

Supporting Information

Boron-Catalyzed Dehydrative Allylation of 1,3-Diketones and β -Ketone Esters with 1,3-DiarylAllyl Alcohols in Water

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General Information.

All reagents and solvents were purchased from Adamas Reagent, Energy Chemical, Alfa Aresa Chemical Co., Aladdin Industrial Corp., Macklin Biochemical Co., Acors Organics, Bide Pharmatech Ltd., and so forth. All chemicals are without purification unless otherwise stated. All heating was done in an oil bath. Thin-layer chromatography (TLC) for analysis was performed on Schleicher&Schuell F1400/LS 254 silica gel plates observed under UV light ($\lambda = 254$ nm). ^1H -NMR spectra were recorded at 300 MHz or 400 MHz, and carbon NMR (^{13}C { ^1H }) were recorded at 101 MHz. CDCl_3 was used as a solvent as an internal standard (0.00 ppm) unless otherwise stated. High-resolution mass spectra were recorded using a Q-TOF time-of-flight mass spectrometer. Coupling constants (J) were reported in Hertz (Hz). Infrared (IR) spectra were recorded on a Jasco ATR MIRacle spectrophotometer. Samples were scanned in the 400-4000 cm^{-1} region with KBr pellet.

1. The synthesis of the substrates

1.1 The synthesis of β -diketone or β -keto ester.

These compounds can be synthesized by Claisen condensation reaction, in which the appropriate ketone was reacted with the ester of without α -hydrogen under the NaH to produce β -diketone. And the ketone were reacted with dimethyl carbonate under the NaH to form β -keto ester^[1]. The Compounds **1b**, **1c**, **1e-1h**, **1k-1s** can be synthesized by this method. **1i**, **1t** and **1u** can be synthesized by selectively reducing the double bond of conjugated unsaturated ketones in the presence of triethyl silane and HFIP catalyzed by $\text{B}(\text{C}_6\text{F}_5)_3$ ^[2].

1.2 The synthesis of 1,3-diarylallyl alcohols.

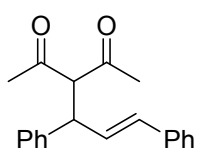
The allyl alcohol could be easily prepared according to literature^[3].

2. General experimental procedure and data

General Process of the allylation of 1,3-diketone and β -keto ester compounds with allyl alcohol.

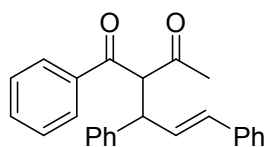
1,3-diketone and β -keto ester (0.3 mmol), allyl alcohol (0.3 mmol), $\text{B}(\text{C}_6\text{F}_5)_3$ (3 mol%), H_2O (1.5 mL) were added to the reaction tube and the reaction mixture was stirred at 100 $^\circ\text{C}$ in oil bath for 1 h. The reaction was monitored by TLC. When the reaction was finished, the mixture was cooled to RT and extracted with ethyl acetate three times (5 mL each). The organic phase was merged and dried with anhydrous MgSO_4 , and vacuum evaporated. Further purification was carried out via silica gel column chromatography and eluted with ethyl acetate and petroleum ether.

(E)-3-(1,3-diphenylallyl)pentane-2,4-dione (**3aa**)^[4a].



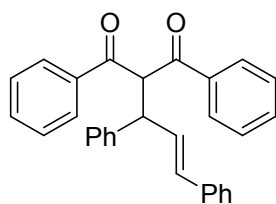
Yield: 78 mg, 89% yield; white solid; m.p.100.5-101.7 $^\circ\text{C}$; Rf: 0.5 (petroleum ether/EtOAc = 7/1). IR (KBr): 3028, 2964, 1724, 1701, 1359, 1263, 1095, 1026, 972, 804, 746, 700 cm^{-1} . ^1H NMR (300 MHz, CDCl_3) δ 7.39-7.15 (m, 10H), 6.45 (d, $J = 15.8$ Hz, 1H), 6.29-6.12 (m, 1H), 4.62-4.09 (m, 2H), 2.26 (s, 3H), 1.94 (s, 3H). ^{13}C { ^1H } NMR (101 MHz, CDCl_3) δ 202.8, 202.7, 140.1, 136.6, 131.7, 129.3, 129.0, 128.5, 127.9, 127.7, 127.3, 126.4, 74.5, 49.2, 30.0, 29.7.

(E)-2-(1,3-diphenylallyl)-1-phenylbutane-1,3-dione (**3ba**)^[4b].



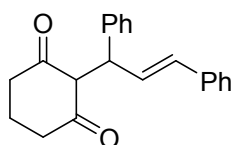
Yield: 88 mg, 83% yield; colorless liquid; Rf: 0.5 (petroleum ether/EtOAc = 7/1). IR (KBr): 3061, 3028, 1713, 1676, 1448, 1358, 1254, 970, 750, 696 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 7.99 (dd, $J = 77.4, 7.1$ Hz, 2H), 7.66-7.03 (m, 13H), 6.64-6.07 (m, 2H), 5.23 (dd, $J = 17.9, 11.2$ Hz, 1H), 4.63 (dd, $J = 11.2, 8.7$ Hz, 1H), 2.14 (d, $J = 103.2$ Hz, 3H). ^{13}C { ^1H } NMR (101 MHz, CDCl_3) δ 203.0, 194.3, 140.7, 140.0, 137.2, 136.9, 136.8, 136.6, 133.8, 133.6, 131.7, 131.6, 130.0, 129.4, 129.0, 128.9, 128.9, 128.7, 128.7, 128.6, 128.5, 128.4, 128.3, 127.9, 127.7, 127.4, 127.3, 127.0, 126.9, 126.4, 126.3, 69.5, 68.7, 49.5, 49.3, 28.1, 27.8.

(E)-2-(1,3-diphenylallyl)-1,3-diphenylpropane-1,3-dione (3ca)^[4b]



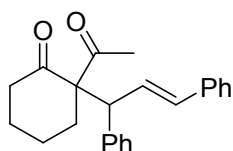
Yield: 88 mg, 83% yield; white solid, m.p.112.1-113.0 °C; Rf: 0.5 (petroleum ether/EtOAc = 7/1). **IR (KBr):** 3063, 3028, 2926, 1693, 1659, 1448, 1284, 1257, 991, 964, 746, 692 cm⁻¹. **¹H NMR (400 MHz, CDCl₃)** δ 8.12-8.04 (m, 2H), 7.90-7.83 (m, 2H), 7.57-7.53 (m, 1H), 7.48-7.30 (m, 7H), 7.28-7.08 (m, 8H), 6.44-6.30 (m, 2H), 6.04 (d, *J* = 10.5 Hz, 1H), 4.86 (ddd, *J* = 10.5, 6.1, 1.3 Hz, 1H). **¹³C {¹H} NMR (101 MHz, CDCl₃)** δ 194.5, 193.8, 141.0, 137.3, 136.9, 136.9, 133.5, 133.3, 131.9, 130.0, 128.9, 128.8, 128.7, 128.6, 128.5, 128.3, 127.4, 126.9, 126.3, 62.5, 50.1.

(E)-2-(1,3-diphenylallyl)cyclohexane-1,3-dione (3da)^[4c]



Yield: 68 mg, 75% yield; white solid, m.p.107.5-108.4 °C; Rf: 0.5 (petroleum ether/EtOAc = 7/1). **IR (KBr):** 3026, 2951, 1771, 1599, 1562, 1493, 1369, 1275, 1192, 1082, 976, 746, 698 cm⁻¹. **¹H NMR (400 MHz, CDCl₃)** δ 7.48-7.16 (m, 10H), 6.94 (dd, *J* = 15.9, 8.1 Hz, 1H), 6.52 (d, *J* = 15.9 Hz, 1H), 5.24 (d, *J* = 8.1 Hz, 1H), 2.41 (t, *J* = 6.0 Hz, 4H), 1.91 (p, *J* = 6.5 Hz, 2H). **¹³C {¹H} NMR (101 MHz, CDCl₃)** δ 142.2, 137.1, 131.8, 130.6, 128.5, 128.5, 127.8, 127.4, 126.4, 126.3, 117.4, 41.9, 20.6.

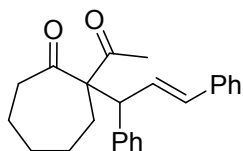
(E)-2-acetyl-2-(1,3-diphenylallyl)cyclohexan-1-one (3ea).



Yield: 85 mg, total yield 86% yield; and **3ea (A)** as a white solid, m.p.80.5-81.4°C; Rf: 0.5 (petroleum ether/EtOAc = 7/1). **IR (KBr):** 3028, 2951, 1695, 1599, 1495, 1452, 1356, 1174, 1126, 972, 748, 700 cm⁻¹. **¹H NMR (400 MHz, CDCl₃)** δ 7.43-7.15 (m, 10H), 6.51-6.46 (m, 2H), 4.44 (dd, *J* = 5.7, 3.6 Hz, 1H), 2.62-2.44 (m, 2H), 2.20 (ddd, *J* = 14.9, 12.9, 6.0 Hz, 1H), 2.04 (s, 3H), 1.88 (dddd, *J* = 12.0, 6.0, 4.3, 2.6 Hz, 1H), 1.81-1.58 (m, 4H). **¹³C {¹H} NMR (101 MHz, CDCl₃)** δ: 208.3, 205.0, 139.6, 136.9, 132.7, 129.6, 128.4, 128.1, 127.7, 127.5, 126.7, 126.4, 72.1, 52.06, 42.68, 31.0, 27.3, 25.7, 20.8.

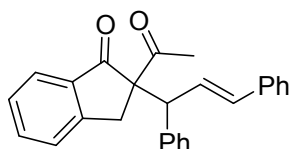
And a colorless liquid **3ea (B)**; Rf: 0.4 (petroleum ether/EtOAc = 7/1). **¹H NMR (400 MHz, CDCl₃)** δ 7.30 (s, 10H), 6.59 (dd, *J* = 15.8, 7.9 Hz, 1H), 6.42-6.33 (m, 1H), 4.42 (dd, *J* = 7.9, 1.2 Hz, 1H), 2.72-2.64 (m, 1H), 2.53 (ddq, *J* = 15.0, 3.5, 1.6 Hz, 1H), 2.30-2.17 (m, 1H), 1.98 (s, 3H), 1.97-1.74 (m, 3H), 1.65-1.60 (m, 2H). **¹³C {¹H} NMR (101 MHz, CDCl₃)** δ: 208.3, 205.0, 139.6, 136.9, 132.7, 129.6, 128.4, 128.1, 127.7, 127.5, 126.7, 126.4, 72.1, 52.0, 42.6, 31.2, 27.3, 25.7, 20.8. **HRMS (ESI) m/z (M+Na)⁺** calcd for C₂₄H₂₆O₂Na: 369.1830; Found: 369.1832.

(E)-2-acetyl-2-(1,3-diphenylallyl)cycloheptan-1-one (3fa).



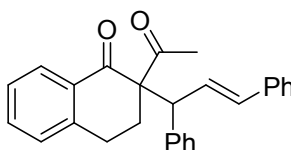
Yield: 81 mg, 78% yield; colorless liquid; Rf: 0.5 (petroleum ether/EtOAc = 7/1). **IR (KBr):** 2929, 2858, 1695, 1601, 1493, 1454, 1356, 1163, 970, 748, 698 cm⁻¹. **¹H NMR (300 MHz, CDCl₃)** δ 7.40-7.15 (m, 10H), 6.83-6.29 (m, 2H), 4.55-4.01 (m, 1H), 2.48-2.23 (m, 2H), 2.20 (s, 4H), 2.00 (dd, *J* = 15.0, 10.2 Hz, 1H), 1.69-1.29 (m, 6H). **¹³C {¹H} NMR (101 MHz, CDCl₃)** δ: 212.5, 211.3, 208.6, 206.2, 139.9, 139.2, 137.2, 136.8, 132.9, 132.2, 129.7, 129.3, 129.0, 128.5, 128.5, 128.4, 128.3, 127.6, 127.5, 127.3, 127.3, 127.0, 126.4, 74.6, 73.4, 56.2, 52.9, 45.3, 43.6, 32.2, 30.3, 28.6, 28.3, 26.2, 25.6, 25.4, 25.4. **HRMS (ESI) m/z (M+Na)⁺** calcd for C₂₃H₂₄O₂Na: 355.1674; Found: 355.1684.

(E)-2-acetyl-2-(1,3-diphenylallyl)-2,3-dihydro-1H-inden-1-one (3ga).



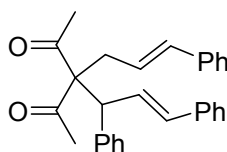
Yield: 91 mg, 83% yield; colorless liquid; Rf: 0.5 (petroleum ether/EtOAc = 7/1). **IR (KBr):** 3030, 2926, 1699, 1605, 1495, 1456, 1356, 1273, 1176, 968, 768, 700 cm^{-1} . **^1H NMR (400 MHz, CDCl_3)** δ 7.85-6.92 (m, 14H), 6.65-6.02 (m, 2H), 4.98-4.91 (m, 1H), 4.20 (dd, $J = 39.4, 17.1$ Hz, 1H), 3.36 (dd, $J = 85.5, 17.2$ Hz, 1H), 2.37 (d, $J = 107.0$ Hz, 3H). **^{13}C { ^1H } NMR (101 MHz, CDCl_3)** δ : 202.7, 202.3, 202.0, 201.9, 154.3, 153.9, 139.1, 138.7, 136.7, 136.6, 135.6, 135.2, 135.1, 134.8, 133.5, 133.1, 129.4, 128.9, 128.7, 128.6, 128.3, 128.2, 128.2, 127.9, 127.7, 127.5, 127.5, 127.3, 127.1, 126.4, 126.4, 126.3, 126.1, 124.5, 124.2, 74.3, 73.5, 53.4, 51.2, 32.0, 29.9, 26.2, 26.0. **HRMS (ESI) m/z ($\text{M}+\text{Na}$) $^+$** calcd for $\text{C}_{26}\text{H}_{22}\text{O}_2\text{Na}$: 389.1517; Found: 389.1525.

(E)-2-acetyl-2-(1,3-diphenylallyl)-3,4-dihydronaphthalen-1(2H)-one (3ha).



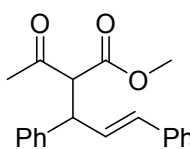
Yield: 100 mg, 88% yield; colorless liquid; Rf: 0.5 (petroleum ether/EtOAc = 7/1). **IR (KBr):** 3028, 2931, 1707, 1674, 1599, 1493, 1450, 1358, 1230, 1173, 970, 748, 700 cm^{-1} . **^1H NMR (400 MHz, CDCl_3)** δ 8.11 (ddd, $J = 23.1, 7.9, 1.5$ Hz, 1H), 7.49-7.10 (m, 13H), 6.99-6.39 (m, 2H), 4.63 (dd, $J = 195.7, 8.9$ Hz, 1H), 3.10 (dddd, $J = 83.7, 17.2, 12.6, 4.6$ Hz, 1H), 2.87-2.68 (m, 2H), 2.25 (s, 1H), 2.09 (d, $J = 7.8$ Hz, 3H). **^{13}C { ^1H } NMR (101 MHz, CDCl_3)** δ : 205.2, 202.8, 197.6, 195.9, 143.8, 143.4, 140.2, 139.3, 137.2, 136.9, 133.9, 133.8, 133.0, 133.0, 132.6, 132.5, 131.9, 130.0, 129.6, 129.3, 128.8, 128.7, 128.5, 128.5, 128.4, 128.2, 128.0, 127.9, 127.6, 127.5, 127.4, 127.2, 127.1, 126.8, 126.8, 126.8, 126.5, 126.5, 126.4, 125.8, 69.4, 67.5, 55.2, 50.4, 29.0, 28.2, 27.5, 26.9, 26.6, 26.2, 26.0, 23.2. **HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$** calcd for $\text{C}_{27}\text{H}_{25}\text{O}_2$: 381.1874; Found: 381.1855.

3-cinnamyl-3-((E)-1,3-diphenylallyl)pentane-2,4-dione (3ia).



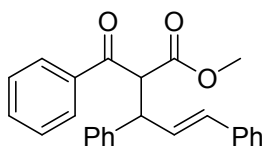
Yield: 62 mg, 51% yield; colorless liquid; Rf: 0.5 (petroleum ether/EtOAc = 7/1). **IR (KBr):** 3028, 2922, 1724, 1701, 1601, 1495, 1360, 1271, 1140, 972, 744, 698, cm^{-1} . **^1H NMR (400 MHz, CDCl_3)** δ 7.38-7.15 (m, 15H), 6.61 (dd, $J = 15.8, 8.5$ Hz, 1H), 6.43-6.32 (m, 2H), 5.88 (dt, $J = 15.7, 7.2$ Hz, 1H), 4.46 (dd, $J = 8.6, 1.1$ Hz, 1H), 2.91 (ddd, $J = 15.3, 7.3, 1.5$ Hz, 1H), 2.81 (ddd, $J = 15.3, 7.2, 1.6$ Hz, 1H), 2.22 (s, 3H), 2.05 (s, 3H). **^{13}C { ^1H } NMR (101 MHz, CDCl_3)** δ : 206.6, 206.1, 139.1, 137.0, 136.8, 133.7, 132.6, 129.6, 128.9, 128.5, 128.5, 128.4, 127.5, 127.3, 126.3, 126.1, 124.3, 74.1, 51.4, 36.1, 30.3, 29.5. **HRMS (ESI) m/z ($\text{M}+\text{Na}$) $^+$** calcd for $\text{C}_{29}\text{H}_{28}\text{O}_2\text{Na}$: 431.1987; Found: 431.1995.

Methyl (E)-2-acetyl-3,5-diphenylpent-4-enoate (3ja)^[4a].



Yield: 64 mg, 69% yield; colorless liquid; Rf: 0.4 (petroleum ether/EtOAc = 7/1). **IR (KBr):** 3028, 2953, 1741, 1714, 1601, 1495, 1450, 1358, 1157, 970, 746, 698 cm^{-1} . **^1H NMR (300 MHz, CDCl_3)** δ 7.40-7.14 (m, 10H), 6.46 (dd, $J = 15.8, 9.1$ Hz, 1H), 6.28 (ddd, $J = 15.9, 14.0, 8.2$ Hz, 1H), 4.31 (ddd, $J = 10.8, 8.1, 2.4$ Hz, 1H), 4.13 (dd, $J = 11.2, 6.9$ Hz, 1H), 3.60 (d, $J = 64.6$ Hz, 3H), 2.17 (d, $J = 81.3$ Hz, 3H). **^{13}C { ^1H } NMR (101 MHz, CDCl_3)** δ : 201.6, 201.4, 168.4, 168.1, 140.4, 140.1, 136.8, 136.6, 132.0, 131.6, 129.5, 129.1, 128.9, 128.8, 128.5, 128.0, 127.9, 127.7, 127.6, 127.2, 127.1, 126.4, 65.4, 65.1, 52.6, 52.4, 49.0, 48.8, 30.1, 30.0.

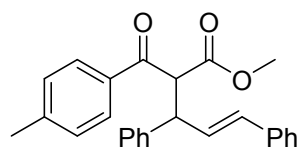
Methyl (E)-2-benzoyl-3,5-diphenylpent-4-enoate (3ka)^[4b].



Yield: 99 mg, 86% yield; white solid; m.p. 112.1-113.0 $^{\circ}\text{C}$; Rf: 0.4 (petroleum ether/EtOAc = 7/1). **IR (KBr):** 3028, 2951, 1736, 1680, 1599, 1495, 1450, 1261, 1157, 970, 744, 698 cm^{-1} . **^1H NMR (300 MHz, CDCl_3)** δ 8.15-8.06 (m, 1H),

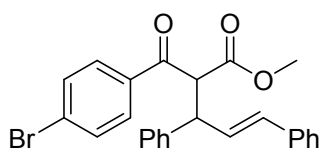
7.99 -7.90 (m, 1H), 7.70-7.04 (m, 13H), 6.63-6.14 (m, 2H), 5.06 (dd, $J = 10.9, 6.1$ Hz, 1H), 4.62 (td, $J = 10.8, 10.3, 8.1$ Hz, 1H), 3.56 (d, $J = 67.8$ Hz, 3H). ^{13}C { ^1H } NMR (101 MHz, CDCl_3) δ 193.2, 192.6, 168.5, 168.0, 140.7, 140.4, 137.0, 136.9, 136.8, 136.6, 133.7, 133.6, 131.9, 131.7, 129.7, 129.7, 128.9, 128.8, 128.7, 128.5, 128.5, 128.3, 128.3, 128.0, 127.5, 127.4, 127.2, 126.9, 126.4, 126.3, 59.7, 59.5, 52.7, 52.5, 49.0, 48.9.

Methyl (E)-2-(4-methylbenzoyl)-3,5-diphenylpent-4-enoate (3la).



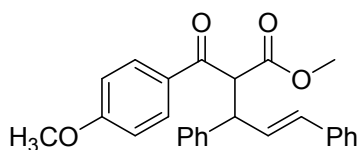
Yield: 92 mg, 79% yield; colorless liquid; Rf: 0.4 (petroleum ether/EtOAc = 7/1). IR (KBr): 3028, 2956, 1736, 1672, 1605, 1452, 1288, 1261, 1186, 1159, 968, 748, 698 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 8.02-7.95 (m, 1H), 7.87-7.80 (m, 1H), 7.41-7.26 (m, 6H), 7.25-7.08 (m, 6H), 6.57-6.17 (m, 2H), 5.00 (td, $J = 11.0, 0.9$ Hz, 1H), 4.65-4.50 (m, 1H), 3.54 (d, $J = 91.6$ Hz, 3H), 2.40 (d, $J = 14.9$ Hz, 3H). ^{13}C { ^1H } NMR (101 MHz, CDCl_3) δ : 192.5, 192.0, 168.5, 168.1, 144.7, 144.5, 140.7, 140.4, 137.0, 136.9, 134.3, 134.0, 131.7, 131.5, 129.8, 129.7, 129.5, 129.3, 128.9, 128.6, 128.6, 128.4, 128.3, 128.3, 127.9, 127.4, 127.3, 127.1, 126.7, 126.3, 126.2, 59.9, 59.0, 52.6, 52.3, 48.9, 48.0, 21.6, 21.6. HRMS (ESI) m/z ($\text{M}+\text{Na}$) $^+$ calcd for $\text{C}_{26}\text{H}_{24}\text{O}_3\text{Na}$: 407.1623; Found: 407.1624.

Methyl (E)-2-(4-bromobenzoyl)-3,5-diphenylpent-4-enoate (3ma).



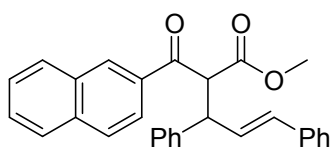
Yield: 114 mg, 85% yield; colorless liquid; Rf: 0.4 (petroleum ether/EtOAc = 7/1). IR (KBr): 3028, 2951, 1736, 1684, 1585, 1493, 1450, 1259, 1290, 1157, 968, 748, 698 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 7.97-7.89 (m, 1H), 7.83-7.73 (m, 1H), 7.62 (dq, $J = 9.1, 2.2$ Hz, 1H), 7.58-7.51 (m, 1H), 7.40-7.08 (m, 10H), 6.57-6.16 (m, 2H), 4.95 (t, $J = 11.1$ Hz, 1H), 4.62-4.49 (m, 1H), 3.55 (d, $J = 92.9$ Hz, 3H). ^{13}C { ^1H } NMR (101 MHz, CDCl_3) δ : 192.1, 191.2, 168.1, 167.7, 140.4, 139.6, 136.8, 136.7, 135.5, 135.2, 132.1, 132.0, 131.7, 130.2, 129.9, 129.4, 129.3, 129.1, 128.9, 128.7, 128.7, 128.5, 128.3, 128.2, 127.9, 127.5, 127.4, 127.2, 126.6, 126.3, 126.2, 60.3, 59.6, 52.8, 52.5, 49.7, 48.1. HRMS (ESI) m/z ($\text{M}+\text{Na}$) $^+$ calcd for $\text{C}_{25}\text{H}_{21}\text{O}_3\text{NaBr}$: 471.0572; Found: 471.0578.

Methyl (E)-2-(4-methoxybenzoyl)-3,5-diphenylpent-4-enoate (3na).



Yield: 98 mg, 82% yield; colorless liquid; Rf: 0.4 (petroleum ether/EtOAc = 7/1). IR (KBr): 3028, 2955, 1741, 1676, 1599, 1454, 1261, 1174, 1026, 970, 748, 698, 580 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 8.15-7.90 (m, 2H), 7.43-7.26 (m, 5H), 7.24-7.08 (m, 5H), 6.99-6.83 (m, 2H), 6.57-6.15 (m, 2H), 4.98 (t, $J = 11.2$ Hz, 1H), 4.65-4.52 (m, 1H), 3.85 (d, $J = 12.1$ Hz, 3H), 3.54 (d, $J = 92.9$ Hz, 3H). ^{13}C { ^1H } NMR (101 MHz, CDCl_3) δ : 192.1, 189.9, 168.7, 167.2, 164.0, 163.9, 141.4, 140.5, 137.0, 136.9, 131.6, 131.5, 131.2, 130.9, 129.9, 129.8, 129.8, 129.5, 128.6, 128.4, 128.3, 128.3, 127.9, 127.4, 127.3, 127.0, 126.7, 126.3, 126.2, 114.0, 113.8, 59.2, 58.7, 55.5, 55.4, 52.6, 52.3, 48.8, 48.7. HRMS (ESI) m/z ($\text{M}+\text{Na}$) $^+$ calcd for $\text{C}_{26}\text{H}_{24}\text{O}_4\text{Na}$: 423.1572; Found: 423.1591.

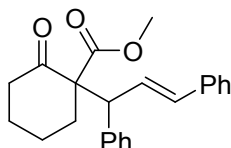
Methyl (E)-2-(2-naphthoyl)-3,5-diphenylpent-4-enoate (3oa).



Yield: 108 mg, 86% yield; colorless liquid; Rf: 0.4 (petroleum ether/EtOAc = 7/1). IR (KBr): 3028, 2953, 1734, 1678, 1628, 1495, 1452, 1358, 1296, 1153, 968, 748, 698 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 8.62 (dd, $J = 53.9, 1.8$ Hz, 1H), 8.16-7.94 (m, 2H), 7.93-7.81 (m, 2H), 7.66-7.60 (m, 1H), 7.59-7.53 (m, 1H), 7.49-7.07 (m, 10H), 5.24 (dd, $J = 12.7, 10.9$ Hz, 1H), 4.76-4.63 (m, 1H),

3.58 (d, $J = 91.3$ Hz, 3H). ^{13}C { ^1H } NMR (101 MHz, CDCl_3) δ : 193.8, 192.4, 168.5, 167.7, 140.7, 140.4, 137.0, 136.8, 135.8, 135.7, 134.2, 133.9, 132.5, 132.0, 131.9, 131.7, 130.8, 130.6, 129.8, 129.8, 129.8, 129.7, 128.9, 128.8, 128.8, 128.7, 128.7, 128.6, 128.5, 128.3, 128.3, 127.9, 127.8, 127.7, 127.5, 127.3, 127.2, 126.9, 126.9, 126.4, 126.3, 124.1, 124.0, 59.7, 59.6, 52.8, 52.5, 49.1, 48.9. HRMS (ESI) m/z ($\text{M}+\text{Na}$) $^+$ calcd for $\text{C}_{29}\text{H}_{24}\text{O}_3\text{Na}$: 443.1623; Found: 443.1619.

Methyl (E)-1-(1,3-diphenylallyl)-2-oxocyclohexane-1-carboxylate (3pa).

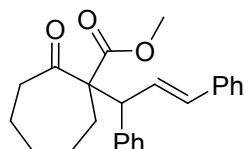


Yield: 80 mg, 76% yield; colorless liquid; Rf: 0.4 (petroleum ether/EtOAc = 7/1).

IR (KBr): 3027, 2931, 1710, 1645, 1496, 1452, 1357, 1234, 970, 748, 700 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 7.44-7.16 (m, 10H), 6.78-6.63 (m, 1H), 6.43 (dd, $J = 15.7, 3.1$ Hz, 1H), 4.22 (dd, $J = 59.7, 9.1$ Hz, 1H), 3.54 (d, $J = 11.5$ Hz, 3H), 2.63

(dq, $J = 12.3, 2.7$ Hz, 1H), 2.53-2.35 (m, 2H), 1.95 (tdd, $J = 9.5, 4.8, 2.3$ Hz, 1H), 1.84-1.47 (m, 4H). ^{13}C { ^1H } NMR (101 MHz, CDCl_3) δ : 206.9, 205.0, 171.6, 171.1, 139.9, 138.9, 137.3, 136.7, 132.4, 130.0, 129.7, 129.1, 128.8, 128.4, 128.4, 128.1, 128.0, 127.3, 127.3, 127.0, 126.8, 126.3, 126.3, 66.2, 66.0, 53.5, 52.2, 52.1, 51.6, 42.0, 34.8, 33.4, 26.9, 26.6, 21.7. HRMS (ESI) m/z ($\text{M}+\text{Na}$) $^+$ calcd for $\text{C}_{23}\text{H}_{24}\text{O}_3\text{Na}$: 371.1623; Found: 371.1621.

Methyl (E)-1-(1,3-diphenylallyl)-2-oxocycloheptane-1-carboxylate (3qa).

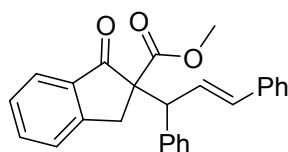


Yield: 92 mg, 85% yield; colorless liquid; Rf: 0.4 (petroleum ether/EtOAc = 7/1).

IR (KBr): 3028, 2937, 1736, 1711, 1599, 1495, 1452, 1223, 1147, 968, 748, 700 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 7.41-7.15 (m, 10H), 6.73 (ddd, $J = 15.7, 12.3, 9.5$ Hz, 1H), 6.42 (dd, $J = 15.7, 5.5$ Hz, 1H), 4.22 (dd, $J = 31.1, 9.4$ Hz, 1H), 3.63

(d, $J = 20.9$ Hz, 3H), 2.56-2.40 (m, 1H), 2.40-2.15 (m, 1H), 2.11-1.87 (m, 2H), 1.69-1.35 (m, 6H). ^{13}C { ^1H } NMR (101 MHz, CDCl_3) δ : 208.8, 208.2, 172.2, 171.8, 139.6, 137.4, 137.2, 132.3, 132.0, 129.7, 129.5, 129.3, 128.8, 128.4, 128.4, 128.2, 128.2, 127.3, 127.2, 127.1, 127.0, 126.3, 68.1, 67.7, 55.5, 54.9, 52.1, 52.0, 44.3, 42.3, 32.9, 31.8, 30.0, 29.8, 25.8, 25.4, 25.3, 25.1. HRMS (ESI) m/z ($\text{M}+\text{Na}$) $^+$ calcd for $\text{C}_{24}\text{H}_{26}\text{O}_3\text{Na}$: 385.1780; Found: 385.1786.

Methyl (E)-2-(1,3-diphenylallyl)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (3ra).

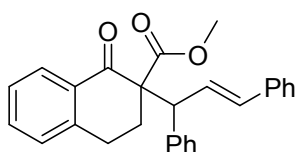


Yield: 102 mg, 88% yield; colorless liquid; Rf: 0.4 (petroleum ether/EtOAc = 7/1). IR (KBr): 3030, 2928, 1741, 1713, 1606, 1495, 1456, 1277, 1236, 1163,

968, 752, 700 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 7.80-7.01 (m, 14H), 6.66-6.48 (m, 1H), 6.45-6.12 (m, 1H), 4.91-4.81 (m, 1H), 4.07-3.97 (m, 1H), 3.74-

3.44 (m, 4H). ^{13}C { ^1H } NMR (101 MHz, CDCl_3) δ : 201.1, 200.1, 169.9, 169.6, 153.4, 153.2, 139.7, 138.3, 137.0, 136.8, 135.5, 135.2, 135.2, 135.1, 133.9, 132.9, 129.4, 128.7, 128.6, 128.6, 128.5, 128.5, 128.3, 128.1, 127.9, 127.7, 127.6, 127.4, 127.4, 127.1, 127.1, 126.4, 126.3, 126.3, 126.0, 124.7, 124.5, 66.4, 65.7, 53.1, 52.9, 52.6, 50.4, 33.6, 32.6. HRMS (ESI) m/z ($\text{M}+\text{Na}$) $^+$ calcd for $\text{C}_{26}\text{H}_{22}\text{O}_3\text{Na}$: 405.1467; Found: 405.1488.

Methyl (E)-2-(1,3-diphenylallyl)-1-oxo-1,2,3,4-tetrahydronaphthalene-2-carboxylate (3sa).



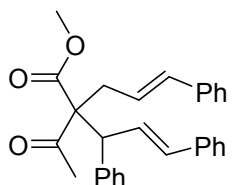
Yield: 92 mg, 79% yield; colorless liquid; Rf: 0.4 (petroleum ether/EtOAc = 7/1). IR (KBr): 3028, 2949, 1732, 1687, 1601, 1450, 1238, 1215, 972, 746,

700 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 8.12 (dd, $J = 7.9, 1.4$ Hz, 1H), 7.53-7.11 (m, 13H), 7.05-6.70 (m, 1H), 6.53 (dd, $J = 19.4, 15.7$ Hz, 1H), 4.53

(dd, $J = 103.9, 9.6$ Hz, 1H), 3.53 (d, $J = 4.9$ Hz, 3H), 3.37-3.03 (m, 1H), 2.90 (dddd, $J = 17.5, 10.1, 4.8, 2.8$ Hz, 1H), 2.69 (tdd, $J = 13.5, 4.5, 2.7$ Hz, 1H), 2.30-2.11 (m, 1H). ^{13}C { ^1H } NMR (101 MHz, CDCl_3) δ : 194.1, 193.1,

170.8, 169.9, 143.0, 142.9, 139.9, 139.4, 137.3, 137.1, 133.5, 133.4, 132.8, 132.4, 132.4, 130.0, 129.9, 129.5, 128.7, 128.4, 128.5, 128.4, 128.3, 128.3, 128.1, 128.1, 127.9, 127.5, 127.3, 127.2, 126.8, 126.7, 126.6, 126.4, 126.4, 63.5, 62.0, 55.4, 53.0, 52.5, 52.4, 30.3, 29.0, 26.9, 26.5, 25.4. **HRMS (ESI) m/z (M+Na)⁺** calcd for C₂₇H₂₄O₃Na: 419.1623; Found: 419.1628.

Methyl (E)-2-acetyl-2-cinnamyl-3,5-diphenylpent-4-enoate (3ta).

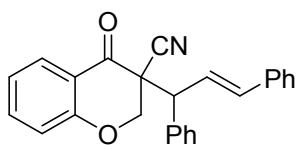


Yield: 30 mg, 23% yield; colorless liquid; Rf: 0.4 (petroleum ether/EtOAc = 7/1).

IR (KBr): 3028, 2951, 1738, 1709, 1601, 1454, 1495, 1448, 1356, 1213, 968, 746, 698 cm⁻¹. **¹H NMR (400 MHz, CDCl₃)** δ 7.39-7.15 (m, 15H), 6.68 (ddd, *J* = 15.7, 9.0, 1.5 Hz, 1H), 6.50-6.29 (m, 2H), 6.07-5.92 (m, 1H), 4.27 (dd, *J* = 8.8, 1.0 Hz, 1H), 3.73 (d, *J* = 55.2 Hz, 3H), 2.92-2.64 (m, 2H), 2.11 (d, *J* = 41.3 Hz, 3H). **¹³C**

{¹H} NMR (101 MHz, CDCl₃) δ: 205.1, 204.7, 171.9, 171.5, 139.5, 139.3, 137.3, 137.0, 137.0, 133.8, 133.7, 132.8, 132.4, 129.3, 129.2, 129.0, 128.6, 128.5, 128.4, 128.4, 128.3, 127.5, 127.3, 127.3, 127.3, 127.3, 127.1, 126.3, 126.3, 126.1, 124.2, 68.4, 67.7, 53.4, 52.7, 52.0, 51.3, 38.2, 37.8, 30.4, 30.1. **HRMS (ESI) m/z (M+Na)⁺** calcd for C₂₉H₂₈O₃Na: 447.1936; Found: 447.1935.

(E)-3-(1,3-diphenylallyl)-4-oxochromane-3-carbonitrile (3ua).

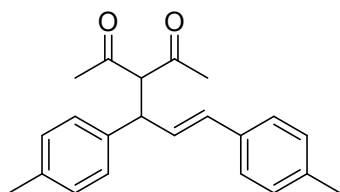


Yield: 88 mg, 80% yield; colorless liquid; Rf: 0.4 (petroleum ether/EtOAc = 7/1).

IR (KBr): 3030, 2924, 2249, 1701, 1606, 1471, 1458, 1302, 1215, 1138, 974, 748, 696 cm⁻¹. **¹H NMR (400 MHz, CDCl₃)** δ 7.88 (dd, *J* = 60.0, 7.7 Hz, 1H), 7.66-7.50 (m, 2H), 7.44 (dt, *J* = 21.0, 8.3 Hz, 3H), 7.26 (s, 4H), 7.24 (d, *J* = 6.3 Hz, 2H), 7.13 (dt, *J* = 9.3, 4.9 Hz, 2H), 6.82- 6.20 (m, 2H), 4.88-4.24 (m, 2H), 4.11 (dd, *J* = 39.4, 9.1 Hz, 1H). **¹³C {¹H} NMR (101 MHz, CDCl₃)** δ 184.6, 184.3, 160.1, 159.9, 137.2, 137.1, 136.8, 136.6, 136.3,

136.0, 136.0, 134.4, 129.2, 129.0, 128.9, 128.7, 128.6, 128.5, 128.5, 128.4, 128.3, 128.3, 128.0, 126.7, 126.7, 125.0, 124.4, 123.0, 123.0, 119.0, 118.3, 117.9, 117.9, 115.5, 115.4, 69.5, 69.3, 54.1, 54.0, 49.6, 48.7. **HRMS (ESI) m/z (M+Na)⁺** calcd for C₂₅H₁₉NO₂Na: 388.1313; Found: 388.1305.

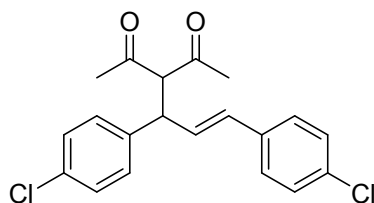
(E)-3-(1,3-di-p-tolylallyl)pentane-2,4-dione (3ab)^[4d].



Yield: 74 mg, 75% yield; white solid; m.p. 70.1-71.3 °C; Rf: 0.5 (petroleum ether/EtOAc = 10/1). **IR (KBr):** 3028, 2922, 1728, 1699, 1645, 1514, 1417, 1360, 1169, 972, 802 cm⁻¹. **¹H NMR (400 MHz, CDCl₃)** δ 7.22-7.11 (m, 6H), 7.12-7.03 (m, 2H), 6.39 (d, *J* = 15.8 Hz, 1H), 6.18-6.08 (m, 1H), 2.31 (s, 6H), 2.25 (s, 3H), 1.94 (s, 3H). **¹³C {¹H} NMR (101 MHz, CDCl₃)** δ 203.1, 202.9, 137.5, 137.1, 136.8, 133.8, 131.3, 129.7, 129.2, 128.4, 127.8, 126.2,

74.6, 48.9, 30.0, 29.8, 21.1, 21.0.

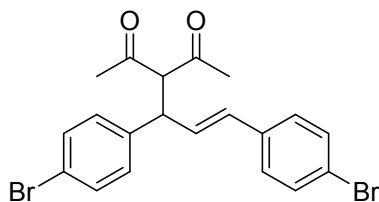
(E)-3-(1,3-bis(4-chlorophenyl)allyl)pentane-2,4-dione (3ac)^[4d].



Yield: 78 mg, 73% yield; white solid; m.p. 83.5-84.0 °C; Rf: 0.5 (petroleum ether/EtOAc = 10/1). **IR (KBr):** 3032, 2922, 1730, 1699, 1491, 144, 1358, 1282, 1147, 1092, 972, 820, 675 cm⁻¹. **¹H NMR (400 MHz, CDCl₃)** δ 7.47-7.41 (m, 2H), 7.41-7.35 (m, 2H), 7.22-6.97 (m, 4H), 6.33 (d, *J* = 15.8 Hz, 1H), 6.12 (ddd, *J* = 15.8, 6.1, 1.8 Hz, 1H), 4.29 (d, *J* = 5.8 Hz, 2H), 2.23 (s, 3H), 1.95 (s,

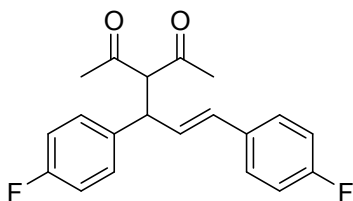
3H). ^{13}C { ^1H } NMR (101 MHz, CDCl_3) δ 202.2, 202.0, 138.9, 135.2, 132.2, 131.8, 131.8, 131.7, 131.0, 129.6, 129.4, 129.0, 127.9, 121.7, 121.3, 74.2, 48.2, 30.0, 29.7.

