Synthesis of *α*-Heterosubstituted Ketones through Sulfur Mediated Difunctionalization of Internal Alkynes

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Supporting Information

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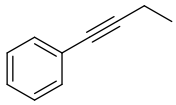
I. General Information

Unless otherwise noted, all materials were purchased from commercial suppliers. Dichloromethane was refluxed over CaH_2, and freshly distilled prior to use. Tetrahydrofuran (THF) was refluxed with sodium/benzophenone, and freshly distilled prior to use. Flash column chromatography was performed using silica gel (normal phase, 200-300 mesh) from Branch of Qingdao Haiyang Chemical. Petroleum ether used for column chromatography were 60-90 ^{o}C fraction, and the removal of residue solvent was accomplished under rotovap with repeated azeotrope with chloroform, and then evaporation under vacuum (< 1 mmHg pressure). Reactions were monitored by thin-layer chromatography on silica gel 60-F254 coated 0.2 mm plates from Yantai Chemical Industry Research Institute. The plates were visualized under UV light, as well as other TLC stains (potassium permanganate: 1% in water; iodine: 10 g iodine absorbed on 30g silica gel). ^{1}H and ^{13}C NMR spectra were recorded on a Bruker 400 MHz spectrometer, usually in $CDCl_3$ with TMS as an internal standard, and the chemical shifts (δ) were reported in parts per million (ppm). The IR spectra (KBr pellets, ν [cm^{-1}]) were take n on a Nicolet 5700 FTIR spectrometer. HRMS measurements were carried out on an Agilent LC/MSD TOF mass spectrometer. The thin layer chromatography silica gel preparative plates were brought from Anhui LiangChen Silicon Material Co. Ltd.

II. Compounds Chart



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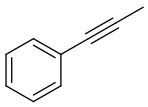

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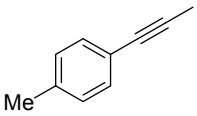

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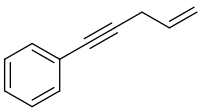

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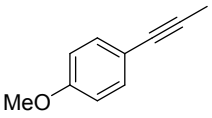

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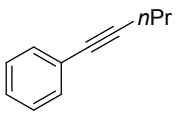

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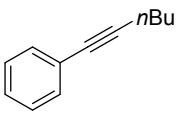

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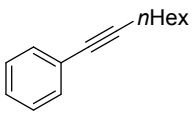

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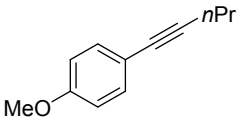

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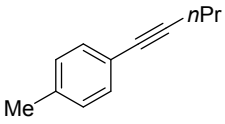

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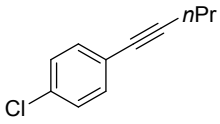

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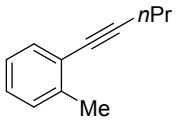

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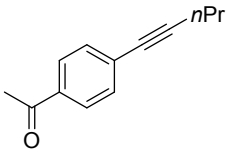

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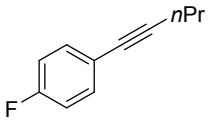

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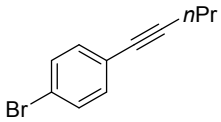

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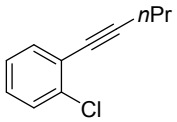

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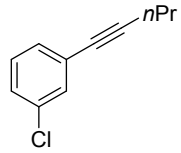

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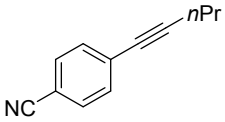

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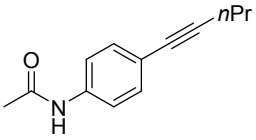

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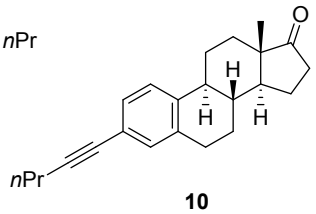

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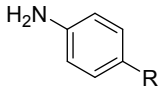

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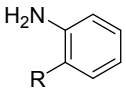

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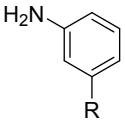

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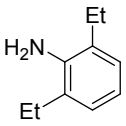

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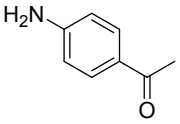

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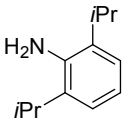

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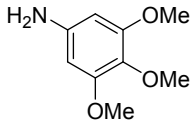

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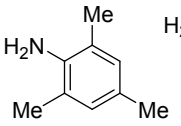

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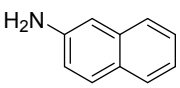

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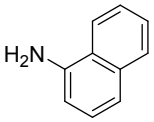

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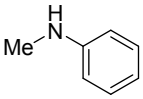

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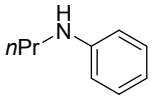

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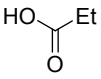

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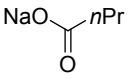

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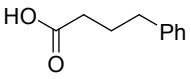

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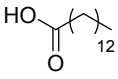

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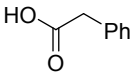

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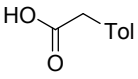

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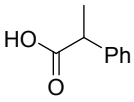

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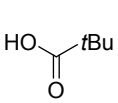

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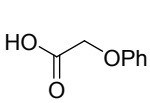

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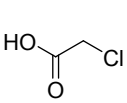

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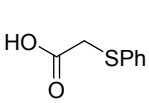

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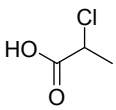

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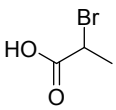

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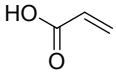

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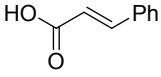

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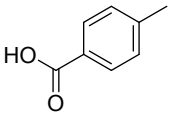

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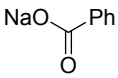

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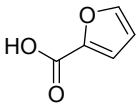

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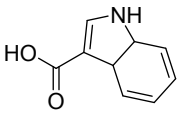

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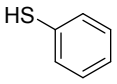

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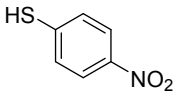

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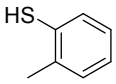

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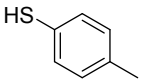

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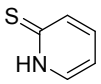

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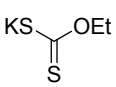

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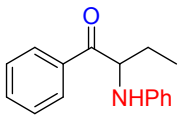

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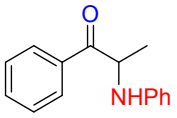

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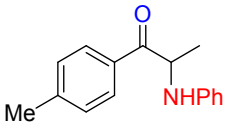

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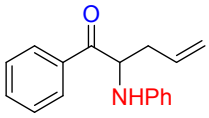

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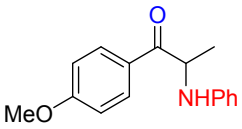

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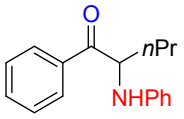

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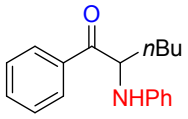

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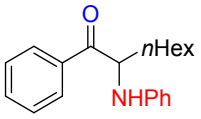

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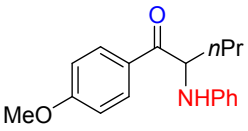

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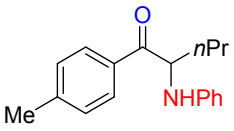

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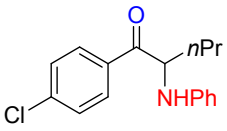

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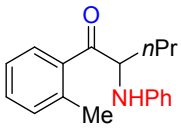

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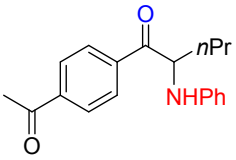

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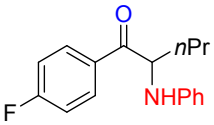

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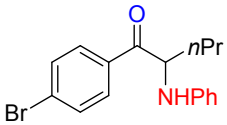

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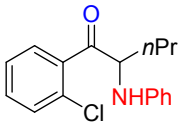

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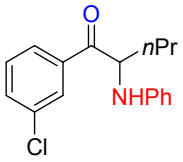

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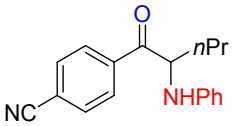

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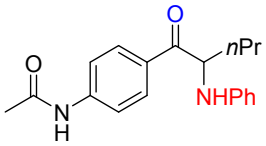

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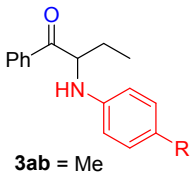

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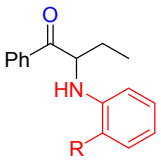
3af= Br
3ag = I
3ah = *t*Bu



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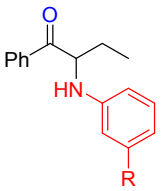

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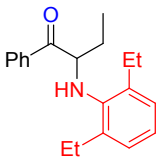

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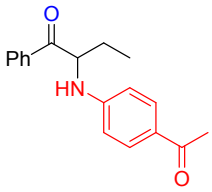

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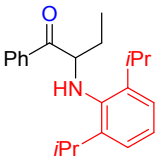

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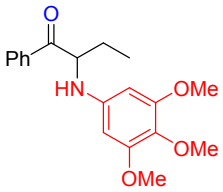

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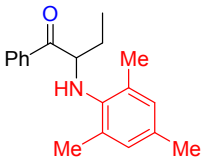

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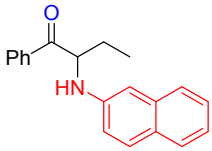

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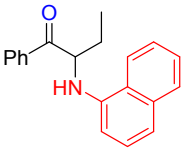

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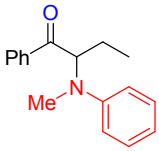

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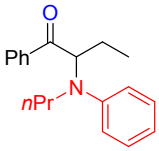

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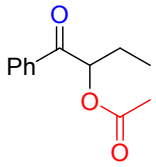

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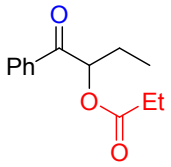

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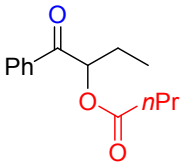

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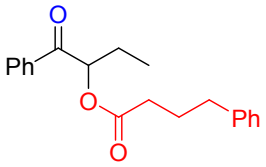

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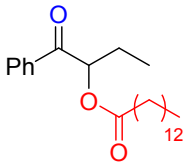

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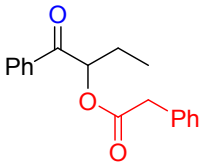

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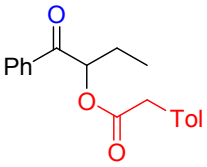

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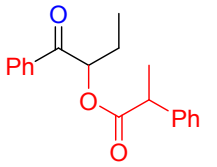

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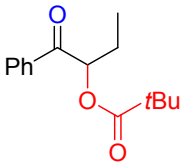

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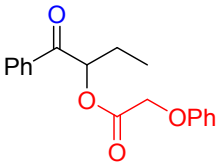

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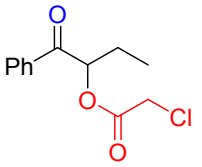

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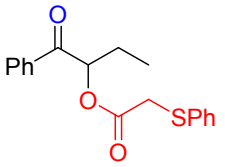

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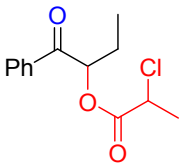

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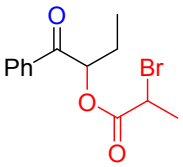

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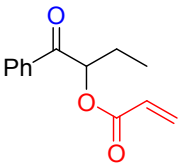

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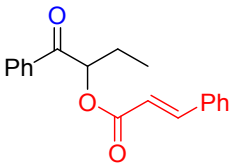

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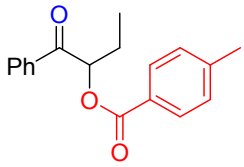

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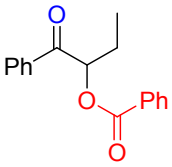

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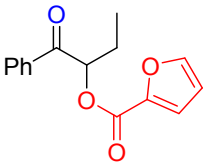

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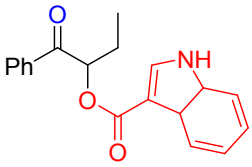

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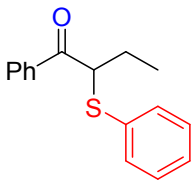

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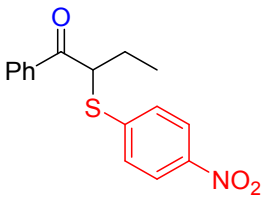

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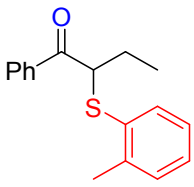

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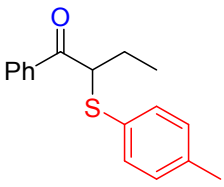

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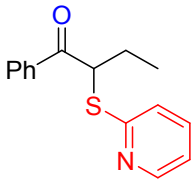

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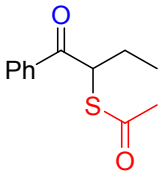

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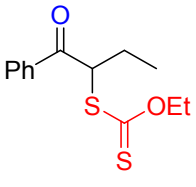

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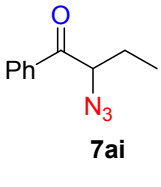

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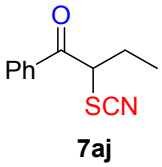

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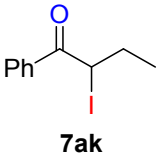

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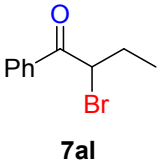

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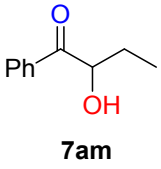

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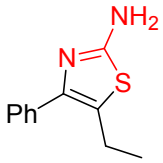

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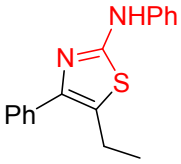

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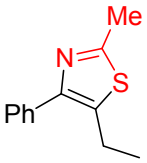

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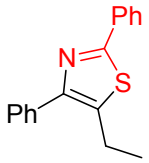

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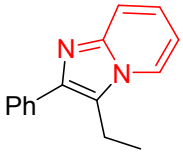

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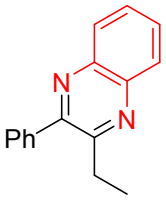

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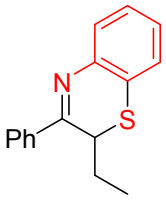

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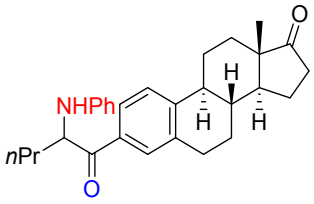

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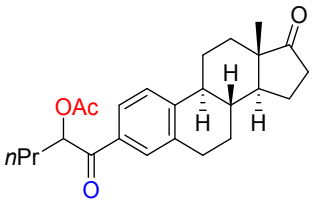

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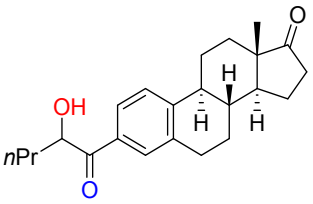
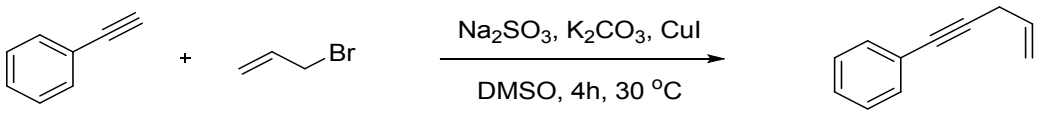


13

III. Experimental Procedures and Characterization Data

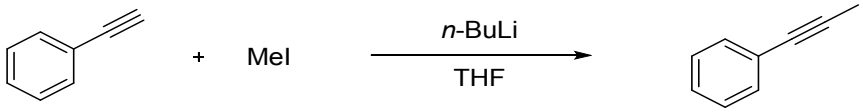
Preparation of alkyne substrates

(1) General Procedure 1 for preparation of alkyne **1d**: [1]



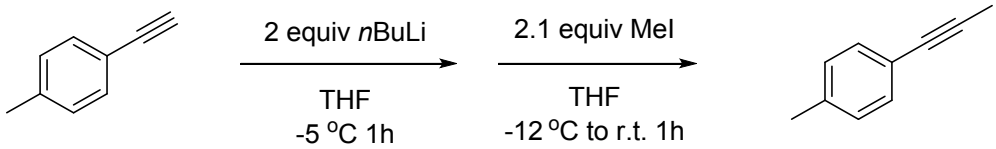
A mixture of 1.5 mmol of allylic bromide, 1.0 mmol of alkyne, 0.5 mmol of Na_2SO_3, 0.02 mmol of CuI , 1.0 mmol of the K_2CO_3 were stirred at 30 °C in 1 mL DMSO for 4 h. After acidic work up and extraction with ether or dichloromethane, the crude products were purified by a short column of silica gel.

(2) General Procedure 2 for preparation of alkynes **1b** and **1e**: [2]



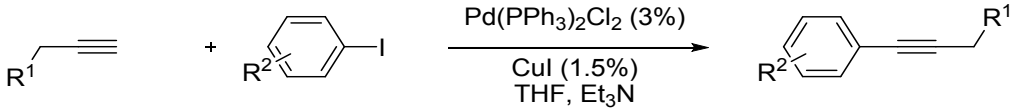
To a flame-dried round-bottom flask under nitrogen was added phenylacetylene (5 mL, 46 mmol, 1 eq) followed by THF (300 mL, 0.15 M). The flask was placed in an ice-water/salt bath and allowed to cool. *n*-Butyllithium (40 mL, 2.5 M in hexanes, 100 mmol, 2 eq) was added slowly and the reaction was allowed to stir for 1 hour. Iodomethane (6 mL, 96.2 mmol, 2.1 eq) was added at -20 °C and the reaction was allowed to stir at room temperature for 1 hour. The reaction was quenched with a saturated solution of ammonium chloride and extracted with dichloromethane. The organics were dried over $MgSO_4$ and the solvent removed under pressure. The residue was purified by a column of silica gel.

(3) Preparation of alkyne **1c**: [3]



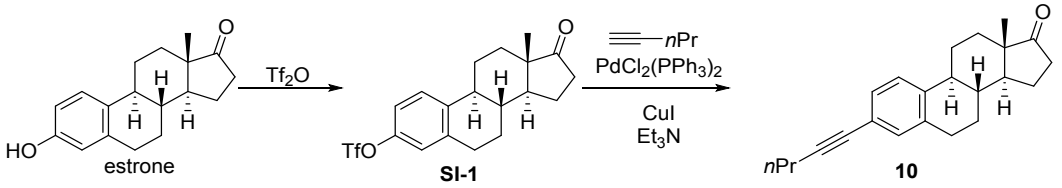
To a dried 200 mL two-neck flask under nitrogen were added 4-ethynyltoluene (581 mg, 5 mmol) and THF (30 mL). The flask was placed in an ice water/salt bath and allowed to cool to around -5 °C. *n*-Butyllithium (2.4 M in hexane, 4.2 mL, 10 mmol) was added dropwise and the reaction mixture was stirred for 1 h. Iodomethane (0.65 mL, 21 mmol) was added at 12 °C and the reaction mixture was stirred at room temperature for 1 h. After quenching with a saturated aqueous NH_4Cl solution, the mixture was extracted with CH_2Cl_2 (20 mL × 3). The combined organic layer was dried over $MgSO_4$ and the solvent was removed under reduced pressure. The residue was purified by a column of silica gel (eluent: hexane) to afford the alkyne **1c** as a colorless oil.

(4) General Procedure 3 for preparation of alkynes **1f**–**1s**: [4]



A dried 50 mL flask was added iodobenzene (361 mg, 4 mmol), $Pd(PPh_3)_2Cl_2$ (42 mg, 0.06 mmol), CuI (23 mg, 0.12 mmol), triethylamine (1.010 g, 10 mmol) and THF (20 mL). The flask was purged with nitrogen and alkynes (4.4 mmol) were added via syringe. After the addition, the reaction mixture was stirred overnight at room temperature. The resulting mixture was filtered to remove the ammonium salt, and the filtrate was concentrated in vacuo. Purification of the residue by column chromatograph (petroleum ether) gave alkynes **1f−1s**.

(5) Preparation of the alkyne **10**: [5]



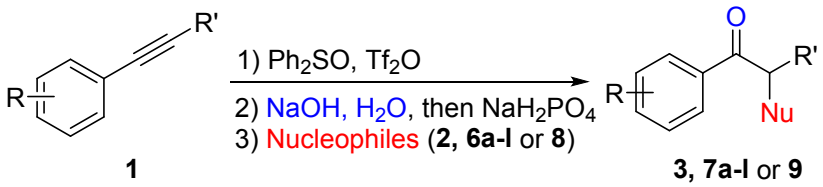
To a solution of estrone (1.35 g, 5 mmol) and pyridine (0.81 g, 10 mmol) in dry CH_2Cl_2 (50 mL) was added Tf_2O (1 ml, 6 mmol) dropwise at 0 °C. After that, the mixture was warmed to rt, and stirred overnight. The mixture was then quenched with HCl (aq, 10%) and extracted with CH_2Cl_2. The combined organic layer was dried over anhydrous Na_2SO_4. The filtrate was concentrated in vacuo and the residue was purified by flash column chromatography to afford **SI-1** (1.83 g, 91 %).
A mixture of **SI-1** (1.61 g, 4 mmol), alkyne (0.5 mL, 5 mmol) trimethylamine (3.0 mL), $Pd(PPh_3)_2Cl_2$ (84 mg, 0.12 mmol) CuI (84 mg, 0.12 mmol) in 15 mL DMF was stirred at 90 °C for 4 h under nitrogen. The mixture was quenched with water and extracted with EtOAc (50 mL x 3). The combined organic phases are washed with brine and dried over Na_2SO_4. The filtrate was concentrated in vacuo and the residue was purified by flash column chromatography to afford crude product. Recrystallization from petroleum ether gave **10** as a white solid (538 mg, 42 %), mp 162-164 °C.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22−7.16 (m, 2H), 7.15 (s, 1H), 2.87 (dd, J = 8.8, 4.0 Hz, 2H), 2.51 (dd, J = 18.8, 8.7 Hz, 1H), 2.44−2.33 (m, 3H), 2.29 (td, J = 10.6, 3.9 Hz, 1H), 2.20−1.94 (m, 4H), 1.68−1.56 (m, 4H), 1.55−1.37 (m, 4H), 1.04 (t, J = 7.4 Hz, 3H), 0.91 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 220.8, 139.3, 136.4, 132.0, 128.9, 125.2, 121.4, 89.5, 80.6, 50.5, 48.0, 44.4, 38.0, 35.8, 31.6, 29.1, 26.4, 25.6, 22.3, 21.6, 21.4, 13.8, 13.5.
IR (KBr) ν (cm^{-1}) 2931, 2868, 1737, 1495, 1499, 1453, 1084, 825..
HRMS (ESI) calcd for $C_{23}H_{29}O^+$ $[M+H]^+$ *m/z:* 321.2213, found: 321.2211.

References:

[1] Bieber, L. W.; Silve, M. F. *Tetrahedron Lett*. **2007**, *48*, 7088.
[2] Stuart, D. R.; Bertrand-Laperle, M.; Burgess, K. M. N.; Fagnou, K. *J. Am. Chem. Soc.* **2008**, *130*, 16474.
[3] Fujihara, T.; Tani, Y.; Semba, K.; Terao, J.; Tsuji, Y. *Angew. Chem., Int. Ed*. **2012**, *51*, 11487.
[4] Yamagami, T.; Shintani, R.; Shirakawa, E.; Hayashi, T. *Org. Lett*. **2007**, *9*, 1045.
[5] Xu, Z. F.; Dai, H, C.; Shan, L. H.; Li, C. Y. *Org. Lett*. **2018**, *20*, 1054.

Synthesis of α-Heterosubstituted Ketones through Sulfur Mediated Difunctionalization of Internal Alkynes

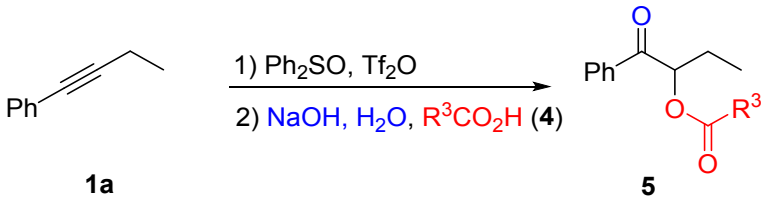
General Procedure A



1 **3, 7a-l** or **9**

To a flame-dried Schlenk tube, alkyne (**1**) (0.4 mmol) and diphenyl sulfoxide (97 mg, 0.48 mmol) were added, and then dissolved with dichloromethane (2 mL) under nitrogen atmosphere before cooling down to -78 ºC (liquid N_2/ethyl acetate bath). Tf_2O (81 µL, 0.48 mmol) was added dropwise. After being stirred for 20 min, the mixture was warmed up to 0 ºC and stirred for additional 20 min. Then NaOH (48 mg, 1.2 mmol) and H_2O (144 mg, 8 mmol) were added to the mixture. After stirring at 40 ºC for 12 h, the NaH_2PO_4•$2H_2O$ (125 mg, 0.8 mmol) and nucleophiles (**2, 6a-l** or **8**) (0.6 mmol) were added to solution, and continued to stir at the same temperature for 12 h. The solution was extracted with CH_2Cl_2 (10 mL×3). The combined organic phases were dried with Na_2SO_4 and concentrated under reduced pressure. The residue was further purified by flash column chromatography on silica gel and afforded the pure product (**3, 7a-l** or **9**).

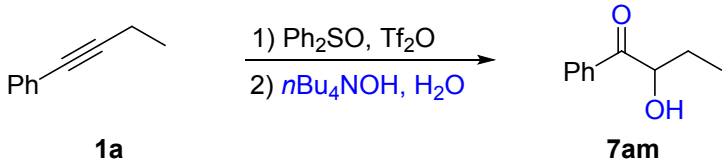
General procedure B



1a **5**

To a flame-dried Schlenk tube, alkyne (**1a**) (52 mg, 0.4 mmol) and diphenyl sulfoxide (97 mg, 0.48 mmol) were added, and then dissolved with dichloromethane (2 mL) under nitrogen atmosphere before cooling down to -78 ºC (liquid N_2/ethyl acetate bath). Tf_2O (81 µL, 0.48 mmol) was added dropwise. After being stirred for 20 min, the mixture was warmed up to 0 ºC and stirred for additional 20 min. Then NaOH (80 mg, 2 mmol), carboxylic acid (2 mmol) and H_2O (108 mg, 6 mmol) were added to the solution and stirred at 40 ºC for 12 h. The solution was extracted with CH_2Cl_2 (10 mL×3). The combined organic phases were dried with Na_2SO_4 and concentrated under reduced pressure. The residue was further purified by flash column chromatography on silica gel, and afforded the pure product (**5**).

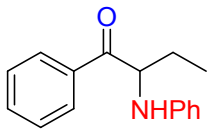
General procedure C



1a **7am**

To a flame-dried Schlenk tube, alkyne (**1a**) (52 mg, 0.4 mmol) and diphenyl sulfoxide (97 mg, 0.48 mmol) were added, and then dissolved with dichloromethane (2 mL) under nitrogen atmosphere before cooling down to -78 ºC (liquid N_2/ethyl acetate bath). Tf_2O (81 μL, 0.48 mmol) was added dropwise. After being stirred for 20 min, the mixture was warmed up to 0 ºC and stirred for additional 20 min. Then added 1.5 mL 10 % aqueous nBu$_4$NOH solution to the mixture and stirred at 40 ºC for 12 h. The solution was extracted with CH_2Cl_2 (10 mL×3). The combined organic phases were dried with Na_2SO_4 and concentrated under reduced pressure. The residue was further purified by flash column chromatography on silica gel, and afforded the pure product (**7am**).

Characterization Data of compounds



3aa

1-Phenyl-2-(phenylamino)butan-1-one (**3aa**)[6] was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **2a** (56 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 69 mg product **3aa** as a yellow solid in 72% yield.

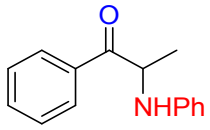
3aa: R_f = 0.62 (petroleum ether/ethyl acetate = 5:1), mp 77-80 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, J = 8.3 Hz, 2H), 7.62 (t, J = 7.4 Hz, 1H), 7.51 (t, J = 7.6 Hz, 2H), 7.23−7.15 (m, 2H), 6.73 (t, J = 7.4 Hz, 3H), 5.10 (t, J = 5.3 Hz, 1H), 4.74 (s, 1H), 2.17−2.00 (m, 1H), 1.82−1.68 (m, 1H), 0.92 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 200.4, 147.0, 135.2, 133.5, 129.3, 128.8, 128.3, 117.8, 113.5, 58.7, 25.9, 9.1.

IR (KBr) v (cm^{-1}) 3395, 3054, 2968, 2934, 2875, 1685, 1602, 1508, 1448, 1318, 749, 694.

HRMS (ESI) calcd for $C_{16}H_{18}ON^+$ $[M+H]^+$ m/z: 240.1383, found: 240.1380.



3ba

1-Phenyl-2-(phenylamino)propan-1-one (**3ba**)[6] was synthesized according to General Procedure A from **1b** (46 mg, 0.4 mmol) and **2a** (56 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 48 mg product **3ba** as a yellow solid in 53% yield.

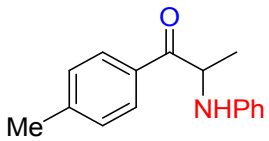
3ba: R_f = 0.23 (petroleum ether/ethyl acetate = 20:1), mp 105-107 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10−7.97 (m, 2H), 7.62 (t, J = 7.4 Hz, 1H), 7.52 (t, J = 7.6 Hz, 2H), 7.20 (t, J = 7.9 Hz, 2H), 6.78−6.61 (m, 3H), 5.15 (q, J = 6.8 Hz, 1H), 4.73 (s, 1H), 1.50 (d, J = 6.8 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 200.6, 146.5, 134.7, 133.6, 129.4, 128.9, 128.4, 117.9, 113.5, 53.3, 19.6.

IR (KBr) v (cm^{-1}) 3354, 2932, 1681, 1596, 2505, 1448, 1226, 1169, 753, 696.

HRMS (ESI) calcd for $C_{15}H_{16}NO^+$ $[M+H]^+$ m/z: 226.1226, found: 226.1235.



3ca

2-(Phenylamino)-1-p-tolylpropan-1-one (**3ca**)[6] was synthesized according to General Procedure A from **1c** (52 mg, 0.4 mmol) and **2a** (56 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 42 mg product **3ca** as a yellow solid in 44% yield.

3ca: R_f = 0.21 (petroleum ether/ethyl acetate = 20:1), mp 103-105 °C

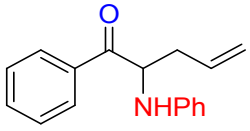
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, J = 8.0 Hz, 2H), 7.32 (d, J = 8.0 Hz, 2H), 7.23−7.16 (m,

2H), 6.78−6.65 (m, 3H), 5.12 (p, J = 6.4 Hz, 1H), 4.75 (s, 1H), 2.45 (s, 3H), 1.49 (d, J = 6.9 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 200.1, 146.6, 144.6, 132.1, 129.5, 129.3, 128.6, 117.7, 113.4, 53.1, 21.7 19.7.

IR (KBr) v (cm^{-1}) 3394, 2979, 1682, 1604, 1505, 1451, 1318, 1156, 971, 748, 693.

HRMS (ESI) calcd for $C_{16}H_{18}NO^+$ $[M+H]^+$ m/z: 240.1383, found: 240.1389.



3da

1-Phenyl-2-(phenylamino)pent-4-en-1-one (**3da**)[6] was synthesized according to General Procedure A from **1d** (57 mg, 0.4 mmol) and **2a** (56 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 52 mg product **3da** as a yellow oil in 52% yield.

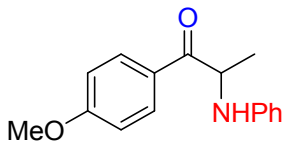
3da: R_f = 0.34 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04−7.99 (m, 2H), 7.62 (t, J = 7.4 Hz, 1H), 7.52 (t, J = 7.6 Hz, 2H), 7.19 (t, J = 7.9 Hz, 2H), 6.78−6.66 (m, 3H), 5.75 (ddt, J = 17.2, 10.1, 7.2 Hz, 1H), 5.17 (dd, J = 12.5, 5.9 Hz, 1H), 5.09 (d, J = 10.1 Hz, 1H), 5.03 (dd, J = 17.0, 1.4 Hz, 1H), 4.68 (d, J = 6.7 Hz, 1H), 2.85−2.70 (m, 1H), 2.57−2.39 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 199.6, 146.6, 135.2, 133.6, 132.6, 129.3, 128.9, 128.4, 118.8, 118.1, 113.7, 57.5, 37.0.

IR (KBr) v (cm^{-1}) 3395, 3055, 1686, 1602, 1507, 1448, 1317, 1225, 994, 920, 749, 690.

HRMS (ESI) calcd for $C_{17}H_{18}NO^+$ $[M+H]^+$ m/z: 252.1383, found: 252.1389.



3ea

1-(4-Methoxyphenyl)-2-(phenylamino)propan-1-one (**3ea**)[6] was synthesized according to General Procedure A from **1e** (58 mg, 0.4 mmol) and **2a** (56 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 21 mg product **3ea** as a yellow oil in 21% yield.

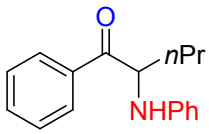
3ea: R_f = 0.21 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, J = 8.9 Hz, 2H), 7.18 (t, J = 7.9 Hz, 2H), 7.00−6.94 (m, 2H), 6.75−6.63 (m, 3H), 5.09 (q, J = 6.9 Hz, 1H), 4.78 (s, 1H), 3.89 (s, 3H), 1.48 (d, J = 6.9 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 199.0, 163.9, 146.6, 130.8, 129.3, 127.5, 117.7, 114.0, 113.4, 77.2, 55.5, 52.9, 19.8.

IR (KBr) v (cm^{-1}) 3391, 2918, 1679, 1601, 1506, 1312, 1258, 1156, 841, 750, 693.

HRMS (ESI) calcd for $C_{16}H_{18}NO_2^+$ $[M+H]^+$ m/z: 256.1332, found: 256.1339.



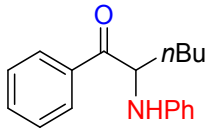
3fa

1-Phenyl-2-(phenylamino)pentan-1-one (**3fa**)[7] was synthesized according to General Procedure A from **1f** (58 mg, 0.4 mmol) and **2a** (56 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, then use preparative TLC to give 81 mg product **3fa** as a yellow oil in 80% yield.

3fa: R_f = 0.28 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, J = 7.6 Hz, 2H), 7.62 (t, J = 7.4 Hz, 1H), 7.52 (t, J = 7.7 Hz, 2H), 7.18 (t, J = 7.7 Hz, 2H), 6.72 (dd, J = 12.1, 7.9 Hz, 3H), 5.10 (s, 1H), 4.67 (s, 1H), 2.03−1.90 (m, 1H), 1.75−1.61 (m, 1H), 1.46 (ddd, J = 12.2, 8.7, 4.7 Hz, 2H), 0.91 (t, J = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 200.8, 147.2, 135.3, 133.5, 129.3, 128.8, 128.3 117.9 113.5, 57.8, 35.4, 18.5, 14.0.



3ga

1-Phenyl-2-(phenylamino)hexan-1-one (**3ga**) was synthesized according to General Procedure A from **1g** (63 mg, 0.4 mmol) and **2a** (56 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 81 mg product **3ga** as a yellow oil in 76% yield.

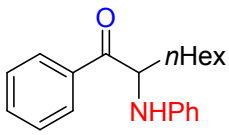
3ga: R_f = 0.40 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04−7.97 (m, 2H), 7.61 (t, J = 7.4 Hz, 1H), 7.51 (t, J = 7.6 Hz, 2H), 7.22−7.13 (m, 2H), 6.77−6.65 (m, 3H), 5.08 (dd, J = 6.5, 4.8 Hz, 1H), 4.65 (s, 1H), 2.02−1.94 (m, 1H), 1.71−1.63 (m, 1H), 1.46−1.22 (m, 4H), 0.84 (t, J = 7.1 Hz, 3H).

^{13}C NMR (101 MHz, DMSO) δ 200.8, 147.2, 135.3, 133.5, 129.3, 129.1, 128.8, 128.3, 117.9, 113.6, 57.9, 33.0, 27.3, 22.6, 13.8.

IR (KBr) v (cm^{-1}) 3393, 2930, 1683, 1602, 1506, 1448, 1316, 1154, 747, 690.

HRMS (ESI) calcd for $C_{18}H_{22}NO^+$ $[M+H]^+$ m/z: 268.1696, found: 268.1705.



3ha

1-Phenyl-2-(phenylamino)octan-1-one (**3ha**) was synthesized according to General Procedure A from **1h** (75 mg, 0.4 mmol) and **2a** (56 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 96 mg product **3ha** as a yellow oil in 81% yield.

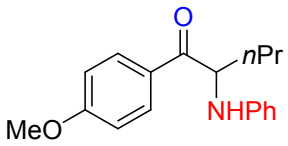
3ha: R_f = 0.37 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, J = 7.5 Hz, 2H), 7.61 (t, J = 7.4 Hz, 1H), 7.51 (t, J = 7.6 Hz, 2H), 7.17 (t, J = 7.8 Hz, 2H), 6.78−6.63 (m, 3H), 5.08 (dd, J = 6.0, 5.2 Hz, 1H), 4.66 (s, 1H), 2.11−1.87 (m, 1H), 1.77−1.63 (m, 1H), 1.48−1.18 (m, 8H), 0.84 (t, J = 6.8 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 200.8, 147.2, 135.3, 133.5, 129.3, 128.8, 128.3, 117.9, 113.6, 58.0, 33.2, 31.5, 29.1, 25.1, 22.5, 14.0.

IR (KBr) v (cm^{-1}) 3395, 3054, 2928, 2856, 1686, 1603, 1508, 1449, 1318, 1261, 749, 692.

HRMS (ESI) calcd for $C_{20}H_{26}NO^+$ $[M+H]^+$ m/z: 296.2009, found: 296.2008.



3ia

1-(4-Methoxyphenyl)-2-(phenylamino)pentan-1-one (**3ia**) was synthesized according to General Procedure A from **1i** (70 mg, 0.4 mmol) and **2a** (56 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 10/1, then use preparative TLC to give 48 mg product **3ia** as a yellow oil in 42% yield.

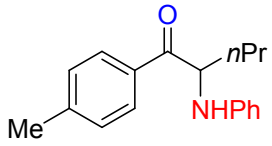
3ia: R_f = 0.60 (petroleum ether/ethyl acetate = 5:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, J = 8.8 Hz, 2H), 7.17 (t, J = 7.2 Hz, 2H), 7.01−6.93 (m, 2H), 6.74−6.66 (m, 3H), 5.05 (dd, J = 6.3, 5.0 Hz, 1H), 4.68 (s, 1H), 3.88 (s, 3H), 2.00−1.90 (m, 1H), 1.73−1.63 (m, 1H), 1.53−1.34 (m, 2H), 0.91 (t, J = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 199.1, 163.8, 147.3, 130.6, 129.3, 128.1, 117.7, 114.0, 113.5, 57.3, 55.4, 35.6, 18.5, 14.0.

IR (KBr) ν (cm^{-1}) 3387, 2959, 1674, 1602, 1509, 1312, 1259, 1175, 841, 750, 693.

HRMS (ESI) calcd for $C_{18}H_{22}NO_2^+$ $[M+H]^+$ *m/z*: 284.1645, found: 284.1651.



3ja

2-(Phenylamino)-1-p-tolylpentan-1-one (**3ja**) was synthesized according to General Procedure A from **1j** (63 mg, 0.4 mmol) and **2a** (56 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 81 mg product **3ja** as a yellow solid in 76% yield.

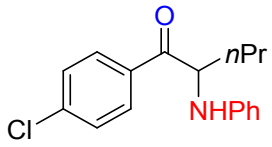
3ja: R_f = 0.72 (petroleum ether/ethyl acetate = 5:1), mp 66-67 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, J = 8.1 Hz, 2H), 7.31 (d, J = 8.1 Hz, 2H), 7.17 (t, J = 7.8 Hz, 2H), 6.75−6.66 (m, 3H), 5.08 (t, J = 4.9 Hz, 1H), 4.68 (s, 1H), 2.44 (s, 3H), 2.01−1.90 (m, 1H), 1.72−1.63 (m, 1H), 1.52−1.34 (m, 2H), 0.90 (t, J = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 200.3, 147.2, 144.4, 132.7, 129.5, 129.3, 128.4, 117.7, 113.5, 57.6, 35.5, 21.7, 18.5, 14.0.

IR (KBr) ν (cm^{-1}) 3394, 3052, 2959, 2872, 1680, 1604, 1505, 1314, 1155, 1000, 823, 749, 692.

HRMS (ESI) calcd for $C_{18}H_{22}NO^+$ $[M+H]^+$ *m/z*: 268.1696, found: 268.1702.

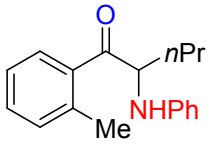


3ka

1-(4-Chlorophenyl)-2-(phenylamino)pentan-1-one (**3ka**) was synthesized according to General Procedure A from **1k** (71 mg, 0.4 mmol) and **2a** (56 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 90 mg product **3ka** as a yellow solid in 78% yield.

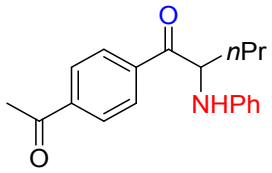
3ka: R_f = 0.72 (petroleum ether/ethyl acetate = 5:1), mp 108-110 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98−7.89 (m, 2H), 7.53−7.44 (m, 2H), 7.19−7.10 (m, 2H), 6.71 (t, J = 7.3 Hz, 1H), 6.68−6.61 (m, 2H), 5.00 (t, J = 5.0 Hz, 1H), 4.58 (s, 1H), 1.99−1.86 (m, 1H), 1.71−1.60 (m, 1H), 1.50−1.30 (m, 2H), 0.89 (t, J = 7.3 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 199.7, 147.0, 140.0, 133.6, 129.7, 129.4, 129.2, 118.1, 113.5, 57.9, 35.3, 18.56, 14.0.
IR (KBr) v (cm^{-1}) 3397, 3054, 2960, 2873, 1685, 1602, 1505, 1400, 1312, 1092, 995, 840, 749, 693.
HRMS (ESI) calcd for $C_{17}H_{19}NClO^+$ $[M+H]^+$ m/z: 288.1150, found: 288.1157.



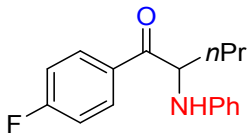
3la

2-(Phenylamino)-1-o-tolylpentan-1-one (**3la**) was synthesized according to General Procedure A from **1l** (63 mg, 0.4 mmol) and **2a** (56 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 82 mg product **3la** as a yellow oil in 77% yield.
3la: R_f = 0.68 (petroleum ether/ethyl acetate = 5:1)
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, J = 8.1 Hz, 2H), 7.32 (d, J = 8.1 Hz, 2H), 7.18 (t, J = 7.9 Hz, 2H), 6.76−6.66 (m, 3H), 5.09 (t, J = 5.0 Hz, 1H), 4.69 (s, 1H), 2.45 (s, 3H), 2.01−1.91 (m, 1H), 1.74−1.64 (m, 1H), 1.53−1.35 (m, 2H), 0.91 (t, J = 7.3 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 200.3, 147.2, 144.4, 132.7, 129.5, 129.3, 128.4, 117.7, 113.5, 57.6, 35.5, 21.6, 18.5, 14.0.
IR (KBr) v (cm^{-1}) 3395, 2959, 1681, 1604, 1505, 1314, 1000, 748, 692.
HRMS (ESI) calcd for $C_{18}H_{22}NO^+$ $[M+H]^+$ m/z: 268.1696, found: 268.1703.



3ma

1-(4-Acetylphenyl)-2-(phenylamino)pentan-1-one (**3ma**) was synthesized according to General Procedure A by using Ph_2SO (0.8 mmol), Tf_2O (0.8 mmol), **1m** (75 mg, 0.4 mmol) and **2a** (56 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 71 mg product **3ma** as a yellow solid in 60% yield.
3ma: R_f = 0.40 (petroleum ether/ethyl acetate = 5:1), mp 85-87 °C
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (s, 4H), 7.17 (dd, J = 8.4, 7.4 Hz, 2H), 6.72 (t, J = 7.3 Hz, 1H), 6.70−6.65 (m, 2H), 5.07(t, J = 5.2 Hz, 1H), 4.60 (s, 1H), 2.65 (s, 3H), 2.00−1.90 (m, 1H), 1.72−1.61 (m, 1H), 1.54−1.33 (m, 2H), 0.90 (t, J = 7.3 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 200.5, 197.3, 146.9, 140.4, 138.5, 129.3, 128.6, 128.5, 118.1, 113.5, 58.3, 35.1, 26.8, 18.5, 13.9.
IR (KBr) v (cm^{-1}) 3392, 2960, 1686, 16023, 1501, 1310, 1263, 995, 750, 693.
HRMS (ESI) calcd for $C_{19}H_{22}NO_2^+$ $[M+H]^+$ m/z: 296.1645, found: 296.1642.



3na

1-(4-Fluorophenyl)-2-(phenylamino)pentan-1-one (**3na**) was synthesized according to General Procedure A from **1n** (65 mg, 0.4 mmol) and **2a** (56 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 80 mg product **3na** as a yellow oil in 74% yield.

3na: R_f = 0.33 (petroleum ether/ethyl acetate = 5:1).

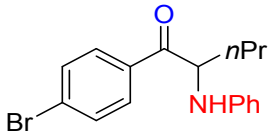
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08−8.00 (m, 2H), 7.21−7.13 (m, 4H), 6.72 (t, J = 7.3 Hz, 1H), 6.68 (d, J = 7.8 Hz, 2H), 5.03 (dd, J = 12.0, 7.0 Hz, 1H), 4.60 (d, J = 7.0 Hz, 1H), 1.99−1.90 (m, 1H), 1.71−1.60 (m, 1H), 1.53−1.34 (m, 2H), 0.91 (t, J = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 199.2, 165.9 (d, J = 255.7 Hz), 147.1, 131.7 (d, J = 3.1 Hz), 131.0 (d, J = 9.3 Hz), 129.4, 118.0, 116.0 (d, J = 21.9 Hz), 113.5, 57.8, 35.4, 18.6, 14.0.

^{19}F NMR (376 MHz, $CDCl_3$) δ -104.15.

IR (KBr) v (cm^{-1}) 3396, 3053, 2960. 2873, 1686, 1600, 1508, 1410, 1312, 1232, 1154, 846, 749, 693.

HRMS (ESI) calcd for $C_{17}H_{19}NFO^+$ $[M+H]^+$ *m/z*: 272.1445, found: 272.1453.



3oa

1-(4-Bromophenyl)-2-(phenylamino)pentan-1-one (**3oa**) was synthesized according to General Procedure A from **1o** (89 mg, 0.4 mmol) and **2a** (56 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 103 mg product **3oa** as a yellow solid in 78% yield.

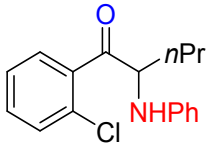
3oa: R_f = 0.79 (petroleum ether/ethyl acetate = 5:1), mp 119-121 ^{o}C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, J = 8.5 Hz, 2H), 7.65 (d, J = 8.6 Hz, 2H), 7.17 (t, J = 7.7 Hz, 2H), 6.73 (t, J = 7.2 Hz, 1H), 6.68 (d, J = 8.2 Hz, 2H), 5.02 (t, J = 5.0 Hz, 1H), 4.60 (s, 1H), 2.00−1.87 (m, 1H), 1.71−1.60 (m, 1H), 1.51−1.33 (m, 2H), 0.90 (t, J = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 199.9, 147.0, 134.0, 132.2, 129. 8, 129.4, 128.7, 118.0, 113.5, 57.9, 35.3, 18.5, 13.9.

IR (KBr) v (cm^{-1}) 3397, 2959, 1685, 1602, 1585, 1505, 1396, 1072, 993, 748, 692.

HRMS (ESI) calcd for $C_{17}H_{19}NBrO^+$ $[M+H]^+$ *m/z*: 332.0645, found: 332.0641.



3pa

1-(2-Chlorophenyl)-2-(phenylamino)pentan-1-one (**3pa**) was synthesized according to General Procedure A from **1p** (71 mg, 0.4 mmol) and **2a** (56 mg, 0.6 mmol), eluted by petroleum ether/ethyl

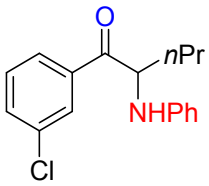
acetate = 100/1, to give 89 mg product **3pa** as a yellow oil in 77% yield.

3pa: R_f = 0.33 (petroleum ether/ethyl acetate = 20:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50−7.44 (m, 2H), 7.44−7.38 (m, 1H), 7.33 (td, J = 7.4, 1.4 Hz, 1H), 7.19 (t, J = 7.9 Hz, 2H), 6.73 (dd, J = 18.2, 7.6 Hz, 3H), 5.00 (dd, J = 6.6, 5.1 Hz, 1H), 4.53 (s, 1H), 1.96−1.87 (m, 1H), 1.65−1.55 (m, 1H), 1.50−1.36 (m, 2H), 0.88 (t, J = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 203.1, 147.0, 137.5, 132.0, 131.3, 130.7, 129.3, 129.1, 126.9, 117.9, 113.6, 61.5, 34.0, 18.5, 13.9.

HRMS (ESI) calcd for $C_{17}H_{19}NClO^+$ $[M+H]^+$ *m/z*: 288.1150, found: 288.1156.



3qa

1-(3-Chlorophenyl)-2-(phenylamino)pentan-1-one (**3qa**) was synthesized according to General Procedure A from **1q** (71 mg, 0.4 mmol) and **2a** (56 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 76 mg product **3qa** as a yellow oil in 66% yield.

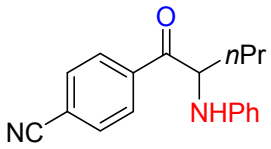
3qa: R_f = 0.38 (petroleum ether/ethyl acetate = 20:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (s, 1H), 7.88 (d, J = 7.8 Hz, 1H), 7.62−7.54 (m, 1H), 7.45 (t, J = 7.9 Hz, 1H), 7.18 (t, J = 7.9 Hz, 2H), 6.74 (t, J = 7.3 Hz, 1H), 6.69 (d, J = 7.8 Hz, 2H), 5.02 (s, 1H), 4.59 (s, 1H), 2.01−1.90 (m, 1H), 1.71−1.61 (m, 1H), 1.53−1.34 (m, 2H), 0.92 (t, J = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 199.7, 146.9, 136.8, 135.2, 133.4, 130.2, 129.4, 128.4, 126.3, 118.1, 113.6, 58.1, 35.2, 18.5, 13.9.

IR (KBr) ν (cm^{-1}) 3398, 3054, 2960, 2872, 1687, 1603, 1509, 1420, 1315, 1156, 800, 750, 692.

HRMS (ESI) calcd for $C_{17}H_{19}NClO^+$ $[M+H]^+$ *m/z*: 288.1150, found: 288.1158.



3ra

4-(2-(Phenylamino)pentanoyl)benzonitrile (**3ra**) was synthesized according to General Procedure A from **1r** (68 mg, 0.4 mmol) and **2a** (56 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 50/1, to give 23 mg product **3ra** as a yellow oil in 21% yield.

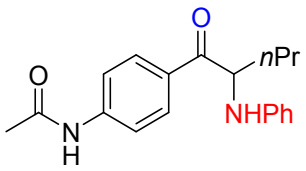
3ra: R_f = 0.55 (petroleum ether/ethyl acetate = 20:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, J = 8.1 Hz, 2H), 7.80 (d, J = 8.1 Hz, 2H), 7.17 (t, J = 7.7 Hz, 2H), 6.74 (t, J = 7.3 Hz, 1H), 6.66 (d, J = 8.0 Hz, 2H), 5.01 (dd, J = 12.3, 7.2 Hz, 1H), 4.50 (d, J = 7.9 Hz, 1H), 1.99−1.86 (m, 1H), 1.71−1.61 (m, 1H), 1.51−1.34 (m, 2H), 0.91 (t, J = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 199.9, 146.7, 138.4, 132.7, 129. 5, 128.7, 118.4, 117.8, 116.8, 113.6, 58.5, 35.0, 18.6, 13.9.

IR (KBr) ν (cm^{-1}) 3391, 2961, 2922, 2230, 1691, 1602, 1602, 1501, 1076, 750, 696.

HRMS (ESI) calcd for $C_{18}H_{19}N_2O^+$ $[M+H]^+$ *m/z*: 279.1492, found: 279.1496.



3sa

N-(4-(2-(Phenylamino)pentanoyl)phenyl)acetamide (**3sa**) was synthesized according to General Procedure A from **1s** (81 mg, 0.4 mmol) and **2a** (56 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 5/1, to give 65 mg product **3sa** as a yellow oil in 52% yield.

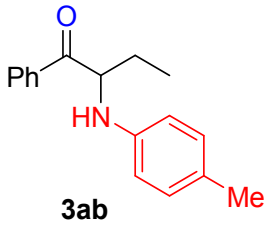
3sa: R_f = 0.19 (petroleum ether/ethyl acetate = 2:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (s, 1H), 7.97 (d, J = 8.7 Hz, 2H), 7.65 (d, J = 8.5 Hz, 2H), 7.14 (dd, J = 8.4, 7.5 Hz, 2H), 6.73−6.61 (m, 3H), 5.03 (dd, J = 5.1, 5.9 Hz, 1H), 4.59 (s, 1H), 2.18 (s, 3H), 1.97−1.88 (m, 1H), 1.70−1.60 (m, 1H), 1.50−1.32 (m, 2H), 0.89 (t, J = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 199.8, 168.9, 147.1, 142.8, 130.7, 129.7, 129.3, 119.1, 117.9, 113.5, 57.5, 35.5, 24.6, 18.6, 13.9.

IR (KBr) ν (cm^{-1}) 3324, 2961, 2934, 1677, 1599, 1528, 1408, 1317, 749.

HRMS (ESI) calcd for $C_{19}H_{23}N_2O_2^+$ $[M+H]^+$ *m/z*: 311.1154, found: 311.1156.



3ab

1-Phenyl-2-(p-tolylamino)butan-1-one (**3ab**)[8] was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **2b** (64 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 63 mg product **3ab** as a yellow solid in 62% yield.

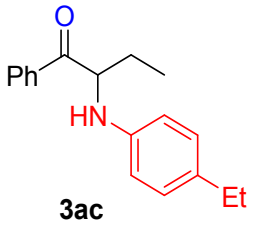
3ab: R_f = 0.59 (petroleum ether/ethyl acetate = 5:1), mp 64-66 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05−7.96 (m, 2H), 7.61 (t, J = 7.4 Hz, 1H), 7.51 (t, J = 7.7 Hz, 2H), 7.01 (d, J = 7.5 Hz, 2H), 6.71−6.59 (m, 2H), 5.07 (t, J = 4.5 Hz, 1H), 4.60 (s, 1H), 2.25 (s, 3H), 2.14−2.02 (m, 1H), 1.80−1.68 (m, 1H), 0.94 (td, J = 7.4, 1.2 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 200.6, 144.7, 135.3, 133.4, 129.8, 128.8, 128.3, 127.0, 113.8, 59.1, 25.9, 20.3, 9.2.

IR (KBr) ν (cm^{-1}) 3392, 2969, 1684, 1619, 1519, 1448, 1316, 1155, 979, 808, 700.

HRMS (ESI) calcd for $C_{17}H_{20}ON^+$ $[M+H]^+$ *m/z*: 254.1539, found: 254.1544.



3ac

2-(4-Ethylphenylamino)-1-phenylbutan-1-one (**3ac**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **2c** (73 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 72 mg product **3ac** as a yellow solid in 67% yield.

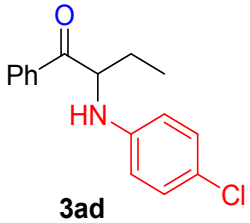
3ac: R_f = 0.61 (petroleum ether/ethyl acetate = 5:1), mp 52-54 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, J = 7.6 Hz, 2H), 7.61 (t, J = 7.4 Hz, 1H), 7.51 (t, J = 7.6 Hz, 2H), 7.03 (d, J = 8.1 Hz, 2H), 6.67 (d, J = 8.3 Hz, 2H), 5.07 (t, J = 5.0 Hz, 1H), 4.63 (s, 1H), 2.55 (q, J = 7.6 Hz, 2H), 2.18−2.02 (m, 1H), 1.82−1.68 (m, 1H), 1.20 (t, J = 7.6 Hz, 3H), 0.94 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 200.7, 145.0, 135.3, 133.6, 133.4, 128.8, 128.6, 128.3, 113.7, 59.1, 27.9, 25.9, 15.8, 9.2.

IR (KBr) v (cm^{-1}) 3396, 2965, 1685, 1617, 1519, 1449, 1315, 1156, 979, 821, 700.

HRMS (ESI) calcd for $C_{18}H_{22}ON^+$ $[M+H]^+$ m/z: 268.1696, found: 268.1699.



3ad

2-(4-Chlorophenylamino)-1-phenylbutan-1-one (**3ad**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **2d** (77 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 50/1, to give 80 mg product **3ad** as a yellow solid in 73% yield.

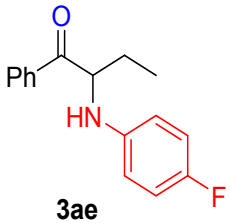
3ad: R_f = 0.58 (petroleum ether/ethyl acetate = 5:1), mp 65-67 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, J = 7.5 Hz, 2H), 7.62 (t, J = 7.4 Hz, 1H), 7.51 (t, J = 7.6 Hz, 2H), 7.16−7.07 (m, 2H), 6.67−6.56 (m, 2H), 5.03 (t, J = 4.9 Hz, 1H), 4.76 (s, 1H), 2.07 (dqd, J = 14.7, 7.4, 4.9 Hz, 1H), 1.86−1.56 (m, 1H), 0.90 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 200.0, 145.6, 135.0, 133.7, 129.1, 128.9, 128.3, 122.3, 114.6, 58.7, 25.7, 9.1.

IR (KBr) v (cm^{-1}) 3396, 3096, 2969, 2934, 1686, 1599, 1509, 1448, 1316, 1093, 979, 816, 700.

HRMS (ESI) calcd for $C_{16}H_{17}OClN^+$ $[M+H]^+$ m/z: 274.0993, found: 274.0998.



3ae

2-(4-Fluorophenylamino)-1-phenylbutan-1-one (**3ae**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **2e** (67 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 71 mg product **3ae** as a yellow solid in 69% yield.

3ae: R_f = 0.62 (petroleum ether/ethyl acetate = 5:1), mp 64-66 °C

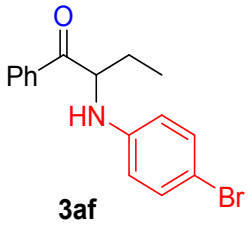
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02−7.96 (m, 2H), 7.65−7.58 (m, 1H), 7.54−7.47 (m, 2H), 6.91−6.84 (m, 2H), 6.67−6.60 (m, 2H), 4.99 (t, J = 5.4 Hz, 1H), 4.58 (s, 1H), 2.05 (dqd, J = 14.8, 7.4, 4.8 Hz, 1H), 1.78−1.66 (m, 1H), 0.92 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 200.5, 157.2, 154.8, 143.5, 135.2, 133.6, 128.9, 128.3, 115.9, 115.6, 114.7, 114.6, 59.6, 25.9, 9.3.

^{19}F NMR (377 MHz, $CDCl_3$) δ -127.38.

IR (KBr) v (cm^{-1}) 3391, 3060, 2969. 1682, 1597, 1515, 1448, 1219, 1156, 979, 821, 699.

HRMS (ESI) calcd for $C_{16}H_{17}OFN^+$ $[M+H]^+$ m/z: 258.1289, found: 258.1294.



3af

2-(4-Bromophenylamino)-1-phenylbutan-1-one (**3af**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **2f** (103 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 50/1, to give 83 mg product **3af** as a yellow oil in 65% yield.

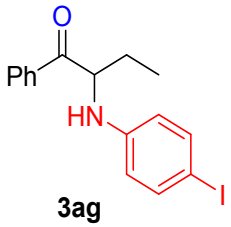
3af: R_f = 0.54 (petroleum ether/ethyl acetate = 5:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01−7.94 (m, 2H), 7.63−7.57 (m, 1H), 7.53−7.46 (m, 2H), 7.27−7.20 (m, 2H), 6.60−6.52 (m, 2H), 5.02 (q, J = 5.2 Hz, 1H), 4.77 (d, J = 4.4 Hz, 1H), 2.06 (dqd, J = 14.8, 7.4, 4.9 Hz, 1H), 1.78−1.66 (m, 1H), 0.87 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 199.9, 145.9, 135.0, 133.7, 132.0, 128.9, 128.3, 115.1, 109.3, 58.6, 25.6, 9.0.

IR (KBr) ν (cm^{-1}) 3395, 2968, 1684, 1596, 1500, 1315, 979, 813, 700.

HRMS (ESI) calcd for $C_{16}H_{17}OBrN^+$ $[M+H]^+$ *m/z*: 318.0488, found: 318.0493.



3ag

2-(4-Iodophenylamino)-1-phenylbutan-1-one (**3ag**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **2g** (131 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 110 mg product **3ag** as a yellow solid in 75% yield.

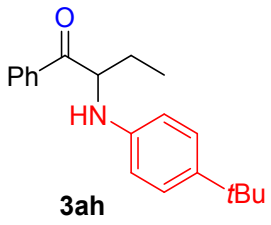
3ag: R_f = 0.62 (petroleum ether/ethyl acetate = 5:1), mp 106-108 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03−7.95 (m, 2H), 7.66−7.58 (m, 1H), 7.54−7.48 (m, 2H), 7.45−7.38 (m, 2H), 6.52−6.45 (m, 2H), 5.03 (t, J = 5.3 Hz, 1H), 4.79 (s, 1H), 2.07 (dqd, J = 14.8, 7.5, 4.9 Hz, 1H), 1.79−1.67 (m, 1H), 0.88 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 199.8, 146.5, 137.9, 135.0, 133.7, 128.9, 128.3, 115.7, 78.3, 58.4, 25.6, 9.0.

IR (KBr) ν (cm^{-1}) 3394, 2967, 1683, 1591, 1499, 1448, 1316, 1247, 811, 700.

HRMS (ESI) calcd for $C_{16}H_{17}OIN^+$ $[M+H]^+$ *m/z*: 366.0349, found: 366.0353.

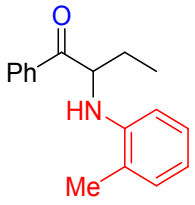


3ah

2-(4-*tert*-Butylphenylamino)-1-phenylbutan-1-one (**3ah**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **2h** (90 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 50/1, to give 63 mg product **3ah** as a yellow oil in 53% yield.

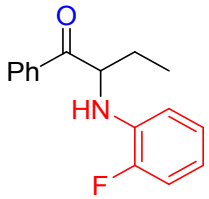
3ah: R_f = 0.32 (petroleum ether/ethyl acetate = 20:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06−7.98 (m, 2H), 7.65−7.58 (m, 1H), 7.56−7.48 (m, 2H), 7.22 (d, J = 8.6 Hz, 2H), 6.68 (d, J = 8.6 Hz, 2H), 5.06 (t, J = 5.4 Hz, 1H), 4.64 (s, 1H), 2.08 (dqd, J = 14.7, 7.4, 4.9 Hz, 1H), 1.81−1.66 (m, 1H), 1.29 (s, 9H), 0.94 (t, J = 7.4 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 200.7, 144.7, 140.5, 135.3, 133.4, 128.8, 128.3, 126.1, 113.2, 59.1, 33.8, 31.5, 26.1, 9.3.
IR (KBr) v (cm^{-1}) 3303, 2963, 1694, 1602, 1519, 1364, 1267, 836, 713.
HRMS (ESI) calcd for $C_{20}H_{26}ON^+$ $[M+H]^+$ m/z: 296.2009, found: 296.2012.



3ai

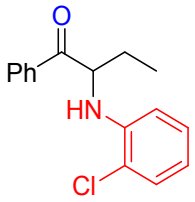
1-Phenyl-2-(o-tolylamino)butan-1-one (**3ai**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **2i** (64 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 50/1, to give 80 mg product **3ai** as a yellow solid in 79% yield.
3ai: R_f = 0.54 (petroleum ether/ethyl acetate = 5:1), mp 100-102 °C
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06−7.98 (m, 2H), 7.63 (t, J = 7.4 Hz, 1H), 7.52 (t, J = 7.8 Hz, 2H), 7.11 (t, J = 6.7 Hz, 2H), 6.72−6.61 (m, 2H), 5.16 (t, J = 4.9 Hz, 1H), 4.70 (s, 1H), 2.30 (s, 3H), 2.22−2.06 (m, 1H), 1.85−1.72 (m, 1H), 0.95−0.88 (m, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 200.5, 144.9, 135.2, 133.5, 130.4, 128.8, 128.3, 127.0, 122.8, 117.3, 110.2, 58.6, 25.8, 17.5, 9.0.
IR (KBr) v (cm^{-1})3419, 3060, 2968, 2933, 1685, 1605, 1511, 1447, 1316, 1160, 980, 746, 700.
HRMS (ESI) calcd for $C_{17}H_{20}ON^+$ $[M+H]^+$ m/z: 254.1539, found: 254.1548.



3aj

2-(2-Fluorophenylamino)-1-phenylbutan-1-one (**3aj**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **2j** (67 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 63 mg product **3aj** as a yellow solid in 61% yield.
3aj: R_f = 0.64 (petroleum ether/ethyl acetate = 5:1), mp 78-80 °C
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05−7.96 (m, 2H), 7.62 (t, J = 7.4 Hz, 1H), 7.51 (t, J = 7.6 Hz, 2H), 7.04−6.91 (m, 2H), 6.76−6.59 (m, 2H), 5.08 (d, J = 3.0 Hz, 1H), 4.93 (s, 1H), 2.09 (dqd, J = 14.8, 7.4, 4.8 Hz, 1H), 1.85−1.70 (m, 1H), 0.94 (t, J = 7.4 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 199.8, 151.9 (d, J = 239.3 Hz), 135.5 (d, J = 11.5 Hz), 135.1, 133.6, 128.9, 128.3, 124.4 (d, J = 3.5 Hz), 117.3 (d, J = 7.0 Hz), 114.9 (d, J = 18.7 Hz), 112.8 (d, J = 3.2 Hz), 58.6, 25.9, 9.2.
^{19}F NMR (376 MHz, $CDCl_3$) δ −135.00.
IR (KBr) v (cm^{-1}) 3414, 3066, 2969, 1686, 1620, 1516, 1448, 1335, 1252, 1191, 979, 742, 700.

HRMS (ESI) calcd for $C_{16}H_{17}OFN^+$ $[M+H]^+$ *m/z*: 258.1289, found: 258.1295.



3ak

2-(2-Chlorophenylamino)-1-phenylbutan-1-one (**3ak**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **2k** (77 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 63 mg product **3ak** as a yellow oil in 58% yield.

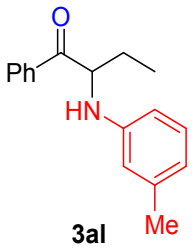
3ak: R_f = 0.70 (petroleum ether/ethyl acetate = 5:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05−7.96 (m, 2H), 7.62 (t, J = 7.4 Hz, 1H), 7.52 (t, J = 7.6 Hz, 2H), 7.29 (dd, J = 7.8, 1.3 Hz, 1H), 7.14−7.06 (m, 1H), 6.70−6.59 (m, 2H), 5.41 (d, J = 7.4 Hz, 1H), 5.12 (dt, J = 7.4, 5.4 Hz, 1H), 2.12 (dqd, J = 14.9, 7.5, 5.0 Hz, 1H), 1.87−1.75 (m, 1H), 0.92 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 199.4, 142.8, 135.0, 133.6, 129.5, 128.9, 128.3, 127.6, 119.9, 117.6, 111.6, 58.5, 25.6, 9.0.

IR (KBr) ν (cm^{-1}) 3396, 2969, 1685, 1597, 1509, 1449, 1323, 1247, 1035, 980, 742, 698.

HRMS (ESI) calcd for $C_{16}H_{17}OClN^+$ $[M+H]^+$ *m/z*: 274.0993, found: 274.0998.



3al

1-Phenyl-2-(m-tolylamino)butan-1-one (**3al**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **2l** (64 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 54 mg product **3al** as a yellow solid in 53% yield.

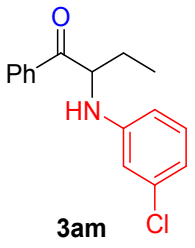
3al: R_f = 0.69 (petroleum ether/ethyl acetate = 5:1), mp 75-78 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05−7.97 (m, 2H), 7.62 (t, J = 7.4 Hz, 1H), 7.52 (t, J = 7.6 Hz, 2H), 7.08 (t, J = 7.9 Hz, 1H), 6.59−6.49 (m, 3H), 5.09 (t, J = 4.6 Hz, 1H), 4.70 (s, 1H), 2.29 (s, 3H), 2.16−2.03 (m, 1H), 1.80−1.68 (m, 1H), 0.92 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 200.4, 147.0, 139.1, 135.3, 133.5, 129.2, 128.8, 128.3, 118.7, 114.4, 110.5, 58.7, 25.9, 21.6, 9.1.

IR (KBr) ν (cm^{-1}) 3396, 3043, 2968, 1682, 1605, 1491, 1448, 1326, 1245, 1179, 979, 844, 770, 700.

HRMS (ESI) calcd for $C_{17}H_{20}ON^+$ $[M+H]^+$ *m/z*: 254.1539, found: 254.1545.



3am

2-(3-Chlorophenylamino)-1-phenylbutan-1-one (**3am**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **2m** (77 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 79 mg product **3am** as a yellow oil in 72% yield.

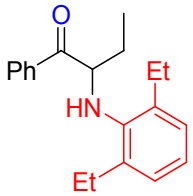
3am: R_f = 0.65 (petroleum ether/ethyl acetate = 5:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04−7.95 (m, 2H), 7.62 (t, J = 7.4 Hz, 1H), 7.52 (t, J = 7.6 Hz, 2H), 7.07 (t, J = 8.3 Hz, 1H), 6.71−6.65 (m, 2H), 6.57 (dd, J = 8.2, 1.5 Hz, 1H), 5.06 (dt, J = 7.6, 5.3 Hz, 1H), 4.89 (d, J = 7.5 Hz, 1H), 2.09 (dqd, J = 14.8, 7.5, 4.9 Hz, 1H), 1.81−1.67 (m, 1H), 0.88 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 199.7, 148.1, 135.0, 134.9, 133.7, 130.3, 128.9, 128.3, 117.5, 113.0, 111.8, 58.3, 25.6, 8.9.

IR (KBr) v (cm^{-1}) 3393, 3063, 1969, 2934, 2876, 1682, 1598, 1505, 1448, 1245, 1154, 988, 767, 700.

HRMS (ESI) calcd for $C_{16}H_{17}OClN^+$ $[M+H]^+$ *m/z*: 274.0993, found: 274.0999.



3an

2-(2,6-Diethylphenylamino)-1-phenylbutan-1-one (**3an**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **2n** (90 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 56 mg product **3an** as a yellow oil in 47% yield.

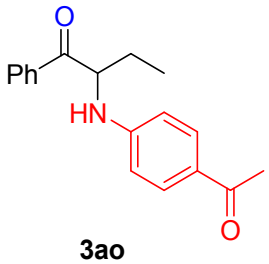
3an: R_f = 0.57 (petroleum ether/ethyl acetate = 5:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, J = 7.7 Hz, 2H), 7.59 (t, J = 7.3 Hz, 1H), 7.47 (t, J = 7.6 Hz, 2H), 7.05 (d, J = 7.5 Hz, 2H), 6.92 (t, J = 7.5 Hz, 1H), 4.93 (t, J = 5.6 Hz, 1H), 4.46 (s, 1H), 2.81 (q, J = 7.6 Hz, 4H), 1.96 (ddq, J = 14.1, 7.2, 7.2 Hz, 1H), 1.87−1.73 (m, 1H), 1.33 (t, J = 7.5 Hz, 6H), 0.88 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 202.5, 143.5, 136.0, 134.9, 133.3, 128.7, 128.2, 126.6, 121.8, 62.1, 26.7, 24.7, 14.4, 9.4.

IR (KBr) v (cm^{-1}) 3379, 2968, 1694, 1451, 1318, 1285, 1168, 1070, 895, 763, 713.

HRMS (ESI) calcd for $C_{20}H_{26}NO^+$ $[M+H]^+$ *m/z*: 296.2009, found: 296.2014.



3ao

2-(4-Acetylphenylamino)-1-phenylbutan-1-one (**3ao**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **2o** (81 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 10/1, to give 78 mg product **3ao** as a yellow oil in 69% yield.

3ao: R_f = 0.21 (petroleum ether/ethyl acetate = 5:1).

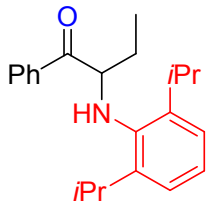
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05−7.95 (m, 2H), 7.83 (d, J = 8.8 Hz, 2H), 7.66−7.58 (m, 1H), 7.52

(t, J = 7.6 Hz, 2H), 6.66 (d, J = 8.8 Hz, 2H), 5.35 (d, J = 7.4 Hz, 1H), 5.18 (dt, J = 7.5, 5.2 Hz, 1H), 2.49 (s, 3H), 2.13 (dqd, J = 14.8, 7.5, 5.0 Hz, 1H), 1.87−1.73 (m, 1H), 0.86 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 199.0, 196.2, 150.7, 134.7, 133.9, 130.8, 129.0, 128.4, 127.0, 112.0, 57.6, 26.0, 25.5, 8.7.

IR (KBr) v (cm^{-1}) 3379, 2970, 2933, 1656, 1599, 1527, 1359, 1279, 1181, 826, 700.

HRMS (ESI) calcd for $C_{18}H_{20}O_2N^+$ $[M+H]^+$ m/z: 282.1489, found: 282.1494.



3ap

2-(2,6-Diisopropylphenylamino)-1-phenylbutan-1-one (**3ap**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **2p** (106 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 10/1, then use preparative TLC to give 70 mg product **3ap** as a yellow oil in 54% yield.

