Practical Synthesis of Phthalimides and Benzamides by a Multicomponent Reaction Involving Arynes, Isocyanides and CO_2/H_2O

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1. General Information

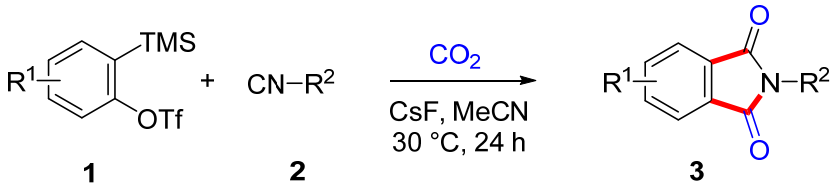
Unless otherwise specified, all reactions were carried out under an atmosphere of argon in flame-dried reaction vessels with Teflon screw caps. For the synthesis of phthalimides, all reactions were carried out under an atmosphere of CO_2 (balloon). THF was purified by distillation over Na-benzophenone and was transferred under argon. Dry CH_3CN was purchased from commercial sources and stored under argon over 4 Å molecular sieves. 18-Crown-6 was recrystallized from dry CH_3CN and KF was dried by heating at 110 °C for 12 h and left to cool under argon. CsF was purchased from Aldrich and dried by heating at 110 °C for 12 h and left to cool under argon. All the isocyanides were purchased from either Aldrich or Alfa Aesar and used without further purification. The 2-(trimethylsilyl)phenyl trifluoromethanesulfonate **1a** and all other symmetric and unsymmetric aryne precursors were synthesized following literature procedure.[1]

Analytical thin layer chromatography was performed on TLC Silica gel 60 F254. Visualization was accomplished with short wave UV light or $KMnO_4$ staining solutions followed by heating. Chromatography was performed on silica gel (230-400 mesh) by standard techniques eluting with solvents as indicated.

All compounds were fully characterized. ^1H and ^{13}C NMR spectra were recorded on Bruker AV 400, in solvents as indicated. Chemical shifts (δ) are given in ppm. The residual solvent signals were used as references and the chemical shifts converted to the TMS scale ($CDCl_3$: δH = 7.26 ppm, δC = 77.16 ppm). Infra-red spectra were recorded on a Bruker Alpha-E Infra-red Spectrophotometer. The wave numbers (n) of recorded IR-signals are quoted in cm^{-1}. HRMS data were recorded either on a Thermo Scientific Q-Exactive, Accela 1250 pump or using Waters SYNAPT G2 High Definition Mass Spectrometry System.

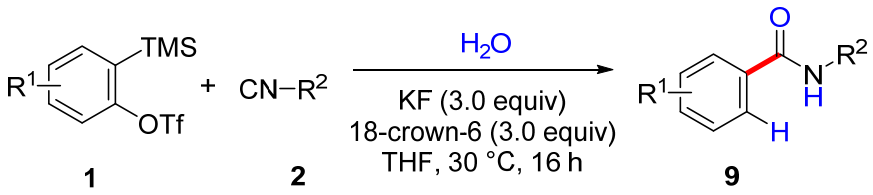
[1] (a) Sato, Y.; Tamura, T.; Kinbara, A.; Morib, M. *Adv. Synth. Catal.* **2007**, *349*, 647. (b) Peña, D.; Cobas, A.; Pérez, D.; Guitián, E. *Synthesis* **2002**, 1454.

2. General Procedure for the MCR Involving Isocyanides, Arynes and CO_2



To a flame-dried screw-capped test tube equipped with a magnetic stir bar was added dry CsF (0.228 g, 1.5 mmol). Then the screw-capped tube was evacuated and backfilled with argon, and CH_3CN (2.0 mL) was added under argon atmosphere. To the stirring solution were added isocyanide **2** (0. 5 mmol), and aryne precursor **1** (0.75 mmol) under argon. Then the reaction mixture was purged with CO_2 (balloon), and subsequently the mixture was stirred at 30 °C for 24 h under an atmosphere of CO_2 (balloon). When TLC control showed the completion of the reaction (after 24 h) the reaction was stopped (The reactions carried out with aromatic isocyanides did not work well at 30 °C. However, the reactivity was improved when the reaction was carried out at 60 °C for 6 h). The solvent was evaporated and the crude residue was purified by column chromatography on silica gel to get the corresponding phthalimide derivative **3** in moderate to excellent yields.

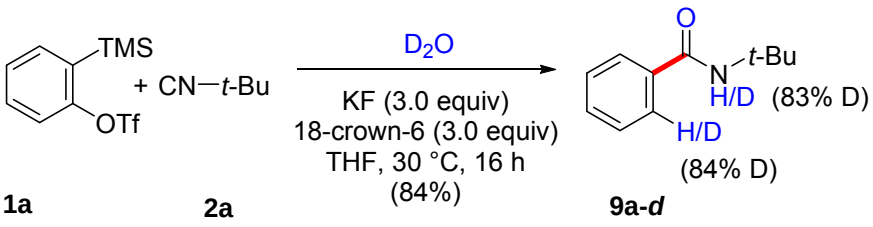
3. General Procedure for the MCR Involving Isocyanides, Arynes and H_2O



1 **2** **9**

To a flame-dried screw-capped test tube equipped with a magnetic stir bar was added 18-crown-6 (0.396 g, 1.5 mmol) and KF (1.5 mmol). Then the screw-capped tube was evacuated and backfilled with argon. The mixture was dissolved in THF (2.0 mL) under argon atmosphere and then the mixture was kept stirring at 30 °C. To this stirring solution was added isocyanide **2** (0.5 mmol) followed by the aryne precursor **1** (0.75 mmol). To this reaction mixture was added H_2O (1.0 mmol) after 10 minutes. Then the reaction mixture was kept stirring for 16 h. When TLC control showed the completion of the reaction (after 16 h) the reaction was stopped. The solvent was evaporated and the crude residue was purified by column chromatography on silica gel to afford the corresponding benzamide derivative **9** in good to excellent yields.

4. Reaction of Isocyanides, and Arynes in D_2O

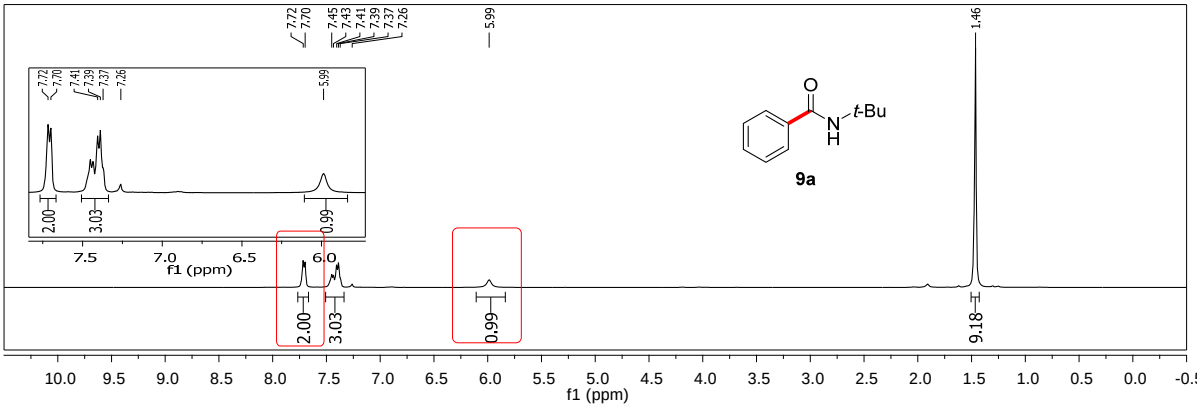


1a **2a** **9a-*d***

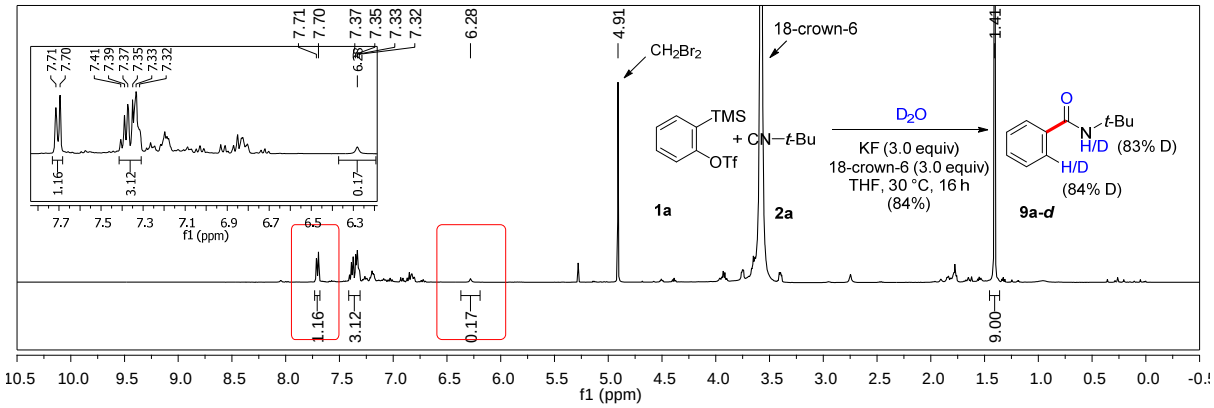
To a flame-dried screw-capped test tube equipped with a magnetic stir bar was added 18-crown-6 (0.198 g, 0.60 mmol) and KF (0.044 g, 0.60 mmol). Then the screw-capped tube was evacuated and backfilled with argon. The mixture was dissolved in THF (1.0 mL) under argon atmosphere. To the resulting stirring solution at 30 ^{o}C was added *tert*-butyl isocyanide **2a** (0.021 g, 28 µL, 0.25 mmol), 2-(trimethylsilyl)phenyl trifluoromethane sulfonate **1a** (0.112 g, 91 µL 0.375 mmol). To this reaction mixture was added D_2O (9.0 µL, 0.5 mmol) after 10 minutes. Then the mixture was then stirred at 30 °C for 16 h. The reaction mixture was diluted with CH_2Cl_2 (2.0 mL) and filtered through a short pad of silica gel and eluted with CH_2Cl_2 (2x10.0 mL). The solvent was evaporated to obtain the crude product whose yield was determined by ^{1}H NMR analysis using CH_2Br_2 (18.0 µL, 0.25 mmol) as the internal standard. ^{1}H-NMR spectrum analysis of crude product showed 84% deuterium incorporation at the 2-position of the ring and 83% deuterium incorporation was observed at the amide functionality.

*The incorporation of deuterium at the 2-position of ring and at the amide functional group indicates that the initially formed aryl anion intermediate **5** (Scheme 4 of the manuscript) is first protonated and the subsequent hydrolysis of the resultant iminium species results to the formation of the benzamide **9**.*

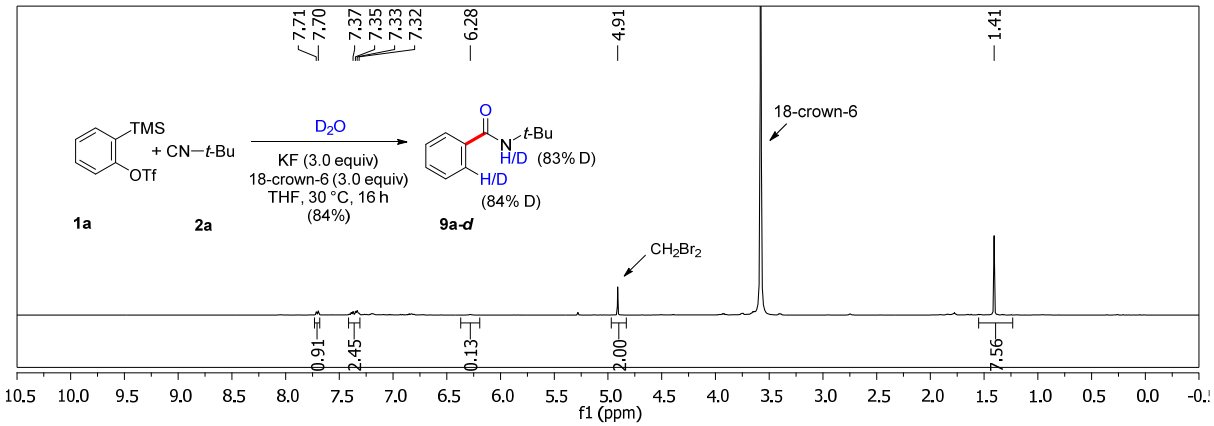
^{1}H-NMR Spectrum of Aromatic Amide 9a



^{1}H-NMR Spectrum of Crude Product (Reaction Using D_2O)

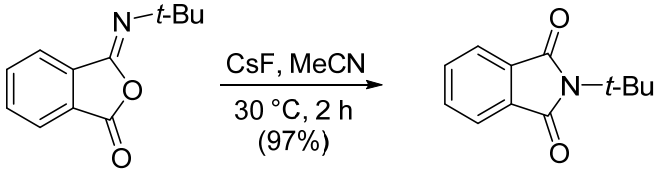


^{1}H-NMR Spectrum of Crude Product (Reaction Using D_2O, NMR yield = 84%)



5. Mechanistic Experiments

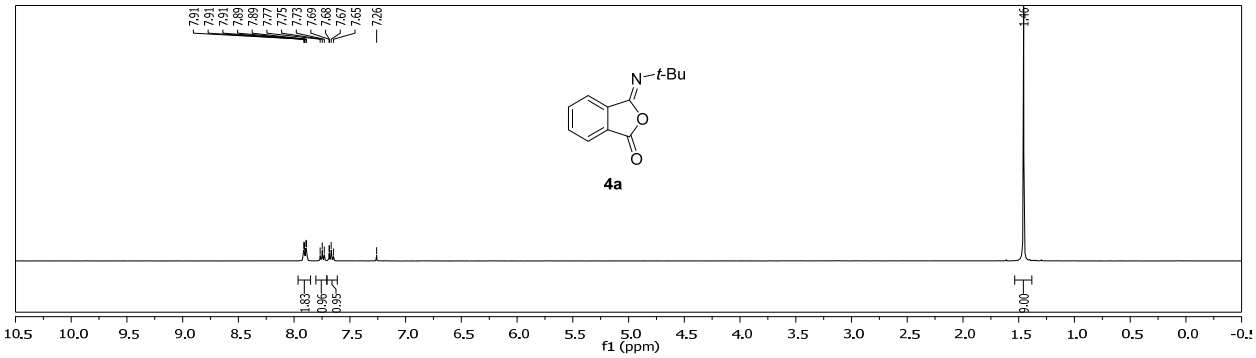
Experiment to Confirm Fluoride-Induced Rearrangement of Isoimide 7 to Phthalimide 3



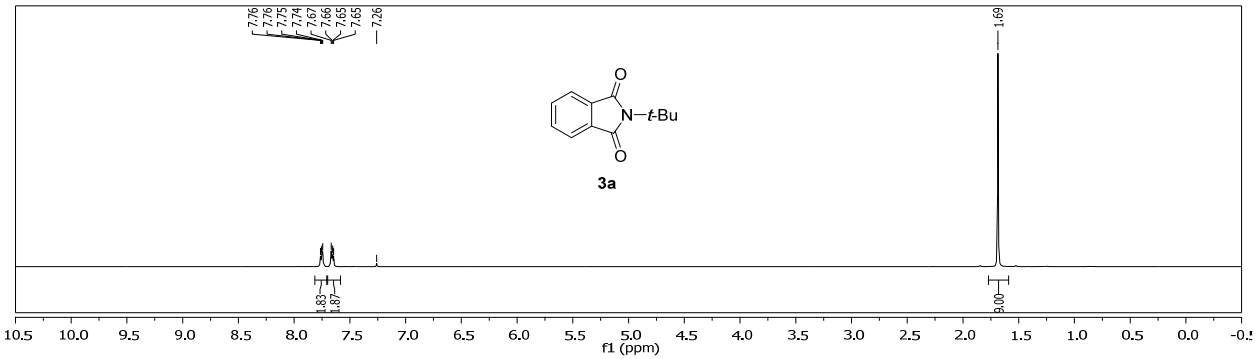
4a **3a**

To a flame-dried screw-capped test tube equipped with a magnetic stir bar was added dry CsF (0.152 g, 1.0 mmol). Then the screw-capped tube was evacuated and backfilled with argon, and CH_3CN (2.0 mL) was added under argon atmosphere. To the stirring solution were added (*tert*-butylimino)isobenzofuran-1(3*H*)-one (phthlaisoimide) **4a** (0.102 g, 0.5 mmol). Then the reaction mixture was stirred at 30 °C for 2 h. The reaction was stopped, and the solvent was evaporated and the crude residue was purified by column chromatography on silica gel to afford the phthalimide **3a** as a white solid (0.099 g, 97%).

^{1}H NMR Spectrum of Isoimide 4a

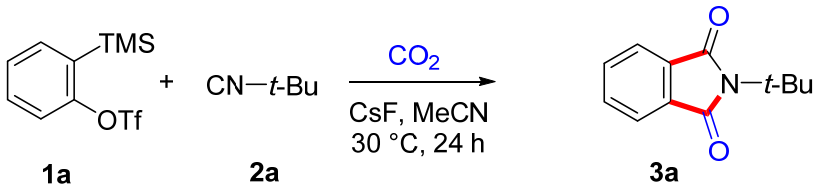


^{1}H NMR Spectrum of Phthalimide 3a



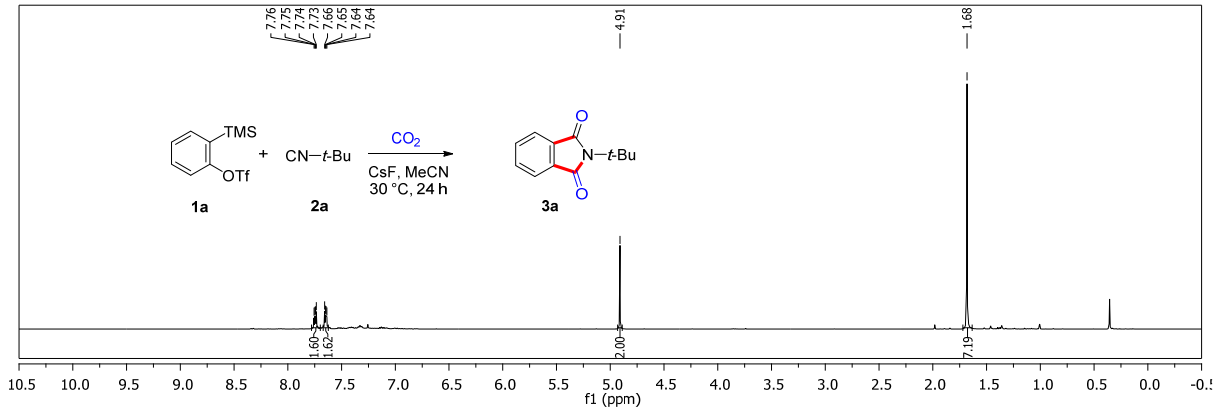
*This experiment indicates the fluoride-induced rearrangement of **7** to **3** as proposed in Scheme 4 of the manuscript.*

Detection of Phthalimide 3a after Filtration (Without Silica gel Column Chromatography)



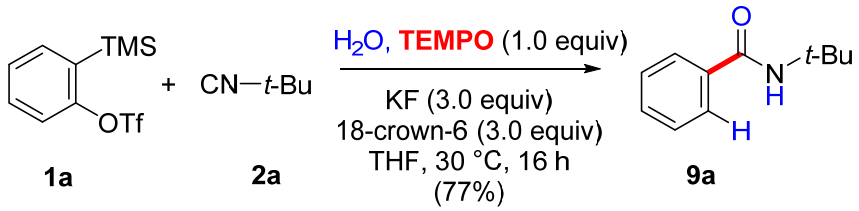
To a flame-dried screw-capped test tube equipped with a magnetic stir bar was added dry CsF (0.114 g, 1.5 mmol). Then the screw-capped tube was evacuated and backfilled with argon, and CH_3CN (1.0 mL) was added under argon atmosphere. To the stirring solution were added isocyanide **2a** (0.021 g, 28 µL, 0.25 mmol), and aryne precursor **1a** (0.112 g, 91 µL 0.375 mmol) under argon. Then the reaction mixture was purged with CO_2 (balloon), and subsequently the mixture was stirred at 30 °C for 24 h under an atmosphere of CO_2 (balloon). When TLC control showed the completion of the reaction (after 24 h) the reaction was stopped, and the reaction mixture diluted with CH_2Cl_2. The mixture was filtered through a filter paper and the residue washed with CH_2Cl_2 (15 ml). The solvent was evaporated to obtain the crude product whose yield was determined by ^{1}H NMR analysis using CH_2Br_2 (18.0 µL, 0.25 mmol) as the internal standard. The 1H NMR showed 80% yield of the phthalimide **3a**.

^{1}H NMR Spectrum of Crude Mixture after Filtration



*This experiment indicates that the rearrangement of **4a** to **3a** is not happening during purification using silica gel column chromatography.*

Reaction of Isocyanide, aryne and H_2O in the presence of TEMPO



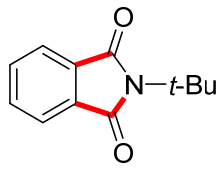
1a **2a** **9a**

To a flame-dried screw-capped test tube equipped with a magnetic stir bar was added 18-crown-6 (0.198 g, 0.75 mmol), KF (0.044 g, 0.75 mmol), TEMPO (0.039 g, 0.25 mmol). Then the screw-capped tube was evacuated and backfilled with argon. The mixture was dissolved in THF (1.0 mL) under argon atmosphere and then the mixture was kept stirring at 30 °C. To this stirring solution was added isocyanide **2a** (0.021 g, 28 µL, 0.25 mmol) followed by the aryne precursor **1a** (0.112 g, 91 µL, 0.375 mmol). To this reaction mixture was added H_2O (9.0 µL, 0.5 mmol) after 10 minutes. Then the reaction mixture was kept stirring for 16 h. The solvent was evaporated and the crude residue was purified by column chromatography on silica gel to afford the *N*-(*tert*-butyl)benzamide **9a** as a white solid (0.034 g, 77%). This indicates that the reaction is proceeding via an ionic mechanism.

*The formation of the benzamide derivative **9a** in 77% yield even in the presence of TEMPO, indicates that this reaction is not proceeding via any radical intermediates.*

6. Synthesis and Characterization of Phthalimides

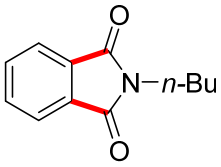
2-(*tert*-Butyl)isoindoline-1,3-dione (3a)[2]



Following the general procedure, 2-(trimethylsilyl)phenyl trifluoromethane sulfonate **1a** (0.224 g, 182 µL, 0.75 mmol) was treated with *tert*-butyl isocyanide **2a** (0.042 g, 57 µL, 0.5 mmol) in the presence of CsF (0.228 g, 1.5 mmol) in MeCN (2.0 mL) under an atmosphere of CO_2 (balloon pressure) at 30 °C for 24 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 95/05) of the crude mixture afforded 2-(*tert*-butyl) isoindoline-1,3-dione **3a** as a white solid (0.078 g, 76%).

***R*$_f$** (Pet. Ether/EtOAc=95/05): 0.30; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.77-7.75 (m, 2H), 7.67-7.65 (m, 2H), 1.69 (s, 9H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 169.82, 133.79, 132.28, 122.70, 57.96, 29.22. **HRMS** calculated $[M+H]^+$ for $C_{12}H_{14}O_2N$: 204.1019, found: 204.1019. **FTIR (cm^{-1}):** 2979, 1770, 1701, 1656, 1367, 1259, 1150, 1100, 872, 748.

2-Butylisoindoline-1,3-dione (3b)[3]

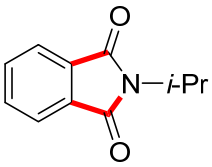


Following the general procedure, 2-(trimethylsilyl)phenyl trifluoromethane sulfonate **1a** (0.224 g, 182 µL, 0.75 mmol) was treated with 1-isocyanobutane **2b** (0.042 g, 52 µL, 0.5 mmol) in the presence of CsF (0.228 g, 1.5 mmol) in MeCN (2.0 mL) under an atmosphere of CO_2 (balloon pressure) at 30 °C for 24 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 95/05) of the crude mixture afforded 2-Butylisoindoline-1,3-dione **3d** as a colourless oil (0.062 g, 64%).

***R*$_f$** (Pet. Ether/EtOAc=80/20): 0.32; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.83-7.81 (m, 2H), 7.70-7.68 (m, 2H), 3.67 (t, *J* = 7.4 Hz 2H), 1.68-1.61 (m, 2H), 1.38-1.31 (m, 2H), 0.93 (t, *J* = 7.32 Hz 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 168.57, 133.93, 132.30, 123.24, 37.90, 30.75, 20.18, 13.74. **HRMS** calculated $[M+H]^+$ for $C_{12}H_{14}O_2N$: 204.1019, found: 204.1017. **FTIR (cm^{-1}):** 3055, 2927, 2870, 1771, 1709, 1615, 1466, 1437, 1395, 1264, 1651, 895, 719.

[2] Yan, X.; Fang, K.; Liu, H.; Xi, C. *Chem. Commun*. **2013**, *49*, 10650.
[3] Du, Y.; Hyster, T. K.; Rovis, T. *Chem. Commun.,* **2011**, *47,* 12074.

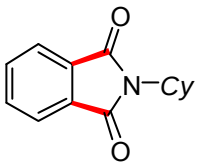
2-isopropylisoindoline-1,3-dione (3c)[3]



Following the general procedure, 2-(trimethylsilyl)phenyl trifluoromethane sulfonate **1a** (0.224 g, 182 μL, 0.75 mmol) was treated with isopropyl isocyanide **2c** (0.035 g, 47 μL, 0.5 mmol) in the presence of CsF (0.228 g, 1.5 mmol) in MeCN (2.0 mL) under an atmosphere of CO_2 (balloon pressure) at 30 °C for 24 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 95/05) of the crude mixture afforded 2-isopropylisoindoline-1,3-dione **3c** as a white solid (0.051 g, 54%).

R_f (pet. ether/EtOAc=95/05): 0.28; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.81-7.79 (m, 2H), 7.69-7.67 (m, 2H), 4.53 (p, *J* = 6.99Hz, 1H), 1.48 (d, *J* = 6.9Hz, 6H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 168.52, 133.89, 132.25, 123.11, 43.12, 20.26. **HRMS** calculated $[M+H]^+$ for $C_{11}H_{12}O_2N$: 190.0863, found: 190.0862. **FTIR (cm^{-1}):** 2987, 1772, 1718, 1458, 1385, 1366, 1044, 876, 793.

2-cyclohexylisoindoline-1,3-dione (3d)[4]

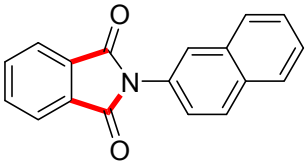


Following the general procedure, 2-(trimethylsilyl)phenyl trifluoromethane sulfonate **1a** (0.224 g, 182 μL, 0.75 mmol) was treated with isocyanocyclohexane **2d** (0.055 g, 62 μL, 0.5 mmol) in the presence of CsF (0.228 g, 1.5 mmol) in MeCN (2.0 mL) under an atmosphere of CO_2 (balloon pressure) at 30 °C for 24 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 95/05) of the crude mixture afforded 2-cyclohexylisoindoline-1,3-dione **3d** as a white solid (0.074 g, 64%).

R_f (Pet. Ether/EtOAc=95/05): 0.28; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.81-7.79 (m, 2H), 7.69-7.67 (m, 2H), 4.14-4.06 (m, 1H), 2.25-2.14 (m, 2H), 1.87-1.84 (m, 2H), 1.73-1.67 (m, 3H), 1.42-1.24 (m, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 168.59, 133.86, 132.20, 123.12, 51.0, 29.99, 26.14, 25.24. **HRMS** calculated $[M+H]^+$ for $C_{14}H_{16}O_2N$: 230.1176, found: 230.1174. **FTIR (cm^{-1}):** 2925, 2853, 1760, 1702, 1465, 1446, 1349, 1264, 1028, 901, 709.

[4] Nageswara Rao, S.; Mohan, D. C.; Adimurthy, S. *Org. Lett.* **2013**, *15*, 1496.

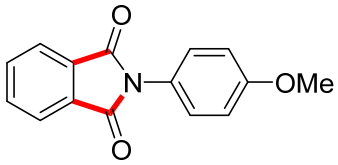
2-(Naphthalen-2-yl)isoindoline-1,3-dione (3e)[5]



Following the general procedure, 2-(trimethylsilyl)phenyl trifluoromethane sulfonate **1a** (0.224 g, 182 µL, 0.75 mmol) was treated with napthyl isocyanide **2e** (0.077 g, 0.5 mmol) in the presence of CsF (0.228 g, 1.5 mmol) in MeCN (2.0 mL) under an atmosphere of CO_2 (balloon pressure) at 60 °C for 6 h. The reaction mixture was cooled and the evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 95/05) of the crude mixture afforded 2-(Naphthalen-2-yl)isoindoline-1,3-dione **3e** as a brown solid (0.066 g, 48%).

R_f (pet. ether/EtOAc=80/20): 0.34; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.00-7.95 (m, 4H), 7.90 (t, *J* = 4.6Hz, 2H), 7.82-7.80 (m, 2H), 7.57-7.53 (m, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ167.58, 134.60, 133.48, 132.76, 131.97, 129.28, 129.13, 128.36, 127.90, 126.85, 126.72, 125.68, 124.33, 123.93. **HRMS** calculated $[M+Na]^+$ for $C_{18}H_{11}O_2NNa$: 296.0682, found: 296.0678. **FTIR (cm^{-1}):** 3056, 2979, 1762, 1708, 1631, 1514, 1364, 1263, 1146, 1013, 903, 732.

2-(4-Methoxyphenyl)isoindoline-1,3-dione (3f)[4]

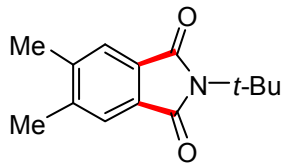


Following the general procedure, 2-(trimethylsilyl)phenyl trifluoromethane sulfonate **1a** (0.224 g, 182 µL, 0.75 mmol) was treated with 1-isocyano-4-methoxybenzene **2f** (0.067 g, 0.5 mmol) in the presence of CsF (0.228 g, 1.5 mmol) in MeCN (2.0 mL) under an atmosphere of CO_2 (balloon pressure) at 60 °C for 6 h. The reaction mixture was cooled and the evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 90/10) of the crude mixture afforded 2-(4-Methoxyphenyl)isoindoline-1,3-dione **3f** as a brown solid (0.056 g, 44%).

R_f (Pet. Ether/EtOAc=80/20): 0.34; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.97-7.95 (m, 2H), 7.81-7.78 (m, 2H), 7.35 (d, *J* = 9.1 Hz, 2H), 7.02 (d, *J* = 9.1 Hz, 2H), 3.86 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 167.71, 159.38, 134.44, 131.94, 128.07, 124.38, 123.79, 114.60, 55.63. **HRMS** calculated $[M+Na]^+$ for $C_{15}H_{11}O_3NNa$: 276.0631, found: 276.0629. **FTIR (cm^{-1}):** 3054, 1716, 1514, 1421, 1387, 1264, 729, 701.

[5] Sawant, D. N.; Wagh, Y. S.; Bhatte, K. D.; Bhanage, B. M. *Eur. J. Org. Chem.* **2011**, 6719.

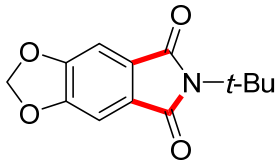
2-(*tert*-Butyl)-5,6-dimethylisoindoline-1,3-dione (3g)[2]



Following the general procedure, 4,5-dimethyl-2-(trimethylsilyl)phenyl trifluoromethanesulfonate **1g** (0.245 g, 182 µL, 0.75 mmol) was treated with *tert*-butyl isocyanide **2a** (0.042 g, 57 µL, 0.5 mmol) in the presence of CsF (0.228 g, 1.5 mmol) in MeCN (2.0 mL) under an atmosphere of CO_2 (balloon pressure) at 30 °C for 24 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 95/05) of the crude mixture afforded 2-(*tert*-Butyl)-5,6-dimethylisoindoline-1,3-dione **3g** as a white solid (0.074 g, 64%).

***R*$_f$** (Pet. Ether/EtOAc=95/05): 0.28; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.49 (s, 2H), 2.37 (s, 6H), 1.67 (s, 9H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 170.20, 143.35, 130.34, 123.60, 57.64, 29.24, 20.63. **HRMS** calculated $[M+H]^+$ for $C_{14}H_{18}O_2N$: 232.1332, found: 232.1331. **FTIR (cm^{-1}):** 2974, 1762, 1708, 1619, 1541, 1460, 1352, 1257, 1206, 1142, 997, 801, 744.

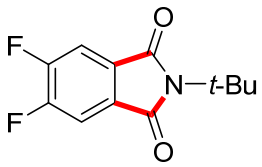
6-(*tert*-Butyl)-5*H*-[1,3]dioxolo[4,5-*f*]isoindole-5,7(6*H*)-dione (3h)[2]



Following the general procedure, 6-(trimethylsilyl)benzo[*d*][1,3]dioxol-5-yl trifluoromethanesulfonate **1h** (0.128 g, 0.375 mmol) was treated with *tert*-butyl isocyanide **2a** (0.021 g, 28 µL, 0.25 mmol) in the presence of CsF (0.114 g, 1.5 mmol) in MeCN (2.0 mL) under an atmosphere of CO_2 (balloon pressure) at 30 °C for 24 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 95/05) of the crude mixture afforded 6-(*tert*-butyl)-5*H*-[1,3]dioxolo[4,5-*f*]isoindole-5,7(6*H*)-dione **3h** as a brown solid (0.043 g, 76%).

***R*$_f$** (Pet. Ether/EtOAc=95/05): 0.28; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.11 (s, 2H), 6.12 (s, 2H), 1.65 (s, 9H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 169.21, 152.53, 127.74, 103.10, 102.83, 57.84, 29.23. **HRMS** calculated $[M+H]^+$ for $C_{13}H_{14}O_4N$: 248.0917, found: 248.0914. **FTIR (cm^{-1}):** 3054, 2987, 1763, 1475, 1349, 1264, 1126, 1007, 816, 732.

2-(*tert*-Butyl)-5,6-difluoroisoindoline-1,3-dione (3i)[2]

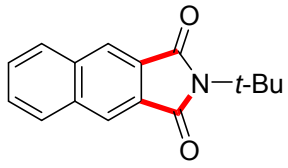


Following the general procedure, 4,5-difluoro-2-(trimethylsilyl)phenyl trifluoromethanesulfonate **1i** (0.251 g, 0.75 mmol) was treated with *tert*-butyl isocyanide **2a** (0.042 g, 57 µL, 0.5 mmol) in the presence of CsF (0.228 g, 1.5 mmol) in MeCN (2.0 mL) under an atmosphere of CO_2

(balloon pressure) at 30 °C for 24 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 95/05) of the crude mixture afforded 2-(*tert*-butyl)-5,6-difluoroisoindoline-1,3-dione **3i** as a white solid (0.066 g, 55%).

***R*$_f$** (Pet. Ether/EtOAc=95/05): 0.32; **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.58 (t, *J* = 7.3 Hz, 2H), 1.67 (s, 9H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ 167.67, 155.86 (dd, J_1 = 15.9 Hz, J_1 = 260.4 Hz), 128.97 (t, *J* = 5.4 Hz), 112.54 (q, *J* = 7.3 Hz) 56.63, 29.10. **HRMS** calculated $[M+H]^+$ for $C_{12}H_{12}F_2NO_2$: 240.0831, found: 240.0829. **FTIR (cm^{-1}):** 3061, 2981, 1773, 1700, 1619, 1465, 1323, 1287, 1205, 1107, 954, 880, 743.

