Organocatalytic, Oxidative, Intermolecular Amination and Hydrazination of Simple Arenes at Ambient Temperature

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Supporting Information:

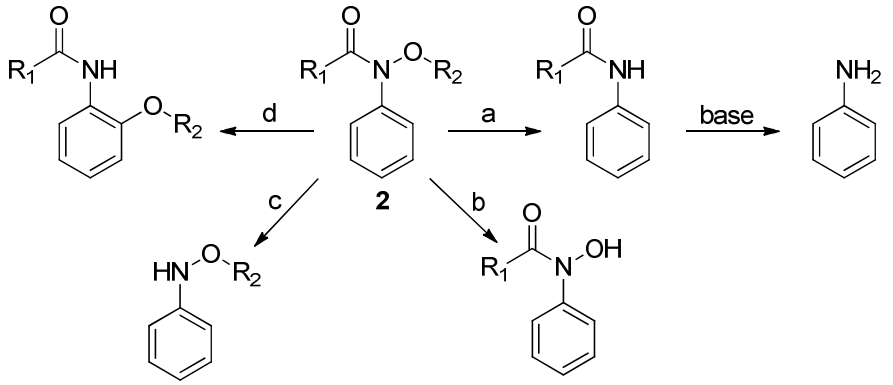
Contents:

General:

Unless otherwise noted, all commercially available compounds were used as provided without further purification. Solvents for chromatography were technical grade. Petroleum ether 40-60°C was used for column chromatography and thin layer chromatography. Dry solvents were purified by the Solvent Purification System M-BRAUN Glovebox Technology SPS-800. Analytical thin-layer chromatography (TLC) was performed on Merck silica gel aluminium plates with F-254 indicator, visualised by irradiation with UV light. Column chromatography was performed using silica gel Merck 60 (particle size 0.040-0.063 mm). Solvent mixtures are understood as volume/volume.

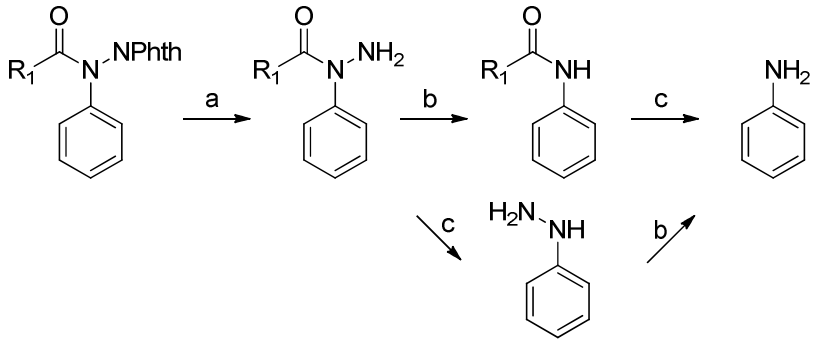
^{1}H-NMR and ^{13}C-NMR were recorded on Bruker DRX300 (300 MHz), Bruker DRX400 (400 MHz), DRX500 (500 MHz) and INOVA500 (500 MHz) spectrometers in $CDCl_3$, $(CD_3)_2SO$, CD_2Cl_2. Data are reported in the following order: chemical shift (δ) in ppm; multiplicities are indicated s (singlet), brs (broad singlet), d (doublet), t (triplet), q (quartet), m (multiplet); coupling constants (*J*) are given in Hertz (Hz). Mass spectra are recorded on a gas chromatograph (HP 6890) with mass detector (HP 5973), coupled to a J & W's fused silica GC column (GC column: stationary phase DB-5ms, 25 m × 0.202 mm× 0.33 µm) using the program (Acquisition time: 3 min, Initial temperature: 50 °C, Initial time: 1 min, Rate of temperature increasing: 40 °C, Final temperature: 300 °C, Final time: 15 min) or on a HPLC-MS system from HP Agilent 1100 series binary pump together with a reversed-phase HPLC column (CC250/4 Nucleosil 120-5 C4 by Macherey-Nagel, flow 1.0 mL/min, from 90% A to 100% B over 15 min; A = 0.1% HCOOH in H_2O, B = 0.1% HCOOH in CH_3CN). High resolution mass spectra were recorded on a LTQ Orbitrap mass spectrometer coupled to an Accela HPLC-System (HPLC column: Hypersyl GOLD, 50 mm × 1 mm × 1.9 µm). Fourier transform infrared spectroscopy (FT-IR) spectra were obtained with a Bruker Tensor 27 spectrometer (ATR, neat) and are reported in terms of frequency of absorption (cm^{-1}). Optical rotations were measured in a Schmidt + Haensch Polartronic HH8 polarimeter. Chemical yields refer to pure isolated substances.

Transformations of *N*-methoxy-*N*-phenylacetamides 2.



Reaction conditions: a) *hv*, MeCN, RT;[*] b) $AlCl_3$, Me_2S, RT;[†] c) NH_2-NH_2·H_2O, THF, RT;[‡] d) $AlCl_3$, THF, RT.[§]

Transformations of derivatives 5.



Reaction conditions: a) NH_2-NH_2·H_2O, EtOH:DCM(4:1), RT; 71% b) $TiCl_3$, EtOH, reflux;[**] c) base.

[*] Chowdhury, N.; Anoop, A.; Singh, N. D. P. *Synthesis*, **2012**, *44*, 1745.

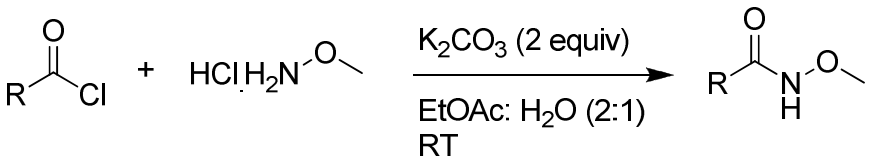
[†] Kawase, M.; Kitamura, T.; Shimada, M.; Kikugawa, Y. *Synth. Commun*. **1990**, *20*, 887.

[‡] Santos, P. F.; Lobo, A. M.; Prabhakar, S. *Synth. Commun*. **1995**, *25*, 3509.

[§] Kikugawa, Y.; Shimada, M. *J. Chem. Soc. Chem. Commun*. **1989**, 1450.

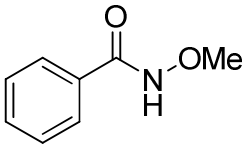
[**] Zhang, Y.; Tang, Q.; Luo, M. *Org. Biomol. Chem*. **2011**, *9*, 4977.

General Procedure for the synthesis of *N*-methoxy amide compounds:



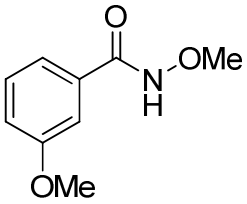
O-Methylhydroxylamine hydrochloride (596 mg, 7 mmol, 1 equiv) was dissolved in water (12 mL) and EtOAc (25 mL) and cooled to 0°C in an ice bath. K_2CO_3 (1.9 g, 14 mmol, 2 equiv) followed by acid chloride (6.3 mmol, 0.9 equiv) were added to the reaction mixture. After stirring at room temperature for 16 h, the aqueous layer was removed and the organic layer was washed with water and brine. The organic layer was dried over anhydrous $MgSO_4$ and concentrated under reduced pressure. The oil products were purified by silica gel column chromatography to get the pure product.

Spectroscopic data for *N*-methoxy amide derivatives:



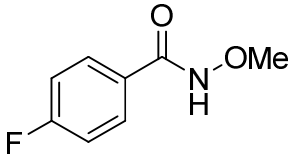
_N_-Methoxybenzamide[1] (1a)

White amorphous solid; Yield: 65%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.02 (brs, 1H), 7.77 – 7.71 (m, 2H), 7.55 – 7.49 (m, 1H), 7.43 (t, *J* = 7.5 Hz, 2H), 3.89 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.52, 132.03, 131.91, 128.63, 127.28, 64.35; FT-IR: $\tilde{\nu}$ = 3220, 1650, 1514, 1478, 13041242, 1150 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_8H_{10}O_2N$: 152.07061 found: 152.07043.



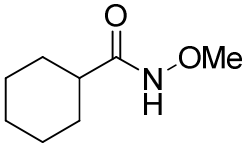
_N_,3-Dimethoxybenzamide[2] (1o)

Colour-less oil; Yield: 86%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 – 7.24 (m, 3H), 7.06 – 7.01 (m, 1H), 3.85 (s, 3H), 3.81 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.94, 133.19, 129.80, 119.03, 118.45, 112.34, 64.52, 55.56; FT-IR: $\tilde{\nu}$ = 3190, 2938, 1643, 1581, 1512, 1482, 14341290, 1240, 1136 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_9H_{12}O_3N$: 182.08117 found: 182.08106.



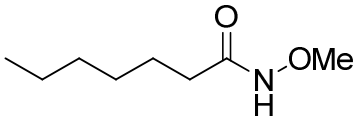
4-Fluoro-*N*-methoxybenzamide[3] **(1n)**

White amorphous solid; Yield: 97%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.78 (dd, *J* = 8.8, 5.3 Hz, 2H), 7.07 (t, *J* = 8.8 Hz, 2H), 3.83 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.37, 165.14 (d, *J* = 252.8 Hz), 129.66 (d, *J* = 8.9 Hz), 127.99, 115.87 (d, *J* = 21.9 Hz), 64.47; FT-IR: $\tilde{\nu}$ = 3222, 1643, 1599, 1486, 1228 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_8H_9O_2NF$: 170.06118 found: 170.06101.



***N*-Methoxycyclohexanecarboxamide (1p)**

White amorphous solid; Yield: 80%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.73 (s, 3H), 2.08-1.97 (s, 1H), 1.81-1.74 (m, 4H), 1.69-1.61 (m, 1H), 1.54 – 1.42 (m, 2H), 1.21 (d, *J* = 8.8 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 174.07, 64.30, 42.49, 29.31, 25.70; FT-IR: $\tilde{\nu}$ = 3211, 2926, 2852, 1650, 1514, 1437, 1388 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_8H_{16}O_2N$: 158.11756 found: 158.11742.

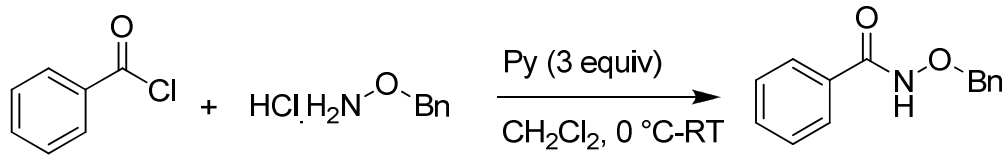


***N*-Methoxyheptanamide (1q)**

Colour-less oil; Yield: 72%; ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.72 (s, 3H), 2.08 (brs, 2H), 1.67-1.57(m, 2H), 1.26 (brs, 6H), 0.90-0.81 (m, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.34, 64.30,

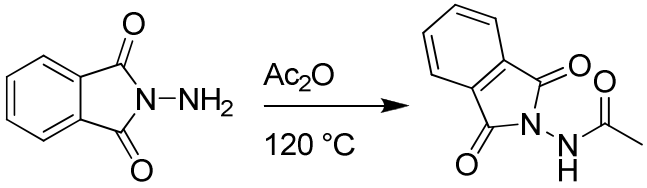
33.31, 31.57, 28.97, 25.49, 22.56, 14.08; FT-IR: $\tilde{\nu}$ = 3175, 2957, 2929, 2859, 1653, 1516, 1461, 1440, 1063 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_8H_{18}O_2N$: 160.13321 found: 160.13308.

Synthesis of *N*-(benzyloxy)benzamide (1m)



O-Benzylhydroxylamine hydrochloride (1.6 g, 10 mmol, 1 equiv) was dissolved in CH_2Cl_2 (15 mL) and cooled to 0 °C in an ice bath. Pyridine (2.5 mL, 30 mmol, 3 equiv) followed by benzoyl chloride (1.18 mL, 10 mmol, 1 equiv) were added to the reaction mixture at the same temperature. Then the mixture was slowly warmed to room temperature. After stirring at room temperature for 16 h, the reaction mixture was diluted with dichloromethane (100 mL) and washed with (1N) hydrochloric acid solution, water, brine. The organic layer was dried over anhydrous $MgSO_4$. The crude solid product was crystallized from EtOAc/petroleum ether (40-60 C) as white crystal in 75% yield. The analytical data was identical with the reported literature value.[4] m.p. 132-134 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 – 7.64 (m, 2H), 7.52 – 7.46 (m, 1H), 7.46 – 7.31 (m, 7H), 5.02 (s, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.13, 135.43, 132.10, 131.91, 129.41, 128.88, 128.76, 128.74, 127.15, 78.39; FT-IR: $\tilde{\nu}$ = 3228, 1640, 1575, 1507, 1482, 14521288, 1207 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{14}H_{14}O_2N$: 228.10191 found: 228.10213.

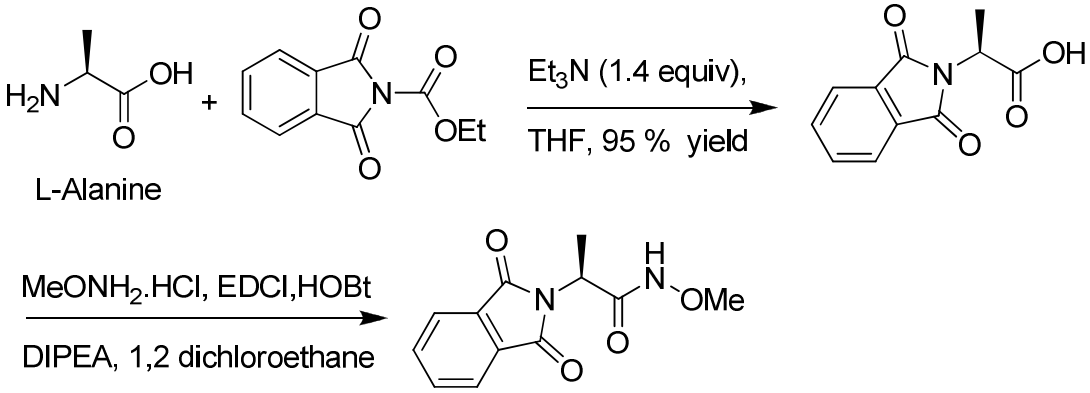
Synthesis of *N*-acetylaminophthalimide (4)



N-Aminophthalimide (1.65 g, 10 mmol) was mixed with acetic anhydride (4.82 mL, 50 mmol) in a round bottom flask and warmed to 120°, forming a homogeneous yellow solution which turned colorless as heating was continued for 1 h. Afterwards, the reaction mixture was

cooled to room temperature, forming colorless prismatic needles. The excess anhydride was drawn off and the residue washed with a small amount of cold ether to give *N*-acetylaminophthalimide in 59% yield. The analytical data were identical to the reported literature data.[5] ^{1}H NMR (500 MHz, DMSO-D_6) δ 10.64 (s, 1H), 8.01 – 7.86 (m, 4H), 2.05 (s, 3H); ^{13}C NMR (126 MHz, DMSO-D_6) δ 168.54, 165.17, 135.19, 129.46, 123.67, 20.11; FT-IR: $\tilde{\nu}$ = 3260, 1740, 1672, 1509, 1466, 1386, 1260, 1201, 1107 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{10}H_9O_3N_2$: 205.06077 found: 205.06082.

Synthesis of (*S*)-2-(1,3-dioxoisoindolin-2-yl)-*N*-methoxypropanamide (1r)



L-Alanine (2.0 g, 22 mmol) was taken in anhydrous THF (50 mL) then triethylamine (4.04 mL, 30.8 mmol) followed by *N*-(ethoxycarbonyl)phthalimide (5.07 g, 22.22 mmol) were added. The reaction mixture was refluxed for 24 h with vigorous stirring under argon atmosphere. The reaction mixture was filtered at ambient temp and the filtrate was concentrated under reduced pressure. The residue was dissolved in dichloromethane and extracted with 10% $NaHCO_3$ solution in water. The basic extract was treated with (2N) HCl to pH =2, and extracted with dichloromethane. The organic extract was washed with water and dried over anhydrous sodium sulfate. The organic extract was concentrated under reduced pressure and crystallized from toluene in 75% yield. The analytical data were identical to the reported literature data.[6]

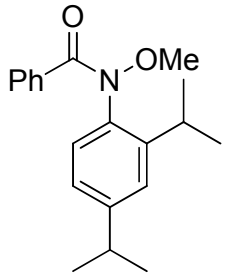
N-phthaloyl-*L*-alanine (548 mg, 2.5 mmol) was dissolved in 10 mL of 1,2 dichloroethane. 1-ethyl-3-(3-(dimethylamino)propyl)carbodiimide hydrochloride (586.8 mg, 3 mmol) and 1-hydroxybenzotriazole (413.6 mg, 3 mmol) were added to the reaction mixture and stirred under argon for 5 minutes. *O*-methylhydroxylamine hydrochloride (277 mg, 3.25 mmol) and

diisopropylethylamine (1.8 mL, 10 mmol) were added to the reaction mixture sequentially. The stirring was continued for 12 h. After completion of the reaction it was diluted with dichloromethane, washes with (1N) HCl solution, water, then with saturated $NaHCO_3$ solution and again with water, brine, dried over anhydrous magnesium sulfate. The organic extract was concentrated under reduced pressure and purified by silica gel column chromatography with 70% EtOAc in petroleum ether in 45% yield as white amorphous solid. ^{1}H NMR (300 MHz, DMSO-D_6) δ 11.36 (s, 1H), 7.99-7.77 (m, 4H), 4.72 (q, *J* = 7.2 Hz, 1H), 3.55 (s, 3H), 1.50 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, DMSO-D_6) δ 167.27, 165.66, 134.48, 131.82, 123.13, 63.14, 46.42, 14.69; FT-IR: $\tilde{\nu}$ = 3122, 2971, 1708, 1685, 1658, 1385, 1047 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{12}H_{13}O_4N_2$: 249.08698 found: 249.08704.

General Procedure for the organocatalytic intermolecular amination reaction

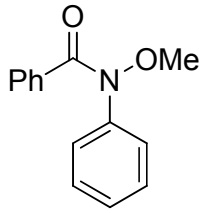
Amination reagent (0.2 mmol) was dissolved in a 4 mL screw-capped vial with 1 mL of 1,2 dichloroethane. Then arene (0.4-4 mmol), followed by organocatalyst (0.02 mmol), peracetic acid (39%, 74.6 µL, 0.44 mmol) and trifluoroacetic acid (78.6 µL, 1 mmol) were added at room temperature. The reaction mixture was stirred at this temperature for 0.6–26h. The reaction mixture was quenched with saturated solution of sodium thiosulphate and extracted with dichloromethane, washed with water, brine, dried over anhydrous Na_2SO_4. The organic extracts were filtered and concentrated under reduced pressure. Purification by column chromatography on silica gel (10% EtOAc in Pet. Ether-3% MeOH in dichloromethane) afforded the pure product.

Spectroscopic data of *N*-aryl-*N*-methoxybenzamides:



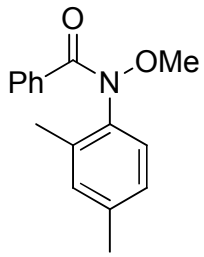
***N*-(2,4-Diisopropylphenyl)-*N*-methoxybenzamide (2i)**

Colour-less oil; ^{1}H NMR (500 MHz, CD_2Cl_2) δ 7.60 (brs, 2H), 7.41 (brs, 1H), 7.35 (brs, 2H), 7.25 (s, 1H), 7.15 (brs, 1H), 7.09 (brs, 1H), 3.67 (brs, 3H), 3.24 (dt, *J* = 13.7, 6.9 Hz, 1H), 2.93 (dt, *J* = 13.7, 6.8 Hz, 1H), 1.27 (s, 3H), 1.26 (s, 3H), 1.21 (s, 6H); ^{13}C NMR (126 MHz, CD_2Cl_2) δ 151.30, 147.75, 135.30, 131.04, 130.52, 129.01, 128.93, 128.45, 125.70, 124.71, 61.07, 34.70, 28.94, 24.22; FT-IR: $\tilde{\nu}$ = 2962, 2930, 2871, 1662, 1494, 1420, 1363 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{20}H_{26}O_2N$: 312.19581 found: 312.19590.



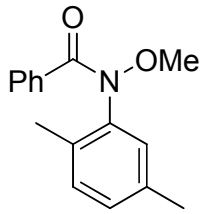
***N*-Methoxy-*N*-phenylbenzamide**[7] **(2b)**

Light yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56 – 7.52 (m, 2H), 7.39 – 7.25 (m, 7H), 7.18 (d, *J* = 7.4 Hz, 1H), 3.62 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.25, 139.39, 134.74, 130.78, 129.10, 128.59, 128.12, 127.20, 124.52, 61.78; FT-IR: $\tilde{\nu}$ = 2932, 1718, 1488, 1347, 1304 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{14}H_{14}O_2N$: 228.10191 found: 228.10194.



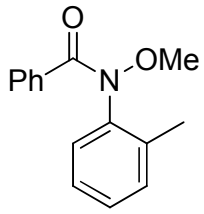
***N*-(2,4-Dimethylphenyl)-*N*-methoxybenzamide (2j)**

Light yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 – 7.43 (m, 2H), 7.42 – 7.26 (m, 3H), 7.13 – 7.00 (m, 2H), 6.97 (s, 1H), 3.72 (s, 3H), 2.30 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 139.46, 136.46, 132.14, 130.70, 128.96, 128.66, 128.43, 128.14, 128.02, 127.29, 60.73, 21.25, 18.05; FT-IR: $\tilde{\nu}$ = 2926, 1657, 1496, 1446, 1038 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{16}H_{18}O_2N$: 256.13321 found: 256.13323.



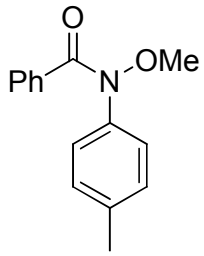
***N*-(2,5-Dimethylphenyl)-*N*-methoxybenzamide (2h)**

Colour-less oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.57 (s, 2H), 7.38 (s, 1H), 7.31 (s, 2H), 7.13 (d, *J* = 7.8 Hz, 1H), 7.07 (d, *J* = 7.3 Hz, 2H), 3.71 (s, 3H), 2.28 (s, 6H); ^{13}C NMR (126 MHz, $CDCl_3$)δ 136.48, 133.57, 133.51, 131.17, 130.78, 130.24, 130.20, 128.52, 128.44, 128.04, 60.97, 20.86, 17.69; FT-IR: $\tilde{\nu}$ = 2925, 1718, 1446, 1342, 1171 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{16}H_{18}O_2N$: 256.13321 found: 256.13318.



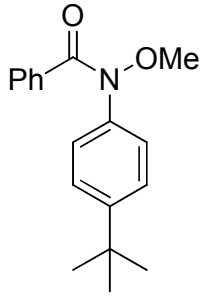
***N*-Methoxy-*N*-*o*-tolylbenzamide**[7] **(2e *ortho* isomer)**

Colour-less oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.67 – 7.06 (m, 9H), 3.72 (s, 3H), 2.36 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 136.84, 134.48, 131.44, 130.76, 129.29, 128.45, 128.06, 126.62, 60.96, 18.21; FT-IR: $\tilde{\nu}$ = 2932, 1658, 1489, 1446, 1343, 1021 cm^{-1}; HRMS: calc. for $[M+Na]^+$ $C_{15}H_{15}O_2NNa$: 264.09950 found: 264.09963.



***N*-Methoxy-*N*-*p*-tolylbenzamide**[7] **(2e *para* isomer)**

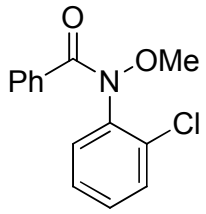
Colour-less oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 (d, *J* = 8.2 Hz, 2H), 7.39 (t, *J* = 7.4 Hz, 1H), 7.33 (t, *J* = 7.4 Hz, 2H), 7.28 (d, *J* = 8.2 Hz, 2H), 7.15 (d, *J* = 8.2 Hz, 2H), 3.70 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.13, 137.48, 136.89, 134.80, 130.67, 129.75, 128.62, 128.08, 125.09, 61.52, 21.20; FT-IR: $\tilde{\nu}$ = 2931, 1658, 1506, 1341, 1304 cm^{-1}; HRMS: calc. for $[M+Na]^+$ $C_{15}H_{15}O_2NNa$: 264.09950 found: 264.09962.



***N*-(4-*tert*-Butylphenyl)-*N*-methoxybenzamide (2f *para* isomer)**

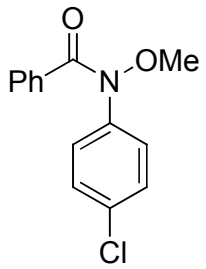
Light yellow oil; ^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.62 (d, *J* = 7.1 Hz, 2H), 7.46 – 7.34 (m, 7H), 3.66 (s, 3H), 1.33 (s, 9H); ^{13}C NMR (101 MHz, CD_2Cl_2) δ 168.62, 150.94, 137.21, 135.56,

131.01, 128.83, 128.48, 126.39, 124.80, 62.01, 35.05, 31.57; FT-IR: $\tilde{\nu}$ = 2961, 1660, 1509, 1340, 1308 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{18}H_{22}O_2N$: 284.16451 found: 284.16451.



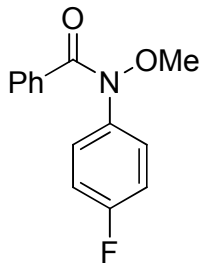
***N*-(2-Chlorophenyl)-*N*-methoxybenzamide**[8] **(2d *ortho* isomer)**

Colour-less oil; ^1H NMR (500 MHz, $CDCl_3$) δ 7.62 (d, *J* = 5.7 Hz, 2H), 7.48 – 7.35 (m, 3H), 7.34 – 7.27 (m, 4H), 3.82 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 140.40, 134.13, 133.55, 131.06, 130.75, 130.48, 129.26, 128.53, 128.11, 127.79, 62.00; FT-IR: $\tilde{\nu}$ = 1665, 1474, 1444, 1341, 1270, 1020 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{14}H_{13}O_2N^{35}Cl$: 262.06293 found: 262.06309; $[M+H]^+$ $C_{14}H_{13}O_3N^{37}Cl$: 264.05998 found: 264.05992.



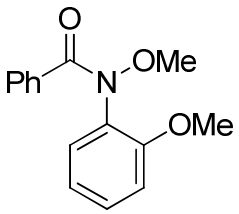
***N*-(4-Chlorophenyl)-*N*-methoxybenzamide**[8] **(2d *para* isomer)**

Colour-less oil; ^1H NMR (400 MHz, CD_2Cl_2) δ 7.66 – 7.60 (m, 2H), 7.51 – 7.31 (m, 7H), 3.63 (s, 3H); ^{13}C NMR (101 MHz, CD_2Cl_2) δ 168.67, 138.49, 135.10, 132.45, 131.32, 129.49, 128.82, 128.59, 125.43, 62.36; FT-IR: $\tilde{\nu}$ = 1660, 1486, 1333, 1295, 1092 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{14}H_{13}O_2N^{35}Cl$: 262.06293 found: 262.06294; $[M+H]^+$ $C_{14}H_{13}O_3N^{37}Cl$: 264.05998 found: 264.05988.



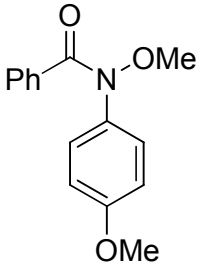
***N*-(4-Fluorophenyl)-*N*-methoxybenzamide (2c *para* isomer)**

Light yellow oil; ^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.64 – 7.58 (m, 2H), 7.48 – 7.42 (m, 3H), 7.41 – 7.35 (m, 2H), 7.12 – 7.05 (m, 2H), 3.65 (s, 3H); ^{13}C NMR (101 MHz, CD_2Cl_2) δ 168.69, 161.71 (d, *J* = 246.6 Hz), 135.97 (d, *J* = 2.8 Hz), 135.10, 131.21, 128.82, 128.55, 127.07 (d, *J* = 8.3 Hz), 116.26 (d, *J* = 22.9 Hz), 62.08; FT-IR: $\tilde{\nu}$ = 1662, 1503, 1344, 1234 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{14}H_{13}O_2NF$: 246.09248 found: 246.09246.



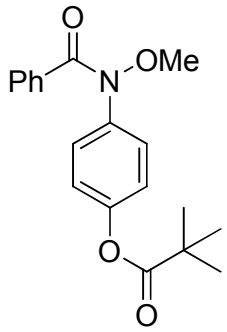
***N*-Methoxy-*N*-(2-methoxyphenyl)benzamide[8] (2a *ortho* isomer)**

Light yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.51 (d, *J* = 7.5 Hz, 2H), 7.35 (dd, *J* = 7.8, 1.6 Hz, 1H), 7.32 – 7.27 (m, 2H), 7.22 (t, *J* = 7.5 Hz, 2H), 6.94 (td, *J* = 7.8, 1.6 Hz, 1H), 6.82 (d, *J* = 8.3 Hz, 1H), 3.84 (s, 3H), 3.69 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 169.66, 155.20, 134.77, 130.86, 130.51, 130.15, 129.32, 128.38, 127.73, 121.03, 112.26, 61.55, 55.70; FT-IR: $\tilde{\nu}$ = 2930, 1665, 1460, 1342, 1281, 1261, 1020 cm^{-1}; HRMS: calc. for $[M+Na]^+$ $C_{15}H_{15}O_3NNa$: 280.09441 found: 280.09448.



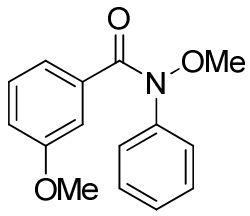
***N*-Methoxy-*N*-(4-methoxyphenyl)benzamide[8] (2a *para* isomer)**

Light yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (d, *J* = 7.3 Hz, 2H), 7.38 (t, *J* = 7.3 Hz, 1H), 7.34-7.26 (m, 4H), 6.86 (d, *J* = 8.9 Hz, 2H), 3.80 (s, 3H), 3.71 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.12, 159.00, 134.67, 132.29, 130.66, 128.66, 128.10, 127.50, 114.40, 61.30, 55.61; FT-IR: $\tilde{\nu}$ = 2929, 1656, 1506, 1354, 1247, 1026 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{15}H_{16}O_3N$: 258.11247 found: 258.11247.



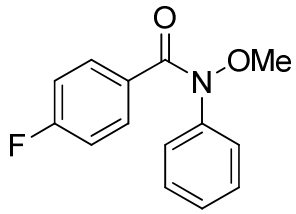
4-(*N*-Methoxybenzamido)phenyl pivalate (2g *para* isomer)

Light yellow amorphous solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, *J* = 7.2 Hz, 2H), 7.51 – 7.32 (m, 5H), 7.07 (d, *J* = 8.9 Hz, 2H), 3.68 (s, 3H), 1.35 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.02, 168.22, 149.64, 136.64, 134.50, 130.90, 128.59, 128.19, 125.34, 122.15, 61.83, 39.26, 27.23; FT-IR: $\tilde{\nu}$ = 2973, 1749, 1662, 1500, 1340, 1164, 1108 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{19}H_{22}O_4N$: 328.15433 found: 328.15453.



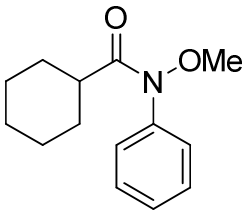
***N*,3-Dimethoxy-*N*-phenylbenzamide (2o)**

Light yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, *J* = 7.9 Hz, 2H), 7.36 (t, *J* = 7.9 Hz, 2H), 7.29-7.19 (m, 2H), 7.18 – 7.12 (m, 2H), 6.97 – 6.92 (m, 1H), 3.77 (s, 3H), 3.71 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.91, 159.24, 139.35, 135.86, 129.19, 129.11, 127.29, 124.63, 120.92, 116.98, 113.59, 61.77, 55.44; FT-IR: $\tilde{\nu}$ = 2933, 1661, 1581, 1487, 1346, 1306, 1287, 1258, 1036 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{15}H_{16}O_3N$: 258.11247 found: 258.11253.



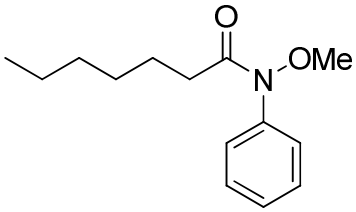
4-Fluoro-*N*-methoxy-*N*-phenylbenzamide (2n)

White amorphous solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (dd, *J* = 8.7, 5.4 Hz, 2H), 7.44 (d, *J* = 7.9 Hz, 2H), 7.37 (t, *J* = 7.9 Hz, 2H), 7.26 (t, *J* = 7.9 Hz, 1H), 7.03 (t, *J* = 8.7 Hz, 2H), 3.69 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.05, 164.12 (d, *J* = 251.6 Hz), 139.22, 131.18 (d, *J* = 8.8 Hz), 130.59 (d, *J* = 3.3 Hz), 129.18, 127.36, 124.52, 115.24 (d, *J* = 21.8 Hz), 61.78; FT-IR: $\tilde{\nu}$ = 1658, 1599, 1489, 1455, 1348, 1306, 1227, 1157 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{14}H_{13}O_2NF$: 246.09248 found: 246.09258.



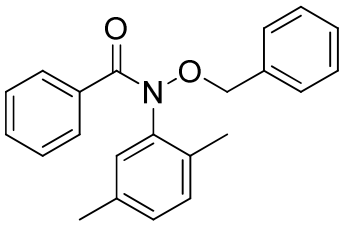
***N*-Methoxy-*N*-phenylcyclohexanecarboxamide (2p)**

Light yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (d, *J* = 7.8 Hz, 2H), 7.39 (t, *J* = 7.8 Hz, 2H), 7.28-7.20 (m, 1H), 3.70 (s, 3H), 2.72 (brs, 1 H), 1.92 – 1.74 (m, 4H), 1.69 (brs, 1H), 1.61-1.48 (m, 2H), 1.36-1.18 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.54, 138.56, 128.94, 126.72, 123.29, 62.07, 41.42, 29.18, 25.86, 25.83; FT-IR: $\tilde{\nu}$ = 2928, 2853, 1674, 1490, 1450, 1378, 1339, 1270 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{14}H_{20}O_2N$: 234.14886 found: 234.14897.



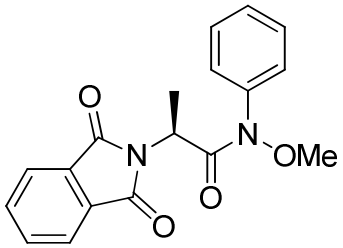
***N*-Methoxy-*N*-phenylheptanamide (2q)**

Light yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (d, *J* = 7.9 Hz, 2H), 7.39 (t, *J* = 7.9 Hz, 2H), 7.29 – 7.19 (m, 1H), 3.70 (s, 3H), 2.53 (brs, 2H), 1.73 – 1.63 (m, 2H), 1.42 – 1.22 (m, 6H), 0.88 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.37, 138.41, 128.96, 126.68, 123.25, 61.84, 33.55, 31.74, 29.16, 24.73, 22.66, 14.19; FT-IR: $\tilde{\nu}$ = 2955, 2929, 2856, 1681, 1491, 1376, 1282, 1064 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{14}H_{22}O_2N$: 236.16451 found: 236.16468.



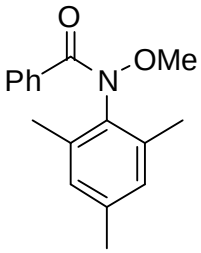
***N*-(Benzyloxy)-*N*-(2,5-dimethylphenyl)benzamide (2m)**

Light yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (brs, 2H), 7.46 – 7.24 (m, 6H), 7.22 – 6.90 (m, 5H), 4.89 (brs, 2H), 2.31 (s, 3H), 2.29 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 136.37, 133.48, 131.15, 130.62, 130.06, 129.53, 128.74, 128.51, 128.49, 128.00, 75.80, 20.94, 17.87; FT-IR: $\tilde{\nu}$ = 1661, 1504, 1448, 1412, 1341 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{22}H_{22}O_2N$: 332.16451 found: 332.16507.



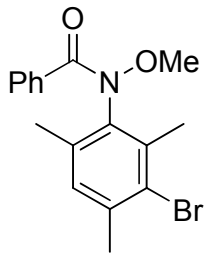
(*S*)-2-(1,3-Dioxoisoindolin-2-yl)-*N*-methoxy-*N*-phenylpropanamide (2r)

Light yellow oil; ^{1}H NMR (300 MHz, CD_2Cl_2) δ 7.85 (dd, *J* = 5.3, 3.1 Hz, 2H), 7.80 – 7.69 (m, 2H), 7.52 – 7.31 (m, 4H), 7.31 – 7.19 (m, 1H), 5.30 – 5.21 (m, 1H), 3.65 (s, 3H), 1.72 (d, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, CD_2Cl_2) δ 168.06, 138.40, 134.67, 132.40, 129.38, 127.34, 123.76, 62.62, 48.50, 15.39; FT-IR: $\tilde{\nu}$ = 1709, 1683, 1381, 1336, 1275, 1064 cm^{-1}; HRMS: calc. for $[M+Na]^+$ $C_{18}H_{16}O_4N_2Na$: 347.10023 found: 347.10087; $[\alpha]_D^{RT}$ = -19.2 (c = 1.0 in CH_2Cl_2).



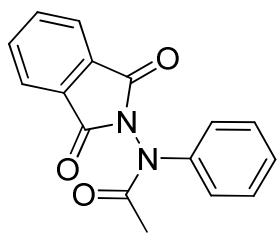
***N*-Mesityl-*N*-methoxybenzamide (2k)**

Colour-less oil; (mixture of rotamers in 1.15:1 ratio): # denotes major rotamer and * denotes minor rotamer; ^{1}H NMR (500 MHz, $CDCl_3$) δ *7.81 (d, *J* = 6.9 Hz, 2H), #*7.52 – 7.42 (m, 3H), *7.33 (d, *J* = 7.5 Hz, 2H), #7.28 (t, *J* = 7.5 Hz, 1H), #7.17 (t, *J* = 7.7 Hz, 2H), *6.97 (s, 2H), #6.84 (s, 2H), #3.86 (s, 3H), *3.49 (s, 3H), *2.35 (s, 6H), *2.32 (s, 3H), #2.25 (s, 3H), #2.23 (s, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 169.06, 167.34, 139.57, 139.31, 137.08, 136.77, 134.93, 134.85, 132.89, 130.74, 130.61, 129.70, 129.44, 128.17, 128.12, 128.11, 127.86, 60.87, 60.47, 21.21, 21.15, 18.65, 18.39; FT-IR: $\tilde{\nu}$ = 2927, 1656, 1446, 1375 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{17}H_{20}O_2N$: 270.14886 found: 270.14886.



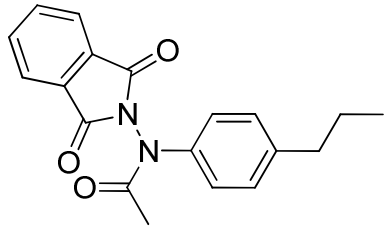
***N*-(3-Bromo-2,4,6-trimethylphenyl)-*N*-methoxybenzamide (2l)**

Colour-less oil; (mixture of rotamers in 1.5:1 ratio): ^{1}H NMR (500 MHz, CD_2Cl_2) # denotes major rotamer and * denotes minor rotamer; δ #7.82 (d, *J* = 7.7 Hz, 2H), *7.56 – 7.46 (m, 5H), #7.32 (d, *J* = 7.7Hz, 2H), #7.21 (t, *J* = 7.7 Hz, 1H), #7.14 (s, 1H), *6.97 (s, 1H), *3.85 (s, 2H), #3.51 (s, 3H), #2.47 (s, 3H), #2.45 (s, 3H), *2.42 (s, 3H), *2.36 (s, 3H), #2.32 (s, 3H), *2.20 (s, 3H); ^{13}C NMR (126 MHz, CD_2Cl_2) δ 169.14, 167.49, 140.32, 140.00, 137.91, 137.75, 136.76, 136.63, 136.21, 134.92, 134.77, 133.72, 131.06, 130.71, 130.52, 130.31, 128.80, 128.43, 128.37, 128.24, 125.94, 125.65, 61.30, 60.78, 24.15, 24.10, 20.55, 19.93, 18.59, 18.31; FT-IR: $\tilde{\nu}$ = 2930, 1659, 1447, 1365 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{17}H_{19}O_2N^{79}Br$: 348.05937 found: 348.05976; $[M+H]^+$ $C_{17}H_{19}O_2N^{81}Br$: 350.05732 found: 350.05750.



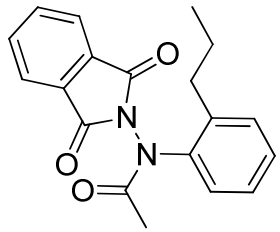
***N*-(1,3-Dioxoisoindolin-2-yl)-*N*-phenylacetamide[9] (5a)**

White amorphous solid; (major rotamer) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.75 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.68 (d, *J* = 6.9 Hz, 2H), 7.49 – 7.38 (m, 3H), 2.10 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.50, 165.00, 140.71, 134.76, 130.10, 129.98, 129.70, 128.91, 124.07, 21.77; FT-IR: $\tilde{\nu}$ = 1732, 1690, 1385, 1369, 1313, 1264, 1115 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{16}H_{13}O_3N_2$: 281.09207 found: 281.09220.



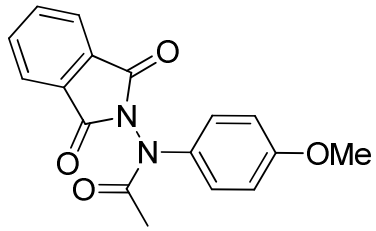
***N*-(1,3-Dioxoisoindolin-2-yl)-*N*-(4-propylphenyl)acetamide (5e *para* isomer)**

White amorphous solid; (major rotamer) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.75 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.58 (d, *J* = 8.3 Hz, 2H), 7.24 (d, *J* = 8.3 Hz, 2H), 2.60 (t, *J* = 7.5 Hz, 2H), 2.10 (s, 3H), 1.70 – 1.56 (m, 2H), 0.93 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.76, 165.04, 144.66, 138.37, 134.72, 130.20, 129.95, 128.72, 124.06, 37.77, 24.52, 21.78, 13.90; FT-IR: $\tilde{\nu}$ = 2959, 2930, 1794, 1735, 1507, 1369, 1310, 1262, 1112 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{19}H_{19}O_3N_2$: 323.13902 found: 323.13925.



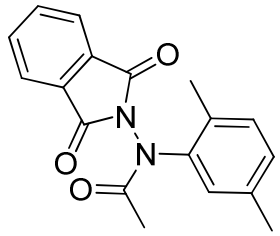
***N*-(1,3-Dioxoisoindolin-2-yl)-*N*-(2-propylphenyl)acetamide (5e *ortho* isomer)**

White amorphous solid; (major rotamer) ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.91 – 7.86 (m, 2H), 7.85 – 7.82 (m, 1H), 7.79 – 7.74 (m, 2H), 7.39 – 7.35 (m, 2H), 7.29 – 7.24 (m, 1H), 3.18-3.10 (m, 1H), 2.88-2.80 (m, 1H), 2.02 (s, 3H), 1.77 – 1.63 (m, 2H), 1.09 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 169.22, 165.96, 165.02, 141.85, 138.89, 134.84, 134.69, 130.38, 130.26, 130.15, 129.99, 127.29, 124.09, 124.00, 32.38, 24.03, 21.39, 14.39; FT-IR: $\tilde{\nu}$ = 2961, 2871, 1737, 1694, 1367, 1301, 1257 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{19}H_{19}O_3N_2$: 323.13902 found: 323.13915.



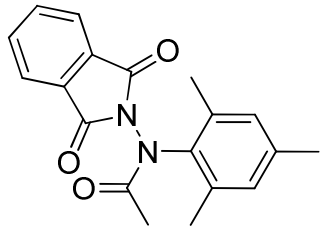
***N*-(1,3-Dioxoisoindolin-2-yl)-*N*-(4-methoxyphenyl)acetamide (5b *para* isomer)**

White amorphous solid; (major rotamer) ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.86 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.74 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.61 (d, *J* = 9.0 Hz, 2H), 6.92 (d, *J* = 9.0 Hz, 2H), 3.81 (s, 3H), 2.08 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.90, 164.97, 160.34, 134.69, 133.54, 130.38, 130.20, 124.02, 114.98, 55.67, 21.69; FT-IR: $\tilde{\nu}$ = 1734, 1692, 1506, 1370, 1247 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{17}H_{15}O_4N_2$: 311.10263 found: 311.10277.



