Electronic Supplementary Information

Light-Mediated Cascade Transformation of Activated Alkenes: BiOBr Nanosheets as Efficient Photocatalysts for the synthesis of α-aryl-β-trifluoromethyl amides

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1. General Information:

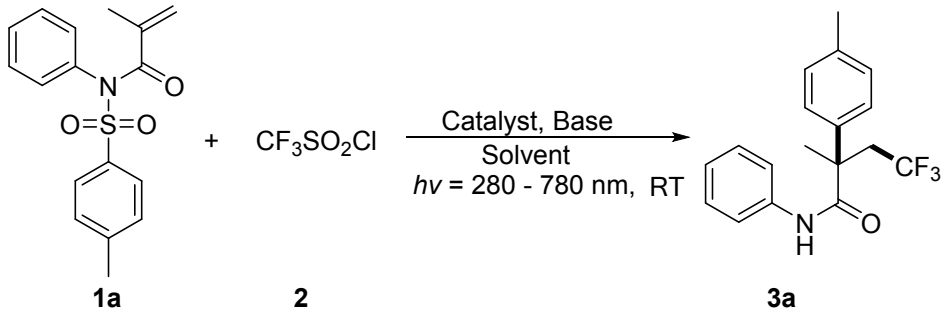
Materials

All purchased reagents were used without further purification. Analytical thin layer chromatography was performed on 0.20 mm Qingdao Haiyang silica gel plates. Silica gel (200-300 mesh) (from Qingdao Haiyang Chem. Company, Ltd.) was used for flash chromatography.

Characterization

The scanning electron microscopy (SEM) images were taken with a Hitachi S-4800 scanning electron microscope (SEM, 5 kV) equipped with the Thermo Scientific energy-dispersion X-ray fluorescence analyzer. Transmission electron microscopy (TEM) and higher-magnification transmission electron microscopy (HRTEM) were carried out with JEOL JEM-2100F system equipped with the EDAX Genesis XM2. The X-ray diffraction patterns (XRD) of the products were recorded with a PANalytical X'Pert PRO X-ray diffractometer with Co Kα radiation (λ = 1.78897 nm) at 40 kV and 40 mA. Then the data was converted into corresponding Cu Kα radiation. The optical properties of the samples were analyzed by a Shimadzu UV-2550 ultraviolet/visible diffuse reflectance spectroscopy (DRS), during which $BaSO_4$ was employed as the internal reflectance standard. ^{1}H, ^{13}C, ^{19}F NMR were recorded on Varian Mercury Plus 400 instruments at 400 MHz (^{1}H NMR), 100 MHz (^{13}C NMR), as well as 376 MHz (^{19}F NMR). Chemical shifts were reported in ppm down field from internal Me_4Si and external CCl_3F, respectively. Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (doublet of doublet), br (broad). Coupling constants were reported in Hertz (Hz). HRMS were recorded on an Agilent Q-TOF spectrometer using the ESI method. IR spectra were recorded on an AVATAR 360 FT-IR spectrometer.

2. Table S1. Optimization of the Reaction Conditions[a]



1a **2** **3a**

Entry	Catalyst	Base	Solvent	Yield (%)
1[b]	BiOBr nanosheets	K_2HPO_4	DMF	74
2[b]	BiOBr nanosheets	K_2HPO_4	DMAC	79
3	BiOBr nanosheets	K_2HPO_4	DMAC	75
4	BiOBr nanosheets	K_2HPO_4	DMSO	-
5	BiOBr nanosheets	K_2HPO_4	NMP	51
6	BiOBr nanosheets	K_2HPO_4	H_2O	-
7	BiOBr nanosheets	K_2HPO_4	CH_3OH	-
8	BiOBr nanosheets	K_2HPO_4	CH_2Cl_2	-
9	BiOBr nanosheets	K_2HPO_4	Toluene	-
10	BiOBr nanosheets	K_2HPO_4	THF	Trace
11[c]	BiOBr nanosheets	K_2HPO_4	DMAC	-
12	-	K_2HPO_4	DMAC	-
13	CdS	Et*i*Pr_2	DMAC	-
14	$Ru(bpy)_3Cl_2.6H_2O$	Et*i*Pr_2	DMAC	-

[a] Reaction conditions: **1a** (0.2 mmol), **2** CF_3SO_2Cl (1.0 mmol), base (0.6 mmol), catalysts (20 mg), solvent (1.0 mL), 300 W Xe arclamp, RT. [b] 0.4 mmol $HSiEt_3$ is added. [c] In the dark.

3. Synthesis and characterizations of BiOBr nanosheets

The procedure was proceed according with the our previous work.[15]

Typically, 0.97 g $Bi(NO_3)_3 \cdot 5H_2O$ was dissolved in 3.0 ml acetic acid, and the resulting solution was added to 30 ml de-ionized water containing 0.24 g KBr under vigorous stirring. Upon the adding of $Bi(NO_3)_3$/HAc solution, yellow precipitates were immediately observed which then became light yellow as addition was completed. After stirring for 20 min at room temperature, the suspension was transferred into a Teflon-lined stainless steel autoclave (40 ml capacity) and heated at 120 ℃ for 6.0 h. The resulting precipitate was filtrated, washed thoroughly with distilled water to remove any possible ionic species in the product, and then dried at 60 ℃ overnight.

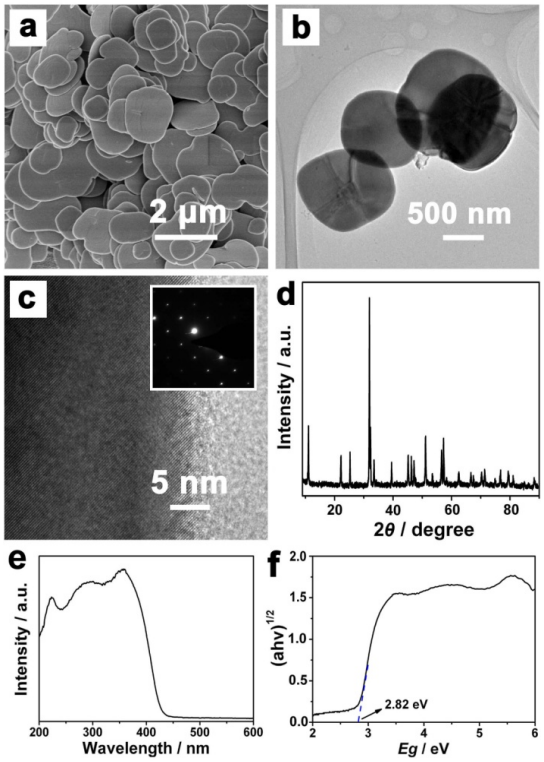
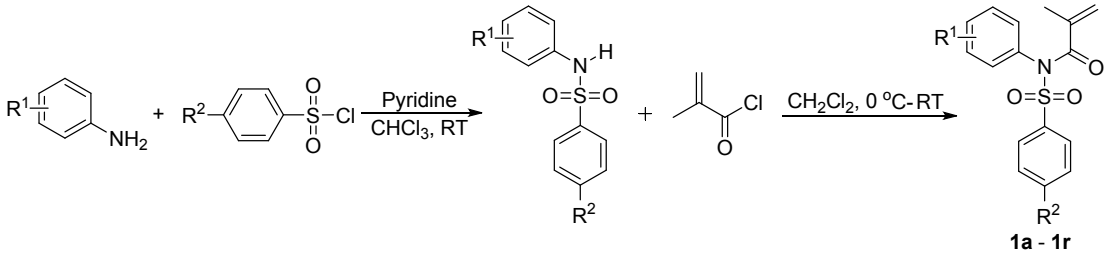


Fig. S1. a) SEM image, b) TEM image, c and its inset) HRTEM image and SAED pattern; nanosheets, d) XRD pattern, e) UV-vis diffuse reflectance spectra (DRS), f) The plots of (ahv)1/2 versus the energy of light (Eg) of the BiOBr nanosheets.

The morphology and structure of the as-prepared BiOBr nanostructures are characterized by using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The corresponding SEM image (Fig. S1a) and TEM image (Fig. S1b) show that the obtained samples are sheet-like structures with a thickness below 50 nm. High resolution transmission electron microscopy (HRTEM) (Fig. S1c) and selected area electron diffraction (SAED) pattern (the inset of Fig. S1c) of BiOBr demonstrate that the as-prepared BiOBr nanosheets are single-crystalline. The X-ray diffraction data of BiOBr samples is shown in Fig. S1d. All of the detectable reflection peaks can be assigned to a pure tetragonal phase of BiOBr (JCPDS No. 73-2061). The UV-vis diffuse reflectance spectra (DRS) of the BiOBr samples (Fig. S1e) displays its absorption range to 440 nm, corresponding to band-gap energy (Eg) of approximately 2.82 eV (Fig. S1f). To monitor the photocatalytic activity of the synthesized BiOBr nanosheets, the as-prepared BiOBr nanosheets are used in the following organic transformations.

4. Typical procedure for the synthesis of substrates

Substrates **1a**-**1r** were synthesized according to the literature[1].



Reference:

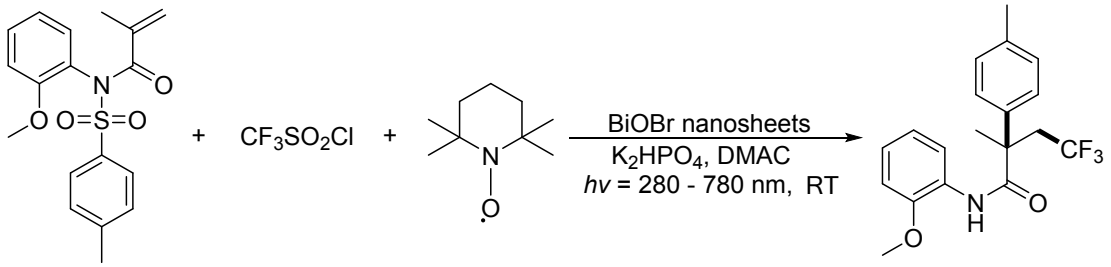
1. a) Kong, W. Q.; Casimiro, M.; Merino, E.; Nevado, C. *J. Am. Chem. Soc.* **2013**, *135*, 14480; b) Kong, W. Q.; Casimiro, M.; Fuentes, N.; Merino, E.; Nevado, C. *Angew. Chem. Int. Ed.* **2013**, *52*, 13086; c) Li, L.; Deng, M.; Zheng, S.-C.; Xiong, Y.-P.; Tan, B.; Liu, X.-Y. *Org. Lett.* **2014**, *16*, 504; d) Fan, J. H.; Yang, J.; Song, R. J.; Li, J. H. *Org. Lett.* **2015**, *17*, 836; e) Li, Y.-M.; Shen, Y. H.; Chang, K.-J.; Yang, S.-D. *Tetrahedron* **2014**, *70*, 1991.

5. General procedure for light-induced BiOBr nanosheets promoted one-pot tandem trifluoromethylation/aryl migration/desulfonylation and N−H bond formation of alkenes

1a (0.2 mmol, 63.1 mg), BiOBr (20.0 mg) and K_2HPO_4 (98%, 0.6 mmol, 106.6 mg) were weighted to a dried 10 mL schlenk flask under air, and then DMAC (1.0 mL) were added. After degassing under vacuum and refilling with Ar (three times), the Schlenk tube was sealed well. CF_3SO_2Cl (98%, 1.0 mmol, 172.0 mg) was added to the mixture via the microinjector. The Schlenk tube was immediately moved to a distance of ca. 30 cm from the light source and the resulting solution was stirred at room temperature under light irradiation until the reaction was completed. Then the reaction mixture was centrifuged. The organic phases were diluted with water (4.0 mL) and extracted with CH_2Cl_2 (3 × 6.0 mL). The combined organic layers were washed with brine, and dried over anhydrous $MgSO_4$. After removal of the solvent, the residue was purified by flash column chromatography on silica gel to give the desired product **3a**. 75% yield, **^{1}H-NMR** (400 MHz, $CDCl_3$) δ 7.36 – 7.29 (m, 6H), 7.25 (d, J = 8.1 Hz, 2H), 7.10 (t, J = 7.1 Hz, 1H), 6.81 (s, 1H), 3.21 (dq, J = 15.5, 11.1 Hz, 1H), 2.87 (dq, J = 15.6, 11.3 Hz, 1H), 2.40 (s, 3H), 1.87 (s, 3H); **^{19}F-NMR** (376 MHz, $CDCl_3$) [ppm] δ –59.04 (t, J = 11.1 Hz, 3F); **^{13}C-NMR** (100 MHz, $CDCl_3$) [ppm] δ 173.6, 138.0, 137.6, 137.5, 129.9, 129.0, 126.8, 124.6, 126.5 (q, J = 277.0 Hz), 119.9, 48.5 (d, J = 3.0 Hz), 42.0 (q, J = 26.0 Hz), 22.8, 21.0; **IR** (KBr) ν (cm^{-1}): 3317, 2923, 2360, 1670, 1602, 1544, 1503, 1444, 1263, 1139, 810, 746, 697, 649; **HRMS** (ESI) found: *m/z* 344.1238 $[M+Na]^+$; calcd. for $C_{18}H_{18}F_3NO$+Na 344.1238.

6. Control experiment

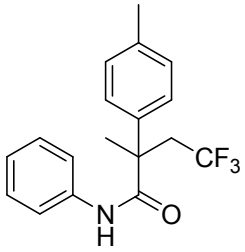
The addition of TEMPO to the reaction system under standard conditions



1c **2** **TEMPO** **3c, 0% yield**

1c (0.1 mmol, 34.5 mg), BiOBr (10.0 mg), K_2HPO_4 (98%, 0.3 mmol, 53.3 mg) and TEMPO (98%, 0.3 mmol, 47.8 mg) were weighted to a dried 10 mL schlenk flask under air and then DMAC (1.0 mL) were added. After degassing under vacuum and refilling with Ar (three times), the schlenk tube was sealed well. CF_3SO_2Cl (98%, 0.5 mmol, 86.0 mg) was added to the mixture via the microinjector. The Schlenk tube was immediately moved to a distance of ca. 30 cm from the light source and the resulting solution was stirred at room temperature under light irradiation for 6.0 h. After that, the light source was moved and α,α,α-trifluorotoluene (internal standard, 98%, 0.1 mmol, 14.3 mg) was added to the Schlenk tube. ^{19}F NMR analysis of this reaction mixture showed that only trace amount of **3c** was formed. This indicated that this one-pot tandem transformation of alkenes by our photocatalic strategy proceeded through a radical pathway.

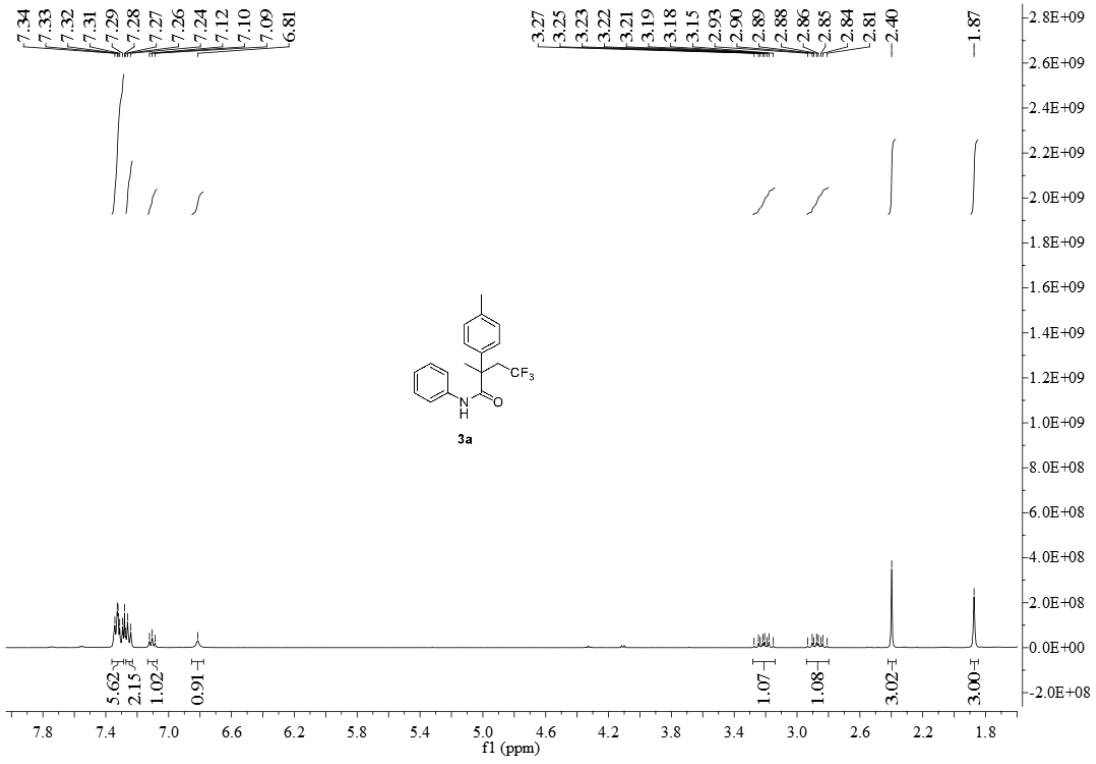
7. Copies of NMR Spectra

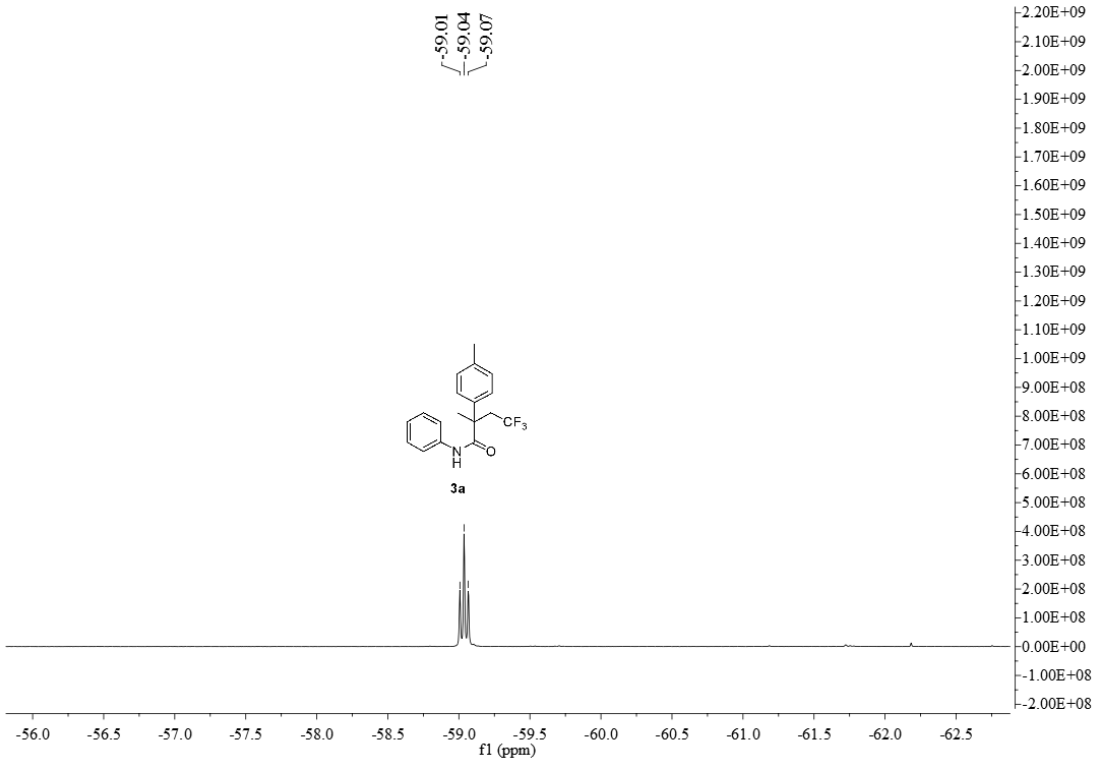


3a

4,4,4-trifluoro-2-methyl-N-phenyl-2-(p-tolyl)butanamide (3a)

M: 48.2 mg, **Yield:** 75%; **^{1}H-NMR** (400 MHz, $CDCl_3$) δ 7.36 – 7.29 (m, 6H), 7.25 (d, J = 8.1 Hz, 2H), 7.10 (t, J = 7.1 Hz, 1H), 6.81 (s, 1H), 3.21 (dq, J = 15.5, 11.1 Hz, 1H), 2.87 (dq, J = 15.6, 11.3 Hz, 1H), 2.40 (s, 3H), 1.87 (s, 3H); **^{19}F-NMR** (376 MHz, $CDCl_3$) [ppm] δ –59.04 (t, J = 11.1 Hz, 3F); **^{13}C-NMR** (100 MHz, $CDCl_3$) [ppm] δ 173.6, 138.0, 137.6, 137.5, 129.9, 129.0, 126.8, 124.6, 126.5 (q, J = 277.0 Hz), 119.9, 48.5 (d, J = 3.0 Hz), 42.0 (q, J = 26.0 Hz), 22.8, 21.0; **IR** (KBr) ν (cm^{-1}): 3317, 2923, 2360, 1670, 1602, 1544, 1503, 1444, 1263, 1139, 810, 746, 697, 649; **HRMS** (ESI) found: *m/z* 344.1238 $[M+Na]^+$; calcd. for $C_{18}H_{18}F_3NO$+Na 344.1238.

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O
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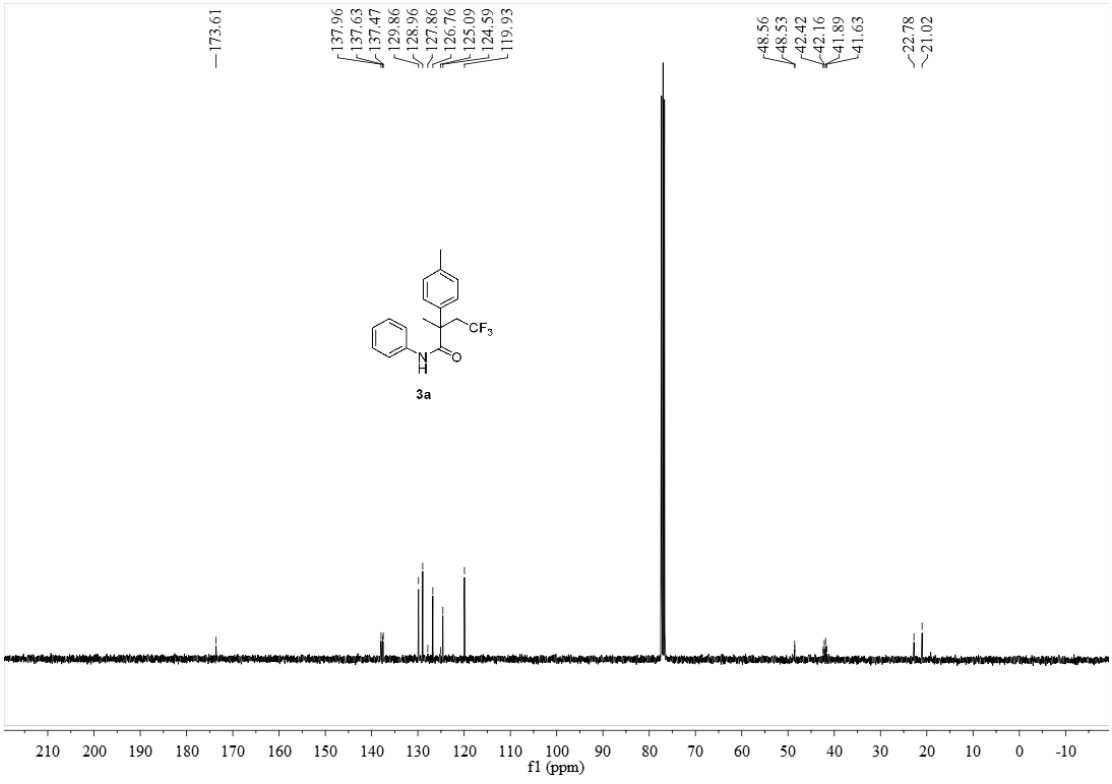
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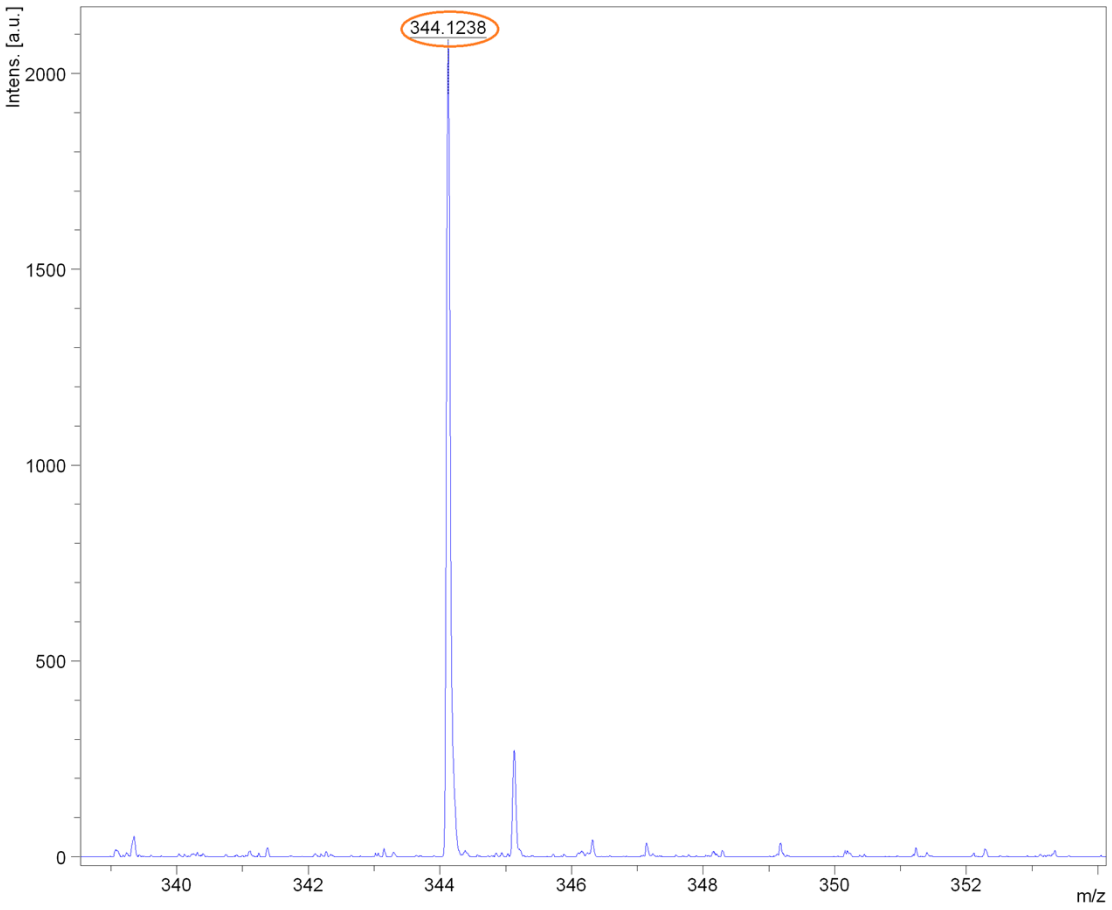
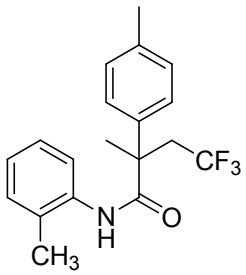
3a
f1 (ppm)

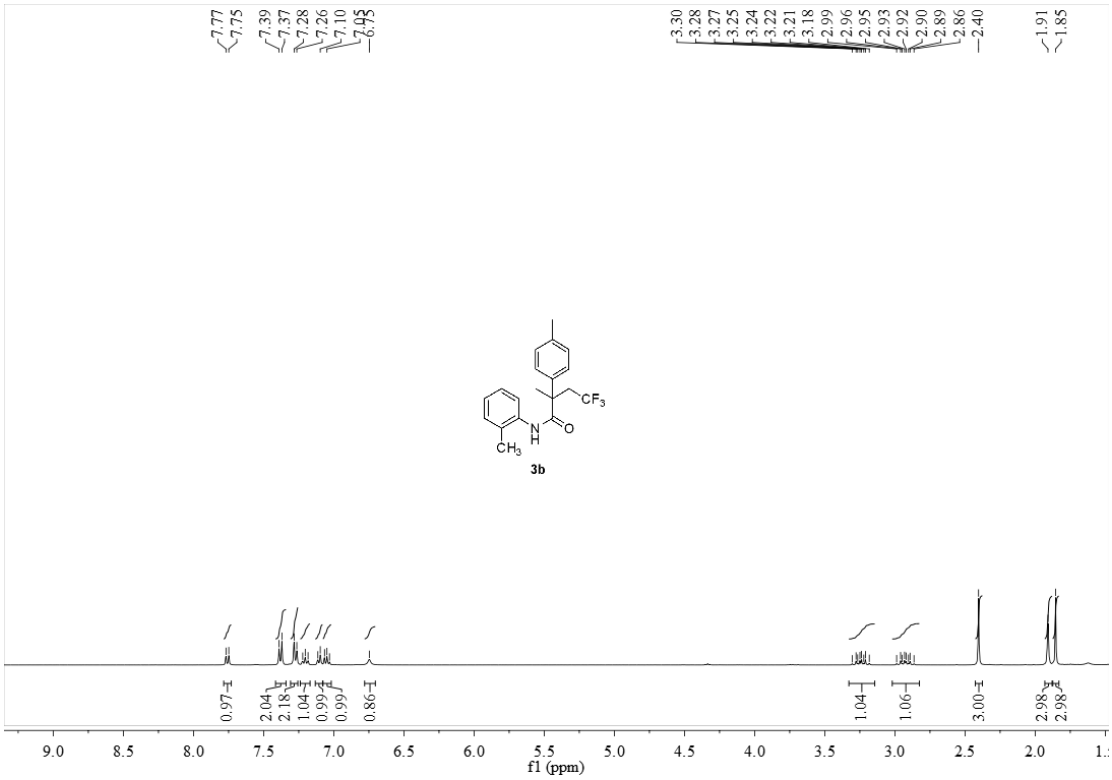




3b

4,4,4-trifluoro-2-methyl-N-(o-tolyl)-2-(p-tolyl)butanamide (3b)