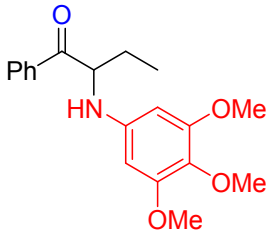
3ap: R_f = 0.57 (petroleum ether/ethyl acetate = 5:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87−7.81 (m, 2H), 7.58−7.52 (m, 1H), 7.46−77.40 (m, 2H), 7.09−7.05 (m, 2H), 7.03−6.97 (m, 1H), 4.72 (dd, J = 6.7, 5.1 Hz, 1H), 4.23 (s, 1H), 3.41 (hept, J = 6.8 Hz, 2H), 2.02−1.89 (m, 7.4 Hz, 1H), 1.81 (dqd, J = 14.8, 7.5, 5.1 Hz, 1H), 1.28 (d, J = 6.9 Hz, 6H), 1.22 (d, J = 6.8 Hz, 6H), 0.87 (t, J = 7.5 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 202.7, 141.7, 141.5, 136.3, 133.3, 128.7, 128.2, 123.6, 123.0, 64.0, 27.7, 26.8, 24.3, 24.1, 9.8.

IR (KBr) v (cm^{-1}) 2964, 1692, 1670, 1450, 1384, 1262, 1167, 894, 762, 713.

HRMS (ESI) calcd for $C_{22}H_{30}ON^+$ $[M+H]^+$ m/z: 324.2322, found: 324.2321.



3aq

1-Phenyl-2-(3,4,5-trimethoxyphenylamino)butan-1-one (**3aq**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **2q** (110 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 4/1, to give 93 mg product **3aq** as a yellow solid in 71% yield.

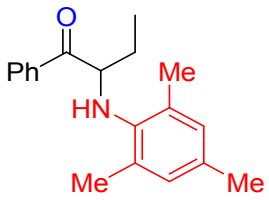
3aq: R_f = 0.39 (petroleum ether/ethyl acetate = 2:1), mp 128-130 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03−7.96 (m, 2H), 7.61 (t, J = 7.4 Hz, 1H), 7.50 (t, J = 7.6 Hz, 2H), 5.91 (s, 2H), 4.99 (t, J = 5.2 Hz, 1H), 4.60 (s, 1H), 3.77 (s, 6H), 3.74 (s, 3H), 2.05 (dqd, J = 14.7, 7.4, 5.2 Hz, 1H), 1.79−1.68 (m, 1H), 0.94 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 200.9, 153.9, 143.8, 135.3, 133.6, 130.5, 128.9, 128.2, 91.4, 61.0, 59.2, 55.9, 26.1, 9.4.

IR (KBr) v (cm^{-1}) 3373, 2936, 1683, 1609, 1510, 1450, 1235, 1126, 1004, 776, 700.

HRMS (ESI) calcd for $C_{19}H_{24}O_4N^+$ $[M+H]^+$ m/z: 330.1700, found: 330.1703.



3ar

2-(Mesitylamino)-1-phenylbutan-1-one (**3ar**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **2r** (81 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 81 mg product **3ar** as a yellow oil in 72% yield.

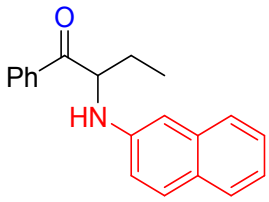
3ar: R_f = 0.70 (petroleum ether/ethyl acetate = 5:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93−7.80 (m, 2H), 7.56 (t, J = 7.4 Hz, 1H), 7.45 (t, J = 7.7 Hz, 2H), 6.79 (s, 2H), 4.94 (t, J = 5.6 Hz, 1H), 4.25 (s, 1H), 2.37 (s, 6H), 2.20 (s, 3H), 1.98−1.84 (m, 1H), 1.81−1.69 (m, 1H), 0.88 (t, J = 7.5 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 202.7, 142.0, 136.0, 133.3, 130.4, 129.7, 128.7, 128.3, 128.1, 61.25, 26.8, 20.4, 19.0, 9.4.

IR (KBr) v (cm^{-1}) 3363, 2919, 1701, 1679, 1491, 1450, 1378, 1271, 1026, 853, 713.

HRMS (ESI) calcd for $C_{19}H_{24}ON^+$ $[M+H]^+$ m/z: 282.1852, found: 282.1860.



3as

2-(Naphthalen-2-ylamino)-1-phenylbutan-1-one (**3as**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **2s** (86 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 76 mg product **3as** as a yellow solid in 66% yield.

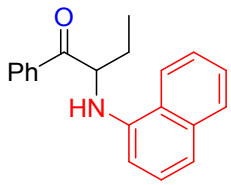
3as: R_f = 0.55 (petroleum ether/ethyl acetate = 5:1), mp 92-95 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09−8.02 (m, 2H), 7.70−7.64 (m, 2H), 7.64−7.58 (m, 2H), 7.53 (t, J = 7.6 Hz, 2H), 7.39−7.33 (m, 1H), 7.24−7.18 (m, 1H), 7.02 (dd, J = 8.8, 2.4 Hz, 1H), 6.88 (d, J = 2.2 Hz, 1H), 5.24 (d, J = 4.2 Hz, 1H), 4.96 (s, 1H), 2.21 (dqd, J = 14.8, 7.5, 5.0 Hz, 1H), 1.88−1.77 (m, 1H), 0.93 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 200.2, 144.6, 135.2, 135.1, 133.6, 129.2, 128.9, 128.4, 127.6, 126.3, 125.8, 122.1, 118.4, 105.0, 58.5, 25.5, 9.0.

IR (KBr) v (cm^{-1}) 3396, 3054, 2968, 1683, 1629, 1602, 1519, 1221, 827, 700.

HRMS (ESI) calcd for $C_{20}H_{20}ON^+$ $[M+H]^+$ m/z: 290.1539, found: 290.1544.



3at

2-(Naphthalen-1-ylamino)-1-phenylbutan-1-one (**3at**) was synthesized according to General

Procedure A from **1a** (52 mg, 0.4 mmol) and **2t** (86 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 83 mg product **3at** as a yellow solid in 72% yield.

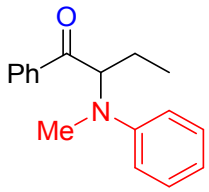
3at: R_f = 0.68(petroleum ether/ethyl acetate = 5:1), mp 103-105 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (d, J = 8.0 Hz, 3H), 7.82 (d, J = 7.5 Hz, 1H), 7.64 (t, J = 7.4 Hz, 1H), 7.58−7.46 (m, 4H), 7.34 (td, J = 8.1, 1.6 Hz, 1H), 7.27 (d, J = 7.3 Hz, 1H), 6.66 (d, J = 7.4 Hz, 1H), 5.64 (d, J = 5.8 Hz, 1H), 5.31 (dd, J = 11.6, 5.6 Hz, 1H), 2.32−2.19 (m, 1H), 1.96−1.83 (m, 1H), 0.96 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 200.4, 142.1, 135.1, 134.5, 133.6, 128.9, 128.6, 128.4, 126.3, 125.8, 124.8, 123.8, 120.3, 117.6, 104.8, 58.6, 25.5, 9.1.

IR (KBr) ν (cm^{-1}) 3423, 3060, 2968, 1682, 1581, 1525, 1480, 1448, 1409, 1337, 1252, 1150, 768, 700.

HRMS (ESI) calcd for $C_{20}H_{20}ON^+$ $[M+H]^+$ m/z: 290.1539, found: 290.1546.



3au

2-(Methyl(phenyl)amino)-1-phenylbutan-1-one (**3au**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **2u** (64 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 64 mg product **3au** as a yellow oil in 63% yield.

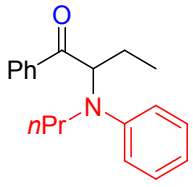
3au: R_f = 0.30 (petroleum ether/ethyl acetate = 5:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92−7.83 (m, 2H), 7.55−7.46 (m, 1H), 7.36 (t, J = 7.8 Hz, 2H), 7.33−7.27 (m, 2H), 6.85 (d, J = 8.3 Hz, 2H), 6.79 (t, J = 7.3 Hz, 1H), 5.07 (t, J = 7.1 Hz, 1H), 2.71 (s, 3H), 2.13 (ddq, J = 14.5, 7.3, 7.3Hz, 1H), 1.83 (ddq, J = 14.7, 7.4, 7.4 Hz, 1H), 1.00 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 198.9, 149.1, 136.3, 133.0, 129.4, 128.5, 128.1, 117.2, 112.6, 63.7, 32.5, 20.8, 11.1.

IR (KBr) ν (cm^{-1}) 2967, 1685, 1597, 1504, 1449, 1261, 1217, 749, 692.

HRMS (ESI) calcd for $C_{17}H_{20}ON^+$ $[M+H]^+$ m/z: 254.1539, found: 254.1544.



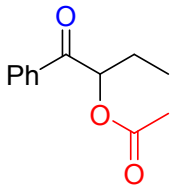
3av

1-Phenyl-2-(phenyl(propyl)amino)butan-1-one (**3av**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **2v** (81 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 62 mg product **3av** as a yellow oil in 55% yield.

3av: R_f = 0.43 (petroleum ether/ethyl acetate = 5:1)

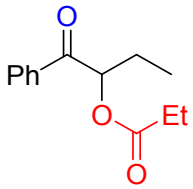
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87−7.81 (m, 2H), 7.52−7.46 (m, 1H), 7.37−7.31 (m, 2H), 7.31−7.26 (m, 2H), 6.85−6.74 (m, 3H), 5.01 (t, J = 7.0 Hz,1H), 3.09 (ddd, J = 15.4, 10.2, 5.4 Hz, 1H), 2.93 (ddd, J = 15.0, 10.0, 6.0 Hz, 1H), 2.22−2.07 (m, 1H), 1.84−1.71 (m, 1H), 1.47−1.30 (m, 2H), 1.01 (t, J = 7.4 Hz, 3H), 0.77 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 198.7, 147.8, 136.5, 133.0, 129.4, 128.5, 128.2, 117.3, 113.6, 64.2, 47.4, 21.4, 21.0, 11.233, 11.226.
IR (KBr) v (cm^{-1}) 2964, 1686, 1596, 1501, 1219, 748, 693.
HRMS (ESI) calcd for $C_{19}H_{24}ON^+$ $[M+H]^+$ m/z: 282.1852, found: 282.1857.



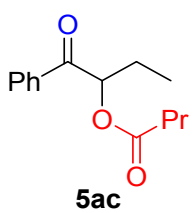
5aa

1-Oxo-1-phenylbutan-2-yl acetate (**5aa**)[9] was synthesized according to General Procedure B from **1a** (52 mg, 0.4 mmol) and using **4a** (196 mg, 2 mmol) instead of NaOH and RCO_2H, eluted by petroleum ether/ethyl acetate = 50/1, to give 75 mg product **5aa** as a yellow oil in 91% yield.
5aa: R_f = 0.58 (petroleum ether/ethyl acetate = 5:1)
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99−7.85 (m, 2H), 7.62−7.52 (m, 1H), 7.46 (t, J = 7.6 Hz, 2H), 5.81 (dd, J = 8.1, 4.3 Hz, 1H), 2.15 (s, 3H), 2.00−1.77 (m, 2H), 1.00 (t, J = 7.4 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 196.5, 170.6, 134.8, 133.4, 128.7, 128.3, 76.3, 24.7, 20.6, 9.8.
IR (KBr) v (cm^{-1}) 2973, 2938, 1739, 1698, 1598, 1449, 1373, 1236, 1104, 904, 699.
HRMS (ESI) calcd for $C_{12}H_{15}O_3^+$ $[M+H]^+$ m/z: 207.1016, found: 207.1010.



5ab

1-Oxo-1-phenylbutan-2-yl propionate (**5ab**) was synthesized according to General Procedure B from **1a** (52 mg, 0.4 mmol) and **4b** (148 mg, 2 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 66 mg product **5ab** as a yellow oil in 75% yield.
5ab: R_f = 0.53 (petroleum ether/ethyl acetate = 20:1)
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, J = 7.6 Hz, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.46 (t, J = 7.7 Hz, 2H), 5.82 (dd, J = 8.0, 4.4 Hz, 1H), 2.45 (qd, J = 7.6, 1.2 Hz, 2H), 2.01−1.79 (m, 2H), 1.16 (t, J = 7.6 Hz, 3H), 1.01 (t, J = 7.4 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 196.7, 174.1, 134.9, 133.4, 128.7, 128.3, 76.1, 27.3, 24.7, 9.8, 9.0.
IR (KBr) v (cm^{-1}) 2977, 2940, 1739, 1698, 1598, 1462, 1449, 1384, 1222, 1186, 1082, 912, 697.
HRMS (ESI) calcd for $C_{13}H_{17}O_3^+$ $[M+H]^+$ m/z: 221.1172, found: 221.1178.



5ac

1-Oxo-1-phenylbutan-2-yl butyrate (**5ac**) was synthesized according to General Procedure B from **1a** (52 mg, 0.4 mmol) and using **4c** (220 mg, 2 mmol) instead of NaOH and RCO_2H, eluted by petroleum ether/ethyl acetate = 100/1, to give 76 mg product **5ac** as a yellow oil in 81% yield.

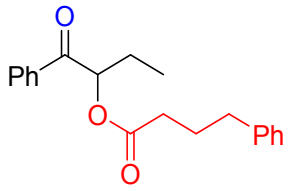
5ac: R_f = 0.58 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, J = 7.2 Hz, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.46 (t, J = 7.8 Hz, 2H), 5.82 (dd, J = 8.2, 4.2 Hz, 1H), 2.41 (dt, J = 1.6, 7.8 Hz, 2H), 2.03−1.78 (m, 2H), 1.73−1.63 (m, 2H), 1.01 (t, J = 7.6 Hz, 3H), 0.96 (t, J = 7.5 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 196.7, 173.3, 134.9, 133.4, 128.7, 128.3, 76.1, 35.8, 24.7, 18.4, 13.6, 9.9.

IR (KBr) v (cm^{-1}) 2968, 2937, 1373, 1698, 1598, 1449, 1384, 1260, 1180, 698.

HRMS (ESI) calcd for $C_{14}H_{19}O_3^+$ $[M+H]^+$ m/z: 235.1329, found: 235.1337.



5ad

1-Oxo-1-phenylbutan-2-yl 4-phenylbutanoate (**5ad**) was synthesized according to General Procedure B from **1a** (328 mg, 0.4 mmol) and **4d** (56 mg, 2 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 96 mg product **5ad** as a yellow oil in 77% yield.

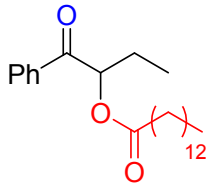
5ad: R_f = 0.54 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ(m, 2H), 7.50−7.43 (m, 1H), 7.39−7.32 (m, 2H), 7.20−7.13 (m, 2H), 7.11−7.05 (m, 3H), 5.72 (dd, J = 8.1, 4.3 Hz, 1H), 2.57 (t, J = 7.8 Hz, 2H), 2.34 (m, 2H), 1.93−1.66 (m, 4H), 0.91 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 196.6, 173.1, 141.4, 134.9, 133.4, 128.7, 128.5, 128.3, 125.9, 76.2, 35.0, 33.2, 26.4, 24.7, 9.9.

IR (KBr) v (cm^{-1}) 2971, 2936, 1737, 1698, 1598, 1496, 1449, 1384, 1222, 1143, 747, 699.

HRMS (ESI) calcd for $C_{20}H_{21}O_3^+$ $[M+H]^+$ m/z: 311.1642 found: 311.1646.



5ae

1-Oxo-1-phenylbutan-2-yl tetradecanoate (**5ae**) was synthesized according to General Procedure B from **1a** (52 mg, 0.4 mmol) and **4e** (457 mg, 2 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 98 mg product **5ae** as a yellow oil in 65% yield.

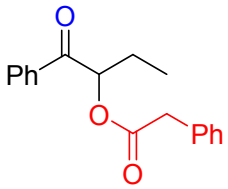
5ae: R_f = 0.71 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, J = 7.6 Hz, 2H), 7.60 (t, J = 7.4 Hz, 1H), 7.49 (t, J = 7.6 Hz, 2H), 5.84 (dd, J = 8.1, 4.3 Hz, 1H), 2.45 (t, J = 7.5 Hz, 2H), 2.03−1.82 (m, 2H), 1.67 (tt, J = 7.2, 7.2 Hz, 2H), 1.28 (s, 20H), 1.05 (t, J = 7.4 Hz, 3H), 0.91 (t, J = 6.6 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 196.8, 173.5, 135.0, 133.4, 128.7, 128.4, 76.08, 34.0, 31.9, 29.7, 29.6, 29.6, 29.4, 29.3, 29.2, 29.1, 24.9, 24.7, 22.7, 14.1, 9.9.

IR (KBr) v (cm^{-1}) 2924, 2853, 1740, 1701, 1598, 1464, 1449, 1222, 1180, 1107, 697.

HRMS (ESI) calcd for $C_{24}H_{39}O_3^+$ $[M+H]^+$ m/z: 375.2894, found: 375.2895.



5af

1-Oxo-1-phenylbutan-2-yl 2-phenylacetate (**5af**) was synthesized according to General Procedure B from **1a** (52 mg, 0.4 mmol) and **4f** (272 mg, 2 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 73 mg product **5af** as a yellow oil in 72% yield.

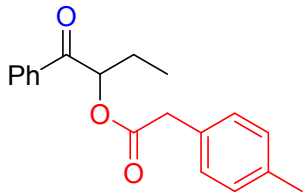
5af: R_f = 0.56 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, J = 7.6 Hz, 2H), 7.57 (t, J = 7.2 Hz, 1H), 7.44 (t, J = 7.4 Hz, 2H), 7.36−7.19 (m, 5H), 5.86−5.78 (m, 1H), 3.76 (s, 2H), 2.03−1.79 (m, 2H), 0.98 (t, J = 7.2 Hz, 3H)

^{13}C NMR (101 MHz, $CDCl_3$) δ 196.5, 171.2, 134.9, 133.6, 133.4, 129.3, 128.7, 128.5, 128.3, 127.1, 76.7, 40. 9, 24.7, 9.8.

IR (KBr) v (cm^{-1}) 2977, 2938, 1747, 1698, 1598, 1449, 1383, 1223, 1179, 1045, 697.

HRMS (ESI) calcd for $C_{13}H_{16}ClO_3^+$ $[M+H]^+$ m/z: 255.0782, found: 255.0791.



5ag

1-Oxo-1-phenylbutan-2-yl 2-p-tolylacetate (**5ag**) was synthesized according to General Procedure B from **1a** (52 mg, 0.4 mmol) and **4g** (300 mg, 2 mmol), eluted by petroleum ether/ethyl acetate = 50/1, to give 85 mg product **5ag** as a yellow oil in 72% yield.

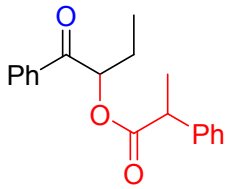
5ag: R_f = 0.53 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94−7.89 (m, 2H), 7.591H), 7.47−7.54 (m, 2H), 7.19 (d, J = 8.0 Hz, 2H), 7.13 (d, J = 8.0 Hz, 2H), 5.82 (dd, J = 8.1, 4.5 Hz, 1H), 3.72 (s, 2H), 2.33 (s, 3H), 2.02−1.81 (m, 2H), 0.99 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 196.5, 171.4, 136.6, 134.9, 133.4, 130.5, 129.2, 128.7, 128.3, 76.6, 40.4, 24.7, 21.0, 9.8.

IR (KBr) v (cm^{-1}) 2973, 2936, 1737, 1698, 1597, 1516, 1448, 1247, 1223, 1155, 977, 697.

HRMS (ESI) calcd for $C_{19}H_{21}O_3^+$ $[M+H]^+$ m/z: 297.1485 found: 297.1491

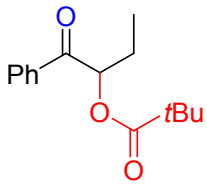


5ah

1-Oxo-1-phenylbutan-2-yl 2-phenylpropanoate (**5ah**) was synthesized according to General Procedure B from **1a** (52 mg, 0.4 mmol) and **4h** (300 mg, 2 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 96 mg product **5ah** as a yellow oil in 81% yield.

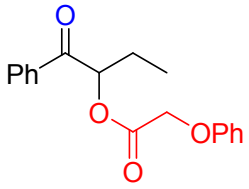
5ah: R_f = 0.63 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, J = 7.7 Hz, 1H), 7.84 (d, J = 7.7 Hz, 1H), 7.59−7.49 (m, 1H), 7.44 (t, J = 7.7 Hz, 1H), 7.38 (t, J = 7.7 Hz, 1H), 7.34−7.28 (m, 4H), 7.28−7.21 (m, 1H), 5.79−5.71 (m, 1H), 3.88 (q, J = 7.2 Hz, 0.5H), 3.83 (q, J = 7.2 Hz, 0.5H), 2.00−1.73 (m, 2H), 1.56 (d, J = 7.2 Hz, 1.5H), 1.53 (d, J = 7.2 Hz, 1.5H), 0.96 (t, J = 7.4 Hz, 1.5H), 0.86 (t, J = 7.4 Hz, 1.5H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 196.64 (196.36), 174.20 (173.99), 140.28 (139.76), 134.87 (134.84), 133.42 (133.25), 128.68 (128.58), 128.54 (128.47), 128.32 (128.28), 127.63 (127.56), 127.12 (127.09), 76.75 (76.52), 45.34 (45.08), 24.68 (24.60), 18.41 (18.31), 9.79 (9.66).
IR (KBr) v (cm^{-1}) 2976, 2936, 1734, 1698, 1598, 1495, 1449, 1382, 1221, 1165, 912, 733, 698.
HRMS (ESI) calcd for $C_{19}H_{21}O_3^+$ $[M+H]^+$ m/z: 297.1485, found: 297.1493.



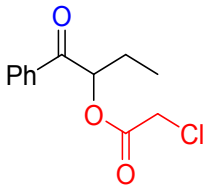
5ai

1-Oxo-1-phenylbutan-2-yl pivalate (**5ai**) was synthesized according to General Procedure B from **1a** (52 mg, 0.4 mmol) and **4i** (204 mg, 2 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 61 mg product **5ai** as a white solid in 61% yield.
5ai: R_f = 0.64 (petroleum ether/ethyl acetate = 20:1), mp 60-63 °C
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95−7.88 (m, 2H), 7.56 (t, J = 7.4 Hz, 1H), 7.45 (t, J = 7.7 Hz, 2H), 5.75 (dd, J = 8.3, 4.3 Hz, 1H), 2.02−1.79 (m, 2H), 1.25 (s, 9H), 1.02 (t, J = 7.4 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 196.8, 178.1, 135.0, 133.3, 128.6, 128.3, 76.0, 38.6, 27.0, 24.6, 9.8.
IR (KBr) v (cm^{-1}) 2973, 2935, 1731, 1699, 1480, 1449, 1283, 1222, 1157, 912, 698.
HRMS (ESI) calcd for $C_{15}H_{21}O_3^+$ $[M+H]^+$ m/z: 249.1485, found: 249.1492.



5aj

1-Oxo-1-phenylbutan-2-yl 2-phenoxyacetate (**5aj**) was synthesized according to General Procedure B from **1a** (52 mg, 0.4 mmol) and **4j** (304 mg, 2 mmol), eluted by petroleum ether/ethyl acetate = 20/1, to give 65 mg product **5aj** as a yellow solid in 54% yield.
5aj: R_f = 0.44 (petroleum ether/ethyl acetate = 20:1), mp 65-67 °C
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, J = 7.6 Hz, 2H), 7.60 (t, J = 7.4 Hz, 1H), 7.48 (t, J = 7.6 Hz, 2H), 7.29 (t, J = 8.0 Hz, 2H), 6.99 (t, J = 7.4 Hz, 1H), 6.93 (d, J = 8.4 Hz, 2H), 5.95 (dd, J = 8.0, 4.2 Hz, 1H), 4.78 (s, 2H), 2.08−1.78 (m, 2H), 0.99 (t, J = 7.4 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 195.8, 168.8, 157.8, 134.6, 133.6, 129.5, 128.8, 128.4, 121.7, 114.6, 77.1, 65.0, 24.7, 9.7.
IR (KBr) v (cm^{-1}) 2973, 2935, 1761, 1696, 1599, 1496, 1222, 1192, 1089, 755, 692.
HRMS (ESI) calcd for $C_{18}H_{19}O_4^+$ $[M+H]^+$ m/z: 299.1278, found: 299.1282.



5ak

1-Oxo-1-phenylbutan-2-yl 2-chloroacetate (**5ak**) was synthesized according to General Procedure B from **1a** (52 mg, 0.4 mmol) and **4k** (190 mg, 2 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 59 mg product **5ak** as a yellow oil in 61% yield.

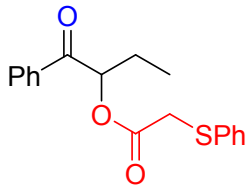
5ak: R_f = 0.57 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96−7.88 (m, 2H), 7.63−7.54 (m, 1H), 7.48 (t, J = 7.7 Hz, 2H), 5.91 (dd, J = 8.1, 4.2 Hz, 1H), 4.19 (s, 2H), 2.07−1.83 (m, 2H), 1.02 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 195.4, 167.0, 134.5, 133.7, 128.8, 128.3, 77.9, 40.6, 24.8, 9.7.

IR (KBr) ν (cm^{-1}) 2973, 1762, 1697, 1597, 1449, 1223, 1181, 976, 697.

HRMS (ESI) calcd for $C_{12}H_{14}ClO_3^+$ $[M+H]^+$ *m/z*: 241.0626, found: 241.0622.



5al

1-Oxo-1-phenylbutan-2-yl 2-(phenylthio)acetate (**5al**) was synthesized according to General Procedure B from **1a** (52 mg, 0.4 mmol) and **4l** (336 mg, 2 mmol), eluted by petroleum ether/ethyl acetate = 50/1, to give 91 mg product **5al** as a yellow oil in 72% yield.

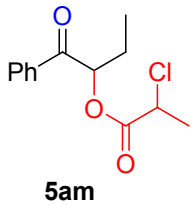
5al: R_f = 0.51 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, J = 7.6 Hz, 2H), 7.58 (t, J = 7.4 Hz, 1H), 7.50−7.38 (m, 4H), 7.29 (t, J = 7.5 Hz, 2H), 7.21 (t, J = 7.3 Hz, 1H), 5.83 (dd, J = 8.0, 4.3 Hz, 1H), 3.80 (d, J = 15.2 Hz, 1H), 3.74 (d, J = 15.2 Hz, 1H), 0.95 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 195.9, 169.4, 134.9, 134.7, 133.6, 129.8, 129.0, 128.7, 128.4, 126.9, 77.3, 36.3, 24.7, 9.7.

IR (KBr) ν (cm^{-1}) 2972, 2928, 1737, 1698, 1598, 1582, 1449, 1384, 1266, 1222, 1130, 738, 67.

HRMS (ESI) calcd for $C_{18}H_{19}O_3S^+$ $[M+H]^+$ *m/z*: 315.1049, found: 315.1054.



5am

1-Oxo-1-phenylbutan-2-yl 2-chloropropanoate (**5am**) was synthesized according to General Procedure B from **1a** (52 mg, 0.4 mmol) and **4m** (217 mg, 2 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 73 mg product **5am** as a yellow oil in 72% yield.

5am: R_f = 0.56 (petroleum ether/ethyl acetate = 20:1)

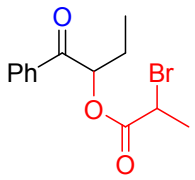
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96−7.91 (m, 2H), 7.65−7.57 (m, 1H), 7.49 (t, J = 7.7 Hz, 2H), 5.89 (dd, J = 8.0, 4.2 Hz, 0.5H), 5.87 (dd, J = 8.0, 4.2 Hz, 0.5H), 4.56 (q, J = 7.0 Hz, 0.5H), 4.52 (q, J =

7.0 Hz, 0.5H), 2.09−1.86 (m, 2H), 1.78 (d, *J* = 7.0 Hz, 1.5H), 1.75 (d, *J* = 7.0 Hz, 1.5H), 1.05 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 195.61 (195.42), 169.84 (169.56), 134.5, 133.66 (133.63), 128.79 (128.76), 128.32 (128.29), 77.61 (77.58), 52.46 (52.03), 24.72 (24.69), 21.56 (21.38), 9.70 (9.68).

IR (KBr) *v* (cm^{-1}) 2977, 2938, 1747, 1698, 1598, 1449, 1383, 1223, 1179, 1045, 697.

HRMS (ESI) calcd for $C_{13}H_{16}ClO_3^+$ $[M+H]^+$ *m/z*: 255.0782, found: 255.0791.



5an

1-Oxo-1-phenylbutan-2-yl 2-bromopropanoate (**5an**) was synthesized according to General Procedure B from **1a** (52 mg, 0.4 mmol) and **4n** (306 mg, 2 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 83 mg product **5an** as a yellow oil in 69% yield.

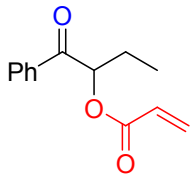
5an: R_f = 0.59 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97−7.90 (m, 2H), 7.63−7.56 (m, 1H), 7.52−7.45 (m, 2H), 5.85 (2dd, *J* = 8.1, 4.2 Hz, 1H), 4.49 (2q, *J* = 6.9 Hz, 1H), 2.08−1.83 (m, 5H), 1.05 (2t, *J* = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 195.77 (195.49), 170.02 (169.63), 134.60, 133.68 (133.63), 128.81 (128.78), 128.38 (128.35), 77.61 (77.55), 39.86 (39.38), 24.79 (24.69), 21.75 (21.52), 9.77.

IR (KBr) *v* (cm^{-1}) 2975, 2933, 1740, 1698, 1448, 1223, 1159, 1074, 698.

HRMS (ESI) calcd for $C_{13}H_{16}BrO_3^+$ $[M+H]^+$ *m/z*: 299.0277, found: 299.0285.



5ao

1-Oxo-1-phenylbutan-2-yl acrylate (**5ao**) was synthesized according to General Procedure B from **1a** (52 mg, 0.4 mmol) and **4o** (144 mg, 2 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 26 mg product **5ao** as a yellow oil in 30% yield.

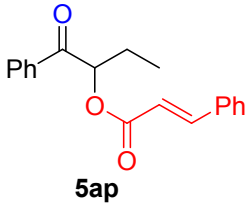
5ao: R_f = 0.50 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03−7.86 (m, 2H), 7.62−7.55 (m, 1H), 7.51−7.44 (m, 2H), 6.49 (dd, *J* = 17.3, 1.3 Hz, 1H), 6.24 (dd, *J* = 17.3, 10.4 Hz, 1H), 5.94−5.86 (m, 2H), 2.11−1.85 (m, 2H), 1.04 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 196.4, 165.7, 134.9, 133.5, 131.7, 128.8, 128.4, 127.7, 76.4, 24.8, 9.9.

IR (KBr) *v* (cm^{-1}) 2970, 2932, 1727, 1698, 11598, 1449, 1407, 1276, 1222, 1195, 972, 768, 699.

HRMS (ESI) calcd for $C_{13}H_{15}O_3^+$ $[M+H]^+$ *m/z*: 219.1016, found: 219.1011.



5ap

1-Oxo-1-phenylbutan-2-yl cinnamate (**5ap**) was synthesized according to General Procedure B from **1a** (52 mg, 0.4 mmol) and **4p** (296 mg, 2 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 46 mg product **5ap** as a yellow oil in 39% yield.

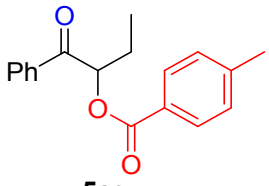
5ap: R_f = 0.53 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89−7.85 (m, 2H), 7.64 (d, J = 16.0 Hz, 1H), 7.50−7.34 (m, 5H), 7.20−7.24 (m, 3H), 6.45 (d, J = 16.0 Hz, 1H), 5.86 (dd, J = 8.0, 4.4 Hz, 1H), 1.98−1.77 (m, 2H), 0.96 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 196.6, 166.4, 145.8, 134.9, 134. 2, 133.5, 130.4, 128.8, 128.7, 128.4, 128.2, 117.2, 76.3, 24.8, 9.9.

IR (KBr) v (cm^{-1}) 2973, 2935, 1697, 1637, 1598, 1449, 1312, 1203, 11701, 982, 768, 697.

HRMS (ESI) calcd for $C_{19}H_{19}O_3^+$ $[M+H]^+$ *m/z*: 295.1329, found: 295.1332.



5aq

1-Oxo-1-phenylbutan-2-yl 4-methylbenzoate (**5aq**) was synthesized according to General Procedure B from **1a** (52 mg, 0.4 mmol) and **4q** (272 mg, 2 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 51 mg product **5aq** as a yellow oil in 45% yield.

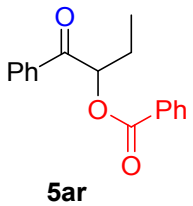
5aq: R_f = 0.60 (petroleum ether/ethyl acetate = 20:1), mp 56 -58 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07−7.91 (m, 4H), 7.62−7.54 (m, 1H), 7.52−7.44 (m, 2H), 7.24 (d, J = 8.1 Hz, 2H), 6.04 (dd, J = 7.9, 4.5 Hz, 1H), 2.41 (s, 3H), 2.16−1.95 (m, 2H), 1.11 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 196.5, 166.2, 144.0, 135.0, 133.4, 129.9, 129.1, 128.7, 128.4, 126.8, 76.5, 24.9, 21.7, 10.0.

IR (KBr) v (cm^{-1}) 2973, 2936, 1719, 1698, 1612, 1448, 1310, 1277, 1109, 752, 698.

HRMS (ESI) calcd for $C_{18}H_{19}O_3^+$ $[M+H]^+$ *m/z*: 283.1329, found: 283.1335.



5ar

1-Oxo-1-phenylbutan-2-yl benzoate (**5ar**)[10] was synthesized according to General Procedure B from **1a** (52 mg, 0.4 mmol) and using **4r** (288 mg, 2 mmol) instead of NaOH and RCO_2H, eluted by petroleum ether/ethyl acetate = 100/1, to give 51 mg product **5ar** as a yellow oil in 48% yield.

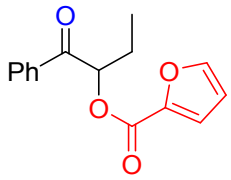
5ar: R_f = 0.55 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (d, J = 7.4 Hz, 2H), 8.01 (d, J = 7.5 Hz, 2H), 7.62 (dd, J = 12.4, 7.1 Hz, 2H), 7.52−7.41 (m, 4H), 6.06 (dd, J = 7.9, 4.5 Hz, 1H), 2.19−1.91 (m, 2H), 1.12 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 196.4, 166.2, 134.9, 133.5, 133.2, 129.8, 129.6, 128.8, 128.4, 128.4, 76.7, 24.9, 9.9.

IR (KBr) v (cm^{-1}) 2973, 2936, 1721, 1697, 1450, 1277, 1250, 1115, 712, 699.

HRMS (ESI) calcd for $C_{17}H_{17}O_3^+$ $[M+H]^+$ *m/z*: 269.1172, found: 269.1176.



5as

1-Oxo-1-phenylbutan-2-yl-furan-2-carboxylate (**5as**) was synthesized according to General Procedure B from **1a** (52 mg, 0.4 mmol) and **4s** (224 mg, 2 mmol), eluted by petroleum ether/ethyl acetate = 20/1, to give 21 mg product **5as** as a yellow oil in 20% yield.

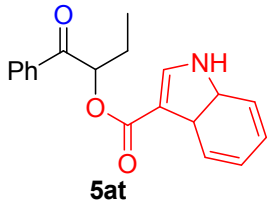
5as: R_f = 0.39 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02−7.94 (m, 2H), 7.63−7.54 (m, 2H), 7.48 (t, J = 7.6 Hz, 2H), 7.27 (d, J = 3.5 Hz, 1H), 6.52 (dd, J = 3.5, 1.7 Hz, 1H), 6.03 (dd, J = 8.0, 4.4 Hz, 1H), 2.15−1.92 (m, 2H), 1.08 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 196.0, 158.1, 146.7, 144.1, 134.8, 133.5, 128.8, 128.4, 118.7, 111.9, 76.6, 24.9, 9.9.

IR (KBr) ν (cm^{-1}) 2975, 2938, 1726, 1697, 1580, 1473, 1397, 1303, 1228, 1181, 1120, 1015, 763, 698.

HRMS (ESI) calcd for $C_{15}H_{15}O_4^+$ $[M+H]^+$ *m/z*: 259.0965, found: 259.0969.



5at

1-Oxo-1-phenylbutan-2-yl-3a,7a-dihydro-1*H*-indole-3-carboxylate (**5at**) was synthesized according to General Procedure B from **1a** (52 mg, 0.4 mmol) and **4t** (326 mg, 2 mmol), eluted by petroleum ether/ethyl acetate = 20/1, to give 28 mg product **5at** as a white solid in 23% yield.

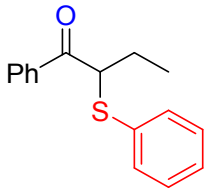
5at: R_f = 0.36 (petroleum ether/ethyl acetate = 20:1), mp 157-159 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.20 (s, 1H), 8.05−8.00 (m, 2H), 7.69 (d, J = 8.0 Hz, 1H), 7.60 (t, J = 7.4 Hz, 1H), 7.50 (t, J = 7.6 Hz, 2H), 7.38−7.33 (m, 2H), 7.28−7.32 (s, 1H), 7.14 (t, J = 7.4 Hz, 1H), 6.09 (dd, J = 8.0, 4.5 Hz, 1H), 2.23−1.97 (m, 2H), 1.14 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 196.1, 161.5, 137.1, 134.7, 133.6, 128.8, 128.5, 127.4, 126.4, 125.6, 122.6, 120.8, 112.0, 109.7, 76.9, 25.0, 9.94.

IR (KBr) ν (cm^{-1}) 3332, 1682, 1527, 1449, 1429, 1244, 1202, 745, 697.

HRMS (ESI) calcd for $C_{19}H_{20}NO_3^+$ $[M+H]^+$ *m/z*: 308.1281, found: 308.1283.



7aa

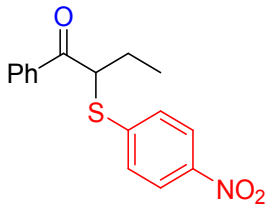
1-Phenyl-2-(phenylthio)butan-1-one (**7aa**)[11] was synthesized according to General Procedure A

from **1a** (52 mg, 0.4 mmol) and **6a** (66 mg, 0.6 mmol) without adding NaH_2PO_4 in step 2, eluted by petroleum ether/ethyl acetate = 100/1, to give 74 mg product **7aa** as a yellow oil in 72% yield.

7aa: R_f = 0.69 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97−7.86 (m, 2H), 7.57−7.51 (m, 1H), 7.45−7.39 (m, 2H), 7.35−7.31 (m, 2H), 7.29−7.21 (m, 3H), 4.37 (t, J = 7.1 Hz, 1H), 2.03 (ddq, J = 14.5, 7.2, 7.2 Hz, 1H), 1.86 (ddq, J = 14.4, 7.2, 7.2 Hz, 1H), 1.05 (t, J = 7.2 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 196.0, 136.3, 134.4, 133.0, 132.0, 128.9, 128.5, 128.5, 53.3, 24.2, 11.9.



7ab

2-(4-Nitrophenylthio)-1-phenylbutan-1-one (**7ab**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **6b** (93 mg, 0.6 mmol) without adding NaH_2PO_4 in step 2, eluted by petroleum ether/ethyl acetate = 100/1, then use preparative TLC to give 79 mg product **7ab** as a yellow oil in 66% yield.

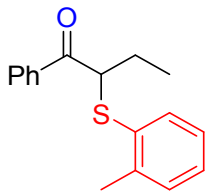
7ab: R_f = 0.69 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (d, J = 8.3 Hz, 2H), 7.96 (d, J = 7.9 Hz, 2H), 7.60 (t, J = 7.3 Hz, 1H), 7.48 (t, J = 7.6 Hz, 2H), 7.42 (d, J = 8.5 Hz, 2H), 4.66 (t, J = 7.0 Hz, 1H), 2.16 (ddq, J = 14.4, 7.2, 7.2 Hz, 1H), 1.96 (ddq, J = 14.4, 7.2, 7.2 Hz, 1H), 1.08 (t, J = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 195.7, 146.5, 143.2, 135.5, 133.6, 131.0, 128.8, 128.5, 123.9, 52.8, 24.7, 11.8.

IR (KBr) ν (cm^{-1}) 2924, 1744, 1450, 1457, 1366, 1235, 1146, 831, 746.

HRMS (ESI) calcd for $C_{16}H_{16}NO_3S^+$ $[M+H]^+$ m/z: 302.0845, found: 302.0846.



7ac

1-Phenyl-2-(o-tolylthio)butan-1-one (**7ac**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **6c** (75 mg, 0.6 mmol) without adding NaH_2PO_4 in step 2, eluted by petroleum ether/ethyl acetate = 50/1, to give 72 mg product **7ac** as a yellow oil in 67% yield.

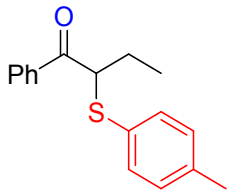
7ac: R_f = 0.55 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89−7.83 (m, 2H), 7.56−7.49 (m, 1H), 7.43−7.34 (m, 3H), 7.20−7.06 (m, 3H), 4.42 (t, J = 7.1 Hz, 1H), 2.30 (s, 3H), 2.19−2.06 (m, 1H), 2.02−1.88 (m, 1H), 1.06 (t, J = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 196.6, 141.3, 136.3, 134.2, 133.0, 132.5, 130.4, 128.4, 128.2, 126.4, 53.4, 24.8, 20.8, 12.1.

IR (KBr) ν (cm^{-1}) 3060, 2968, 2874, 1680, 1596, 1448, 1261, 1221, 984, 751, 689.

HRMS (ESI) calcd for $C_{17}H_{19}SO^+$ $[M+H]^+$ *m/z*: 271.1151, found: 271.1155.



7ad

1-Phenyl-2-(p-tolylthio)butan-1-one (**7ad**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **6d** (75 mg, 0.6 mmol) without adding NaH_2PO_4 in step 2, eluted by petroleum ether/ethyl acetate = 50/1, to give 69 mg product **7ad** as a yellow oil in 64% yield.

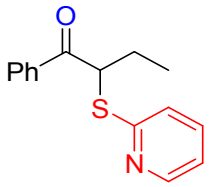
7ad: R_f = 0.55 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98−7.91 (m, 2H), 7.56 (t, J = 7.4 Hz, 1H), 7.45 (t, J = 7.6 Hz, 2H), 7.22 (d, J = 8.0 Hz, 2H), 7.08 (d, J = 7.9 Hz, 2H), 4.31 (t, J = 7.2 Hz, 1H), 2.33 (s, 3H), 2.00 (ddq, J = 14.6, 7.3, 7.3 Hz, 1H), 1.85 (ddq, J = 14.3, 7.3, 7.3 Hz, 1H), 1.06 (t, J = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 195.8, 138.9, 136.4, 135.1, 132.9, 129.7, 128.5, 127.9, 53.3, 24.0, 21.2, 11.9.

IR (KBr) ν (cm^{-1}) 3968, 2874, 1680, 1597, 1492, 1448, 1263, 1221, 808, 689.

HRMS (ESI) calcd for $C_{17}H_{19}SO^+$ $[M+H]^+$ *m/z*: 271.1151, found: 271.1150.



7ae

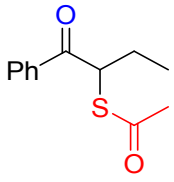
1-Phenyl-2-(pyridin-2-ylthio)butan-1-one (**7ae**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **6e** (67 mg, 0.6 mmol) without adding NaH_2PO_4 in step 2, eluted by petroleum ether/ethyl acetate = 20/1, to give 86 mg product **7ae** as a yellow oil in 84% yield.

7ae: R_f = 0.26 (petroleum ether/ethyl acetate = 5:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.39 (dd, J = 4.8, 0.6 Hz, 1H), 8.08 (d, J = 7.4 Hz, 2H), 7.53 (t, J = 7.4 Hz, 1H), 7.50−7.39 (m, 3H), 7.16 (d, J = 8.1 Hz, 1H), 7.03−6.95 (m, 1H), 5.71 (t, J = 7.0 Hz, 1H), 2.18 (ddq, J = 14.4, 7.3, 7.3 Hz, 1H), 2.02−1.87 (m, 1H), 1.04 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 198.1, 157.0, 149.2, 136.3, 136.1, 133.0, 128.6, 128.5, 122.4, 119.8, 48.7, 25.3, 11.8.

HRMS (ESI) calcd for $C_{15}H_{16}SNO^+$ $[M+H]^+$ *m/z*: 258.0947, found: 258.0951.



7af

S-1-Oxo-1-phenylbutan-2-yl ethanethioate (**7af**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **6f** (69 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate =

100/1, to give 60 mg product **7af** as a yellow oil in 67% yield.

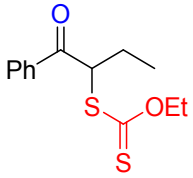
7af: R_f = 0.65 (petroleum ether/ethyl acetate = 5:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05−7.91 (m, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.46 (t, J = 7.7 Hz, 2H), 5.17 (t, J = 7.1 Hz, 1H), 2.35 (s, 3H), 2.10 (ddq, J = 14.3, 7.3, 7.3 Hz, 1H), 1.90−1.75 (m, 1H), 0.98 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 197.2, 194.3, 135.5, 133.5, 128.7, 128.5, 48.5, 30.2, 25.2, 11.5.

IR (KBr) ν (cm^{-1}) 2968, 1685, 1596, 1579, 1448, 1355, 1263, 1106, 807, 691.

HRMS (ESI) calcd for $C_{12}H_{15}O_2S^+$ $[M+H]^+$ m/z: 223.0787, found: 223.0779.



7ag

O-Ethyl *S*-1-oxo-1-phenylbutan-2-yl carbonodithioate (**7ag**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **6g** (96 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 90 mg product **7ag** as a yellow oil in 84% yield.

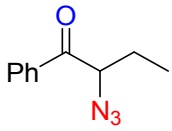
7ag: R_f = 0.72 (petroleum ether/ethyl acetate = 5:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, J = 7.5 Hz, 2H), 7.60 (t, J = 7.4 Hz, 1H), 7.49 (t, J = 7.7 Hz, 2H), 5.42 (t, J = 6.9 Hz, 1H), 4.62 (q, J = 7.1 Hz, 2H), 2.15 (ddq, J = 14.3, 7.2, 7.2 Hz, 1H), 1.93 (ddq, J = 14.5, 7.3, 7.3 Hz, 1H), 1.35 (t, J = 7.1 Hz, 3H), 1.04 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 213.2, 196.6, 135.7, 133.6, 128.7, 128.6, 70.6, 56.1, 25.0, 13.7, 11.7.

IR (KBr) ν (cm^{-1}) 2970, 1686, 1596, 1578, 1448, 1218, 1112, 1050, 984, 809, 692.

HRMS (ESI) calcd for $C_{13}H_{17}O_2S_2^+$ $[M+H]^+$ m/z: 269.0664, found: 269.0665.



7ai

2-Azido-1-phenylbutan-1-one (**7ai**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **6i** (39 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 50/1, to give 50 mg product **7ai** as a yellow oil in 66% yield.

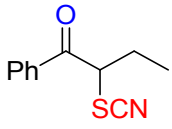
7ai: R_f = 0.35 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, J = 7.7 Hz, 2H), 7.61 (t, J = 7.4 Hz, 1H), 7.49 (t, J = 7.7 Hz, 2H), 4.54 (dd, J = 8.3, 5.1 Hz, 1H), 2.05−1.79 (m, 2H), 1.07 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 196.6, 134.8, 133.8, 128.9, 128.5, 64.5, 24.8, 10.6.

IR (KBr) ν (cm^{-1}) 2973, 2104, 1691, 1597, 1579, 1449, 1214, 989, 694.

HRMS (ESI) calcd for $C_{10}H_{12}ON_3^+$ $[M+H]^+$ m/z: 190.0975, found: 190.0971.



7aj

1-Phenyl-2-thiocyanatobutan-1-one (**7aj**)[12] was synthesized according to General Procedure A

from **1a** (52 mg, 0.4 mmol) and **6j** (58 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 60 mg product **7aj** as a yellow oil in 73% yield.

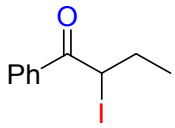
7aj: R_f = 0.33 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, J = 8.0 Hz, 2H), 7.66 (t, J = 7.3 Hz, 1H), 7.53 (t, J = 7.5 Hz, 2H), 5.01 (t, J = 5.8 Hz, 1H), 2.44−2.14 (m, 2H), 1.05 (t, J = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 194.6, 134.5, 133.8, 129.1, 128.6, 111.4, 55.8, 26.0, 10.0.

IR (KBr) ν (cm^{-1}) 2972, 2155, 1682, 1596, 1579, 1449, 1268, 1202, 986, 764, 692.

HRMS (ESI) calcd for $C_{11}H_{12}NOS^+$ $[M+H]^+$ m/z: 206.0634, found: 206.0634.



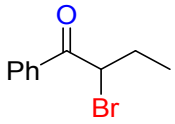
7ak

2-Iodo-1-phenylbutan-1-one (**7ak**)[13] was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **6k** (100 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 94 mg product **7ak** as a yellow oil in 86% yield.

7ak: R_f = 0.42 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03−7.97 (m, 2H), 7.62−7.54 (m, 1H), 7.47 (t, J = 7.6 Hz, 2H), 5.27 (t, J = 7.2 Hz, 1H), 2.17 (p, J = 7.3 Hz, 2H), 1.05 (t, J = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 194.5, 134.1, 133.5, 128.7, 128.6, 28.2, 28.1, 14.2.



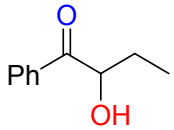
7al

2-Bromo-1-phenylbutan-1-one (**7al**)[14] was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **6l** (71 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 72 mg product **7al** as a yellow oil in 79% yield.

7al: R_f = 0.48 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06−7.98 (m, 2H), 7.63−7.57 (m, 1H), 7.49 (t, J = 7.6 Hz, 2H), 5.08 (dd, J = 7.7, 6.5 Hz, 1H), 2.31−2.06 (m, 2H), 1.09 (t, J = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 193.2, 134.0, 133.6, 128.8, 128.7, 49.0, 26.9, 12.1.



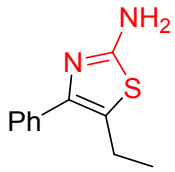
7am

2-Hydroxy-1-phenylbutan-1-one (**7am**)[15] was synthesized according to General Procedure C from **1a** (52 mg, 0.4 mmol), eluted by petroleum ether/ethyl acetate = 25/1, to give 42 mg product **7am** as a yellow oil in 64% yield.

7am: R_f = 0.37 (petroleum ether/ethyl acetate = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, J = 7.5 Hz, 2H), 7.62 (t, J = 7.4 Hz, 1H), 7.50 (t, J = 7.7 Hz, 2H), 5.06 (ddd, J = 6.6, 6.6, 3.9 Hz, 1H), 3.70 (d, J = 6.4 Hz, 1H), 1.96 (dqd, J = 14.8, 7.5, 3.9 Hz, 1H), 1.61 (ddd, J = 14.3, 7.2, 7.2 Hz, 1H), 0.94 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 202.1, 133.9, 133.8, 128.8, 128.5, 73.9, 28.8, 8.8.



9aa

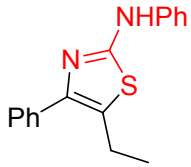
5-Ethyl-4-phenylthiazol-2-amine (**9aa**)[16] was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **8a** (46 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 5/1, to give 52 mg product **9aa** as a yellow oil in 64% yield.

9aa: R_f = 0.35 (petroleum ether/ethyl acetate = 1:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (d, J = 7.3 Hz, 2H), 7.38 (t, J = 7.6 Hz, 2H), 7.29 (t, J = 7.3 Hz, 1H), 5.27 (s, 2H), 2.79 (q, J = 7.5 Hz, 2H), 1.24 (t, J = 7.5 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 164.2, 145.4, 135.4, 128.4, 128.2, 127.2, 125.9, 20.5, 16.7.

HRMS (ESI) calcd for $C_{11}H_{13}N_2S^+$ $[M+H]^+$ *m/z*: 205.0794, found: 205.0804.



9ab

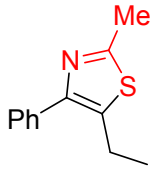
5-Ethyl-*N*,4-diphenylthiazol-2-amine (**9ab**)[17] was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **8b** (91 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 20/1, to give 92 mg product **9ab** as a yellow solid in 82% yield.

9ab: R_f = 0.52 (petroleum ether/ethyl acetate = 5:1), mp : 124-126 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (s, 1H), 7.58 (d, J = 7.7 Hz, 2H), 7.38 (t, J = 7.4 Hz, 2H), 7.33−7.15 (m, 5H), 7.07−6.95 (m, 1H), 2.86 (q, J = 7.5 Hz, 2H), 1.28 (t, J = 7.5 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 161.6, 145.4, 140.6, 135.2, 129.3, 128.6, 128.3, 127.4, 124.8, 122.6, 118.2, 20.5, 16.7.

HRMS (ESI) calcd for $C_{17}H_{17}N_2S^+$ $[M+H]^+$ *m/z*: 281.1107, found: 281.1112.



9ac

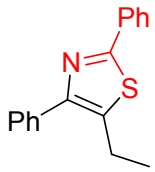
5-Ethyl-2-methyl-4-phenylthiazole (**9ac**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **8c** (45 mg, 0.6 mmol) and added extra 2 ml dichloroethane and stirred at 60 °C in step 3. Eluted by petroleum ether/ethyl acetate = 50/1, to give 52 mg product **9ac** as a yellow oil in 64% yield.

9ac: R_f = 0.58 (petroleum ether/ethyl acetate = 5:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, J = 7.3 Hz, 2H), 7.42 (t, J = 7.6 Hz, 2H), 7.33 (t, J = 7.4 Hz, 1H), 2.92 (q, J = 7.5 Hz, 2H), 2.69 (s, 3H), 1.30 (t, J = 7.5 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 161.9, 149.7, 135.8, 135.3, 128.6, 128.3, 127.4, 20.7, 19.1, 16.8.

HRMS (ESI) calcd for $C_{12}H_{14}NS^+$ $[M+H]^+$ *m/z*: 204.0841, found: 204.0847.



9ad

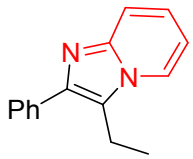
5-Ethyl-2,4-diphenylthiazole (**9ad**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **8d** (82 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 50/1, to give 62 mg product **9ad** as a yellow oil in 58% yield.

9ad: R_f = 0.53 (petroleum ether/ethyl acetate = 5:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, J = 7.6 Hz, 2H), 7.70 (d, J = 7.7 Hz, 2H), 7.50−7.35 (m, 6H), 3.02 (q, J = 7.5 Hz, 2H), 1.38 (t, J = 7.5 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.8, 151.3, 136.7, 135.3, 133.9, 129.6, 128.8, 128.7, 128.4, 127.6, 126.3, 21.0, 16.8.

HRMS (ESI) calcd for $C_{17}H_{16}NS^+$ $[M+H]^+$ *m/z*: 266.0998, found: 266.1003.



9ae

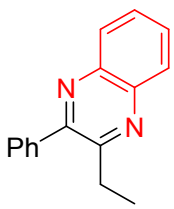
3-Ethyl-2-phenylimidazo[1,2-*a*]pyridine (**9ae**)[18] was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **8e** (56 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 2/1, to give 28 mg product **9ae** as a yellow oil in 31% yield.

9ae: R_f = 0.28 (petroleum ether/ethyl acetate = 1:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, J = 6.8 Hz, 1H), 7.79 (d, J = 7.2 Hz, 2H), 7.64 (d, J = 9.0 Hz, 1H), 7.46 (t, J = 7.6 Hz, 2H), 7.35 (t, J = 7.4 Hz, 1H), 7.22−7.08 (m, 1H), 6.80 (t, J = 6.8 Hz, 1H), 3.10 (q, J = 7.5 Hz, 2H), 1.35 (t, J = 7.5 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 144.3, 141.9, 134.9, 128.5, 128.1, 127.3, 123.4, 122.8, 121.7, 117.7, 111.9, 17.0, 12.2.

HRMS (ESI) calcd for $C_{15}H_{15}N_2^+$ $[M+H]^+$ *m/z*: 223.1230, found: 223.1237.



9af

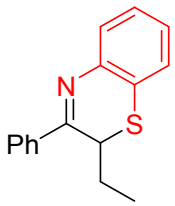
2-Ethyl-3-phenylquinoxaline (**9af**)[19] was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **8f** (65 mg, 0.6 mmol) and added 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) (272 mg, 1.2 mmol) in one-pot after step 3. Eluted by petroleum ether/ethyl acetate = 5/1, to give 70 mg product **9af** as a yellow solid in 75% yield.

9af: R_f = 0.58 (petroleum ether/ethyl acetate = 5:1), mp : 47-49 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17−8.05 (m, 2H), 7.78−7.68 (m, 2H), 7.65−7.59 (m, 2H), 7.56−7.45 (m, 3H), 3.06 (q, J = 7.5 Hz, 2H), 1.31 (t, J = 7.5 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 157.0, 154.9, 141.5, 140.7, 139.1, 129.5, 129.12, 129.1, 128.8, 128.8, 128.5, 128.5, 29.3, 12.9.

HRMS (ESI) calcd for $C_{16}H_{15}N_2^+$ $[M+H]^+$ *m/z*: 235.1230, found: 235.1237.



9ag

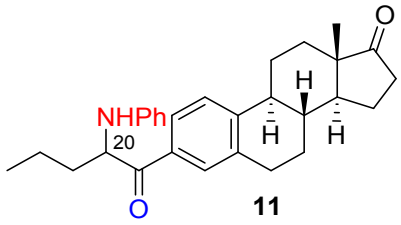
2-Ethyl-3-phenyl-2H-benzo[*b*][1,4]thiazine (**9ag**) was synthesized according to General Procedure A from **1a** (52 mg, 0.4 mmol) and **8g** (75 mg, 0.6 mmol), eluted by petroleum ether/ethyl acetate = 100/1, to give 76 mg product **9ag** as a yellow oil in 75% yield.

9ag: R_f = 0.19 (petroleum ether/ethyl acetate = 50:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07−7.97 (m, 2H), 7.51 (d, J = 7.9 Hz, 1H), 7.48−7.43 (m, 3H), 7.33 (d, J = 7.7 Hz, 1H), 7.23 (t, J = 7.6 Hz, 1H), 7.12 (t, J = 7.5 Hz, 1H), 3.90 (dd, J = 9.8, 4.9 Hz, 1H), 1.74−1.58 (m, 1H), 1.57−1.40 (m, 1H), 0.96 (t, J = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 159.5, 142.8, 137.6, 130.7, 128.7, 127.8, 127.8, 127.4, 126.7, 126.2, 120.2, 37.5, 23.0, 11.0.

HRMS (ESI) calcd for $C_{16}H_{16}NS^+$ $[M+H]^+$ *m/z*: 254.0998, found: 254.1001.



11

(20*R*+*S*)-(8*R*,9*S*,13*S*,14*S*)-13-Methyl-3-(2-(phenylamino)pentanoyl)-6,7,8,9,11,12,13,14,15,16-decahydro-17*H*-cyclopenta[*a*]phenanthren-17-one (**11**) was synthesized according to General Procedure A from **10** (64 mg, 0.2 mmol) and **2a** (28 mg, 0.3 mmol), eluted by petroleum ether/ethyl acetate = 10/1, to give 47 mg product **11** as a yellow oil in 55% yield.

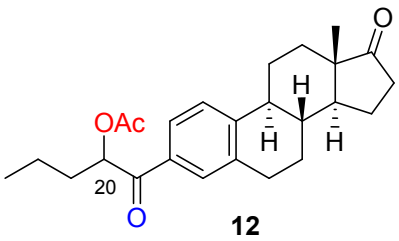
11: R_f = 0.32 (petroleum ether/ethyl acetate = 5:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, J = 8.2 Hz, 1H), 7.73 (s, 1H), 7.41 (d, J = 8.2 Hz, 1H), 7.15 (t, J = 7.8 Hz, 2H), 6.69 (t, J = 8.7 Hz, 3H), 5.05 (t, J = 5.3 Hz, 1H), 4.65 (s, 1H), 3.05−2.94 (m, 2H), 2.57−2.43 (m, 2H), 2.40−2.30 (m, 1H), 2.21−1.90 (m, 5H), 1.70−1.61 (m, 3H), 1.58−1.32 (m, 6H), 0.92 (s, 3H), 0.88 (t, J = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 220.4, 200.5, 147.2, 146.0, 137.3, 132.8, 129.3, 129.0, 125.77, 125.72, 117.7, 113.5, 57.55 (57.52), 50.5, 47.8, 44.7, 37.7, 35.8, 35.4, 31.5, 29.33 (29.28), 26.2, 25.5, 21.5, 18.4, 13.98 (13.76).

IR (KBr) ν (cm^{-1}) 3381, 2931, 2871, 1738, 1679, 1602, 1506, 1454, 748.

HRMS (ESI) calcd for $C_{29}H_{36}NO_2^+$ $[M+H]^+$ *m/z*: 430.2741, found: 430.2741.



12

(20*R*+*S*)-1-((8*R*,9*S*,13*S*,14*S*)-13-Methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta[*a*]phenanthren-3-yl)-1-oxopentan-2-yl acetate (**12**) was synthesized according to General Procedure B from **11** (64 mg, 0.2 mmol) and **4a** (98 mg, 1 mmol), eluted by petroleum ether/ethyl acetate = 10/1, to give 55 mg product **12** as a yellow oil in 69% yield.

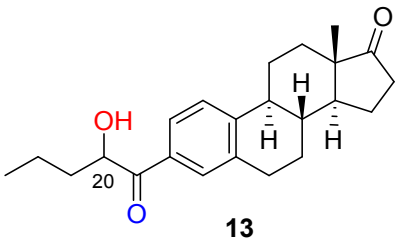
12: R_f = 0.26 (petroleum ether/ethyl acetate = 5:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, J = 8.3 Hz, 1H), 7.67 (s, 1H), 7.38 (d, J = 8.1 Hz, 1H), 5.93−5.75 (m, 1H), 3.05−2.89 (m, 2H), 2.58−2.39 (m, 2H), 2.38−2.28 (m, 1H), 2.20−2.01 (m, 6H), 2.00−1.94 (m, 1H), 1.85−1.76 (m, 2H), 1.68−1.41 (m, 8H), 0.96−0.88 (m, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 220.4, 196.46 (196.33), 170.6, 145.9, 137.1, 132.32 (132.27), 128.98 (128.96), 125.7, 74.96 (74.93), 50.4, 47.8, 44.7, 37.7, 35.7, 33.4, 31.5, 29.27 (29.20), 26.2, 25.4, 21.5, 20.6, 18.8, 13.73 (13.66).

IR (KBr) v (cm^{-1}) 2932, 2872, 1736, 1691, 1604, 1564, 1236, 1009.

HRMS (ESI) calcd for $C_{25}H_{33}O_4^+$ $[M+H]^+$ m/z: 397.2373, found: 397.2373.



13

(20*R*+*S*)(8*R*,9*S*,13*S*,14*S*)-3-(2-Hydroxypentanoyl)-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17*H*-cyclopenta[*a*]phenanthren-17-one (**13**) was synthesized according to General Procedure C from **10** (64 mg, 0.2 mmol), eluted by petroleum ether/ethyl acetate = 7/1, to give 46 mg product **13** as a colorless oil in 65% yield.

13: R_f = 0.23 (petroleum ether/ethyl acetate = 5:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70−7.64 (m, 2H), 7.41 (d, J = 8.6 Hz, 1H), 5.13−4.93 (m, 1H), 3.71 (d, J = 6.4 Hz, 1H), 3.10−2.88 (m, 2H), 2.58−2.42 (m, 2H), 2.41−2.31 (m, 1H), 2.22−1.97 (m, 4H), 1.88−1.77 (m, 1H), 1.69−1.40 (m, 9H), 0.96−0.87 (m, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 220.3, 201.94 (201.88), 146.5, 137.3, 131.28 (131.22), 129.11 (129.07), 125.91 (125.86), 125.8, 72.76 (72.74), 50.5, 47.8, 44.7, 38.1, 37.7, 35.7, 31.5, 29.30 (29.21), 26.17 (26.14), 25.5, 21.5, 18.2, 13.83 (13.75).

IR (KBr) v (cm^{-1}) 3467, 2932, 2871, 1739, 1675, 1604, 1564, 1454, 1258, 1132, 1009.

HRMS (ESI) calcd for $C_{23}H_{131}O_3^+$ $[M+H]^+$ m/z: 355.2268, found: 355.2269.

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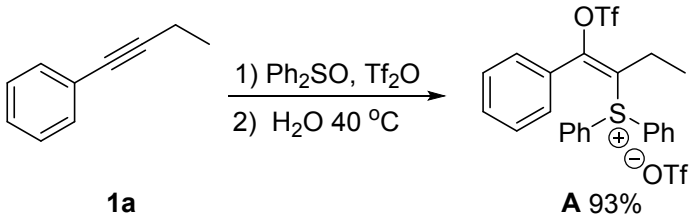
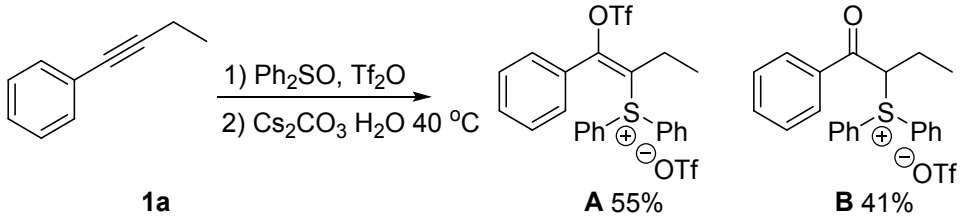
Isolation of crude intermediates

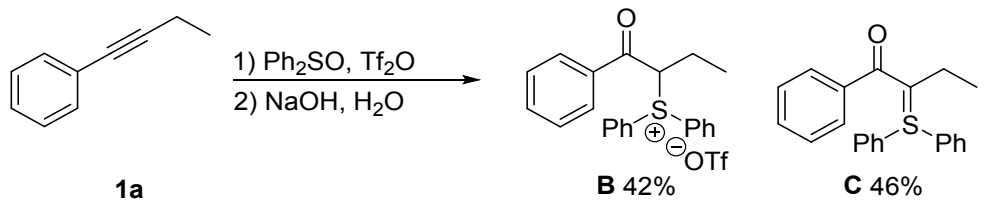


To a flame-dried Schlenk tube, alkyne (**1a**) (52 mg, 0.4 mmol) and diphenyl sulfoxide (97 mg, 0.48 mmol) were added, and then dissolved with dichloromethane (2 mL) under nitrogen atmosphere before cooling down to -78 ºC (liquid N_2/ethyl acetate bath). Tf_2O (81 μL, 0.48 mmol) was added dropwise and stirred for 20 min. The mixture was warmed up to 0 ºC and stirred for another 20 min. Then H_2O (144 mg, 8 mmol) was added to the solution. After stirring at 40 °C for 12 h, the solution was extracted with CH_2Cl_2 (10 mL×3). The combined organic phases were dried with Na_2SO_4 and concentrated under reduced pressure. The residue was further purified by flash column chromatography on silica gel, and eluted by CH_2Cl_2/CH_3OH = 50/1 to afford the intermediate **A** as a yellow oil (229 mg, 93%).



To a flame-dried Schlenk tube, alkyne (**1a**) (52 mg, 0.4 mmol) and diphenyl sulfoxide (97 mg, 0.48 mmol) were added, and then dissolved with dichloromethane (2 mL) under nitrogen atmosphere before cooling down to -78 ºC (liquid N_2/ethyl acetate bath). Tf_2O (81 μL, 0.48 mmol) was added dropwise and stirred for 20 min. The mixture was warmed up to 0 ºC and stirred for another 20 min. Then Cs_2CO_3 (652 mg, 2 mmol) and H_2O (144mg, 8 mmol) were added to the solution. After stirring at 40 °C for 12 h, the solution was extracted with CH_2Cl_2 (10 mL×3). The combined organic phases were dried with Na_2SO_4 and concentrated under reduced pressure. The residue was further purified by flash column chromatography on silica gel and eluted by CH_2Cl_2/CH_3OH = 50/1 to afford

intermediates **A** (136 mg, 55%) and **B** (79 mg, 41%).



1a **B** 42% **C** 46%

To a flame-dried Schlenk tube, alkyne (**1a**) (52 mg, 0.4 mmol) and diphenyl sulfoxide (97 mg, 0.48 mmol) were added, and then dissolved with dichloromethane (2 mL) under nitrogen atmosphere before cooling down to -78 ºC (liquid N_2/ethyl acetate bath). Tf_2O (81 μL, 0.48 mmol) was added dropwise and stirred for 20 min. The mixture was warmed up to 0 ºC and stirred for another 20 min. Then NaOH (80 mg, 2 mmol) and H_2O (144 mg, 8 mmol) were added to the solution. After stirring at 40 ºC for 2.5 h, the solution was extracted with CH_2Cl_2 (10 mL×3). The combined organic phases were dried with Na_2SO_4 and concentrated under reduced pressure. The residue was further purified by flash column chromatography on silica gel and eluted by CH_2Cl_2/CH_3OH = 50/1 to afford intermediates **B** (81 mg, 42%) and **C** (62 mg, 46%).

Intermediate **A**

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 − 7.42 (m, 15H), 2.81 (q, J = 7.4 Hz, 2H), 0.79 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 158.4, 135.2, 133.3, 132.1, 132.0, 131.6, 131.3, 130.9, 130.8, 129.7, 129.6, 128.0, 125.9, 122.5, 120.9 (q, J = 321.0 Hz), 117.8 (q, J = 321.3 Hz), 22.8, 12.7.

^{19}F NMR (377 MHz, $CDCl_3$) δ -73.23, -78.14.

HRMS (ESI) calcd for $C_{23}H_{20}O_3F_3S_2^+$ $(M\text{-}OTf)^+$ *m/z*: 465.0800, found: 465.0803.

Intermediate **B**

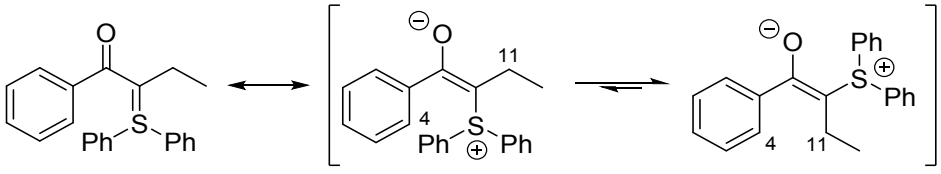
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.38−8.32 (m, 2H), 8.23 (d, J = 7.7 Hz, 2H), 8.18 (d, J = 6.9 Hz, 2H), 7.77−7.70 (m, 3H), 7.62 (t, J = 6.9 Hz, 1H), (m, 5H), 7.38−7.32 (s, 1H), 2.48−2.33 (m, 1H), 2.08−1.93 (m, 1H), 0.93 (t, J = 7.5 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 193.57 (193.53), 135.51 (135.49), 135.17 (135.15), 134.38 (134.36), 133.01 (133.00), 132.31, 131.85, 131.45 (131.43), 130.58, 129.59 (129.58), 129.41, 124.90 (124.89), 121.88 (121.87), 121.13 (q, J = 321.38 Hz, CF3), 69.33, 69.26, 22.79, 8.80.

^{19}F NMR (377 MHz, $CDCl_3$) δ -78.11.

HRMS (ESI) calcd for $C_{23}H_{22}O_4F_3S_2^+$ $[M\text{-}OTf]^+$ *m/z*: 333.1308, found: 333.1317.

Intermediate **C**



SI-2 minor **SI-3 major**

The major isomer was determined by the ^{13}C NMR spectrum. Because of steric interactions between

H attached C-4 and H attached C-11, the electron cloud density of C-11 would enhance. This γ-gauche effect led to the carbon signal of **SI-3** C-11 shifted upfield.[20, 21]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63−7.59 (m, 1H), 7.56−7.46 (m, 6H), 7.45−7.37 (m, 5H), 7.35−7.28 (m, 3H), 2.52 (q, J = 7.2 Hz, 2H, major), 2.28 (q, J = 7.2 Hz, 2H, minor), 0.74 (t, J = 7.2 Hz, 3H, major), 0.64 (t, J = 7.2 Hz, 3H, minor).

^{13}C NMR (101 MHz, $CDCl_3$) δ 187.2, 143.4, 133.2, 131.2, 131.1, 129.9, 129.7, 129.6, 128.7, 127.8, 127.6, 127.2, 71.4, 20.0 (22.3), 13.9 (17.0). (Note: partial ^{13}C signals of the isomer **SI-2** was missing for trace amount.)

HRMS (ESI) calcd for $C_{23}H_{22}O_4F_3S_2^+$ $[M+H]^+$ *m/z*: 333.1308, found: 333.1315.

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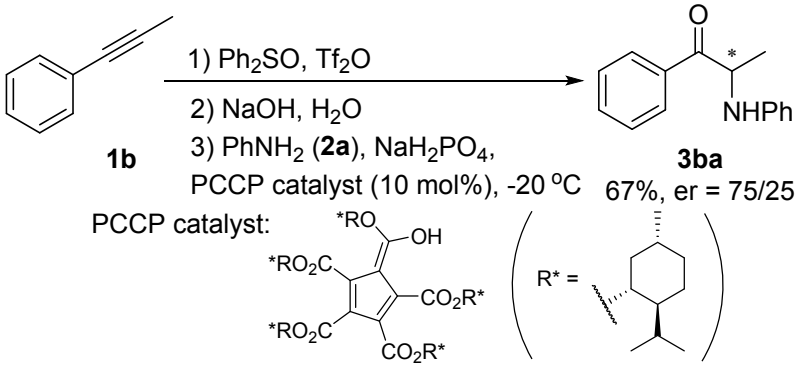
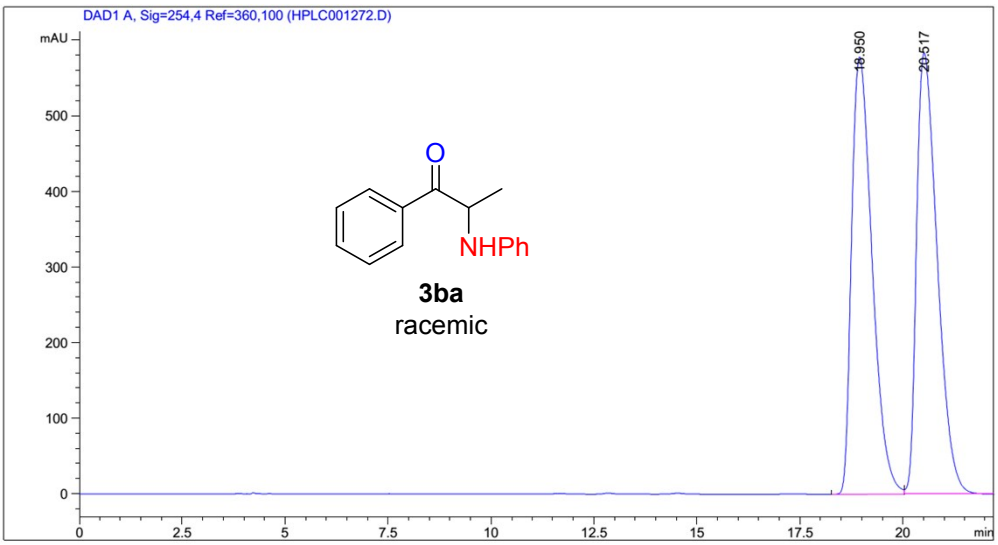
Preliminary results for a catalytic asymmetric version.