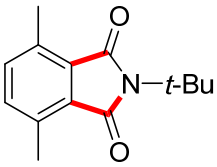
2-(*tert*-Butyl)isoindoline-1,3-dione(3j)



Following the general procedure, 3-(trimethylsilyl)naphthalen-2-yl trifluoromethanesulfonate **1j** (0.131 g, 0.375 mmol) was treated with *tert*-butyl isocyanide **2a** (0.042 g, 57 μL, 0.25 mmol) in the presence of CsF (0.114 g, 0.75 mmol) in MeCN (1.0 mL) under an atmosphere of CO_2 (balloon pressure) at 30 °C for 24 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 95/05) of the crude mixture afforded 2-(*tert*-Butyl)-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione **3j** as a pale yellow solid (0.028 g, 44%).

***R*$_f$** (Pet. ether/EtOAc=95/05): 0.28; **^{1}H NMR (400 MHz, CDCl$_3$)** δ 8.25, (s, 2H), 8.04-8.02 (m, 2H), 7.67-7.65 (m, 2H), 1.75 (s, 9H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ 169.53, 135.72, 130.25, 128.93, 128.30, 123.87, 58.33, 29.25. **HRMS** calculated $[M+H]^+$ for $C_{16}H_{16}O_2N$: 254.1176, found: 254.1173. **FTIR (cm^{-1}):** 3058, 3978, 1761, 1760, 1686, 1625, 1607, 1589, 1478, 1398, 1274, 1150, 1011, 905, 750.

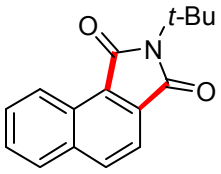
2-(*tert*-Butyl)-4,7-dimethylisoindoline-1,3-dione (3k)



Following the general procedure, 3,6-dimethyl-2-(trimethylsilyl)phenyl trifluoromethanesulfonate **1k** (0.245 g, 0.75 mmol) was treated with *tert*-butyl isocyanide **2a** (0.042 g, 57 μL, 0.5 mmol) in the presence of CsF (0.228 g, 1.5 mmol) in MeCN (2.0 mL) under an atmosphere of CO_2 (balloon pressure) at 30 °C for 24 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 95/05) of the crude mixture afforded 2-(*tert*-Butyl)-4,7-dimethylisoindoline-1,3-dione **3k** as a white solid (0.044 g, 38%).

***R*$_f$** (pet. ether/EtOAc=95/05): 0.46; **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.39, (d, *J* = 7.68Hz, 1H), 7.28 (d, *J* = 7.68 Hz, 1H), 2.66 (s, 3H), 2.63 (s, 3H), 1.46 (s, 9H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ166.16, 137.06, 136.55, 134.98, 133.56, 56.15, 30.61, 30.55, 30.50, 18.40, 17.49. **HRMS** calculated $[M+H]^+$ for $C_{14}H_{18}NO_2$: 232.1332, found: 232.1331. **FTIR (cm^{-1}):** 2968, 2928, 2872, 1781, 1698, 1503, 1457, 1362, 1238, 1110, 824, 764.

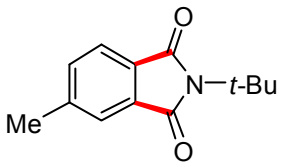
2-(*tert*-Butyl)-1*H*-benzo[*e*]isoindole-1,3(2*H*)-dione (3l)[2]



Following the general procedure, 1-(trimethylsilyl)naphthalen-2-yl trifluoromethanesulfonate **1l** (0.261 g, 0.75 mmol) was treated with *tert*-butyl isocyanide **2a** (0.042 g, 57 μL, 0.5 mmol) in the presence of CsF (0.228 g, 1.5 mmol) in MeCN (2.0 mL) under an atmosphere of CO_2 (balloon pressure) at 30 °C for 24 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 95/05) of the crude mixture afforded 2-(*tert*-Butyl)-1*H*-benzo[*e*]isoindole-1,3(2*H*)-dione **3l** as a white solid (0.103 g, 81%).

***R*$_f$** (Pet. Ether/EtOAc=95/05): 0.36; **^{1}H NMR (400 MHz, CDCl$_3$)** δ 8.94 (d, *J* = 8.4 Hz, 1H), 8.10 (d, *J* = 8.4 Hz, 1H), 7.92 (d, *J* = 8.2 Hz, 1H), 7.77(d, *J* = 8.3 Hz, 1H), 7.71-7.67 (m, 1H), 7.64-7.60 (m, 1H), 1.74 (s, 9H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ 171.22, 170.31, 136.65, 134.75, 131.54, 129.40, 128.84, 128.50, 127.85, 127.04, 125.04, 118.16, 57.83, 29.36. **HRMS** calculated $[M+Na]^+$ for $C_{16}H_{15}O_2NNa$: 276.0995, found: 276.0991. **FTIR (cm^{-1}):** 3057, 2970, 2936, 1922, 1763, 1696, 1635, 1598, 1478, 1337, 1275, 1099, 868, 783.

2-(*tert*-Butyl)-5-methylisoindoline-1,3-dione (3m)[2]



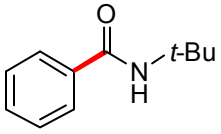
Following the general procedure, 5-methyl-2-(trimethylsilyl)phenyl trifluoromethanesulfonate **1m** (0.234 g, 0.75 mmol) was treated with *tert*-butyl isocyanide **2a** (0.042 g, 57 μL, 0.5 mmol) in the presence of CsF (0.228 g, 1.5 mmol) in MeCN (2.0 mL) under an atmosphere of CO_2 (balloon pressure) at 30 °C for 24 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 95/05) of the crude mixture afforded 2-(*tert*-Butyl)-5-methylisoindoline-1,3-dione **3m** as a white solid (0.095 g, 87%).

***R*$_f$** (Pet. Ether/EtOAc=95/05): 0.28; **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.63 (d, *J* = 7.5 Hz, 1H), 7.54 (s, 1H), 7.42 (d, *J* = 7.6 Hz, 1H), 2.47 (s, 3H), 1.67 (s, 9H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ

169.98, 169.92, 144.92, 134.38, 132.67, 129.74, 123.15, 122.61, 57.8, 29.23, 22.07. **HRMS** calculated $[M+Na]^+$ for $C_{13}H_{15}O_2NNa$: 240.0995, found: 240.0992. **FTIR (cm^{-1}):** 2980, 2922, 1759, 1698, 1621, 1485, 1400, 1370, 1314, 1227, 1100, 796.

7. Synthesis and Characterization of Aromatic Amides

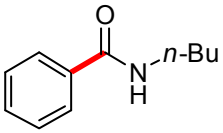
***N*-(*tert*-Butyl)benzamide (9a)**[6]



Following the general procedure, 2-(trimethylsilyl)phenyl trifluoromethane sulfonate **1a** (0.224 g, 182 µL, 0.75 mmol) was treated with *tert*-butyl isocyanide **2a** (0.042 g, 57 µL, 0.5 mmol) in the presence of KF (0.087 g, 1.5 mmol) and 18-crown-6 (0.396 g, 1.5 mmol) in THF (2.0 mL) at 30 °C. To this reaction mixture was added H_2O (18 µL, 1 mmol) after 10 minutes. The mixture was then stirred at 30 °C for 16 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 92/08) of the crude mixture afforded *N*-(*tert*-butyl)benzamide **9a** as a white solid (0.072 g, 81%).

***R*$_f$** (Pet. Ether/EtOAc=80/20): 0.44; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.71 (d, *J* = 7.2 Hz, 2H), 7.46-7.37 (m, 3H), 5.99 (br s, 1H), 1.46 (s, 9H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 167.03, 136.01, 131.16, 128.56, 126.80, 51.68, 28.96. **HRMS** calculated $[M+Na]^+$ for $C_{11}H_{15}ONNa$: 200.1046, found: 200.1046. **FTIR (cm^{-1}):** 1116, 2964, 2928, 1633, 1602, 1578, 1532, 1275, 719.

***N*-Butylbenzamide (9b)**[7]



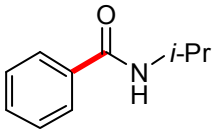
Following the general procedure, 2-(trimethylsilyl)phenyl trifluoromethane sulfonate **1a** (0.224 g, 182 µL, 0.75 mmol) was treated with 1-isocyanobutane **2b** (0.042 g, 52 µL, 0.5 mmol) in the presence of KF (0.087 g, 1.5 mmol) and 18-crown-6 (0.396 g, 1.5 mmol) in THF (2.0 mL) at 30 °C. To this reaction mixture was added H_2O (18 µL, 1 mmol) after 10 minutes. The mixture was then stirred at 30 °C for 16 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 92/08) of the crude mixture afforded *N*-butylbenzamide **9b** as a colorless oil (0.061 g, 69%).

***R*$_f$** (Pet. Ether/EtOAc=80/20): 0.26; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.75 (d, *J* = 7.1 Hz, 2H), 7.48-7.46 (m, 1H), 6.33 (bs, 1H), 3.44 (q, 2H), 3.61-3.55 (m, 2H), 3.43-3.36 (m, 2H), 0.94 (t, *J* =

[6] Xia, Z.; Zhu, Q. *Org. Lett*. **2013**, *15*, 4110.
[7] Mali, S. M.; Bhaisare, R. D.; Gopi, H. N. *J. Org. Chem.* **2013**, *78*, 5550.

7.3 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 167.70, 134.96, 131.36, 128.59, 126.96, 39.91, 31.82, 20.25, 13.88. **HRMS** calculated $[M+Na]^+$ for $C_{11}H_{13}ONNa$: 200.1046, found: 200.1045. **FTIR (cm^{-1}):** 3310, 2957, 2931, 2871, 1635, 1577, 1540, 1435, 1376, 1074, 693.

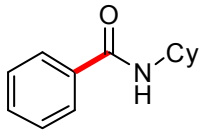
***N*-isopropylbenzamide (9c)**[8]



Following the general procedure, 2-(trimethylsilyl)phenyl trifluoromethane sulfonate **1a** (0.224 g, 182 μL, 0.75 mmol) was treated with 2-isocyanopropane **2c** (0.035 g, 47 μL, 0.5 mmol) in the presence of KF (0.087 g, 1.5 mmol) and 18-crown-6 (0.396 g, 1.5 mmol) in THF (2.0 mL) at 30 °C. To this reaction mixture was added H_2O (18 μL, 1 mmol) after 10 minutes. The mixture was then stirred at 30 °C for 16 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 90/10) of the crude mixture afforded *N*-isopropylbenzamide **9c** as a white solid (0.068 g, 83%).

***R*$_f$** (Pet. Ether/EtOAc=80/20): 0.26; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.75 (d, *J* = 7.4 Hz, 2H), 7.49-7.46 (m, 1H), 7.43-7.39 (m, 2H), 5.99 (bs, 1H), 4.32-4.24 (m, 1H), 1.26 (d, *J* = 6.4 Hz, 6H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 166.84, 136.13, 131.37, 128.61, 126.94, 42.01, 22.93. **HRMS** calculated $[M+Na]^+$ for $C_{10}H_{13}ONNa$: 186.0889, found: 186.0888. **FTIR (cm^{-1}):** 3302, 3057, 2973, 1632, 1603, 1578, 1533, 1386, 1264, 1074, 733.

***N*-cyclohexylbenzamide (9d)**[8]



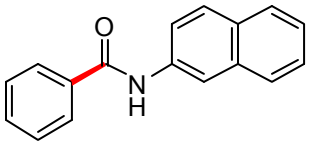
Following the general procedure, 2-(trimethylsilyl)phenyl trifluoromethane sulfonate **1a** (0.224 g, 182 μL, 0.75 mmol) was treated with isocyanocyclohexane **2d** (0.055 g, 62 μL, 0.5 mmol) in the presence of KF (0.087 g, 1.5 mmol) and 18-crown-6 (0.396 g, 1.5 mmol) in THF (2.0 mL) at 30 °C. To this reaction mixture was added H_2O (18 μL, 1 mmol) after 10 minutes. The mixture was then stirred at 30 °C for 16 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 92/08) of the crude mixture afforded *N*-cyclohexylbenzamide **9d** as a white solid (0.069 g, 68%).

***R*$_f$** (Pet. Ether/EtOAc=80/20): 0.32; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.74 (d, *J* = 7.8 Hz, 2H), 7.47-7.37 (m, 3H), 6.14 (bs, 1H), 3.99-3.92 (m, 1H), 2.02-1.93 (m, 2H), 1.76-1.71 (m, 2H), 1.66-1.62 (m, 1H), 1.44-1.34 (m, 2H), 1.23-1.15 (m, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 166.76,

[8] Zhao, Q.; Li, H.; Wang, L. *Org. Biomol. Chem.* **2013**, *11*, 6772.

135.18, 131.28, 128.55, 126.95, 48.78, 33.27, 25.64, 25.02. **HRMS** calculated $[M+Na]^+$ for $C_{13}H_{17}O_2NNa$: 226.1202, found: 226.1201. **FTIR (cm^{-1}):** 3329, 3247, 3067, 2929, 1716, 1627, 1603, 1536, 1452, 1291, 1241, 1028, 790.

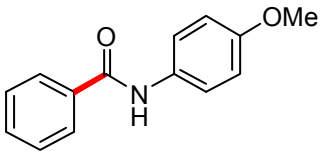
***N*-(naphthalen-2-yl)benzamide (9e)**[9]



Following the general procedure, 2-(trimethylsilyl)phenyl trifluoromethane sulfonate **1a** (0.224 g, 182 μL, 0.75 mmol) was treated with 2-isocyanonaphthalene **2e** (0.077 g, 0.5 mmol) in the presence of KF (0.087 g, 1.5 mmol) and 18-crown-6 (0.396 g, 1.5 mmol) in THF (2.0 mL) at 60 °C. To this reaction mixture was added H_2O (18 μL, 1 mmol) after 10 minutes. The mixture was then stirred at 60 °C for 3 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 92/08) of the crude mixture afforded *N*-(naphthalen-2-yl)benzamide **9e** as a brown solid (0.032 g, 26%).

***R*$_f$** (Pet. Ether/EtOAc=80/20): 0.44; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.34 (s, 1H), 8.14 (bs, 1H), 7.91 (d, *J* = 7.1 Hz, 2H), 7.82-7.78 (m, 3H), 7.61-7.53 (m, 2H), 7.50-7.40 (m, 4H),. **^{13}C NMR (100 MHz, $CDCl_3$)** δ 166.10, 136.51, 136.05, 133.97, 132.02, 130.90, 128.93, 127.86, 127.70, 127.20, 126.67, 125.26, 120.28, 117.24. **HRMS** calculated $[M+Na]^+$ for $C_{17}H_{13}ONNa$: 270.0889, found: 270.0884. **FTIR (cm^{-1}):** 3054, 2986, 1771, 1708, 1540, 1467, 1370, 1013, 873, 732.

***N*-(4-Methoxyphenyl)benzamide (9f)**[7]

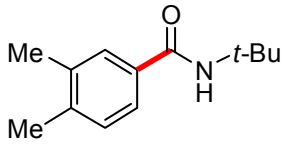


Following the general procedure, 2-(trimethylsilyl)phenyl trifluoromethane sulfonate **1a** (0.224 g, 182 μL, 0.75 mmol) was treated with 1-isocyano-4-methoxybenzene **2f** (0.067 g, 0.5 mmol) in the presence of KF (0.087 g, 1.5 mmol) and 18-crown-6 (0.396 g, 1.5 mmol) in THF (2.0 mL) at 60 °C. To this reaction mixture was added H_2O (18 μL, 1 mmol) after 10 minutes. The mixture was then stirred at 60 °C for 3 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 88/12) of the crude mixture afforded *N*-(4-methoxyphenyl)benzamide **9f** as a pale brown solid (0.065 g, 57%).

[9] Young, D. D.; Connelly, C. M.; Grohmann, C.; Deiters, A. *J. Am. Chem. Soc.* **2010**, *132*, 7976.

***R*$_f$** (Pet. Ether/EtOAc=80/20): 0.28; **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.86 (d, *J* = 7.12 Hz, 3H), 7.56-7.44 (m, 5H), 6.89 (d, *J* = 8.74 Hz, 2H), 3.81 (s, 3H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ 166.81, 146.75, 136.14, 131.82, 131.13, 128.86, 127.13, 122.28, 114.36, 56.61. **HRMS** calculated $[M+Na]^+$ for $C_{14}H_{13}O_2NNa$: 250.0838, found: 250.0836. **FTIR (cm^{-1}):** 3330, 3053, 1645, 1601, 1577, 1513, 1410, 1348, 1264, 792.

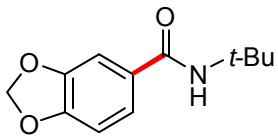
***N*-(*tert*-Butyl)-3,4-dimethylbenzamide (9g)**



Following the general procedure, 4,5-dimethyl-2-(trimethylsilyl)phenyl trifluoromethanesulfonate **1g** (0.245 g, 182 μL, 0.75 mmol) was treated with *tert*-butyl isocyanide **2a** (0.042 g, 57 μL, 0.5 mmol) in the presence of KF (0.087 g, 1.5 mmol) and 18-crown-6 (0.396 g, 1.5 mmol) in THF (2.0 mL) at 30 °C. To this reaction mixture was added H_2O (18 μL, 1 mmol) after 10 minutes. The mixture was then stirred at 30 °C for 16 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 92/08) of the crude mixture afforded *N*-(*tert*-butyl)-3,4-dimethylbenzamide **9g** as a white solid (0.080 g, 78%).

***R*$_f$** (Pet. Ether/EtOAc=80/20): 0.50; **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.90 (s, 1H), 7.42 (d, *J* = 7.8 Hz, 1H), 7.33 (d, *J* = 7.7 Hz, 1H) , 5.96 (bs, 1H), 2.27 (s, 6H), 1.46 (s, 9H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ 167.23, 140.11, 136.86, 133.46, 129.69, 128.11, 124.11, 51.56, 28.93, 19.81. **HRMS** calculated $[M+H]^+$ for $C_{13}H_{20}ON$: 206.1539, found: 206.1539. **FTIR (cm^{-1}):** 3309, 2968, 2921, 1636, 1612, 1575, 1533, 1450, 1313, 1235, 1141, 829.

***N*-(*tert*-Butyl)benzo[*d*][1,3]dioxole-5-carboxamide (9h)**[10]

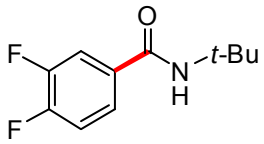


Following the general procedure, 6-(trimethylsilyl)benzo[*d*][1,3]dioxol-5-yl trifluoromethanesulfonate **1h** (0.257 g, 0.75 mmol) was treated with *tert*-butyl isocyanide **2a** (0.042 g, 57 μL, 0.5 mmol) in the presence of KF (0.087 g, 1.5 mmol) and 18-crown-6 (0.396 g, 1.5 mmol) in THF (2.0 mL) at 30 °C. To this reaction mixture was added H_2O (18 μL, 1 mmol) after 10 minutes. The mixture was then stirred at 30 °C for 16 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 92/08) of the crude mixture afforded *N*-(*tert*-Butyl)benzo[*d*][1,3]dioxole-5-carboxamide **9h** as a white solid (0.102 g, 92%).

[10] Callens, E.; Burtonb, A. J.; Barrett, A. G. M. *Tetrahedron Lett.* **2006**, *47*, 8699.

***R*$_f$** (Pet. Ether/EtOAc=80/20): 0.32; **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.23-7.21 (m, 2H), 6.78 (d, *J* = 8.7 Hz, 1H), 5.99 (s, 2H), 5.86 (bs, 1H), 1.44 (s, 9H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ 166.30, 150.06, 147.99, 130.35, 121.22, 107.93, 107.60, 101.70, 51.83, 28.99. **HRMS** calculated $[M+Na]^+$ for $C_{12}H_{15}O_3NNa$: 244.0944, found: 244.0943. **FTIR (cm^{-1}):** 3332, 2970, 2904, 1732, 1716, 1698, 1645, 1621, 1539, 1483, 1435, 1112, 838

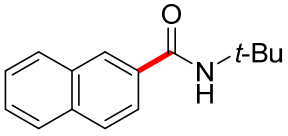
_N_-(_tert_-Butyl)-3,4-difluorobenzamide (9i)



Following the general procedure, 4,5-difluoro-2-(trimethylsilyl)phenyl trifluoromethanesulfonate **1i** (0.251 g, 0.75 mmol) was treated with *tert*-butyl isocyanide **2a** (0.042 g, 57 μL, 0.5 mmol) in the presence of KF (0.087 g, 1.5 mmol) and 18-crown-6 (0.396 g, 1.5 mmol) in THF (2.0 mL) at 30 °C. To this reaction mixture was added H_2O (18 μL, 1 mmol) after 10 minutes. The mixture was then stirred at 30 °C for 16 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 92/08) of the crude mixture afforded *N*-(*tert*-Butyl)-3,4-difluorobenzamide **9i** as a white solid (0.071 g, 66%).

***R*$_f$** (Pet. Ether/EtOAc=90/10): 0.26; **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.59-7.54 (m, 1H), 7.46-7.42 (m, 1H), 7.20-7.13 (m, 1H), 5.91 (bs, 1H), 1.46 (s, 9H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ 166.84, 153.64 (dd, J_1 = 13.0 Hz, J_2 = 253.4 Hz), 151.59 (dd, J_1 = 13.1 Hz, J_2 = 250.32 Hz), 133.13 (t, J = 4.1 Hz), 123.17 (q, J = 3.6 Hz), 117.34 (d, J = 13 Hz), 116.71(d, J = 13 Hz), 52.06, 28.89. **HRMS** calculated $[M+Na]^+$ for $C_{11}H_{13}ONF_2Na$: 236.0857, found: 236.0854. **FTIR (cm^{-1}):** 3308, 3054, 2985, 1667, 1606, 1541, 1504, 1456, 1421, 1394, 1264, 834, 731.

_N_-(_tert_-Butyl)-2-naphthamide (9j)[11]



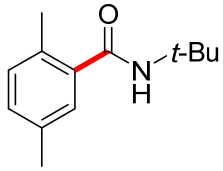
Following the general procedure, 3-(trimethylsilyl)naphthalen-2-yl trifluoromethanesulfonate **1j** (0.261 g, 0.75 mmol) was treated with *tert*-butyl isocyanide **2a** (0.042 g, 57 μL, 0.5 mmol) in the presence of KF (0.087 g, 1.5 mmol) and 18-crown-6 (0.396 g, 1.5 mmol) in THF (2.0 mL) at 30 °C. To this reaction mixture was added H_2O (18 μL, 1 mmol) after 10 minutes. The mixture was then stirred at 30 °C for 16 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc

[11] Jiang, H.; Liu, B.; Li, Y.; Wang, A.; Huang, H. *Org. Lett.* **2011**, *13*, 1028.

= 92/08) of the crude mixture afforded *N*-(*tert*-Butyl)-2-naphthamide **9j** as a white solid (0.081 g, 72%).

***R*$_f$** (pet. ether/EtOAc=80/20): 0.58; **^{1}H NMR (400 MHz, CDCl$_3$)** δ 8.22 (s, 1H), 7.91-7.78 (m, 4H), 7.56-7.50 (m, 2H), 6.14 (bs, 1H), 1.52 (s, 9H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ167.13, 134.65, 133.24, 132.74, 126.77, 123.71, 51.87, 29.04. **HRMS** calculated $[M+H]^+$ for $C_{15}H_{18}ON$: 228.1383, found: 228.1382. **FTIR (cm^{-1}):** 3329, 3055, 2978, 1636, 1626, 1594, 1541, 1456, 1319, 1139, 952, 763.

N-(*tert*-Butyl)-2,5-dimethylbenzamide (9k)

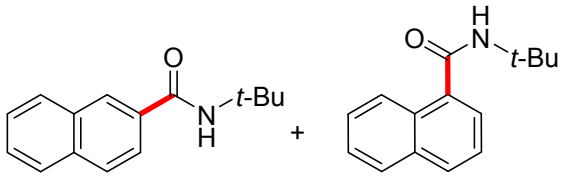


Following the general procedure, 3,6-dimethyl-2-(trimethylsilyl)phenyl trifluoromethanesulfonate **1k** (0.245 g, 0.75 mmol) was treated with *tert*-butyl isocyanide **2a** (0.042 g, 57 μL, 0.5 mmol) in the presence of KF (0.087 g, 1.5 mmol) and 18-crown-6 (0.396 g, 1.5 mmol) in THF (2.0 mL) at 30 °C. To this reaction mixture was added H_2O (18 μL, 1 mmol) after 10 minutes. The mixture was then stirred at 30 °C for 16 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 92/08) of the crude mixture afforded *N*-(*tert*-Butyl)-2,5-dimethylbenzamide **9k** as a white solid (0.078 g, 76%).

***R*$_f$** (pet. ether/EtOAc=80/20): 0.52; **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.11 (s, 1H), 7.06 (s, 2H), 5.61 (bs, 1H), 2.36 (s, 3H), 2.30 (s, 3H), 1.45 (s, 9H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ 169.99, 137.84, 135.28, 130.12, 127.14, 51.77, 28.95, 20.87, 19.13. **HRMS** calculated $[M+Na]^+$ for $C_{13}H_{19}ONNa^+$: 228.1359, found: 228.1359. **FTIR (cm^{-1}):** 3053, 2987, 1662, 1508, 1454, 1364, 1264, 730.

N-(*tert*-Butyl)-2-naphthamide (9l)[12] and N-(*tert*-Butyl)-1-naphthamide (9l')[11]

Following the general procedure, 1-(trimethylsilyl)naphthalen-2-yl trifluoromethanesulfonate **1l** (0.261 g, 0.75 mmol) was treated with *tert*-butyl isocyanide **2a** (0.042 g, 57 μL, 0.5 mmol) in the presence of KF (0.087 g, 1.5 mmol) and 18-crown-6 (0.396 g, 1.5 mmol) in THF (2.0 mL) at 30 °C.

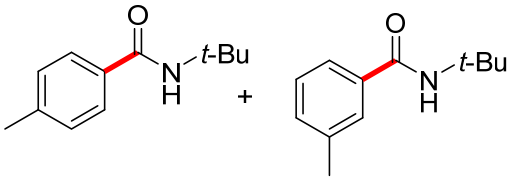


[12] Reddy, K. L. *Tetrahedron Lett.* **2003,** *44,* 1453.

To this reaction mixture was added H_2O (18 μL, 1 mmol) after 10 minutes. The mixture was then stirred at 30 °C for 16 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 92/08) of the crude mixture afforded *N*-(*tert*-Butyl)-2-naphthamide and *N*-(*tert*-butyl)-1-naphthamide as an inseparable mixture of regioisomers in 1.2:1 ratio **9l : 9l'** as a white solid (0.090 g, 79%).

R_f (Pet. Ether/EtOAc=90/10): 0.29; **^{1}H NMR (400 MHz, $CDCl_3$) of Major isomer:** δ 8.27 (d, *J* = 8.2 Hz, 1H), 7.56-7.48 (m, 5H), 7.42 (t, *J* = 7.17 Hz, 1H), 5.86 (bs, 1H), 1.53 (s, 9H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 169.27, 136.04, 133.74, 130.15, 130.11, 128.36, 127.51, 127.06, 126.40, 125.43, 124.52, 52.13, 29.02. **^{1}H NMR (400 MHz, $CDCl_3$) of Minor isomer:** δ 8.22 (s, 1H), 7.91-7.84 (m, 5H), 7.80-7.77 (m, 1H), 6.16 (bs, 1H), 1.52 (s, 9H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 167.08, 134.63, 133.24, 132.73, 128.93, 128.42, 127.81, 127.06, 126.75, 124.85, 123.71, 51.83, 29.02. **HRMS** calculated $[M+H]^+$ for $C_{15}H_{18}ON$: 228.1383, found: 228.1381. **FTIR (cm^{-1}):** 3317, 3054, 2976, 1638, 1592, 1577, 1516, 1476, 1451, 1303, 1218, 731.

***N*-(*tert*-Butyl)-3-methylbenzamide (9m)**[6] **and *N*-(*tert*-Butyl)-4-methylbenzamide (9m')**[13]



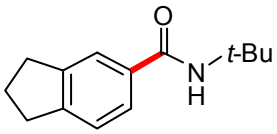
Following the general procedure, 4-methyl-2-(trimethylsilyl)phenyl trifluoromethanesulfonate **1m** (0.234 g, 0.75 mmol) was treated with *tert*-butyl isocyanide **2a** (0.042 g, 57 μL, 0.5 mmol) in the presence of KF (0.087 g, 1.5 mmol) and 18-crown-6 (0.396 g, 1.5 mmol) in THF (2.0 mL) at 30 °C. To this reaction mixture was added H_2O (18 μL, 1 mmol) after 10 minutes. The mixture was then stirred at 30 °C for 16 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 92/08) of the crude mixture afforded *N*-(*ter*t-Butyl)-3-methylbenzamide **9m** and **9m'** as inseparable mixture of regioisomers in 1.2:1 ratio (white solid, 0.056 g, 59%).

R_f (pet. ether/EtOAc=80/20): 0.52; **^{1}H NMR (400 MHz, $CDCl_3$) of Major isomer;** δ 7.60 (d, *J* = 7.62 Hz, 2H), 7.19 (d, *J* = 7.74 Hz, 2H), 5.91 (bs, 1H), 2.37 (s, 3H), 1.45 (s, 9H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 167.03, 141.53, 133.15, 129.23, 126.81, 51.63, 29.03, 21.50. **^{1}H NMR (400 MHz, $CDCl_3$) of Minor isomer;** δ 7.53 (s, 1H), 7.47 (d, *J* = 5.91Hz, 1H), 7.27-7.25 (m, 2H), 5.91 (bs, 1H), 2.37 (s, 3H), 1.45 (s, 9H). **^{13}C NMR (100 MHz, $CDCl_3$) of Minor isomer;** δ 167.28, 138.43, 136.02, 131.91, 130.01, 128.46, 127.61, 123.77, 51.63, 29.00, 21.48. **HRMS**

[13] Mokhtary, M.; Najafizadehe, F. *J. Chem.* (*Hindawi*) **2012**, 9, 576.

calculated $[M+Na]^+$ for $C_{12}H_{17}ONNa$: 214.1202, found: 214.1200. **FTIR (cm^{-1}):** 3317, 3053, 2967, 1716, 1638, 1611, 1586, 1573, 1453, 1305, 1220, 1020, 743.

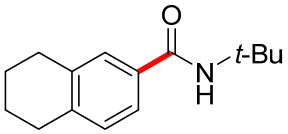
***N*-(*tert*-Butyl)-2,3-dihydro-1*H*-indene-5-carboxamide (9n)**



Following the general procedure, 6-(trimethylsilyl)-2,3-dihydro-1*H*-inden-5-yl trifluoromethanesulfonate **1o** (0.254 g, 0.75 mmol) was treated with *tert*-butyl isocyanide **2a** (0.042 g, 57 µL, 0.5 mmol) in the presence of KF (0.087 g, 1.5 mmol) and 18-crown-6 (0.396 g, 1.5 mmol) in THF (2.0 mL) at 30 °C. To this reaction mixture was added H_2O (18 µL, 1 mmol) after 10 minutes. The mixture was then stirred at 30 °C for 16 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 92/08) of the crude mixture afforded *N*-(*tert*-Butyl)-2,3-dihydro-1*H*-indene-5-carboxamide **9n** as a white solid (0.079 g, 73%).

***R*$_f$** (Pet. Ether/EtOAc=90/10): 0.34; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.57 (s, 1H), 7.49 (d, *J* = 7.6 Hz, 1H), 7.23 (d, *J* = 7.6 Hz, 1H), 5.94 (bs, 1H), 2.92 (t, *J* = 7.2 Hz, 4H), 2.13-2.06 (m, 2H), 1.46 (s, 9H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 167.53, 147.88, 144.77, 134.09, 124.96, 124.34, 122.88, 51.61, 32.91, 32.77, 29.04, 25.56. **HRMS** calculated $[M+Na]^+$ for $C_{14}H_{19}ONNa$: 240.1359, found: 240.1356. **FTIR (cm^{-1}):** 3053, 2986, 2362, 1733, 1654, 1575, 1518, 1482, 1364, 1083, 733

***N*-(*tert*-Butyl)-5,6,7,8-tetrahydronaphthalene-2-carboxamide (9o)**

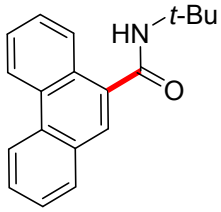


Following the general procedure, 3-(trimethylsilyl)-5,6,7,8-tetrahydronaphthalen-2-yl trifluoromethanesulfonate **1o** (0.132 g, 0.375 mmol) was treated with *tert*-butyl isocyanide **2a** (0.021 g, 29 µL, 0.25 mmol) in the presence of KF (0.044 g, 0.75 mmol) and 18-crown-6 (0.198 g, 0.75 mmol) in THF (1.0 mL) at 30 °C. To this reaction mixture was added H_2O (9 µL, 0.5 mmol) after 10 minutes. The mixture was then stirred at 30 °C for 16 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 92/08) of the crude mixture afforded *N*-(*tert*-butyl)-5,6,7,8-tetrahydronaphthalene-2-carboxamide **9o** as a white solid (0.036 g, 62%).

***R*$_f$** (pet. ether/EtOAc=80/20): 0.5; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.41 (m, 2H), 7.08 (d, *J* = 7.6 Hz, 1H), 5.92 (bs, 1H), 2.78 (s, 4H), 1.8 (s, 4H), 1.46 (s, 9H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 167.26, 140.79, 137.48, 133.06, 127.72, 123.69, 51.60, 29.49, 29.04, 23.14, 23.07. **HRMS**

calculated $[M+H]^+$ for $C_{15}H_{22}ON$: 232.1696, found: 232.1693. **FTIR (cm^{-1}):** 2966, 2928, 2859, 1637, 1610, 1572, 1493, 1391, 1276, 917, 815.

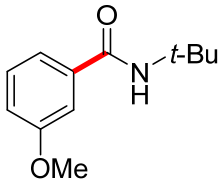
***N*-(*tert*-Butyl)phenanthrene-9-carboxamide (9p)**



Following the general procedure, 10-(trimethylsilyl)phenanthren-9-yl trifluoromethanesulfonate **1p** (0.299 g, 0.75 mmol) was treated with *tert*-butyl isocyanide **2a** (0.042 g, 57 µL, 0.5 mmol) in the presence of KF (0.087 g, 1.5 mmol) and 18-crown-6 (0.396 g, 1.5 mmol) in THF (2.0 mL) at 30 °C. To this reaction mixture was added H_2O (18 µL, 1 mmol) after 10 minutes. The mixture was then stirred at 30 °C for 16 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 92/08) of the crude mixture afforded *N*-(*tert*-butyl)phenanthrene-9-carboxamide **9p** as a white solid (0.113 g, 81%).

***R*$_f$** (pet. ether/EtOAc=80/20): 0.58; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.66-8.61 (m, 2H), 8.30-8.28 (m, 1H), 7.84 (d, *J* = 7.87Hz, 1H), 7.76 (s, 1H), 7.67-7.58 (4H, m), 6.03 (bs, 1H), 1.56 (s, 9H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ169.31, 134.83, 130.59, 127.07, 125.59, 122.90, 122.67, 52.15, 28.98. **HRMS** calculated $[M+Na]^+$ for $C_{19}H_{19}ONNa$: 300.1359, found: 300.1355. **FTIR (cm^{-1}):** 3282, 3057, 2967, 2927, 1638, 1594, 1530, 1506, 1449, 1315, 1271, 1165, 1039, 949, 895, 790, 702.