M: 40.9 mg, **Yield:** 61%; **^{1}H-NMR** (400 MHz, $CDCl_3$) [ppm] δ 7.76 (d, J = 8.0 Hz, 1H), 7.38 (d, J = 8.2 Hz, 2H), 7.27 (d, J = 7.6 Hz, 2H), 7.20 (t, J = 7.6 Hz, 1H), 7.11 (d, J = 6.8 Hz, 1H), 7.05 (t, J = 7.1 Hz, 1H), 6.75 (s, 1H), 3.24 (dq, J = 15.6, 11.0 Hz, 1H), 2.93 (dq, J = 15.6, 11.3 Hz, 1H), 2.40 (s, 3H), 1.91 (s, 3H), 1.85 (s, 3H); **^{19}F-NMR** (376 MHz, $CDCl_3$) [ppm] δ –58.90 (t, J = 11.1 Hz, 3F); **^{13}C-NMR** (100 MHz, $CDCl_3$) [ppm] δ 173.7, 138.0, 137.9, 135.4, 130.4, 129.8, 128.7, 126.8, 126.7, 126.6 (q, J = 279.0 Hz), 125.2, 122.3, 48.6 (d, J = 1.0 Hz), 41.7 (q, J = 28.0 Hz), 22.9, 21.0, 17.0; **IR** (KBr) ν (cm^{-1}): 3277, 2923, 2856, 2360, 1666, 1516, 1455, 1366, 1303, 1260, 1157, 1121, 1080, 1038, 953, 815, 749, 672, 581; **HRMS** (ESI) found: *m/z* 358.1392 $[M+Na]^+$; calcd. for $C_{19}H_{20}F_3NO$+Na 358.1395.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O
CH3
3b
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2.18
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0.99
0.99
0.86
1.04
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3.00
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2.98
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8.0
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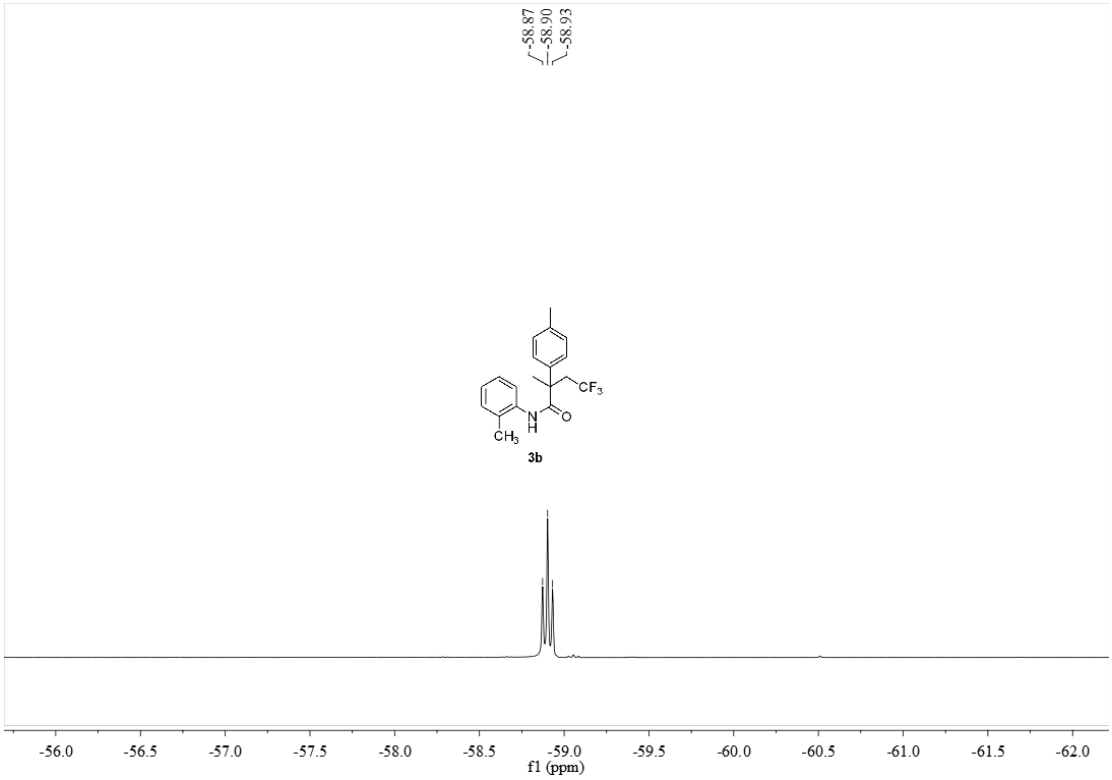
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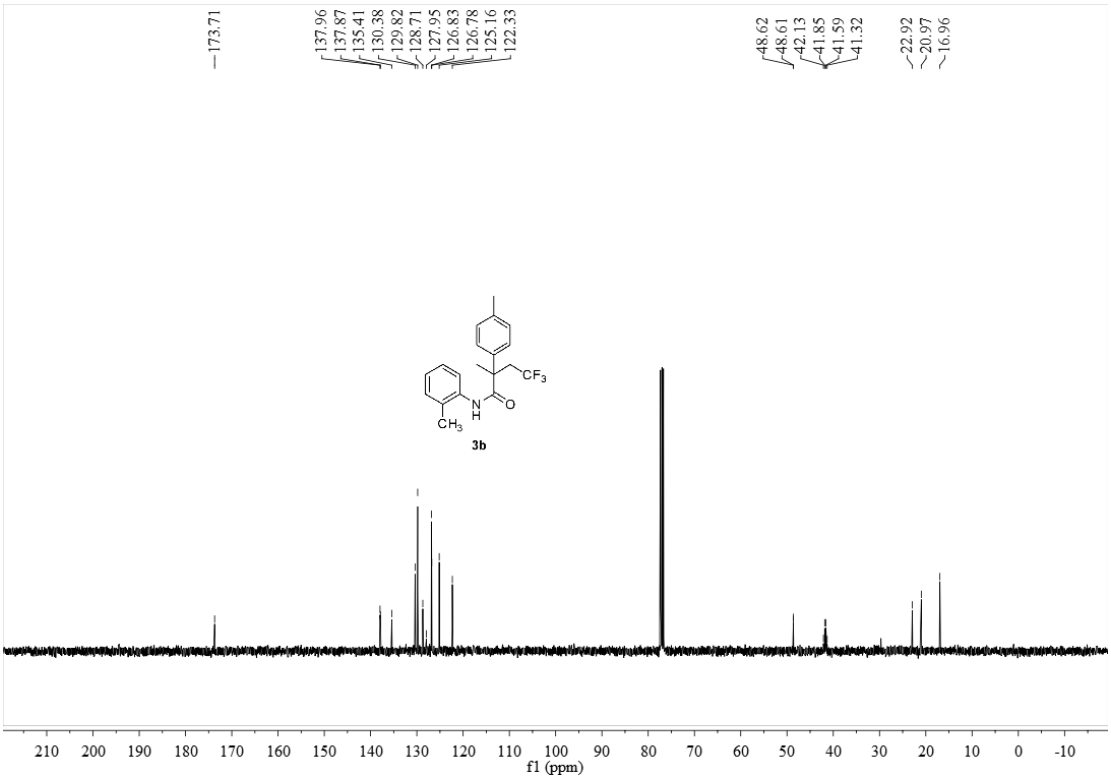
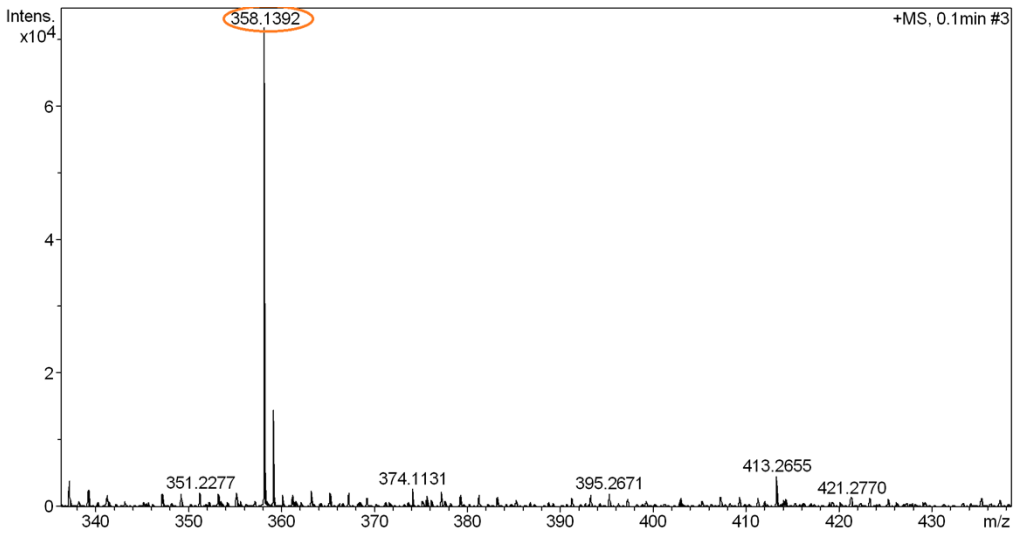
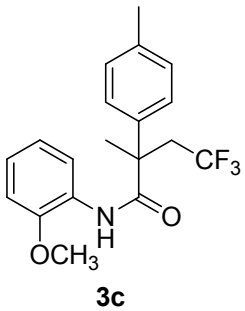




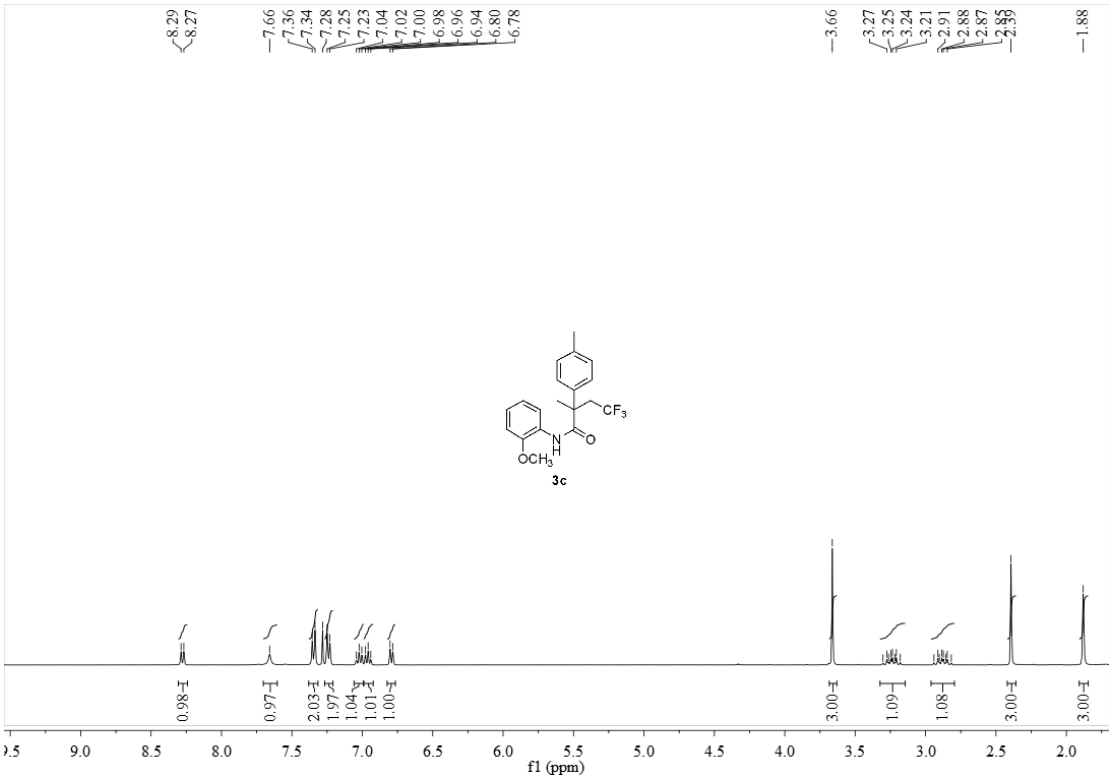


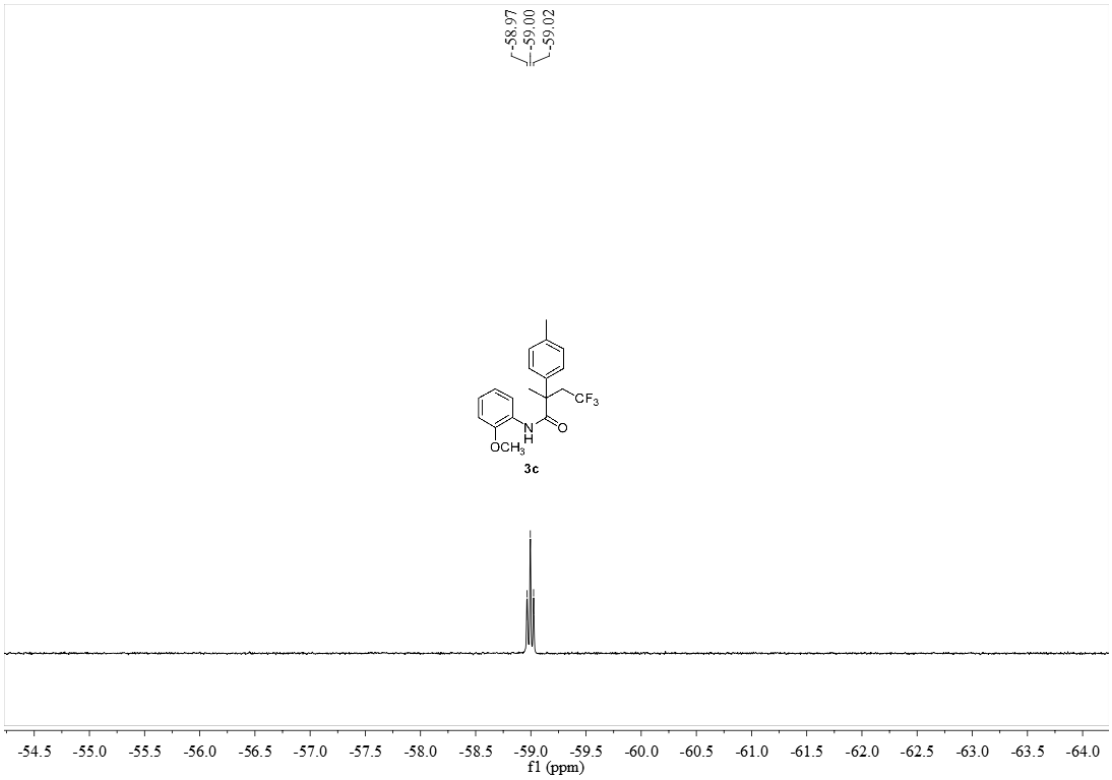
3c

4,4,4-trifluoro-N-(2-methoxyphenyl)-2-methyl-2-(p-tolyl)butanamide (3c)

M: 53.4 mg, **Yield:** 76%; **^{1}H-NMR** (400 MHz, $CDCl_3$) [ppm] δ 8.28 (d, J = 7.8 Hz, 1H), 7.66 (s,

1H), 7.35 (d, J = 8.1 Hz, 2H), 7.24 (d, J = 8.0 Hz, 2H), 7.02 (t, J = 7.6 Hz, 1H), 6.96 (t, J = 7.6 Hz, 1H), 6.79 (d, J = 8.0 Hz, 1H), 3.66 (s, 3H), 3.24 (dq, J = 15.5, 11.1 Hz, 1H), 2.88 (dq, J = 15.5, 11.3 Hz, 1H), 2.39 (s, 3H), 1.88 (s, 3H); **^{19}F-NMR** (376 MHz, $CDCl_3$) [ppm] δ –59.00 (t, J = 11.1 Hz, 3F); **^{13}C-NMR** (100 MHz, $CDCl_3$) [ppm] δ 173.2, 148.2, 138.0, 137.5, 129.6, 127.5, 126.3, 126.6 (q, J = 279.0 Hz), 123.9, 121.1, 119.6, 110.1, 55.7, 48.7 (d, J = 1.0 Hz), 41.9 (q, J = 26.0 Hz), 29.7, 21.0; **IR** (KBr) ν (cm^{-1}): 3381, 2919, 2851, 2359, 1684, 1601, 1526, 1463, 1364, 1257, 1119, 1034, 938, 745; **HRMS** (ESI) found: *m/z* 374.1338 $[M+Na]^+$; calcd. for $C_{19}H_{20}F_3NO_2$+Na 374.1344.

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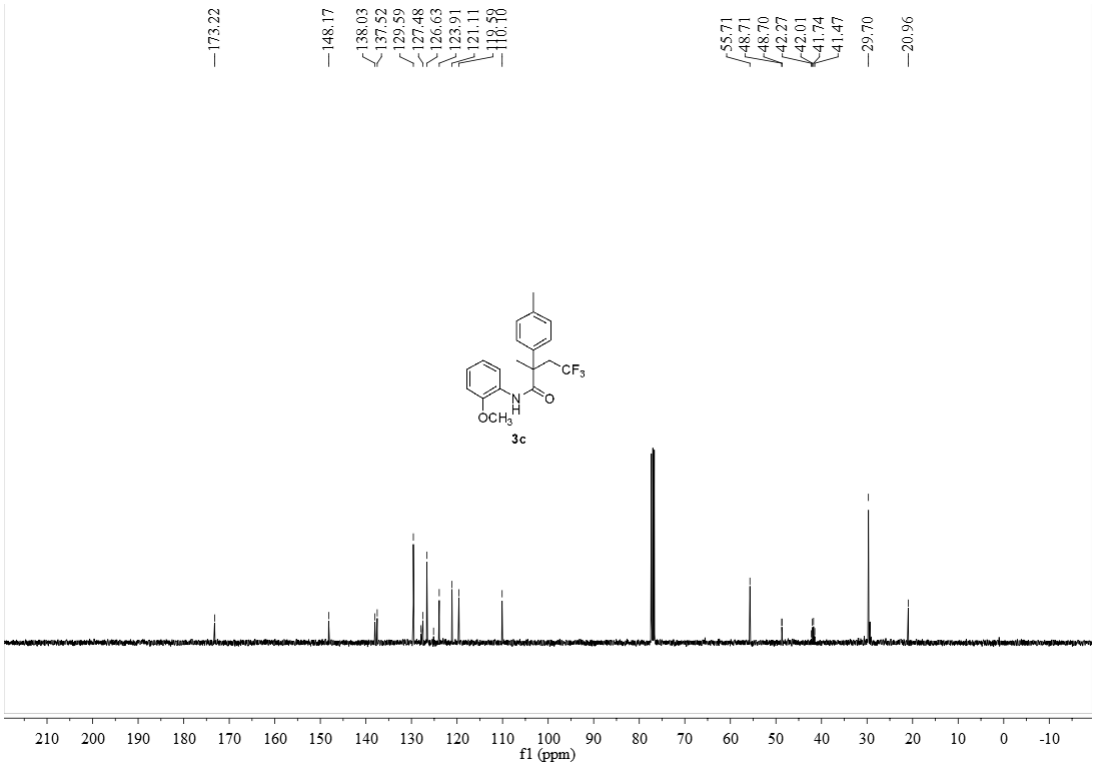
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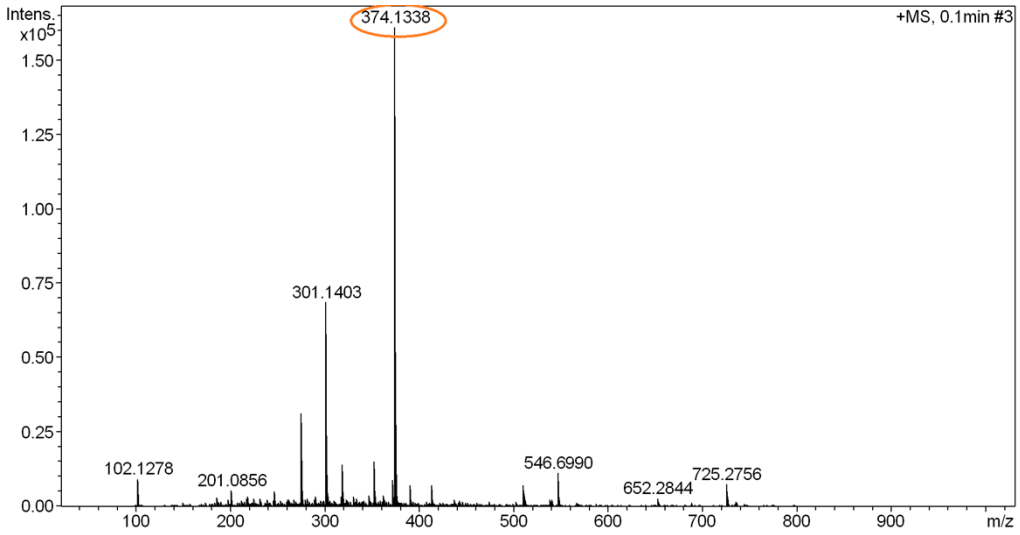
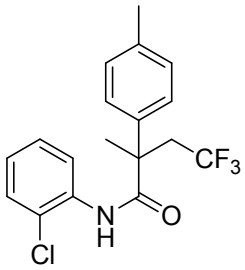
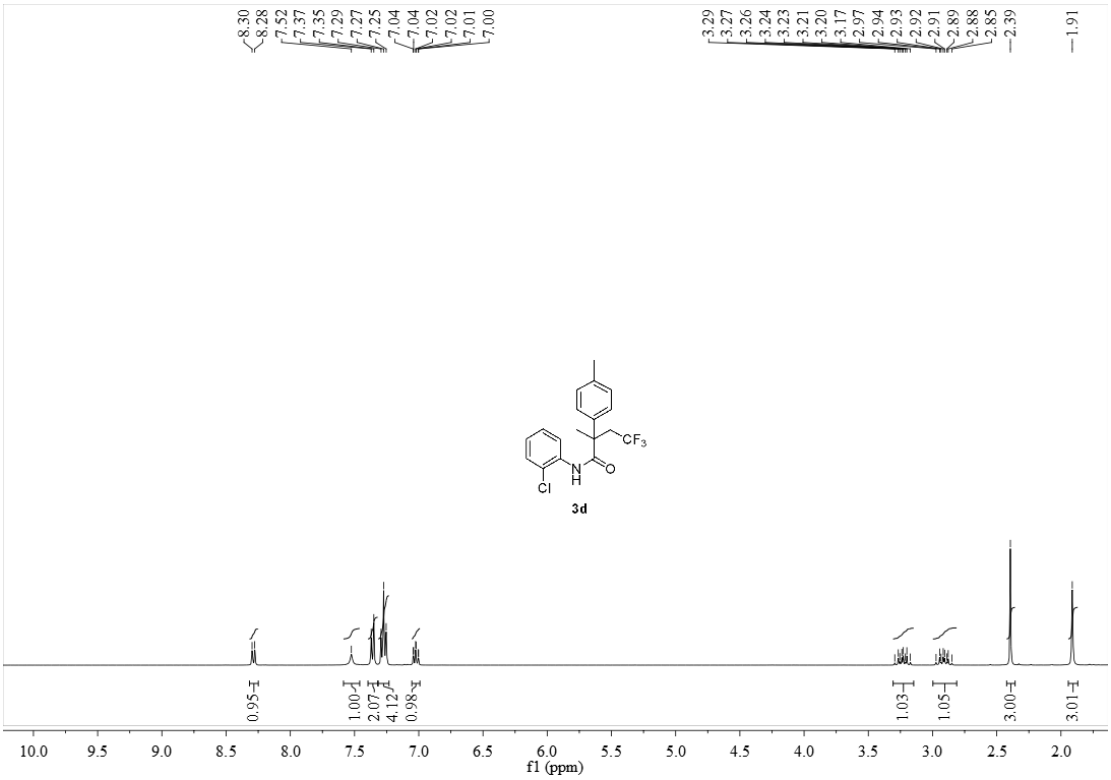




3d

N-(2-chlorophenyl)-4,4,4-trifluoro-2-methyl-2-(p-tolyl)butanamide (3d)

M: 37.7 mg, **Yield:** 53%; **^{1}H-NMR** (400 MHz, $CDCl_3$) [ppm] δ 8.29 (d, J = 8.3 Hz, 1H), 7.52 (s, 1H), 7.36 (d, J = 8.2 Hz, 2H), 7.27 (t, J = 8.0 Hz, 4H), 7.02 (td, J = 7.8, 1.4 Hz, 1H), 3.23 (dq, J = 15.6, 11.0 Hz, 1H), 2.91 (dq, J = 15.6, 11.2 Hz, 1H), 2.39 (s, 3H), 1.91 (s, 3H); **^{19}F-NMR** (376 MHz, $CDCl_3$) [ppm] δ –58.97 (t, J = 11.0 Hz, 3F); **^{13}C-NMR** (100 MHz, $CDCl_3$) [ppm] δ 173.6, 138.0, 137.3, 134.4, 129.9, 128.9, 127.7, 126.7, 126.4 (q, J = 276.0 Hz), 124.8, 123.2, 121.4, 48.8 (d, J = 1.0 Hz), 41.8 (q, J = 28.0 Hz), 22.7, 21.0; **IR** (KBr) ν (cm^{-1}): 3379, 2986, 2928, 2360, 1693, 1592, 1520, 1438, 1370, 1304, 1260, 1123, 1081, 1037, 940, 819, 753, 633, 566; **HRMS** (ESI) found: *m/z* 378.0849 $[M+Na]^+$; calcd. for $C_{18}H_{17}ClF_3NO$+Na 378.0849.


