

Supporting Information for

CsOH / γ -Al₂O₃: A heterogeneous reusable basic catalyst for one pot synthesis of 2-amino-4,6-diaryl pyrimidines

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1. General information

Unless otherwise stated, all reagents were purchased from Sigma-Aldrich (India) and used without purification. Melting points are uncorrected. IR spectra were recorded on Frontier Perkin Elmer IR spectrometer. ^1H NMR and ^{13}C NMR spectra were obtained on a Bruker AVANCE 300 MHz instrument in CDCl_3 using TMS as internal standard. Chemical shifts (δ) are expressed in ppm. Mass spectra were recorded on Thermofisher Scientific Polaris Q instrument. Elemental analysis (C, H, N) were carried out on elemental analyzer EA 3000, Euro Vector, Italy.

2. Characterization data of selected compounds

Example 1. 4-(4-chlorophenyl)-6-phenylpyrimidin-2-amine (3b)

white solid, yield: 80%; mp:160 °C; IR (cm⁻¹): 3489.99, 3305.30, 1629.78, 766.92, 694.11; ¹H-NMR (CDCl₃): 5.43 (s, 2H, NH₂), 7.38-7.49 (m, 6H, aromatic), 7.97-8.03 (m, 4H, aromatic); ¹³C-NMR(CDCl₃): 103.9, 127.1, 128.4, 128.7, 128.9, 130.5, 136.1, 136.5, 137.6, 163.6, 164.8, 166.4; MS (m/z) 281.31; Elem. Anal. Calculated for C₁₆H₁₂N₃Cl: C, 68.20; H, 4.29; N, 14.91. Found: C, 68.42; H, 4.31; N, 14.68%.

Example 2. 4-phenyl-6-(p-tolyl) pyrimidin-2-amine (3e)

white solid, yield: 80%; mp:128 °C; IR (cm⁻¹): 3309.20, 3193.44, 2980.37, 1630.45, 1528.36, 1363.12, 767.83; ¹H-NMR (CDCl₃): 2.40 (s, 3H, CH₃), 5.44 (s, 2H, NH₂), 7.26 (d, 2H, aromatic), 7.41 (s, 1H, aromatic), 7.46-7.49 (m, 3H, aromatic), 8.01-8.04 (m, 4H, aromatic); ¹³C-NMR(CDCl₃): 21.4, 103.9, 127.0, 127.1, 128.7, 129.4, 130.3, 134.9, 137.9, 140.7, 163.6, 166.10, 166.16; MS (m/z) 261.31; Elem. Anal. Calculated for C₁₇H₁₅N₃: C, 78.13; H, 5.78; N, 16.07. Found: C, 78.46; H, 5.52; N, 15.87%.

Example 3. 4-(4-bromophenyl)-6-(4-fluorophenyl) pyrimidin-2-amine (3g)

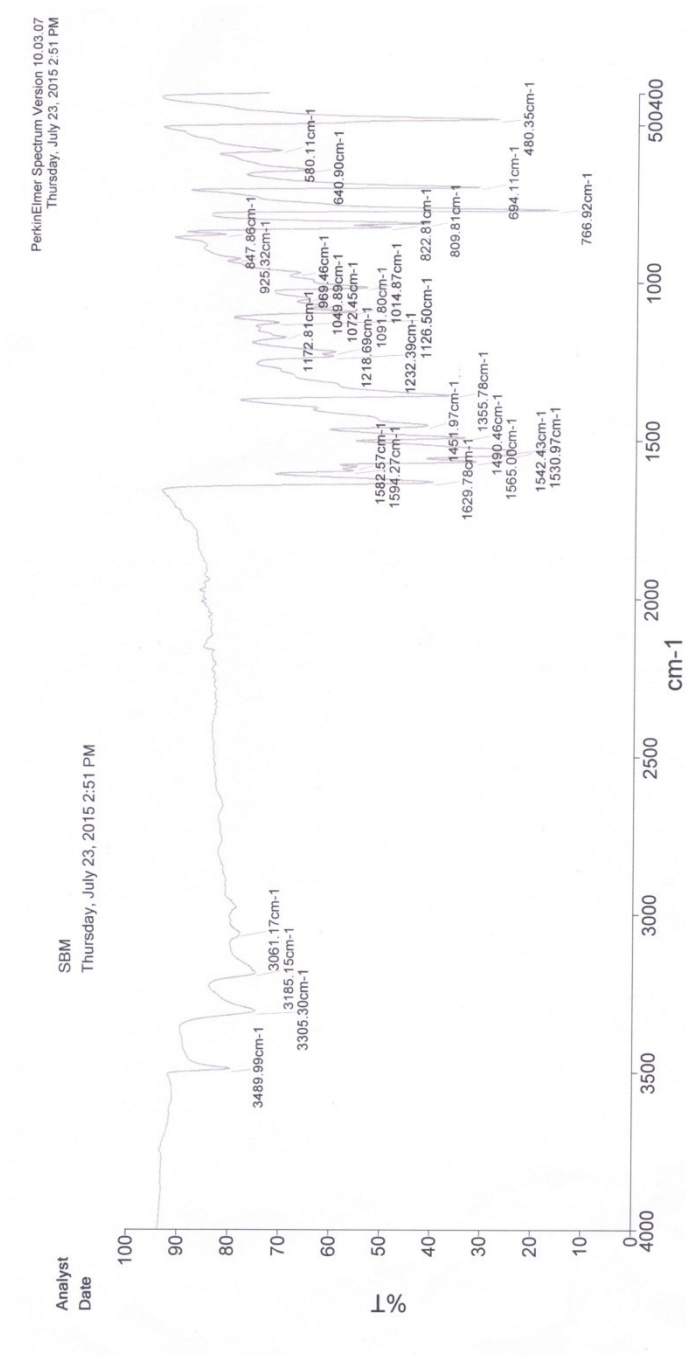
white solid, yield: 72%; mp:154°C; IR (cm⁻¹): 3491.84, 3327.78, 3206.99, 1633.65, 1364.77, 1218.69, 806.84; ¹H-NMR (CDCl₃): 5.16 (s, 2H, NH₂), 7.18 (d, 2H, aromatic), 7.38 (s, 1H, aromatic), 7.61 (dd, 2H, aromatic), 8.04-8.06 (m, 4H, aromatic); ¹³C-NMR(CDCl₃): 103.5, 115.6, 115.9, 125.1, 128.6, 129.0, 129.2, 131.9, 133.6, 136.5, 163.5, 165.0, 165.3; MS (m/z) 343.39; Elem. Anal. Calculated for C₁₆H₁₁N₃BrF: C, 55.83; H, 3.22; N, 12.20. Found: C, 55.75; H, 3.34; N, 12.42%.

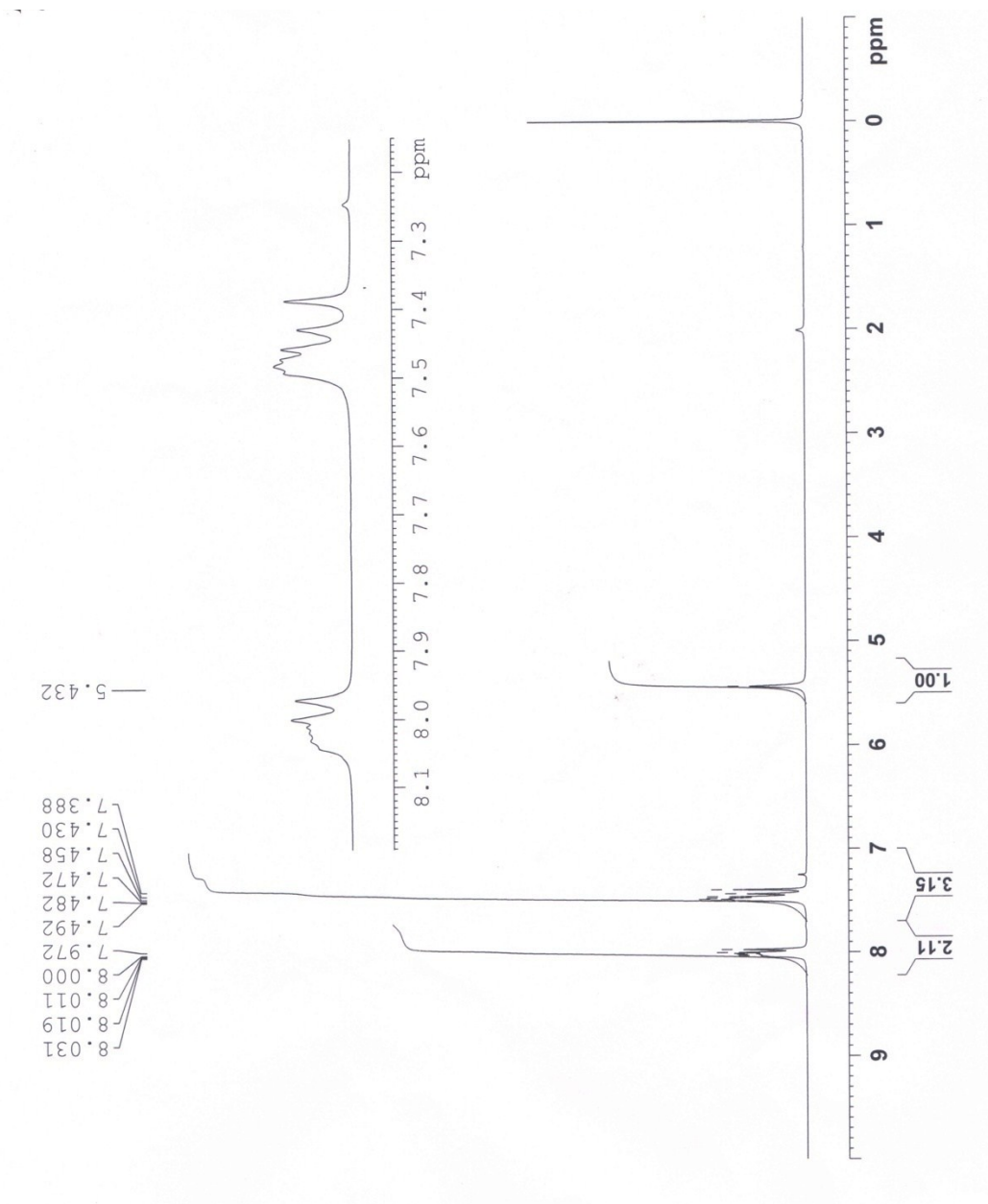
Example 4. (2-(2-amino-6-(4-bromophenyl) pyrimidin-4-yl)cyclopenta-2, 4-dien-1-yl) (cyclopenta-2,4-dien-1-yl) iron (3h)

