Facile Synthesis of 2-Anilino Nicotinic Acids via Catalysis Cu-Mg-Al LDO Catalysed Amination of 2-Chloronicotinic acid

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1. Experimental

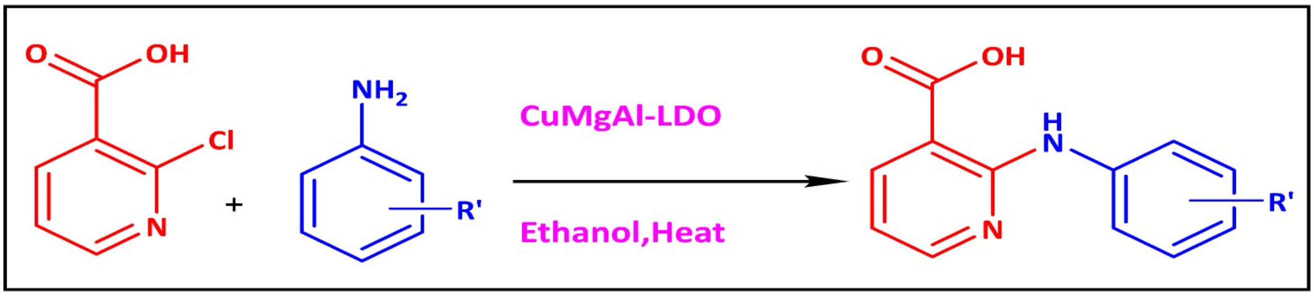
All the reagents used for the reactions were analytical grade with a high degree of purity. The metal salts (Mg $(NO_3)_2·6H_2O$, Al $(NO_3)_3·9H_2O$, and Cu $(NO_3)_2·3H_2O$) of were procured from Avra synthesis private. Ltd. Mumbai. Sodium hydroxide and sodium carbonate were purchased from S.D. Fine Chem. Ltd. Mumbai. The progress of each reaction was monitored by thin-layer chromatography (TLC) using 0.25 mm E-Merck silica gel 60 F-254 pre-coated plates and visualized under UV light (254 nm). The proton (^{1}H) NMR spectra were recorded on an 'Agilent 500 MHz spectrometer' with a suitable solvent (CDCl3 & DMSO-D6-*d6*), and chemical shifts are expressed in δ ppm using Tetramethylsilane (TMS) as an internal standard. The JASCO FT/IR-4100 spectrophotometer was used to record IR frequency graphs using the ATR technique. All melting points are uncorrected and are presented in degrees Celsius.

2. General procedure

2.1 Procedure for catalyst preparation:

The Cu-Mg-Al LDO material with a molar ratio of 5/62/33 was prepared by co-precipitation. The aqueous solutions of the metal nitrates (Mg $(NO_3)_2·6H_2O$, Al $(NO_3)_3·9H_2O$, and Cu $(NO_3)_2·3H_2O$) of desired concentration were added to the solution containing 0.75 M Na_2CO_3 and 0.1 M NaOH under vigorous stirring. The pH of the mixture was kept steady at 10 while mixing by adding NaOH solution. The slurry obtained was aged for 3 hours at 60 °C. After the room temperature, the precipitate was filtered and washed until the pH was 7. The catalyst was then dried overnight at 120 °C. Finally, it was calcined at 600°C for 12 hours. [1,2]

2.2 General procedure for preparation of 2-Anilino nicotinic acid derivatives:



Reaction condition: 2-chloronicotinic acid (1.0 mmol), aniline (1.5 mmol), Solvent (7.8 mL), 23.6 mg Catalyst mass (15 wt.%), Temperature (80°C). [b] Isolated yield.

The round-bottom glass flask with a double surface condenser was charged with a mixture of ethanol (7.8 mL) and Cu-Mg-Al LDO (15 wt.%). The reaction mixture was stirred using a magnetic stirrer for 2 minutes. 2-chloro nicotinic acid (1.0 mmol) and aniline derivative (1.5 mmol) were added to the solution of Cu-Mg-Al LDO in ethanol. The reaction mixture was heated to reflux (Reflux reaction mass) and stirred at 200 rpm until complete conversion was observed. Thin-layer chromatography (TLC) was used to track the reaction's progress with mobile phase dichloromethane: methanol (9.5:0.5). TLC was visualized under UV light at 254 nm wavelength. After complete conversion, the heating was removed, the reaction mixture was filtered, and the filtrate was cooled to 5-10°C. After precipitation, the pure product was isolated through filtration. Some of the isolated solids were purified in ethanol. Yields obtained ranged from 20 % to 92 % for various products.

3. Catalyst Characterizations.

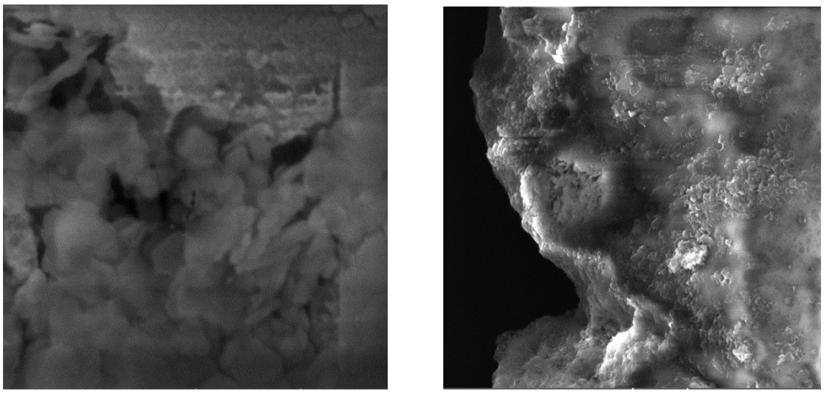
The physicochemical properties of the catalyst are presented in Table S1.

Table S1. Characteristics of the catalyst

BET surface area (m^2/g)[a]	Pore volume (cm^3/g)[b]	Pore size (nm)[c]	Metal content (mol%)	Metal content (wt. %)
33.5	0.0512	6.1	Cu=5.2, Mg= 62.7, Al=32.1	Cu=7.3, Mg= 33.4, Al=18.9,

[a] determined from the BET equation, [b] obtained from N_2 absorption-desorption curve, [c] estimated from the BJH pore size distribution,

SEM images of the fresh catalyst are shown in Figure S1. The porous nature of the catalyst is quite evident. The elemental composition of the metal is shown in Table S1. The SEM EDX profile of the fresh catalyst is shown in Figure S2.



