Electronic Supplementary Information (ESI)

New Journal of Chemistry

Three-component synthesis of 4*H*-pyran scaffolds accelerated by a Gabapentin-based natural deep eutectic solvent

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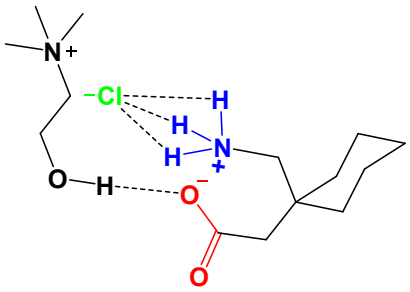
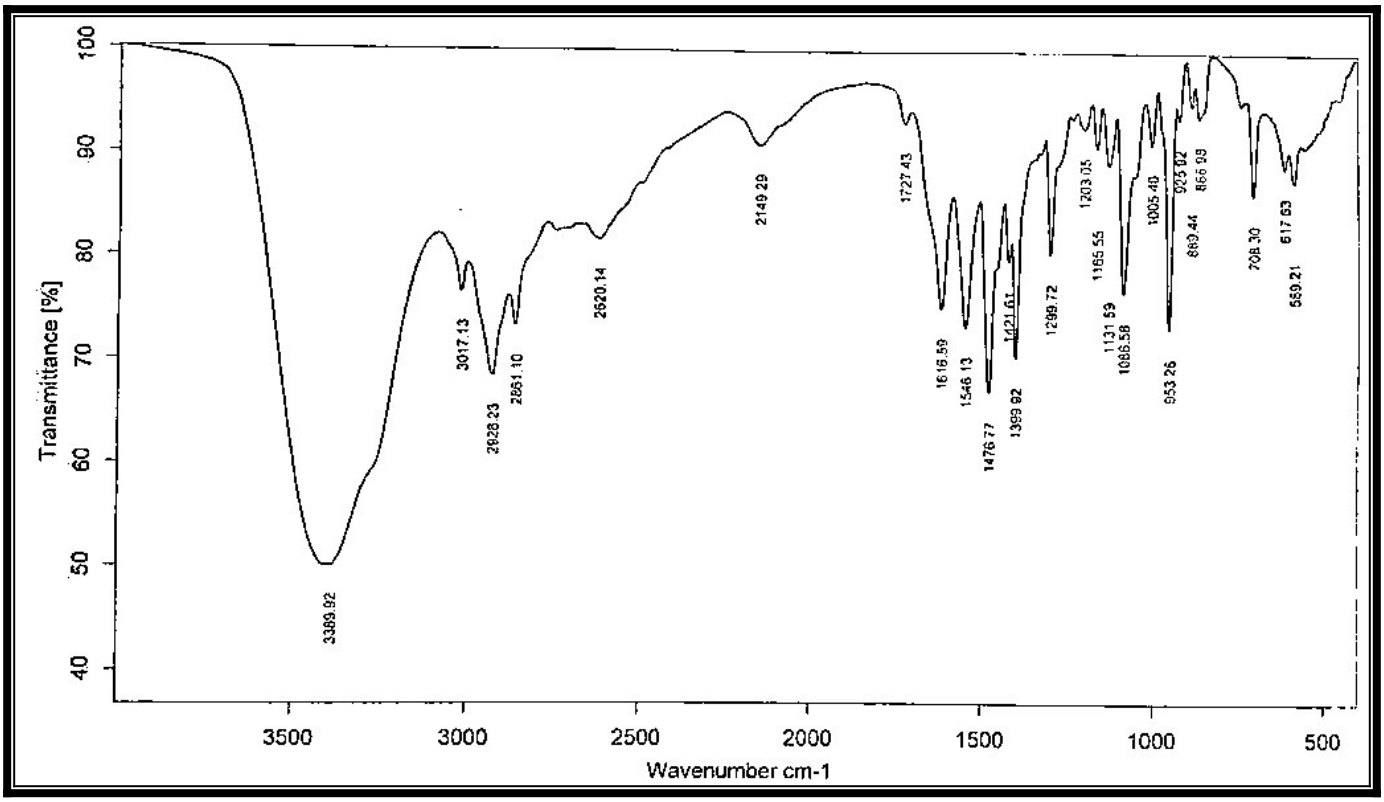
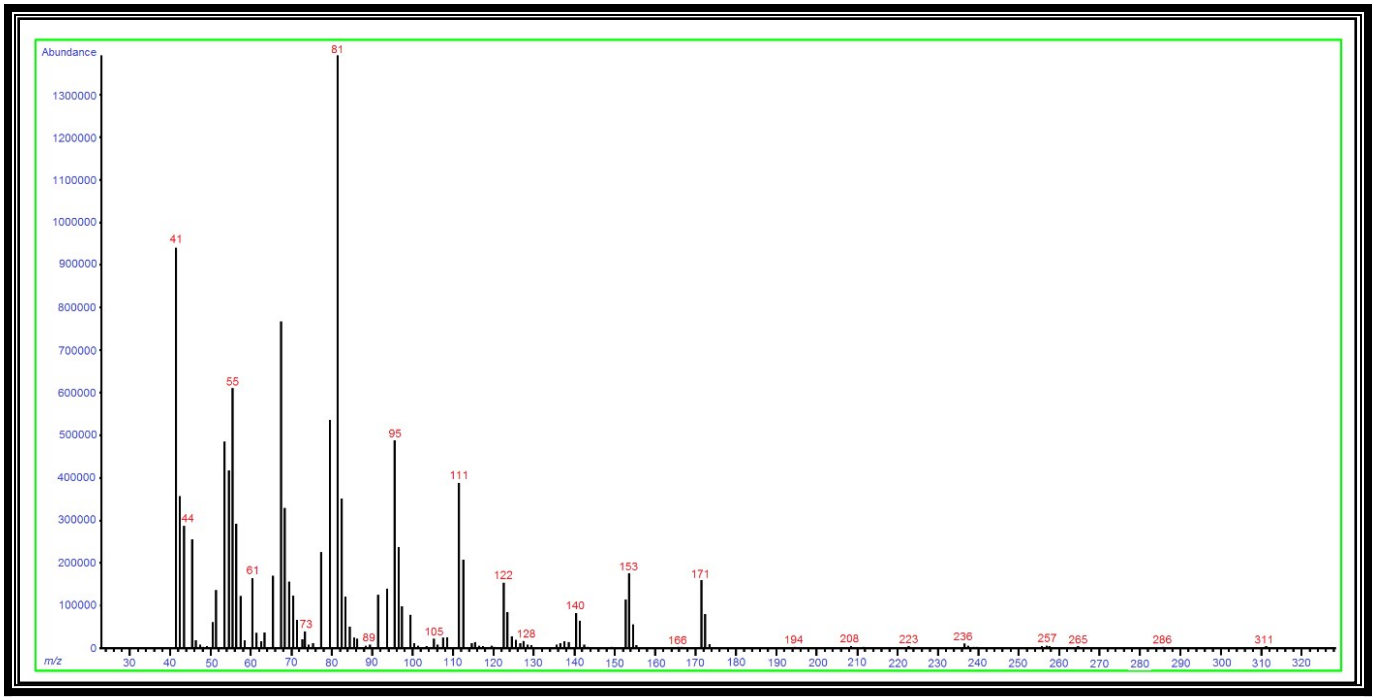
Spectral data for [Ch]Cl:Gab NADES : White highly viscous liquid; M.P. 25 °C; MS: *m/z* = 311 (M^+); FT-IR (KBr, cm^{-1}) υ_{max}: 3389, 3017, 2928, 2861, 2620, 2149, 1616, 1546, 1476, 1399, 1299, 1087, 953, 708, 618, 589; ^{1}H NMR (400 MHz, $CDCl_3$-*d6*): δ (ppm) 1.20-1.41 (m, H), 2.22 (s, 2H), 2.64 (s, 2H), 3.44 (t, *j*=4 Hz, 2H), 3.8 2(q, *j*=4 Hz, 2H), 4.00-5.00 (w, NH & OH); ^{13}C NMR (100 MHz, $CDCl_3$-*d6*): δ(ppm) 21.5, 22.8, 26.0, 34.1, 34.7, 36.6, 53.6, 55.5, 67.4, 175.1 (COO^-).

