Synthesis of 2-hydroxy-3-alkyl-2-phenyl-2, 3-dihydroquinazolin-4(1H)-one via molybdenum hexacarbonyl mediated CO gas-and ligand free carbonylative reactions.


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Supporting Information P. No

Section A: General Information .................................................................2

Section B: Experimental Procedure, Table & data........................................3

Section C: 1H, 13C, HRMS and IR spectrum............................................... 12

Electronic Supplementary Material (ESI) for RSC Advances.
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**General methods:** Unless stated otherwise, reactions were performed under nitrogen atmosphere using oven dried glassware. Reactions were monitored by thin layer chromatography (TLC) on silica gel plates (60 F254), visualizing with ultraviolet light or iodine spray. Flash chromatography was performed on silica gel (100-200 mesh) using distilled hexane, ethyl acetate, dichloromethane. $^1$H NMR and $^{13}$C NMR spectra were determined in CDCl$_3$ or DMSO-$d_6$ solution by using 400 or 100 MHz spectrometers, respectively. Proton chemical shifts (δ) are relative to tetramethylsilane (TMS, δ = 0.00) as internal standard and expressed in ppm. Spin multiplicities are given as s (singlet), d (doublet), t (triplet) and m (multiplet) as well as b (broad). Coupling constants (J) are given in hertz. Melting points were determined using melting point B-540 apparatus and are uncorrected. HRMS was determined using waters LCT premier XETOF ARE-047 apparatus.
**General process for the synthesis of substituted 2-hydroxy-2, 3-dihydroquinazolin-4-(1H)-one:**

![Chemical structure](image)

**General procedure:** A mixture of Isatoic anhydride (500 mg, 3.0 mmol), aryl halides (868 mg, 3.0 mmol), amine (344 mg, 3.4 mmol), Mo (CO)$_6$ (820 mg, 3.0 mmol), Et$_4$NBr (200 mg, 0.6 mmol), and Bu$_3$N (689 mg, 3.7 mmol), in DMF (5 mL) was heated to 150°C under argon balloon. Maintained the reaction mass for 15 to 18 hours and check the progress of reaction as indicated by TLC. The reaction mixture was cooled to room temperature and added water (20 mL) and ethyl acetate (20 mL), total reaction mixture was filtered through celite bed and bed washed with Ethyl acetate (3*20 mL) and both layers were separated and aqueous layer was back extracted with ethyl acetate (20 mL) and then combined organic layers was washed with water (20 mL) and dry over anhydrous Na$_2$SO$_4$ and the total organic layer was concentrated under reduced pressure to afford the crude hydroxy compounds. The above crude was purified by (100-200 mesh silica gel) gravity column purification and product was eluted at 8 to 10 % of EtOAc/Hexanes to give pure hydroxy compounds of 10a to 15o.
2-[(1, 1’-biphenyl)-4-yl]-3-hexyl-2-hydroxy-2, 3-dihydroquinazolin-4(1H)-one (12a):
White solid, Yield-72 %, mp: 97-105°C, IR (KBr) = 3325, 2922, 1672, 1548, 1310, 1076, 984, 858, 797, 745, 692, 450 cm\(^{-1}\), \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta=12.16\) (s, 1H), 8.84 (d, \(J = 8.4\) Hz, 1H), 8.12 (d, \(J = 8.4\) Hz, 2H), 7.74 (d, \(J = 8.4\) Hz, 2H), 7.65 (d, \(J = 7.6\) Hz, 2H), 7.55 – 7.26 (m, 5H), 7.13 (t, \(J = 7.6\) Hz, 1H), 6.28 (s, br, 1H), 3.50 – 3.43 (m, 2H), 1.68 – 1.56 (m, 2H), 1.40 – 1.25 (m, 6H), 0.91 – 0.89 (m, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \(\delta =169.2, 144.54, 140.10, 139.83, 133.55, 132.51, 128.91-(2C), 127.97-(2C), 127.92-(2C), 127.42-(2C), 127.23-(2C), 126.4, 122.8, 121.6, 120.8, 40.1, 31.4, 29.4, 26.6, 22.5, 13.9; HRMS: m/z calcd for C\(_{26}\)H\(_{29}\)N\(_2\)O\(_2\) (M+H) 401.2249, found 401.2241.

