Supporting Information

Metal-free synthesis of ketones by visible-light induced aerobic oxidative radical addition of aryl hydrazines to alkenes

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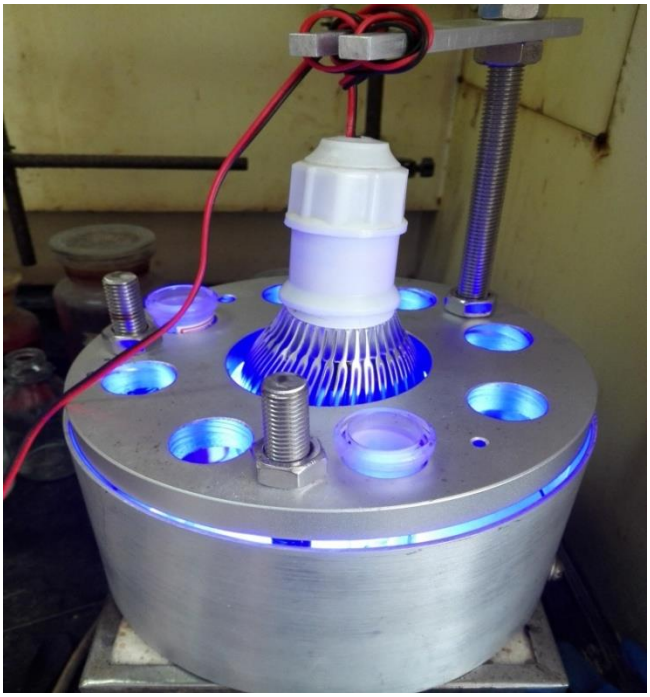
1. General Information

Column chromatography was generally performed on silica gel (200-300 mesh) and reactions were monitored by thin layer chromatography (TLC) using UV light to visualize the course of the reactions. The ^{1}H (400MHz) and ^{13}C NMR (100MHz)data were recorded on Bruker AVANCE Ⅱ 400MHz spectrometer using $CDCl_3$ as solvent. The chemical shifts (δ) are reported in ppm and coupling constants (*J*) in Hz. ^{1}H NMR spectra was recorded with tetramethylsilane (δ = 0.00 ppm)as internal reference; ^{13}C NMR spectra was recorded with $CDCl_3$ (δ = 77.00 ppm) as internal reference.

Reaction Apparatus:

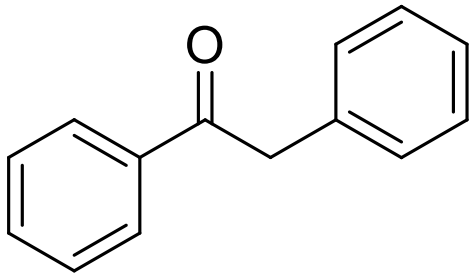
Photochemical reactions were carried out under visible light irradiation by a blue led bulb at room temperature.



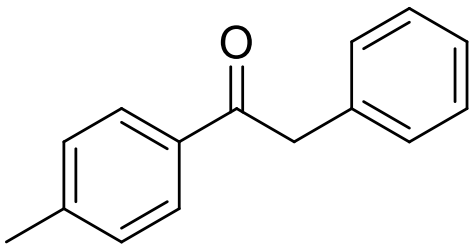
2. General procedures for synthesis of 3aa-3ap and 3ba-3bd

To a solution of aryl hydrazines (0.5 mmol) and alkene (1.5 mmol) in MeCN (1.5 mL) was added Methylene Blue (0.01mmol) and DABCO (0.5 mmol). The reaction mixture was stirred at 25℃ under air atmosphere (open vial) and irradiated by blue LED(7 W). The reaction was monitored by thin layer chromatography (TLC). When the reaction was completed, it was diluted with water and extracted with ethyl acetate 3 times. Removal of solvent followed by column chromatography afforded desired products.

3. Compound characterizations

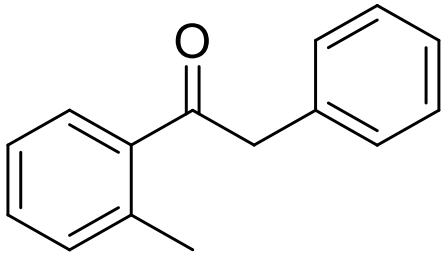


1,2-diphenylethanone (3aa).[1] Petroleum ether/ethyl acetate = 30:1, white solid, 81% yield (162 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00-8.03 (m, 2H), 7.56 (t, *J*= 7.2 Hz, 1H), 7.44-7.48 (m, 2H), 7.31-7.35 (m, 2H), 7.26-7.28 (m, 3H), 4.29 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.7, 136.7, 134.6, 133.2, 129.5, 128.7, 128.7, 128.7, 126.9, 45.5.

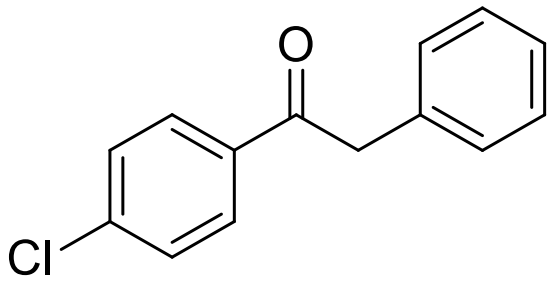


2-phenyl-1-(p-tolyl)ethanone (3ab).[1] Petroleum ether/ethyl acetate = 30:1, white

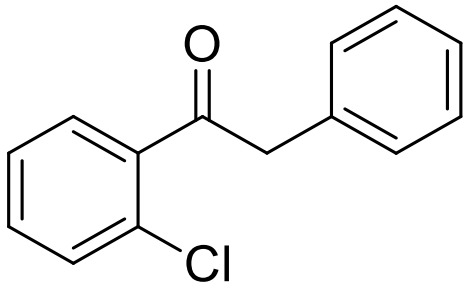
solid, 85% yield (170 mg). ^{1}H NMR(400 MHz, $CDCl_3$) δ 7.94-7.96 (m, 2H), 7.33-7.36 (m, 2H), 7.28-7.30 (m, 5H), 4.29 (s, 2H), 2.43 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.2, 143.9, 134.7, 134.0, 129.4, 129.2, 128.7, 128.6, 126.7, 45.3, 21.6.



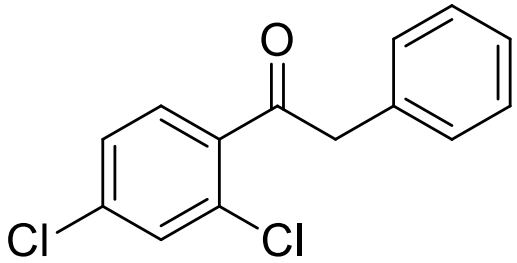
2-phenyl-1-(o-tolyl)ethanone (3ac). [2] Petroleum ether/ethyl acetate = 30:1, white solid, 70 %yield (140 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (dd, J_1= 7.6 Hz, J_2= 1.2 Hz, 1H), 7.28-7.38 (m, 4H), 7.23-7.26 (m, 4H), 4.23 (s, 2H), 2.45 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.4, 138.5, 137.5, 134.4, 131.9, 131.3, 129.5, 129.0, 128.6, 126.8, 125.6, 48.4, 21.2.



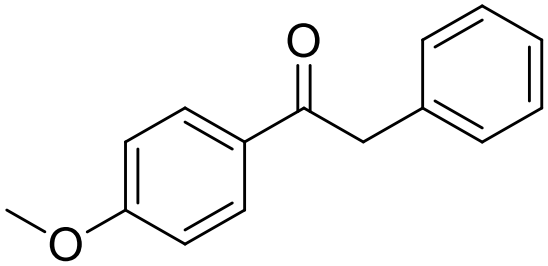
1-(4-chlorophenyl)-2-phenylethanone (3ad).[3] Petroleum ether/ethyl acetate = 30:1, white solid, 83% yield (166 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, J= 8.8 Hz, 2H), 7.43-7.45 (m, 2H), 7.33-7.35 (m, 2H), 7.25-7.29 (m, 3H), 4.27 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.4, 139.6, 134.8, 134.1, 130.0, 129.3, 128.9, 128.7, 127.0, 45.5.



1-(2-chlorophenyl)-2-phenylethanone (3ae).[4] Petroleum ether/ethyl acetate = 30:1, yellow oil, 65% yield (130 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24-7.41 (m, 9H), 4.27 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.9, 139.2, 133.6, 131.6, 130.7, 130.3, 129.6, 129.0, 128.6, 127.0, 126.8, 49.5.

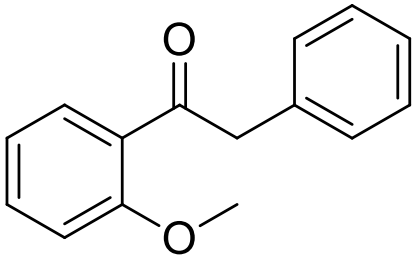


1-(2,4-dichlorophenyl)-2-phenylethanone (3af).[5] Petroleum ether/ethyl acetate = 30:1, white solid, 76% yield (152 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (d, *J*= 1.6 Hz, 1H), 7.26-7.36 (m, 5H), 7.22-7.24 (m, 2H), 4.25 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.6, 137.3, 137.2, 133.3, 131.9, 130.3, 130.2, 129.6, 128.7, 127.2, 127.2, 49.4.

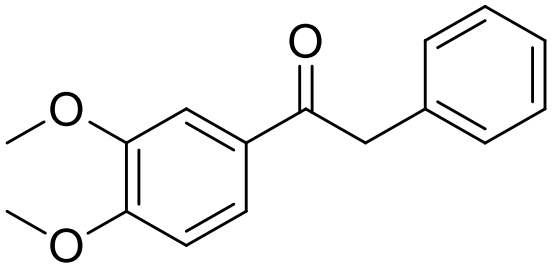


1-(4-methoxyphenyl)-2-phenylethanone (3ag). [4] Petroleum ether/ethyl acetate = 30:1, white solid, 84% yield (168 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, *J*= 9.2

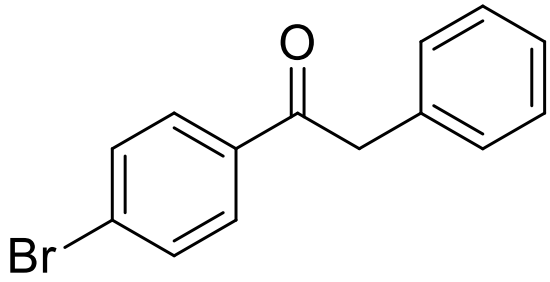
Hz, 2H), 7.25-7.35 (m, 5H), 6.94 (d, *J*= 8.8 Hz, 2H), 4.25 (s, 2H), 3.87 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.2, 163.5, 134.9, 130.9, 129.6, 129.3, 128.6, 126.7, 113.7, 55.4, 45.2.



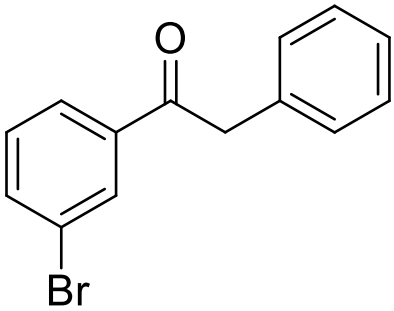
1-(2-methoxyphenyl)-2-phenylethanone (3ah).[4] Petroleum ether/ethyl acetate = 30:1, yellow oil, 65% yield (170 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66-7.68 (m, 1H), 7.43-7.47 (m, 1H), 7.28-7.32 (m, 2H), 7.21-7.24 (m, 3H), 6.95-7.00 (m, 2H), 4.30 (s, 2H), 3.92 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.1, 158.3, 135.2, 133.5, 130.6, 129.6, 128.3, 126.5, 120.7, 111.4, 55.4, 50.1.



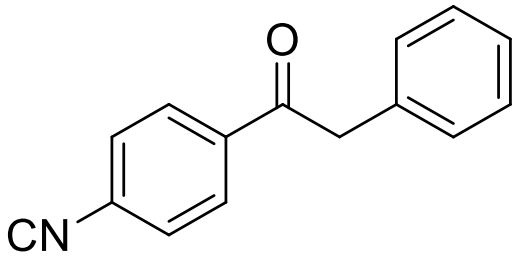
1-(3,4-dimethoxyphenyl)-2-phenylethanone (3ai).[6] Petroleum ether/ethyl acetate = 30:1, white solid, 76% yield (152 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (dd, J_1= 8.4 Hz, J_2= 2 Hz, 1H), 7.56 (d, *J*= 2.0 Hz, 1H), 7.25-7.33 (m, 5H), 6.88 (d, *J*= 8.4 Hz, 1H), 4.25 (s, 2H), 3.94 (s, 3H), 3.92 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.3, 153.3, 149.0, 135.0, 129.7, 129.3, 128.6, 126.8, 123.4, 110.6, 110.0, 56.0, 55.9, 45.2.



1-(4-bromophenyl)-2-phenylethanone (3aj).[7] Petroleum ether/ethyl acetate = 30:1, yellow solid, 78% yield (156 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87-7.89 (m, 2H), 7.59-7.62 (m, 2H), 7.34 (t, *J*= 7.2 Hz, 2H), 7.25-7.29 (m, 3H), 4.26 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.6, 135.2, 134.1, 131.9, 130.1, 129.3, 128.8, 128.4, 127.0, 45.5.

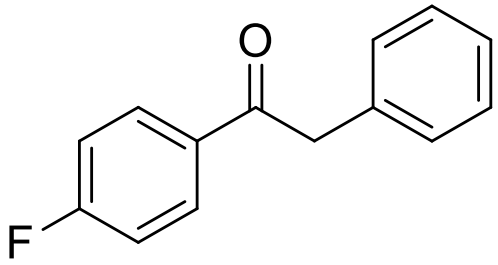


1-(3-bromophenyl)-2-phenylethanone (3ak).[1] Petroleum ether/ethyl acetate = 30:1, white solid, 68% yield (136 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (t, *J*= 2.0 Hz, 1H), 7.93-7.95 (m, 1H), 7.70 (dq, J_1= 8.0 Hz, J_2= 1.2 Hz, 1H), 7.33-7.37 (m, 3H), 7.26-7.30 (m, 3H), 4.27 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.2, 138.3, 136.0, 133.9, 131.6, 130.2, 129.4, 128.7, 127.1, 127.1, 123.0, 45.5.

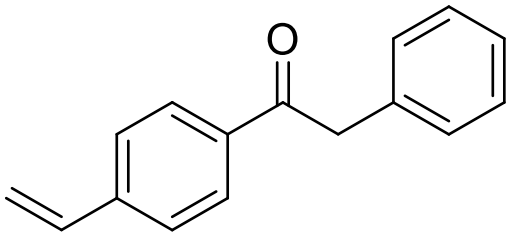


4-(2-phenylacetyl)benzonitrile (3al).[1] Petroleum ether/ethyl acetate = 30:1,

white solid, 85% yield (170 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08-8.10 (m, 2H), 7.76-7.78 (m, 2H), 7.34-7.37 (m, 2H), 7.29-7.31 (m, 1H), 7.24-7.26 (m, 2H), 4.31 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.2, 139.4, 133.4, 132.5, 129.3, 129.0, 128.9, 127.3, 117.8, 116.4, 45.8.

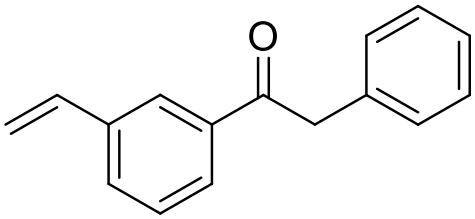


1-(4-fluorophenyl)-2-phenylethanone (3am).[8] Petroleum ether/ethyl acetate = 30:1, white solid, 85% yield (170 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03-8.07 (m, 2H), 7.33-7.37 (m, 2H), 7.26-7.29 (m, 3H), 7.14 (t, *J*= 8.8 Hz, 2H), 4.27 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.0, 165.7 (d, *J*= 254.2 Hz), 134.3, 132.9 (d, *J*= 2.5 Hz), 131.2 (d, *J*= 9.1 Hz), 129.3, 128.7, 126.9, 115.7 (d, *J*= 22.2 Hz), 45.4.

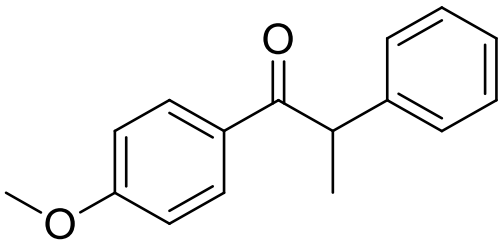


2-phenyl-1-(4-vinylphenyl)ethanone (3an). Petroleum ether/ethyl acetate = 30:1, white solid, 68% yield (136 mg), mp: 88-90 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J*= 8.4 Hz, 2H), 7.49 (d, *J*= 8.4 Hz, 2H), 7.32-7.36 (m, 2H), 7.26-7.29 (m, 3H), 6.76 (dd, J_1= 17.6 Hz, J_2= 10.8 Hz, 1H), 5.88 (dd, J_1= 17.1 Hz, J_2= 0.8 Hz, 1H), 5.41 (dd, J_1= 10.8 Hz, J_2= 0.4 Hz, 1H), 4.29 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.0, 142.1, 135.9, 135.7, 134.6, 129.4, 129.0, 128.7, 126.9, 126.4, 116.8, 45.5. IR

(film) ν/cm^{-1} 3090 (m), 3020 (m), 3058 (m), 2932 (w), 1660 (vs), 690 (s). MS(ESI, *m/z*) 223.1 (M + H$^+$), 245.1 (M + Na$^+$). Anal.calcd for $C_{16}H_{14}O$: C, 86.45; H, 6.35; O, 7.20. Found: C, 86.18; H, 6.30; O, 7.52.

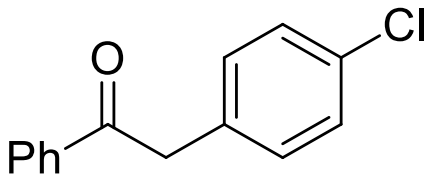


2-phenyl-1-(3-vinylphenyl)ethanone (3ao). Petroleum ether/ethyl acetate = 30:1, white oil, 87% yield (174 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07-8.09 (m, 1H), 7.92-7.94 (m, 1H), 7.62-7.64 (m, 1H), 7.45 (t, J= 7.6 Hz, 1H), 7.36-7.39 (m, 2H), 7.28-7.32 (m, 3H), 6.79 (dd, J_1= 17.6 Hz, J_2= 10.8 Hz, 1H), 5.86 (d, J= 17.6 Hz, 1H), 5.38 (d, J= 10.8 Hz, 1H), 4.33 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.5, 138.0, 136.8, 135.9, 134.4, 130.6, 129.4, 128.8, 128.6, 127.9, 126.8, 126.3, 115.2, 45.5. IR (film) ν/cm^{-1} 3095 (m), 3014 (m), 2833 (s), 1686 (vs), 760 (w), 694 (m). MS(ESI, *m/z*) 223.1 (M + H$^+$), 245.1 (M + Na$^+$). Anal.calcd for $C_{16}H_{14}O$: C, 86.45; H, 6.35; O, 7.20. Found: C, 86.22; H, 6.23; O, 7.55.

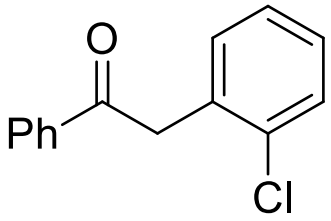


1-(4-methoxyphenyl)-2-phenylpropan-1-one (3ap).[9] Petroleum ether/ethyl acetate = 30:1, white solid, 76% yield (152 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ

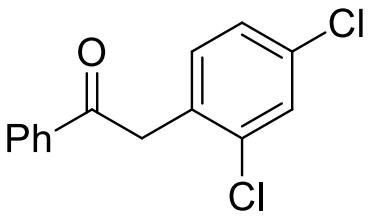
7.93-7.96 (m, 2H), 7.27-7.29 (m, 4H), 7.18-7.21 (m, 1H), 6.84-6.87 (m, 2H), 4.64 (q, *J*= 6.8 Hz, 1H), 3.81 (s, 3H), 1.51 (d, *J*= 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.8, 163.1, 141.9, 131.0, 129.4, 128.9, 127.6, 126.7, 113.6, 55.3, 47.5, 19.5.



2-(4-chlorophenyl)-1-phenylethanone (3ba).[8] Petroleum ether/ethyl acetate = 30:1, white solid, 80% yield (160 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98-8.00 (m, 2H), 7.57 (t, *J*= 7.2 Hz, 1H), 7.46 (t, *J*= 7.6 Hz, 2H), 7.28-7.30 (m, 2H), 7.18-7.20 (m, 2H), 4.25 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.0, 136.4, 133.3, 132.9, 132.8, 130.9, 128.7, 128.7, 128.5, 44.6.

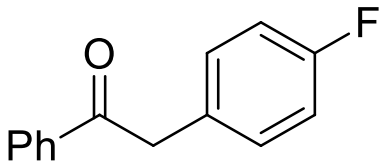


2-(2-chlorophenyl)-1-phenylethanone (3bb).[5] Petroleum ether/ethyl acetate = 30:1, white solid, 70% yield (140 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06-8.09 (m, 2H), 7.60-7.64 (m, 1H), 7.52 (t, *J*= 7.2 Hz, 2H), 7.43-7.45 (m, 1H), 7.25-7.28 (m, 3H), 4.47 (s, 2H). ^{13}CNMR (100 MHz, $CDCl_3$) δ 196.2, 136.5, 134.3, 133.2, 133.0, 131.6, 129.4, 128.6, 128.4, 128.2, 126.8, 43.1.

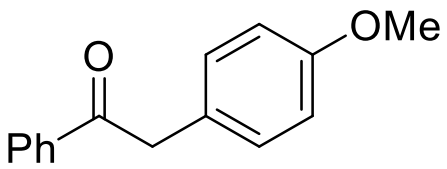


2-(2,4-dichlorophenyl)-1-phenylethanone (3bc).[5] Petroleum ether/ethyl acetate

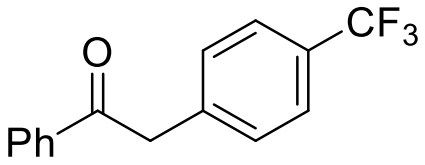
= 30:1, white solid, 75% yield (150 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98-8.00 (m, 2H), 7.59 (t, *J*= 7.2 Hz, 1H), 7.48 (t, *J*= 7.6 Hz, 2H), 7.35-7.40 (m, 2H), 7.08-7.10 (m, 1H), 4.24 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.4, 136.1, 134.5, 133.5, 132.5, 131.5, 131.1, 130.4, 129.0, 128.8, 128.4, 44.2.



