Supporting Information

Ceric Ammonium Nitrate (CAN) catalyzed efficient one-pot three component aza-Diels-Alder reactions for a facile synthesis of tetrahydropyranoquinoline derivatives

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4. Experimental

4.1 Materials and methods

All the commercial reagents and solvents were used without further purification unless otherwise stated. Melting points were recorded on a Buchi 535 melting point apparatus and are uncorrected. All the reactions were monitored by thin layer chromatography performed on precoated silica gel 60F₂₅₄ plates (Merck). Compounds were visualized with UV light at 254 nm and 365 nm, I₂ and heating plates after dipping in 2% phosphomolybdic acid in 15% aq. H₂SO₄ soln. IR spectra were recorded on a Perkin-Elmer 683 or a 1310 FT-IR spectrometers with KBr pellets. NMR spectra were recorded on a Varian Unity-400 MHz and BRUKER AMX 300 spectrometers using TMS as an internal standard. ¹³C NMR was recorded on a Varian Unity 100 MHz using CDCl₃ as internal standard. Mass spectra were recorded on a VG Micromass 7070H and a Finnigan Mat 1020B mass spectrometers operating at 70eV.

Typical experimental procedure for the preparation of compounds tetrahydroquinolines:

Benzaldehyde (1.1 mmol), Aniline (1.1 mmol), 3,4-dihydro-2*H*-pyran (1.3 mmol) and in acetonitrile (5 mL) were mixed in a flask and CAN (10 mol %) was added at room temperature. The resulting mixture was stirred at room temperature for 30 min and completion of the reaction was monitored by TLC (ethylacetate : hexane 4:6). The reaction mixture was extracted into CH₃Cl and dried over Na₂SO₄ and solvent was evaporated and the crude residue obtained was purified by column chromatography [silica gel, ethyl acetate-hexane 10:90] to give pure tetrahydropyranoquinolines **4** and **5** in 97 % yield.

5-Phenyl-3,4,4a,5,6,10b-hexahydro-2H-pyrano[3,2-c]quinoline (4&5 a) [*Cis isomer*]: Pale Yellow Solid; m.p. 131-133 °C. IR (KBr): $\nu_{\max}$ 3311, 3020, 2923, 2857, 1605, 1493, 1066, 752 cm^{-1} . ^1H NMR (300 MHz, CDCl_3), δ : 1.27-1.53 (m, 4H), 2.16-2.20 (m, 1H), 3.40 (d, $J = 2.4$, 11.3 Hz, 1H), 3.55 (dd, $J = 2.4$, 11.3 Hz, 1H), 3.80 (s, 1H), 4.70 (d, $J = 2.7$ Hz, 1H), 5.30 (d, $J = 5.5$ Hz, 1H), 6.55 (d, $J = 8.0$ Hz, 1H), 6.78 (t, $J = 8.0$ Hz, 1H), 7.05 (t, $J = 7.8$ Hz, 1H), 7.25-7.45 (m, 6H).; MS (ESI): $m/z = 265$ ($[\text{M}+\text{H}]^+$).

5-Phenyl-3,4,4a,5,6,10b-hexahydro-2H-pyrano[3,2-c]quinoline [*Trans isomer*]: viscous oil; IR (KBr): $\nu_{\max}$ 3378, 3061, 2945, 2857, 1612, 1499, 1366, 708 cm^{-1} .; MS (ESI): $m/z = 265$ ($[\text{M}+\text{H}]^+$). ^1H NMR (300 MHz, CDCl_3), δ 1.27 (m, 1H), 1.58 (m, 1H), 1.69 (m, 1H), 1.85 (m, 1H), 3.76 (dt, $J = 2.5$, 11.6 Hz, 1H), 4.11 (m, 2H), 4.35 (d, $J = 3.1$ Hz, 1H), 4.72 (d, $J = 10.9$ Hz, 1H), 6.59 (d, $J = 7.4$ Hz, 1H), 6.73 (t, $J = 7.2$ Hz, 1H), 7.11 (t, $J = 7.2$ Hz, 1H), 7.27 (d, $J = 7.4$ Hz, 1H), 7.39-7.46 (m, 5H).