2-[(1, 1’-biphenyl)-4-yl]-3-benzyl-2-hydroxy-2, 3-dihydroquinazolin-4(1H)-one (12b):
White solid, Yield- 67 %, mp:154-158°C , IR (KBr) = 3325, 2922, 1672, 1548, 1310, 1076, 984, 745, 692, 450 cm\(^{-1}\), \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta =12.20\) (s, 1H), 8.86 (d, \(J = 8.4\) Hz, 1H), 8.13 (d, \(J = 8.4\) Hz, 2H), 7.75 (d, \(J = 8.0\) Hz, 2H), 7.56 -7.32 (m, 12H), 7.12 (t, \(J = 7.6\) Hz, 1H), 6.56 (s, 1H), 4.68 (d, \(J = 5.6\) Hz, 2H), \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \(\delta =167.0, 144.3, 139.9, 138.1, 132.9, 132.8, 128.8, 128.7, 128.6, 127.97-(2C), 127.93-(2C), 127.90-(2C), 127.83, 127.81, 127.77, 127.60, 127.48, 127.40, 127.21, 127.17, 126.5, 122.8, 121.6, 44.0; HRMS: m/z calcd for C\(_{27}\)H\(_{23}\)N\(_2\)O\(_2\) (M+H) 406.1760, found 406.1757.
2-((1, 1'-biphenyl)-4-yl)-2-hydroxy-3-(4-methoxybenzyl)-2, 3-dihydroquinazolin-4(1H)-one (12c): Brown solid, Yield-58 %, mp : 168-172°C, IR (KBr) = 3328, 2926, 1638, 1512, 1428, 1249, 1027, 809, 746, 580, 520 cm\(^{-1}\), \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta= 12.22\) (s, 1H), 8.86 (d, \(J= 8.4\) Hz, 1H), 8.14 (d, \(J= 8.4\) Hz, 1H), 7.86 (d, \(J= 8.4\) Hz, 2H), 7.75 (d, \(J= 8.4\) Hz, 1H), 7.66 (d, \(J= 6.4\) Hz, 2H), 7.56-7.49 (m, 5H), 7.44-7.38 (m, 1H), 7.38-7.26 (m, 2H), 6.91-6.85 (m, 2H), 6.36 (s, 1H), 4.61 (d, \(J= 4.8\) Hz, 2H), 3.80 (s, 3H), \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \(\delta = 169.00, 166.30, 159.10, 144.27, 140.05, 133.51, 133.05, 132.76, 130.25, 129.27, 129.24, 128.88, 128.87, 127.95, 127.92, 127.45, 127.39, 127.20, 127.17, 127.14, 126.56, 122.78, 121.57, 120.26, 114.24, 114.14, 55.28, 43.6; HRMS: m/z calcd for C\(_{28}\)H\(_{25}\)N\(_2\)O\(_3\) (M+H) 437.1865, found 437.1865.

2-((1, 1'-biphenyl)-4-yl)-3-cycloheptyl-2-hydroxy-2, 3-dihydroquinazolin-4 (1H)-one (12d): White solid, Yield- 70 %, mp : 139-142°C , IR (KBr) = 3337, 2923, 1628, 1512, 1446, 1278, 1006, 901, 743, 662, 546 cm\(^{-1}\), \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta = 12.09\) (s, 1H), 8.82 (d, \(J= 8.4\) Hz, 1H), 8.12 (d, \(J= 8.4\) Hz, 2H), 7.74 (d, \(J= 8.4\) Hz, 2H), 7.59-7.41 (m, 7H), 7.10 (t, \(J= 7.2\) Hz, 1H), 6.23 (s, br, 1H), 4.19-4.17 (m, 1H), 2.08-2.02 (m, 2H), 1.67-1.56 (m, 10H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \(\delta = 168.02, 144.44, 143.97, 140.04, 140.01, 139.81, 133.70, 132.43, 128.86-(2C), 128.85-(2C), 127.92-(2C), 127.89-(2C), 127.30-(2C), 127.12, 50.95, 50.88, 35.16, 35.00, 27.99, 27.96, 24.11, 24.00; HRMS: m/z calcd for C\(_{27}\)H\(_{29}\)N\(_2\)O\(_2\) (M+H) 413.2229, found 413.2228.
2-[(1, 1'-biphenyl]-4-yl]-2-hydroxy-3-(R)-1-phenylethyl]-2, 3-dihydroquinazolin-4 (1H)-one, (12e): Colorless solid, Yield-62 %, mp = 205-211°C, IR (KBr) = 3334, 2969, 1670, 1591, 1259, 1160, 883, 753, 558 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ= 12.10 (s, 1H), 8.84 (d, J= 8.4 Hz, 1H), 8.09 (d, J= 8.0 Hz, 2H), 7.73 (d, J= 8.4 Hz, 2H), 7.65 (d, J= 7.2 Hz, 2H), 7.56-7.48 (m, 4H), 7.46-7.38 (m, 5H), 7.36-7.30 (m, 1H), 7.13 (t, J= 7.6 Hz, 1H), 6.48 (d, J= 7.2 Hz, 1H), 5.37-5.30 (m, 1H), 1.65 (d, J= 6.8 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ= 166.24, 144.31, 143.11, 140.01, 133.22, 128.89-(2C), 128.76-(2C), 127.97-(3C), 127.48-(3C), 127.43-(3C), 127.22-(3C), 127.19-(3C), 126.26-(2H), 49.24, 21.72, HRMS: m/z calcd for C₂₈H₂₅N₂O₂ (M+H) 421.1896 found 421.1896.