8.30
8.28
7.52
7.37
7.35
7.29
7.27
7.25
7.04
7.04
7.02
7.02
7.01
7.00
3.29
3.27
3.26
3.24
3.23
3.21
3.20
3.17
2.97
2.94
2.93
2.92
2.91
2.89
2.88
2.85
2.39
1.91
CF3
Cl
N
H
O
3d
0.95
1.00
2.07
4.12
0.98
1.03
1.05
3.00
3.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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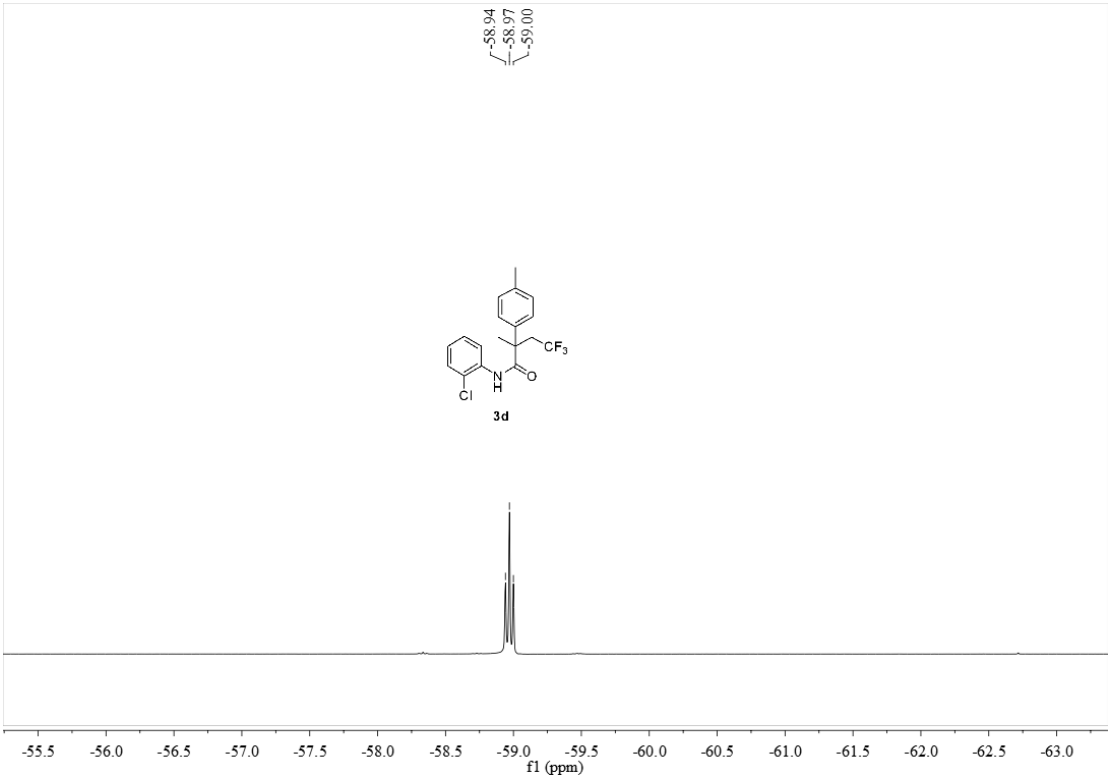
3.5
3.0
2.5
2.0
f1 (ppm)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-58.97
-59.00
CF3
Cl
N
H
O
3d
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-56.5
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-58.0
-58.5
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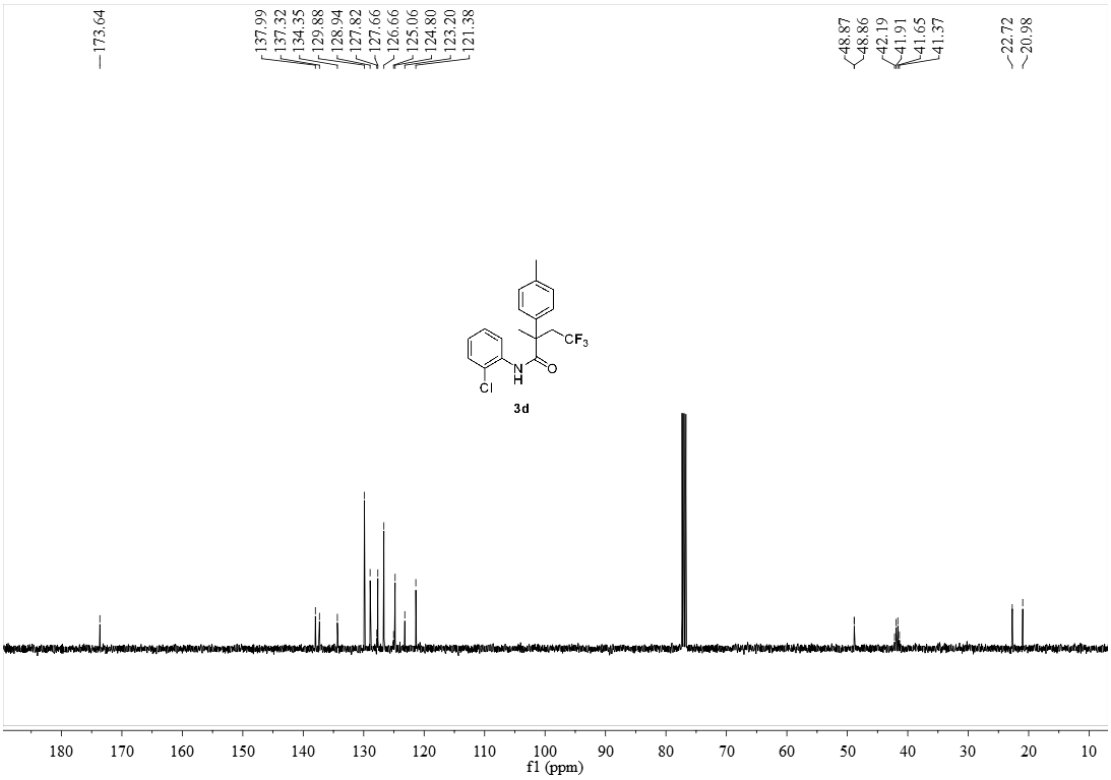
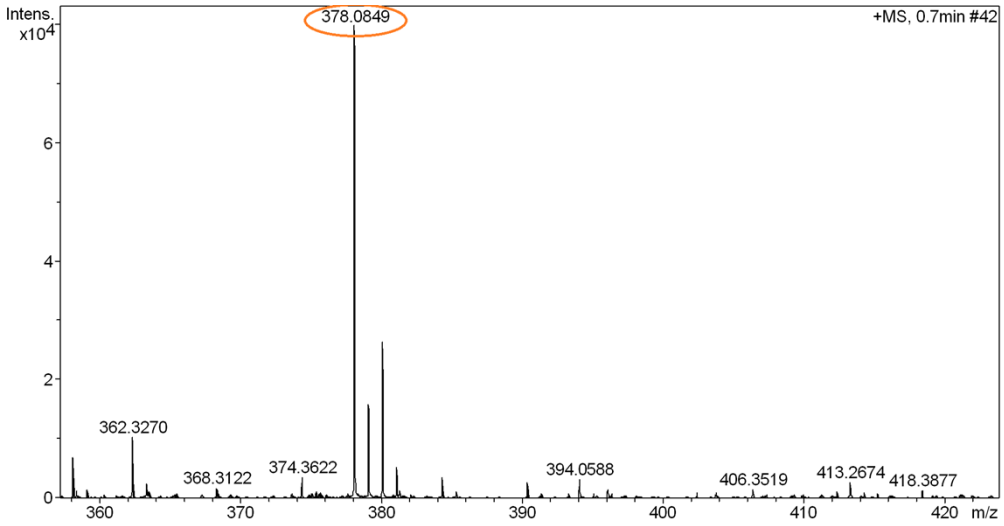
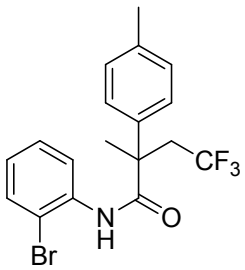
-62.0
-62.5
-63.0
f1 (ppm)





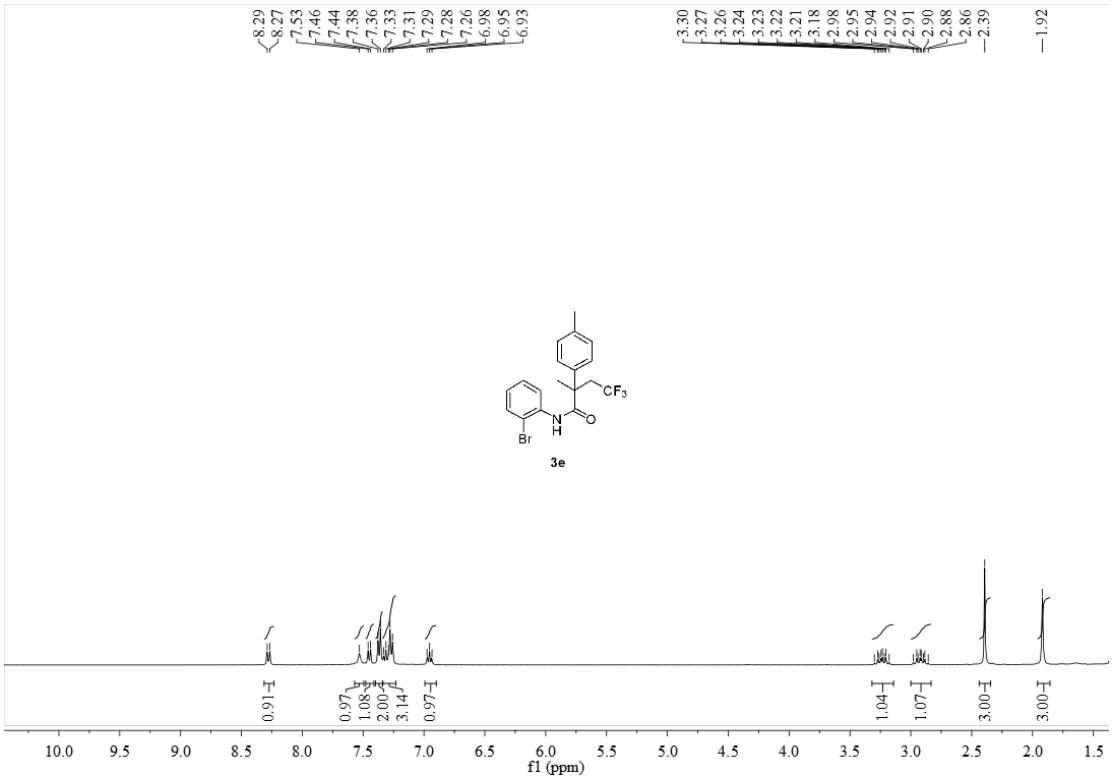


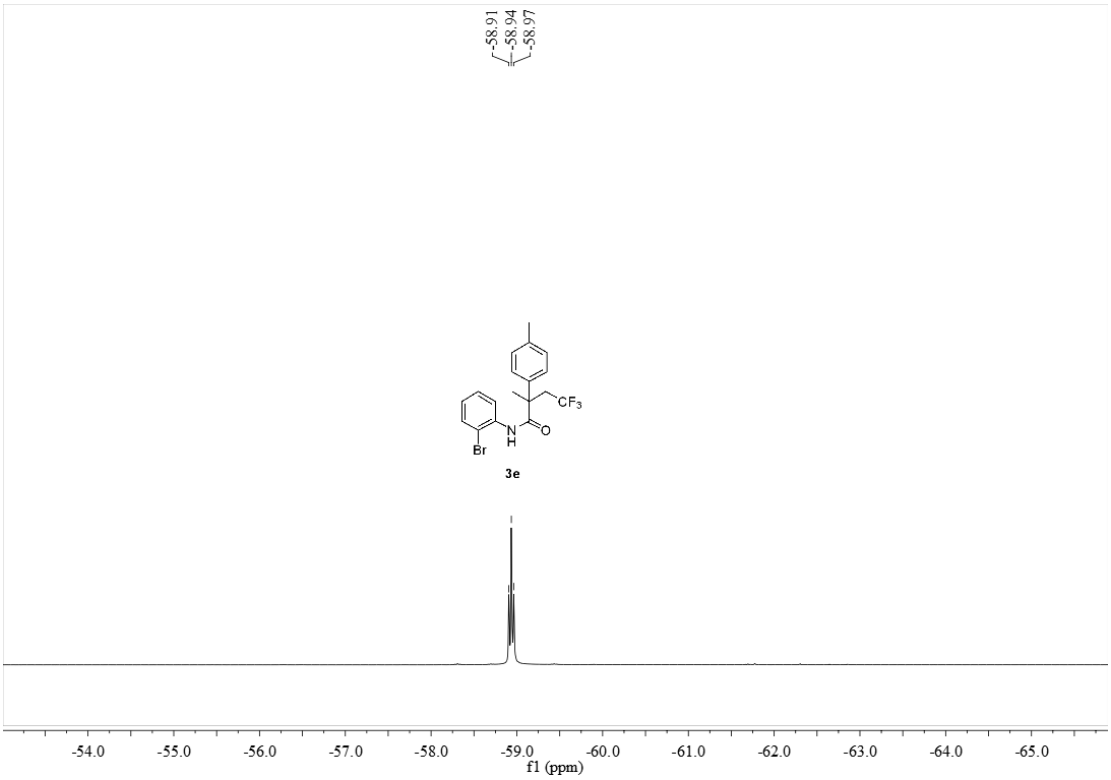
3e

N-(2-bromophenyl)-4,4,4-trifluoro-2-methyl-2-(p-tolyl)butanamide (3e)

M: 42.4 mg, **Yield:** 53%; **^{1}H-NMR** (400 MHz, $CDCl_3$) [ppm] δ 8.28 (d, J = 8.2 Hz, 1H), 7.53 (s,

1H), 7.45 (d, J = 8.0 Hz, 1H), 7.37 (d, J = 8.2 Hz, 2H), 7.34–7.23 (m, 3H), 6.95 (t, J = 8.2 Hz, 1H), 3.24 (dq, J = 15.5, 11.0 Hz, 1H), 2.92 (dq, J = 15.5, 11.2 Hz, 1H), 2.39 (s, 3H), 1.92 (s, 3H); **^{19}F-NMR** (376 MHz, $CDCl_3$) [ppm] δ –58.94 (t, J = 11.0 Hz, 3F); **^{13}C-NMR** (100 MHz, $CDCl_3$) [ppm] δ 173.7, 138.0, 137.3, 135.5, 132.2, 129.9, 128.3, 126.7, 126.5 (q, J = 277.0 Hz), 125.3, 121.6, 113.6, 48.9 (d, J = 1.0 Hz), 41.8 (q, J = 28.0 Hz), 22.7, 21.0; **IR** (KBr) ν (cm^{-1}): 3361, 2924, 2855, 2361, 1693, 1587, 1518, 1434, 1368, 1301, 1260, 1206, 1158, 1121, 1080, 1032, 941, 816, 752, 668, 564; **HRMS** (ESI) found: *m/z* 422.0335 $[M+Na]^+$; calcd. for $C_{18}H_{17}BrF_3NO$+Na 422.0344.

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-58.94
-58.97
CF3
N
H
O
Br
3e
-54.0
-55.0
-56.0
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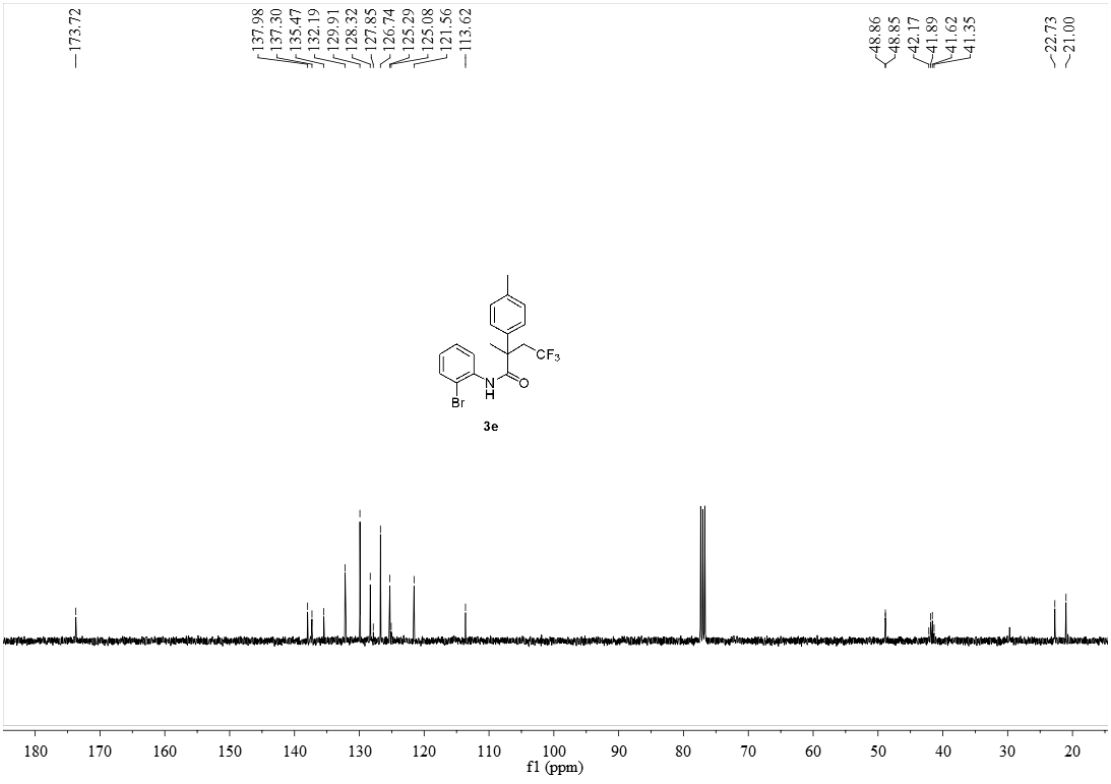
-62.0
-63.0
-64.0
-65.0
f1 (ppm)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129.91
128.32
127.85
126.74
125.29
125.08
121.56
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48.86
48.85
42.17
41.89
41.62
41.35
22.73
21.00
CF3
N
H
O
Br
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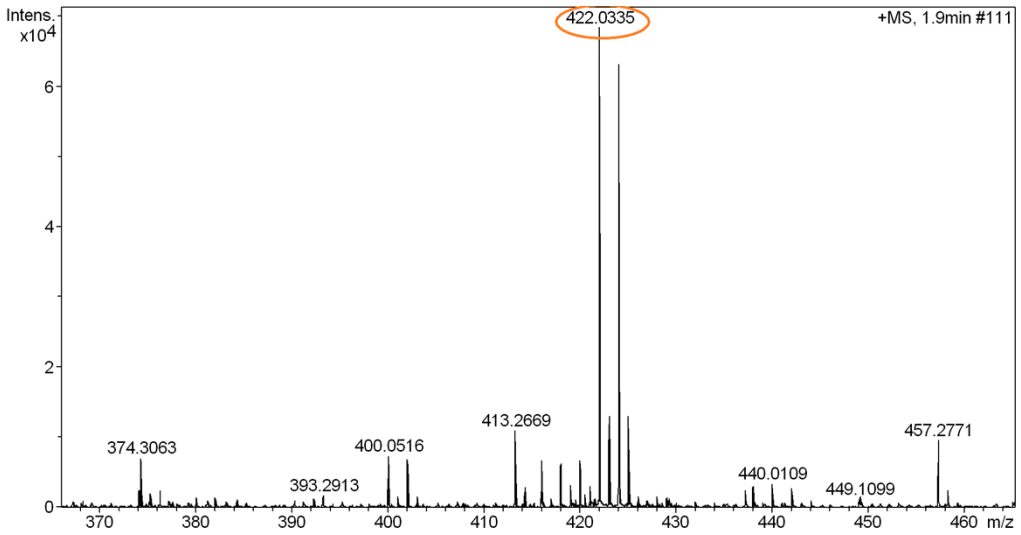
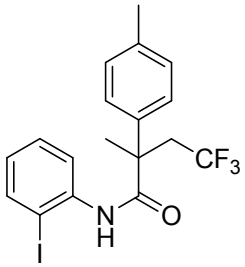
80
70
60
50
40
30
20
f1 (ppm)

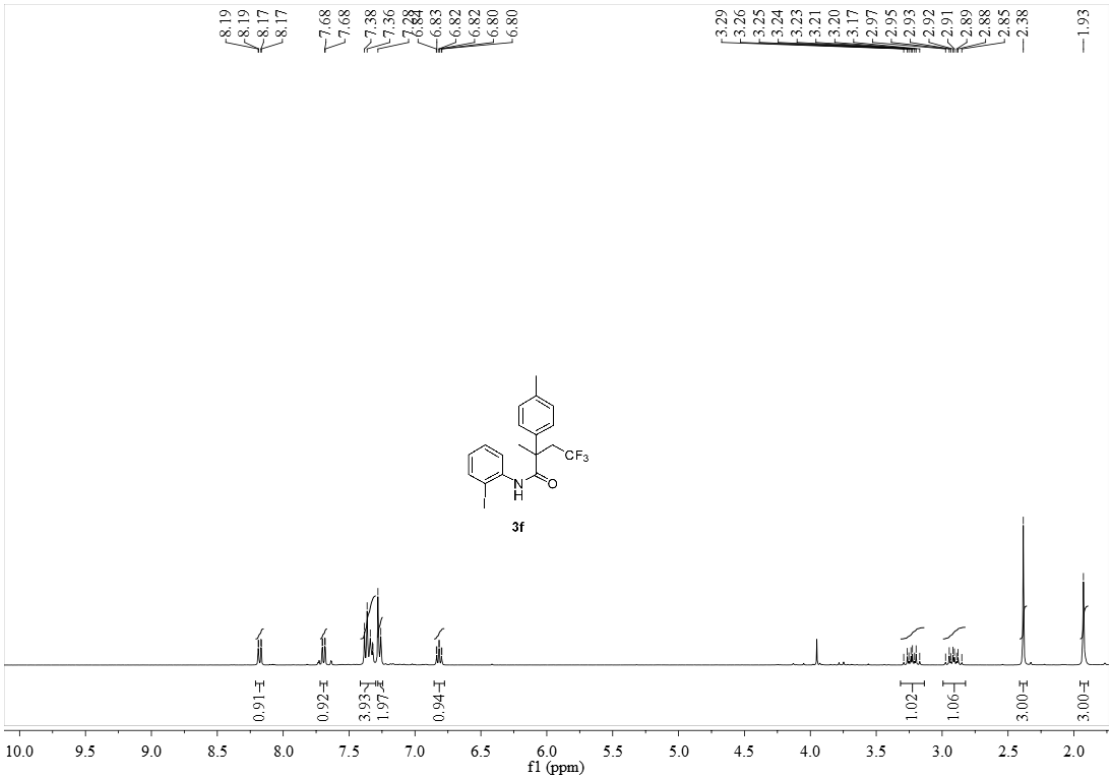




3f

4,4,4-trifluoro-N-(2-iodophenyl)-2-methyl-2-(p-tolyl)butanamide (3f)

M: 36.7 mg, **Yield:** 41%; **^{1}H-NMR** (400 MHz, $CDCl_3$) [ppm] δ 8.18 (dd, J = 8.2, 1.3 Hz, 1H), 7.69 (dd, J = 8.0, 1.3 Hz, 1H), 7.41–7.30 (m, 4H), 7.27 (d, J = 8.8 Hz, 2H), 6.82 (td, J = 7.8, 1.5 Hz, 1H), 3.23 (dq, J = 15.6, 11.0 Hz, 1H), 2.91 (dq, J = 15.6, 11.2 Hz, 1H), 2.38 (s, 3H), 1.93 (s, 3H); **^{19}F-NMR** (376 MHz, $CDCl_3$) [ppm] δ –58.93 (t, J = 11.1 Hz, 3F); **^{13}C-NMR** (100 MHz, $CDCl_3$) [ppm] δ 173.9, 138.8, 138.0, 137.9, 137.4, 130.0, 129.2, 126.9, 126.5 (q, J = 277.0 Hz), 126.0, 121.5, 89.5, 48.8 (d, J = 1.0 Hz), 41.8 (q, J = 27.0 Hz), 22.8, 21.0; **IR** (KBr) ν (cm^{-1}): 3346, 2956, 2866, 2359, 1690, 1581, 1515, 1431, 1366, 1297, 1259, 1121, 1080, 1032, 938, 816, 751, 652, 561; **HRMS** (ESI) found: *m/z* 470.0212 $[M+Na]^+$; calcd. for $C_{18}H_{17}IF_3NO$+Na 470.0205.


8.19
8.19
8.17
8.17
7.68
7.68
7.38
7.36
7.28
6.83
6.82
6.82
6.80
6.80
3.29
3.26
3.25
3.24
3.23
3.21
3.20
3.17
2.97
2.95
2.93
2.92
2.91
2.89
2.88
2.85
2.38
1.93
CF3
N
H
O
I
3f
0.91
0.92
3.93
1.97
0.94
1.02
1.06
3.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
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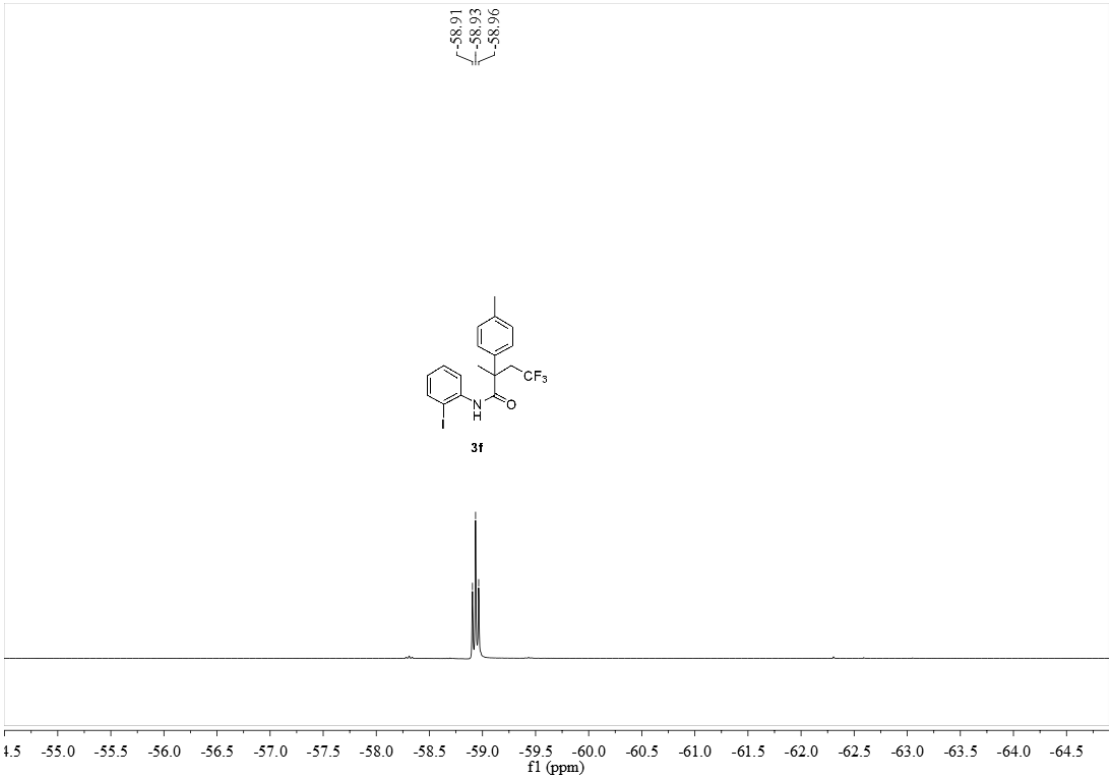
3.5
3.0
2.5
2.0
f1 (ppm)


-58.91
-58.93
-58.96
CF3
N
H
O
I
3f
-55.0
-55.5
-56.0
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-57.5
-58.0
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-59.5
-60.0
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-61.5
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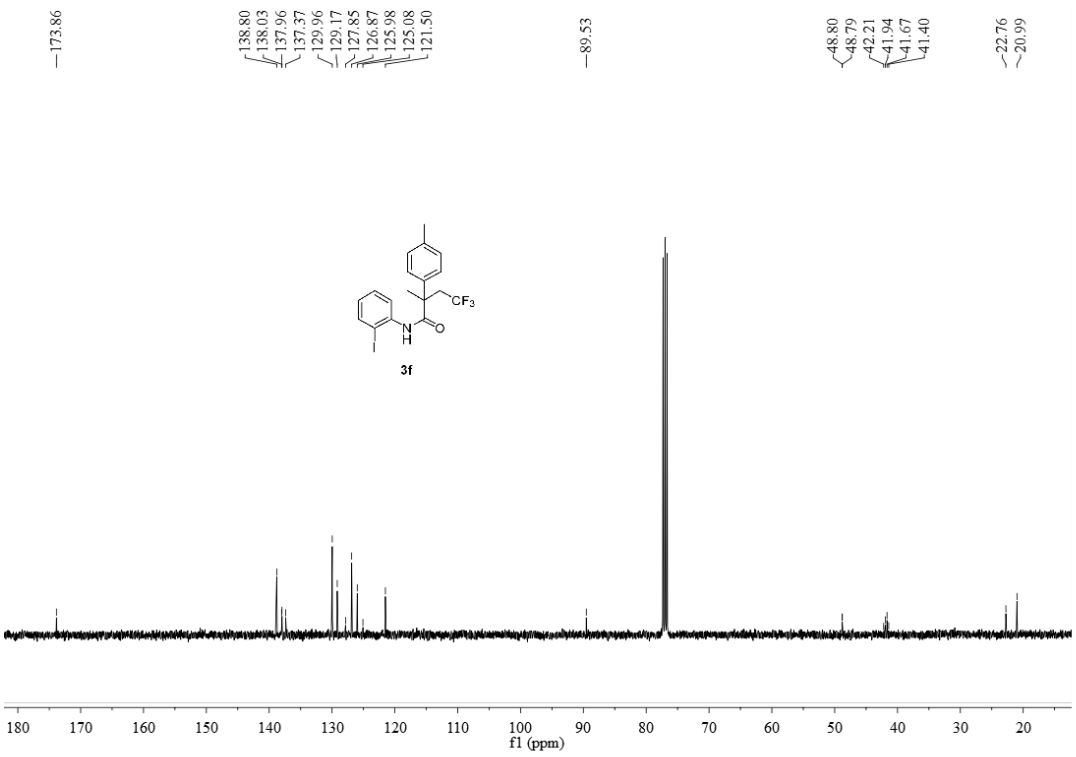
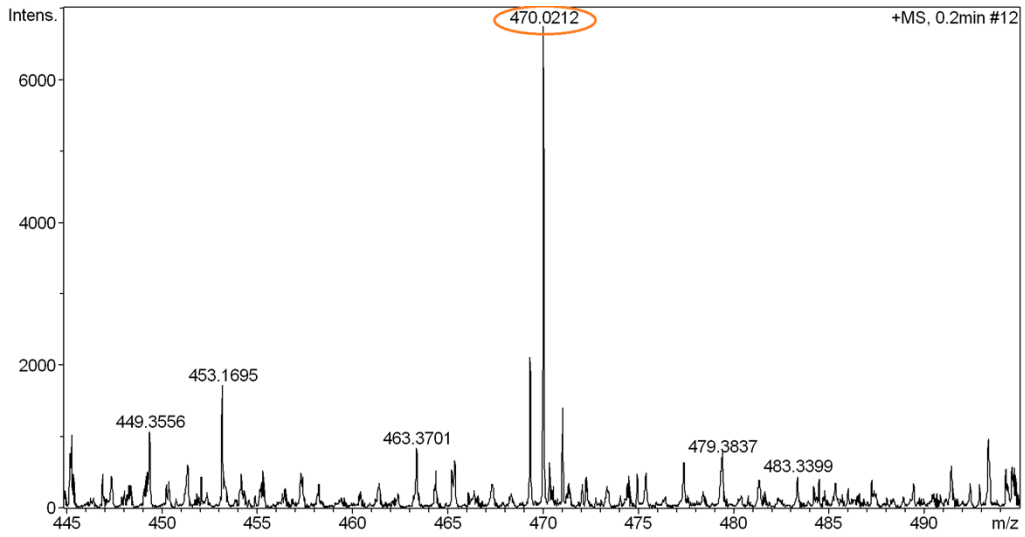
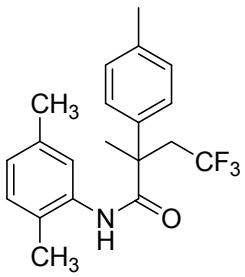
-62.5
-63.0
-63.5
-64.0
-64.5
f1 (ppm)