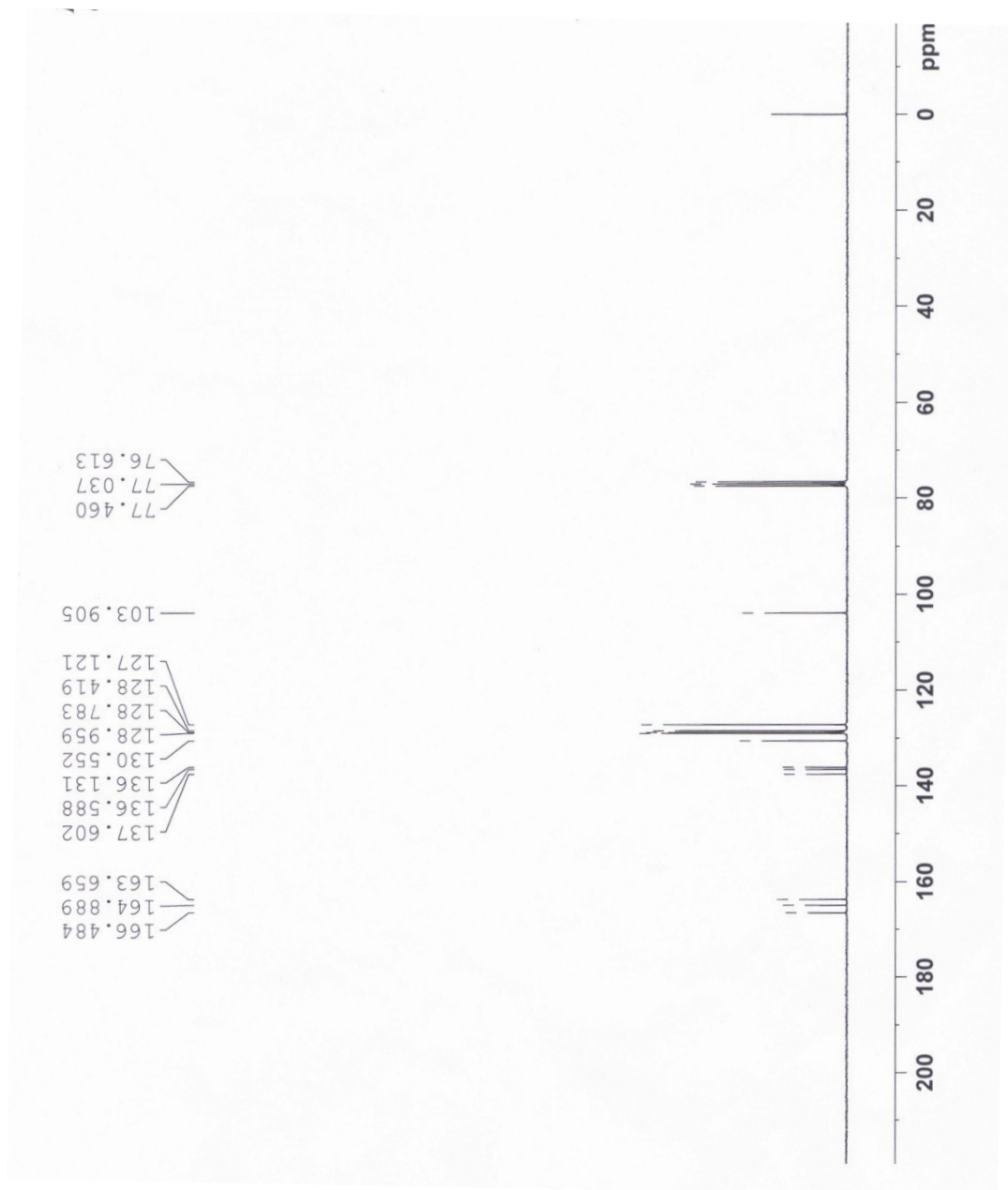
red solid, Yield: 78%; mp: 188°C; IR (cm⁻¹): 3307.26, 3190.28, 2923.48, 1632.95, 1529.99, 812.24, 748.24; ¹H-NMR (CDCl₃): 4.10 (s, 5H, cp ring), 4.46 (t, 2H, cp ring), 4.96 (t, 2H, cp ring), 5.20 (s, 2H, NH₂), 7.07 (s, 1H, aromatic), 7.60 (d, 2H, aromatic), 7.89 (d, 2H, aromatic); ¹³C-NMR(CDCl₃): 68.0, 69.9, 70.8, 80.9, 103.5, 124.7, 128.5, 131.8, 136.7, 163.3, 163.4, 169.7; MS (m/z) 433.51; Elem. Anal. Calculated for C₂₀H₁₆BrFeN₃: C, 55.33; H, 3.71; N, 9.67. Found: C, 55.52; H, 3.82; N, 9.91%.

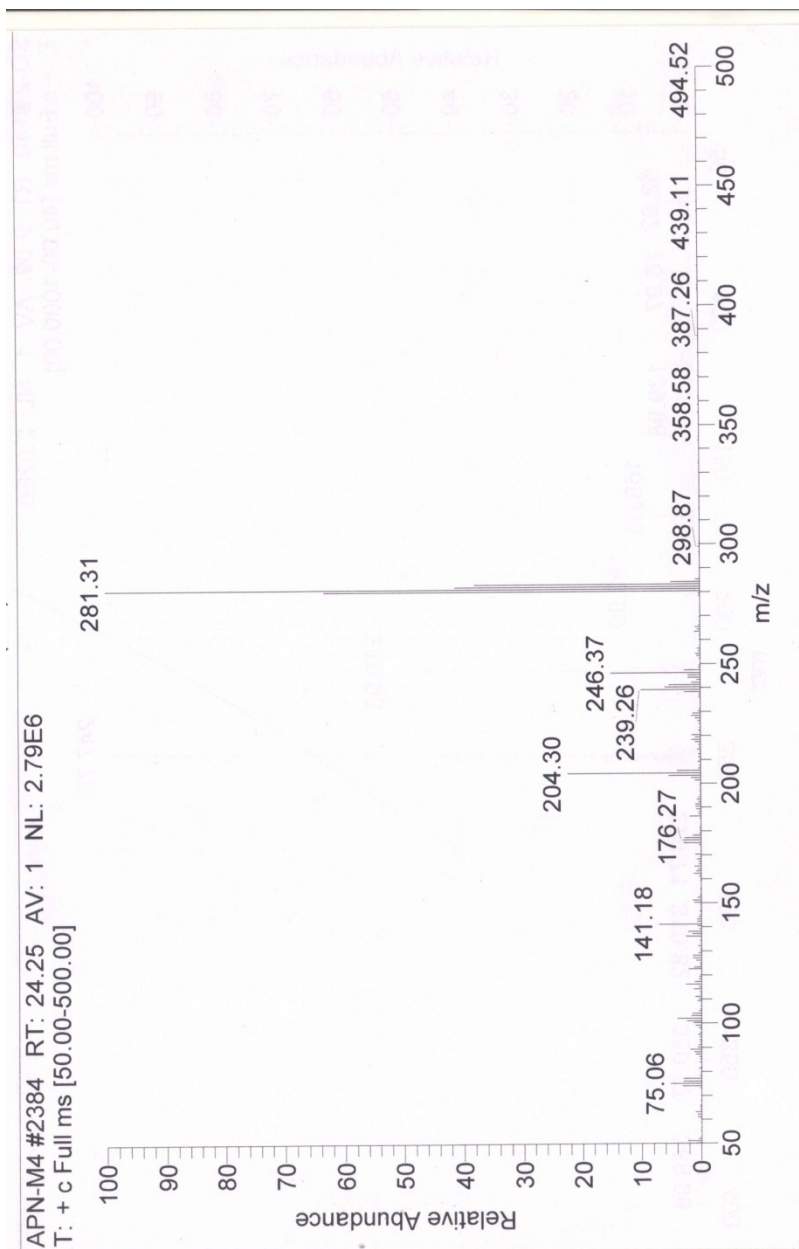
Spectra:

Example 1. 4-(4-chlorophenyl)-6-phenylpyrimidin-2-amine (**3b**)

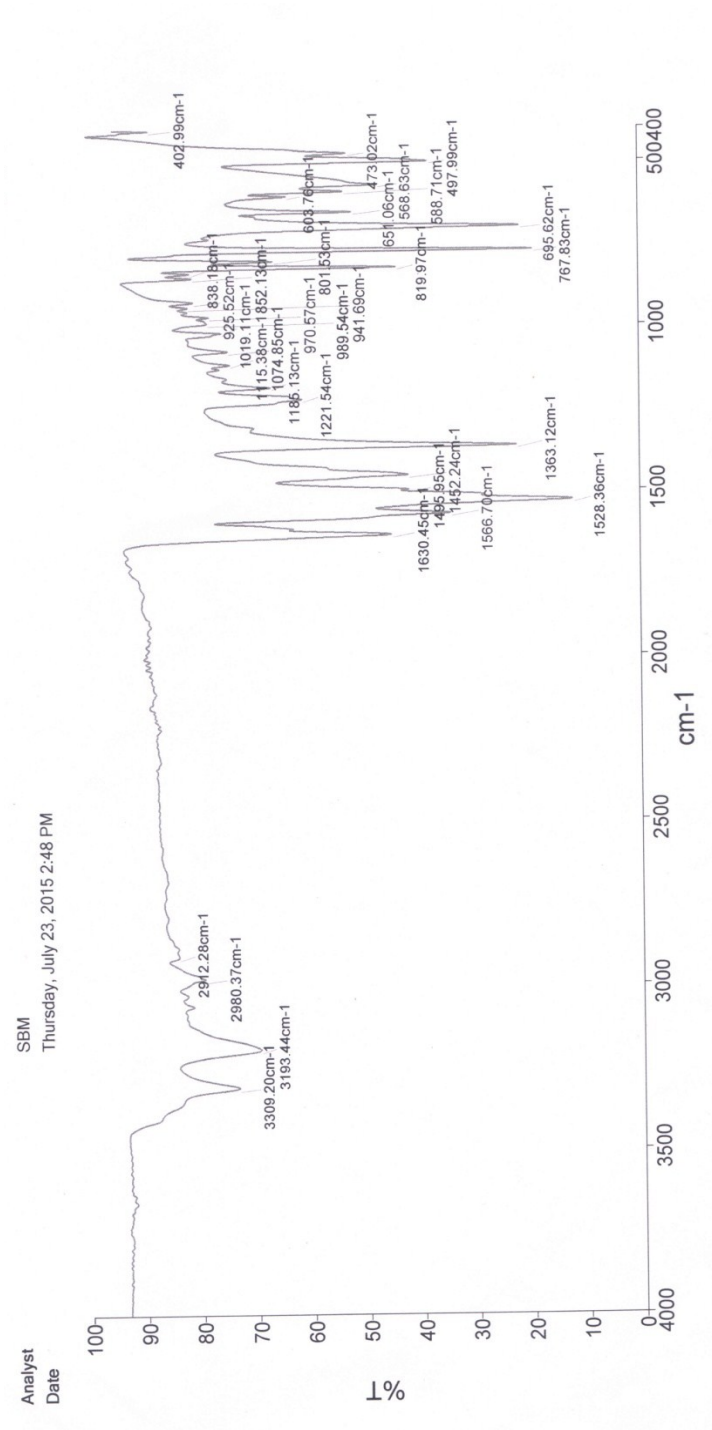


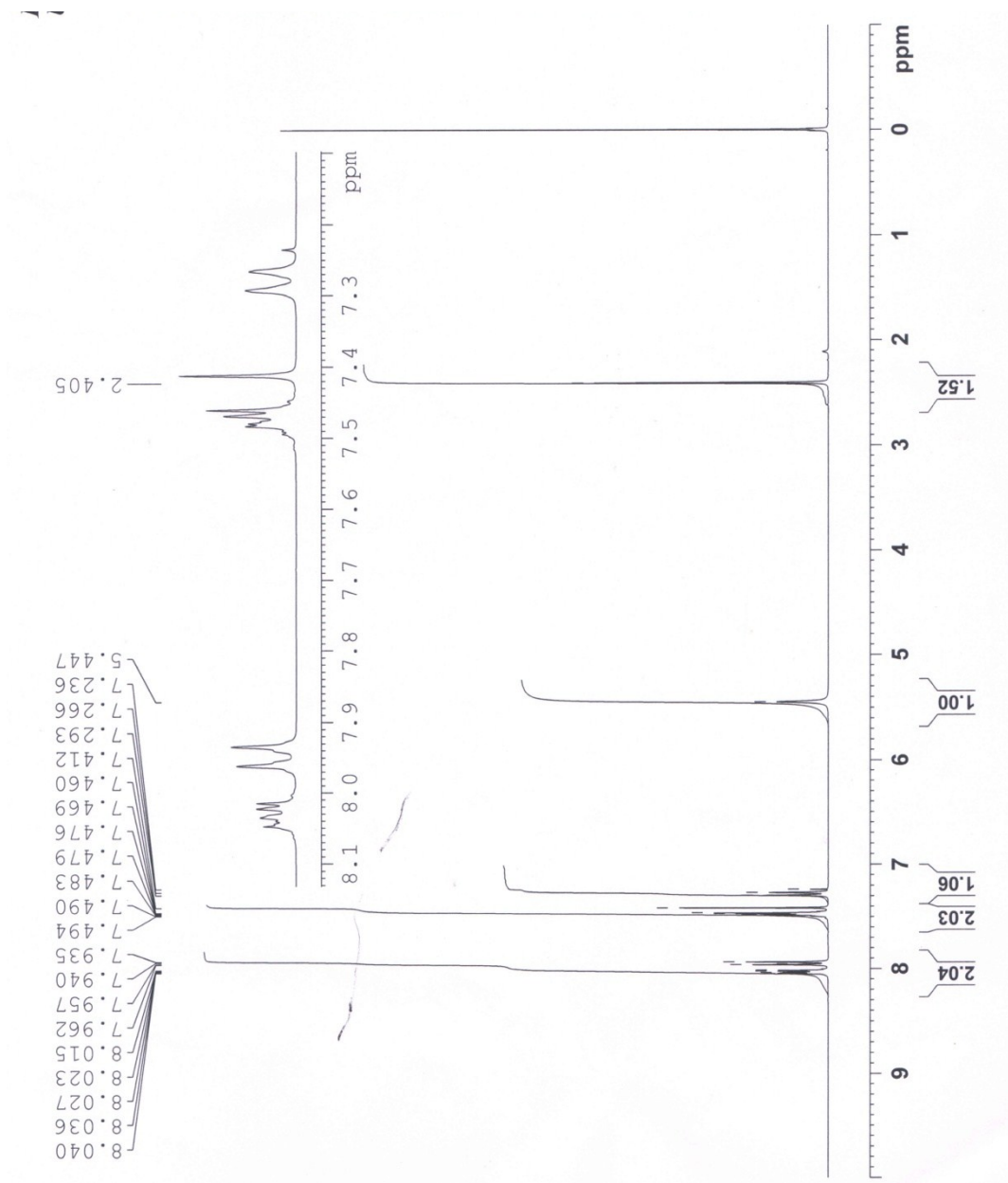


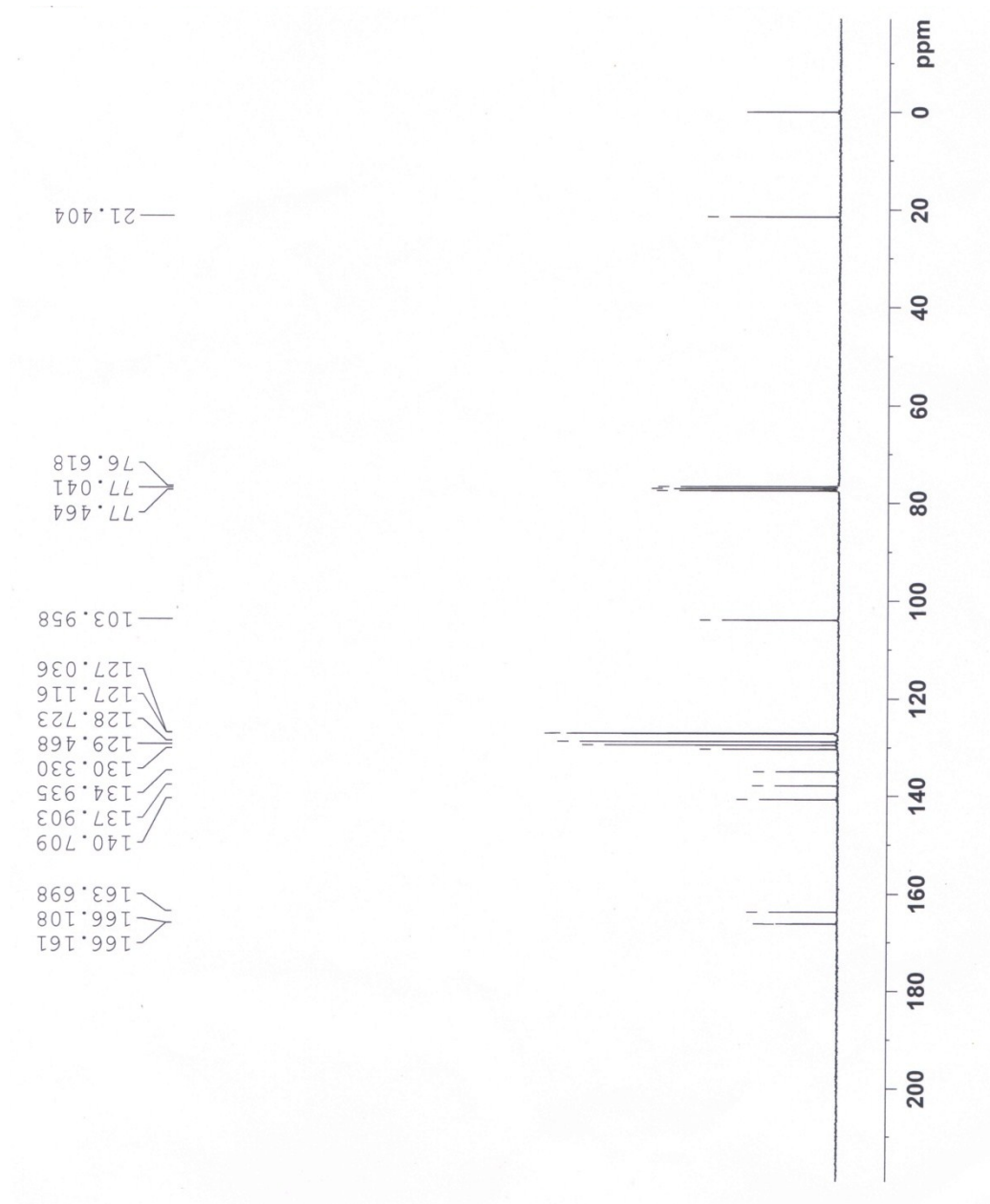


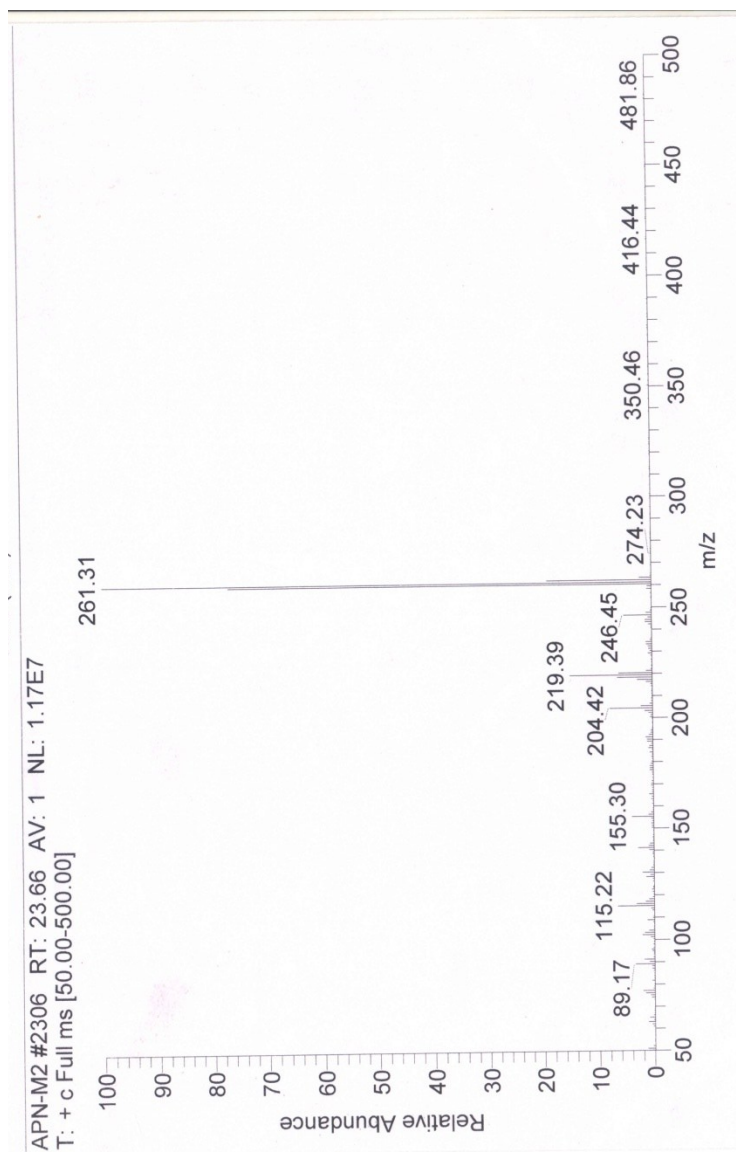


Example 2. 4-phenyl-6-(p-tolyl) pyrimidin-2-amine (**3e**)