5-(3-Methoxy-phenyl)-3,4,4a,5,6,10b-hexahydro-2H-pyrano[3,2-c]quinoline (4&5b):
Cis isomer : Thick syrup; R_f (20% EtOAc/n-hexane) 0.5.; IR (KBr) ($\nu_{\max}$, cm^{-1}): 3374 (N-H), 3040, 2923, 2855, 1515, 1345, 1068, 759; ^1H NMR (300 MHz, CDCl_3), δ : 1.31-1.68 (m, 4H), 2.09-2.18 (m, 1H); 3.67 (m, 1H), 3.81 (s, 3H), 4.64 (d, $J = 2.2$ Hz, 1H), 5.27 (d, $J = 5.2$ Hz, 1H), 6.54 (d, $J = 8.3$ Hz, 1H), 6.78 (t, $J = 11.3$ Hz, 1H), 7.04 (t, $J = 7.5$ Hz, 3H), 7.24 (d, $J = 7.5$ Hz, 1H), 7.37 (d, 1H); MS (ESI) m/z 296 ($[\text{M}+\text{H}]^+$). Anal. Calcd. for $\text{C}_{19}\text{H}_{21}\text{NO}_2$: C, 77.26; H, 7.17; N, 4.74; O, 10.83. Found: C, 77.23; H, 7.17; N, 4.75.

Trans isomer : Mp 99-100 °C; R_f (20% EtOAc/n-hexane) 0.48.; IR (KBr) ($\nu_{\max}$, cm^{-1}): 3374 (N-H), 3040, 2923, 2855, 1515, 1345, 1068, 759; ^1H NMR (300 MHz, CDCl_3), δ : 1.37-

1.55 (m, 4H), 2.09-2.21 (m, 1H), 3.69 (dt, $J = 3.0, 9.8$ Hz, 1H), 3.79 (s, 3H), 4.09 (dd, $J = 3.7, 11.3$ Hz, 1H), 4.34 (d, $J = 2.2$ Hz, 1H), 4.67 (d, $J = 10.5$ Hz, 1H), 6.48 (d, $J = 7.5$ Hz, 1H), 6.65 (t, $J = 8.3$ Hz, 1H), 6.81 (dd, $J = 3.7, 8.3$ Hz, 1H), 6.94 (d, $J = 8.3$ Hz, 2H), 7.04 (dt, $J = 1.51, 7.5$ Hz, 1H), 7.16 (dd, $J = 1.5, 7.5$ Hz, 1H), 7.24 (dd, $J = 1.5, 7.5$ Hz, 1H); MS (ESI) m/z 296 ($[M+H]^+$). Anal. Calcd. for $C_{19}H_{21}NO_2$: C, 77.26; H, 7.17; N, 4.74; O, 10.83. Found: C, 77.25; H, 7.18; N, 4.74.

5-(2,4-Dichloro-phenyl)-3,4,4a,5,6,10b-hexahydro-2H-pyrano[3,2-c]quinoline (4& 5e): *Cis isomer*; Mp 133-136 °C; R_f (20% EtOAc/n-hexane) 0.5.; IR (KBr) (ν_{max}, cm^{-1}): 3374 (N-H), 3040, 2923, 2855, 1515, 1345, 1068, 759; 1H NMR (300 MHz, $CDCl_3$), δ : 1.39-1.57 (m, 4H), 2.18-2.38 (m, 1H), 3.42 (dt, $J = 3.0, 11.3$ Hz, 1H), 3.58 (dd, $J = 3.7, 11.3$ Hz, 1H), 5.01 (d, $J = 2.2$ Hz, 1H), 5.26 (d, $J = 5.2$ Hz, 1H), 6.55 (d, $J = 6.8$ Hz, 1H), 6.77 (t, $J = 7.5$ Hz, 1H), 7.03 (t, $J = 7.5$ Hz, 1H), 7.27 (d, $J = 8.3$ Hz, 1H), 7.64 (m, 2H), 7.62 (d, $J = 8.3$ Hz, 2H); MS (ESI) m/z 334 ($[M+H]^+$). Anal. Calcd. for $C_{18}H_{17}Cl_2NO$: C, 64.68; H, 5.13; Cl, 21.21; N, 4.19; O, 4.79. Found: C, 64.66; H, 5.14; N, 4.18.