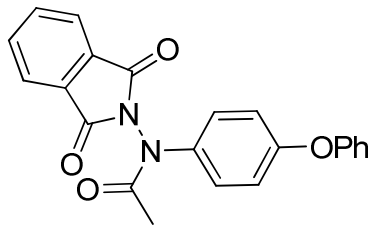
***N*-(2,5-Dimethylphenyl)-*N*-(1,3-dioxoisoindolin-2-yl)acetamide (5q)**

White amorphous solid; (major rotamer) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 – 7.83 (m, 2H), 7.75 (dd, *J* = 5.5, 3.0 Hz, 2H), 7.58 (s, 1H), 7.19 (d, *J* = 7.8 Hz, 1H), 7.12 (d, *J* = 6.7 Hz, 1H), 2.60 (s, 3H), 2.32 (s, 3H), 2.04 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.93, 165.72, 165.12, 139.22, 137.50, 135.29, 134.82, 134.69, 134.09, 131.57, 130.69, 129.75, 127.49, 124.38, 124.01, 20.95, 20.90, 17.75; FT-IR: $\tilde{\nu}$ = 1735, 1694, 1367, 1307, 1283, 1259 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{18}H_{17}O_3N_2$: 309.12337 found: 309.12345.



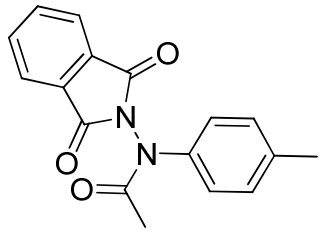
***N*-(1,3-Dioxoisoindolin-2-yl)-*N*-mesitylacetamide (5r)**

White amorphous solid; (major rotamer) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.74 (dd, *J* = 5.5, 3.1 Hz, 2H), 6.96 (s, 2H), 2.43 (s, 6H), 2.29 (s, 3H), 2.04 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.68, 164.57, 139.27, 137.47, 134.69, 134.45, 130.21, 130.07, 123.87, 21.07, 20.80, 19.27; FT-IR: $\tilde{\nu}$ = 2919, 1734, 1686, 1466, 1368, 1312, 1250 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{19}H_{19}O_3N_2$: 323.13902 found: 323.13921.



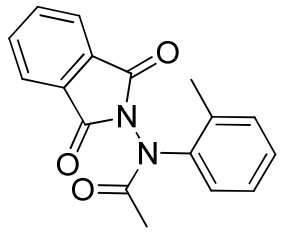
***N*-(1,3-Dioxoisoindolin-2-yl)-*N*-(4-phenoxyphenyl)acetamide (5c *para* isomer)**

White amorphous solid; (major rotamer) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.76 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.64 (d, *J* = 8.8 Hz, 2H), 7.40 – 7.33 (m, 2H), 7.19 – 7.13 (m, 1H), 7.05 – 6.99 (m, 4H), 2.12 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.71, 164.96, 158.61, 156.00, 135.28, 134.76, 130.56, 130.10, 124.40, 124.06, 120.21, 119.80, 119.07, 21.76; FT-IR: $\tilde{\nu}$ = 1736, 1696, 1487, 1369, 1315, 1262, 1230 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{22}H_{17}O_4N_2$: 373.11828 found: 373.11835.



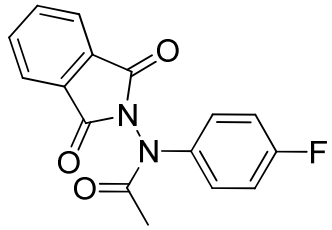
***N*-(1,3-Dioxoisoindolin-2-yl)-*N*-*p*-tolylacetamide (5d *para* isomer)**

White amorphous solid; (major rotamer) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.74 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.56 (d, *J* = 8.2 Hz, 2H), 7.24 (d, *J* = 8.2 Hz, 2H), 2.37 (s, 3H), 2.09 (s, 3H)L; ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.71, 164.98, 139.92, 138.20, 134.71, 130.53, 130.14, 128.71, 124.03, 21.74, 21.32; FT-IR: $\tilde{\nu}$ = 1735, 1694, 1369, 1312, 1262, cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{17}H_{15}O_3N_2$: 295.10772 found: 295.10783.



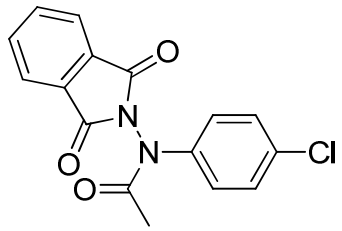
***N*-(1,3-Dioxoisoindolin-2-yl)-*N*-*o*-tolylacetamide (5d *ortho* isomer)**

White amorphous solid; (major rotamer) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 – 7.85 (m, 2H), 7.81 – 7.74 (m, 3H), 7.32 (d, *J* = 4.0 Hz, 2H), 7.29 – 7.25 (m, 1H), 2.65 (s, 3H), 2.04 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.92, 166.07, 165.75, 139.49, 137.52, 135.33, 134.86, 134.74, 131.84, 129.92, 129.73, 127.57, 124.41, 124.07, 21.03, 18.24; FT-IR: $\tilde{\nu}$ = 1736, 1695, 1369, 1305, 1259 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{17}H_{15}O_3N_2$: 295.10772 found: 295.10776.



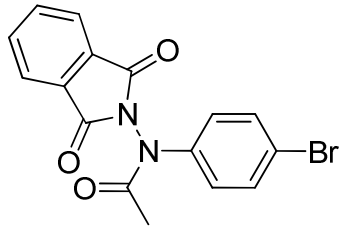
***N*-(1,3-Dioxoisoindolin-2-yl)-*N*-(4-fluorophenyl)acetamide (5f *para* isomer)**

White amorphous solid; (major rotamer) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.76 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.72 – 7.65 (m, 2H), 7.12 (t, *J* = 8.5 Hz, 2H), 2.08 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.42, 164.94, 162.90 (d, *J* = 250.5 Hz), 136.73 (d, *J* = 3.3 Hz), 134.84, 131.09 (d, *J* = 9.0 Hz), 130.03, 124.12, 116.93 (d, *J* = 22.9 Hz), 21.72; FT-IR: $\tilde{\nu}$ = 1734, 1688, 1500, 1384, 1366, 1315, 1288, 1261, 1222, 1192 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{16}H_{12}O_3N_2F$: 299.08265 found: 299.08274.



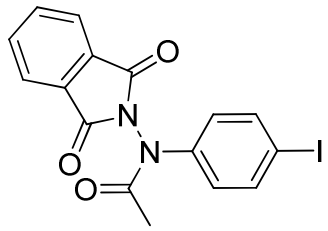
***N*-(4-Chlorophenyl)-*N*-(1,3-dioxoisoindolin-2-yl)acetamide (5g *para* isomer)**

White amorphous solid; (major rotamer) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.76 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.62 (d, *J* = 8.6 Hz, 2H), 7.42 (d, *J* = 8.6 Hz, 2H), 2.10 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.18, 164.93, 139.17, 135.71, 134.87, 130.33, 130.21, 130.00, 124.15, 21.72; FT-IR: $\tilde{\nu}$ = 1735, 1698, 1487, 1369, 1308, 1259, 1087 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{16}H_{12}O_3N_2{}^{35}Cl$: 315.05310 found: 315.05337; $[M+H]^+$ $C_{16}H_{12}O_3N_2{}^{37}Cl$: 317.05015 found: 317.05026.



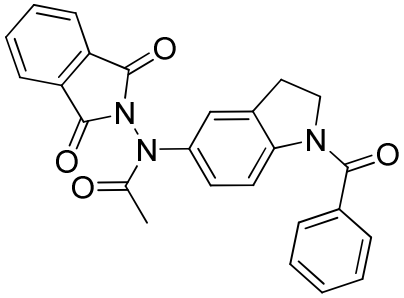
***N*-(4-Bromophenyl)-*N*-(1,3-dioxoisoindolin-2-yl)acetamide (5h *para* isomer)**

White amorphous solid; (major rotamer) ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.89 (dd, *J* = 5.4, 3.2 Hz, 2H), 7.77 (dd, *J* = 5.4, 3.2 Hz, 2H), 7.61 – 7.54 (m, 4H), 2.11 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.11, 164.94, 139.73, 134.88, 133.25, 130.62, 130.03, 124.16, 123.84, 21.71; FT-IR: $\tilde{\nu}$ = 1734, 1699, 1485, 1369, 1308, 1259, 1068 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{16}H_{12}O_3N_2{}^{79}Br$: 359.00258 found: 359.00291; $[M+H]^+$ $C_{16}H_{12}O_3N_2{}^{81}Br$: 361.00053 found: 361.00072.



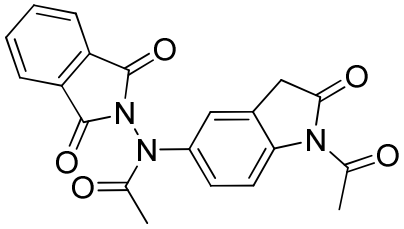
***N*-(1,3-Dioxoisoindolin-2-yl)-*N*-(4-iodophenyl)acetamide[10] (5i *para* isomer)**

White amorphous solid; (major rotamer) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.81 – 7.73 (m, 4H), 7.41 (d, *J* = 8.4 Hz, 2H), 2.10 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.12, 164.92, 140.39, 139.27, 134.88, 130.74, 130.00, 124.16, 95.57, 21.74; FT-IR: $\tilde{\nu}$ = 1735, 1698, 1482, 1369, 1308, 1259 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{16}H_{12}O_3N_2I$: 406.98871 found: 406.98831.



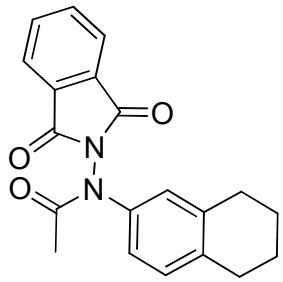
_N_-(1-Benzoylindolin-5-yl)-_N_-(1,3-dioxoisoindolin-2-yl)acetamide (5n)

White amorphous solid; (major rotamer) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.79 – 7.74 (m, 2H), 7.57 – 7.39 (m, 8H), 4.16 – 4.05 (m, 2H), 3.14 (t, *J* = 8.3 Hz, 2H), 2.11 (s, 3H); ^{13}C NMR of mixture (101 MHz, $CDCl_3$) δ 169.47, 168.87, 168.79, 165.24, 165.00, 164.71, 141.40, 137.45, 136.37, 135.39, 134.81, 134.78, 134.51, 131.31, 130.84, 130.13, 130.10, 129.31, 128.83, 128.36, 127.17, 126.22, 126.02, 125.99, 125.47, 124.46, 124.10, 124.08, 54.01, 21.76, 20.74; FT-IR: $\tilde{\nu}$ = 1734, 1695, 1644, 1483, 1372, 1266 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{25}H_{20}O_4N_3$: 426.14483 found: 426.14456.



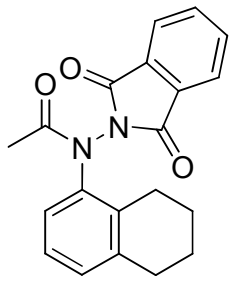
_N_-(1-Acetyl-2-oxoindolin-5-yl)-_N_-(1,3-dioxoisoindolin-2-yl)acetamide (5o)

White amorphous solid; (major rotamer) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30 (d, *J* = 8.7 Hz, 1H), 7.89 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.77 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.65 (dd, *J* = 8.7, 2.0 Hz, 1H), 7.62 (s, 1H), 3.75 (s, 2H), 2.67 (s, 3H), 2.10 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.66, 170.90, 168.40, 165.01, 142.20, 137.43, 134.89, 130.04, 129.34, 125.31, 124.71, 124.17, 117.89, 36.53, 26.80, 21.76; FT-IR: $\tilde{\nu}$ = 1757, 1736, 1701, 1482, 1371, 1292 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{20}H_{16}O_5N_3$: 378.10845 found: 378.10858.



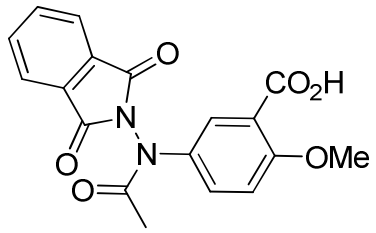
***N*-(1,3-Dioxoisoindolin-2-yl)-*N*-(5,6,7,8-tetrahydronaphthalen-2-yl)acetamide (5p major isomer)**

White amorphous solid; (major rotamer) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.74 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.41 – 7.34 (m, 2H), 7.11 (d, *J* = 8.3 Hz, 1H), 2.77 (d, *J* = 3.5 Hz, 4H), 2.11 (s, 3H), 1.84-1.74 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.86, 164.99, 139.10, 139.08, 138.00, 134.69, 130.51, 130.17, 129.10, 125.76, 124.01, 29.40, 29.27, 22.96, 22.84, 21.76; FT-IR: $\tilde{\nu}$ = 2928, 1736, 1692, 1368, 1314, 1264 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{20}H_{19}O_3N_2$: 335.13902 found: 335.13955.



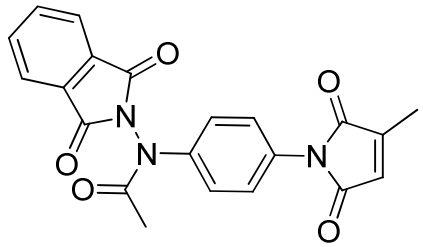
***N*-(1,3-Dioxoisoindolin-2-yl)-*N*-(5,6,7,8-tetrahydronaphthalen-1-yl)acetamide (5p minor isomer)**

White amorphous solid; (major rotamer) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 – 7.85 (m, 2H), 7.76 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.64 (dd, *J* = 6.9, 2.1 Hz, 1H), 7.20 – 7.12 (m, 2H), 3.43 (dt, *J* = 17.4, 6.1 Hz, 1H), 2.87-2.75 (m, 3H), 2.05 (s, 3H), 1.92 – 1.72 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.12, 165.82, 165.22, 139.73, 139.18, 136.76, 134.82, 134.70, 130.85, 130.30, 130.08, 127.07, 126.59, 124.03, 124.00, 29.77, 25.35, 22.75, 21.10; FT-IR: $\tilde{\nu}$ = 2927, 1736, 1692, 1460, 1367, 1302, 1262 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{20}H_{19}O_3N_2$: 335.13902 found: 335.13938.



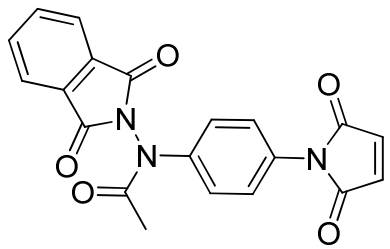
5-(*N*-(1,3-Dioxoisoindolin-2-yl)acetamido)-2-methoxybenzoic acid (5m)

White amorphous solid; (major rotamer) ^{1}H NMR (400 MHz, DMSO-D_6) δ 12.97 (s, 1H), 8.02 – 7.90 (m, 4H), 7.84 (d, *J* = 2.7 Hz, 1H), 7.75 (dd, *J* = 8.9, 2.7 Hz, 1H), 7.26 (d, *J* = 8.9 Hz, 1H), 3.85 (s, 3H), 2.07 (s, 3H); ^{13}C NMR (101 MHz, DMSO-D_6) δ 168.15, 166.23, 164.80, 158.46, 135.62, 133.07, 132.21, 130.39, 129.15, 124.09, 122.14, 113.67, 56.24, 21.25; FT-IR: $\tilde{\nu}$ = 1732, 1692, 1673, 1388, 1375, 1271 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{18}H_{15}O_6N_2$: 355.09246 found: 355.09277.



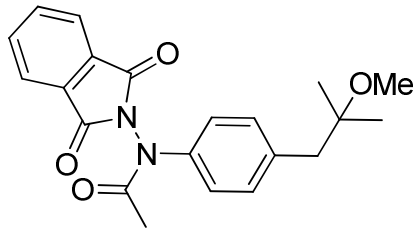
***N*-(1,3-Dioxoisoindolin-2-yl)-*N*-(4-(3-methyl-2,5-dioxo-2,5-dihydro-1*H*-pyrrol-1-yl)phenyl)acetamide (5l)**

Light yellow oil; (major rotamer) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (dd, *J* = 5.3, 3.0 Hz, 2H), 7.81 – 7.71 (m, 4H), 7.47 (d, *J* = 8.6 Hz, 2H), 6.50 (d, *J* = 1.4 Hz, 1H), 2.16 (d, *J* = 1.2 Hz, 3H), 2.14 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.26, 169.15, 168.38, 164.89, 146.18, 139.42, 134.84, 132.78, 130.01, 129.66, 127.73, 126.93, 124.11, 21.85, 11.31; FT-IR: $\tilde{\nu}$ = 1737, 1707, 1510, 1383, 1313, 1264 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{21}H_{16}O_5N_3$: 390.10845 found: 390.10940.



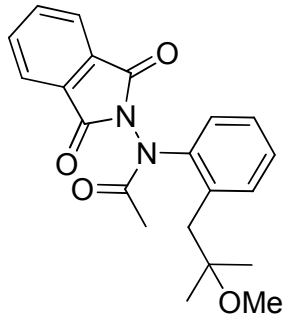
***N*-(4-(2,5-Dioxo-2,5-dihydro-1*H*-pyrrol-1-yl)phenyl)-*N*-(1,3-dioxoisoindolin-2-yl)acetamide (5k)**

Light yellow oil; (major rotamer) ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.88 (dd, *J* = 5.3, 3.1 Hz, 2H), 7.81 – 7.73 (m, 4H), 7.48 (d, *J* = 8.6 Hz, 2H), 6.87 (s, 2H), 2.14 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 169.10, 168.27, 164.91, 139.78, 134.84, 134.48, 132.43, 130.08, 129.75, 127.16, 124.12, 21.82; FT-IR: $\tilde{\nu}$ = 1737, 1711, 1510, 1371, 1309, 1262 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{20}H_{14}O_5N_3$: 376.09280 found: 376.09304.



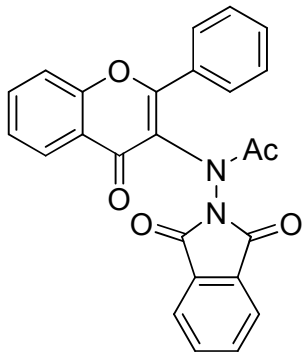
***N*-(1,3-Dioxoisoindolin-2-yl)-*N*-(4-(2-methoxy-2-methylpropyl)phenyl)acetamide (5j *para* isomer)**

Light yellow amorphous solid; (major rotamer) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.75 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.57 (d, *J* = 8.2 Hz, 2H), 7.26 (d, *J* = 8.2 Hz, 2H), 3.26 (s, 3H), 2.78 (s, 2H), 2.10 (s, 3H), 1.12 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.74, 165.02, 140.39, 138.77, 134.73, 131.96, 130.18, 128.29, 124.07, 75.10, 49.57, 45.84, 24.88, 21.78; FT-IR: $\tilde{\nu}$ = 2973, 1736, 1698, 1369, 1311, 1264, 1123, 1073 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{21}H_{23}O_4N_2$: 367.16523 found: 367.16657.



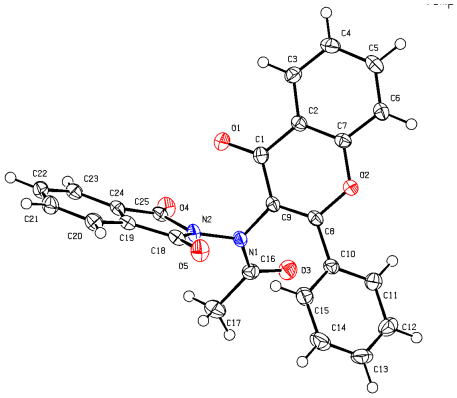
***N*-(1,3-Dioxoisoindolin-2-yl)-*N*-(2-(2-methoxy-2-methylpropyl)phenyl)acetamide (5j *ortho* isomer)**

Light yellow amorphous solid; (major rotamer) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 – 7.84 (m, 3H), 7.79-7.73 (m, 3H), 7.37 (td, *J* = 7.8, 1.3 Hz, 1H), 7.30 (td, *J* = 7.8, 1.3 Hz, 1H), 3.41 (s, 3H), 3.26 (d, *J* = 15.2 Hz, 1H), 3.14 (d, *J* = 15.2 Hz, 1H), 2.02 (s, 3H), 1.35 (s, 3H), 1.09 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.62, 166.10, 165.37, 139.67, 138.33, 134.87, 134.76, 132.01, 130.43, 130.36, 130.10, 129.68, 127.47, 124.11, 124.02, 76.05, 49.20, 37.39, 25.92, 24.74, 21.58; FT-IR: $\tilde{\nu}$ = 2947, 1739, 1698, 1367, 1294, 1264, 1073 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{21}H_{23}O_4N_2$: 367.16523 found: 367.16693.



_N_-(1,3-Dioxoisoindolin-2-yl)-_N_-(4-oxo-2-phenyl-4_H_-chromen-3-yl)acetamide (5s)

Light yellow crystalline soild; mp: 251-252 °C; (Mixture of rotamers in 1.5:1 ratio) # denotes major rotamer and * denotes minor rotamer; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.32 (d, *J* = 7.7 Hz, 1H), 8.19-8.08 (m, 2H), 7.99 (d, *J* = 7.2 Hz, 2H), 7.87 – 7.62 (m, 8H), 7.60 – 7.44 (m, 7H), #2.15 (s, 3H), *2.02 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, mixture of rotamers) δ 174.11, 173.65, 169.71, 168.76, 164.51, 164.17, 164.01, 163.71, 163.54, 162.59, 155.60, 135.08, 135.01, 134.55, 134.49, 134.11, 132.55, 131.86, 131.13, 130.42, 129.92, 129.57, 129.02, 128.64, 128.33, 126.69, 126.20, 126.01, 125.55, 124.62, 123.93, 123.87, 123.70, 123.40, 122.90, 119.49, 118.20, 118.11, 20.97, 20.73; FT-IR: $\tilde{\nu}$ = 1728, 1631, 1614, 1465, 1390, 1363, 1302, 1281, 1238, 1218, 112 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{25}H_{17}O_5N_2$: 425.11320 found: 425.11298.

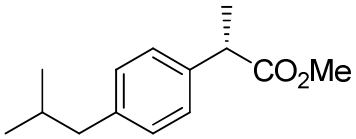


Crystal data and structure refinement for *N*-(1,3-dioxoisoindolin-2-yl)-*N*-(4-oxo-2-phenyl-4*H*-chromen-3-yl)acetamide. [Deposition number at Cambridge Crystallographic Data Centre: CCDC 889063]

Empirical formula	C25 H16 N2 O5	
Formula weight	424.40	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$ (14)	
Unit cell dimensions	a = 12.7844(6) Å	α= 90°.
	b = 12.8542(5) Å	β= 92.052(5)°.
	c = 11.9648(6) Å	γ = 90°.
Volume	1964.95(16) $Å^3$	
Z	4	
Density (calculated)	1.435 Mg/m^3	
Absorption coefficient	0.102 mm^{-1}	
F(000)	880	
Crystal size	0.40 x 0.20 x 0.20 mm^3	
Theta range for data collection	2.25 to 27.00°.	
Index ranges	-16<=h<=15, -16<=k<=16, -15<=l<=14	
Reflections collected	16750	
Independent reflections	4282 [R(int) = 0.0360]	
Completeness to theta = 27.00°	99.7 %	
Max. and min. transmission	0.9800 and 0.9605	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4282 / 0 / 290	
Goodness-of-fit on F^2	1.000	
Final R indices [I>2sigma(I)]	R1 = 0.0354, wR2 = 0.0631	
R indices (all data)	R1 = 0.0645, wR2 = 0.0657	
Largest diff. peak and hole	0.183 and -0.243 $e.Å^{-3}$	

Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

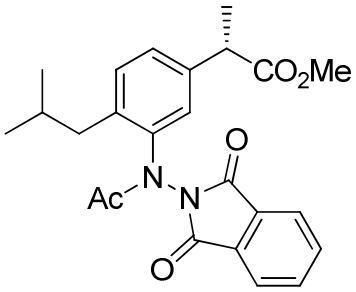
	x	y	z	U(eq)
C(1)	6621(1)	410(1)	7770(1)	21(1)
C(2)	5671(1)	985(1)	8024(1)	20(1)
C(3)	5298(1)	1020(1)	9107(1)	25(1)
C(4)	4364(1)	1499(1)	9313(1)	28(1)
C(5)	3780(1)	1938(1)	8431(1)	28(1)
C(6)	4130(1)	1926(1)	7363(1)	24(1)
C(7)	5084(1)	1454(1)	7176(1)	21(1)
C(8)	6343(1)	1042(1)	5849(1)	20(1)
C(9)	6898(1)	463(1)	6608(1)	18(1)
C(10)	6577(1)	1224(1)	4667(1)	22(1)
C(11)	5800(1)	1088(1)	3849(1)	26(1)
C(12)	6021(1)	1248(1)	2740(1)	33(1)
C(13)	6996(1)	1577(1)	2448(1)	37(1)
C(14)	7769(1)	1735(1)	3265(1)	38(1)
C(15)	7565(1)	1553(1)	4378(1)	31(1)
C(16)	7626(1)	-873(1)	5377(1)	23(1)
C(17)	8603(1)	-1377(1)	4983(1)	36(1)
C(18)	9342(1)	656(1)	7050(1)	22(1)
C(19)	10110(1)	309(1)	7921(1)	20(1)
C(20)	10981(1)	809(1)	8359(1)	26(1)
C(21)	11555(1)	307(1)	9201(1)	28(1)
C(22)	11269(1)	-669(1)	9573(1)	26(1)
C(23)	10406(1)	-1180(1)	9107(1)	22(1)
C(24)	9836(1)	-672(1)	8282(1)	20(1)
C(25)	8889(1)	-1012(1)	7639(1)	20(1)
N(1)	7753(1)	-143(1)	6230(1)	20(1)
N(2)	8657(1)	-181(1)	6897(1)	21(1)
O(1)	7129(1)	-82(1)	8483(1)	32(1)
O(2)	5432(1)	1505(1)	6106(1)	23(1)
O(3)	6770(1)	-1043(1)	4965(1)	30(1)
O(4)	8414(1)	-1819(1)	7650(1)	27(1)
O(5)	9267(1)	1473(1)	6559(1)	31(1)



(*S*)-Methyl 2-(4-isobutylphenyl)propanoate[11]

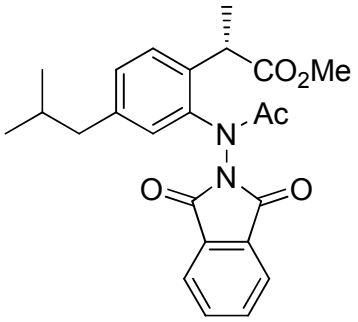
This compound has been synthesized by a literature known procedure and the spectroscopic data were identical to the reported one. [11]

Colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.20 (d, *J* = 7.9 Hz, 2H), 7.10 (d, *J* = 7.9 Hz, 2H), 3.71 (q, *J* = 7.2 Hz, 1H), 3.66 (s, 3H), 2.45 (d, *J* = 7.2 Hz, 2H), 1.86 (m, 1H), 1.50 (d, *J* = 7.2 Hz, 3H), 0.91 (d, *J* = 6.6, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.32, 140.67, 137.89, 129.47, 127.26, 52.08, 45.19, 45.17, 30.30, 22.53, 18.75.



(*S*)-Methyl 2-(3-(*N*-(1,3-dioxoisoindolin-2-yl)acetamido)-4-isobutylphenyl)propanoate (5t major isomer)

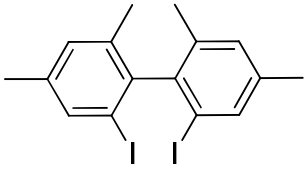
Light yellow oil; (Mixture of rotamers in 1:1 ratio) ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.92 – 7.85 (m, 2H), 7.84 – 7.79 (m, 1H), 7.79 – 7.73 (m, 2H), 7.34 – 7.27 (m, 2H), 3.79 – 3.72 (m, 1H), 3.69 (d, *J* = 8.3 Hz, 3H), 2.91 – 2.80 (m, 2H), 2.11 – 2.02 (m, 1H), 2.00 (d, *J* = 0.7 Hz, 3H), 1.52 (dd, *J* = 11.8, 7.2 Hz, 3H), 1.06 (d, *J* = 6.7 Hz, 3H), 0.90 (d, *J* = 6.7 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 174.73, 169.27, 166.15, 164.95, 139.73, 139.67, 139.46, 139.32, 139.26, 134.85, 134.68, 131.09, 131.02, 130.45, 130.34, 130.17, 128.84, 128.56, 124.13, 123.98, 52.27, 52.25, 44.82, 38.83, 38.81, 28.52, 23.07, 21.97, 21.52, 18.71, 18.56; FT-IR: $\tilde{\nu}$ = 2955, 1736, 1696, 13661290, 1261, 1202, 1163 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{24}H_{27}O_5N_2$: 423.19145 found: 423.19137; $[\alpha]_D^{RT}$ = 32.2 (c = 1.0 in CH_2Cl_2).



(*S*)-Methyl 2-(2-(*N*-(1,3-dioxoisoindolin-2-yl)acetamido)-4-isobutylphenyl)propanoate (5t minor isomer)

Color-less oil; (Mixture of rotamers in 2.2:1 ratio, major rotamer); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.91 – 7.87 (m, 2H), 7.79 – 7.74 (m, 3H), 7.71 (d, *J* = 1.6 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 1H), 7.21 (dd, *J* = 8.0, 1.6 Hz, 1H), 4.98 (q, *J* = 7.0 Hz, 1H), 3.68 (s, 3H), 2.55 – 2.46 (m, 2H), 2.08 (s, 3H), 1.93 – 1.83 (m, 1H), 1.43 (d, *J* = 7.0 Hz, 3H), 0.92 (d, *J* = 6.6 Hz, 3H), 0.90 (d, *J* = 6.6 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$, mixture of rotamers) δ 175.25, 169.84, 165.84, 165.40, 142.71, 142.42, 138.15, 137.85, 137.31, 134.92, 134.82, 134.79, 134.65, 131.76, 131.62, 131.51, 131.07, 130.25, 130.09, 128.54, 128.49, 124.19, 124.11, 124.00, 123.96, 52.15, 44.75, 38.93, 38.23, 30.17, 24.66, 22.54, 22.34, 22.31, 21.68, 21.43, 19.86, 18.87; FT-IR: $\tilde{\nu}$ = 2956, 1734, 1697, 1377, 1270, 1168, 1073 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_{24}H_{27}O_5N_2$: 423.19145 found: 423.19130; $[\alpha]_D^{RT}$ = 68.7 (c = 1.0 in CH_2Cl_2).

Synthesis of 2,2'-diiodo-4,4',6,6'-tetramethylbiphenyl[12]

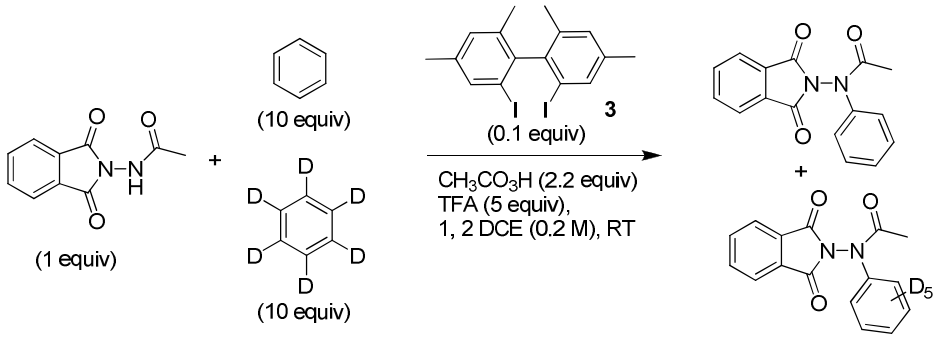


This compound has been synthesized by a literature known procedure and the spectroscopic data were identical to the reported one.

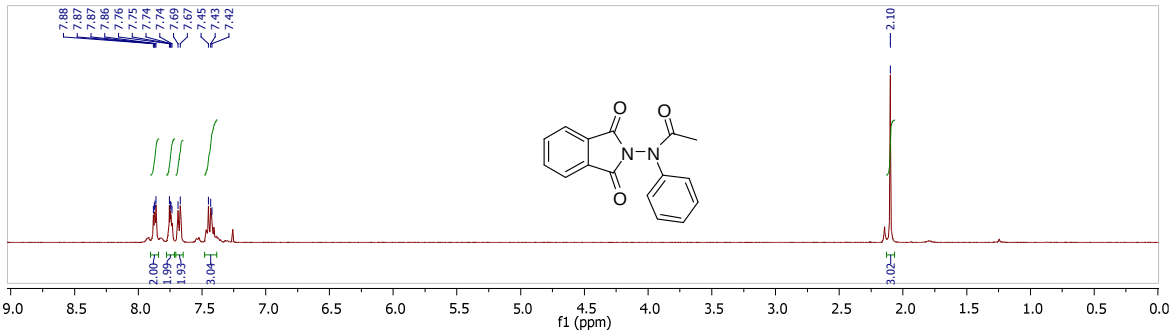
Kinetic Isotope Effect Study:

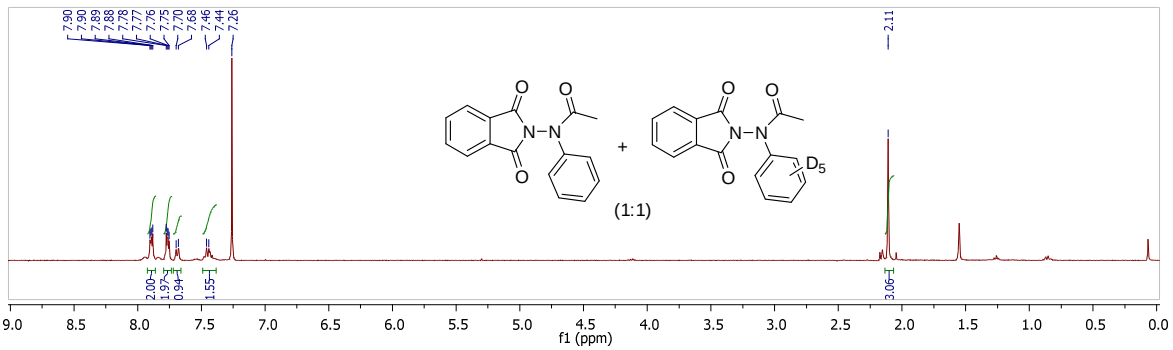
Compound *N*-acetylaminophthalimide (0.2 mmol) was dissolved in a 4 mL screw-capped vial with 1 mL of 1,2 dichloroethane. Then benzene (2 mmol), [$^{2}H_6$]-benzene (2 mmol) followed

by organocatalyst **3** (0.02 mmol), peracetic acid (39%, 74.6 µL, 0.44 mmol) and trifluoroacetic acid (78.6 µL, 1 mmol) were added at room temperature. The reaction mixture was stirred at this temperature for 40 min. It was quenched with saturated solution of sodium thiosulphate and extracted with dichloromethane, washed with water, brine, dried over anhydrous Na_2SO_4. The organic extracts were filtered and concentrated under reduced pressure.

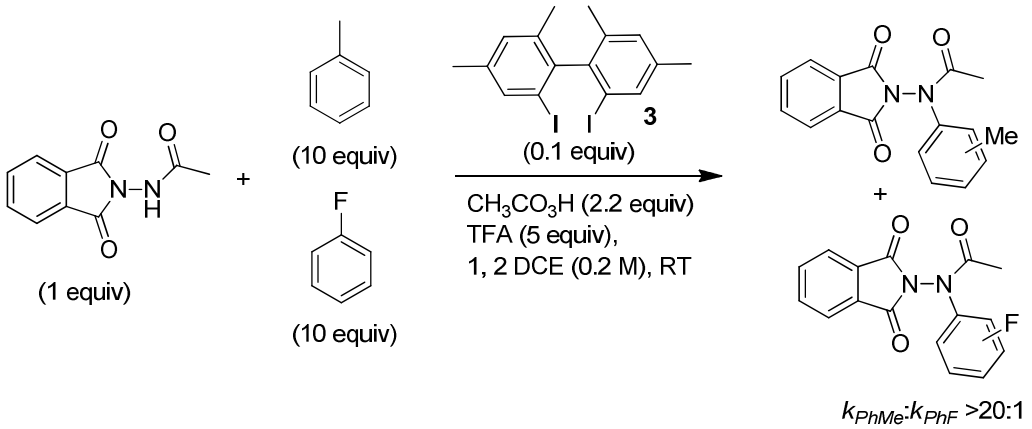


Purification by column chromatography on silica gel (15% EtOAc in Pet. Ether) afforded the pure product, which was submitted for ^{1}H NMR measurement in $CDCl_3$ solvent. The Kinetic Isotope Effect was measured from the ^{1}H NMR spectra given below.





Competition reactions for the organocatalytic hydrazination of electron rich and electron deficient arene:



Compound *N*-acetylaminophthalimide (0.2 mmol) was dissolved in a 4 mL screw-capped vial with 1 mL of 1,2 dichloroethane. Then toluene (2 mmol), fluorobenzene (2 mmol) followed by organocatalyst **3** (0.02 mmol), peracetic acid (39%, 74.6 µL, 0.44 mmol) and trifluoroacetic acid (78.6 µL, 1 mmol) were added at room temperature. The reaction mixture was stirred at this temperature for 40 min. It was quenched with saturated solution of sodium thiosulphate and extracted with dichloromethane, washed with water, brine, dried over anhydrous Na_2SO_4. The organic extracts were filtered and concentrated under reduced pressure to afford the mixture of products. The mixture of products was submitted for HPLC-LCMS and ^{1}H NMR measurement in $CDCl_3$ solvent. The amount of product formed was analysed by the ^{1}H NMR spectra of the mixture.

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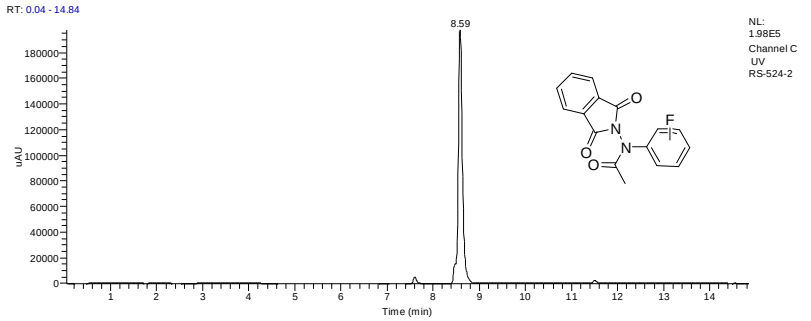

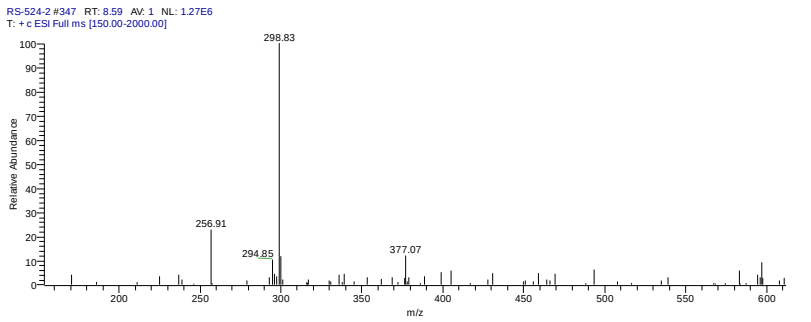

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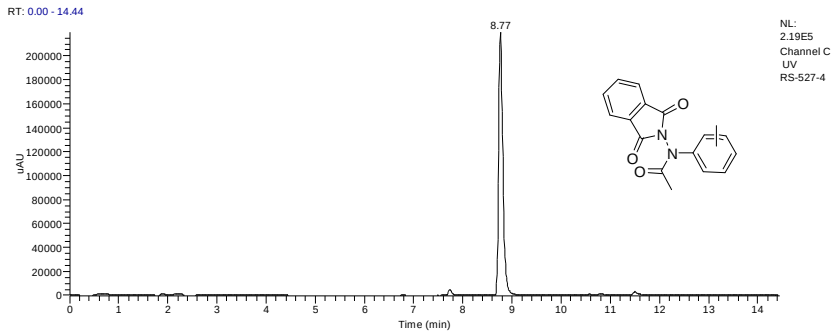
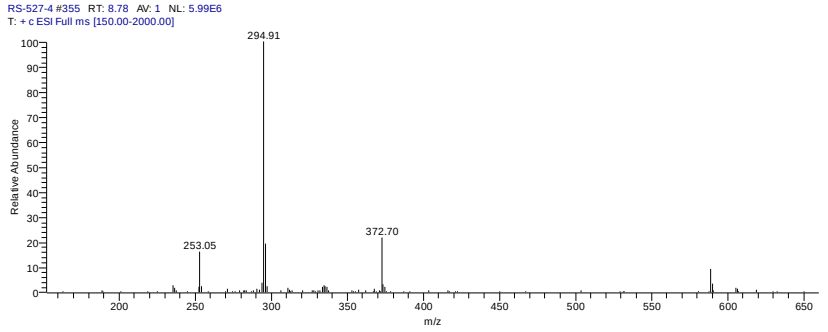
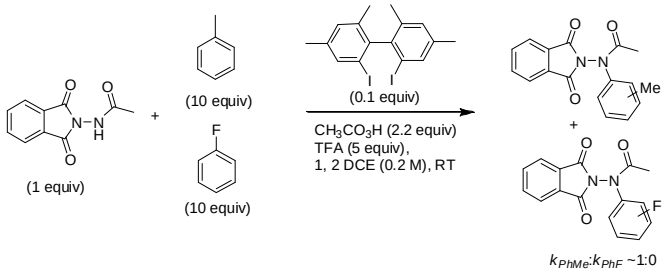
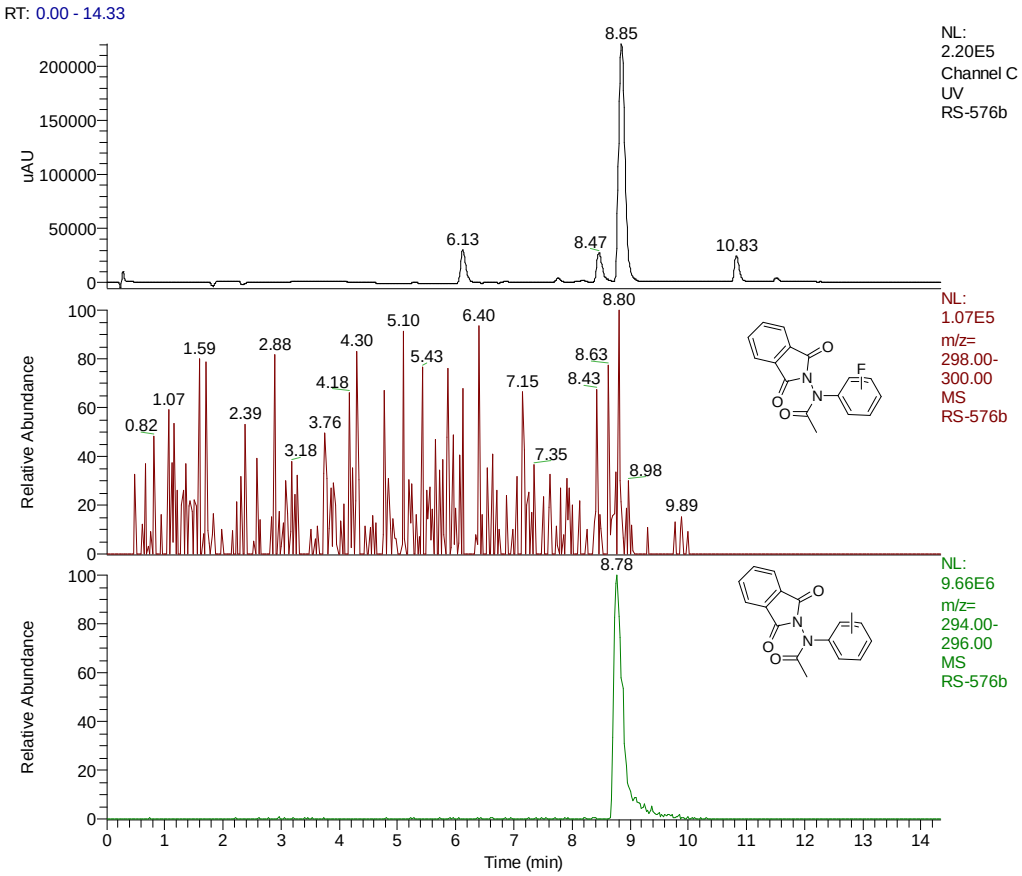
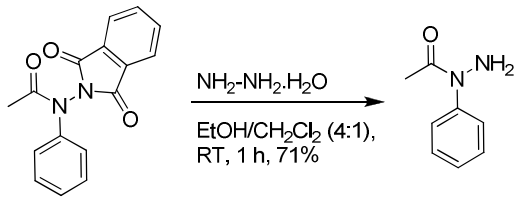





(1 equiv)
(10 equiv)
(10 equiv)
(0.1 equiv)
CH3CO3H (2.2 equiv)
TFA (5 equiv),
1, 2 DCE (0.2 M), RT
Me
F
kPhMe:kPhF ~1:0


RT: 0.00 - 14.33
NL:
2.20E5
Channel C
UV
RS-576b
uAU
8.85
6.13
8.47
10.83
NL:
1.07E5
m/z=
298.00-
300.00
MS
RS-576b
Relative Abundance
8.80
5.10
6.40
1.59
2.88
4.30
5.43
8.63
4.18
7.15
8.43
1.07
2.39
0.82
3.76
3.18
7.35
8.98
9.89
NL:
9.66E6
m/z=
294.00-
296.00
MS
RS-576b
8.78
Time (min)

Phthalimide group deprotection:



Compound *N*-(1,3-dioxoisoindolin-2-yl)-*N*-phenylacetamide (48 mg, 0.17 mmol, 1 equiv) was taken in EtOH/CH_2Cl_2 (1.2 mL/ 0.3 mL) and hydrazine monohydrate (50 wt %, 105 µL, 1.68 mmol, 10 equiv) was added. The resulting mixture was stirred for 1h at room temperature for the completion of the reaction. White precipitate was observed after completion of the reaction. The reaction mixture was filtered and the filtrate was concentrated under reduced pressure. The desired product *N*-phenylacetohydrazide was purified by flash silica gel column chromatography with 70% EtOAc in petroleum ether in 71% yield as white amorphous solid. The analytical data were identical to the known literature data.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 (t, *J* = 7.7 Hz, 2H), 7.31 (dd, *J* = 19.7, 7.3 Hz, 2H), 4.81 (s, 2H), 2.00 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 169.44, 142.99, 129.48, 127.99, 126.72, 21.84; FT-IR: $\tilde{\nu}$ = 3324, 3217, 3059, 1630, 1596, 1493, 1458, 1399, 1255 cm^{-1}; HRMS: calc. for $[M+H]^+$ $C_8H_{11}ON_2$: 151.08659 found: 151.08637.