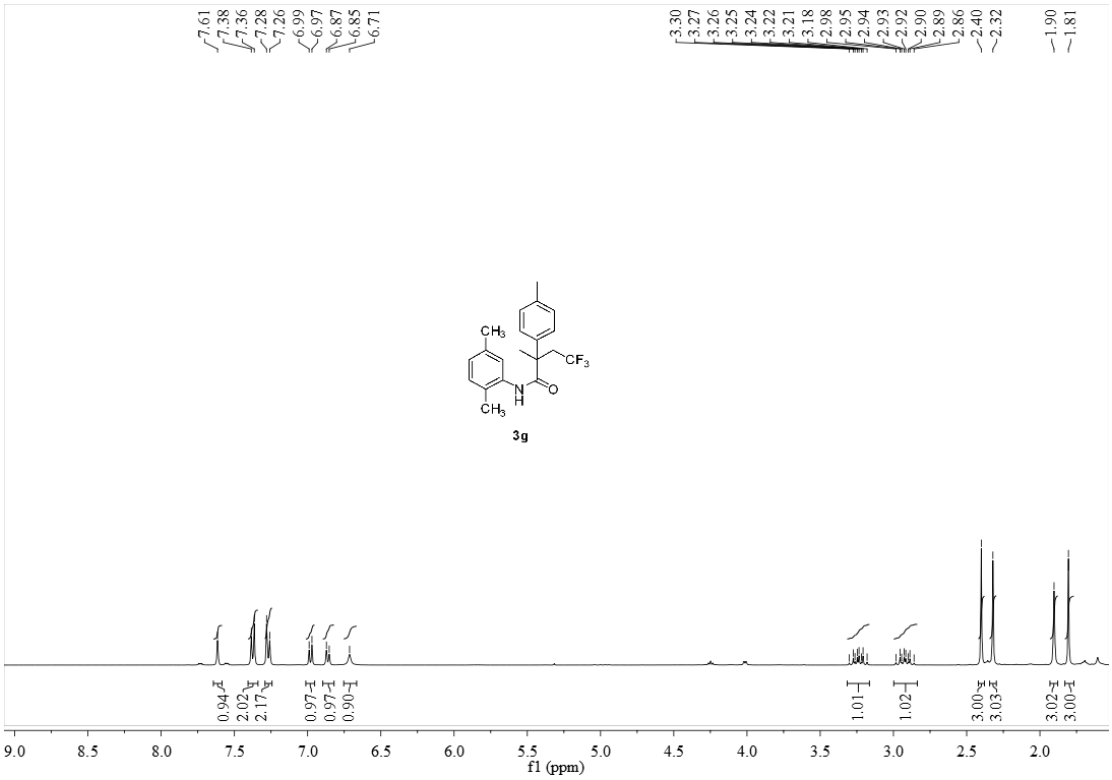


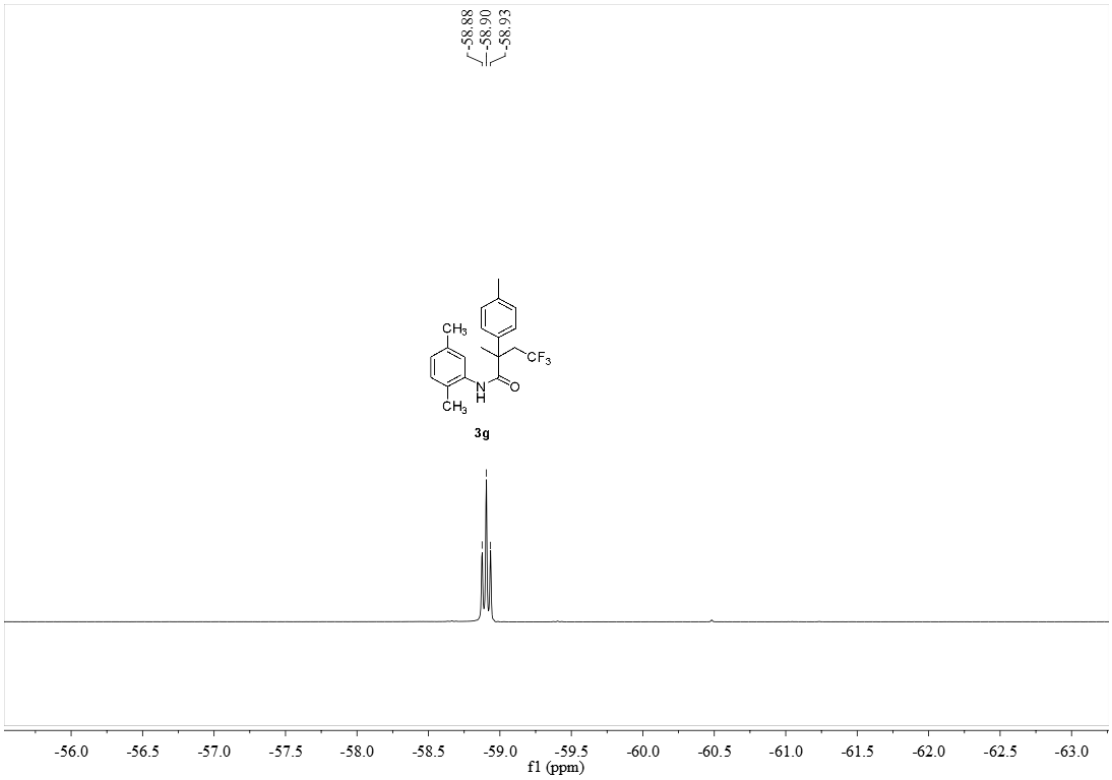
3g

N-(2,5-dimethylphenyl)-4,4,4-trifluoro-2-methyl-2-(p-tolyl)butanamide (3g)

M: 36.3 mg, **Yield:** 52%; **^{1}H-NMR** (400 MHz, $CDCl_3$) [ppm] δ 7.61 (s, 1H), 7.37 (d, J = 8.2 Hz,

2H), 7.27 (d, J = 8.7 Hz, 2H), 6.98 (d, J = 7.7 Hz, 1H), 6.86 (d, J = 7.6 Hz, 1H), 6.71 (s, 1H), 3.24 (dq, J = 15.6, 11.0 Hz, 1H), 2.92 (dq, J = 15.6, 11.3 Hz, 1H), 2.40 (s, 3H), 2.32 (s, 3H), 1.90 (s, 3H), 1.81 (s, 3H); **^{19}F-NMR** (376 MHz, $CDCl_3$) [ppm] δ –58.90 (t, J = 11.1 Hz, 3F); **^{13}C-NMR** (100 MHz, $CDCl_3$) [ppm] δ 173.7, 137.9, 137.9, 136.6, 135.2, 130.2, 129.8, 126.8, 126.6 (q, J = 277.0 Hz), 125.9, 125.5, 122.8, 48.6 (d, J = 1.0 Hz), 41.7 (q, J = 27.0 Hz), 22.9 (t, J = 1.5 Hz), 21.1, 21.0, 16.5; **IR** (KBr) ν (cm^{-1}): 3312, 2928, 2864, 2359, 1728, 1669, 1581, 1524, 1369, 1262, 1163, 1124, 1079, 1039, 950, 810, 646, 576; **HRMS** (ESI) found: *m/z* 372.1549 $[M+Na]^+$; calcd. for $C_{20}H_{22}F_3NO$+Na 372.1551.




-58.88
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-58.93
CH3
CF3
N
H
O
CH3
3g
-56.0
-56.5
-57.0
-57.5
-58.0
-58.5
-59.0
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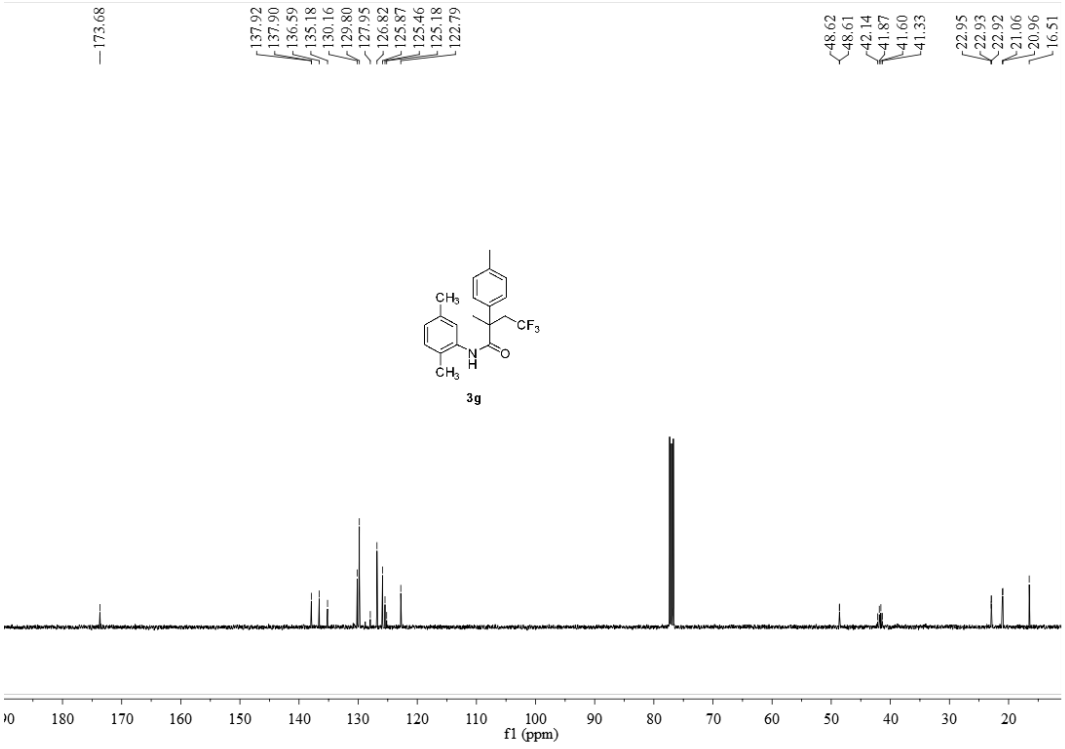
-60.0
-60.5
-61.0
-61.5
-62.0
-62.5
-63.0
f1 (ppm)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136.59
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129.80
127.95
126.82
125.87
125.46
125.18
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48.61
42.14
41.87
41.60
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22.95
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22.92
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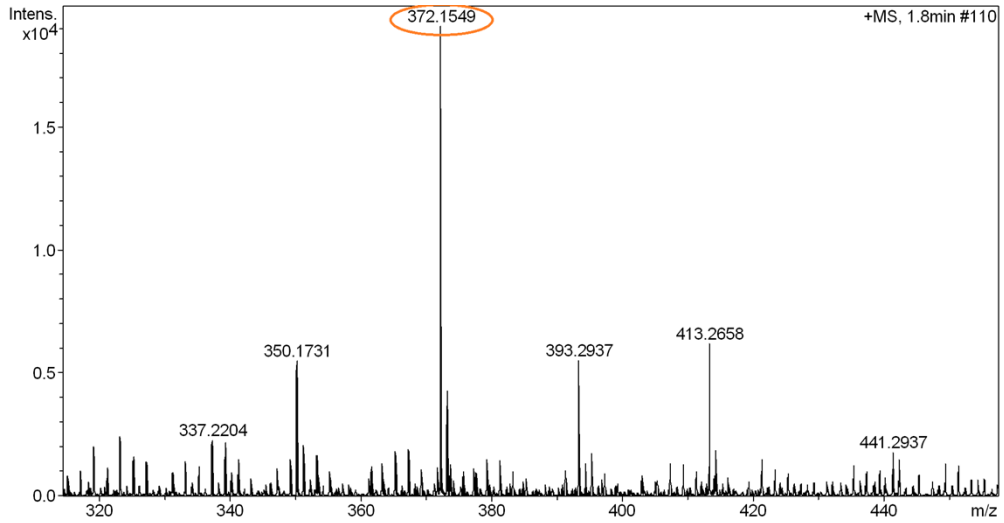
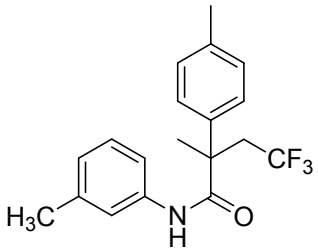
CF3
N
H
O
CH3
3g
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)

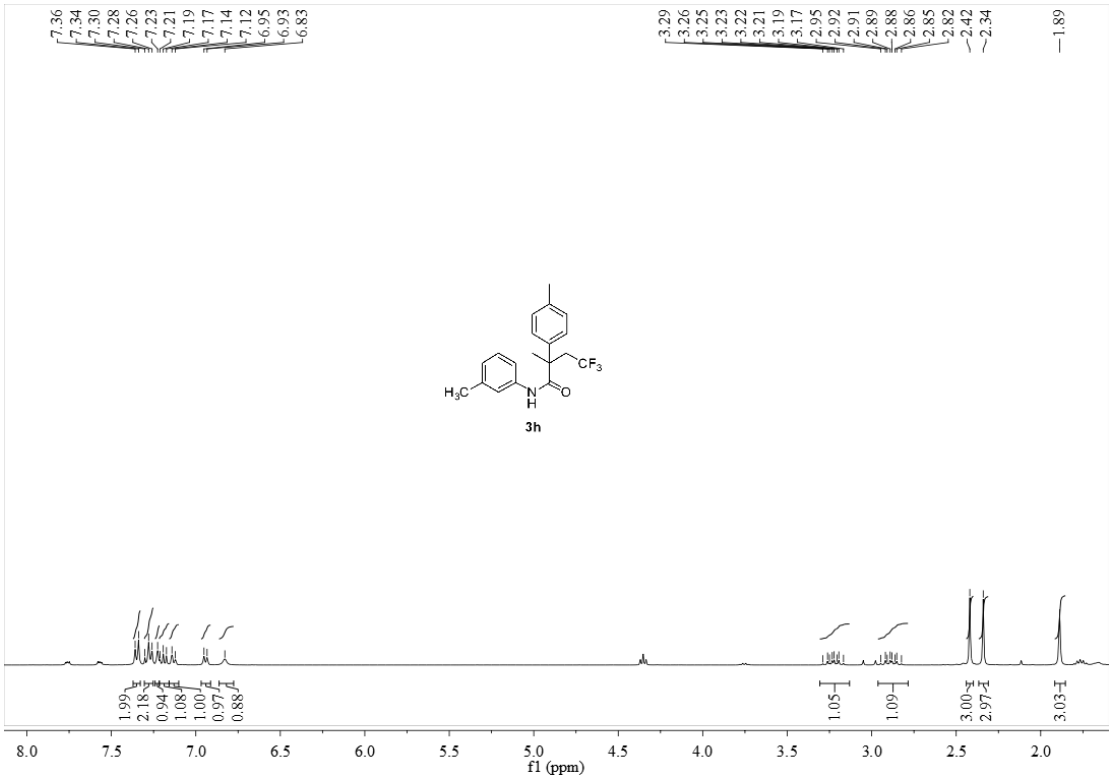




3h

4,4,4-trifluoro-2-methyl-N-(m-tolyl)-2-(p-tolyl)butanamide (3h)

M: 24.8 mg, **Yield:** 37%; **^{1}H-NMR** (400 MHz, $CDCl_3$) [ppm] δ 7.35 (d, J = 8.2 Hz, 2H), 7.28 (t, J = 8.5 Hz, 2H), 7.23 (s, 1H), 7.19 (t, J = 7.8 Hz, 1H), 7.13 (d, J = 8.1 Hz, 1H), 6.94 (d, J = 7.3 Hz, 1H), 6.83 (s, 1H), 3.23 (dq, J = 15.6, 11.1 Hz, 1H), 2.89 (dq, J = 15.5, 11.2 Hz, 1H), 2.42 (s, 3H), 2.34 (s, 3H), 1.89 (s, 3H); **^{19}F-NMR** (376 MHz, $CDCl_3$) [ppm] δ –59.02 (t, J = 11.1 Hz, 3F); **^{13}C-NMR** (100 MHz, $CDCl_3$) [ppm] δ 173.6, 139.0, 137.9, 137.4, 130.9, 129.9, 128.8, 126.8, 126.5 (q, J = 277.0 Hz), 125.4, 120.6, 117.0, 48.6 (d, J = 1.0 Hz), 42.1 (q, J = 26.5 Hz), 22.8, 21.4, 21.0; **IR** (KBr) ν (cm^{-1}): 3317, 2924, 2866, 2359, 1722, 1672, 1603, 1532, 1487, 1366, 1263, 1125, 1078, 1040, 949, 777, 688, 562; **HRMS** (ESI) found: *m/z* 358.1388 $[M+Na]^+$; calcd. for $C_{19}H_{20}F_3NO$+Na 358.1395.


7.36
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7.19
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7.12
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6.83
3.29
3.26
3.25
3.23
3.22
3.21
3.19
3.17
2.95
2.92
2.91
2.89
2.88
2.86
2.85
2.82
2.42
2.34
1.89
CF3
H3C
N
H
O
3h
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0.94
1.08
1.00
0.97
0.88
1.05
1.09
3.00
2.97
3.03
8.0
7.5
7.0
6.5
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5.5
5.0
4.5
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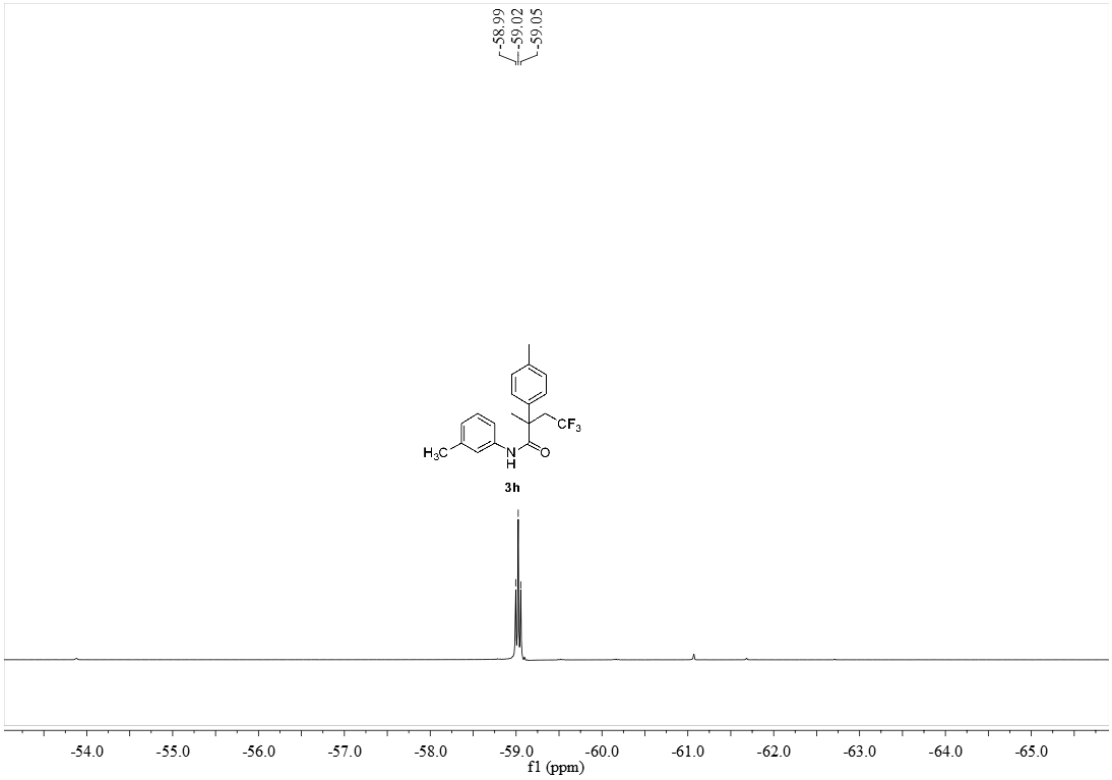
3.5
3.0
2.5
2.0
f1 (ppm)


-58.99
-59.02
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CF3
H3C
N
H
O
3h
-54.0
-55.0
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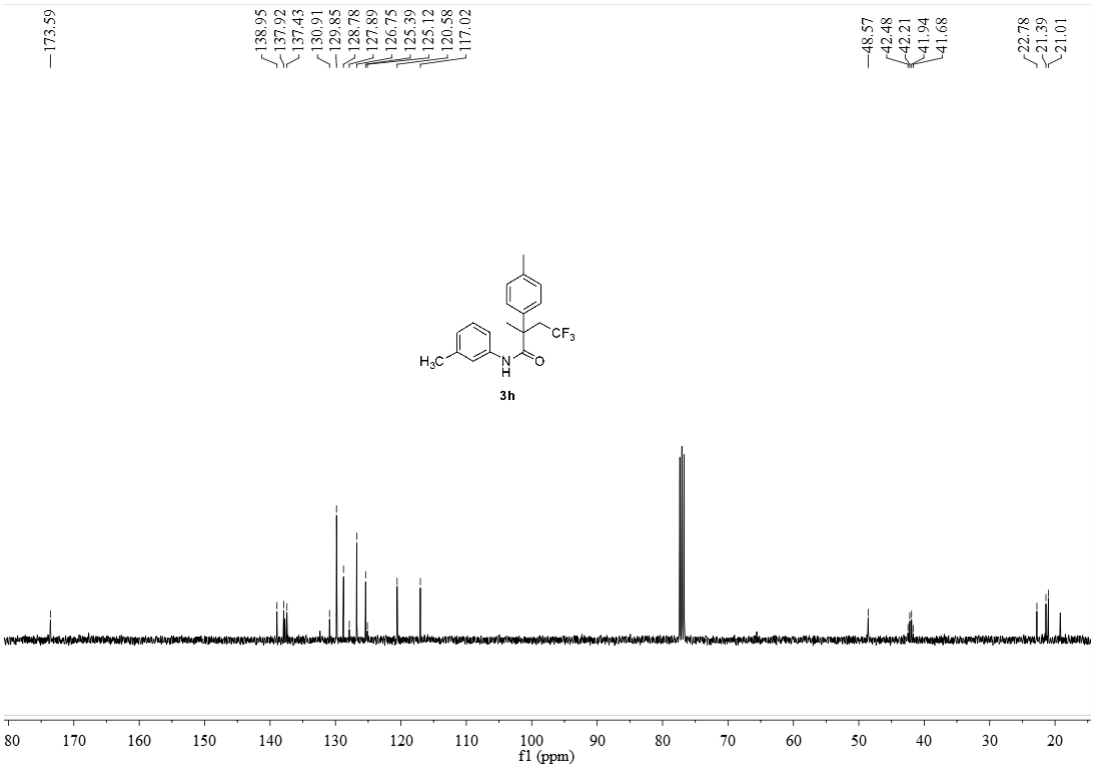
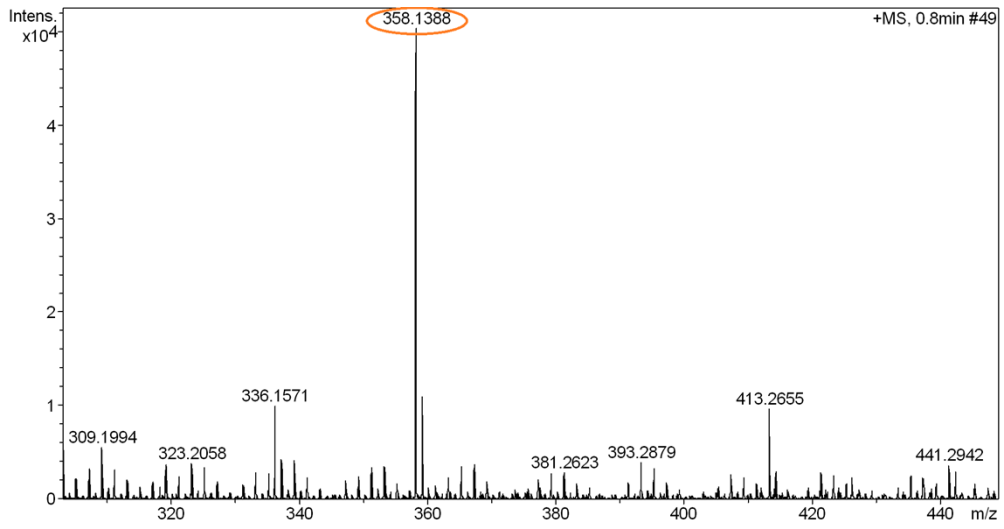
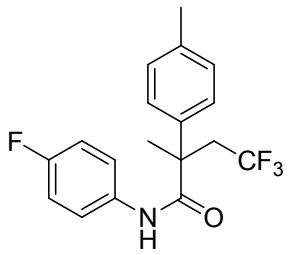
-59.0
-60.0
-61.0
-62.0
-63.0
-64.0
-65.0
f1 (ppm)





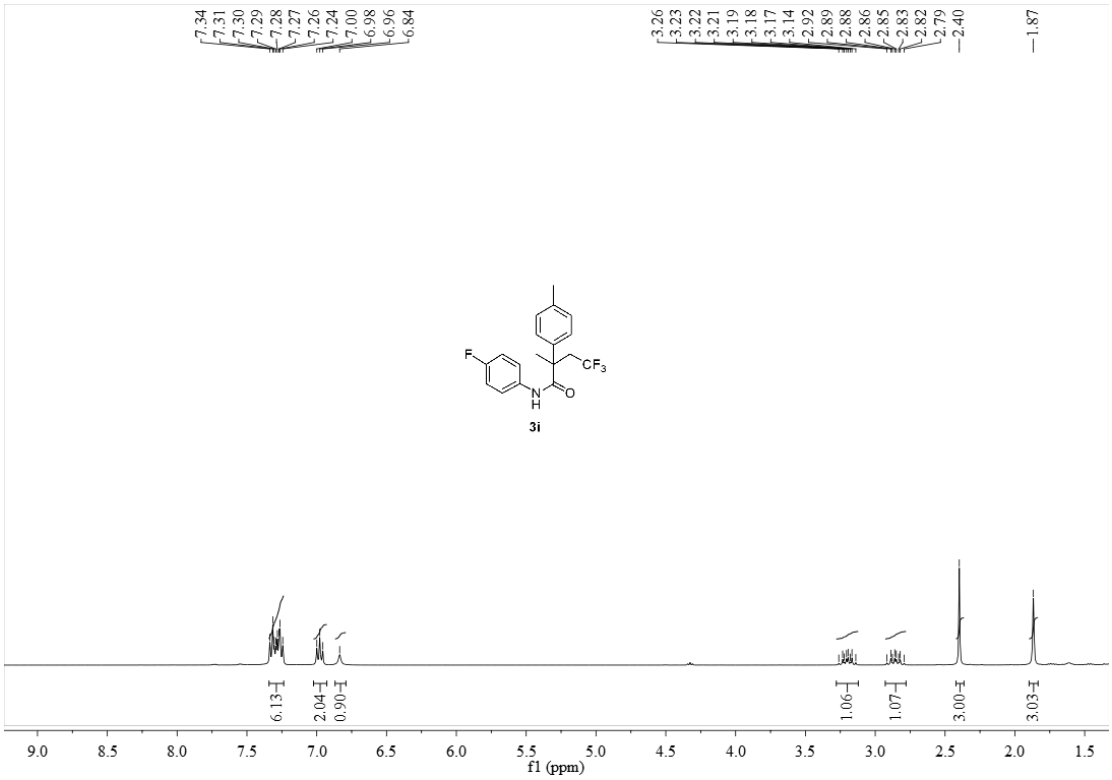


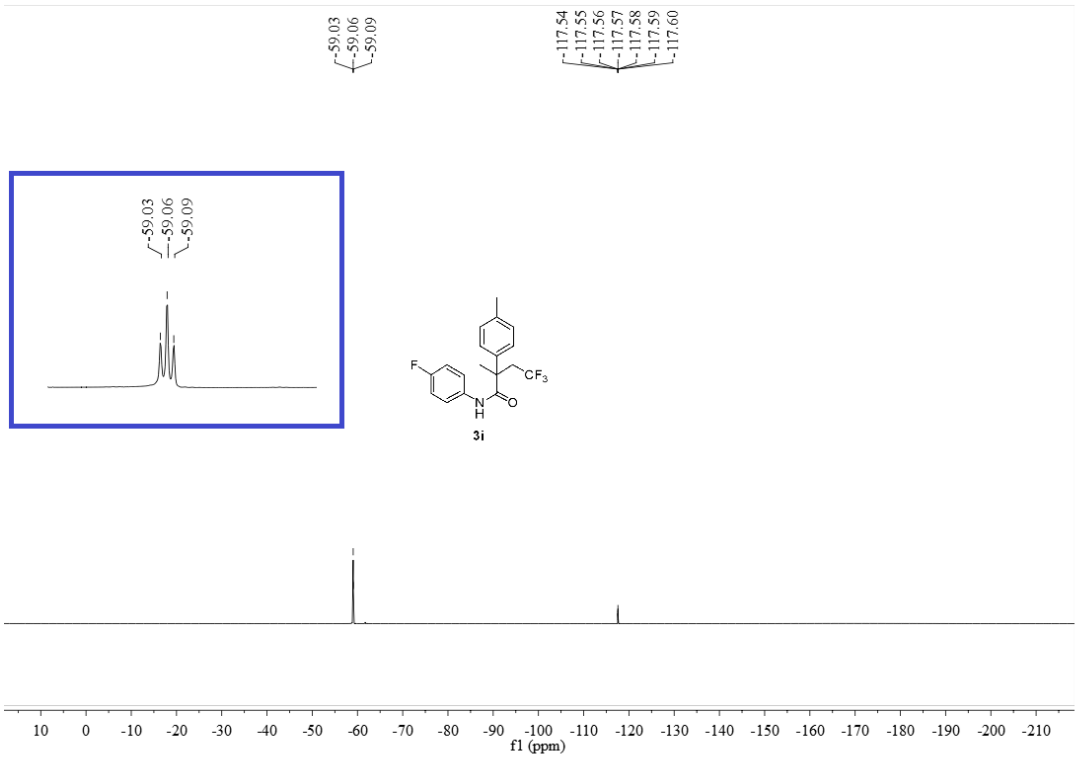
3i

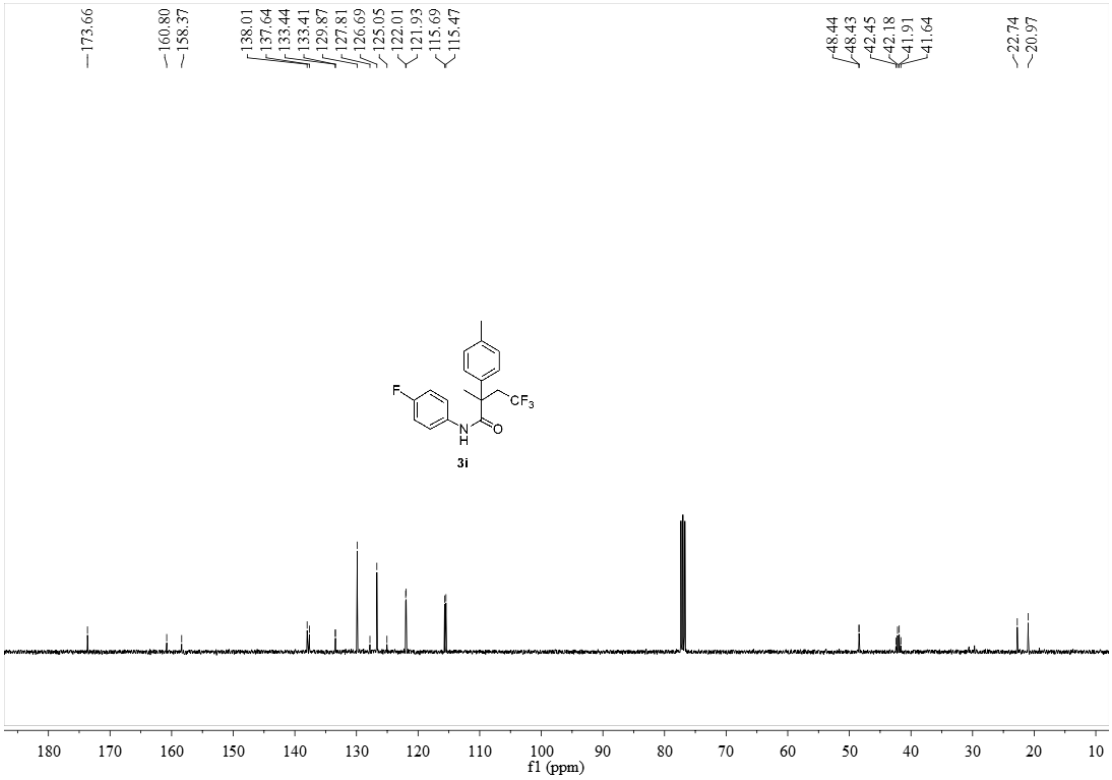
4,4,4-trifluoro-N-(4-fluorophenyl)-2-methyl-2-(p-tolyl)butanamide (3i)

