Electronic Supplementary Material (ESI) for RSC Advances.

ELECTRONIC SUPPLEMENTARY INFORMATION

A one pot protocol

to convert nitro-arenes into *N*-aryl amides

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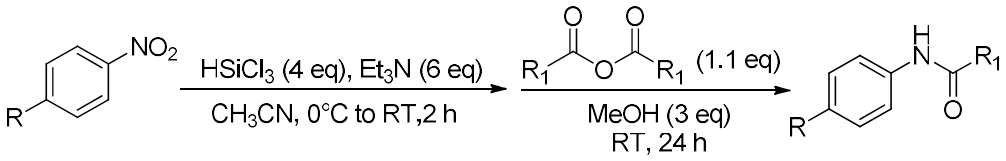
General Information

Methods: Reactions were monitored by analytical thin-layer chromatography (TLC) using silica gel 60 F254 pre-coated glass plates (0.25 mm thickness) and visualized using UV light. Flash chromatography was carried out on silica gel (230-400 mesh). ^{1}H NMR spectra were recorded on spectrometers operating at 300 MHz (Bruker Fourier 300); proton chemical shifts are reported in ppm (δ) with the solvent reference relative to tetramethylsilane (TMS) employed as the internal standard ($CDCl_3$ δ = 7.26 ppm). ^{13}C NMR spectra were recorded on 300 MHz spectrometers (Bruker Fourier 300) operating at 75 MHz, with complete proton decoupling; carbon chemical shifts are reported in ppm (δ) relative to TMS with the respective solvent resonance as the internal standard ($CDCl_3$, δ = 77.0 ppm). 19F NMR spectra were recorded on 300 MHz spectrometers (Bruker Fourier 300) operating at 282.1 MHz; fluorine chemical shifts are reported in ppm (δ) relative to CF_3Cl with the respective solvent resonance as the internal standard ($CDCl_3$, δ = 77.0 ppm). Mass spectra and accurate mass analysis were carried out on a VG AUTOSPEC- M246 spectrometer (double-focusing magnetic sector instrument with EBE geometry) equipped with EI source or with LCQ Fleet ion trap mass spectrometer, ESI source, with acquisition in positive ionization mode in the mass range of 50-2000 m/z.

Materials: Dry solvents were purchased and stored under nitrogen over molecular sieves (bottles with crown caps). All chemicals were purchased from commercial suppliers and used without further purification unless otherwise specified.

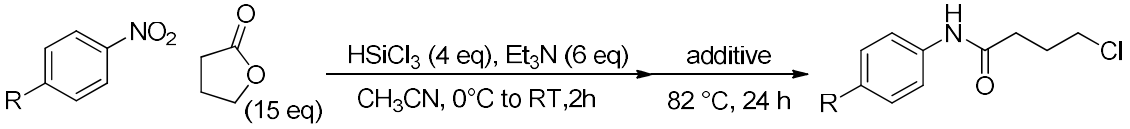
General procedures for the one pot reduction-amidation reactions

General procedure for the one-pot amidation with anhydrides



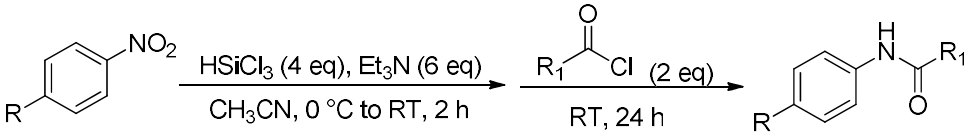
Working under N_2 atmosphere, a solution of $HSiCl_3$ (2.54 mmol, 4 eq) in dry MeCN (1 mL) was added to a solution of the proper nitroarene (0.64 mmol, 1 eq) and Et_3N (3.8 mmol, 6 eq) in dry MeCN (4 mL) at 0 °C. After 30 minutes, the reaction mixture was allowed to RT and kept under stirring for 2 hours. Subsequently, MeOH (1.92 mmol, 3eq) and the anhydride (0.7 mmol, 1.1 eq) were added. The mixture was kept under stirring at RT for 24 hours. After this period, the reaction was quenched with 10% NaOH (5 mL), stirred for 10 minutes and then extracted with CH_2Cl_2. The collected organic phases were washed with 1M HCl and dried over Na_2SO_4; the solvent was removed under reduced pressure. Typically, no flash column chromatography purification was needed.

General procedure for the one-pot amidation with lactones



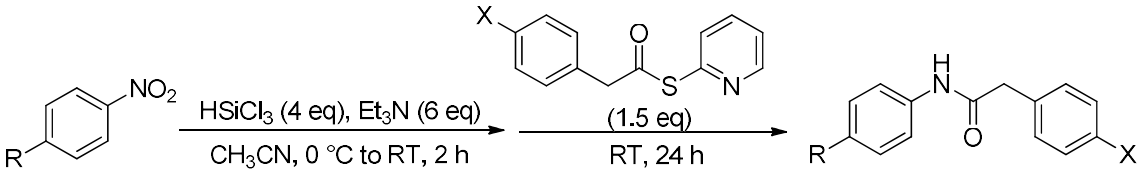
Working under N_2 atmosphere, a solution of $HSiCl_3$ (2.54 mmol, 4 eq) in dry MeCN (1 mL) was added to a solution of the proper nitroarene (0.64 mmol, 1 eq), γ-butyrolactone (9.6 mmol, 15 eq) and Et_3N (3.8 mmol, 6 eq) in dry MeCN (4 mL) at 0 °C. After 30 minutes, the reaction mixture was allowed to RT and kept under stirring for 2 hours. Subsequently, the additive (when needed) was added. The mixture was heated to reflux (82 °C) and kept under stirring for 24 hours. After this period, the reaction was quenched with 10% NaOH (5 mL), stirred for 10 minutes, filtered over celite, washed with EtOAc, dried over Na_2SO_4 and filtered; the solvent was removed under reduced pressure. The product was purified via flash column chromatography.

General procedure for the one-pot amidation with acetyl chlorides



Working under N_2 atmosphere, a solution of $HSiCl_3$ (2.54 mmol, 4 eq) in dry MeCN (1 mL) was added to a solution of the proper nitroarene (0.64 mmol, 1 eq) and Et_3N (3.8 mmol, 6 eq) in dry MeCN (4 mL) at 0 °C. After 30 minutes, the reaction mixture was allowed to RT and kept under stirring for 2 hours. Subsequently, the acyl chloride (1.28 mmol, 2 eq) was added. The mixture was kept under stirring at RT for 24 hours. After this period, the reaction was quenched with 10% NaOH (5 mL), stirred for 10 minutes and then extracted with CH_2Cl_2. The collected organic phases were washed with 1M HCl and dried over Na_2SO_4; the solvent was removed under reduced pressure. The product was purified via flash column chromatography.

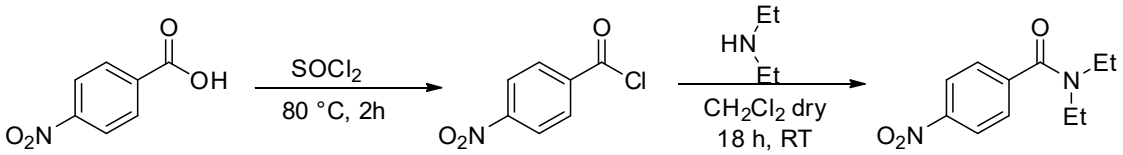
General procedure for the one-pot amidation with thioesters



Working under N_2 atmosphere, a solution of $HSiCl_3$ (2.54 mmol, 4 eq) in dry MeCN (1 mL) was added to a solution of the proper nitroarene (0.64 mmol, 1 eq) and Et_3N (3.8 mmol, 6 eq) in dry MeCN (4 mL) at 0 °C. After 30 minutes, the reaction mixture was allowed to RT and kept under stirring for 2 hours. Subsequently, the thioester (0.95 mmol, 1.5 eq) was added. The mixture was kept under stirring at RT for 24 hours. After this period, the reaction was quenched with 10% NaOH (5 mL), stirred for 10 minutes and then extracted with CH_2Cl_2. The collected organic phases were washed with 1M HCl and dried over Na_2SO_4; the solvent was removed under reduced pressure. The product was purified via flash column chromatography using CH_2Cl_2:EtOAc 95:5 as eluent.

