Regioselective Rhodium(I)-Catalyzed Hydroarylation of Protected Allylic Amines with Arylboronic Acids

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Supporting Information

Table of Contents

General Experimental. Unless otherwise noted, reactions were carried out under argon atmosphere, in flame-dried, single-neck, round bottom flasks fitted with a rubber septum, with magnetic stirring. Air- or water-sensitive liquids and solutions were transferred via syringe or stainless steel canula. Organic solutions were concentrated by rotary evaporation at 23–40 °C under 40 Torr (house vacuum). Analytical thin layer chromatography (TLC) was performed with Silicycle™ normal phase glass plates (0.25 mm, 60-A pore size, 230-400 mesh). Visualization was done under a 254 nm UV light source and generally by immersion in potassium permanganate ($KMnO_4$), followed by heating using a heat gun. Purification of reaction products was generally done by flash chromatography with Silicycle™ Ultra-Pure 230-400 mesh silica gel, as described by Still *et al*.[1]

Materials. $[Rh(COD)OH]_2$, $[Rh(COD)Cl]_2$, BINAP and other phosphine ligands were purchased from Strem Chemicals Inc. and used as received. Supplies of $[Rh(COD)_2]OTf$ and Josiphos ligands were generously provided by Solvias Inc. Unless otherwise indicated, boronic acids were obtained from Combi-Blocks, Inc. and Sigma-Aldrich, Inc., and used without further purification. Tetrahydrofuran, 1,4-dioxane and toluene were purified by distillation under N_2 from Na/benzophenone immediately prior to use. Ether and dichloromethane were purified by the method of Pangborn *et al*.[2] Hexanes used for chromatography was purified by simple distillation before use. Substrates 1a,[3] 1b,[3] 1c,[4] 1d,[5] 1e,[5] 1f,[6] 1g,[7] 1h,[8] 1j,[8] 1l,[9] 4a,[10]

[1] Still, W. C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923.
[2] Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518.
[3] Morino, Y.; Hidaka, I.; Oderaotoshi, Y.; Komatsu, M.; Minakata, S. *Tetrahedron* **2006**, *62*, 12247.
[4] Whitehead, C. W.; Traverso, J. J. *J. Am. Chem. Soc.* **1958**, *80*, 2182.
[5] De Amici, M.; De Micheli, C.; Misani, V. *Tetrahedron* **1990**, *46*, 1975.
[6] Bowen, R. D.; Maccoll, A. *Org. Mass Spectrom.* **1989**, *24*, 113.

4b[3] and 4c[11] are known compounds and were prepared according to the literature procedures. *N*-Vinylphthalimide **4c** was purchased from Sigma-Aldrich, Inc.

Instrumentation. Proton nuclear magnetic resonance spectra (^{1}H NMR) spectra and carbon nuclear magnetic resonance spectra (^{13}C NMR) were recorded at 23 °C with a Varian Mercury 400 (400 MHz/100 MHz) NMR spectrometer equipped with a Nalorac4N-400 probe, or a Varian 400 (400 MHz/100 MHz) NMR spectrometer equipped with ATB8123-400 probe. Recorded shifts for protons are reported in parts per million (δ scale) downfield from tetramethylsilane and are referenced to residual protium in the NMR solvents ($CHCl_3$: δ 7.26, $CHDCl_2$: δ 5.29, C_6HD_5: δ 7.15, CD_2HOD: δ 3.30). Chemical shifts for carbon resonances are reported in parts per million (δ scale) downfield from tetramethylsilane and are referenced to the carbon resonances of the solvent ($CDCl_3$: δ 77.0, CH_2Cl_2: δ 53.8, C_6D_6: δ 128.0, CD_3OD: δ 49.2). Data are represented as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet,, qn = quintuplet, sx = sextet, sp = septuplet, m = multiplet, br = broad), and coupling constant (*J*, Hz). Infrared (IR) spectra were obtained using a Perkin-Elmer Spectrum 1000 FT-IR spectrometer as a neat film on a NaCl plate. Data is presented as frequency of absorption (cm^{-1}). High resolution mass spectra were obtained from a SI2 Micromass 70S-250 mass spectrometer (EI) or an ABI/Sciex Qstar mass spectrometer (ESI). Melting points were taken on a Fisher-Johns melting point apparatus and are uncorrected.

Experimental Procedures. General procedure for Rh(I)-catalyzed hydroarylation of protected allylic amines with arylboronic acids. To an oven-dried 2 dram vial equipped with a magnetic stir bar was added $[Rh(COD)OH]_2$ (3.0 mg, 0.0066 mmol, 2 mol%) and BINAP (12.0 mg, 0.019 mmol, 6 mol%). The vial was sealed and flushed with argon, then distilled 1,4-dioxane (1.0 mL) was added and the mixture was stirred for 10 min in a 50 ^{o}C oil bath, the solution turned orange/reddish indicating the formation of the catalyst. Protected allylic amine (0.32 mmol, 1.0 eq.) and phenylboronic acid (98 mg, 0.80 mmol, 2.5 eq.) were added together as a solution in distilled 1,4-dioxane (1.5 mL) and the reaction mixture was heated in a 75 ^{o}C oil bath. After 12 hrs, the solvent was removed *in vacuo*. The crude was applied to the top of a column of silica gel and purified by column chromatography (EtOAc/hexanes as elution gradient).

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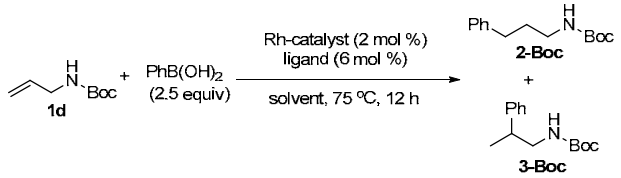
[7] Stille, J. K.; Becker, Y. *J. Org. Chem.* **1980**, *45*, 2139.
[8] Gheorghe, A.; Quiclet-Sire, B.; Vila, X.; Zard, S. *Tetrahedron* **2007**, *63*, 7187.
[9] Sarkar, N.; Banerjee, A.; Nelson, S. G. *J. Am. Chem. Soc.* **2008**, *130*, 9222.
[10] Taillier, C.; Hameury, T.; Bellosta, V.; Cossy, J. *Tetrahedron* **2007**, *63*, 4472.
[11] Marcotullio, M. C.; Campagna, V.; Sternativo, S.; Costantino, F.; Curini, M. *Synthesis* **2006**, 2760.

Optimization Data. Screening of Rh catalyst, ligand and solvent is shown in the following table:

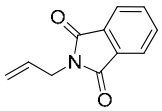


Entry	***Rh-catalyst***	***Ligand***	***Solvent***	***NMR yield (%)[a]***
1[c]	$[Rh(COD)OH]_2$	BINAP	dioxane	70[b]
2	$[Rh(COD)Cl]_2$	BINAP	dioxane	<5
3	$[Rh(COD)_2]OTf$	BINAP	dioxane	12
4	$[Rh(COD)OH]_2$	/	dioxane	<5
5	$[Rh(COD)OH]_2$	Tol-BINAP	dioxane	20
6	$[Rh(COD)OH]_2$	Xyl-BINAP	dioxane	<5
7	$[Rh(COD)OH]_2$	Trost ligand	dioxane	<5
8	$[Rh(COD)OH]_2$	Xyl-P-Phos	dioxane	27
9	$[Rh(COD)OH]_2$	P-Phos	dioxane	28
10	$[Rh(COD)OH]_2$	PPF-P^{t}Bu$_2$	dioxane	<5
11	$[Rh(COD)OH]_2$	PPF-PCy$_2$	dioxane	<5
12	$[Rh(COD)OH]_2$	Tunephos	dioxane	<5
13	$[Rh(COD)OH]_2$	DPPM	dioxane	22
14	$[Rh(COD)OH]_2$	DPPE	dioxane	12
15	$[Rh(COD)OH]_2$	DPPP	dioxane	27
16	$[Rh(COD)OH]_2$	DPPB	dioxane	<5
17	$[Rh(COD)OH]_2$	BIPHEP	dioxane	66[d]
18	$[Rh(COD)OH]_2$	S-Phos	dioxane	<5
19	$[Rh(COD)OH]_2$	X-Phos	dioxane	<5
20	$[Rh(COD)OH]_2$	Xantphos	dioxane	<5
21	$[Rh(COD)OH]_2$	PPh_3	dioxane	<5
22	$[Rh(COD)OH]_2$	Dolefin-type	dioxane	13
23	$[Rh(COD)OH]_2$	BINAP	toluene	<5
24	$[Rh(COD)OH]_2$	BINAP	MeCN	<5
25	$[Rh(COD)OH]_2$	BINAP	DMF	<5
26	$[Rh(COD)OH]_2$	BINAP	MeOH	<5
27	$[Rh(COD)OH]_2$	BINAP	THF	45
28	$[Rh(COD)OH]_2$	BINAP	DME	<5
29	$[Rh(COD)OH]_2$	BINAP	DCE	<5

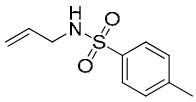
[a] Determined by crude ^{1}H NMR (400MHz). [b] Isolated yield. [c] Additional parameters were screened using the $[Rh(COD)OH]_2$/BINAP/dioxane system (NMR yield in bracket): 24 °C (36%), 6 h (26%), added Cs_2CO_3 (1.0 equiv) (8%), added $Sc(OTf)_3$ (0.1 equiv) (<5%); using potassium phenyltrifluoroborate and phenylboronic acid pinacol ester in dioxane/H_2O gave <10% yield of the desired products; using less than 2.5 equiv boronic acid led to incomplete conversion. [d] BIPHEP gave comparable yield, but the cost of racemic BINAP is much less than BIPHEP (Aldrich).

Characterization Data.

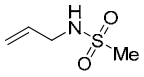
Substrates:



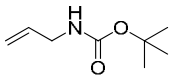
(1a)[3]: 2-allylisoindoline-1,3-dione. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89-7.83 (2H, m), 7.75-7.70 (2H, m), 5.95-5.84 (1H, m), 5.29-5.18 (2H, m), 4.31-4.29 (2H, m) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 167.9, 133.9, 132.1, 131.5, 123.2, 117.7, 40.0 ppm.



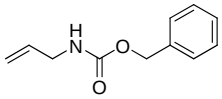
(1b)[3]: *N*-allyl-4-methylbenzenesulfonamide. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 (2H, d, J = 8.1 Hz), 7.31 (2H, d, J = 7.9 Hz), 5.77-5.67 (1H, m), 5.19-5.07 (2H, m), 4.45 (1H, br. s), 3.58 (2H, br. d, J = 5.7 Hz), 2.43 (3H, s) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 143.4, 136.9, 132.9, 129.7, 127.1, 117.6, 45.7, 21.5 ppm.



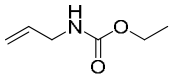
(1c)[4]: *N*-allylmethanesulfonamide. ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.93-5.83 (1H, m), 5.34-5.20 (2H, m), 4.86 (1H, br. s), 3.79-3.75 (2H, m), 2.98 (3H, s) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 133.4, 117.7, 45.5, 40.9 ppm.



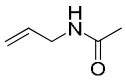
(1d)[5]: *tert*-butyl allylcarbamate. ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.89-5.79 (1H, m), 5.20-5.09 (2H, m), 4.61 (1H, br. s), 3.75 (2H, br. t, J = 5.6 Hz), 1.45 (9H, s) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 155.7, 134.9, 115.6, 79.3, 43.0, 28.3 ppm.



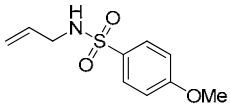
(1e)[5]: benzyl allylcarbamate. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36-7.29 (5H, m), 5.88-5.79 (1H, m), 5.21-5.10 (4H, m), 4.89 (1H, br. s), 3.81 (2H, br. t, J = 5.2 Hz) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 156.2, 136.5, 134.4, 128.5, 128.1, 116.0, 66.7, 43.4 ppm.



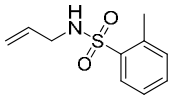
(1f)[6]: ethyl allylcarbamate. ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.90-5.80 (1H, m), 5.22-5.10 (2H, m), 4.86 (1H, br. s), 4.13 (2H, q, J = 7.0 Hz), 3.80 (2H, br. t, J = 5.6 Hz), 1.25 (3H, t, J = 7.0 Hz) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 156.5, 134.6, 115.8, 60.8, 43.3, 14.6 ppm.



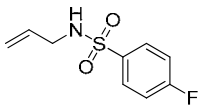
(1g)[7]: *N*-allylacetamide. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.43 (1H, br. s), 5.88-5.78 (1H, m), 5.22-5.10 (2H, m), 3.88-3.83 (2H, m), 2.00 (3H, s). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 170.1, 134.1, 116.0, 41.9, 22.9 ppm.



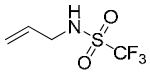
(1h)[8]: *N*-allyl-4-methoxybenzenesulfonamide. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81 (2H, d, J = 9.0 Hz), 6.98 (2H, d, J = 9.0 Hz), 5.77-5.67 (1H, m), 5.19-5.08 (2H, m), 4.67 (1H, br. t, J = 6.1 Hz), 3.87 (3H, s), 3.58 (2H, tt, J = 1.5, 6.1 Hz) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 162.9, 133.0, 131.6, 129.2, 117.6, 114.2, 55.6, 45.7 ppm.



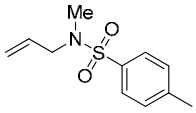
(1i)[8]: *N*-allyl-2-methylbenzenesulfonamide. Prepared according to the literature procedure. The product was obtained as a colourless oil in quantitative yield (405 mg). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.97 (1H, dd, J = 1.3, 8.2 Hz), 7.46 (1H, dt, J = 1.3, 7.5 Hz), 7.34-7.29 (2H, m), 5.71 (1H, tdd, J = 5.9, 10.2, 17.0 Hz), 5.17 (1H, ddd, J = 1.5, 2.8, 17.1 Hz), 5.09 (1H, ddd, J = 1.2, 2.5, 10.2 Hz), 4.75 (1H, br. s), 3.58 (2H, tt, J = 1.5, 6.1 Hz), 2.66 (3H, s) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 137.8, 137.0, 133.0, 132.8, 132.5, 129.5, 126.1, 117.7, 45.5, 20.3 ppm. **IR** (NaCl, neat film): 3302, 3063, 2986, 2932, 2863, 1651, 1597, 1427, 1165, 1134, 1072, 926, 764 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{10}H_{13}NO_2S$ [M^+]: 211.0667; found: 211.0665.



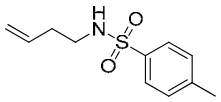
(1j)[8]: *N*-allyl-4-fluorobenzenesulfonamide. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93-7.88 (2H, m), 7.23-7.17 (2H, m), 5.76-5.66 (1H, m), 5.20-5.08 (2H, m), 4.91 (1H, br. t, J = 5.6 Hz), 3.61 (2H, tt, J = 1.5, 6.1 Hz) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 166.3, 163.8, 132.7, 129.9, 129.8, 117.8, 116.4, 116.2, 45.7 ppm.



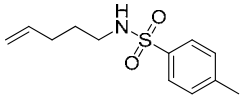
(1k)[8]: *N*-allyl-1,1,1-trifluoromethanesulfonamide. Prepared according to the literature procedure. The product was obtained as a colourless oil in 56% yield (382 mg). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 5.86 (1H, tdd, J = 5.9, 10.2, 17.0 Hz), 5.33 (1H, dtd, J = 0.9, 1.6, 17.1 Hz), 5.29 (1H, ddd, J = 1.3, 2.1, 10.2 Hz), 5.00 (1H, br. s), 3.9 (2H, d, J = 5.7 Hz) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 132.0, 119.8 (q, J = 320.9 Hz, CF_3), 118.9, 46.7 ppm. **IR** (NaCl, neat film): 3649, 3318, 2947, 1651, 1435, 1373, 1196, 1057, 934, 856 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_4H_6F_3NO_2S$ [M^+]: 189.0071; found: 189.0075.



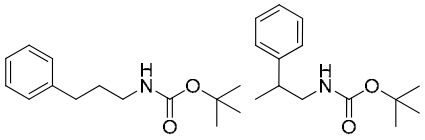
(1l)[9]: *N*-allyl-*N*-4-dimethylbenzenesulfonamide. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68 (2H, d, J = 8.3 Hz), 7.32 (2H, d, J = 7.9 Hz), 5.76-5.66 (1H, m), 5.22-5.16 (2H, m), 3.63 (2H, d, J = 6.3 Hz), 2.66 (3H, s), 2.44 (3H, s) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 143.3, 134.4, 132.6, 129.6, 127.4, 119.0, 53.0, 34.2, 21.5 ppm.



(4a)[3]: *N*-(but-3-enyl)-4-methylbenzenesulfonamide. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (2H, d, J = 8.3 Hz), 7.31 (2H, d, J = 8.3 Hz), 5.68-5.58 (1H, m), 5.07-5.00 (2H, m), 4.70 (1H, br. s), 3.01 (2H, q, J = 6.7 Hz), 2.43 (3H, s), 2.20 (2H, d, J = 6.8 Hz) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 143.4, 136.9, 134.1, 129.7, 127.1, 118.0, 42.1, 33.6, 21.5 ppm.

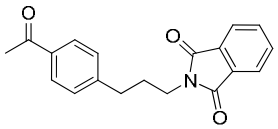


(4b)[11]: 4-methyl-*N*-(pent-4-enyl)benzenesulfonamide. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 (2H, d, J = 8.3 Hz), 7.31 (2H, d, J = 8.0 Hz), 5.75-5.65 (1H, m), 4.99-4.92 (2H, m), 4.79 (1H, br. t, J = 6.1 Hz), 2.94 (2H, q, J = 6.9 Hz), 2.43 (3H, s), 2.04 (2H, q, J = 7.0 Hz), 1.59-1.52 (2H, m) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 143.4, 137.3, 137.1, 129.7, 127.1, 115.5, 42.7, 30.7, 28.7, 21.5 ppm.

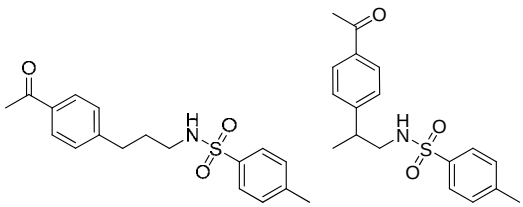


(2-Boc, 3-Boc): *tert*-butyl 3-phenylpropylcarbamate (linear).

***tert*-butyl 2-phenylpropylcarbamate (branched).** Prepared according to the general procedure described above. The product was obtained as a colourless oil in 70% yield (77 mg). Inseparable mixture of linear and branched regioisomers (1:5 ratio). **^{1}H NMR** (400 MHz, $CDCl_3$): major regioisomer (branched) δ 7.35-7.27 (2H, m), 7.25-7.16 (3H, m), 4.44 (1H, br. s), 3.46-3.36 (1H, m), 3.23-3.14 (1H, m), 2.98-2.85 (1H, m), 1.41 (9H, s), 1.26 (3H, d, J = 7.0 Hz) ppm; characteristic peaks for minor regioisomer (linear) δ 4.54 (1H, br. s), 2.64 (2H, t, J = 7.8 Hz), 1.86-1.77 (2H, m), 1.45 (9H, s) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 155.9, 144.2, 141.5, 128.6, 128.4, 128.3, 127.2, 126.5, 125.9, 79.1, 47.3, 40.1, 33.1, 31.7, 28.4, 28.3, 19.1 ppm. **IR** (NaCl, neat film): 3341, 2978, 2955, 1697, 1512, 1366, 1173, 756, 702 cm^{-1}. **HRMS** *m/z* (ESI): calcd. for $C_{14}H_{21}NO_2Na$ [M-Na$^+$]: 258.1464; found: 258.1466.



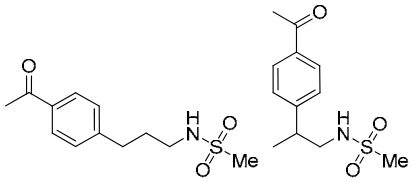
(2aa): 2-(3-(4-acetylphenyl)propyl)isoindoline-1,3-dione. Prepared according to the general procedure described above. The product was obtained as a white solid in 91% yield (90 mg). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.85-7.80 (4H, m), 7.70 (2H, dd, J = 3.1, 5.4 Hz), 7.29 (2H, d, J = 8.1 Hz), 3.75 (2H, t, J = 7.1 Hz), 2.75 (2H, t, J = 7.7 Hz), 2.55 (3H, s), 2.11-2.03 (2H, m) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 197.6, 168.3, 146.7, 135.1, 133.9, 132.0, 128.5 (2), 123.1, 37.6, 33.2, 29.3, 26.5 ppm. **IR** (NaCl, neat film): 2932, 1775, 1713, 1682, 1605, 1397, 1358, 1265, 1018 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{19}H_{17}NO_3$ [M$^+$]: 307.1208; found: 307.1201. **M.p.** 80-82 ^{o}C.



(2ba): *N*-(3-(4-acetylphenyl)propyl)-4-methylbenzenesulfonamide (linear).

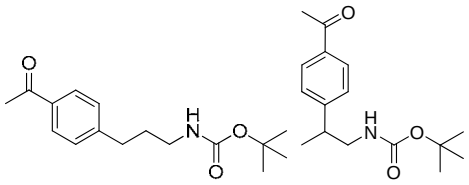
***N*-(2-(4-acetylphenyl)propyl)-4-methylbenzenesulfonamide (branched).** Prepared according to the general procedure described above. The product was obtained as a colourless oil in 92% yield (98 mg). Inseparable mixture of linear and branched regioisomers (6:1 ratio). **^{1}H NMR** (400 MHz, $CDCl_3$): major regioisomer (linear) δ 7.84 (2H, d, J = 8.4 Hz), 7.74 (2H, d, J = 8.3 Hz), 7.30 (2H, d, J = 7.9 Hz), 7.18 (2H,

d, J = 8.4 Hz), 4.94 (1H, br. t, J = 5.8 Hz), 2.96 (2H, q, J = 6.7 Hz), 2.67 (2H, t, J = 7.7 Hz), 2.57 (3H, s), 2.43 (3H, s), 1.86-1.75 (2H, m) ppm; characteristic peaks for minor regioisomer (branched) δ 7.67 (2H, d, J = 8.3 Hz), 3.23-3.14 (1H, m), 3.09-3.02 (1H, m), 1.25 (3H, d, J = 6.9 Hz) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 197.8, 146.7, 143.4, 136.8, 135.2, 129.7, 128.8, 128.6, 128.5, 127.4, 127.0, 126.4, 49.3, 42.4, 39.9, 32.6, 30.8, 26.5, 21.5, 18.9 ppm. **IR** (NaCl, neat film): 3271, 2955, 2893, 1674, 1604, 1435, 1327, 1273, 1157, 1096, 810 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{18}H_{21}NO_3S$ [M^+]: 331.1242; found: 331.1238.



(2ca): N-(3-(4-acetylphenyl)propyl)methanesulfonamide (linear).

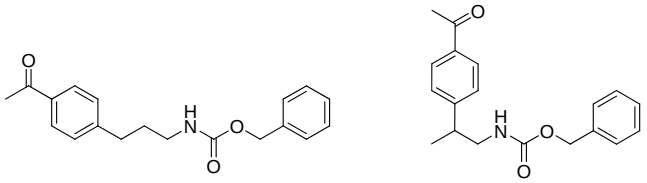
N-(2-(4-acetylphenyl)propyl)methanesulfonamide (branched). Prepared according to the general procedure described above. The product was obtained as a white solid in 83% yield (69 mg). Inseparable mixture of linear and branched regioisomers (5:1 ratio). **^{1}H NMR** (400 MHz, $CDCl_3$): major regioisomer (linear) δ 7.89 (2H, d, J = 8.2 Hz), 7.28 (2H, d, J = 8.5 Hz), 4.59 (1H, br. s), 3.16 (2H, d, J = 6.7 Hz), 2.95 (3H, s), 2.76 (2H, t, J = 7.7 Hz), 2.59 (3H, s), 1.97-1.89 (2H, m) ppm; minor regioisomer (branched) δ 7.93 (2H, d, J = 8.2 Hz), 7.33 (2H, d, J = 8.4 Hz), 4.38 (1H, br. s), 3.41-3.24 (3H, m), 2.83 (3H, s), 2.59 (3H, s), 1.34 (3H, d, J = 7.0 Hz) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): linear regioisomer δ 197.8, 146.6, 135.3, 128.7, 128.6, 42.6, 40.2, 32.6, 31.3, 26.5 ppm. **IR** (NaCl, neat film): 3241, 2955, 1674, 1435, 1304, 1150,833, 764 cm^{-1}. **HRMS** *m/z* (ESI): calcd. for $C_{12}H_{18}NO_3S$ [MH^+]: 256.1001; found: 256.1000. **M.p.** 113-115 ^{o}C.



(2da): *tert*-butyl 3-(4-acetylphenyl)propylcarbamate (linear).

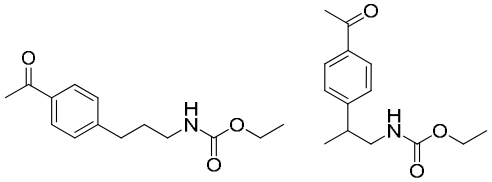
***tert*-butyl 2-(4-acetylphenyl)propylcarbamate (branched).** Prepared according to the general procedure described above. The product was obtained as a colourless oil in 92% yield (120 mg). Inseparable mixture of linear and branched regioisomers (2:1 ratio). **^{1}H NMR** (400 MHz, $CDCl_3$): major regioisomer (linear) δ 7.88 (2H, d, J = 8.2 Hz), 7.27 (2H, d, J = 8.1 Hz), 4.69 (1H, br. s), 3.16 (2H, br. q, J = 7.1 Hz), 2.70 (2H, t, J = 7.8 Hz), 2.58 (3H, s), 1.88-1.79 (2H, m), 1.44 (9H, s) ppm; minor regioisomer (branched) δ 7.91 (2H, d, J = 8.3 Hz), 7.30 (2H, d, J = 8.3 Hz), 4.56 (1H, br. s), 3.44-3.35 (1H, m), 3.26-3.20 (1H, m), 3.07-2.97 (1H, m), 2.59 (3H, s), 1.41 (9H, s), 1.28 (3H, d, J = 7.0 Hz) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 197.7, 155.9, 155.8, 150.0, 147.4, 135.6, 135.1, 128.9, 128.6, 128.5, 128.2, 127.4, 127.3, 115.3, 79.2, 79.1, 47.0, 40.1,

40.0, 33.0, 31.3, 28.3, 26.5, 26.4, 18.7 ppm. **IR** (NaCl, neat film): 3341, 2955, 2893, 1682, 1605, 1528, 1366, 1273, 1173, 756 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{12}H_{15}NO_3$ [M minus $C_4H_8^+$]: 221.1052; found: 221.1057.



(2ea): benzyl 3-(4-acetylphenyl)propylcarbamate (linear).

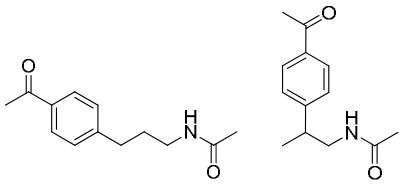
benzyl 2-(4-acetylphenyl)propylcarbamate (branched). Prepared according to the general procedure described above. The product was obtained as a colourless oil in 86% yield (86 mg). Inseparable mixture of linear and branched regioisomers (2.5:1 ratio). **^{1}H NMR** (400 MHz, $CDCl_3$): major regioisomer (linear) δ 7.90-7.84 (2H, m, overlapping with minor regioisomer), 7.37-7.23 (7H, m, overlapping with minor regioisomer), 5.09 (2H, s), 4.94 (1H, br. s), 3.23 (2H, br. q, J = 6.6 Hz), 2.69 (2H, t, J = 7.7 Hz), 2.56 (3H, s), 1.91-1.81 (2H, m) ppm; minor regioisomer (branched) δ 7.90-7.84 (2H, m, overlapping with major regioisomer), 7.37-7.23 (7H, m, overlapping with major regioisomer), 5.06 (2H, s), 4.79 (1H, br. s), 3.51-3.43 (1H, m), 3.33-3.26 (1H, m), 3.06-2.99 (1H, m), 2.57 (3H, s), 1.28 (3H, d, J = 7.0 Hz) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 197.7 (2), 156.4, 156.2, 149.6, 147.2, 136.5, 136.4, 135.7, 135.1, 128.7, 128.5 (2), 128.4, 128.0, 127.4, 66.6, 47.4, 40.5, 40.1, 32.9, 31.2, 26.5, 18.8 ppm. **IR** (NaCl, neat film): 3341, 3032, 2940, 1682, 1604, 1358, 1265, 1134, 1011, 841,756 cm^{-1}. **HRMS** *m/z* (ESI): calcd. for $C_{19}H_{21}NO_3$ [M^+]: 311.1521; found: 311.1519.



(2fa): ethyl 3-(4-acetylphenyl)propylcarbamate (linear).

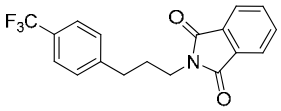
ethyl 2-(4-acetylphenyl)propylcarbamate (branched). Prepared according to the general procedure described above. The product was obtained as a colourless oil in 65% yield (54 mg). Inseparable mixture of linear and branched regioisomers (2:1 ratio). **^{1}H NMR** (400 MHz, $CDCl_3$): major regioisomer (linear) δ 7.88 (2H, d, J = 8.3 Hz), 7.27 (2H, d, J = 8.5 Hz), 4.81 (1H, br. s), 4.15-4.04 (2H, m, overlapping with minor regioisomer), 3.21 (2H, br. q, J = 6.5 Hz), 2.71 (2H, t, J = 7.8 Hz), 2.58 (3H, s), 1.90-1.81 (2H, m), 1.24 (3H, t, J = 7.1 Hz) ppm; minor regioisomer (branched) δ 7.92 (2H, d, J = 8.4 Hz), 7.31 (2H, d, J = 8.3 Hz), 4.67 (1H, br. s), 4.15-4.04 (2H, m, overlapping with major regioisomer), 3.50-3.40 (1H, m), 3.33-3.25 (1H, m), 3.08-2.97 (1H,m), 2.59 (3H, s), 1.29 (3H, d, J = 7.0 Hz), 1.23-1.17 (3H, m, overlapping with major regioisomer) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 197.7 (2), 156.7, 156.5, 149.8, 147.2, 135.7,

135.1, 128.7, 128.5 (2), 127.4, 60.7, 47.3, 40.4, 40.2, 33.0, 31.3, 26.5, 18.8, 14.6, 14.5 ppm. **IR** (NaCl, neat film): 3341, 2955, 1682, 1605, 1528, 1366, 1265, 1150, 833 cm^{-1}. **HRMS** *m/z* (ESI): calcd. for $C_{14}H_{20}NO_3$ $[MH^+]$: 250.1437; found: 250.1438.