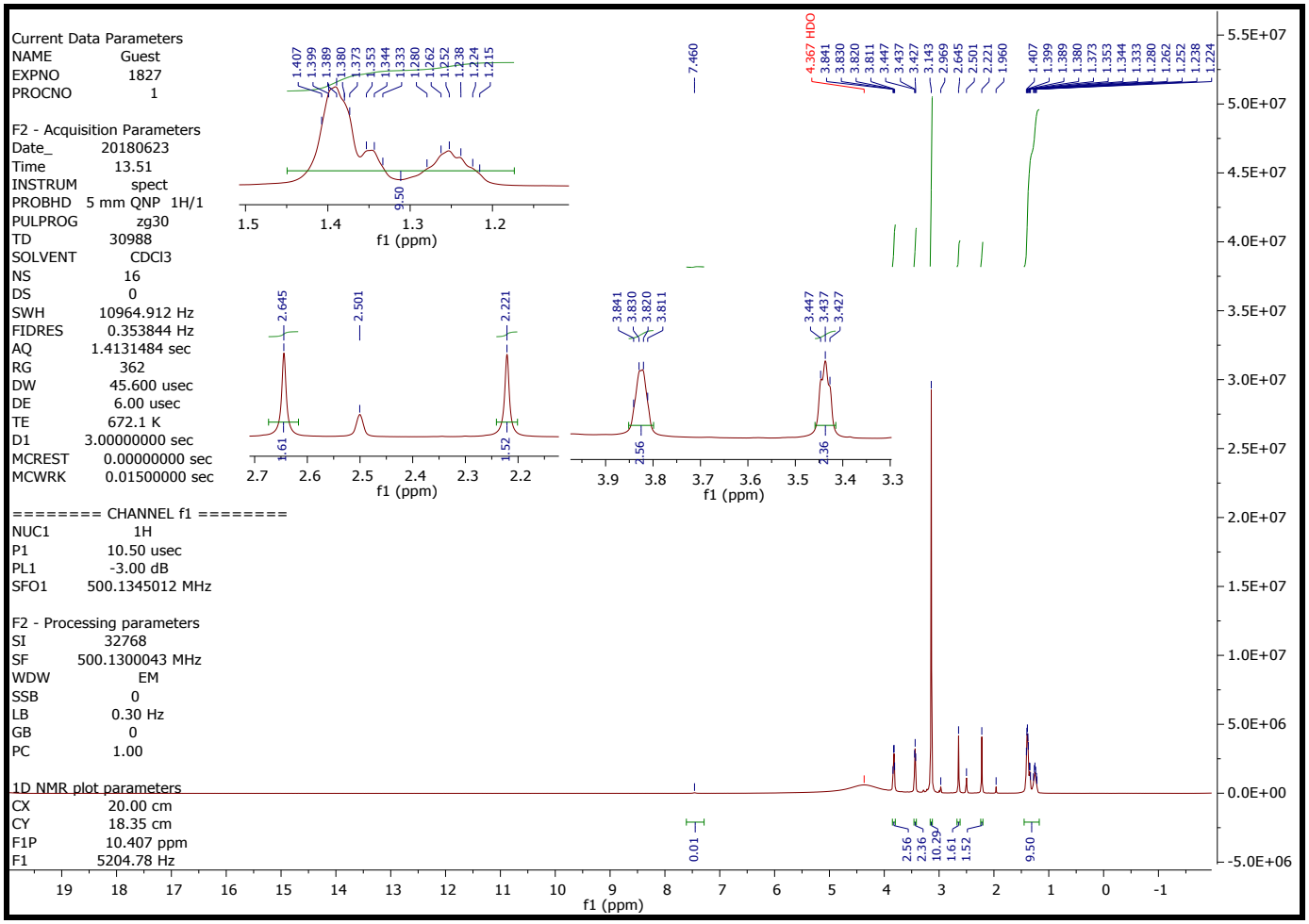




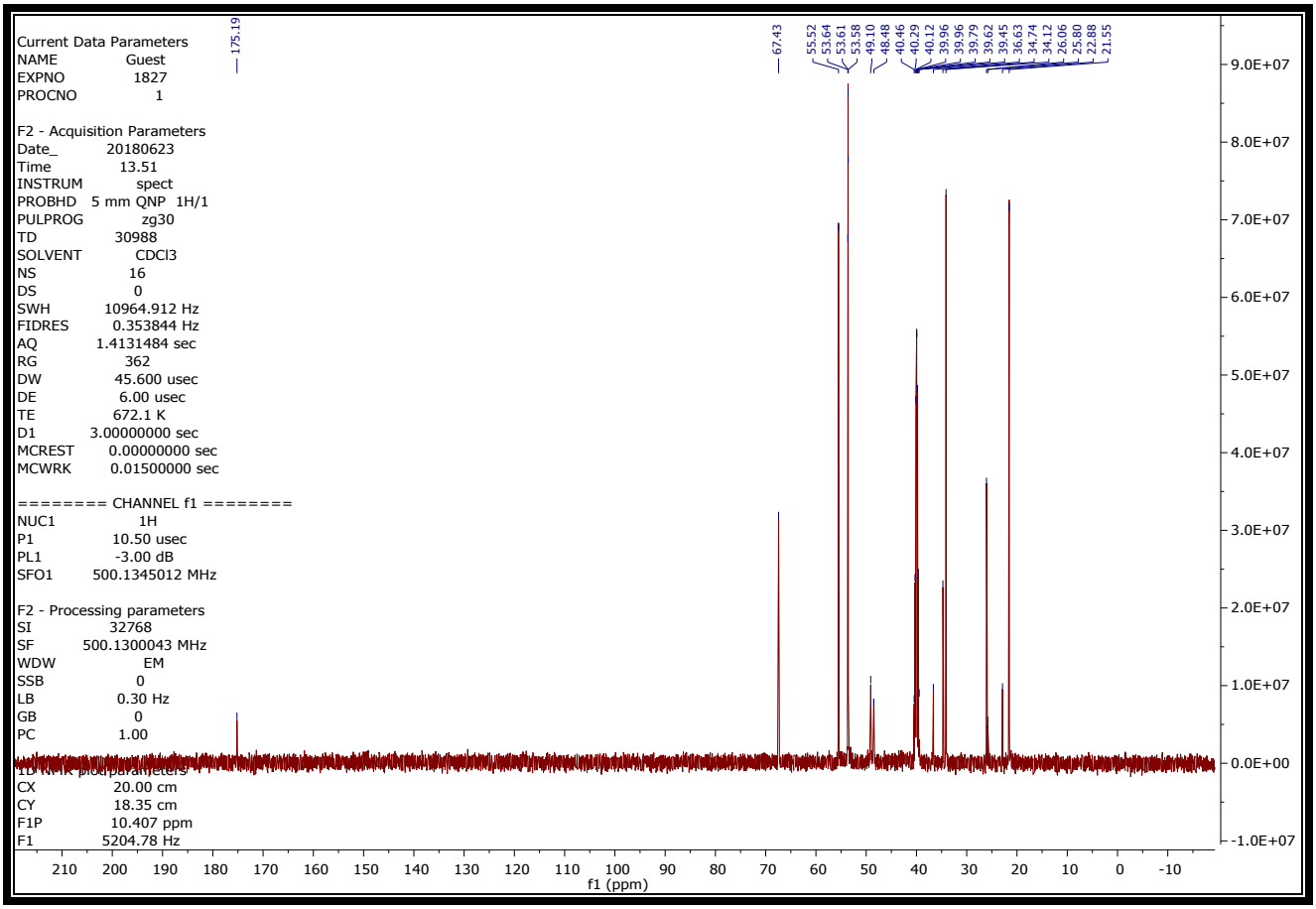
S 1: FT-IR spectrum of [Ch]Cl:Gab NADES.



S 2: Mass spectrum of the [Ch]Cl:Gab NADES.



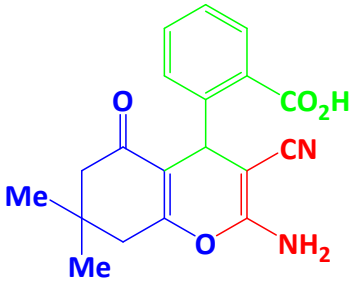
S 3: ^{1}H NMR spectrum of the [Ch]Cl:Gab NADES.