(E)-3-(1,3-bis(4-bromophenyl)allyl)pentane-2,4-dione (3ad)^[4d].



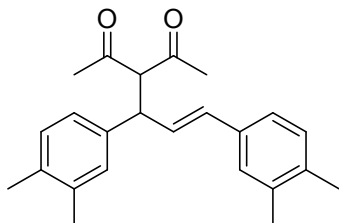
Yield: 107 mg, 80% yield; white solid; m.p. 78.5-79.3 °C; (petroleum ether/EtOAc = 7/1). IR (KBr): 3030, 1726, 1699, 1487, 1412, 1360, 1269, 1142, 972, 810, 658 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 7.46-7.42 (m, 2H), 7.40-7.35 (m, 2H), 7.18-7.08 (m, 4H), 6.33 (d, J = 15.8 Hz, 1H), 6.12 (ddd, J = 15.8, 6.1, 1.8 Hz, 1H), 4.29 (d, J = 5.8 Hz, 2H), 2.23 (s, 3H), 1.95 (s, 3H). ^{13}C { ^1H } NMR (101 MHz, CDCl_3) δ 202.2, 202.0, 138.9, 135.2, 132.2, 131.7, 131.0, 129.6, 129.4, 127.9, 121.7, 121.3, 74.3, 48.2, 30.0, 29.7.

(E)-3-(1,3-bis(4-fluorophenyl)allyl)pentane-2,4-dione (3ae)^[4d].



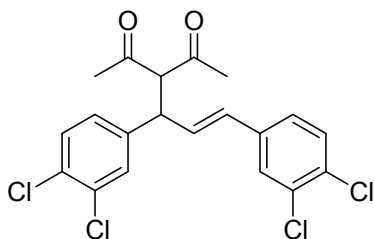
Yield: 74 mg, 76% yield; white solid; m.p. 92.1-93.5 °C; Rf: 0.5 (petroleum ether/EtOAc = 7/1). IR (KBr): 3041, 2924, 1730, 1699, 1603, 1510, 1454, 1358, 1227, 1159, 970, 839, 754, 675 cm^{-1} . ^1H NMR (300 MHz, CDCl_3) δ 7.36-7.16 (m, 4H), 6.98 (dddd, J = 17.2, 8.7, 6.8, 2.1 Hz, 4H), 6.36 (d, J = 15.8 Hz, 1H), 6.14-6.00 (m, 1H), 4.31 (d, J = 7.3 Hz, 2H), 2.24 (s, 3H), 1.93 (s, 3H). ^{13}C { ^1H } NMR (101 MHz, CDCl_3) δ 202.5, 202.3, 163.6, 163.0, 161.1, 160.6, 135.9, 135.8, 132.6, 132.6, 130.6, 129.5, 129.4, 128.8, 128.8, 127.9, 127.9, 116.0, 115.8, 115.6, 115.3, 74.5, 48.1, 30.0, 29.7. ^{19}F NMR (CDCl_3 , 376 MHz): δ -114.0, -114.9.

(E)-3-(1,3-bis(3,4-dimethylphenyl)allyl)pentane-2,4-dione (3af).



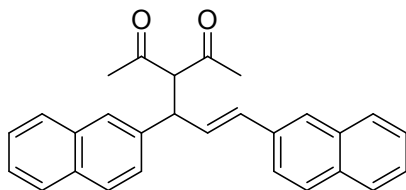
Yield: 42 mg, 41% yield; colorless liquid.; Rf: 0.5 (petroleum ether/EtOAc = 7/1). IR (KBr): 3009, 2922, 1728, 1699, 1605, 1508, 1352, 1153, 970, 818, 742, 675 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 7.15-6.94 (m, 6H), 6.37 (d, J = 15.8 Hz, 1H), 6.12 (dd, J = 15.8, 8.1 Hz, 1H), 4.48-4.15 (m, 2H), 2.26-2.20 (m, 15H), 1.95 (s, 3H). ^{13}C { ^1H } NMR (101 MHz, CDCl_3) δ : 203.2, 203.0, 137.6, 137.1, 136.5, 136.1, 135.4, 134.3, 131.2, 130.1, 129.7, 129.1, 128.3, 127.5, 124.6, 123.8, 74.5, 48.9, 31.3, 29.8, 19.8, 19.6, 19.4, 19.3. HRMS (ESI) m/z ($\text{M}+\text{H}$)⁺ calcd for $\text{C}_{24}\text{H}_{29}\text{O}_2$: 349.2168; Found: 349.2166.

(E)-3-(1,3-bis(3,4-dichlorophenyl)allyl)pentane-2,4-dione (3ag).



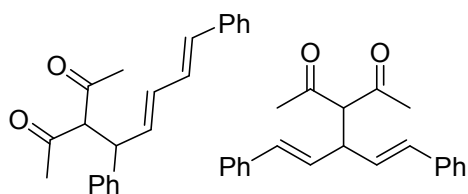
Yield: 70 mg, 54% yield; colorless liquid.; Rf: 0.5 (petroleum ether/EtOAc = 10/1). IR (KBr): 3010, 2924, 1732, 1701, 1591, 1556, 1471, 1358, 1263, 1136, 1030, 968, 883, 816, 708, 673, 621, 577 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ 7.40 (d, J = 8.3 Hz, 1H), 7.37-7.31 (m, 3H), 7.15-7.05 (m, 2H), 6.31 (d, J = 15.8 Hz, 1H), 6.14-6.07 (m, 1H), 4.36-4.23 (m, 2H), 2.23 (s, 3H), 2.00 (s, 3H). ^{13}C { ^1H } NMR (101 MHz, CDCl_3) δ : 201.6, 201.4, 139.9, 136.1, 133.1, 132.7, 131.7, 131.6, 131.4, 130.5, 130.3, 130.0, 129.8, 128.0, 127.2, 125.6, 73.4, 47.6, 30.4, 29.6. HRMS (ESI) m/z ($\text{M}+\text{H}$)⁺ calcd for $\text{C}_{20}\text{H}_{17}\text{Cl}_2\text{O}_2$: 428.9973; Found: 428.9966.

(E)-3-(1,3-di(naphthalen-2-yl)allyl)pentane-2,4-dione (3ah)^[4d].



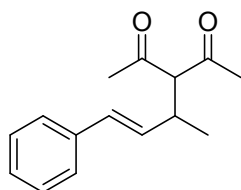
Yield: 75 mg, 64% yield; white solid; m.p. 93.5-94.8 °C; Rf: 0.6 (petroleum ether/EtOAc = 7/1). **IR (KBr):** 3053, 2924, 1726, 1699, 1628, 1599, 1417, 1358, 1269, 1149, 966, 895, 860, 816, 748, 621 cm⁻¹. **¹H NMR (400 MHz, CDCl₃)** δ 7.84-7.71 (m, 7H), 7.65 (s, 1H), 7.53-7.37 (m, 6H), 6.62 (d, *J* = 15.8 Hz, 1H), 6.39 (dd, *J* = 15.8, 7.6 Hz, 1H), 4.63-4.48 (m, 2H), 2.31 (s, 3H), 1.95 (s, 3H). **¹³C {¹H} NMR (101 MHz, CDCl₃)** δ 202.8, 202.6, 137.5, 133.9, 133.6, 133.5, 133.0, 132.6, 132.1, 129.5, 128.9, 128.2, 127.9, 127.8, 127.7, 127.6, 126.7, 126.4, 126.3, 126.0, 125.9, 123.4, 74.5, 49.2, 30.0, 29.9.

3-((2E,4E)-1,5-diphenylpenta-2,4-dien-1-yl)pentane-2,4-dione (3ai)^[4e].



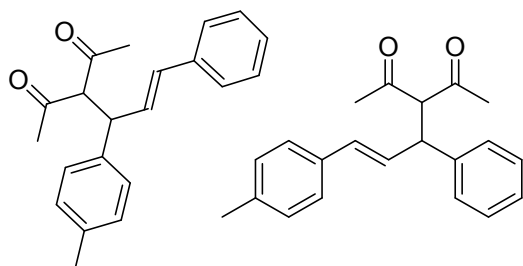
Yield: 51 mg, 59% yield; yellow liquid; Rf: 0.6 (petroleum ether/EtOAc = 10/1). **IR (KBr):** 3028, 2926, 1726, 1699, 1628, 1599, 1493, 1540, 1419, 1358, 1186, 1153, 972, 754, 700, 623 cm⁻¹. **¹H NMR (400 MHz, CDCl₃)** δ 7.37-7.23 (m, 10H), 6.71-6.46 (m, 2H), 6.28-5.79 (m, 2H), 4.35-3.95 (m, 2H), 2.32-1.91 (m, 6H). **¹³C {¹H} NMR (101 MHz, CDCl₃)** δ 202.9, 202.7, 140.0, 137.0, 133.2, 132.8, 132.3, 132.2, 129.0, 128.6, 128.1, 127.9, 127.8, 127.7, 127.6, 127.3, 126.3, 126.3, 74.5, 73.8, 49.0, 46.4, 30.0, 29.7.

(E)-3-(4-phenylbut-3-en-2-yl)pentane-2,4-dione (3aj)^[4d].



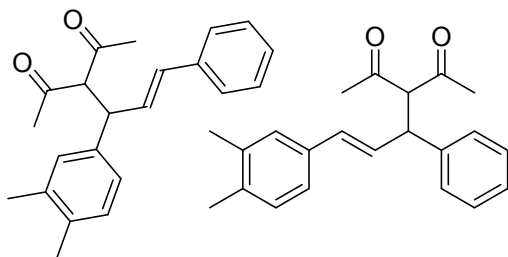
Yield: 14 mg, 20% yield; colorless liquid; Rf: 0.6 (petroleum ether/EtOAc = 7/1). **IR (KBr):** 3026, 2968, 2929, 2875, 1697, 1599, 1547, 1493, 1452, 1421, 1358, 1273, 1190, 1156, 970, 750, 696 cm⁻¹. **¹H NMR (300 MHz, CDCl₃)** δ 7.34-7.23 (m, 6H), 6.44 (dd, *J* = 15.9, 0.9 Hz, 1H), 6.00 (dd, *J* = 15.9, 8.5 Hz, 1H), 3.69 (d, *J* = 10.4 Hz, 1H), 3.28-3.14 (m, 1H), 2.23 (s, 3H), 2.13 (s, 3H), 1.09 (d, *J* = 6.7 Hz, 3H). **¹³C {¹H} NMR (101 MHz, CDCl₃)** δ 203.6, 203.5, 136.8, 131.0, 130.9, 128.6, 128.5, 127.6, 126.2, 75.6, 37.9, 30.0, 29.7, 18.8.

(E)-3-(3-phenyl-1-(p-tolyl)allyl)pentane-2,4-dione (3ak)^[4d].



Yield: 70 mg, 80% yield; white solid mixture; Rf: 0.5 (petroleum ether/EtOAc = 7/1). **IR (KBr):** 3028, 2949, 2920, 1726, 1701, 1601, 1514, 1417, 1360, 1275, 1171, 1142, 972, 816, 860, 816, 764, 739, 698, 623 cm⁻¹. **¹H NMR (400 MHz, CDCl₃)** δ 7.34-7.27 (m, 3H), 7.25-7.11 (m, 5H), 7.08 (d, *J* = 7.9 Hz, 1H), 6.41 (dd, *J* = 15.8, 6.8 Hz, 1H), 6.24-6.09 (m, 1H), 4.32 (t, *J* = 6.4 Hz, 2H), 2.31 (s, 3H), 2.25 (s, 3H), 1.93 (d, *J* = 5.1 Hz, 3H). **¹³C {¹H} NMR (101 MHz, CDCl₃)** δ: 203.0, 202.9, 202.8, 202.8, 140.2, 137.5, 136.9, 136.9, 136.6, 133.7, 131.5, 131.4, 129.7, 129.4, 129.1, 129.0, 128.5, 128.3, 128.1, 127.9, 127.76, 127.6, 127.2, 126.3, 126.2, 125.9, 74.5, 49.2, 48.8, 30.0, 30.0, 29.7, 29.7, 21.1, 21.0.

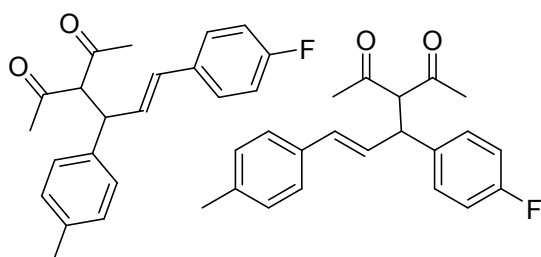
(E)-3-(1-(3,4-dimethylphenyl)-3-phenylallyl)pentane-2,4-dione (3al).



Yield: 59 mg, 61% yield; colorless liquid; Rf: 0.4 (petroleum ether/EtOAc = 7/1). **IR (KBr):** 3026, 2924, 1728, 1699, 1603, 1498, 1450, 1356, 1269, 1153, 968, 822, 762, 698, 617 cm^{-1} . **^1H NMR (400 MHz, CDCl_3)** δ 7.42-7.18 (m, 5H), 7.18-6.99 (m, 3H), 6.44 (t, J = 16.3 Hz, 1H), 6.27-6.13 (m, 1H), 4.43-4.26 (m, 2H), 2.25 (dd, 9H), 1.97 (d, J = 11.4 Hz, 3H). **^{13}C { ^1H }**

NMR (101 MHz, CDCl_3) δ : 203.0, 202.9, 202.8, 202.8, 140.3, 137.5, 137.2, 136.7, 136.6, 136.2, 135.5, 134.2, 131.6, 131.3, 130.2, 129.8, 129.7, 129.2, 129.0, 128.5, 128.0, 127.9, 127.6, 127.6, 127.2, 126.3, 125.1, 123.9, 74.6, 74.4, 49.2, 48.1, 30.0, 30.0, 29.8, 29.7, 19.9, 19.7, 19.5, 19.3. **HRMS (ESI) m/z ($\text{M}+\text{Na}$) $^+$** calcd for $\text{C}_{22}\text{H}_{24}\text{O}_2\text{Na}$: 343.1674; Found: 343.1678.

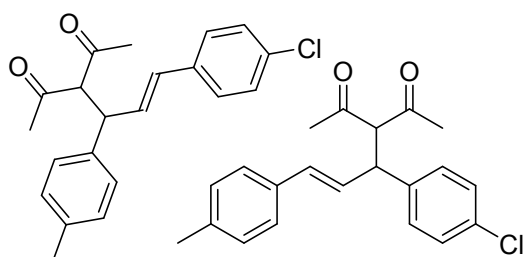
(E)-3-(3-(4-fluorophenyl)-1-(p-tolyl)allyl)pentane-2,4-dione (3am).



Yield: 80 mg, 82% yield; colorless liquid; Rf: 0.4 (petroleum ether/EtOAc = 7/1). **IR (KBr):** 3026, 2924, 1730, 1699, 1603, 1510, 1419, 1358, 1227, 1157, 970, 835, 812, 750, 723, 619 cm^{-1} . **^1H NMR (400 MHz, CDCl_3)** δ 7.24-7.07 (m, 6H), 7.00 (dd, J = 16.5, 7.9 Hz, 2H), 6.39 (dd, J = 15.8, 3.5 Hz, 1H), 6.13 (dt, J = 15.4, 7.1 Hz, 1H), 4.39-4.26 (m, 2H),

2.31 (s, 3H), 2.25 (d, J = 2.4 Hz, 3H), 1.94 (s, 3H). **^{13}C { ^1H }** **NMR (101 MHz, CDCl_3)** δ : 202.9, 202.7, 202.6, 202.5, 163.5, 163.0, 161.0, 160.5, 137.7, 136.9, 136.1, 136.0, 133.6, 132.8, 132.8, 131.6, 130.2, 129.8, 129.7, 129.5, 129.4, 129.3, 129.2, 129.1, 127.9, 127.9, 127.8, 127.7, 126.2, 125.8, 115.9, 115.7, 115.5, 115.3, 115.1, 114.9, 74.6, 74.4, 48.4, 48.2, 29.8, 29.6, 21.1, 21.0. **^{19}F NMR (CDCl_3 , 376MHz):** δ -114.2, -115.0. **HRMS (ESI) m/z ($\text{M}+\text{Na}$) $^+$** calcd for $\text{C}_{21}\text{H}_{21}\text{O}_2\text{NaF}$: 347.1423; Found: 347.1426.

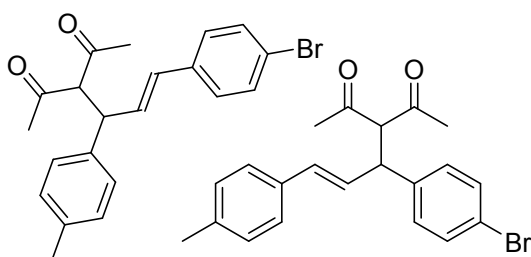
(E)-3-(3-(4-chlorophenyl)-1-(p-tolyl)allyl)pentane-2,4-dione (3an)^[4d].



Yield: 82 mg, 81% yield; white solid mixture; Rf: 0.4 (petroleum ether/EtOAc = 7/1). **IR (KBr):** 3028, 2924, 1730, 1699, 1512, 1491, 1358, 1282, 1149, 972, 812, 723, 636 cm^{-1} . **^1H NMR (400 MHz, CDCl_3)** δ 7.34-7.07 (m, 8H), 6.39 (dd, J = 15.8, 8.5 Hz, 1H), 6.14 (dddd, J = 25.0, 15.7, 6.7, 1.2 Hz, 1H), 4.38-4.26 (m, 2H), 2.33 (s, 3H), 2.26 (s, 3H), 1.96 (d,

J = 6.8 Hz, 3H). **^{13}C { ^1H }** **NMR (101 MHz, CDCl_3)** δ : 202.8, 202.6, 202.5, 202.3, 138.8, 137.7, 137.0, 136.7, 135.0, 133.5, 133.2, 132.9, 131.9, 130.2, 130.1, 129.7, 129.2, 129.2, 129.1, 128.6, 127.7, 127.5, 126.2, 74.5, 74.4, 48.6, 48.3, 30.0, 29.9, 29.8, 29.6, 21.1, 21.0.

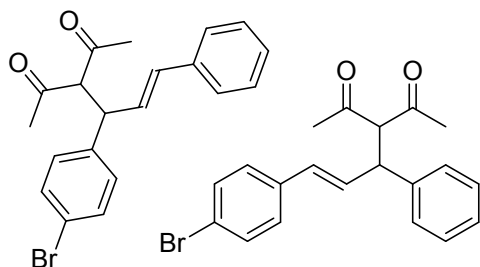
(E)-3-(3-(4-bromophenyl)-1-(p-tolyl)allyl)pentane-2,4-dione (3ao).



Yield: 99 mg, 86% yield; white solid mixture; Rf: 0.4 (petroleum ether/EtOAc = 7/1). **IR (KBr):** 3020, 2920, 1730, 1699, 1512, 1485, 1417, 1358, 1284, 1146, 1070, 1009, 972, 810, 721, 663, 629 cm^{-1} . **^1H NMR (400 MHz, CDCl_3)** δ 7.50-7.26 (m, 2H),

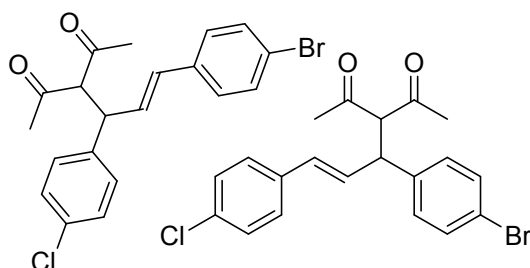
7.23-7.07 (m, 6H), 6.38 (t, $J = 15.5$ Hz, 1H), 6.25-6.06 (m, 1H), 4.39-4.26 (m, 2H), 2.33 (s, 3H), 2.26 (d, $J = 1.7$ Hz, 3H), 1.96 (d, $J = 7.7$ Hz, 3H). ^{13}C { ^1H } NMR (101 MHz, CDCl_3) δ : 202.8, 202.5, 202.4, 202.3, 139.3, 137.7, 137.0, 136.6, 135.5, 133.5, 132.0, 131.9, 131.5, 130.3, 130.2, 129.7, 129.6, 129.2, 127.8, 127.7, 127.4, 126.2, 121.4, 121.0, 74.4, 74.4, 48.7, 48.4, 30.0, 29.9, 29.8, 29.6, 21.1, 21.0. HRMS (ESI) m/z ($\text{M}+\text{Na}$) $^+$ calcd for $\text{C}_{21}\text{H}_{21}\text{O}_2\text{NaBr}$: 407.0623; Found: 407.0620.

(E)-3-(1-(4-bromophenyl)-3-phenylallyl)pentane-2,4-dione (3ap).



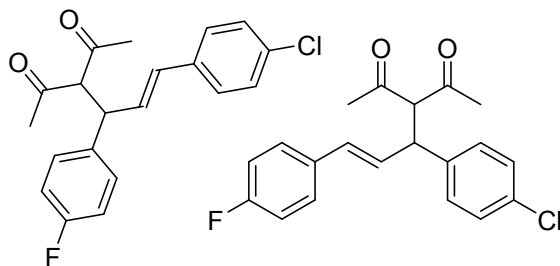
Yield: 88 mg, 82% yield; white solid mixture; Rf: 0.5 (petroleum ether/EtOAc = 7/1). IR (KBr): 3028, 2918, 1724, 1699, 1489, 1360, 1273, 1173, 1140, 1072, 1009, 972, 818, 762, 702 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ : 7.49-7.18 (m, 7H), 7.13 (d, $J = 8.5$ Hz, 2H), 6.39 (dd, $J = 22.8, 15.8$ Hz, 1H), 6.24-6.09 (m, 1H), 4.39-4.26 (m, 2H), 2.24 (d, $J = 1.8$ Hz, 3H), 1.94 (d, $J = 12.6$ Hz, 3H). ^{13}C { ^1H } NMR (101 MHz, CDCl_3) δ : 202.6, 202.4, 202.3, 202.2, 139.7, 139.2, 136.3, 135.4, 132.1, 132.0, 131.6, 130.5, 130.1, 129.6, 129.1, 128.5, 128.5, 127.9, 127.9, 127.3, 126.3, 121.4, 74.3, 74.3, 49.0, 48.3, 30.1, 30.0, 29.8, 29.6. HRMS (ESI) m/z ($\text{M}+\text{Na}$) $^+$ calcd for $\text{C}_{20}\text{H}_{19}\text{O}_2\text{NaBr}$: 393.0466; Found: 393.0464.

(E)-3-(3-(4-bromophenyl)-1-(4-chlorophenyl)allyl)pentane-2,4-dione (3aq).



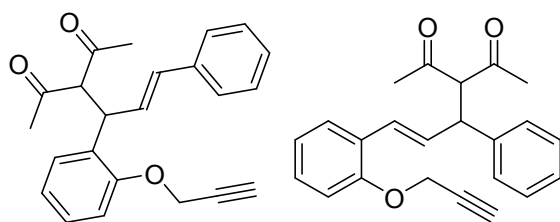
Yield: 90 mg, 75% yield; white solid mixture; Rf: 0.4 (petroleum ether/EtOAc = 7/1). IR (KBr): 3030, 2924, 1730, 1489, 1412, 1358, 1282, 1146, 1090, 1011, 970, 818, 717, 629 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ : 7.42 (dd, $J = 25.3, 8.4$ Hz, 2H), 7.30 (d, $J = 8.4$ Hz, 1H), 7.27-7.17 (m, 3H), 7.14 (dd, $J = 8.6, 2.0$ Hz, 2H), 6.36 (dd, $J = 15.8, 6.2$ Hz, 1H), 6.14 (dt, $J = 15.8, 8.2$ Hz, 1H), 4.33 (d, $J = 6.4$ Hz, 2H), 2.24 (s, 3H), 1.96 (s, 3H). ^{13}C { ^1H } NMR (101 MHz, CDCl_3) δ : 202.2, 202.0, 138.9, 138.3, 135.2, 134.8, 133.4, 133.1, 132.1, 131.6, 130.9, 130.8, 129.6, 129.4, 129.3, 129.2, 129.2, 128.7, 127.9, 127.5, 121.6, 121.2, 74.2, 74.2, 48.2, 48.1, 30.0, 29.7. HRMS (ESI) m/z ($\text{M}+\text{Na}$) $^+$ calcd for $\text{C}_{20}\text{H}_{18}\text{O}_2\text{NaClBr}$: 427.0076; Found: 427.0074.

(E)-3-(3-(4-chlorophenyl)-1-(4-fluorophenyl)allyl)pentane-2,4-dione (3ar).



Yield: 85 mg, 83% yield; white solid mixture; Rf: 0.4 (petroleum ether/EtOAc = 7/1). IR (KBr): 1730, 1701, 1601, 1510, 1416, 1358, 1281, 1225, 1159, 1090, 1011, 974, 823, 764, 600 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ : 7.33-7.17 (m, 6H), 7.09-6.93 (m, 2H), 6.38 (dd, $J = 15.8, 7.0$ Hz, 1H), 6.20-6.03 (m, 1H), 4.40-4.27 (m, 2H), 2.25 (s, 3H), 1.96 (d, $J = 4.9$ Hz, 3H). ^{13}C { ^1H } NMR (101 MHz, CDCl_3) δ : 202.4, 202.3, 202.2, 202.1, 138.5, 135.6, 135.5, 134.8, 133.4, 133.1, 132.4, 130.8, 130.6, 129.6, 129.5, 129.4, 129.2, 129.2, 128.7, 128.4, 128.3, 127.9, 127.8, 127.5, 116.0, 115.8, 115.6, 115.3, 74.5, 74.4, 48.2, 48.1, 30.0, 29.9, 29.6, 29.6. ^{19}F NMR (CDCl_3 , 376 MHz) δ : -113.8, -114.8. HRMS (ESI) m/z ($\text{M}+\text{Na}$) $^+$ calcd for $\text{C}_{20}\text{H}_{18}\text{O}_2\text{NaClF}$: 367.0877; Found: 367.0876.

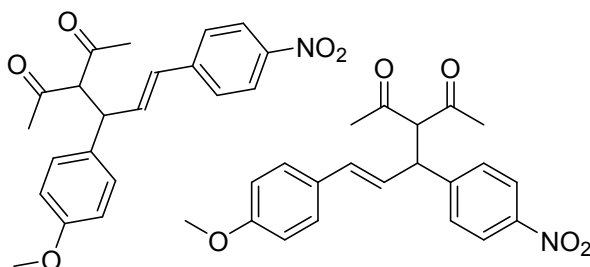
(E)-3-(3-phenyl-1-(2-(prop-2-yn-1-yl)oxy)phenyl)pentane-2,4-dione (3as).



Yield: 77 mg, 75% yield; colorless liquid; Rf: 0.4 (petroleum ether/EtOAc = 5/1). **IR (KBr):** 3294, 3030, 2924, 2121, 1724, 1699, 1601, 1491, 1452, 1360, 1271, 1219, 1142, 1024, 980, 924, 756, 694, 631 cm^{-1} . **^1H NMR (400 MHz, CDCl_3)** δ 7.47-7.15 (m, 7H), 7.04-6.93 (m, 2H), 6.58 (dd, 1H), 6.37-6.24

(m, 1H), 4.76 (dd, $J = 43.0, 2.4$ Hz, 2H), 4.70-4.35 (m, 2H), 2.54 (t, $J = 2.4$ Hz, 1H), 2.27 (s, 3H), 1.98 (d, $J = 19.7$ Hz, 3H). **^{13}C { ^1H } NMR (101 MHz, CDCl_3)** δ : 203.3, 202.9, 154.8, 154.7, 140.3, 136.8, 131.8, 130.1, 129.5, 128.9, 128.7, 128.5, 128.4, 128.2, 127.9, 127.4, 127.1, 126.9, 126.3, 126.1, 122.0, 121.6, 112.8, 112.7, 78.5, 78.4, 75.6, 75.6, 74.6, 72.6, 56.3, 56.0, 48.9, 44.4, 30.5, 30.2, 29.6, 29.1. **HRMS (ESI) m/z ($\text{M}+\text{Na}$) $^+$** calcd for $\text{C}_{23}\text{H}_{22}\text{O}_3\text{Na}$: 369.1467; Found: 369.1467.

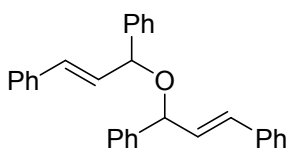
(E)-3-(1-(4-methoxyphenyl)-3-(4-nitrophenyl)allyl)pentane-2,4-dione (3at)^[4f].



Yield: 93 mg, 85% yield; yellow liquid; Rf: 0.5 (petroleum ether/EtOAc = 3/1). **IR (KBr):** 3003, 2935, 2839, 1728, 1699, 1643, 1601, 1516, 1419, 1344, 1254, 1180, 1153, 1111, 1032, 972, 831, 741, 694 cm^{-1} . **^1H NMR (300 MHz, CDCl_3)** δ 8.25-8.04 (m, 2H), 7.38 (d, $J = 8.9$ Hz, 2H), 7.16 (d, $J =$

8.7 Hz, 2H), 6.85 (d, $J = 8.7$ Hz, 2H), 6.50-6.26 (m, 2H), 4.33 (d, $J = 3.3$ Hz, 3H), 2.24 (s, 3H), 1.93 (d, 3H). **^{13}C { ^1H } NMR (101 MHz, CDCl_3)** δ : 202.5, 202.2, 201.8, 201.6, 158.8, 158.7, 148.2, 146.8, 143.1, 143.1, 134.6, 134.3, 130.9, 129.7, 129.3, 129.1, 129.1, 129.0, 128.8, 128.6, 128.6, 128.5, 128.2, 127.6, 126.8, 125.0, 124.1, 124.0, 123.9, 123.7, 123.6, 74.1, 55.2, 55.2, 48.5, 48.1, 30.1, 29.9, 29.8, 29.6.

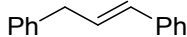
bis(1,3-diphenylallyl) ether (4aa)^[4g].



The target compound was obtained according to general procedure, but the reaction temperature is 30 $^{\circ}\text{C}$, and the crude product was purified by silica gel chromatography using petroleum ether/EtOAc = 100/1 as an eluent to give pure product **4aa** (30 mg, 26% yield) as a yellow liquid; Rf: 0.5

(petroleum ether/EtOAc = 70/1). **IR (KBr):** 3055, 2924, 1591, 1479, 1433, 1090, 854, 744, 696 cm^{-1} . **^1H NMR (400 MHz, CDCl_3)** δ 7.49-7.26 (m, 20H), 6.63 (dd, $J = 15.7, 4.2$ Hz, 2H), 6.38 (ddd, $J = 20.9, 15.9, 7.0$ Hz, 2H), 5.13 (t, $J = 7.7$ Hz, 2H). **^{13}C { ^1H } NMR (101 MHz, CDCl_3)** δ 141.3, 141.2, 136.6, 136.6, 131.6, 131.4, 130.5, 130.3, 128.6, 128.5, 127.8, 127.7, 127.7, 127.1, 126.7, 126.6, 126.6, 79.2, 79.1.

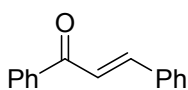
(E)-prop-1-ene-1,3-diylidibenzene (5aa)^[4g].



Compound **4aa** (0.3 mmol), $\text{B}(\text{C}_6\text{F}_5)_3$ (3 mol%), H_2O (1.5 mL) were added to the reaction tube and the reaction mixture was stirred at 100 $^{\circ}\text{C}$ in oil bath for 1 h. and the

crude product was purified by silica gel chromatography using petroleum ether/EtOAc = 200/1 as an eluent to give pure product **5aa** (20 mg, 26% yield) as a colorless liquid; Rf: 0.4 (petroleum ether/EtOAc = 100/1). **IR (KBr):** 3028, 1603, 1498, 1450, 1227, 966, 742, 696 cm^{-1} . **^1H NMR (300 MHz, CDCl_3)** δ 7.46-7.20 (m, 10H), 6.58-6.34 (m, 2H), 3.61 (d, $J = 6.3$ Hz, 2H). **^{13}C { ^1H } NMR (101 MHz, CDCl_3)** δ 140.2, 137.6, 133.4, 131.2, 129.3, 128.8, 128.6, 127.2, 126.3, 126.2, 39.4.

(E)-chalcone (6aa)^[4g].

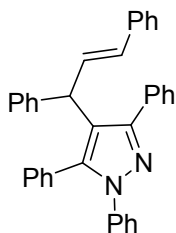


Compound **4aa** (0.3 mmol), B(C₆F₅)₃ (3 mol%), H₂O (1.5 mL) were added to the reaction tube and the reaction mixture was stirred at 100 °C in oil bath for 1 h. and the crude product was purified by silica gel chromatography using petroleum ether/EtOAc = 100/1 as an eluent to give pure product **6aa** (12 mg, 20% yield) as a yellow solid; Rf: 0.4 (petroleum ether/EtOAc = 50/1). **IR (KBr):** 3061, 1664, 1605, 1448, 1336, 1215, 1016, 982, 787, 748, 690 cm⁻¹. **¹H NMR (300 MHz, CDCl₃)** δ 8.07-7.98 (m, 2H), 7.82 (d, *J* = 15.7 Hz, 1H), 7.70-7.48 (m, 6H), 7.47-7.38 (m, 3H). **¹³C {¹H} NMR (101 MHz, CDCl₃)** δ 190.5, 144.8, 138.2, 134.9, 132.8, 130.6, 129.0, 128.6, 128.5, 128.5, 122.1.

Scale-up of the Transformation on a 10 mmol Scale.

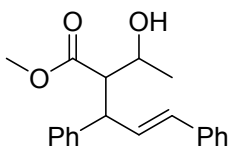
Acetylacetone (1 mol), allyl alcohol (1 mol), B(C₆F₅)₃ (3 mol%), H₂O (20 mL) were added to the reaction tube and the reaction mixture was stirred at 100 °C in oil bath for 1 h. The reaction was monitored by TLC. When the reaction was finished, cooled to room temperature, extraction with ethyl acetate was done three times, the organic phase was merged and dried with anhydrous MgSO₄, and vacuum evaporated. Further purification was carried out via silica gel column chromatography and eluted with ethyl acetate and petroleum ether.

Synthesis of 4 according to the Following Procedure.



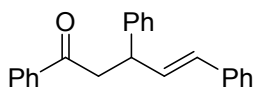
3ca (0.3 mmol) and phenylhydrazine (1.2 eq) in acetic acid (1.5 mL) at 100 °C for one hour. And the crude product was purified by silica gel chromatography using petroleum ether/EtOAc = 100/1 as an eluent to give pure product **4** (101 mg, 69% yield) as a colorless liquid; Rf: 0.5 (petroleum ether/EtOAc = 50/1). **IR (KBr):** 3059, 2928, 1597, 1497, 1448, 1358, 1155, 1072, 968, 744, 696 cm⁻¹. **¹H NMR (300 MHz, CDCl₃)** δ 7.58 (dd, *J* = 6.6, 3.0 Hz, 2H), 7.39-7.08 (m, 23H), 6.47-6.28 (m, 2H), 5.05 (d, *J* = 6.3 Hz, 1H). **¹³C {¹H} NMR (101 MHz, CDCl₃)** δ: 151.8, 143.9, 141.7, 140.0, 137.3, 133.8, 131.5, 131.4, 131.0, 130.6, 129.0, 128.6, 128.4, 128.4, 128.3, 128.2, 128.2, 128.1, 127.8, 127.2, 126.8, 126.2, 126.1, 124.8, 120.6, 44.2. **HRMS (ESI) m/z (M+Na)⁺** calcd for C₃₆H₂₈N₂Na: 511.2150; Found: 511.2143.

Synthesis of 5 according to the Following Procedure.



3ja (0.2 mmol) and NaBH₄ (0.5 eq) in MeOH (1.5 mL) at 0 °C for one hour. And the crude product was purified by silica gel chromatography using petroleum ether/EtOAc = 7/1 as an eluent to give pure product **5** (31 mg, 51% yield) as a colorless liquid; Rf: 0.5 (petroleum ether/EtOAc = 50/1). **IR (KBr):** 3028, 2968, 1718, 1601, 1495, 1452, 1267, 1201, 1165, 1130, 1032, 968, 748, 698 cm⁻¹. **¹H NMR (400 MHz, CDCl₃)** δ 7.42-7.18 (m, 10H), 6.75-6.22 (m, 2H), 4.18-3.99 (m, 2H), 3.54 (d, *J* = 109.8 Hz, 3H), 2.87 (ddd, *J* = 11.2, 5.2, 2.9 Hz, 1H), 1.22 (dd, *J* = 43.7, 6.5 Hz, 3H). **¹³C {¹H} NMR (101 MHz, CDCl₃)** δ: 174.8, 173.7, 142.0, 141.0, 137.1, 136.9, 132.3, 130.9, 130.5, 129.8, 128.9, 128.5, 128.4, 128.4, 127.9, 127.8, 127.5, 127.4, 126.9, 126.8, 126.3, 126.2, 77.3, 77.0, 76.7, 65.9, 65.5, 57.5, 56.4, 51.6, 50.7, 49.0, 22.6, 22.4. **HRMS (ESI) m/z (M+H)⁺** calcd for C₂₀H₂₃O₃: 311.1640; Found: 311.1647.

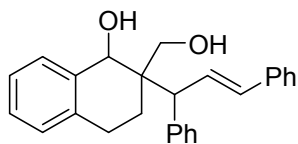
Synthesis of 6^[4h] according to the Following Procedure.



3ka (0.2 mmol) in 20% NaOH (3 mL) at 100 °C for 5 h. And the crude product was purified by silica gel chromatography using petroleum ether/EtOAc = 30/1 as an eluent to give pure product **6** (34 mg, 56% yield) as a yellow liquid; Rf:

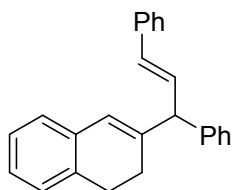
0.5 (petroleum ether/EtOAc = 15/1). **IR (KBr):** 3030, 1678, 1597, 1493, 1448, 1417, 1230, 968, 750, 692, cm^{-1} . **^1H NMR (400 MHz, CDCl_3)** δ 8.00-7.93 (m, 2H), 7.58 (t, $J = 7.4$ Hz, 1H), 7.47 (t, $J = 7.5$ Hz, 2H), 7.38-7.14 (m, 10H), 6.42 (d, 2H), 4.33 (td, $J = 6.8, 4.5$ Hz, 1H), 3.61-3.45 (m, 2H). **^{13}C NMR (101 MHz, CDCl_3)** δ 198.2, 143.3, 137.2, 137.2, 133.1, 132.6, 130.1, 128.7, 128.6, 128.4, 128.1, 127.8, 127.2, 126.6, 126.2, 44.5, 43.9.

Synthesis of 7 according to the Following Procedure.



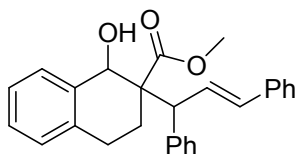
3sa (0.5 mmol) and diisobutylaluminum hydride (4 eq) in THF (5 mL) at -20 °C for 12 hour. And the crude product was purified by silica gel chromatography using petroleum ether/EtOAc = 7/1 as an eluent to give pure product **7** (151 mg, 82% yield) as a white solid; m.p. 82.5-83.6 °C; Rf: 0.5 (petroleum ether/EtOAc = 3/1). **IR (KBr):** 1703, 1635, 1103, 804, 746, 698 cm^{-1} . **^1H NMR (400 MHz, CDCl_3)** δ 7.48 (dd, $J = 27.7, 5.9$ Hz, 2H), 7.40-7.13 (m, 12H), 6.78 (dd, $J = 15.0, 9.3$ Hz, 1H), 6.35 (dd, $J = 40.6, 15.7$ Hz, 1H), 4.58 (d, $J = 122.8$ Hz, 1H), 4.02 (dd, $J = 133.5, 10.1$ Hz, 1H), 3.65-3.48 (m, 2H), 3.04-2.91 (m, 1H), 2.78 (ddt, $J = 26.1, 17.4, 8.2$ Hz, 1H), 2.06 (d, $J = 71.7$ Hz, 1H), 1.86 (dt, $J = 10.1, 5.5$ Hz, 1H), 1.69 (s, 1H), 1.54 (dd, $J = 13.1, 6.9$ Hz, 1H). **^{13}C { ^1H } NMR (101 MHz, CDCl_3)** δ : 141.5, 141.0, 137.8, 137.3, 135.9, 135.6, 132.6, 131.5, 131.4, 131.2, 129.6, 129.5, 128.8, 128.5, 128.4, 128.3, 128.3, 127.8, 127.7, 127.4, 127.2, 127.0, 126.6, 126.5, 126.4, 126.3, 126.3, 126.2, 73.4, 72.7, 65.6, 65.1, 53.4, 50.8, 45.0, 25.8, 25.4, 24.4, 24.4. **HRMS (ESI) m/z ($\text{M}+\text{Na}$) $^+$** calcd for $\text{C}_{26}\text{H}_{26}\text{O}_2\text{Na}$: 393.1830; Found: 393.1831.