2-(4-fluorophenyl)-1-phenylethanone (3bd).[8] Petroleum ether/ethyl acetate = 30:1, white solid, 85% yield (170 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00-8.03 (m, 2H), 7.59 (t, *J*= 7.6 Hz, 1H), 7.49 (t, *J*= 7.6 Hz, 2H), 7.22-7.25 (m, 2H), 7.01-7.05 (m, 2H), 4.28 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.3, 161.8 (d, *J*= 243.7 Hz), 136.4, 133.2, 131.0 (d, *J*= 7.3 Hz), 130.1 (d, *J*= 2.9 Hz), 128.6, 128.4, 115.4 (d, *J*= 21.2 Hz), 44.4.



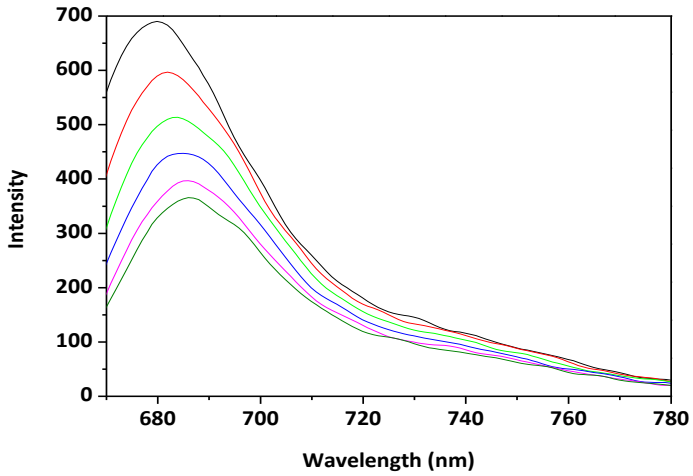
2-(4-methoxyphenyl)-1-phenylethan-1-one (3be).[10] Petroleum ether/ethyl acetate = 20:1, white solid, 72% yield (144 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00-8.02 (m, 2H), 7.56 (t, *J*= 7.2 Hz, 1H), 7.44-7.48 (m, 2H), 7.17-7.20 (m, 2H), 6.86-6.88 (m, 2H), 4.23 (s, 2H), 3.79(s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.9, 158.6, 136.7, 133.1, 130.5, 128.6, 128.6, 126.5, 114.2, 55.3, 44.4.

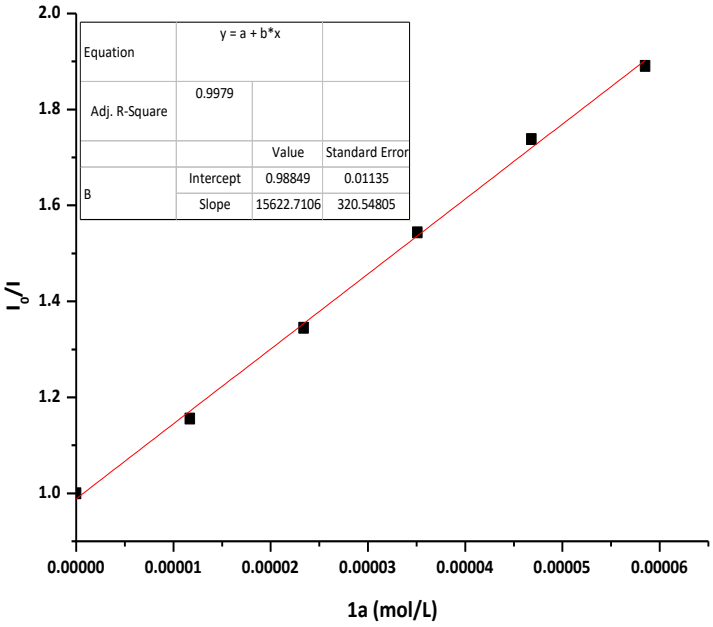


1-phenyl-2-(4-(trifluoromethyl)phenyl)ethan-1-one (3bf).[11] Petroleum ether/ethyl acetate = 20:1, white solid, 86% yield (172 mg). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00-8.03 (m, 2H), 7.58-7.62 (m, 3H), 7.47-7.51 (m, 2H), 7.38-7.40 (m, 2H), 4.36 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.7, 138.6, 136.4, 133.5, 130.0, 128.8, 128.5, 125.5 (q, *J*= 4.5 Hz), 45.0.

4. Luminescence quenching by compound 1a

A Varian Cary Eclipse fluorescence spectrometer was used to record the emission intensities. All the solutions were excited at 664 nm and the emission intensity at 685 nm was observed. CH_3CN was degassed with a stream of N_2 for 30 min. In a typical experiment, the emission spectrum of a 5×10^{-5} M solution of Methylene Blue in CH_3CN was collected. Then, appropriate amount of quencher was added to the measured solution in a quartz cuvette and the emission spectrum of the sample was collected. I_0 and I represent the intensities of the emission in the absence and presence of the quencher at 685 nm.





5. References

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[8] Gao K, Yorimitsu H, Osuka A. *Angewandte Chemie*, 2016.

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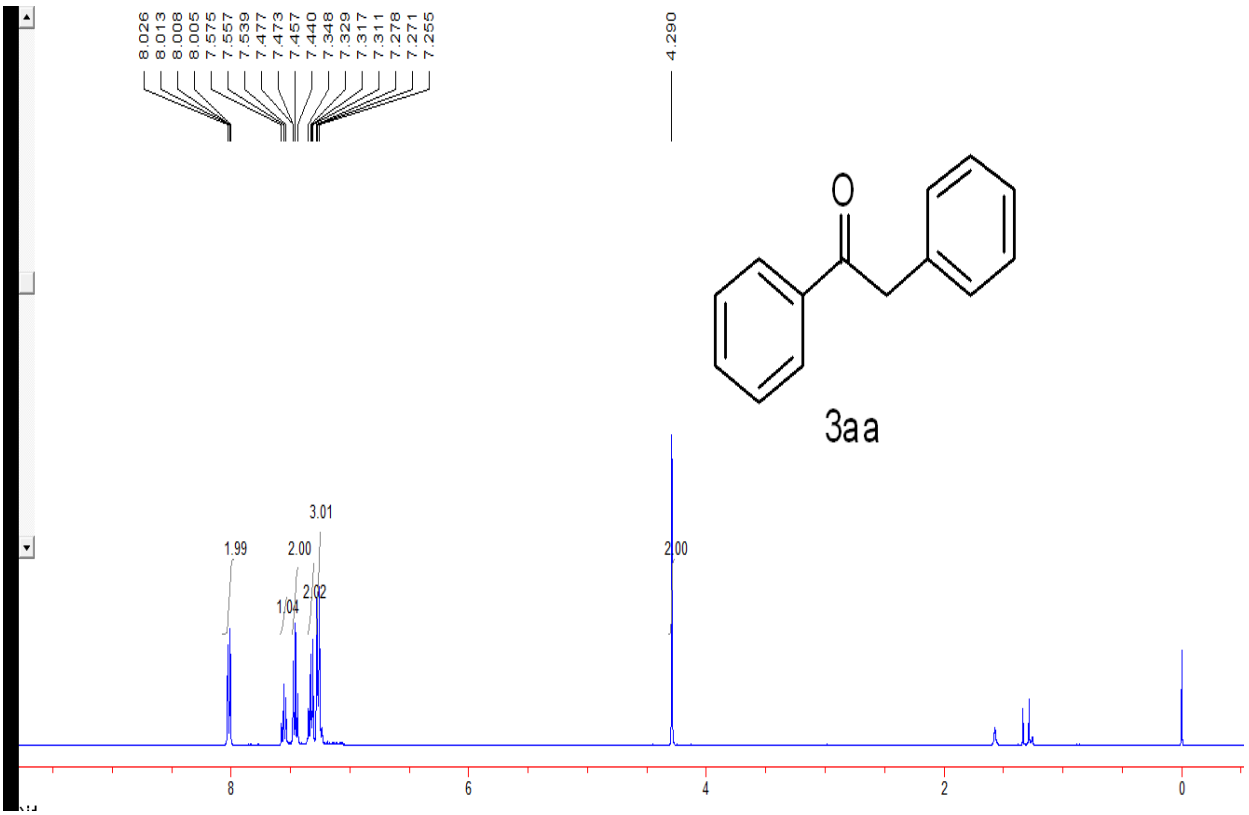
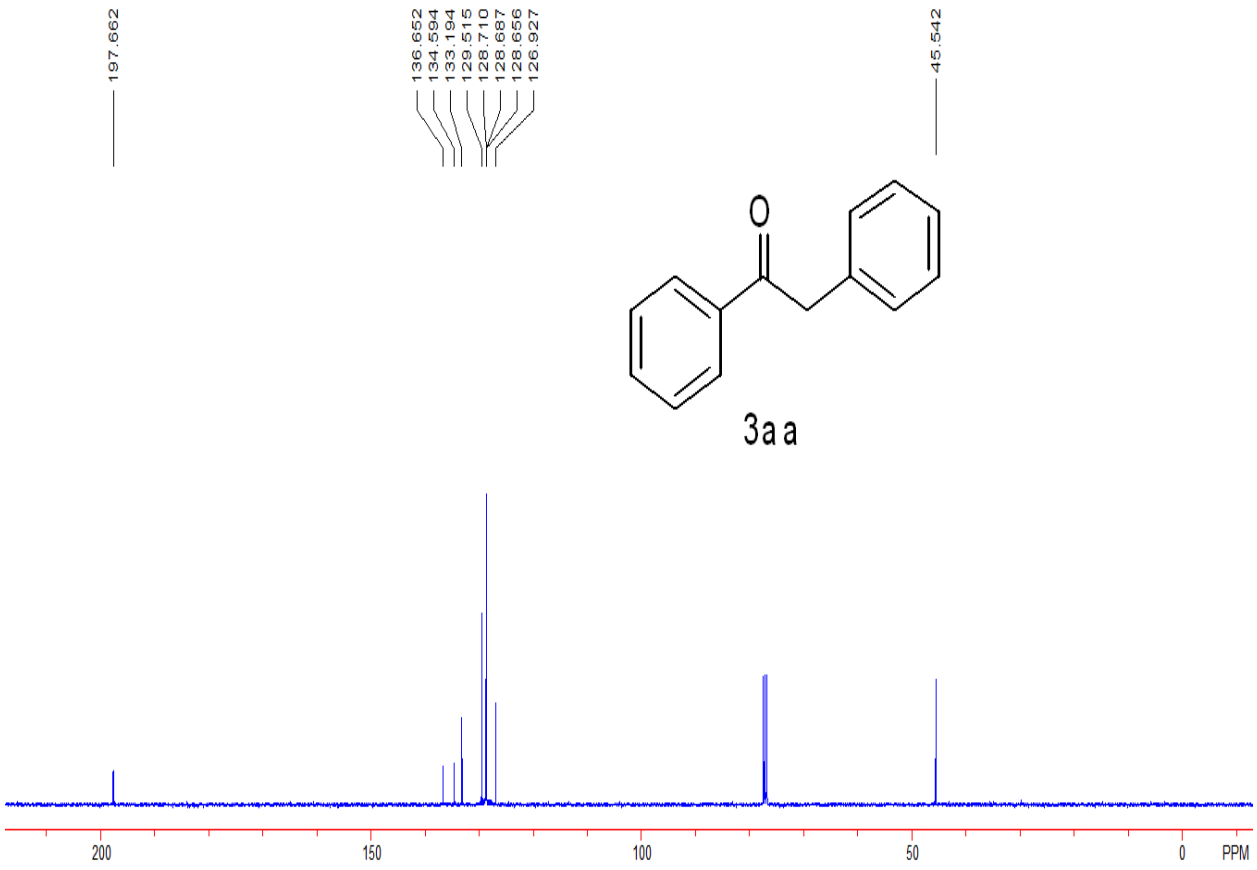
[10] Gao K, Yorimitsu H, Osuka A. *Angewandte Chemie*, 2016.

[11] Cao C, Wang L, Cai Z, et al. *European Journal of Organic Chemistry*, 2011, 2011(8): 1570-1574.

