Supporting Information

Preparation of Tri- and Difluoromethylated Amines from Aldimines Using (Trifluoromethyl)trimethylsilane

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General:

Aldimines **1** were prepared by condensation reactions of corresponding aldehydes and anilines, except for benzilideneaniline **1a**, which was commercially available. All other chemicals were purchased from commercial sources. THF was freshly distilled over sodium. Column chromatography was carried out using silica gel 63-200 μm from Solvent Technologies. NMR spectra were obtained on a 400 MHz superconducting spectrometer using CDCl_3 as solvent, TMS as an internal standard for ^1H and ^{13}C , and CFCl_3 as an internal standard for ^{19}F . GCMS data were recorded on a Thermo Finnigan TRACE GC/DSQ spectrometer at 70 eV.

Typical Procedure for Nucleophilic Trifluoromethylation of aldimines with TMSCF_3 . Under nitrogen atmosphere, into a 25 ml round-bottomed flask containing benzilideneaniline **1a** (181 mg, 1.0 mmol) and tetrabutylammonium triphenyldifluorosilicate (TBAT) (81 mg, 0.15 mmol) in THF (5 ml) at room temperature, was added dropwise TMSCF_3 (213 mg, 1.5 mmol) via a syringe. The reaction mixture was stirred at room temperature for 1 hour, and then quenched by adding 1N HCl aqueous solution (0.2 ml). The quenched solution was washed with aqueous saturated NaHCO_3 solution followed by water and saturated aqueous NaCl solution. The combined organic phase was dried over MgSO_4 , filtered, and the solvent was removed under reduced pressure. The residue was further purified by silica gel column chromatography (hexane : ethyl acetate = 9 : 1) to give *N*-(2,2,2-trifluoro-1-phenylethyl)aniline **3a** (188 mg, 75% yield) as a pale yellow oil. ^1H NMR δ 4.32 (br. d, J = 7.3Hz, 1H), 4.91 (qd, J = 7.6, 7.3Hz, 1H), 6.62 (d, J = 7.9Hz, 2H), 6.76 (t, J = 7.5Hz, 1H), 7.14 (dd, J = 8.4, 7.5Hz, 2H), 7.32-7.45 (m, 5H); ^{19}F NMR δ -74.5 (d, J = 7.6Hz, 3F); ^{13}C NMR δ 60.5 (q, J = 29.8Hz), 113.9, 119.2, 125.1 (q, J = 282Hz), 127.9, 128.7, 128.9, 129.1, 129.4, 129.7; GCMS m/z 250 ($\text{M}^+\text{-H}$), 181 ($\text{M}^+\text{-CF}_3\text{H}$).

***N*-(2,2,2-trifluoro-1-phenylethyl)-4-methoxy-*N*-(trimethylsilyl)aniline 2b:** pale yellow oil, yield: 21%. ^1H NMR δ 0.11 (s, 9H), 3.75 (s, 3H), 4.78 (q, J = 9.2Hz, 1H), 6.54 (m, 2H), 6.62 (m, 2H), 7.03 (m, 2H), 7.24 (m, 3H); ^{19}F NMR δ -68.6 (d, J = 9.2Hz, 3F); ^{13}C NMR δ 0.5, 55.2, 63.7 (q, J = 29.0Hz), 113.0, 126.3 (q, J = 286Hz), 128.0, 128.0, 129.6, 134.2, 134.8, 135.1, 157.4; GCMS m/z 353 (M^+), 351, 283 ($\text{M}^+\text{-CF}_3\text{H}$).

