Supporting Information

DIRECT WASTE-FREE SYNTHESIS OF AMIDES FROM NON-ACTIVATED CARBOXYLIC ACIDS AND AMINES: APPLICATION TO THE SYNTHESIS OF TETRAHYDROISOQUINOLINES

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General Information: Amines and carboxylic acids were purchased from Sigma-Aldrich, USA. Silica gel (60-120 mesh) used for column chromatography was purchased from Sisco Research Laboratories Pvt. Ltd., India and all other chemicals were purchased from Spectrochem, India, Merck, Germany and Sigma-Aldrich, USA and were used without further purification. NMR spectra were recorded on a Bruker Avance-300 and 600 spectrometers. Mass spectra were recorded on QTOF-Micro of Waters Micromass.

General experimental procedure for the synthesis of amides: To a suspension of carboxylic acid (1.0 mmol) in dry toluene (20 mL) equipped with Dean-Stark apparatus was added amine (1.0 mmol) and boric acid (0.08 mmol) at room temperature and then refluxed for 8 h (Reactions without boric acid were performed for 14 h). On completion (as monitored by TLC), reaction mixture was precipitated in to amides by adding 20-25 mL *n*-hexane and filtered to get pure amide. The crude product was analyzed by NMR without further purification.

General experimental procedure for the synthesis of tetrahydroisoquinolines: To a stirred suspension of amide (1.0 mmol) in DCM in a round bottom flask equipped with condenser was added phosphorus oxychloride (3 mmol) and the resulting mixture was refluxed for 12 h. The reaction mixture was cooled to 0 °C and neutralized with Na₂CO₃. The resulting mixture was extracted with EtOAc to afford the crude dihydroisoquinolines. To the solution of dihydroisoquinolines in MeOH cooled up to 0 °C, was added NaBH₄ (1.2 equiv.), and stirred for 3 h at rt. The reaction mixture was cooled and extracted with DCM. The tetrahydroisoquinoline was purified by column chromatography over silica-gel (60-120 mesh) with appropriate mixture of *n*-hexane and ethyl acetate.

¹H, ¹³C NMR and Mass spectroscopic data of compounds

N-(Phenylethyl)-2-phenylacetamide (Table 1, entry 3a)

¹H NMR (CDCl₃, 600 MHz): δ = 2.62 (t, 2H, *J* = 6.6, 6.6 Hz), 3.34-3.37 (m, 2H), 3.43 (s, 2H), 5.25 (brs, 2H), 6.92 (d, 2H, *J* = 7.8 Hz), 7.06-7.23 (m, 8H). ¹³C NMR (CDCl₃, 150 MHz): δ = 34.3, 39.4, 42.7, 125.2, 126.1, 127.4, 127.5, 127.8, 128.2, 133.6, 137.5, 169.6. HRMS (ESI, +): calcd for C₁₆H₁₈NO [M+H]⁺ 240.1388, found 240.1397.

N-(Phenylethyl)-2-(2-iodophenyl)acetamide (Table 1, entry 3b)

^1H NMR (CD_3OD , 600 MHz): δ = 2.81 (s, 2H), 3.44 (s, 2H), 3.65 (s, 2H), 6.97 (s, 1H), 7.19-7.32 (m, 7H), 7.84-7.85 (m, 1H). ^{13}C NMR (CD_3OD , 150 MHz): δ = 36.6, 42.3, 101.9, 127.5, 129.6, 129.7, 129.9, 132.1, 140.1, 140.6, 140.8, 172.6. HRMS (ESI, +): calcd for $\text{C}_{16}\text{H}_{17}\text{INO}$ $[\text{M}+\text{H}]^+$ 366.0355, found 366.0364.

***N*-(Phenylethyl)-2-(2-bromophenyl)acetamide (Table 1, entry 3c)¹**

^1H NMR (CDCl_3 , 600 MHz): δ = 2.67-2.69 (m, 2H), 3.39-3.43 (m, 2H), 3.61 (s, 2H), 5.41 (brs, 1H), 6.99-7.00 (m, 2H), 7.07-7.22 (m, 6H), 7.49-7.50 (m, 1H). ^{13}C NMR (CDCl_3 , 150 MHz): δ = 34.3, 39.5, 42.9, 123.8, 125.2, 126.8, 127.4, 127.5, 127.9, 130.5, 131.9, 133.6, 137.4, 168.2. HRMS (ESI, +): calcd for $\text{C}_{16}\text{H}_{17}\text{BrNO}$ $[\text{M}+\text{H}]^+$ 318.0494, found 318.0487.

***N*-(Phenylethyl)-2-(2-bromo-4-methoxyphenyl)acetamide (Table 1, entry 3d)**

^1H NMR (CDCl_3 , 600 MHz): δ = 2.73-2.76 (m, 2H), 3.46-3.49 (m, 2H), 3.60 (s, 2H), 3.80 (s, 3H), 5.34 (brs, 1H), 6.80-6.82 (m, 1H), 7.06-7.08 (m, 2H), 7.10-7.11 (m, 1H),

7.15-7.19 (m, 2H), 7.22-7.26 (m, 2H). ^{13}C NMR (CDCl_3 , 150 MHz): δ = 35.5, 40.7, 43.1, 55.6, 114.1, 118.4, 125.2, 126.4, 126.7, 128.6, 128.7, 132.0, 138.7, 159.4, 169.9. HRMS (ESI, +): calcd for $\text{C}_{17}\text{H}_{19}\text{BrNO}_2$ $[\text{M}+\text{H}]^+$ 348.0599, found 348.0578.

***N*-(Phenylethyl)-2-(3,4-dimethoxyphenyl)acetamide (Table 1, entry 3e)**

^1H NMR (CDCl_3 , 600 MHz): δ = 2.71-2.73 (m, 2H), 3.43-3.47 (m, 4H), 3.82 (s, 3H), 3.88 (s, 3H), 5.41 (brs, 1H), 6.66 (s, 1H), 6.68-6.69 (m, 1H), 6.78-6.79 (m, 1H), 7.01-7.02 (m, 2H), 7.17-7.19 (m, 1H), 7.21-7.24 (m, 2H). ^{13}C NMR (CDCl_3 , 150 MHz): δ = 35.4, 40.6, 43.5, 55.8, 55.9, 111.5, 112.4, 113.9, 121.6, 126.5, 127.2, 128.6, 128.7, 129.6, 138.6, 148.3, 149.2, 171.3. HRMS (ESI, +): calcd for $\text{C}_{18}\text{H}_{22}\text{NO}_3$ $[\text{M}+\text{H}]^+$ 300.1600, found 300.1623.

***N*-(Phenylethyl)-2-(3,4-methylenedioxyphenyl)acetamide (Table 1, entry 3f)**

^1H NMR (CDCl_3 , 600 MHz): δ = 2.77-2.78 (m, 2H), 3.47-3.52 (m, 4H), 5.53 (brs, 1H), 6.00 (s, 2H), 6.64-6.65 (m, 1H), 6.69 (s, 1H), 6.77-6.78 (m, 1H), 7.09-7.10 (m, 2H), 7.23-7.25 (m, 1H), 7.28-7.31 (m, 2H). ^{13}C NMR (CDCl_3 , 150 MHz): δ = 35.4, 40.7, 43.4, 101.2, 108.6, 109.8, 122.7, 126.5, 128.3, 128.6, 128.7, 138.7, 146.9, 148.1, 171.2. HRMS (ESI, +): calcd for $\text{C}_{17}\text{H}_{18}\text{NO}_3$ $[\text{M}+\text{H}]^+$ 284.1287, found 284.1273.

