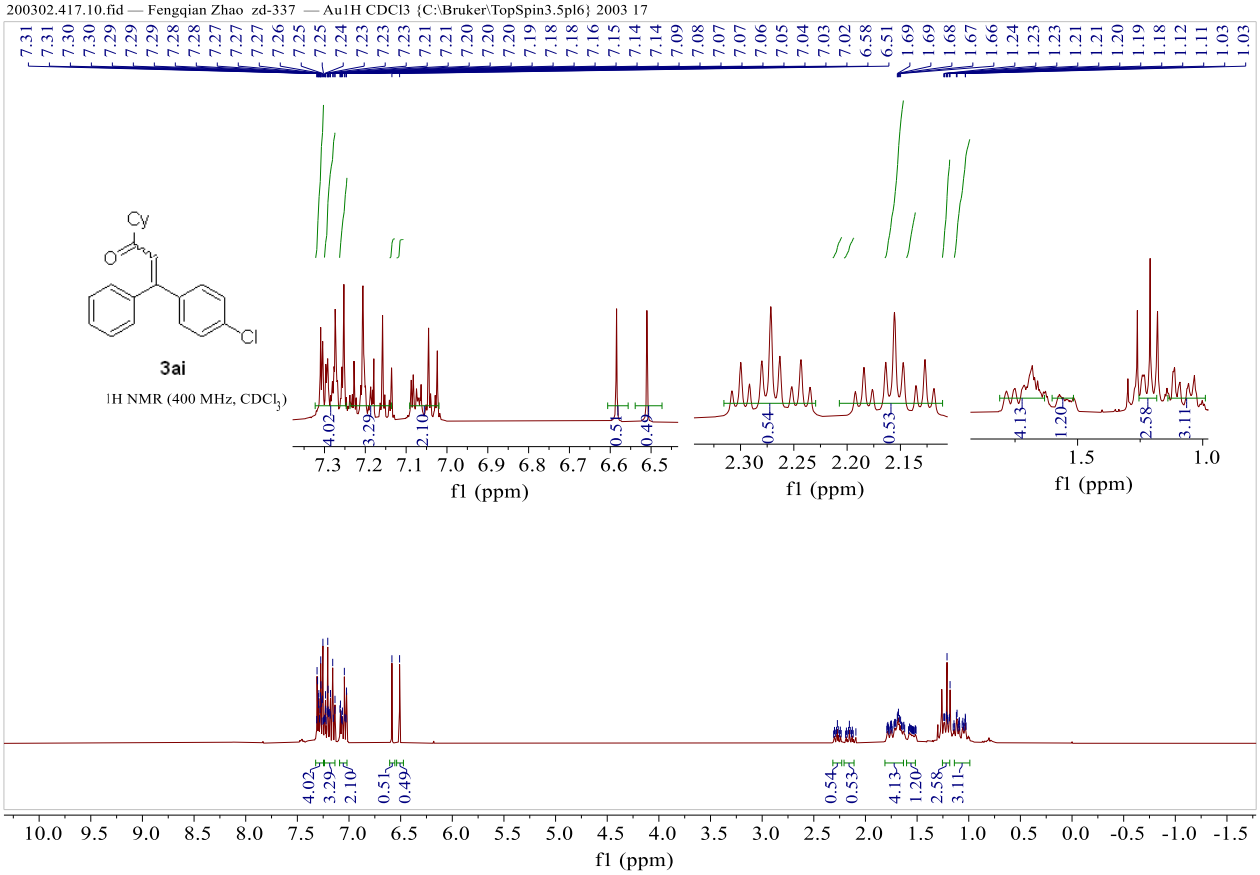
200217.304.10.fid — Fengqian Zhao zd-316 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 4



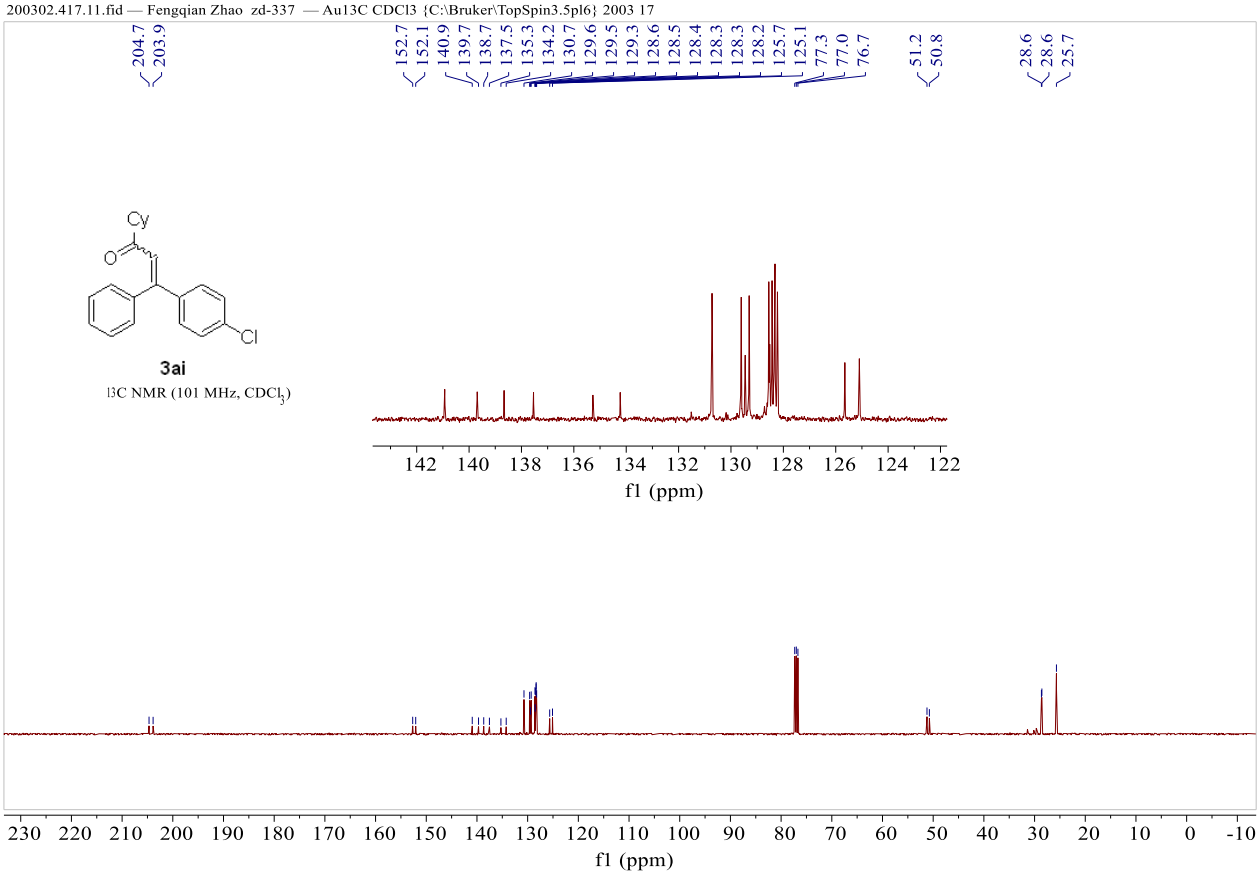
200207.f340.11.fid — Fengqian Zhao zd-316 — 19F(H-entk) CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 40

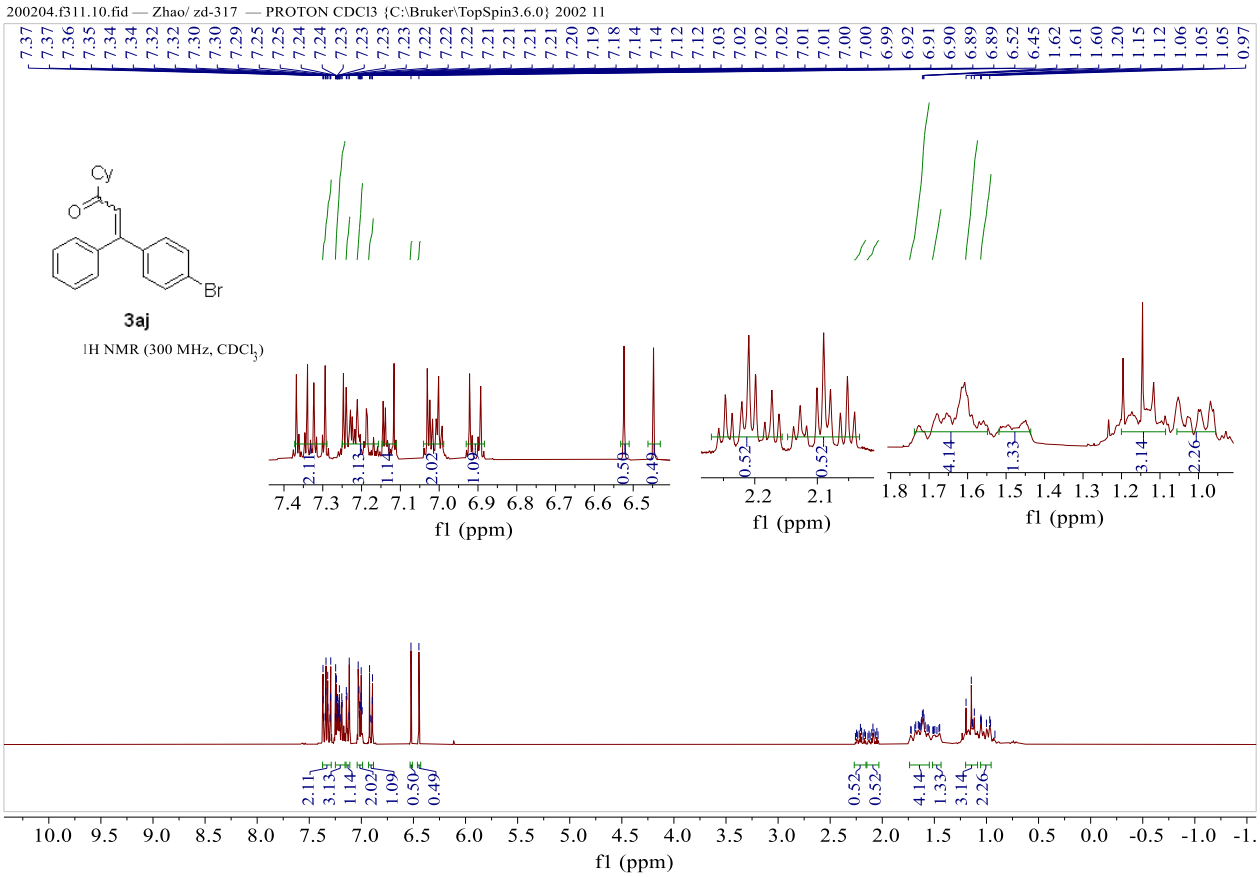


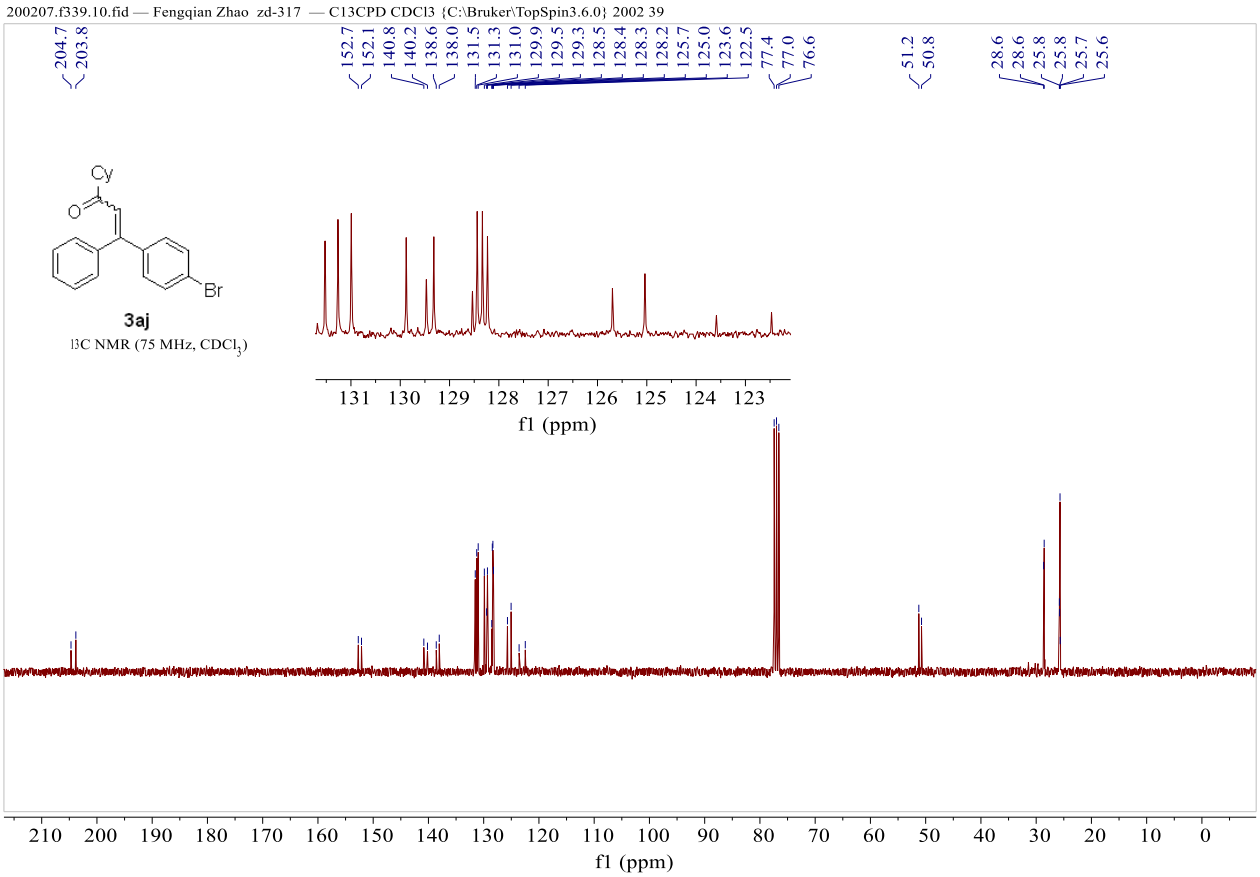
200302.417.10.fid — Fengqian Zhao zd-337 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 17

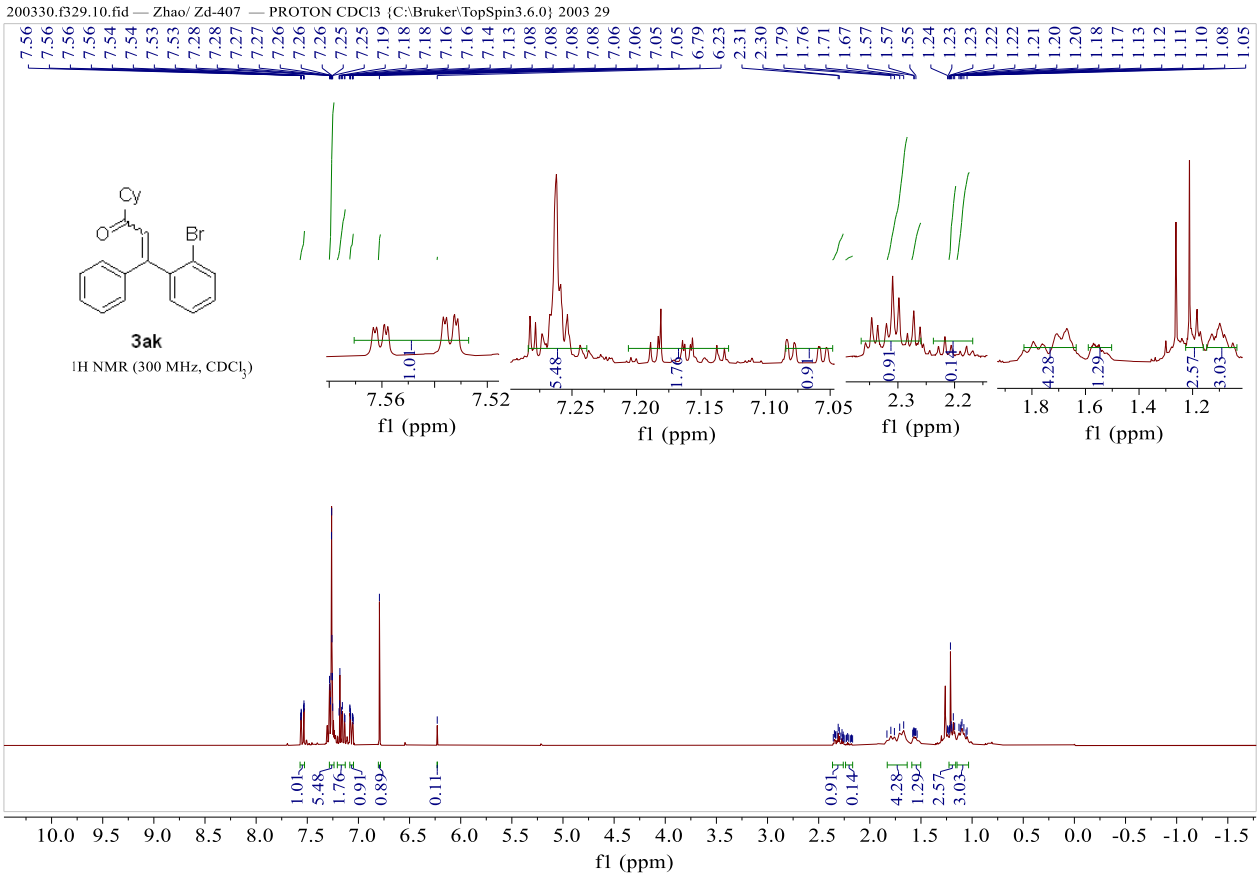


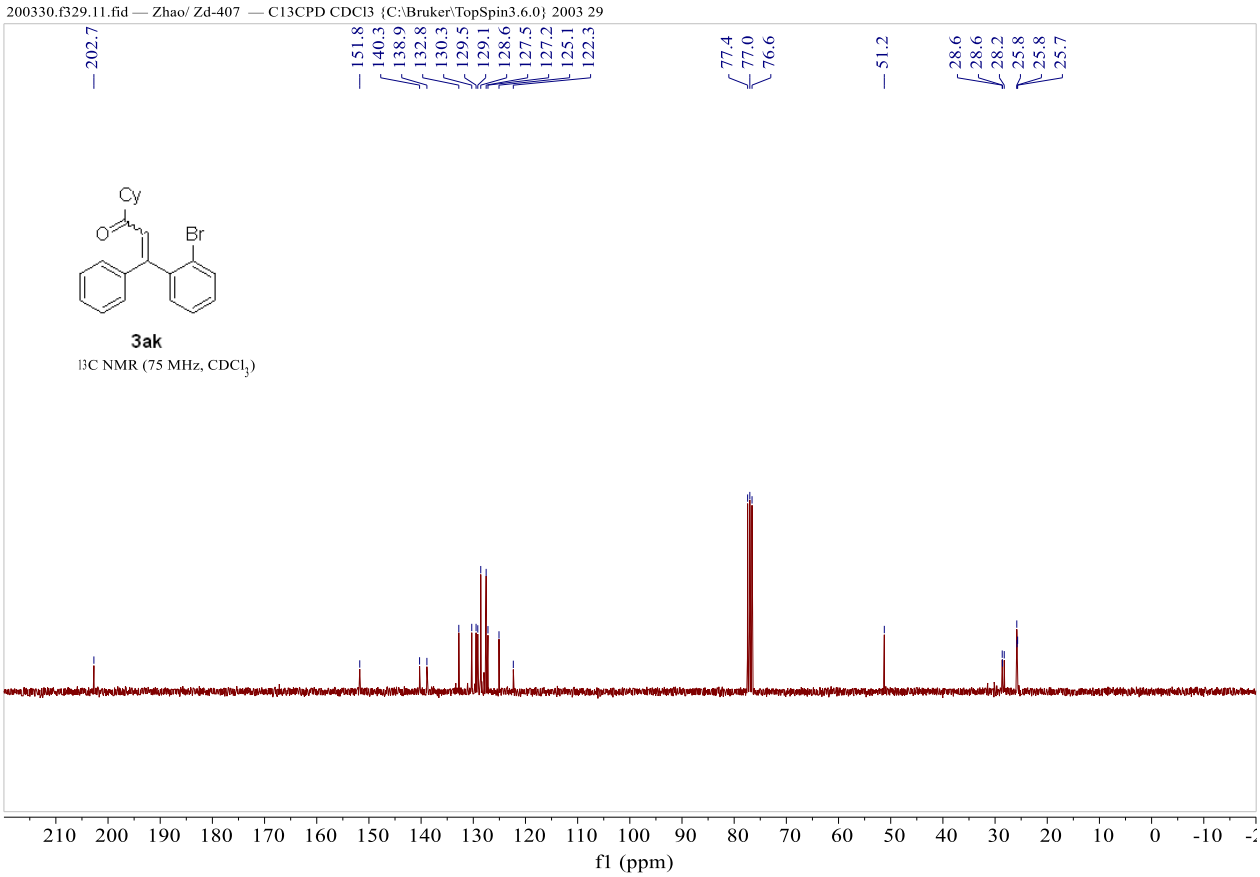
200302.417.11.fid — Fengqian Zhao zd-337 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 17