3-hexyl-2-hydroxy-2-phenyl-2, 3-dihydroquinazilin-4 (1H)-one (12f): Syrupy liquid, Yield- 68 %, IR (KBr) = 3345, 2928, 1642, 1525, 1449, 1302, 1159, 1029, 758, 26 cm⁻¹, ¹H NMR (400 MHz, CDCl₃): δ= 12.09 (s, 1H), 8.82 (d, J= 8.4 Hz, 1H), 8.05 (d, J= 7.6 Hz, 2H), 7.54-7.42 (m, 5H), 7.13 (t, J= 7.6 Hz, 1H), 6.2 (s, 1H), 3.48-3.43 (m, 2H), 1.66-1.59 (m, 2H), 1.40-1.31 (m, 6H), 0.93-0.89 (m, 3H), ¹³CNMR (100 MHz, CDCl₃): δ = 169.18, 139.77, 134.82, 132.48, 131.77, 131.27, 128.73, 128.52, 127.37, 126.78, 126.45, 122.80, 121.60, 120.86, 40.11, 31.44, 29.40, 26.64, 22.53, 13.97, HRMS: m/z calcd for C₂₀H₂₅N₂O₂ (M+H) 325.1916, found 325.1911.

3-benzyl-2-hydroxy-2-phenyl-2, 3-dihydroquinazolin-4 (1H)-one: (12g): Syrupy liquid, Yield- 68 %, IR (KBr) = 3294, 2925, 1680, 1525, 1157, 1044, 796, 755, 580 cm⁻¹, ¹H NMR
(400 MHz, CDCl₃): δ = 12.10 (s, 1H), 8.83 (d, J= 8.4 Hz, 1H), 8.05 (d, J= 6.8 Hz, 2H), 7.57-7.50 (m, 5H), 7.39-7.31 (m, 5H), 7.10 (t, J= 7.6Hz, 1H), 6.60 (s, br, 1H), 4.66 (d, J= 5.6 Hz, 2H). ¹³C NMR (100 MHz, CDCl₃): δ = 169.06, 139.88, 137.43, 134.73, 132.75, 131.82, 131.53, 128.85, 128.74, 128.65, 128.56, 127.82, 127.78, 127.71, 127.59, 127.35, 126.90, 126.58, 122.82, 121.56, 44.02, HRMS: m/z calcd for C₂₁H₁₉N₂O₂ (M+H) 331.1447 found 331.1440.

3-cycloheptyl-2-hydroxy-2-phenyl-2, 3-dihydroquinazolin-4 (1H)-one (12h): Brown solid, Yield-65 %, mp: 89-94°C, IR (KBr) = 3327, 2856, 1630, 1491, 1073, 887, 752, 671 cm⁻¹, ¹H NMR (400 MHz, CDCl₃): δ = 12.10 (s, 1H), 8.80 (d, J= 8.0 Hz, 1H), 8.05 (d, J= 7.2 Hz, 2H), 7.46-7.40 (m, 5H), 7.12 (t, J= 7.6 Hz, 1H), 6.20 (s, 1H), 4.16 (s, 1H), 2.04-2.03 (m, 2H), 1.66-1.54 (m, 10H); ¹³C NMR (100 MHz, CDCl₃): δ = 168.50, 132.52, 131.76, 131.20, 128.75, 128.50-2C), 127.41-2C), 126.78-2C), 126.33, 122.79, 50.93, 50.86, 35.20, 35.08, 28.05, 28.01, 24.15, 24.04, HRMS: m/z calcd for C₂¹H₂₅N₂O₂ (M+H) 337.1916 found 337.1890.

