Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

2-Pyridinylmethyl Borrowing: Base-promoted C-Alkylation of (Pyridin-2-yl)methyl Alcohols with Ketones *via* Cleavage of Unstrained C(sp^3)–C(sp^3) Bonds

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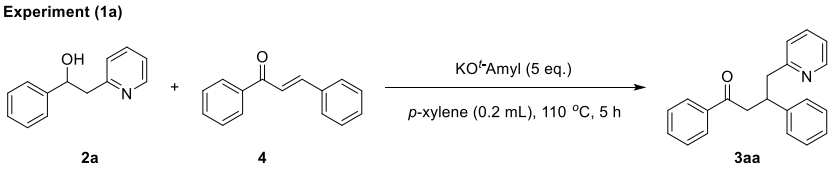
1. General Information

^{1}H and **^{13}C** NMR spectra were recorded on a Bruker Avance 600 MHz and 400 MHz instruments. Chemical shifts were reported in parts per million (ppm), and the residual solvent peak was used as an internal reference: proton (chloroform δ 7.26), carbon (chloroform δ 77.0) or tetramethylsilane (TMS δ 0.00) was used as a reference. Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (doublet of doublet), bs (broad singlet). Coupling constants were reported in Hertz (Hz). All high resolution mass spectra (**HRMS**) were obtained on a Bruker Apex-2. For thin layer chromatography (**TLC**), Qingdao Haiyang Chemical were used, and compounds were visualized with a UV light at 254 nm. Flash chromatography separations were performed on Qingdao Haiyang Chemical 300-400 mesh silica gel. All reactions were carried out under nitrogen atmosphere. All commercially available reagents were used as received for the reactions without any purification. The (pyridin-2-yl)methyl alcohols are commercially available or synthesis via the known procedures.[1]

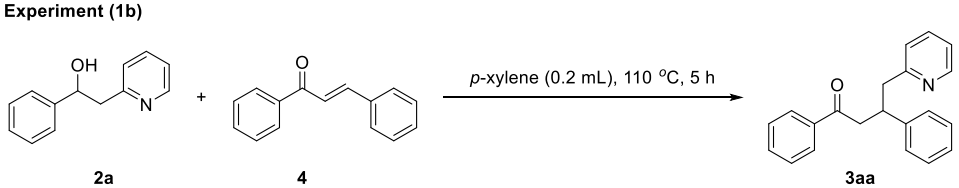
2. Experimental Procedures

To a vacuum reaction tube equipped with a dried stir bar was added (pyridin-2-yl)methyl alcohols (0.2 mmol), ketones (0.1 mmol), KO^{t}Amyl (0.5 mmol), *p*-xylene in the glovebox. The reaction mixture was taken outside the glovebox. Then, the reaction mixture was allowed to stir at the settle temperature for 20 hours. The reaction mixture was added to water (10 mL), extracted with EtOAc (3 × 5 mL). The organic layer was washed with aqueous $NaHCO_3$ and brine and dried over Na_2SO_4. And the residue was purified by column chromatography with silica gel to give pure products.

3. Mechanistic studies



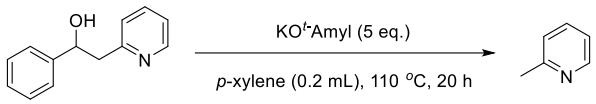
Experiment (1a): To a vacuum reaction tube equipped with a dried stir bar was added (pyridin-2-yl)methyl alcohol **2a** (0.2 mmol), chalcone **4** (0.1 mmol), KO^{t}Amyl (0.5 mmol), *p*-xylene (0.2 mL) in the glovebox. The reaction mixture was taken outside the glovebox. Then, the reaction mixture was allowed to stir at the settle temperature for 5 hours. The reaction mixture was added to water (10 mL), extracted with EtOAc (3 × 5 mL). The organic layer was washed with aqueous $NaHCO_3$ and brine and dried over Na_2SO_4. And the residue was purified by column chromatography with silica gel to give pure products. We got the corresponding product **3aa** with 21% yield, showing that KO^{t}Amyl improves the cleavage of C-C((pyridin-2-yl)methyl) and the conjugate 1,4-addition of the 2-pyridinylmethyl moiety to chalcone.



Experiment (1b): To a vacuum reaction tube equipped with a dried stir bar was added (pyridin-2-yl)methyl alcohol **2a** (0.2 mmol), chalcone **4** (0.1 mmol), *p*-xylene (0.2 mL) in the glovebox. The reaction mixture was taken outside the

glovebox. Then, the reaction mixture was allowed to stir at the settle temperature for 5 hours. No product **3aa** was observed, further demonstrating the conclusion of experiment **(1a)**.

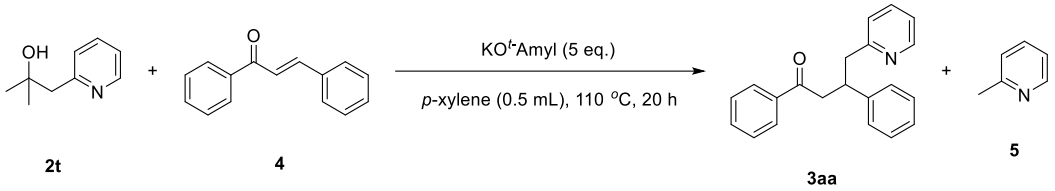
Experiment (2)



2a **5**

Experiment (2): To a vacuum reaction tube equipped with a dried stir bar was added (pyridin-2-yl)methyl alcohol **2a** (0.2 mmol), chalcone **4** (0.1 mmol), KO^{t}-Amyl (0.5 mmol), *p*-xylene (0.5 mL) in the glovebox. The reaction mixture was taken outside the glovebox. Then, the reaction mixture was allowed to stir at the settle temperature for 20 hours. The crude reaction mixture was given HRMS. We got the 2-methylpyridine **5** (HRMS (m/z): $[M+H]^+$ C_6H_8N found 94.0656), showing that KO^{t}-Amyl improves the cleavage of C-C((pyridin-2-yl)methyl).

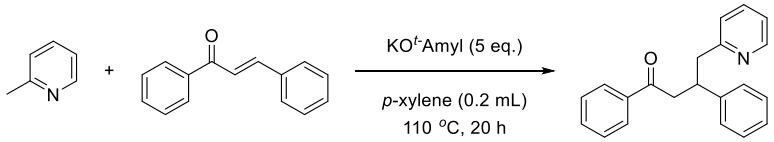
Experiment (3)



2t **4** **3aa** **5**

Experiment (3): To a vacuum reaction tube equipped with a dried stir bar was added (pyridin-2-yl)methyl alcohol **2a** (0.2 mmol), chalcone **4** (0.1 mmol), KO^{t}-Amyl (0.5 mmol), *p*-xylene (0.5 mL) in the glovebox. The reaction mixture was taken outside the glovebox. Then, the reaction mixture was allowed to stir at the settle temperature for 20 hours. The crude reaction mixture was given HRMS. We got the corresponding product **3aa** (HRMS (m/z): $[M+H]^+$ $C_{21}H_{20}NO$ found 302.1539), and 2-methylpyridine **5** (HRMS (m/z): $[M+H]^+$ C_6H_8N found 94.0653), which shows that KO^{t}-Amyl improves the cleavage of C-C((pyridin-2-yl)methyl) of tertiary alcohol and the conjugate 1,4-addition of the 2-pyridinylmethyl moiety to chalcone.

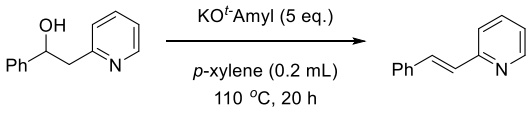
Experiment (4)



5 **4** **3aa,** not detected

Experiment (4): To a vacuum reaction tube equipped with a dried stir bar was added 2-methylpyridine **5** (0.2 mmol), chalcone **4** (0.1 mmol), KO^{t}-Amyl (0.5 mmol), *p*-xylene (0.2 mL) in the glovebox. The reaction mixture was taken outside the glovebox. Then, the reaction mixture was allowed to stir at the settle temperature for 20 hours. And we did not monitor the corresponding products **3aa** and some of the starting material was recovered, which indicated that KO^{t}-Amyl cannot take benzyl hydrogen from 2-methylpyridine, which indicated that the *β*-carbon elimination of 2-pyridinylmethyl secondary alcohol could active the 2-pyridinylmethyl of the starting material in this transformation.

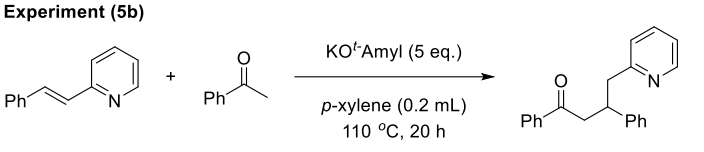
Experiment (5a)



2a **P1**
30% yield

Experiment (5a): To a vacuum reaction tube equipped with a dried stir bar was added (pyridin-2-yl)methyl alcohol **2a** (0.2 mmol), KO^{t}-Amyl (0.5 mmol), *p*-xylene (0.2 mL) in the glovebox. The reaction mixture was taken outside the glovebox. Then, the reaction mixture was allowed to stir at the settle temperature for 20 hours. The reaction mixture was added to water (10 mL), extracted with EtOAc (3 × 5 mL). The organic layer was washed with aqueous $NaHCO_3$ and brine and dried over Na_2SO_4. And the residue was purified by column chromatography with silica gel to give pure products. We got 2-alkenyl pyridine **P1** with 30% yield.

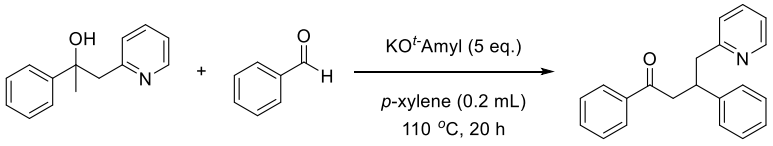
Experiment (5b)



P1 **1a** **3aa,** 21% yield
6% total yield of two steps

Experiment (5b): To a vacuum reaction tube equipped with a dried stir bar was added 2-alkenyl pyridine **P1** (0.2 mmol), acetophenone **1a**, KO^{t}-Amyl (0.5 mmol), *p*-xylene (0.2 mL) in the glovebox. The reaction mixture was taken outside the glovebox. Then, the reaction mixture was allowed to stir at the settle temperature for 20 hours. The reaction mixture was added to water (10 mL), extracted with EtOAc (3 × 5 mL). The organic layer was washed with aqueous $NaHCO_3$ and brine and dried over Na_2SO_4. And the residue was purified by column chromatography with silica gel to give pure products. We got the **3aa** with 21% yield, and the total yield of the two steps (experiment 5a, 5b) was 6%, which indicated that the mechanism is not the main mechanism for this transformation, including the dehydration of **2a** to give 2-alkenyl pyridine, followed by Michael addition of **1a** to 2-alkenyl pyridine. And the mechanism of 2-pyridinylmethyl borrowing via the C-C bond cleavage occupying a leading position.

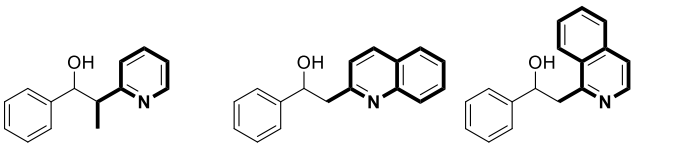
Experiment (6): 2-pyridinylmethyl tertiary alcohol and benzaldehyde were also examined under the standard reaction conditions as following: To a vacuum reaction tube equipped with a dried stir bar was added 2-phenyl-1-(pyridin-2-yl)propan-2-ol (0.2 mmol), benzaldehyde (0.1 mmol), KO^{t}-Amyl (0.5 mmol), *p*-xylene (0.5 mL) in the glovebox. The reaction mixture was taken outside the glovebox. Then, the reaction mixture was allowed to stir at the settle temperature for 20 hours. However, the corresponding products was not detected. In this reaction, trace amounts of by-products such as 2-methylpyridine and acetophenone were observed by TLC.



S7 **S8** **3aa,** not detected

Substrate limitations:

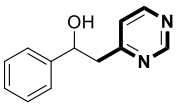
The following substrates were examined under the standard reaction conditions (see section 2 experimental procedures). However, low conversion of the alcohols were noted, as shown below.



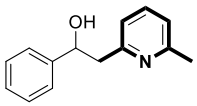
S2s
ND

S2u
20% yield

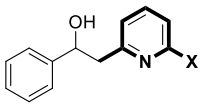
S2v
ND



S2w
ND



S2x
<5% yield

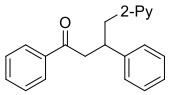


S2y, X = F, ND
S2z, X = Cl, ND

ND = Not Determined.

4. Characterization data of products

1,3-diphenyl-4-(pyridin-2-yl)butan-1-one (3aa)[2]

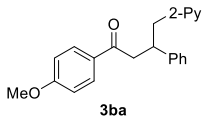


3aa

The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.29 in hexane:ethyl acetate = 3:1) resulting in 18.0 mg of colorless solid in 60% yield, melting point 89-90 ^{o}C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.48 (d, *J* = 5.4 Hz, 1H), 7.85 (d, *J* = 7.8 Hz, 2H), 7.52-7.47 (m, 2H), 7.40 (t, *J* = 7.8 Hz, 2H), 7.24-7.20 (m, 4H), 7.16-7.11 (m, 1H), 7.06 (dd, *J* = 7.2 Hz, 4.8 Hz, 1H), 7.03 (d, *J* = 7.8 Hz, 1H), 3.95-3.89 (m, 1H), 3.44-3.30 (m, 2H), 3.24-3.19 (m, 1H), 3.17-3.12 (m 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 198.68, 159.89, 149.16, 144.05, 137.20, 136.15, 132.84, 128.46, 128.41, 128.06, 127.58, 126.42, 123.69, 121.25, 45.10, 44.39, 41.71.

1-(4-methoxyphenyl)-3-phenyl-4-(pyridin-2-yl)butan-1-one (3ba)

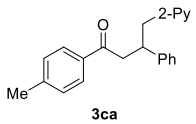


3ba

The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.15 in hexane:ethyl acetate = 3:1) resulting in 16.9 mg of gray solid in 51% yield, melting point 109-110 ^{o}C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.48 (d, *J* = 4.8 Hz, 1H), 7.84 (d, *J* = 8.4 Hz, 2H), 7.52-7.47 (m, 1H), 7.24-7.19 (m, 4H), 7.16-7.10 (m, 1H), 7.07-7.01 (m, 2H), 6.87 (d, *J* = 8.4 Hz, 2H), 3.93-3.87 (m, 1H), 3.84 (s, 3H), 3.37-3.26 (m, 2H), 3.22 (dd, *J* = 13.2 Hz, 7.8 Hz, 1H), 3.14 (dd, *J* = 13.8 Hz, 7.8 Hz, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 197.21, 163.33, 159.98, 149.12, 144.14, 136.12, 130.34, 128.37, 127.59, 126.36, 123.67, 121.20, 113.61, 55.41, 45.09, 44.08, 41.88.
HRMS (ESI): $C_{22}H_{22}NO_2$ $[M+H]^+$ calculated 332.1645, found 332.1643.

3-phenyl-4-(pyridin-2-yl)-1-(*p*-tolyl)butan-1-one (3ca)



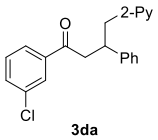
3ca

The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.35 in

hexane:ethyl acetate = 3:1) resulting in 14.2 mg of colorless solid in 45% yield, melting point 114-115 ^{o}C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.48 (d, *J* = 4.8 Hz, 1H), 7.76 (d, *J* = 7.8 Hz, 2H), 7.46-7.52 (m, 1H), 7.24-7.20 (m, 4H), 7.19 (d, *J* = 7.8 Hz, 2H), 7.15-7.10 (m, 1H), 7.06 (dd, *J* = 7.8 Hz, 4.8 Hz, 1H), 7.03 (d, *J* = 7.8 Hz, 1H), 3.96-3.86 (m, 1H), 3.41-3.29 (m, 2H), 3.22 (dd, *J* = 13.8 Hz, 7.8 Hz, 1H), 3.14 (dd, *J* = 13.8 Hz, 7.2 Hz, 1H), 2.37 (s, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 198.31, 159.94, 149.11, 144.10, 143.59, 136.14, 134.74, 129.14, 128.38, 128.19, 127.59, 126.38, 123.68, 121.22, 45.08, 44.29, 41.78, 21.57.
HRMS (ESI): $C_{22}H_{22}NO$ $[M+H]^+$ calculated 316.1696, found 316.1694.

1-(3-chlorophenyl)-3-phenyl-4-(pyridin-2-yl)butan-1-one (3da)

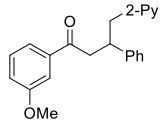


3da

The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.40 in hexane:ethyl acetate = 3:1) resulting in 19.8 mg of yellow oil in 59% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.50 (d, *J* = 4.8 Hz, 1H), 7.83-7.79 (m, 1H), 7.72 (d, *J* = 7.8 Hz, 1H), 7.54-7.50 (m, 1H), 7.49-7.46 (m, 1H), 7.34 (t, *J* = 7.8 Hz, 1H), 7.25-7.20 (m, 4H), 7.17-7.13 (m, 1H), 7.10-7.06 (m, 1H), 7.03 (d, *J* = 7.8 Hz, 1H), 3.95-3.86 (m, 1H), 3.38-3.29 (m, 2H), 3.24-3.12 (m, 2H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 197.45, 159.71, 149.20, 143.80, 138.68, 136.23, 134.81, 132.77, 129.79, 128.48, 128.25, 127.53, 126.56, 126.13, 123.75, 121.35, 45.00, 44.43, 41.66.
HRMS (ESI): $C_{21}H_{19}ClNO$ $[M+H]^+$ calculated 336.1150, found 336.1147.

1-(3-methoxyphenyl)-3-phenyl-4-(pyridin-2-yl)butan-1-one (3ea)

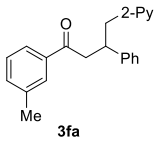


3ea

The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.43 in hexane:ethyl acetate = 3:1) resulting in 23.9 mg of yellow oil in 72% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.49 (d, *J* = 4.8 Hz, 1H), 7.50 (t, *J* = 7.8 Hz, 1H), 7.44 (d, *J* = 7.8 Hz, 1H), 7.37 (s, 1H), 7.30 (t, *J* = 8.4 Hz, 1H), 7.24-7.19 (m, 4H), 7.17-7.11 (m, 1H), 7.08-7.03 (m, 2H), 7.02 (d, *J* = 7.8 Hz, 1H), 3.95-3.88 (m, 1H), 3.80 (s, 3H), 3.41-3.30 (m, 2H), 3.21 (dd, *J* = 13.2 Hz, 7.8 Hz, 1H), 3.14 (dd, *J* = 13.8 Hz, 7.2 Hz, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 198.51, 159.87, 159.76, 149.17, 144.02, 138.55, 136.15, 129.44, 128.41, 127.58, 126.43, 123.69, 121.25, 120.72, 119.48, 112.26, 55.40, 45.08, 44.49, 41.75.
HRMS (ESI): $C_{22}H_{22}NO_2$ $[M+H]^+$ calculated 332.1645, found 332.1642.

3-phenyl-4-(pyridin-2-yl)-1-(*m*-tolyl)butan-1-one (3fa)

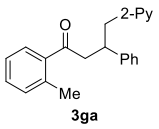


3fa

The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.50 in hexane:ethyl acetate = 3:1) resulting in 20.9 mg of colorless solid in 66% yield, melting point 60-61 ^{o}C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.49 (d, *J* = 4.8 Hz, 1H), 7.69-7.62 (m, 2H), 7.53-7.47 (m, 1H), 7.34-7.26 (m, 2H), 7.24-7.20 (m, 4H), 7.16-7.11 (m, 1H), 7.07-7.04 (m, 1H), 7.04 (d, *J* = 7.8 Hz, 1H), 3.95-3.88 (m, 1H), 3.41-3.30 (m, 2H), 3.22 (dd, *J* = 13.2 Hz, 7.8 Hz, 1H), 3.14 (dd, *J* = 13.2 Hz, 7.2 Hz, 1H), 2.36 (s, 3H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 198.87, 159.95, 149.14, 144.11, 138.21, 137.24, 136.12, 133.59, 128.61, 128.38, 128.33, 127.59, 126.39, 125.26, 123.67, 121.22, 45.07, 44.46, 41.73, 21.29.
HRMS (ESI): $C_{22}H_{22}NO$ $[M+H]^+$ calculated 316.1696, found 316.1695.

3-phenyl-4-(pyridin-2-yl)-1-(*o*-tolyl)butan-1-one (3ga)



3ga

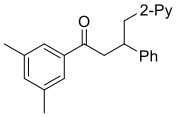
The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.43 in hexane:ethyl acetate = 3:1) resulting in 17.1 mg of yellow oil in 54% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.50 (d, *J* = 4.8 Hz, 1H), 7.52-7.47 (m, 1H), 7.45 (d, *J* = 7.2 Hz, 1H), 7.30 (t, *J* = 7.8 Hz, 1H), 7.23-7.17 (m, 3H), 7.17-7.12 (m, 4H), 7.09-7.04 (m, 1H), 7.00 (d, *J* = 7.2 Hz, 1H), 3.89-3.78 (m, 1H), 3.43-3.24 (m, 2H), 3.21-3.05 (m, 2H), 2.18 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 203.24, 159.82, 149.22, 143.70, 138.47, 137.79, 136.12, 131.69, 130.89, 128.39, 128.06, 127.66, 126.45, 125.46, 123.66, 121.26, 47.48, 45.29, 42.04, 20.61.

HRMS (ESI): $C_{22}H_{22}NO$ $[M+H]^+$ calculated 316.1696, found 316.1693.

1-(3,5-dimethylphenyl)-3-phenyl-4-(pyridin-2-yl)butan-1-one (3ha)



3ha

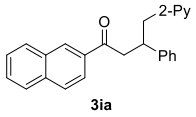
The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.50 in hexane:ethyl acetate = 3:1) resulting in 22.8 mg of yellow oil in 69% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.49 (d, *J* = 4.2 Hz, 1H), 7.53-7.48 (m, 1H), 7.45 (s, 2H), 7.24-7.20 (m, 4H), 7.16-7.12 (m, 2H), 7.10-7.02 (m, 2H), 3.94-3.85 (m, 1H), 3.40-3.27 (m, 2H), 3.22 (dd, *J* = 13.8 Hz, 7.8 Hz, 1H), 3.14 (dd, *J* = 13.2 Hz, 7.2 Hz, 1H), 2.32 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 199.03, 160.01, 149.14, 144.17, 138.05, 137.31, 136.12, 134.46, 128.37, 127.61, 126.37, 125.87, 123.67, 121.21, 45.05, 44.52, 41.75, 21.17.