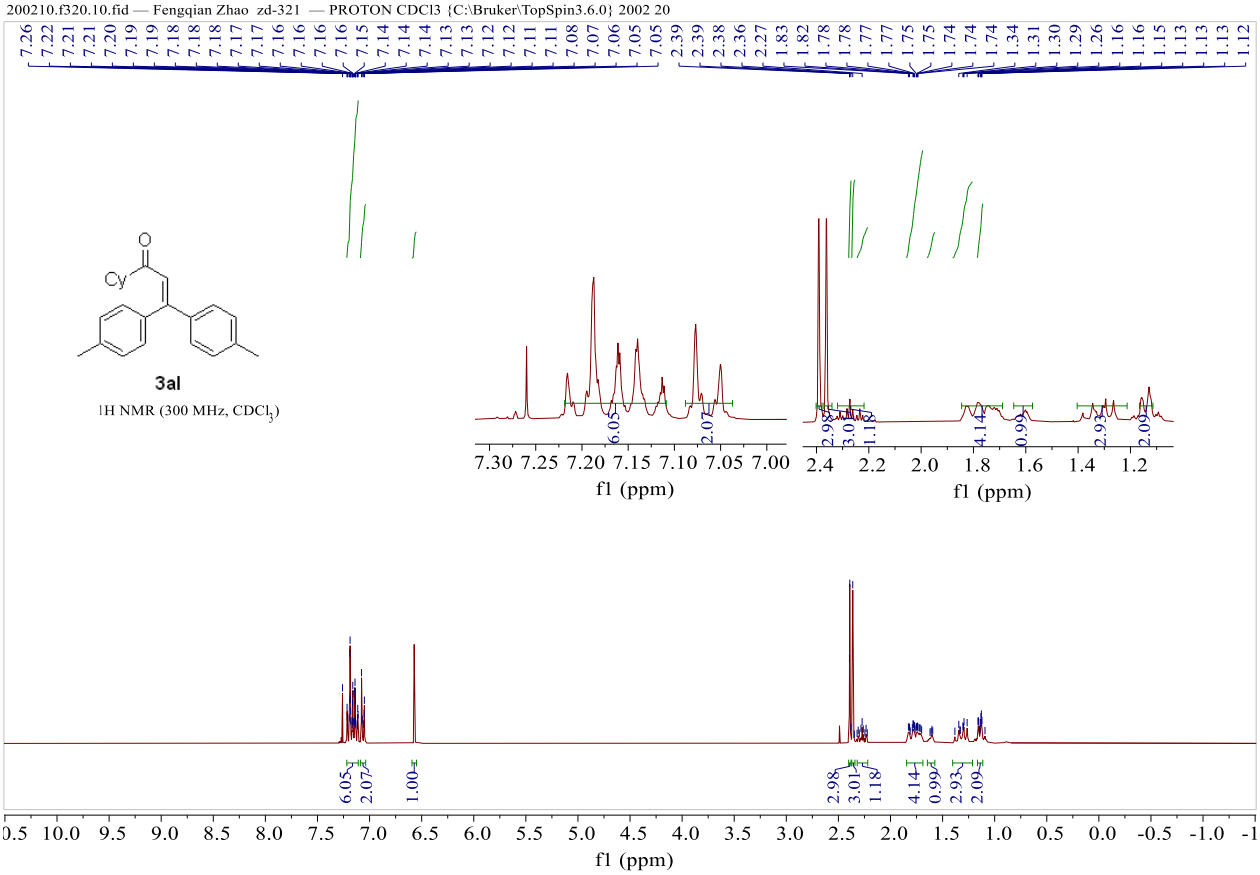

200204.f311.10.fid — Zhao/ zd-317 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 11
Cy
O
Br
3aj
1H NMR (300 MHz, CDCl3)
f1 (ppm)


200207.f339.10.fid — Fengqian Zhao zd-317 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 39
Cy
O
Br
3aj
13C NMR (75 MHz, CDCl3)
f1 (ppm)

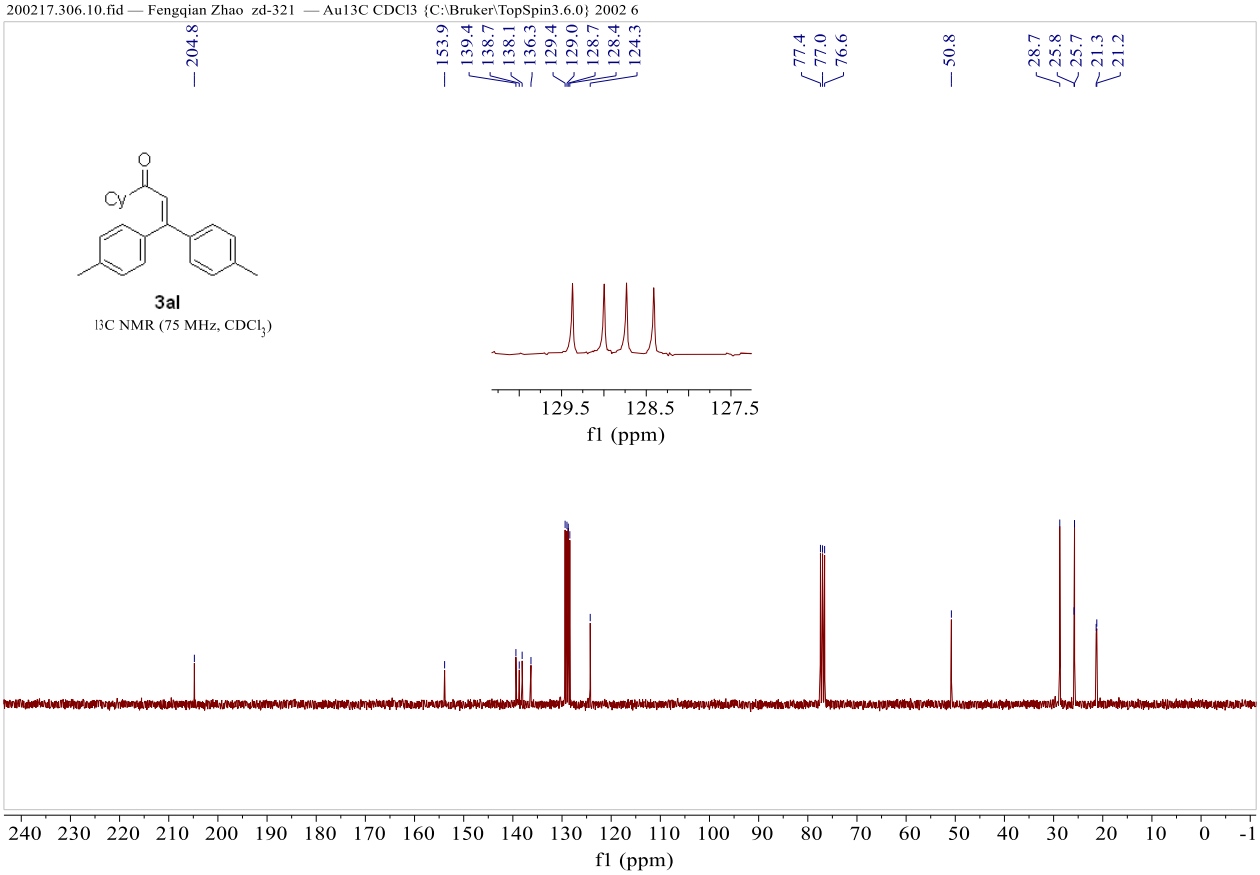

200330.f329.10.fid — Zhao/ Zd-407 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2003 29
Cy
O
Br
3ak
1H NMR (300 MHz, CDCl3)
f1 (ppm)


200330.f329.11.fid — Zhao/ Zd-407 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2003 29
Cy
O
Br
3ak
13C NMR (75 MHz, CDCl3)
f1 (ppm)

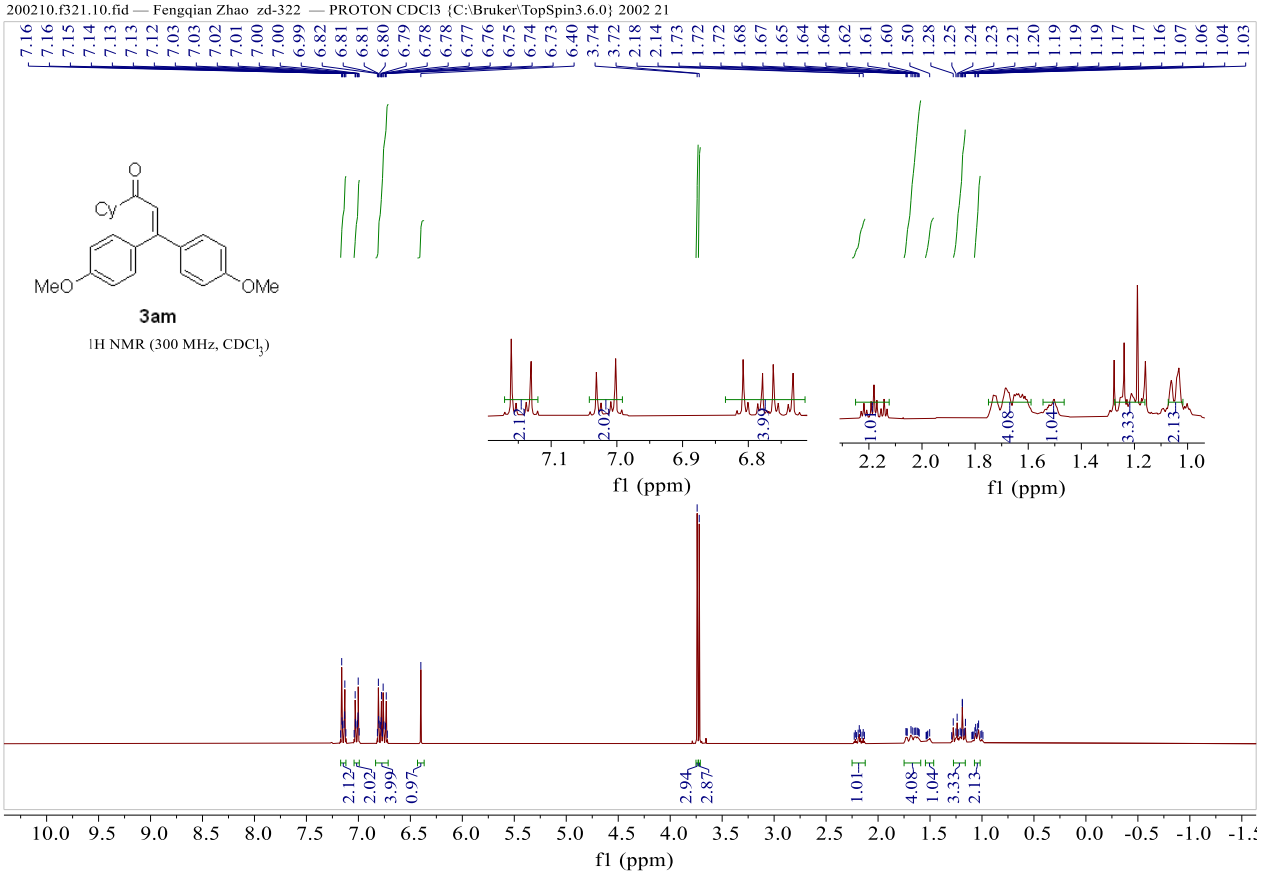
200210.f320.10.fid — Fengqian Zhao zd-321 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 20



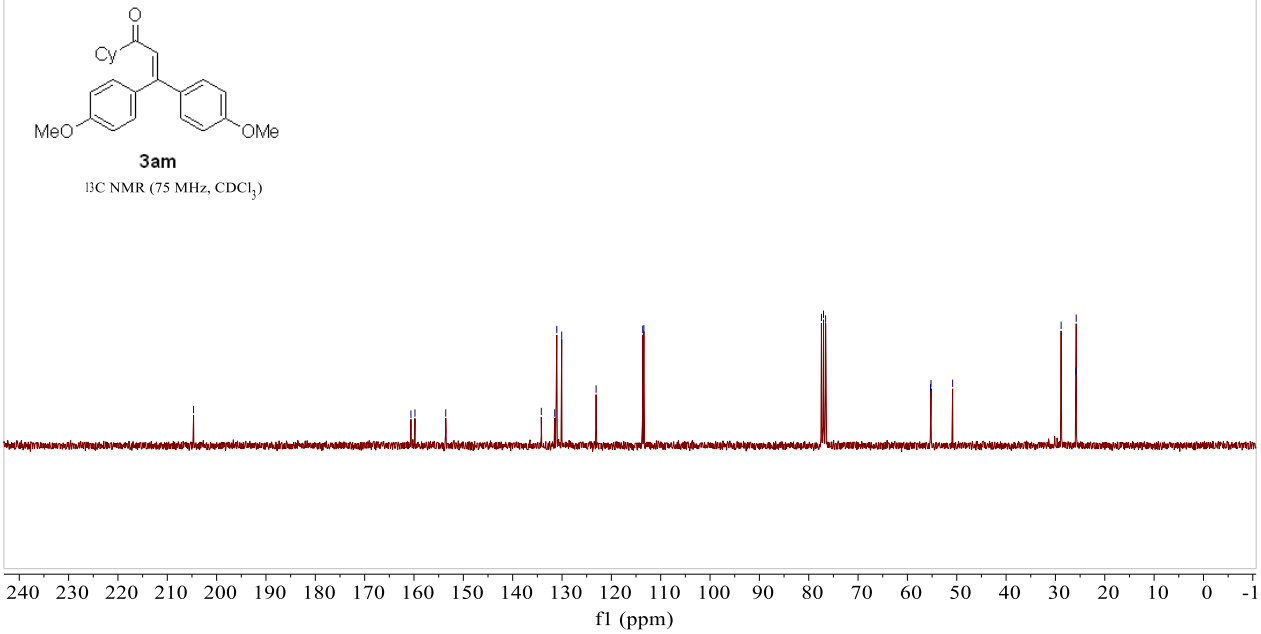
200217.306.10.fid — Fengqian Zhao zd-321 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 6



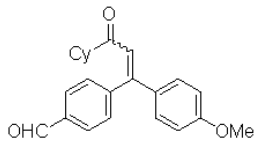
200210.f321.10.fid — Fengqian Zhao zd-322 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 21



200217.307.10.fid — Fengqian Zhao zd-322 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 7



200313.f320.10.fid — Fengqian Zhao zd-376 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2003 20

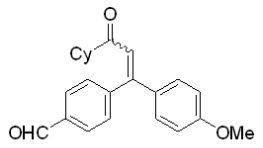


3an

^{1}H NMR (300 MHz, $CDCl_3$)

f1 (ppm)

200408.f341.11.fid — Fengqian Zhao zd-376 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2004 41



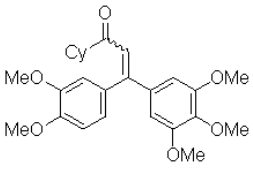
3an

^{13}C NMR (75 MHz, $CDCl_3$)

acetone

f1 (ppm)

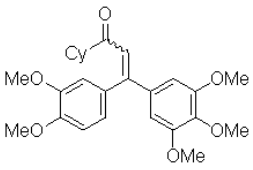
200313.f321.10.fid — Fengqian Zhao zd-377 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2003 21



3ao

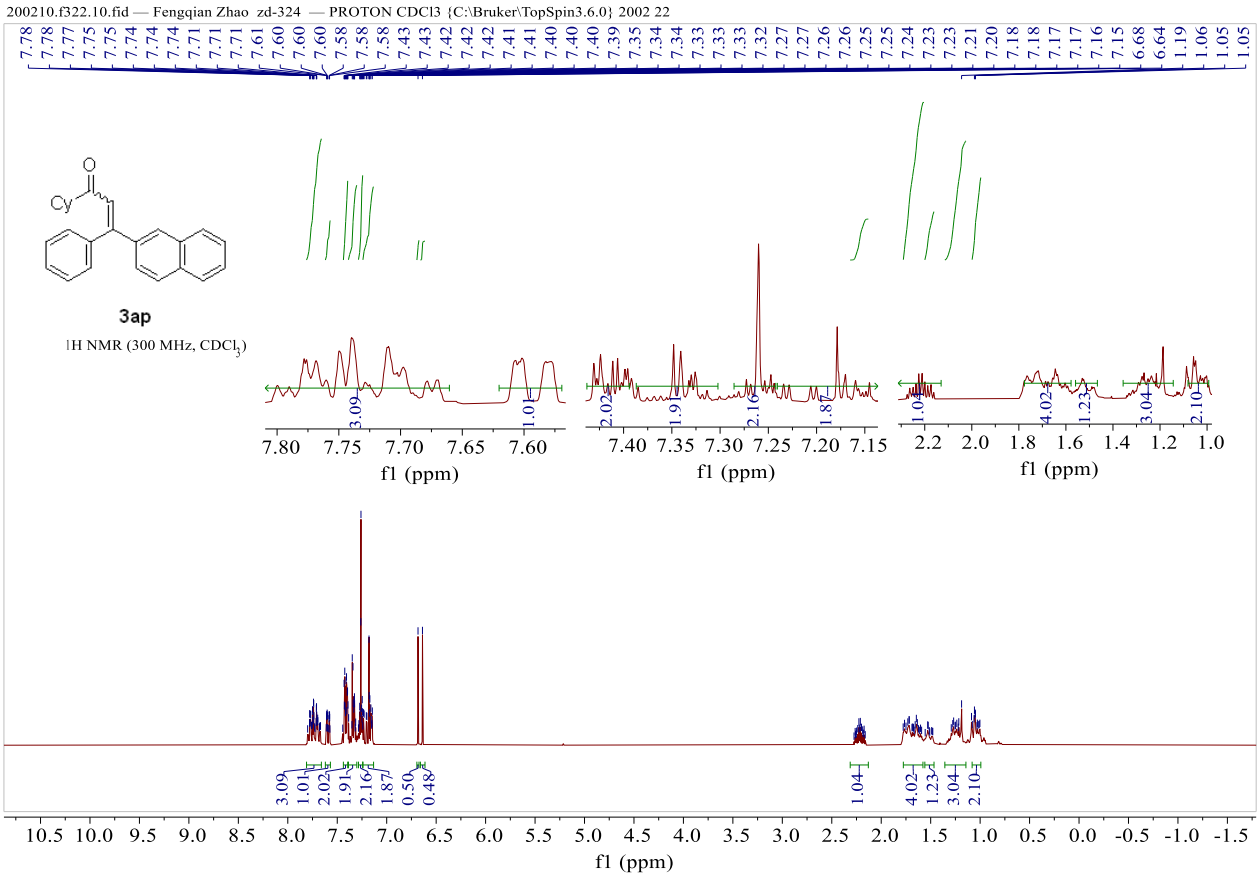
^{1}H NMR (300 MHz, $CDCl_3$)