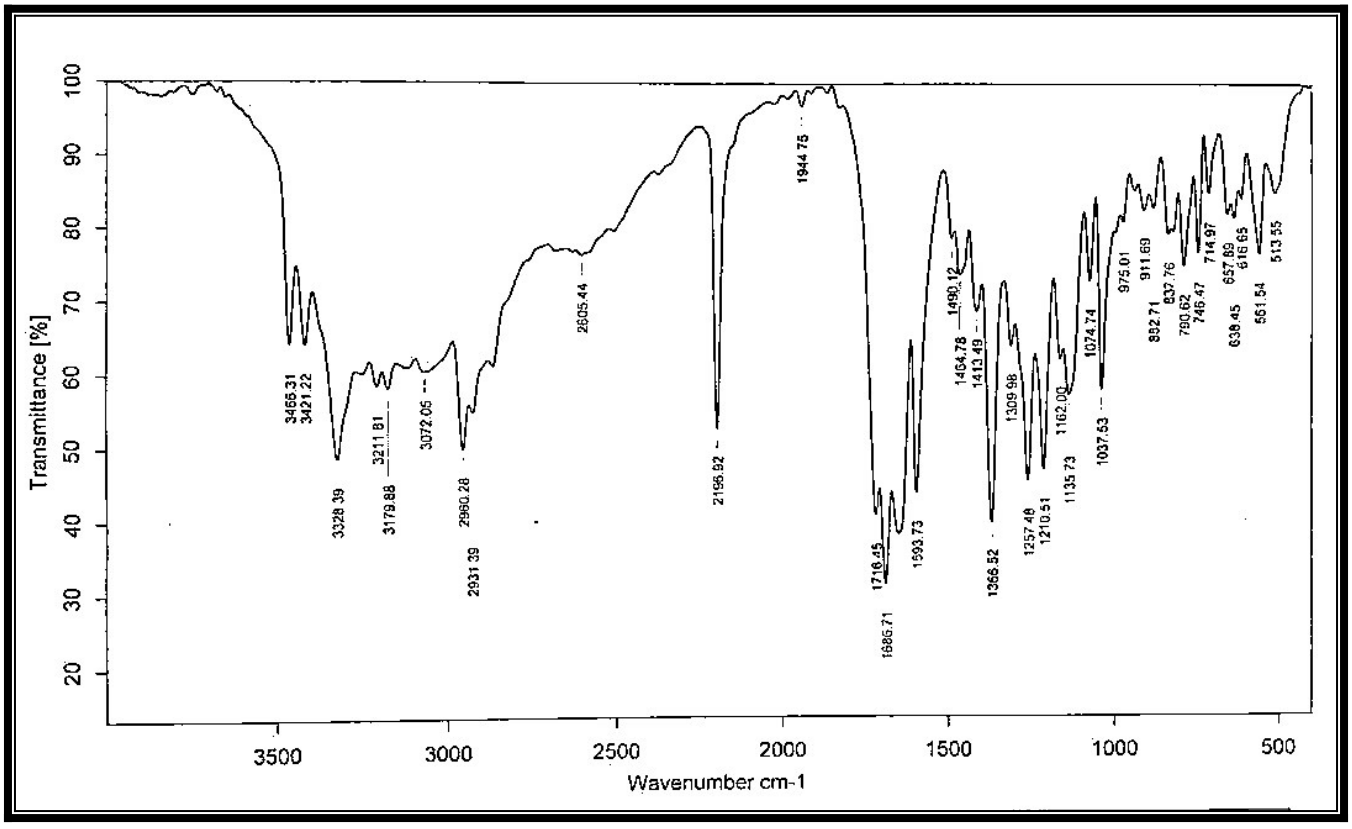


S 4: ^{13}C NMR spectrum of the [Ch]Cl:Gab NADES.

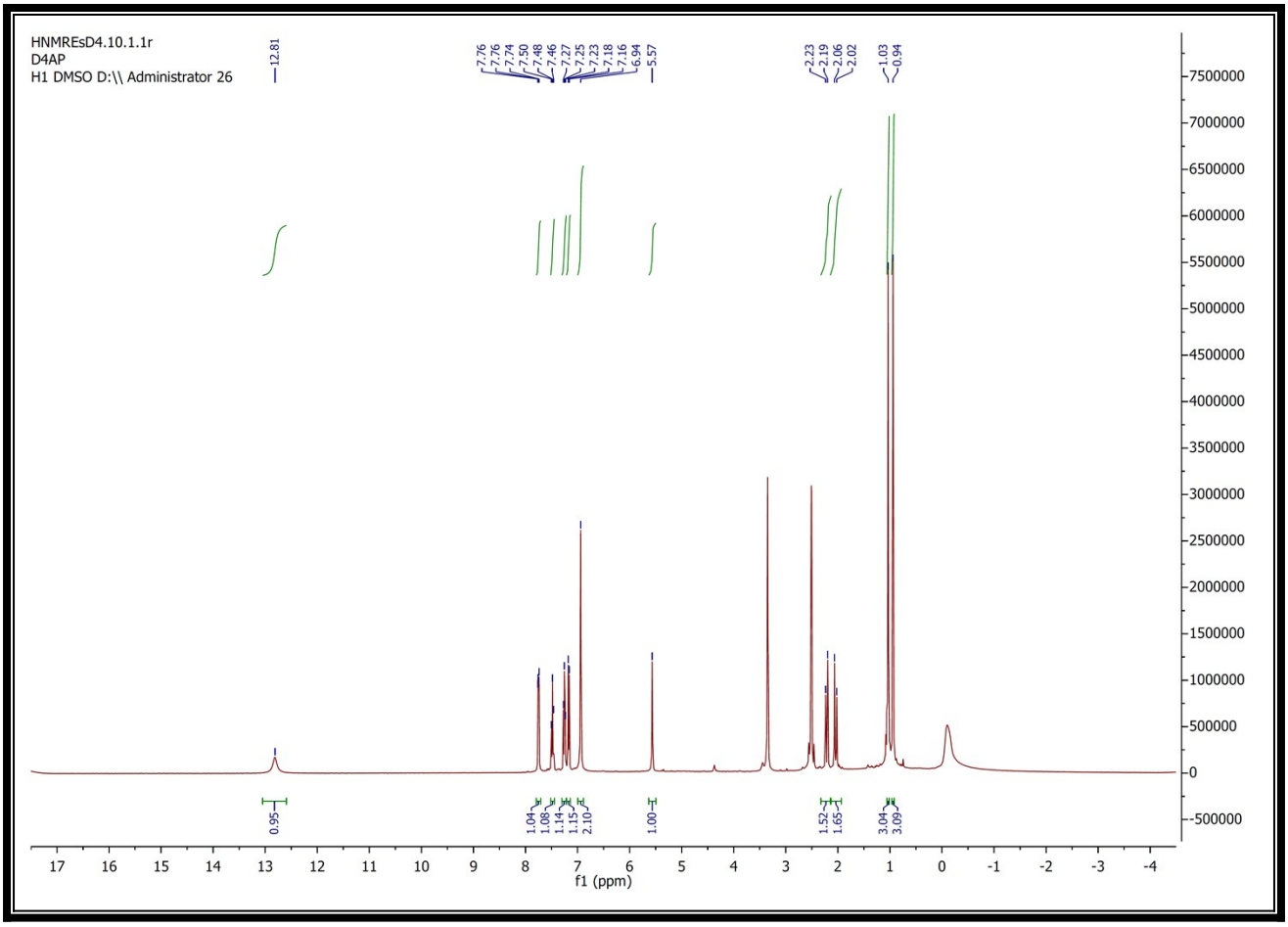
2-(2-Amino-3-cyano-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4H-chromen-4-yl)benzoic acid (4b).

Yellow powder; yield 86%; M.P. 189-192 °C; FT-IR (KBr, *v*, cm^{-1}): 3466, 3421, 3328, 3212, 3179, 2960, 2931, 2196, 1716, 1687, 1594; ^{1}H NMR (400 MHz, DMSO-d_6): δ (ppm) 0.94 (s, 3H, $-CH_3$), 1.03 (s, 3H, $-CH_3$), 2.04 (d, *J*=16 Hz, 2H, $-CH_2$), 2.21 (d, *J*=16 Hz, 2H, $-CH_2$), 5.57 (s, 1H, -CH), 6.94 (brs, 2H, NH_2), 7.17 (d, *J*=8 Hz, 1H, CH-Py), 7.25 (t, *J*=8 Hz, 1H, CH-Py), 7.48 (t, *J*=8 Hz, 1H, CH-Py), 7.75 (d, *J*=8 Hz, 1H, CH-Py), 12.81 (brs, 1H, CO_2H) ; ^{13}C NMR (100 MHz, DMSO-d_6): δ (ppm) 27.3 & 28.7, 30.2, 32.3, 39.9, 50.3, 58.8, 113.7, 119.8, 126.6, 129.2, 130.1, 130.7, 132.3, 146.7, 159.1, 162.8, 168.8, 196.1.