HRMS (ESI): $C_{23}H_{24}NO$ $[M+H]^+$ calculated 330.1852, found 330.1850.

1-(naphthalen-2-yl)-3-phenyl-4-(pyridin-2-yl)butan-1-one (3ia)



3ia

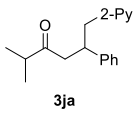
The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.30 in hexane:ethyl acetate = 3:1) resulting in 24.3 mg of yellow oil in 69% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.50 (d, *J* = 4.8 Hz, 1H), 8.37 (s, 1H), 7.91 (d, *J* = 8.4 Hz, 2H), 7.83 (t, *J* = 7.8 Hz, 2H), 7.58 (t, *J* = 7.2 Hz, 1H), 7.55-7.49 (m, 2H), 7.28-7.21 (m, 4H), 7.14 (t, *J* = 7.2 Hz, 1H), 7.09-7.05 (m, 2H), 4.01-3.92 (m, 1H), 3.53-3.47 (m, 2H), 3.27 (dd, *J* = 13.2 Hz, 7.8 Hz, 1H), 3.19 (dd, *J* = 13.8 Hz, 7.8 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 198.65, 159.96, 149.18, 144.10, 136.18, 135.51, 134.52, 132.51, 129.72, 129.55, 128.44, 128.32, 128.29, 127.72, 127.61, 126.65, 126.46, 123.93, 123.73, 121.28, 45.15, 44.46, 41.95.

HRMS (ESI): $C_{25}H_{22}NO$ $[M+H]^+$ calculated 352.1696, found 352.1693.

2-methyl-5-phenyl-6-(pyridin-2-yl)hexan-3-one (3ja)



3ja

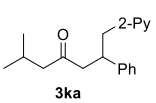
The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.28 in hexane:ethyl acetate = 3:1) resulting in 13.9 mg of yellow oil in 52% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.49 (d, *J* = 4.8 Hz, 1H), 7.53-7.47 (m, 1H), 7.25-7.21 (m, 2H), 7.20-7.12 (m, 3H), 7.09-7.04 (m, 1H), 7.00 (d, *J* = 7.8 Hz, 1H), 3.77-3.69 (m, 1H), 3.14-3.04 (m, 2H), 2.87 (dd, *J* = 16.8 Hz, 8.4 Hz, 1H), 2.80 (dd, *J* = 16.8 Hz, 6.0 Hz, 1H), 2.45-2.39 (m, 1H), 0.94 (d, *J* = 6.6 Hz, 3H), 0.89 (d, *J* = 7.2 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 213.09, 159.90, 149.12, 144.05, 136.12, 128.38, 127.54, 126.40, 123.61, 121.22, 46.35, 44.89, 41.43, 41.13, 17.83, 17.79.

HRMS (ESI): $C_{18}H_{22}NO$ $[M+H]^+$ calculated 268.1696, found 268.1700.

6-methyl-2-phenyl-1-(pyridin-2-yl)heptan-4-one (3ka)



3ka

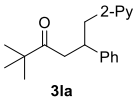
The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.51 in hexane:ethyl acetate = 3:1) resulting in 20.3 mg of yellow oil in 72% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.50-8.48 (m 1H), 7.52-7.46 (m, 1H), 7.23 (t, *J* = 7.2 Hz, 2H), 7.19-7.12 (m, 3H), 7.06 (dd, *J* = 7.2 Hz, 4.8 Hz, 1H), 6.97 (d, *J* = 7.8 Hz, 1H), 3.79-3.64 (m, 1H), 3.15-3.01(m, 2H), 2.87-2.61 (m, 2H), 2.17-2.08 (m, 2H), 2.03-1.93 (m, 1H), 0.77-0.73 (m, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 209.30, 159.81, 149.15, 143.85, 136.10, 128.39, 127.54, 126.43, 123.65, 121.23, 52.38, 48.95, 45.11, 41.50, 24.29, 22.48, 22.38.

HRMS (ESI): $C_{19}H_{24}NO$ $[M+H]^+$ calculated 282.1852, found 282.1852

2,2-dimethyl-5-phenyl-6-(pyridin-2-yl)hexan-3-one (3la)



3la

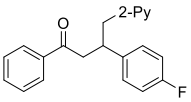
The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.50 in hexane:ethyl acetate = 3:1) resulting in 9.3 mg of yellow oil in 33% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.48 (d, *J* = 4.8 Hz, 1H), 7.52-7.46 (m, 1H), 7.22 (t, *J* = 7.2 Hz, 2H), 7.19-7.16 (m, 2H), 7.13 (t, *J* = 7.2 Hz, 1H), 7.05 (dd, *J* = 7.2 Hz, 4.8 Hz, 1H), 7.02 (d, *J* = 7.8 Hz, 1H), 3.78-3.70 (m, 1H), 3.17-3.03 (m, 2H), 3.00-2.75 (m, 2H), 0.97 (s, 9H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 213.90, 160.08, 149.06, 144.28, 136.07, 128.28, 127.65, 126.29, 123.47, 121.14, 44.62, 44.06, 42.72, 41.18, 26.04.

HRMS (ESI): $C_{19}H_{24}NO$ $[M+H]^+$ calculated 282.1852, found 282.1853

3-(4-fluorophenyl)-1-phenyl-4-(pyridin-2-yl)butan-1-one (3ab)



3ab

The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.33 in hexane:ethyl acetate = 3:1) resulting in 20.2 mg of colorless solid in 63% yield, melting point 104-105 ^{o}C.

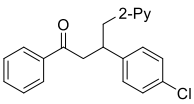
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.49 (d, *J* = 4.8 Hz, 1H), 7.85 (d, *J* = 7.8 Hz, 2H), 7.51 (t, *J* = 7.2 Hz, 2H), 7.40 (t, *J* = 7.8 Hz, 2H), 7.18-7.13 (m, 2H), 7.09-7.05 (m, 1H), 7.02 (d, *J* = 7.8 Hz, 1H), 6.90 (t, *J* = 8.4 Hz, 2H), 3.95-3.88 (m, 1H), 3.35 (d, *J* = 7.2 Hz, 2H), 3.21 (dd, *J* = 13.8 Hz, 7.8 Hz, 1H), 3.10 (dd, *J* = 13.2 Hz, 7.2 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 198.50, 161.42 (d, *J* = 244.0 Hz), 159.63, 149.18, 139.60 (d, *J* = 3.0 Hz), 137.10, 136.23, 132.94, 129.00 (d, *J* = 8.0 Hz), 128.51, 128.03, 123.69, 121.35, 115.16 (d, *J* = 21.0 Hz), 45.15, 44.47, 41.03.

^{19}F NMR (376 MHz, $CDCl_3$) δ -116.67.

HRMS (ESI): $C_{21}H_{19}FNO$ $[M+H]^+$ calculated 320.1445, found 320.1443

3-(4-chlorophenyl)-1-phenyl-4-(pyridin-2-yl)butan-1-one (3ac)



3ac

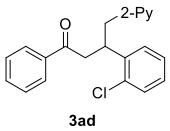
The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.25 in hexane:ethyl acetate = 3:1) resulting in 17.1 mg of colorless solid in 51% yield, melting point 108-109 ^{o}C.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.52-8.47 (m, 1H), 7.87-7.81 (m, 2H), 7.56-7.47 (m, 2H), 7.41 (t, *J* = 7.2 Hz, 2H), 7.19 (d, *J* = 8.4 Hz, 2H), 7.14 (d, *J* = 8.4 Hz, 2H), 7.08-7.05 (m, 1H), 7.01 (d, *J* = 7.8 Hz, 1H), 3.95-3.88 (m, 1H), 3.36 (d, *J* =6.6 Hz, 2H), 3.20 (dd, *J* = 13.8 Hz, 7.8 Hz, 1H), 3.10 (dd, *J* = 13.8 Hz, 7.8 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 198.34, 159.48, 149.26, 142.50, 137.03, 136.24, 133.01, 132.06, 128.98, 128.53, 128.03, 123.66, 121.38, 44.96, 44.26, 41.06.

HRMS (ESI): $C_{21}H_{19}ClNO$ $[M+H]^+$ calculated 336.1150, found 336.1149

3-(2-chlorophenyl)-1-phenyl-4-(pyridin-2-yl)butan-1-one (3ad)



3ad

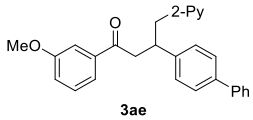
The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.36 in hexane:ethyl acetate = 3:1) resulting in 21.5 mg of yellow oil in 64% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.47-8.44 (m, 1H), 7.91-7.86 (m, 2H), 7.54-7.48 (m, 2H), 7.41 (t, *J* = 7.8 Hz, 2H), 7.32-7.29 (m, 1H), 7.28-7.25 (m, 1H), 7.17-7.13 (m, 1H), 7.12-7.08 (m, 2H), 7.07-7.04 (m, 1H), 4.47-4.38 (m, 1H), 3.52-3.37 (m, 2H), 3.25-3.16 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 198.26, 159.61, 149.07, 141.13, 137.03, 136.22, 133.97, 132.94, 129.84, 128.49, 128.22, 128.07, 127.53, 126.89, 123.59, 121.34, 43.39, 42.75, 37.98.

HRMS (ESI): $C_{21}H_{19}ClNO$ $[M+H]^+$ calculated 336.1150, found 336.1151

3-([1,1'-biphenyl]-4-yl)-1-(3-methoxyphenyl)-4-(pyridin-2-yl)butan-1-one (3ae)



3ae

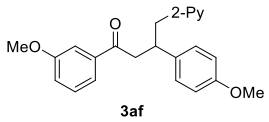
The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.34 in hexane:ethyl acetate = 3:1) resulting in 28.6 mg of yellow oil in 70% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.53-8.50 (m, 1H), 7.57-7.51 (m, 3H), 7.48 (d, *J* =7.8 Hz, 3H), 7.42-7.38 (m, 3H), 7.34-7.29 (m, 4H), 7.10-7.05 (m, 3H), 4.01-3.95 (m, 1H), 3.81 (s, 3H), 3.47-3.34 (m, 2H), 3.27-3.17 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 198.50, 159.84, 159.79, 149.22, 143.19, 140.90, 139.24, 138.55, 136.20, 129.46, 128.67, 127.99, 127.11, 127.05, 126.95, 123.72, 121.30, 120.74, 119.50, 112.30, 55.41, 45.08, 44.45, 41.39.

HRMS (ESI): $C_{28}H_{26}NO_2$ $[M+H]^+$ calculated 408.1958, found 408.1957

1-(3-methoxyphenyl)-3-(4-methoxyphenyl)-4-(pyridin-2-yl)butan-1-one (3af)



3af

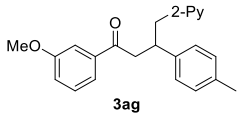
The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.23 in hexane:ethyl acetate = 3:1) resulting in 14.1 mg of yellow oil in 39% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.51-8.47 (m, 1H), 7.52-7.47 (m, 1H), 7.44 (d, *J* = 7.2 Hz, 1H), 7.40-7.34 (m, 1H), 7.31 (t, *J* = 7.8 Hz, 1H), 7.17-7.10 (m, 2H), 7.08-7.04 (m, 2H), 7.02 (d, *J* = 7.8 Hz, 1H), 6.77 (d, *J* = 8.4 Hz, 2H), 3.89-3.82 (m, 1H), 3.81 (s, 3H), 3.74 (s, 3H), 3.37-3.27 (m, 2H), 3.18 (dd, *J* = 13.8 Hz, 7.8 Hz, 1H), 3.11 (dd, *J* = 13.8 Hz, 7.8 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 198.68, 159.99, 159.76, 158.06, 149.16, 138.59, 136.13, 136.05, 129.43, 128.48, 123.69, 121.21, 120.74, 119.45, 113.81, 112.27, 55.40, 55.16, 45.28, 44.75, 41.03.

HRMS (ESI): $C_{23}H_{24}NO_3$ $[M+H]^+$ calculated 362.1751, found 362.1750

1-(3-methoxyphenyl)-4-(pyridin-2-yl)-3-(*p*-tolyl)butan-1-one (3ag)



3ag

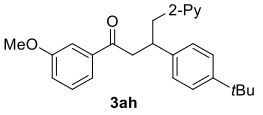
The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.42 in hexane:ethyl acetate = 3:1) resulting in 14.9 mg of yellow oil in 43% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.49 (d, *J* = 4.8 Hz, 1H), 7.54-7.48 (m, 1H), 7.44 (d, *J* = 7.8 Hz, 1H), 7.38-7.35 (m, 1H), 7.30 (t, *J* = 8.4 Hz, 1H), 7.11 (d, *J* = 7.8 Hz, 2H), 7.07-7.01 (m, 5H), 3.93-3.84 (m, 1H), 3.80 (s, 3H), 3.38-3.28 (m, 2H), 3.21-3.09 (m, 2H), 2.26 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 198.62, 160.00, 159.74, 149.15, 140.98, 138.57, 136.16, 135.86, 129.42, 129.12, 127.40, 123.70, 121.23, 120.75, 119.47, 112.25, 55.40, 45.16, 44.60, 41.36, 20.99.

HRMS (ESI): $C_{23}H_{24}NO_2$ $[M+H]^+$ calculated 346.1802, found 346.1800

3-(4-(*tert*-butyl)phenyl)-1-(3-methoxyphenyl)-4-(pyridin-2-yl)butan-1-one (3ah)



3ah

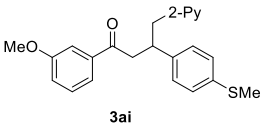
The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.40 in hexane:ethyl acetate = 3:1) resulting in 10.9 mg of yellow oil in 28% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.48 (d, *J* = 5.4 Hz, 1H), 7.56-7.49 (m, 1H), 7.43 (d, *J* = 7.8 Hz, 1H), 7.37-7.33 (m, 1H), 7.29 (t, *J* = 7.8 Hz, 1H), 7.25 (d, *J* = 8.4 Hz, 2H), 7.17 (d, *J* = 7.8 Hz, 2H), 7.09-7.02 (m, 3H), 3.92-3.85 (m, 1H), 3.80 (s, 3H), 3.39-3.27 (m, 2H), 3.23-3.12 (m, 2H), 1.27 (s, 9H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 198.71, 160.10, 159.72, 149.12, 149.11, 141.06, 138.62, 136.16, 129.39, 127.11, 125.29, 123.70, 121.23, 120.73, 119.39, 112.26, 55.39, 45.09, 44.46, 41.17, 34.33, 31.34.

HRMS (ESI): $C_{26}H_{30}NO_2$ $[M+H]^+$ calculated 388.2271, found 388.2274

1-(3-methoxyphenyl)-3-(4-(methylthio)phenyl)-4-(pyridin-2-yl)butan-1-one (3ai)



3ai

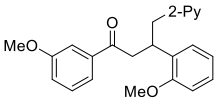
The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.34 in hexane:ethyl acetate = 3:1) resulting in 19.7 mg of yellow oil in 52% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.52-8.46 (m, 1H), 7.53-7.48 (m, 1H), 7.44 (d, *J* = 7.2 Hz, 1H), 7.38-7.35 (m, 1H), 7.31 (t, *J* = 8.4 Hz, 1H), 7.16-7.09 (m, 4H), 7.08-7.04 (m, 2H), 7.02 (d, *J* = 7.8 Hz, 1H), 3.91-3.85 (m, 1H), 3.81 (s, 3H), 3.38-3.28 (m, 2H), 3.19 (dd, *J* = 13.2 Hz, 7.8 Hz, 1H), 3.11 (dd, *J* = 13.8 Hz, 7.8 Hz, 1H), 2.42 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 198.41, 159.78, 159.74, 149.21, 141.06, 138.50, 136.20, 136.03, 129.46, 128.11, 126.94, 123.69, 121.30, 120.70, 119.50, 112.28, 55.41, 45.01, 44.46, 41.22, 16.01.

HRMS (ESI): $C_{23}H_{24}NO_2S$ $[M+H]^+$ calculated 378.1522, found 378.1521

3-(2-methoxyphenyl)-1-(3-methoxyphenyl)-4-(pyridin-2-yl)butan-1-one (3aj)



3aj

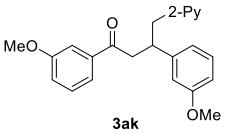
The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.33 in hexane:ethyl acetate = 3:1) resulting in 18.8 mg of yellow oil in 52% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.49-8.44 (m, 1H), 7.52-7.43 (m, 2H), 7.42-7.36 (m, 1H), 7.30 (t, *J* = 7.8 Hz, 1H), 7.17-7.10 (m, 2H), 7.08-7.00 (m, 3H), 6.83 (t, *J* = 7.2 Hz, 1H), 6.80 (d, *J* = 7.8 Hz, 1H), 4.25-4.14 (m, 1H), 3.80 (s, 3H), 3.77 (s, 3H), 3.45 (dd, *J* = 16.2 Hz, 7.8 Hz, 1H), 3.32 (dd, *J* = 16.2 Hz, 6.0 Hz, 1H), 3.27-3.14 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 199.06, 160.56, 159.70, 157.26, 148.98, 138.73, 135.98, 131.80, 129.33, 128.46, 127.40, 123.60, 121.04, 120.78, 120.51, 119.27, 112.31, 110.72, 55.39, 55.27, 43.08, 43.02, 36.72.

HRMS (ESI): $C_{23}H_{24}NO_3$ $[M+H]^+$ calculated 362.1751, found 362.1750

1,3-bis(3-methoxyphenyl)-4-(pyridin-2-yl)butan-1-one (3ak)



3ak

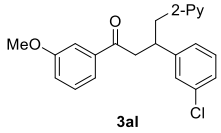
The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.19 in hexane:ethyl acetate = 3:1) resulting in 16.7 mg of yellow oil in 46% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.52-8.45 (m, 1H), 7.54-7.48 (m, 1H), 7.45 (d, *J* = 7.2 Hz, 1H), 7.39-7.35 (m, 1H), 7.31 (t, *J* = 8.4 Hz, 1H), 7.15 (t, *J* = 7.8 Hz, 1H), 7.09-7.02 (m, 3H), 6.83 (d, *J* = 7.8 Hz, 1H), 6.77-6.73 (m, 1H), 6.70-6.67 (m, 1H), 3.94-3.86 (m, 1H), 3.81 (s, 3H), 3.73 (s, 3H), 3.41-3.28 (m, 2H), 3.22-3.09 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 198.46, 159.85, 159.77, 159.58, 149.18, 145.75, 138.58, 136.15, 129.43, 129.37, 123.71, 121.25, 120.71, 119.87, 119.46, 113.51, 112.28, 111.77, 55.40, 55.11, 45.04, 44.38, 41.74.

HRMS (ESI): $C_{23}H_{24}NO_3$ $[M+H]^+$ calculated 362.1751, found 362.1751

3-(3-chlorophenyl)-1-(3-methoxyphenyl)-4-(pyridin-2-yl)butan-1-one (3al)



3al

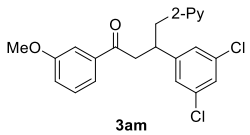
The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.32 in hexane:ethyl acetate = 3:1) resulting in 26.4 mg of yellow oil in 72% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.50 (d, *J* = 4.8 Hz, 1H), 7.52 (t, *J* = 7.8 Hz, 1H), 7.46 (d, *J* = 7.8 Hz, 1H), 7.38 (s, 1H), 7.32 (t, *J* = 8.4 Hz, 1H), 7.21 (s, 1H), 7.17-7.13 (m, 1H), 7.13-7.09 (m, 2H), 7.09-7.06 (m, 2H), 7.02 (d, *J* = 7.8 Hz, 1H), 3.94-3.87 (m, 1H), 3.82 (s, 3H), 3.36 (d, *J* = 7.2 Hz, 2H), 3.19 (dd, *J* = 13.8 Hz, 7.8 Hz, 1H), 3.10 (dd, *J* = 13.2 Hz, 7.2 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 198.04, 159.81, 159.37, 149.29, 146.21, 138.38, 136.25, 134.16, 129.64, 129.50, 127.73, 126.65, 125.95, 123.68, 121.41, 120.68, 119.62, 112.25, 55.42, 44.85, 44.17, 41.35.

HRMS (ESI): $C_{22}H_{21}ClNO_2$ $[M+H]^+$ calculated 366.1255, found 366.1254

3-(3,5-dichlorophenyl)-1-(3-methoxyphenyl)-4-(pyridin-2-yl)butan-1-one (3am)



3am

The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.48 in hexane:ethyl acetate = 3:1) resulting in 14.4 mg of yellow oil in 36% yield.

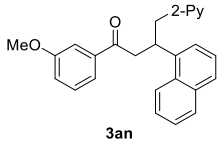
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.54-8.46 (m, 1H), 7.57-7.51 (m, 1H), 7.46 (d, *J* = 7.2 Hz, 1H), 7.40-7.36 (m, 1H), 7.34 (t, *J* = 7.8 Hz, 1H), 7.15-7.13 (m, 1H), 7.12-7.07 (m, 4H), 7.03 (d, *J* = 7.8 Hz, 1H), 3.96-3.87 (m, 1H), 3.82 (s, 3H), 3.41-3.28 (m, 2H), 3.17 (dd, *J* = 13.8 Hz, 7.8 Hz, 1H), 3.08 (dd, *J* = 13.8 Hz, 7.2 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 197.60, 159.86, 158.91, 149.38, 147.68, 138.21, 136.36, 134.78, 129.56, 126.71, 126.30, 123.67, 121.58, 120.65, 119.74, 112.25, 55.44, 44.58, 43.87, 41.08.

HRMS (ESI): $C_{22}H_{20}Cl_2NO_2$ $[M+H]^+$ calculated 400.0866, found 400.0865

.

1-(3-methoxyphenyl)-3-(naphthalen-1-yl)-4-(pyridin-2-yl)butan-1-one (3an)



3an

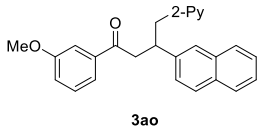
The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.32 in hexane:ethyl acetate = 3:1) resulting in 16.8 mg of yellow oil in 44% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.49-8.44 (m, 1H), 8.27 (d, *J* = 8.4 Hz, 1H), 7.85-7.79 (m, 1H), 7.69 (d, *J* = 7.8 Hz, 1H), 7.52-7.48 (m, 1H), 7.48-7.42 (m, 4H), 7.39 (t, *J* = 7.2 Hz, 1H), 7.37-7.34 (m, 1H), 7.30 (t, *J* = 7.8 Hz, 1H), 7.07-7.04 (m, 1H), 7.04-6.99 (m, 2H), 4.96-4.85 (m, 1H), 3.78 (s, 3H), 3.58-3.47 (m, 2H), 3.38-3.27 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 198.53, 159.92, 159.76, 149.16, 140.30, 138.57, 136.08, 134.01, 131.60, 129.42, 128.84, 126.97, 126.06, 125.44, 125.33, 123.60, 123.29, 121.23, 120.72, 119.55, 112.22, 55.38, 44.42, 44.20.