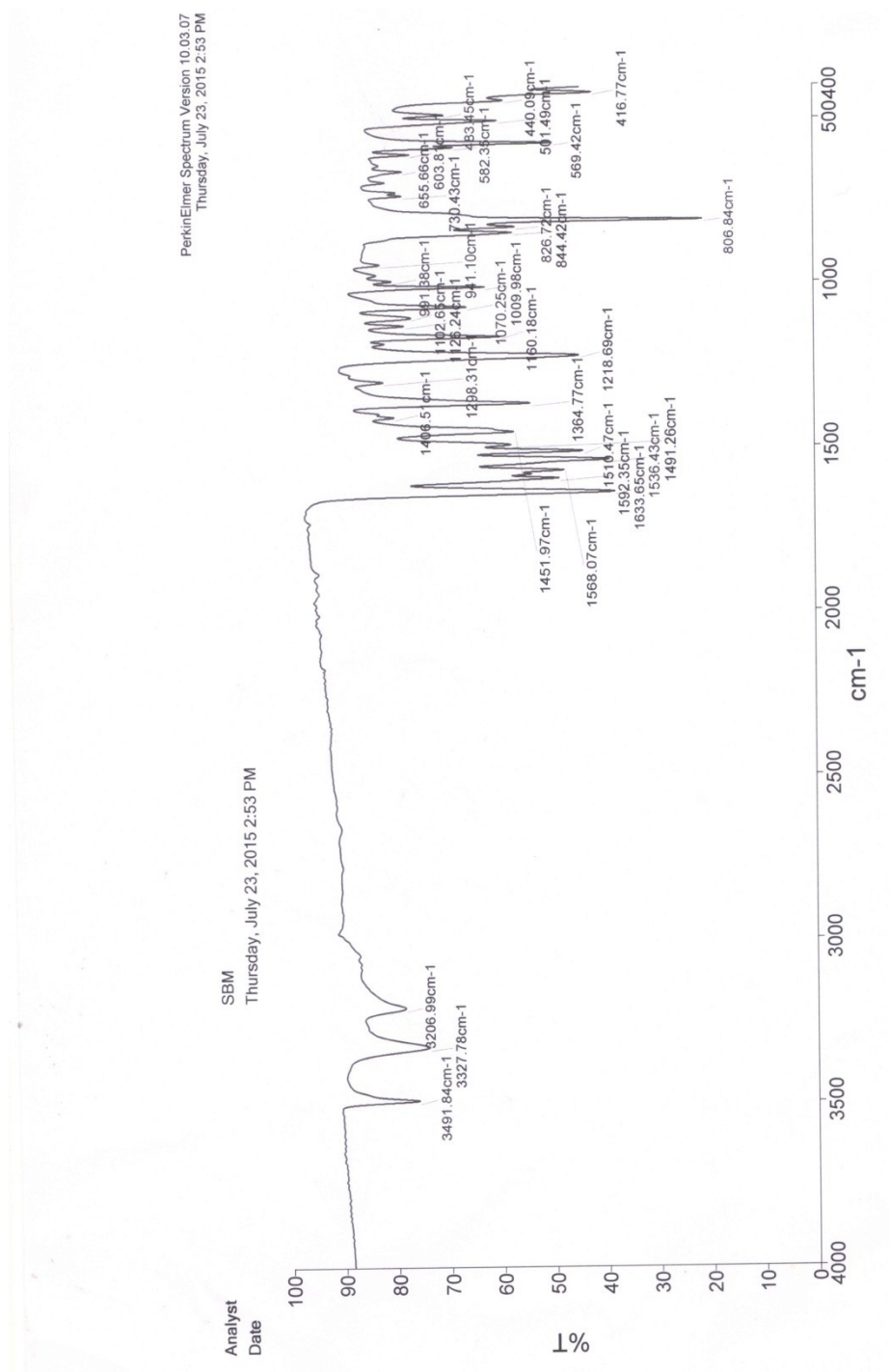


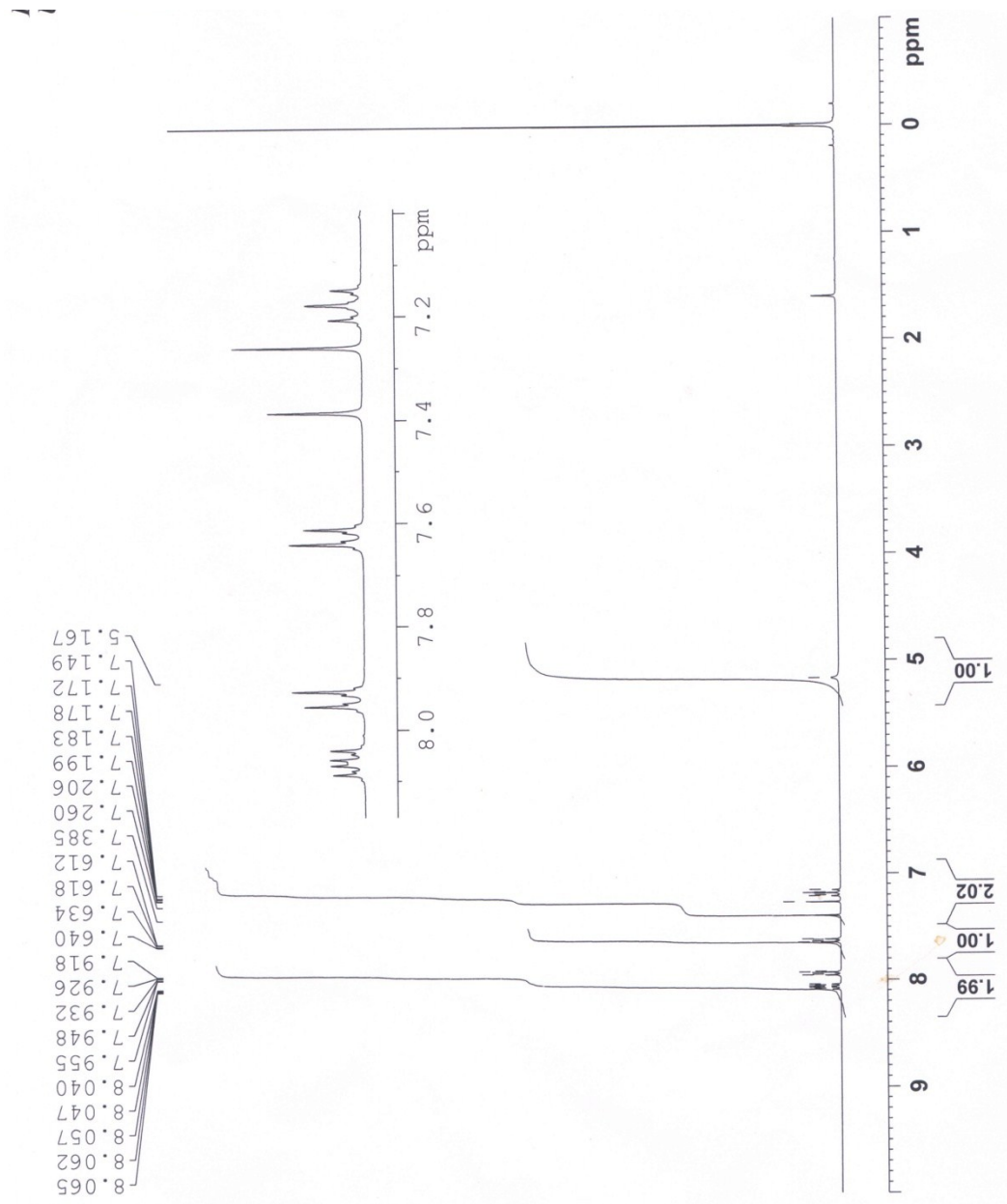


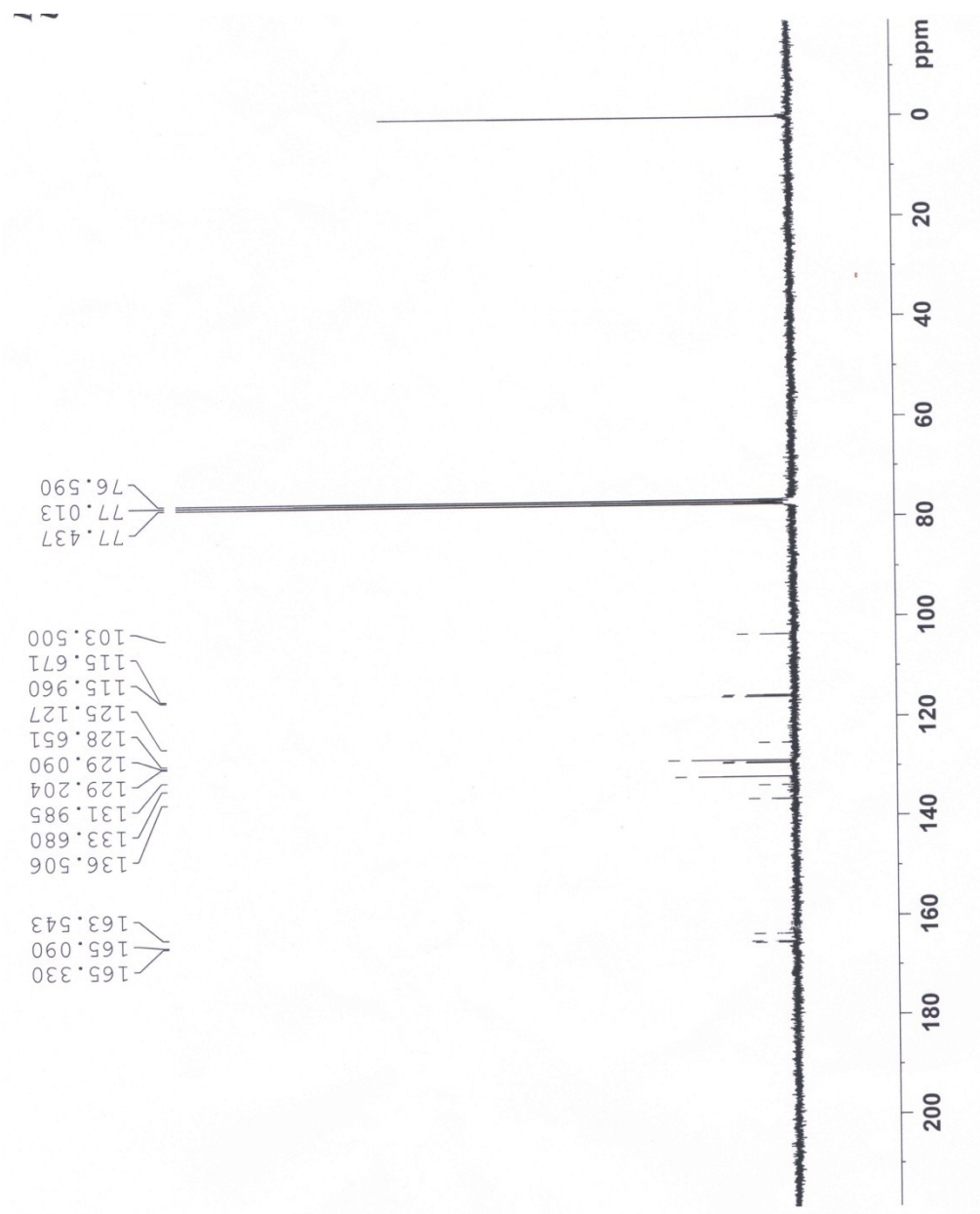