***N*-(2,2,2-trifluoro-1-phenylethyl)-4-methoxyaniline 3b:** pale yellow oil, yield: 72%. ^1H NMR δ 3.71 (s, 3H), 4.10 (br. d, J = 6.6Hz, 1H), 4.82 (qd, J = 7.6, 6.6Hz, 1H), 6.61 (m, 2H), 6.75 (m, 2H), 7.39 (m, 3H), 7.45 (m, 2H); ^{19}F NMR δ -74.5 (d, J = 7.6Hz, 3F); ^{13}C NMR δ 55.6, 61.7 (q, J = 29.8Hz), 114.8,

115.7, 125.1 (q, J= 282Hz), 127.9, 128.9, 129.1, 134.3, 139.5, 153.3; GCMS m/z 281 (M^+), 280 (M^+-H), 212 (M^+-CF_3), 211 (M^+-CF_3H), 121

***N*-(2,2,2-trifluoro-1-*p*-torylethyl)aniline 3c:** pale yellow oil, yield: 46%. 1H NMR δ 2.33 (s, 3H), 4.28 (br. d, J= 7.3Hz, 1H), 4.87 (dq, J= 7.3, 6.1Hz, 1H), 6.63 (m, 2H), 6.76 (m, 1H), 7.12-7.19 (m, 4H), 7.33 (m, 2H); ^{19}F NMR δ -74.6 (d, J= 6.1Hz, 3F); ^{13}C NMR δ 21.1, 60.3 (q, J= 29.8Hz), 113.9, 119.1, 125.1 (q, J= 282Hz), 127.7, 129.3, 129.6, 131.0, 139.0, 145.6; GCMS m/z 264 (M^+-H), 195 (M^+-CF_3H). HRMS (EI) m/z 265.1070, calcd for $C_{15}H_{14}F_3N$ 265.1078.

***N*-(2,2,2-trifluoro-1-(4-methoxyphenyl)ethyl)aniline 3d:** pale yellow oil, yield: 38%. 1H NMR δ 3.76 (s, 3H), 4.28 (br. d, J= 6.4Hz, 1H), 4.85 (dq, J= 6.4, 6.1Hz, 1H), 6.62 (m, 2H), 6.76 (m, 1H), 6.89 (m, 2H), 7.14 (m, 2H), 7.35 (m, 2H); ^{19}F NMR δ -74.8 (d, J= 6.1Hz, 3F); ^{13}C NMR δ 55.2, 60.4 (q, J= 29.8Hz), 113.9, 114.3, 119.1, 125.1 (q, J= 282Hz), 125.9, 129.0, 129.3, 145.6, 160.1; GCMS m/z 280 (M^+-H), 211 (M^+-CF_3H), 188 (M^+-PhNH_2)

***N*-(1-(4-chlorophenyl)-2,2,2-trifluoroethyl)aniline 3e:** pale yellow oil, yield: 54%. 1H NMR δ 4.30 (br. d, J= 6.6Hz, 1H), 4.88 (qd, J= 7.6, 6.6Hz, 1H), 6.59 (m, 2H), 6.78 (m, 1H), 7.15 (m, 2H), 7.33-7.40 (m, 4H); ^{19}F NMR δ -74.6 (d, J= 7.6Hz, 3F); ^{13}C NMR δ 60.0 (q, J= 29.8Hz), 113.9, 119.5, 124.5 (q, J= 282Hz), 129.1, 129.3, 129.4, 132.5, 135.1, 145.1; GCMS m/z 286/284 (M^+), 217/215 (M^+-CF_3), 216/214 (M^+-CF_3H).

***N*-(2,2,2-trifluoro-1-(4-fluorophenyl)ethyl)aniline 3f:** pale yellow oil, yield: 67%. 1H NMR δ 4.29 (br. d, J= 7.0Hz, 1H), 4.88 (dq, J= 7.6, 7.0Hz, 1H), 6.59 (m, 2H), 6.77 (m, 1H), 7.04 (m, 2H), 7.14 (m, 2H), 7.40 (m, 2H); ^{19}F NMR δ -74.7 (d, J= 7.6Hz, 3F), -112.9 (m, 1F); ^{13}C NMR δ 60.0 (q, J= 29.8Hz), 113.9, 115.9 (d, J= 22.1Hz), 119.4, 124.9 (q, J= 280Hz), 129.4, 129.7 (d, J=8.4Hz), 129.8 (br.), 145.2, 163.1 (d, J= 248Hz); GCMS m/z 268 (M^+-H), 119 (M^+-CF_3H). HRMS (EI) m/z 269.0820, calcd for $C_{14}H_{11}F_4N$ 269.0827.