2-hydroxy-2-phenyl-3-((R)-1-phenylethyl)-2, 3-dihydroquinazolin-4 (1H)-one (12i): White solid, Yield-60 %, mp: 101-106°C, IR (KBr) = 3305, 2927, 1634, 1446, 1209, 1028, 928, 754, 700, 551 cm⁻¹, ¹H NMR (400 MHz, CDCl₃): δ = 12.04 (s, 1H), 8.81 (d, J= 8.8 Hz, 1H), 8.01 (d, J= 7.6 Hz, 1H), 7.78 (d, J= 7.6 Hz, 2H), 7.54-7.47 (m, 2H), 7.43-7.36 (m, 6H), 7.31-7.25 (m, 2H), 6.34 (s, br, 1H), 5.37-5.30 (m, 1H), 1.61 (d, J= 6.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ = 166.53, 143.09, 134.59, 132.75, 131.76, 131.44, 128.85, 128.74-2C), 128.54-2C), 127.66, 127.46, 127.40, 126.88, 126.43, 126.23, 126.16, 122.80, 121.67, 49.20, 21.69, HRMS: m/z calcd for C₂₂H₂₁N₂O₂ (M+H) 345.1503 found 345.1595.
3-hexyl-2-hydroxy-2-(4-methoxyphenyl)-2, 3- dihydroquinazolin-4 (1H)-one, (12j):
Brown solid, Yield-48 %, mp:68-72°C, IR (KBr) = 3337, 2923, 1682, 1512, 1446, 1278, 1006, 901, 662, 546 cm⁻¹, ¹H NMR (400 MHz, CDCl₃): δ = 12.10 (s, 1H), 8.79 (d, J= 8.4 Hz, 1H), 8.01 (d, J= 8.4 Hz, 2H), 7.52-7.47 (m, 2H), 7.05-6.91 (m, 2H), 6.69-6.63 (m, s, 1H), 6.32 (s, 1H), 3.87 (s, 3H), 3.48-3.40 (m, 2H), 1.66-1.56 (m, 2H), 1.41-1.27 (m, 6H), 0.91-0.88 (m, 3H), ¹³C NMR (100 MHz, CDCl₃): δ= 169.18, 165.59, 139.77, 134.82, 132.48, 131.77, 131.27, 128.73, 127.37, 126.8, 126.45, 122.80, 121.60, 120.86, 55.42, 40.11, 31.44, 29.40, 26.64, 22.53, 13.97; HRMS: m/z calcd for C₂₁H₂₇N₂O₃ (M+H) 355.2022 found 355.2007.

3-benzyl-2-hydroxy-2-(4-methoxyphenyl)-2, 3-dihydroquinazolin-4- (1H)-one, (12k):
White solid, Yield-48 %, mp: 115-119°C, IR (KBr) =3327, 2856, 1630, 1556, 1073, 887, 752, 671, 585 cm⁻¹, ¹H NMR (400 MHz, CDCl₃): δ= 12.04 (s, 1H), 8.81 (d, J= 8.4 Hz, 1H), 8.03 (d, J= 8.8 Hz, 2H), 7.56-7.49 (m, 3H), 7.39-7.29 (m, 5H), 7.01 (d, J= 8.4 Hz, 2H), 6.32 (s, 1H), 4.61 (d, J= 5.2 Hz, 2H), 3.87 (s, 3H), ¹³C NMR (100 MHz, CDCl₃): δ= 169.14, 162.51, 140.30, 137.45, 135.03, 132.86, 132.40, 129.32, 128.77, 128.42, 127.85, 127.79, 127.55, 127.05, 126.42, 122.57, 121.61, 120.10, 117.35, 116.59,55.42, 44.11;HRMS: m/z calcd for C₂₂H₂₁N₂O₂ (M+H) 361.1552 found 361.1537.

3-cycloheptyl-2-hydroxy-2-(4-methoxyphenyl)-2, 3-dihydroquinazolin-4(1H)-one, (12l):
Syrupy liquid, Yield-52 %, IR (KBr) = 3314, 2856, 1678, 1591, 1412, 1173, 1022, 834, 755, 666, 573, 528 cm⁻¹, ¹H NMR (400 MHz, CDCl₃): δ = 11.99 (s, 1H), 8.78 (d, J= 8.4 Hz, 1H), 8.02 (d, J= 8.8 Hz, 2H), 7.54-7.45 (m, 2H), 7.10 (t, J= 8.0 Hz, 1H), 7.01 (d, J= 8.8 Hz, 2H),
6.18 (s, br, 1H), 4.17-4.15 (m, 1H), 3.87 (s, 3H), 2.07-2.01 (m, 2H), 1.68-1.53 (m, 10 H), $^{13}$C NMR (100 MHz, CDCl$_3$): δ = 168.07, 162.42, 140.00, 132.45, 129.28-(2C), 127.15-(2C), 126.36, 122.52-(2C), 121.52, 120.90, 113.92, 55.41, 50.90, 35.03-(2C), 27.98-(2C), 23.99-(2C), HRMS: m/z calcd for C$_{22}$H$_{27}$N$_2$O$_3$ (M+H) 367.2022 found 367.2030.