References:

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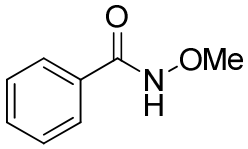
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H
OMe

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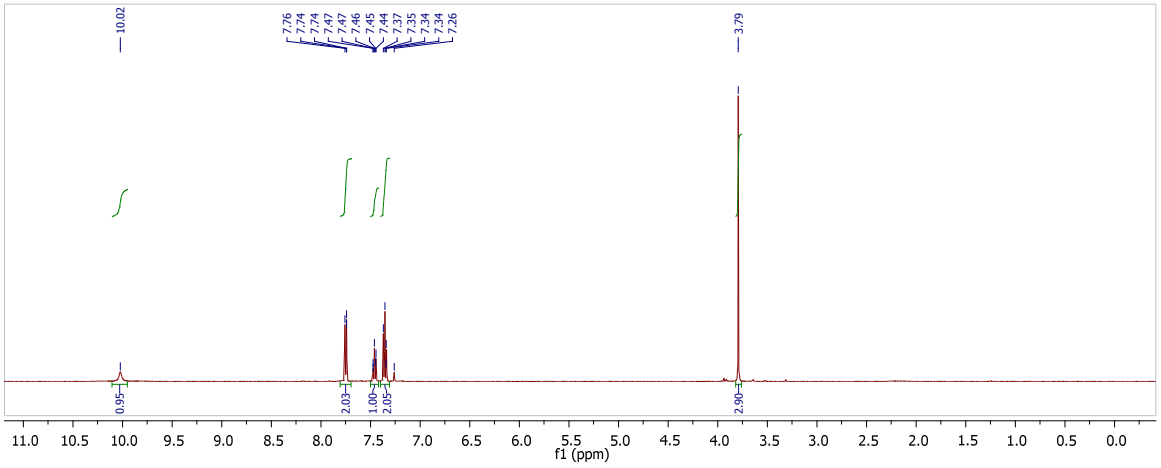
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7.45
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7.34
7.26
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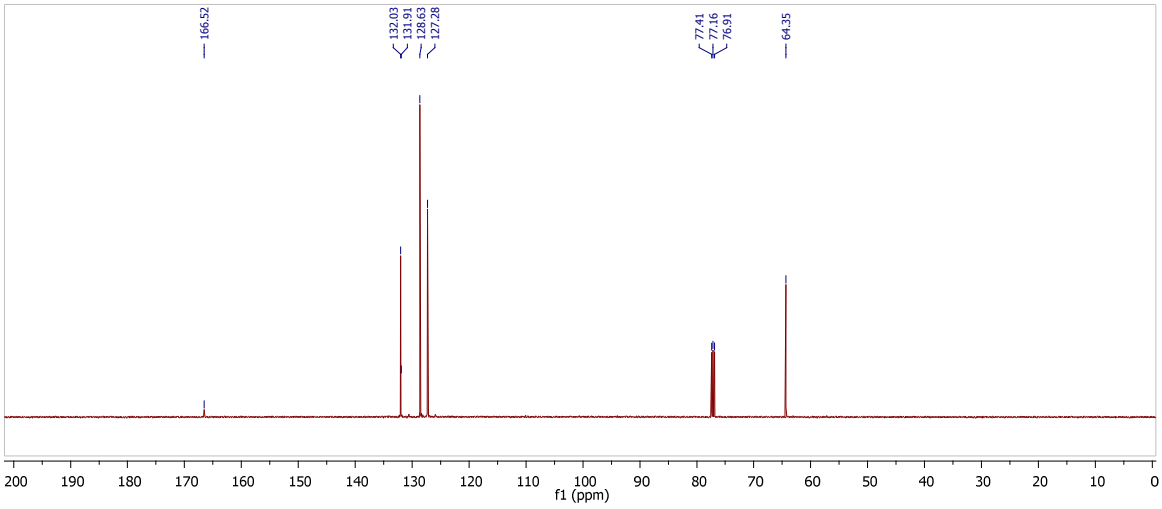
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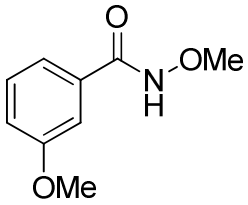
f1 (ppm)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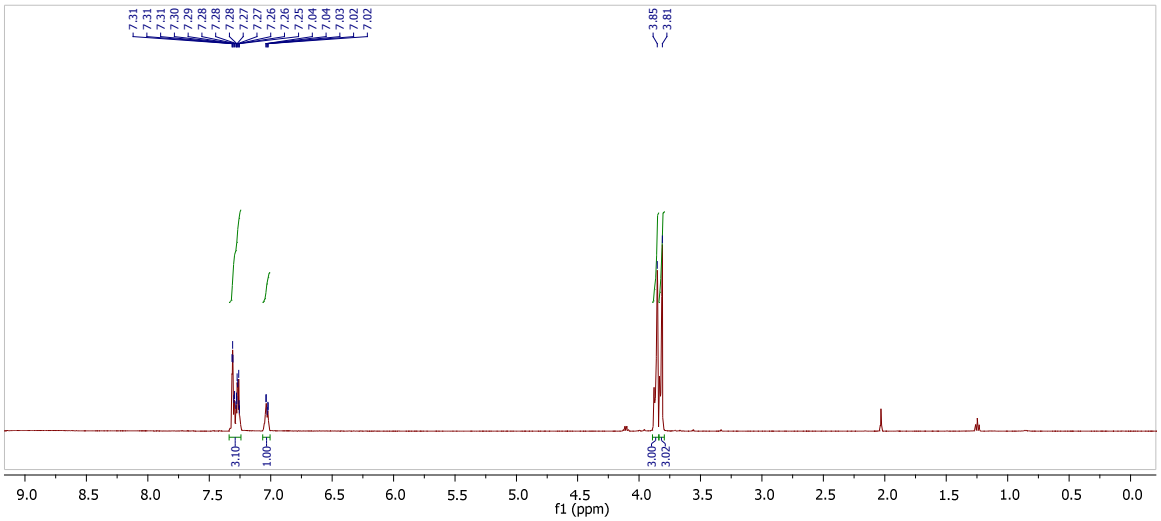
N
H
OMe
OMe

1o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7.27
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7.25
7.04
7.04
7.03
7.02
7.02
3.85
3.81
3.10
1.00
3.00
3.02
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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2.5
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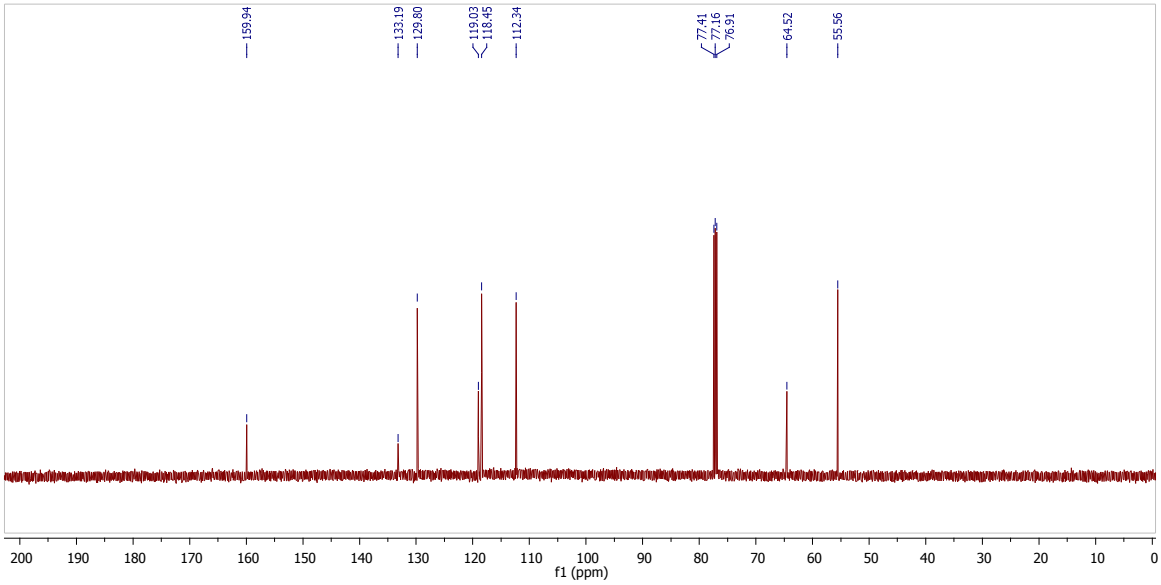
f1 (ppm)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77.16
76.91
64.52
55.56
200
190
180
170
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120
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100
90
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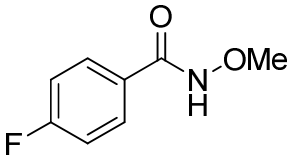
70
60
50
40
30
20
10
0
f1 (ppm)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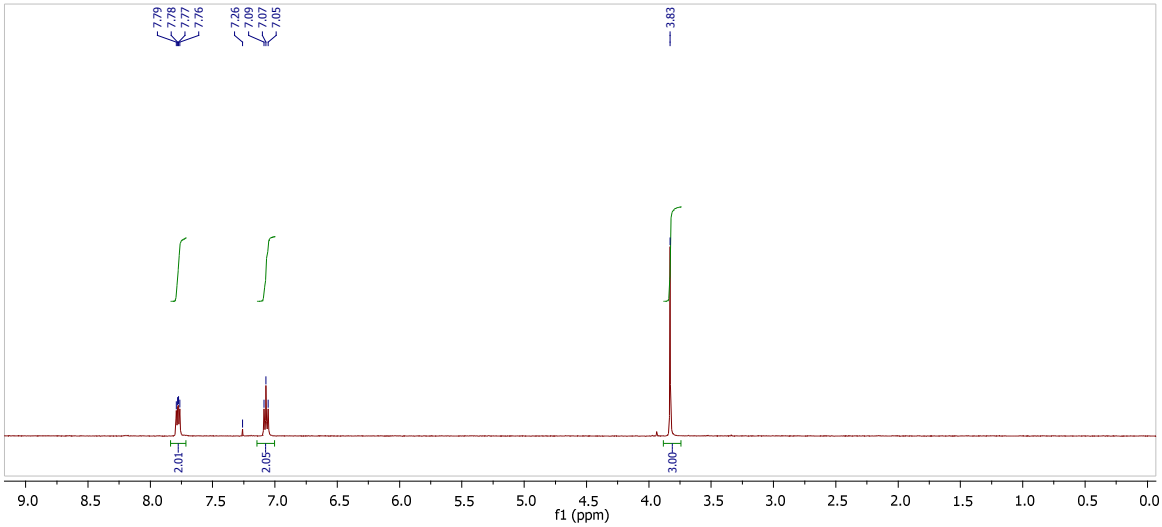
N
H
OMe
F

1n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7.76
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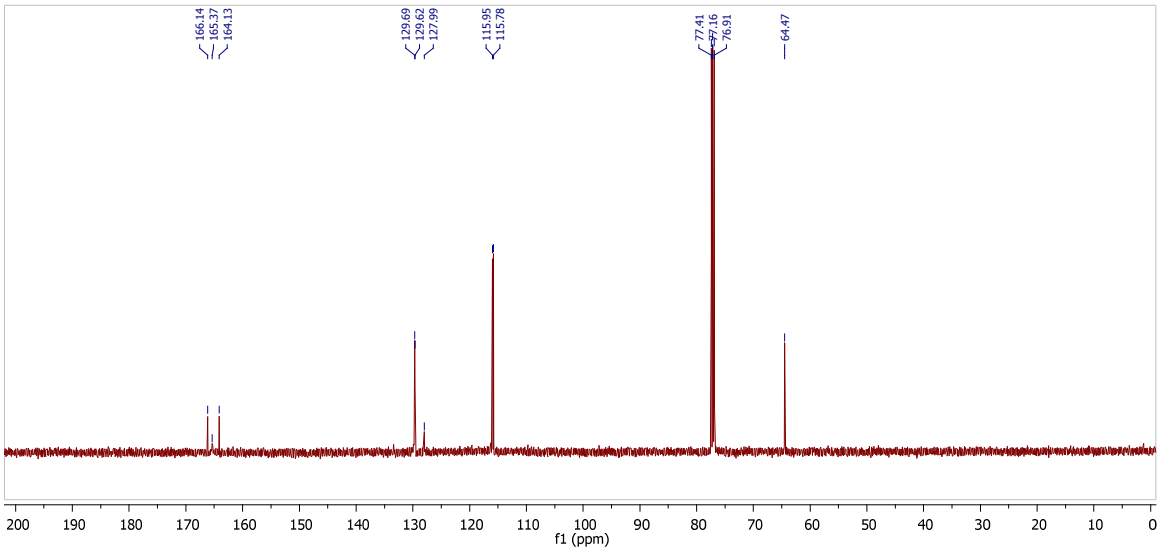
7.07
7.05
3.83
2.01
2.05
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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77.41
77.16
76.91
64.47
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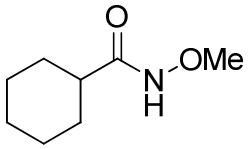
f1 (ppm)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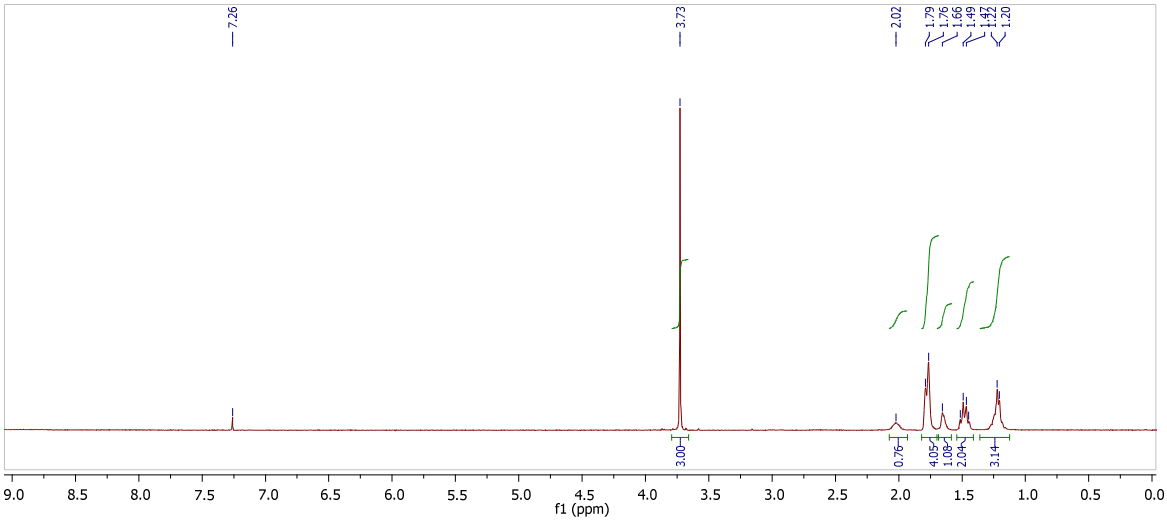
H
OMe

1p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1.79
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1.47
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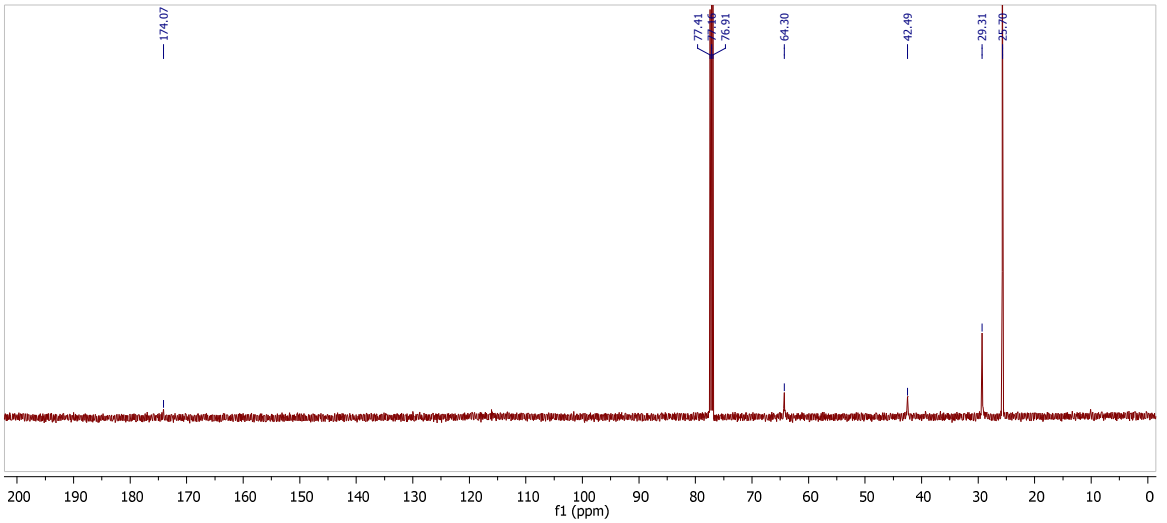
1.20
3.00
0.76
4.05
1.08
2.04
3.14
f1 (ppm)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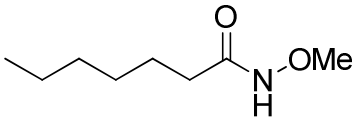
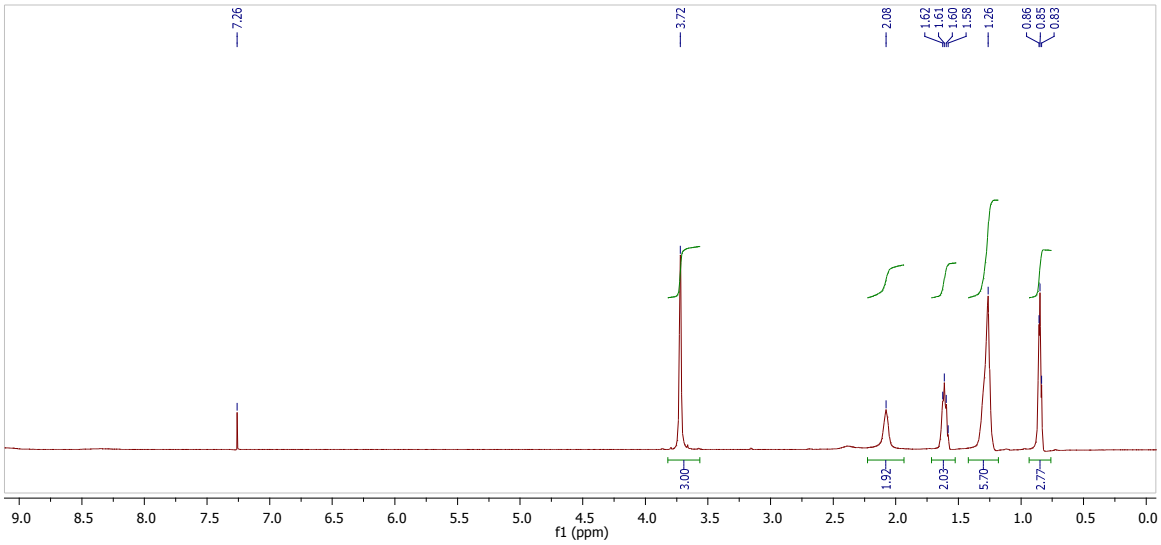
76.91
64.30
42.49
29.31
25.70
f1 (ppm)


O
OMe
N
H

1q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1.61
1.60
1.58
1.26
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0.85
0.83
3.00
1.92
2.03
5.70
2.77
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
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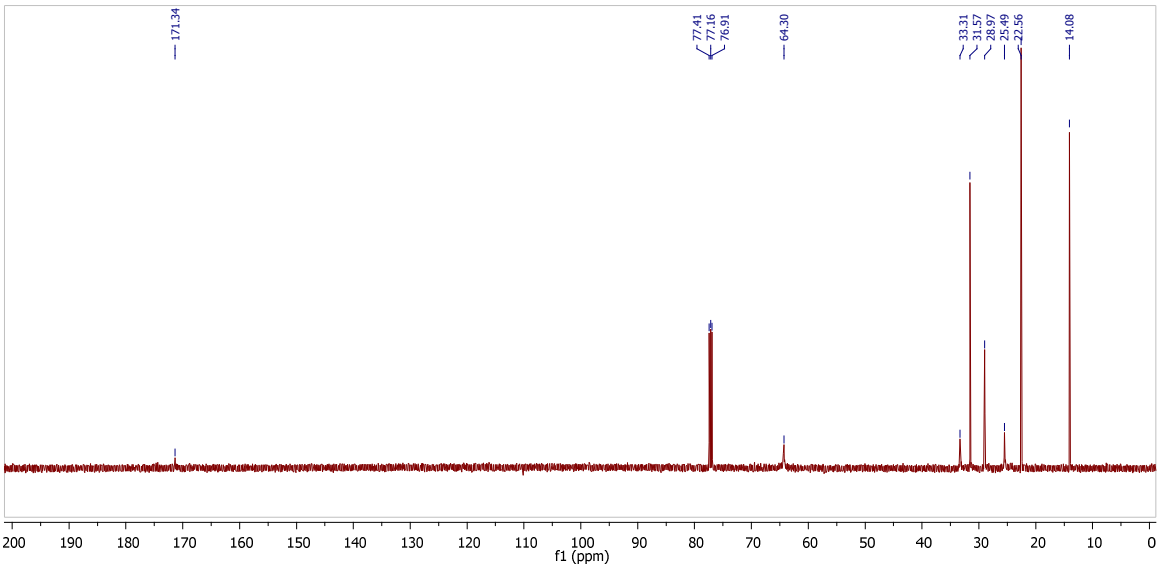
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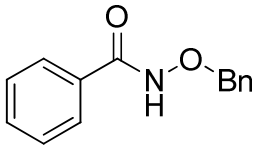
77.41
77.16
76.91
64.30
33.31
31.57
28.97
25.49
22.56
14.08
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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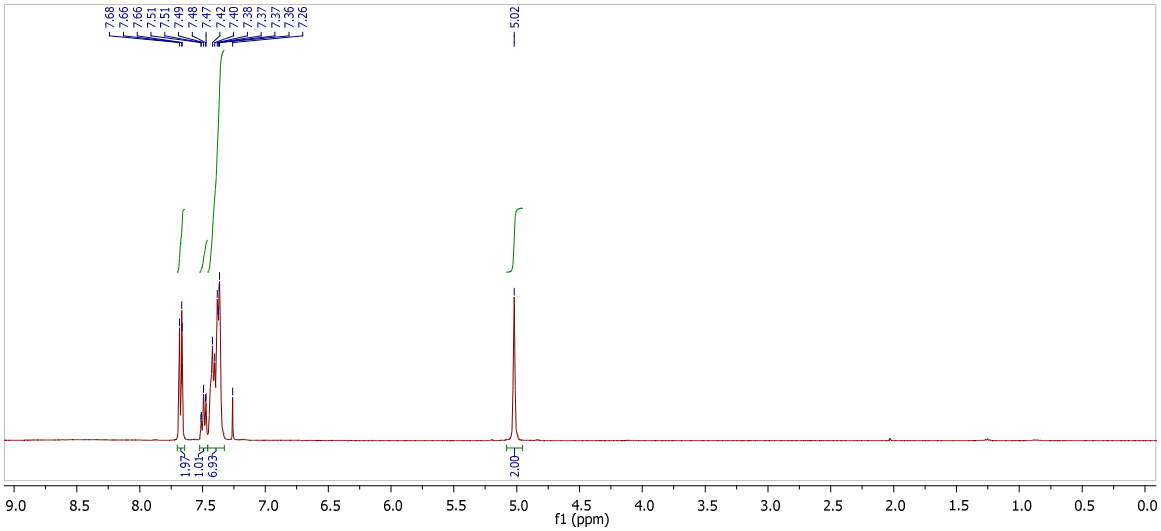
N
H
O
Bn

1m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7.37
7.36
7.26
5.02
1.97
1.01
6.93
2.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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2.5
2.0
1.5
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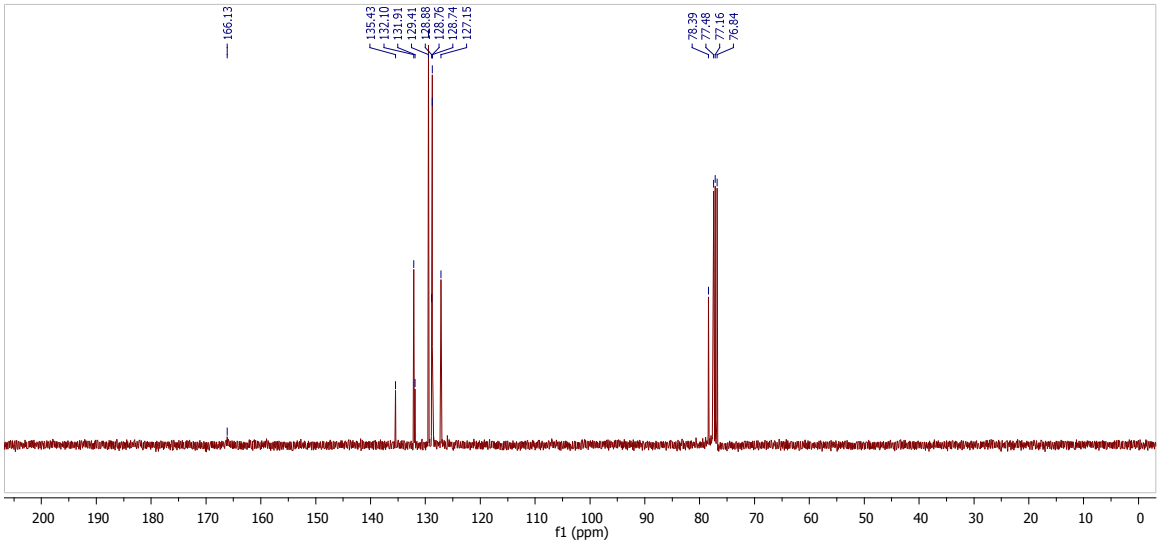
f1 (ppm)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128.74
127.15
78.39
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77.16
76.84
200
190
180
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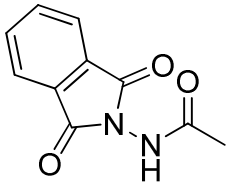
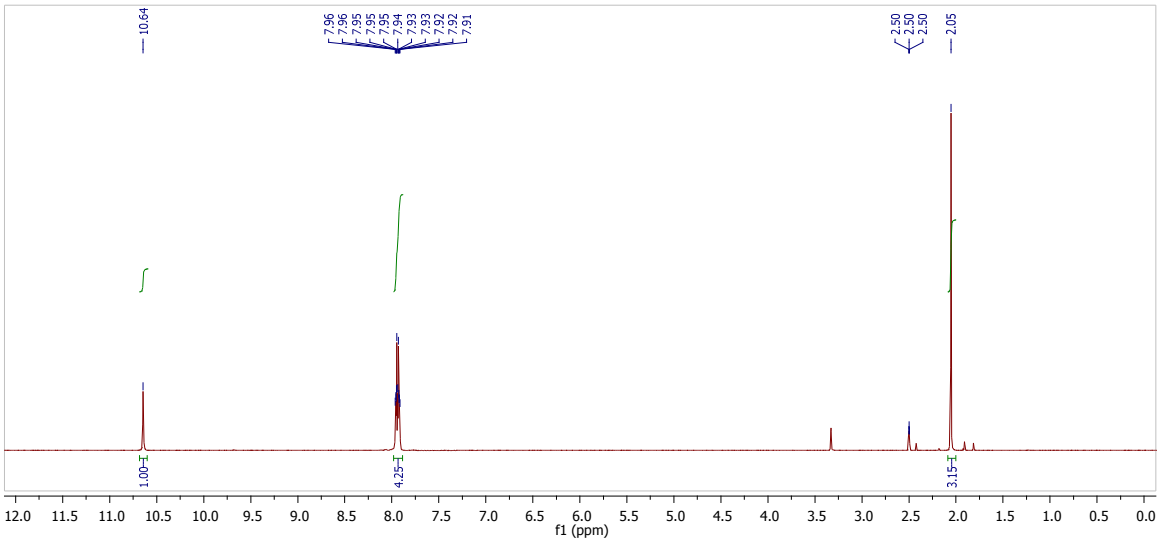
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


O
O
N
N
H
O

4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7.94
7.93
7.93
7.92
7.92
7.91
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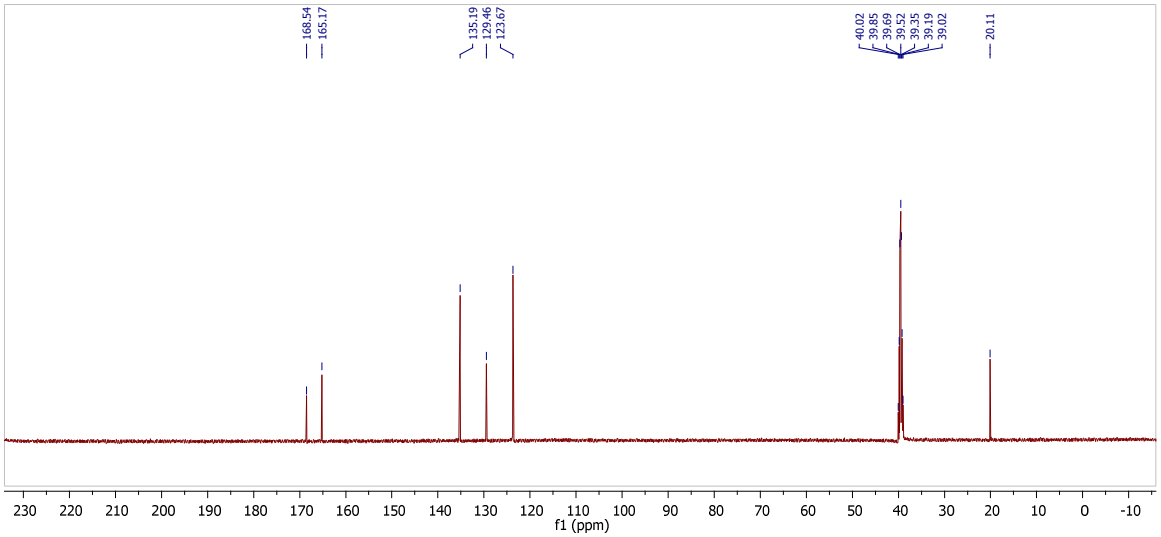
2.50
2.50
2.05
1.00
4.25
3.15
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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39.69
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39.19
39.02
20.11
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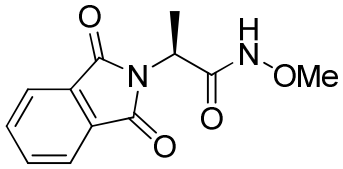
f1 (ppm)



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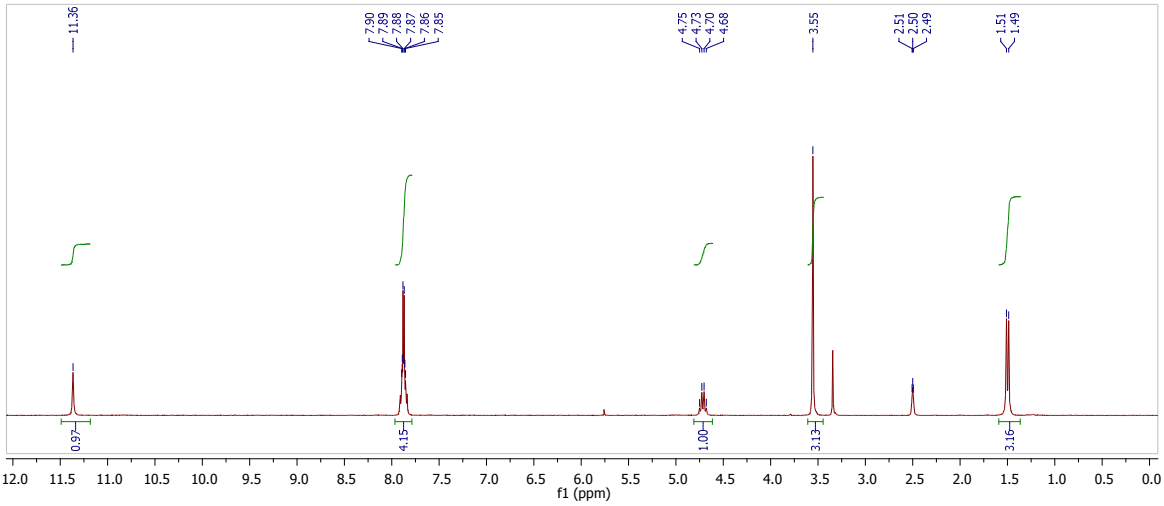
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7.85
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4.70
4.68
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2.49
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1.49
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4.15
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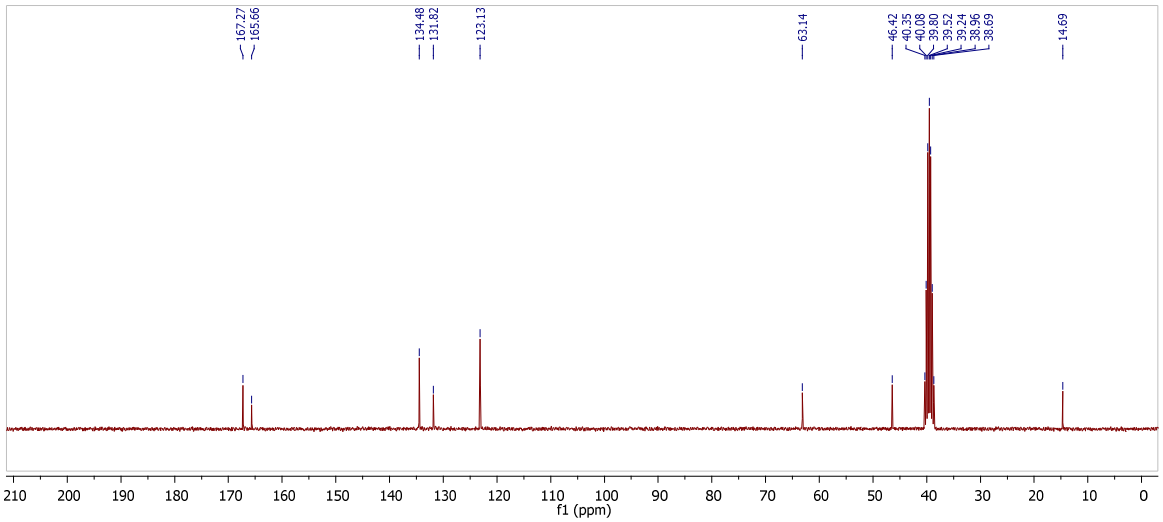
3.13
3.16
f1 (ppm)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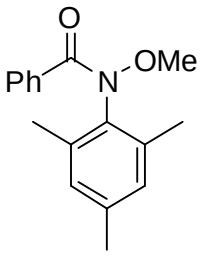
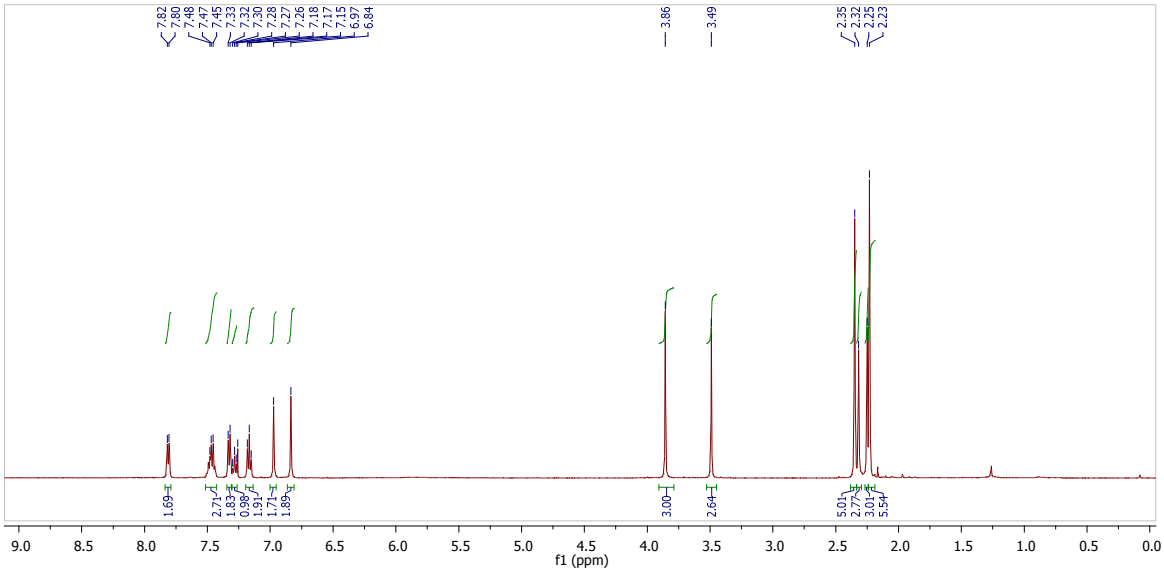

O
Ph
N
OMe

2k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7.26
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7.17
7.15
6.97
6.84
3.86
3.49
2.35
2.32
2.25
2.23
1.69
2.71
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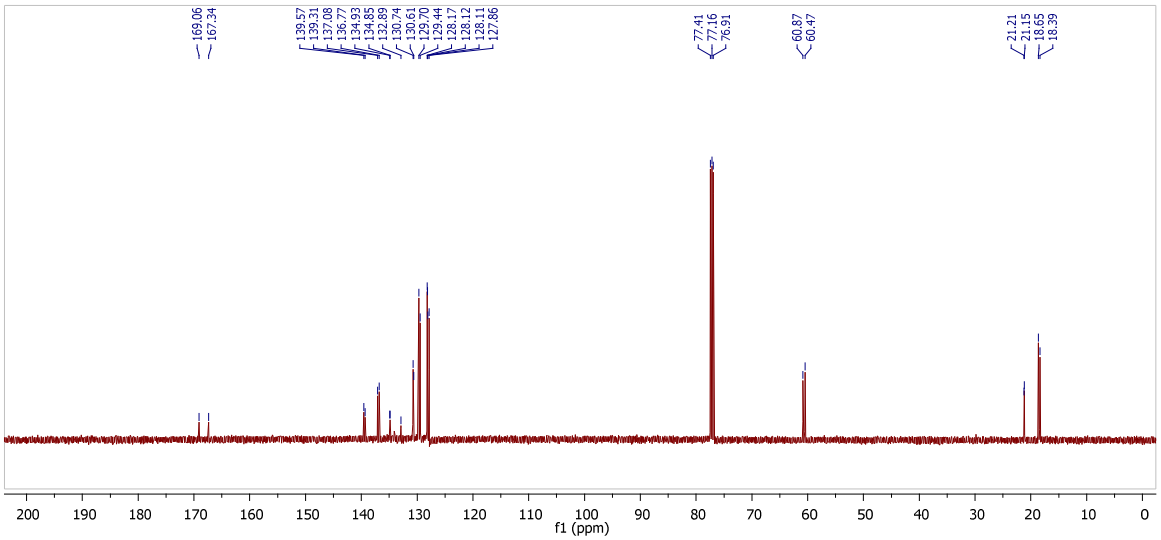
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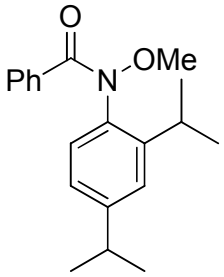
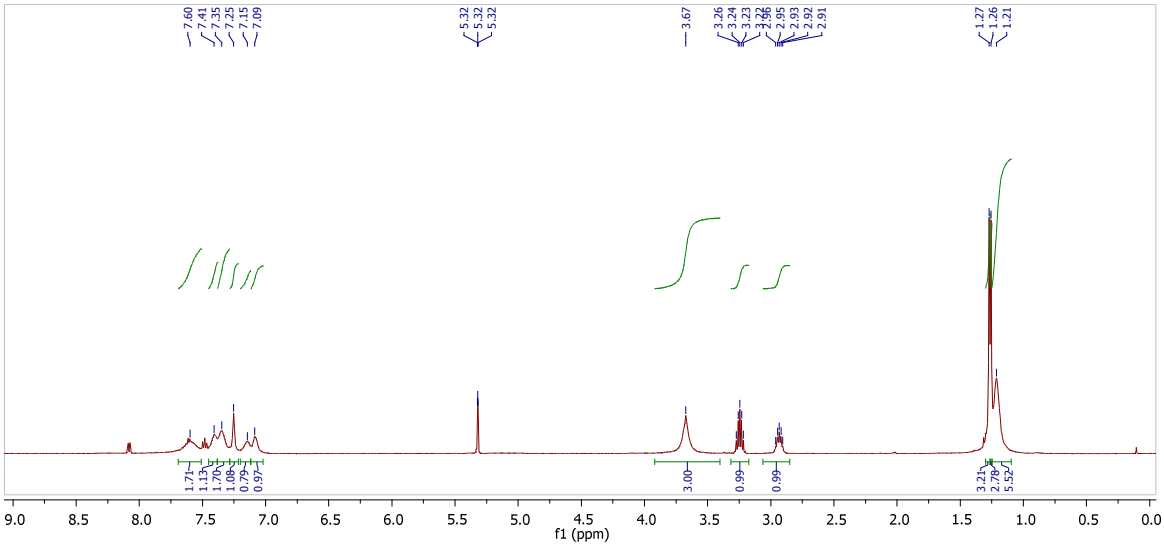
18.39
f1 (ppm)