***N*-(2-Fluorophenylethyl)-2-(2-bromophenyl)acetamide (Table 1, entry 3g)**

^1H NMR (CD_3OD , 600 MHz): δ = 2.86 (s, 2H), 3.45 (s, 2H), 3.65 (s, 2H), 7.03-7.08 (m, 2H), 7.16 (s, 1H), 7.23-7.29 (m, 4H), 7.56-7.57 (m, 1H). ^{13}C NMR (CD_3OD , 150 MHz): δ = 30.0, 40.9, 44.2, 116.3, 116.4, 125.5, 126.2, 127.3, 127.5, 129.0, 129.6, 129.7, 130.1, 132.6, 133.1, 134.0, 136.7, 162.1, 163.8, 172.8. MS (ESI, +): calcd for $\text{C}_{16}\text{H}_{16}\text{BrFNO}$ $[\text{M}+\text{H}]^+$ 336.03, found 336.03.

***N*-(2-Fluorophenylethyl)-2-(3,4-dihydroxyphenyl)acetamide (Table 1, entry 3h)**

^1H NMR (CD_3OD , 600 MHz): δ = 2.96 (s, 2H), 3.08 (s, 2H), 3.33 (s, 2H), 6.61-6.67 (m, 2H), 6.76 (s, 1H), 7.09-7.15 (m, 2H), 7.27-7.29 (m, 2H). ^{13}C NMR (CD_3OD , 150 MHz): δ = 28.5, 40.7, 45.4, 116.2, 116.5, 117.5, 121.6, 125.8, 125.9, 130.4, 130.6, 132.4, 144.7, 146.1, 161.9, 163.5, 180.8. HRMS (ESI, +): calcd for $\text{C}_{16}\text{H}_{17}\text{FNO}_3$ $[\text{M}+\text{H}]^+$ 290.1192, found 290.1183.

***N*-(4-Methoxyphenylethyl)-2-(2-bromophenyl)acetamide (Table 1, entry 3j)¹**

^1H NMR (CDCl_3 , 600 MHz): δ = 2.61 (t, 2H, J = 6.6, 7.2 Hz), 3.37 (q, 2H, J = 6.6, 6.6 Hz), 3.59 (s, 2H), 3.70 (s, 3H), 5.31 (brs, 1H), 6.69 (d, 2H, J = 8.4 Hz), 6.89 (d, 2H, J = 8.4 Hz), 7.06-7.09 (m, 1H), 7.18-7.20 (m, 2H), 7.49 (d, 1H, J = 8.4 Hz). ^{13}C NMR (CDCl_3 , 150 MHz): δ = 28.5, 33.3, 42.9, 54.0, 112.8, 123.8, 126.8, 127.8, 128.4, 129.4, 130.5, 131.9, 133.6, 156.9, 168.1. HRMS (ESI, +): calcd for $\text{C}_{17}\text{H}_{19}\text{BrNO}_2$ $[\text{M}+\text{H}]^+$ 348.0599, found 348.0587.

***N*-(4-Methoxyphenylethyl)-2-(2-iodophenyl)acetamide (Table 1, entry 3k)**

^1H NMR (CD_3OD , 600 MHz): δ = 2.74 (s, 2H), 3.39 (s, 2H), 3.65 (s, 2H), 3.76 (s, 3H), 6.81-6.82 (m, 2H), 6.97 (s, 1H), 7.09-7.10 (m, 1H), 7.23 (s, 2H), 7.31 (s, 2H), 7.84-7.85 (m, 1H). ^{13}C NMR (CD_3OD , 150 MHz): δ = 35.6, 42.4, 48.7, 55.7, 101.8, 115.0, 129.7, 129.9, 130.9, 131.9, 132.4, 140.1, 140.7, 159.8, 172.5. HRMS (ESI, +): calcd for $\text{C}_{17}\text{H}_{19}\text{INO}_2$ $[\text{M}+\text{H}]^+$ 396.0461, found 396.0488.

***N*-(4-Methoxyphenylethyl)-2-(4-bromophenyl)acetamide (Table 1, entry 3l)**

^1H NMR (CDCl_3 , 600 MHz): δ = 2.63-2.66 (m, 2H), 3.38-3.44 (m, 4H), 3.77 (s, 3H), 5.45 (brs, 1H), 6.76 (d, 2H, J = 8.4 Hz), 6.91 (d, 2H, J = 8.4 Hz), 7.02 (d, 2H, J = 8.4 Hz), 7.41 (d, 2H, J = 8.4 Hz). ^{13}C NMR (CDCl_3 , 150 MHz): δ = 34.5, 40.8, 43.1, 55.3, 114.0,

121.3, 129.6, 130.5, 131.1, 132.0, 133.9, 158.4, 170.3. HRMS (ESI, +): calcd for C₁₇H₁₉BrNO₂ [M+H]⁺ 348.0599, found 348.0584.

***N*-(4-Methoxyphenylethyl)-2-(4-bromo-4-methoxyphenyl) acetamide (Table 1, entry 3m)**

¹H NMR (CDCl₃, 600 MHz): δ = 2.67-2.69 (m, 2H), 3.41-3.45 (m, 2H), 3.56 (s, 2H), 3.78 (s, 3H), 3.80 (s, 3H), 5.37 (brs, 1H), 6.75-6.77 (m, 2H), 6.80-6.82 (m, 1H), 6.97 (d, 2H, J = 9.0 Hz), 7.10-7.11 (m, 1H), 7.15 (d, 2H, J = 8.4 Hz). ¹³C NMR (CDCl₃, 150 MHz): δ = 34.6, 40.8, 43.2, 55.2, 55.6, 114.0, 118.4, 125.2, 126.7, 129.7, 130.6, 132.1, 158.2, 159.4, 169.9. HRMS (ESI, +): calcd for C₁₈H₂₁BrNO₃ [M+H]⁺ 378.0705, found 378.0788.

***N*-(4-Methoxyphenylethyl)-2-(3,4-dihydroxyphenyl) acetamide (Table 1, entry 3n)**

¹H NMR (CD₃OD, 600 MHz): δ = 2.18 (s, 1H), 2.70 (s, 2H), 2.91 (s, 2H), 3.19 (s, 2H), 3.63 (s, 3H), 6.48 (s, 1H), 6.52-6.53 (m, 1H), 6.63 (s, 1H), 6.75-6.76 (m, 2H), 6.97-7.02 (m, 2H). ¹³C NMR (CD₃OD, 150 MHz): δ = 34.2, 42.4, 48.8, 55.9, 115.5, 116.3, 117.6, 121.7, 126.5, 129.4, 130.1, 130.8, 130.9, 144.8, 146.2, 160.5, 180.9. MS (ESI, +): calcd for C₁₇H₂₀NO₄ [M+H]⁺ 302.13, found 302.13.