Trans isomer : Thick syrup; R_f (20% EtOAc/n-hexane) 0.45.; IR (KBr) (ν_{max}, cm^{-1}): 3374 (N-H), 3040, 2923, 2855, 1515, 1345, 1068, 759; 1H NMR (300 MHz, $CDCl_3$), δ : 1.39-1.57 (m, 4H), 2.18-2.38 (m, 1H), 3.63 (dt, $J = 3.0, 11.3$ Hz, 1H), 3.92 (dd, $J = 3.7, 11.3$ Hz, 1H), 4.37 (d, $J = 3.7$ Hz, 1H), 5.09 (d, $J = 8.3$ Hz, 1H), 6.49 (d, $J = 6.8$ Hz, 1H), 6.69 (t, $J = 7.5$ Hz, 1H), 7.05 (t, $J = 8.3$ Hz, 1H), 7.24 (m, 3H), 7.39-7.45 (m, 2H); MS (ESI) m/z 334 ($[M+H]^+$). Anal. Calcd. for $C_{18}H_{17}Cl_2NO$: C, 64.68; H, 5.13; Cl, 21.21; N, 4.19; O, 4.79. Found: C, 64.68; H, 5.12; N, 4.20

5-(4-chloro-phenyl)-3,4,4a,5,6,10b-hexahydro-2H-pyrano[3,2-c]quinoline (4 & 5d): *Cis isomer*; Mp 132-134 °C; R_f (20% EtOAc/n-hexane) 0.5.; IR (KBr) (ν_{max}, cm^{-1}): 3374 (N-H), 3040, 2923,

2855, 1515, 1345, 1068, 759; ^1H NMR (300 MHz, CDCl_3), δ : 1.39-1.57 (m, 4H), 2.18-2.38 (m, 1H), 3.42 (dt, $J = 3.0, 11.3$ Hz, 1H), 3.58 (dd, $J = 3.7, 11.3$ Hz, 1H), 5.01 (d, $J = 2.2$ Hz, 1H), 5.26 (d, $J = 5.2$ Hz, 1H), 6.55 (d, $J = 6.8$ Hz, 1H), 6.77 (t, $J = 7.5$ Hz, 1H), 7.03 (t, $J = 7.5$ Hz, 1H), 7.27 (d, $J = 8.3$ Hz, 1H), 7.64 (m, 2H), 7.62 (d, $J = 8.3$ Hz, 2H); MS (ESI) m/z 300 ($[\text{M}+\text{H}]^+$).

HRMS: calcd for $\text{C}_{18}\text{H}_{18}\text{ON}$: 300.11497 Found 300.11414.

Trans isomer : Thick syrup; R_f (20% EtOAc/n-hexane) 0.45.; IR (KBr) (ν_{max} , cm^{-1}): 3374 (N-H), 3040, 2923, 2855, 1515, 1345, 1068, 759; ^1H NMR (300 MHz, CDCl_3), δ : 1.39-1.57 (m, 4H), 2.18-2.38 (m, 1H), 3.63 (dt, $J = 3.0, 11.3$ Hz, 1H), 3.92 (dd, $J = 3.7, 11.3$ Hz, 1H), 4.37 (d, $J = 3.7$ Hz, 1H), 5.09 (d, $J = 8.3$ Hz, 1H), 6.49 (d, $J = 6.8$ Hz, 1H), 6.69 (t, $J = 7.5$ Hz, 1H), 7.05 (t, $J = 8.3$ Hz, 1H), 7.24 (m, 3H), 7.39-7.45 (m, 2H); MS (ESI) m/z 300 ($[\text{M}+\text{H}]^+$).

HRMS: calcd for $\text{C}_{18}\text{H}_{18}\text{ON}$: 300.11497 Found 300.11448.