***N*-(*tert*-Butyl)-3-methoxybenzamide (9q)**[14]

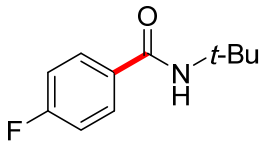


Following the general procedure, 2-methoxy-6-(trimethylsilyl)phenyl trifluoromethanesulfonate **1q** (0.246 g, 0.75 mmol) was treated with *tert*-butyl isocyanide **2a** (0.042 g, 57 µL, 0.5 mmol) in the presence of KF (0.087 g, 1.5 mmol) and 18-crown-6 (0.396 g, 1.5 mmol) in THF (2.0 mL) at 30 °C. To this reaction mixture was added H_2O (18 µL, 1 mmol) after 10 minutes. The mixture was then stirred at 30 °C for 16 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 92/08) of the crude mixture afforded N-(*tert*-butyl)-3-methoxybenzamide **9q** as a white solid (0.086 g, 83%).

[14] Sephton, M. A.; Emerson, C. R.; Zakharov, L. N.; Blakemore, P. R. *Chem. Commun.* **2010**, *46*, 2094.

***R*$_f$** (pet. ether/EtOAc=80/20): 0.34; **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.33 (s, 1H), 7.29 (d, *J* = 7.89 Hz, 1H), 7.21(d, *J* = 7 .51 Hz, 1H), 7.02 (dd, J_1 = 2.23Hz, J_2 = 8.12 Hz, 1H), 5.94 (bs, 1H), 3.84 (s, 3H), 1.47 (s, 9H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ 166.85, 159.94, 137.54, 129.55, 118.52, 117.49, 121.27, 55.58, 51.76, 28.99. **HRMS** calculated $[M+Na]^+$ for $C_{12}H_{17}O_2NNa$: 230.1152, found: 230.1149. **FTIR (cm^{-1}):** 2965, 2918, 1849, 1732, 1703, 1644, 1582, 1486, 1392, 1275, 1149, 946, 750.

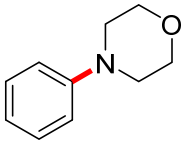
_N_-(_tert_-Butyl)-4-fluorobenzamide (9r)[11]



Following the general procedure, 5-fluoro-2-(trimethylsilyl)phenyl trifluoromethanesulfonate **1r** (0.237 g, 0.75 mmol) was treated with *tert*-butyl isocyanide **2a** (0.042 g, 57 µL, 0.5 mmol) in the presence of KF (0.087 g, 1.5 mmol) and 18-crown-6 (0.396 g, 1.5 mmol) in THF (2.0 mL) at 30 °C. To this reaction mixture was added H_2O (18 µL, 1 mmol) after 10 minutes. The mixture was then stirred at 30 °C for 16 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc = 92/08) of the crude mixture afforded *N*-(*tert*-butyl)-4-fluorobenzamide **9r** as a white solid (0.060 g, 61%). The regioisomer ratio (12:1) was calculated by ^{1}H NMR spectroscopy of the crude reaction mixture.

***R*$_f$** (Pet. Ether/EtOAc=90/10): 0.30; **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.72-7.69 (m, 2H), 7.06 (t, 2H), 5.96 (br s, 1H), 1.45 (s, 9H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ 166.01, 164.55 (d, *J* = 251.2 Hz), 132.16 (d, *J* = 3.13 Hz), 129.12 (d, *J* = 8.8 Hz), 115.55 (d, *J* = 21.77 Hz), 51.82, 28.94. **HRMS** calculated $[M+Na]^+$ for $C_{11}H_{14}ONFNa$: 218.0952, found: 218.0949. **FTIR (cm^{-1}):** 3310, 3052, 2967, 2934, 1732, 1657, 1633, 1602, 1547, 1500, 1454, 1392, 1318, 1264, 1095, 703.

4-Phenylmorpholine[15]



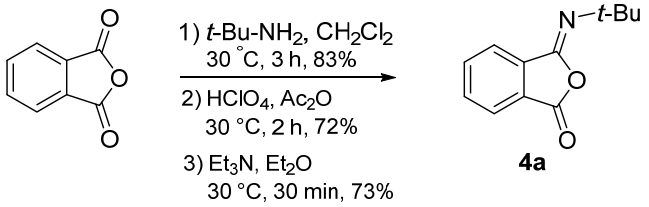
Following the general procedure, 2-(trimethylsilyl)phenyl trifluoromethane sulfonate **1a** (0.224 g, 182 µL, 0.75 mmol) was treated with 4-(2-isocyanoethyl)morpholine **2g** (0.070 g, 69 µL, 0.5 mmol) in the presence of KF (0.087 g, 1.5 mmol) and 18-crown-6 (0.396 g, 1.5 mmol) in THF (2.0 mL) at 30 °C. To this reaction mixture was added H_2O (18 µL, 1 mmol) after 10 minutes. The mixture was then stirred at 30 °C for 16 h. Evaporation of solvent followed by column chromatography (Pet. ether/EtOAc

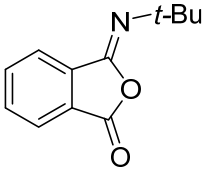
[15] Nguyen, M. H.; Smith, A. B., III. *Org. Lett*. **2013**, *15*, 4872.

= 92/08) of the crude mixture afforded 4-phenylmorpholine **10** as a pale brown solid (0.046 g, 56%).

***R*$_f$** (Pet. ether/EtOAc=90/10): 0.56; **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.71 (d, *J* = 7.2 Hz, 2H), 7.32-7.28 (m, 2H), 6.96-6.91 (m, 3H), 3.88 (t, *J* = 4.7 Hz, 4H), 3.17 (t, *J* = 4.8 Hz, 4H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ 151.30, 129.30, 120.26, 115.88, 67.01, 49.52.

3-(*tert*-Butylimino)isobenzofuran-1(3*H*)-one (4a)





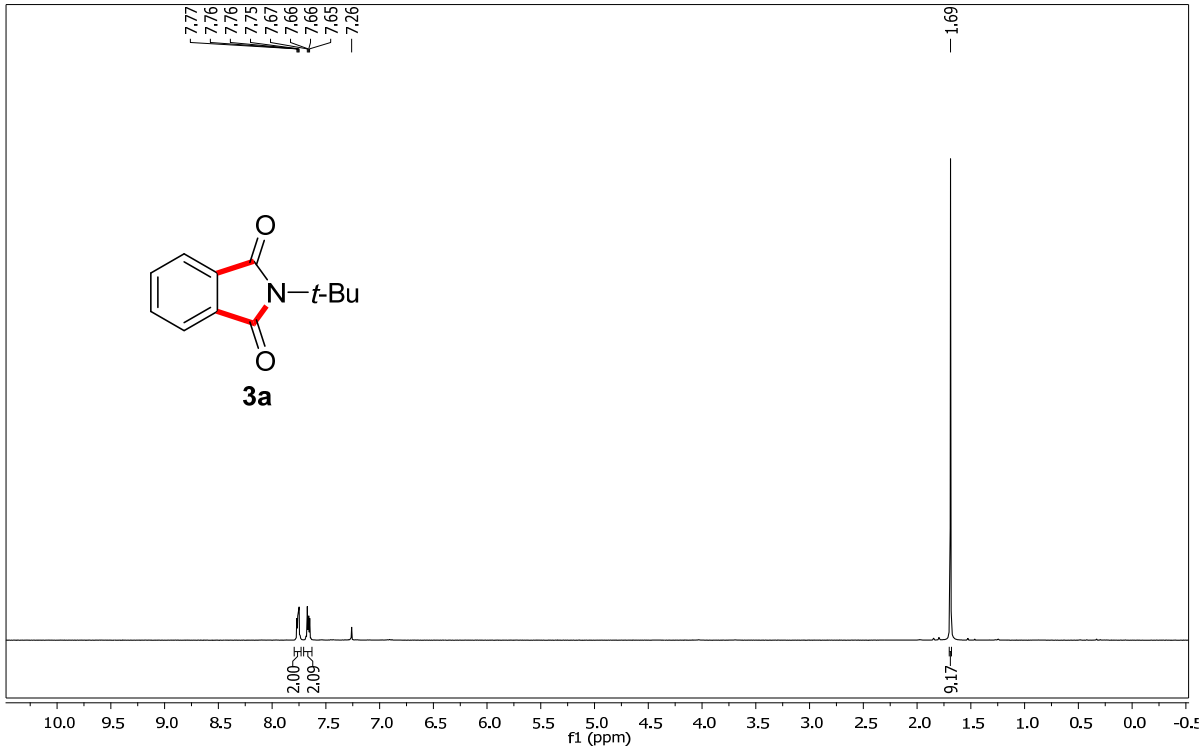
3-(*tert*-Butylimino)isobenzofuran-1(3*H*)-one was synthesized following the literature procedures.[16] Initially the phthalic anhydride (5 g, 1.0 equiv) was dissolved in 40 ml CH_2Cl_2. To the stirring solution *tert*-butylamine (7.5 mL, 1.0 equiv) was added slowly at 30 °C, and the reaction mixture was stirred for 3 h, then the precipitate was filtered and washed with pet. ether, to obtain the phthalamic acid derivative as a white solid (6.2 gm, 83% yield). The perchloric acid (3.1 mL) was added slowly to the suspension of phthalamic acid (5.5 gm, 25 mmol), in acetic anhydride (30 ml). The mixture was stirred at 30 °C for 2 h, then the precipitate was filtered and washed with diethyl ether to obtain the phthalisoimidium perchlorate as a white solid (5.4 gm, 72%). To the phthalisoimidium salt (3.0 g, 1.0 equiv) added 40 mL diethyl ether, followed by slow addition of triethyl amine (1.4 mL, 1.0 equiv), and then stirred for 30 min to obtain the phthalisoimide derivative **4a** as a pale brown solid (1.4 g, 73%).

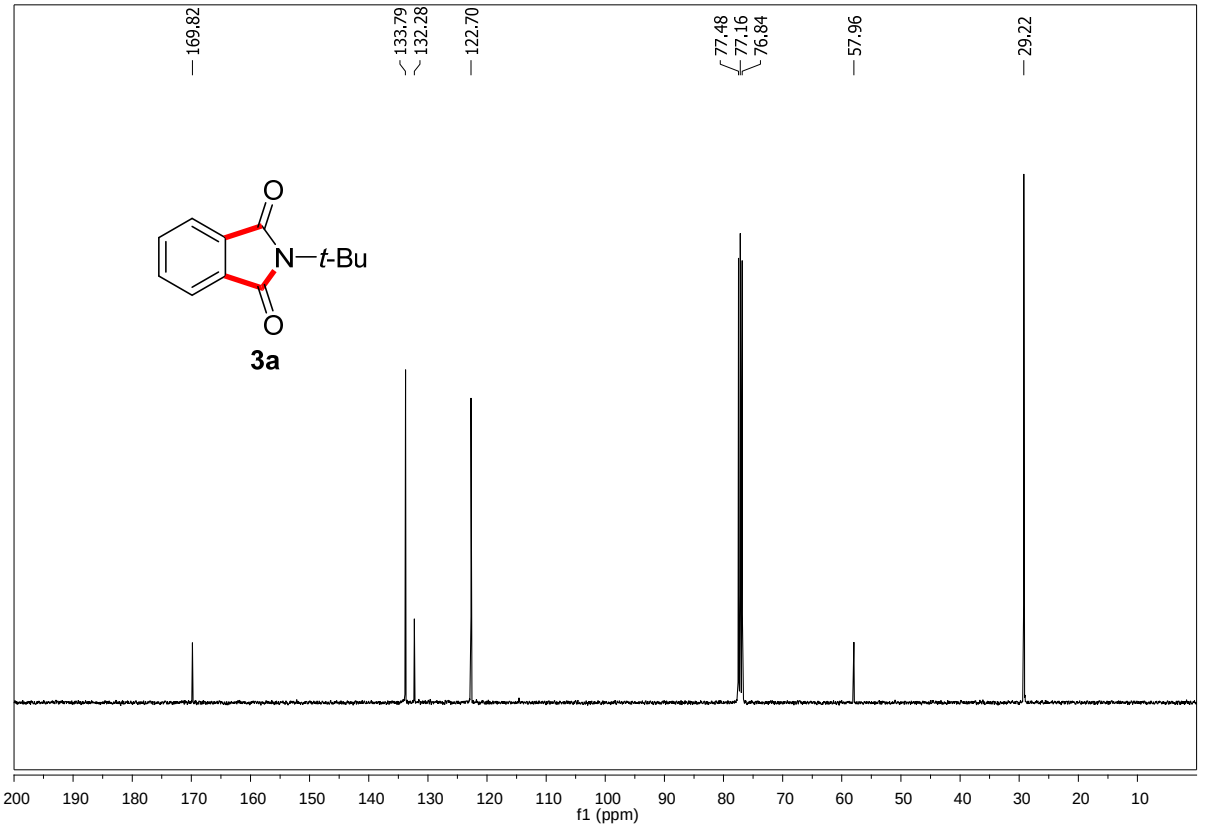
***R*$_f$** (Pet. Ether/EtOAc=95/05): 0.28; **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.91-7.89 (m, 2H), 7.77-7.73 (m, 1H), 7.69-7.67 (m, 1H), 1.46 (s, 9H). **^{13}C NMR (100 MHz, CDCl$_3$)** δ 165.67, 144.54, 137.86, 135.13, 132.42, 127.70, 124.96, 123.36, 56.22, 30.50. **FTIR (cm^{-1}):** 2971, 1804, 1775, 1709, 1467, 1213, 1095, 917, 890, 692.

[16] (a) Verbicky, J. W., Jr.; Williams, L. *J. Org. Chem*. **1981**, *46*, 175. (b) Boyd, G. B.; Monteil, R. L. *J. Chem. Soc., Perkin Trans 1*, **1978**, 1338.

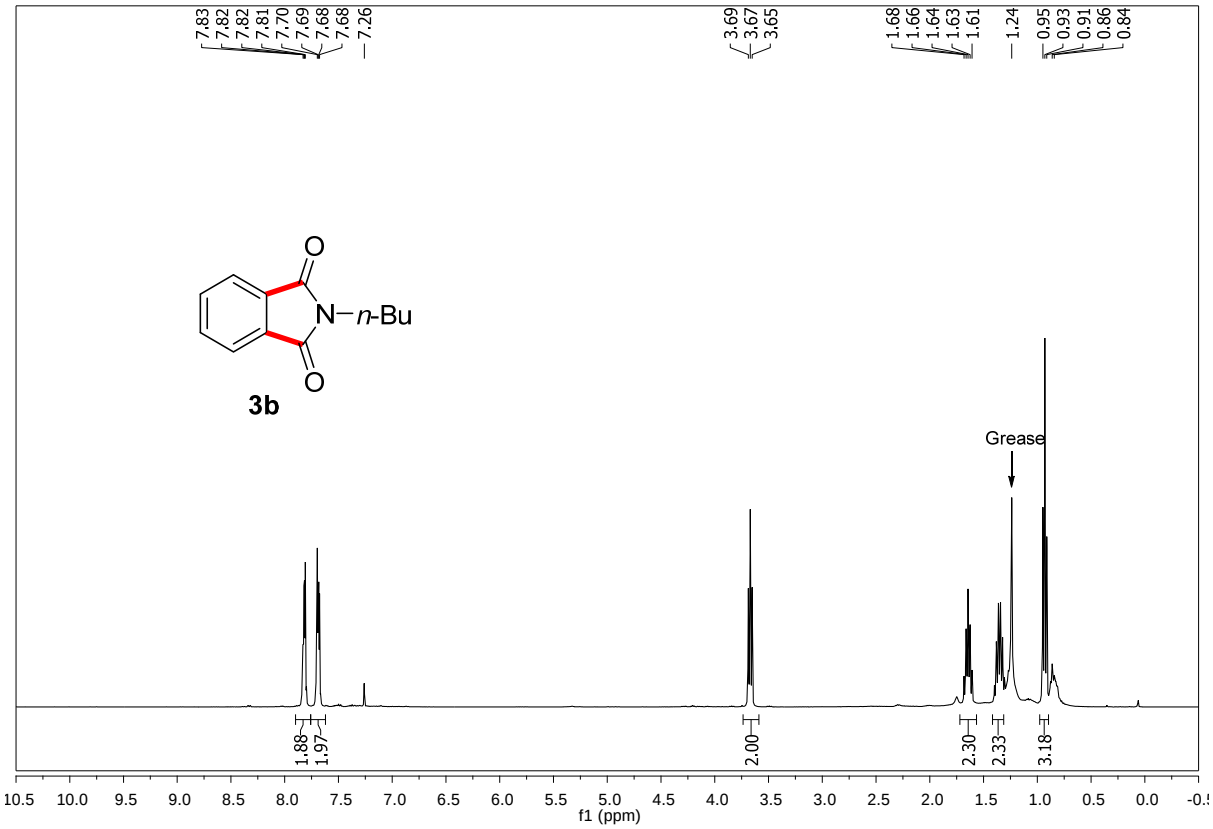
8. ^{1}H and ^{13}C NMR Spectra of Phthalimides and Aromatic Amides

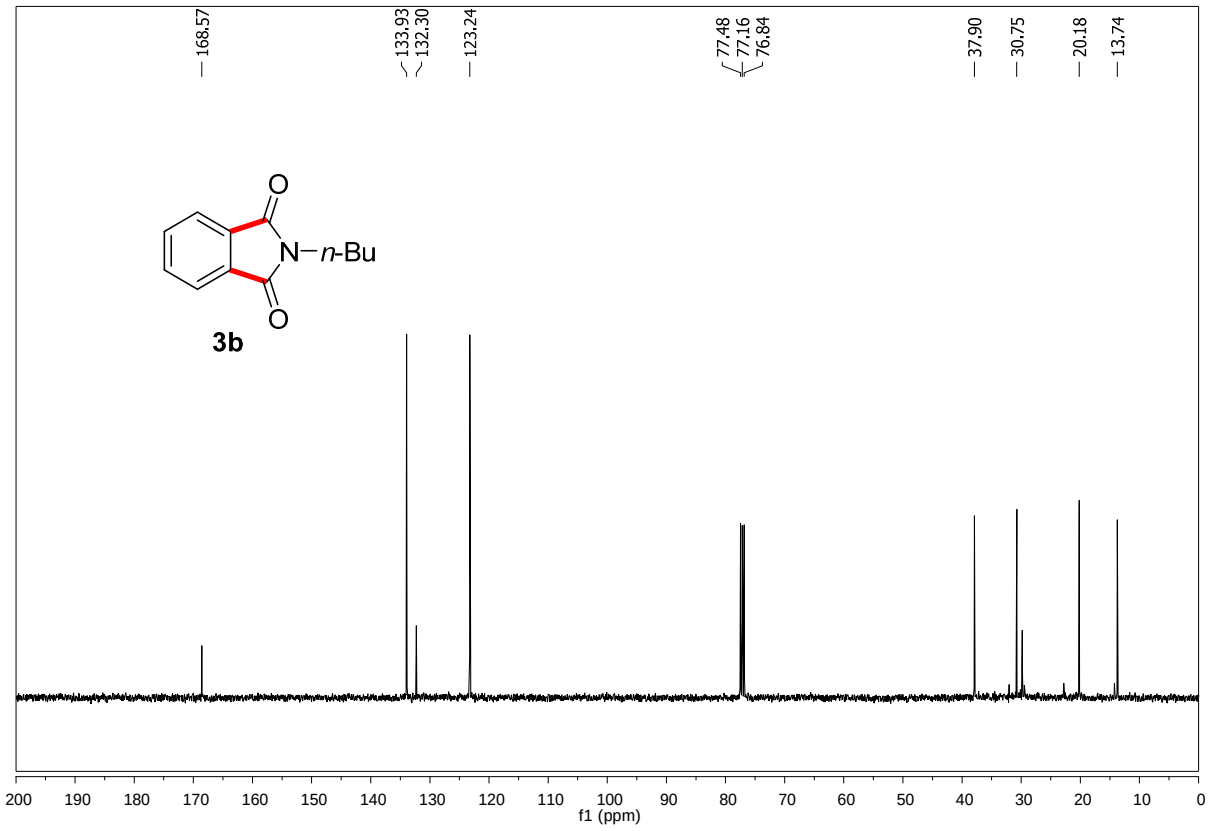
2-(*tert*-Butyl)isoindoline-1,3-dione (3a)



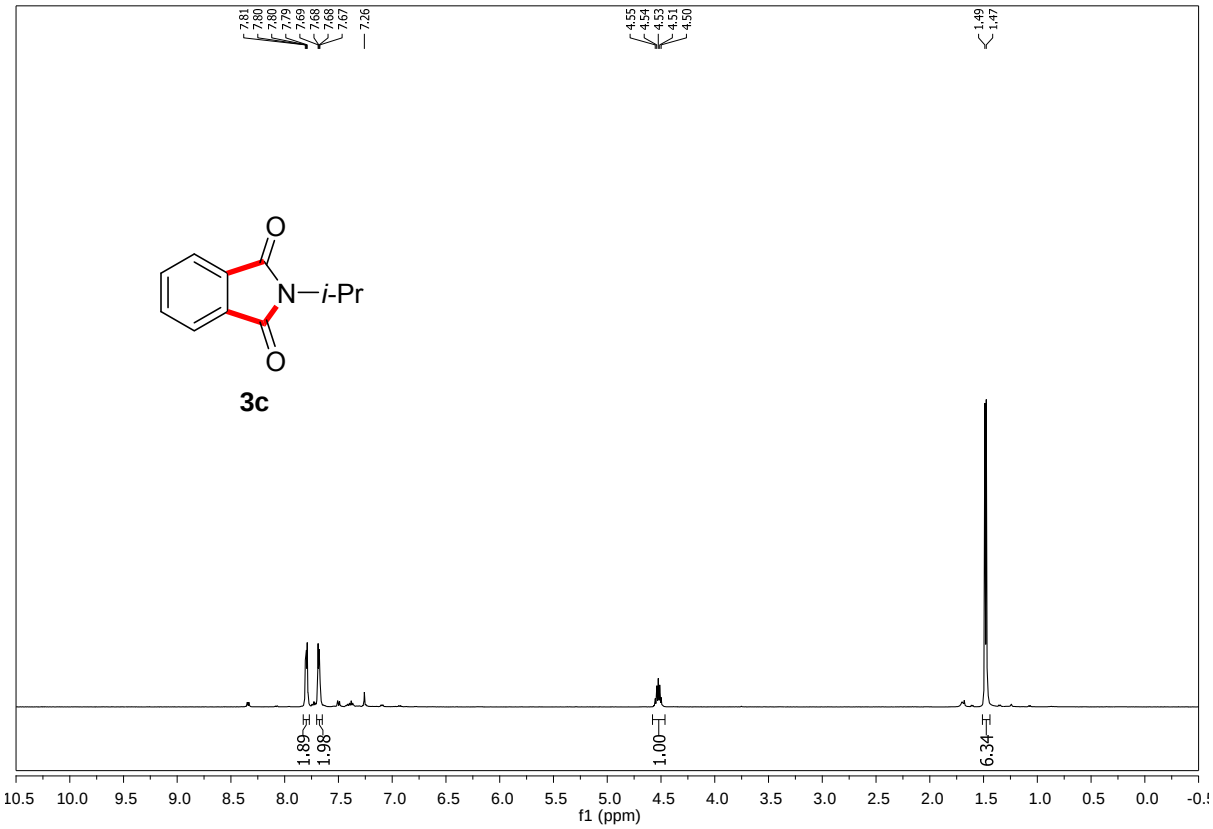


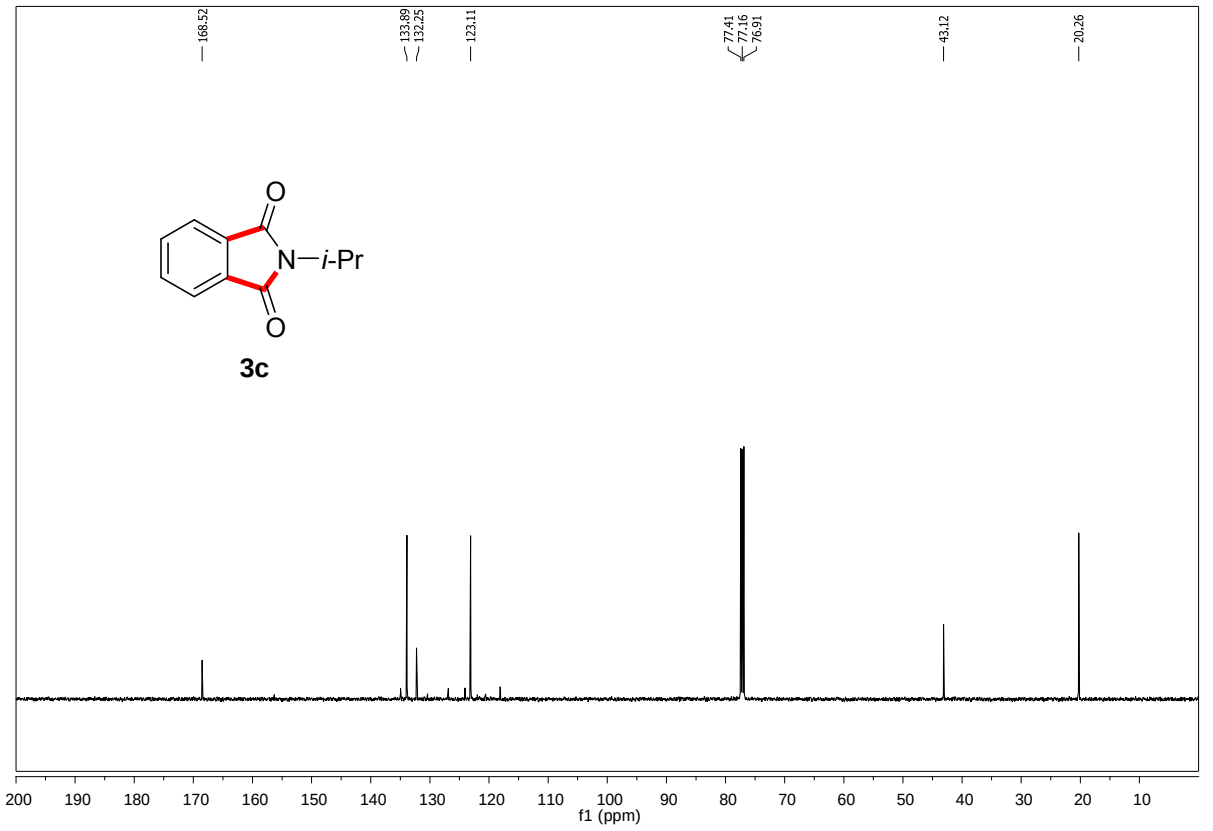
2-Butylisoindoline-1,3-dione (3b)



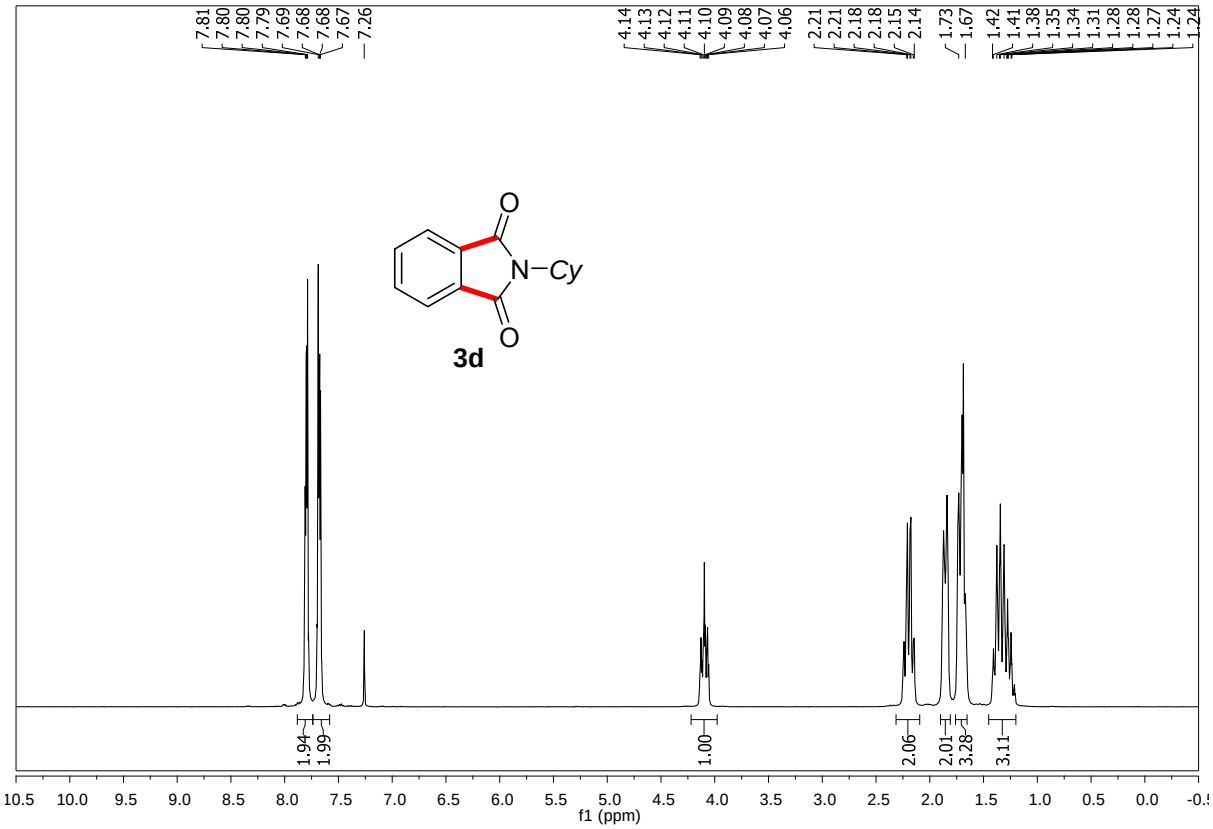


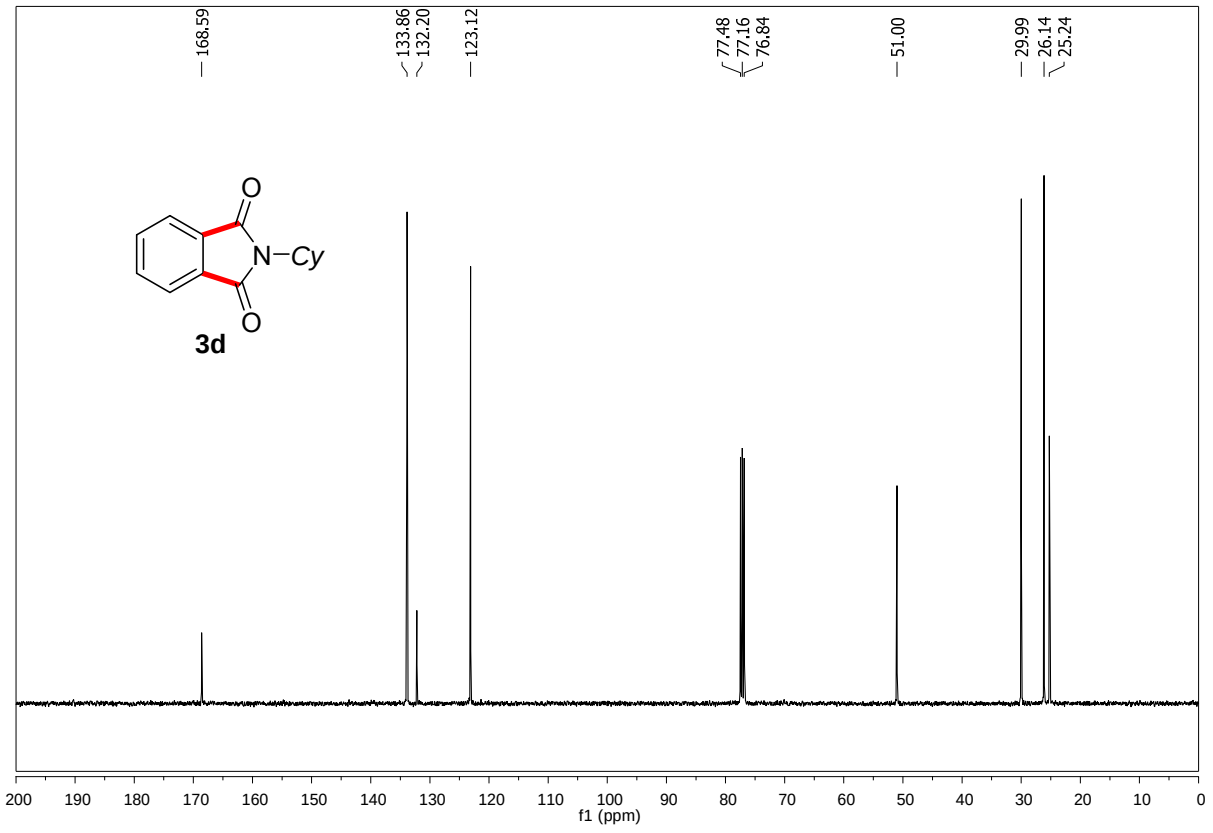
2-Isopropylisoindoline-1,3-dione (3c)