***N*-(1-(4-bromophenyl)-2,2,2-trifluoroethyl)aniline 3g:** pale yellow oil, yield: 50%. 1H NMR δ 4.29 (br. d, J= 7.1Hz, 1H), 4.86 (qd, J= 7.6, 7.1Hz, 1H), 6.58 (m, 2H), 6.77 (m, 1H), 7.14 (m, 2H), 7.31 (m, 2H), 7.49 (m, 2H); ^{19}F NMR δ -74.5 (d, J= 7.6Hz, 3F); ^{13}C NMR δ 60.0 (q, J= 30.5Hz), 113.9, 119.5, 123.3, 124.7 (q, J= 282Hz), 129.4, 129.5, 132.1, 133.0, 145.0; GCMS m/z 330/328 (M^+-H), 261/259 (M^+-CF_3H). HRMS (EI) m/z 329.0016, calcd for $C_{14}H_{11}BrF_3N$ 329.0026.

***N*-(1-(2,4-dichlorophenyl)-2,2,2-trifluoroethyl)aniline 3h**: pale yellow oil, yield: 77%. ^1H NMR δ 4.44 (br. d, J = 7.7Hz, 1H), 5.61 (dq, J = 7.7, 6.1Hz, 1H), 6.63 (m, 2H), 6.83 (m, 1H), 7.20 (m, 2H), 7.27 (m, 1H), 7.46-7.56 (m, 2H); ^{19}F NMR δ -74.5 (d, J = 6.1Hz, 3F); ^{13}C NMR δ 55.8 (q, J = 31.3Hz), 113.7, 119.6, 124.6 (q, J = 282Hz), 127.9, 129.4, 129.5, 129.7, 130.6, 135.4, 135.6, 144.6; GCMS m/z 323/321/319 (M^+), 322/320/318 (M^+-H), 321/319/317 (M^+-H_2), 253/251/249 ($\text{M}^+-\text{CF}_3\text{H}$). HRMS (EI) m/z 319.0148, calcd for $\text{C}_{14}\text{H}_{10}\text{Cl}_2\text{F}_3\text{N}$ 319.0142.

***N*-(2,2,2-trifluoro-1-(2-fluorophenyl)ethyl)aniline 3i**: pale yellow oil, yield: 74%. ^1H NMR δ 4.39 (br. d, J = 8.6Hz, 1H), 5.36 (m, 1H), 6.65 (m, 2H), 6.78 (m, 1H), 7.08-7.19 (m, 4H), 7.33 (m, 1H), 7.45 (m, 1H); ^{19}F NMR δ -74.9 (dd, J = 6.1, 6.1Hz, 3F), -118.1 (m, 1F); ^{13}C NMR δ 53.7 (qd, J = 31.3, 3.8Hz), 113.7, 115.9 (d, J = 22.1Hz), 119.4, 121.3 (d, J =13.7Hz), 124.7 (d, J =3.8Hz), 124.8 (br. q, J = 280Hz), 128.6 (br.), 129.4, 130.8 (d, J = 9.2Hz), 124.6, 161.0 (d, J = 248Hz); GCMS m/z 268 (M^+-H), 199 ($\text{M}^+-\text{CF}_3\text{H}$). HRMS (EI) m/z 269.0818, calcd for $\text{C}_{14}\text{H}_{11}\text{F}_4\text{N}$ 269.0827.

***N*-(1-(2-bromophenyl)-2,2,2-trifluoroethyl)aniline 3j**: pale yellow oil, yield: 83%. ^1H NMR δ 4.43 (br. d, J = 7.9Hz, 1H), 5.63 (dq, J = 7.9, 7.6Hz, 1H), 6.62 (m, 2H), 6.76 (m, 1H), 7.12-7.21 (m, 3H), 7.31 (m, 1H), 7.50 (m, 1H), 7.61 (m, 1H); ^{19}F NMR δ -74.2 (d, J = 7.6Hz, 3F); ^{13}C NMR δ 58.6 (q, J = 29.8Hz), 113.7, 119.3, 124.9 (q, J = 282Hz), 125.2, 128.1, 128.6, 129.4, 130.5, 133.2, 133.6, 144.9; GCMS m/z 330/328 (M^+-H), 329/327 (M^+-H_2), 261/259 ($\text{M}^+-\text{CF}_3\text{H}$).