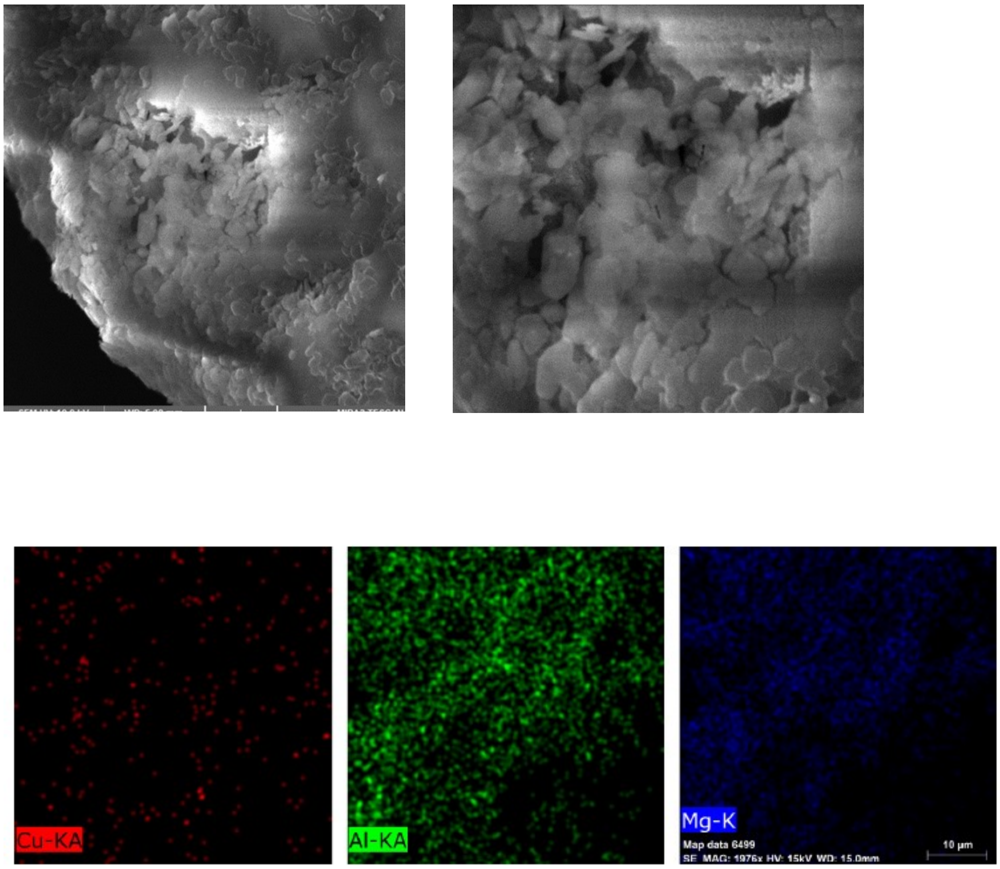


Figure S1. SEM-EDX images of the fresh catalyst

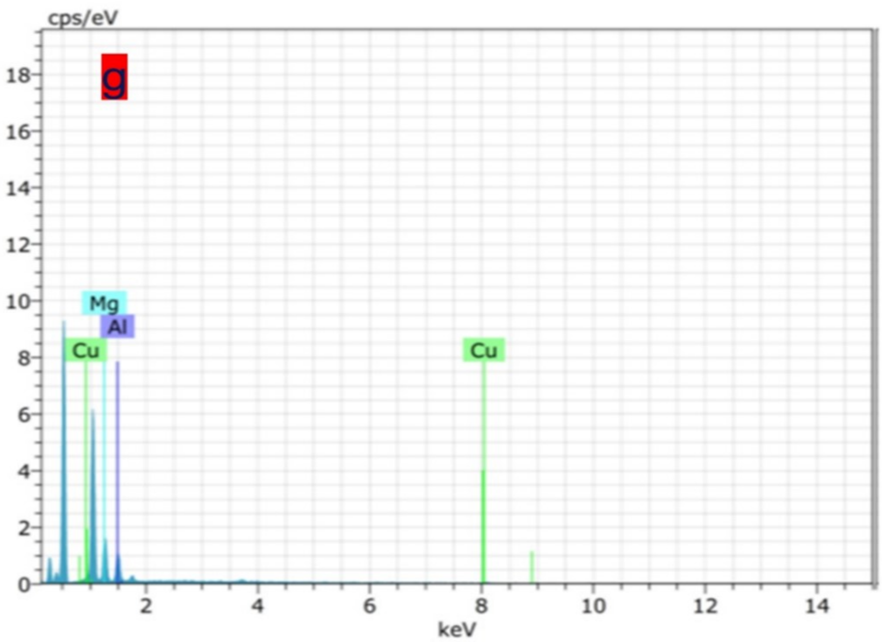
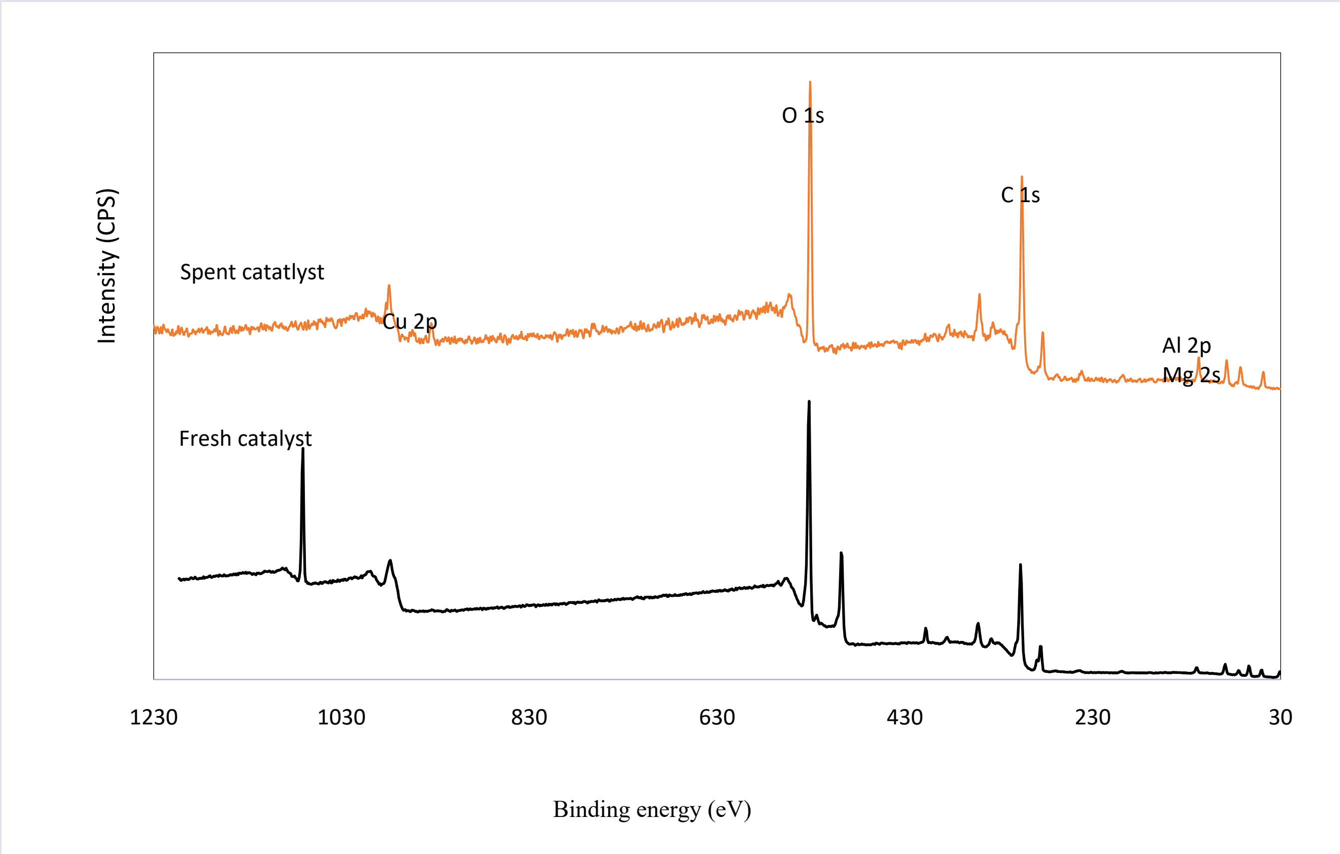
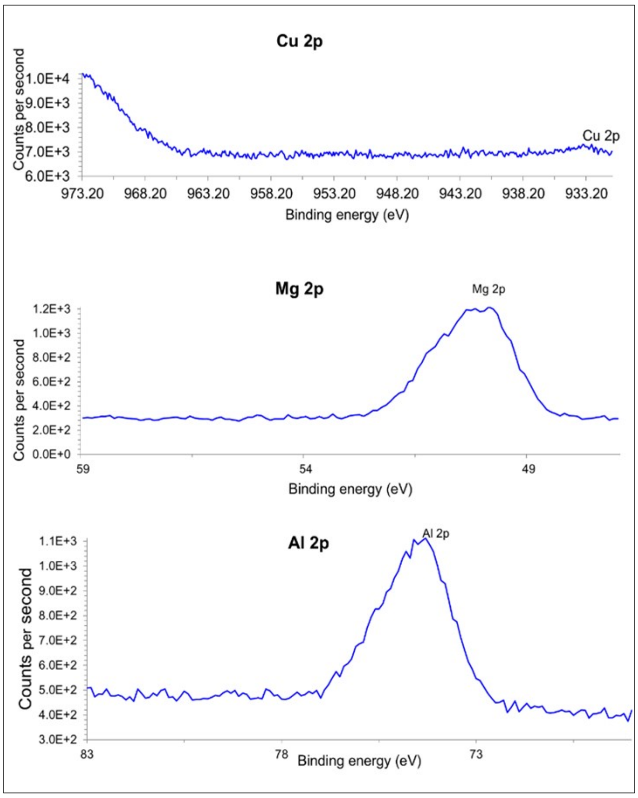