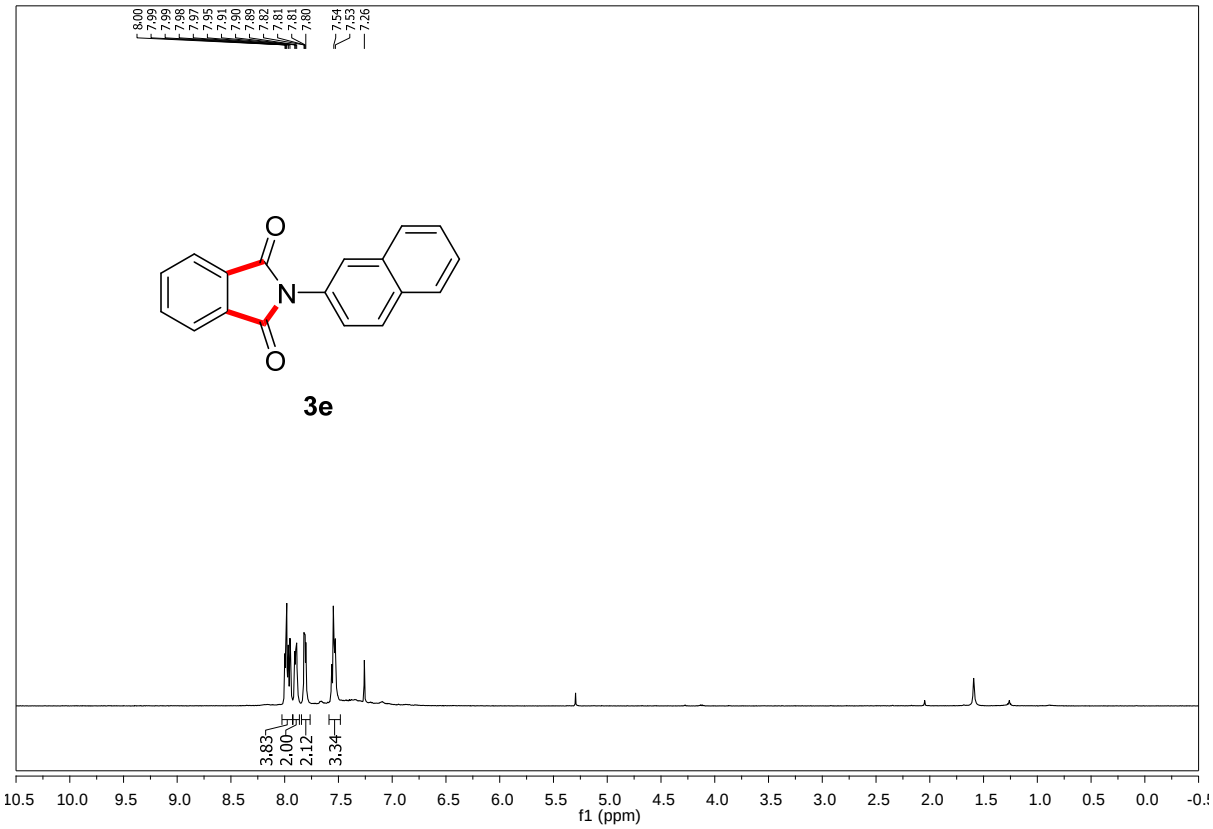


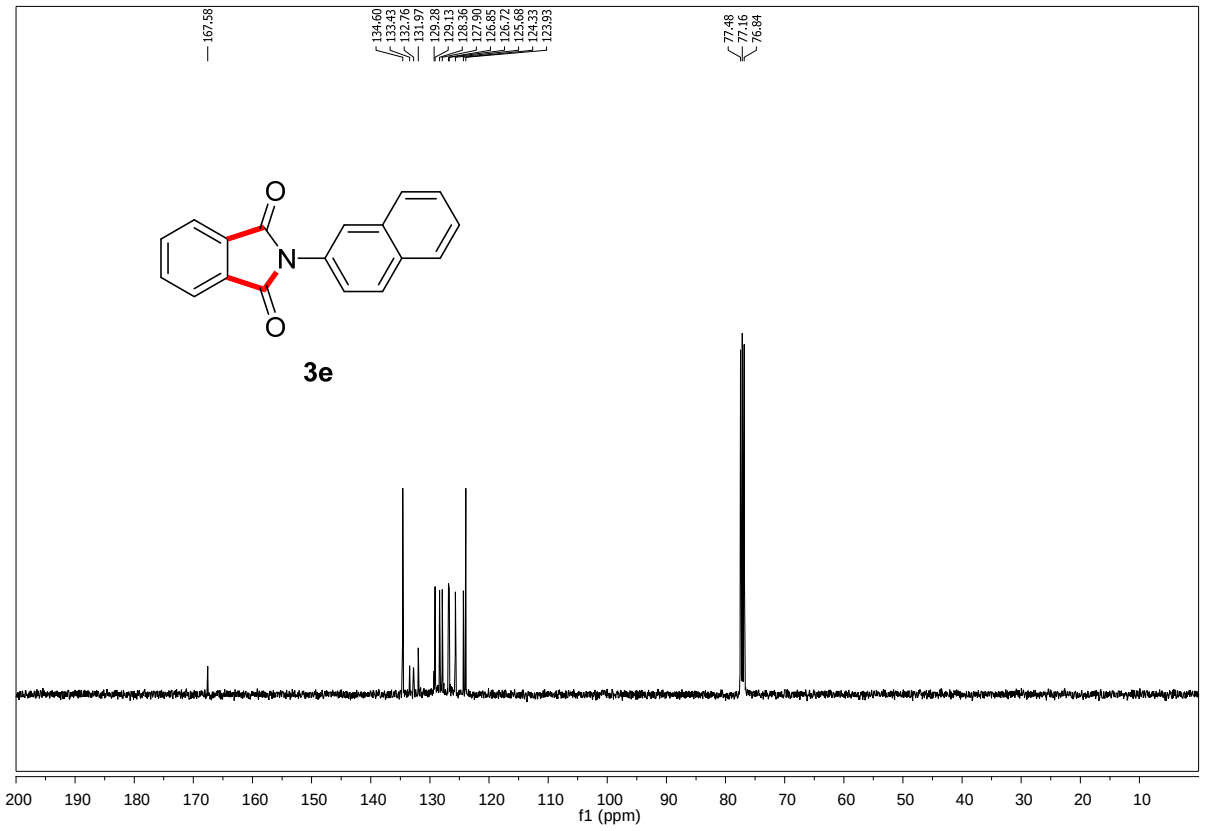
2-cyclohexylisoindoline-1,3-dione (3d)



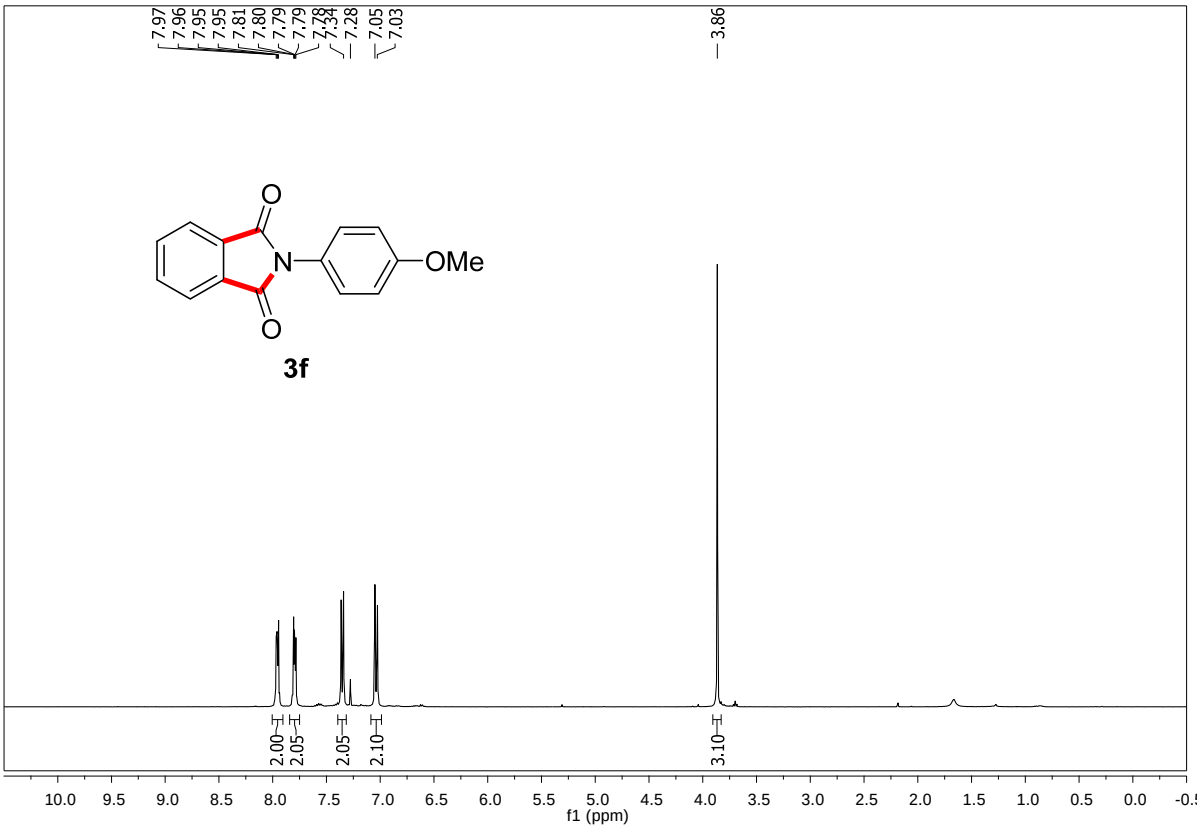


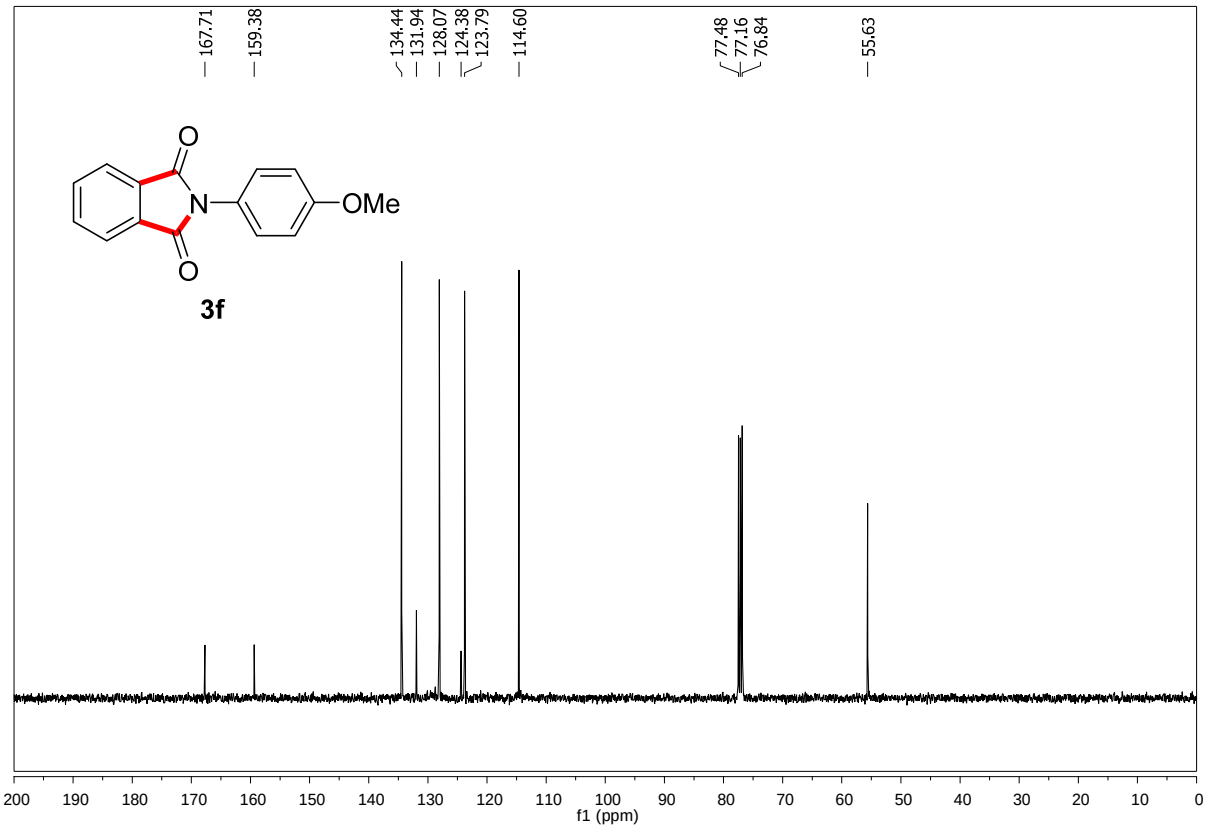
2-(Naphthalen-2-yl)isoindoline-1,3-dione (3e)



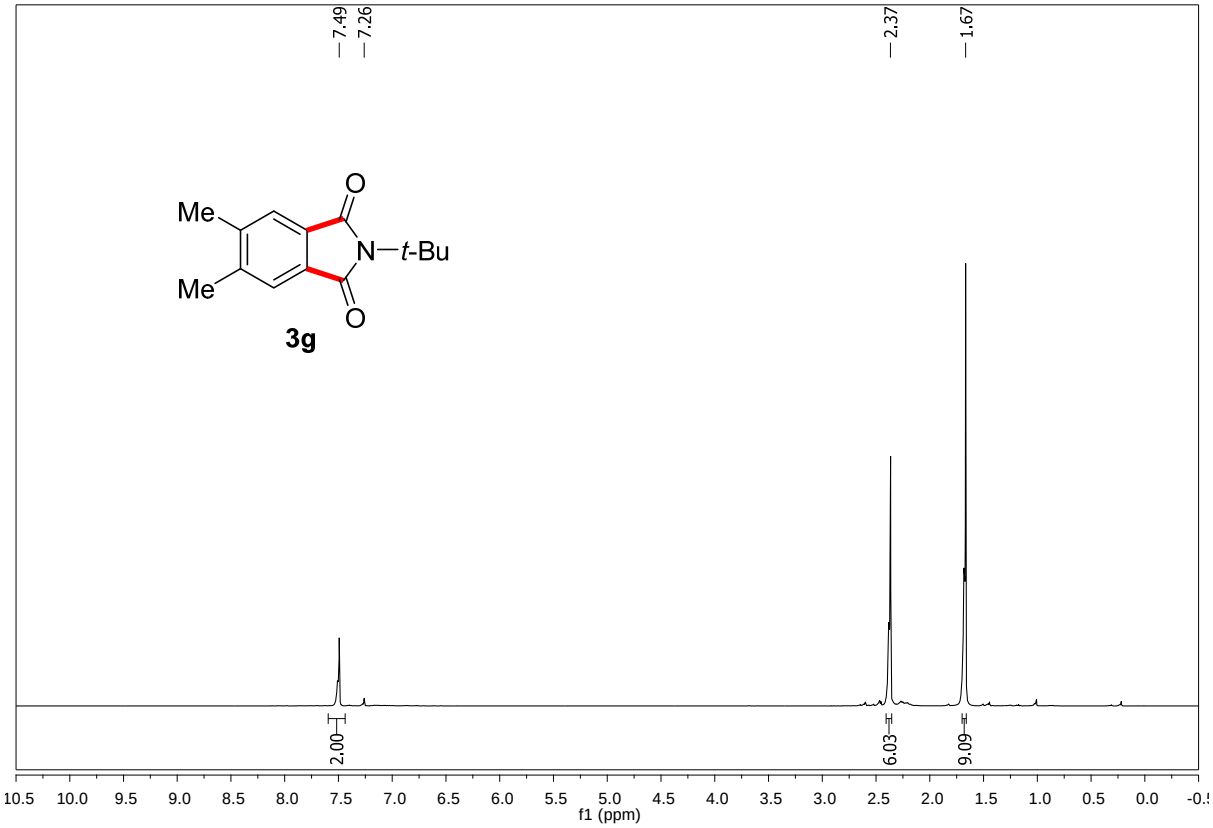


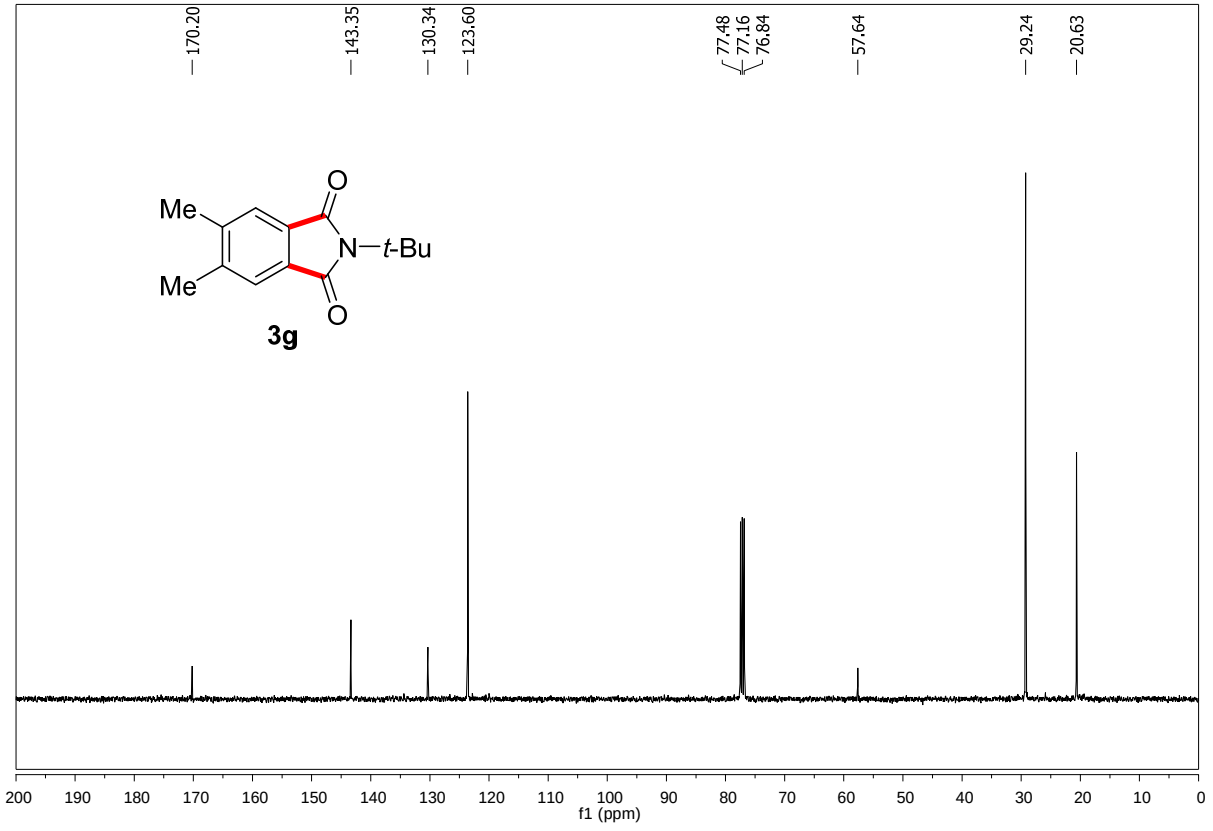
2-(4-Methoxyphenyl)isoindoline-1,3-dione (3f)



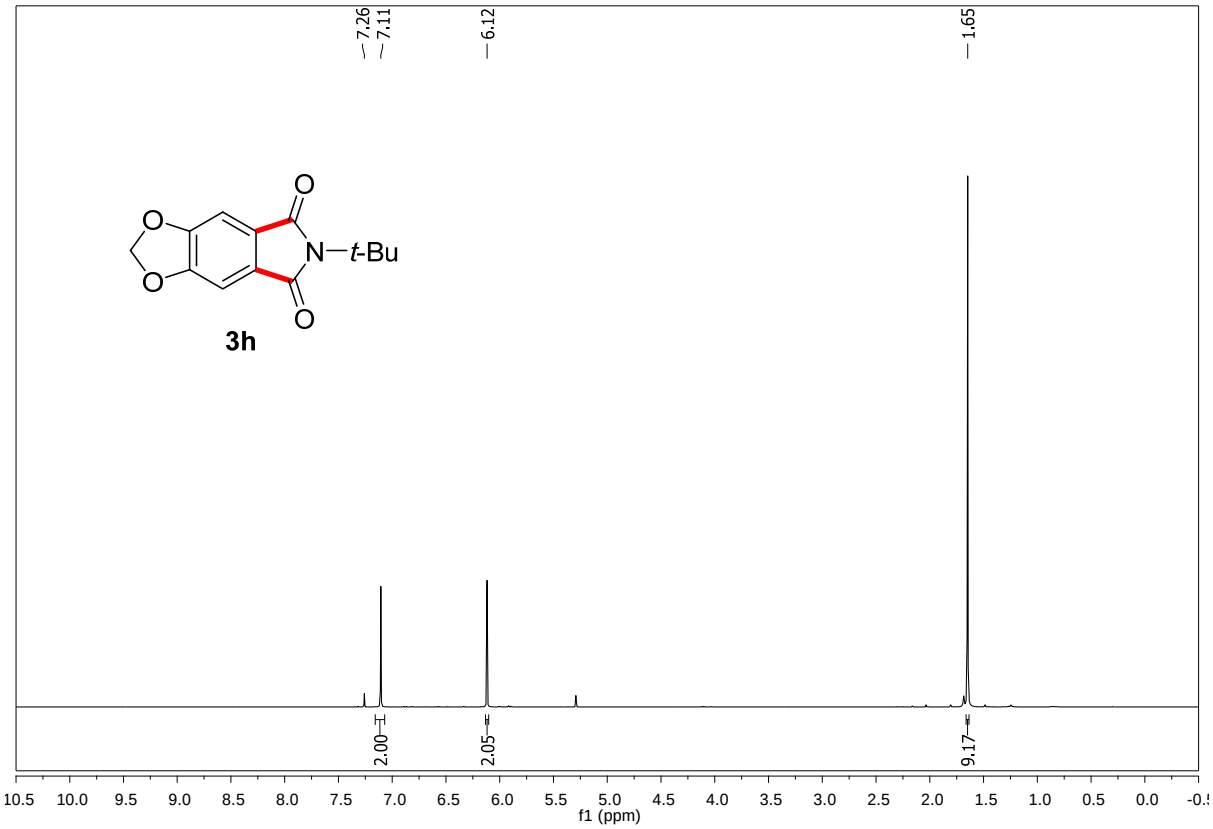


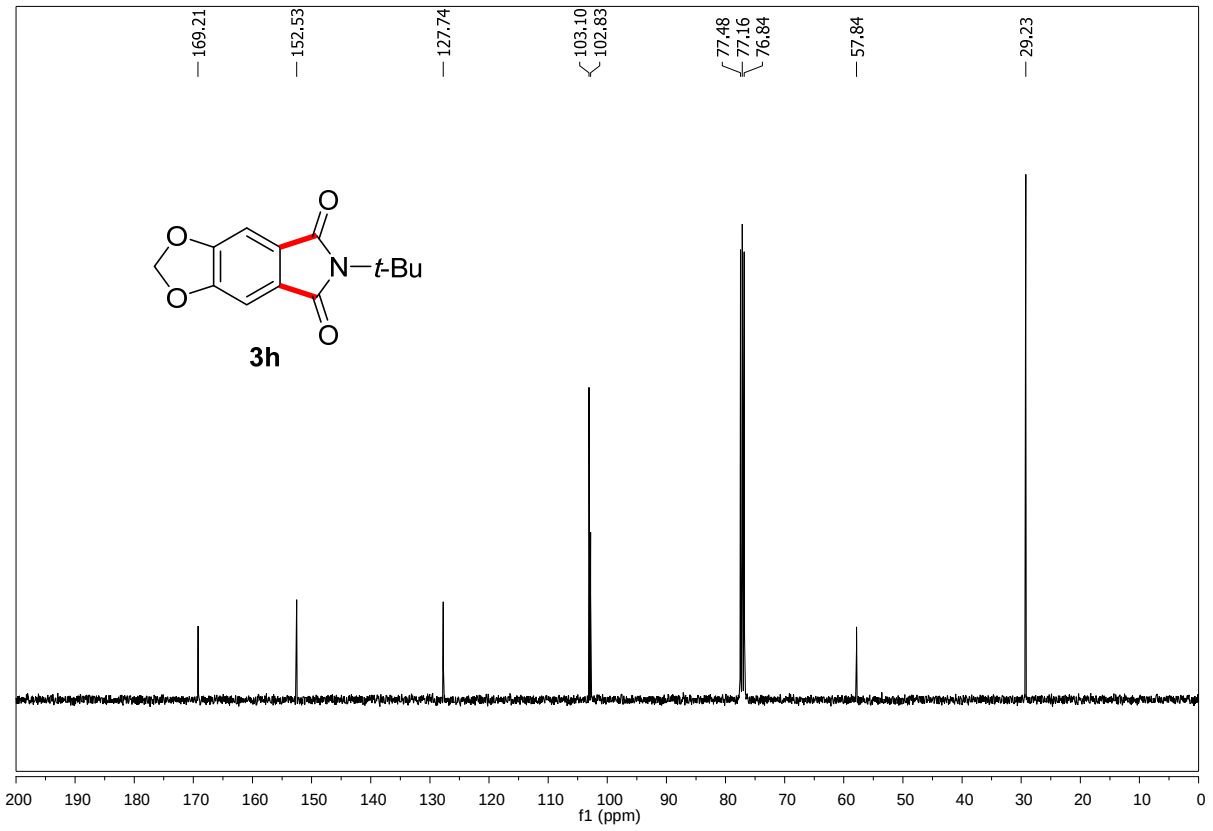
2-(*tert*-Butyl)-5,6-dimethylisoindoline-1,3-dione (3g)



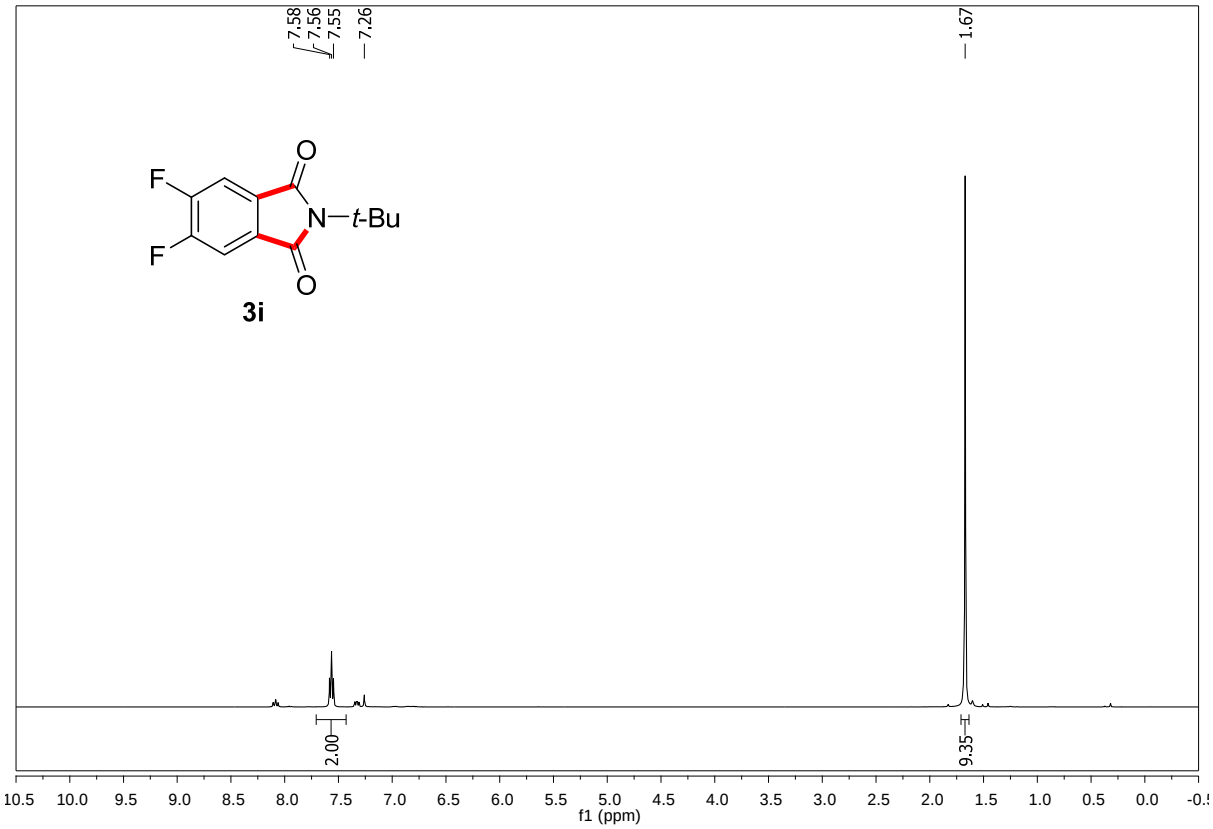


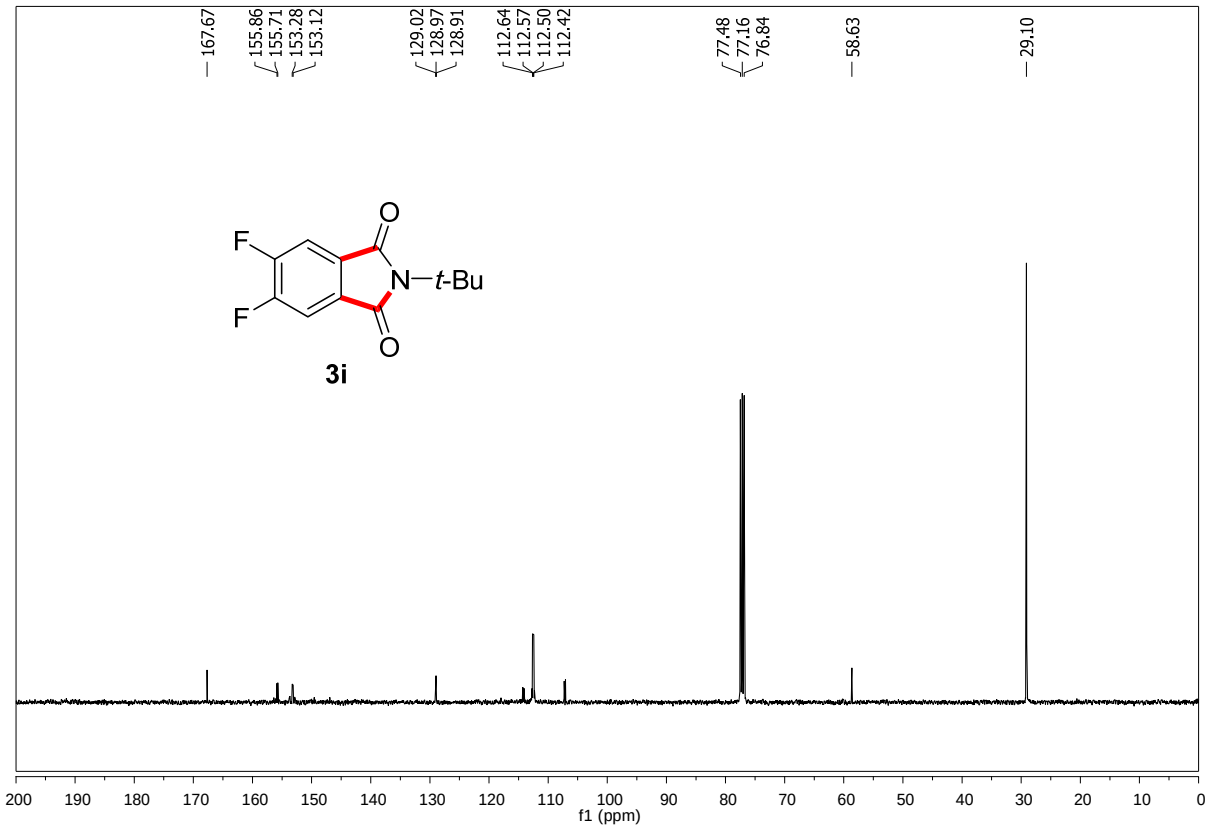
6-(*tert*-Butyl)-5*H*-[1,3]dioxolo[4,5-*f*]isoindole-5,7(6*H*)-dione (3h)



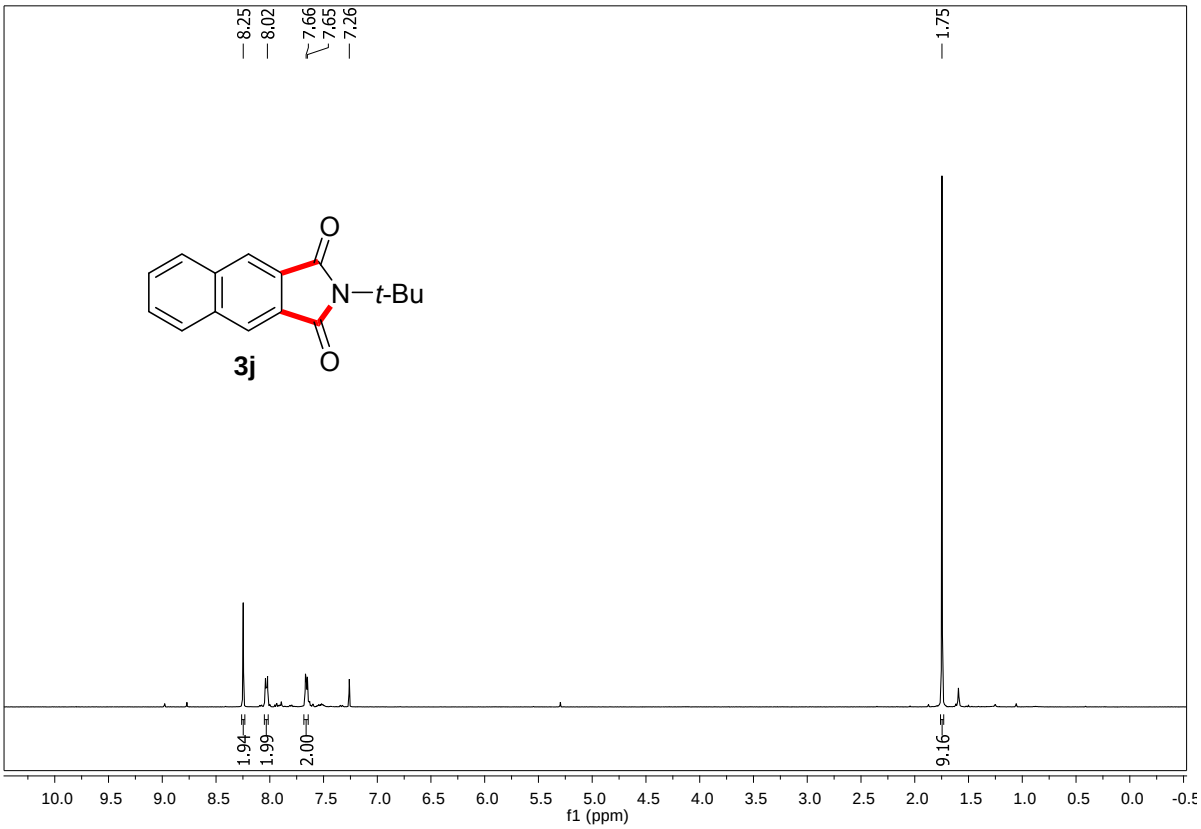


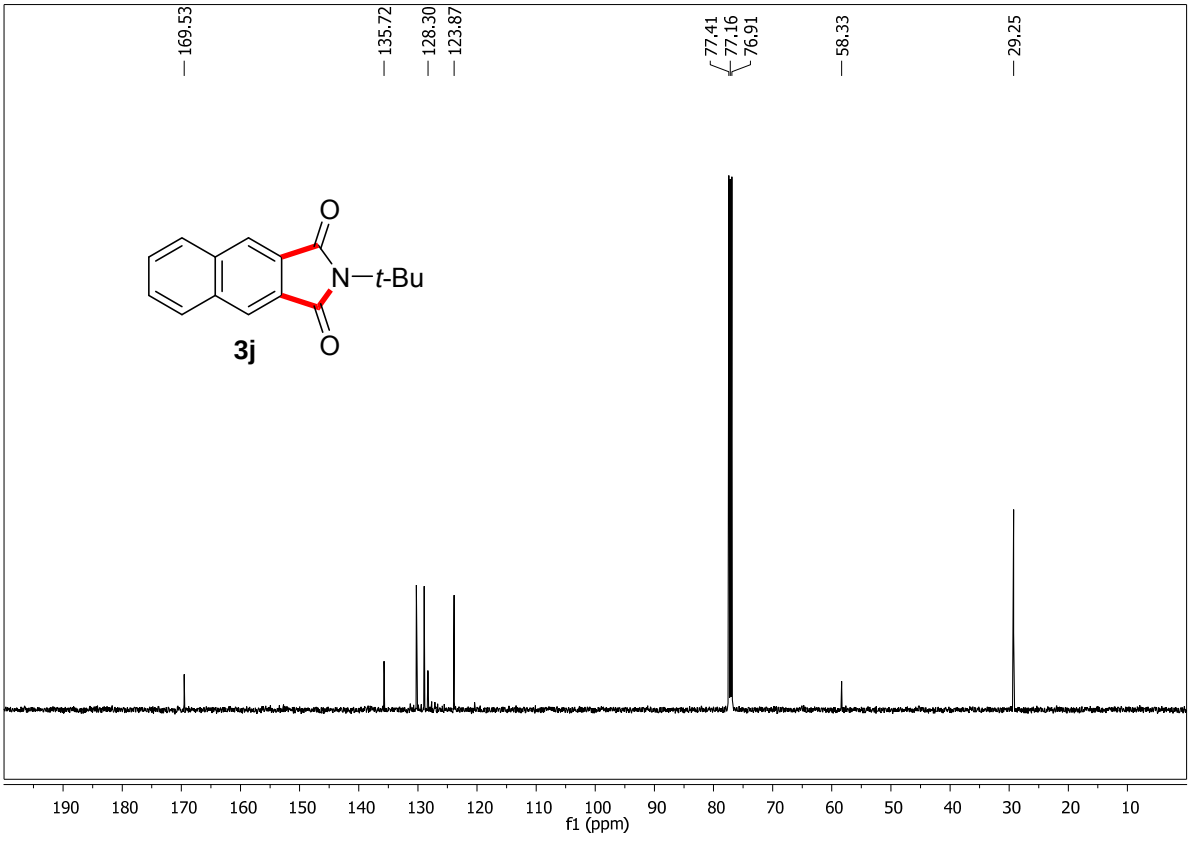
2-(*tert*-Butyl)-5,6-difluoroisoindoline-1,3-dione (3i)