Synthesis of 8 according to the Following Procedure.



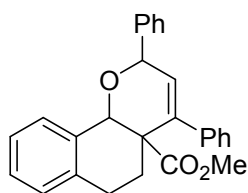
7 (0.2 mmol) and *p*-TsOH (1 eq) in toluene (2 mL) and DMSO (0.3 mL) at 80 °C for 12 hour. And the crude product was purified by silica gel chromatography using petroleum ether/EtOAc = 150/1 as an eluent to give pure product **8** (48 mg, 75% yield) as a colorless liquid; Rf: 0.5 (petroleum ether/EtOAc = 25/1). **IR (KBr):** 3059, 3026, 2929, 1599, 1491, 1450, 1072, 1030, 970, 750, 698 cm^{-1} . **^1H NMR (400 MHz, CDCl_3)** δ 7.49-7.26 (m, 10H), 7.21-7.05 (m, 4H), 6.62 (dd, $J = 15.9, 7.4$ Hz, 1H), 6.50-6.40 (m, 2H), 4.38 (d, $J = 7.3$ Hz, 1H), 2.83 (t, $J = 7.2$ Hz, 2H), 2.26 (t, $J = 8.1$ Hz, 2H). **^{13}C { ^1H } NMR (101 MHz, CDCl_3)** δ : 143.0, 141.8, 137.7, 134.6, 134.5, 131.4, 131.0, 128.6, 128.5, 128.5, 127.3, 127.2, 126.6, 126.6, 126.4, 126.3, 126.0, 124.3, 55.6, 28.3, 26.7. **HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$** calcd for $\text{C}_{25}\text{H}_{23}$: 323.1800; Found: 323.1800.

Synthesis of 9 according to the Following Procedure.



3sa (0.5 mmol) and diisobutylaluminum hydride (2 eq) in THF (5 mL) at -20 °C for 2 hour. And the crude product was purified by silica gel chromatography using petroleum ether/EtOAc = 20/1 as an eluent to give pure product **9** (151 mg, 76% yield) as a white solid; m.p. 123.4-124.1 °C; Rf: 0.3 (petroleum ether/EtOAc = 7/1). **IR (KBr):** 3730, 3508, 3028, 2931, 1703, 1601, 1510, 1452, 1356, 1225, 1016, 970, 810, 746, 698 cm^{-1} . **^1H NMR (300 MHz, CDCl_3)** δ 7.45 (t, $J = 7.3$ Hz, 2H), 7.37-7.30 (m, 5H), 7.29-7.15 (m, 6H), 7.09 (d, $J = 7.3$ Hz, 1H), 6.92 (ddd, $J = 31.7, 15.6, 10.3$ Hz, 1H), 6.70-6.49 (m, 1H), 4.92 (d, $J = 127.8$ Hz, 1H), 4.08 (dd, $J = 66.4, 10.3$ Hz, 1H), 3.48 (s, 3H), 2.89-2.74 (m, 2H), 2.48-1.98 (m, 2H), 1.64 (s, 1H). **^{13}C { ^1H } NMR (101 MHz, CDCl_3)** δ 173.2, 173.0, 140.6, 140.3, 138.0, 137.4, 137.3, 137.0, 135.9, 135.3, 132.4, 132.2, 130.1, 129.8, 129.2, 129.1, 129.0, 128.9, 128.6, 128.5, 128.4, 128.3, 128.2, 128.1, 128.0, 127.3, 127.2, 126.9, 126.8, 126.4, 126.3, 126.3, 126.1, 69.9, 68.5, 55.1, 54.6, 54.0, 52.6, 51.2, 26.2, 26.0, 24.8, 23.5. **HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$** calcd for $\text{C}_{27}\text{H}_{27}\text{O}_3$: 399.1960; Found: 399.1966.

Synthesis of **10** according to the Following Procedure.

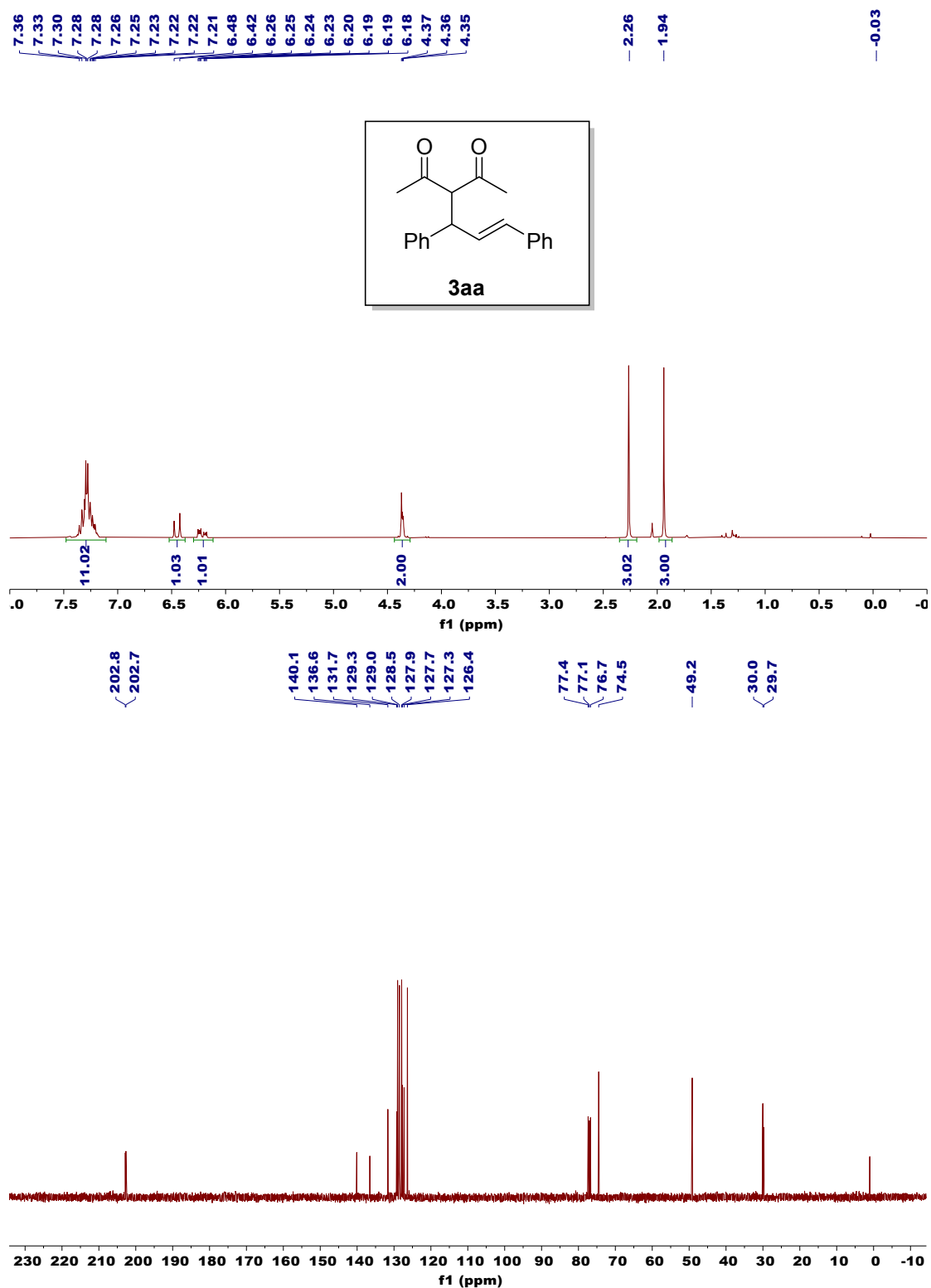


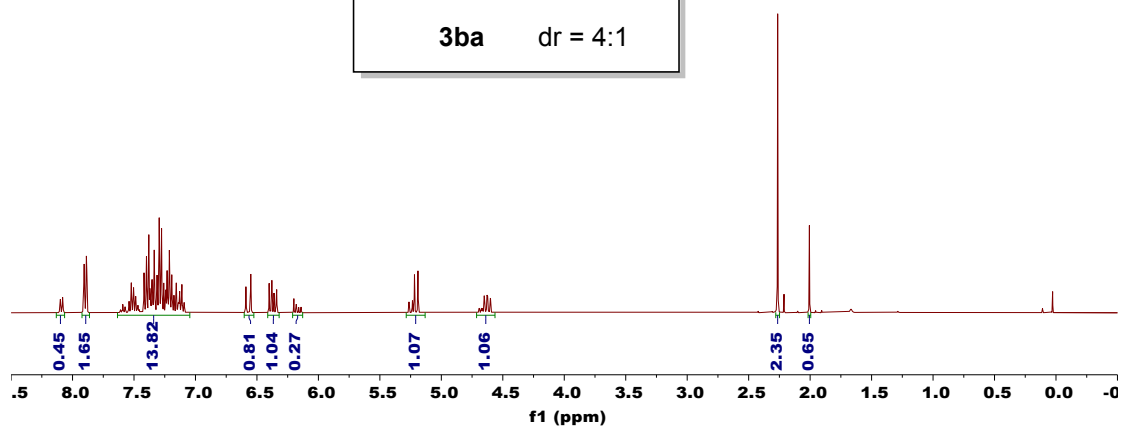
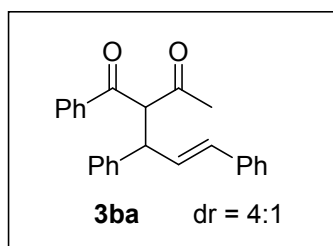
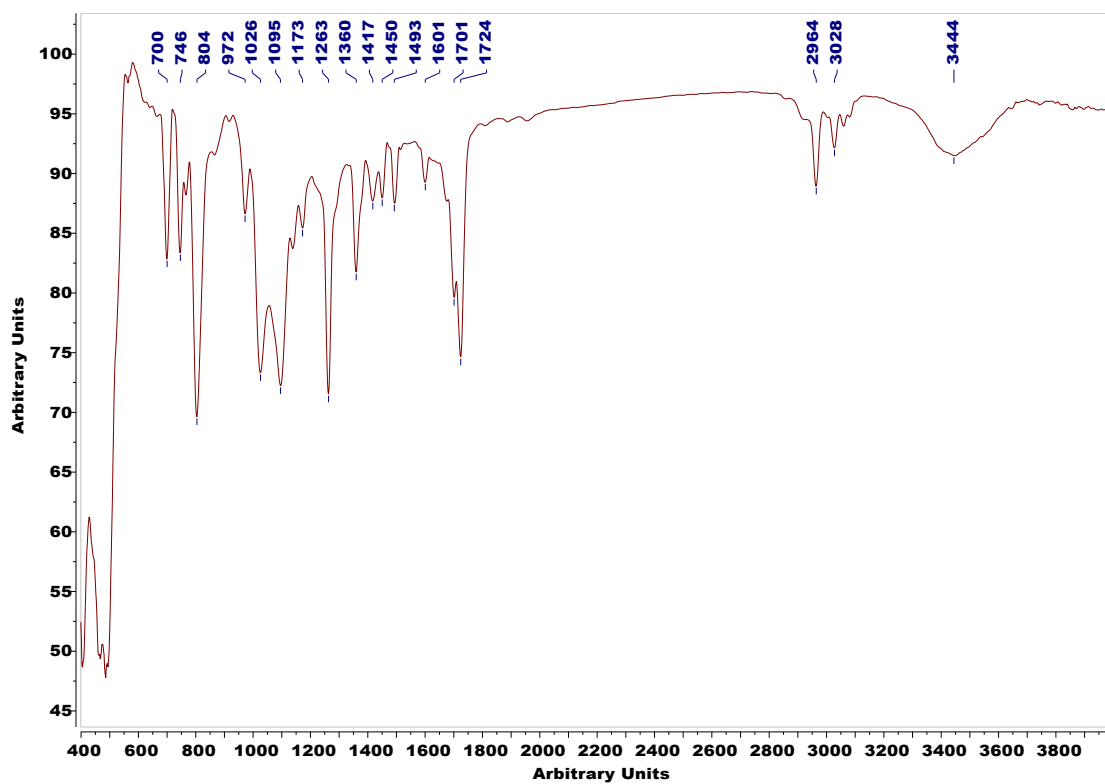
Compound **9** (0.2 mmol) and DDQ (1.2 eq) and 80 mg 4 Å MS in CH₂Cl₂ (10 mL) at rt for 12 hour. And the crude product was purified by silica gel chromatography using petroleum ether/EtOAc = 200/1 as an eluent to give pure product **10** (36 mg, 46% yield) as a colorless liquid; R_f: 0.3 (petroleum ether/EtOAc = 25/1). **IR (KBr)**: 3028, 2924, 1732, 1493, 1441, 1381, 1244, 1217, 974, 933, 823, 746, 698 cm⁻¹. **¹H NMR (300 MHz, CDCl₃)** δ 7.49 (dd, *J* = 12.8, 7.9 Hz, 3H), 7.43-7.35 (m, 4H), 7.33-7.21 (m, 7H), 7.11 (t, *J* = 4.3 Hz, 1H), 7.02 (d, *J* = 15.7 Hz, 1H), 6.58 (d, *J* = 15.7 Hz, 1H), 6.07 (s, 1H), 3.76 (s, 4H), 2.68 (ddd, *J* = 15.0, 11.0, 4.0 Hz, 1H), 2.44-2.34 (m, 1H), 2.26 (dt, *J* = 14.0, 4.5 Hz, 1H), 1.79-1.63 (m, 1H). **¹³C {¹H} NMR (101 MHz, CDCl₃)** δ: 172.9, 140.5, 139.5, 136.6, 133.4, 131.3, 129.5, 129.2, 128.5, 128.5, 128.2, 128.1, 127.6, 127.1, 126.8, 126.5, 124.6, 87.5, 76.1, 56.9, 52.1, 27.6, 26.3. **HRMS (ESI) m/z (M+H)⁺** calcd for C₂₇H₂₅O₃: 397.1804; Found: 397.1805.

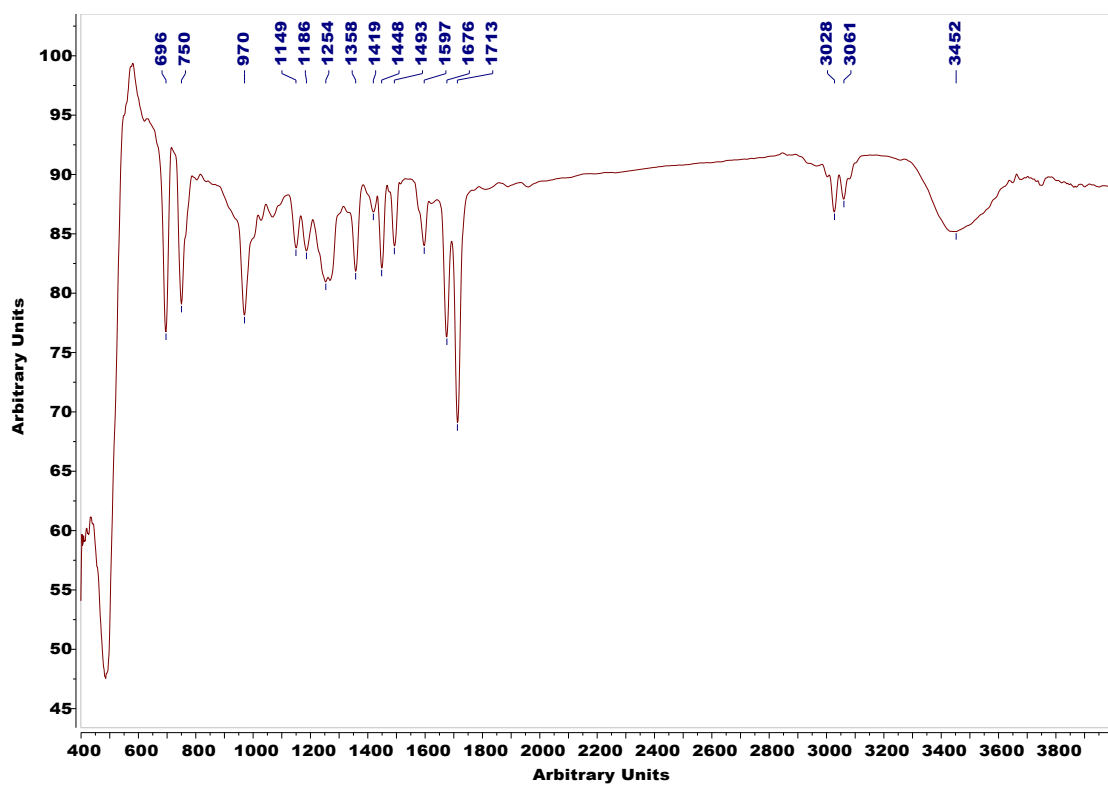
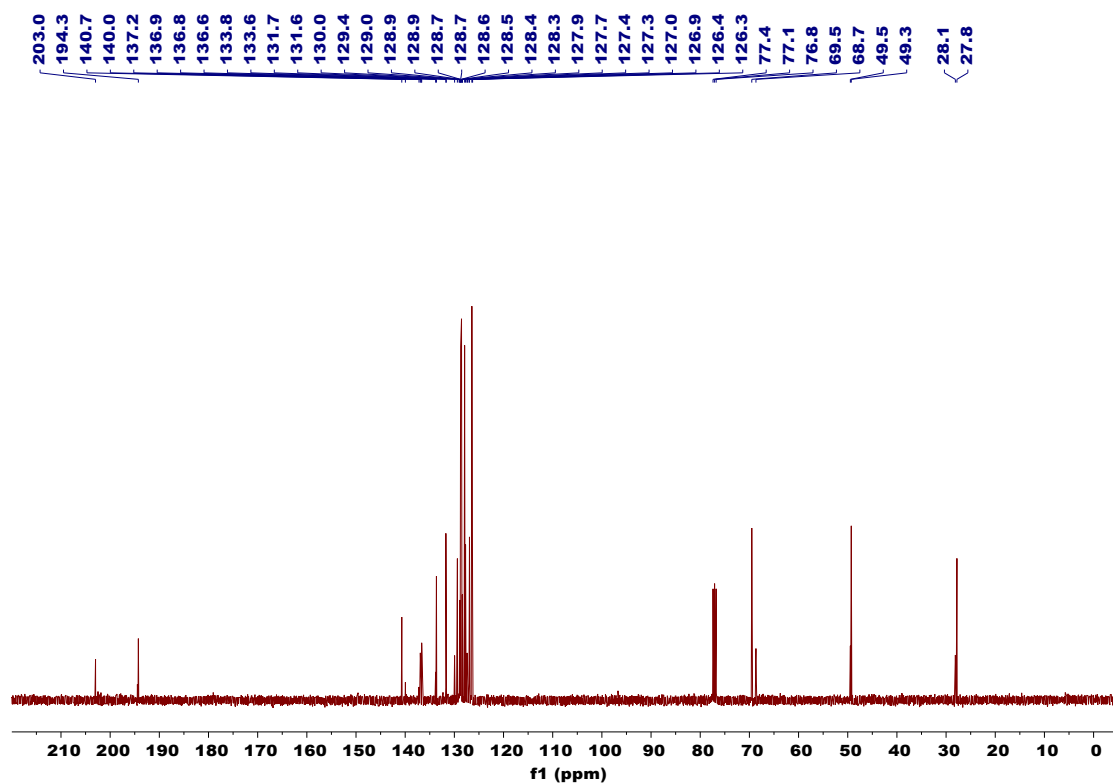
3. Reference

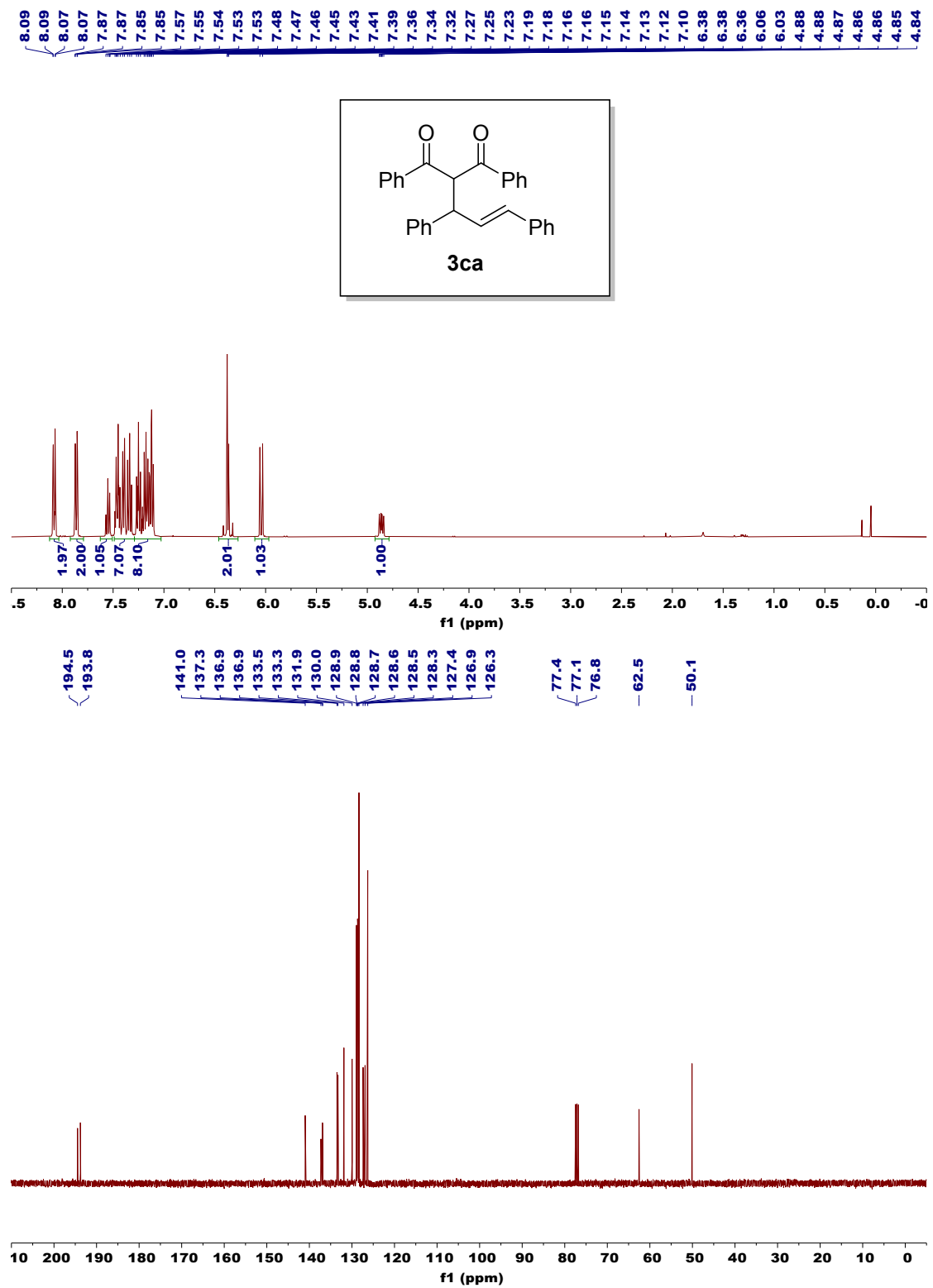
1. L.-D. Zhang, X.-J. Song, N.-Y. Wang, L.-F. Zhao, F. Qiang, X.-Y. You, C.-T. Peng, T.-T. Gao, M.-H. Xiong, C. Gao, Y. Luo, Y. Xu, Q.-Y. Zhang and L.-T. Yu, *RSC Adv.*, 2015, **5**, 25967-25978.
2. X.-Y. Zhan, H. Zhang, Y. Dong, J. Yang, S. He, Z.-C. Shi, L. Tang and J.-Y. Wang, *J. Org. Chem.*, 2020, **85**, 6578-6592.
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4. (a) A. Cullen, J. M. Alfred and D. B. G. Williams, *RSC Adv.*, **2017**, *7*, 42168-42171.; (b) D.-P. Chen, K. Yuan, X.-L. Xu and J.-Z. Yan, *Tetrahedron Lett.*, 2015, *13*, 1641-1644.; (c) X.-S. Xiao, S.-P. Huang, S.-S. Tang, G.-K. Jia, G.-C. Ou and Y.-Y. Li, *J. Org. Chem.*, **2019**, *84*, 7618-7629.; (d) R. Nallagond, R. R. Reddy and P. Ghorai, *Chem. Eur. J.*, **2015**, *21*, 14732-14736.; (e) W.-D. Rao, L.-P. Wai, H. L. T. Adeline, P.-J. Goh, J. M. L. Choy, J. Kaijie-Ke, and P. W. H. Chan, *Tetrahedron Lett.*, **2008**, *49*, 122-126.; (f) M. Prat, J. Ribas and M. Moreno-Mañas, *Tetrahedron Lett.*, **1992**, *48*, 1695-1706.; (g) W. Huang, Z.-X. Zhang, X. Xiang, R.-T. Liu, X.-P. Zhou and J.-L. Wang, *J. Org. Chem.*, **2009**, *74*, 3299-3304.; (h) Y.-G. Zhu, B.-G. Zhao and Y. Shi, *Org. Lett.*, **2013**, *15*, 992-995.

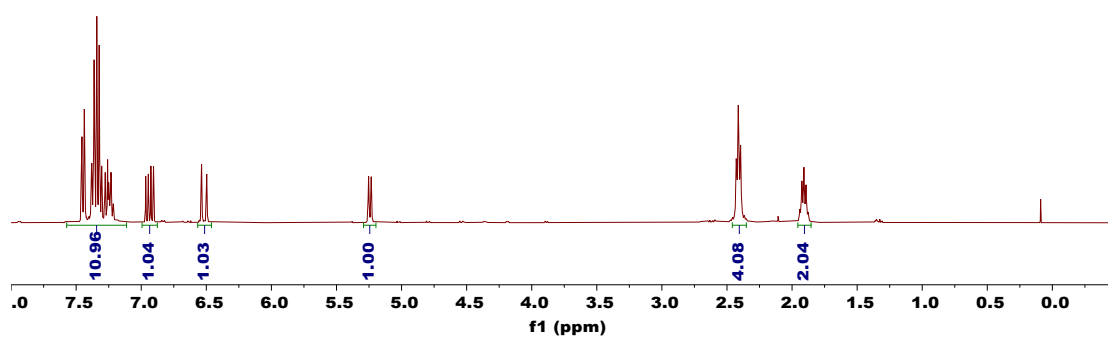
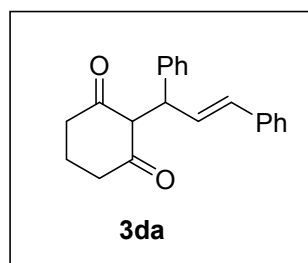
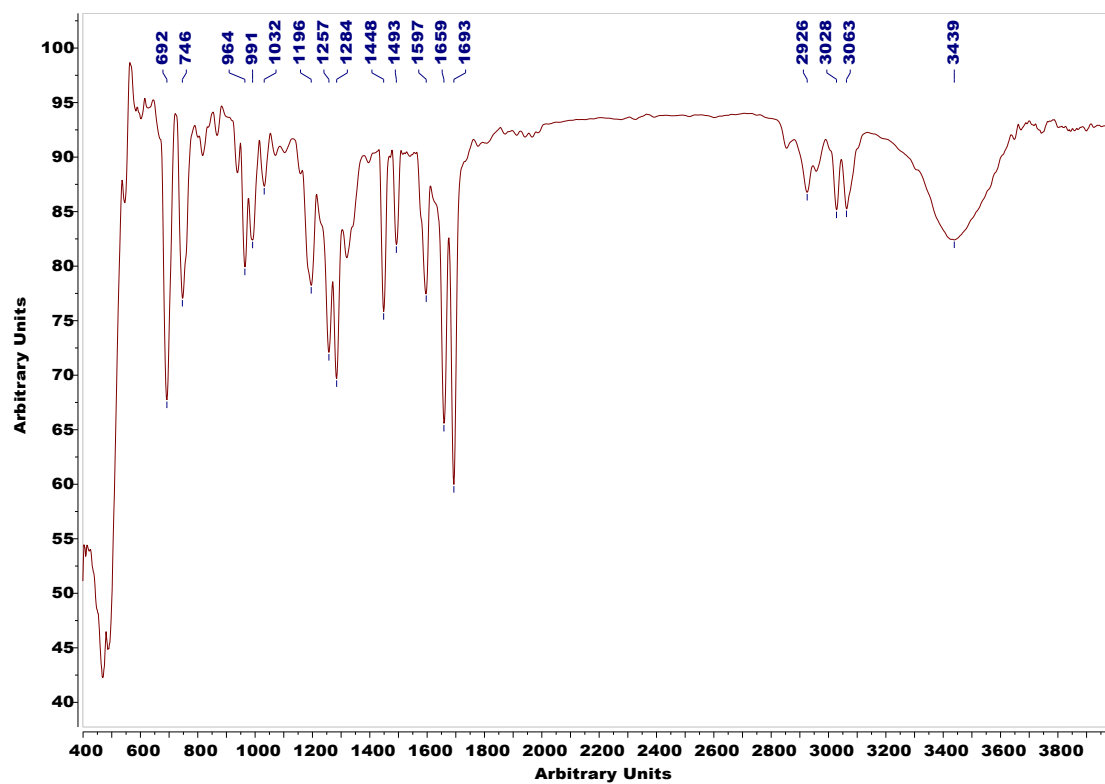
4 ^1H NMR, ^{13}C NMR and IR

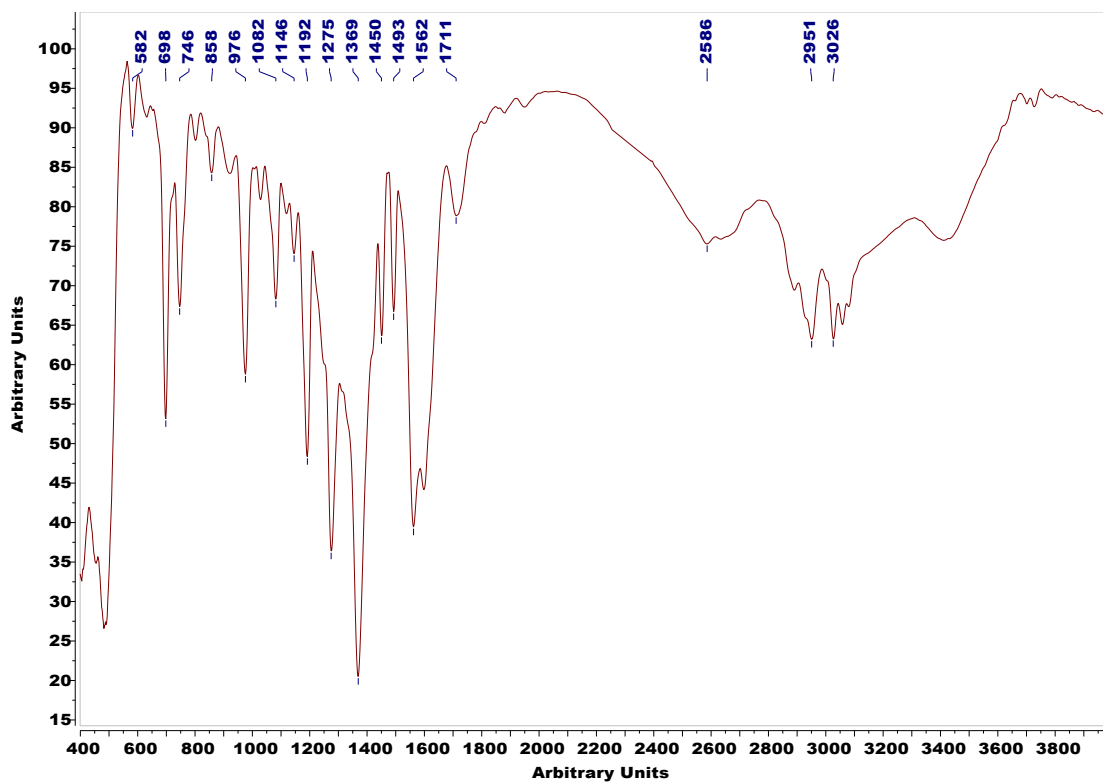
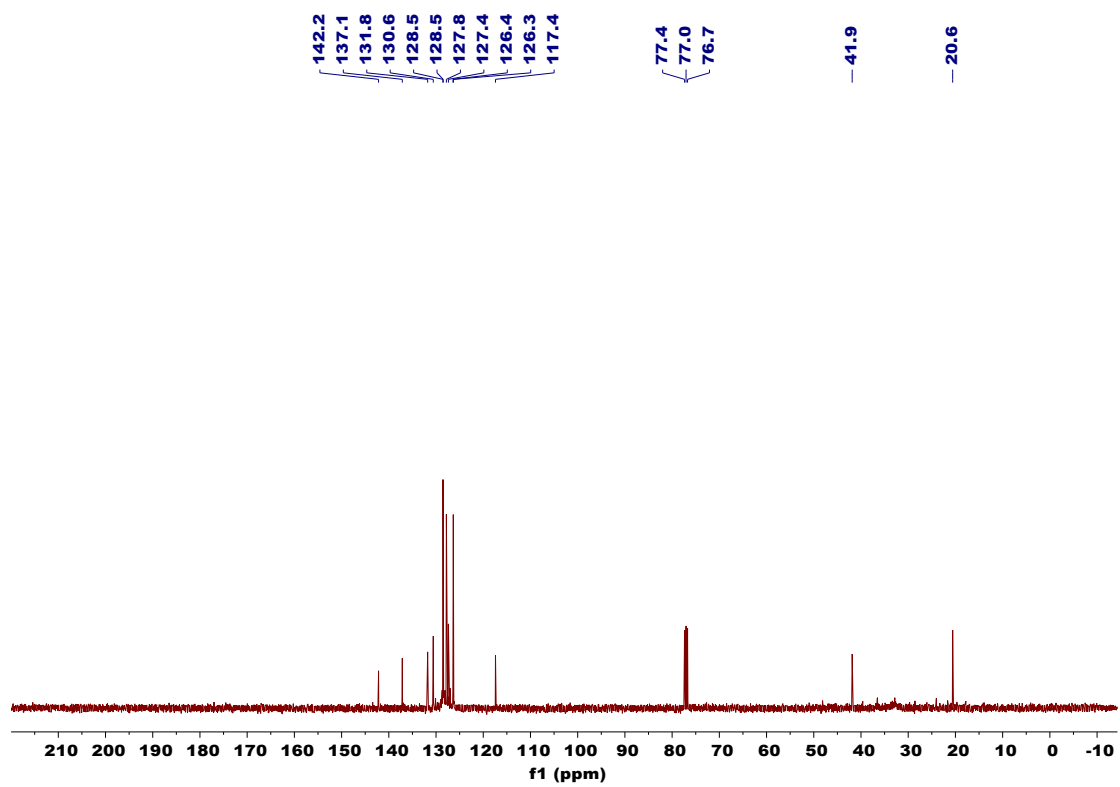


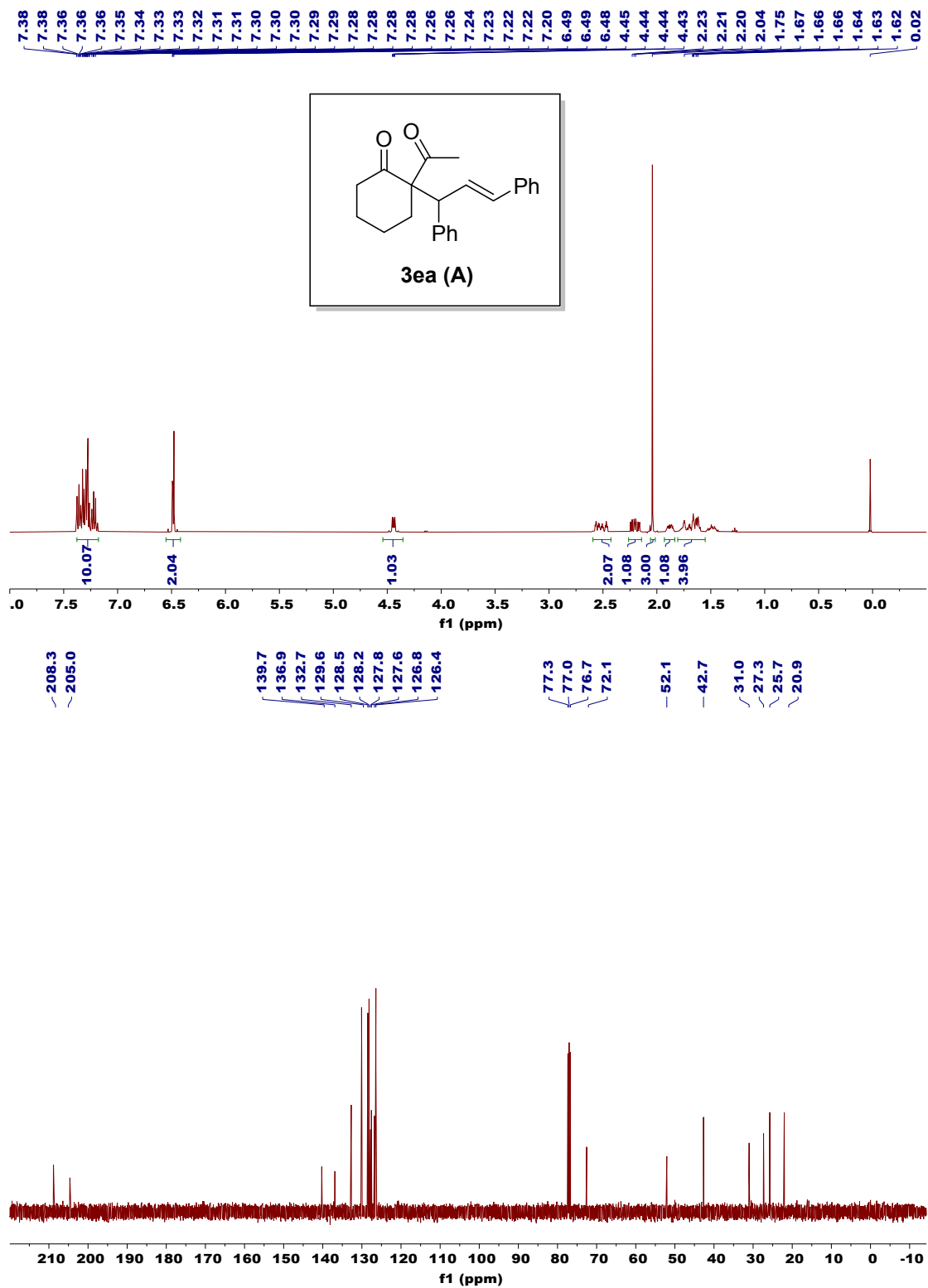


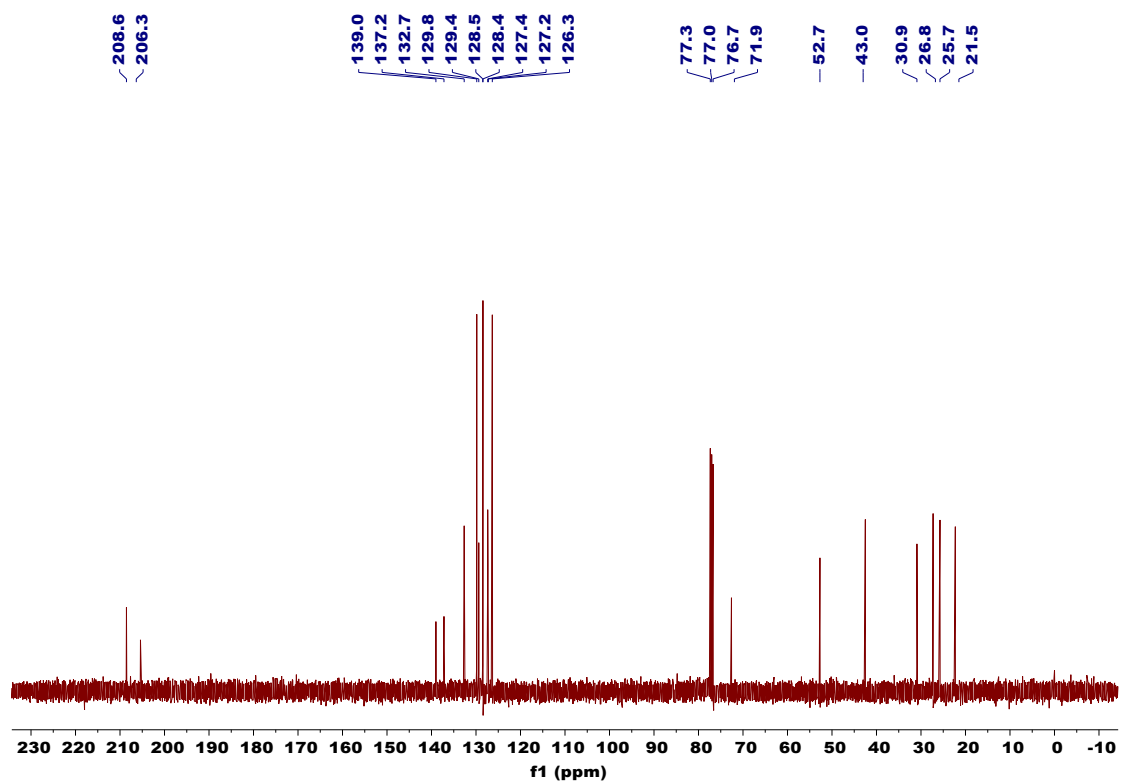
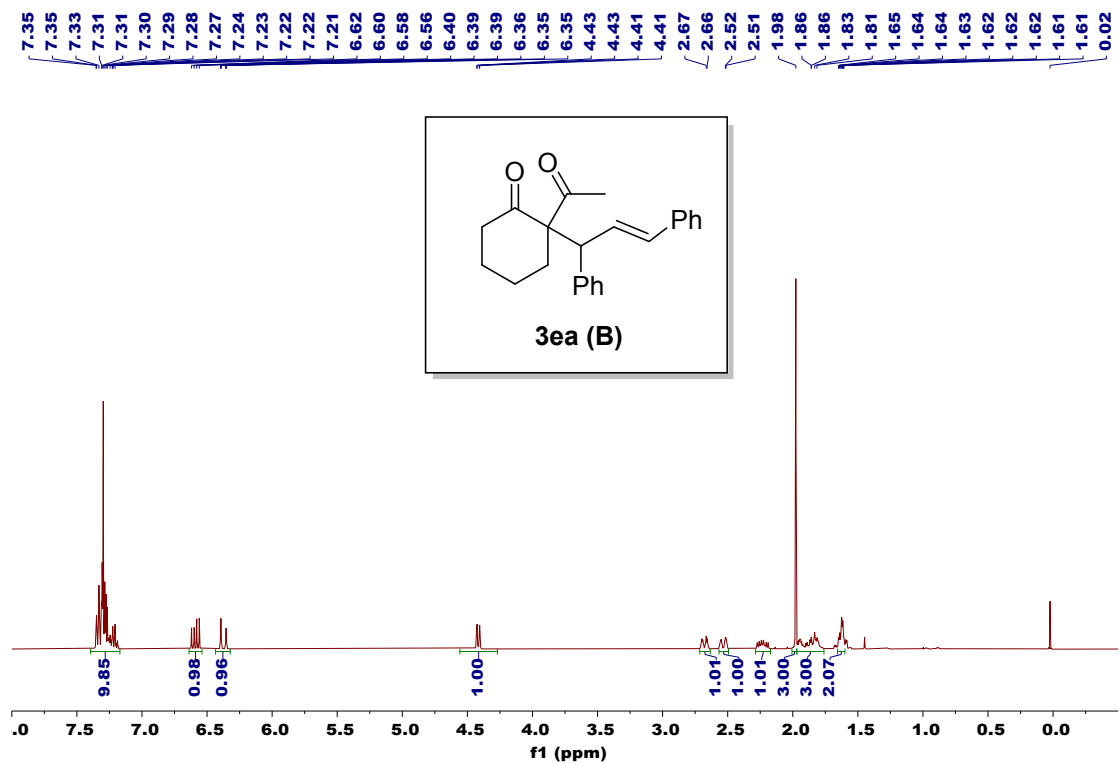