***N*-(1-cyclohexyl-2,2,2-trifluoroethyl)aniline 3k**: pale yellow oil, yield: 47%. ^1H NMR δ 1.09-1.20 (m, 2H), 1.22-1.34 (m, 3H), 1.62-1.93 (m, 6H), 3.71 (br., 1H), 3.74 (m, 1H), 6.66 (m, 2H), 6.75 (m, 1H), 7.18 (m, 2H); ^{19}F NMR δ -72.3 (d, J = 7.6Hz); ^{13}C NMR δ 25.9, 26.1, 27.3, 30.3, 38.5, 59.9 (q, J = 27.5Hz), 113.3, 118.5, 126.3 (q, J = 285Hz), 129.4, 147.1; GCMS m/z 256 (M^+-H), 187 ($\text{M}^+-\text{CF}_3\text{H}$), 173 ($\text{M}^+-\text{C}_6\text{H}_{12}$). HRMS (EI) m/z 329.0026, calcd for $\text{C}_{14}\text{H}_{11}\text{BrF}_3\text{N}$ 329.0026.

***N*-(1-(4-chlorophenyl)-2,2-difluoroethylidene)aniline 4e**: (syn/anti=1:1 mixture) pale yellow oil, yield: 36%. ^1H NMR δ 6.35 (t, J = 55.3Hz, 1H), 6.37 (t, J = 52.9Hz), 6.72 (m, 2H), 6.89 (m, 2H), 7.06 (m, 2H),

7.17-7.26 (m, 6H), 7.39 (m, 2H), 7.45 (m, 2H), 8.02 (m, 2H); ^{19}F NMR δ -116.4 (d, J = 53.4Hz), -117.4 (d, J = 55.0Hz); GCMS m/z 266/264(M^+), 215/213 (M^+ - CF_2H), 212.

Compound 6e: pale yellow oil, yield: 13%. ^1H NMR δ 4.73 (br. d, J = 9.3Hz, 1H), 5.38 (ddd, J = 16.8, 9.3, 7.6Hz, 1H), 6.56 (m, 2H), 6.64 (m, 2H), 6.75 (m, 1H), 6.89 (m, 2H), 7.00 (m, 1H), 7.12-7.20 (m, 6H), 7.34 (m, 2H), 7.43 (m, 2H); ^{19}F NMR δ -100.5 (dd, J = 262, 7.6Hz, 1F), -110.3 (dd, J = 262, 16.8Hz); GCMS m/z 482/480/478(M^+), 481/479/477 (M^+ -H), 462/460/458 (M^+ -HF), 461/459/457 (M^+ - H_2F), 216/214.

Typical Procedure for Direct Preparation of Difluorinated Amines from Imines with TMSCF_3 .

Under nitrogen atmosphere, into a 50 ml round-bottomed flask containing benzilideneaniline **1a** (181 mg, 1.0 mmol) and tetramethylammonium fluoride (TMAF) (47 mg, 0.5 mmol) in THF (5 ml) at room temperature, was added TMSCF_3 (213 mg, 1.5 mmol). dropwise via a syringe The reaction mixture was stirred at room temperature for 1 hour, and then the solvent was removed under reduced pressure. The residue was dissolved in EtOH (10 ml), and NaBH_4 (757 mg, 20 mmol) was added gradually. The reaction mixture was stirred at room temperature for another 1 hour, and the solvent was removed again. Water (20 ml) was added, and extracted with Et_2O . The combined organic phase was dried over MgSO_4 , filtered, and the solvent was removed under reduced pressure. The residue was further purified by silica gel column chromatography (hexane : ethyl acetate = 60 : 1) to give *N*-(2,2-difluoro-1-phenylethyl)aniline **8a** (114 mg, 62% yield) as a pale yellow oil. ^1H NMR δ 4.36 (br. d, J = 6.4Hz, 1H), 4.68 (dddd, J = 13.7, 13.7, 6.4, 3.1Hz, 1H), 5.95 (ddd, J = 56.5, 56.5, 3.1Hz, 1H), 6.58 (m, 2H), 6.72 (m, 1H), 7.11 (m, 2H), 7.30-7.40 (m, 5H); ^{19}F NMR δ -125.4 (ddd, J = 279, 56.5, 13.7Hz, 1F), -127.6 (ddd, J = 279, 56.5, 13.7Hz, 1F); ^{13}C NMR δ 60.1 (dd, J = 22.1, 22.1Hz), 113.4, 115.7 (dd, J = 247, 247Hz), 118.6, 127.7, 128.5, 128.8, 129.2, 135.3 (br.), 146.0; GCMS m/z 232 (M^+ -H), 181 (M^+ - CF_2H_2). HRMS (EI) m/z 233.1016, calcd for $\text{C}_{14}\text{H}_{13}\text{F}_2\text{N}$ 233.1016.

