Ortho Arylation of Acetanilides *via* Pd(II)-Catalyzed C-H Functionalization

Shangdong Yang, Bijie Li, Xiaobing Wan, and Zhangjie Shi*

Beijing National Laboratory of Molecular Sciences (BNLMS) and Key Laboratory of Bioorganic Chemistry and Molecular Engineering of Ministry of Education, College of Chemistry and Green Chemistry Center, Peking University, Beijing 100871 and State Key Laboratory of Organometallic Chemistry, Chinese Academy of Sciences, Shanghai 200032, China

Supporting Information

General. All the reactions were carried out under air atmosphere. Dioxane was dried by sodium and freshly distilled. Anhydrous $Cu(OTf)_2$ and AgF were purchased from Alfa Aesar and Acros Chemical and used without further purification (Important! the sources of $Cu(OTf)_2$ is very critical in this transformation.). $Pd(OAc)_2$ was purchased from Alfa Aesar Chemical. ^1H NMR (300 MHz) and ^{13}C NMR (75 MHz) were registered on Varian 300M spectrometers with $CDCl_3$ as solvent and tetramethylsilane (TMS) as internal standard. Chemical shifts were reported in units (ppm) by assigning TMS resonance in the ^1H spectrum as 0.00 ppm and $CDCl_3$ resonance in the ^{13}C spectrum as 77.0 ppm. All coupling constants (*J* values) were reported in Hertz (Hz). Column chromatography was performed on silica gel 200-300 mesh. IR, GC, MS, and HRMS were performed by the State-authorized Analytical Center in Peking University.

General procedures for *ortho* arylation of acetanilide:

In a 30 mL Schlenck tube, Acetanilide **1a** (0.2 mmol, 1.0 equiv), $Pd(OAc)_2$ (0.01 mmol, 0.05 equiv), anhydrous $Cu(OTf)_2$ (0.4 mmol, 2.0 equiv) and anhydrous AgF (0.4 mmol, 2.0 equiv) was dissolved in 5.0 mL of dried 1, 4-Dioxane, then (trimethoxyl)phenysilane (80 μL, 0.4 mmol, 2.0 equiv) was added via syringe. The tube was sealed with a Teflon lined cap, and the reaction mixture was stirred and heated to 110 °C in an oil bath for 48 h. After the reaction was completed, the dark solid was removed by filtration through Celite, and the Celite bed was washed with 3 x 5 mL diethyl ether. In most cases, the combined filtrate was immediately concentrated, and the residue was purified by silica gel chromatography with a proper eluent. The purified material was dried under oil-pump vacuum.

***N*-Biphenyl-2-yl-acetamide (3aa).**[1] Following the general procedures, the reaction was performed on a 0.2 mmol scale at 110 °C for 36 h. A white powder was obtained after silica gel chromatography (70:30 hexanes:EtOAc), yield 0.031 g (74%) (With $Cu(OTf)_2$ from different companies, such as Aldrich and Acros, the reaction could take place. However, the starting material **1a** could not completely convert into product **3aa** occasionally and the yield was ranged from 45-70% with the recovered starting material **1a**). ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.24 (d, *J* = 7.80 Hz, 1H), 7.51-7.33 (m, 6H), 7.25-7.15 (m, 3H), 2.01 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.19, 138.11, 134.62, 132.19, 130.01, 129.16, 129.02, 128.35, 127.90, 124.32, 121.68, 24.52; *m/z* (EI) 211 (M^+, 33.3%), 169 (100%); IR (KBr plate, $CDCl_3$) ν 3261, 3031, 1663, 1520, 1445, 1293, 757.

***N*-(4'-Methoxy-biphenyl-2-yl)-acetamide (3ac).**[2] Following the general procedures, the reaction was performed on a 0.2 mmol and 10 mol % $Pd(OAc)_2$ scale at 110 °C for 48 h. A white powder was obtained after silica gel chromatography (50:50 hexanes:EtOAc), yield 0.034 g (71%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.22 (d, *J* = 8.10 Hz, 1H), 7.51 (d, *J* = 7.80 Hz, 1H), 7.36 – 7.18 (m, 6H), 7.01 (d, *J* = 9.00 Hz, 2H), 3.86 (s, 3H), 2.03 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.25, 159.23, 134.71, 131.94, 130.28, 130.07, 128.79, 127.98, 124.31, 123.97, 121.60, 119.75, 114.38, 55.25, 24.43; *m/z* (EI) 241 (M^+, 58.8%), 199 (100%); IR (KBr plate, $CDCl_3$) ν 3267, 1663, 1517, 1445, 1298, 1246, 1179, 1035, 834, 760.

***N*-(4'-Methyl-biphenyl-2-yl)-acetamide (3ad).**[2] Following the general procedures, the reaction was performed on a 0.2 mmol scale at 110 °C for 48 h. A white powder was obtained after silica gel chromatography (65:35 hexanes:EtOAc), yield 0.028 g (62%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.25 (d, *J* = 7.80 Hz, 1H), 7.37 -7.17 (m, 8H), 2.42 (s, 3H), 2.03 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.21, 137.75, 135.01, 134.67, 132.01, 130.04, 129.75, 129.03, 128.17, 124.24, 121.41, 24.61, 21.18; *m/z* (EI) 225 (M^+, 46.9%), 183 (100%); IR (KBr plate, $CDCl_3$) ν 3264, 3024, 1663, 1520, 1445, 1303, 1289, 821, 757.

***N*-(4'-Fluoro-biphenyl-2-yl)-acetamide (3ae).**[2] Following the general procedures, the reaction was performed on a 0.2 mmol scale at 110 °C for 48 h. A white powder was obtained after silica gel chromatography (65:35 hexanes:EtOAc), yield 0.029 g (63%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.20 (d, *J* = 8.40 Hz, 1H), 7.40-7.32 (m, 3H), 7.27-7.15 (m, 4H), 7.06 (s, 1H), 2.03 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.29, 164.06, 160.77, 134.60, 134.05, 131.47, 130.96, 130.86, 130.09, 128.54, 124.55, 122.09, 116.16, 115.88, 115.75, 24.50; *m/z* (EI) 229 (M^+, 98.2%), 185 (100%); IR (KBr plate, $CDCl_3$) ν 3259, 3039, 1663, 1515, 1446, 1222, 1159, 838, 759.

***N*-(4'-Chloro-biphenyl-2-yl)-acetamide (3af).** Following the general procedures, the reaction was performed on a 0.2 mmol scale at 110 °C for 48 h. A white powder was obtained after silica gel chromatography (65:35 hexanes:EtOAc), yield 0.024 g (46%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.16 (8.16, *J* = 8.10 Hz, 1H), 7.47 – 7.27 (m, 5H), 7.20 (d, *J* = 6.60 Hz, 2H), 7.09 (s, 1H), 2.03 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.35, 136.56, 134.44, 133.99, 131.42, 130.52, 129.95, 129.19, 128.68, 124.69, 122.36, 24.44; *m/z* (EI) 247 (M^+, ^{37}Cl, 12.5%), 245 (M^+, ^{35}Cl, 34.1%), 203 (100%); IR (KBr plate, $CDCl_3$) ν 3248,

3027, 1652, 1524, 1291, 857, 701. Anal. Calcd. for $C_{14}H_{12}NOCl$: C, 68.44; H, 4.92; N, 5.70. Found: C, 68.29; H, 4.99; N, 5.59.

***N*-(3'-Methoxy-biphenyl-2-yl)-acetamide (3ag).**[2] Following the general procedures, the reaction was performed on a 0.2 mmol scale at 110 °C for 48 h. A white powder was obtained after silica gel chromatography (50:50 hexanes:EtOAc), yield 0.025 g (52%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.28 (d, *J* = 8.10 Hz, 1H), 7.39 (m, 2H), 7.34-7.17 (m, 3H), 6.97-6.89 (m, 3H), 3.84 (s, 3H), 2.03 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.24, 160.02, 139.43, 134.62, 131.83, 130.08, 129.87, 128.43, 124.20, 121.38, 121.30, 114.71, 113.54, 55.29, 24.65; *m/z* (EI) 241 (M^+, 52.7%), 199 (100%); IR (KBr plate, $CDCl_3$) ν 3267, 1664, 1586, 1522, 1442, 1303, 759.

***N*-Biphenyl-2-yl-benzamide (3ca).** Following the general procedures, the reaction was performed on a 0.2 mmol scale at 110 °C for 48 h. A white powder was obtained after silica gel chromatography (90:10 hexanes:EtOAc), yield 0.021 g (38%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.53 (d, *J* = 8.40 Hz, 1H), 8.01 (s, 1H), 7.60 (d, *J* = 7.20 Hz, 2H), 7.54 -7.29 (m, 8H), 7.24-7.19 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 164.97, 138.02, 134.87, 134.73, 132.33, 131.69, 129.96, 129.51, 129.32, 129.19, 128.98, 128.71, 128.57, 128.15, 126.77, 124.35, 121.13, 115.33; *m/z* (EI) 273 (M^+, 55.4%), 105 (100%); IR (KBr plate, $CDCl_3$) ν 3295, 1664, 1581, 1519, 1489, 1449, 1306, 1073, 1027, 796, 759, 702; Anal. Calcd. For $C_{19}H_{15}NO$: C, 83.49; H, 5.53; N, 5.12. Found: C, 83.27; H, 5.63; N, 4.95.

***N*-Biphenyl-2-yl-3-phenyl-propionamide (3ha).** Following the general procedures, the reaction was performed on a 0.2 mmol scale at 110 °C for 48 h. A white powder was obtained after silica gel chromatography (90:10 hexanes:EtOAc), yield 0.028 g (46%).^{1}H NMR (300 MHz, $CDCl_3$) δ 8.29 (d, *J* = 8.10 Hz, 1H), 7.43-7.29 (m, 5H), 7.27 - 7.14 (m, 8H), 7.07 (s, 1H), 2.95 (d, *J* = 7.05 Hz, 2H), 2.47 (d, *J* = 7.65 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.11, 140.50, 137.95, 134.50, 132.05, 129.98, 129.83, 129.16, 129.02, 128.77, 128.54, 128.36, 128.30, 128.14, 127.87, 127.24, 126.28, 124.27, 121.48, 39.54, 31.35; *m/z* (EI) 301 (M^+, 98.8%), 167 (100%); IR (KBr plate, $CDCl_3$) ν 3281, 3025, 1662, 1584, 1518, 1494, 1478, 1447, 1278, 1218, 1075, 751, 699; Anal. Calcd. For $C_{21}H_{19}NO$: C, 83.69; H, 6.35; N, 4.65. Found: C, 83.54; H, 6.46; N, 4.81.

***N*-(5-Methoxy-biphenyl-2-yl)-acetamide (3ja).** Following the general procedures, the reaction was performed on a 0.2 mmol scale at 110 °C for 48 h. A white powder was obtained after silica gel chromatography (70:30 hexanes:EtOAc), yield 0.033 g (68%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.98 (d, *J* = 5.40 Hz, 1H), 7.49-7.35 (m, 5H), 6.97-6.88 (m, 2H), 6.80 (d, *J* = 2.40 Hz, 1H), 3.81 (s, 3H), 1.99 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.41, 156.41, 138.19, 134.70, 129.11, 129.02, 128.89, 127.92, 127.61, 124.33, 115.35, 113.36, 55.46, 24.19; *m/z* (EI) 241 (M^+, 58.8%), 199 (100%); IR (KBr plate, $CDCl_3$) ν 3285, 1669, 1522, 1442, 1315, 1172, 1126, 1115, 1035, 758; Anal. Calcd. For $C_{15}H_{15}NO_2$: C, 74.67; H, 6.27; N, 5.81. Found: C, 74.54; H, 6.20; N, 5.68.

Acetic acid 6-acetylamino-biphenyl-3-yl ester (3ka). Following the general procedures, the reaction was performed on a 0.2 mmol scale at 110 °C for 48 h. A white powder was obtained after silica gel chromatography (90:10 hexanes:EtOAc), yield 0.034 g (64%). ^{1}H

NMR (300 MHz, $CDCl_3$) δ 8.29 (d, *J* = 8.70 Hz, 1H), 7.50-7.43 (m, 3H), 7.38(d, *J* = 8.10 Hz, 2H), 7.12 -7.01 (m, 2H), 7.00 (s, 1H), 2.30 (s, 3H), 2.03 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.24, 165.17, 149.26, 137.18, 133.62, 133.40, 132.38, 130.11, 129.31, 129.11, 128.54, 128.27, 123.11, 122.80, 17.86, 16.52; *m/z* (EI) 269 (M^+, 50.6%) , 167 (100%); HRMS: Anal. Calcd. for $C_{16}H_{15}NO_3$ 269.10519. Found: 269.10537; IR (KBr plate, $CDCl_3$) ν 3280, 1736, 1669, 1518, 1451, 1247, 800, 754, 703.

Benzoic acid 6-acetylamino-biphenyl-3-yl ester (3la). Following the general procedures, the reaction was performed on a 0.2 mmol scale at 110 °C for 48 h. A white powder was obtained after silica gel chromatography (90:10 hexanes:EtOAc), yield 0.042 g (64%). ^1H NMR (300 MHz, $CDCl_3$) δ 8.32 (d, *J* = 8.40 Hz, 1H), 7.19 (d, *J* = 10.20 Hz, 2H), 7.66 -7.53 (m, 1H), 7.52 -7.38 (m, 6H), 7.23 -7.13 (m, 3H), 2.03 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.24, 165.17, 146.96, 137.18, 133.62, 133.40, 132.38, 130.11, 129.31, 129.11, 128.54, 128.27, 123.11, 122.80, 121.36, 24.52; *m/z* (EI) 331 (M^+, 48.3%), 105 (100%); HRMS: Anal. Calcd. for $C_{21}H_{17}NO_3$ 331.12084. Found: 331.12101; IR (KBr plate, $CDCl_3$) ν 3280, 1736, 1669, 1518, 1451, 1247, 800, 754, 703.