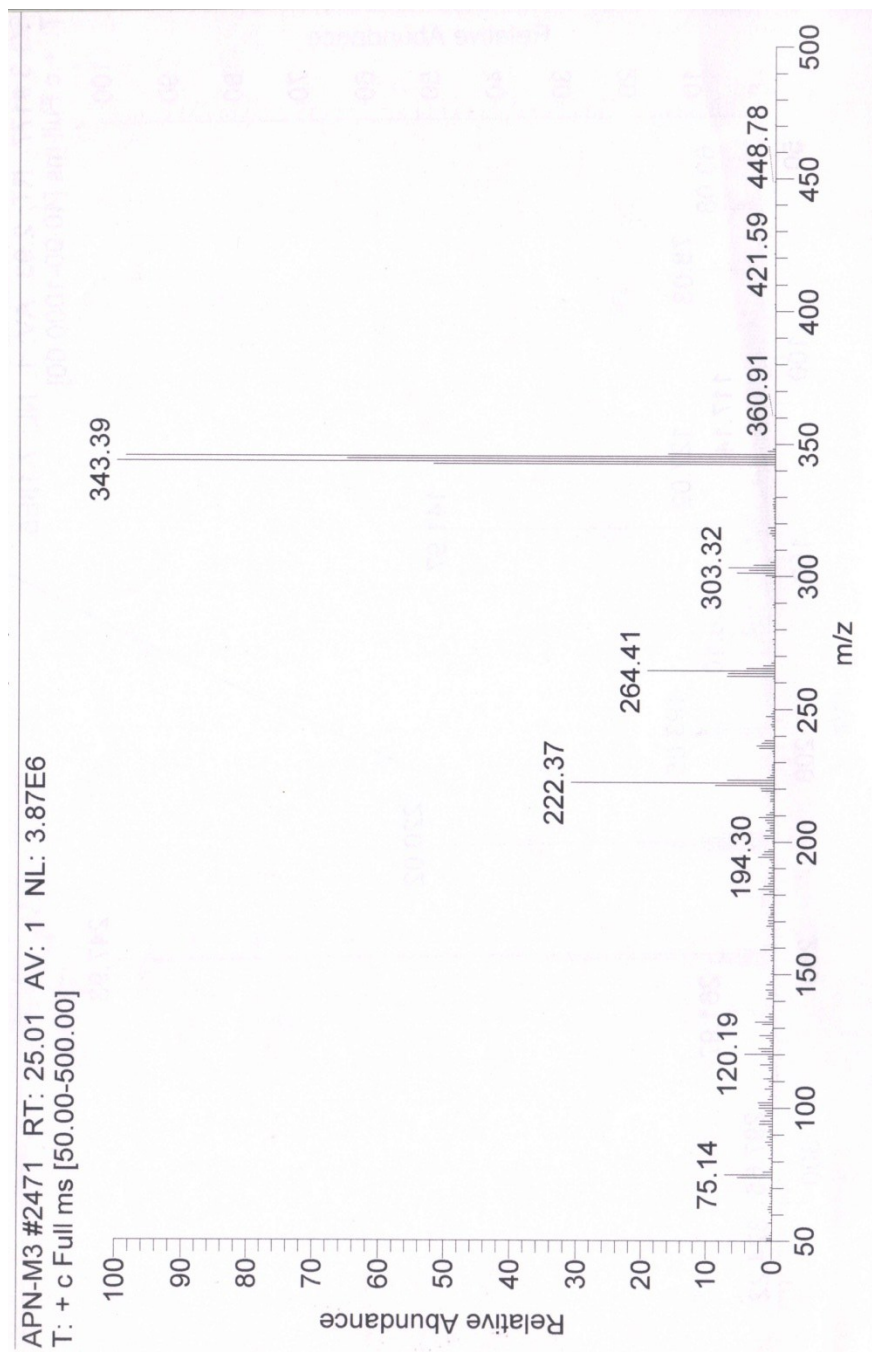


Example 3. 4-(4-bromophenyl)-6-(4-fluorophenyl) pyrimidin-2-amine (3g)