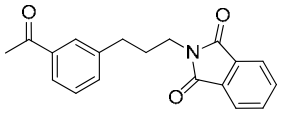


(2ga): *N*-(3-(4-acetylphenyl)propyl)acetamide (linear).

***N*-(2-(4-acetylphenyl)propyl)acetamide (branched).** Prepared according to the general procedure described above. The product was obtained as a colourless oil in 50% yield (36 mg). Linear regioisomer: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.88 (2H, d, J = 8.3 Hz), 7.27 (2H, d, J = 8.2 Hz), 5.73 (1H, br. s), 3.29 (2H, q, J = 7.0 Hz), 2.71 (2H, t, J = 7.7 Hz), 2.58 (3H, s), 1.96 (3H, s), 1.91-1.82 (2H, m) ppm. Branched regioisomer: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.92 (2H, d, J = 8.2 Hz), 7.31 (2H, d, J = 8.2 Hz), 5.45 (1H, br. s), 3.65-3.57 (1H, m), 3.32-3.24 (1H, m), 3.09-2.99 (1H, m), 2.59 (3H, s), 1.90 (3H, s), 1.29 (3H, d, J = 7.0 Hz) ppm. Linear regioisomer: **^{13}C NMR** (100 MHz, $CDCl_3$): δ 197.8, 170.2, 147.2, 135.2, 128.6, 128.5, 39.1, 33.2, 30.9, 26.5, 23.2 ppm. Branched regioisomer: **^{13}C NMR** (100 MHz, $CDCl_3$): δ 197.7, 170.0, 149.8, 135.8, 128.8, 127.4, 45.9, 39.8, 26.5, 23.2, 19.1 ppm. **IR** (NaCl, neat film): 3295, 2955, 1682, 1605, 1559, 1435, 1366, 1273, 833 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{13}H_{17}NO_2$ $[M^+]$: 219.1259; found: 219.1263.

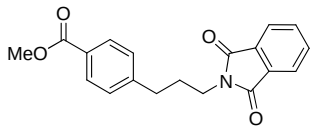


(2ab): 2-(3-(4-(trifluoromethyl)phenyl)propyl)isoindoline-1,3-dione. Prepared according to the general procedure described above. The product was obtained as a white solid in 76% yield (77 mg). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.81 (2H, dd, J = 3.1, 5.4 Hz), 7.70 (2H, dd, J = 3.0, 5.5 Hz), 7.47 (2H, d, J = 8.1 Hz), 7.30 (2H, d, J = 8.0 Hz), 3.75 (2H, t, J = 7.0 Hz), 2.75 (2H, t, J = 7.7 Hz), 2.12-2.03 (2H, m) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.3, 145.1, 133.9, 132.0, 128.6, 125.2 (q, J = 3.8 Hz, CF_3), 123.1, 37.6, 33.0, 29.3 ppm. **IR** (NaCl, neat film): 2940, 1775, 1713, 1620, 1397, 1327, 1165, 1119, 1065, 1018, 718 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{18}H_{14}F_3NO_2$ $[M^+]$: 333.0977; found: 333.0964. **M.p.** 60-61 ^oC.

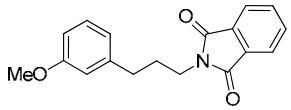


(2ac): 2-(3-(3-acetylphenyl)propyl)isoindoline-1,3-dione. Prepared according to the general procedure described above. The product was obtained as a colourless oil in 66% yield (65 mg). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.81-7.77 (3H, m), 7.72-7.67 (3H, m), 7.40-7.30 (2H, m), 3.74 (2H, t, J = 7.1 Hz), 2.73 (2H, t, J

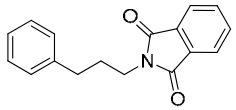
= 7.8 Hz), 2.57 (3H, s). 2.09-2.01 (2H, m) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 198.1, 168.3, 141.5, 137.2, 133.9, 133.1, 132.0, 128.6, 128.1, 126.0, 123.1, 37.6, 32.9, 29.6, 26.6 ppm. **IR** (NaCl, neat film): 2940, 1771, 1701, 1678, 1586, 1439, 1397, 1358, 1273, 1188, 1026, 721 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{19}H_{17}NO_3$ [M^+]: 307.1208; found: 307.1214.



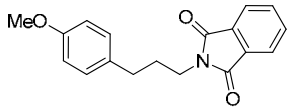
(2ad): methyl 4-(3-(1,3-dioxoisoindolin-2-yl)propyl)benzoate. Prepared according to the general procedure described above. The product was obtained as a white solid in 67% yield (69 mg). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.91 (2H, d, J = 8.3 Hz), 7.82 (2H, dd, J = 3.0, 5.5 Hz), 7.70 (2H, dd, J = 3.1, 5.4 Hz), 7.26 (2H, d, J = 8.2 Hz), 3.88 (3H, s), 3.75 (2H, t, J = 7.1 Hz), 2.74 (2H, t, J = 7.8 Hz), 2.10-2.02 (2H, m) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.3, 167.0, 146.5, 133.9, 132.0, 129.7, 128.3, 127.9, 123.1, 51.9, 37.6, 33.2, 29.4 ppm. **IR** (NaCl, neat film): 2939, 1775, 1713, 1613, 1397, 1281, 1111, 718 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{19}H_{17}NO_4$ [M^+]: 323.1158; found: 323.1156. **M.p.** 90-92 ^{o}C.



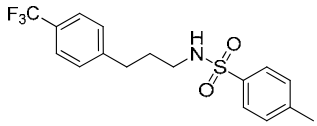
(2ae): 2-(3-(3-methoxyphenyl)propyl)isoindoline-1,3-dione. Prepared according to the general procedure described above. The product was obtained as a colourless oil in 55% yield (53 mg). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.82 (2H, dd, J = 3.1, 5.4 Hz), 7.69 (2H, dd, J = 3.0, 5.5 Hz), 7.15 (1H, t, J = 7.9 Hz), 6.78 (1H, d, J = 7.6 Hz), 6.75-6.73 (1H, m), 6.68-6.65 (1H, m), 3.77 (3H, s), 3.74 (2H, t, J = 7.2 Hz), 2.66 (2H, t, J = 7.8 Hz), 2.08-1.99 (2H, m) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.3, 159.5, 142.6, 133.8, 132.1, 129.3, 123.1, 120.6, 113.8, 111.4, 55.1, 37.7, 33.2, 29.6 ppm. **IR** (NaCl, neat film): 3024, 2940, 1775, 1713, 1605, 1397, 1265, 1157, 1026, 887, 756 cm^{-1}. **HRMS** *m/z* (ESI): calcd. for $C_{18}H_{18}NO_3$ [MH^+]: 296.1281; found: 296.1290.



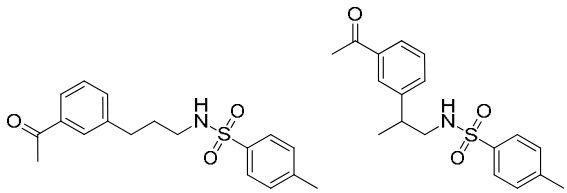
(2af): 2-(3-phenylpropyl)isoindoline-1,3-dione. Prepared according to the general procedure described above. The product was obtained as a white solid in 65% yield (55 mg). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.83-7.80 (2H, m), 7.70-7.67 (2H, m), 7.27-7.17 (4H, m), 7.16-7.10 (1H, m), 3.74 (2H, t, J = 7.2 Hz), 2.68 (2H, t, J = 7.8 Hz), 2.08-1.99 (2H, m) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.3, 140.9, 133.8, 132.1, 128.3, 128.2, 125.9, 123.1, 37.7, 33.1, 29.8 ppm. **IR** (NaCl, neat film): 3024, 2940, 1775, 1713, 1397, 1366, 1018 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{17}H_{15}NO_2$ [M^+]: 265.1103; found: 265.1111. **M.p.** 42-45 ^{o}C.



(2ag): 2-(3-(4-methoxyphenyl)propyl)isoindoline-1,3-dione. Prepared according to the general procedure described above. The product was obtained as a white solid in 35% yield (34 mg). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.82 (2H, dd, J = 3.0, 5.5 Hz), 7.70 (2H, dd, J = 3.1, 5.4 Hz), 7.11 (2H, d, J = 8.8 Hz), 6.79 (2H, d, J = 8.7 Hz), 3.75 (3H, s), 3.73 (2H, t, J = 7.2 Hz), 2.63 (2H, t, J = 7.8 Hz), 2.04-1.96 (2H, m) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.4, 157.8, 133.8, 133.0, 132.1, 129.2, 123.1, 113.8, 55.2, 37.8, 32.2, 30.0 ppm. **IR** (NaCl, neat film): 2940, 1775, 1713, 1613, 1512, 1397, 1242, 1026, 718 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{18}H_{18}NO_3$ [MH^+]: 296.1281; found: 296.1287. **M.p.** 88-90 ^{o}C.

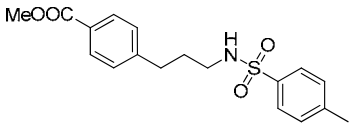


(2bb): 4-methyl-*N*-(3-(4-(trifluoromethyl)phenyl)propyl)benzenesulfonamide. Prepared according to the general procedure described above. The product was obtained as a white solid in 79% yield (91 mg). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.73 (2H, d, J = 8.3 Hz), 7.50 (2H, d, J = 8.1 Hz), 7.30 (2H, d, J = 8.1 Hz), 7.20 (2H, d, J = 8.0 Hz), 4.51 (1H, br. t, J = 6.2 Hz), 2.96 (2H, q, J = 6.7 Hz), 2.68 (2H, t, J = 7.6 Hz), 2.43 (3H, s), 1.85-1.77 (2H, m) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 145.0, 143.5, 136.8, 129.7, 128.7, 127.1, 125.3 (q, J = 3.8 Hz, CF_3), 42.3, 32.4, 30.9, 21.5 ppm. **IR** (NaCl, neat film): 3248, 2931, 1620, 1435, 1420, 1327, 1157, 1065, 818, 756 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{17}H_{18}F_3NO_2S$ [M^+]: 357.1010; found: 357.1010. **M.p.**: 97-99 ^{o}C

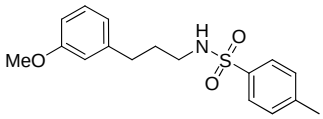


(2bc): *N*-(3-(3-acetylphenyl)propyl)-4-methylbenzenesulfonamide **(linear).**

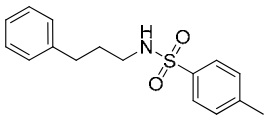
N-(2-(3-acetylphenyl)propyl)-4-methylbenzenesulfonamide **(branched).** Prepared according to the general procedure described above. The product was obtained as a pale yellow oil in 79% yield (84 mg). Inseparable mixture of linear and branched regioisomers (7.7:1 ratio). **^{1}H NMR** (400 MHz, $CDCl_3$): major regioisomer (linear) δ 7.77-7.71 (4H, m), 7.31-7.28 (4H, m), 4.81 (1H, br. t, J = 6.2 Hz), 2.96 (2H, q, J = 6.7 Hz), 2.67 (2H, t, J = 7.6 Hz), 2.58 (3H, s), 2.42 (3H, s), 1.84-1.77 (2H, m) ppm; characteristic peaks for minor regioisomer (branched) δ 4.56 (1H, br. t, J = 6.2 Hz), 3.22-3.15 (1H, m), 3.09-3.02 (1H, m), 2.56 (3H, s), 1.24 (3H, d, J = 6.9 Hz) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): major regioisomer (linear) δ 198.4, 143.3, 141.5, 137.2, 136.8, 133.2, 129.6, 128.6, 128.1, 127.0, 126.1, 42.4, 32.4, 31.0, 26.6, 21.4 ppm. **IR** (NaCl, neat film): 3271, 3063, 3028, 2928, 2866, 1678, 1585, 1431, 1327, 1157, 1096, 961, 899, 814, 667 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{18}H_{21}NO_3S$ [M^+]: 331.1242; found: 331.1247.



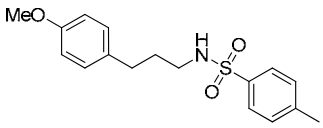
(2bd): methyl 4-(3-(4-methylphenylsulfonamido)propyl)benzoate. Prepared according to the general procedure described above. The product was obtained as a white solid in 56% yield (63 mg). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.90 (2H, d, J = 8.3 Hz), 7.74 (2H, d, J = 8.3 Hz), 7.29 (2H, d, J = 8.0 Hz), 7.13 (2H, d, J = 8.3 Hz), 4.94 (1H, br. t, J = 5.8 Hz), 3.89 (3H, s), 2.94 (2H, q, J = 6.7 Hz), 2.66 (2H, t, J = 7.7 Hz), 2.42 (3H, s), 1.83-1.75 (2H, m) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 167.0, 146.4, 143.4, 136.8, 129.7(2), 128.3, 128.0, 127.0, 52.0, 42.4, 32.6, 30.8, 21.5 ppm. **IR** (NaCl, neat film): 3271, 2955, 1721, 1605, 1435, 1327, 1281, 1157, 1072, 764 cm^{-1}. **HRMS** *m/z* (ESI): calcd. for $C_{18}H_{22}NO_4S$ [MH^+]: 348.1264; found: 348.1259. **M.p.** 95-97 ^{o}C.



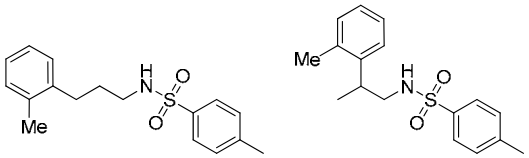
(2be): *N*-(3-(3-methoxyphenyl)propyl)-4-methylbenzenesulfonamide. Prepared according to the general procedure described above. The product was obtained as a colourless oil in 62% yield (63 mg). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.79 (2H, d, J = 8.3 Hz), 7.32 (2H, d, J = 8.0 Hz), 7.20-7.17 (1H, m), 6.76-6.68 (3H, m), 5.09 (1H, br. t, J = 6.1 Hz), 3.80 (3H, s), 2.98 (2H, q, J = 6.7 Hz), 2.60 (2H, t, J = 7.6 Hz), 2.45 (3H, s), 1.84-1.76 (2H, m) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 159.6, 143.2, 142.5, 136.8, 129.6, 129.3, 127.0, 120.6, 114.0, 111.3, 55.0, 42.5, 32.6, 30.9, 21.4 ppm. **IR** (NaCl, neat film): 3279, 2940, 2866, 2835, 1586, 1489, 1435, 1323, 1261, 1157, 1096, 1042, 814, 664 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{17}H_{21}NO_3S$ [M^+]: 319.1242; found: 319.1245.



(2bf): 4-methyl-*N*-(3-phenylpropyl)benzenesulfonamide. Prepared according to the general procedure described above. The product was obtained as a colourless oil in 65% yield (89 mg). **^{1}H NMR** (300 MHz, $CDCl_3$): δ 7.73 (2H, d, J = 8.3 Hz), 7.29 (2H, d, J = 7.9 Hz), 7.27-7.21 (2H, m), 7.19-7.14 (1H, m), 7.09-7.05 (2H, m), 4.63 (1H, br. s), 2.96 (2H, q, J = 6.9 Hz), 2.59 (2H, t, J = 7.6 Hz), 2.42 (3H, s), 1.82-1.74 (2H, m) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 143.2, 140.9, 136.9, 129.6, 128.3, 128.2, 127.0, 125.9, 42.5, 32.6, 31.0, 21.4 ppm. **IR** (NaCl, neat film): 3271, 2955, 2893, 1604, 1435, 1327, 1149, 1072, 810 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{16}H_{19}NO_2S$ [M^+]: 289.1137; found: 289.1144.

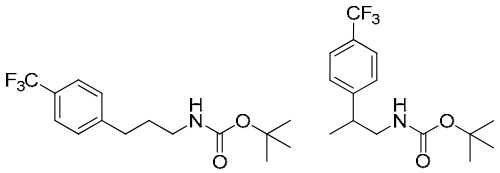


(2bg): *N*-(3-(4-methoxyphenyl)propyl)-4-methylbenzenesulfonamide. Prepared according to the general procedure described above. The product was obtained as a pale yellow oil in 36% yield (35 mg). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.73 (2H, d, J = 8.3 Hz), 7.30 (2H, d, J = 8.0 Hz), 6.98 (2H, d, J = 8.6 Hz), 6.78 (2H, d, J = 8.7 Hz), 4.65 (1H, br. t, J = 5.6 Hz), 3.77 (3H, s), 2.94 (2H, q, J = 6.8 Hz), 2.53 (2H, t, J = 7.6 Hz), 2.43 (3H, s), 1.78-1.70 (2H, m) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 157.9, 143.3, 136.9, 132.9, 129.7, 129.2, 127.1, 113.8, 55.2, 42.5, 31.7, 31.3, 21.5 ppm. **IR** (NaCl, neat film): 3287, 2955, 2361, 1605, 1512, 1327, 1250, 1157, 1096, 1034, 810 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{17}H_{21}NO_3S$ [M^+]: 319.1242; found: 319.1249



(2bh): 4-methyl-N-(3-o-tolylpropyl)benzenesulfonamide (linear).

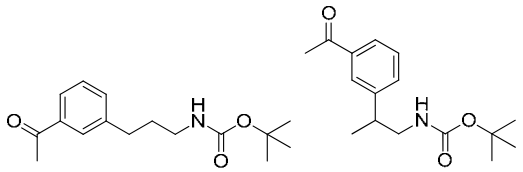
4-methyl-N-(2-p-tolylpropyl)benzenesulfonamide (branched). Prepared according to the general procedure described above. The product was obtained as a colourless oil in 65% yield (63 mg). Branched regioisomer: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.67 (2H, d, J = 8.3 Hz), 7.29 (2H, d, J = 8.1 Hz), 7.16-7.08 (3H, m), 7.00-6.96 (1H, m), 4.27 (1H, br. s), 3.24-3.12 (2H, m), 3.08-3.00 (1H, m), 2.43 (3H, s), 2.26 (3H, s), 1.18 (3H, d, J = 6.7 Hz) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 143.4, 140.9, 136.9, 136.2, 130.8, 129.7, 127.0, 126.6, 126.5, 124.9, 48.8, 34.6, 21.5, 19.4, 19.0 ppm. **IR** (NaCl, neat film): 3287, 2955, 2924, 1605, 1435, 1327, 1157, 1072, 818, 764 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{17}H_{21}NO_2S$ [M^+]: 303.1293; found: 303.1293. Linear regioisomer: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.74 (2H, d, J = 8.3 Hz), 7.30 (2H, d, J = 7.9 Hz), 7.14-7.06 (3H, m), 7.02-6.99 (1H, m), 4.43 (1H, br. t, J = 5.6 Hz), 3.02 (2H, q, J = 6.8 Hz), 2.58 (2H, t, J = 7.8 Hz), 2.43 (3H, s), 2.23 (3H, s), 1.77-1.69 (2H, m) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 143.4, 139.1, 136.9, 135.8, 130.2, 129.7, 128.7, 127.1, 126.2, 126.0, 43.0, 30.2, 29.8, 21.5, 19.2 ppm. **IR** (NaCl, neat film): 3271, 2955, 1605, 1435, 1327, 1157, 1096, 810, 764 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{17}H_{21}NO_2S$ [M^+]: 303.1293; found: 303.1299



(2db): *tert*-butyl 3-(4-(trifluoromethyl)phenyl)propylcarbamate (linear).

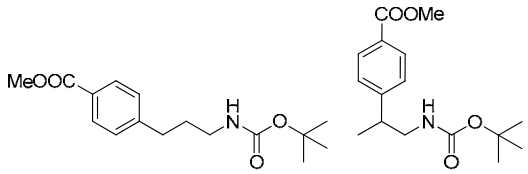
***tert*-butyl 2-(4-(trifluoromethyl)phenyl)propylcarbamate (branched).** Prepared according to the general procedure described above. The product was obtained as a white solid in 69% yield (67 mg). Branched

regioisomer: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.57 (2H, d, J = 8.3 Hz), 7.32 (2H, d, J = 8.1 Hz), 4.44 (1H, br. s), 3.44-3.34 (1H, m), 3.25-3.17 (1H, m), 3.06-2.95 (1H, m), 1.40 (9H, s), 1.28 (3H, d, J = 7.0 Hz) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 155.8, 148.4, 127.6, 125.5 (q, J = 3.7 Hz, CF_3), 122.9, 79.4, 47.2, 40.2, 28.3, 18.9 ppm. **IR** (NaCl, neat film): 3341, 2978, 2932, 1697, 1512, 1327, 1250, 1165, 1126, 1072, 833 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{11}H_{12}NO_2F_3$ [M minus $C_4H_8^+$]: 247.0820; found: 247.0815. **M.p.** 100-103 ^{o}C. Linear regioisomer: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.53 (2H, d, J = 8.2 Hz), 7.29 (2H, d, J = 8.0 Hz), 4.57 (1H, br. s), 3.16 (2H, br. q, J = 6.3 Hz), 2.70 (2H, t, J = 7.8 Hz), 1.87-1.78 (2H, m), 1.44 (9H, s) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 155.9, 145.7, 128.6, 125.5 (q, J = 3.9 Hz, CF_3), 122.9, 79.2, 40.0, 32.9, 31.5, 28.4 ppm. **IR** (NaCl, neat film): 3341, 2955, 2893, 1705, 1528, 1327, 1165, 1126, 1072, 764 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{11}H_{12}NO_2F_3$ [M minus $C_4H_8^+$]: 247.0820; found: 247.0820. **M.p.** 55-57 ^{o}C.



(2dc): *tert*-butyl (3-(3-acetylphenyl)propyl)carbamate (linear).

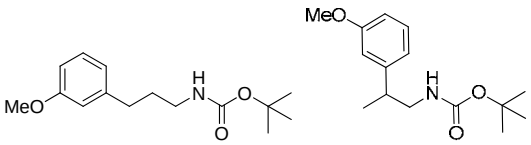
***tert*-butyl (2-(3-acetylphenyl)propyl)carbamate (branched).** Prepared according to the general procedure described above. The product was obtained as a colourless oil in 68% yield (60 mg). Inseparable mixture of linear and branched regioisomers (1.3:1 ratio). **^{1}H NMR** (400 MHz, $CDCl_3$): linear regioisomer δ 7.81-7.74 (2H, m, overlapping with branched regioisomer), 7.41-7.35 (2H, m, overlapping with branched regioisomer), 4.50 (1H, br. s), 3.17-3.11 (2H, m), 2.68 (2H, t, J = 7.7 Hz), 2.57 (3H, s), 1.85-1.78 (2H, m), 1.42 (9H, s); branched regioisomer δ 7.81-7.74 (2H, m, overlapping with linear regioisomer), 7.41-7.35 (2H, m, overlapping with linear regioisomer), 4.63 (1H, br. s), 3.41-3.34 (1H, m), 3.24-3.18 (1H, m), 3.03-2.95 (1H, m), 2.58 (3H, s), 1.38 (9H, s), 1.27 (3H, d, J = 7.0 Hz) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 198.2, 198.1, 155.9, 155.8, 144.8, 142.0, 137.3, 137.2, 133.1, 132.0, 128.7, 128.5, 128.0, 126.9, 126.7, 126.1, 79.4, 79.1, 47.1, 40.0, 32.8, 31.6, 28.3, 28.2, 28.1, 26.6, 18.9 ppm. **IR** (NaCl, neat film): 3356, 2974, 2932, 2870, 1717, 1678, 1601, 1520, 1439, 1366, 1273, 1250, 1173, 799, 694 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{16}H_{23}NO_3$ [M^+]: 277.1678; found: 277.1676.



(2dd): methyl 4-(3-(*tert*-butoxycarbonylamino)propyl)benzoate (linear).

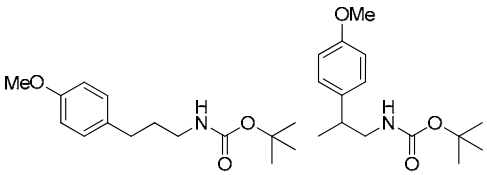
methyl 4-(1-(*tert*-butoxycarbonylamino)propan-2-yl)benzoate (branched). Prepared according to the general procedure described above. The product was obtained as a white solid (branched product) and a colourless oil (linear product) in 50% yield (45 mg). Branched regioisomer: **^{1}H NMR** (400 MHz, $CDCl_3$): δ

7.99 (2H, d, J = 8.4 Hz), 7.28 (2H, d, J = 8.7 Hz), 4.43 (1H, br. s), 3.91 (3H, s), 3.45-3.35 (1H, m), 3.25-3.16 (1H, m), 3.06-2.95 (1H, m), 1.41 (9H, s), 1.28 (3H, d, J = 7.0 Hz) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 167.0, 155.8, 149.7, 129.9, 128.6, 127.3, 79.3, 52.0, 47.2, 40.2, 28.3, 18.8 ppm. **IR** (NaCl, neat film): 3379, 2955, 1721, 1528, 1281, 1150, 1111, 764 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{15}H_{20}NO_3$ [M minus CH_3O^+]: 262.1443; found: 262.1444. **M.p.** 72-74 ^{o}C. Linear regioisomer: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.95 (2H, d, J = 8.3 Hz), 7.24 (2H, d, J = 8.5 Hz), 4.57 (1H, br. s), 3.90 (3H, s), 3.16 (2H, br. q, J = 6.3 Hz), 2.69 (2H, t, J = 7.8 Hz), 1.87-1.78 (2H, m), 1.44 (9H, s) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 167.0, 155.9, 147.1, 129.8, 128.4, 128.0, 79.2, 52.0, 40.1, 33.1, 31.4, 28.4 ppm. **IR** (NaCl, neat film): 3379, 2955, 1713, 1528, 1435, 1281, 1173, 1111, 764 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{12}H_{15}NO_4$ [M minus $C_4H_8^+$]: 237.1001; found: 237.0999.



(2de): *tert*-butyl (3-(3-methoxyphenyl)propyl)carbamate (linear).

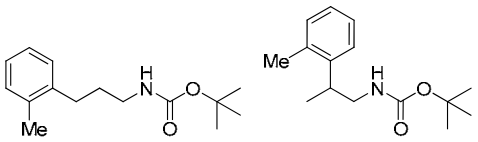
***tert*-butyl (2-(3-methoxyphenyl)propyl)carbamate (branched).** Prepared according to the general procedure described above. The product was obtained as a colourless oil in 71% yield (60 mg). Inseparable mixture of linear and branched regioisomers (1.3:1 ratio). **^{1}H NMR** (400 MHz, $CDCl_3$): linear regioisomer δ 7.29-7.20 (1H, m, overlapping with branched regioisomer), 6.84-6.75 (3H, m, overlapping with branched regioisomer), 4.61 (1H, br. s), 3.82 (3H, s), 3.23-3.15 (2H, m, overlapping with branched regioisomer), 2.65 (2H, t, J = 7.8 Hz), 1.87-1.80 (2H, m), 1.47 (9H, s) ppm; branched regioisomer δ 7.29-7.20 (1H, m, overlapping with linear regioisomer), 6.84-6.75 (3H, m, overlapping with linear regioisomer), 4.50 (1H, br. s), 3.83 (s, 3H), 3.45-3.40 (m, 1H), 3.23-3.15 (1H, m, overlapping with linear regioisomer), 2.96-2.87 (m, 1H), 1.45 (9H, s), 1.28 (3H, d, J = 7.0 Hz) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 159.7, 159.6, 155.9, 145.9, 143.1, 129.5, 129.3, 120.7, 119.5, 114.1, 113.1, 111.6, 111.2, 79.1, 55.1, 47.2, 40.2, 40.1, 33.1, 31.6, 28.4, 28.3, 19.1 ppm. **IR** (NaCl, neat film): 3356, 2974, 2932, 1686, 1601, 1586, 1512, 1489, 1366, 1250, 1165, 1042, 779, 698 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{15}H_{23}NO_3$ [M^+]: 265.1678; found: 265.1684.



(2df): *tert*-butyl 3-(4-methoxyphenyl)propylcarbamate (linear).

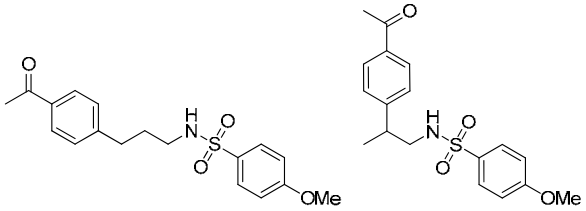
***tert*-butyl 2-(4-methoxyphenyl)propylcarbamate (branched).** Prepared according to the general procedure described above. The product was obtained as a colourless oil in 30% yield (27 mg). Inseparable mixture of linear and branched regioisomers (1:2 ratio). **^{1}H NMR** (400 MHz, $CDCl_3$): major regioisomer (branched) δ 7.12 (2H, d, J = 8.6 Hz), 6.86 (2H, d, J = 8.7 Hz), 4.44 (1H, br. s), 3.79 (3H, s), 3.42-3.33 (1H, m), 3.18-3.09 (1H, m, overlapping with minor regioisomer), 2.93-2.81 (1H, m), 1.41 (9H, s), 1.23 (3H, d, J

= 7.0 Hz) ppm; minor regioisomer (linear) δ 7.09 (2H, d, J = 8.7 Hz), 6.82 (2H, d, J = 8.6 Hz), 4.55 (1H, br. s), 3.78 (3H, s), 3.18-3.09 (2H, m, overlapping with major regioisomer), 2.58 (2H, t, J = 7.7 Hz), 1.81-1.74 (2H, m), 1.44 (9H, s) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 158.2, 157.8, 155.9, 136.2, 133.6, 129.2, 128.1, 114.0, 113.8, 79.1, 55.2, 47.5, 40.2, 39.2, 32.2, 31.9, 28.4, 28.3, 19.3 ppm. **IR** (NaCl, neat film): 3364, 2955, 2932, 1697, 1512, 1366, 1250, 1173, 1034, 833, 756 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{11}H_{15}NO_3$ [M minus $C_4H_8^+$]: 209.1052; found: 209.1059.



(2dg): *tert*-butyl 3-o-tolylpropylcarbamate (linear).