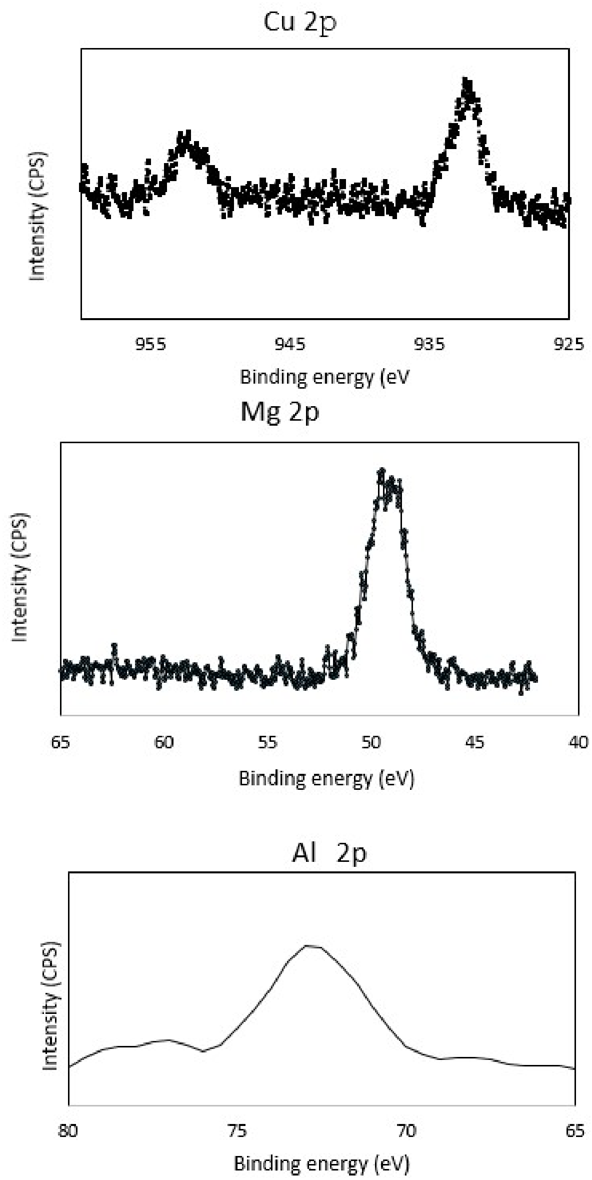
Figure S2. SEM-EDX elemental profile


Intensity (CPS)
O 1s
C 1s
Spent catatlyst
Cu 2p
Al 2p
Mg 2s
Fresh catalyst
1230
1030
830
630
430
230
30
Binding energy (eV)