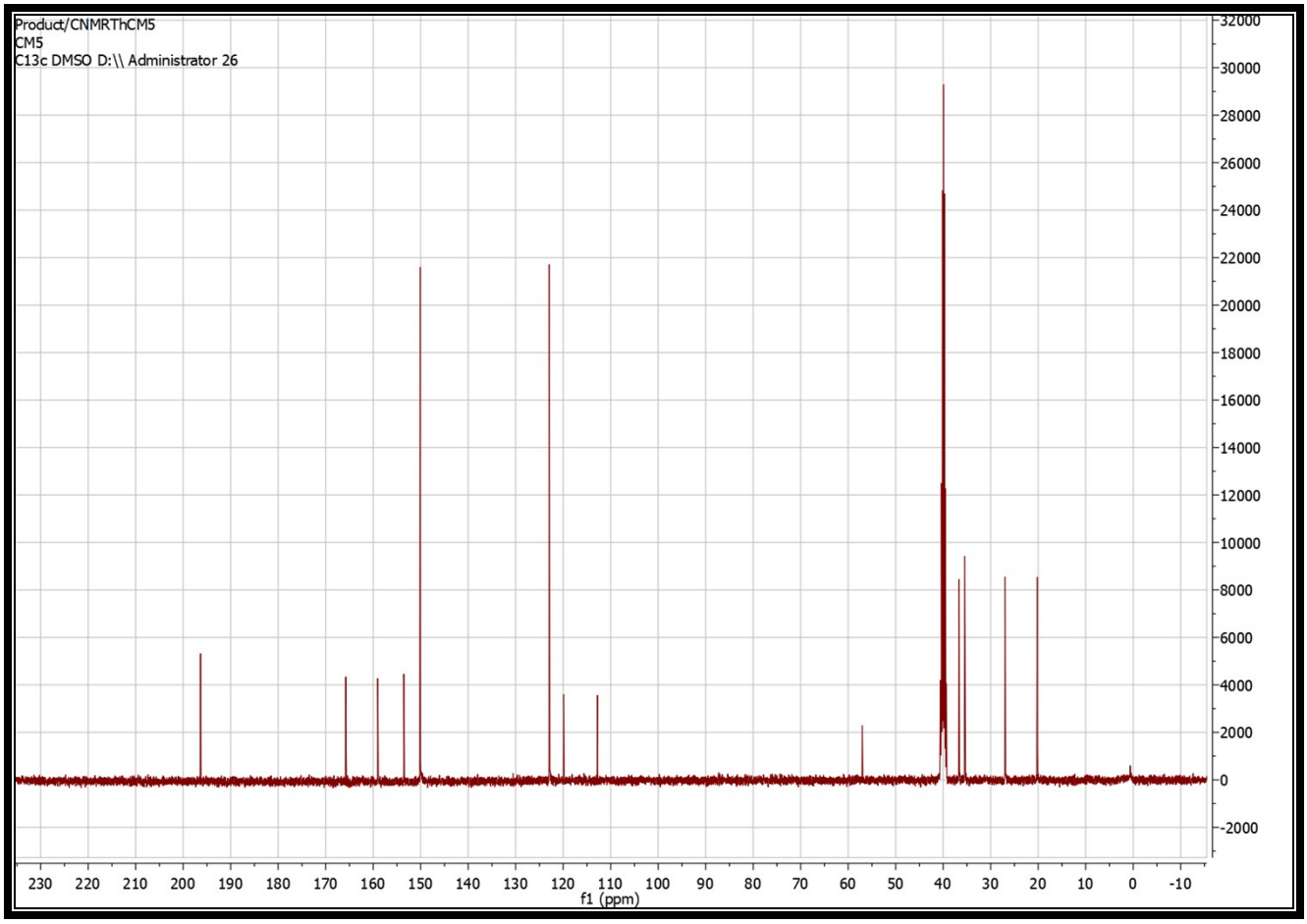




S 5: FT-IR spectrum of 2-(2-Amino-3-cyano-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4H-chromen-4-yl)benzoic acid (4b).



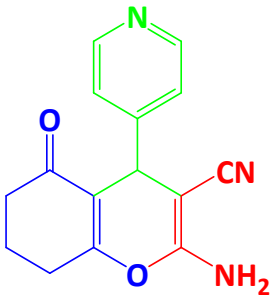
S 6: ^{1}H NMR spectrum of 2-(2-Amino-3-cyano-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4H-chromen-4-yl)benzoic acid (4b).

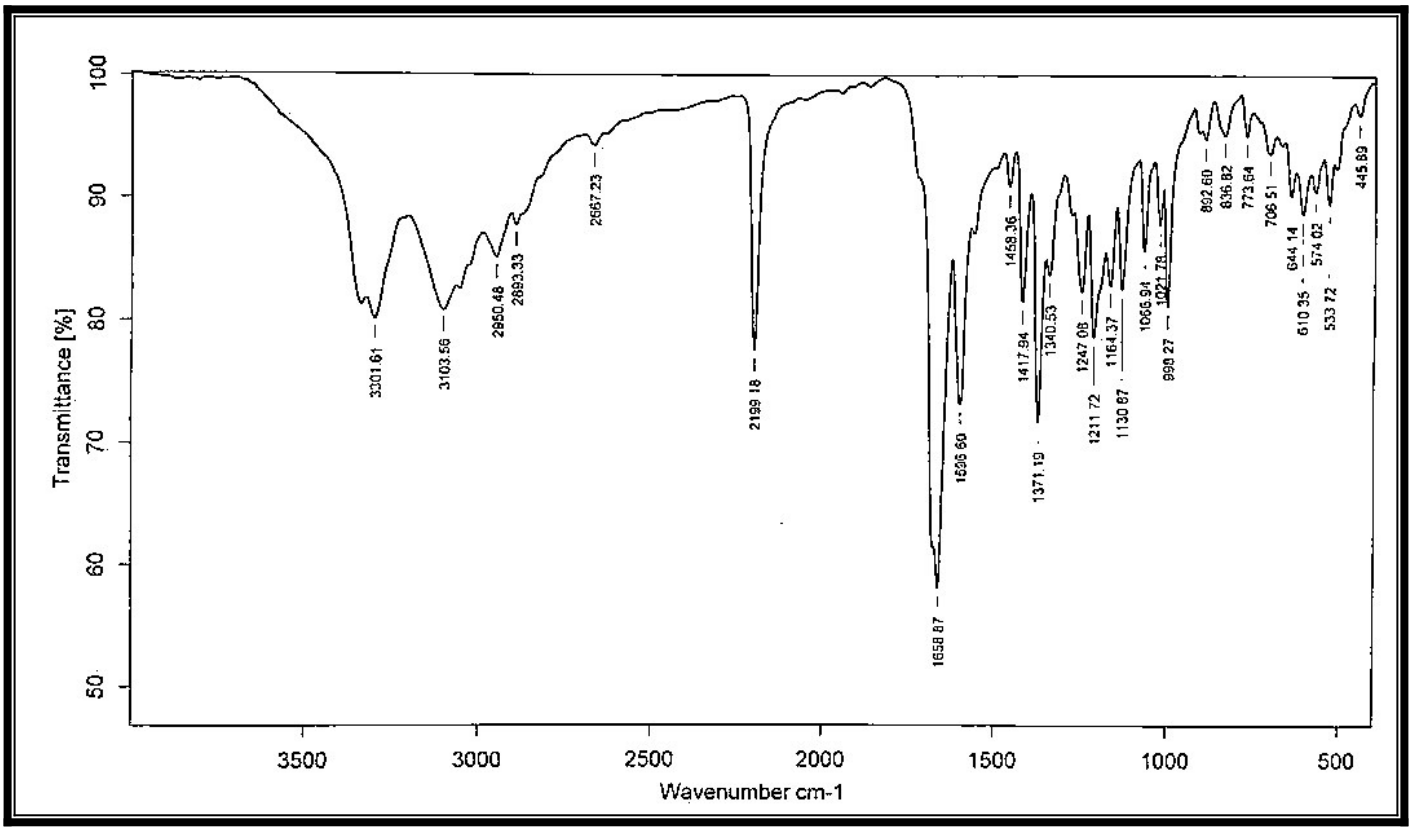


S 7: ^{13}C NMR spectrum of 2-(2-Amino-3-cyano-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4H-chromen-4-yl)benzoic acid (4b).

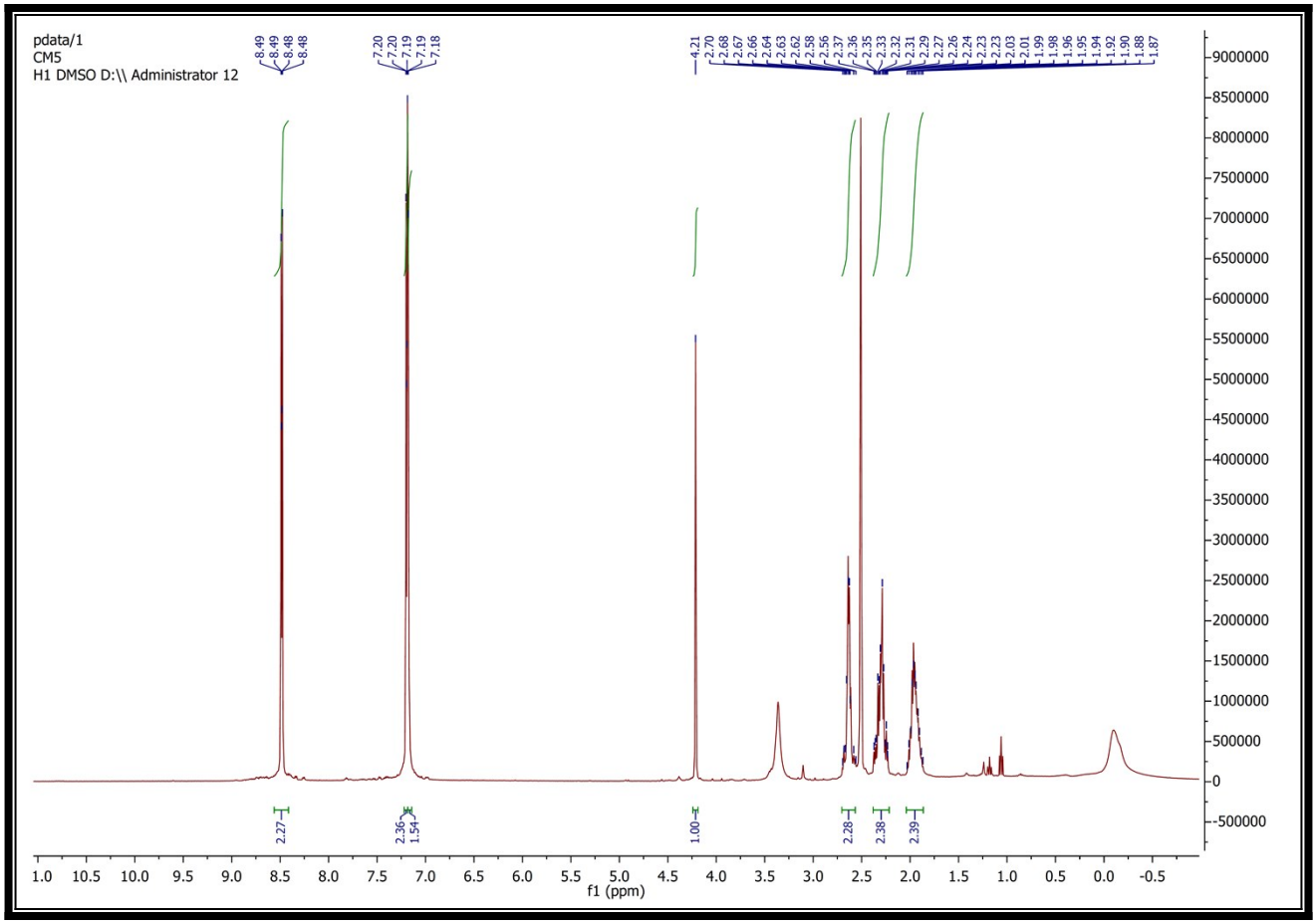
***2-Amino-5-oxo-4-(pyridin-4-yl)-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile (4l*)**.