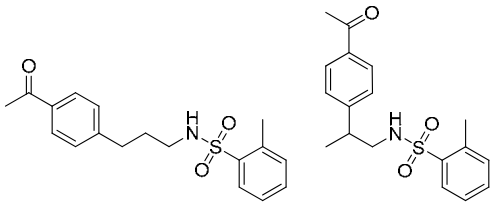
***tert*-butyl 2-o-tolylpropylcarbamate (branched).** Prepared according to the general procedure described above. The product was obtained as a colourless oil in 40% yield (48 mg). Inseparable mixture of linear and branched regioisomers (1:1 ratio). **^{1}H NMR** (400 MHz, $CDCl_3$): linear regioisomer δ 7.22-7.09 (4H, m, overlapping with branched regioisomer), 4.49 (1H, br. s), 3.30-3.14 (2H, m, overlapping with branched regioisomer), 2.62 (2H, t, J = 7.9 Hz), 2.35 (3H, s), 1.82-1.73 (2H, m), 1.46 (9H, s) ppm; branched regioisomer δ 7.22-7.09 (4H, m, overlapping with linear regioisomer), 4.59 (1H, br. s), 3.46-3.37 (1H, m), 3.30-3.14 (2H, m, overlapping with linear regioisomer), 2.30 (3H, s), 1.43 (9H, s), 1.23 (3H, d, J = 6.7 Hz) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 155.9 (2), 142.4, 139.7, 136.2, 135.8, 130.5, 130.2, 128.7, 126.3, 126.1, 126.0 (2), 125.2, 79.1, 46.7, 40.5, 34.9, 30.5, 30.4, 28.4 (2), 19.5, 19.2, 18.9 ppm. **IR** (NaCl, neat film): 3356, 2955, 2932, 1690, 1512, 1458, 1250, 1173, 764 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{11}H_{15}NO_2$ [M minus $C_4H_8^+$]: 193.1103; found: 193.1108.



(2h): *N*-(3-(4-acetylphenyl)propyl)-4-methoxybenzenesulfonamide (linear).

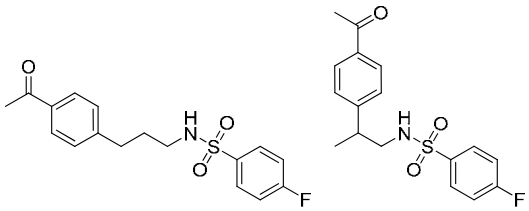
***N*-(2-(4-acetylphenyl)propyl)-4-methoxybenzenesulfonamide (branched).** Prepared according to the general procedure described above. The product was obtained as a pale yellow oil in 92% yield (99 mg). Inseparable mixture of linear and branched regioisomers (6:1 ratio). **^{1}H NMR** (400 MHz, $CDCl_3$): major regioisomer (linear) δ 7.83 (2H, d, J = 8.3 Hz), 7.79 (2H, d, J = 9.0 Hz), 7.18 (2H, d, J = 8.4 Hz), 6.96 (2H, d, J = 9.0 Hz), 5.04 (1H, br. t, J = 6.1 Hz), 3.86 (3H, s), 2.94 (2H, q, J = 6.7 Hz), 2.67 (2H, t, J = 7.7 Hz), 2.57 (3H, s), 1.84-1.77 (2H, m) ppm; characteristic peaks for minor regioisomer δ 7.71 (2H, d, J = 9.0 Hz), 4.77 (1H, br. t, J = 6.2 Hz), 3.85 (3H, s), 3.20-3.01 (3H, m), 2.56 (3H, s), 1.24 (3H, d, J = 6.9 Hz) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): major regioisomer (linear) δ 197.8, 162.8, 146.8, 135.1, 131.3, 129.1, 128.5 (2),

114.2, 55.6, 42.3, 32.6, 30.7, 26.5 ppm. **IR** (NaCl, neat film): 3295, 1682, 1597, 1497, 1412, 1327, 1265, 1157, 1096, 833 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{18}H_{21}NO_4S$ [M^+]: 347.1191; found: 347.1191.



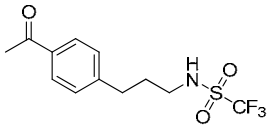
(2i): *N*-(3-(4-acetylphenyl)propyl)-2-methylbenzenesulfonamide (linear).

***N*-(2-(4-acetylphenyl)propyl)-2-methylbenzenesulfonamide (branched).** Prepared according to the general procedure described above. The product was obtained as a colourless oil in 88% yield (93 mg). Inseparable mixture of linear and branched regioisomers (8:1 ratio). **^{1}H NMR** (400 MHz, $CDCl_3$): major regioisomer (linear) δ 7.94 (1H, d, J = 7.5 Hz), 7.83 (2H, d, J = 8.3 Hz), 7.48-7.42 (1H, m), 7.31 (2H, d, J = 7.4 Hz), 7.15 (2H, d, J = 8.3 Hz), 5.11 (1H, br. t, J = 6.0 Hz), 2.97 (2H, q, J = 6.8 Hz), 2.69-2.62 (2H, m), 2.65 (3H, s), 2.56 (3H, s), 1.84-1.76 (2H, m) ppm; characteristic peaks for minor regioisomer (branched) δ 7.23 (1H, d, J = 7.6 Hz), 4.80 (1H, br. t, J = 6.0 Hz), 3.25-3.03 (3H, m), 2.57 (3H, s), 2.41 (3H, s), 1.22 (3H, d, J = 6.9 Hz) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): major regioisomer (linear) δ 197.9, 146.7, 137.8, 136.9, 135.1, 132.7, 132.5, 129.3, 128.5 (2), 126.1, 42.3, 32.6, 30.9, 26.5, 20.2 ppm. **IR** (NaCl, neat film): 3295, 1682, 1613, 1412, 1319, 1273, 1165, 1064, 756 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{18}H_{21}NO_3S$ [M^+]: 331.1242; found: 331.1235.

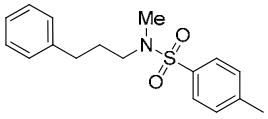


(2j): *N*-(3-(4-acetylphenyl)propyl)-4-fluorobenzenesulfonamide (linear).

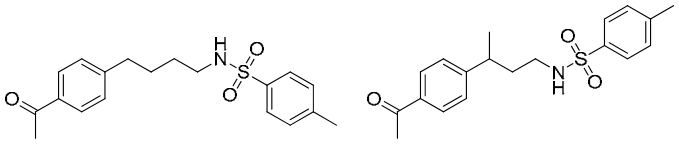
***N*-(2-(4-acetylphenyl)propyl)-4-fluorobenzenesulfonamide (branched).** Prepared according to the general procedure described above. The product was obtained as a colourless oil in 86% yield (94 mg). Inseparable mixture of linear and branched regioisomers (8:1 ratio). **^{1}H NMR** (400 MHz, $CDCl_3$): major regioisomer (linear) δ 7.90-7.83 (4H, m, overlapping with minor regioisomer), 7.22-7.14 (4H, m, overlapping with minor regioisomer), 4.94 (1H, br. t, J = 5.9 Hz), 2.98 (2H, q, J = 6.8 Hz), 2.69 (2H, t, J = 7.6 Hz), 2.58 (3H, s), 1.87-1.79 (2H, m) ppm; characteristic peaks for minor regioisomer δ 4.70 (1H, br. t, J = 6.2 Hz), 3.25-3.04 (3H, m), 1.26 (3H, d, J = 6.9 Hz) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): major regioisomer (linear) δ 197.8, 166.3, 163.8, 146.5, 135.9 (d, J = 3.3 Hz, C-F), 135.3, 129.8, 129.7, 128.6, 128.5, 116.4, 116.2, 42.5, 32.6, 30.8, 26.5 ppm. **IR** (NaCl, neat film): 3295, 1682, 1605, 1497, 1327, 1273, 1157, 1096, 841 cm^{-1}. **HRMS** *m/z* (ESI): calcd. for $C_{17}H_{19}FNO_3S$ [MH^+]: 336.1064; found: 336.1081.



(2k): *N*-(3-(4-acetylphenyl)propyl)-1,1,1-trifluoromethanesulfonamide. Prepared according to the general procedure described above. The product was obtained as a white solid in 43% yield (40 mg). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.89 (2H, d, J = 8.3 Hz), 7.28 (2H, d, J = 8.4 Hz), 5.44 (1H, br. s), 3.34 (2H, t, J = 7.0 Hz), 2.77 (2H, t, J = 7.7 Hz), 2.59 (3H, s), 2.02-1.94 (2H, m) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 198.5, 146.2, 135.4, 128.8, 128.6, 119.9 (q, J = 321.0 Hz, CF_3), 43.8, 32.4, 31.4, 26.5 ppm. **IR** (NaCl, neat film): 3202, 1667, 1605, 1435, 1373, 1273, 1188, 1080, 964, 818 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{12}H_{14}F_3NO_3S$ [M^+]: 309.0647; found: 309.0645. **M.p.** 60-62 ^{o}C.

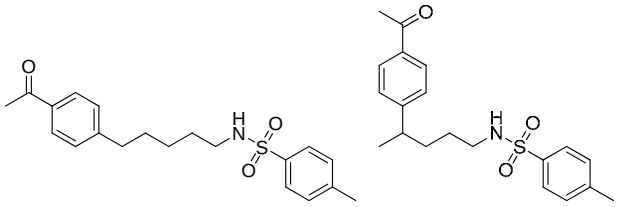


(2l): *N*-4-dimethyl-*N*-(3-phenylpropyl)benzenesulfonamide. Prepared according to the general procedure described above. The product was obtained as a colourless oil in 68% yield (66 mg). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.65 (2H, d, J = 8.3 Hz), 7.31-7.25 (4H, m), 7.21-7.16 (3H, m), 3.03 (2H, t, J = 7.1 Hz), 2.71 (3H, s), 2.66 (2H, t, J = 7.8 Hz), 2.42 (3H, s), 1.89-1.81 (2H, m) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 143.2, 141.3, 134.5, 129.6, 128.4, 128.3, 127.4, 125.9, 49.7, 34.7, 32.7, 29.3, 21.4 ppm. **IR** (NaCl, neat film): 2924, 1597, 1458, 1343, 1165, 964, 818 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{17}H_{21}NO_2S$ [M^+]: 303.1293; found: 303.1287.



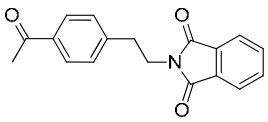
(5a): *N*-(4-(4-acetylphenyl)butyl)-4-methylbenzenesulfonamide (linear).

***N*-(3-(4-acetylphenyl)butyl)-4-methylbenzenesulfonamide (branched).** Prepared according to the general procedure described above. The product was obtained as a colourless oil in 62% yield (73 mg). Inseparable mixture of linear and branched regioisomers (1:1 ratio). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.84 (4H, t, J = 8.3 Hz), 7.74 (2H, t, J = 8.3 Hz), 7.69 (2H, t, J = 8.3 Hz), 7.30-7.26 (4H, m), 7.20-7.17 (4H, m), 4.86 (2H, br. t, J = 6.1 Hz), 2.95 (2H, q, J = 6.7 Hz, linear regioisomer), 2.86-2.77 (3H, m), 2.61 (2H, t, J = 7.6 Hz, linear regioisomer), 2.57 (6H, s), 2.41 (6H, s), 1.79-1.73 (2H, m), 1.66-1.58 (2H, m), 1.53-1.46 (2H, m), 1.20 (3H, d, J = 7.0 Hz) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 197.8 (2), 151.7, 147.6, 143.4, 143.3, 136.9, 136.8, 135.4, 135.0, 129.6, 128.7, 128.5 (2), 127.1, 127.0, 42.8, 41.3, 37.5, 37.0, 35.2, 29.0, 27.7, 26.5, 21.8, 21.4 ppm. **IR** (NaCl, neat film): 3295, 2940, 1682, 1597,1327, 1273, 1165, 1096 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{19}H_{23}NO_3S$ [M^+]: 345.1399; found: 345.1396.



(5b): *N*-(5-(4-acetylphenyl)pentyl)-4-methylbenzenesulfonamide (linear).

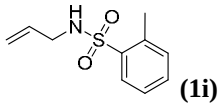
***N*-(4-(4-acetylphenyl)pentyl)-4-methylbenzenesulfonamide (branched).** Prepared according to the general procedure described above. The product was obtained as a colourless oil in 48% yield (73 mg). Inseparable mixture of linear and branched regioisomers (1:1.6 ratio). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.86 (4H, d, J = 8.3 Hz, linear+branched), 7.74 (2H, d, J = 8.3 Hz, linear), 7.71 (2H, d, J = 8.3 Hz, branched), 7.31-7.26 (4H, m, linear+branched), 7.23-7.18 (4H, m, linear+branched), 4.57 (1H, br. t, J = 6.1 Hz, linear), 4.53 (1H, br. t, J = 6.2 Hz, linear), 2.95-2.86 (4H, m, linear+branched), 2.69 (1H, sextet, J = 7.1 Hz, branched), 2.61 (2H, t, J = 7.7 Hz, linear), 2.58 (3H, s, linear), 2.57 (3H, s, branched), 2.42 (6H, br. s, linear+branched), 1.61-1.24 (10H, m, linear+branched), 1.21 (3H, d, J = 6.9 Hz, branched) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 197.9, 197.8, 152.6, 148.1, 143.3, 136.9 (2), 135.2, 134.9, 129.6, 128.6, 128.5, 128.4, 127.1, 127.0 (2), 43.0 (2), 39.5, 35.6, 34.7, 30.4, 29.3, 27.6, 26.5, 26.0, 21.9, 21.4 ppm. **IR** (NaCl, neat film): 3295, 2940, 1682, 1613, 1327, 1273, 1165, 1096 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{20}H_{25}NO_3S$ [M^+]: 359.1555; found: 359.1547.



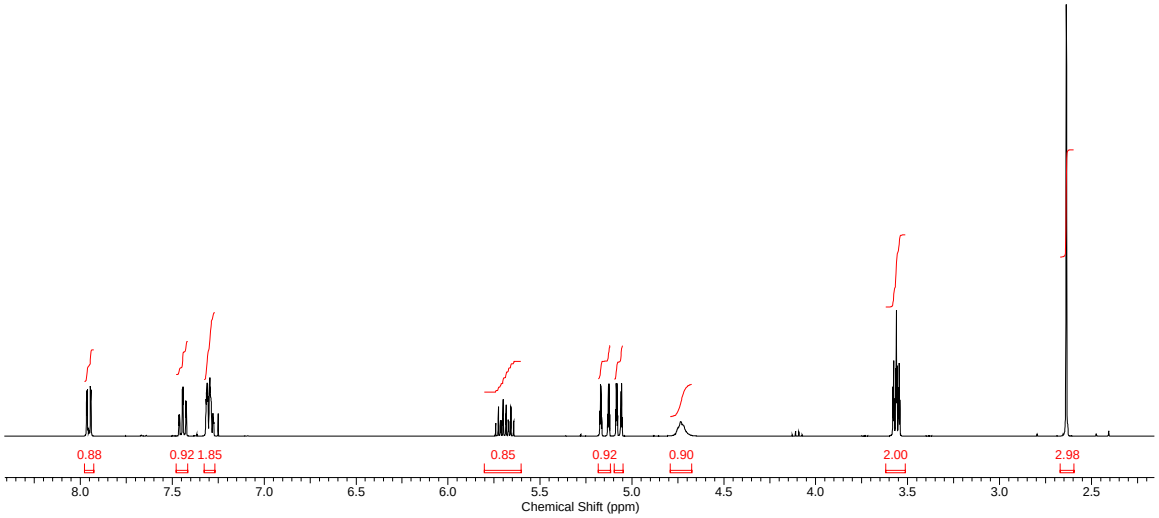
(5c): 2-(4-acetylphenethyl)isoindoline-1,3-dione. Prepared according to the general procedure described above. The product was obtained as a white solid in 35% yield (32 mg). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.88 (2H, d, J = 8.2 Hz), 7.83 (2H, dd, J = 3.1, 5.4 Hz), 7.71 (2H, dd, J = 3.0, 5.5 Hz), 7.34 (2H, d, J = 8.1 Hz), 3.96 (2H, t, J = 7.5 Hz), 3.07 (2H, t, J = 7.5 Hz), 2.58 (3H, s) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 197.7, 168.0, 143.6, 135.7, 134.0, 131.9, 129.1, 128.7, 123.3, 38.7, 34.5, 26.5 ppm. **IR** (NaCl, neat film): 2940, 1705, 1674, 1366, 1018, 725 cm^{-1}. **HRMS** *m/z* (EI): calcd. for $C_{18}H_{15}NO_3$ [M^+]: 293.1052; found: 293.1047. **M.p.** 178-180 ^{o}C.

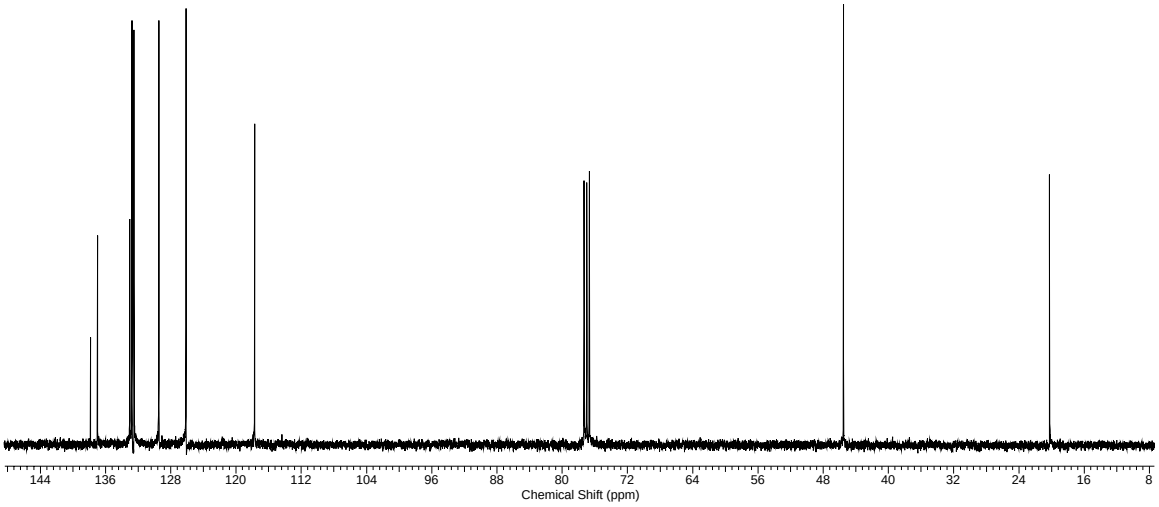
Spectra.

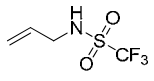
Substrates:



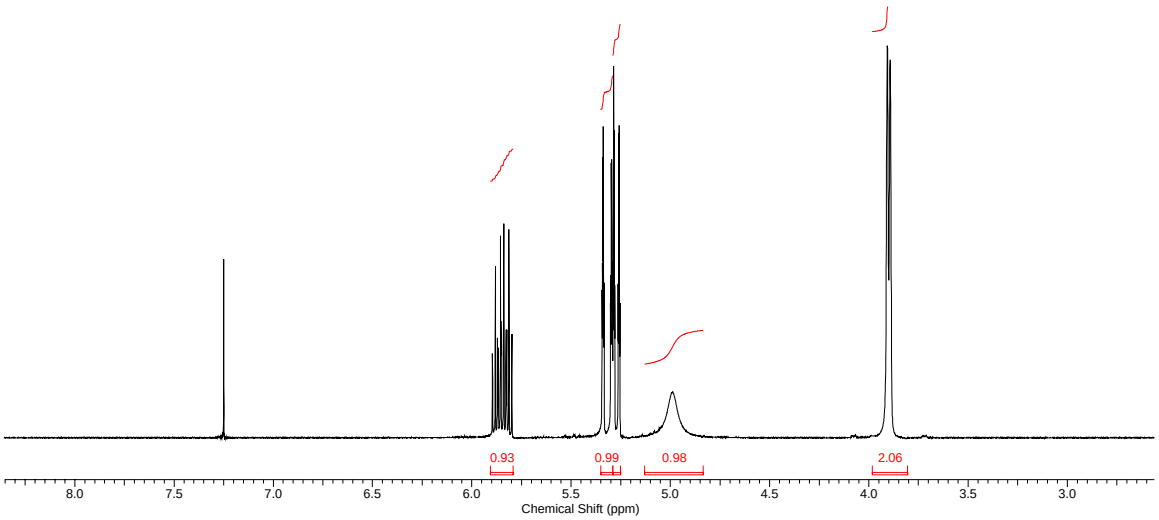
(1i)







(1k)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0.98
2.06
8.0
7.5
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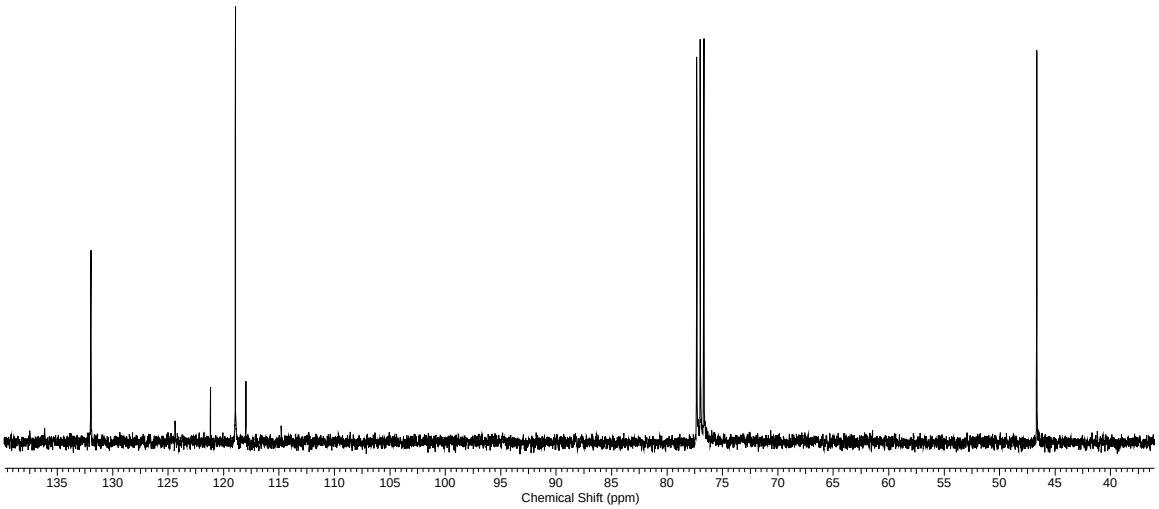
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
Chemical Shift (ppm)


135
130
125
120
115
110
105
100
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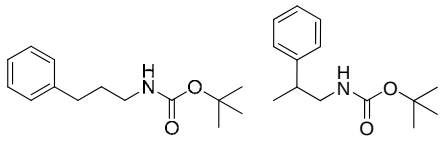
85
80
75
70
65
60
55
50
45
40
Chemical Shift (ppm)

Equation 2:



(2-Boc, 3-Boc)

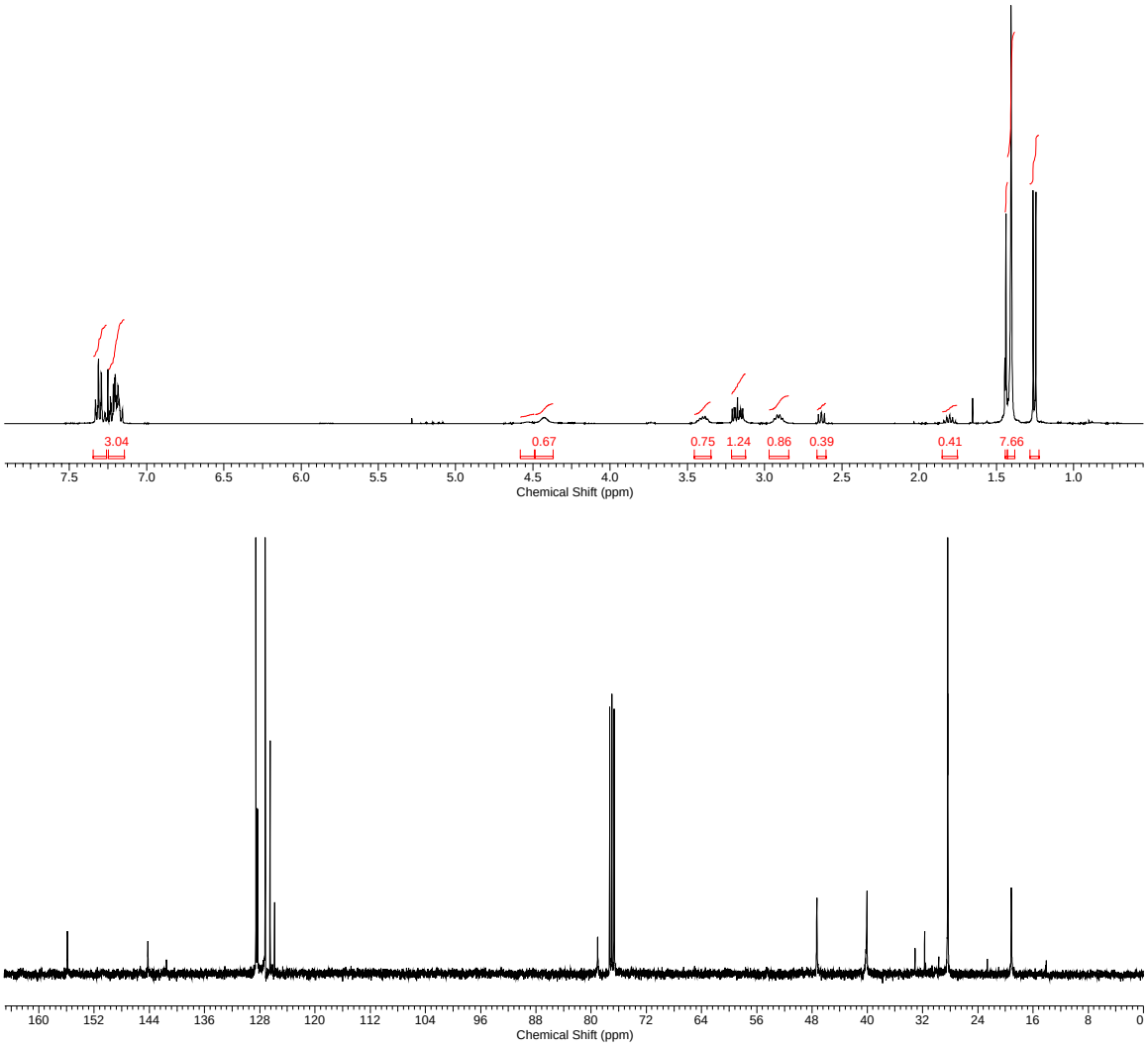
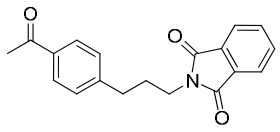
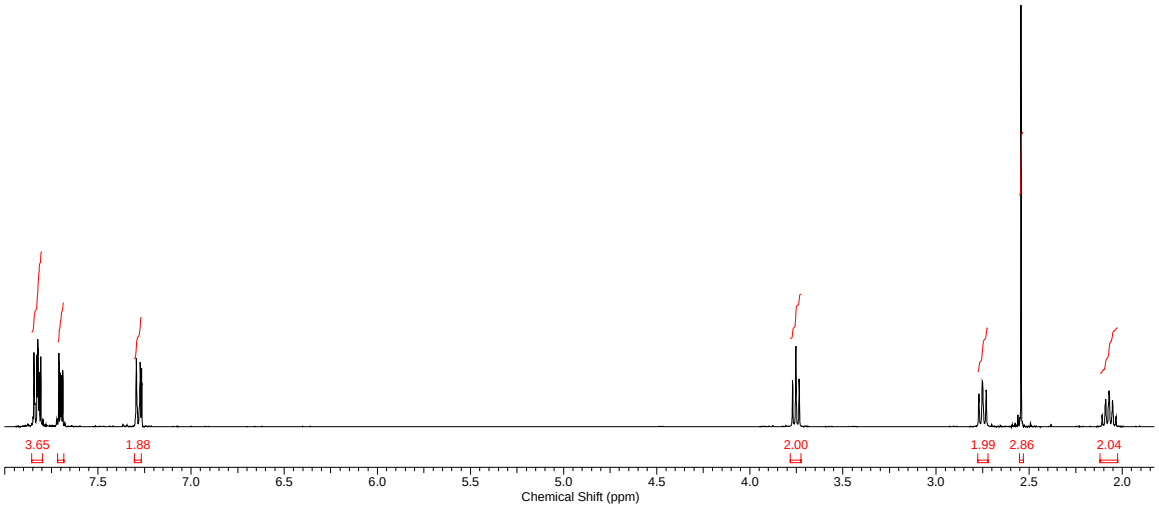


Table 1:



(2aa)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1.99
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7.0
6.5
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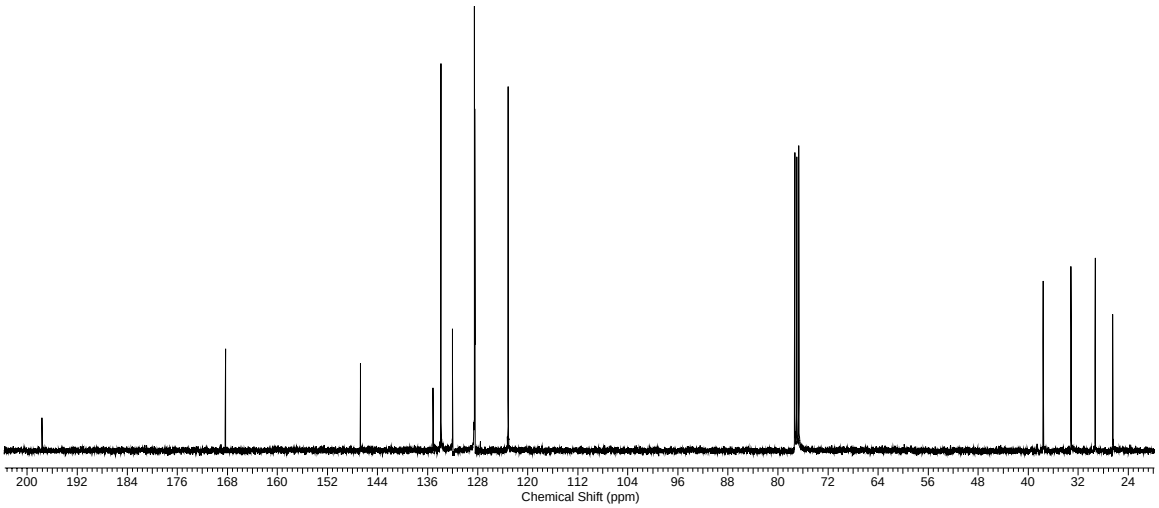
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
Chemical Shift (ppm)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56
48
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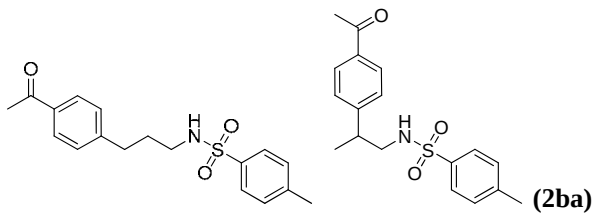
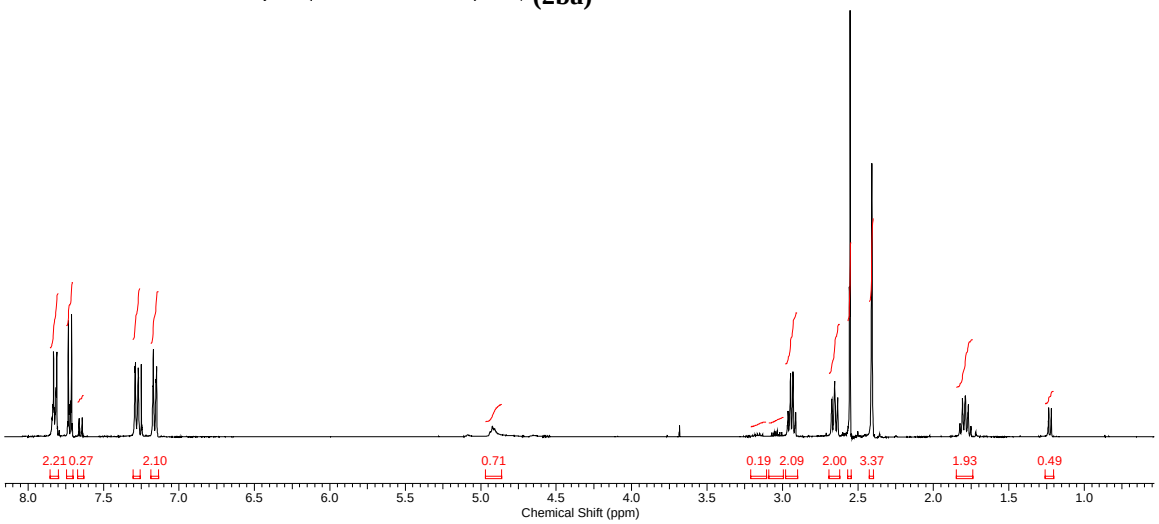
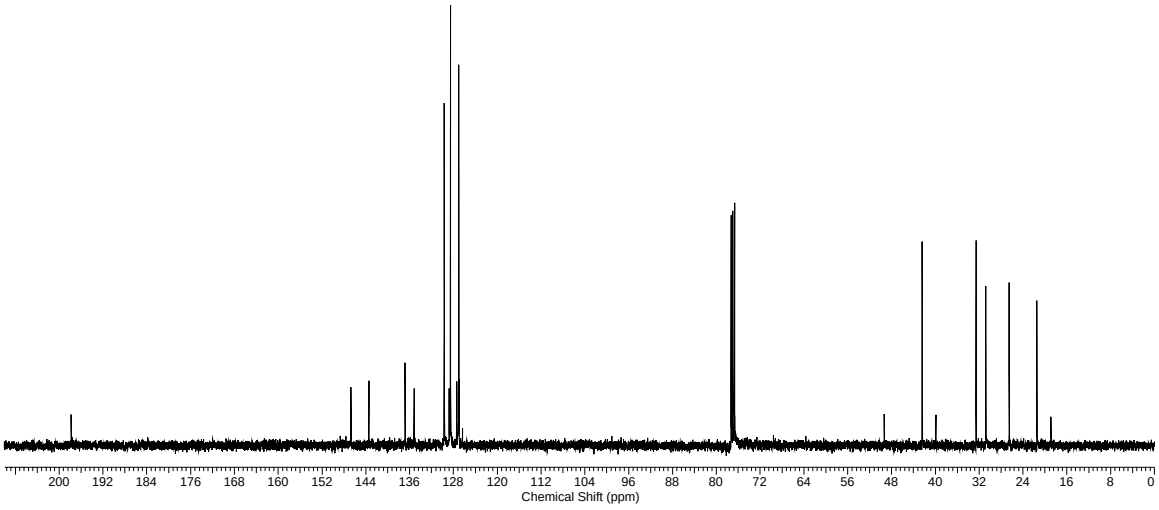
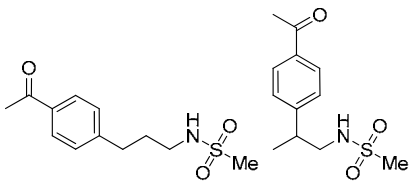
32
24
Chemical Shift (ppm)



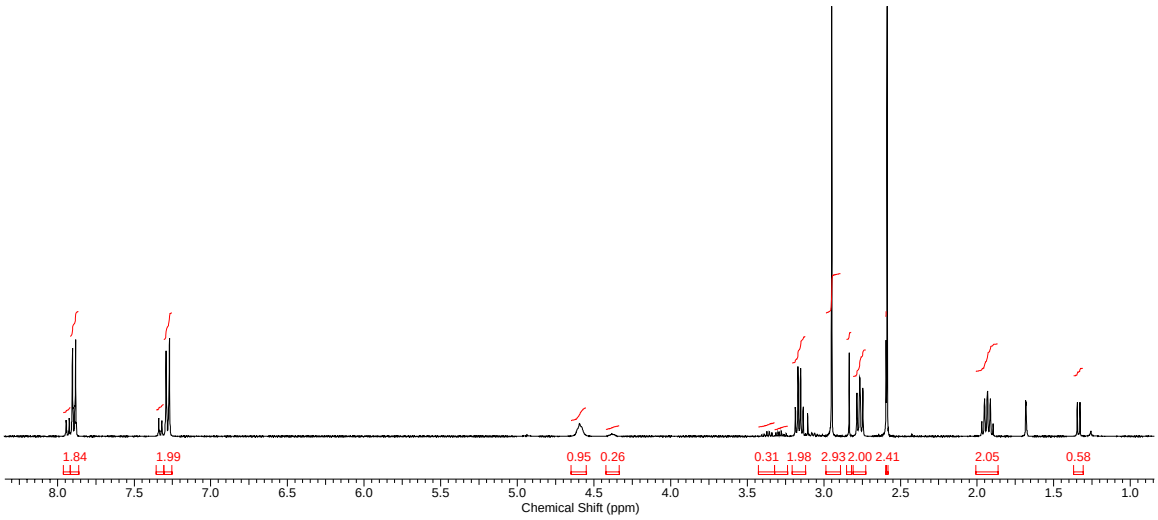
(2ba)

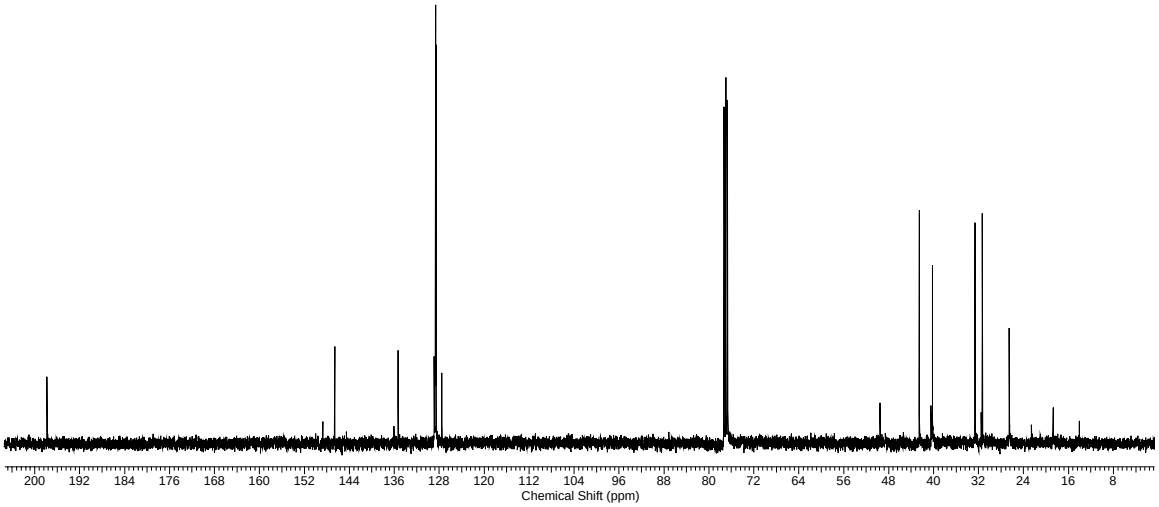

2.21 0.27
2.10
0.71
0.19 2.09
2.00 3.37
1.93
0.49
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
Chemical Shift (ppm)

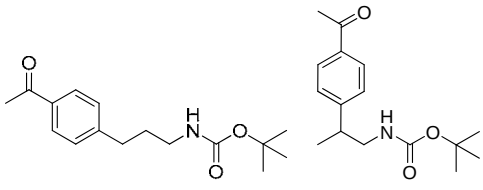

200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)


O
H
N
S
O
O
Me
O
H
N
S
O
O
Me

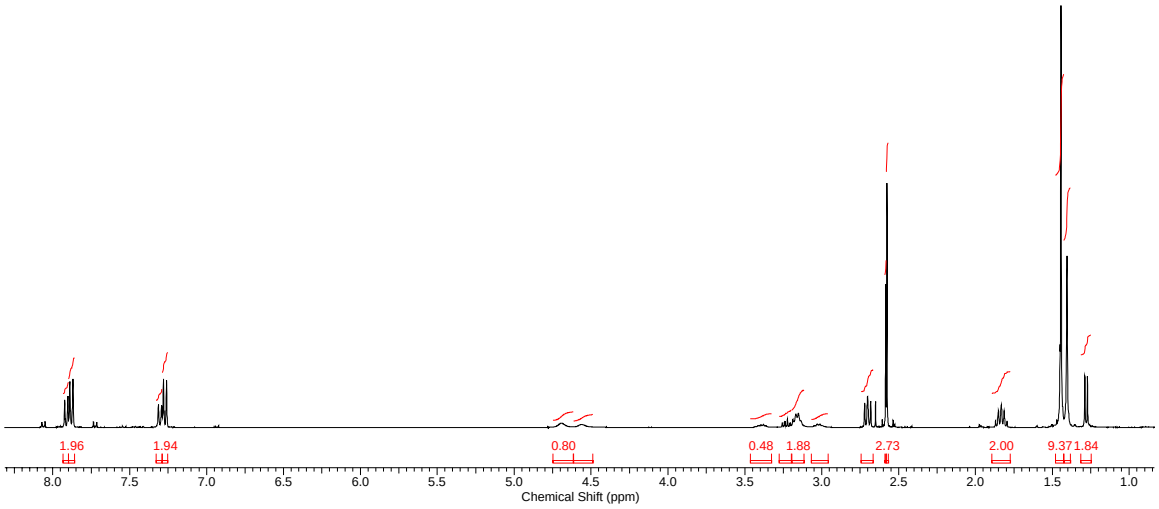
(2ca)

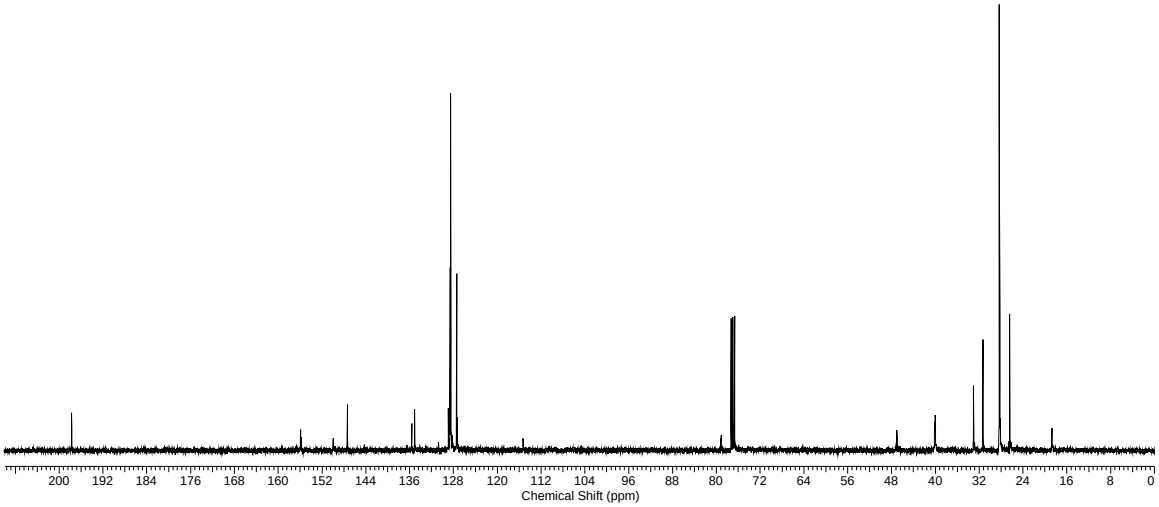

1.84
1.99
0.95
0.26
0.31
1.98
2.93
2.00
2.41
2.05
0.58
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
Chemical Shift (ppm)

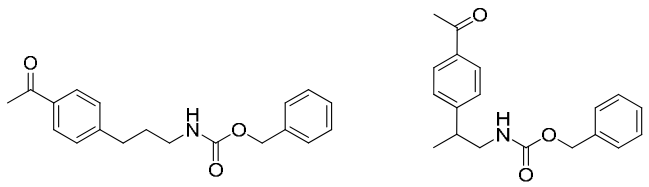

200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
Chemical Shift (ppm)



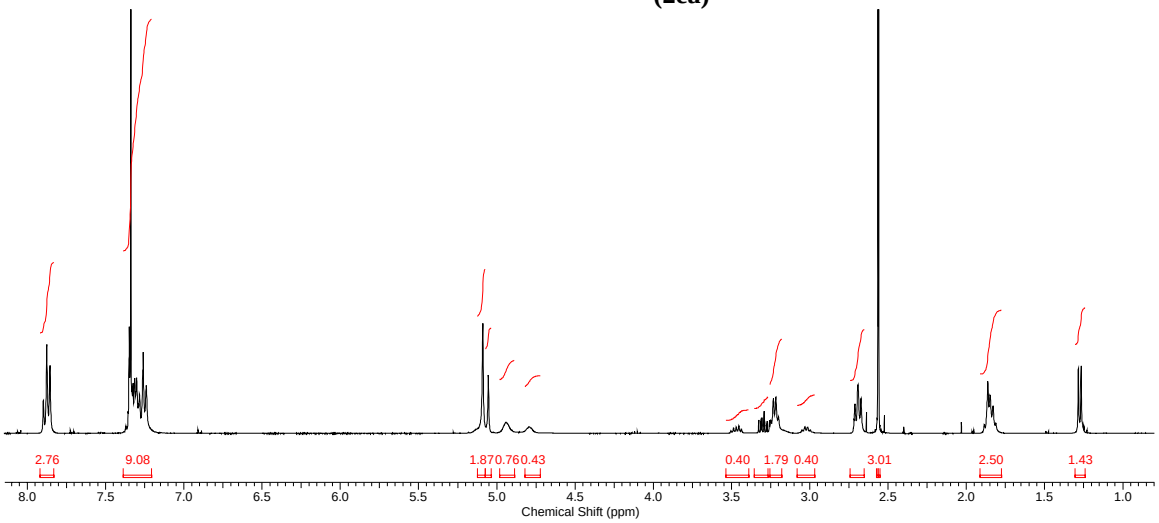
(2da)

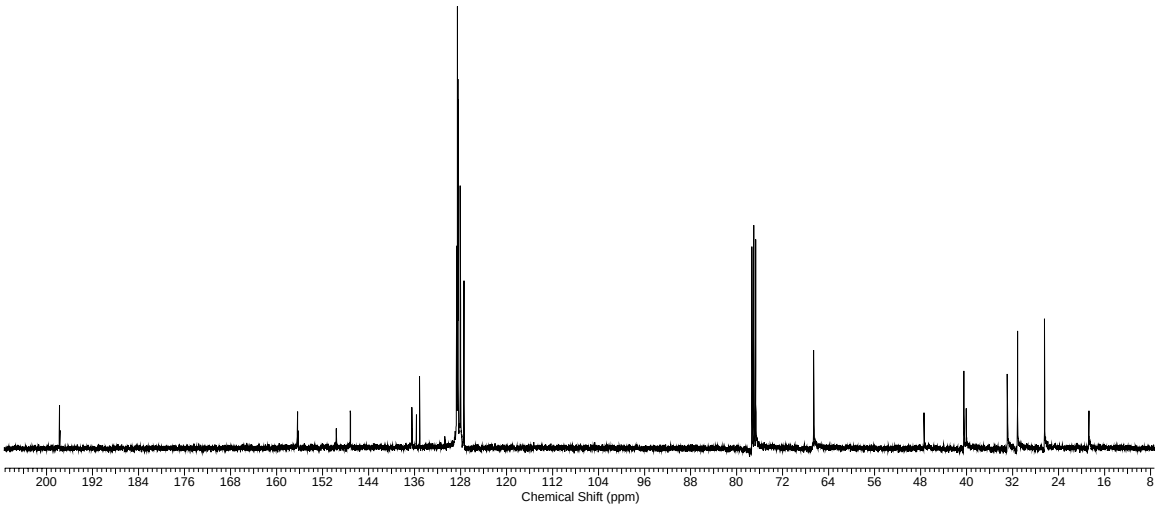

1.96
1.94
0.80
0.48
1.88
2.73
2.00
9.37
1.84
Chemical Shift (ppm)

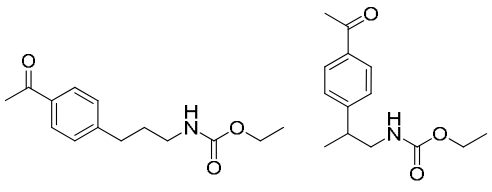

Chemical Shift (ppm)



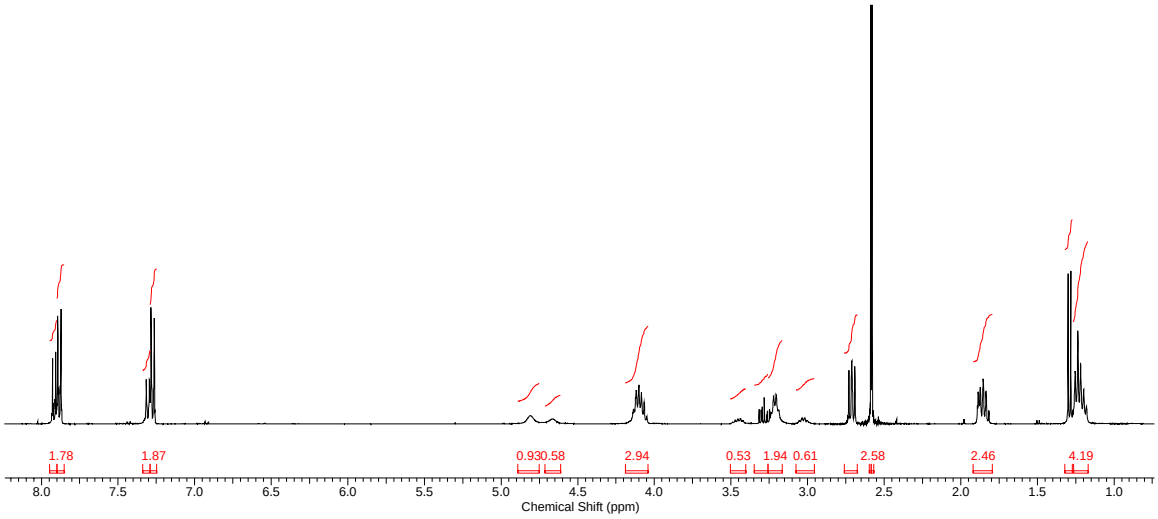
(2ea)

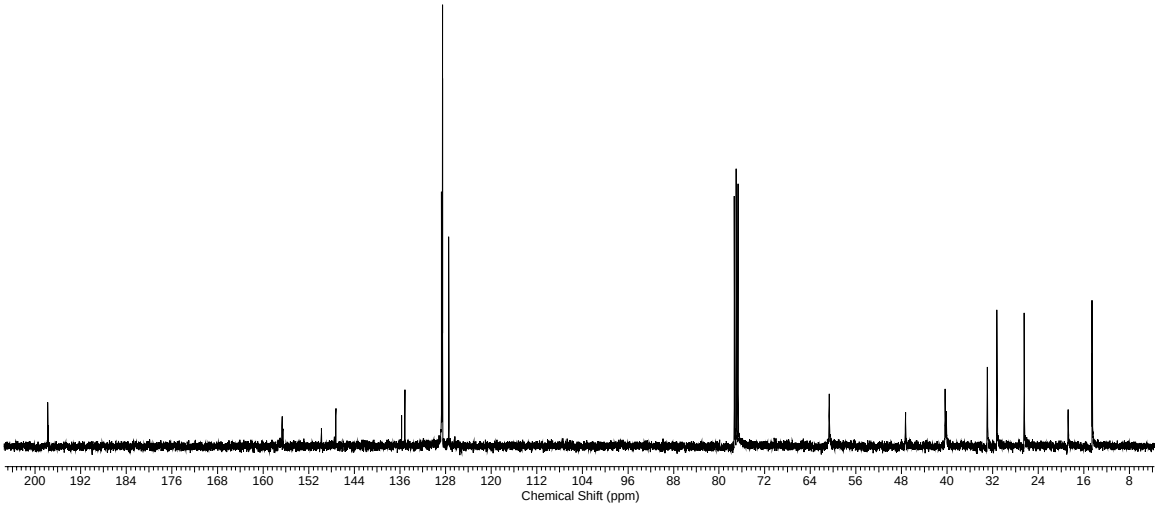

2.76
9.08
1.87
0.76
0.43
0.40
1.79
0.40
3.01
2.50
1.43
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
Chemical Shift (ppm)

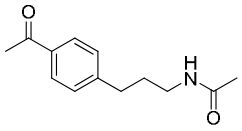

200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
Chemical Shift (ppm)



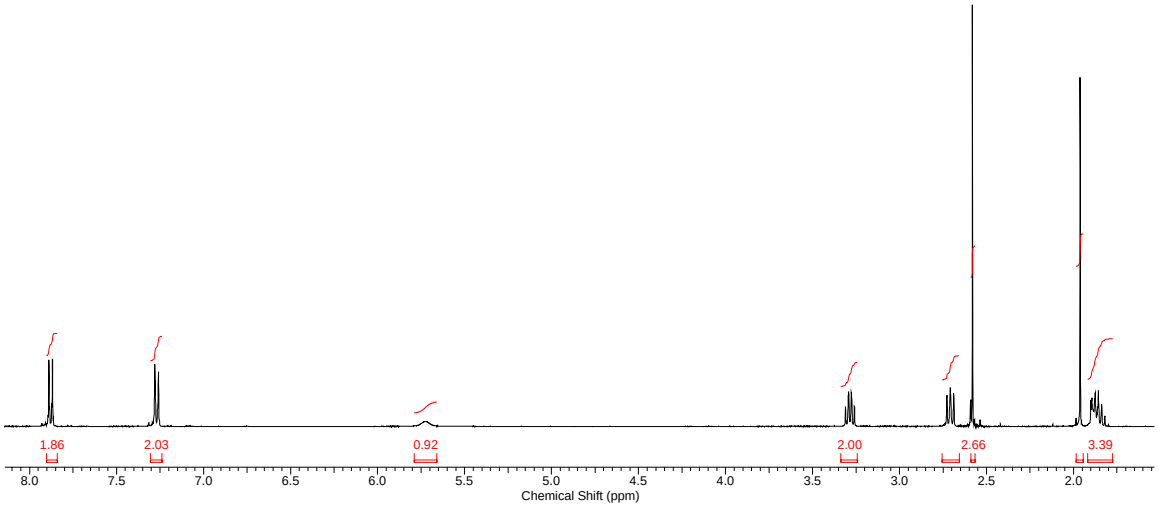
(2fa)

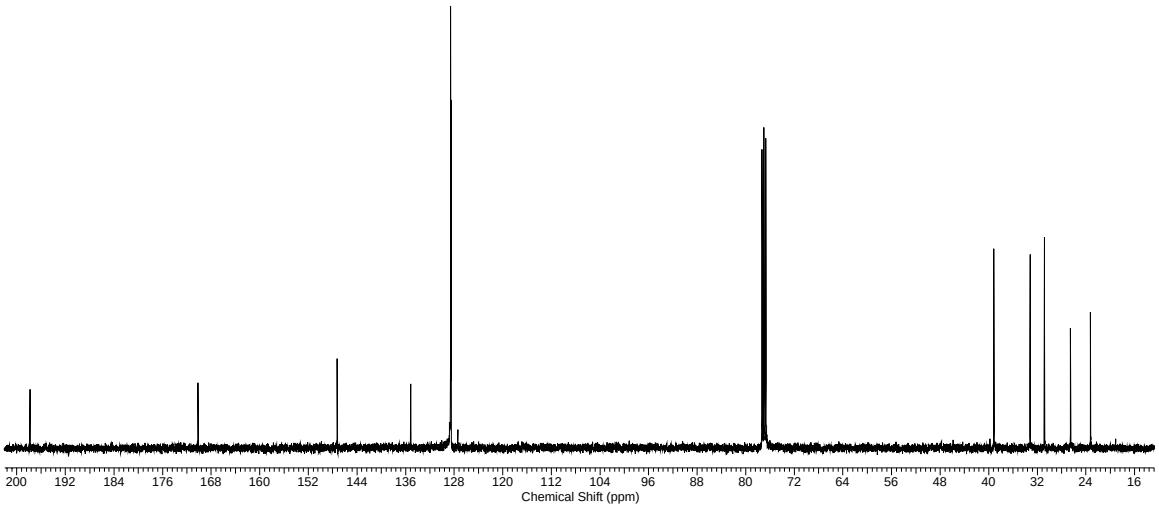

1.78
1.87
0.93
0.58
2.94
0.53
1.94
0.61
2.58
2.46
4.19
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
Chemical Shift (ppm)

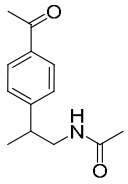

200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
Chemical Shift (ppm)



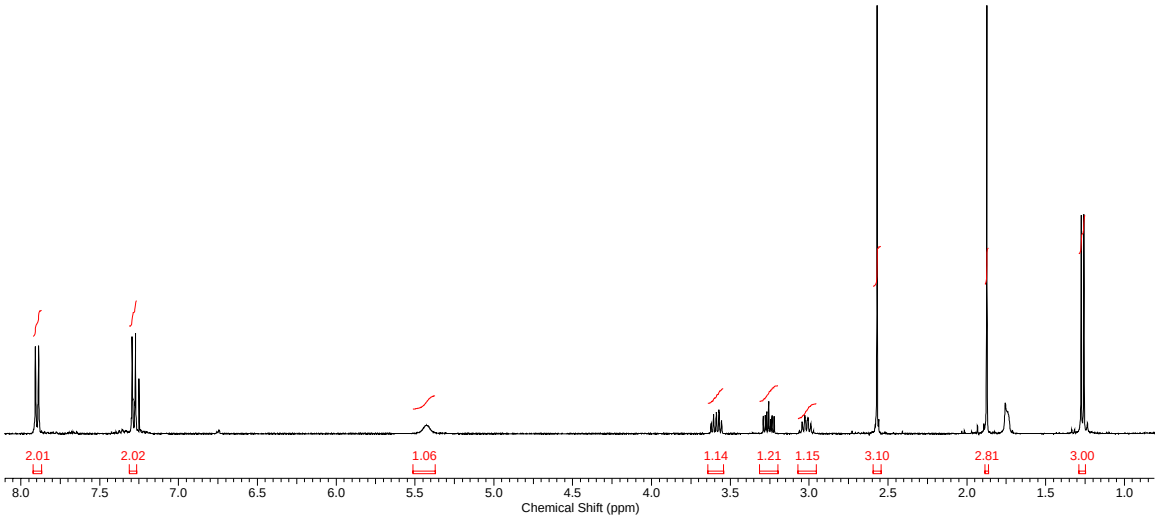
(2ga) Linear regioisomer







(2ga) Branched regioisomer



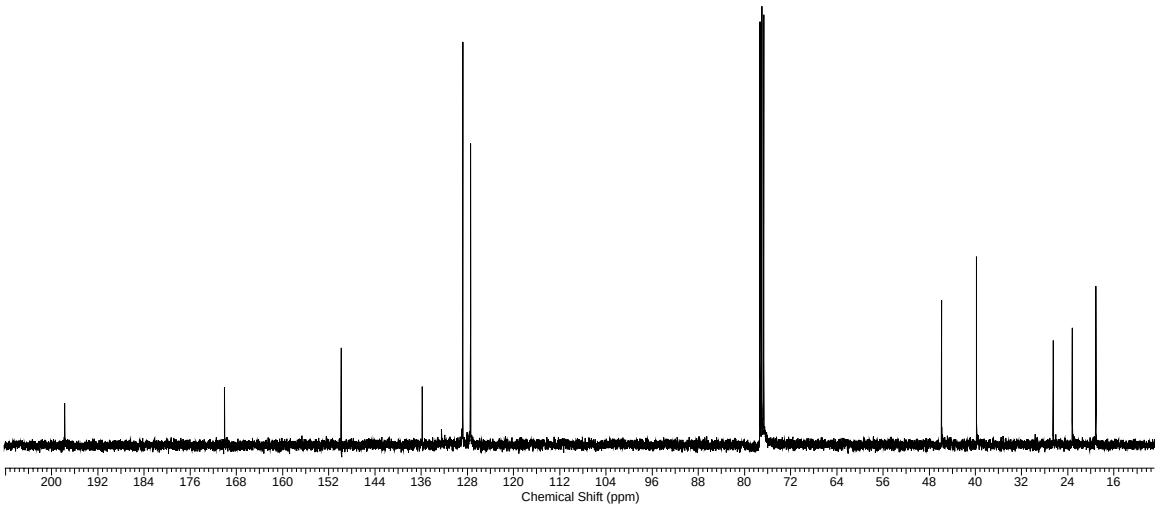
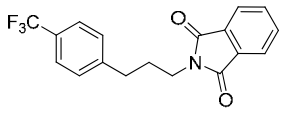
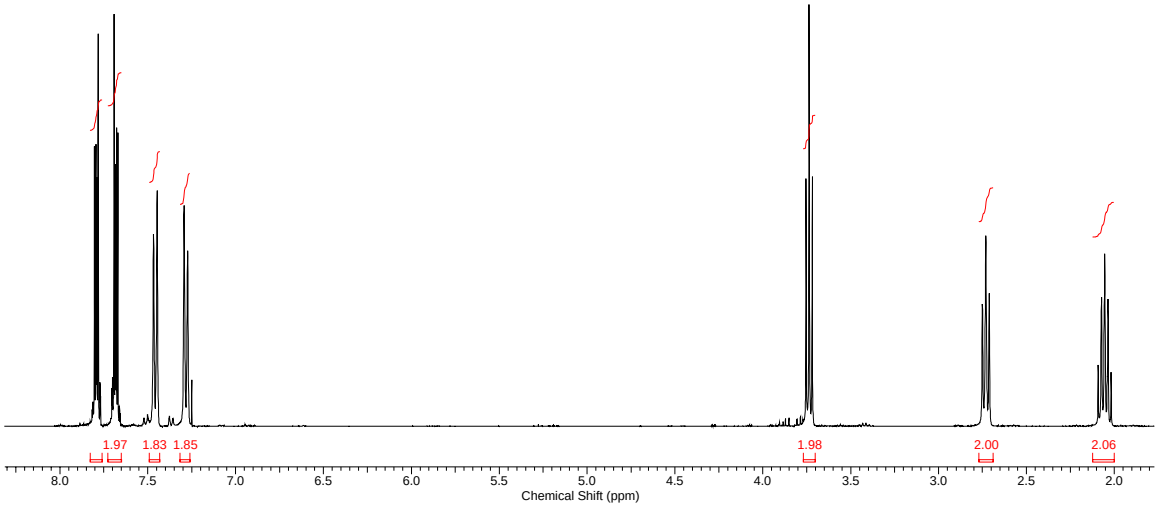
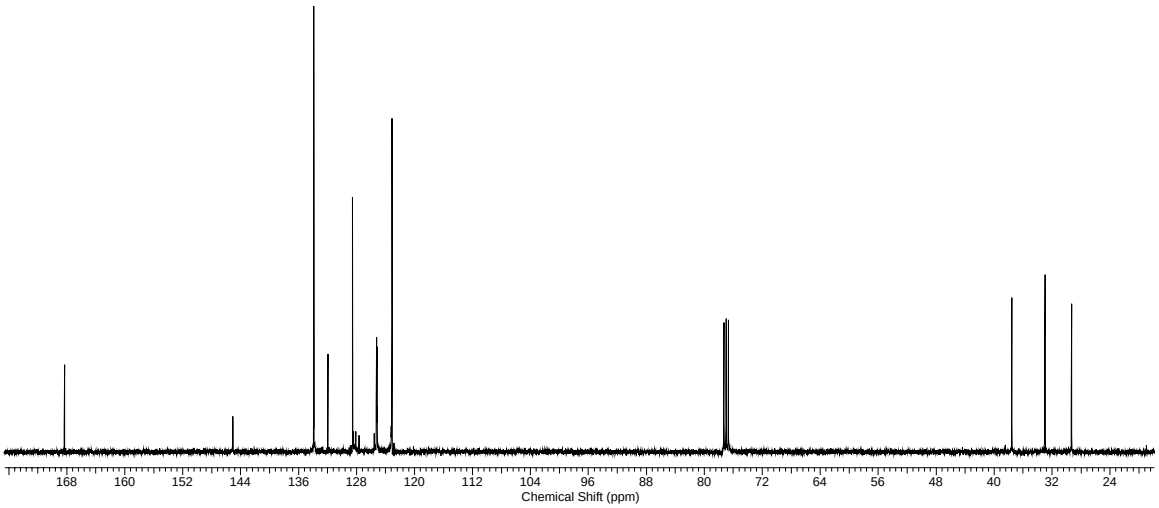