***N*-(4-Methoxyphenylethyl)-2-(3,4-dimethoxyphenyl) acetamide (Table 1, entry 3o)**

^1H NMR (CDCl_3 , 600 MHz): δ = 2.64 (t, 2H, J = 6.6, 6.6 Hz), 3.39 (q, 2H, J = 6.6, 6.6 Hz), 3.45 (s, 2H), 3.76 (s, 3H), 3.82 (s, 3H), 3.86 (s, 3H), 5.45 (brs, 1H), 6.65-6.66 (m, 1H), 6.67-6.69 (m, 1H), 6.73-6.76 (m, 2H), 6.78-6.79 (m, 1H), 6.90 (s, 1H), 6.92 (s, 1H). ^{13}C NMR (CDCl_3 , 150 MHz): δ = 34.2, 40.6, 43.2, 54.9, 55.6, 55.7, 111.2, 112.1, 113.7, 121.4, 127.0, 129.4, 130.3, 148.1, 149.0, 157.9, 171.1. HRMS (ESI, +): calcd for $\text{C}_{19}\text{H}_{24}\text{NO}_4$ $[\text{M}+\text{H}]^+$ 330.1705, found 330.1775.

***N*-(4-Methoxyphenylethyl)-2-(3,4-methylenedioxyphenyl)acetamide (Table 1, entry 3p)**

^1H NMR (CDCl_3 , 600 MHz): δ = 2.67 (t, 2H, J = 6.6, 7.2 Hz), 3.42 (q, 4H, J = 5.4, 6.6 Hz), 3.79 (s, 3H), 5.43 (brs, 1H), 5.97 (s, 2H), 6.61 (d, 1H, J = 7.8 Hz), 6.65 (s, 1H), 6.74 (d, 1H, J = 7.8 Hz), 6.79 (d, 2H, J = 8.4 Hz), 6.97 (d, 2H, J = 8.4 Hz). ^{13}C NMR (CDCl_3 , 150 MHz): δ = 34.5, 40.6, 43.5, 55.2, 101.2, 108.6, 109.8, 113.9, 122.7, 128.4, 129.7, 130.6, 146.9, 148.1, 158.2, 170.9. HRMS (ESI, +): calcd for $\text{C}_{18}\text{H}_{20}\text{NO}_4$ $[\text{M}+\text{H}]^+$ 314.1392, found 314.1379.

***N*-(2-Methoxyphenylethyl)-2-phenylacetamide (Table 1, entry 3q)**

^1H NMR (CD_3OD , 600 MHz): δ = 2.66 (t, 2H, J = 6.6, 7.2 Hz), 3.19 (s, 2H), 3.27-3.29 (m, 2H), 3.67 (s, 3H), 6.69-6.71 (m, 1H), 6.78-6.80 (m, 1H), 6.88-6.90 (m, 1H), 7.03-7.07 (m, 1H), 7.09-7.13 (m, 3H), 7.16-7.19 (m, 2H). ^{13}C NMR (CD_3OD , 150 MHz): δ = 29.7, 39.5, 42.6, 54.4, 110.2, 120.5, 126.5, 127.4, 128.2, 128.7, 130.2, 135.5, 157.7, 172.6. HRMS (ESI, +): calcd for $\text{C}_{17}\text{H}_{20}\text{NO}_2$ $[\text{M}+\text{H}]^+$ 270.1494, found 270.1482.

***N*-(2-Methoxyphenylethyl)-2-(2-bromophenyl)acetamide (Table 1, entry 3r)**

^1H NMR (CD_3OD , 600 MHz): δ = 2.81 (s, 2H), 3.42 (s, 2H), 3.64 (s, 2H), 3.79 (s, 3H), 6.84-6.91 (m, 2H), 7.08-7.09 (m, 1H), 7.17 (s, 2H), 7.27-7.29 (m, 2H), 7.55-7.56 (m, 1H). ^{13}C NMR (CD_3OD , 150 MHz): δ = 31.4, 41.1, 44.3, 56.1, 111.8, 121.8, 126.3, 128.7, 129.1, 129.2, 130.2, 131.8, 133.1, 134.1, 136.9, 159.3, 172.7. HRMS (ESI, +): calcd for $\text{C}_{17}\text{H}_{19}\text{BrNO}_2$ $[\text{M}+\text{H}]^+$ 348.0599, found 366.0584.

***N*-(3,4-Dimethoxyphenylethyl)-2-phenylacetamide (Table 1, entry 3s)**

^1H NMR (CD_3OD , 600 MHz): δ = 2.86 (t, 2H, J = 7.2, 7.2 Hz), 3.45-3.46 (m, 2H), 3.53-3.56 (m, 2H), 3.93 (s, 3H), 3.95 (s, 3H), 6.81-6.83 (m, 2H), 6.92-6.95 (m, 2H), 6.97 (s, 1H), 7.34-7.43 (m, 3H). ^{13}C NMR (CD_3OD , 150 MHz): δ = 34.5, 40.6, 42.6, 55.1, 55.3, 112.1, 112.7, 120.9, 126.4, 128.2, 128.7, 132.0, 135.5, 147.8, 149.2, 172.6. HRMS (ESI, +): calcd for $\text{C}_{18}\text{H}_{22}\text{NO}_3$ $[\text{M}+\text{H}]^+$ 300.1600, found 300.1612.

***N*-(3,4-Dimethoxyphenylethyl)-2-(2-bromophenyl) acetamide (Table 1, entry 3t)¹**

^1H NMR (CD_3OD , 600 MHz): δ = 2.75 (s, 2H), 3.43 (s, 2H), 3.65 (m, 2H), 3.79-3.83 (m, 6H), 6.74 (s, 1H), 6.82 (s, 2H), 7.16 (s, 1H), 7.24-7.28 (m, 2H), 7.55-7.56 (m, 1H). ^{13}C NMR (CD_3OD , 150 MHz): δ = 36.2, 42.4, 44.3, 56.7, 56.8, 113.5, 114.1, 122.5, 126.2, 129.1, 130.2, 133.1, 133.6, 134.1, 136.8, 149.4, 150.7, 172.8. HRMS (ESI, +), calcd for $\text{C}_{18}\text{H}_{21}\text{BrNO}_3$ $[\text{M}+\text{H}]^+$ 378.0705, found 378.0787.

***N*-(3,4-Dimethoxyphenylethyl)-2-(4-bromophenyl) acetamide (Table 1, entry 3u)**

^1H NMR (CDCl_3 , 600 MHz): δ = 2.69-2.71 (m, 2H), 3.45-3.49 (m, 4H), 3.85 (s, 3H), 3.89 (s, 3H), 5.43 (brs, 1H), 6.43-6.54 (m, 1H), 6.63 (s, 1H), 6.74 (d, 1H, J = 8.4 Hz),

7.06 (d, 2H, $J = 8.4$ Hz), 7.45 (d, 2H, $J = 8.4$ Hz). ^{13}C NMR (CDCl_3 , 150 MHz): $\delta = 34.9, 40.7, 43.2, 55.8, 55.9, 111.2, 111.7, 120.6, 121.3, 130.9, 131.1, 131.5, 132.0, 133.8, 147.7, 149.1, 170.3$. MS (ESI, +): calcd for $\text{C}_{18}\text{H}_{21}\text{BrNO}_3$ $[\text{M}+\text{H}]^+$ 378.07, found 378.07.