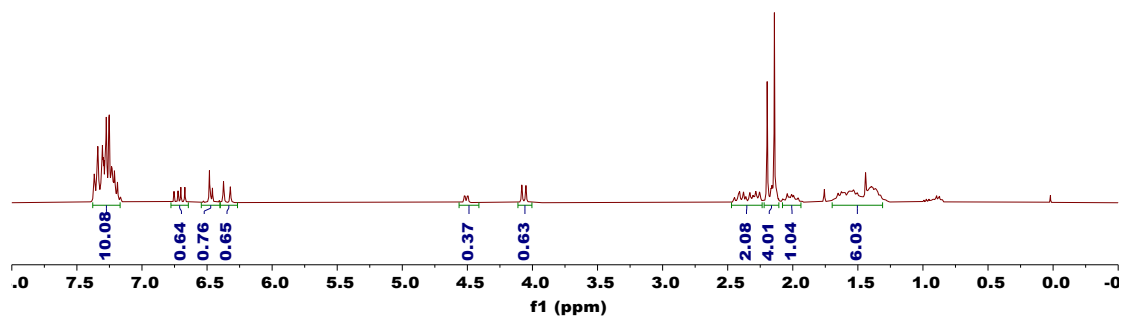
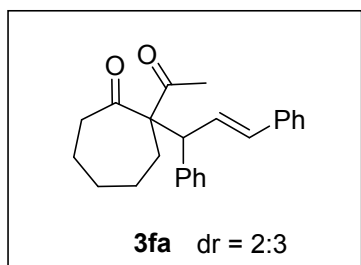
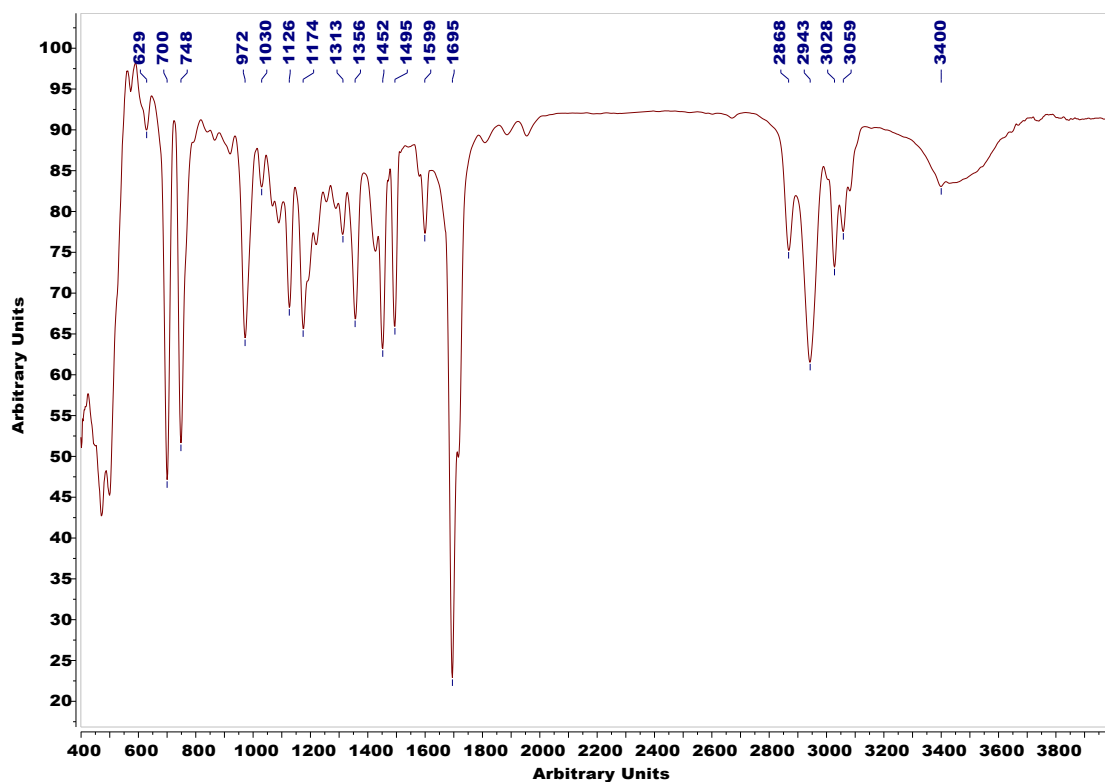


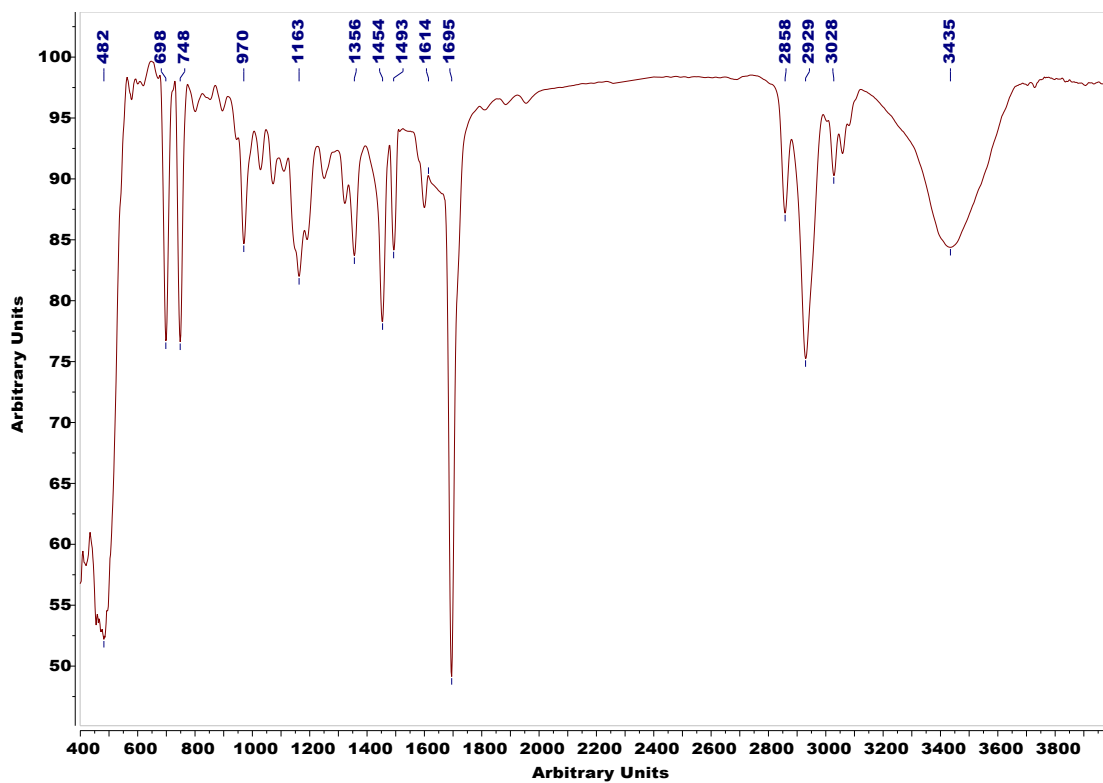
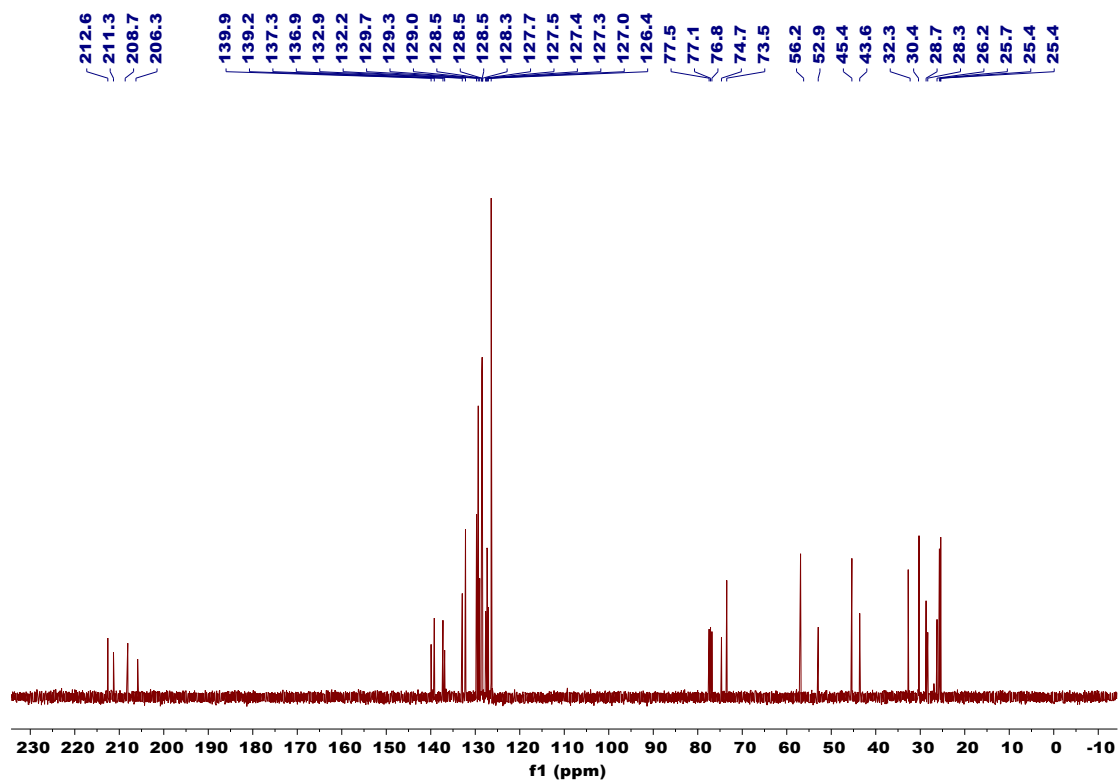


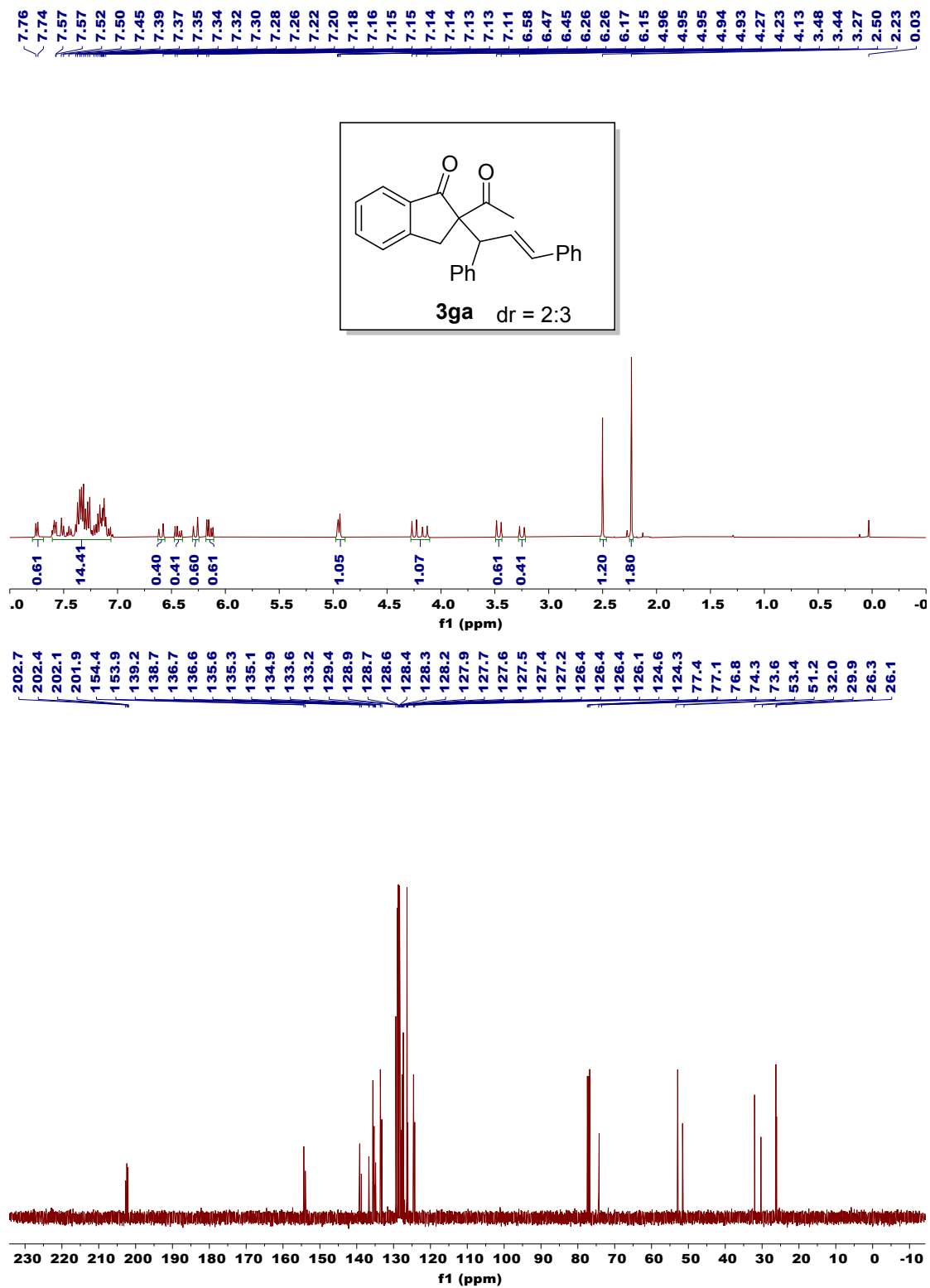


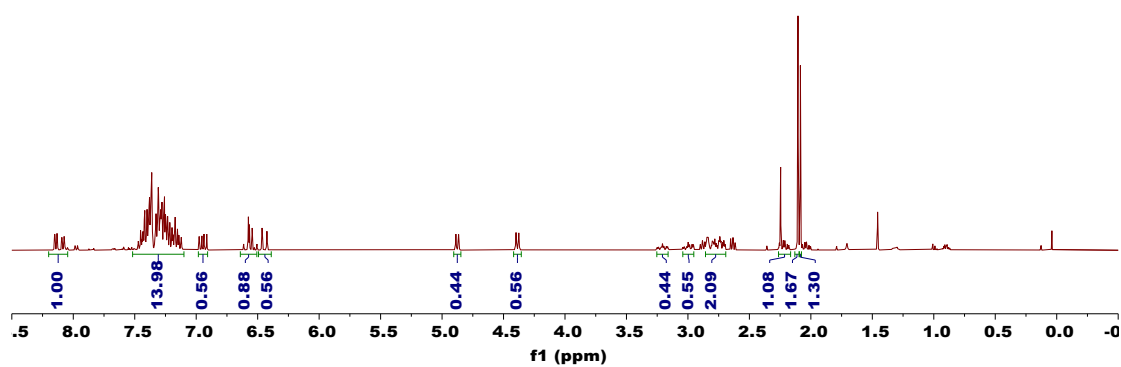
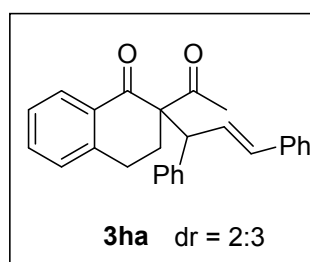
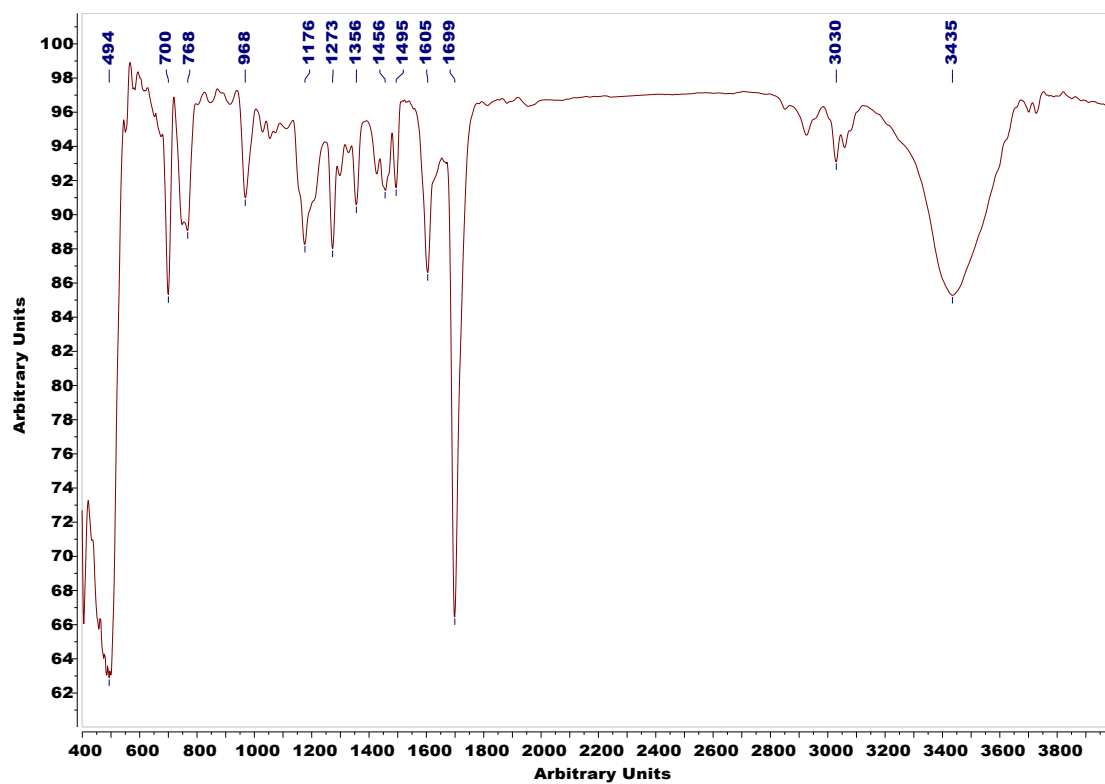


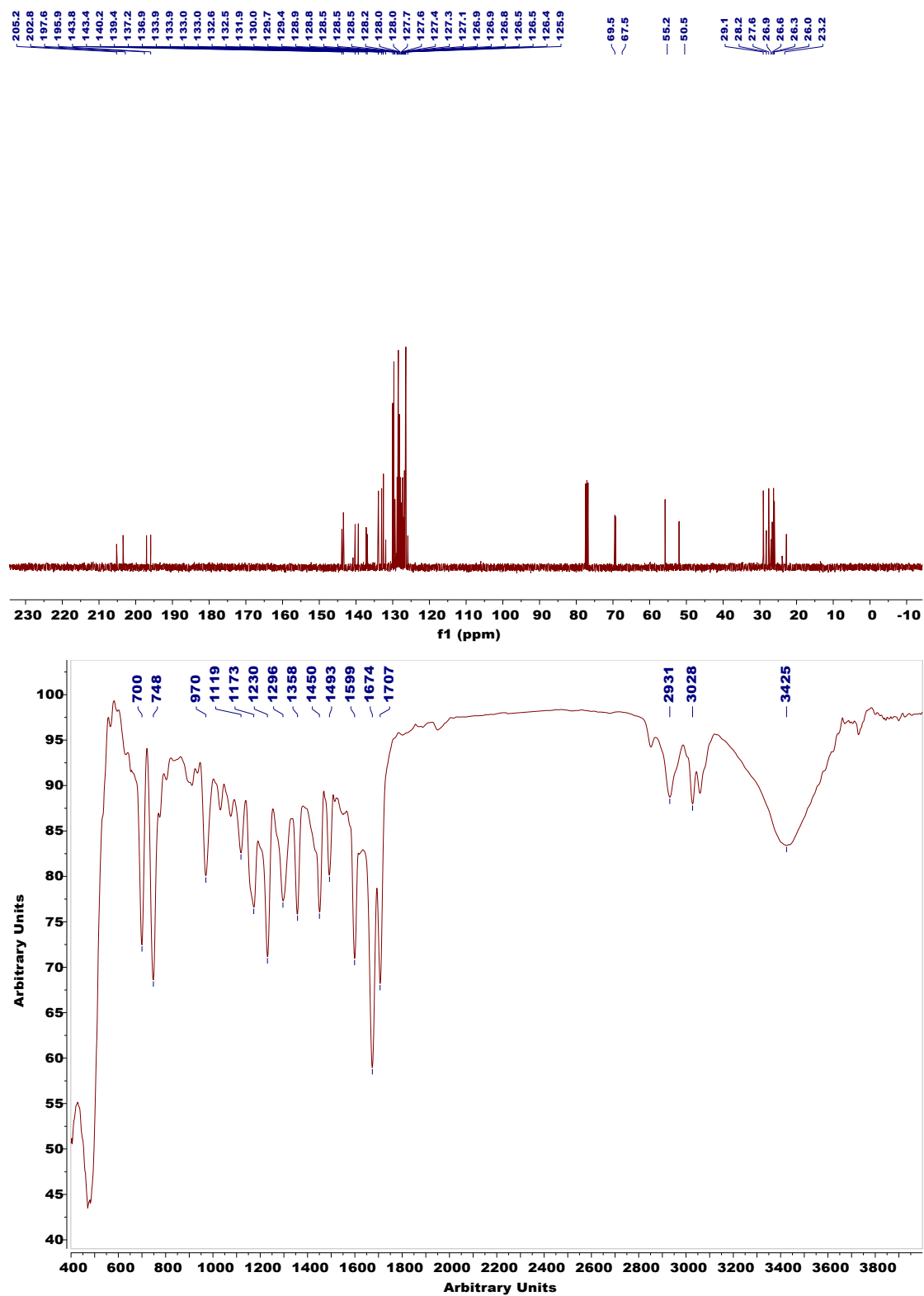


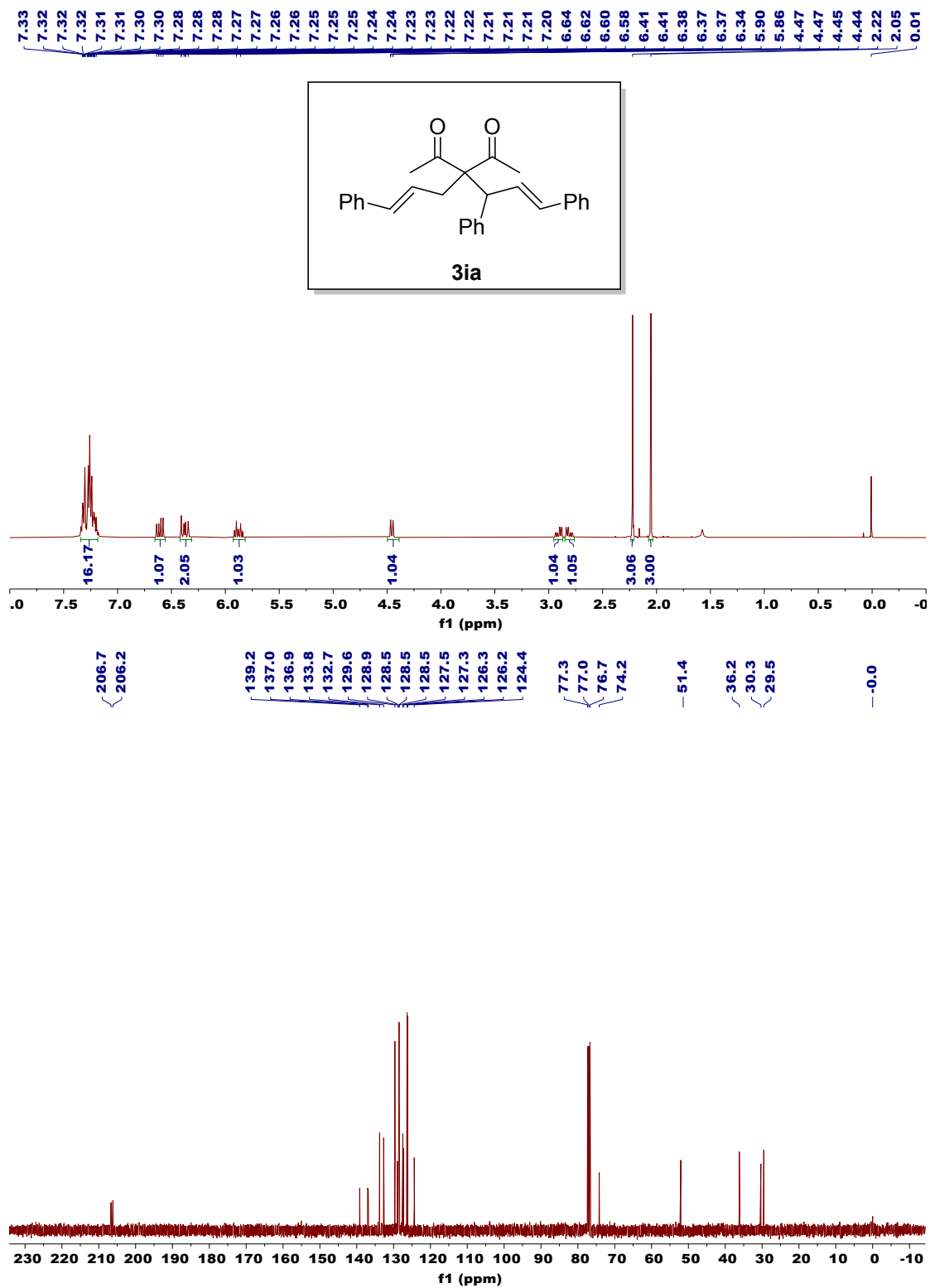


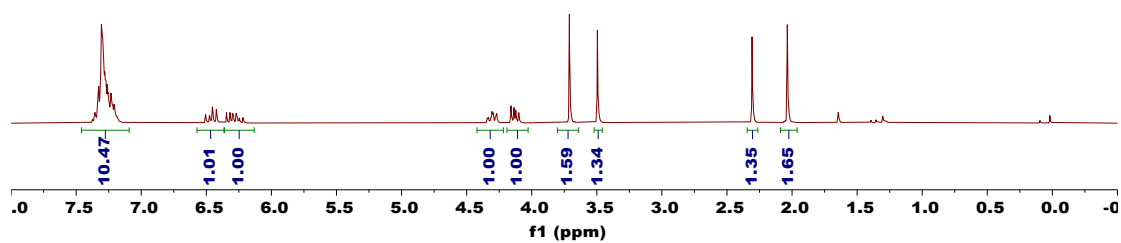
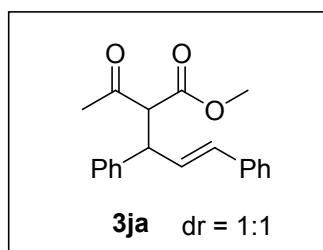
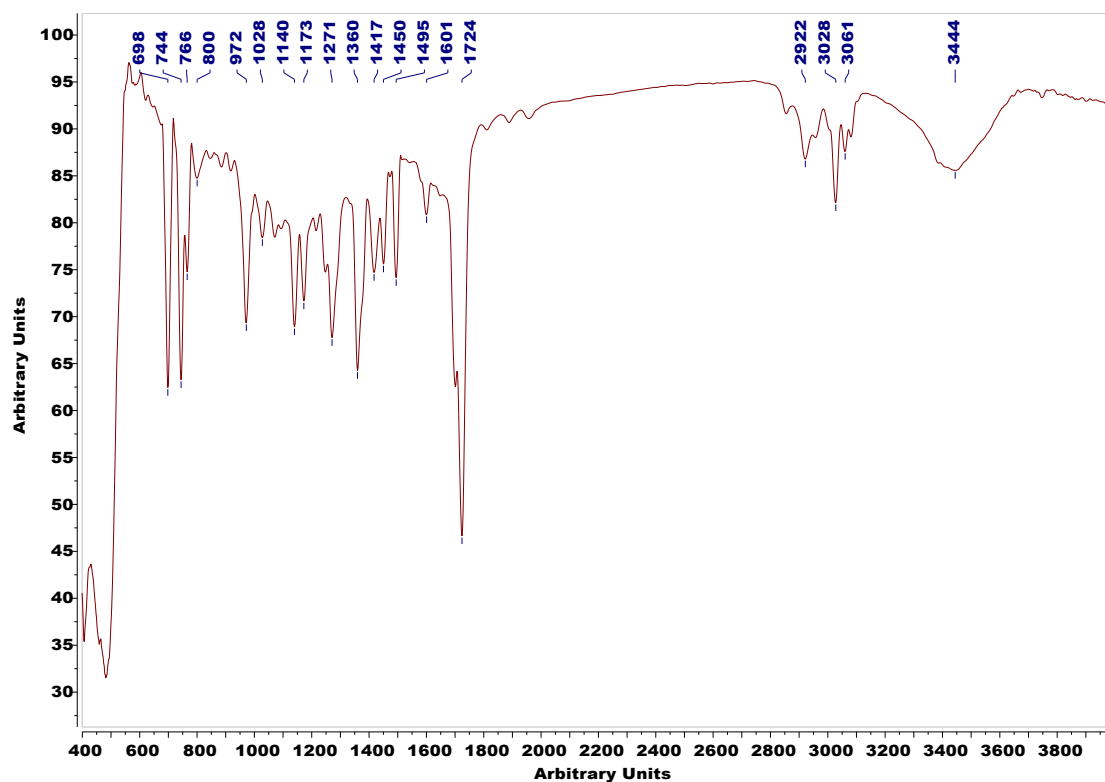


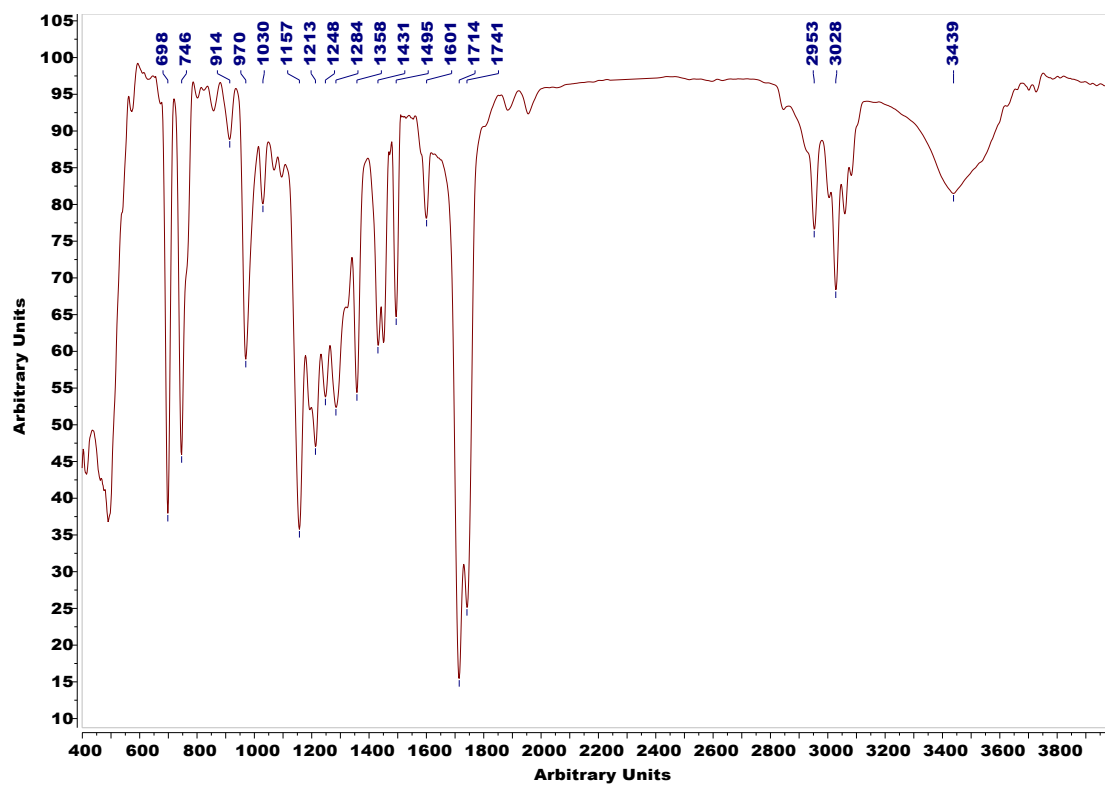
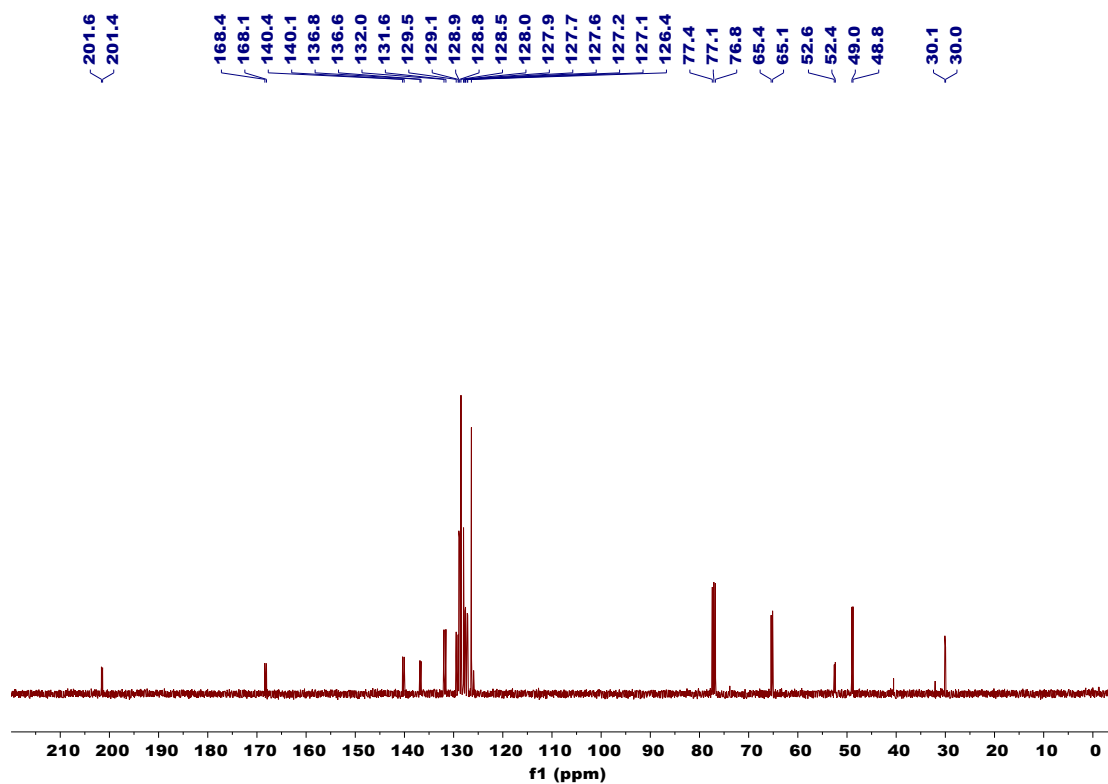