HRMS (ESI): $C_{26}H_{24}NO_2$ $[M+H]^+$ calculated 382.1802, found 382.1799

1-(3-methoxyphenyl)-3-(naphthalen-2-yl)-4-(pyridin-2-yl)butan-1-one (3ao)



3ao

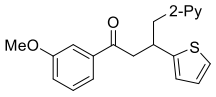
The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.36 in hexane:ethyl acetate = 3:1) resulting in 18.0 mg of yellow oil in 47% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.53-8.47 (m, 1H), 7.78-7.70 (m, 3H), 7.64 (s, 1H), 7.48-7.44 (m, 2H), 7.43-7.38 (m, 3H), 7.38-7.35 (m, 1H), 7.30 (t, *J* = 7.8 Hz, 1H), 7.07-7.01 (m, 3H), 4.14-4.08 (m, 1H), 3.78 (s, 3H), 3.52-3.39 (m, 2H), 3.35-3.21 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 198.50, 159.84, 159.79, 149.22, 143.19, 140.90, 139.24, 138.55, 136.20, 129.46, 128.67, 127.99, 127.11, 127.05, 126.95, 123.72, 121.30, 120.74, 119.50, 112.30, 55.41, 45.08, 44.45, 41.39.

HRMS (ESI): $C_{26}H_{24}NO_2$ $[M+H]^+$ calculated 382.1802, found 382.1800

1-(3-methoxyphenyl)-4-(pyridin-2-yl)-3-(thiophen-2-yl)butan-1-one (3ap)



3ap

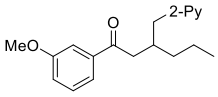
The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.30 in hexane:ethyl acetate = 3:1) resulting in 13.5 mg of yellow oil in 40% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.51 (d, *J* = 4.8 Hz, 1H), 7.56-7.51 (m, 1H), 7.48 (d, *J* = 7.8 Hz, 1H), 7.43-7.39 (m, 1H), 7.33 (t, *J* = 8.4 Hz, 1H), 7.11-7.05 (m, 4H), 6.85-6.82 (m, 1H), 6.79-6.76 (m, 1H), 4.32-4.24 (m, 1H), 3.83 (s, 3H), 3.43-3.31 (m, 2H), 3.28-3.17 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 198.05, 159.80, 159.41, 149.26, 147.80, 138.45, 136.23, 129.49, 126.54, 124.17, 123.68, 123.10, 121.41, 120.74, 119.60, 112.29, 55.43, 45.83, 45.40, 36.96.

HRMS (ESI): $C_{20}H_{20}NO_2S$ $[M+H]^+$ calculated 338.1209, found 338.1208

1-(3-methoxyphenyl)-3-(pyridin-2-ylmethyl)hexan-1-one (3aq)



3aq

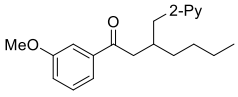
The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.63 in hexane:ethyl acetate = 3:1) resulting in 15.5 mg of yellow oil in 52% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.52-8.48 (m, 1H), 7.60-7.55 (m, 1H), 7.45 (d, *J* = 7.8 Hz, 1H), 7.43-7.40 (m, 1H), 7.32 (t, *J* = 7.8 Hz, 1H), 7.19 (d, *J* = 7.8 Hz, 1H), 7.11-7.05 (m, 2H), 3.84 (s, 3H), 2.99-2.90 (m, 2H), 2.90-2.73 (m, 2H), 2.67-2.57 (m, 1H), 1.43-1.34 (m, 4H), 0.95-0.81 (m, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 200.01, 160.94, 159.79, 149.14, 138.76, 136.20, 129.45, 123.68, 121.09, 120.76, 119.35, 112.31, 55.41, 42.93, 42.79, 36.61, 35.35, 19.98, 14.25.

HRMS (ESI): $C_{19}H_{24}NO_2$ $[M+H]^+$ calculated 298.1802, found 298.1801

1-(3-methoxyphenyl)-3-(pyridin-2-ylmethyl)heptan-1-one (3ar)



3ar

The title compound was prepared according to the general procedure as described, silica gel flash column chromatography was performed using hexanes and ethyl acetate (10:1) (R*f* = 0.62 in hexane:ethyl acetate = 3:1) resulting in 16.5 mg of yellow oil in 53% yield.

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.50 (d, *J* = 4.8 Hz, 1H), 7.60-7.55 (m, 1H), 7.45 (d, *J* = 7.8 Hz, 1H), 7.42 (s, 1H), 7.32 (t, *J* = 7.8 Hz, 1H), 7.19 (d, *J* = 7.8 Hz, 1H), 7.12-7.05 (m, 2H), 3.84 (s, 3H), 2.99-2.89 (m, 2H), 2.89-2.72 (m, 2H), 2.64-2.58 (m, 1H), 1.43-1.37 (m, 2H), 1.37-1.30 (m, 2H), 1.30-1.23 (m, 2H), 0.85 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 200.02, 160.94, 159.78, 149.13, 138.77, 136.20, 129.45, 123.69, 121.09, 120.76, 119.35, 112.30, 55.41, 42.92, 42.80, 35.51, 33.95, 29.00, 22.83, 14.00.

HRMS (ESI): $C_{20}H_{26}NO_2$ $[M+H]^+$ calculated 312.1958, found 312.1956

5. References

[1] T. Niwa, H. Yorimitsu and K. Oshima, *Angew. Chem. Int. Ed.*, 2007, **46**, 2643.

[2] H. Komai, T. Yoshino, S. Matsunaga and M. Kanai, *Org. Lett.* 2011, **13**, 1706.

6. NMR spectra of the products

HCM-1H.74.fid
HCM-177a

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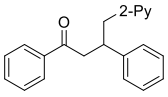
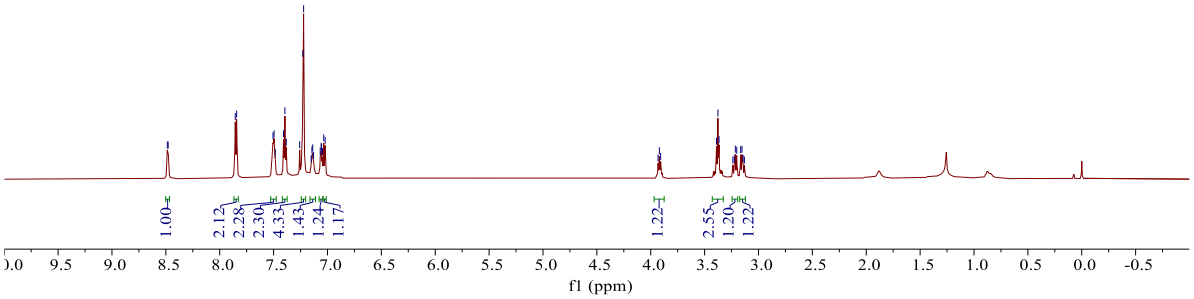


3aa



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HCM-134

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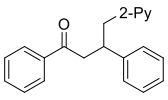

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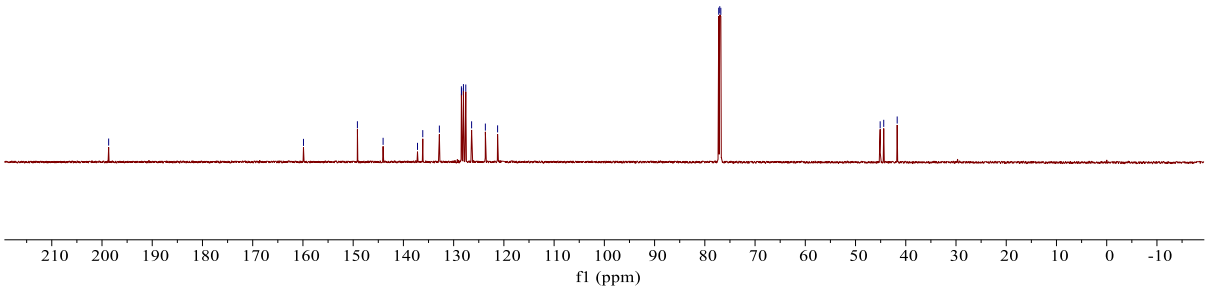

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HCM-187b

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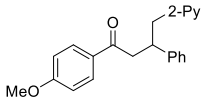
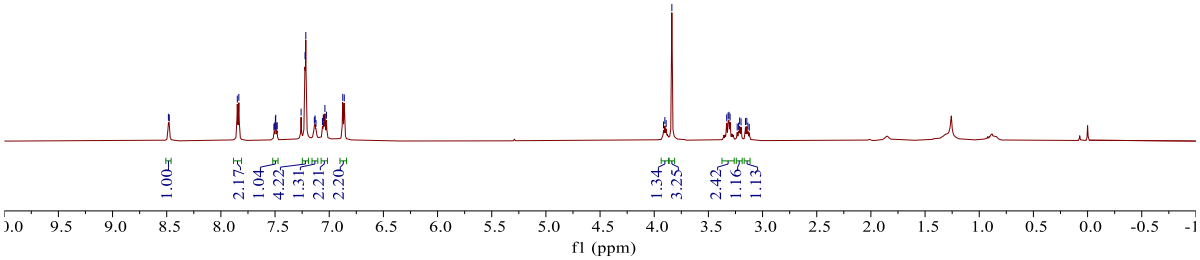


3ba



HCM-13C.3.fid
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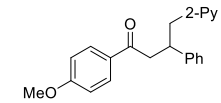

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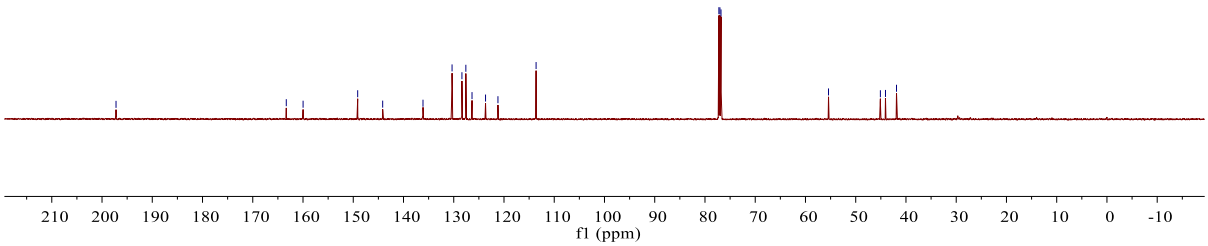

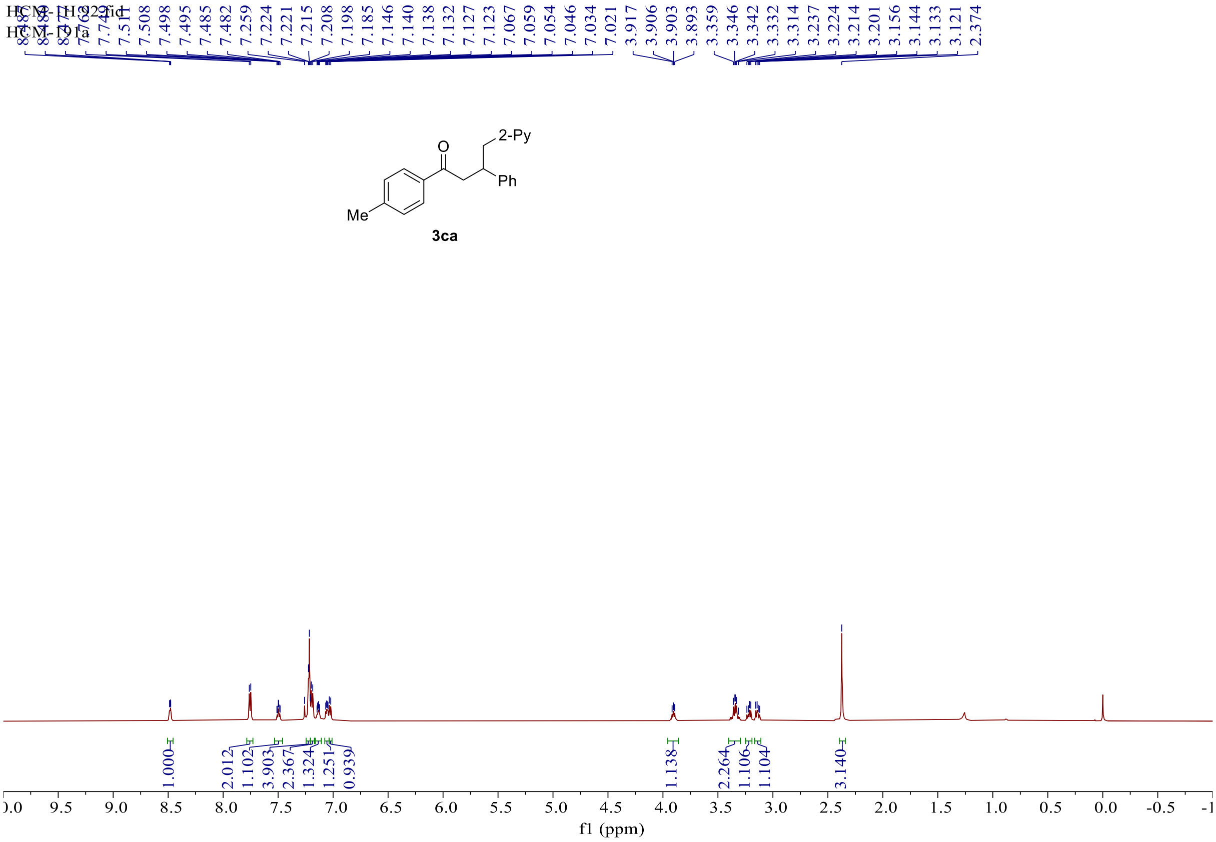
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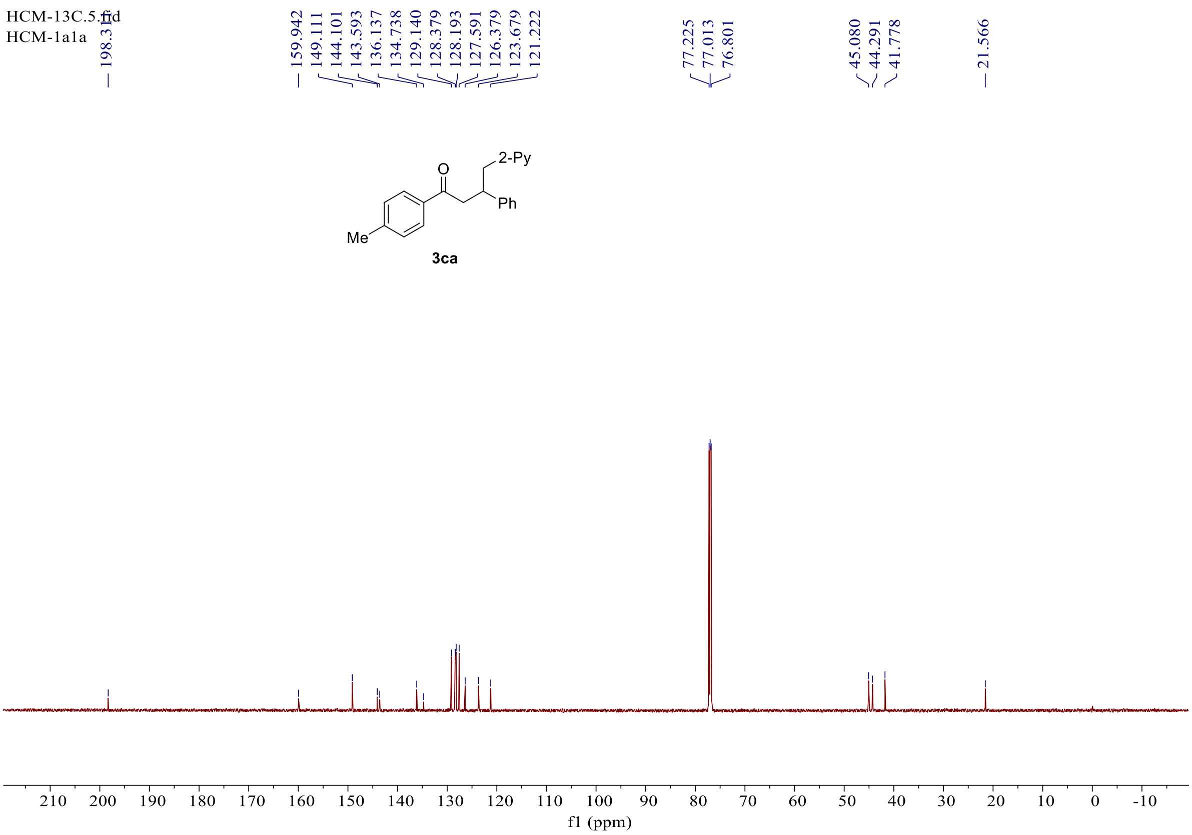
f1 (ppm)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76.801
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O
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Ph
Me
3ca
f1 (ppm)

HCM-1H.76.fid
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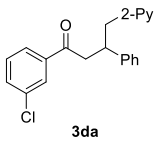

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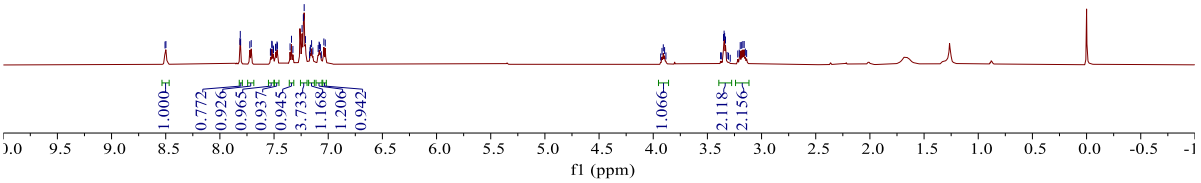
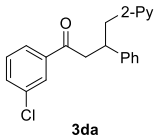


HCM-13C.37.fid
HCM-196b



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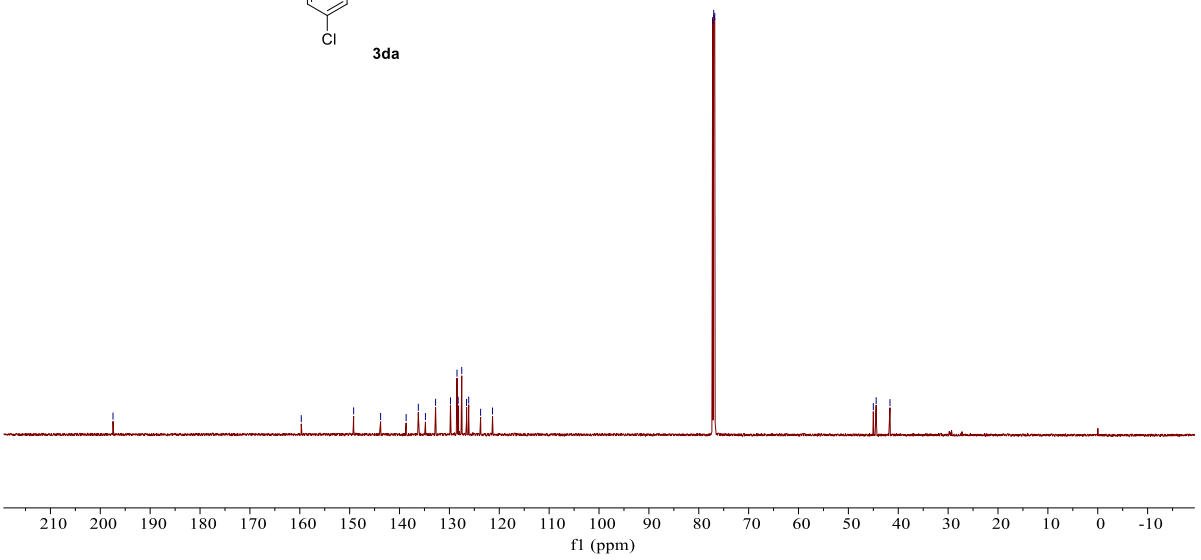