***N*-(5-Methyl-biphenyl-2-yl)-acetamide (3ma).**[2] Following the general procedures, the reaction was performed on a 0.2 mmol scale at 110 °C for 48 h. A white powder was obtained after silica gel chromatography (70:30 hexanes:EtOAc), yield 0.032 g (71%). ^1H NMR (300 MHz, $CDCl_3$) δ 8.04 (d, *J* = 8.10 Hz, 1H), 7.49-7.33 (m, 5H), 7.18-7.11 (m, 2H), 7.06 (s, 1H), 2.34 (s, 3H), 1.99 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.26, 138.24, 134.07, 132.50, 131.93, 130.56, 129.08, 128.87, 128.81, 127.73, 122.09, 24.36, 20.79; *m/z* (EI) 225 (M^+, 40.0%), 183 (100%); IR (KBr plate, $CDCl_3$) ν 3281, 3023, 1655, 1525, 1443, 1298, 1133, 830, 754.

***N*-(5-Chloro-biphenyl-2-yl)-acetamide (3na).**[2] Following the general procedures, the reaction was performed on a 0.2 mmol scale at 110 °C for 48 h. A white powder was obtained after silica gel chromatography (70:30 hexanes:EtOAc), yield 0.030 g (61%). ^1H NMR (300 MHz, $CDCl_3$) δ 8.24 (d, *J* = 8.70 Hz, 1H), 7.51-7.45 (m, 3H), 7.37-7.31 (m, 3H), 7.25 (d, *J* = 8.70 Hz, 1H), 7.23 (s, 1H), 2.02 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.24, 136.75, 133.58, 133.25, 129.75, 129.49, 129.24, 128.99, 128.46, 128.22, 122.78, 114.44, 24.55; *m/z* (EI) 247 (M^+, ^{37}Cl), 245 (M^+, ^{35}Cl); IR (KBr plate, $CDCl_3$) ν 3266, 2924, 1666, 1510, 1480, 1442, 1294, 1097, 787, 755.

***N*-(5-Acetyl-biphenyl-2-yl)-acetamide(3oa).** Following the general procedures, the reaction was performed on a 0.2 mmol scale at 110 °C for 48 h. A white powder was obtained purified by silica gel chromatography (hexanes:EtOAc, 65:35), yield 0.030 g (59%). ^1H NMR (300 MHz, $CDCl_3$) δ 8.51 (d, *J* = 8.40 Hz, 1H), 7.96 (q, *J* = 4.20 Hz, 1H), 7.85 (d, *J* = 5.40 Hz, 1H), 7.53 - 7.51 (m, 3H), 7.49 -7.37 (m, 3H), 2.59 (s, 3H), 2.04 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.03, 168.38, 139.10, 136.94, 132.52, 130.24, 129.40, 129.15, 128.55, 128.42, 119.98, 26.48, 24.86; *m/z* (EI) 253 (M^+, 50.6%) 196 (100%); HRMS: Anal. Calcd. for $C_{16}H_{15}NO_2$ 283.11028. Found: 253.11064; IR (KBr plate, $CDCl_3$) ν 3311, 2983, 1712, 1514, 1443, 1296, 1235, 1108, 753, 702.

***N*-(4-Methoxy-biphenyl-2-yl)-acetamide (3pa).** Following the general procedures, the

reaction was performed on a 0.2 mmol scale at 110 °C for 48 h. A white powder was S8obtained after silica gel chromatography (50:50 hexanes:EtOAc), yield 0.025 g (52%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.98 (d, *J* = 2.70 Hz, 1H), 7.49 - 7.33 (m, 5H), 7.23 (s, 1H), 7.14 (d, *J* = 8.40 Hz, 1H), 6.74 (d, *J* = 2.70 Hz, 1H), 3.85 (s, 3H), 2.02 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.27, 159.48, 137.90, 135.58, 130.68, 129.36, 129.07, 127.61, 124.26, 110.52, 106.21, 55.40, 24.74; *m/z* (EI) 241 (M^+, 52.7%), 199 (100%); IR (KBr plate, $CDCl_3$) ν 3262, 2934, 1666, 1583, 1527, 14666, 1303, 1236, 1166, 1049, 764, 701; Anal. Calcd. for $C_{15}H_{15}NO_2$: C, 74.67; H, 6.27; N, 5.81. Found: C, 74.57; H, 6.15; N, 5.68.

***N*-(3-Methoxy-biphenyl-2-yl)-acetamide (3qa).[3]** Following the general procedures, the reaction was performed on a 0.2 mmol scale at 110 °C for 48 h. A white powder was S8obtained after silica gel chromatography (50:50 hexanes:EtOAc), yield 0.039 g (80%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36-7.31 (m, 6H), 6.93 (d, *J* = 8.10 Hz, 2H), 6.70 (s, 1H), 3.86 (s, 3H), 1.99 (d, *J* = 5.10 Hz, 2H), 1.65 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.62, 154.99, 140.98, 139.37, 128.94, 128.53, 128.34, 127.47, 122.20, 122.29, 110.43, 55.86, 23.10; *m/z* (EI) 241 (M^+, 52.7%), 199 (100%); IR (KBr plate, $CDCl_3$) ν 3128, 2974, 1656, 1466, 1260, 1121, 1020, 759, 705.

***N*-(3, 5-Dimethyl-biphenyl-2-yl)-acetamide (3ra).[2]** Following the general procedures, the reaction was performed on a 0.2 mmol scale at 110 °C for 48 h. A white powder was obtained after silica gel chromatography (hexanes:EtOAc, 65:35), yield 0.035 g (73%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.43-7.30 (m, 6H), 7.09 (s, 1H), 7.00 (s, 1H), 6.60 (s, 1H), 2.34 (s, 3H), 2.29 (s, 3H), 1.99 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.58, 139.68, 139.37, 137.21, 136.52, 130.84, 129.87, 128.98, 128.80, 128.58, 128.46, 128.25, 23.03, 20.96, 18.46; *m/z* (EI) 239 (M^+, 52.7%), 197 (100%); IR (KBr plate, $CDCl_3$) ν 3248, 3027, 1652, 1524, 1291, 857, 701.

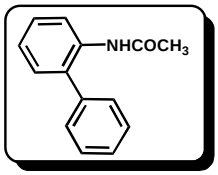
***N*-(3-Methoxy-5-methyl-biphenyl-2-yl)-acetamide (3sa).** Following the general procedures, the reaction was performed on a 0.2 mmol scale at 110 °C for 48 h. A white powder was obtained after silica gel chromatography (65:35 hexanes:EtOAc), yield 0.033 g (65%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.88 (s, 1H), 7.45 (d, *J* = 6.90, 2H), 7.39-7.32 (m, 3H), 7.18 (s, 1H), 7.01 (s, 1H), 3.88 (s, 3H), 2.20 (s, 3H), 2.02 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.28, 157.31, 138.17, 133.25, 131.63, 129.53, 128.97, 127.43, 123.83, 122.56, 103.85, 55.49, 24.59, 15.70; *m/z* (EI) 255 (M^+, 52.7%), 213 (100%); IR (KBr plate, $CDCl_3$) ν 3282, 3003, 1666, 1517, 1398, 1233, 1143, 1050, 753, 702; Anal. Calcd. for $C_{16}H_{17}NO_2$: C, 75.27; H, 6.71; N, 5.49. Found: C, 75.33; H, 6.79; N, 5.53.

Acetic acid 6-acetylamino-4-methoxy-biphenyl-3-yl ester (3ta). Following the general procedures, the reaction was performed on a 0.2 mmol scale at 110 °C for 48 h. A white powder was obtained after silica gel chromatography (50:50 hexanes:EtOAc), yield 0.037 g (62%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.30 (s, 1H), 7.76 (s, 1H), 7.53-7.34 (m, 6H), 3.98 (s, 3H), 3.86 (s, 3H), 2.06 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.51, 165.87, 159.83, 139.61, 136.66, 133.58, 129.35, 129.33, 128.15, 122.71, 114.31, 103.56, 56.20, 51.88, 25.02; *m/z* (EI) 299 (M^+, 52.7%), 226 (100%); IR (KBr plate, $CDCl_3$) ν 3422, 3017, 1719, 1693, 1580, 1464, 1399, 1224, 1090, 1081, 753; Anal. Calcd. for $C_{17}H_{14}NO_4$:

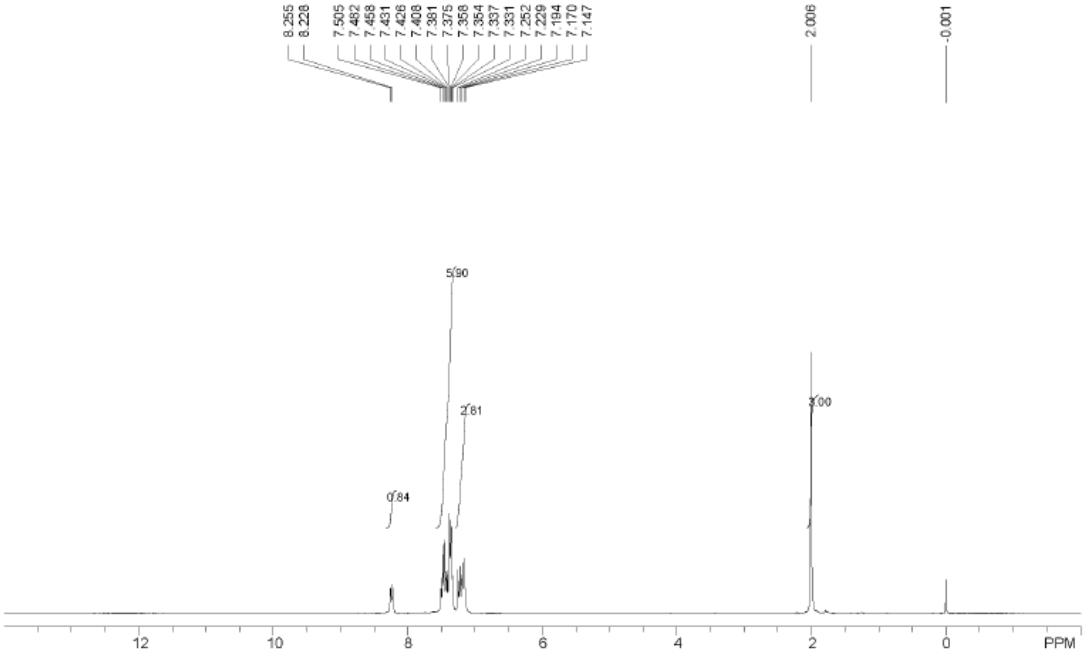
C, 68.21; H, 5.72; N, 4.68; Found: C, 68.00; H, 5.80; N, 4.54.

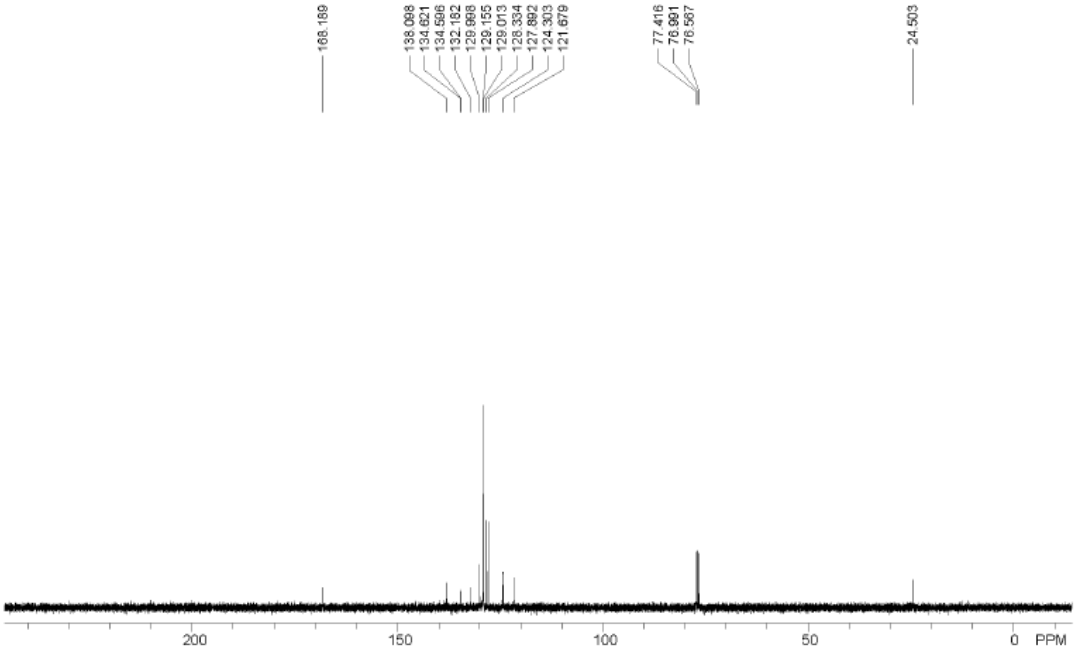
References:

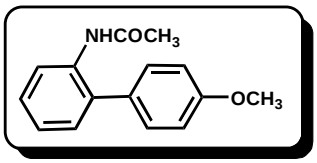
1. Chauhan, K. K.; Frost, C. G.; Love, I.; Waite, D. *Synlett* **1999**, 1743–1744.
2. Tsang, W. C. P.; Zheng, N.; Buchwald, S. L. *J. Am. Chem. Soc.* **2005**, *127*, 14560-14561.
3. Kim, H. J.; Asif, R.; Chung, D. S.; Hong, J. I. *Tetrahedron Lett.* **2003**, *44*, 4355-4359.