***N*-(3,4-Dimethoxyphenylethyl)-2-(2-bromo-4-methoxyphenyl) acetamide (Table 1, entry 3v)**

^1H NMR (CDCl_3 , 600 MHz): $\delta = 2.60$ (s, 2H), 3.36-3.37 (m, 2H), 3.49 (s, 2H), 3.57-3.75 (m, 9H), 5.35 (brs, 1H), 6.49-6.53 (m, 2H), 6.61-6.62 (m, 1H), 6.70-6.71 (m, 1H), 6.99 (s, 1H), 7.05-7.06 (m, 1H). ^{13}C NMR (CDCl_3 , 150 MHz): $\delta = 35.02, 40.7, 43.2, 55.6, 55.8, 55.9, 111.2, 111.7, 113.9, 118.3, 120.6, 125.2, 126.6, 131.1, 132.0, 147.6, 148.9, 159.4, 169.9$. HRMS (ESI, +): calcd for $\text{C}_{19}\text{H}_{23}\text{BrNO}_4$ $[\text{M}+\text{H}]^+$ 408.0810, found 408.0807.

***N*-(3,4-Dimethoxyphenylethyl)-2-(3,4-dihydroxyphenyl) acetamide (Table 1, entry 3w)**

^1H NMR (CD_3OD , 600 MHz): $\delta = 2.84$ (s, 2H), 3.07 (s, 2H), 3.31-3.82 (m, 2H), 3.80-3.82 (m, 6H), 6.62-6.67 (m, 2H), 6.76-6.79 (m, 2H), 6.85 (s, 1H), 6.89-6.91 (m, 1H). ^{13}C NMR (CD_3OD , 150 MHz): $\delta = 34.0, 41.7, 44.9, 56.1, 56.1, 113.1, 113.3, 115.7, 117.0, 121.2, 121.8, 130.3, 130.5, 144.2, 145.6, 149.3, 150.4, 180.3$. HRMS (ESI, +): calcd for $\text{C}_{18}\text{H}_{22}\text{NO}_5$ $[\text{M}+\text{H}]^+$ 332.1498, found 332.1487.

***N*-(3,4-Dimethoxyphenylethyl)-2-(3,4-methylenedioxyphenyl) acetamide (Table 1, entry 3x)**

^1H NMR (CDCl_3 , 600 MHz): δ = 2.68 (t, 2H, J = 6.6, 6.6 Hz), 3.43-3.46 (m, 4H), 3.83 (s, 3H), 3.85 (s, 3H), 5.42 (brs, 1H), 5.96 (s, 2H), 6.56-6.61 (m, 3H), 6.63-6.63 (m, 1H), 6.72-6.75 (m, 2H). ^{13}C NMR (CDCl_3 , 150 MHz): δ = 35.0, 40.7, 43.5, 55.8, 55.9, 101.2, 108.6, 109.7, 111.2, 111.7, 120.6, 122.6, 128.3, 131.1, 146.9, 147.7, 148.1, 149.0, 171.0. HRMS (ESI, +): calcd for $\text{C}_{19}\text{H}_{22}\text{NO}_5$ $[\text{M}+\text{H}]^+$ 344.1498, found 344.1487.

***N*-(3,4-Dimethoxyphenylethyl)-2-(3,4-dimethoxyphenyl) acetamide (Table 1, entry 3y)**

^1H NMR (CD_3OD , 600 MHz): δ = 2.57 (t, 2H, J = 6.6, 7.2 Hz), 3.25-3.28 (m, 4H), 3.62 (s, 3H), 3.64 (s, 3H), 3.65 (s, 3H), 3.67 (s, 3H), 6.49-6.51 (m, 1H), 6.59-6.66 (m, 3H), 6.69-6.72 (m, 2H). ^{13}C NMR (CD_3OD , 150 MHz): δ = 34.4, 40.5, 42.1, 54.9, 54.9, 55.1, 55.2, 111.6, 111.9, 112.3, 112.6, 120.7, 121.1, 128.2, 131.8, 147.6, 148.1, 148.9, 149.0, 172.9. HRMS (ESI, +): calcd for $\text{C}_{20}\text{H}_{26}\text{NO}_5$ $[\text{M}+\text{H}]^+$ 360.1811, found 360.1807.

Table 2, entry 6a

^1H NMR (CD_3OD , 600 MHz): δ = 1.15-1.25 (m, 4H), 1.42-1.49 (m, 4H), 2.00-2.03 (m, 2H), 2.14-2.17 (m, 2H), 2.65-2.68 (m, 2H), 3.19-3.29 (m, 2H), 7.05-7.09 (m, 3H), 7.14-7.17 (m, 2H). ^{13}C NMR (CD_3OD , 150 MHz): δ = 24.6, 25.5, 28.5, 33.7, 35.1, 35.6,

40.5, 125.9, 128.0, 128.4, 139.1, 174.8, 176.4. MS (ESI, +): calcd for $C_{16}H_{24}NO_3$ $[M+H]^+$ 278.17, found 278.17.

Table 2, entry 6b

 1H NMR ($CDCl_3$, 600 MHz): δ = 1.26-1.37 (m, 4H), 1.58-1.63 (m, 4H), 2.09-2.13 (m, 2H), 2.31-2.36 (m, 2H), 2.74-2.76 (m, 2H), 3.47-3.49 (m, 2H), 3.79 (s, 3H), 5.46-5.51 (m, 1H), 6.85 (d, 2H, J = 8.4 Hz), 7.09-7.13 (m, 2H). ^{13}C NMR ($CDCl_3$, 150 MHz): δ = 24.5, 25.4, 28.5, 28.6, 28.7, 34.0, 36.6, 40.7, 55.3, 114.1, 129.7, 130.8, 158.3, 173.1, 178.3. MS (ESI, +): calcd for $C_{17}H_{26}NO_4$ $[M+H]^+$ 308.18, found 308.18.

Table 2, entry 6c

 1H NMR ($CDCl_3$, 600 MHz): δ = 1.25-1.37 (m, 4H), 1.59-1.65 (m, 4H), 2.09-2.14 (m, 2H), 2.32-2.35 (m, 2H), 2.75-2.77 (m, 2H), 3.48-3.51 (m, 2H), 3.86 (s, 3H), 3.87 (s, 3H), 5.50-5.59 (m, 1H), 6.71-6.72 (m, 2H), 6.80-6.81 (m, 2H). ^{13}C NMR ($CDCl_3$, 150 MHz): δ = 24.7, 25.5, 28.6, 28.7, 28.8, 33.8, 35.2, 36.6, 40.6, 55.9, 111.5, 111.9, 120.7, 131.4, 147.8, 149.1, 173.2, 178.6. MS (ESI, +): calcd for $C_{18}H_{28}NO_5$ $[M+H]^+$ 338.1967, found 338.1987.

Table 2, entry 6d

^1H NMR (CD_3OD , 600 MHz): δ = 1.15-1.25 (m, 4H), 1.42-1.49 (m, 4H), 2.00-2.03 (m, 2H), 2.14-2.17 (m, 2H), 2.66-2.68 (m, 2H), 3.19-3.29 (m, 2H), 7.05-7.09 (m, 3H), 7.14-7.17 (m, 2H). ^{13}C NMR (CD_3OD , 150 MHz): δ = 24.3, 25.7, 28.5, 33.7, 35.1, 35.6, 40.5, 125.9, 128.1, 128.4, 128.6, 139.1, 174.8, 176.4. MS (ESI, +): calcd for $\text{C}_{15}\text{H}_{22}\text{NO}_3$ $[\text{M}+\text{H}]^+$ 264.16, found 264.16.