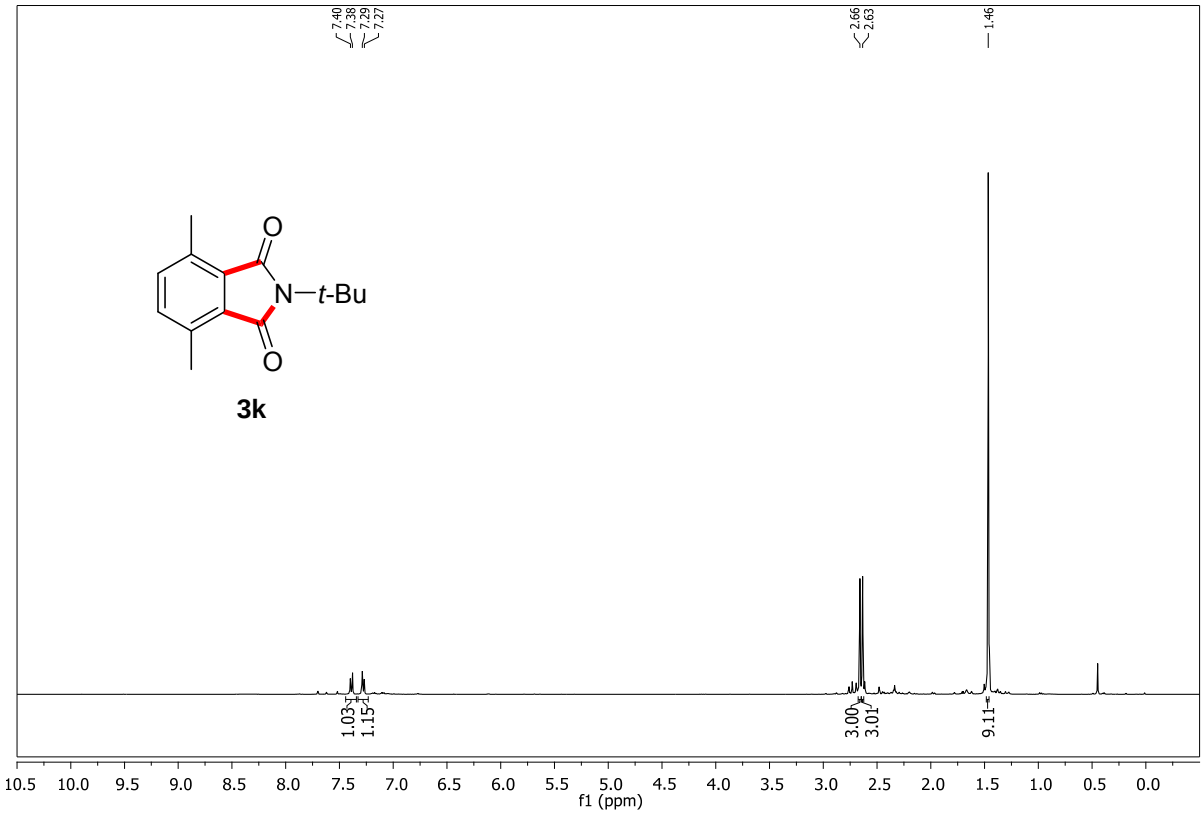


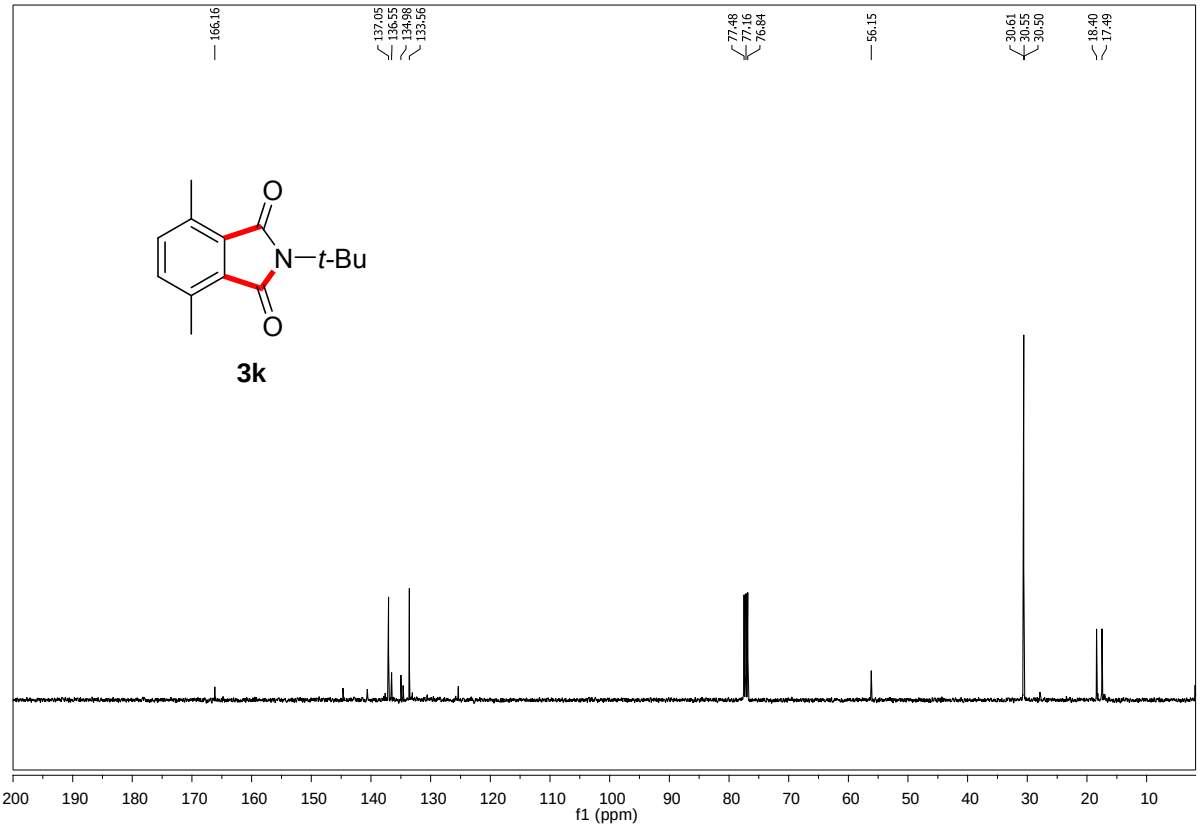
2-(*tert*-Butyl)-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (3j)



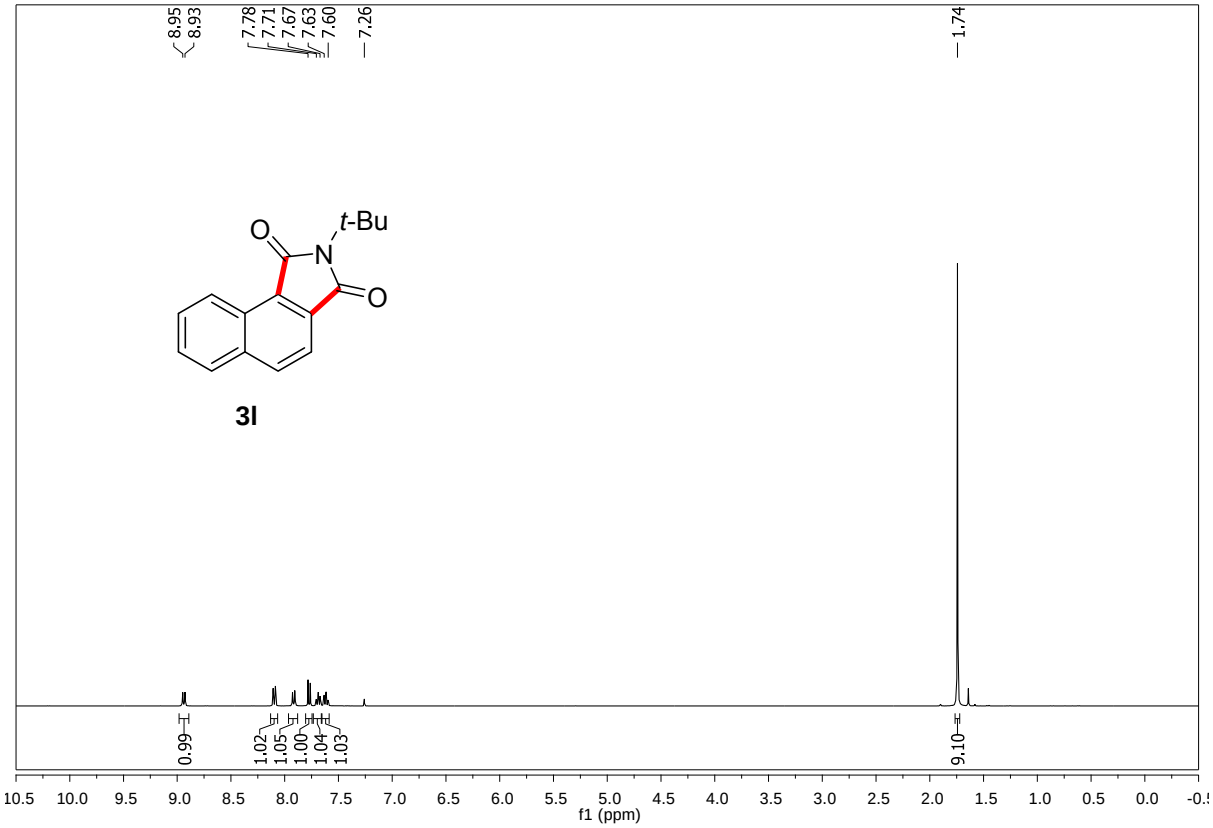


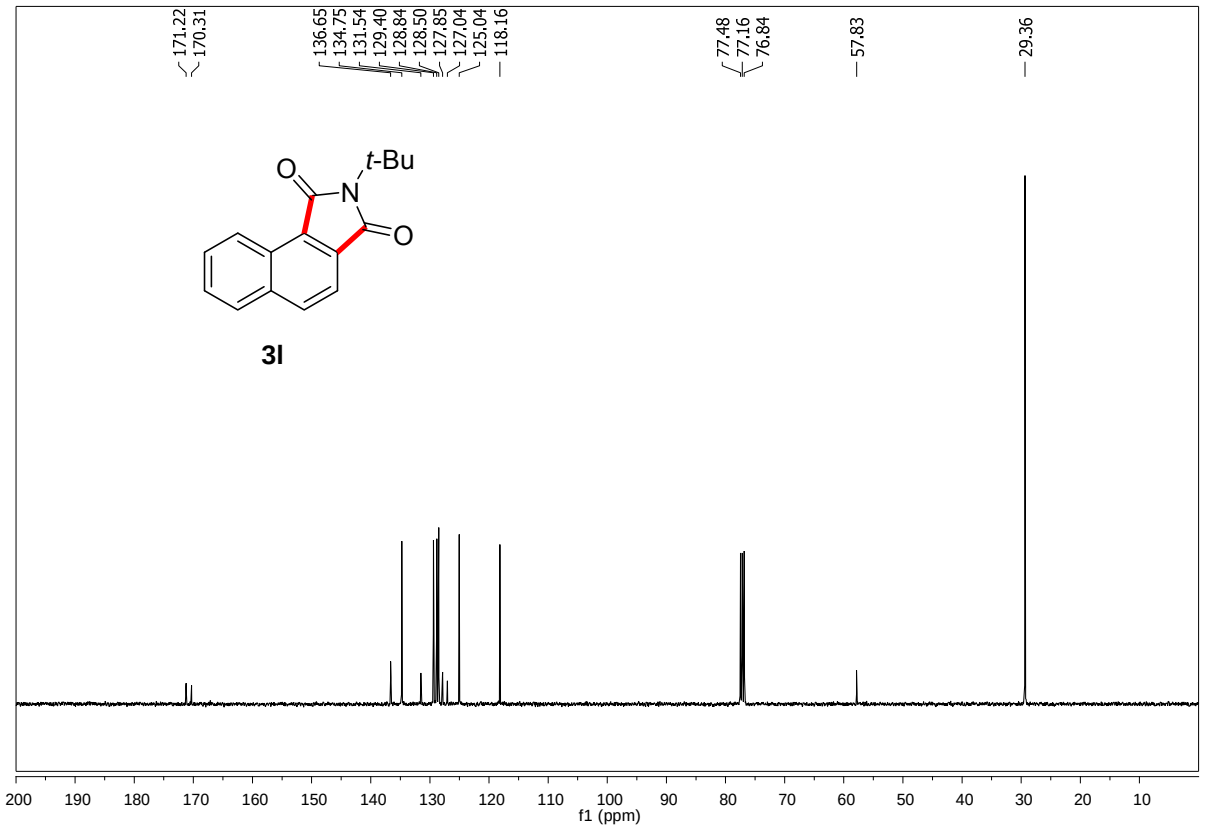
2-(*tert*-Butyl)-4,7-dimethylisoindoline-1,3-dione (3k)



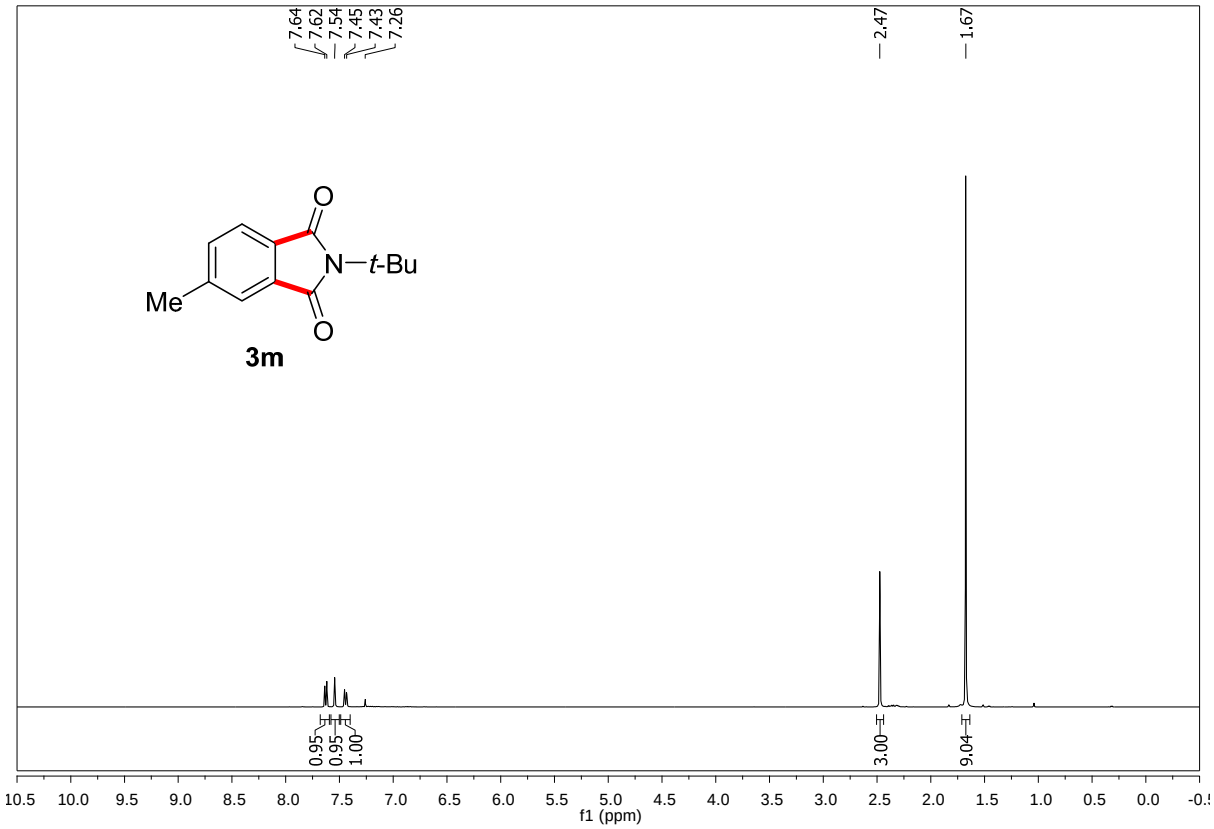


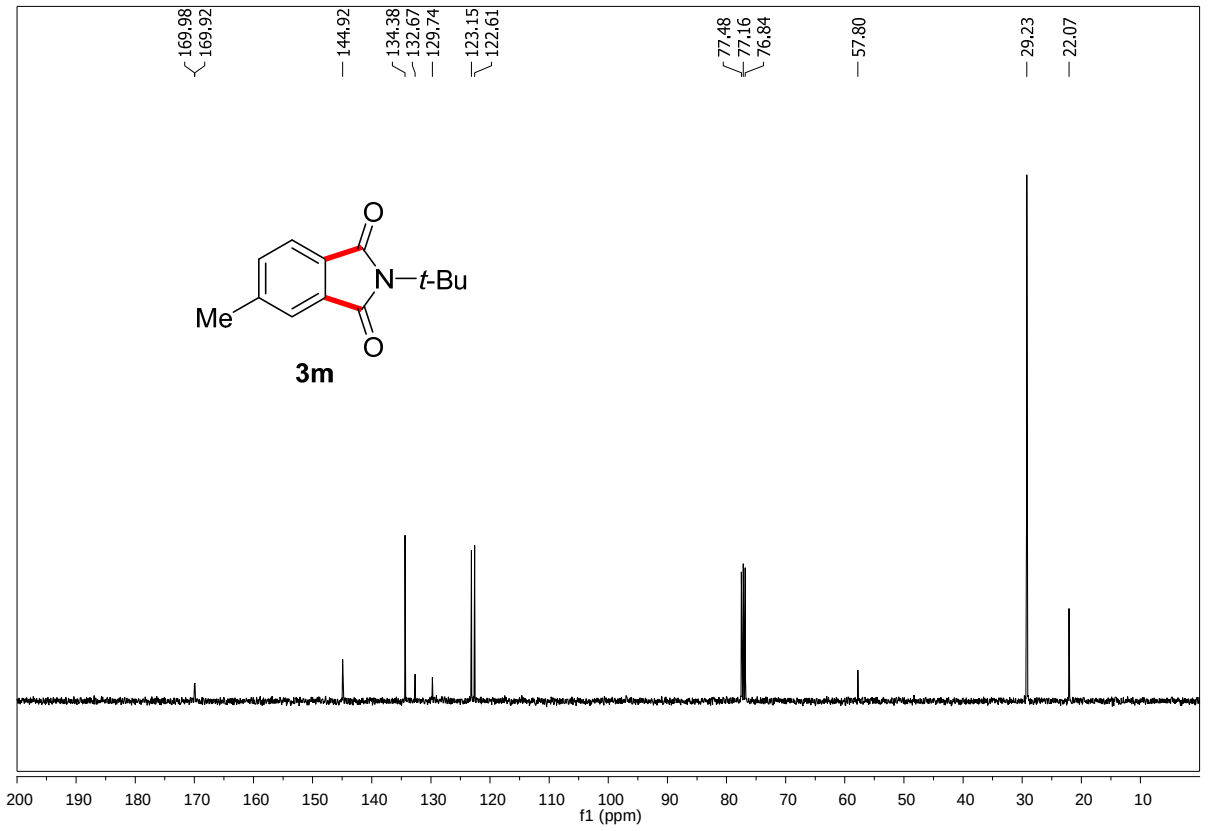
2-(*tert*-Butyl)-1*H*-benzo[*e*]isoindole-1,3(2*H*)-dione (3l)



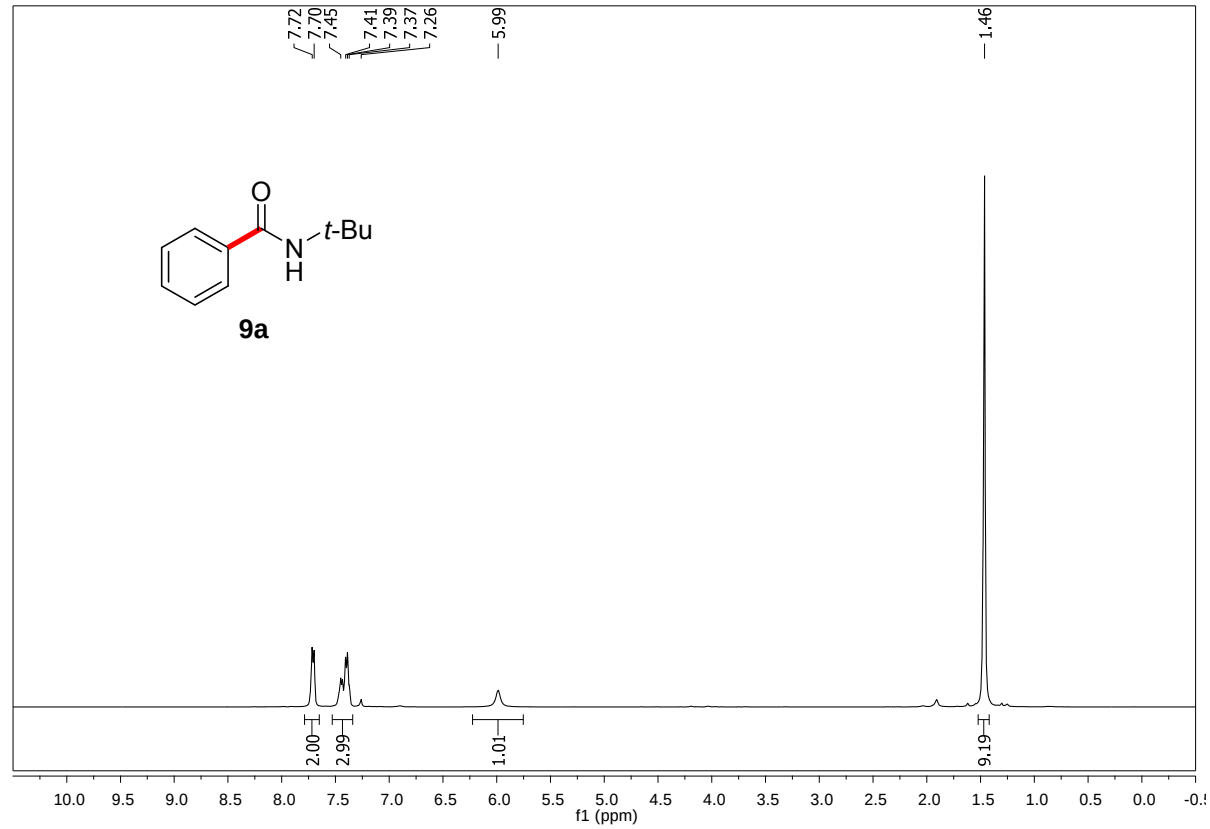


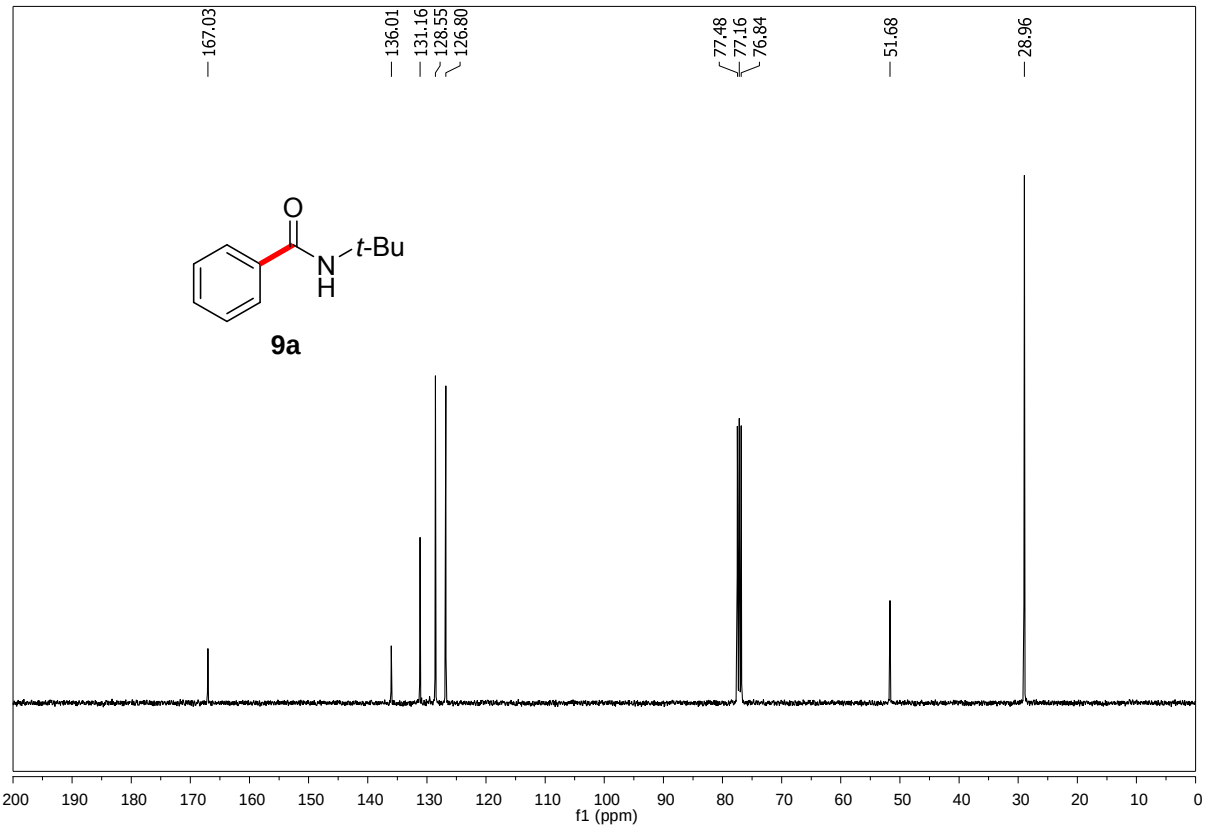
2-(*tert*-Butyl)-5-methylisoindoline-1,3-dione (3m)



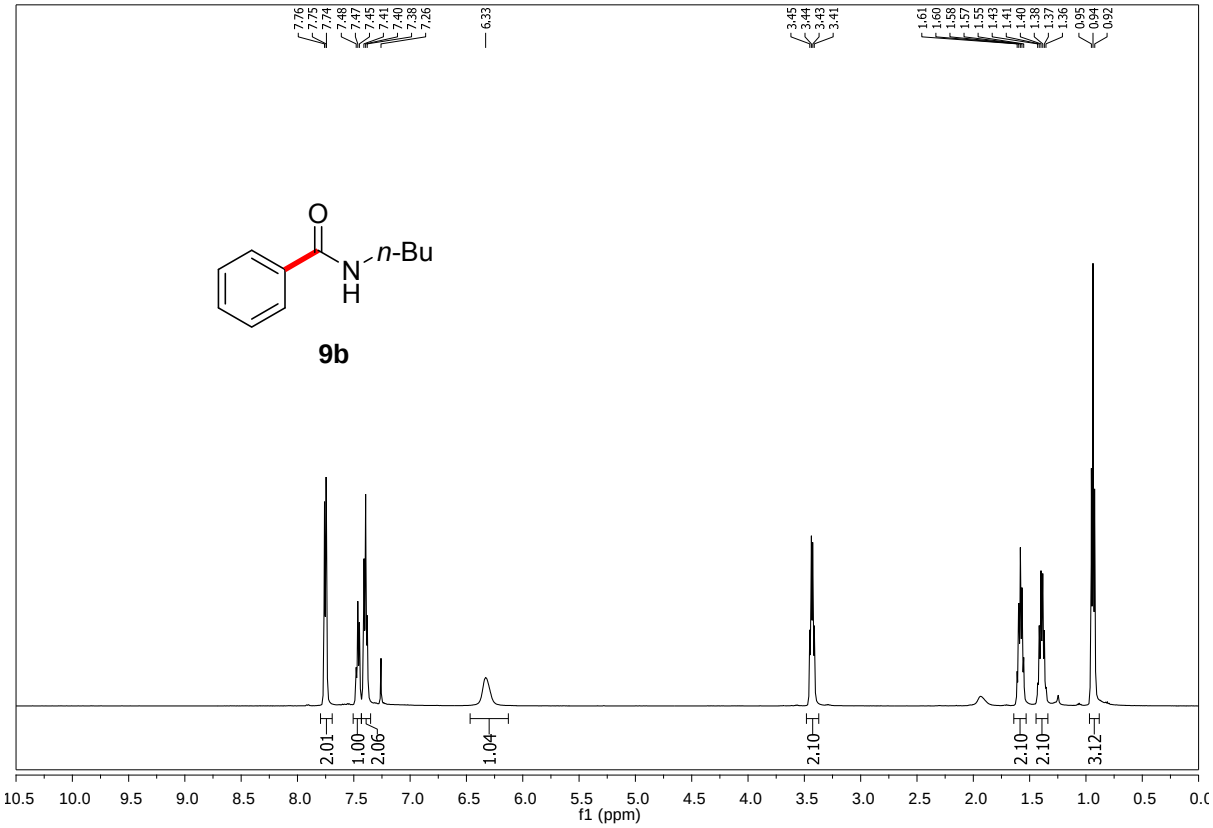


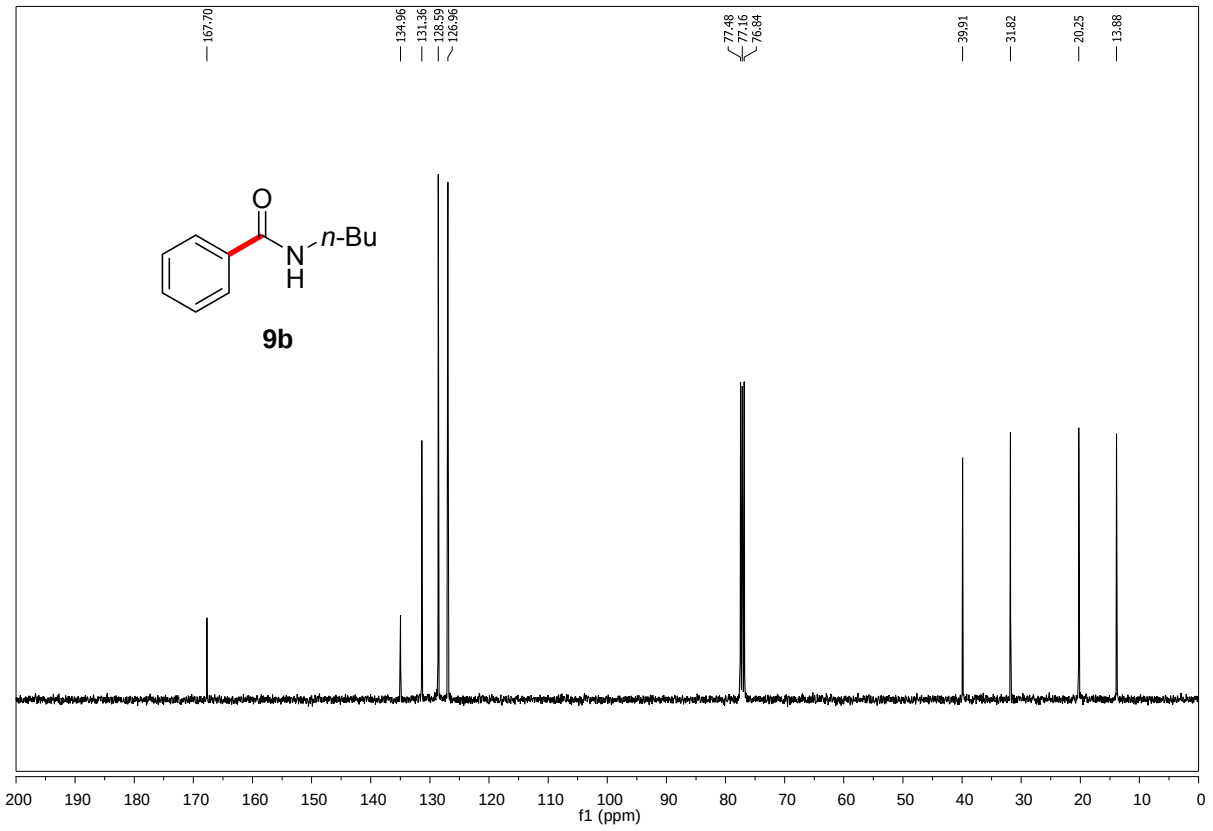
N-(*tert*-Butyl)benzamide (9a)



