

Supplementary Information

Coupling of Amide with Ketone *via* C-N / C-H Bonds Cleavage: A Mild Synthesis of 1,3-Diketone

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1. General Information

All reagents were purchased and used without further purification. ^1H spectra were recorded in CDCl_3 or DMSO-d_6 on 500 MHz NMR spectrometers and data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet), coupling constants (Hz) and integration. ^{13}C spectra were recorded in CDCl_3 or DMSO-d_6 on 126 MHz NMR spectrometers and resonances (δ) are given in ppm. High resolution mass spectra was recorded on a time of flight (TOF) mass spectrometer. ^1H NMR and ^{13}C NMR were recorded on Agilent Technologies (U. S. A.) NMR spectrometer (500 MHz for ^1H).

2. General procedure for the synthesis of starting materials

Starting materials (**1**, **2** and **4-9**) were synthesized according to the reported methods. ^[1, 2]

To a solution of amine (1.0 equiv) and Et_3N (1.5 equiv) in dichloromethane was added sulfonyl chloride (1.1 equiv) at 0 °C. The resulting solution was stirred overnight at 0 °C to room temperature. The reaction was diluted with ethyl acetate and washed with HCl (1 M), saturated Na_2CO_3 , and saturated NaCl. The organic layer was dried over anhydrous MgSO_4 , filtered, and concentrated under vacuum to afford sulfonamide and used without further purification.

To a solution of acyl chloride (purchased or synthesized from the acid^[3]) (1.1 equiv), sulfonamide (1.0 equiv) and Et_3N (1.5 equiv) in dichloromethane was added DMAP (0.1 equiv) at 0 °C. The resulting solution was stirred overnight at 0 °C to room temperature. The reaction was diluted with ethyl acetate and washed with HCl (1 M), saturated Na_2CO_3 , and saturated NaCl. The organic layer was dried over anhydrous MgSO_4 , filtered, and concentrated under vacuum. The solids were washed with ethyl acetate in hexane (25%) to afford pure product **1-9**.

Starting materials (**10**, **11**, **12**, **13**, **14** and **15**) were synthesized following the corresponding literatures. ^[1, 2, 4]

Other starting materials (**16**, **17** and **18**) were purchased and used without further purification.

3. General procedure for the synthesis of 1,3-diketones (**3**) from tertiary amides

To a 20 mL vial was charged with tertiary amide (0.3 mmol, 1.0 equiv), ketone (0.75 mmol, 2.5 equiv) and diethyl ether (3 mL), and then LiHMDS (0.75 mL, 2.5 equiv, 1 M in toluene) was added under argon atmosphere. The resulting solution was stirred for 4 h. The reaction was quenched with hydrogen chloride (5 mL, 1M in water) and diluted with ethyl acetate (50 mL). The organic layer was washed with brine (10 mL $\times$ 2), dried with anhydrous MgSO_4 , filtered and concentrated under vacuum. The crude product was purified by silica gel column chromatography with ethyl acetate/hexane or dichloromethane/hexane to afford the product.

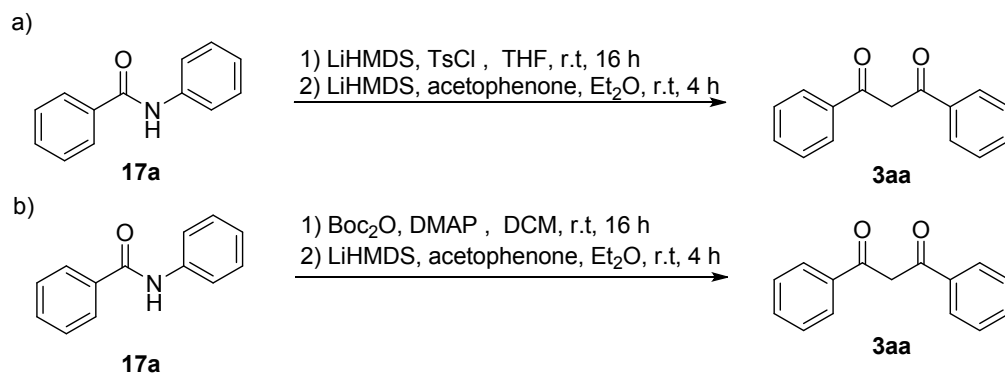
For substrates (**3do**, **3dp**, **3dq**) were synthesized with corresponding ketone (0.3 mmol, 1.0 equiv).

4. General procedure for the synthesis of 1,3-diketones (**3**) from secondary amides in one pot

Synthesis of 1,3-diphenylpropane-1,3-dione (**3aa**) as an example

Procedure A: To the solution of *N*-phenylbenzamide (**17a**) (98.6 mg, 0.5 mmol, 1.0 equiv) in THF (1 mL) was added LiHMDS (0.75 mL, 0.75 mmol, 1.5 equiv, 1 M in THF) under argon protected. After stirred for 1 h at room temperature, TsCl (114.4 mg, 0.6 mmol, 1.2 equiv) was added. The resulting solution was stirred for 16 h at room temperature. To the mixture was added acetophenone (150.1 mg, 1.25 mmol, 2.5 equiv), diethyl ether (5 mL) and LiHMDS (1.25 mL, 1.25 mmol, 2.5 equiv, 1 M in toluene). The resulting solution was stirred for 4 h under argon protected. The reaction was quenched with hydrogen chloride (5 mL, 1M in water) and diluted with ethyl acetate (50 mL). The organic layer was washed with brine (10 mL $\times$ 2), dried with anhydrous MgSO_4 , filtered and concentrated under vacuum. The crude product was purified by silica gel column chromatography with ethyl acetate in hexane (0 to 4%) to afford 1,3-diphenylpropane-1,3-dione (**3aa**) (78.5 mg, 0.35 mmol, 70% yield) as white solid. (**Scheme S1a**)

Procedure B: To the solution of *N*-phenylbenzamide (**17a**) (98.6 mg, 0.5 mmol, 1.0 equiv) in CH_2Cl_2 (1 mL) was added di-*tert*-butyl dicarbonate (163.7 mg, 0.75 mmol, 1.5 equiv) and DMAP (6.1 mg, 0.05 mmol, 0.1 equiv). The resulting solution was stirred for 16 h at room temperature. The dichloromethane was evaporated under vacuum and then acetophenone (150.1 mg, 1.25 mmol, 2.5 equiv), diethyl ether (5 mL) and LiHMDS (1.25 mL, 1.25 mmol, 2.5 equiv, 1 M in toluene) were added. The resulting solution was stirred for 4 h under argon protected. The reaction was quenched with hydrogen chloride (5 mL, 1M in water) and diluted with ethyl acetate (50 mL). The organic layer was washed with brine (10 mL $\times$ 2), dried with anhydrous MgSO_4 , filtered and concentrated under vacuum. The crude product was treated with hydrogen chloride (4 M in dioxane) for 1h. (Through deprotection to remove leaving group). The solids were filtered out, the filtrate was concentrated under vacuum and purified by silica gel column chromatography with ethyl acetate in hexane (0 to 4%) to afford 1,3-diphenylpropane-1,3-dione (**3aa**) (90.8 mg, 0.41 mmol, 81% yield) as white solid. (**Scheme S1b**)

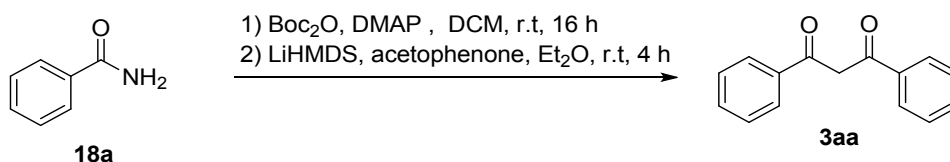


Scheme S1. Synthesis of **3aa** from **17a** in one pot

5. General procedure for the synthesis of 1,3-diketones (3) from primary amides in one pot

Synthesis of 1,3-diphenylpropane-1,3-dione (**3aa**) as an example

To the solution of benzamide (**18a**) (98.6 mg, 0.5 mmol, 1.0 equiv) in CH₂Cl₂ (1 mL) was added di-*tert*-butyl dicarbonate (272.8 mg, 1.25 mmol, 2.5 equiv) and DMAP (6.1 mg, 0.05 mmol, 0.1 equiv). The resulting solution was stirred for 16 h at room temperature. The dichloromethane was evaporated under vacuum and then acetophenone (150.1 mg, 1.25 mmol, 2.5 equiv), diethyl ether (5 mL) and LiHMDS (1.25 mL, 1.25 mmol, 2.5 equiv, 1 M in toluene) were added. The resulting solution was stirred for 4 h under argon protected. The reaction was quenched with hydrogen chloride (5 mL, 1M in water) and diluted with ethyl acetate (50 mL). The organic layer was washed with brine (10 mL × 2), dried with anhydrous MgSO₄, filtered and concentrated under vacuum. The crude product was purified by silica gel column chromatography with ethyl acetate in hexane (0 to 4%) to afford 1,3-diphenylpropane-1,3-dione (**3aa**) (95.3 mg, 0.43 mmol, 85% yield) as white solid. (Scheme S2).

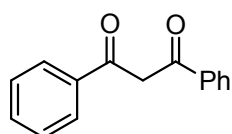


Scheme S2. Synthesis of **3aa** from **18a** in one pot

6. Procedure for the gram-scale reaction

To a 250 mL round-bottom flask was charged with 4-methylbenzamide (1.00 g, 7.4 mmol, 1.0 equiv), di-*tert*-butyl dicarbonate (4.04 g, 18.5 mmol, 2.5 equiv), DMAP (90 mg, 0.74 mmol, 0.1 equiv) and CH₂Cl₂ (15 mL). The resulting solution was stirred for 16 h at room temperature. The dichloromethane was evaporated under vacuum and then 4'-methylacetophenone (2.48 g, 18.5 mmol, 2.5 equiv), diethyl ether (70 mL) were added. After LiHMDS (18.5 mL, 18.5 mmol, 2.5 equiv, 1 M in toluene) was added through syringe under protecting with argon and cooling with ice bath, and then removing the ice bath. The resulting solution was stirred for 4 h with temperature rise naturally. The reaction was quenched with hydrogen chloride (20 mL, 1M in water) and diluted with ethyl acetate (100 mL). The organic layer was washed with brine (30 mL × 2), dried with anhydrous MgSO₄, filtered and concentrated under vacuum. The crude product was purified twice by silica gel column chromatography with ethyl acetate in hexane (0 to 5%) to afford 1,3-di-*p*-tolylpropane-1,3-dione (1.68 g, 6.7 mmol, 90% yield) as white solid.

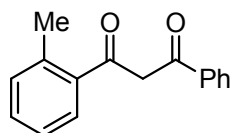
7. Experiment Data



1,3-Diphenylpropane-1,3-dione ^[5] (**3aa**) [contain 97% enol form]

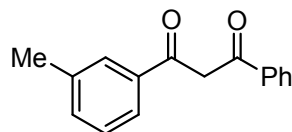
N-Phenyl-*N*-tosylbenzamide (105.4 mg, 0.30 mmol) afforded 1,3-diphenylpropane-1,3-dione (**3aa**) (56.1 mg, 0.25 mmol, 84% yield) as

white solid by silica gel column chromatography with ethyl acetate in hexane (0% to 5%); *N*-phenylbenzamide (98.6 mg, 0.50mmol) afforded **3aa** (78.5 mg, 0.35 mmol, 70% yield) under *Procedure A*; *N*-phenylbenzamide (98.6 mg, 0.50mmol) afforded **3aa** (95.3 mg, 0.41 mmol, 81% yield) under *Procedure B*; benzamide (60.6 mg, 0.50 mmol) afforded **3aa** (90.8 mg, 0.43 mmol, 85% yield) under the one-pot procedure; Mp: 77–78 °C; ¹H NMR (500 MHz, CDCl₃) δ 16.88 (s, 1H), 8.01 (d, *J* = 7.2 Hz, 4H), 7.57 (t, *J* = 7.2 Hz, 2H), 7.51 (t, *J* = 7.5 Hz, 4H), 6.88 (s, 1H); ¹³C NMR (126 MHz, CDCl₃) δ 185.8, 135.5, 132.5, 128.7, 127.2, 93.2.



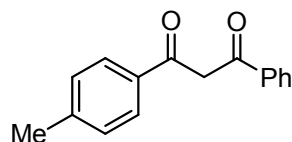
1-Phenyl-3-(*o*-tolyl)propane-1,3-dione ^[6] (**3ba**) [contain 97% enol form]

2-Methyl-*N*-phenyl-*N*-tosylbenzamide (109.6 mg, 0.30 mmol) afforded 1-phenyl-3-(*o*-tolyl)propane-1,3-dione (**3ba**) (21.4 mg, 0.09 mmol, 31% yield) as yellow oil by silica gel column chromatography with ethyl acetate in hexane (0% to 5%); ¹H NMR (500 MHz, CDCl₃) δ 16.66 (s, 1H), 7.97 (d, *J* = 7.7 Hz, 2H), 7.62–7.53 (m, 2H), 7.49 (t, *J* = 7.6 Hz, 2H), 7.39 (t, *J* = 7.5 Hz, 1H), 7.33–7.25 (m, 2H), 6.55 (s, 1H), 2.58 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 190.5, 184.9, 137.1, 136.7, 135.3, 132.4, 131.5, 130.7, 128.7, 128.3, 127.1, 125.8, 97.3, 20.7.



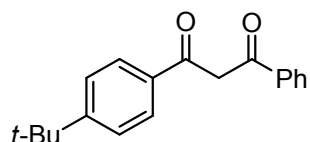
1-Phenyl-3-(*m*-tolyl)propane-1,3-dione ^[6] (**3ca**) [contain 97% enol form]

3-Methyl-*N*-phenyl-*N*-tosylbenzamide (109.6 mg, 0.30 mmol) afforded 1-phenyl-3-(*m*-tolyl)propane-1,3-dione (**3ca**) (60.8 mg, 0.26 mmol, 85% yield) as light yellow oil by silica gel column chromatography with ethyl acetate in hexane (0% to 5%); ¹H NMR (500 MHz, CDCl₃) δ 16.91 (s, 1H), 8.01 (d, *J* = 8.0 Hz, 2H), 7.84–7.78 (m, 2H), 7.59–7.54 (m, 1H), 7.50 (t, *J* = 7.6 Hz, 7H), 7.41–7.35 (m, 2H), 6.86 (s, 1H), 2.46 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 186.0, 185.6, 138.5, 135.6, 135.5, 133.3, 132.4, 128.7, 128.6, 127.7, 127.2, 124.4, 93.2, 21.5.



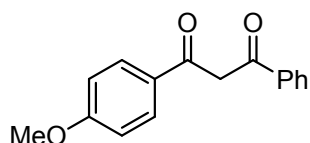
1-Phenyl-3-(*p*-tolyl)propane-1,3-dione ^[5] (**3da** and **3ad**) [contain 97% enol form]

4-Methyl-*N*-phenyl-*N*-tosylbenzamide (109.6 mg, 0.30 mmol) afforded 1-phenyl-3-(*p*-tolyl)propane-1,3-dione (**3da**) (65.8 mg, 0.28 mmol, 92% yield) as white solid by silica gel column chromatography with ethyl acetate in hexane (0% to 5%); *N*-phenylbenzamide (98.6 mg, 0.50mmol) afforded **3ad** (79.8 mg, 0.34 mmol, 67% yield) under *Procedure A*; *N*-phenylbenzamide (98.6 mg, 0.50mmol) afforded **3ad** (97.7 mg, 0.41 mmol, 82% yield) under *Procedure B*; benzamide (60.6 mg, 0.50 mmol) afforded **3ad** (107.2 mg, 0.45 mmol, 90% yield) under the one-pot procedure; Mp: 86–89 °C; ¹H NMR (500 MHz, CDCl₃) δ 16.94 (s, 1H), 8.00 (d, *J* = 8.0 Hz, 2H), 7.91 (d, *J* = 8.2 Hz, 2H), 7.56 (t, *J* = 7.3 Hz, 1H), 7.50 (t, *J* = 7.6 Hz, 2H), 7.30 (d, *J* = 8.3 Hz, 2H), 6.85 (s, 1H), 2.44 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 186.1, 185.2, 143.3, 135.6, 132.8, 132.3, 129.4, 128.7, 127.3, 127.1, 92.8, 21.7.



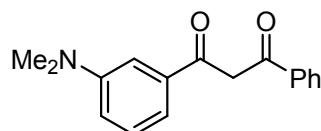
1-(4-(*tert*-Butyl)phenyl)-3-phenylpropane-1,3-dione ^[7] (**3ea**)
[contain 95% enol form]

4-(*tert*-Butyl)-*N*-phenyl-*N*-tosylbenzamide (122.3 mg, 0.30 mmol) afforded 1-(4-(*tert*-butyl)phenyl)-3-phenylpropane-1,3-dione (**3ea**) (74.9 mg, 0.27 mmol, 89% yield) as light yellow oil by silica gel column chromatography with ethyl acetate in hexane (0% to 5%) ¹H NMR (500 MHz, CDCl₃) δ 16.95 (s, 1H), 8.01 (d, *J* = 7.2 Hz, 2H), 7.96 (d, *J* = 8.5 Hz, 2H), 7.59–7.48 (m, 5H), 6.87 (s, 1H), 1.38 (s, 9H); ¹³C NMR (126 MHz, CDCl₃) δ 185.9, 185.3, 156.3, 135.6, 132.8, 132.3, 128.7, 127.13, 127.1, 125.7, 92.9, 35.1, 31.1.



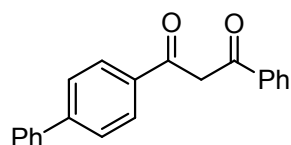
1-(4-Methoxyphenyl)-3-phenylpropane-1,3-dione ^[5] (**3fa**)
[contain 94% enol form]

4-Methoxy-*N*-phenyl-*N*-tosylbenzamide (114.4 mg, 0.30 mmol) afforded 1-(4-methoxyphenyl)-3-phenylpropane-1,3-dione (**3fa**) (61.8 mg, 0.24 mmol, 81% yield) as white solid by silica gel column chromatography with ethyl acetate in hexane (0% to 10%); Mp: 127–128 °C; ¹H NMR (500 MHz, CDCl₃) δ 17.01 (s, 1H), 8.04–7.93 (m, 4H), 7.58–7.44 (m, 3H), 6.99 (d, *J* = 8.8 Hz, 2H), 6.80 (s, 1H), 3.89 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 186.2, 184.0, 163.2, 135.5, 132.2, 129.3, 128.7, 128.2, 127.0, 114.0, 92.4, 55.5.



1-(3-(Dimethylamino)phenyl)-3-phenylpropane-1,3-dione (**3ga**) [contain 95% enol form]

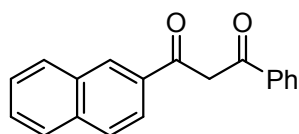
3-(Dimethylamino)-*N*-phenyl-*N*-tosylbenzamide (118.4 mg, 0.30 mmol) afforded 1-(3-(dimethylamino)phenyl)-3-phenylpropane-1,3-dione (**3ga**) (54.5 mg, 0.20 mmol, 68% yield) as yellow solid by silica gel column chromatography with ethyl acetate in hexane (0% to 10%); Mp: 72–73 °C; ¹H NMR (500 MHz, CDCl₃) δ 16.96 (s, 1H), 8.04–7.97 (m, 2H), 7.56 (d, *J* = 7.5 Hz, 1H), 7.50 (t, *J* = 7.4 Hz, 2H), 7.39–7.29 (m, 3H), 6.96–6.90 (m, 1H), 6.86 (s, 1H), 3.04 (s, 6H); ¹³C NMR (126 MHz, CDCl₃) δ 187.2, 185.0, 150.7, 136.4, 135.6, 132.3, 129.2, 128.7, 127.1, 116.4, 115.3, 110.6, 93.4, 40.6; HRMS (ESI-TOF) *m/z*: [M + H]⁺ Calcd for C₁₇H₁₈NO₂ 268.1338; Found 268.1338.



1-([1,1'-Biphenyl]-4-yl)-3-phenylpropane-1,3-dione ^[8] (**3ha**)
[contain 96% enol form]

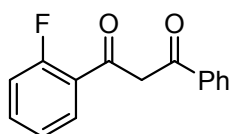
4-Methyl-*N*-phenyl-*N*-tosylbenzamide (128.3 mg, 0.30 mmol) afforded 1-([1,1'-biphenyl]-4-yl)-3-phenylpropane-1,3-dione (**3ha**) (65.8 mg, 0.22 mmol, 73% yield) as white solid by silica gel column chromatography with ethyl acetate in hexane (0% to 10%); Mp: 109–111 °C; ¹H NMR (500 MHz, CDCl₃) δ 16.97 (s, 1H), 8.09 (d, *J* = 8.4 Hz, 2H), 8.03 (d, *J* = 7.1 Hz, 2H), 7.73 (d, *J* = 8.4 Hz, 2H), 7.67 (d, *J* = 7.2 Hz, 2H), 7.58 (t, *J* = 7.3 Hz, 1H), 7.55–7.46 (m, 4H), 7.43 (t, *J* = 7.4 Hz, 1H), 6.92 (s, 1H); ¹³C NMR (126 MHz, CDCl₃) δ 185.7, 185.3, 145.2, 139.9, 135.6, 134.2, 132.5, 129.0,

128.7, 128.2, 127.7, 127.3, 127.24, 127.19, 93.1.



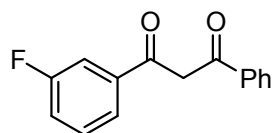
1-(naphthalen-2-yl)-3-phenylpropane-1,3-dione ^[5] (**3ia**) [contain 96% enol form]

N-Phenyl-*N*-tosyl-2-naphthamide (120.5 mg, 0.30 mmol) afforded 1-(naphthalen-2-yl)-3-phenylpropane-1,3-dione (**3ia**) (70.8 mg, 0.26 mmol, 86% yield) as light yellow solid by silica gel column chromatography with ethyl acetate in hexane (0% to 10%); Mp: 93–94 °C; ¹H NMR (500 MHz, CDCl₃) δ 16.99 (s, 1H), 8.55 (s, 1H), 8.10–8.02 (m, 3H), 7.99 (d, *J* = 7.9 Hz, 1H), 7.94 (d, *J* = 8.6 Hz, 1H), 7.90 (d, *J* = 7.9 Hz, 1H), 7.64–7.48 (m, 5H), 7.02 (s, 1H); ¹³C NMR (126 MHz, CDCl₃) δ 185.8, 185.5, 135.6, 135.3, 132.7, 132.5, 129.4, 128.7, 128.5, 128.4, 128.2, 127.8, 127.2, 126.8, 123.3, 93.5.



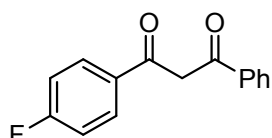
1-(2-Fluorophenyl)-3-phenylpropane-1,3-dione ^[6] (**3ja**) [contain 96% enol form]

2-Fluoro-*N*-phenyl-*N*-tosylbenzamide (110.8 mg, 0.30 mmol) afforded 1-(2-fluorophenyl)-3-phenylpropane-1,3-dione (**3ja**) (68.3 mg, 0.28 mmol, 94% yield) as white solid by silica gel column chromatography with ethyl acetate in hexane (0% to 5%); Mp: 62–63 °C; ¹H NMR (500 MHz, CDCl₃) δ 16.79 (s, 1H), 8.07–7.96 (m, 3H), 7.57 (t, *J* = 7.3 Hz, 1H), 7.54–7.46 (m, 3H), 7.32–7.25 (m, 1H), 7.21–7.14 (m, 1H), 6.99 (s, 1H); ¹³C NMR (126 MHz, CDCl₃) δ 186.5, 181.3 (d, *J* = 3.7 Hz), 161.2 (d, *J* = 255.8 Hz), 135.3, 133.7 (d, *J* = 9.2 Hz), 132.6, 130.2 (d, *J* = 1.9 Hz), 128.7, 127.4, 124.6 (d, *J* = 3.5 Hz), 123.8 (d, *J* = 10.4 Hz), 116.6 (d, *J* = 23.9 Hz), 97.9 (d, *J* = 13.4 Hz).



1-(3-Fluorophenyl)-3-phenylpropane-1,3-dione ^[6] (**3ka**) [contain 97% enol form]

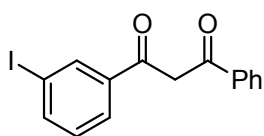
3-Fluoro-*N*-phenyl-*N*-tosylbenzamide (110.8 mg, 0.30 mmol) afforded 1-(3-fluorophenyl)-3-phenylpropane-1,3-dione (**3ka**) (66.8 mg, 0.28 mmol, 92% yield) as white solid by silica gel column chromatography with ethyl acetate in hexane (0% to 5%); Mp: 82–83 °C; ¹H NMR (500 MHz, CDCl₃) δ 16.77 (s, 1H), 8.04–7.96 (m, 2H), 7.78 (d, *J* = 7.8 Hz, 1H), 7.72–7.65 (m, 1H), 7.58 (t, *J* = 7.3 Hz, 1H), 7.54–7.43 (m, 3H), 7.30–7.22 (m, 1H), 6.83 (s, 1H); ¹³C NMR (126 MHz, CDCl₃) δ 186.1, 184.1, 162.9 (d, *J* = 248.2 Hz), 137.8 (d, *J* = 7.0 Hz), 135.2, 132.7, 130.3 (d, *J* = 7.9 Hz), 128.8, 127.2, 122.8 (d, *J* = 2.9 Hz), 119.3 (d, *J* = 21.4 Hz), 114.1 (d, *J* = 22.7 Hz), 93.3.



1-(4-Fluorophenyl)-3-phenylpropane-1,3-dione ^[5] (**3la** and **3al**) [contain 97% enol form]

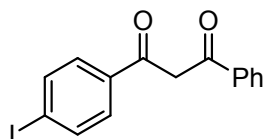
4-Fluoro-*N*-phenyl-*N*-tosylbenzamide (110.8 mg, 0.30 mmol) afforded 1-(4-fluorophenyl)-3-phenylpropane-1,3-dione (**3la**) (68.3 mg, 0.28 mmol, 94% yield) as white solid by silica gel column chromatography with ethyl acetate in

hexane (0% to 5%); *N*-phenylbenzamide (98.6 mg, 0.50 mmol) afforded **3al** (72.7 mg, 0.30 mmol, 60% yield) under *Procedure A*; *N*-phenylbenzamide (98.6 mg, 0.50 mmol) afforded **3al** (110.2 mg, 0.46 mmol, 91% yield) under *Procedure B*; benzamide (60.6 mg, 0.50 mmol) afforded **3al** (106.6 mg, 0.44 mmol, 88% yield) under the one-pot procedure; Mp: 78–79 °C; ¹H NMR (500 MHz, CDCl₃) δ 16.87 (s, 1H), 8.09–7.92 (m, 4H), 7.57 (t, *J* = 7.3 Hz, 1H), 7.50 (t, *J* = 7.5 Hz, 2H), 7.18 (t, *J* = 8.6 Hz, 2H), 6.81 (s, 1H); ¹³C NMR (126 MHz, CDCl₃) δ 185.12, 185.11, 165.4 (d, *J* = 254.5 Hz), 135.2, 132.5, 131.9 (d, *J* = 3.0 Hz), 129.6 (d, *J* = 9.1 Hz), 128.7, 127.1, 115.8 (d, *J* = 22.7 Hz), 92.8.



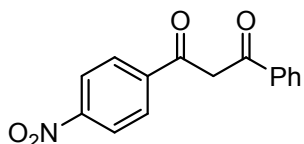
1-(3-Iodophenyl)-3-phenylpropane-1,3-dione (3ma) [contain 97% enol form]

3-Iodo-*N*-phenyl-*N*-tosylbenzamide (143.2 mg, 0.30 mmol) afforded 1-(3-iodophenyl)-3-phenylpropane-1,3-dione (**3ma**) (98.8 mg, 0.28 mmol, 94% yield) as pink solid by silica gel column chromatography with ethyl acetate in hexane (0% to 5%); Mp: 53–54 °C; ¹H NMR (500 MHz, CDCl₃) δ 16.75 (s, 1H), 8.31 (s, 1H), 8.00 (d, *J* = 7.6 Hz, 2H), 7.94 (d, *J* = 7.7 Hz, 1H), 7.88 (d, *J* = 7.8 Hz, 1H), 7.58 (t, *J* = 7.3 Hz, 1H), 7.51 (t, *J* = 7.5 Hz, 2H), 7.23 (t, *J* = 7.8 Hz, 1H), 6.80 (s, 1H); ¹³C NMR (126 MHz, CDCl₃) δ 186.0, 184.0, 141.1, 137.5, 136.0, 135.2, 132.8, 130.3, 128.8, 127.3, 126.3, 94.5, 93.2; HRMS (ESI-TOF) *m/z*: [*M* + Na]⁺ Calcd for C₁₅H₁₁IO₂Na 372.9701; Found 372.9700.



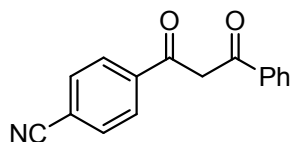
1-(4-Iodophenyl)-3-phenylpropane-1,3-dione ^[9] (**3na**) [contain 97% enol form]

4-Iodo-*N*-phenyl-*N*-tosylbenzamide (143.2 mg, 0.30 mmol) afforded 1-(4-iodophenyl)-3-phenylpropane-1,3-dione (**3na**) (94.6 mg, 0.27 mmol, 90%) as white solid by silica gel column chromatography with ethyl acetate in hexane (0% to 5%); Mp: 109–110 °C; ¹H NMR (500 MHz, CDCl₃) δ 16.81 (s, 1H), 8.03–7.94 (m, 2H), 7.84 (d, *J* = 8.6 Hz, 2H), 7.70 (d, *J* = 8.6 Hz, 2H), 7.57 (t, *J* = 7.3 Hz, 1H), 7.50 (t, *J* = 7.6 Hz, 2H), 6.81 (s, 1H); ¹³C NMR (126 MHz, CDCl₃) δ 186.0, 184.6, 138.0, 135.3, 134.9, 132.7, 128.7, 128.6, 127.2, 99.9, 93.0.



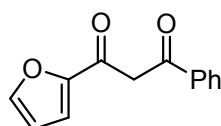
1-(4-Nitrophenyl)-3-phenylpropane-1,3-dione ^[10] (**3oa**) [contain 97% enol form]

4-Nitro-*N*-phenyl-*N*-tosylbenzamide (118.9 mg, 0.30 mmol) afforded 1-(4-nitrophenyl)-3-phenylpropane-1,3-dione (**3oa**) (74.3 mg, 0.28 mmol, 92% yield) as yellow solid by silica gel column chromatography with ethyl acetate in hexane (0% to 10%); Mp: 159–160 °C; ¹H NMR (500 MHz, CDCl₃) δ 16.69 (s, 1H), 8.34 (d, *J* = 8.8 Hz, 2H), 8.14 (d, *J* = 8.8 Hz, 2H), 8.02 (d, *J* = 7.1 Hz, 2H), 7.66–7.57 (m, 1H), 7.53 (t, *J* = 7.5 Hz, 2H), 6.91 (s, 1H); ¹³C NMR (126 MHz, CDCl₃) δ 187.8, 181.6, 149.8, 140.9, 135.1, 133.2, 128.9, 128.0, 127.4, 123.9, 94.2.



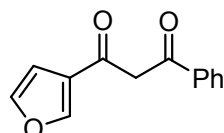
4-(3-Oxo-3-phenylpropanoyl)benzonitrile ^[11] (**3pa**) [contain 98% enol form]

4-Cyano-*N*-phenyl-*N*-tosylbenzamide (112.9 mg, 0.30 mmol) afforded 4-(3-oxo-3-phenylpropanoyl)benzonitrile (**3pa**) (65.8 mg, 0.26 mmol, 88% yield) as white solid by silica gel column chromatography with ethyl acetate in hexane (0% to 10%); Mp: 142–143 °C; ¹H NMR (500 MHz, CDCl₃) δ 16.71 (s, 1H), 8.07 (d, *J* = 8.3 Hz, 2H), 8.00 (d, *J* = 7.2 Hz, 2H), 7.78 (d, *J* = 8.2 Hz, 2H), 7.60 (t, *J* = 7.4 Hz, 1H), 7.52 (t, *J* = 7.6 Hz, 2H), 6.87 (s, 1H); ¹³C NMR (126 MHz, CDCl₃) δ 187.5, 182.2, 139.3, 135.1, 133.1, 132.5, 128.8, 127.5, 127.4, 118.1, 115.5, 93.9.



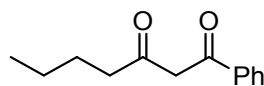
1-(Furan-2-yl)-3-phenylpropane-1,3-dione ^[5] (**3qa**) [contain 95% enol form]

N-Phenyl-*N*-tosylfuran-2-carboxamide (102.4 mg, 0.30 mmol) afforded 1-(furan-2-yl)-3-phenylpropane-1,3-dione (**3qa**) (61.0 mg, 0.29 mmol, 95% yield) as red solid by silica gel column chromatography with ethyl acetate in hexane (0% to 10%); Mp: 64–65 °C; ¹H NMR (500 MHz, CDCl₃) δ 16.23 (s, 1H), 7.98 (d, *J* = 7.3 Hz, 2H), 7.63 (s, 1H), 7.56 (t, *J* = 7.2 Hz, 1H), 7.49 (t, *J* = 7.4 Hz, 2H), 7.30–7.22 (m, 1H), 6.78 (s, 1H), 6.60 (d, *J* = 1.8 Hz, 1H); ¹³C NMR (126 MHz, CDCl₃) δ 182.4, 177.7, 151.0, 146.1, 134.6, 132.4, 128.7, 127.0, 115.9, 112.7, 92.7.



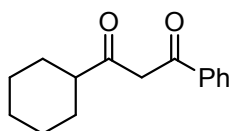
1-(Furan-3-yl)-3-phenylpropane-1,3-dione (**3ra**) [contain 94% enol form]

N-Phenyl-*N*-tosylfuran-3-carboxamide (102.4 mg, 0.30 mmol) afforded 1-(furan-3-yl)-3-phenylpropane-1,3-dione (**3ra**) (54.6 mg, 0.26 mmol, 85% yield) as white solid by silica gel column chromatography with ethyl acetate in hexane (0% to 10%); Mp: 85–87 °C; ¹H NMR (500 MHz, CDCl₃) δ 16.42 (s, 1H), 8.12 (s, 1H), 7.95 (d, *J* = 7.2 Hz, 2H), 7.56 (t, *J* = 7.3 Hz, 1H), 7.53–7.45 (m, 3H), 6.81 (d, *J* = 1.0 Hz, 1H), 6.51 (s, 1H); ¹³C NMR (126 MHz, CDCl₃) δ 184.0, 181.7, 145.7, 144.3, 135.0, 132.4, 128.7, 127.0, 125.2, 108.2, 94.1; HRMS (ESI-TOF) *m/z*: [M + Na]⁺ Calcd for C₁₃H₁₀O₃Na 237.0528; Found 237.0529.



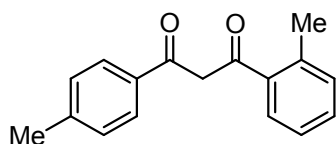
1-Phenylheptane-1,3-dione ^[5] (**3sa**) [contain 91% enol form]

N-Phenyl-*N*-tosylpentanamide (99.4 mg, 0.30 mmol) afforded 1-phenylheptane-1,3-dione (**3sa**) (40.5 mg, 0.20 mmol, 66% yield) as light yellow oil by silica gel column chromatography with ethyl acetate in hexane (0% to 5%); ¹H NMR (500 MHz, CDCl₃) δ 16.23 (s, 1H), 7.90 (d, *J* = 7.2 Hz, 2H), 7.53 (t, *J* = 7.3 Hz, 1H), 7.46 (t, *J* = 7.5 Hz, 2H), 6.19 (s, 1H), 2.44 (t, *J* = 7.6 Hz, 2H), 1.73–1.63 (m, 2H), 1.47–1.36 (m, 2H), 0.96 (t, *J* = 7.3 Hz, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 197.0, 183.5, 135.1, 132.2, 128.6, 127.0, 96.1, 39.0, 27.9, 22.4, 13.9.



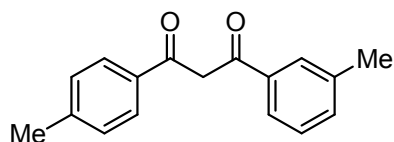
1-Cyclohexyl-3-phenylpropane-1,3-dione ^[5] (**3ta**) [contain 94% enol form]

N-Phenyl-*N*-tosylcyclohexanecarboxamide (107.3 mg, 0.30 mmol) afforded 1-cyclohexyl-3-phenylpropane-1,3-dione (**3ta**) (34.5 mg, 0.15 mmol, 50% yield) as light yellow oil by silica gel column chromatography with ethyl acetate in hexane (0% to 5%); ¹H NMR (500 MHz, CDCl₃) δ 16.33 (s, 1H), 7.90 (d, *J* = 7.2 Hz, 2H), 7.53 (t, *J* = 7.3 Hz, 1H), 7.46 (t, *J* = 7.5 Hz, 2H), 6.20 (s, 1H), 2.38–2.28 (m, 1H), 1.99–1.69 (m, 5H), 1.53–1.41 (m, 2H), 1.38–1.23 (m, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 199.9, 184.3, 135.3, 132.2, 128.6, 127.0, 94.5, 47.4, 29.6, 25.84, 25.82.



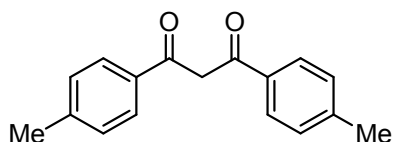
1-(*o*-Tolyl)-3-(*p*-tolyl)propane-1,3-dione (**3db**) [contain 96% enol form]

4-Methyl-*N*-phenyl-*N*-tosylbenzamide (109.6 mg, 0.30 mmol) afforded 1-(*o*-tolyl)-3-(*p*-tolyl)propane-1,3-dione (**3db**) (68.9 mg, 0.27 mmol, 91% yield) as yellow oil by silica gel column chromatography with ethyl acetate in hexane (0% to 5%); ¹H NMR (500 MHz, CDCl₃) δ 16.76 (s, 1H), 7.88 (d, *J* = 8.1 Hz, 2H), 7.59 (d, *J* = 8.0 Hz, 1H), 7.39 (t, *J* = 7.4 Hz, 1H), 7.34–7.25 (m, 4H), 6.53 (s, 1H), 2.58 (s, 3H), 2.45 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 189.9, 185.3, 143.3, 137.0, 136.7, 132.6, 131.4, 130.7, 129.5, 128.3, 127.2, 125.8, 97.0, 21.7, 20.8; HRMS (ESI-TOF) *m/z*: [M + Na]⁺ Calcd for C₁₇H₁₆O₂Na 275.1048; Found 275.1047.



1-(*m*-Tolyl)-3-(*p*-tolyl)propane-1,3-dione (**3dc**) [contain 96% enol form]

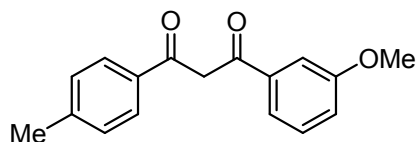
4-Methyl-*N*-phenyl-*N*-tosylbenzamide (109.6 mg, 0.30 mmol) afforded 1-(*m*-tolyl)-3-(*p*-tolyl)propane-1,3-dione (**3dc**) (70.4 mg, 0.28 mmol, 93% yield) as light yellow oil; ¹H NMR (500 MHz, CDCl₃) δ 16.97 (s, 1H), 7.89 (d, *J* = 8.1 Hz, 2H), 7.82–7.74 (m, 2H), 7.41–7.32 (m, 2H), 7.28 (d, *J* = 8.1 Hz, 2H), 6.81 (s, 1H), 2.42 (s, 3H), 2.41 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 186.1, 185.6, 143.4, 138.6, 135.7, 133.3, 133.0, 129.6, 128.7, 127.8, 127.4, 124.5, 93.0, 21.9, 21.6; HRMS (ESI-TOF) *m/z*: [M + Na]⁺ Calcd for C₁₇H₁₆O₂Na 275.1048; Found 275.1047.



1,3-Di-*p*-tolylpropane-1,3-dione ^[12] (**3dd**) [contain 95% enol form]

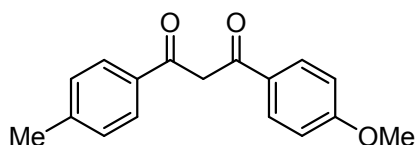
4-Methyl-*N*-phenyl-*N*-tosylbenzamide (109.6 mg, 0.30 mmol) afforded 1,3-di-*p*-tolylpropane-1,3-dione (**3dd**) (71.1 mg, 0.28 mmol, 94% yield) as white solid by silica gel column chromatography with ethyl acetate in hexane (0% to 5%); 4-methyl-*N*-phenylbenzamide (105.6 mg, 0.50 mmol) afforded **3dd** (116.1 mg, 0.46 mmol, 92% yield) under *Procedure B*; 4-methylbenzamide (67.6 mg, 0.50 mmol) afforded **3dd** (107.2 mg, 0.425 mmol, 85% yield) under the one-pot procedure; Mp: 125–127 °C; ¹H NMR (500 MHz, CDCl₃) δ 17.02 (s, 1H), 7.88 (d, *J* = 8.1 Hz, 4H), 7.28 (d, *J*

= 8.1 Hz, 4H), 6.81 (s, 1H), 2.42 (s, 6H); ^{13}C NMR (126 MHz, CDCl_3) δ 185.5, 143.2, 132.8, 129.4, 127.2, 92.5, 21.7.



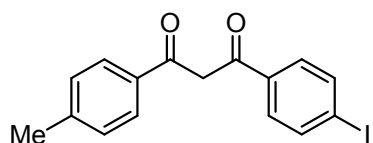
1-(3-Methoxyphenyl)-3-(*p*-tolyl)propane-1,3-dione (3de)
[contain 96% enol form]

4-Methyl-*N*-phenyl-*N*-tosylbenzamide (109.6 mg, 0.30 mmol) afforded 1-(3-methoxyphenyl)-3-(*p*-tolyl)propane-1,3-dione (**3de**) (62.8 mg, 0.23 mmol, 78% yield) as white solid by silica gel column chromatography with ethyl acetate in hexane (0% to 10%); Mp: 49–51 °C; ^1H NMR (500 MHz, CDCl_3) δ 16.92 (s, 1H), 7.88 (d, J = 8.1 Hz, 2H), 7.58–7.48 (m, 2H), 7.38 (t, J = 7.9 Hz, 1H), 7.28 (d, J = 7.9 Hz, 2H), 7.10–7.04 (m, 1H), 6.80 (s, 1H), 3.87 (s, 3H), 2.42 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 185.9, 185.4, 160.0, 143.5, 137.3, 132.9, 129.8, 129.6, 127.4, 119.7, 118.6, 112.1, 93.2, 55.6, 21.9; HRMS (ESI-TOF) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{17}\text{H}_{16}\text{O}_3\text{Na}$ 291.0997; Found 291.0996.



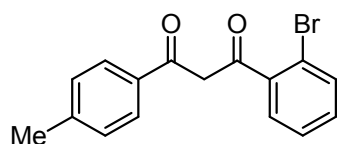
1-(4-Methoxyphenyl)-3-(*p*-tolyl)propane-1,3-dione (3df) [13]
[contain 93% enol form]

4-Methyl-*N*-phenyl-*N*-tosylbenzamide (109.6 mg, 0.30 mmol) afforded 1-(4-methoxyphenyl)-3-(*p*-tolyl)propane-1,3-dione (**3df**) (73.2 mg, 0.27 mmol, 91% yield) as white solid by silica gel column chromatography with ethyl acetate in hexane (0% to 10%); Mp: 99–101 °C; ^1H NMR (500 MHz, CDCl_3) δ 17.12 (s, 1H), 7.98 (d, J = 8.9 Hz, 2H), 7.89 (d, J = 8.1 Hz, 2H), 7.29 (d, J = 8.0 Hz, 2H), 6.98 (d, J = 8.8 Hz, 2H), 6.79 (s, 1H), 3.89 (s, 3H), 2.44 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 185.7, 184.3, 163.1, 143.0, 132.8, 129.4, 129.2, 128.2, 127.1, 113.9, 92.0, 55.5, 21.7.