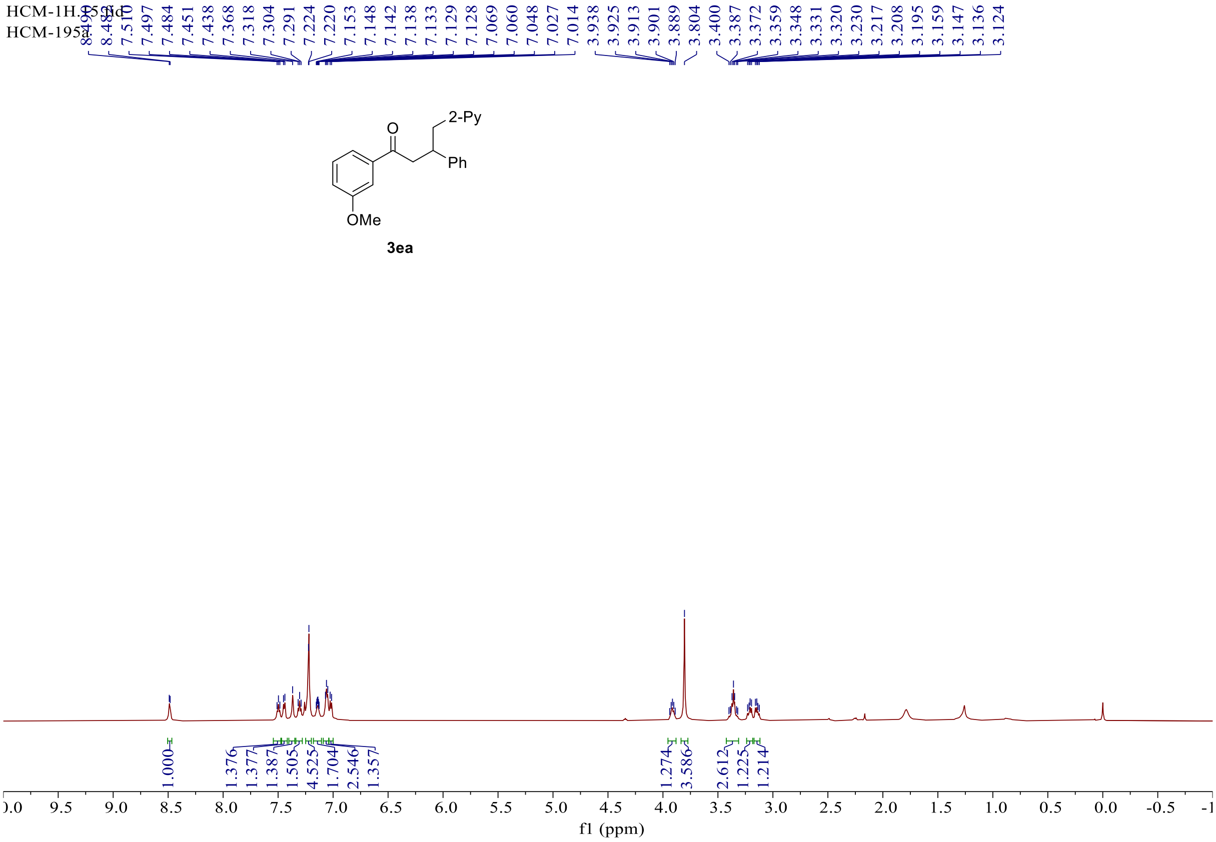
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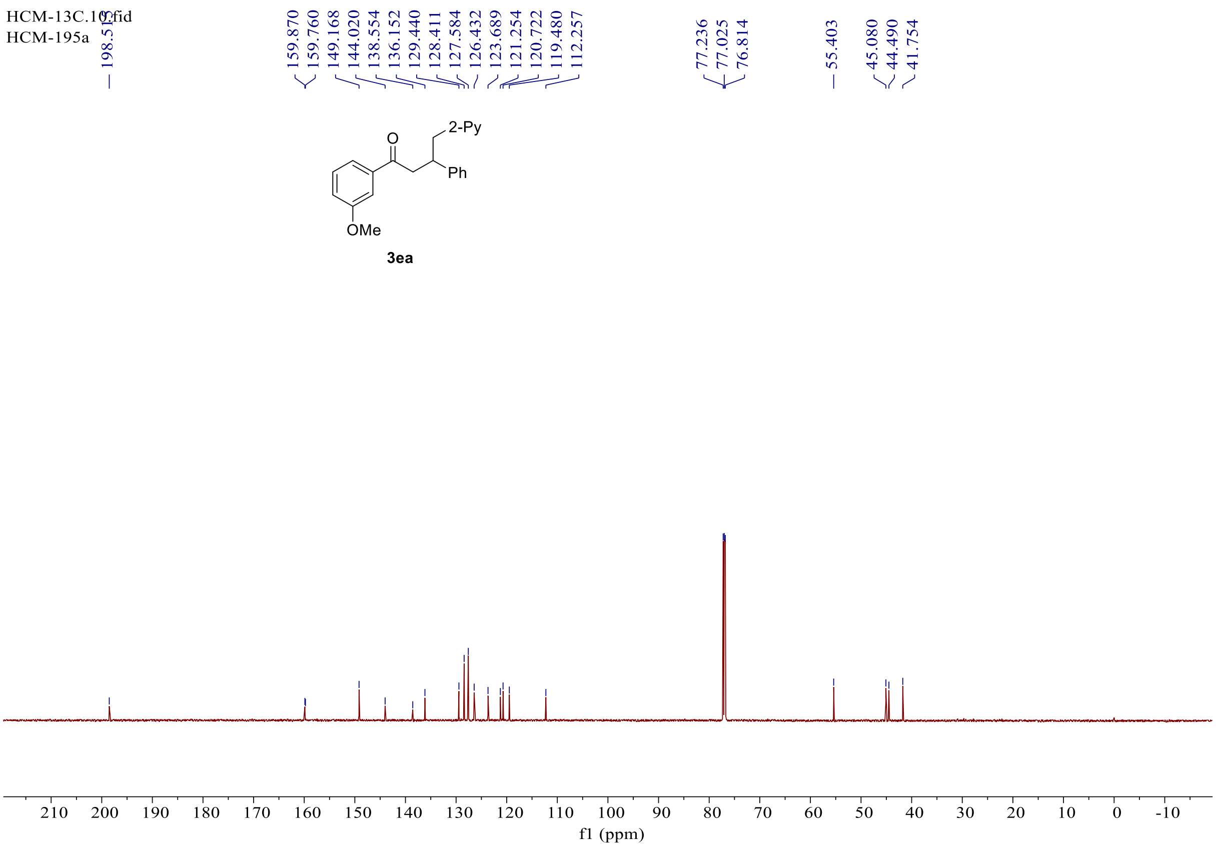
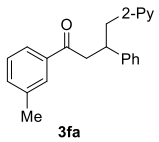
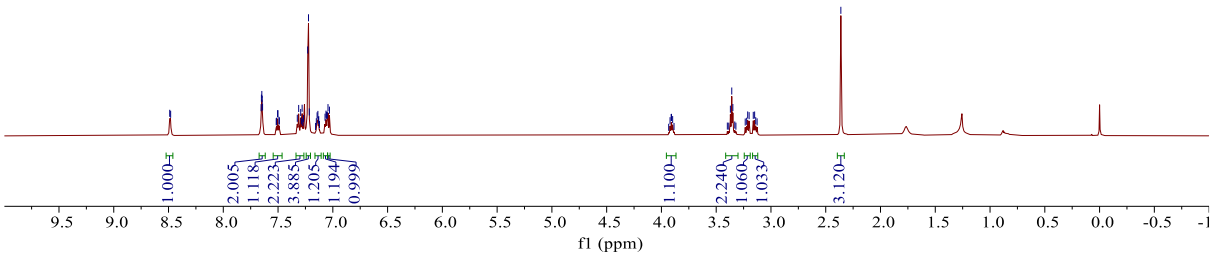
7.291
7.224
7.220
7.153
7.148
7.142
7.138
7.133
7.129
7.128
7.069
7.060
7.048
7.027
7.014
3.938
3.925
3.913
3.901
3.889
3.804
3.400
3.387
3.372
3.359
3.348
3.331
3.320
3.230
3.217
3.208
3.195
3.159
3.147
3.136
3.124
2-Py
O
Ph
OMe
3ea
1.000
1.376
1.377
1.387
1.505
4.525
1.704
2.546
1.357
1.274
3.586
2.612
1.225
1.214
f1 (ppm)


HCM-195a
159.870
159.760
149.168
144.020
138.554
136.152
129.440
128.411
127.584
126.432
123.689
121.254
120.722
119.480
112.257
77.236
77.025
76.814
55.403
45.080
44.490
41.754
2-Py
O
Ph
OMe
3ea
f1 (ppm)

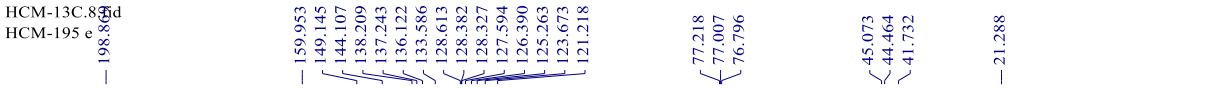
HCM-1H.3.fid
HCM-195e

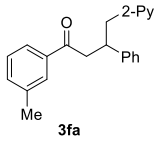


3fa

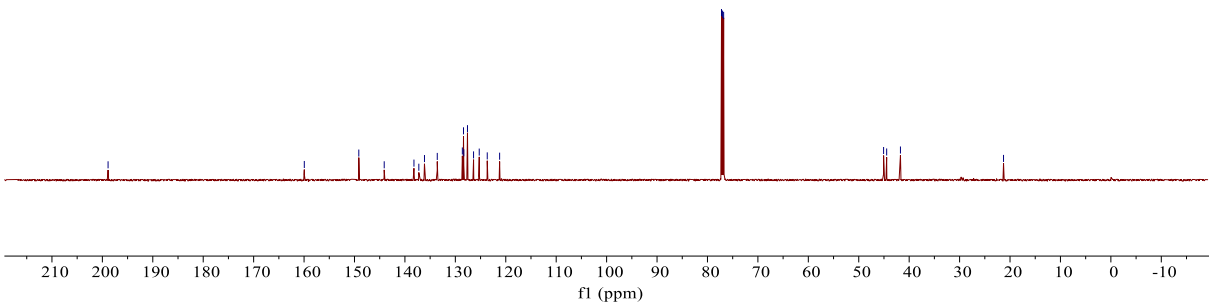


HCM-13C.8.fid
HCM-195 e



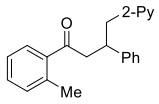


3fa

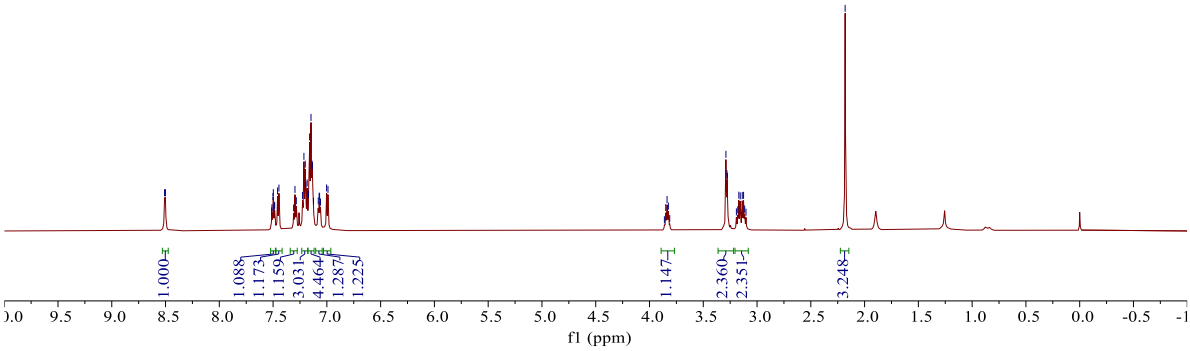


HCM-1H.45.fid
HCM-195B

8.509 8.501 7.514 7.511 7.501 7.498 7.488 7.485 7.458 7.446 7.309 7.296 7.284 7.226 7.213 7.201 7.196 7.183 7.171 7.160 7.147 7.137 7.134 7.125 7.080 7.072 7.068 7.059 7.002 6.990 3.862 3.849 3.837 3.824 3.812 3.289 3.280 3.275 3.192 3.179 3.169 3.156 3.138 3.125 3.115 3.103 2.181

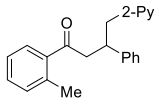


3ga

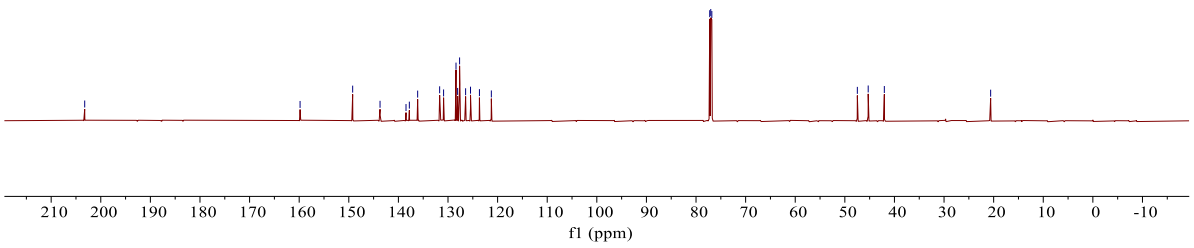


HCM-13C.25.fid
HCM-195B

203.340 159.821 149.220 143.703 138.467 137.793 136.119 131.686 130.892 128.387 128.062 127.664 126.451 125.462 123.664 121.262 77.239 77.027 76.816 47.479 45.291 42.040 20.608

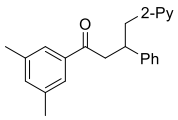


3ga

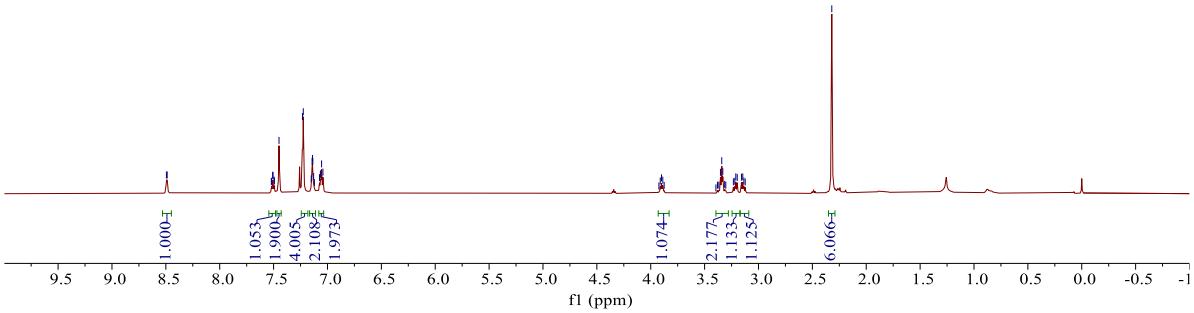


HCM-1H.98.fid
HCM-207a

8.495 8.488 7.521 7.518 7.509 7.505 7.496 7.493 7.450 7.232 7.224 7.150 7.143 7.139 7.136 7.129 7.121 7.074 7.066 7.062 7.055 7.042 3.924 3.912 3.900 3.888 3.875 3.395 3.381 3.368 3.353 3.341 3.331 3.314 3.304 3.233 3.220 3.210 3.197 3.158 3.146 3.136 3.123 2.320

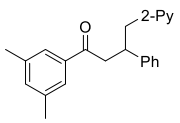


3ha

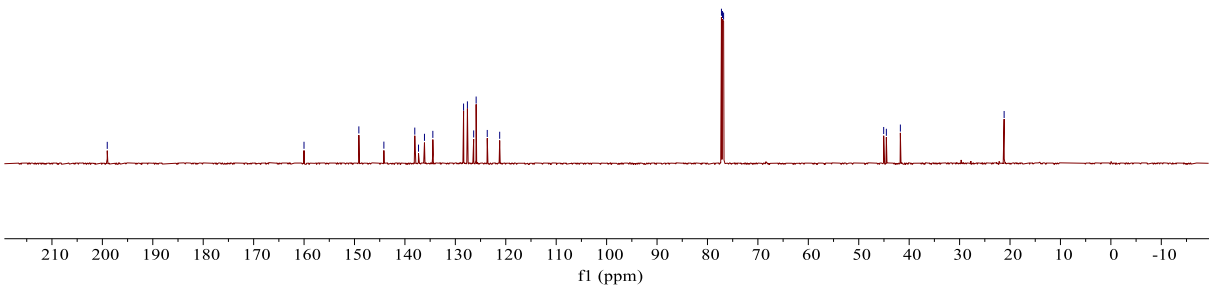


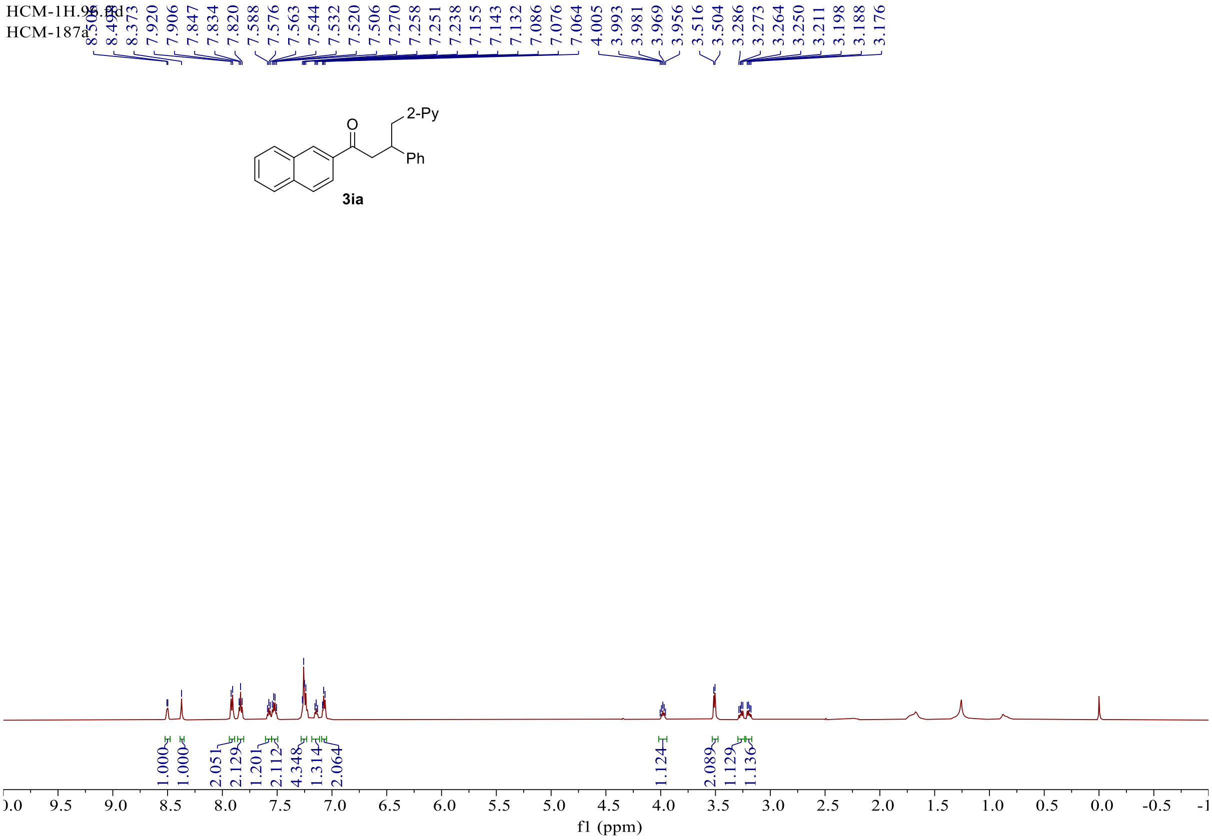
HCM-13C.7[illegible]fid
HCM-207a

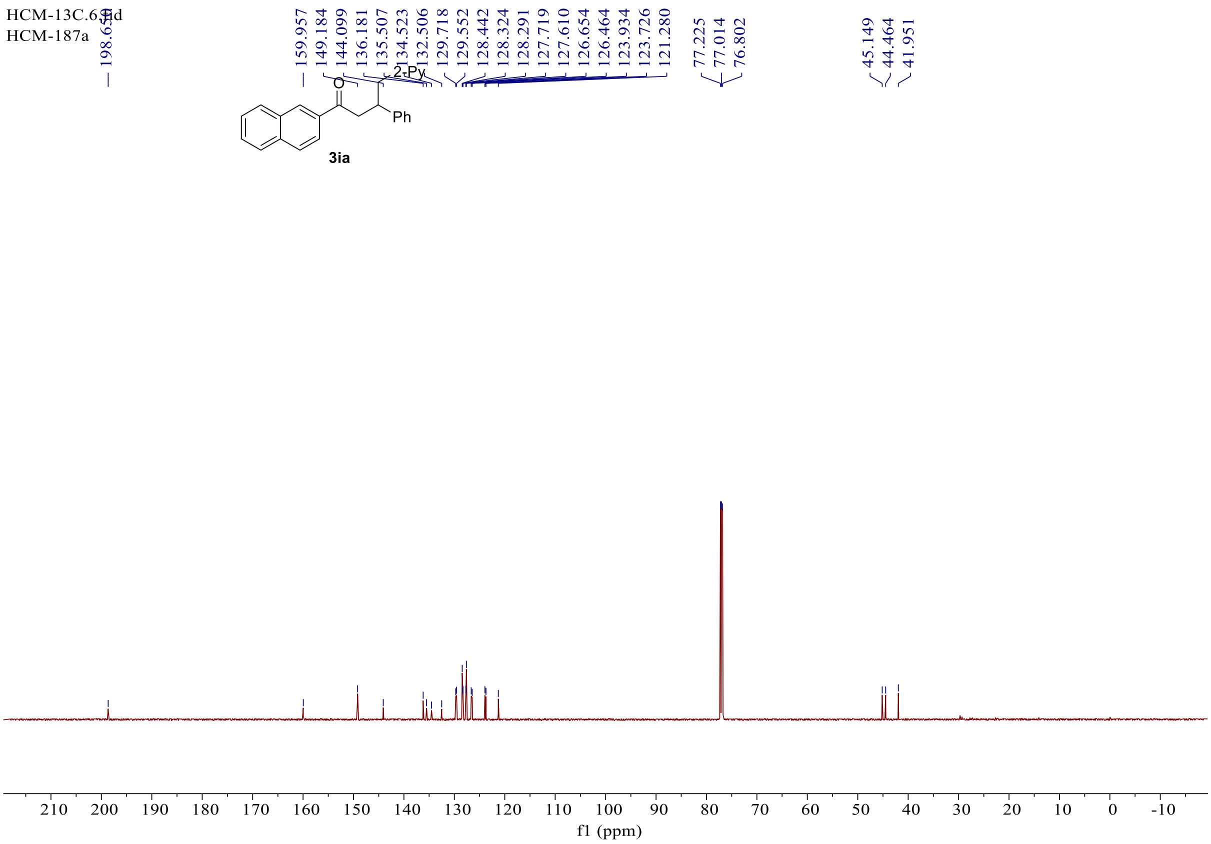
199.022 160.014 149.136 144.174 138.045 137.308 136.123 134.464 128.369 127.608 126.366 125.874 123.672 121.207 77.226 77.015 76.803 45.054 44.524 41.747 21.170