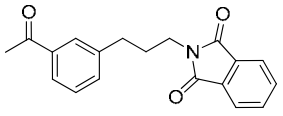
Table 2:


F3C
O
N
O

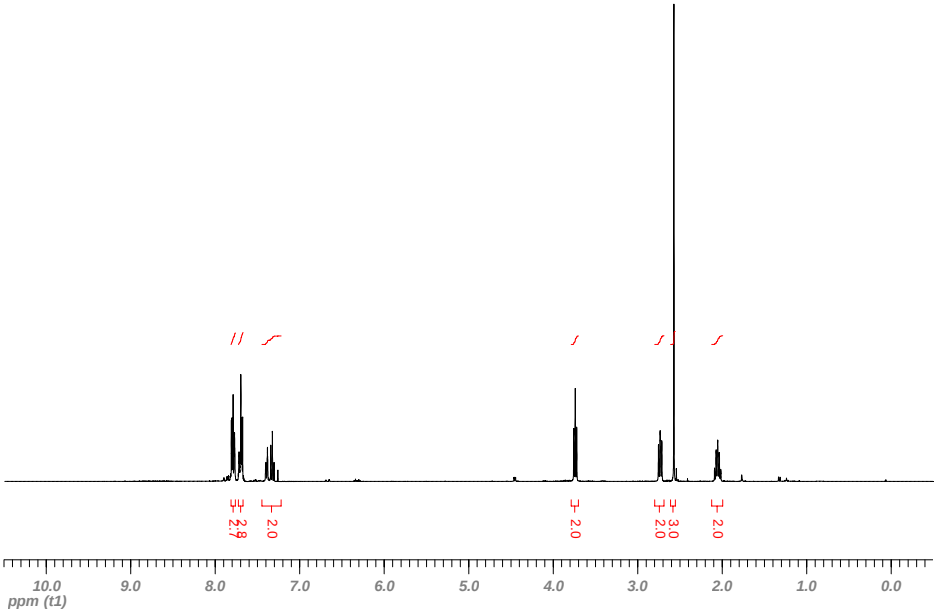
(2ab)

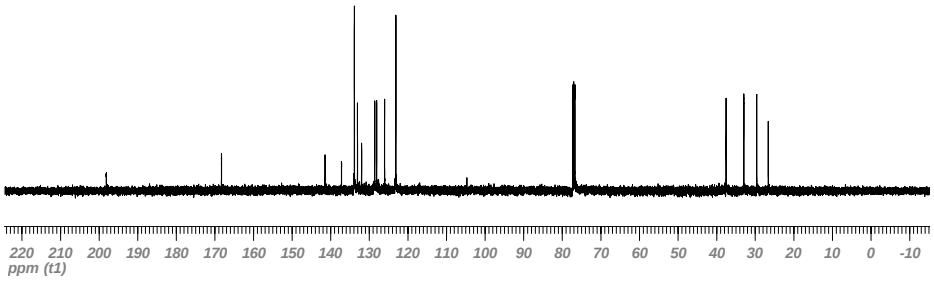

1.97
1.83
1.85
1.98
2.00
2.06
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
Chemical Shift (ppm)

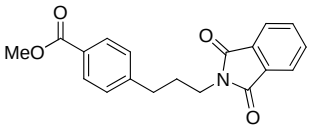

168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
Chemical Shift (ppm)



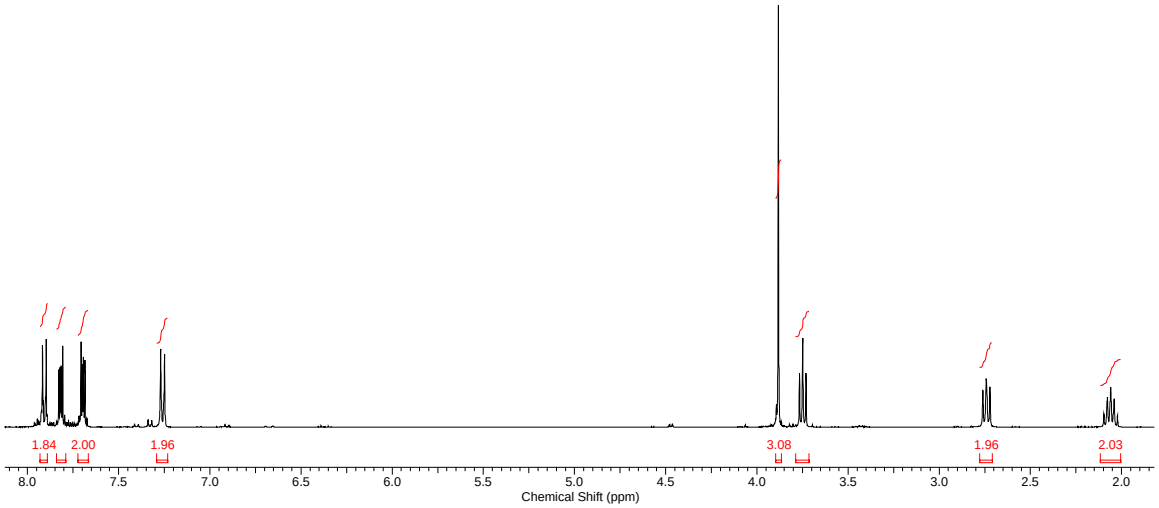
(2ac)

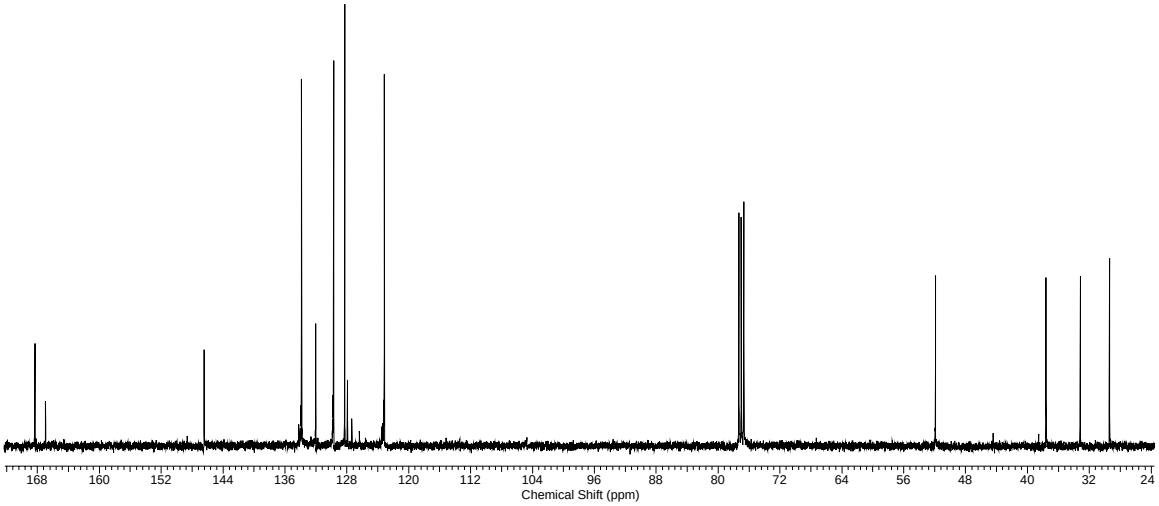

2.9
2.0
2.0
3.0
2.0
2.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

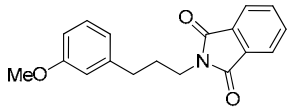

220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
ppm (t1)


O
MeO
O
N
O

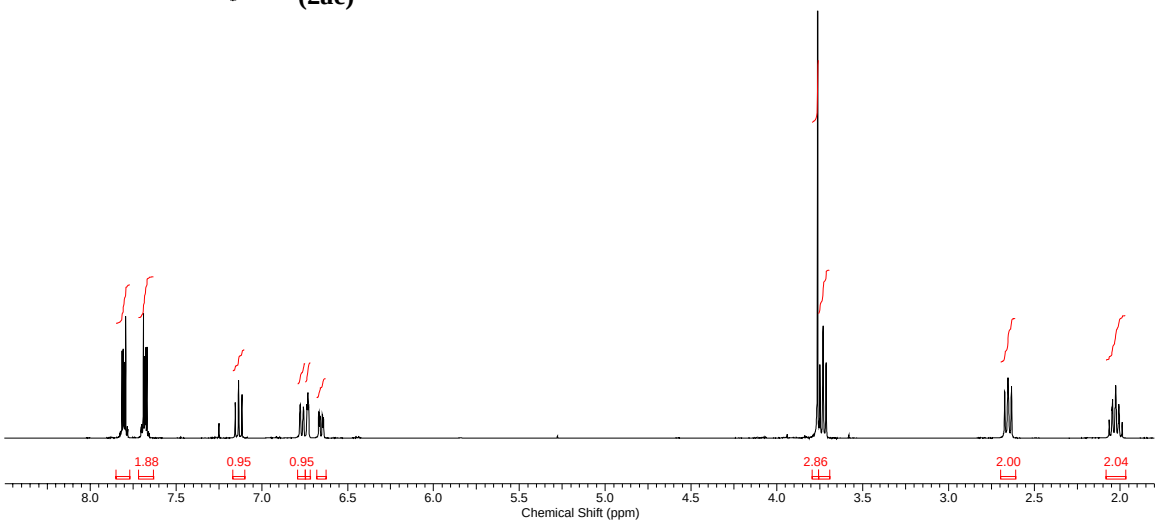
(2ad)

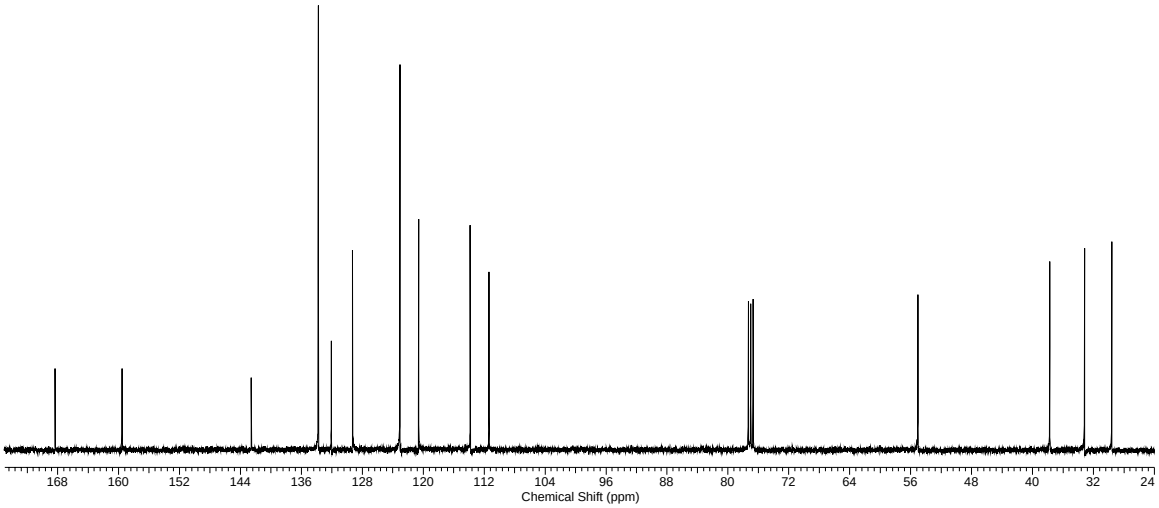

1.84
2.00
1.96
3.08
1.96
2.03
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
Chemical Shift (ppm)

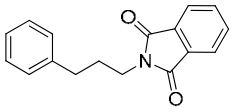

168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
Chemical Shift (ppm)


MeO
O
N
O

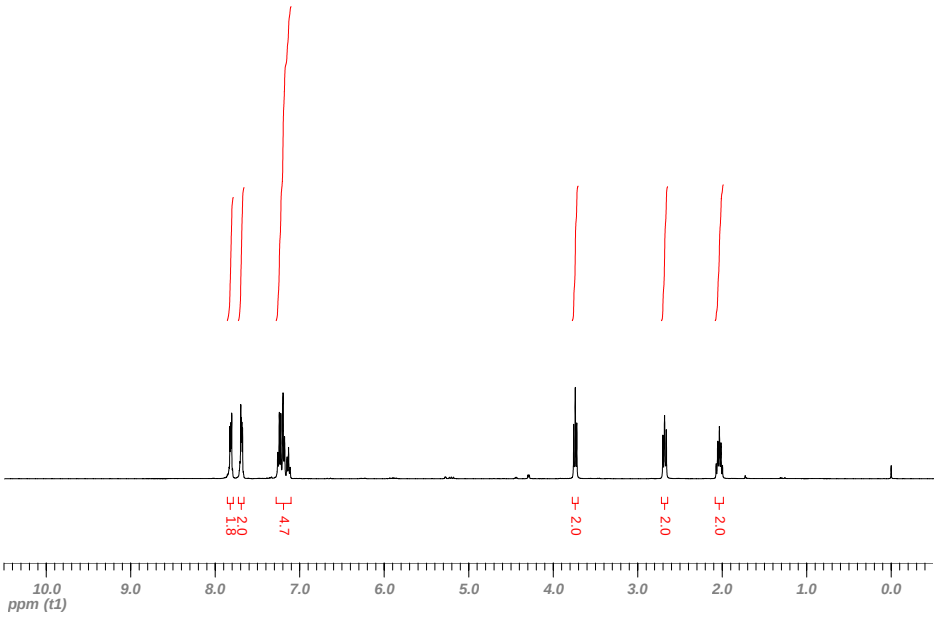
(2ae)

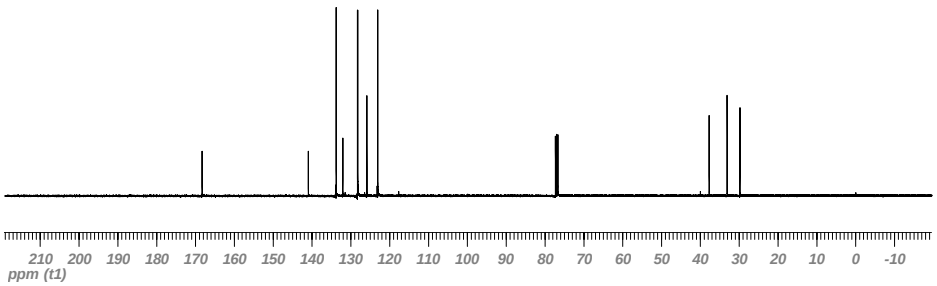

1.88
0.95
0.95
2.86
2.00
2.04
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
Chemical Shift (ppm)

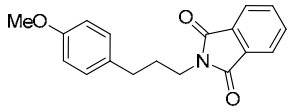

168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
Chemical Shift (ppm)



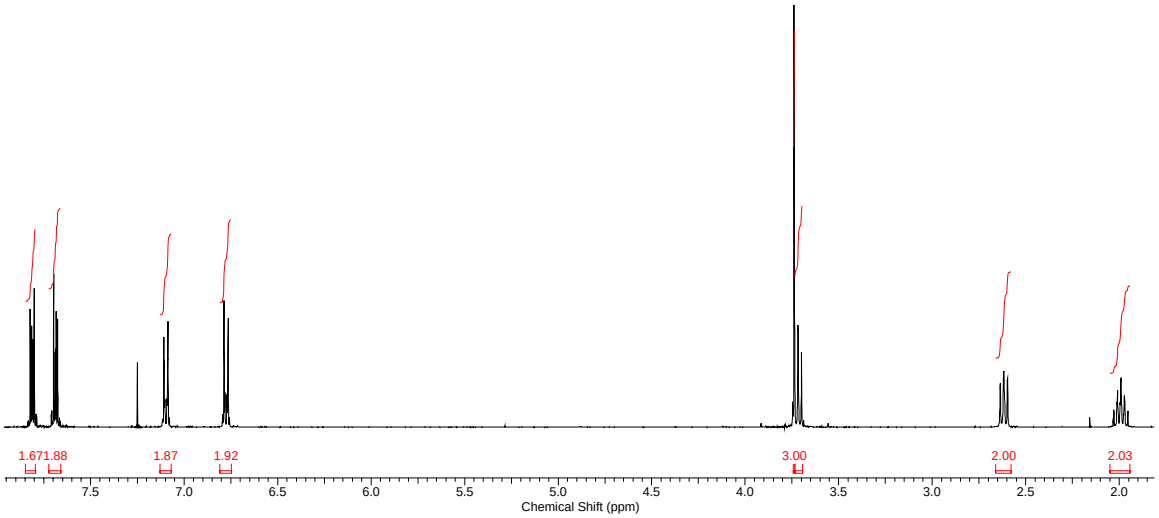
(2af)

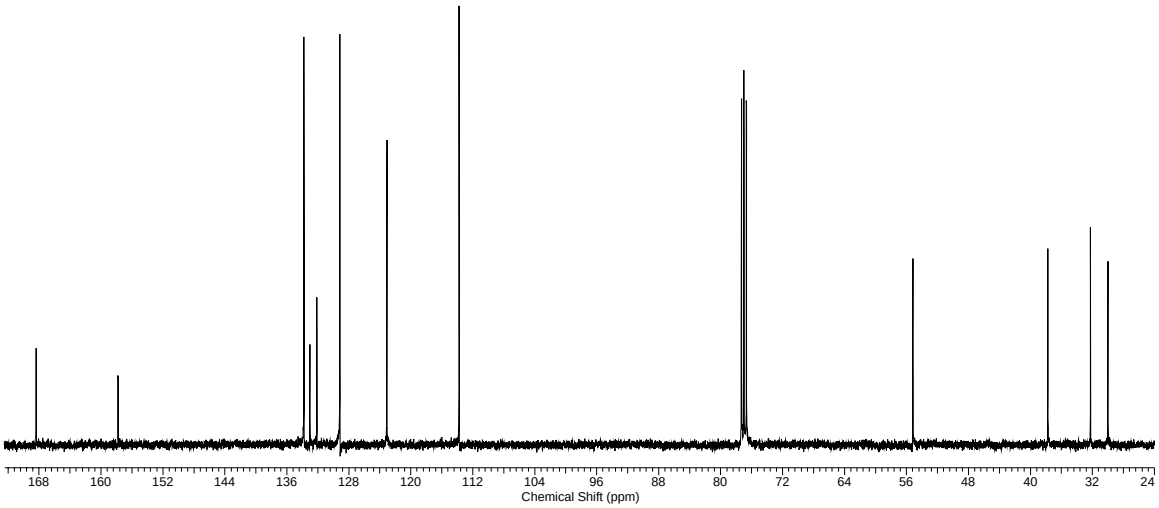

1.8
2.0
4.7
2.0
2.0
2.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

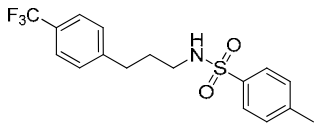

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
ppm (t1)


MeO
O
N
O

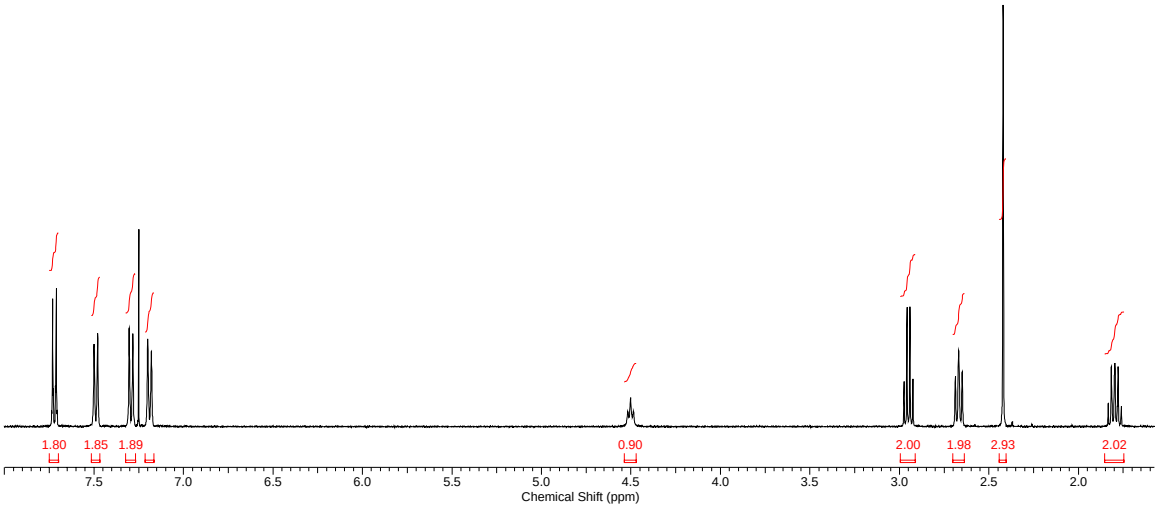
(2ag)

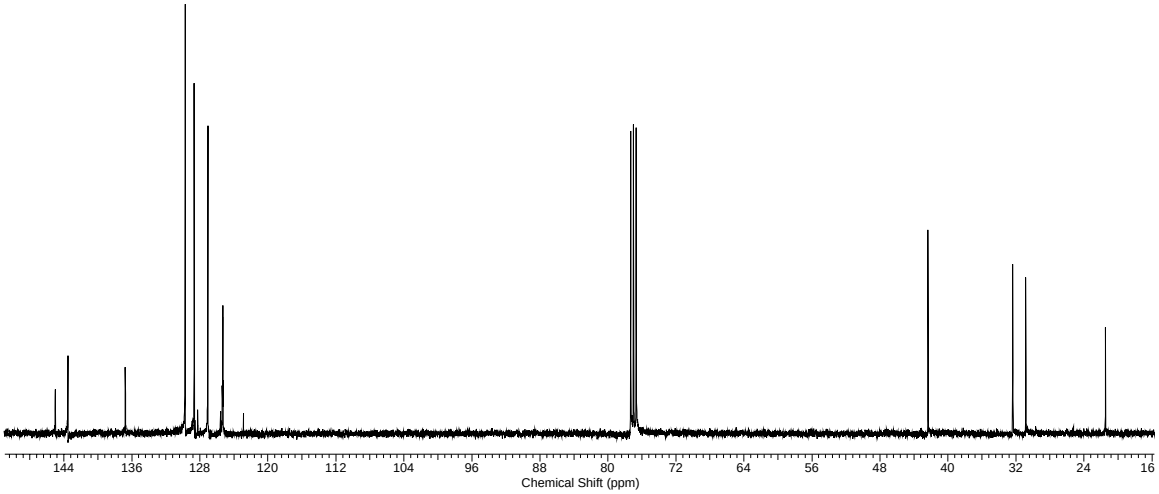

1.67
1.88
1.87
1.92
3.00
2.00
2.03
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
Chemical Shift (ppm)

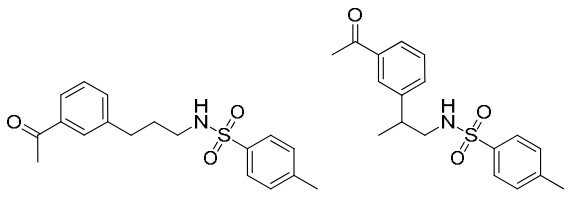

168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
Chemical Shift (ppm)



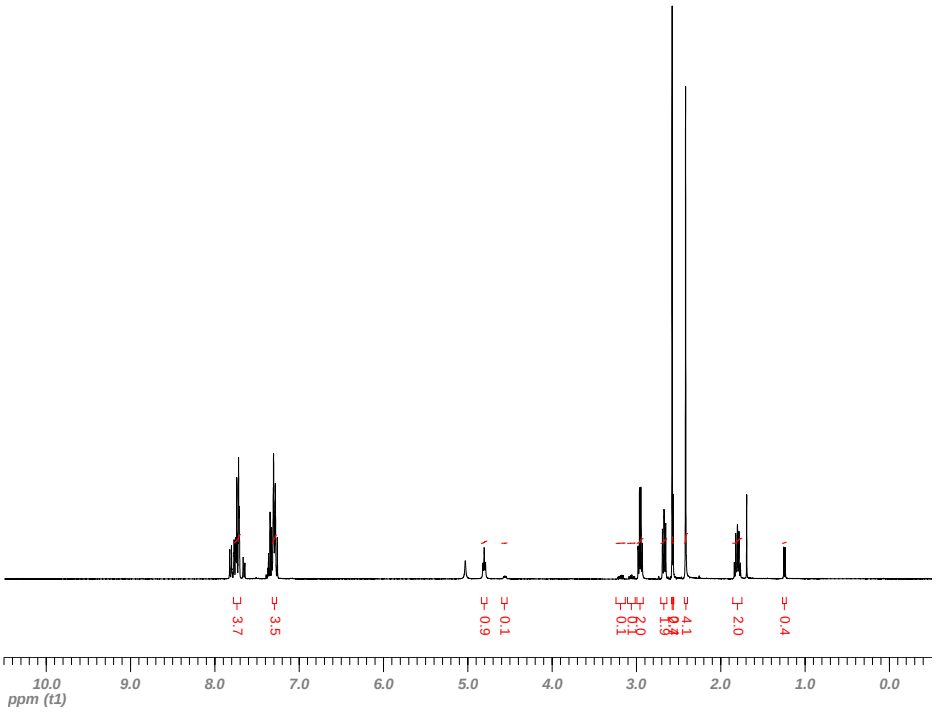
(2bb)

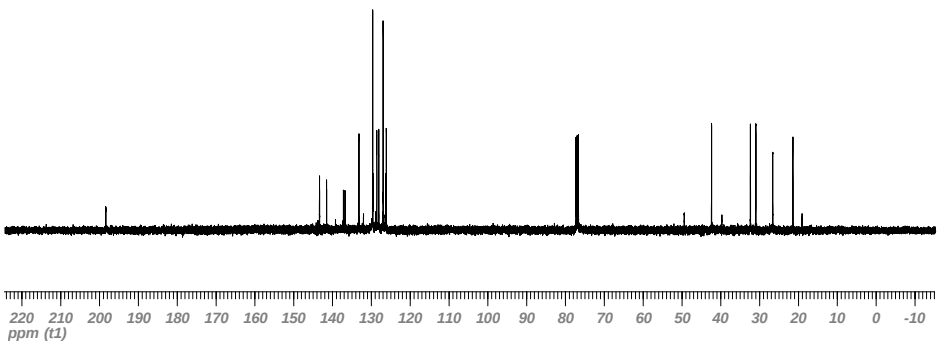

1.80
1.85
1.89
0.90
2.00
1.98
2.93
2.02
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
Chemical Shift (ppm)

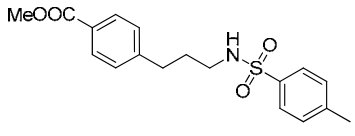

144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
Chemical Shift (ppm)