(a)


Cu 2p
Counts per second
1.0E+4
9.0E+3
8.0E+3
7.0E+3
6.0E+3
973.20
968.20
963.20
958.20
953.20
948.20
943.20
938.20
933.20
Binding energy (eV)
Cu 2p
Mg 2p
Mg 2p
1.2E+3
1.0E+3
8.0E+2
6.0E+2
4.0E+2
2.0E+2
0.0E+0
59
54
49
Binding energy (eV)
Al 2p
Al 2p
1.1E+3
1.0E+3
9.0E+2
8.0E+2
7.0E+2
6.0E+2
5.0E+2
4.0E+2
3.0E+2
83
78
73
Binding energy (eV)

(b)

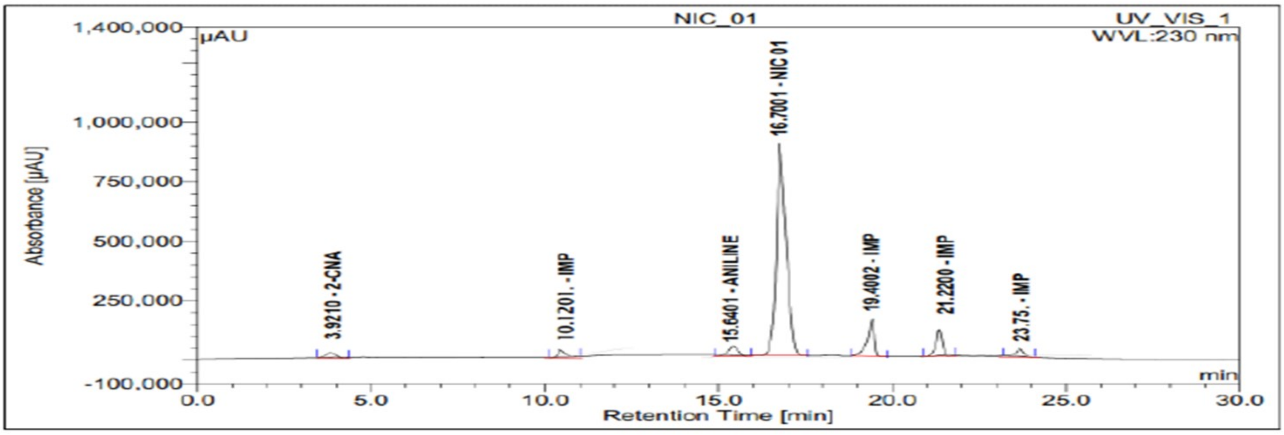


(c)

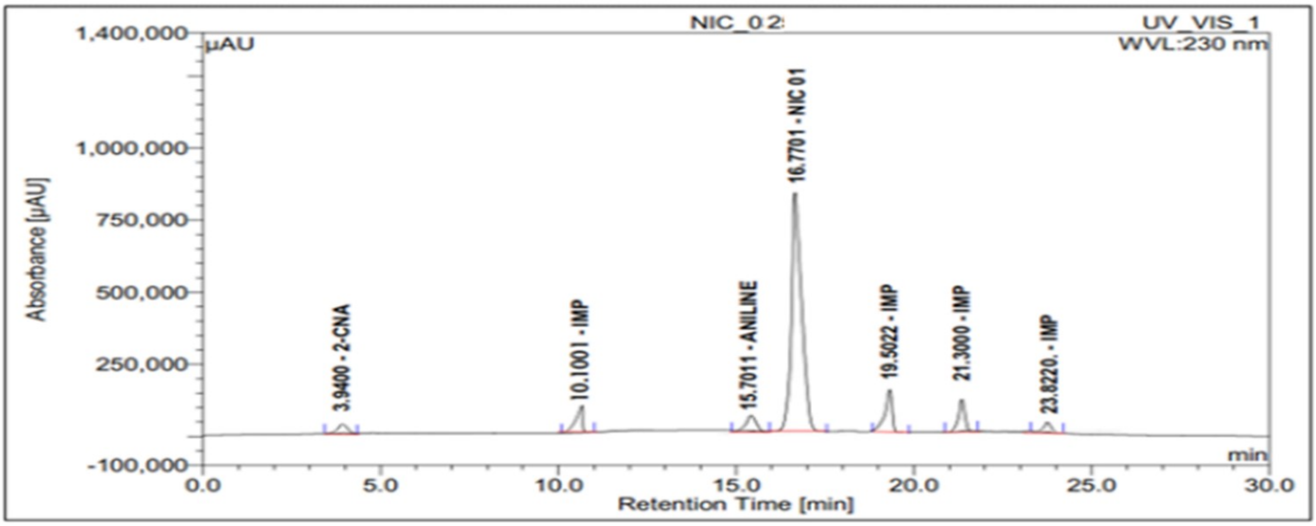
Figure S3. XPS profile for fresh and spent catalyst (a), XPS spectra of Cu 2p, Mg 2p and Al 2p for fresh (b) and spent (c) catalyst