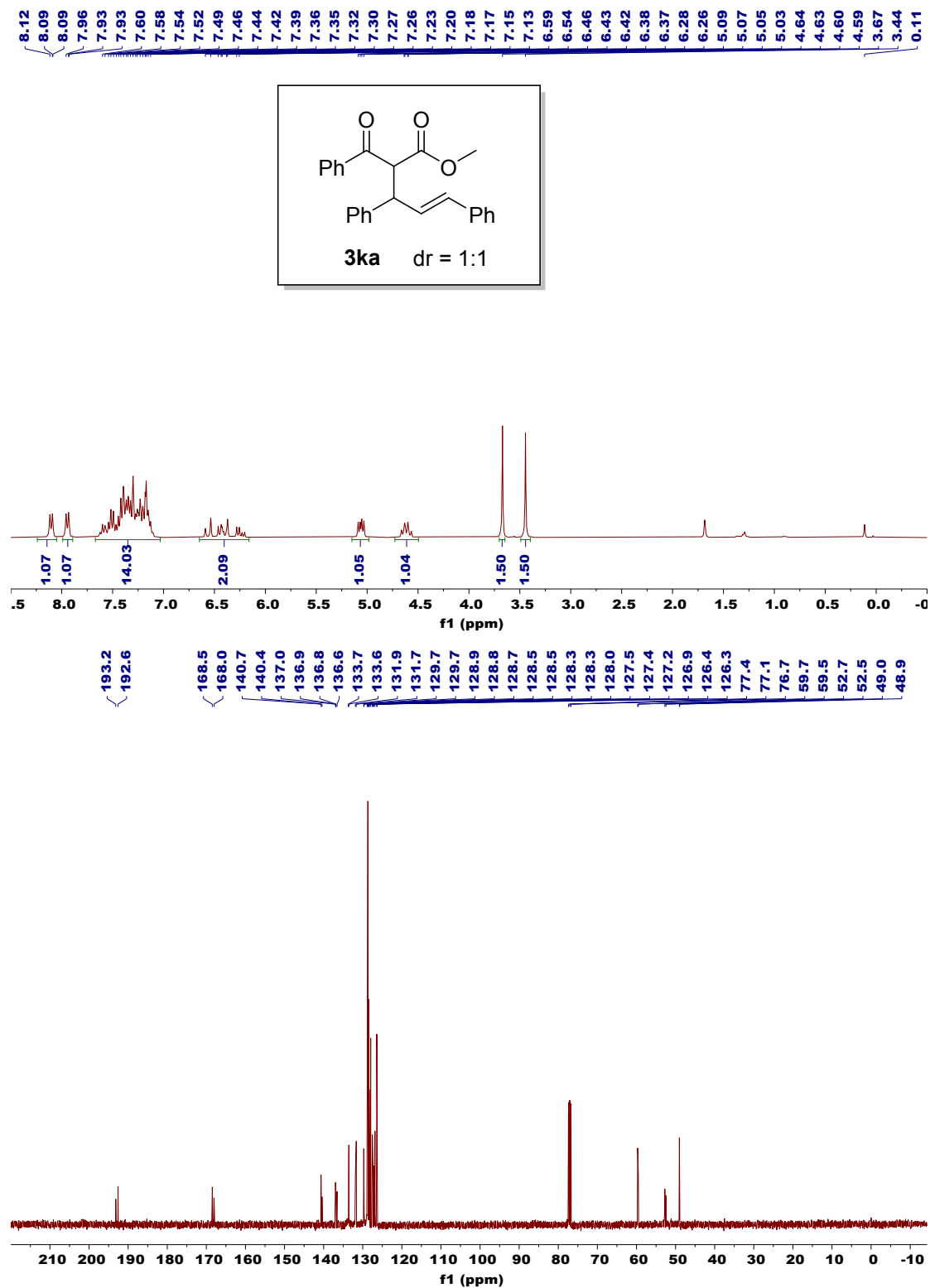


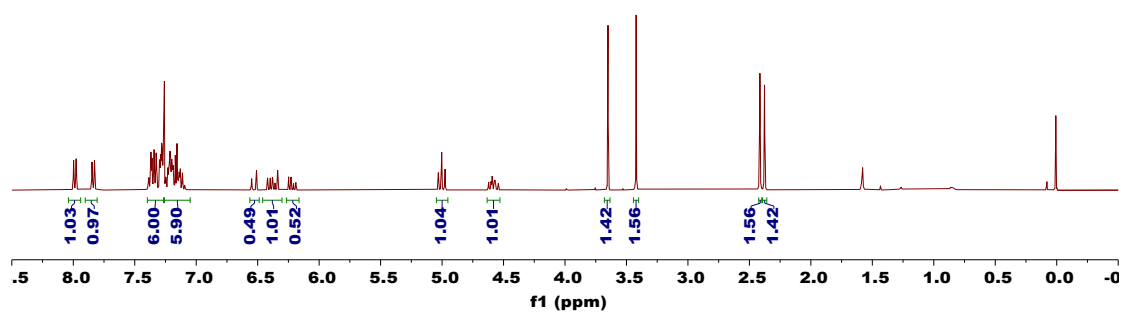
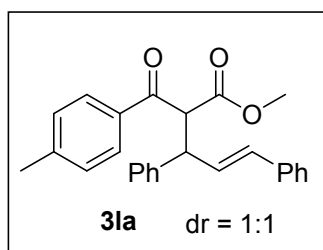
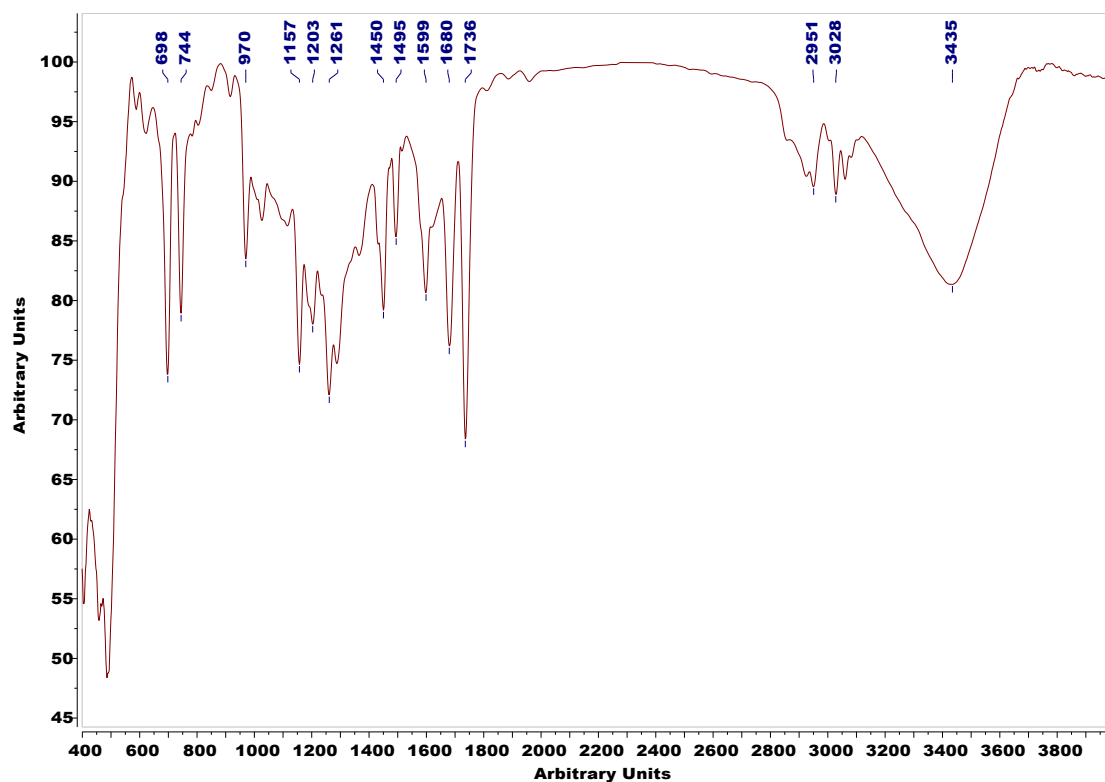


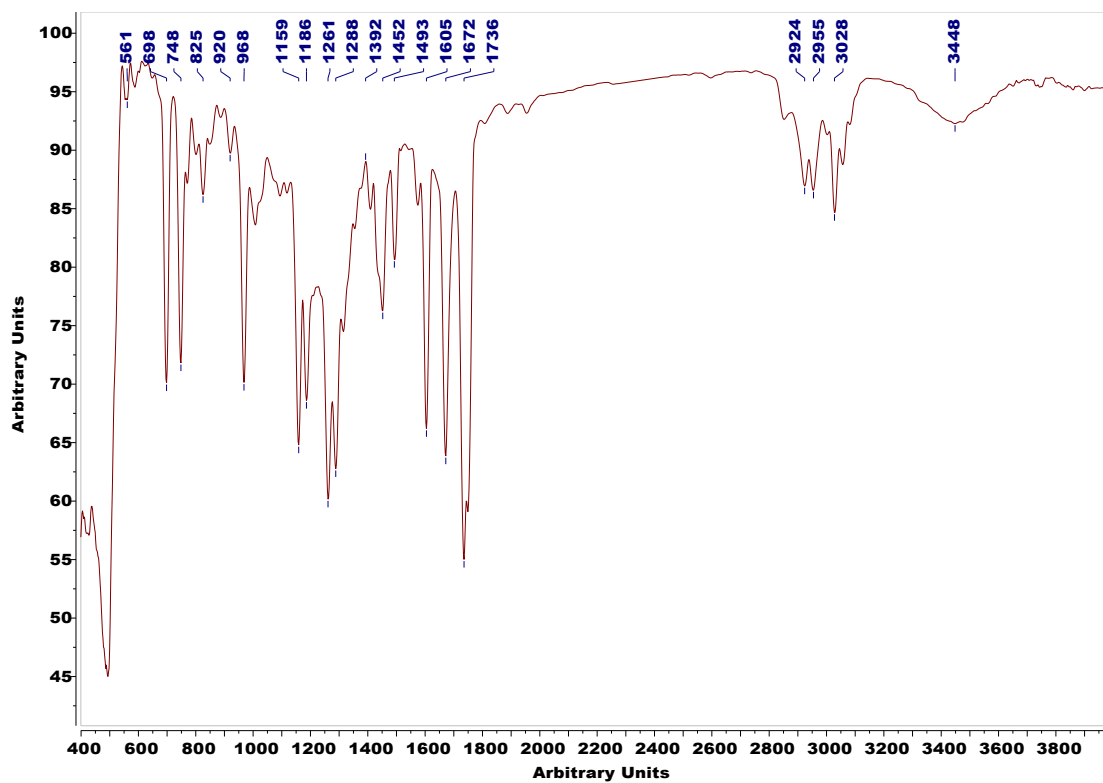
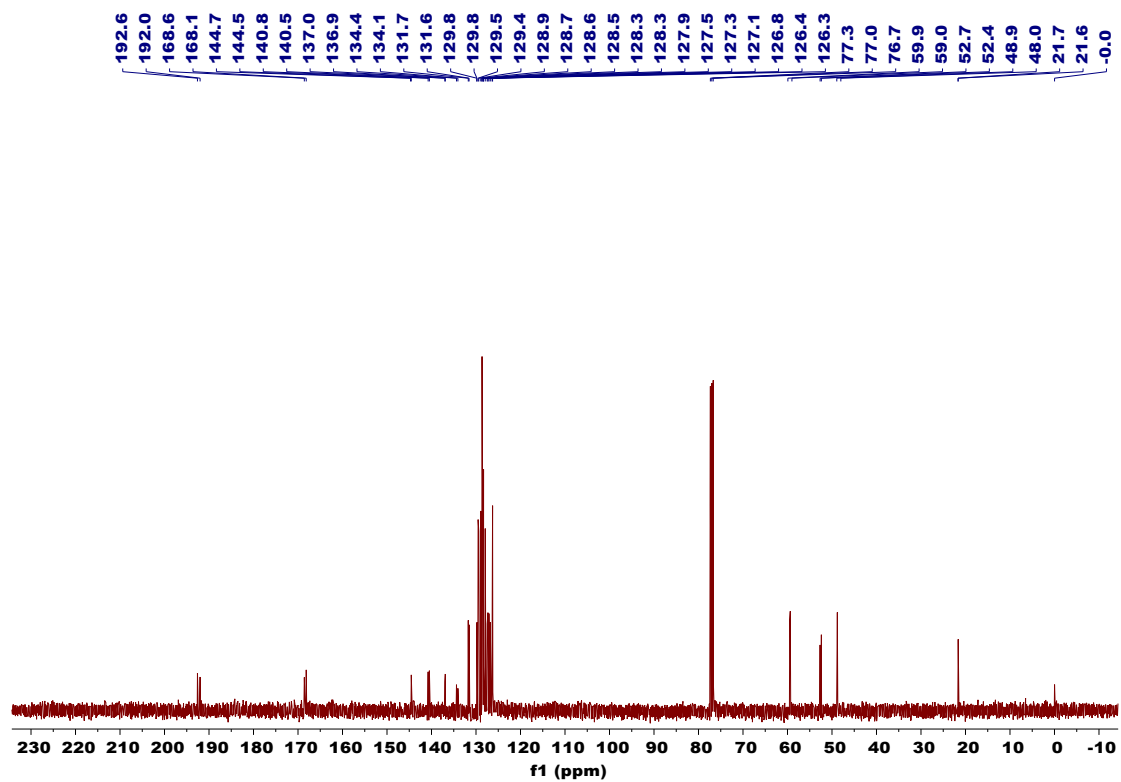


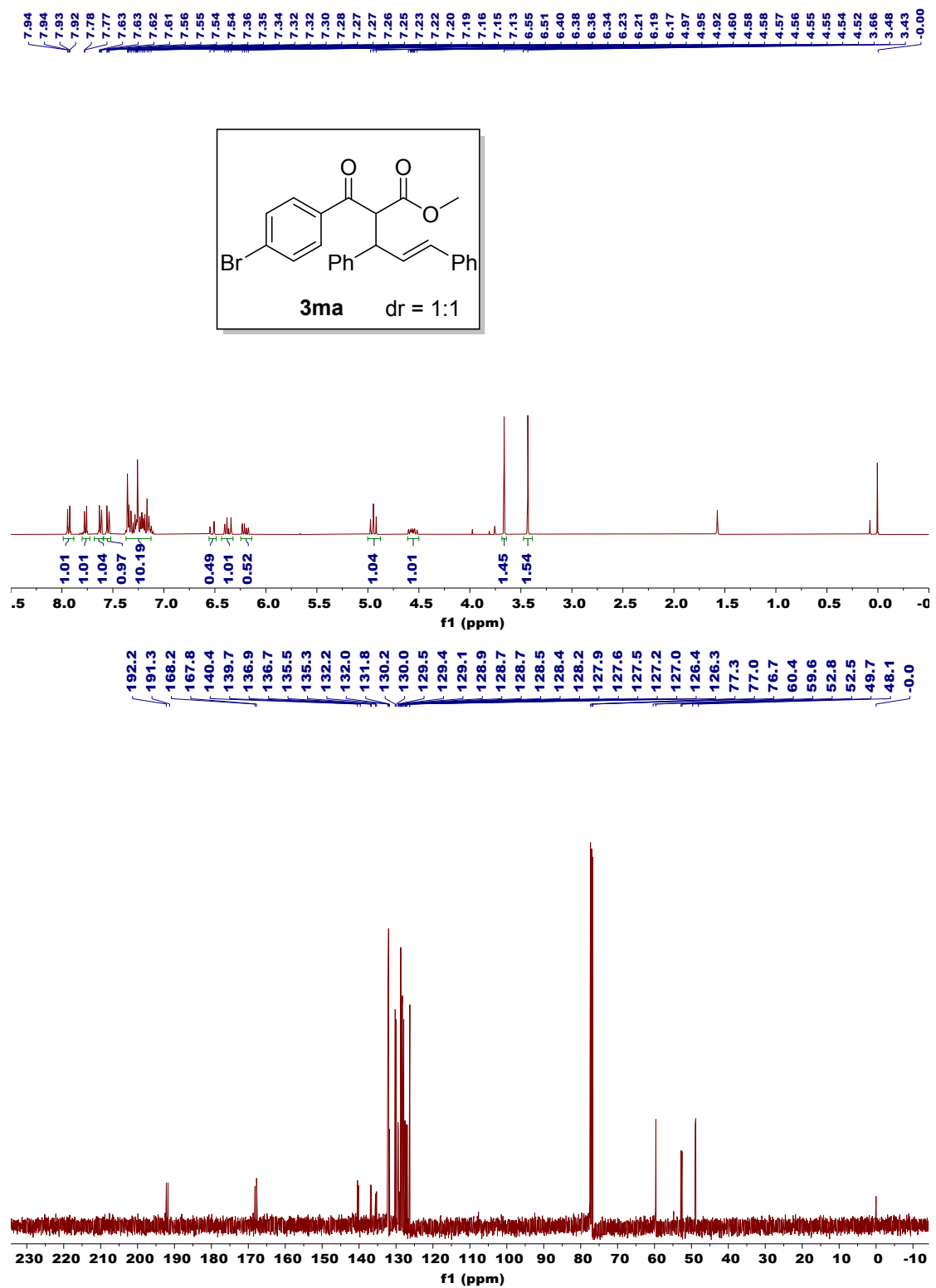


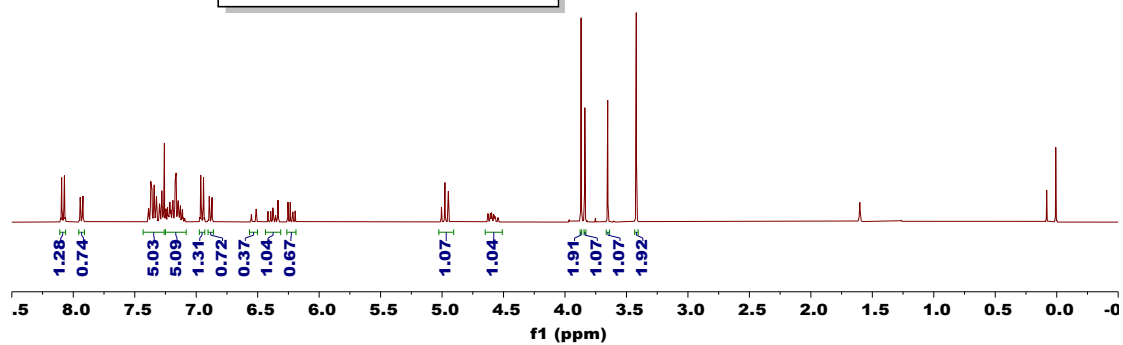
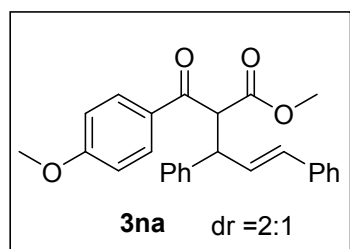
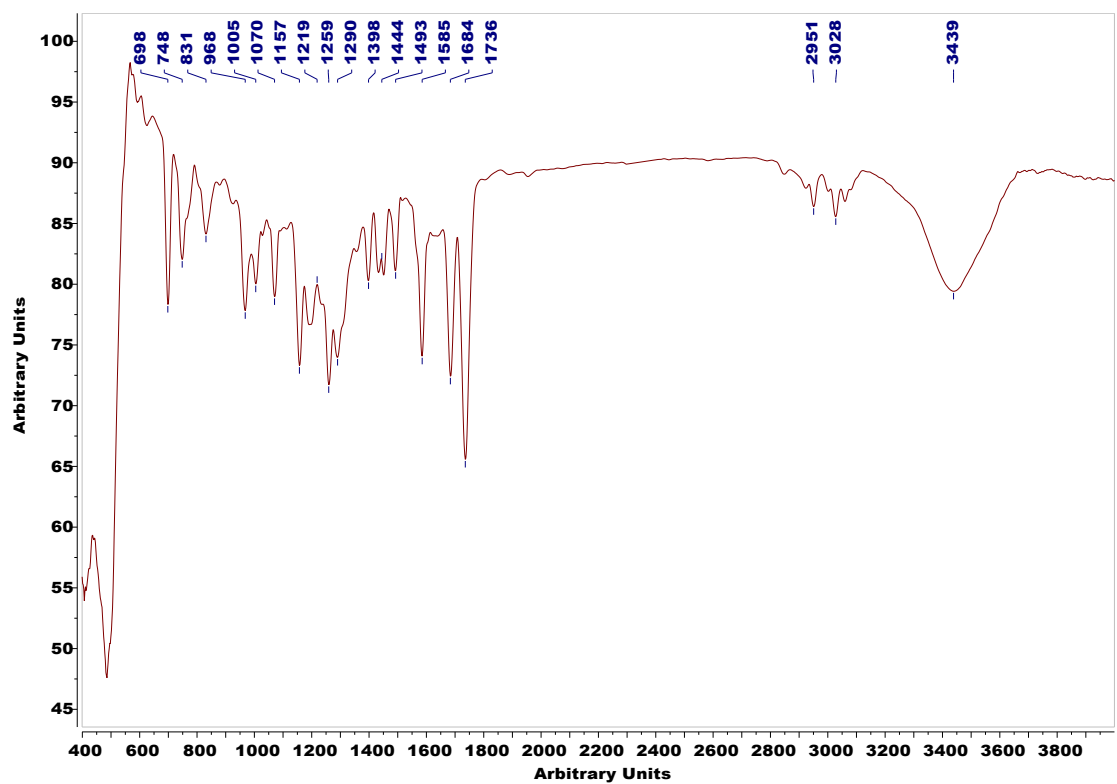


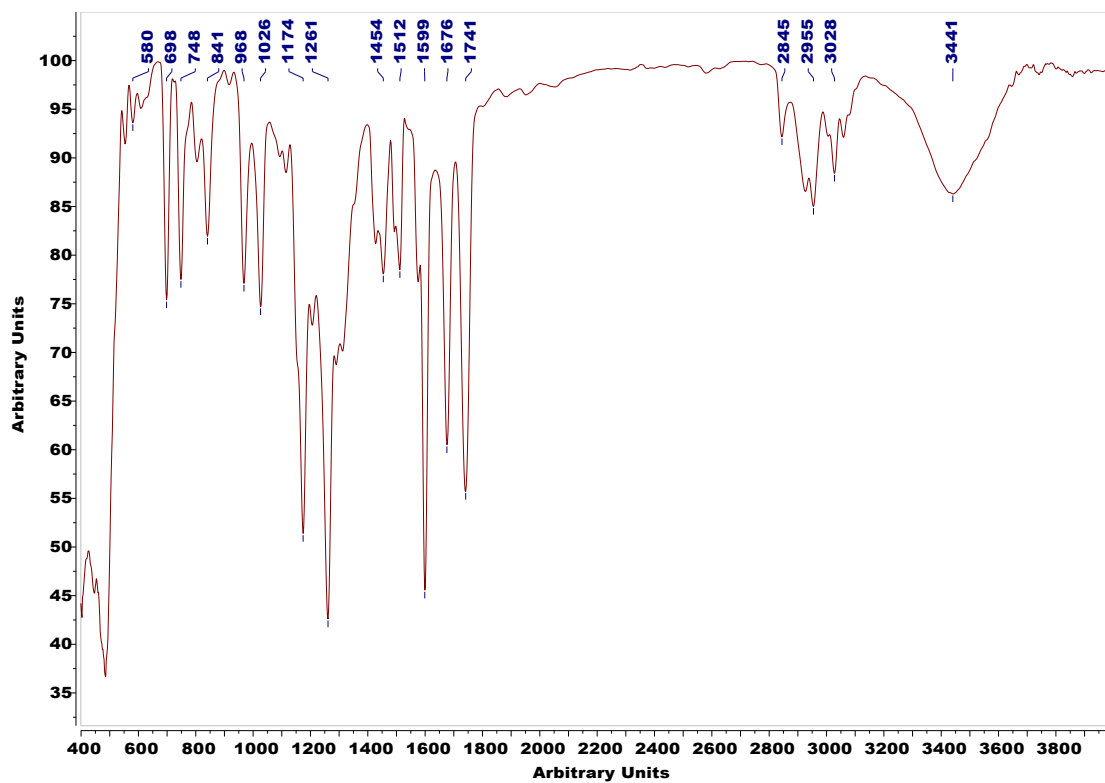
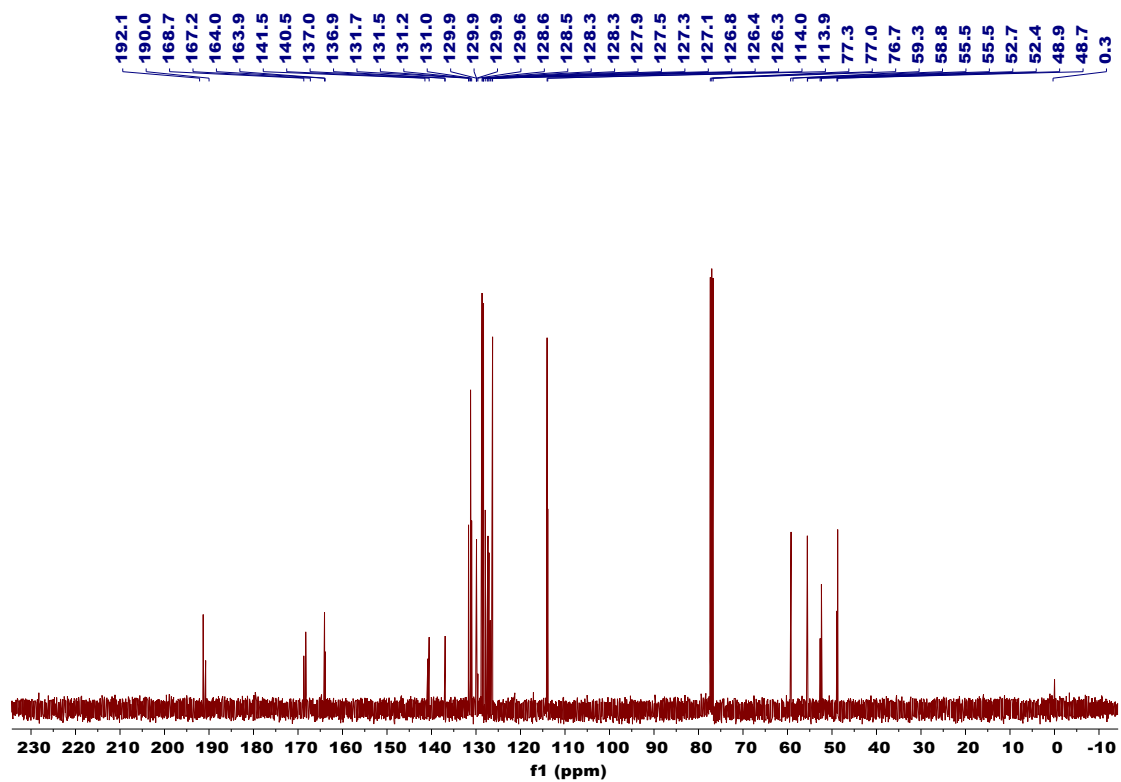


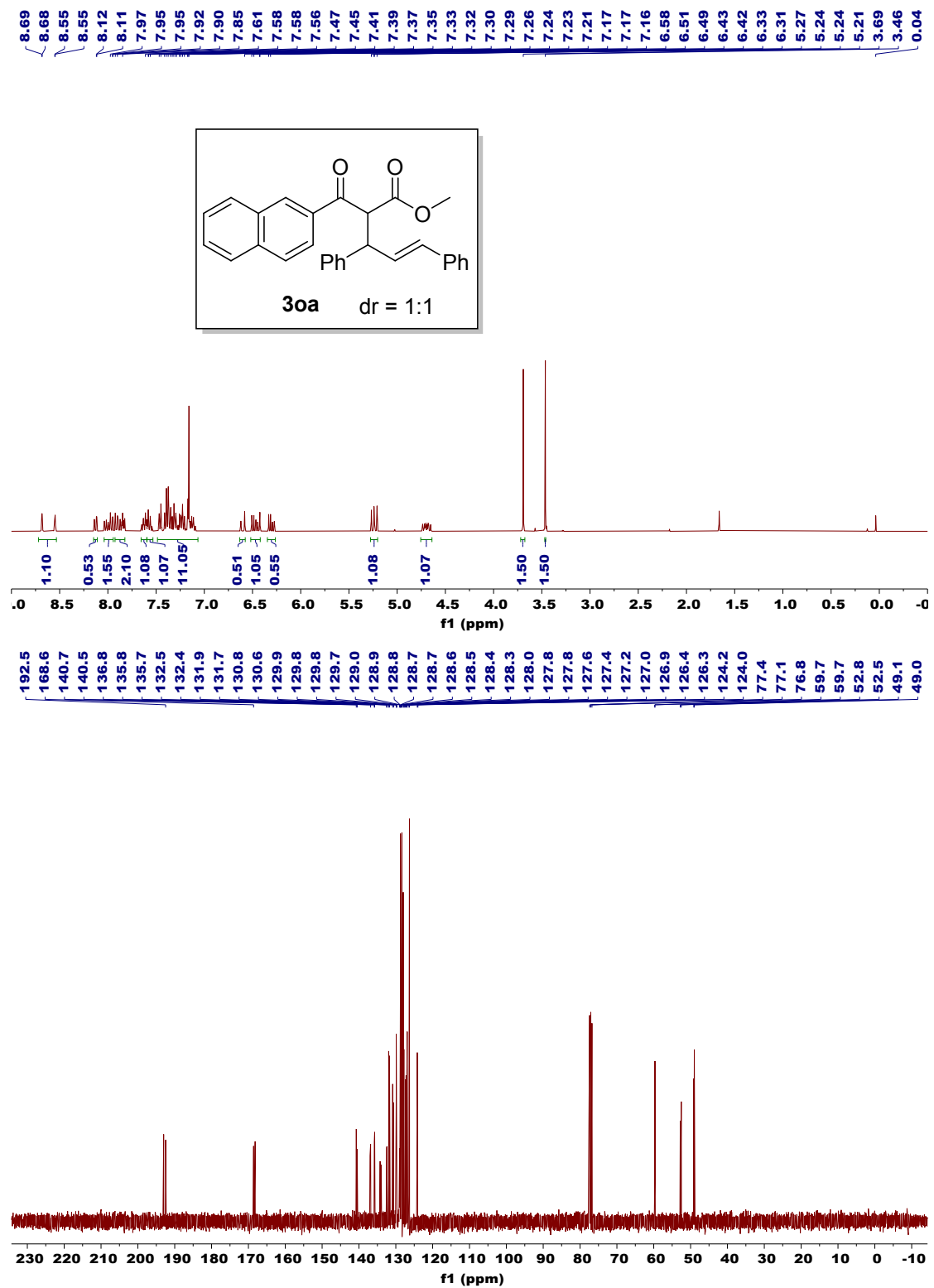


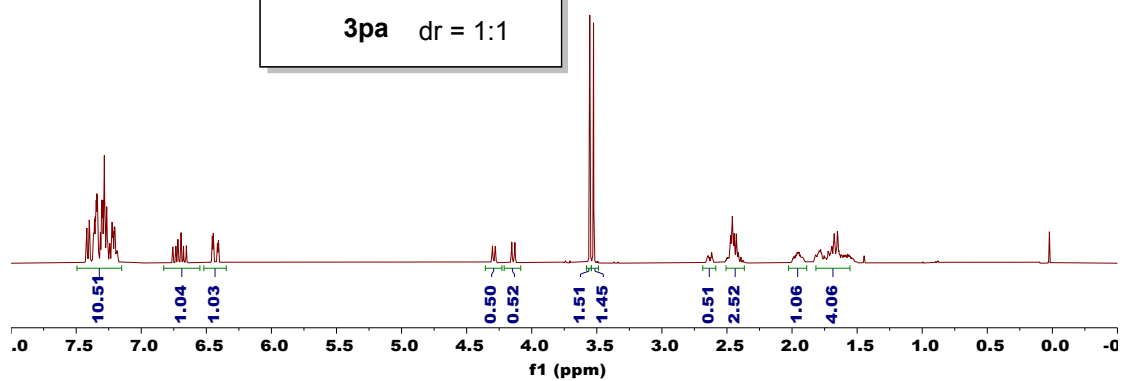
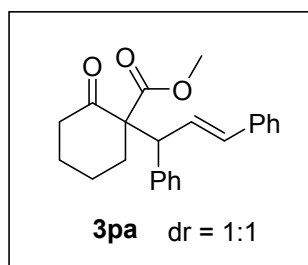
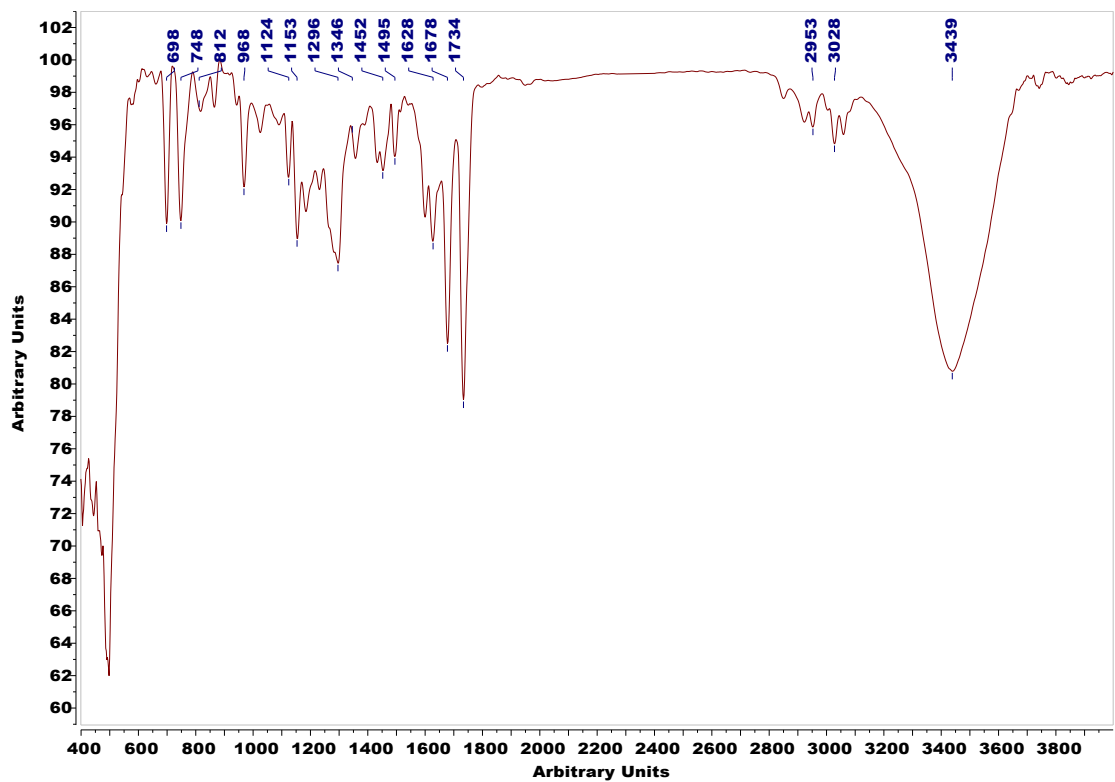


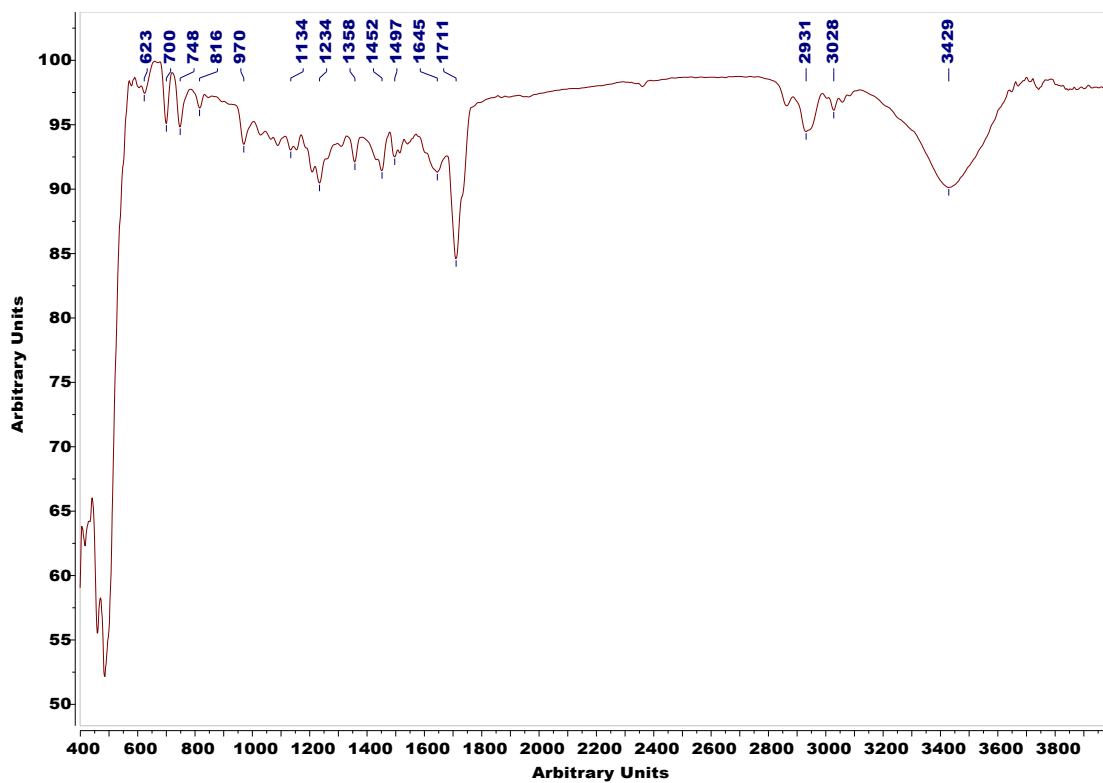
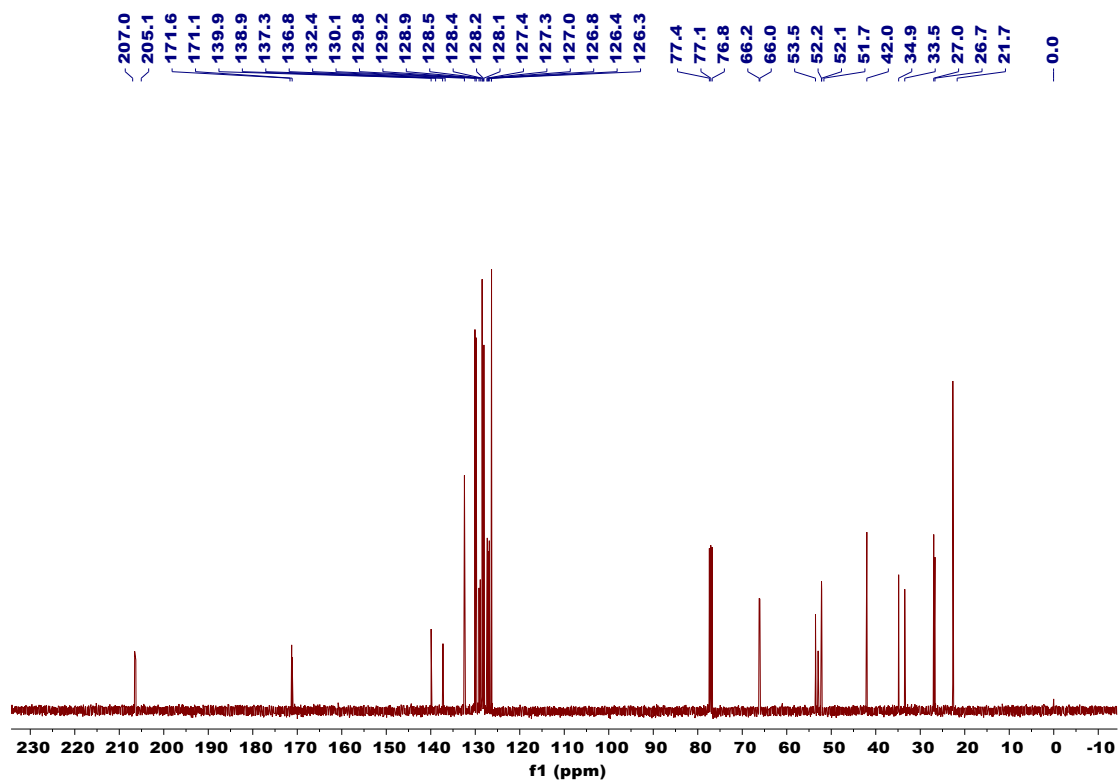