Table 2, entry 6e

^1H NMR (CDCl_3 , 600 MHz): δ = 0.84-0.87 (m, 3H), 1.22-1.28 (m, 16H), 1.54-1.56 (m, 2H), 2.07-2.09 (m, 2H), 2.78-2.79 (m, 2H), 3.46-3.51 (m, 2H), 5.37 (brs, 1H), 7.16 (d, 2H, J = 7.8 Hz), 7.19-7.22 (m, 1H), 7.29 (t, 2H, J = 7.2, 7.2 Hz). ^{13}C NMR (CDCl_3 , 150 MHz): δ = 14.1, 22.7, 25.8, 29.2, 29.3, 29.4, 29.5, 29.6, 31.9, 35.7, 36.9, 40.5, 126.5, 128.6, 128.8, 138.9, 173.1. MS (ESI, +): calcd for $\text{C}_{20}\text{H}_{34}\text{NO}$ $[\text{M}+\text{H}]^+$ 300.24, found 300.24.

Table 2, entry 6f

^1H NMR (CDCl_3 , 600 MHz): δ = 0.87-0.89 (m, 3H), 1.25-1.29 (m, 16H), 1.57-1.59 (m, 2H), 2.09-2.12 (m, 2H), 2.74-2.76 (m, 2H), 3.47-3.49 (m, 2H), 3.79 (s, 3H), 5.39 (brs, 1H), 6.85 (d, 2H, J = 8.4 Hz), 7.11 (d, 2H, J = 8.4 Hz). ^{13}C NMR (CDCl_3 , 150 MHz): δ = 14.1, 22.7, 25.8, 29.3, 29.4, 29.5, 29.6, 31.9, 34.8, 36.9, 40.7, 55.3, 114.1, 129.7, 130.9, 158.3, 173.1. HRMS (ESI, +): calcd for $\text{C}_{21}\text{H}_{36}\text{NO}_2$ $[\text{M}+\text{H}]^+$ 334.2746, found 334.2764.

Table 2, entry 6g

 ^1H NMR (CDCl_3 , 600 MHz): δ = 0.87-0.89 (m, 3H), 1.25-1.29 (m, 16H), 1.58-1.59 (m, 2H), 2.10-2.13 (m, 2H), 2.75-2.77 (m, 2H), 3.48-3.52 (m, 2H), 3.86 (s, 3H), 3.87 (s, 3H), 5.41 (brs, 1H), 6.72-6.73 (m, 2H), 6.80-6.82 (m, 2H). ^{13}C NMR (CDCl_3 , 150 MHz): δ = 14.1, 22.7, 25.8, 29.3, 29.4, 29.5, 29.6, 31.9, 35.3, 36.9, 40.6, 55.8, 55.9, 111.4, 111.9, 120.7, 131.5, 147.7, 149.1, 173.1. HRMS (ESI, +): calcd for $\text{C}_{22}\text{H}_{38}\text{NO}_3$ $[\text{M}+\text{H}]^+$ 364.2852, found 364.2864.

Table 2, entry 6h

 ^1H NMR (CDCl_3 , 600 MHz): δ = 0.87-0.89 (m, 3H), 1.25-1.29 (m, 16H), 1.63-1.68 (m, 2H), 2.19-2.22 (m, 2H), 4.44-4.45 (m, 2H), 5.72 (brs, 1H), 7.26-7.28 (m, 3H), 7.32-7.35 (m, 2H); ^{13}C NMR (CDCl_3 , 150 MHz): δ = 14.1, 22.7, 25.8, 29.3, 29.4, 29.5, 29.6, 31.9, 36.9, 43.6, 127.5, 127.9, 128.7, 138.4, 172.9. HRMS (ESI, +): calcd for $\text{C}_{19}\text{H}_{32}\text{NO}$ $[\text{M}+\text{H}]^+$ 290.2484, found 290.2497.

Table 2, entry 6i

^1H NMR (CDCl_3 , 600 MHz): δ = 0.88-0.89 (m, 3H), 1.23-1.28 (m, 28H), 1.54-1.57 (m, 2H), 2.07-2.09 (m, 2H), 2.78-2.80 (m, 2H), 3.48-3.51 (m, 2H), 5.40 (brs, 1H), 7.14-7.17 (m, 2H), 7.21-7.23 (m, 1H), 7.27-7.29 (m, 2H). ^{13}C NMR (CDCl_3 , 150 MHz): δ = 14.1, 22.7, 25.8, 29.3, 29.4, 29.5, 29.6, 29.7, 31.9, 35.7, 36.9, 40.5, 125.3, 126.5, 128.2, 128.6, 128.8, 129.0, 138.9, 173.1. MS (ESI, +): calcd for $\text{C}_{26}\text{H}_{46}\text{NO}$ $[\text{M}+\text{H}]^+$ 388.35, found 388.35.

Table 2, entry 6j

^1H NMR (CDCl_3 , 600 MHz): δ = 0.88-0.89 (m, 3H), 1.25-1.29 (m, 28H), 1.57-1.59 (m, 2H), 2.09-2.12 (m, 2H), 2.74-2.76 (m, 2H), 3.47-3.49 (m, 2H), 3.79 (s, 3H), 5.39 (brs, 1H), 6.85 (d, 2H, J = 7.8 Hz), 7.10 (d, 2H, J = 8.4 Hz). ^{13}C NMR (CDCl_3 , 150 MHz): δ = 14.1, 22.7, 25.8, 29.3, 29.5, 29.6, 29.7, 31.9, 34.8, 36.9, 40.6, 55.3, 114.1, 129.7, 130.9, 158.3, 173.0. HRMS (ESI, +): calcd for $\text{C}_{27}\text{H}_{48}\text{NO}_2$ $[\text{M}+\text{H}]^+$ 418.3685, found 418.3677.

Table 2, entry 6k

^1H NMR (CDCl_3 , 600 MHz): δ = 0.86-0.88 (m, 3H), 1.25 (m, 28H), 1.57-1.59 (m, 2H), 2.09-2.12 (m, 2H), 2.74-2.77 (m, 2H), 3.48-3.51 (m, 2H), 3.85 (s, 3H), 3.86 (s, 3H), 5.43 (brs, 1H), 6.72 (d, 2H, J = 8.4 Hz), 6.80 (d, 2H, J = 7.8 Hz). ^{13}C NMR (CDCl_3 , 150 MHz): δ = 14.1, 22.7, 25.8, 29.3, 29.4, 29.5, 29.6, 29.7, 31.9, 35.3, 36.9, 40.6, 55.8, 55.9, 111.4, 111.9, 120.7, 131.5, 147.7, 149.1, 173.1. HRMS (ESI, +): calcd for $\text{C}_{28}\text{H}_{50}\text{NO}_3$ $[\text{M}+\text{H}]^+$ 448.3791, found 448.3856.

Table 2, entry 6l

^1H NMR (CDCl_3 , 600 MHz): δ = 0.77-0.78 (m, 3H), 1.15-1.19 (m, 28H), 1.52-1.55 (m, 2H), 2.08-2.10 (m, 2H), 4.31-4.32 (m, 2H), 5.78 (brs, 1H), 7.15-7.16 (m, 3H), 7.20-7.23 (m, 2H). ^{13}C NMR (CDCl_3 , 150 MHz): δ = 14.1, 22.7, 24.9, 25.8, 29.2, 29.3, 29.4, 29.5, 29.6, 29.7, 31.9, 36.8, 43.6, 127.5, 127.8, 128.7, 138.5, 173.1. HRMS (ESI, +): calcd for $\text{C}_{25}\text{H}_{44}\text{NO}$ $[\text{M}+\text{H}]^+$ 374.3423, found 374.3454.