1-(4-iodophenyl)-3-(*p*-tolyl)propane-1,3-dione (3dg) [9]
[contain 97% enol form]

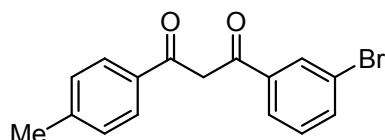
4-Methyl-*N*-phenyl-*N*-tosylbenzamide (109.6 mg, 0.30 mmol) afforded 1-(4-iodophenyl)-3-(*p*-tolyl)propane-1,3-dione (**3dg**) (87.4 mg, 0.24 mmol, 80% yield) as white solid by silica gel column chromatography with ethyl acetate in hexane (0% to 5%); Mp: 171–173 °C; ^1H NMR (500 MHz, CDCl_3) δ 16.91 (s, 1H), 7.89 (d, J = 8.1 Hz, 2H), 7.84 (d, J = 8.5 Hz, 2H), 7.70 (d, J = 8.5 Hz, 2H), 7.30 (d, J = 8.1 Hz, 2H), 6.80 (s, 1H), 2.44 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 186.31 (s), 184.02 (s), 143.58 (s), 137.9, 135.0, 132.6, 129.5, 128.5, 127.3, 99.7, 92.6, 21.7.



1-(2-Bromophenyl)-3-(*p*-tolyl)propane-1,3-dione (3dh)
[contain 98% enol form]

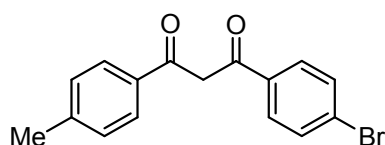
4-Methyl-*N*-phenyl-*N*-tosylbenzamide (109.6 mg, 0.30 mmol)

afforded 1-(2-bromophenyl)-3-(*p*-tolyl)propane-1,3-dione (**3dh**) (82.8 mg, 0.26 mmol, 87% yield) as white solid by silica gel column chromatography with ethyl acetate in hexane (0% to 5%); Mp: 80–81 °C; ¹H NMR (500 MHz, CDCl₃) δ 16.37 (s, 1H), 7.87 (d, *J* = 8.2 Hz, 2H), 7.68 (d, *J* = 8.0 Hz, 1H), 7.61 (d, *J* = 7.6 Hz, 1H), 7.46–7.38 (m, 1H), 7.36–7.24 (m, 3H), 6.65 (s, 1H), 2.44 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 188.0, 184.3, 143.6, 138.5, 133.9, 132.0, 131.6, 130.0, 129.5, 127.5, 127.3, 120.2, 97.9, 21.7; HRMS (ESI-TOF) *m/z*: [M + Na]⁺ Calcd for C₁₆H₁₃BrO₂Na 338.9997; Found 338.9998.



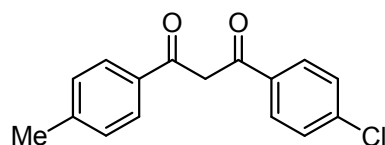
1-(3-Bromophenyl)-3-(*p*-tolyl)propane-1,3-dione (3di)
[contain 97% enol form]

4-Methyl-*N*-phenyl-*N*-tosylbenzamide (109.6 mg, 0.30 mmol) afforded 1-(3-bromophenyl)-3-(*p*-tolyl)propane-1,3-dione (**3di**) (70.4 mg, 0.22 mmol, 74% yield) as white solid by silica gel column chromatography with ethyl acetate in hexane (0% to 5%); Mp: 103–104 °C; ¹H NMR (500 MHz, CDCl₃) δ 16.86 (s, 1H), 8.11 (s, 1H), 7.96–7.85 (m, 3H), 7.67 (d, *J* = 7.9 Hz, 1H), 7.41–7.36 (m, 1H), 7.31 (d, *J* = 8.0 Hz, 2H), 6.79 (s, 1H), 2.45 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 186.4, 183.4, 143.7, 137.6, 135.1, 132.4, 130.2, 130.1, 129.5, 127.3, 125.6, 122.9, 93.0, 21.8; HRMS (ESI-TOF) *m/z*: [M + Na]⁺ Calcd for C₁₆H₁₃BrO₂Na 338.9997; Found 338.9997.



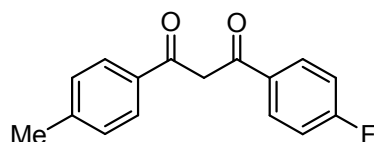
1-(4-Bromophenyl)-3-(*p*-tolyl)propane-1,3-dione [8] (3dj)
[contain 96% enol form]

4-Methyl-*N*-phenyl-*N*-tosylbenzamide (109.6 mg, 0.30 mmol) afforded 1-(4-bromophenyl)-3-(*p*-tolyl)propane-1,3-dione (**3gj**) (79.9 mg, 0.25 mmol, 84% yield) as white solid by silica gel column chromatography with ethyl acetate in hexane (0% to 5%); Mp: 163–164 °C; ¹H NMR (500 MHz, CDCl₃) δ 16.92 (s, 1H), 7.89 (d, *J* = 8.2 Hz, 2H), 7.85 (d, *J* = 8.6 Hz, 2H), 7.62 (d, *J* = 8.6 Hz, 2H), 7.30 (d, *J* = 8.2 Hz, 2H), 6.80 (s, 1H), 2.44 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 186.2, 184.0, 143.6, 134.4, 132.5, 131.9, 129.5, 128.6, 127.3, 127.1, 92.7, 21.7.



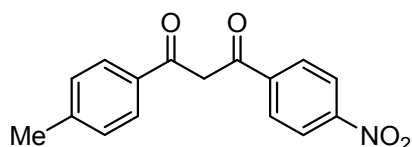
1-(4-Chlorophenyl)-3-(*p*-tolyl)propane-1,3-dione [13] (3dk and 3ud) [contain 96% enol form]

4-Methyl-*N*-phenyl-*N*-tosylbenzamide (109.6 mg, 0.30 mmol) afforded 1-(4-chlorophenyl)-3-(*p*-tolyl)propane-1,3-dione (**3dk**) (66.3 mg, 0.24 mmol, 81% yield) as white solid by silica gel column chromatography with ethyl acetate in hexane (0% to 5%); 4-chloro-*N*-phenylbenzamide (115.8 mg, 0.50 mmol) afforded **3ud** (117.3 mg, 0.43 mmol, 86% yield) under *Procedure B*; 4-chlorobenzamide (77.8 mg, 0.50 mmol) afforded **3ud** (102.3 mg, 0.375 mmol, 75% yield) under the one-pot procedure; Mp: 148–150 °C; ¹H NMR (500 MHz, CDCl₃) δ 16.93 (s, 1H), 7.93 (d, *J* = 8.6 Hz, 2H), 7.90 (d, *J* = 8.2 Hz, 2H), 7.46 (d, *J* = 8.6 Hz, 2H), 7.30 (d, *J* = 7.9 Hz, 2H), 6.80 (s, 1H), 2.44 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 186.1, 183.9, 143.5, 138.5, 134.0, 132.5, 129.5, 129.0, 128.5, 127.3, 92.7, 21.7.



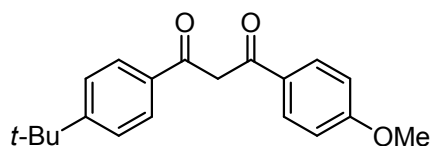
1-(4-Fluorophenyl)-3-(*p*-tolyl)propane-1,3-dione ^[13] (**3dl**)
[contain 95% enol form]

4-Methyl-*N*-phenyl-*N*-tosylbenzamide (109.6 mg, 0.30 mmol) afforded 1-(4-fluorophenyl)-3-(*p*-tolyl)propane-1,3-dione (**3dl**) (69.2 mg, 0.27 mmol, 90% yield) as white solid by silica gel column chromatography with ethyl acetate in hexane (0% to 5%); 4-methyl-*N*-phenylbenzamide (105.6 mg, 0.50 mmol) afforded **3dl** (120.5 mg, 0.47 mmol, 94% yield) under *Procedure B*; 4-methylbenzamide (67.6 mg, 0.50 mmol) afforded **3dl** (116.6 mg, 0.455 mmol, 91% yield) under the one-pot procedure; Mp: 96–97 °C; ¹H NMR (500 MHz, CDCl₃) δ 16.93 (s, 1H), 8.05–7.94 (m, 2H), 7.87 (d, *J* = 8.1 Hz, 2H), 7.28 (d, *J* = 7.9 Hz, 2H), 7.18–7.10 (m, 2H), 6.76 (s, 1H), 2.42 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 185.4, 184.5, 165.3 (d, *J* = 254.5 Hz), 143.4, 132.5, 131.94 (d, *J* = 3.0 Hz), 129.5 (d, *J* = 8.8 Hz), 129.4, 127.2, 115.8 (d, *J* = 21.4 Hz), 92.5, 21.7.



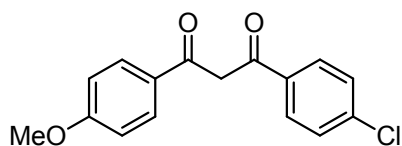
1-(4-Nitrophenyl)-3-(*p*-tolyl)propane-1,3-dione (**3dm**)
[contain 96% enol form]

4-Methyl-*N*-phenyl-*N*-tosylbenzamide (109.6 mg, 0.30 mmol) afforded 1-(4-nitrophenyl)-3-(*p*-tolyl)propane-1,3-dione (**3dm**) (69.7 mg, 0.25 mmol, 82% yield) as yellow solid by silica gel column chromatography with ethyl acetate in hexane (0% to 10%); Mp: 186–187 °C; ¹H NMR (500 MHz, DMSO-*d*₆) δ 16.97 (s, 1H), 8.42–8.27 (m, 4H), 8.09 (d, *J* = 7.7 Hz, 2H), 7.42 (s, 1H), 7.37 (d, *J* = 7.6 Hz, 2H), 2.39 (s, 3H); ¹³C NMR (126 MHz, DMSO-*d*₆) δ 188.2, 180.9, 150.1, 144.7, 140.4, 132.3, 123.0, 129.0, 128.3, 124.3, 95.0; HRMS (ESI-TOF) *m/z*: [M + Na]⁺ Calcd for C₁₆H₁₃NO₄Na 306.0742; Found 306.0743.



1-(4-(*tert*-Butyl)phenyl)-3-(4-methoxyphenyl)propane-1,3-dione ^[14] (**3ef**) [contain 93% enol form]

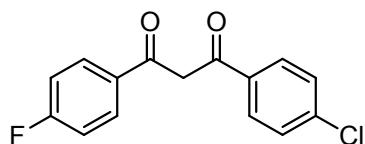
4-(*tert*-Butyl)-*N*-phenyl-*N*-tosylbenzamide (122.3 mg, 0.30 mmol) afforded 1-(4-(*tert*-butyl)phenyl)-3-(4-methoxyphenyl)propane-1,3-dione (**3ef**) (88.5 mg, 0.29 mmol, 95% yield) as white solid by silica gel column chromatography with ethyl acetate in hexane (0% to 10%); Mp: 76–77 °C; ¹H NMR (500 MHz, CDCl₃) δ 17.08 (s, 1H), 7.99 (d, *J* = 8.8 Hz, 2H), 7.93 (d, *J* = 8.5 Hz, 2H), 7.51 (d, *J* = 8.5 Hz, 2H), 6.99 (d, *J* = 8.8 Hz, 2H), 6.80 (s, 1H), 3.89 (s, 3H), 1.36 (d, *J* = 13.2 Hz, 9H); ¹³C NMR (126 MHz, CDCl₃) δ 185.8, 184.2, 163.1, 155.9, 132.7, 129.2, 128.3, 126.9, 125.6, 114.0, 92.1, 55.5, 35.1, 31.2.



1-(4-Chlorophenyl)-3-(4-methoxyphenyl)propane-1,3-dione ^[7] (**3fk**) [contain 96% enol form]

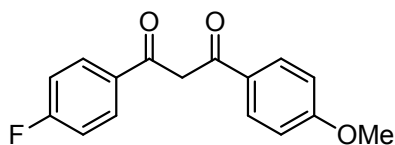
4-Methoxy-*N*-phenyl-*N*-tosylbenzamide (114.4 mg, 0.30 mmol) afforded 1-(4-chlorophenyl)-3-(4-methoxyphenyl)propane-1,3-dione (**3fk**) (68.4 mg, 0.24 mmol, 79% yield) as white solid by

silica gel column chromatography with dichloromethane in hexane (50%); Mp: 116–117 °C; ¹H NMR (500 MHz, CDCl₃) δ 16.96 (s, 1H), 7.98 (d, *J* = 8.7 Hz, 2H), 7.91 (d, *J* = 8.4 Hz, 2H), 7.46 (d, *J* = 8.4 Hz, 2H), 6.99 (d, *J* = 8.7 Hz, 2H), 6.75 (s, 1H), 3.90 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 186.2, 182.8, 163.4, 138.4, 134.0, 129.3, 128.9, 128.3, 127.9, 114.0, 92.2, 55.5.



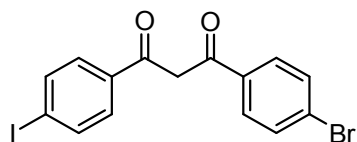
1-(4-Chlorophenyl)-3-(4-fluorophenyl)propane-1,3-dione (3lk) [contain 97% enol form]

4-Fluoro-*N*-phenyl-*N*-tosylbenzamide (110.8 mg, 0.30 mmol) afforded 1-(4-chlorophenyl)-3-(4-fluorophenyl)propane-1,3-dione (**3lk**) (69.7 mg, 0.25 mmol, 84% yield) as white solid by silica gel column chromatography with ethyl acetate in hexane (0% to 5%); Mp: 122–123 °C; ¹H NMR (500 MHz, CDCl₃) δ 16.84 (s, 1H), 8.07–7.97 (m, 2H), 7.92 (d, *J* = 8.5 Hz, 2H), 7.46 (d, *J* = 8.5 Hz, 2H), 7.23–7.12 (m, 2H), 6.76 (s, 1H); ¹³C NMR (126 MHz, CDCl₃) δ 185.1, 183.9, 166.5 (d, *J* = 254.5 Hz), 138.8, 133.7, 131.6 (d, *J* = 2.9 Hz), 129.7 (d, *J* = 9.2 Hz), 129.0, 128.5, 115.9 (d, *J* = 21.4 Hz), 92.7; HRMS (ESI-TOF) *m/z*: [M + Na]⁺ Calcd for C₁₅H₁₀ClFO₂Na 299.0251; Found 299.0250.



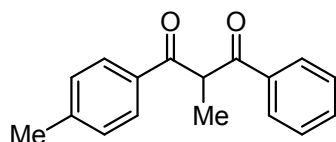
1-(4-Fluorophenyl)-3-(4-methoxyphenyl)propane-1,3-dione ^[17] (**3lf**) [contain 94% enol form]

4-Fluoro-*N*-phenyl-*N*-tosylbenzamide (110.8 mg, 0.30 mmol) afforded 1-(4-fluorophenyl)-3-(4-methoxyphenyl)propane-1,3-dione (**3lf**) (70.3 mg, 0.26 mmol, 86% yield) as white solid by silica gel column chromatography with ethyl acetate in hexane (0% to 10%); Mp: 143–144 °C; ¹H NMR (500 MHz, CDCl₃) δ 17.04 (s, 1H), 8.04–7.93 (m, 4H), 7.20–7.12 (m, 2H), 6.99 (d, *J* = 8.8 Hz, 2H), 6.74 (s, 1H), 3.89 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 185.6, 183.4, 165.2 (d, *J* = 253.3 Hz), 163.3, 131.9 (d, *J* = 2.9 Hz), 129.4 (d, *J* = 8.8 Hz), 129.3, 127.9, 115.8 (d, *J* = 21.4 Hz), 114.0, 92.0, 55.5; HRMS (ESI-TOF) *m/z*: [M + Na]⁺ Calcd for C₁₆H₁₃FO₃Na 295.0746; Found 295.0746.



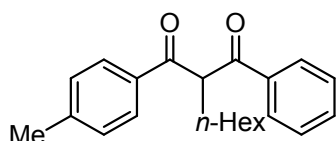
1-(4-Bromophenyl)-3-(4-iodophenyl)propane-1,3-dione (3nj) [contain 97% enol form]

4-Iodo-*N*-phenyl-*N*-tosylbenzamide (143.2 mg, 0.30 mmol) afforded 1-(4-bromophenyl)-3-(4-iodophenyl)propane-1,3-dione (**3nj**) (84.9 mg, 0.20 mmol, 66% yield) as white solid by silica gel column chromatography with ethyl acetate in hexane (0% to 5%); Mp: 218–219 °C; ¹H NMR (500 MHz, CDCl₃) δ 16.76 (s, 1H), 7.90–7.79 (m, 4H), 7.70 (d, *J* = 8.5 Hz, 2H), 7.64 (d, *J* = 8.5 Hz, 2H), 7.27 (s, 1H), 6.78 (s, 1H); ¹³C NMR (126 MHz, CDCl₃) δ 184.9, 184.8, 138.0, 134.7, 134.2, 132.0, 128.7, 128.6, 127.5, 100.1, 92.8; HRMS (ESI-TOF) *m/z*: [M + Na]⁺ Calcd for C₁₅H₁₀BrIO₂Na 450.8807; Found 450.8808.



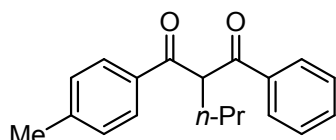
2-Methyl-1-phenyl-3-(*p*-tolyl)propane-1,3-dione ^[15] (**3do**)

4-Methyl-*N*-phenyl-*N*-tosylbenzamide (109.6 mg, 0.30 mmol) afforded 2-methyl-1-phenyl-3-(*p*-tolyl)propane-1,3-dione (**3do**) (57.5 mg, 0.23 mmol, 76% yield) as white solid by silica gel column chromatography with ethyl acetate in hexane (0% to 20%); Mp: 84–85 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.96 (d, *J* = 7.3 Hz, 2H), 7.88 (d, *J* = 8.1 Hz, 2H), 7.56 (t, *J* = 7.3 Hz, 1H), 7.49–7.40 (m, 2H), 7.26 (d, *J* = 8.0 Hz, 2H), 5.26 (q, *J* = 7.0 Hz, 1H), 2.40 (s, 3H), 1.60 (d, *J* = 7.0 Hz, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 197.2, 196.9, 144.5, 135.7, 133.4, 133.1, 129.6, 128.8, 128.7, 128.5, 50.9, 21.7, 14.4.



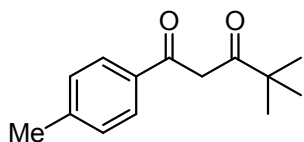
2-Hexyl-1-phenyl-3-(*p*-tolyl)propane-1,3-dione (**3dp**)

4-Methyl-*N*-phenyl-*N*-tosylbenzamide (109.6 mg, 0.30 mmol) afforded 2-hexyl-1-phenyl-3-(*p*-tolyl)propane-1,3-dione (**3dp**) (84.1 mg, 0.26 mmol, 87% yield) as colorless oil by silica gel column chromatography with ethyl acetate in hexane (5% to 10%); ¹H NMR (500 MHz, CDCl₃) δ 7.96 (d, *J* = 7.4 Hz, 2H), 7.88 (d, *J* = 8.1 Hz, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.49–7.39 (m, 2H), 7.25 (d, *J* = 8.1 Hz, 2H), 5.16 (t, *J* = 6.6 Hz, 1H), 2.40 (s, 3H), 2.11 (dt, *J* = 14.0, 6.8 Hz, 2H), 1.46–1.22 (m, 8H), 0.86 (t, *J* = 6.7 Hz, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 196.2, 195.8, 144.4, 136.2, 133.6, 133.3, 129.6, 128.8, 128.7, 128.5, 57.3, 31.5, 29.6, 29.3, 28.3, 22.6, 21.7, 14.1; HRMS (ESI-TOF) *m/z*: [M + Na]⁺ Calcd for C₂₂H₂₆O₂Na 345.1830; Found 345.1830.



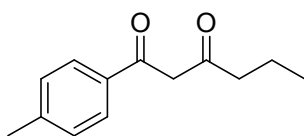
1-Phenyl-2-propyl-3-(*p*-tolyl)propane-1,3-dione (**3dq**)

4-Methyl-*N*-phenyl-*N*-tosylbenzamide (109.6 mg, 0.30 mmol) afforded 1-phenyl-2-propyl-3-(*p*-tolyl)propane-1,3-dione (**3dq**) (69.0 mg, 0.25 mmol, 82% yield) as light yellow oil by silica gel column chromatography with ethyl acetate in hexane (5% to 10%); 4-methyl-*N*-phenylbenzamide (105.6 mg, 0.50 mmol) with 1-phenylpentan-1-one (1.0 equiv) under *Procedure B* to afford **3dq** (47.7 mg, 0.17 mmol, 34% yield); 4-methyl-*N*-phenylbenzamide (105.6 mg, 0.50 mmol) with 1-phenylpentan-1-one (1.0 equiv) under *Procedure B* and reacted for 20 h to afford **3dq** (67.3 mg, 0.24 mmol, 48% yield); 4-methylbenzamide (67.6 mg, 0.50 mmol) with 1-phenylpentan-1-one (1.0 equiv) under the one-pot procedure to afford **3dq** (71.5 mg, 0.255 mmol, 51% yield); 4-methylbenzamide (67.6 mg, 0.50 mmol) with 1-phenylpentan-1-one (1.0 equiv) under the one-pot procedure and reacted for 20 h to afford **3dq** (70.1 mg, 0.25 mmol, 50% yield); ¹H NMR (500 MHz, CDCl₃) δ 7.97 (d, *J* = 7.4 Hz, 2H), 7.89 (d, *J* = 8.1 Hz, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.47–7.41 (m, 2H), 7.25 (d, *J* = 7.9 Hz, 2H), 5.19 (t, *J* = 6.6 Hz, 1H), 2.40 (s, 3H), 2.10 (dt, *J* = 14.2, 6.9 Hz, 2H), 1.51–1.40 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 196.2, 195.8, 144.4, 136.2, 133.6, 133.4, 129.6, 128.8, 128.7, 128.5, 57.0, 31.6, 21.7, 21.6, 14.2; HRMS (ESI-TOF) *m/z*: [M + Na]⁺ Calcd for C₁₉H₂₀O₂Na 303.1361; Found 303.1362.



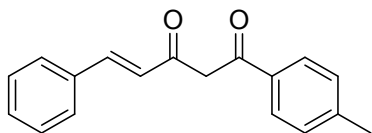
4,4-Dimethyl-1-(*p*-tolyl)pentane-1,3-dione ^[16] (**3dr**) [contain 96% enol form]

4-Methyl-*N*-phenyl-*N*-tosylbenzamide (109.6 mg, 0.30 mmol) afforded 4,4-dimethyl-1-(*p*-tolyl)pentane-1,3-dione (**3dr**) (62.9 mg, 0.29 mmol, 96% yield) as light yellow oil; 4-methyl-*N*-phenylbenzamide (105.6 mg, 0.50 mmol) afforded **3dr** (92.9 mg, 0.455 mmol, 91% yield) under *Procedure B*; 4-methylbenzamide (67.6 mg, 0.50 mmol) afforded **3dr** (94.0 mg, 0.46 mmol, 92% yield) under the one-pot procedure; ¹H NMR (500 MHz, CDCl₃) δ 16.58 (s, 1H), 7.80 (d, *J* = 8.2 Hz, 2H), 7.26 (d, *J* = 8.2 Hz, 2H), 6.29 (s, 1H), 2.42 (s, 3H), 1.26 (s, 9H); ¹³C NMR (126 MHz, CDCl₃) δ 202.1, 185.0, 142.8, 132.9, 129.3, 127.0, 91.7, 39.7, 27.4, 26.2, 21.6.



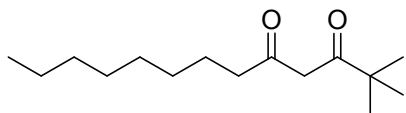
1-(*p*-tolyl)hexane-1,3-dione (**3ds**) [contain 89% enol form]

4-Methyl-*N*-phenyl-*N*-tosylbenzamide (109.6 mg, 0.30 mmol) afforded 1-(*p*-tolyl)hexane-1,3-dione (**3ds**) (51.5 mg, 0.25 mmol, 84% yield) as light yellow oil by silica gel column chromatography with ethyl acetate in hexane (0% to 5%); ¹H NMR (500 MHz, CDCl₃) δ 16.24 (br s, 1H), 7.91–7.71 (m, 2H), 7.25 (d, *J* = 7.8 Hz, 2H), 6.15 (s, 1H), 2.62–2.31 (m, 5H), 1.80–1.59 (m, 2H), 1.09–0.84 (m, 2H); ¹³C NMR (126 MHz, CDCl₃) δ 195.9, 184.1, 142.9, 132.4, 129.3, 127.1, 95.7, 40.9, 21.6, 19.3, 13.8; HRMS (FD-TOF) *m/z*: [M]⁺ Calcd for C₁₃H₁₆O₂ 204.1145; Found 204.1147.



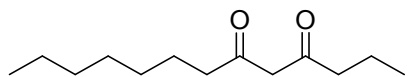
(*E*)-5-Phenyl-1-(*p*-tolyl)pent-4-ene-1,3-dione ^[18] (**3vd**) [contain 97% enol form]

N-(*p*-Tolyl)-*N*-tosylcinnamamide (117.5 mg, 0.30 mmol) afforded (*E*)-5-phenyl-1-(*p*-tolyl)pent-4-ene-1,3-dione (**3vd**) (44.4 mg, 0.17 mmol, 56% yield) as yellow solid by silica gel column chromatography with ethyl acetate in hexane (0% to 10%); Mp: 95–96 °C; ¹H NMR (500 MHz, CDCl₃) δ 16.09 (s, 1H), 7.78 (d, *J* = 8.1 Hz, 2H), 7.59 (d, *J* = 15.8 Hz, 1H), 7.50–7.44 (m, 2H), 7.34–7.27 (m, 3H), 7.21–7.08 (m, 3H), 6.56 (d, *J* = 15.8 Hz, 1H), 6.24 (s, 1H), 2.33 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 189.5, 178.8, 143.4, 139.7, 135.1, 133.6, 129.9, 129.4, 128.9, 128.0, 127.5, 123.4, 97.6, 21.7.



2,2-Dimethyltridecane-3,5-dione (**3wr**) [contain 86% enol form]

N-Phenyl-*N*-tosylnonanamide (116.2 mg, 0.30 mmol) afforded 2,2-dimethyltridecane-3,5-dione (**3wr**) (30.3 mg, 0.13 mmol, 42% yield) as yellow oil by silica gel column chromatography with ethyl acetate in hexane (0% to 5%); ¹H NMR (500 MHz, cdcl₃) δ [15.78 (br s, 0.86H), 5.53 (s, 0.86H), 3.56 (s, 0.28H)], [2.43 (t, *J* = 7.1 Hz, 0.28H), 2.23 (t, *J* = 7.1 Hz, 1.72H)], 1.59–1.48 (m, 2H), 1.29–1.17 (m, 10H), 1.09 (d, *J* = 5.5 Hz, 9H), 0.81 (t, *J* = 6.9 Hz, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 200.3, 195.7, 95.0, 39.0, 38.8, 31.8, 29.3, 29.2, 29.1, 27.3, 25.8, 22.6, 14.0; HRMS (FD-TOF) *m/z*: [M]⁺ Calcd for C₁₅H₂₈O₂ 240.2084; Found 240.2087.



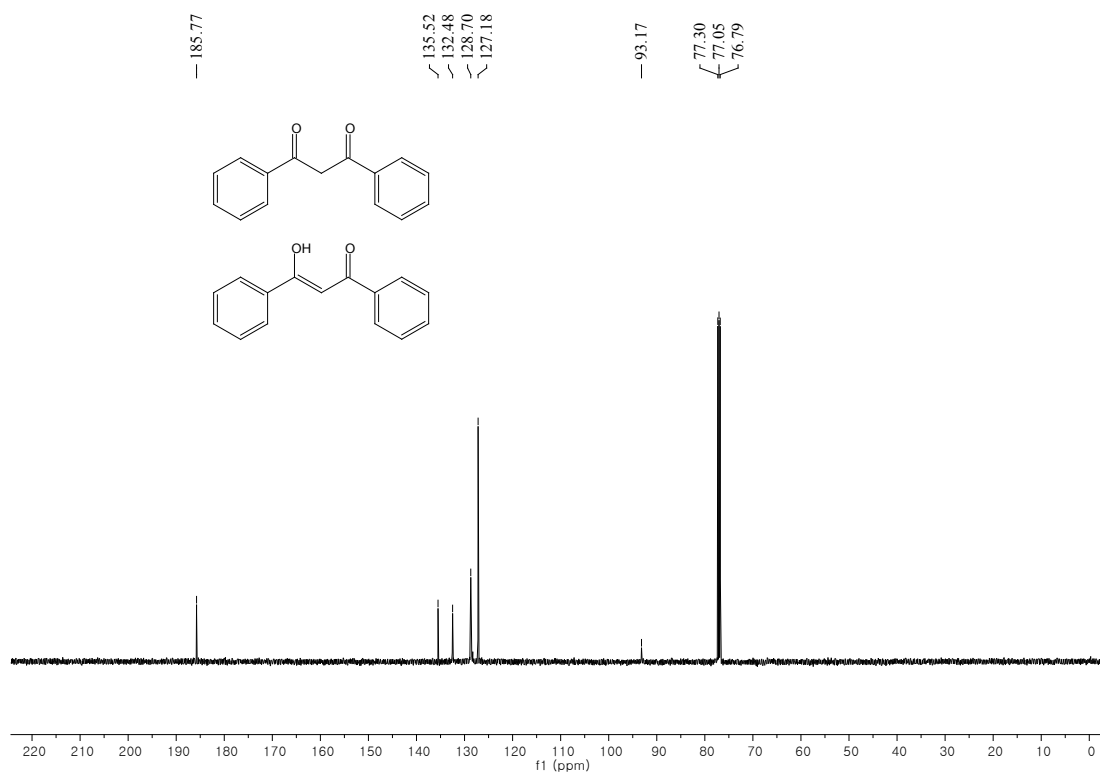
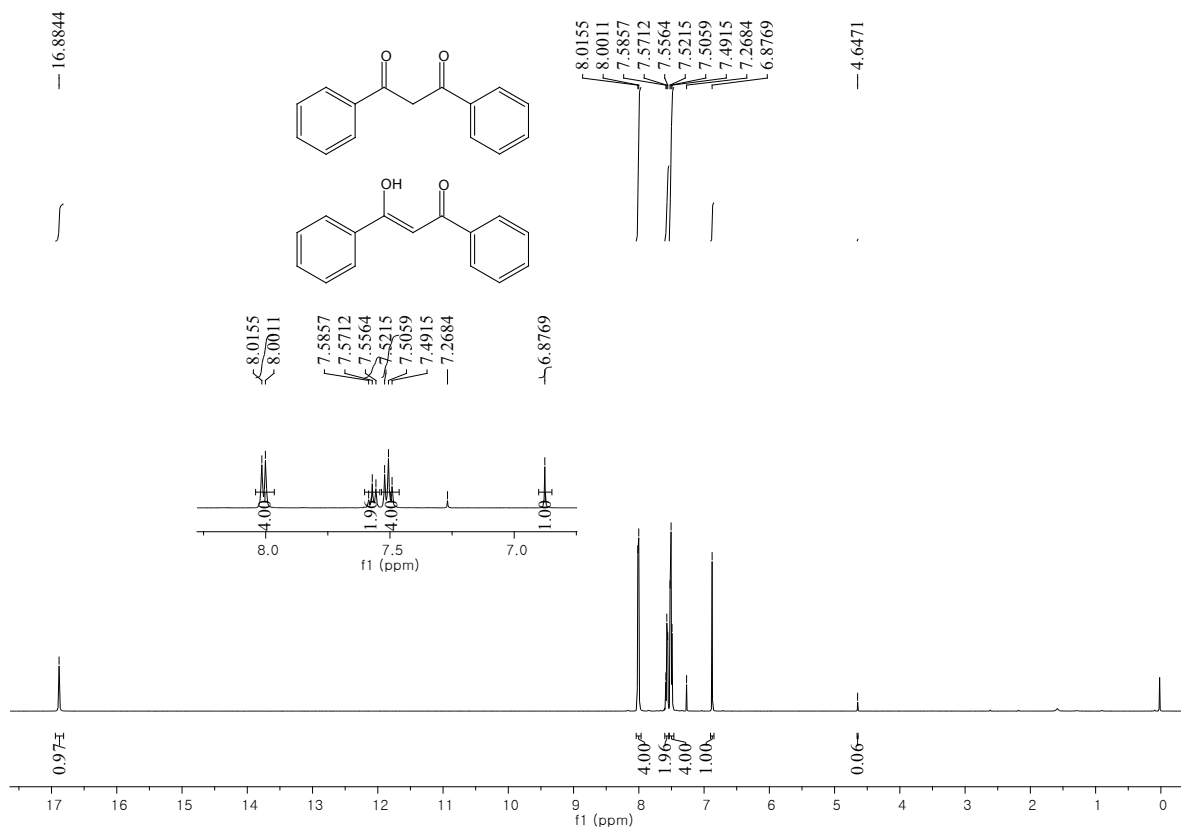
Tridecane-4,6-dione (3xs) ^[19] [contain 80% enol form]

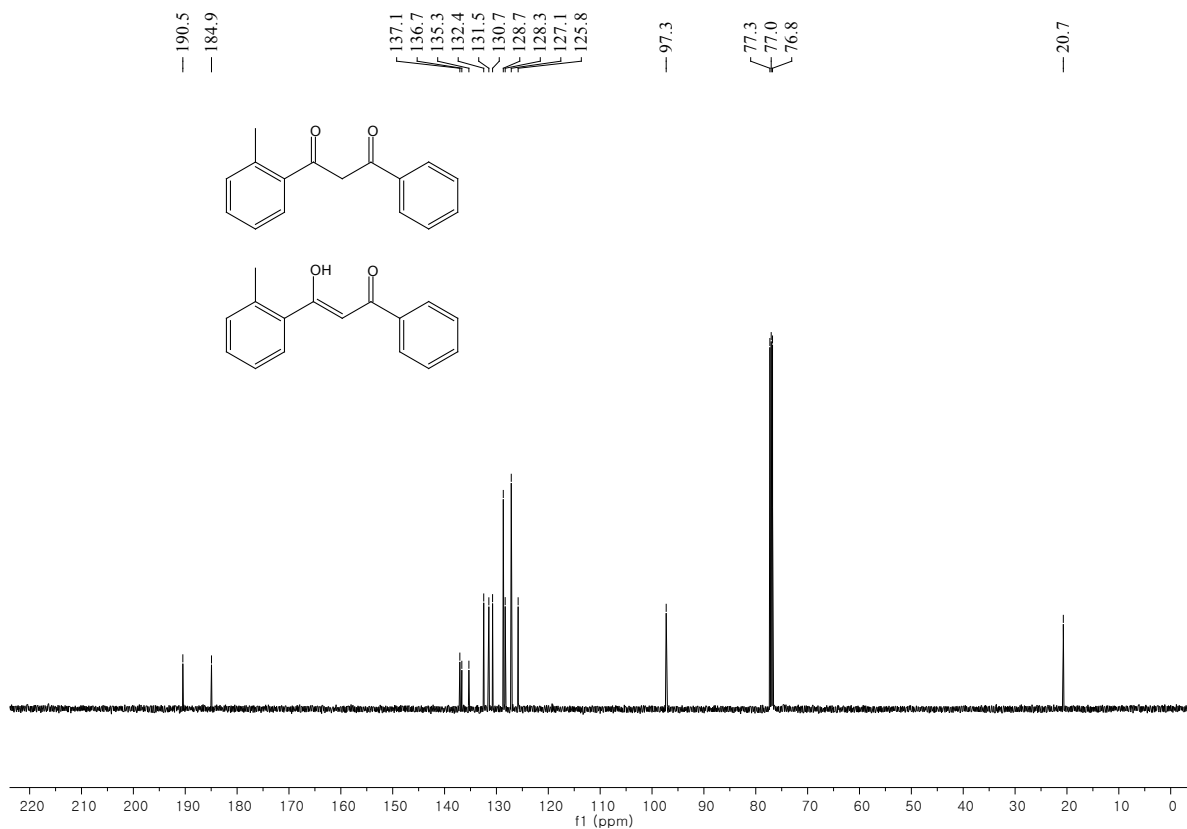
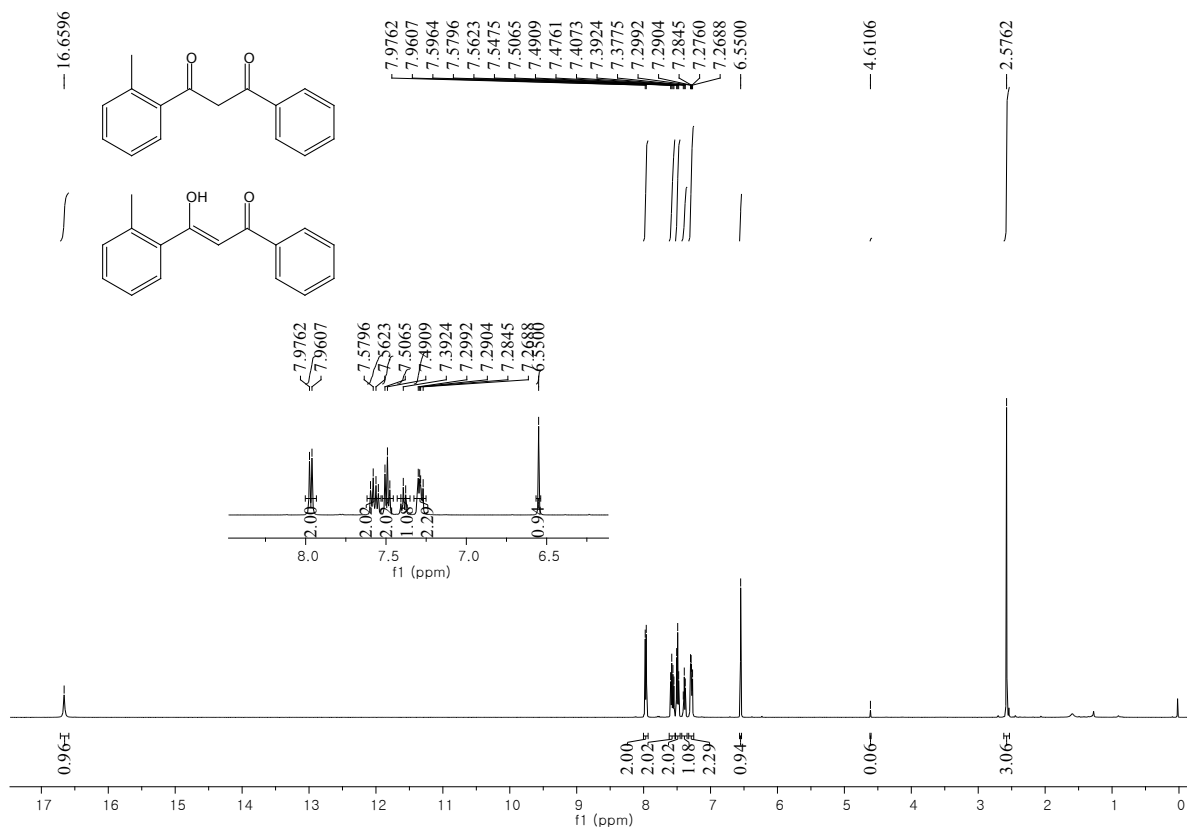
N-Phenyl-*N*-tosyloctanamide (112.1 mg, 0.30 mmol) afforded tridecane-4,6-dione (**3xs**) (19.7 mg, 0.09 mmol, 31% yield) as yellow oil by silica gel column chromatography with ethyl acetate in hexane (0% to 5%); ¹H NMR (500 MHz, CDCl₃) δ [15.52 (br s, 0.8H), 5.47 (s, 0.8H), 3.54 (s, 0.4H)], 2.62–2.21 (m, 4H), 1.70–1.52 (m, 4H), 1.38–1.17 (m, 8H), 1.02–0.81 (m, 6H); ¹³C NMR (126 MHz, CDCl₃) δ 204.5, 204.3, 194.7^{enol}, 194.1^{enol}, 99.1^{enol}, 57.2, 45.7, 43.8, 40.2^{enol}, 38.5^{enol}, 31.7^{enol}, 31.6, 29.2^{enol}, 29.01^{enol}, 28.99, 25.7^{enol}, 23.4, 22.60^{enol}, 22.58, 19.1^{enol}, 16.9, 14.1^{enol}, 13.7^{enol}, 13.6.