6. Spectroscopic Data for Products

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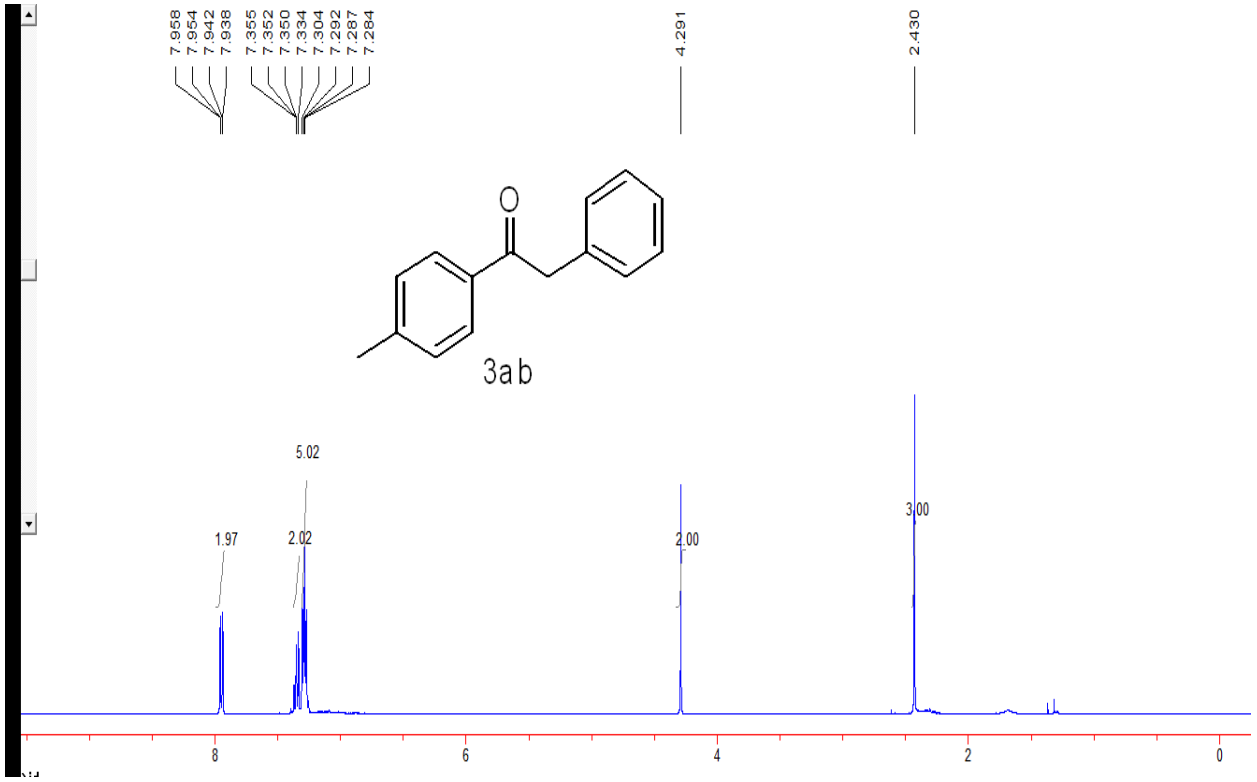
0
PPM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8
6
4
2
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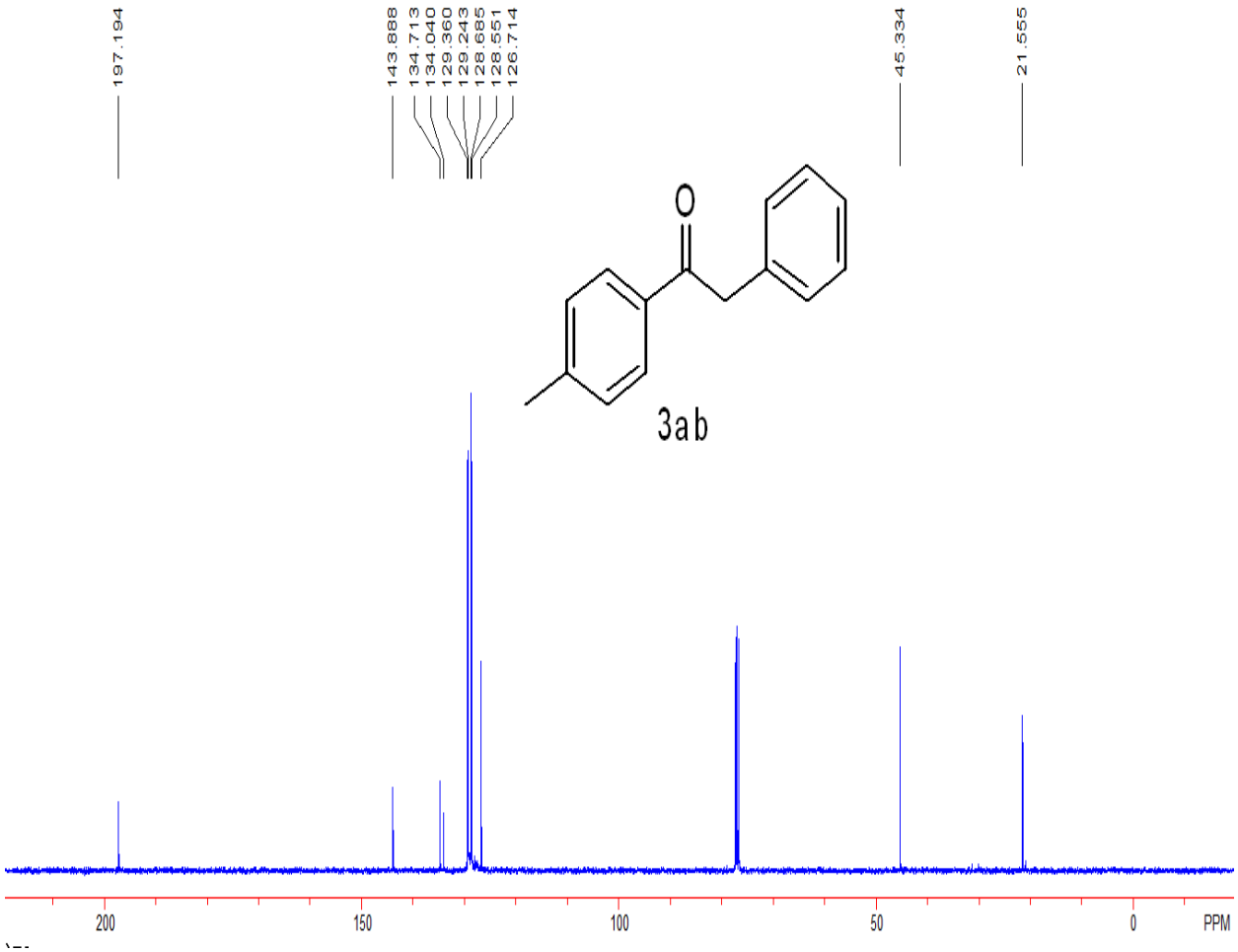
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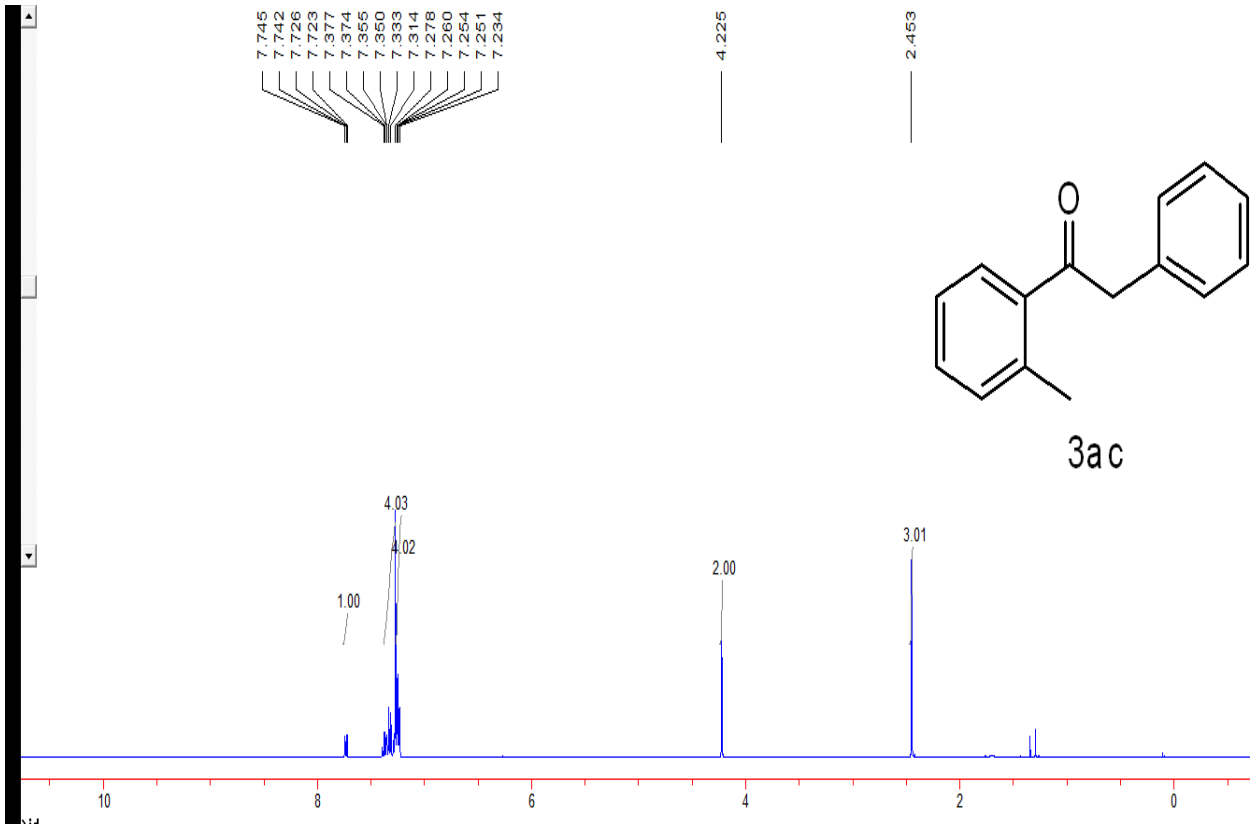
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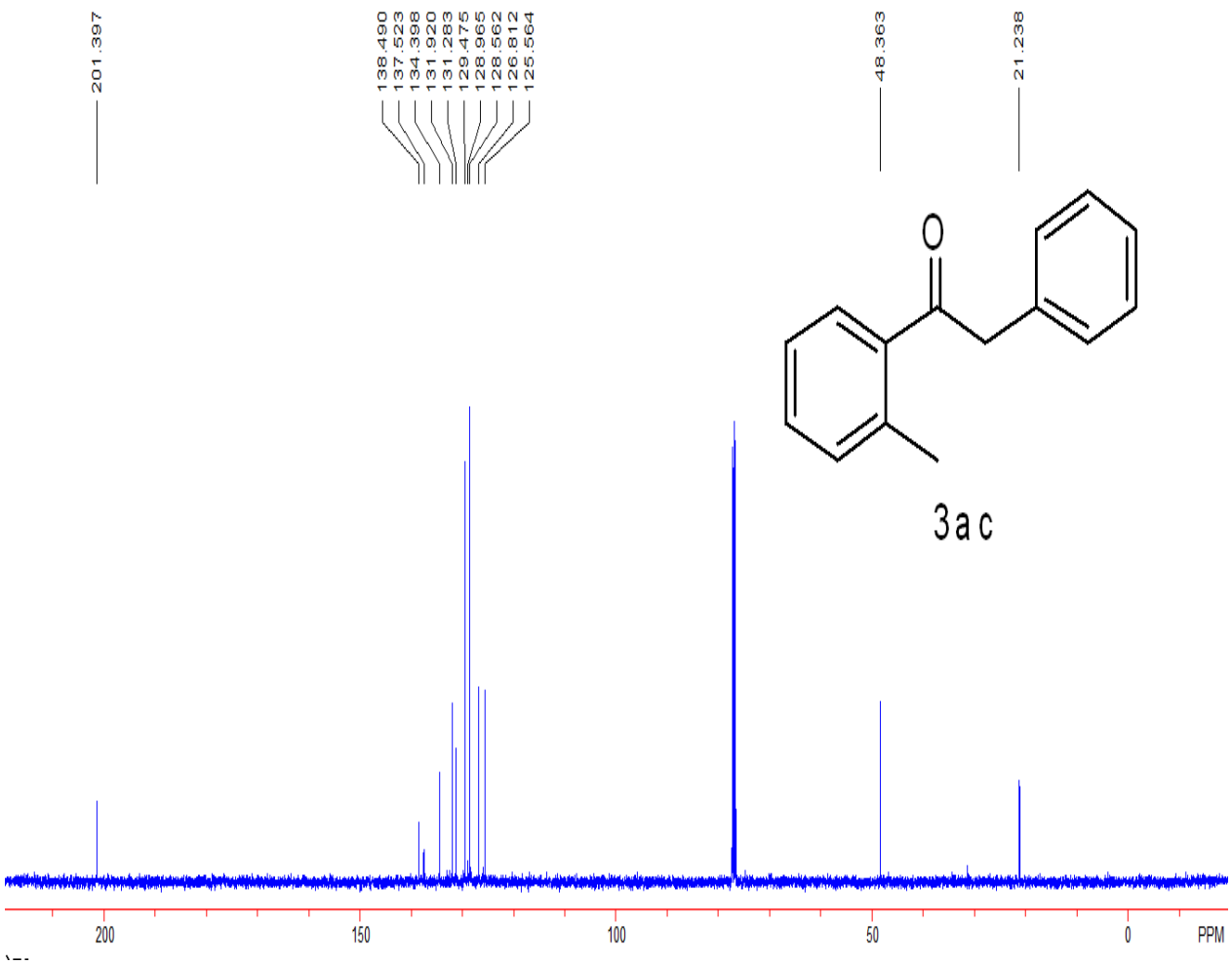
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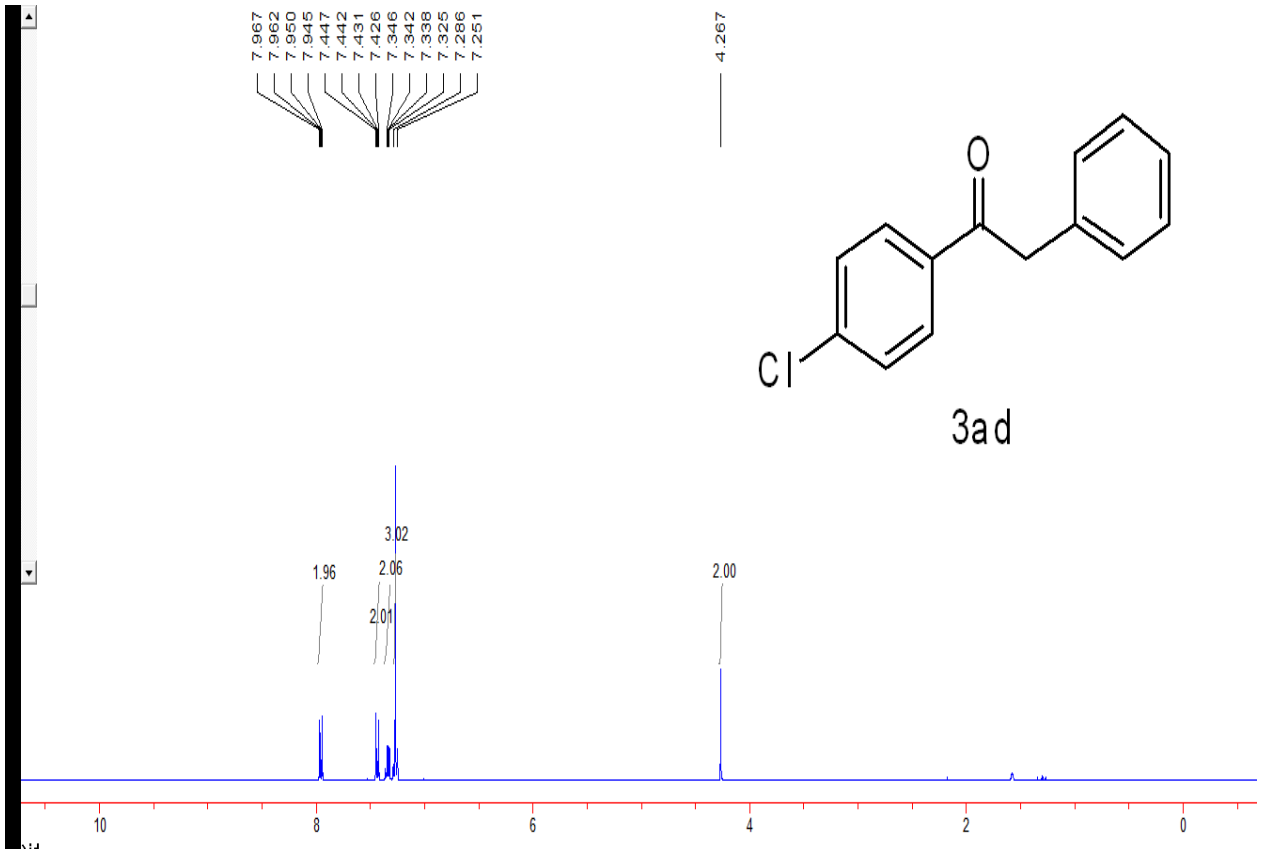
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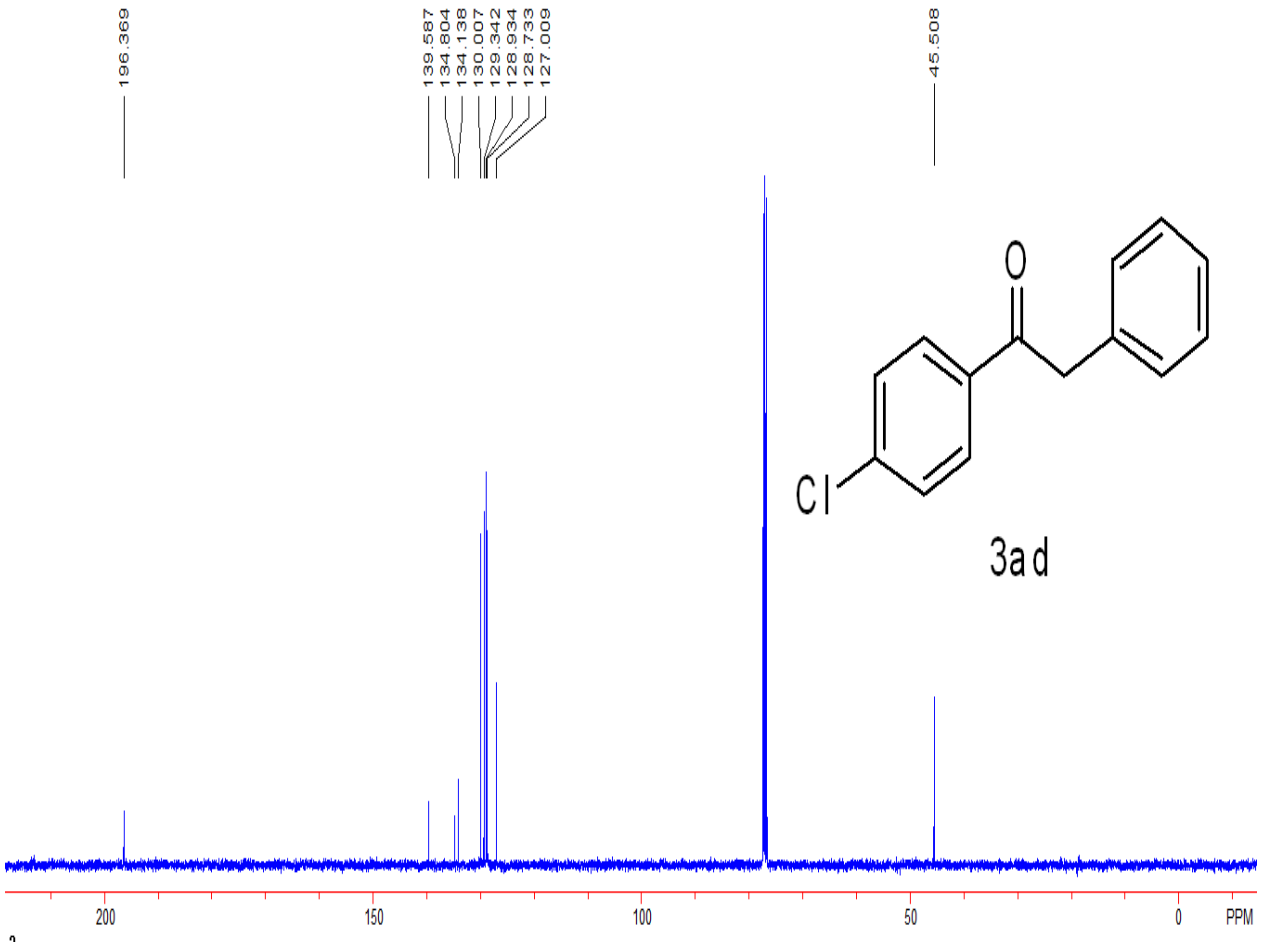
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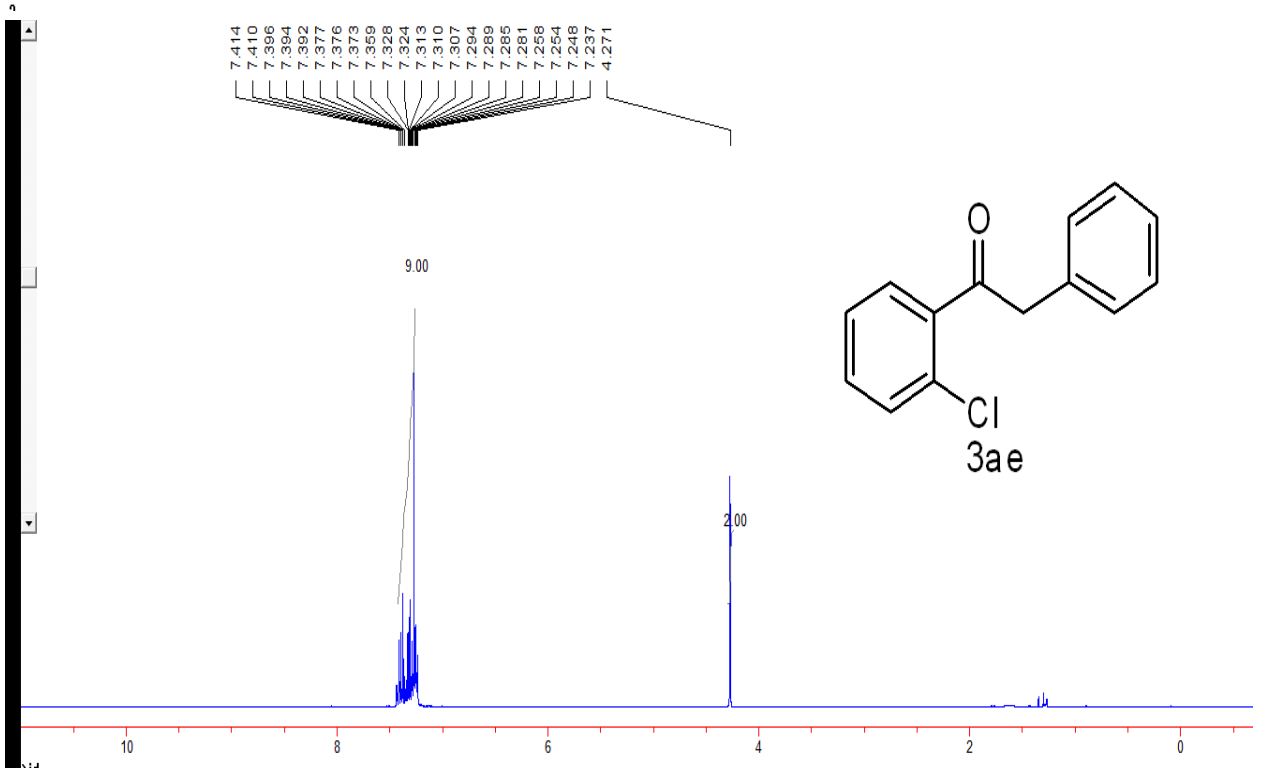
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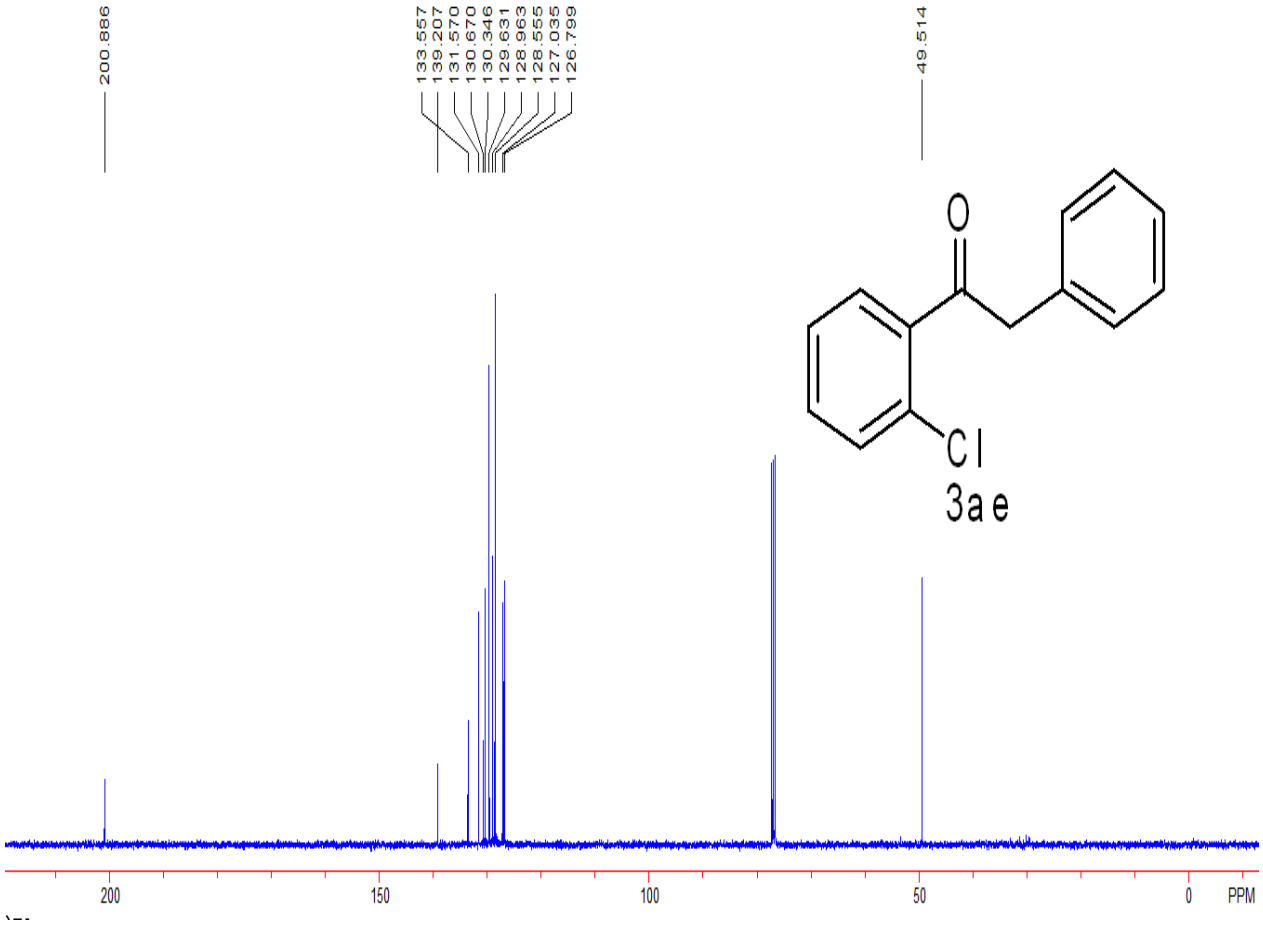
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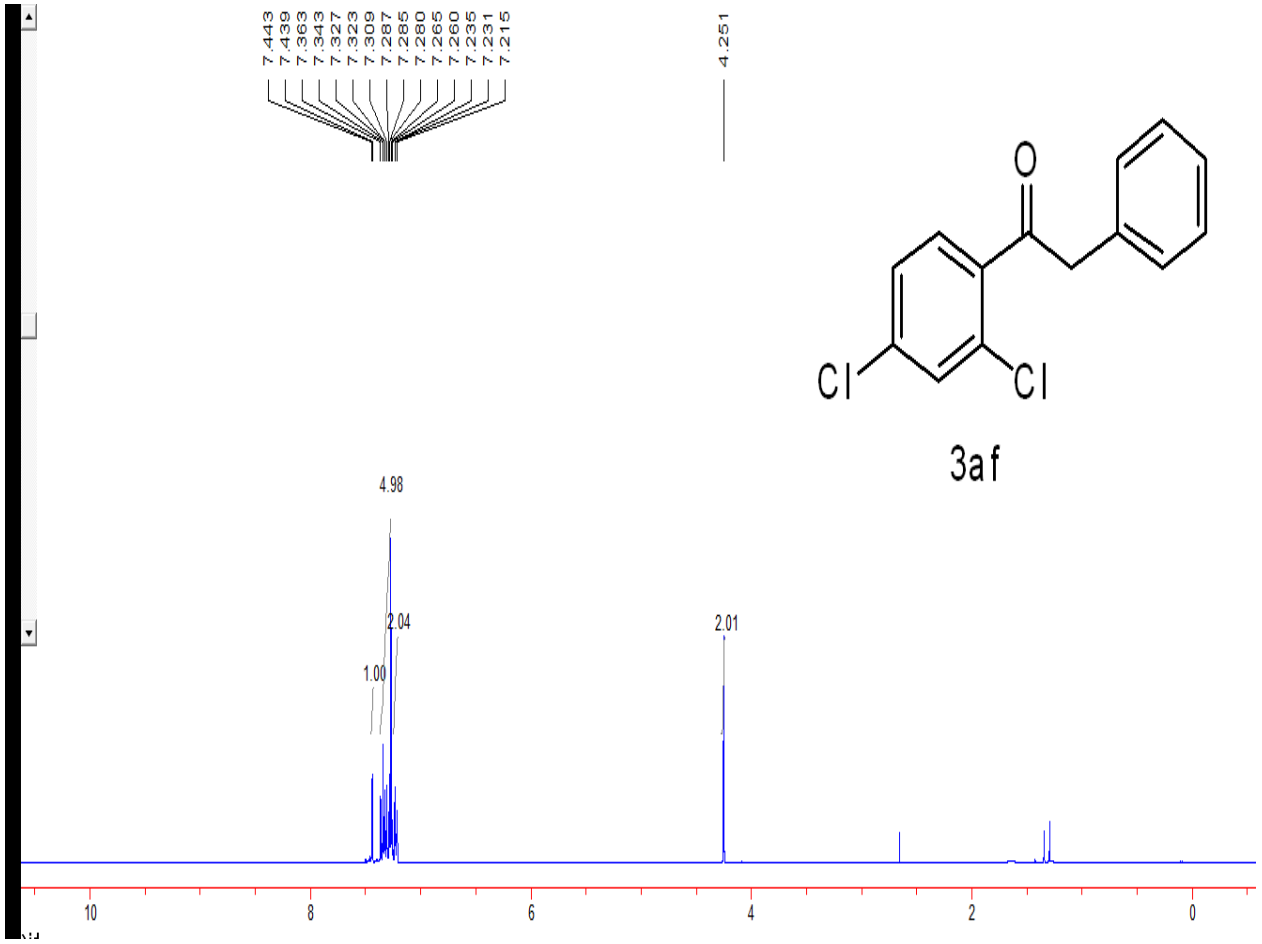
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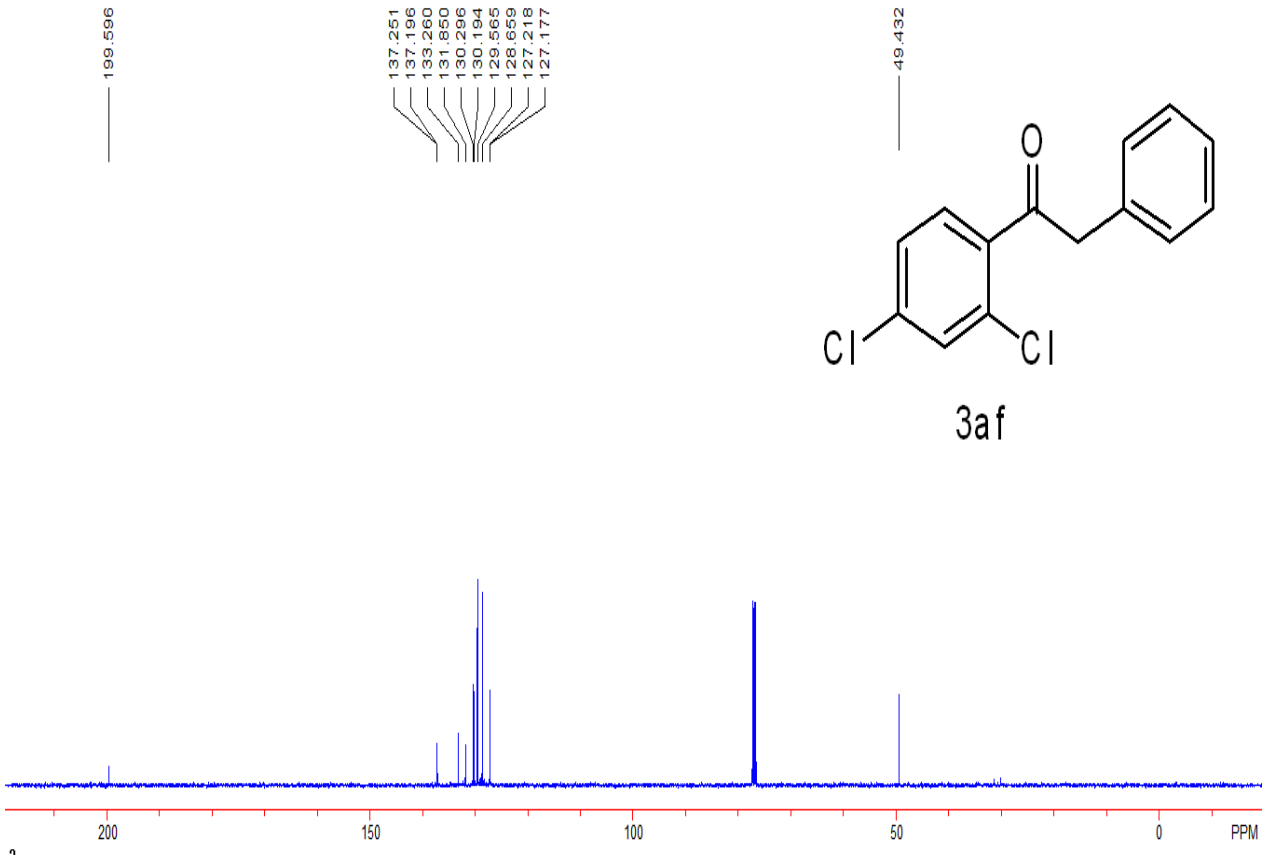