O
Ph
N
OMe

2i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2.92
2.91
1.27
1.26
1.21
1.71
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3.21
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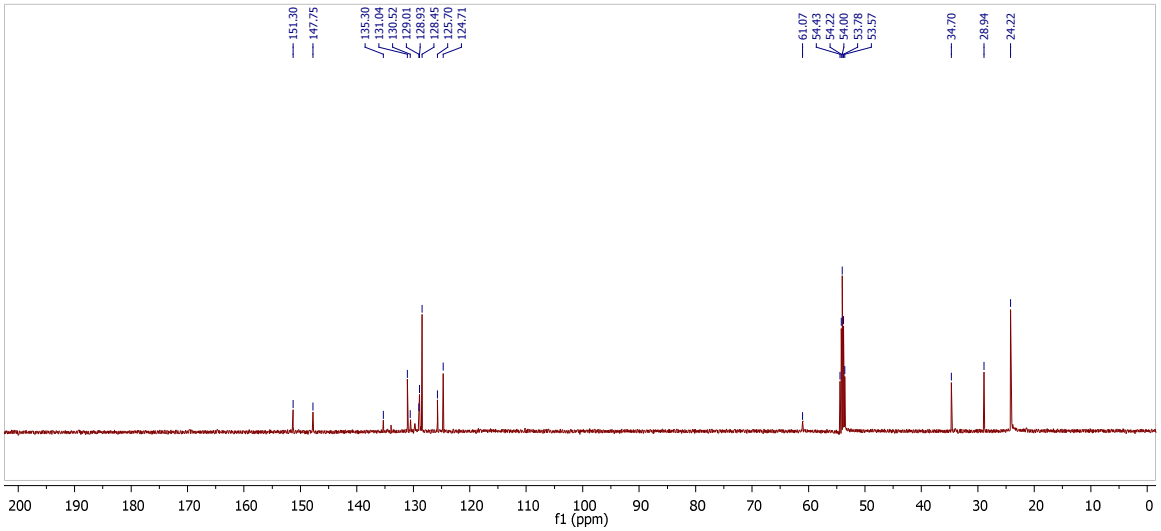
5.52
f1 (ppm)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129.01
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128.45
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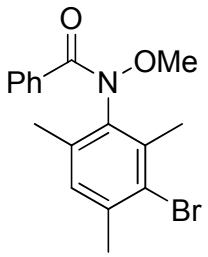
24.22
f1 (ppm)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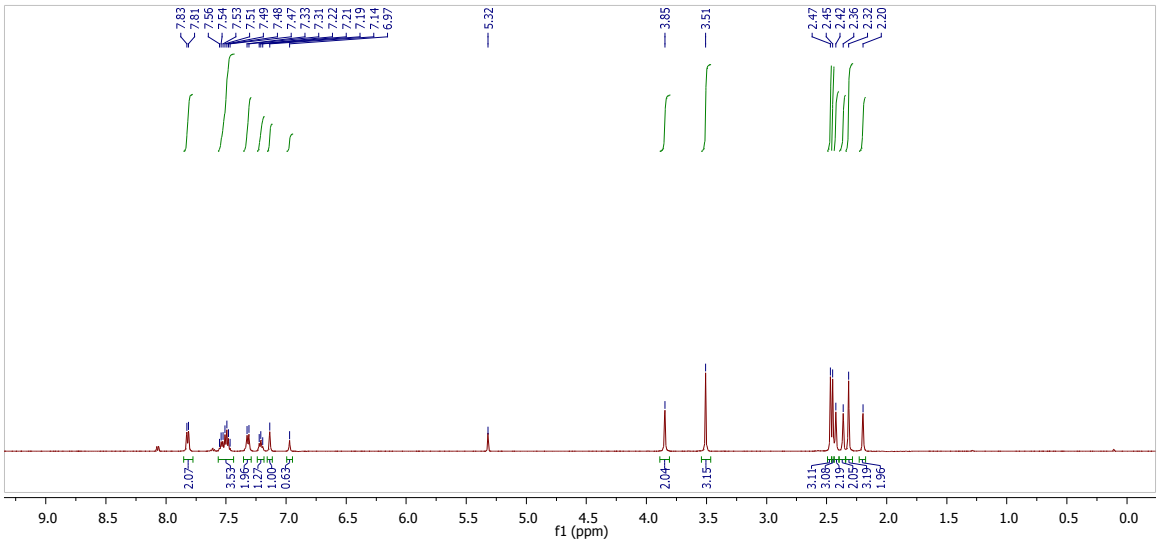
Br

2l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7.19
7.14
6.97
5.32
3.85
3.51
2.47
2.45
2.42
2.36
2.32
2.20
2.07
3.53
1.96
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2.19
2.05
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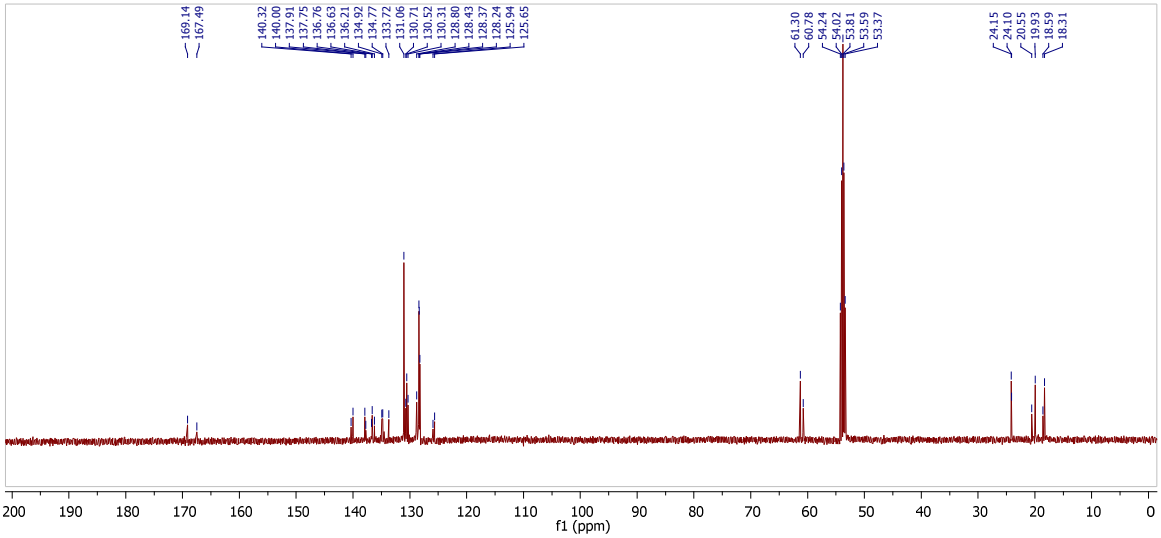
f1 (ppm)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131.06
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130.52
130.31
128.80
128.43
128.37
128.24
125.94
125.65
61.30
60.78
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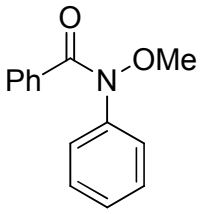
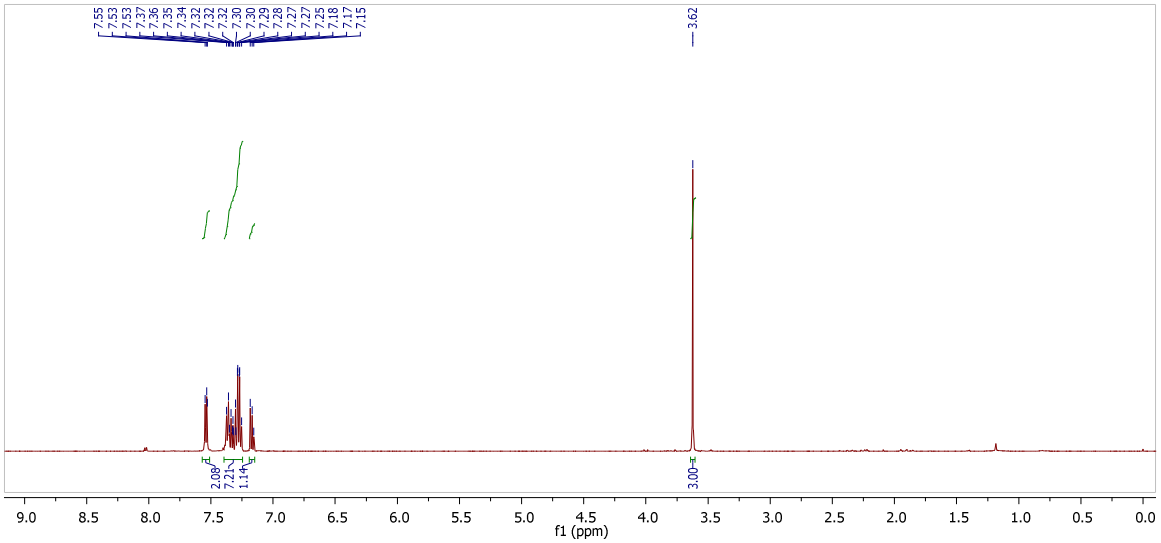
18.31
f1 (ppm)


O
Ph
N
OMe

2b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7.25
7.18
7.17
7.15
3.62
2.08
7.21
1.14
3.00
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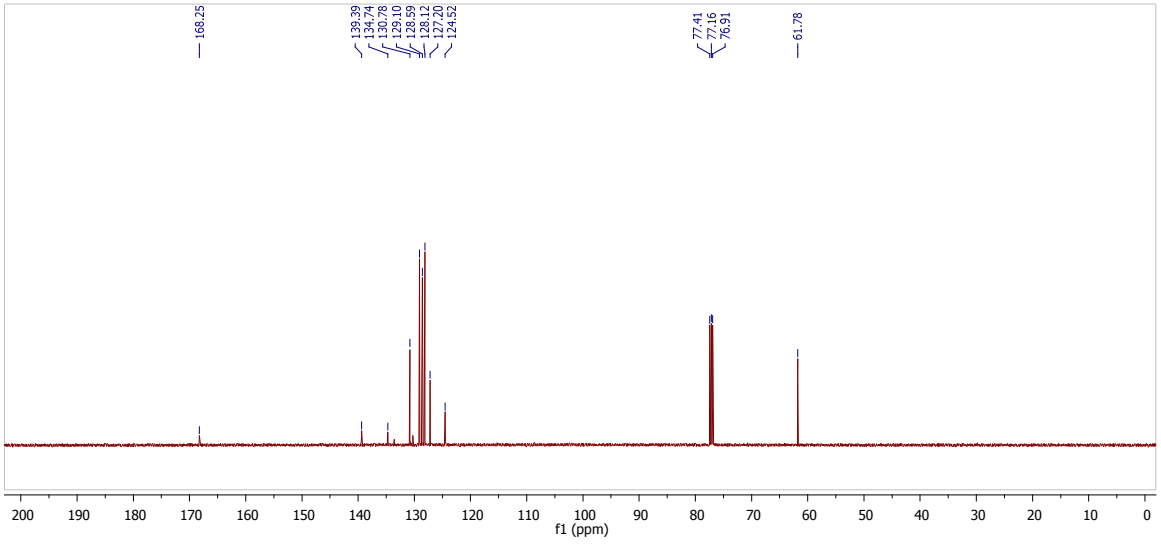
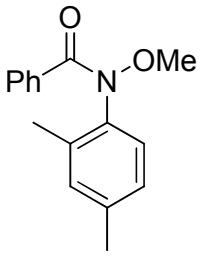
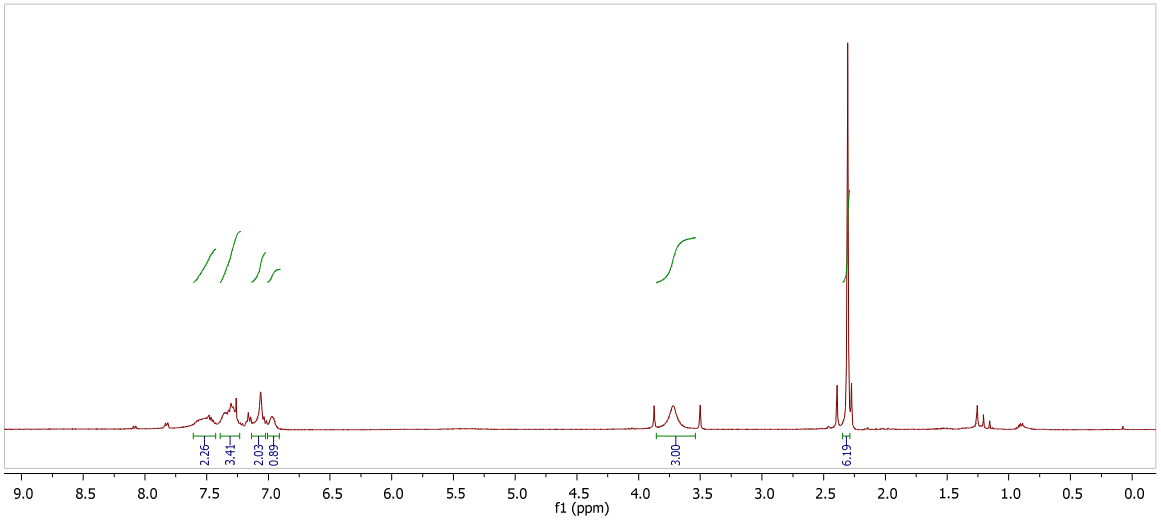
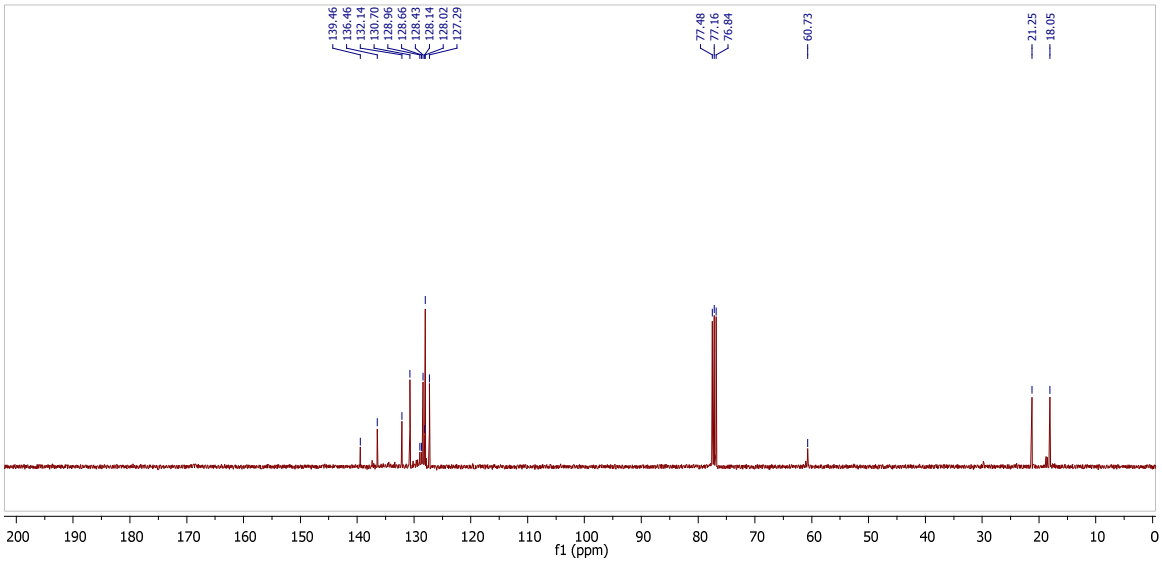
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


168.25
139.39
134.74
130.78
129.10
128.59
128.12
127.20
124.52
77.41
77.16
76.91
61.78
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


O
Ph
N
OMe

2j


2.26
3.41
2.03
0.89
3.00
6.19
f1 (ppm)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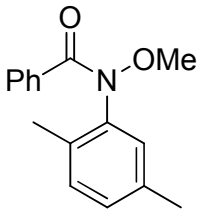
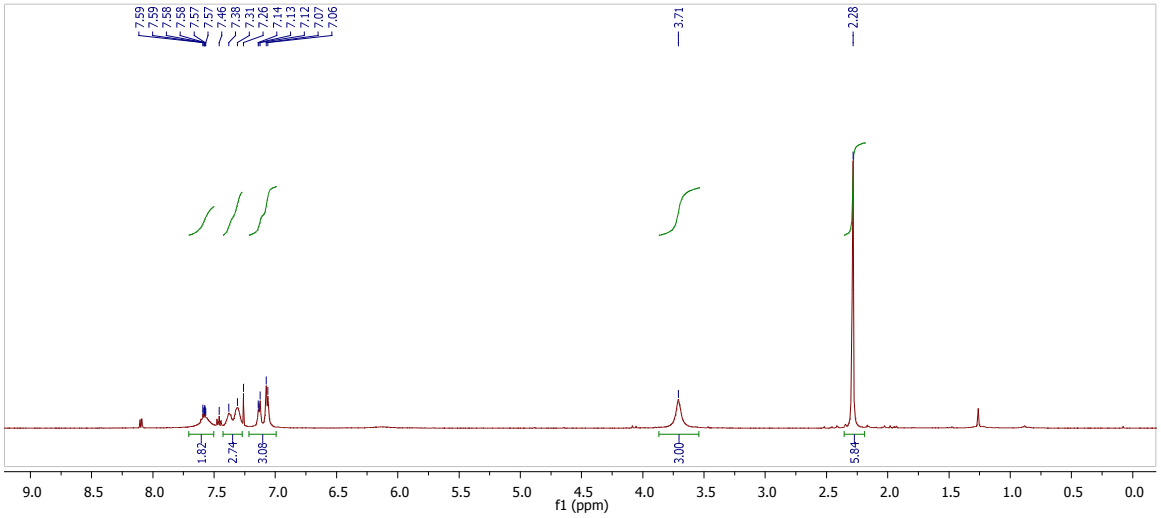
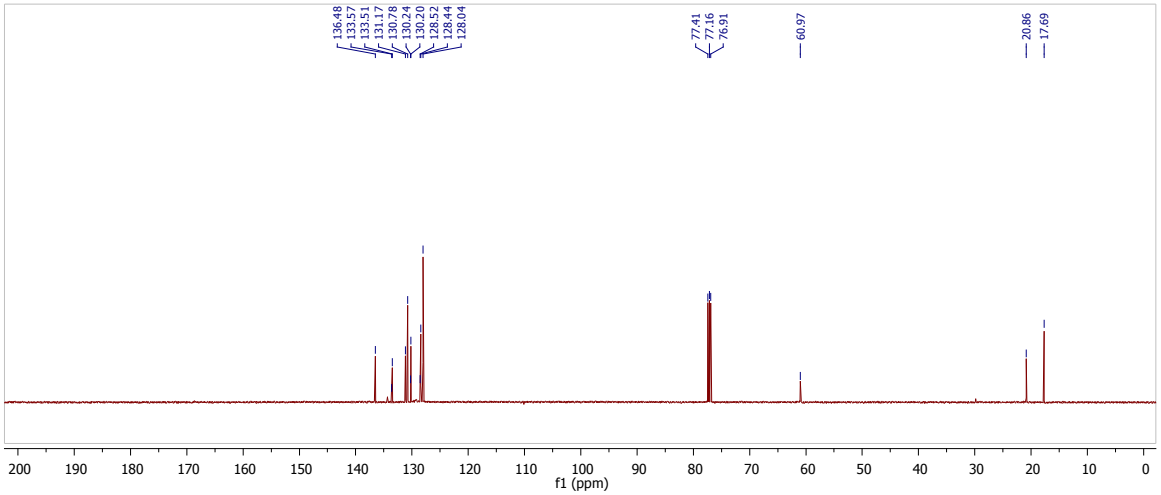
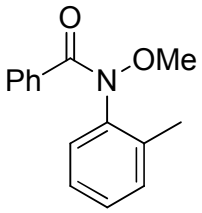
136.46
132.14
130.70
128.96
128.66
128.43
128.14
128.02
127.29
77.48
77.16
76.84
60.73
21.25
18.05
f1 (ppm)


O
Ph
N
OMe

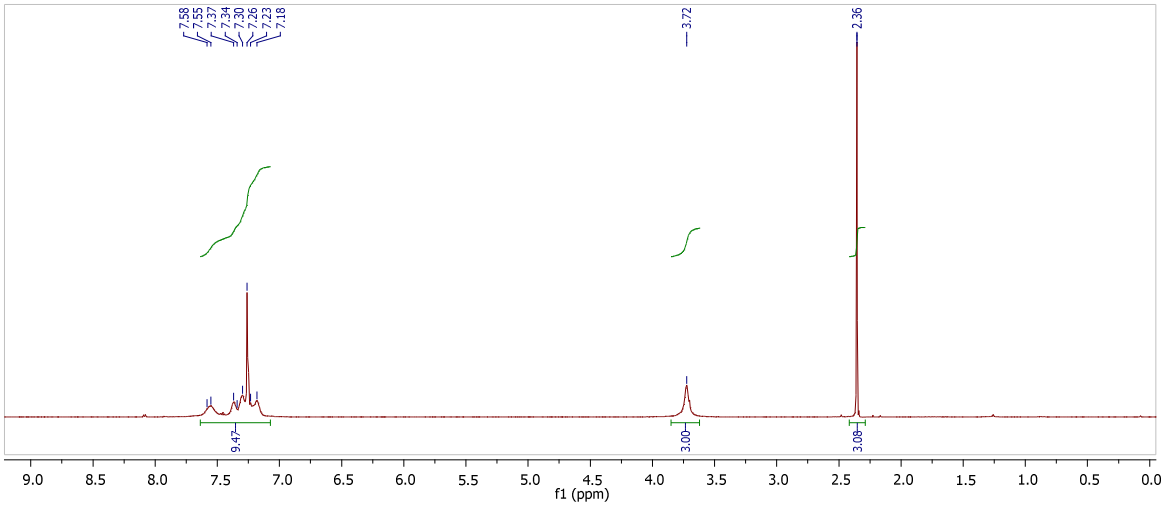
2h

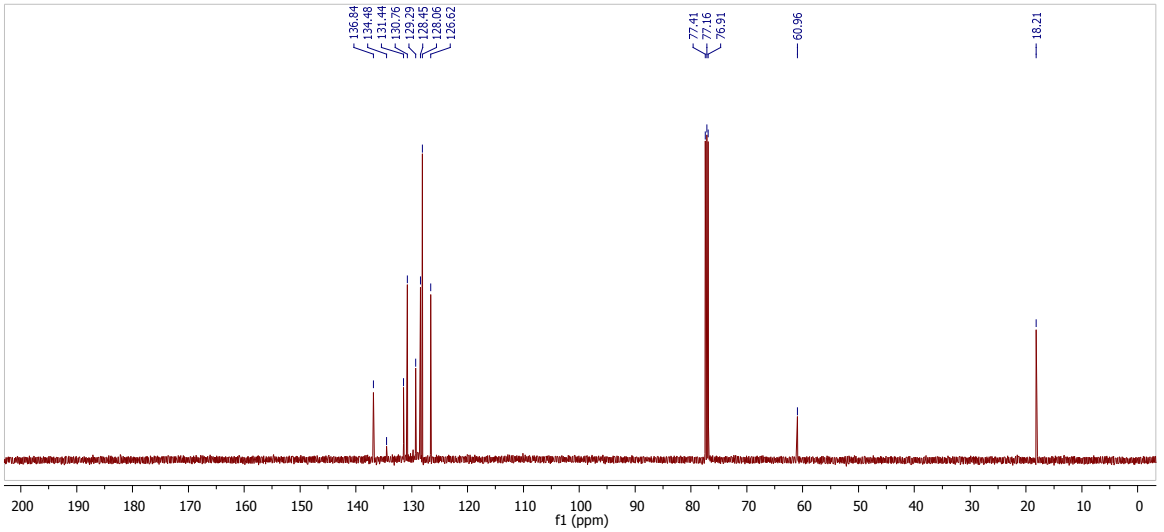

7.59
7.59
7.58
7.58
7.57
7.57
7.46
7.38
7.31
7.26
7.14
7.13
7.12
7.07
7.06
3.71
2.28
1.82
2.74
3.08
3.00
5.84
f1 (ppm)

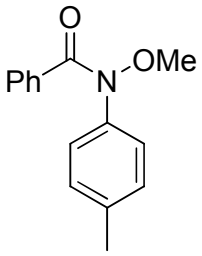

136.48
133.57
133.51
131.17
130.78
130.24
130.20
128.52
128.44
128.04
77.41
77.16
76.91
60.97
20.86
17.69
f1 (ppm)