White powder; yield 88%; M.P. 228-231 °C; FT-IR (KBr, *v*, cm^{-1}): 3301, 33103, 2950, 2893, 2199, 1659, 1597; ^{1}H NMR (400 MHz, DMSO-d_6): δ (ppm) 1.96 (m, 2H, -CH_2), 2.29 (m, 2H, -CH_2), 2.64 (m, 2H, -CH_2), 4.21(s, 1H, -CH), 7.18 (brs, 2H, NH_2), 7.20 (d, J_1=4.6 Hz, J_2=1.6 Hz 2H, CH-Py), 8.49 (d, J_1=4.4 Hz, J_2=1.6 Hz, 2H, CH-Py); ^{13}C NMR (100 MHz, DMSO-d_6): δ (ppm) 20.2, 26.9, 35.4, 36.6, 57.0, 112.8, 119.8, 122.9, 150.1, 153.5, 159.0, 165.7, 196.3.

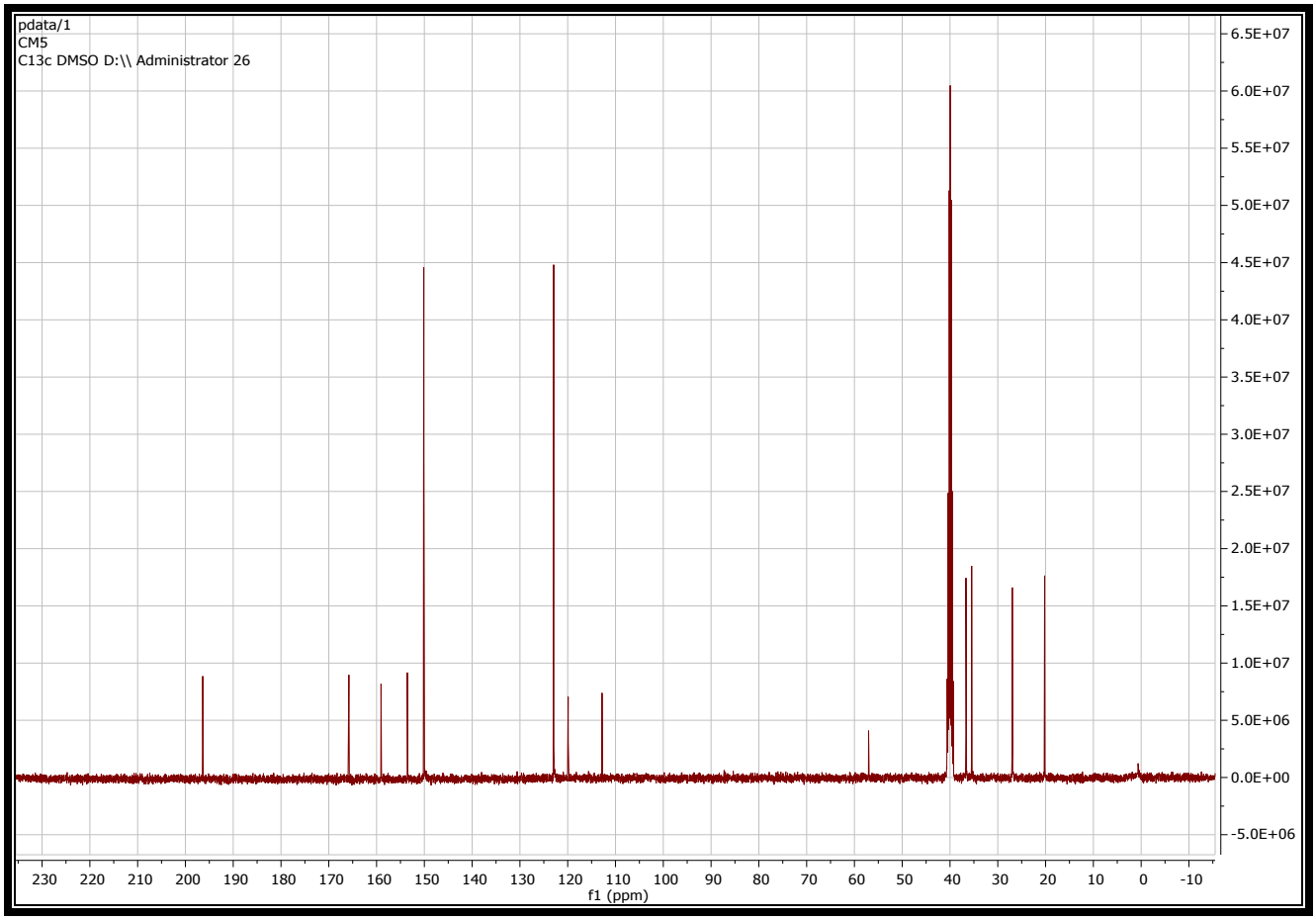




S 8: FT-IR spectrum of 2-Amino-5-oxo-4-(pyridin-4-yl)-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile (4l).



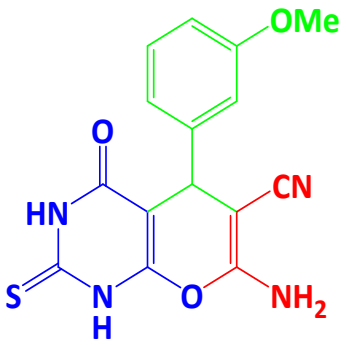
S 9: ^{1}H-NMR spectrum of 2-Amino-5-oxo-4-(pyridin-4-yl)-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile (4l).

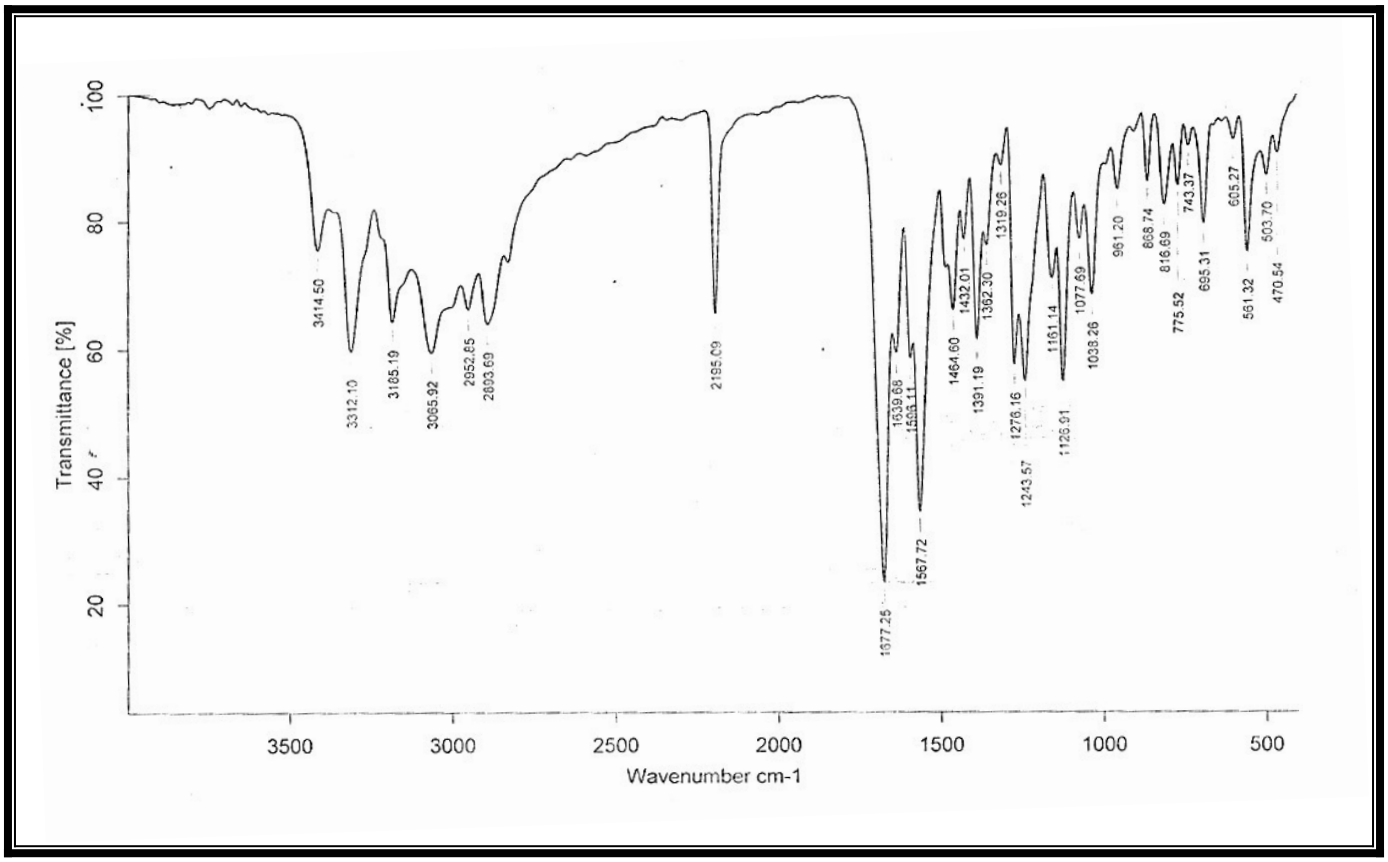


S 10: ^{13}C-NMR spectrum of 2-Amino-5-oxo-4-(pyridin-4-yl)-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile (4l).