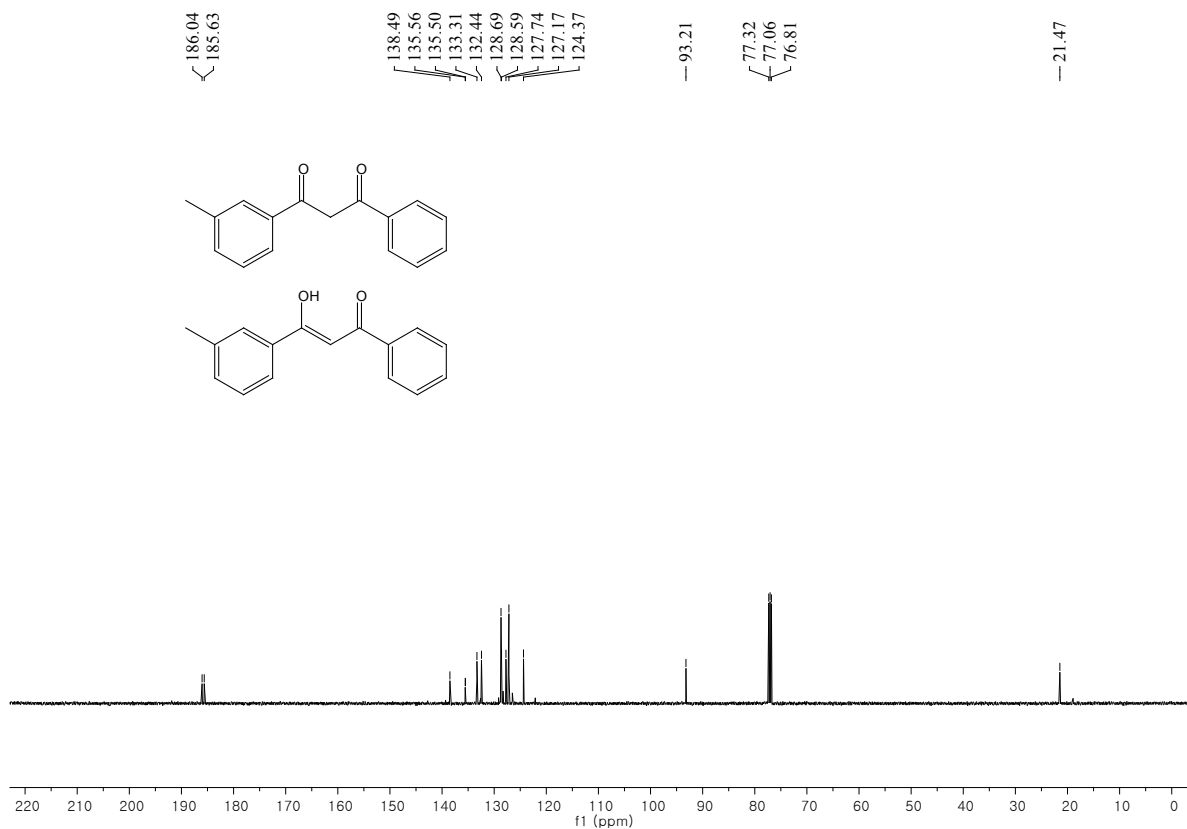
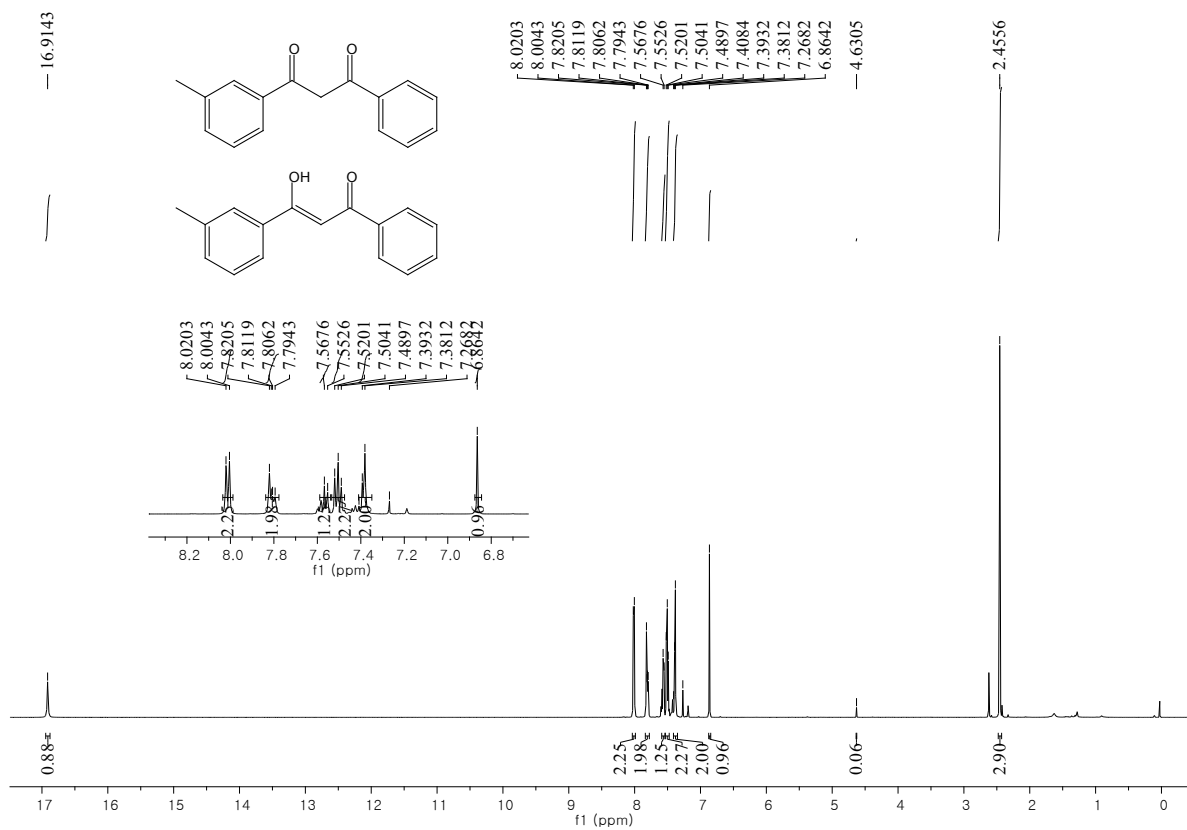
8. References

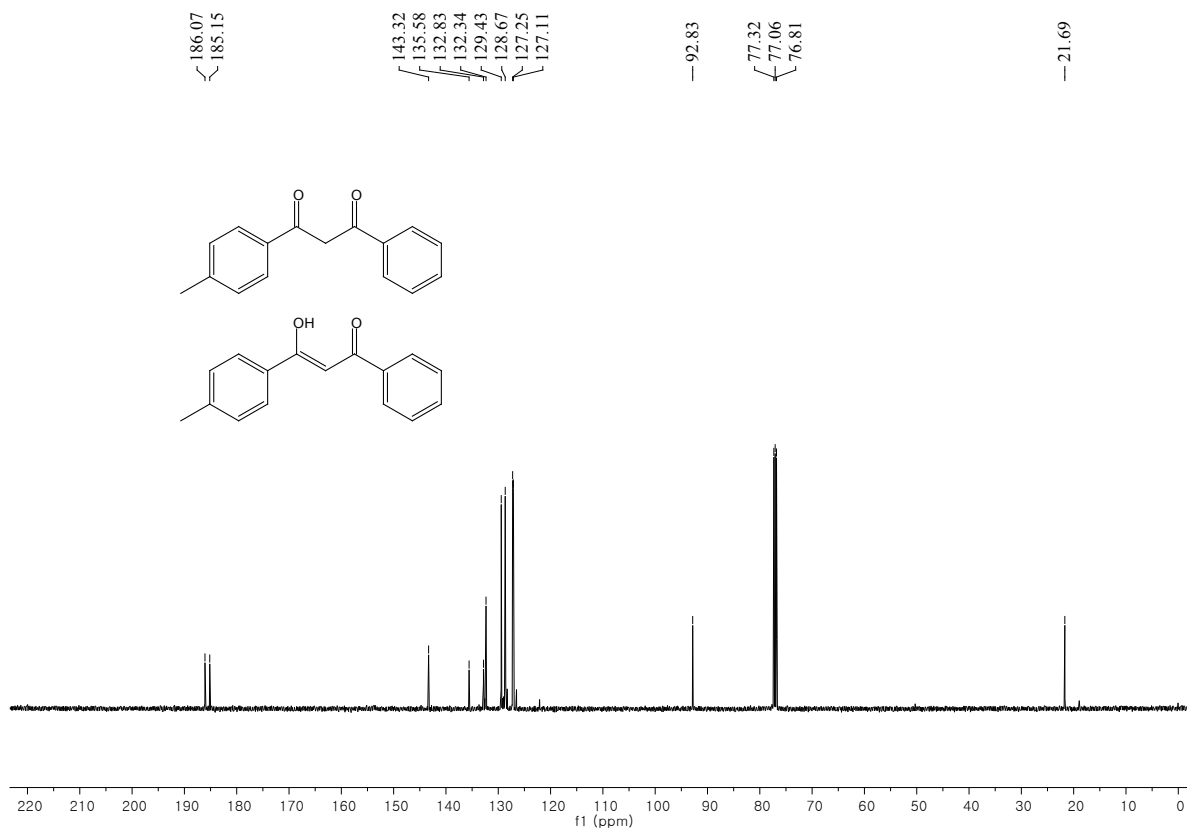
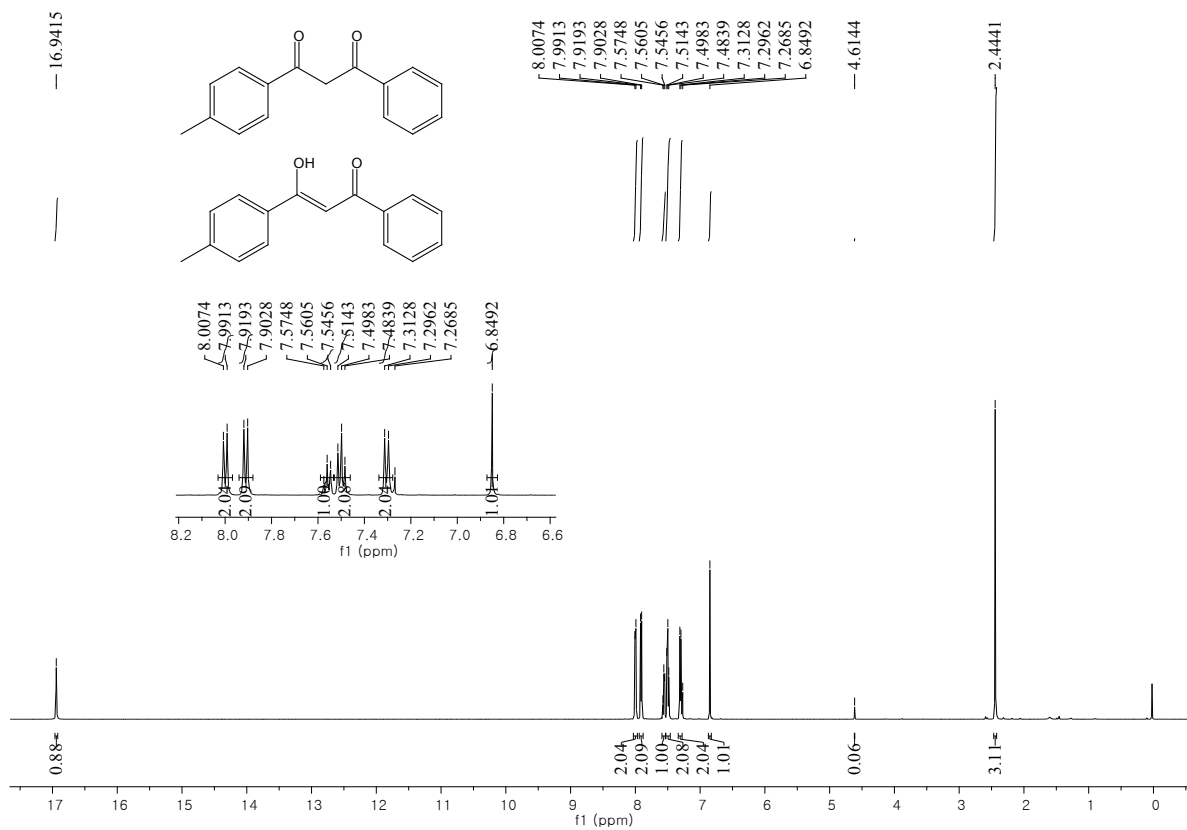
- [1] S. Shi, G. Meng and M. Szostak, *Angew. Chem. Int. Ed.*, 2016, **55**, 6959.
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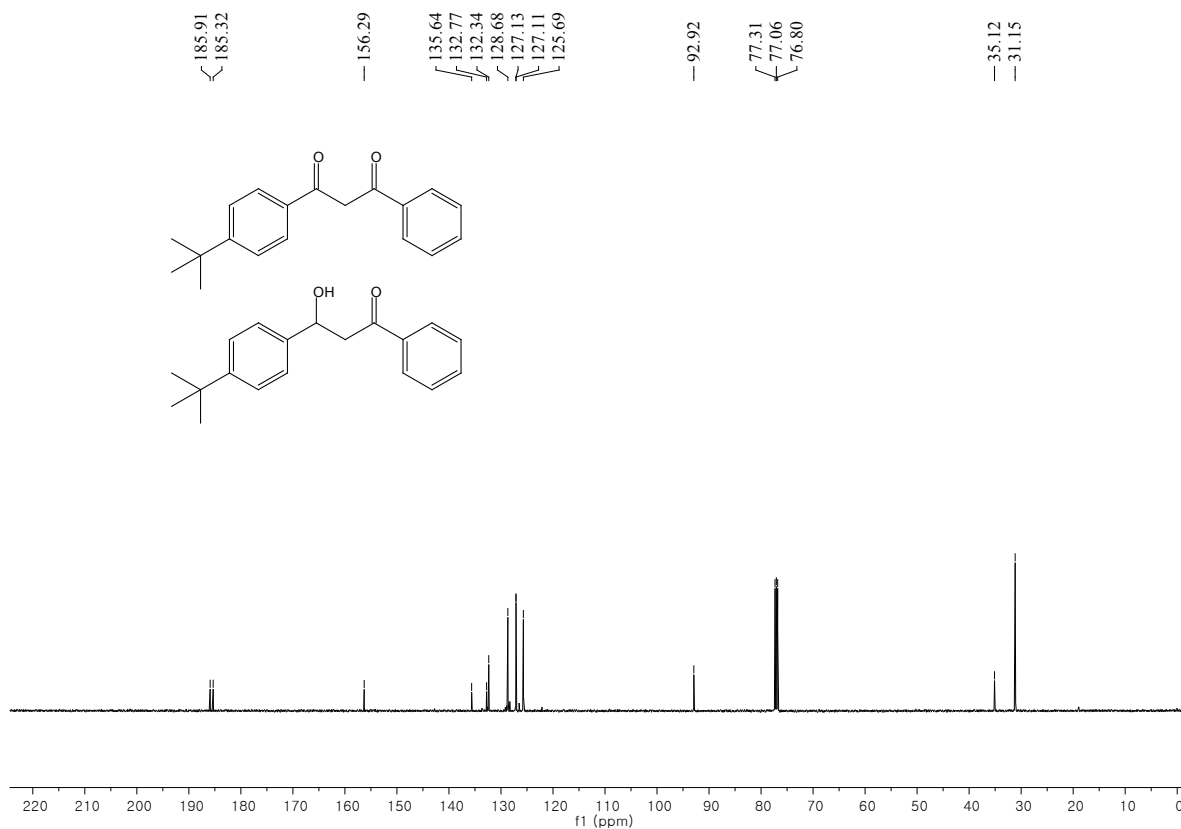
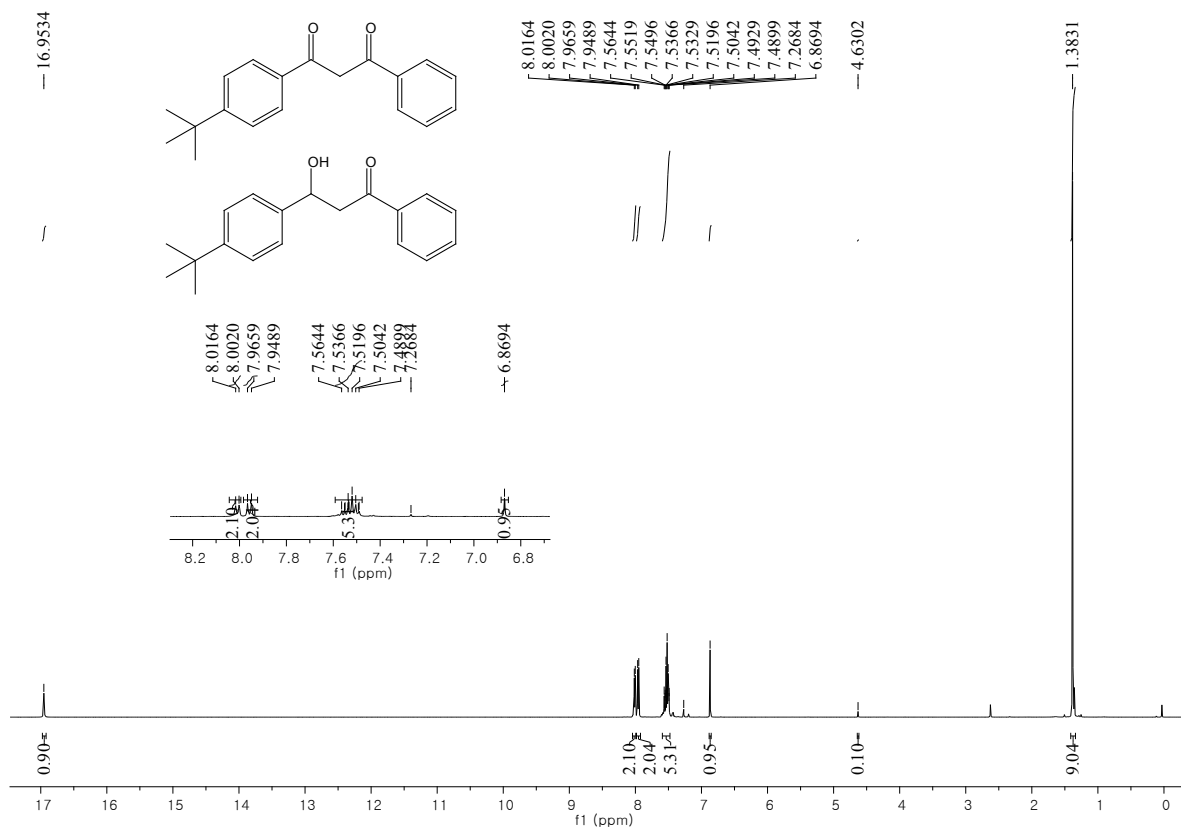
9. Copy of NMR Spectra

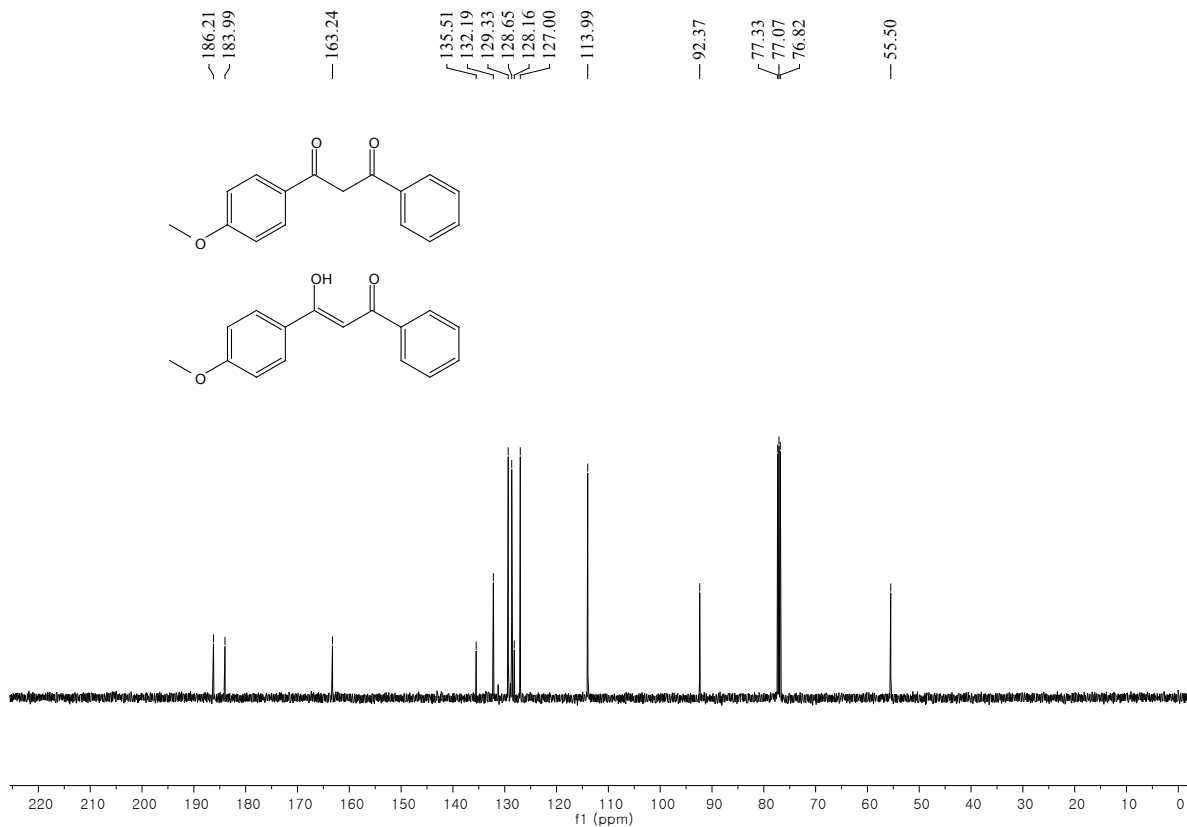
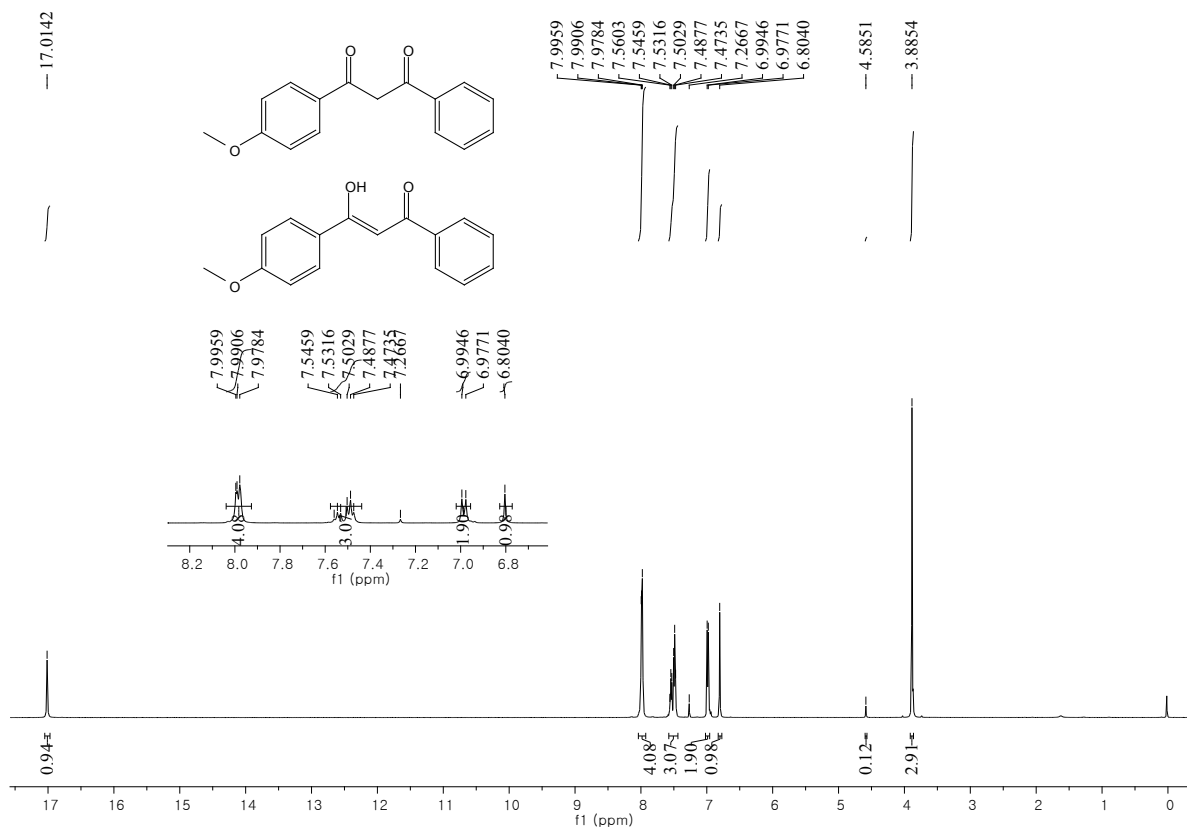


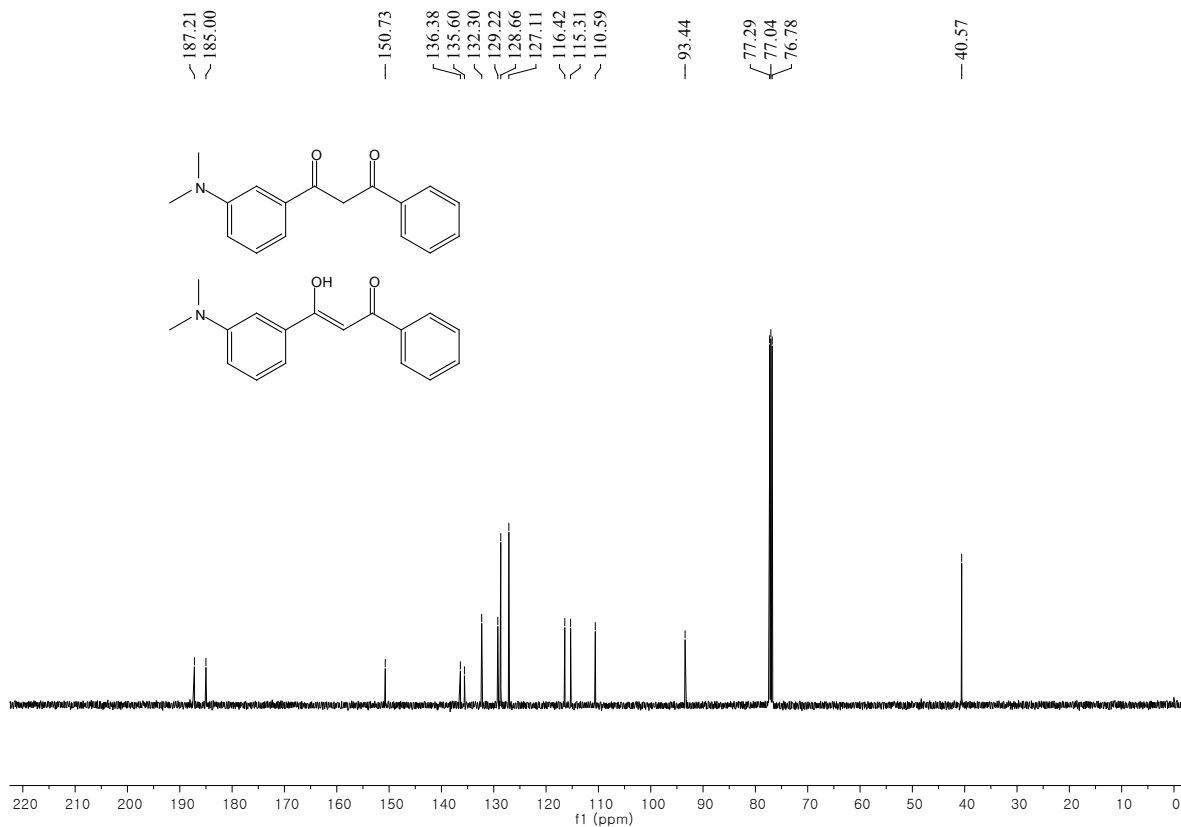
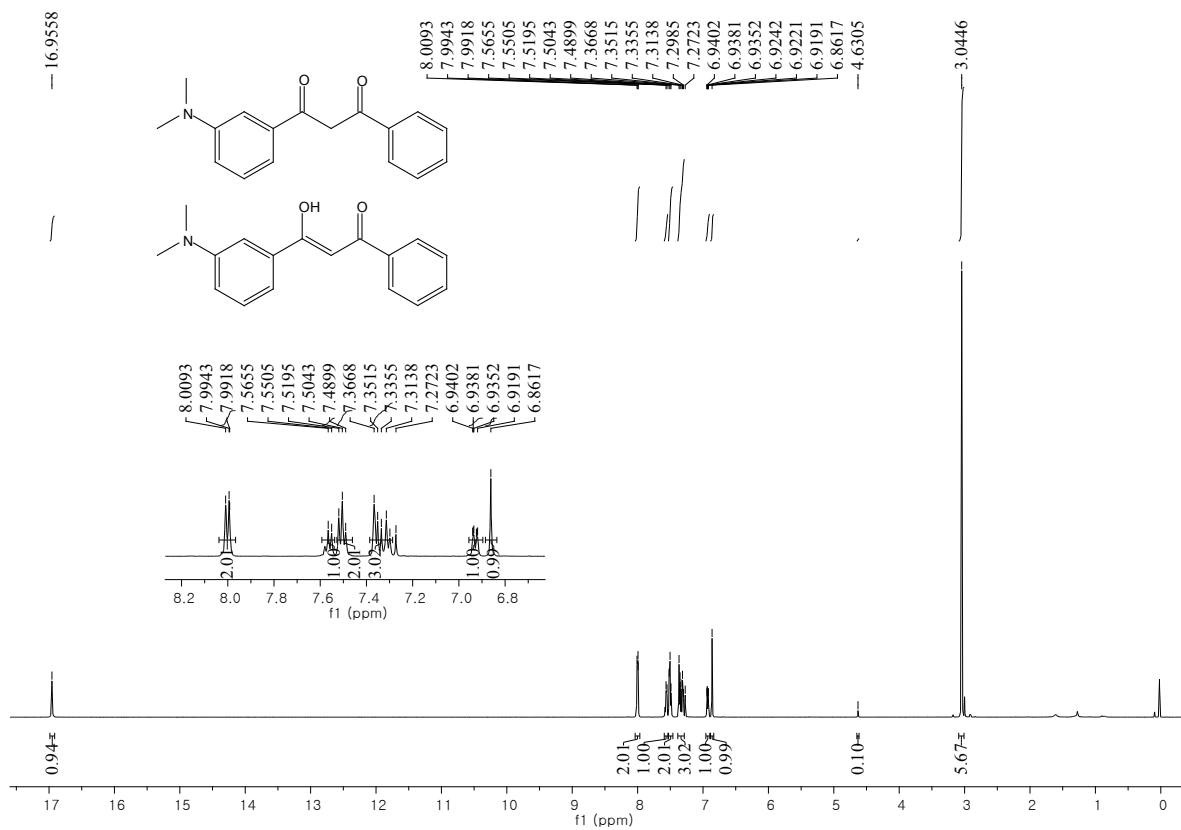


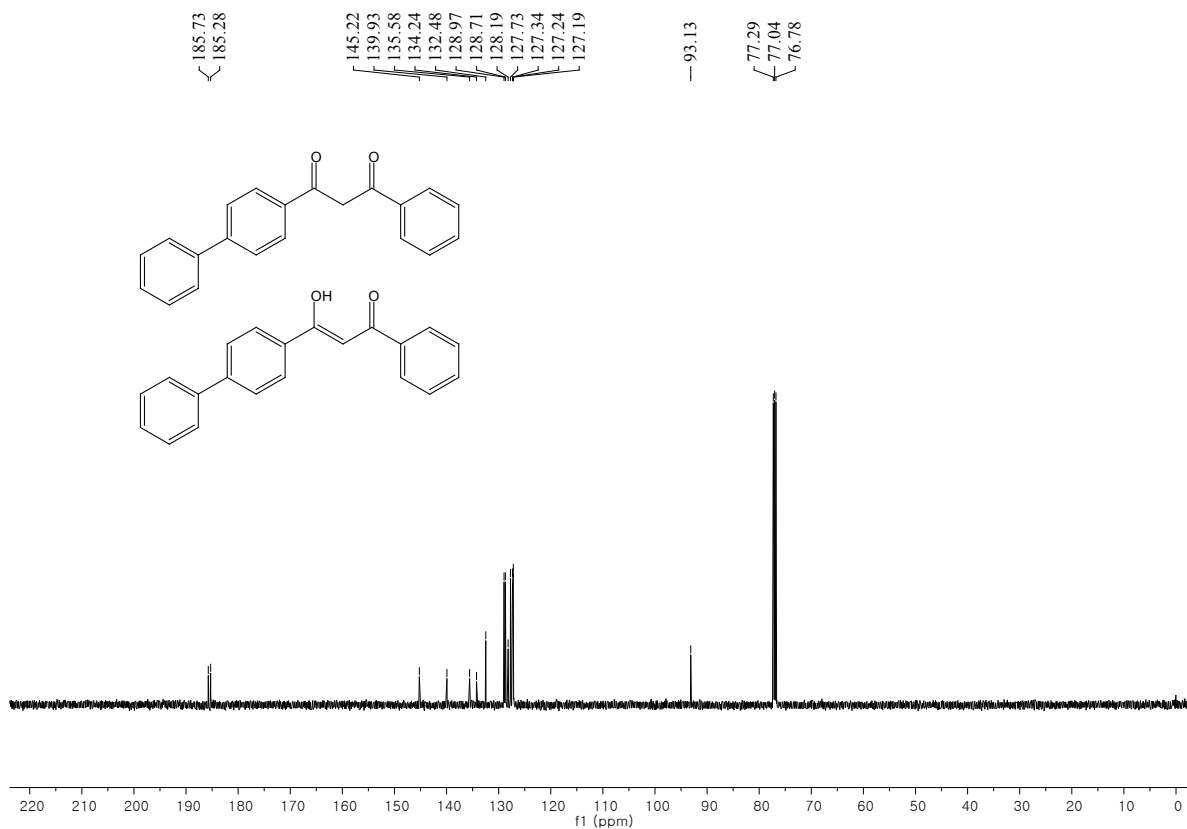
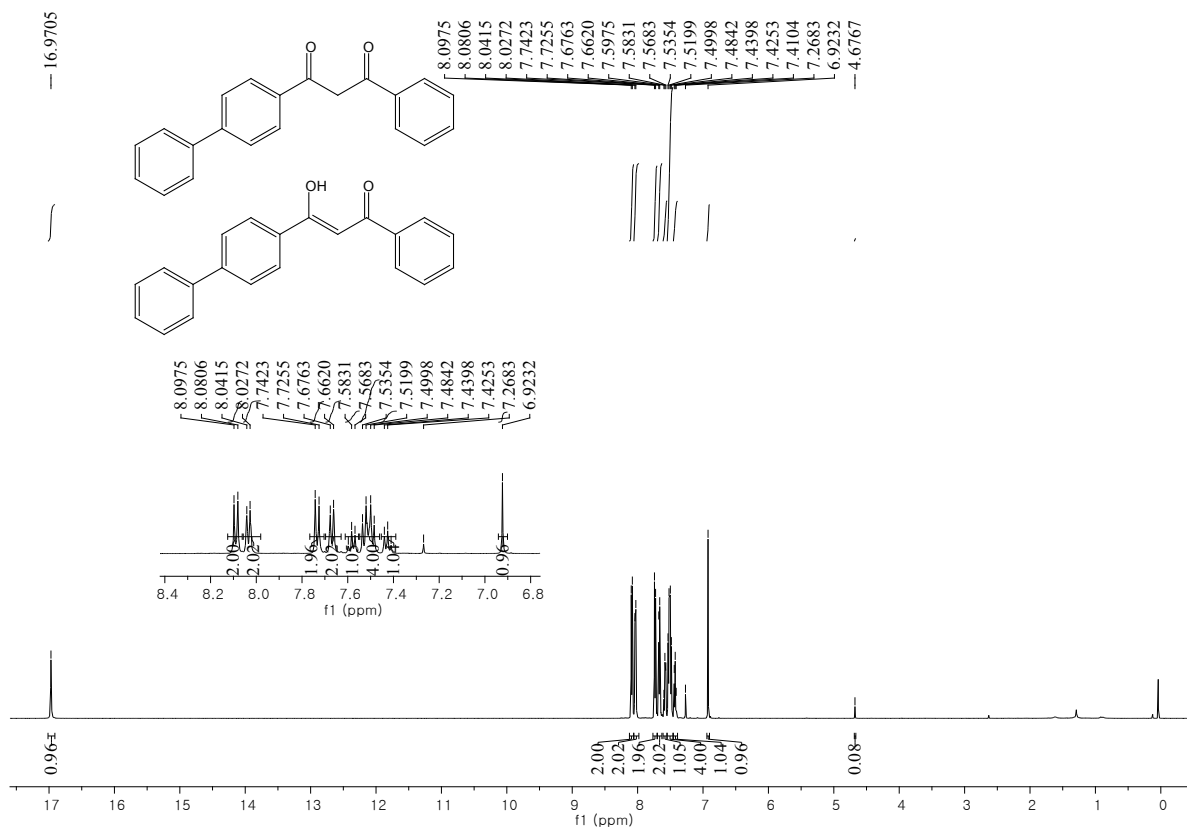


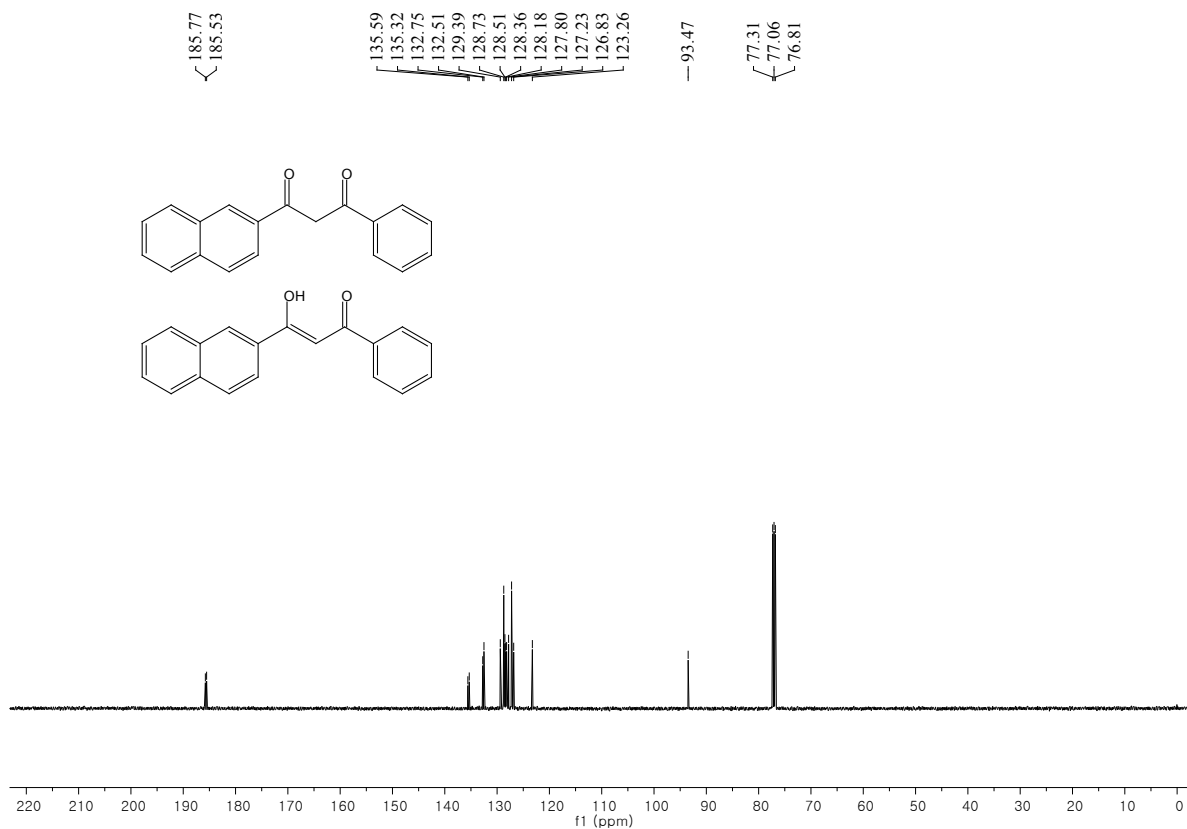
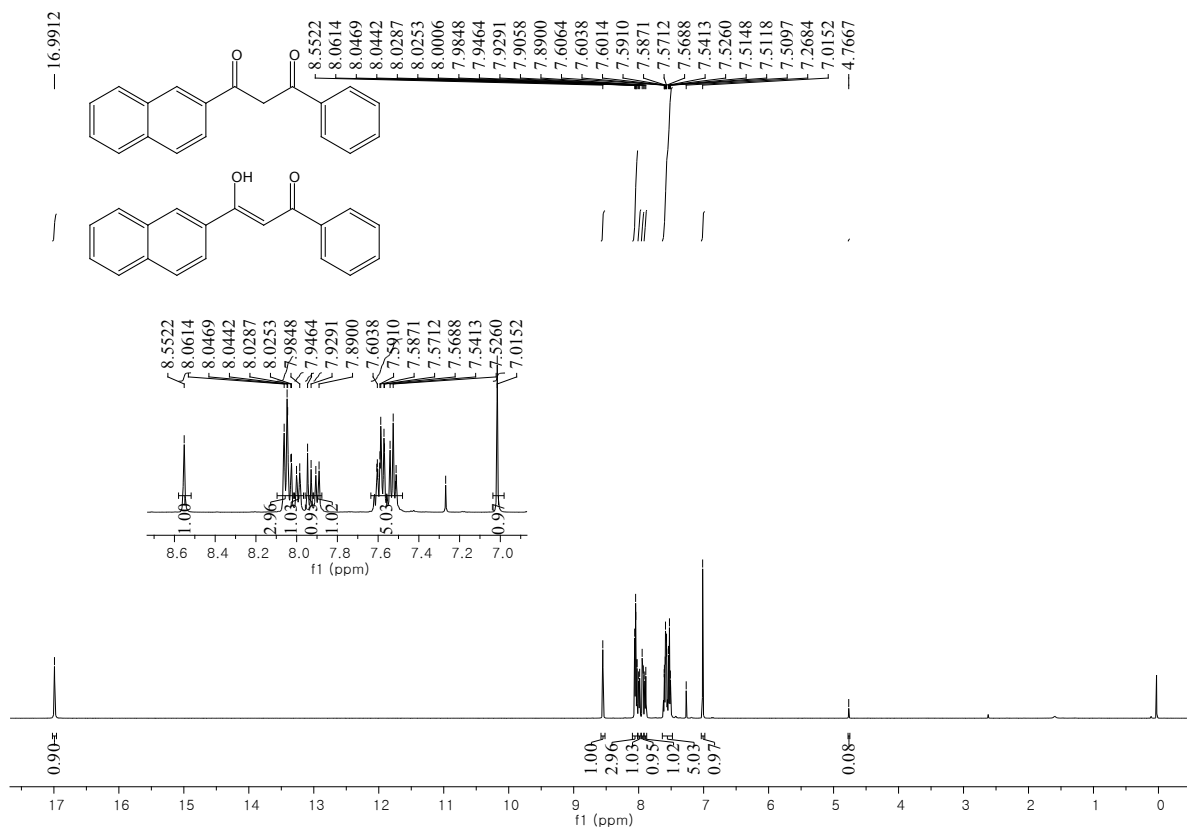