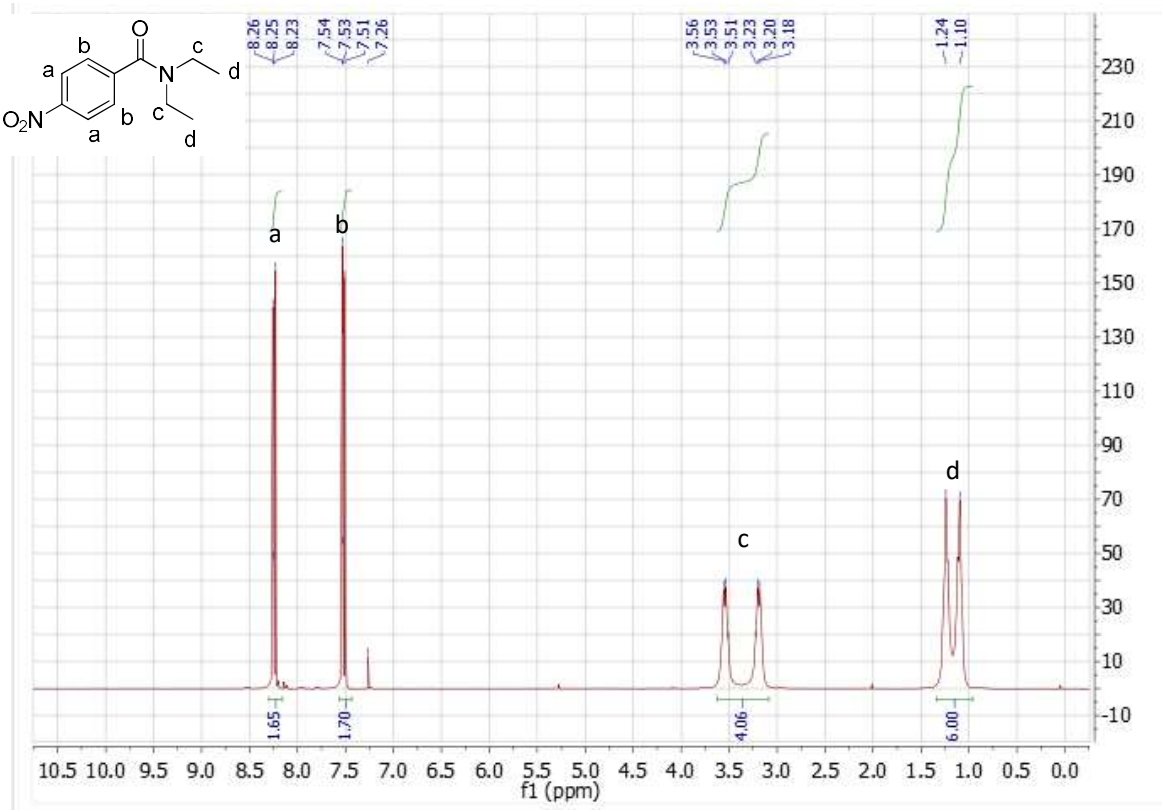
Substrate synthesis

Synthesis of *N,N*-diethyl-4-nitrobenzamide

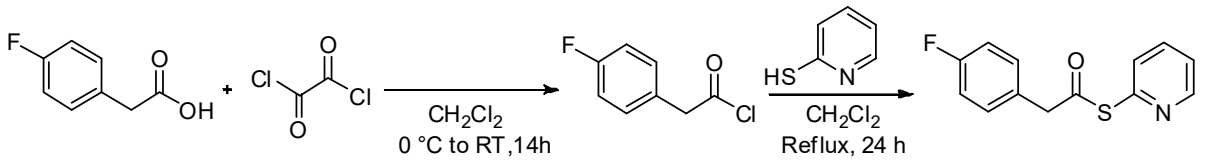


Working under N_2 atmosphere, thionyl chloride (50.3 mmol, 12 eq) was added dropwise to the carboxylic acid (4.19 mmol, 1 eq) at room temperature. The mixture was stirred at 80 °C for 2h. The residual thionyl chloride was removed under reduced pressure. The crude was then dissolved in dry CH_2Cl_2 (3.0 mL) and a solution of diethylamine (10.5 mmol, 2.5 eq) in dry CH_2Cl_2 (1.0 mL) was added. The crude was treated with $NaHCO_3$ss and extracted with CH_2Cl_2; the collected organic phases were dried over Na_2SO_4 and the solvent was removed under reduced pressure. The crude was purified via flash column chromatography with the mixture Hex:EtOAc 3:7 as eluent (Rf = 0.40). The desired product was obtained as a white solid in 63% yield. Analytical data are in agreement with those reported in the literature.[1]

^{1}H-NMR (300 MHz, $CDCl_3$) δ 8.24 (d, J = 8.7 Hz, 2H), 7.52 (d, J = 8.7 Hz, 2H), 3.55 (bd, J = 6.8 Hz, 2H), 3.19 (bd, J = 6.8 Hz, 2H), 1.24 (bt, 3H), 1.10 (bt, 3H).



Synthesis of 4-chlorophenylaceticpyridylthioester



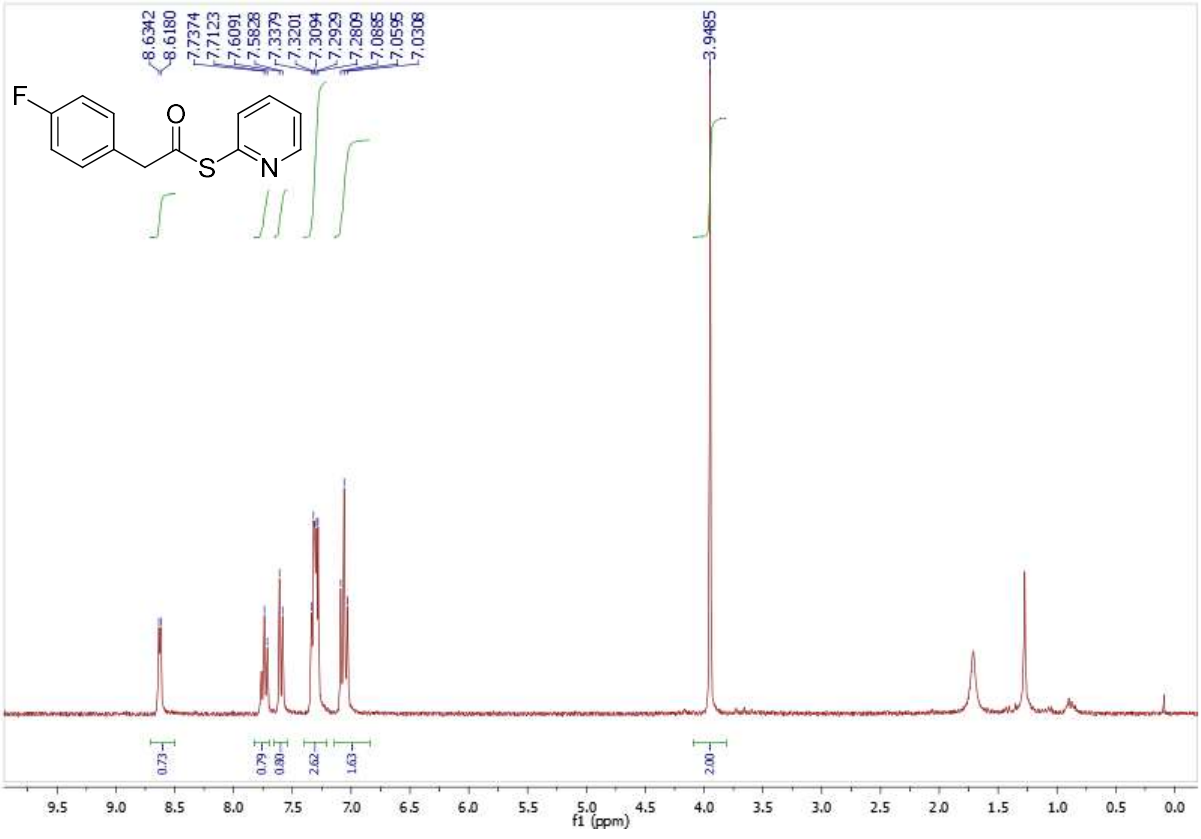
Working under N_2 atmosphere, phenylacetic acid (2.6 mmol,1 eq) is dissolved in 2.60 mL of dry CH_2Cl_2.The reaction mixture was then cooled to 0 °C and oxalyl chloride (7.79 mmol, 3 eq) is added dropwise; the reaction mixture was allowed to room temperature and kept under stirring for 14 h. After this period, the solvent and the residual oxalyl chloride were removed under reduced pressure. The so formed acyl chloride was dissolved in 0.4 mL of dry DCM and added dropwise to a solution of 2-mecaptopyridin (2.6 mmol,1 eq) in dry CH_2Cl_2 (2.3 mL) at 0 °C. The reaction mixture was kept under stirring at this temperature for 10 minutes, then allowed to room temperature and kept under stirring for further 40 minutes. The reaction mixture was treated $NaHCO_3$ss and extracted with CH_2Cl_2 as organic solvent for the extraction; the collected organic phases were dried over Na_2SO_4 and the solvent was removed under reduced pressure. The crude was purified via flash column chromatography using the mixture with the mixture CH_2Cl_2:EtOAc 8:2 as eluent (Rf = 0.45). The product was obtained as an orange oil in 73% yield.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.61 (d, *J* = 4.9 Hz, 1H), 7.72 (td, *J* = 7.6, 1.6 Hz, 1H), 7.57 (d, *J* = 7.9 Hz, 1H), 7.32-7.27 (m, 3H), 7.04 (t, *J* = 8.7 Hz, 2H), 3.93 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 194.46 (s), 162.39 (d, *J* = 246.4 Hz), 151.30 (s), 150.42 (s), 137.15 (s), 131.43 (s), 131.32 (s), 130.07 (s), 128.56 (s), 123.63 (s), 115.82 (s), 115.53 (s), 49.59 (s).

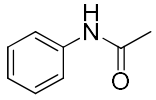
^{19}F NMR (282 MHz, $CDCl_3$) δ -114.64.

HRMS (EI): calcd for C13H10FNOS: 247.046714, found: 247.046750



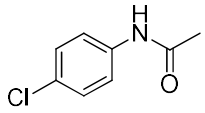
Products characterization

N-phenylacetamide **2a**[2]



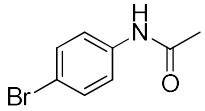
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50 (d, *J* = 8.0 Hz, 2H), 7.31 (t, *J* = 7.8 Hz, 2H), 7.10 (t, *J* = 7.3 Hz, 1H), 2.16 (s, 3H).

N-(4-Chlorophenyl)acetamide **2b**[2]



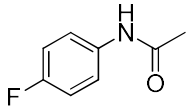
^{1}H NMR (300 MHz, CD_3OD) δ 7.57-7.54 (d, 2H, J = 9.00 Hz), 7.32-7.29 (d, 2H, J = 6.00 Hz), 2.13 (s, 3H). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47 (d, J = 8.6 Hz, 1H), 7.27 (t, J = 13.6 Hz, 2H), 7.22 (bs, 1H), 2.19 (s, 3H).

N-(4-Bromophenyl)acetamide **2c**[3]



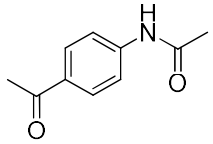
^{1}H NMR (300 MHz, CD_3OD) δ 7.52-7.43 (m, 4H), 2.13 (s, 3H). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.44-7.36 (m, 4H), 2.16 (s, 3H).

N-(4-Fluorophenyl)acetamide **2d**[4]



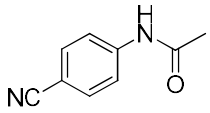
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.63 (bs, 1H), 7.47-7.42 (m, 2H), 6.98 (t, J = 8.7 Hz, 1H), 2.15 (s, 3H).

N-(4-acetylphenyl)acetamide **2e**[4]



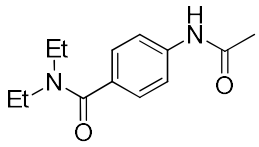
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.95 (d, J = 8.8 Hz, 2H),, 7.80 (bs, 1H), 7.62 (d, J = 8.6 Hz, 2H), 2.57 (s, 3H), 2.21 (s, 3H).

N-(4-Cyanoophenyl)acetamide **2f**[5]



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.65 (AB system, J = 8.9 Hz, 2H), 7.60 (AB system, J = 8.9 Hz, 2H), 2.22 (s, 3H).