NHCOCH3

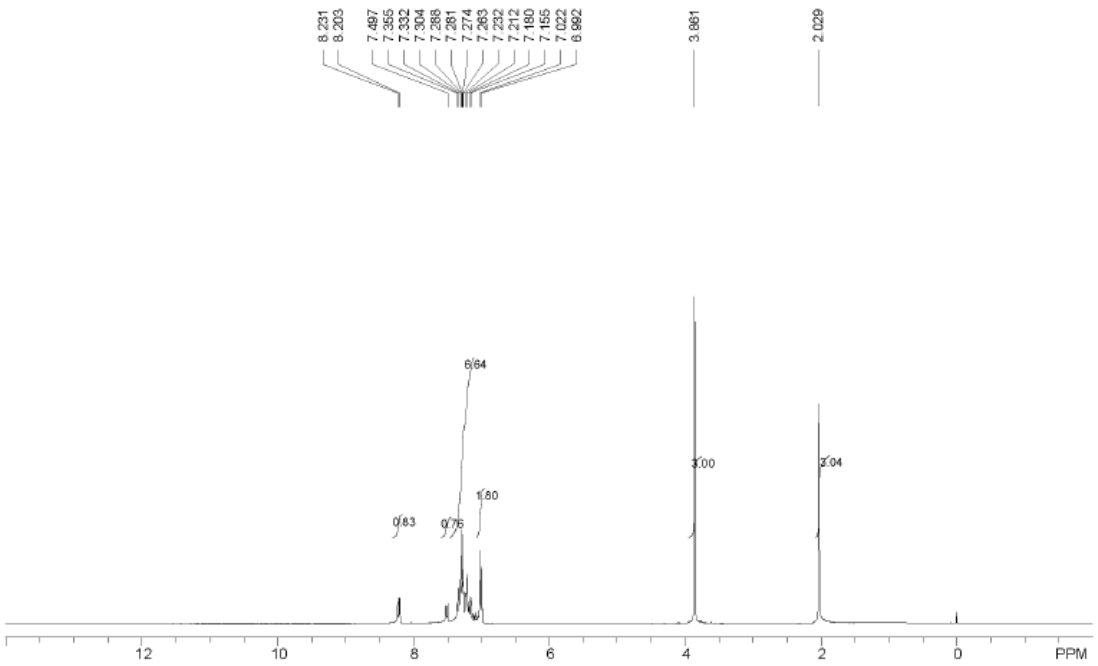
3aa

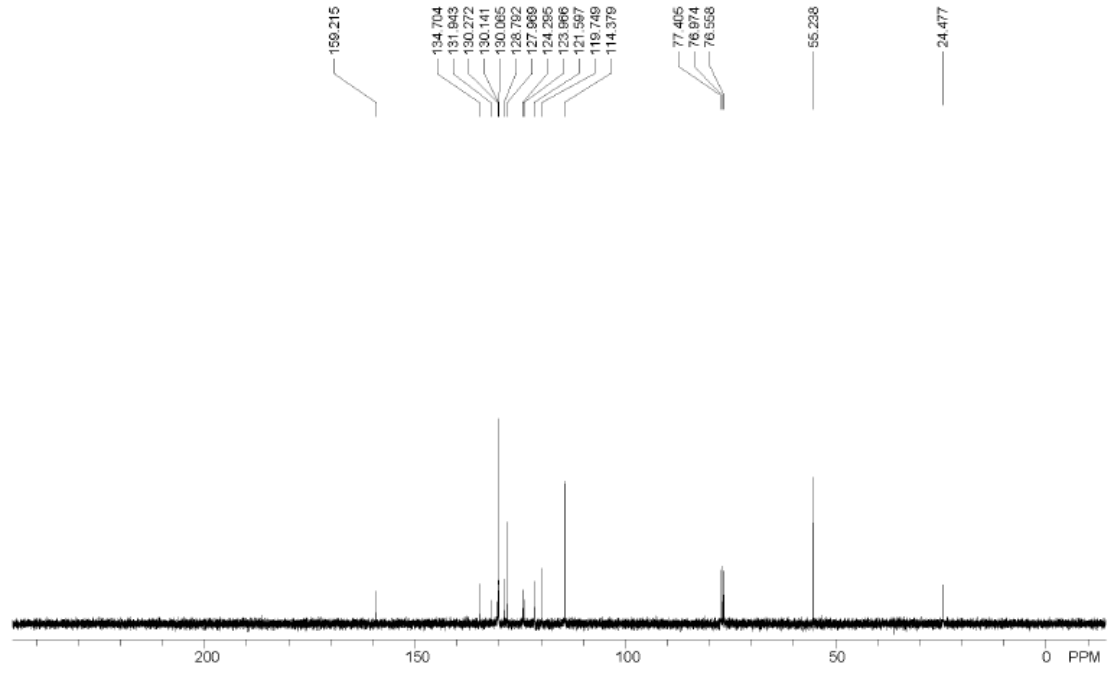

8.255
8.228
7.505
7.482
7.458
7.431
7.426
7.408
7.391
7.375
7.358
7.354
7.337
7.331
7.252
7.229
7.194
7.170
7.147
2.006
-0.001
5.90
2.81
3.00
0.84
12
10
8
6
4
2
0
PPM

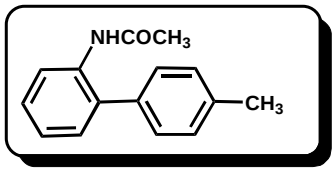

168.189
138.098
134.621
134.596
132.182
129.998
129.155
129.013
128.334
127.892
124.303
121.679
77.416
76.991
76.567
24.503
200
150
100
50
0
PPM


NHCOCH3
OCH3

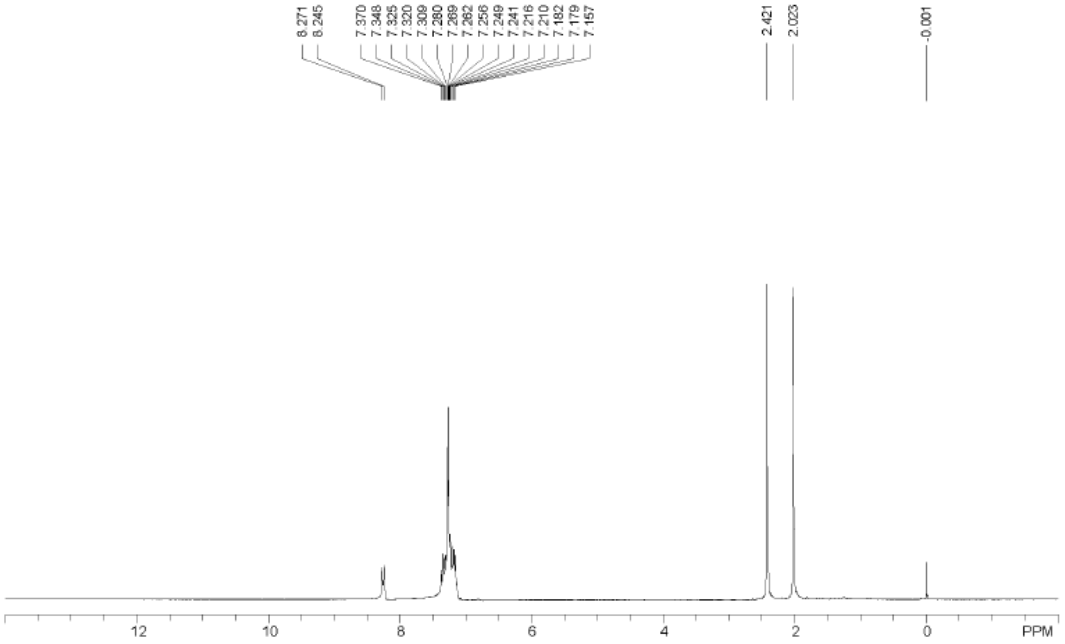
3ac

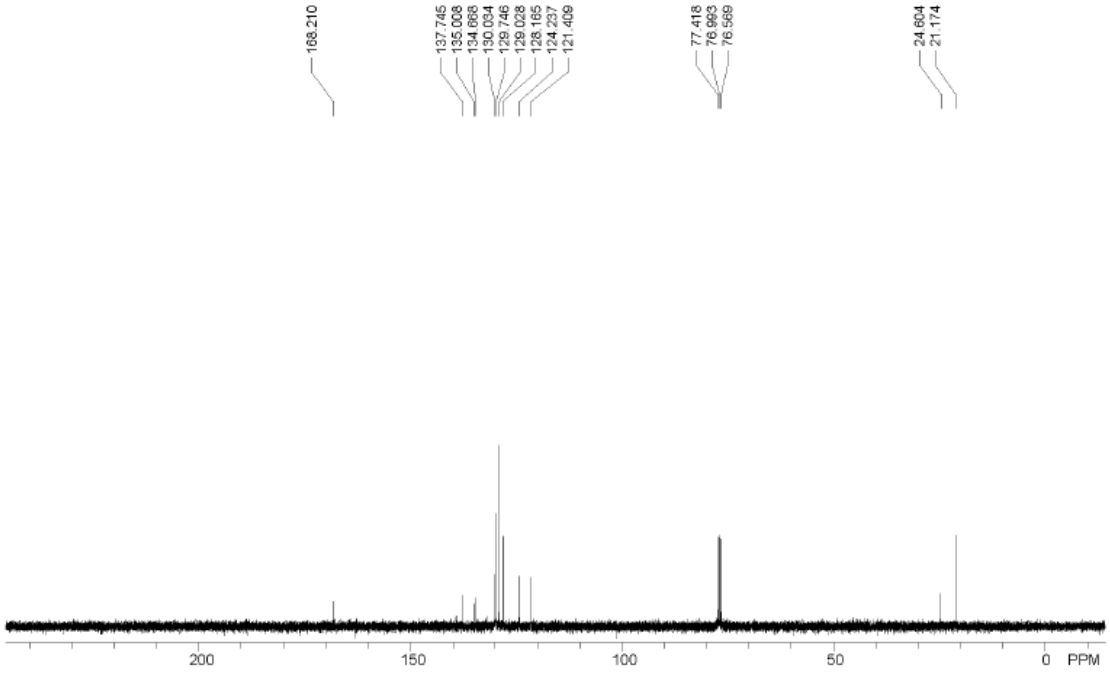

8.231
8.203
7.497
7.355
7.332
7.304
7.288
7.281
7.274
7.263
7.232
7.212
7.190
7.155
7.022
6.992
3.861
2.029
6.64
3.00
3.04
1.80
0.83
0.75
12
10
8
6
4
2
0
PPM

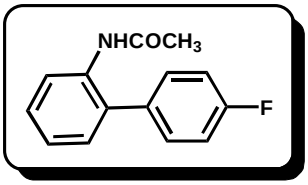

159.215
134.704
131.943
130.272
130.141
130.065
128.792
127.969
124.295
123.966
121.597
119.749
114.379
77.405
76.974
76.558
55.238
24.477
200
150
100
50
0
PPM


NHCOCH3
CH3

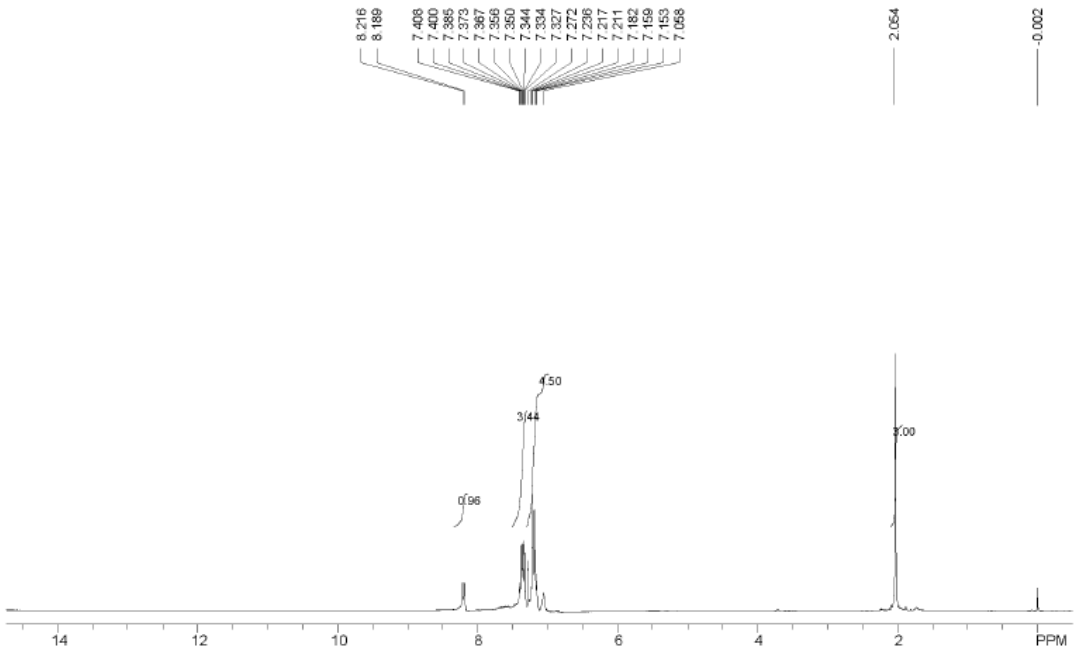
3ad

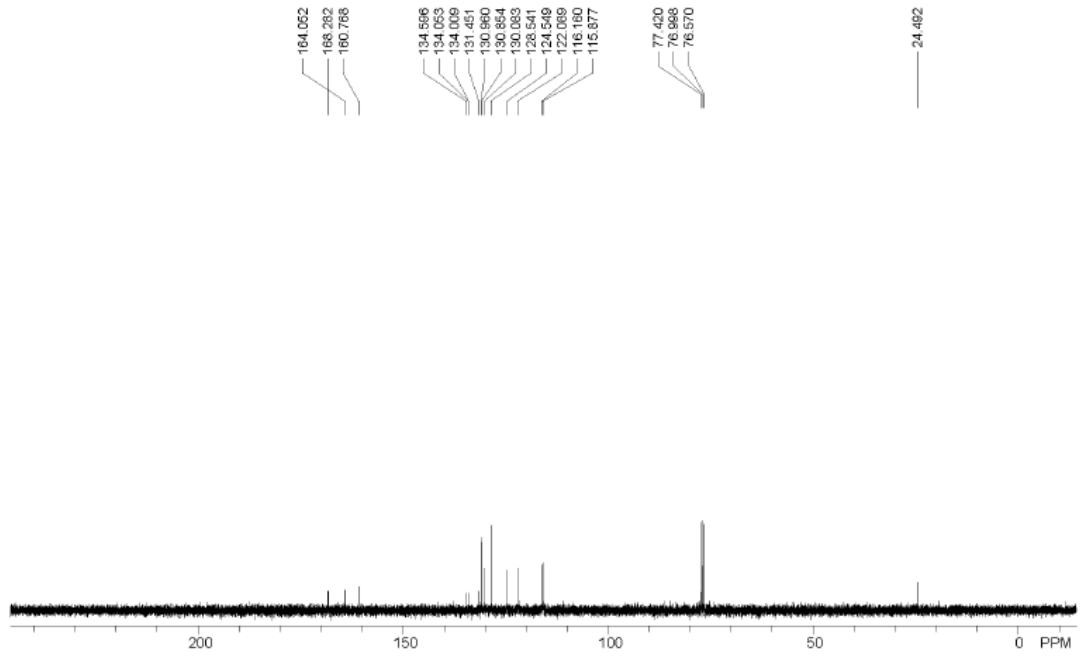

8.271
8.245
7.370
7.348
7.325
7.320
7.309
7.280
7.269
7.262
7.256
7.249
7.241
7.216
7.210
7.182
7.179
7.157
2.421
2.023
0.001
12
10
8
6
4
2
0
PPM