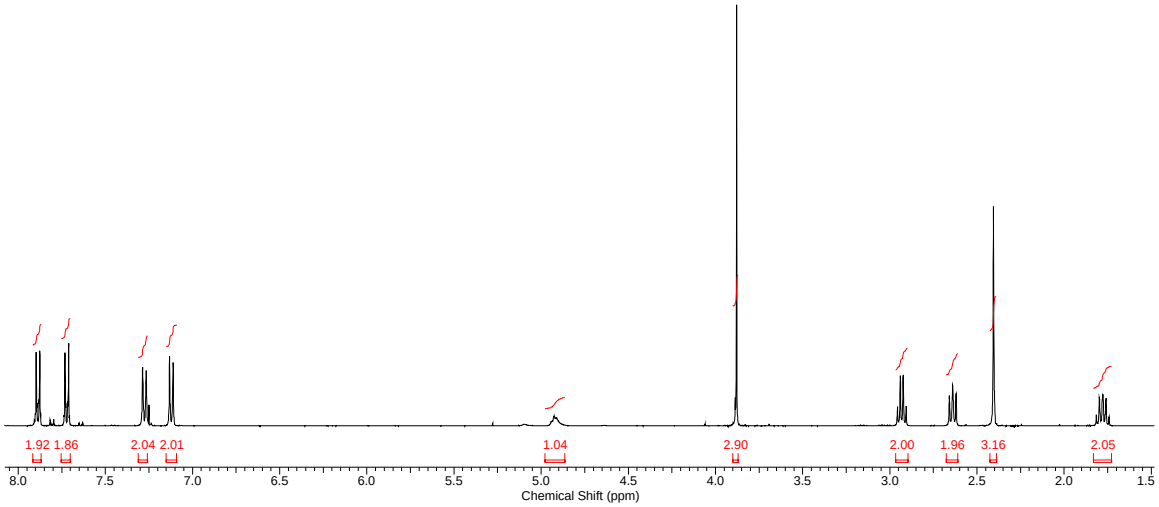
(2bc)

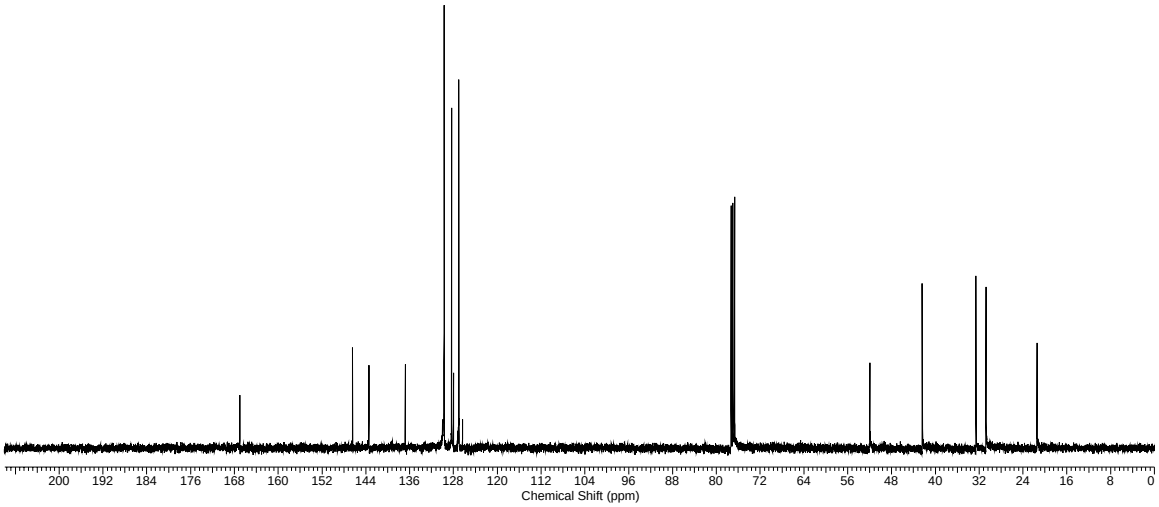

3.7
3.5
0.9
0.1
2.0
0.4
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

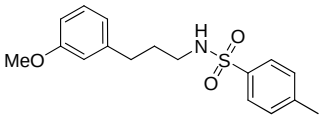

220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
ppm (t1)


MeOOC
H
N
S
O
O

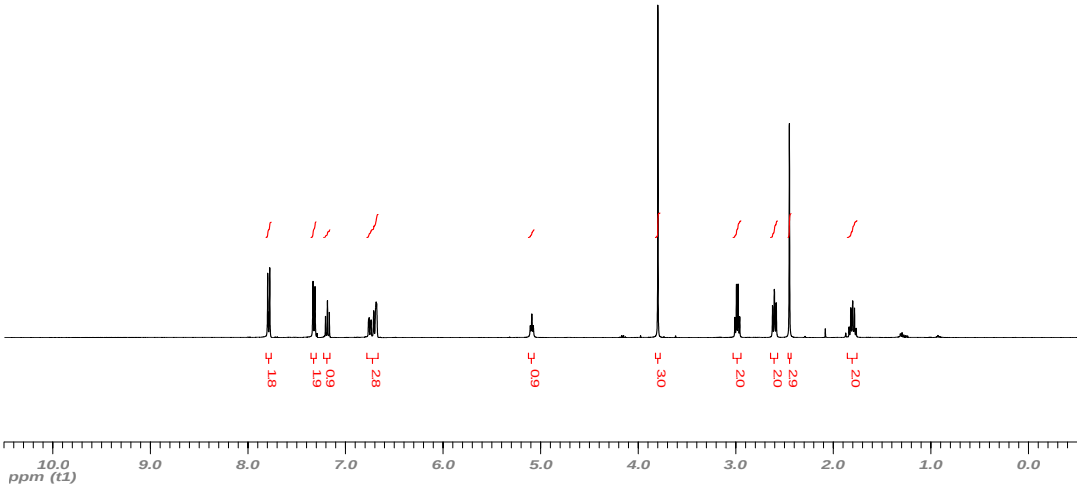
(2bd)

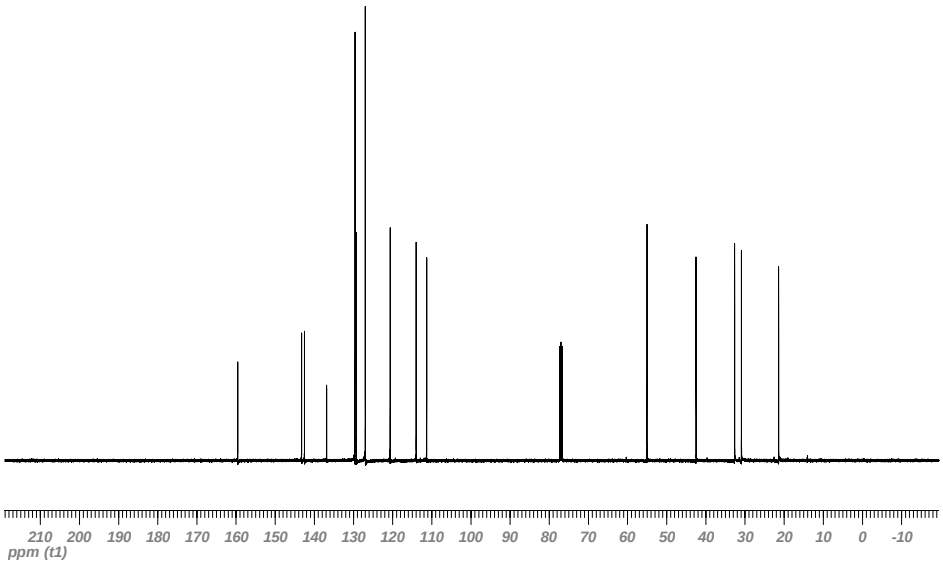

1.92 1.86 2.04 2.01 1.04 2.90 2.00 1.96 3.16 2.05
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5
Chemical Shift (ppm)

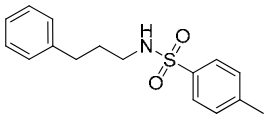

200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16 8 0
Chemical Shift (ppm)


MeO
H
N
O
S
O

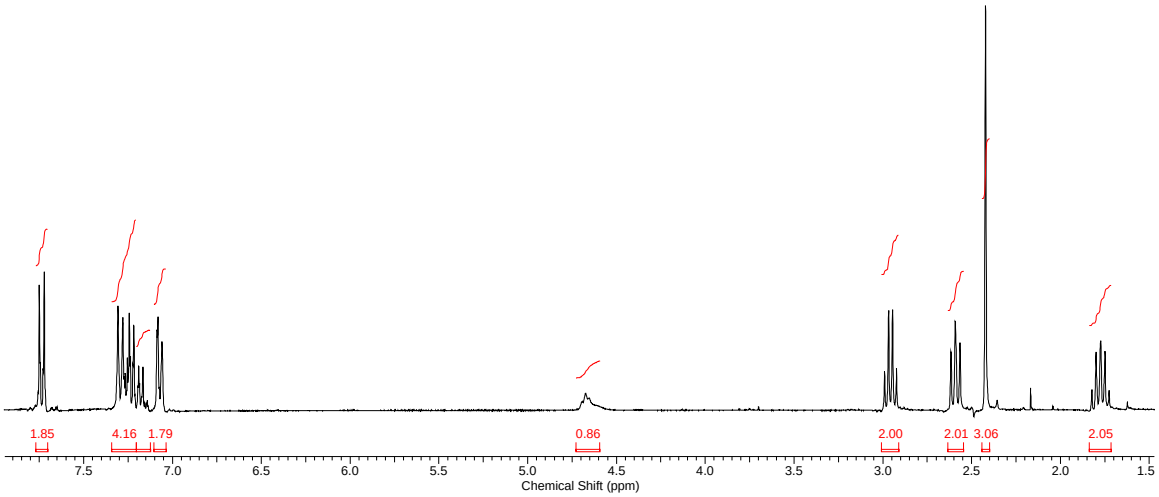
(2be)

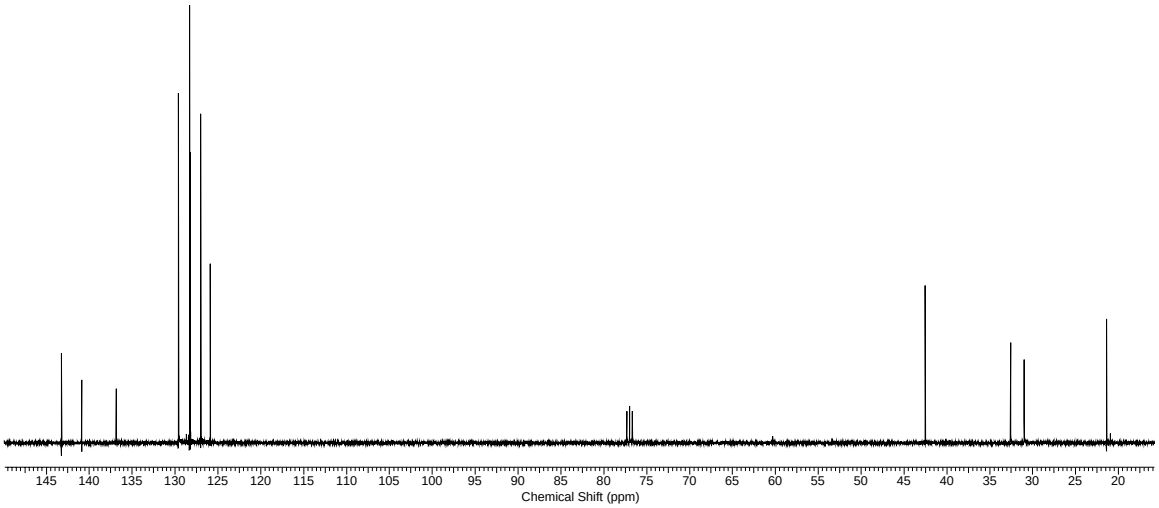

1.18
1.19
0.90
2.28
0.99
3.30
2.20
2.20
2.29
2.20
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

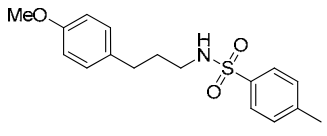

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
ppm (t1)



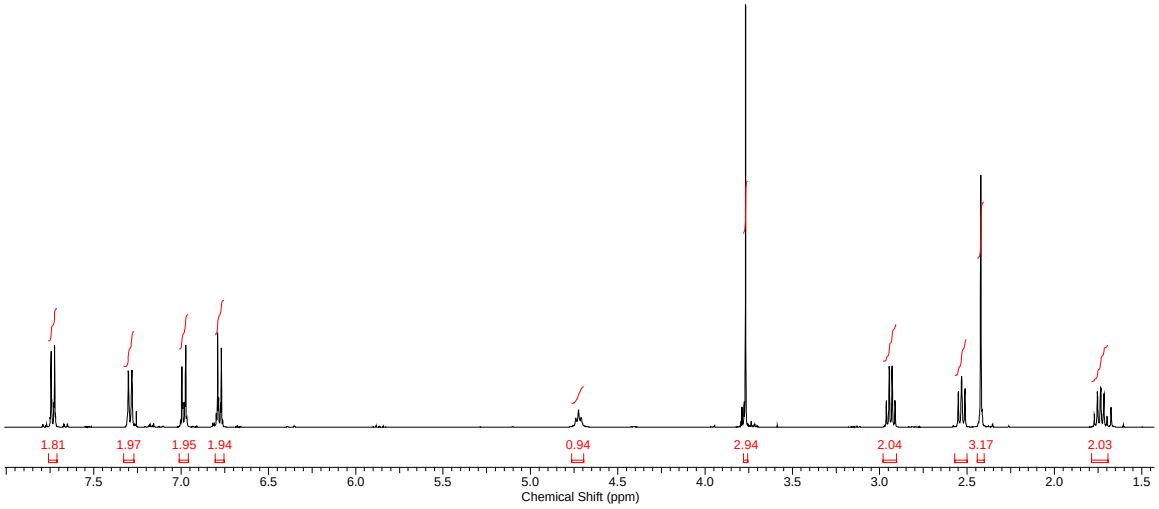
(2bf)

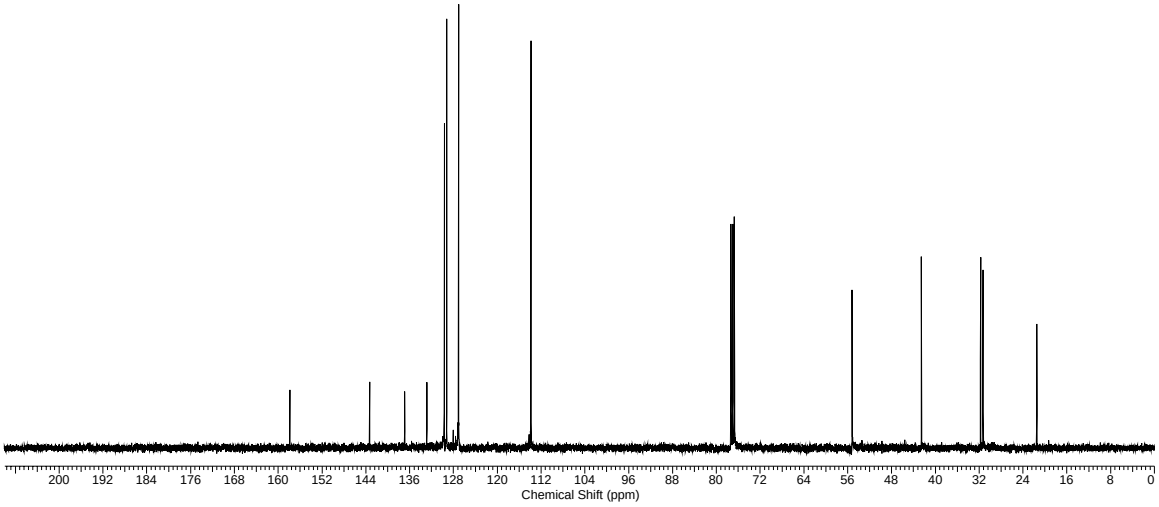

1.85
4.16
1.79
0.86
2.00
2.01
3.06
2.05
Chemical Shift (ppm)

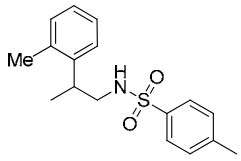

Chemical Shift (ppm)


MeO
H
N
S
O
O

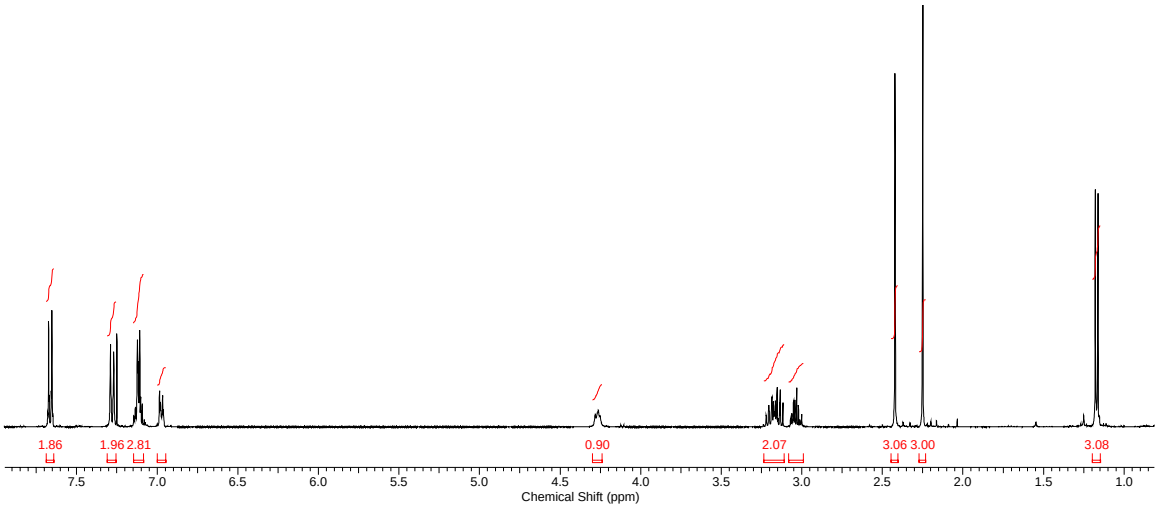
(2bg)

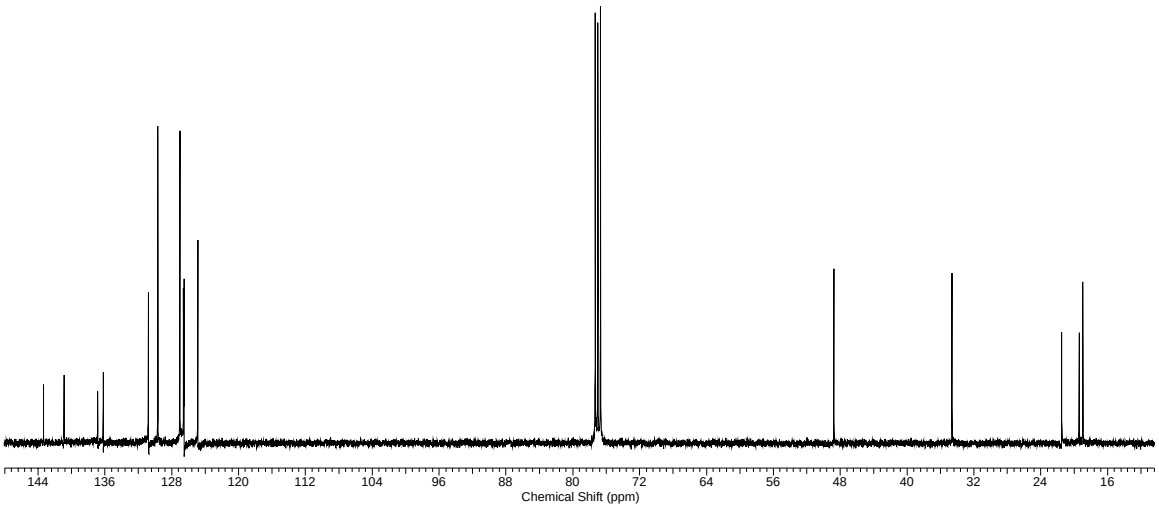

1.81
1.97
1.95
1.94
0.94
2.94
2.04
3.17
2.03
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
Chemical Shift (ppm)

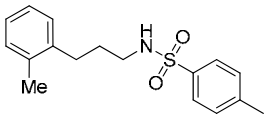

200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)