4-acetamido-*N,N*-diethylbenzamide **2g**

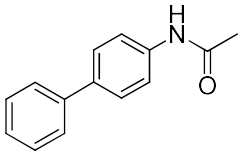


^{1}H NMR (300 MHz, $CDCl_3$) δ 8.48 (bs, 1H), 7.40 (d, J = 8.3 Hz, 2H), 7.22 (d, J = 8.3 Hz, 2H), 3.51 (bs, 2H), 3.27 (s, 2H), 2.13 (s, 3H), 1.25-1.12 (m, 6H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.26 (s), 168.92 (s), 139.12 (s), 132.33 (s), 127.03 (s), 119.83 (s), 43.46 (s), 39.51 (s), 24.36 (s), 14.22 (s), 12.96 (s).

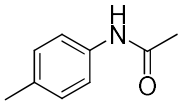
HRMS (EI): calcd for C13H18N2O2: 234.136828, found:234.136040

N-([1,1'-biphenyl]-4-yl)acetamide **2h**[6]



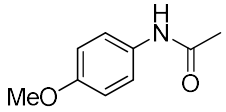
^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.60-7.53 (m, 6H), 7.46 (bs, 1H), 7.43 (t, J = 7.5 Hz, 1H), 7.35-7.31 (m, 1H)

N-(p-tolyl)acetamide **2i**[2]



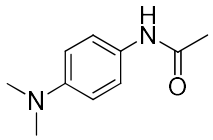
^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.43 (bs, 1H), 7.37 (d, J = 8.3 Hz, 2H), 7.10 (d, J = 8.2 Hz, 2H), 2.30 (s, 3H), 2.14 (s, 3H)

N-(4-Methoxyphenyl)acetamide **2l**[2]



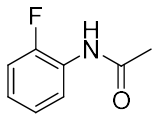
^{1}H-NMR (300 MHz, CD_3OD) δ 7.43 (d, J = 8.8 Hz, 2H), 6.88 (d, J = 8.7 Hz, 2H), 3.79 (s, 3H), 2.11 (s, 3H). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39 (d, J = 9.0 Hz, 2H), 6.86 (d, J = 9.0 Hz, 2H), 3.79 (s, 3H), 2.15 (d, J = 2.9 Hz, 3H).

N-(4-(dimethylamino)phenyl)acetamide **2m**[5]



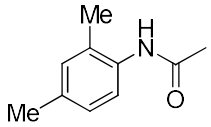
^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.31 (d, J = 8.9 Hz, 2H), 7.01 (d, J = 8.6 Hz. 1H), 6.68 (d, J = 9.0 Hz, 2H), 2.90 (s, 3H), 2.12 (s, 3H).

N-(2-Fluorophenyl)acetamide **2n**[7]



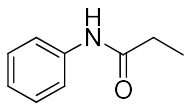
^{1}H NMR (300 MHz, $CDCl_3$) δ 8.29 (t, J = 7.9 Hz, 1H), 7.40 (bs, 1H), 7.15-7.02 (m, 3H), 2.22 (s, 3H).

N-(2,4-Dimethylphenyl)acetamide **2o**[5]



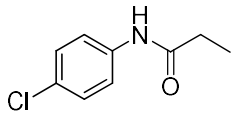
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.52 (d, J = 8.6 Hz, 1H), 7.00 (bs, 2H), 2.28 (s, 3H), 2.21 (s, 3H), 2.17 (s, 3H).

N-Phenylpropionamide **2p**[8]



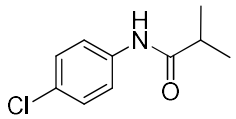
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.58 (bs, 1H), 7.51 (d, J = 8.0 Hz, 2H), 7.29 (t, J = 7.7 Hz, 2H), 7.08 (t, J = 7.3 Hz, 1H), 2.38 (q, J = 7.5 Hz, 2H), 1.23 (t, J = 7.4 Hz, 3H).

N-(4-chlorophenylpropionamide **2q**[9]



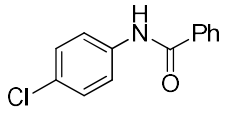
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47 (d, J = 8.7 Hz, 2H), 7.29-7.24 (m, 3H), 2.39 (q, J = 7.6 Hz, 2H), 1.24 (t, J = 7.5 Hz, 3H).

N-(4-chlorophenyl)isobutyramide **2r**[11]



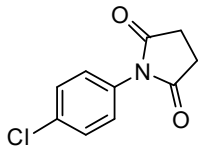
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48 (d, *J* = 8.8 Hz, 2H), 7.28 (d, *J* = 9.6 Hz, 2H), 7.10 (bs, 1H), 2.65-2.43 (m, 1H), 1.26 (d, *J* = 6.9 Hz, 6H).

N-(4-chlorophenyl)benzamide **2s**[11]



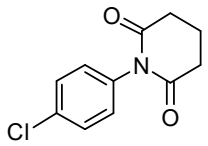
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.86 (d, J = 7.0 Hz, 2H), 7.81 (bs, 1H), 7.68 – 7.40 (m, 5H), 7.34 (d, J = 8.8 Hz, 2H).

N-(4-chlorophenyl)succinimide **3a**[12]



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.45 (d, J = 8.7 Hz, 2H), 7.26 (d, J = 8.7 Hz, 2H), 2.90 (s, 3H). ^{1}H NMR (300 MHz, MeOD) δ 7.50 (d, J = 8.7 Hz, 2H), 7.32 (d, J = 8.7 Hz, 2H), 2.87 (s, 3H).

N-(4-chlorophenyl)glutarimide **3b**

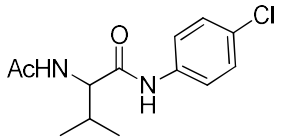


^{1}H NMR (300 MHz, $CDCl_3$) δ 7.44 (d, J = 8.6 Hz, 2H), 7.05 (d, J = 8.6 Hz, 2H), 2.84 (t, J = 6.6 Hz, 4H), 2.22-1.99 (m, 2H).

^{3}C NMR (75 MHz, $CDCl_3$) δ 172.45 (s), 134.42 (s), 133.60 (s), 129.90 (s), 129.47 (s), 32.97 (s), 17.17 (s).

HRMS (EI): calcd for C11H10N1O2Cl1: 223.040006, found: 223.039470

2-acetamido-*N*-(4-chlorophenyl)-3-methylbutanamide **4**

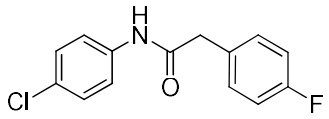


^{1}H NMR (300 MHz, $CDCl_3$) δ 9.15 (bs, 1H), 7.51 (d, J = 8.8 Hz, 2H), 7.25 (d, J = 8.8 Hz, 2H), 6.56 (d, J = 8.8 Hz, 1H), 4.57-4.51 (m, 1H), 2.18-2.05 (m, 1H), 2.08 (s, 3H), 1.00 (d, J = 4.6 Hz, 3H), 0.98 (d, J = 4.7 Hz, 3H).

^{3}C NMR (75 MHz, $CDCl_3$) δ 170.69 (s), 170.06 (s), 136.43 (s), 129.37 (s), 128.92 (s), 121.20 (s), 59.39 (s), 31.31 (s), 23.31 (s), 19.24 (s), 18.42 (s).

HRMS (EI): calcd for C13H17ClN2O2: 268.097856, found: 268.093990

N-(4-chlorophenyl)-2-(4-fluorophenyl)acetamide **5a**



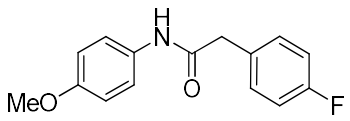
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38 (AB, *J* = 8.8 Hz, 2H), 7.33-7.24 (m, 4H), 7.09 (t, *J* = 8.6 Hz, 2H), 6.99 (bs, 1H), 3.71 (s, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 168.82 (s), 162.20 (d, J = 147.15), 136.10 (s), 131.13 (s), 131.07 (s), 129.92 (s), 129.60 (s), 121.10 (s), 116.10 (s), 116.30 (s), 43.80 (s).

^{19}F NMR (282 MHz, $CDCl_3$) δ -114.33 (s).

HRMS (EI): calcd for C14H11ClFNO: 263.051320, found: 263.051610

N-(4-methoxyphenyl)-2-(4-fluorophenyl)acetamide **5b**



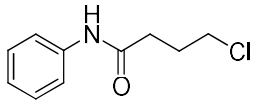
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33-7.29 (m, 3H), 7.08 (t, J = 8.6 Hz, 2H), 6.93 (bs, 1H), 6.83 (d, J = 9.0 Hz, 2H), 3.77 (s, 3H), 3.69 (s, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 168.76 (s), 163.83 (s), 160.57 (s), 156.60 (s), 131.13 (s), 131.02 (s), 130.61 (s), 130.37 (s), 130.32 (s), 121.84 (s), 116.14 (s), 115.85 (s), 114.09 (s), 55.46 (s), 43.63 (s).

^{19}F NMR (282 MHz, $CDCl_3$) δ -114.69 (s).

HRMS (EI): calcd for C15H14FNO2: 259.100857, found: 259.100830

4-chloro-*N*-phenylbutanamide **6a**



White solid Hex/EtOAc 6/4, Rf= 0.40

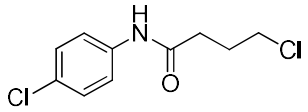
m.p. 188-190°C, then dec; IR (nujol): ν 3410, 1770, 1602, 1496, 1455, 1379 cm^{-1}

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47 (d, J = 8.6 Hz, 2H), 7.29 (d, J = 8.7 Hz, 2H), 7.18 (bs, 2H), 3.67 (t, J = 6.0 Hz, 2H), 2.57 (t, J = 7.0 Hz, 2H), 2.21 (p, J = 6.6 Hz, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 170.07 (s), 137.70 (s), 129.04 (s), 124.44 (s), 119.93 (s), 44.47 (s), 34.14 (s), 27.92 (s).

HRMS (EI): calcd for C10H12ClNO: 197.060742, found: 197.061230

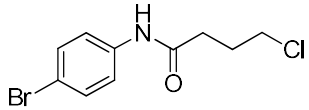
4-chloro-*N*-(4-chlorophenyl)butanamide **6b**[13]



White solid Hex/EtOAc 8/2, Rf= 0.25

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47 (d, *J* = 8.6 Hz, 2H), 7.29 (d, *J* = 8.7 Hz, 2H), 7.18 (bs, 2H), 3.67 (t, *J* = 6.0 Hz, 2H), 2.57 (t, *J* = 7.0 Hz, 2H), 2.21 (p, *J* = 6.6 Hz, 2H).