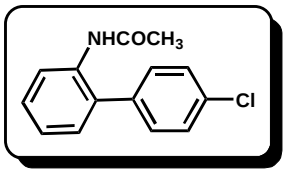

168.210
137.745
135.008
134.668
130.034
129.746
129.028
128.165
124.237
121.409
77.418
76.993
76.569
24.604
21.174
200
150
100
50
0
PPM


NHCOCH3
F

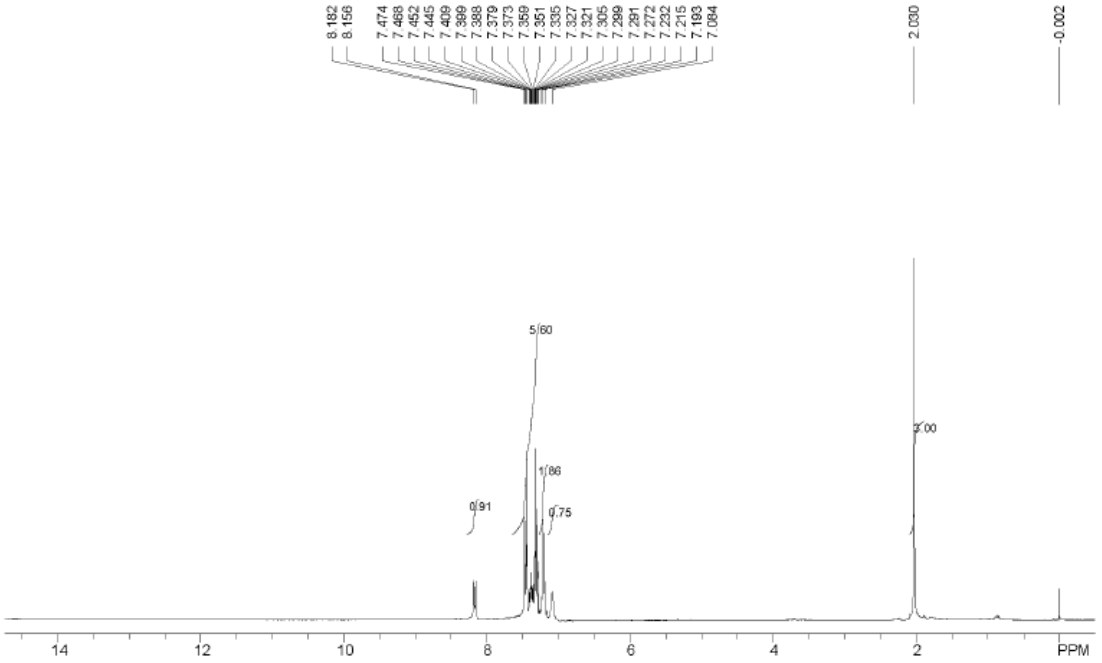
3ae

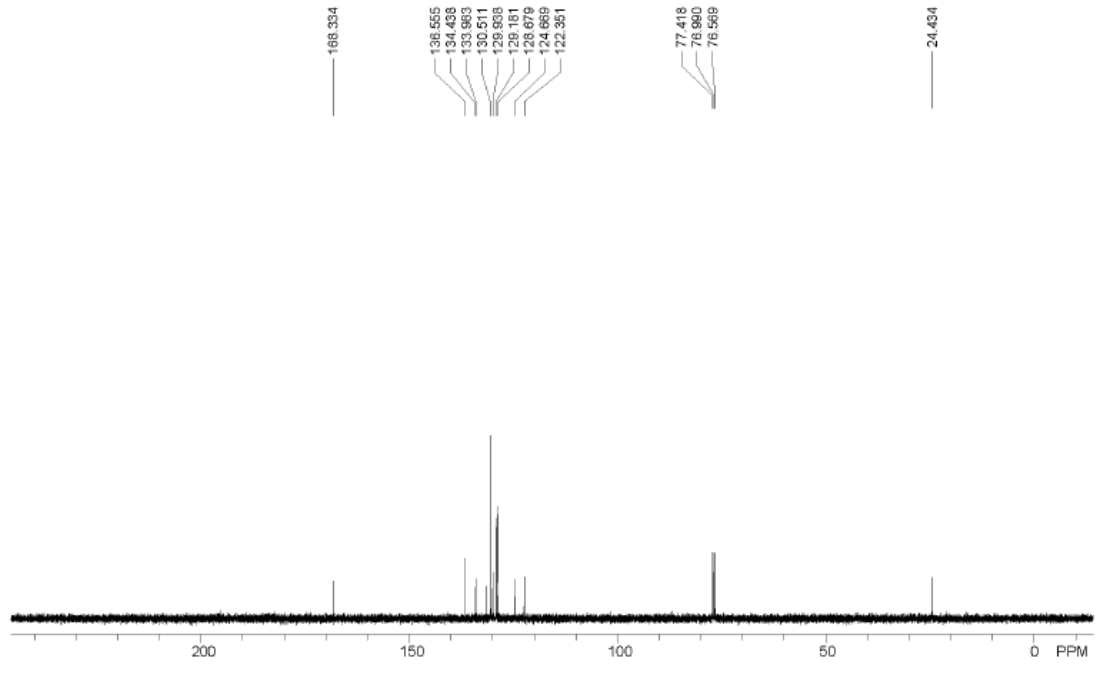

8.216
8.189
7.408
7.400
7.385
7.373
7.367
7.356
7.350
7.344
7.334
7.327
7.272
7.236
7.217
7.211
7.182
7.159
7.153
7.058
2.054
-0.002
4.50
3.44
2.00
0.96
14
12
10
8
6
4
2
PPM

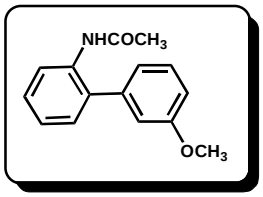

164.052
163.282
160.768
134.586
134.053
134.009
131.451
130.960
130.854
130.083
128.541
124.549
122.069
116.160
115.877
77.420
76.998
76.570
24.492
200
150
100
50
0
PPM


NHCOCH3
Cl

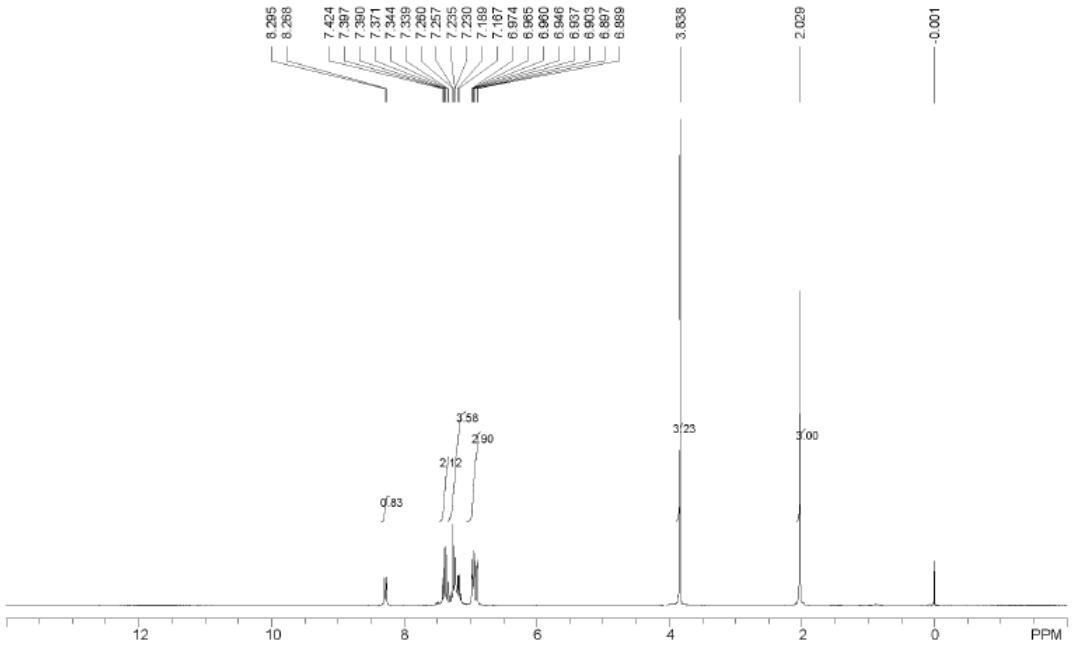
3af

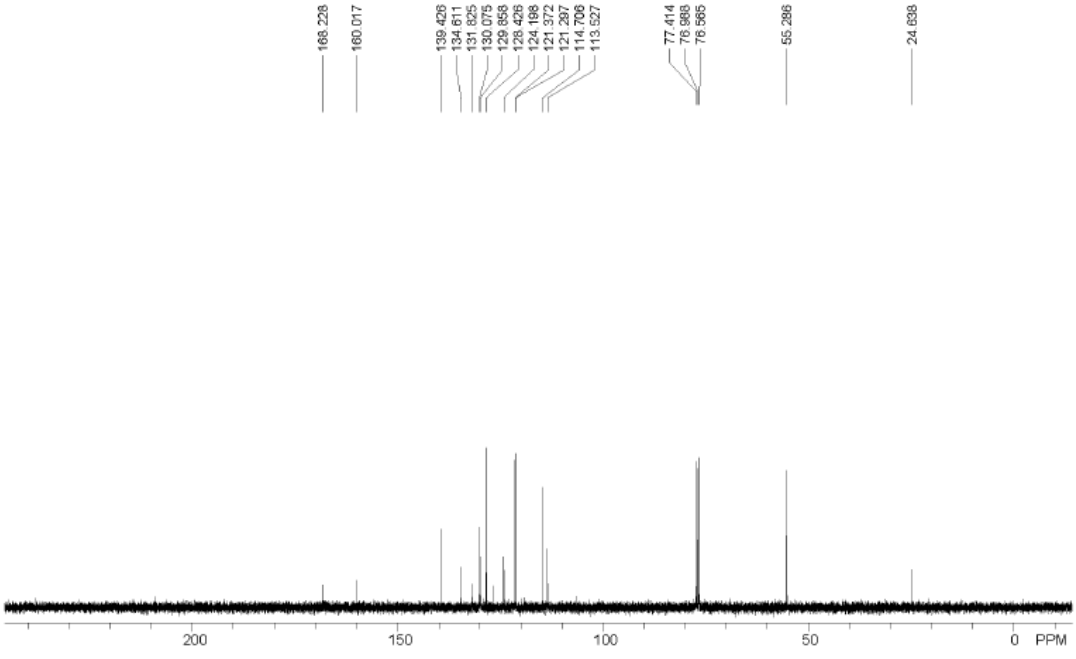

8.182
8.156
7.474
7.468
7.452
7.445
7.409
7.389
7.388
7.379
7.373
7.359
7.351
7.335
7.327
7.321
7.305
7.299
7.291
7.272
7.232
7.215
7.193
7.084
2.030
-0.002
5.60
3.00
1.86
0.91
0.75
14
12
10
8
6
4
2
PPM

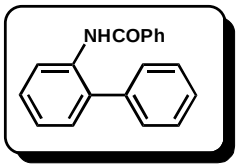

168.334
136.555
134.438
133.983
130.511
129.938
129.181
128.679
124.669
122.351
77.418
76.990
76.569
24.434
200
150
100
50
0
PPM