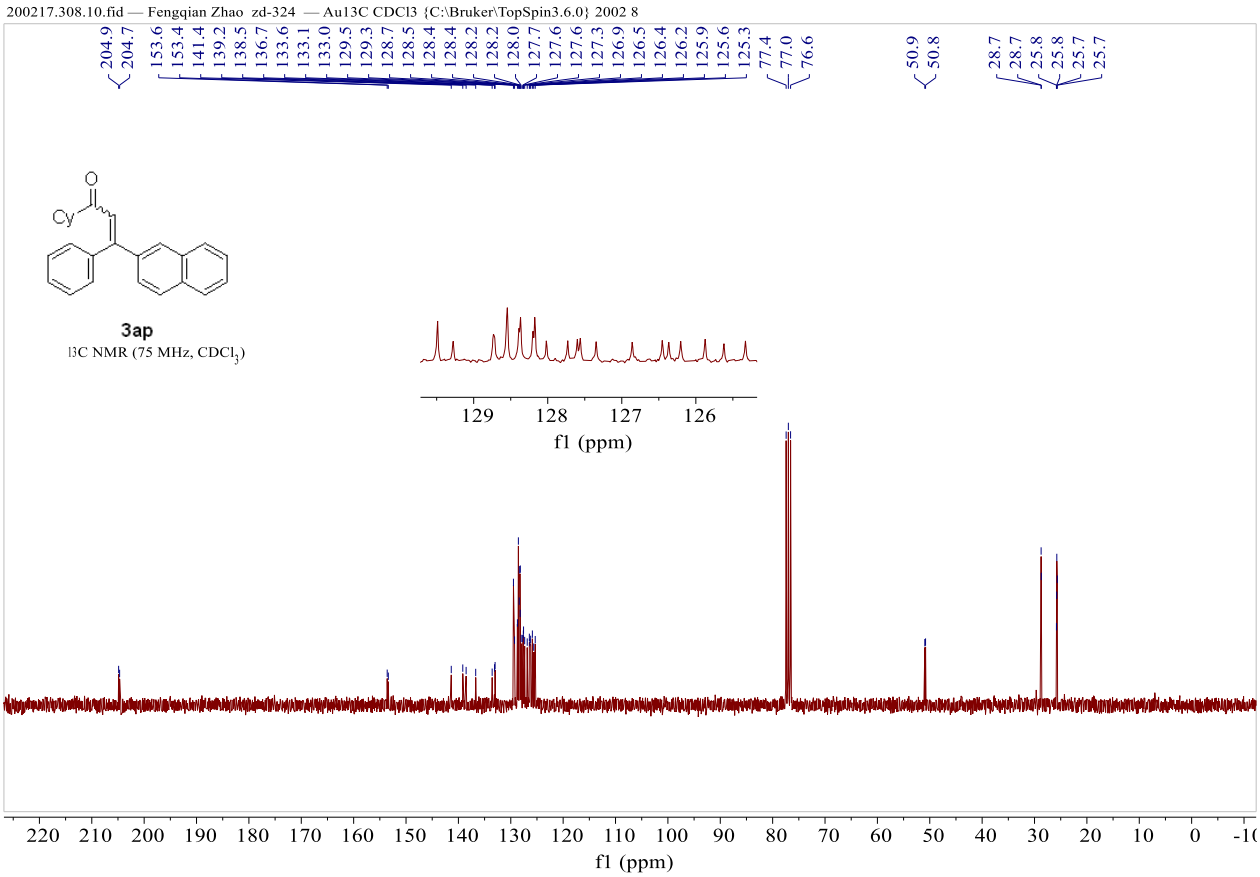
200313.f321.11.fid — Fengqian Zhao zd-377 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2003 21

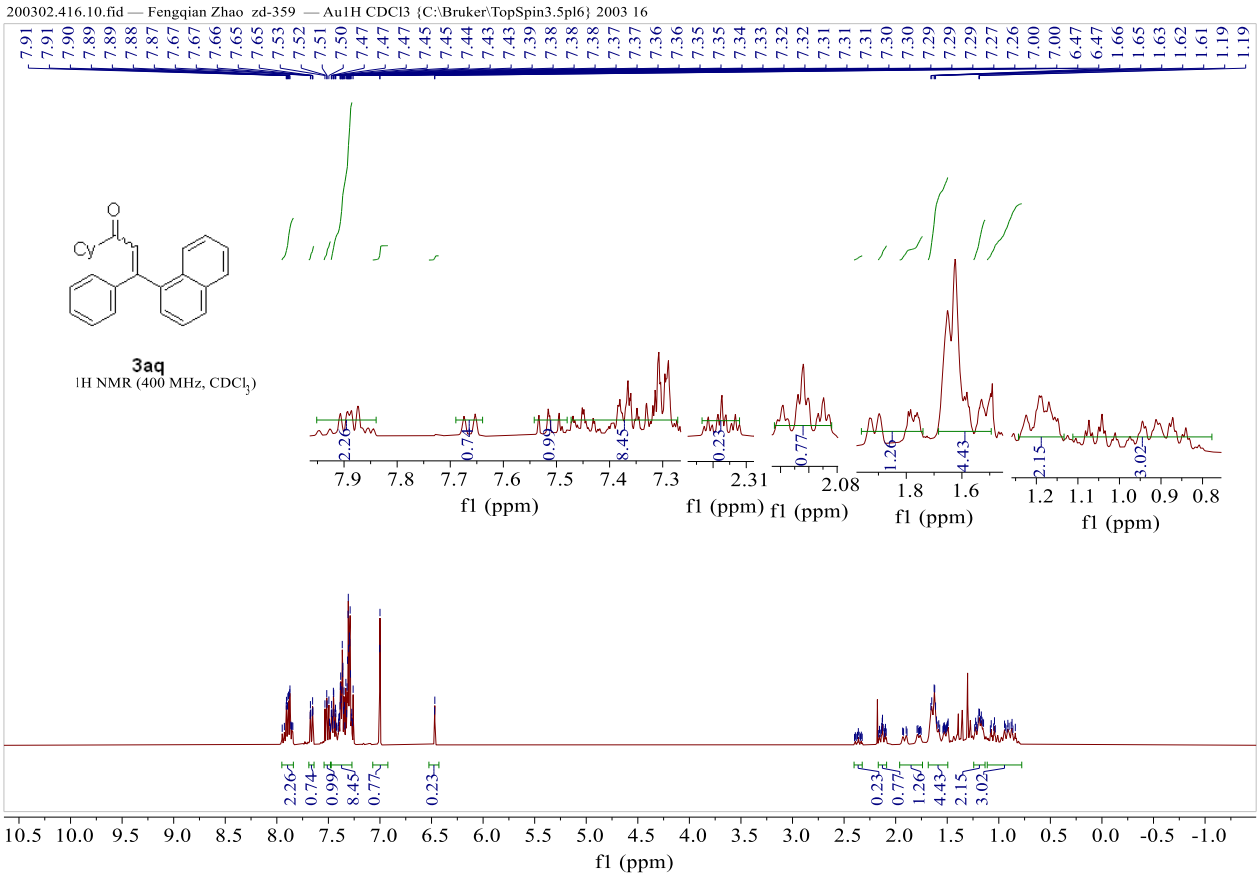


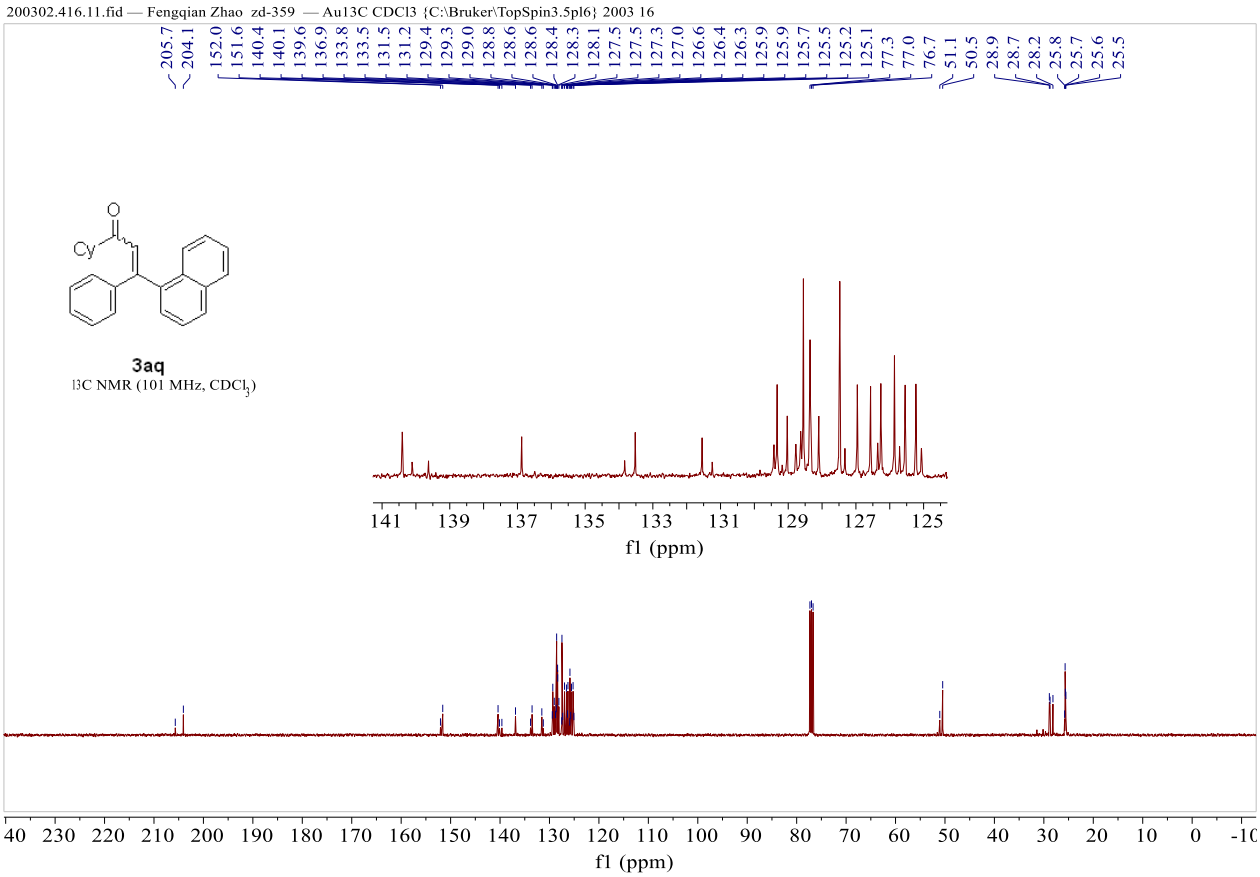
3ao

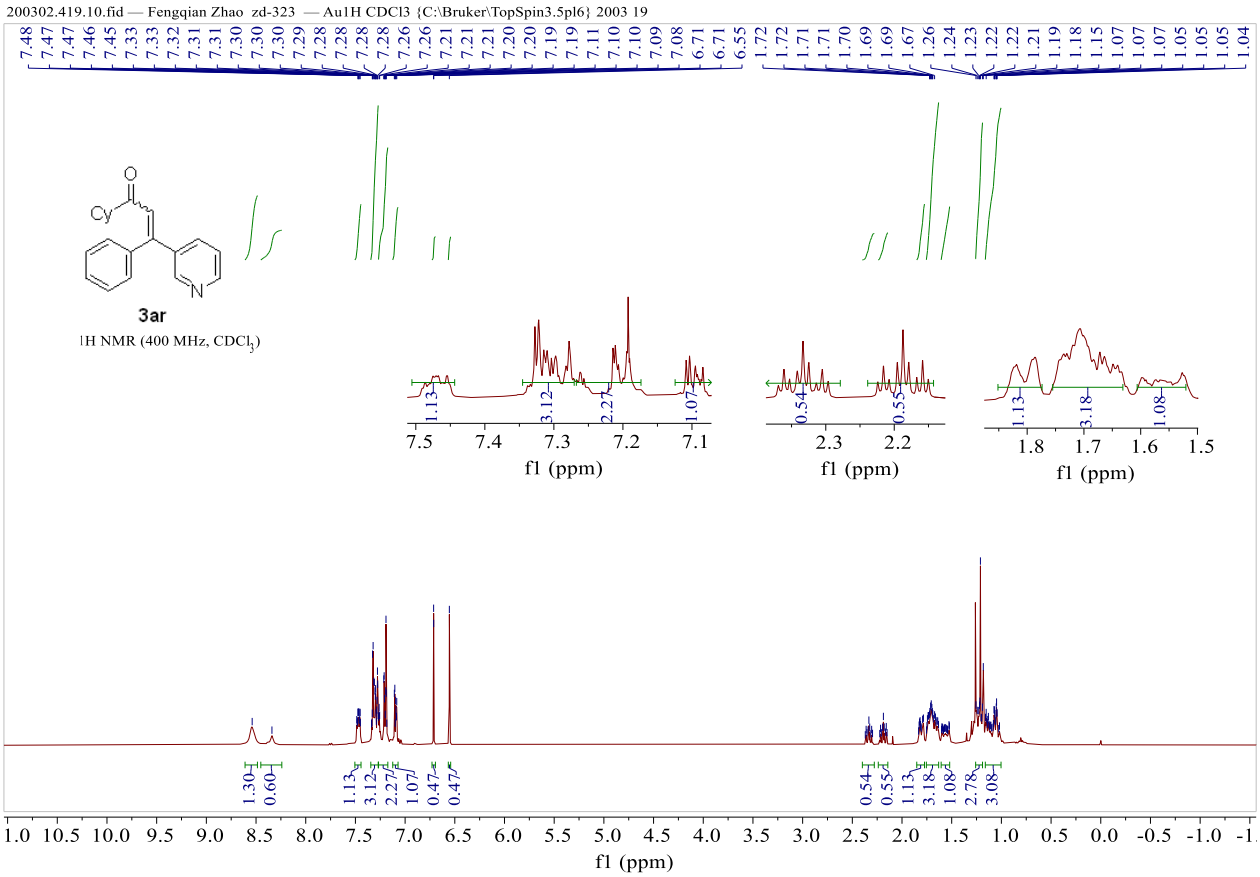
^{13}C NMR (75 MHz, $CDCl_3$)

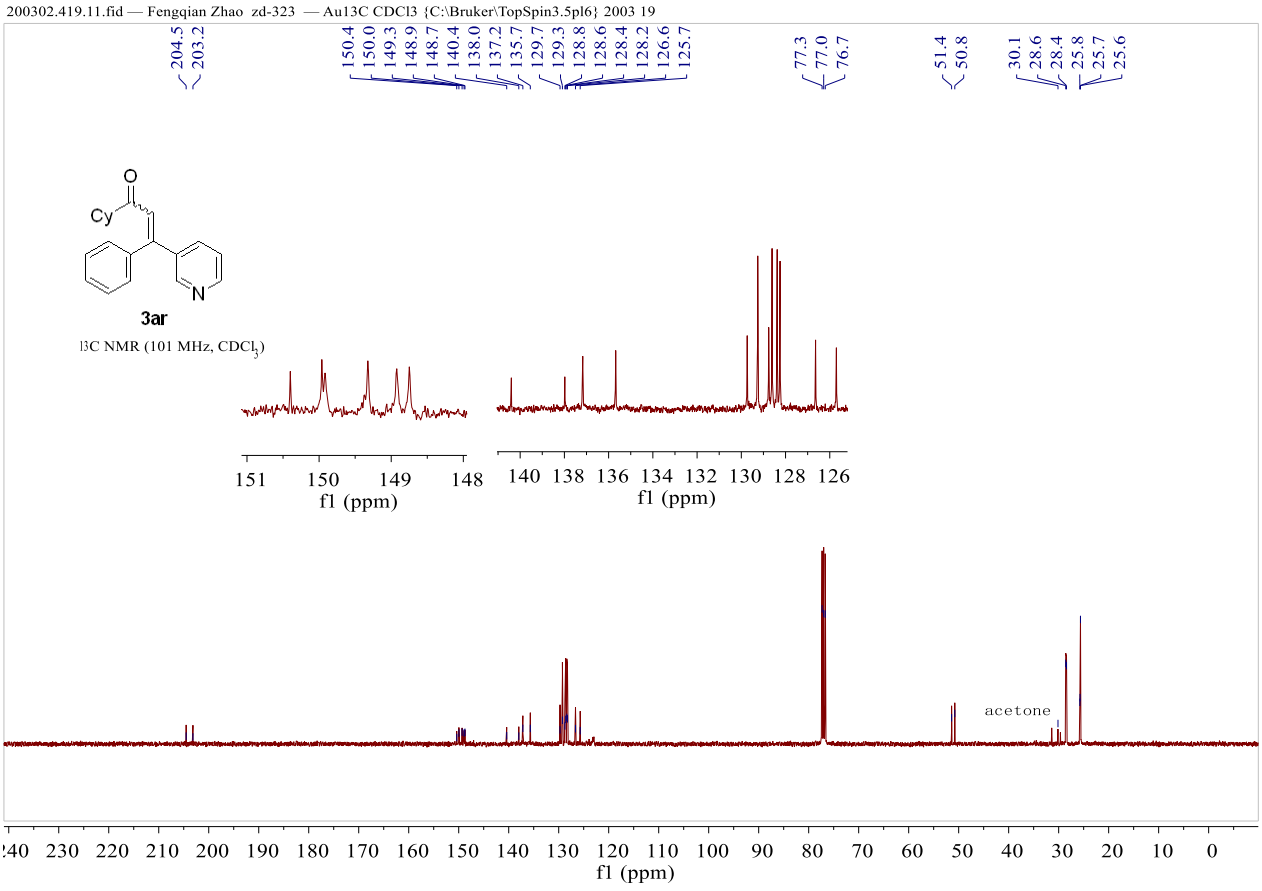

200210.f322.10.fid — Fengqian Zhao zd-324 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 22
Cy
O
3ap
1H NMR (300 MHz, CDCl3)
f1 (ppm)

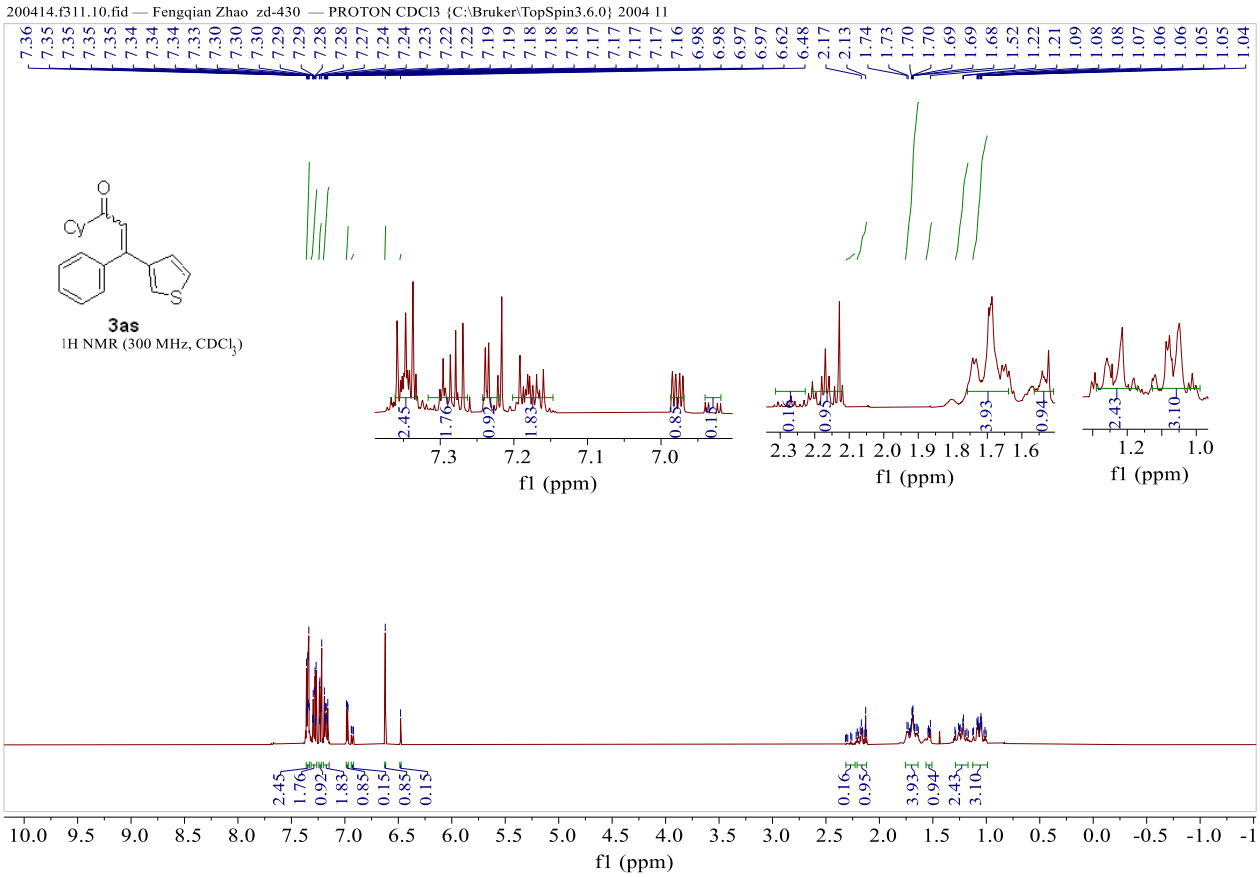

200217.308.10.fid — Fengqian Zhao zd-324 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 8
Cy
O
3ap
13C NMR (75 MHz, CDCl3)
f1 (ppm)