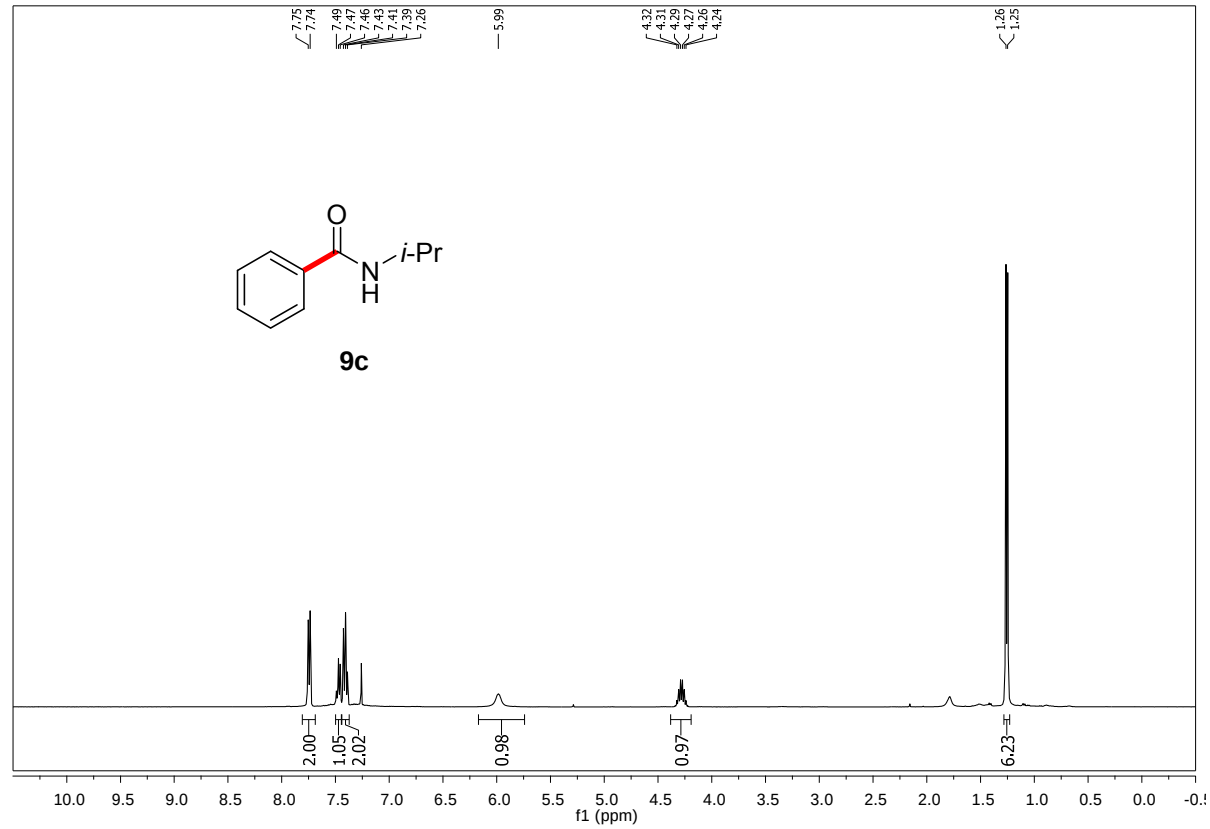


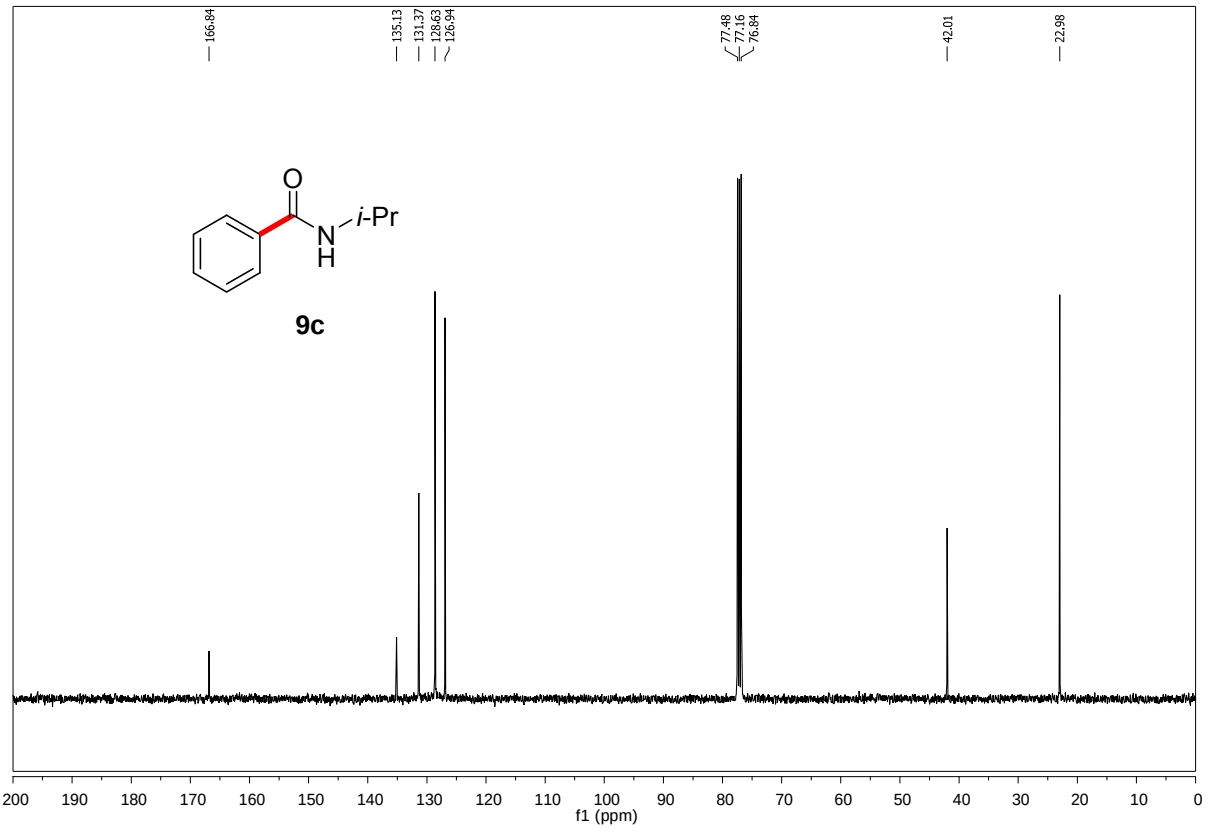
N-Butylbenzamide (9b)



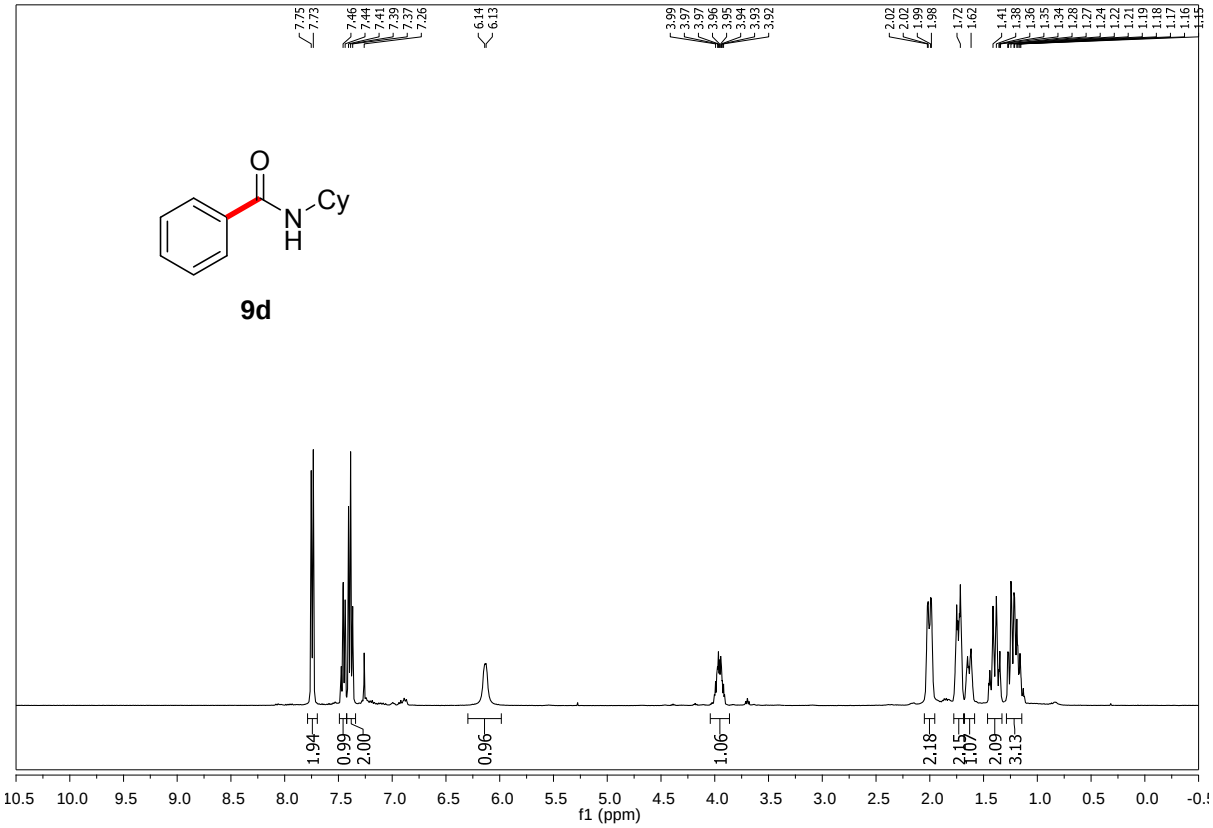


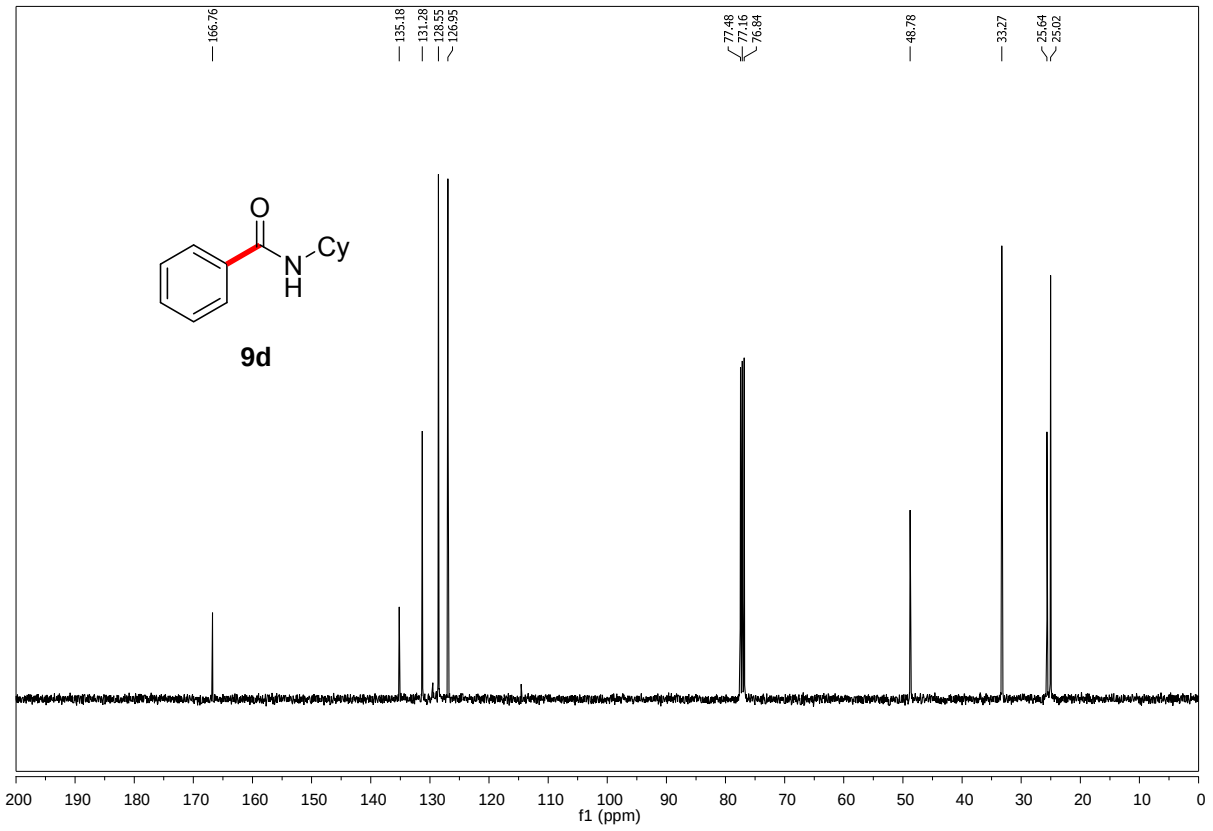
N-Isopropylbenzamide (9c)



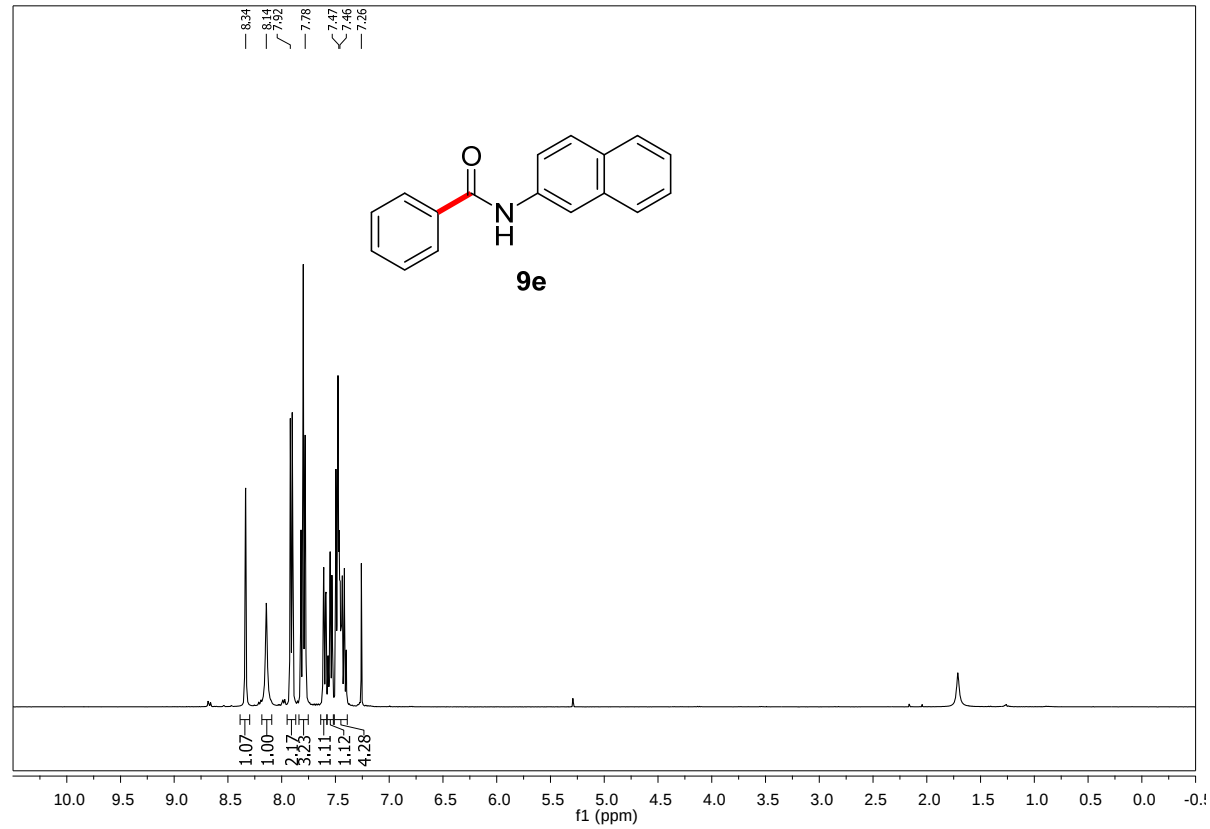


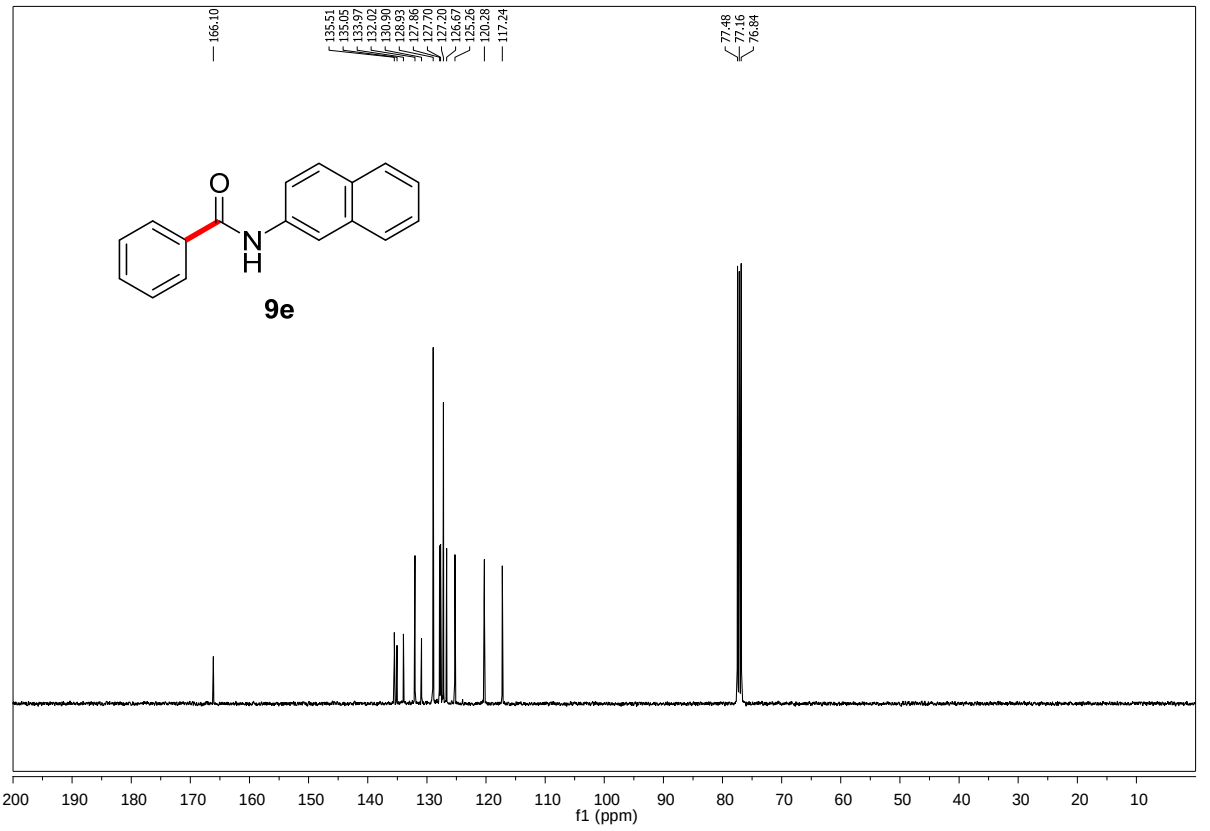
N-Cyclohexylbenzamide (9d)



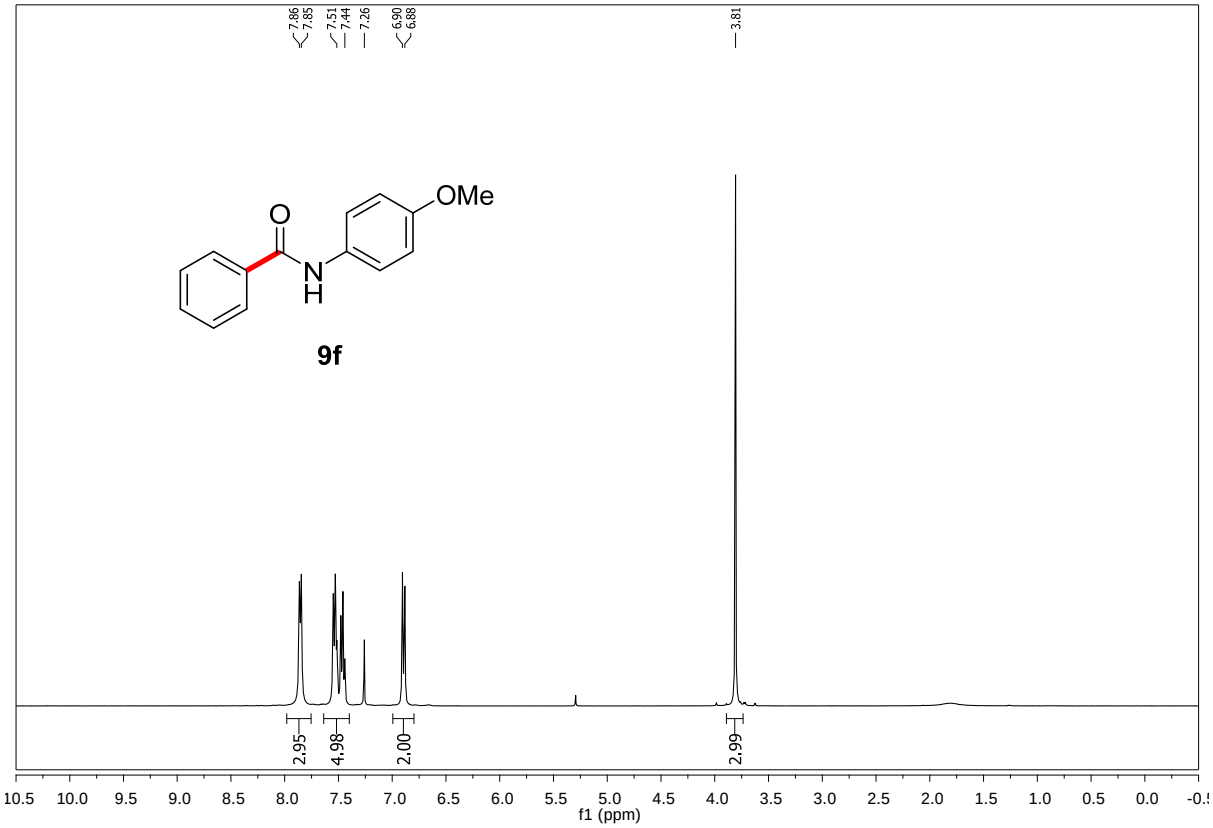


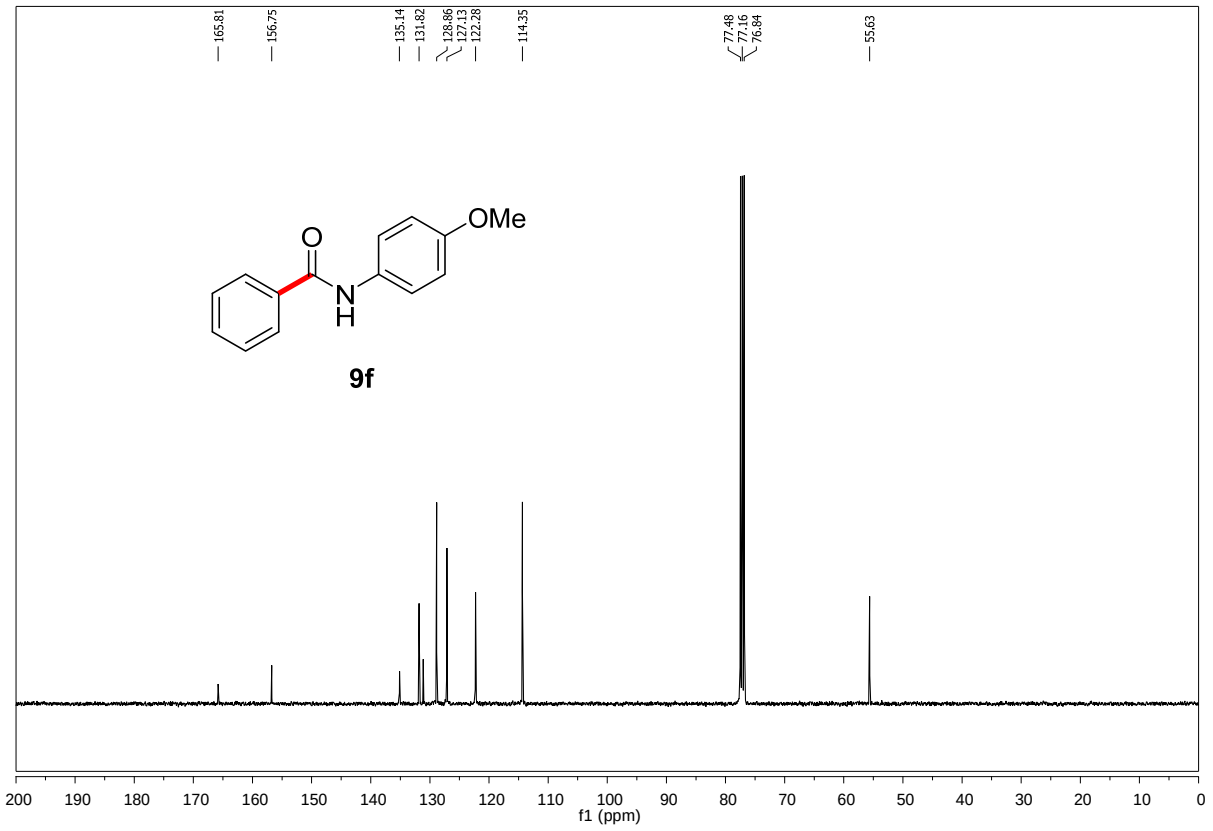
N-(Naphthalen-2-yl)benzamide (9e)



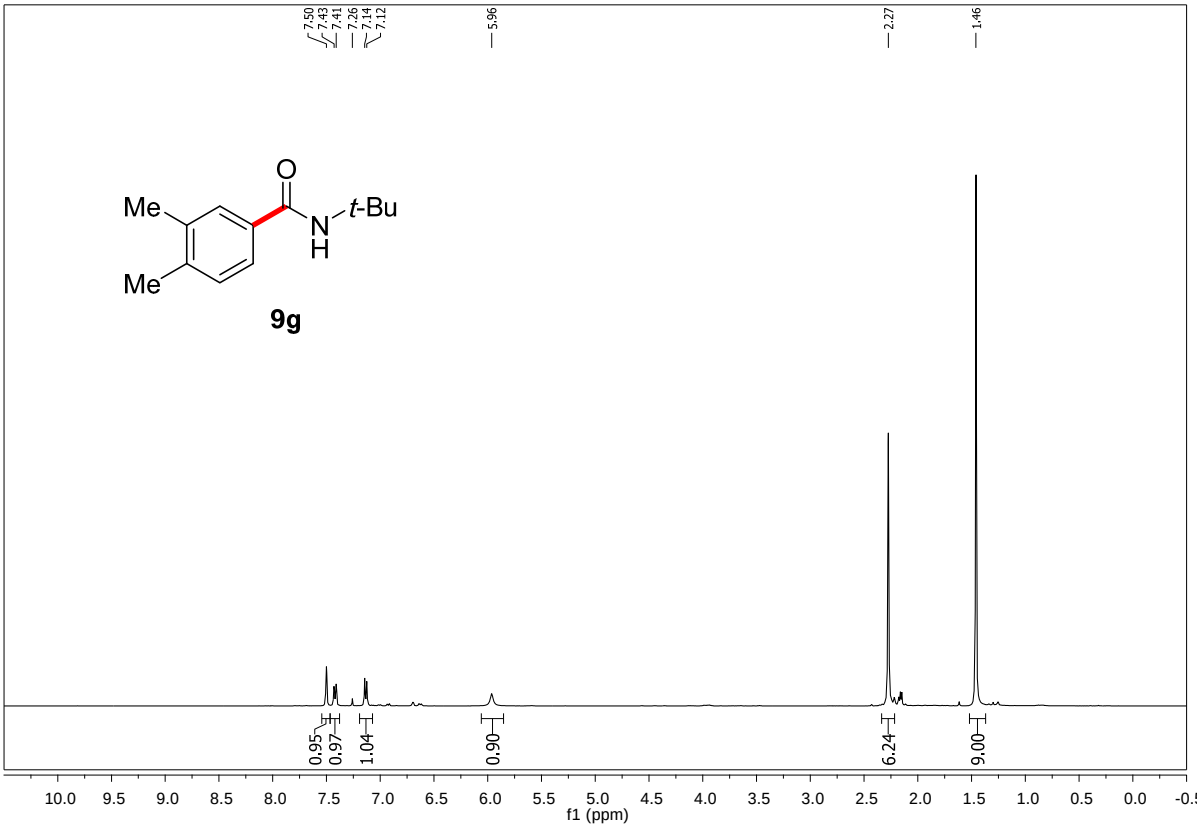


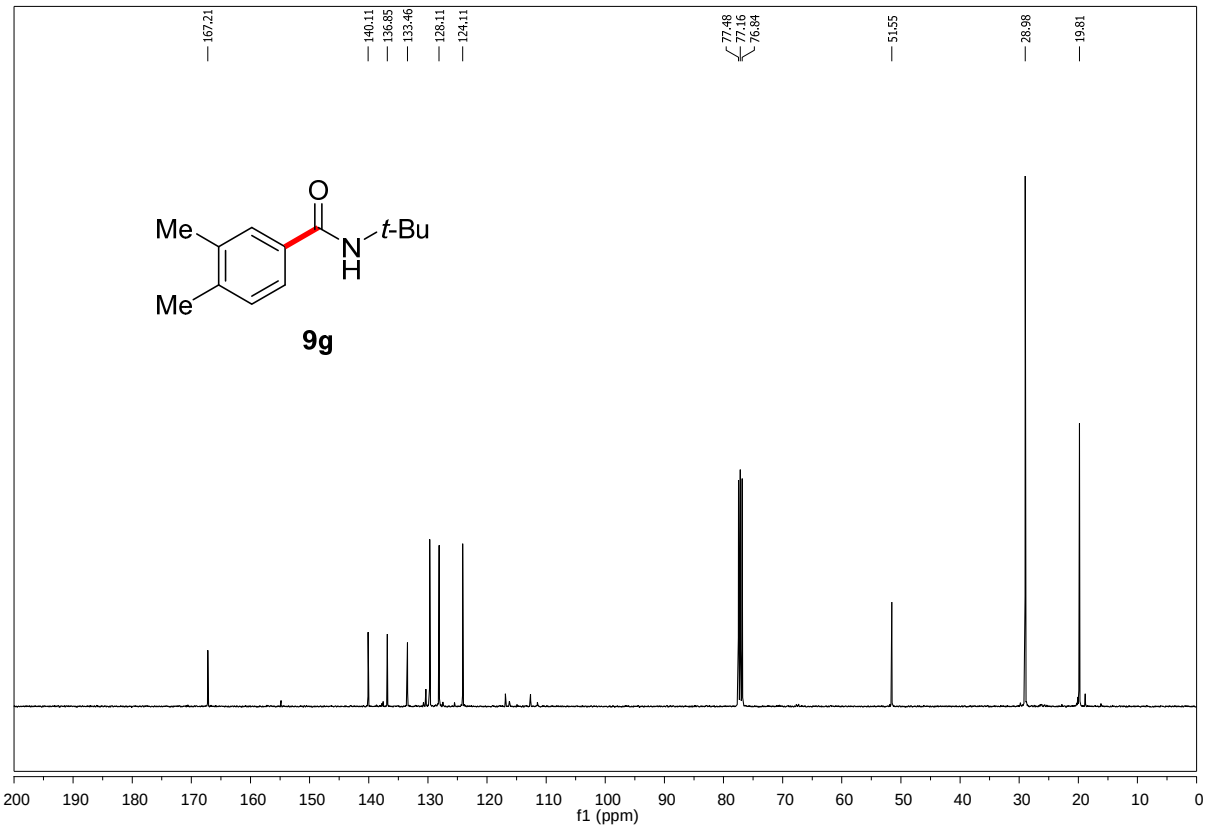
N-(4-Methoxyphenyl)benzamide (9f)



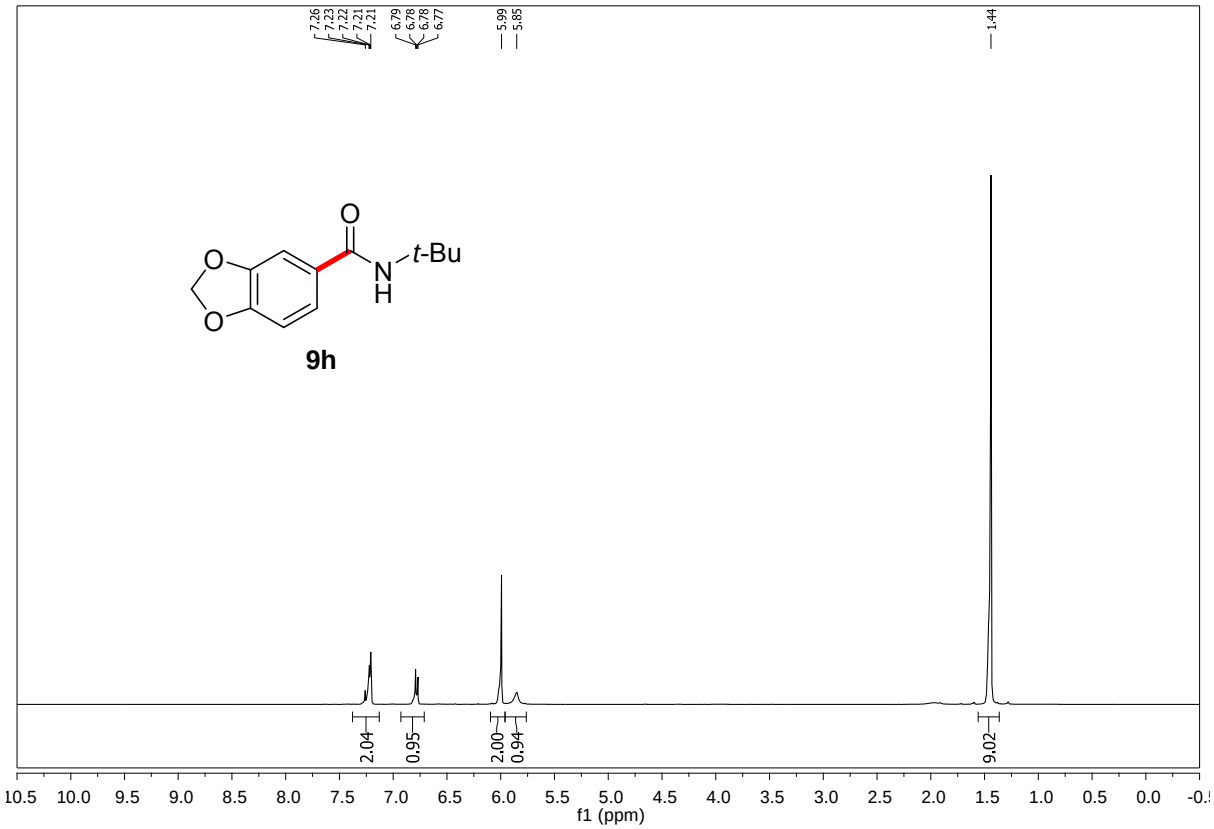


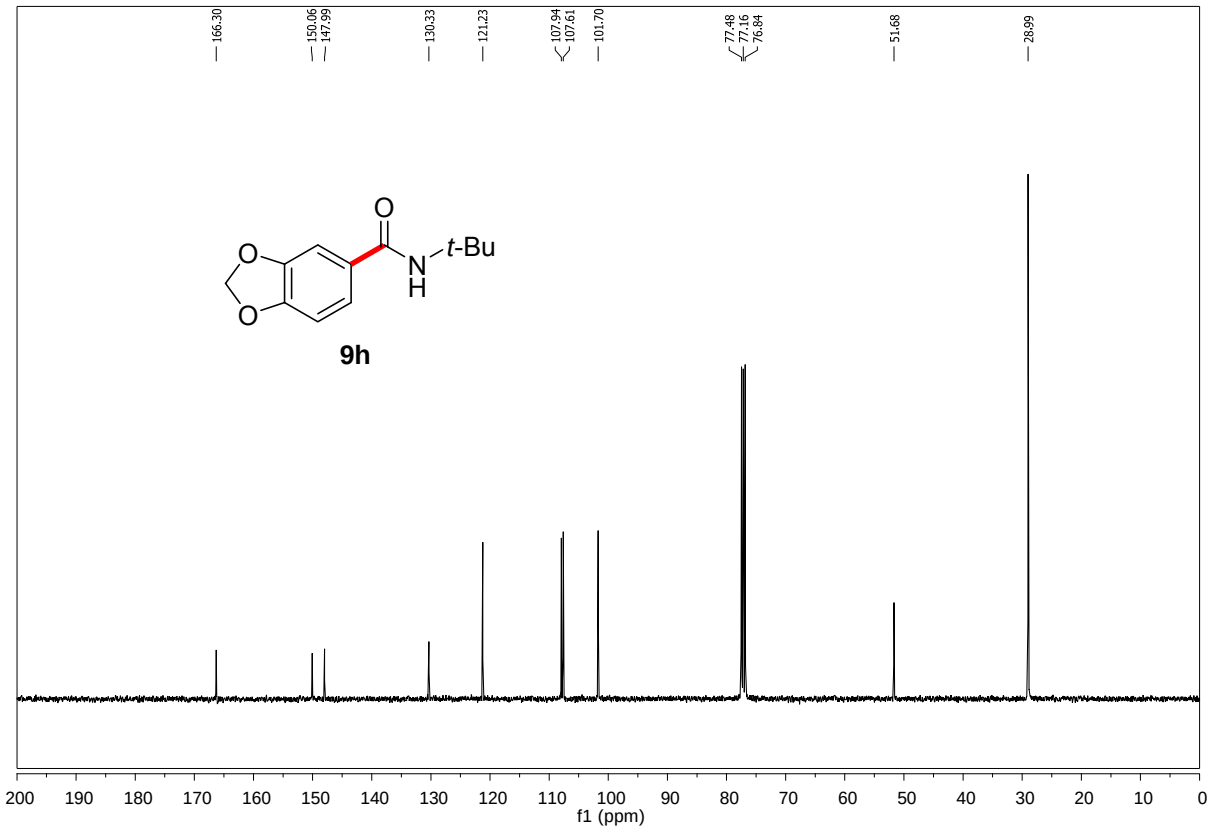
***N*-(*tert*-Butyl)-3,4-dimethylbenzamide (9g)**