1-(Benzyl)-1,2,3,4-tetrahydroisoquinoline (Table 3, entry 8a)

^1H NMR (CDCl_3 , 600 MHz): δ = 2.75-2.89 (m, 6H), 3.49 (s, 1H), 6.95-7.01 (m, 2H), 7.11-7.28 (m, 7H). ^{13}C NMR (CDCl_3 , 150 MHz): δ = 29.7, 36.3, 49.6, 50.9, 115.4, 124.1, 126.7, 127.7, 128.3, 129.5, 131.2, 139.9.

1-(2-Iodobenzyl)-1,2,3,4-tetrahydroisoquinoline (Table 3, entry 8b)¹

^1H NMR (CD_3OD , 600 MHz) : δ = 2.67-2.73 (m, 4H), 2.77-2.82 (m, 3H), 6.78-6.81 (m, 1H), 7.02-7.11 (m, 3H), 7.14-7.24 (m, 3H), 7.67-7.69 (m, 1H). ^{13}C NMR (CD_3OD , 150 MHz) : δ = 28.7, 34.5, 48.3, 49.8, 98.9, 125.4, 127.4, 127.5, 127.6, 127.7, 127.8, 127.9, 128.3, 128.9, 138.7, 141.4.

1-(2-Bromo-4-methoxybenzyl)-1,2,3,4- tetrahydroisoquinoline (Table 3, entry 8c)

^1H NMR (CDCl_3 , 600 MHz): δ = 2.18-2.1 (m, 5H), 2.88 (s, 2H), 3.79 (s, 3H), 6.77-6.79 (m, 1H), 7.09-7.19 (m, 1H), 7.19-7.23 (m, 3H), 7.29-7.31 (m, 2H). ^{13}C NMR (CDCl_3 , 150 MHz): δ = 35.4, 36.0, 49.3, 50.7, 55.5, 113.6, 118.1, 124.5, 126.2, 128.5, 128.7, 131.0, 139.8, 158.6.

1-(Benzyl)-1,2,3,4-tetrahydro-7-methoxytetrahydroisoquinoline (Table 3, entry 8d)

^1H NMR (CDCl_3 , 600 MHz): δ = 2.08-2.09 (m, 2H), 2.85-2.88 (m, 2H), 2.91-2.93 (m, 1H), 2.98-3.03 (m, 2H), 3.84 (s, 3H), 6.85-6.92 (m, 2H), 7.11-7.33 (m, 6H). ^{13}C NMR (CDCl_3 , 150 MHz): δ = 29.7, 34.7, 49.9, 50.1, 55.3, 114.1, 126.5, 128.5, 128.7, 129.7, 130.6, 158.3.

1-(2-Bromobenzyl)-1,2,3,4-tetrahydro-7-methoxytetrahydroisoquinoline (Table 3, entry 8e)¹

^1H NMR (CD_3OD , 600 MHz): δ = 2.61-2.63 (m, 2H), 2.71-2.73 (m, 3H), 2.80-2.83 (m, 2H), 3.64 (s, 3H), 6.70-6.72 (m, 2H), 6.91-6.99 (m, 3H), 7.14-7.19 (m, 1H), 7.41-7.42 (m, 1H). ^{13}C NMR (CD_3OD , 150 MHz): δ = 34.2, 35.4, 48.6, 50.5, 54.3, 113.6, 123.9, 127.5, 127.9, 129.2, 130.6, 131.3, 132.6, 138.7, 158.3.

1-(2-Iodobenzyl)-1,2,3,4-tetrahydro-7-methoxytetrahydroisoquinoline (Table 3, entry 8f)

^1H NMR (CD_3OD , 600 MHz): δ = 2.76-2.78 (m, 2H), 2.86-2.90 (m, 5H), 3.68 (s, 3H), 6.73-6.79 (m, 1H), 6.80-6.81 (m, 1H), 6.99-7.01 (m, 1H), 7.06-7.07 (m, 1H), 7.12-7.14 (m, 1H), 7.16-7.17 (m, 1H), 7.69-7.70 (m, 1H). ^{13}C NMR (CD_3OD , 150 MHz): δ = 28.8, 29.3, 38.5, 48.2, 54.4, 99.3, 110.3, 120.4, 127.5, 127.9, 128.5, 129.6, 130.0, 139.6, 140.8, 157.6.

1-(-Benzyl)-1,2,3,4-tetrahydro-5-methoxytetrahydroisoquinoline (Table 3, entry 8g)

^1H NMR (CDCl_3 , 600 MHz): δ = 2.70-2.83 (m, 7H), 3.70 (s, 3H), 6.73-6.78 (m, 2H), 7.07-7.13 (m, 3H), 7.16-7.19 (m, 3H). ^{13}C NMR (CDCl_3 , 150 MHz): δ = 30.6, 36.1, 49.4, 50.8, 55.2, 110.4, 120.5, 126.2, 127.5, 128.2, 128.5, 128.7, 129.5, 130.4, 139.9, 157.6.

1-(2-Bromobenzyl)-1,2,3,4-tetrahydro-5-methoxytetrahydroisoquinoline (Table 3, entry 8h)

^1H NMR (CD_3OD , 600 MHz): δ = 2.69-2.80 (m, 7H), 3.66 (s, 3H), 6.69-6.77 (m, 2H), 6.96-6.97 (m, 2H), 7.03-7.12 (m, 2H), 6.39 (d, 1H, J = 7.8 Hz). ^{13}C NMR (CD_3OD , 150 MHz): δ = 29.9, 35.4, 48.6, 48.9, 54.4, 110.2, 120.2, 123.9, 127.4, 127.5, 127.9, 129.9, 130.6, 132.6, 138.8, 157.6.

1-(Benzyl)-1,2,3,4-tetrahydro-6,7-dimethoxytetrahydroisoquinoline (Table 3, entry 8i)²

^1H NMR (CD_3OD , 600 MHz): δ = 2.75 (s, 2H), 3.10-3.11 (m, 2H), 3.13-3.23 (m, 3H, mix with solvent signal), 3.48 (s, 3H), 3.65 (s, 3H), 6.34 (s, 1H), 6.59 (s, 1H), 7.15-7.16 (m, 3H), 7.21-7.22 (m, 2H). ^{13}C NMR (CD_3OD , 150 MHz): δ = 26.7, 39.1, 41.2, 54.9, 55.0, 56.2, 110.2, 111.8, 125.6, 126.6, 127.2, 128.5, 129.4, 137.6, 147.3, 148.3.

1-(2-Bromobenzyl)-1,2,3,4-tetrahydro-6,7-dimethoxytetrahydroisoquinoline (Table 3, entry 8j)¹

^1H NMR (CD_3OD , 600 MHz): δ = 3.00-3.30 (m, 3H), 3.40 (s, 3H), 3.43-3.54 (m, 4H), 3.73 (s, 3H), 6.04 (s, 1H), 6.70 (s, 1H), 7.14-7.16 (m, 1H), 7.23-7.27 (m, 2H), 7.55 (d,

1H, $J = 7.8$ Hz). ^{13}C NMR (CD_3OD , 150 MHz): $\delta = 25.3, 39.2, 40.8, 54.9, 55.6, 55.8, 110.8, 112.8, 123.7, 124.8, 125.7, 128.7, 130.0, 133.3, 133.8, 136.0, 148.3, 149.9$.