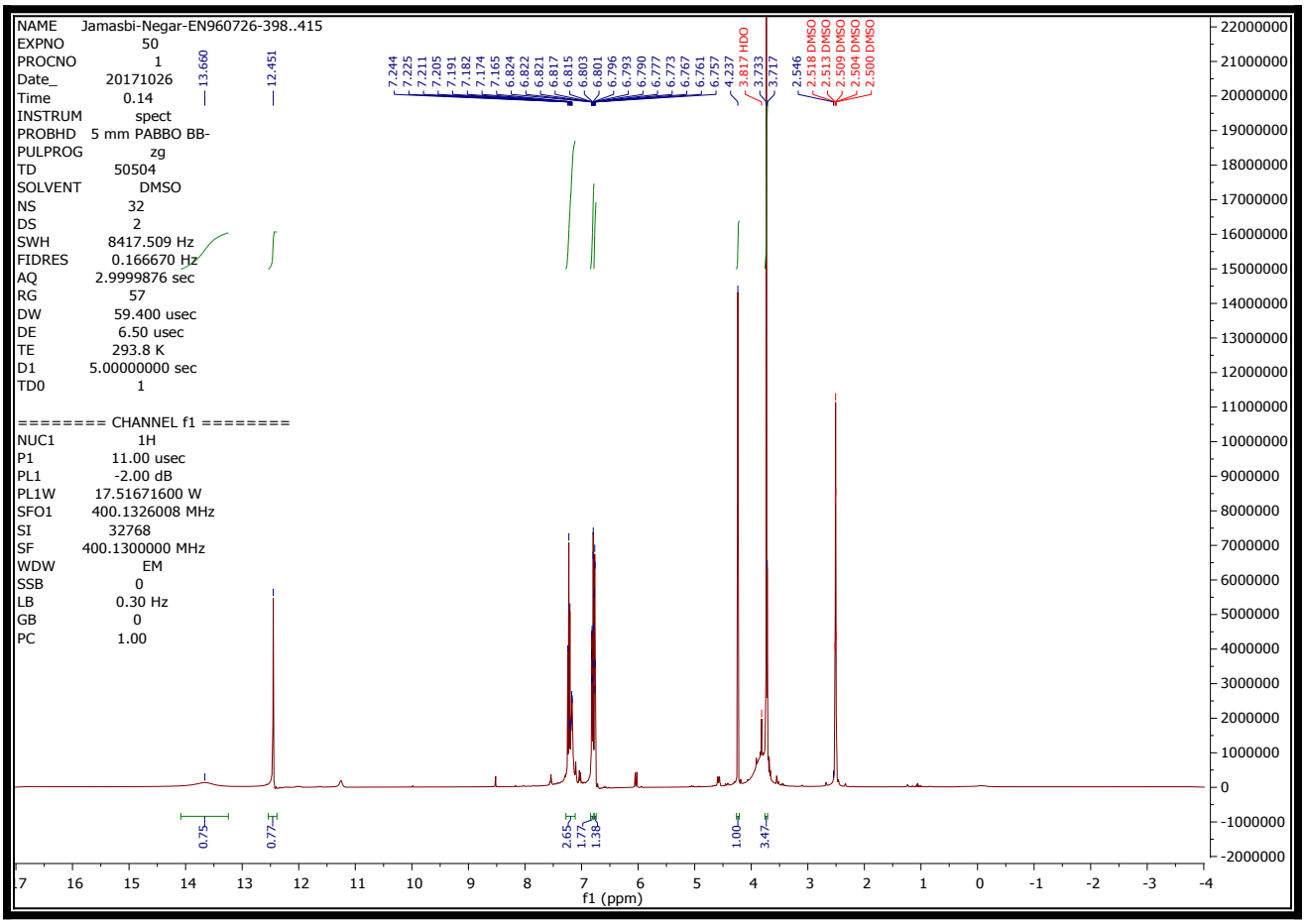
7-Amino-5-(3-methoxyphenyl)-4-oxo-2-thioxo-1,3,4,5-tetrahydro-2H-pyrano[2,3-d]pyrimidine-6-carbonitrile (4x)

White powder; yield 88%; M.P. 235-238 ^{o}C; FT-IR (KBr, *v*, cm^{-1}): 3414, 3312, 3185, 3065, 2195, 1677, 1567; ^{1}H NMR (400 MHz, DMSO-d_6): δ (ppm) 3.73 (s, 3H, CH_3), 4.23 (s, 1H, -CH), 6.76 (t, *J* = 2.4 Hz, 1H, CH-Ph), 6.81 (dd, J_1=8.4 Hz, J_2= 2.4 Hz, 2H, CH-Ph), 7.17 (brs, 2H, NH_2), 7.22 (t, *J* = 8 Hz, 1H, CH-Ph), 12.45 (s, 1H, NH), 13.65 (brs, 1H, NH); ^{13}C NMR (100 MHz, DMSO-d_6): δ (ppm) 35.5, 54.9, 93.3, 111.7, 113.6, 118.9, 119.5, 129.4, 145.0, 151.6, 157.3, 159.1, 160.0, 160.1, 173.7.

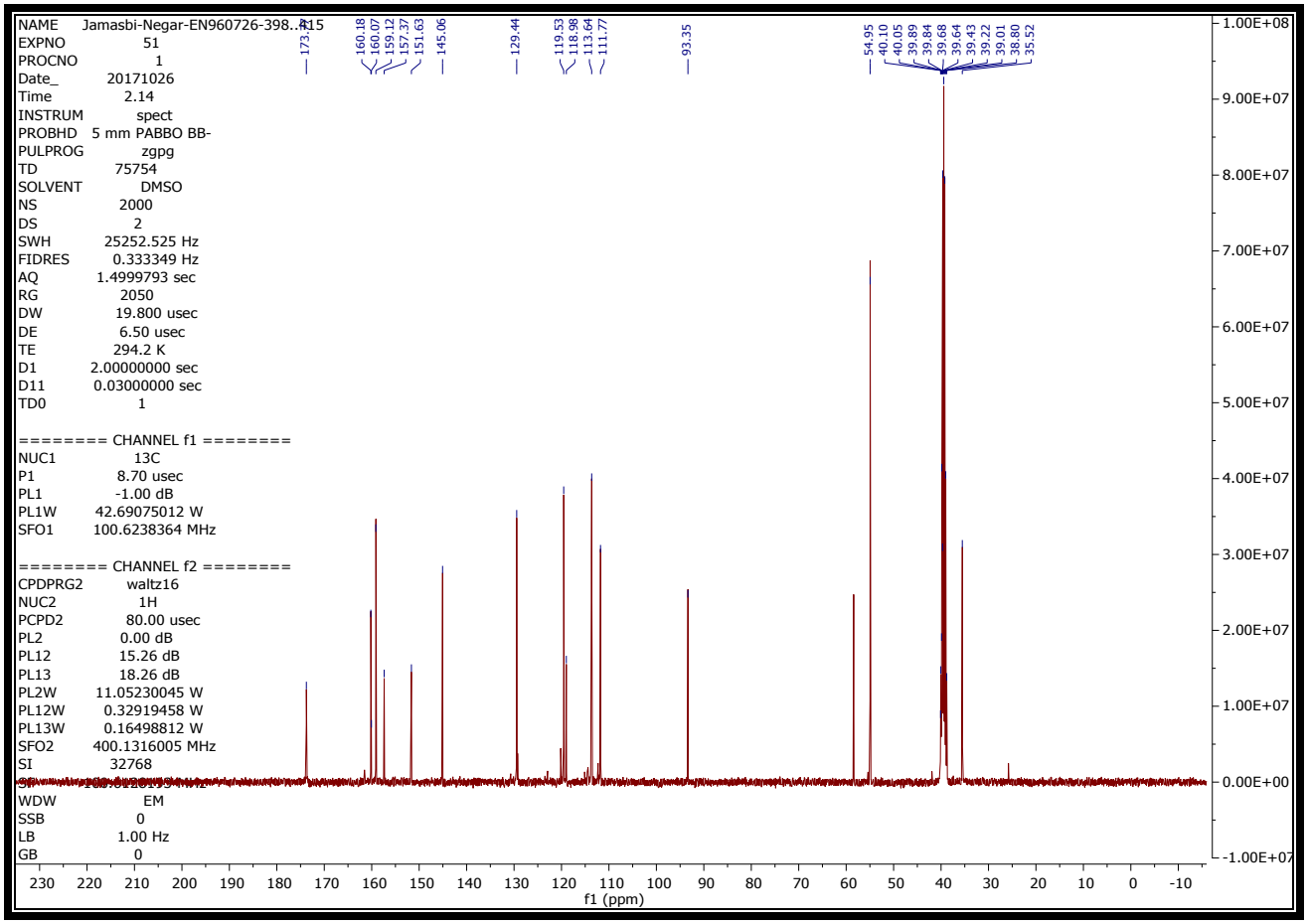




S 11: FT-IR spectrum of 7-Amino-5-(3-methoxyphenyl)-4-oxo-2-thioxo-1,3,4,5-tetrahydro-2H-pyrano[2,3-d]pyrimidine-6-carbonitrile (4x)