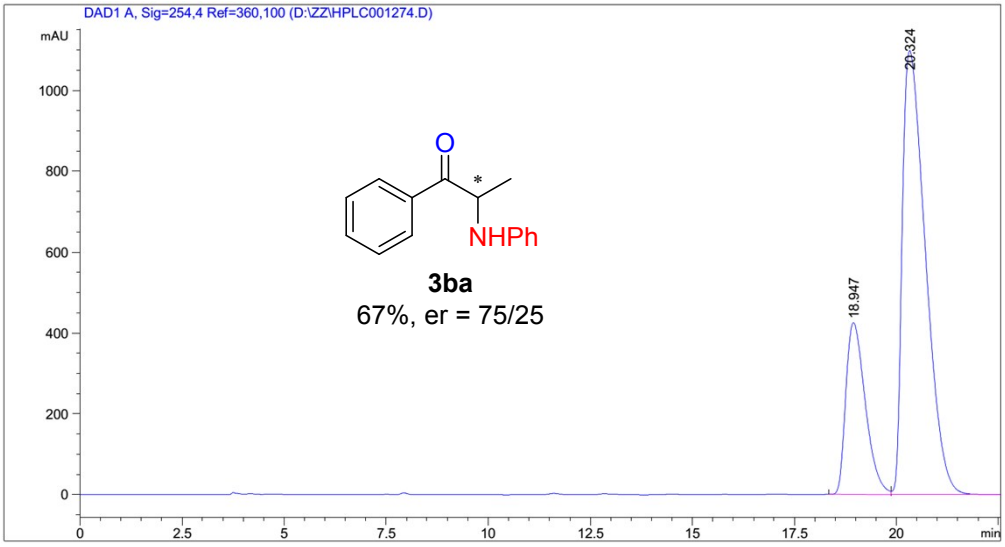


To a flame-dried Schlenk tube, alkyne (**1b**) (23 mg, 0.2 mmol) and diphenyl sulfoxide (49 mg, 0.24 mmol) were added, and then dissolved with dichloromethane (1 mL) under nitrogen atmosphere before cooling down to -78 ºC (liquid N_2/ethyl acetate bath). Tf_2O (41 μL, 0.24 mmol) were added dropwise. After being stirred for 20 min, the mixture was warmed up to 0 ºC and stirred for additional 20 min. Then NaOH (24 mg, 0.6 mmol) and H_2O (72 mg, 4 mmol) were added to the solution and stirred at 40 °C for 12 h. Cooled down to -20 °C, NaH_2PO_4•$2H_2O$ (31 mg, 0.2 mmol), PCCP catalyst (19 mg, 10 mol%), $PhNH_2$ (28 mg, 0.3 mmol) and $CHCl_3$ (1 mL) were added to the solution and continued to stir for 24 h. The solution was quenched with H_2O (10 mL) and extracted with CH_2Cl_2 (10 mL×3). The combined organic phases were dried with Na_2SO_4 and concentrated under reduced pressure. The residue was further purified by flash column chromatography on silica gel and eluted by petroleum ether/ethyl acetate = 100/3 to give 30 mg product **3ba** as a yellow solid in 67% yield (er = 75/25).

The er was determined by HPLC analysis on a Daicel CHIRALPAK AS-H column, *i*PrOH/hexane = 2/98 (V/V), flow rate = 0.8 mL/min): t_R = 19.0 min (25%): t_R = 20.5 min (75%).

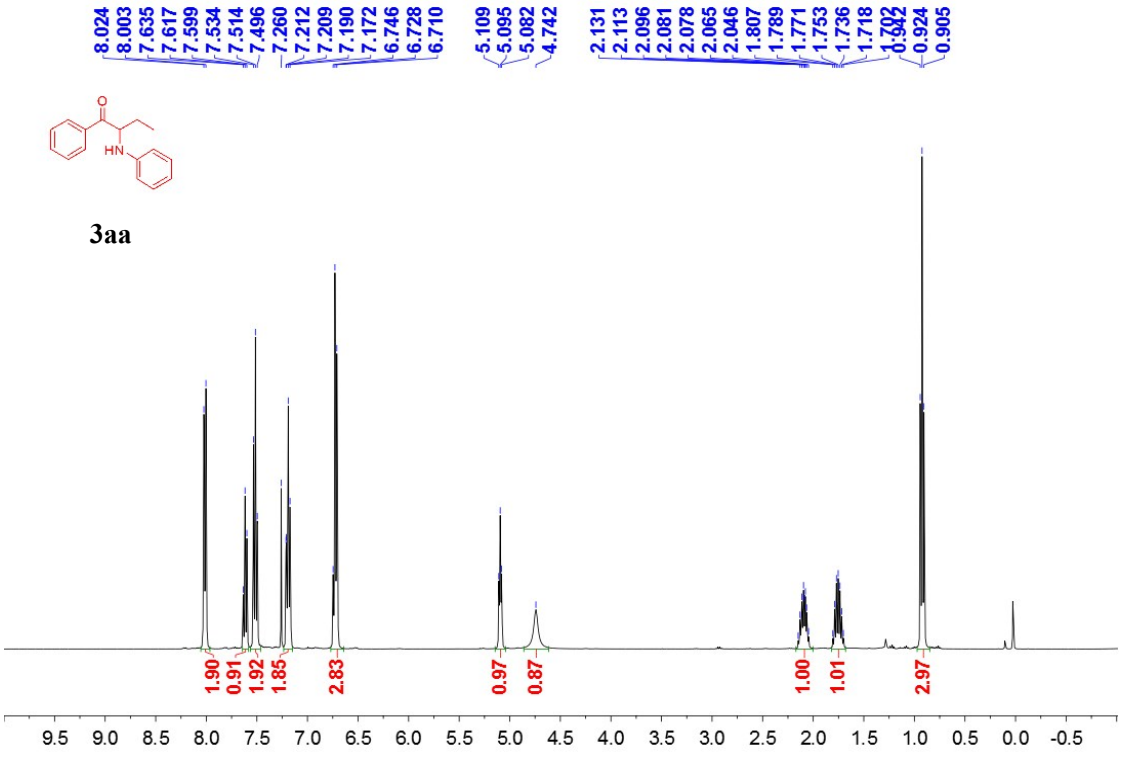


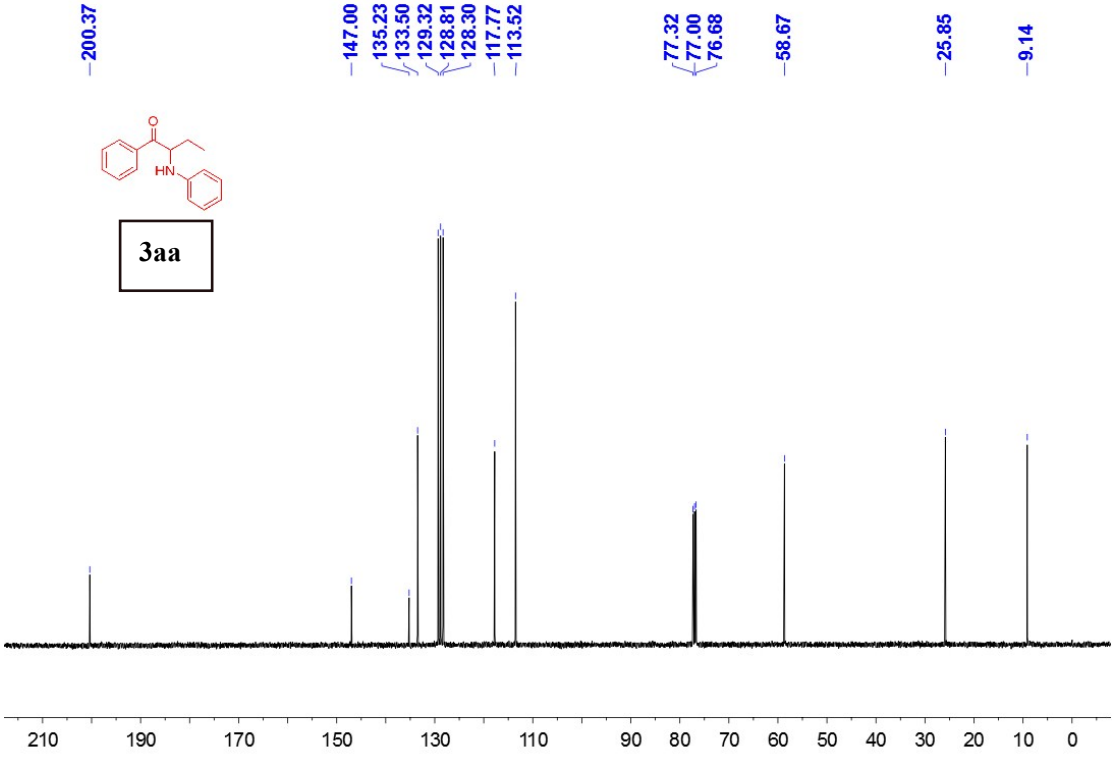
Peak #	Ret. Time [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.950	BV	0.5427	2.00685e4	578.55688	49.6151
2	20.517	VBA	0.5431	2.03799e4	584.01227	50.3849

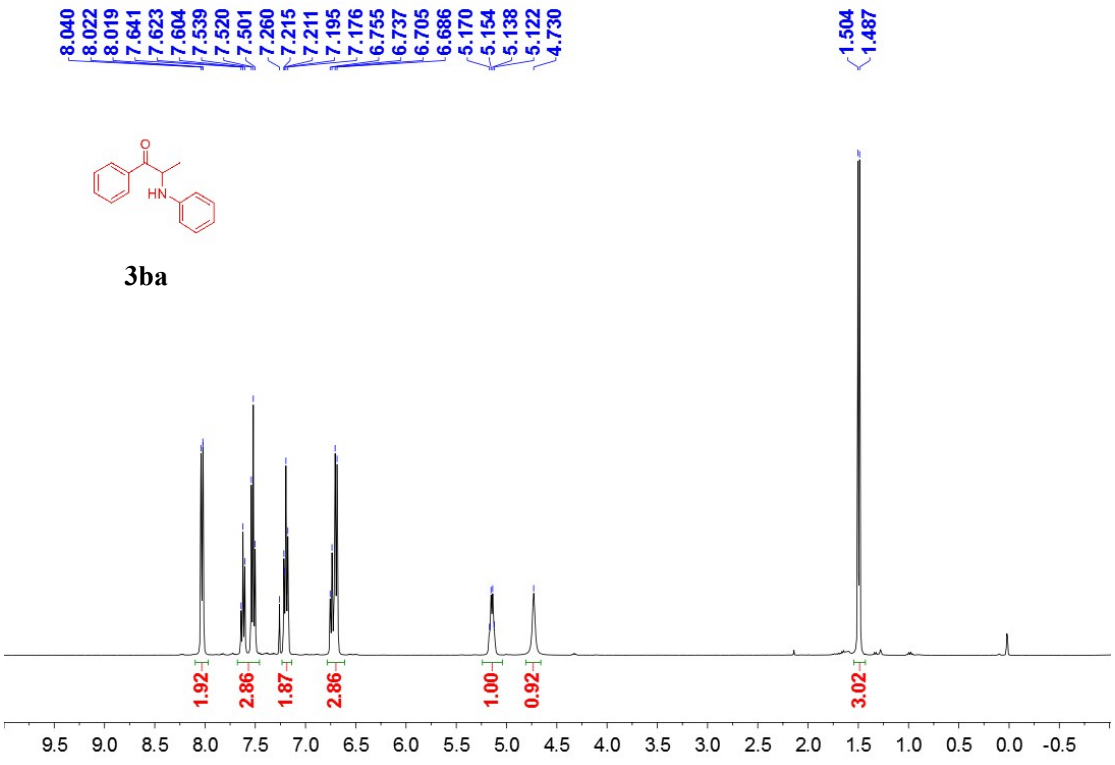


Peak #	Ret. Time [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.947	BV	0.5190	1.41005e4	425.11749	24.5733
2	20.324	VBA	0.6197	4.32809e4	1098.31506	75.4267

IV. Copies of ^{1}H, ^{13}C and ^{19}F NMR Spectra



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6.755
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6.705
6.686
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5.154
5.138
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4.730
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1.487
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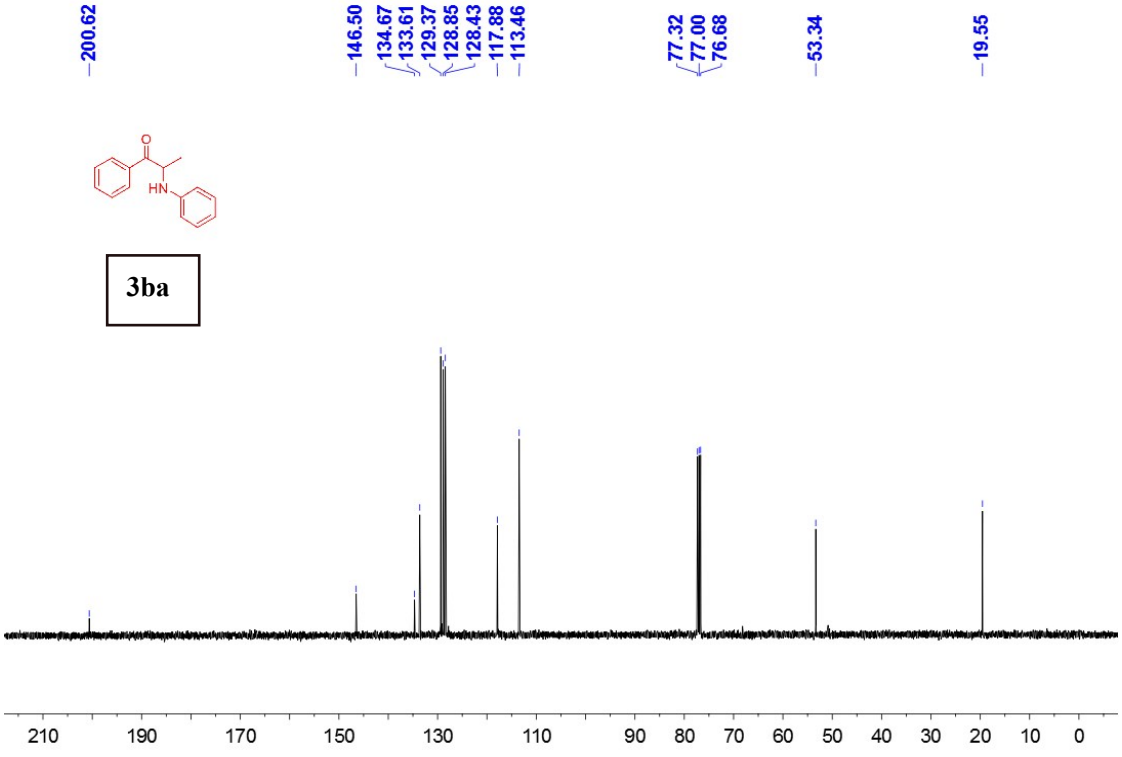
2.86
1.87
2.86
1.00
0.92
3.02
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5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19.55
3ba
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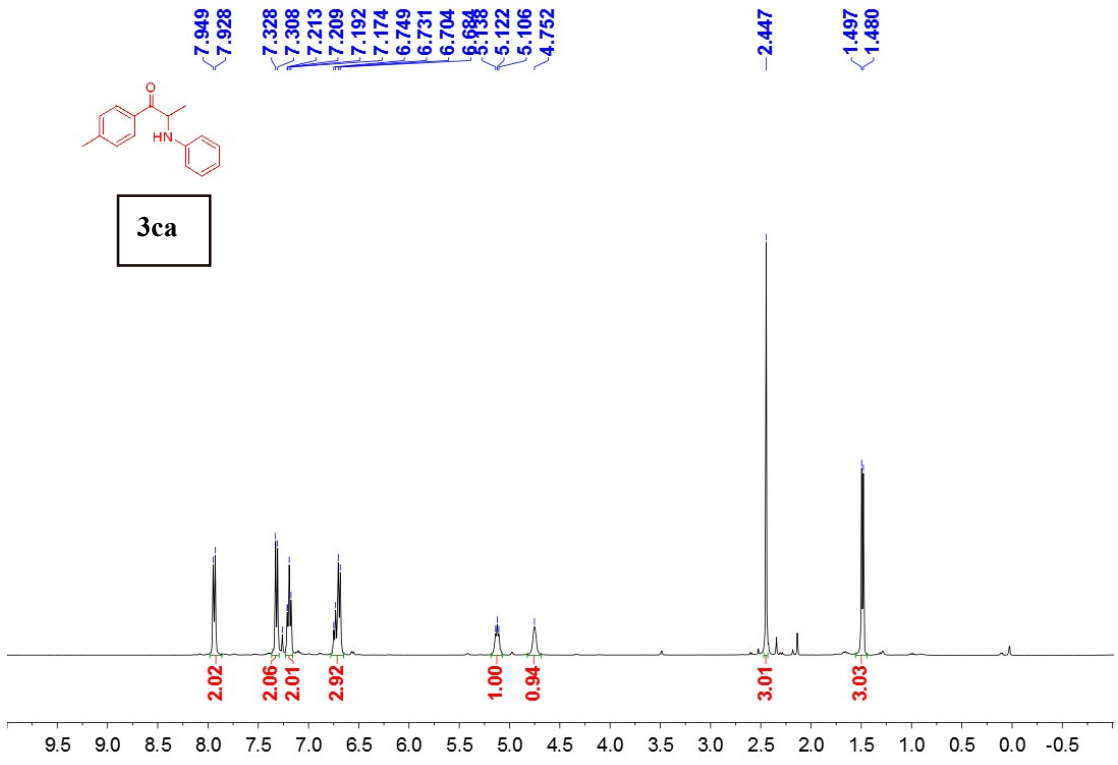
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1.480
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2.06
2.01
2.92
1.00
0.94
3.01
3.03
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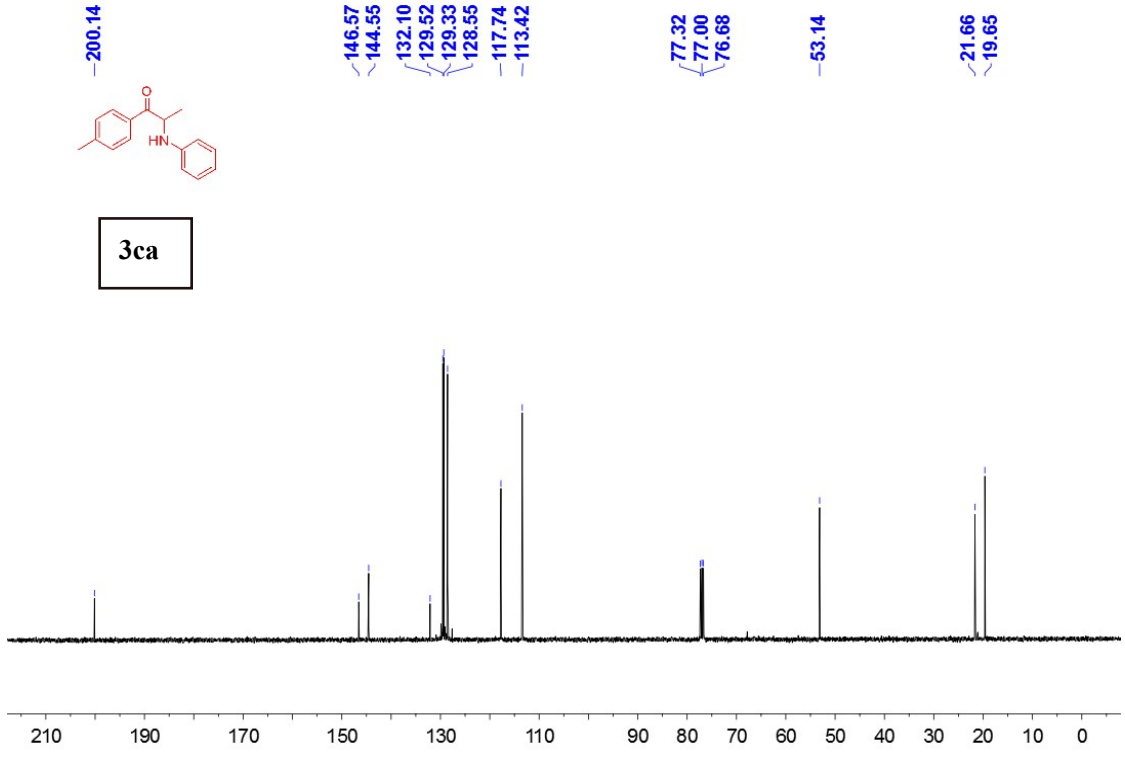
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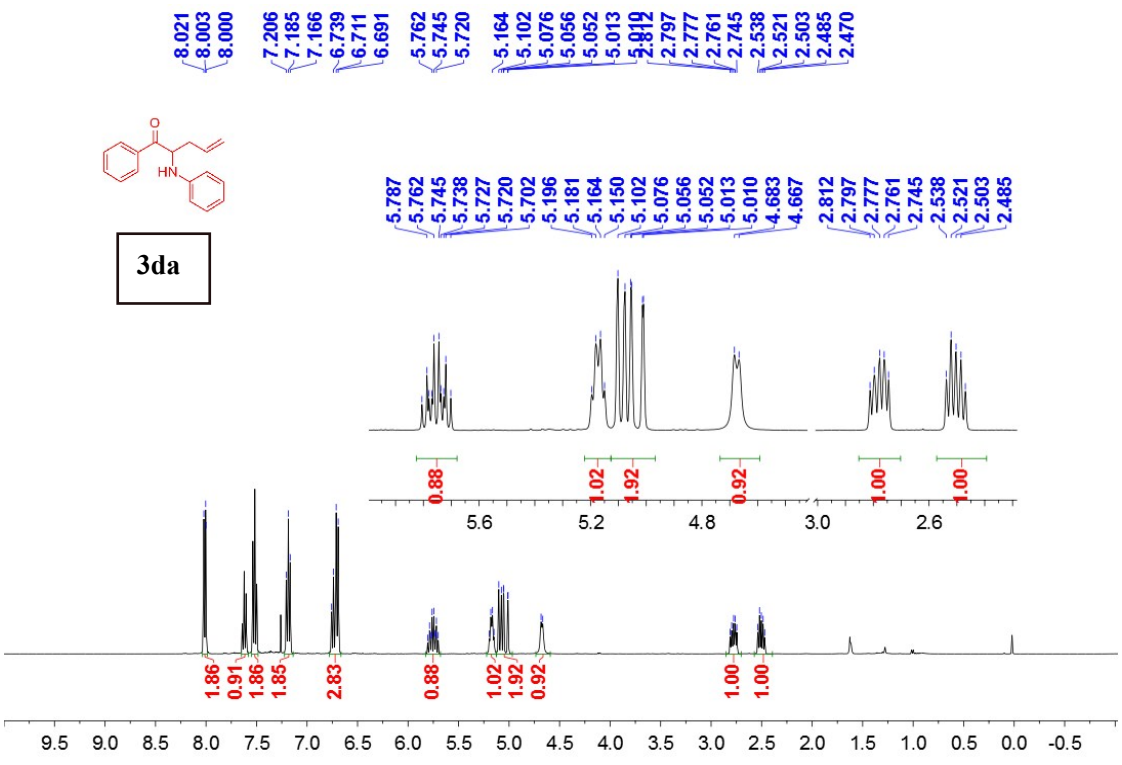
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5.102
5.076
5.056
5.052
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2.777
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2.521
2.503
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0.92
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1.00
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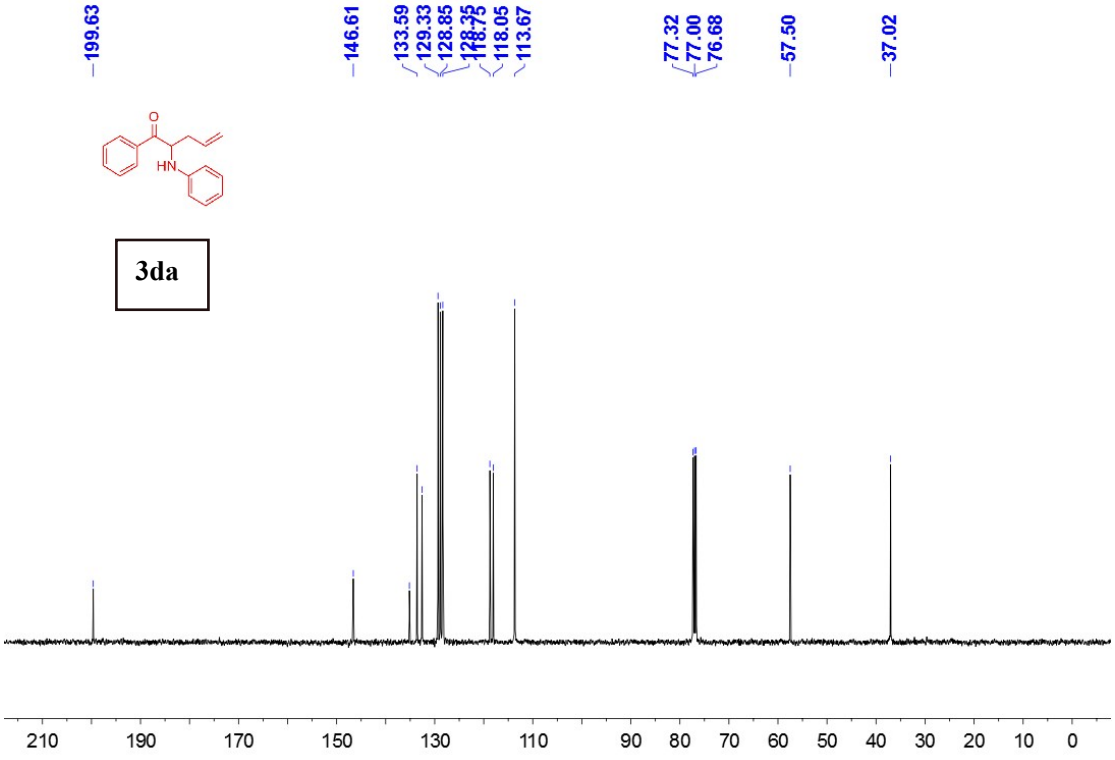
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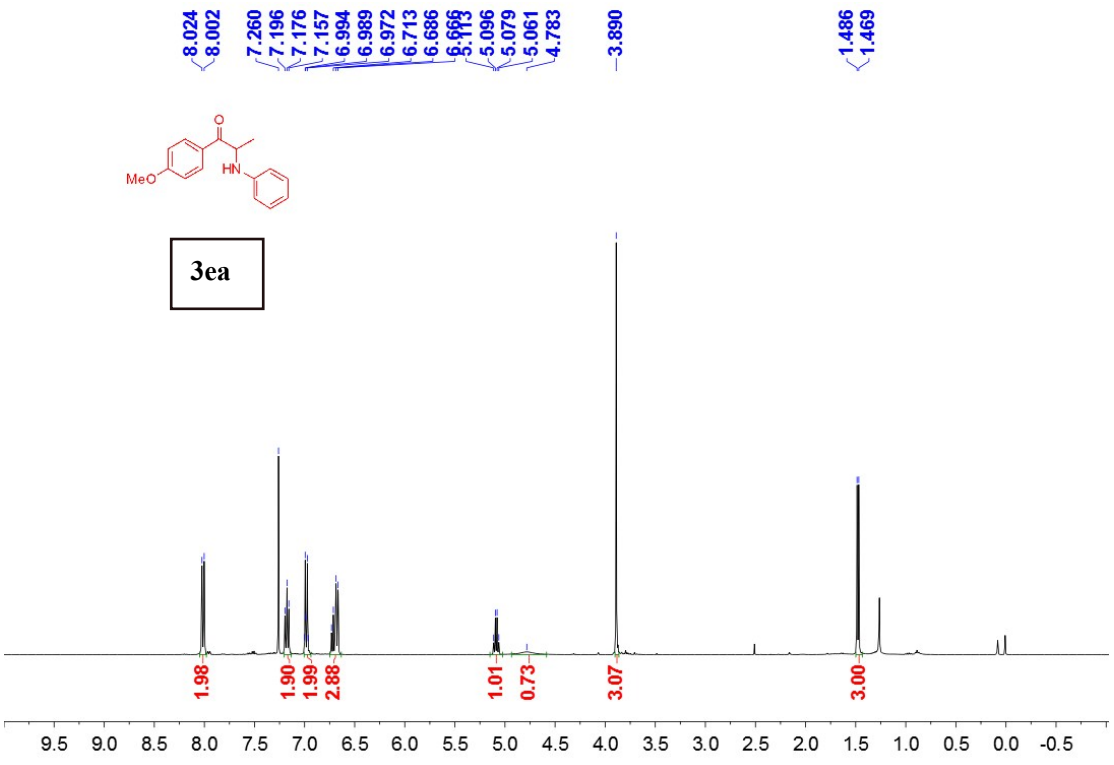
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1.469
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1.99
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3.00
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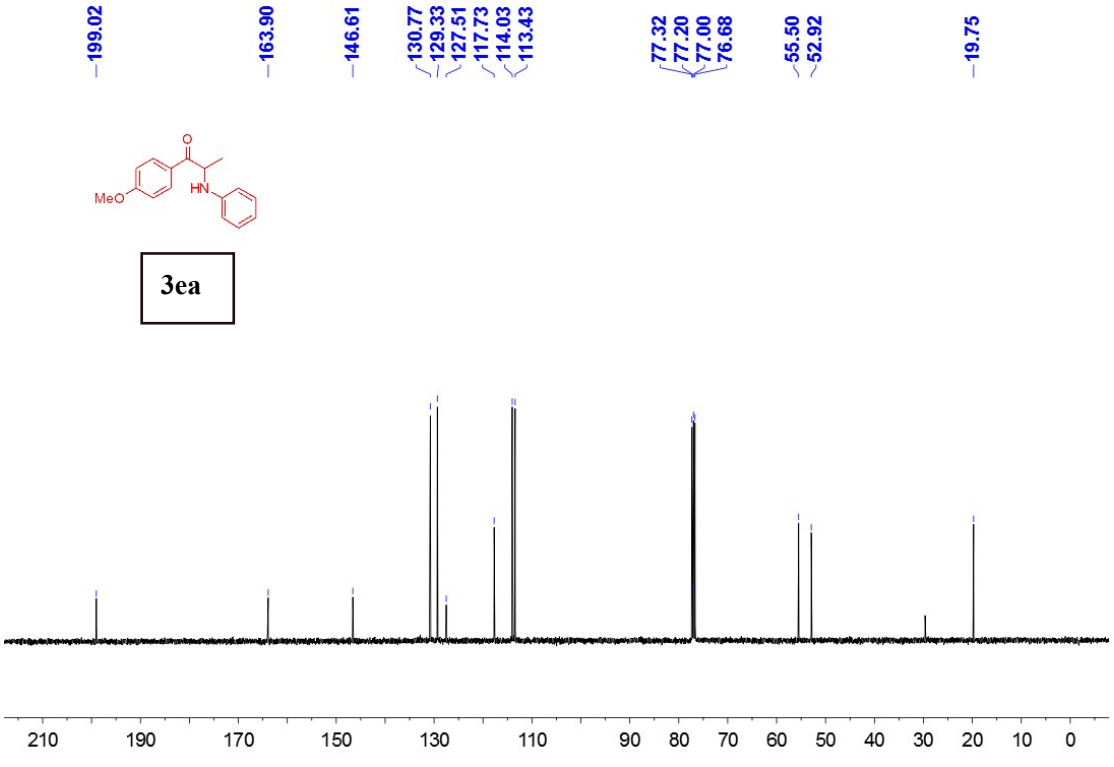
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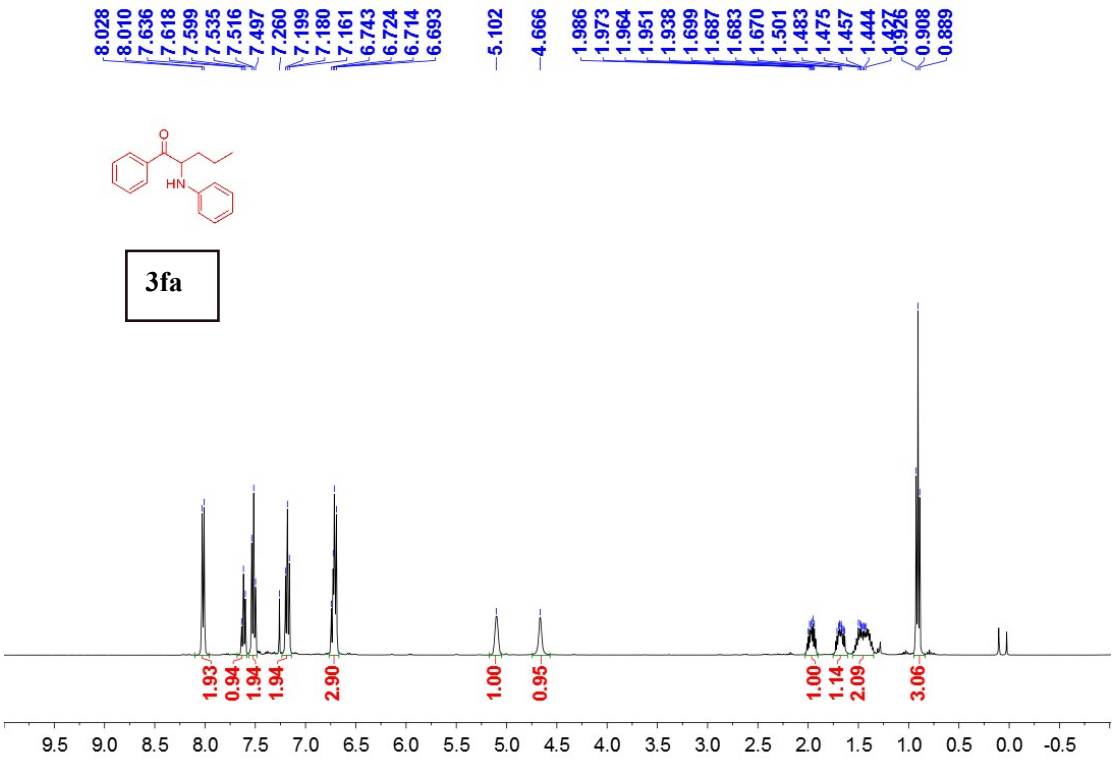
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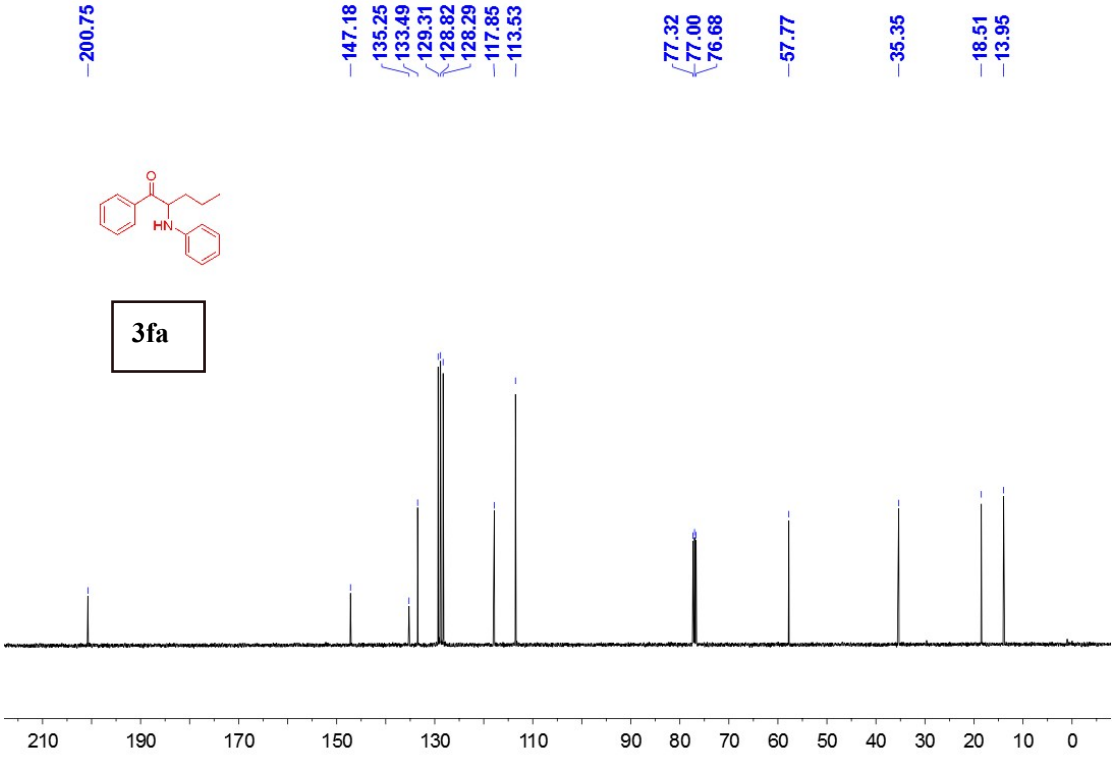
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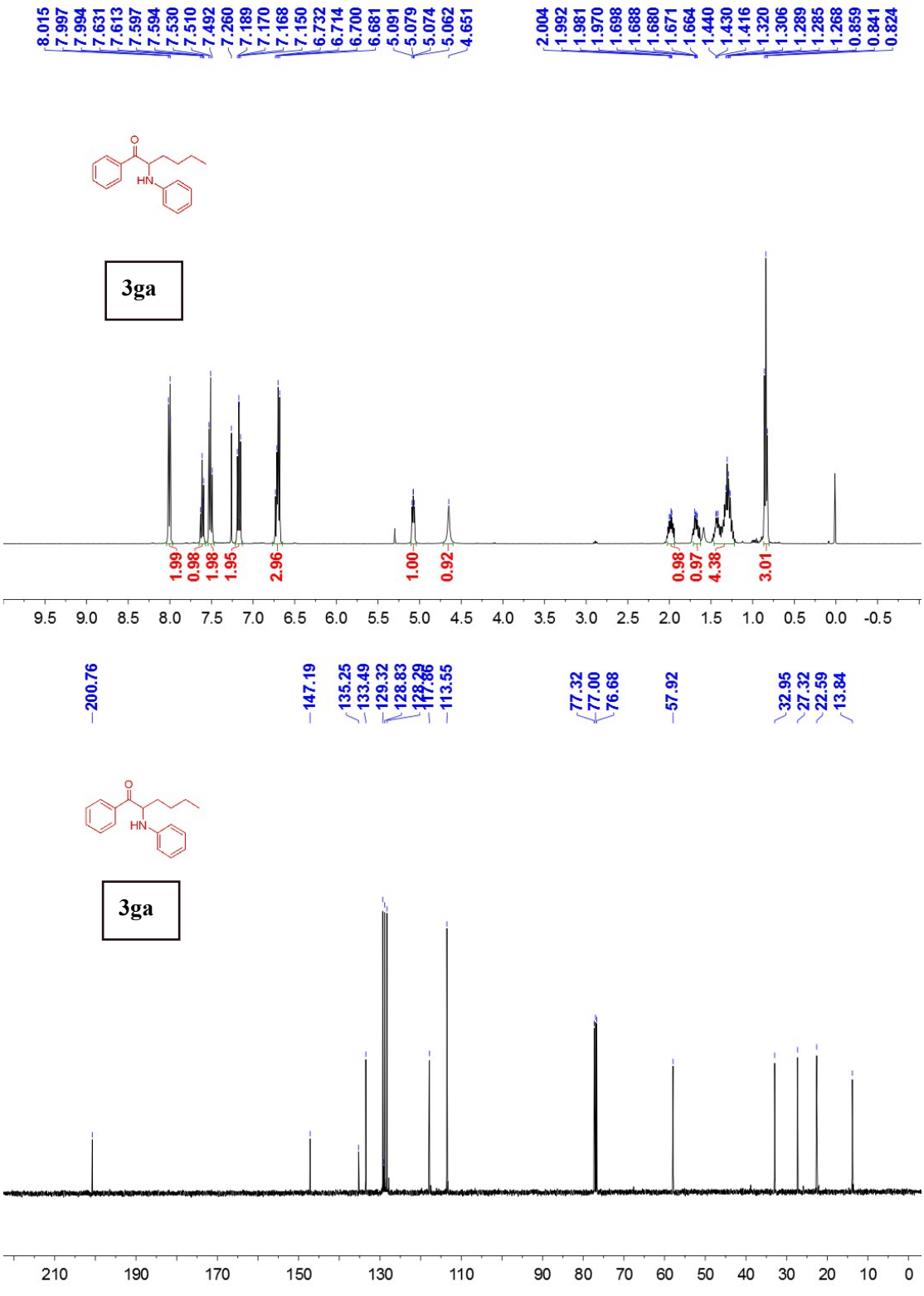
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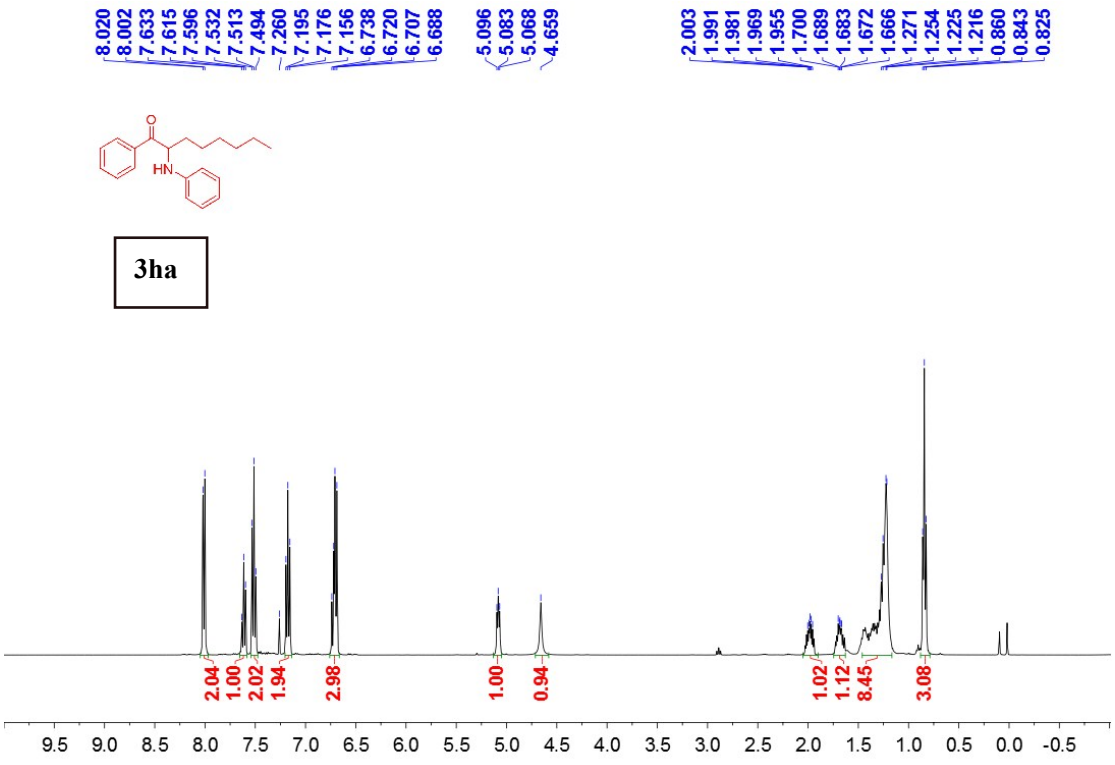
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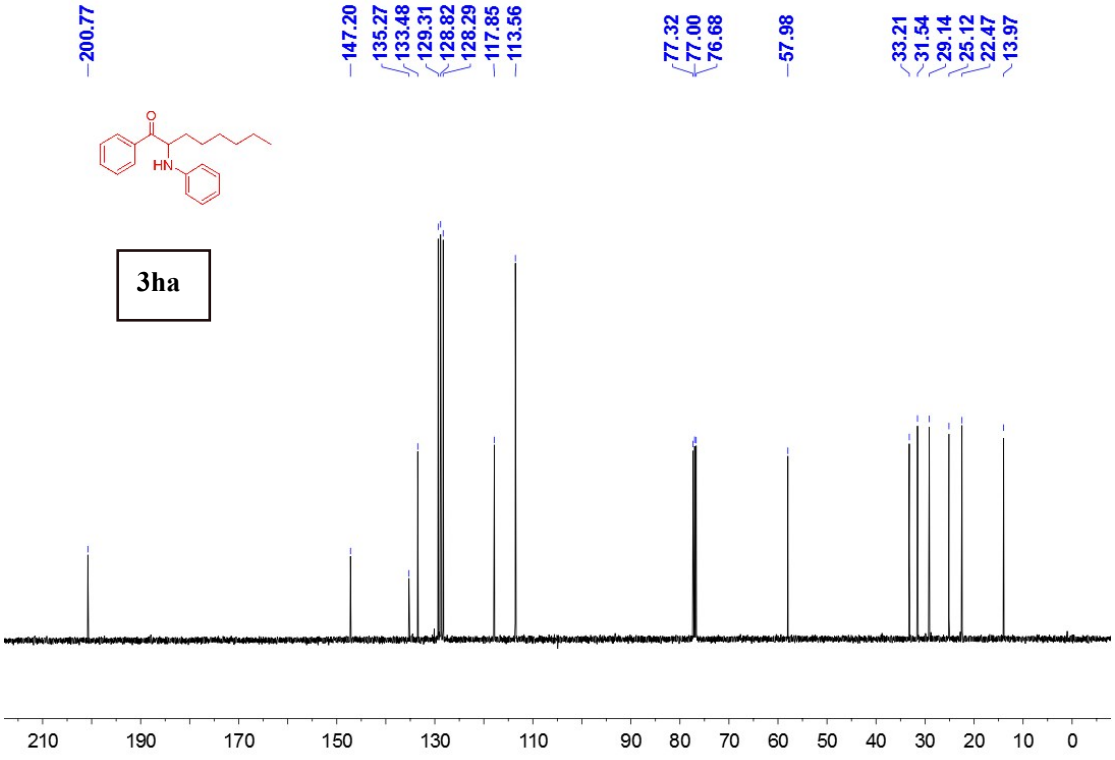
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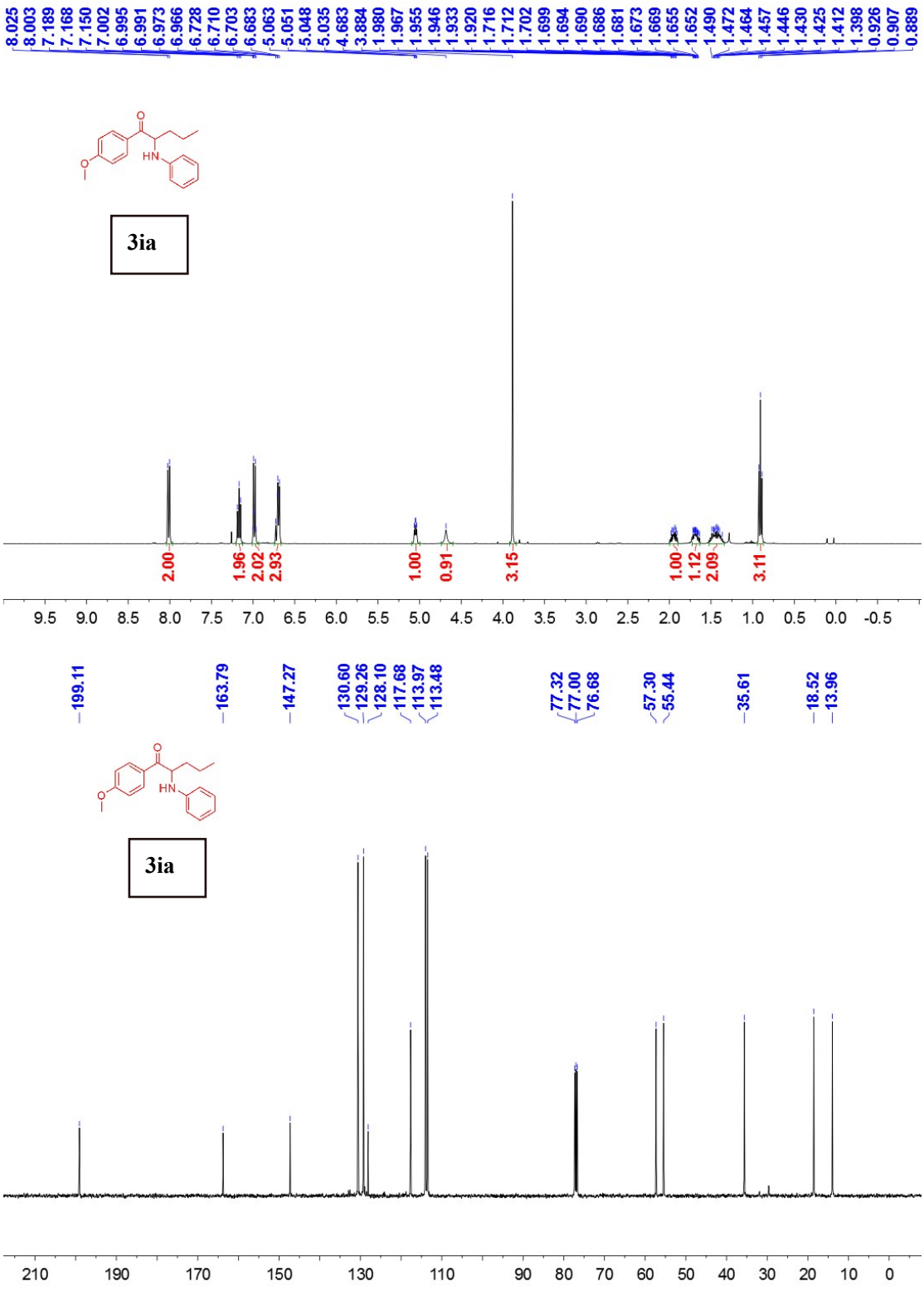
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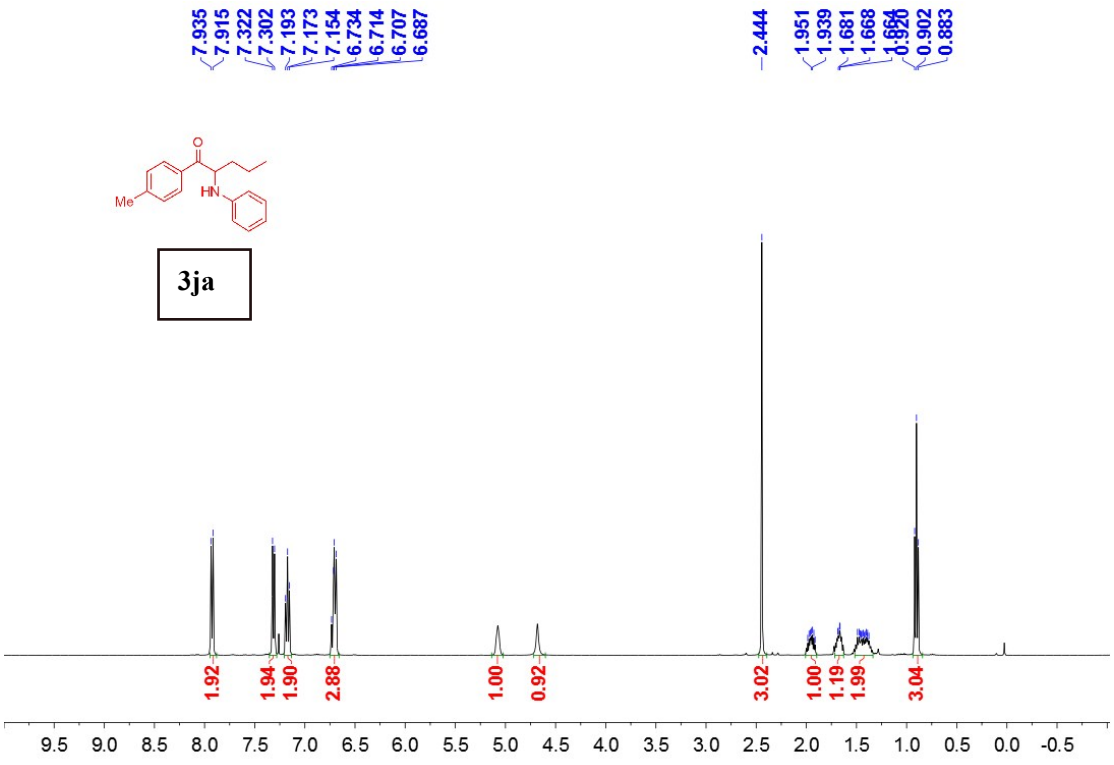
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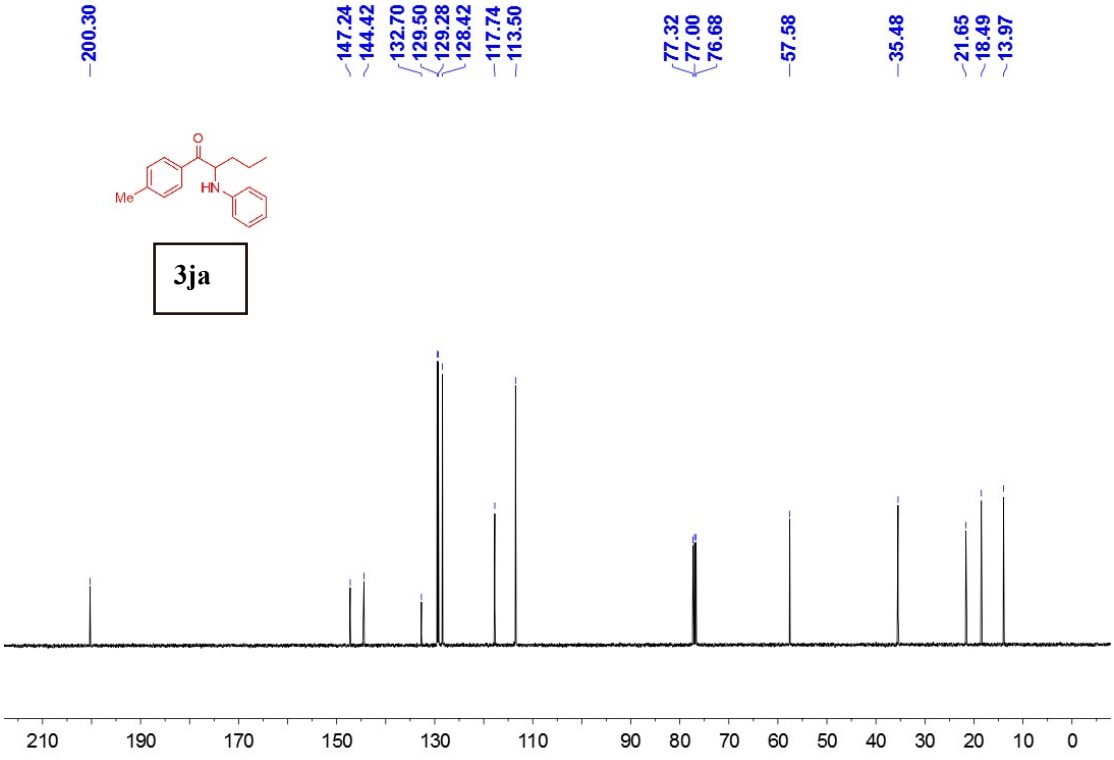
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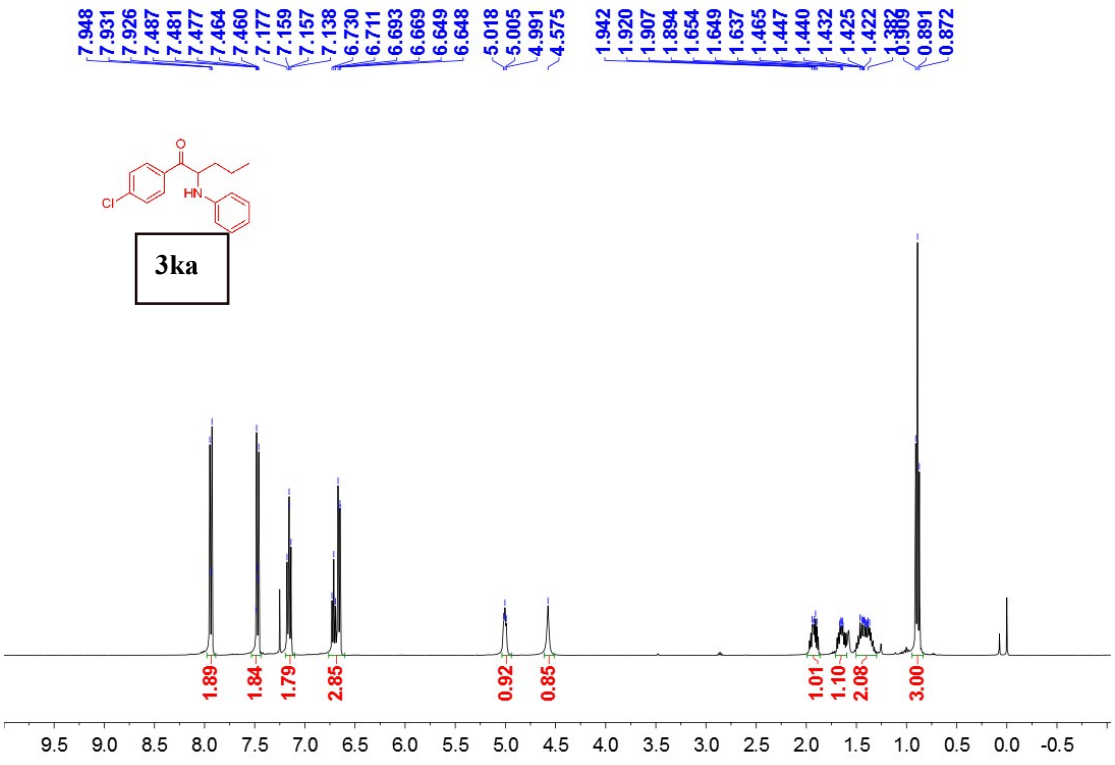
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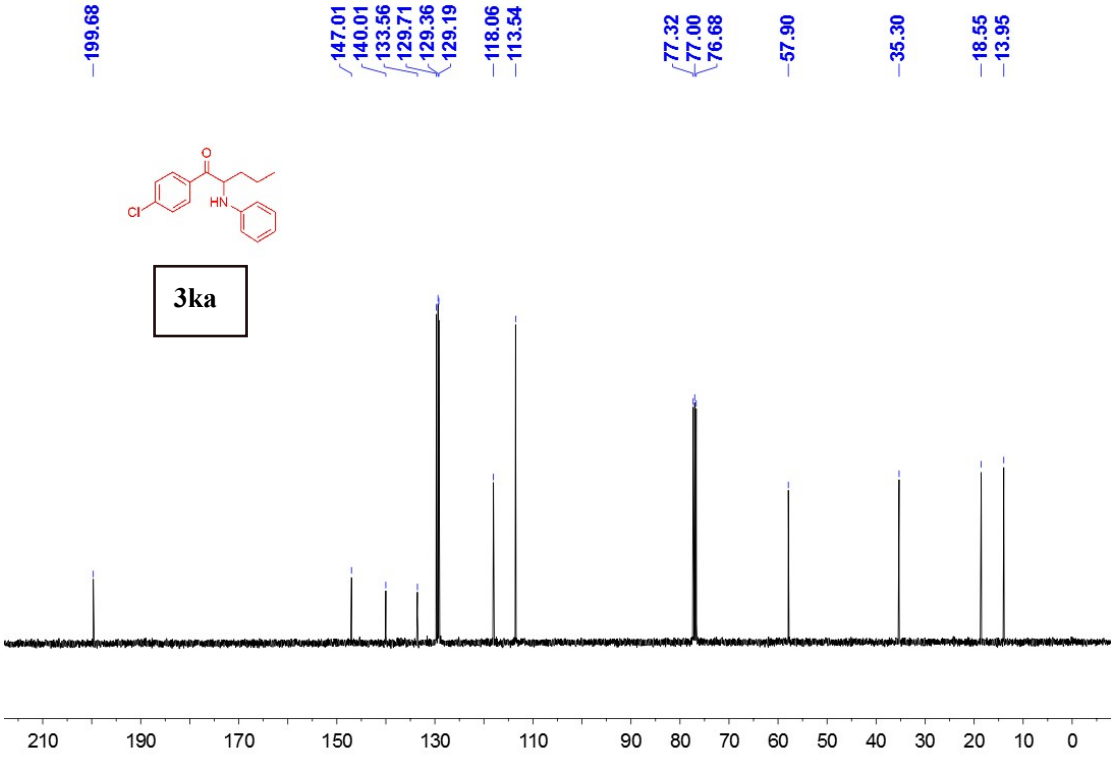
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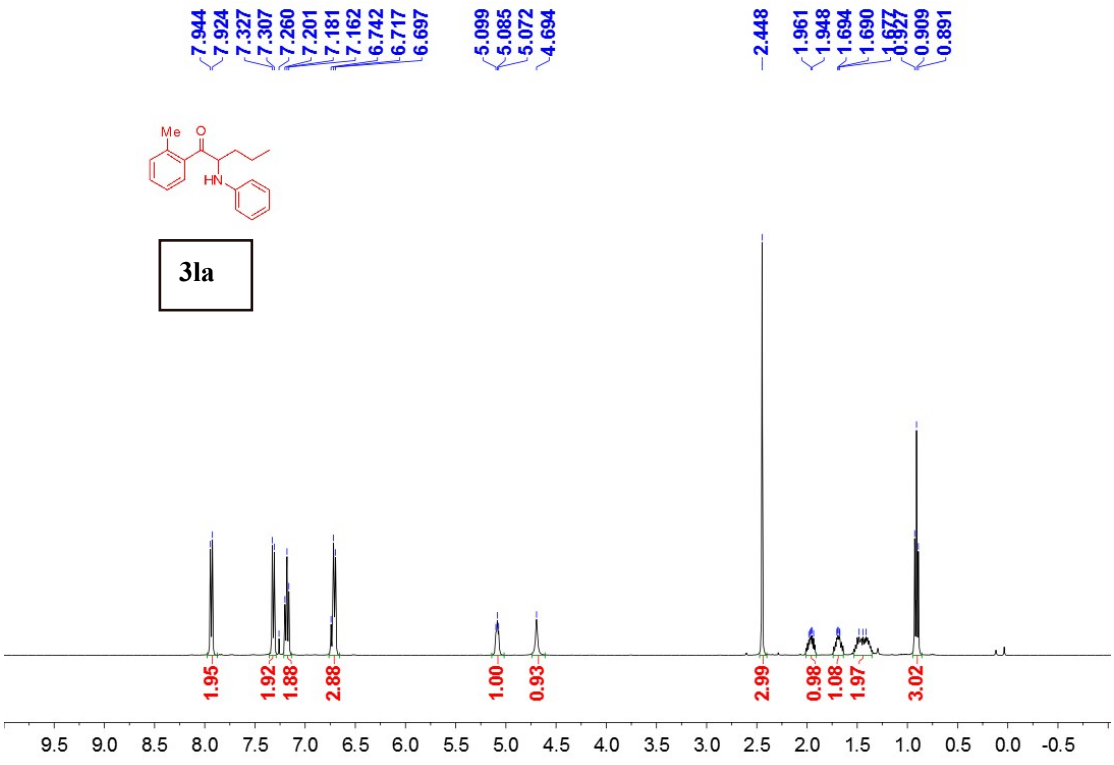
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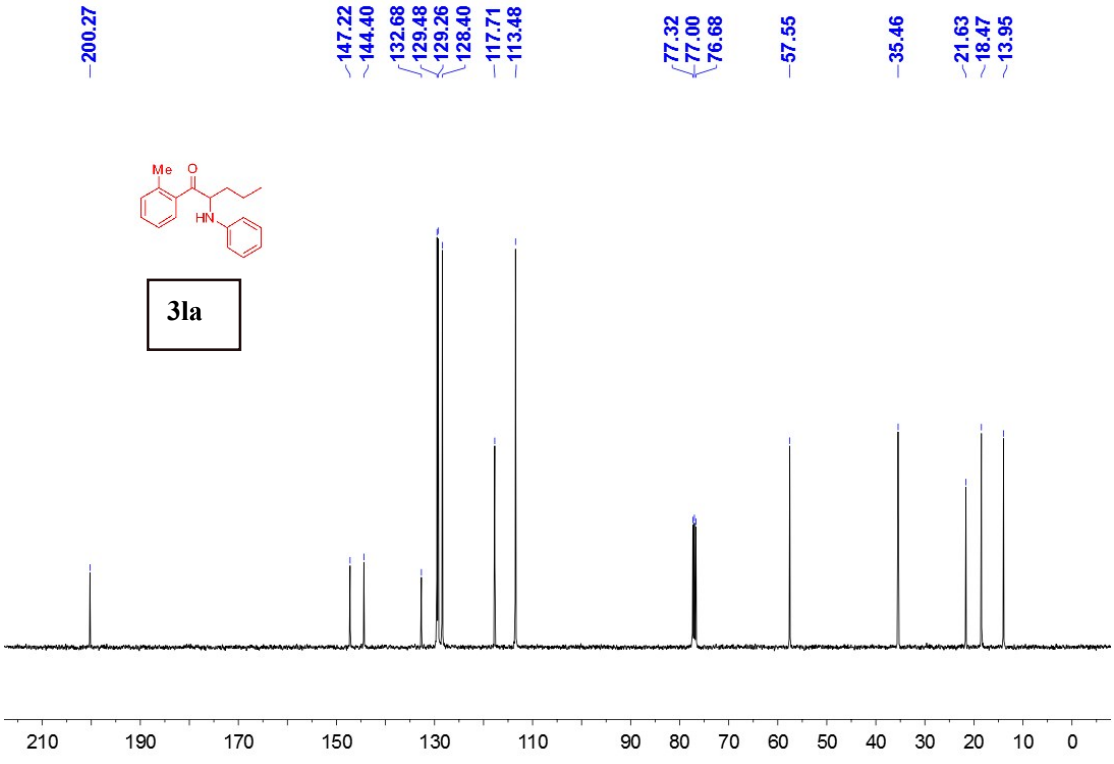
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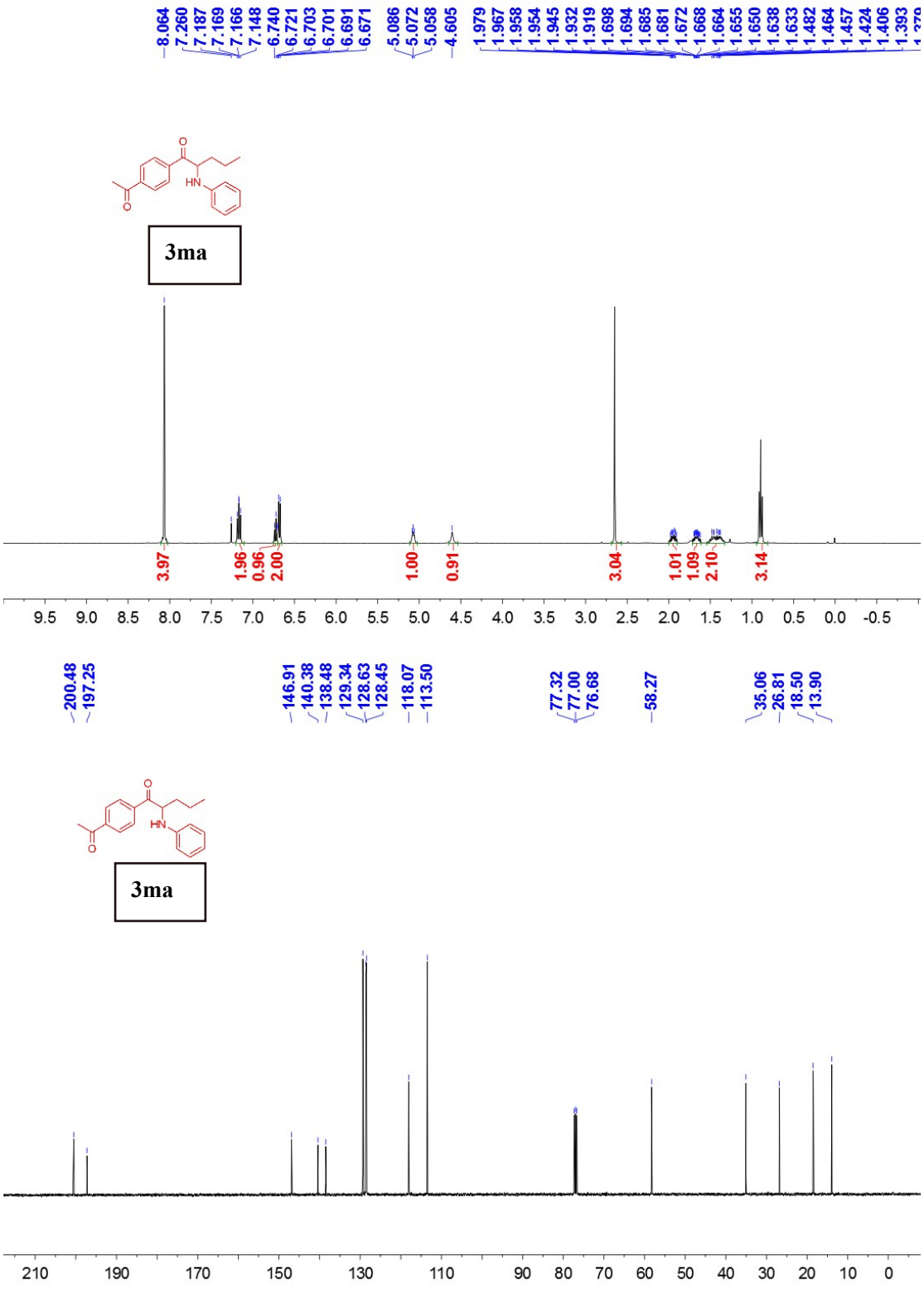
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1.00
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1.01
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2.10
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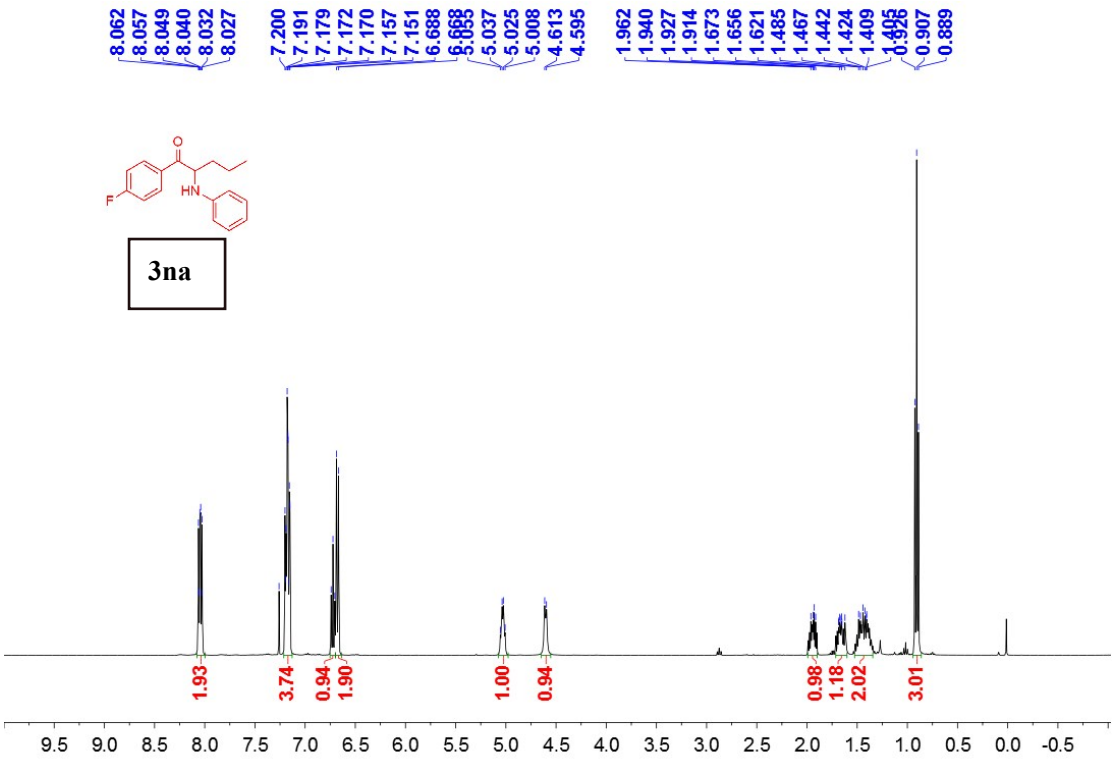
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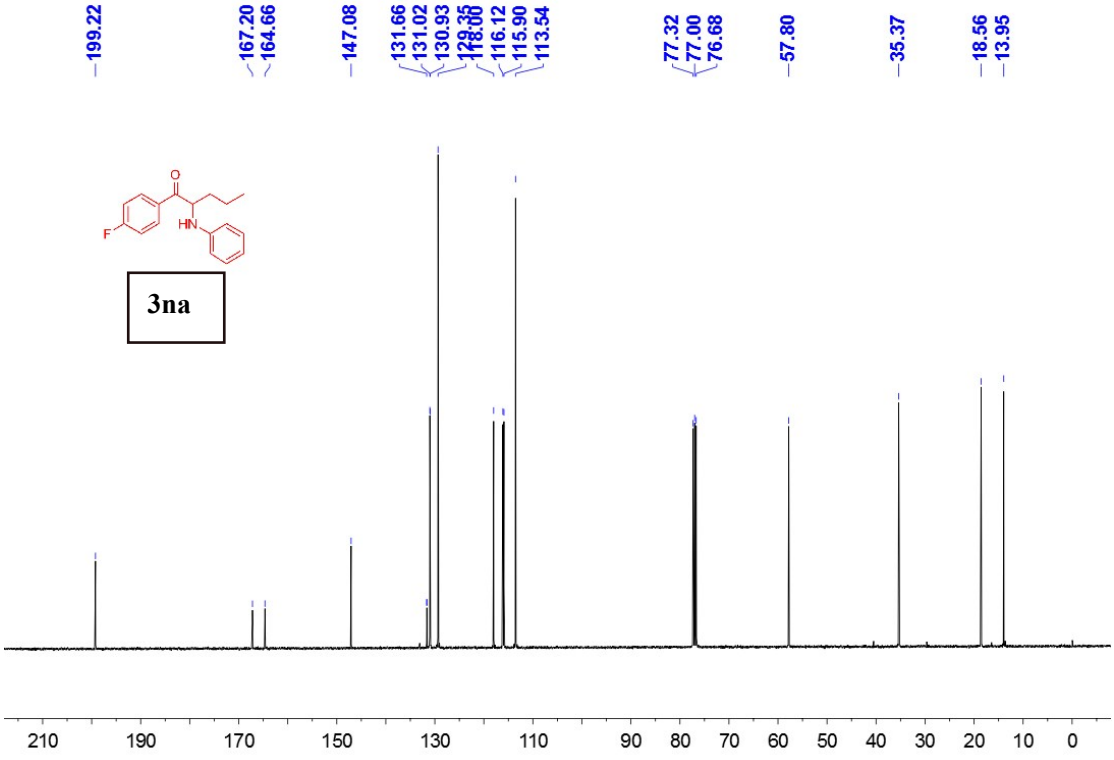
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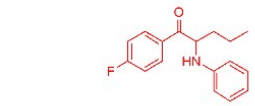