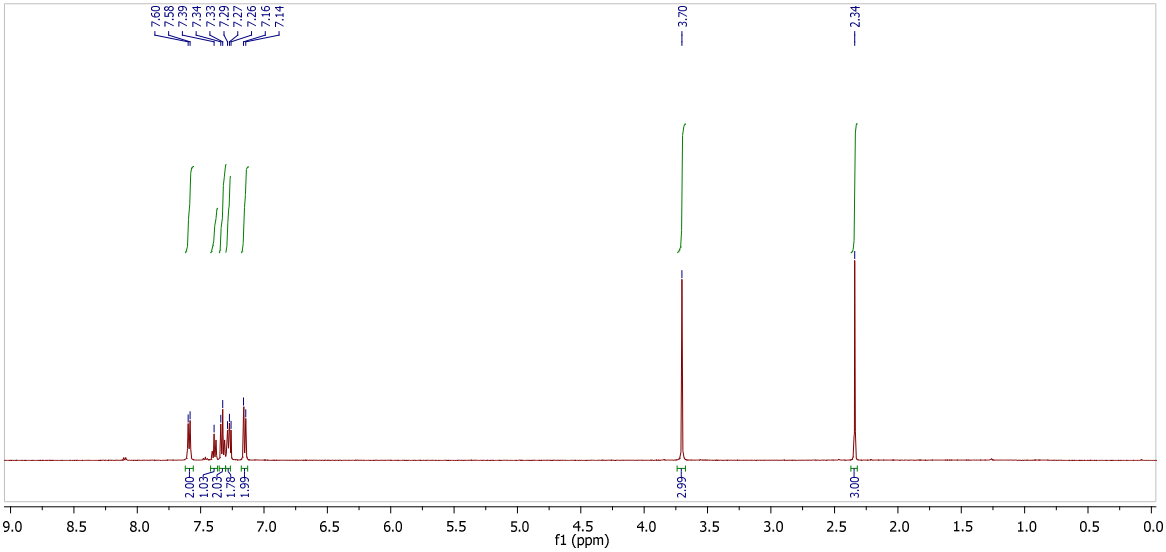
2e *ortho* isomer

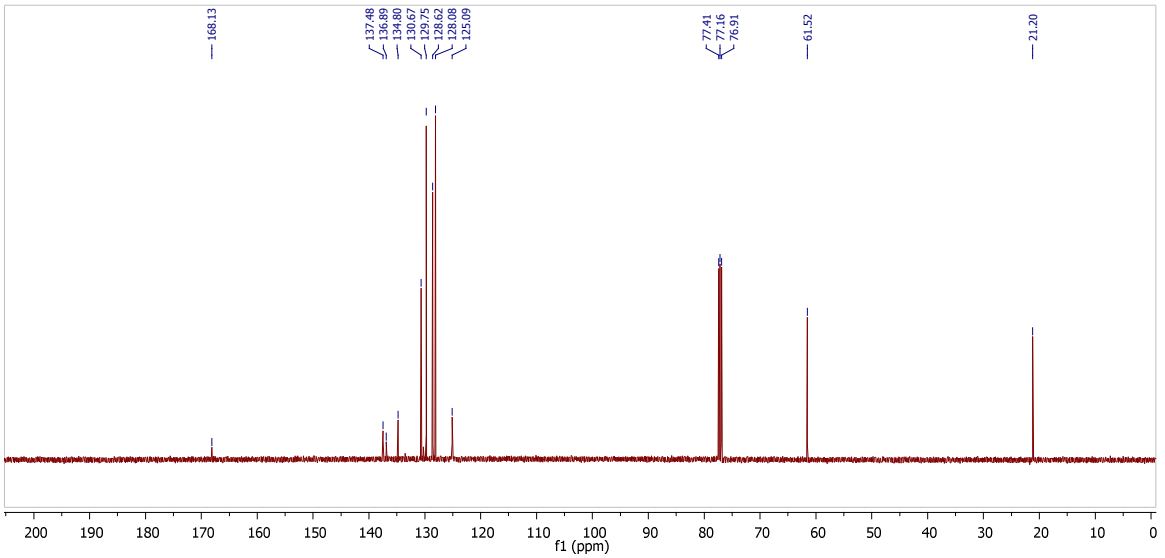


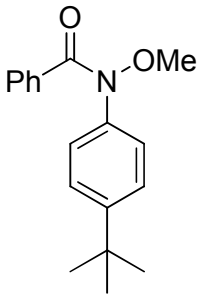




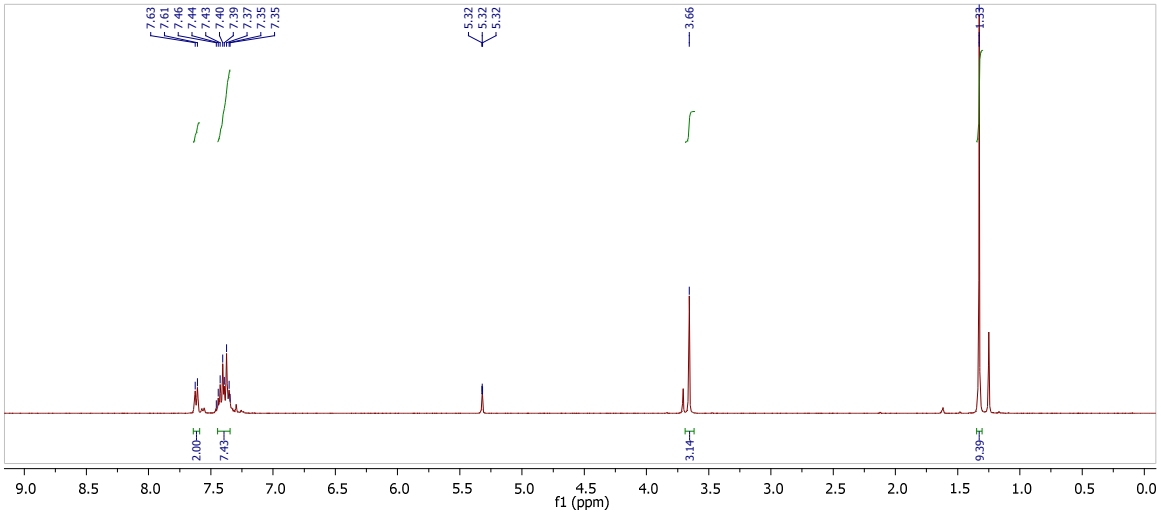
2e *para* isomer

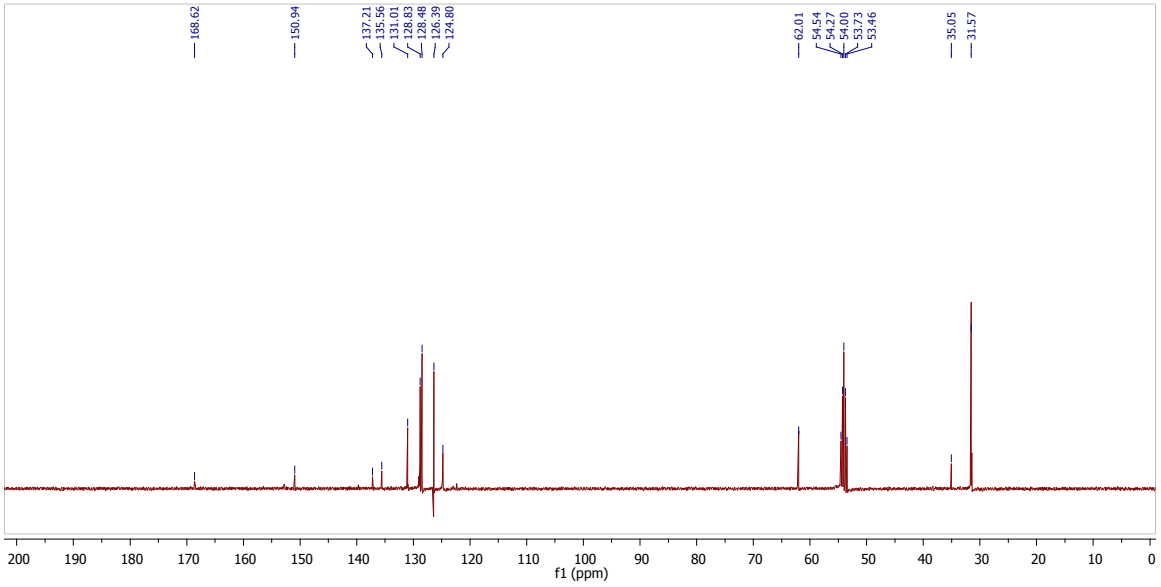


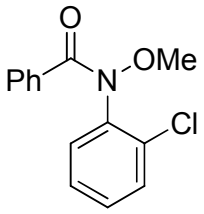




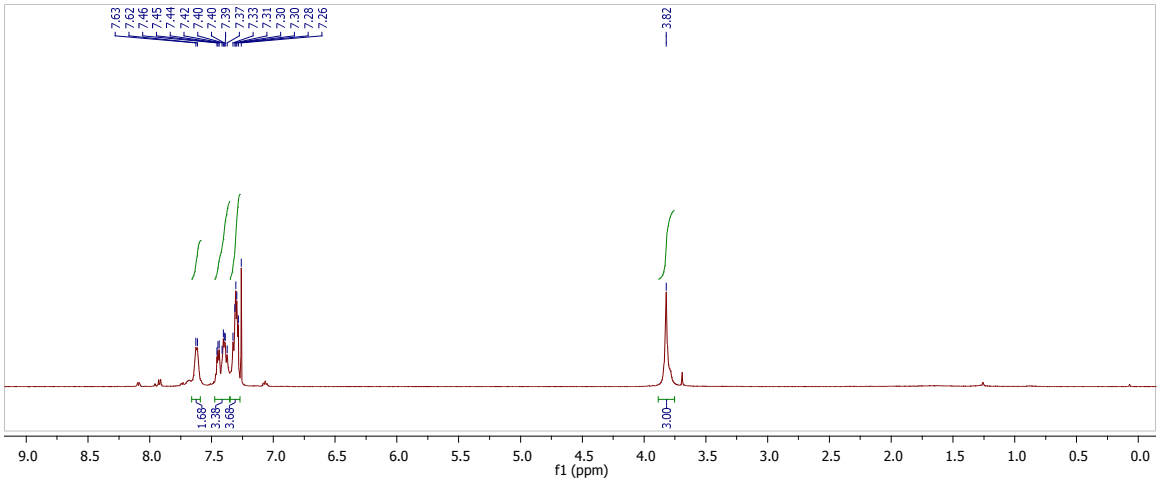
2f *para* isomer

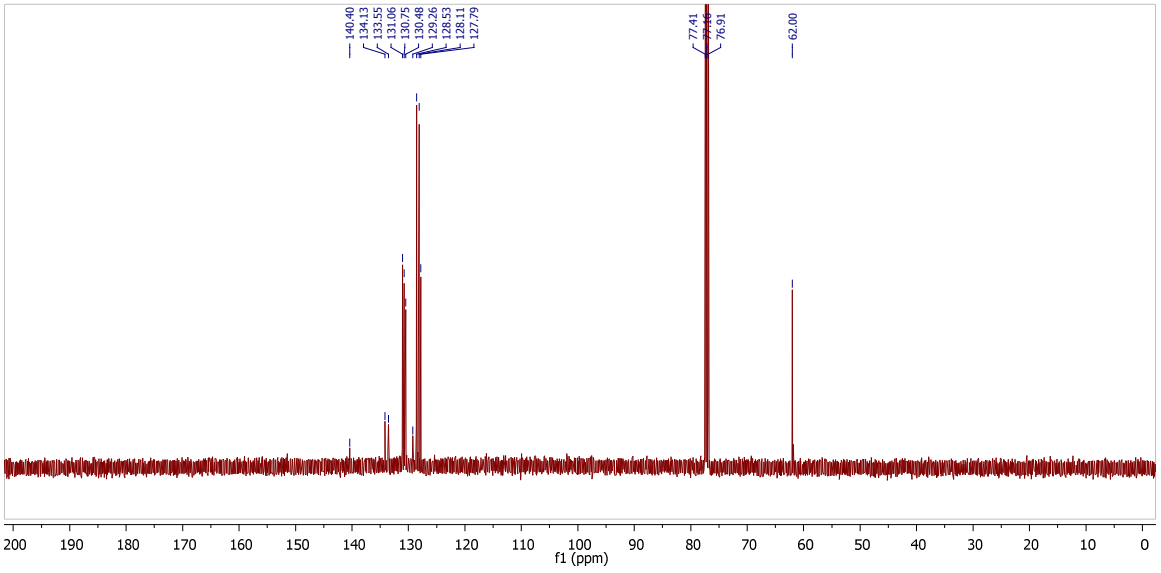


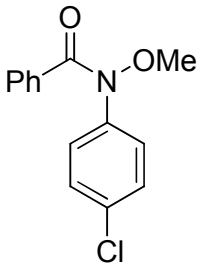




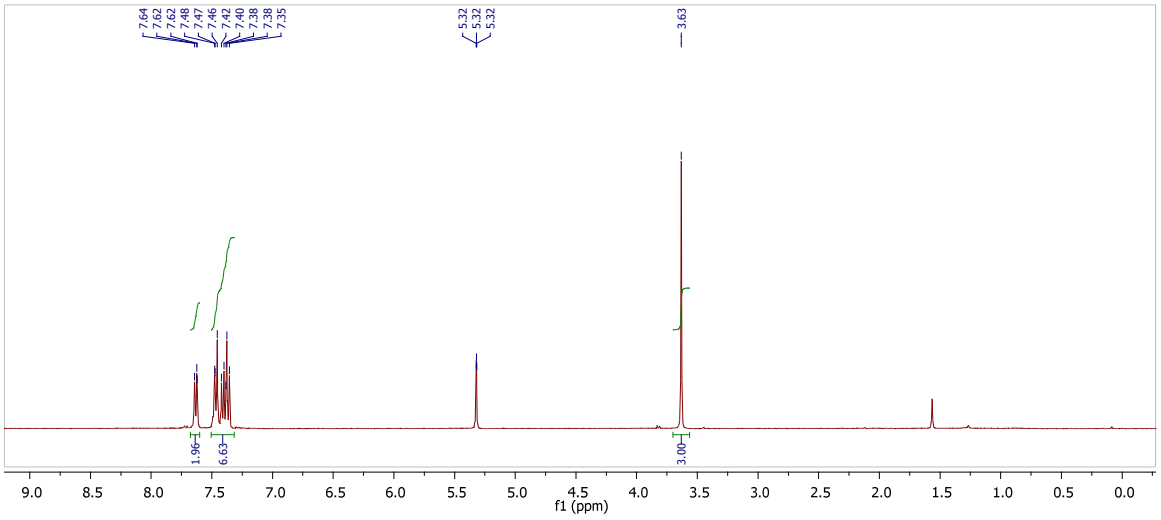
2d *ortho* isomer

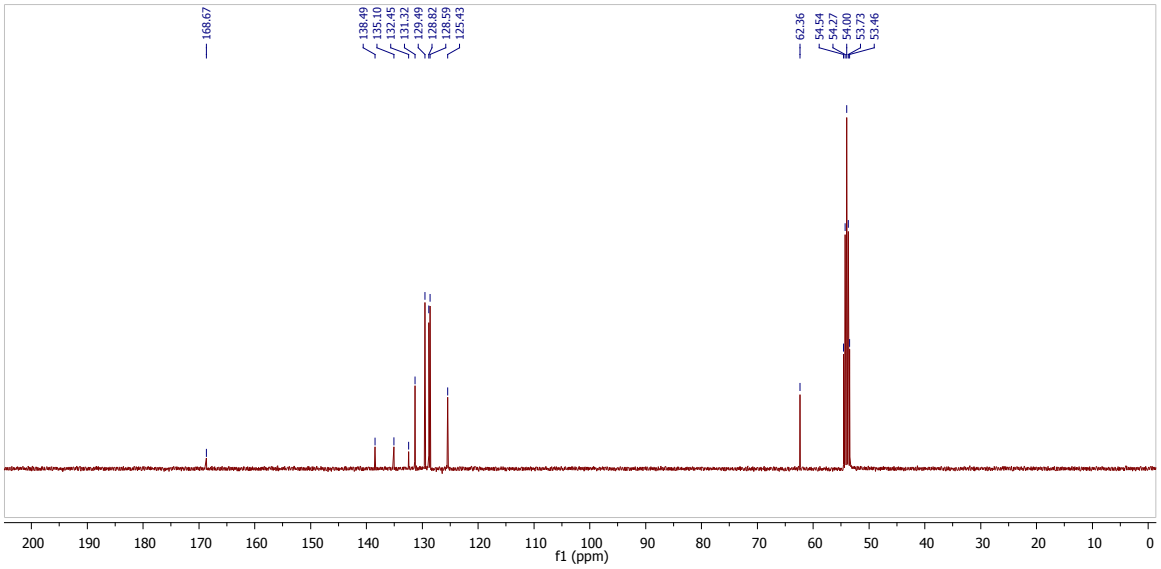


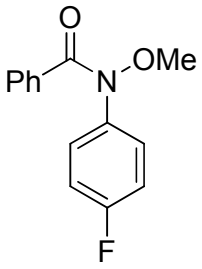




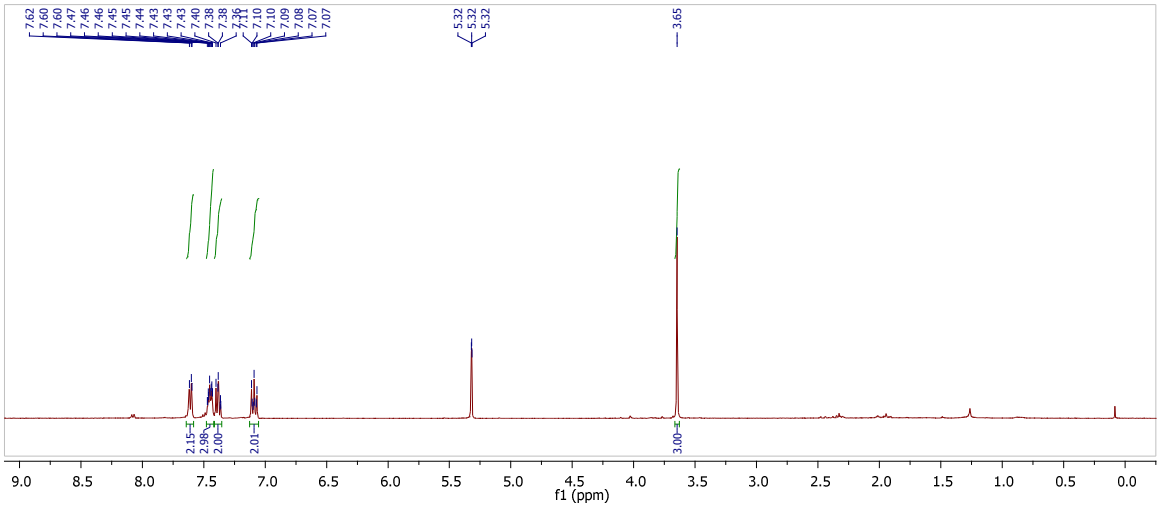
2d *para* isomer

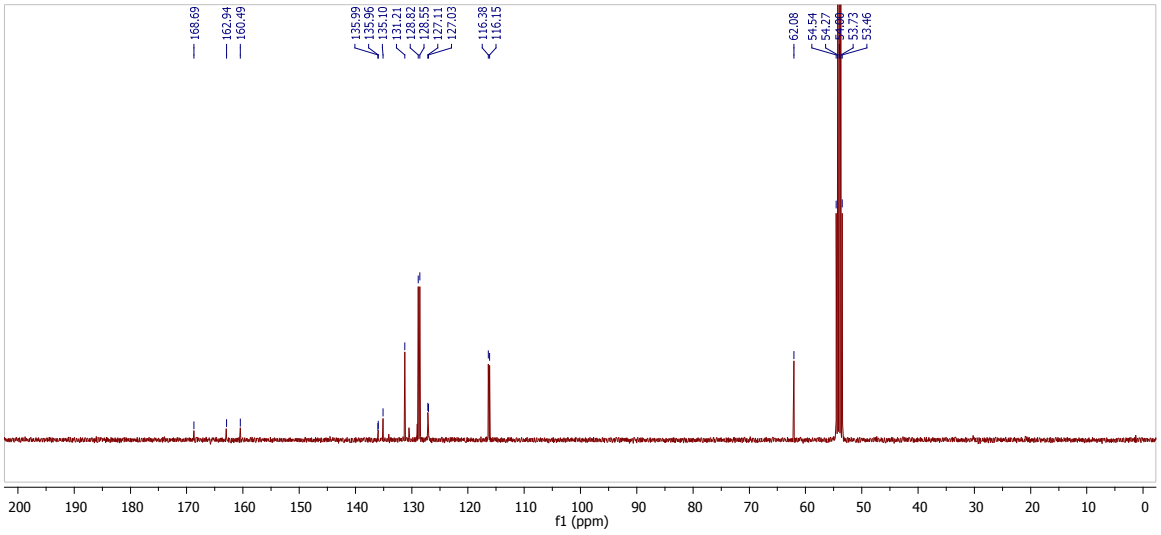


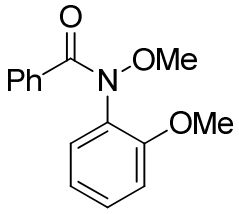




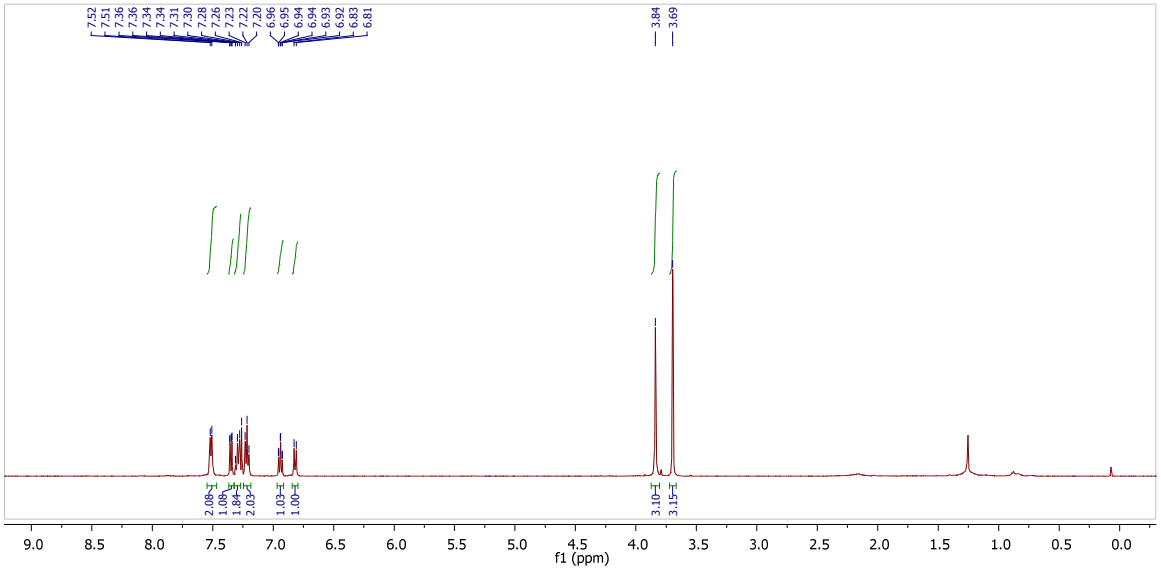
2c *para* isomer

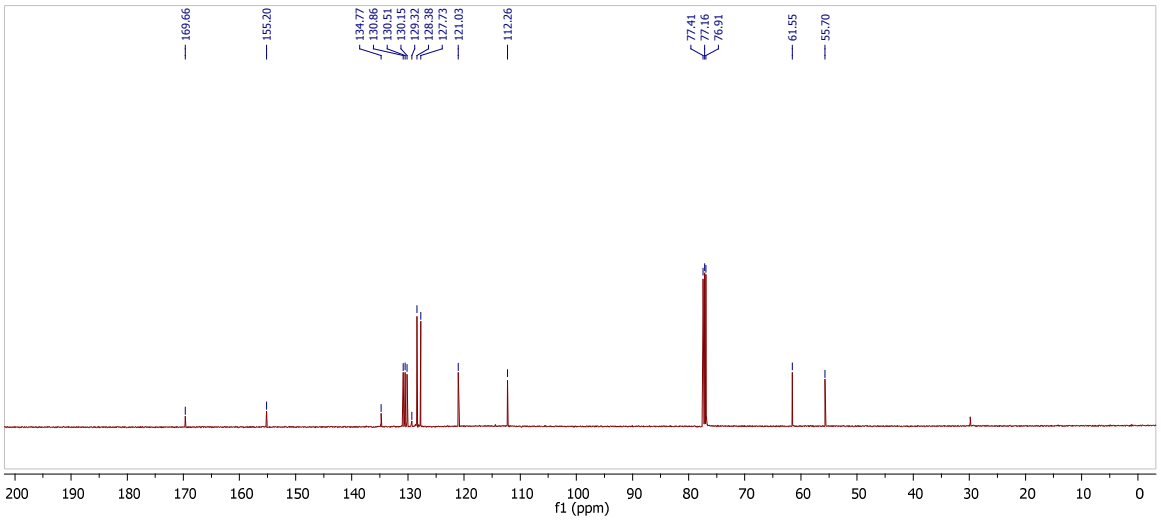


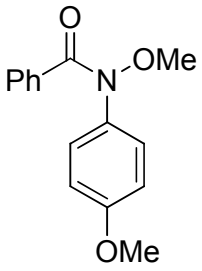




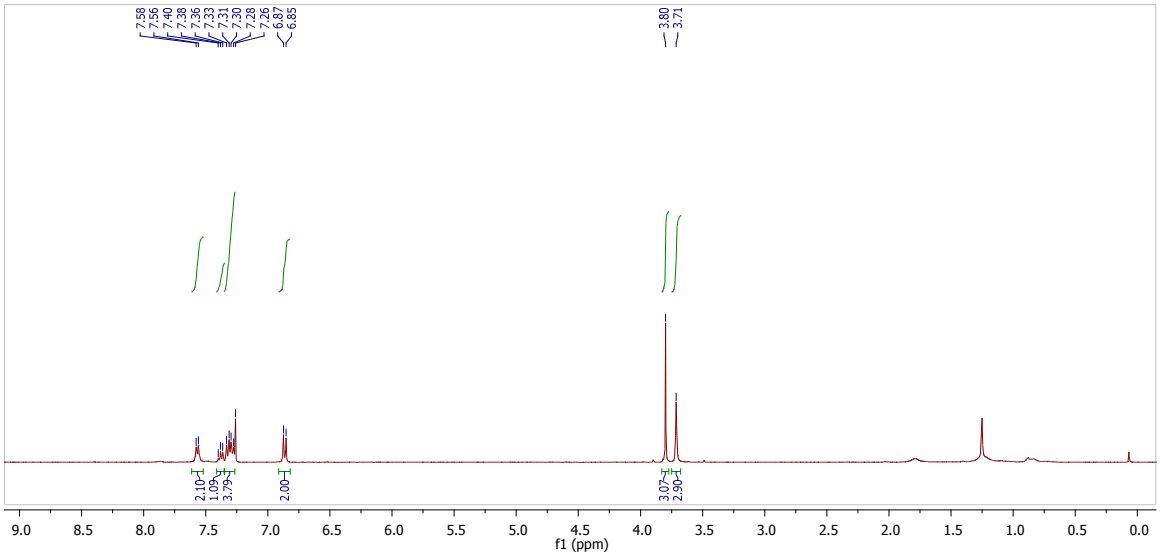
2a *ortho* isomer

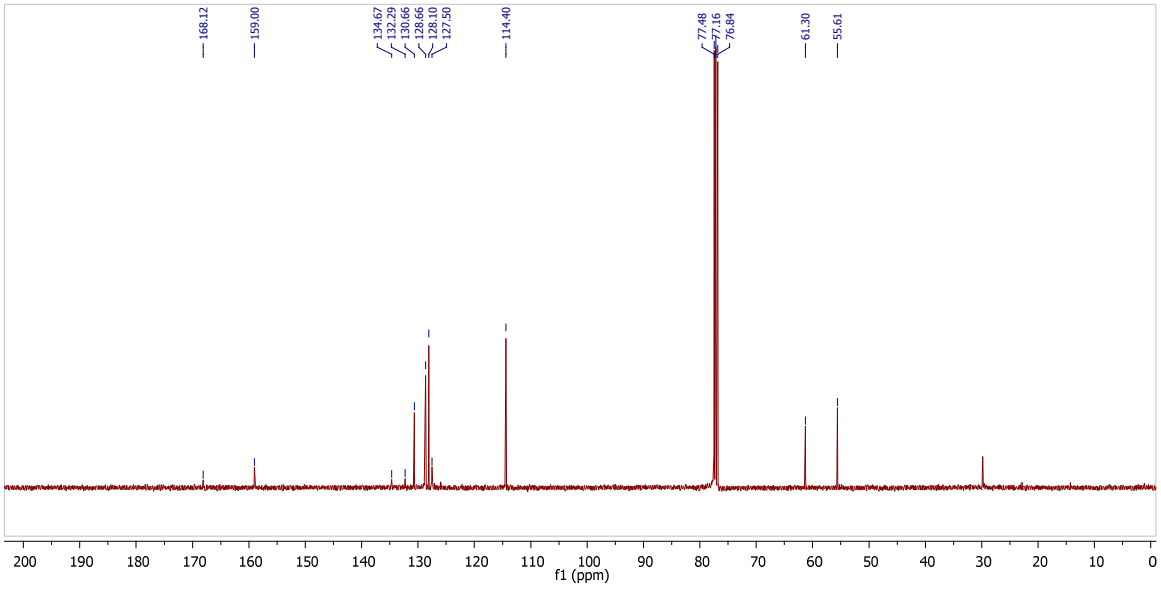


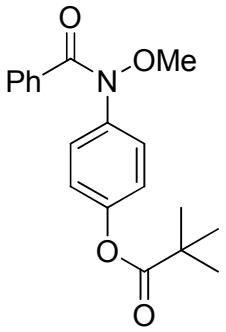




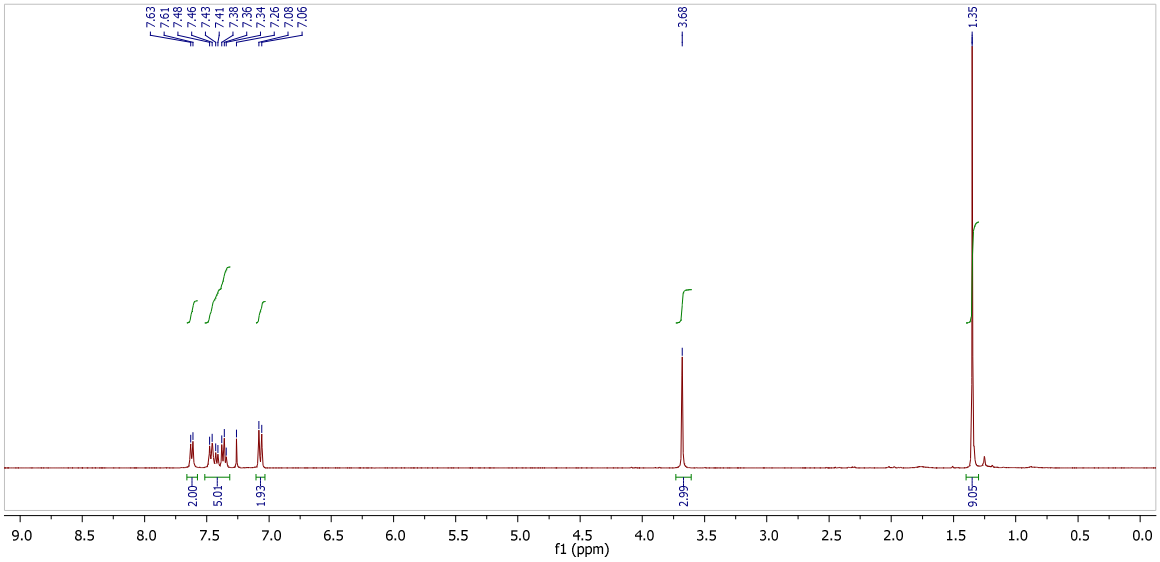
2a *para* isomer

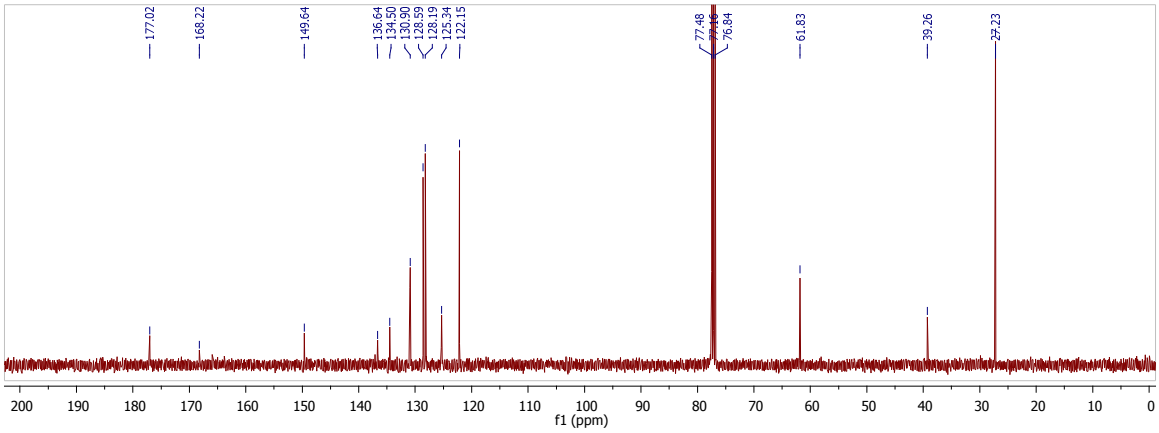


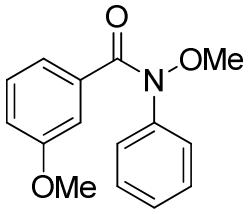




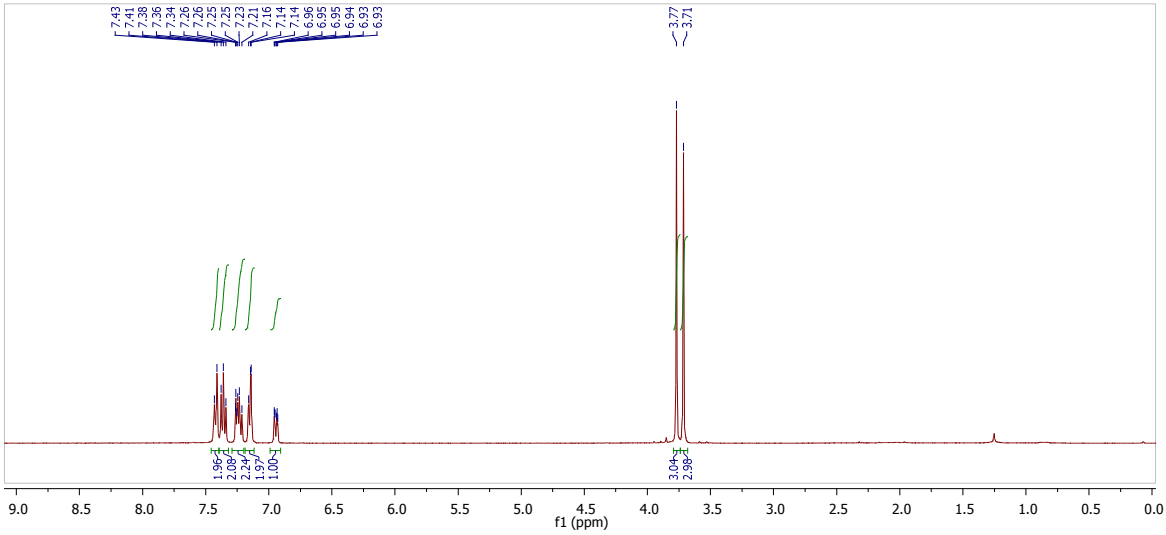
2g *para* isomer






O
N
OMe
OMe

2o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7.38
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7.34
7.26
7.26
7.25
7.25
7.23
7.21
7.16
7.14
7.14
6.96
6.95
6.95
6.94
6.93
6.93
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3.71
1.96
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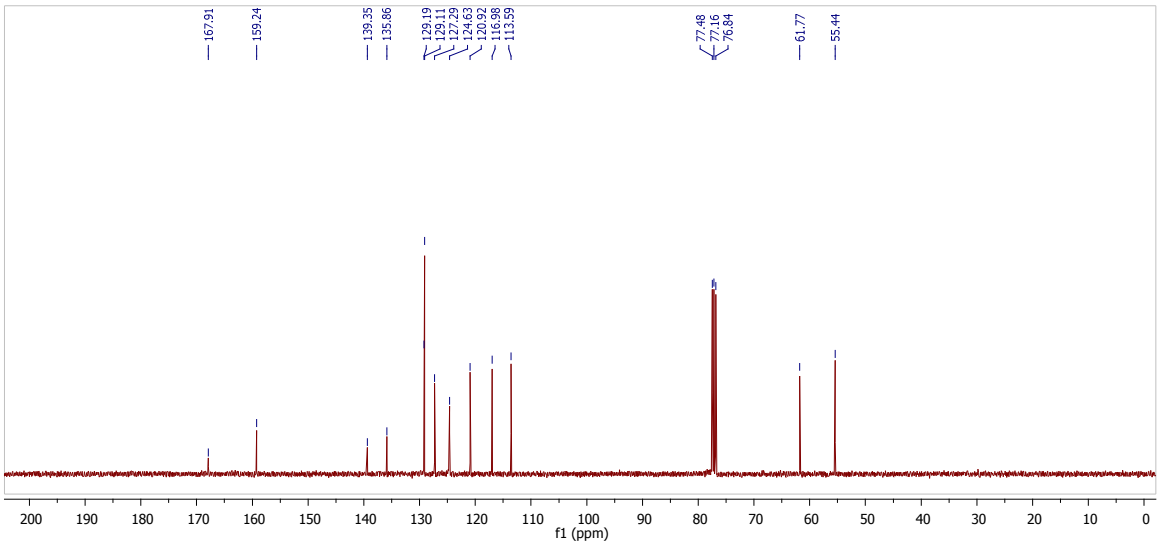
1.97
1.00
3.04
2.98
f1 (ppm)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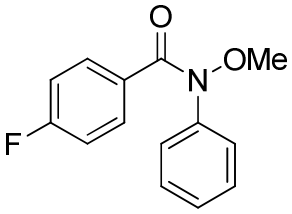
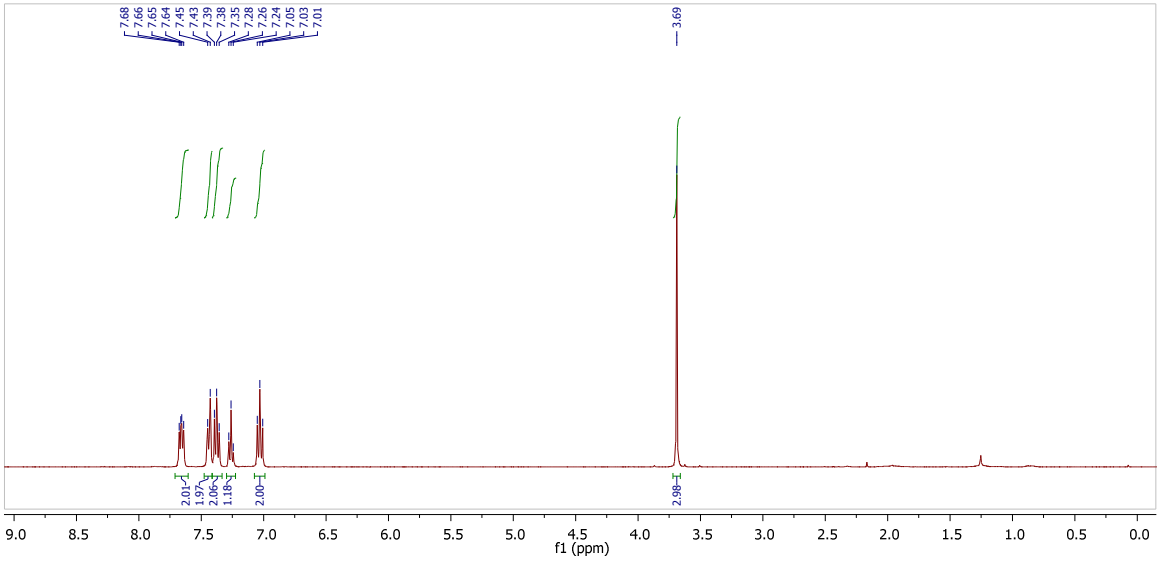
159.24
139.35
135.86
129.19
129.11
127.29
124.63
120.92
116.98
113.59
77.48
77.16
76.84
61.77
55.44
f1 (ppm)


O
N
OMe
F

2n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7.66
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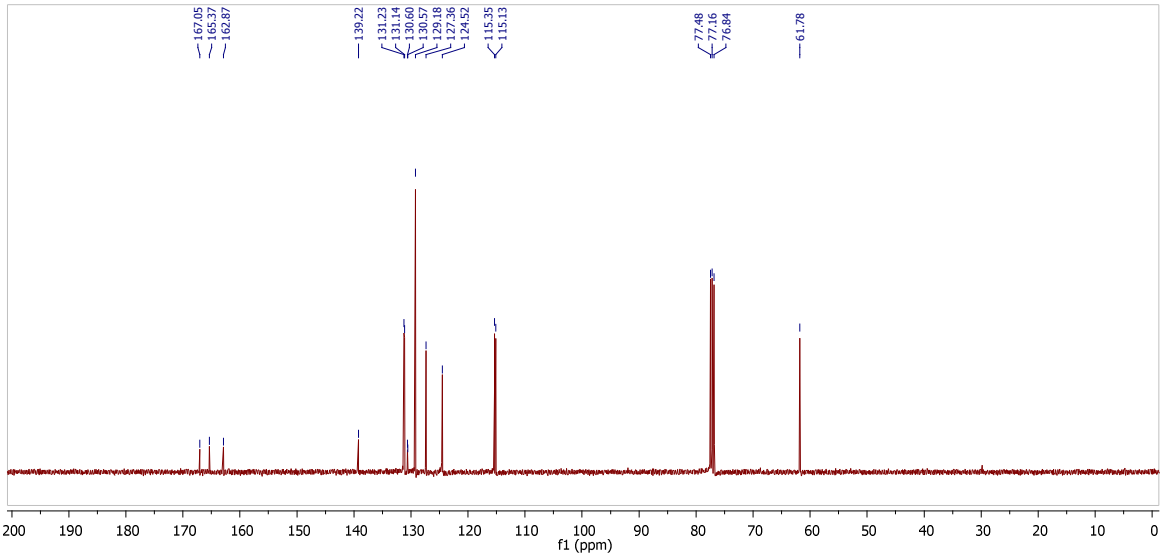
7.64
7.45
7.43
7.39
7.38
7.35
7.28
7.26
7.24
7.05
7.03
7.01
3.69
2.01
1.97
2.06
1.18
2.00
2.98
f1 (ppm)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131.14
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127.36
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115.35
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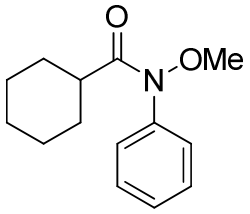
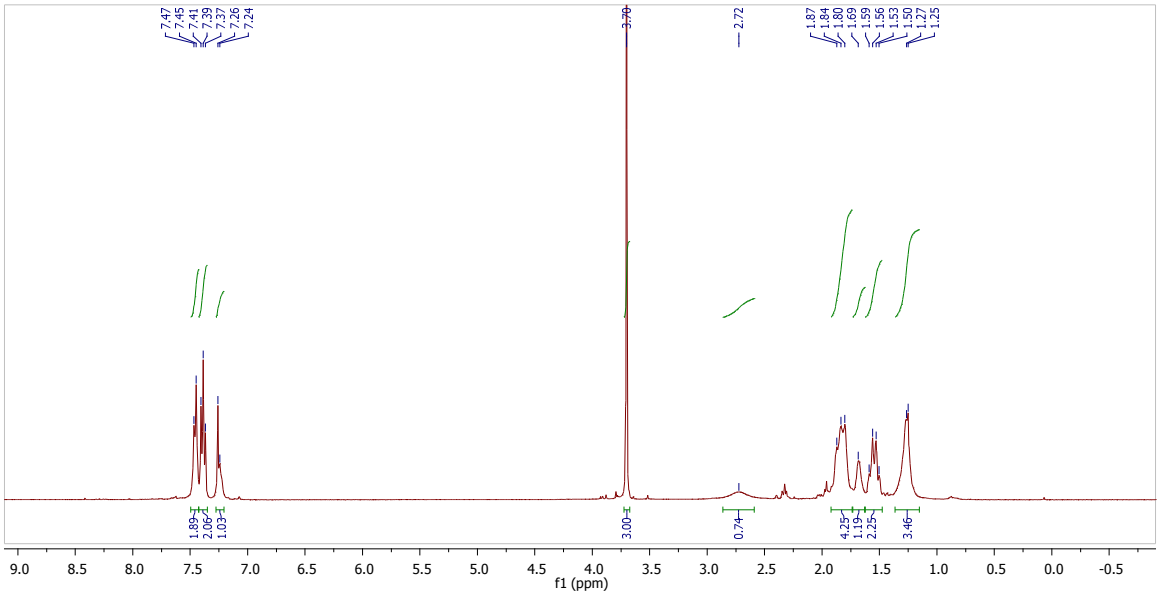
77.48
77.16
76.84
61.78
f1 (ppm)


O
N
OMe

2p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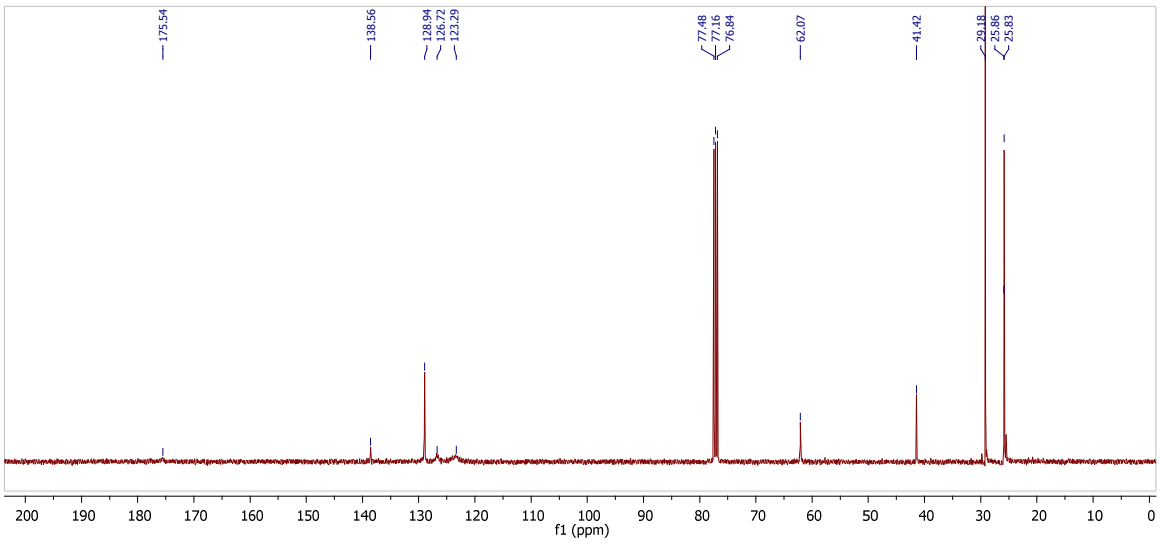
7.45
7.41
7.39
7.37
7.26
7.24
3.70
2.72
1.87
1.84
1.80
1.69
1.59
1.56
1.53
1.50
1.27
1.25
1.89
2.06
1.03
3.00
0.74
4.25
1.19
2.25
3.46
f1 (ppm)


175.54
138.56
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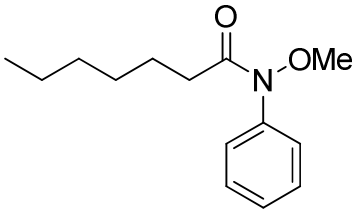
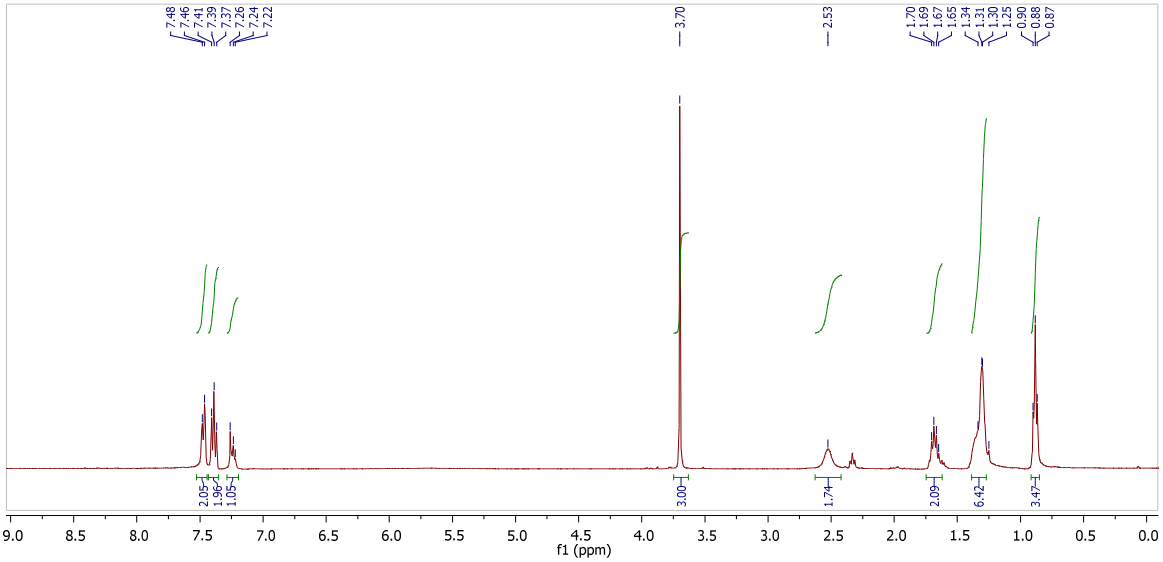
123.29
77.48
77.16
76.84
62.07
41.42
29.18
25.86
25.83
f1 (ppm)


O
N
OMe

2q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7.46
7.41
7.39
7.37
7.26
7.24
7.22
3.70
2.53
1.70
1.69
1.67
1.65
1.34
1.31
1.30
1.25
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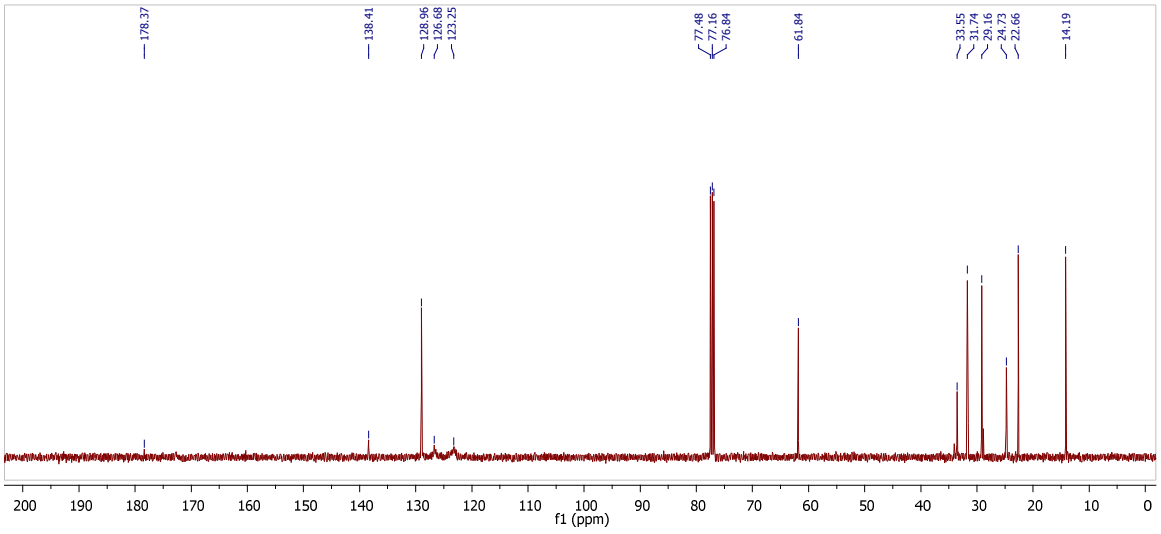
0.87
2.05
1.96
1.05
3.00
1.74
2.09
6.42
3.47
f1 (ppm)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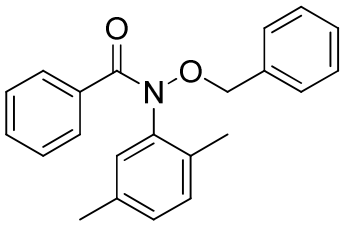
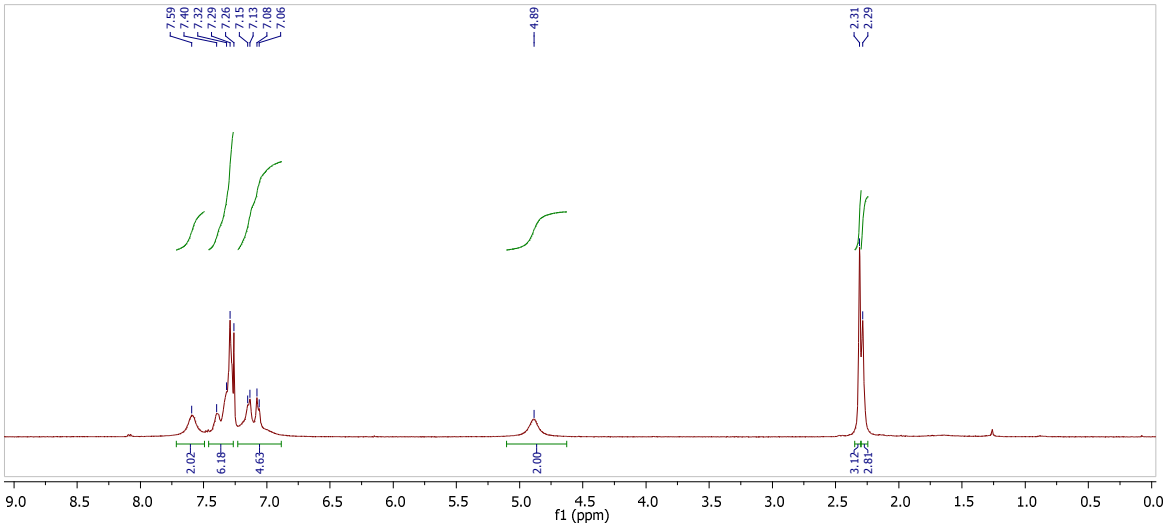
76.84
61.84
33.55
31.74
29.16
24.73
22.66
14.19
f1 (ppm)



2m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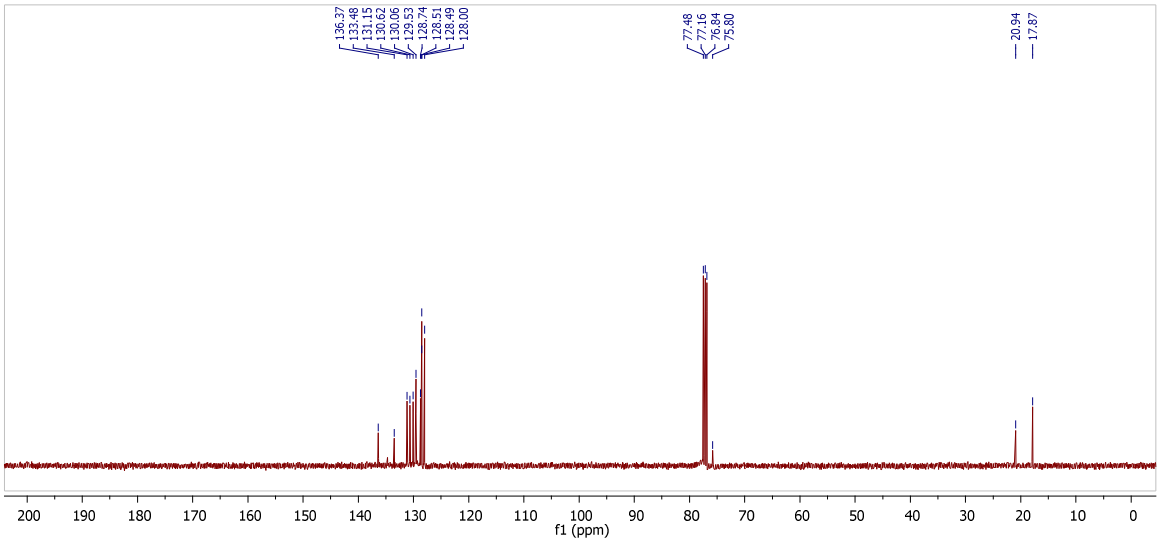
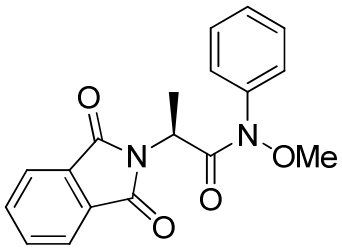
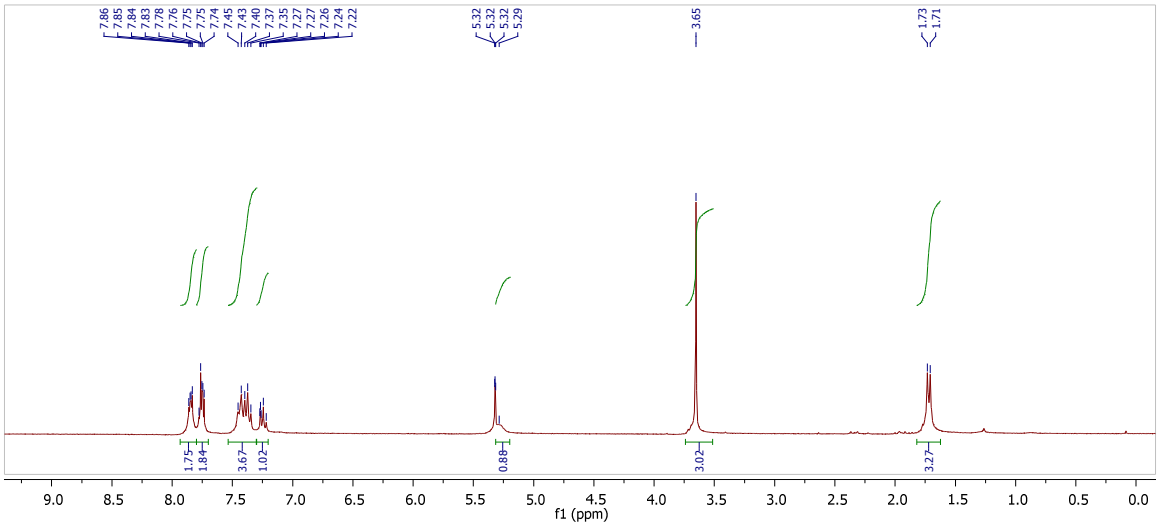
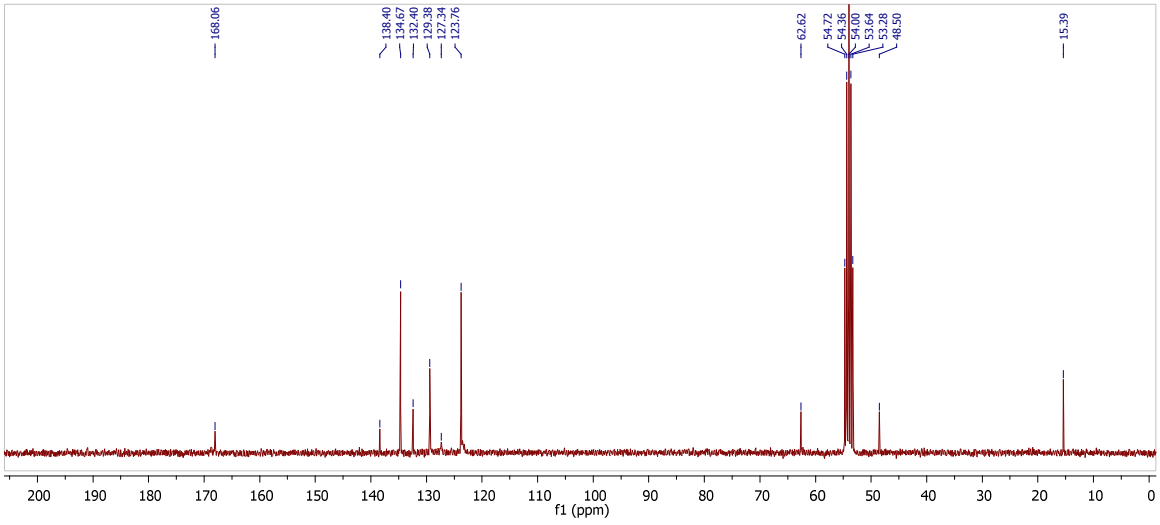
7.29
7.26
7.15
7.13
7.08
7.06
4.89
2.31
2.29
2.02
6.18
4.63
2.00
3.12
2.81
f1 (ppm)

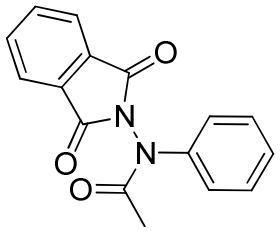

136.37
133.48
131.15
130.62
130.06
129.53
128.74
128.51
128.49
128.00
77.48
77.16
76.84
75.80
20.94
17.87
f1 (ppm)