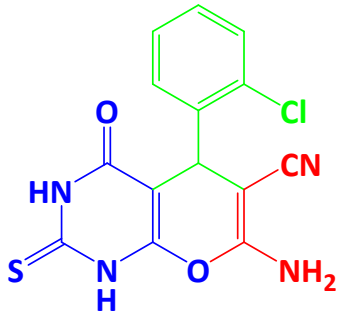
S 12: ^{1}H-NMR spectrum of 7-Amino-5-(3-methoxyphenyl)-4-oxo-2-thioxo-1,3,4,5-tetrahydro-2H-pyrano[2,3-d]pyrimidine-6-carbonitrile (4x)

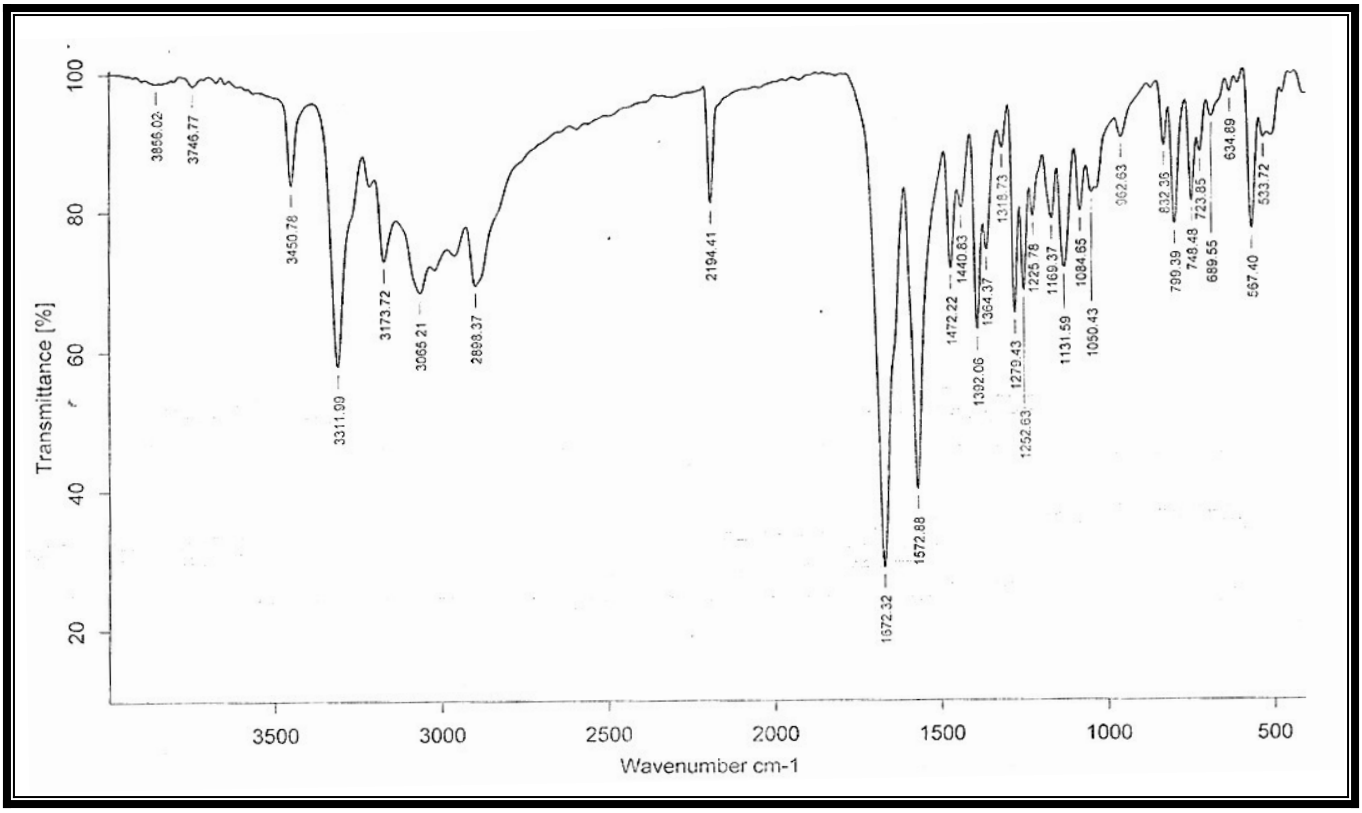


S 13: ^{13}C-NMR spectrum of 7-Amino-5-(3-methoxyphenyl)-4-oxo-2-thioxo-1,3,4,5-tetrahydro-2H-pyrano[2,3-d]pyrimidine-6-carbonitrile (4x).

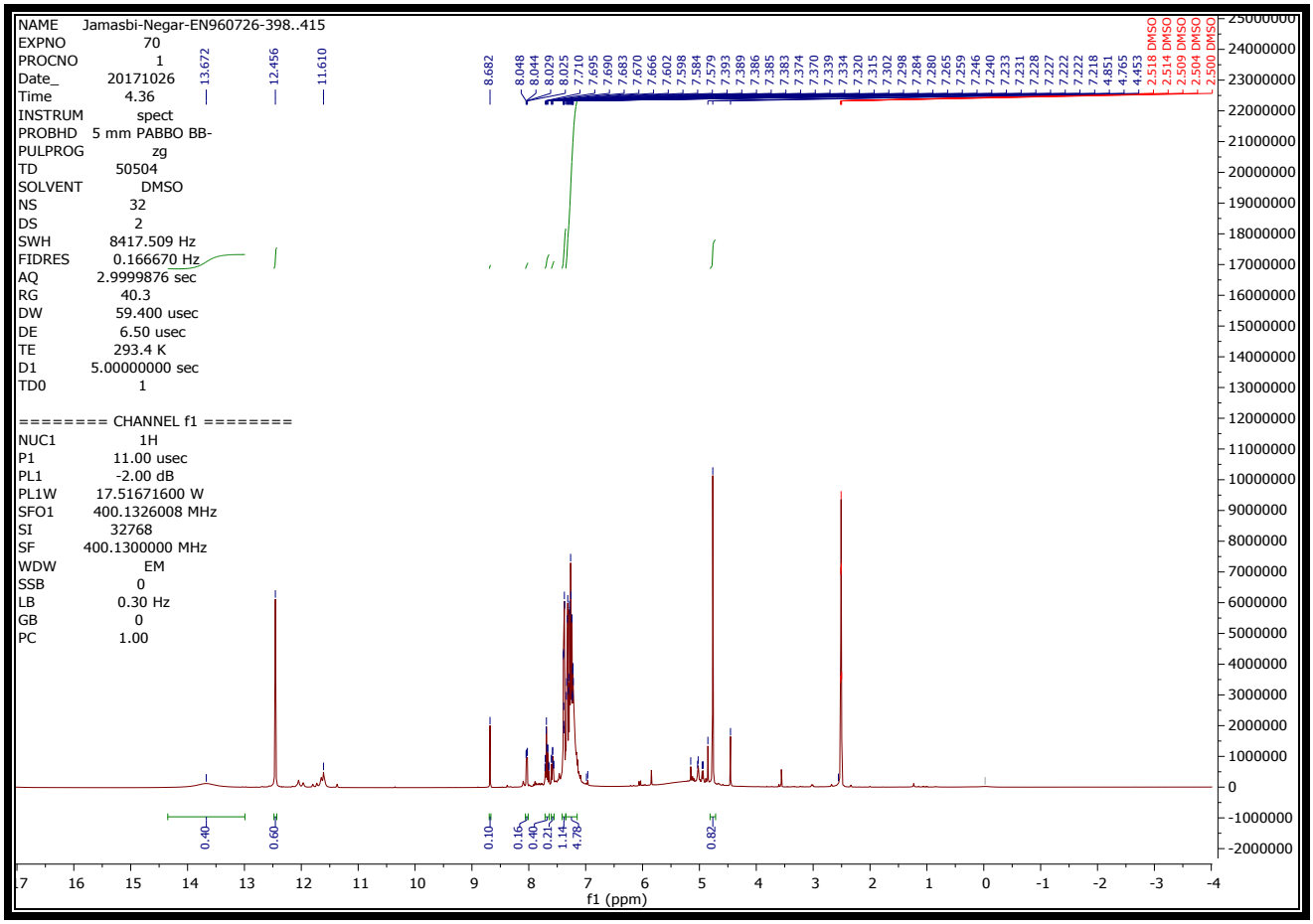
7-Amino-5-(2-chlorophenyl)-4-oxo-2-thioxo-1,3,4,5-tetrahydro-2H-pyrano[2,3-d]pyrimidine-6-carbonitrile (4y).