***N*-(2,2-difluoro-1-phenylethyl)-4-methoxyaniline 8b:** pale yellow oil, yield: 37%. ^1H NMR δ 3.70 (s, 3H), 4.15 (br. s, 1H), 4.63 (br. m, 1H), 5.97 (ddd, J = 55.9, 55.9, 3.1Hz, 1H), 6.57 (m, 2H), 6.73 (m, 2H), 7.32-7.42 (m, 5H); ^{19}F NMR δ -125.7 (ddd, J = 279, 56.5, 12H.2z, 1F), -127.1 (ddd, J = 279, 56.5, 12.2Hz, 1F); GCMS m/z 262 (M^+ -H), 211 (M^+ - CF_2H_2), 210. HRMS (EI) m/z 263.1129, calcd for $\text{C}_{15}\text{H}_{15}\text{F}_2\text{NO}$ 263.1122.

***N*-(1-(4-chlorophenyl)-2,2-difluoroethyl)aniline 8e:** pale yellow oil, yield: 78%. ^1H NMR δ 4.37 (br. d, J = 6.2Hz, 1H), 4.67 (dddd, J = 13.7, 12.2, 6.6, 2.9Hz, 1H), 5.97 (ddd, J = 54.9, 54.9, 2.9Hz, 1H), 6.57 (m, 2H), 6.65 (m, 1H), 7.13 (m, 2H), 7.36 (m, 4H); ^{19}F NMR δ -126.3 (ddd, J = 281, 54.9, 13.7Hz, 1F), -127.1 (ddd, J = 281, 54.9, 12.2Hz, 1F); ^{13}C NMR δ 59.5 (dd, J = 21.4, 21.4Hz), 113.9, 115.3 (dd, J = 247, 247Hz), 118.9, 129.0, 129.1, 129.3, 133.7, 134.4, 145.5; GCMS m/z 268/266 (M^+), 267/265 ($\text{M}^+ - \text{H}$), 217/215 ($\text{M}^+ - \text{CF}_2\text{H}$), 216/214 ($\text{M}^+ - \text{CF}_2\text{H}_2$), 179. HRMS (EI) m/z 267.0632, calcd for $\text{C}_{14}\text{H}_{12}\text{ClF}_2\text{N}$ 267.0626.

***N*-(2,2-difluoro-1-(4-fluorophenyl)ethyl)aniline 8f:** pale yellow oil, yield: 79%. ^1H NMR δ 4.35 (br. d, J = 6.4Hz, 1H), 4.68 (dddd, J = 13.7, 12.2, 6.4, 2.9Hz, 1H), 5.95 (ddd, J = 54.9, 54.9, 2.9Hz, 1H), 6.57 (m, 2H), 6.74 (m, 1H), 7.05 (m, 2H), 7.13 (m, 2H), 7.38 (m, 2H); ^{19}F NMR δ -114.0 (m, 1F), -126.3 (ddd, J = 279, 54.9, 13.7Hz, 1F), -127.2 (ddd, J = 279, 54.9, 12.2Hz, 1F); ^{13}C NMR δ 59.5 (dd, J = 22.1, 22.1Hz), 113.9, 115.5 (dd, J = 247, 247Hz), 115.8 (d, J = 22.1Hz), 118.9, 129.3, 129.4 (d, J = 8.4Hz), 130.9 (br.), 145.7, 162.8 (d, J = 247Hz); GCMS m/z 250 ($\text{M}^+ - \text{H}$), 199 ($\text{M}^+ - \text{CF}_2\text{H}_2$). HRMS (EI) m/z 251.0923, calcd for $\text{C}_{14}\text{H}_{12}\text{F}_3\text{N}$ 251.0922.