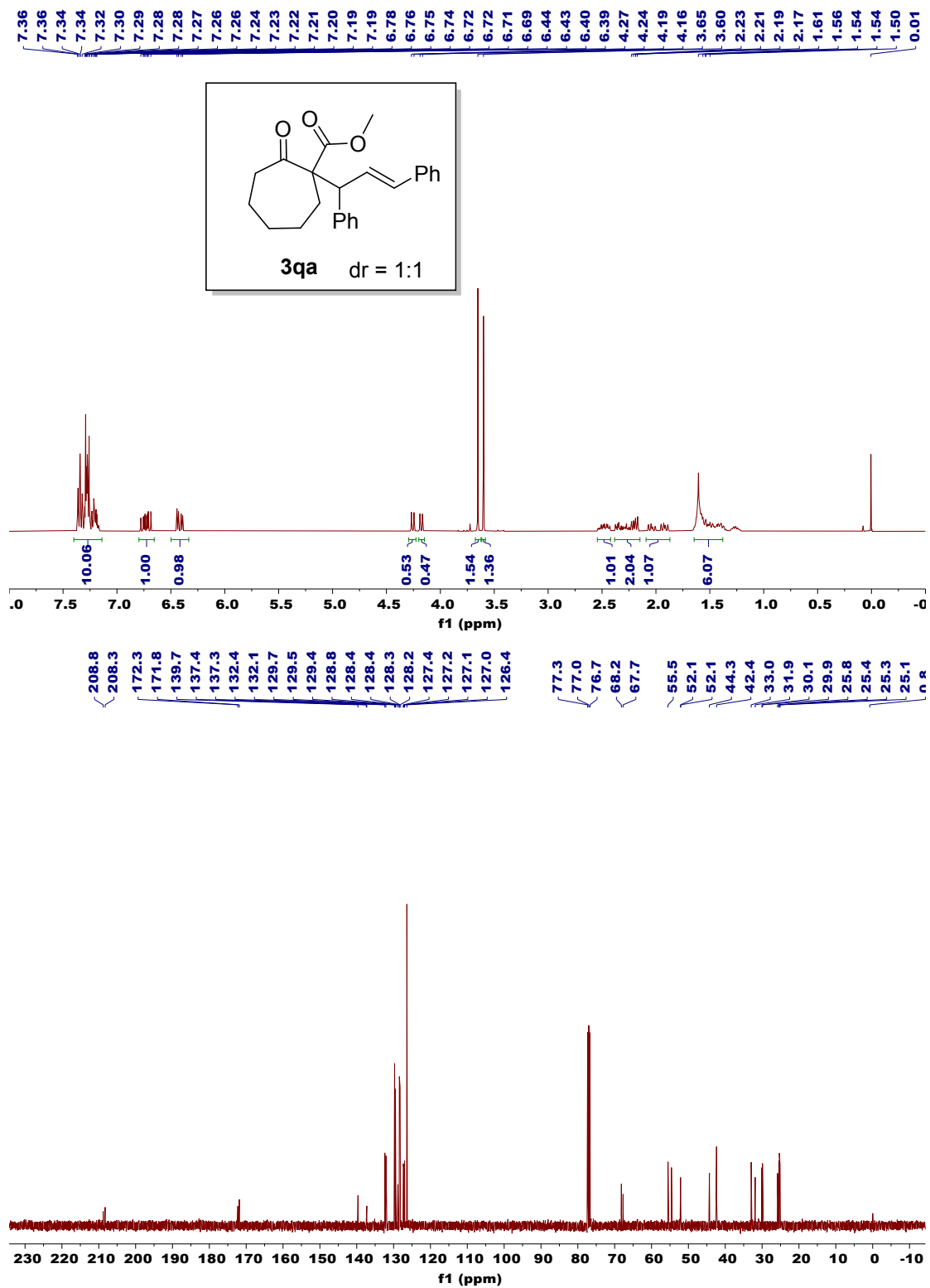


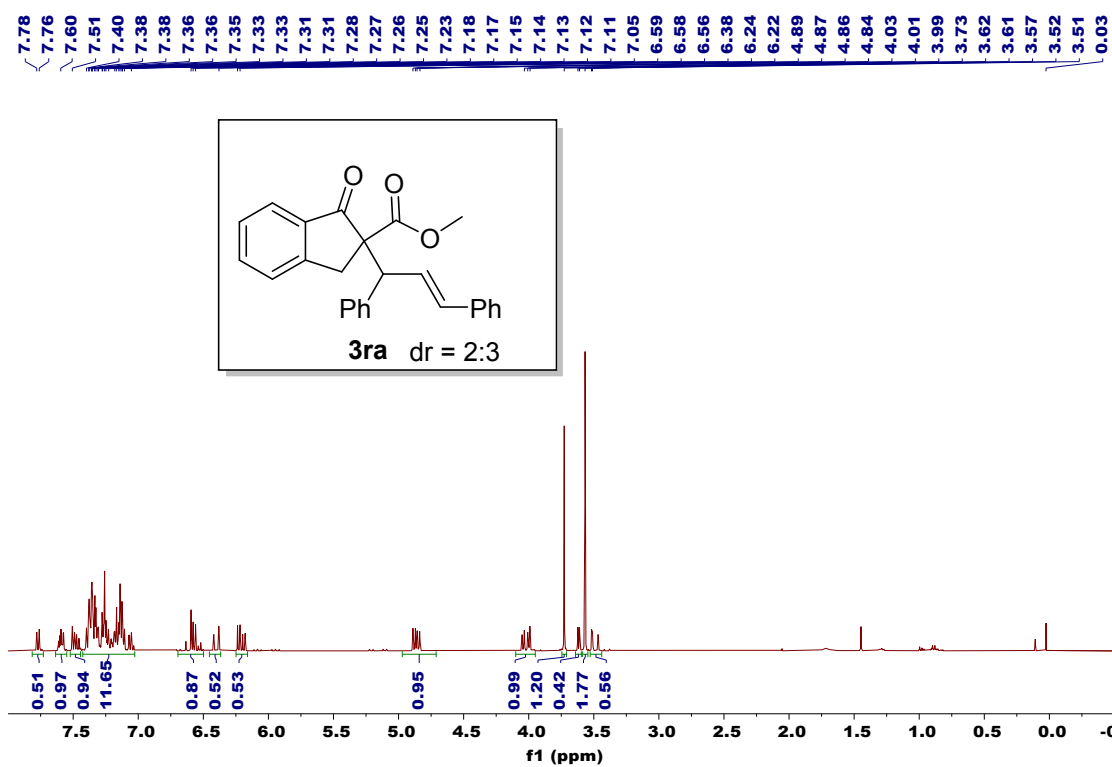
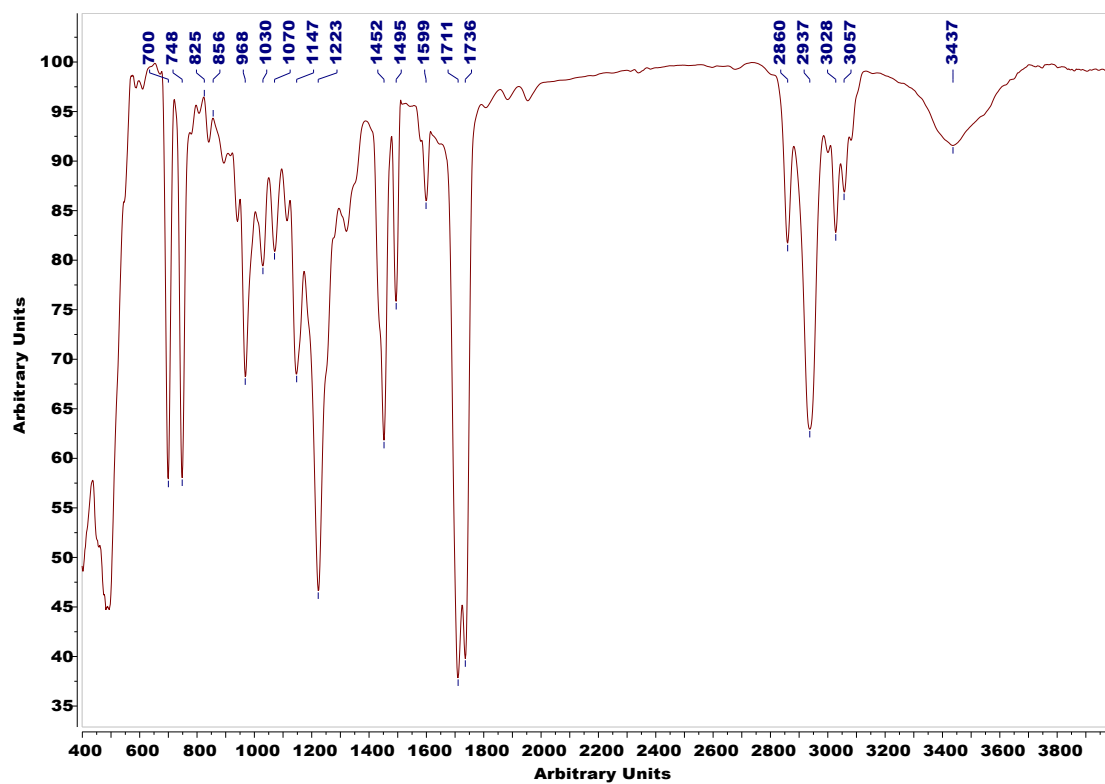


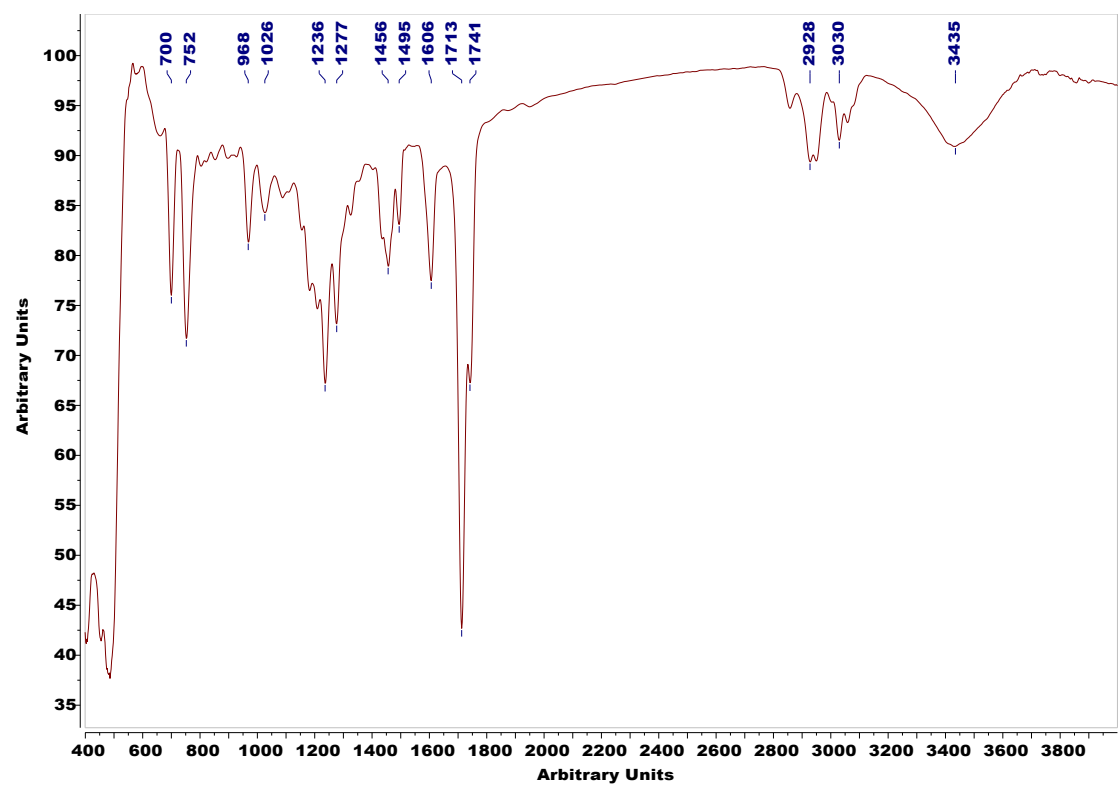
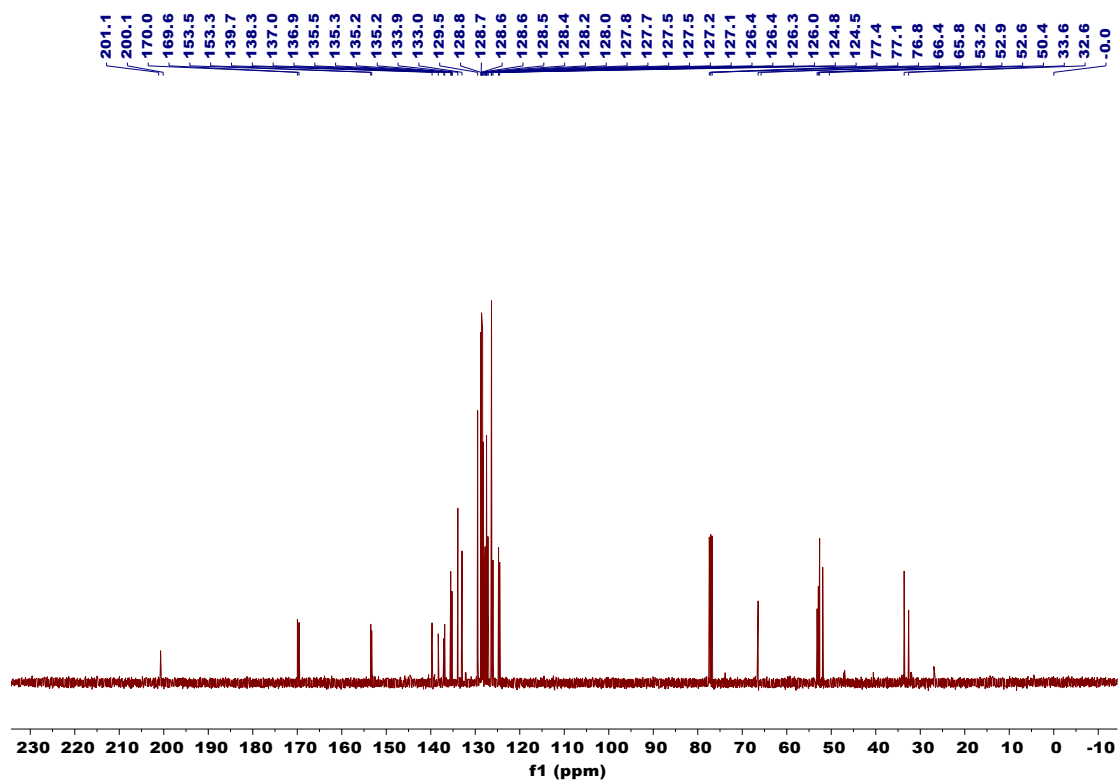


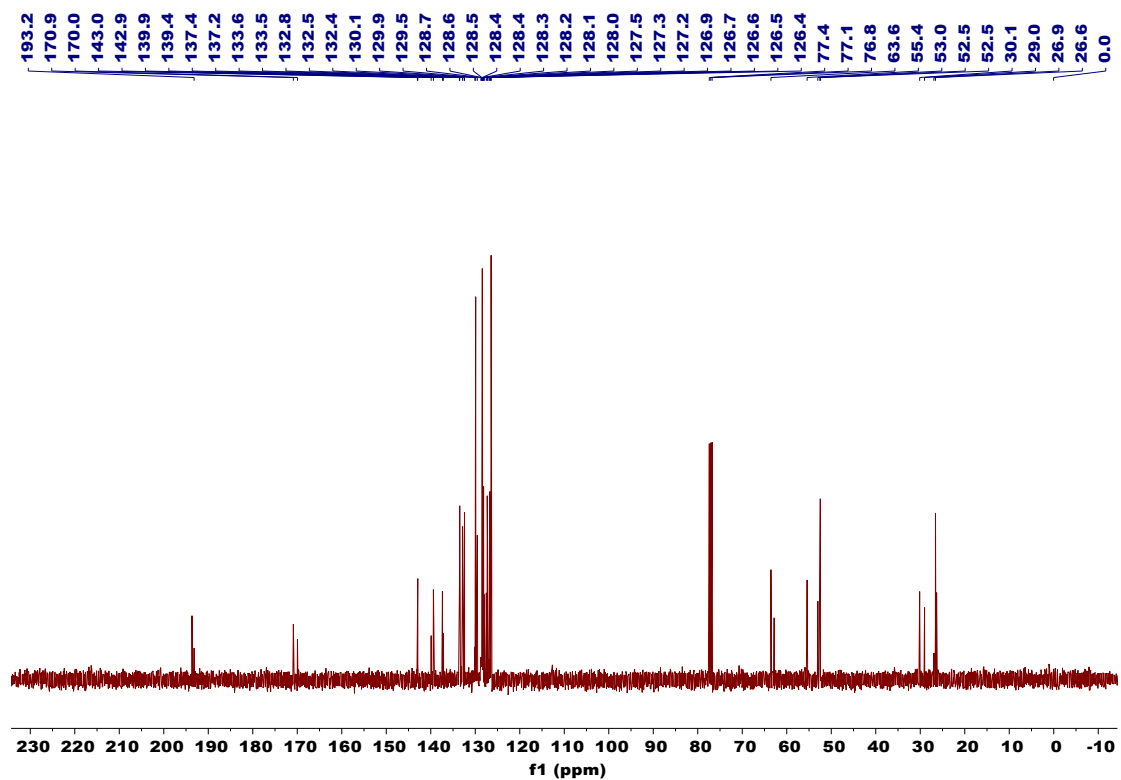
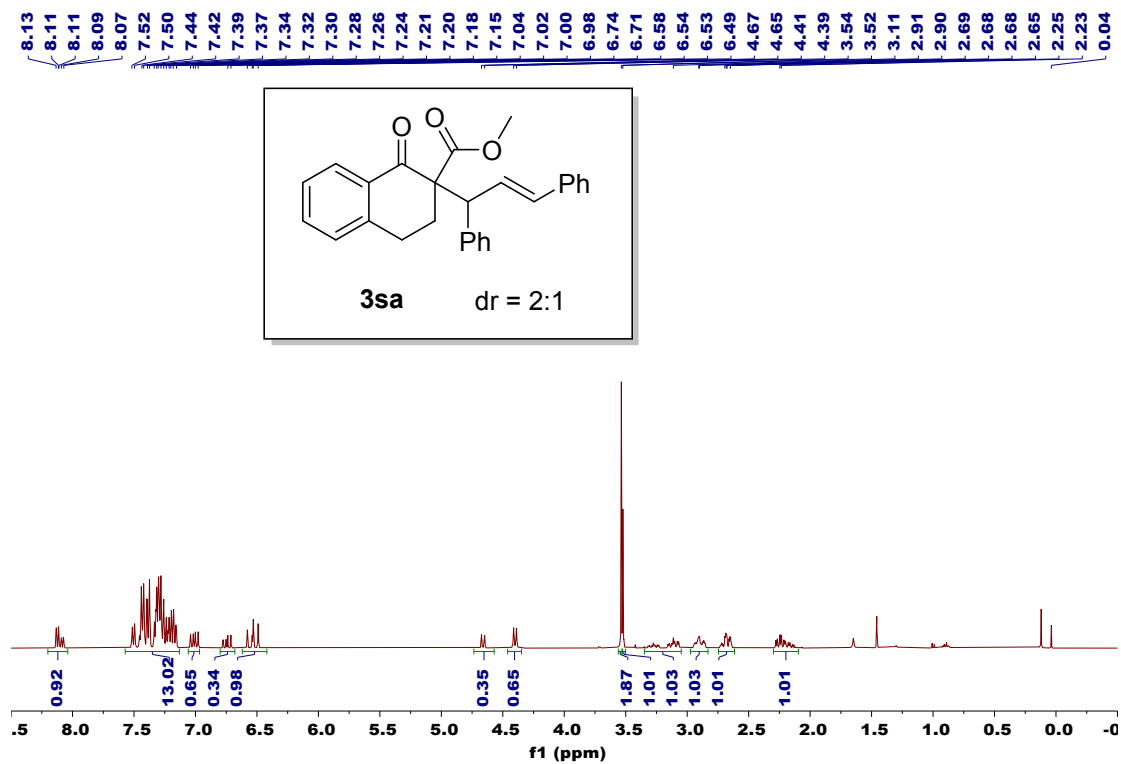


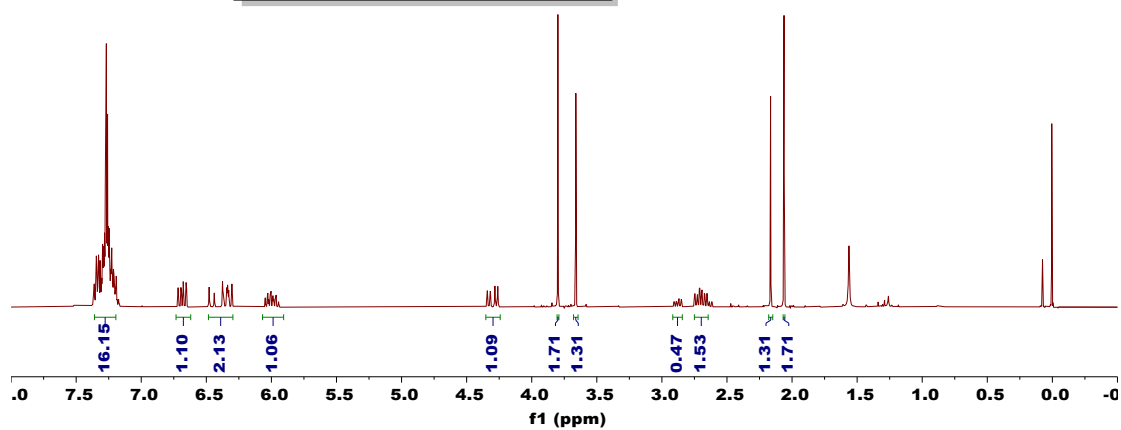
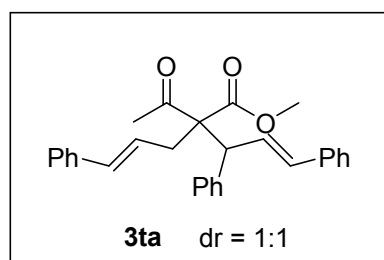
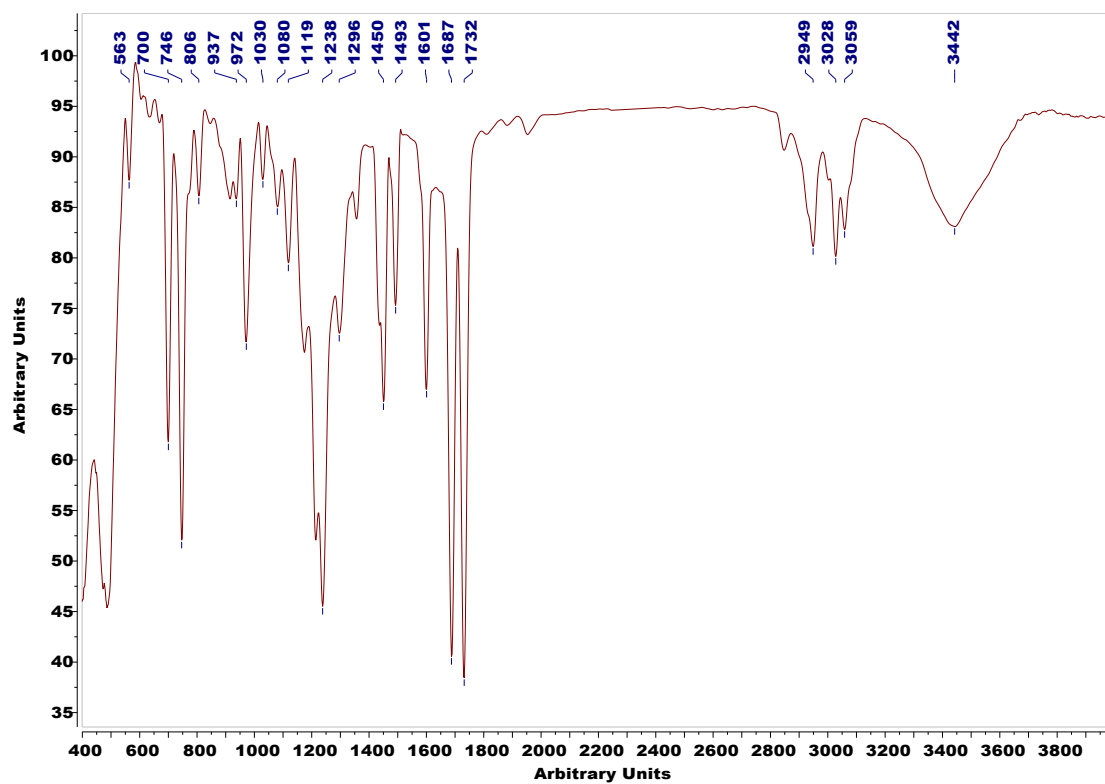


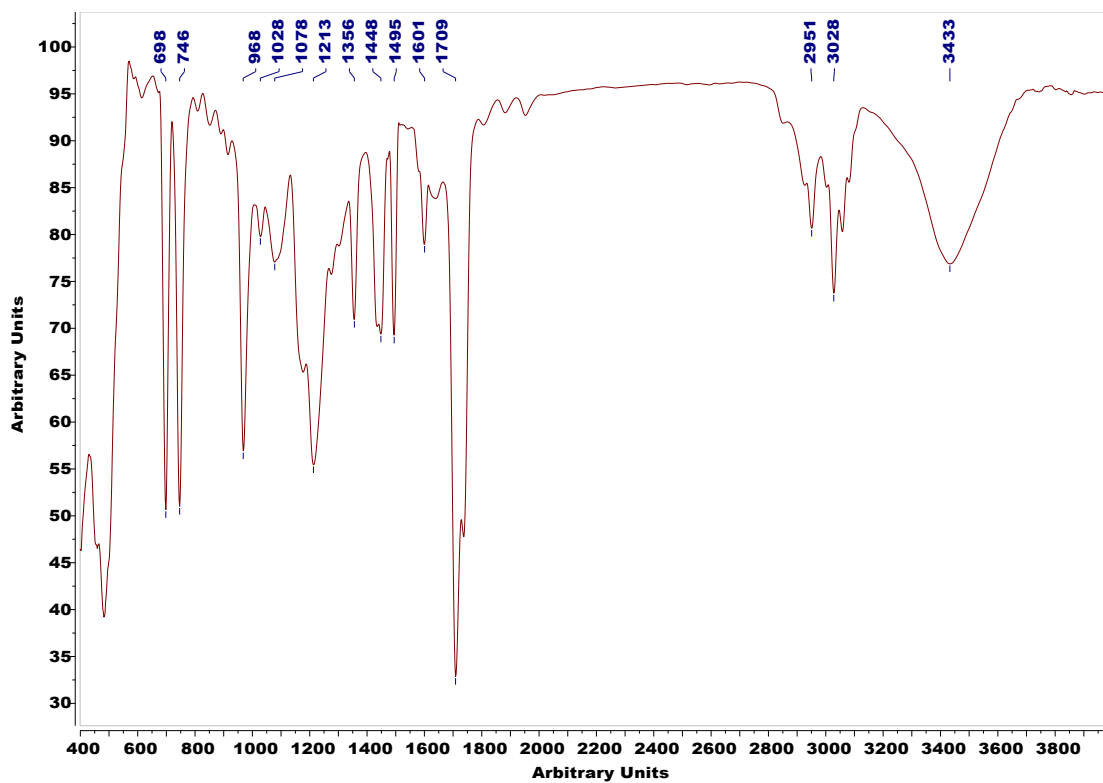
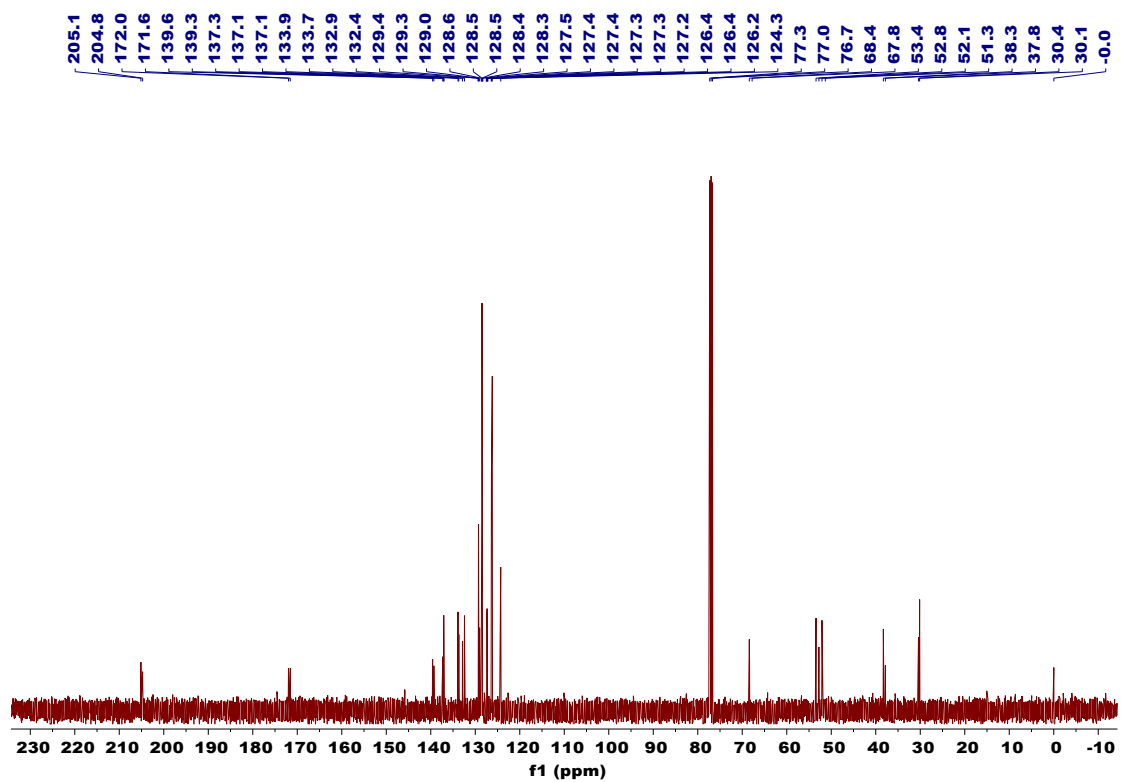


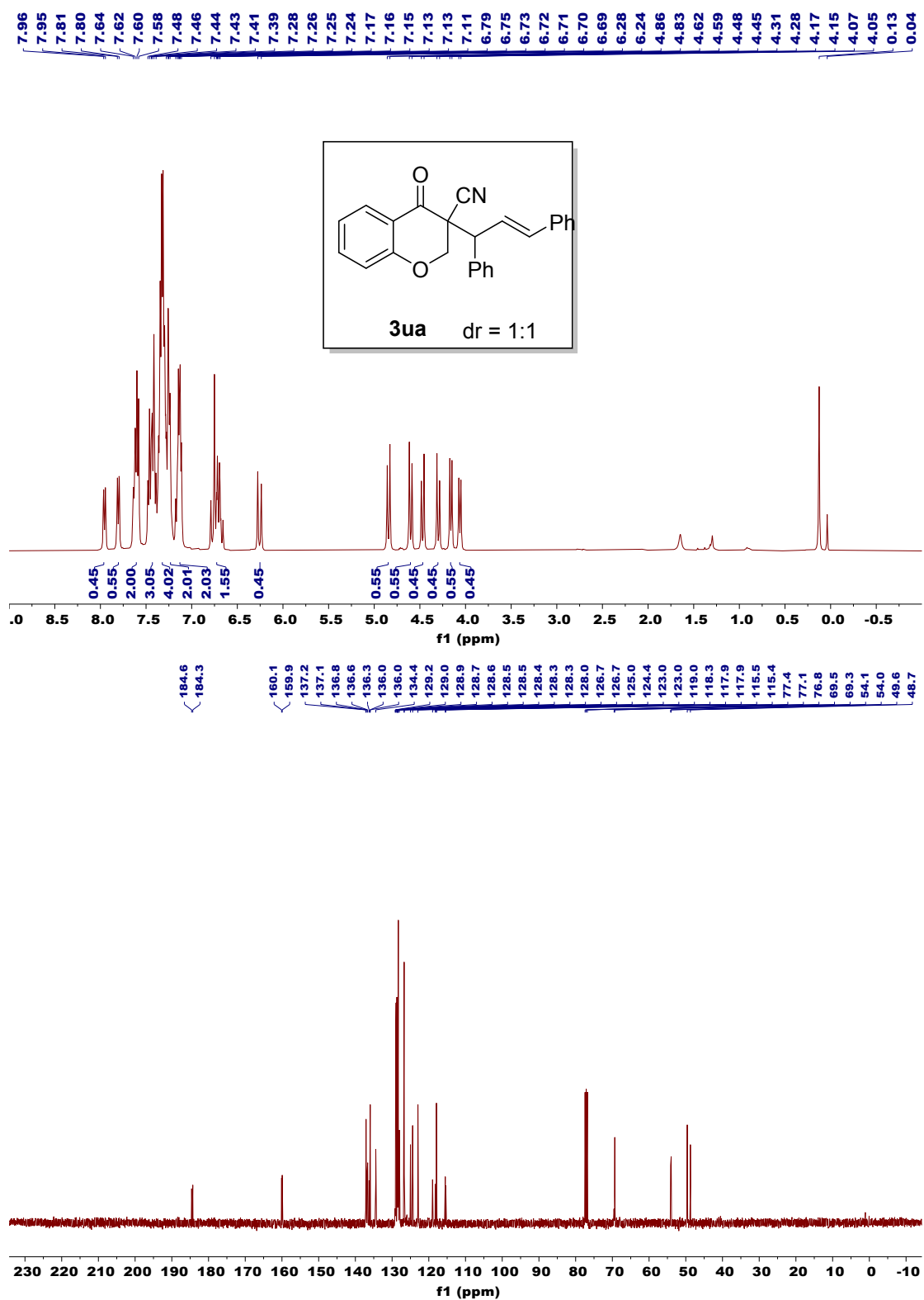


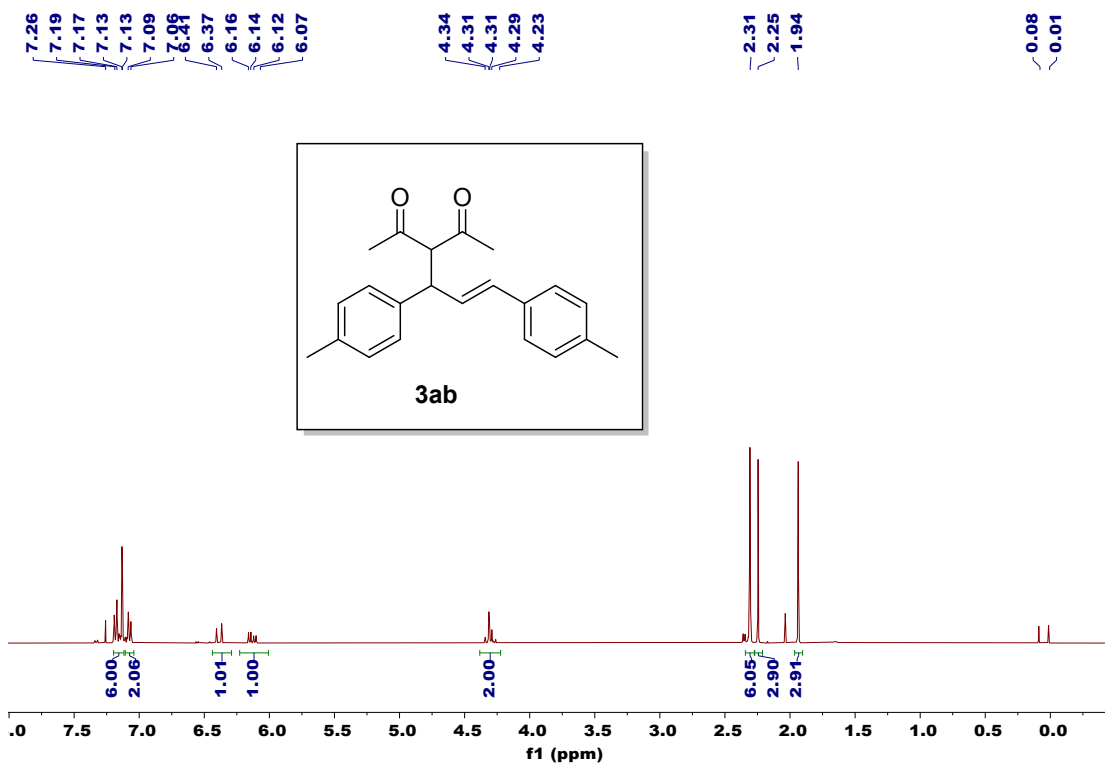
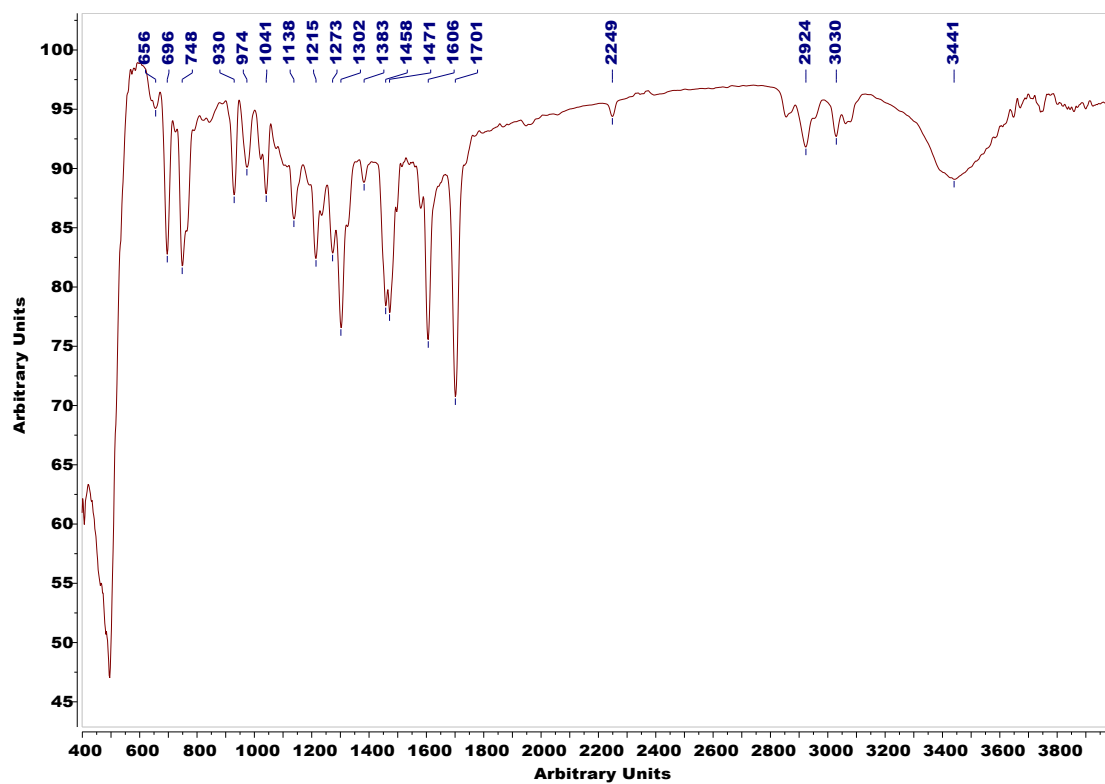


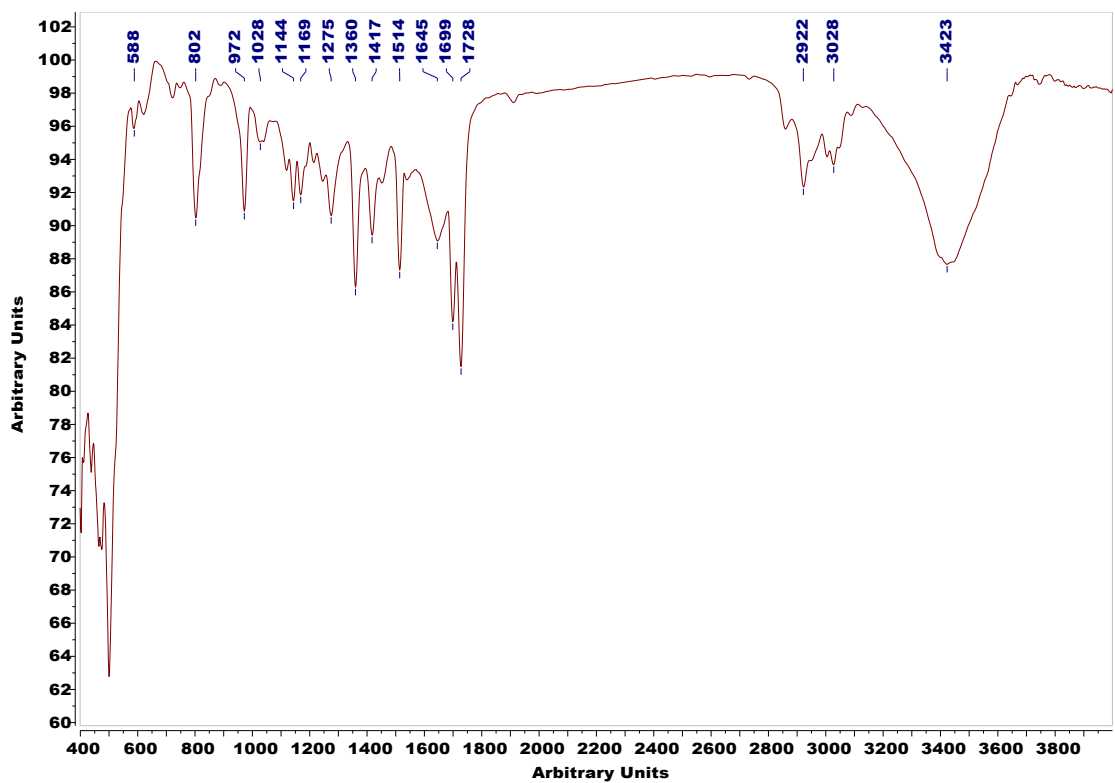
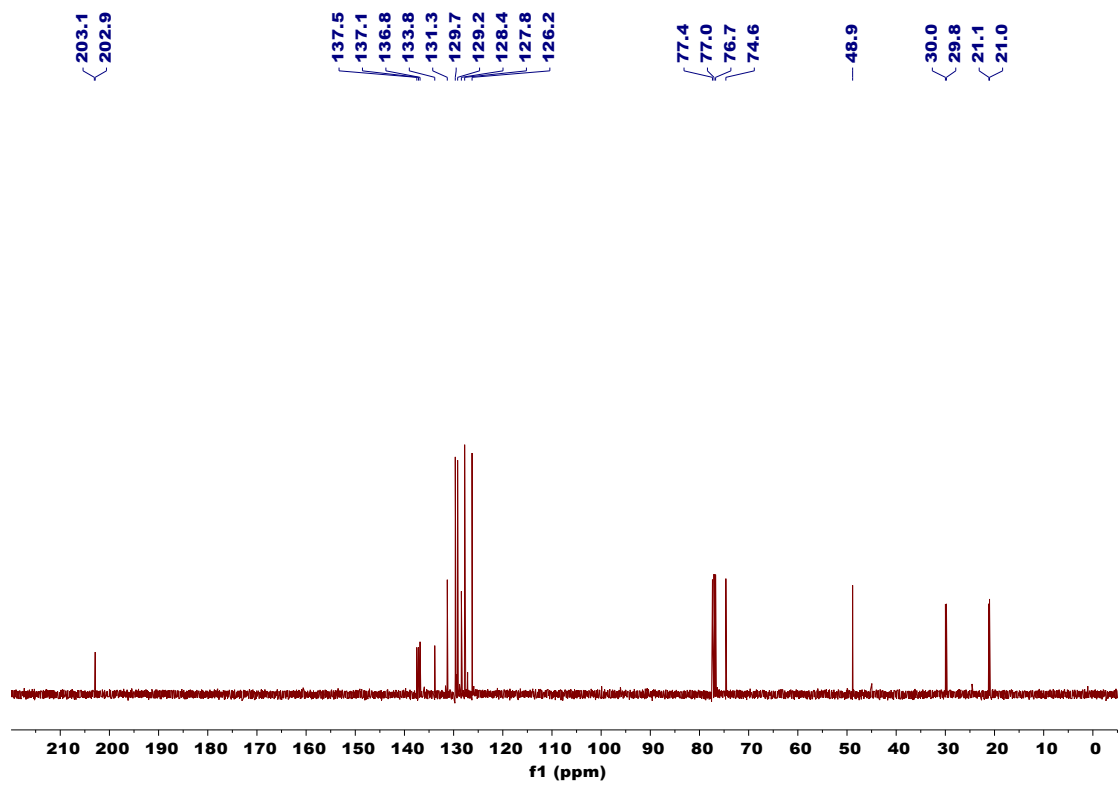