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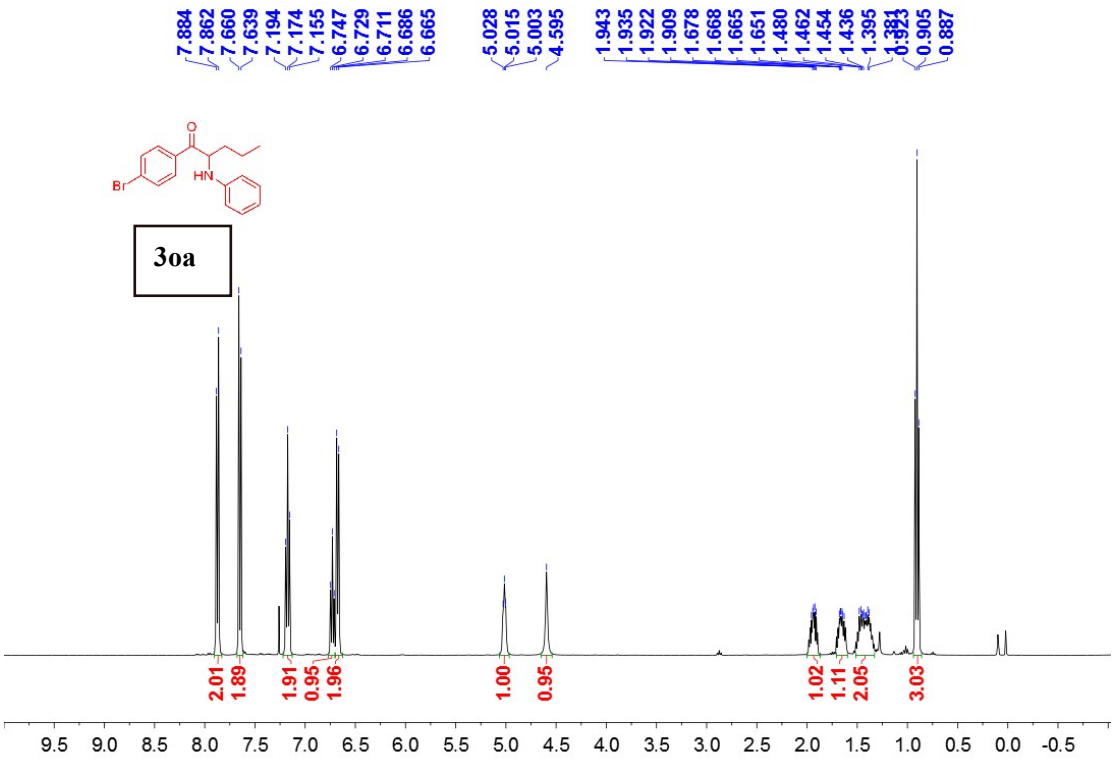
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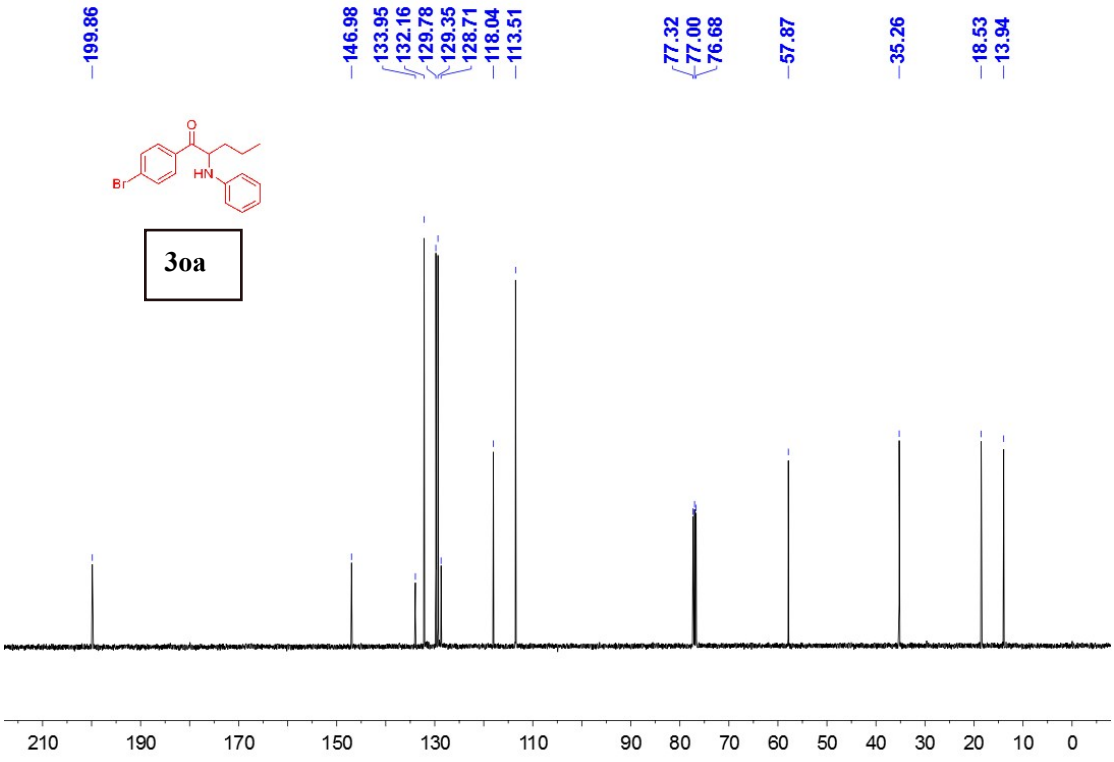
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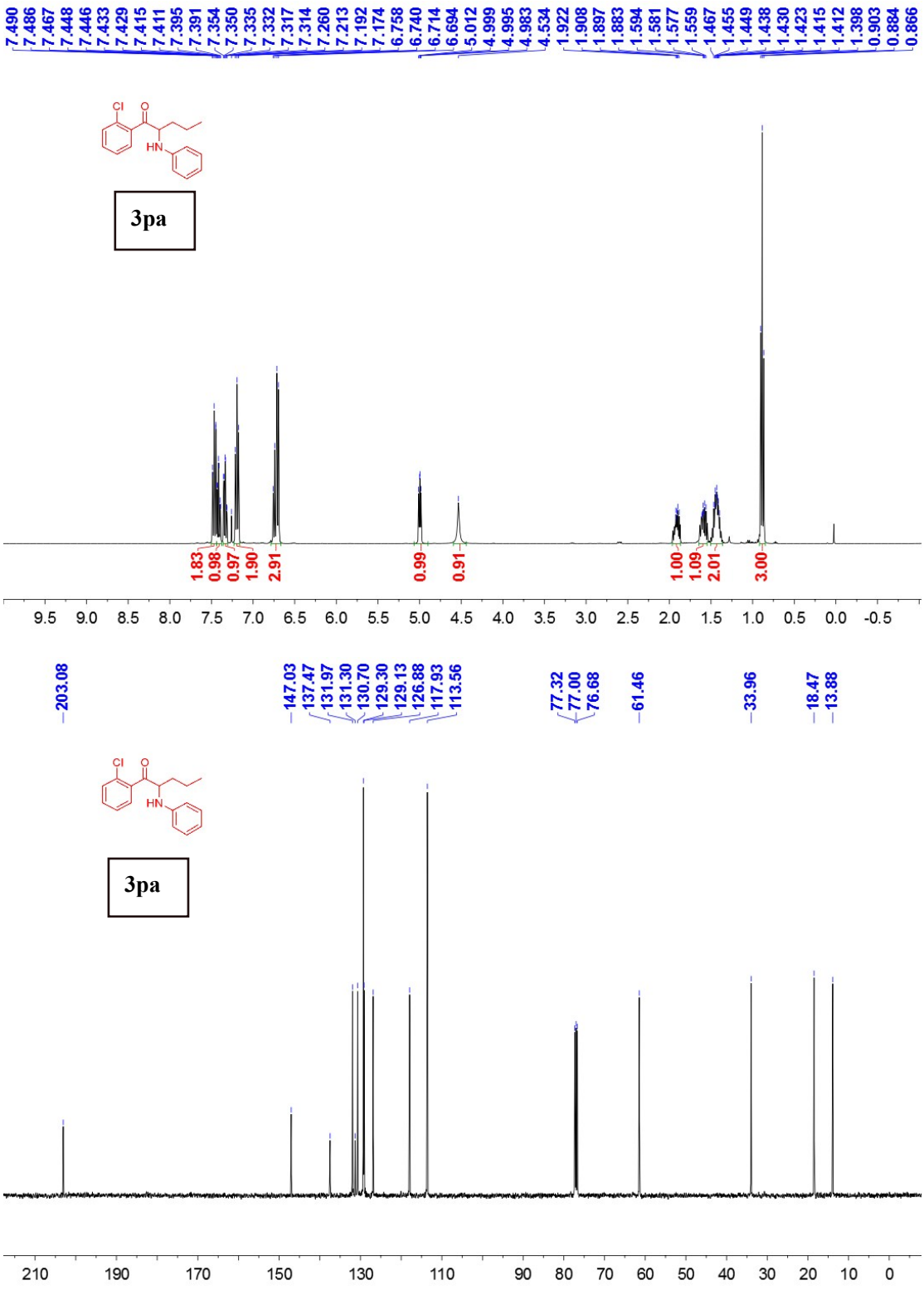
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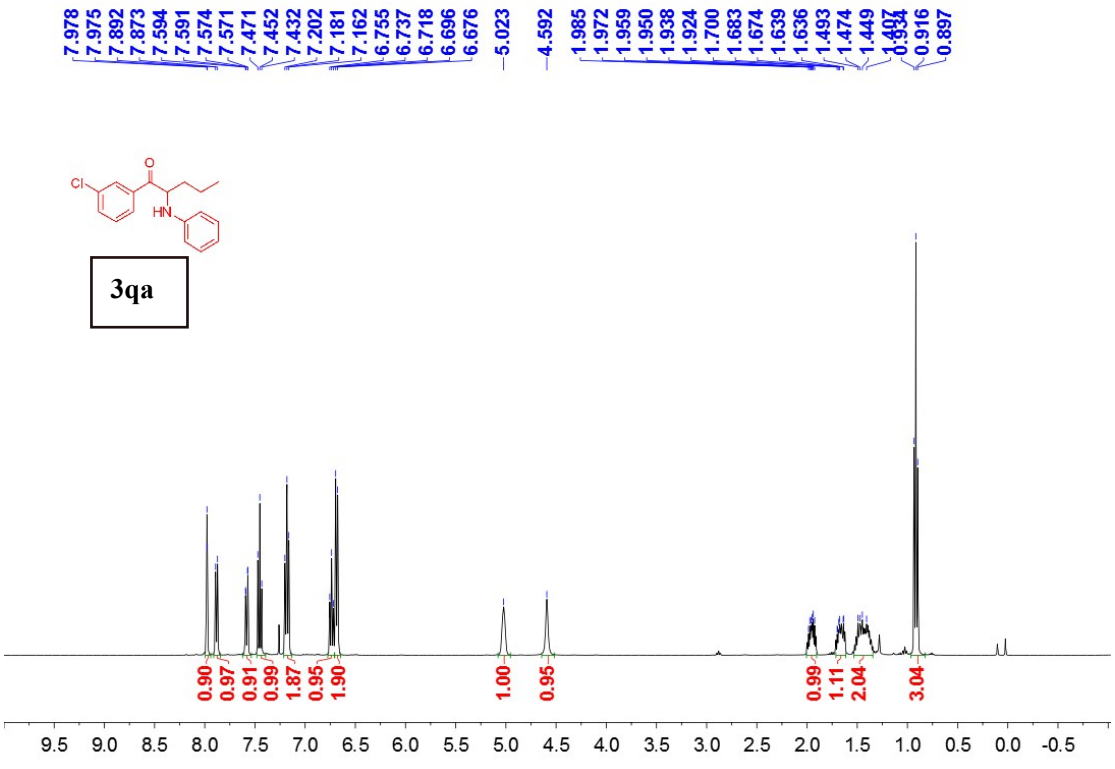
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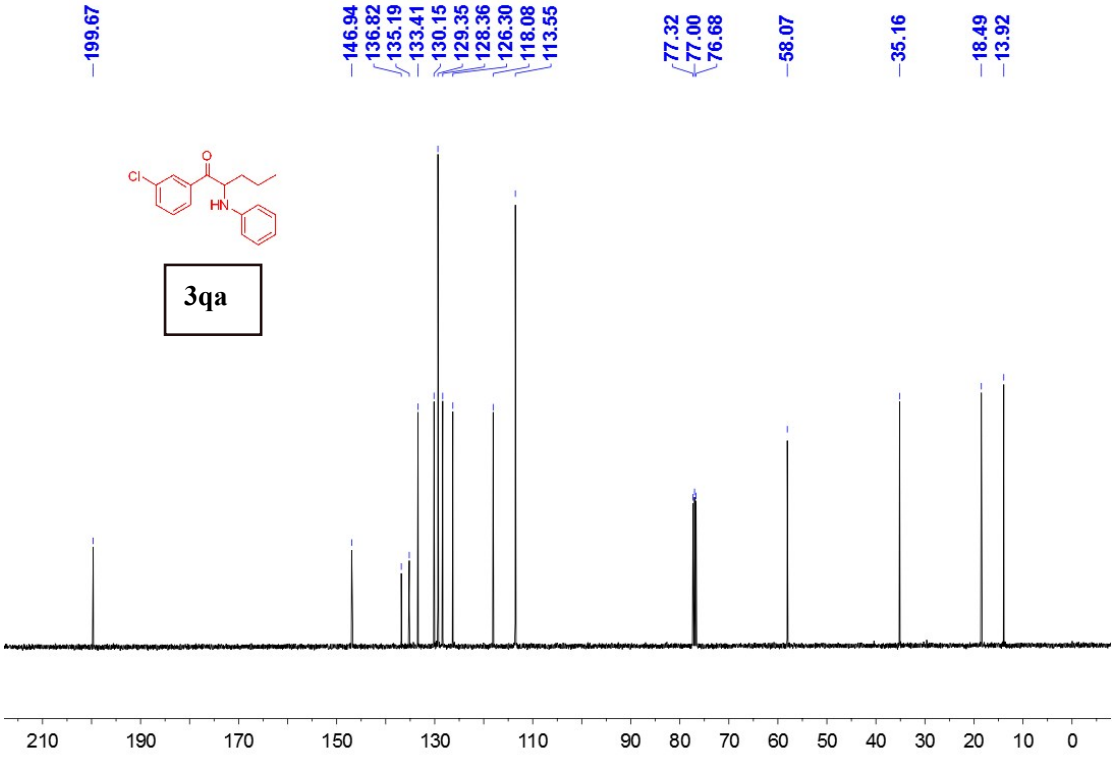
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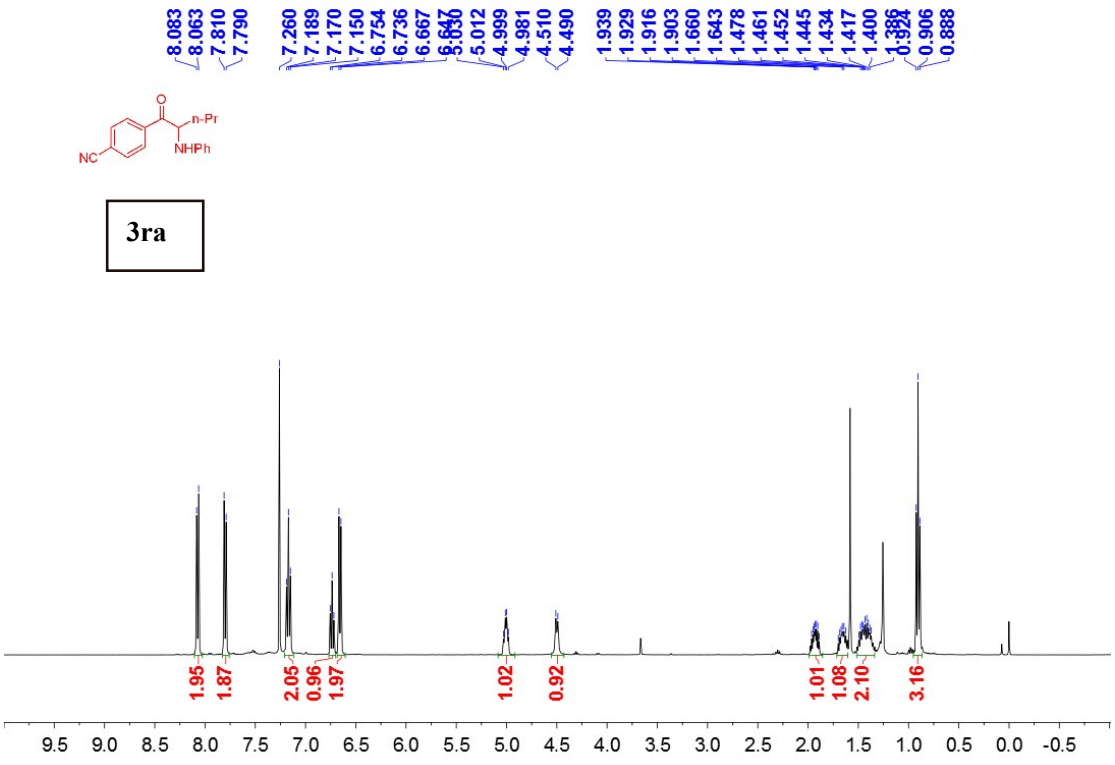
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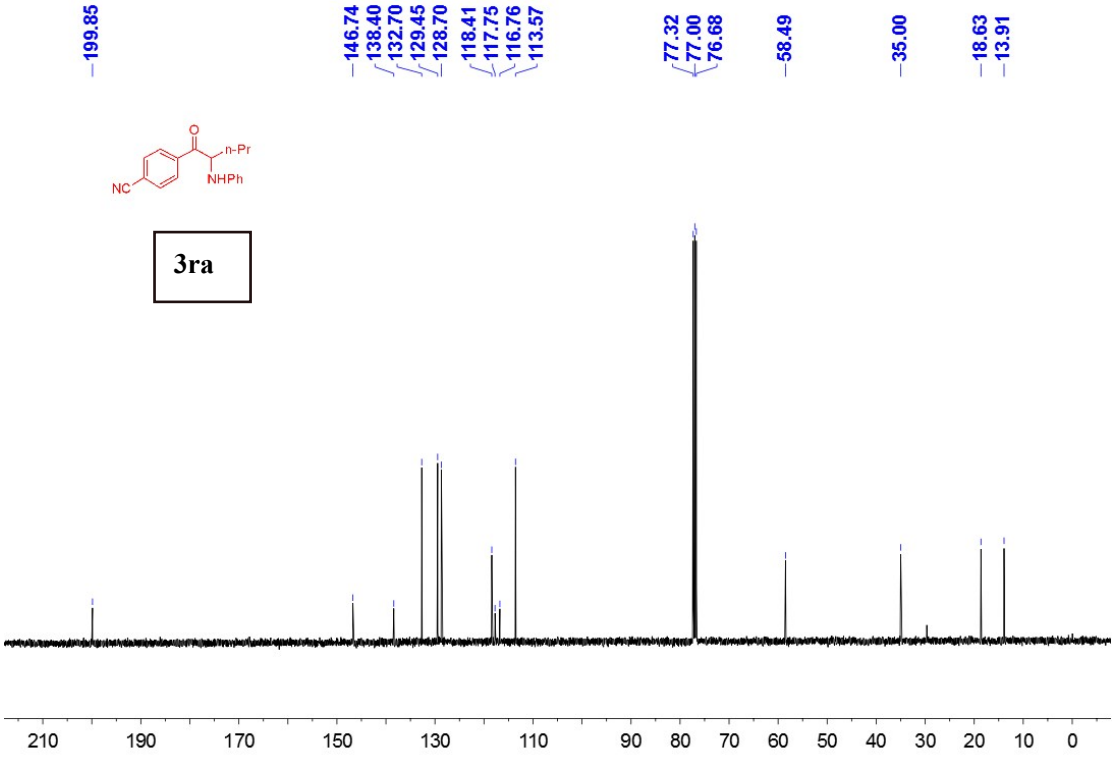
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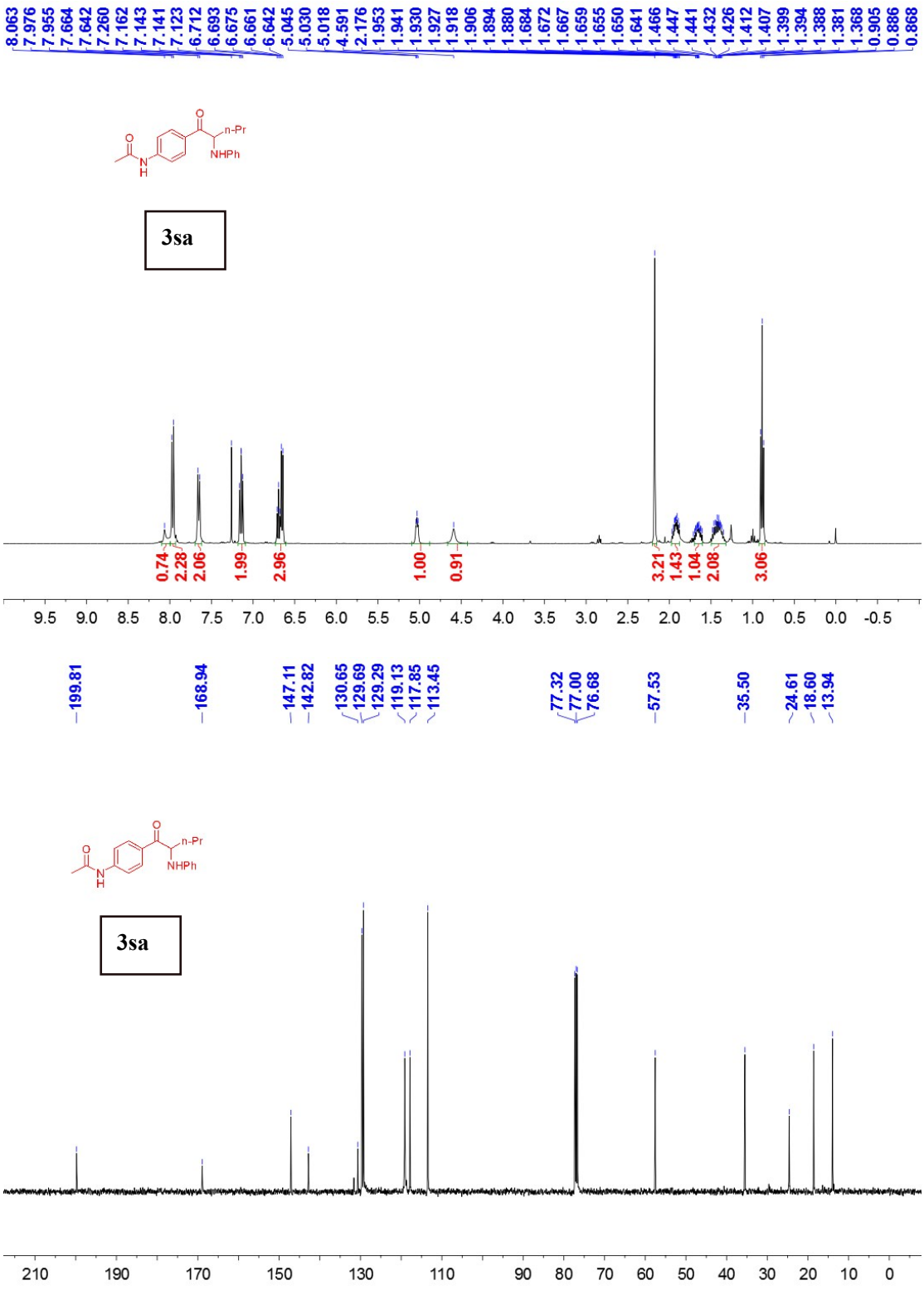
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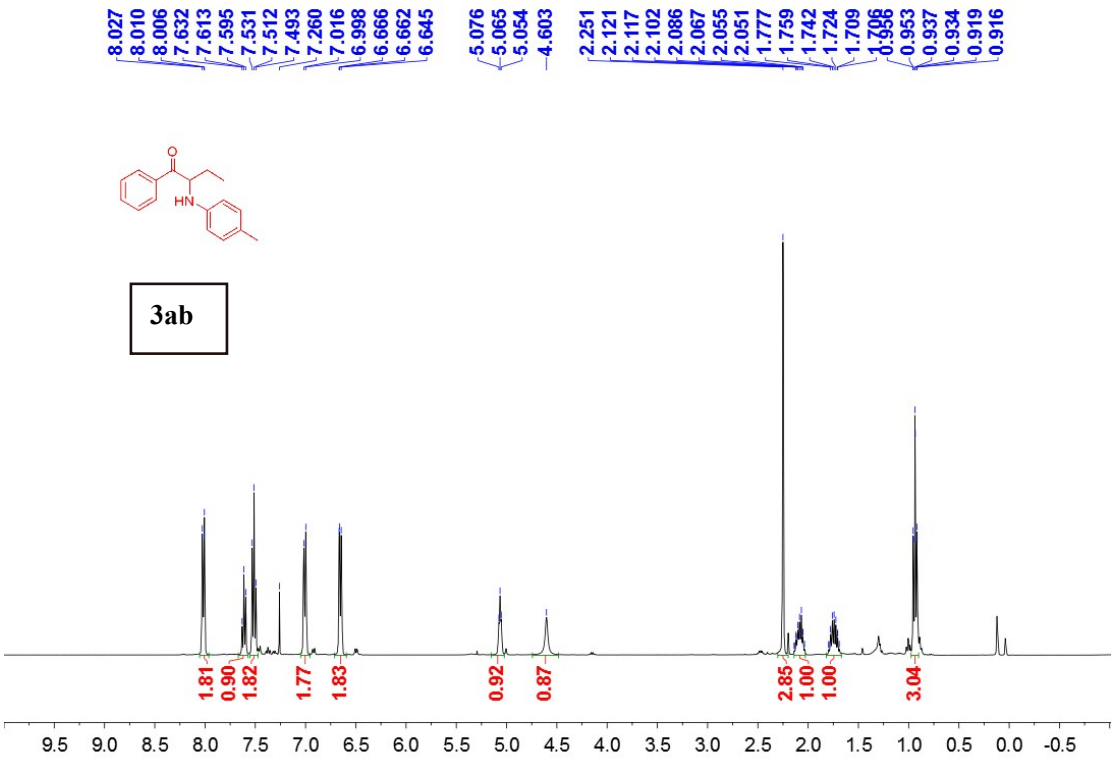
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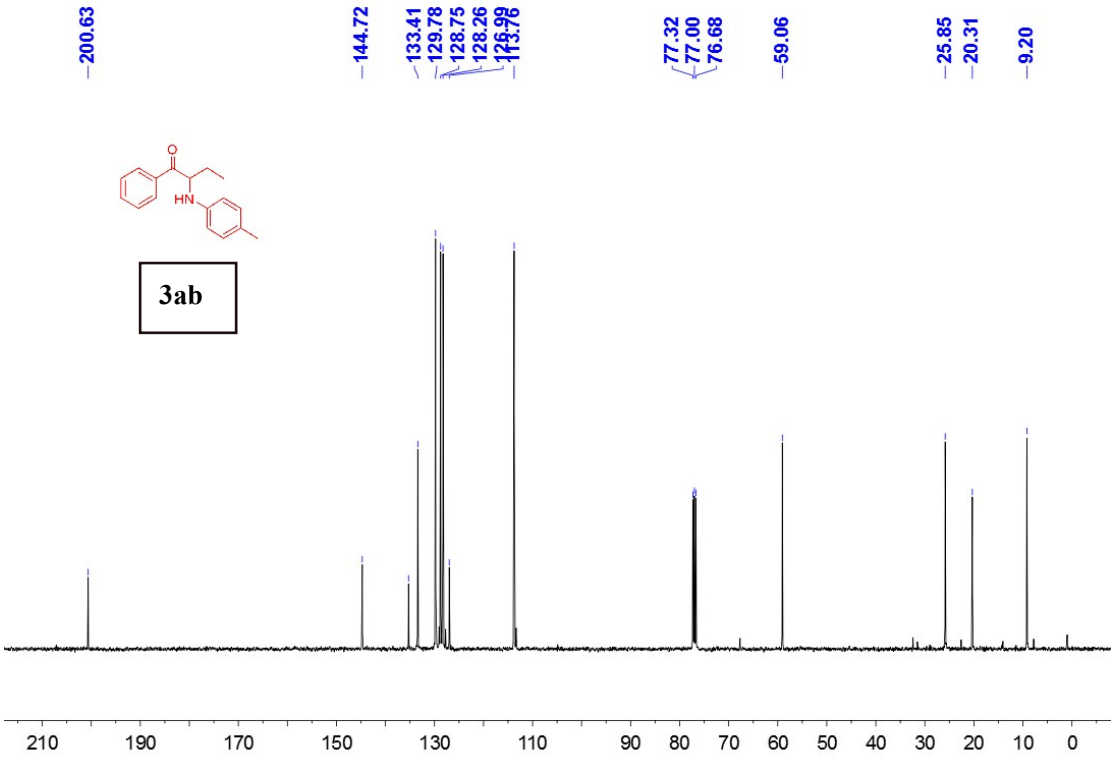
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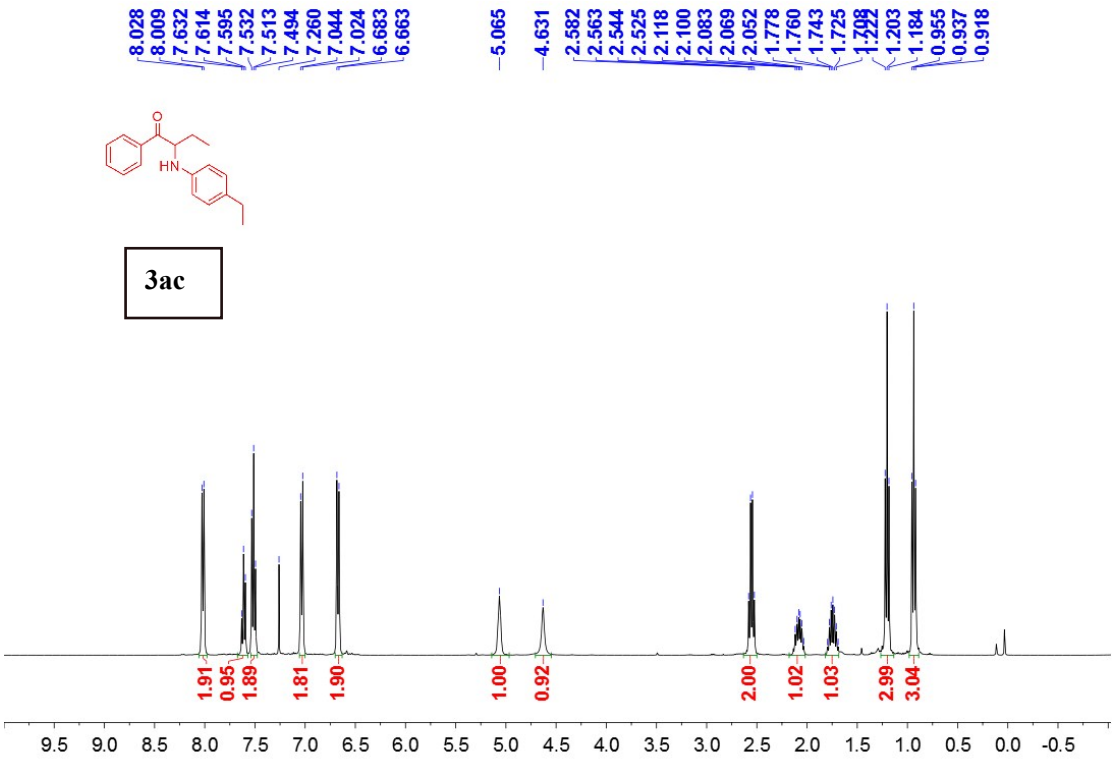
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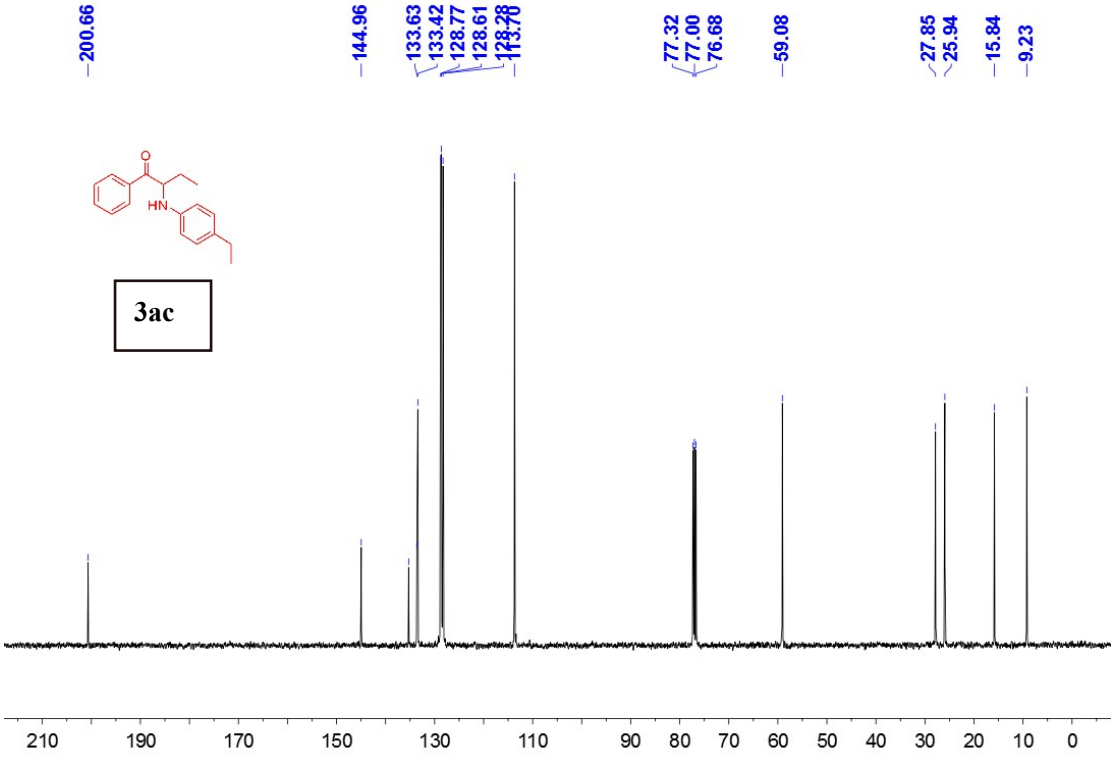
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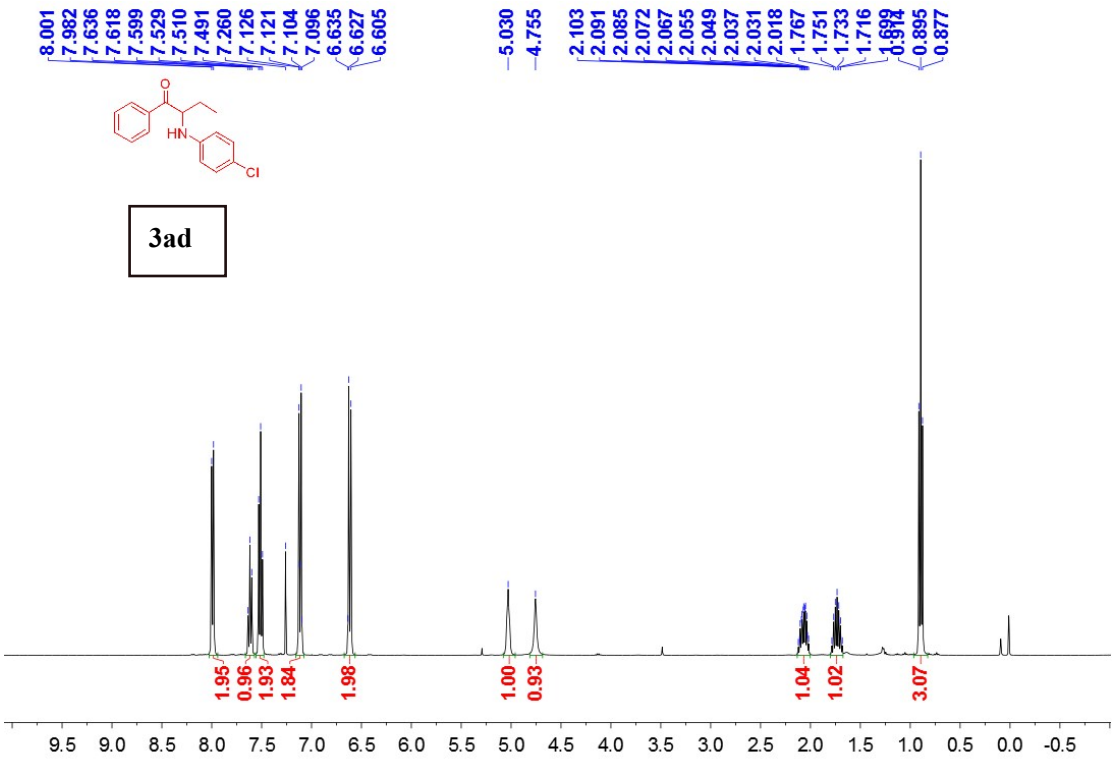
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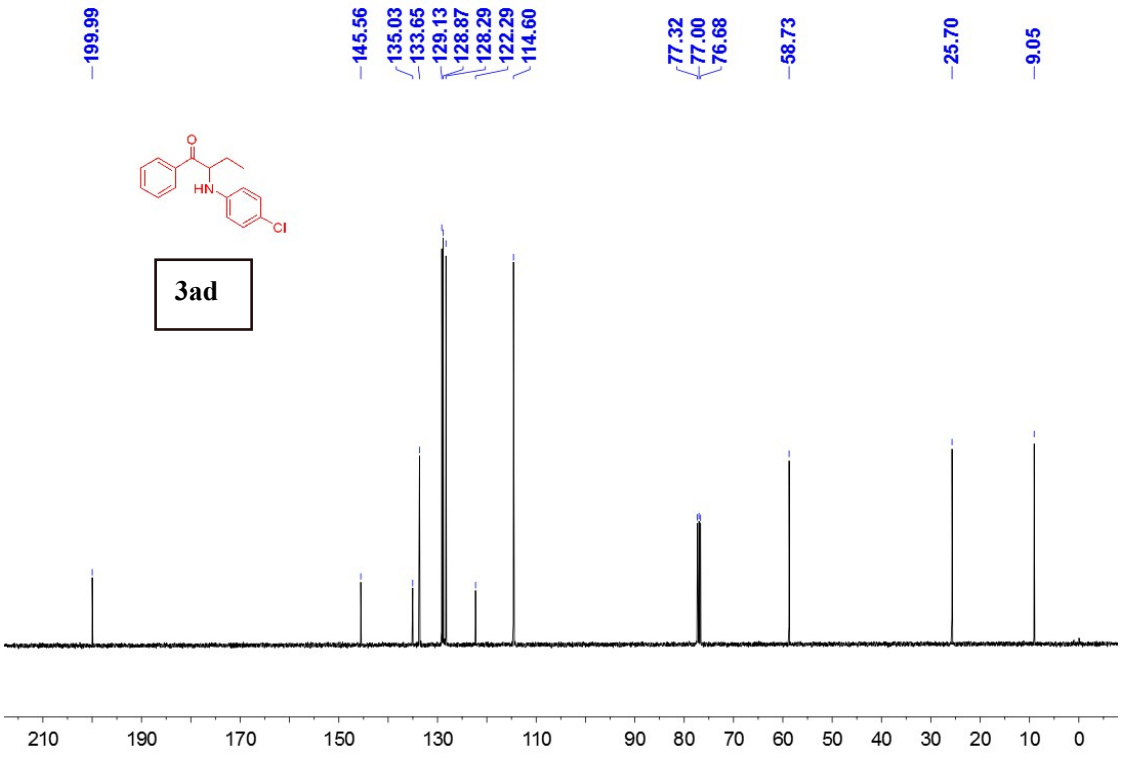
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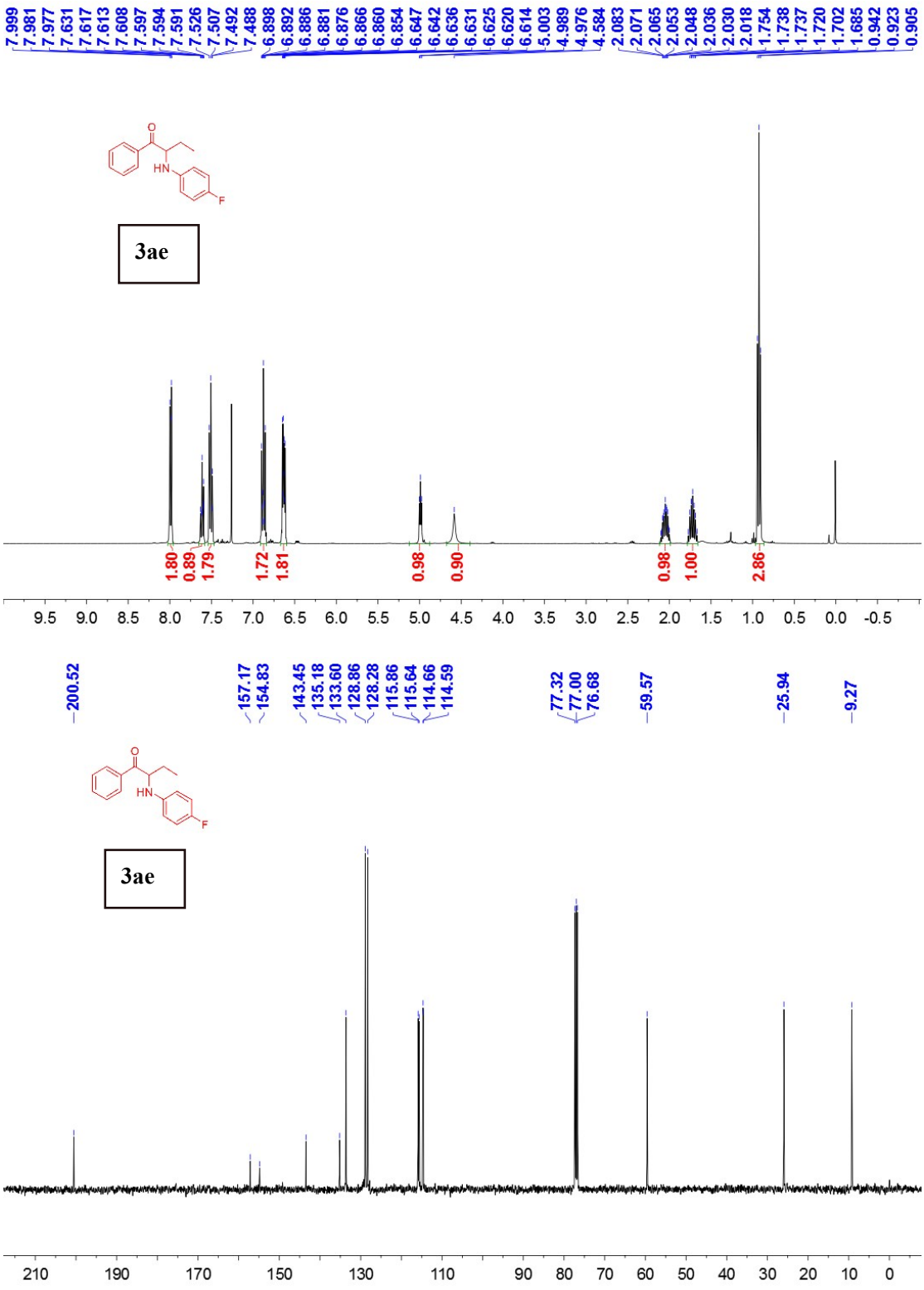
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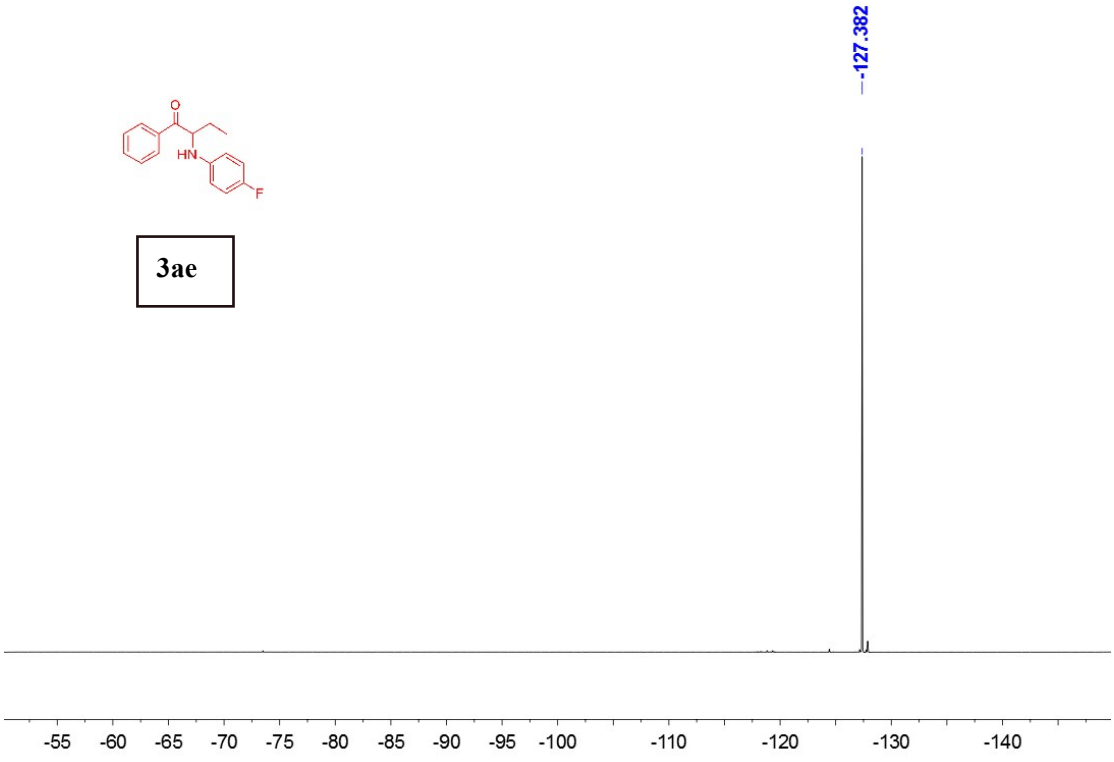
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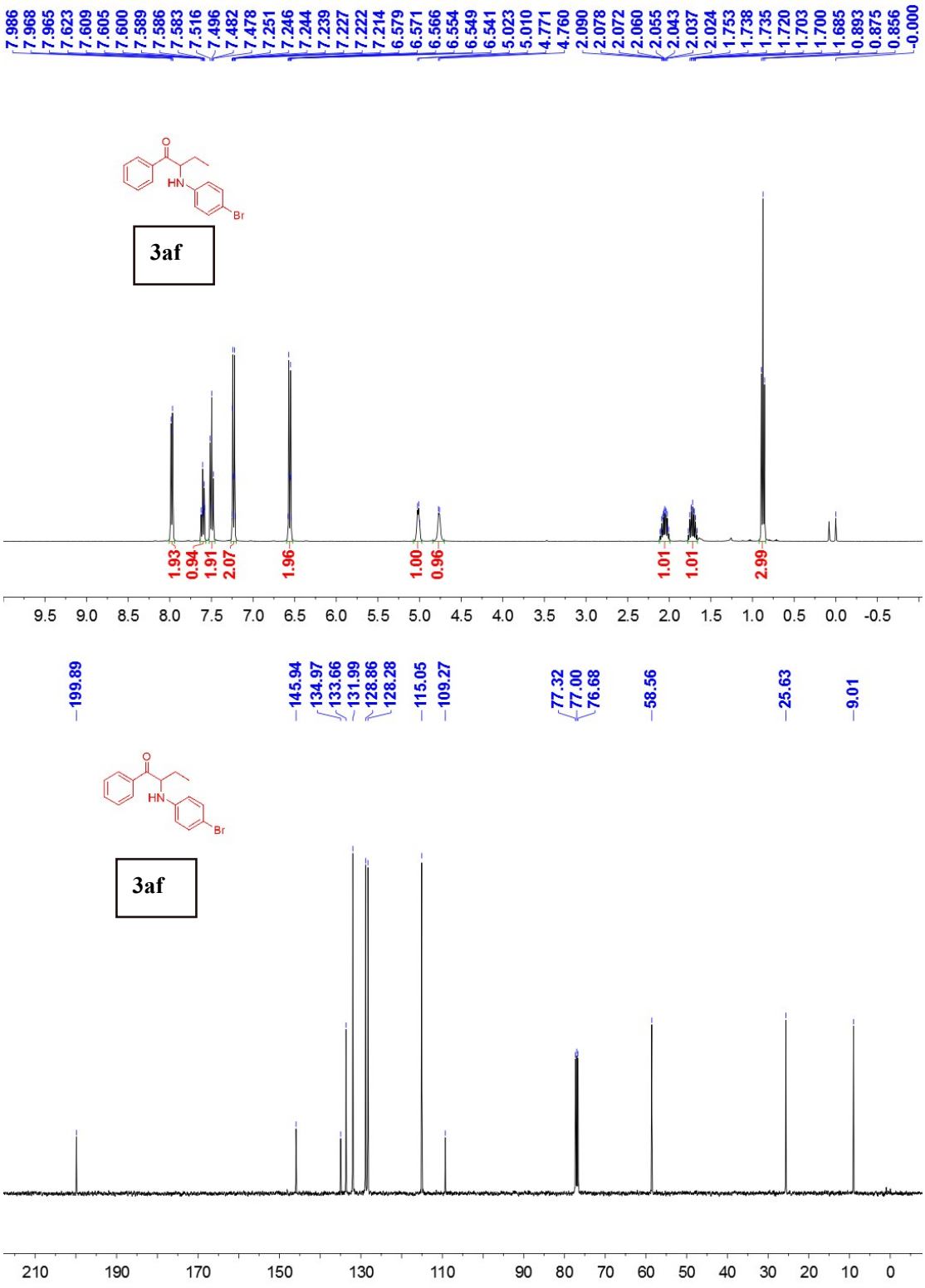
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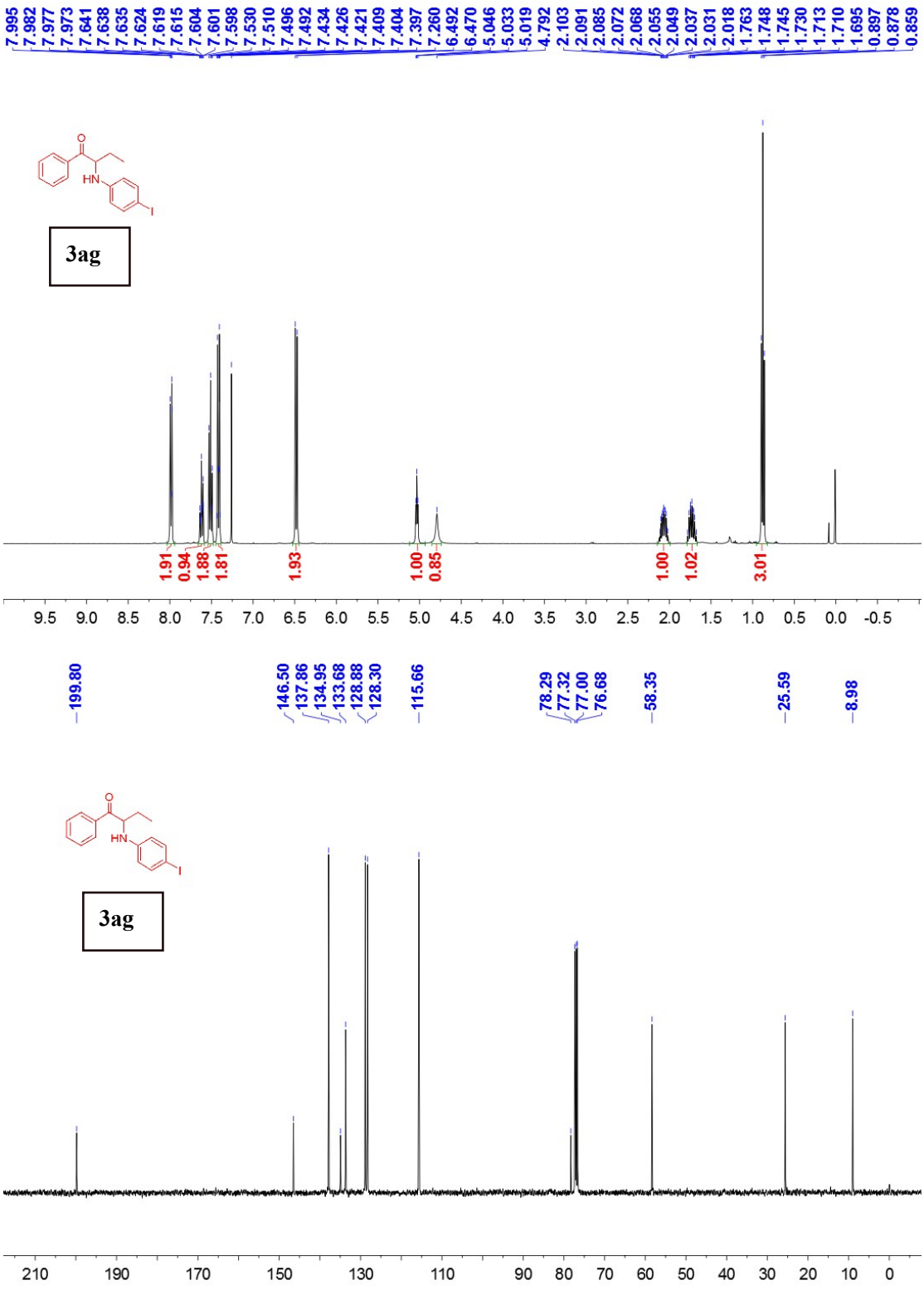
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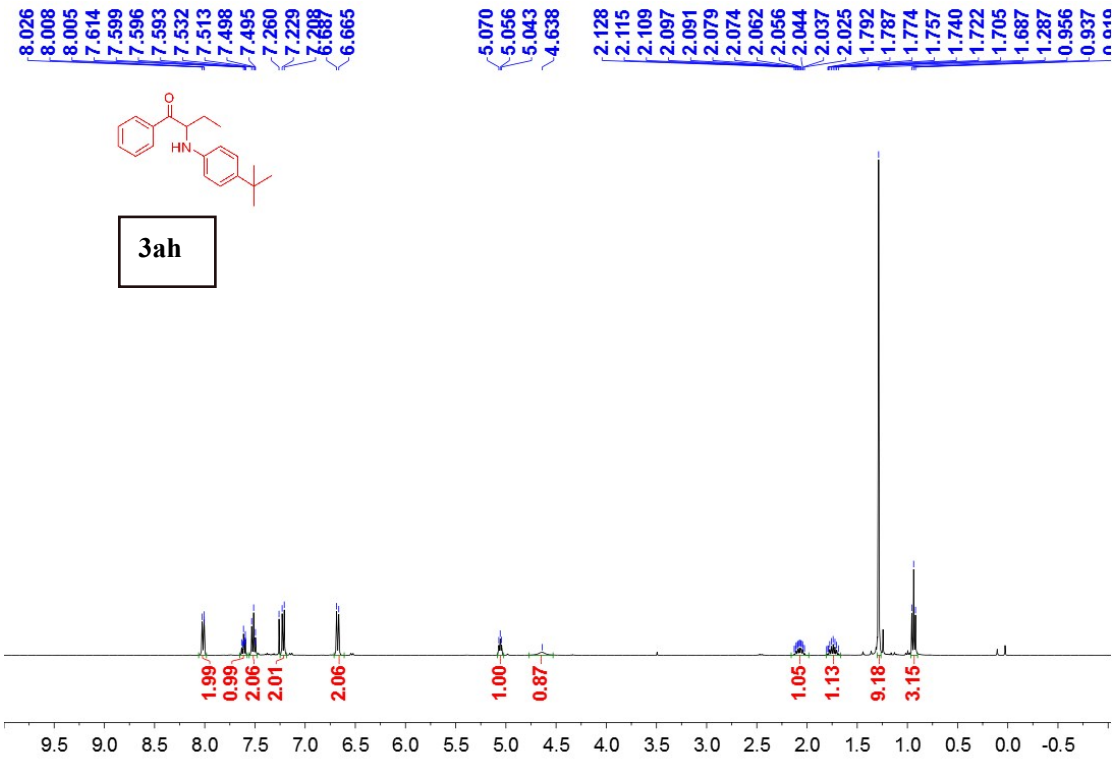
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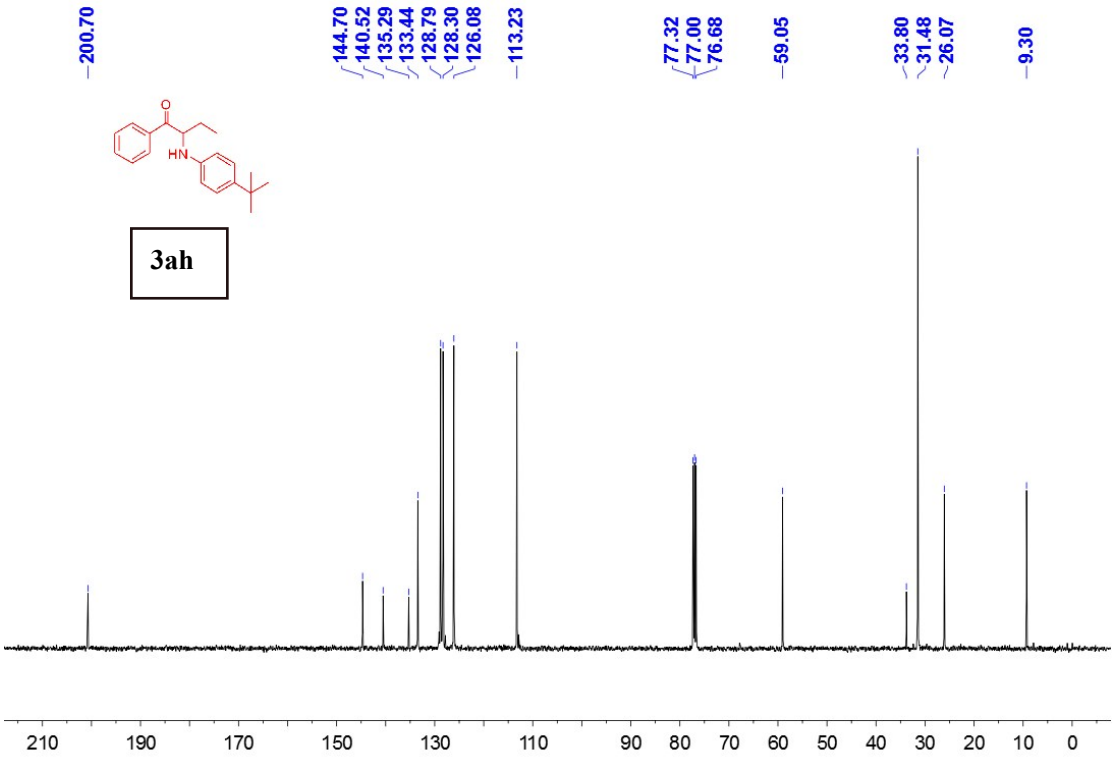
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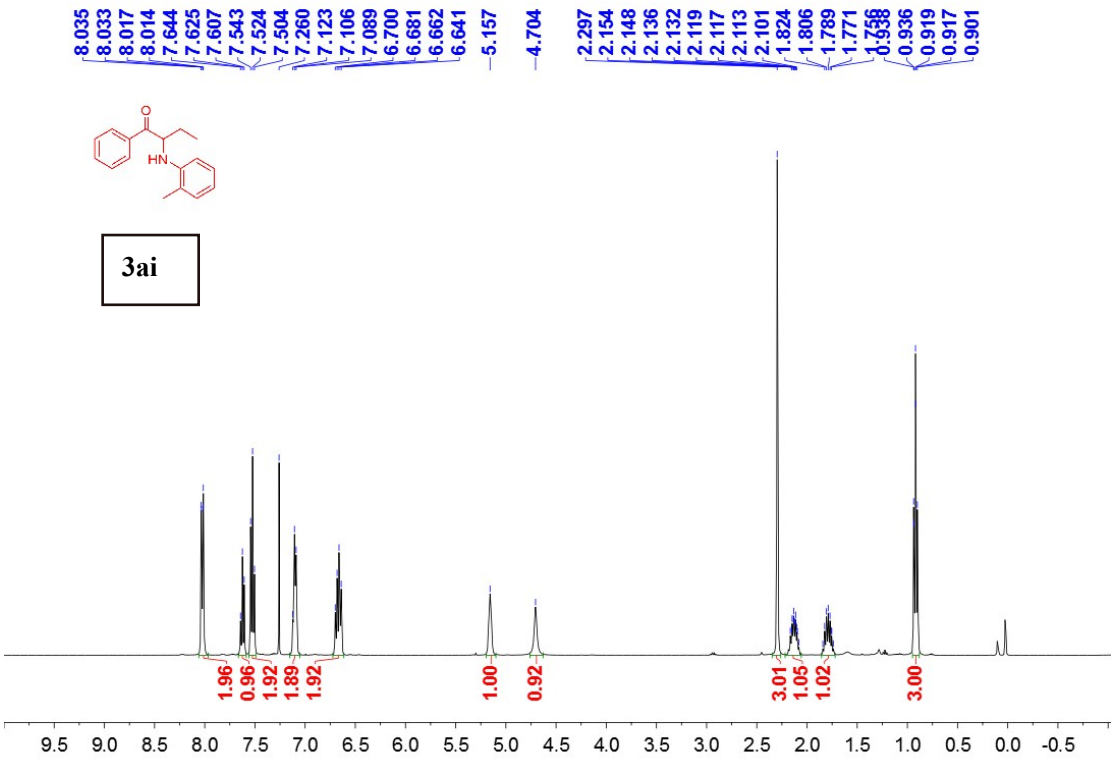
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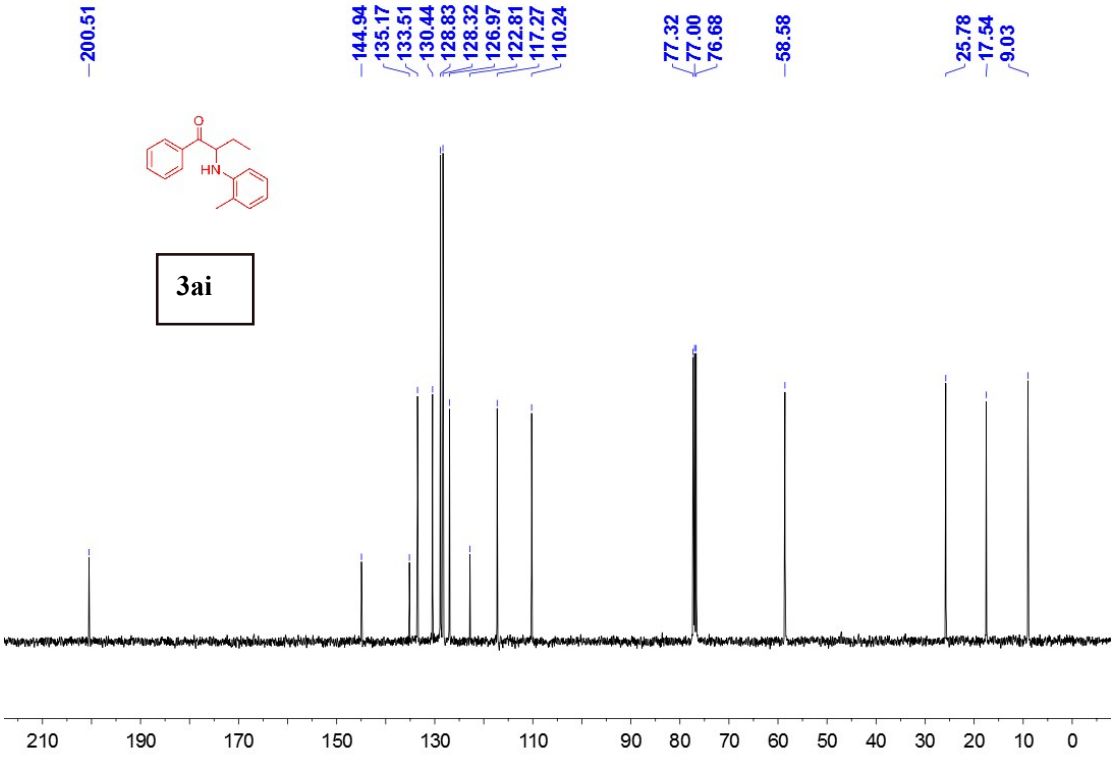
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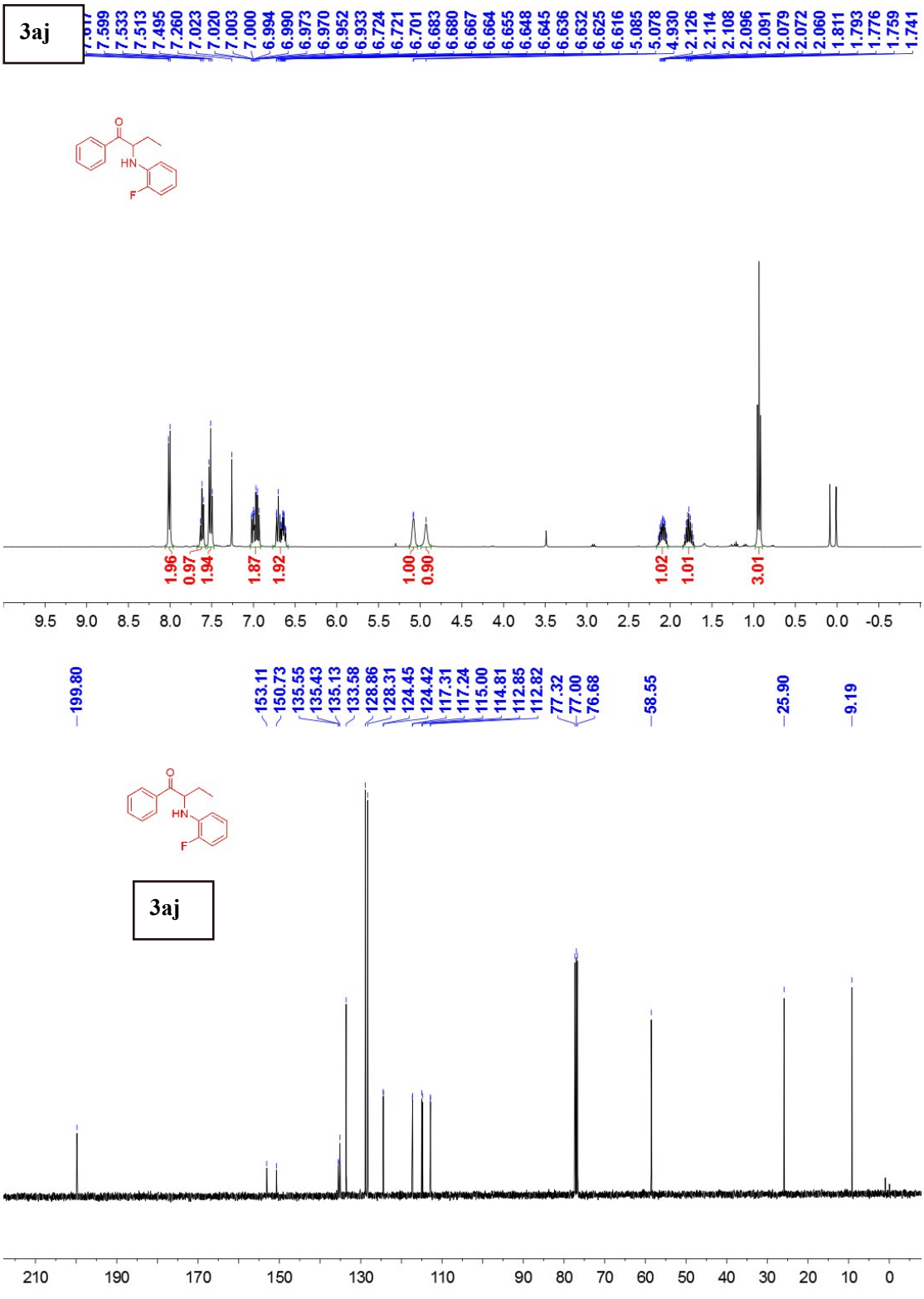
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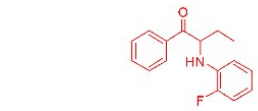