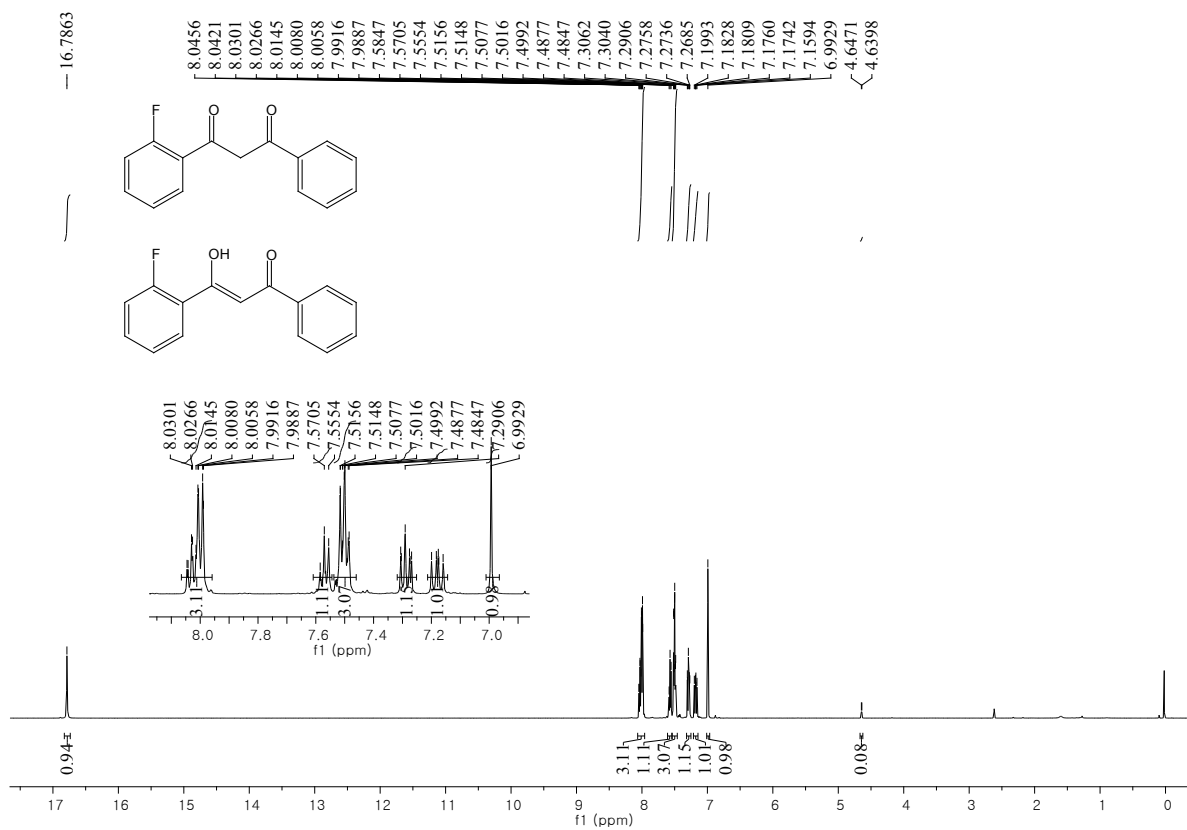




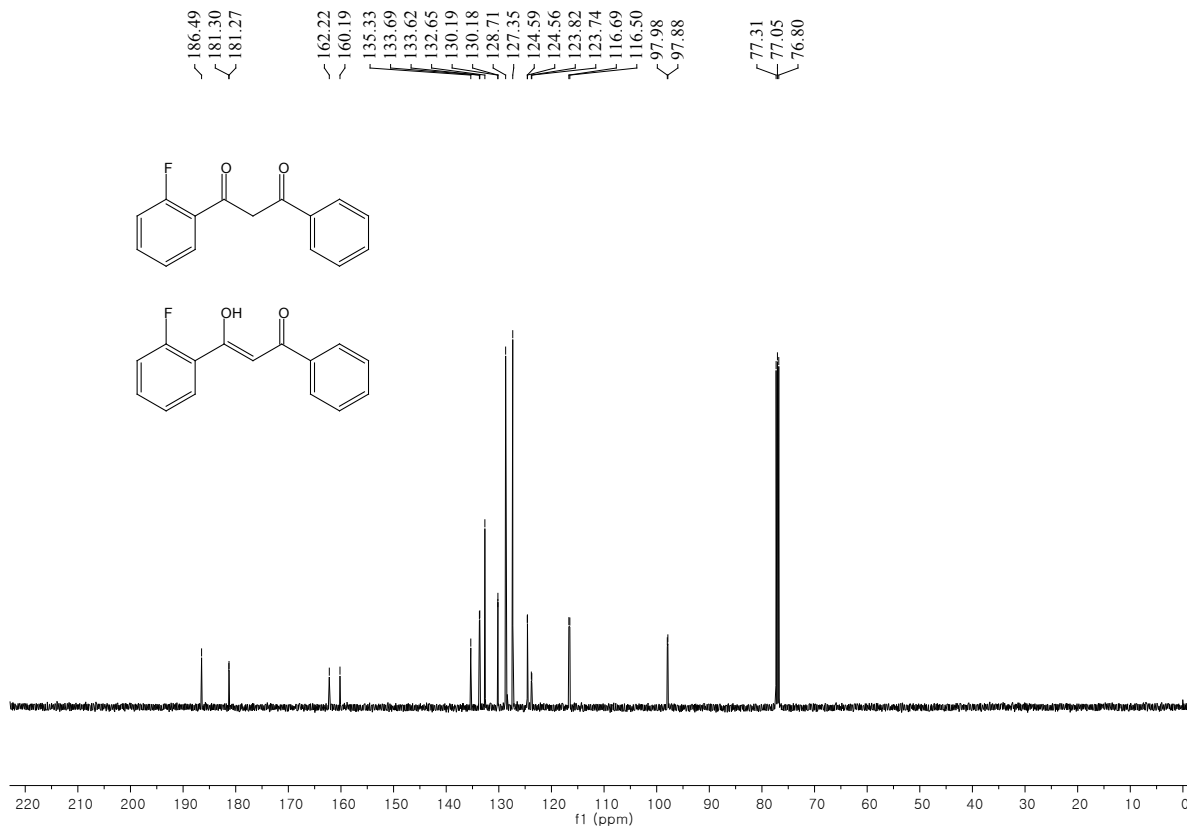




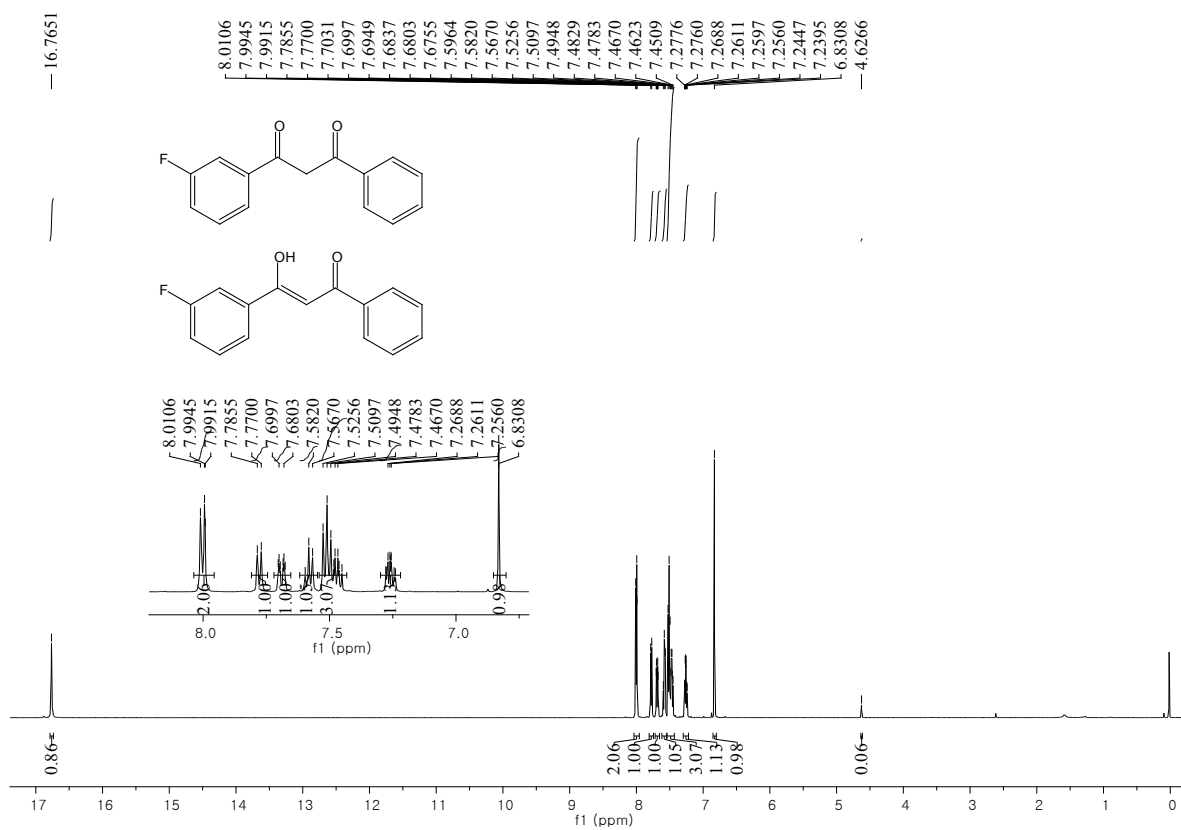




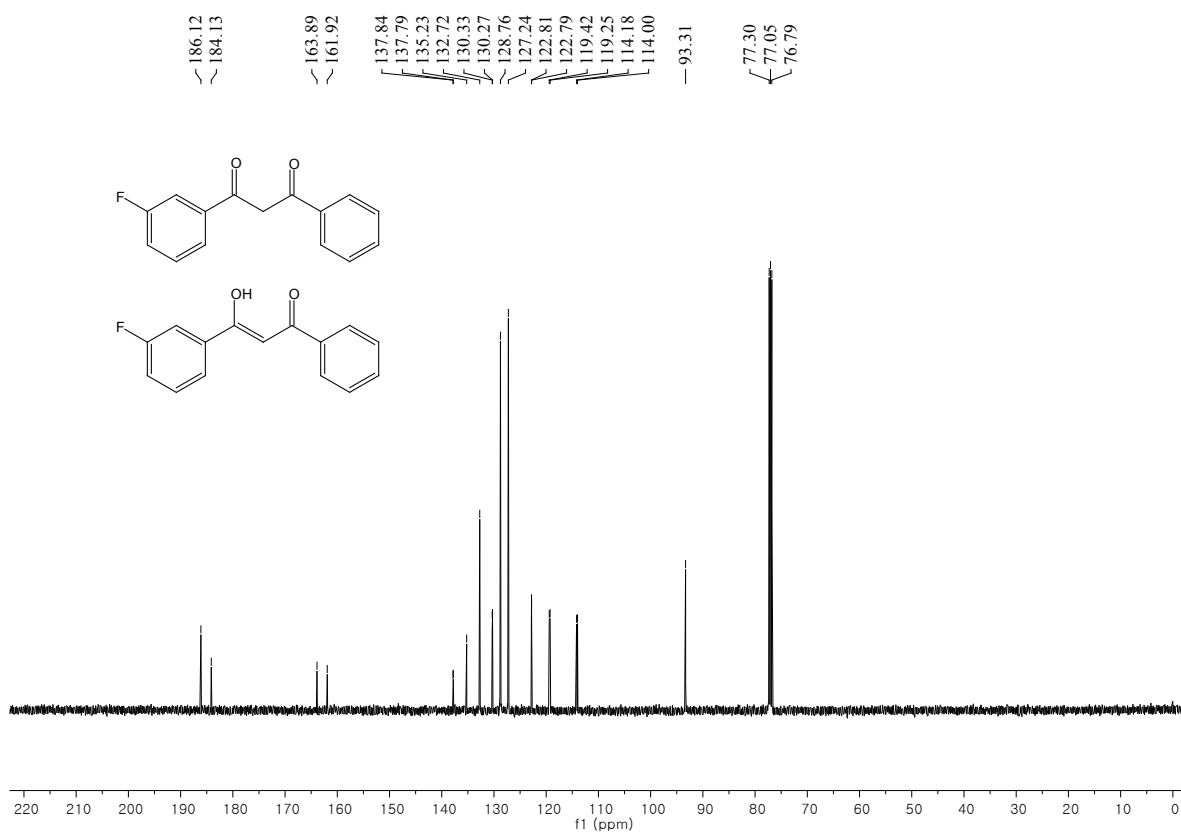
¹H NMR (500 MHz, CDCl₃): **3ja**



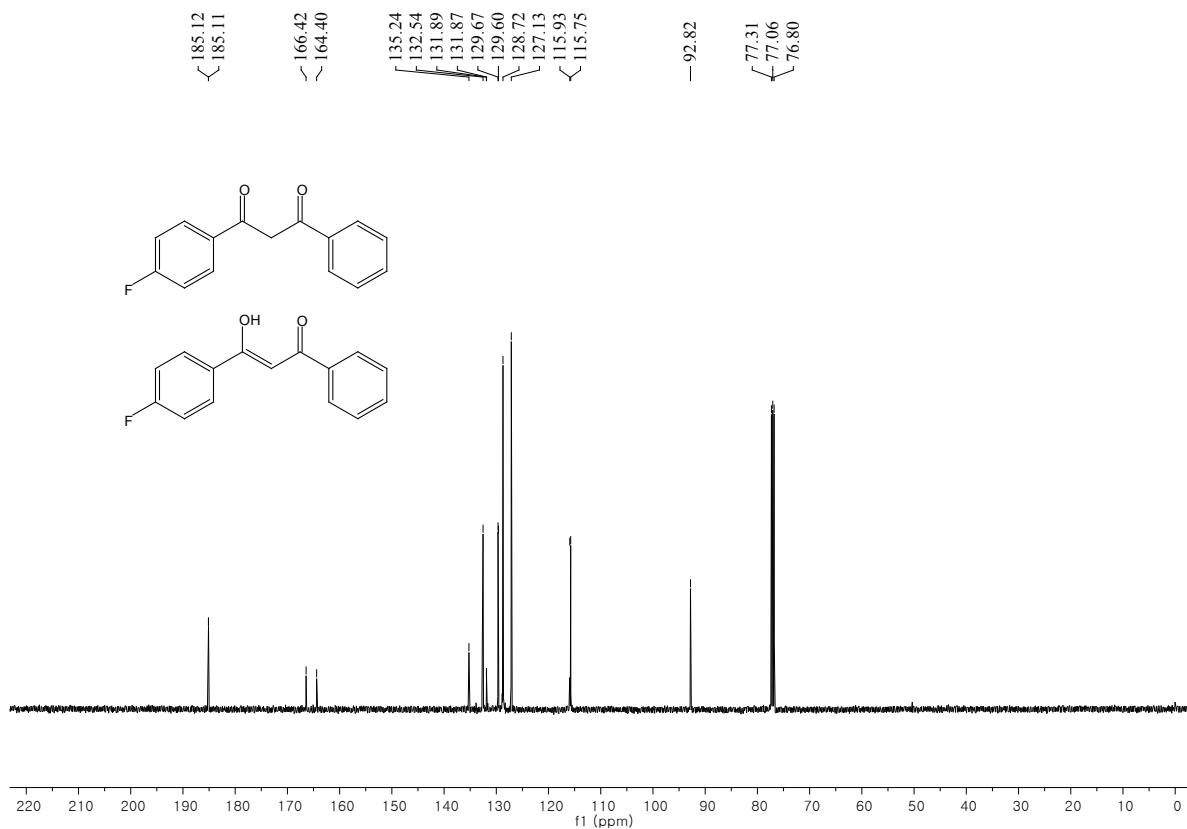
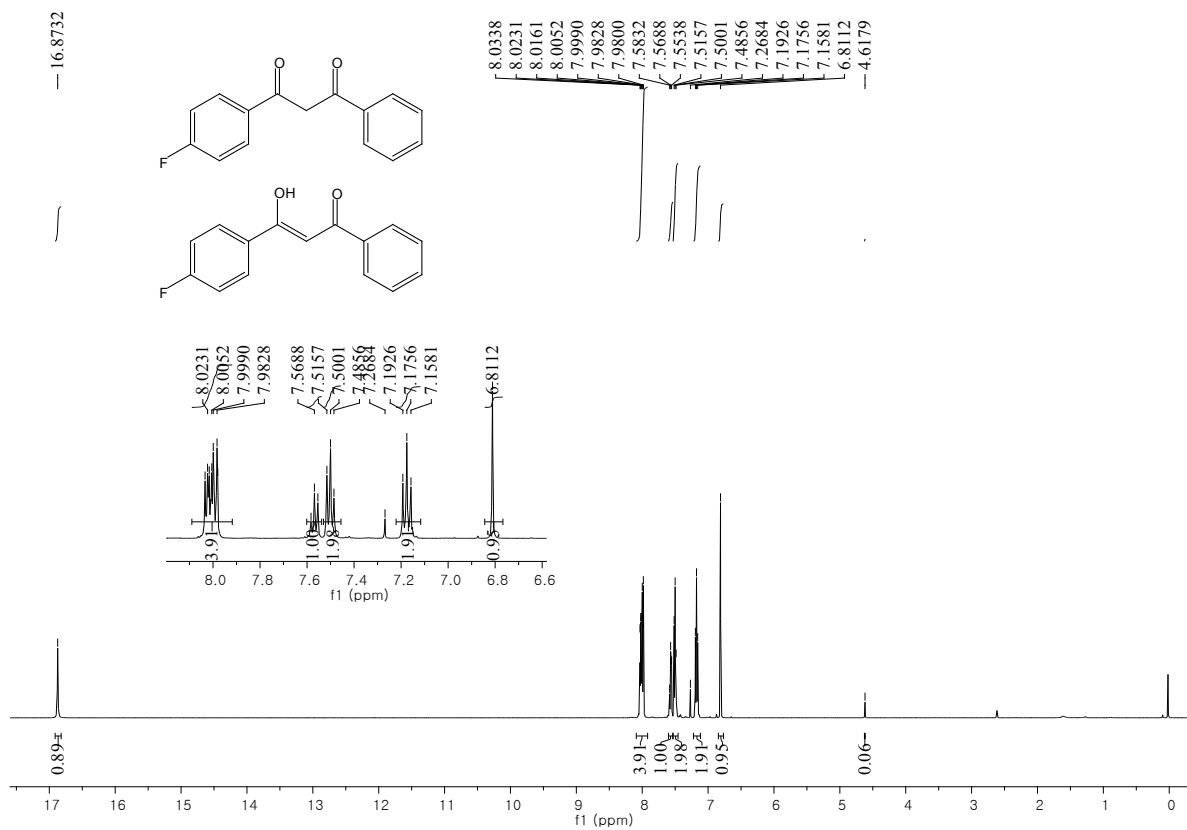
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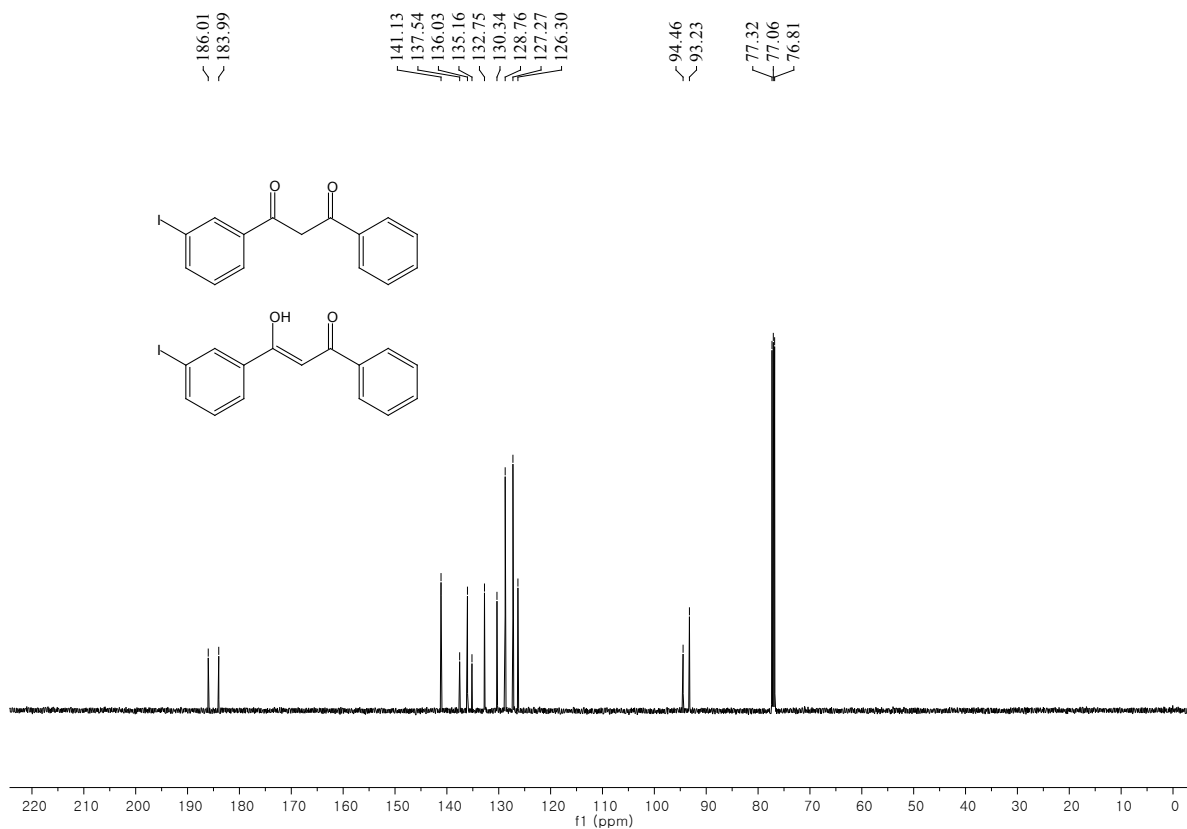
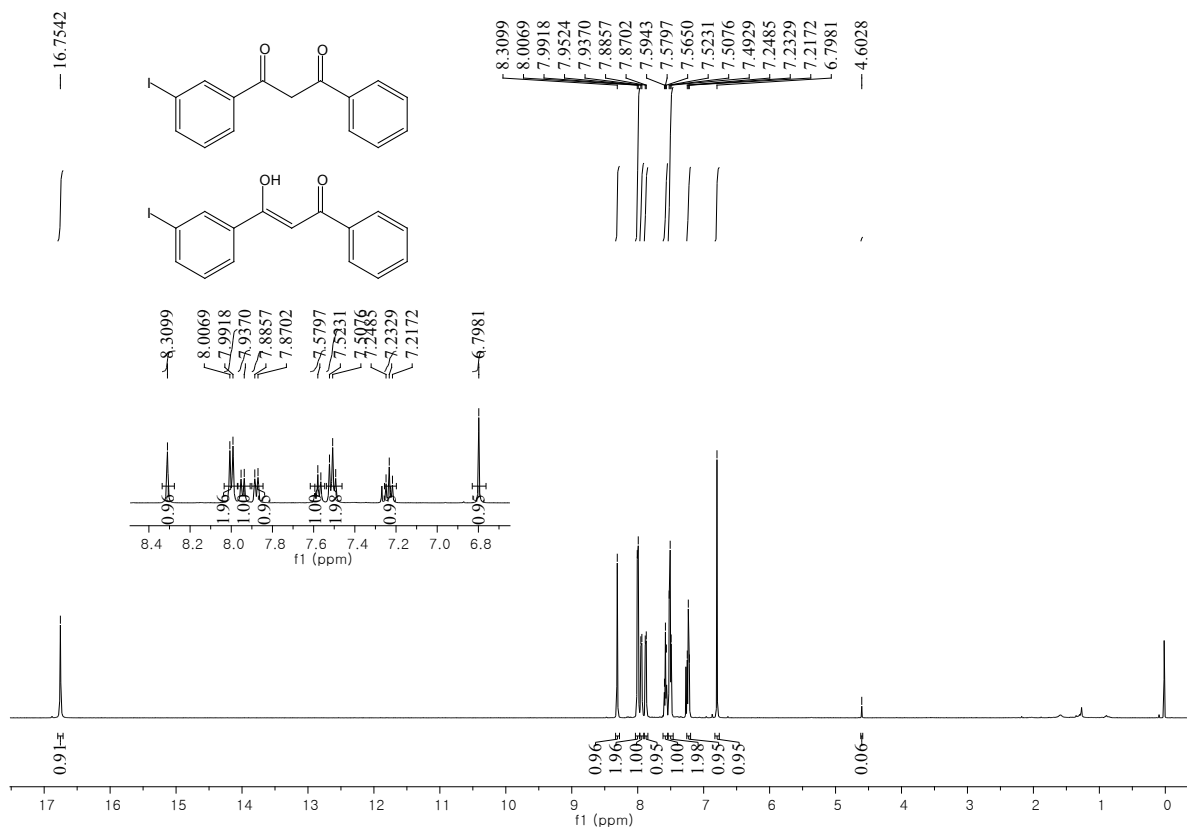


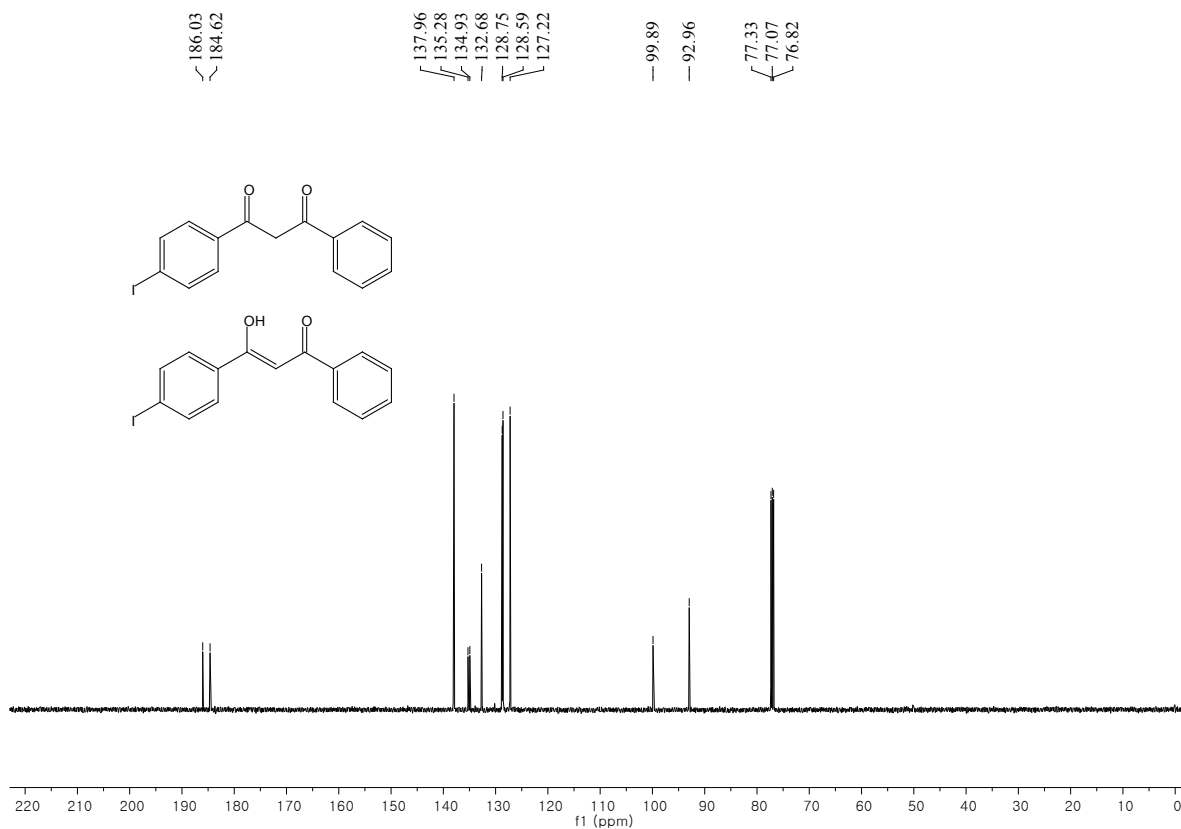
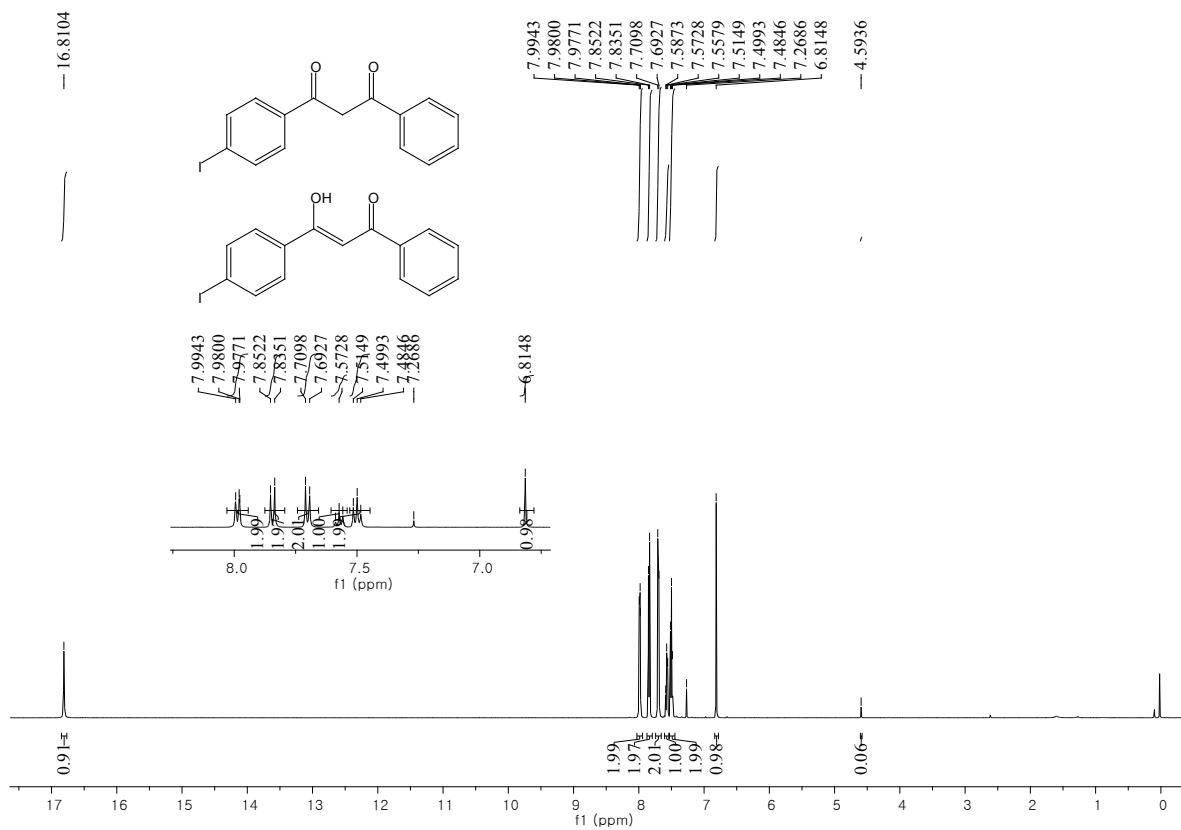
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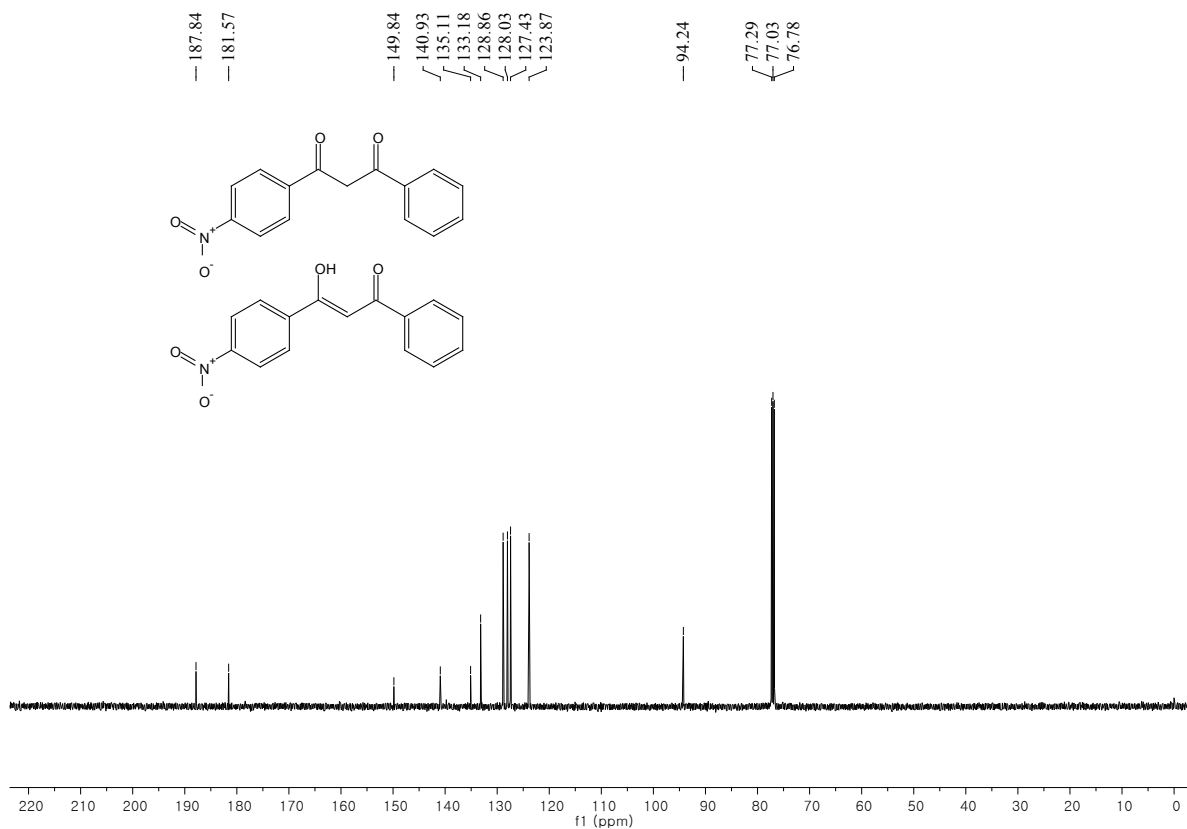
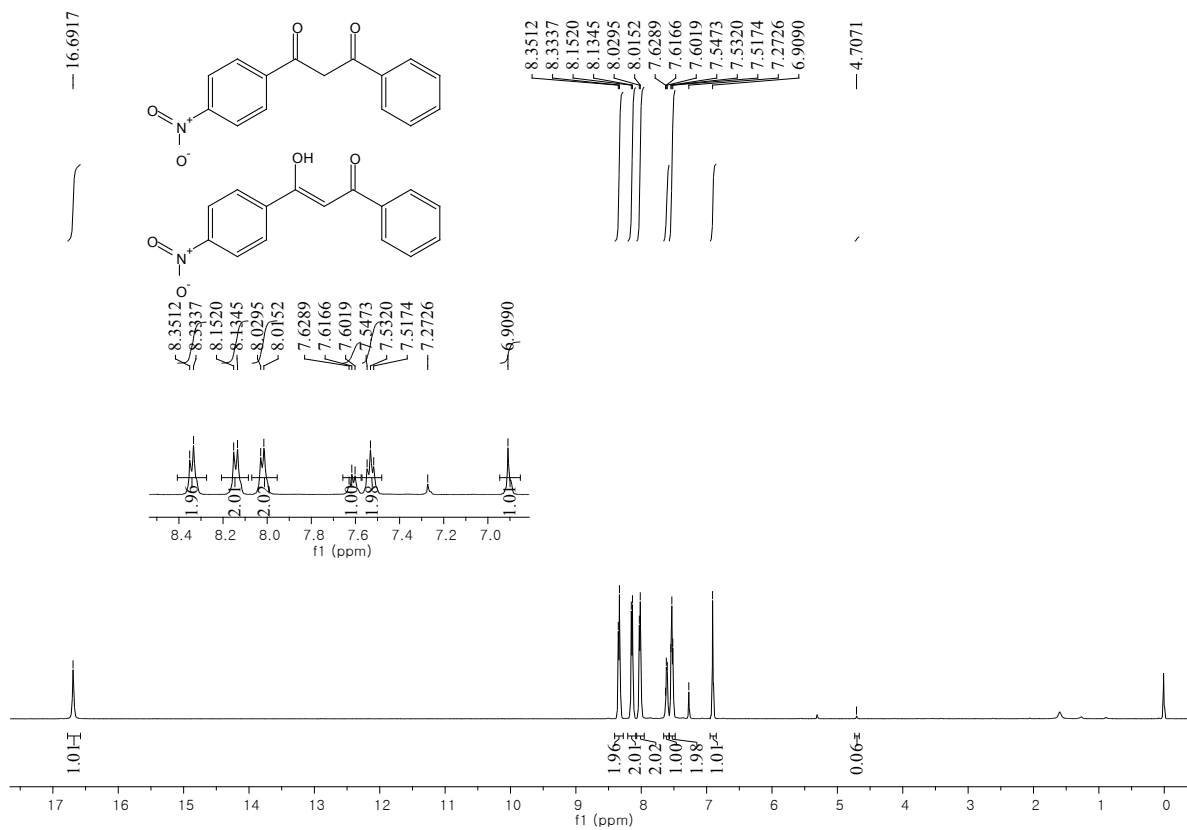


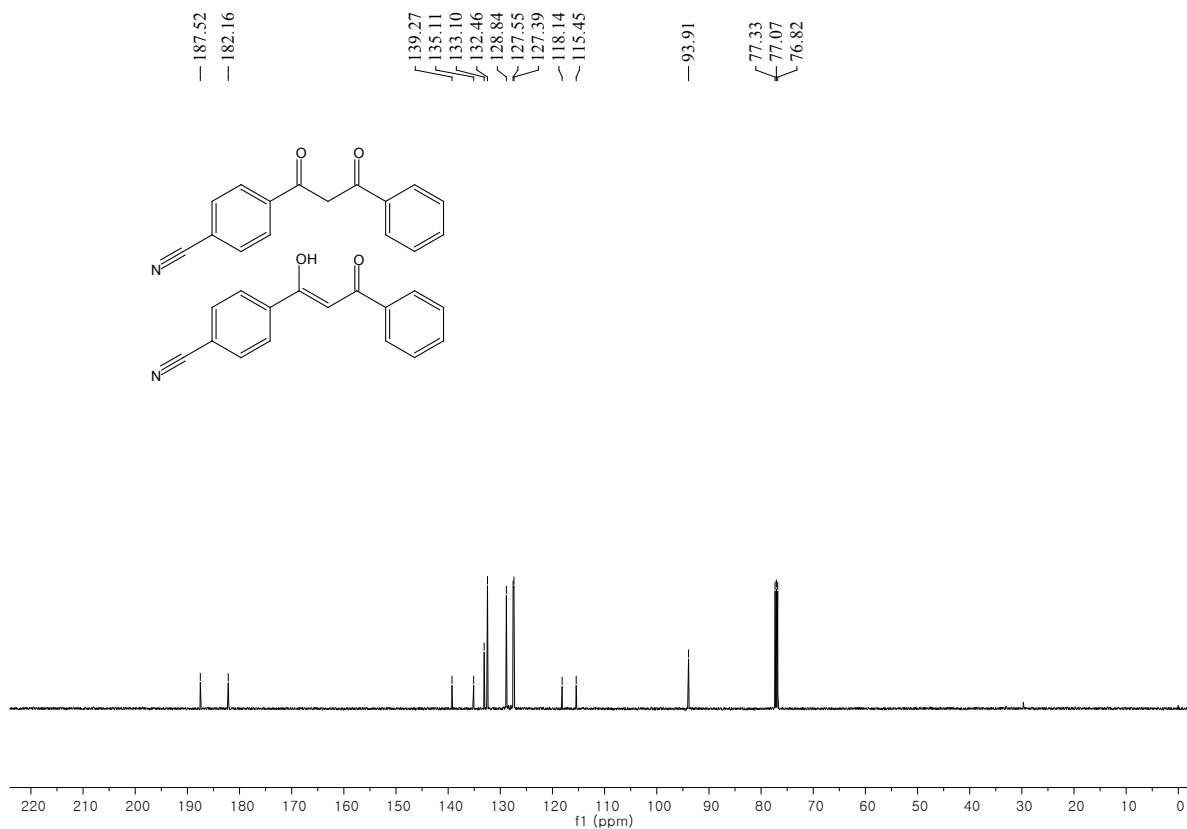
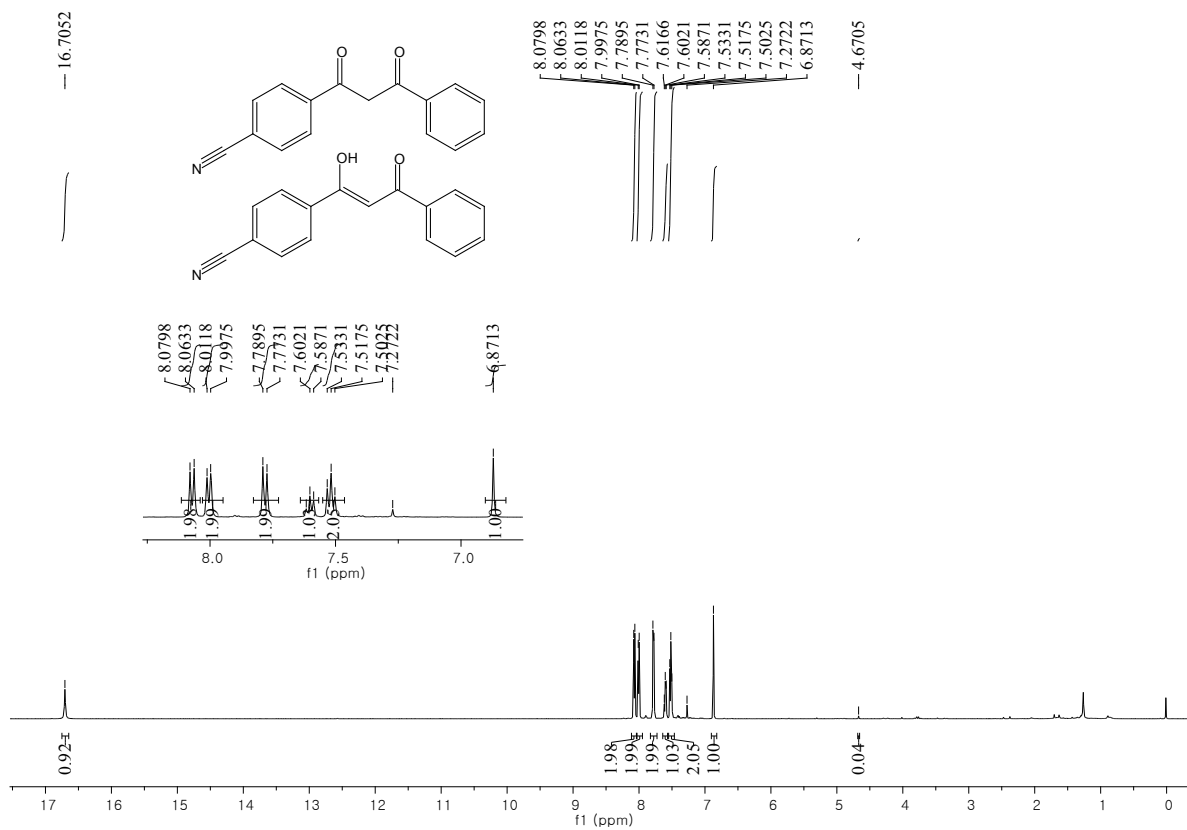
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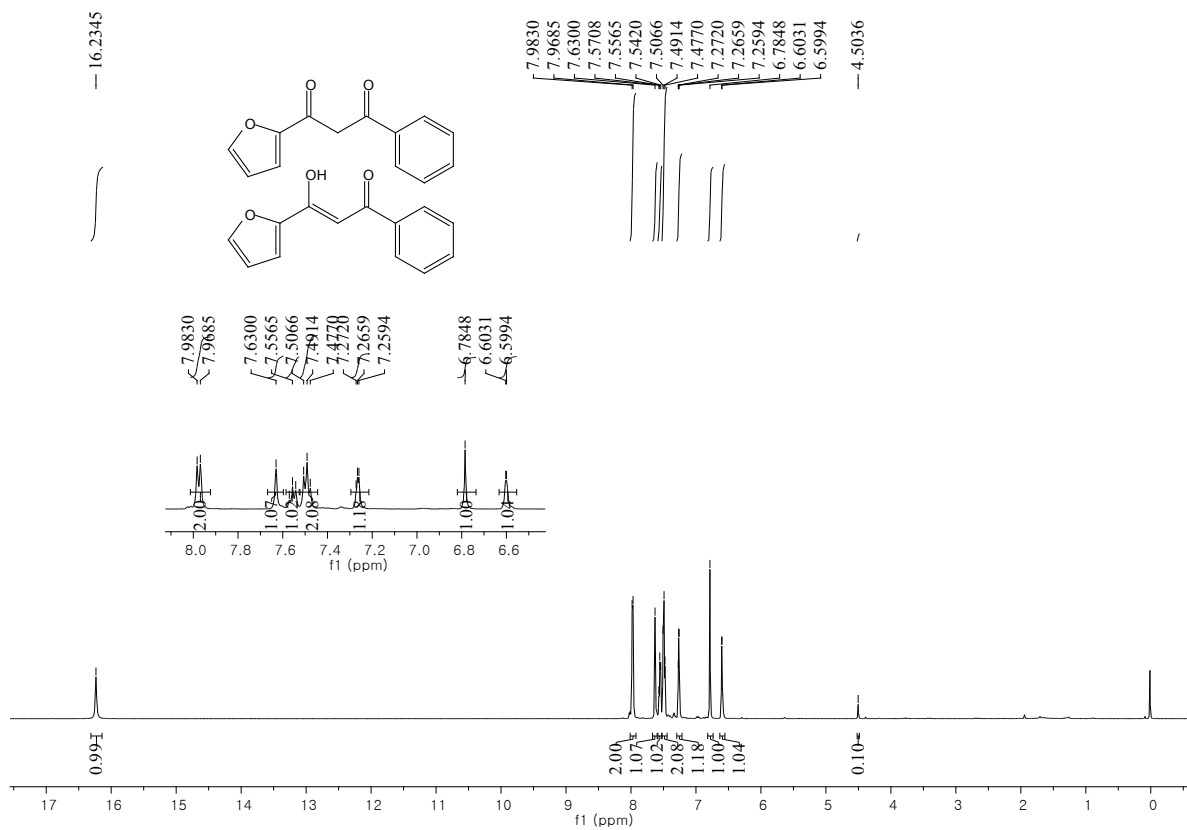