M: 44.1 mg, **Yield:** 65%; **^{1}H-NMR** (400 MHz, $CDCl_3$) [ppm] δ 7.29 (ddd, J = 15.4, 11.4, 8.2 Hz,

6H), 6.98 (t, J = 8.6 Hz, 2H), 6.84 (s, 1H), 3.20 (dq, J = 15.5, 11.0 Hz, 1H), 2.85 (dq, J = 15.5, 11.2 Hz, 1H), 2.40 (s, 3H), 1.87 (s, 3H); **^{19}F-NMR** (376 MHz, $CDCl_3$) [ppm] δ –59.06 (t, J = 11.1 Hz, 3F), –117.51 to –117.63 (m, F); **^{13}C-NMR** (100 MHz, $CDCl_3$) [ppm] δ 173.7, 159.6 (d, $^1J_{CF}$ = 243.0 Hz), 138.0, 137.6, 134.4 (d, $^4J_{CF}$ = 3.0 Hz), 129.9, 126.7, 126.4 (q, J = 276.0 Hz), 122.0 (d, $^3J_{CF}$ = 8.0 Hz), 115.6 (d, $^2J_{CF}$ = 22.0 Hz), 48.4 (d, J = 1.0 Hz), 42.0 (q, J = 27.0 Hz), 22.7, 21.0; **IR** (KBr) ν (cm^{-1}): 3317, 2925, 2857, 2362, 1662, 1612, 1513, 1405, 1367, 1306, 1261, 1216, 1156, 1124, 1080, 1042, 954, 829, 784, 666, 624, 515; **HRMS** (ESI) found: *m/z* 362.1137 $[M+Na]^+$; calcd. for $C_{18}H_{17}F_4NO$+Na 362.1144.




-59.03
-59.06
-59.09
-117.54
-117.55
-117.56
-117.57
-117.58
-117.59
-117.60
F
CF3
N
H
O
3i
f1 (ppm)


173.66
160.80
158.37
138.01
137.64
133.44
133.41
129.87
127.81
126.69
125.05
122.01
121.93
115.69
115.47
48.44
48.43
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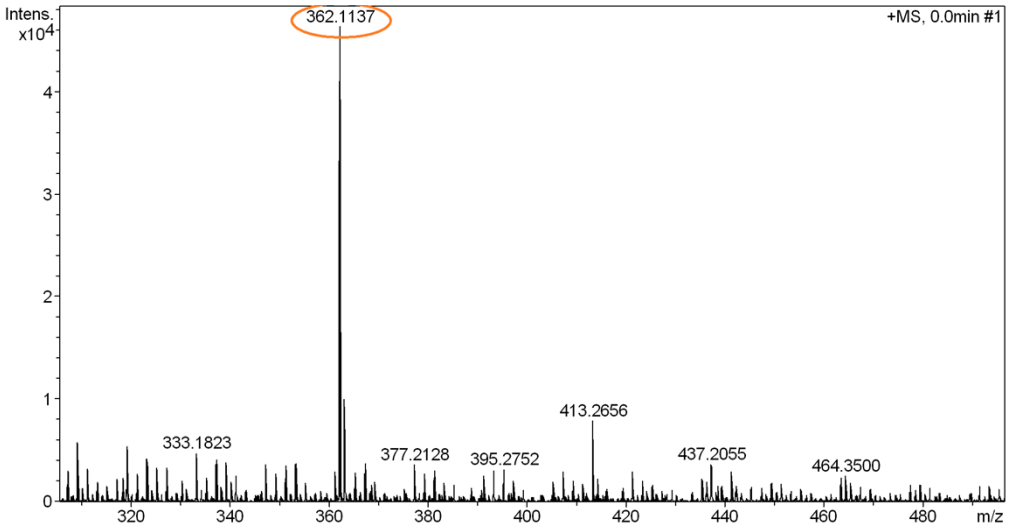
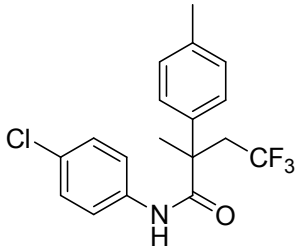
22.74
20.97
F
CF3
N
H
O
3i
f1 (ppm)

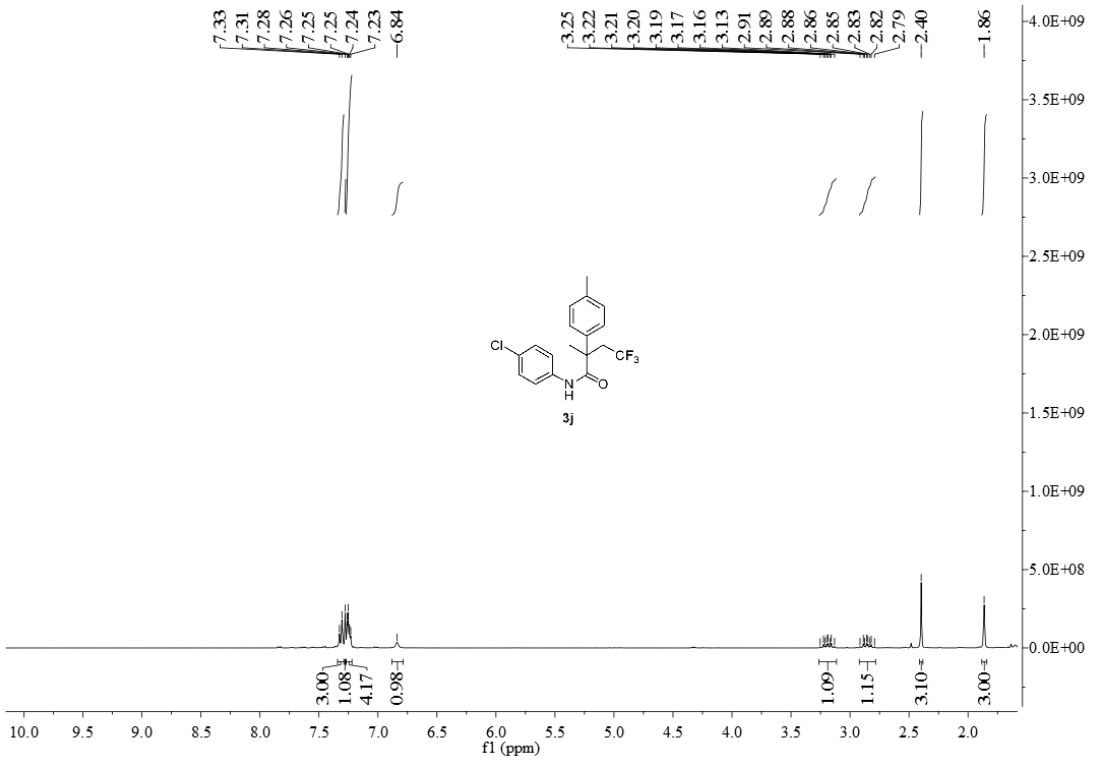


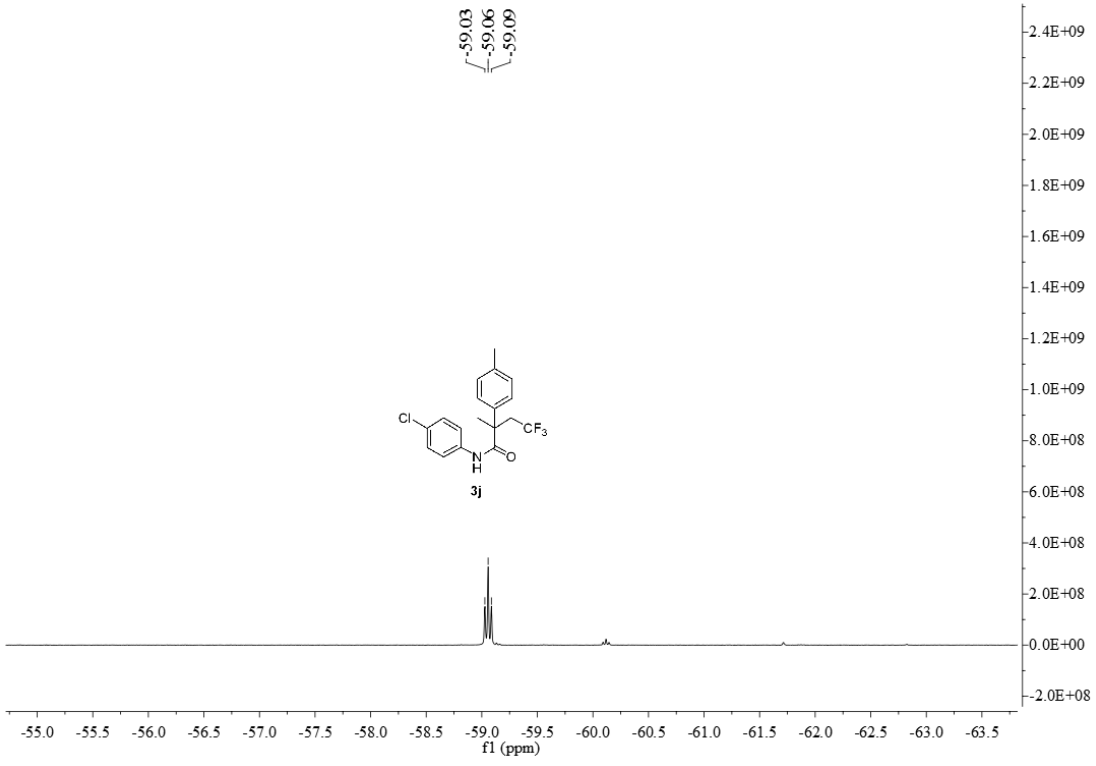


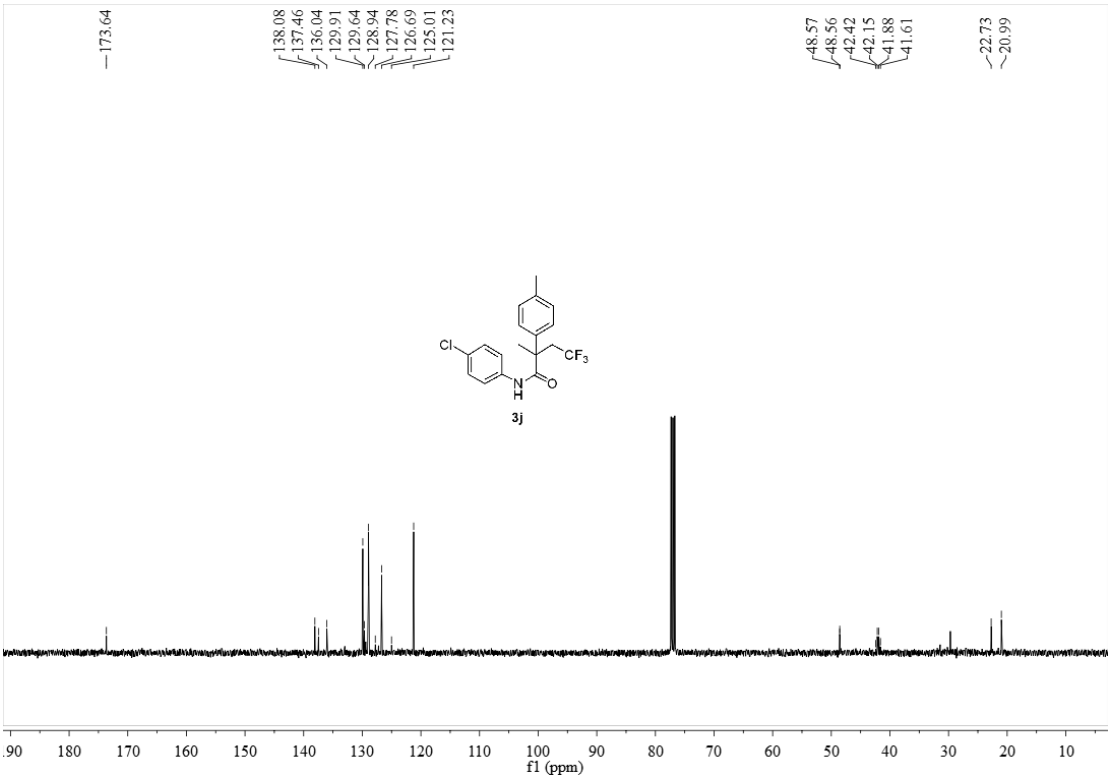
3j

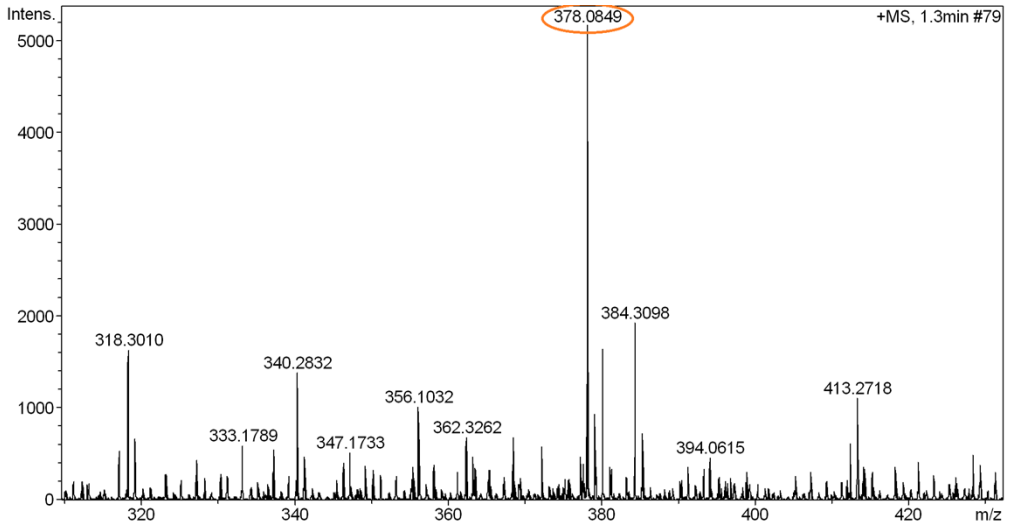
N-(4-chlorophenyl)-4,4,4-trifluoro-2-methyl-2-(p-tolyl)butanamide (3j)

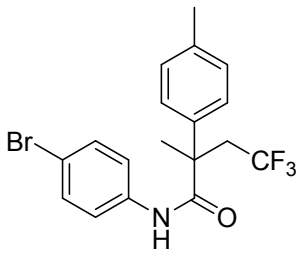
M: 43.4 mg, **Yield:** 61%; **^{1}H-NMR** (400 MHz, $CDCl_3$) [ppm] δ 7.32 (d, J = 8.3 Hz, 3H), 7.28 (s, 1H), 7.27 – 7.22 (m, 4H), 6.84 (s, 1H), 3.19 (dq, J = 15.6, 11.0 Hz, 1H), 2.85 (dq, J = 15.6, 11.2 Hz, 1H), 2.40 (s, 3H), 1.86 (s, 3H); **^{19}F-NMR** (376 MHz, $CDCl_3$) [ppm] δ –59.06 (t, J = 11.1 Hz, 3F); **^{13}C-NMR** (100 MHz, $CDCl_3$) [ppm] δ 173.6, 138.1, 137.5, 136.0, 129.9, 129.6, 128.9, 126.7, 126.4 (q, J = 276.0 Hz), 121.2, 48.6 (d, J = 1.0 Hz), 42.0 (q, J = 27.0 Hz), 22.7, 21.0; **IR** (KBr) ν (cm^{-1}): 3290, 2923, 2856, 2359, 1662, 1594, 1516, 1368, 1306, 1259, 1124, 1083, 1040, 954, 820, 756, 668, 604, 570; **HRMS** (ESI) found: *m/z* 378.0849 $[M+Na]^+$; calcd. for $C_{18}H_{17}ClF_3NO$+Na 378.0849.


7.33
7.31
7.28
7.26
7.25
7.25
7.24
7.23
6.84
3.25
3.22
3.21
3.20
3.19
3.17
3.16
3.13
2.91
2.89
2.88
2.86
2.85
2.83
2.82
2.79
2.40
1.86
Cl
CF3
N
H
O
3j
3.00
1.08
4.17
0.98
1.09
1.15
3.10
3.00
f1 (ppm)


-59.03
-59.06
-59.09
Cl
CF3
N
H
O
3j
f1 (ppm)





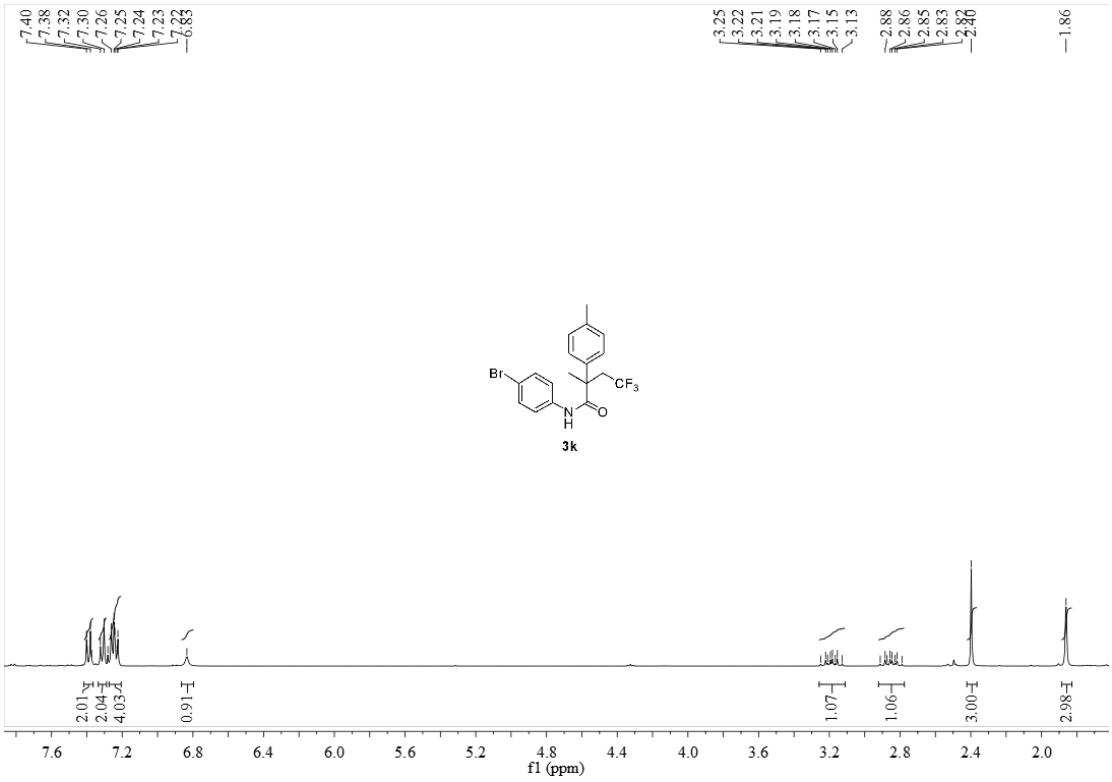


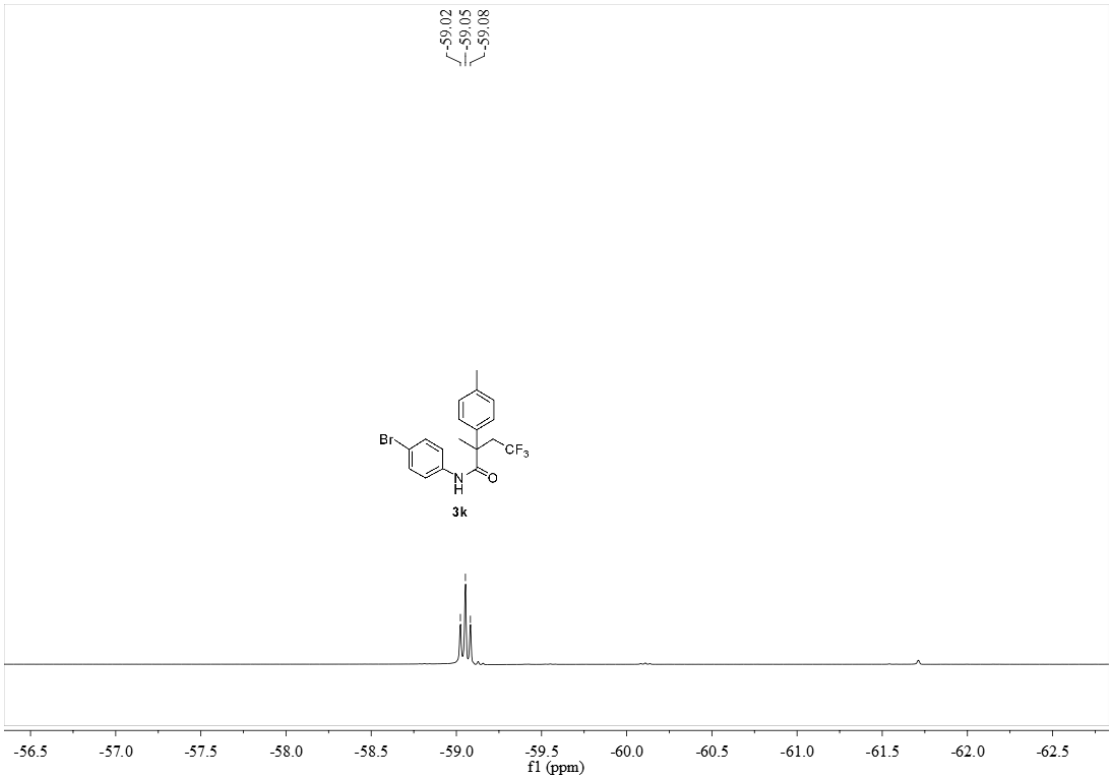
3k

N-(4-bromophenyl)-4,4,4-trifluoro-2-methyl-2-(p-tolyl)butanamide (3k)

M: 49.6 mg, **Yield:** 62%; **^{1}H-NMR** (400 MHz, $CDCl_3$) [ppm] δ 7.39 (dd, J = 9.3, 2.3 Hz, 2H),

7.31 (d, J = 8.3 Hz, 2H), 7.27–7.21 (m, 4H), 6.83 (s, 1H), 3.19 (dq, J = 15.6, 11.1 Hz, 1H), 2.85 (dq, J = 15.6, 11.2 Hz, 1H), 2.40 (s, 3H), 1.86 (s, 3H); **^{19}F-NMR** (376 MHz, $CDCl_3$) [ppm] δ –59.05 (t, J = 11.1 Hz, 3F); **^{13}C-NMR** (100 MHz, $CDCl_3$) [ppm] δ 173.6, 138.1, 137.4, 136.6, 131.9, 129.9, 126.7, 126.4 (q, J = 276.0 Hz), 121.5, 117.2, 48.6 (d, J = 2.0 Hz), 42.0 (q, J = 28.0 Hz), 22.7, 21.0; **IR** (KBr) ν (cm^{-1}): 3306, 2928, 2359, 1667, 1592, 1516, 1370, 1307, 1260, 1125, 1076, 1041, 951, 817, 752, 658, 590, 555; **HRMS** (ESI) found: *m/z* 422.0335 $[M+Na]^+$; calcd. for $C_{18}H_{17}BrF_3NO$+Na 422.0344.