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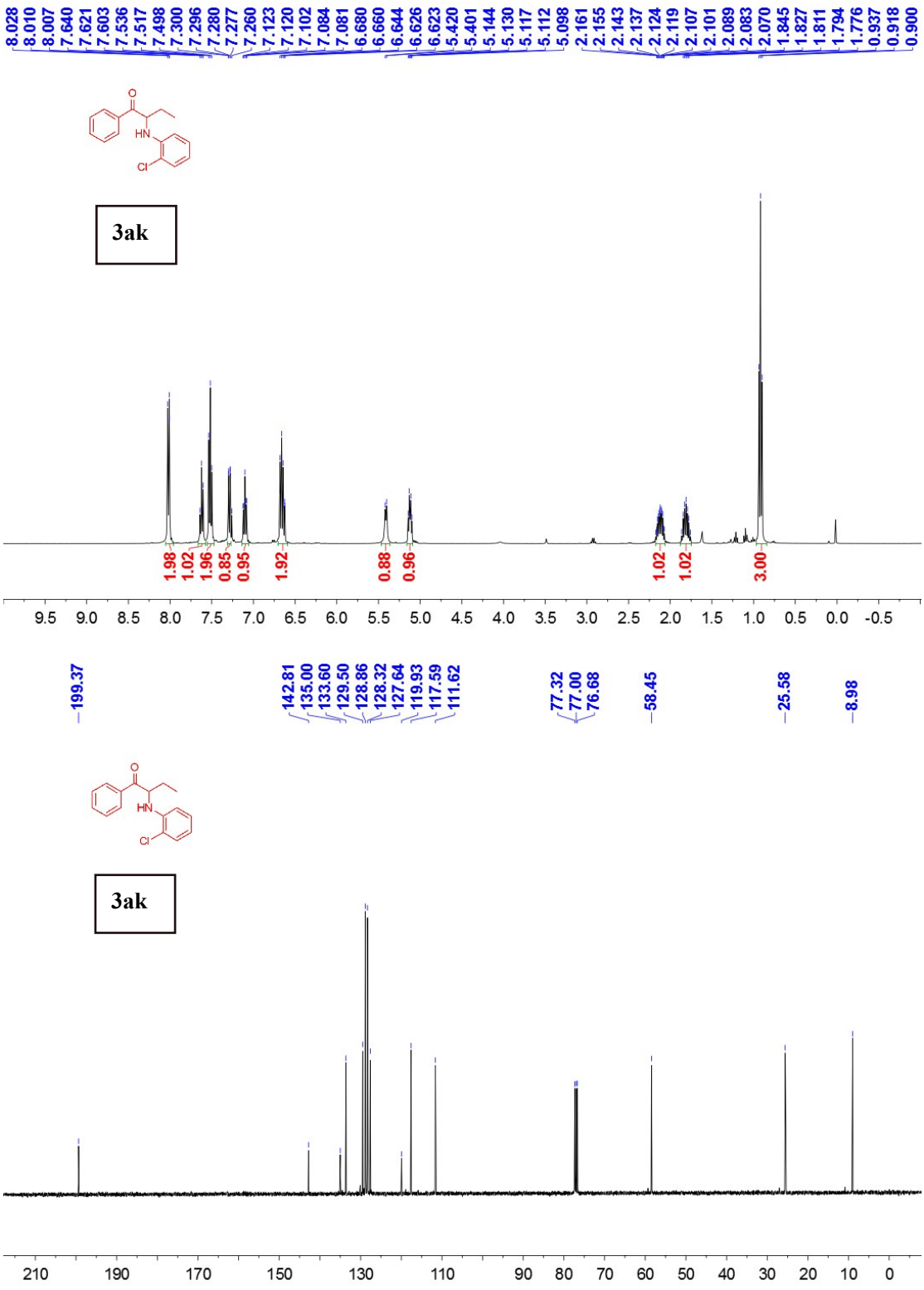
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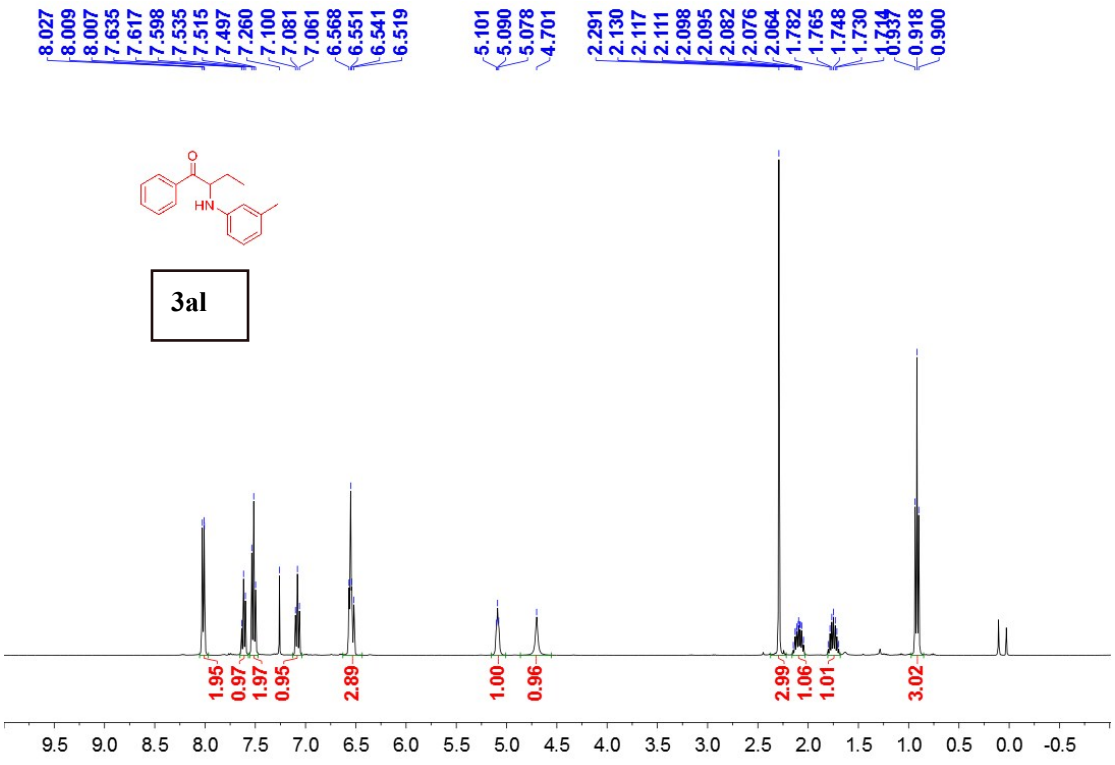
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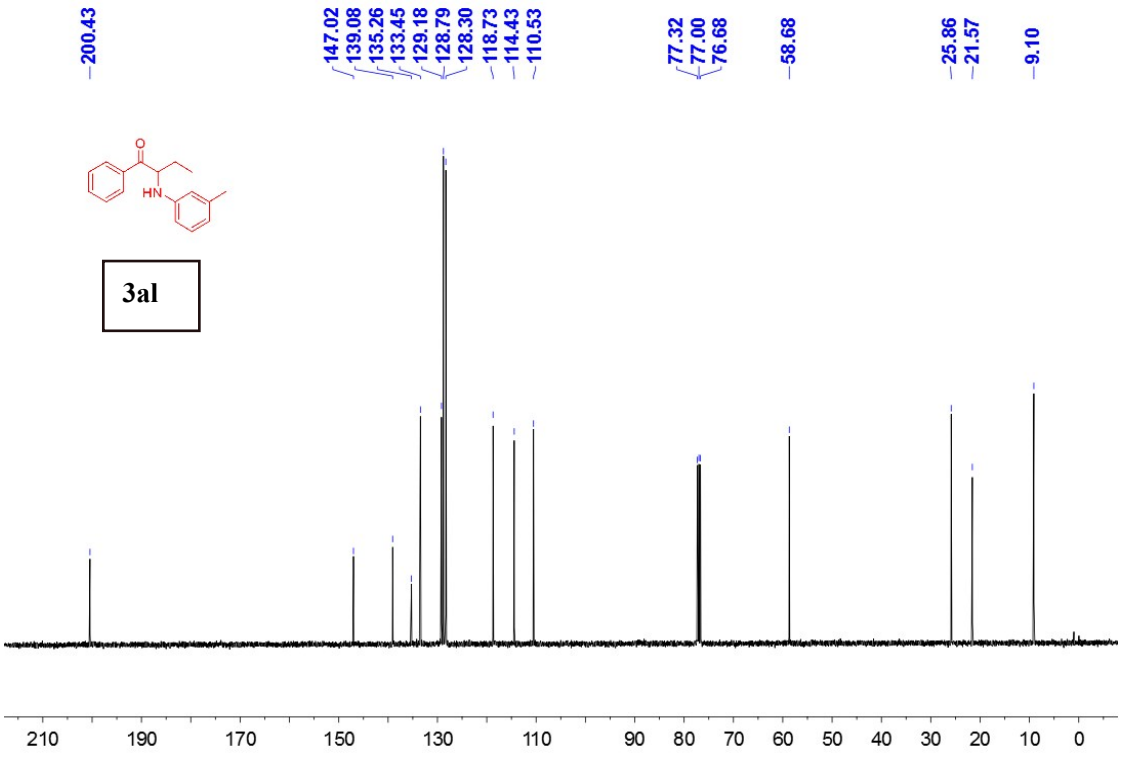
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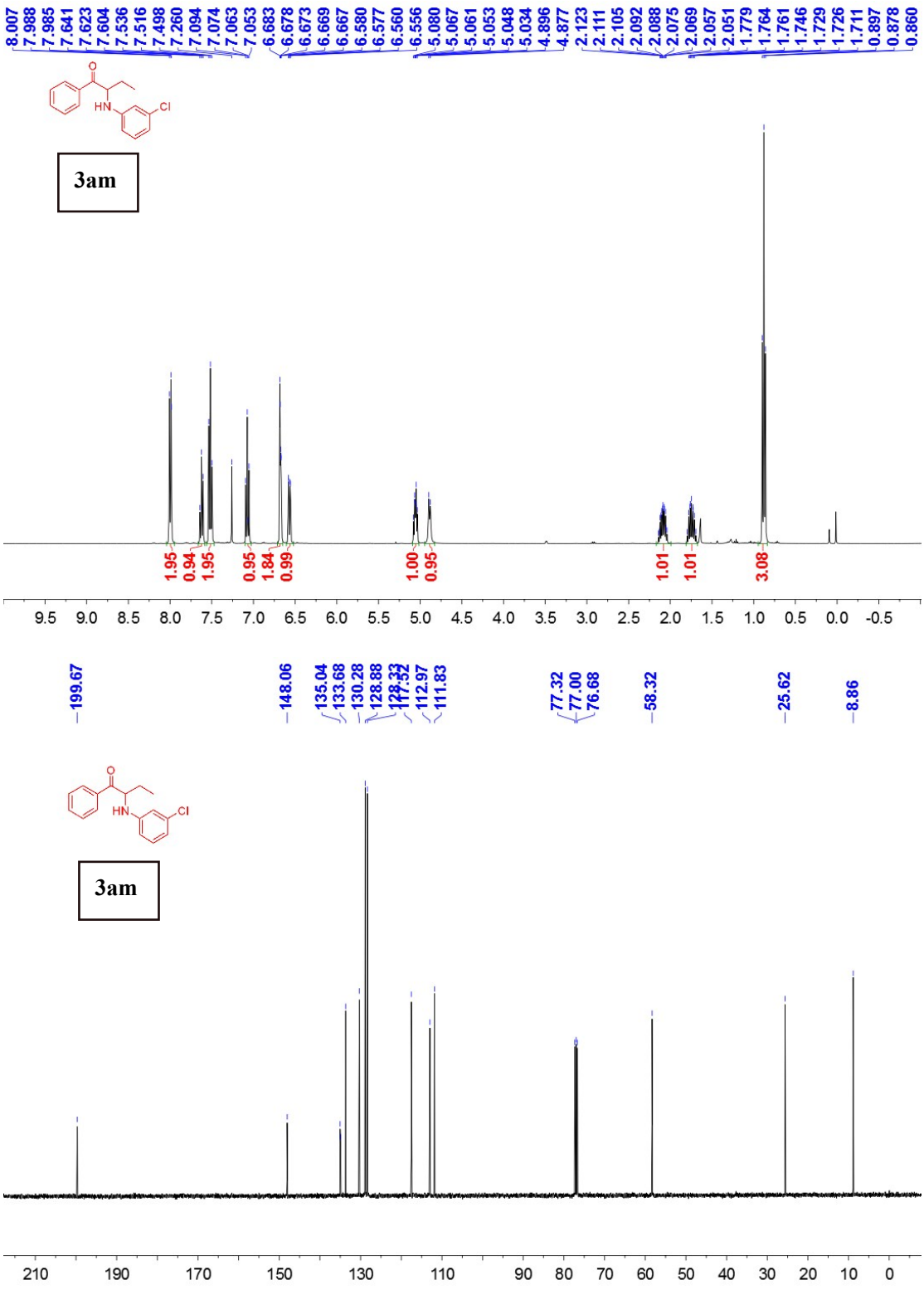
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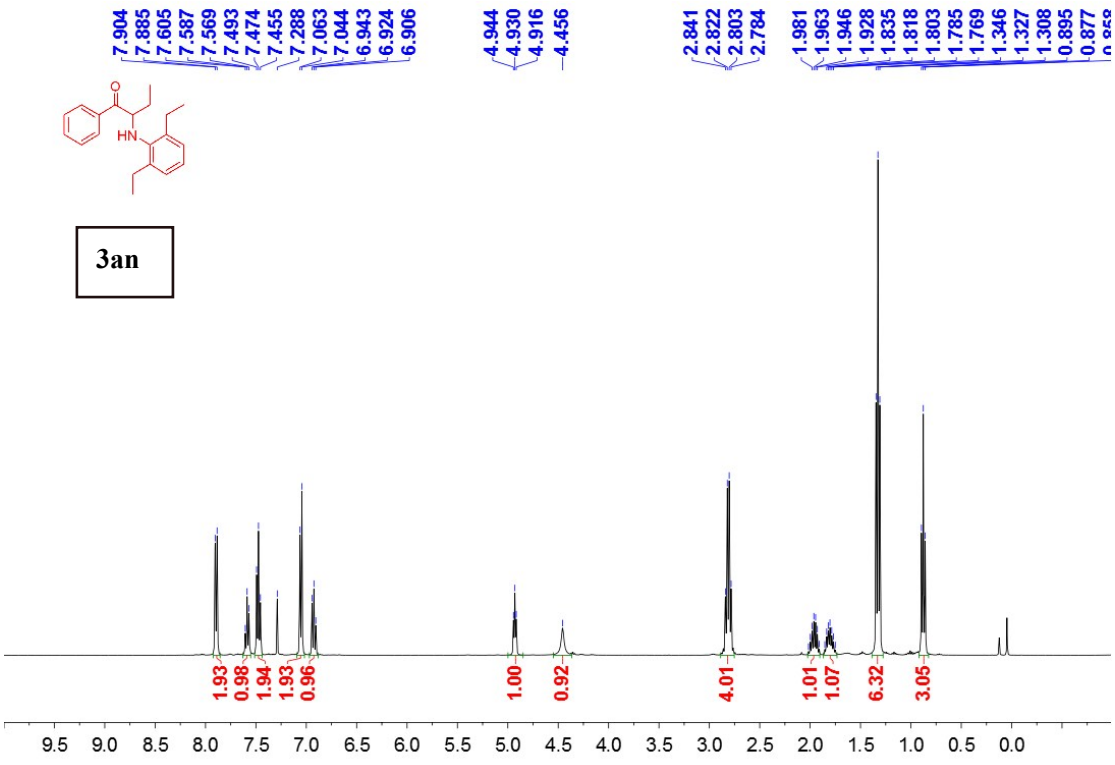
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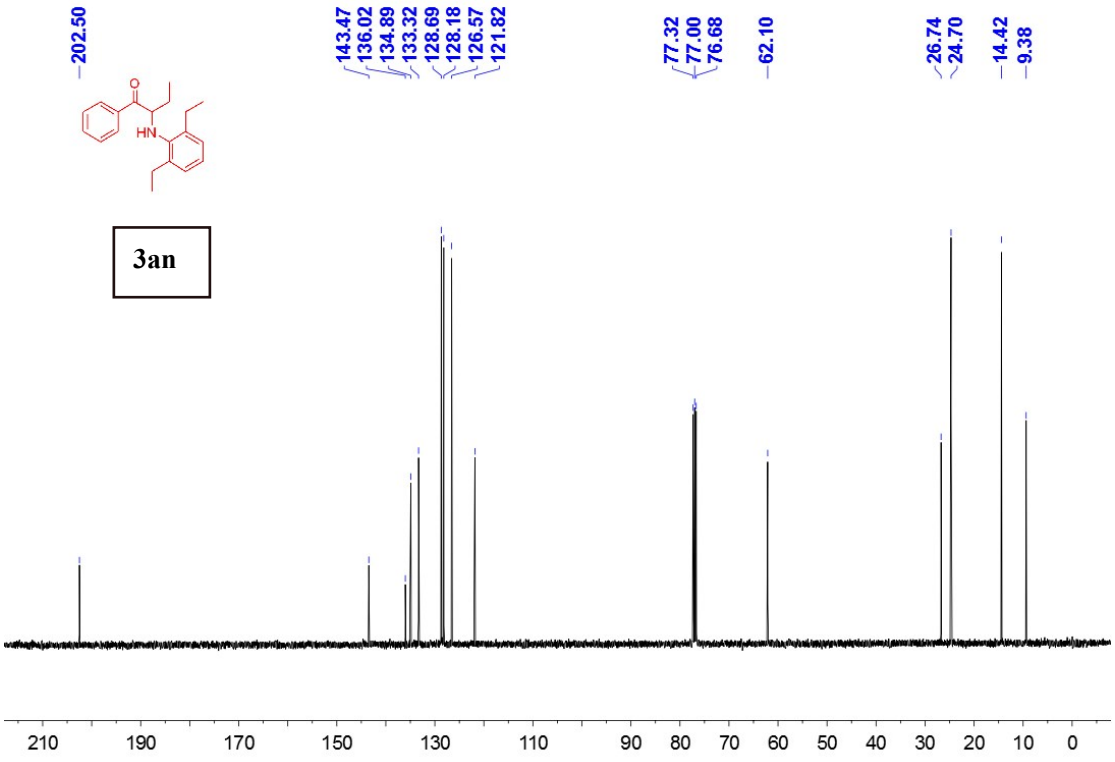
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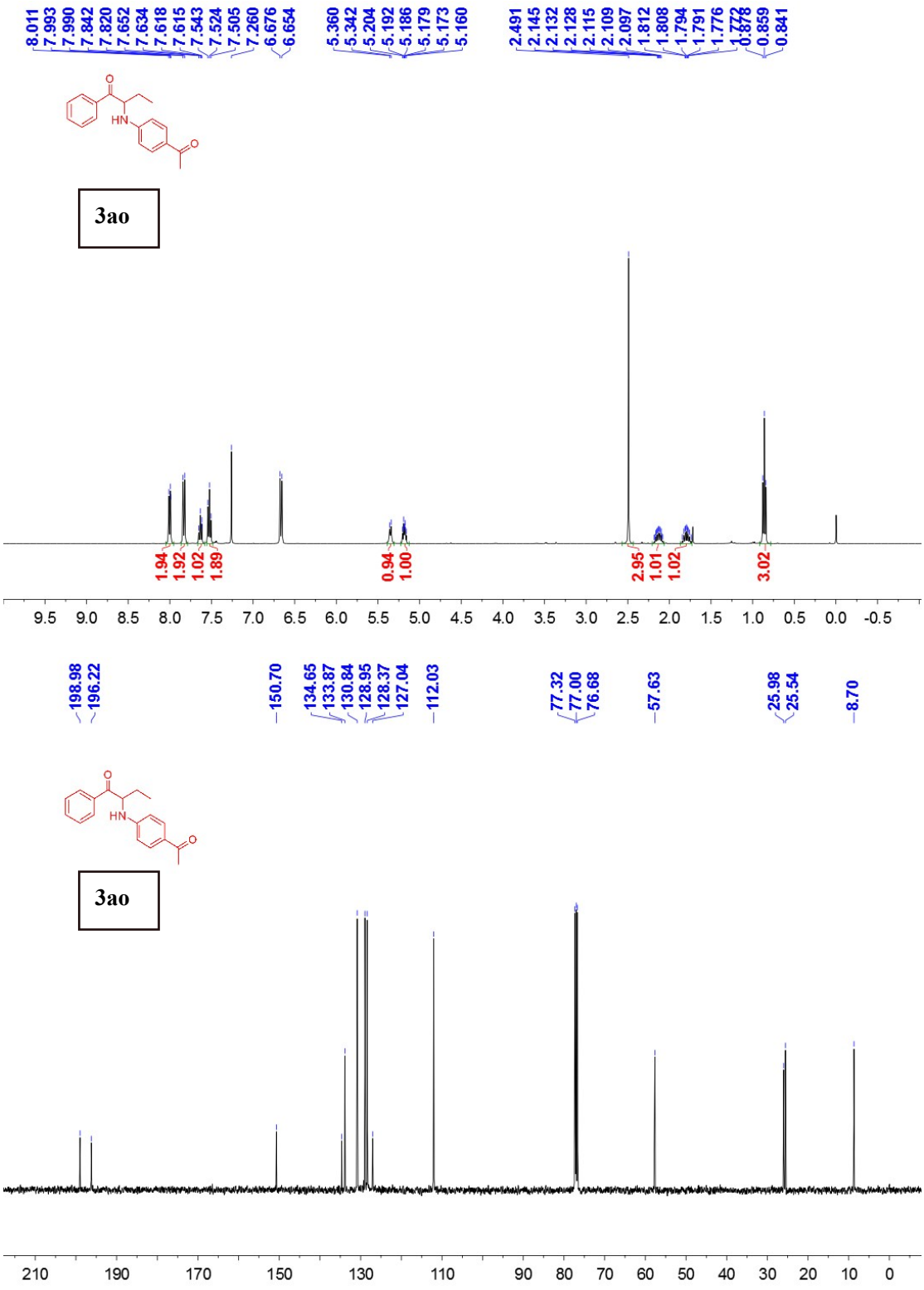
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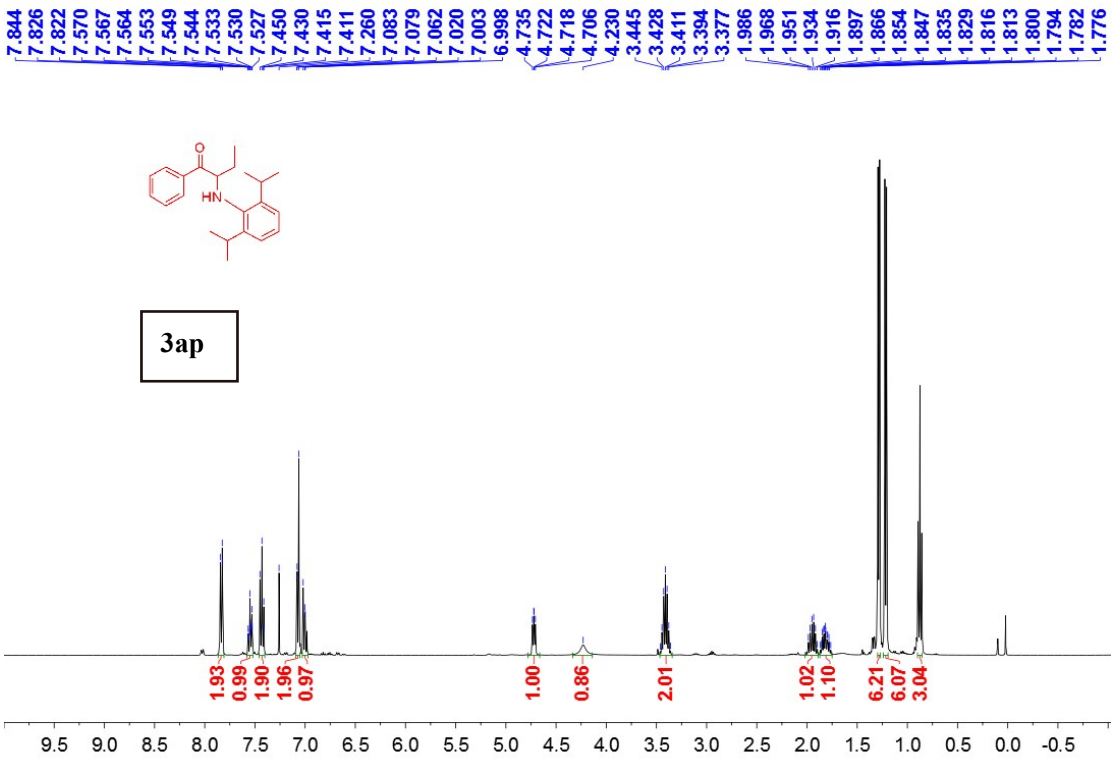
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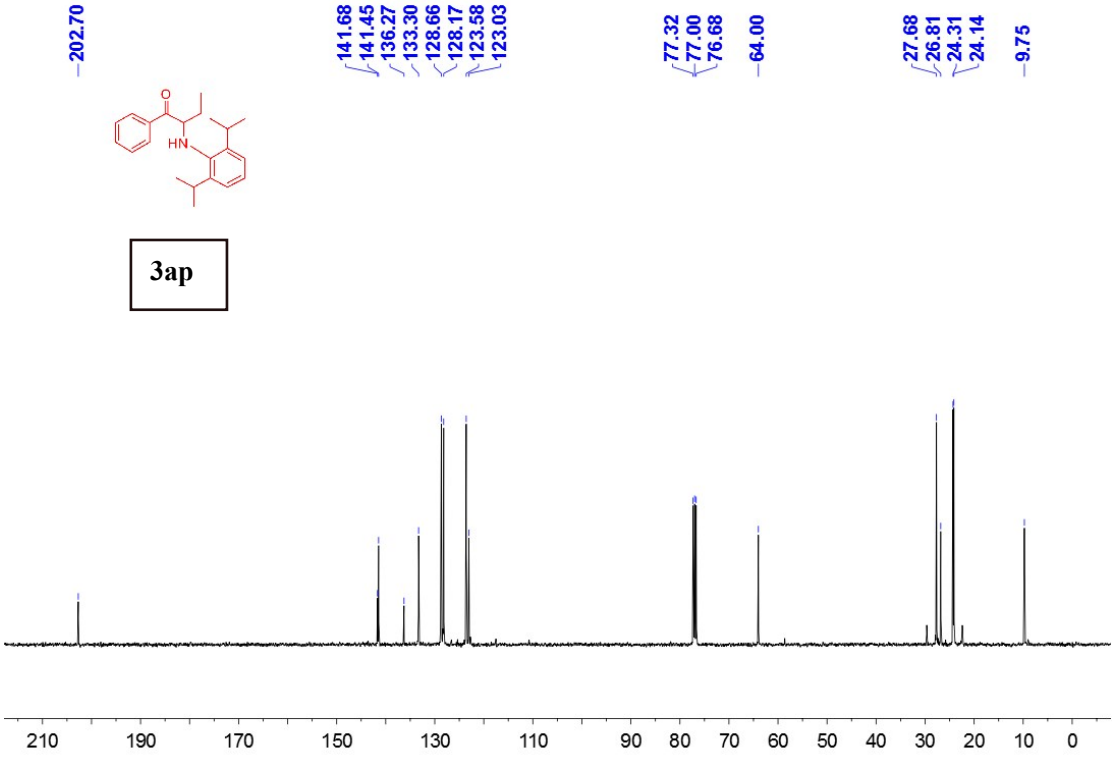
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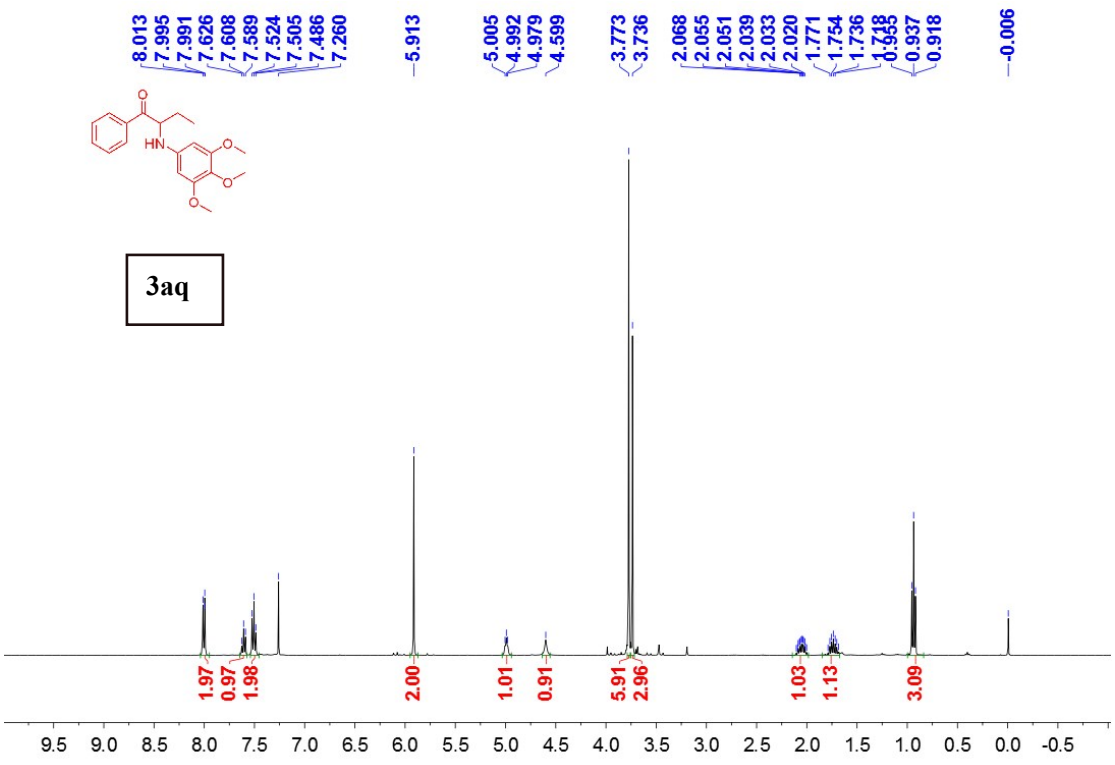
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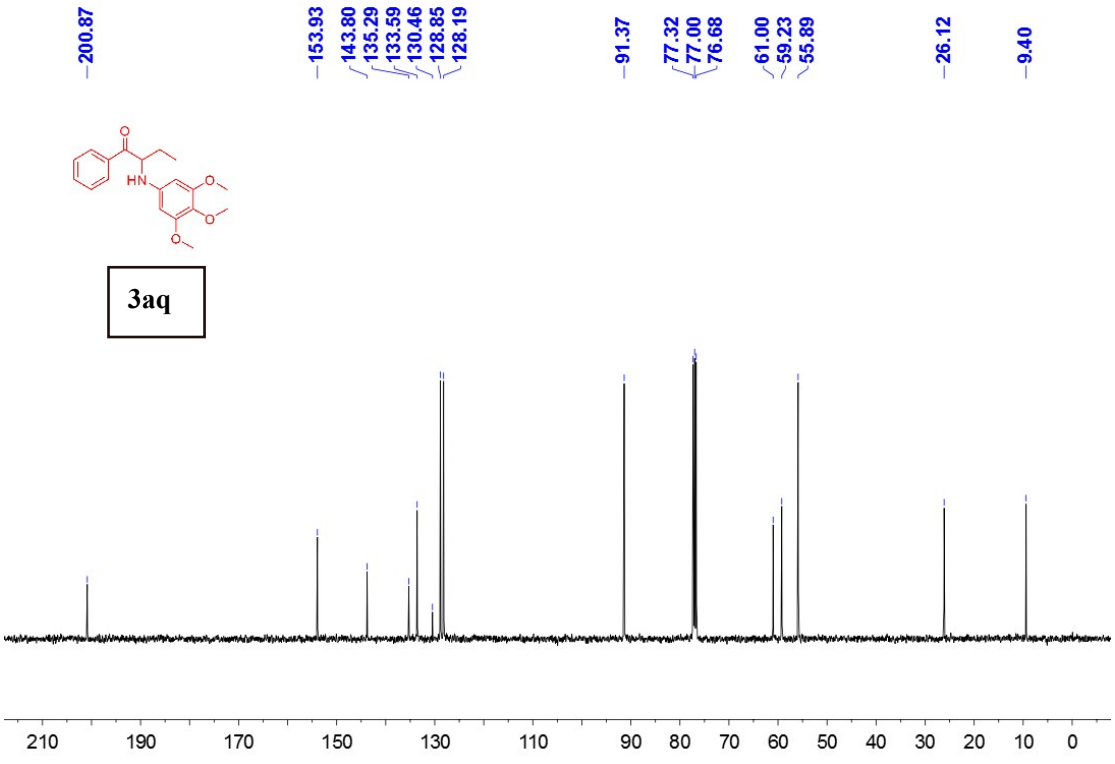
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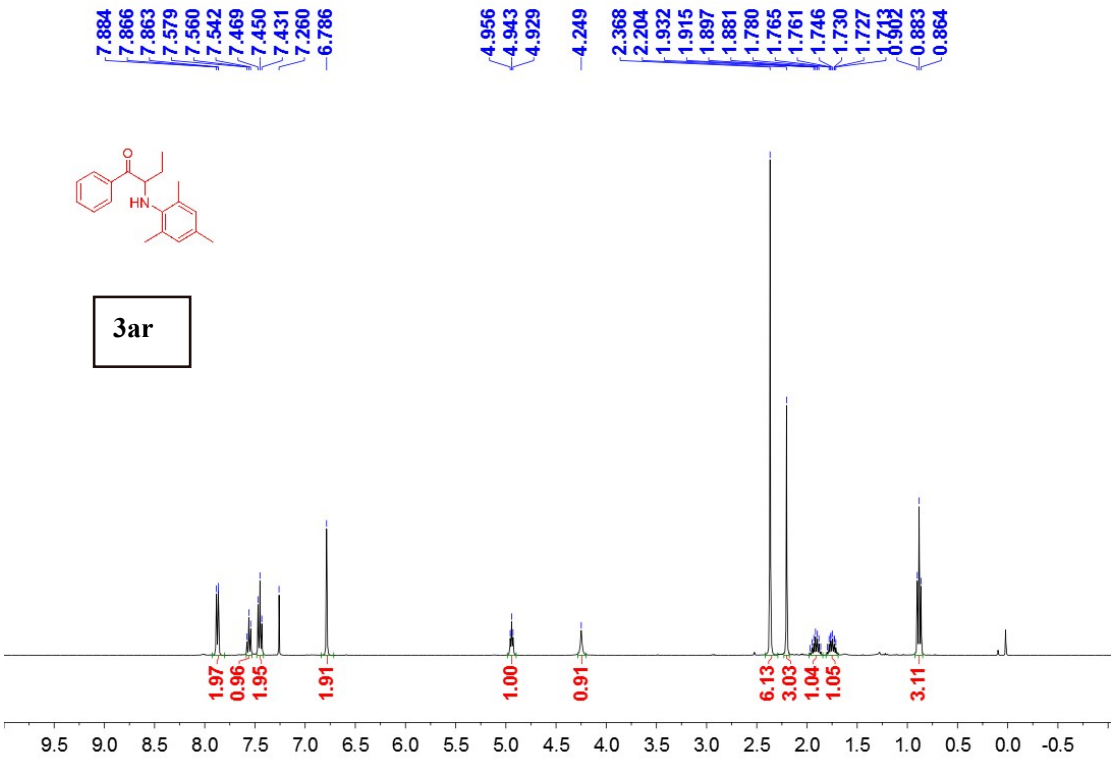
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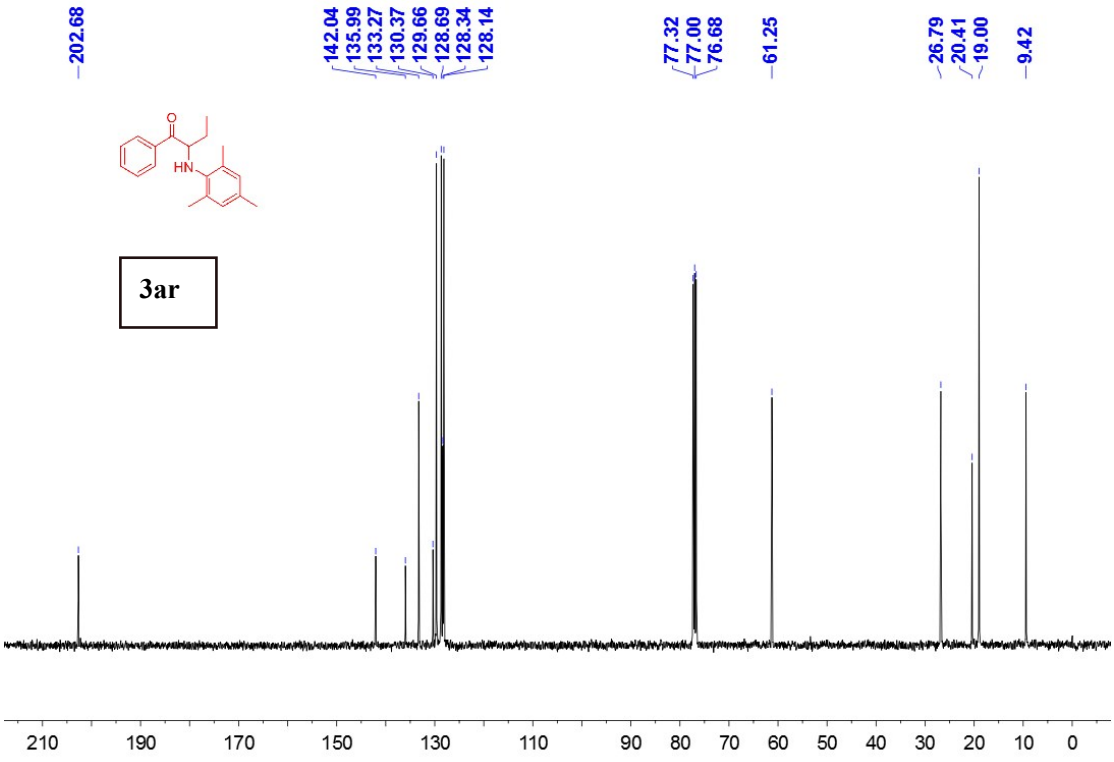
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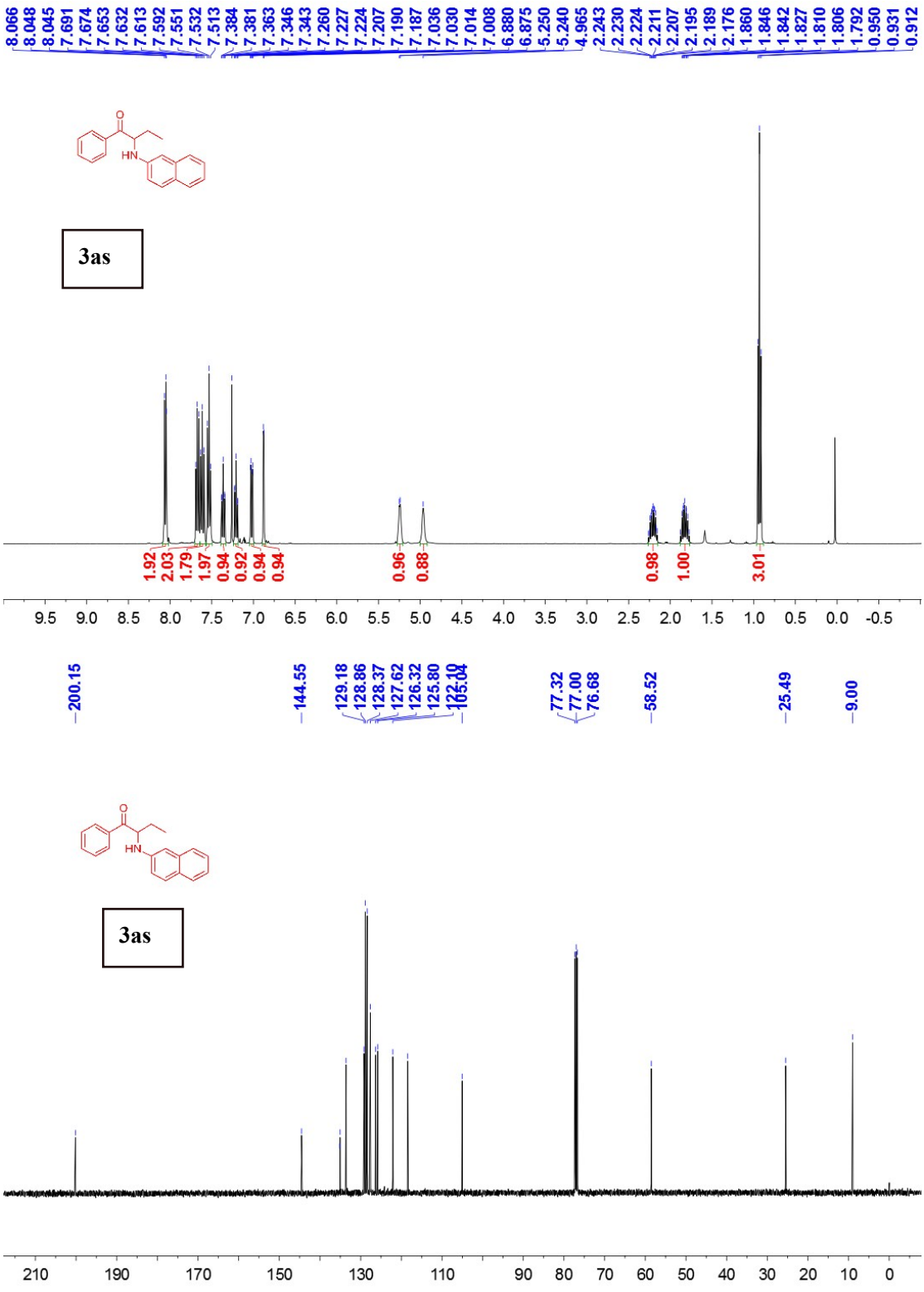
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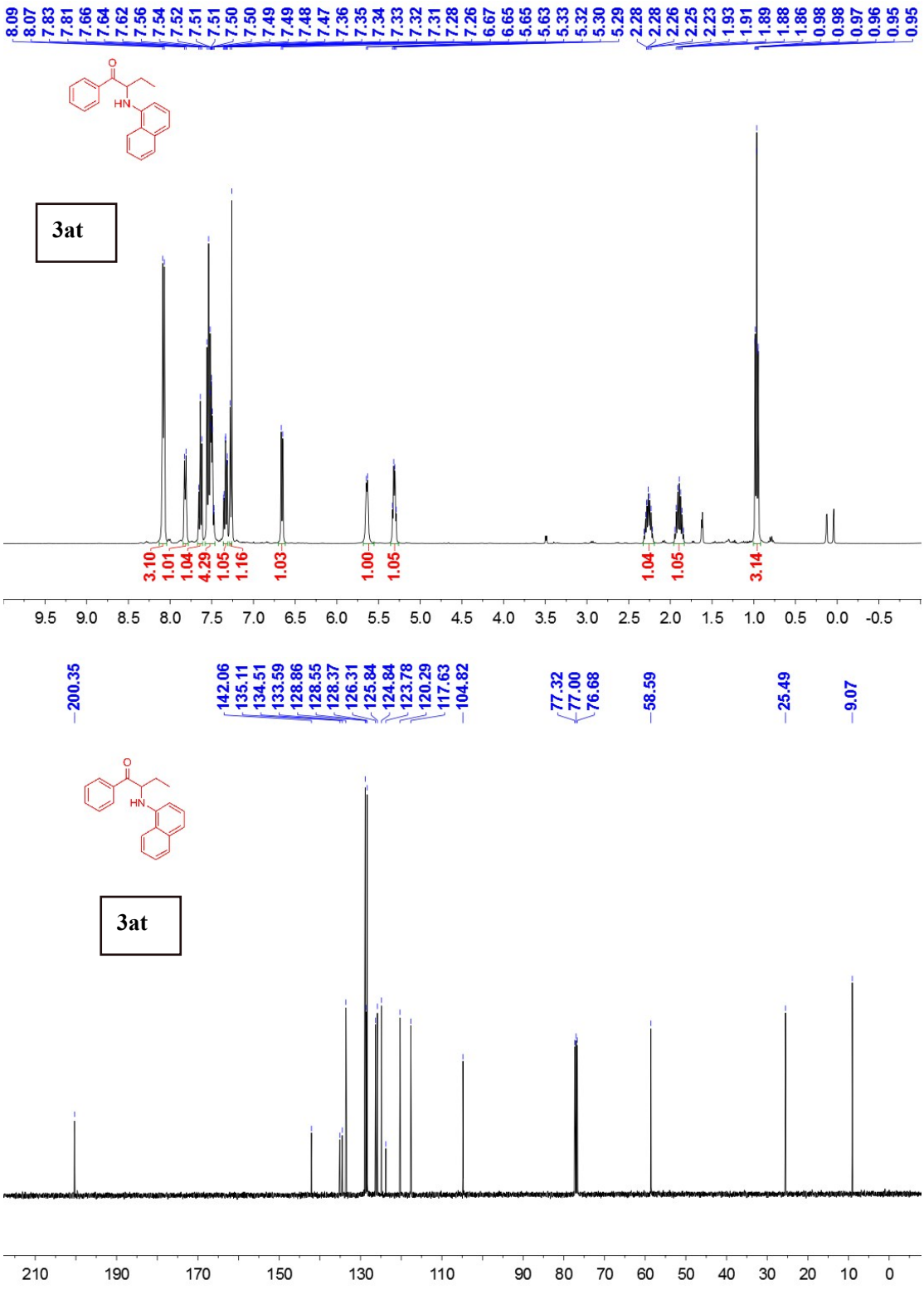
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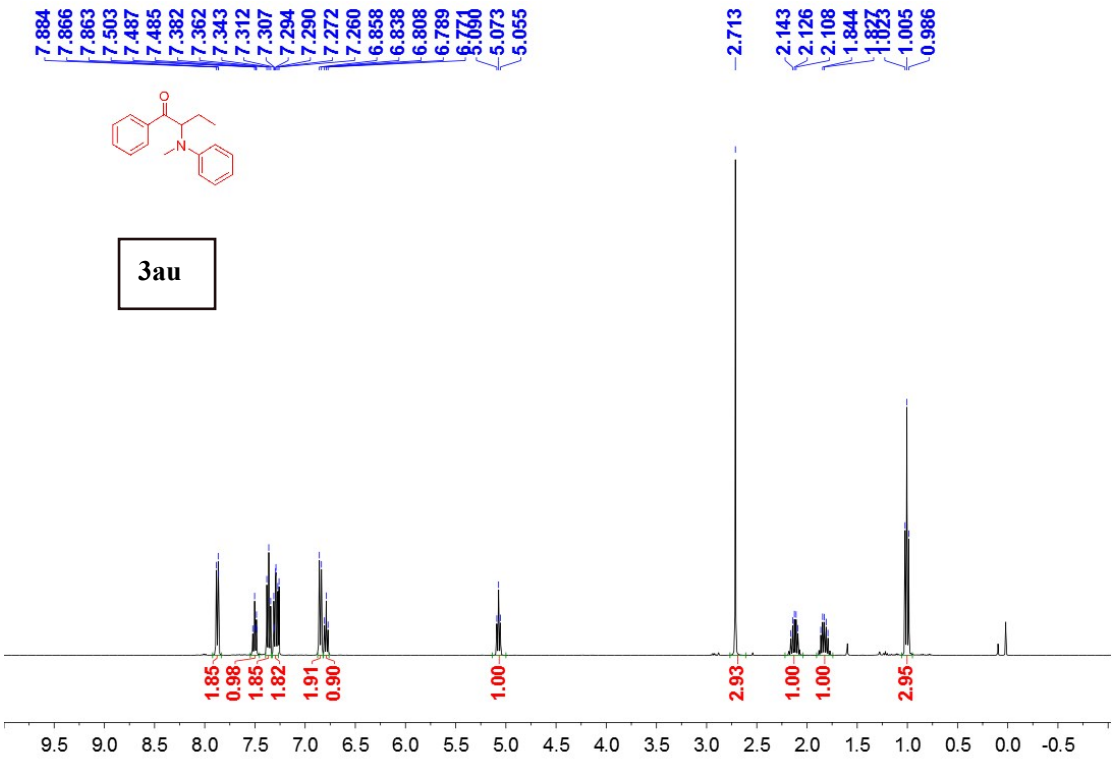
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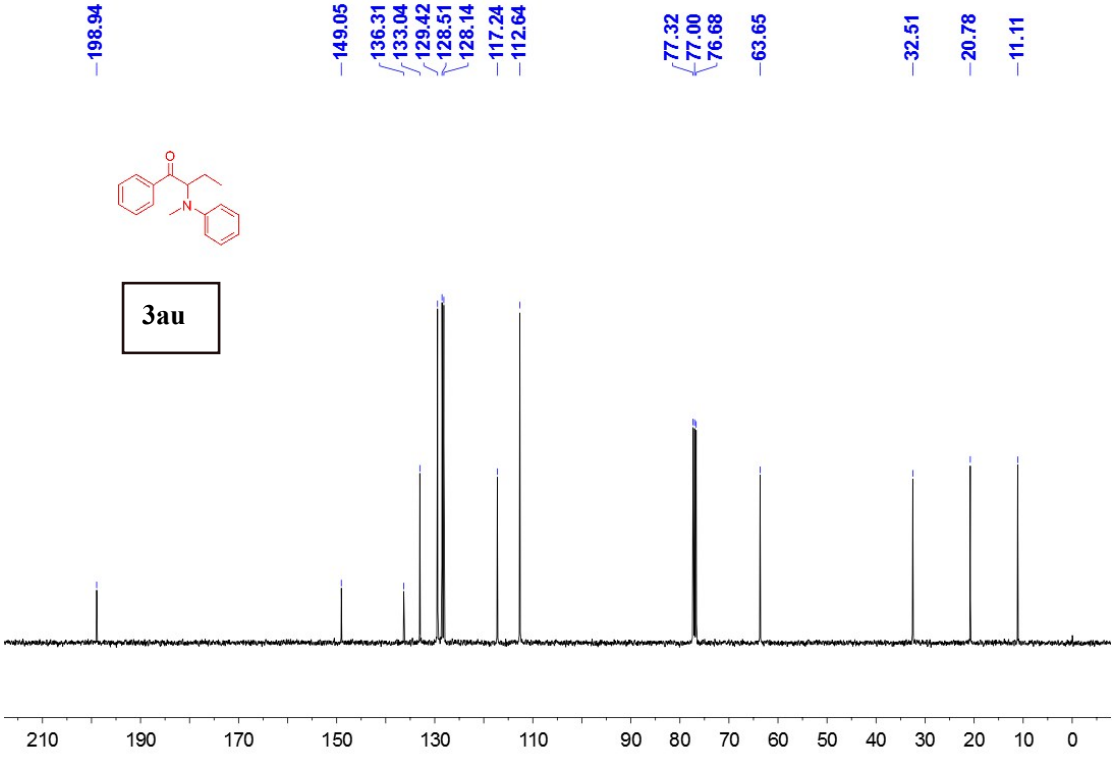
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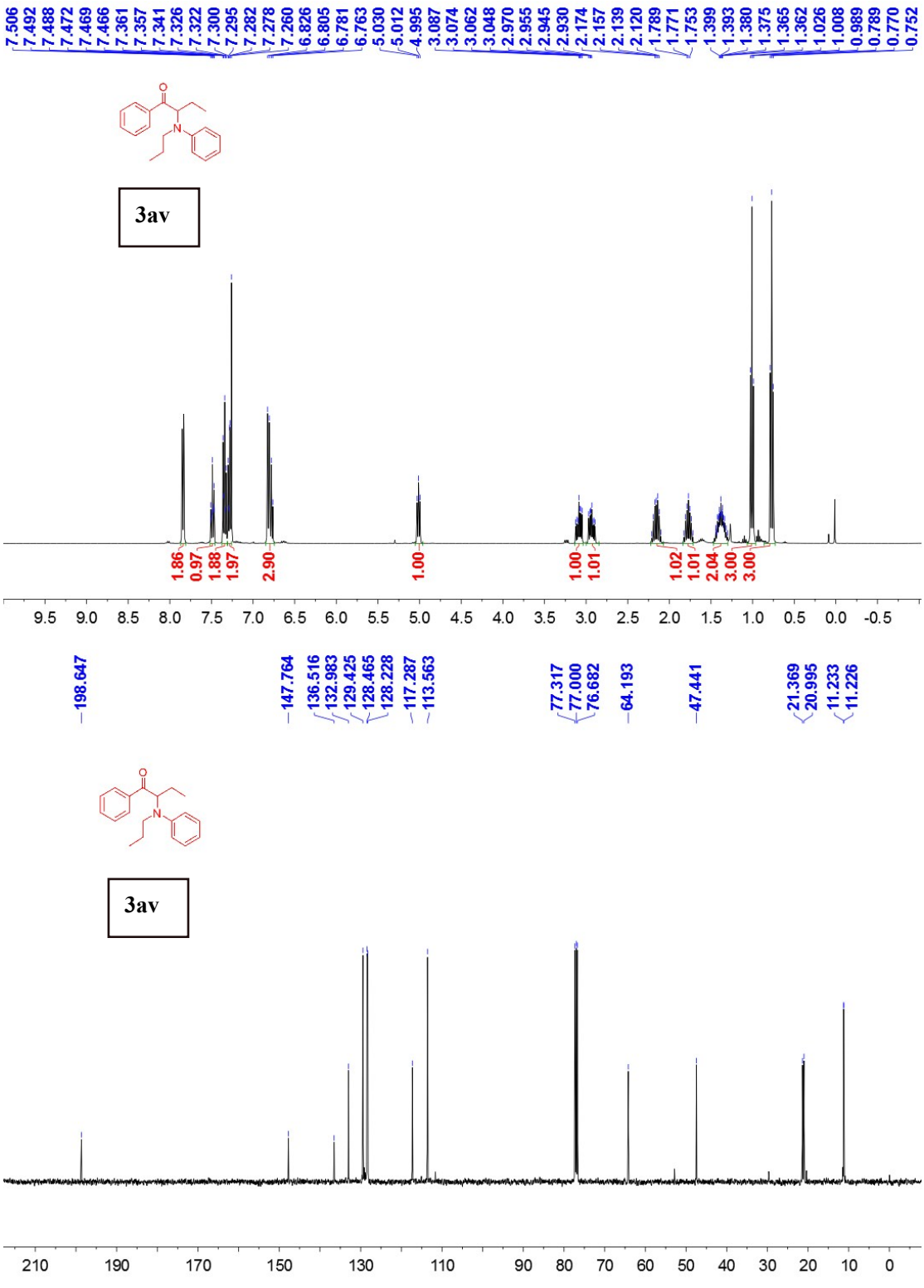
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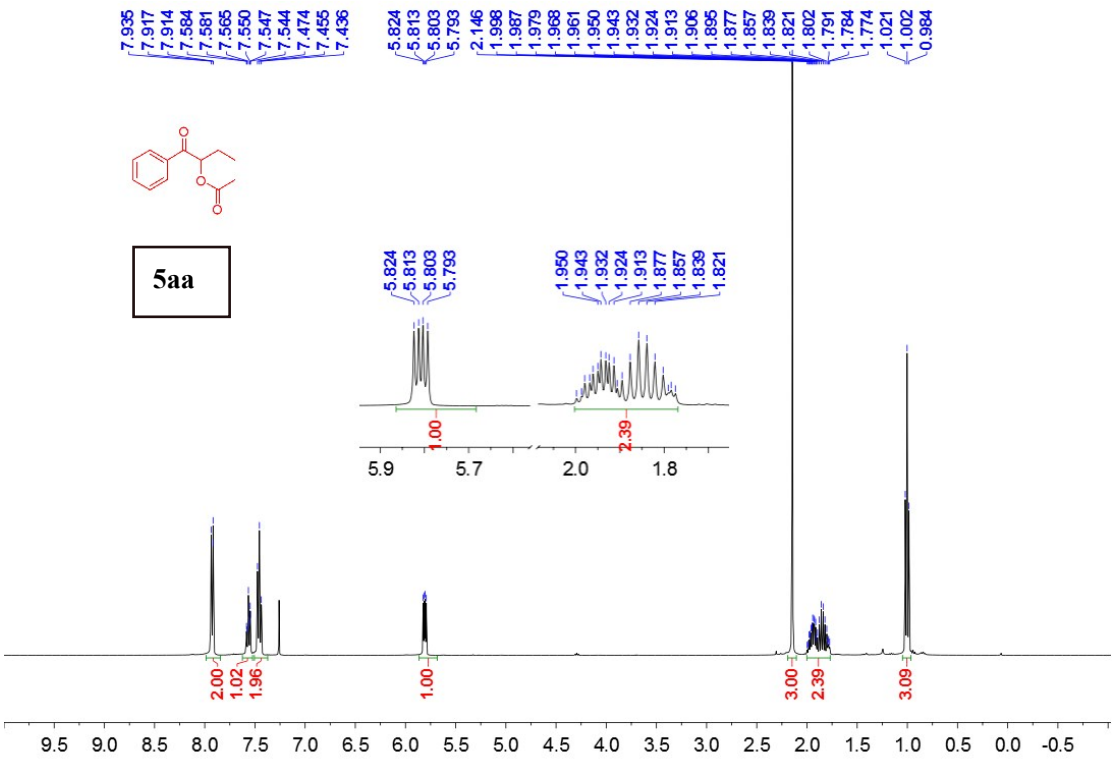
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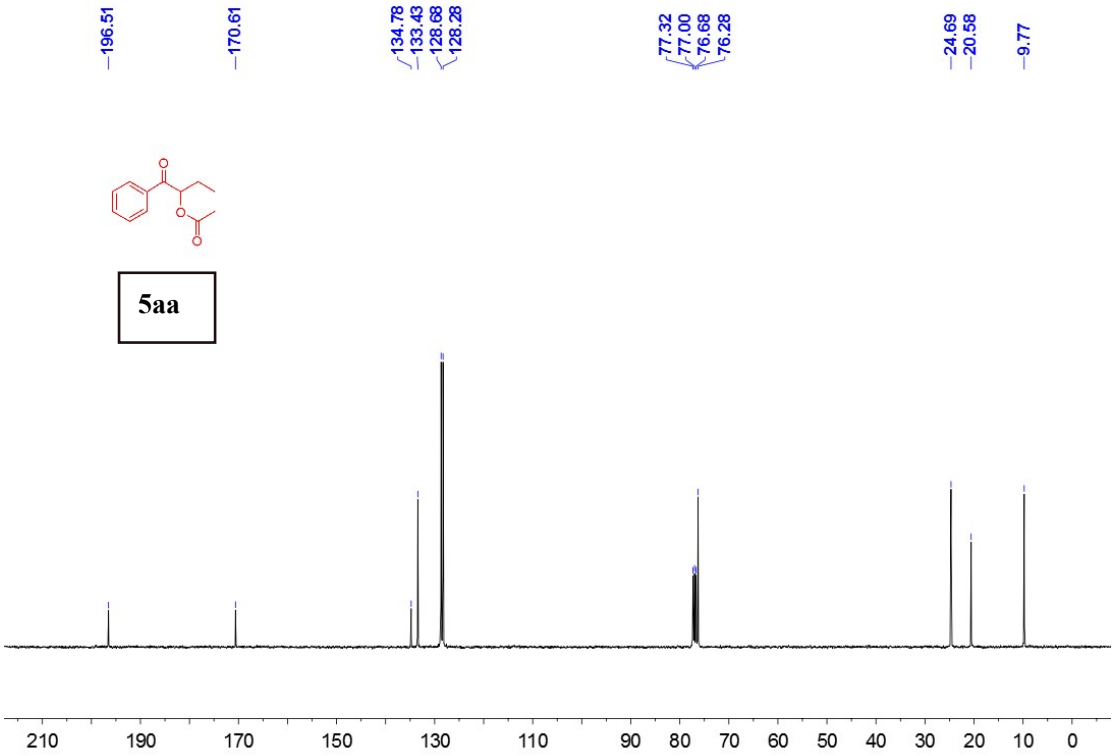
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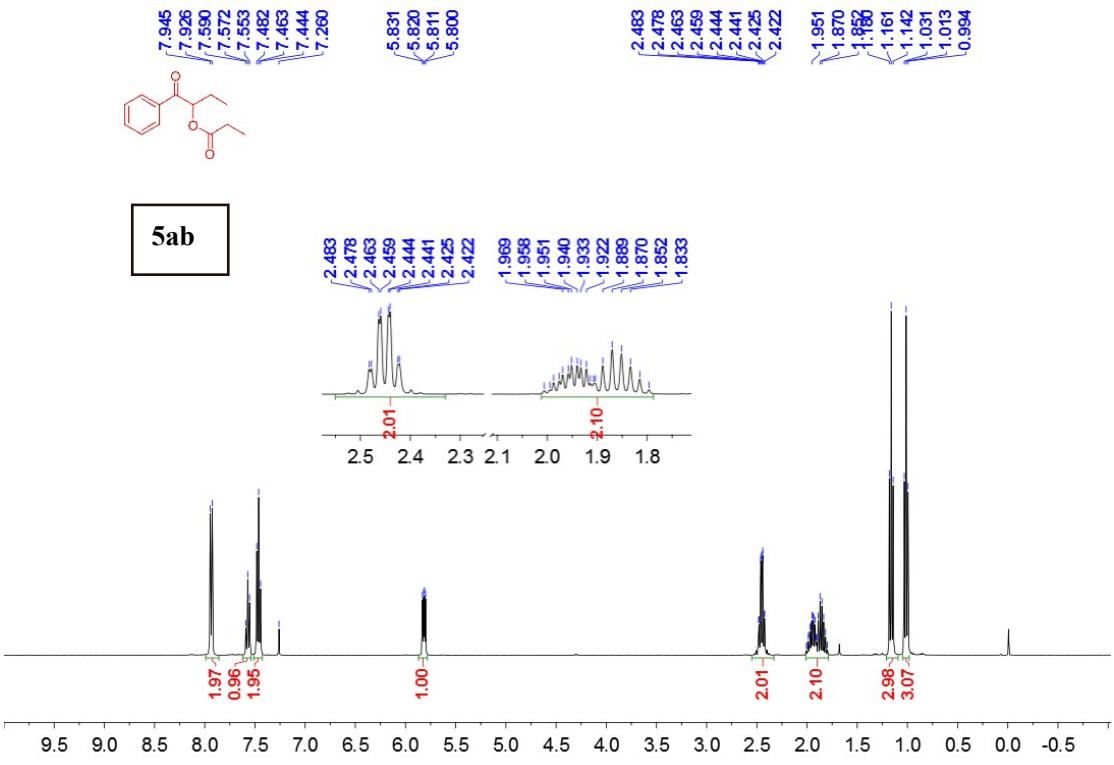
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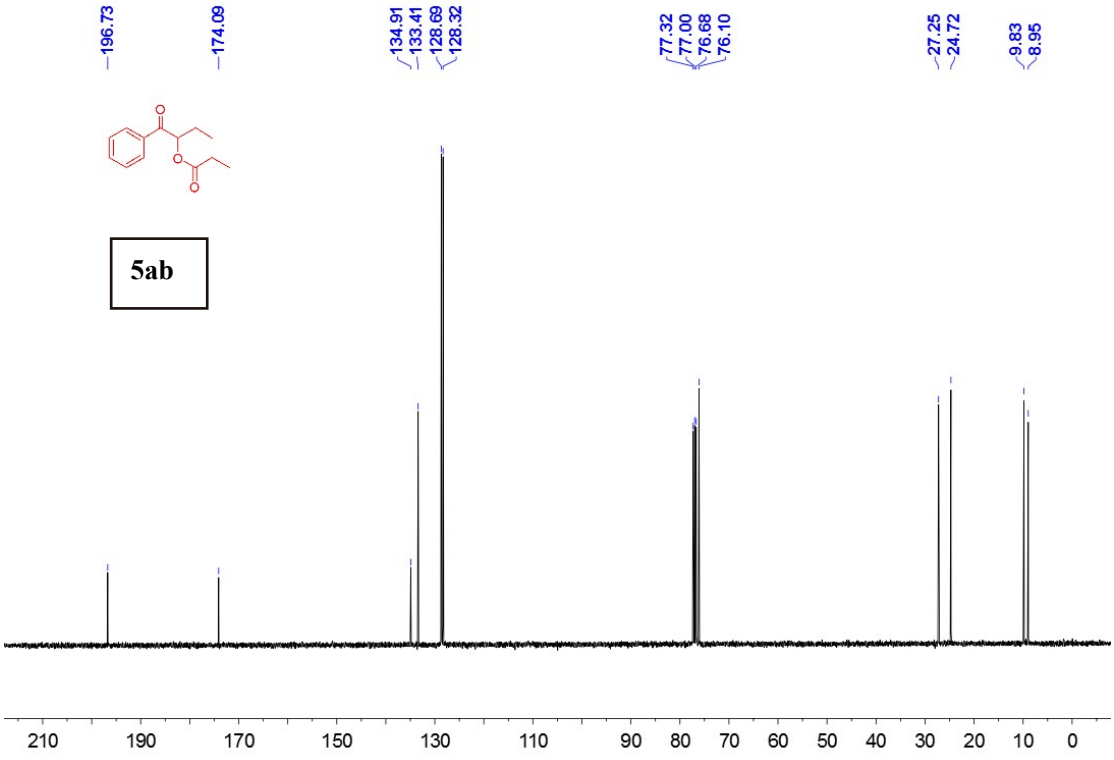
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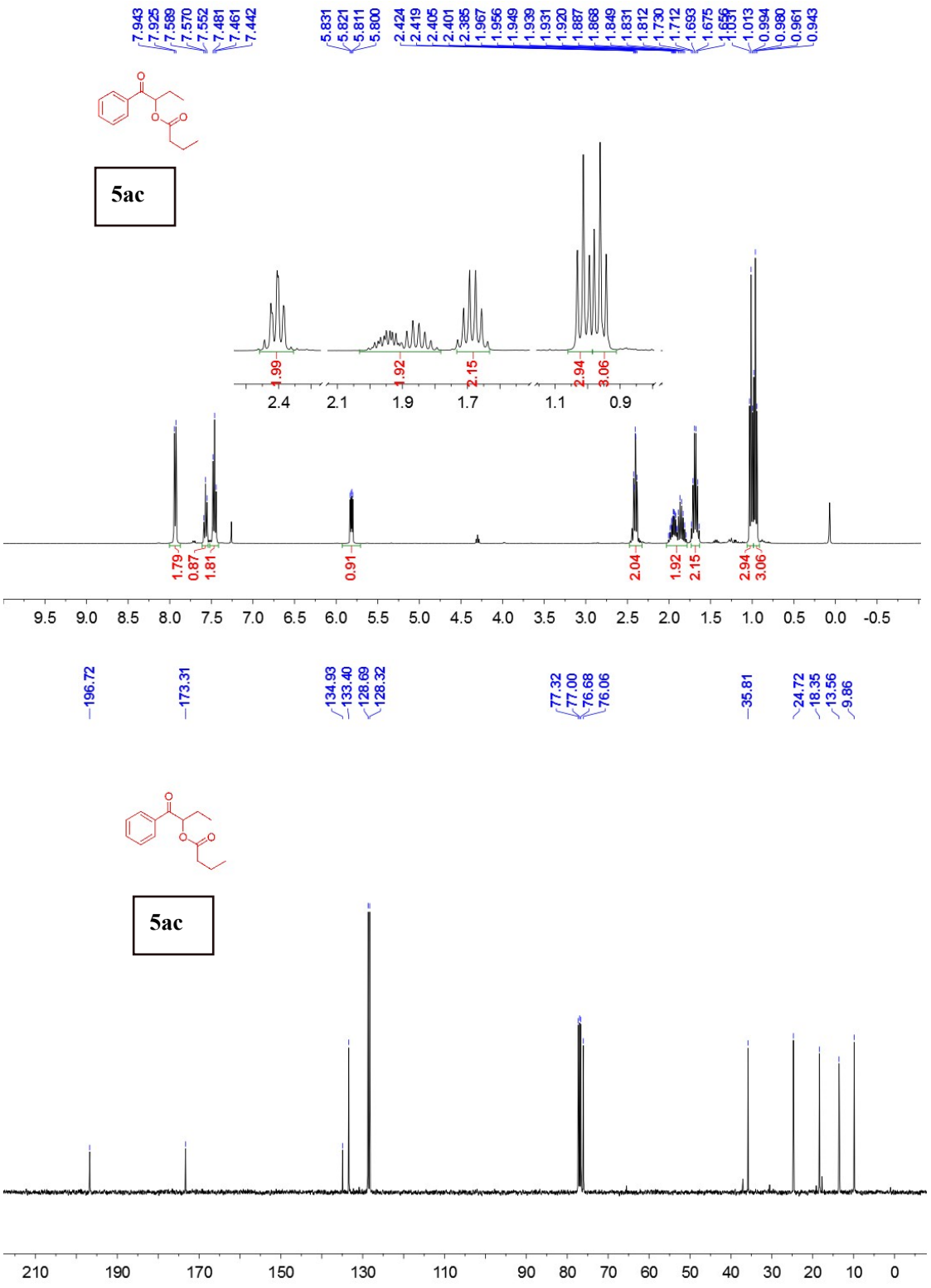
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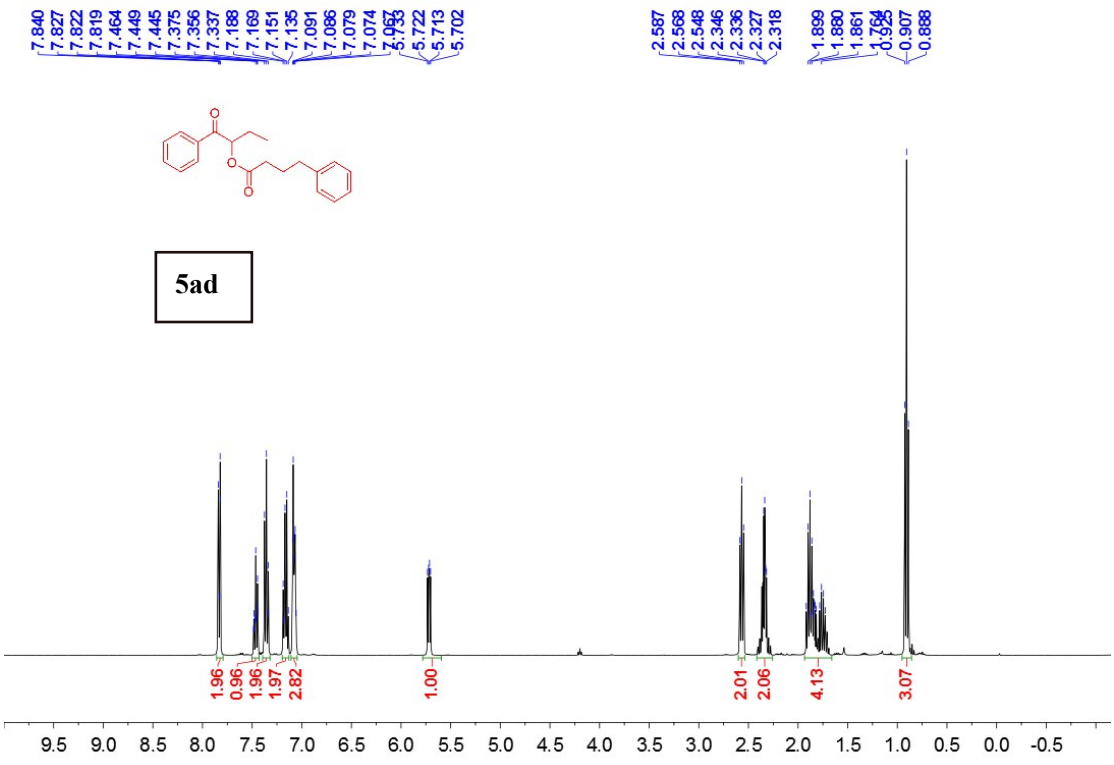
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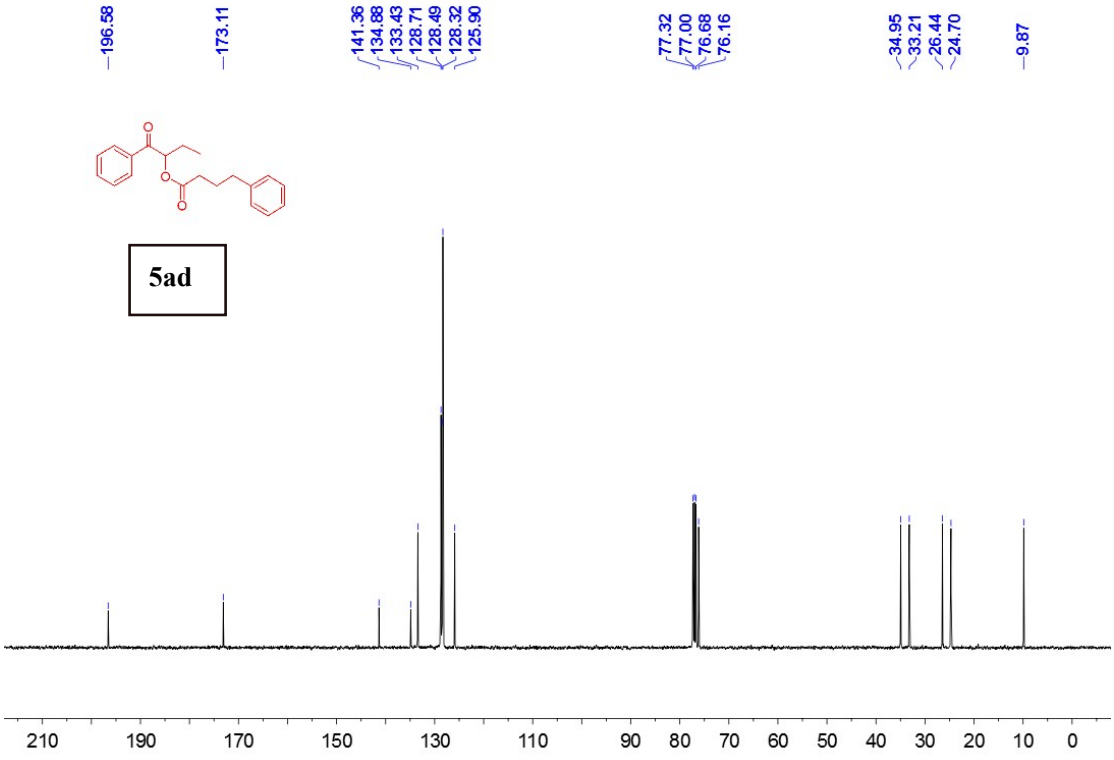
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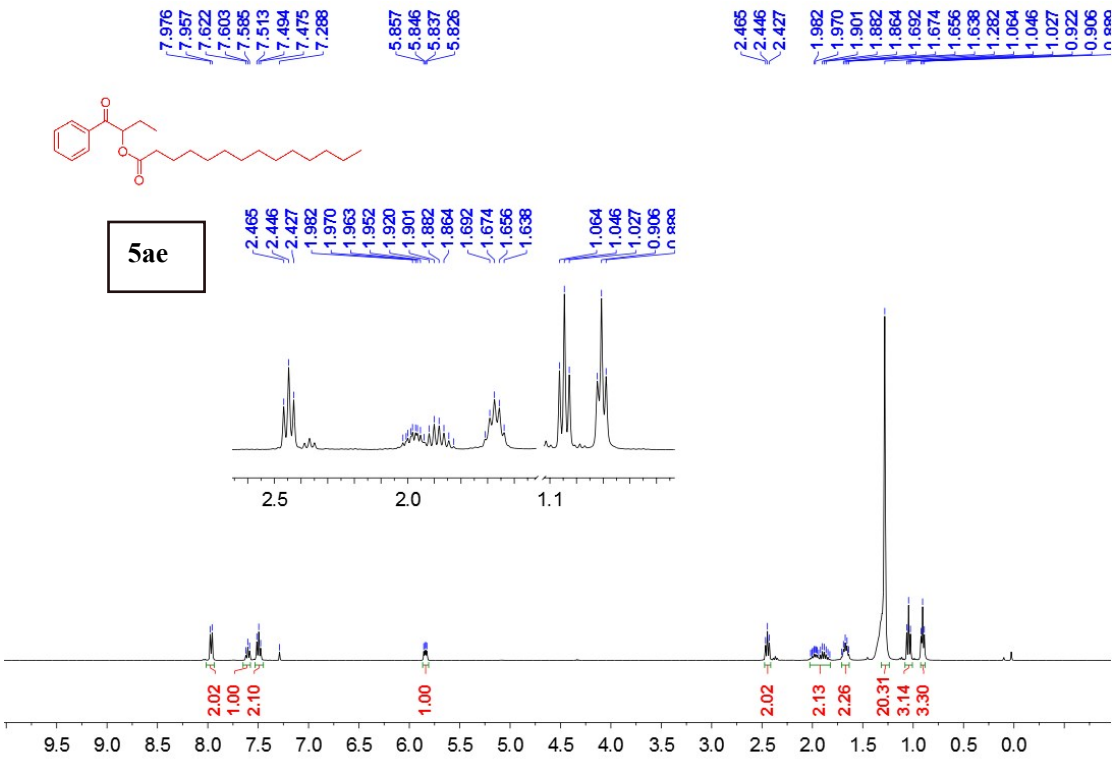
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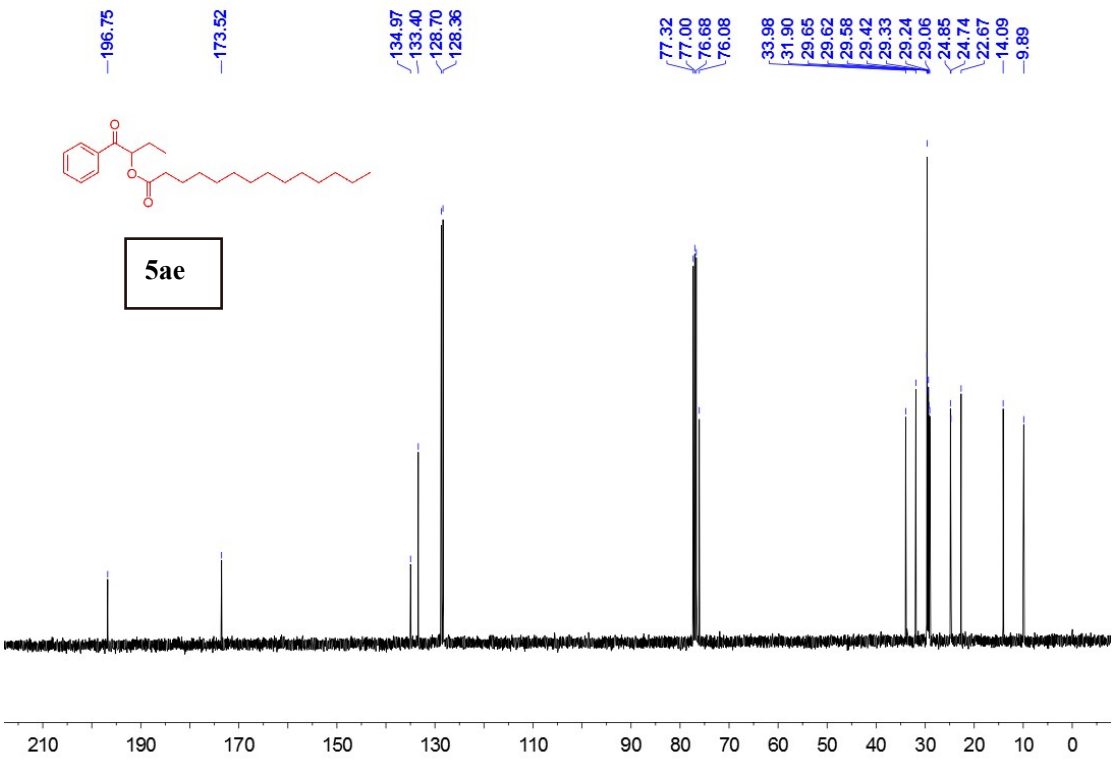
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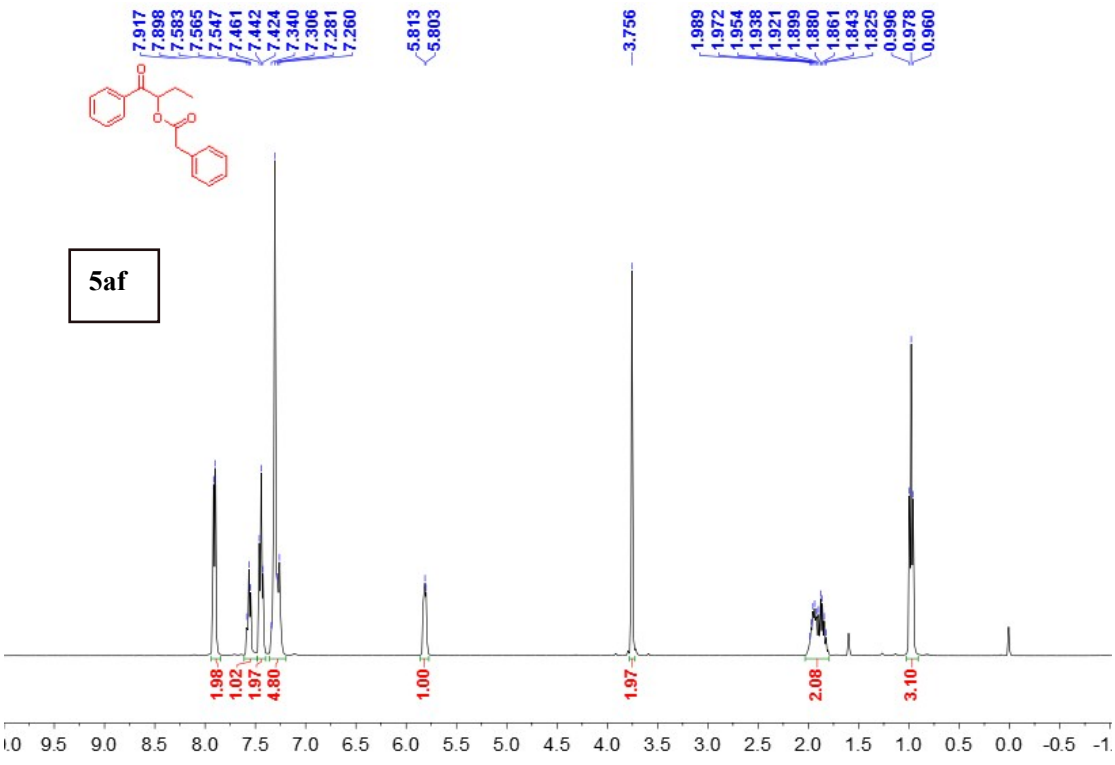
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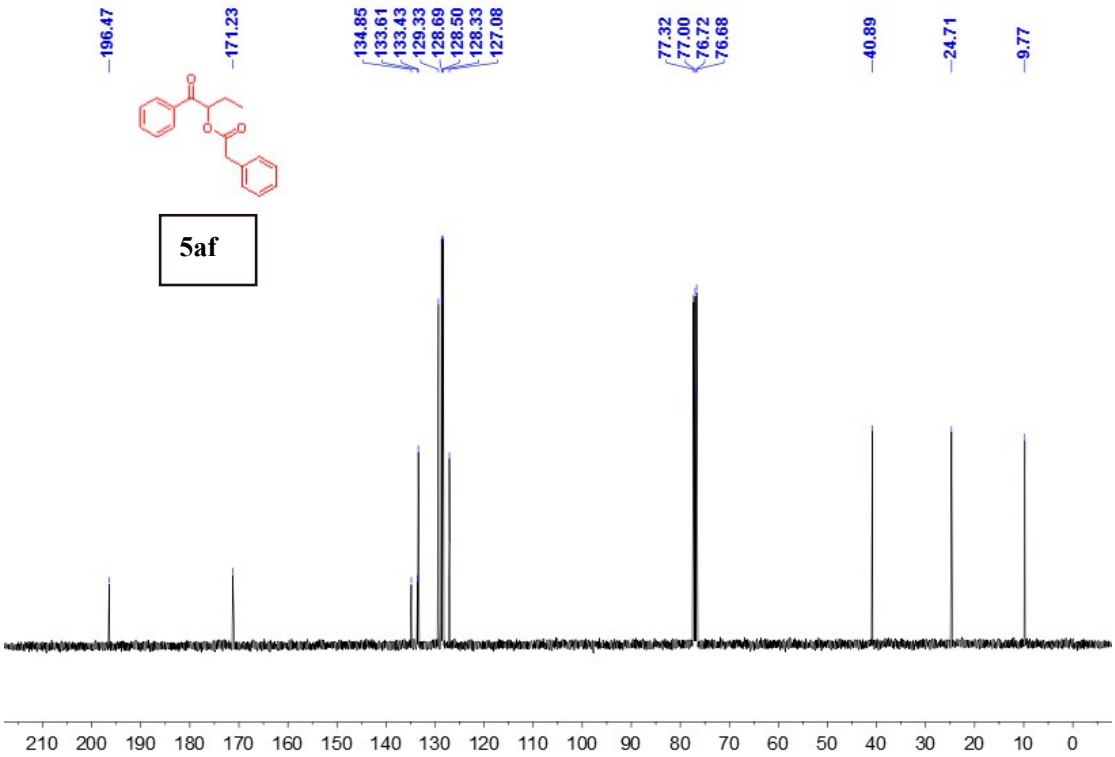
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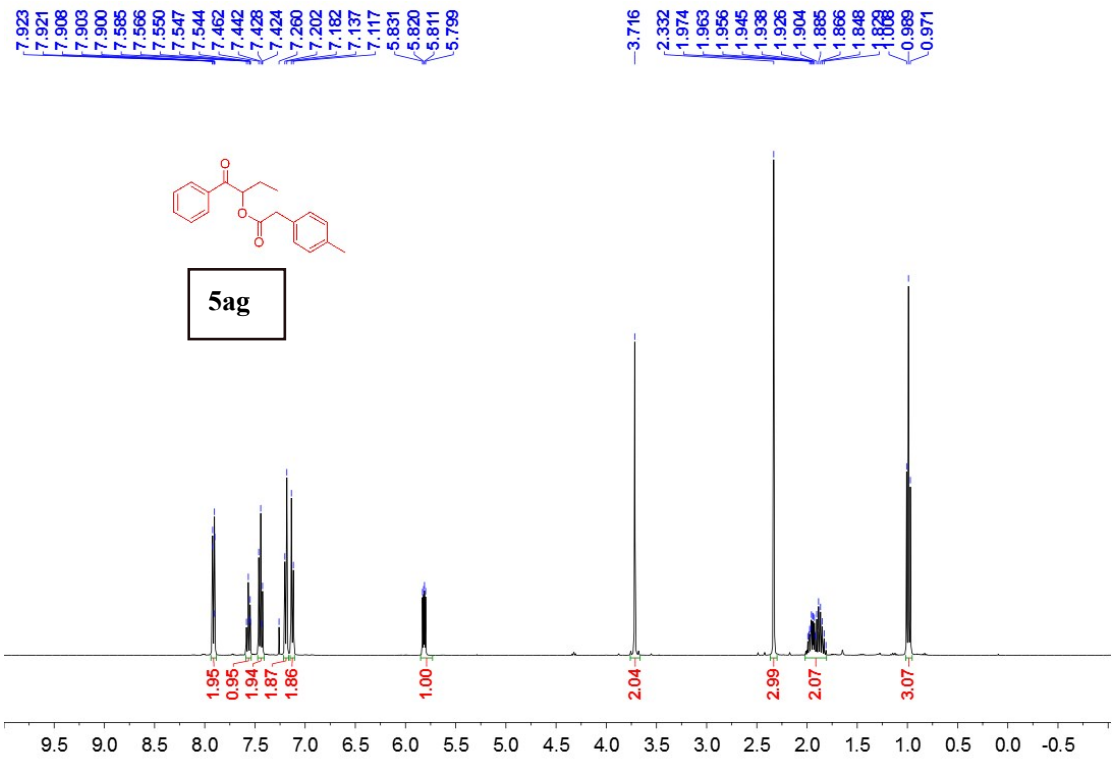
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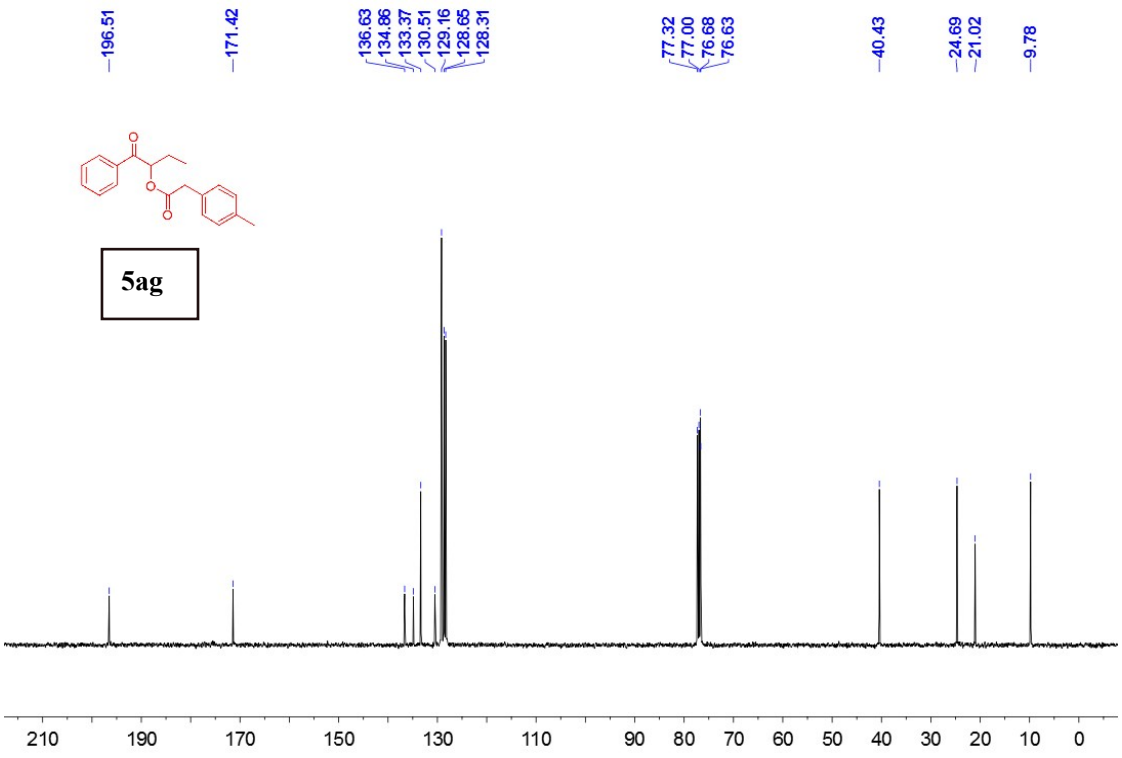
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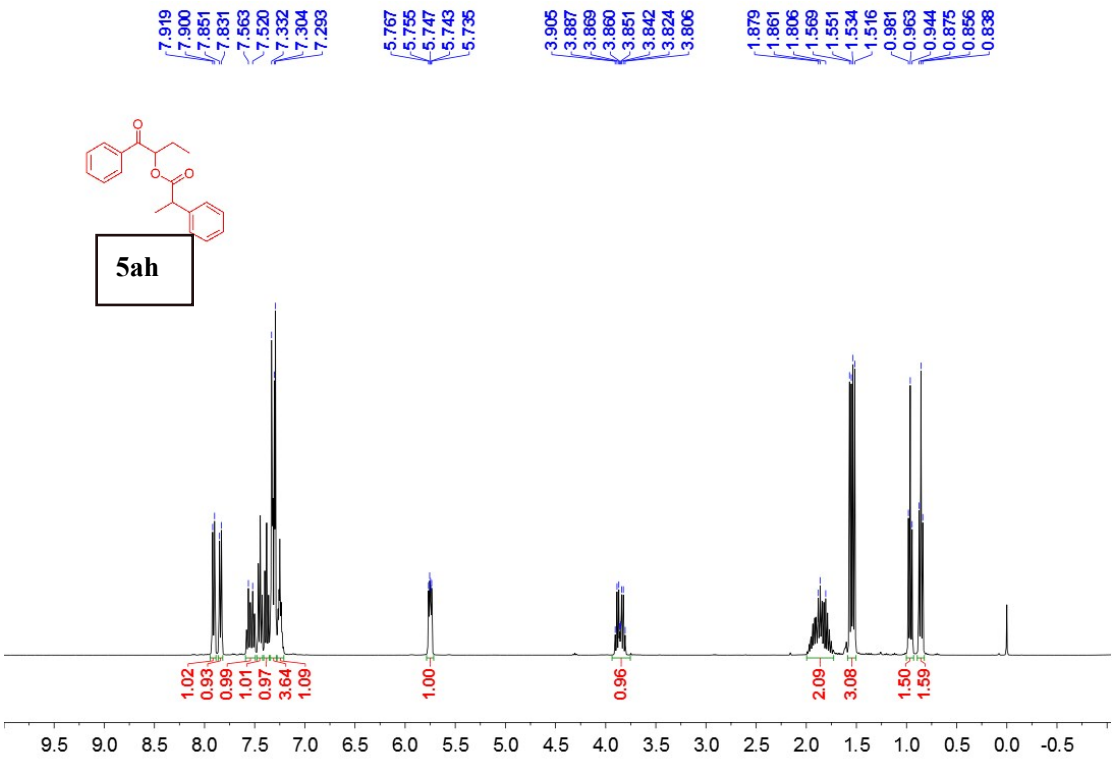
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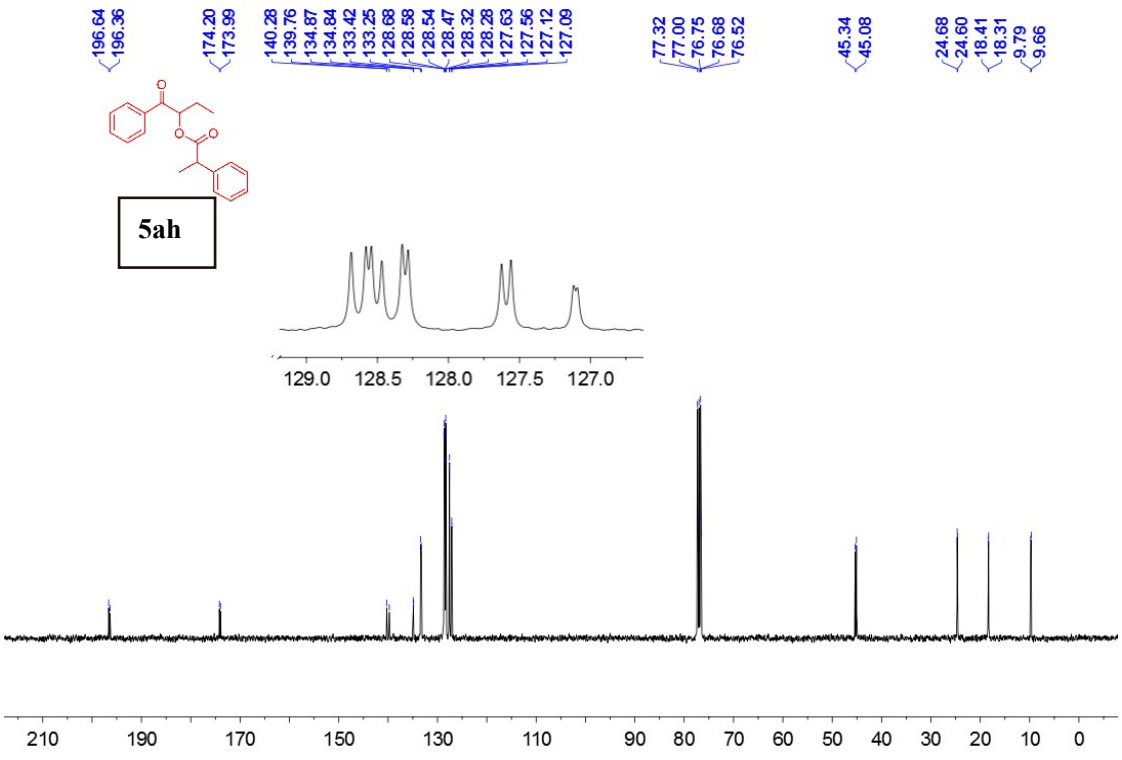
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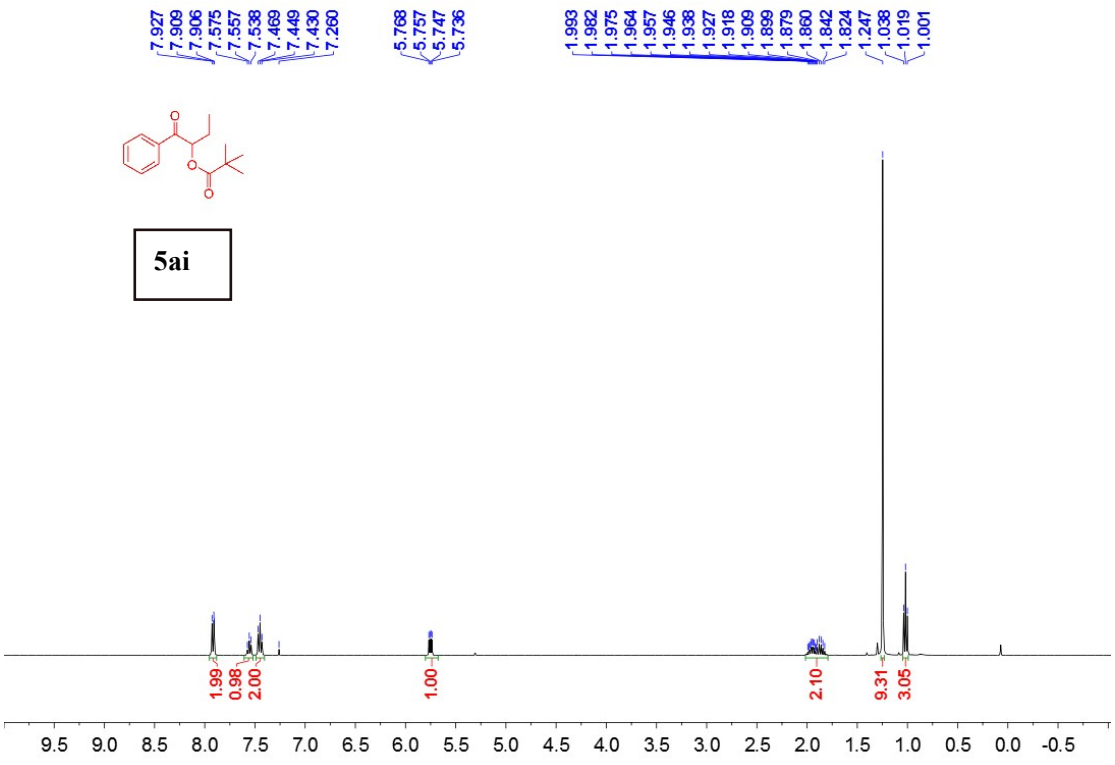
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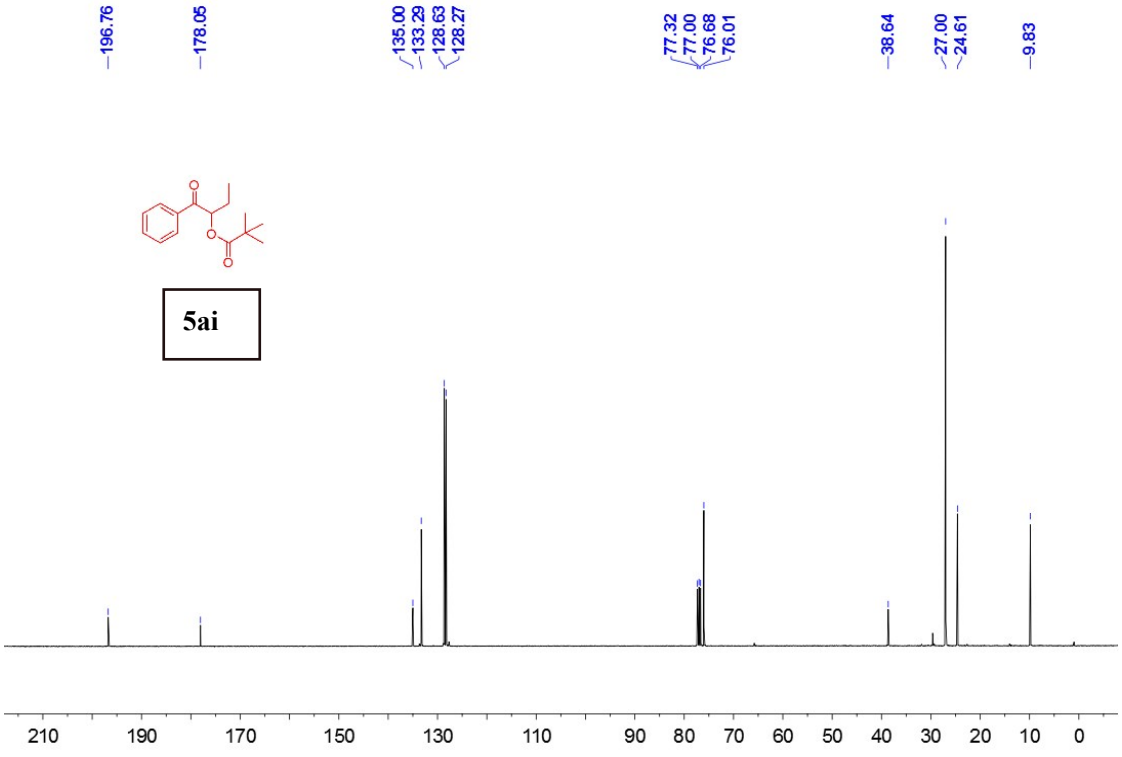
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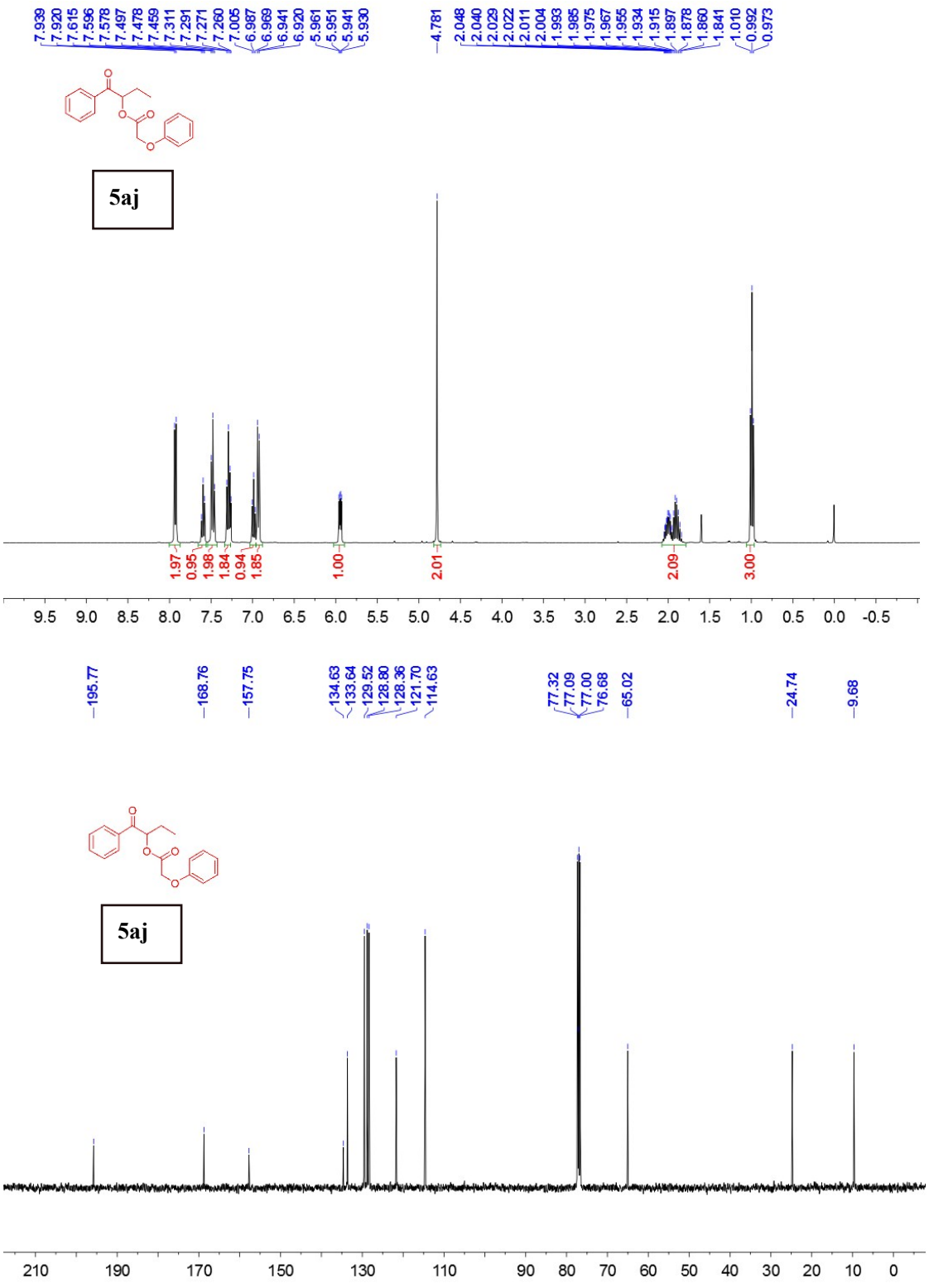
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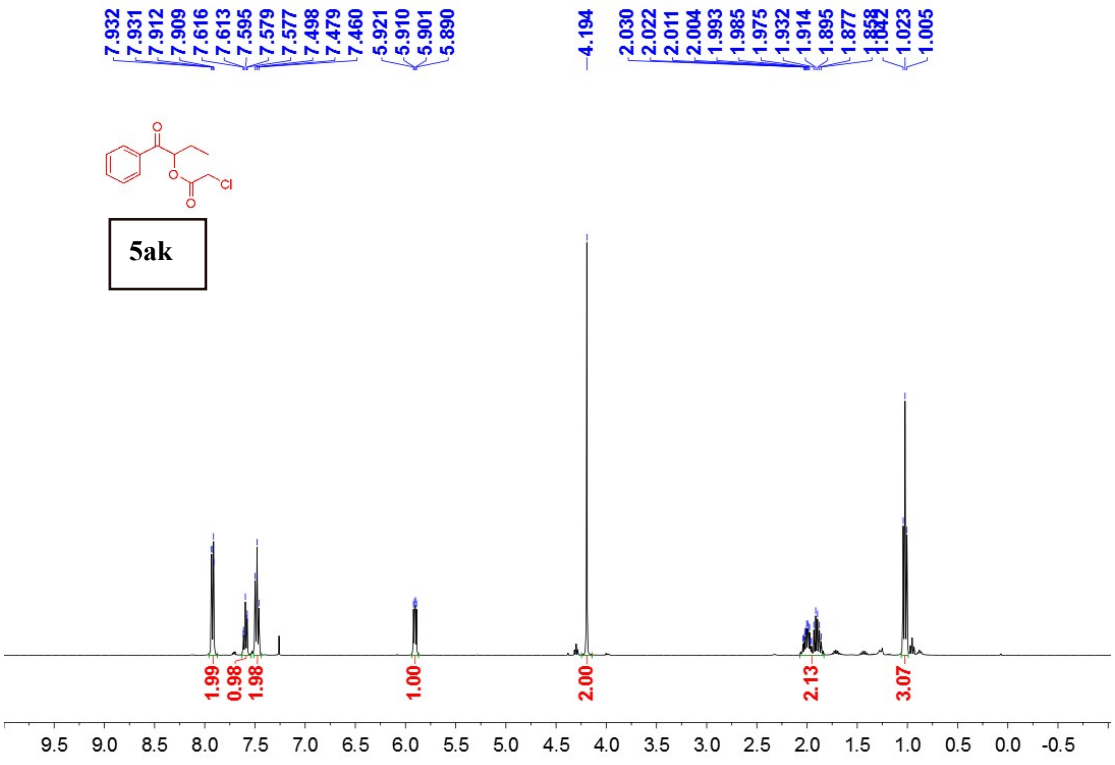
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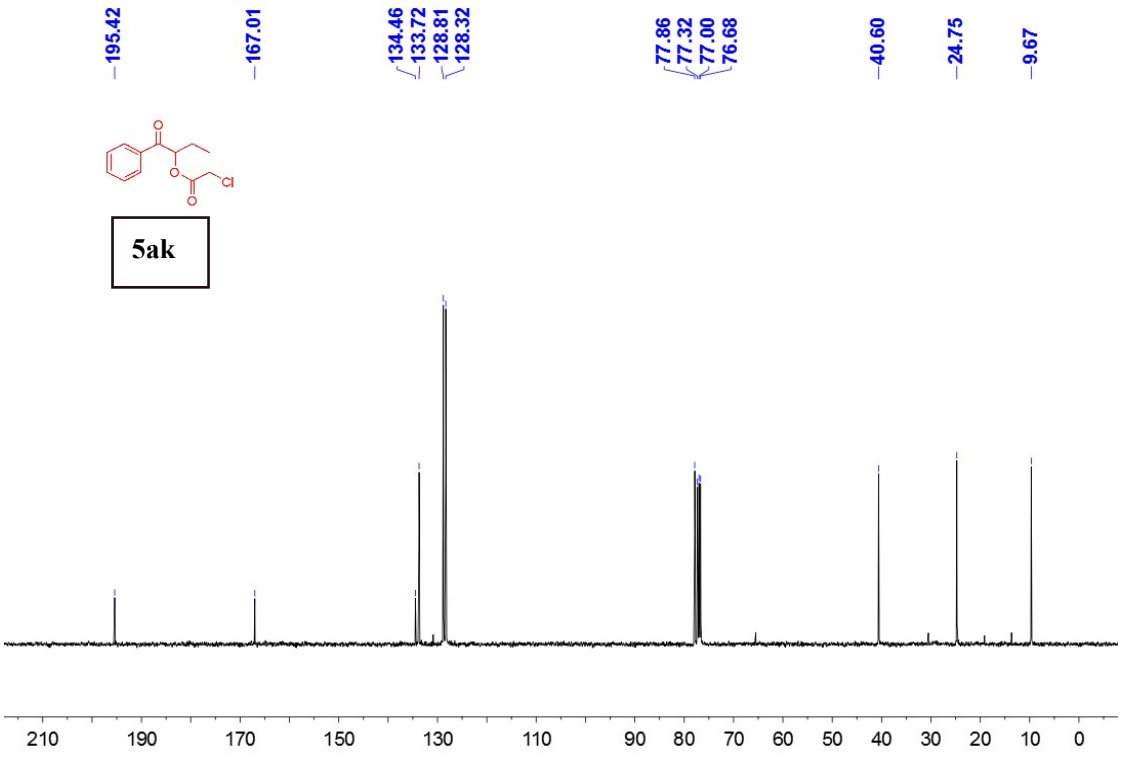
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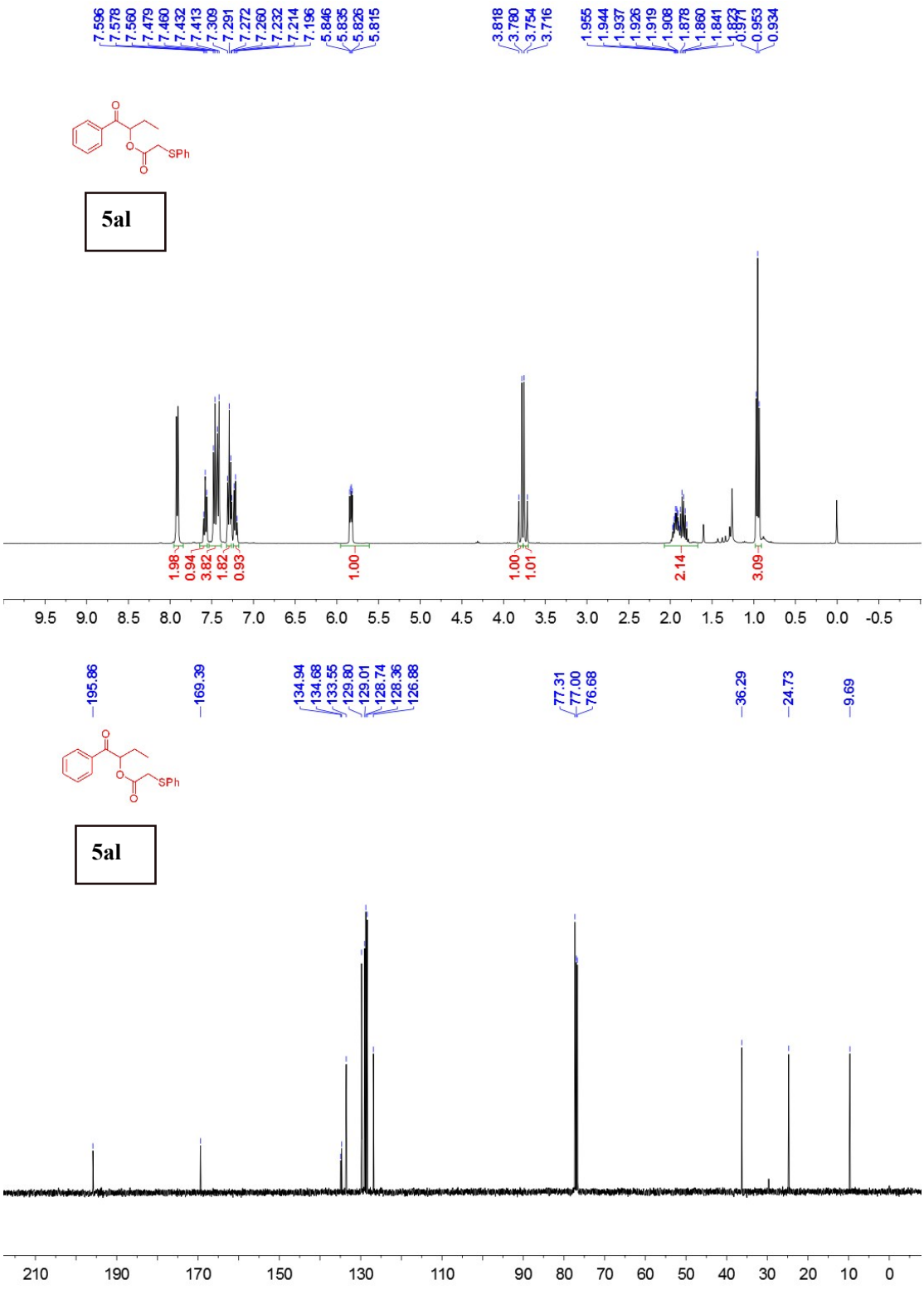
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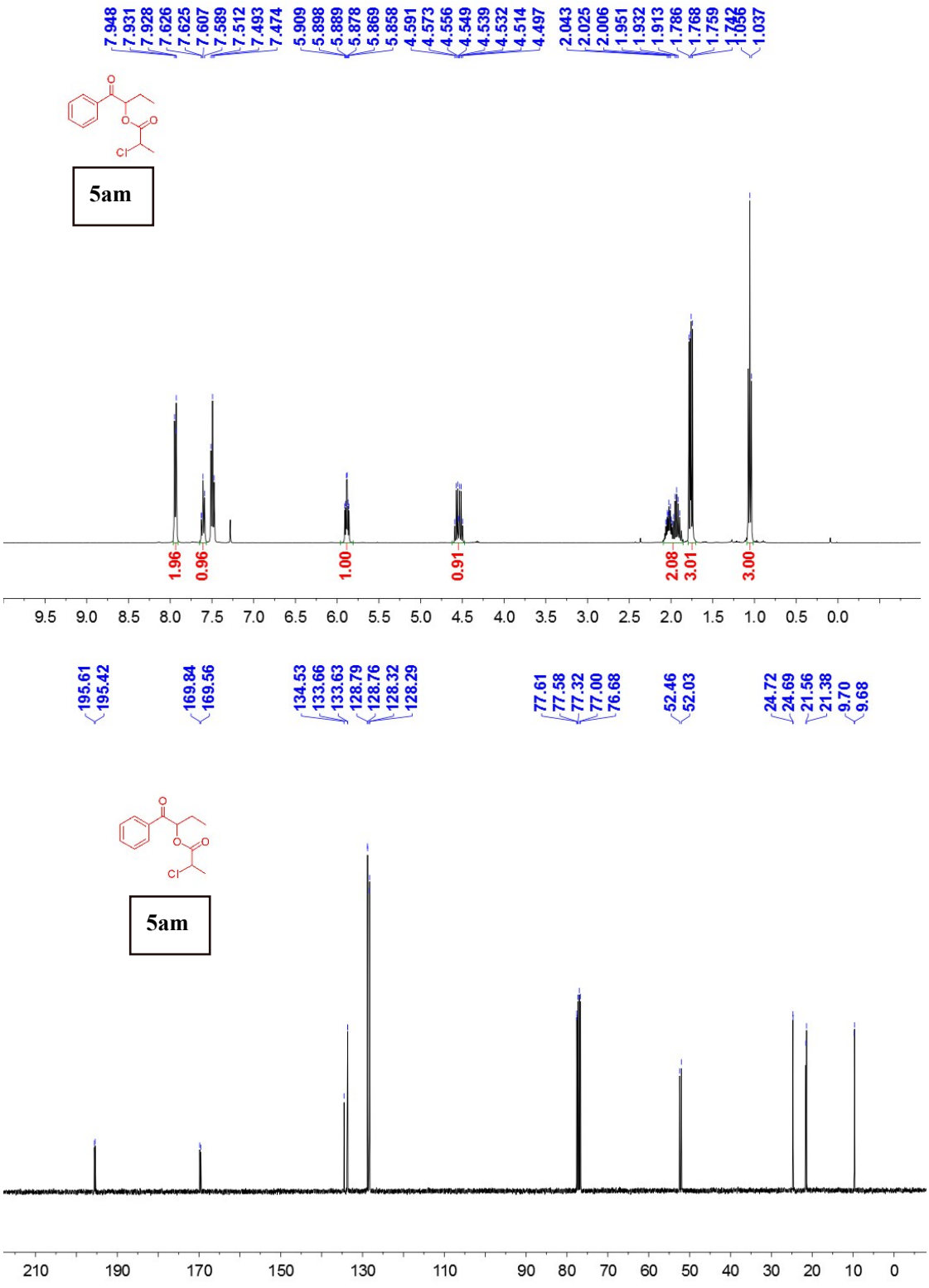
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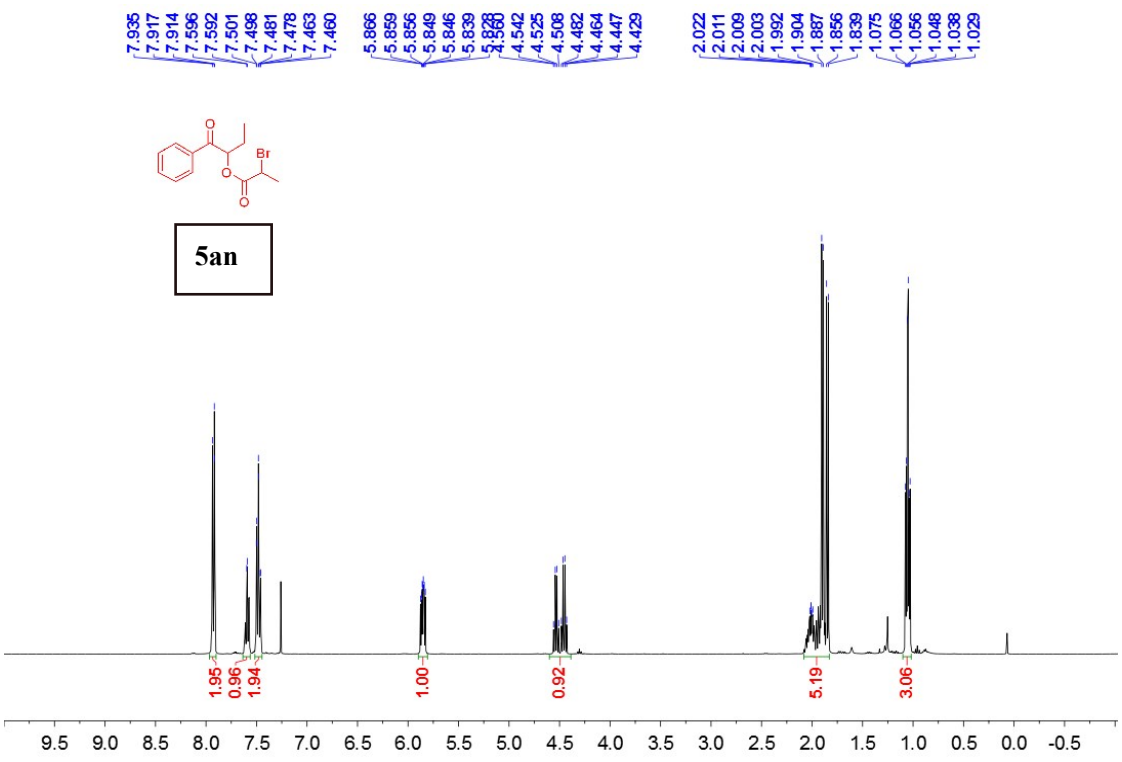
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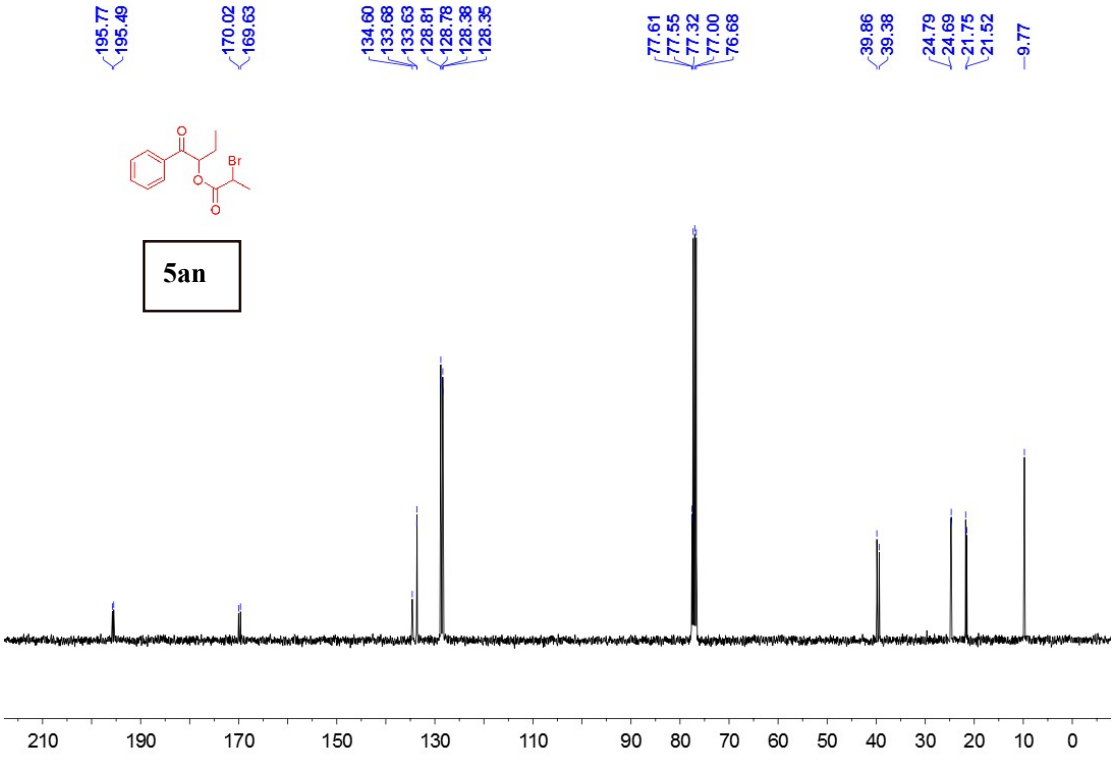
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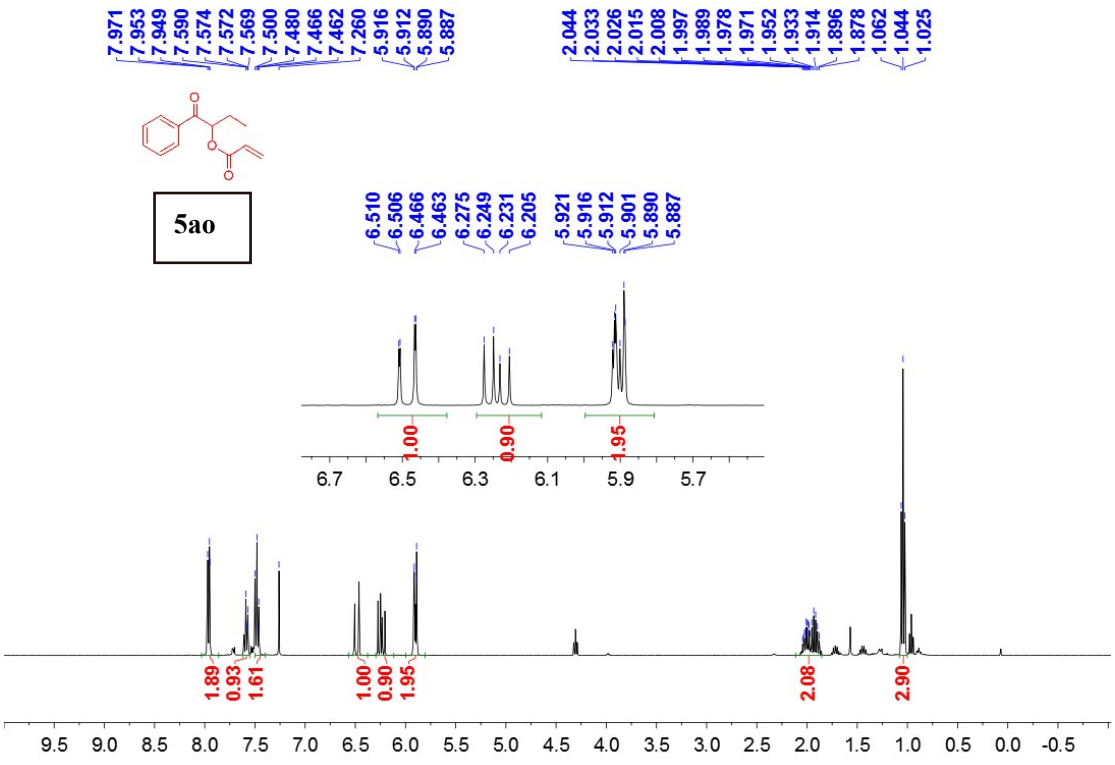
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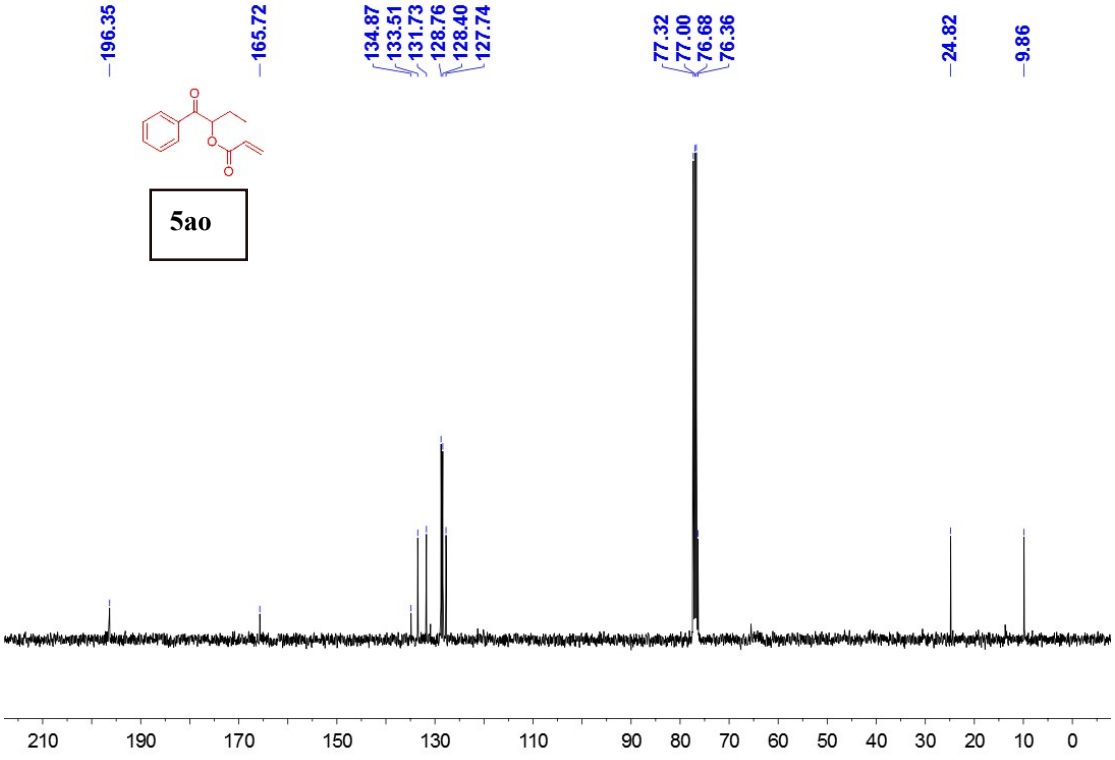
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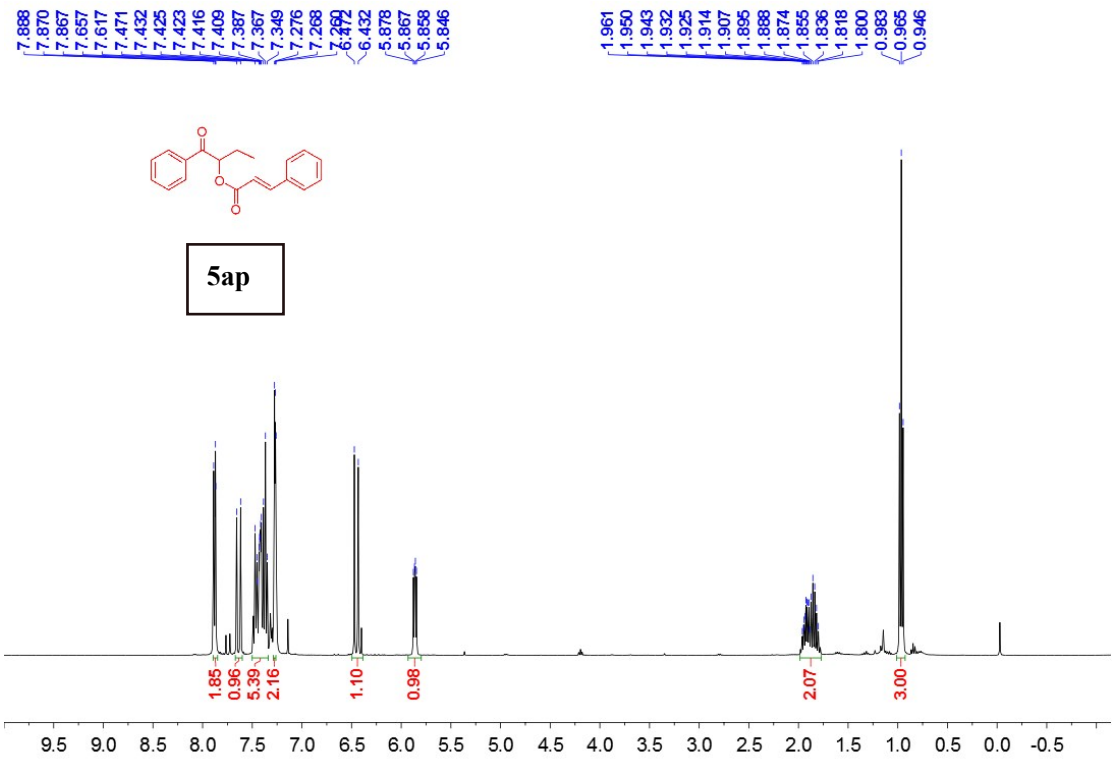
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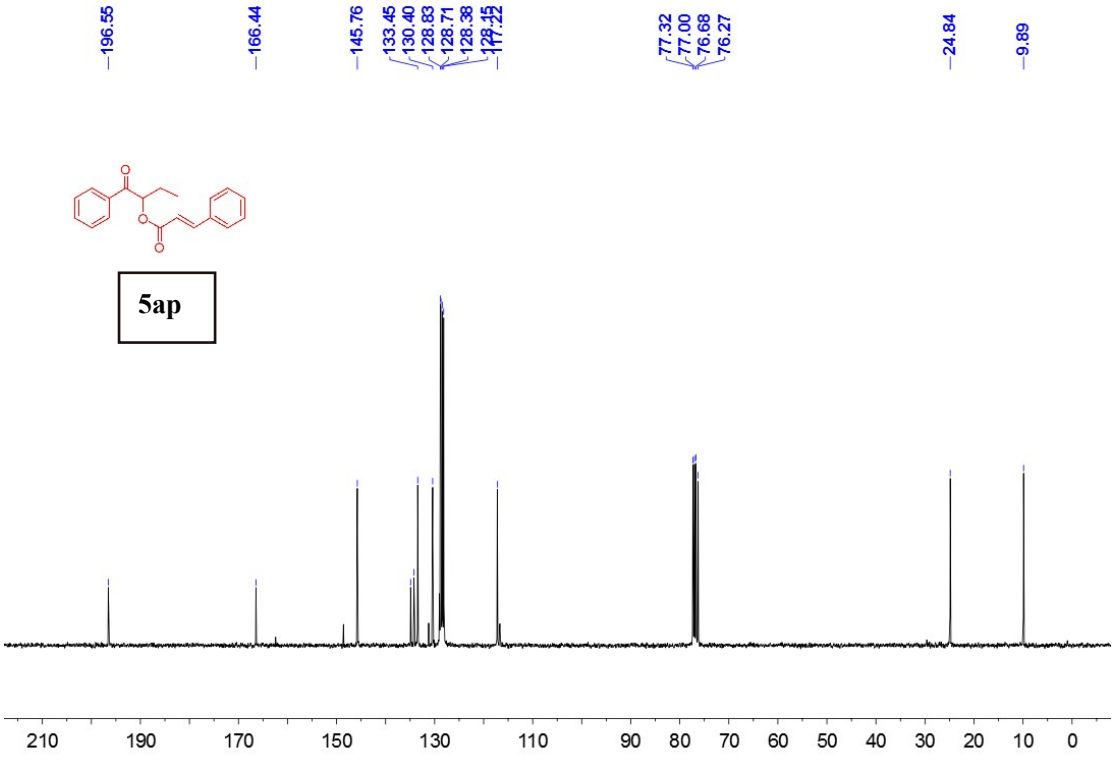
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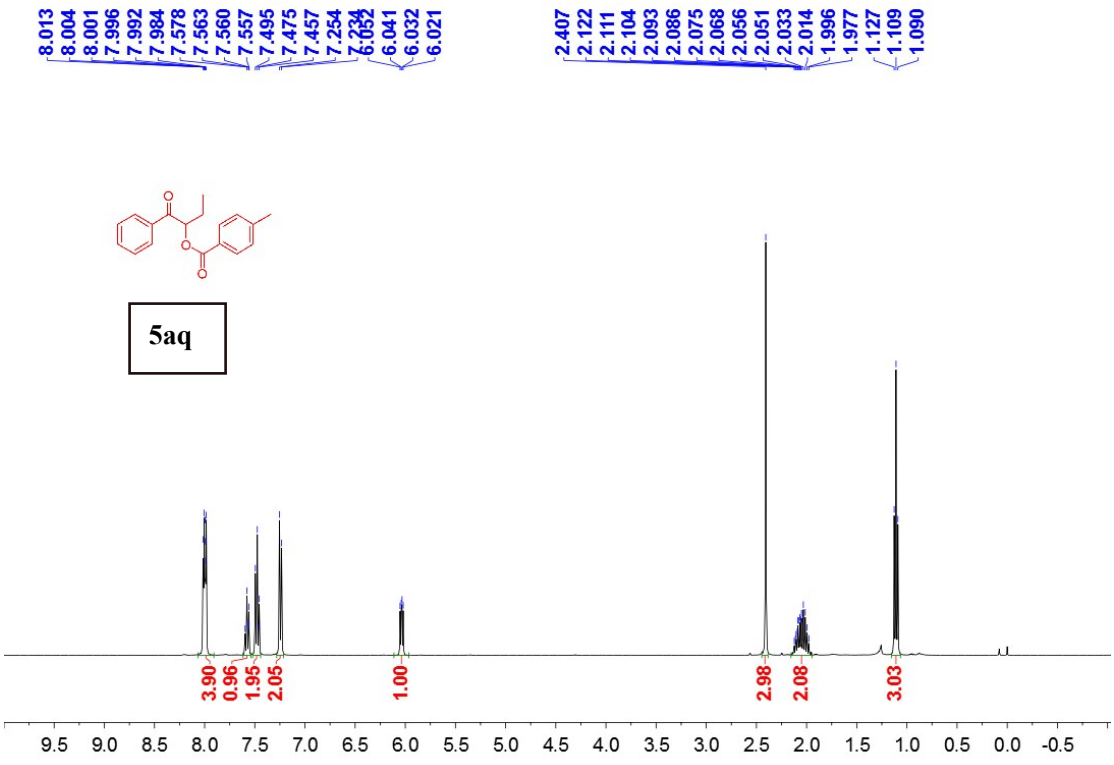
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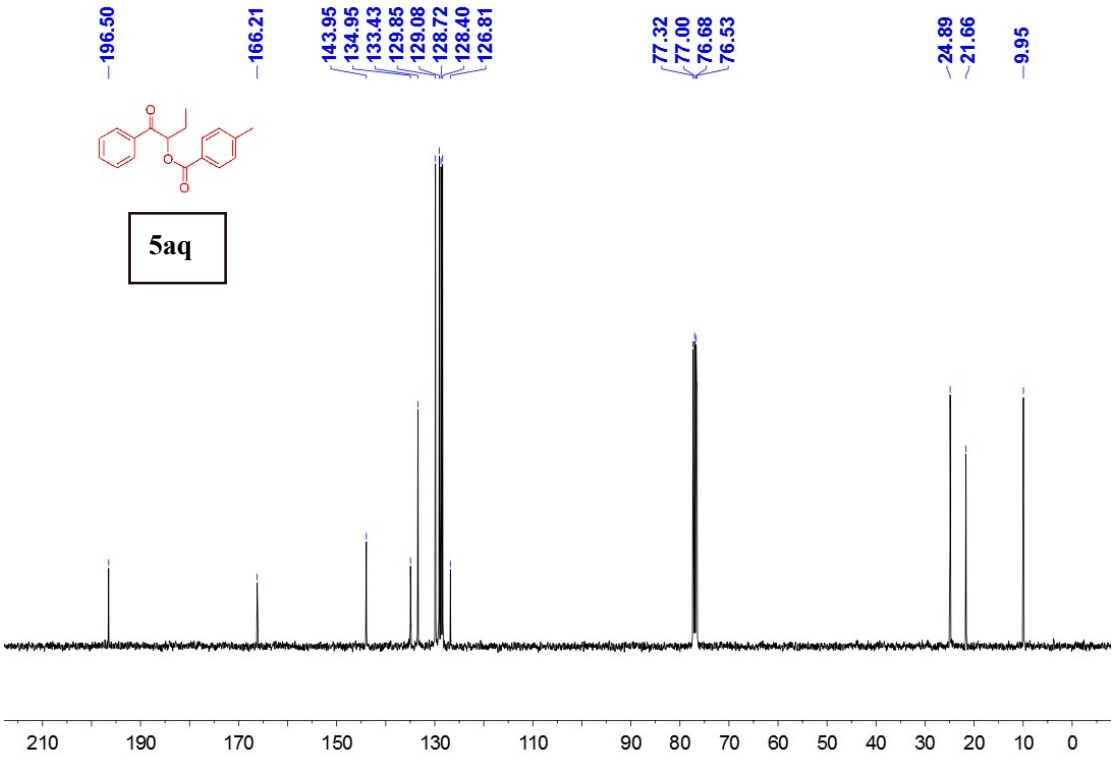
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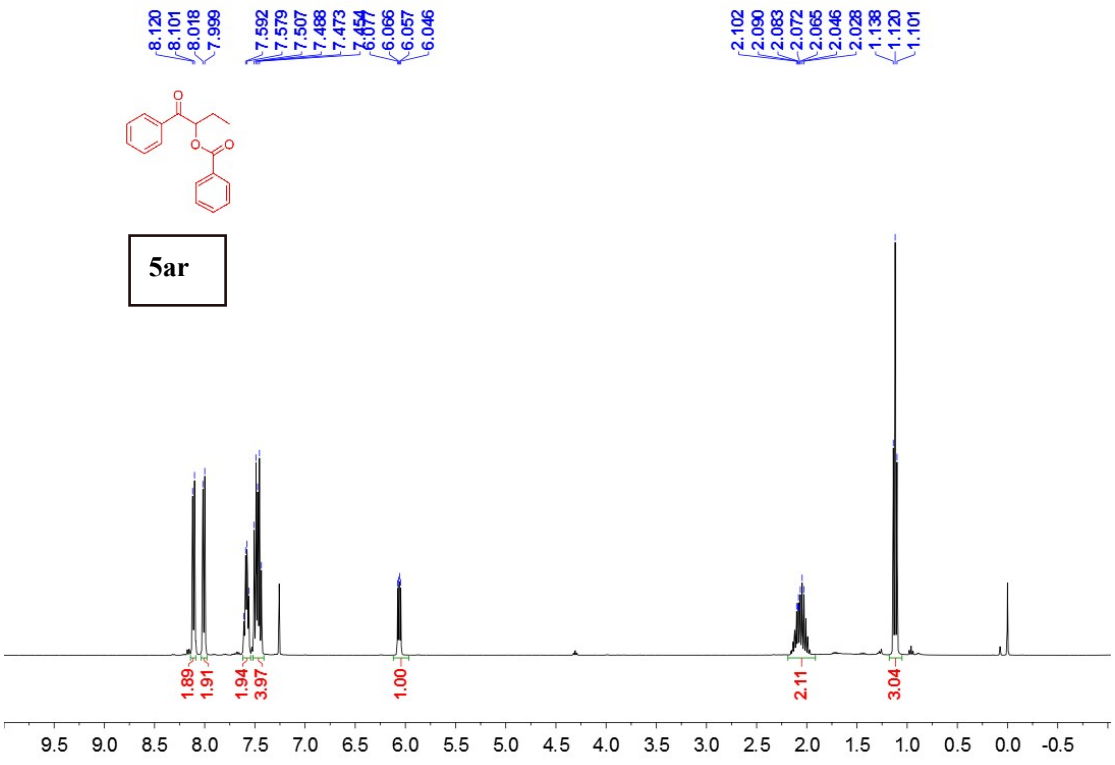
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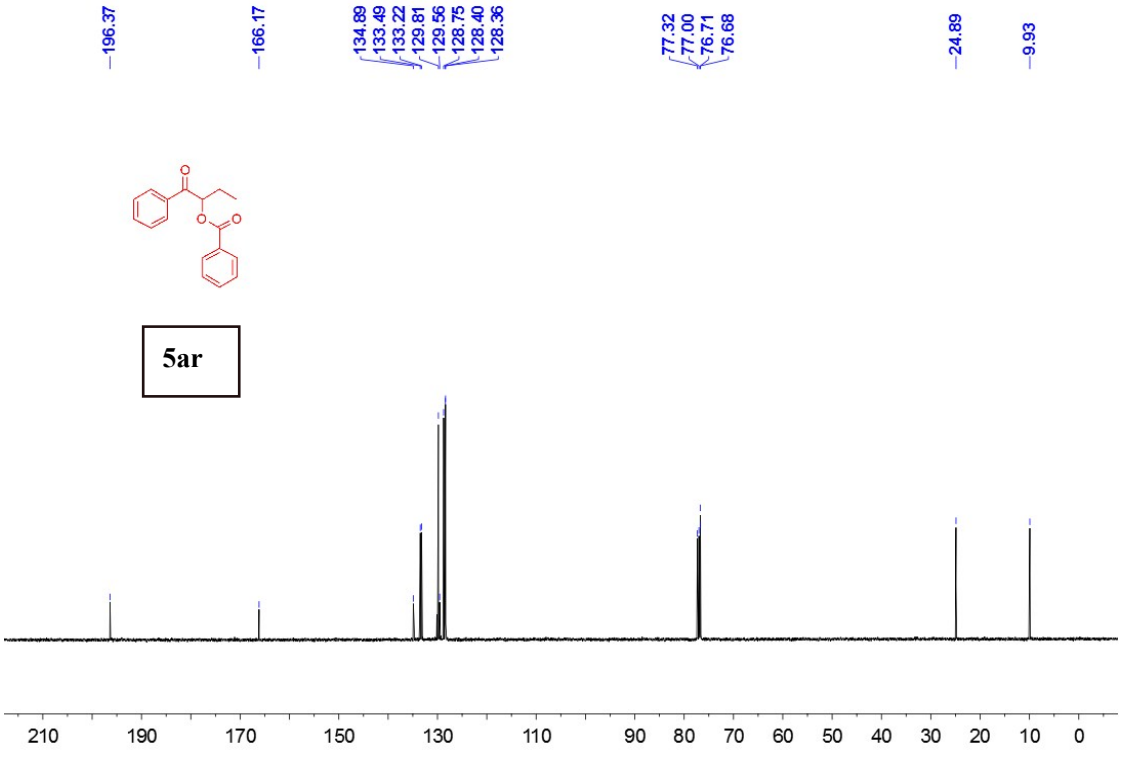
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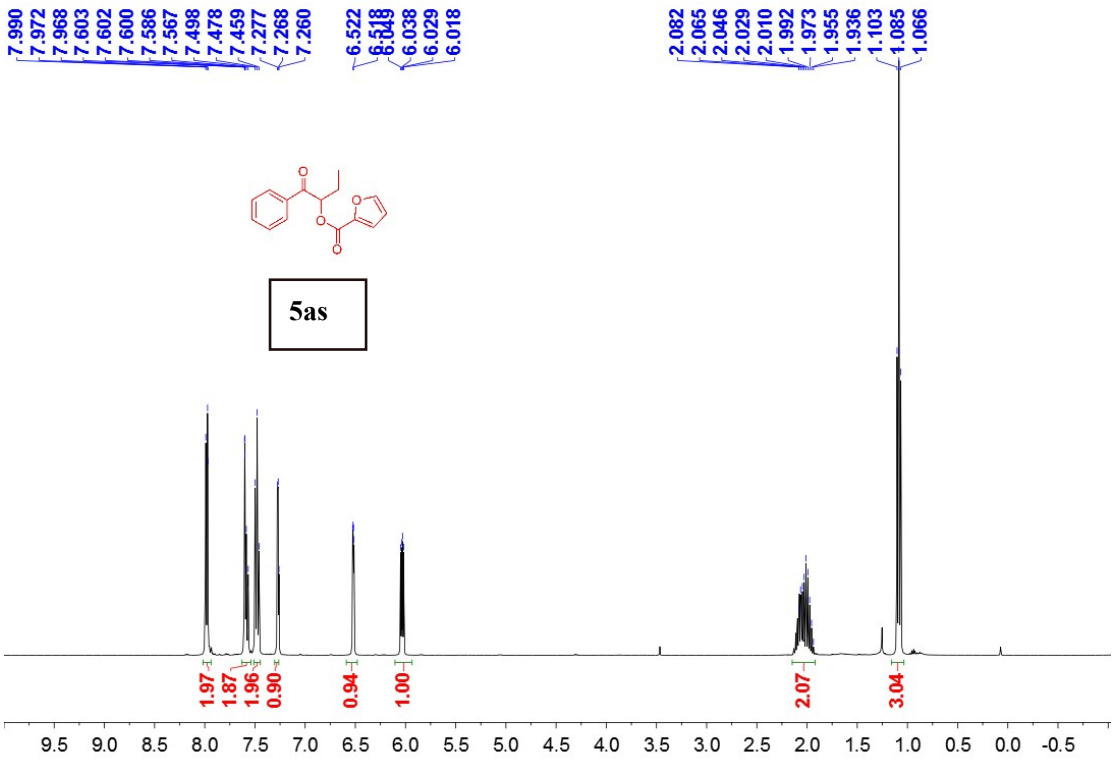
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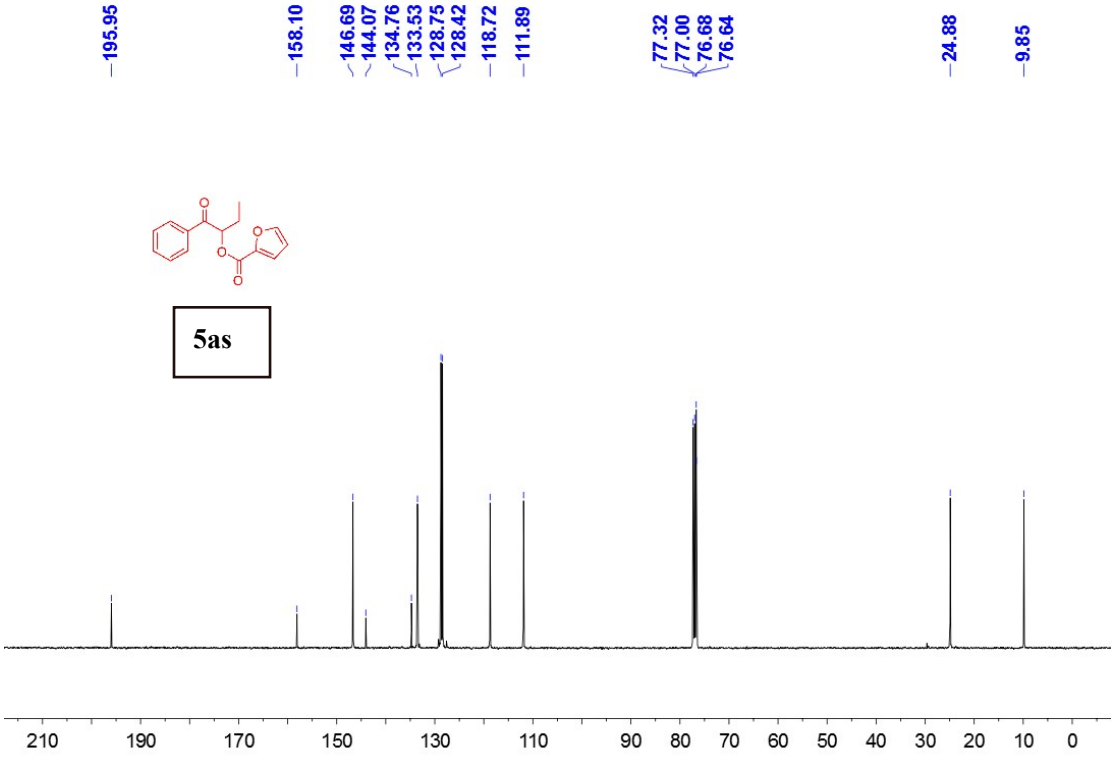
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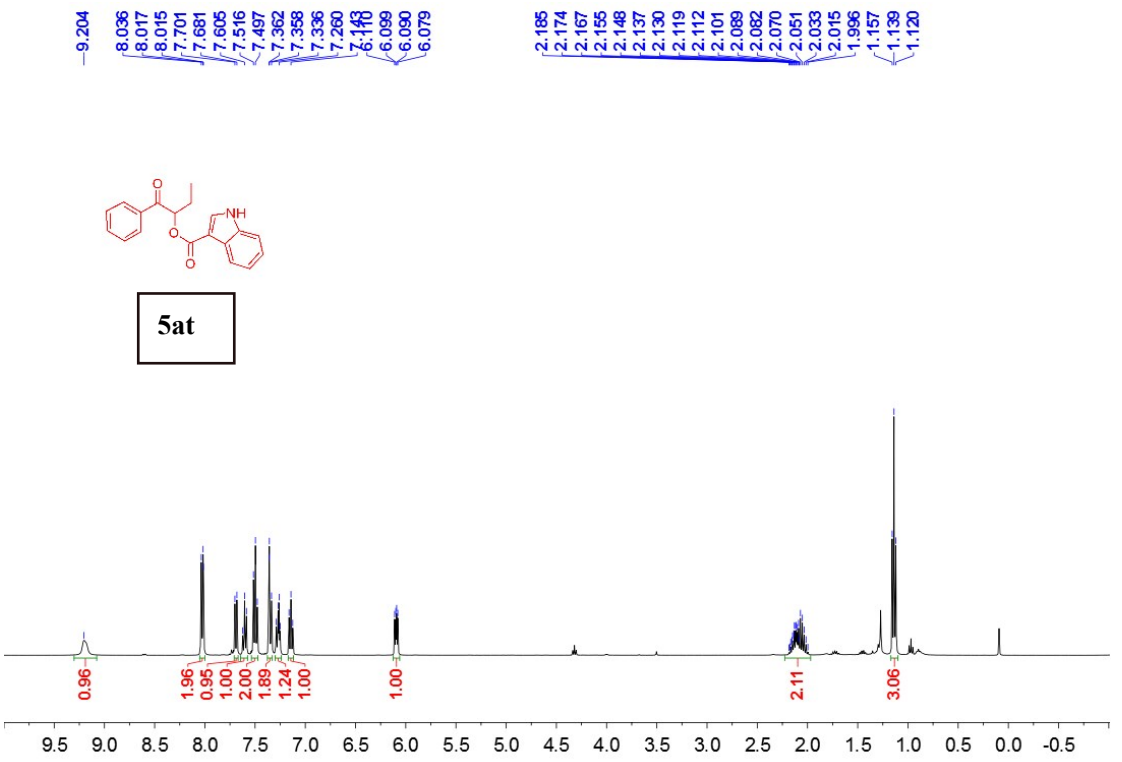
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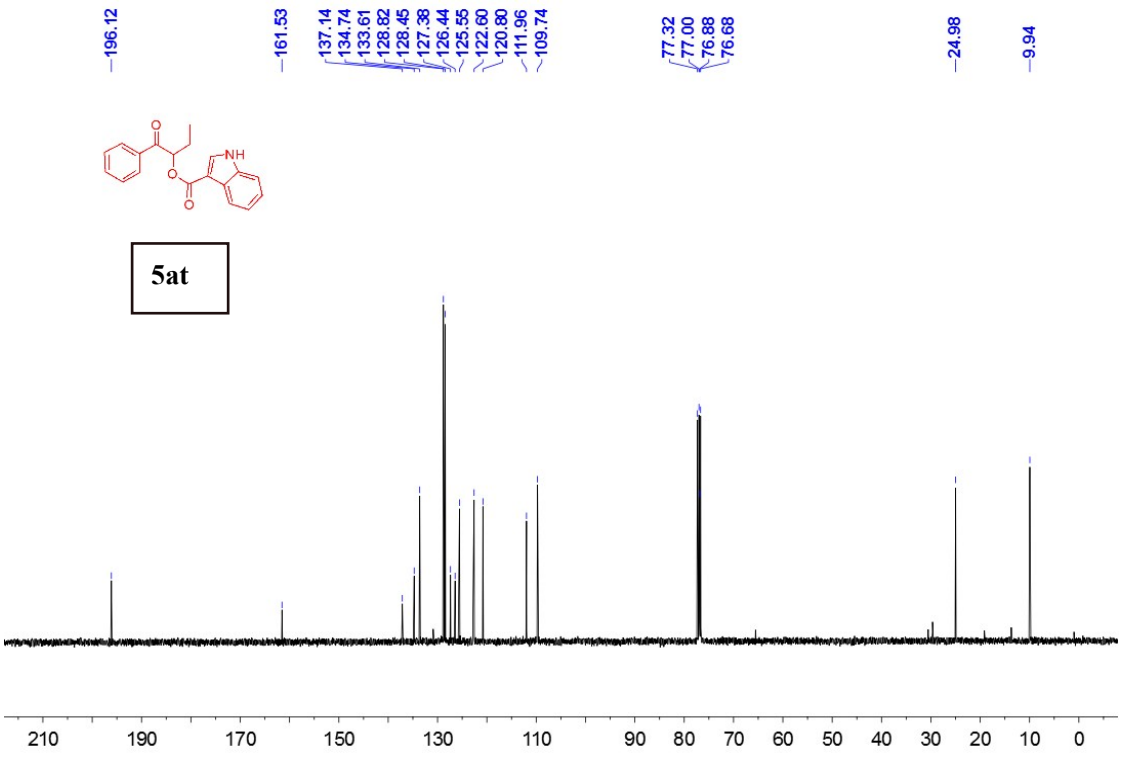
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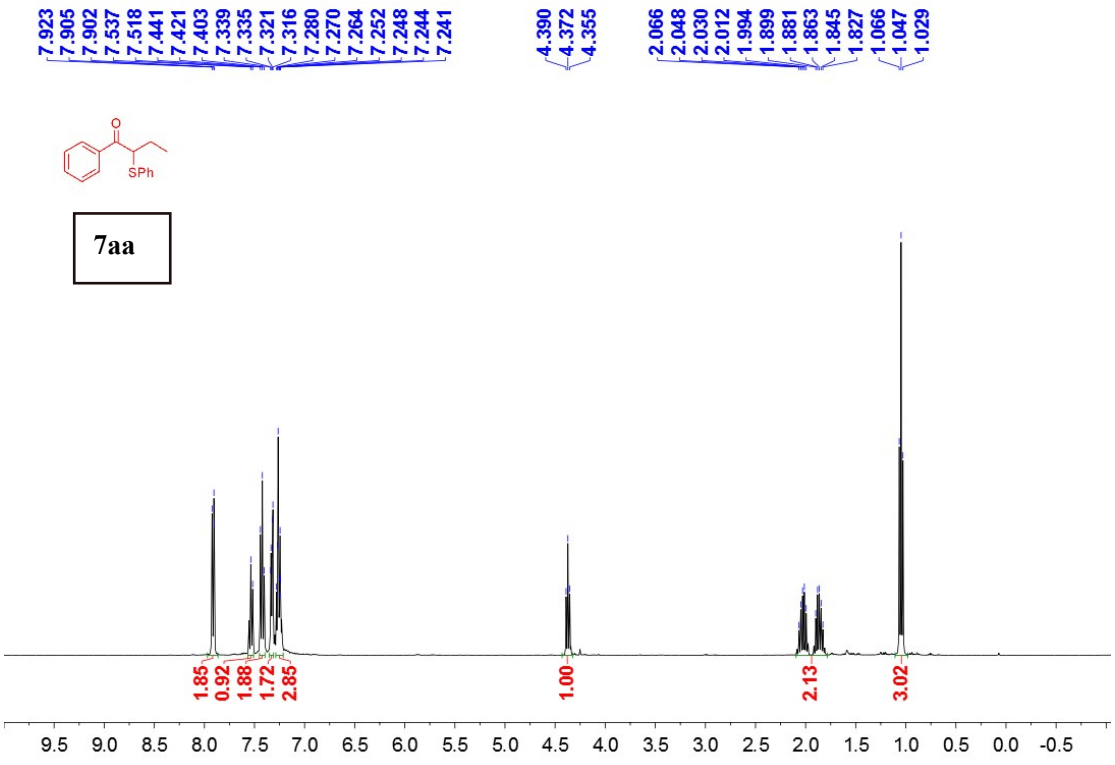
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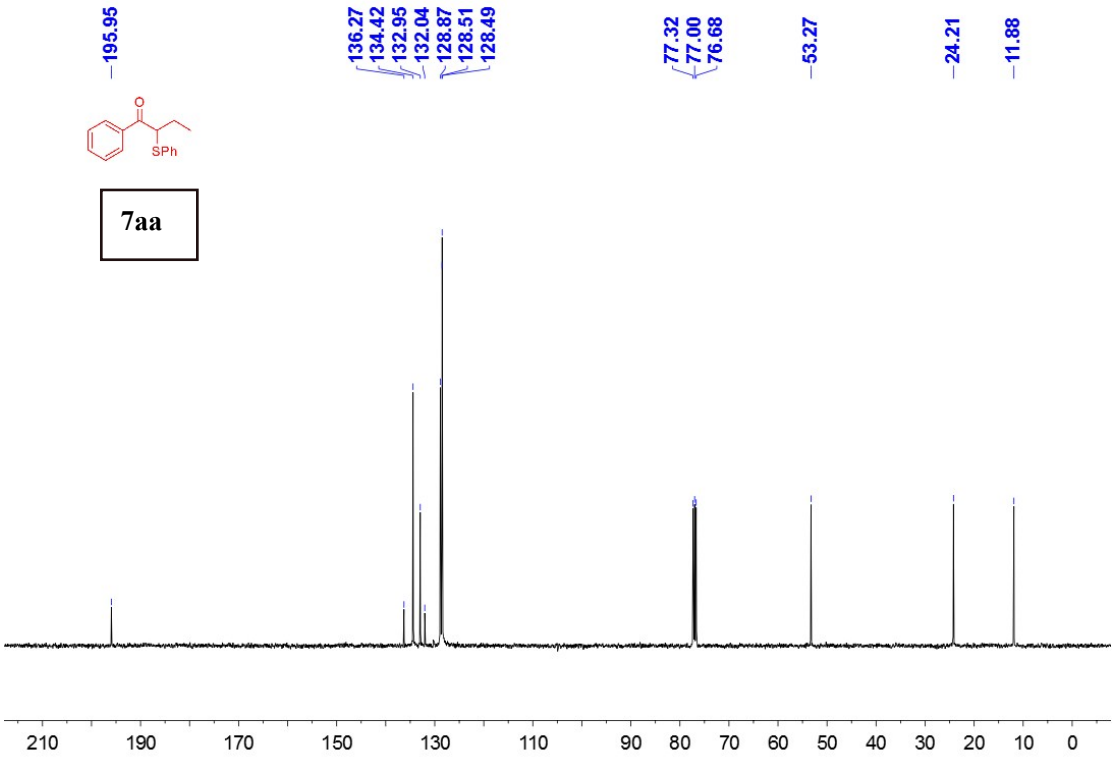
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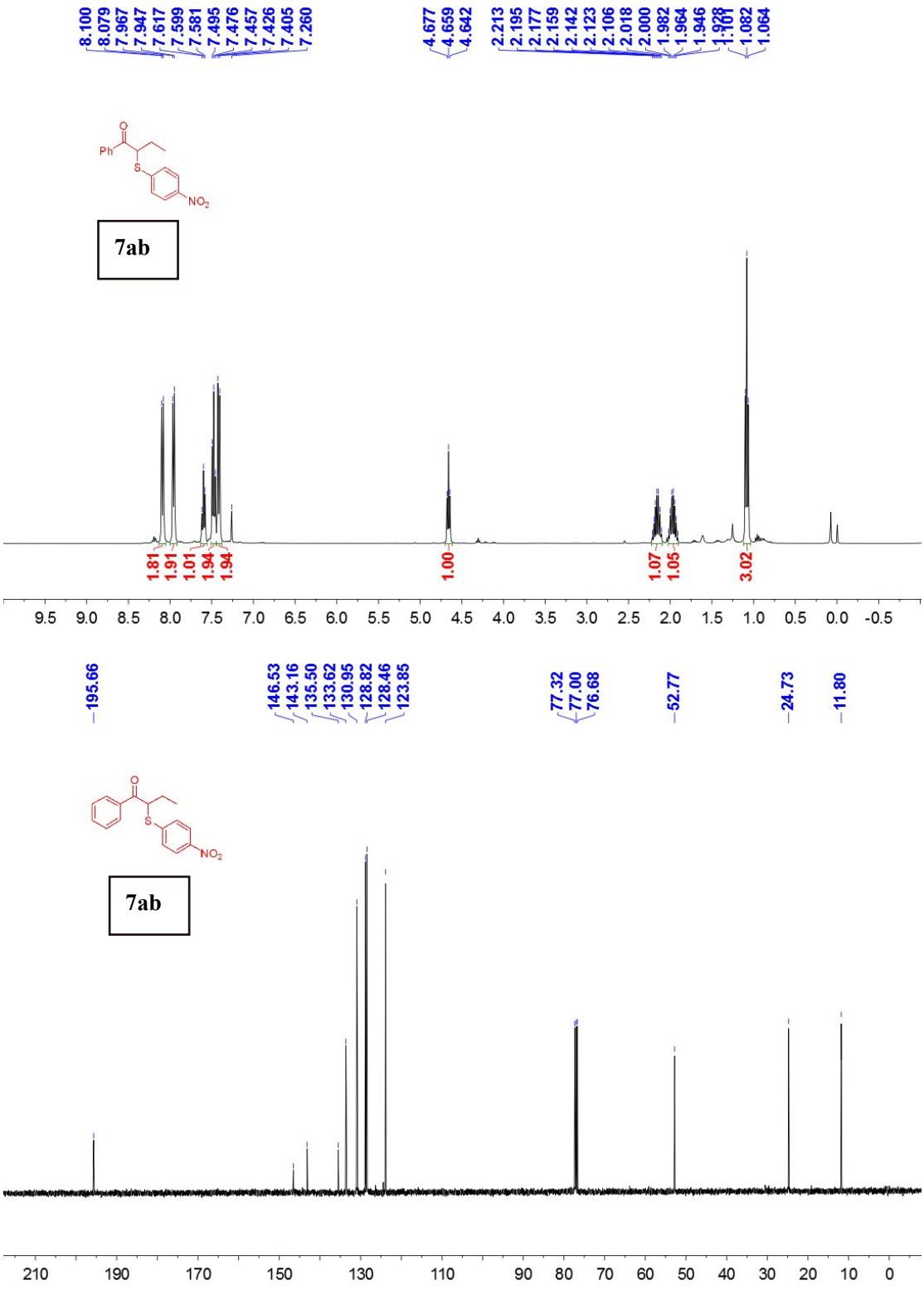