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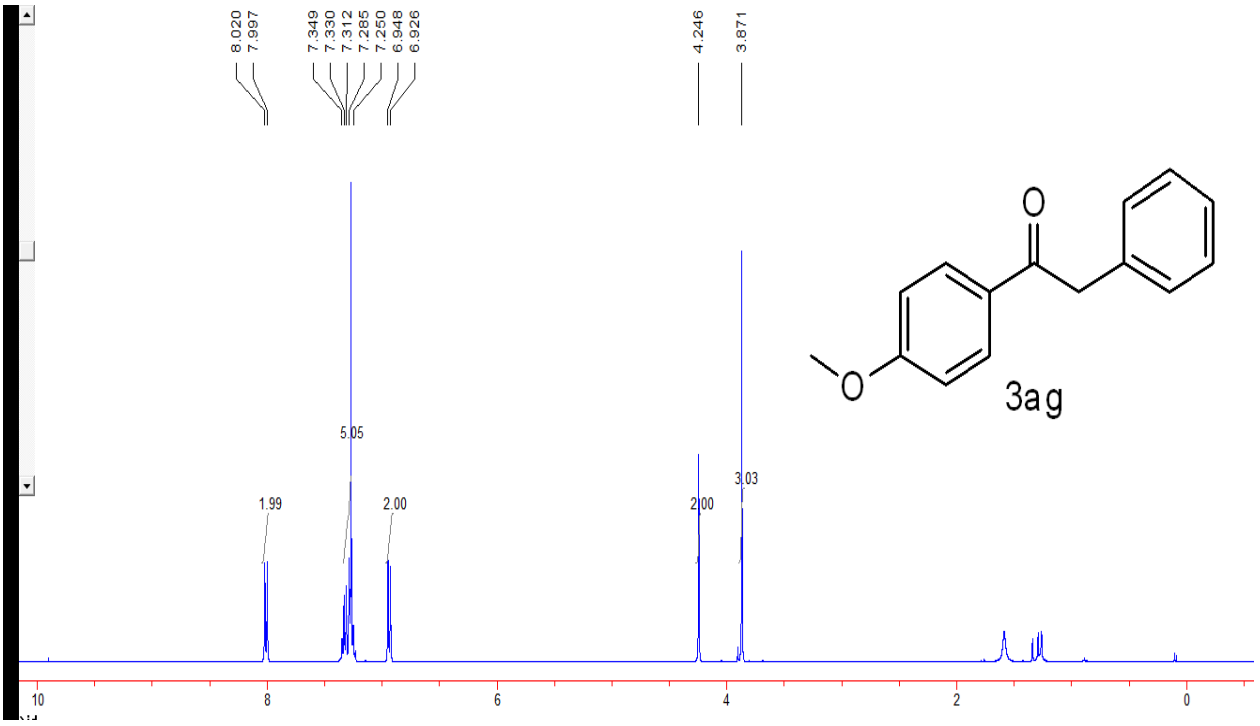
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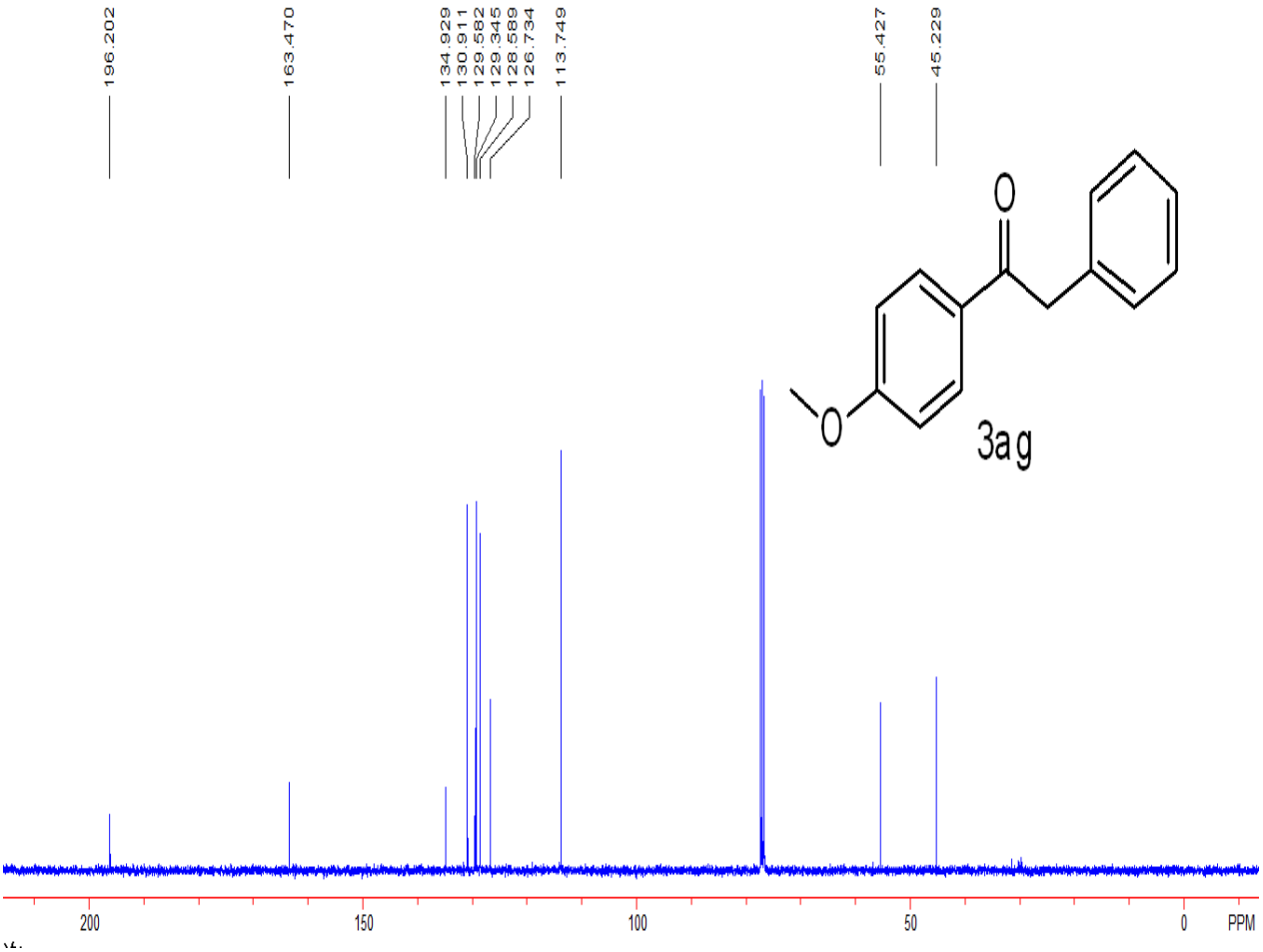
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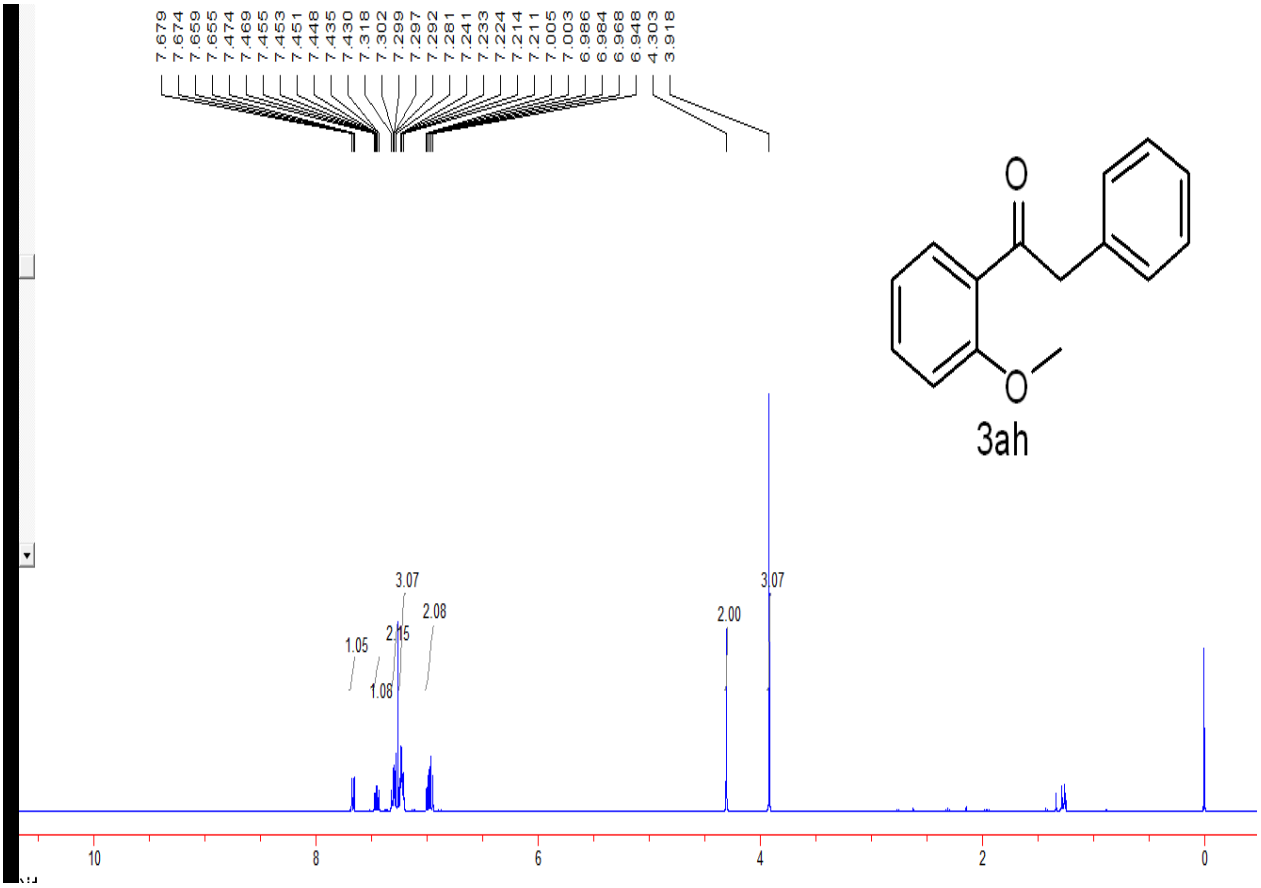
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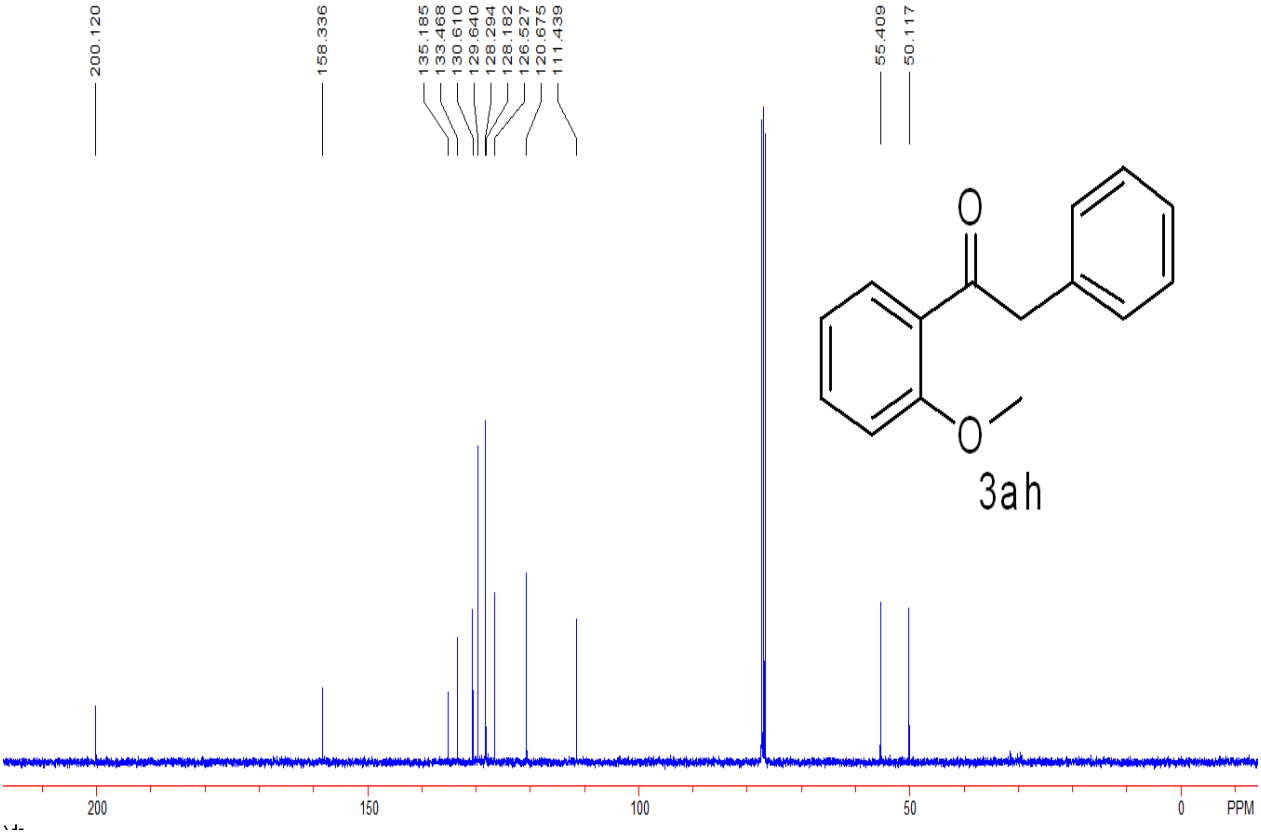
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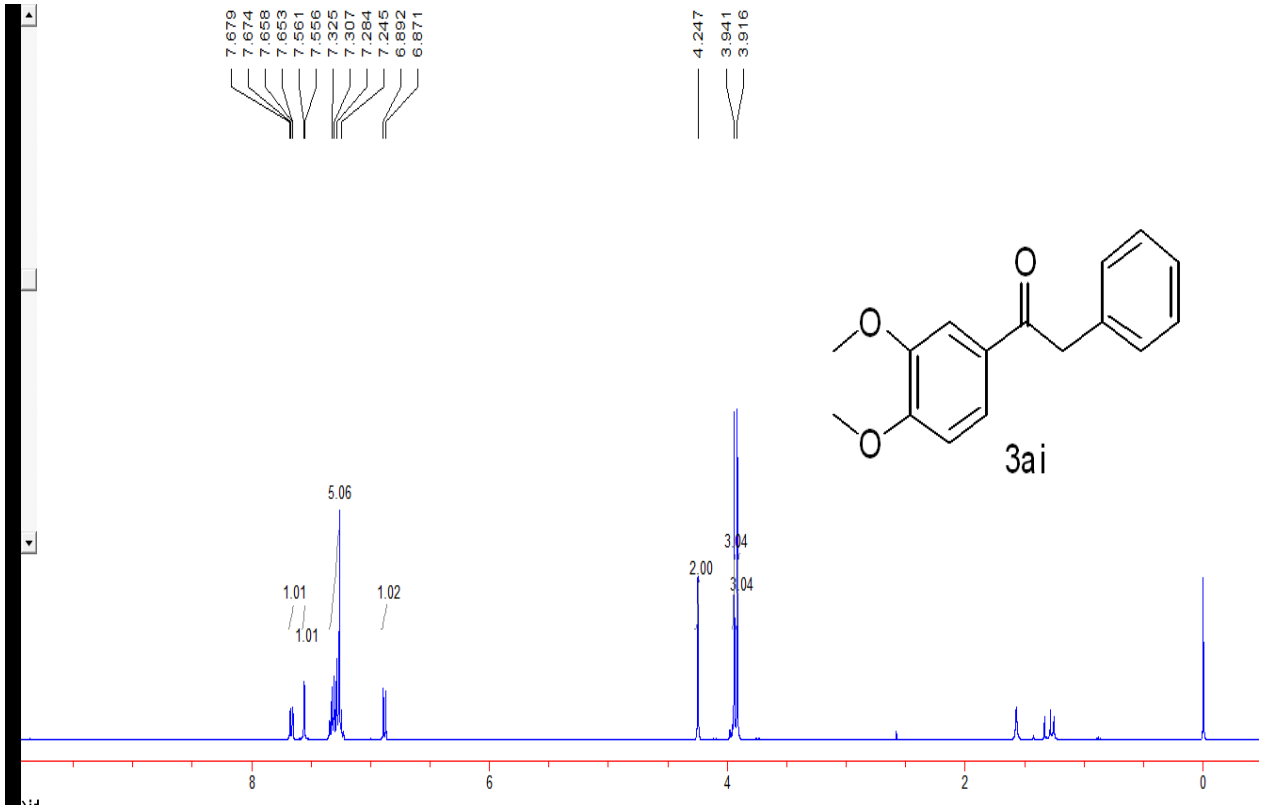
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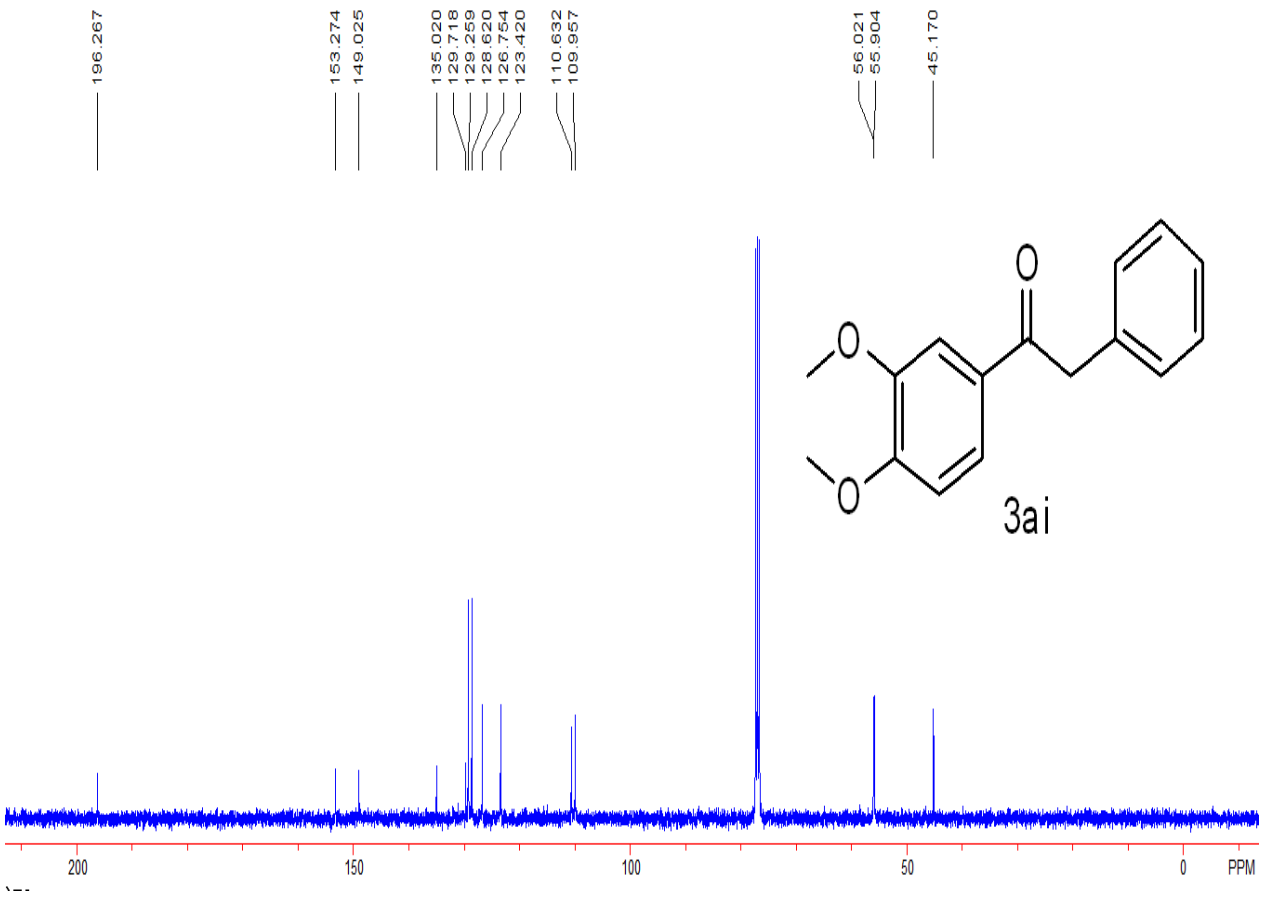
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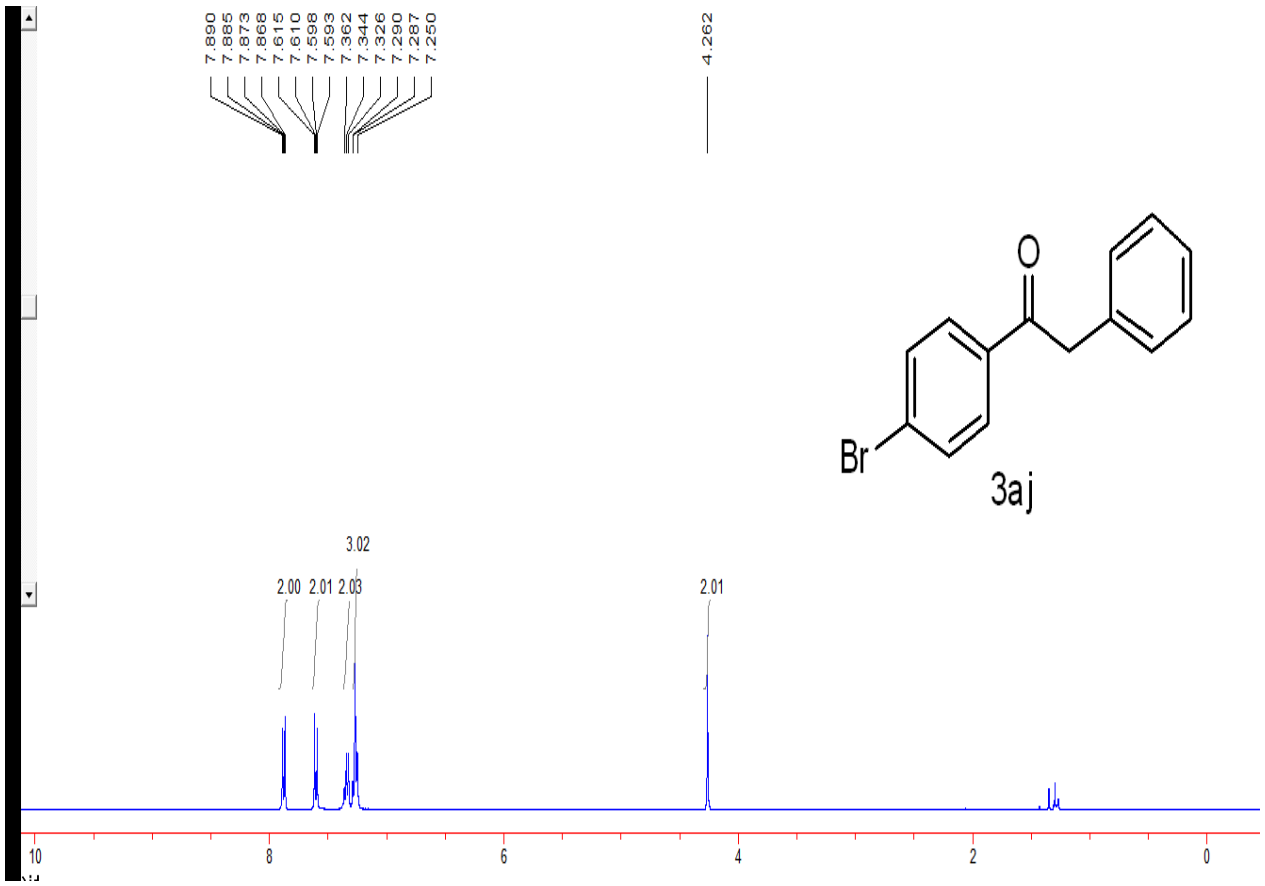
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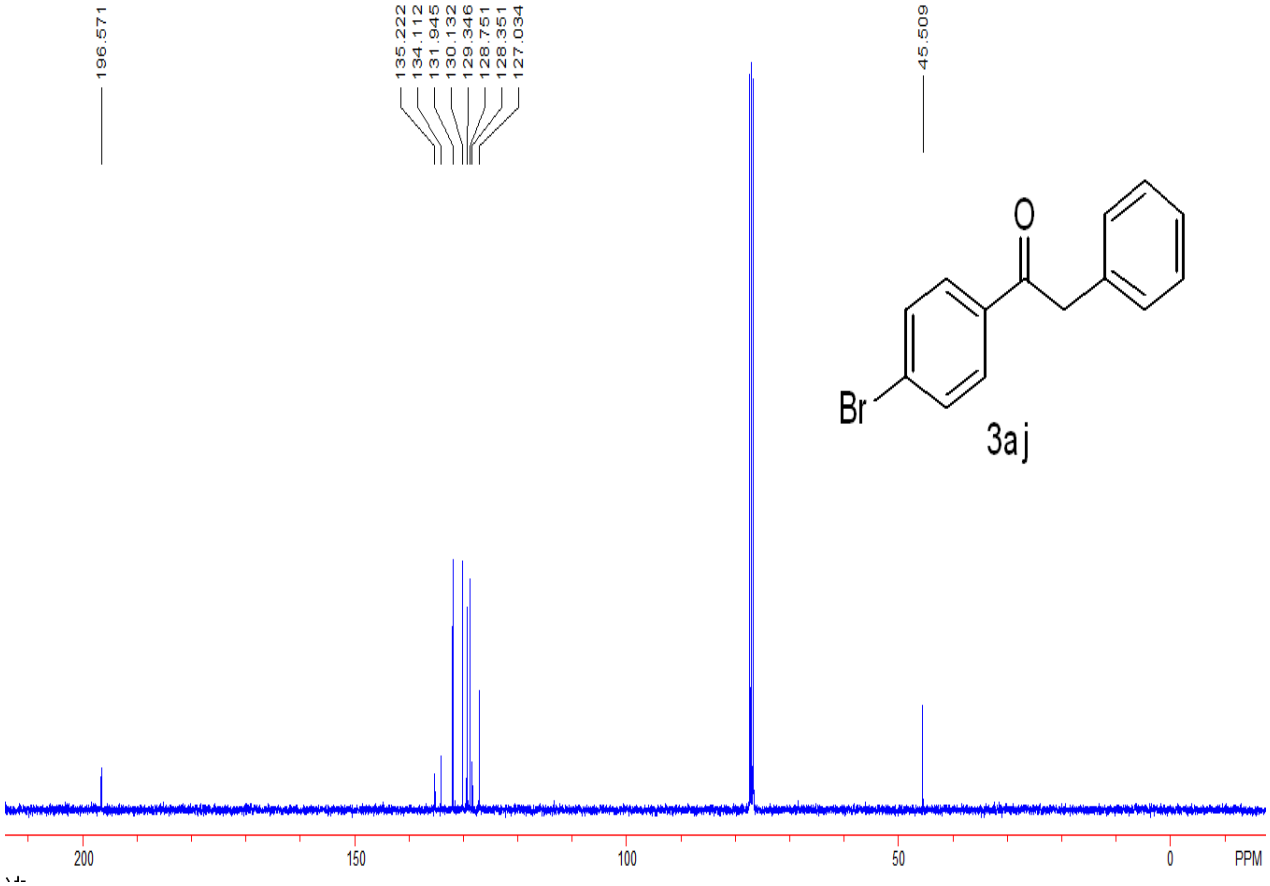
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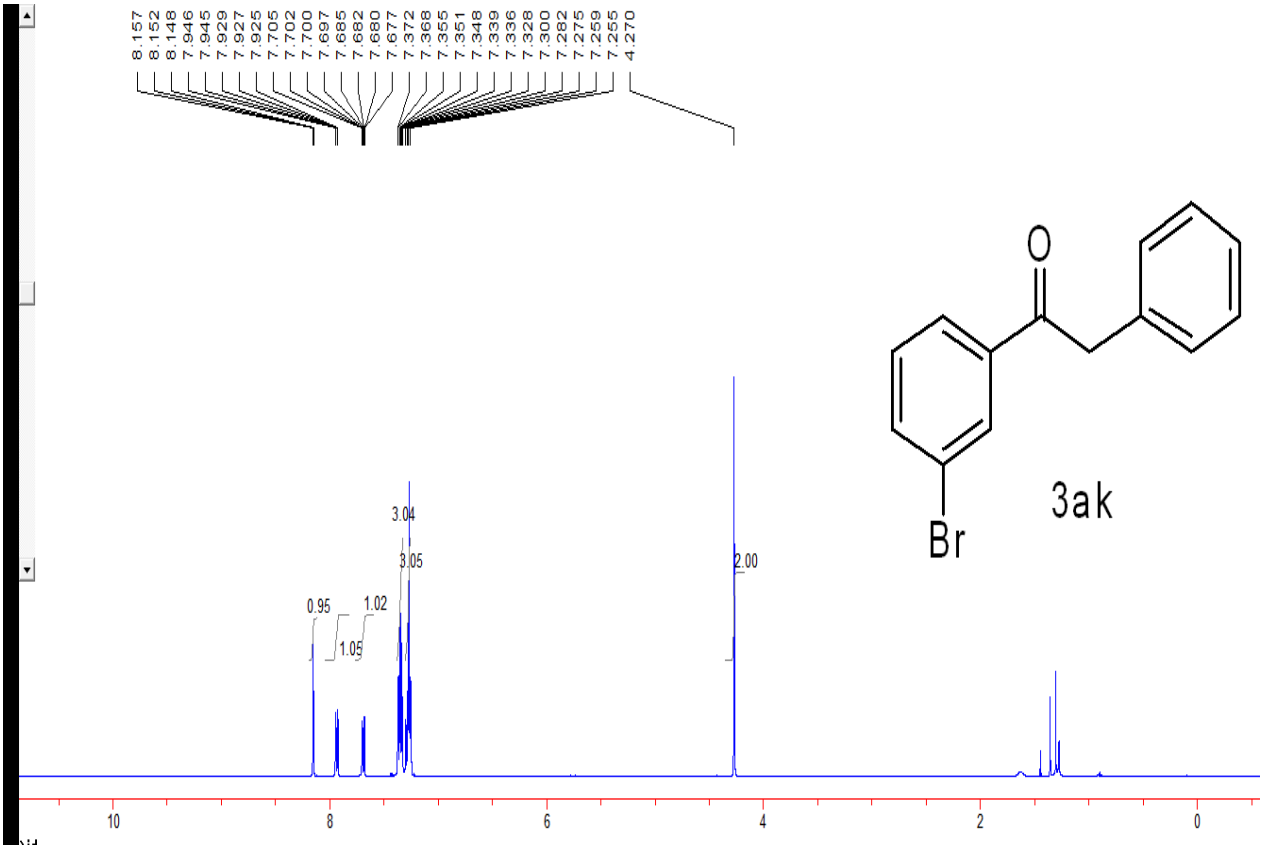
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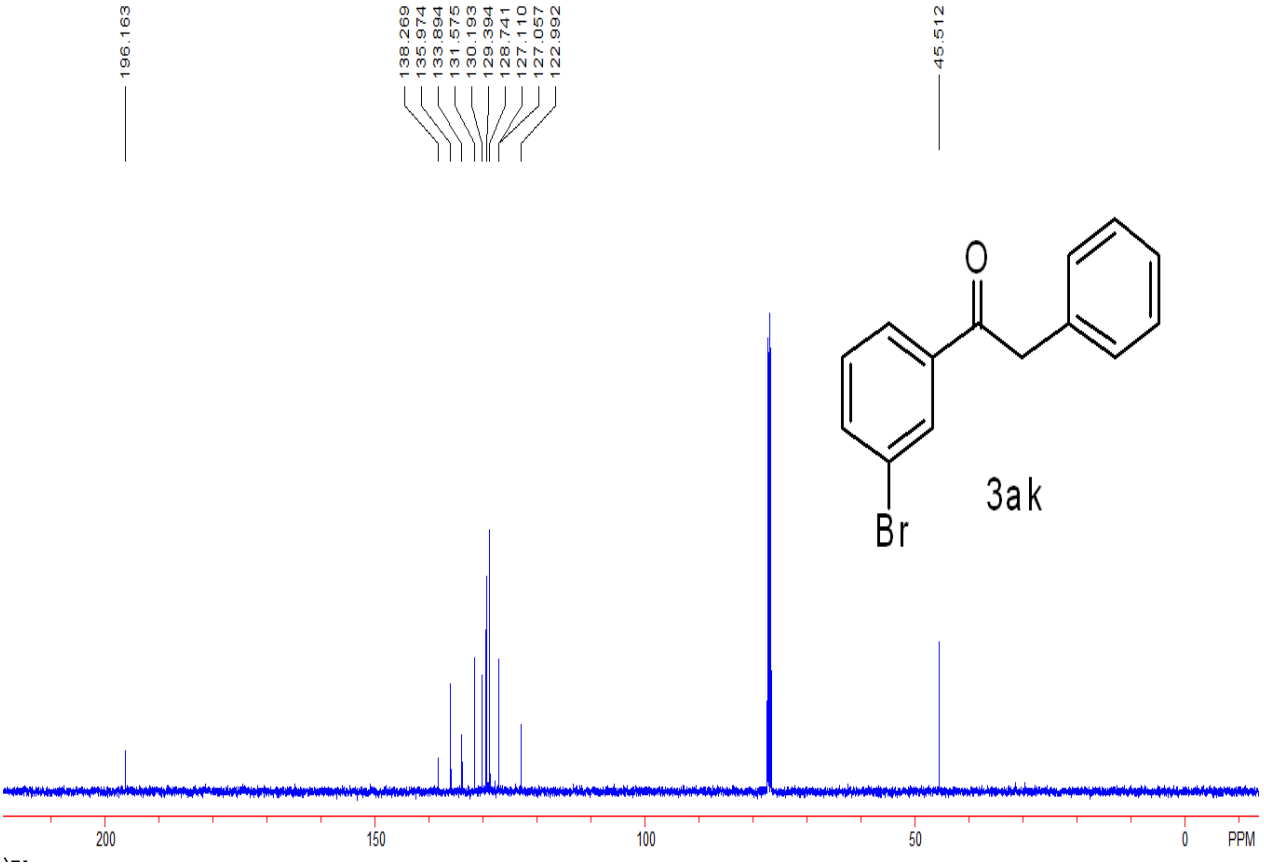
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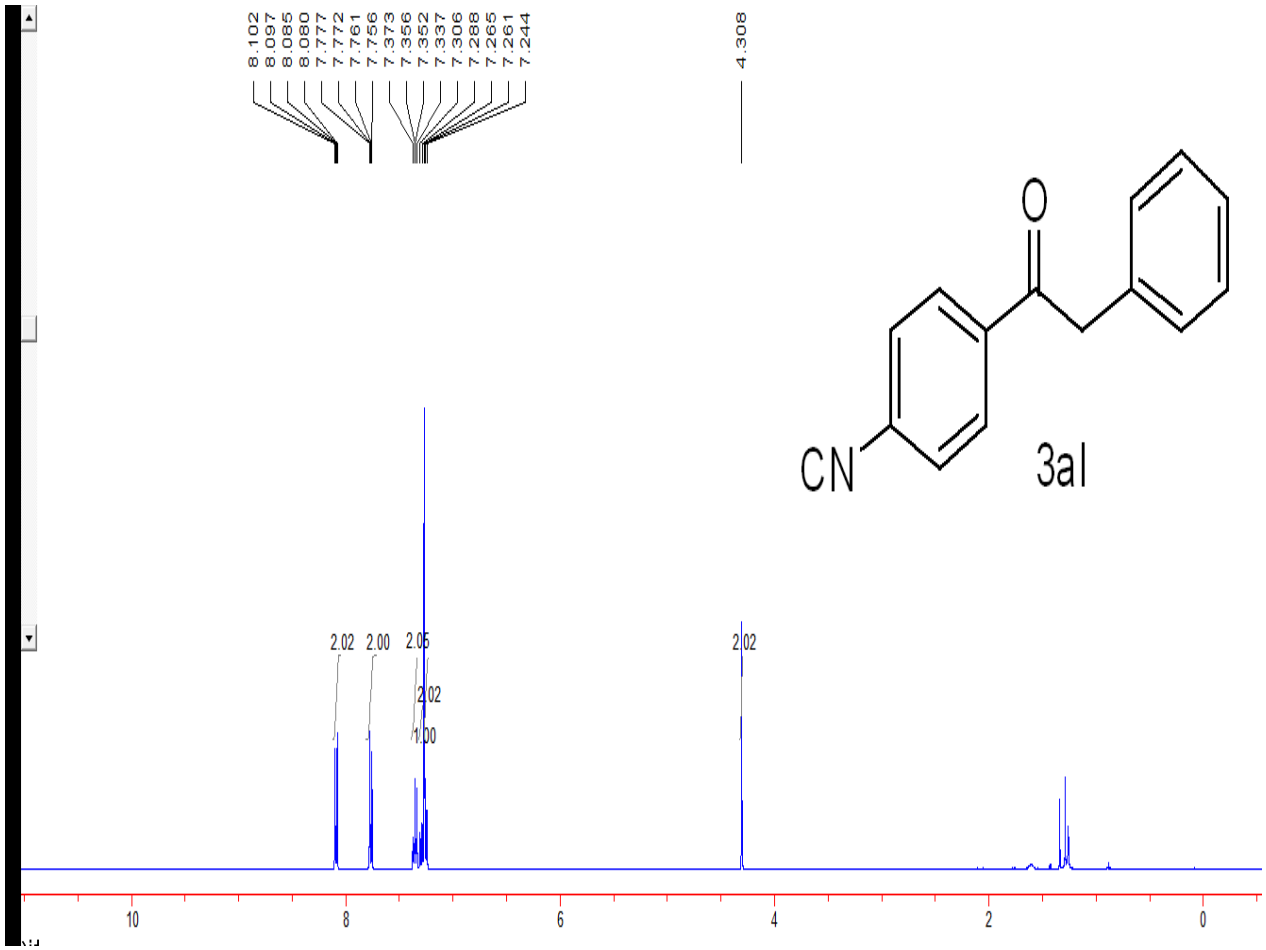