4. Assessment of product purity.

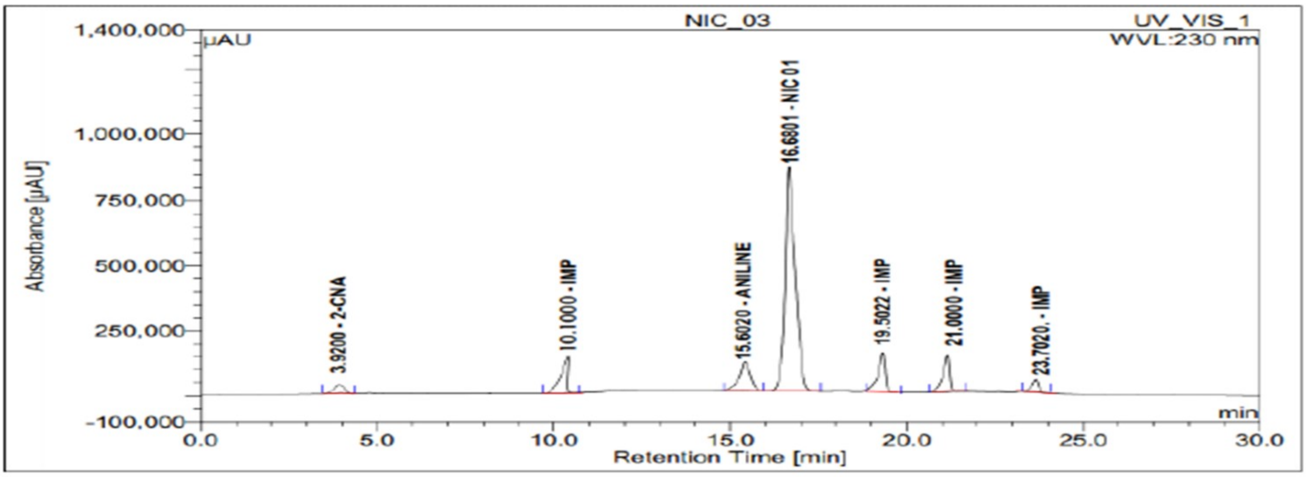
The reusability of the Cu–Mg–Al layered double oxide (LDO) catalyst was evaluated over five consecutive reaction cycles. After each cycle, the reaction product (NIC_01 to NIC_05) was analysed by high-performance liquid chromatography (HPLC) to assess product purity. The representative chromatograms are shown below.



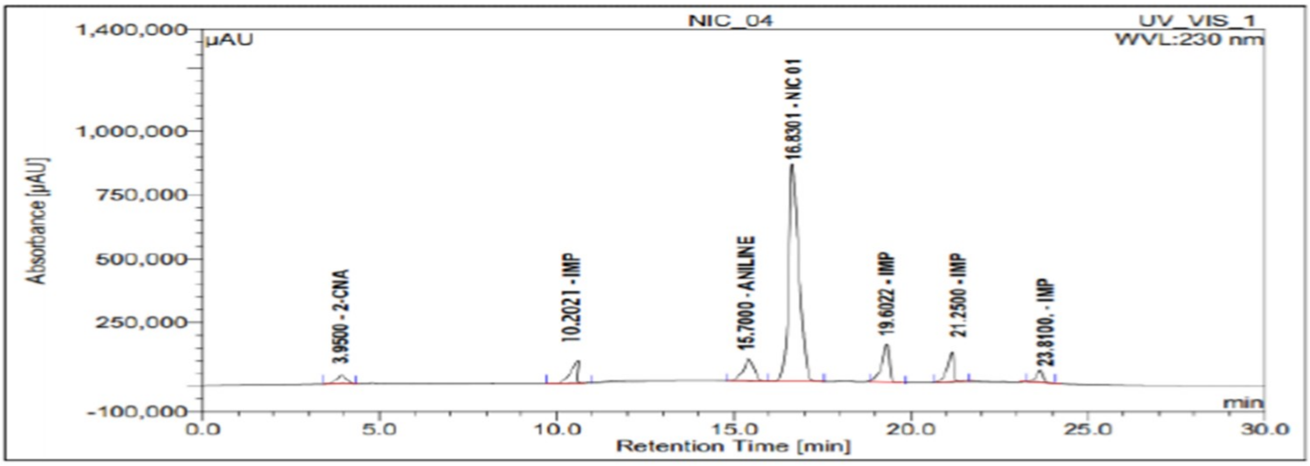
Sr No	Peak Name	RT (min)	Rel. Ret. Time %	Area µAU	Area %
1	2-CNA	3.92	0.23	312.38	0.10
2	Unk	10.12	0.60	2186.67	0.70
3	ANILINE	15.64	0.93	4373.33	1.40
4	NIC	16.70	1.00	292076.24	93.50
5	Unk	19.40	1.16	6560.00	2.10
6	Unk	21.22	1.27	5935.24	1.90
7	Unk	23.75	1.42	937.14	0.30
					100.00



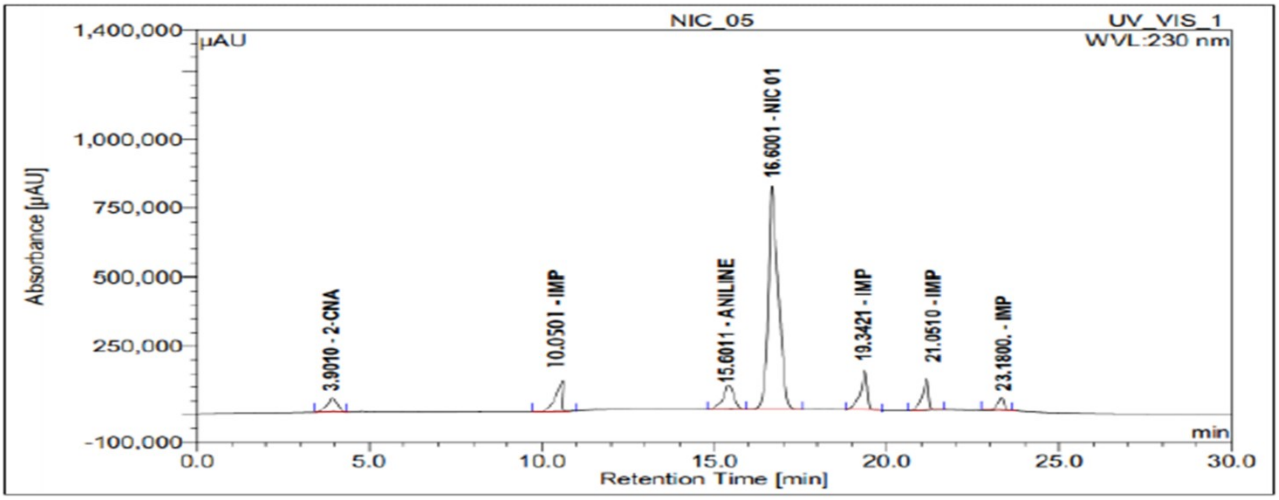
Sr No	Peak Name	RT (min)	Rel. Ret. Time %	Area µAU	Area %
1	2-CNA	3.94	0.23	503.98	0.15
2	Unk	10.10	0.60	4199.84	1.25
3	ANILINE	15.70	0.94	4367.83	1.30
4	NIC	16.77	1.00	308100.08	91.70
5	Unk	19.50	1.16	8903.66	2.65
6	Unk	21.30	1.27	8231.68	2.45
7	Unk	23.82	1.42	1679.94	0.50
					100.00