4-chloro-*N*-(4-bromophenyl)butanamide **6c**



Pale yellow solid Hex/EtOAc 1:1, Rf= 0.45

m.p. 230-231°C, then dec; IR (nujol): ν 3383, 1770, 1538, 1486, 1460, 1376 cm^{-1}

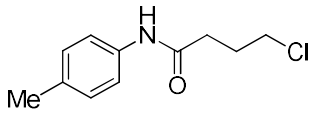
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47-7.38 (m, 4H), 7.19 (bs, 1H), 3.66 (t, *J* = 6.1 Hz, 2H), 2.56 (t, *J* = 7.0 Hz, 2H), 2.20 (p, *J* = 6.5 Hz, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 170.16 (s), 136.76 (s), 131.99 (s), 121.52 (s), 117.03 (s), 44.40 (m), 34.11 (s), 27.81 (s).

HRMS (EI): calcd for C10H11BrClNO: 274.971253, calcd for C10H11BrNO

[M-Cl]: 240.002400, found: 240.002190

4-chloro-*N*-(4-methylphenyl)butanamide **6d**



White solid Hex/EtOAc 7:3, Rf= 0.54

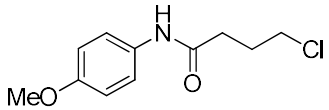
m.p. 201°C, then dec; IR (nujol): ν 3414, 1771, 1596, 1508, 1460, 1380 cm^{-1}

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.55 (bs, 1H), 7.40 (d, J = 8.2 Hz, 2H), 7.12 (d, J = 7.9 Hz, 2H), 3.65 (t, J = 6.0 Hz, 2H), 2.54 (t, J = 7.0 Hz, 2H), 2.33 (s, 3H), 2.21 (d, J = 6.4 Hz, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 170.06 (s), 135.18 (s), 134.06 (s), 129.48 (s), 120.12 (s), 44.50 (s), 34.08 (s), 28.02 (s), 20.86 (s).

HRMS (EI): calcd for C11H14ClNO: 211.076392, found: 211.078630

4-chloro-*N*-(4-methoxyphenyl)butanamide **6e**[14]

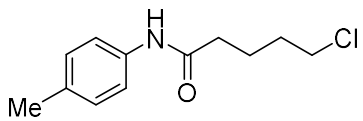


White solid Hex/EtOAc 3:7, Rf= 0.40

m.p. 208°C, then dec; IR (nujol): ν 3382, 1770, 1604, 1510, 1457, 1380 cm^{-1}

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39 (d, J = 9.0 Hz, 2H), 7.26 (bs, 1H), 6.85 (d, J = 8.9 Hz, 2H), 3.73 (s, 3H), 3.65 (t, J = 6.1 Hz, 2H), 2.53 (t, J = 7.0 Hz, 2H), 2.15-2.23 (m, 2H).

5-chloro-*N*-(4-methylphenyl)-pentanamide **7**



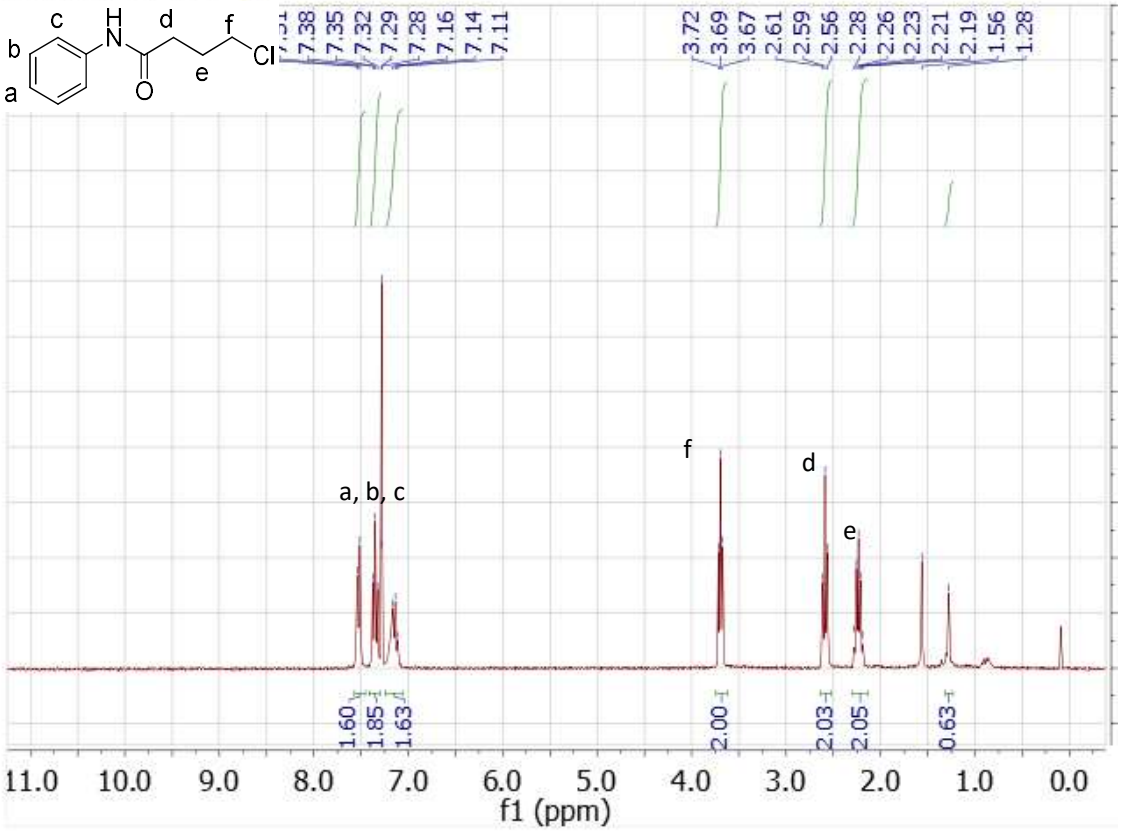
Pale yellow solid. Hex/EtOAc 7:3, Rf= 0.52

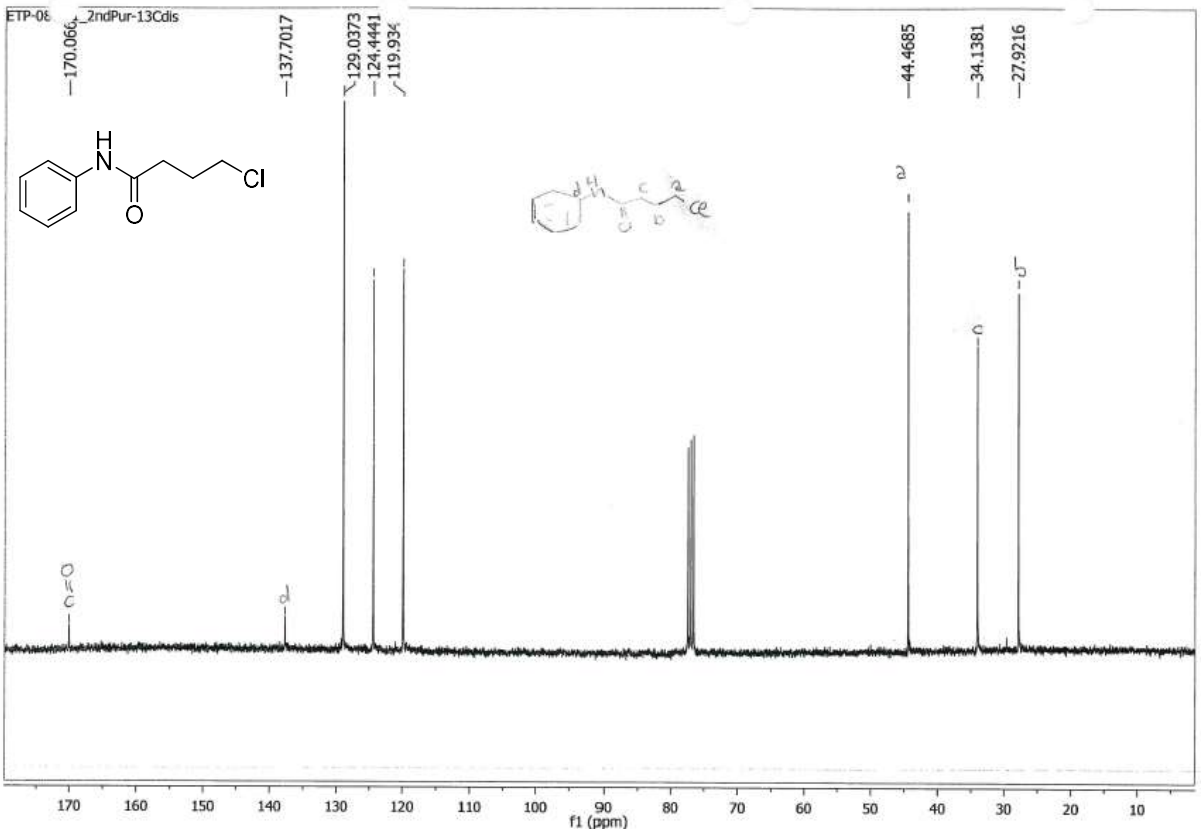
m.p. 219°C, then dec; IR (nujol): ν 3382, 1771, 1597, 1508, 1457, 1378 cm^{-1}