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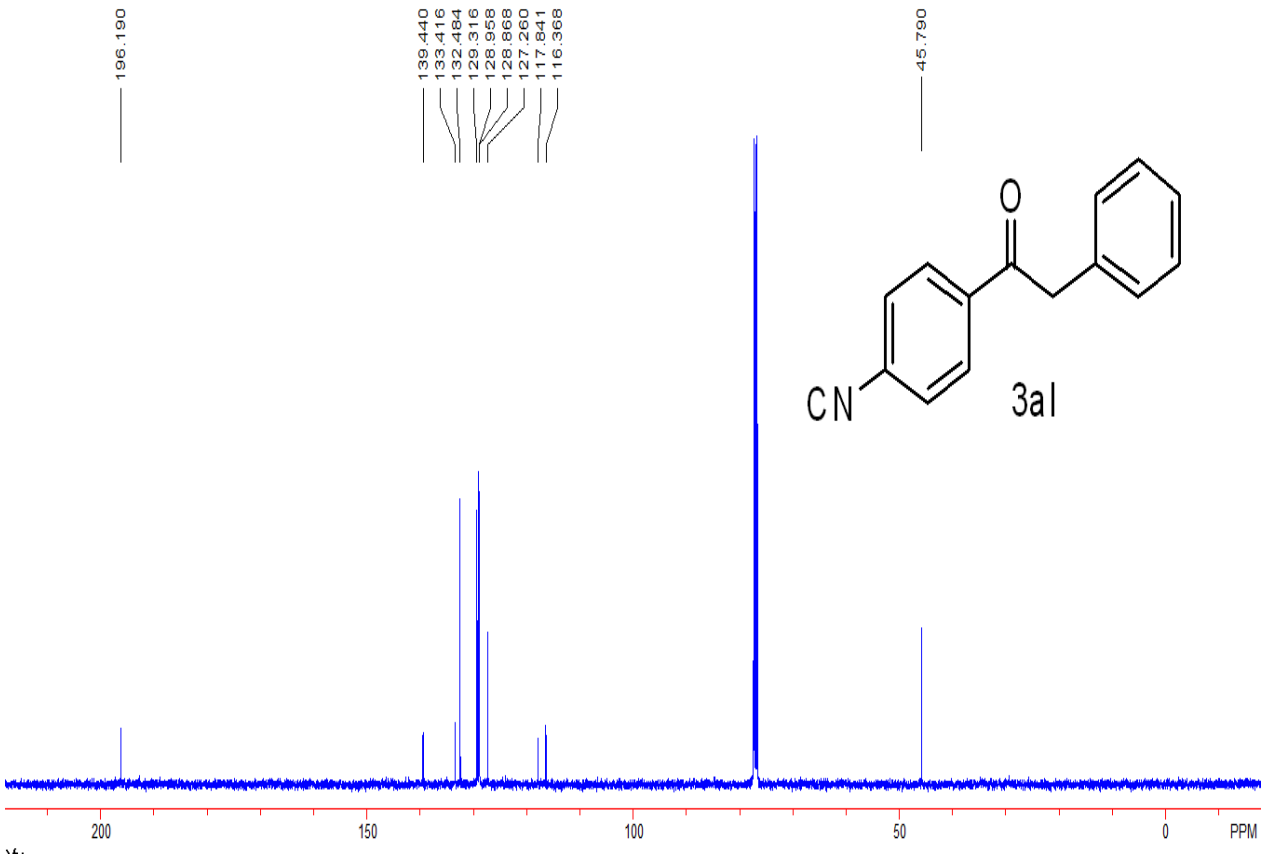
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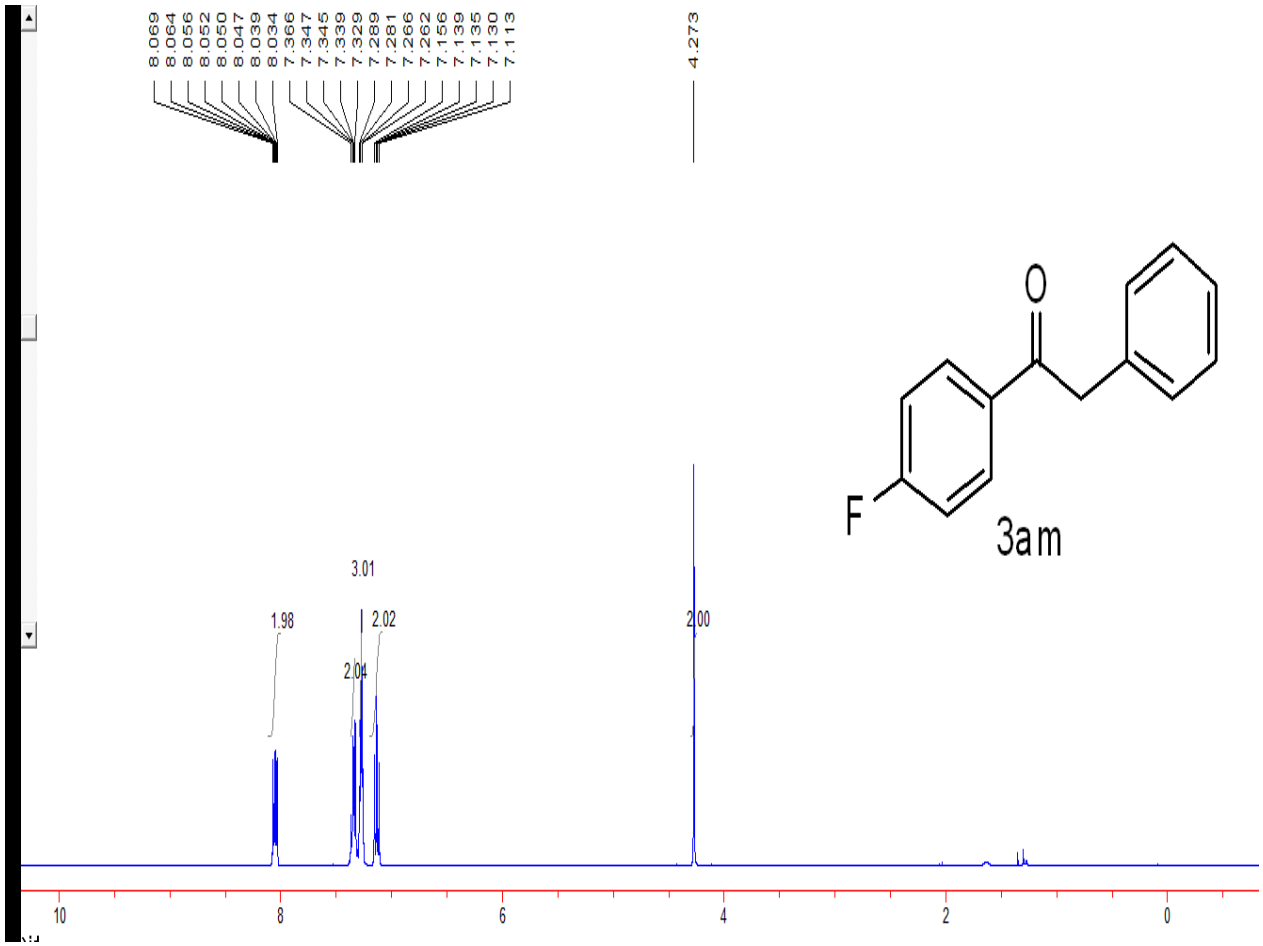
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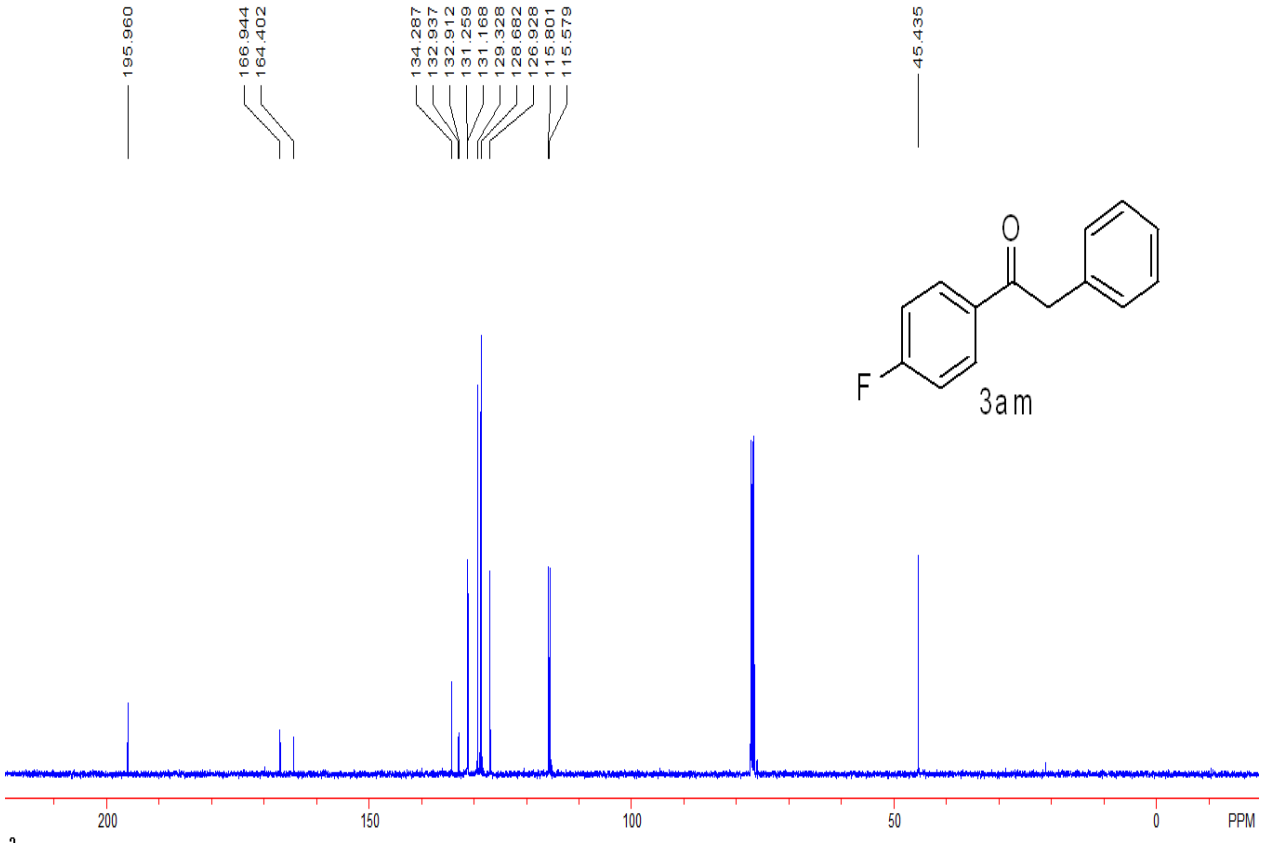
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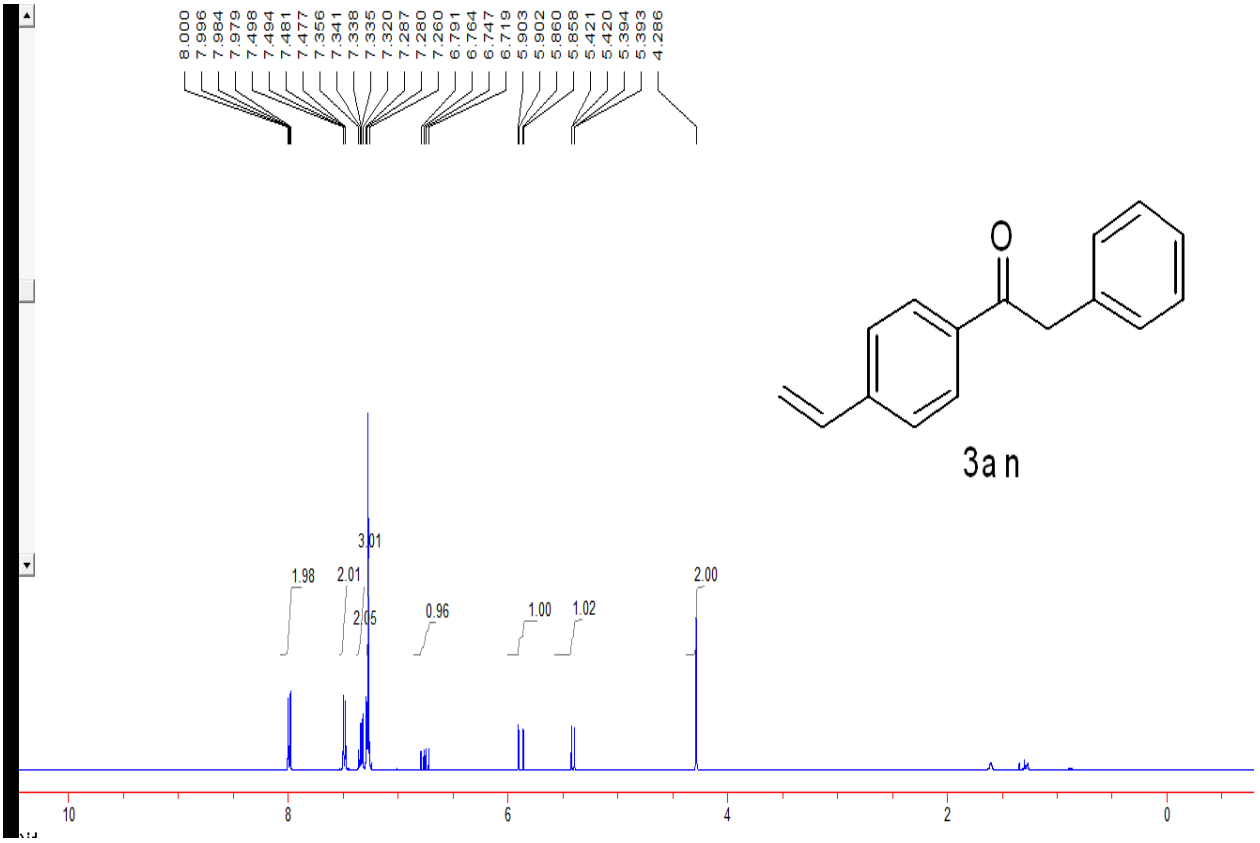
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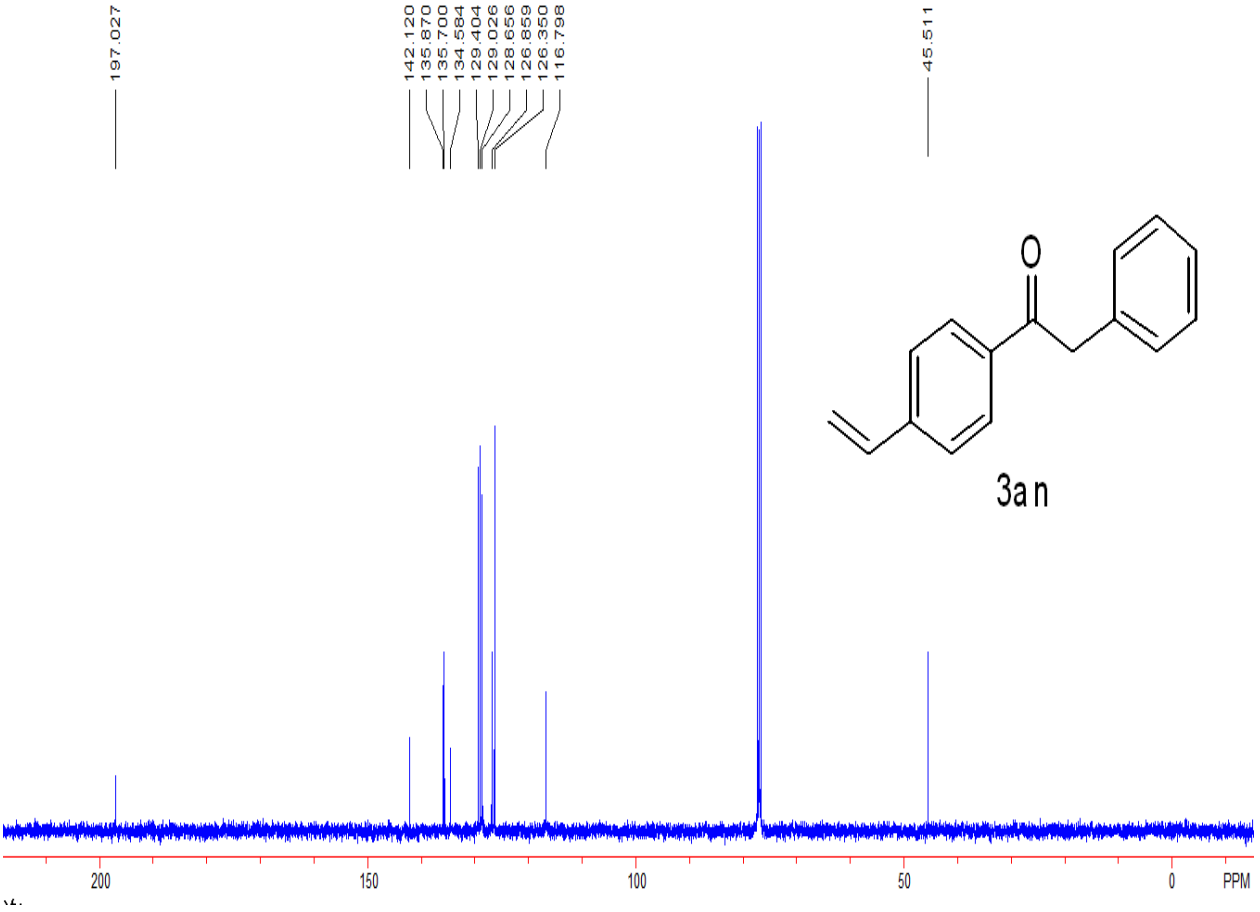
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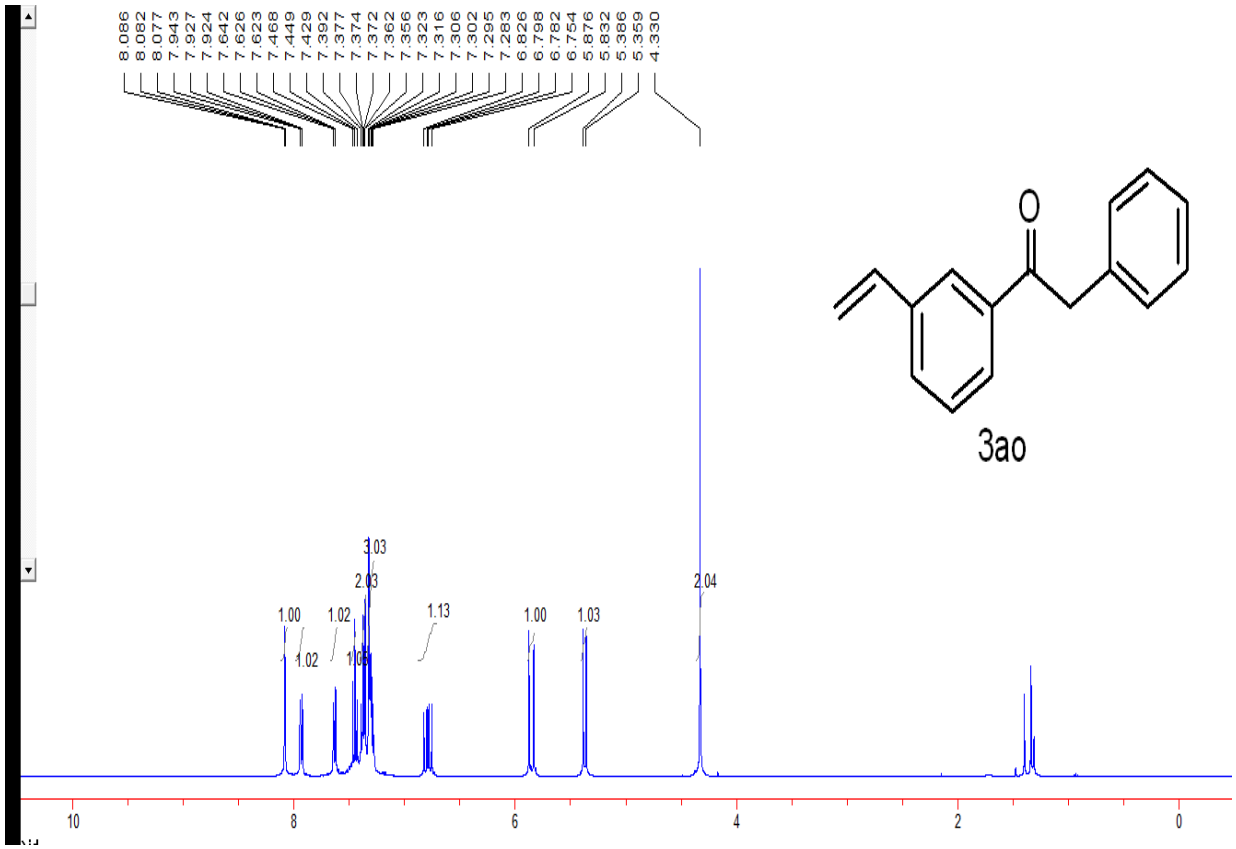
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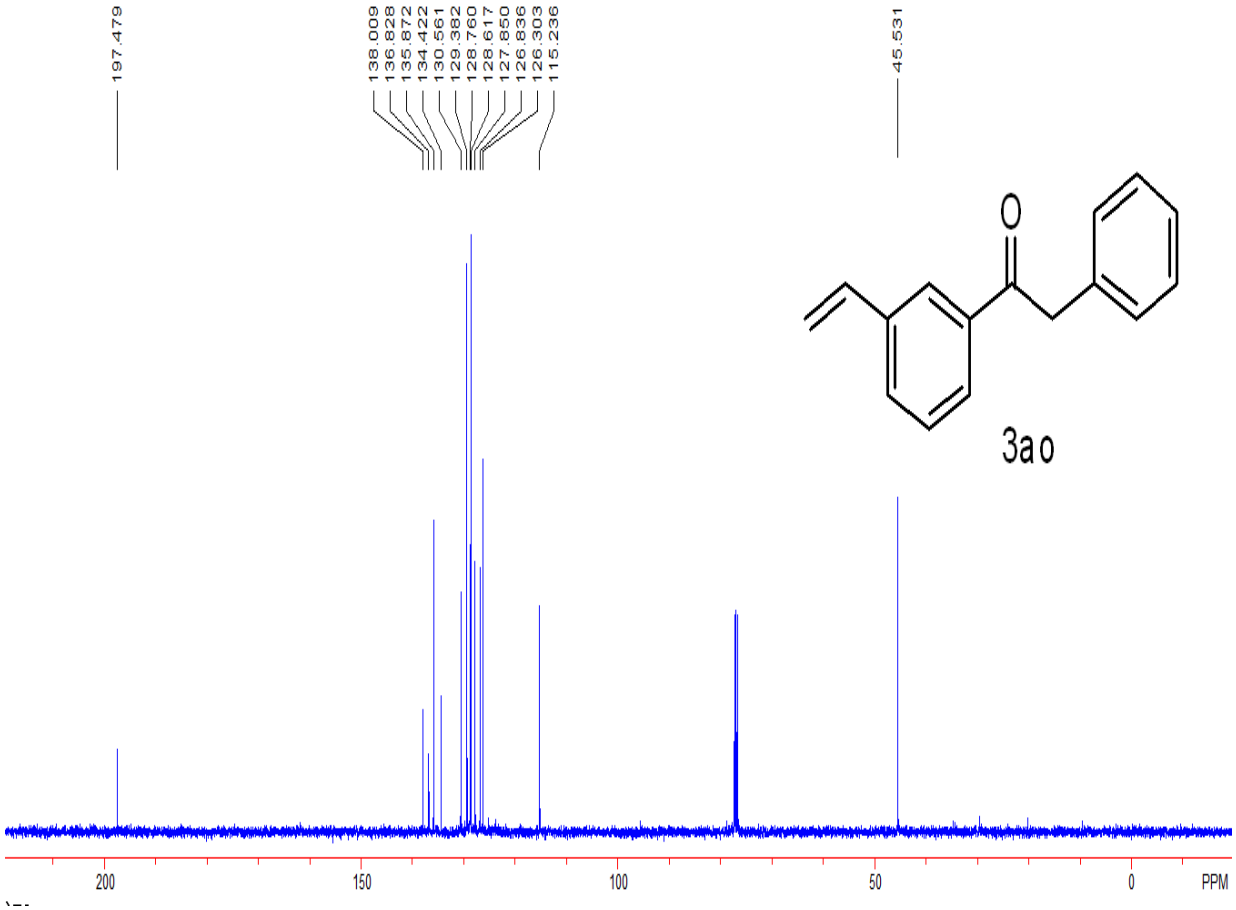
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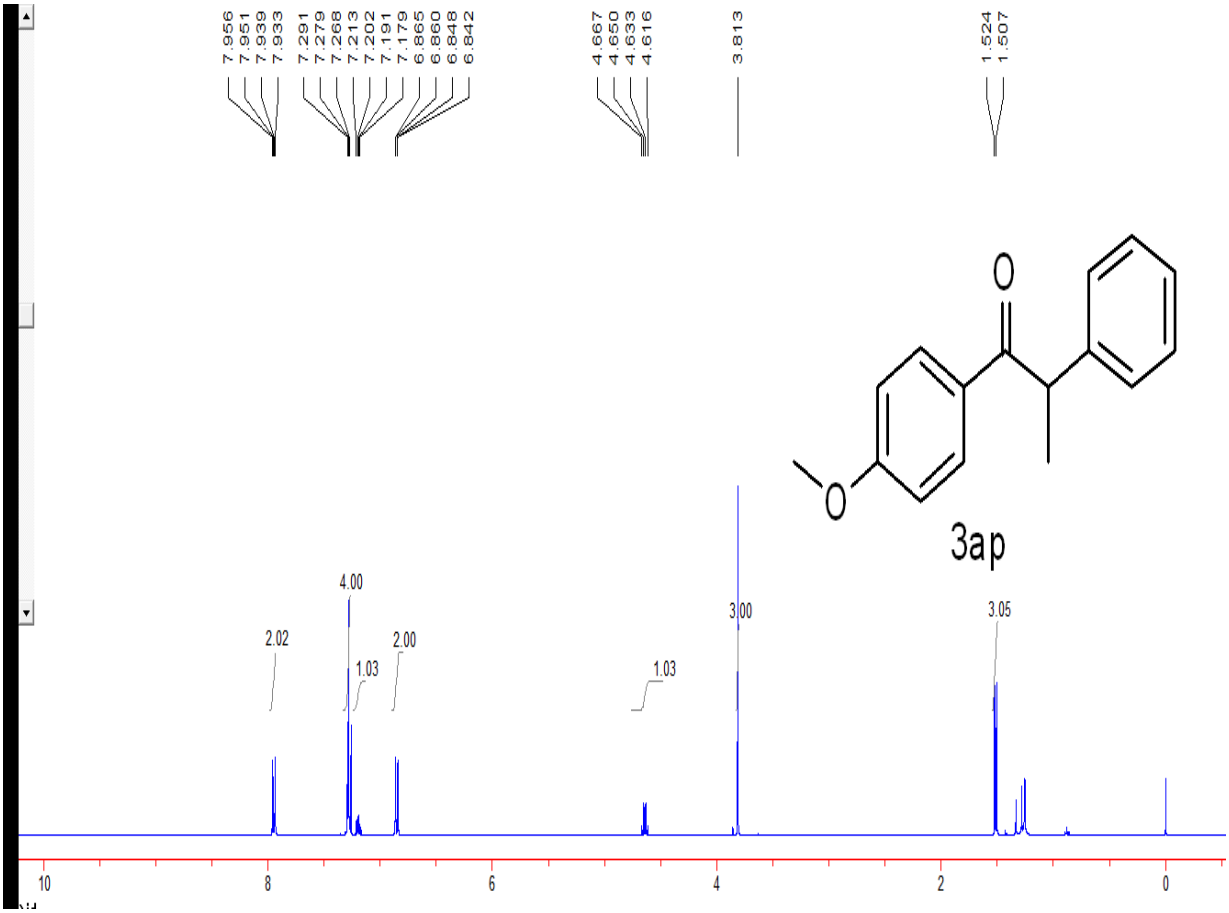
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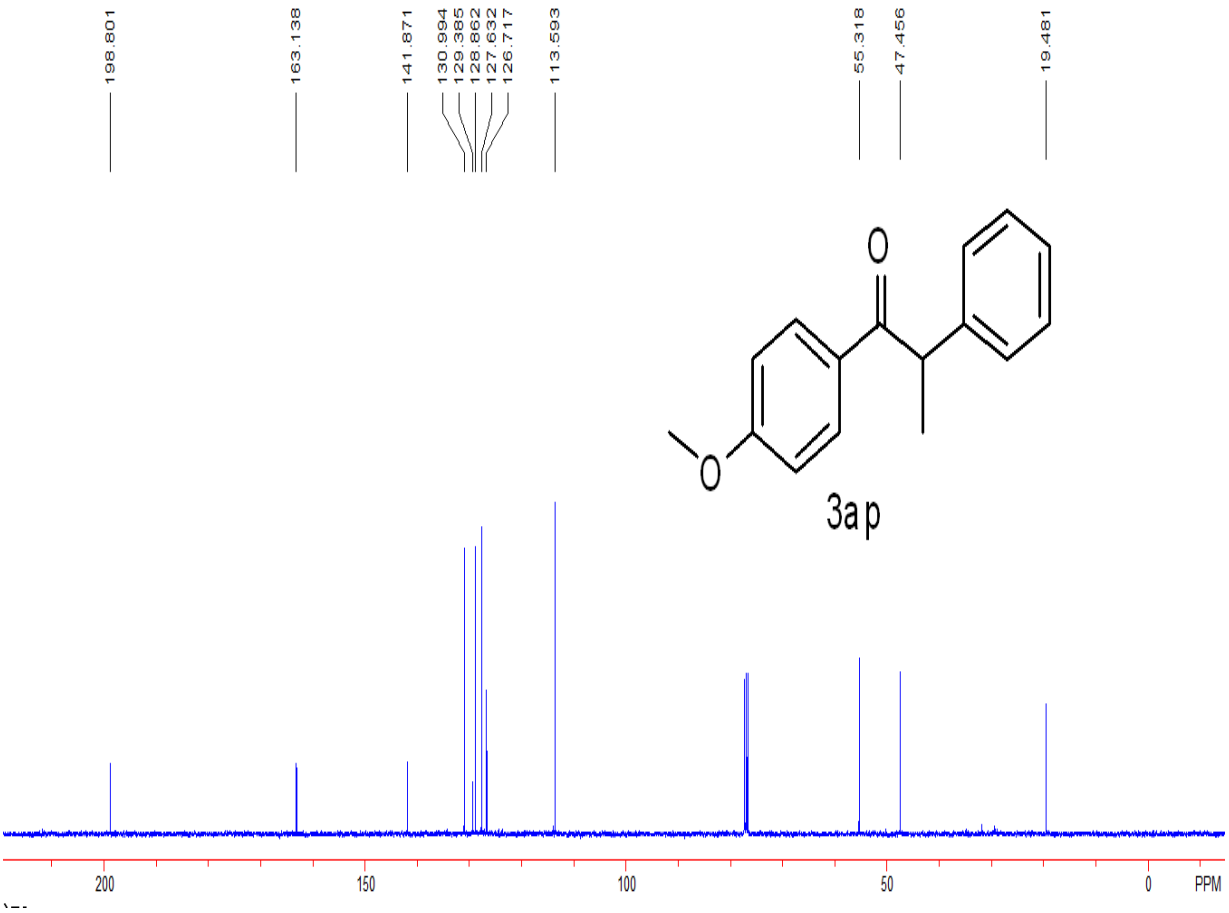
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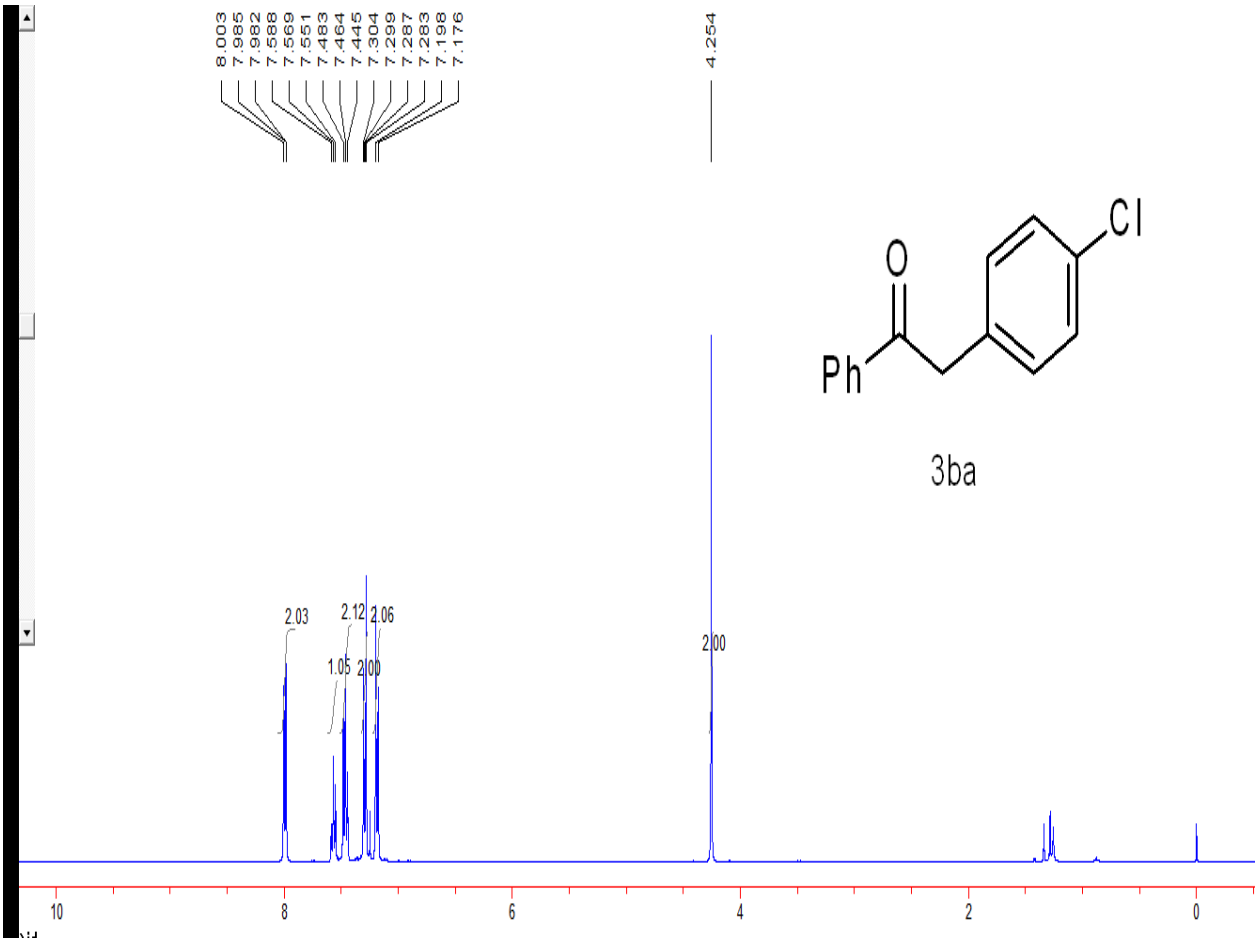
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7.176
4.254
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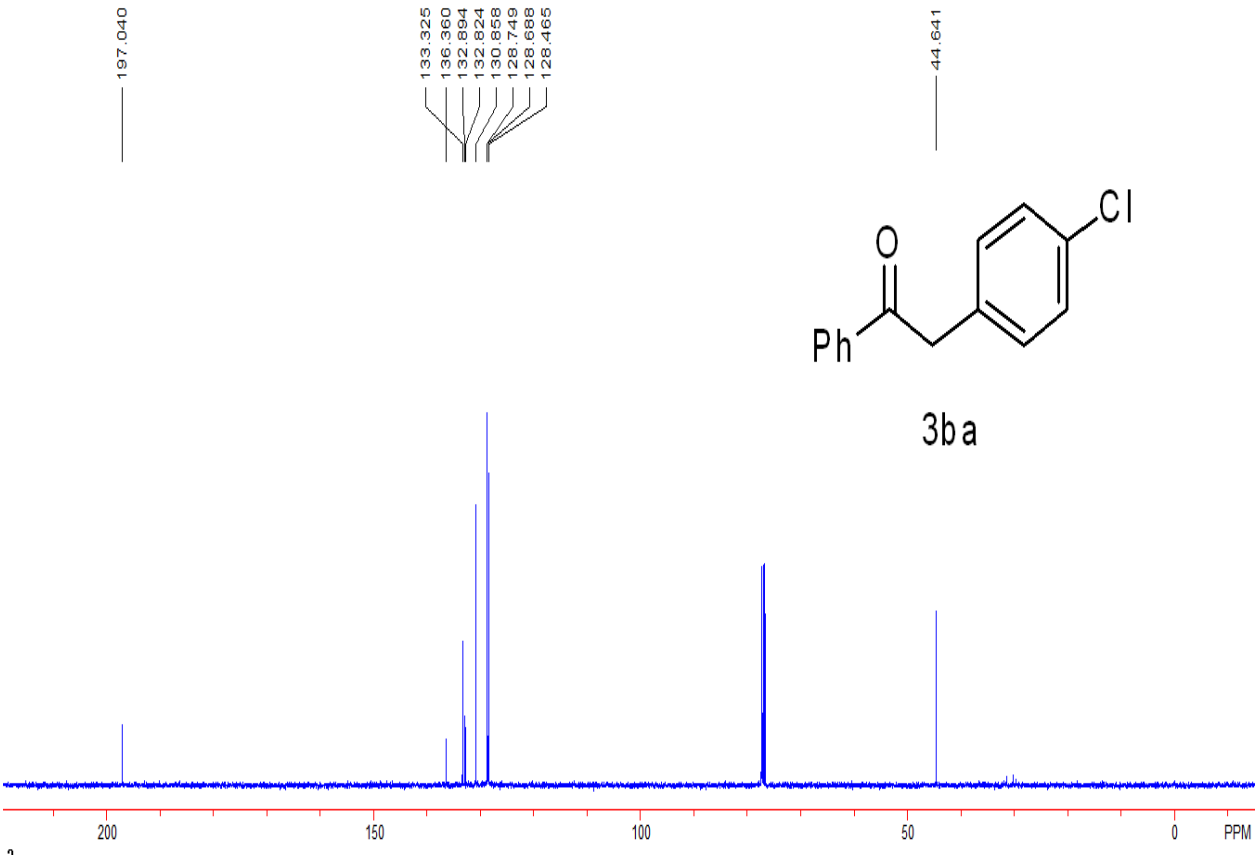
O
Cl
Ph