-59.02
-59.05
-59.08
Br
CF3
N
H
O
3k
-56.5
-57.0
-57.5
-58.0
-58.5
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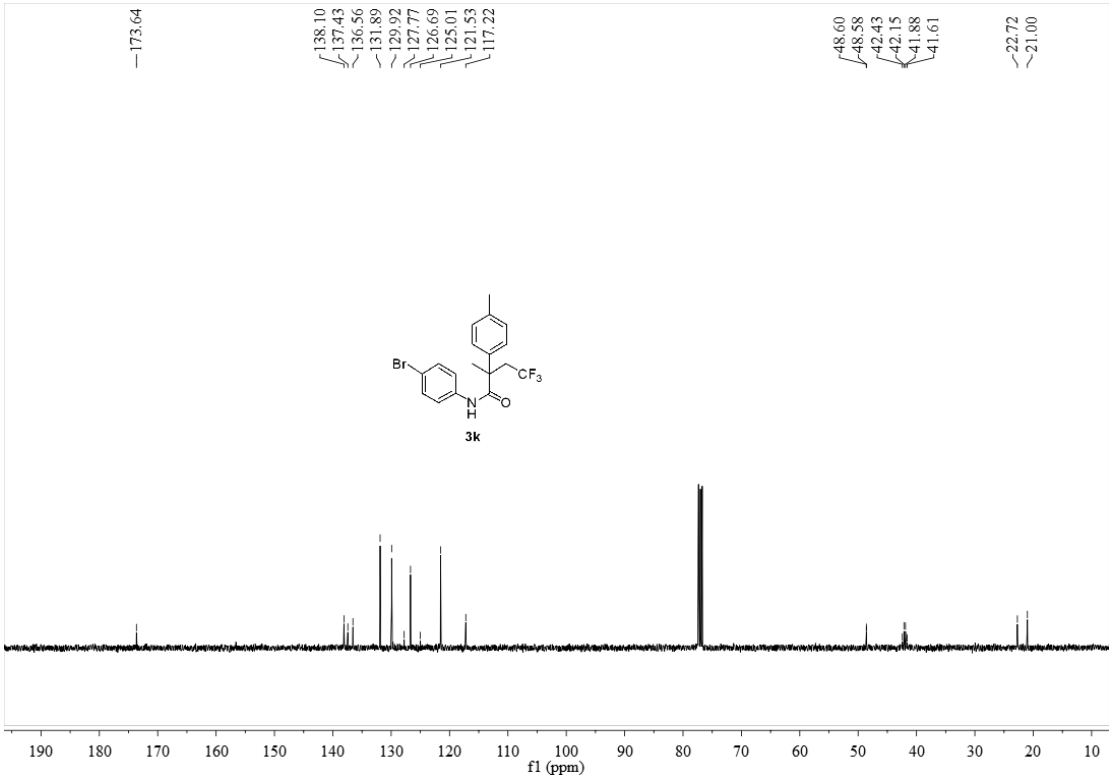
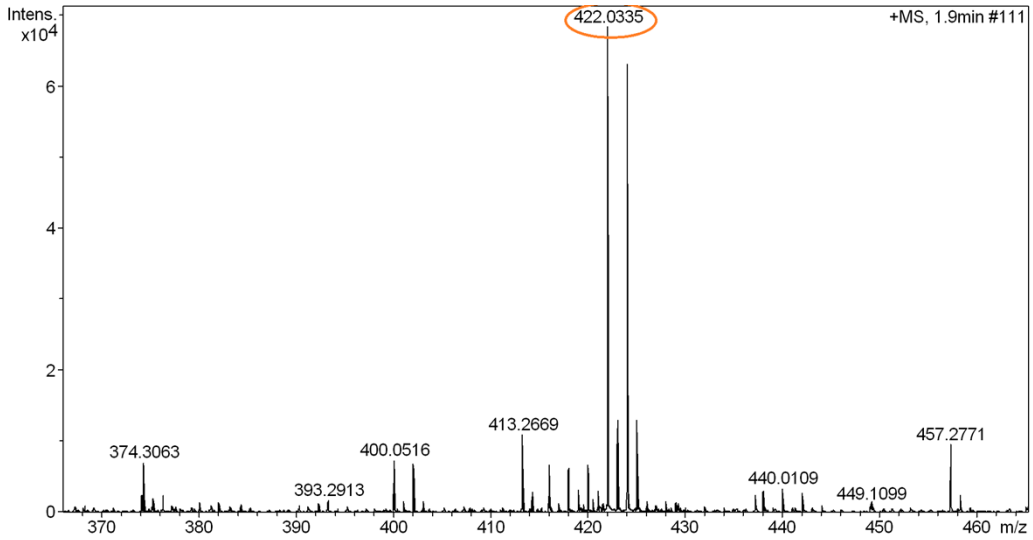
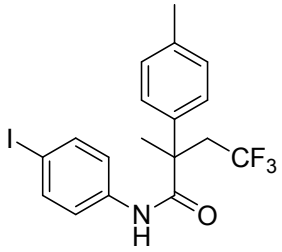
-59.5
-60.0
-60.5
-61.0
-61.5
-62.0
-62.5
f1 (ppm)


173.64
138.10
137.43
136.56
131.89
129.92
127.77
126.69
125.01
121.53
117.22
48.60
48.58
42.43
42.15
41.88
41.61
22.72
21.00
Br
CF3
N
H
O
3k
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

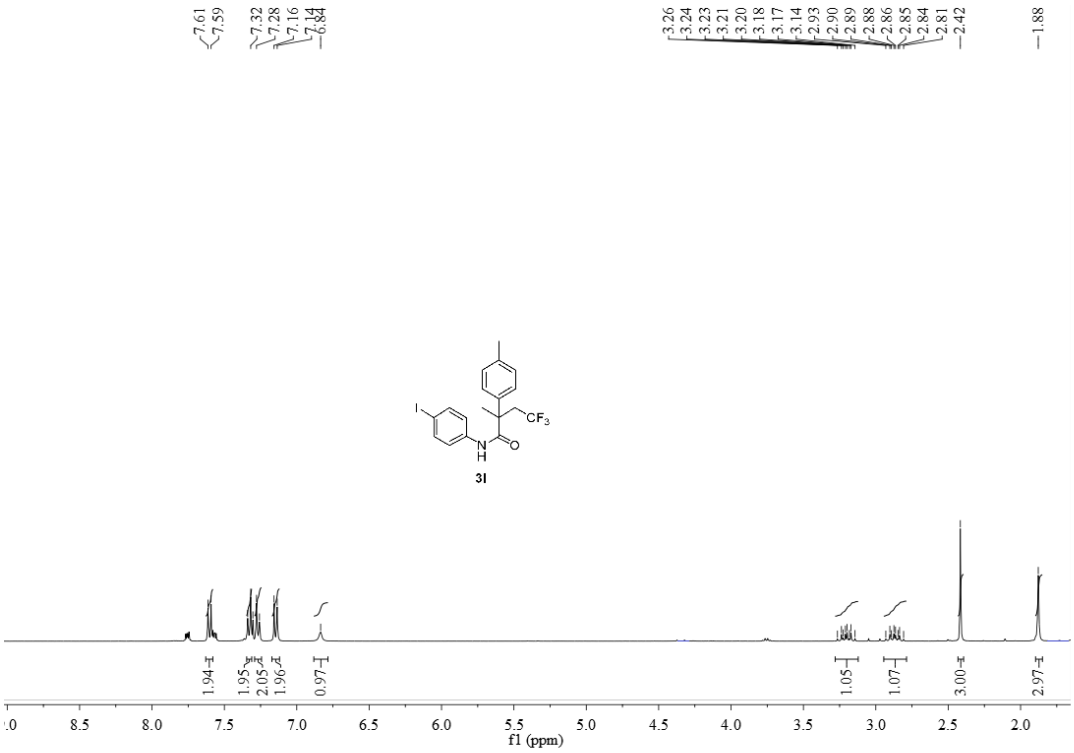


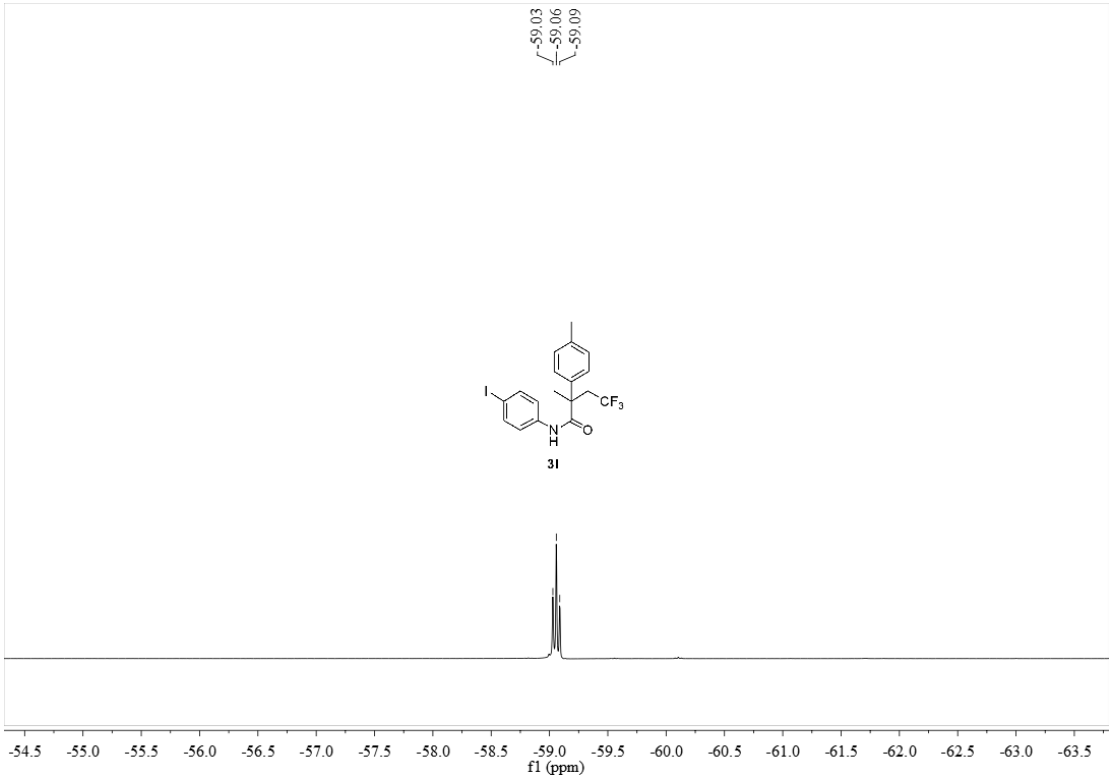


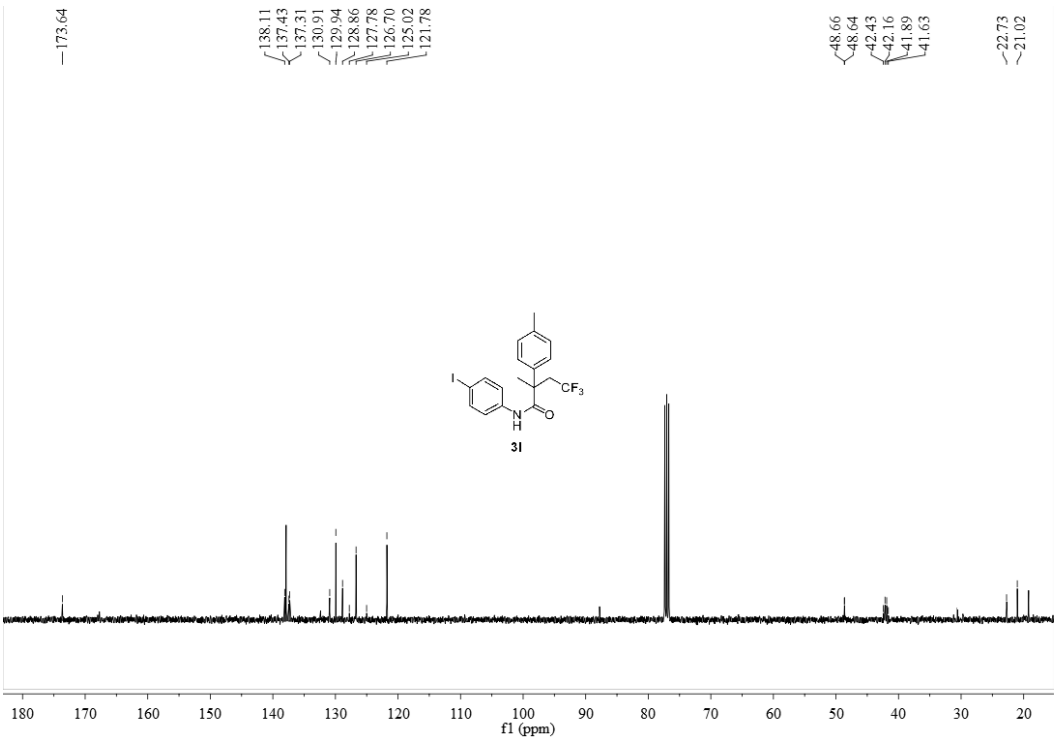
3l

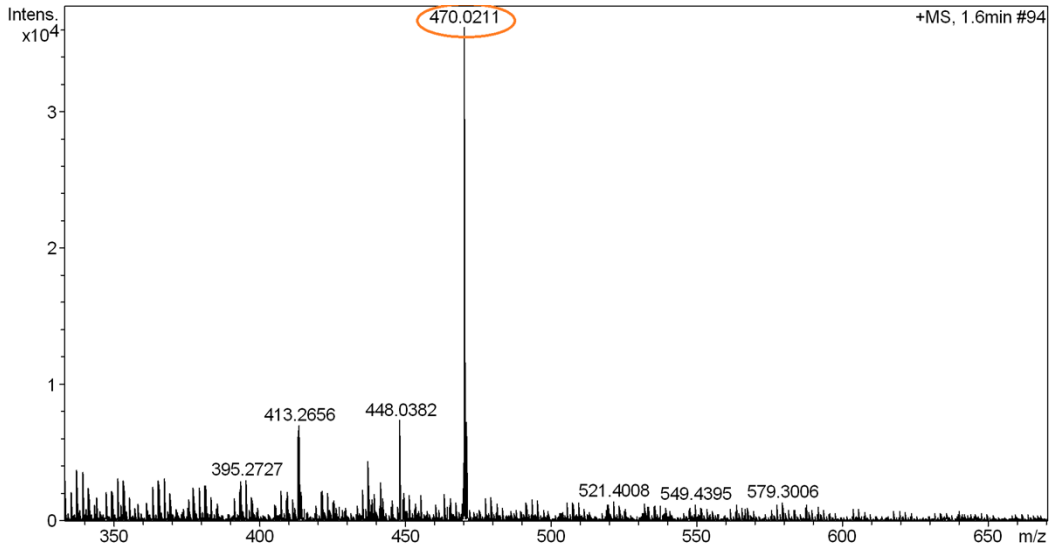
4,4,4-trifluoro-N-(4-iodophenyl)-2-methyl-2-(p-tolyl)butanamide (3l)

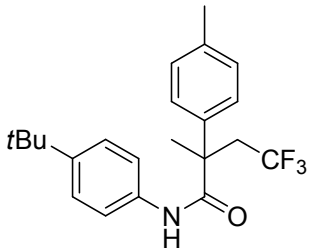
M: 40.2 mg, **Yield:** 45%; **^{1}H-NMR** (400 MHz, $CDCl_3$) [ppm] δ 7.60 (d, J = 8.8 Hz, 2H), 7.33 (d, J = 8.3 Hz, 2H), 7.27 (d, J = 8.1 Hz, 2H), 7.15 (d, J = 8.8 Hz, 2H), 6.84 (s, 1H), 3.20 (dq, J = 15.6, 11.0 Hz, 1H), 2.87 (dq, J = 15.6, 11.2 Hz, 1H), 2.42 (s, 3H), 1.88 (s, 3H); **^{19}F-NMR** (376 MHz, $CDCl_3$) [ppm] δ –59.06 (t, J = 11.1 Hz, 3F); **^{13}C-NMR** (100 MHz, $CDCl_3$) [ppm] δ 173.6, 138.1, 137.4, 137.3, 130.9, 129.9, 127.9, 126.7, 126.4 (q, J = 276.0 Hz), 121.8, 48.6 (d, J = 2.0 Hz), 42.0 (q, J = 26.5 Hz), 22.7, 21.0; **IR** (KBr) ν (cm^{-1}): 3310, 2958, 2871, 2360, 1723, 1685, 1588, 1514, 1366, 1268, 1126, 1075, 951, 817, 747, 664; **HRMS** (ESI) found: *m/z* 470.0211 $[M+Na]^+$; calcd. for $C_{18}H_{17}IF_3NO$+Na 470.0205.


7.61
7.59
7.32
7.28
7.16
7.14
6.84
3.26
3.24
3.23
3.21
3.20
3.18
3.17
3.14
2.93
2.90
2.89
2.88
2.86
2.85
2.84
2.81
2.42
1.88
CF3
I
N
H
O
3l
1.94
1.95
2.05
1.96
0.97
1.05
1.07
3.00
2.97
f1 (ppm)


59.03
59.06
59.09
CF3
I
N
H
O
3l
f1 (ppm)





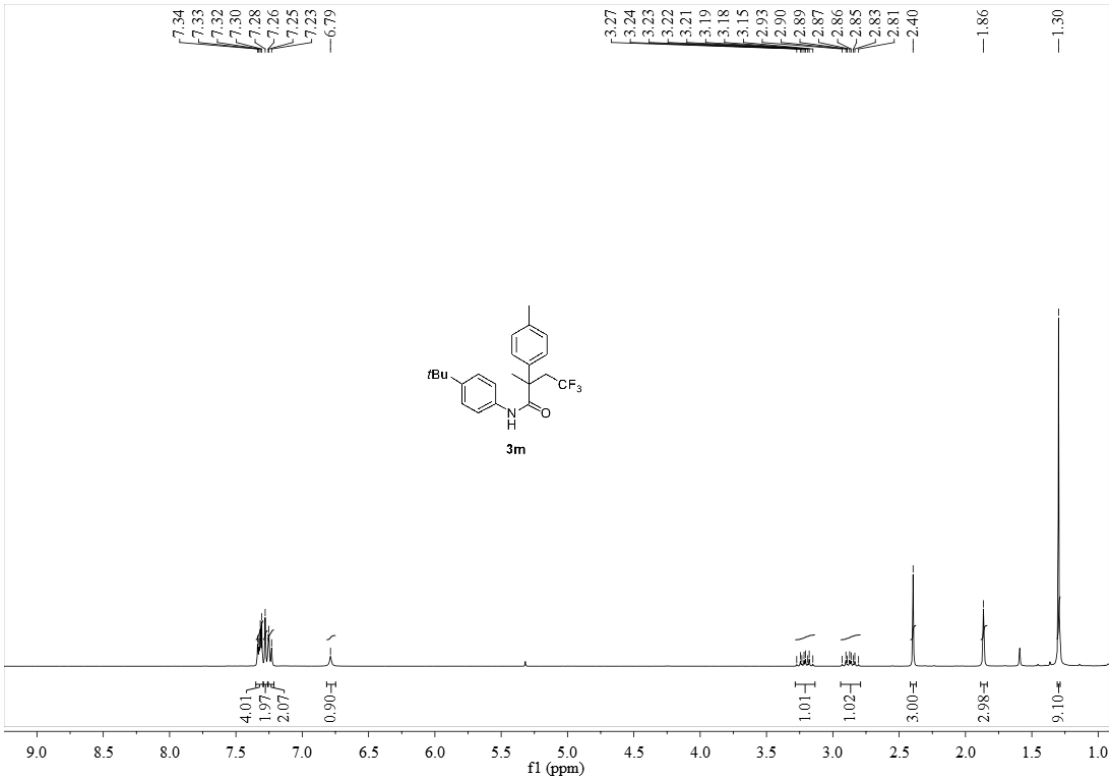


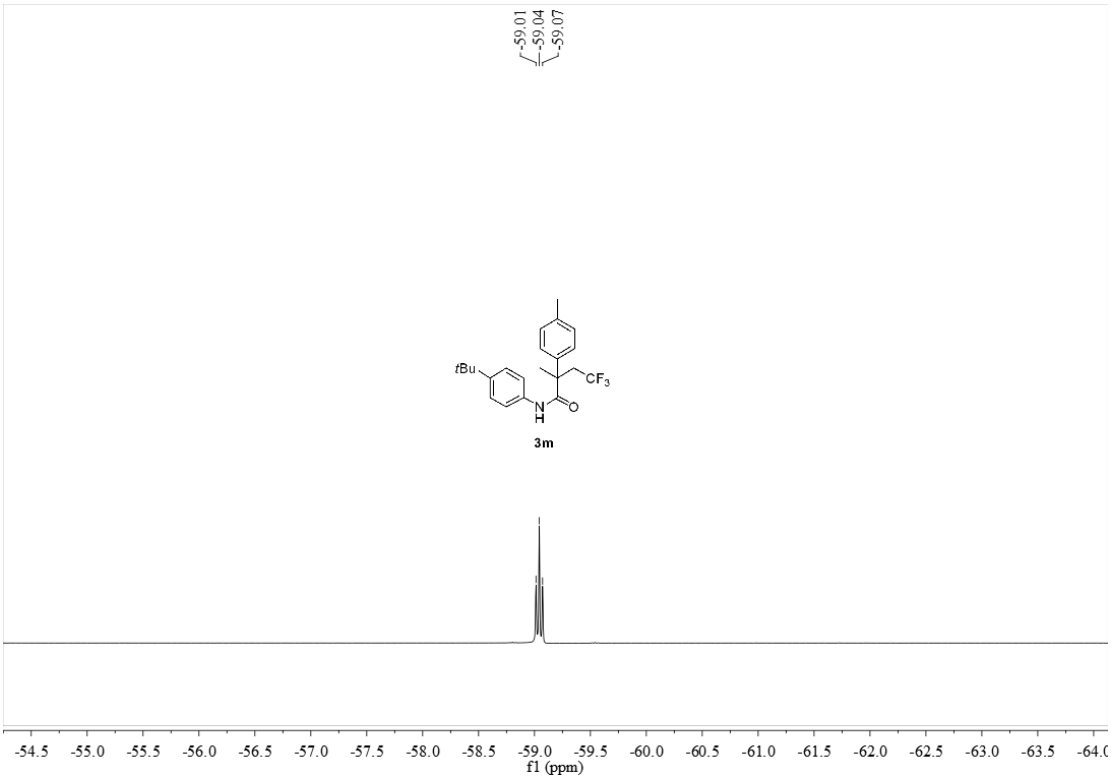
3m

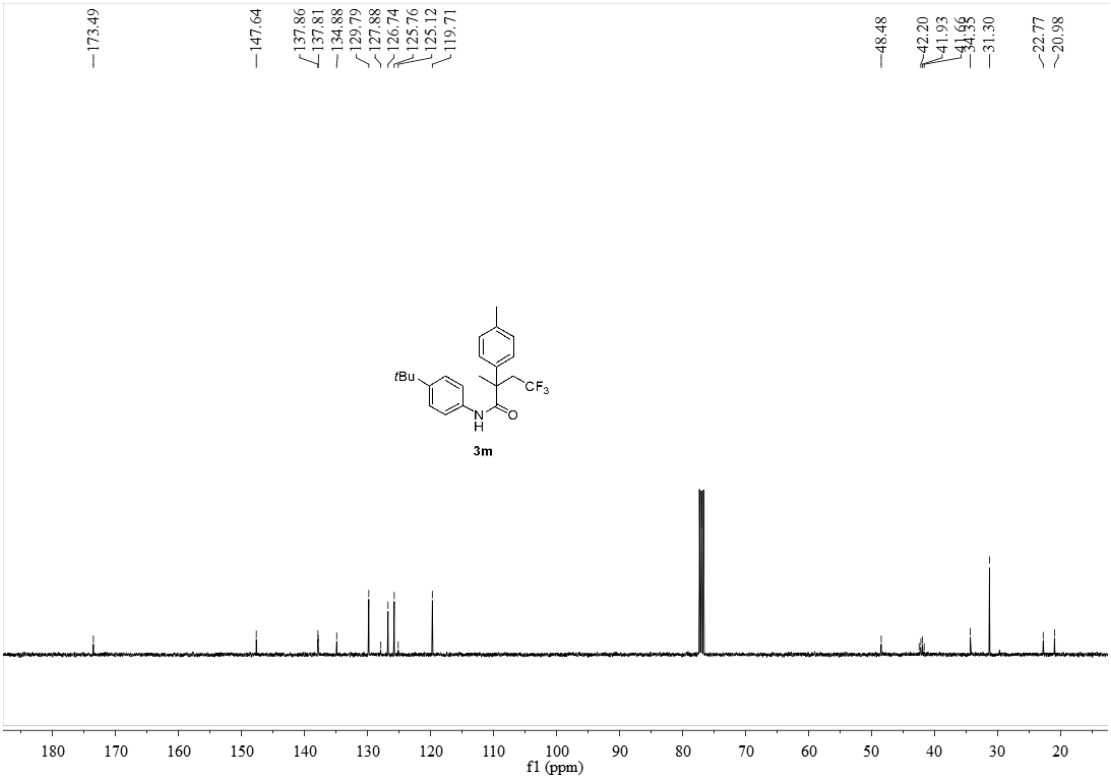
N-(4-(tert-butyl)phenyl)-4,4,4-trifluoro-2-methyl-2-(p-tolyl)butanamide (3m)

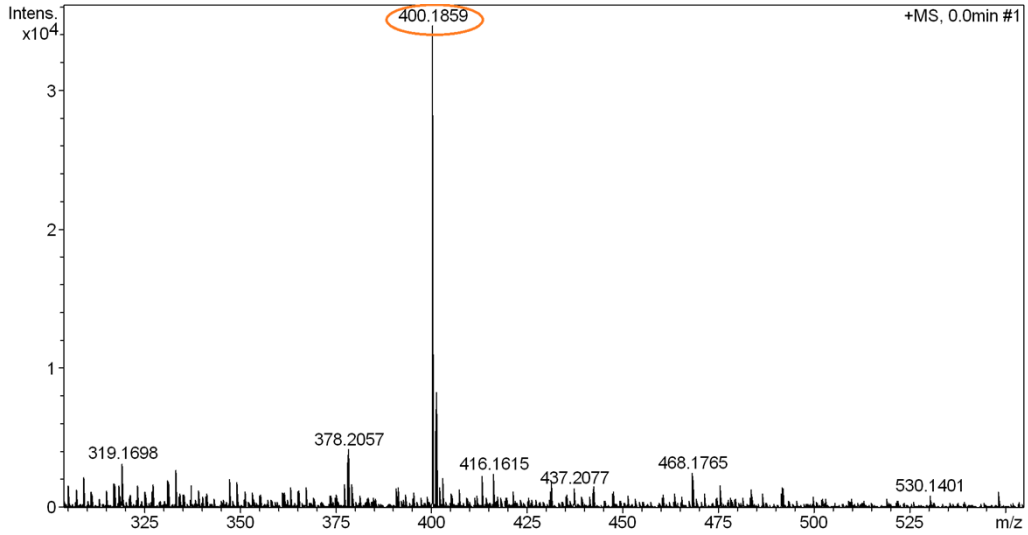
M: 43.0 mg, **Yield:** 57%; **^{1}H-NMR** (400 MHz, $CDCl_3$) [ppm] δ 7.32 (dd, J = 8.5, 4.3 Hz, 4H),

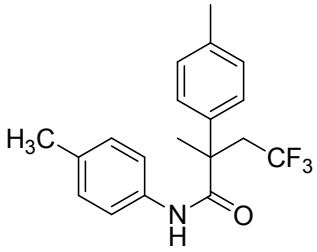
7.28 (s, 2H), 7.24 (d, J = 8.2 Hz, 2H), 6.79 (s, 1H), 3.21 (dq, J = 15.6, 11.1 Hz, 1H), 2.87 (dq, J = 15.6, 11.2 Hz, 1H), 2.40 (s, 3H), 1.86 (s, 3H), 1.30 (s, 9H); **^{19}F-NMR** (376 MHz, $CDCl_3$) [ppm] δ –59.04 (t, J = 11.1 Hz, 3F); **^{13}C-NMR** (100 MHz, $CDCl_3$) [ppm] δ 173.5, 147.6, 137.9, 137.8, 134.9, 129.8, 126.7, 126.5 (q, J = 276.0 Hz), 125.8, 119.7, 48.5 (d, J = 1.0 Hz), 42.1 (q, J = 26.5 Hz), 34.4, 31.3, 22.8, 21.0; **IR** (KBr) ν (cm^{-1}): 3306, 2960, 2868, 2362, 2336, 1664, 1595, 1520, 1404, 1365, 1317, 1261, 1120, 1080, 1040, 952, 822, 668, 614, 555; **HRMS** (ESI) found: *m/z* 400.1859 $[M+Na]^+$; calcd. for $C_{22}H_{26}F_3NO$+Na 400.1864.