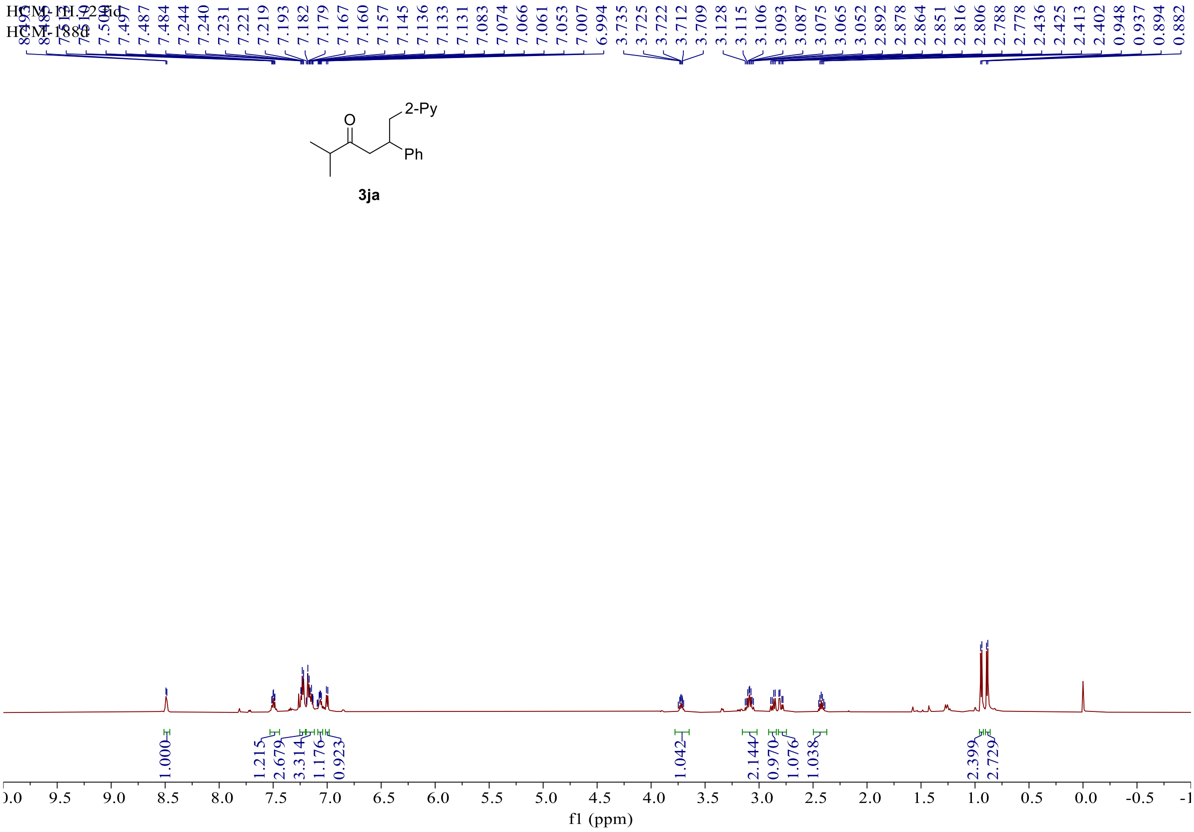


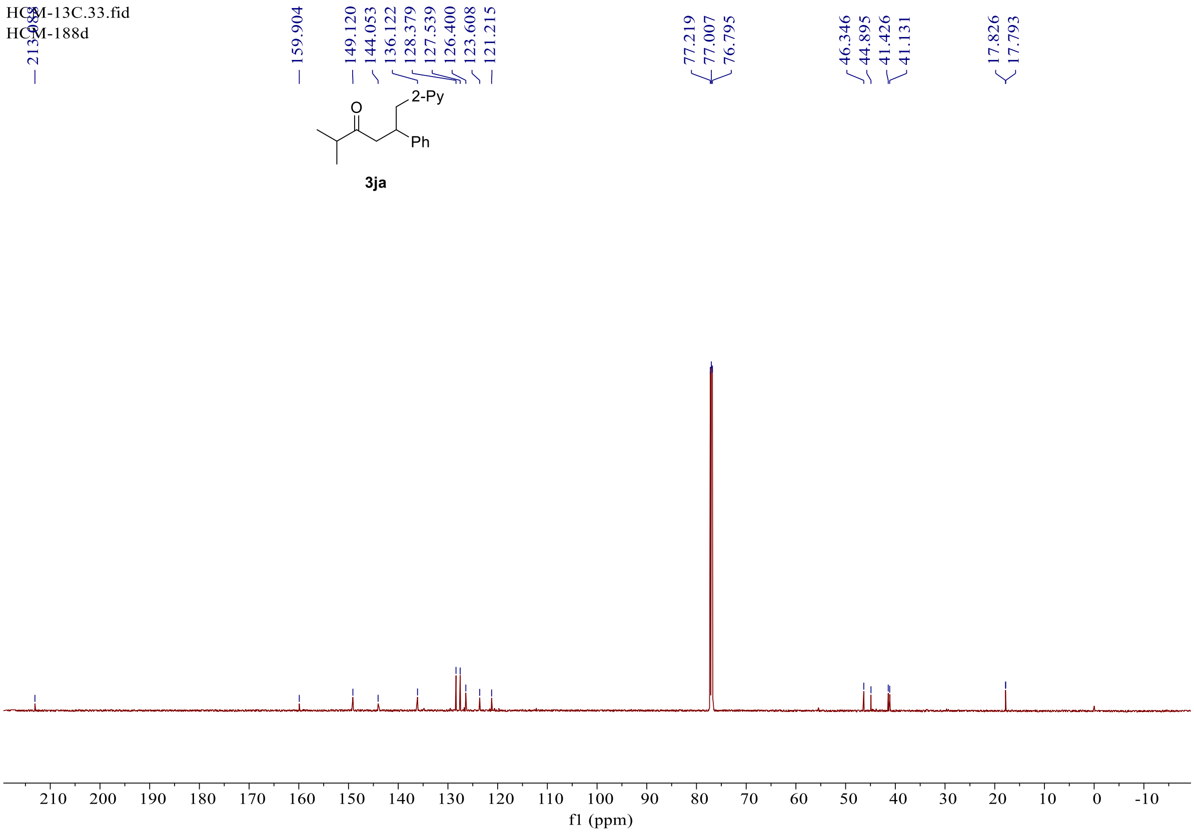
3ha

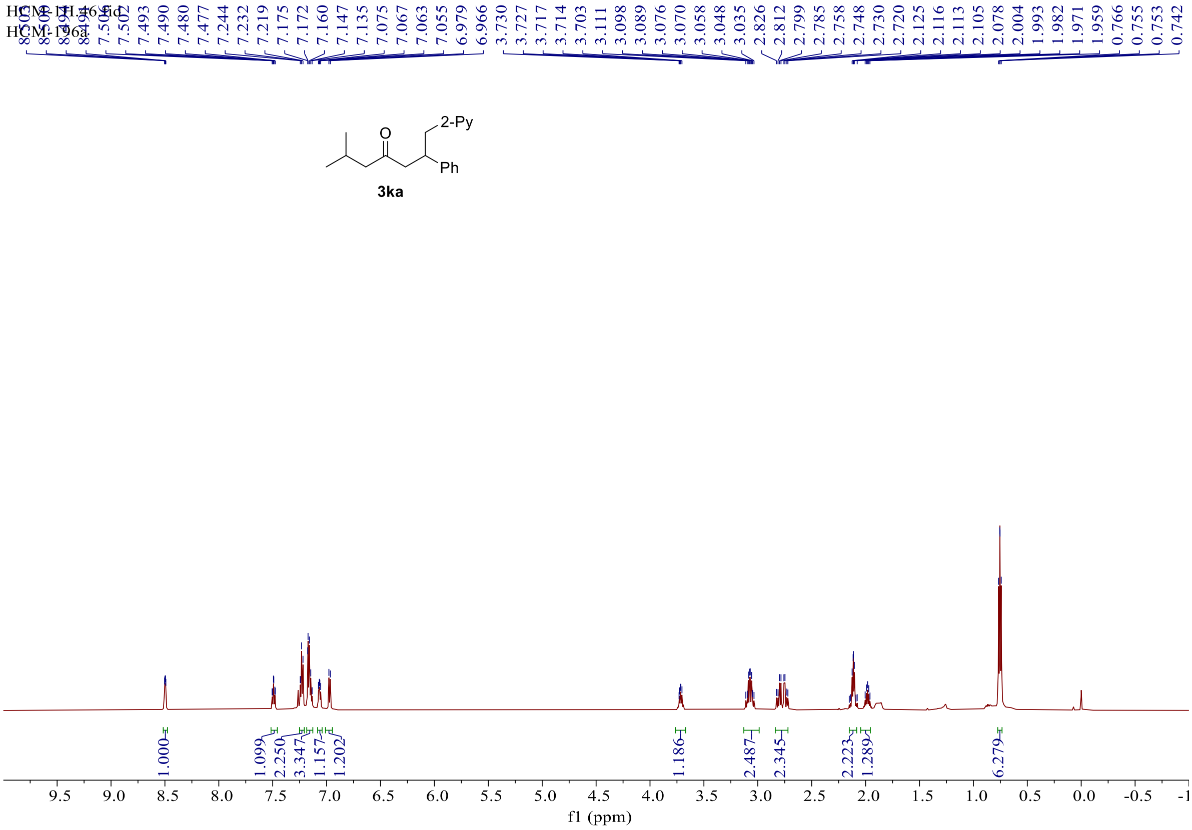


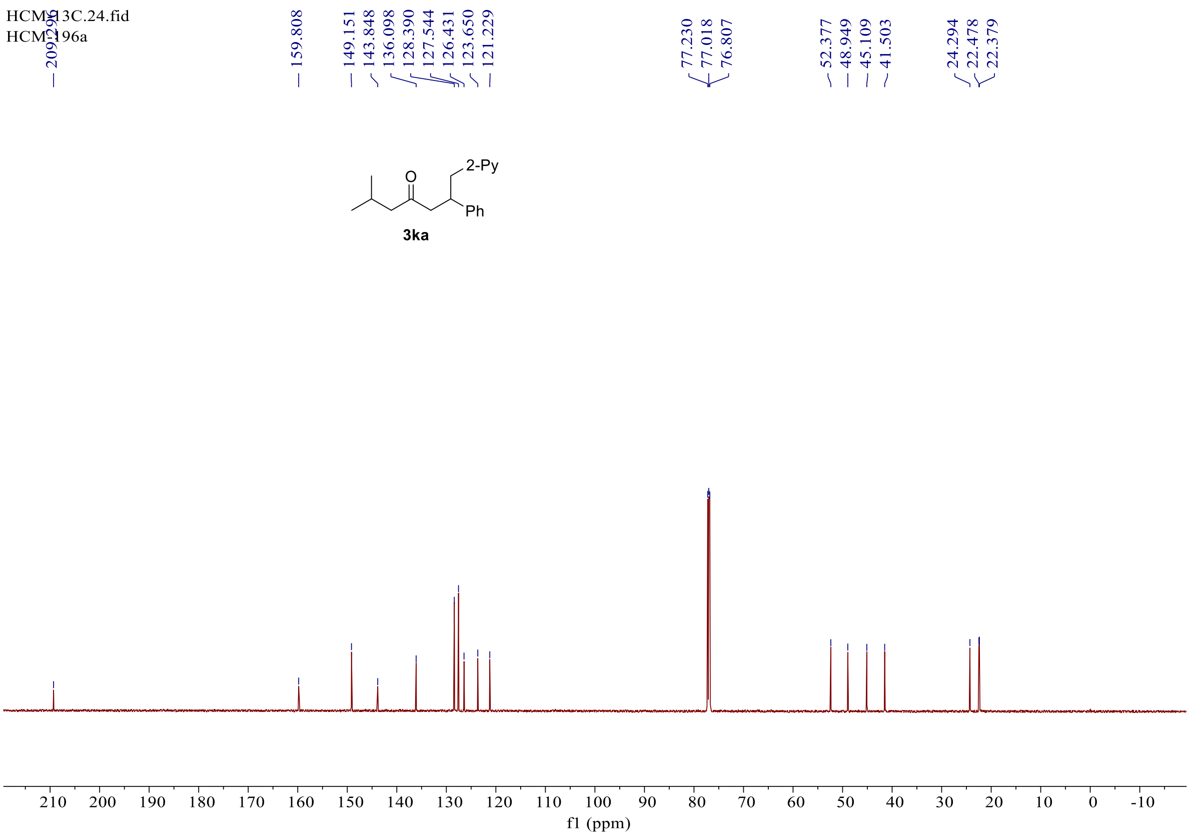

HCM-1H.96.fid
HCM-187a
8.373
7.920
7.906
7.847
7.834
7.820
7.588
7.576
7.563
7.544
7.532
7.520
7.506
7.270
7.258
7.251
7.238
7.155
7.143
7.132
7.086
7.076
7.064
4.005
3.993
3.981
3.969
3.956
3.516
3.504
3.286
3.273
3.264
3.250
3.211
3.198
3.188
3.176
O
2-Py
Ph
3ia
1.000
1.000
2.051
2.129
1.201
2.112
4.348
1.314
2.064
1.124
2.089
1.129
1.136
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
f1 (ppm)


HCM-13C.6.fid
HCM-187a
198.6
159.957
149.184
144.099
136.181
135.507
134.523
132.506
129.718
129.552
128.442
128.324
128.291
127.719
127.610
126.654
126.464
123.934
123.726
121.280
77.225
77.014
76.802
45.149
44.464
41.951
O
2-Py
Ph
3ia
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


HCM-1H.72.fid
HCM-188d
7.487 7.484 7.244 7.240 7.231 7.221 7.219 7.193 7.182 7.179 7.167 7.160 7.157 7.145 7.136 7.133 7.131 7.083 7.074 7.066 7.061 7.053 7.007 6.994 3.735 3.725 3.722 3.712 3.709 3.128 3.115 3.106 3.093 3.087 3.075 3.065 3.052 2.892 2.878 2.864 2.851 2.816 2.806 2.788 2.778 2.436 2.425 2.413 2.402 0.948 0.937 0.894 0.882
O
2-Py
Ph
3ja
1.000 1.215 2.679 3.314 1.176 0.923 1.042 2.144 0.970 1.076 1.038 2.399 2.729
f1 (ppm)

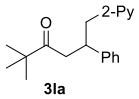

HCM-13C.33.fid
HCM-188d
213.08
159.904 149.120 144.053 136.122 128.379 127.539 126.400 123.608 121.215
77.219 77.007 76.795
46.346 44.895 41.426 41.131
17.826 17.793
O
2-Py
Ph
3ja
f1 (ppm)


HCM-1H.46.fid
HCM-196a
7.490
7.480
7.477
7.244
7.232
7.219
7.175
7.172
7.160
7.147
7.135
7.075
7.067
7.063
7.055
6.979
6.966
3.730
3.727
3.717
3.714
3.703
3.111
3.098
3.089
3.076
3.070
3.058
3.048
3.035
2.826
2.812
2.799
2.785
2.758
2.748
2.730
2.720
2.125
2.116
2.113
2.105
2.078
2.004
1.993
1.982
1.971
1.959
0.766
0.755
0.753
0.742
O
2-Py
Ph
3ka
1.000
1.099
2.250
3.347
1.157
1.202
1.186
2.487
2.345
2.223
1.289
6.279
f1 (ppm)


HCM-13C.24.fid
HCM-196a
159.808
149.151
143.848
136.098
128.390
127.544
126.431
123.650
121.229
77.230
77.018
76.807
52.377
48.949
45.109
41.503
24.294
22.478
22.379
O
2-Py
Ph
3ka
f1 (ppm)

HCM-1H.9.fid
HCM-208 e

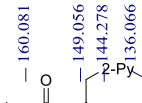
8.483 8.475 7.509 7.506 7.497 7.494 7.484 7.481 7.233 7.221 7.208 7.182 7.179 7.168 7.145 7.133 7.121 7.063 7.055 7.051 7.043 7.025 7.012 3.767 3.754 3.745 3.741 3.731 3.719 3.143 3.130 3.120 3.107 3.086 3.073 3.064 3.051 2.975 2.961 2.946 2.932 2.801 2.792 2.772 2.763 0.975



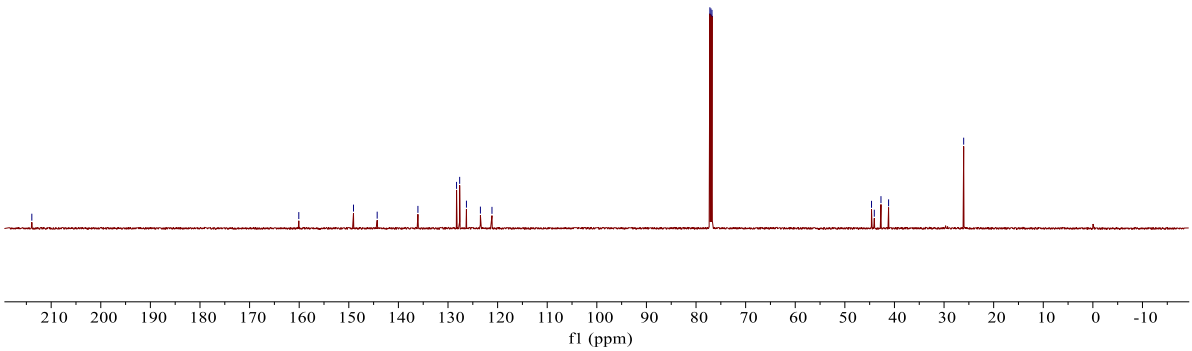
3la

HCM-13C.9.fid
HCM-208e

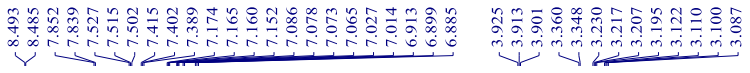
213.010 160.081 149.056 144.278 136.066 128.279 127.649 126.285 123.467 121.138 77.207 76.995 76.784 44.621 44.064 42.718 41.182 26.044