2-(4-fluorophenyl)-3-hexyl-2-hydroxy-2, 3-dihydroquinazolin-4 (1H)-one, (12m): White solid, Yield-26 %, mp:170-176°C, IR (KBr) = 3327, 2856, 1681, 1525, 1491, 1073, 887, 671, 585, 527 cm$^{-1}$, $^1$H NMR (400 MHz, CDCl$_3$): δ = 12.10 (s, 1H), 8.82 (d, J= 8.8 Hz, 1H), 8.07-8.06 (m, 2H), 7.55-7.49 (m, 2H), 7.20-7.08 (m, 3H), 6.21 (s, br, 1H), 3.49-3.42 (m, 2H), 1.67-1.63 (m, 2H), 1.41-1.33 (m, 6H), 0.91-0.88 (m, 3H), $^{13}$C NMR (100 MHz, CDCl$_3$): δ= 169.7, 165.8 (C-F), 129.83, 129.75 (2C), 126.33, 122.90 (2C), 121.55 (2C), 115.88 (2C), 115.66 (2C), 40.12, 31.42, 29.38, 26.63, 22.53, 13.99, HRMS: m/z calcd for C$_{20}$H$_{24}$FN$_2$O$_2$ (M+H) 343.1803 found 343.1822.

3-hexyl-2-hydroxy-2-(thiophen-2-yl)-2, 3-dihydroquinazolin-4-(1H)-one, (12n): Syrupy liquid, Yield-80 %, IR (KBr) = 3334, 2868, 1670, 1591, 1259, 1160, 883, 753, 558 cm$^{-1}$; $^1$H NMR (400MHz, CDCl$_3$): δ = 12.16 (s, 1H), 8.73 (d, J= 8.4 Hz, 1H), 7.77 (d, J= 3.2 Hz, 1H), 7.54-7.47 (m, 3H), 7.14-7.07 (m, 2H), 6.28 (s, br, 1H), 3.49-3.41 (m, 2H), 1.70-1.60 (m, 2H), 1.42-1.32 (m, 6H), 0.94-0.86 (m, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ= 169.14, 160.32, 139.73, 132.63 (2C), 130.97, 128.53, 127.91, 126.38, 122.72, 121.37, 120.23, 40.13, 31.45, 29.39, 26.65, 22.53, 13.98; HRMS: m/z calcd for C$_{18}$H$_{23}$SN$_2$O$_2$ (M+H) 331.1469 found 331.1480.
2-hydroxy-3-((R)-1-Phenylethyl)-2-(thiophen-2-yl)-2, 3-dihydroquinazolin-4 (1H)-one, (18): Brown solid, Yield-77 %, mp: 143-149°C, IR (KBr) = 3334, 2969, 1626, 1591, 1259, 1160, 1090, 883, 753 cm⁻¹, ¹H NMR (400 MHz, CDCl₃): δ = 12.1 (s, 1H), 8.72 (d, J = 8.0 Hz, 1H), 7.73 (d, J= 4 Hz, 1H), 7.41-7.25 (m, 8H), 7.13-7.05 (m, 2H), 6.52 (s, br, 1H), 5.38-5.29 (m, 1H), 1.68-1.64 (d, J= 16.8 Hz, 3H), ¹³C NMR (100 MHz, CDCl₃): δ= 168.32, 142.47, 140.43, 139.91, 132.86, 131.01, 129.88, 128.88, 128.75, 128.55, 128.00, 127.90, 127.71, 127.55, 126.42, 126.19, 122.71, 121.42, 49.33, 21.60, HRMS: m/z calcd for C₂₀H₁₉SN₂O₂ (M+H) 351.1167 found 351.1176.