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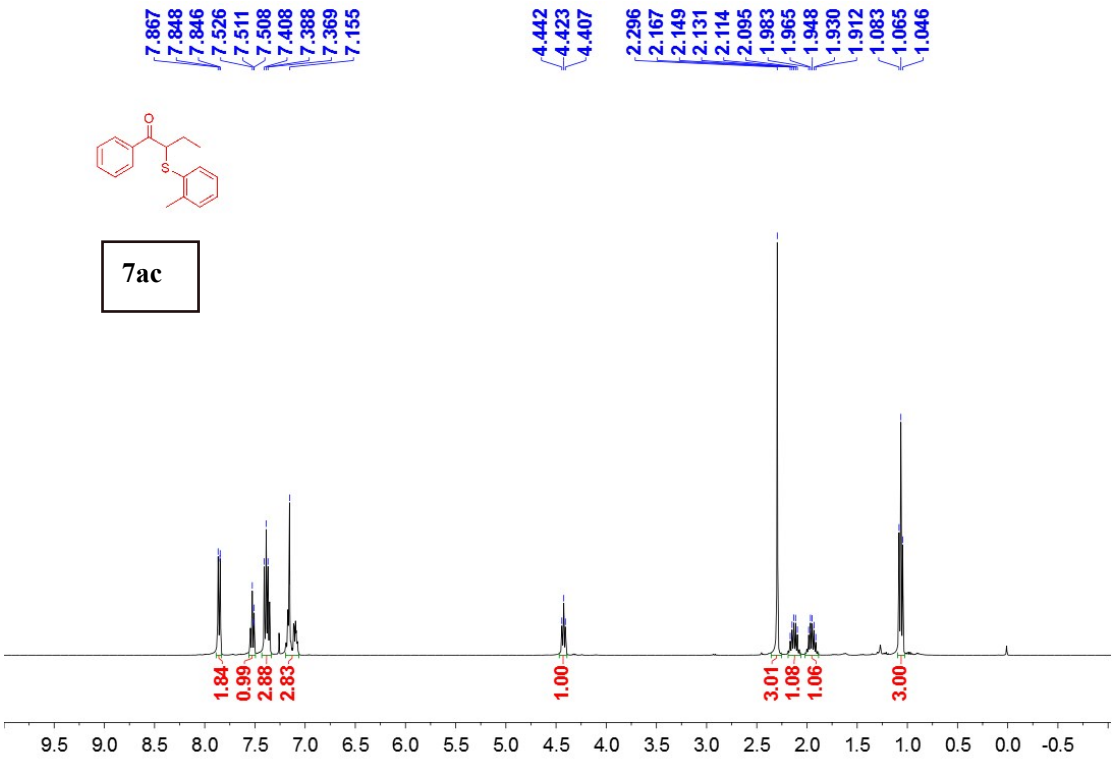
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1.08
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3.00
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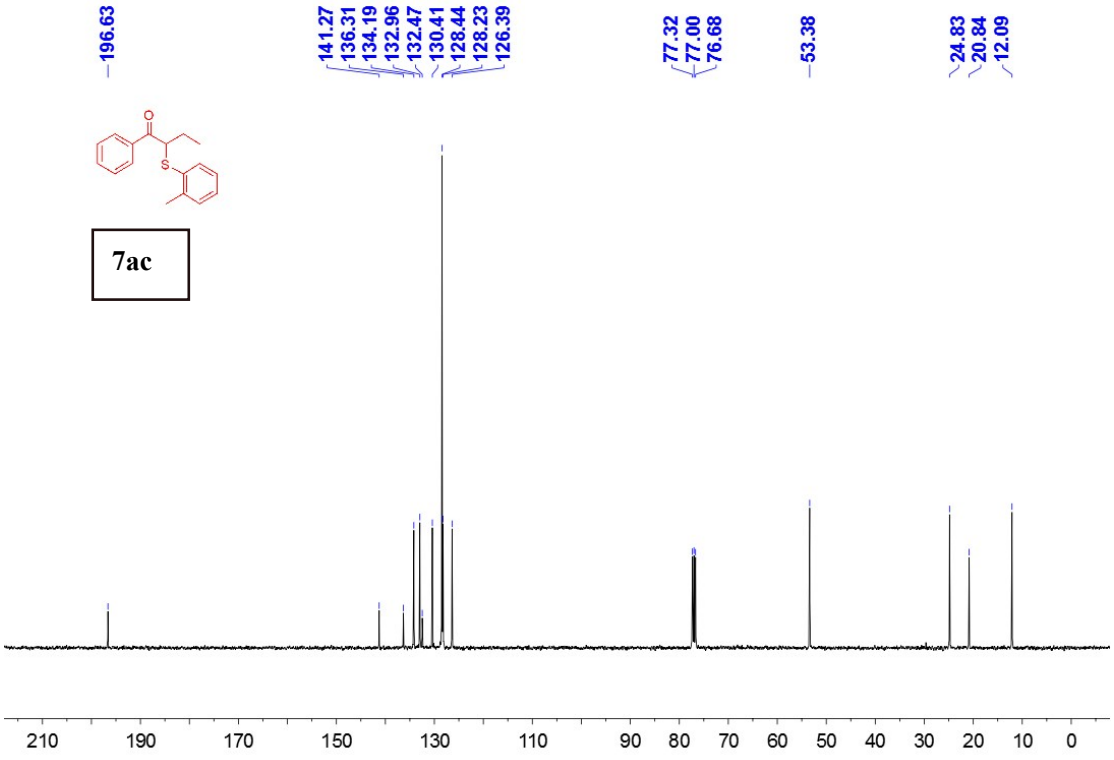
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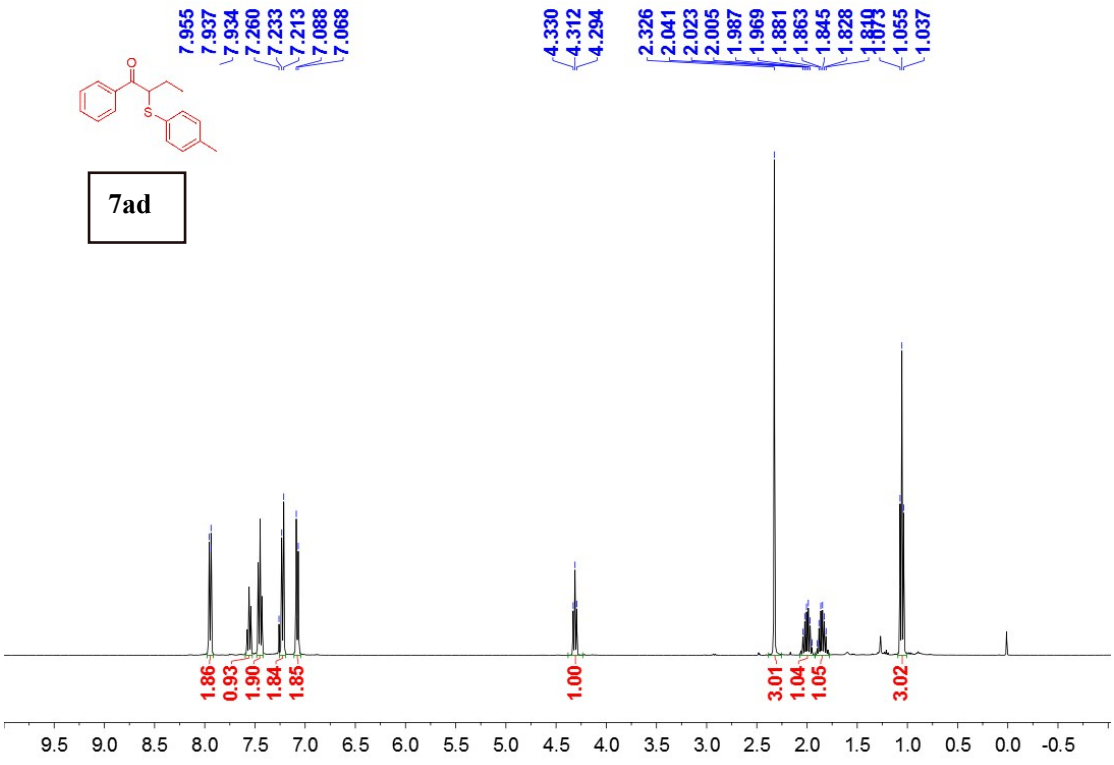
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1.05
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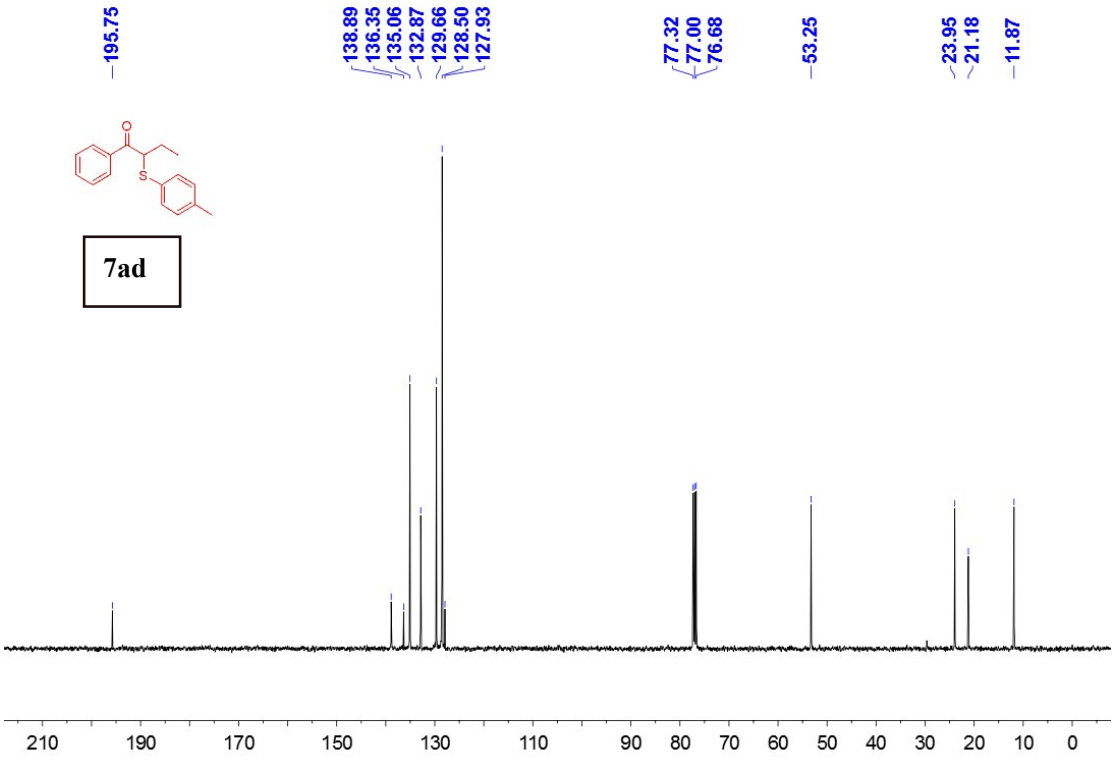
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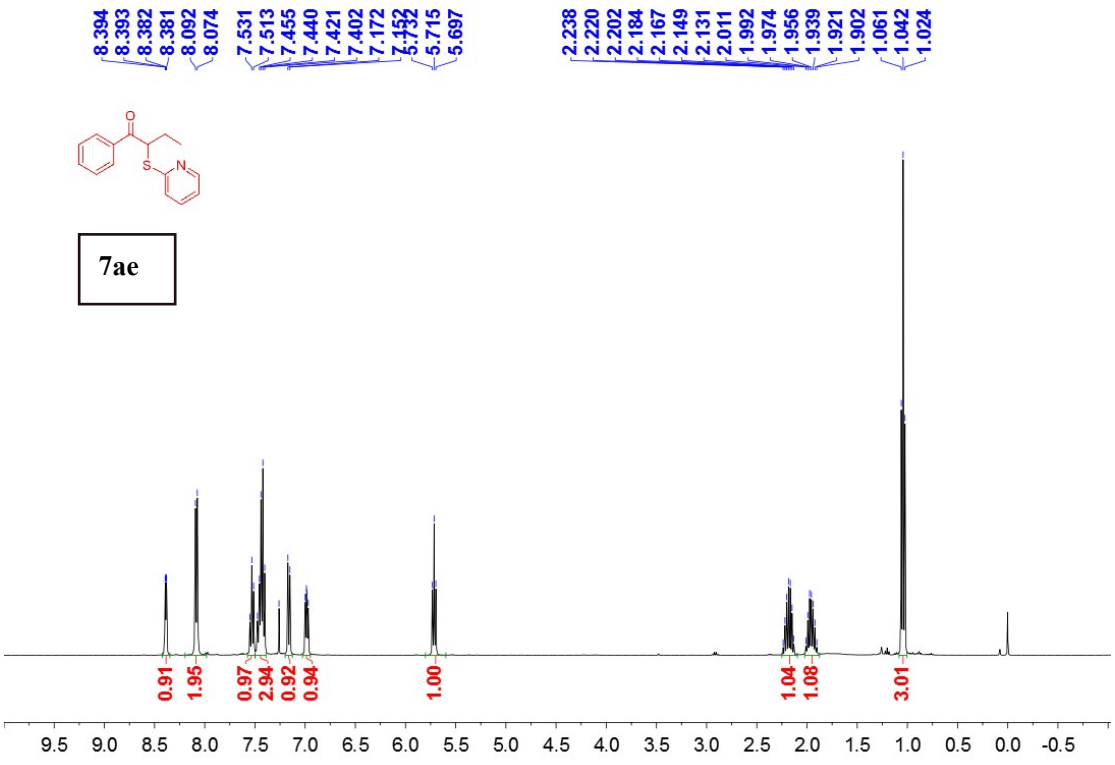
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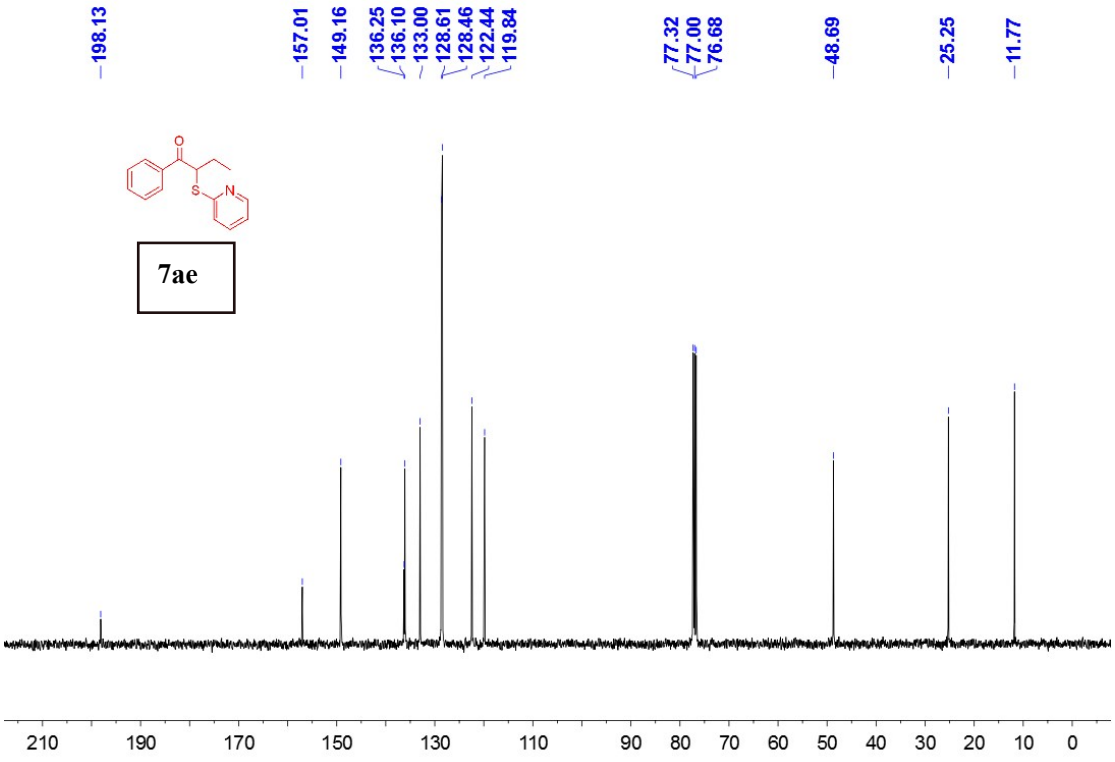
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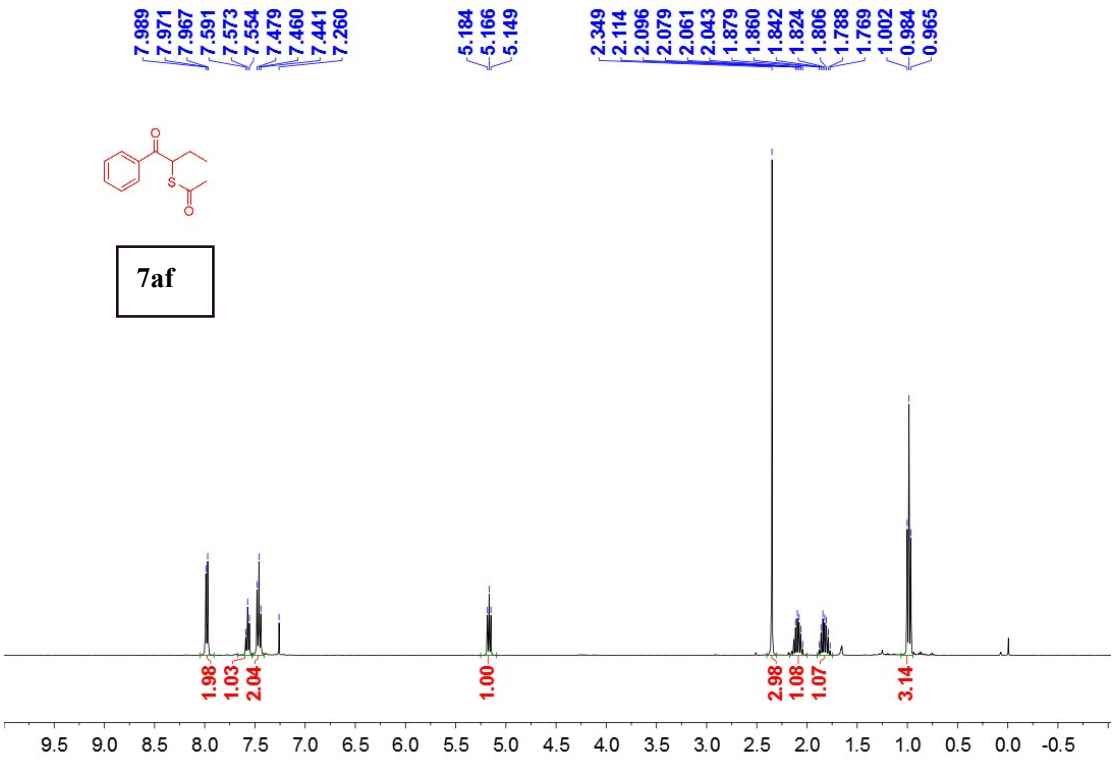
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1.08
1.07
3.14
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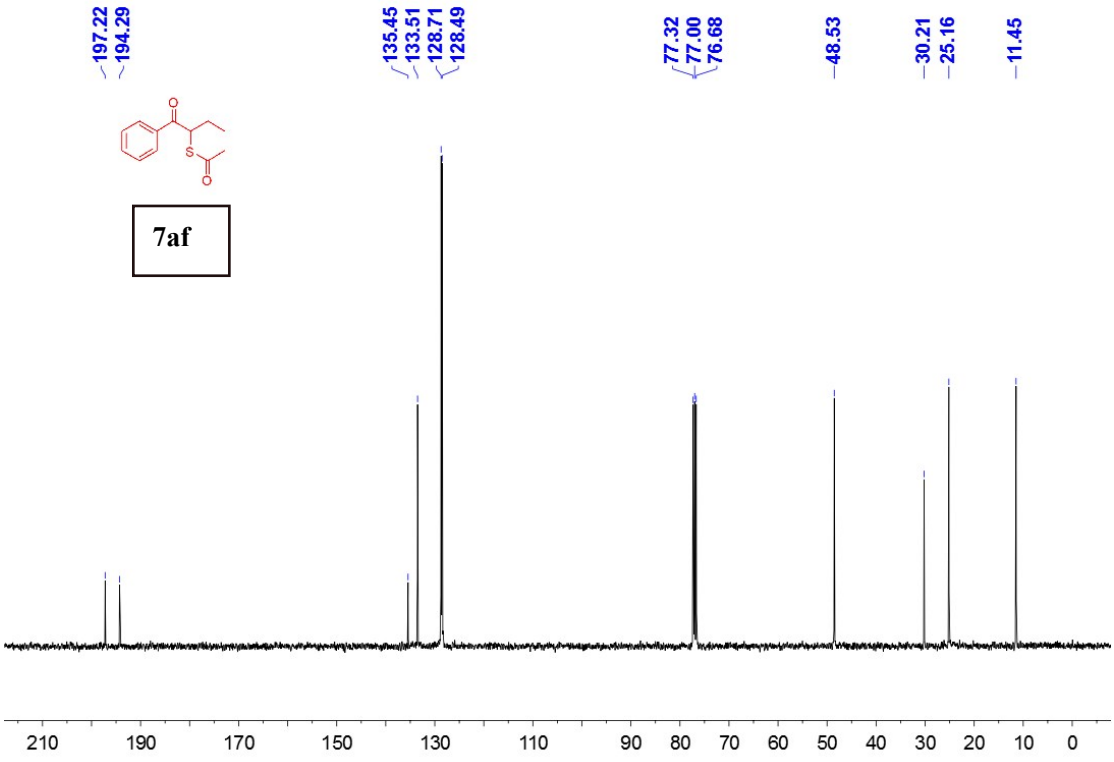
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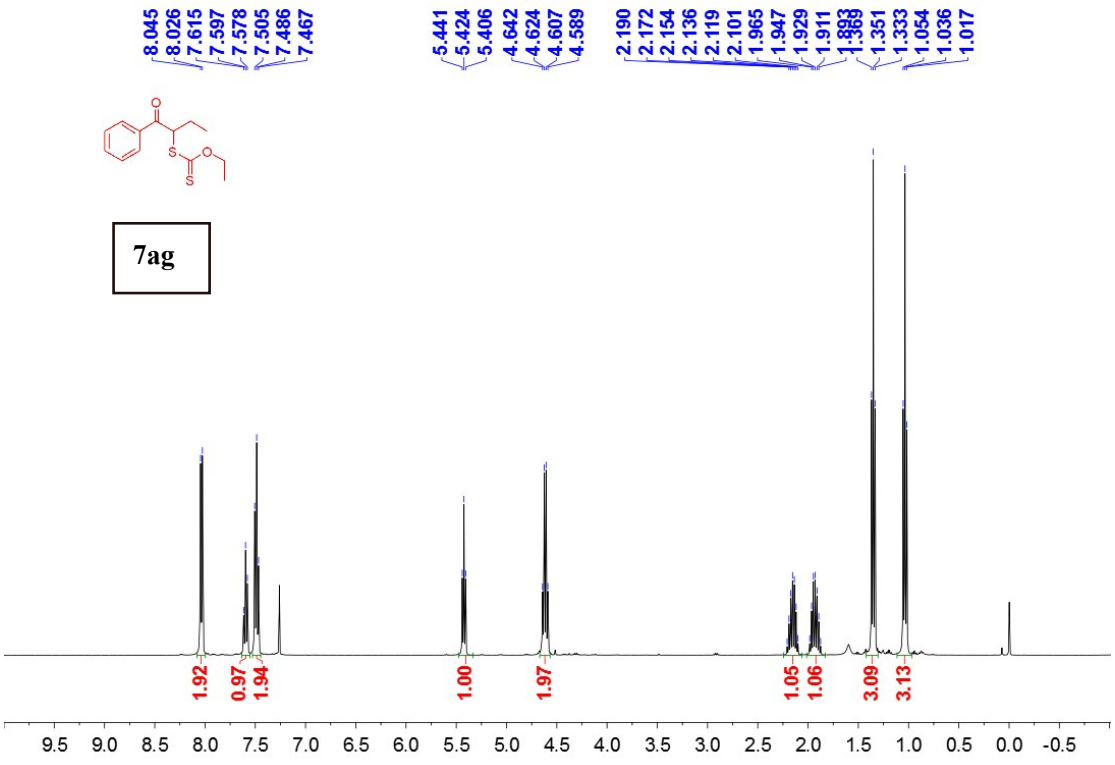
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1.06
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3.13
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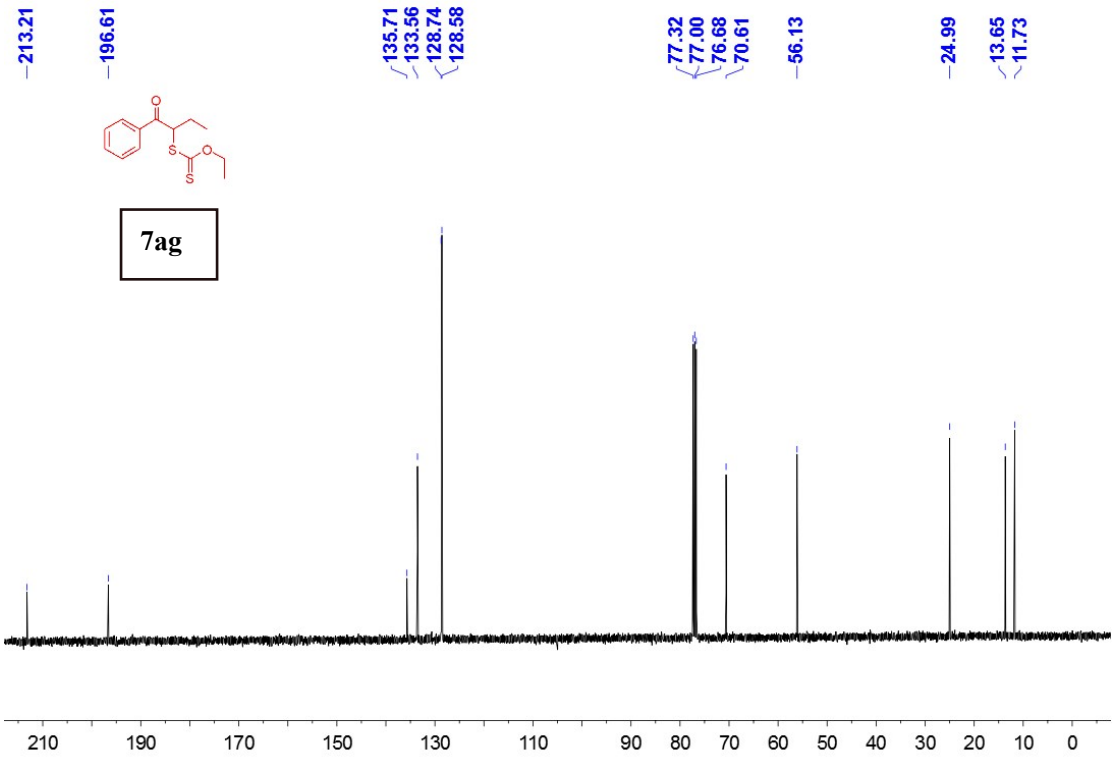
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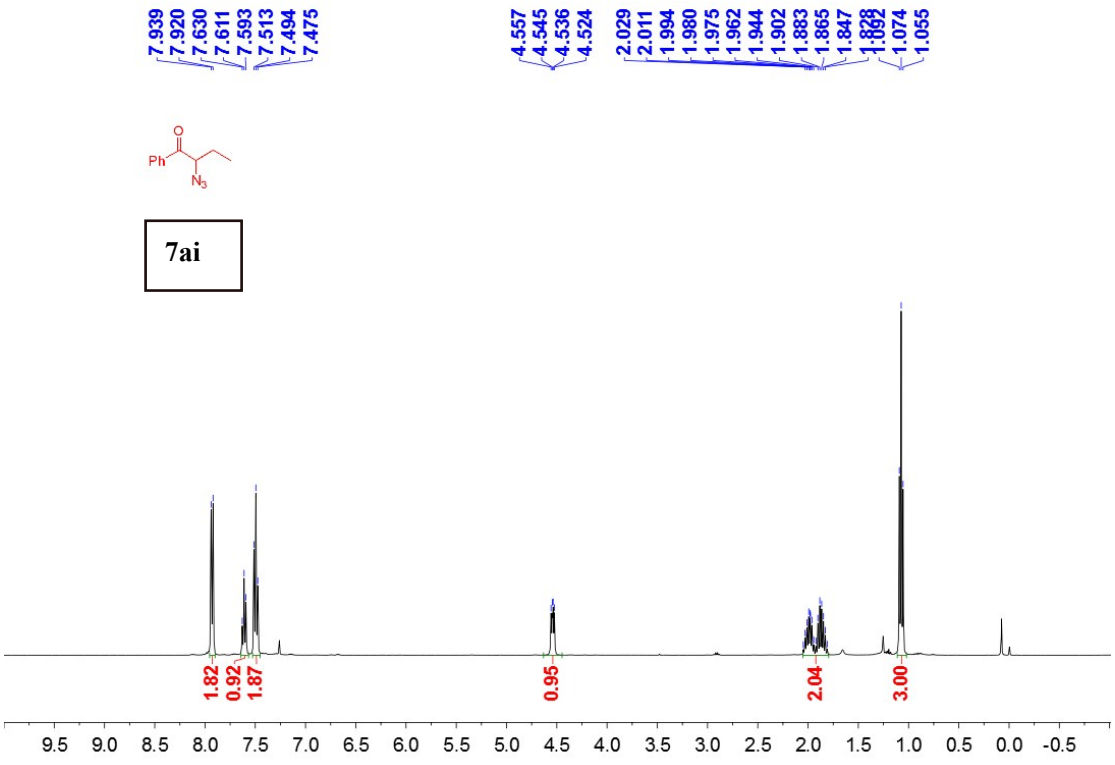
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1.055
Ph
N3
7ai
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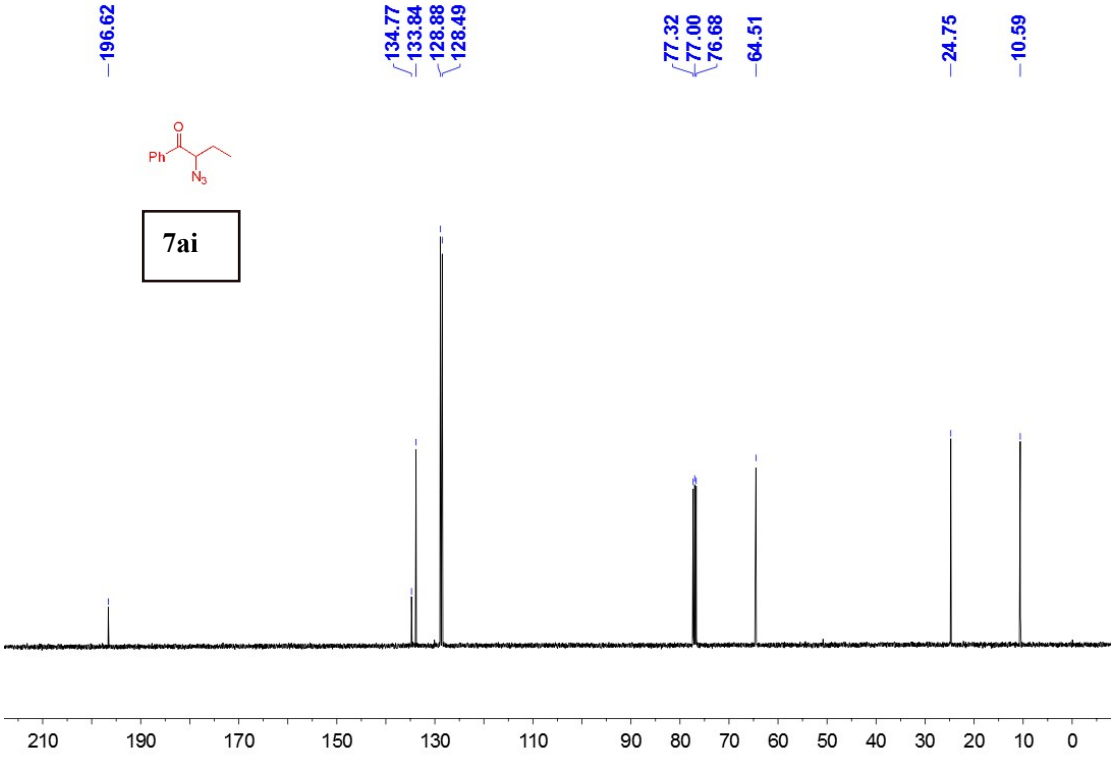
0.92
1.87
0.95
2.04
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5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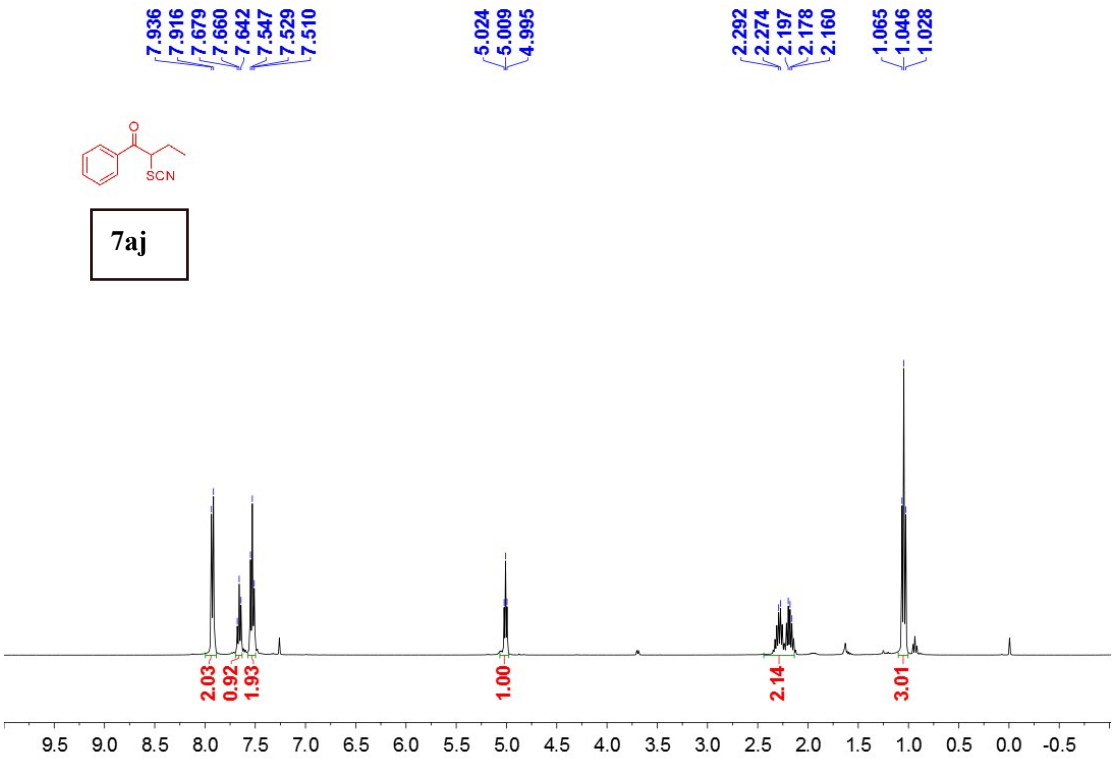
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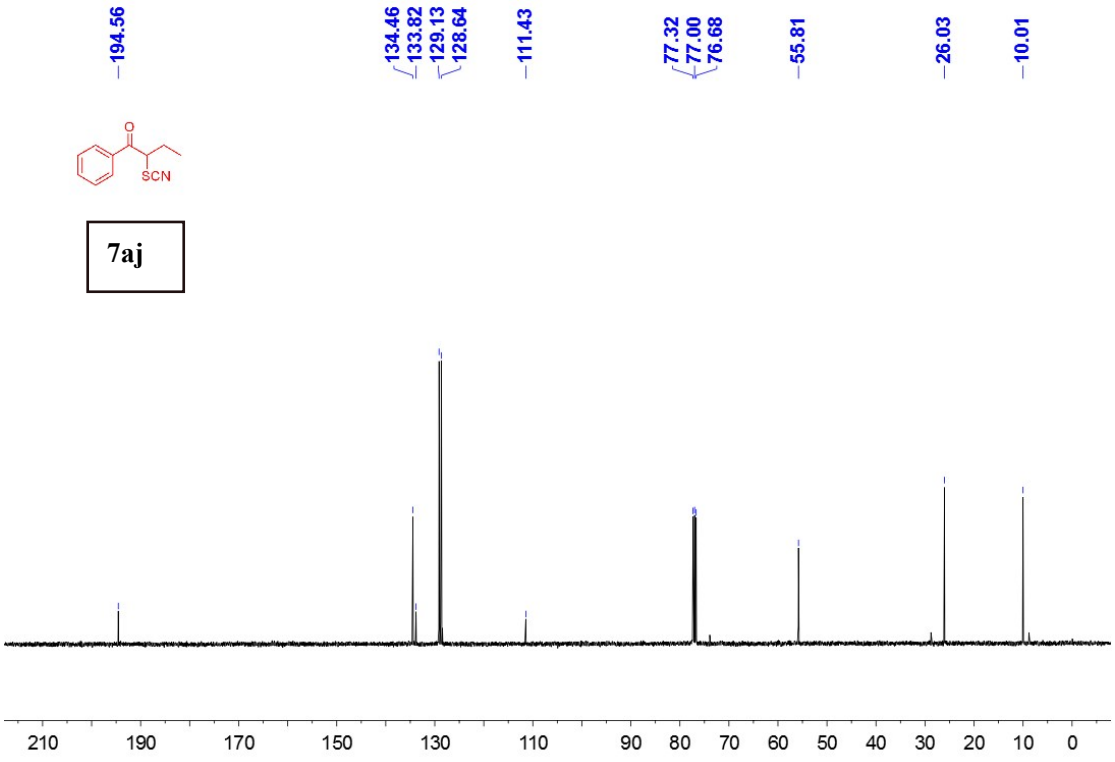
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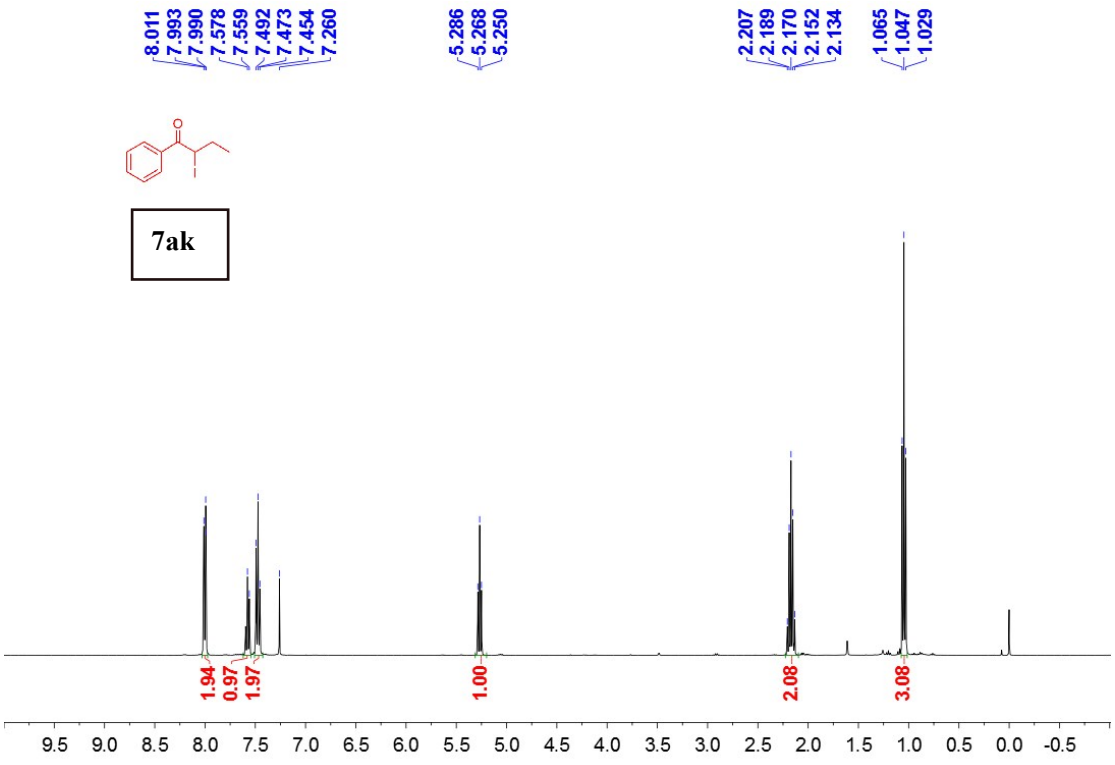
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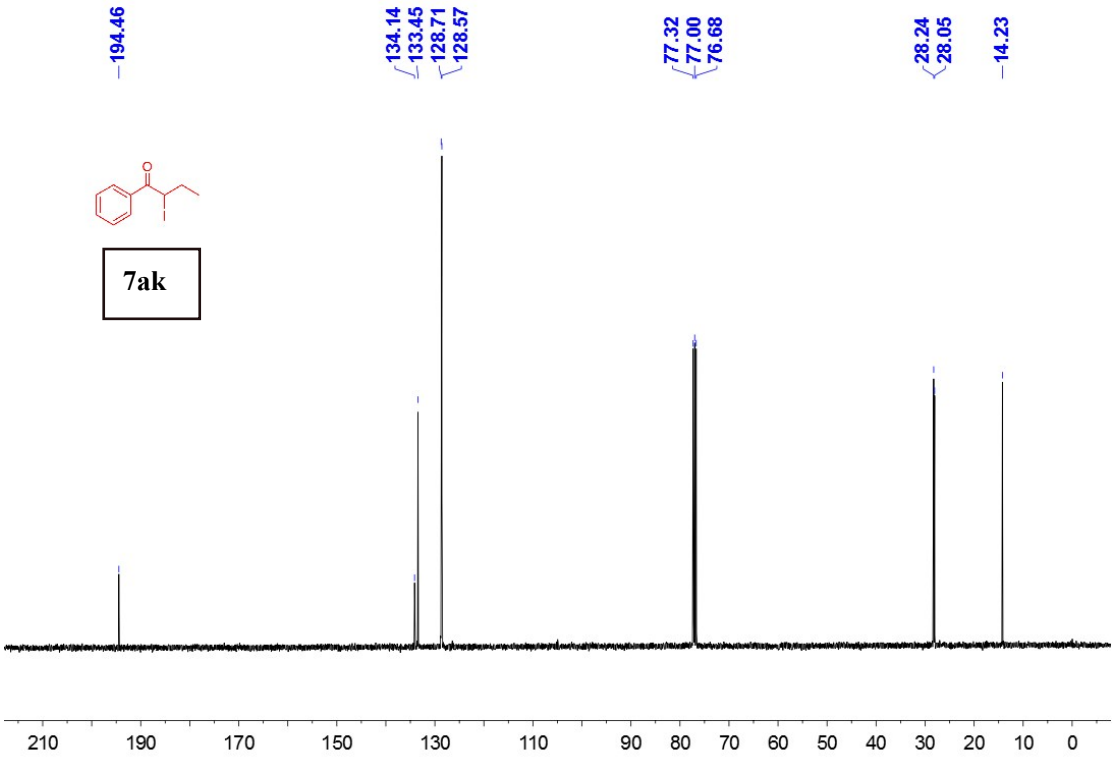
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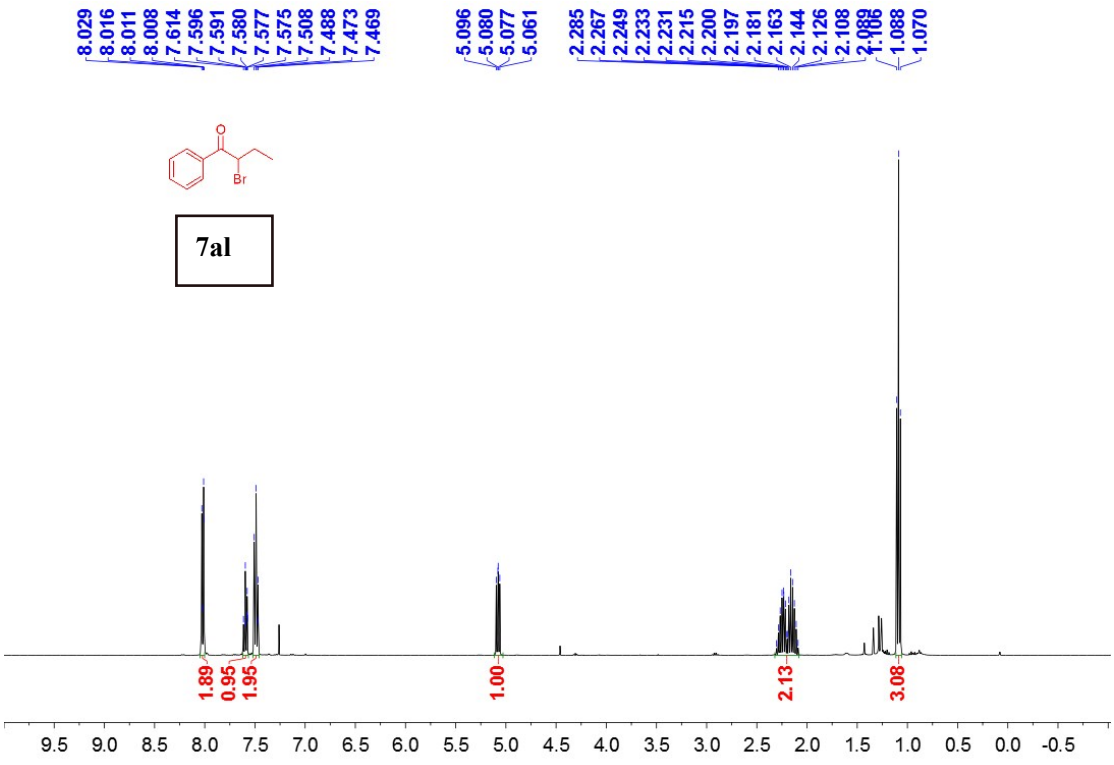
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Br
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2.13
3.08
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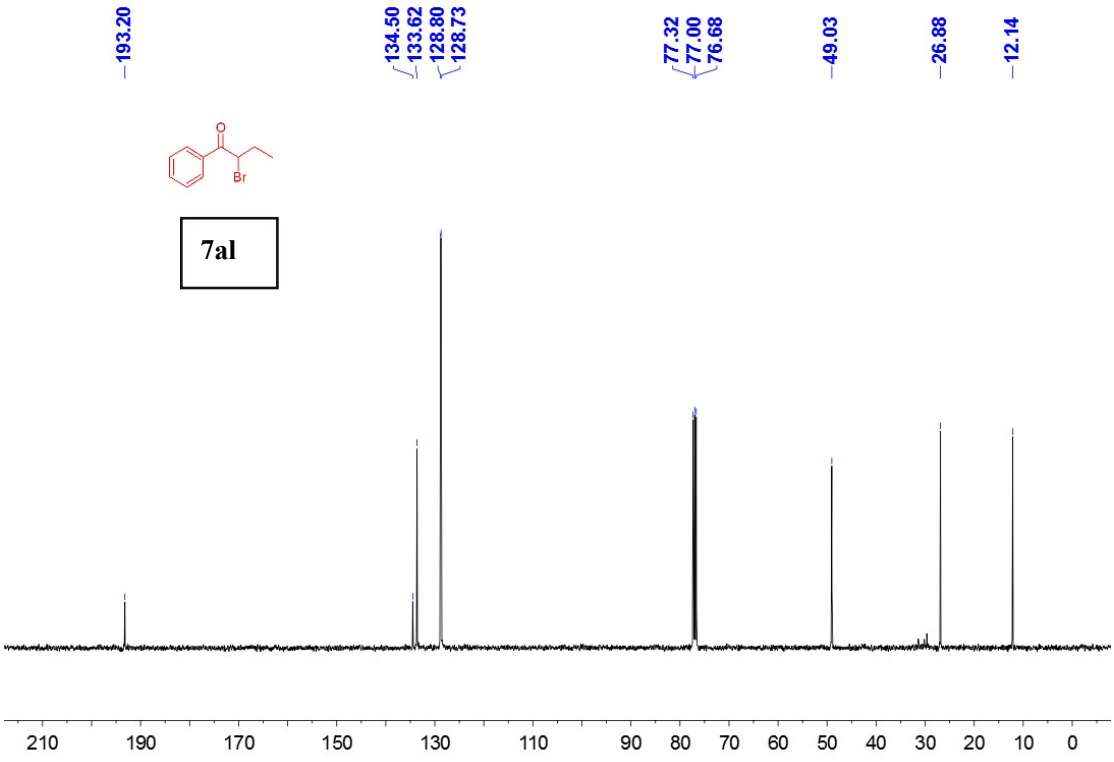
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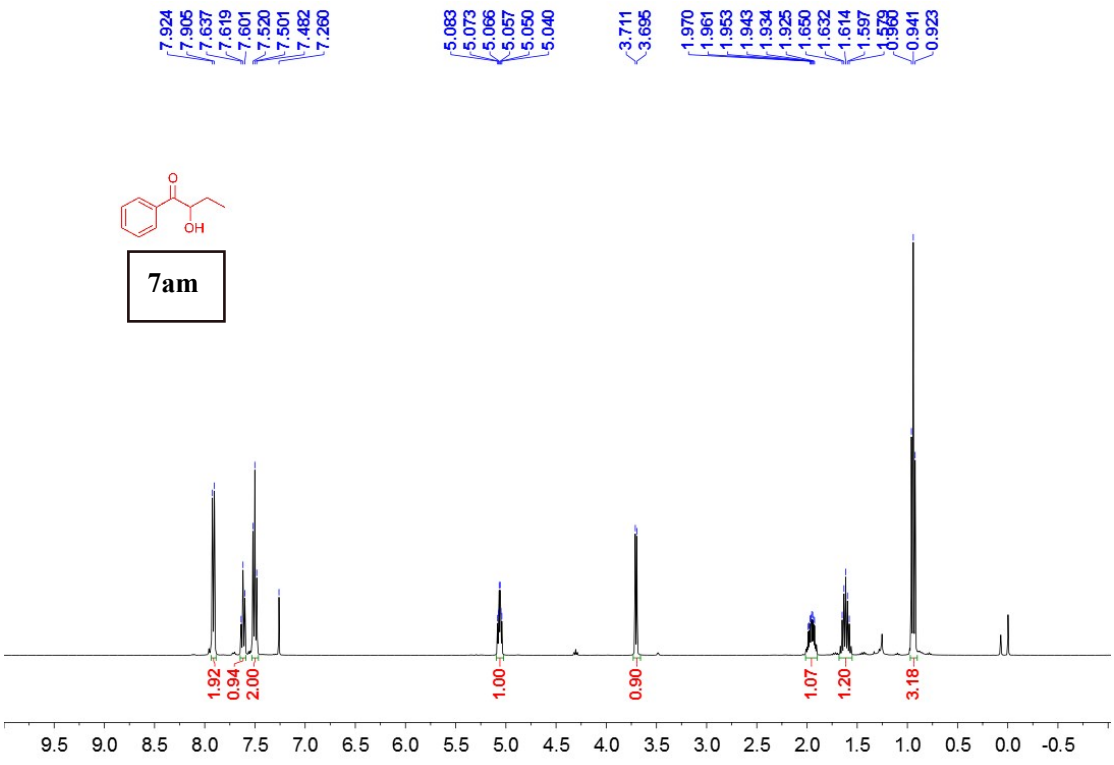
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OH
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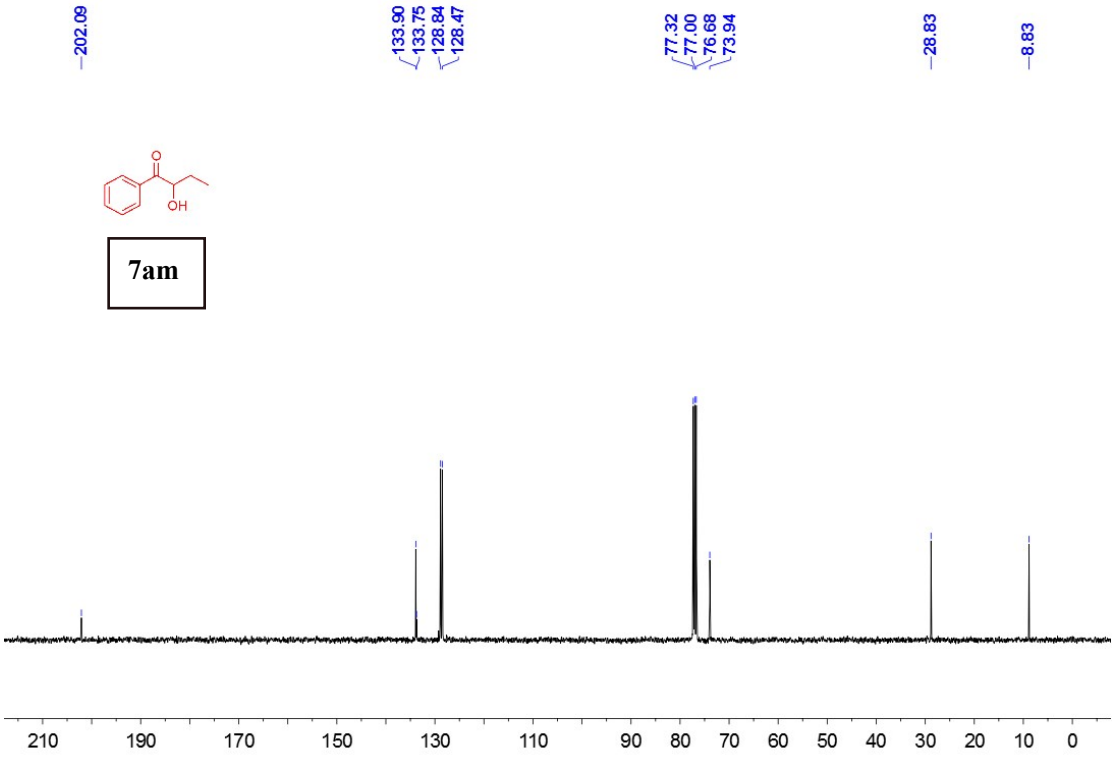
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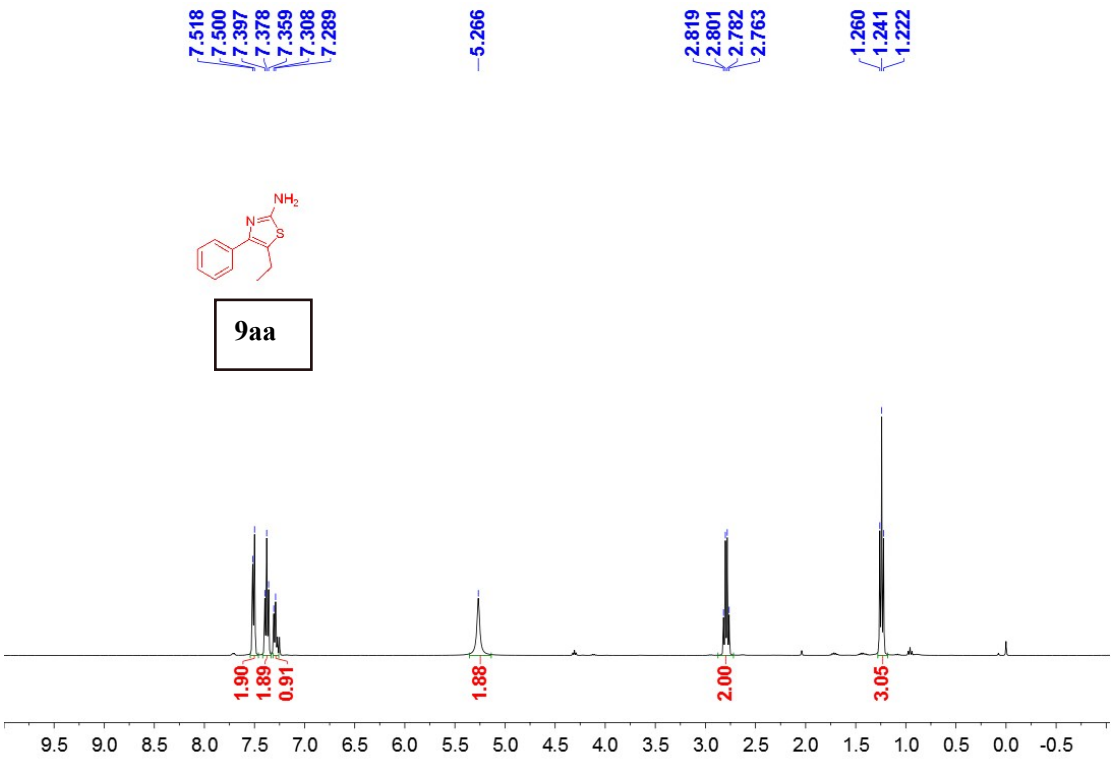
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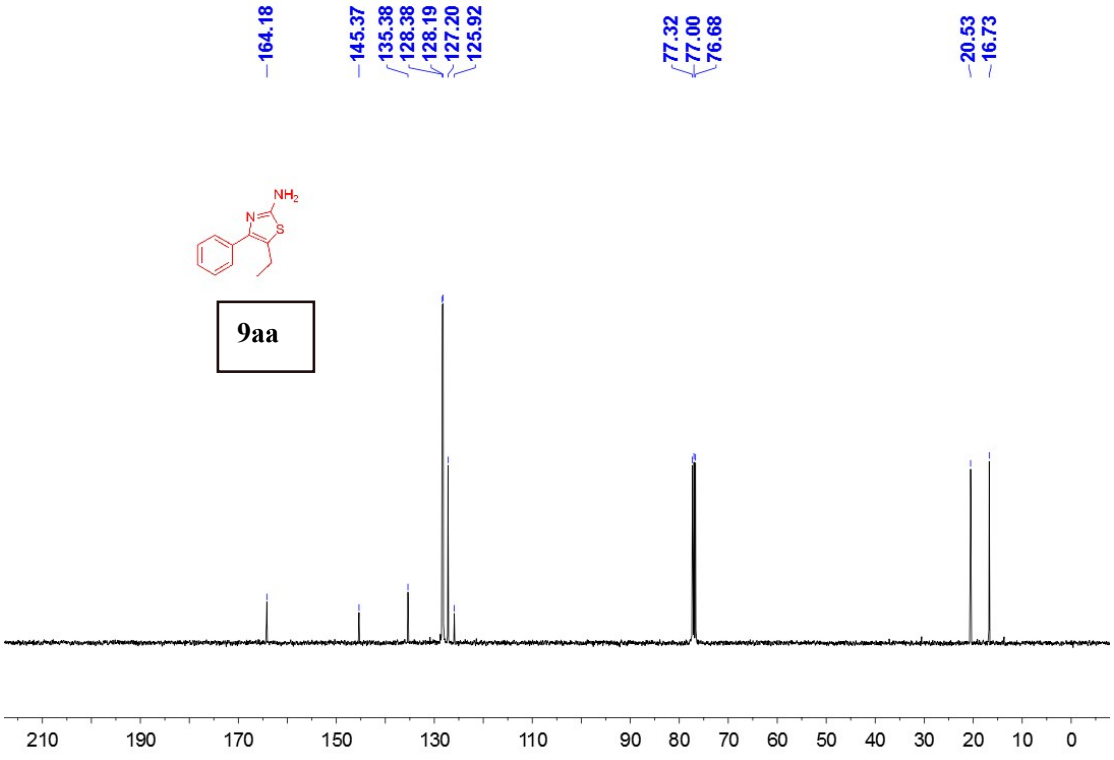
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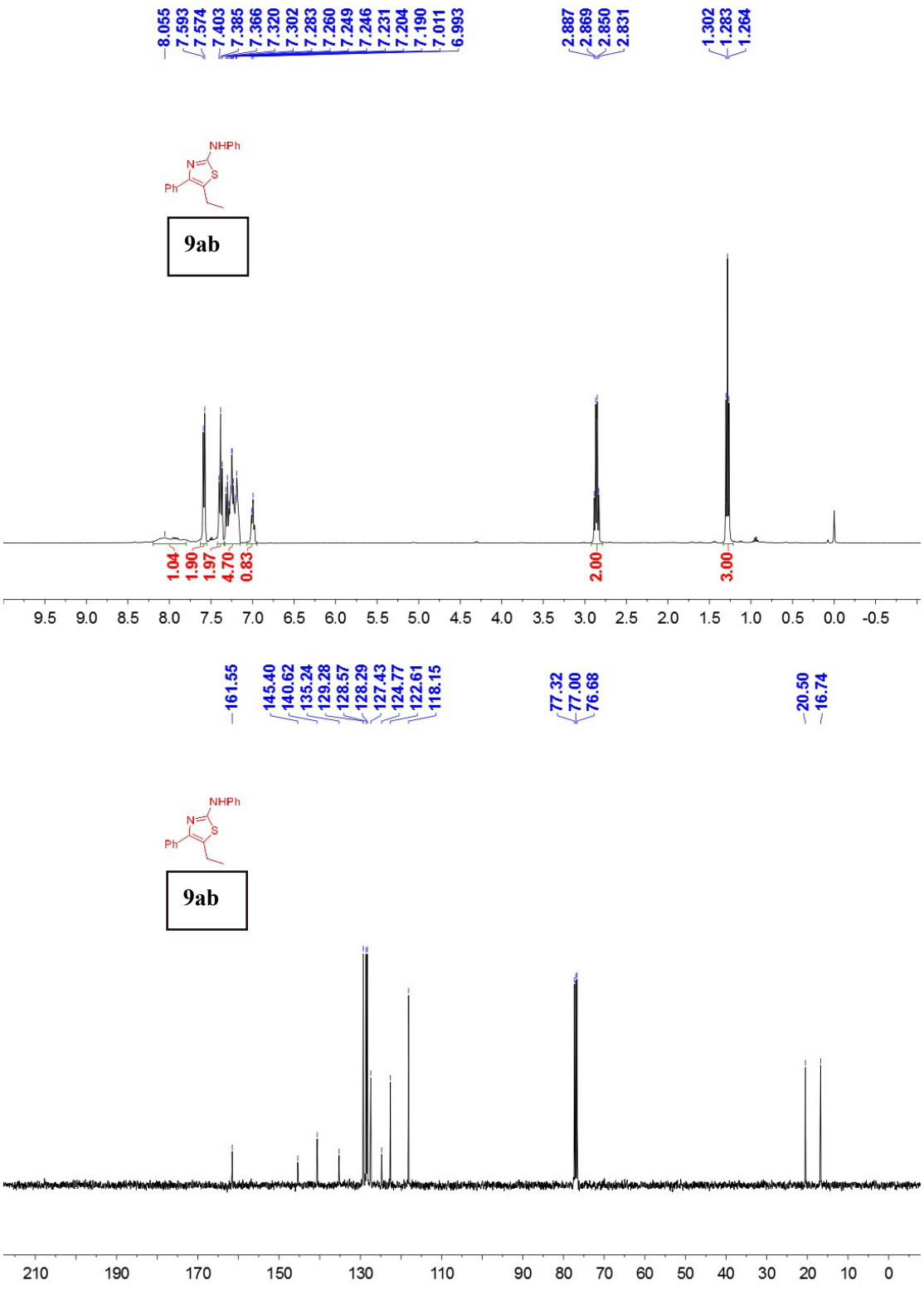
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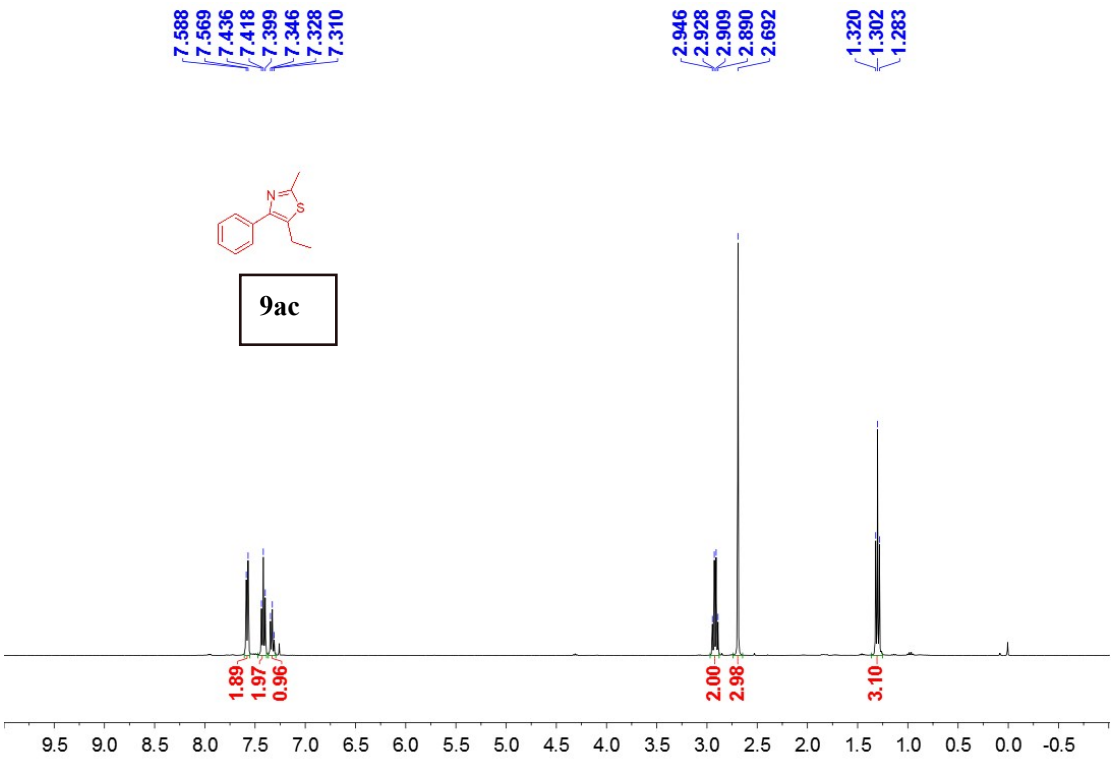
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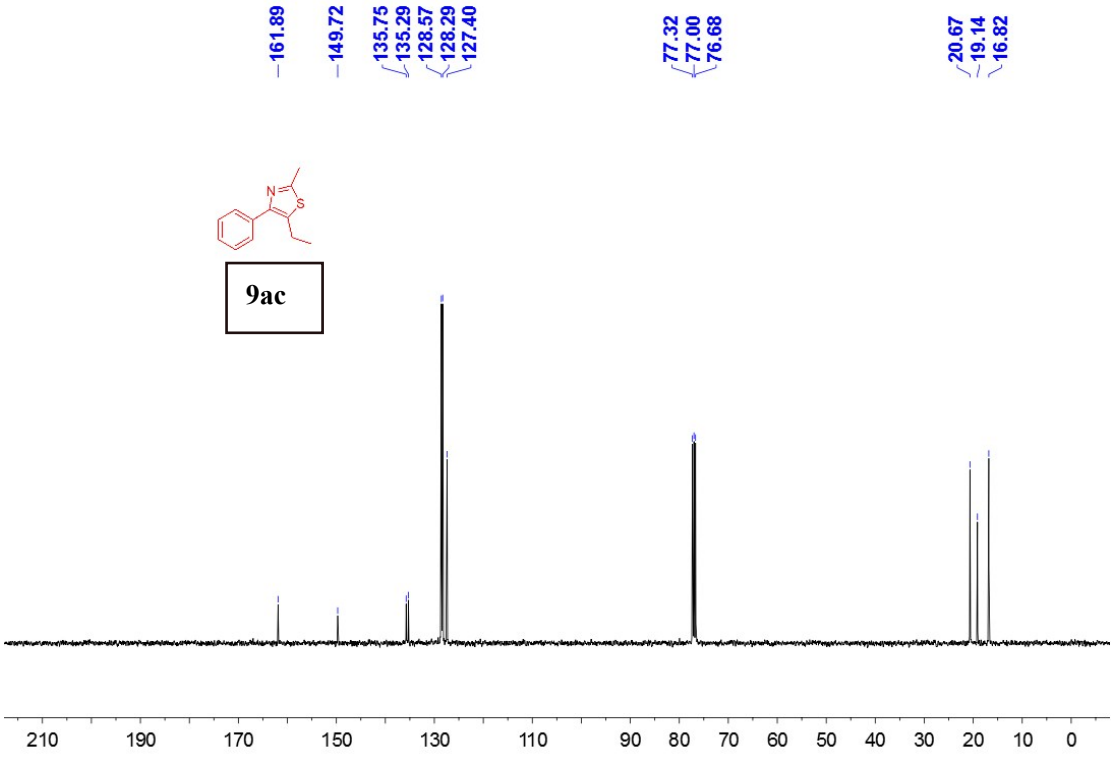
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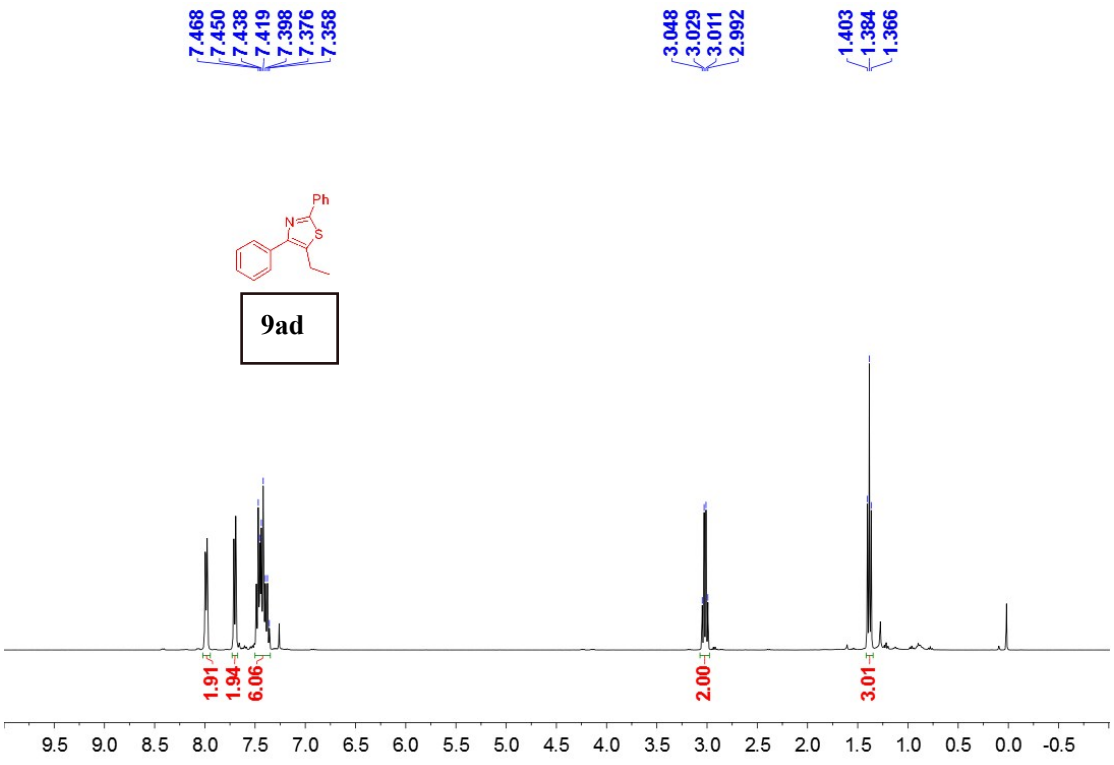
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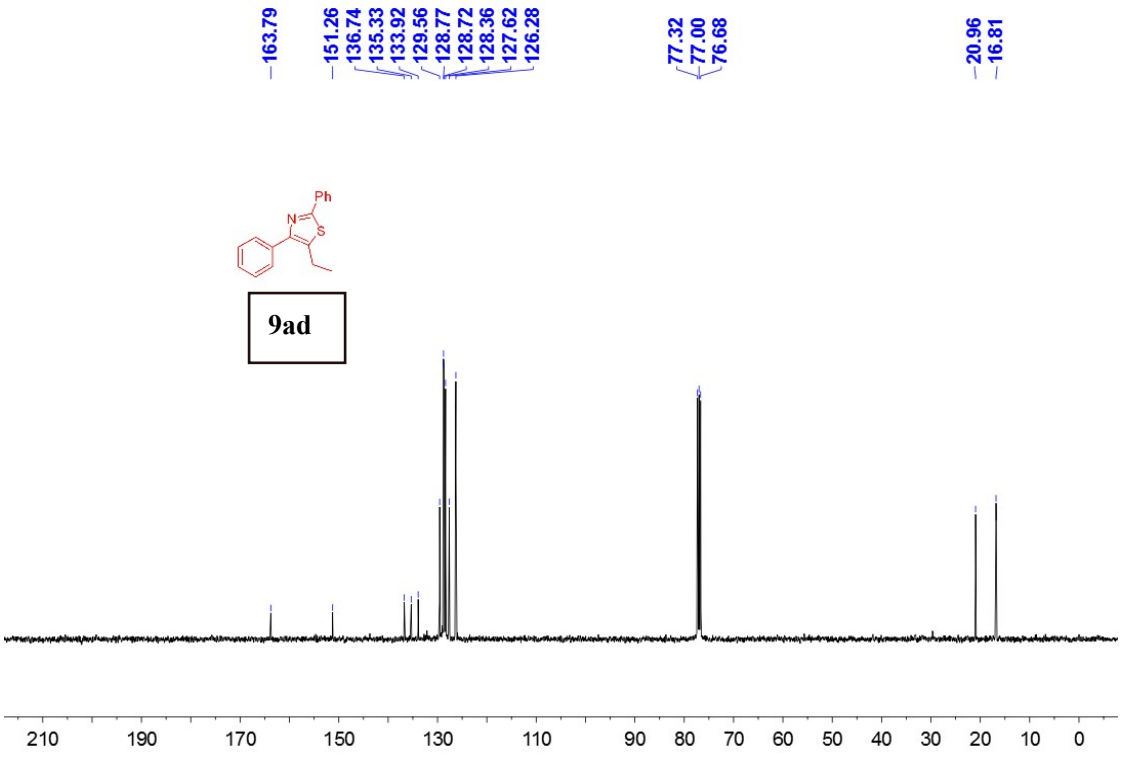
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S
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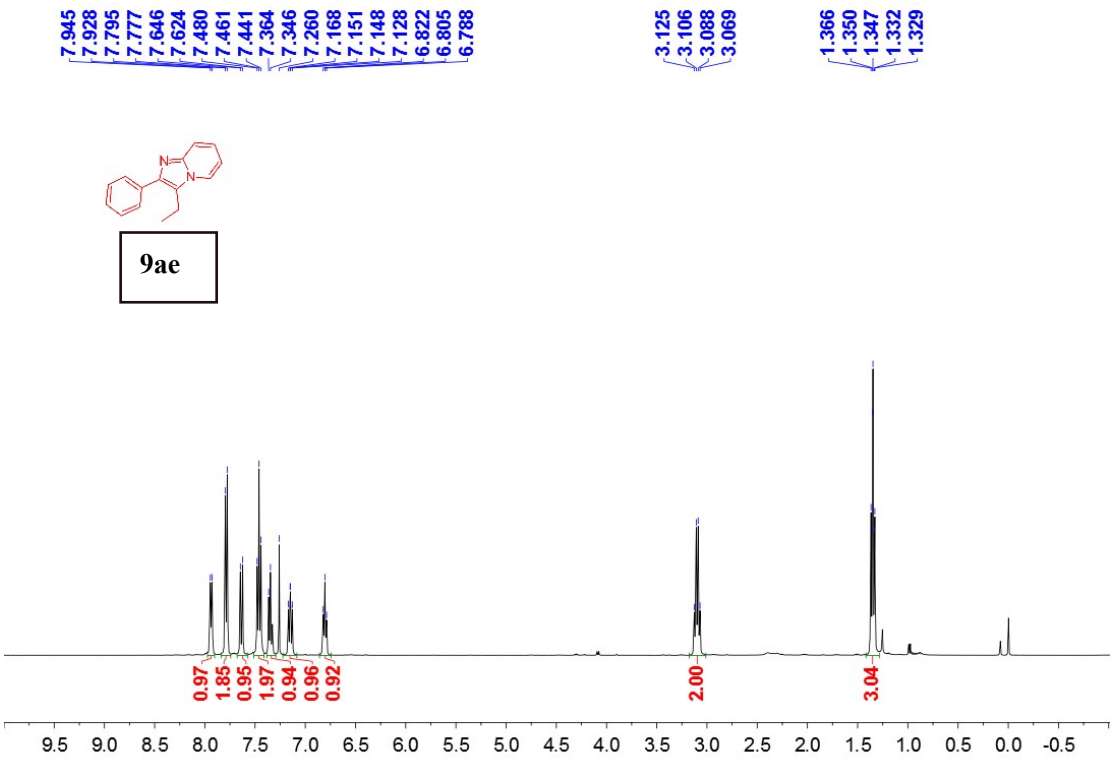
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7.128
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6.805
6.788
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3.106
3.088
3.069
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1.347
1.332
1.329
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1.85
0.95
1.97
0.94
0.96
0.92
2.00
3.04
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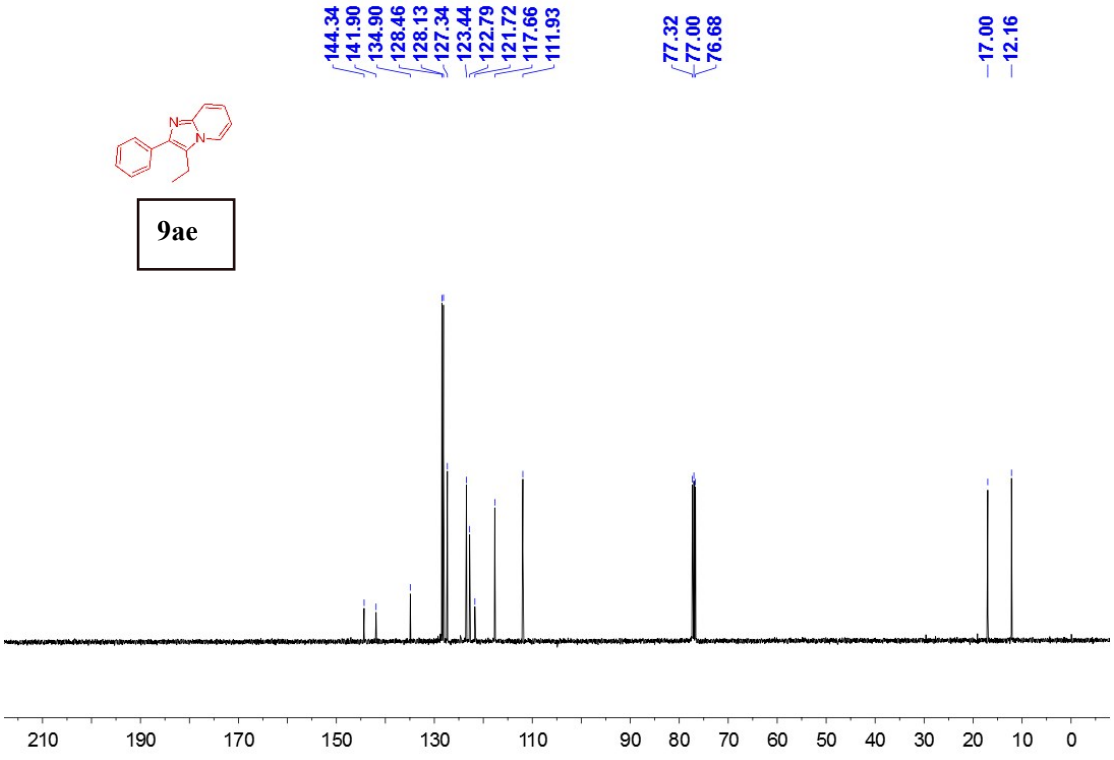
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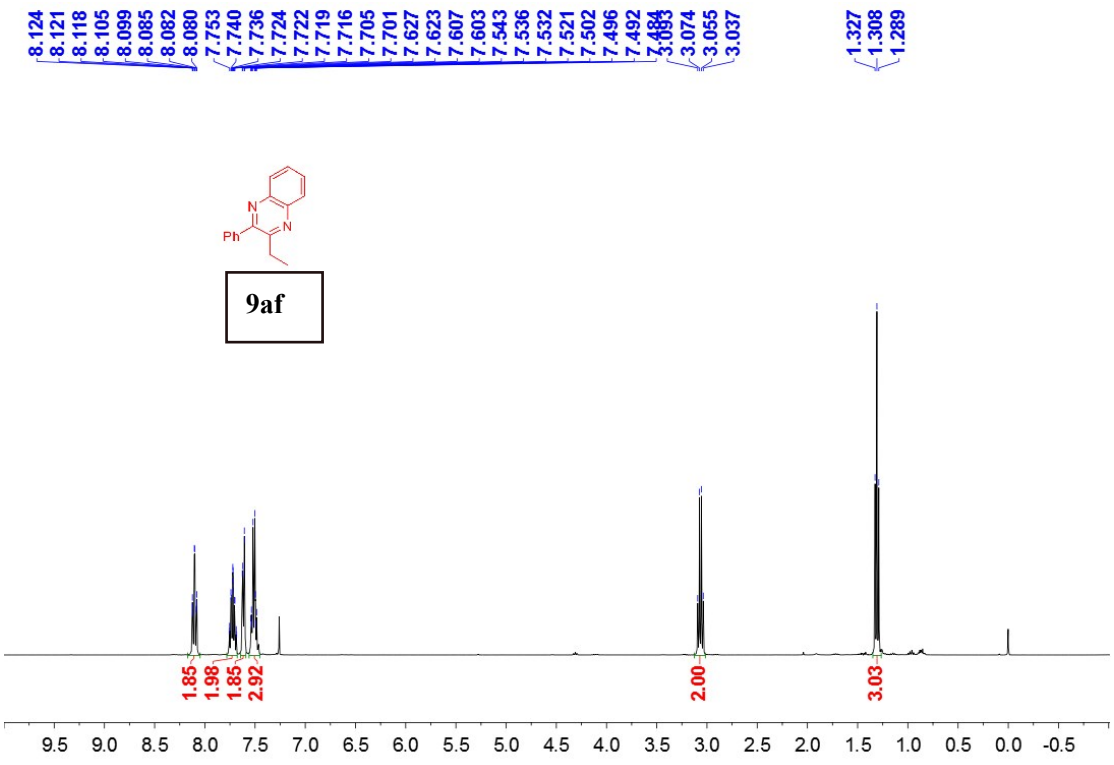
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7.532
7.521
7.502
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3.037
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1.289
Ph
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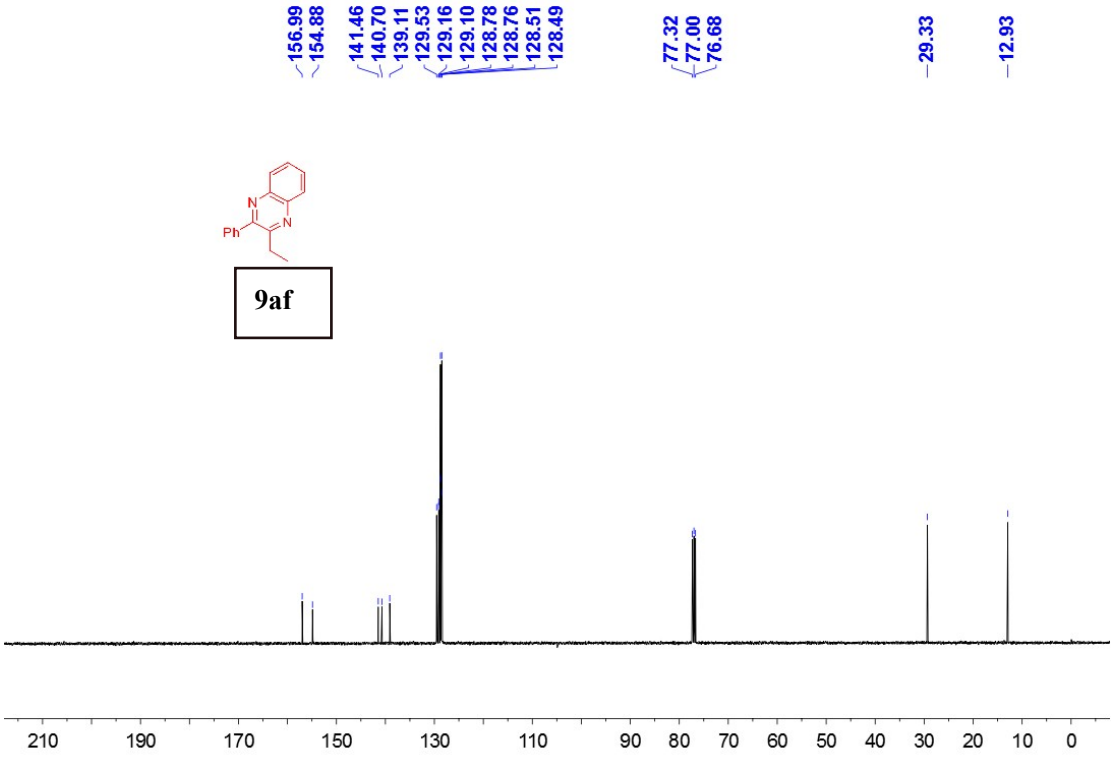
1.85
1.98
1.85
2.92
2.00
3.03
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5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Ph
9af
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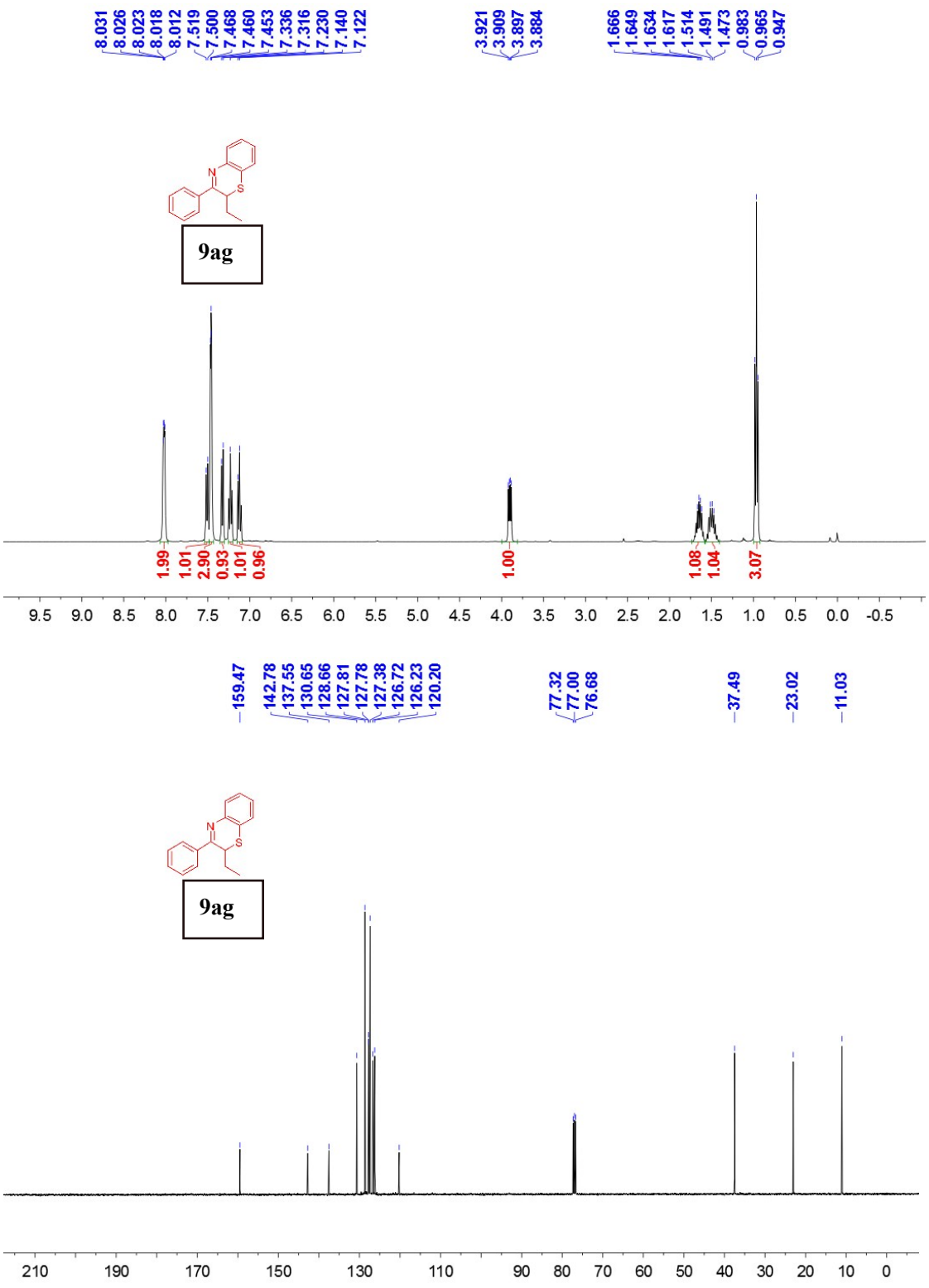
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3.909
3.897
3.884
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1.649
1.634
1.617
1.514
1.491
1.473
0.983
0.965
0.947
9ag
1.99
1.01
2.90
0.93
1.01
0.96
1.00
1.08
1.04
3.07
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
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137.55
130.65
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127.78
127.38
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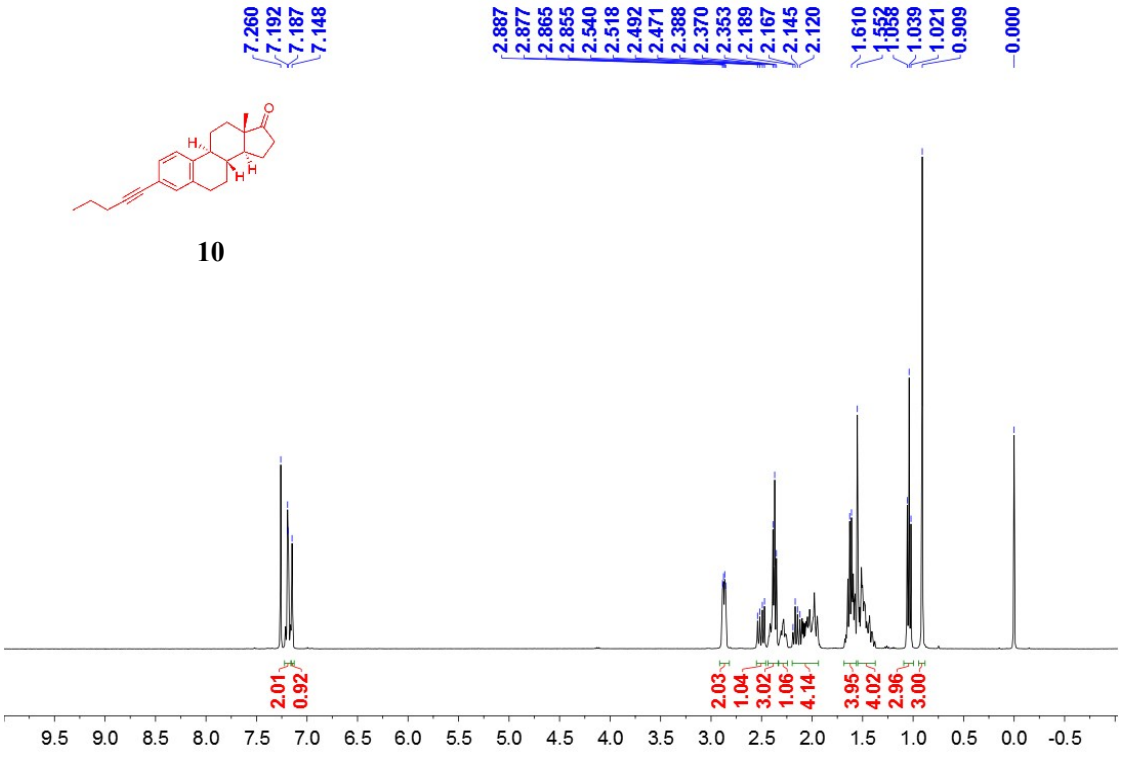
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2.855
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2.518
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2.471
2.388
2.370
2.353
2.189
2.167
2.145
2.120
1.610
1.552
1.039
1.021
0.909
0.000
2.01
0.92
2.03
1.04
3.02
1.06
4.14
3.95
4.02
2.96
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5