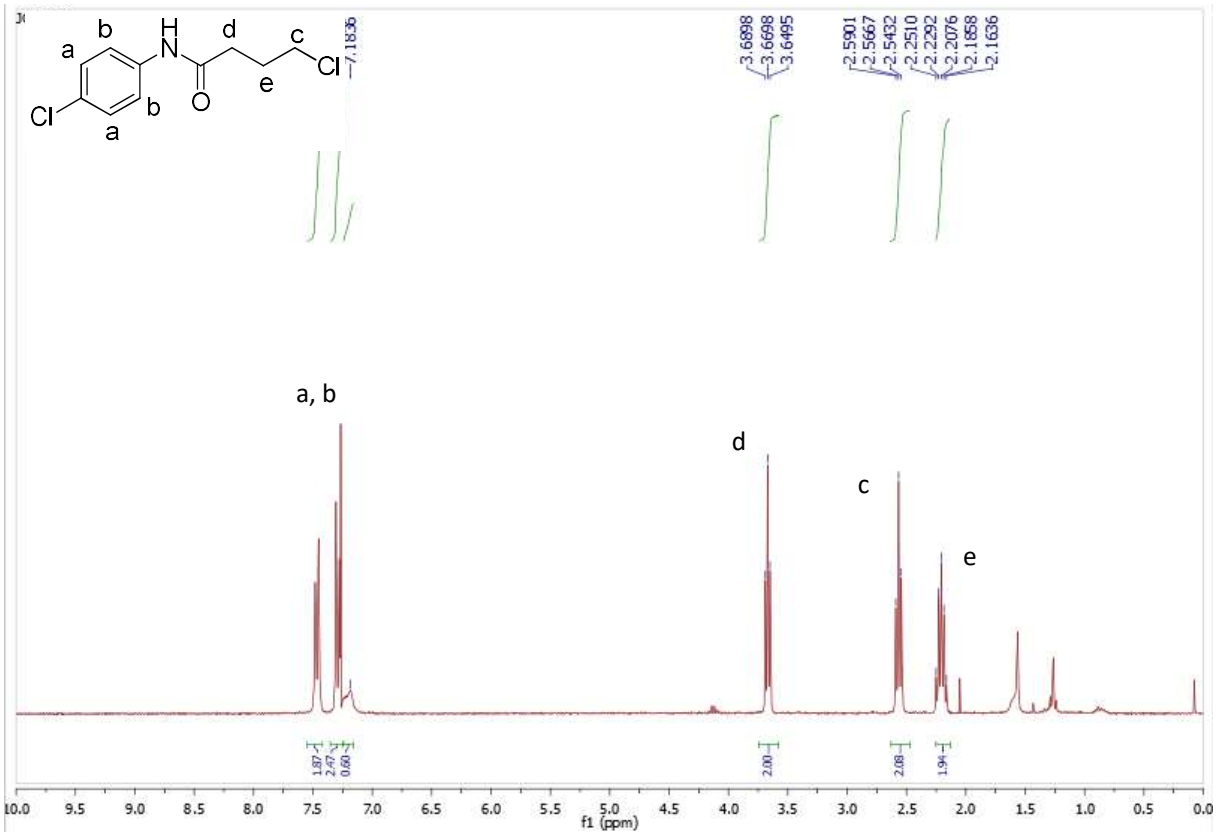
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.82 (Bs, 1H), 7.40 (d, J = 8.4 Hz, 2H), 7.11 (d, J = 8.2 Hz, 2H), 3.62-3.37 (m, 2H), 2.43-2.33 (m, 2H), 2.32 (s, 3H), 2.00-1.66 (m, 4H)

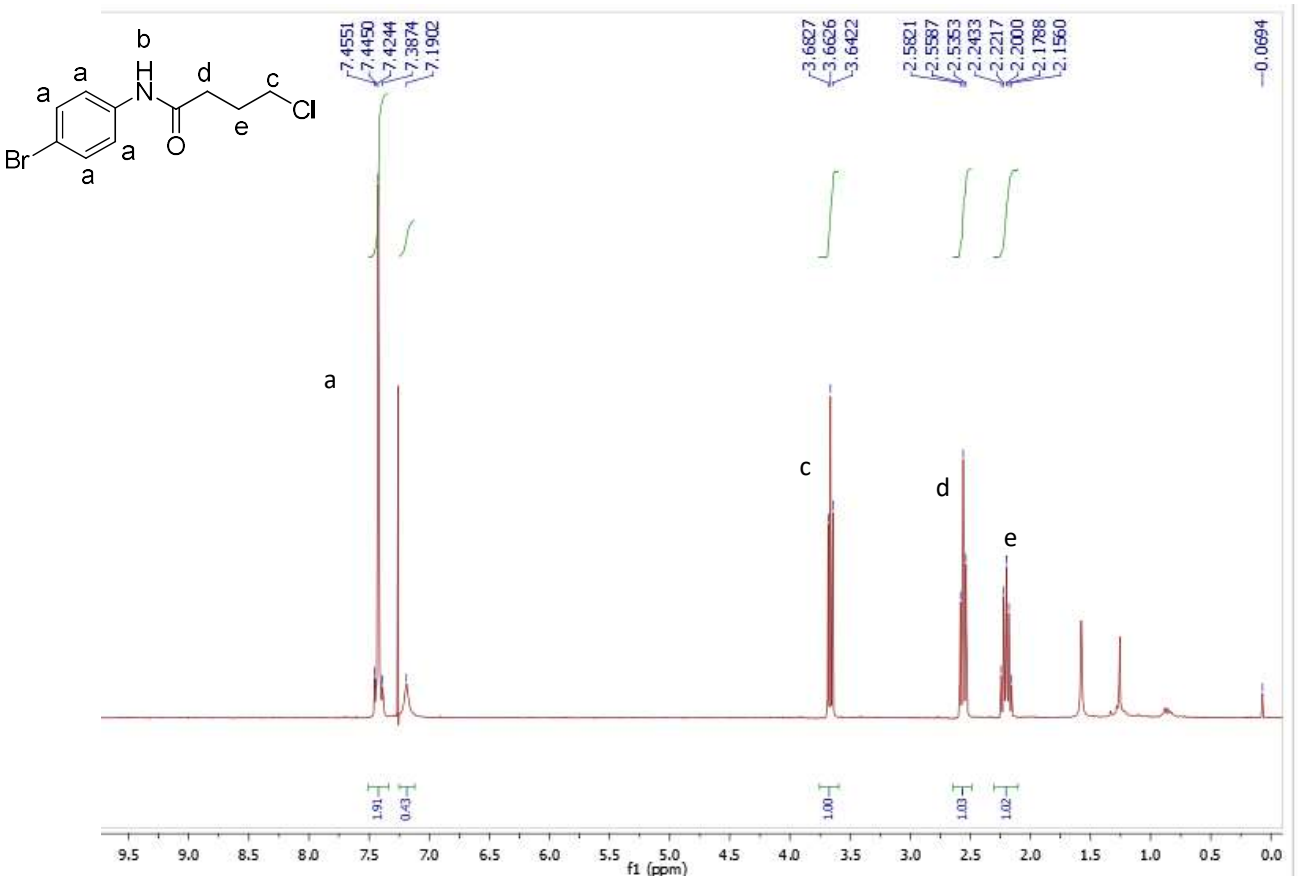
^{13}C NMR (75 MHz, $CDCl_3$) δ 171.07 (s), 135.33 (s), 133.97 (s), 129.41 (s), 120.30 (s), 44.61 (s), 36.45 (s), 31.94 (s), 22.93 (s), 20.87 (s).

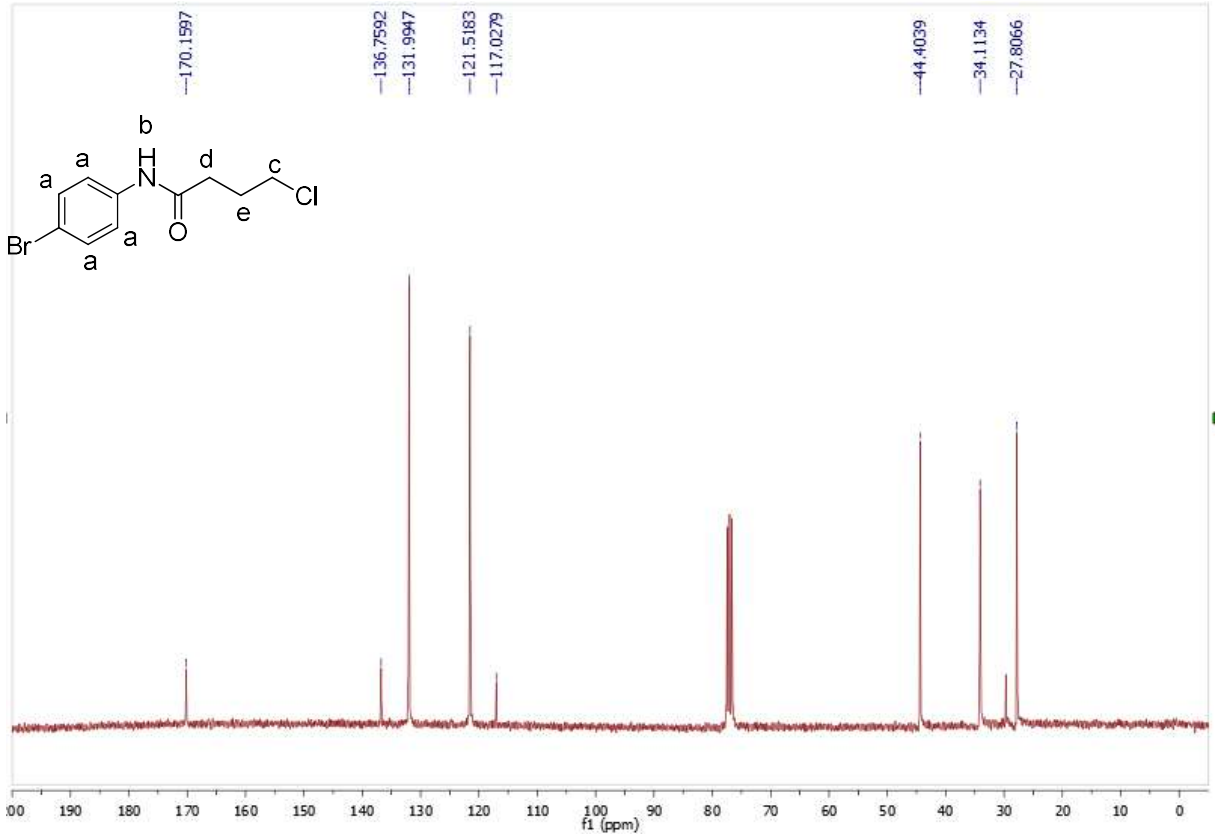
^{1}H and ^{13}C NMR spectra of lactones derived butanamides