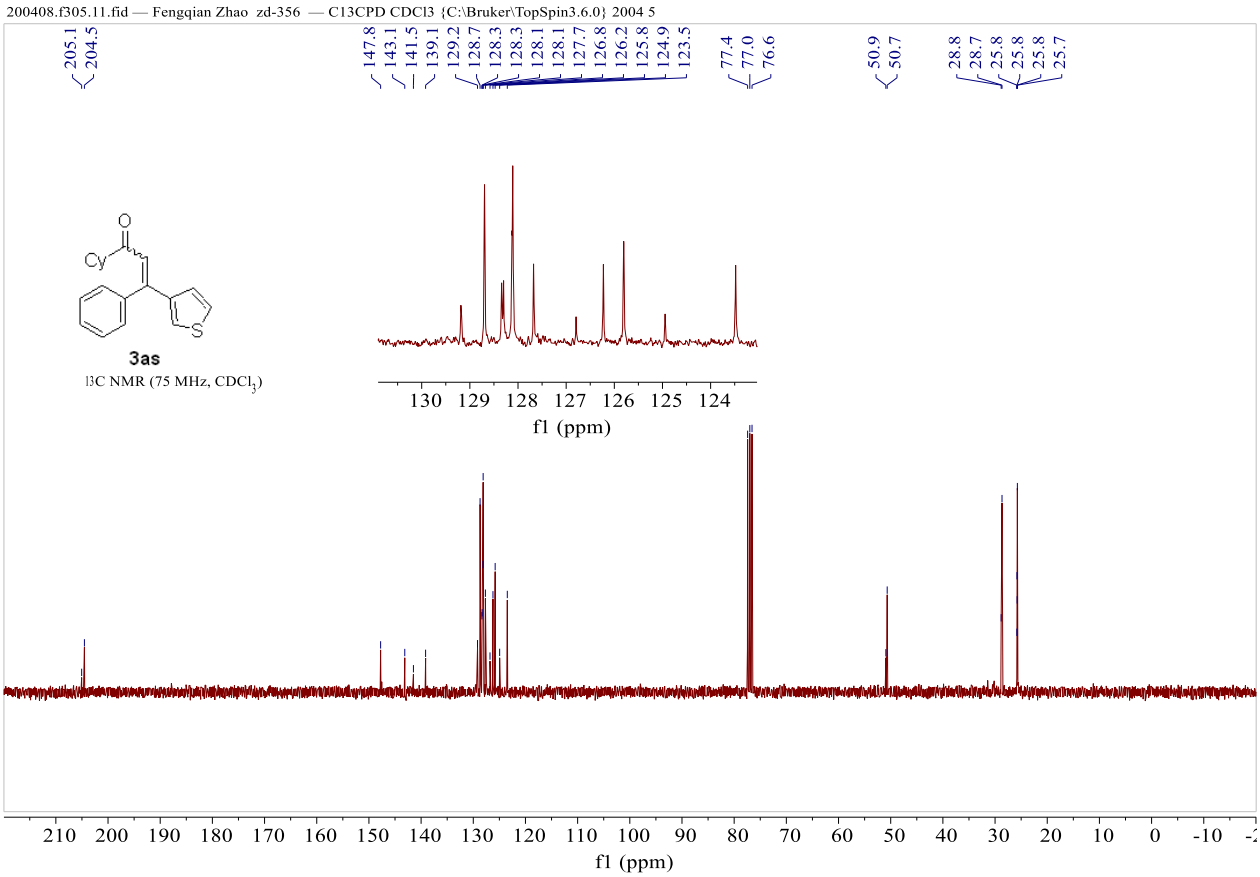

200302.416.10.fid — Fengqian Zhao zd-359 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 16
Cy
3aq
1H NMR (400 MHz, CDCl3)
f1 (ppm)

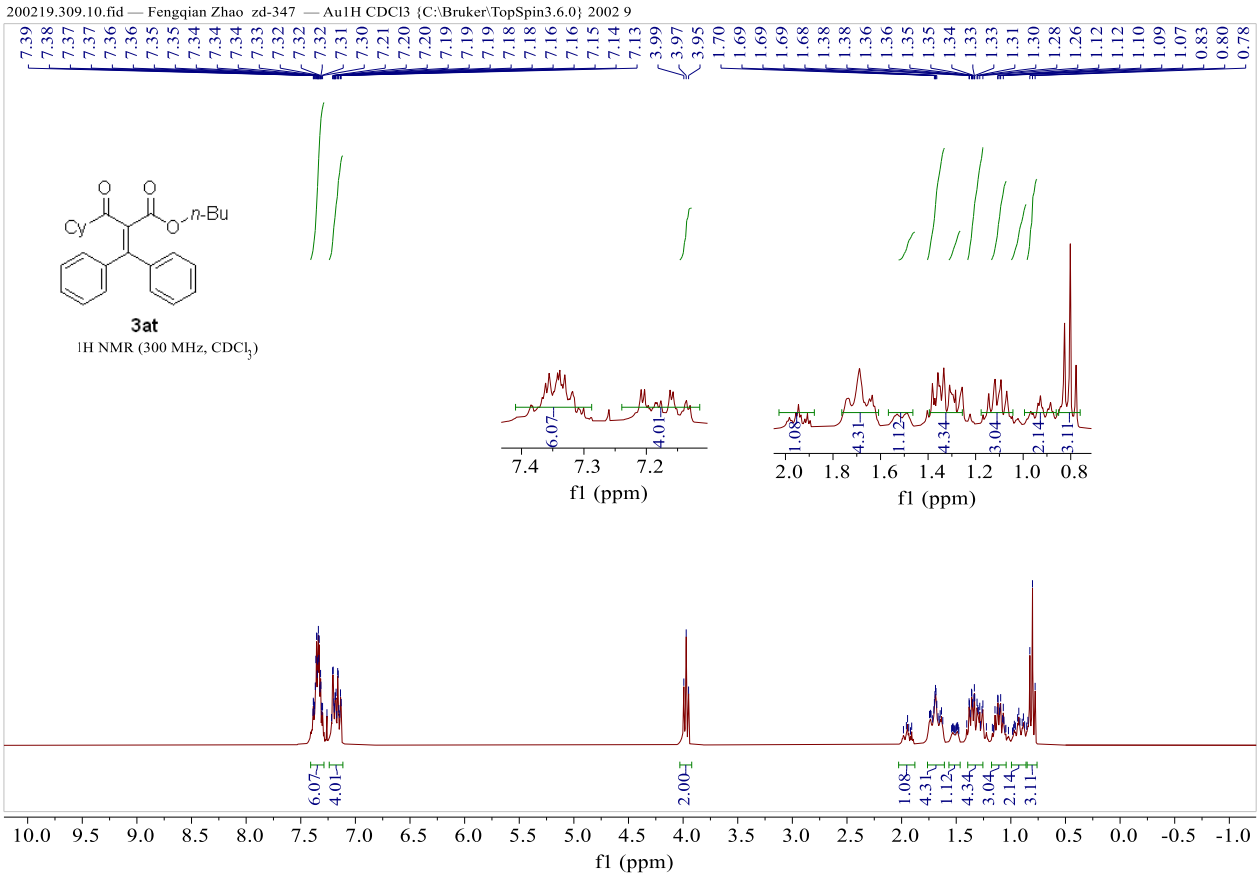

200302.416.11.fid — Fengqian Zhao zd-359 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 16
Cy
3aq
13C NMR (101 MHz, CDCl3)
f1 (ppm)

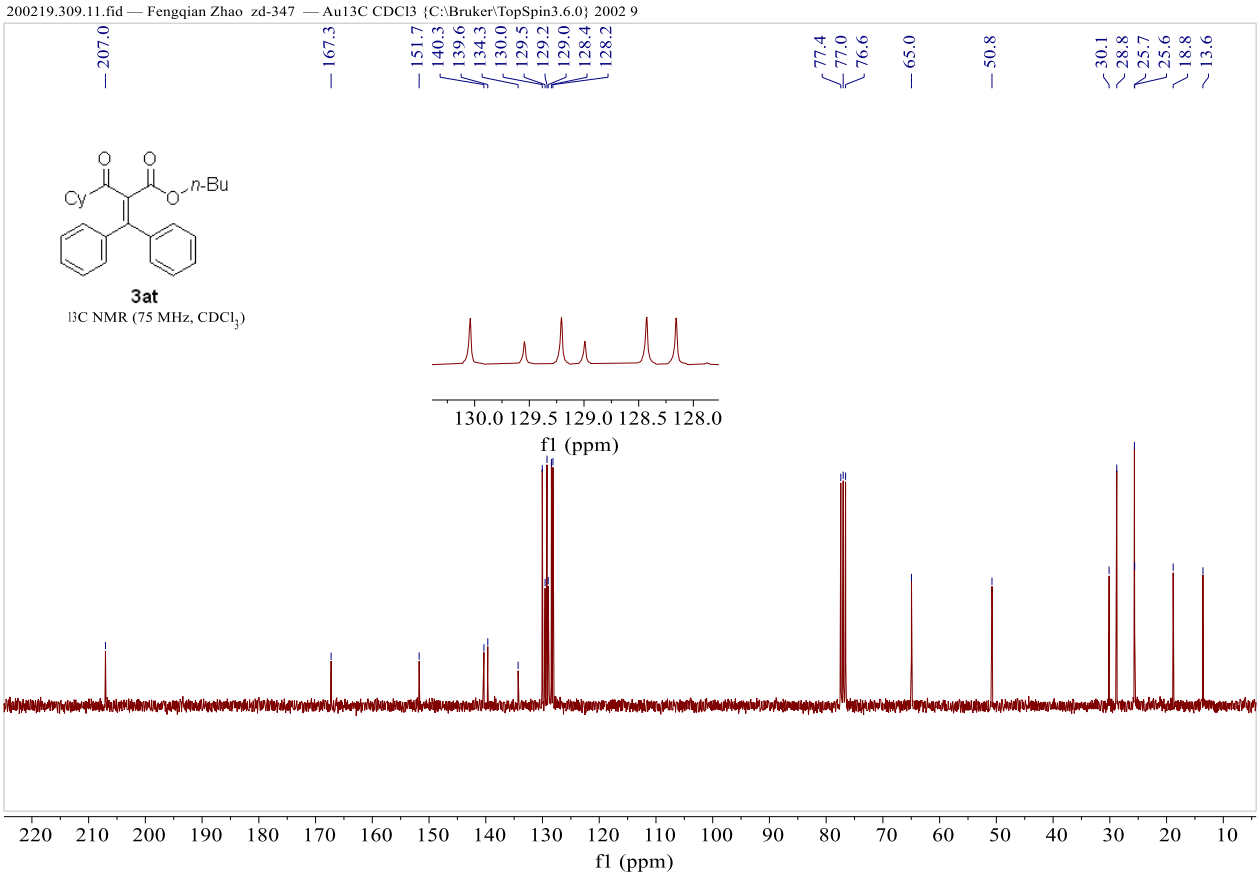

200302.419.10.fid — Fengqian Zhao zd-323 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 19
3ar
1H NMR (400 MHz, CDCl3)
f1 (ppm)

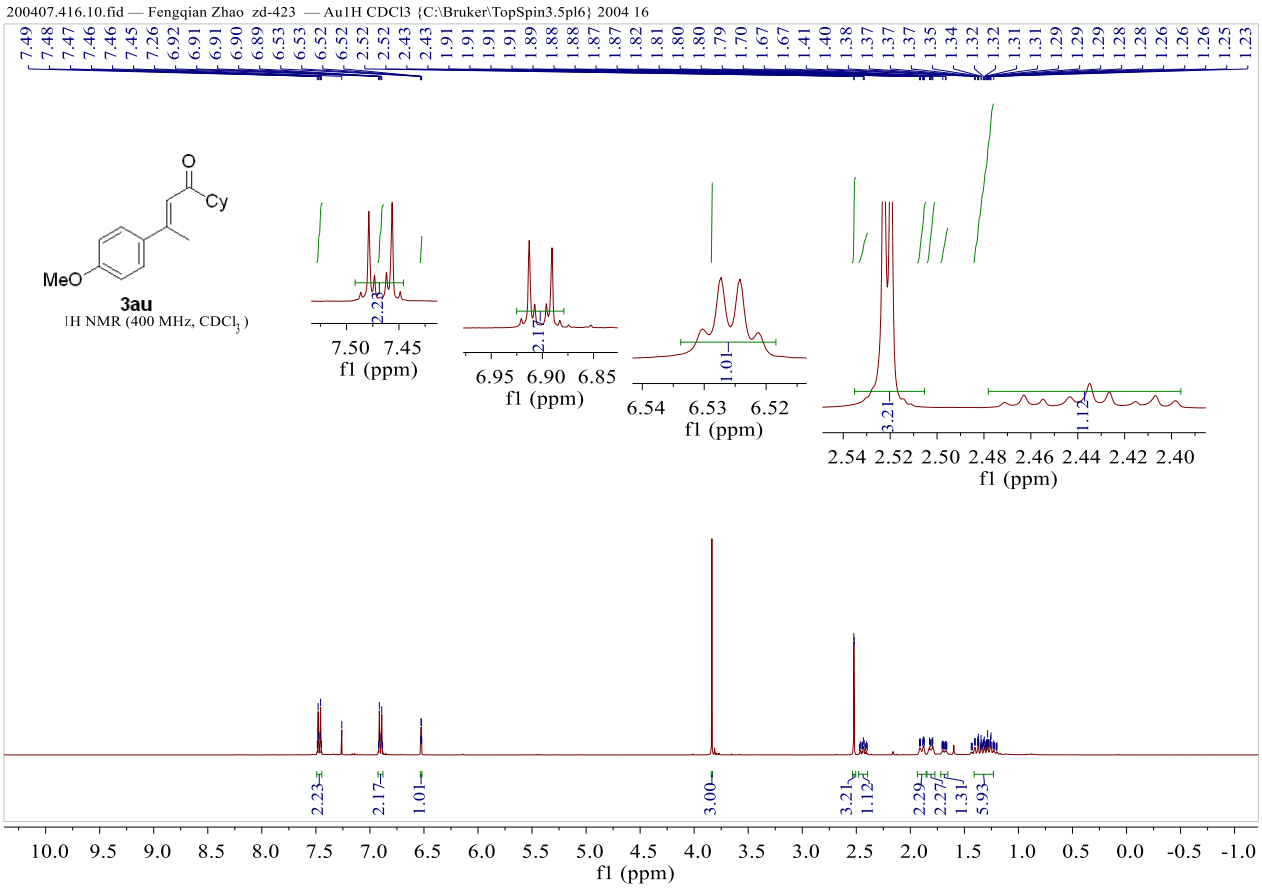

200302.419.11.fid — Fengqian Zhao zd-323 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 19
3ar
13C NMR (101 MHz, CDCl3)
acetone
f1 (ppm)

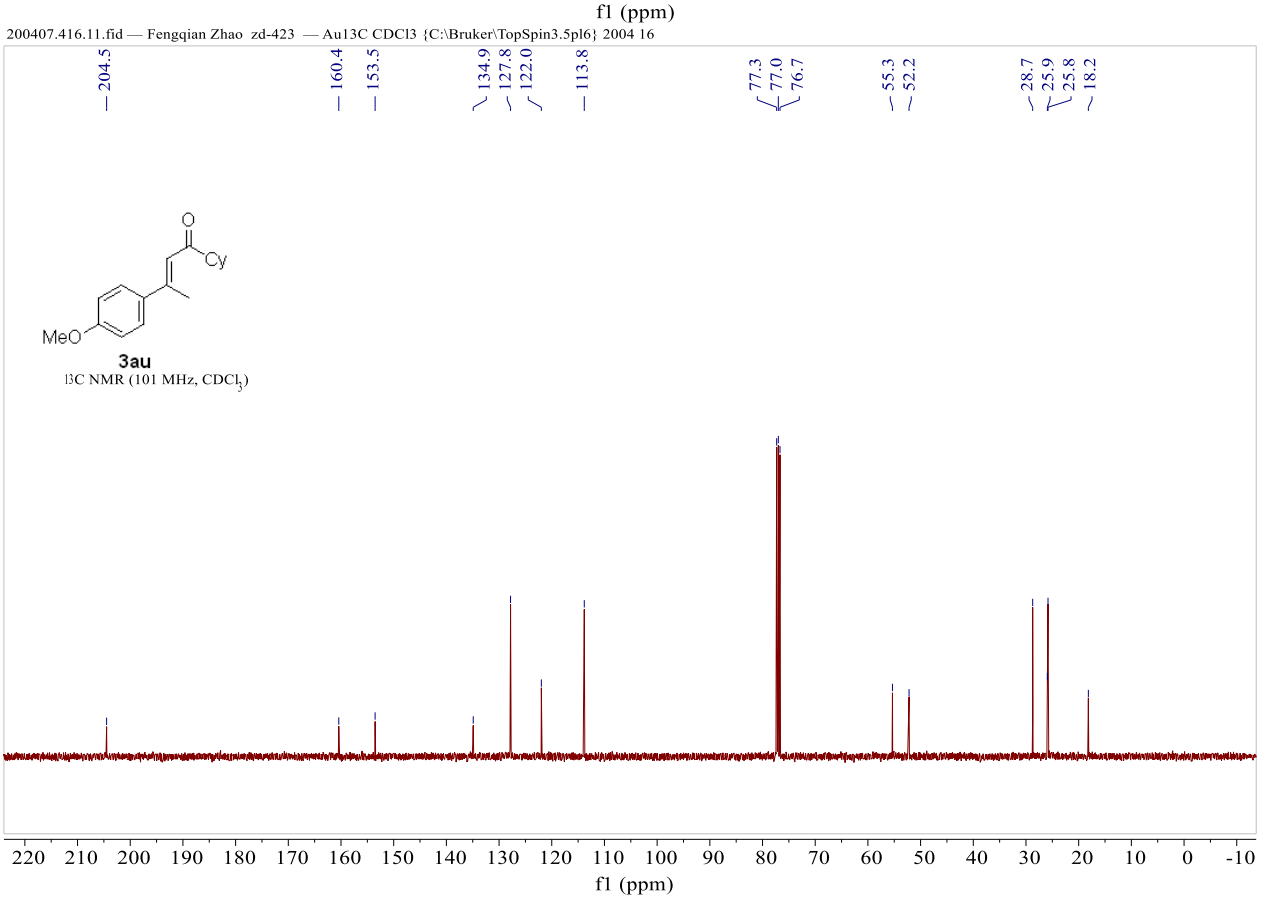

200414.f311.10.fid — Fengqian Zhao zd-430 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2004 11
3as
1H NMR (300 MHz, CDCl3)
f1 (ppm)

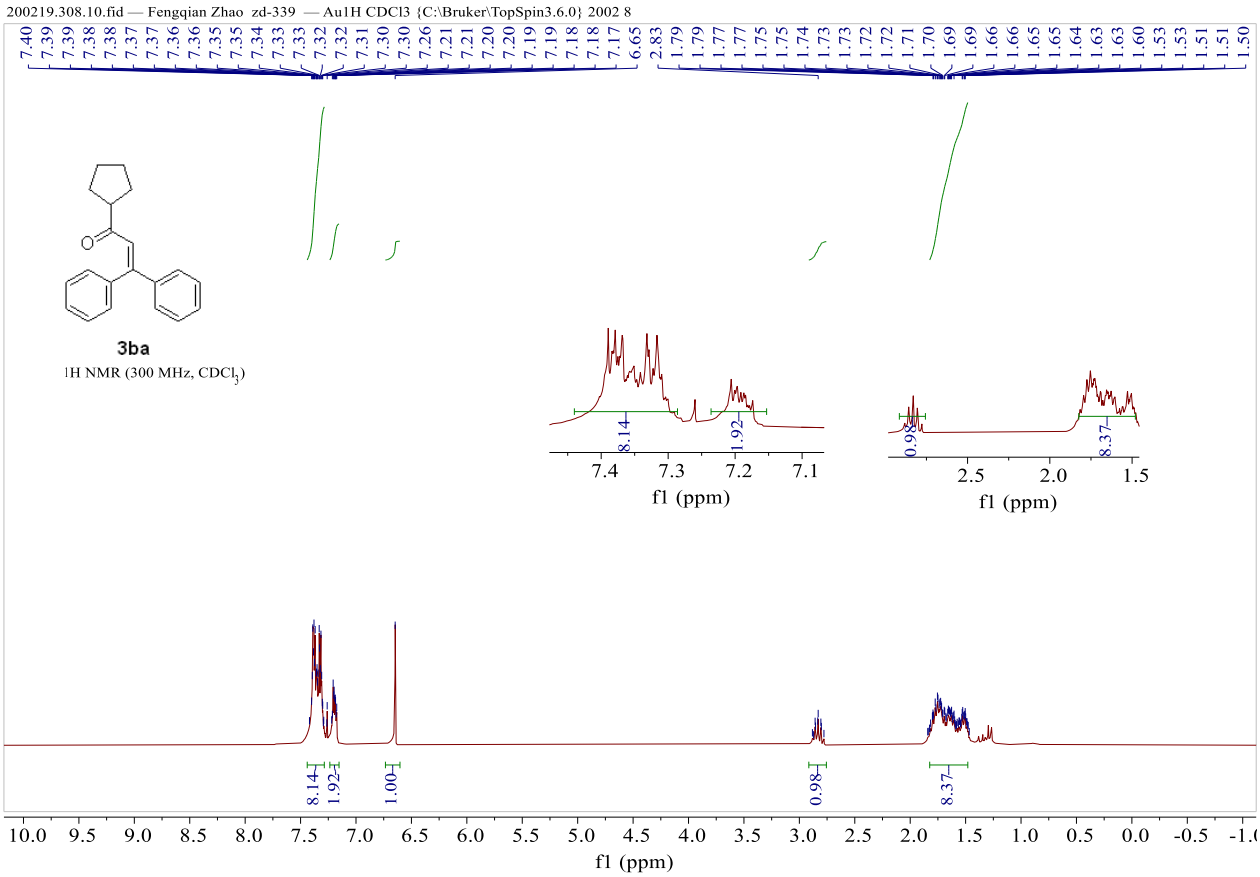

200408.f305.11.fid — Fengqian Zhao zd-356 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2004 5
3as
13C NMR (75 MHz, CDCl3)
f1 (ppm)