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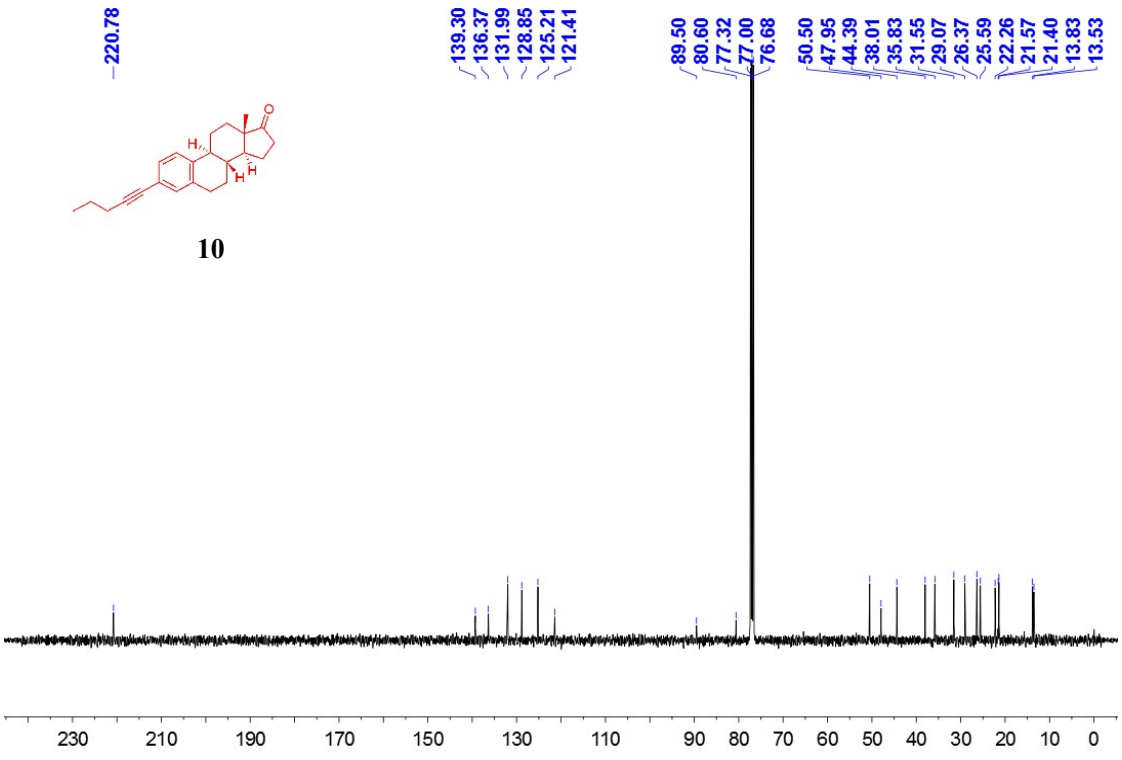
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47.95
44.39
38.01
35.83
31.55
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13.83
13.53
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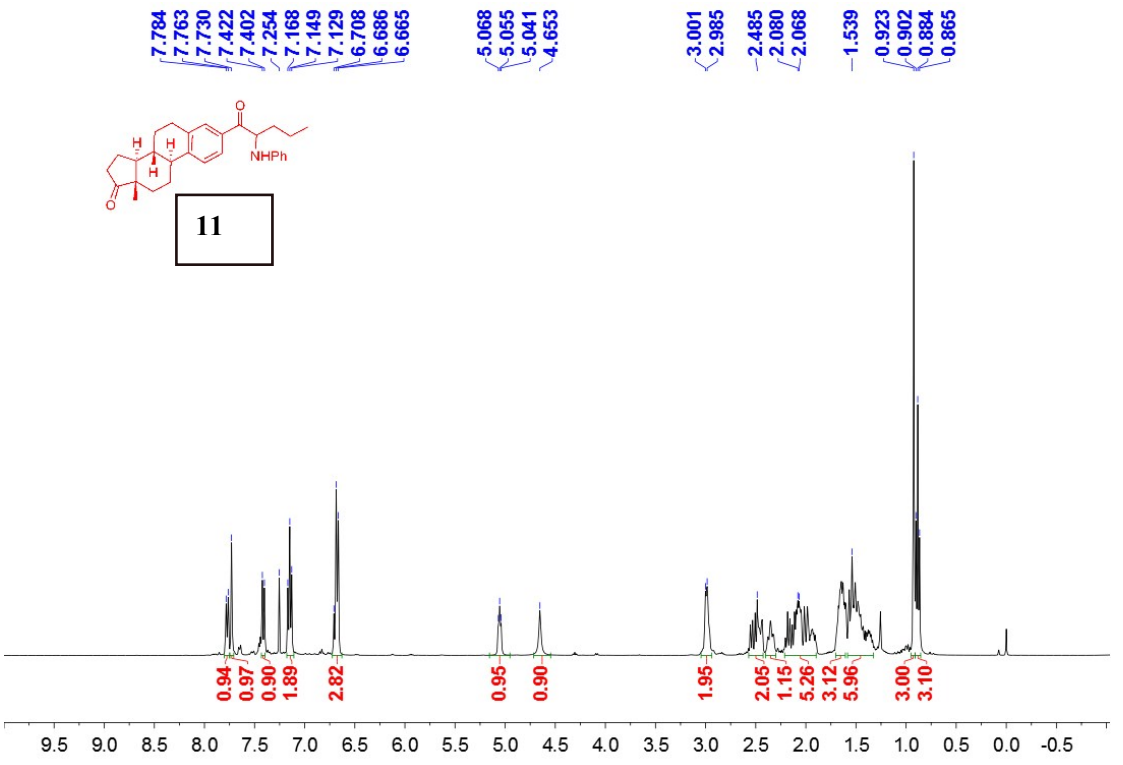
10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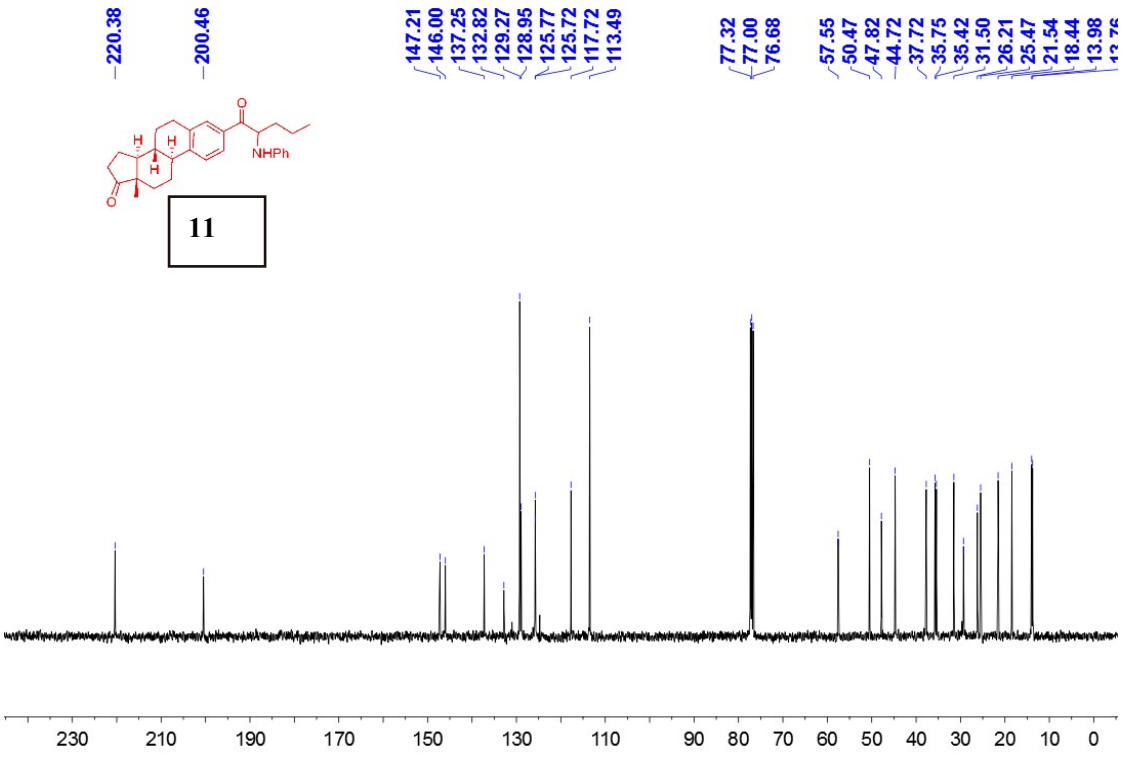
5.068
5.055
5.041
4.653
3.001
2.985
2.485
2.080
2.068
1.539
0.923
0.902
0.884
0.865
NHPh
11
0.94
0.97
0.90
1.89
2.82
0.95
0.90
1.95
2.05
1.15
5.26
3.12
5.96
3.00
3.10
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5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77.32
77.00
76.68
57.55
50.47
47.82
44.72
37.72
35.75
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25.47
21.54
18.44
13.98
NHPh
11
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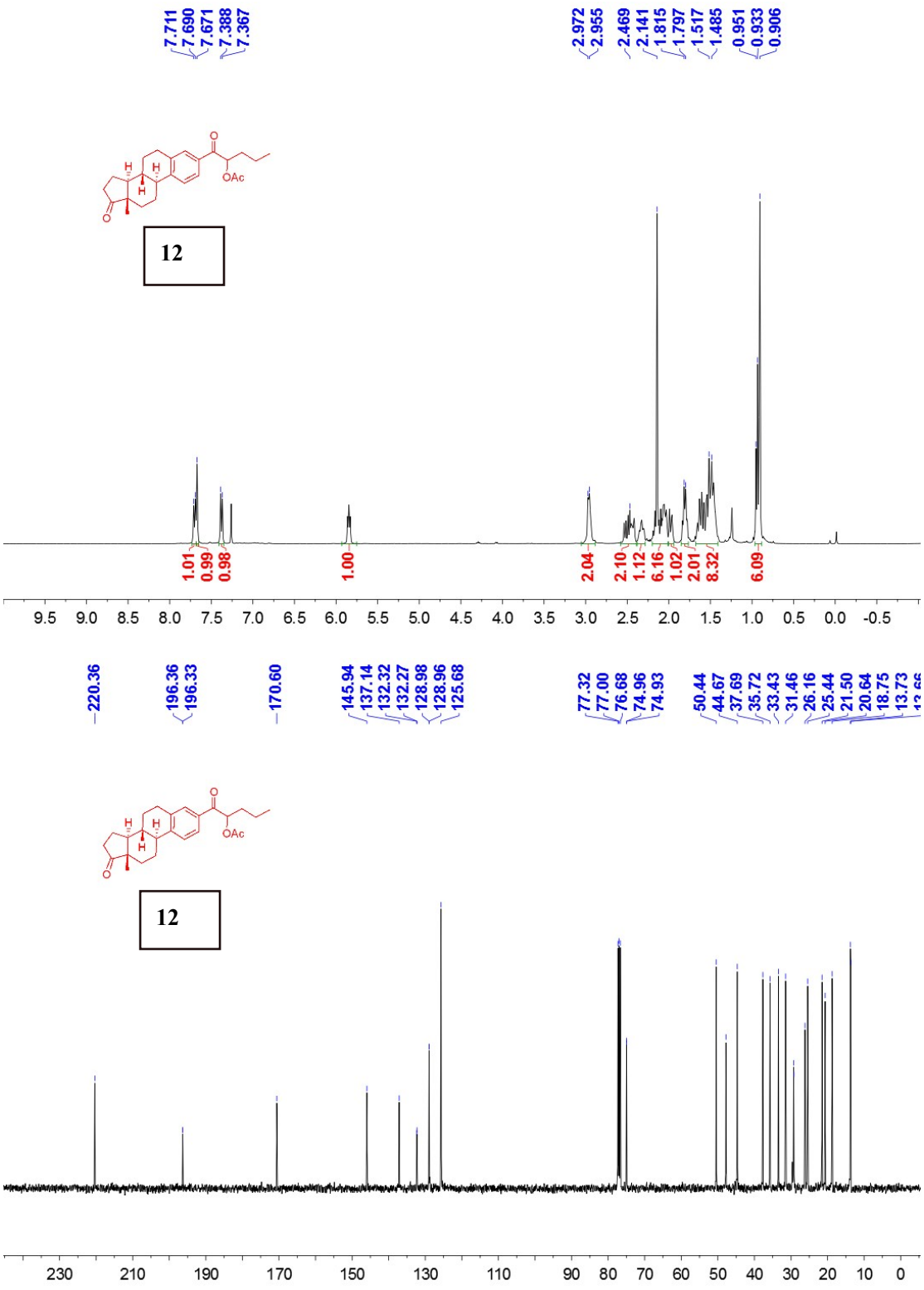
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0.951
0.933
0.906
OAc
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1.01
0.99
0.98
1.00
2.04
2.10
1.12
6.16
1.02
2.01
8.32
6.09
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
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196.36
196.33
170.60
145.94
137.14
132.32
132.27
128.98
128.96
125.68
77.32
77.00
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74.93
50.44
44.67
37.69
35.72
33.43
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OAc
12
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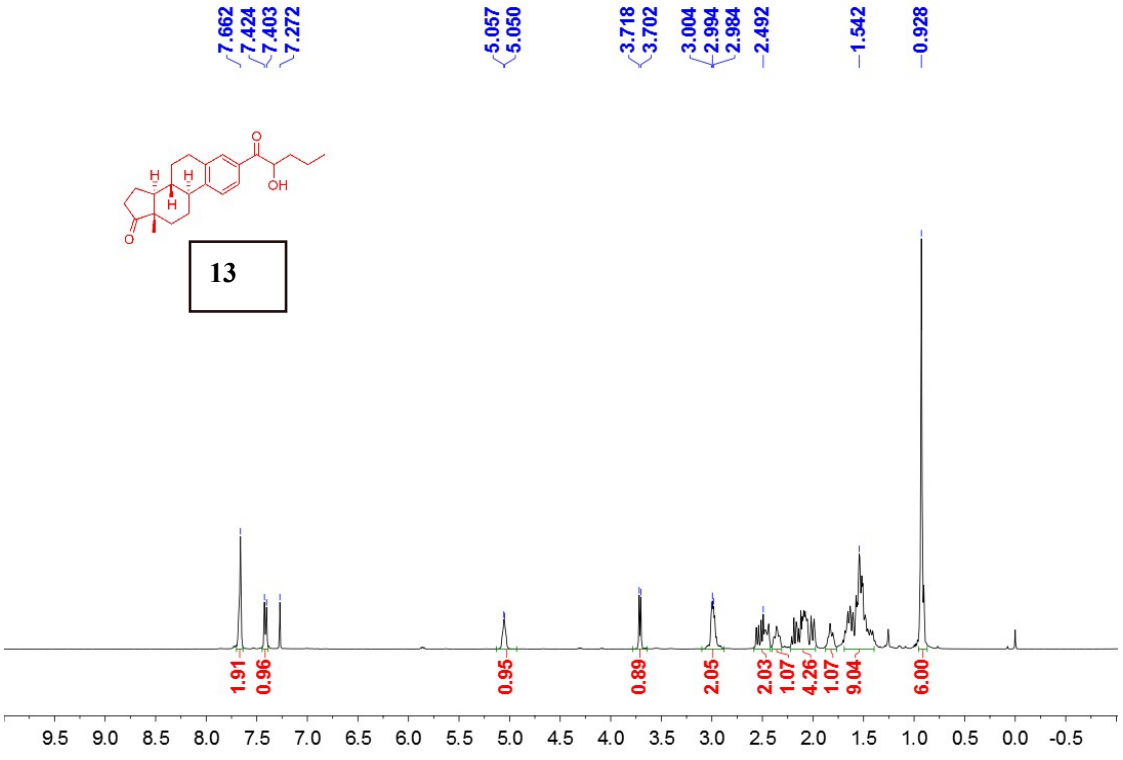
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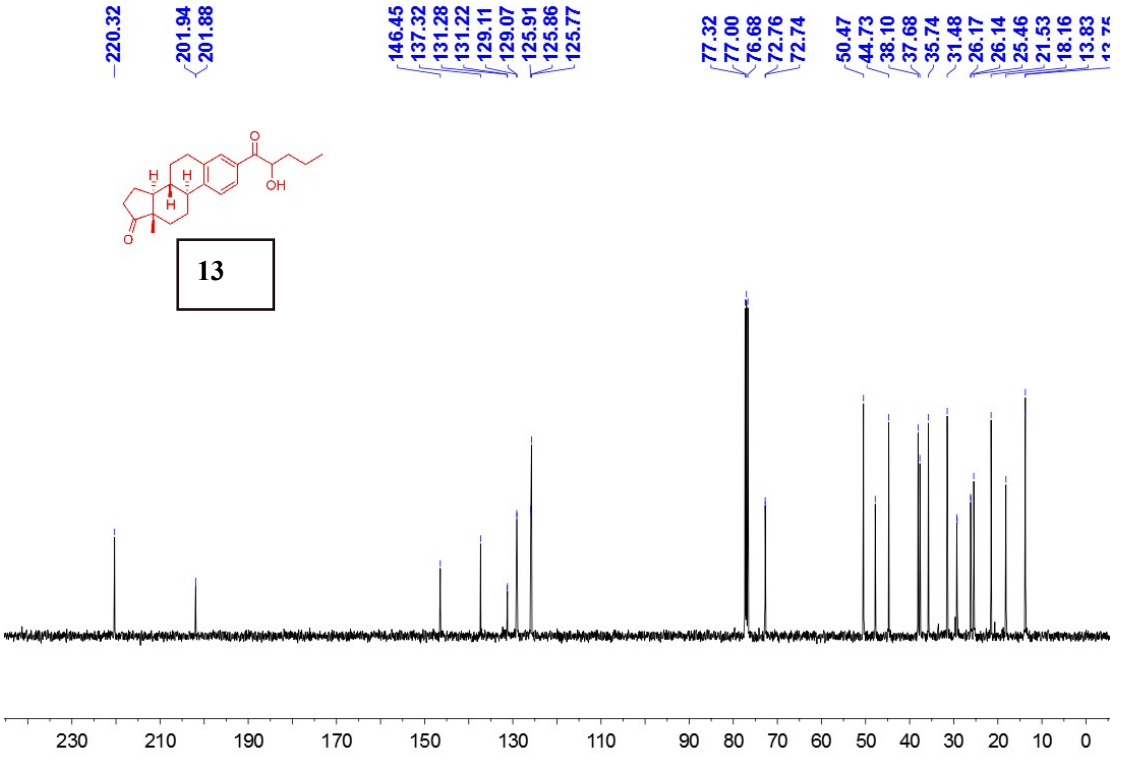
13
1.91
0.96
0.95
0.89
2.05
2.03
1.07
4.26
1.07
9.04
6.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5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125.86
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77.00
76.68
72.76
72.74
50.47
44.73
38.10
37.68
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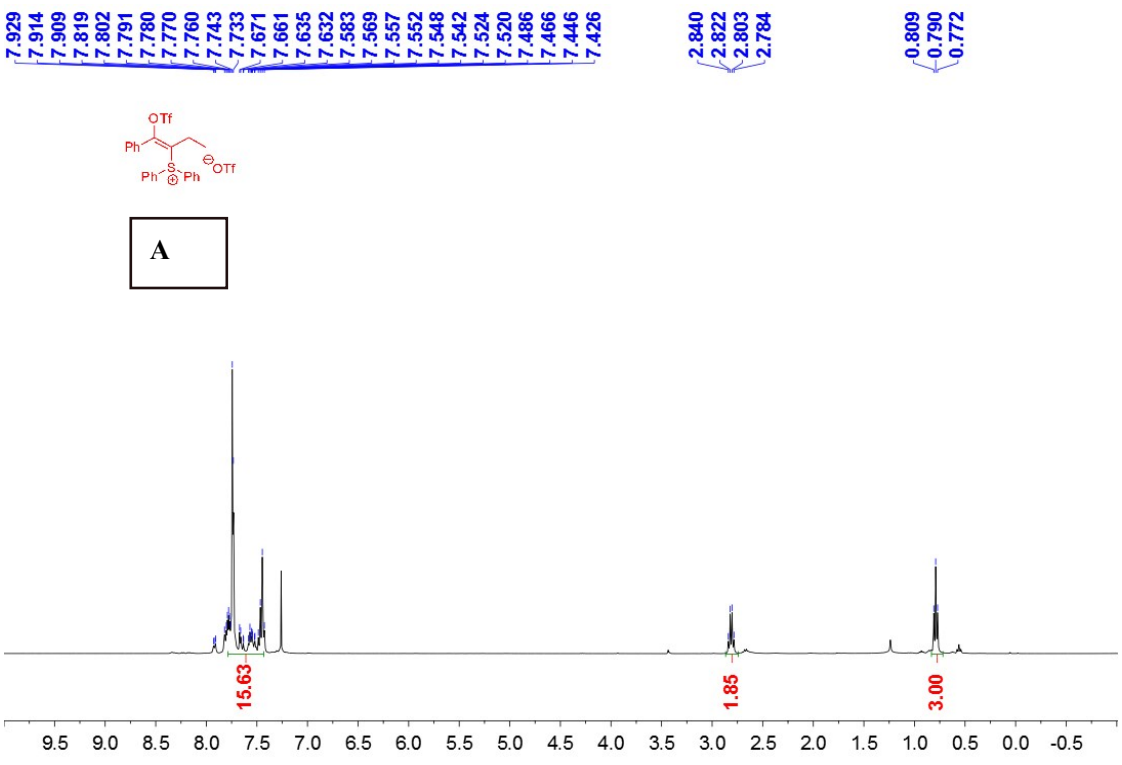
26.14
25.46
21.53
18.16
13.83
13
230 210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0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Ph
Ph
S
Ph
OTf
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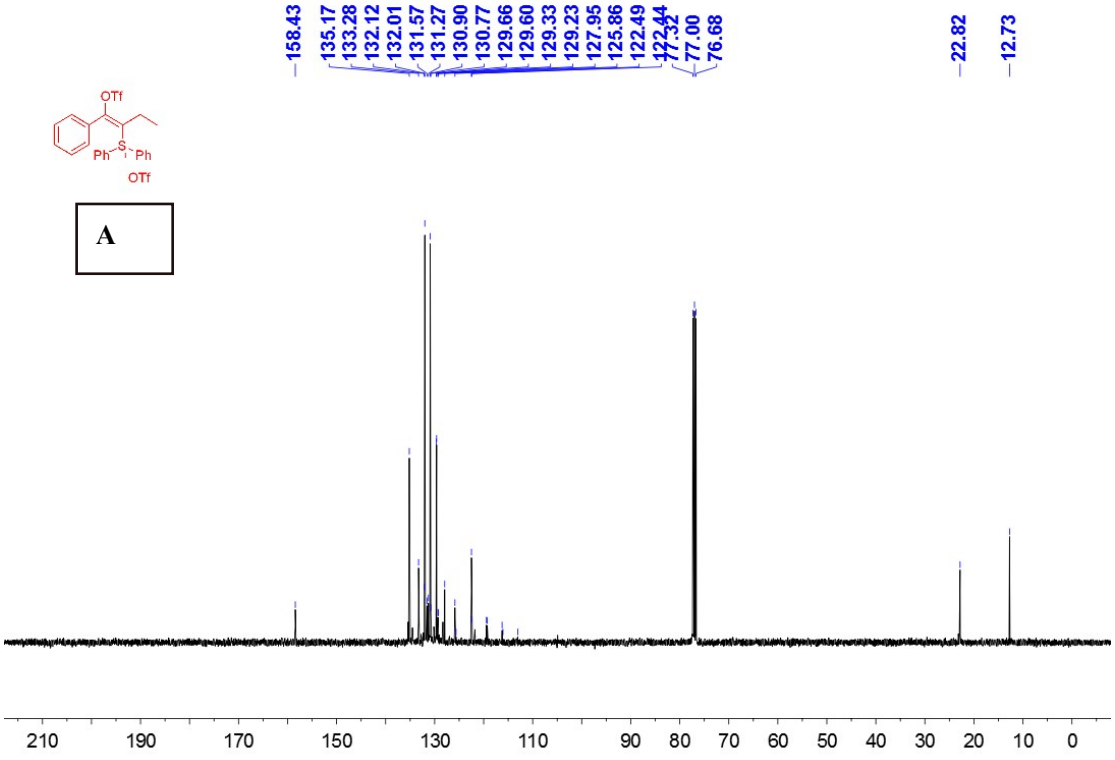
0.0
-0.5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OTf
Ph
S
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10
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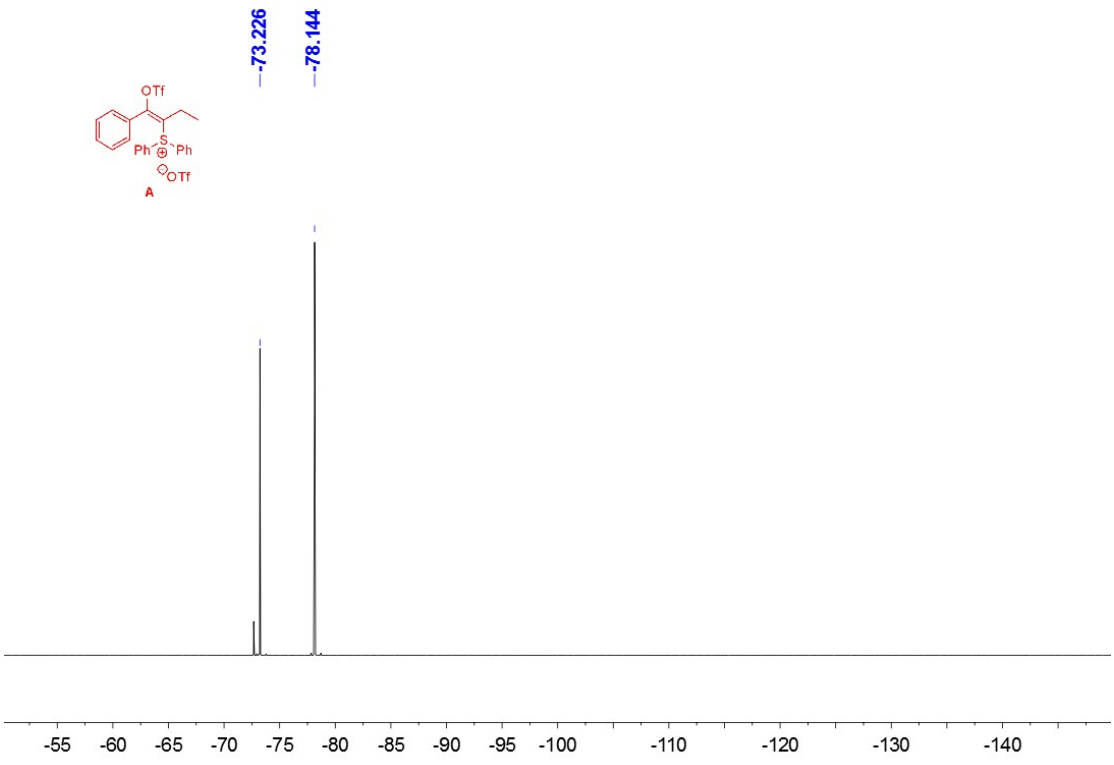
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Ph
OTf
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-90
-95
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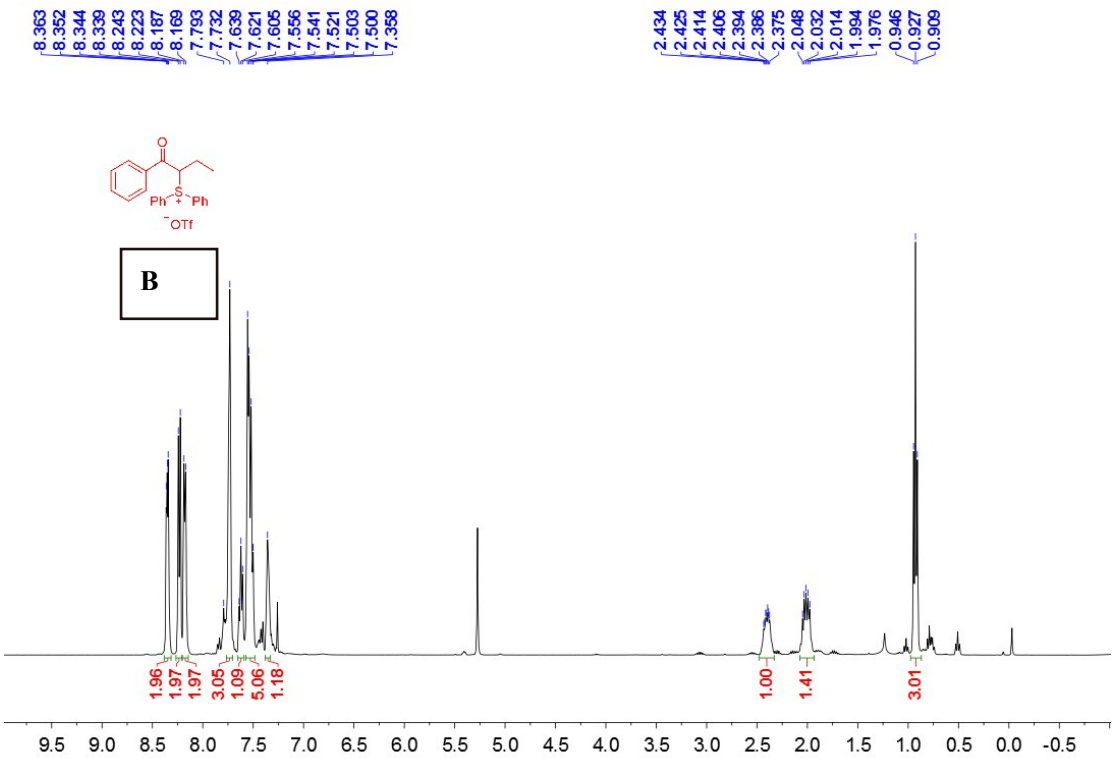
-110
-120
-130
-140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2.032
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0.927
0.909
B
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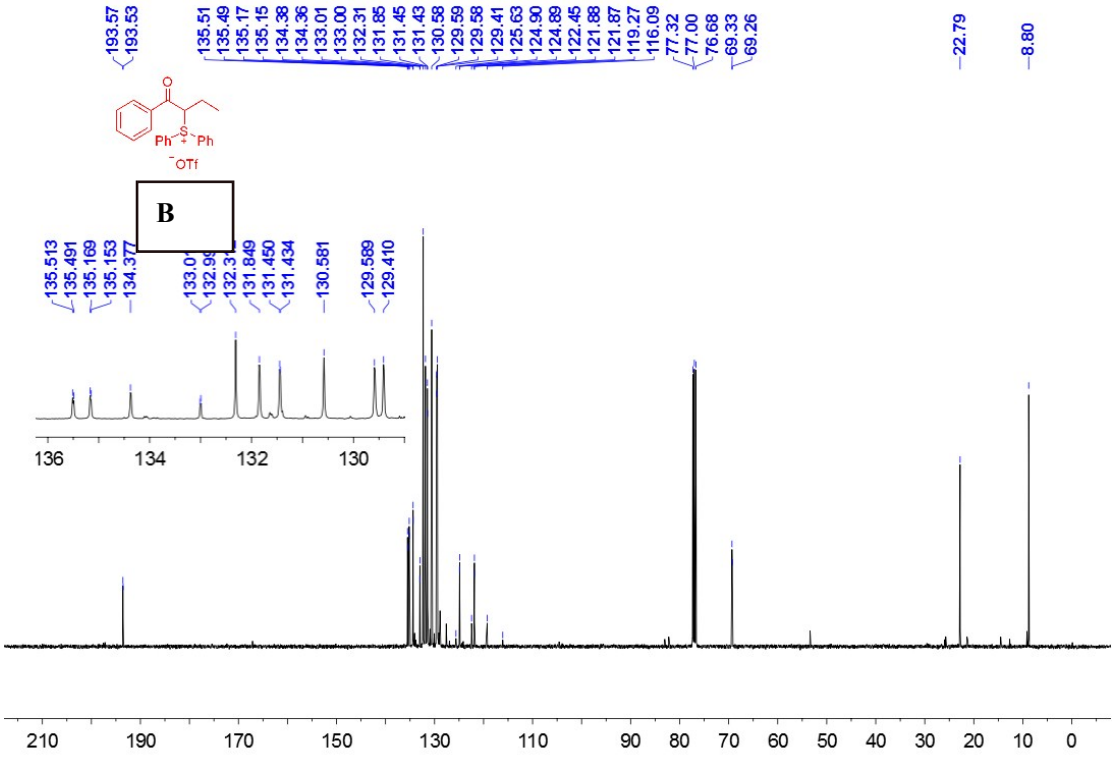
1.97
1.97
3.05
1.09
5.06
1.18
1.00
1.41
3.01
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5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129.58
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B
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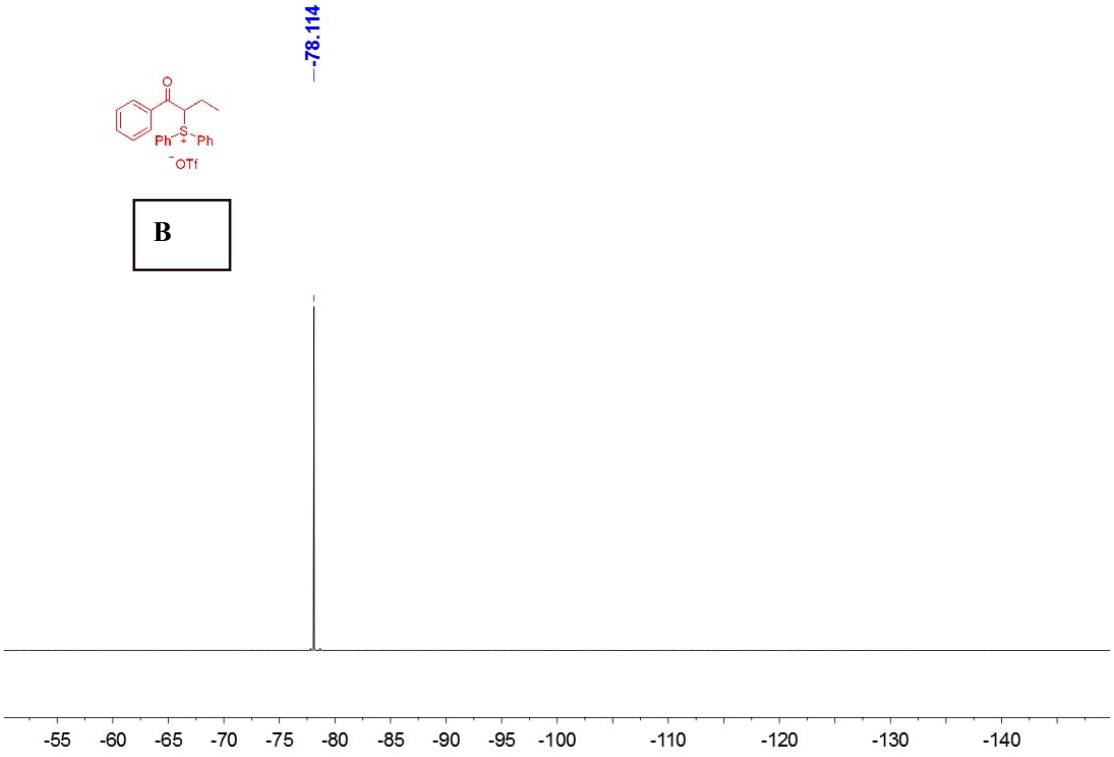
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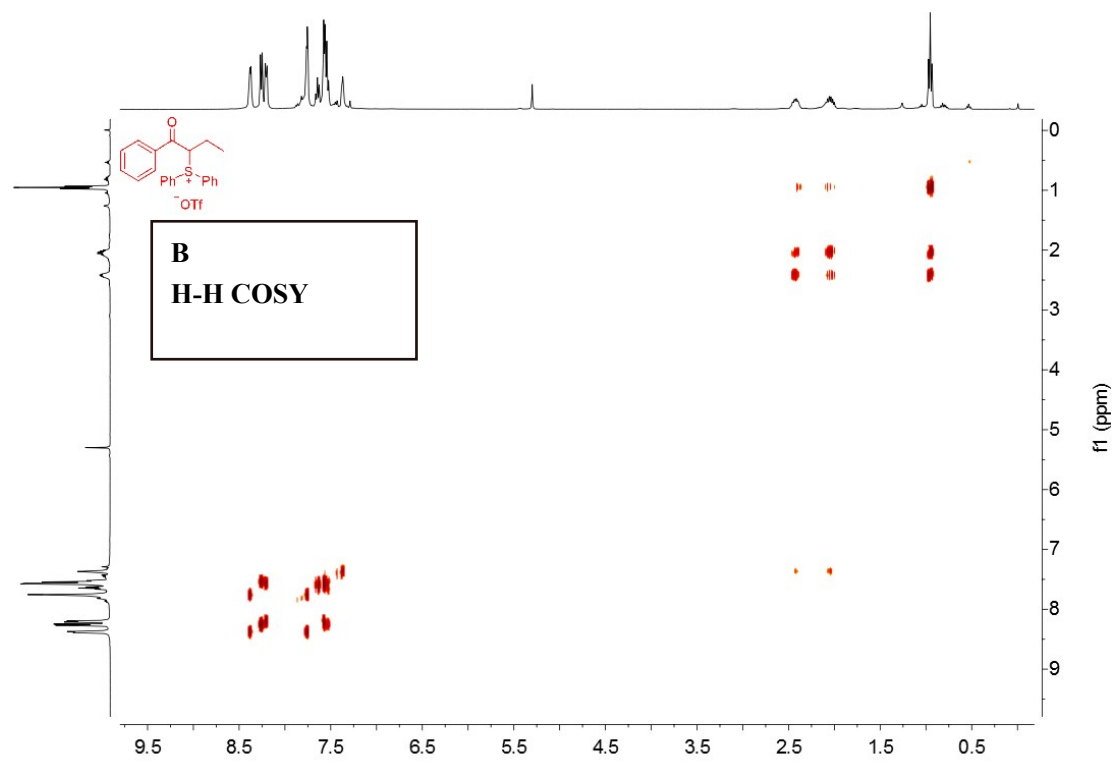
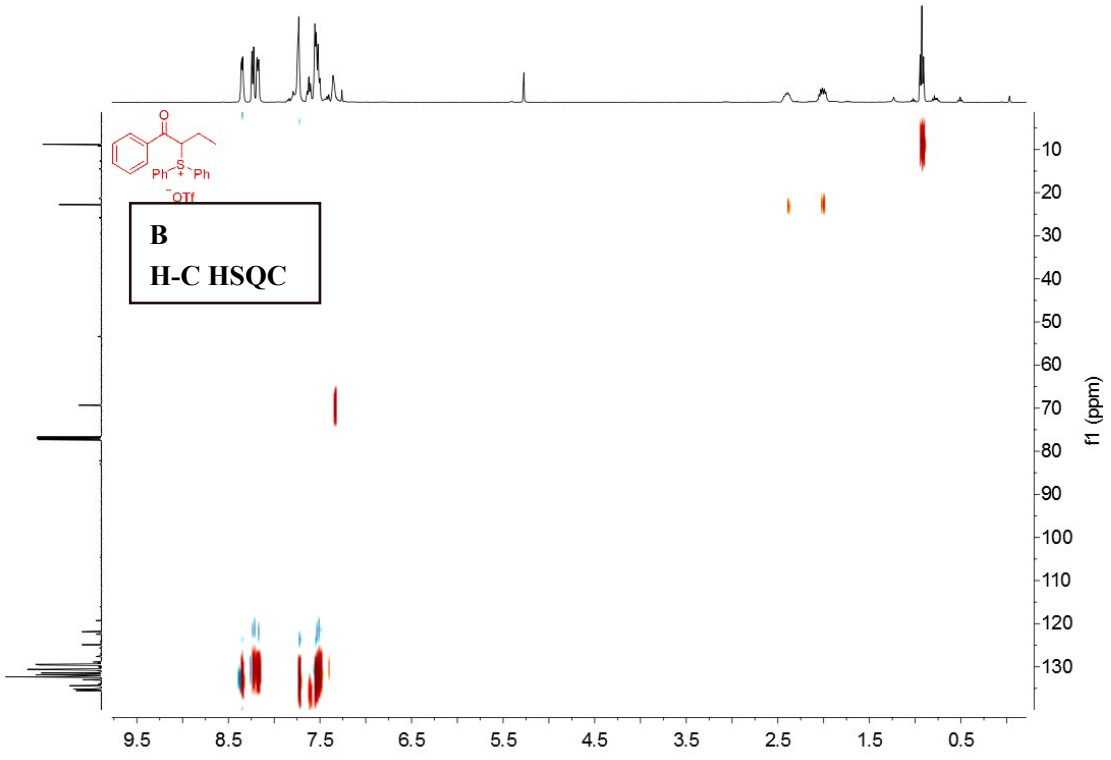
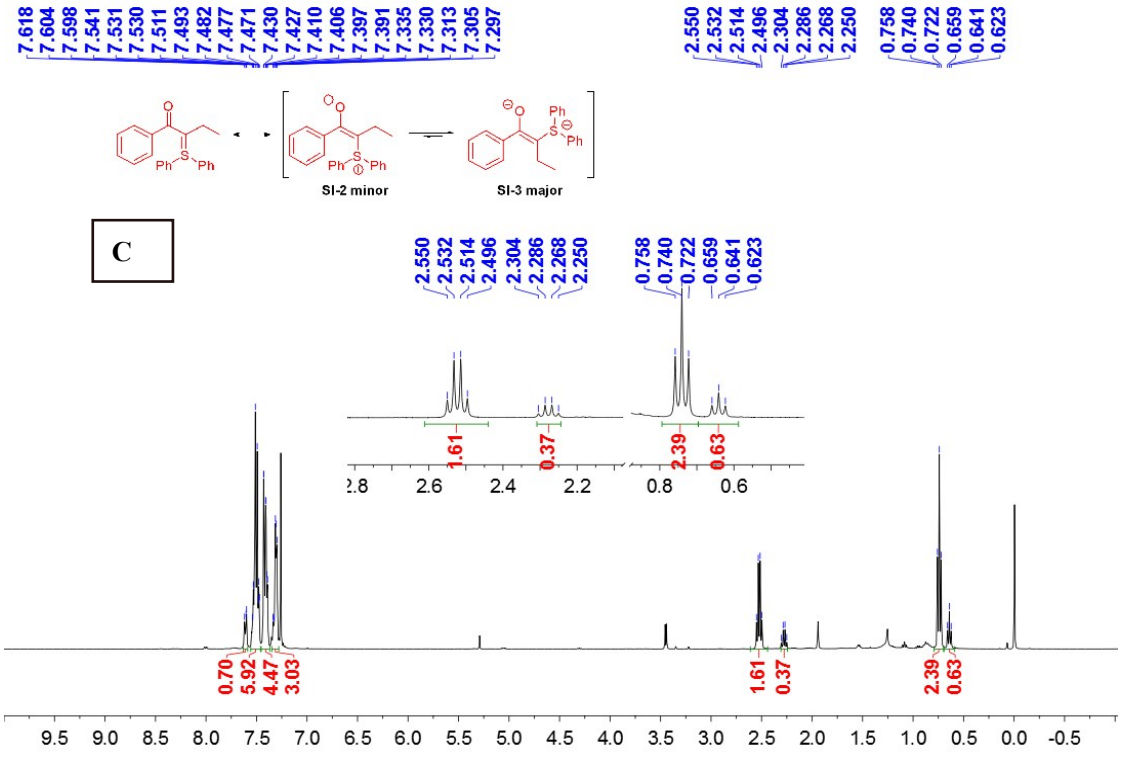
B
-55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -110 -120 -130 -140