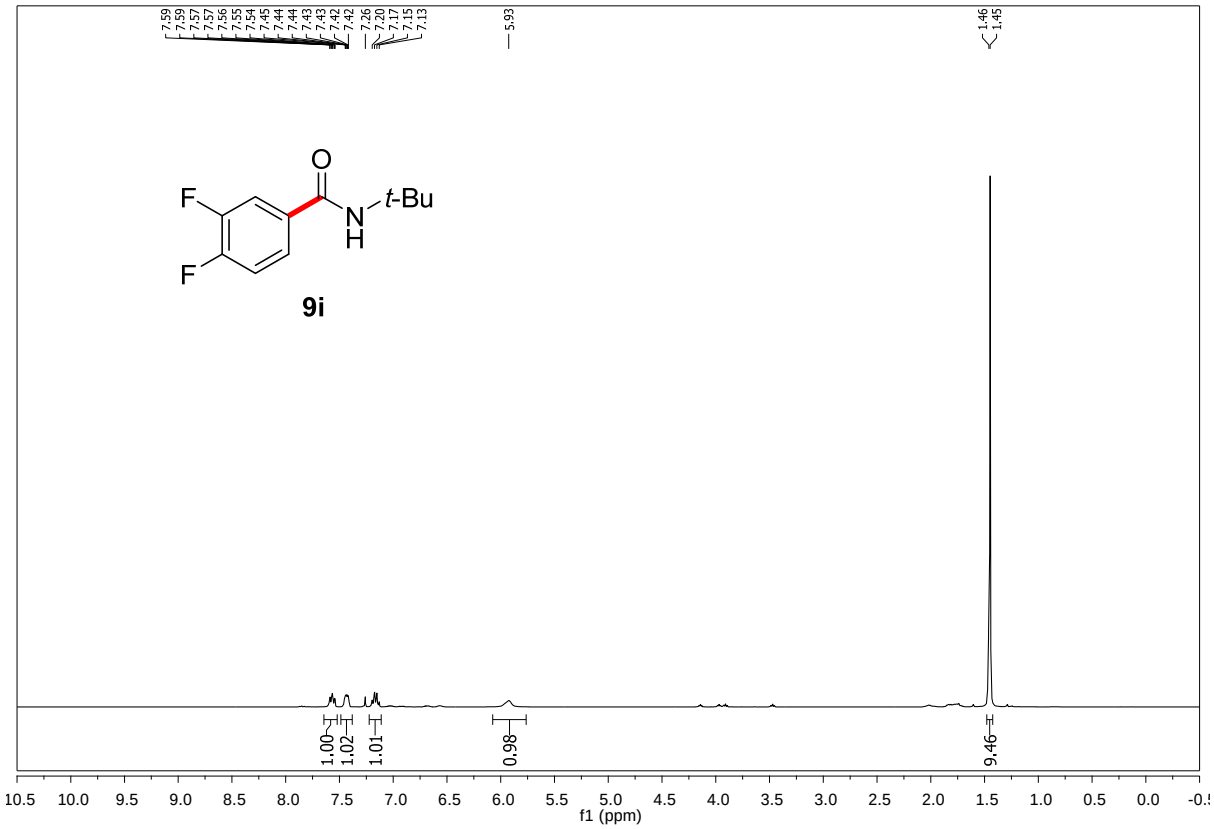


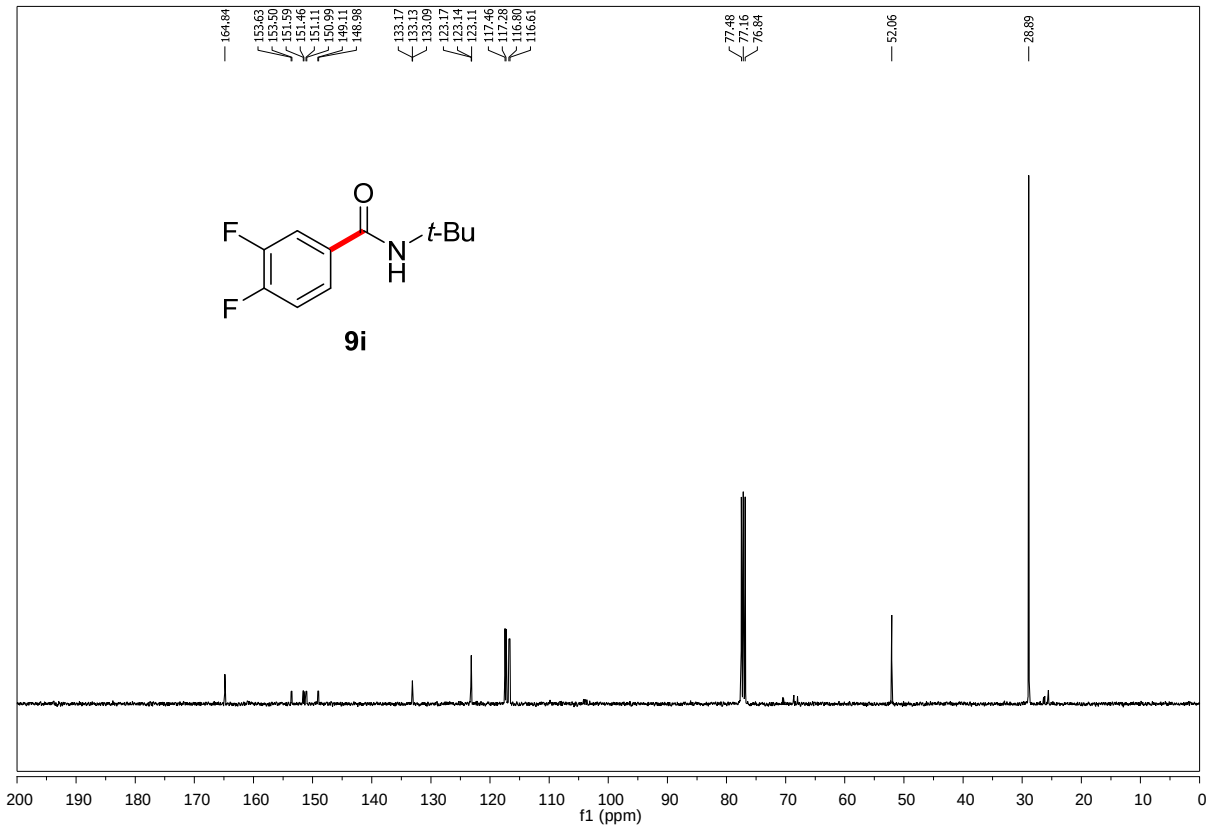
***N*-(*tert*-Butyl)benzo[*d*][1,3]dioxole-5-carboxamide (9h)**



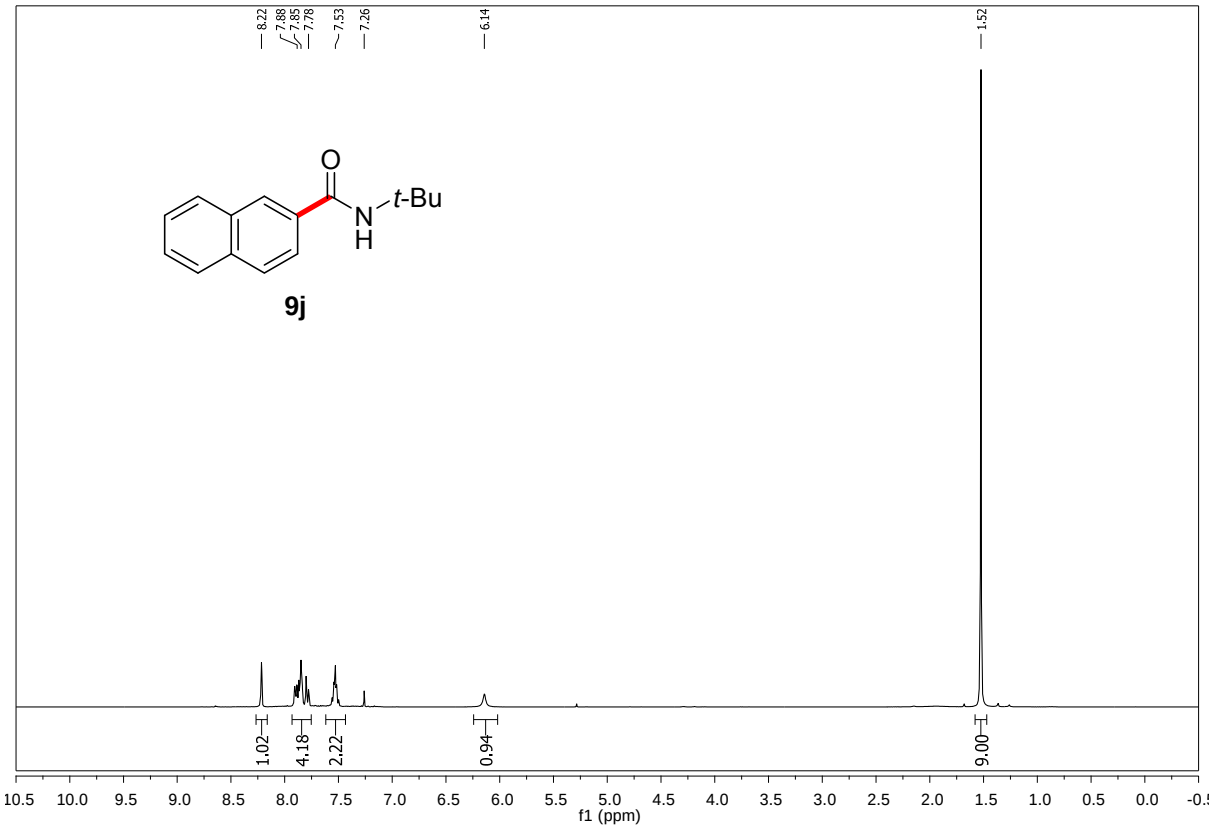


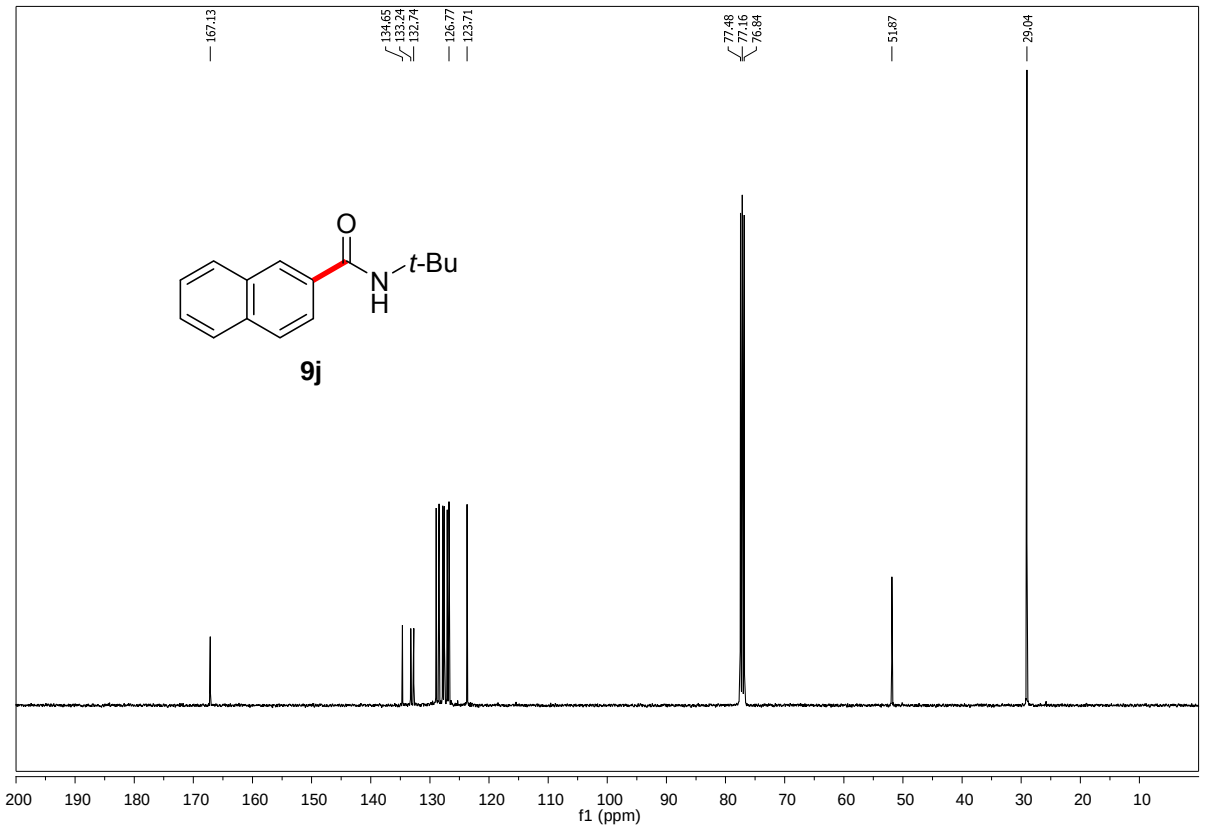
N-(*tert*-Butyl)-3,4-difluorobenzamide (9i)



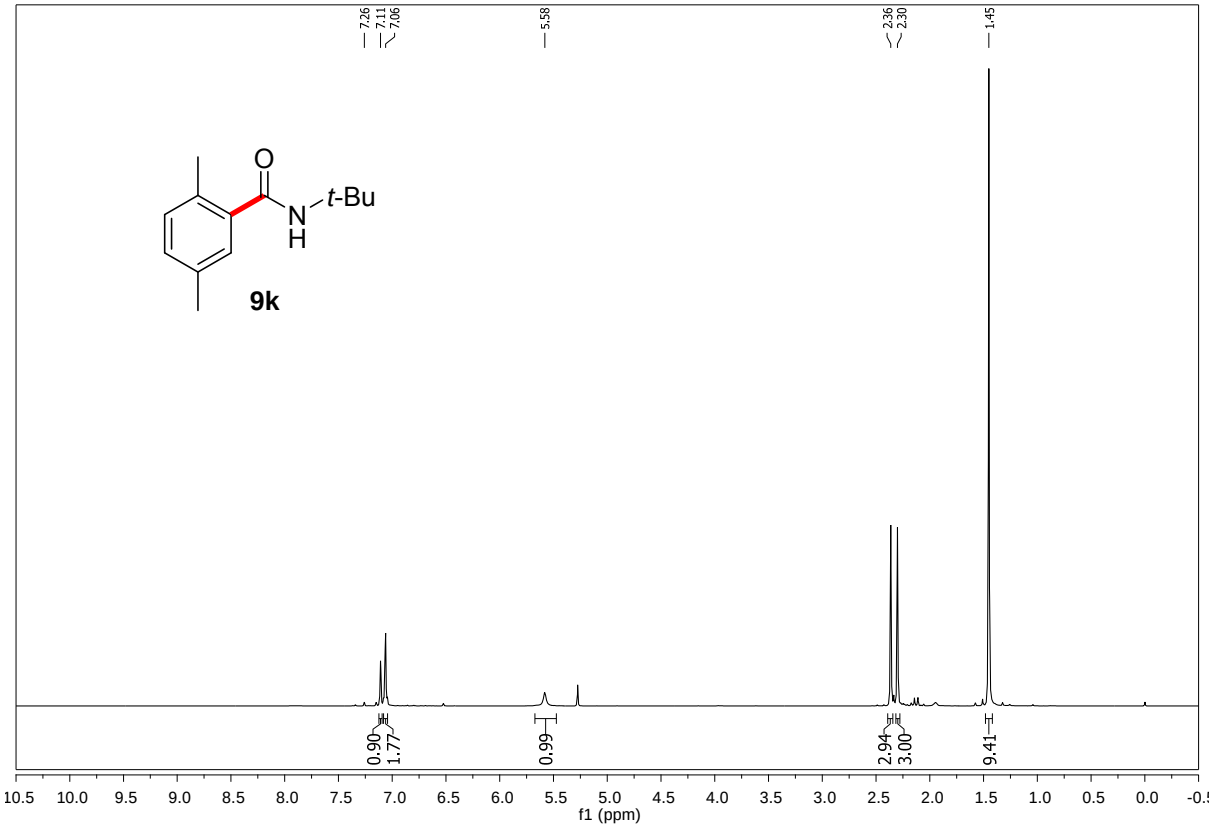


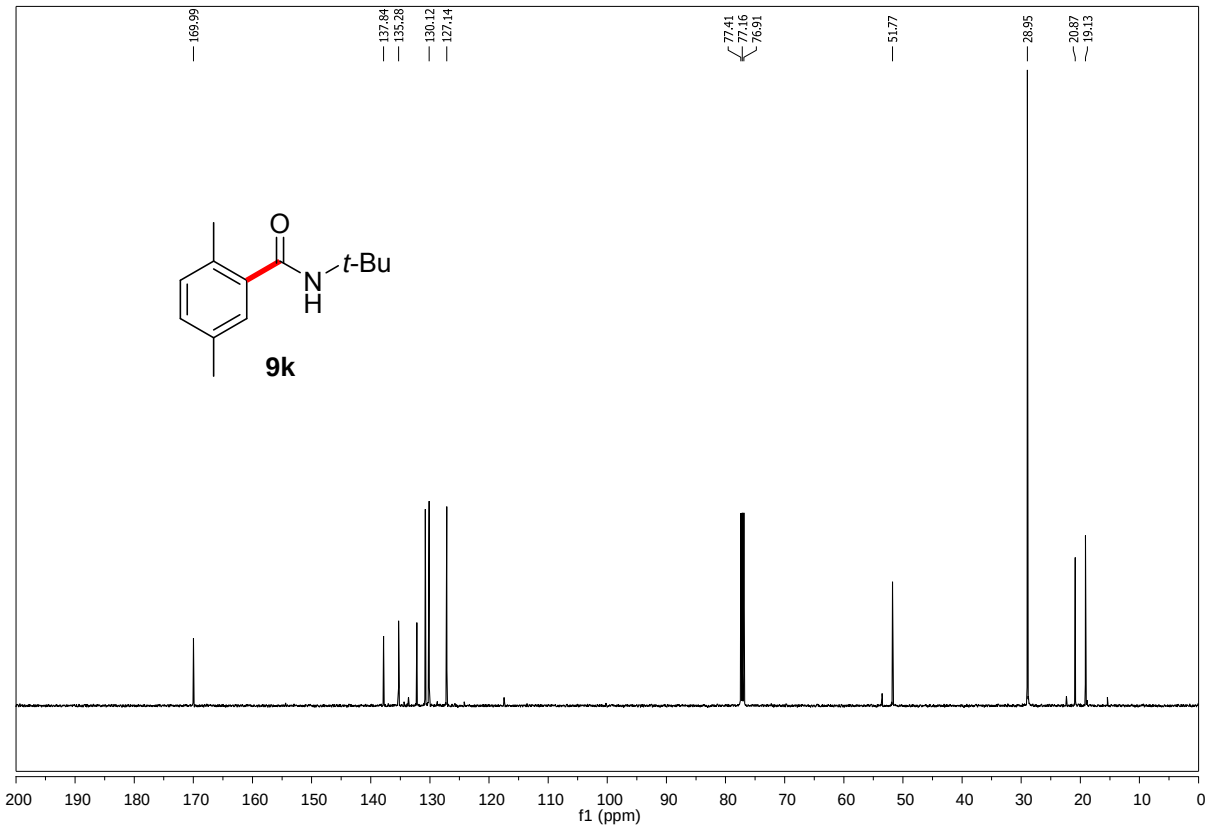
N-(*tert*-Butyl)-2-naphthamide (9j)



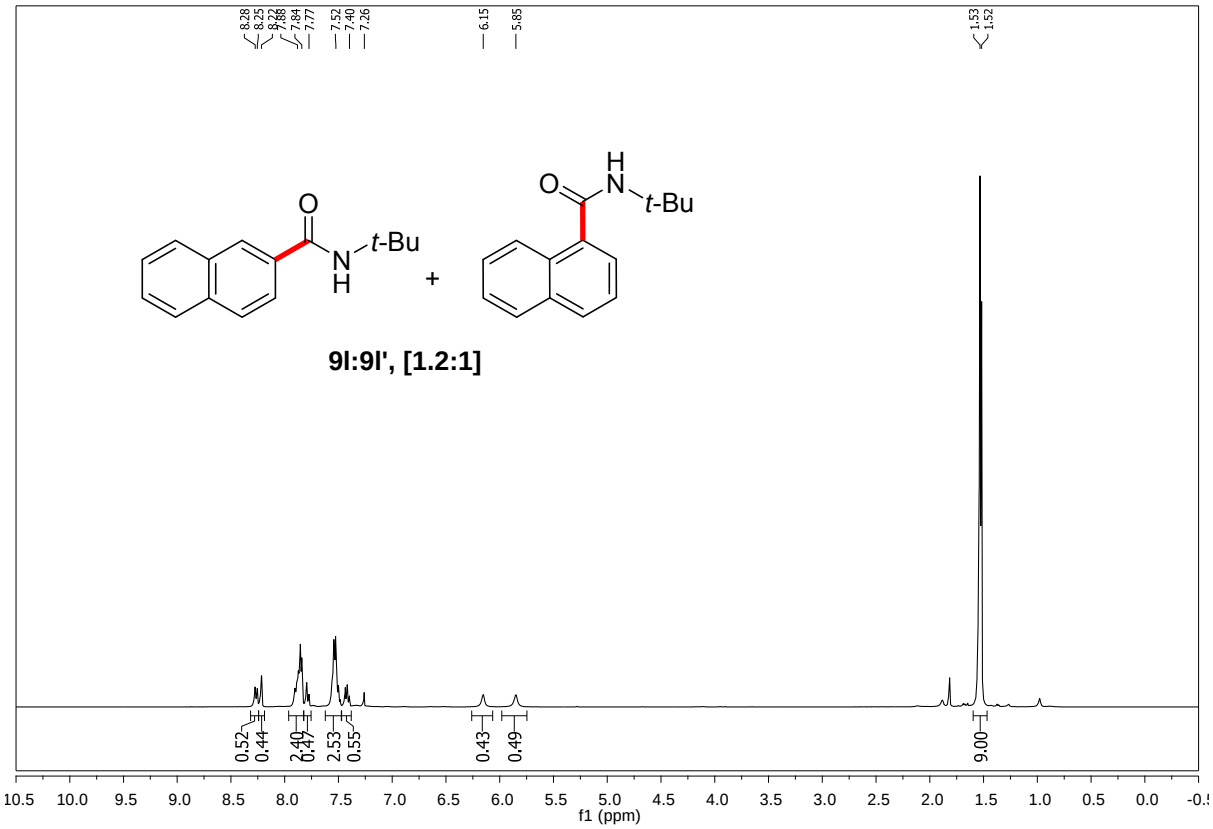


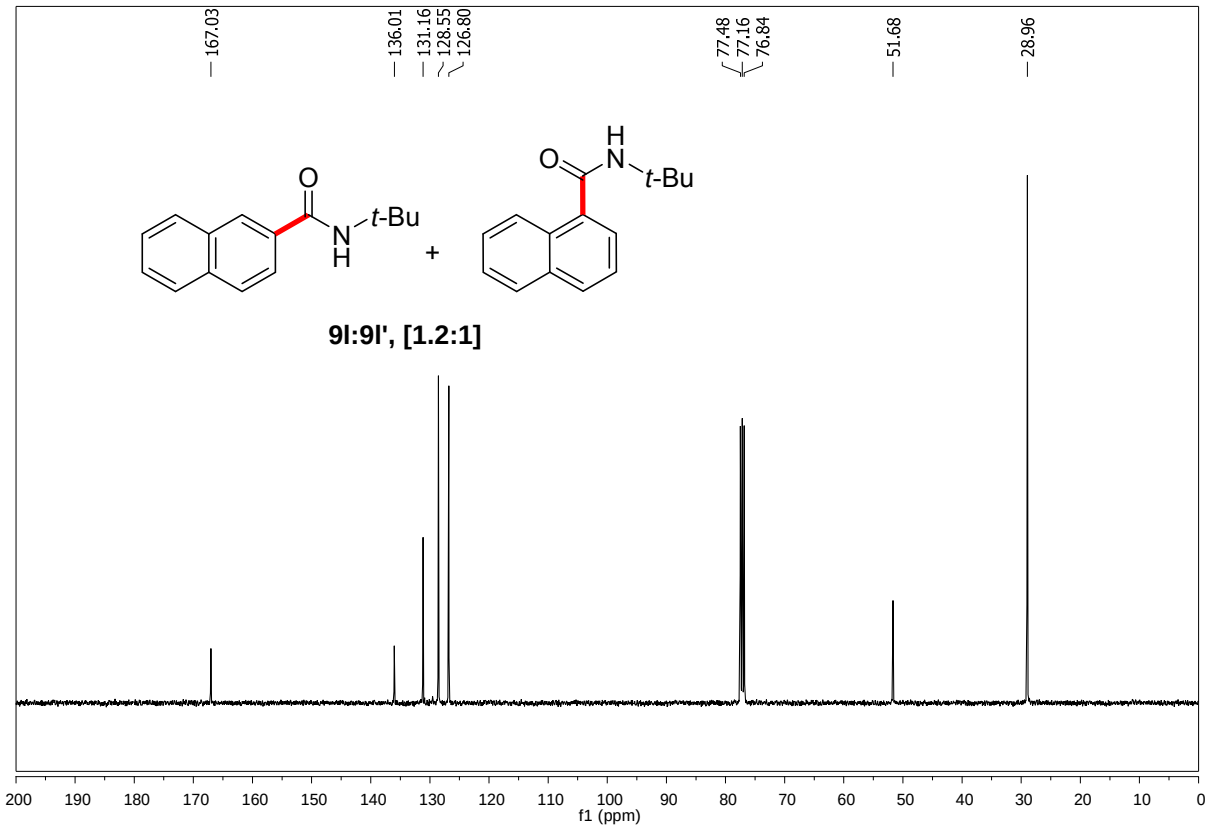
_N_-(_tert_-Butyl)-2,5-dimethylbenzamide (9k)



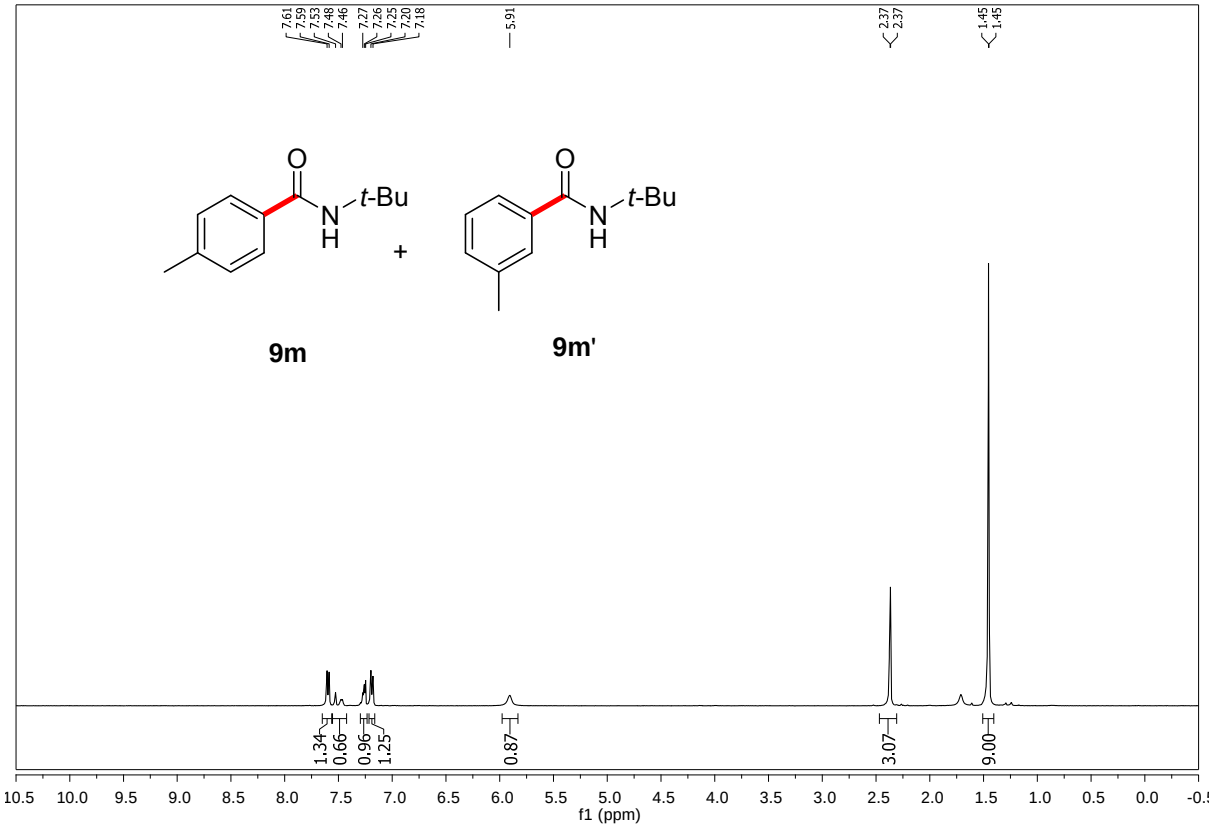


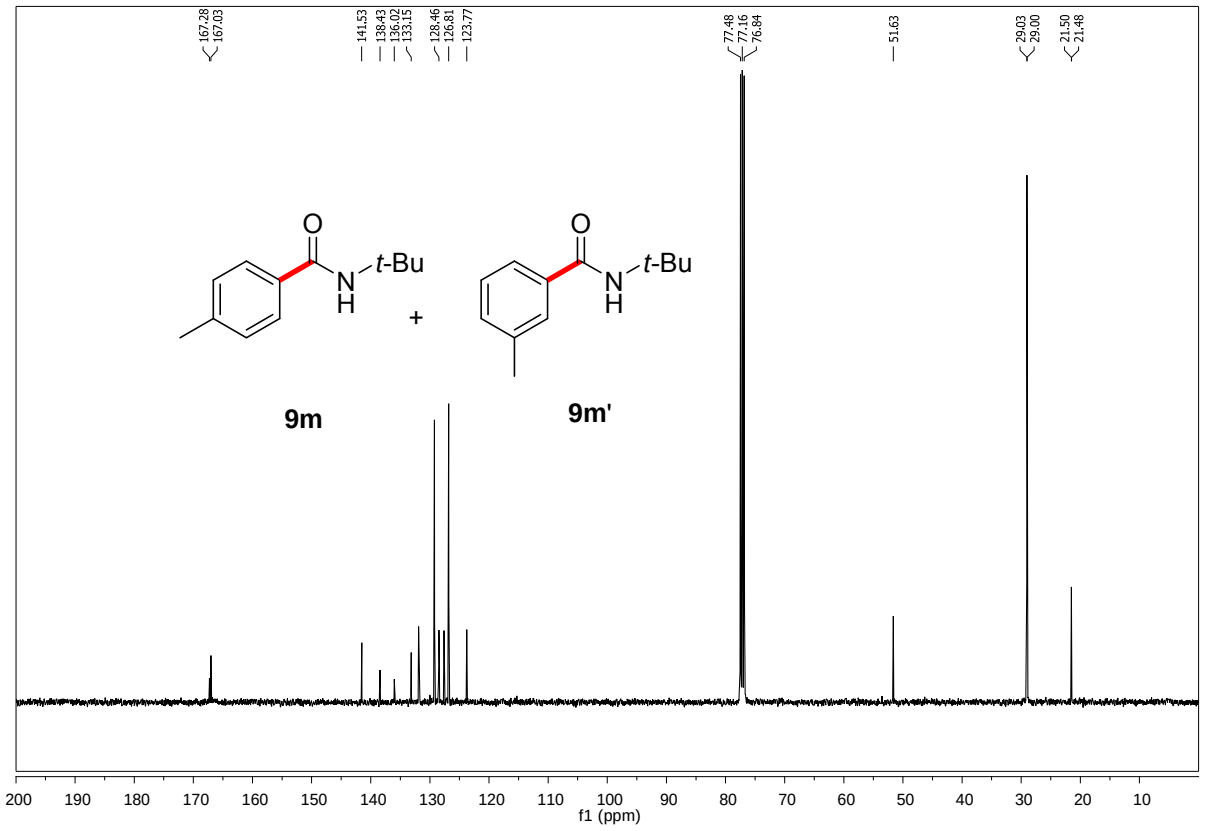
***N*-(*tert*-Butyl)benzamide 9l and 9l'**



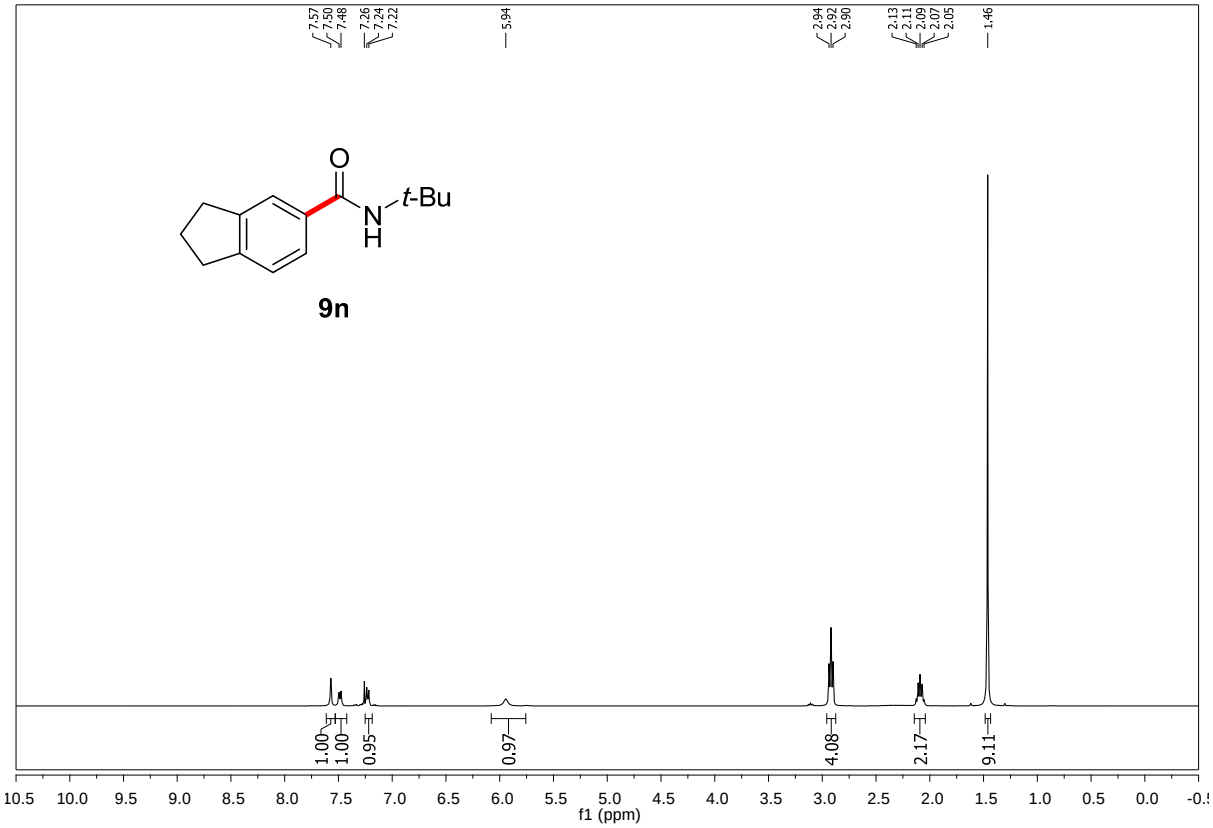


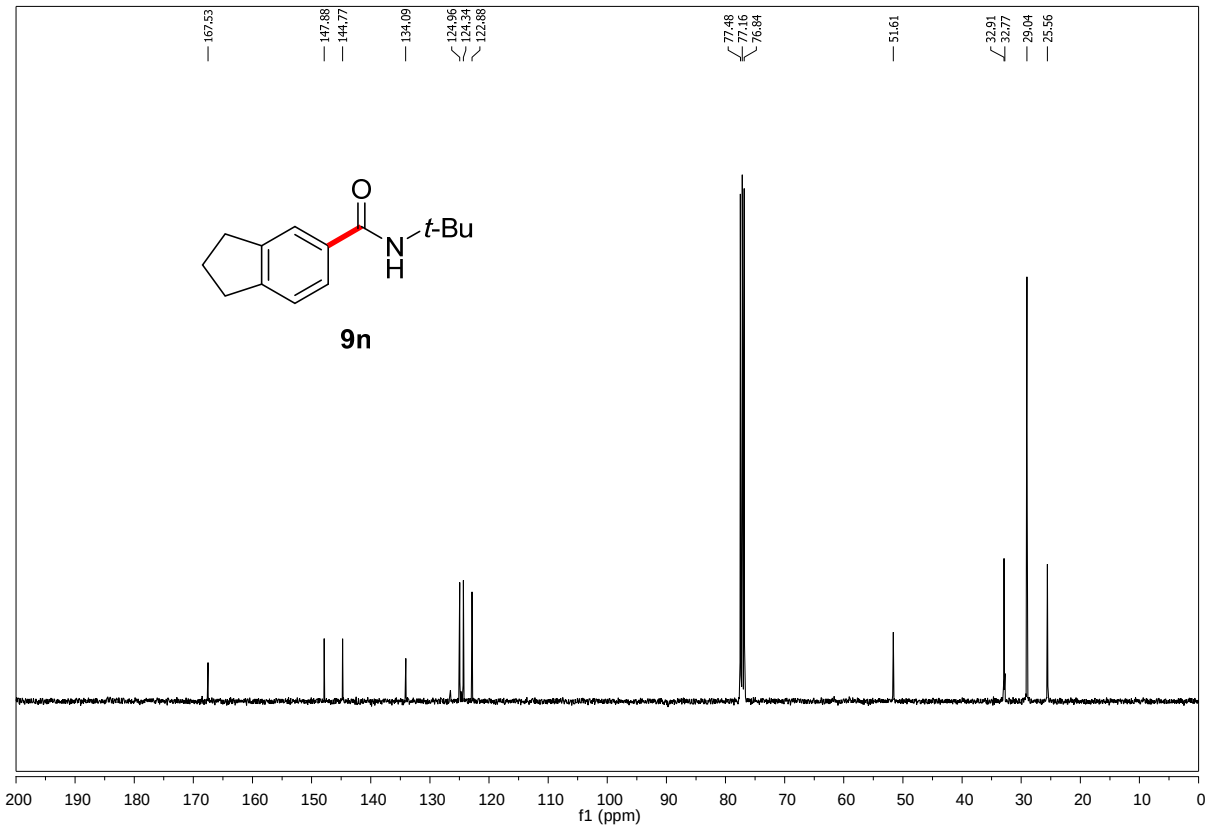
_N_-(_tert_-Butyl)-3-methylbenzamide and _N_-(_tert_-Butyl)-4-methylbenzamide (9m, 9m')