5-(4-Nitro-phenyl)-3,4,4a,5,6,10b-hexahydro-2H-pyrano[3,2-c]quinoline (4 & 5q): Cis isomer; Mp 163-165 $^{\circ}\text{C}$; R_f (20% EtOAc/n-hexane) 0.5.; IR (KBr) (ν_{max} , cm^{-1}): 3374 (N-H), 3040, 2923, 2855, 1515, 1345, 1068, 759 ; ^1H NMR (300 MHz, CDCl_3), δ : 1.17-1.59 (m, 4H), 2.16-2.24 (m, 1H), 3.41 (dt, $J = 2.9, 9.5$ Hz, 1H), 3.61 (dd, $J = 3.2, 11.9$ Hz, 1H), 3.81 (s, 1H), 4.68 (d, $J = 2.2$ Hz, 1H), 5.31 (d, $J = 5.8$ Hz, 1H), 6.61 (d, $J = 8.1$ Hz, 1H), 6.81 (t, $J = 7.3$ Hz, 1H), 7.07 (t, $J = 7.3$ Hz, 1H), 7.42 (d, $J = 8.1$ Hz, 1H), 7.64 (d, $J = 8.8$ Hz, 2H), 8.26 (d, $J = 8.8$ Hz, 2H); MS (ESI) m/z 311 ($[\text{M}+\text{H}]^+$). Anal. Calcd. for $\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}_3$: C, 69.66; H, 5.85; N, 9.03; O, 15.47. Found: C, 69.64; H, 5.84; N, 9.00.

Trans isomer : Mp 136-137 $^{\circ}\text{C}$; R_f (20% EtOAc/n-hexane) 0.45.; IR (KBr) (ν_{max} , cm^{-1}): 3377 (N-H), 3070, 2929, 2852, 1517, 1344, 1080, 750; ^1H NMR (300 MHz, CDCl_3), δ : 1.20-1.49 (m,

4H), 2.11-2.22 (m, 1H), 3.44 (dt, $J = 2.9, 9.5$ Hz, 1H), 3.77 (dd, $J = 3.2, 1.9$ Hz, 1H), 4.04 (s, 1H), 4.39 (d, $J = 2.9$ Hz, 1H), 4.85 (d, $J = 10.2$ Hz, 1H), 6.56 (d, $J = 7.3$ Hz, 1H), 6.74 (t, $J = 7.3$ Hz, 1H), 7.11 (t, $J = 7.3$ Hz, 1H), 7.23 (d, $J = 8.8$ Hz, 1H), 7.64 (d, $J = 8.8$ Hz, 2H), 8.26 (d, $J = 8.8$ Hz, 2H); MS (ESI) m/z 311 ($[M+H]^+$). Anal. Calcd. for $C_{18}H_{18}N_2O_3$: C, 69.66; H, 5.85; N, 9.03; O, 15.47. Found: C, 69.66; H, 5.84; N, 9.01.

5-(4-Bromo-phenyl)-9-isopropyl-3,4,4a,5,6,10b-hexahydro-2H-pyrano[3,2-]quinoline (4 & 5i): *Cis isomer*: Mp 101-104 °C; R_f (20% EtOAc/n-hexane) 0.5.; IR (KBr) ($\nu_{\max}$, cm^{-1}): 3368 (N-H), 2936, 2860, 1493, 1067, 818; ^1H NMR (300 MHz, CDCl_3), δ : 1.21 (s, 3H), 1.25 (s, 3H), 1.42-1.59 (m, 2H), 2.02-2.13 (m, 1H), 2.74-2.88 (m, 1H), 3.32 (dt, $J = 2.9, 10.9$ Hz, 2H), 3.52 (dd, $J = 3.6, 10.9$ Hz, 2H), 4.92 (d, $J = 2.18$ Hz, 1H), 5.22 (d, $J = 5.0$ Hz, 1H), 6.45 (d, $J = 8.0$ Hz, 1H), 6.92 (dd, $J = 2.1, 8.0$ Hz, 1H), 7.22-7.30 (m, 3H), 7.40 (d, $J = 8.7$ Hz, 2H), 7.69 (d, $J = 8.7$ Hz, 1H); MS (ESI) m/z 386 ($[M+H]^+$). Anal. Calcd. for $C_{21}H_{24}\text{BrNO}$: C, 65.29; H, 6.26; Br, 20.68; N, 3.63; O, 4.14. Found: C, 65.26; H, 6.26; N, 3.61.