-59.01
-59.04
-59.07
tBu
CF3
N
H
O
3m
-54.5 -55.0 -55.5 -56.0 -56.5 -57.0 -57.5 -58.0 -58.5 -59.0 -59.5 -60.0 -60.5 -61.0 -61.5 -62.0 -62.5 -63.0 -63.5 -64.0
f1 (ppm)


173.49
147.64
137.86
137.81
134.88
129.79
127.88
126.74
125.76
125.12
119.71
48.48
42.20
41.93
41.66
34.35
31.30
22.77
20.98
tBu
CF3
N
H
O
3m
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
f1 (ppm)

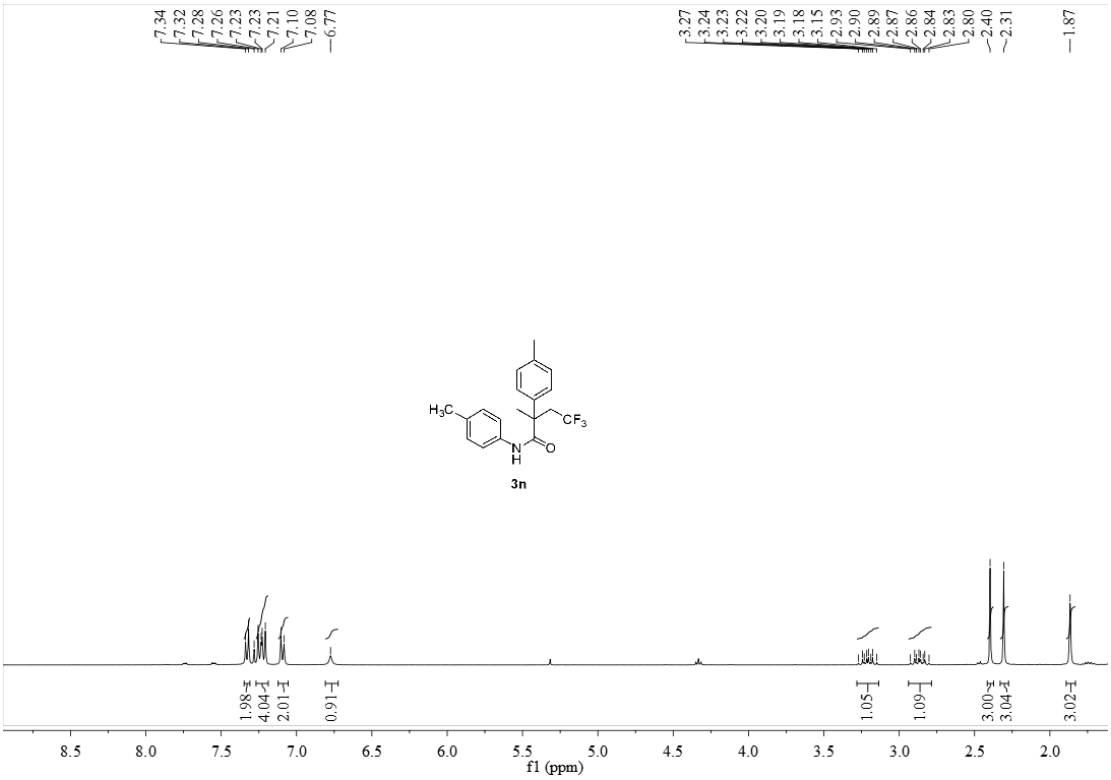


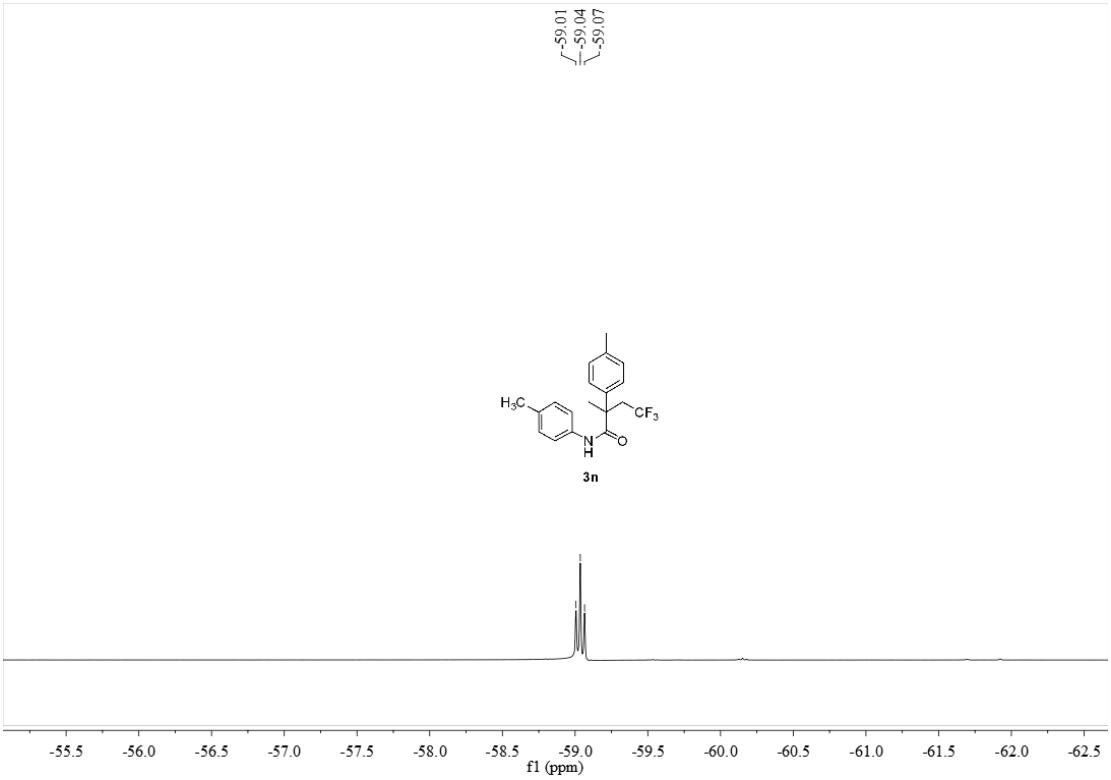


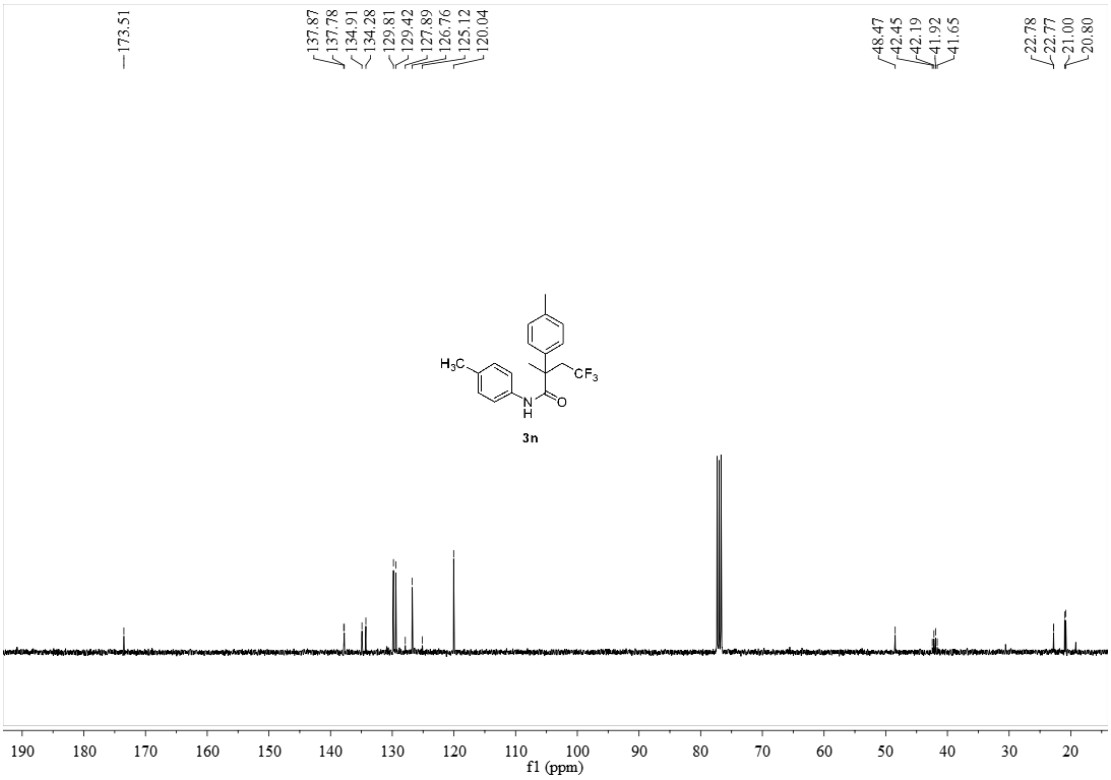
3n

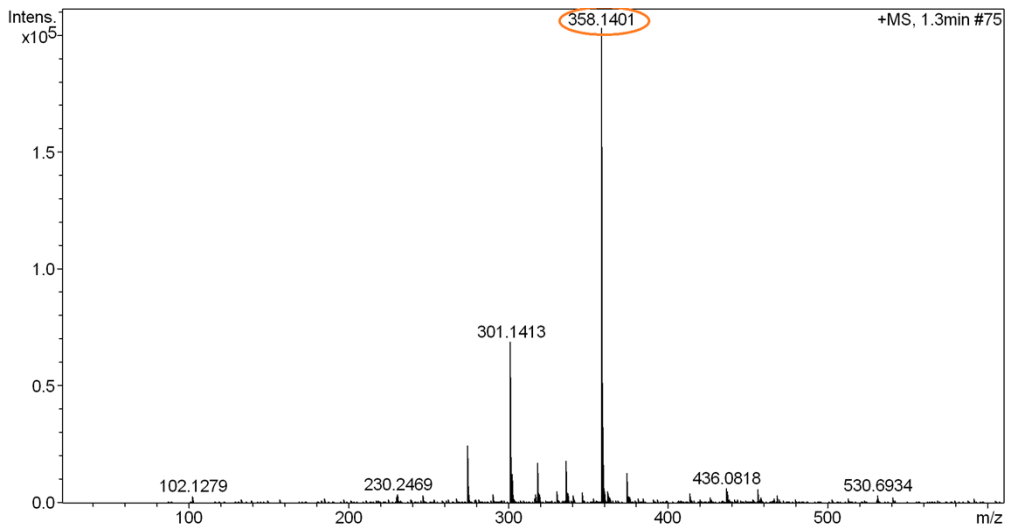
4,4,4-trifluoro-2-methyl-N,2-di-p-tolylbutanamide (3n)

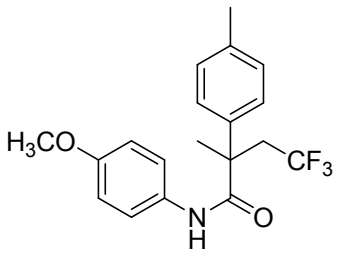
M: 42.3 mg, **Yield:** 63%; **^{1}H-NMR** (400 MHz, $CDCl_3$) [ppm] δ 7.33 (d, J = 8.2 Hz, 2H), 7.23 (dd, J = 10.9, 8.3 Hz, 4H), 7.09 (d, J = 8.3 Hz, 2H), 6.77 (s, 1H), 3.21 (dq, J = 15.6, 11.1 Hz, 1H), 2.86 (dq, J = 15.6, 11.3 Hz, 1H), 2.40 (s, 3H), 2.31 (s, 3H), 1.87 (s, 3H); **^{19}F-NMR** (376 MHz, $CDCl_3$) [ppm] δ –59.04 (t, J = 11.1 Hz, 3F); **^{13}C-NMR** (100 MHz, $CDCl_3$) [ppm] δ 173.5, 137.9, 137.8, 134.9, 134.3, 129.8, 129.4, 126.8, 126.5 (q, J = 277.0 Hz), 120.0, 48.5 (d, J = 1.0 Hz), 42.1 (q, J = 26.5 Hz), 27.8 (d, J = 1.0 Hz), 21.0, 20.8; **IR** (KBr) ν (cm^{-1}): 3334, 2928, 2870, 2360, 1668, 1599, 1520, 1462, 1367, 1308, 1260, 1124, 1079, 1040, 950, 813, 623, 513; **HRMS** (ESI) found: *m/z* 358.1401 $[M+Na]^+$; calcd. for $C_{19}H_{20}F_3NO$+Na 358.1395.


7.34
7.32
7.28
7.26
7.23
7.23
7.21
7.10
7.08
6.77
3.27
3.24
3.23
3.22
3.20
3.19
3.18
3.15
2.93
2.90
2.89
2.87
2.86
2.84
2.83
2.80
2.40
2.31
1.87
H3C
CF3
N
H
O
3n
1.98
4.04
2.01
0.91
1.05
1.09
3.00
3.04
3.02
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
f1 (ppm)


59.01
59.04
59.07
H3C
CF3
N
H
O
3n
-55.5
-56.0
-56.5
-57.0
-57.5
-58.0
-58.5
-59.0
-59.5
-60.0
-60.5
-61.0
-61.5
-62.0
-62.5
f1 (ppm)





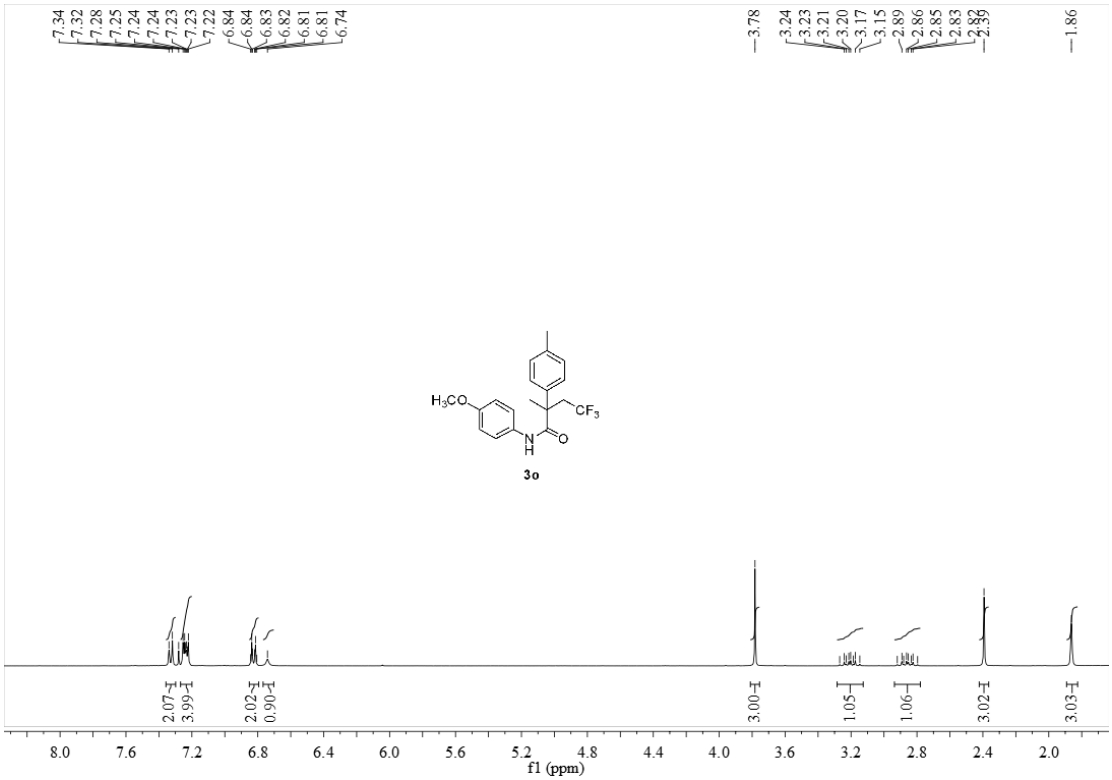


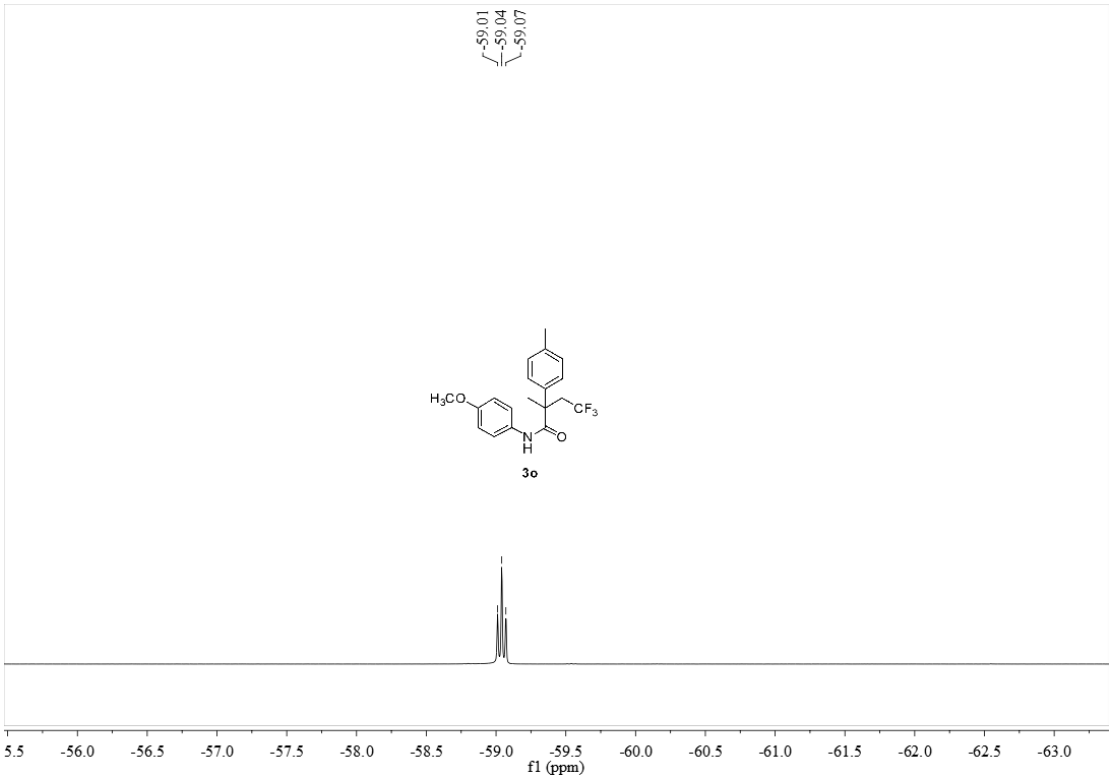
3o

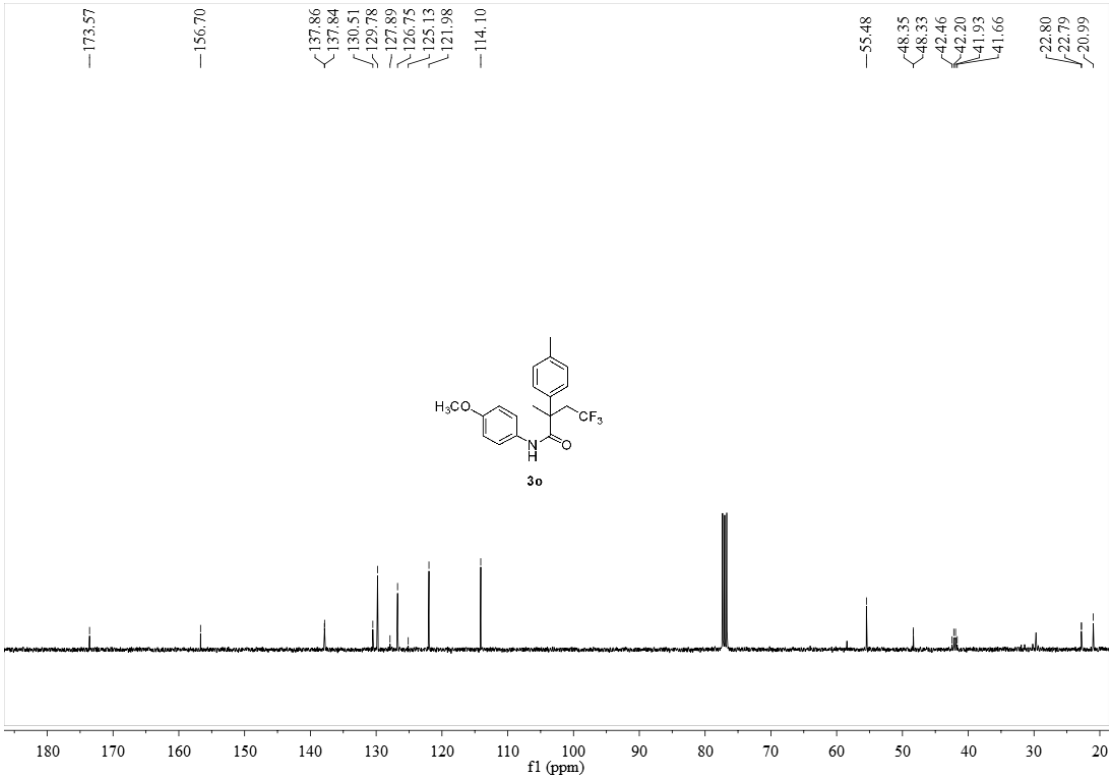
4,4,4-trifluoro-N-(4-methoxyphenyl)-2-methyl-2-(p-tolyl)butanamide (3o)

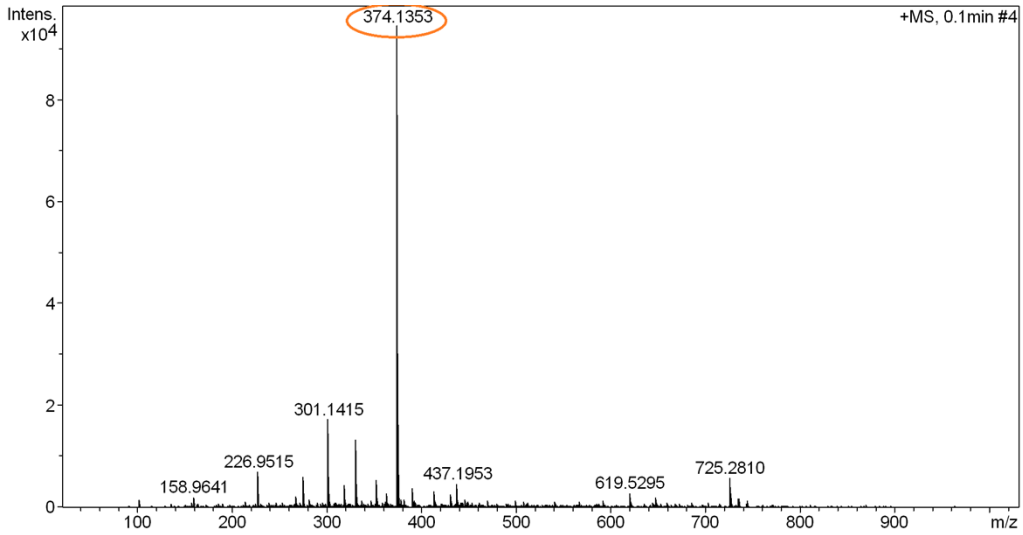
M: 41.5 mg, **Yield:** 59%; **^{1}H-NMR** (400 MHz, $CDCl_3$) [ppm] δ 7.33 (d, J = 8.3 Hz, 2H), 7.27–

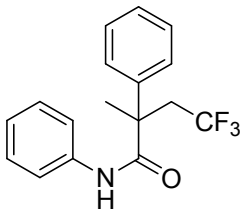
7.20 (m, 4H), 6.85–6.79 (m, 2H), 6.74 (s, 1H), 3.78 (s, 3H), 3.21 (dq, J = 15.6, 11.1 Hz, 1H), 2.86 (dq, J = 15.6, 11.2 Hz, 1H), 2.39 (s, 3H), 1.86 (s, 3H); **^{19}F-NMR** (376 MHz, $CDCl_3$) [ppm] δ – 59.04 (t, J = 11.2 Hz, 3F); **^{13}C-NMR** (100 MHz, $CDCl_3$) [ppm] δ 173.6, 156.7, 137.9, 137.8, 130.5, 129.8, 126.8, 126.5 (q, J = 276.0 Hz), 122.0, 114.1, 55.5, 48.3 (d, J = 2.0 Hz), 42.1 (q, J = 26.5 Hz), 22.8 (d, J = 1.0 Hz), 21.0; **IR** (KBr) ν (cm^{-1}): 3332, 2924, 2853, 2359, 1664, 1604, 1517, 1464, 1411, 1368, 1305, 1251, 1123, 1080, 957, 824, 534; **HRMS** (ESI) found: *m/z* 374.1353 $[M+Na]^+$; calcd. for $C_{19}H_{20}F_3NO_2$+Na 374.1344.




-59.01
-59.04
-59.07
H3CO
CF3
N
H
O
3o
5.5
-56.0
-56.5
-57.0
-57.5
-58.0
-58.5
-59.0
-59.5
-60.0
-60.5
-61.0
-61.5
-62.0
-62.5
-63.0
f1 (ppm)


173.57
156.70
137.86
137.84
130.51
129.78
127.89
126.75
125.13
121.98
114.10
55.48
48.35
48.33
42.46
42.20
41.93
41.66
22.80
22.79
20.99
H3CO
CF3
N
H
O
3o
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)

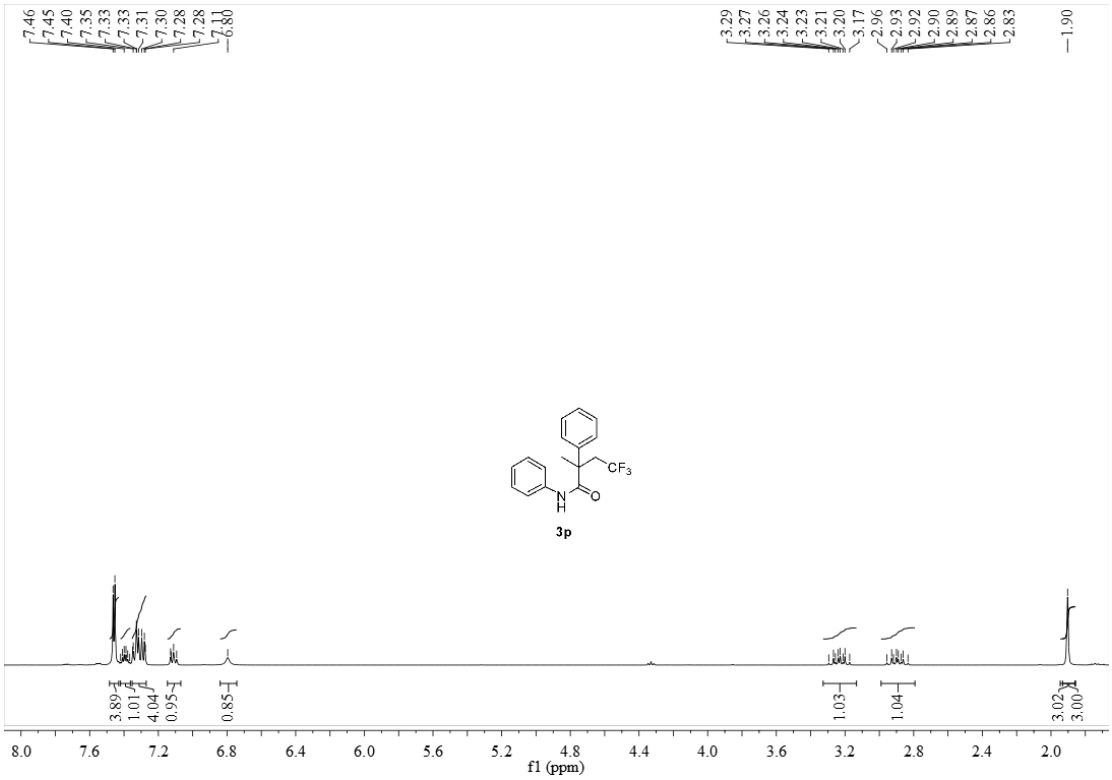


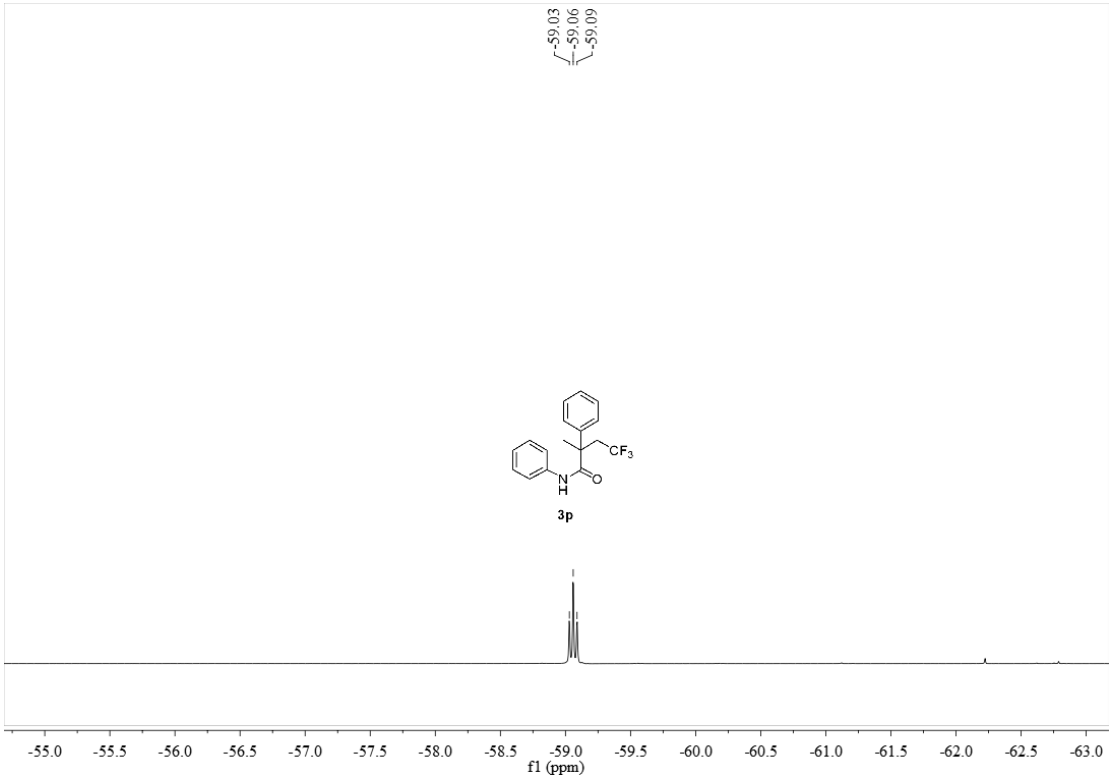


3p

4,4,4-trifluoro-2-methyl-N,2-diphenylbutanamide (3p)

M: 33.2 mg, **Yield:** 54%; **^{1}H-NMR** (400 MHz, $CDCl_3$) [ppm] δ 7.46 (d, J = 4.2 Hz, 4H), 7.39 (dq, J = 8.7, 4.4 Hz, 1H), 7.31 (qd, J = 8.6, 1.8 Hz, 4H), 7.11 (dd, J = 9.8, 4.3 Hz, 1H), 6.80 (s, 1H), 3.23 (dq, J = 15.7, 11.0 Hz, 1H), 2.89 (dq, J = 15.6, 11.2 Hz, 1H), 1.90 (s, 3H), 1.90 (s, 3H); **^{19}F-NMR** (376 MHz, $CDCl_3$) [ppm] δ –59.06 (t, J = 11.1 Hz, 3F); **^{13}C-NMR** (100 MHz, $CDCl_3$) [ppm] δ 173.3, 140.8, 137.4, 129.2, 129.0, 128.1, 126.8, 126.4 (q, J = 277.0 Hz), 124.6, 120.0, 48.9 (d, J = 1.0 Hz), 42.1 (q, J = 27.0 Hz), 22.6; **IR** (KBr) ν (cm^{-1}): 3319, 3058, 2955, 2359, 1666, 1598, 1526, 1440, 1368, 1313, 1259, 1125, 1040, 948, 749, 695, 642, 544; **HRMS** (ESI) found: *m/z* 330.1086 $[M+Na]^+$; calcd. for $C_{17}H_{16}F_3NO$+Na 330.1082.