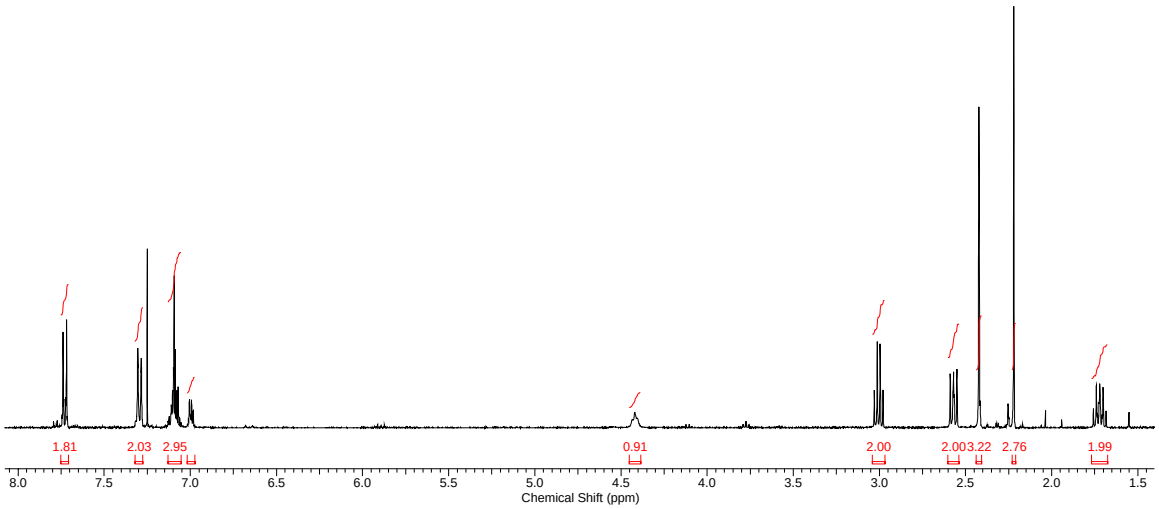
(2bh) Branched regioisomer

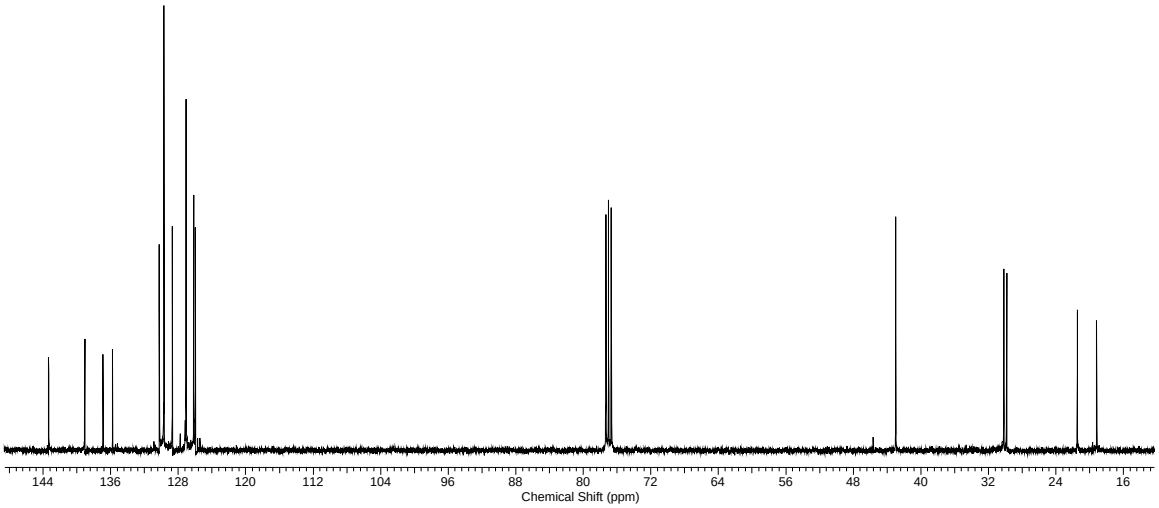


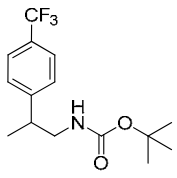




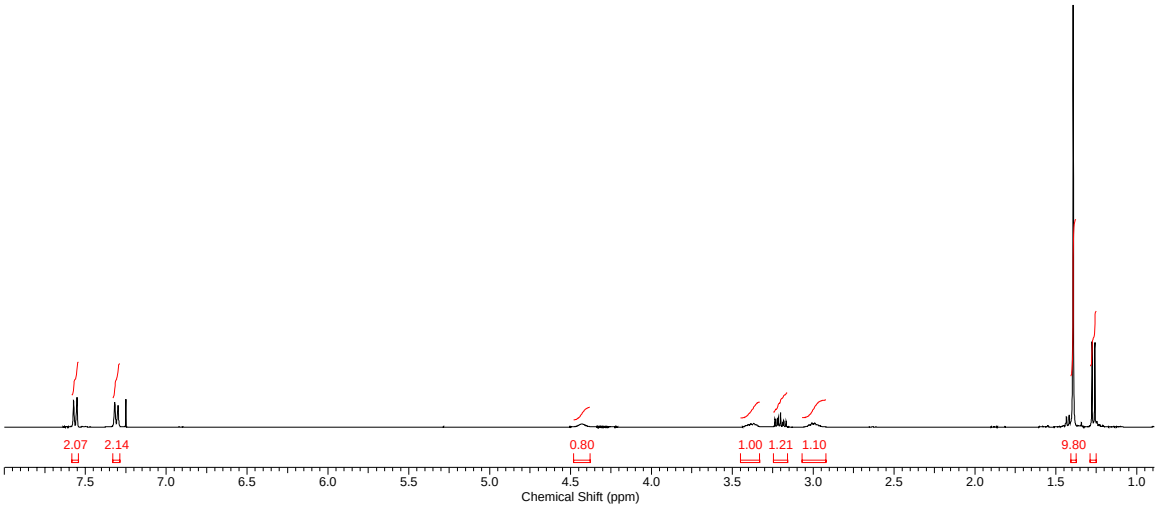
(2bh) Linear regioisomer

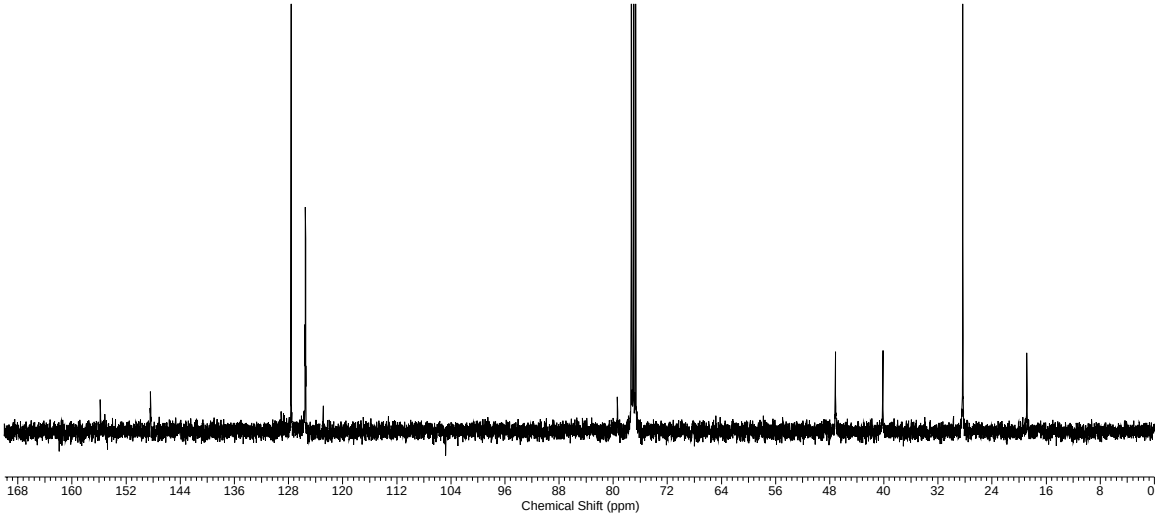


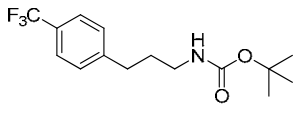




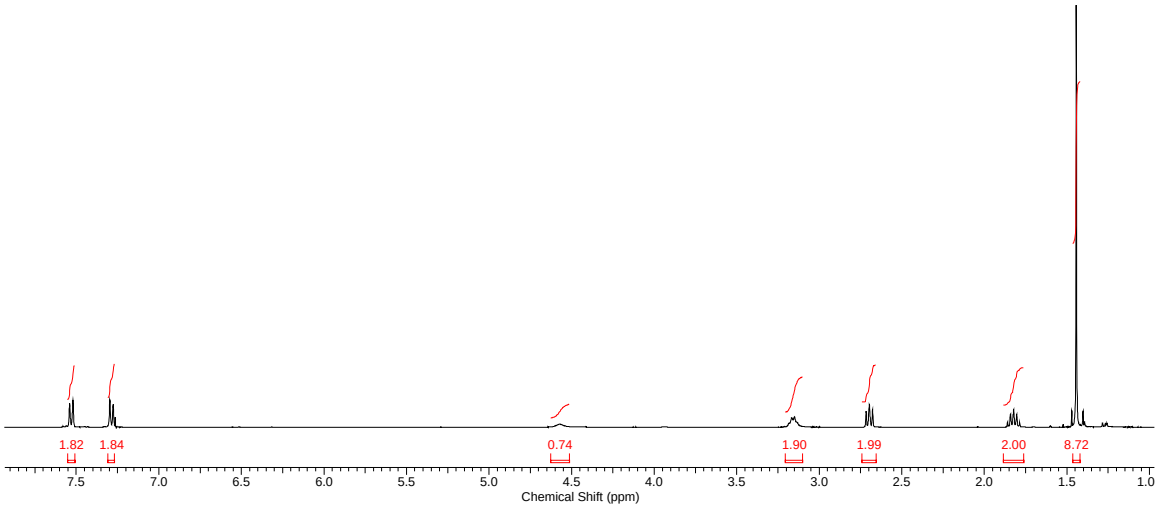
(2db) Branched regioisomer

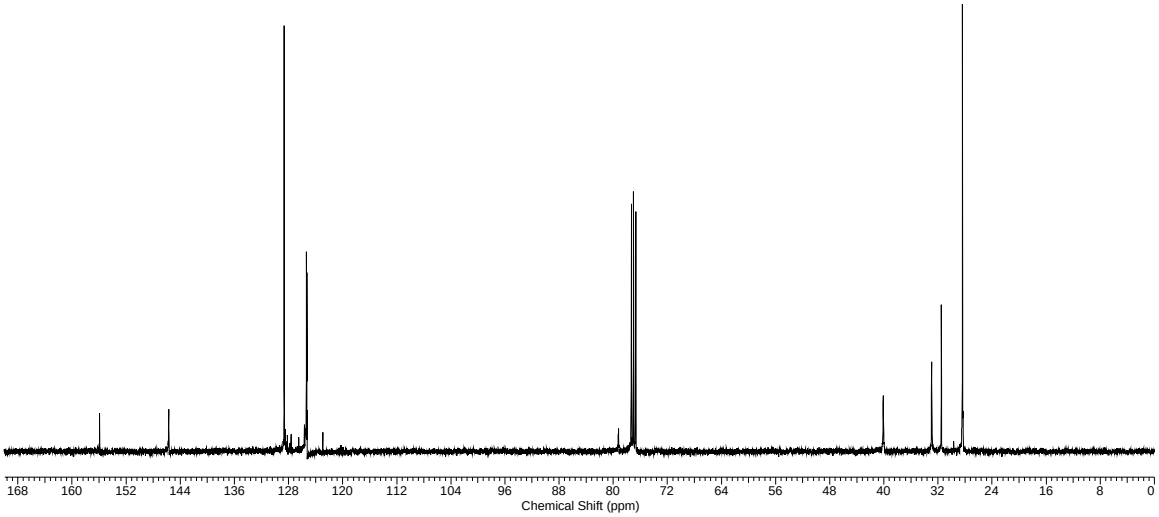


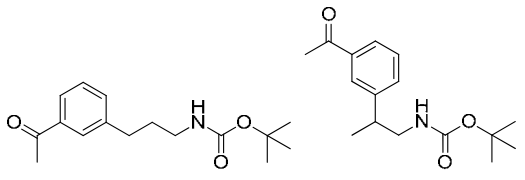




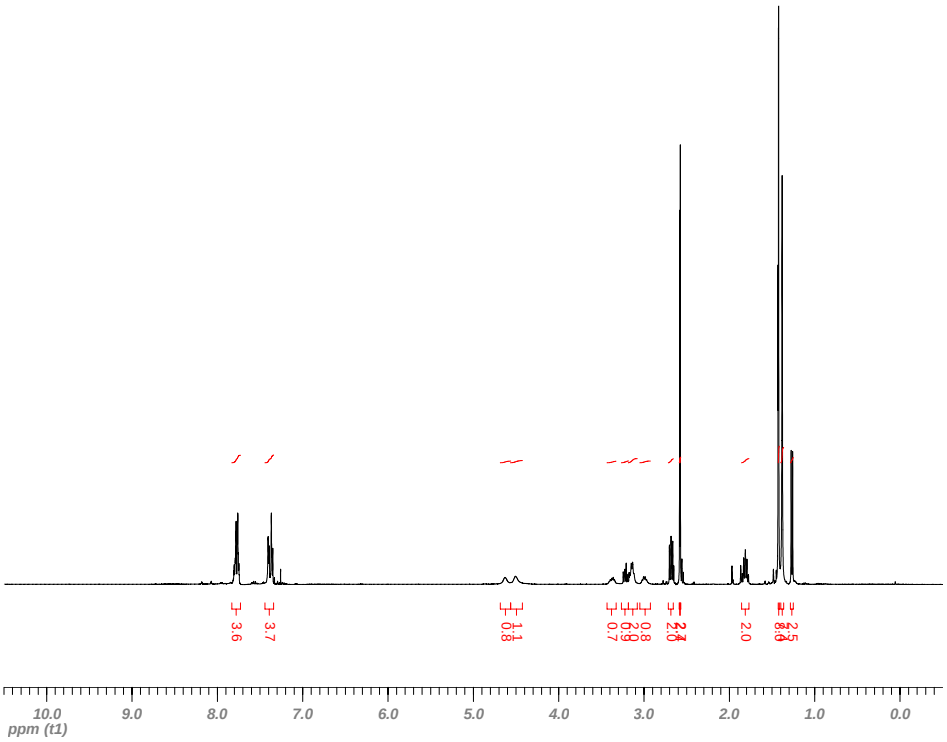
(2db) Linear regioisomer

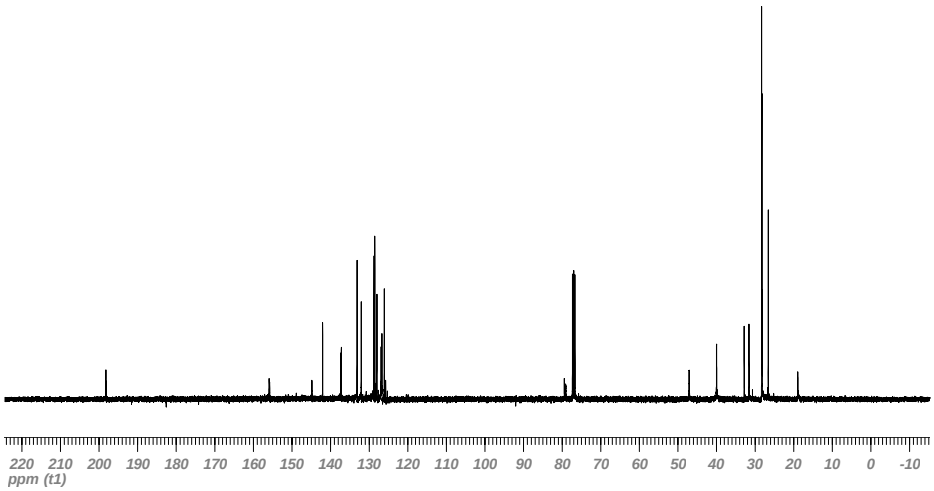


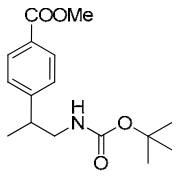




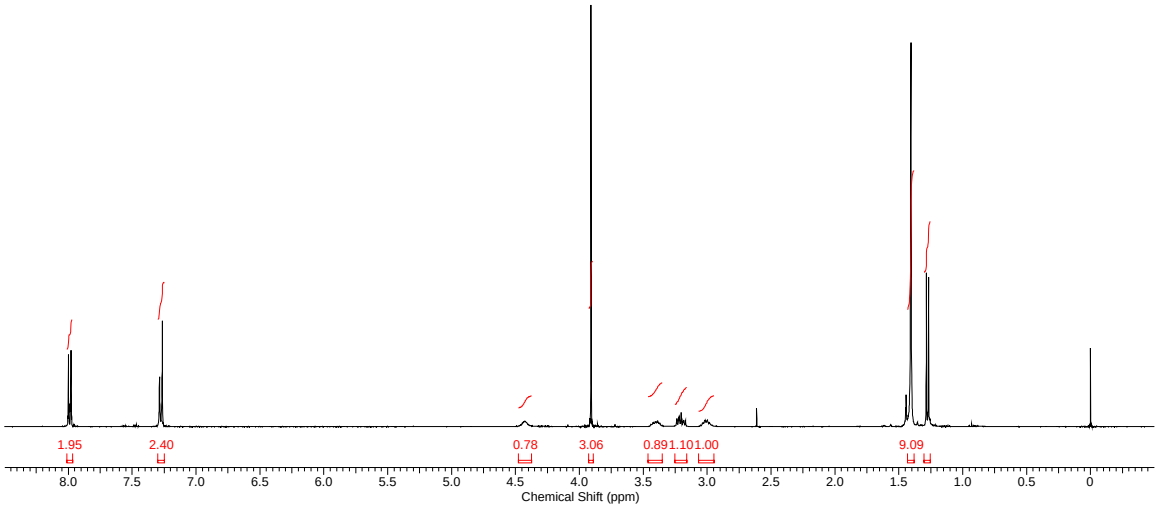
(2dc)

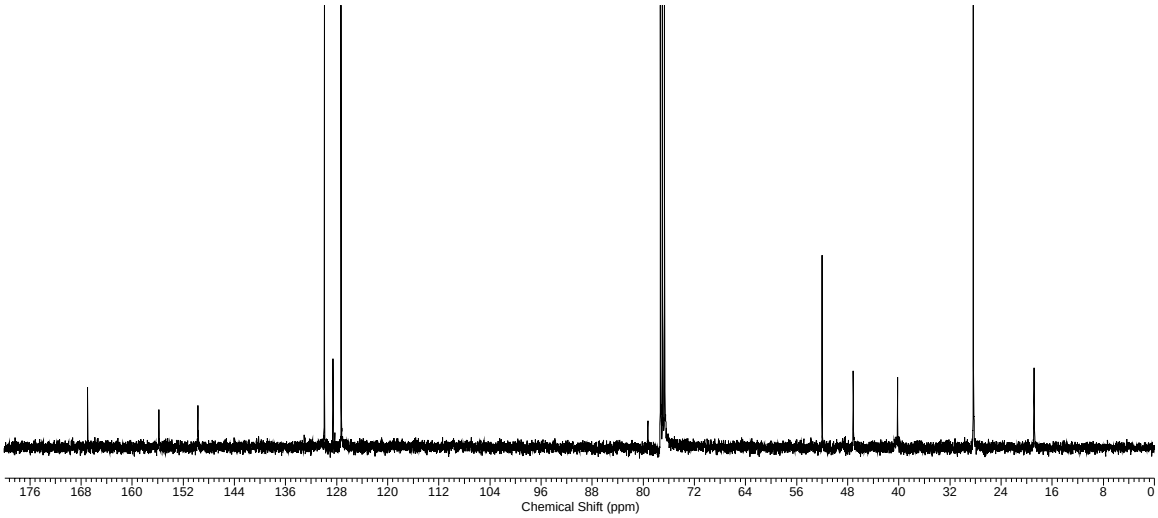

3.6
3.7
1.1
0.8
0.8
2.0
0.7
2.0
2.5
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

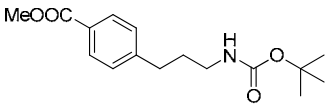

220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
ppm (t1)



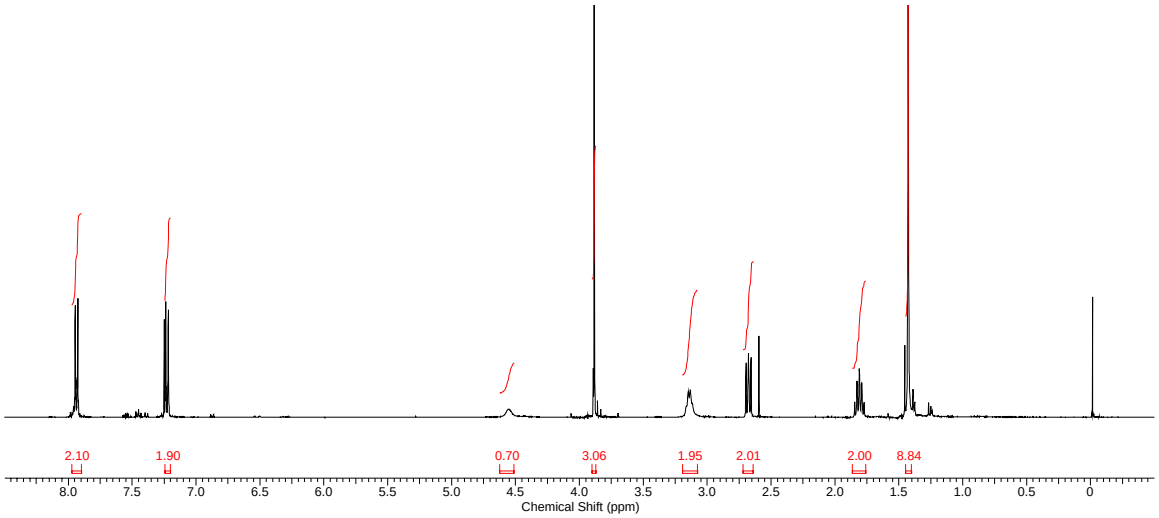
(2dd) Branched regioisomer

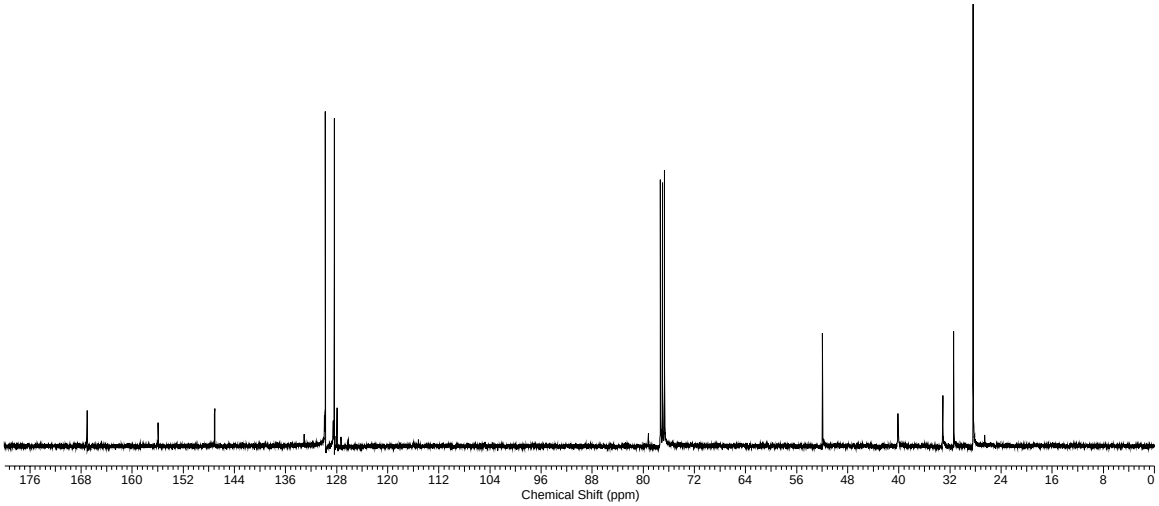


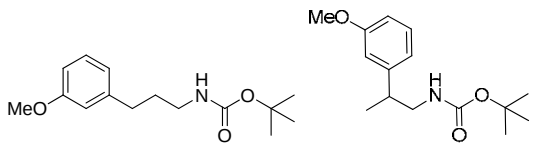




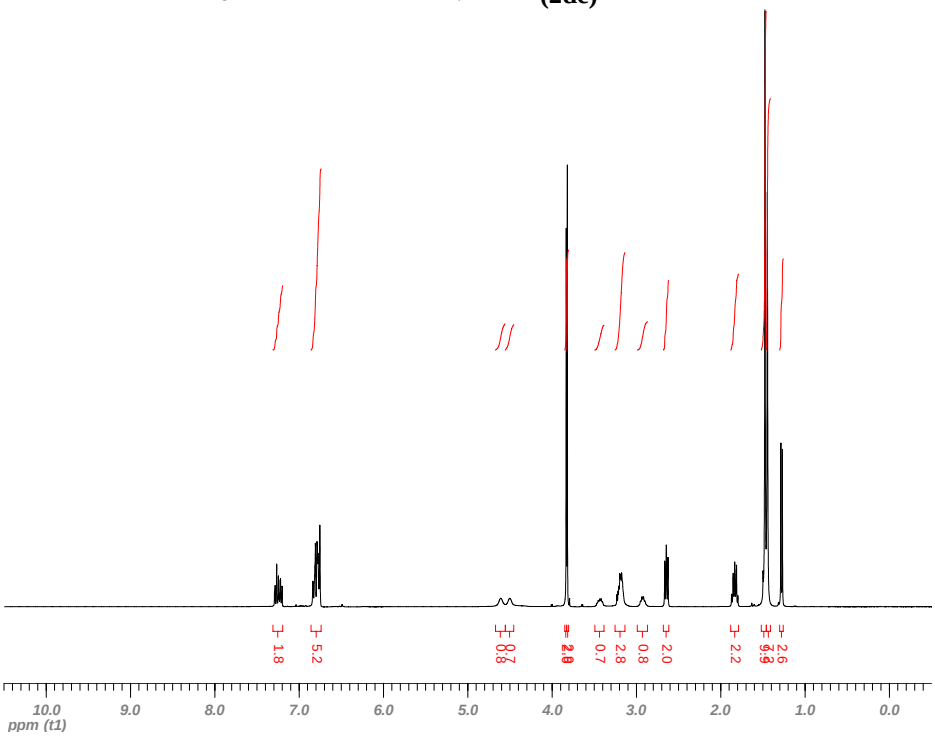
(2dd) Linear regioisomer

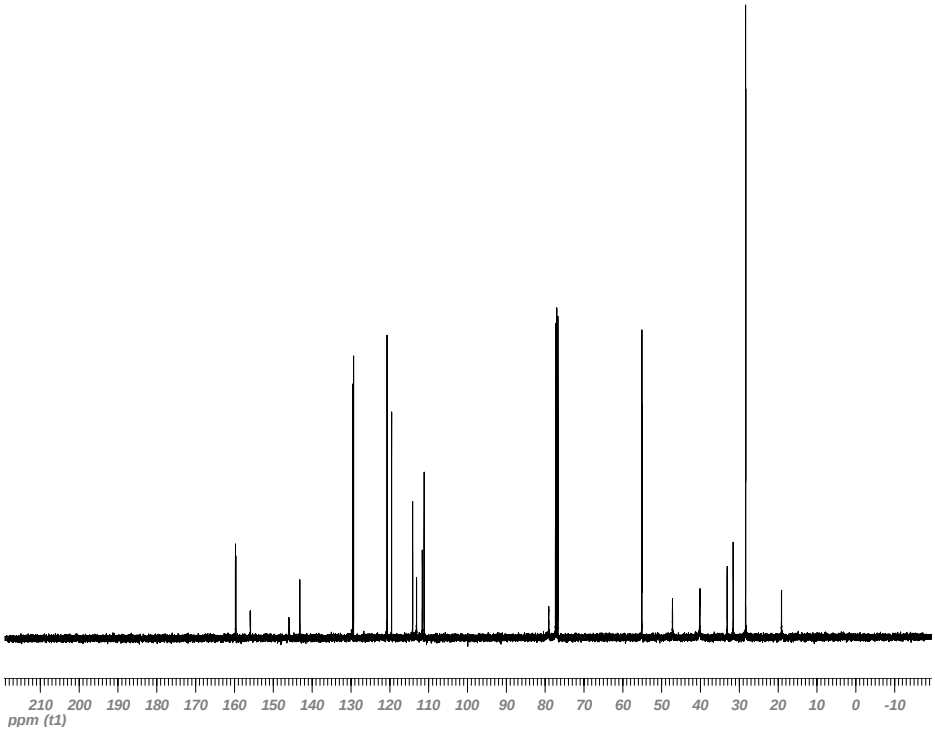


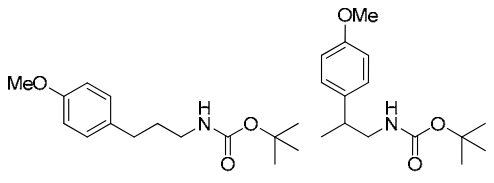



MeO
H
N
O
O
MeO
H
N
O
O

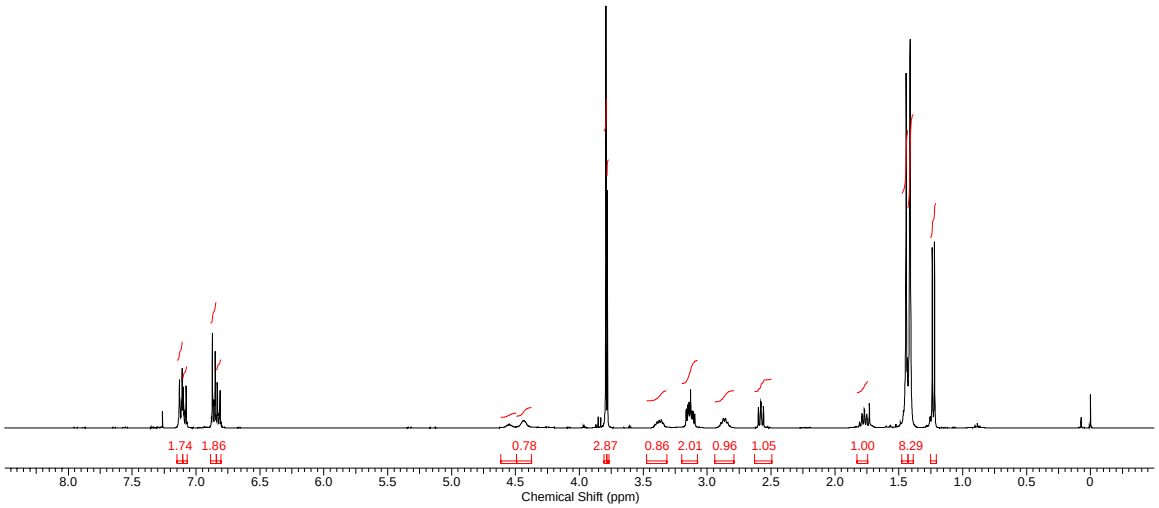
(2de)

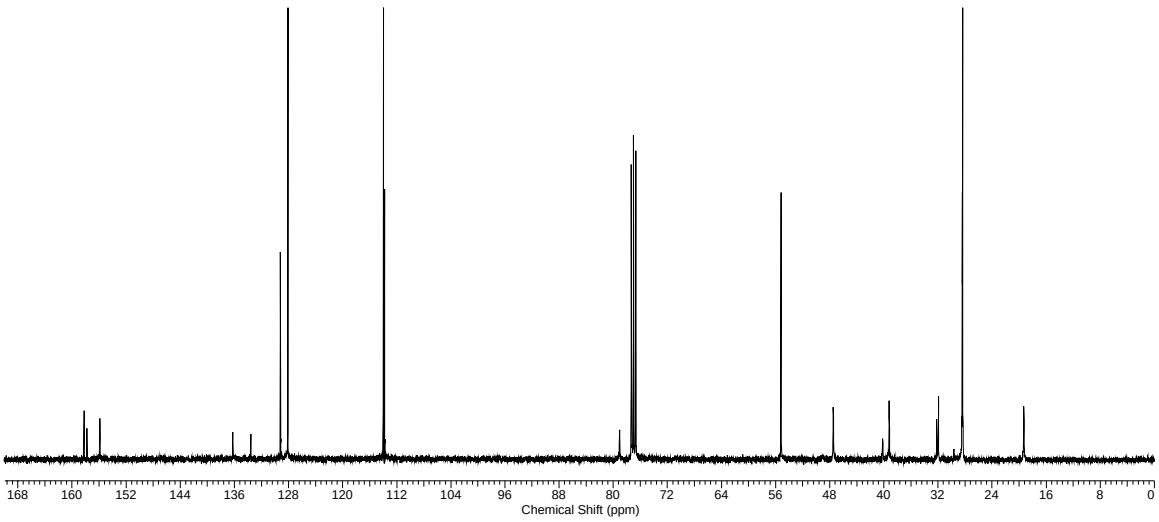

1.8
5.2
0.8
0.7
0.7
2.8
0.8
2.0
2.2
2.6
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

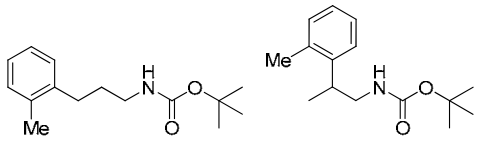

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
ppm (t1)


MeO
H
N
O
O
OMe
H
N
O
O

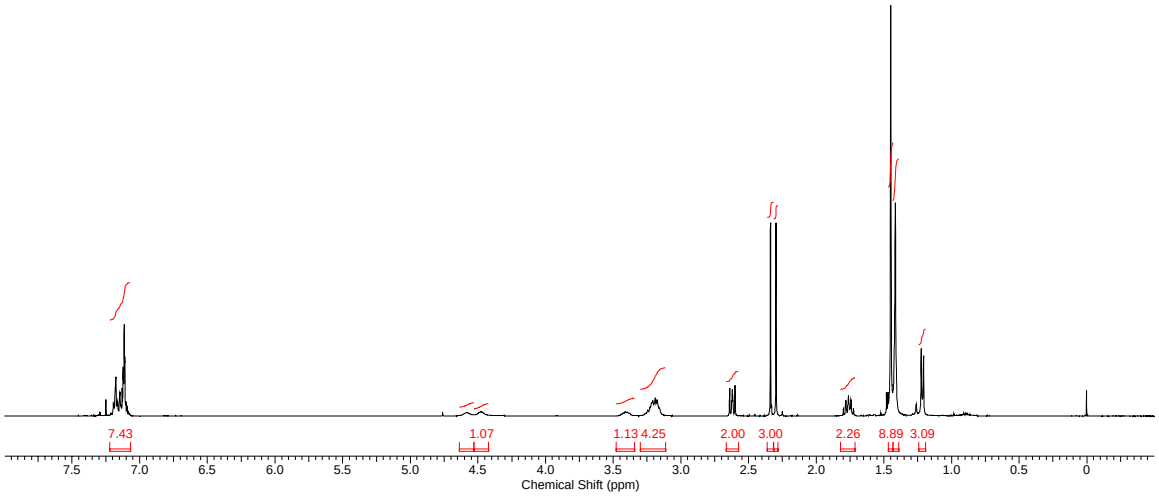
(2df)


1.74
1.86
0.78
2.87
0.86
2.01
0.96
1.05
1.00
8.29
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
Chemical Shift (ppm)


168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)


Me
H
N
O
O
Me
H
N
O
O

(2dg)


7.43
1.07
1.13
4.25
2.00
3.00
2.26
8.89
3.09
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
Chemical Shift (ppm)

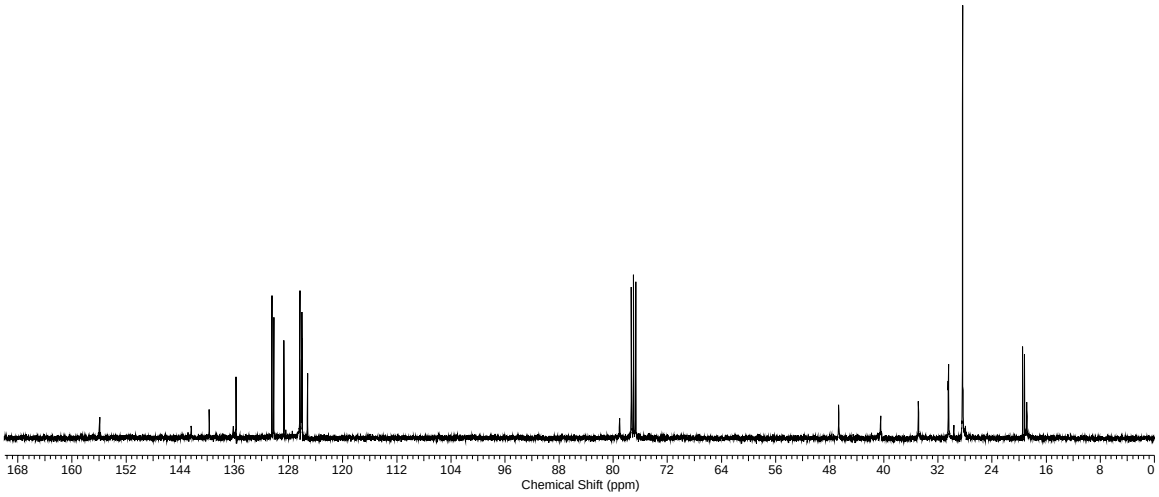
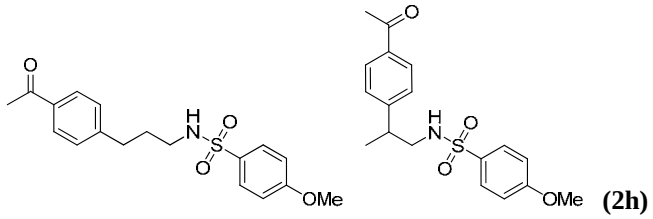
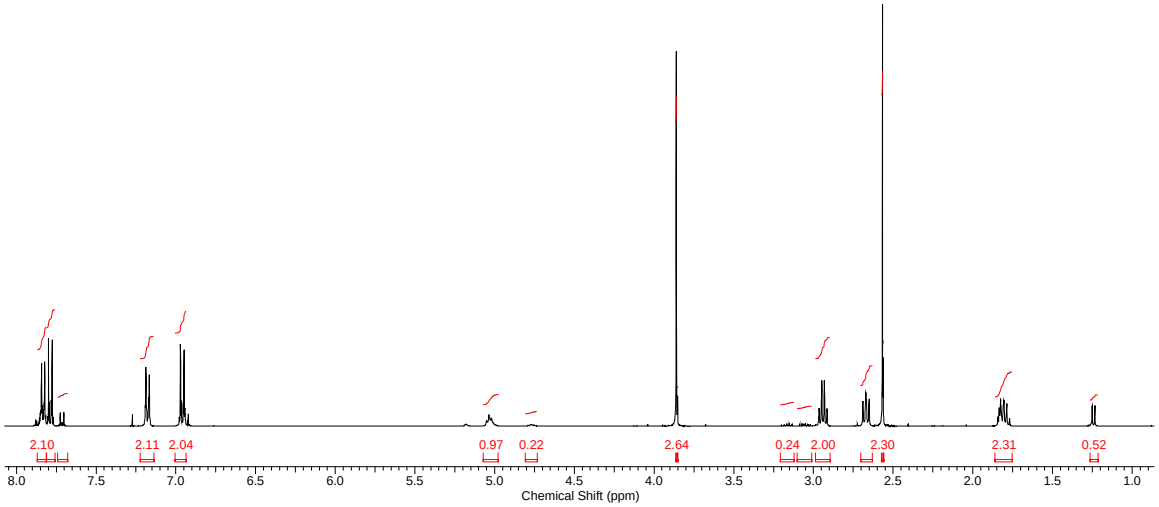
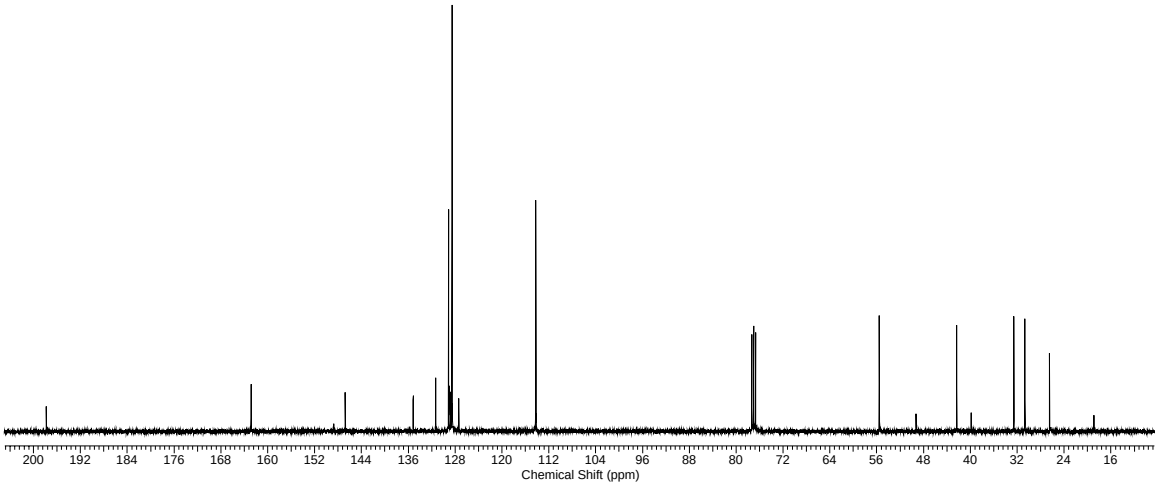