Sr No	Peak Name	RT (min)	Rel. Ret. Time %	Area μAU	Area %
1	2-CNA	3.92	0.24	569.48	0.17
2	Unk	10.10	0.61	5259.30	1.57
3	ANILINE	15.60	0.94	5560.78	1.66
4	NIC	16.68	1.00	304771.17	90.98
5	Unk	19.35	1.16	9547.13	2.85
6	Unk	21.00	1.26	7202.22	2.15
7	Unk	23.70	1.42	2076.92	0.62
					100.00



Sr No	Peak Name	RT (min)	Rel. Ret. Time %	Area μAU	Area %
1	2-CNA	3.95	0.23	662.58	0.20
2	Unk	10.20	0.61	6957.07	2.10
3	ANILINE	15.70	0.93	6029.46	1.82
4	NIC	16.83	1.00	295012.85	89.05
5	Unk	19.60	1.16	11760.76	3.55
6	Unk	21.25	1.26	8282.23	2.50
7	Unk	23.81	1.41	2584.05	0.78
					100.00



Sr No	Peak Name	RT (min)	Rel. Ret. Time %	Area µAU	Area %
1	2-CNA	3.90	0.23	992.69	0.30
2	Unk	10.05	0.61	7941.53	2.40
3	ANILINE	15.60	0.94	6287.04	1.90
4	NIC	16.60	1.00	290858.46	87.90
5	Unk	19.34	1.17	12805.71	3.87
6	Unk	21.05	1.27	8669.50	2.62
7	Unk	23.18	1.39	3342.06	1.01
					100.00

Figure S4: HPLC analysis of the reaction mixtures (NIC_01 to NIC_05).

5 Characterization data of some selected compounds.

5.1 Melting point, IR & ^{1H}NMR Data

1) 2-(phenylamino) nicotinic acid :(**NIC/01)**[4]

MP: 151-153°C, FT-IR (KBr)cm^{-1}: 3244.6,3057.5,2783.7,1649.8,1594.8,1234.2,747.2.^{1}H NMR (500 MHz, DMSO-d6) δ 13.57 (s, 1H), 10.43 (s, 1H), 8.37 (d, *J* = 4.9 Hz, 1H), 8.23 (d, *J* = 7.7 Hz, 1H), 7.69 (d, *J* = 8.2 Hz, 2H), 7.30 (t, *J* = 7.5 Hz, 2H), 6.99 (t, *J* = 7.4 Hz, 1H), 6.91 – 6.76 (m, 1H).

2) 2-(3-tolylamino) nicotinic acid: **(NIC/02)**[5]

MP: 154-156°C, FT-IR (KBr)cm-1: 3773.0,3248.5,1590.99,1494.5,1204.9,767.3,592.0.1H NMR (500 MHz, DMSO-d6) δ 10.30 (s, 1H), 8.25 (dd, J = 4.8, 2.1 Hz, 1H), 8.10 (dd, J = 7.7, 2.1 Hz, 1H), 7.44 (d, J = 10.6 Hz, 1H), 7.34 (s, 1H), 7.06 (t, J = 7.8 Hz, 1H), 6.80 – 6.62 (m, 2H), 2.16 (s, 3H).

3) 2-((2,3-dimethylphenyl) amino) nicotinic acid: (**NIC/04)**[4,5]

MP:234-236°C. ,FT-IR (KBr)cm^{-1}: 3783.6,3235,2761.56,1580.3,1491.6,1230.3,959.4,767.5,581.4. ^{1}H NMR (500 MHz, DMSO-d6) δ 13.42 (s, 1H), 10.09 (s, 1H), 8.25 (dd, *J* = 4.8, 2.1 Hz, 1H), 8.19 (dd, *J* = 7.8, 2.1 Hz, 1H), 7.78 (d, *J* = 8.0 Hz, 1H), 7.03 (t, *J* = 7.8 Hz, 1H), 6.89 (d, *J* = 7.4 Hz, 1H), 6.76 (dd, *J* = 7.7, 4.7 Hz, 1H), 2.24 (s, 3H), 2.12 (s, 3H)

4) 2-((2-fluorophenyl) amino) nicotinic acid :(**NIC/005)**

MP :116-117°C ,FT-IR (KBr)cm^{-1}:3694.94,3320.82,2973.7,1662.2,1587.13,1419,1289,1227,1045,729. ^1H NMR (500 MHz, DMSO-d6)

δ 13.69 (s, 1H), 10.64 (s, 1H), 8.57 (td, *J* = 8.3, 1.7 Hz, 2H), 8.41 (dd, *J* = 4.8, 2.1 Hz, 2H), 8.27 (dd, *J* = 7.7, 2.0 Hz, 2H), 7.25 (dd, *J* = 10.9, 8.9 Hz, 1H), 7.20 – 7.13 (m, 1H), 7.01 (qd, *J* = 7.4, 1.9 Hz, 1H), 6.94 – 6.88 (m, 1H).

5) 2-((3-chlorophenyl) amino) nicotinic acid :((**NIC/007)**[4]

MP :200-202°C ,FT-IR (KBr)cm-1:3323.7,3076.8,1587.1,1412.6,1230.3,869.7,756.9,660.5,519.7.^1H NMR (500 MHz, DMSO-D6) δ 13.70 (s, 1H), 10.54 (s, 1H), 8.41 (dd, J = 4.8, 2.1 Hz, 2H), 8.24 (dd, J = 7.7, 2.1 Hz, 2H), 8.07 (s, 1H), 7.46 (d, J = 8.1 Hz, 1H), 7.29 (t, J [4]= 8.1 Hz, 2H), 7.02 (d, J = 8.0 Hz, 1H), 6.90 (dd, J = 7.8, 4.7 Hz, 2H).

6) 2-((4-chlorophenyl) amino) nicotinic acid: (**NIC/008)**[4,5]

MP:208-210°C**,** FT-IR (KBr)cm^{-1}: 3776.9,3036.3,1635.3,1549.5,1216.8,1079.9,846.5.^1H NMR (500 MHz, DMSO-D6) δ δ 13.55 (s, 1H), 10.43 (s, 1H), 8.31 (d, *J* = 2.9 Hz, 1H), 8.18 (d, *J* = 7.8 Hz, 1H), 7.68 (d, *J* = 8.8 Hz, 2H), 7.27 (d, *J* = 8.9 Hz, 2H), 6.81 (dd, *J* = 7.8, 4.7 Hz, 1H).