7.46
7.45
7.40
7.35
7.33
7.31
7.30
7.28
7.28
7.11
6.80
3.29
3.27
3.26
3.24
3.23
3.21
3.20
3.17
2.96
2.93
2.92
2.90
2.89
2.87
2.86
2.83
1.90
CF3
N
H
O
3p
3.89
1.01
4.04
0.95
0.85
1.03
1.04
3.02
3.00
8.0
7.6
7.2
6.8
6.4
6.0
5.6
5.2
4.8
4.4
4.0
3.6
3.2
2.8
2.4
2.0
f1 (ppm)


-59.03
-59.06
-59.09
CF3
N
H
O
3p
-55.0
-55.5
-56.0
-56.5
-57.0
-57.5
-58.0
-58.5
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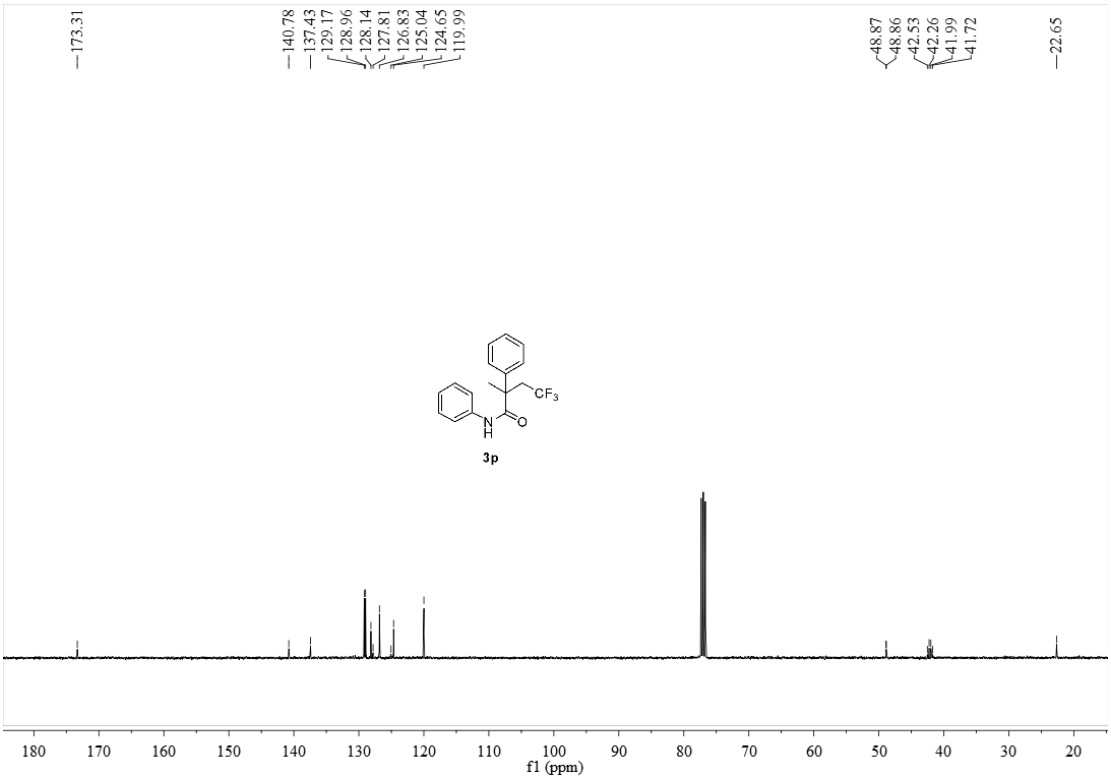
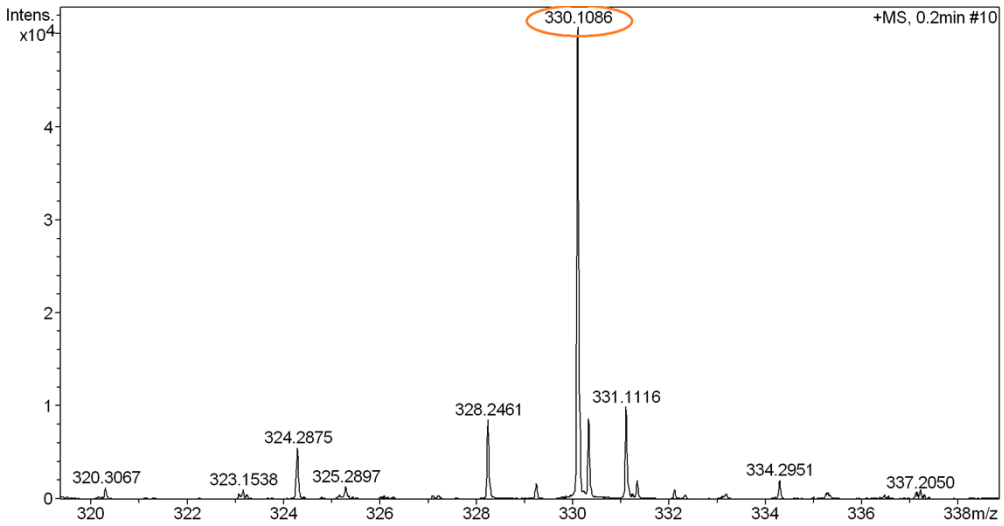
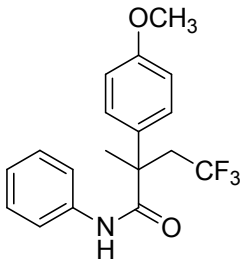
-62.0
-62.5
-63.0
f1 (ppm)





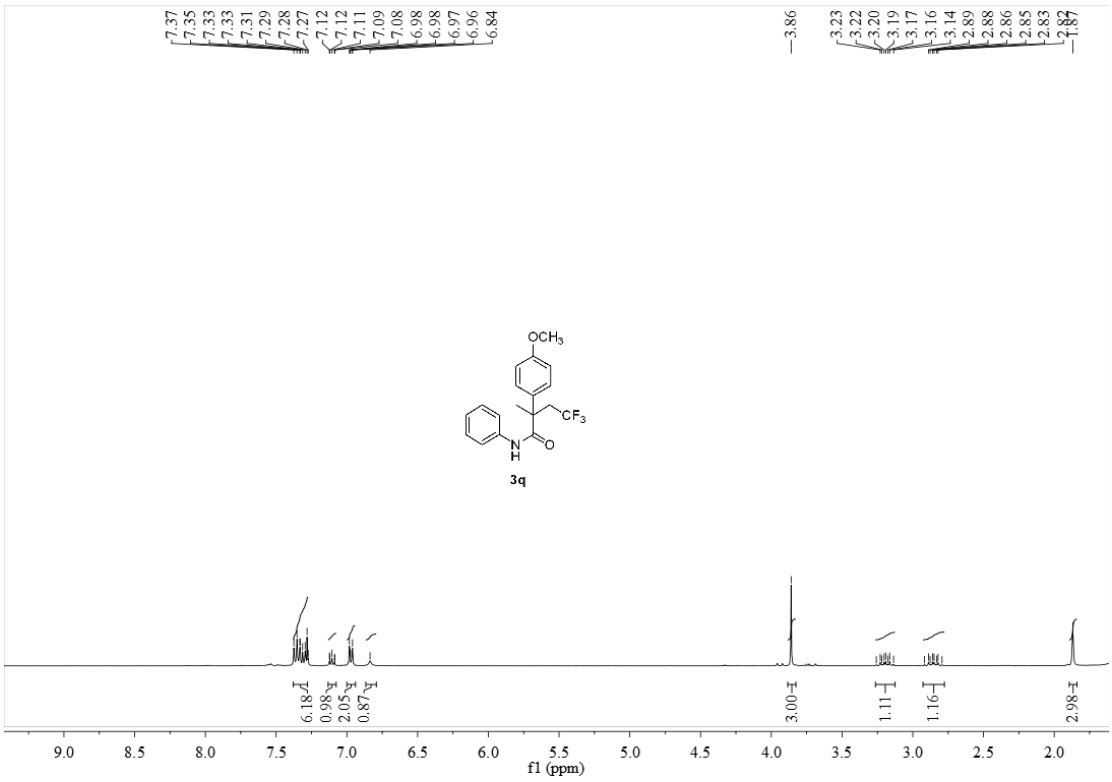


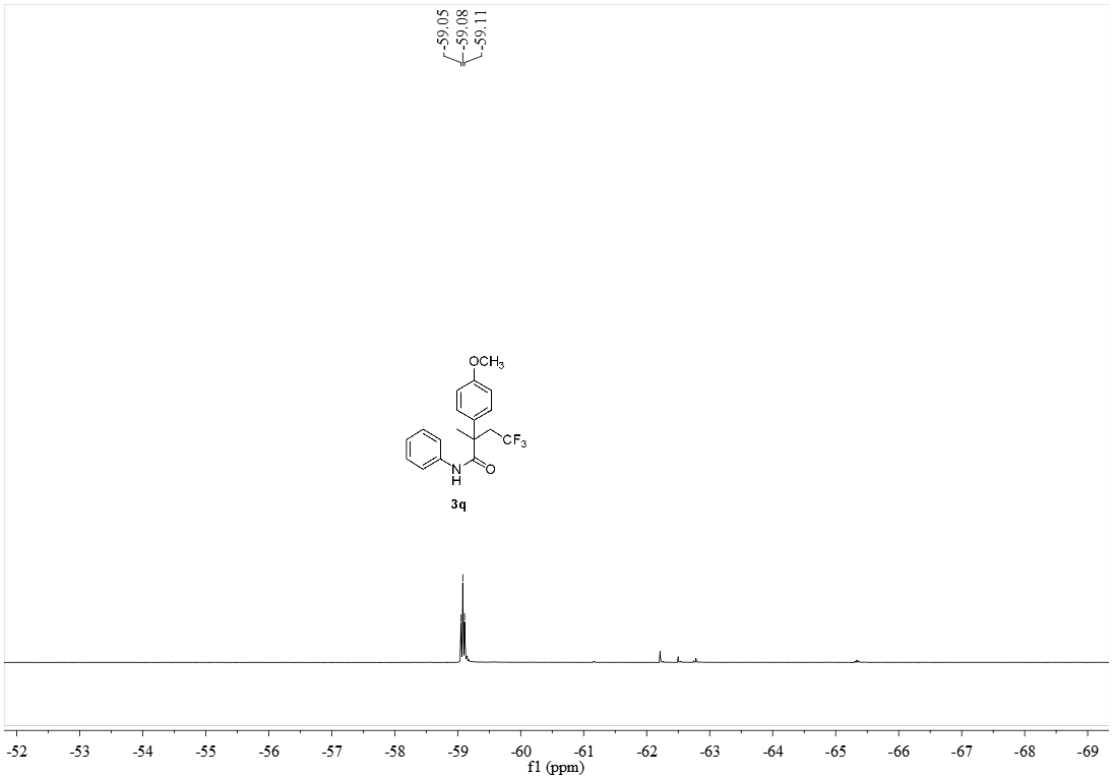
3q

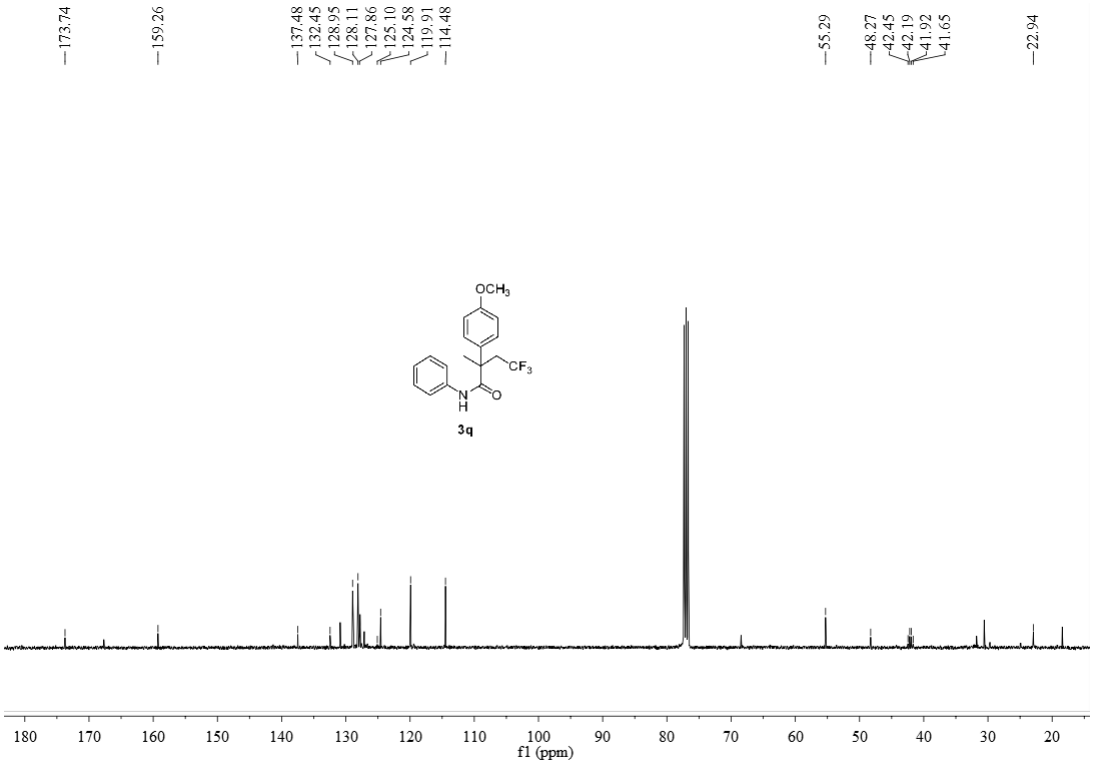
4,4,4-trifluoro-2-(4-methoxyphenyl)-2-methyl-N-phenylbutanamide (3q)

M: 27.6 mg, **Yield:** 41%; **^{1}H-NMR** (400 MHz, $CDCl_3$) [ppm] δ 7.38–7.28 (m, 6H), 7.13–7.08 (m,

1H), 7.00–6.94 (m, 2H), 6.84 (s, 1H), 3.86 (s, 3H), 3.20 (dq, J = 15.6, 11.0 Hz, 1H), 2.85 (dq, J = 15.6, 11.3 Hz, 1H), 1.87 (s, 3H); **^{19}F-NMR** (376 MHz, $CDCl_3$) [ppm] δ –59.08 (t, J = 11.1 Hz, 3F); **^{13}C-NMR** (100 MHz, $CDCl_3$) [ppm] δ 173.7, 159.3, 137.5, 132.4, 129.0, 128.1, 126.5 (q, J = 276.0 Hz), 124.6, 119.9, 114.5, 55.3, 48.3, 42.0 (q, J = 26.5 Hz), 22.9; **IR** (KBr) ν (cm^{-1}): 3316, 2929, 2855, 2360, 1723, 1678, 1604, 1516, 1443, 1366, 1292, 1258, 1125, 1077, 1036, 951, 833, 752, 695, 557; **HRMS** (ESI) found: *m/z* 336.1220 $[M-H]^-$; calcd. for $C_{17}H_{16}F_3NO$-H 336.1212.




-59.05
-59.08
-59.11
OCH3
CF3
N
H
O
3q
-52 -53 -54 -55 -56 -57 -58 -59 -60 -61 -62 -63 -64 -65 -66 -67 -68 -69
f1 (ppm)


173.74
159.26
137.48
132.45
128.95
128.11
127.86
125.10
124.58
119.91
114.48
55.29
48.27
42.45
42.19
41.92
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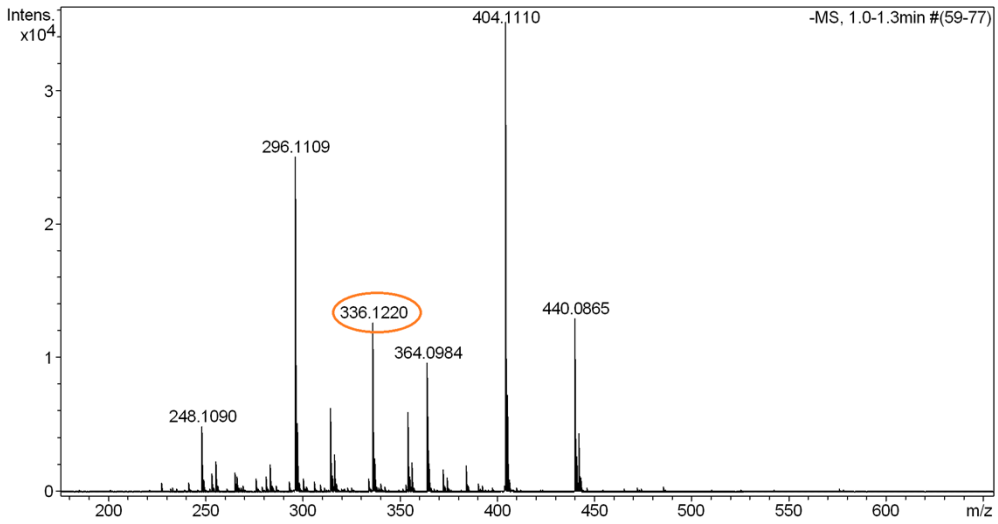
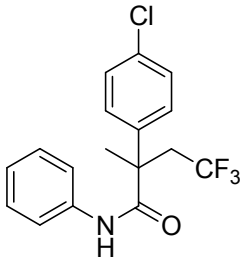
OCH3
CF3
N
H
O
3q
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
f1 (ppm)

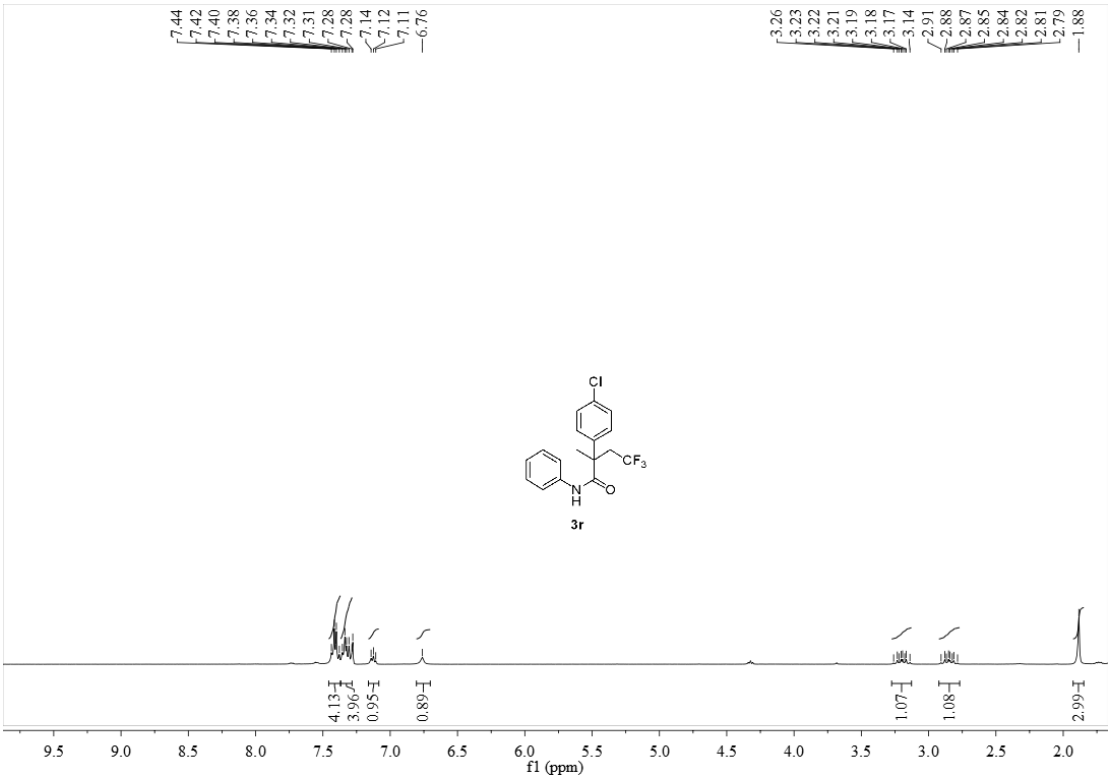


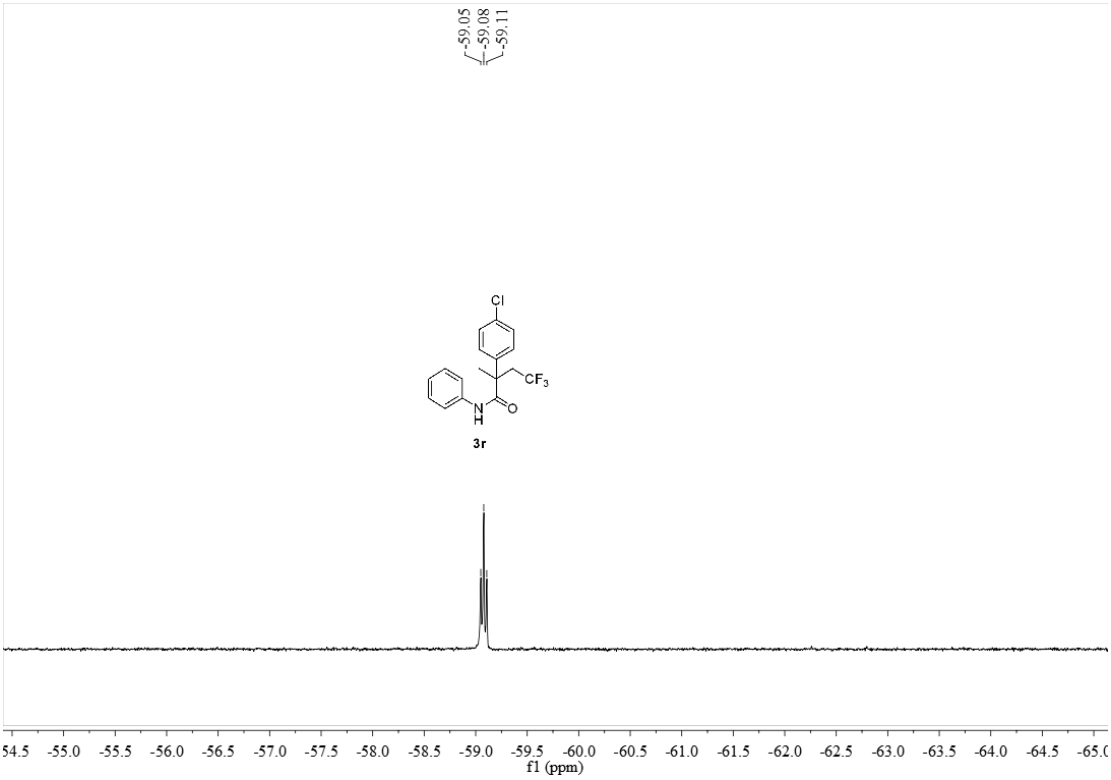


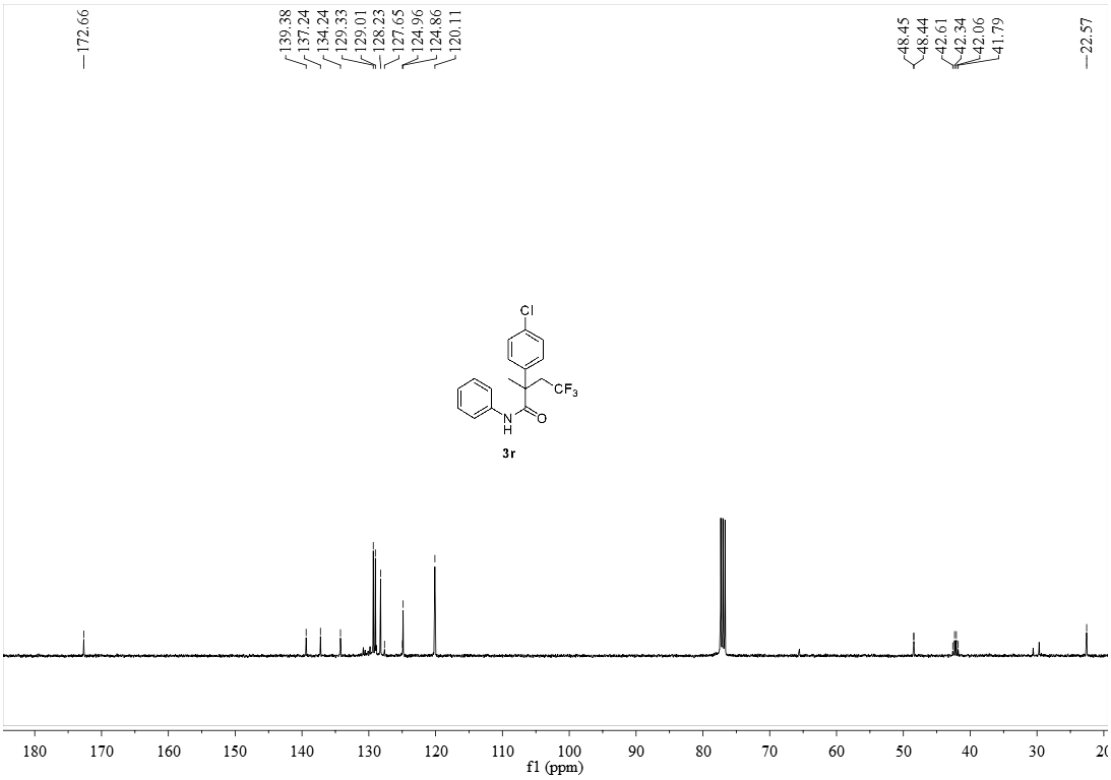
3r

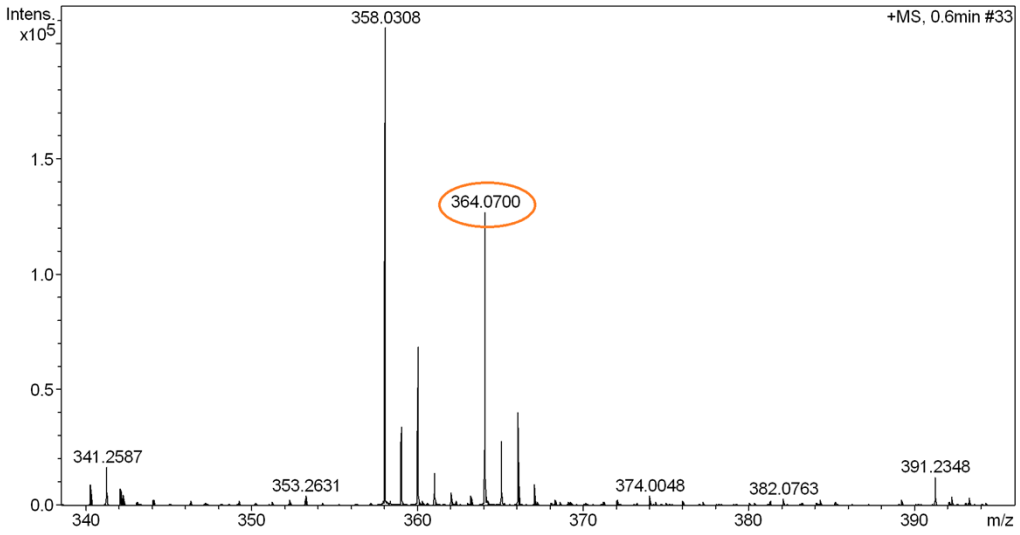
2-(4-chlorophenyl)-4,4,4-trifluoro-2-methyl-N-phenylbutanamide (3r)

M: 20.5 mg, **Yield:** 30%; **^{1}H-NMR** (400 MHz, $CDCl_3$) [ppm] δ 7.41 (q, J = 8.8 Hz, 4H), 7.32 (dt, J = 15.9, 8.1 Hz, 4H), 7.12 (t, J = 7.0 Hz, 1H), 6.76 (s, 1H), 3.20 (dq, J = 15.5, 10.9 Hz, 1H), 2.85 (dq, J = 15.6, 11.1 Hz, 1H), 1.88 (s, 3H); **^{19}F-NMR** (376 MHz, $CDCl_3$) [ppm] δ –59.08 (t, J = 11.0 Hz, 3F); **^{13}C-NMR** (100 MHz, $CDCl_3$) [ppm] δ 172.7, 139.4, 137.2, 134.2, 129.3, 129.0, 128.2, 126.3 (q, J = 269.0 Hz), 124.9, 120.1, 48.4 (d, J = 1.0 Hz), 42.2 (q, J = 27.0 Hz), 22.6; **IR** (KBr) ν (cm^{-1}): 3319, 2919, 2850, 2360, 1673, 1596, 1525, 1440, 1364, 1309, 1259, 1122, 1038, 947, 830, 752, 692, 556; **HRMS** (ESI) found: *m/z* 364.0700 $[M+Na]^+$; calcd. for $C_{17}H_{15}ClF_3NO$+Na 364.0692.


7.44
7.42
7.40
7.38
7.36
7.34
7.32
7.31
7.28
7.28
7.14
7.12
7.11
6.76
3.26
3.23
3.22
3.21
3.19
3.18
3.17
3.14
2.91
2.88
2.87
2.85
2.84
2.82
2.81
2.79
1.88
Cl
CF3
N
H
O
3r
4.13
3.96
0.95
0.89
1.07
1.08
2.99
f1 (ppm)


-59.05
-59.08
-59.11
Cl
CF3
N
H
O
3r
f1 (ppm)


172.66
139.38
137.24
134.24
129.33
129.01
128.23
127.65
124.96
124.86
120.11
48.45
48.44
42.61
42.34
42.06
41.79
22.57
Cl
CF3
N
H
O
3r
f1 (ppm)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374.0048
382.0763
391.2348
m/z