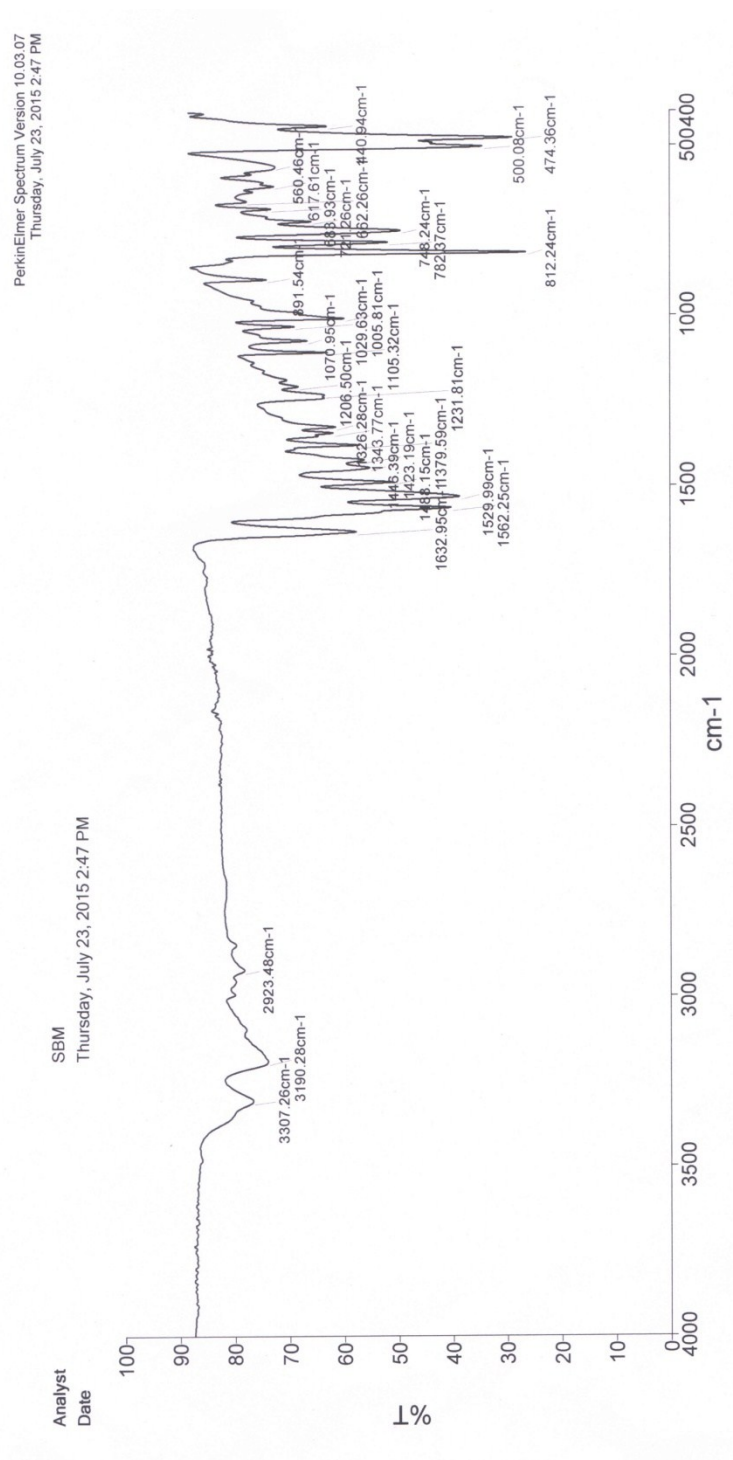


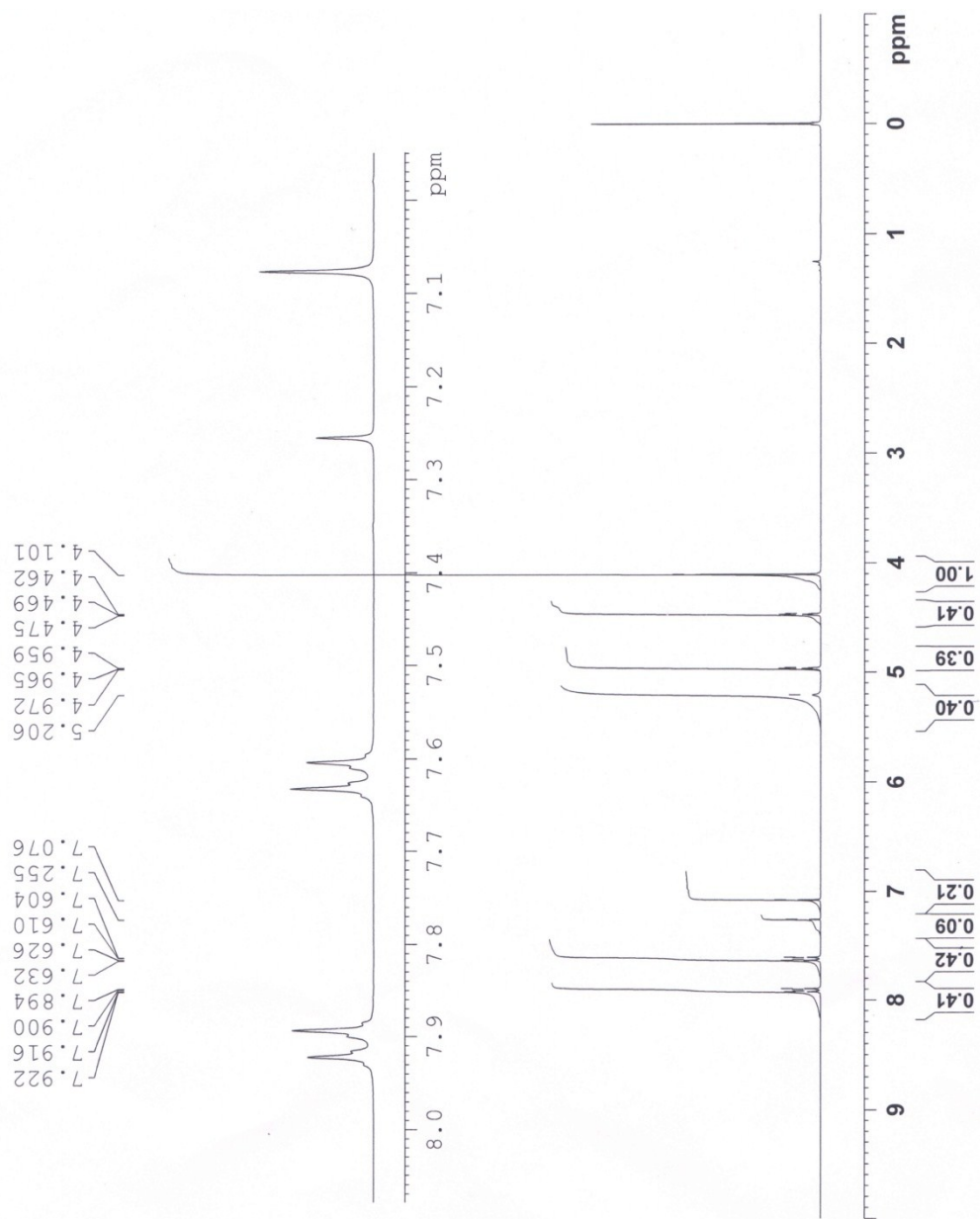


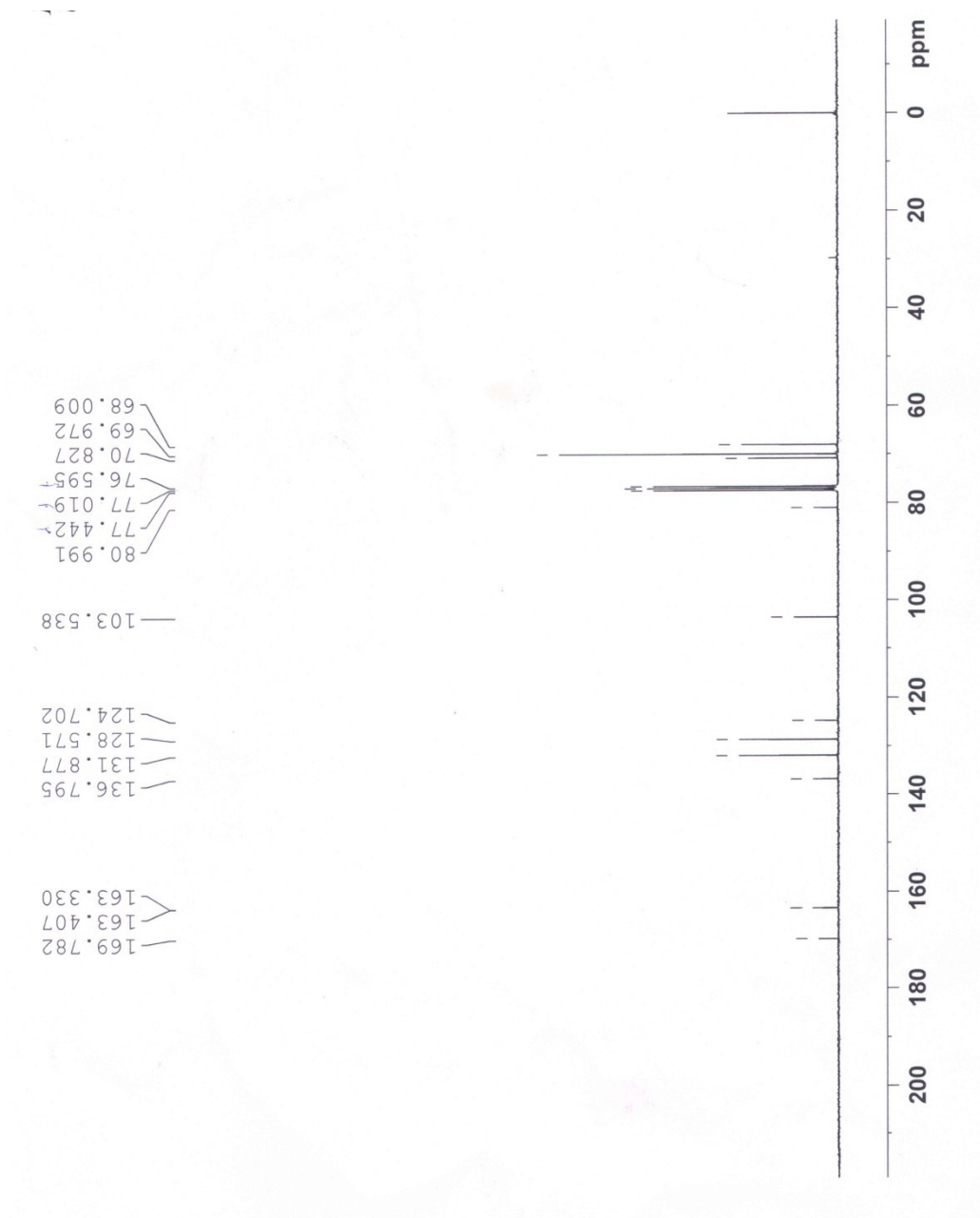