O
N
O
N
OMe
O

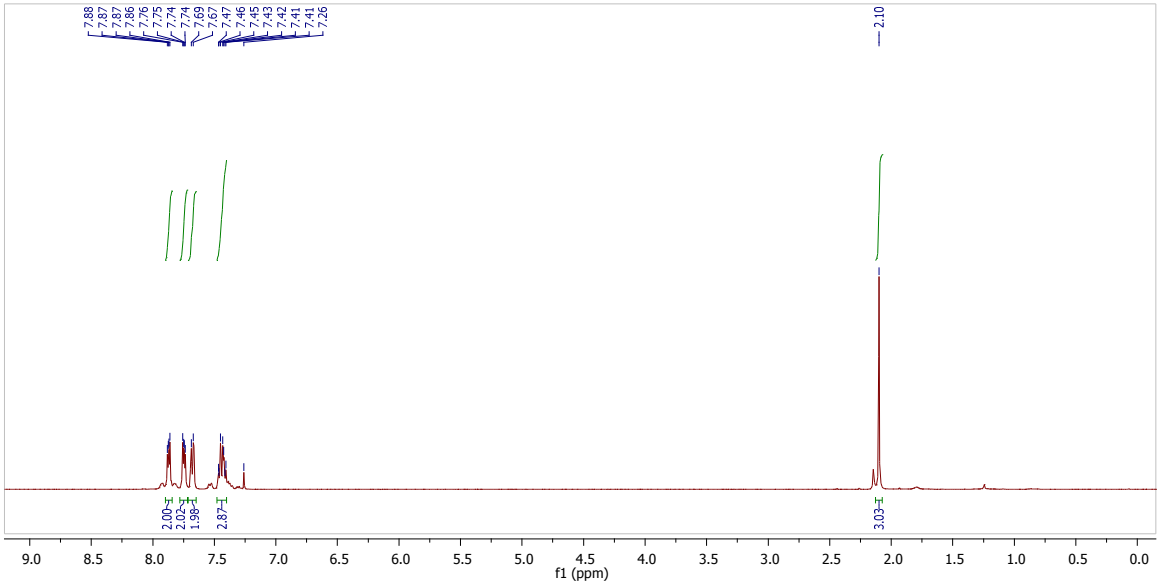
2r

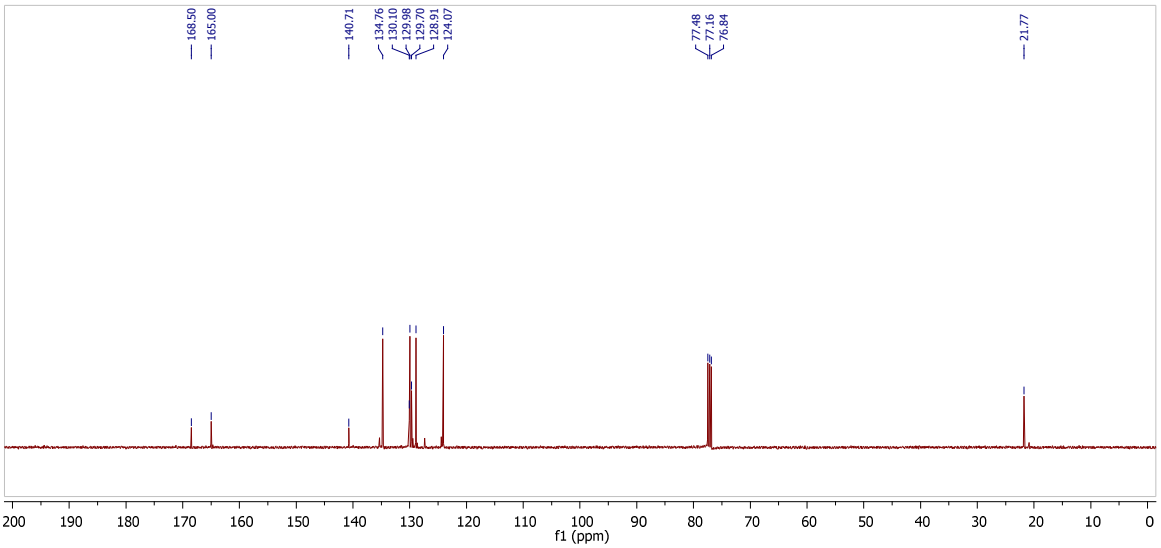

7.86
7.85
7.84
7.83
7.78
7.76
7.75
7.75
7.74
7.45
7.43
7.40
7.37
7.35
7.27
7.27
7.26
7.24
7.22
5.32
5.32
5.32
5.29
3.65
1.73
1.71
1.75
1.84
3.67
1.02
0.88
3.02
3.27
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

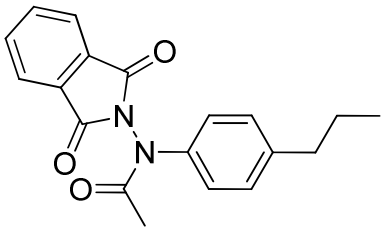

168.06
138.40
134.67
132.40
129.38
127.34
123.76
62.62
54.72
54.36
54.00
53.64
53.28
48.50
15.39
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



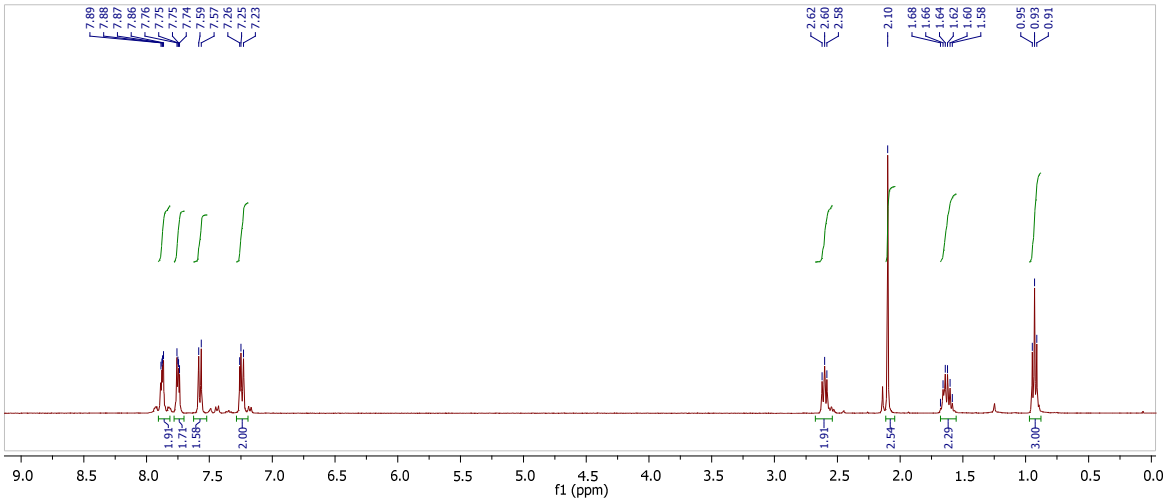
5a

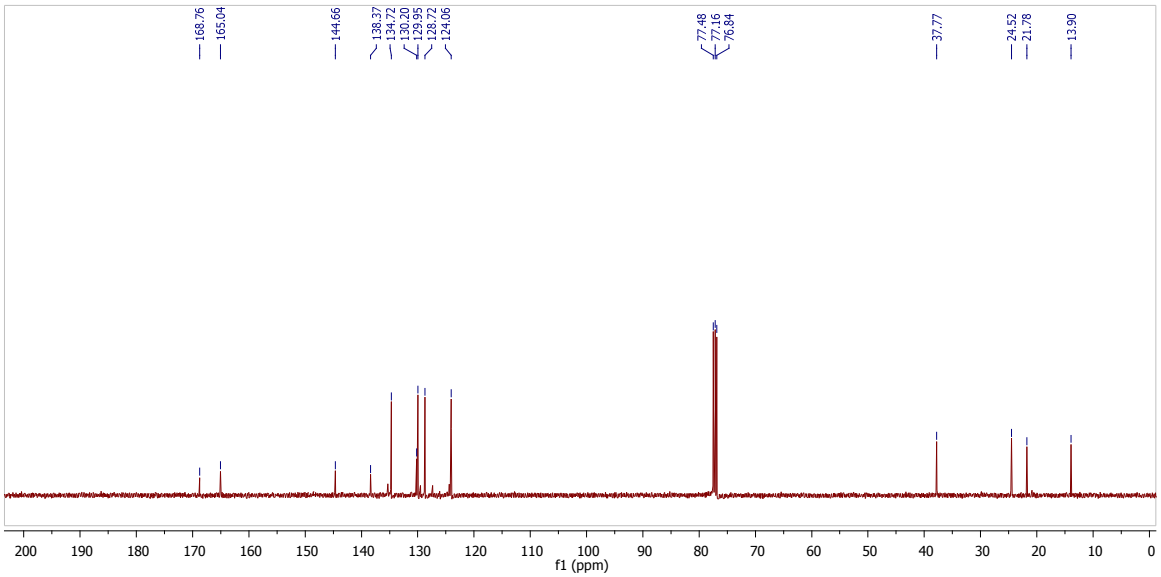

7.88
7.87
7.87
7.86
7.76
7.75
7.74
7.74
7.69
7.67
7.47
7.46
7.45
7.43
7.42
7.41
7.41
7.26
2.10
2.00
2.02
1.98
2.87
3.03
f1 (ppm)

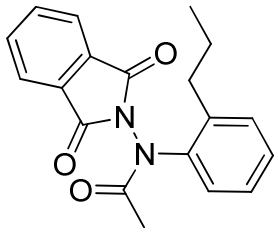

168.50
165.00
140.71
134.76
130.10
129.98
129.70
128.91
124.07
77.48
77.16
76.84
21.77
f1 (ppm)



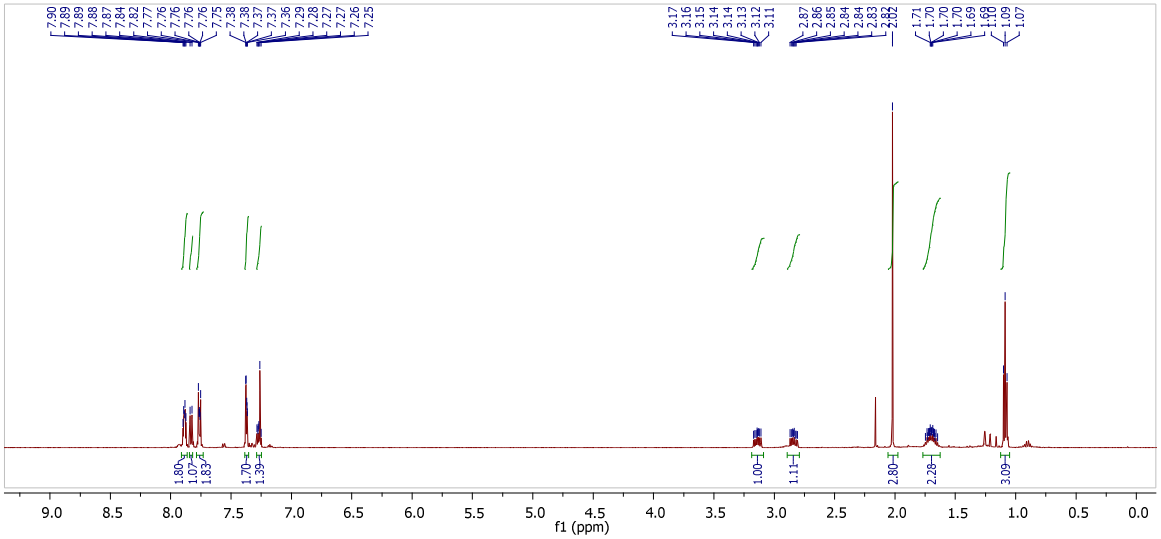
5e *para* isomer

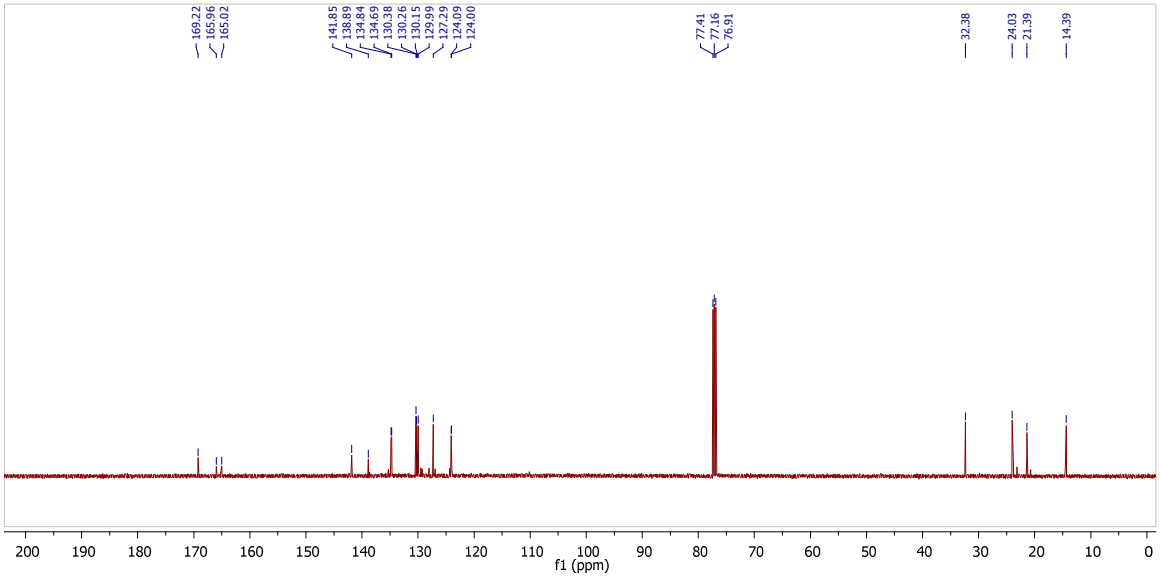


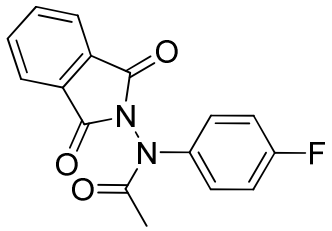




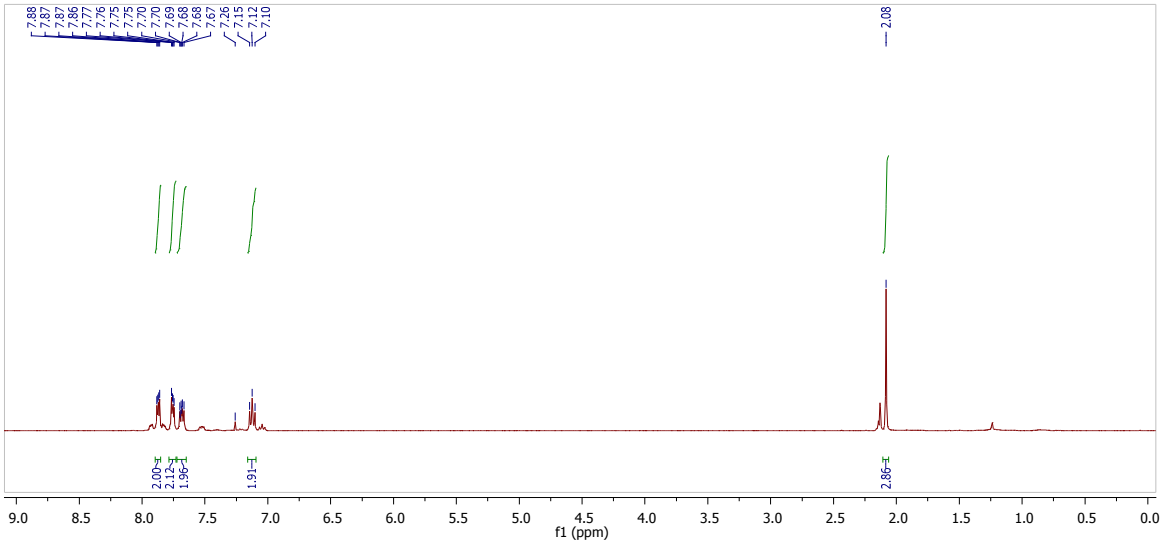
5e *ortho* isomer

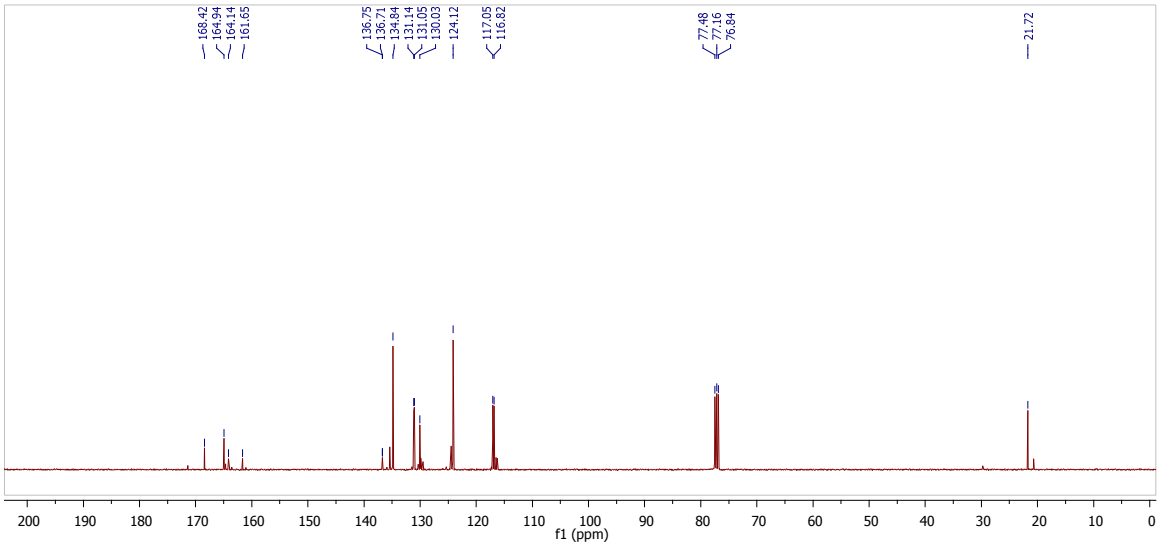


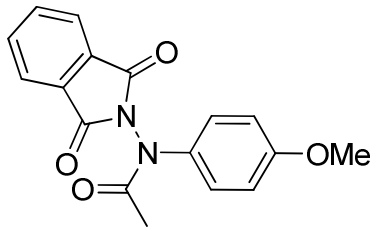




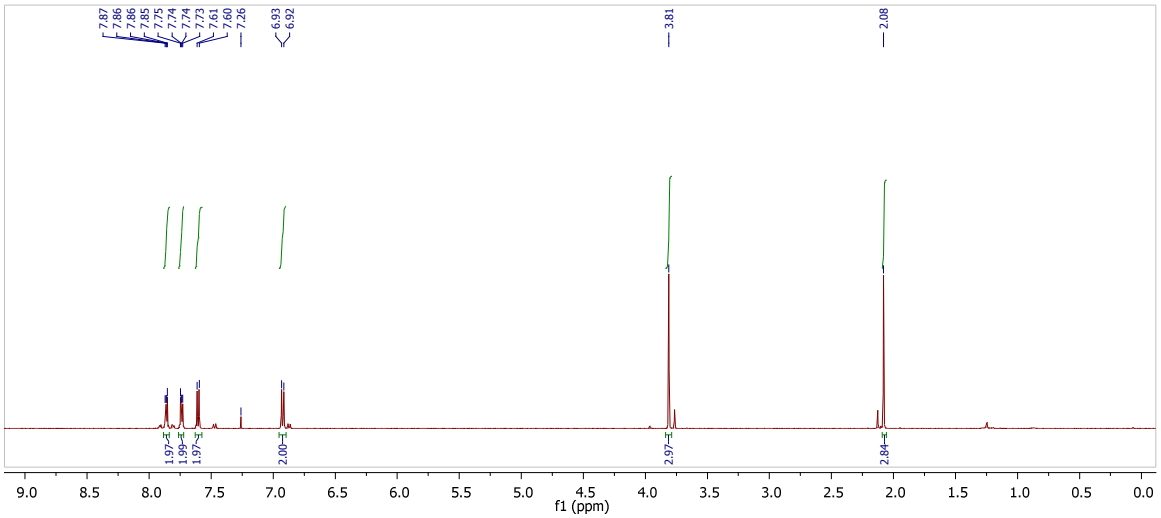
5f *para* isomer

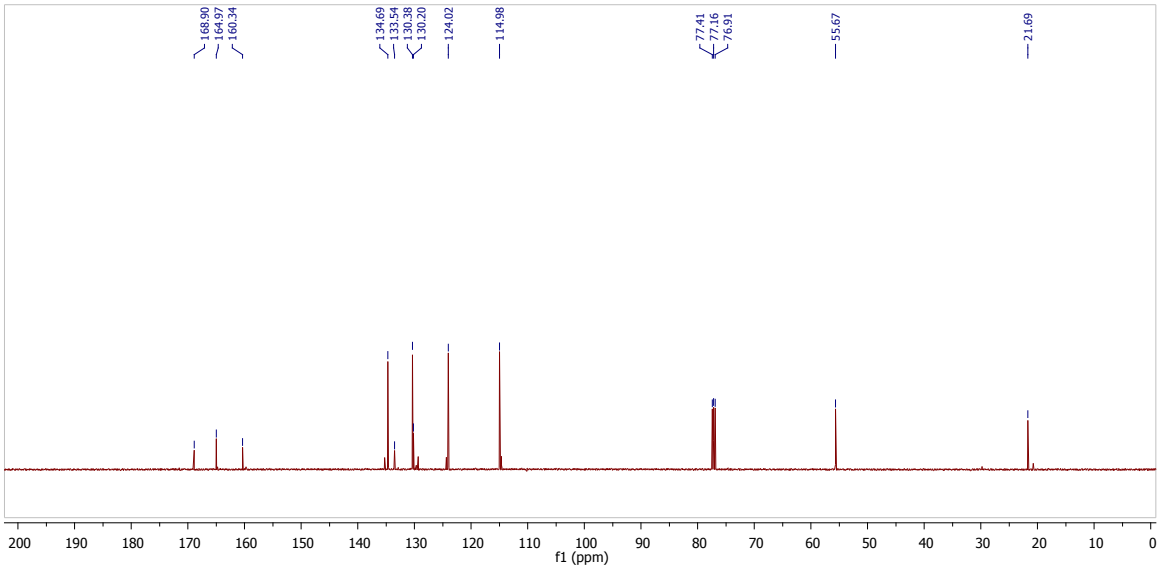


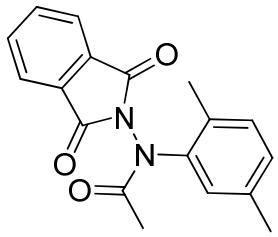




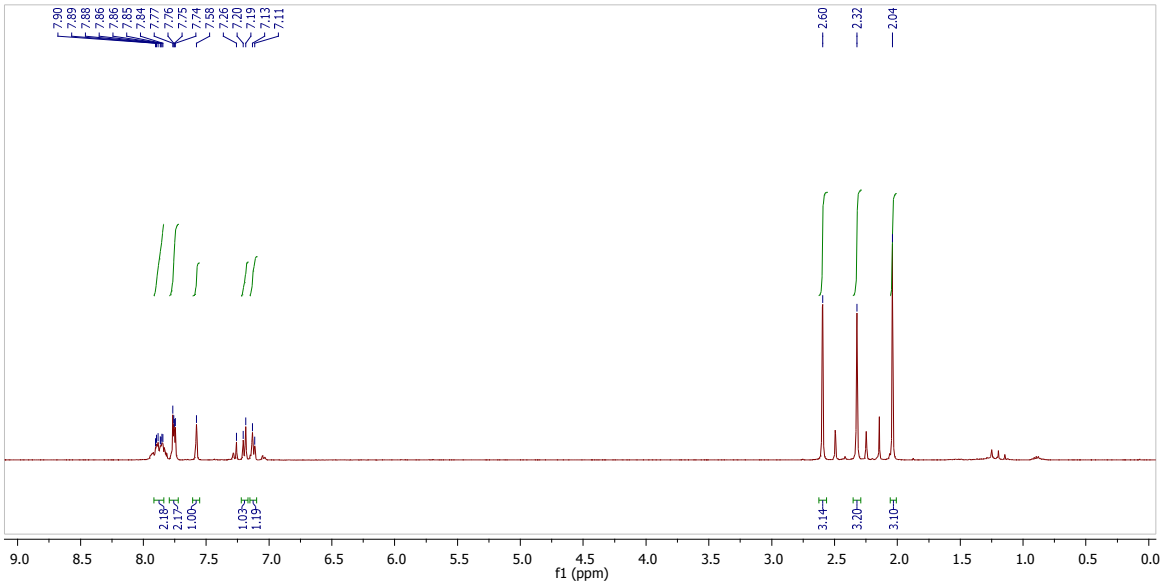
5b *para* isomer

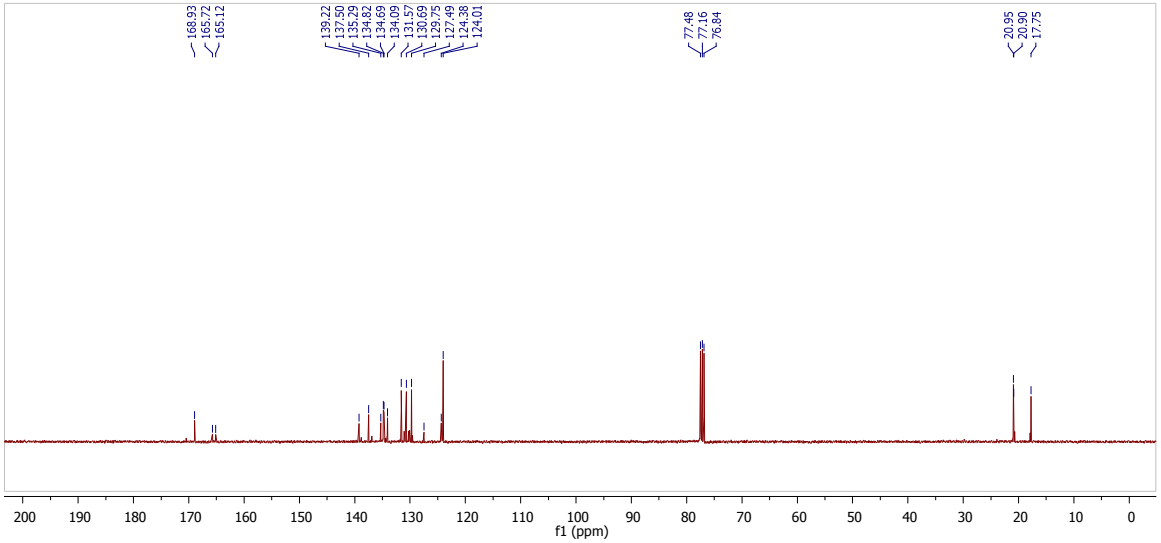






5q


7.90
7.89
7.88
7.86
7.86
7.85
7.84
7.77
7.76
7.75
7.74
7.58
7.26
7.20
7.19
7.13
7.11
2.60
2.32
2.04
2.18
2.17
1.00
1.03
1.19
3.14
3.20
3.10
f1 (ppm)


168.93
165.72
165.12
139.22
137.50
135.29
134.82
134.69
134.09
131.57
130.69
129.75
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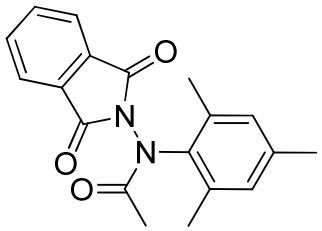
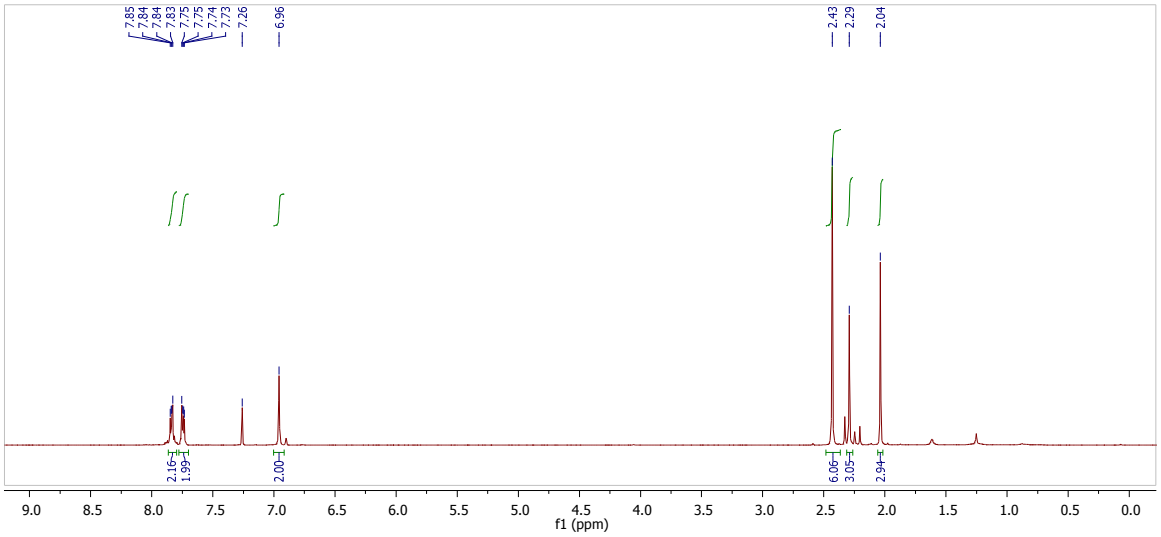
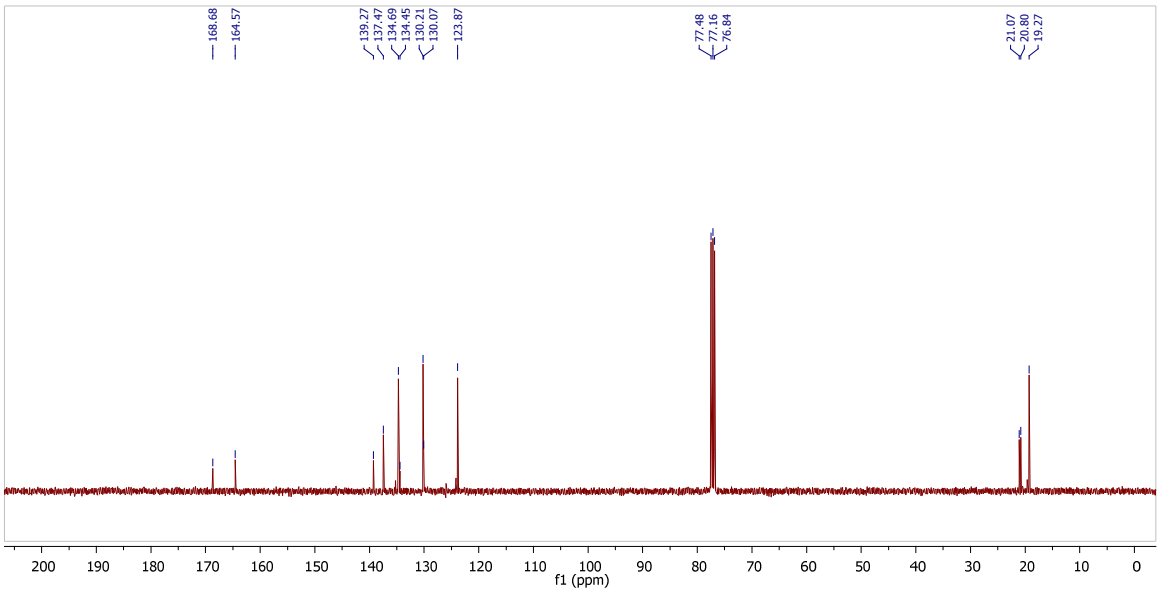
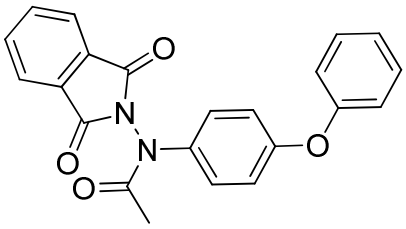
124.01
77.48
77.16
76.84
20.95
20.90
17.75
f1 (ppm)



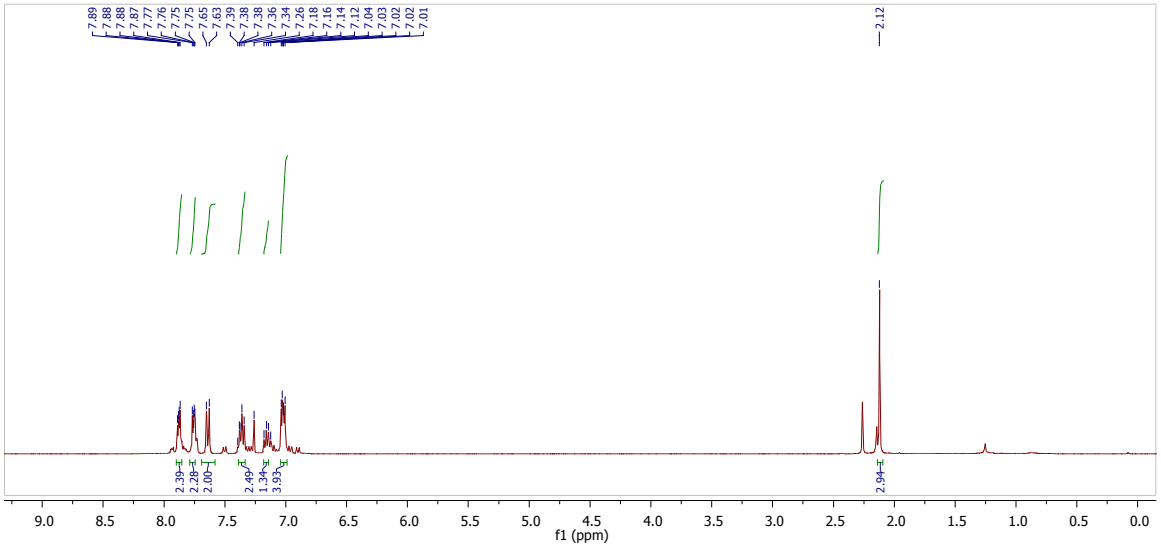
5r

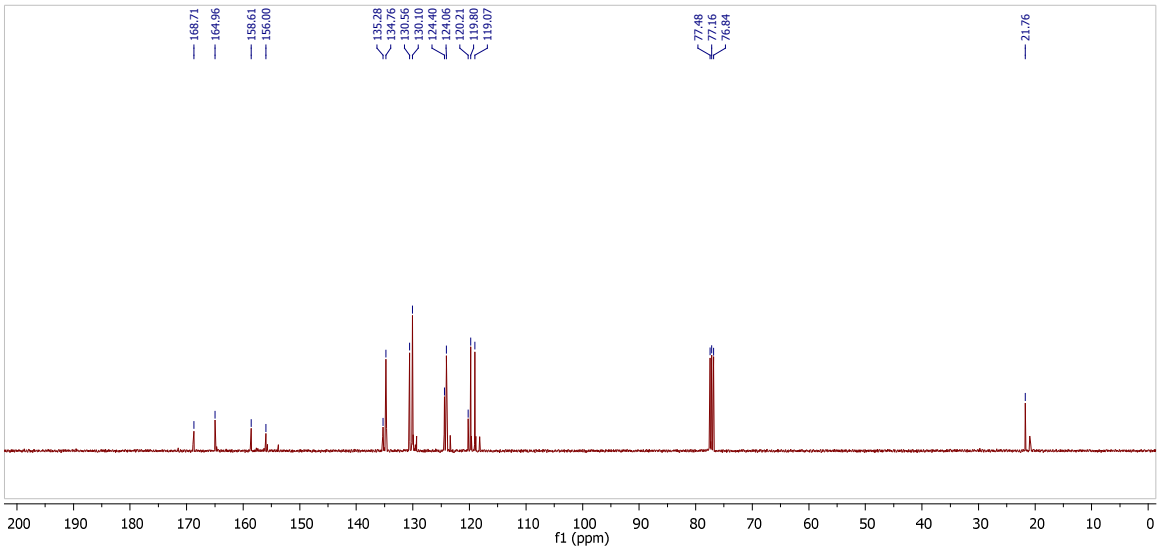

7.85
7.84
7.84
7.83
7.75
7.75
7.74
7.73
7.26
6.96
2.43
2.29
2.04
2.16
1.99
2.00
6.06
3.05
2.94
f1 (ppm)

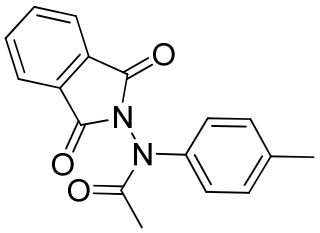

168.68
164.57
139.27
137.47
134.69
134.45
130.21
130.07
123.87
77.48
77.16
76.84
21.07
20.80
19.27
f1 (ppm)