***N*-(1-(4-bromophenyl)-2,2-difluoroethyl)aniline 8g:** pale yellow oil, yield: 63%. ^1H NMR δ 4.33 (br. d, J = 6.4Hz, 1H), 4.63 (dddd, J = 13.7, 13.7, 6.4, 2.9Hz, 1H), 5.92 (ddd, J = 55.9, 55.9, 2.9Hz, 1H), 6.55 (m, 2H), 6.73 (m, 1H), 7.12 (m, 2H), 7.26 (m, 2H), 7.46 (m, 2H); ^{19}F NMR δ -126.1 (ddd, J = 281, 56.5, 13.7Hz, 1F), -127.0 (ddd, J = 281, 56.5, 13.7Hz, 1F); ^{13}C NMR δ 59.6 (dd, J = 22.1, 22.1Hz), 113.9, 115.2 (dd, J = 247, 247Hz), 118.9, 122.6, 129.3, 129.4, 131.9, 134.2, 145.5; GCMS m/z 312/310 ($\text{M}^+ - \text{H}$), 261/259 ($\text{M}^+ - \text{CF}_2\text{H}_2$). HRMS (EI) m/z 311.0130, calcd for $\text{C}_{14}\text{H}_{12}\text{BrF}_2\text{N}$ 311.0121.

***N*-(1-(2,4-dichlorophenyl)-2,2-difluoroethyl)aniline 8h:** pale yellow oil, yield: 58%. ^1H NMR δ 4.51 (br. d, J = 6.6Hz, 1H), 5.26 (dddd, J = 16.8, 10.7, 6.6, 2.2Hz, 1H), 6.07 (ddd, J = 54.9, 54.9, 2.2Hz, 1H), 6.53 (m, 2H), 6.76 (m, 1H), 7.14 (m, 2H), 7.24 (m, 1H), 7.43 (m, 2H), 7.46 (m, 1H); ^{19}F NMR δ -124.6 (ddd, J = 278, 54.9, 10.7Hz, 1F), -131.3 (ddd, J = 278, 54.9, 16.8Hz, 1F); ^{13}C NMR δ 56.1 (dd, J = 20.6, 20.6Hz), 113.5, 114.3 (dd, J = 249, 246Hz), 119.0, 127.9, 129.4, 129.4, 130.0, 131.4, 134.3, 135.0, 145.1; GCMS m/z 304/302/300 ($\text{M}^+ - \text{H}$), 253/251/249 ($\text{M}^+ - \text{CF}_2\text{H}_2$). HRMS (EI) m/z 301.0231, calcd for $\text{C}_{14}\text{H}_{11}\text{Cl}_2\text{F}_2\text{N}$ 301.0236.

***N*-(2,2-difluoro-1-(2-fluorophenyl)ethyl)aniline 8i:** pale yellow oil, yield: 35%. ^1H NMR δ 4.42 (br. d, J = 7.9Hz, 1H), 5.13 (m, 1H), 6.08 (ddd, J = 56.5, 54.9, 2.7Hz, 1H), 6.62 (m, 2H), 6.75 (m, 1H), 7.08-7.17 (m, 4H), 7.31 (m, 1H), 7.41 (m, 1H); ^{19}F NMR δ -119.1 (m, 1F), -125.2 (dddd, J = 279, 54.9, 12.2, 3.1Hz, 1F), -128.7 (br. ddd, J = 279, 56.5, 13.7Hz, 1F); ^{13}C NMR δ 53.9 (ddd, J = 20.6, 20.6, 1.5Hz), 112.4, 114.9 (br. dd, J = 248, 248Hz), 115.6 (d, J = 21.4Hz), 118.9, 112.4 (br. d, J = 16.8Hz), 124.6 (d, J = 3.8Hz), 129.1 (d, J = 3.8Hz), 129.4, 130.2 (d, J = 8.4Hz), 145.6, 160.9 (d, J = 246Hz); GCMS m/z 250 (M^+ -H), 199 (M^+ -CF₂H₂). HRMS (EI) m/z 251.0926, calcd for C₁₄H₁₂F₃N 251.0921.