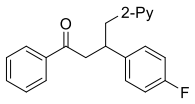


3la

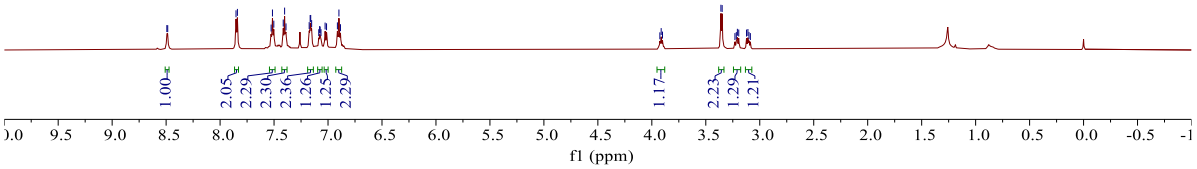


HCM-1H.64.fid
HCM-164a

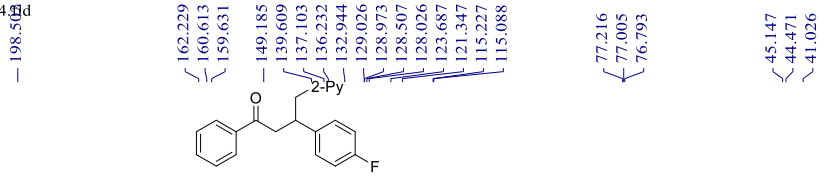




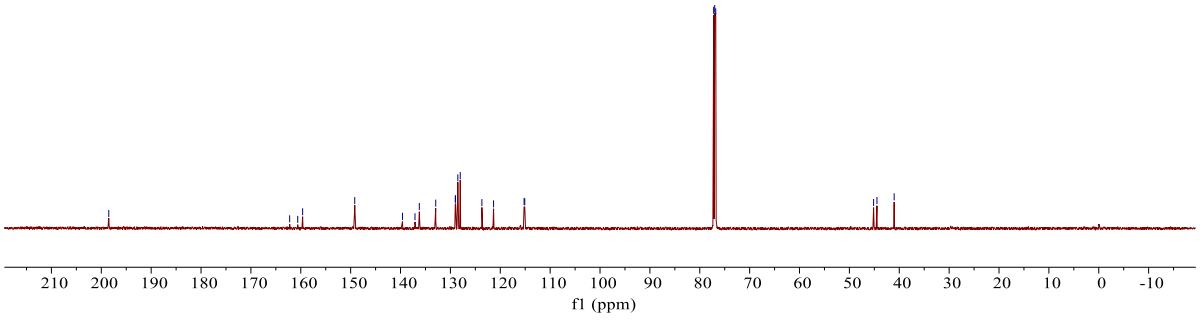
3ab



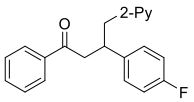
HCM-13C.43.fid
HCM-164a



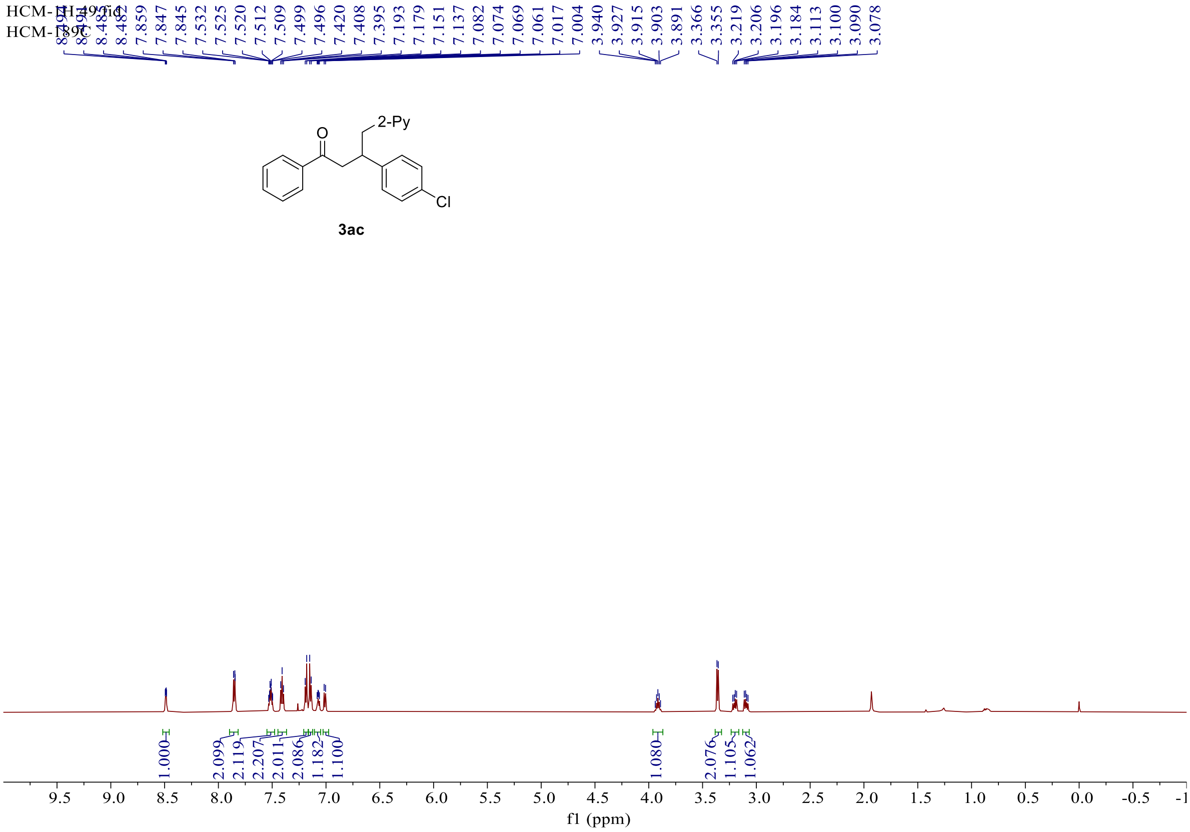
3ab



HCM-19F
HCM-164A.1.fid



3ab


HCM-1H.49.fid
HCM-189c
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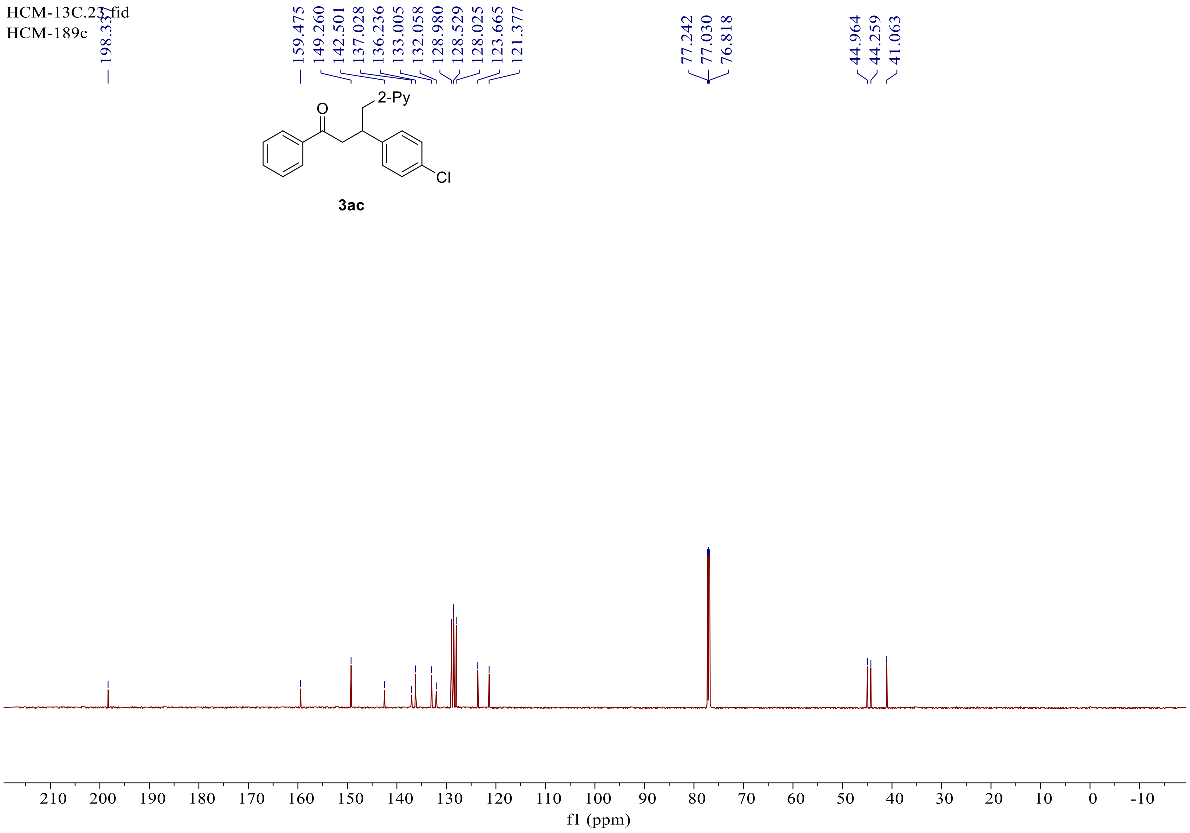
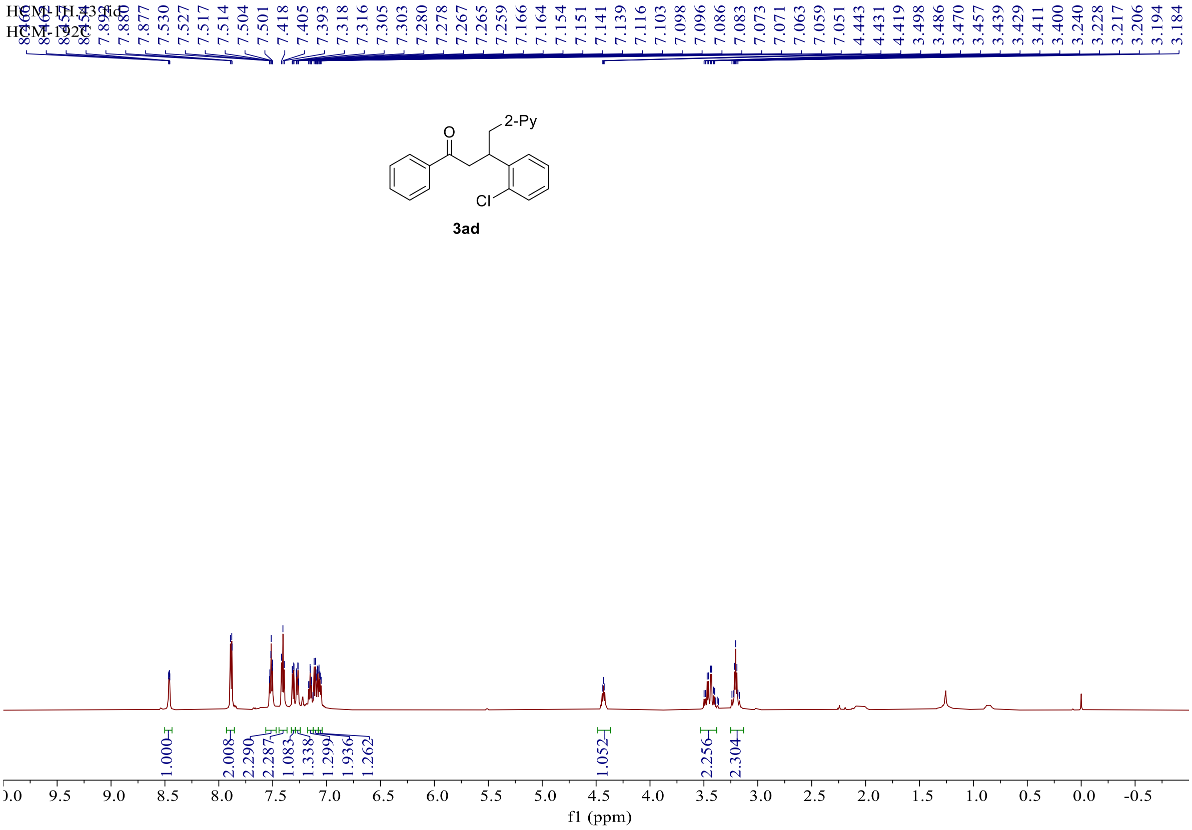
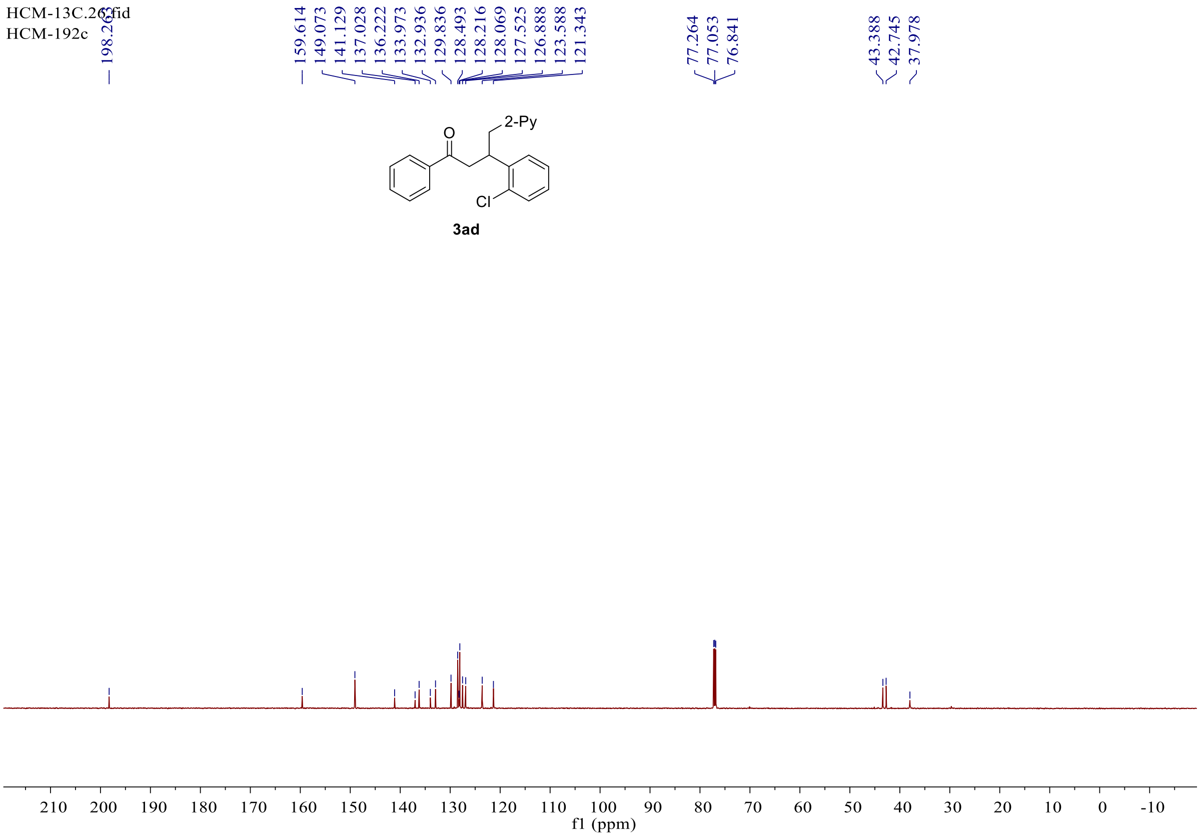
7.859
7.847
7.845
7.532
7.525
7.520
7.512
7.509
7.499
7.496
7.420
7.408
7.395
7.193
7.179
7.151
7.137
7.082
7.074
7.069
7.061
7.017
7.004
3.940
3.927
3.915
3.903
3.891
3.366
3.355
3.219
3.206
3.196
3.184
3.113
3.100
3.090
3.078
O
2-Py
Cl
3ac
1.000
2.099
2.119
2.207
2.011
2.086
1.182
1.100
1.080
2.076
1.105
1.062
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
f1 (ppm)


HCM-13C.23.fid
HCM-189c
198.33
159.475
149.260
142.501
137.028
136.236
133.005
132.058
128.980
128.529
128.025
123.665
121.377
77.242
77.030
76.818
44.964
44.259
41.063
O
2-Py
Cl
3ac
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

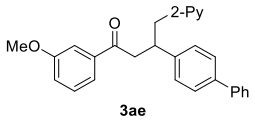


7.877
7.530
7.527
7.517
7.514
7.504
7.501
7.418
7.405
7.393
7.318
7.316
7.305
7.303
7.280
7.278
7.267
7.265
7.259
7.166
7.164
7.154
7.151
7.141
7.139
7.116
7.103
7.098
7.096
7.086
7.083
7.073
7.071
7.063
7.059
7.051
4.443
4.431
4.419
3.498
3.486
3.470
3.457
3.439
3.429
3.411
3.400
3.240
3.228
3.217
3.206
3.194
3.184
2-Py
O
Cl
3ad
1.000
2.008
2.290
2.287
1.083
1.338
1.299
1.936
1.262
1.052
2.256
2.304
f1 (ppm)

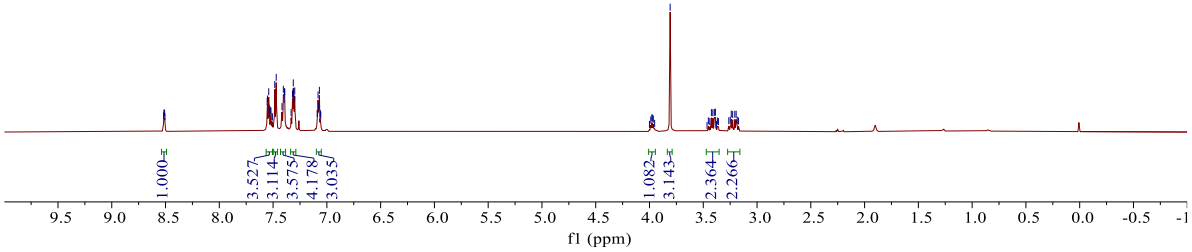


HCM-192c
198.26
159.614
149.073
141.129
137.028
136.222
133.973
132.936
129.836
128.493
128.216
128.069
127.525
126.888
123.588
121.343
77.264
77.053
76.841
43.388
42.745
37.978
2-Py
O
Cl
3ad
f1 (ppm)

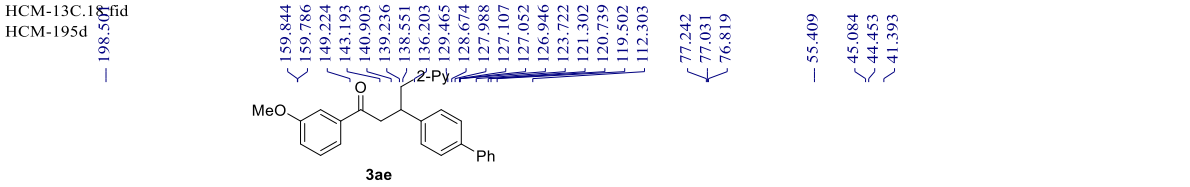
HCM-1H.57.fid
HCM-214a



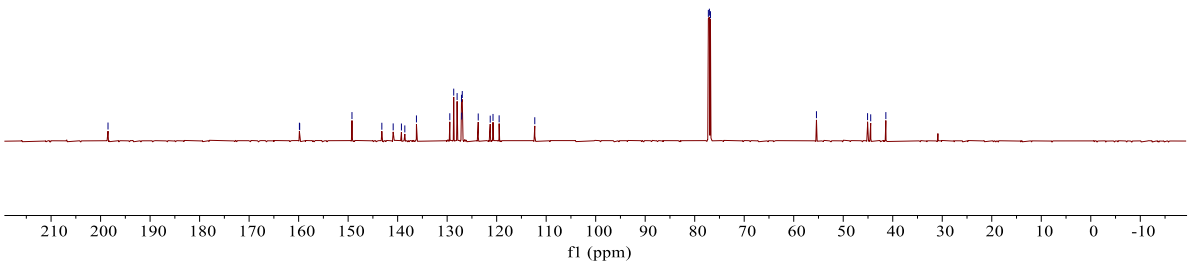
3ae



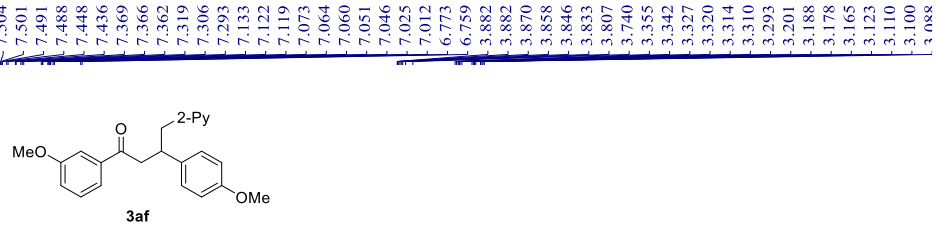
HCM-13C.1fid
HCM-195d



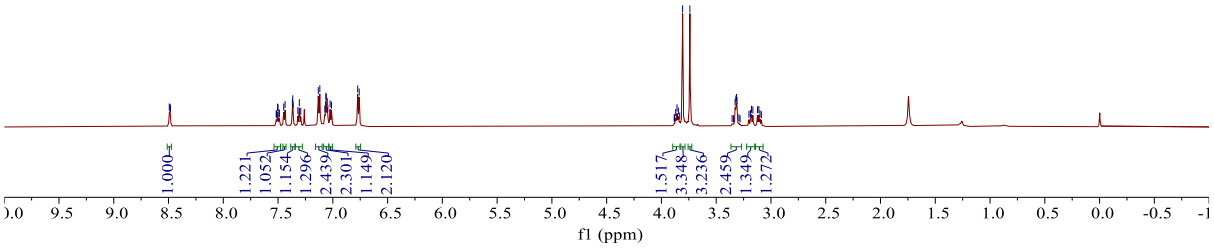
3ae



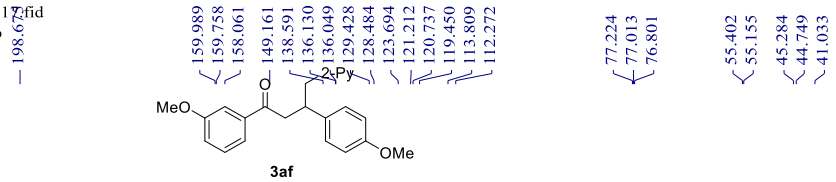
HCM-1H.33.fid
HCM-214



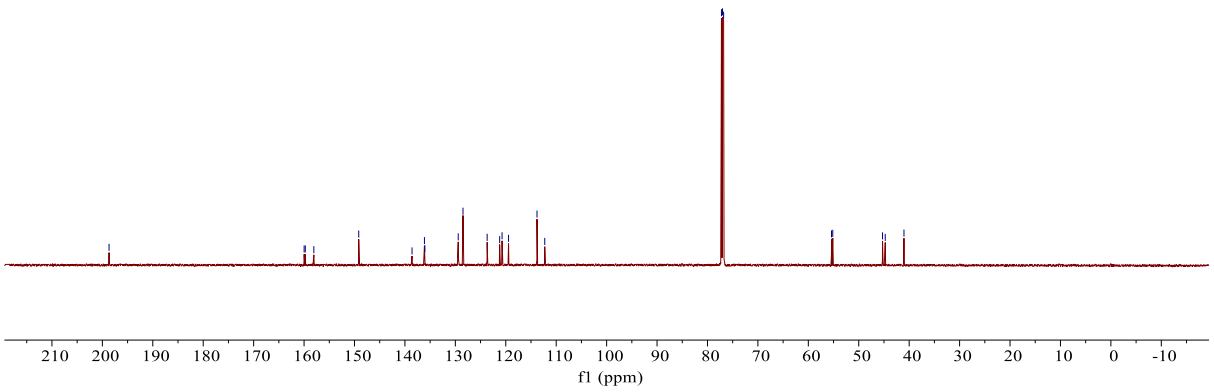
3af



HCM-13C.17.fid
HCM-214b



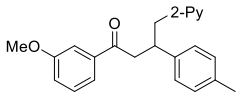
3af



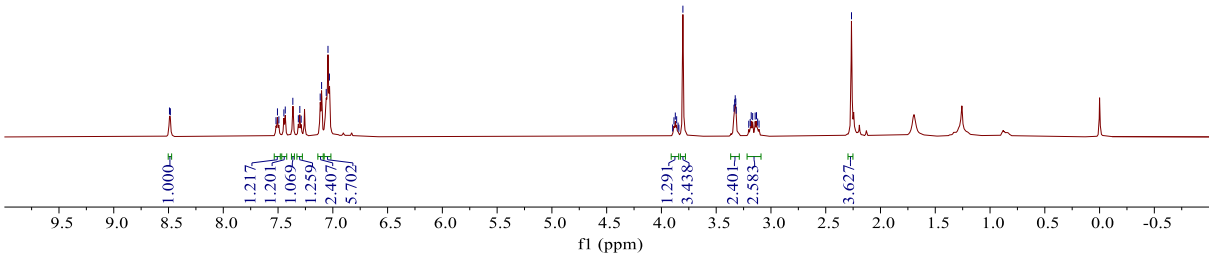
HCM-1H.14.fid
HCM-214c

8.491 8.483 7.519 7.505 7.493 7.447 7.434 7.365 7.315 7.301 7.289 7.115 7.102 7.060 7.044 7.031

3.898 3.885 3.873 3.861 3.848 3.841 3.804 3.338 3.331 3.326 3.321 3.317 3.203 3.190 3.180 3.167 3.144 3.132 3.122 3.109 2.265



3ag



HCM-13C.1.fid
HCM-214c

198.624

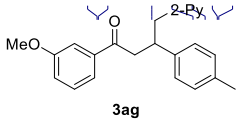
160.001 159.743 149.147 140.983 138.568 136.160 135.864 129.421 129.116 127.403 123.696 121.229 120.747 119.465 112.248

77.228 77.017 76.805

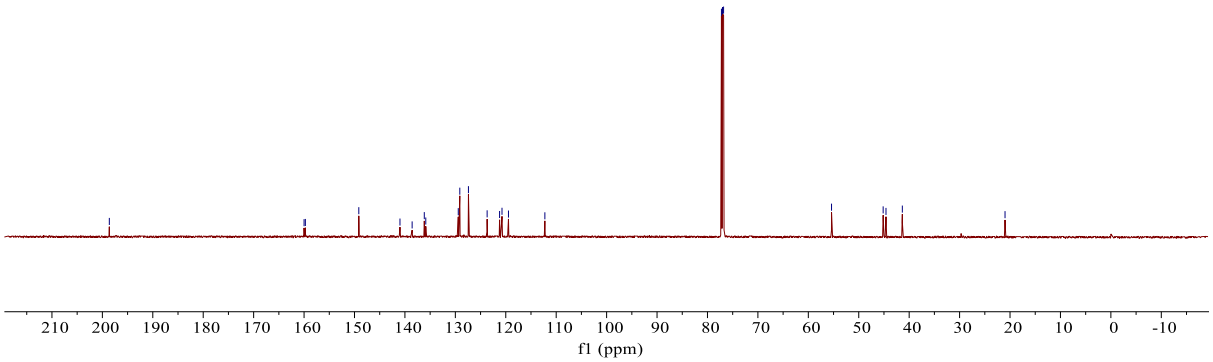
55.399

45.160 44.599 41.356

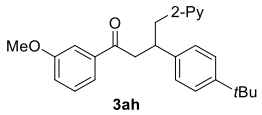
20.991



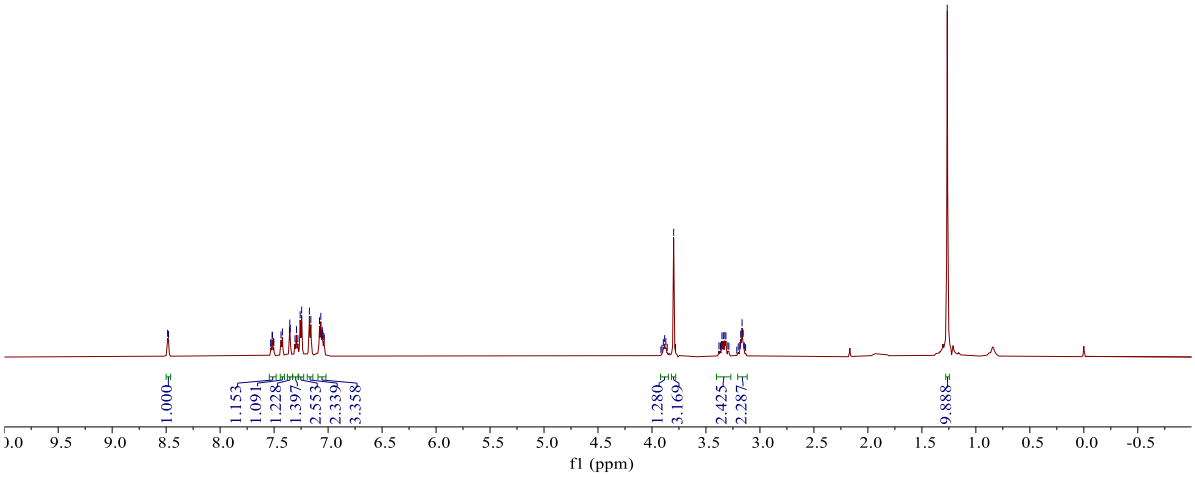
3ag



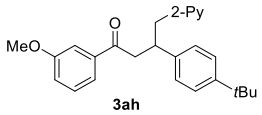
HCM-1H.65.fid
HCM-219c



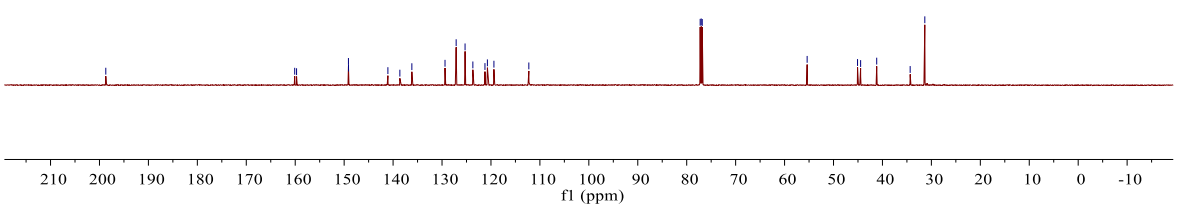
3ah



HCM-13C.38.fid
HCM-219c

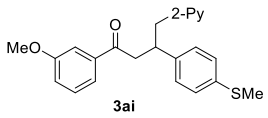


3ah

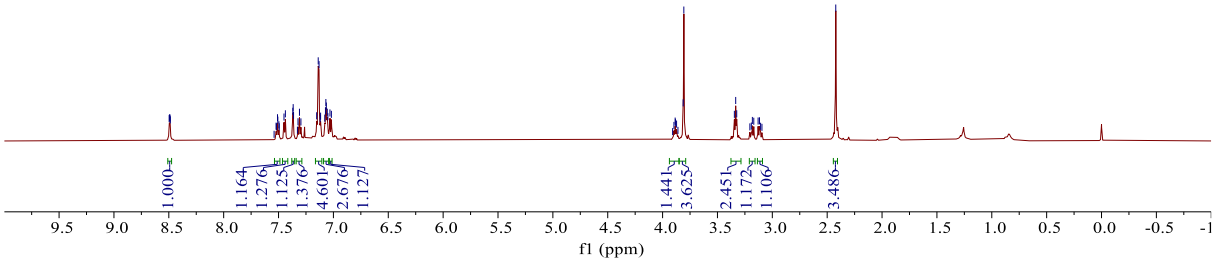


HCM-1H.75.fid
HCM-224b

8.497 8.494 8.489 8.486 7.541 7.522 7.519 7.509 7.506 7.497 7.494 7.450 7.438 7.370 7.367 7.363 7.328 7.323 7.309 7.296 7.153 7.138 7.131 7.120 7.117 7.079 7.073 7.069 7.064 7.056 7.050 7.031 7.018 3.909 3.896 3.886 3.883 3.873 3.860 3.812 3.806 3.346 3.334 3.324 3.204 3.191 3.182 3.169 3.130 3.117 3.107 3.095 2.422