3-Benzyl-2-hydroxy-2-(3-methoxyphenyl)-2, 3-dihydro quinazolin-4(1H) one (12p): White solid, Yield-49 %, ¹H NMR (400 MHz, CDCl₃): δ = 12.13 (s, 1H), 8.78 (d, J= 8.4 Hz, 1H), 7.59-7.58 (m, 2H), 7.50-7.49 (m, 2H), 7.41-7.25 (m, 6H), 7.07-7.05 (m, 2H), 6.75 (s, br, 1H), 4.65-4.62 (m, 2H), 3.87 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ= 169.2, 167.3, 165.4, 159.9, 159.7, 139.7, 136.3, 132.4, 129.7, 129.4, 126.4, 122.8, 121.5, 120.8, 119.1, 118.5, 118.3, 117.4, 112.4, 112.2, 53.4, 41.1; HRMS: m/z calcd for C₂₂H₂₁N₂O₃ (M+H) 361.1552 found 361.1533.
3-Hexyl-2-hydroxy-2-(3-methoxy phenyl)-2, 3-dihydro quinazolin-4(1H) one (12q): white solid, Yield-52 %, ¹H NMR (400 MHz, CDCl₃): δ = 12.10 (s, 1H), 8.78 (d, J= 8.4 Hz, 1H), 7.49-7.47 (m, 2H), 7.42-7.38 (m, 2H), 7.34-7.28 (m, 1H), 7.08-7.05 (m, 2H), 6.51 (s, br, 1H), 3.87 (s, 3H), 3.44-3.41 (m, 2H), 1.63-1.60 (m, 2H), 1.38-1.24 (m, 6H), 0.89-0.86, (m, 3H); ¹³C NMR (100 MHz, CDCl₃): δ= 169.9, 165.4, 139.7, 136.3, 132.4, 129.7, 129.4, 126.4, 122.8, 120.8, 120.0, 119.1, 118.5, 112.4, 112.2, 53.4, 40.1, 31.4, 29.4, 26.6, 22.5, 13.9; MS (EI) m/z:186.26, 355.20(M+H)+, 731.30.

3-Benzyl-2-hydroxy-2-(2-methoxy phenyl)-2,3-dihydro quinazolin-4(1H) one (12r): Light green color solid, Yield-35 %, ¹H NMR (400 MHz, CDCl₃): δ = 11.72 (s, 1H), 8.69 (d, J= 8.4 Hz, 1H), 7.50-7.44 (m, 3H), 7.35-7.25 (m, 7H), 7.18 (d, J= 1.2 Hz, 1H), 7.0 (d, J= 16Hz, 1H), 6.49 (s, br, 1H), 4.64-4.59 (m, 2H), 4.01 (s, 3H);¹³C NMR (100 MHz, CDCl₃): δ= 169.1, 167.3, 165.4, 159.9, 159.7, 139.7, 136.3, 132.4, 129.7, 129.4, 126.4, 122.8, 121.5, 120.8, 119.1, 118.5, 118.3, 117.4, 112.4, 112.2, 55.4, 40.1; HRMS: m/z calcd for C₂₂H₂₁N₂O₃ (M+H) 361.1552 found 361.1537.

3-Hexyl-2-phenyl quinazolin-4-(3H)-one (13a): Syrupy liquid,¹H NMR (400MHz, CDCl₃): δ=8.34 (d, J = 7.6 Hz, 1H), 7.78-7.72 (m, 2H), 7.54-7.49 (m, 5H), 3.99-3.95 (m, 2H), 1.63-1.56 (m, 2H), 1.20-1.07 (m, 6H), 0.84-0.78 (m, 3H);¹³C NMR (100 MHz, CDCl₃): δ =162.16, 156.29, 147.15, 135.51, 134.29, 133.61, 130.15, 129.79, 128.74, 128.45, 127.79, 127.39, 126.97, 126.77, 45.94, 30.97, 28.55, 26.28, 22.30, 13.87;HRMS: m/z calcd for C₂₀H₂₃N₂O (M+H) 307.1810, found 307.1805.
3-Hexyl-2-(4-methoxyphenyl) quinazolin-4 (3H)-one (22): Syrupy liquid, $^1$H NMR (400 MHz, CDCl$_3$): 8.32 (d, J = 7.2 Hz, 1H), 7.74 – 7.72 (m, 1H), 7.50-7.49 (m, 2H), 7.31-7.26 (m, 2H), 7.04-7.01 (m, 1H), 6.87-6.83 (m, 1H), 4.02-3.98 (m, 2H), 3.88 (s, 3H), 1.62-1.54 (m, 2H), 1.25-1.14 (m, 6H), 0.86-0.79 (m, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta = 162.33$, 160.64, 156.18, 147.25, 134.19, 133.22, 134.19, 133.22, 128.04, 127.36, 126.76, 126.72, 120.83, 119.18, 114.16, 114.10, 55.43, 45.97, 31.04, 27.51, 26.31, 22.33, 13.89; HRMS: m/z calcd for C$_{21}$H$_{25}$N$_2$O$_2$ (M+H) 337.1916, found 337.1916.
**Single Mass Analysis**

Tolerance: 5.0 PPM  /  DBE: min = -1.5, max = 100.0
Element prediction: C0.1
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
42 formula(s) evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 0-3  H: 0-3  N: 0-3  O: 0-3

Minimum: 150  Maximum: 206.0

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<th>Calc. Mass</th>
<th>mDa</th>
<th>ppm</th>
<th>DBE</th>
<th>i-FIT</th>
<th>Formula</th>
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<tbody>
<tr>
<td>401.2241</td>
<td>401.2229</td>
<td>1.2</td>
<td>3.0</td>
<td>13.2</td>
<td>33.6</td>
<td>C26 H29 N1 O2</td>
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</tbody>
</table>