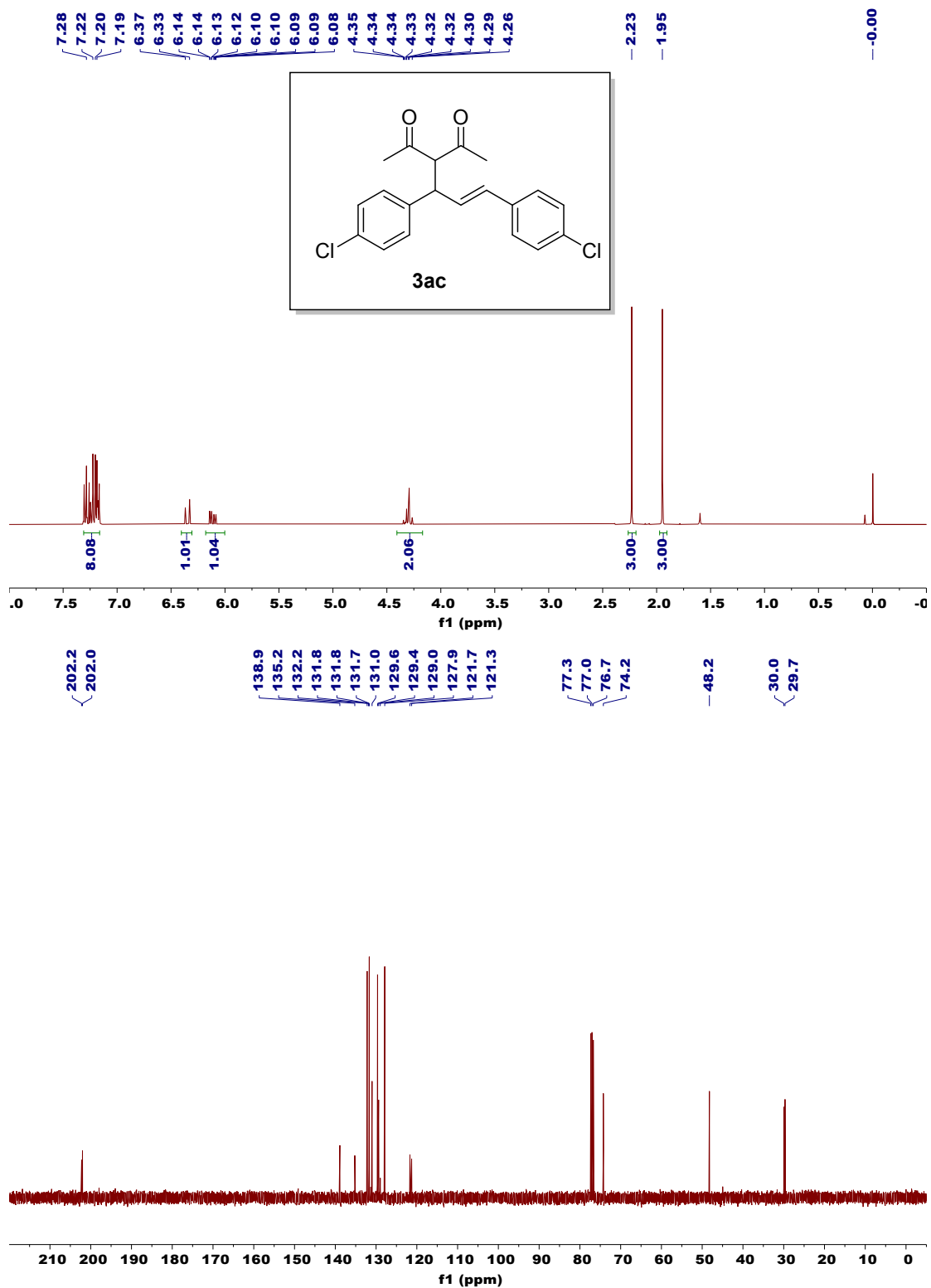


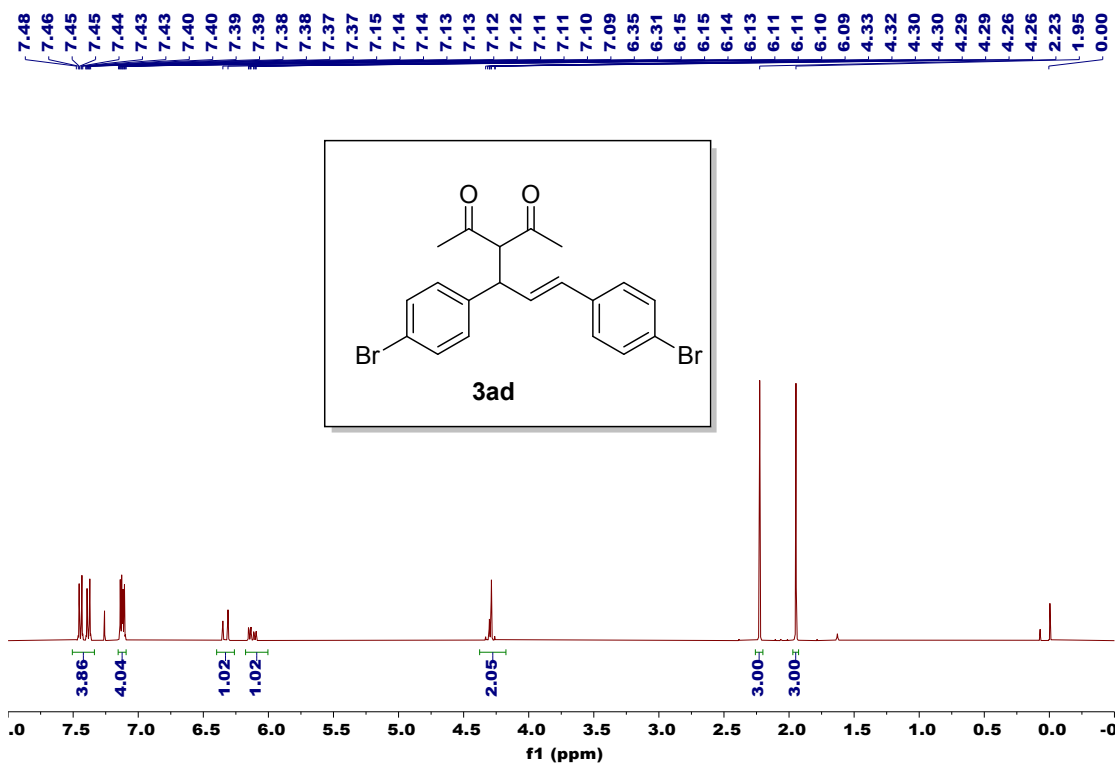
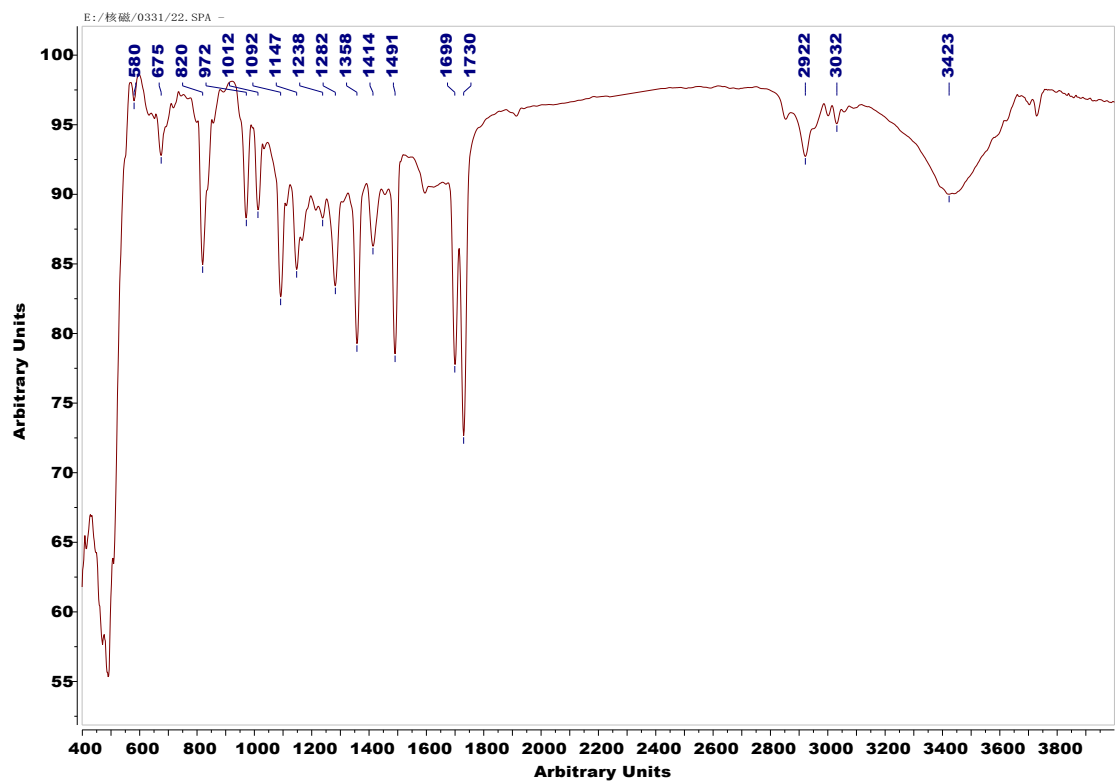


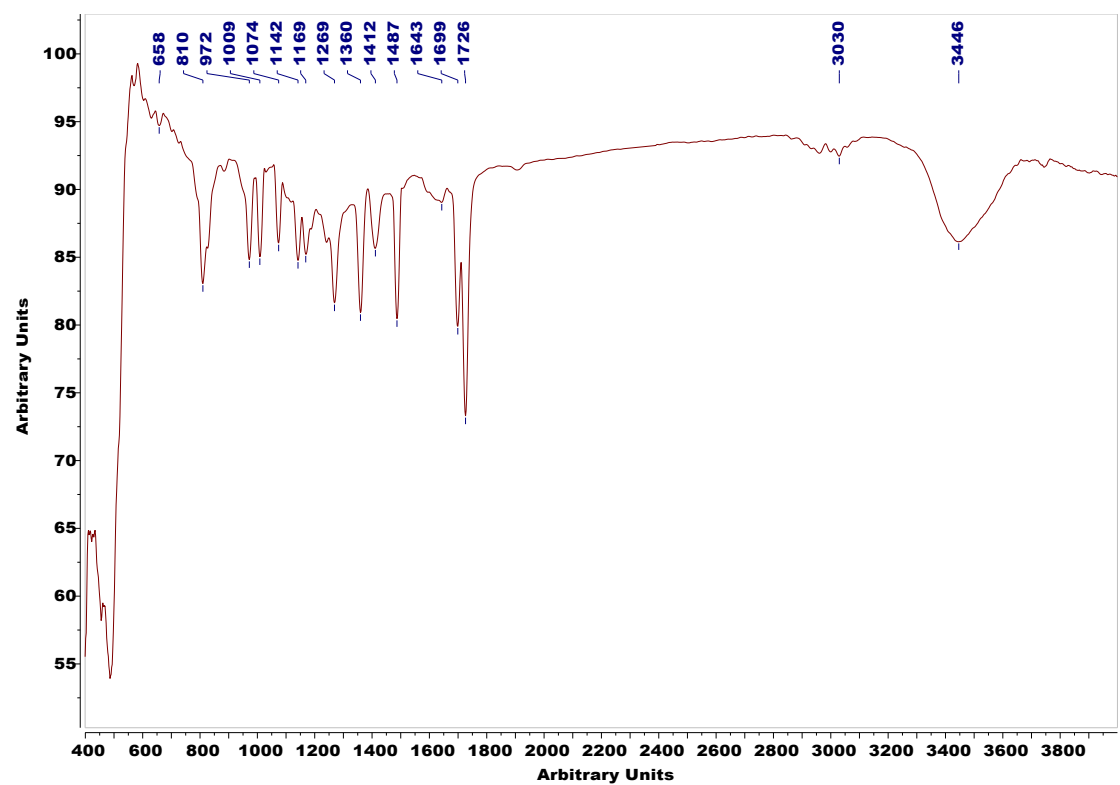
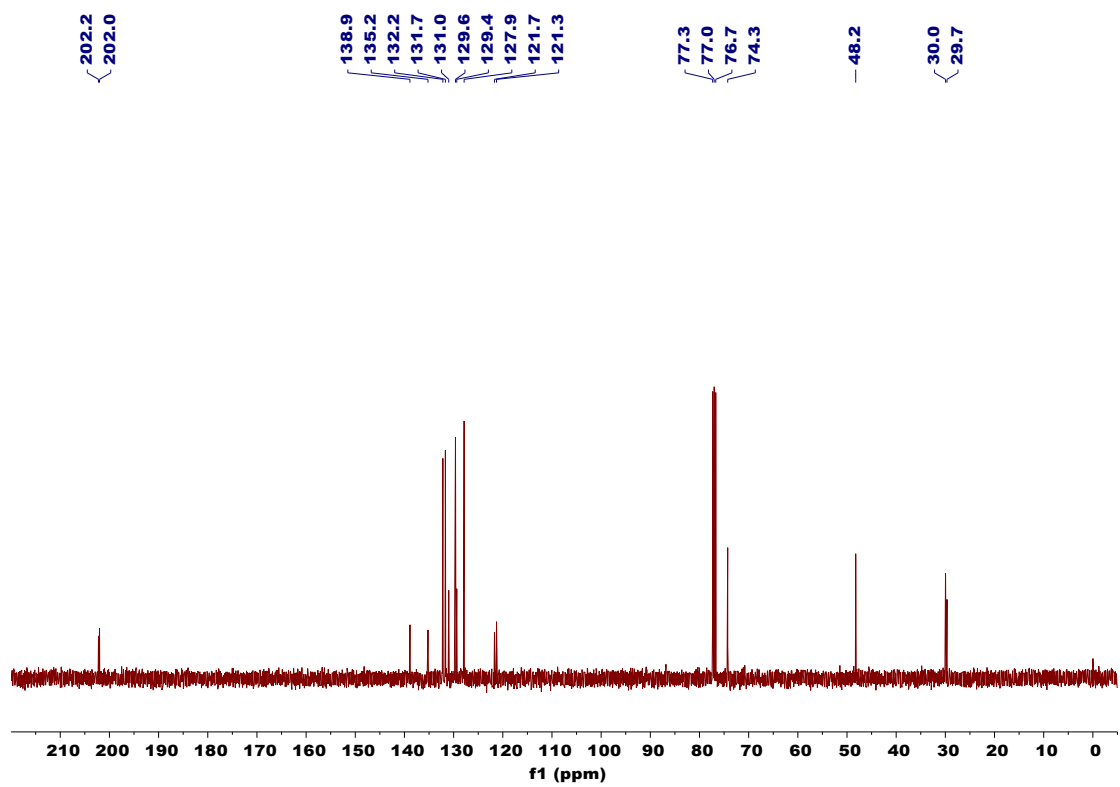


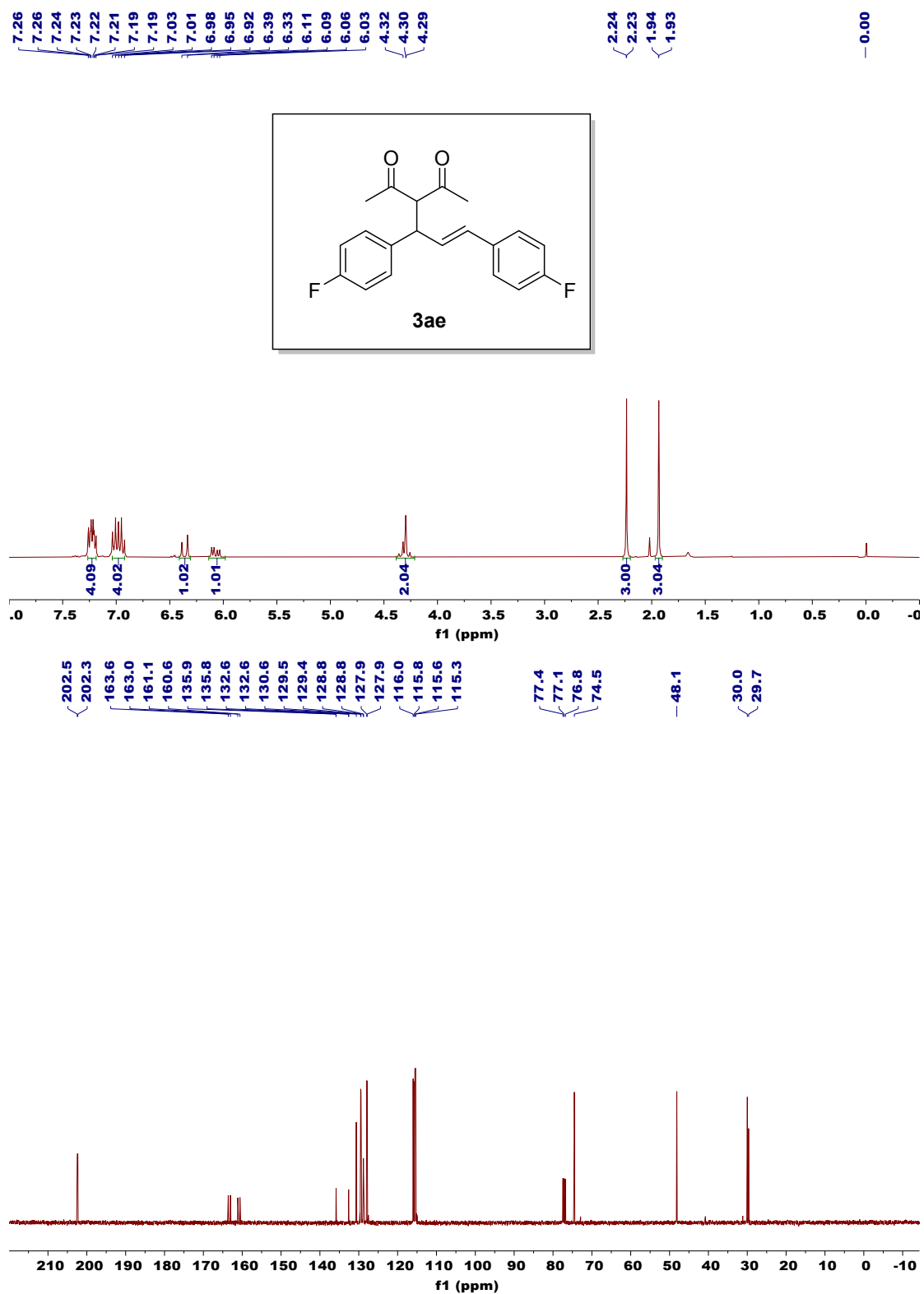


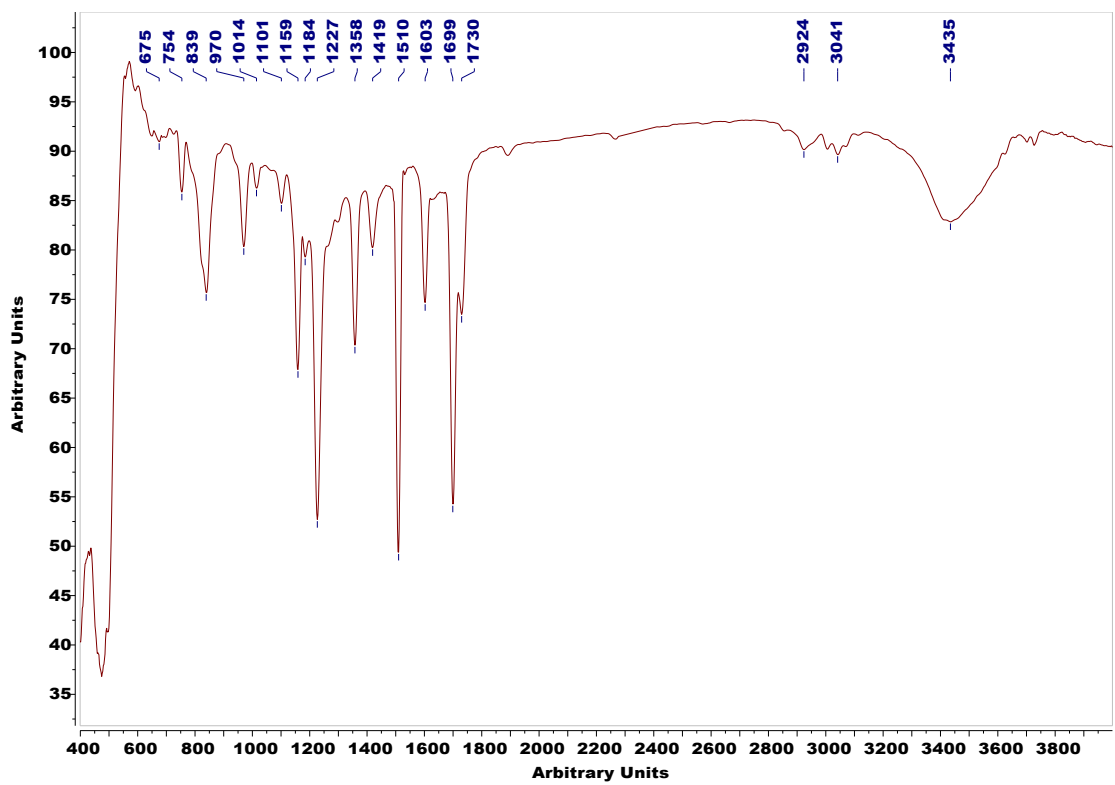
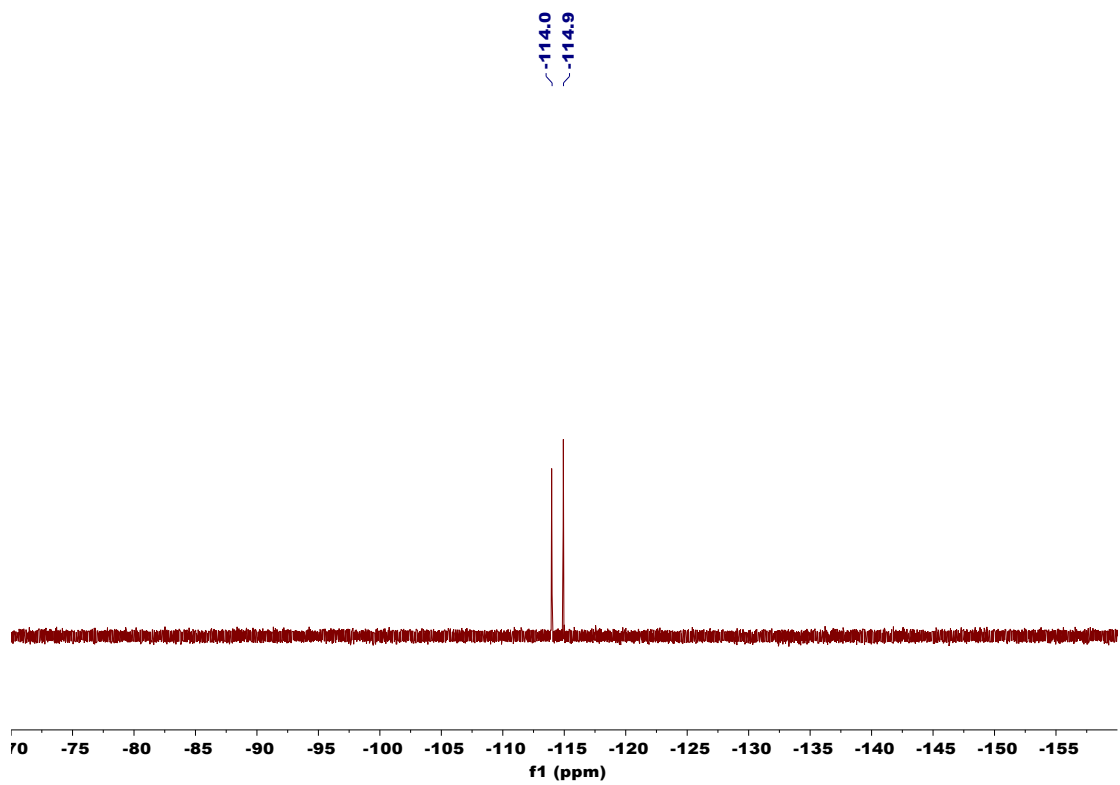


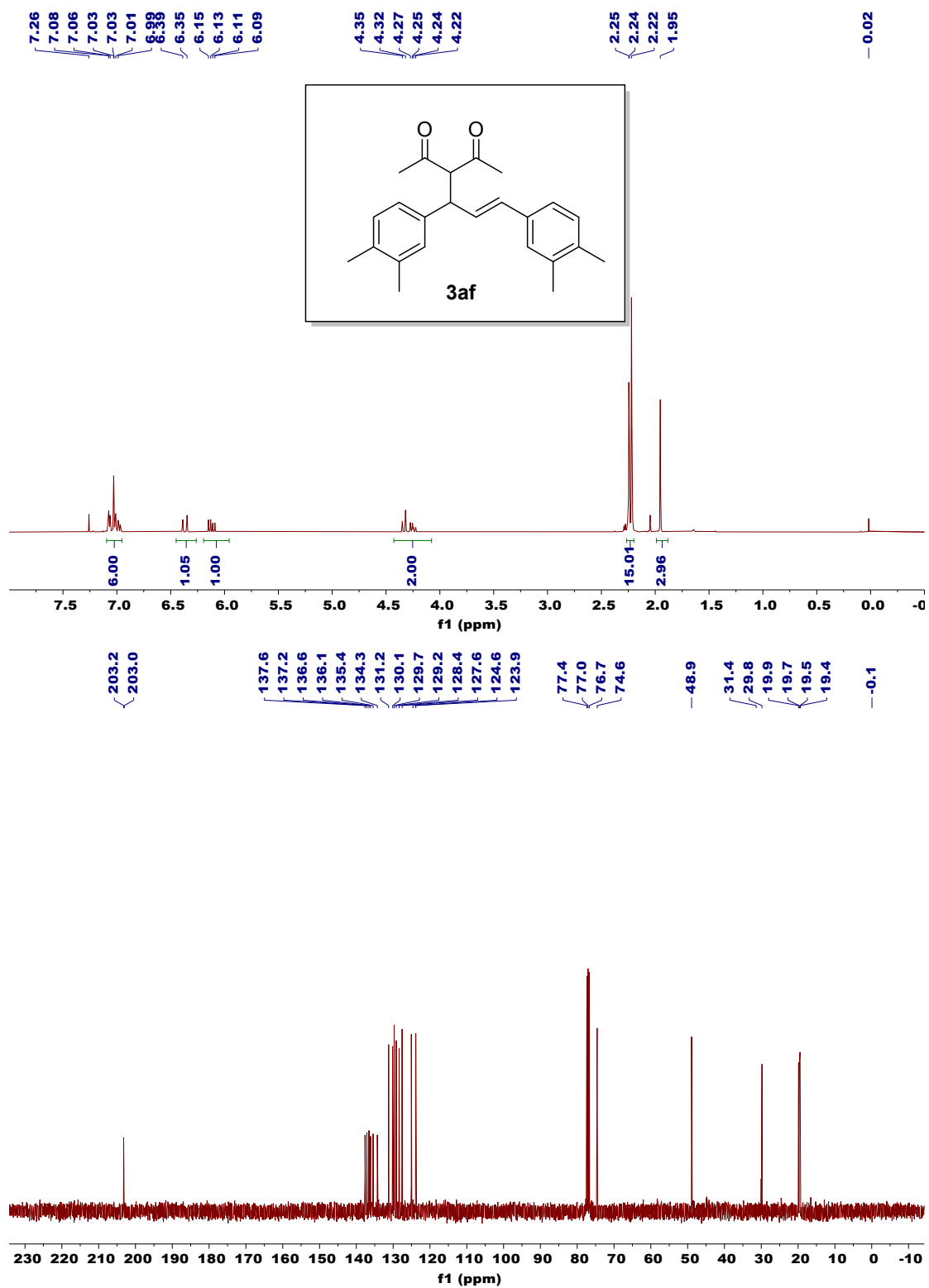


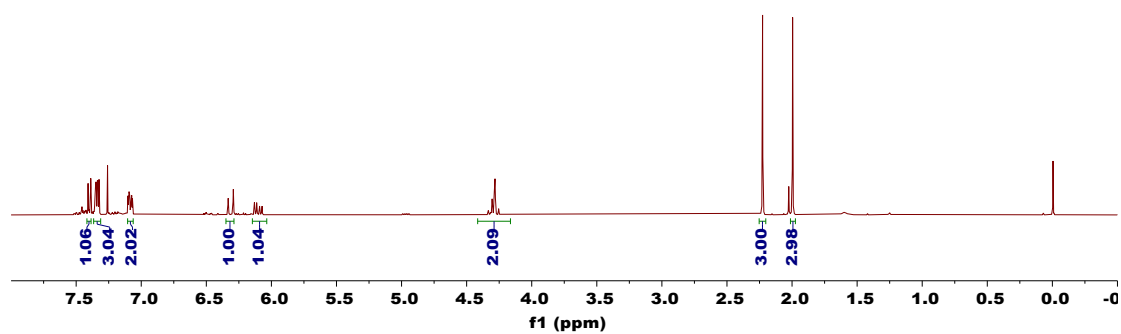
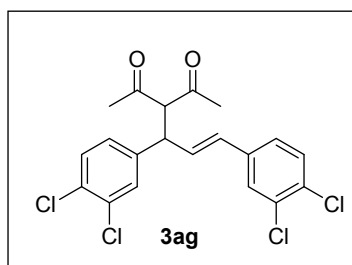
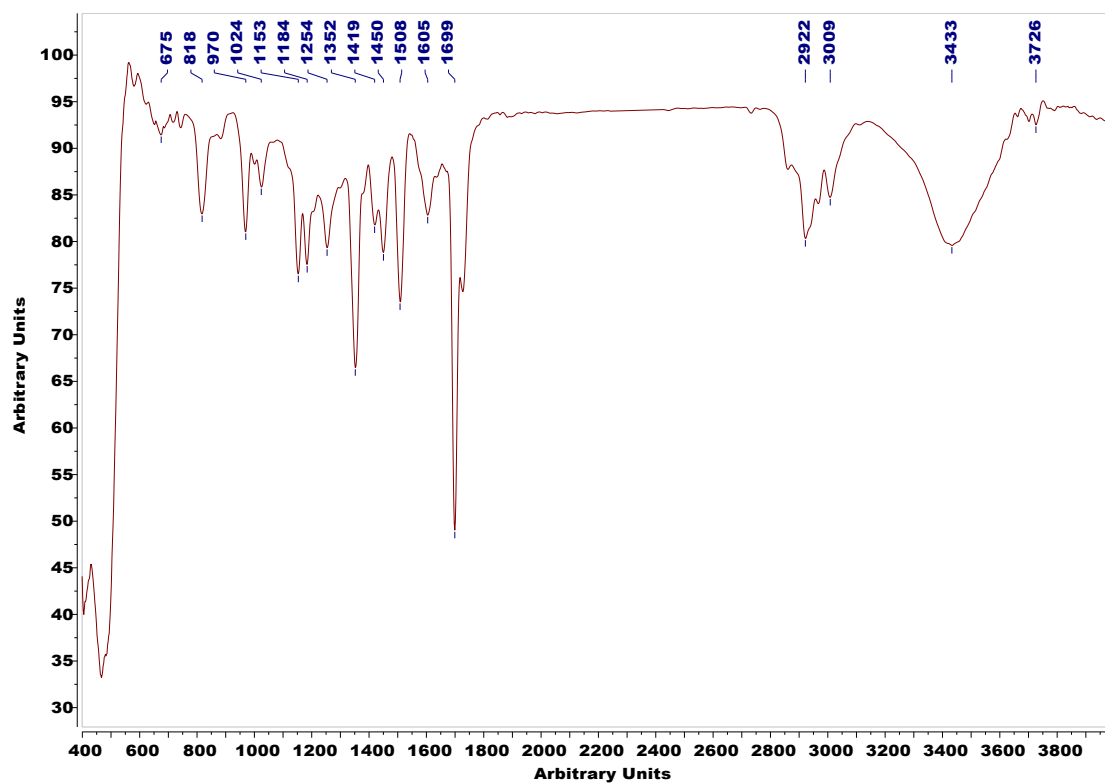


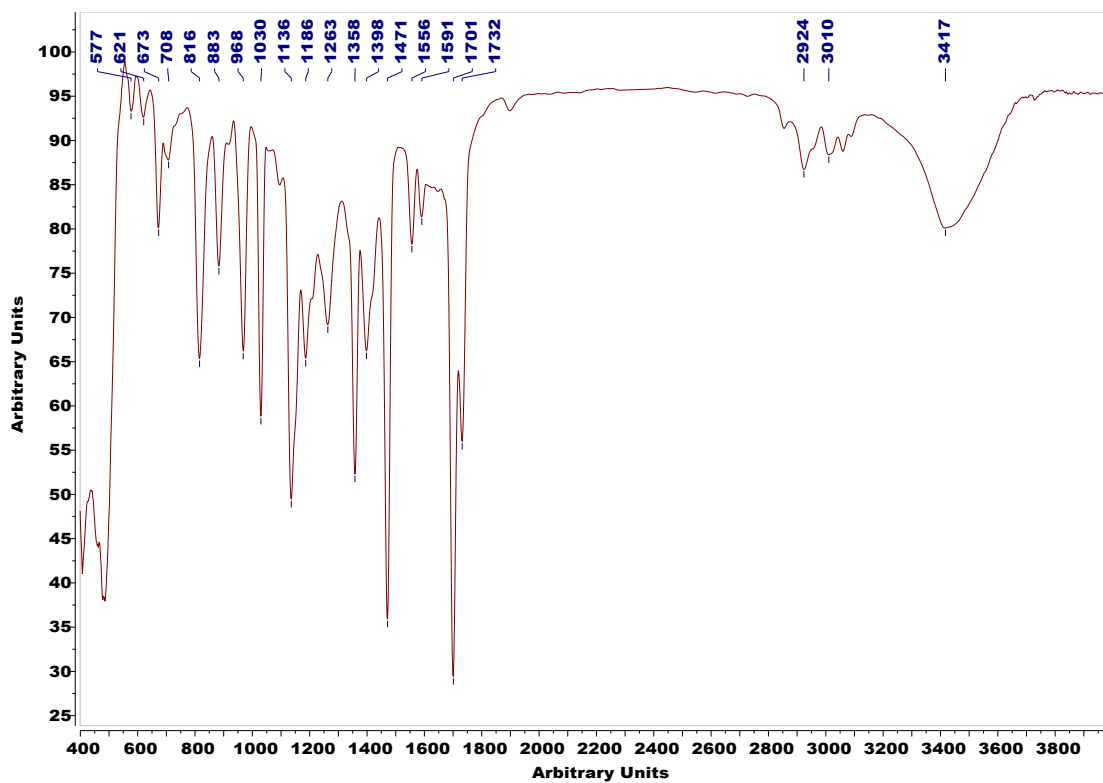
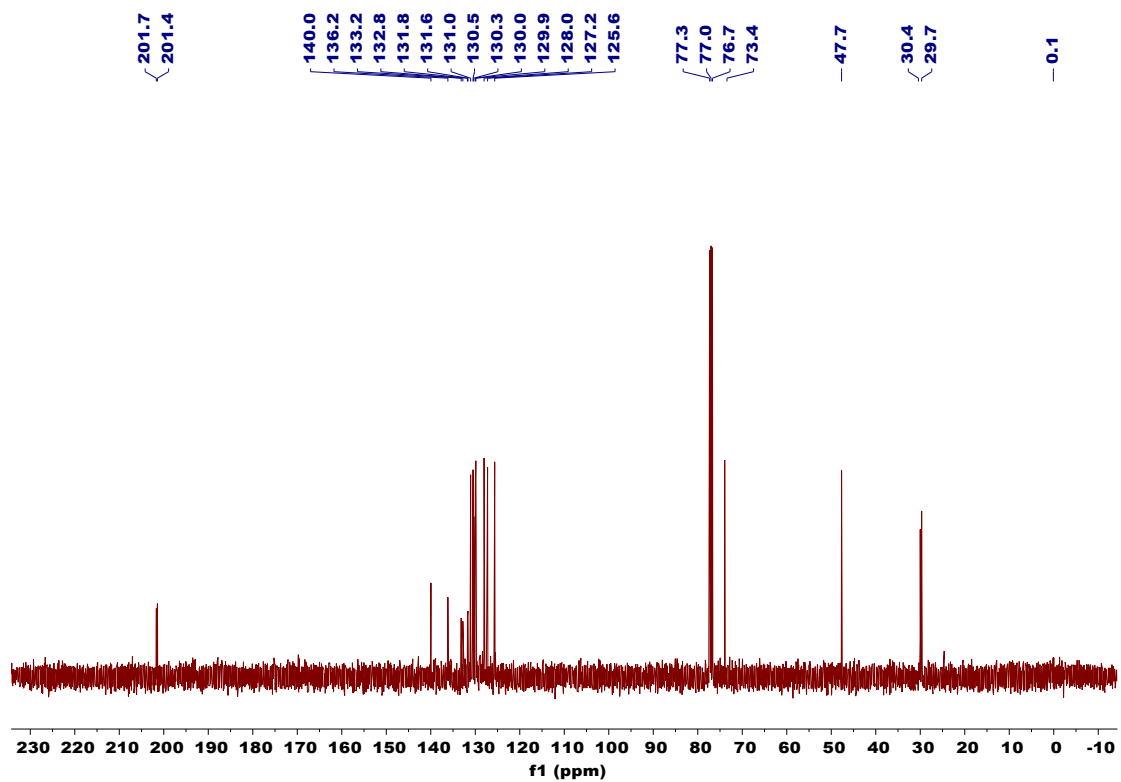