168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
0
Chemical Shift (ppm)

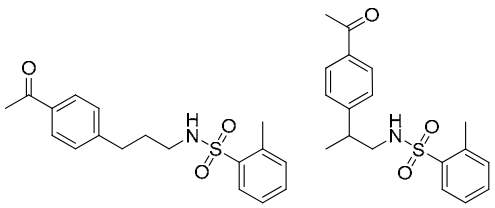
Table 3:



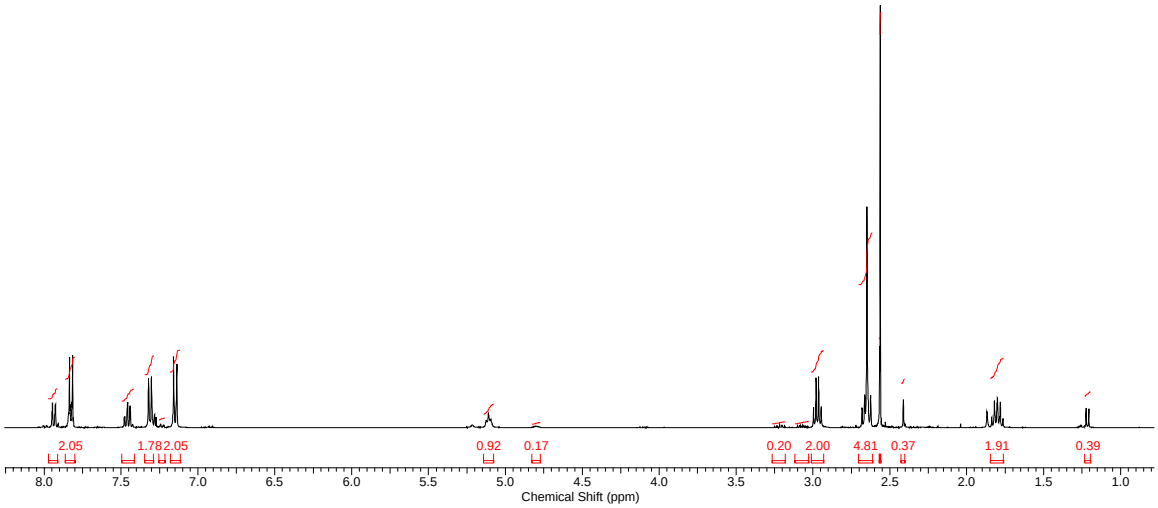
(2h)

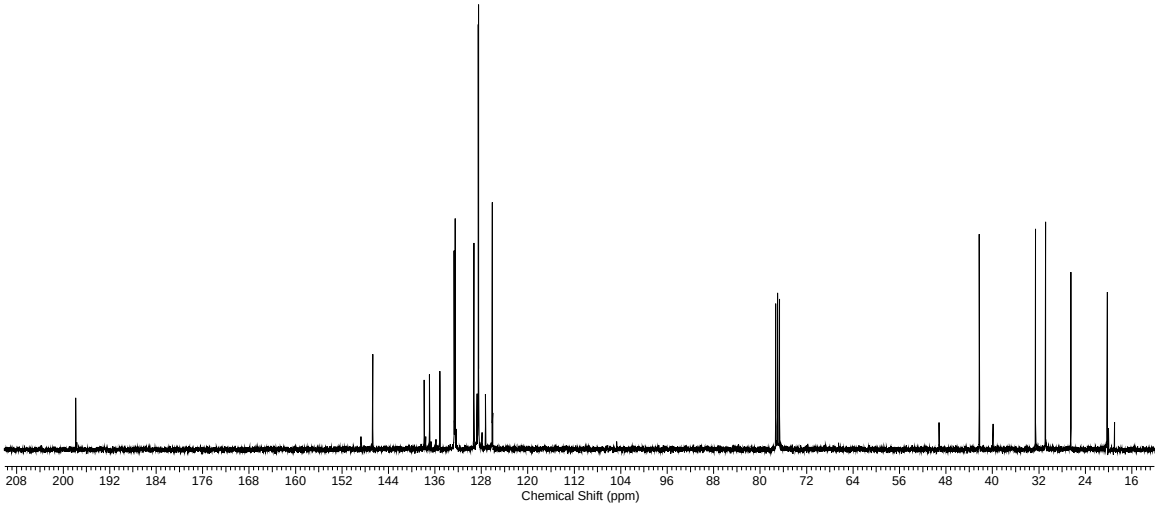

2.10
2.11
2.04
0.97
0.22
2.64
0.24
2.00
2.30
2.31
0.52
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
Chemical Shift (ppm)

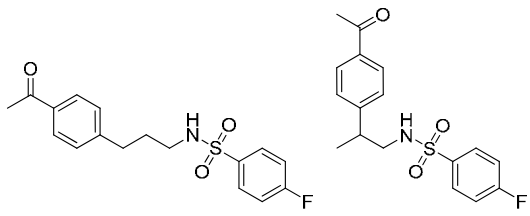

200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
Chemical Shift (ppm)



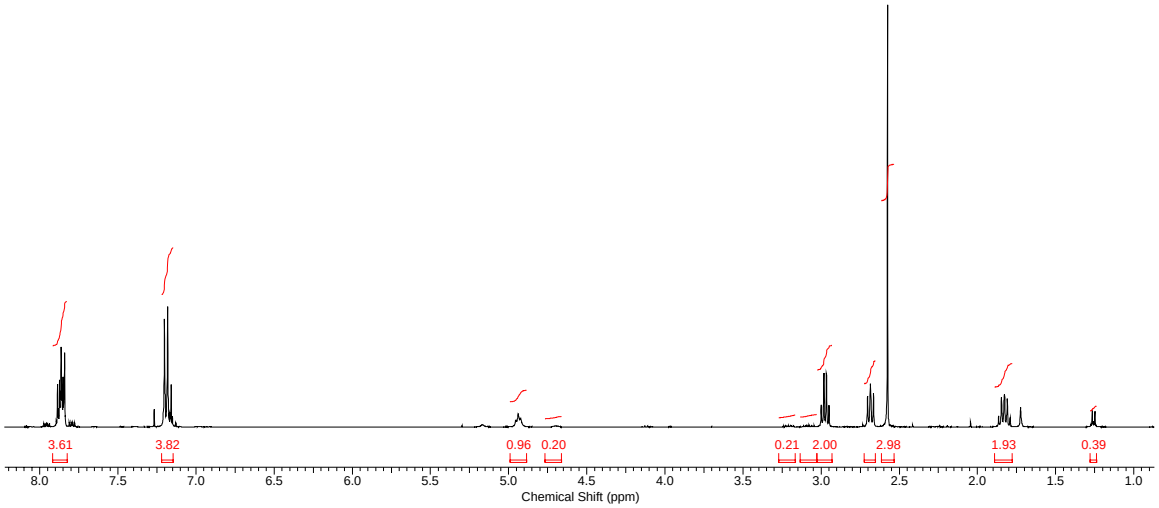
(2i)

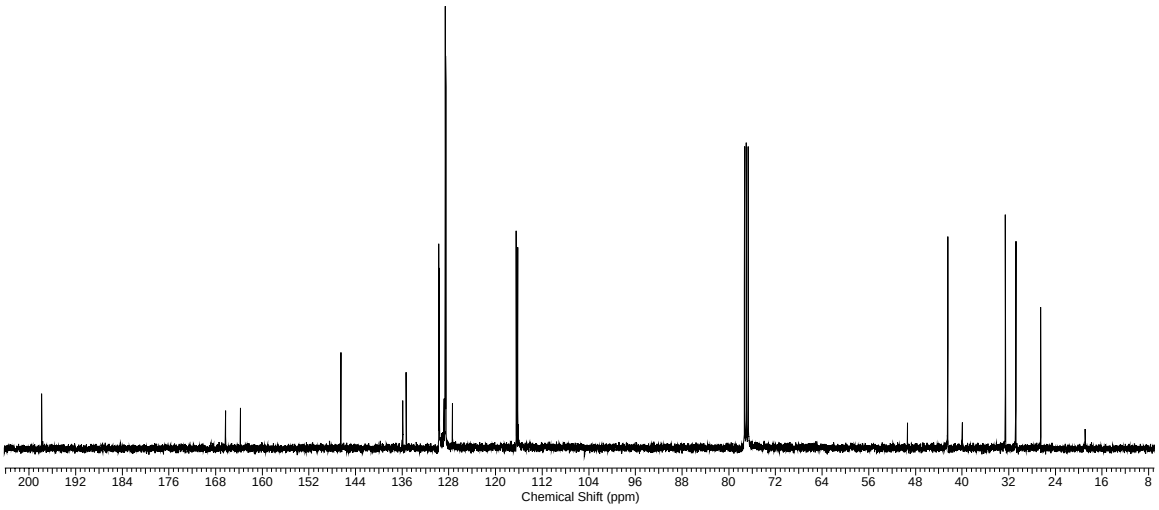

2.05
1.78
2.05
0.92
0.17
0.20
2.00
4.81
0.37
1.91
0.39
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
Chemical Shift (ppm)

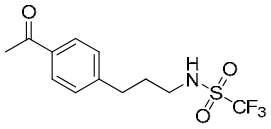

208
200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
Chemical Shift (ppm)



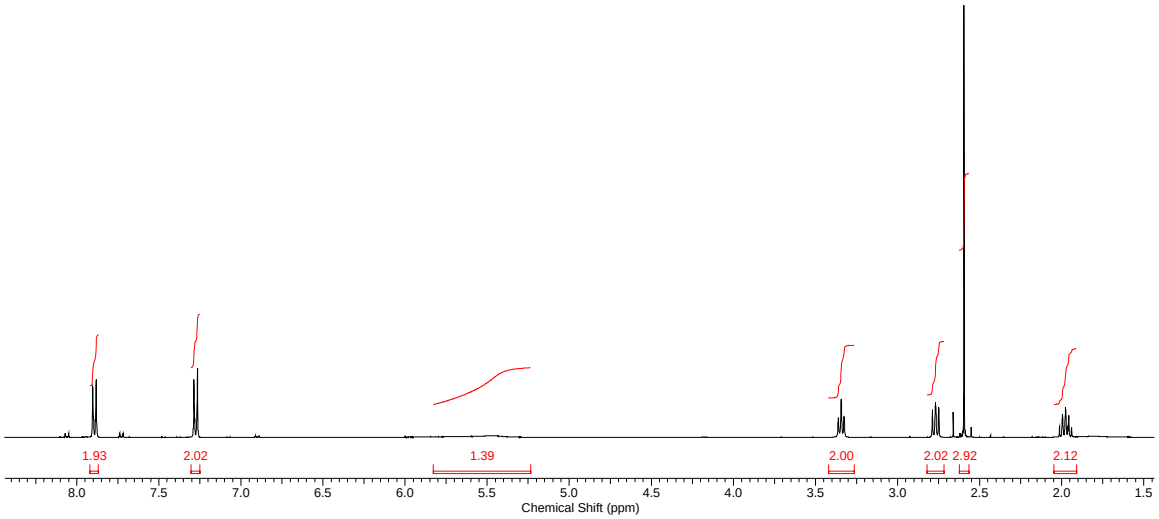
(2j)

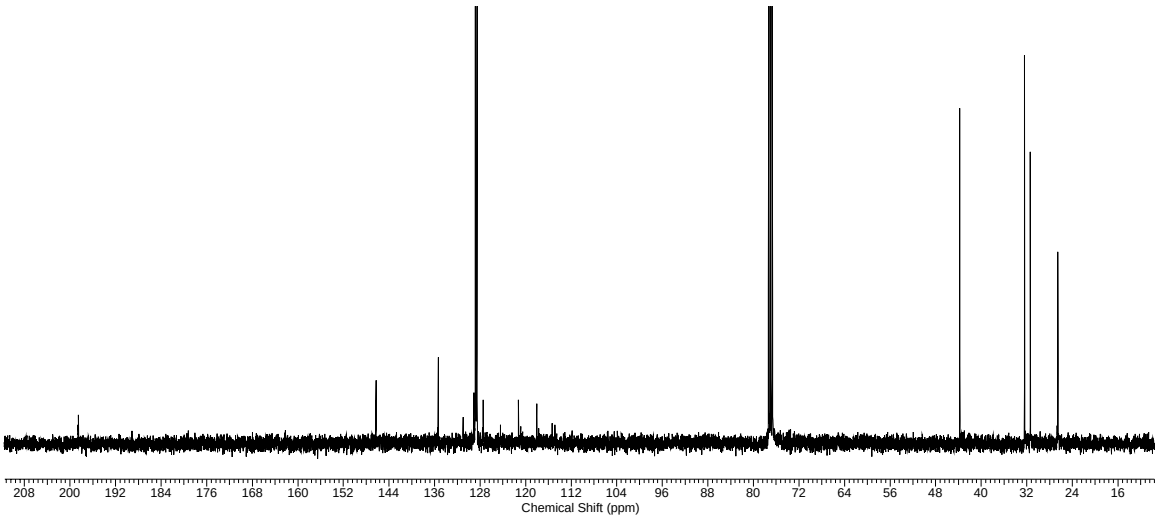

3.61
3.82
0.96
0.20
0.21
2.00
2.98
1.93
0.39
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
Chemical Shift (ppm)


200
192
184
176
168
160
152
144
136
128
120
112
104
96
88
80
72
64
56
48
40
32
24
16
8
Chemical Shift (ppm)

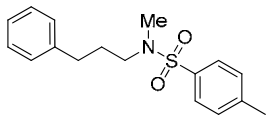


(2k)

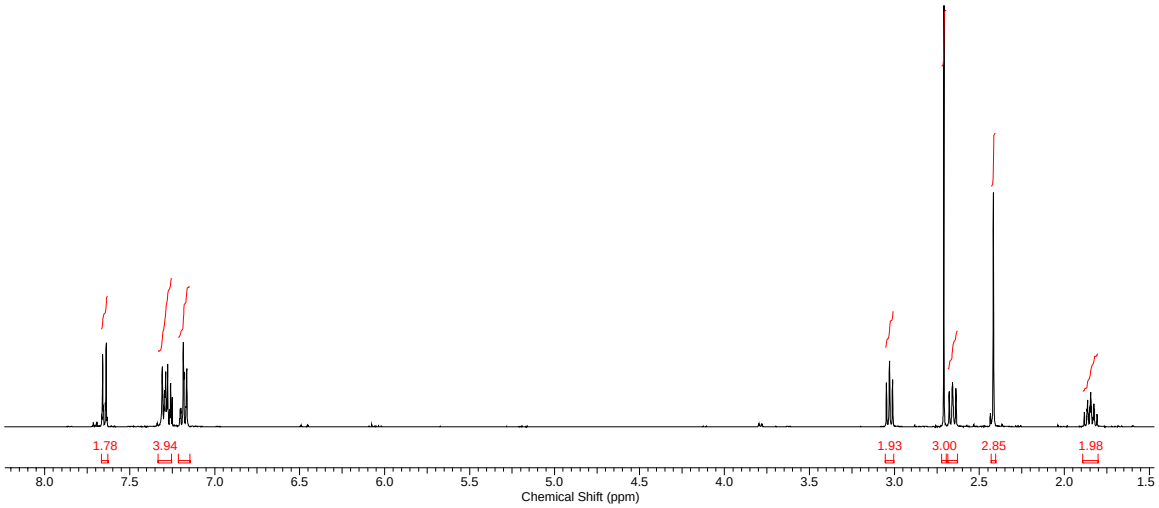

1.93
2.02
1.39
2.00
2.02
2.92
2.12
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5
Chemical Shift (ppm)


208 200 192 184 176 168 160 152 144 136 128 120 112 104 96 88 80 72 64 56 48 40 32 24 16
Chemical Shift (ppm)

Equation 3:



(2l)



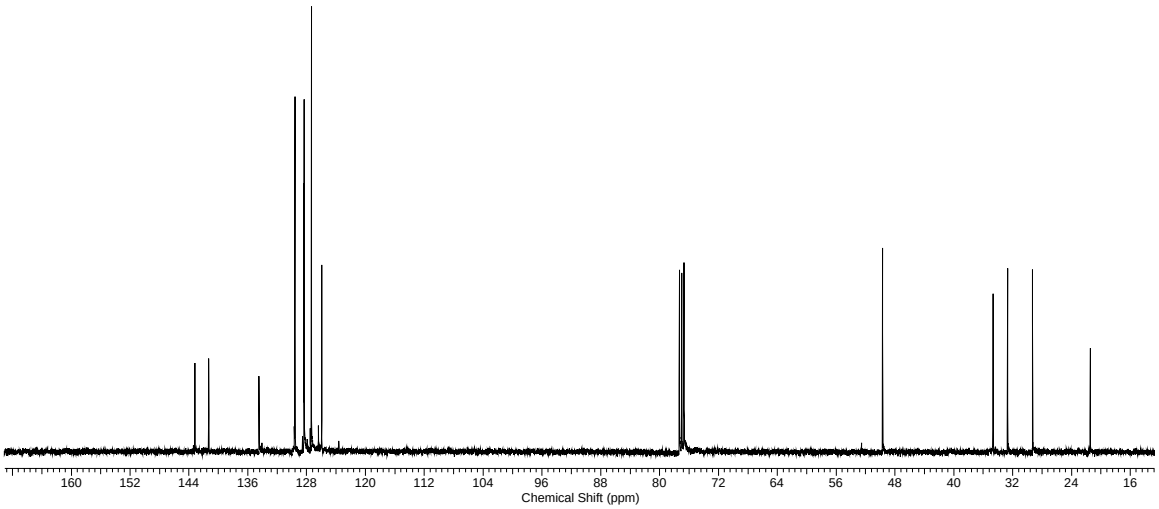
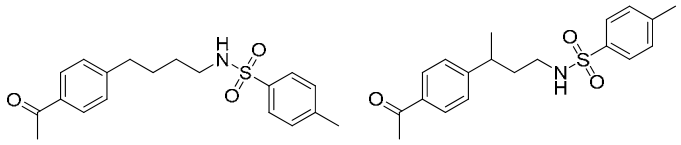
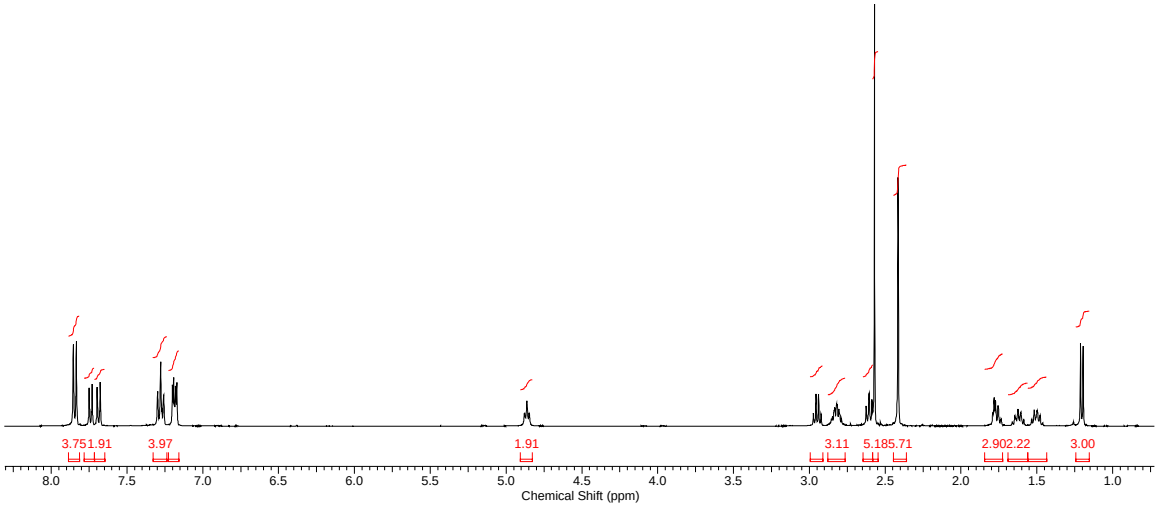
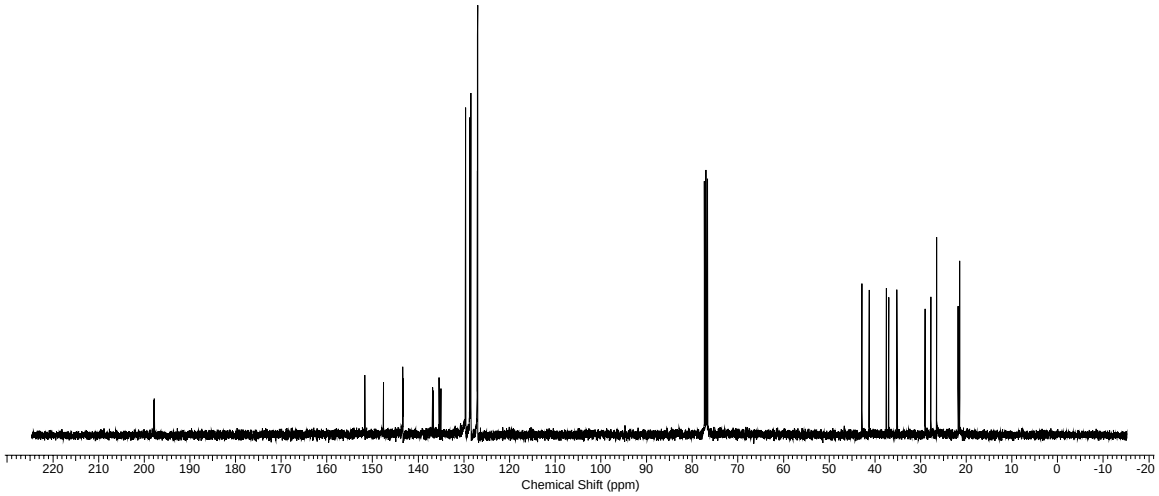


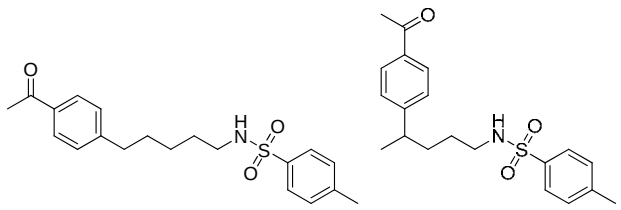
Table 4:



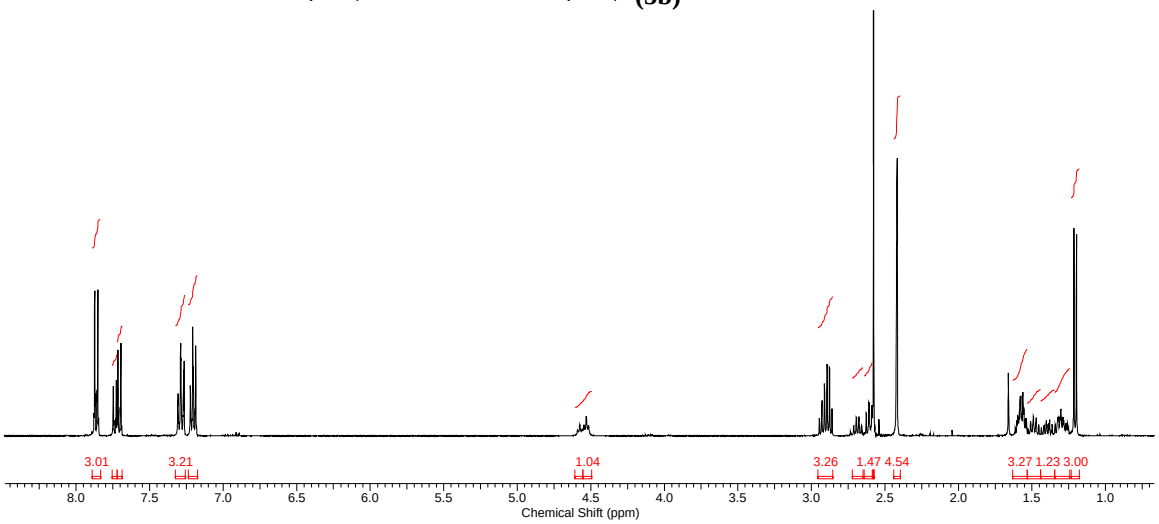
(5a)

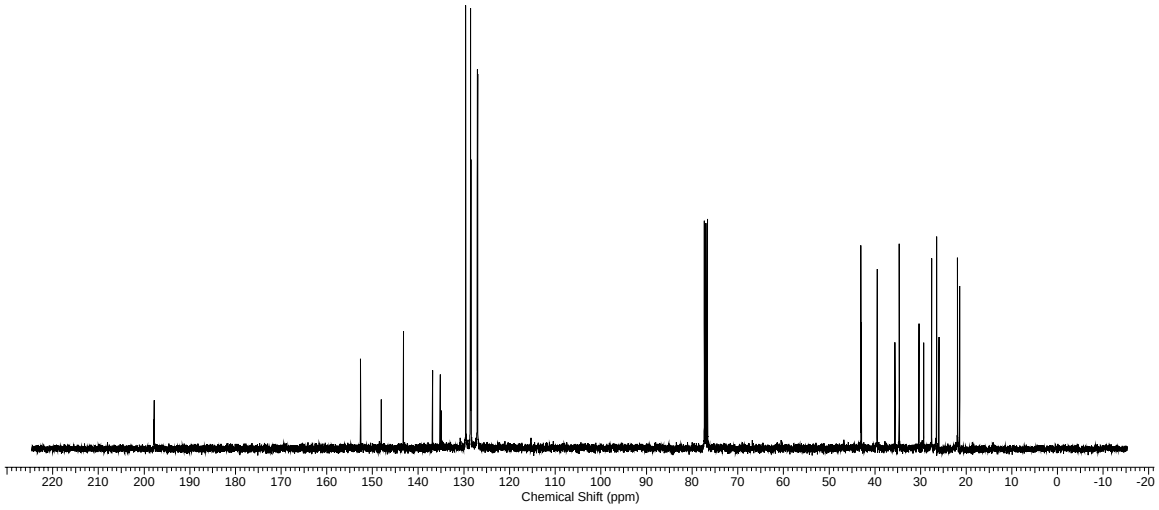

3.751.91
3.97
1.91
3.11
5.185.71
2.902.22
3.00
Chemical Shift (ppm)

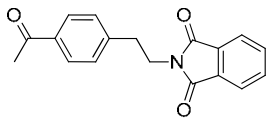

Chemical Shift (ppm)



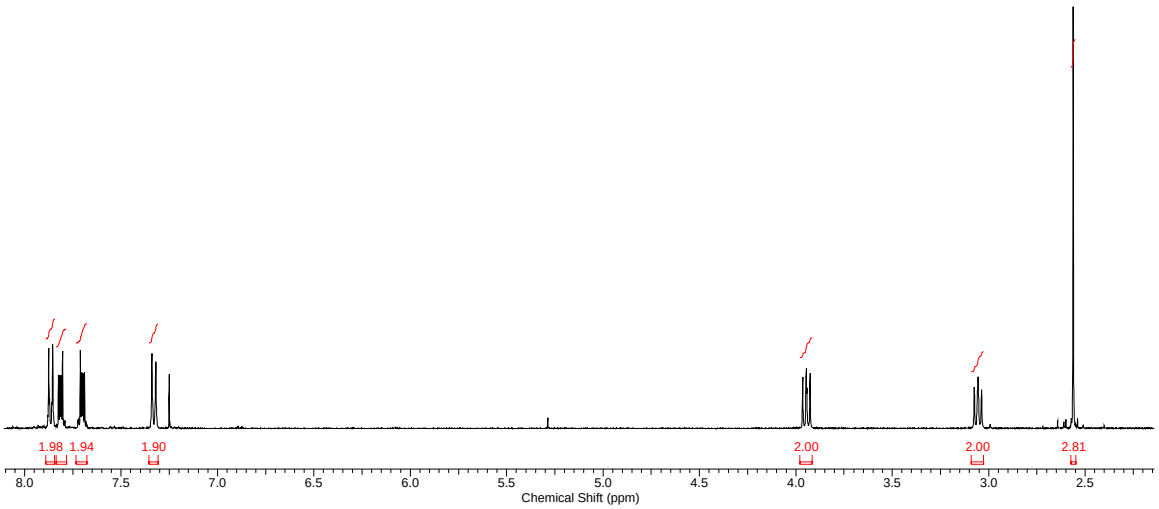
(5b)

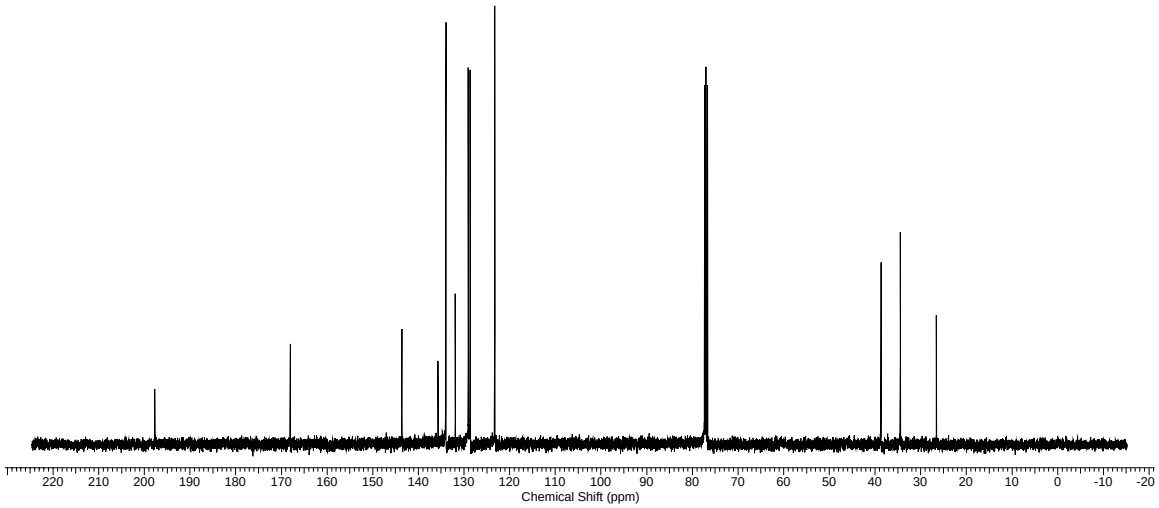

3.01
3.21
1.04
3.26
1.47
4.54
3.27
1.23
3.00
Chemical Shift (ppm)


Chemical Shift (ppm)



(5c)


1.98
1.94
1.90
2.00
2.00
2.81
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
Chemical Shift (ppm)


220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
Chemical Shift (ppm)