**NMR Spectrogram**

- Chemical shifts range from 180 to 20 ppm.
- Various peaks at specific ppm values are observed.
Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for r-IFIT = 2

Monoisotopic Mass, Even Electron Ions
32 formula(s) evaluated with 1 results within limits (up to 20 closest results for each mass)
Elements Used:

C: 0.30 H: 0.30 N: 0.3 O: 0.3

C15H12N2O3 (172.1464 ± 0.0001) Cn+1 (172.1464 ± 0.0001)

1: TOF MS EI+ 2.14e10

Minimum:
Maximum:
Mass Calc. Mass m/z PPM DBE r-IFIT Formula
407.1768 407.1760 -0.4 -0.5 17.5 24.5 C27 H23 N2 O2

R. Koppes, m/z 407.1768
Single Mass Analysis

Tolerance = 5.0 ppm / DDC, min = -1.5, max = 100.0

Element prediction: Off

Number of factape peaks used for I-FFT = 2

Monoisotope Mass, Even Electron Ion

01 formula(s) evaluated with 1 results within limits (up to 10 closest results for each mass)

Elements Used:

C: 5.35 H: 0.35 N: 0.0 O: 0.0

Calculation: C13H13N3O3

1102781.89 [M] (+) 25.40

Minimum:                             Maximum:                             
125.0041                           166.0059                           125.0041                           166.0059

318.1531                           402.1909                           318.1531                           402.1909

Calc. Mass: 5.0 5.6 125.0 166.0 1102781.89 
Mass:      5.0 5.6 17.5 25.4 1102781.89

I-FFT Formula: C13H13N3O3
TDC-164  C3H3/CONH1/043
AR. No.:  MR1265/1714
Analyzer: Wallakeshen
Date: Dec 17 2015
Program: 85
Freq (MHz): 400.22
Shift: 9.30THz
Sample 014 by pet2 Date Friday, February 05, 2016
**Single Mass Analysis**

- **Tolerance**: ± 5.0 PPM
- **DBE**: min = -1.5, max = 106.0
- **Element precision**: C: 0.01, H: 0.02
- **Number of isotope peaks used for i-FIT**: 2

**Microscopic Mass, Even Electron Ion**
72 formulas evaluated with 1 result within limits (up to 10 closest results for each mass)

**Elements Used:**
- C: 3.22, H: 0.35
- N: 0.3, O: 0.5

**Calc/GC/MS/HR MS**
150228

<table>
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<th>PPM</th>
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Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE; min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Tof/Elec Electron Ion
29 formula(s) evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 5.29 H: 0.26 N: 0.3 O: 0.4

C23H26N2O4

325.1911
325.3405
325.2956

Mass Calc. Mass mDa PPM DBE i-FIT Formula
325.191 325.1916 -0.5 -1.5 9.5 5702.3 C20 H25 N2 O2
Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 ppm / DBE: min. = -1.6, max. = 100.0
Element prediction: Off
Number of isotope peaks used for I-RT = 2

Monoisotopic Mass, Elveen Electron ion
22 formulae evaluated with 2 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 0.32 % 5.9 0.0 0.0 0.0 0.0
H: 0.20 % 0.0 0.0 0.0 0.0 0.0
N: 0.32 % 0.0 0.0 0.0 0.0 0.0
O: 0.0 9.7 0.0 0.0 0.0 0.0
CI: 0.0 0.0 0.0 0.0 0.0 0.0

Minimum: 9.9 9.9 9.9
Maximum: 100.0

Mass  Calc. Mass  m/z  PPM  DBE  I-FIT  Formula
355.2057  355.2072  -1.5  -4.2  9.3  328453.5  CP1 N7 N2 O3

FertinEler Spectrometer E3 Version 10.03.06
Friday, February 05, 2010 8:04 PM

Peak Table Group:
DR.REDDY'S LABORATORIES LIMITED,CPS
Analytical Research Department
Single Mass Analysis

Tolerance = 0.5 ppm / DDE: min = -1.5, max = 100.0

Element prediction: C, H

Monoisotopic Mass, Even Electron Ions
80 formulas(s) evaluated with 1 results within limits (up to 10 closest results for each mass)

Elements: C:0-32, H:0-36, O:0-8

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<td>C22 H21 N2 O2</td>
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</tbody>
</table>

DR. REDDY'S LABORATORIES LIMITED, CPS
Analytical Research Department

PerkinElmer Spectrum ES Version 10.03.05
Friday, February 05, 2016 8:24 PM

C383-CJSR1-052_1_1 Sample 021 By pet2 Date Friday, February 05 2016
Elemental Composition Report