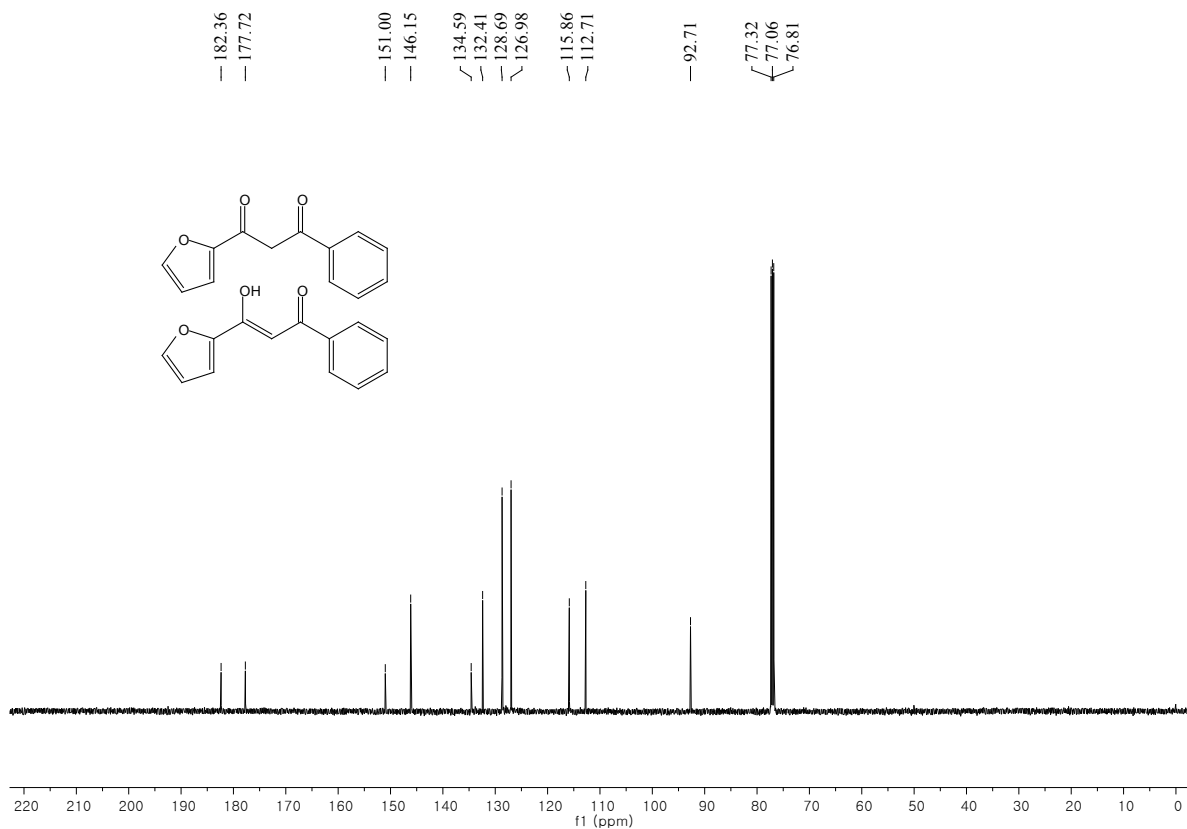




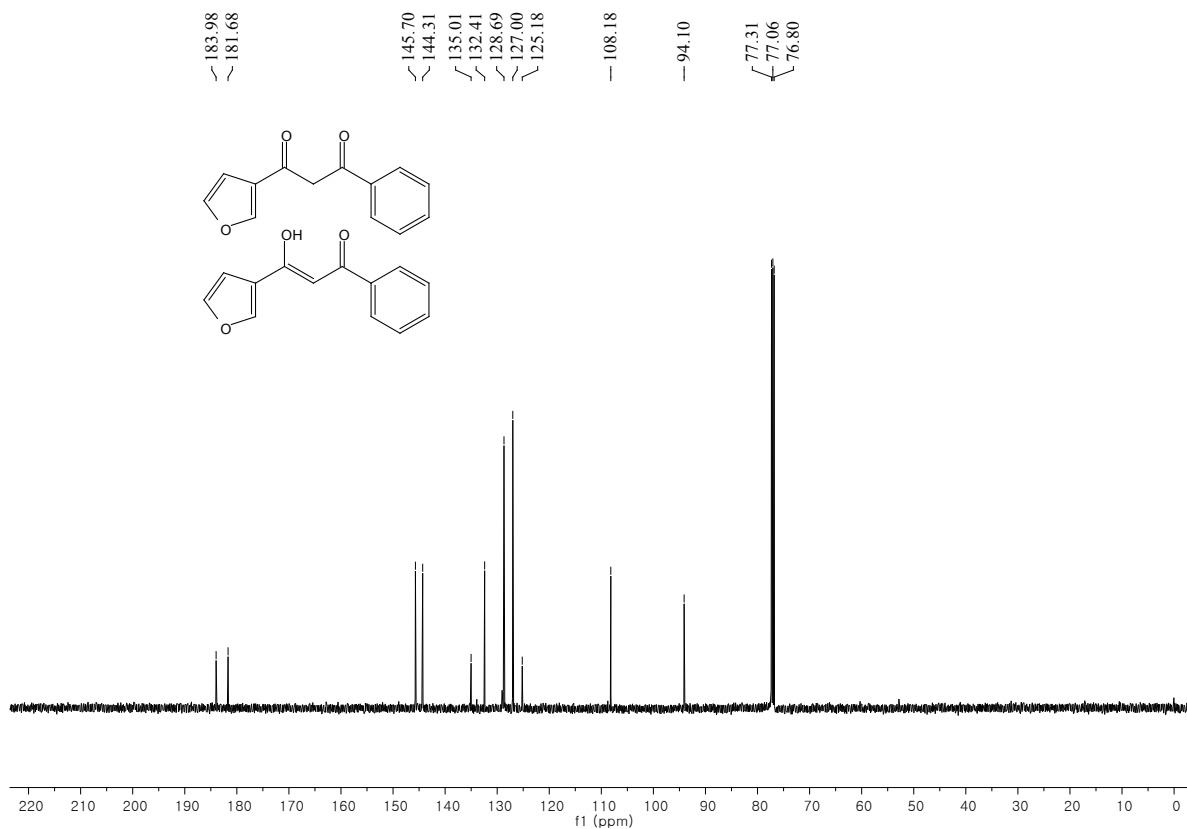
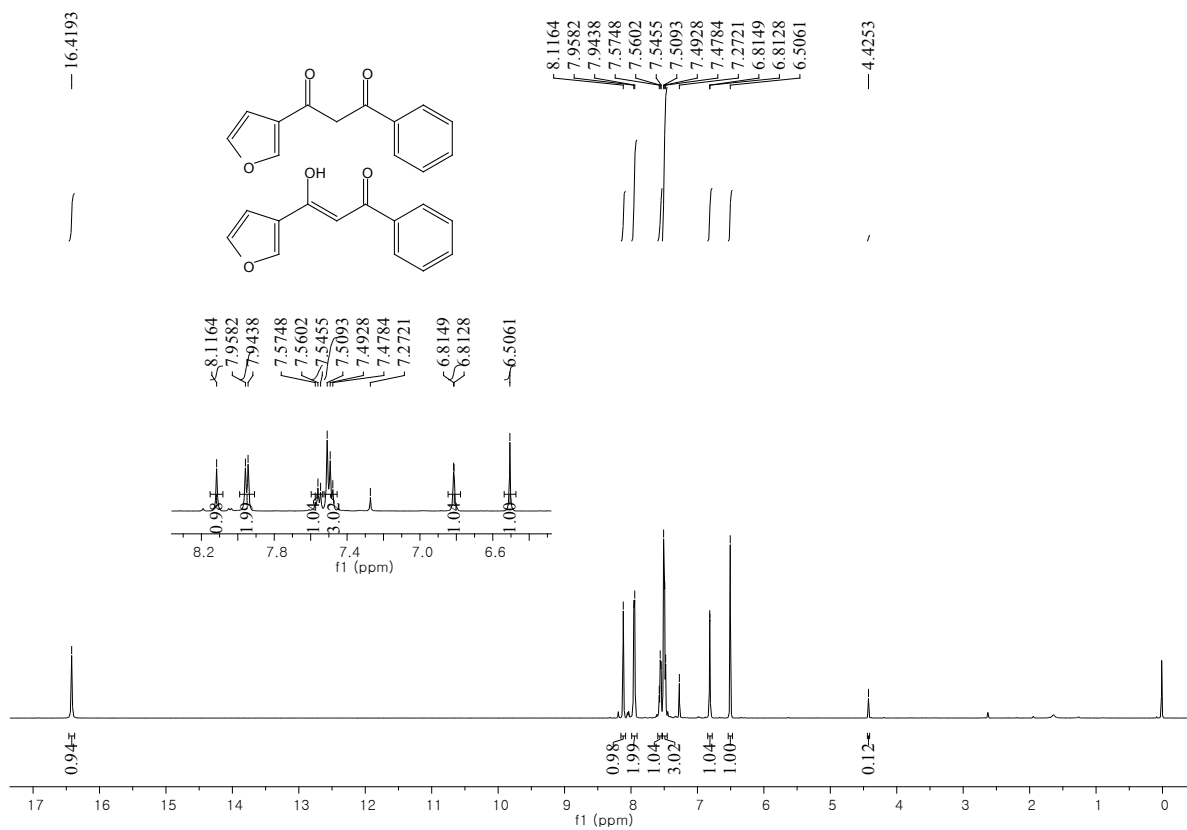


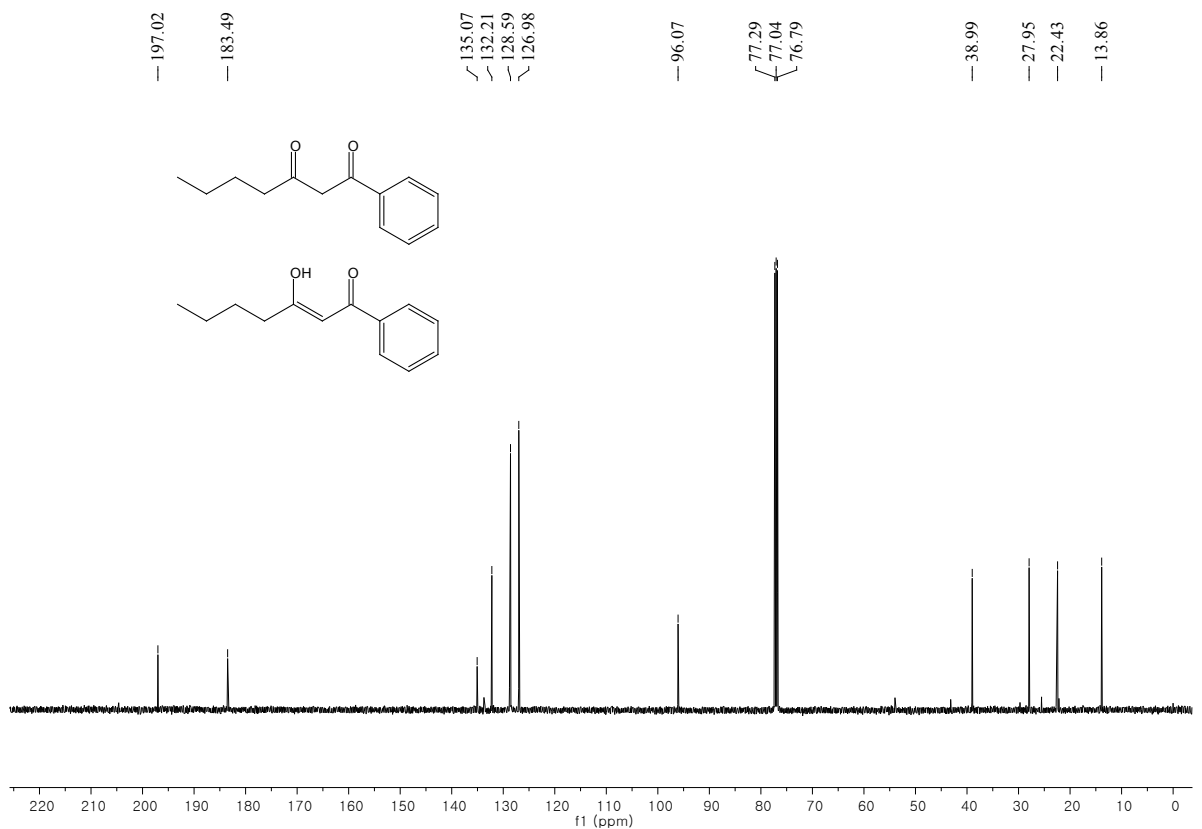
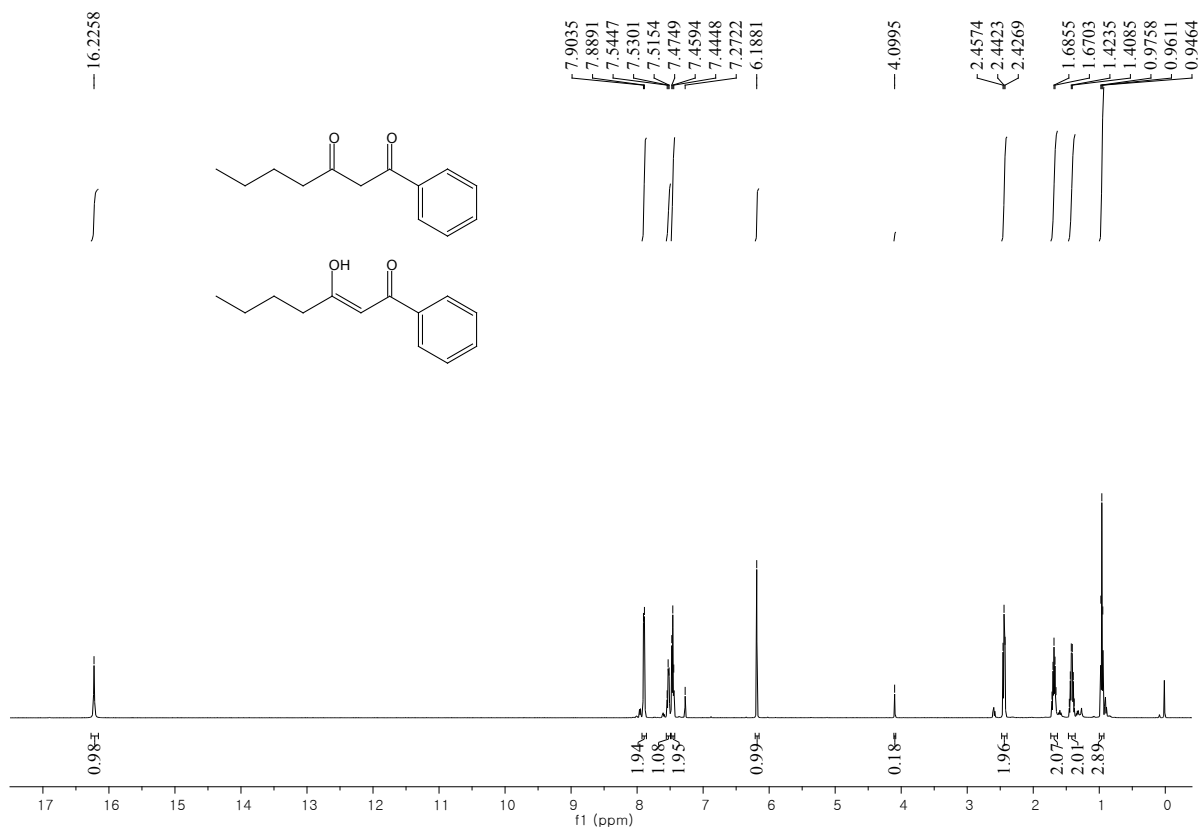


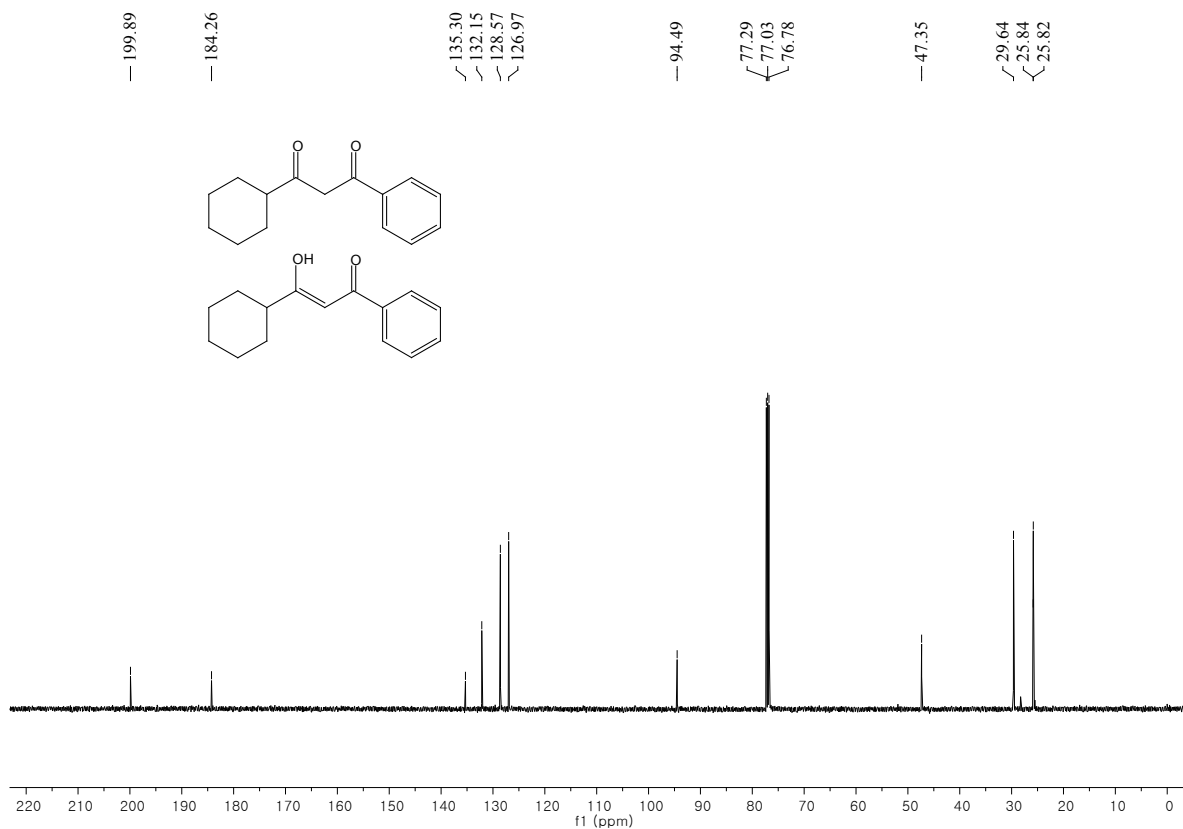
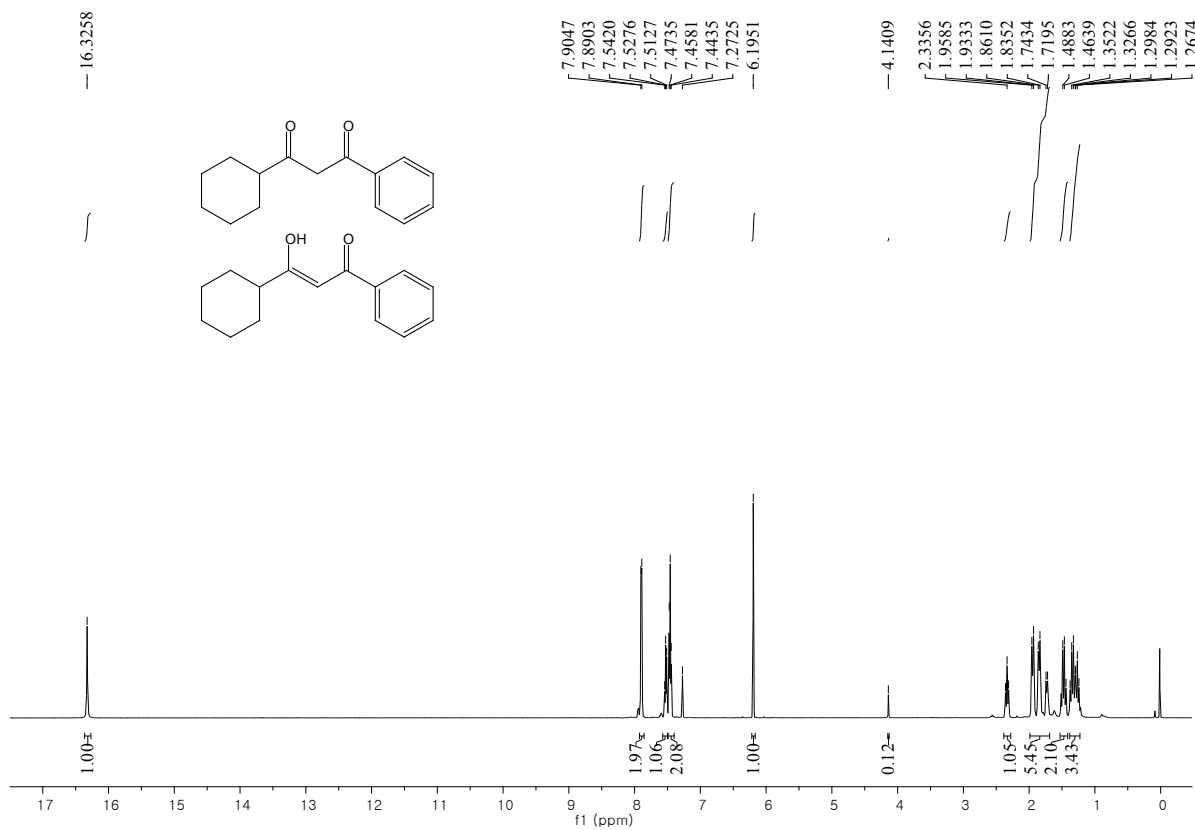
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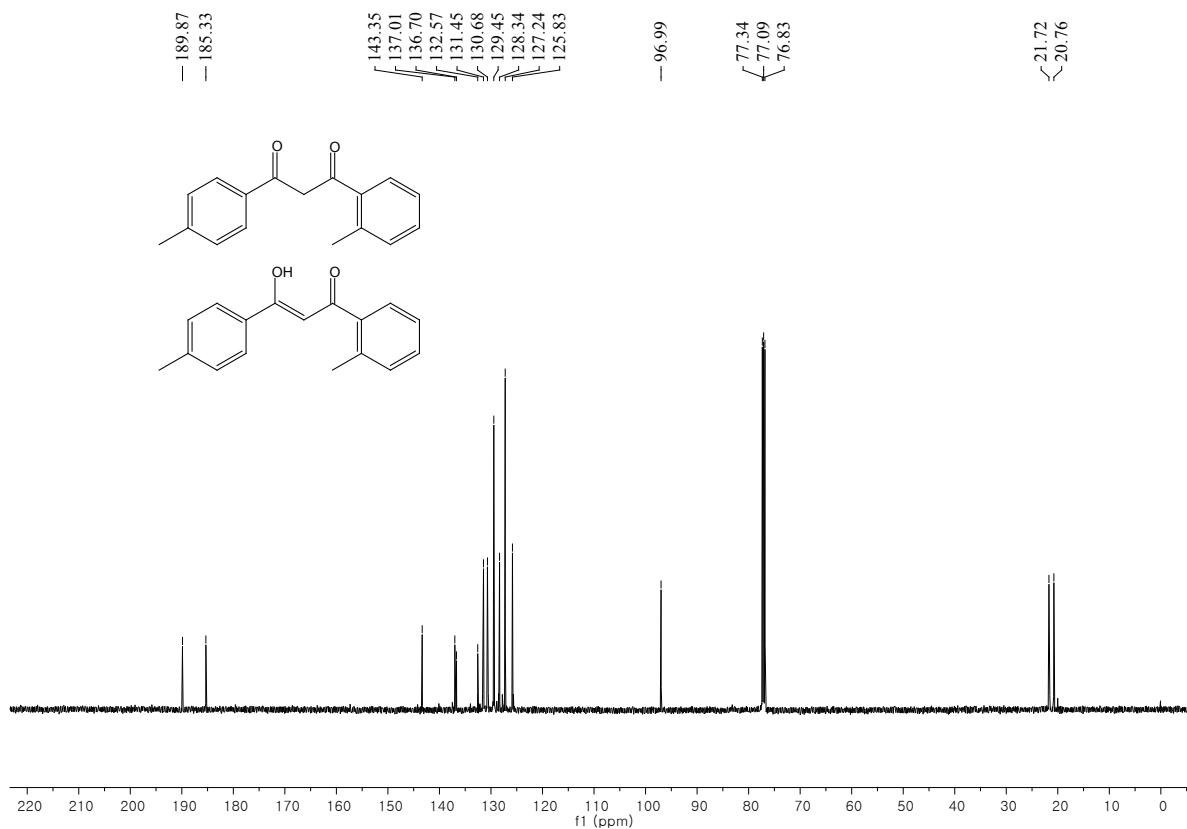
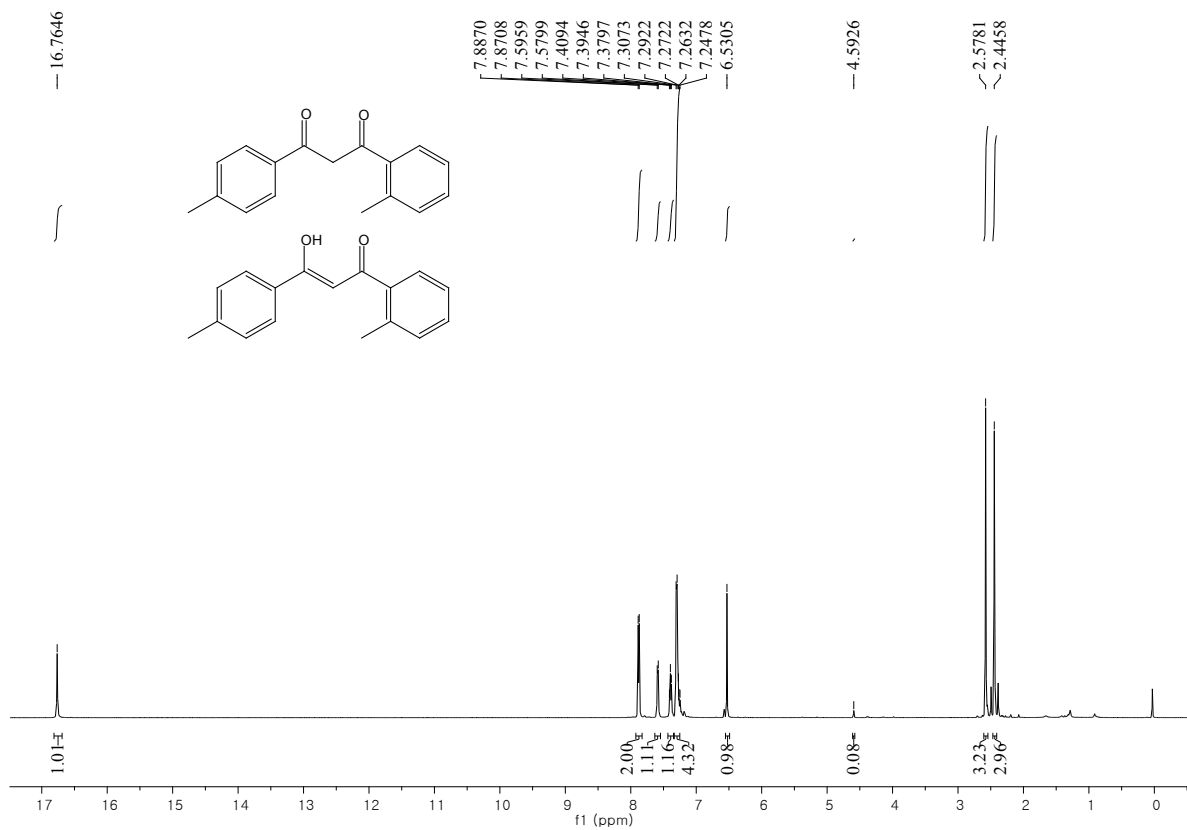


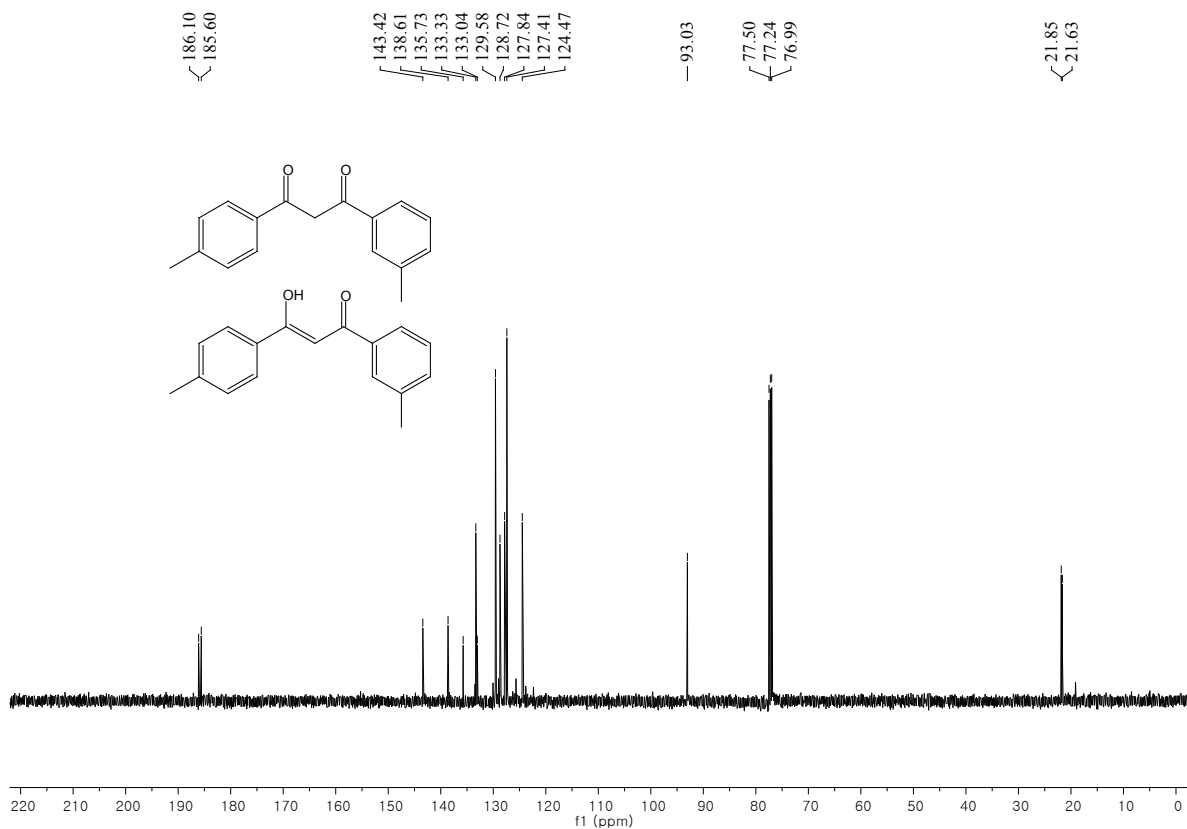
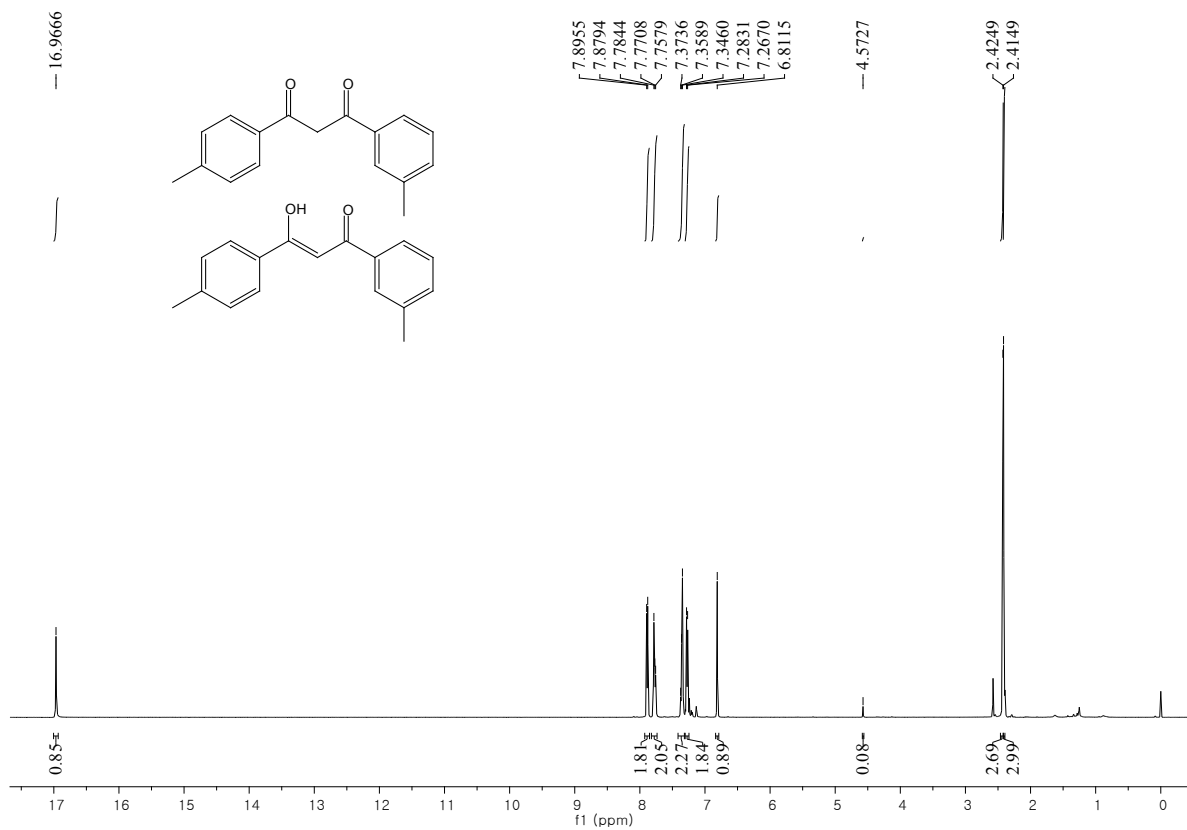
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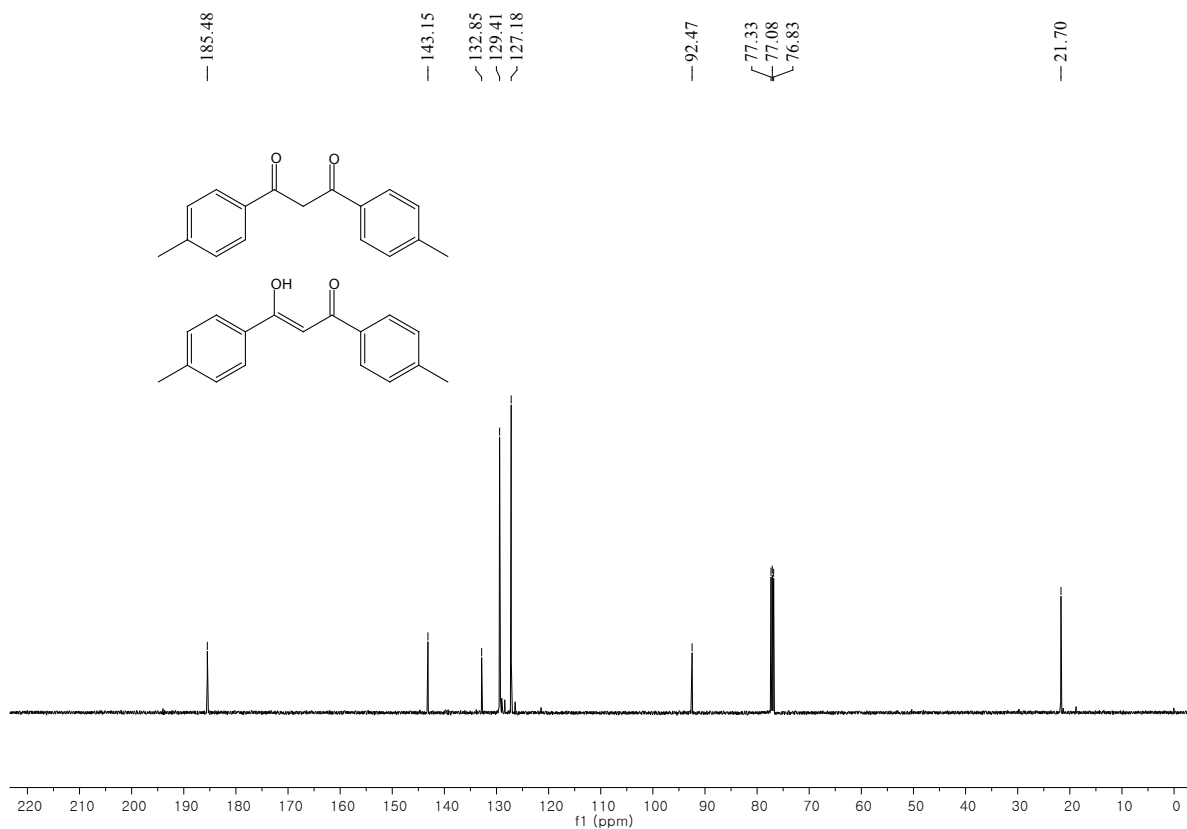
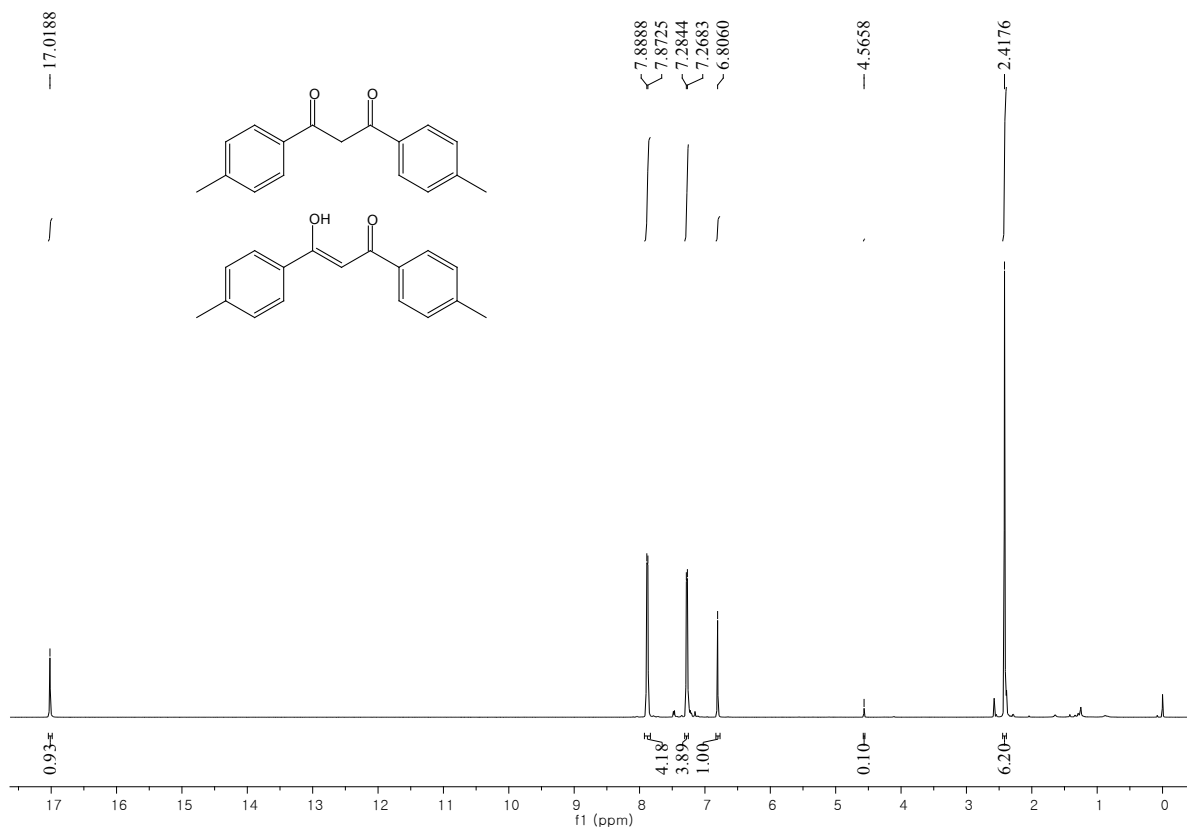