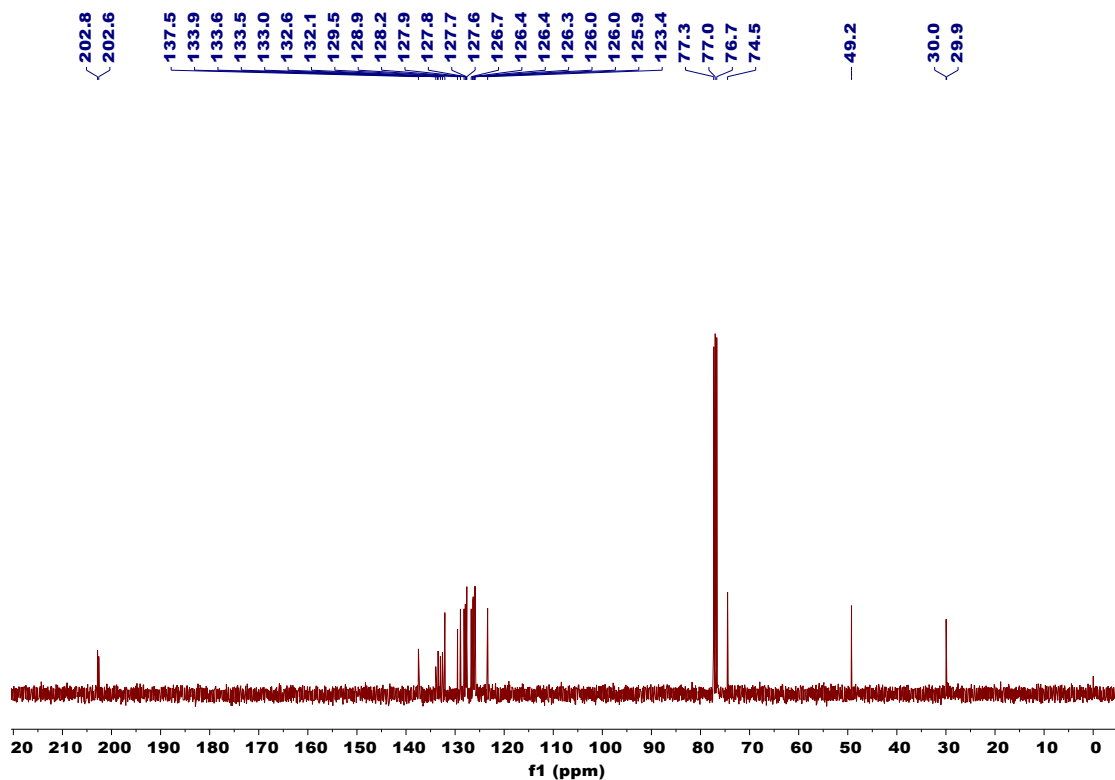
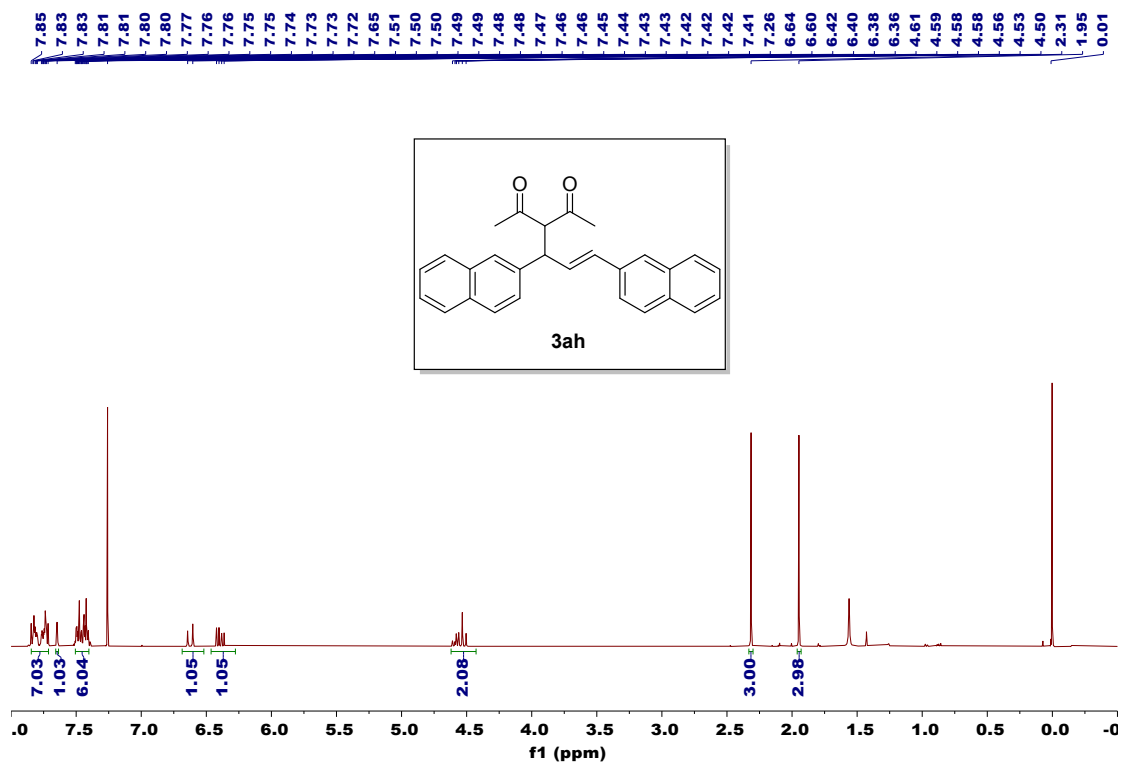


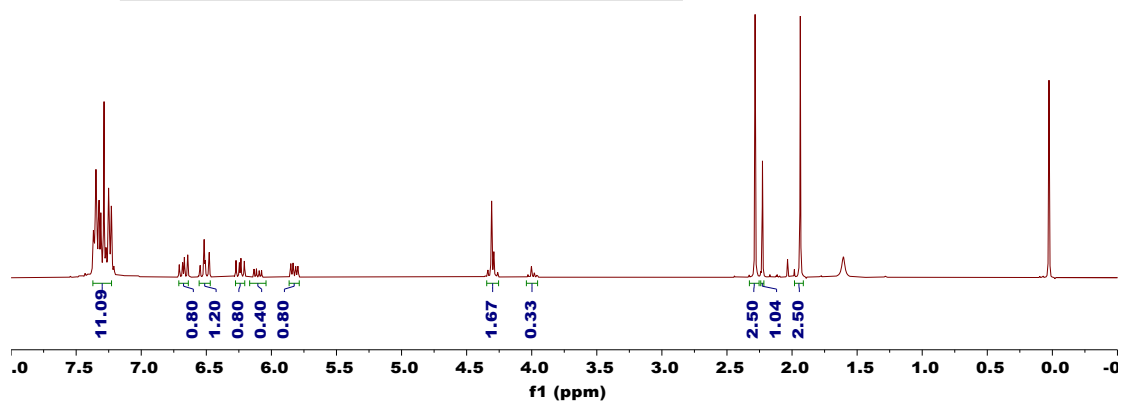
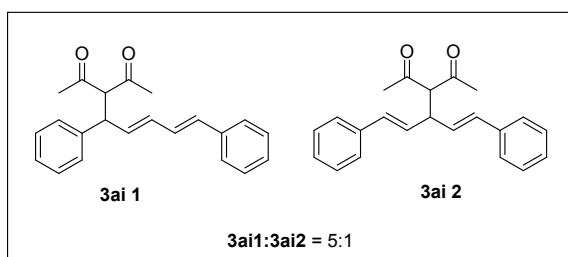
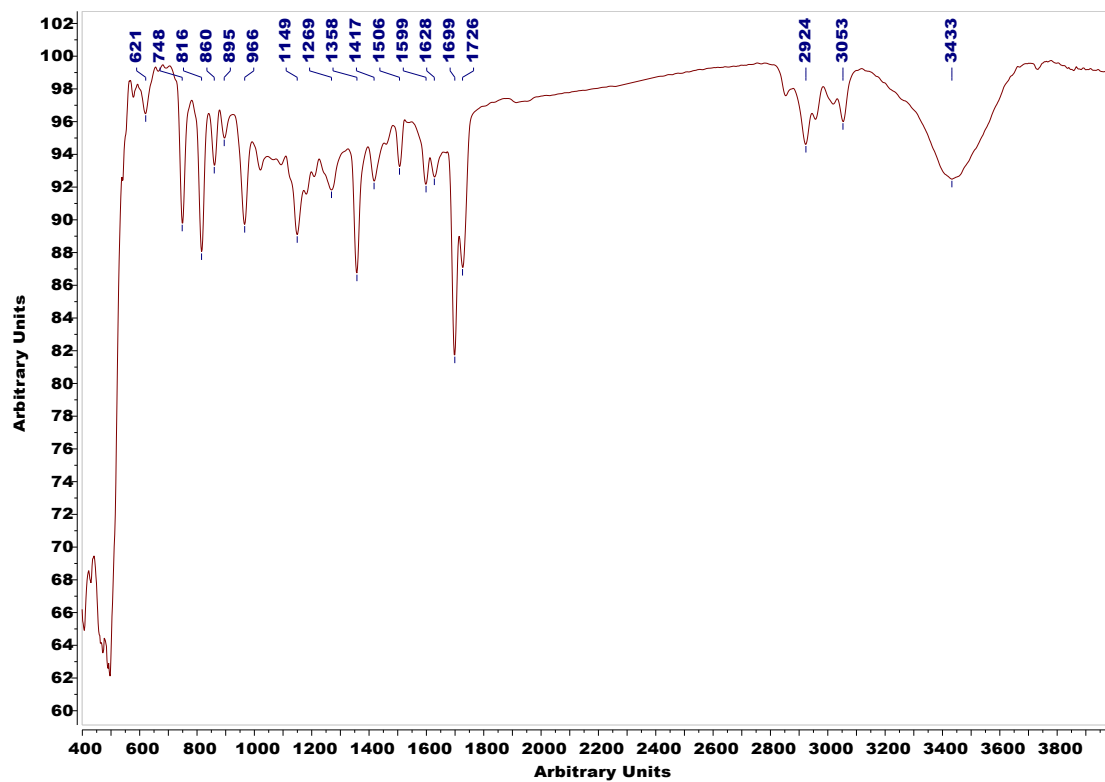


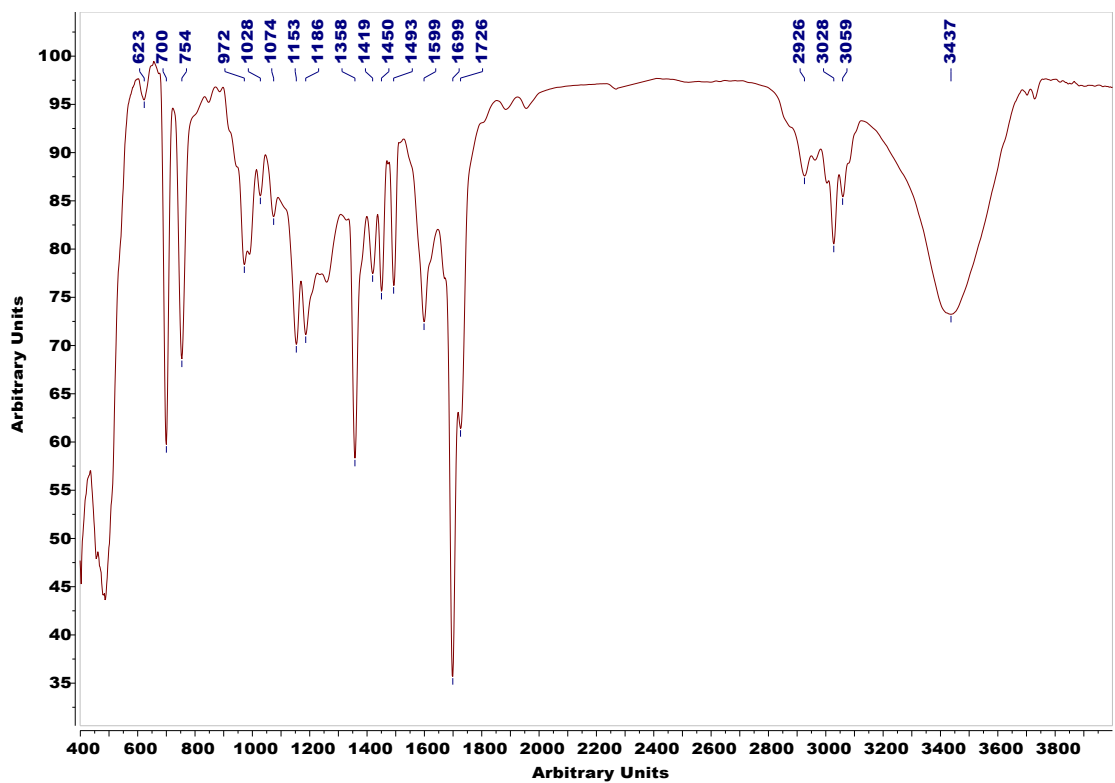
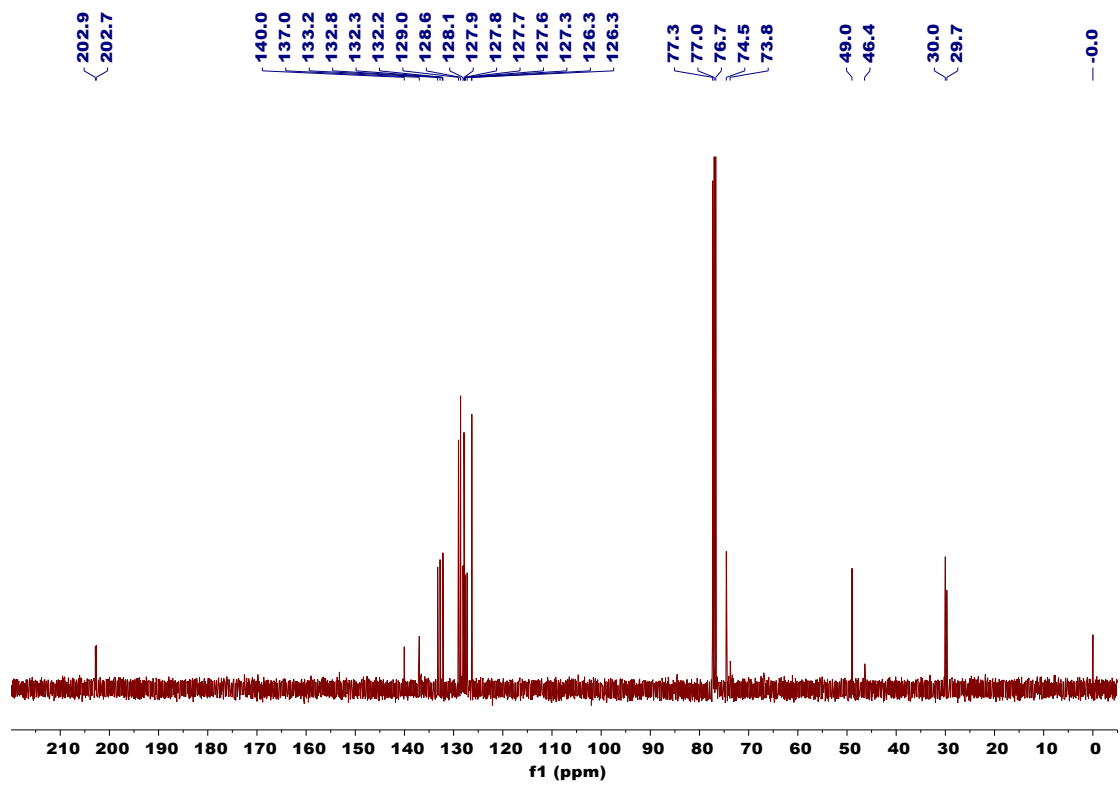


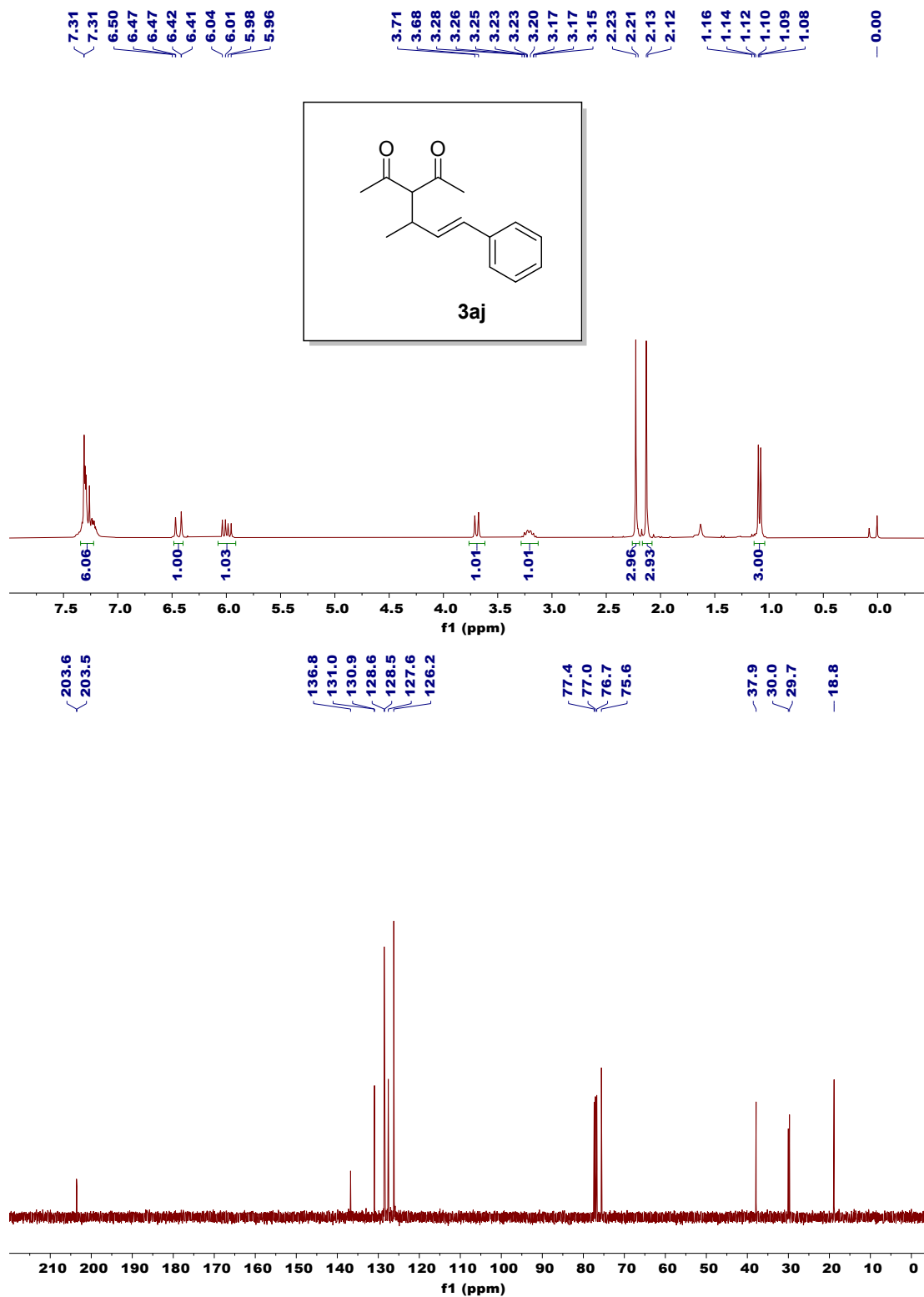


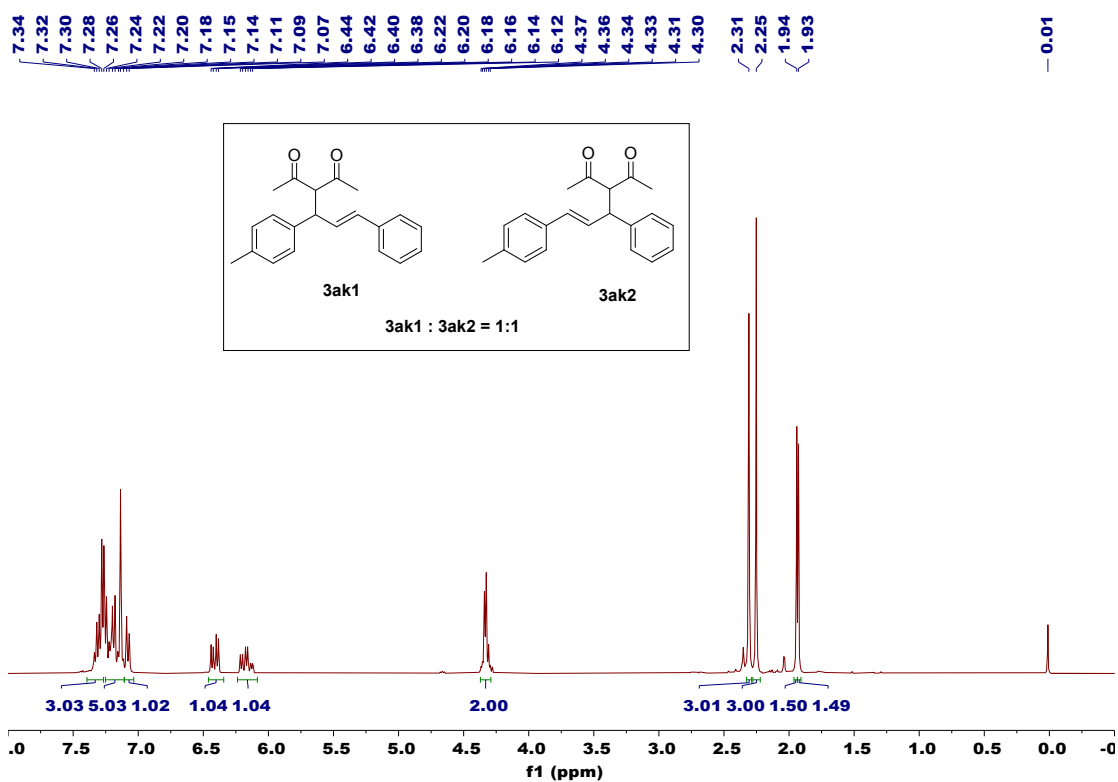
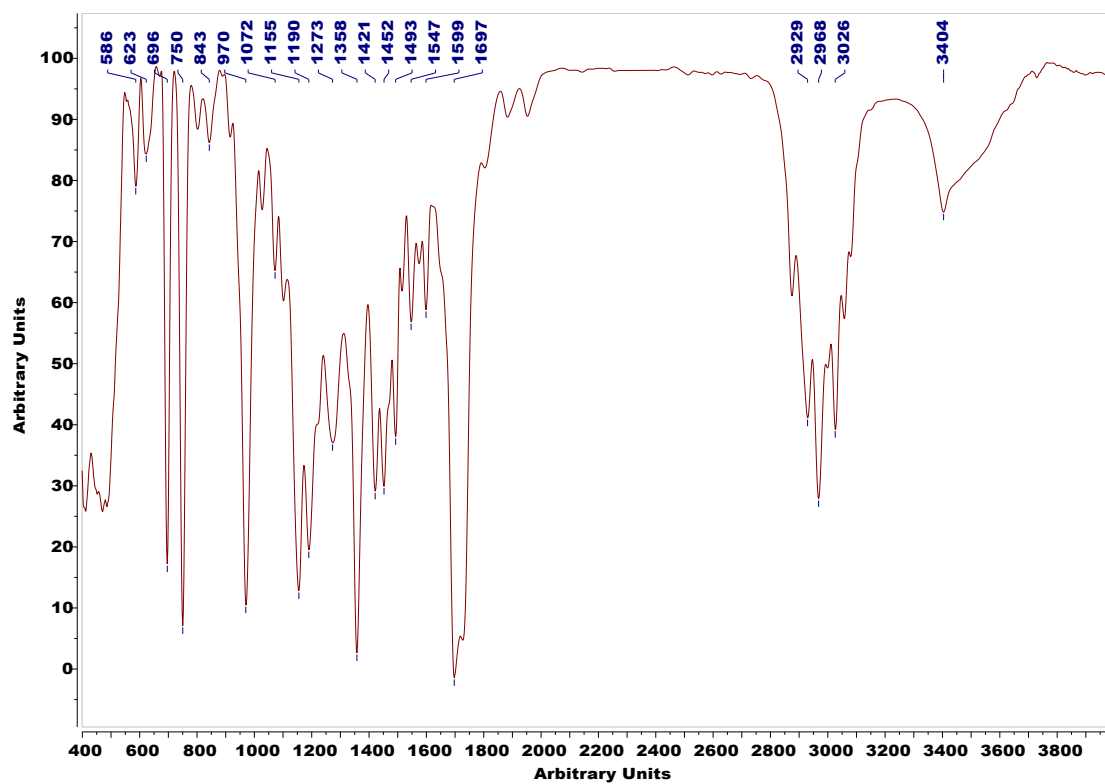


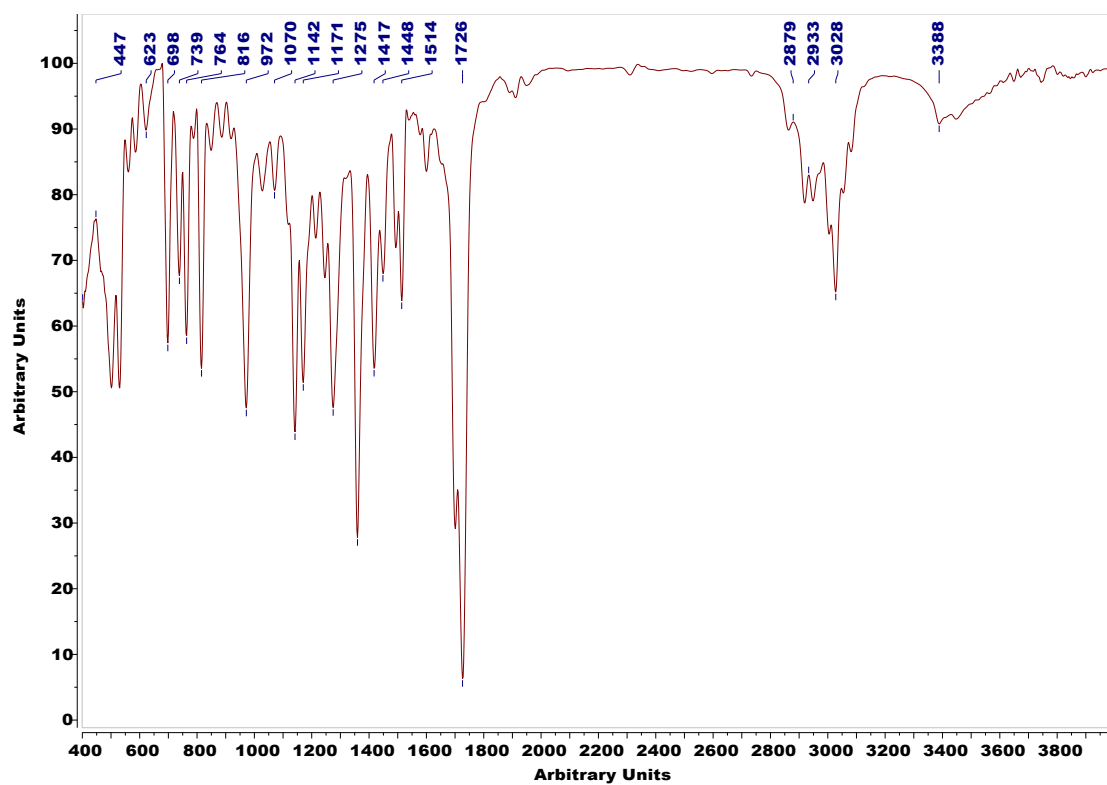
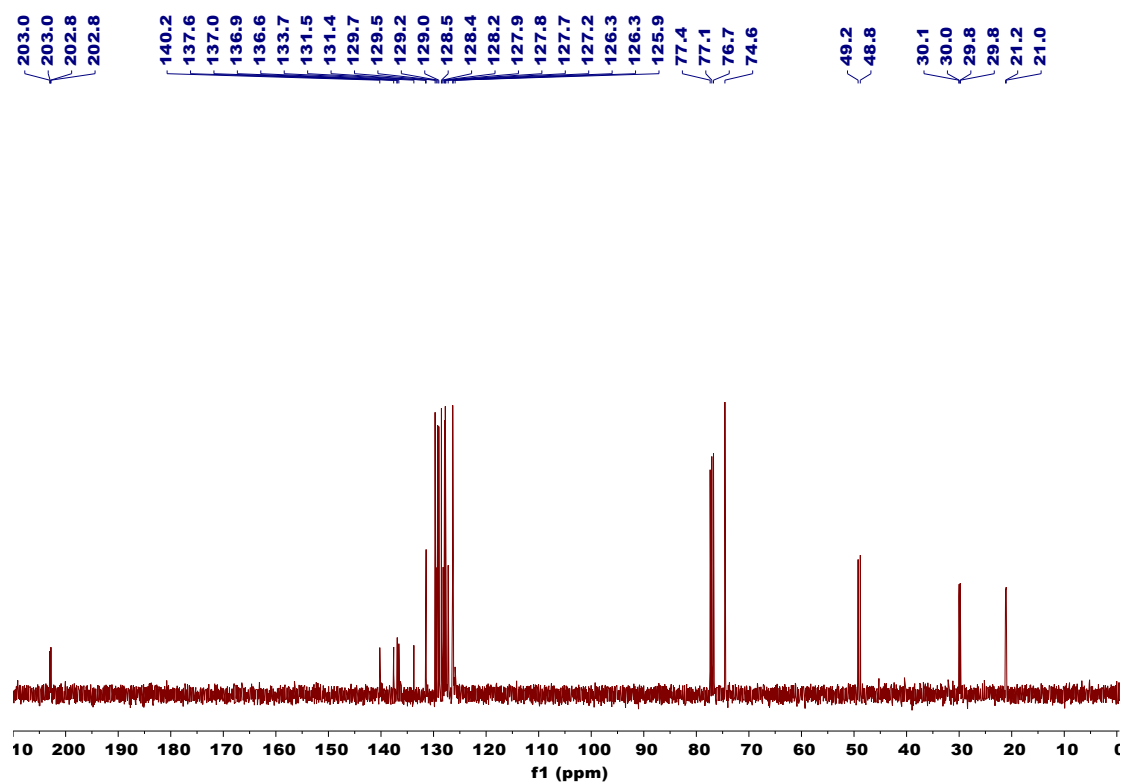


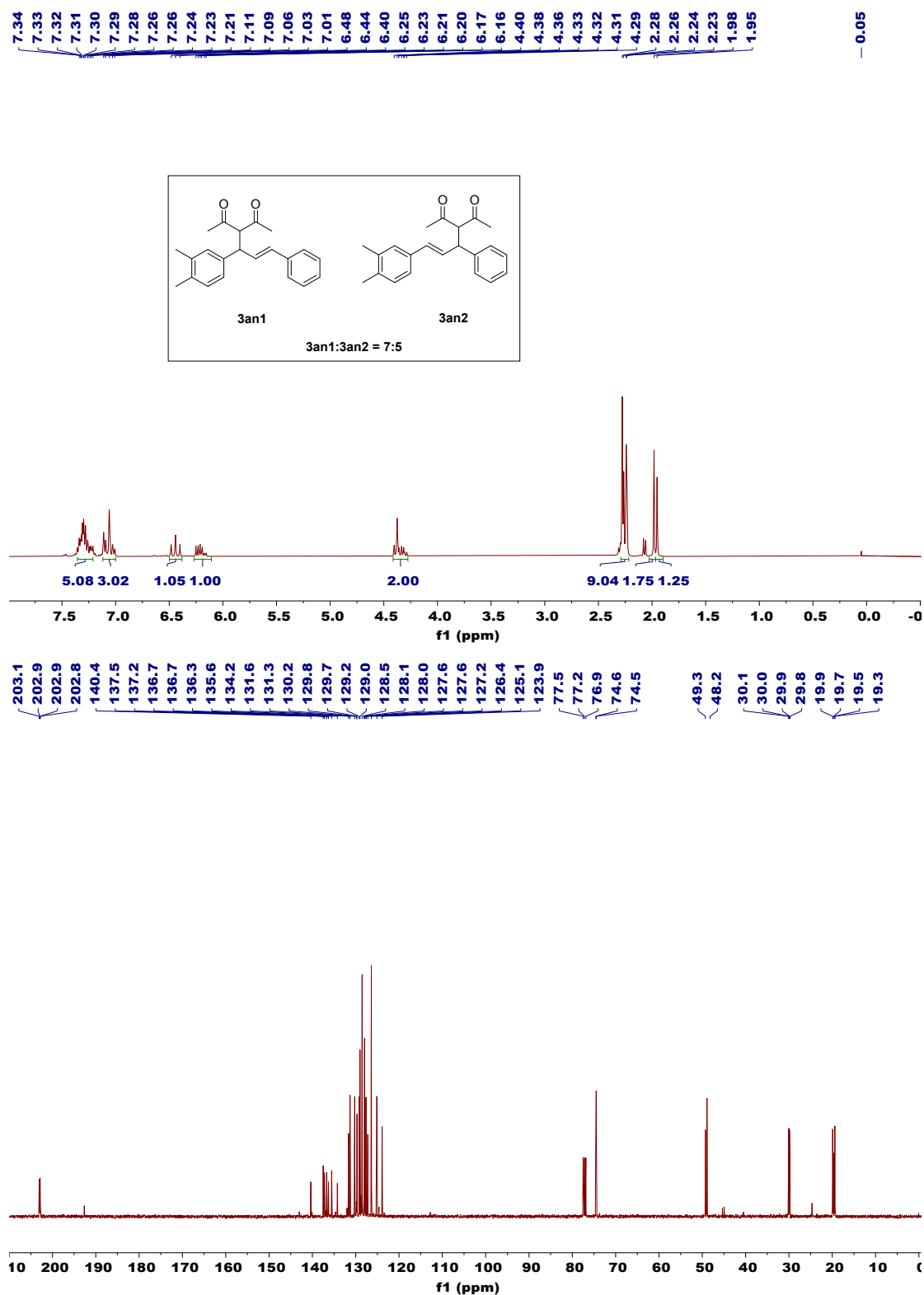


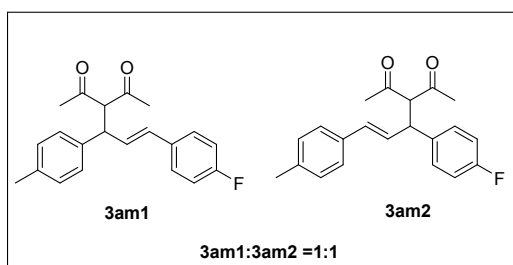
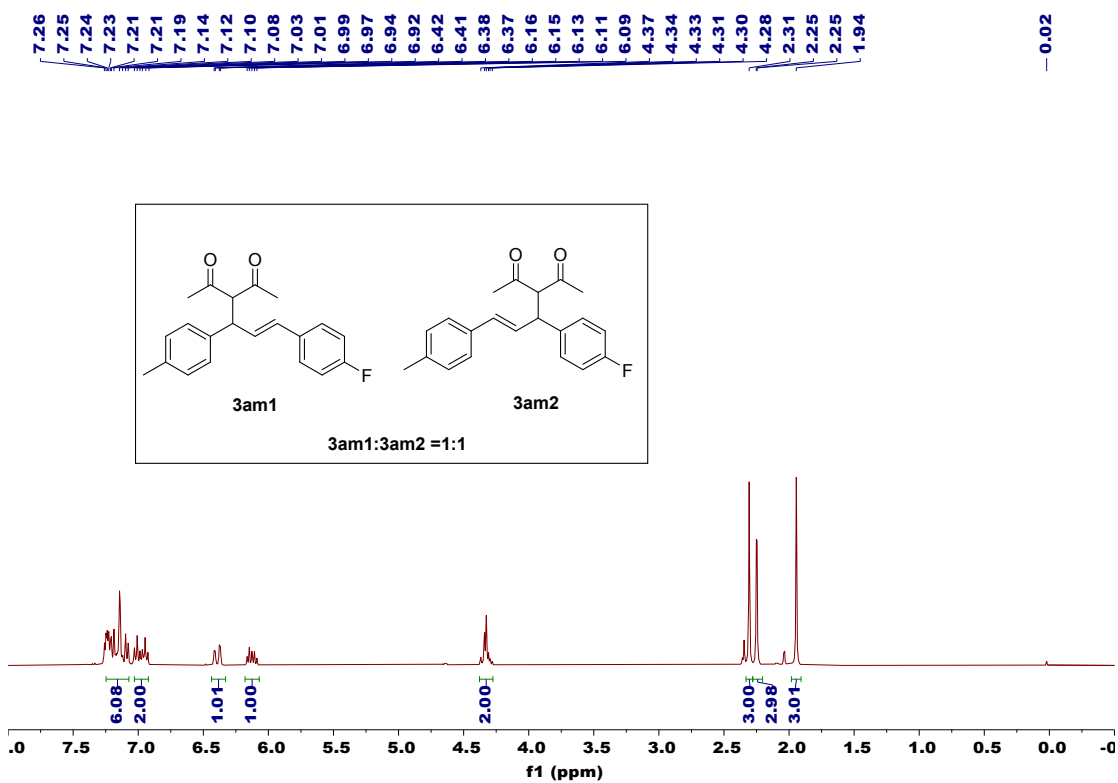
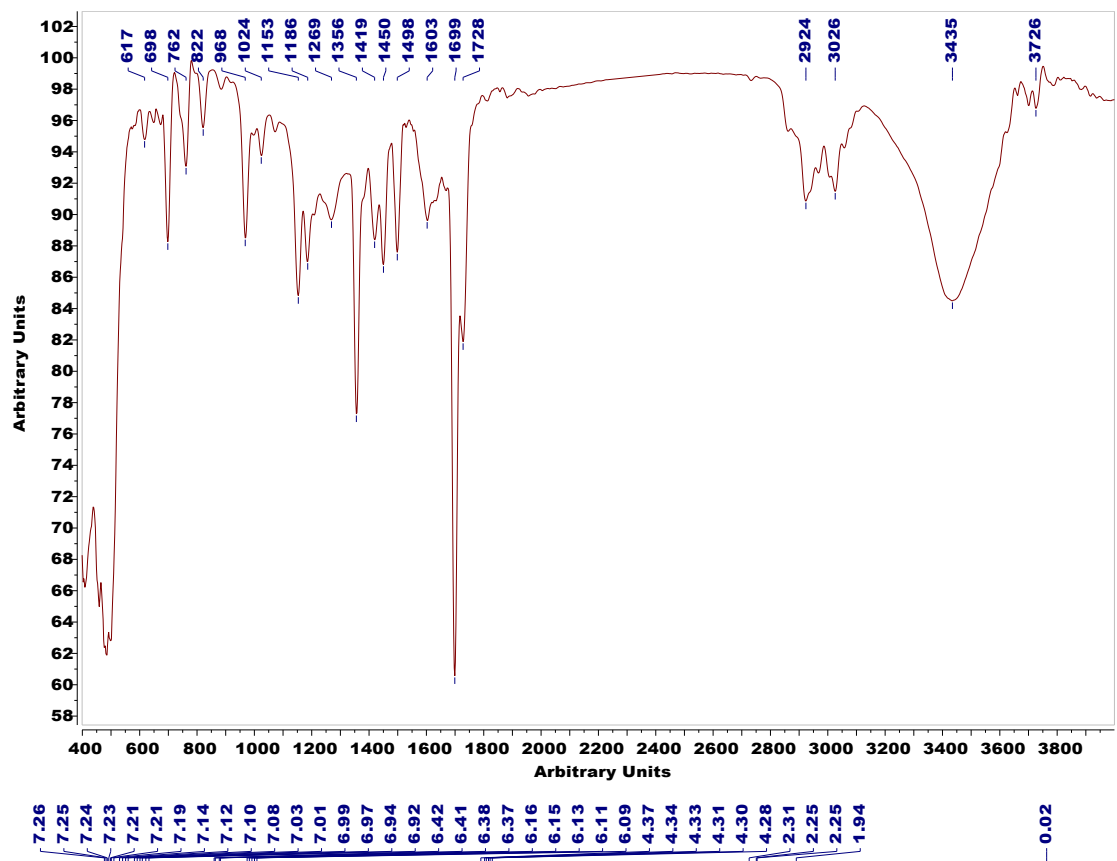


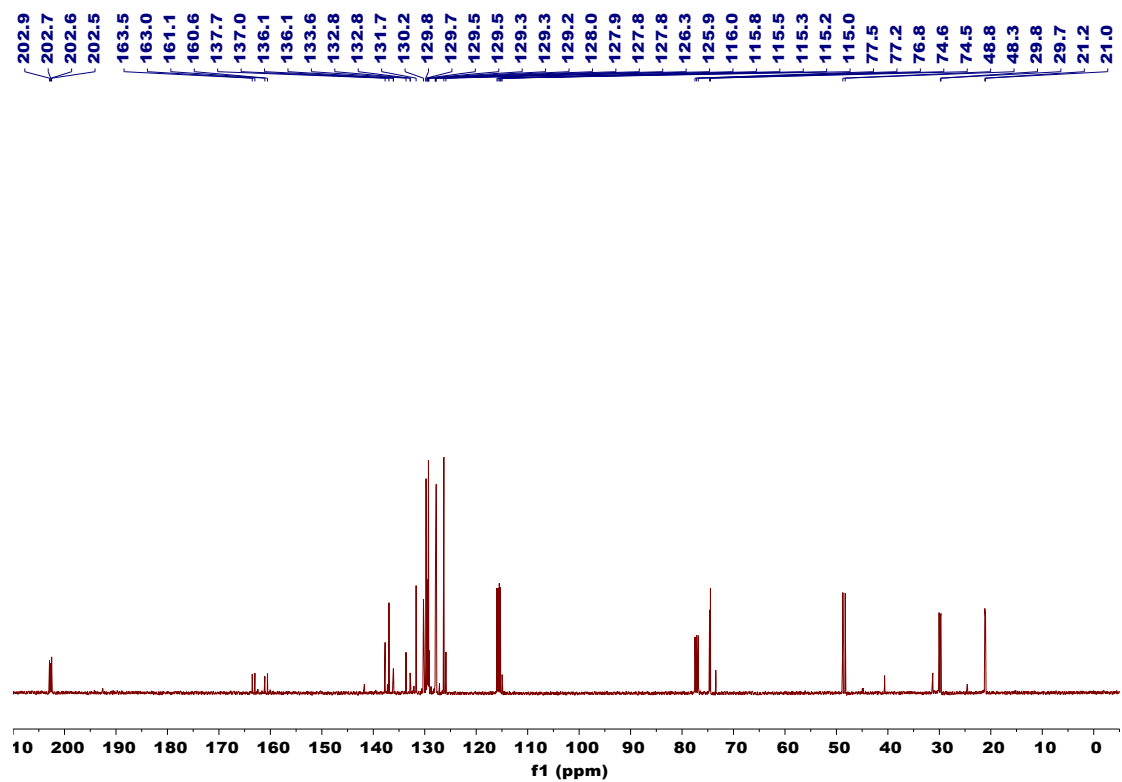












~114.2
~115.0