Trans isomer: Mp 94-95 °C; R_f (20% EtOAc/n-hexane) 0.45.; IR (KBr) ($\nu_{\max}$, cm^{-1}): 3368 (N-H), 2936, 2860, 1493, 1067, 818; ^1H NMR (300 MHz, CDCl_3), δ : 1.94 (s, 3H), 1.23 (s, 3H), 1.39-1.54 (m, 2H), 2.02-2.13 (m, 1H), 2.72-2.82 (m, 1H), 3.62 (dt, $J = 2.1, 11.65$ Hz, 2H), 4.04 (dd, $J = 2.1, 11.6$ Hz, 2H), 4.30 (d, $J = 2.1$ Hz, 1H), 4.60 (d, $J = 10.9$ Hz, 1H), 6.45 (d, $J = 8.0$ Hz, 1H), 6.95 (dd, $J = 2.1, 8.0$ Hz, 1H), 7.01-7.02 (d, $J = 2.18, 2\text{H}$), 7.28 (d, $J = 8.01$ Hz, 2H), 7.48 (d, $J = 8.7$ Hz, 1H); MS (ESI) m/z 386 ($[M+H]^+$). Anal. Calcd. for $C_{21}H_{24}\text{BrNO}$: C, 65.29; H, 6.26; Br, 20.68; N, 3.63; O, 4.14. Found: C, 65.28; H, 6.27; N, 3.65.

9-Fluoro-5-phenyl-3,4,4a,5,6,10b-hexahydro-2H-pyrano[3,2-c]quinoline (4 & 5l): *Cis isomer*

: Mp 158-159 °C; *R_f* (20% EtOAc/n-hexane) 0.5.; ¹H NMR (300 MHz, CDCl₃), δ: 1.37-1.55 (m, 4H), 2.14 (m, 1H), 3.42 (dt, *J* = 3.02, 11.3Hz, 1H), 3.60 (dd, *J* = 3.02, 11.3 Hz, 1H), 3.70 (br s, 1H), 4.63 (d, *J* = 3.0 Hz, 1H), 5.23 (d, *J* = 6.0 Hz 1H), 6.45-6.49 (d, *J* = 4.5 Hz, 1H), 6.78 (dt, *J* = 3.0, 8.3 Hz, 1H), 7.08-7.12 (dd, *J* = 3.0, 8.3 Hz, 1H), 7.24-7.39 (m, 5H); MS (ESI) *m/z* 284 ([M+H])⁺. Anal. Calcd. for C₁₈H₁₈FNO: C, 76.30; H, 6.40; F, 6.71; N, 4.94; O, 5.65. Found: C, 76.29; H, 6.40; N, 4.91.

Trans isomer : Mp 82-84 °C; *R_f* (20% EtOAc/n-hexane) 0.48.; ¹H NMR (300 MHz, CDCl₃), δ: 1.31-1.50 (m, 2H), 1.57-1.68 (m, 1H), 1.75-1.87 (m, 1H); 2.07 (m, 1H), 3.71 (dt, *J* = 2.2, 11.3 Hz, 1H), 3.91 (br s, 1H), 4.08 (dd, *J* = 3.7, 10.5 Hz, 1H), 4.31 (d, *J* = 2.2 Hz, 1H), 4.64 (d, *J* = 10.5 Hz, 1H), 6.39-6.44 (2d, *J* = 4.5 Hz, 1H), 6.81(dt, *J* = 3.0, 8.3 Hz, 1H), 6.88-6.92 (dd, *J* = 3.0, 9.0 Hz, 1H), 7.24-7.39 (m, 5H); MS (ESI) *m/z* 284 ([M+H])⁺. Anal. Calcd. for C₁₈H₁₈FNO: C, 76.30; H, 6.40; F, 6.71; N, 4.94; O, 5.65. Found: C, 76.29; H, 6.41; N, 4.95.