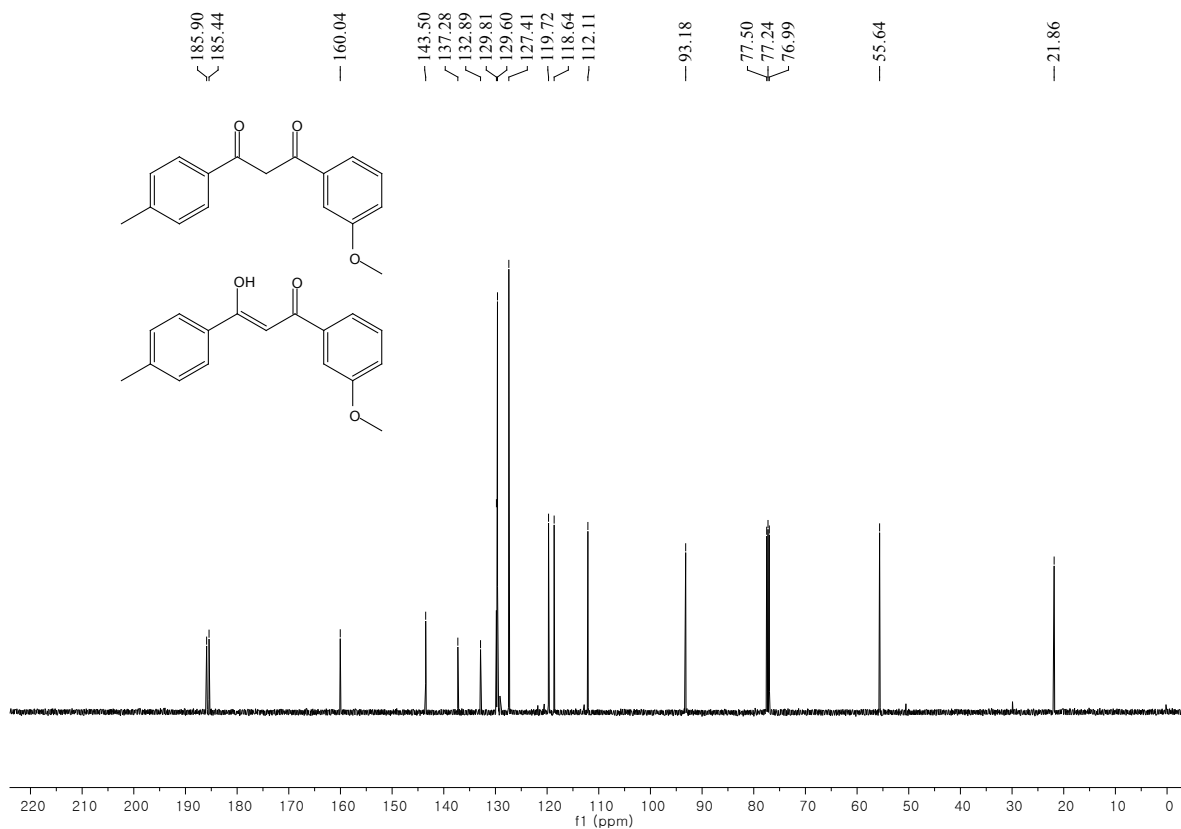
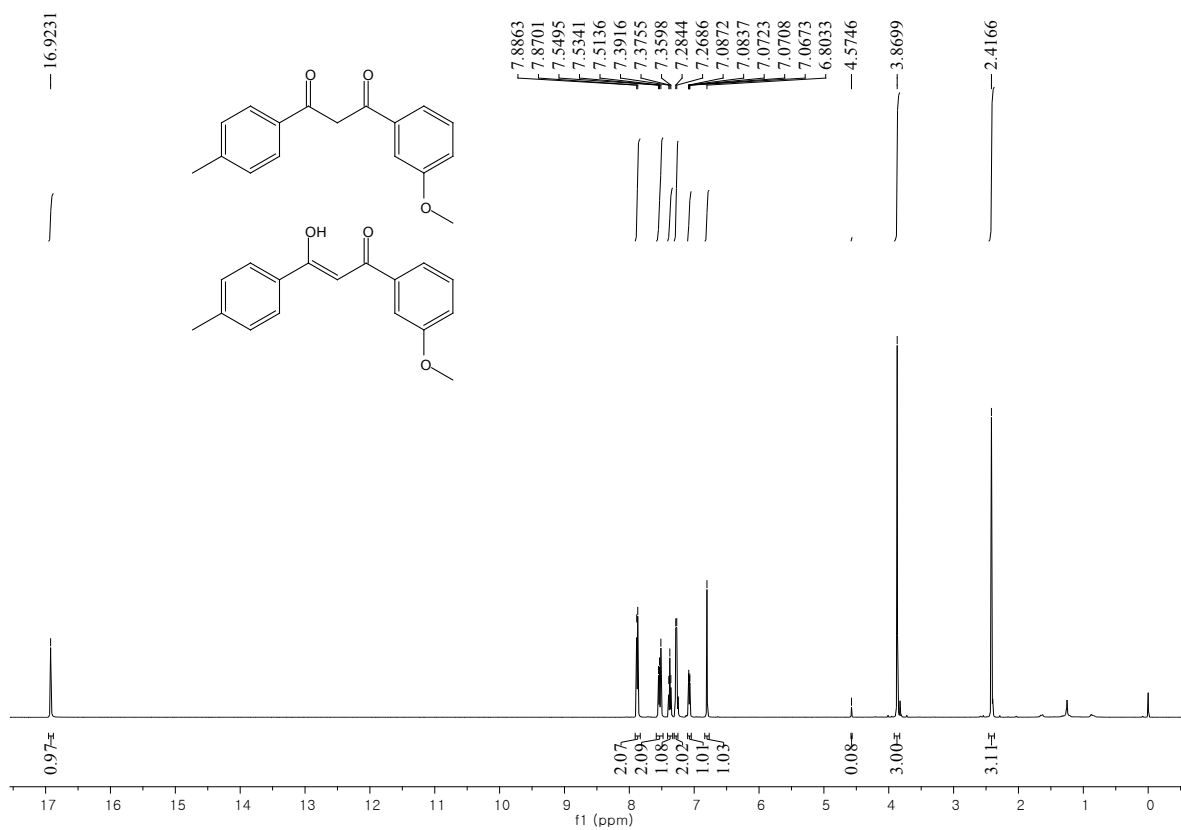


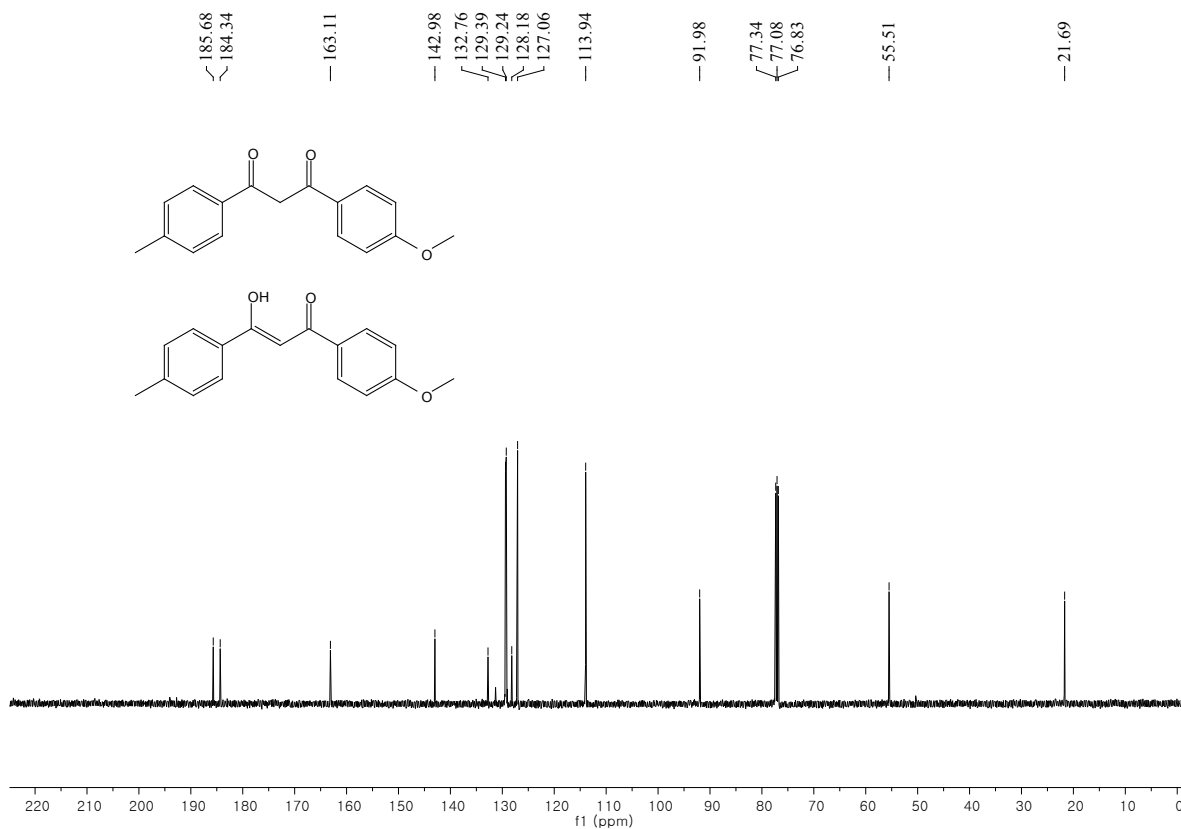
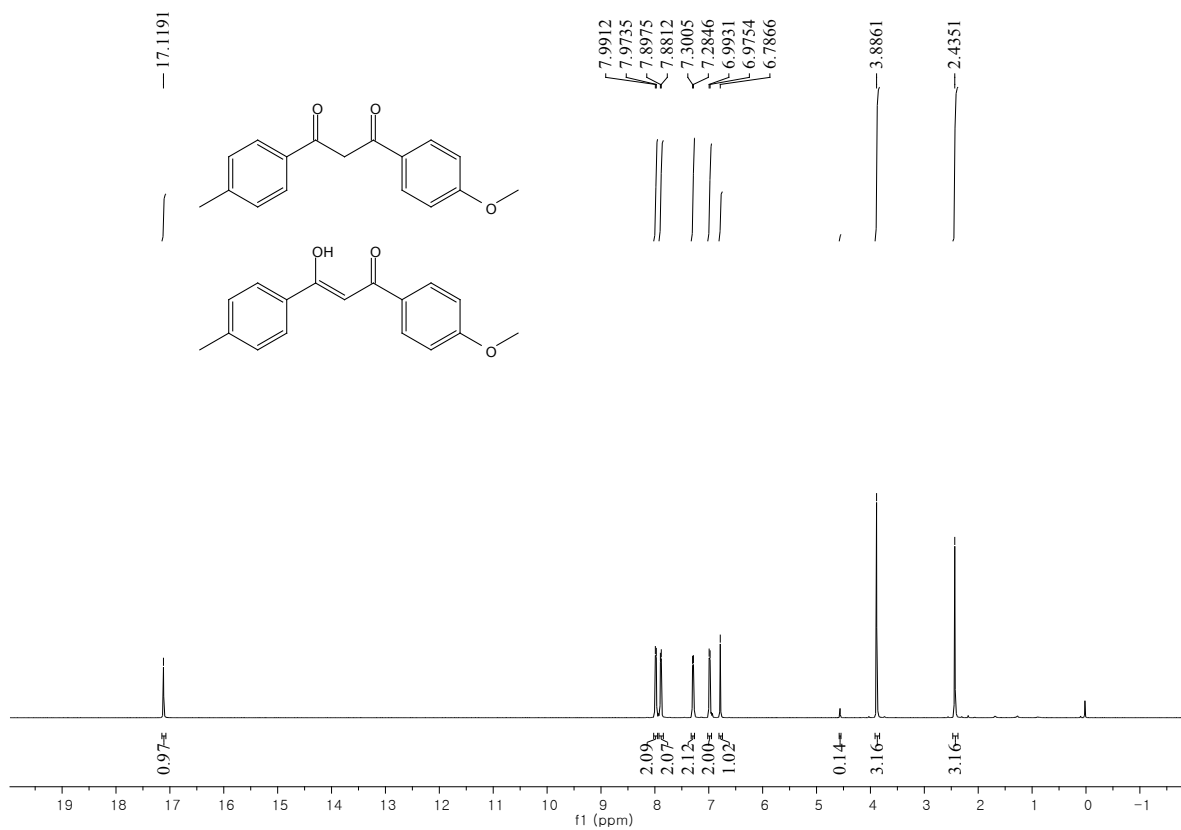


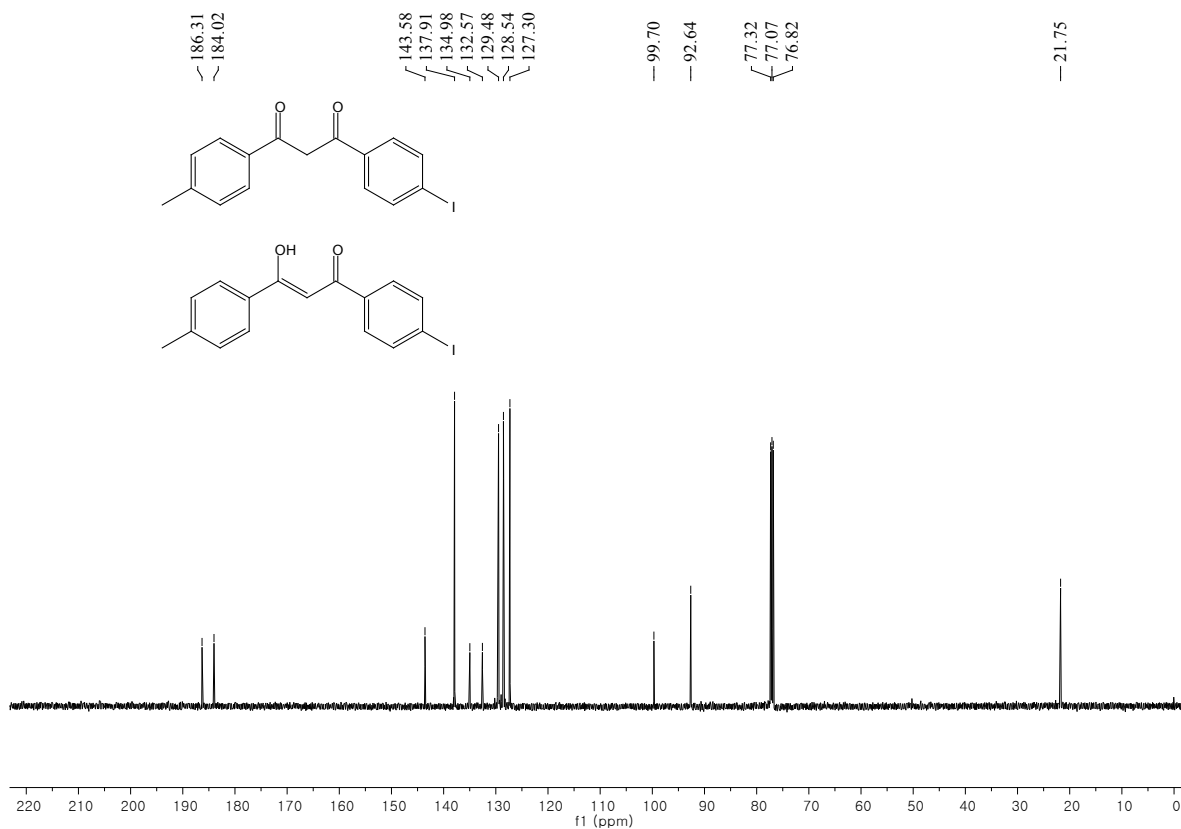
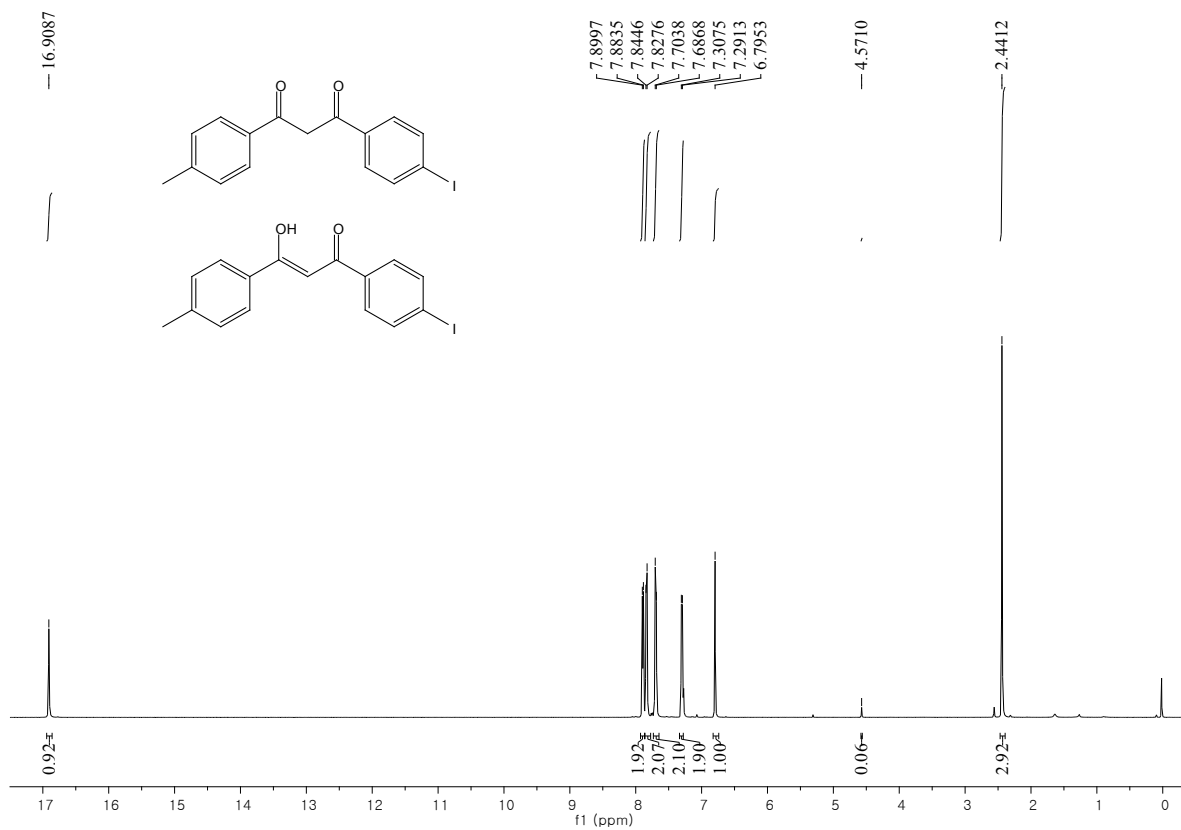


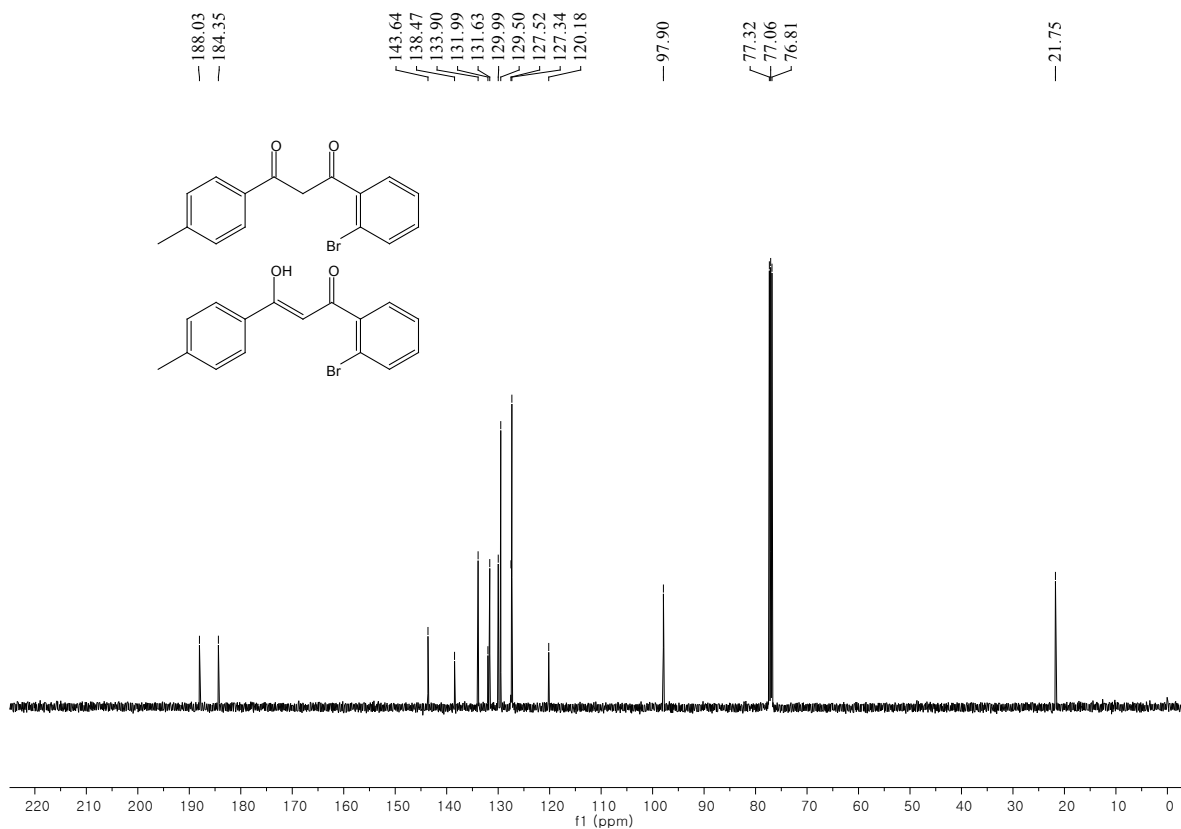
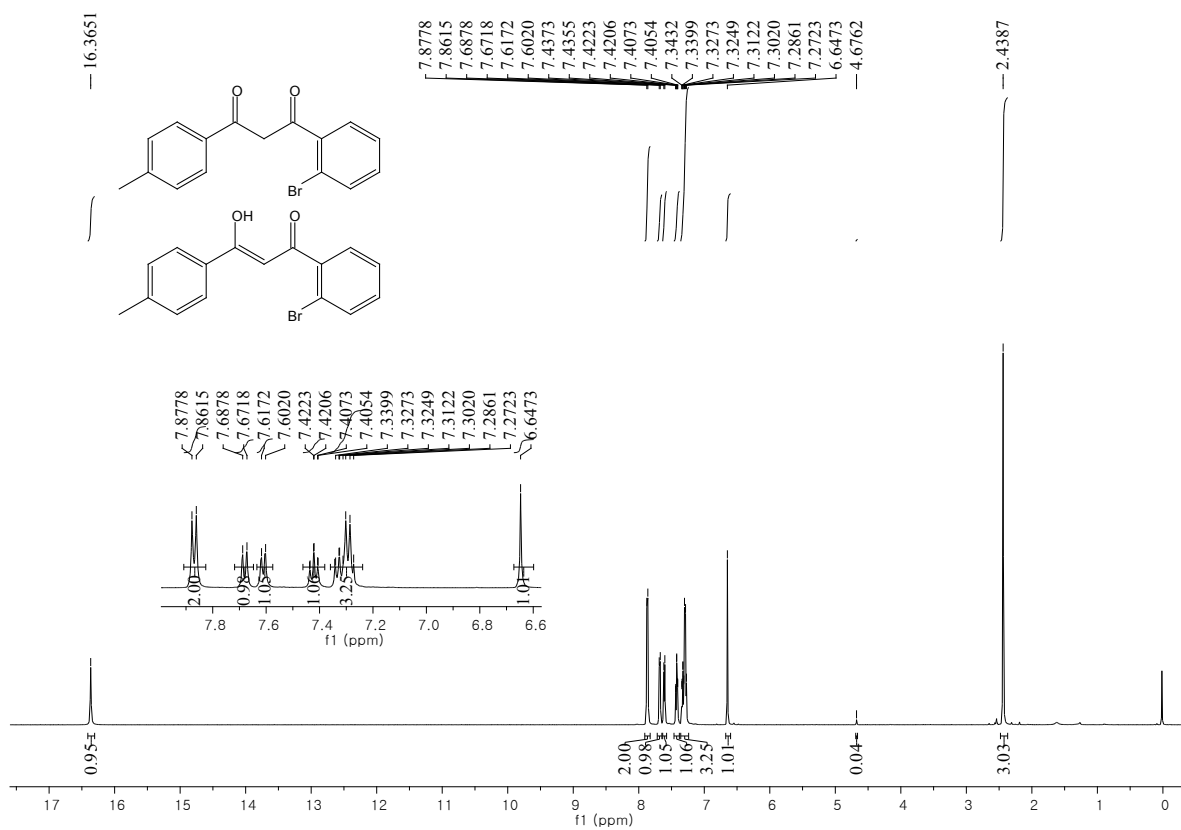


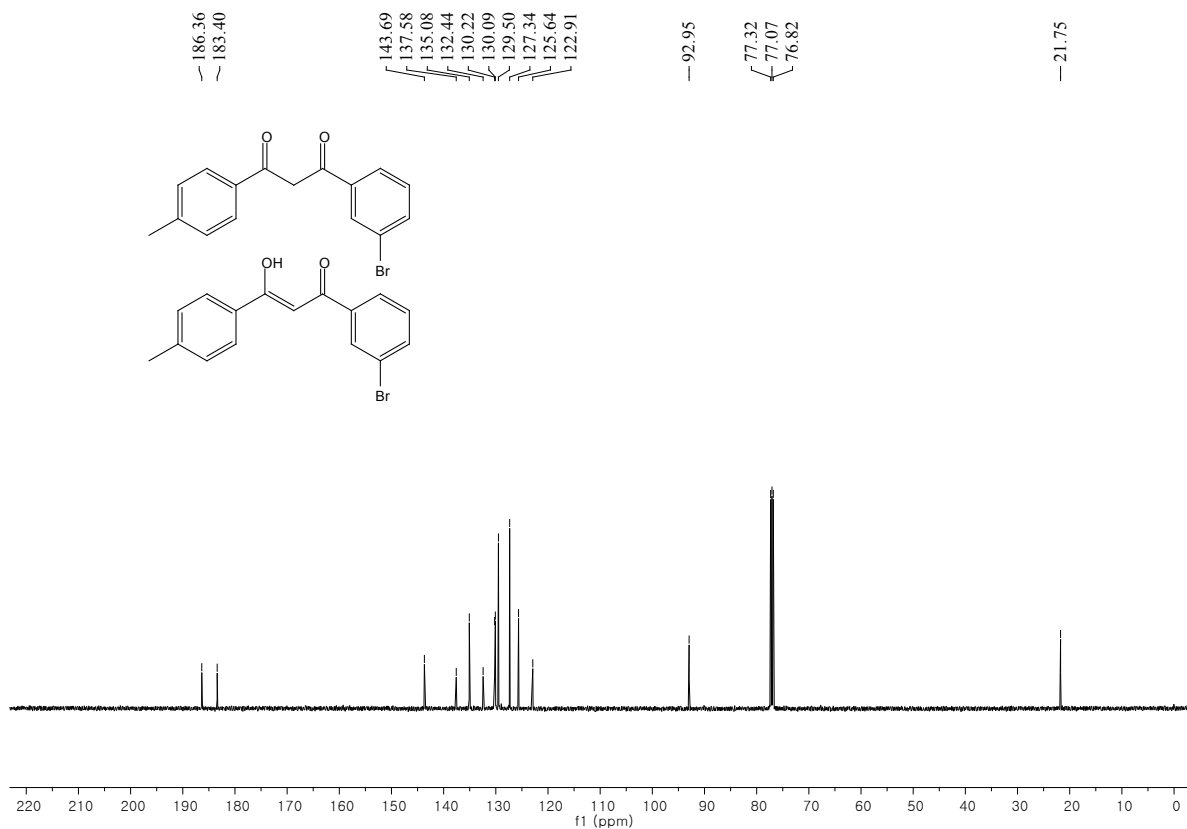
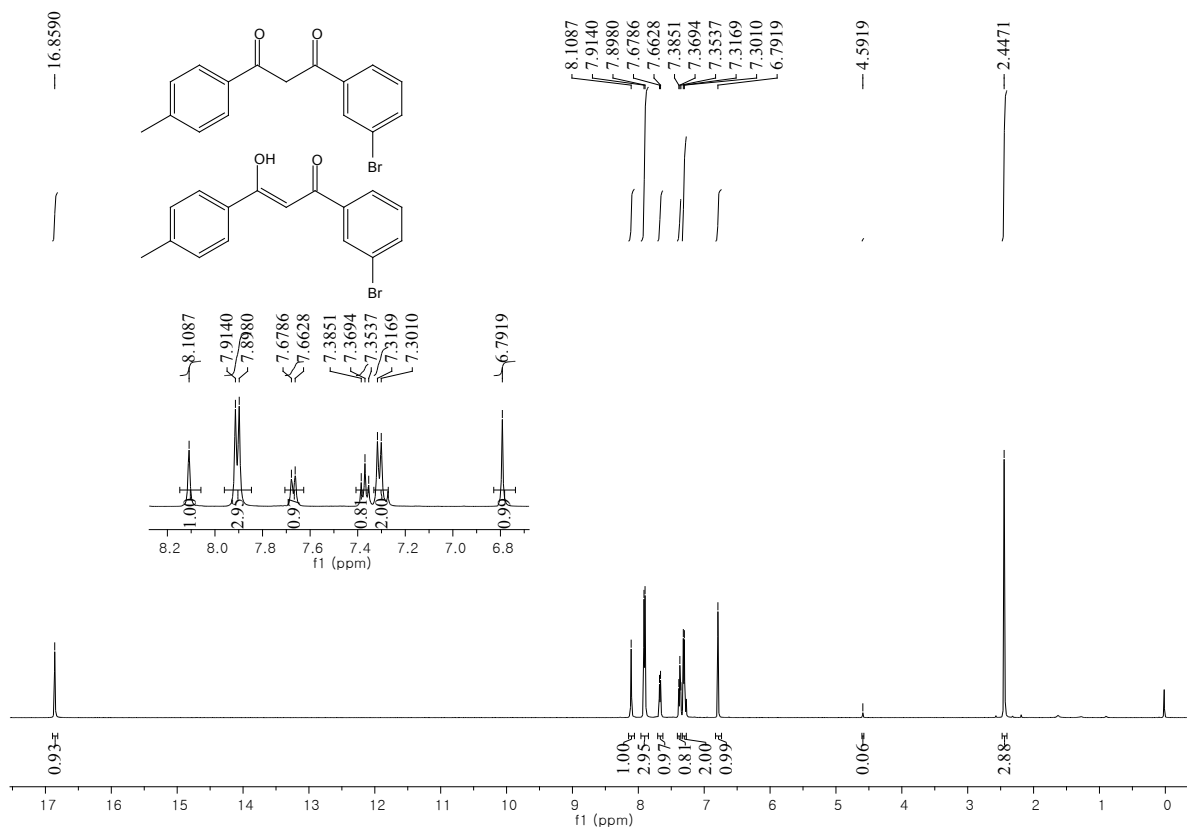


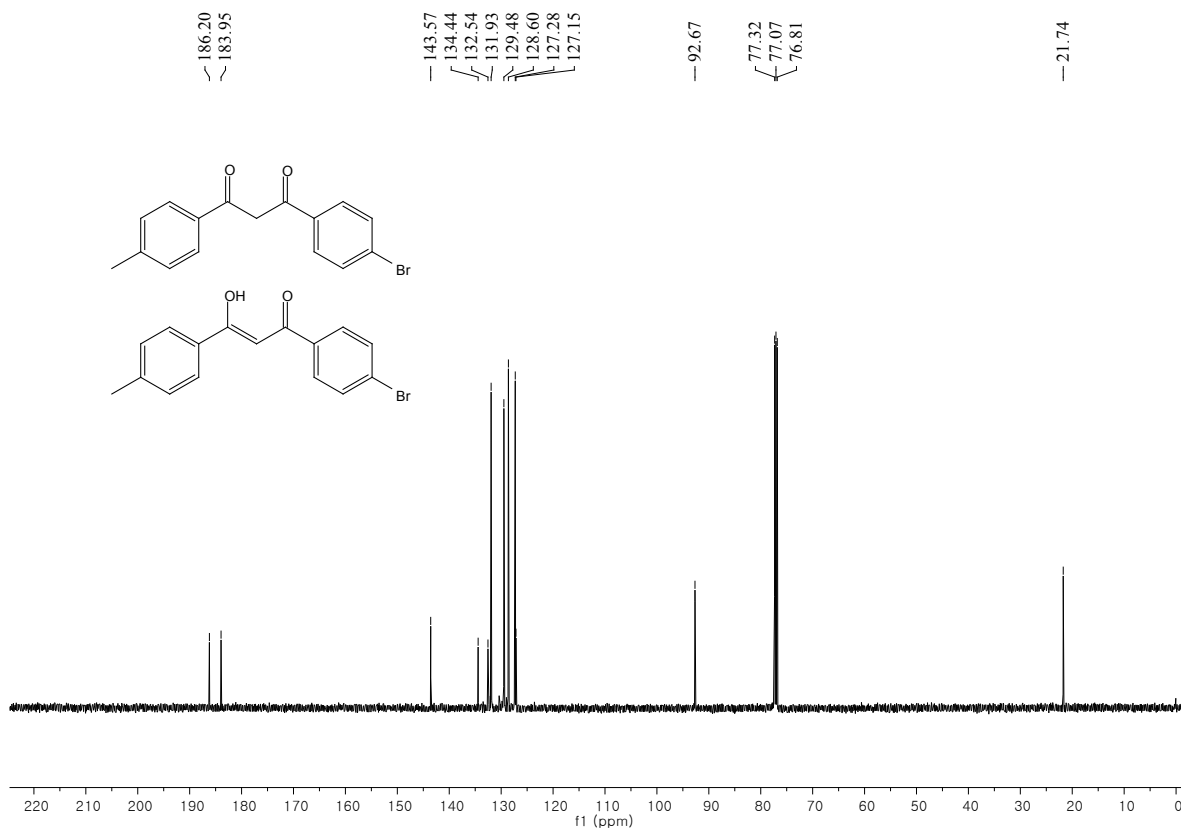
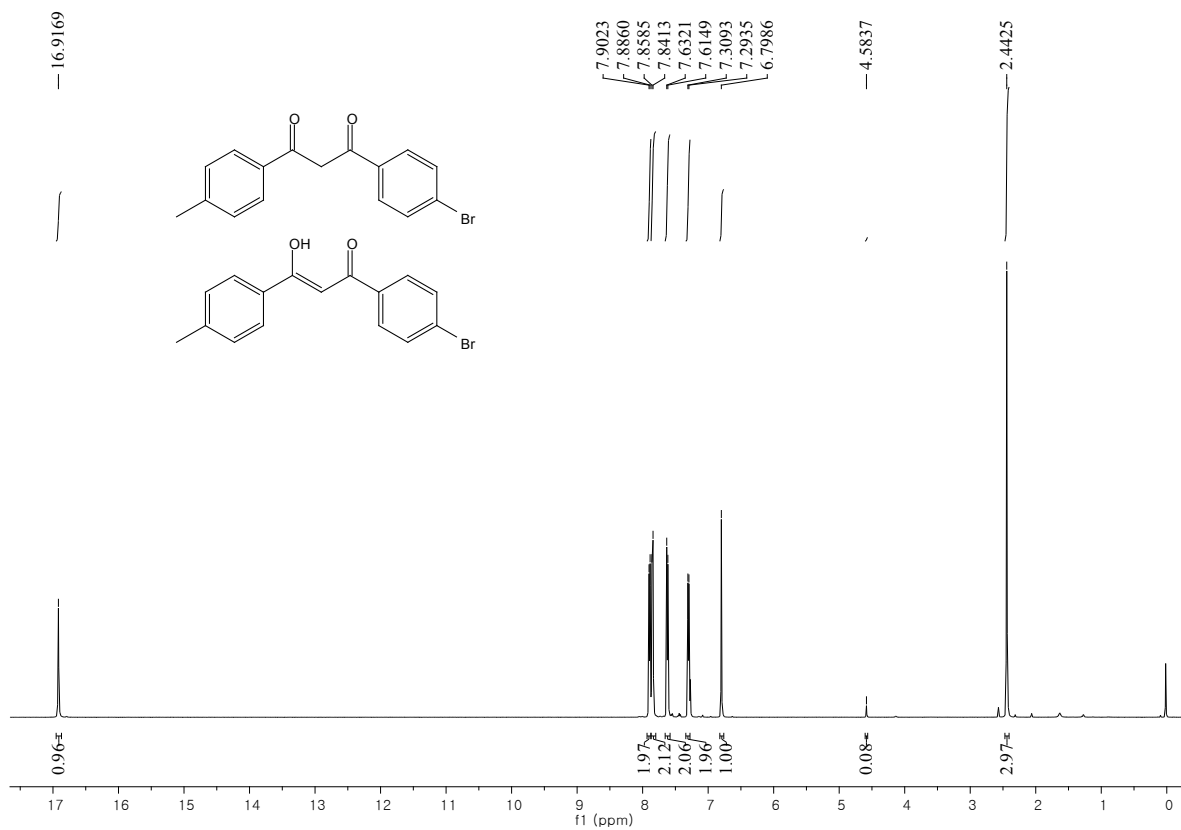


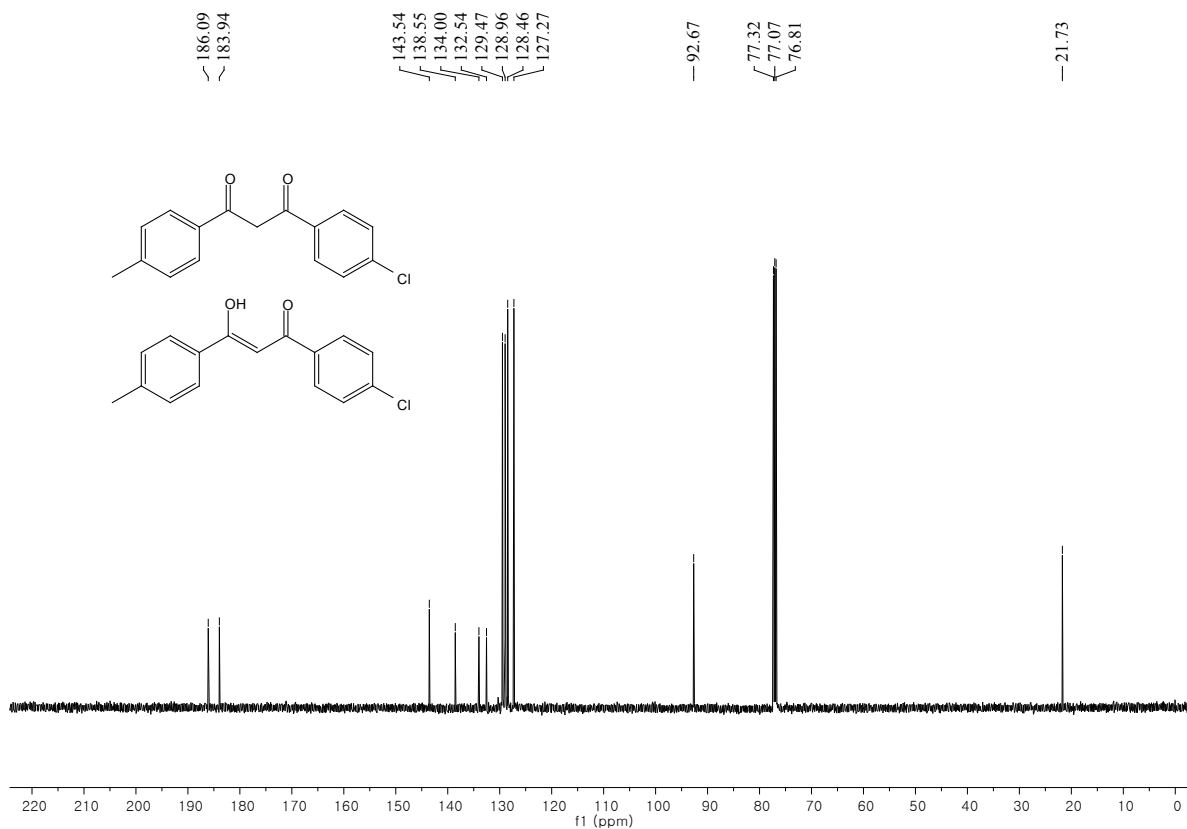
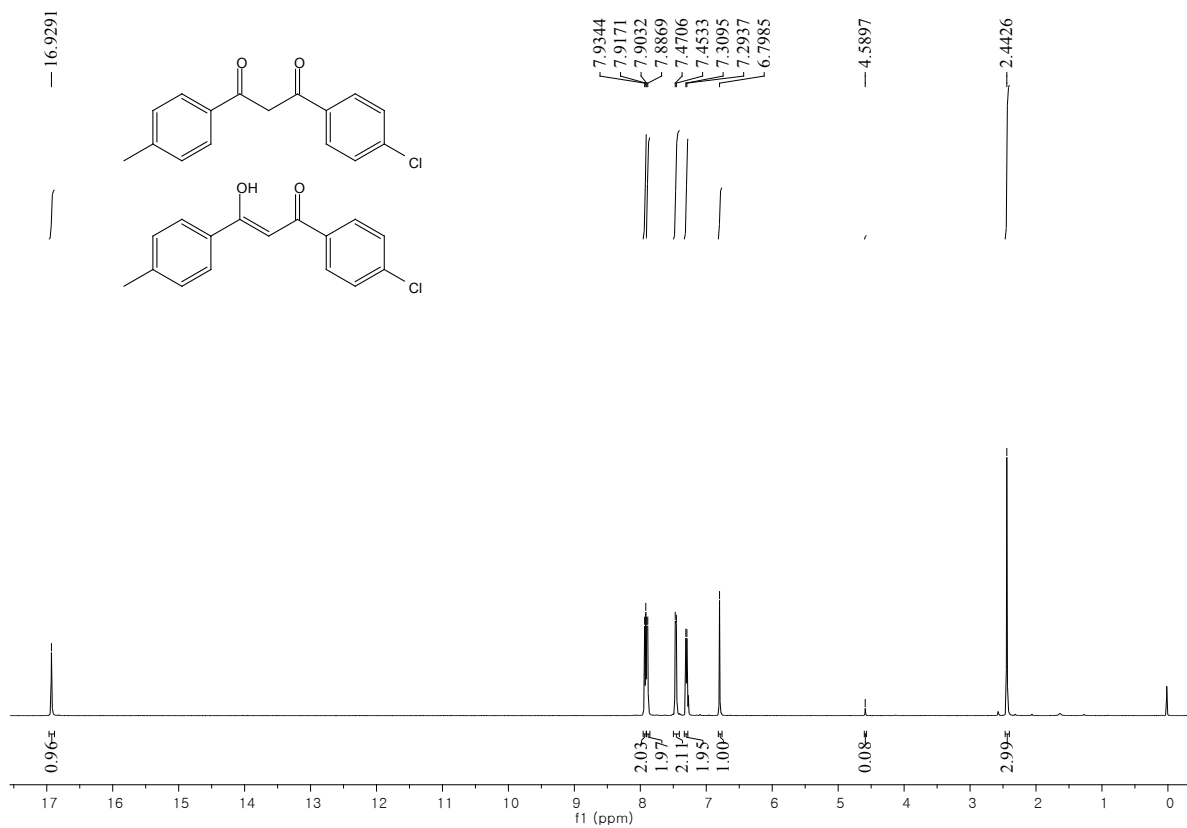


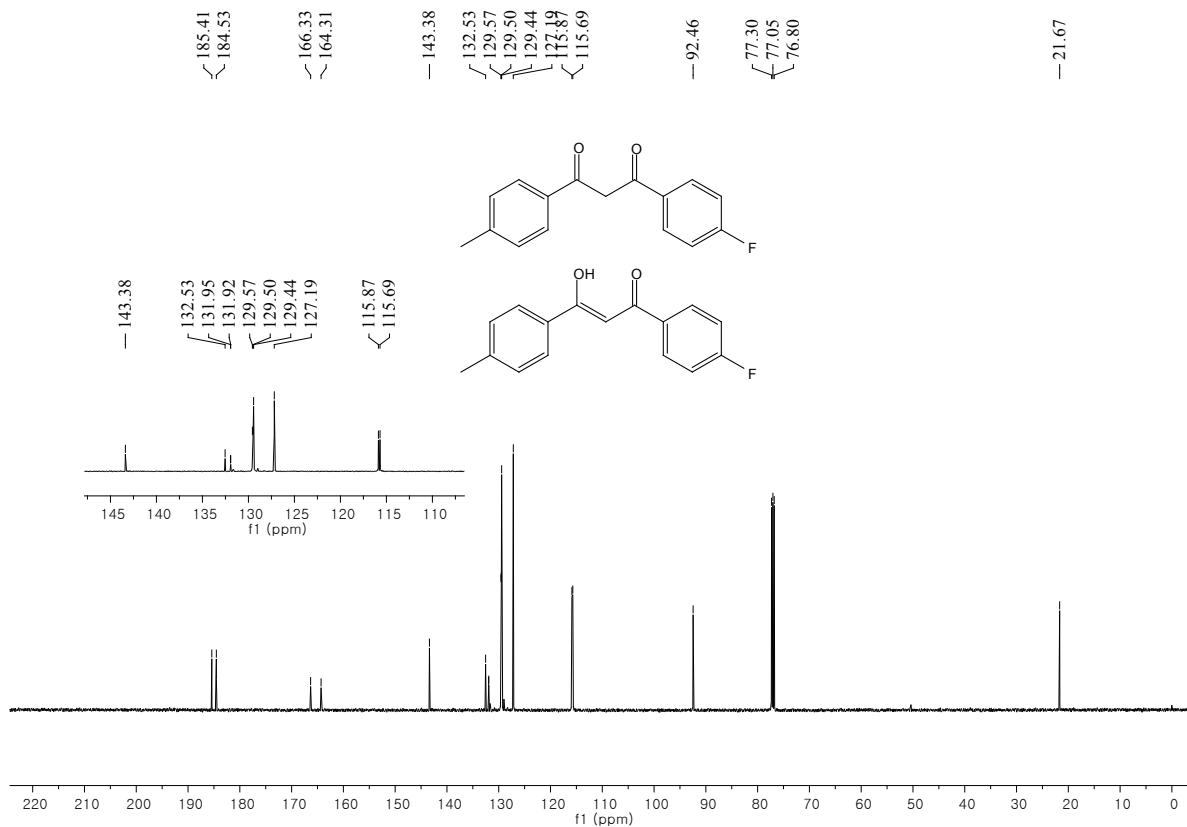
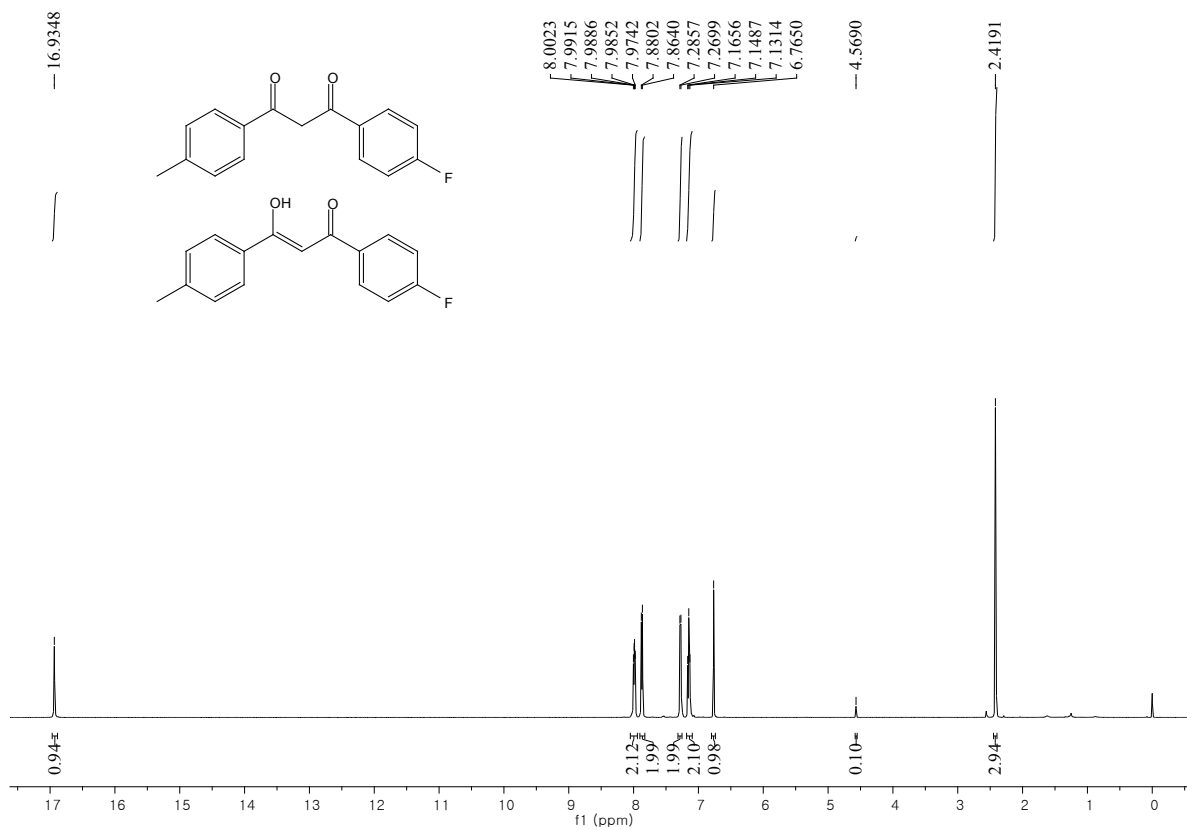