7) 2-((3-chloro-2-methylphenyl) amino) nicotinic acid: (**NIC/009)**

MP:232-234°C, FT-IR (KBr)cm^{-1}: 3224.4,2764.4,1663.3,1580.3,1494.5,1227.4,1131,966.1,763.6. ^1H NMR (500 MHz, DMSO-D6) δ 13.61 (s, 1H), 10.27 (s, 1H), 8.31 (dt, *J* = 4.8, 2.1 Hz, 1H), 8.23 (dt, *J* = 7.7, 2.3 Hz, 1H), 8.08 (d, *J* = 8.0 Hz, 1H), 7.23 – 7.09 (m, 2H), 6.84 (dt, *J* = 8.6, 4.3 Hz, 1H), 2.29 (s, 3H).

8) 2-((3-nitrophenyl) amino) nicotinic acid: **(NIC/10)**[6]

MP:242.243°C. FT-IR (KBr)cm-1:3316.0,3073.9,2842.5,1673.9,1571.7,1426.1,1147.4,734.7,526.4. 1H NMR (500 MHz, DMSO-d6) δ 13.77 (s, 1H), 10.75 (s, 1H), 8.85 (dd, J = 4.4, 2.3 Hz, 1H), 8.41 (s, 1H), 8.24 (d, J = 7.8 Hz, 1H), 7.88 (d, J = 8.2 Hz, 1H), 7.77 (d, J = 8.2 Hz, 1H), 7.51 (t, J = 8.2 Hz, 1H), 7.00 – 6.85 (m, 1H).

9) 2-((2-methyl-3-(trifluoromethyl) phenyl) amino) nicotinic acid: (**NIC/12)**[5,7]

MP :233-235°C**.** FT-IR (KBr)cm^{-1}:3231.15,2775,1666.2,1577.4,1491.6,1302.2,1234.2,1113.6,767.5,578.5. ^1H NMR (500 MHz, DMSO-d6) δ 13.69 (s, 1H), 10.64 (s, 1H), 8.57 (td, *J* = 8.3, 1.7 Hz, 2H), 8.41 (dd, *J* = 4.8, 2.1 Hz, 2H), 8.27 (dd, *J* = 7.7, 2.0 Hz, 2H), 7.25 (dd, *J* = 10.9, 8.9 Hz, 1H), 7.20 – 7.13 (m, 1H), 7.01 (qd, *J* = 7.4, 1.9 Hz, 1H), 6.94 – 6.88 (m, 1H).

10) 2-((4-methoxyphenyl) amino) nicotinic acid: (**NIC/13)**[5,8]

MP :209-211°C ,FT-IR (KBr)cm^{-1}:3230.18,3064.33,2767.3,1669,1594.8,1578.4,1238,1028.8,767.5. ^1H NMR (500 MHz, DMSO-d6) δ 13.43 (s, 1H), 10.17 (s, 1H), 8.27 (dd, *J* = 4.7, 2.1 Hz, 1H), 8.16 (dd, *J* = 7.7, 2.1 Hz, 1H), 7.52 (d, *J* = 9.1 Hz, 2H), 6.85 (d, *J* = 9.1 Hz, 2H), 6.74 (dd, *J* = 7.7, 4.7 Hz, 1H), 3.68 (s, 3H).