NHCOCH3
OCH3

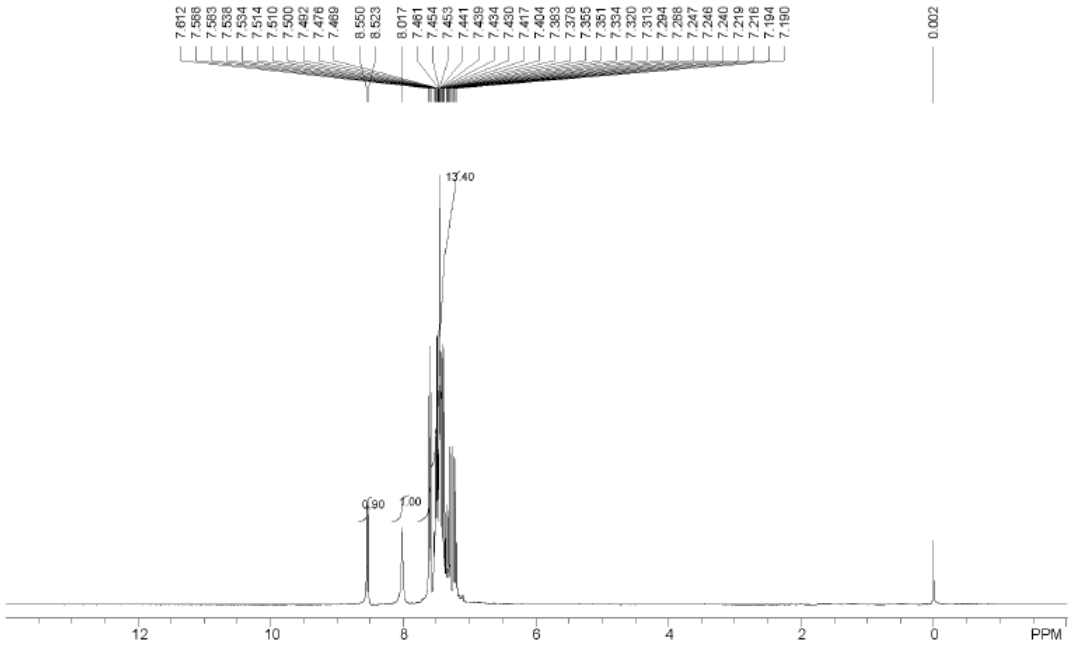
3ag

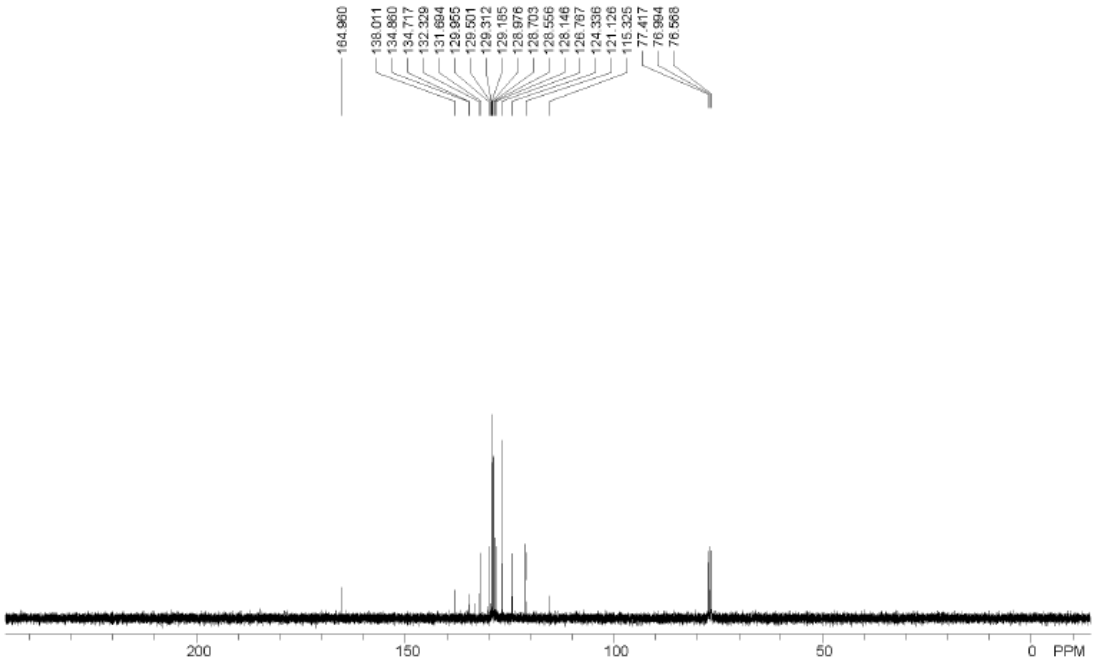

8.295
8.268
7.424
7.397
7.390
7.371
7.344
7.339
7.260
7.257
7.235
7.230
7.189
7.167
6.974
6.965
6.960
6.946
6.937
6.903
6.897
6.889
3.838
2.029
-0.001
0.83
2.12
2.90
3.58
3.23
3.00
12
10
8
6
4
2
0
PPM

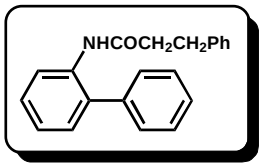

168.228
160.017
139.426
134.611
131.825
130.075
129.858
128.426
124.198
121.372
121.287
114.706
113.527
77.414
76.988
76.565
55.286
24.638
200
150
100
50
0
PPM


NHCOPh

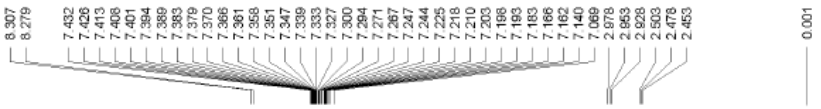
3ca

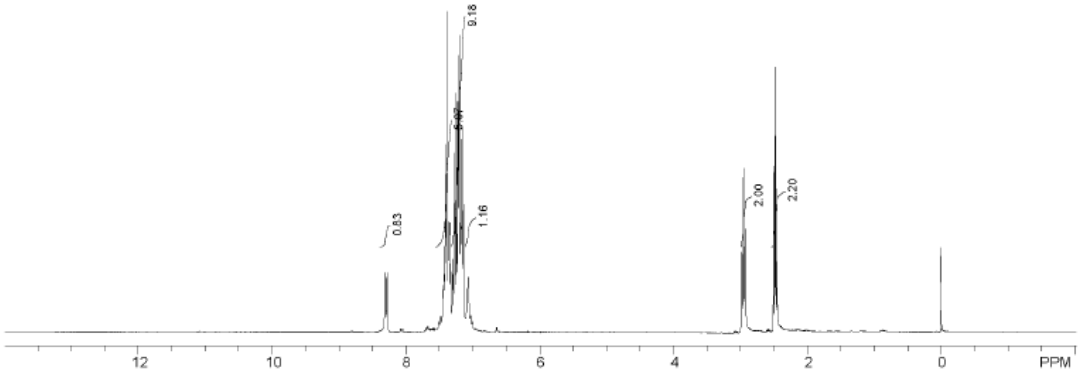

7.612
7.586
7.583
7.538
7.534
7.514
7.510
7.500
7.492
7.476
7.469
8.550
8.523
8.017
7.461
7.454
7.453
7.441
7.439
7.434
7.430
7.417
7.404
7.393
7.378
7.365
7.351
7.334
7.320
7.313
7.294
7.288
7.247
7.246
7.240
7.219
7.216
7.194
7.190
0.002
13.40
0.90
1.00
12
10
8
6
4
2
0
PPM

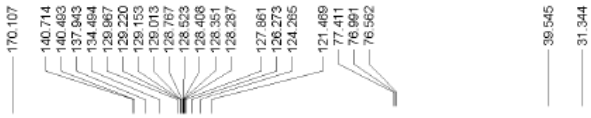

164.980
138.011
134.860
134.717
132.329
131.694
129.955
129.501
129.312
129.185
128.976
128.703
128.556
128.146
126.787
124.336
121.126
115.325
77.417
76.994
76.568
200
150
100
50
0
PPM

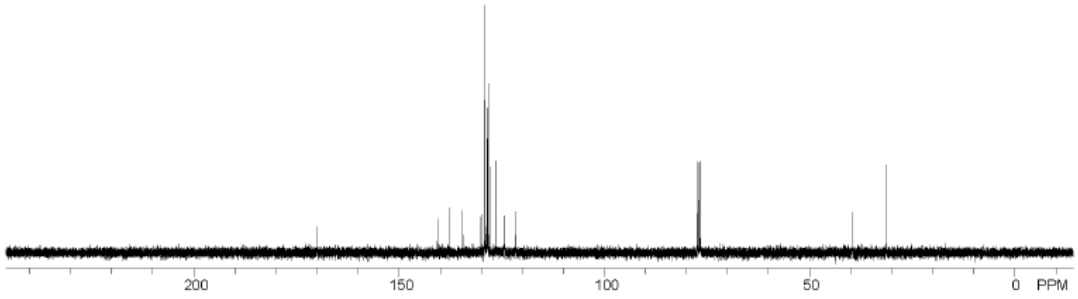

NHCOCH2CH2Ph

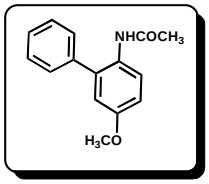
3ha


8.307
8.279
7.432
7.426
7.413
7.406
7.401
7.394
7.389
7.383
7.379
7.370
7.366
7.361
7.358
7.351
7.347
7.339
7.333
7.327
7.300
7.294
7.271
7.267
7.247
7.244
7.225
7.218
7.210
7.203
7.196
7.193
7.183
7.166
7.162
7.140
7.069
2.978
2.953
2.928
2.903
2.478
2.453
0.001

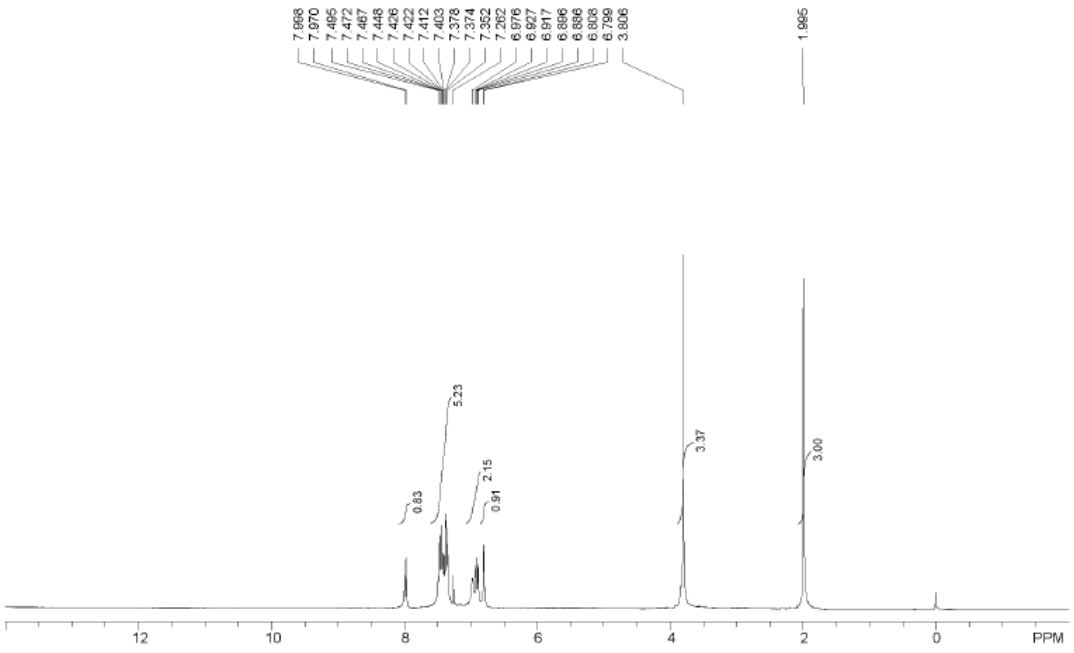

9.18
8.00
0.83
1.16
2.00
2.20
12
10
8
6
4
2
0
PPM

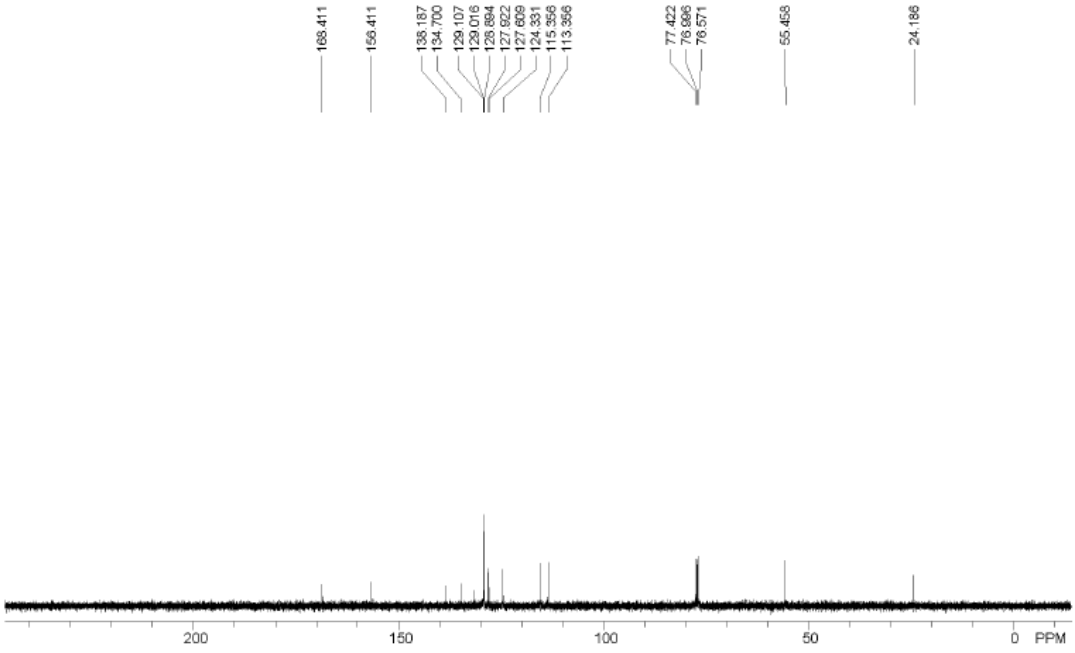

170.107
140.714
140.493
137.943
134.494
129.867
129.220
129.153
129.013
128.767
128.523
128.408
128.351
128.287
127.861
126.273
124.265
121.469
77.411
76.991
76.562
39.545
31.344

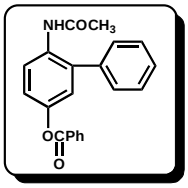

200
150
100
50
0
PPM


NHCOCH3
H3CO

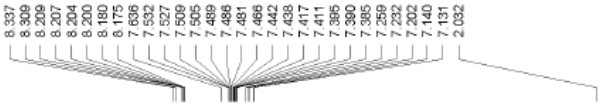
3ja

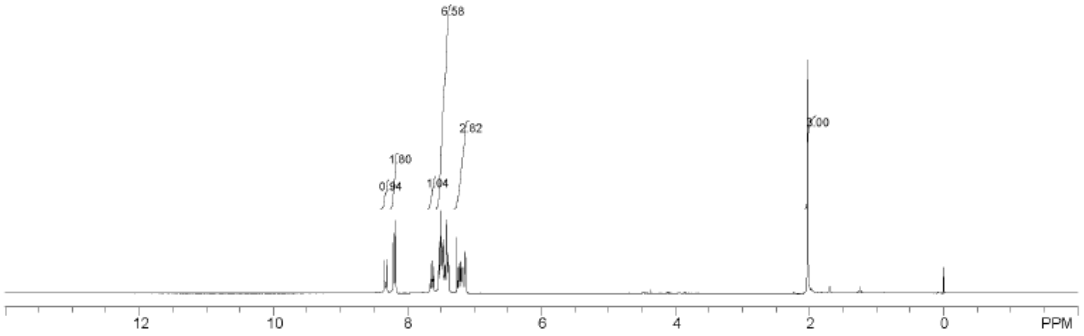

7.998
7.970
7.495
7.472
7.467
7.448
7.426
7.422
7.412
7.403
7.378
7.374
7.352
7.262
6.976
6.927
6.917
6.896
6.886
6.808
6.799
3.806
1.995
0.83
5.23
2.15
0.91
3.37
3.00
12
10
8
6
4
2
0
PPM