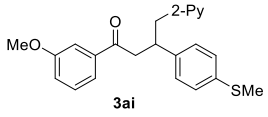


3ai

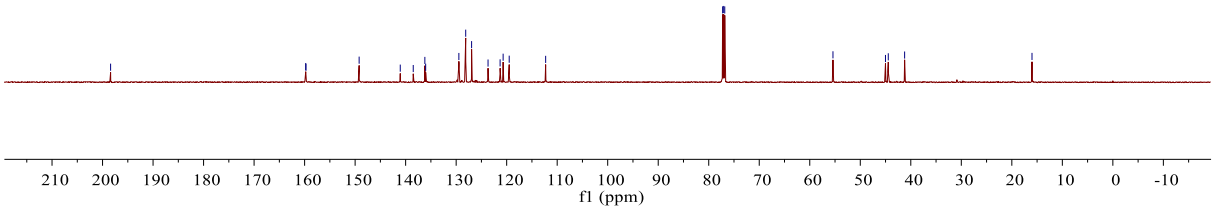


HCM-13C.34.fid
HCM-224b

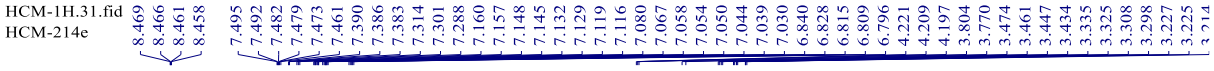
198.402 159.778 159.737 149.206 141.065 138.496 136.196 136.035 129.462 128.109 126.941 123.688 121.298 120.701 119.497 112.284 77.250 77.039 76.827 55.405 45.012 44.457 41.222 16.006

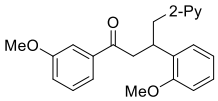


3ai

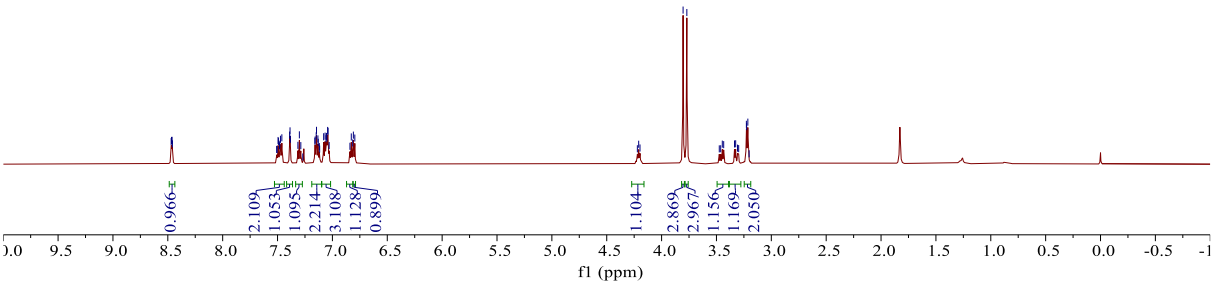


HCM-1H.31.fid
HCM-214e

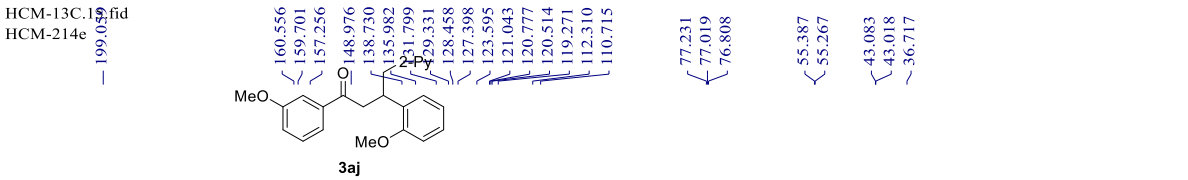




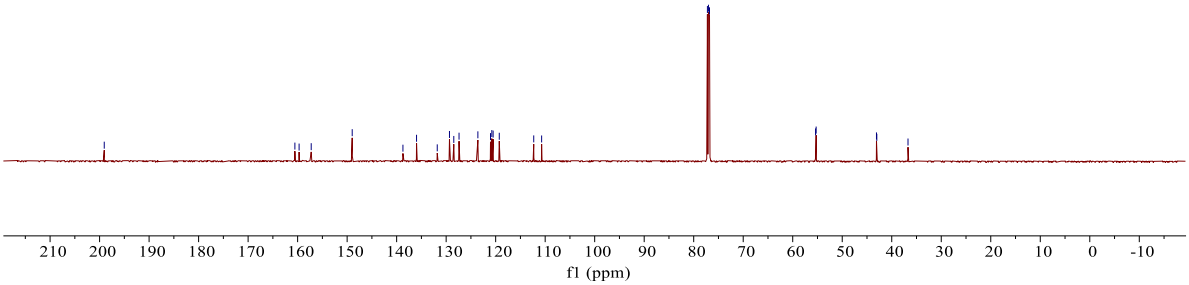
3aj

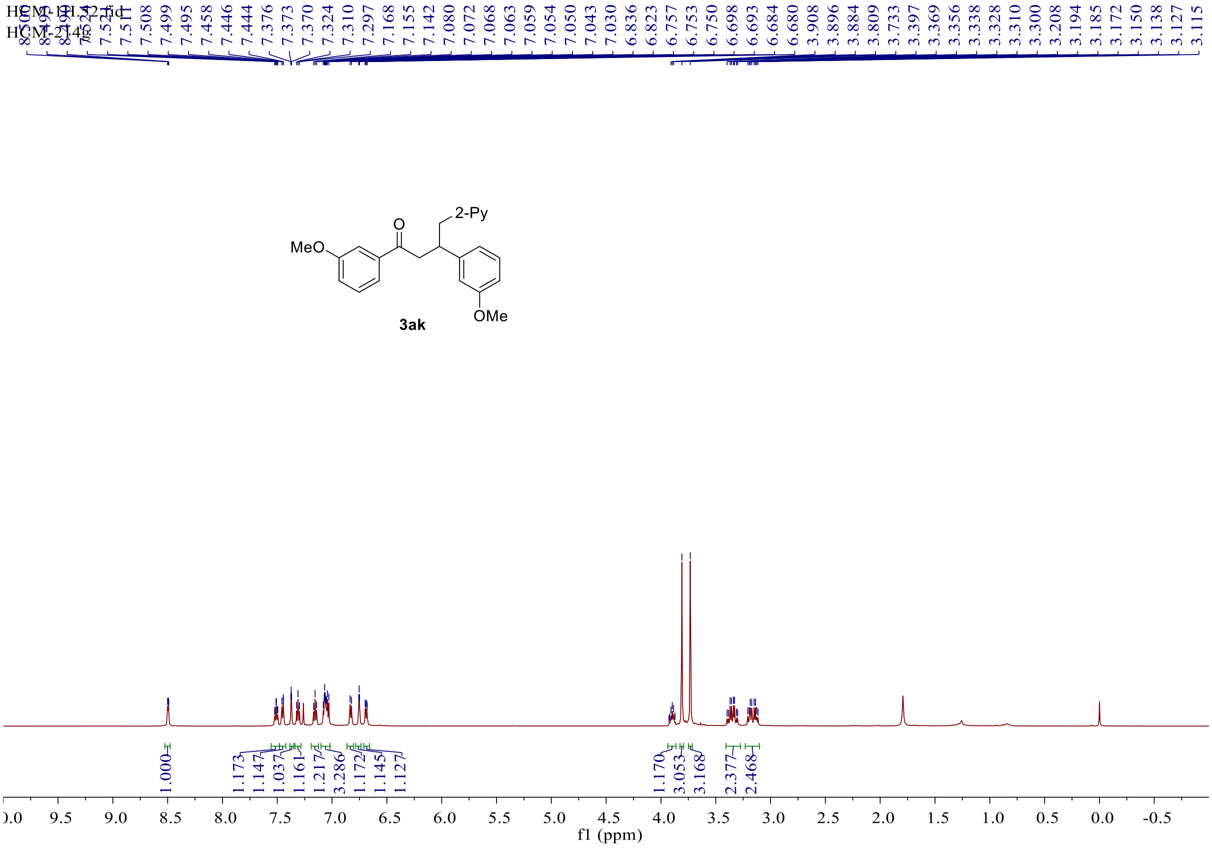


HCM-13C.15.fid
HCM-214e

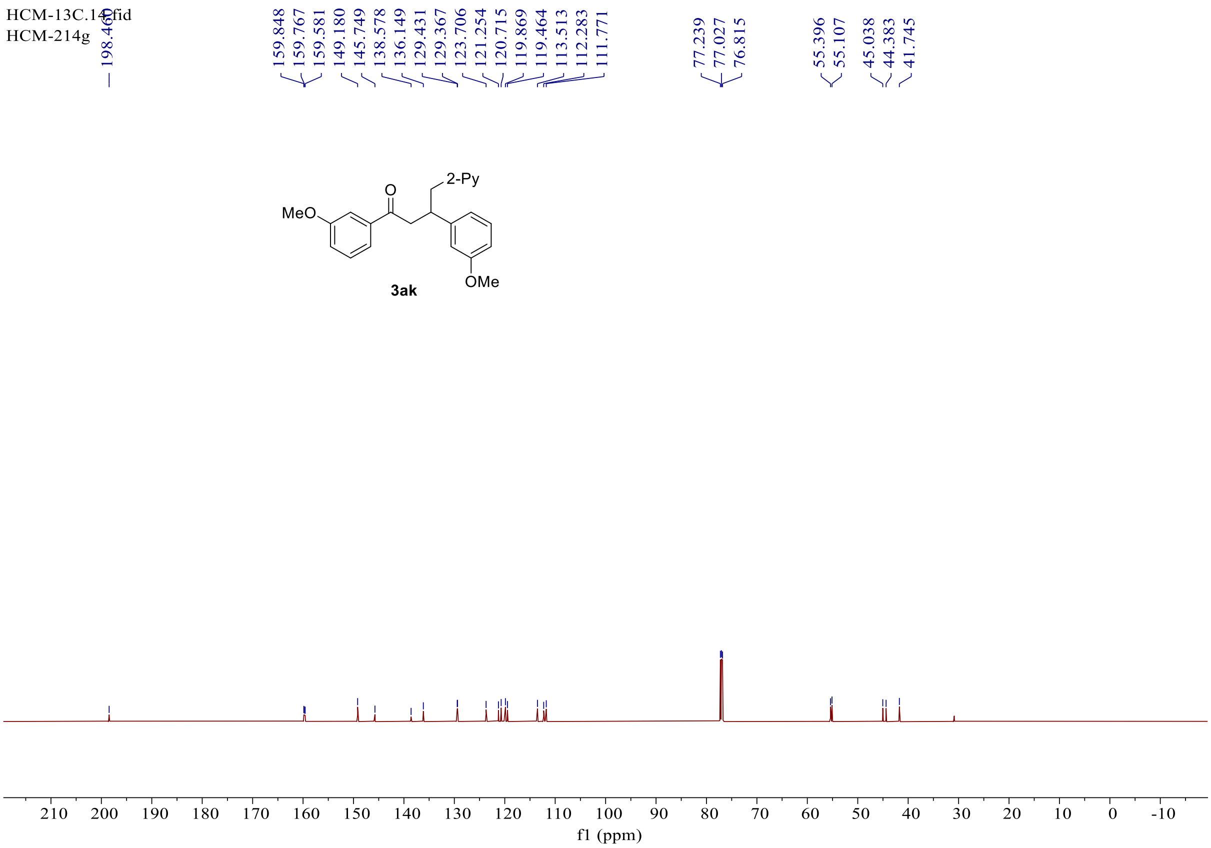


3aj





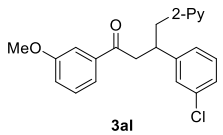
HCM-1H.52.fid
HCM-214g
7.511
7.508
7.499
7.495
7.458
7.446
7.444
7.376
7.373
7.370
7.324
7.310
7.297
7.168
7.155
7.142
7.080
7.072
7.068
7.063
7.059
7.054
7.050
7.043
7.030
6.836
6.823
6.757
6.753
6.750
6.698
6.693
6.684
6.680
3.908
3.896
3.884
3.809
3.733
3.397
3.369
3.356
3.338
3.328
3.310
3.300
3.208
3.194
3.185
3.172
3.150
3.138
3.127
3.115
2-Py
O
MeO
OMe
3ak
1.000
1.173
1.147
1.037
1.161
1.217
3.286
1.172
1.145
1.127
1.170
3.053
3.168
2.377
2.468
f1 (ppm)



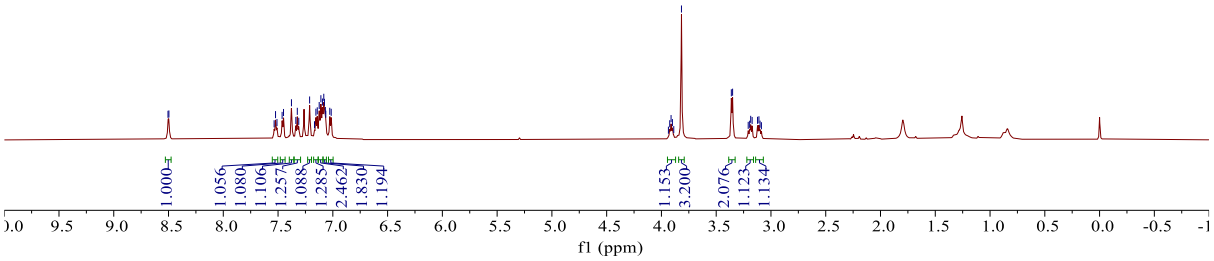
HCM-13C.14.fid
HCM-214g
198.461
159.848
159.767
159.581
149.180
145.749
138.578
136.149
129.431
129.367
123.706
121.254
120.715
119.869
119.464
113.513
112.283
111.771
77.239
77.027
76.815
55.396
55.107
45.038
44.383
41.745
2-Py
O
MeO
OMe
3ak
f1 (ppm)

HCM-1H.21.fid
HCM-214h

8.506 8.497 7.536 7.523 7.510 7.463 7.450 7.378 7.338 7.324 7.311 7.212 7.167 7.155 7.142 7.124 7.109 7.095 7.083 7.077 7.068 7.064 7.028 7.015 3.936 3.924 3.912 3.900 3.887 3.817 3.362 3.350 3.208 3.195 3.185 3.172 3.122 3.110 3.100 3.087

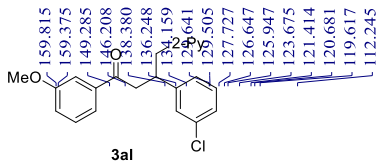


3al

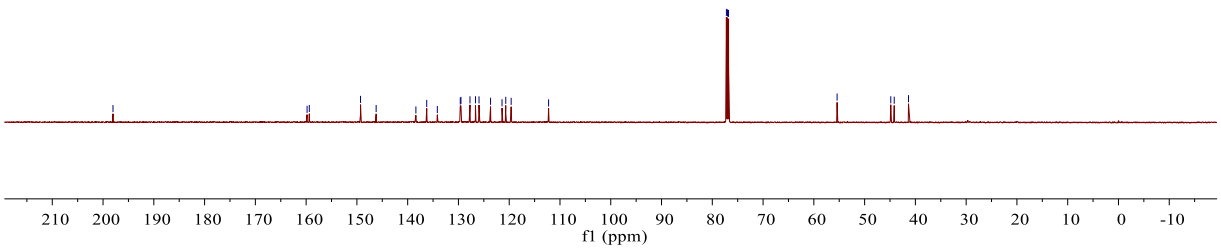


HCM-13C.12.fid
HCM-214h

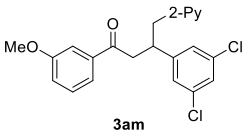
198.041 159.815 159.375 149.285 146.208 138.380 136.248 134.159 132.641 129.505 127.727 126.647 125.947 123.675 121.414 120.681 119.617 112.245 77.224 77.013 76.801 55.421 44.846 44.169 41.352