1-(3,4-dimethoxybenzyl)-1,2,3,4-tetrahydro-6,7-dimethoxytetrahydroisoquinoline (Table 3, entry 8k)

^1H NMR (CD_3OD , 600 MHz): $\delta = 2.65\text{-}2.66$ (m, 2H), $2.77\text{-}2.84$ (m, 2H), $3.07\text{-}3.11$ (m, 2H), 3.53 (s, 3H), 3.61 (s, 6H), $3.70\text{-}3.76$ (m, 1H), $6.57\text{-}6.61$ (m, 2H), $6.72\text{-}6.73$ (m, 1H), $6.75\text{-}6.83$ (m, 2H). ^{13}C NMR (CD_3OD , 150 MHz): $\delta = 28.4, 39.8, 41.6, 54.9, 55.1, 55.2, 56.5, 110.3, 112.0, 113.0, 121.6, 127.1, 129.9, 131.5, 147.1, 147.9, 149.2$.

^1H and ^{13}C NMR Spectra of compounds:

***N*-(Phenylethyl)-2-phenylacetamide (Table 1, entry 3a)**

1H Experiment

N-(Phenylethyl)-2-(2-Iodophenyl)acetamide (Table 1, entry 3b)

N-(Phenylethyl)-2-(2-bromophenyl)acetamide (Table 1, entry 3c)

N-(Phenylethyl)-2-(2-bromo-4-methoxyphenyl)acetamide (Table 1, entry 3d)

N-(Phenylethyl)-2-(3,4-dimethoxyphenyl)acetamide (Table 1, entry 3e)

***N*-(Phenylethyl)-2-(3,4-methylenedioxyphenyl)acetamide (Table 1, entry 3f)**

1H Experiment

Cur: aas30ar
 NAME: aas30ar
 EXPNO: 1
 PROCNO: 1

F2 - Acquisition Parameters
 Date_: 20140226
 Time: 10.40
 INSTRUM: spect
 PROBHD: 5 mm PABBO BB/
 PULPROG: zg30
 TD: 65536
 SOLVENT: CDCl3
 NS: 16
 DS: 2
 SWH: 12019.230 Hz
 FIDRES: 0.183399 Hz
 AQ: 2.7263477 sec
 RG: 31.77
 DW: 41.600 usec
 DE: 6.50 usec
 TE: 291.9 K
 D1: 1.00000000 sec
 TDO: 1

===== CHANNEL f1 =====
 SPOL: 600.3029254 MHz
 NUC1: 1H
 P1: 11.47 usec
 PLW1: 25.00000000 W

F2 - Processing parameters
 SI: 65536
 SP: 600.2999860 MHz
 WDW: EM
 SSB: 0
 LB: 0.30 Hz
 GB: 0
 PC: 1.40

13C Experiment

Cur: rs
 NAME: aas30ar
 EXPNO: 2
 PROCNO: 1

***N*-(2-Fluorophenylethyl)-2-(2-bromophenyl)acetamide (Table 1, entry 3g)**

1H Experiment

BRUKER

Cur
NAME: aas35b
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20120914
Time: 11.06
INSTRUM: spect
PROBHD: 5 mm F4BBO BB/
PULPROG: zg30
TD: 65536
SOLVENT: MeOD
NS: 16
DS: 2
SWH: 12019.230 Hz
FIDRES: 0.183399 Hz
AQ: 2.7263477 sec
RG: 77.74
DW: 41.600 usec
DE: 6.50 usec
TE: 295.7 K
D1: 1.00000000 sec
TD0: 1

===== CHANNEL f1 =====
SFO1: 600.3029254 MHz
NUC1: 1H
P1: 11.47 usec
PLW1: 25.00000000 W

F2 - Processing parameters
SI: 65536
SF: 600.3000140 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.40

13C Experiment

BRUKER

Cu: rs
NAME: aas35b
EXPNO: 2
PROCNO: 1

N-(2-Fluorophenylethyl)-2-(3,4-dihydroxyphenyl)acetamide (Table 1, entry 3h)

1H Experiment

N-(4-Methoxyphenylethyl)-2-(2-bromophenyl)acetamide (Table 1, entry 3j)

1H Experiment

N-(4-Methoxyphenylethyl)-2-(2-iodophenyl)acetamide (Table 1, entry 3k)

¹H Experiment

Cur
NAME
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121022
Time 11.34
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 86.1
DW 41.600 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 600.3029254 MHz
NUC1 1H
P1 11.47 usec
PLW1 25.00000000 W

F2 - Processing parameters
SI 65536
SF 600.3000140 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

¹³C Experiment

Cur
NAME
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121022
Time 12.15
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 796
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 182
DW 13.867 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
NUC1 13C
P1 11.15 usec
PLW1 100.00000000 W
SFO1 150.9606489 MHz
CPDPRG2
NUC2 1H
PLW2 25.00000000 W
PLW12 0.67123002 W
PLW13 0.32890001 W
SFO2 600.3024012 MHz

F2 - Processing parameters
SI 32768
SF 150.9453344 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

N-(4-Methoxyphenylethyl)-2-(4-bromophenyl)acetamide (Table 1, entry 3l)

1H Experiment

Current Data Parameters
NAME aas88b
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140225
Time 11.32
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 28.15
DW 41.600 usec
DE 6.50 usec
TE 292.1 K
D1 1.00000000 sec
TDO 1

===== CHANNEL f1 =====
SFO1 600.3029254 MHz
NUC1 1H
P1 11.47 usec
PLW1 25.00000000 W

F2 - Processing parameters
SI 65536
SF 600.3000228 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

Current Data Parameters
NAME aas88b
EXPNO 2
PROCNO 1

***N*-(4-Methoxyphenylethyl)-2-(4-bromo-4-methoxyphenyl)acetamide (Table 1, entry 3m)**

N-(4-Methoxyphenylethyl)-2-(3,4-dihydroxyphenyl)acetamide (Table 1, entry 3n)

1H Experiment

13C Experiment

N-(4-Methoxyphenylethyl)-2-(3,4-dimethoxyphenyl)acetamide (Table 1, entry 3o)

1H Experiment

N-(2-Methoxyphenylethyl)-2-phenylacetamide (Table 1, entry 3q)

1H Experiment

Current Data Parameters
NAME: aas66b
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20131007
Time: 10.00
INSTRUM: spect
PROBHD: 5 mm PABBO BB/
PULPROG: zg30
TD: 65536
SOLVENT: MeOD
NS: 16
DS: 2
SWH: 12019.230 Hz
FIDRES: 0.183399 Hz
AQ: 2.7263477 sec
RG: 44.08
DW: 41.600 usec
DE: 6.50 usec
TE: 311.7 K
D1: 1.00000000 sec
TDO: 1

==== CHANNEL f1 =====
SFO1: 600.3029254 MHz
NUC1: 1H
P1: 11.47 usec
PLW1: 25.00000000 W

F2 - Processing parameters
SI: 65536
SF: 600.3000781 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.40

N-(2-Methoxyphenylethyl)-2-(2-bromophenyl)acetamide (Table 1, entry 3r)