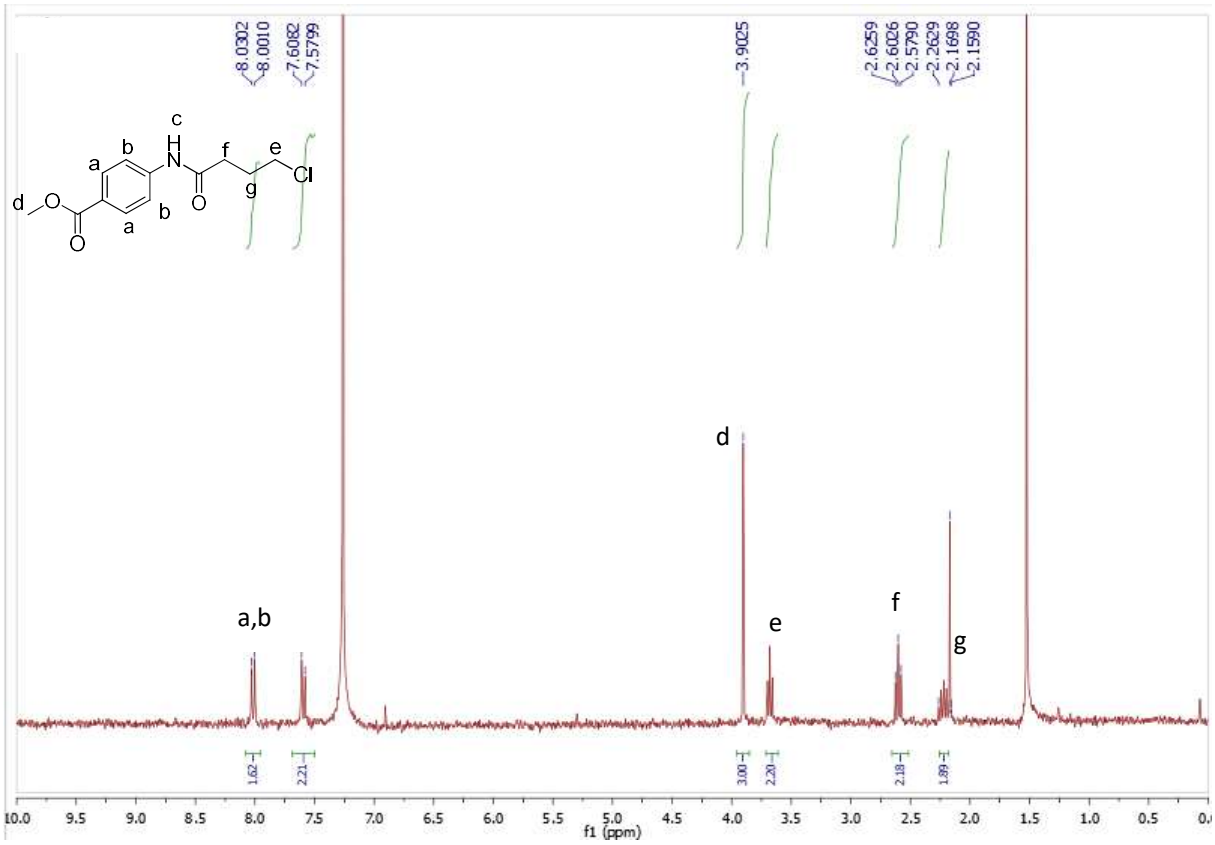


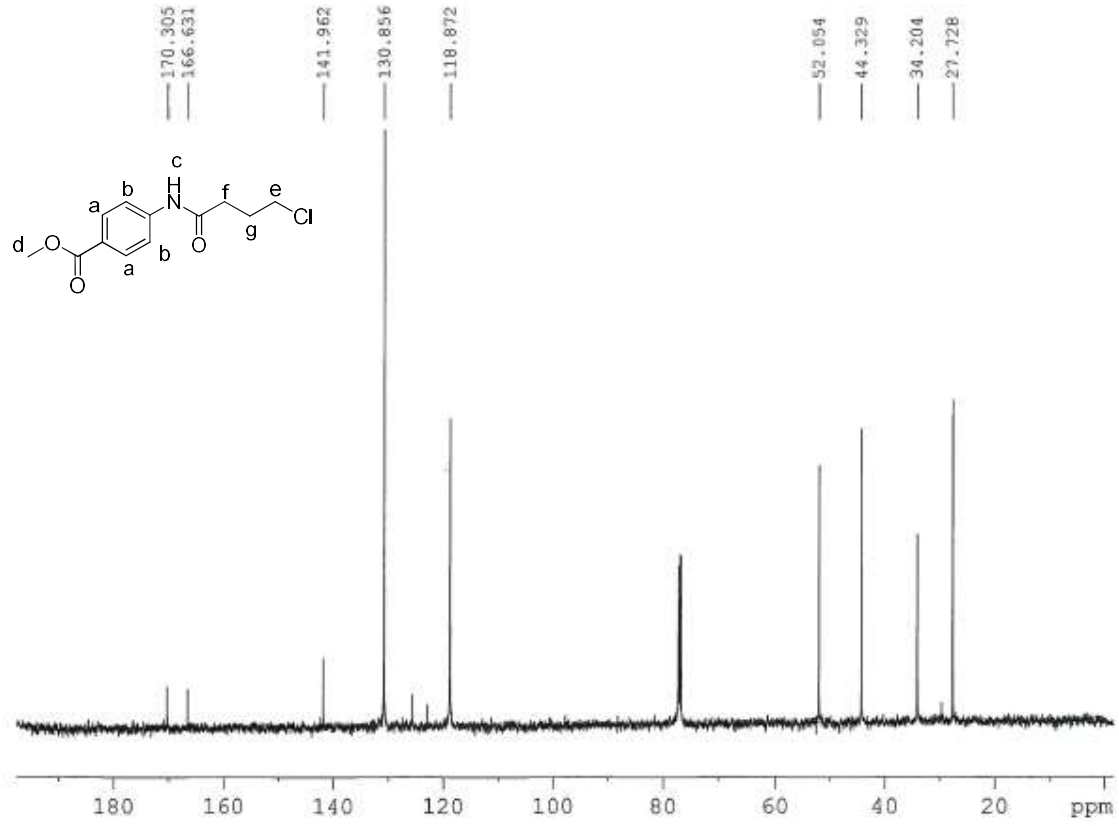


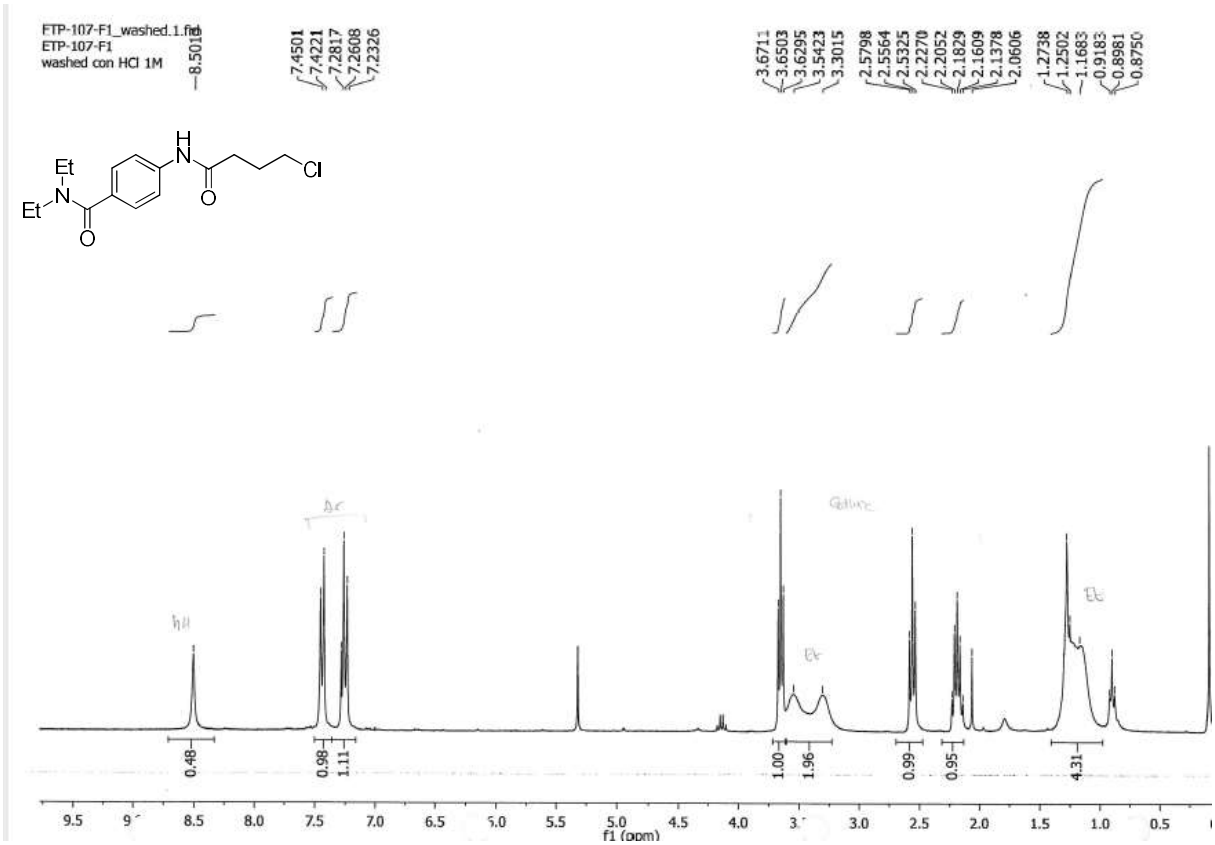

a
b
H
N
d
c
Cl
e
O
b
a
Cl
7.1836
3.6898
3.6698
3.6495
2.5901
2.5667
2.5432
2.2510
2.2292
2.2076
2.1858
2.1636
a, b
d
c
e
1.87
2.47
0.60
2.00
2.08
1.94
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