White powder; yield 89%; M.P. 240 °C (Dec.);FT-IR (KBr, *v*, cm^{-1}): 3450, 3311, 3173, 3065, 2194, 1672, 1572; ^{1}H NMR (400 MHz, DMSO-d_6): δ (ppm) 4.76 (s, 1H, -CH), 7.33-7.21 (m, 5H, CH-Ph and NH_2), 7.38 (dd, J_1= 9.2 Hz, J_2= 1.6 Hz, 1H, CH-Ph) 12.45 (s, 1H, NH), 13.67 (brs, 1H, NH); ^{13}C NMR (100 MHz, DMSO-d_6): δ (ppm) 56.93, 92.52, 118.5, 127.4, 127.9, 128.5, 129.4, 130.3, 132.2, 134.8, 152.1, 157.6, 157.9, 160.0, 173.8.

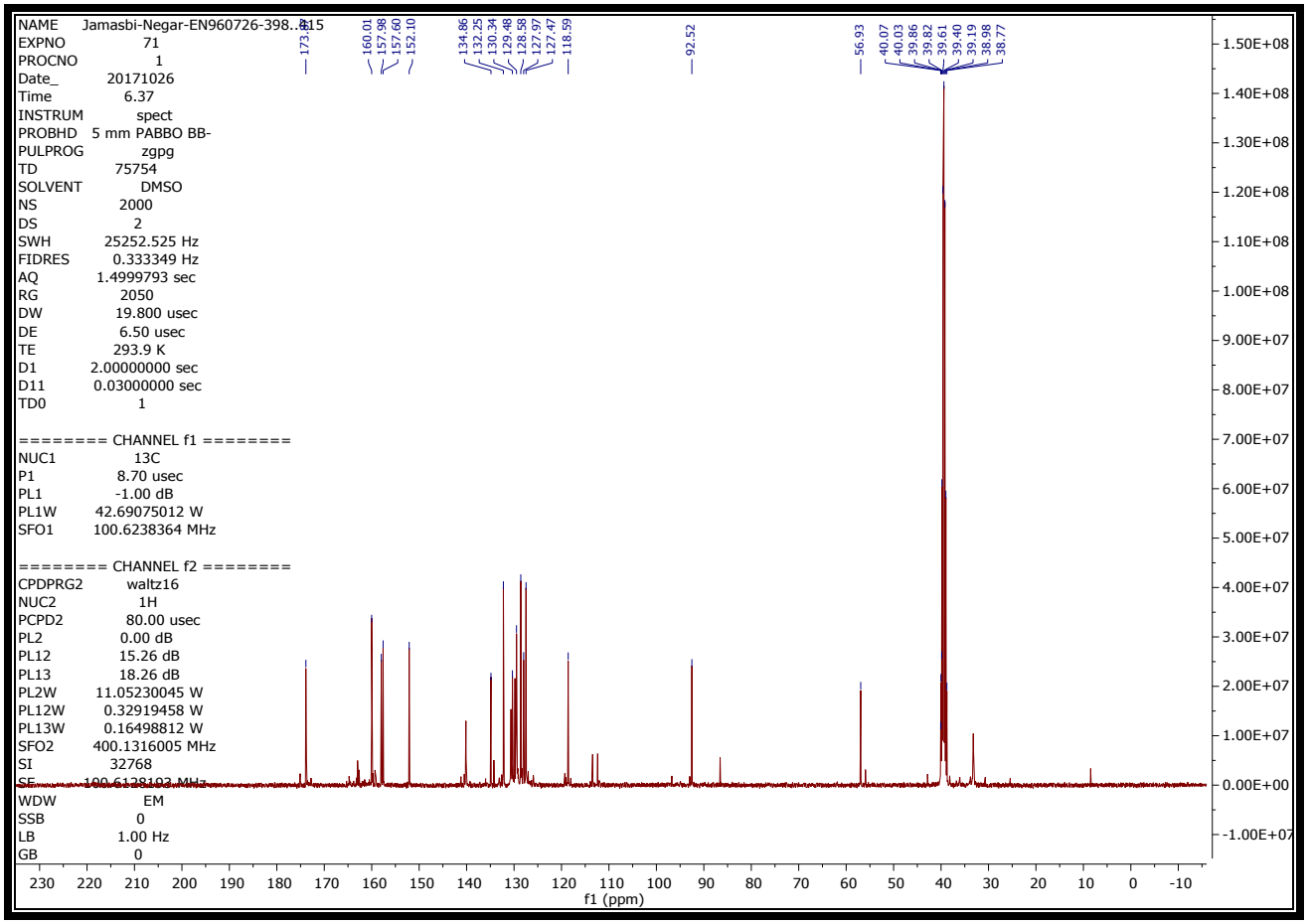




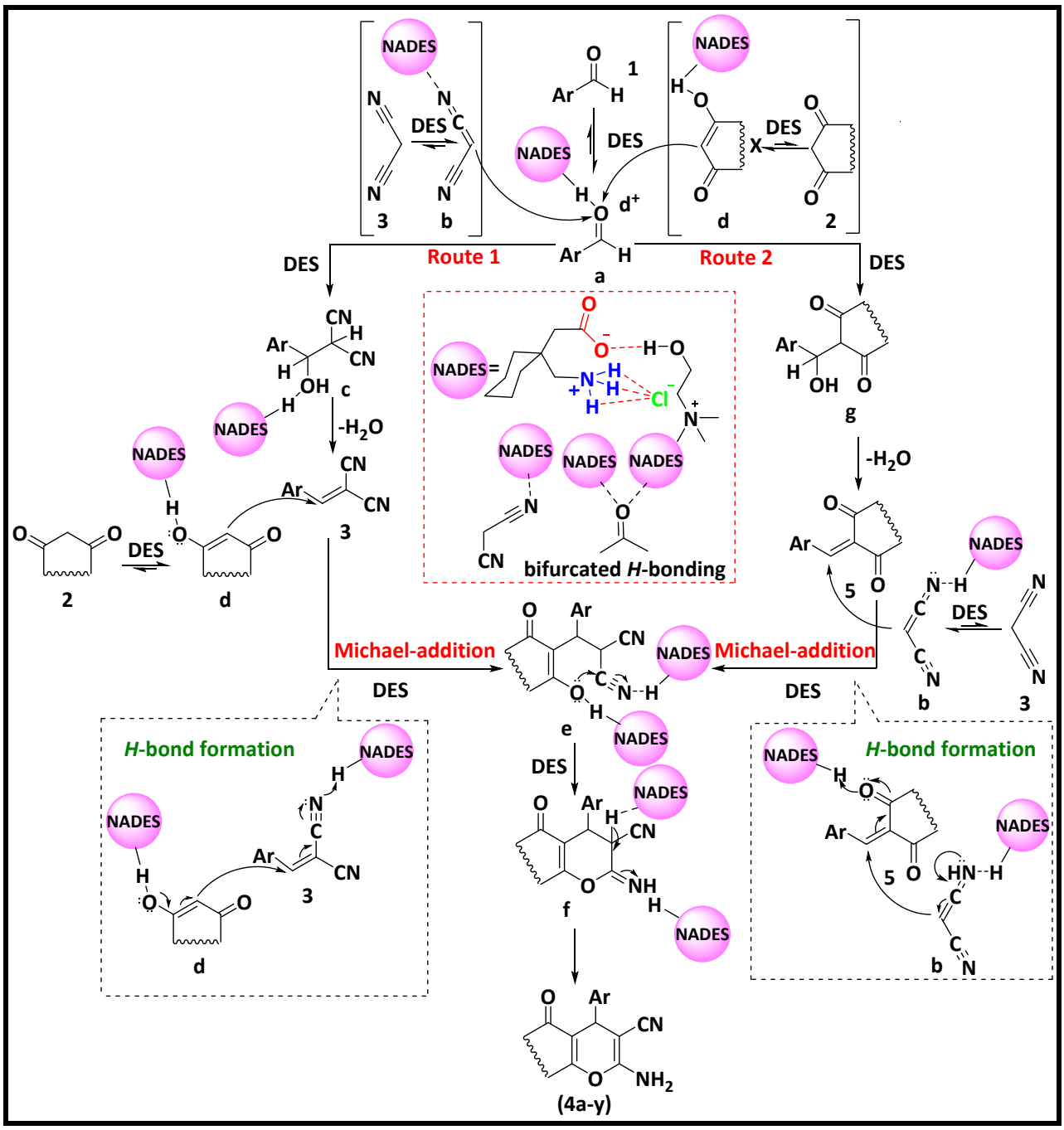
S 14: FT-IR spectrum of 7-Amino-5-(2-chlorophenyl)-4-oxo-2-thioxo-1,3,4,5-tetrahydro-2H-pyrano[2,3-d]pyrimidine-6-carbonitrile (4y).



S 15: ^{1}H-NMR spectrum of 7-Amino-5-(2-chlorophenyl)-4-oxo-2-thioxo-1,3,4,5-tetrahydro-2H-pyrano[2,3-d]pyrimidine-6-carbonitrile (4y).



S 16: ^{13}C-NMR spectrum of 7-Amino-5-(2-chlorophenyl)-4-oxo-2-thioxo-1,3,4,5-tetrahydro-2H-pyrano[2,3-d]pyrimidine-6-carbonitrile (4y).



S 17: Plausible mechanism for the synthesis of 4*H*-pyran derivatives in the presence of [Ch]Cl:Gab NADES.