1H Experiment

Cur NAME: aas34b
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20120903
Time: 9.42
INSTRUM: spect
PROBHD: 5 mm PABBO BB/
PULPROG: zg30
TD: 65536
SOLVENT: MeOD
NS: 16
DS: 2
SWH: 12019.230 Hz
FIDRES: 0.183399 Hz
AQ: 2.7263477 sec
RG: 40.07
DW: 41.600 usec
DE: 6.50 usec
TE: 296.1 K
D1: 1.00000000 sec
TD0: 1

===== CHANNEL f1 =====
SF01: 600.3029254 MHz
NUC1: 1H
P1: 11.47 usec
PIN1: 25.00000000 W

F2 - Processing parameters
SI: 65536
SP: 600.3000140 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.40

13C Experiment

Cur NAME: aas34b
EXPNO: 2
PROCNO: 1

N-(3,4-Dimethoxyphenylethyl)-2-phenylacetamide (Table 1, entry 3s)

¹H Experiment

Current Data Parameters
NAME aas64a
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20131003
Time 12.20
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 40.07
DW 41.600 usec
DE 6.50 usec
TE 312.4 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 600.3029254 MHz
NUC1 ¹H
P1 11.47 usec
PLW1 25.00000000 W

F2 - Processing parameters
SI 65536
SF 600.2999219 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

¹³C Experiment

Current Data Parameters
NAME aas64a
EXPNO 2
PROCNO 1

N-(3,4-Dimethoxyphenylethyl)-2-(2-bromophenyl)acetamide (Table 1, entry 3t)

¹H Experiment

Cur
NAME
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120911
Time 14.24
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 63.12
DW 41.600 usec
DE 6.50 usec
TE 295.9 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 600.3029254 MHz
NUC1 1H
P1 11.47 usec
PLW1 25.00000000 W

F2 - Processing parameters
SI 65536
SF 600.3000140 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

¹³C Experiment

N-(3,4-Dimethoxyphenylethyl)-2-(4-bromophenyl)acetamide (Table 1, entry 3u)

1H Experiment

N-(3,4-Dimethoxyphenylethyl)-2-(2-bromo-4-methoxyphenyl)acetamide (Table 1, entry 3v)

1H Experiment

N-(3,4-Dimethoxyphenylethyl)-2-(3,4-dihydroxyphenyl)acetamide (Table 1, entry 3w)

¹H Experiment

Cur
NAME_000000
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120917
Time 16.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 77.74
DW 41.600 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 600.3029254 MHz
NUC1 1H
P1 11.47 usec
PLW1 25.00000000 W

F2 - Processing parameters
SI 65536
SF 600.3000140 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

¹³C Experiment

Cur
NAME_000000
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120917
Time 16.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 182
DW 13.867 usec
DE 6.50 usec
TE 299.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
NUC1 13C
P1 11.15 usec
PLW1 100.00000000 W
SFO1 150.9606489 MHz
CPDPRG2
NUC2 1H
PLW2 25.00000000 W
PLW12 0.67123002 W
PLW13 0.32890001 W
SFO2 600.3024012 MHz

F2 - Processing parameters
SI 32768
SF 150.9454038 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

N-(3,4-Dimethoxyphenylethyl)-2-(3,4-methylenedioxyphenyl)acetamide (Table 1, entry 3x)

N-(3,4-Dimethoxyphenylethyl)-2-(3,4-dimethoxyphenyl)acetamide (Table 1, entry 3y)

1H Experiment

Table 2, entry 6a

¹H Experiment

Current Data Parameters
NAME: aas92a
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20140227
Time: 14.13
INSTRUM: spect
PROBHD: 5 mm PABBO BB/
PULPROG: zg30
TD: 65536
SOLVENT: MeOD
NS: 26
DS: 2
SWH: 12019.230 Hz
FIDRES: 0.183399 Hz
AQ: 2.7263477 sec
RG: 28.15
DW: 41.600 usec
DE: 6.50 usec
TE: 293.9 K
D1: 1.00000000 sec
TDO: 1

===== CHANNEL f1 =====
SFO1: 600.3029254 MHz
NUC1: ¹H
P1: 11.47 usec
PLW1: 25.00000000 W

F2 - Processing parameters
SI: 65536
SF: 600.3000800 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.40

¹³C Experiment

Current Data Parameters
NAME: aas92a
EXPNO: 2
PROCNO: 1

Table 2, entry 6b

1H Experiment

Table 2, entry 6c

¹H Experiment

Table 2, entry 6c

1H Experiment

Table 2, entry 6f

1H Experiment

Table 2, entry 6g

1H Experiment

Table 2, entry 6h

1H Experiment

Table 2, entry 6i

¹H Experiment

Table 2, entry 6j

1H Experiment

Table 2, entry 6k

¹H Experiment

Table 2, entry 61

1H Experiment

1-(2-Iodobenzyl)-1,2,3,4-tetrahydroisoquinoline (Table 3, entry 8b)

1-(2-Bromo-4-methoxybenzyl)-1,2,3,4-tetrahydroisoquinoline (Table 3, entry 8c)

1-(Benzyloxy)-1,2,3,4-tetrahydro-7-methoxytetrahydroisoquinoline (Table 3, entry 8d)

1-(2-Bromobenzyl)-1,2,3,4-tetrahydro-7-methoxytetrahydroisoquinoline (Table 3, entry 8e)

1-(2-Iodobenzyl)-1,2,3,4-tetrahydro-7-methoxytetrahydroisoquinoline (Table 3, entry 8f)

1-(-Benzyl)-1,2,3,4-tetrahydro-5-methoxytetrahydroisoquinoline (Table 3, entry 8g)

1-(2-Bromobenzyl)-1,2,3,4-tetrahydro-5-methoxytetrahydroisoquinoline (Table 3, entry 8h)

1-(Benzyl)-1,2,3,4-tetrahydro-6,7-dimethoxytetrahydroisoquinoline (Table 3, entry 8i)

Current Data Parameters
NAME aas64a1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20131010
Time 12.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 12019.230
FIDRES 0.183399
AQ 2.7263477
RG 18.64
DW 41.600
DE 6.50
TE 296.2
D1 4.00000000
TD0 1

----- CHANNEL f1 -----
SFO1 600.3029254
NUC1 1H
P1 11.47
PLW1 25.00000000

F2 - Processing parameters
SI 65536
SF 600.3000831
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.40

Current Data Parameters
NAME aas64a1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20131010
Time 12.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 36057.1
FIDRES 0.550000
AQ 0.908800
RG 13.1
DW 13.1
DE 6.5
TE 296.2
D1 2.000000
d11 0.030000
DELTA 1.899999
TD0 1
NUC1 13C
P1 11.47
PLW1 100.000000
SFO1 150.960600
CPDPRG2
NUC2 13C
PLW2 25.000000
PLW12 0.671230
PLW13 0.328900
SFO2 600.302400

F2 - Processing parameters
SI 32768
SF 150.945500
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.40

1-(2-Bromobenzyl)-1,2,3,4-tetrahydro-6,7-dimethoxytetrahydroisoquinoline (Table 3, entry 8j)

1-(3,4-dimethoxybenzyl)-1,2,3,4-tetrahydro-6,7-dimethoxytetrahydroisoquinoline (Table 3, entry 8k)

References:

- 1 M. Lafrance, N. Blaquiere and K. Fagnou, *Eur. J. Org. Chem.* 2007, **2007**, 811-825.
- 2 E. Awuah and A. Capretta, *J. Org. Chem.* **2010**, 75, 5627-5634.