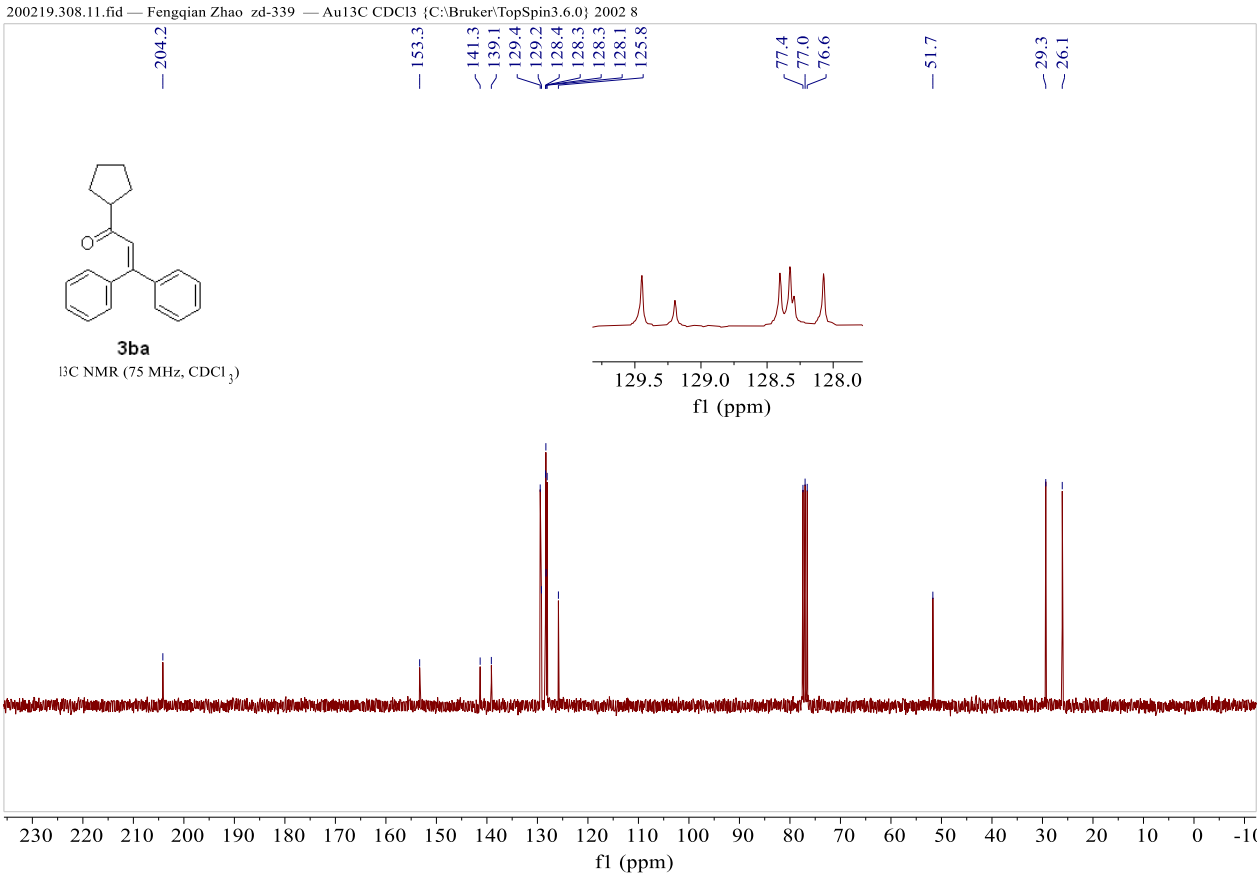

200219.309.10.fid — Fengqian Zhao zd-347 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 9
Cy
n-Bu
3at
1H NMR (300 MHz, CDCl3)
f1 (ppm)


200219.309.11.fid — Fengqian Zhao zd-347 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 9
Cy
n-Bu
3at
13C NMR (75 MHz, CDCl3)
f1 (ppm)

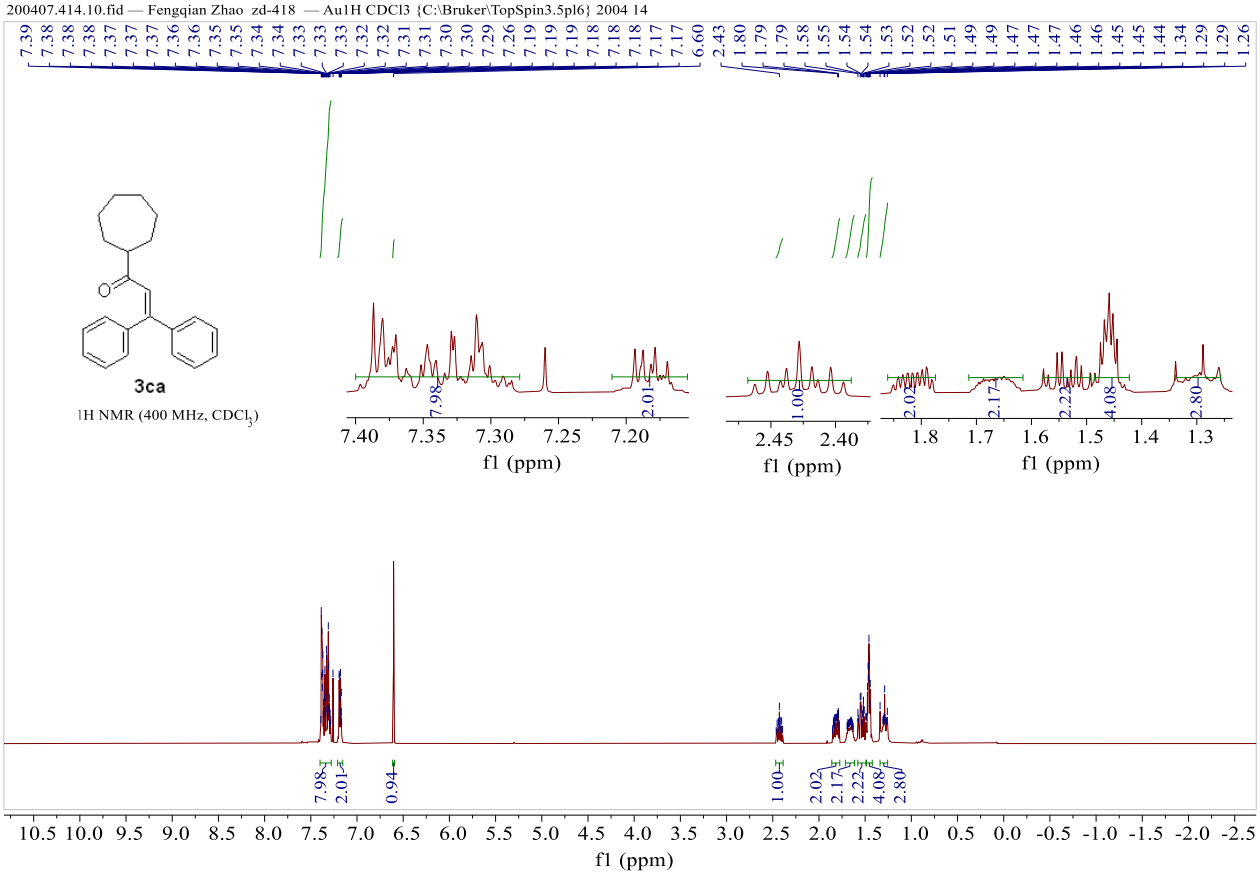

200407.416.10.fid — Fengqian Zhao zd-423 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2004 16
O
Cy
MeO
3au
1H NMR (400 MHz, CDCl3)
f1 (ppm)


200407.416.11.fid — Fengqian Zhao zd-423 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2004 16
O
Cy
MeO
3au
13C NMR (101 MHz, CDCl3)
f1 (ppm)

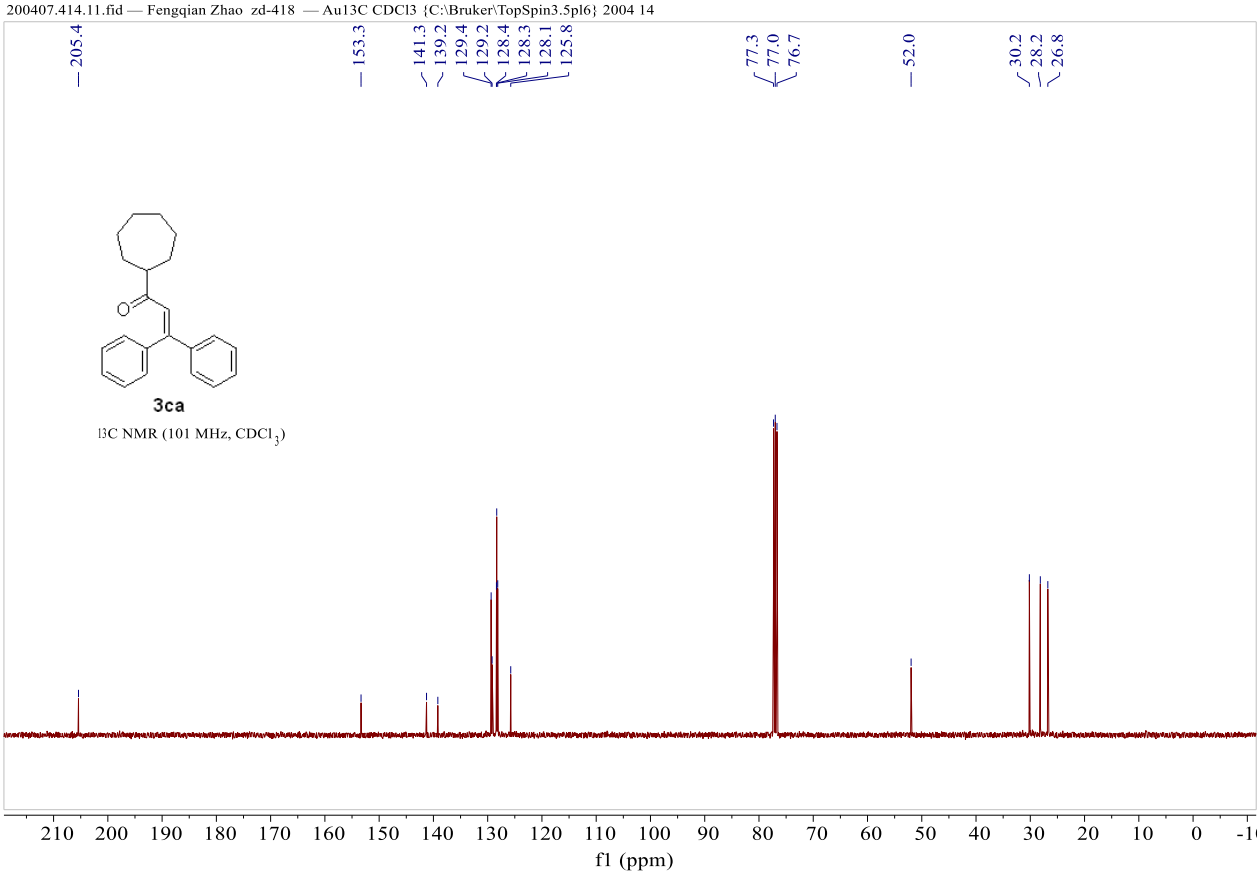

200219.308.10.fid — Fengqian Zhao zd-339 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 8
3ba
1H NMR (300 MHz, CDCl3)
f1 (ppm)

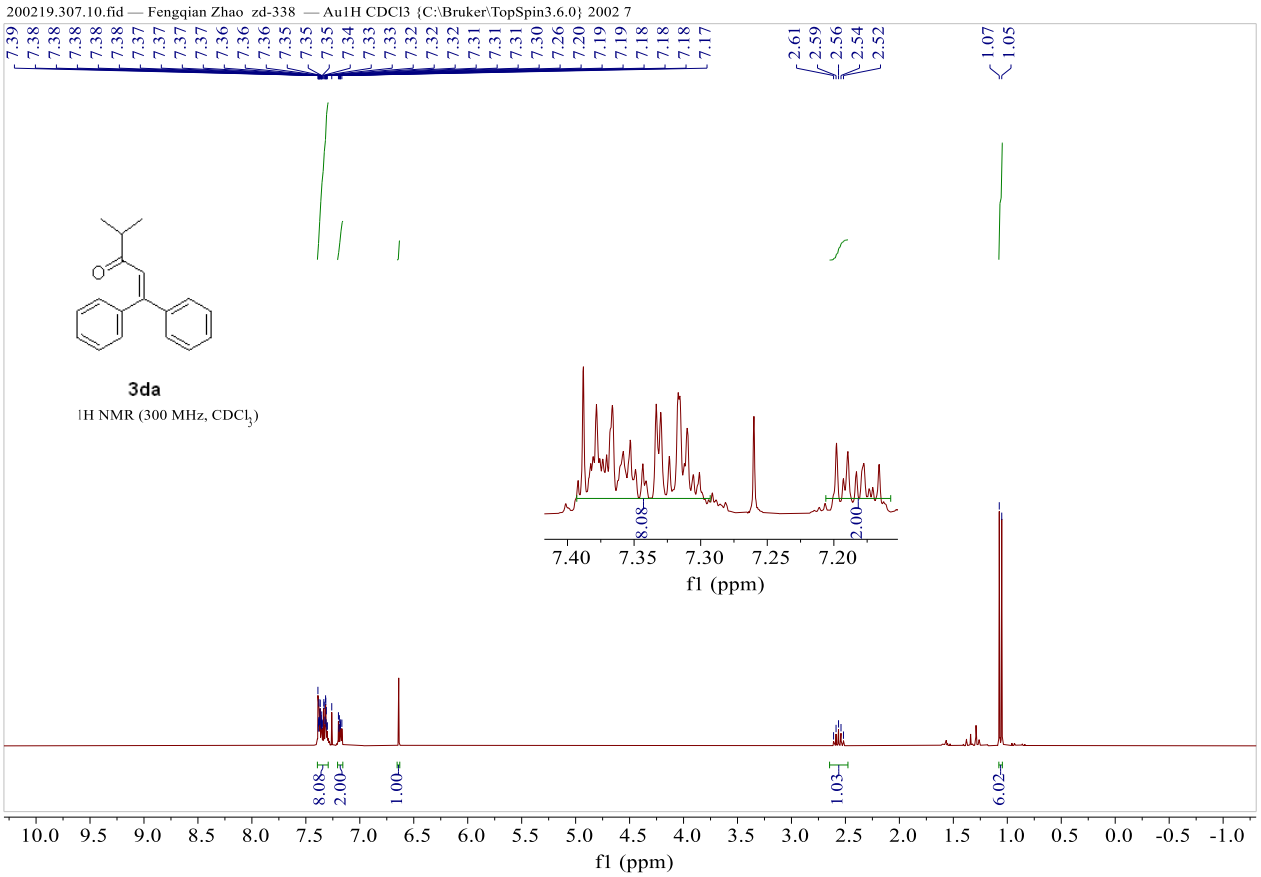

200219.308.11.fid — Fengqian Zhao zd-339 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 8
3ba
13C NMR (75 MHz, CDCl3)
f1 (ppm)

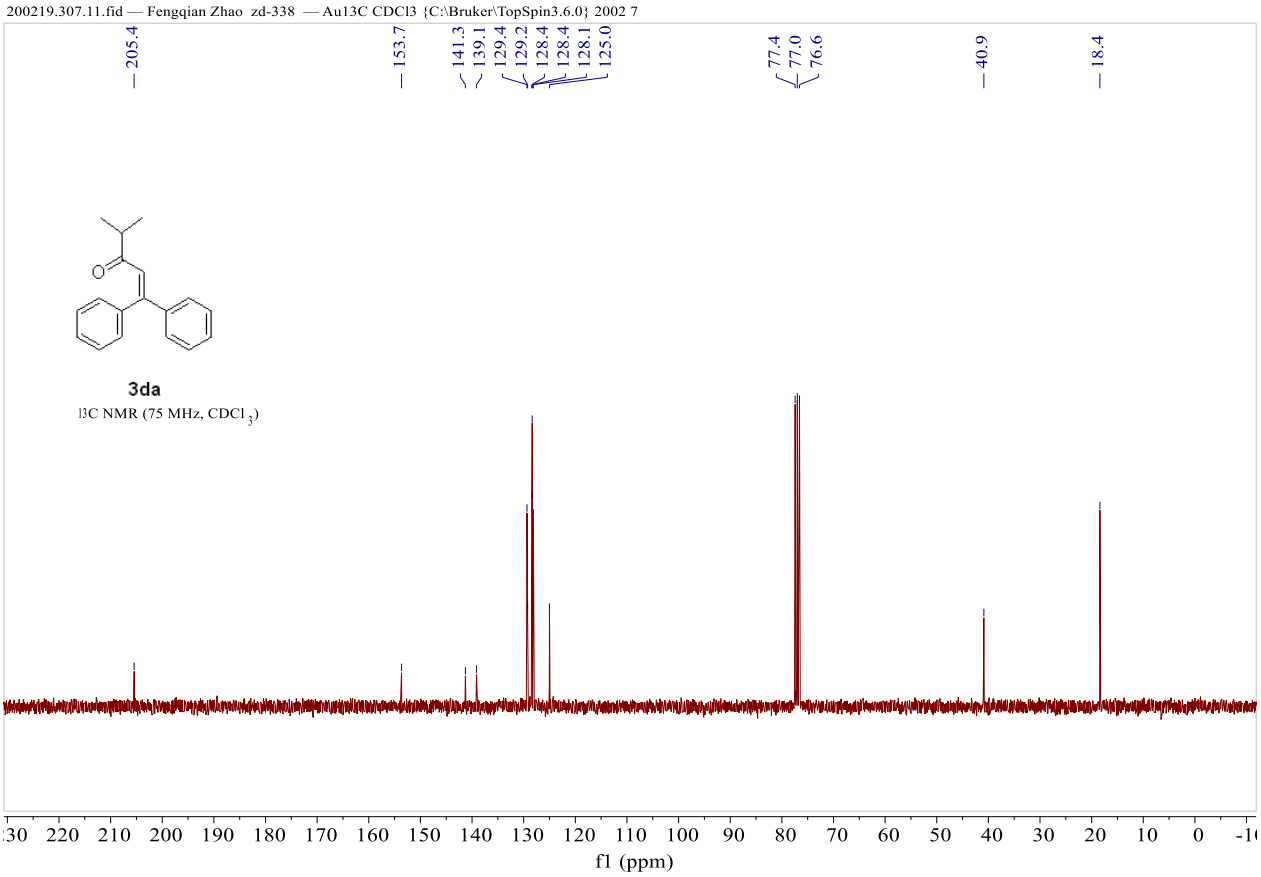
200407.414.10.fid — Fengqian Zhao zd-418 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2004 14