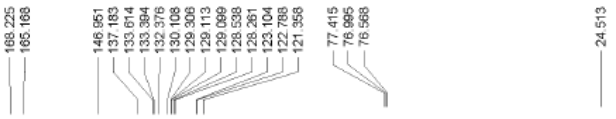

168.411
156.411
138.187
134.700
129.107
129.016
126.894
127.922
127.609
124.331
115.356
113.356
77.422
76.996
76.571
55.458
24.186
200
150
100
50
0
PPM

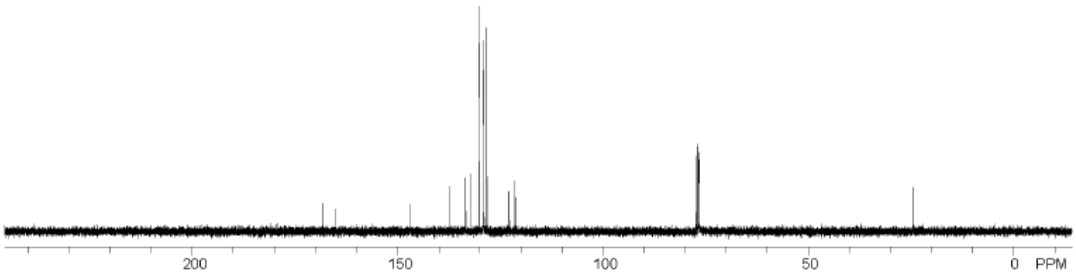

NHCOCH3
OCPh
O

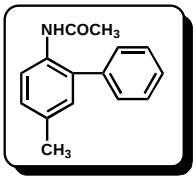
3la


8.337
8.309
8.209
8.207
8.204
8.200
8.180
8.175
7.536
7.532
7.527
7.509
7.505
7.489
7.486
7.481
7.466
7.442
7.438
7.417
7.411
7.395
7.390
7.385
7.259
7.232
7.202
7.140
7.131
2.032

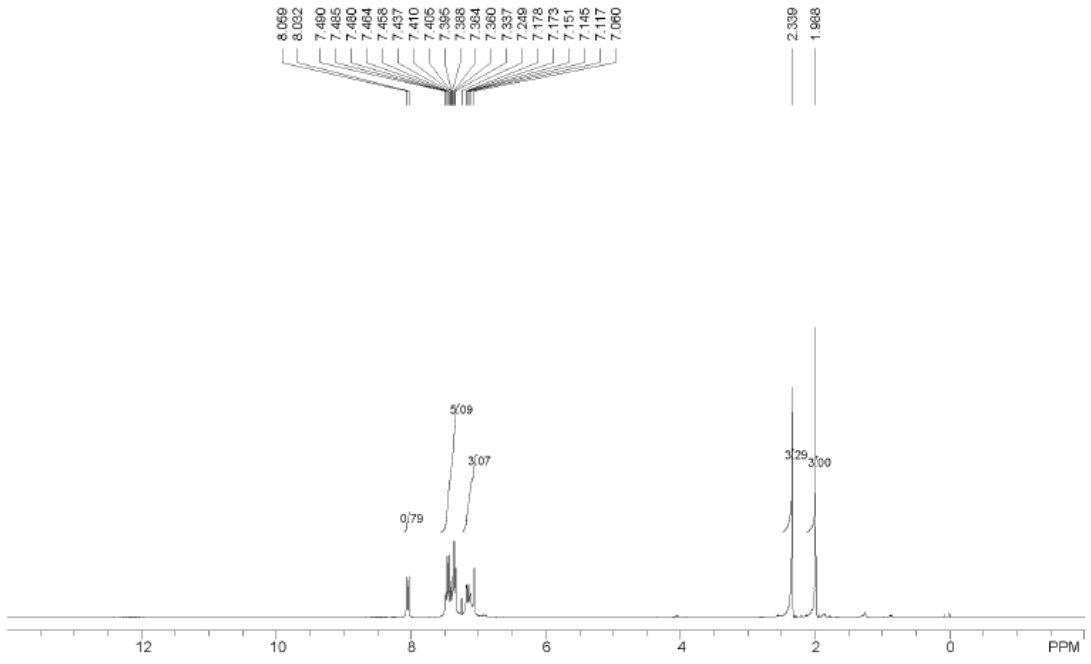

6.58
2.82
3.00
1.80
0.94
1.04
12
10
8
6
4
2
0
PPM

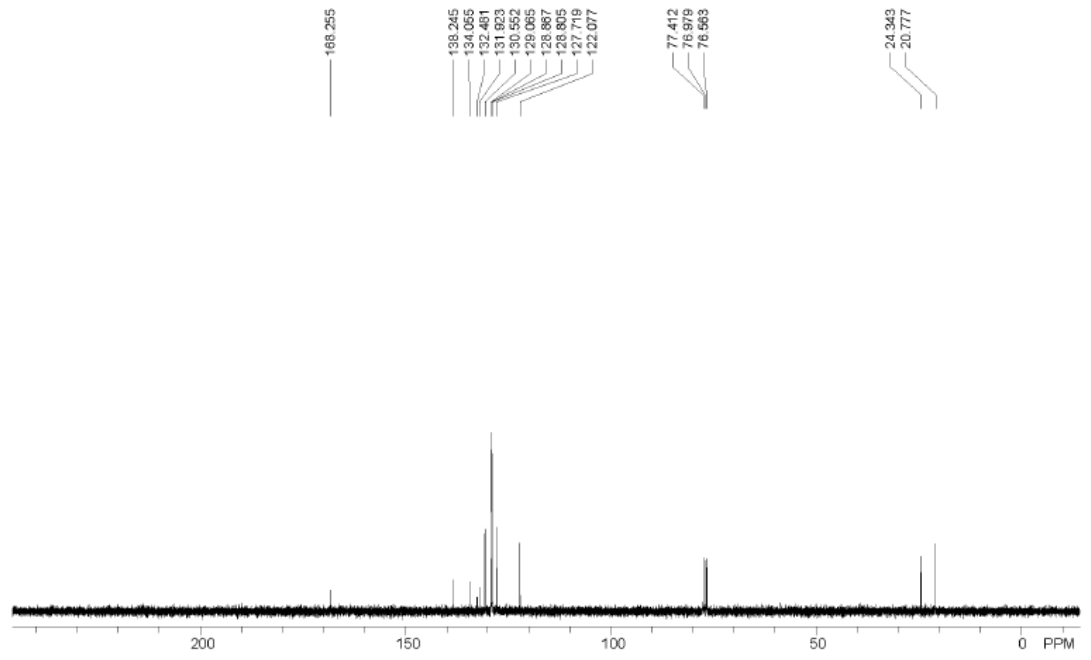

168.225
165.168
146.951
137.183
133.614
133.394
132.376
130.108
129.306
129.113
129.099
128.538
128.261
123.104
122.798
121.358
77.415
76.995
76.588
24.513

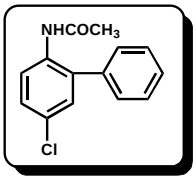

200
150
100
50
0
PPM


NHCOCH3
CH3

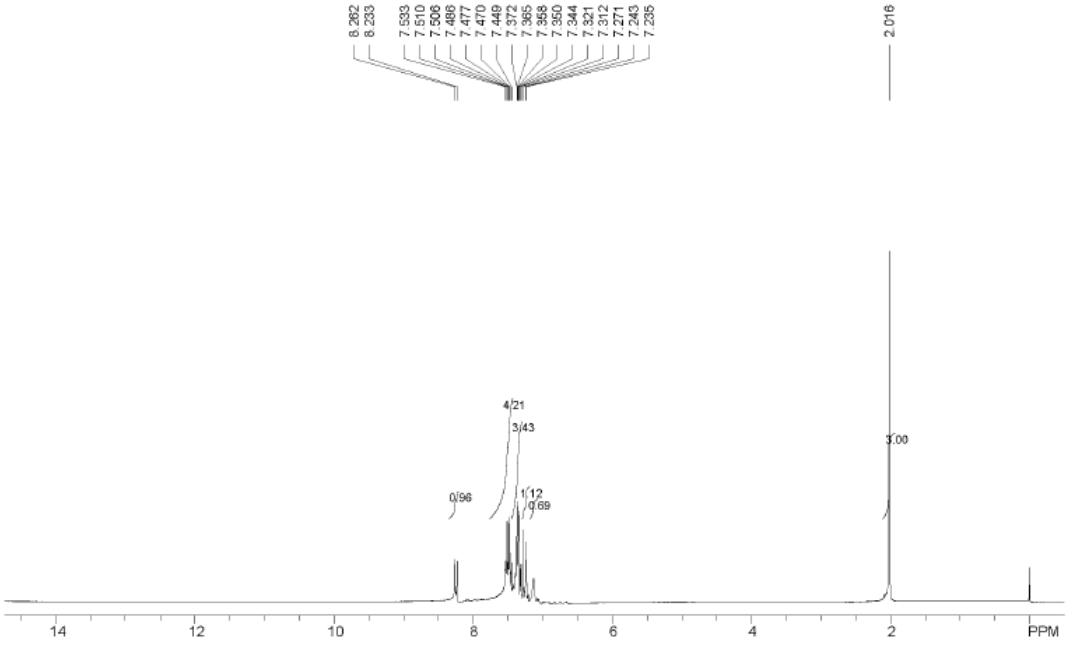
3ma

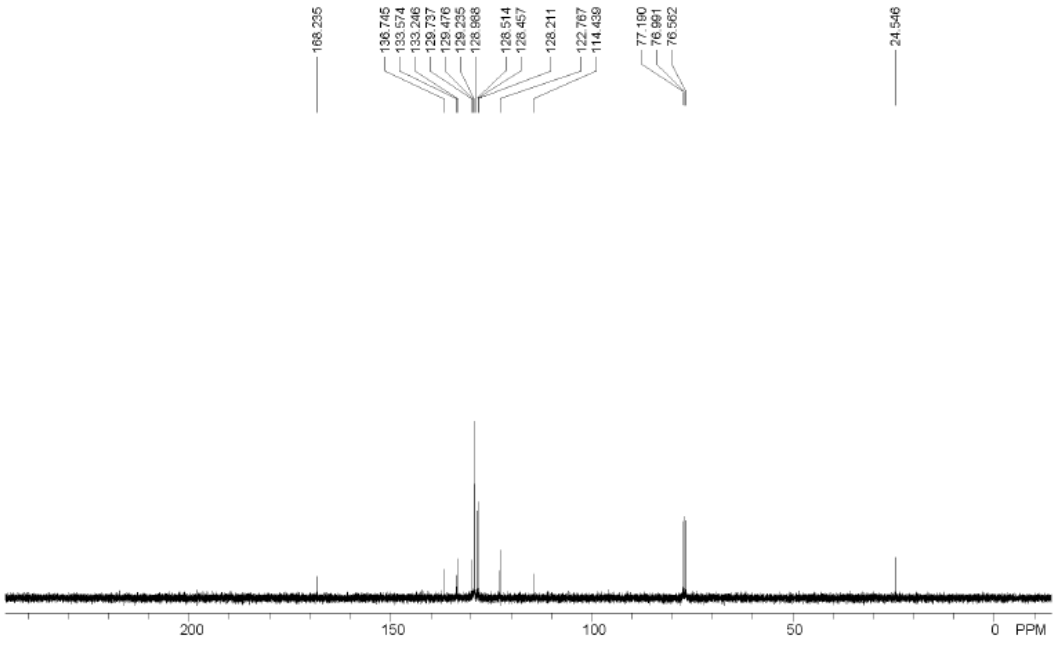

8.059
8.032
7.490
7.485
7.480
7.464
7.458
7.437
7.410
7.406
7.395
7.388
7.384
7.380
7.337
7.249
7.178
7.173
7.151
7.145
7.117
7.060
2.339
1.988
5.09
3.07
0.79
3.29
3.00
12
10
8
6
4
2
0
PPM

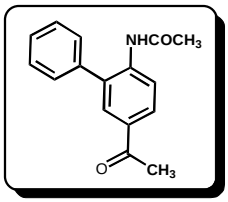

168.255
138.245
134.055
132.481
131.923
130.552
129.065
128.867
128.805
127.719
122.077
77.412
76.979
76.563
24.343
20.777
200
150
100
50
0
PPM