3ba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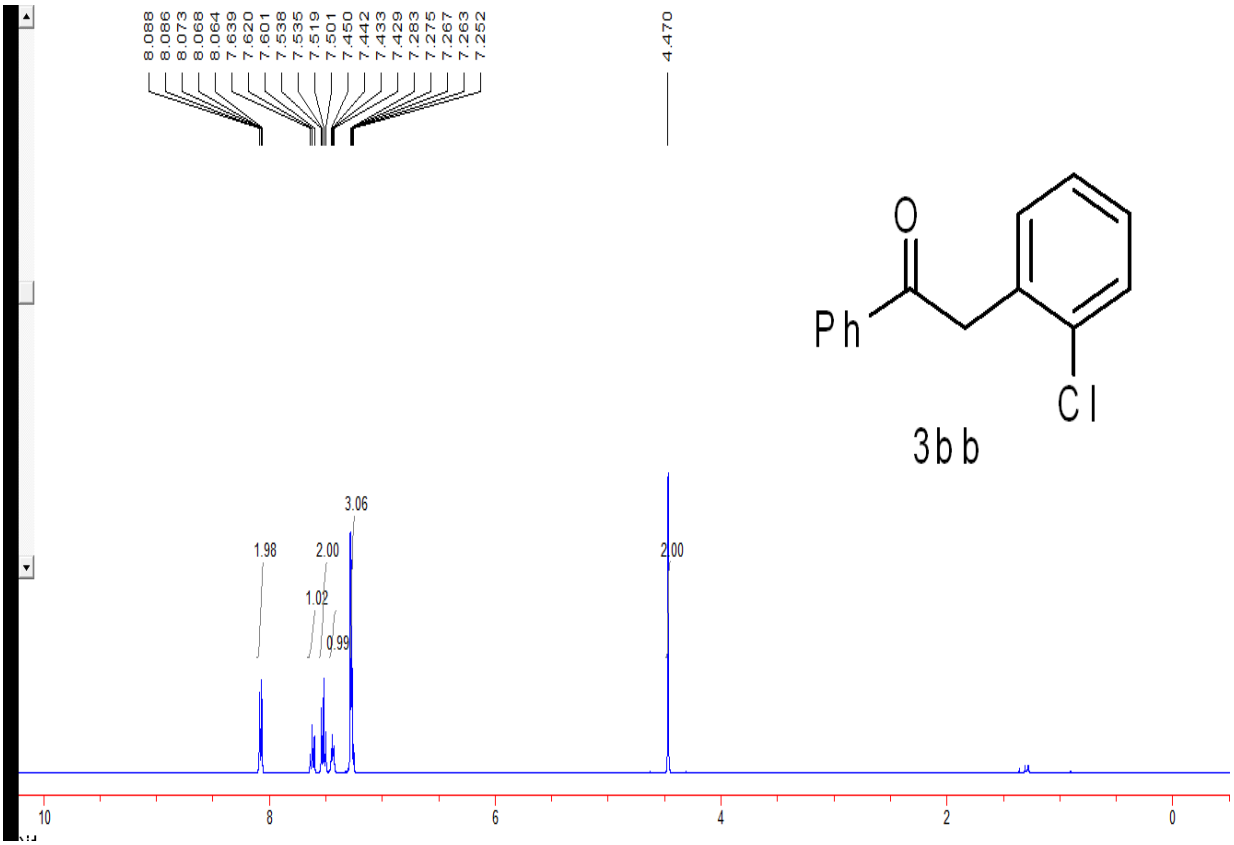
Ph