***N*-(1-(2-bromophenyl)-2,2-difluoroethyl)aniline 8j:** Obtained only in trace amounts, ^1H NMR δ 4.61 (br. d, J = 7.0Hz, 1H), 5.32 (dddd, J = 18.3, 10.7, 7.0, 2.0Hz, 1H), 6.10 (ddd, J = 54.9, 54.9, 2.0Hz, 1H), Aromatics; ^{19}F NMR δ -123.7 (ddd, J = 276, 54.9, 10.7Hz, 1F), -132.4 (ddd, J = 276, 54.9, 18.3Hz, 1F); GCMS m/z 312/310 (M^+ -H), 311/309 (M^+ -H₂), 261/259 (M^+ -CF₂H₂).

VL

INDEX	FREQUENCY	PPM	HEIGHT
1	-25806.716	-68.616	108.5
2	-25817.071	-68.641	103.3

2b

\\xcalibur\data\mog\050713-f2

07/14/2005 12:15:31 PM

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+: + c Full ms [50.00-650.00]

3a

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1	-28013.611	-74.478	151.7
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3a

08/01/2005 01:25:43 PM

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T: + c Full ms [50.00-650.00]

3b

3b

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3b

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T: + c Full ms [50.00-650.00]

3c

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2	-28074.646	-74.641	145.2

3c

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08/26/2005 03:39:35 PM

050826-2-f2 #4749 RT: 19.66 AV: 1 NL: 8.06E7

Full ms [50.00-650.00]

3d.

3d

INDEX	FREQUENCY	PPM	HEIGHT
1	-28128.052	-74.783	127.1
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3d

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08/23/2005 05:43:59 PM

050823-2-f2 #5329 RT: 21.58 AV: 1 NL: 1.32E8
T: + c Full ms [50.00-650.00]

3e

INDEX	FREQUENCY	PPM	HEIGHT
1	-28068.542	-74.624	154.2
2	-28076.172	-74.845	154.5

D:\Xcalibur\data\mog\050810-1

08/10/2005 01:54:29 PM

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[+ c Full ms [50.00-650.00]

3f

INDEX	FREQUENCY	PPM	HEIGHT
1	-28114.319	-74.746	116.4
2	-28121.948	-74.766	109.1
3	-42469.632	-112.866	22.4

3f

3g

3g

INDEX	FREQUENCY	PPM	HEIGHT
1	-28047.100	-74.568	129.6
2	-28054.610	-74.588	150.9

050822
f11

Br-151 x N-161
H CF₃

3g

050822
f m
Br [C] x m-127
" " CF₃

3g

3g

08/22/2005 07:36:17 PM

C:\xcalibur\data\tomog\050822-2-f1

050822-2-f1 #5326 RT: 21.56 AV: 1 NL: 3.69E7

T: + c Full ms [50.00-650.00]

INDEX	FREQUENCY	PPM	HEIGHT
1	-27896.826	-74.434	156.4
2	-28002.930	-74.450	130.0

3h

0 -20 -40 -60 -80 -100 -120 -140 -160 ppm

3h

09/09/2005 05:21:20 PM

C:\xcalibur\data\mog\050909-f3

050909-f3 #5365 RT: 21.68 AV: 1 NL: 7.99E7

T: + c Full ms [50.00-650.00]

3i

C:\Xcalibur\data\mog\050915-f2

050915-f2 #4279 RT: 18.11 AV: 1 NL: 7.69E7
T: + c Full ms [50.00-650.00]

3i

$$\begin{array}{c}
 \text{H} \\
 | \\
 \text{H} - \text{C} - \text{C} - \text{H} \\
 | \quad | \\
 \text{H} \quad \text{H}
 \end{array}$$