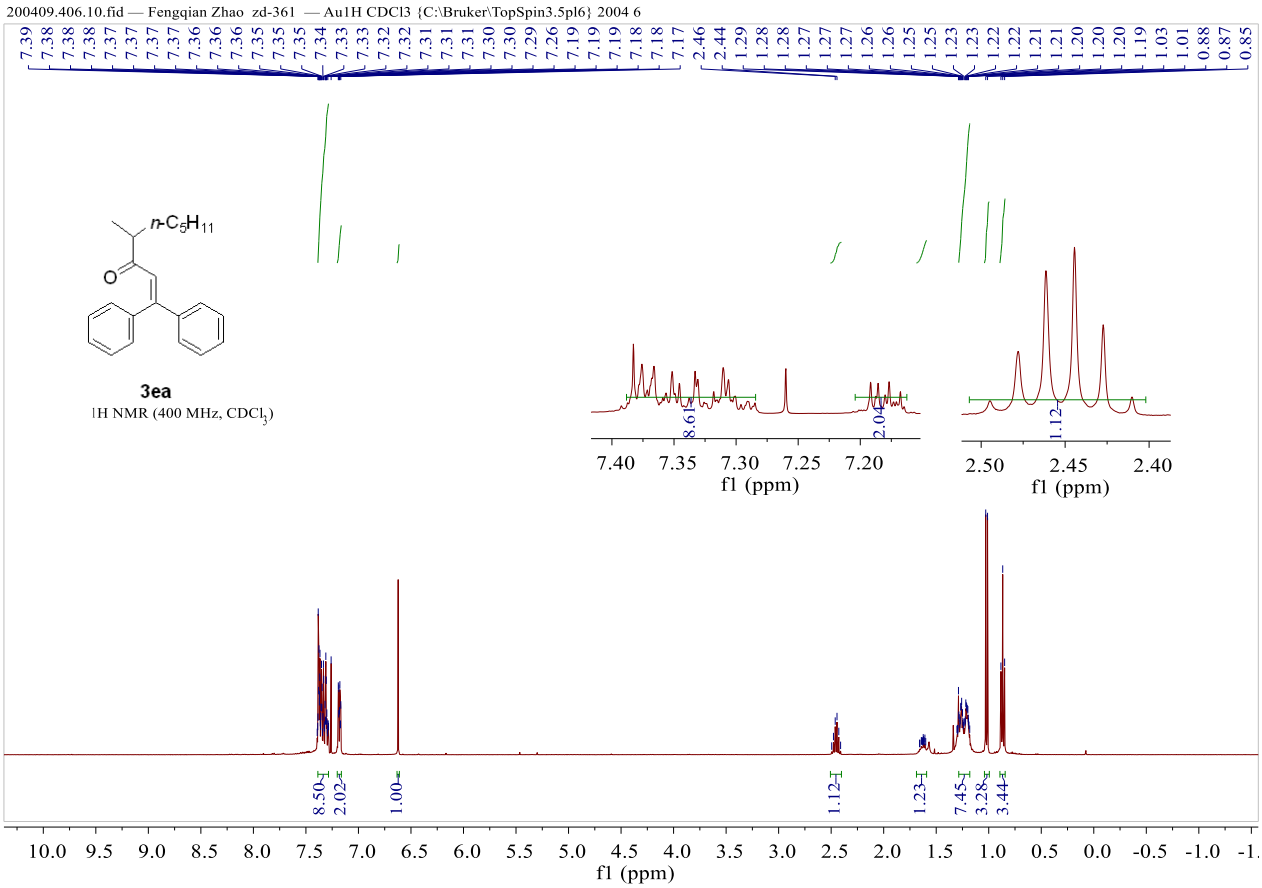


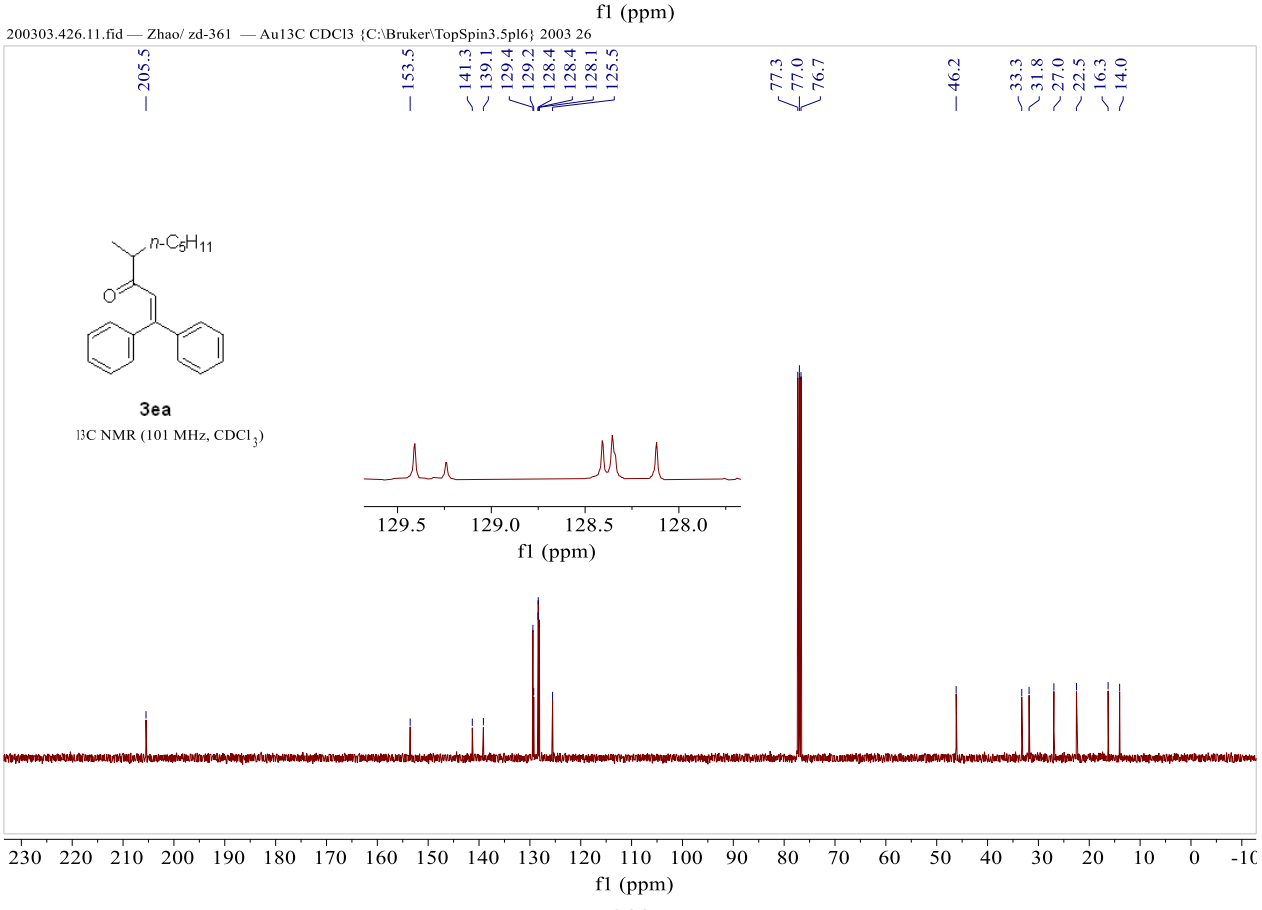
200407.414.11.fid — Fengqian Zhao zd-418 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2004 14

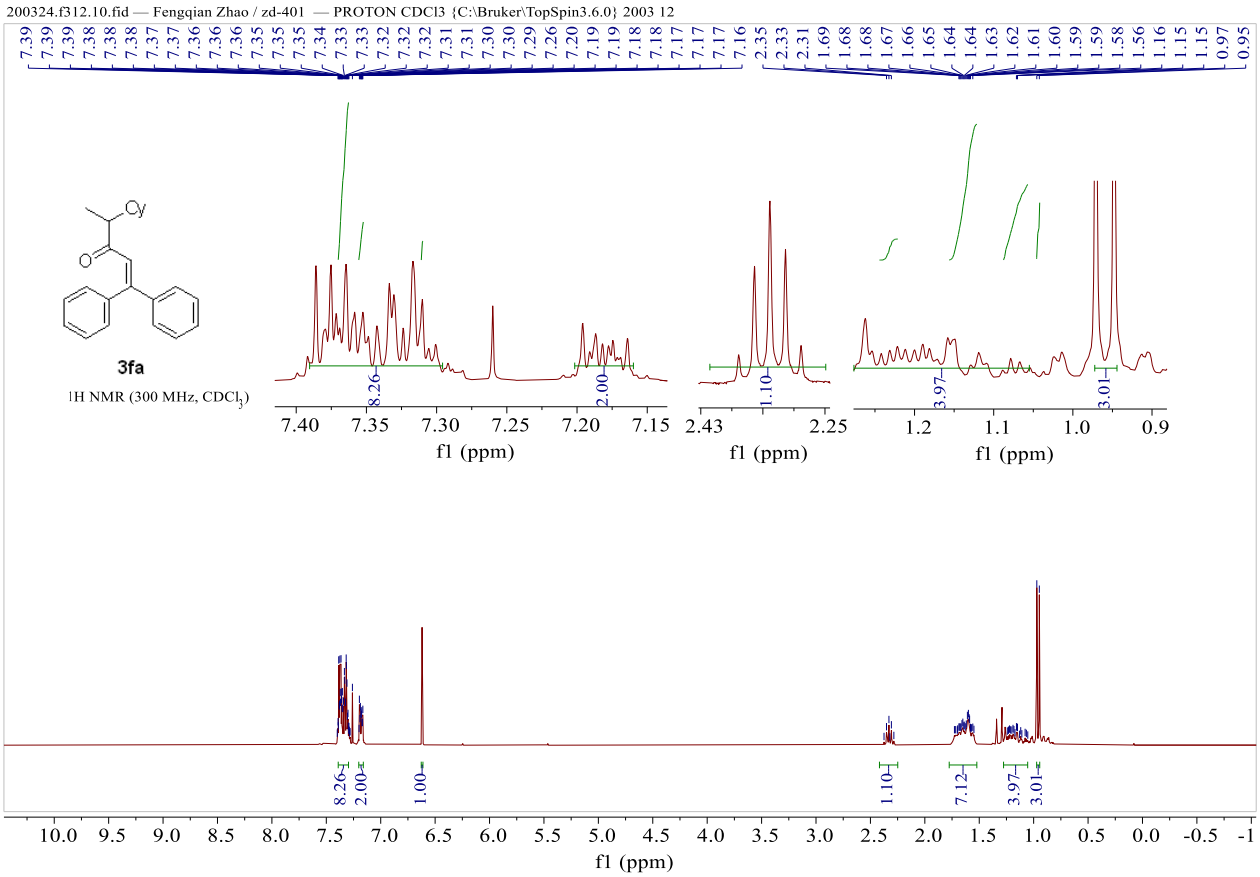


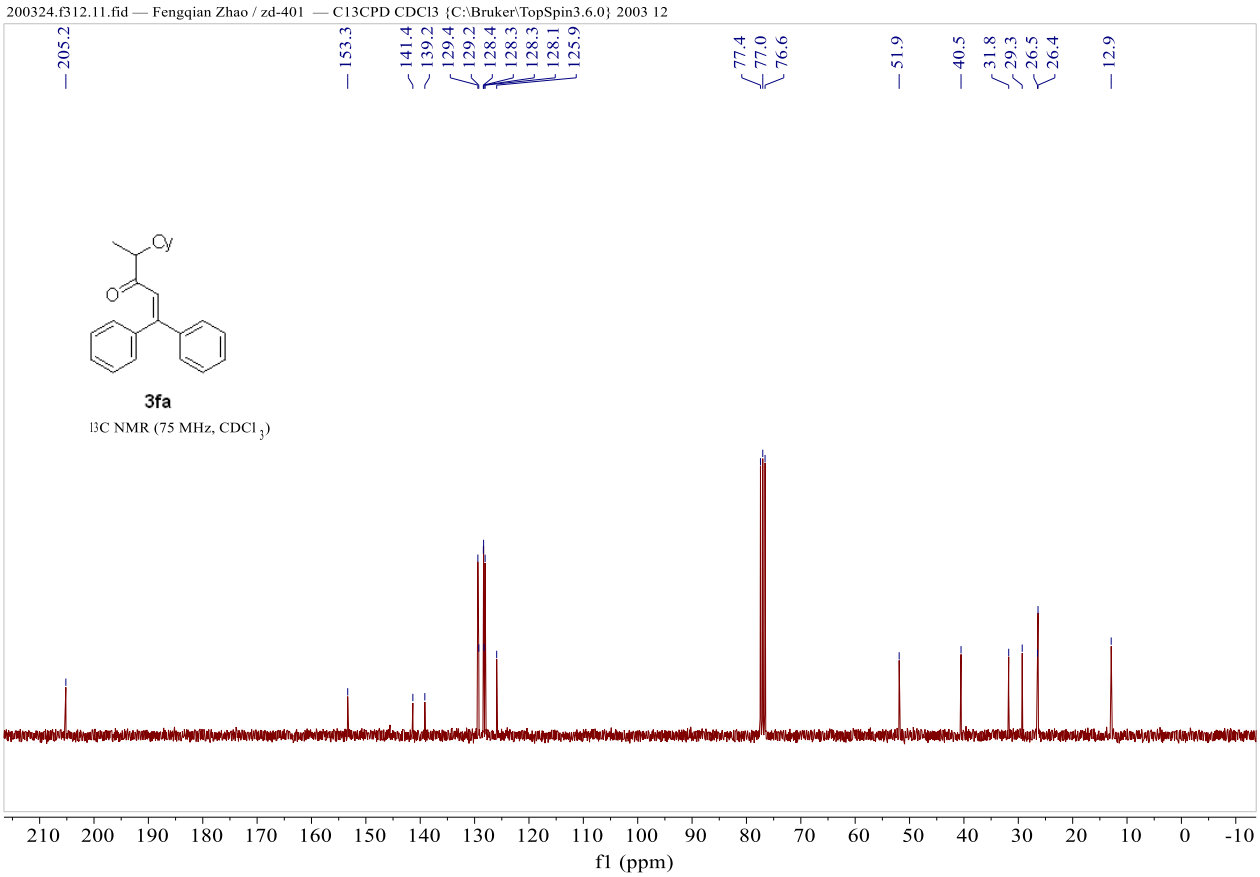

200219.307.10.fid — Fengqian Zhao zd-338 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 7
3da
1H NMR (300 MHz, CDCl3)
f1 (ppm)

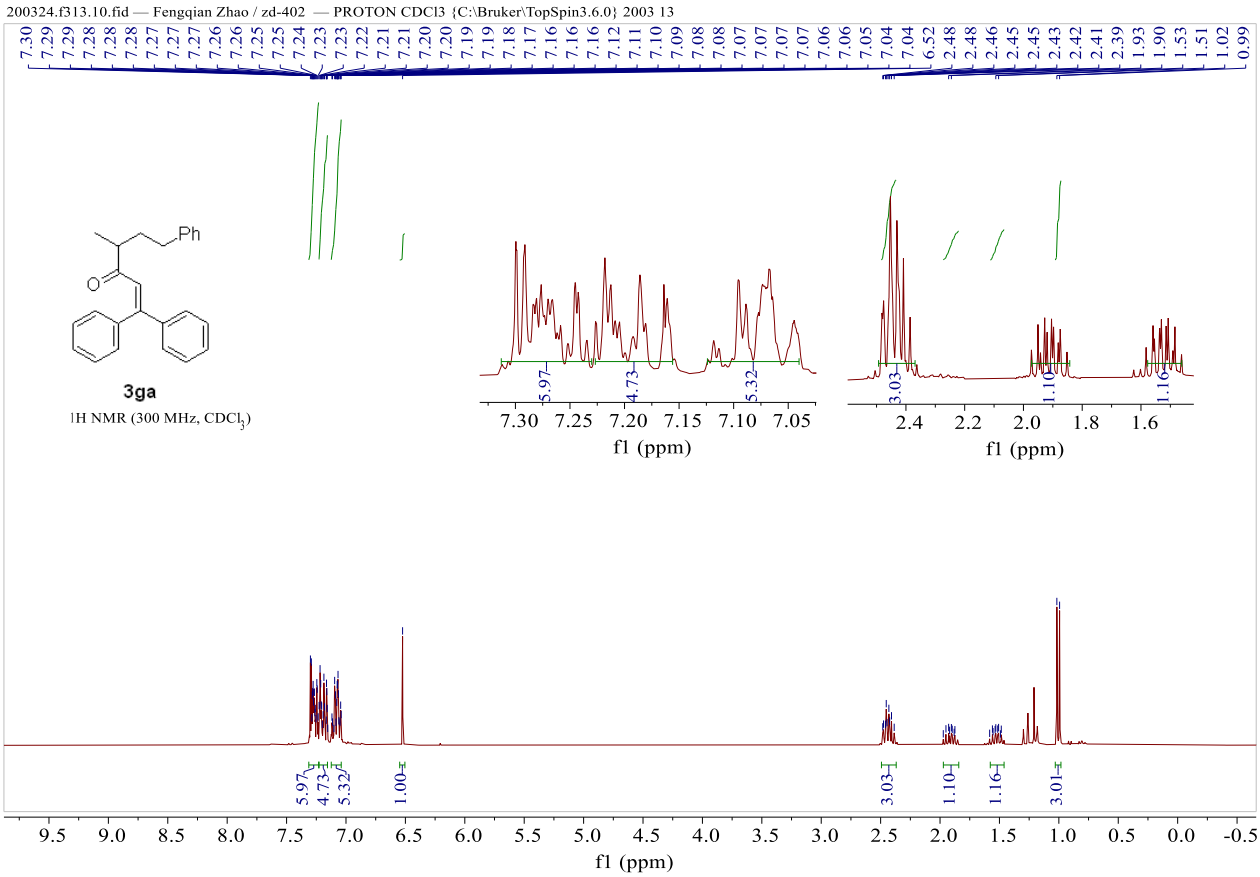

200219.307.11.fid — Fengqian Zhao zd-338 — Au13C CDCl3 {C:\Bruker\TopSpin3.6.0} 2002 7
3da
13C NMR (75 MHz, CDCl3)
f1 (ppm)

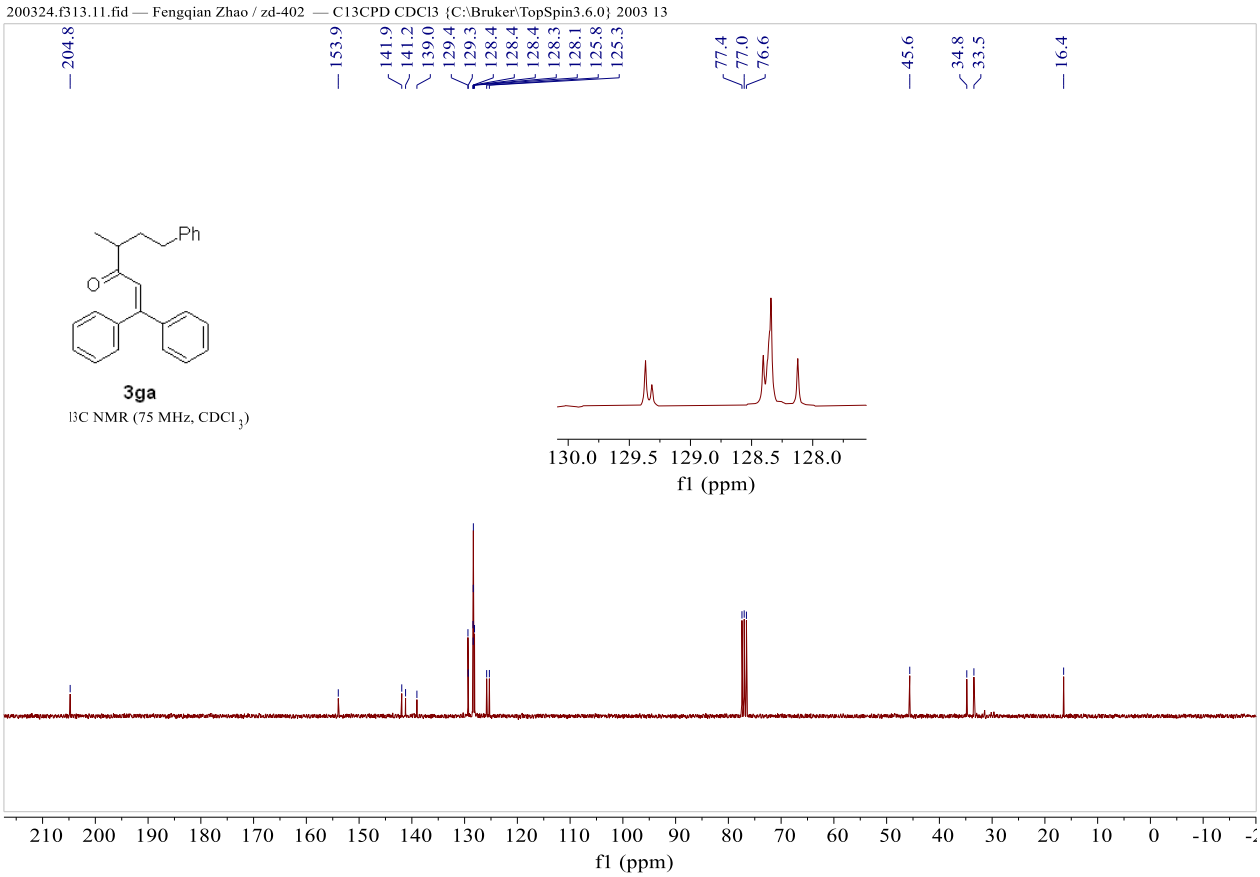

200409.406.10.fid — Fengqian Zhao zd-361 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2004 6
n-C5H11
O
3ea
1H NMR (400 MHz, CDCl3)
f1 (ppm)