3ba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7.429
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7.252
4.470
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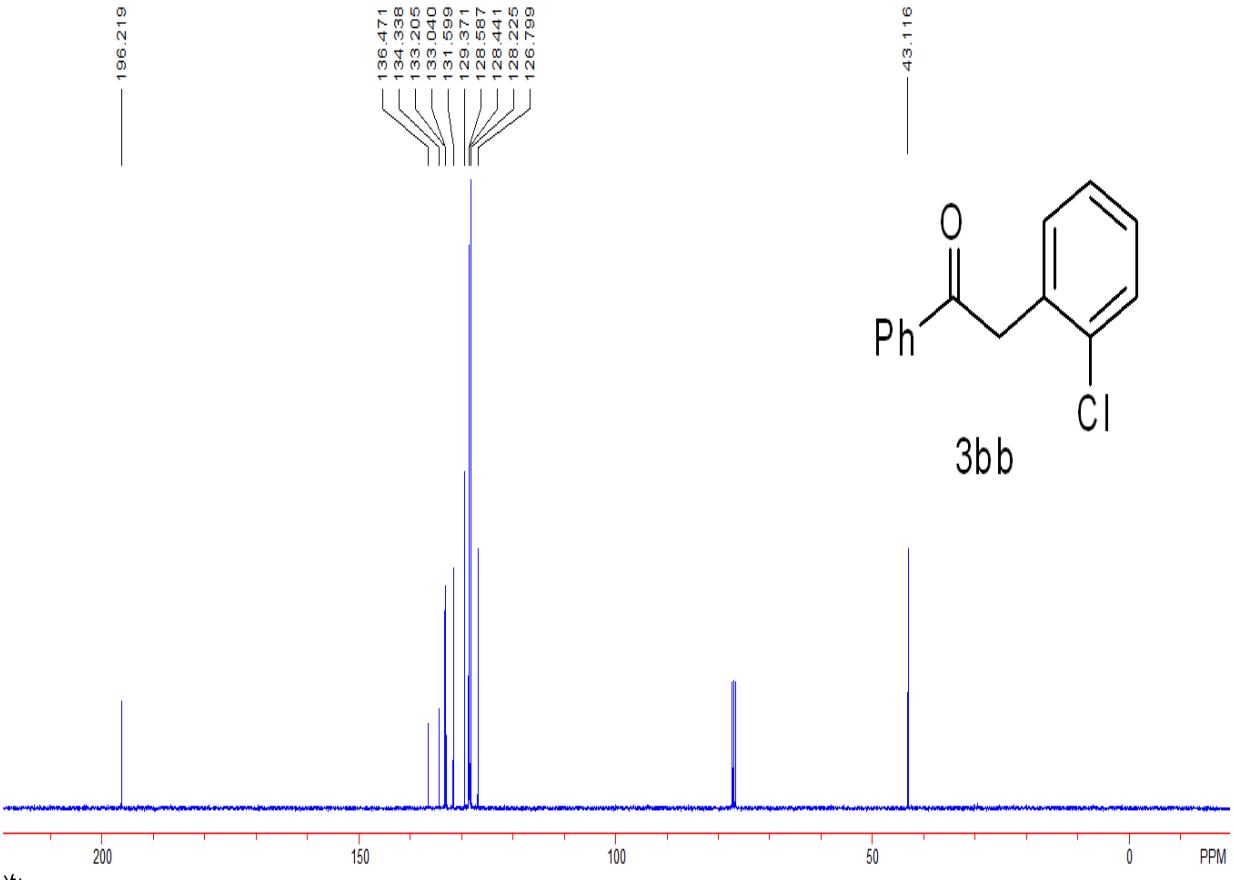
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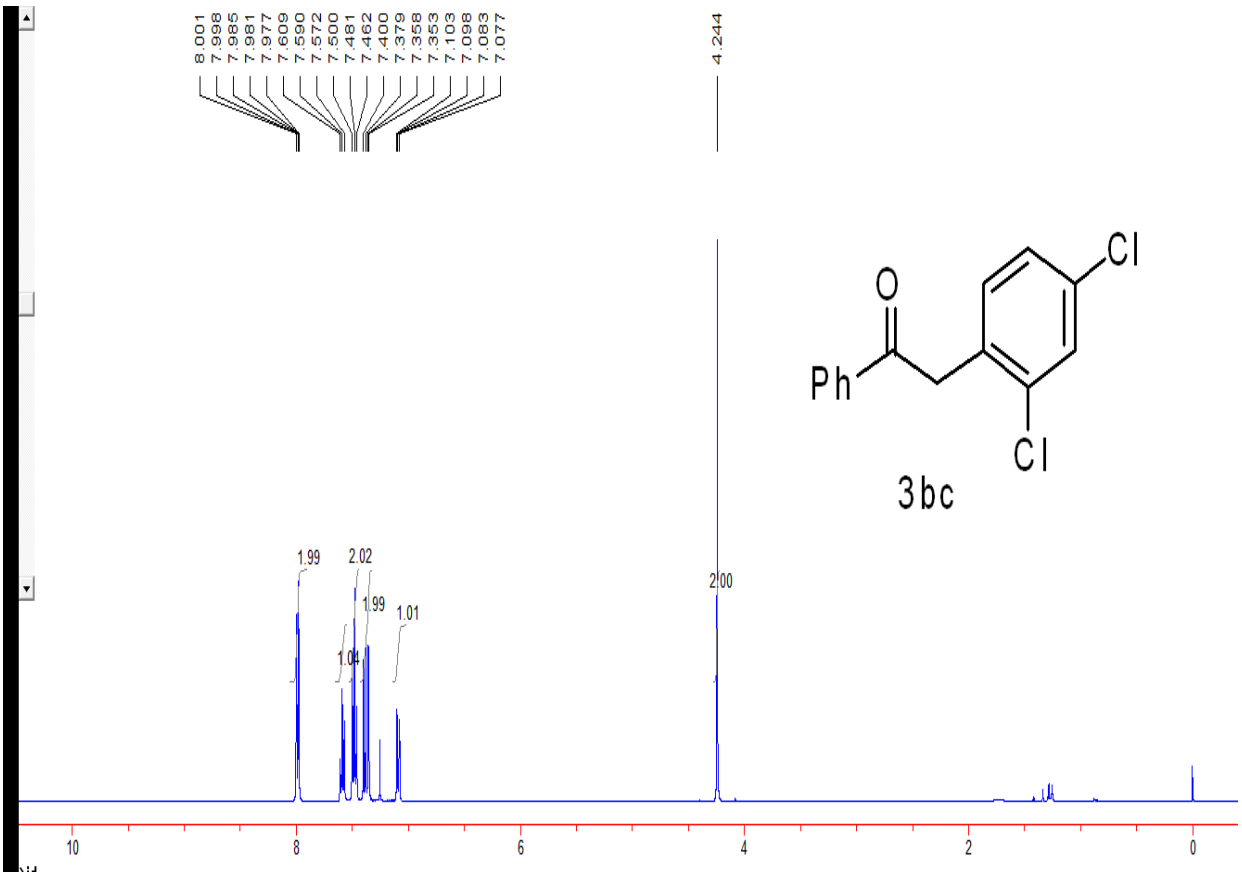
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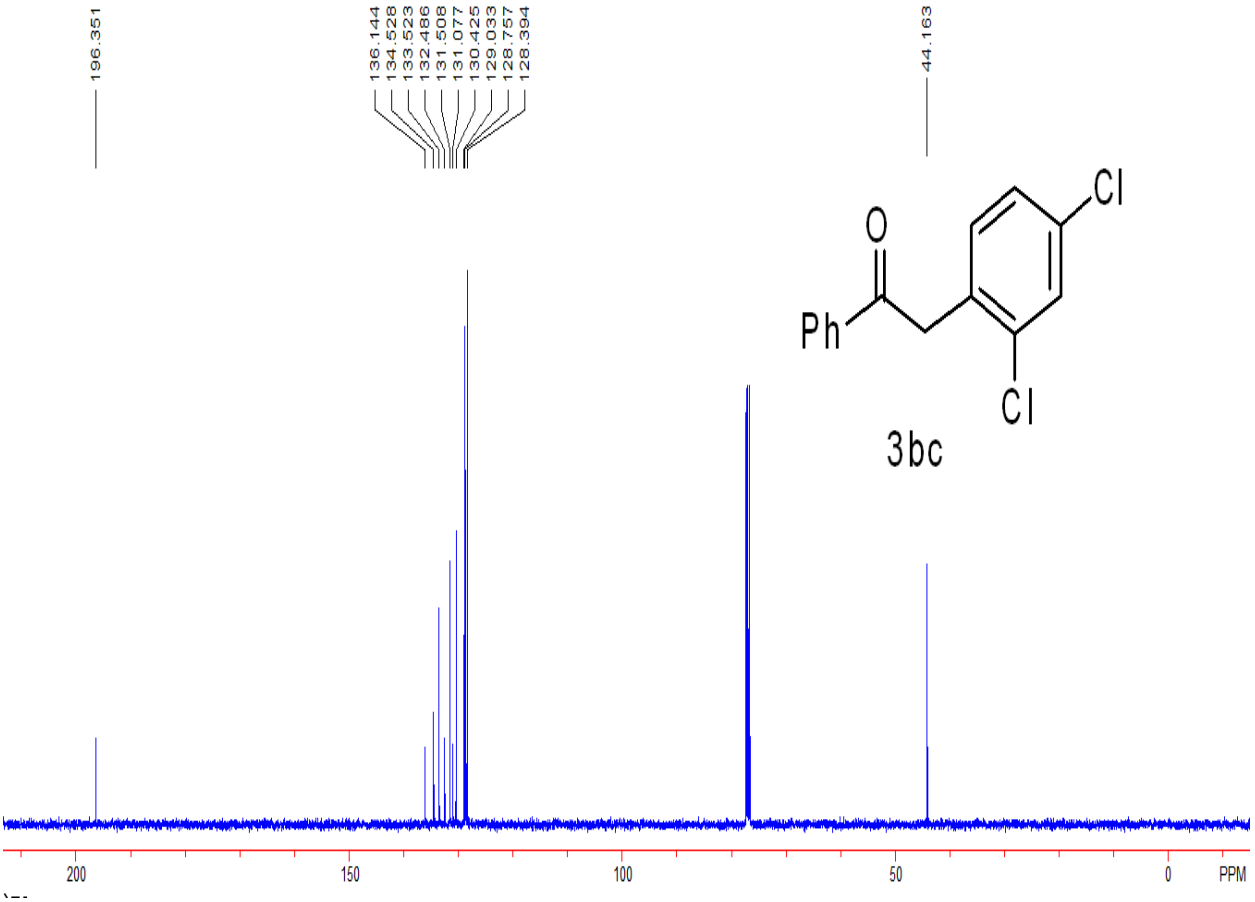
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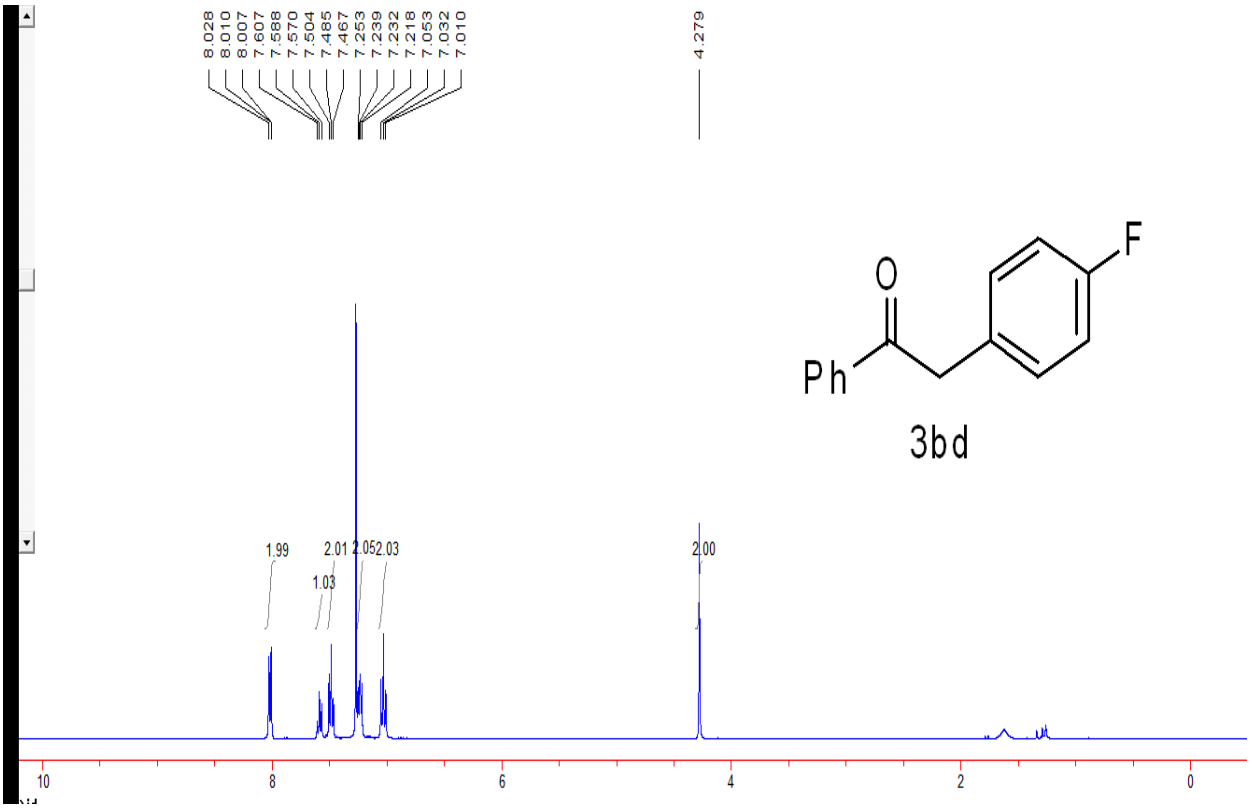
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7.010
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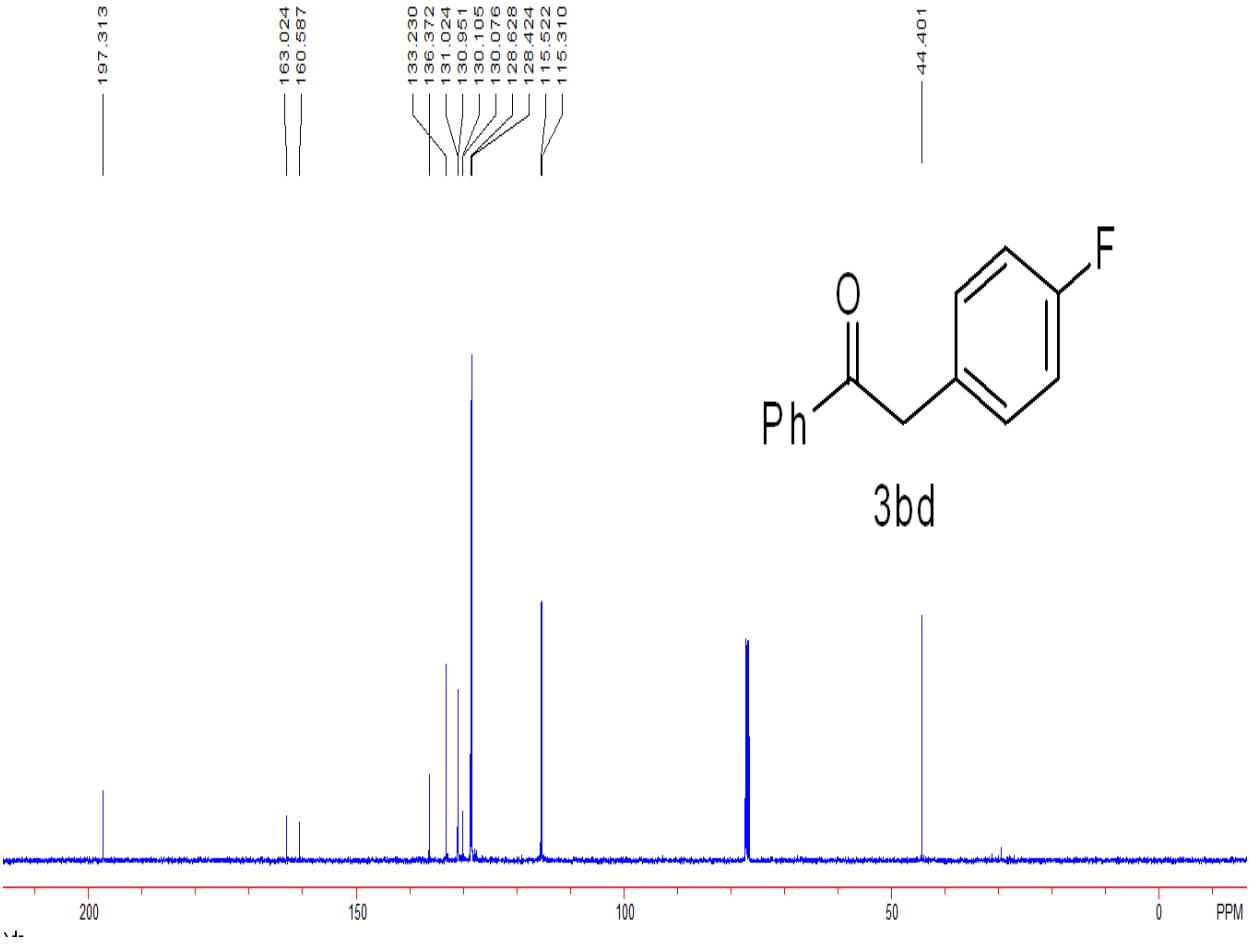
O
F
Ph