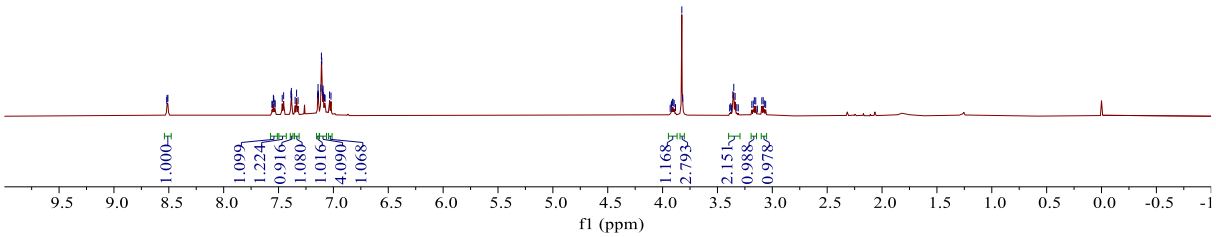
3al



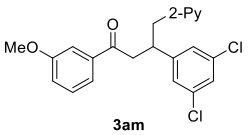
HCM-1H.77.fid
HCM-219a



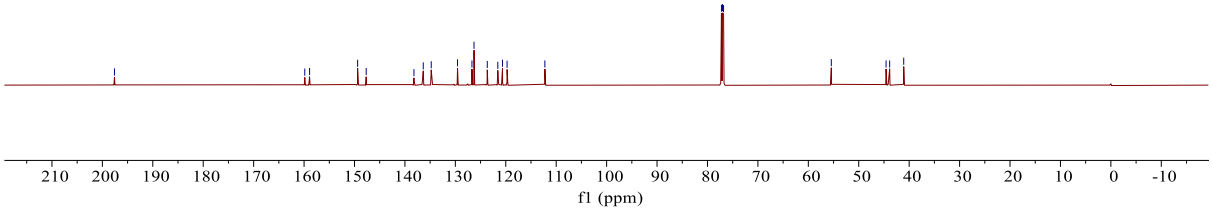
3am

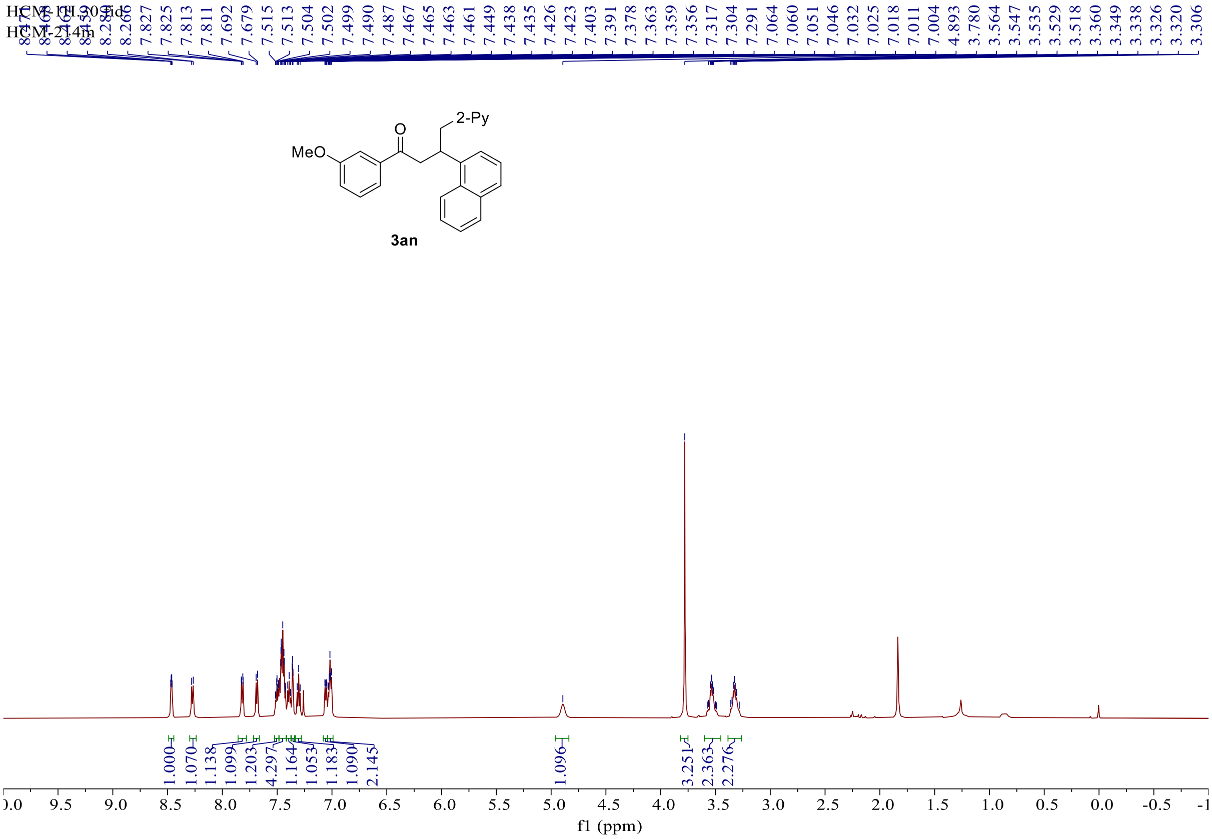


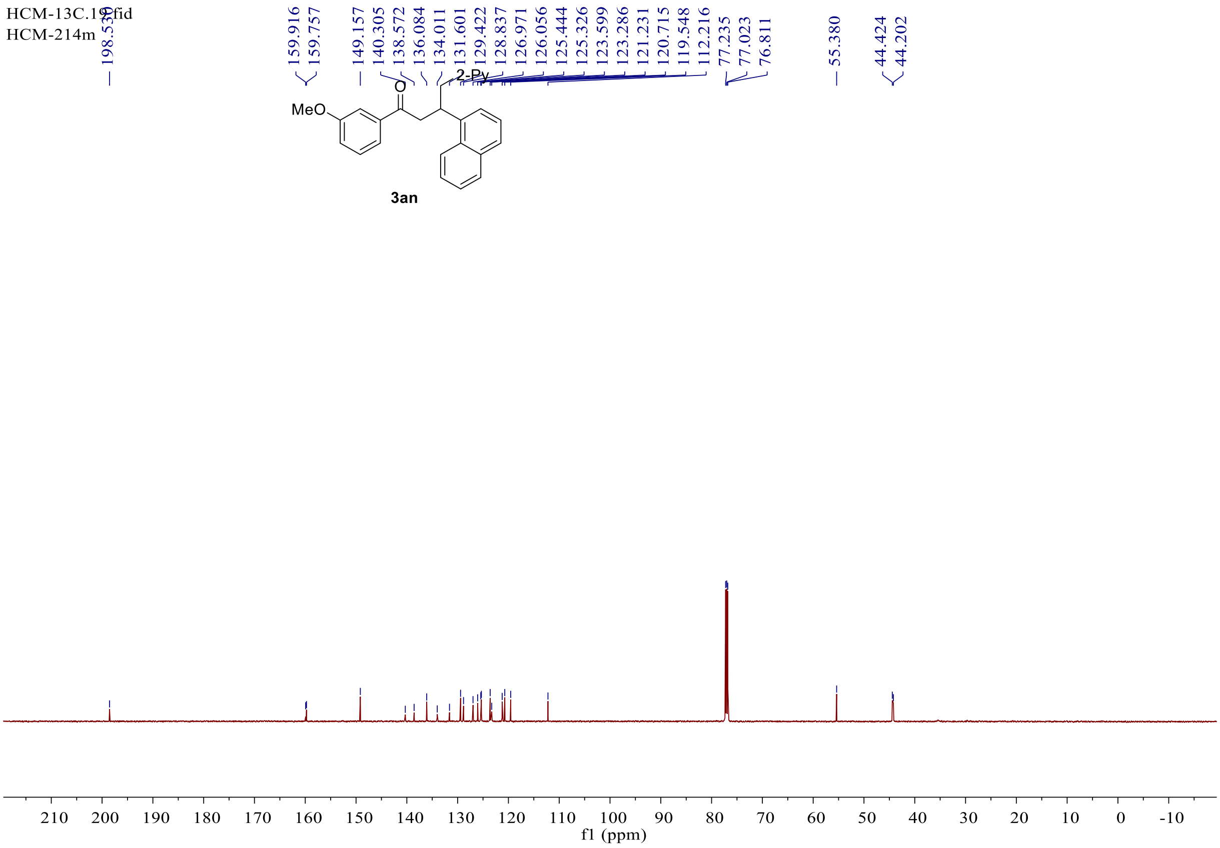
HCM-13C.36.fid
HCM-219a



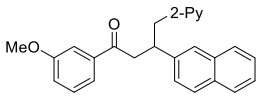
3am



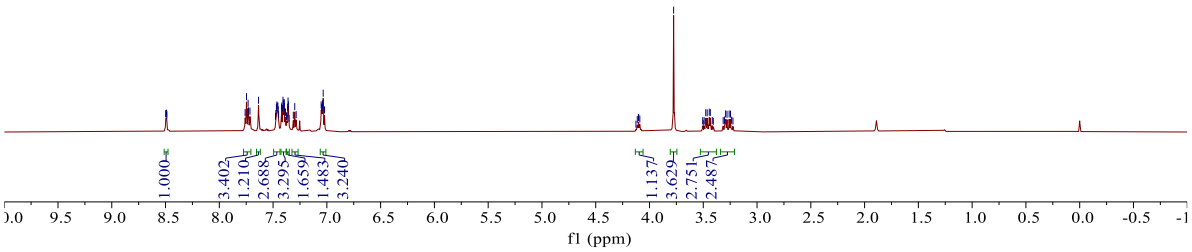

HCM-1H.20.fid
HCM-214m
7.827 7.825 7.813 7.811 7.692 7.679 7.515 7.513 7.504 7.502 7.499 7.490 7.487 7.467 7.465 7.463 7.461 7.449 7.438 7.435 7.426 7.423 7.403 7.391 7.378 7.363 7.359 7.356 7.317 7.304 7.291 7.064 7.060 7.051 7.046 7.032 7.025 7.018 7.011 7.004 4.893 3.780 3.564 3.547 3.535 3.529 3.518 3.360 3.349 3.338 3.326 3.320 3.306
MeO
O
2-Py
3an
1.000 1.070 1.138 1.099 1.203 4.297 1.164 1.053 1.183 1.090 2.145 1.096 3.251 2.363 2.276
f1 (ppm)


HCM-13C.19.fid
HCM-214m
198.53
159.916 159.757 149.157 140.305 138.572 136.084 134.011 131.601 129.422 128.837 126.971 126.056 125.444 125.326 123.599 123.286 121.231 120.715 119.548 112.216 77.235 77.023 76.811 55.380 44.424 44.202
MeO
O
2-Py
3an
f1 (ppm)

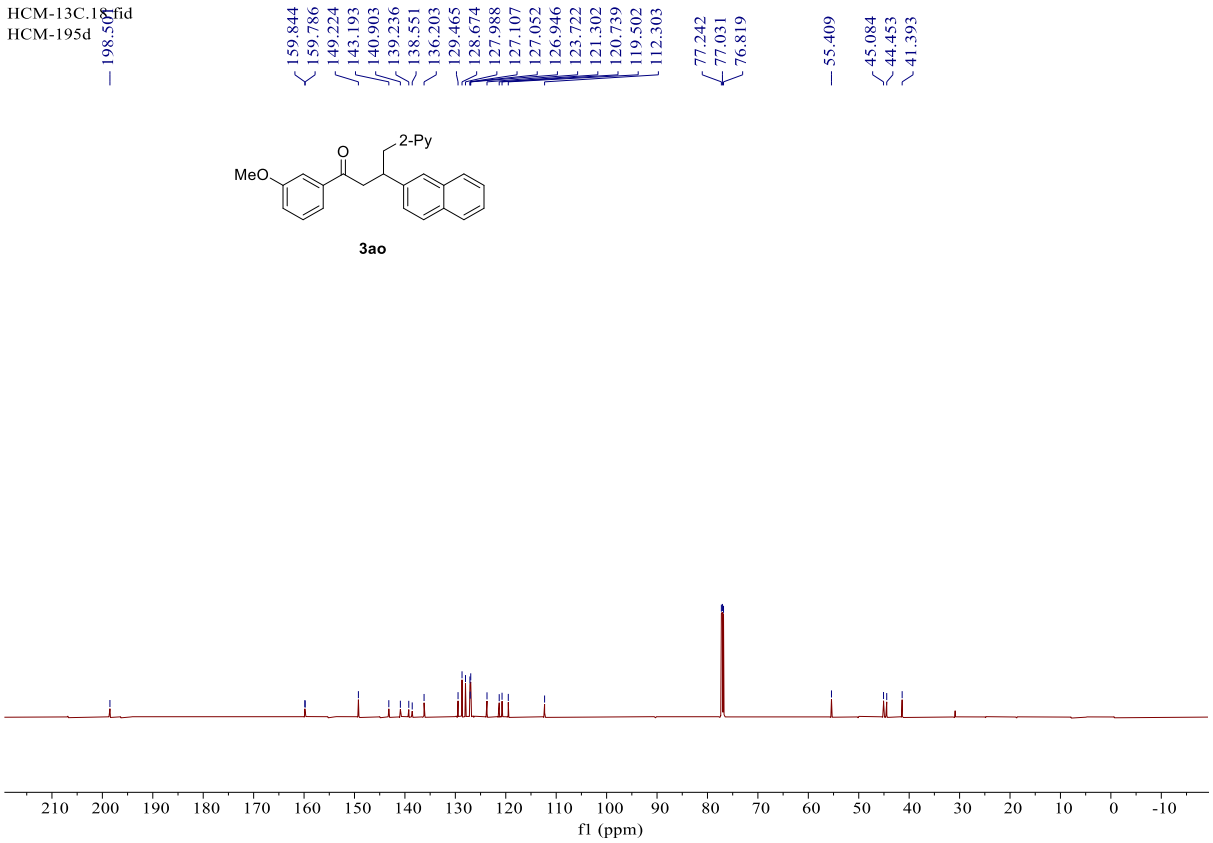
HCM-13H.1.fid
HCM-214L



3ao

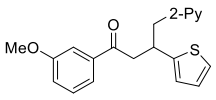


HCM-13C.1.fid
HCM-195d

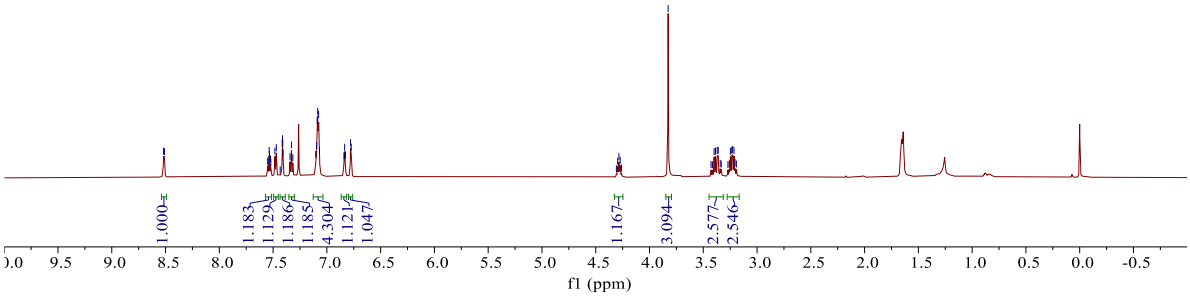


3ao

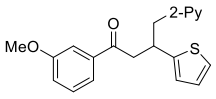
HCM-1H.55.fid
HCM-214o



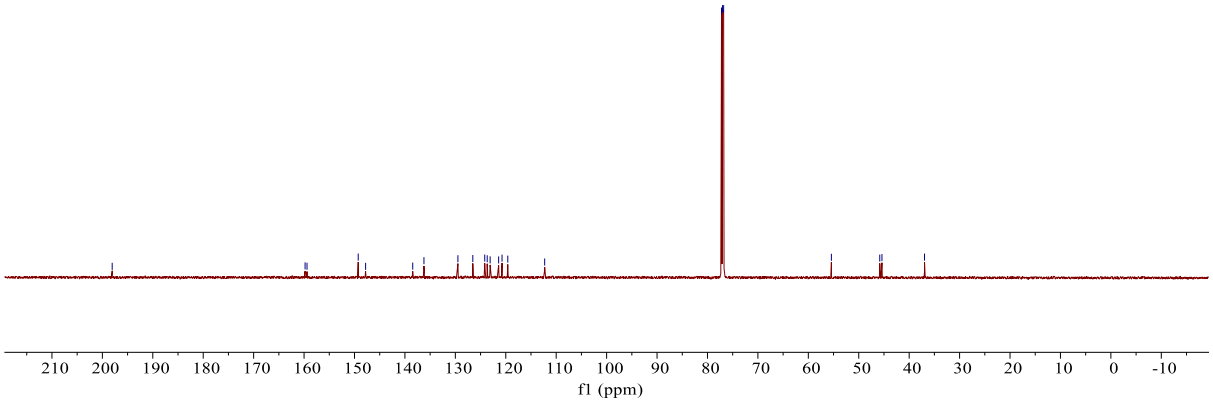
3ap



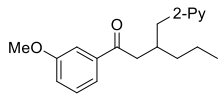
HCM-13C.20.fid
HCM-214o



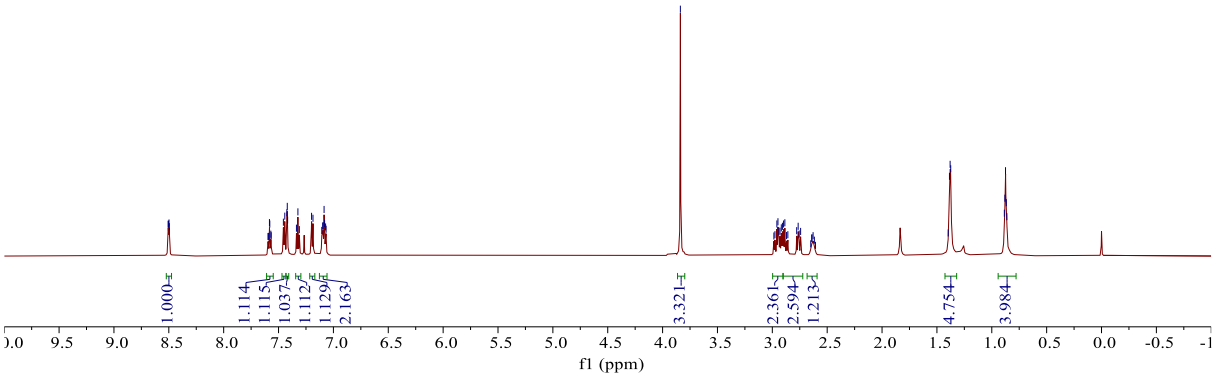
3ap



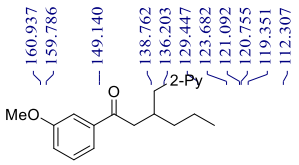
HCM-1H.56.fid
HCM-214v



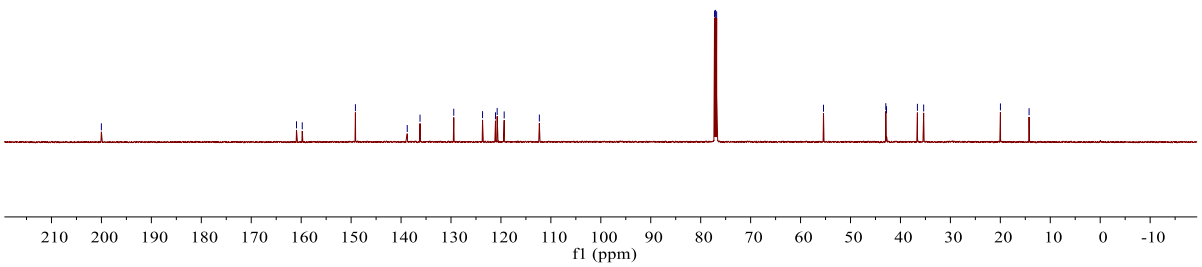
3aq

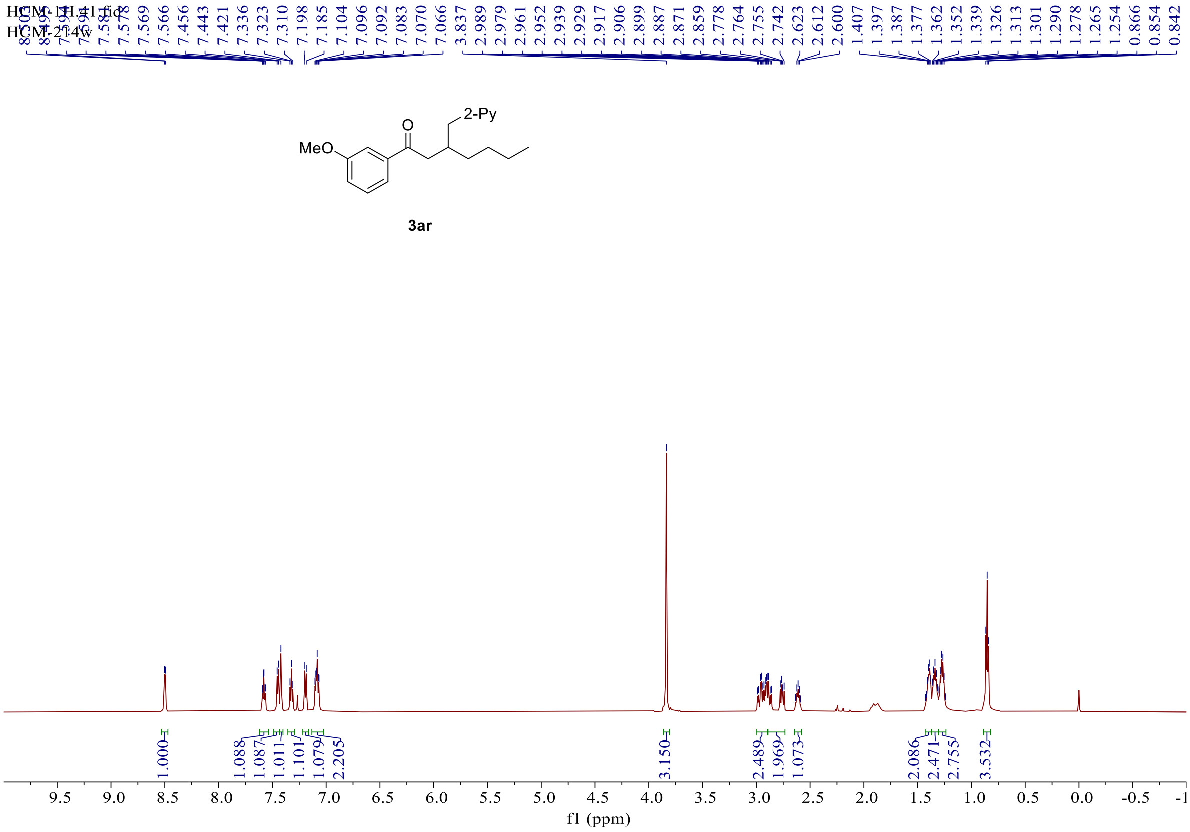


HCM-13C.27.fid
HCM-214V



3aq




HCM-1H.41.fid
HCM-214W
7.566
7.456
7.443
7.421
7.336
7.323
7.310
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0.854
0.842
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O
MeO
3ar
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1.087
1.011
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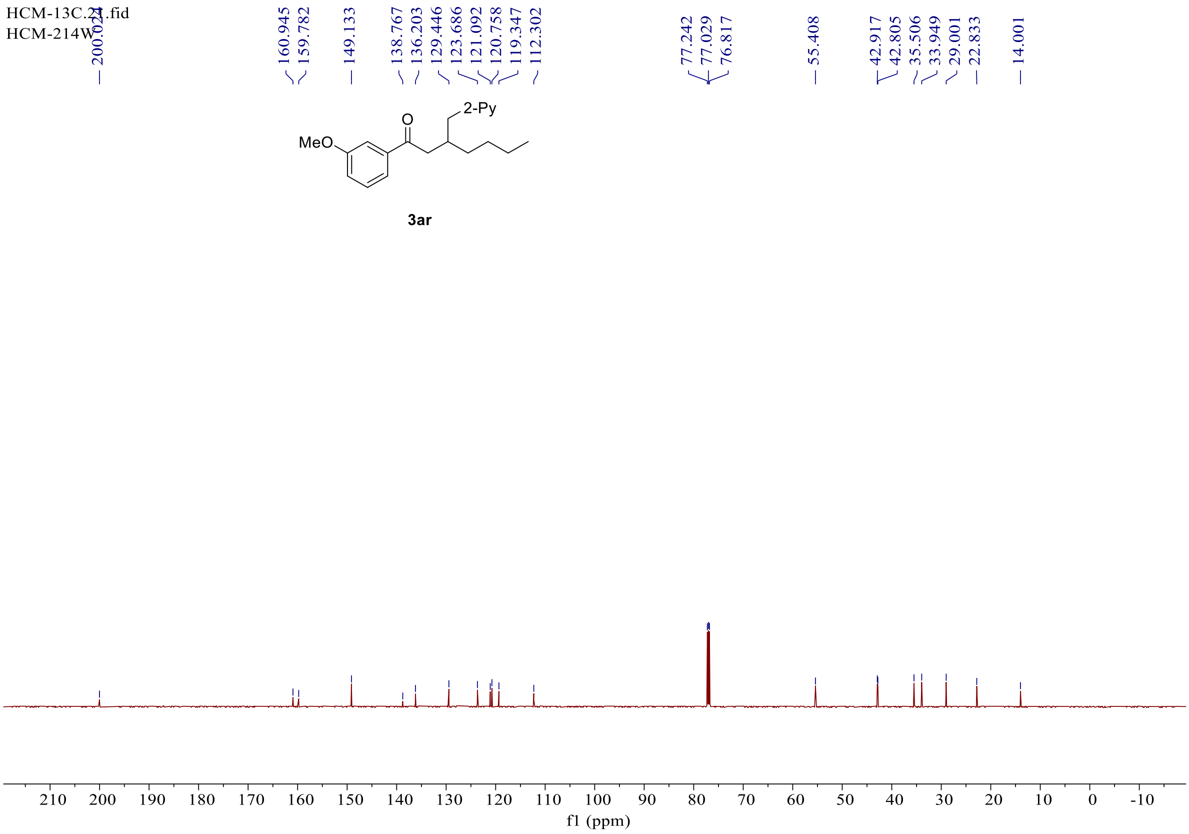
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