2-Fluoro-6-(4-fluoro-phenyl)-5,6a,7,8,9,10a-hexahydro-6H-phenanthridin-10-one (4 & 5n):

Cis isomer : Mp 118-120 °C; *R_f* (20% EtOAc/n-hexane) 0.5.; ¹H NMR (300 MHz, CDCl₃), δ: 1.56-1.94 (m, 3H), 2.26-2.36 (m, 2H), 2.58-2.74 (m, 2H), 4.36-4.40 (m, 1H), 4.64 (d, *J* = 3.1 Hz, 1H), 6.40-6.46 (m, 2H), 6.72-6.81 (t, *J* = 7.8 Hz, 1H), 7.32-7.34 (m, 4H); MS (ESI) *m/z* 314 ([M+H])⁺. Anal. Calcd. for C₁₉H₁₇F₂NO: C, 72.83; H, 5.47; F, 12.13; N, 4.47; O, 5.11. Found: C, 72.80; H, 5.46; N, 4.45.

Trans isomer : Mp 114-115 °C; *R_f* (20% EtOAc/n-hexane) 0.45.; ¹H NMR (300 MHz, CDCl₃), δ: 1.68-1.79 (m, 2H); 1.99-2.30 (m, 2H), 2.38-2.45 (m, 2H), 2.69-2.77 (m, 2H), 4.36-4.42 (m, 1H), 4.54 (d, *J* = 2.0 Hz, 1H), 6.50-6.55 (m, 2H), 6.79-6.81 (t, *J* = 8.3 Hz, 1H), 7.21-

7.32 (m, 4H); MS (ESI) m/z 314 ($[M+H]^+$). Anal. Calcd. for $C_{19}H_{17}F_2NO$: C, 72.83; H, 5.47; F, 12.13; N, 4.47; O, 5.11. Found: C, 72.85; H, 5.47; N, 4.45.

12-(2-Chloro-phenyl)-2,3,4a,11,12,12a-hexahydro-1H-4-oxa-11-aza-chrysene (Table 2, entry r): **Cis isomer** : m.p. 152-154 °C; R_f (20% EtOAc/n-hexane) 0.5.; 1H NMR (300 MHz, $CDCl_3$), δ : 1.21-1.59 (m, 3H), 2.48 (m, 1H), 3.29-3.36 (m, 1H), 3.52-3.61 (m, 1H), 4.29-4.30 (m, 1H), 5.18 (d, $J = 2.6$ Hz, 1H), 5.45 (d, $J = 6.4$ Hz, 1H), 7.17-7.35 (m, 2H), 7.37-7.45 (m, 5H), 7.48-7.54 (m, 2H), 7.93 (d, $J = 2.07$ Hz, 1H); MS (ESI) m/z 350 ($[M+H]^+$). Anal. Calcd. for $C_{22}H_{20}ClNO$: C, 75.53; H, 5.76; Cl, 10.13; N, 4.00; O, 4.57. Found: C, 75.53; H, 5.74; N, 4.01.

Trans isomer : Thick syrup; R_f (20% EtOAc/n-hexane) 0.48.; 1H NMR (300 MHz, $CDCl_3$), δ : 1.20-1.65 (m, 3H), 2.29 (m, 1H), 3.36-3.55 (m, 1H), 3.63-3.64 (m, 1H), 4.19-4.60 (m, 1H), 4.61 (d, $J = 3.6$ Hz, 1H), 5.31 (d, $J = 10.9$ Hz, 1H), 6.67-7.46 (m, 6H), 7.62-7.78 (m, 4H); MS(ESI) m/z 350 ($[M+H]^+$). Anal. Calcd. for $C_{22}H_{20}ClNO$: C, 75.53; H, 5.76; Cl, 10.13; N, 4.00; O, 4.57. Found: C, 75.55; H, 5.75; N, 4.04.

FBSLAD #12-30 RT: 0.33-0.86 AV: 19 SB: 9 0.01-0.24 SM: 15G NL: 1.86E4
T: + p ESI Full ms [50.00-2000.00]

Indian Institute of Chemical Technology, Hyderabad
FTIR Analysis Report

Sample Name:
Sample Preparation: KBr
Collection time: Mon Dec 18 15:03:05 GMT+05:30
Bench: Thermo Nicolet Nexus 670 Spectrometer
Resolution: 4 cm⁻¹

Detector: DTGS KBr
Beamsplitter: KBr
Source: IR

Indian Institute of Chemical Technology, Hyderabad
FTIR Analysis Report

IR SPECTRUM OF *trans* 5-Phenyl-3,4,4a,5,6,10b-hexahydro-2H-pyrano[3,2-c]quinoline (5a)