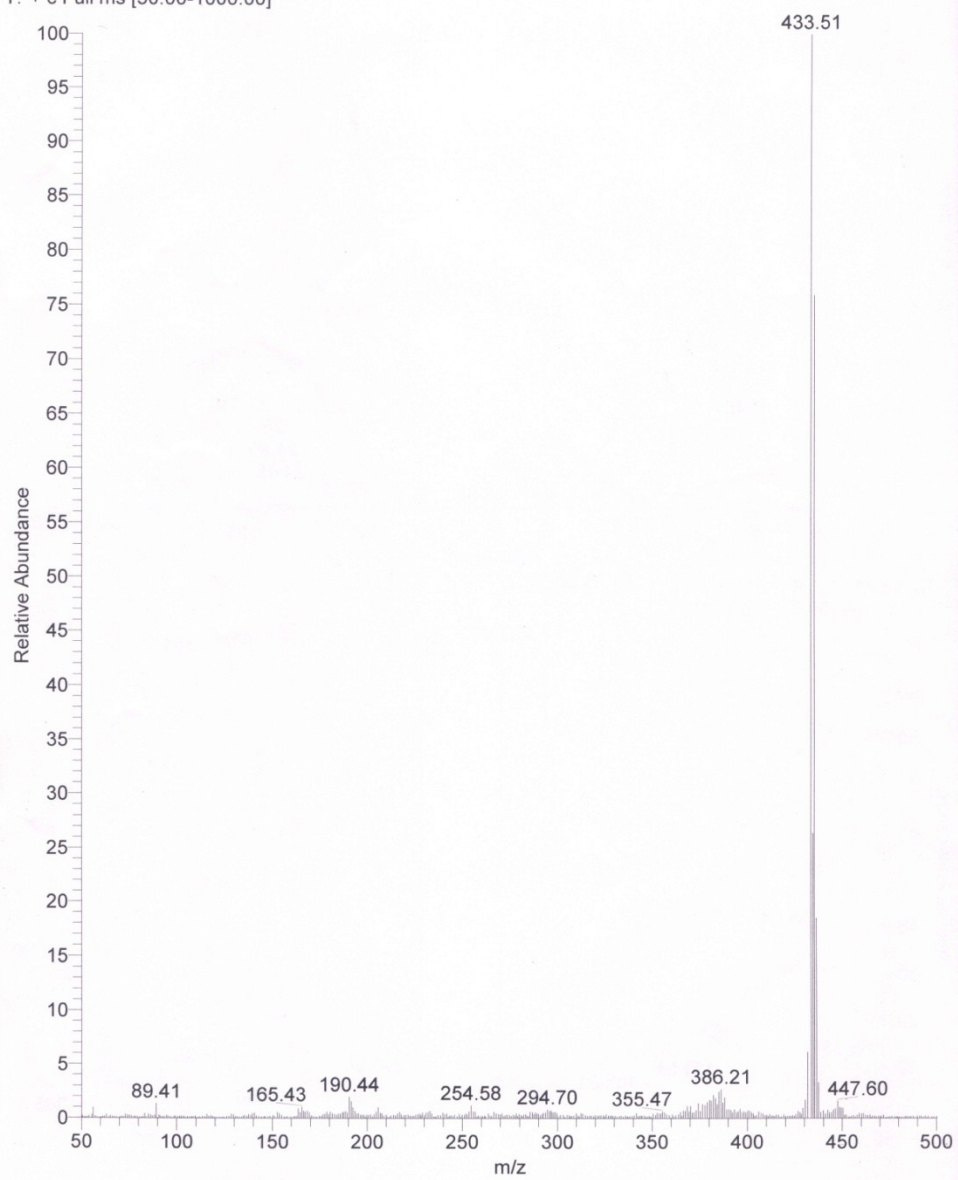
Example 4. (2-(2-amino-6-(4-bromophenyl) pyrimidin-4-yl)cyclopenta-2, 4-dien-1-yl) (cyclopenta-2,4-dien-1-yl) iron (**3h**)