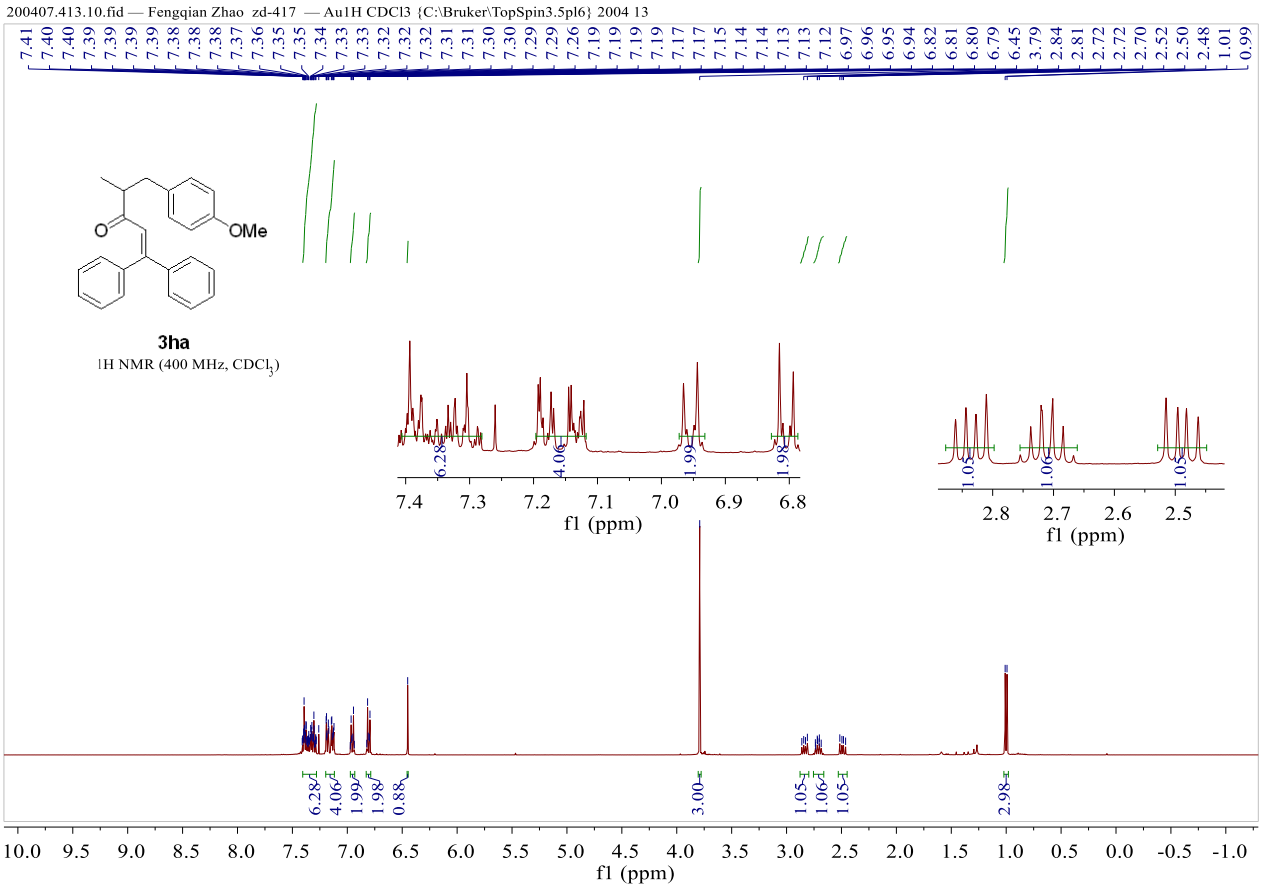

200303.426.11.fid — Zhao/ zd-361 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2003 26
n-C5H11
O
3ea
13C NMR (101 MHz, CDCl3)
f1 (ppm)

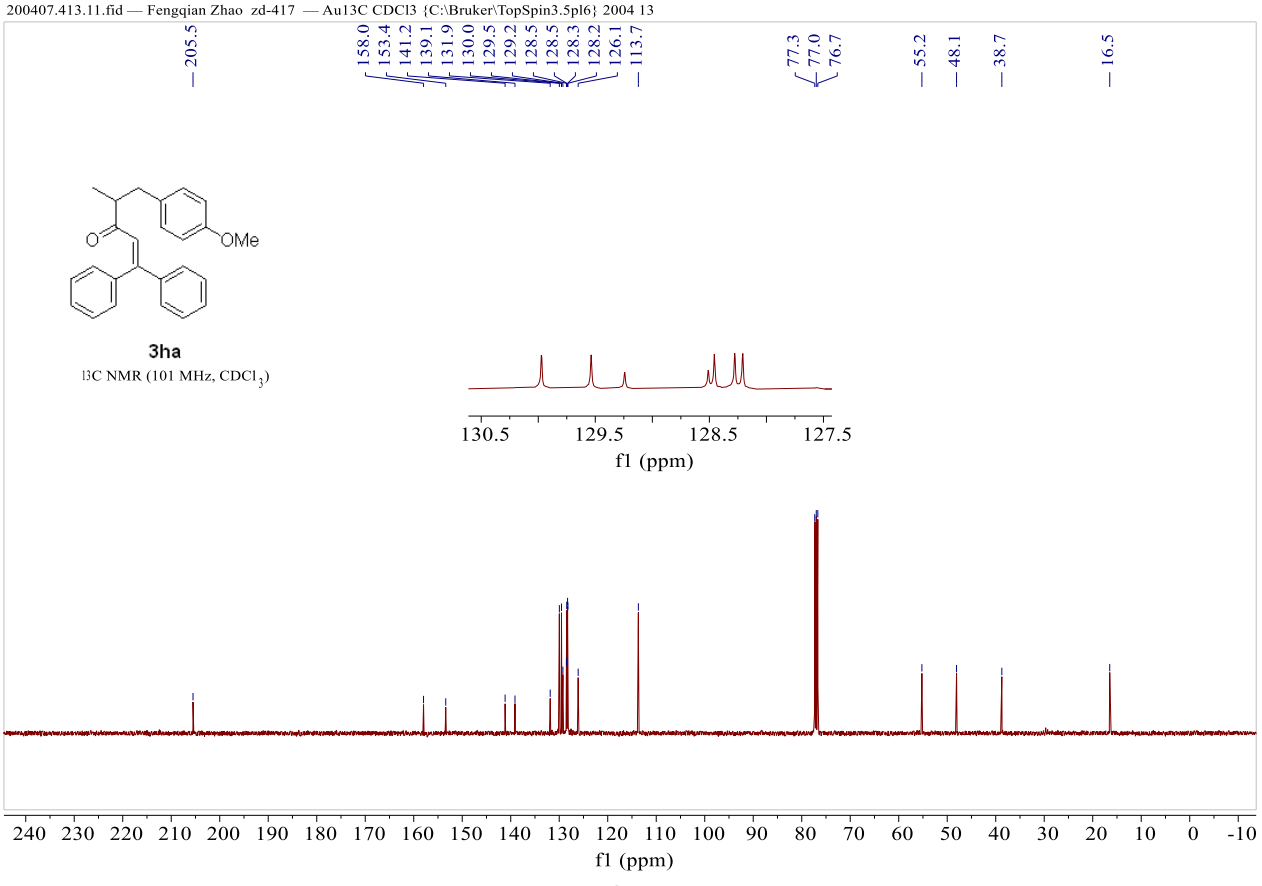

200324.f312.10.fid — Fengqian Zhao / zd-401 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2003 12
Cy
O
3fa
1H NMR (300 MHz, CDCl3)
8.26
2.00
1.10
3.97
3.01
f1 (ppm)
8.26
2.00
1.00
1.10
7.12
3.97
3.01
f1 (ppm)

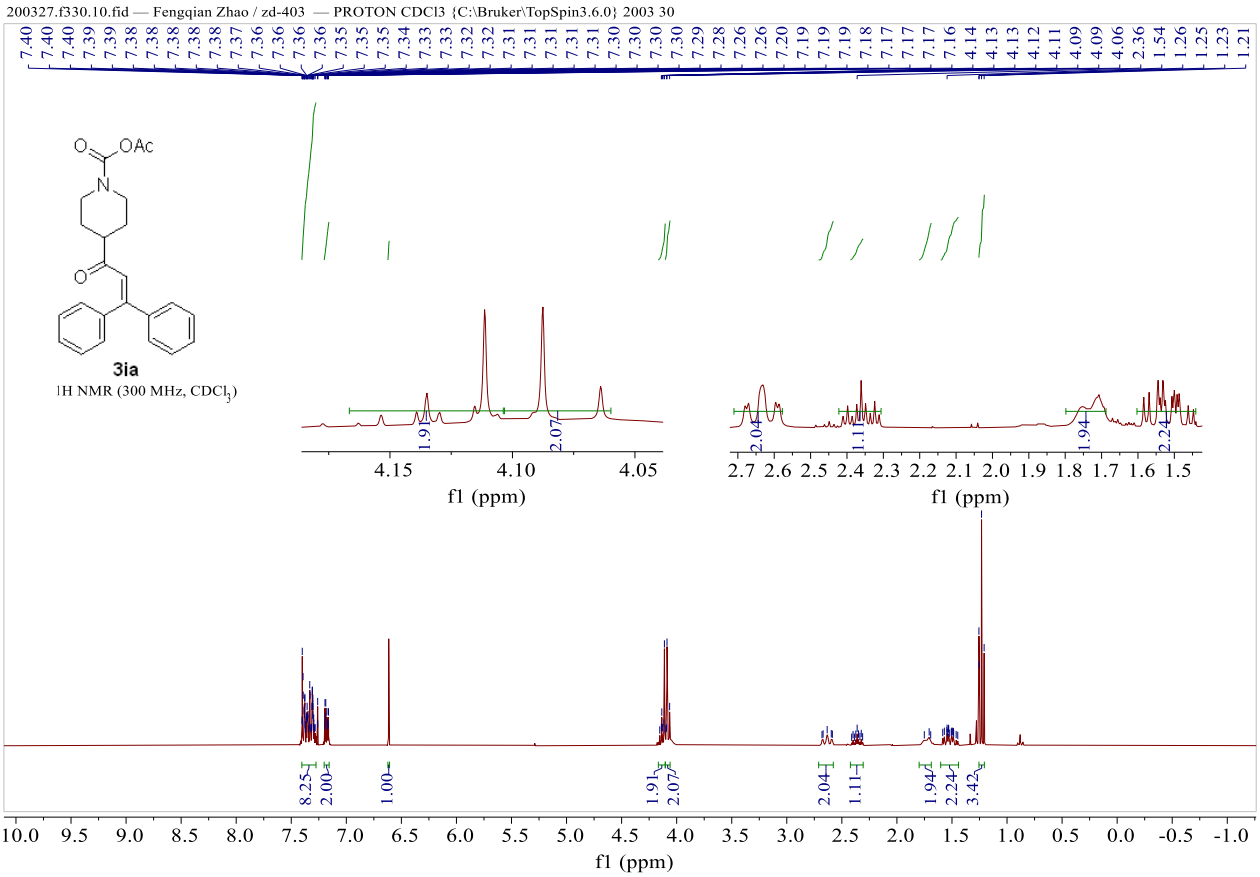

200324.f312.11.fid — Fengqian Zhao / zd-401 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2003 12
205.2
153.3
141.4
139.2
129.4
129.2
128.4
128.3
128.3
128.1
125.9
77.4
77.0
76.6
51.9
40.5
31.8
29.3
26.5
26.4
12.9
Cy
O
3fa
13C NMR (75 MHz, CDCl3)
f1 (ppm)

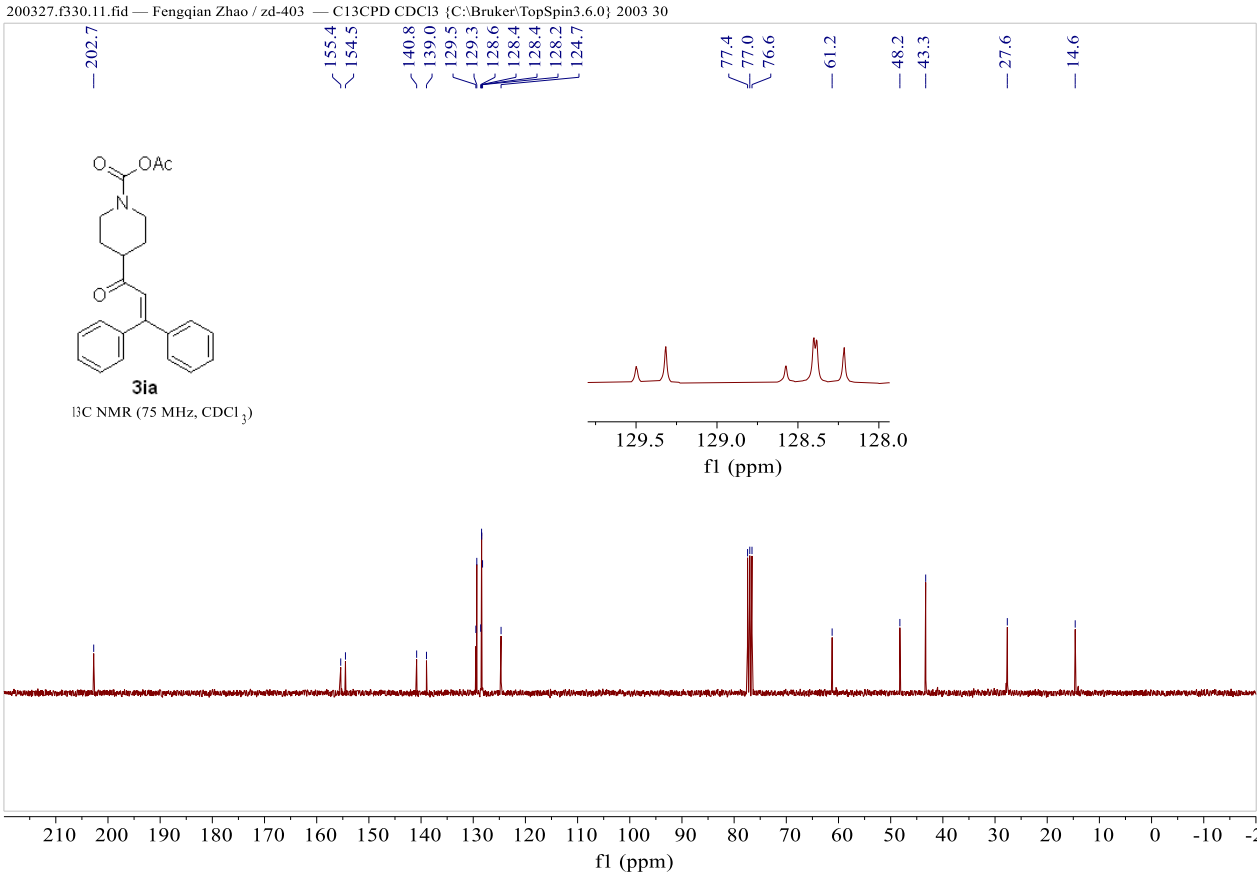

200324.f313.10.fid — Fengqian Zhao / zd-402 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2003 13
Ph
O
3ga
1H NMR (300 MHz, CDCl3)
f1 (ppm)

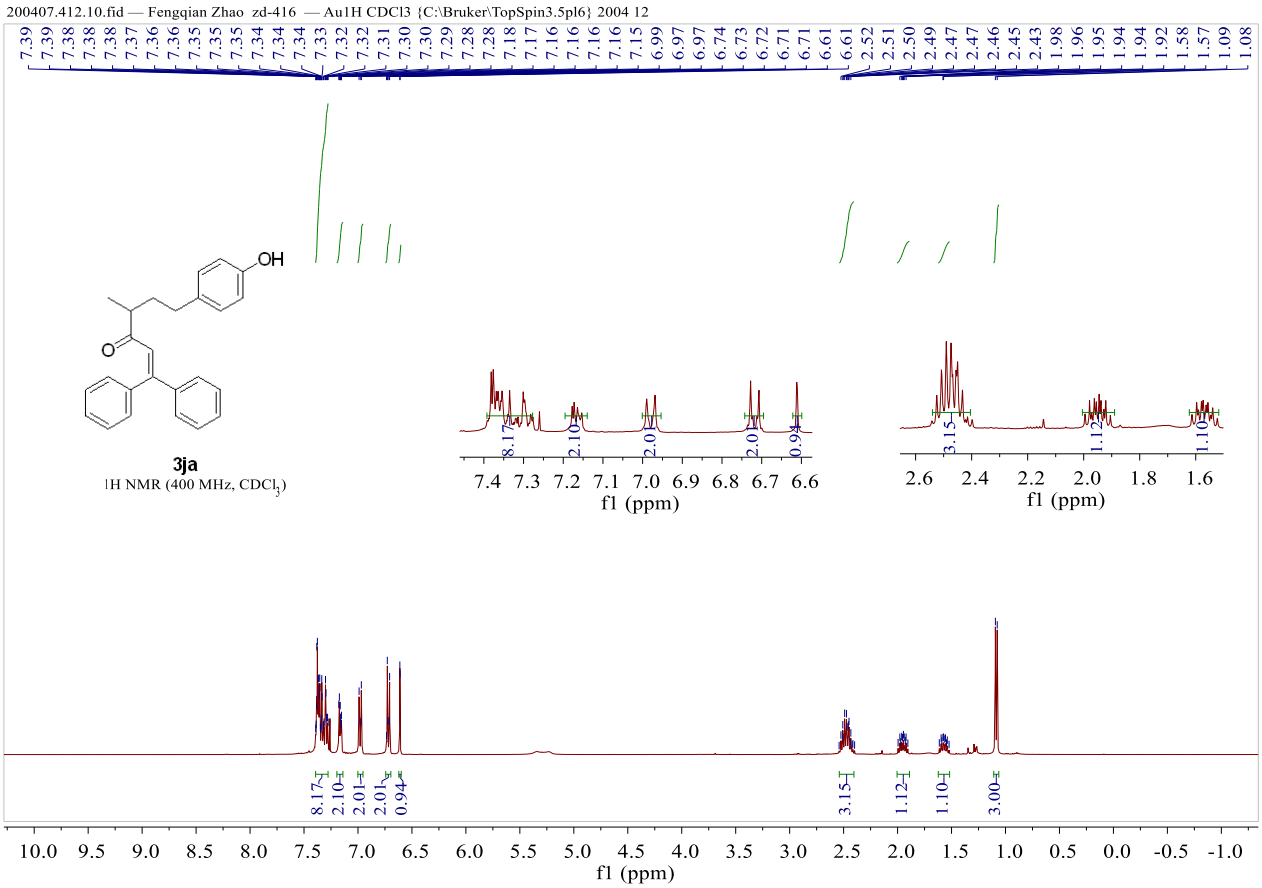

200324.f313.11.fid — Fengqian Zhao / zd-402 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2003 13
Ph
O
3ga
13C NMR (75 MHz, CDCl3)
f1 (ppm)

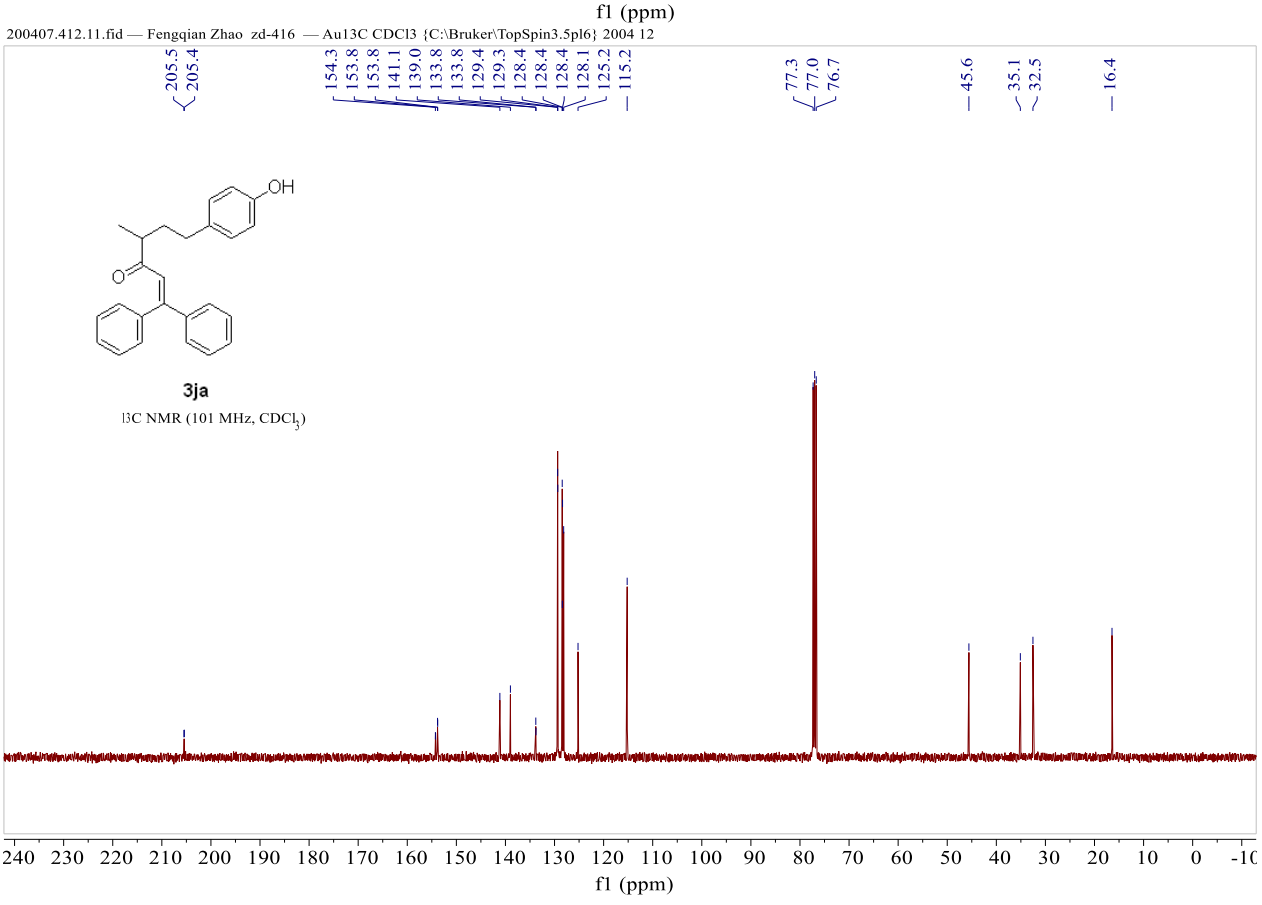

200407.413.10.fid — Fengqian Zhao zd-417 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2004 13
OMe
3ha
1H NMR (400 MHz, CDCl3)
f1 (ppm)