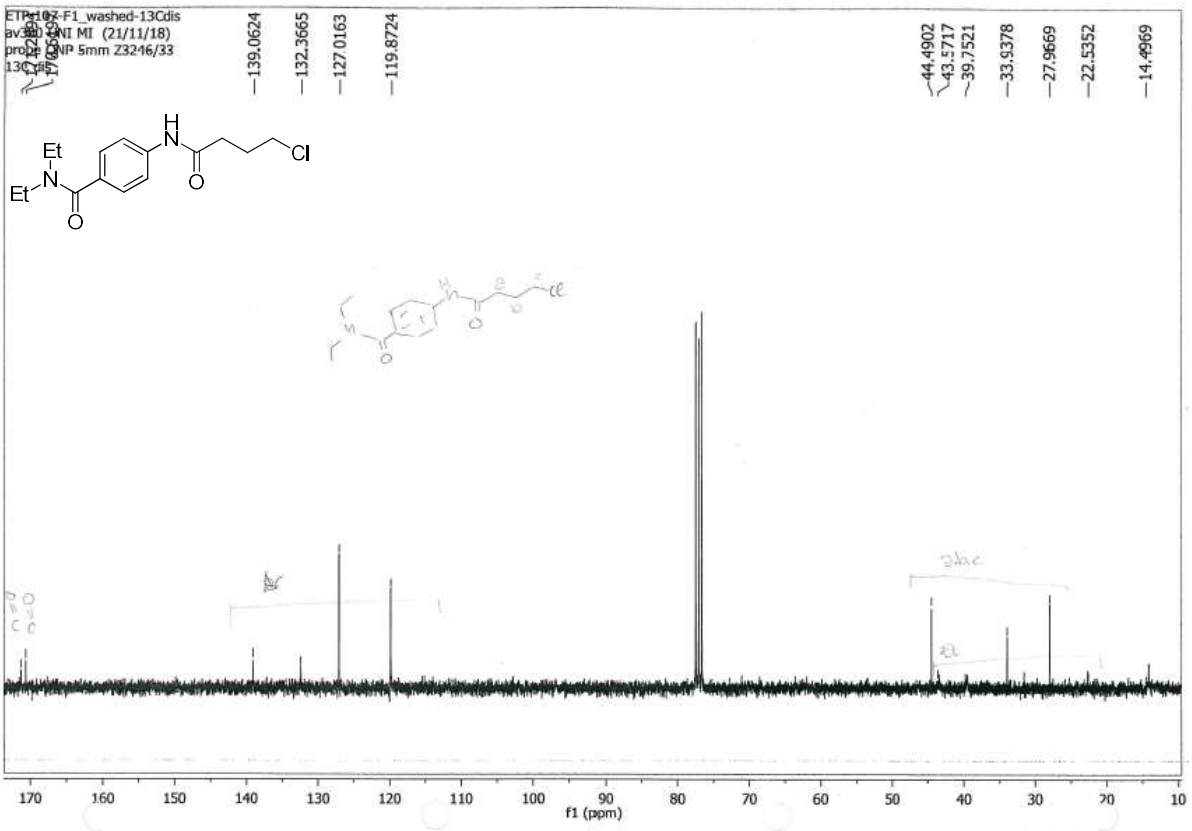

b
a
a
H
N
d
c
Cl
a
e
Br
a
O
a
7.4551
7.4450
7.4244
7.3874
7.1902
3.6827
3.6626
3.6422
2.5821
2.5587
2.5353
2.2433
2.2217
2.2000
2.1788
2.1560
0.0694
a
c
d
e
1.91
0.43
1.00
1.03
1.02
f1 (ppm)

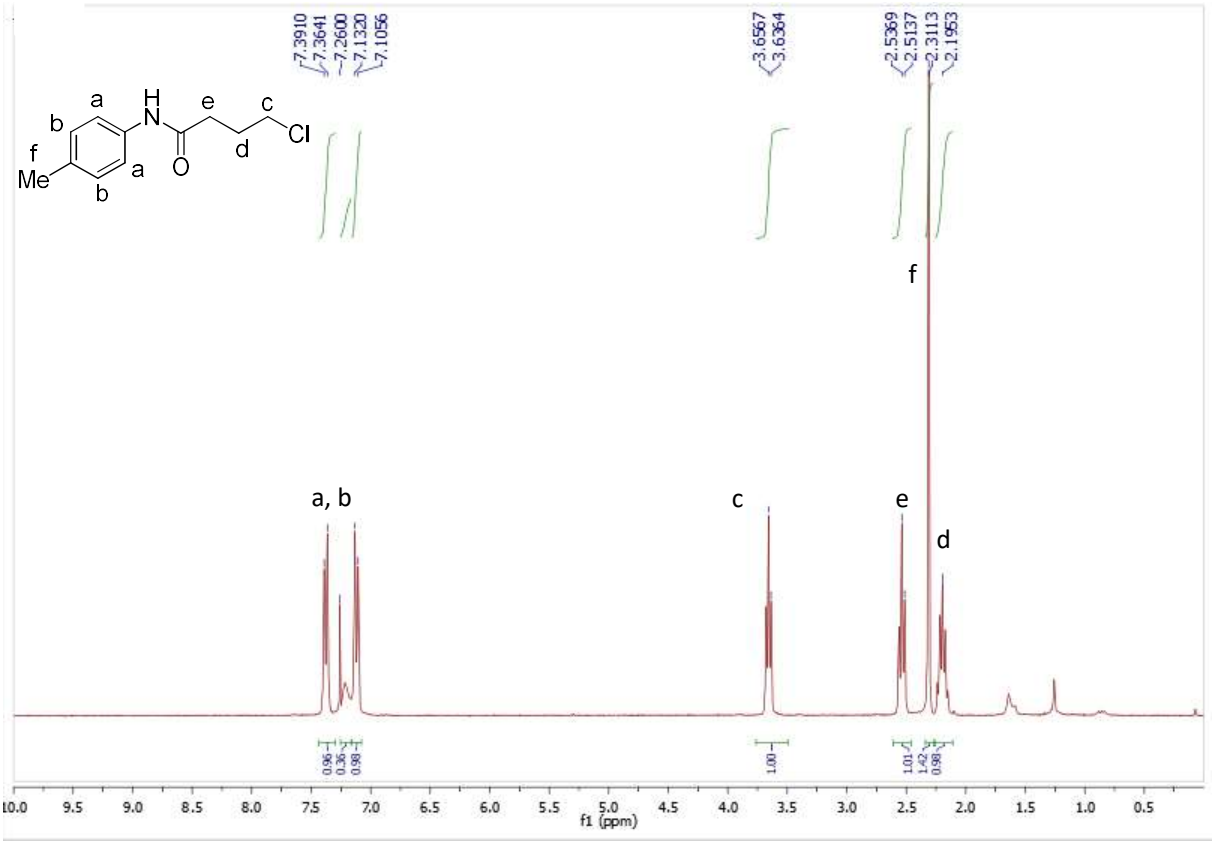

170.1597
136.7592
131.9947
121.5183
117.0279
44.4039
34.1134
27.8066
b
a
a
H
N
d
c
Cl
e
Br
a
O
a
f1 (ppm)

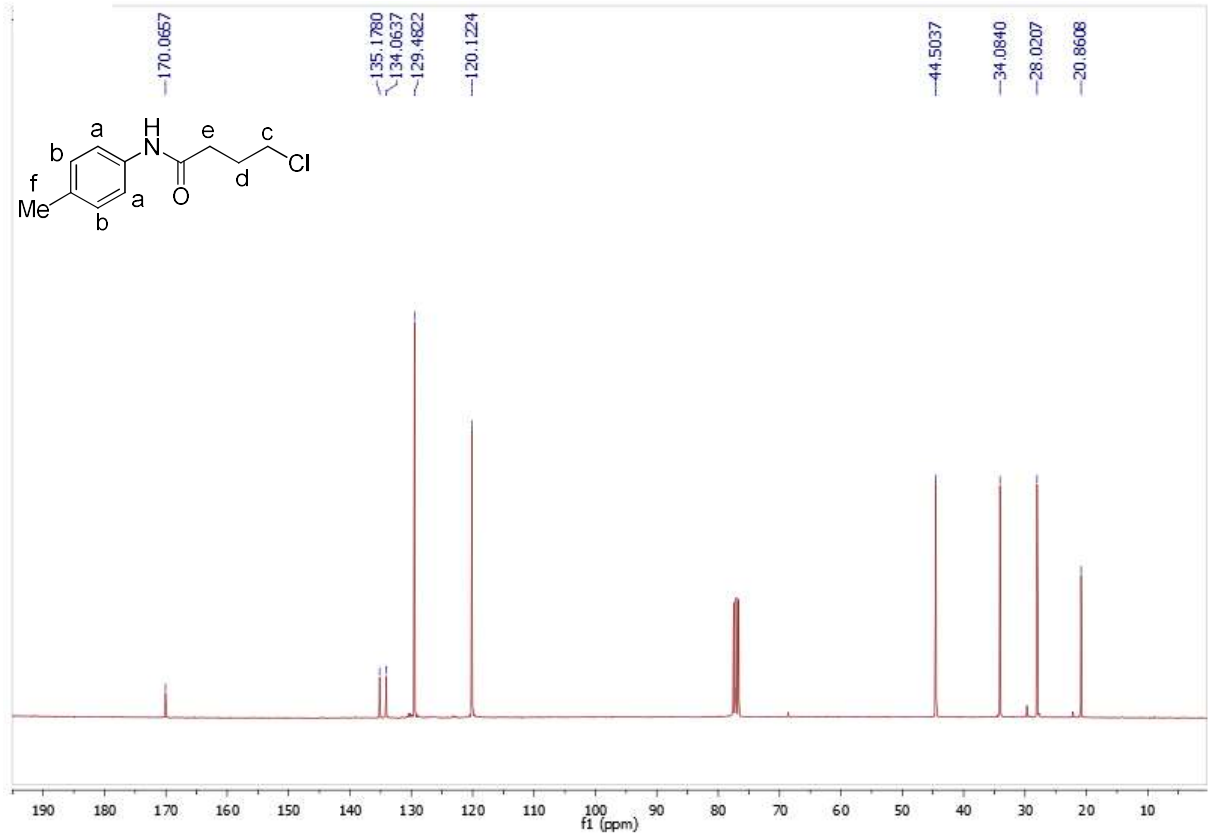

8.0302
8.0010
7.6082
7.5799
3.9025
2.6259
2.6026
2.5790
2.2629
2.1698
2.1590
a,b
d
e
f
g
1.62
2.21
3.00
2.20
2.18
1.89
f1 (ppm)