3bd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Ph

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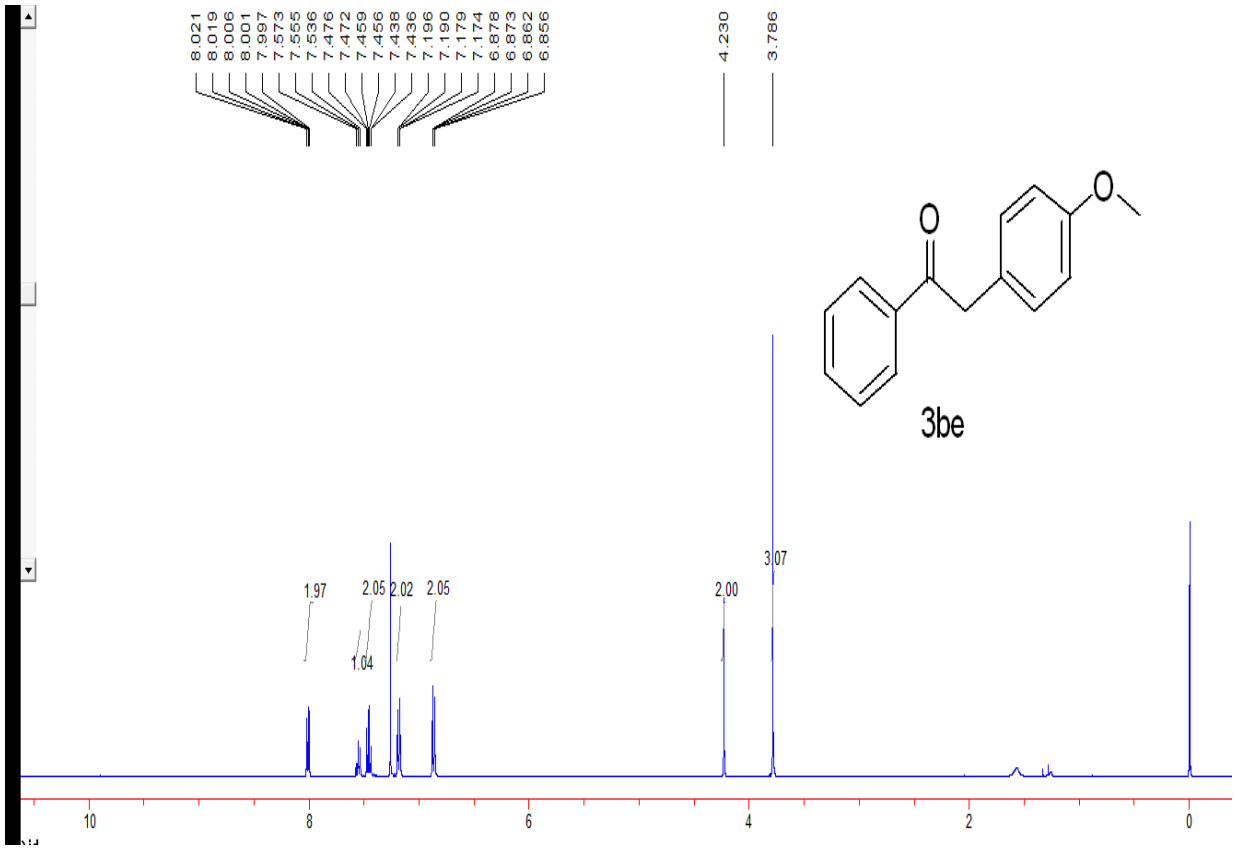
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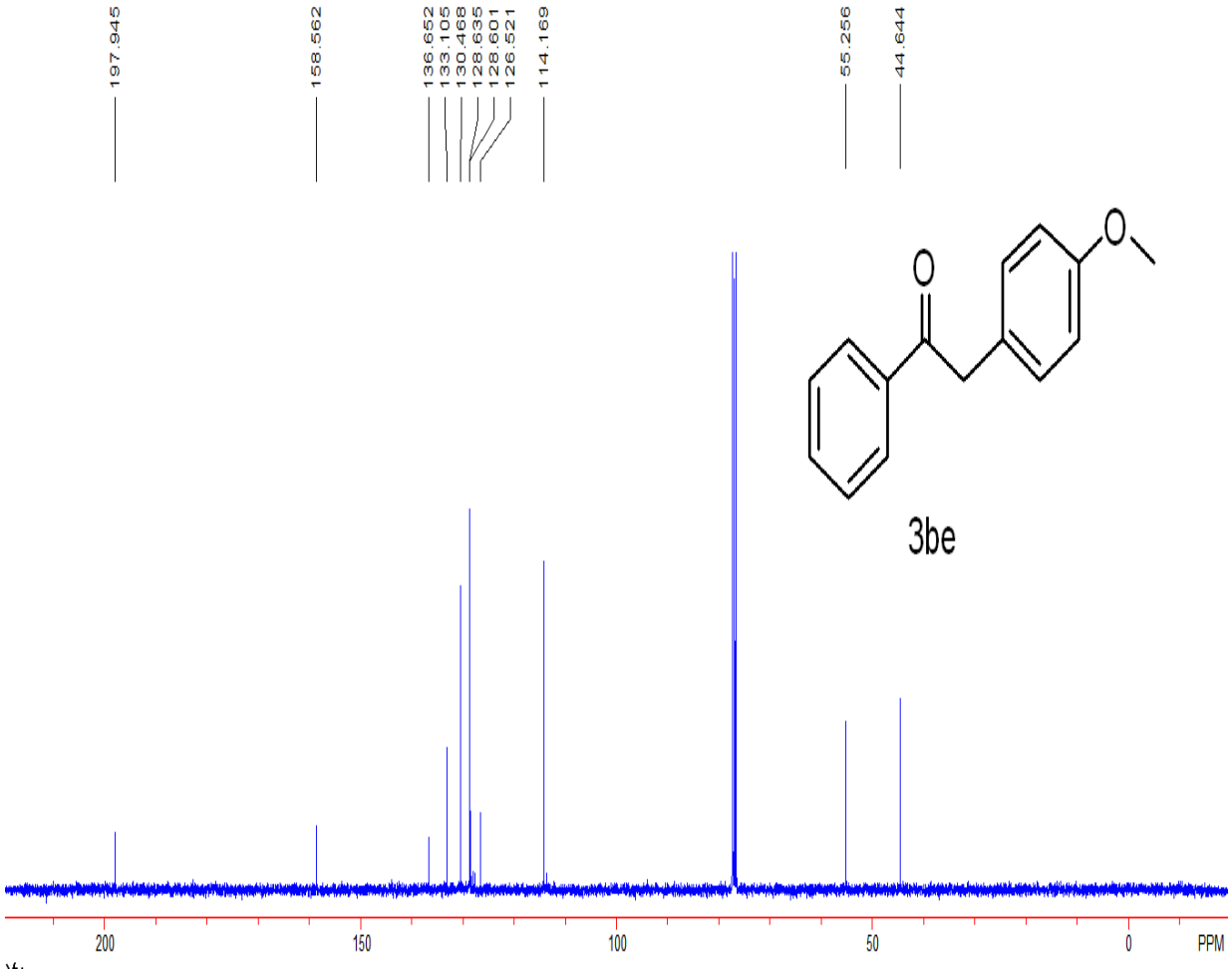
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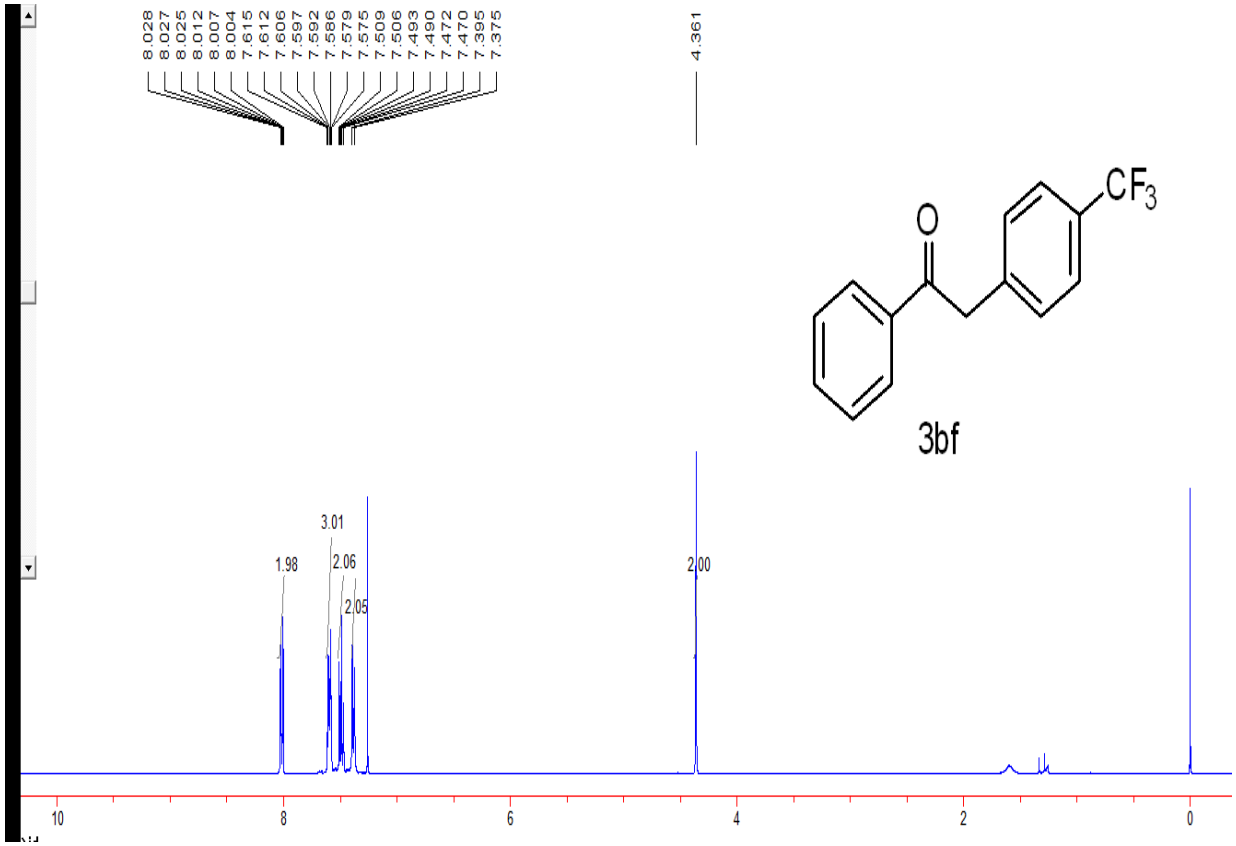
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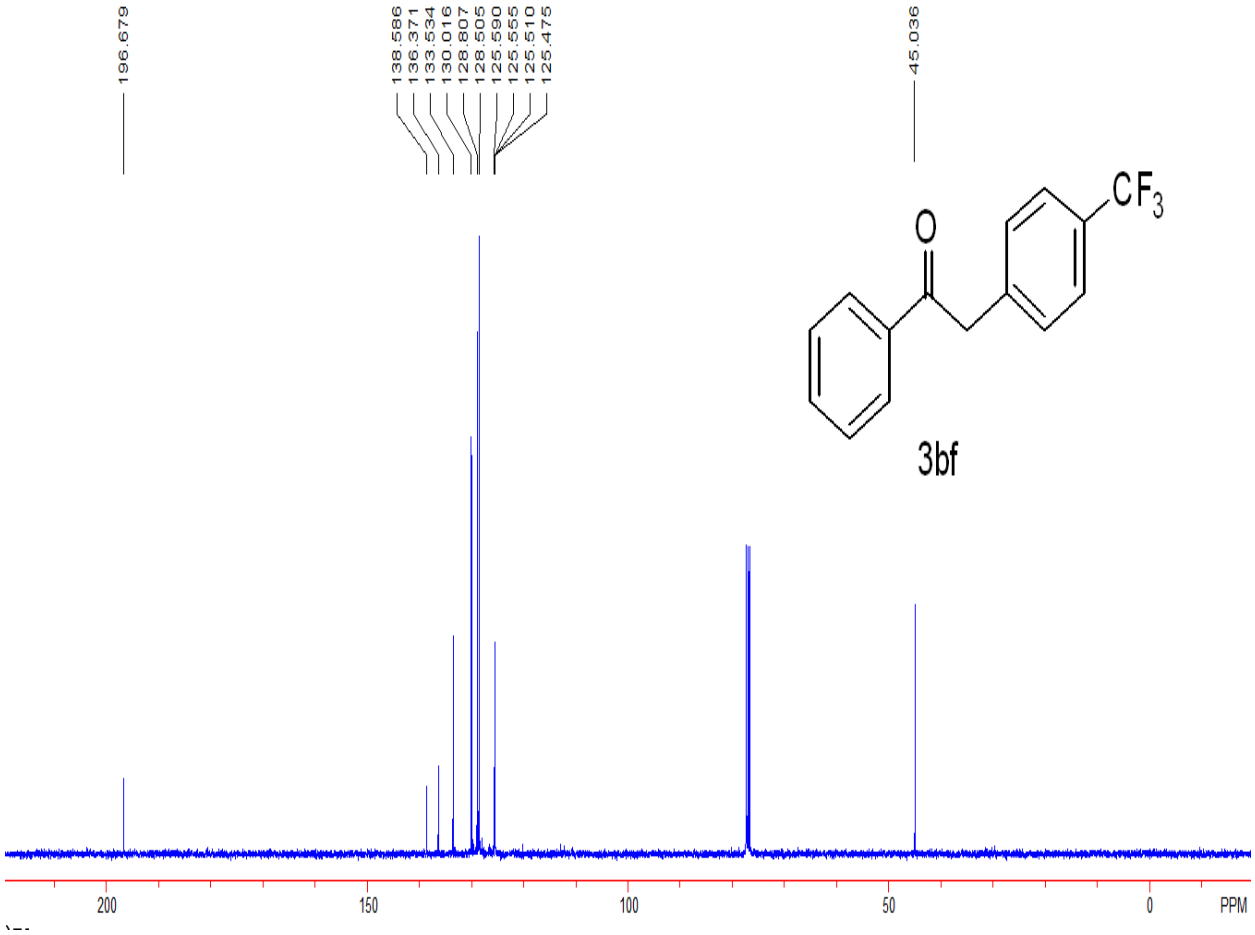
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