File Name:
Sample Preparation: NEAT
Acquisition time: Mon Dec 18 15:23:29 (GMT+05:30)
Instrument: Thermo Nicolet Nexus 670 Spectrometer
Resolution: 4 cm⁻¹

Detector: DTGS KBr
Beamsplitter: KBr
Source: IR
Analyst Name:

National Centre for Mass Spectrometry
CSIR-Indian Institute of Chemical Technology
File Name: C:\VICT-HRMS\14.03.2014\NL-4FBENZ
Sample Name: NL-4FBENZ
Sample ID: V H REDDY
Date and Time: 14-03-14 13:06:50
NL-4FBENZ#3-85 RT: 0.02-0.30 AV: 83 SB: 326 0.80-1.90 NL: 2.54E7
T: FTMS (1,1) + p ESI Full ms [100.00-2000.00]

NL-4FBENZ#8-30 RT: 0.04-0.11 AV: 23
T: FTMS (1,1) + p ESI Full ms [100.00-2000.00]
m/z= 283.35-284.55

m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
284.14365	44110032.0	100.00	284.14452	-3.04	9.5	C ₁₈ H ₁₉ ONF

HRMS SPECTRUM of *cis*-5-(4-Fluorophenyl)-3,4,4a,5,6,10b-hexahydro-2H-pyrano[3,2-c]quinoline (4g)

National Centre for Mass Spectrometry
CSIR-Indian Institute of Chemical Technology

File Name C:\ICT-HRMS\14.03.2014\NL-4CL-BENZ
Sample Name V H REDDY
Sample ID V H REDDY
Date and Time 14-03-14 13:06:09
NL-4CL-BENZ #2-85 RT: 0.02-0.30 AV: 84 SB: 327 0.80-1.90 NL: 1.04E7
T: FTMS (1,1) + p ESI Full ms [100.00-2000.00]

NL-4CL-BENZ#8-30 RT: 0.04-0.12 AV: 23
T: FTMS (1,1) + p ESI Full ms [100.00-2000.00]
m/z= 299.92-300.29

m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
300.11387	11623931.0	100.00	300.11497	-3.65	9.5	C ₁₈ H ₁₉ ONCl

HRMS SPECTRUM of cis-5-(4-Chlorophenyl)-3,4,4a,5,6,10b-hexahydro-2H-pyrano[3,2-c]quinoline (4d)

National Centre for Mass Spectrometry
CSIR-Indian Institute of Chemical Technology

File Name C:\ICT-HRMS\14.03.2014\NL-4-FBAB
Sample Name V H REDDY
Sample ID V H REDDY
Date and Time 14-03-14 13:14:12
NL-4-FBAB #2-83 RT: 0.02-0.30 AV: 82 SB: 326 0.80-1.90 NL: 1.14E8
T: FTMS (1,1) + p ESI Full ms [100.00-2000.00]

NL-4-FBAB#8-30 RT: 0.05-0.12 AV: 23
T: FTMS (1,1) + p ESI Full ms [100.00-2000.00]
m/z= 284.14391

m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
284.14391	157627152.0	100.00	284.14452	-2.13	9.5	C ₁₈ H ₁₉ ONF

HRMS SPECTRUM of trans-5-(4-Fluorophenyl)-3,4,4a,5,6,10b-hexahydro-2H-pyrano[3,2-c]quinoline (5g)

File Name C:\ICT-HRMS\14.03.2014\NL-4-CBA

Sample Name

Sample ID V H REDDY

Date and Time 14-03-14 13:11:31

NL-4-CBA#2-84 RT: 0.02-0.30 AV: 83 SB: 326 0.80-1.90 NL: 3.52E7
T: FTMS (1,1) + p ESI Full ms [100.00-2000.00]

NL-4-CBA#8-30 RT: 0.04-0.12 AV: 23

T: FTMS (1,1) + p ESI Full ms [100.00-2000.00]

m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
300.11448	59652108.0	100.00	300.11497	-1.62	9.5	C ₁₈ H ₁₉ ONCl

HRMS SPECTRUM of trans-5-(4-Chlorophenyl)-3,4,4a,5,6,10b-hexahydro-2H-pyrano[3,2-c] quinoline (5d)