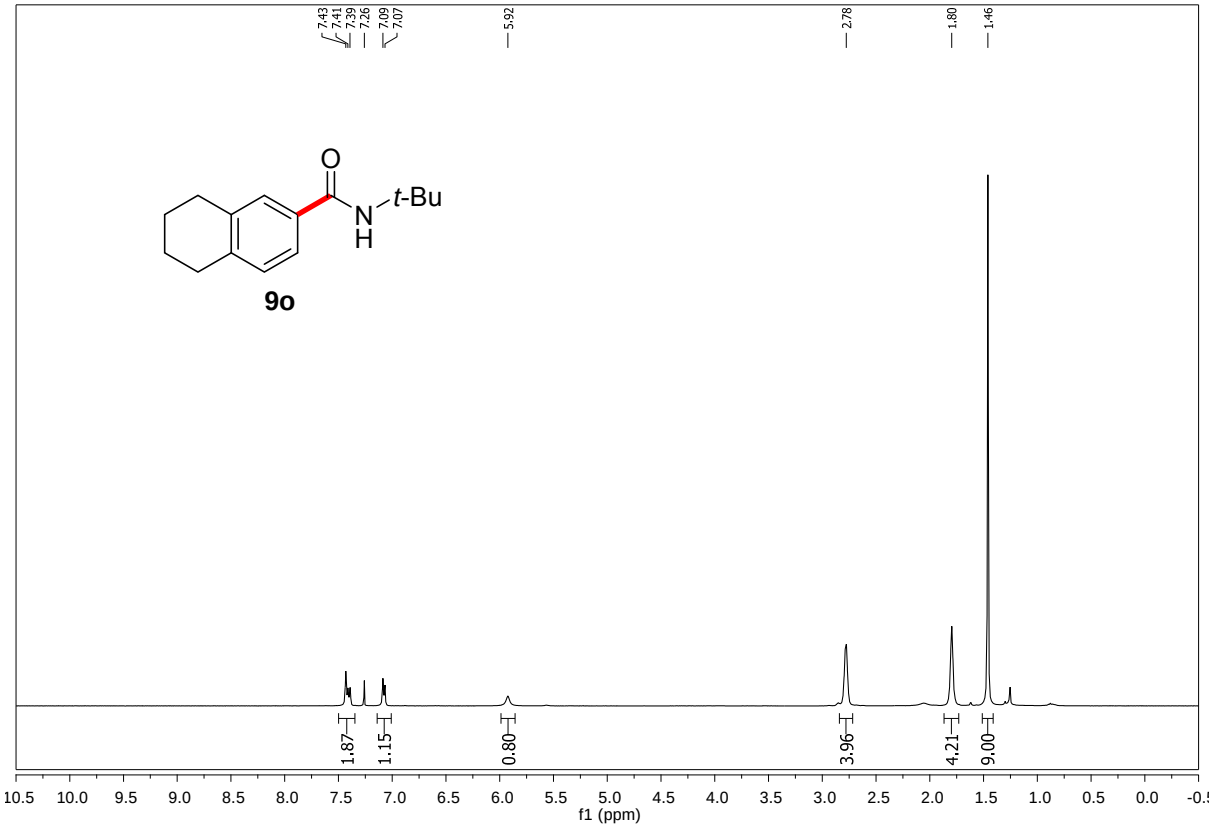


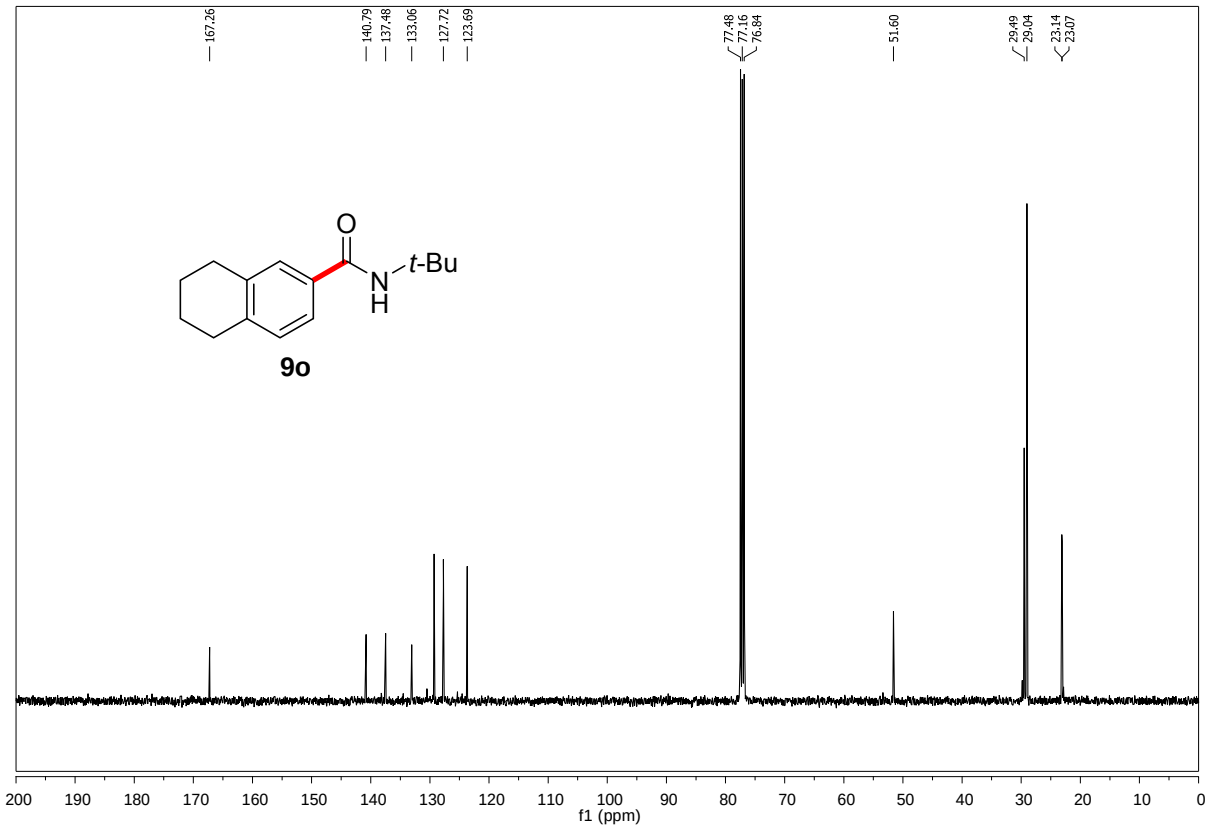
N-(*tert*-Butyl)-2,3-dihydro-1*H*-indene-5-carboxamide (9n)



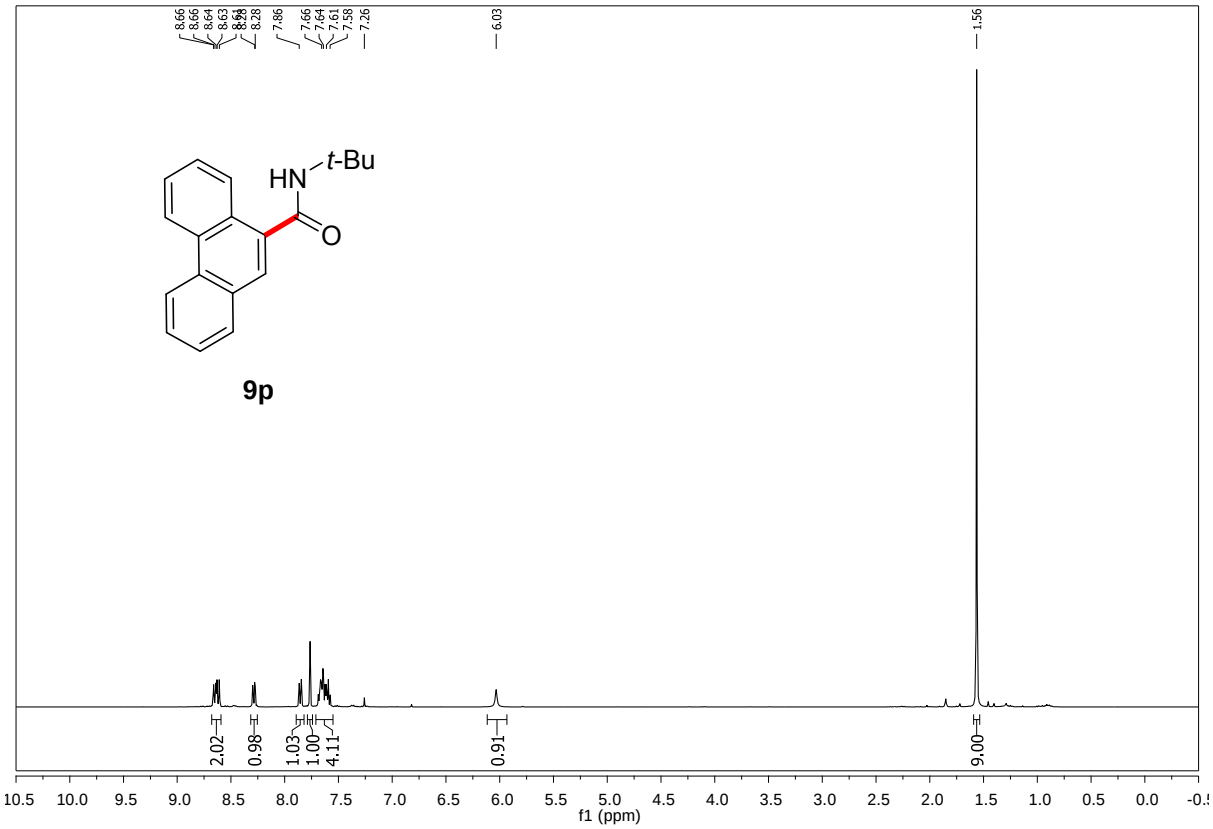


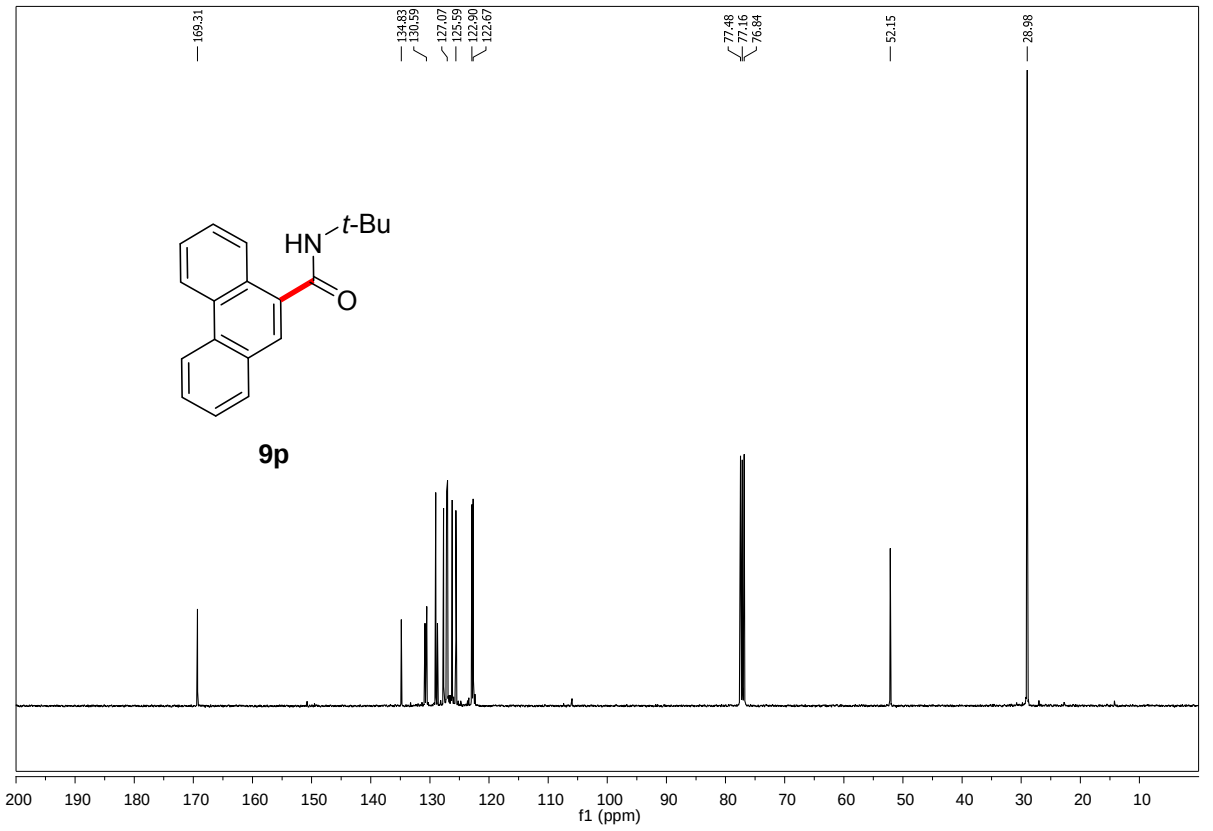
***N*-(*tert*-Butyl)-5,6,7,8-tetrahydronaphthalene-2-carboxamide (9o)**



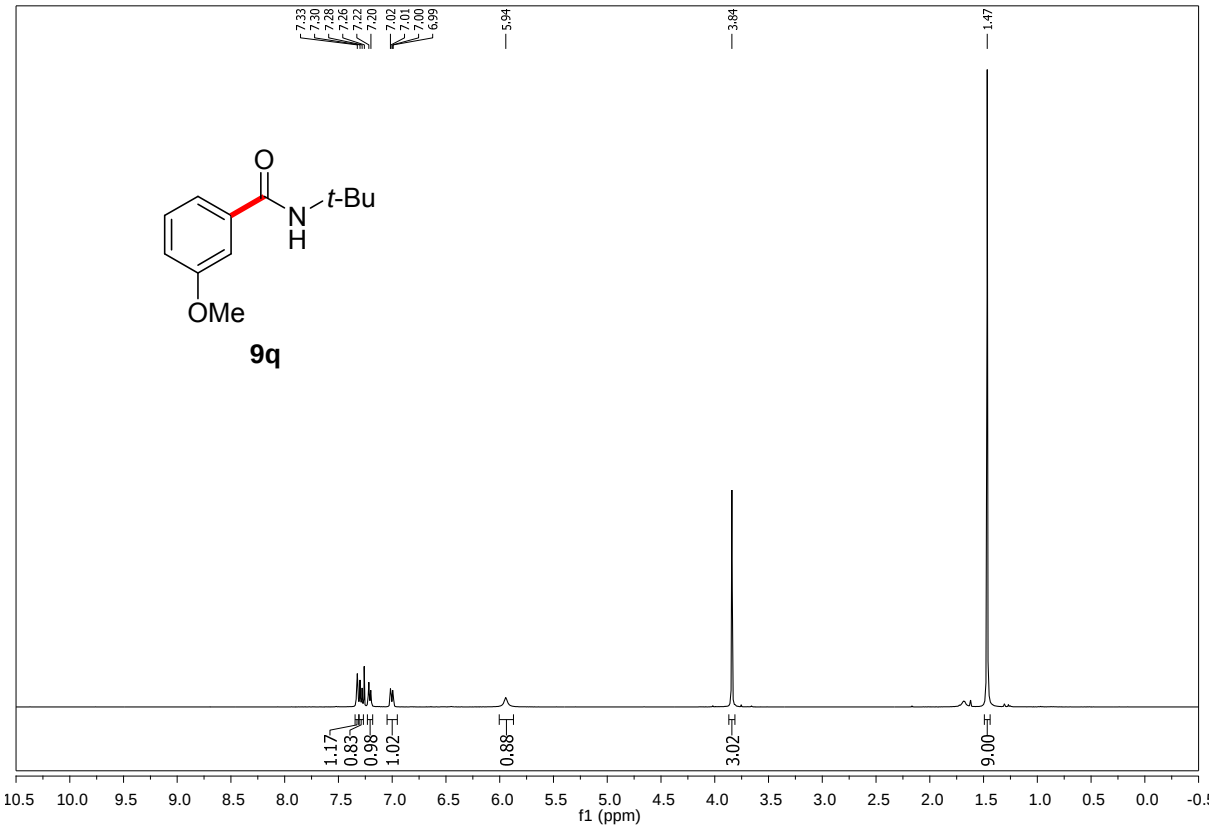


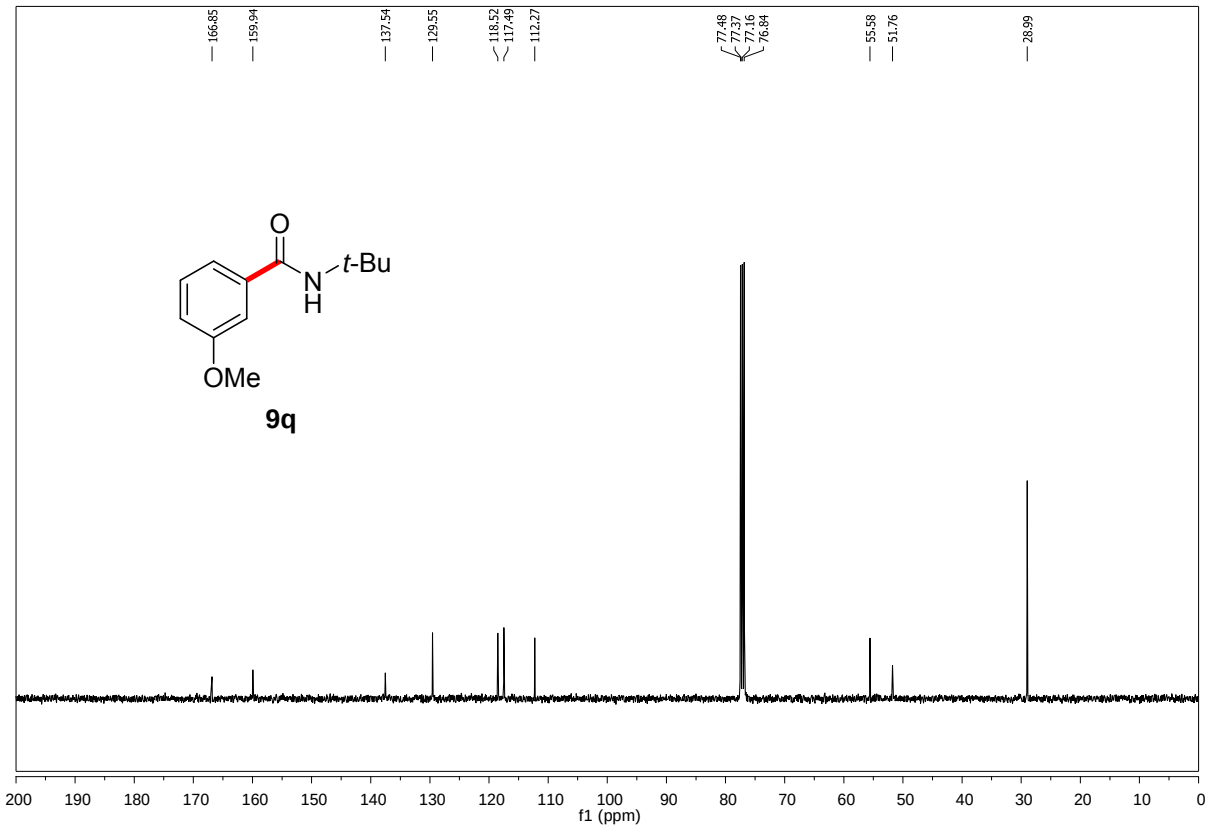
N-(*tert*-Butyl)phenanthrene-9-carboxamide (9p)



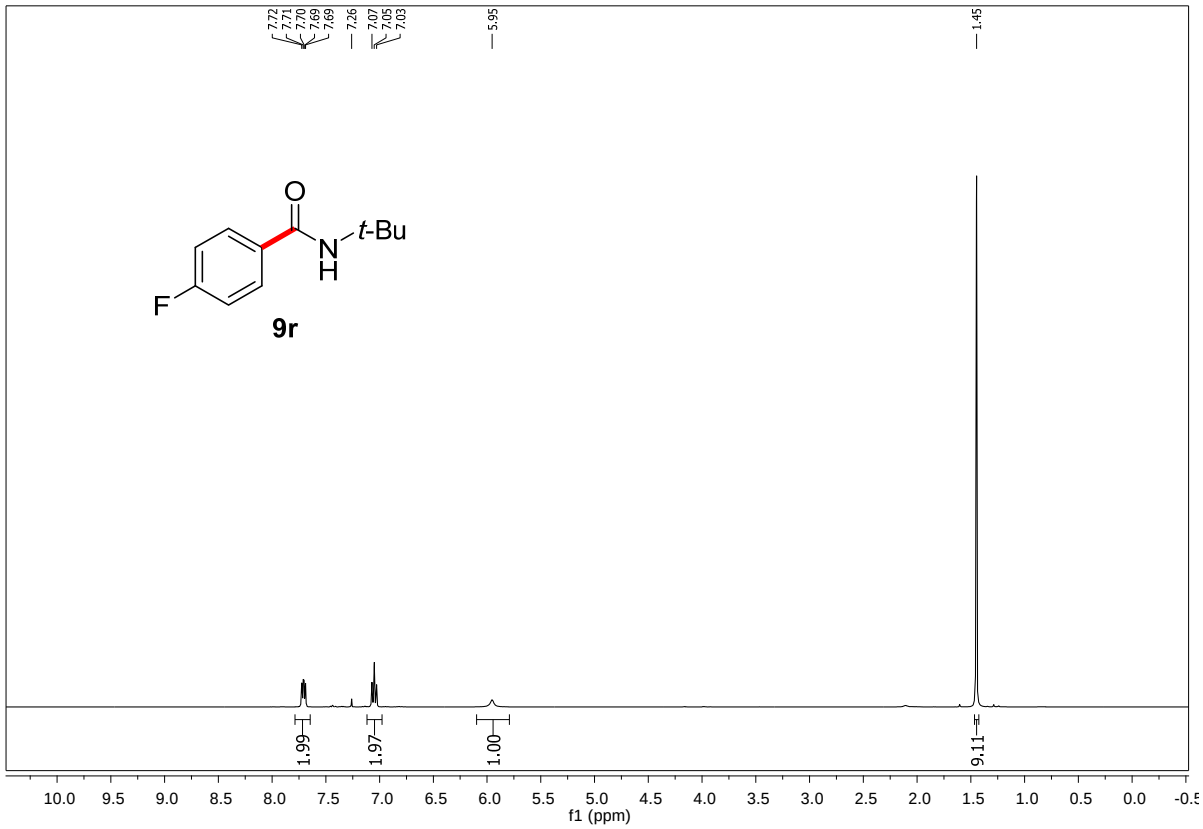


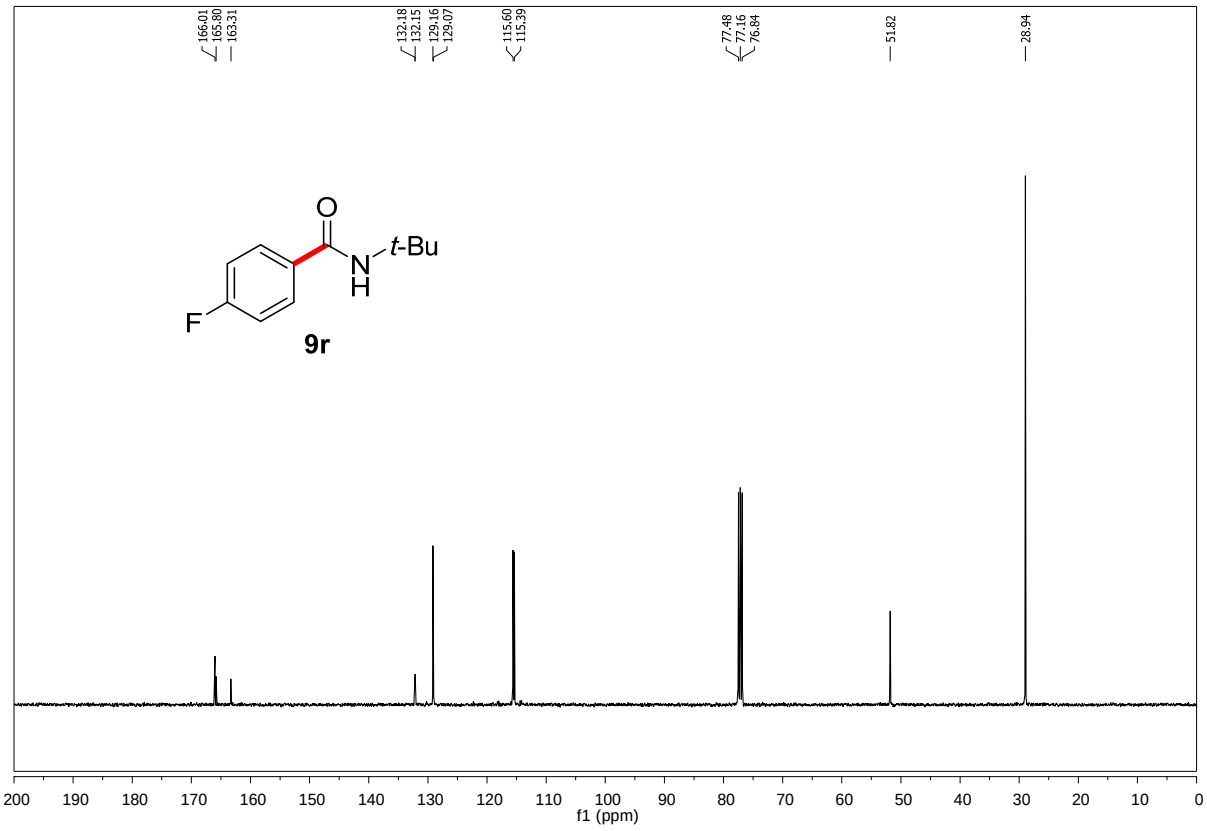
N-(*tert*-Butyl)-3-methoxybenzamide (9q)



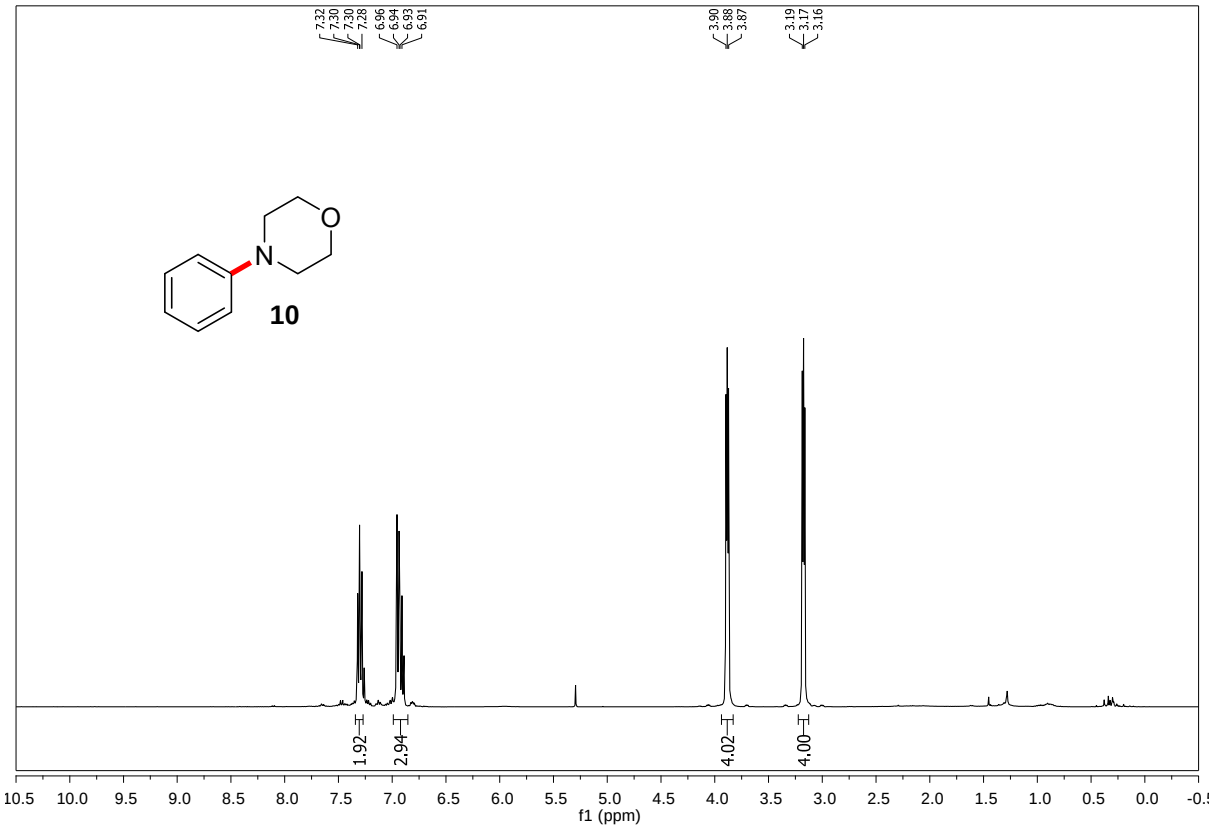


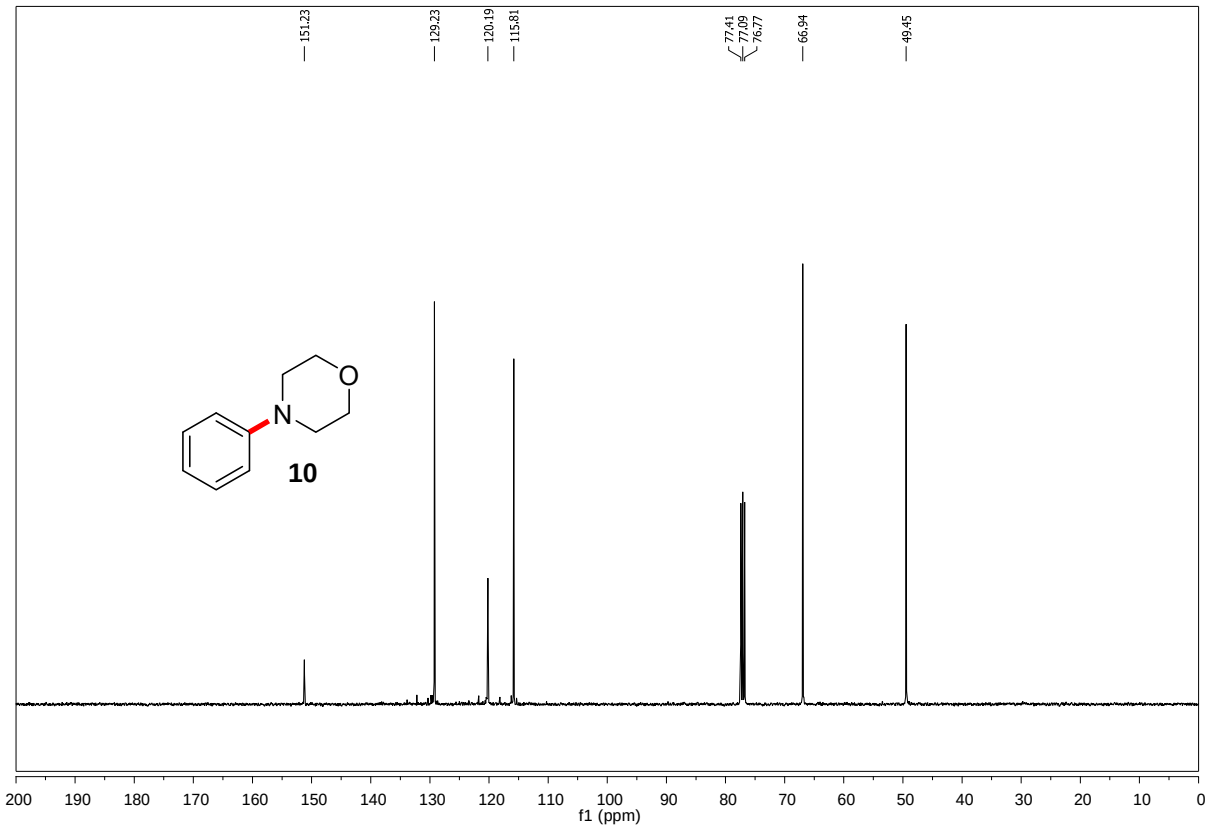
N-(*tert*-Butyl)-4-fluorobenzamide (9r)





4-Phenylmorpholine (10)





3-(*tert*-Butylimino)isobenzofuran-1(3*H*)-one (4a)