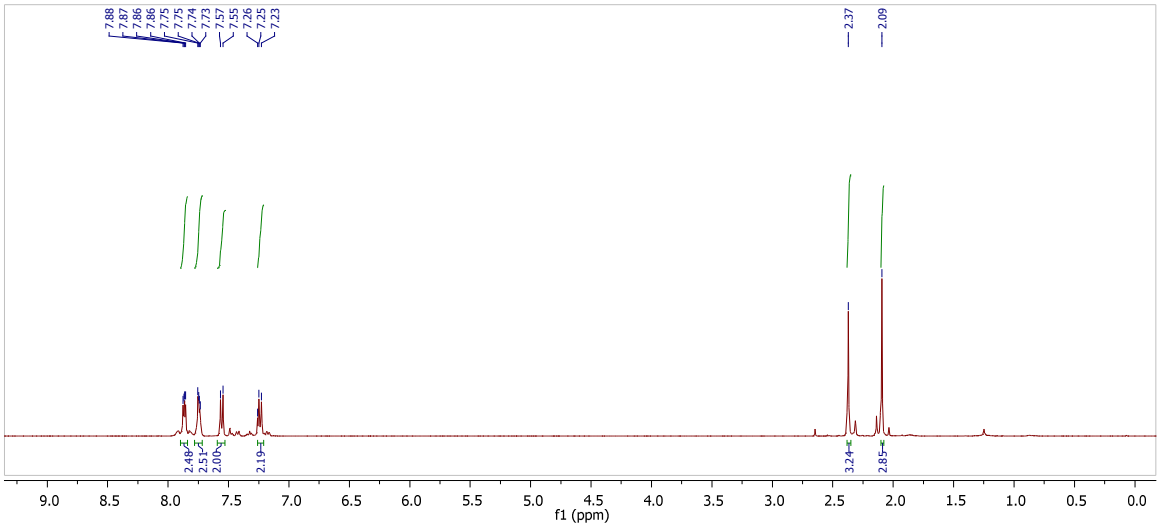
5c *para* isomer

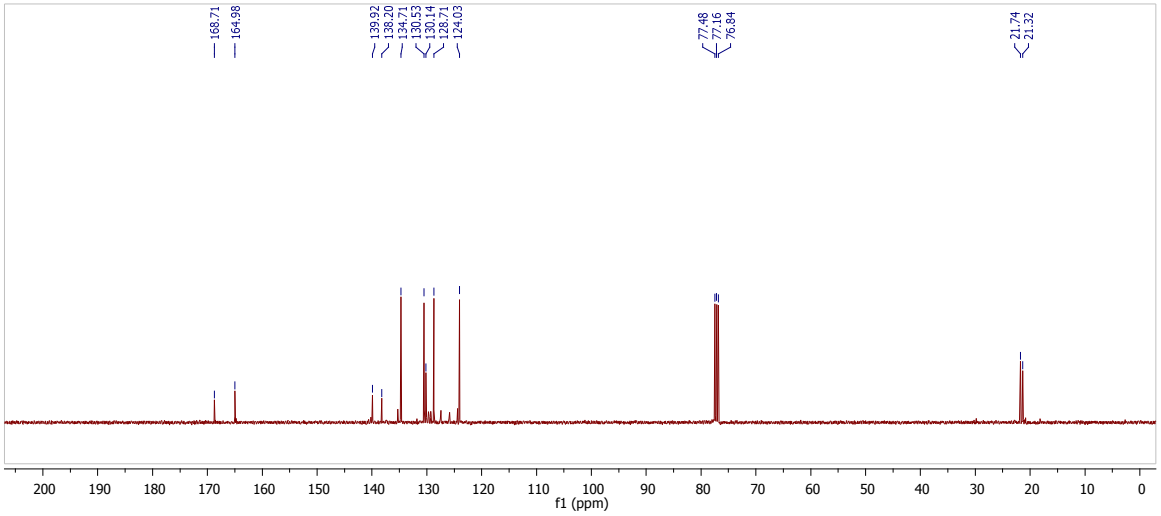


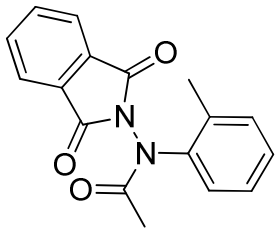




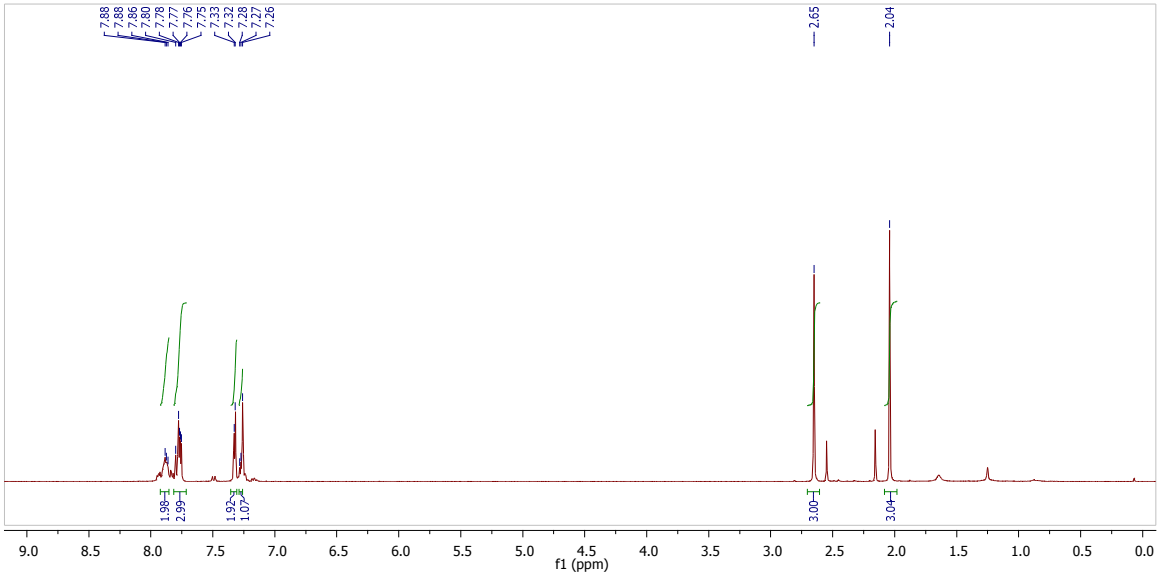
5d *para* isomer

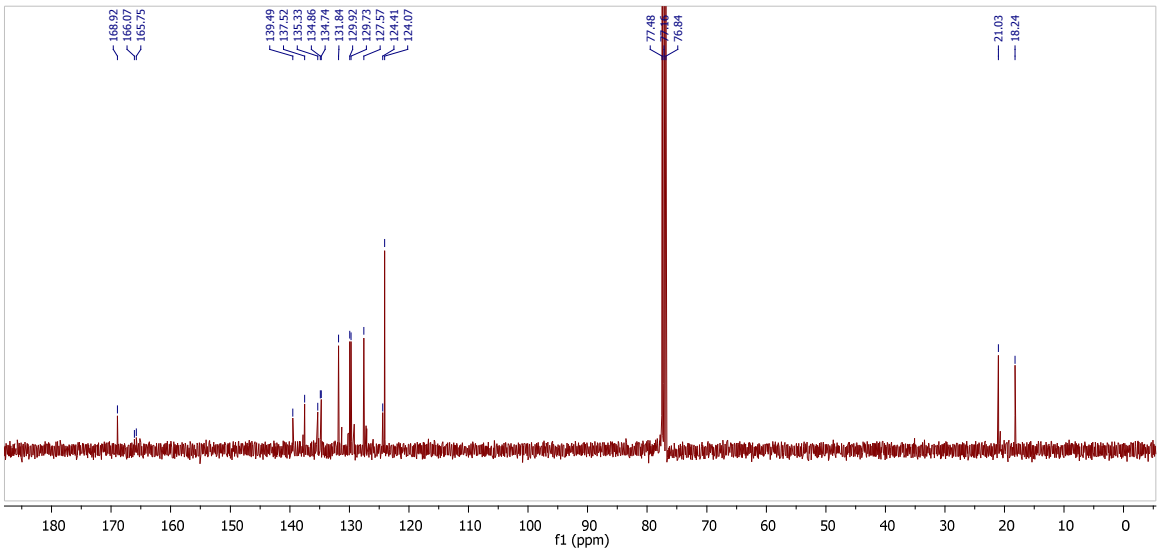


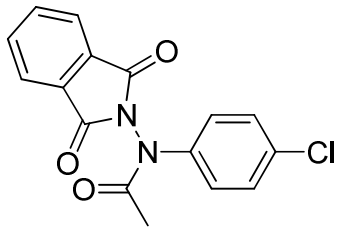




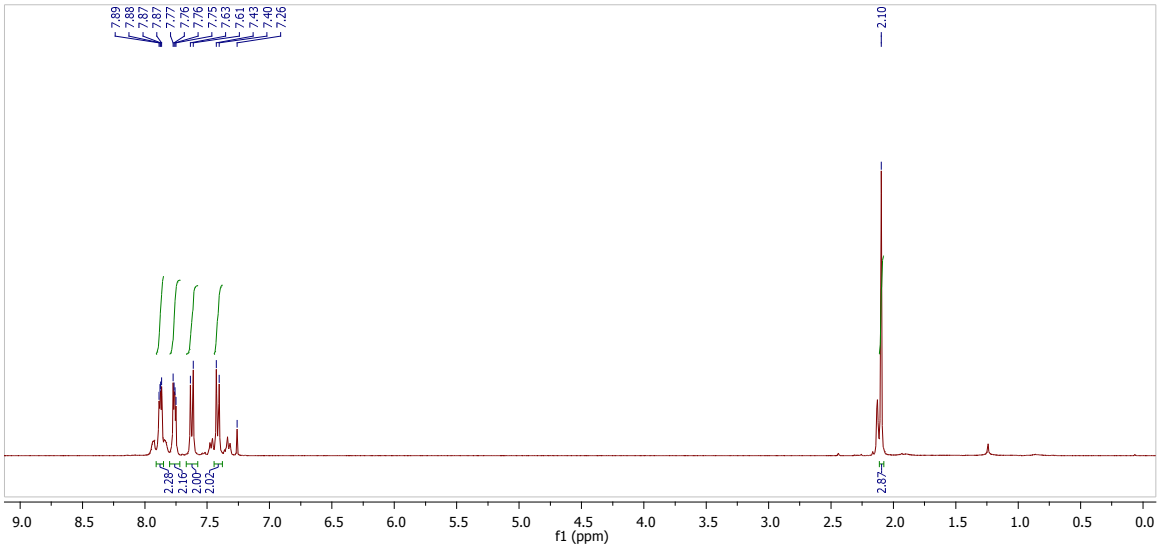
5d *ortho* isomer

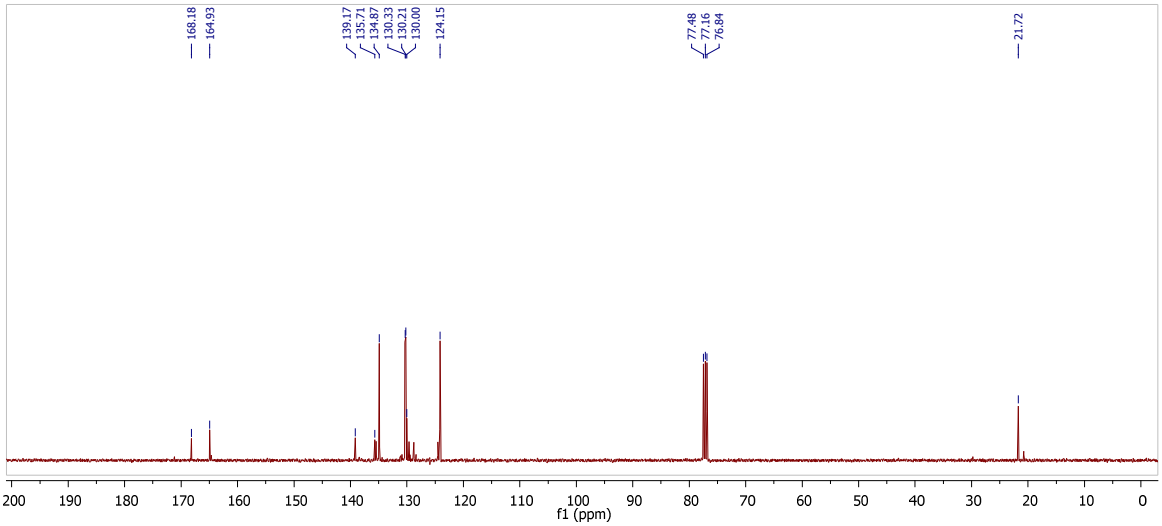


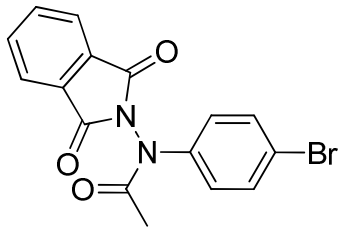




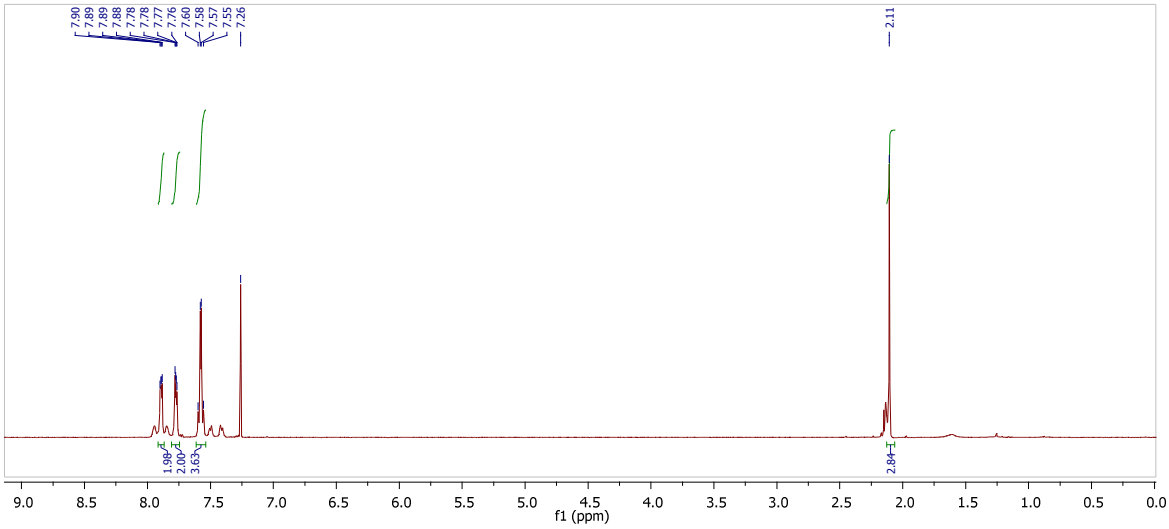
5g *para* isomer

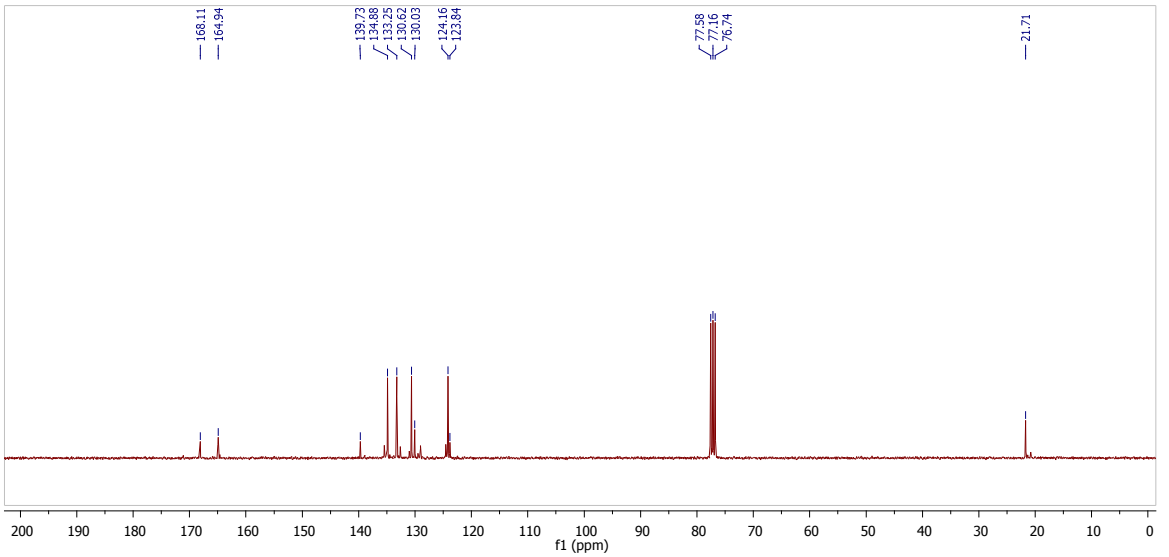


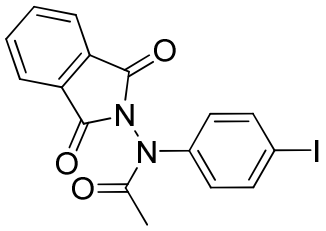




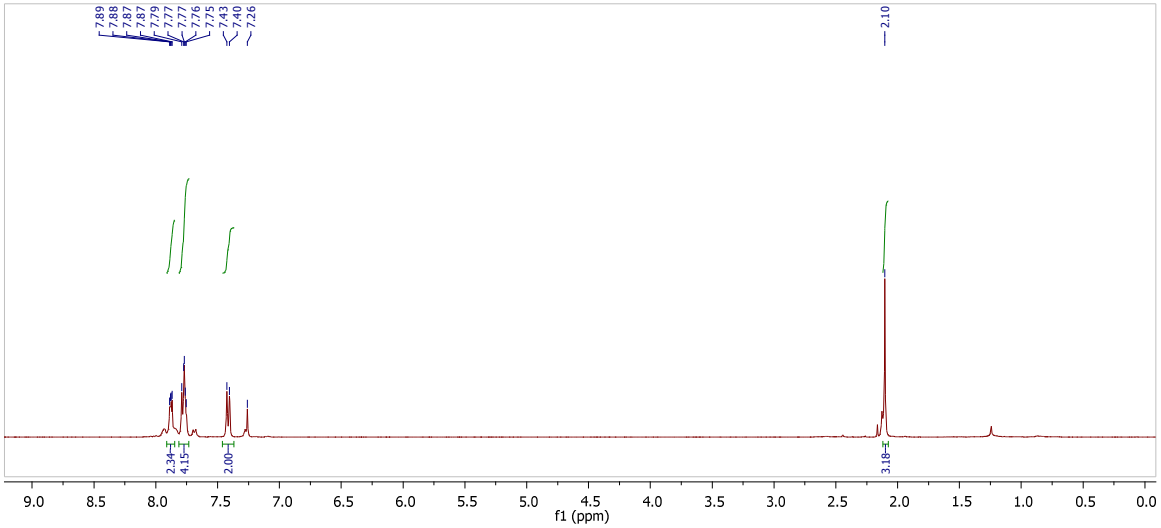
5h *para* isomer

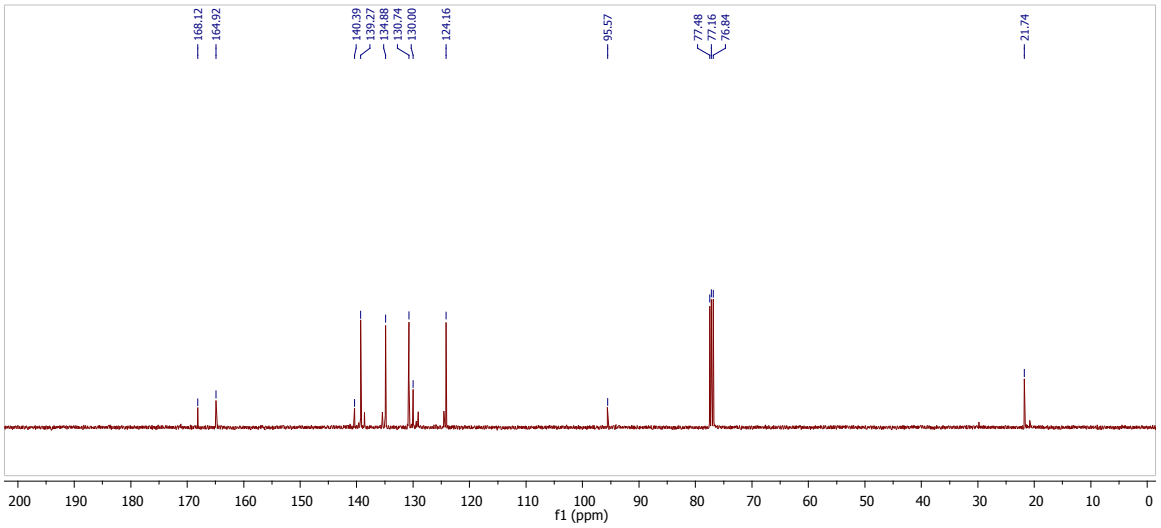


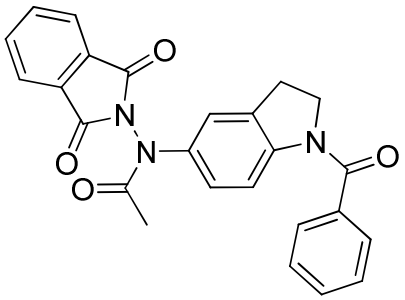




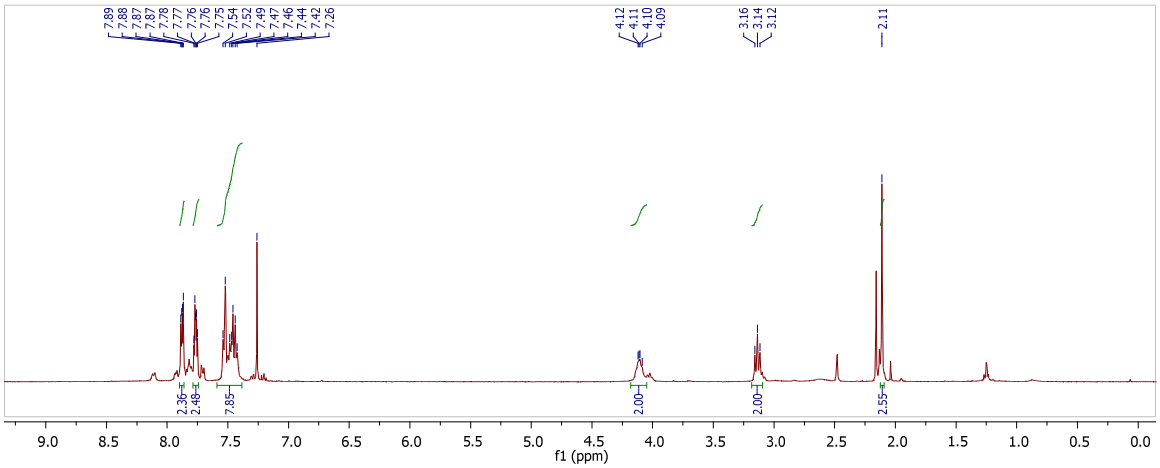
5i *para* isomer

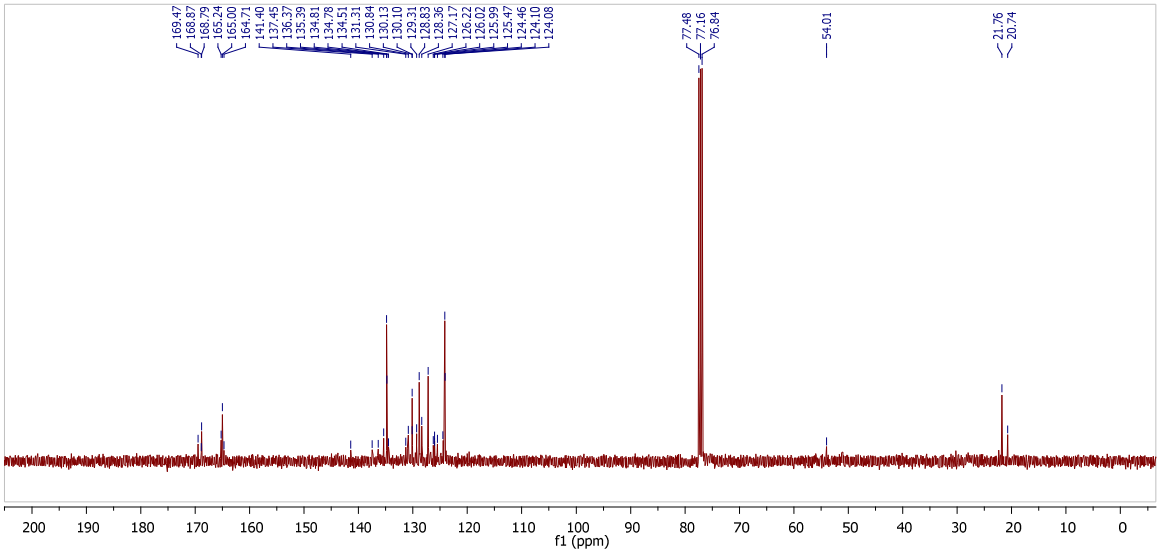






5n


7.89
7.88
7.87
7.87
7.78
7.77
7.76
7.76
7.75
7.54
7.52
7.49
7.47
7.46
7.44
7.42
7.26
4.12
4.11
4.10
4.09
3.16
3.14
3.12
2.11
2.36
2.48
7.85
2.00
2.00
2.55
f1 (ppm)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168.79
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164.71
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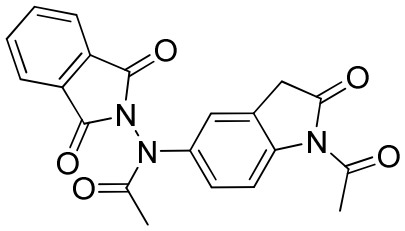
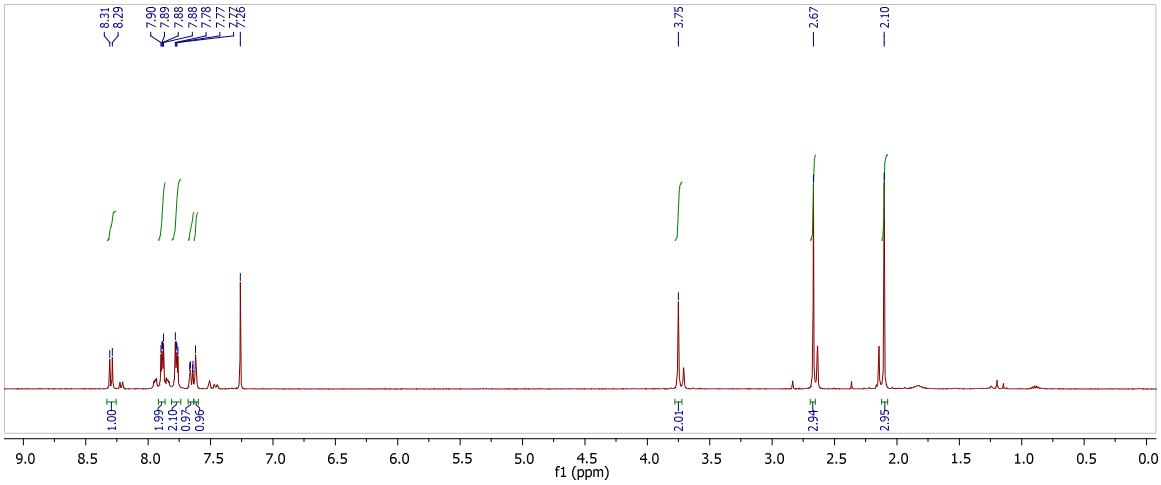
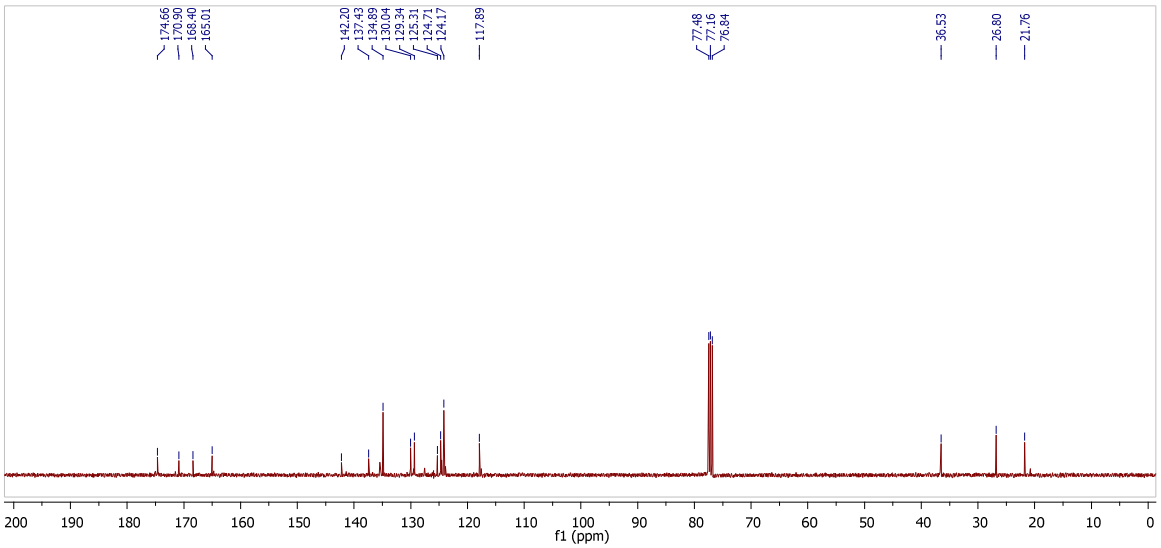
135.39
134.81
134.78
134.51
131.31
130.84
130.13
130.10
129.31
128.83
128.36
127.17
126.22
126.02
125.99
125.47
124.46
124.10
124.08
77.48
77.16
76.84
54.01
21.76
20.74
f1 (ppm)

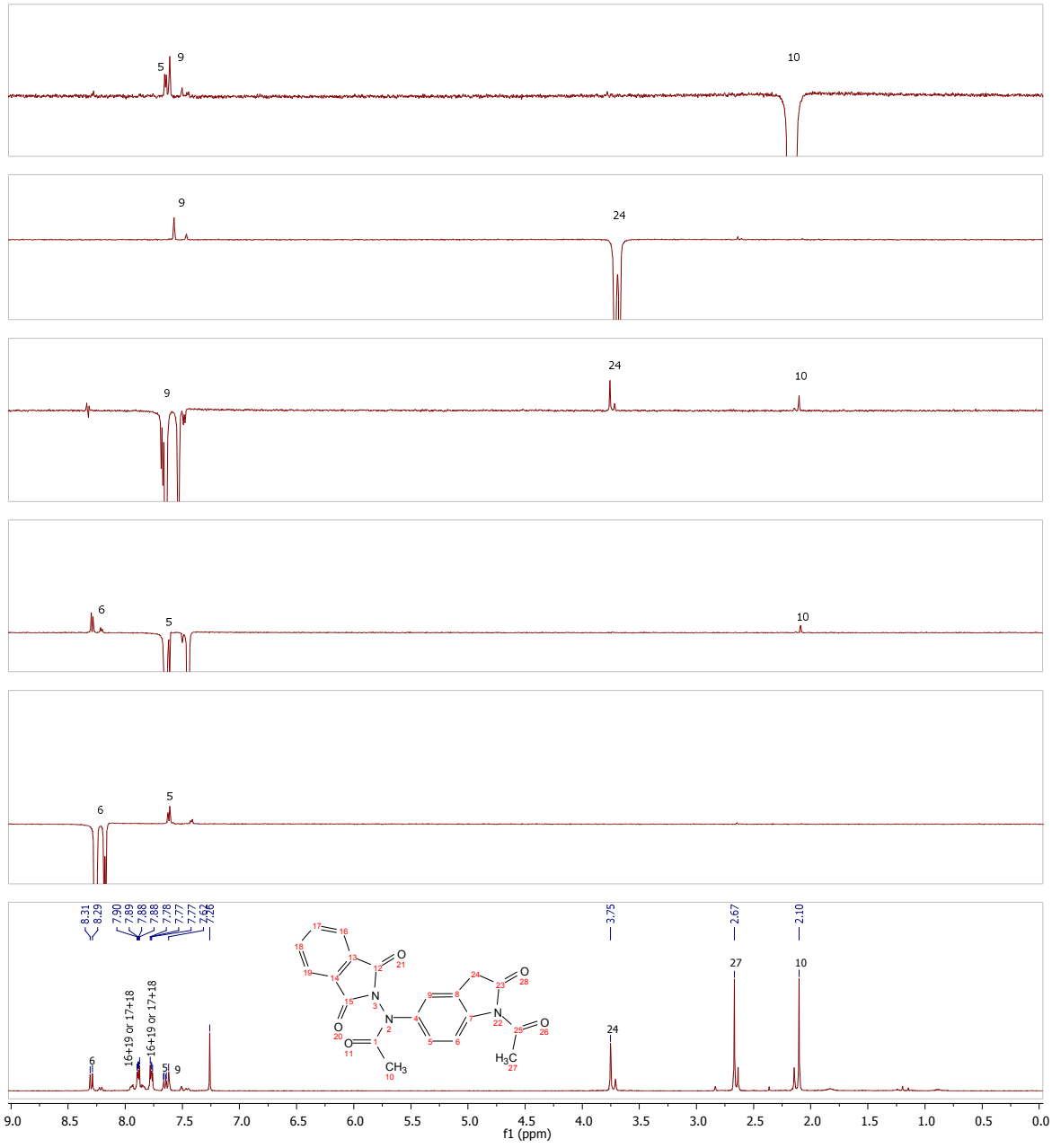


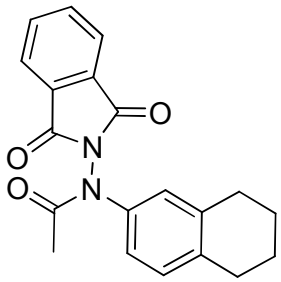
5o


8.31
8.29
7.90
7.89
7.88
7.88
7.78
7.77
7.77
7.26
3.75
2.67
2.10
1.00
1.99
2.10
0.97
0.96
2.01
2.94
2.95
f1 (ppm)

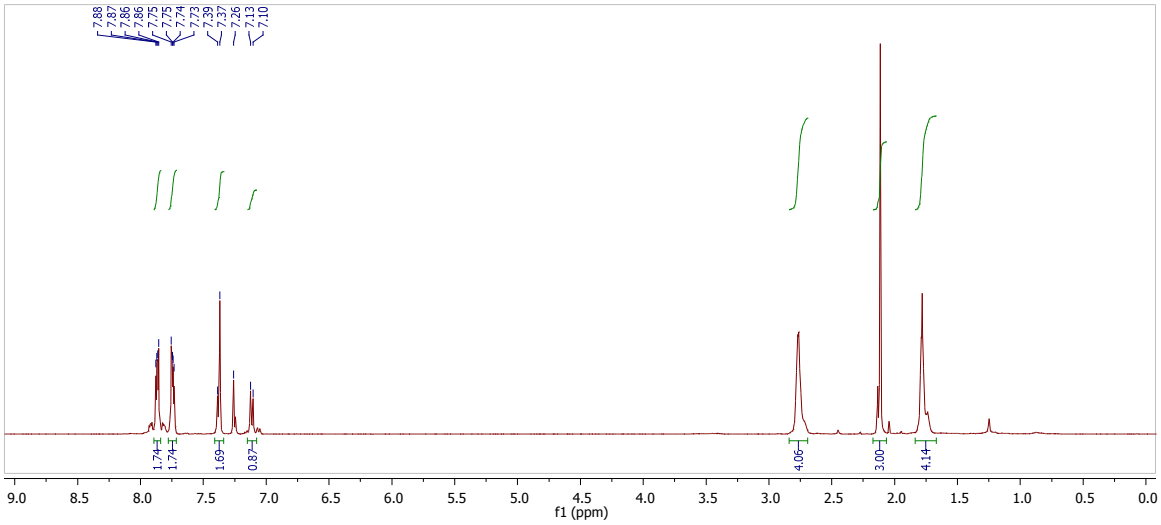

174.66
170.90
168.40
165.01
142.20
137.43
134.89
130.04
129.34
125.31
124.71
124.17
117.89
77.48
77.16
76.84
36.53
26.80
21.76
f1 (ppm)

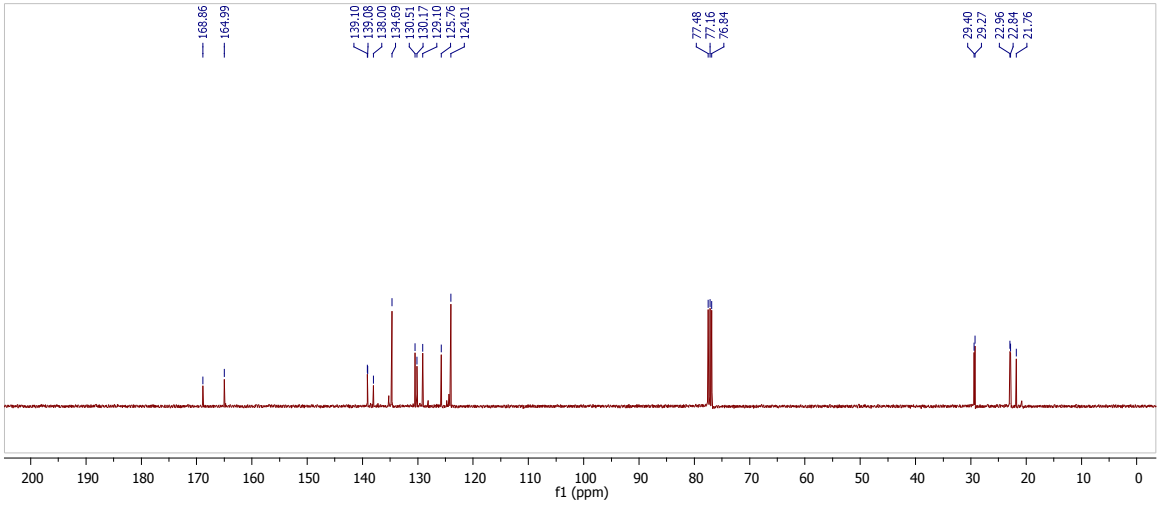
1D NOESY Spectra

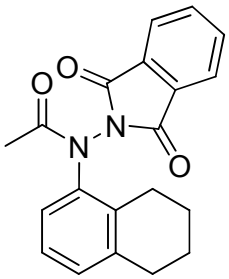




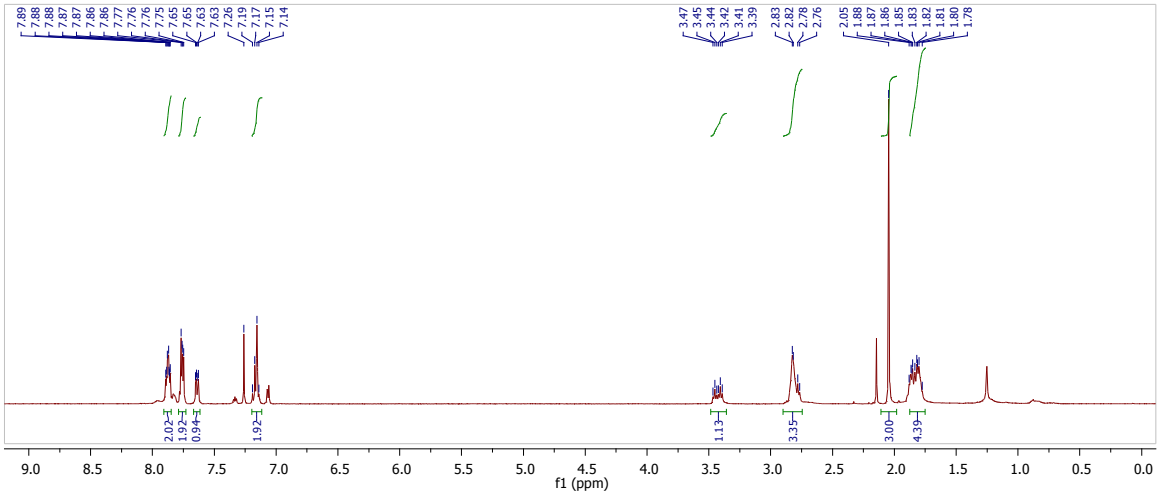
5p major isomer

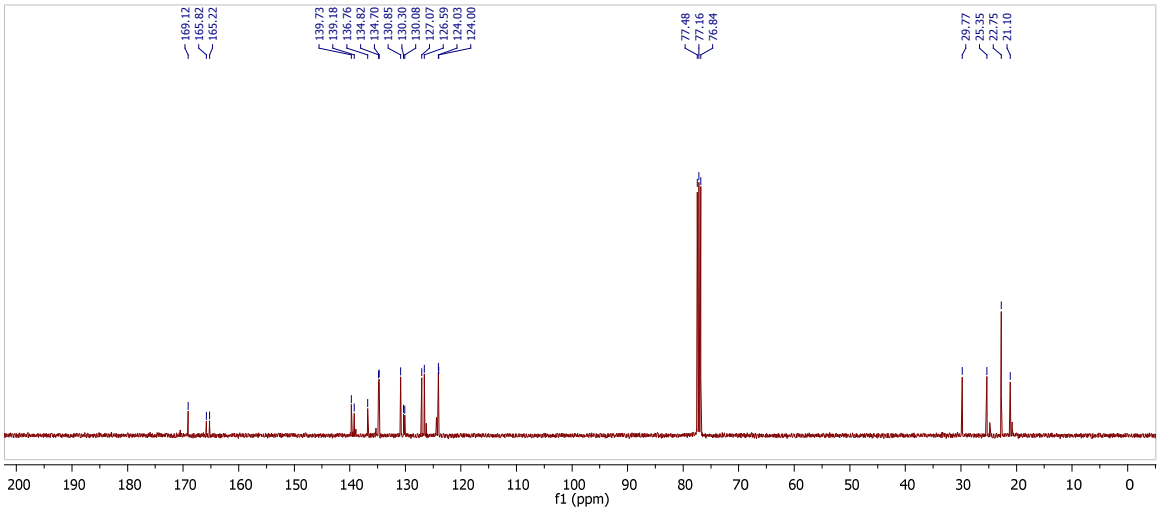


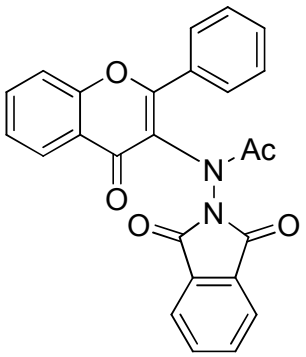




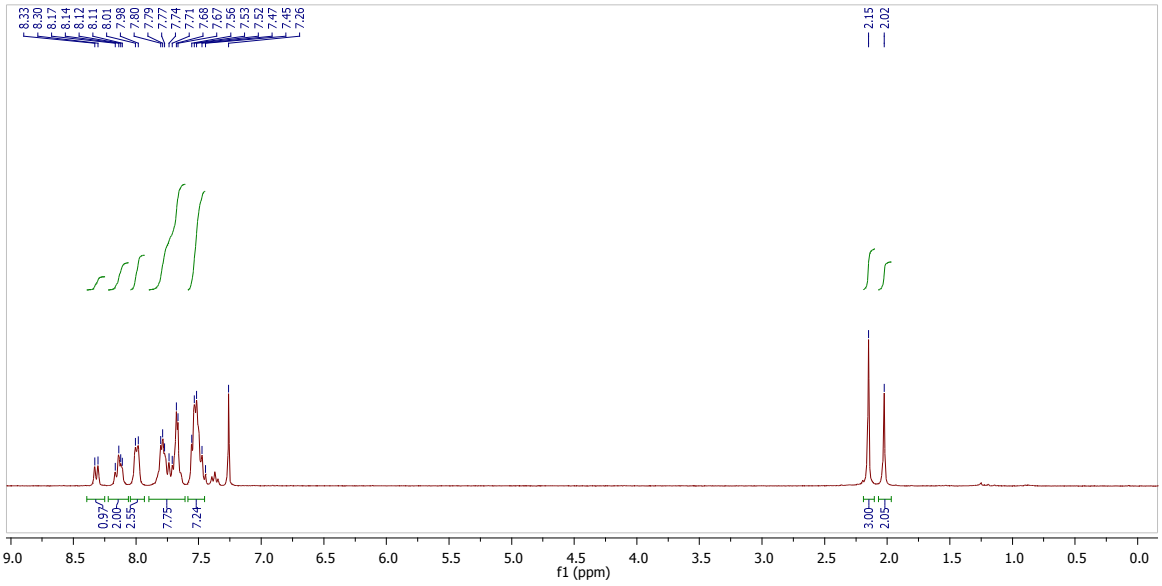
5p minor isomer

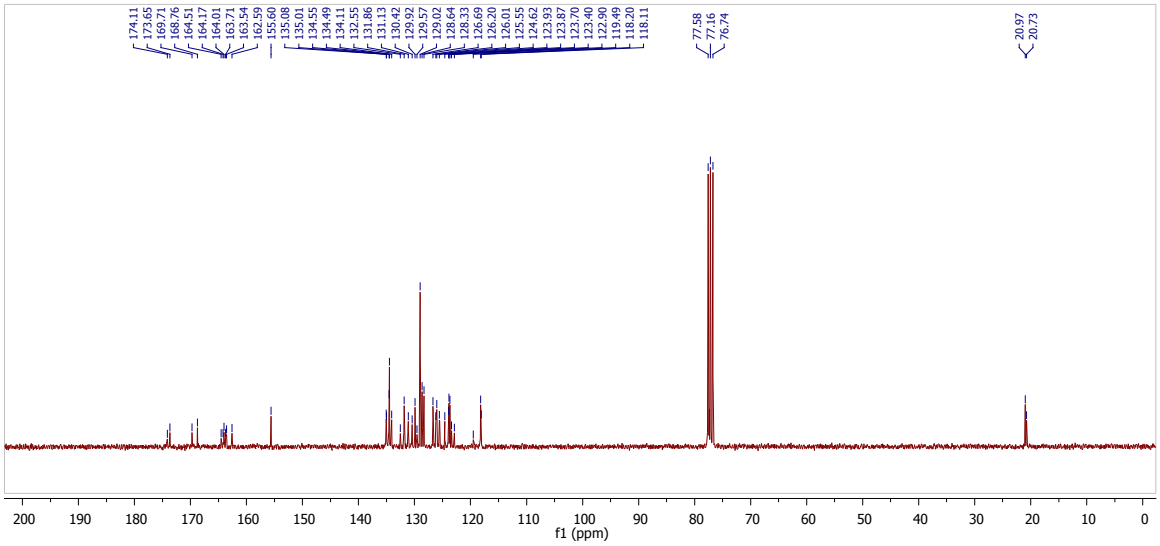


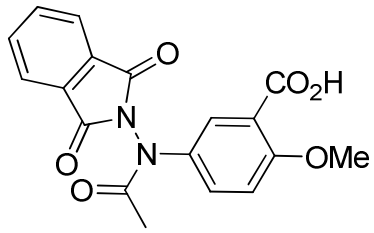



O
Ac
N
O
N
O
O

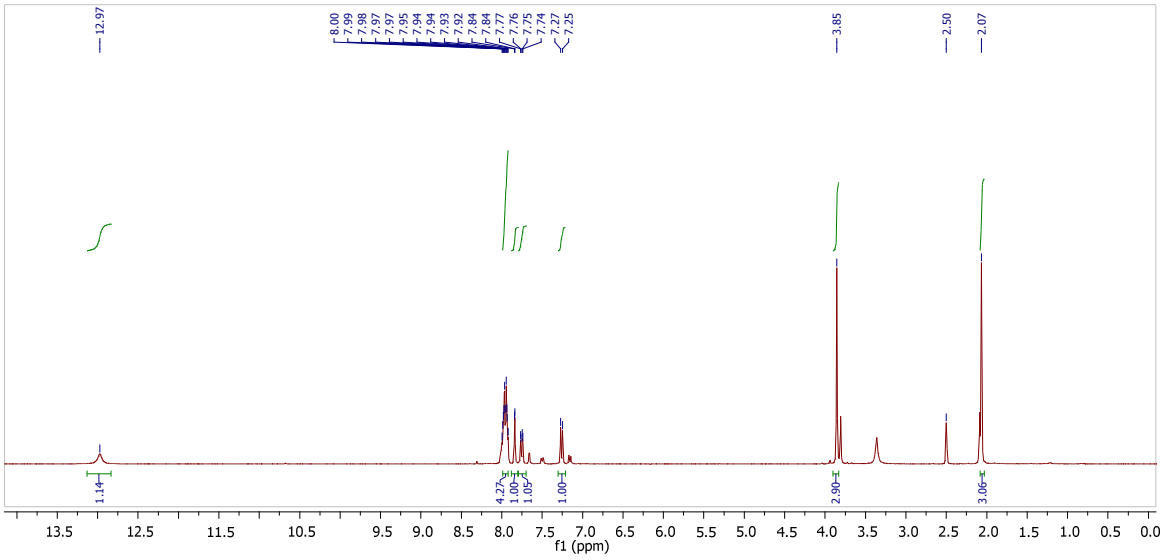
5s

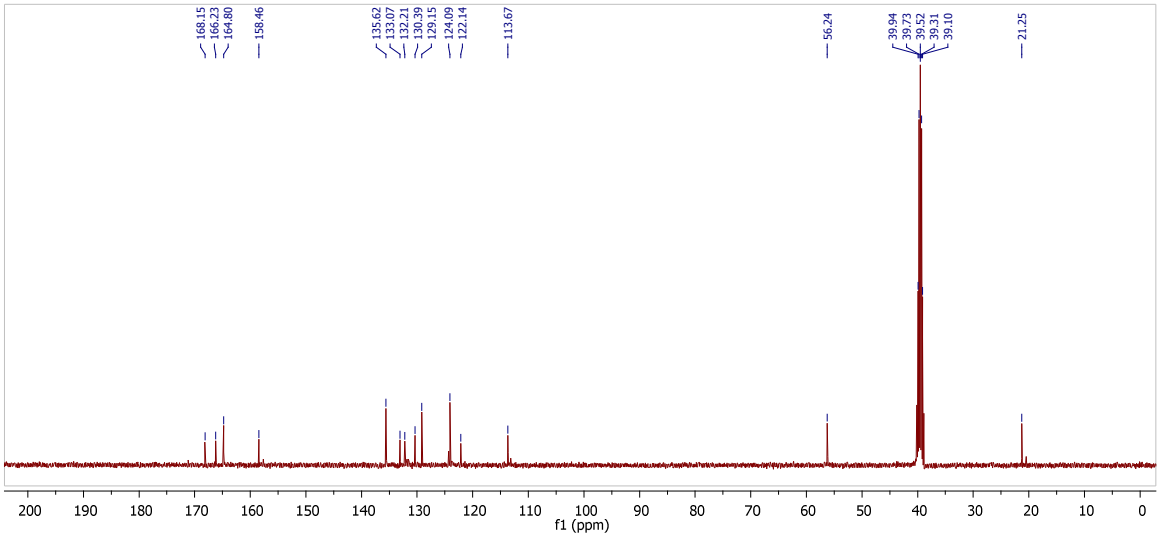

8.33
8.30
8.17
8.14
8.12
8.11
8.01
7.98
7.80
7.79
7.77
7.74
7.71
7.68
7.67
7.56
7.53
7.52
7.47
7.45
7.26
2.15
2.02
0.97
2.00
2.55
7.75
7.24
3.00
2.05
f1 (ppm)

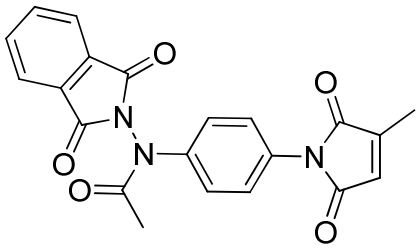

174.11
173.65
169.71
168.76
164.51
164.17
164.01
163.71
163.54
162.59
155.60
135.08
135.01
134.55
134.49
134.11
132.55
131.86
131.13
130.42
129.92
129.57
129.02
128.64
128.33
126.69
126.20
126.01
125.55
124.62
123.93
123.87
123.70
123.40
122.90
119.49
118.20
118.11
77.58
77.16
76.74
20.97
20.73
f1 (ppm)