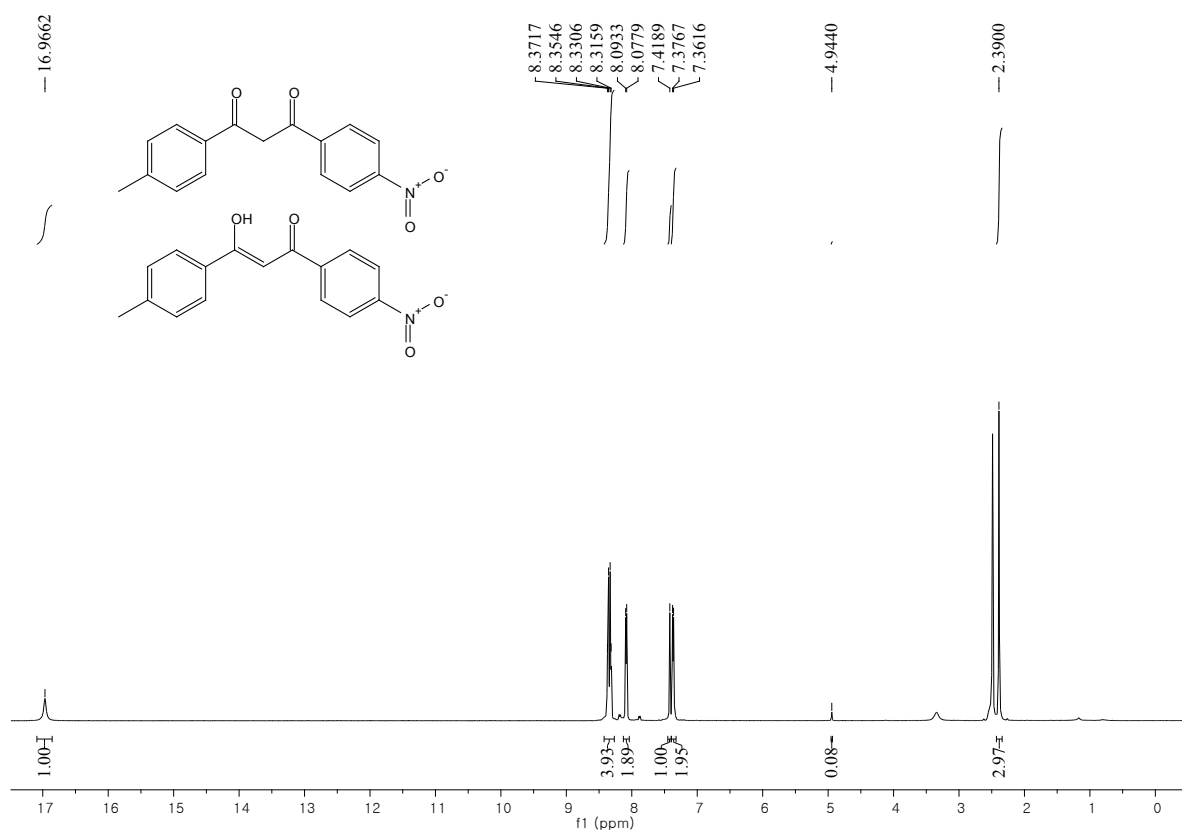




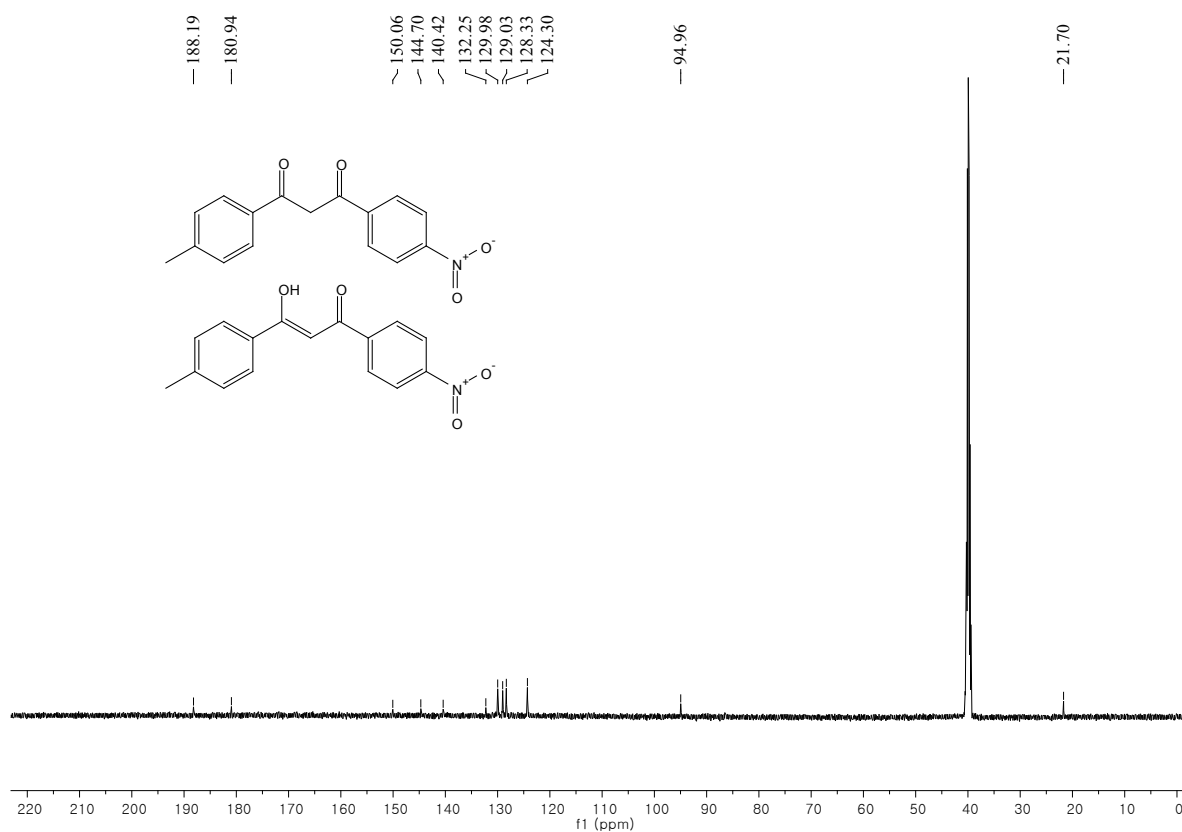




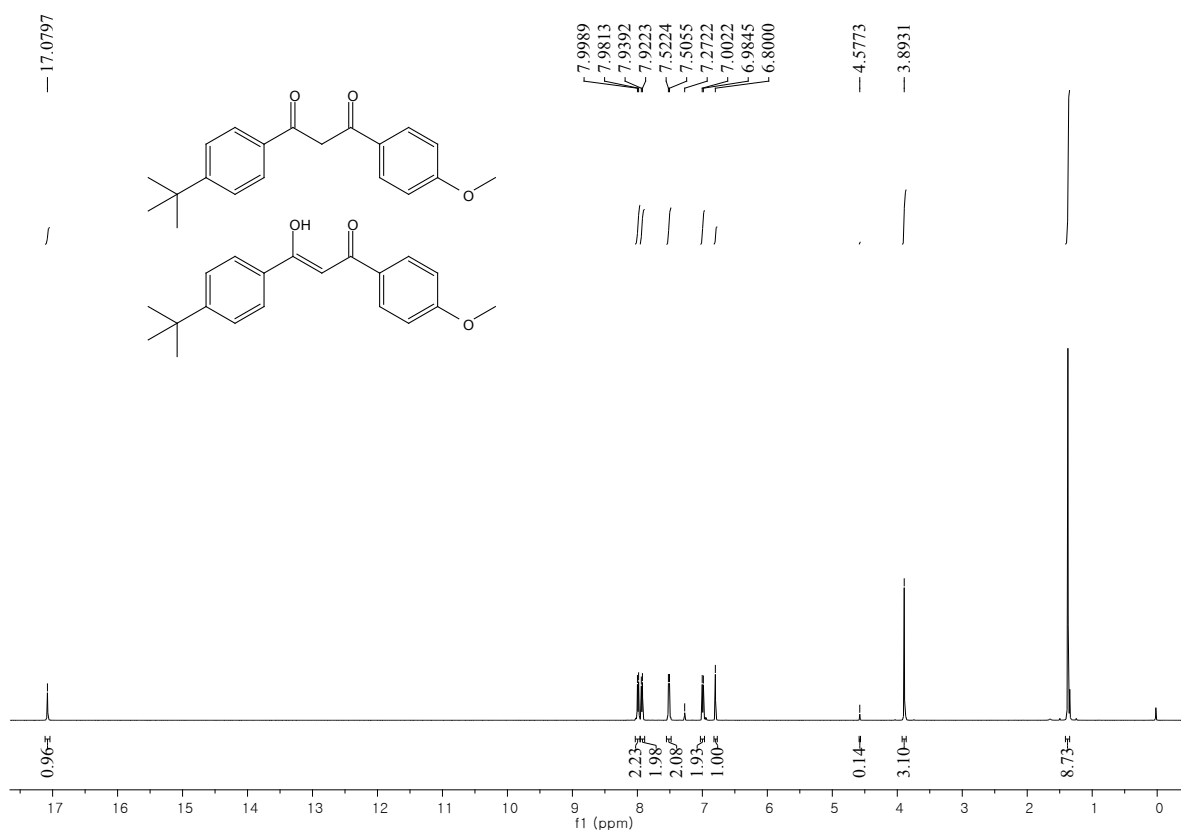
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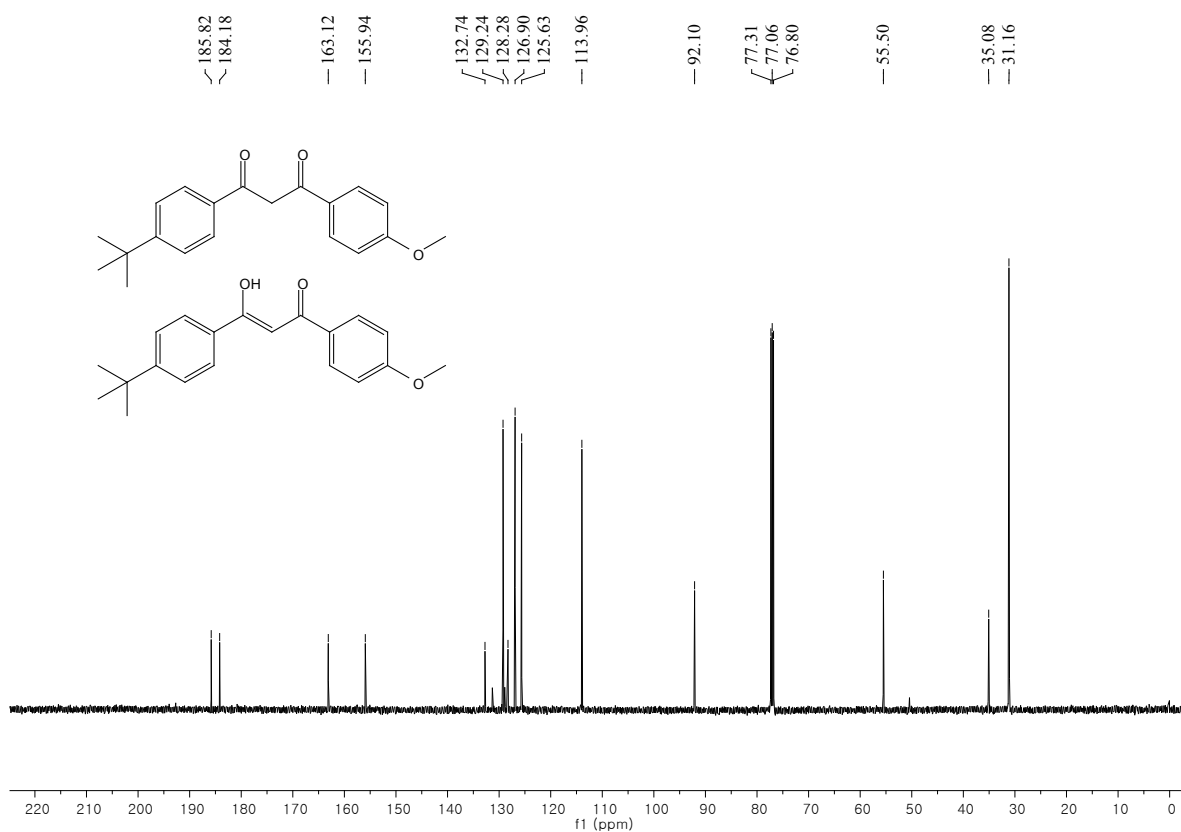
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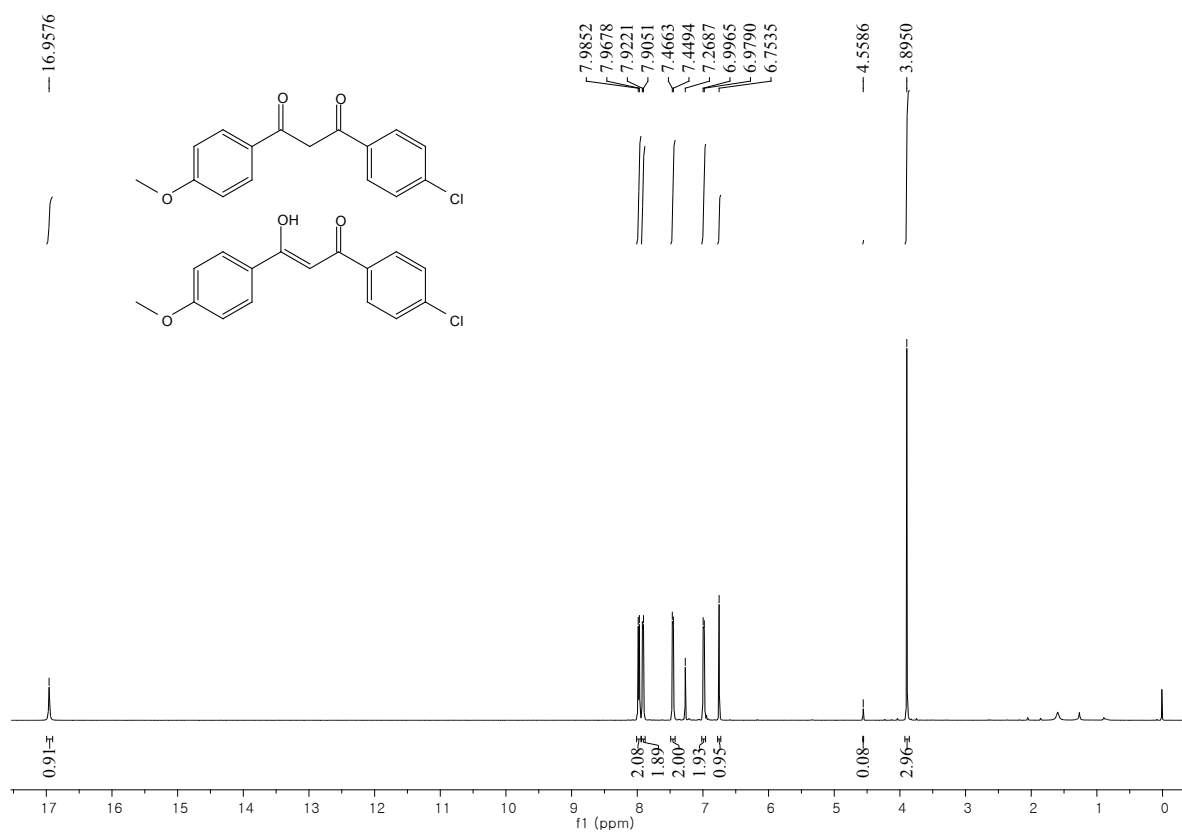
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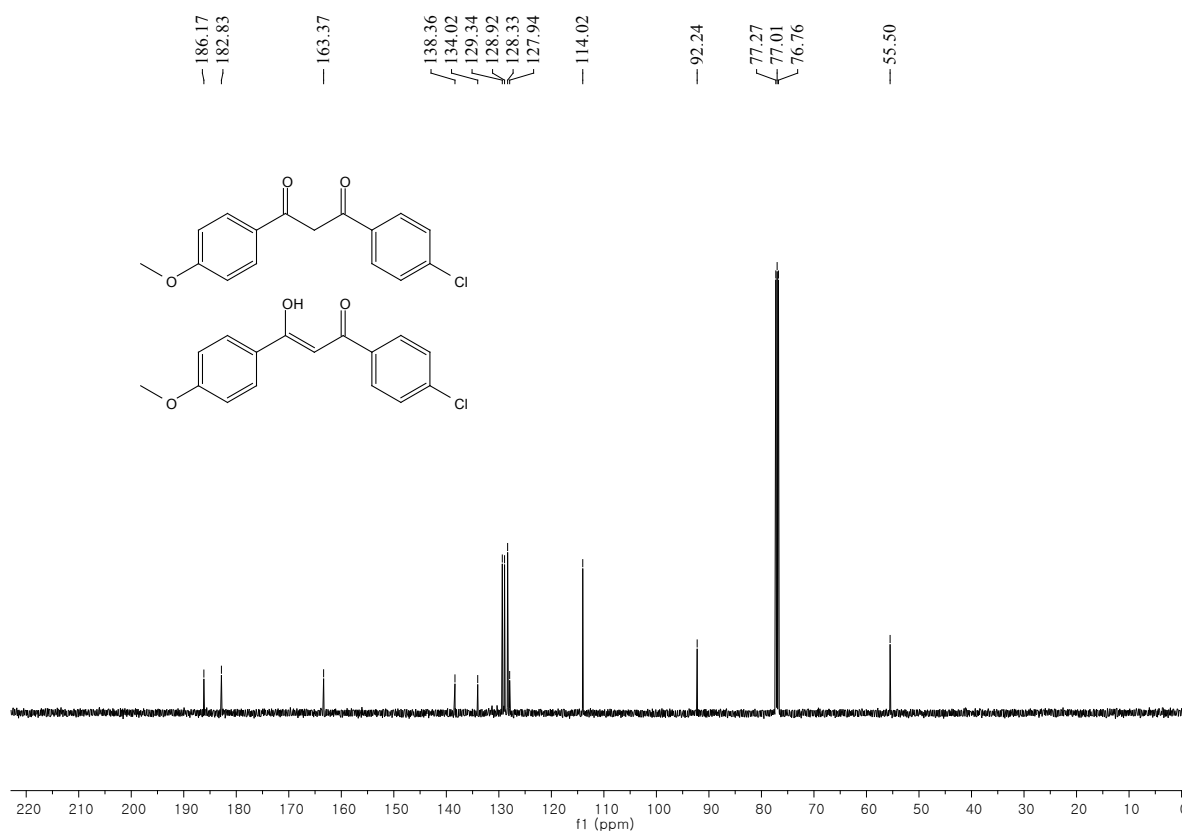
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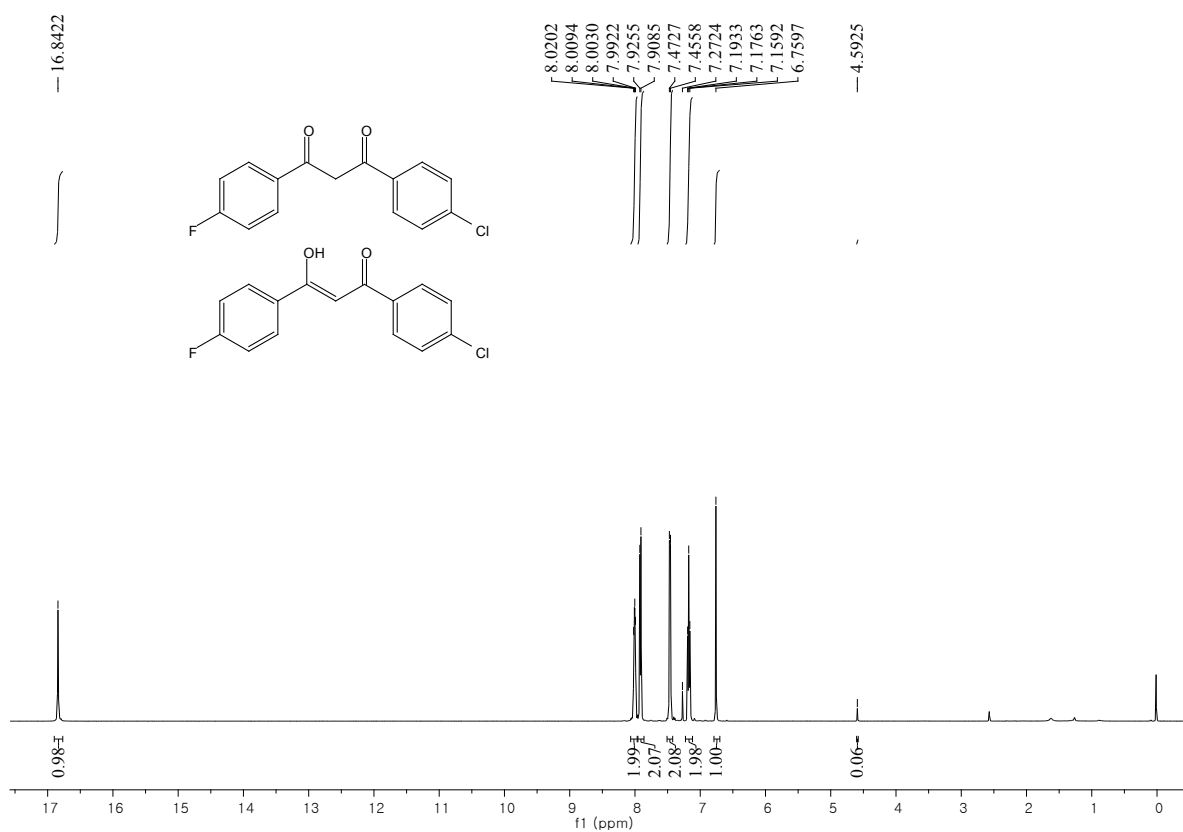
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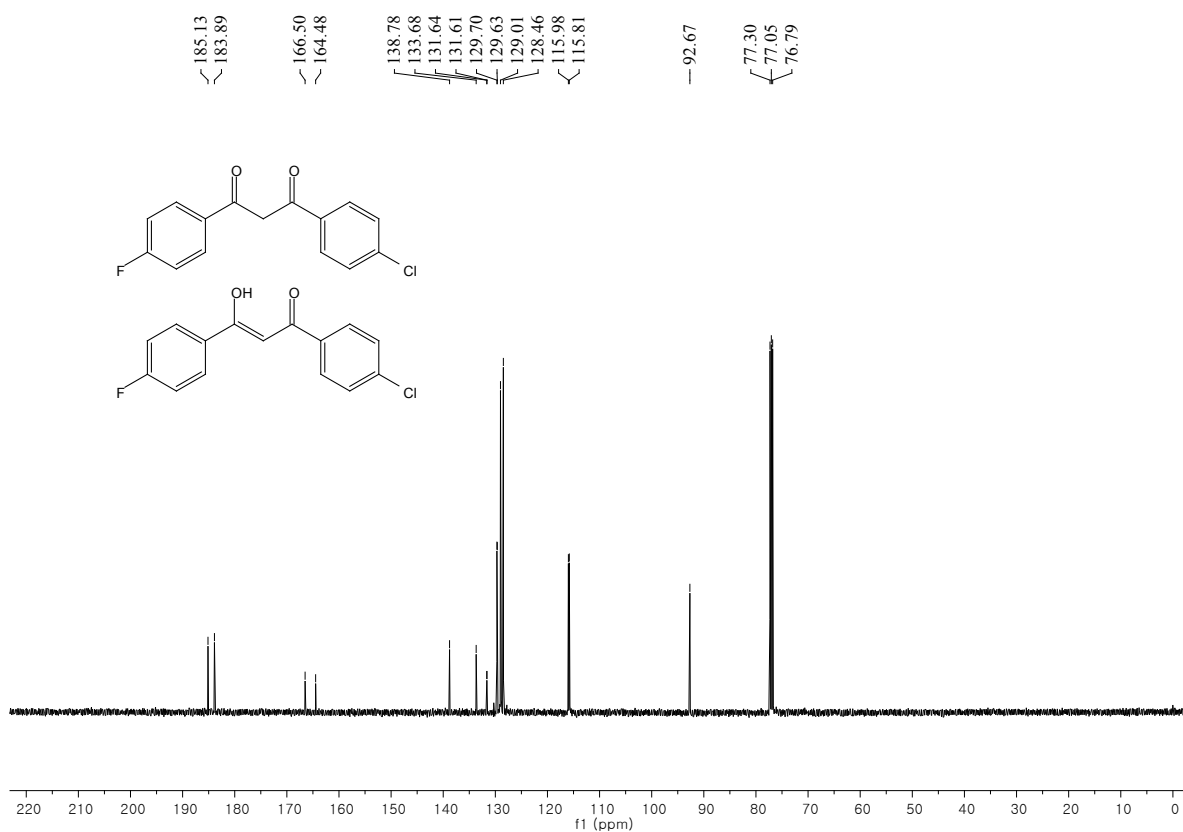
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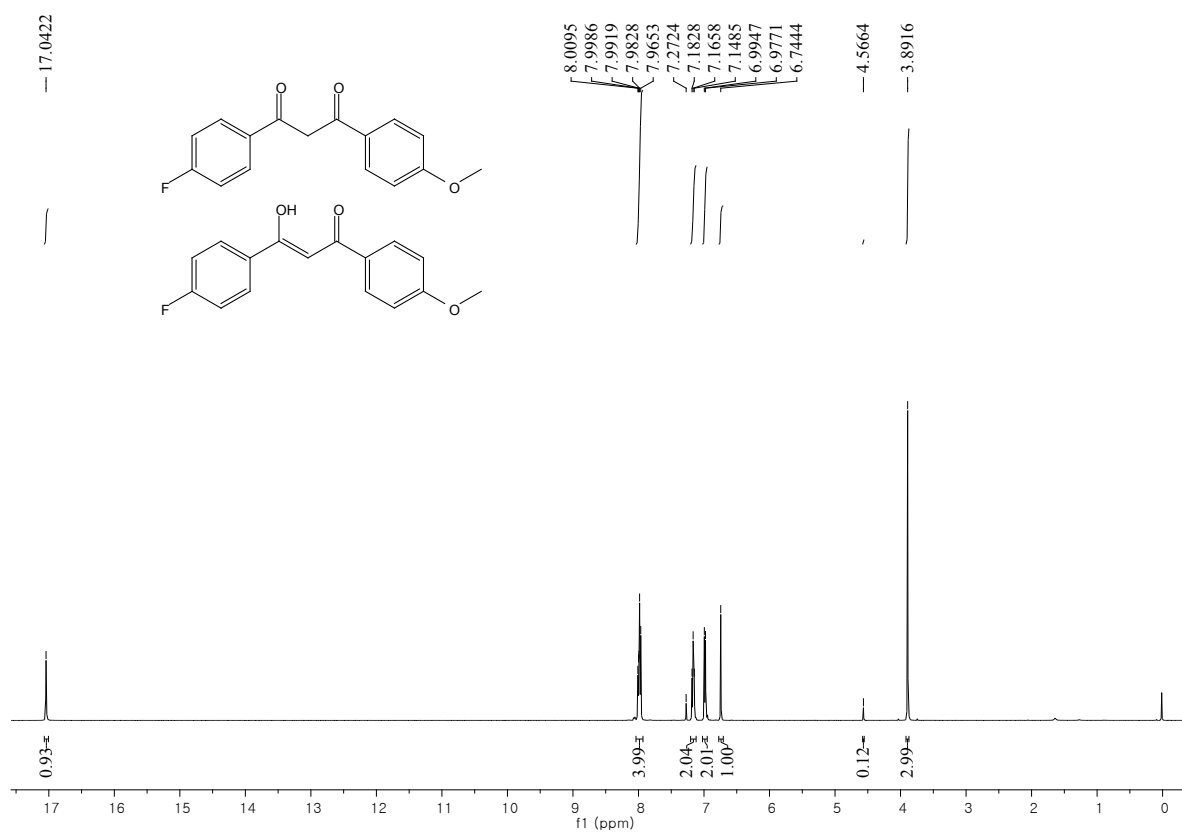
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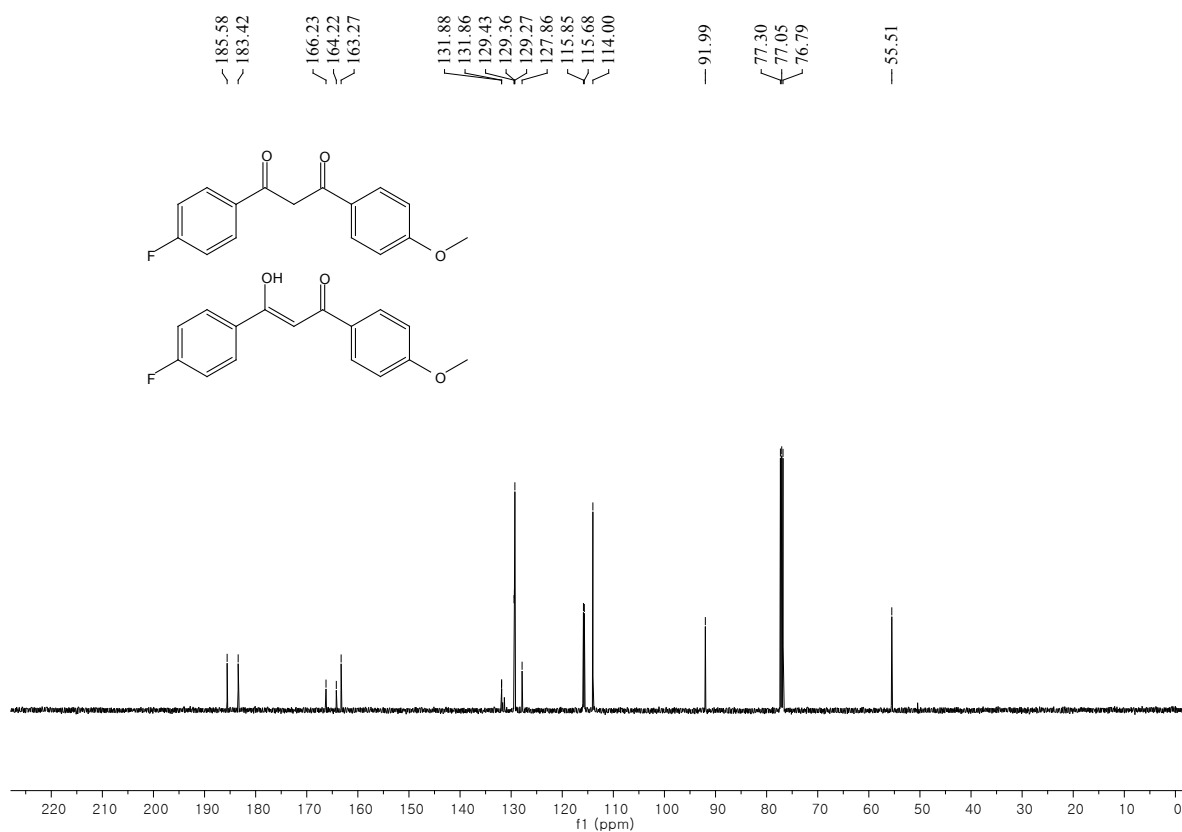
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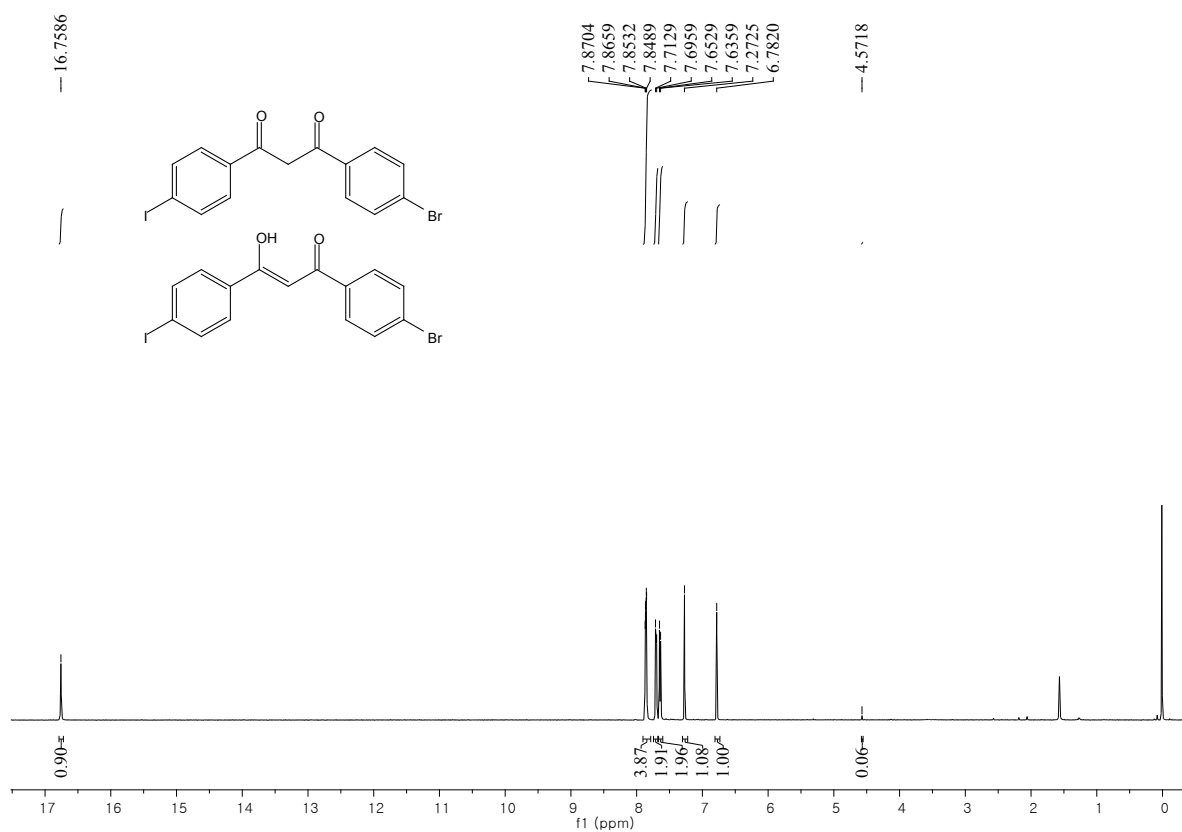
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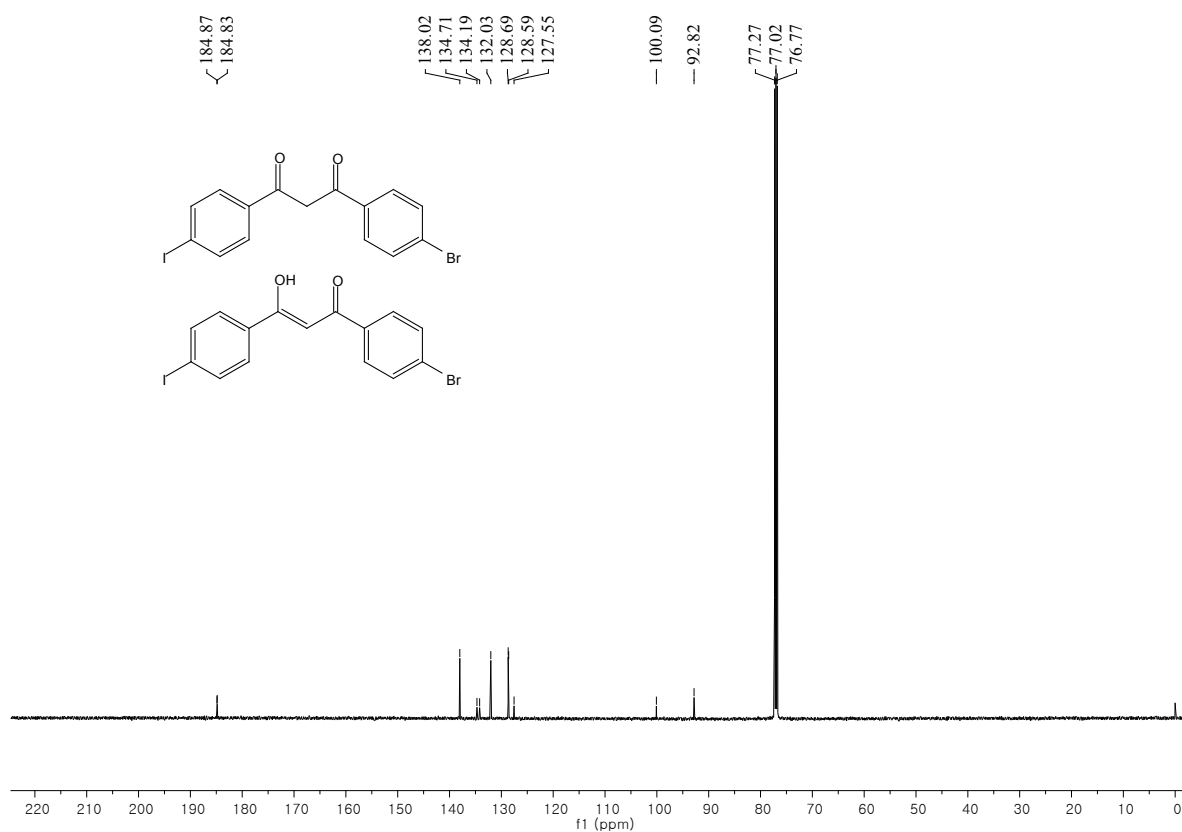
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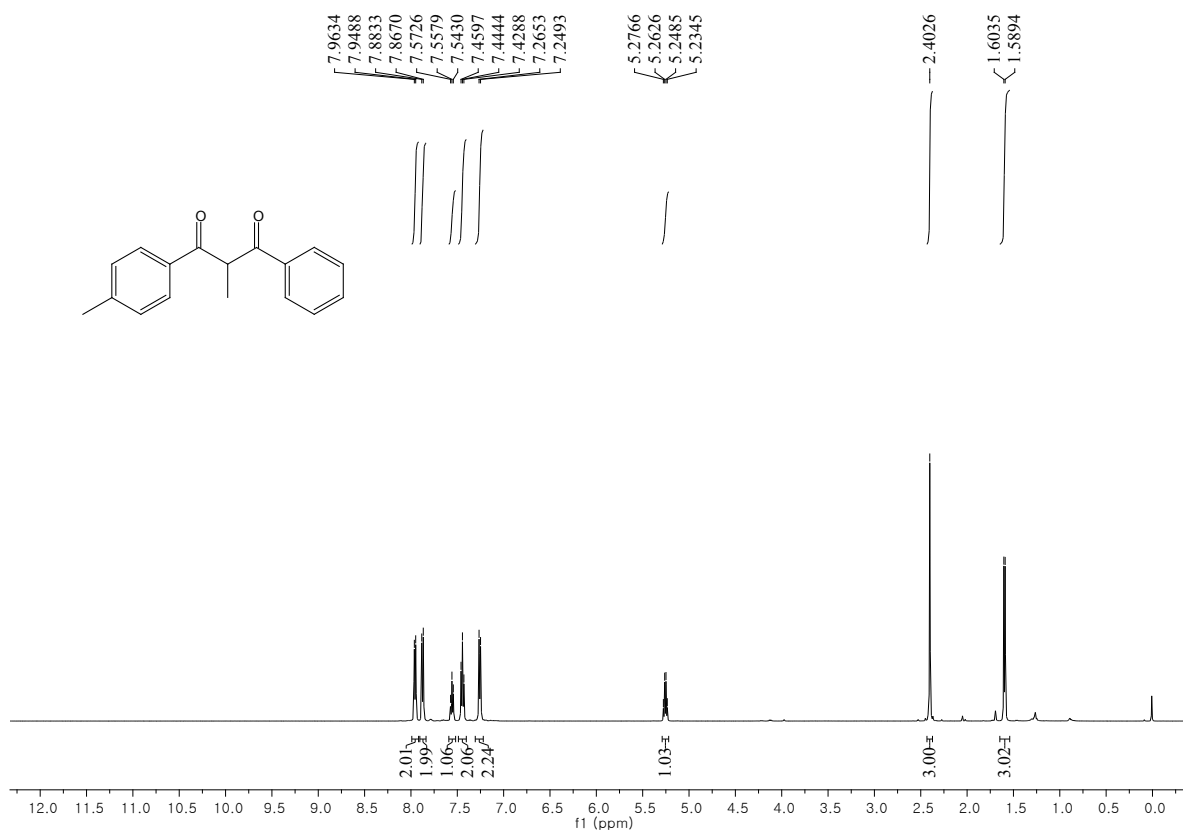
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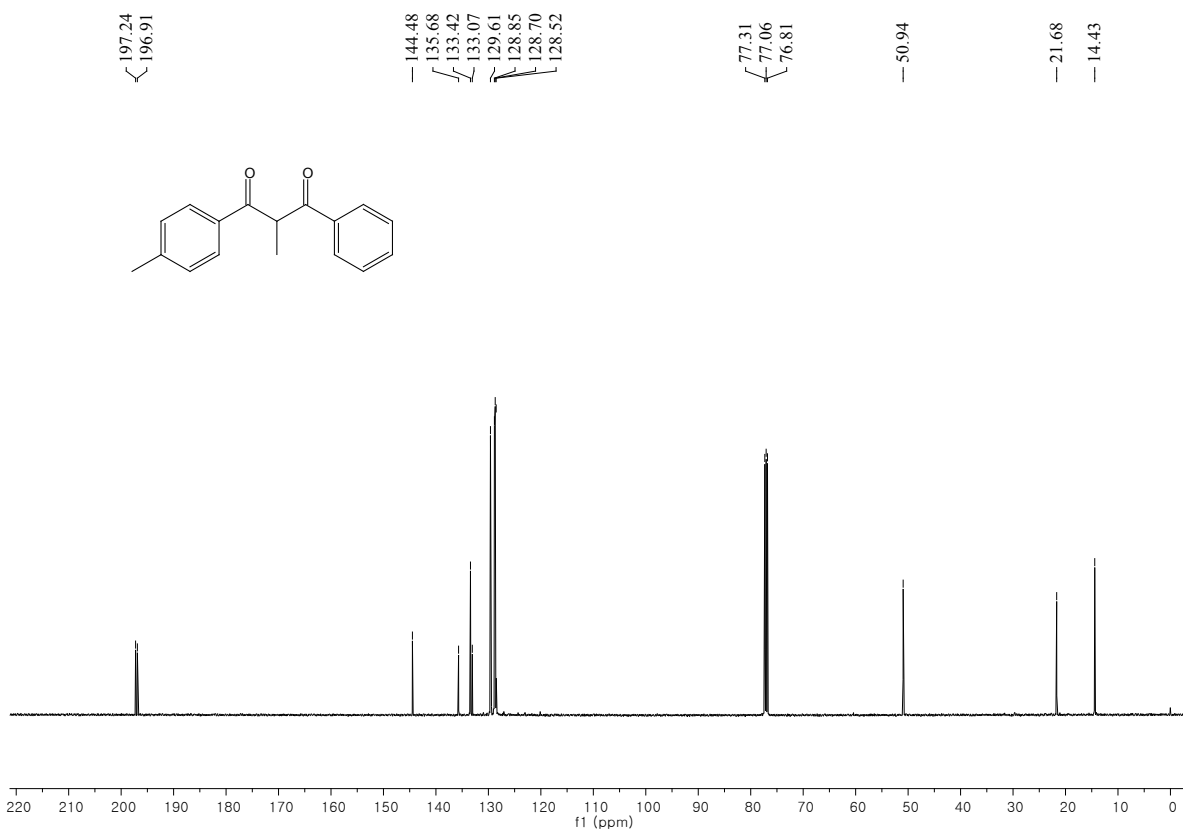
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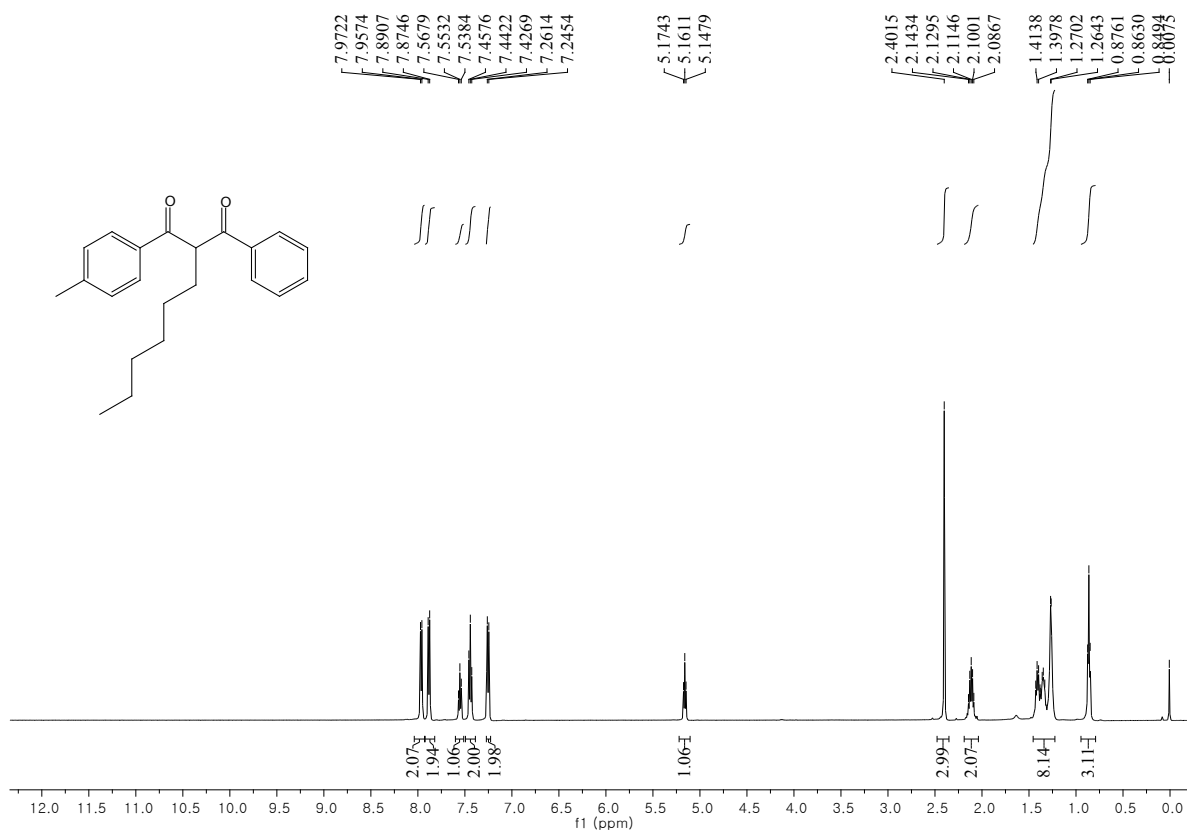
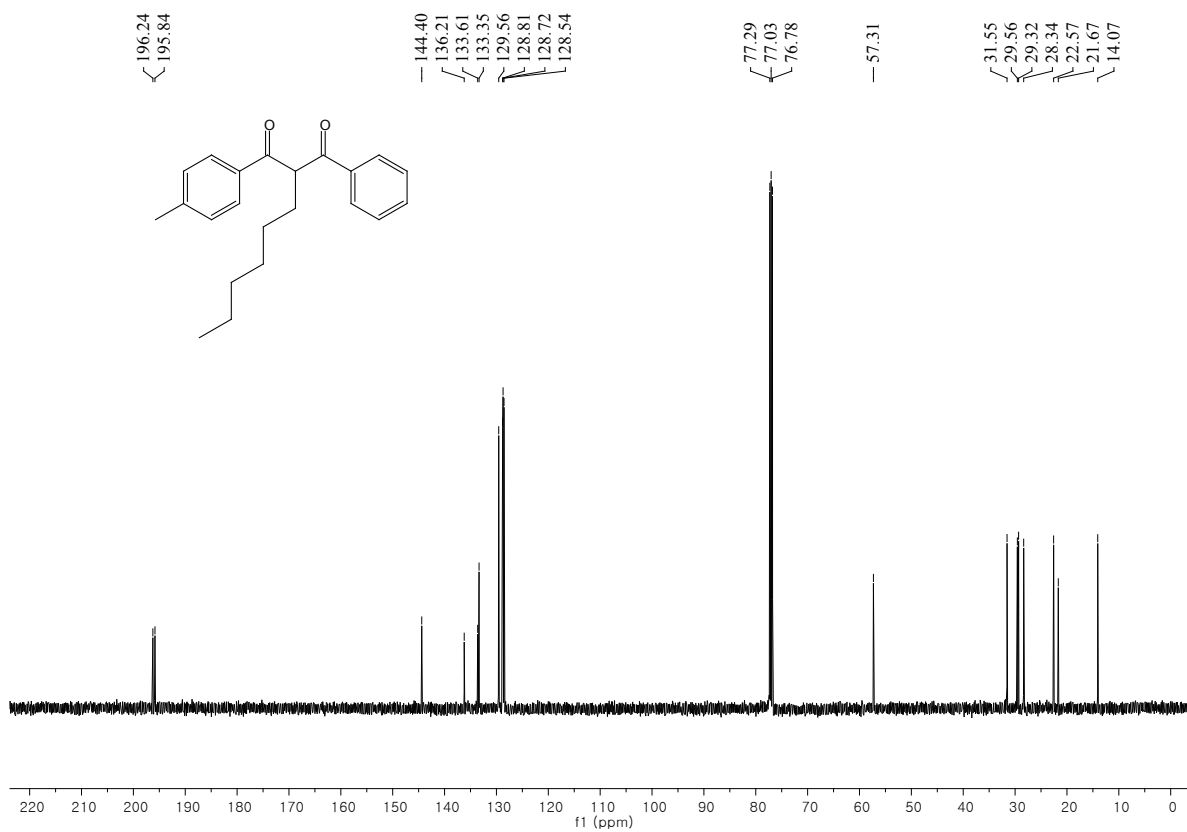