NHCOCH3
Cl

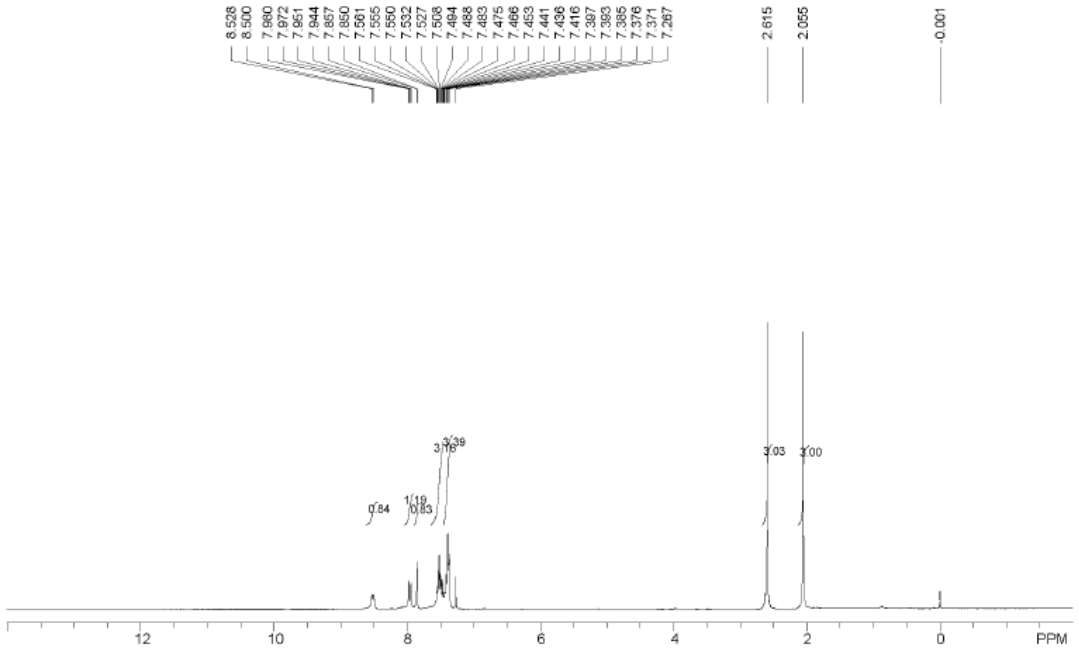
3na

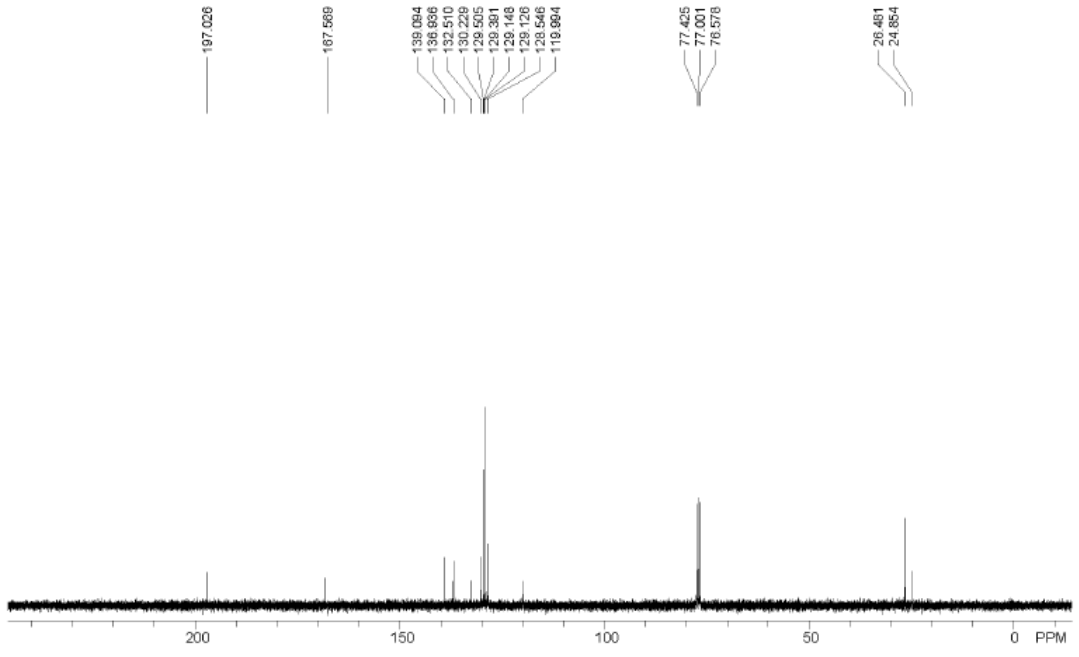

8.262
8.233
7.533
7.510
7.506
7.486
7.477
7.470
7.449
7.372
7.365
7.358
7.350
7.344
7.321
7.312
7.271
7.243
7.235
2.016
4.21
3.43
3.00
0.96
1.12
0.69
14
12
10
8
6
4
2
PPM

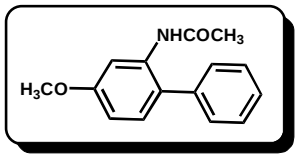

168.235
136.745
133.574
133.246
129.737
129.476
129.235
128.868
126.514
126.457
128.211
122.767
114.439
77.190
76.991
76.562
24.546
200
150
100
50
0
PPM


NHCOCH3
O
CH3

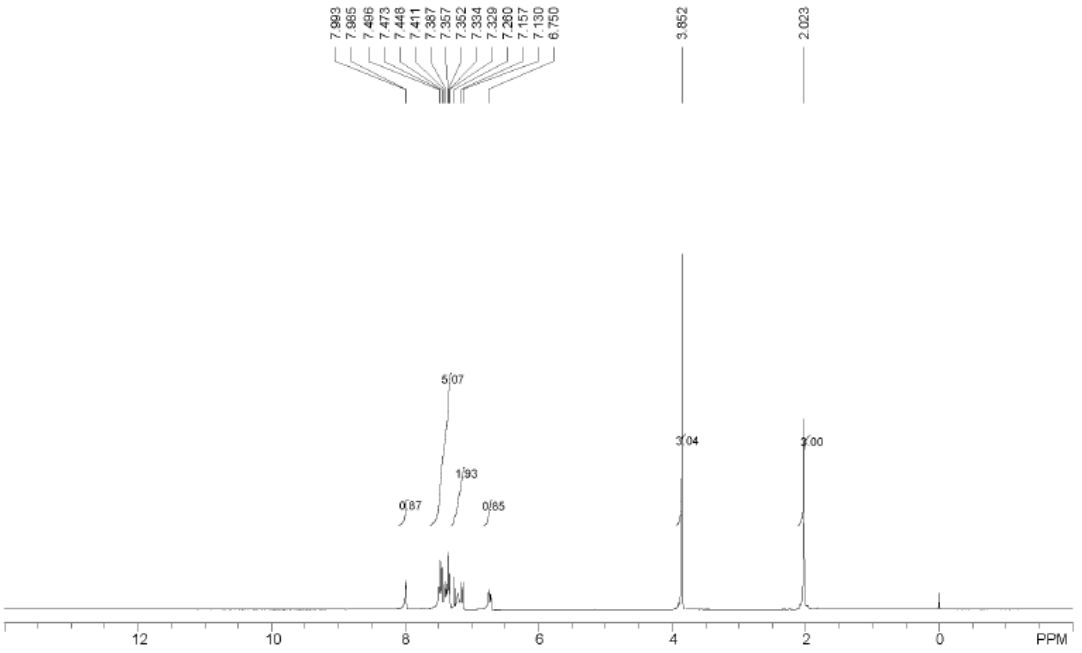
3oa

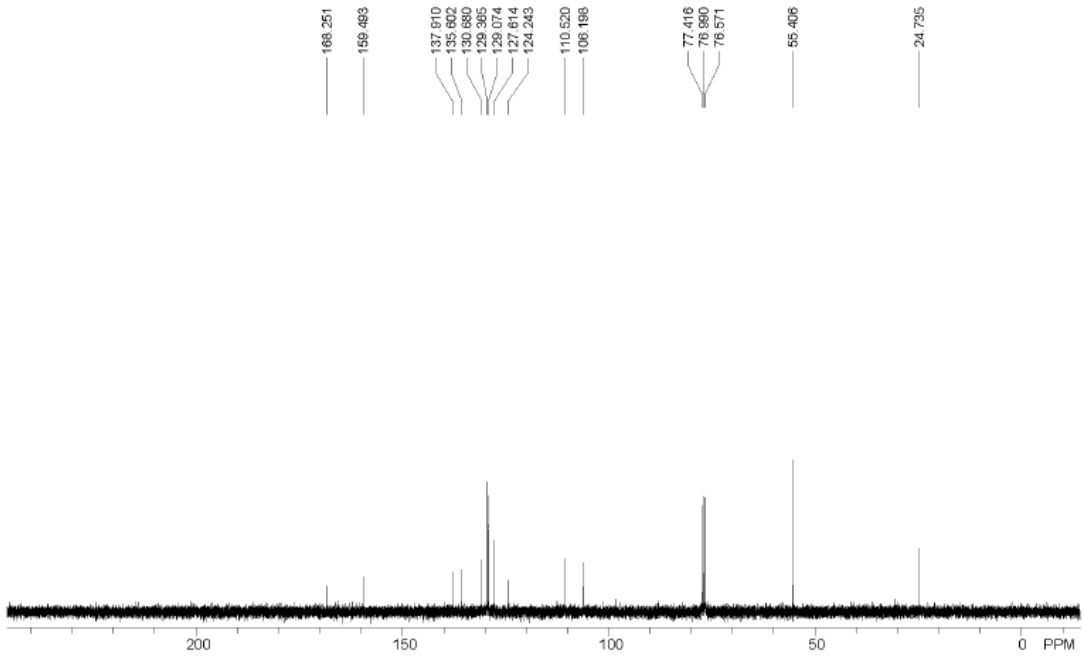

8.528
8.500
7.990
7.972
7.951
7.944
7.857
7.850
7.561
7.555
7.550
7.532
7.527
7.508
7.494
7.488
7.483
7.475
7.466
7.453
7.441
7.436
7.416
7.397
7.383
7.385
7.376
7.371
7.267
2.615
2.055
0.001
0.84
1.19
0.83
3.39
3.78
3.03
3.00
12
10
8
6
4
2
0
PPM

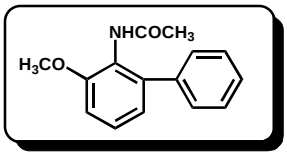

197.026
167.569
139.084
136.896
132.510
130.229
129.505
129.381
129.146
129.126
128.546
119.994
77.425
77.001
76.578
26.461
24.854
200
150
100
50
0
PPM


NHCOCH3
H3CO

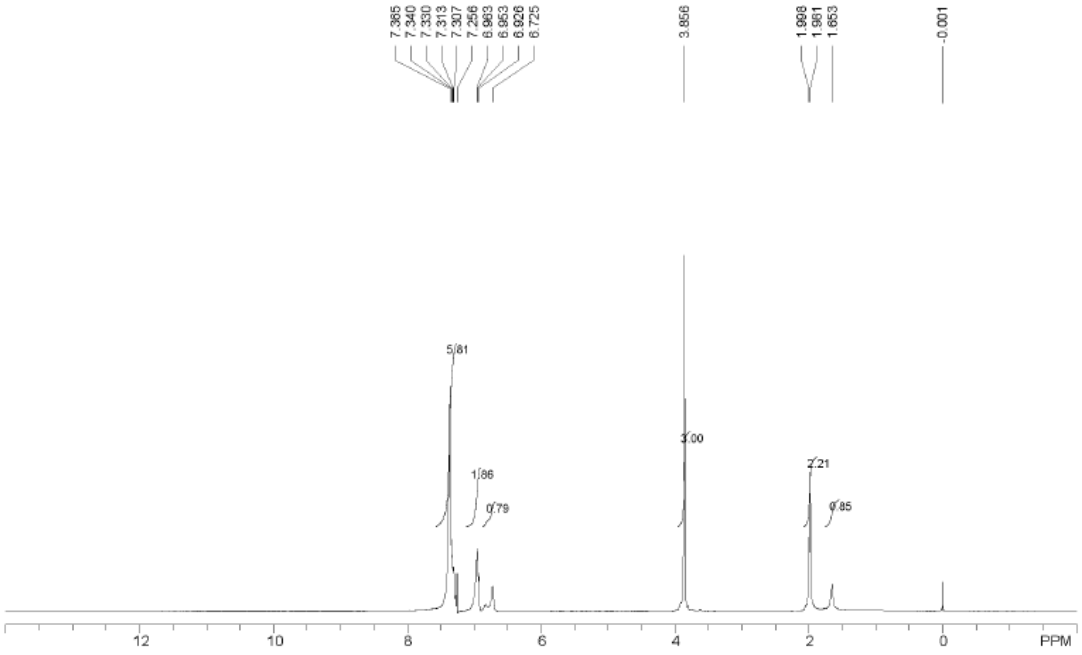
3pa

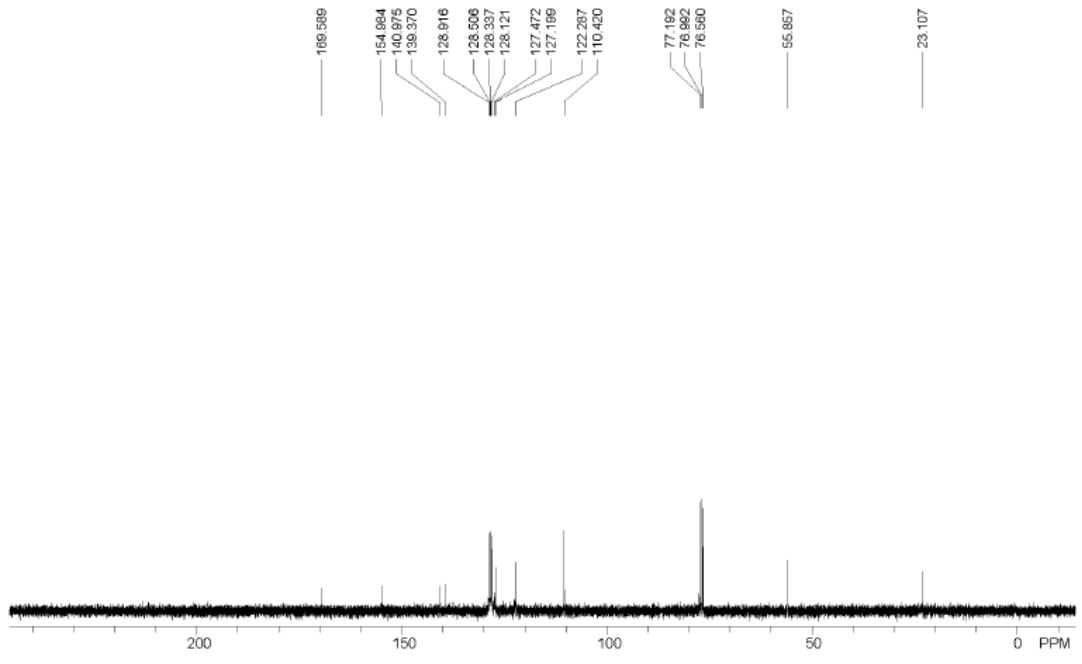

7.993
7.985
7.496
7.473
7.446
7.411
7.387
7.357
7.352
7.334
7.329
7.260
7.157
7.130
6.750
3.852
2.023
5.07
3.04
2.00
1.93
0.87
0.85
12
10
8
6
4
2
0
PPM