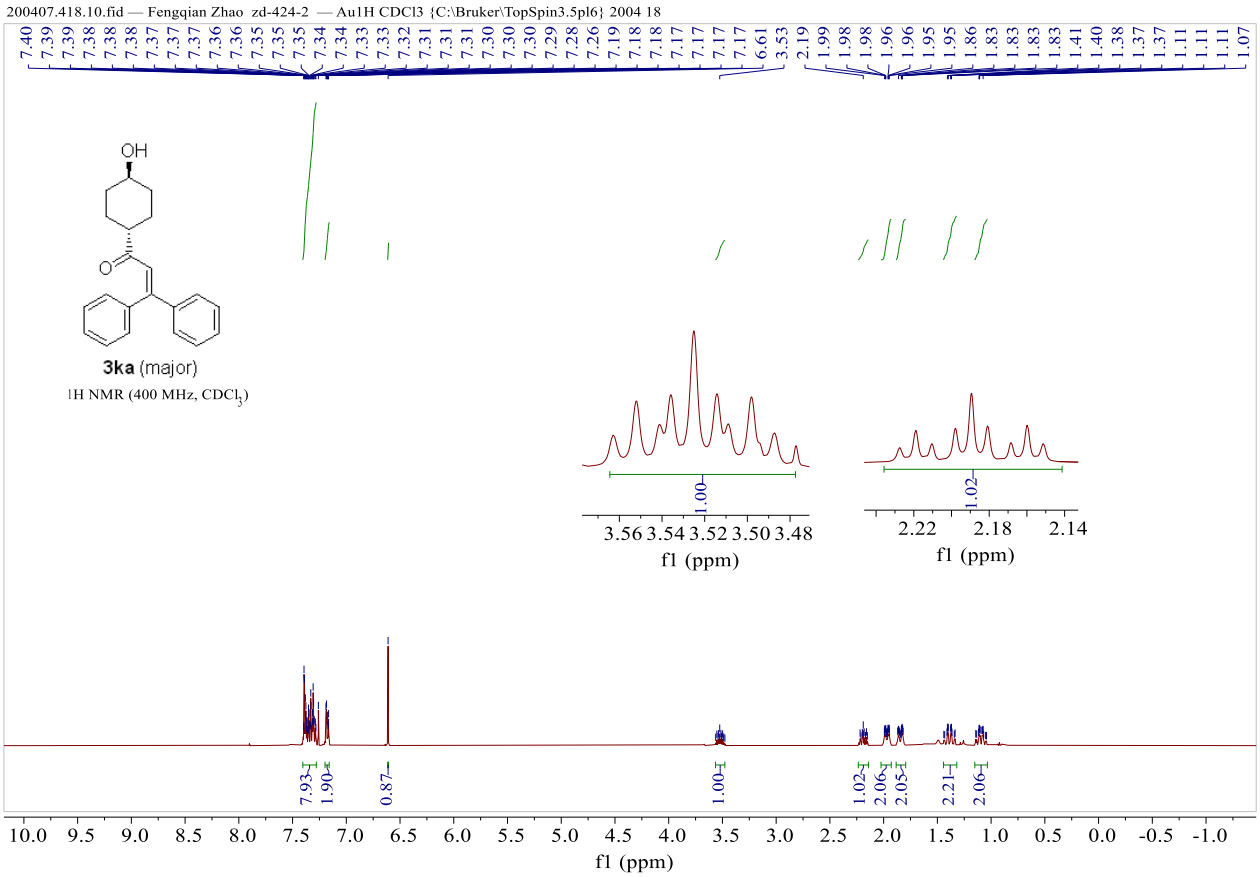

200407.413.11.fid — Fengqian Zhao zd-417 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2004 13
OMe
3ha
13C NMR (101 MHz, CDCl3)
f1 (ppm)

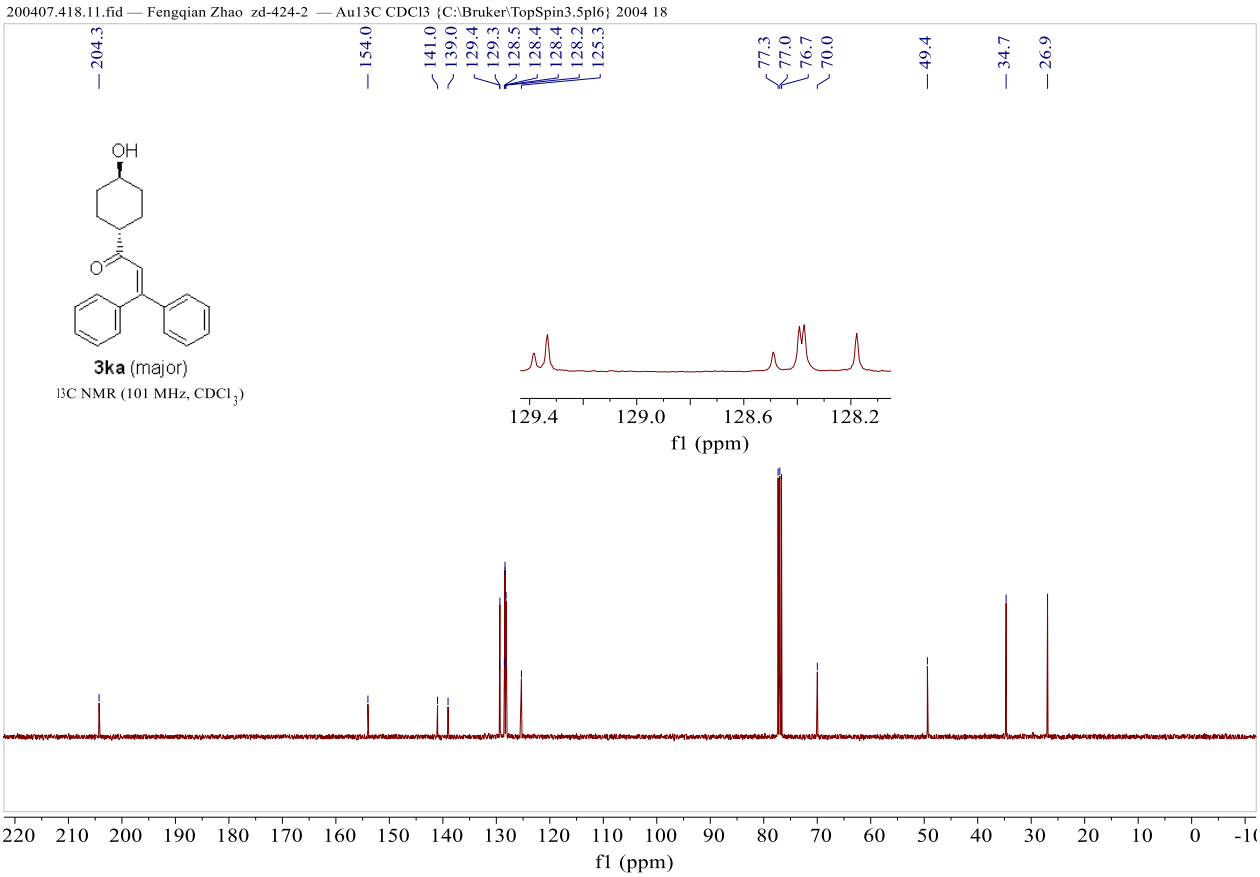

200327.f330.10.fid — Fengqian Zhao / zd-403 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2003 30
3ia
1H NMR (300 MHz, CDCl3)
f1 (ppm)

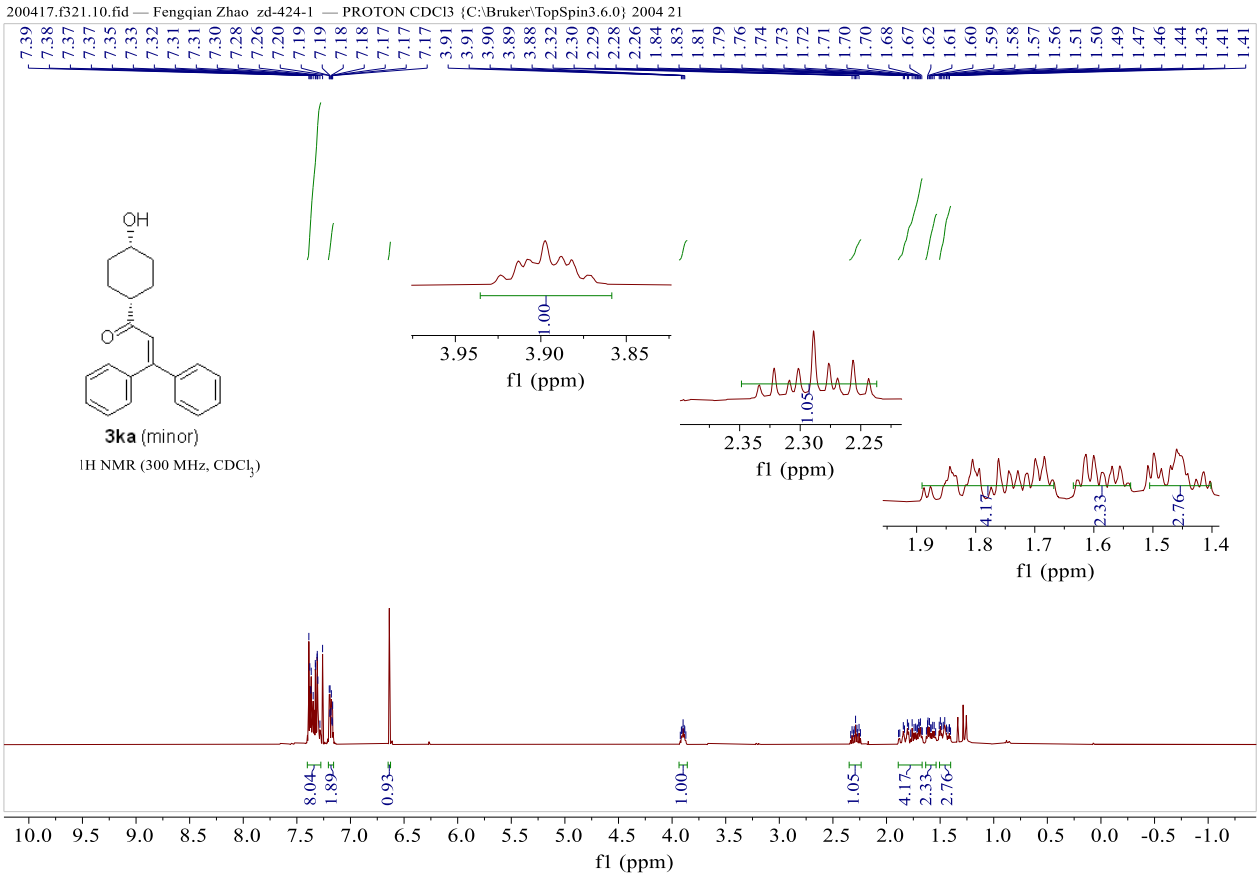

200327.f330.11.fid — Fengqian Zhao / zd-403 — C13CPD CDCl3 {C:\Bruker\TopSpin3.6.0} 2003 30
3ia
13C NMR (75 MHz, CDCl3)
f1 (ppm)

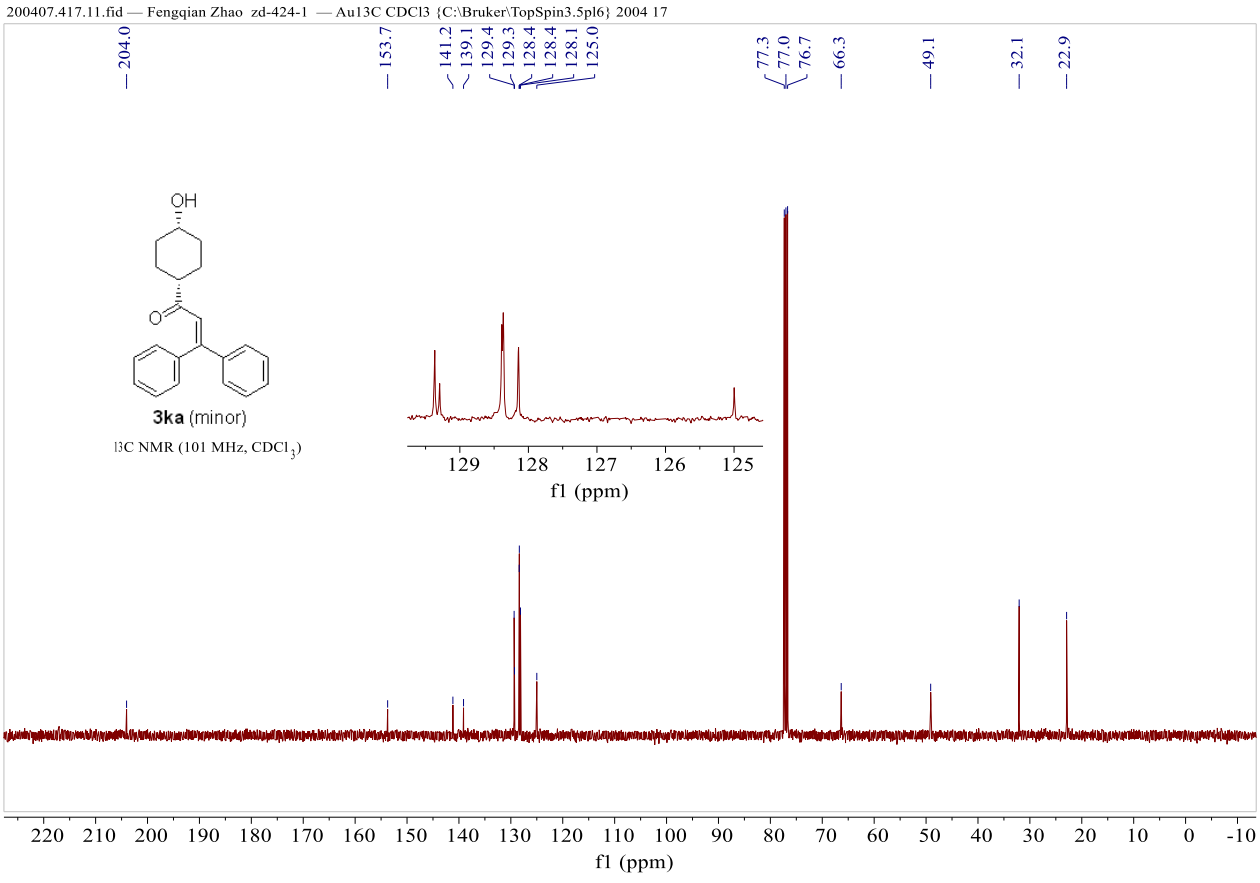

200407.412.10.fid — Fengqian Zhao zd-416 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2004 12
OH
O
3ja
1H NMR (400 MHz, CDCl3)
f1 (ppm)

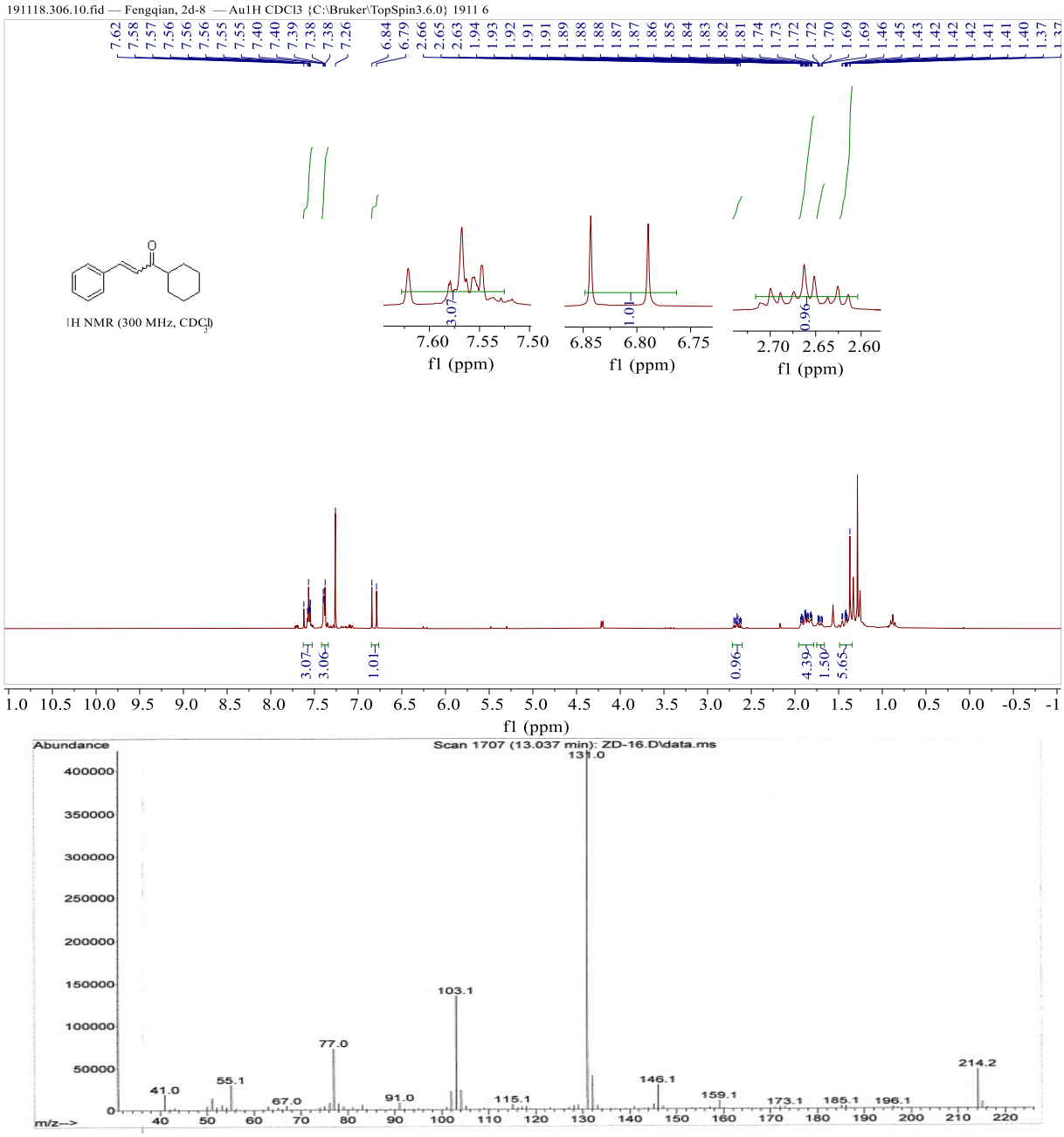

200407.412.11.fid — Fengqian Zhao zd-416 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2004 12
OH
O
3ja
13C NMR (101 MHz, CDCl3)
f1 (ppm)


200407.418.10.fid — Fengqian Zhao zd-424-2 — Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 2004 18
OH
O
3ka (major)
1H NMR (400 MHz, CDCl3)
1.00
1.02
f1 (ppm)
7.93
1.90
0.87
1.00
1.02
2.06
2.05
2.21
2.06
f1 (ppm)


200407.418.11.fid — Fengqian Zhao zd-424-2 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2004 18
204.3
154.0
141.0
139.0
129.4
129.3
128.5
128.4
128.4
128.2
125.3
77.3
77.0
76.7
70.0
49.4
34.7
26.9
OH
O
3ka (major)
13C NMR (101 MHz, CDCl3)
f1 (ppm)
f1 (ppm)


200417.f321.10.fid — Fengqian Zhao zd-424-1 — PROTON CDCl3 {C:\Bruker\TopSpin3.6.0} 2004 21
OH
O
3ka (minor)
1H NMR (300 MHz, CDCl3)
f1 (ppm)
f1 (ppm)
f1 (ppm)
f1 (ppm)


200407.417.11.fid — Fengqian Zhao zd-424-1 — Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 2004 17
OH
O
3ka (minor)
13C NMR (101 MHz, CDCl3)
f1 (ppm)
f1 (ppm)


191118.306.10.fid — Fengqian, 2d-8 — Au1H CDCl3 {C:\Bruker\TopSpin3.6.0} 1911 6
1H NMR (300 MHz, CDCl3)
f1 (ppm)

Abundance
Scan 1707 (13.037 min): ZD-16.D\data.ms
m/z-->