Single Mass Analysis
Tolerance = 6.0 PPM / DBE: min = -1.5, max = 103.0
Element precision: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
evaluated with 1 results within limits (up to 5 closest results for each mass)
Elements Used:
C: 3-22  H: 0-30  N: 0-4  O: 0-4  F: 0-1

1000.0000  (3.0000)  1000.0000  (3.0000)

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</table>

Page 1
DR. REDDY'S LABORATORIES LIMITED, CPS
Analytical Research Department

[Chemical structure image]

Peak Table

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[Chemical structure image]

[Additional chemical structure image]
Single Mass Analysis
Tolerance = 5.0 PPM / DRM: min = 1.5, max = 110.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
150 formulas evaluated with 1 results within limits (up to 10 closest results for each mass)

Elements Used:

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<th>O-100</th>
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<td>2</td>
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</table>

Calc. Mass 531.1469

C18 H23 N2 O2 S

Peak Table Graph

DR.REDDY'S LABORATORIES LIMITED, CPS
Analytical Research Department

PerkinElmer Spectrum ES Version 10.03.
Friday, February 05, 2016 7:16 1

Name     Description cm⁻¹
C385-CJ9P1-025_ Sample 007 By plt2 Date Friday, February 05 2016
Elemental Composition Report

Single Mass Analysis
Tolerance = 6.0 ppm / DDE: min = -1.5, max = 100.0
Elements prediction: Cfr
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
150 formulas are evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 0.24 H: 0.59 N: 0.0 O: 0.0 S: 0.0

C Naz/CuH9NS0
1622.04013 (E,ESI) 01:70M 55I.ESI
1,444.005

Minimum
Maximum
Mass Calc. Mass m/z DDE i-FIT Formula
351.1176 351.1169 0.8 1.5 25.0 N2O 100.0

PerkinElmer Spectrum ES Version: 10.03.06
Friday, February 06, 2016 1:16 PM

Peak Table Graph:

DR. REDDY'S LABORATORIES LIMITED, CPS
Analytical Research Department

C36S-CJER-136_1-1
Sample 007 By pet2 Date Friday, February 06 2016

Analized By:
pet2
Analized Date:
2/6/2016 7:13:23 PM
Elemental Composition Report

Single Mass Analysis

Element prediction: OR

Monoisotopic Mass, Even Electron Ions
26 formulations evaluated with 1 result within limits (up to 2 closest results for each mass)

Elements Used:
C: 0.30  H: 0.76  N: 0.03  O: 0.54

C21H19N2

DR. REDDY'S LABORATORIES LIMITED, GPS
Analytical Research Department

Sample 007 By ssd Date Friday, February 06 2015

Analyzed By: ssd
Analyzed Date: 2/6/2015 1:13:23 PM
Checked By:
Checked Date:
Elemental Composition Report

Single Mass Analysis
Tolerance = 2.0 PPM / DBE: min = 1.5, max = 100.0
Element prediction: On
Number of isotope peaks used for i-RIT: 2

Monoisotopic Mass, Exact Electron Ion
34 formula(s) evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 0.25 H: 0.29 N: 0.3 O: 0.4

Minimum:

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Mass Analysis Report

Data Filename: 36090005.d
Sample Name: CH3C6H5/67 PH-2
User Spectra

--- End Of Report ---

Dr. Reddy's
C6H25C6H5/66 (Ph-1)
1H NMR CDCl3
21-06-2016
ANALYSED BY: KRP

Bruker

Current Data Parameters
MODE: 600.136912 MHz
MCRE: 12.1327 MHz
SUSP: 0.0000 MHz
SNR: 30.13
Noise: 0.0000 ppm
Proton: 600.136912 MHz
Frequency: 30.13 ppm

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Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monotopic Mass, Even Electron Ions
70 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 0-20 H: 0-25 N: 0-5 O: 0-4
C36H44N2O2
1211292500 25 (0.473) Cn (0.23)

Mass Calc. Mass m/z PPM DBE i-FIT Formula
367.1805 367.1810 0.5 101.0 100 545.9 C20 H23 N2 O

Aurigene
Inorganic Chemistry
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotopic peaks used for i-FT = 2

Monoisotopic Mass, Even Electron Ion
47 formula(s) evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 0.02 H: 0.05 N: 0.5 O: 0.4
C13H11NO5S
181/2004 205 5.69% Cl (39-39)

Mass    Calc. Mass   a/Na   RM  DBE   i-FT   Formula
137.1916 137.1916  0.0  9.0  10.5  53.9  C21 H25 N2 O2