O
N
N
O
O
CO2H
OMe

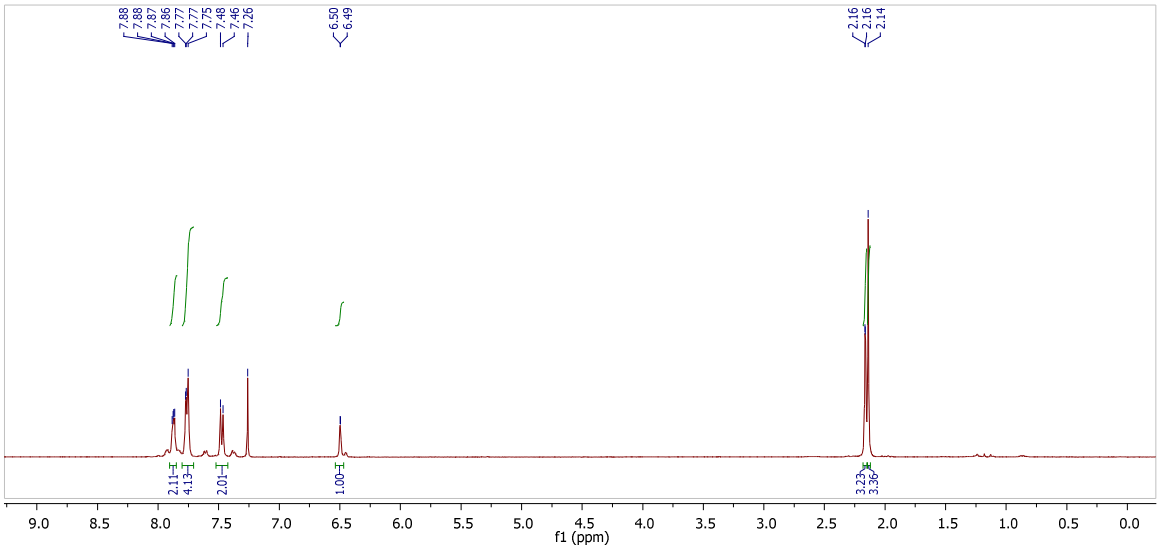
5m

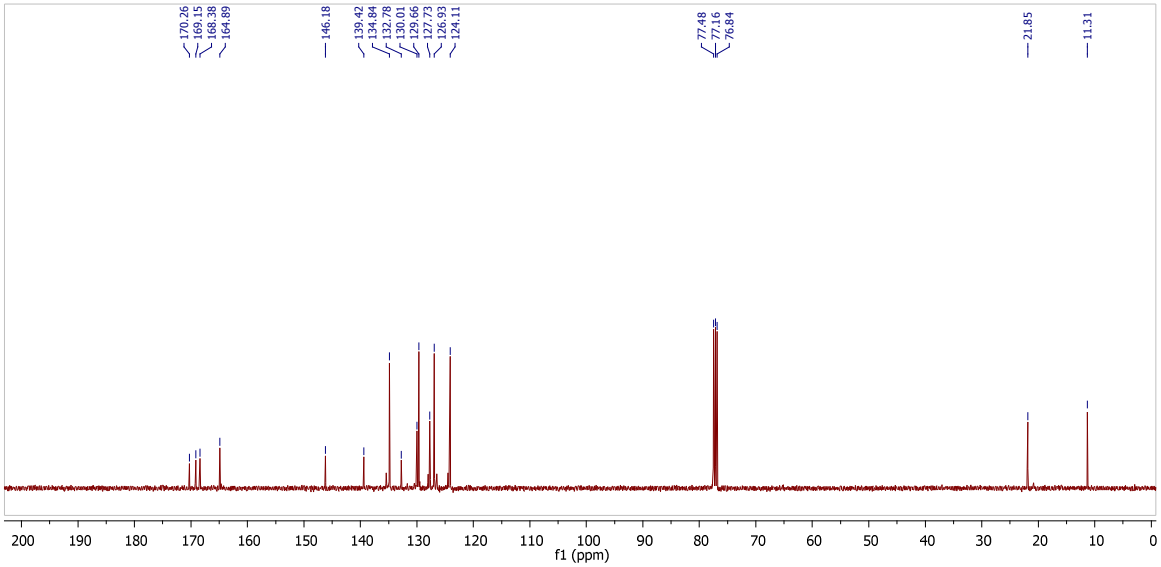

12.97
8.00
7.99
7.98
7.97
7.97
7.95
7.94
7.94
7.93
7.92
7.84
7.84
7.77
7.76
7.75
7.74
7.27
7.25
3.85
2.50
2.07
1.14
4.27
1.08
1.05
1.00
2.90
3.06
f1 (ppm)

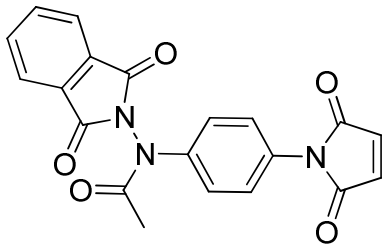

168.15
166.23
164.80
158.46
135.62
133.07
132.21
130.39
129.15
124.09
122.14
113.67
56.24
39.94
39.73
39.52
39.31
39.10
21.25
f1 (ppm)



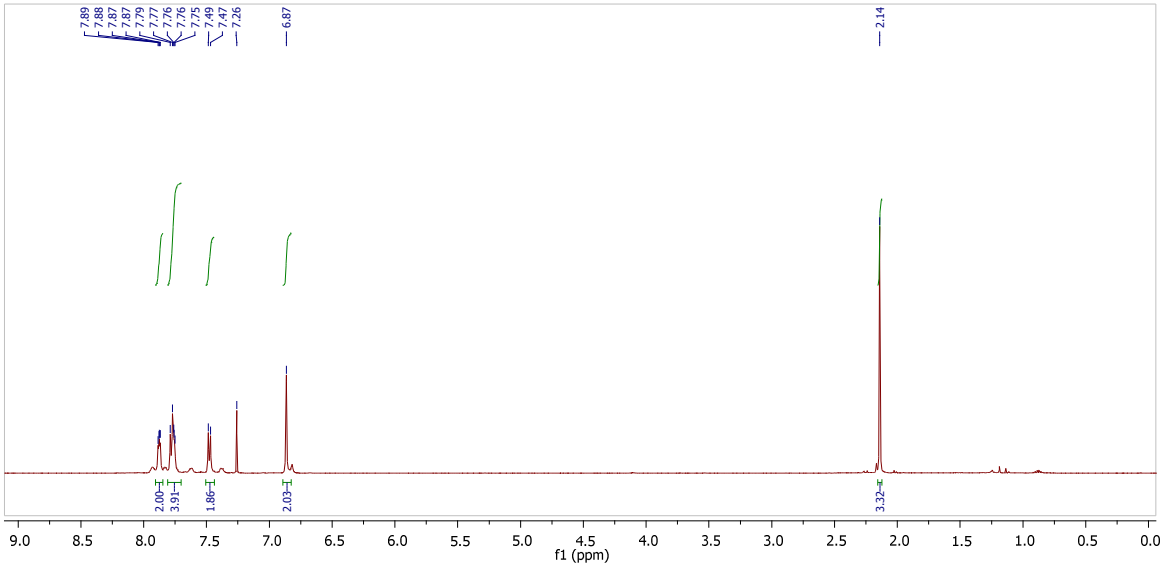
5l

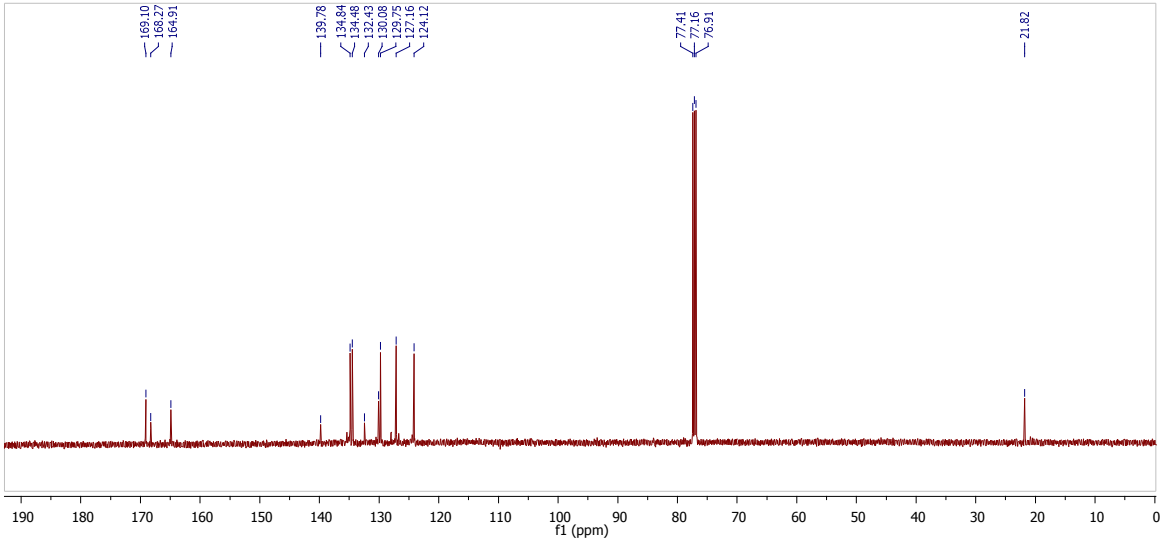

7.88
7.88
7.87
7.86
7.77
7.77
7.75
7.48
7.46
7.26
6.50
6.49
2.16
2.16
2.14
2.11
4.13
2.01
1.00
3.23
3.36
f1 (ppm)

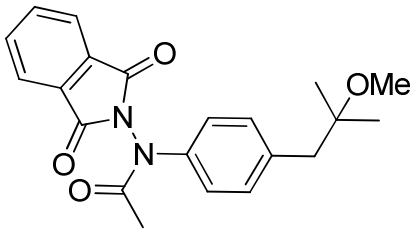

170.26
169.15
168.38
164.89
146.18
139.42
134.84
132.78
130.01
129.66
127.73
126.93
124.11
77.48
77.16
76.84
21.85
11.31
f1 (ppm)



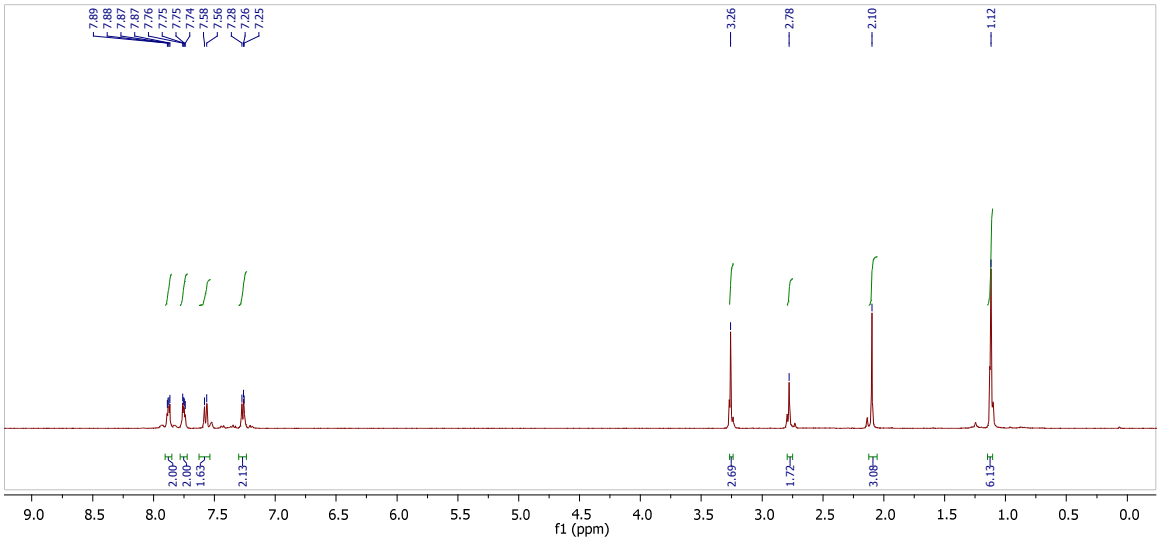
5k

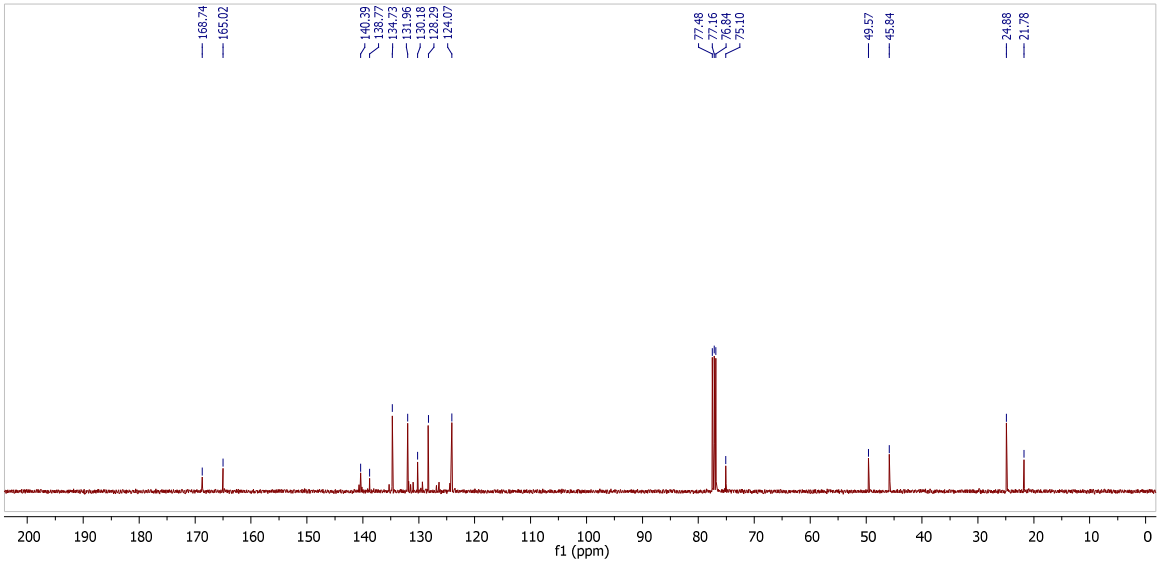

7.89
7.88
7.87
7.87
7.79
7.77
7.76
7.76
7.75
7.49
7.47
7.26
6.87
2.14
2.00
3.91
1.86
2.03
3.32
f1 (ppm)

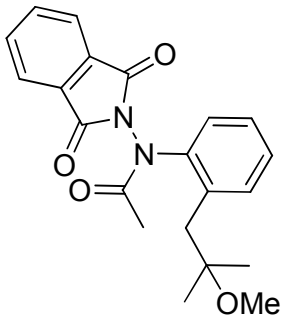

169.10
168.27
164.91
139.78
134.84
134.48
132.43
130.08
129.75
127.16
124.12
77.41
77.16
76.91
21.82
f1 (ppm)



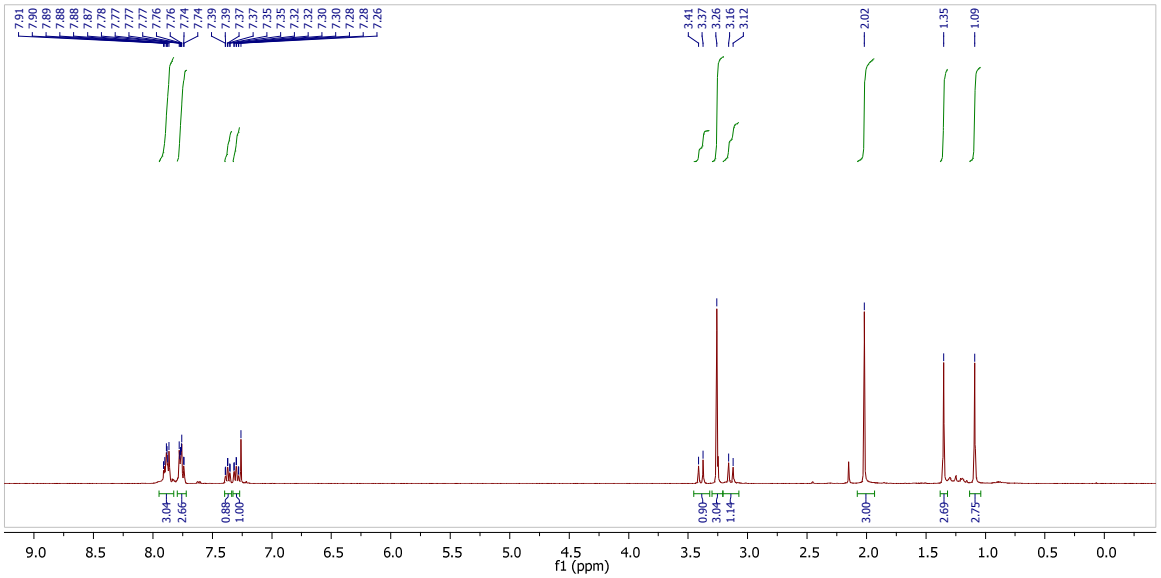
5j *para* isomer

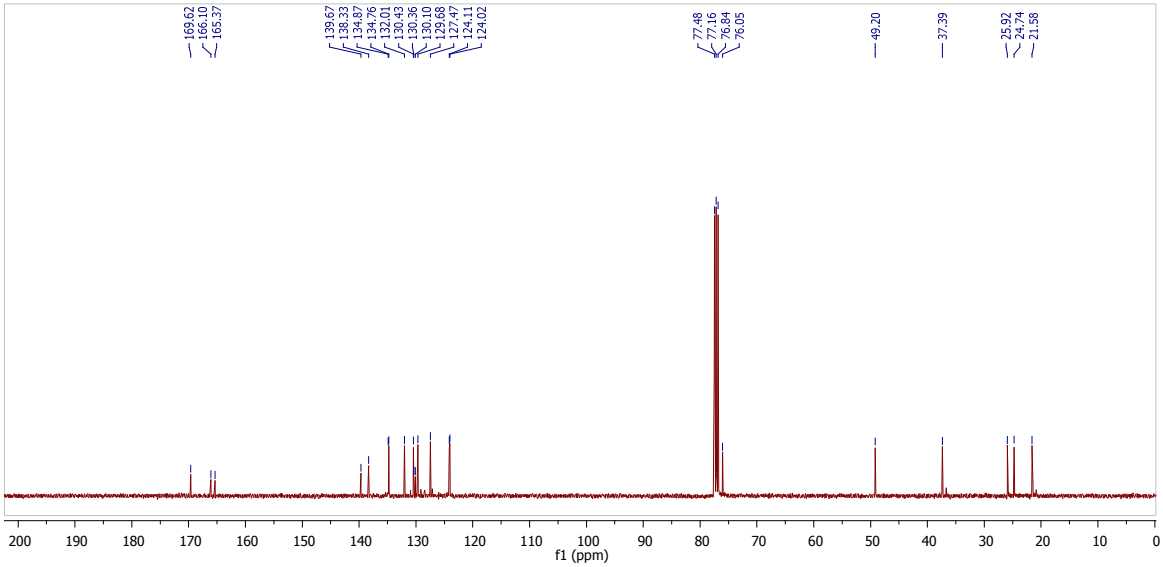


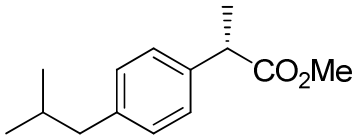


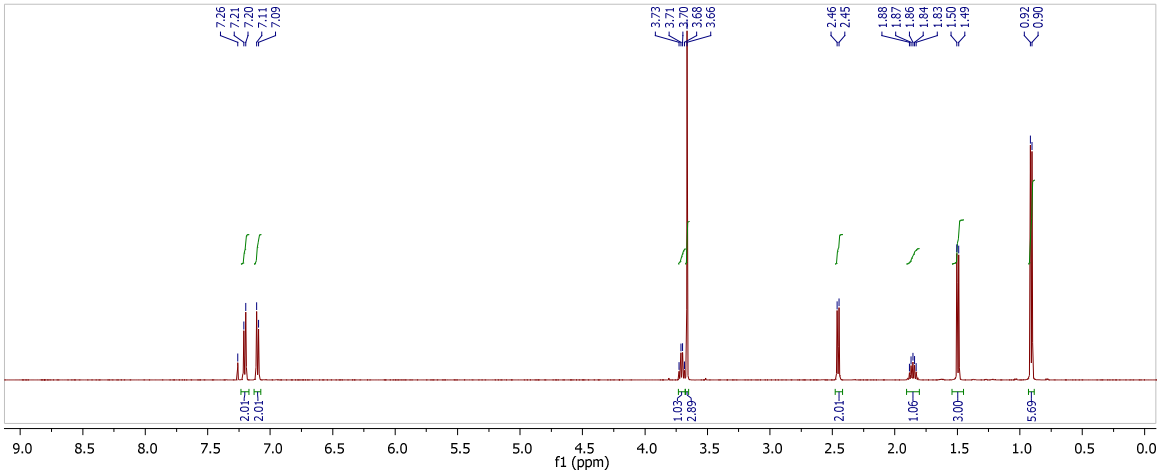


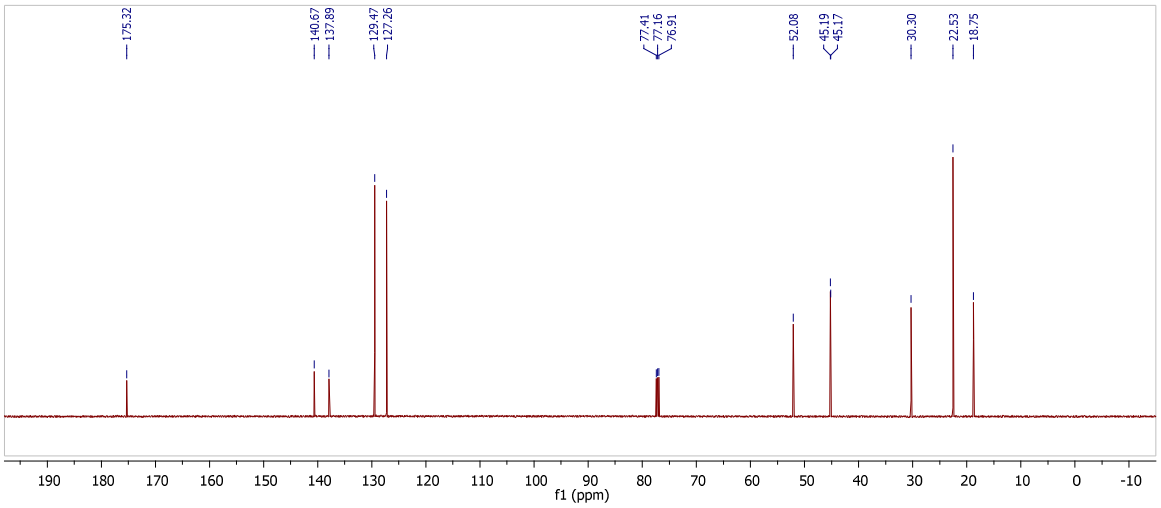
5j *ortho* isomer

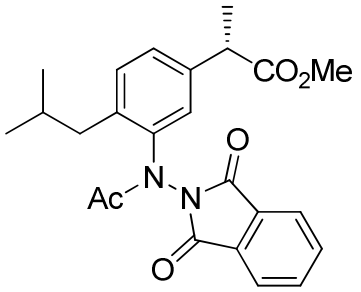




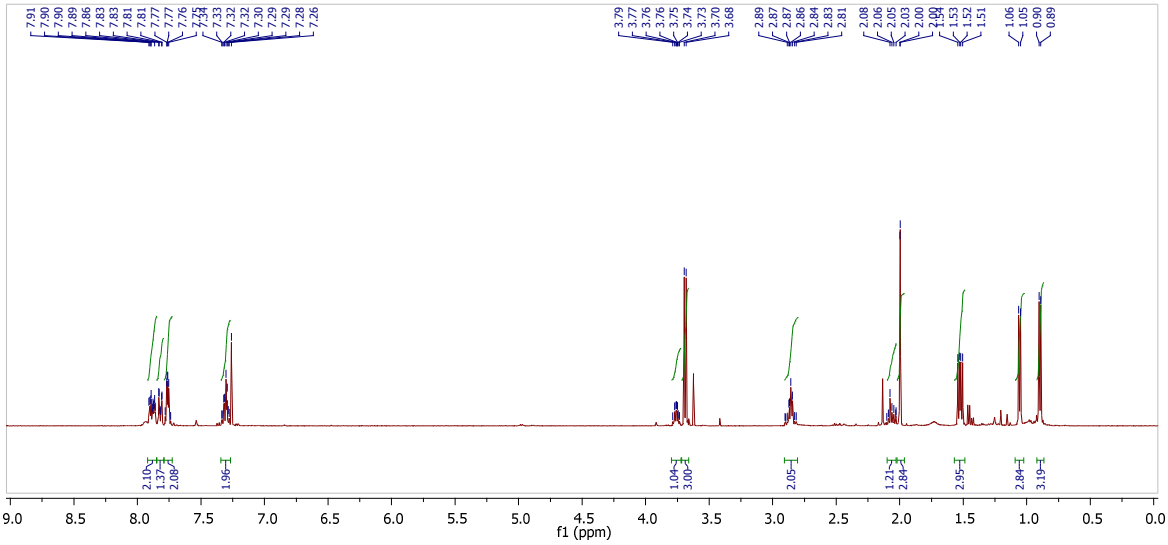

CO2Me

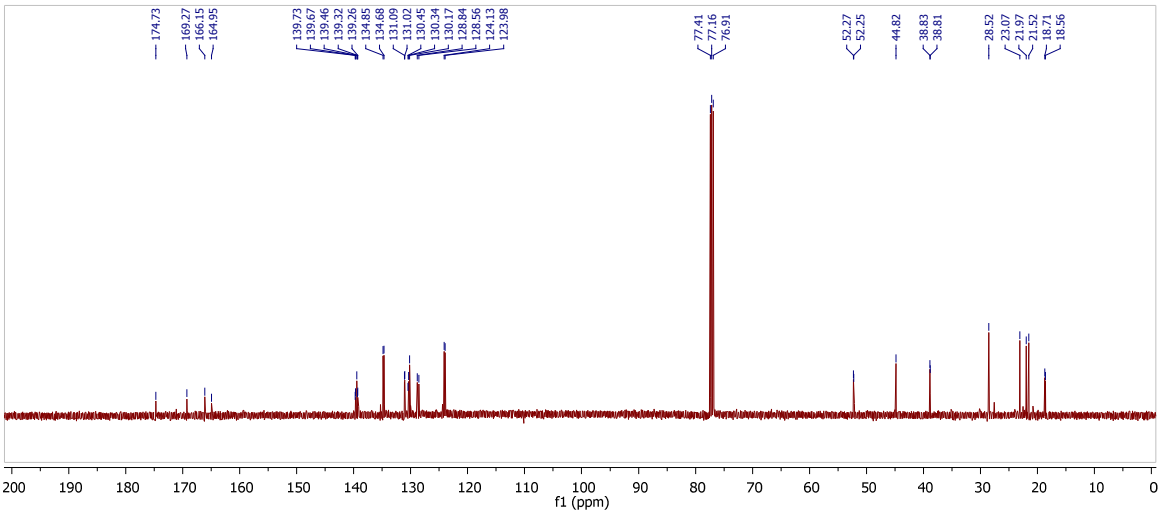

7.26
7.21
7.20
7.11
7.09
3.73
3.71
3.70
3.68
3.66
2.46
2.45
1.88
1.87
1.86
1.84
1.83
1.50
1.49
0.92
0.90
2.01
2.01
1.03
2.89
2.01
1.06
3.00
5.69
f1 (ppm)

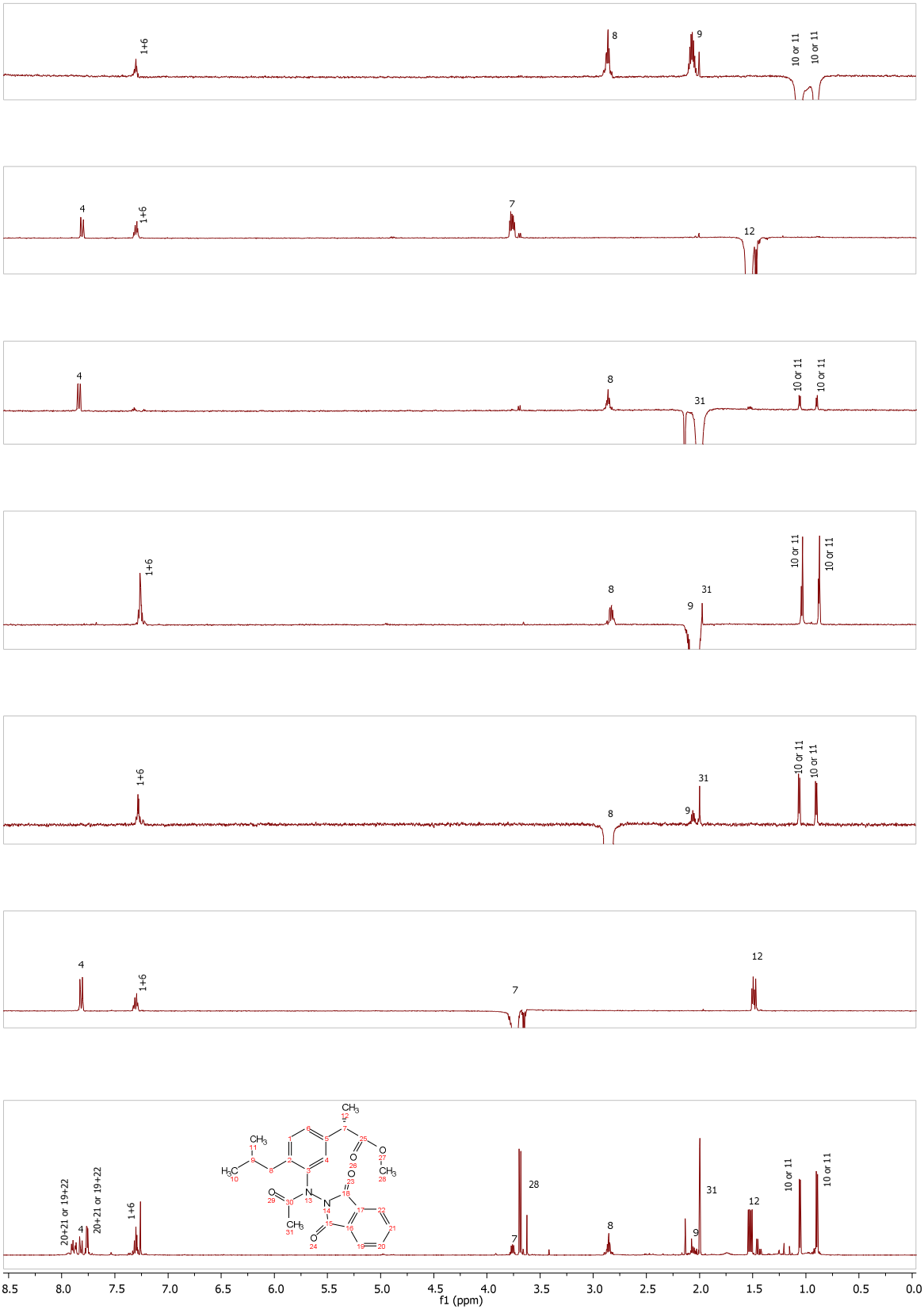

175.32
140.67
137.89
129.47
127.26
77.41
77.16
76.91
52.08
45.19
45.17
30.30
22.53
18.75
f1 (ppm)

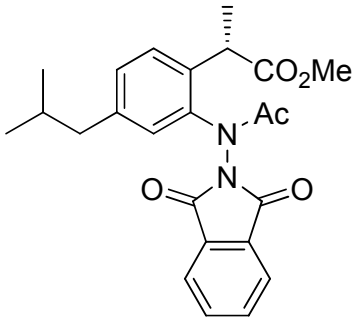


5t major isomer

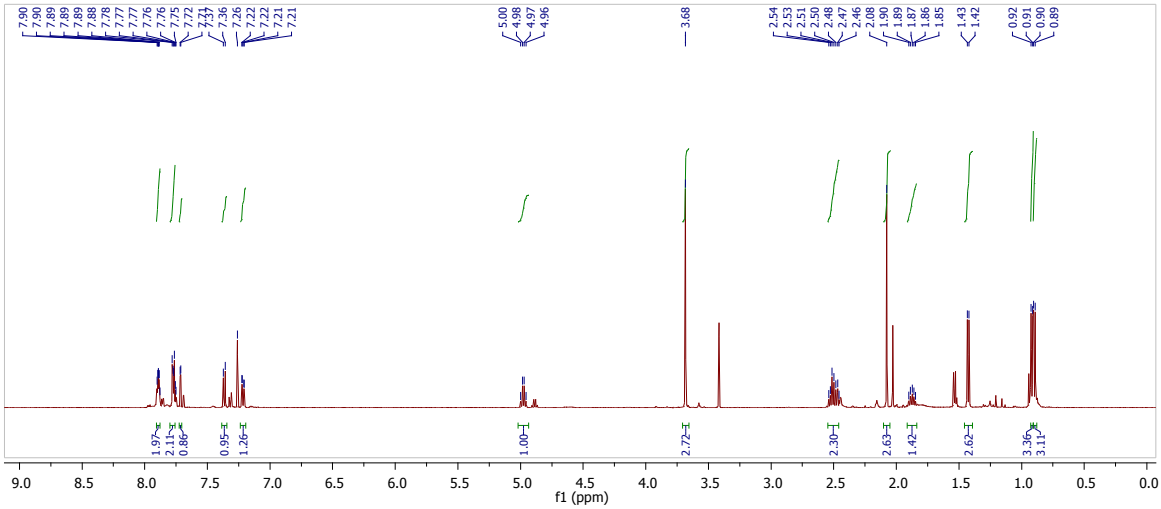


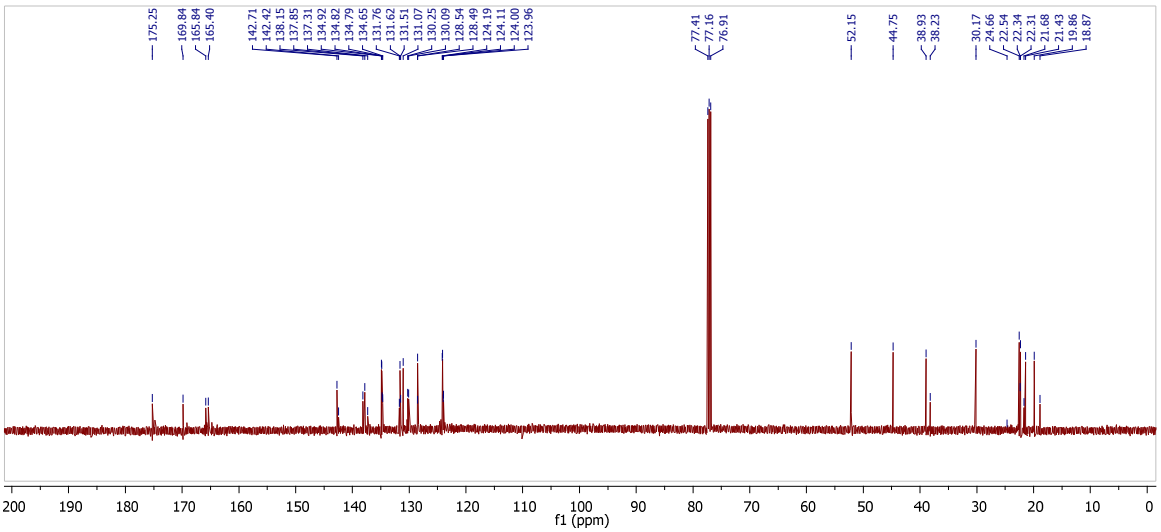


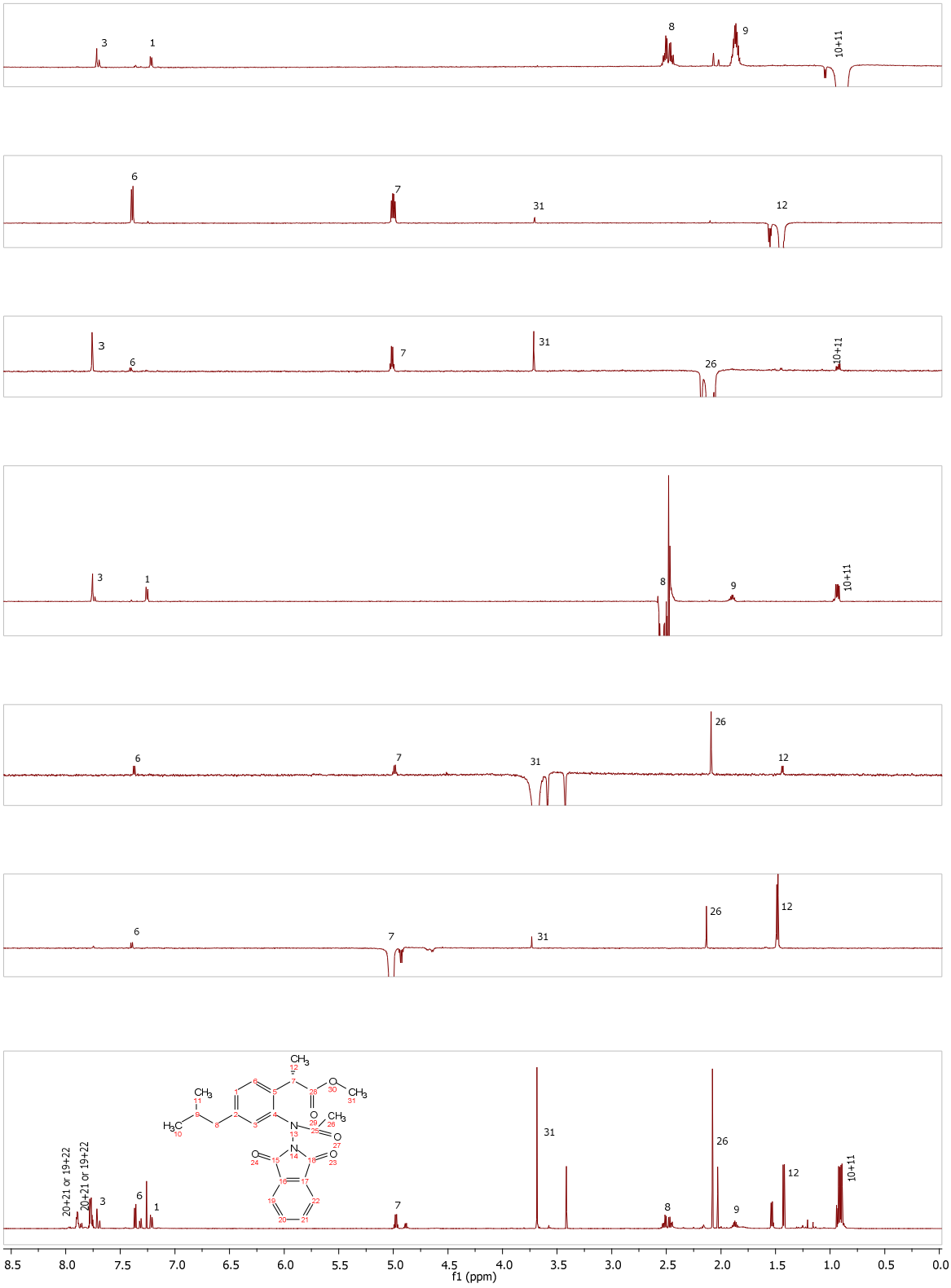

1+6
8
9
10 or 11
10 or 11
4
1+6
7
12
4
8
31
10 or 11
10 or 11
1+6
8
9
31
10 or 11
10 or 11
1+6
8
9
31
10 or 11
10 or 11
4
1+6
7
12
20+21 or 19+22
4
20+21 or 19+22
1+6
7
28
8
9
31
12
10 or 11
10 or 11
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

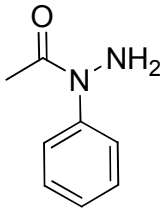


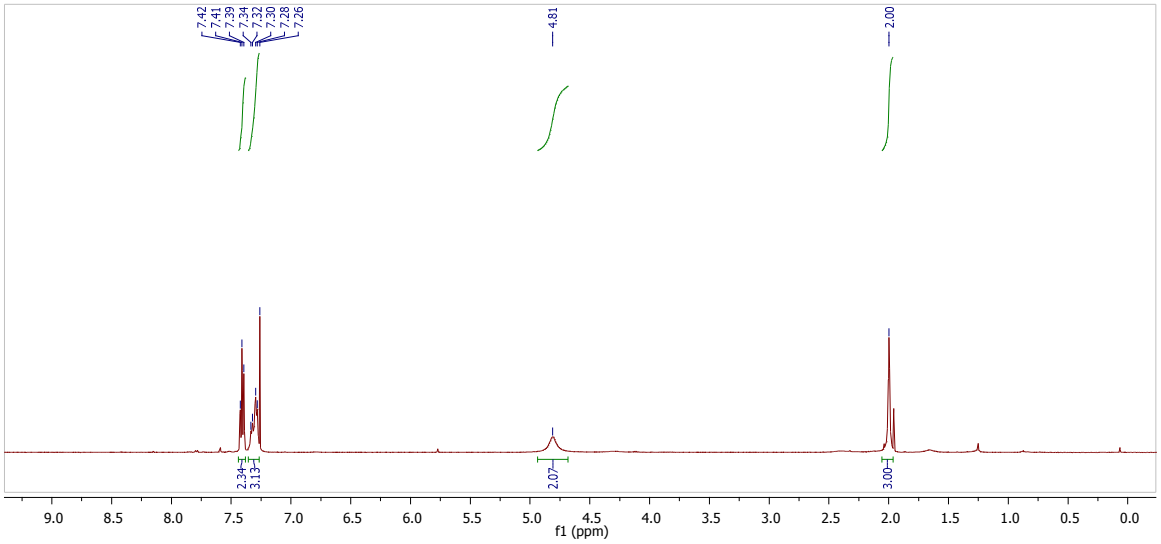
5t minor isomer

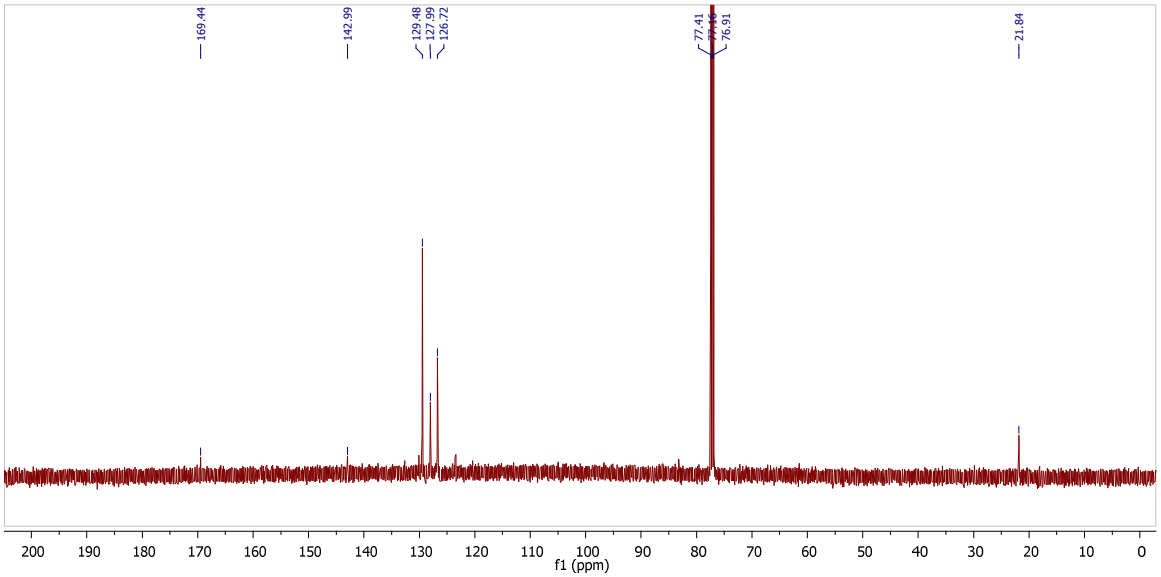





3
1
8
9
10+11
6
7
31
12
3
6
7
31
26
10+11
3
1
8
9
10+11
6
7
31
26
12
6
7
31
26
12
20+21 or 19+22
20+21 or 19+22
3
6
1
7
31
8
26
9
12
10+11
f1 (ppm)


O
N
NH2


7.42
7.41
7.39
7.34
7.32
7.30
7.28
7.26
4.81
2.00
2.34
3.13
2.07
3.00
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
f1 (ppm)


169.44
142.99
129.48
127.99
126.72
77.41
77.16
76.91
21.84
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)