B
H-H COSY
9.5 8.5 7.5 6.5 5.5 4.5 3.5 2.5 1.5 0.5
0 1 2 3 4 5 6 7 8 9
f1 (ppm)


B
H-C HSQC
f1 (ppm)
10
20
30
40
50
60
70
80
90
100
110
120
130
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5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7.297
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2.496
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2.286
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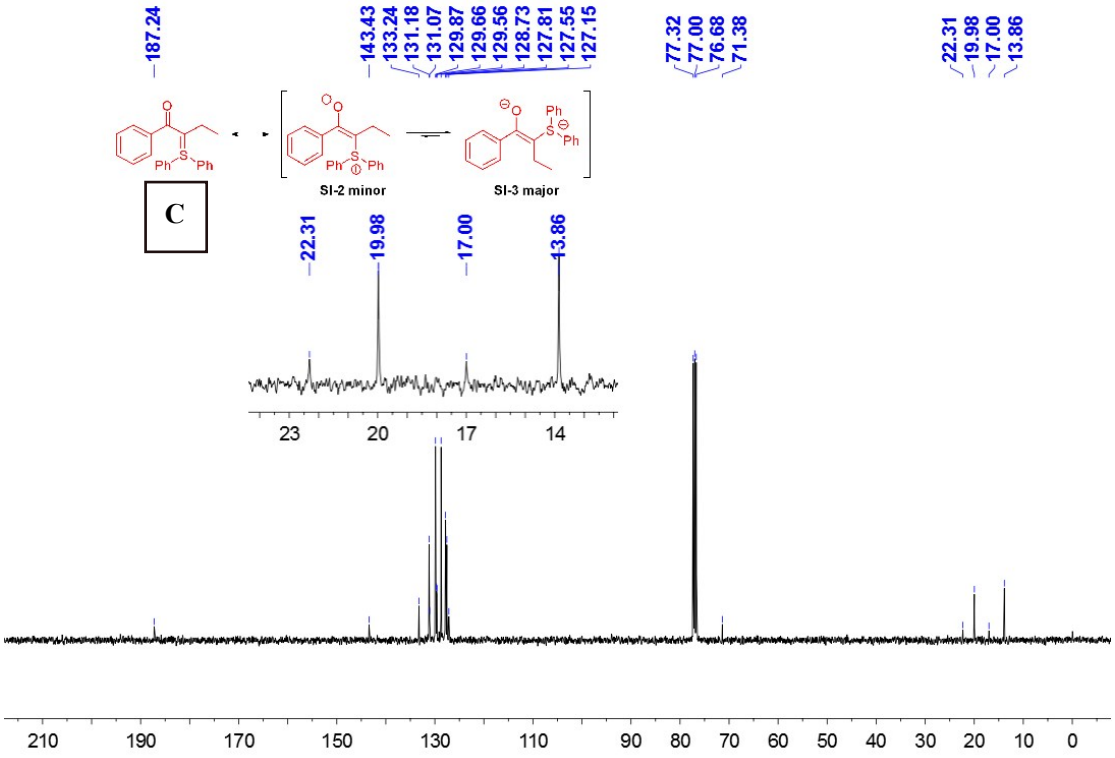
0.740
0.722
0.659
0.641
0.623
SI-2 minor
SI-3 major
C
1.61
0.37
2.39
0.63
0.70
5.92
4.47
3.03
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5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77.00
76.68
71.38
22.31
19.98
17.00
13.86
SI-2 minor
SI-3 major
C
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0