APN-M1_150801140026 #285 RT: 4.06 AV: 1 NL: 4.87E7
T: + c Full ms [50.00-1000.00]



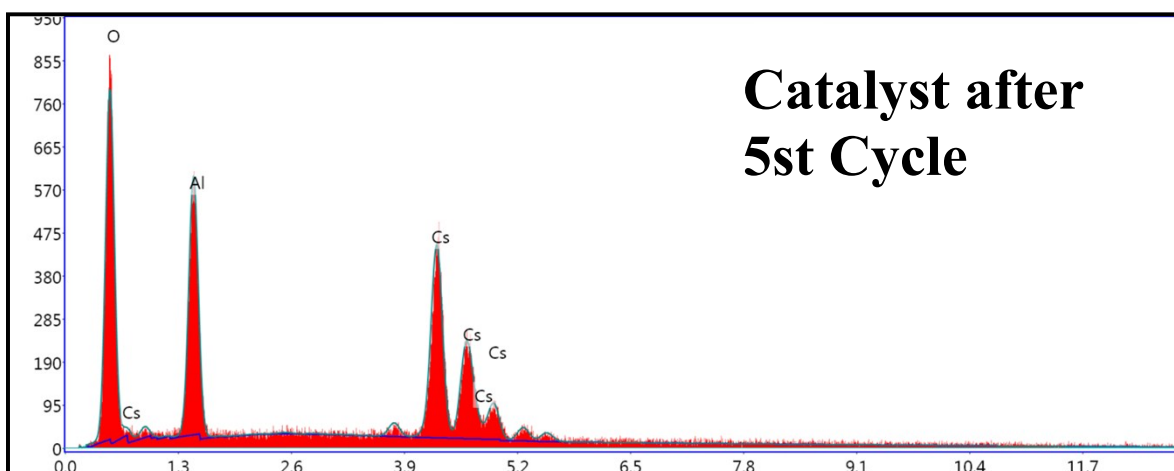
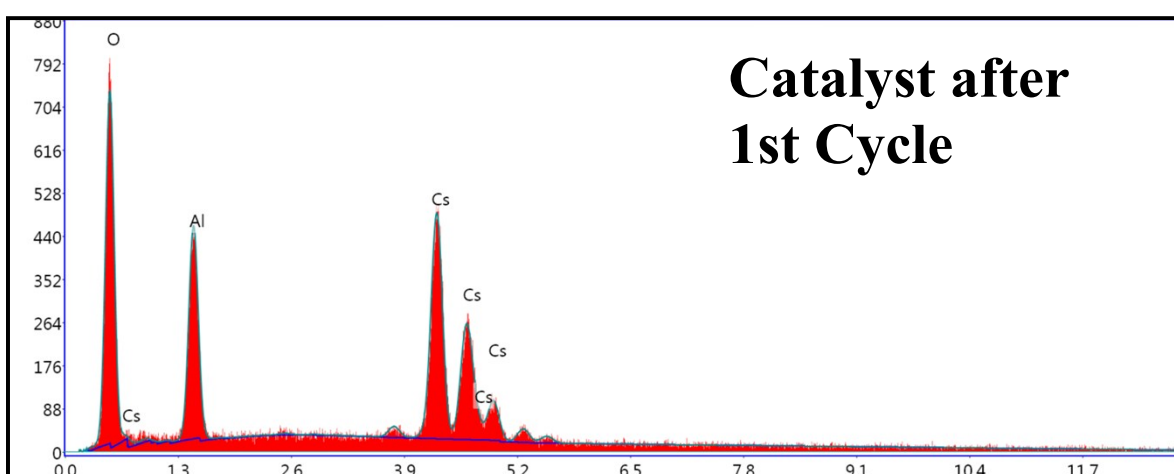
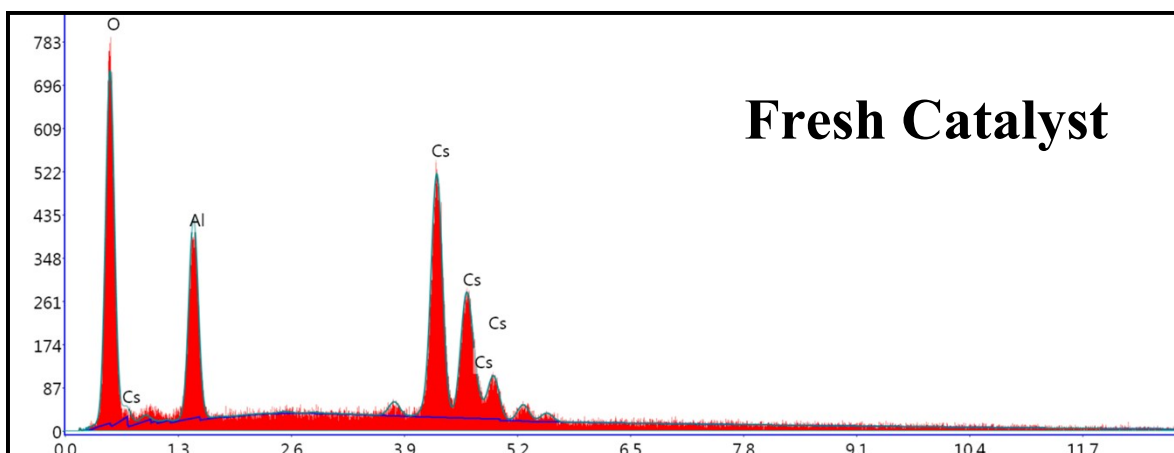


Fig. EDAX pattern of the fresh catalyst, catalyst after 1st cycle of reuse and catalyst after 5th cycle of reuse

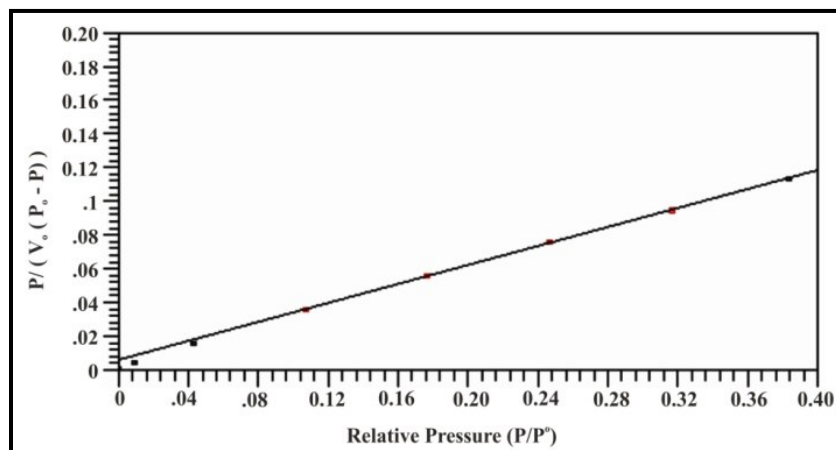


Fig. BET plot of CsOH/ γ -Al₂O₃ catalyst