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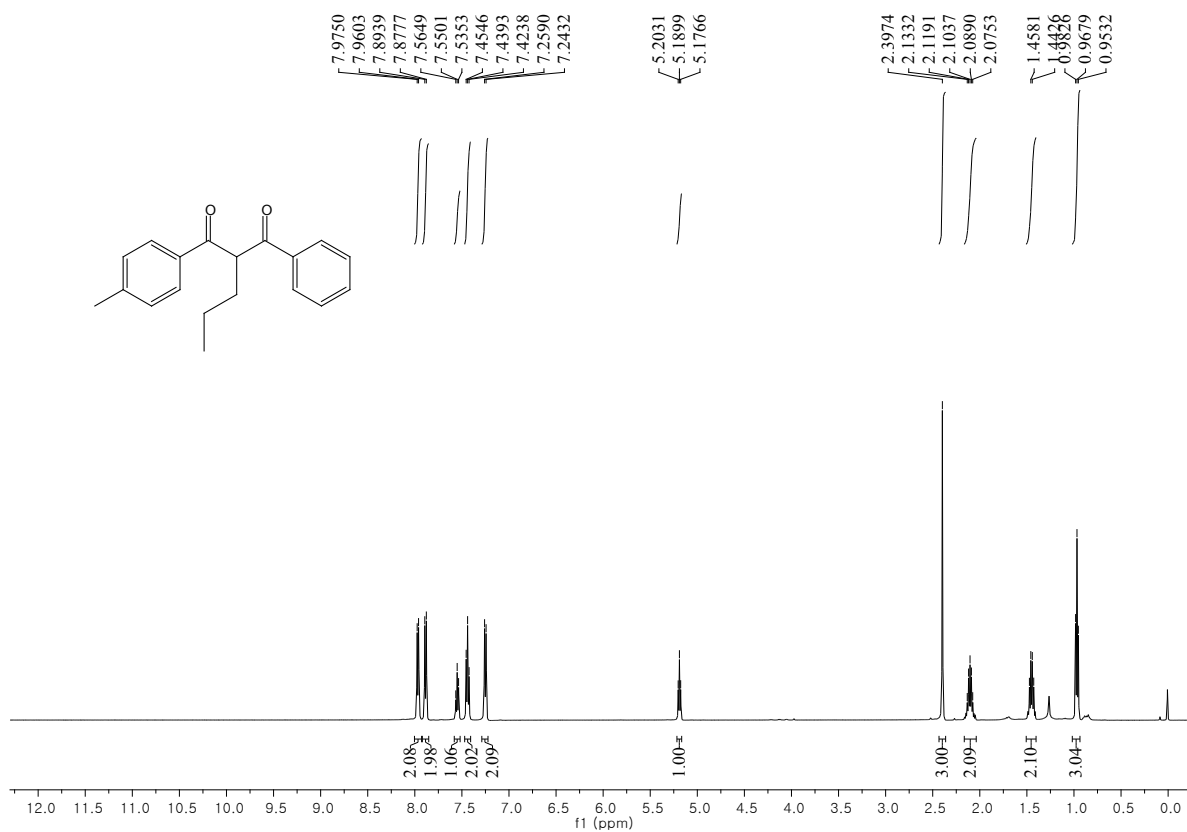


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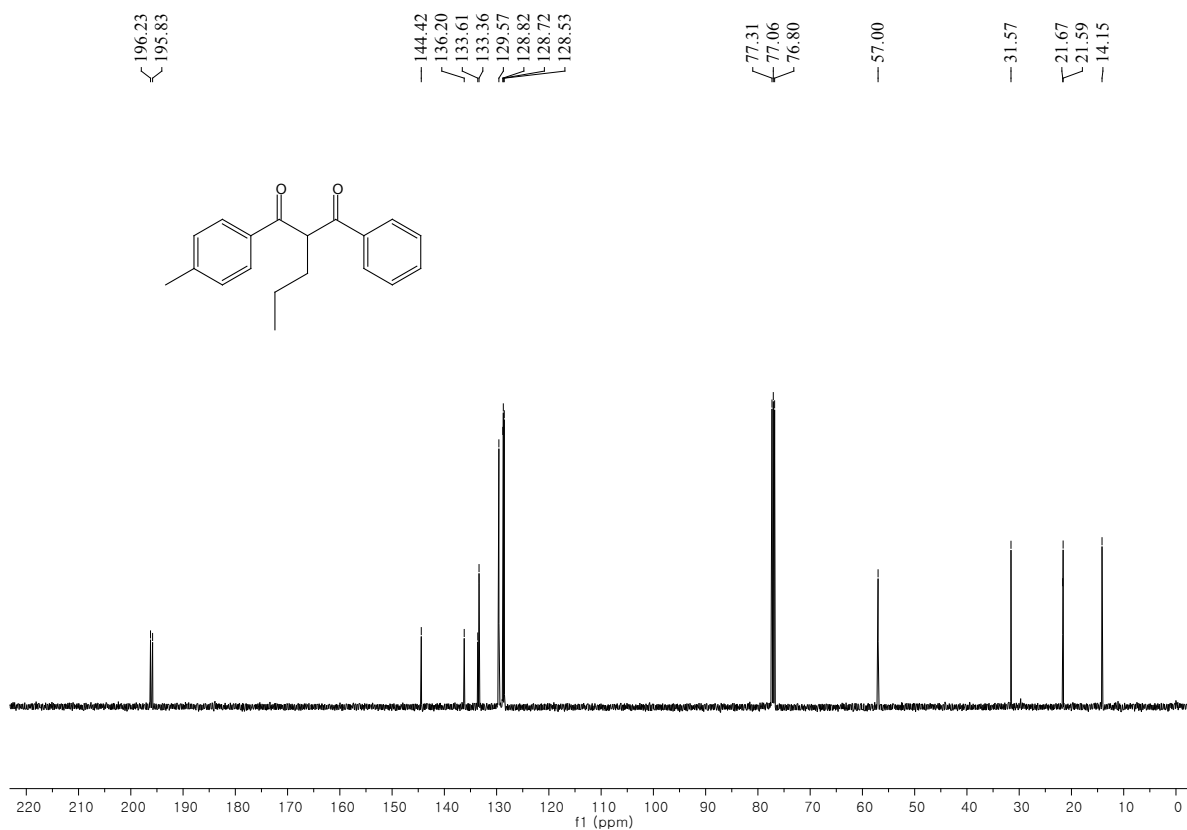


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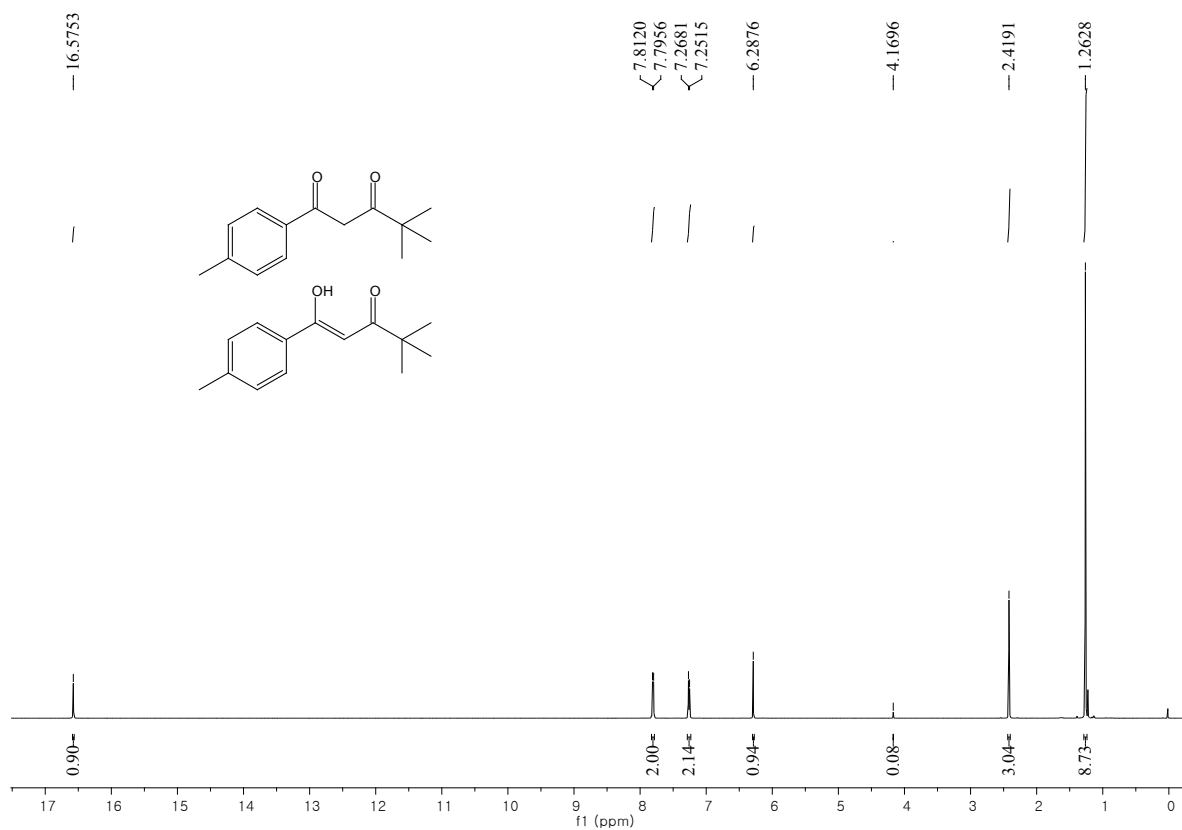
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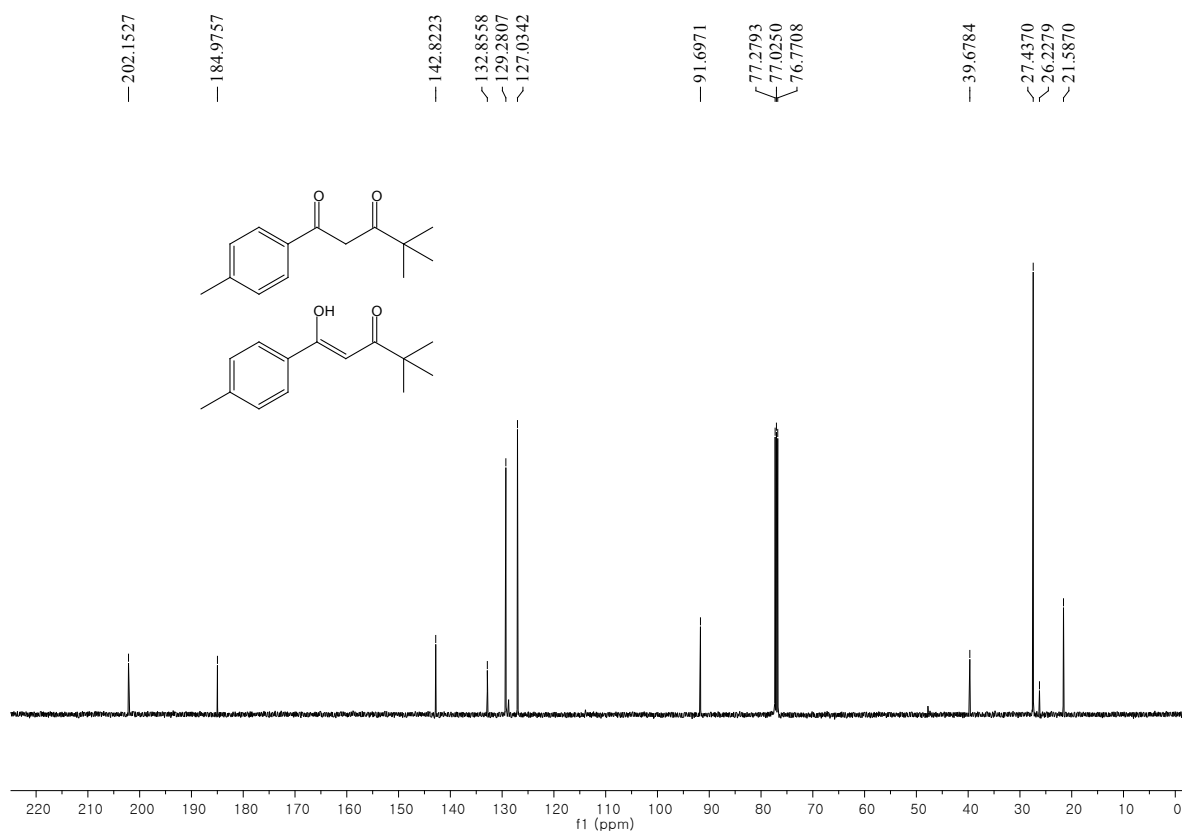
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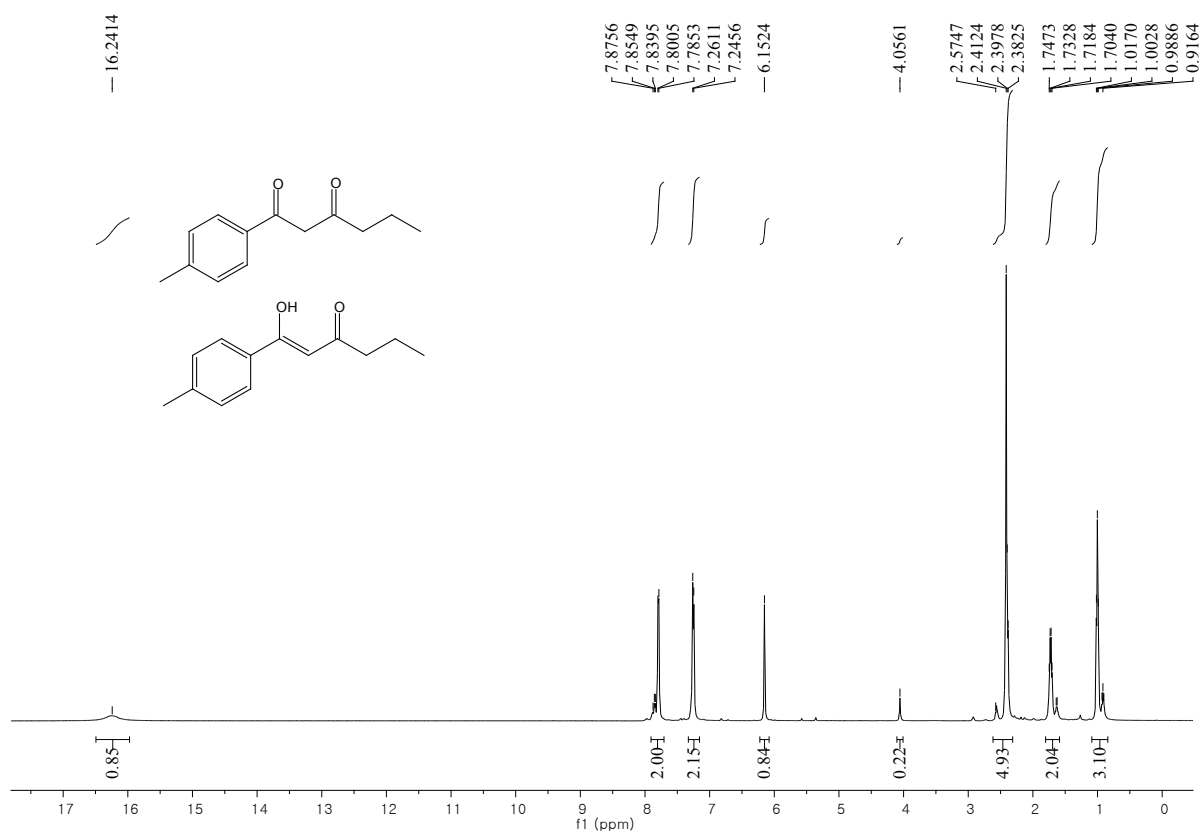
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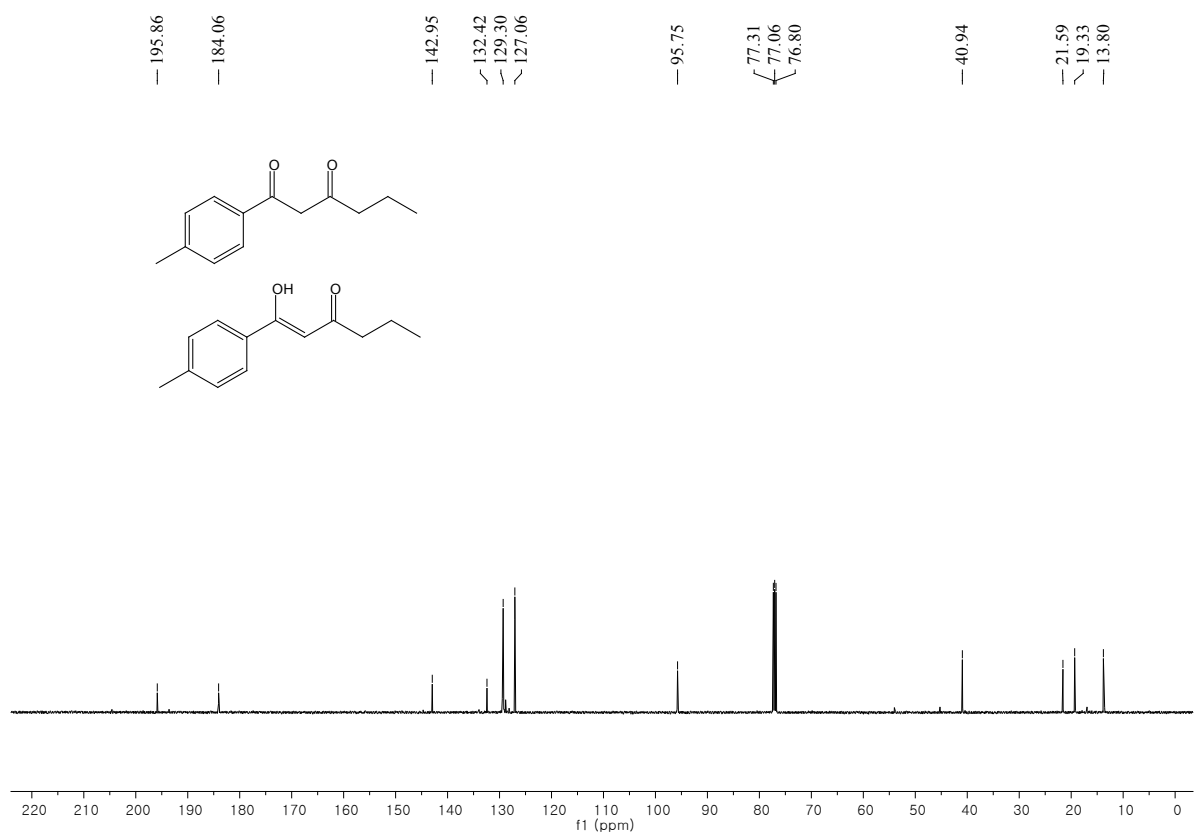
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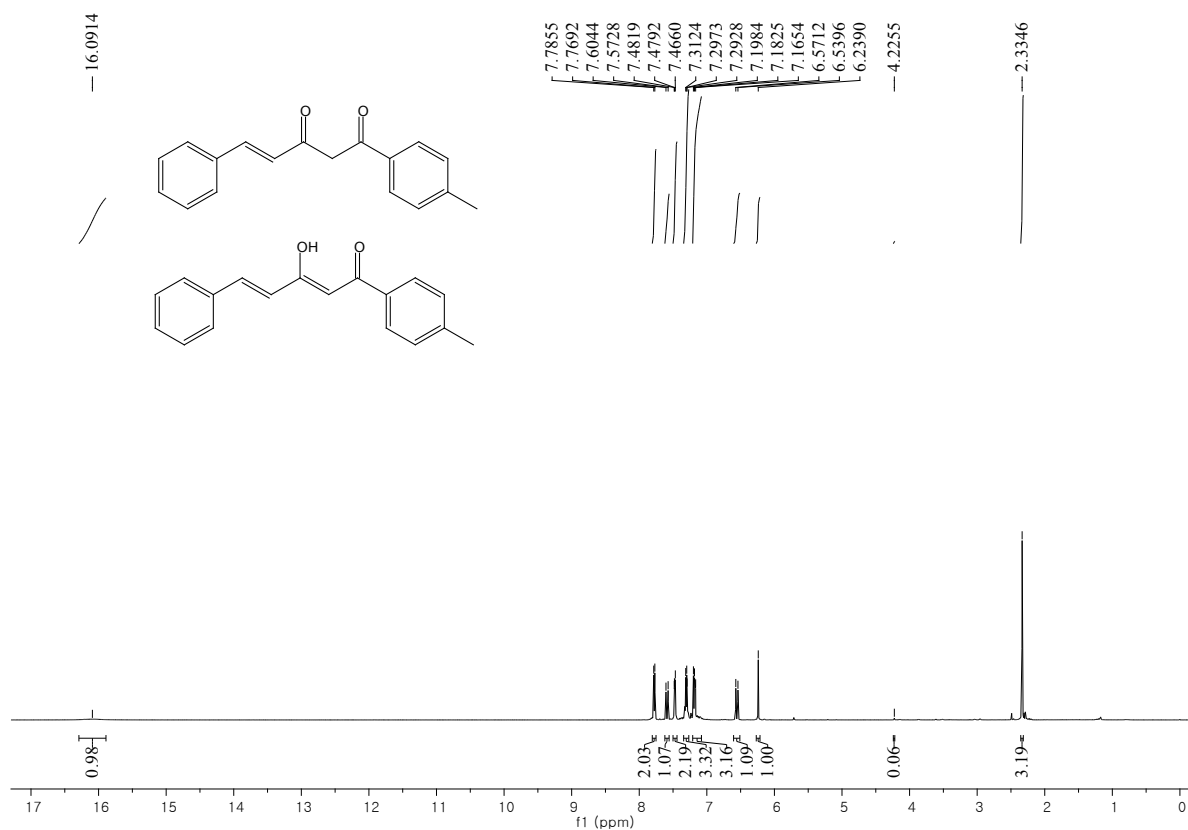
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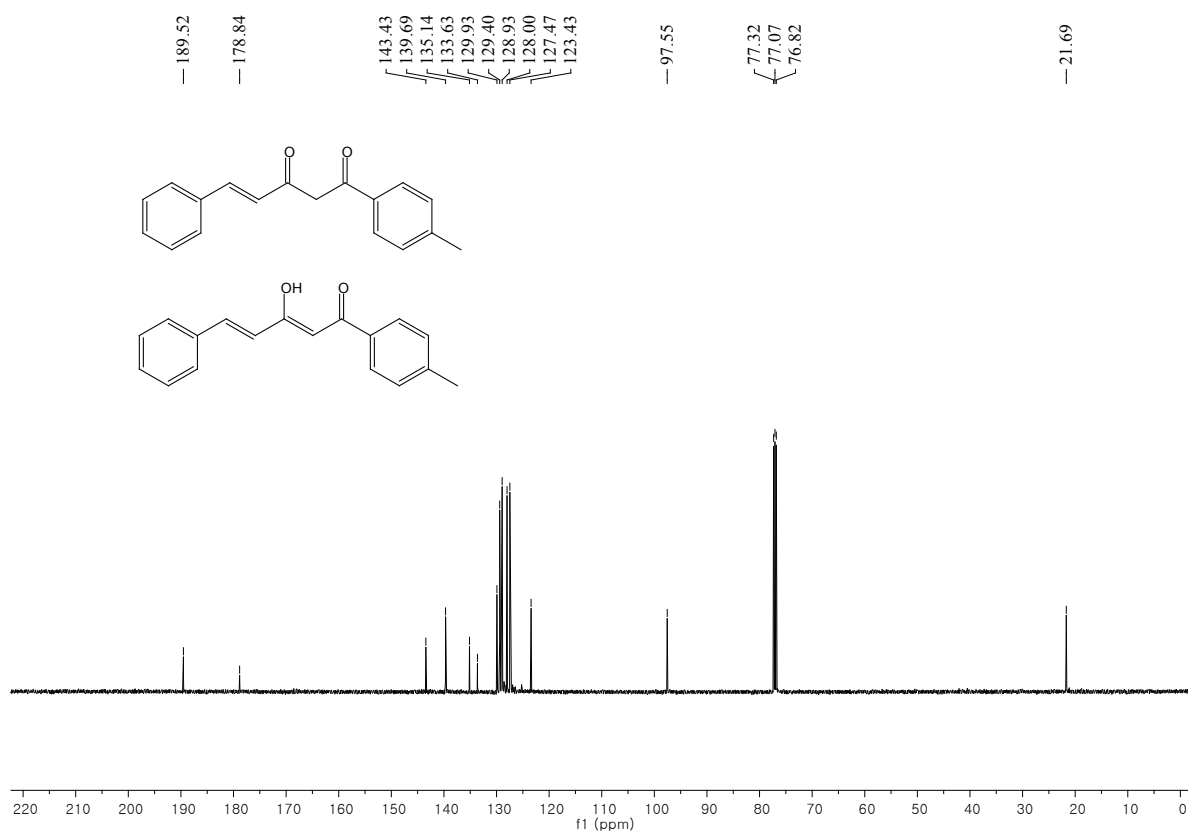
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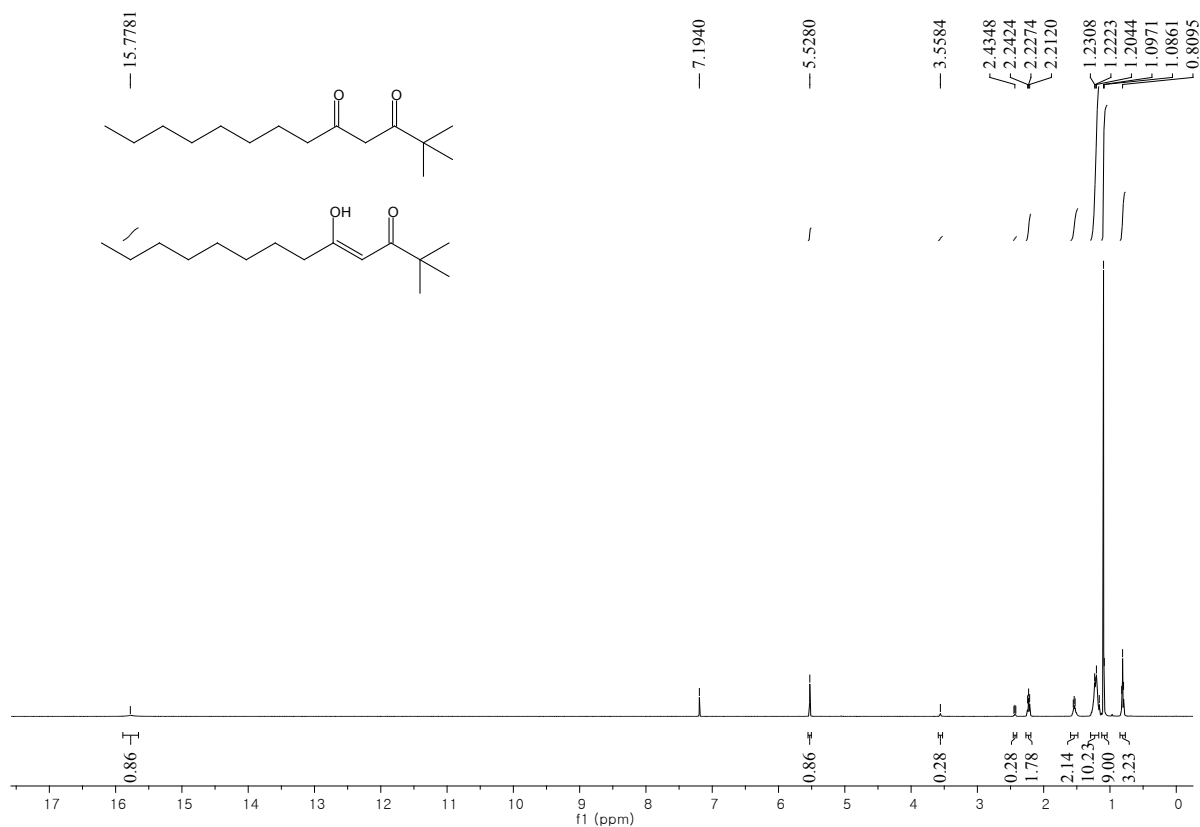
¹³C NMR (126 MHz, CDCl₃): **3ds**



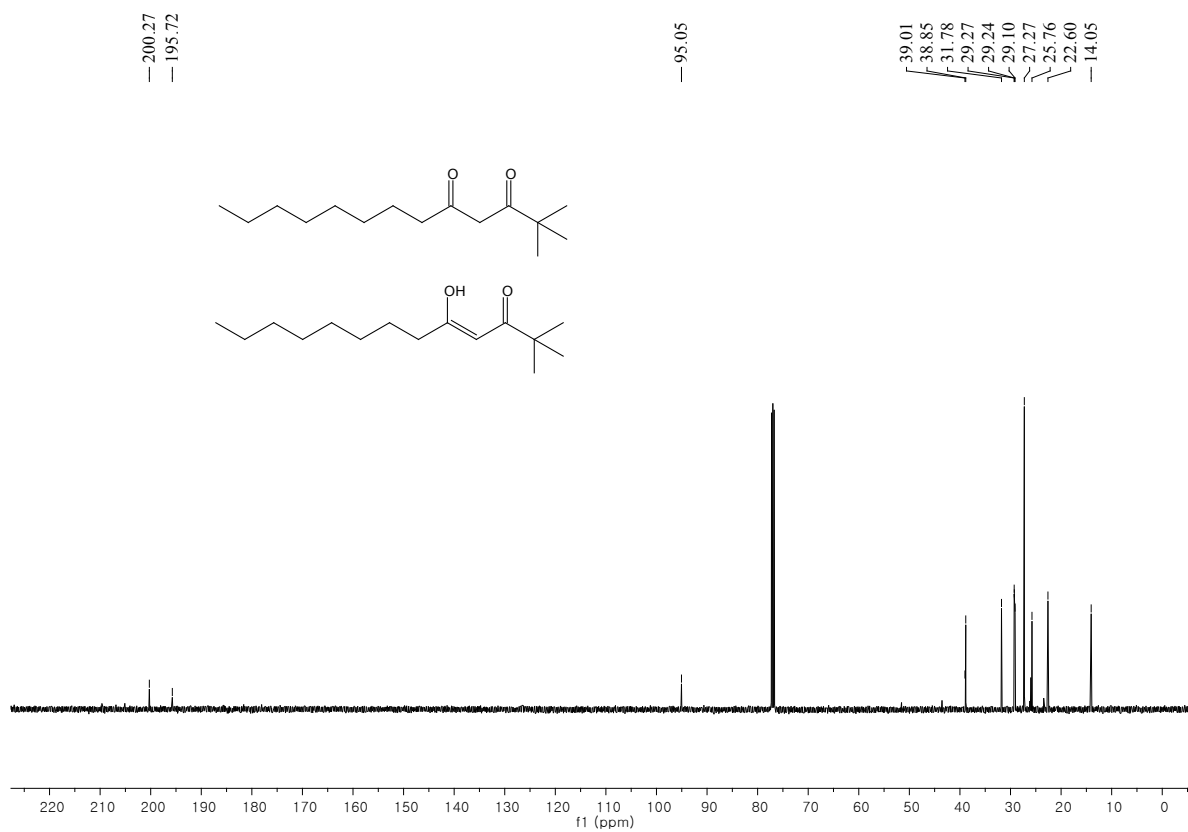
¹H NMR (500 MHz, CDCl₃): **3vd**



^{13}C NMR (126 MHz, CDCl_3): **3vd**



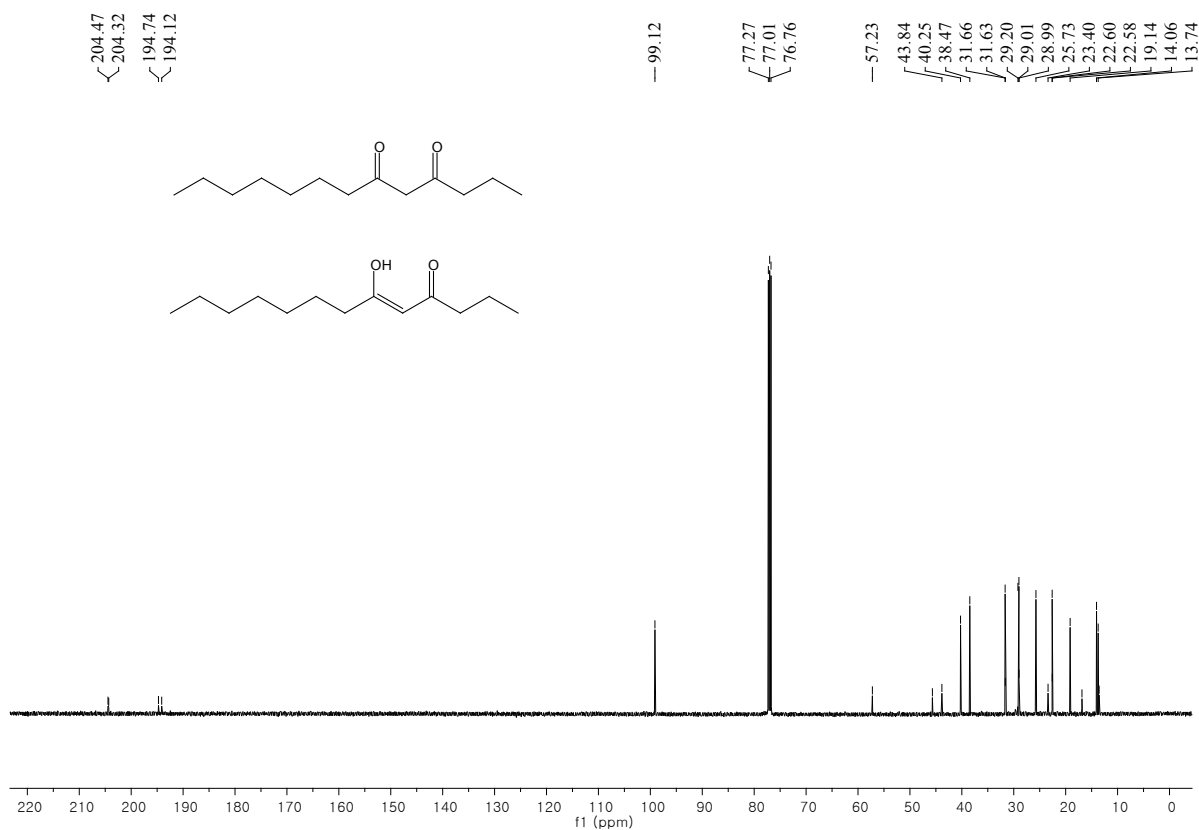
^1H NMR (500 MHz, CDCl_3): **3wr**



¹³C NMR (126 MHz, CDCl₃): **3wr**



¹H NMR (500 MHz, CDCl₃): **3xs**



^{13}C NMR (126 MHz, CDCl_3): **3xs**