3j

09/19/2005 03:59:33 PM

C:\Xcalibur\data\tm050919-12

050919-12 #5238 RT: 21.27 AV: 1 NL: 1.31E8

T: + c Full ms [50.00-650.00]

3k

3k

3k

D:\Kcalibur\data\nm050916-F2

09/16/2005 04:38:27 PM

050916-F2 #4492 RT: 18.81 AV: 1 NL: 8.93E7

[-]: + c Full ms [50.00-650.00]

4e (syn: anti = 1:1) + ketone

(hydrolyzed product)

4e

¹⁹F NMR

4e (syn: anti = 1:1) + Ketone
(hydrolysed product)

INDEX	FREQUENCY	PPM	HEIGHT
1	-0.080	-0.080	26.4
2	-43753.052	-116.324	76.4
3	-45006.456	-116.466	66.4
4	-44143.677	-117.363	80.9
5	-44198.608	-117.509	106.4

4e (syn: anti = 1:1) + ketone
Chiral product

D:\Xcalibur\data\nog\050815-f1

08/16/2005 12:25:32 PM

050815-f1 #4970 RT: 20.38 AV: 1 NL: 4.57E7
T: + c Full ms [50.00-650.00]

INDEX	FREQUENCY	PPM	HEIGHT
1	-37863.185	-100.186	58.4
2	-37890.735	-100.207	50.2
3	-37945.557	-100.884	61.8
4	-37953.186	-100.904	62.3
5	-41528.320	-110.409	43.9
6	-41545.105	-110.454	45.2
7	-41790.771	-111.107	41.9
8	-41807.556	-111.152	40.9

050816
f(2)

$\delta = 100.5$, $\eta = 6.6$ Hz
 $\delta = 100.5$, $\eta = 6.6$ Hz

$\delta = 110.3$

$\delta = 100.5$, $\eta = 6.6$ Hz
 $\delta = 100.5$, $\eta = 6.6$ Hz

C:\calibur\data\nogio050816-f2

08/16/2005 08:40:48 PM

6e

050816-f2 #10571 RT: 38.86 AV: 1 NL: 5.33E6

T: + c Full ms [50.00-650.00]

8a

09/07/2005 05:55:50 PM

C:\xcalibur\data\mog\050907-f3

050907-f3 #4848 RT: 19.98 AV: 1 NL: 1.71E8

T: + c Full ms [50.00-650.00]

8b

C:\Xcalibur\data\mogl\050908-f4

09/08/2005 06:16:42 PM

050908-f4 #5645 RT: 22.61 AV: 1 NL: 5.89E7

T: + c Full ms [50.00-650.00]

8e

80

D:\Xcalibur\data\mog\050817-5

08/17/2005 07:27:32 PM

050817-5 #5473 RT: 22.05 AV: 1 NL: 4.00E7
Full ms [50.00-650.00]

8f

08/19/2005 05:43:59 PM

C:\Xcalibur\data\tomog\050819-2.f3

050819-2.f3 #4785 RT: 19.77 AV: 1 NL: 9.65E7
T: + c Full ms [50.00-650.00]

8g

8g

8%

2:\calibur\data\mogl\050823-f2

08/23/2005 01:22:38 PM

050823-f2 #5891 RT: 23.43 AV: 1 NL: 1.24E8
F: + c Full ms [50.00-650.00]

D:\Xcalibur\data\mogl\050913-13

09/13/2005 12:15:59 PM

050913-13 #5819 RT: 23.19 AV: 1 NL: 1.28E8
T: + c Full ms [50.00-650.00]

8i

D:\calibur\data\mogi\050916-14

09/16/2005 01:33:42 PM

050916-14 #4713 RT: 19.53 AV: 1 NL: 1.26E8
[+ c Full ms [50.00-650.00]]

$R_F = CF_3 : CF_2H (10 : 2)$

3j : 8j

8j

R_F = CF₃ : CF₂H (10 : 2)

3j : 8j

0 -20 -40 -60 -80 -100 -120 -140 -160 ppm

8j

D:\Xcalibur\data\mogl\050921-11

09/21/2005 04:43:16 PM

050921-11 #5581 RT: 22.40 AV: 1 NL: 7.17E7
 T: + c Full ms [50.00-650.00]