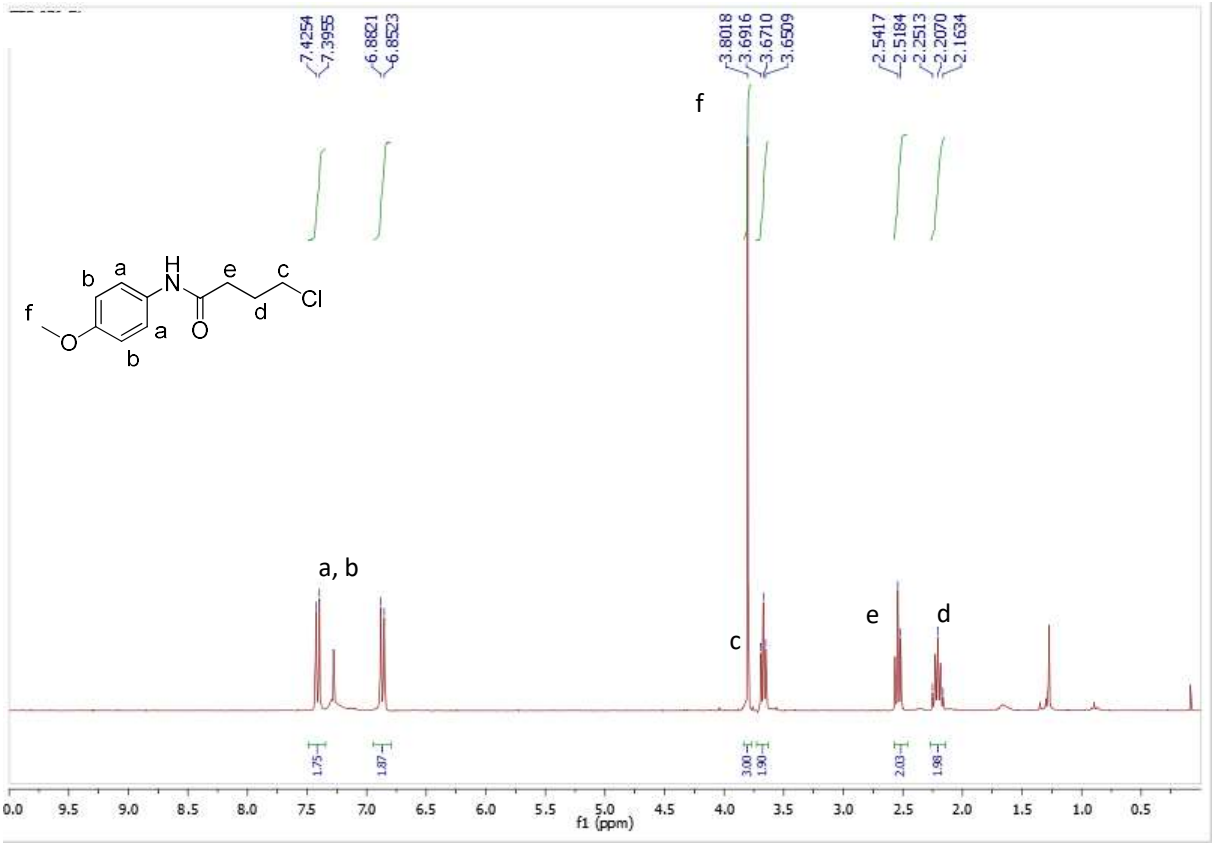

170.305
166.631
141.962
130.856
118.872
52.054
44.329
34.204
27.728
ppm

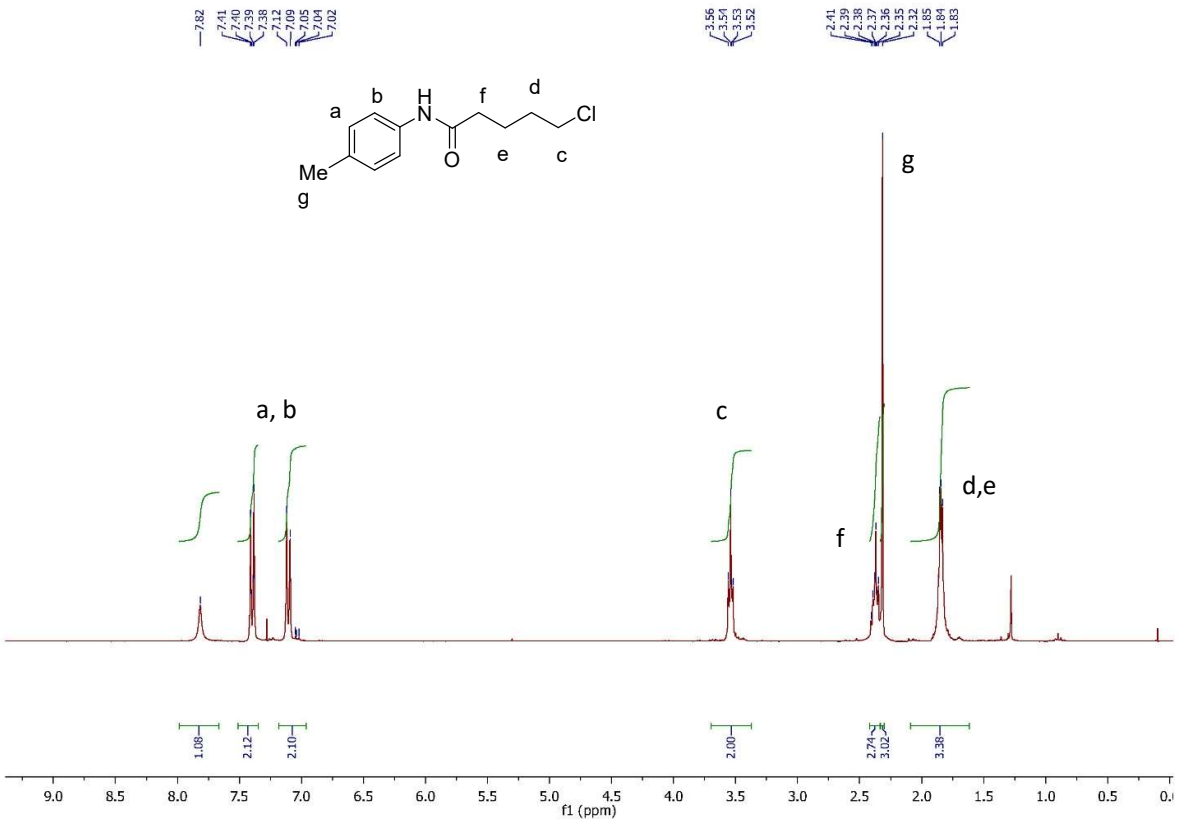

FTP-107-F1_washed.1.fid
ETP-107-F1
washed con HCl 1M
8.5018
7.4501
7.4221
7.2817
7.2608
7.2326
3.6711
3.6503
3.6295
3.5423
3.3015
2.5798
2.5564
2.5325
2.2270
2.2052
2.1829
2.1609
2.1378
2.0606
1.2738
1.2502
1.1683
0.9183
0.8981
0.8750
Et
N
Et
O
H
N
O
Cl
0.48
0.98
1.11
1.00
1.96
0.99
0.95
4.31
f1 (ppm)


139.0624
132.3665
127.0163
119.8724
44.4902
43.5717
39.7521
33.9378
27.9669
22.5352
14.4969
Et
N
Et
O
H
N
O
Cl
f1 (ppm)


7.3910
7.3641
7.2600
7.1320
7.1056
3.6567
3.6364
2.5369
2.5137
2.3113
2.1953
a
b
f
Me
a
b
H
N
O
e
c
d
Cl
a, b
c
e
f
d
0.96
0.36
0.98
1.00
1.01
1.42
0.98
f1 (ppm)


170.0657
135.1780
134.0637
129.4822
120.1224
44.5037
34.0840
28.0207
20.8608
a
b
f
Me
a
b
H
N
O
e
c
d
Cl
f1 (ppm)


7.4254
7.3955
6.8821
6.8523
3.8018
3.6916
3.6710
3.6509
2.5417
2.5184
2.2513
2.2070
2.1634
f
a, b
c
e
d
1.75
1.87
3.00
1.90
2.03
1.98
f1 (ppm)


7.82
7.41
7.40
7.39
7.38
7.12
7.09
7.05
7.04
7.02
3.56
3.51
3.53
3.52
2.41
2.39
2.38
2.37
2.36
2.35
2.32
1.85
1.84
1.83
a
b
H
N
f
d
Cl
Me
g
O
e
c
g
a, b
c
f
d,e
1.08
2.12
2.10
2.00
2.74
3.02
3.38
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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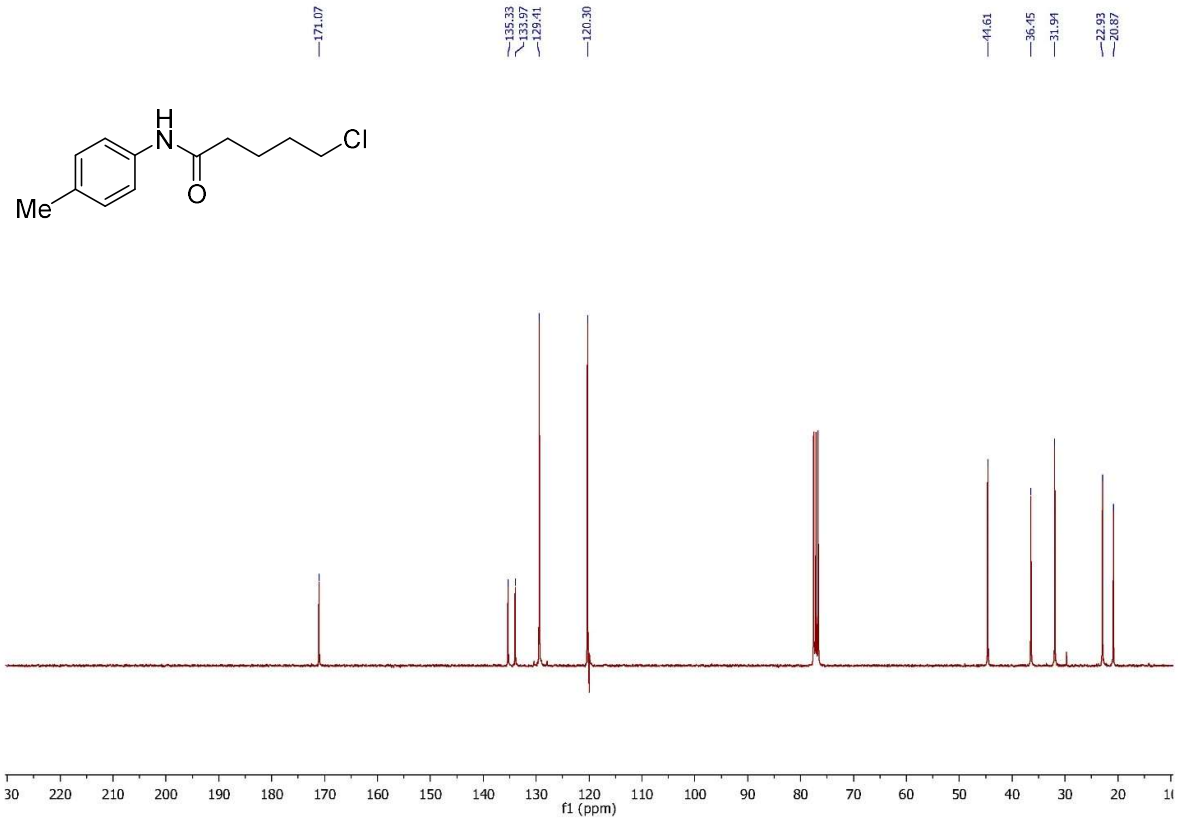
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


171.07
135.33
133.97
129.41
120.30
44.61
36.45
31.91
22.93
20.87
H
N
Cl
Me
O
30
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)

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