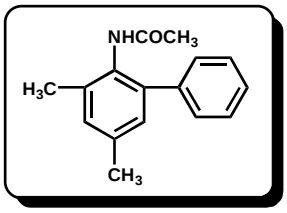

168.251
159.493
137.910
135.602
130.680
129.365
129.074
127.614
124.243
110.520
106.198
77.416
76.990
76.571
55.406
24.735
200
150
100
50
0
PPM


NHCOCH3
H3CO

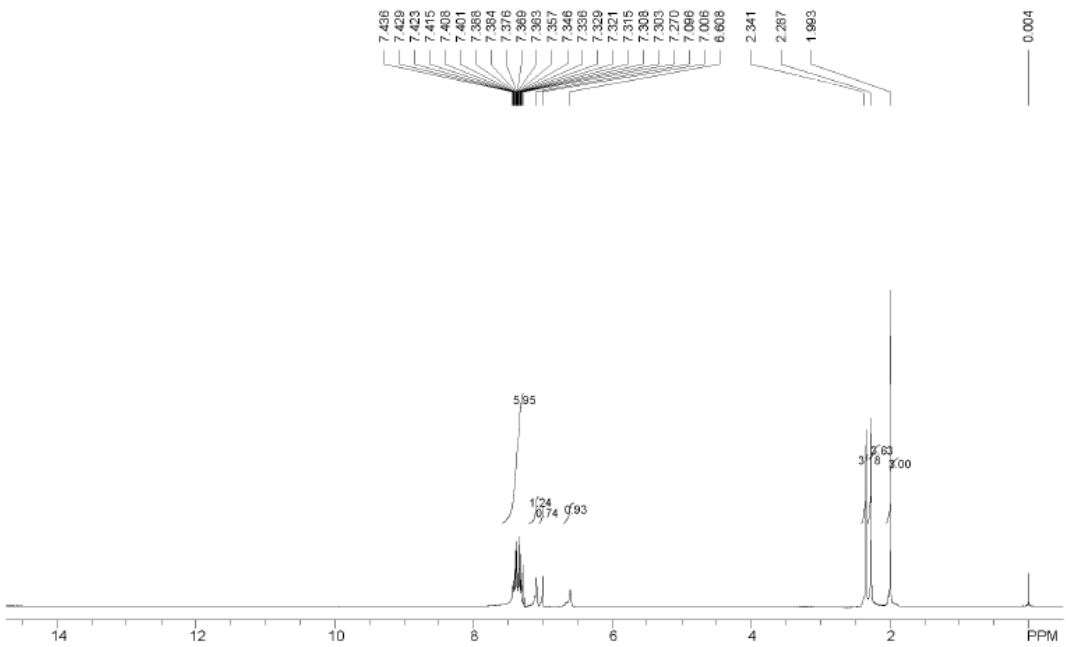
3qa

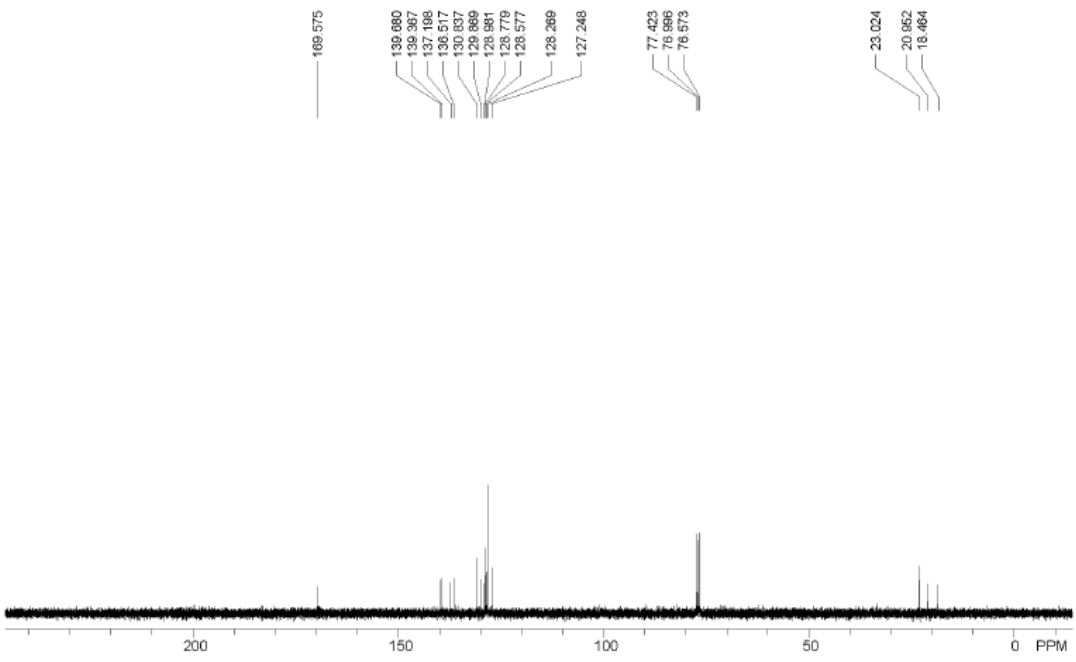

7.365
7.340
7.330
7.313
7.307
7.256
6.983
6.953
6.926
6.725
3.856
1.998
1.961
1.653
0.001
5.81
1.86
0.79
3.00
2.21
0.85
12
10
8
6
4
2
0
PPM

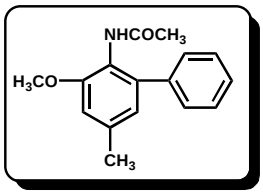

169.589
154.984
140.975
139.370
128.916
128.506
128.337
128.121
127.472
127.199
122.287
110.420
77.192
76.992
76.580
55.857
23.107
200
150
100
50
0
PPM


NHCOCH3
H3C
CH3

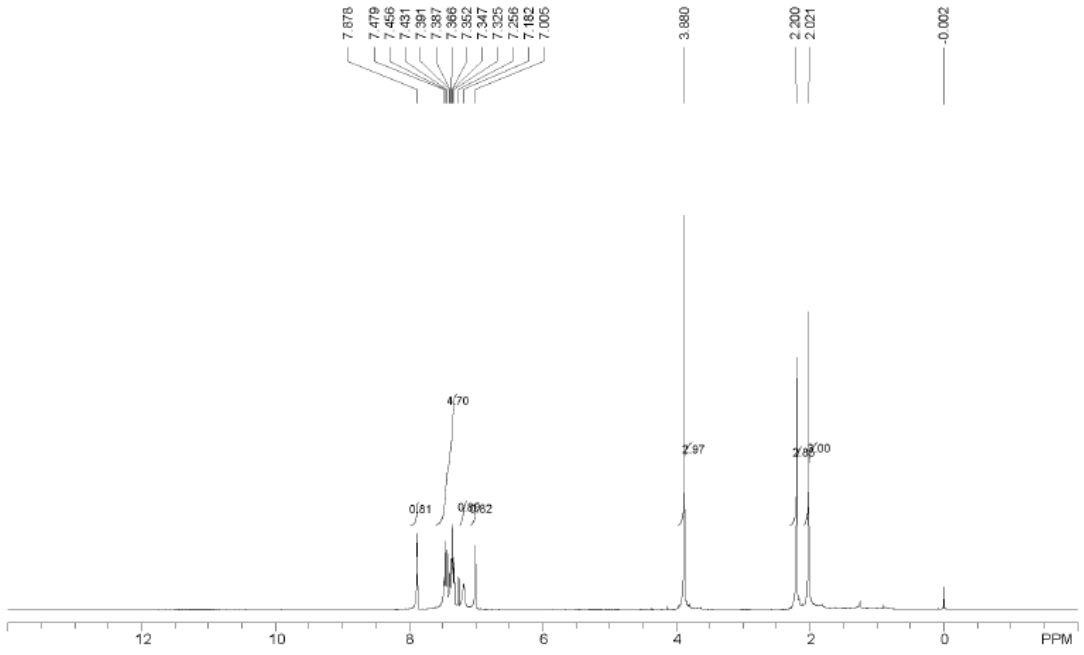
3ra

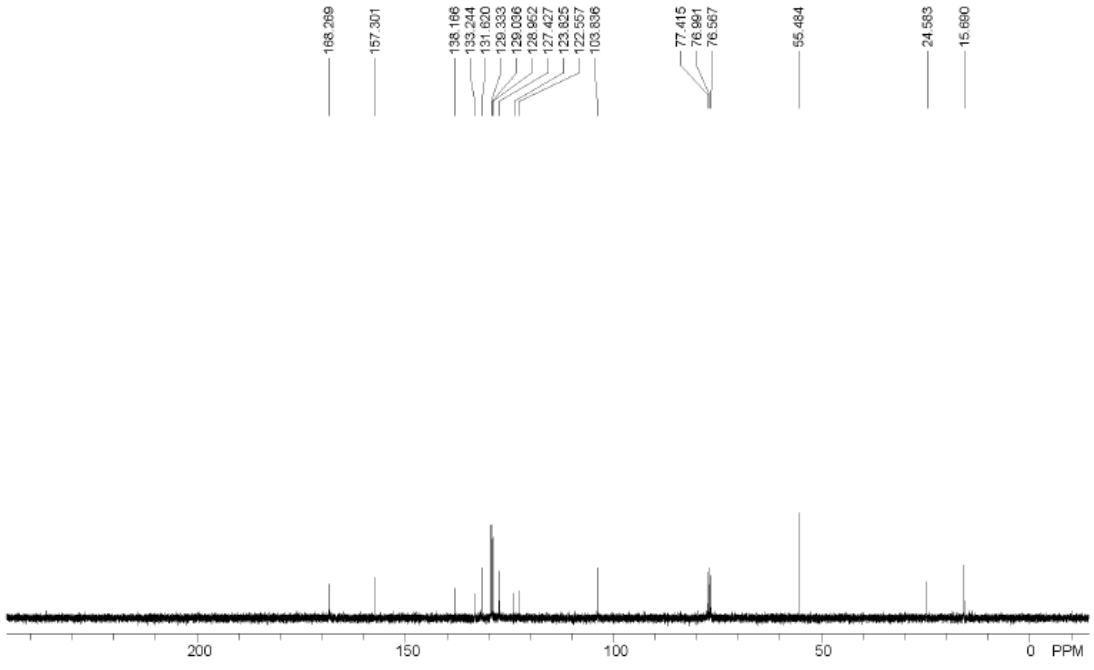

7.436
7.429
7.423
7.415
7.408
7.401
7.388
7.384
7.376
7.369
7.363
7.357
7.346
7.336
7.329
7.321
7.315
7.308
7.303
7.270
7.096
7.006
6.608
2.341
2.287
1.993
0.004
5.95
1.24
0.74
0.93
3.63
3.00
14
12
10
8
6
4
2
PPM

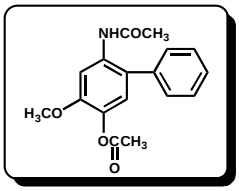

169.575
139.680
139.367
137.198
136.517
130.837
129.869
128.981
128.779
128.577
128.269
127.248
77.423
76.996
76.573
23.024
20.952
18.464
200
150
100
50
0
PPM


NHCOCH3
H3CO
CH3

3sa

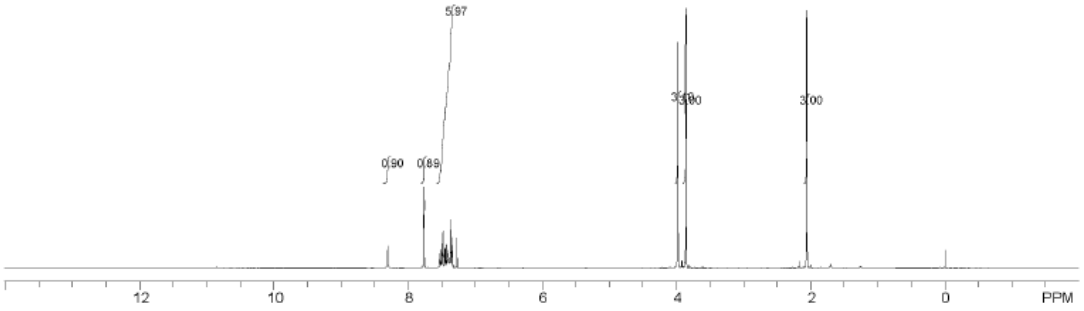

7.878
7.479
7.456
7.431
7.391
7.387
7.366
7.352
7.347
7.325
7.256
7.182
7.005
3.880
2.200
2.021
-0.002
4.70
2.97
0.81
12
10
8
6
4
2
0
PPM


168.269
157.301
138.166
133.244
131.620
129.333
129.036
128.852
127.427
123.825
122.557
103.836
77.415
76.991
76.567
55.484
24.583
15.680
200
150
100
50
0
PPM

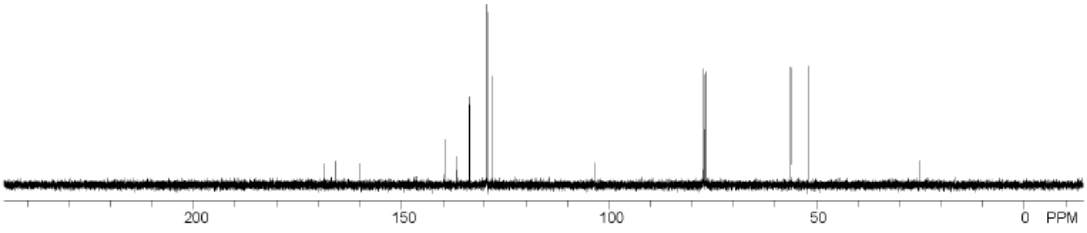

NHCOCH3
H3CO
OCCH3
O

3ta


8.302
7.502
7.450
7.426
7.402
7.371
7.366
7.349
7.344
7.339
7.270
3.979
3.868
2.058
0.002


5.97
3.00
3.00
0.90
0.89
12
10
8
6
4
2
0
PPM


168.501
165.866
159.823
139.608
136.648
133.574
129.345
129.320
128.141
103.556
77.414
76.988
76.564
56.191
51.869
25.012


200
150
100
50
0
PPM