6 References:

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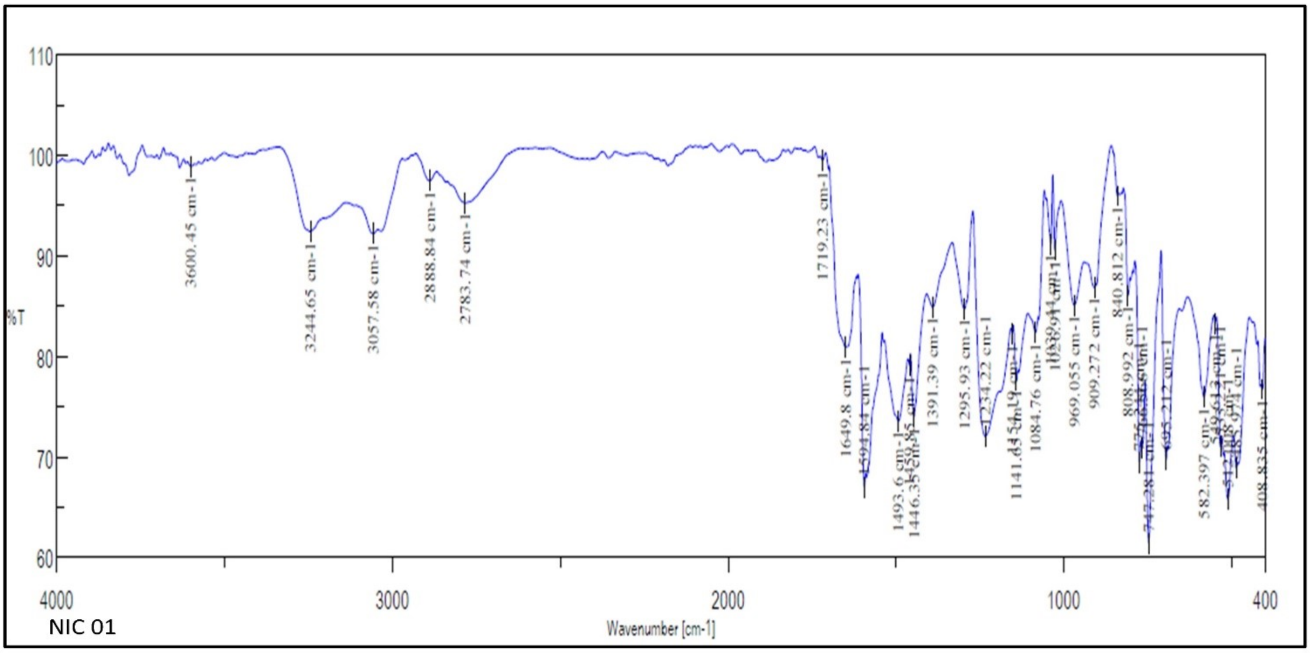
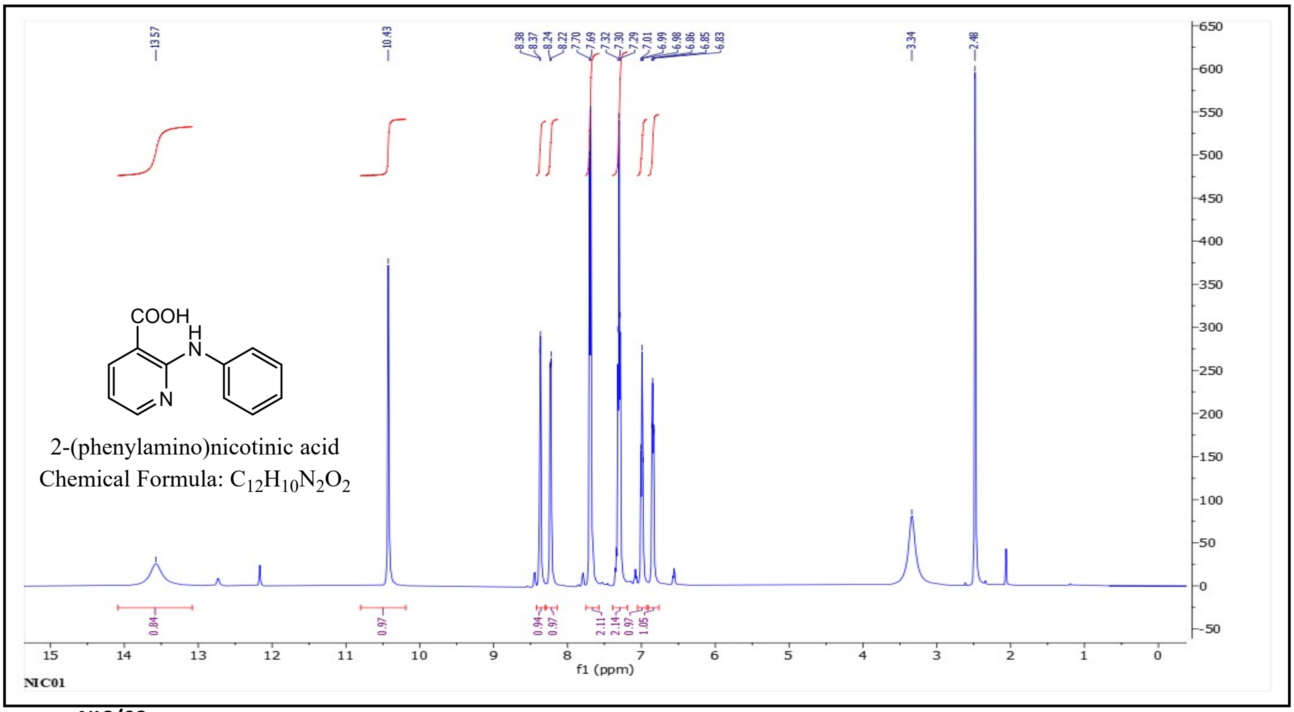
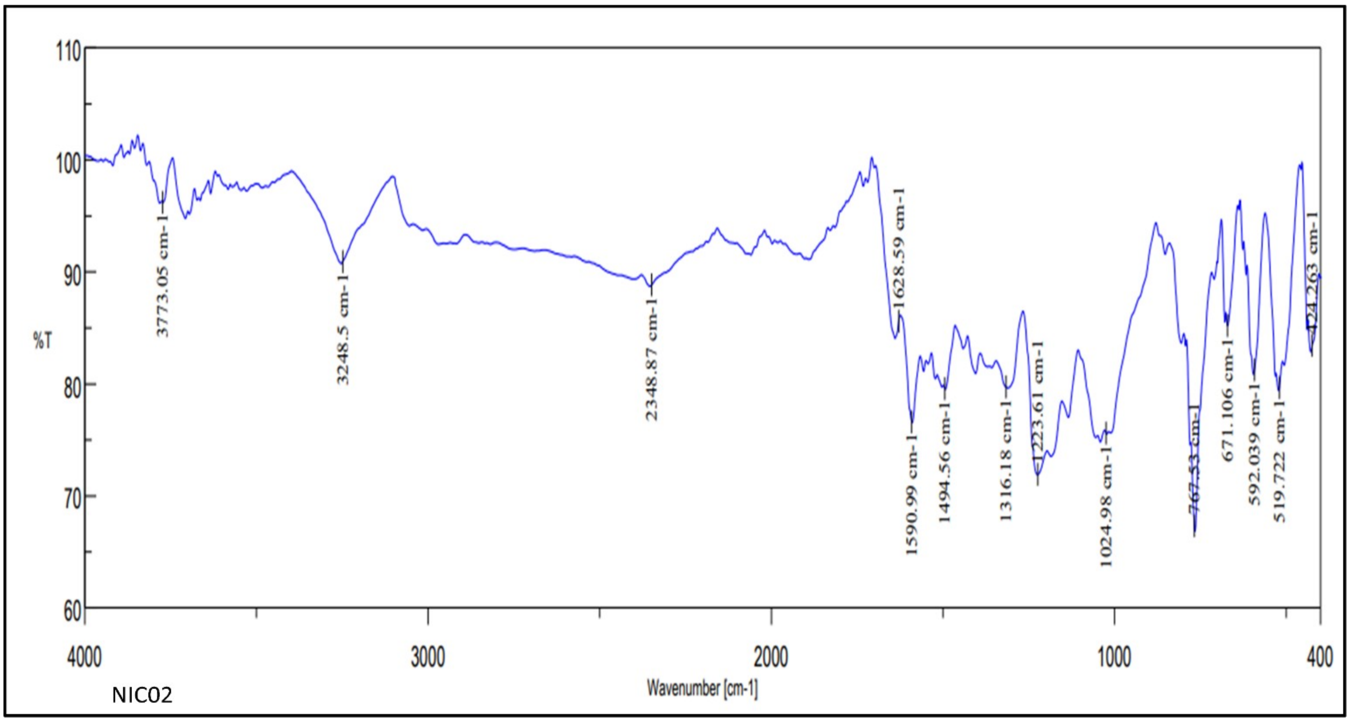
7. [1]H NMR spectra of products, FTIR.

NIC /01:





NIC/02



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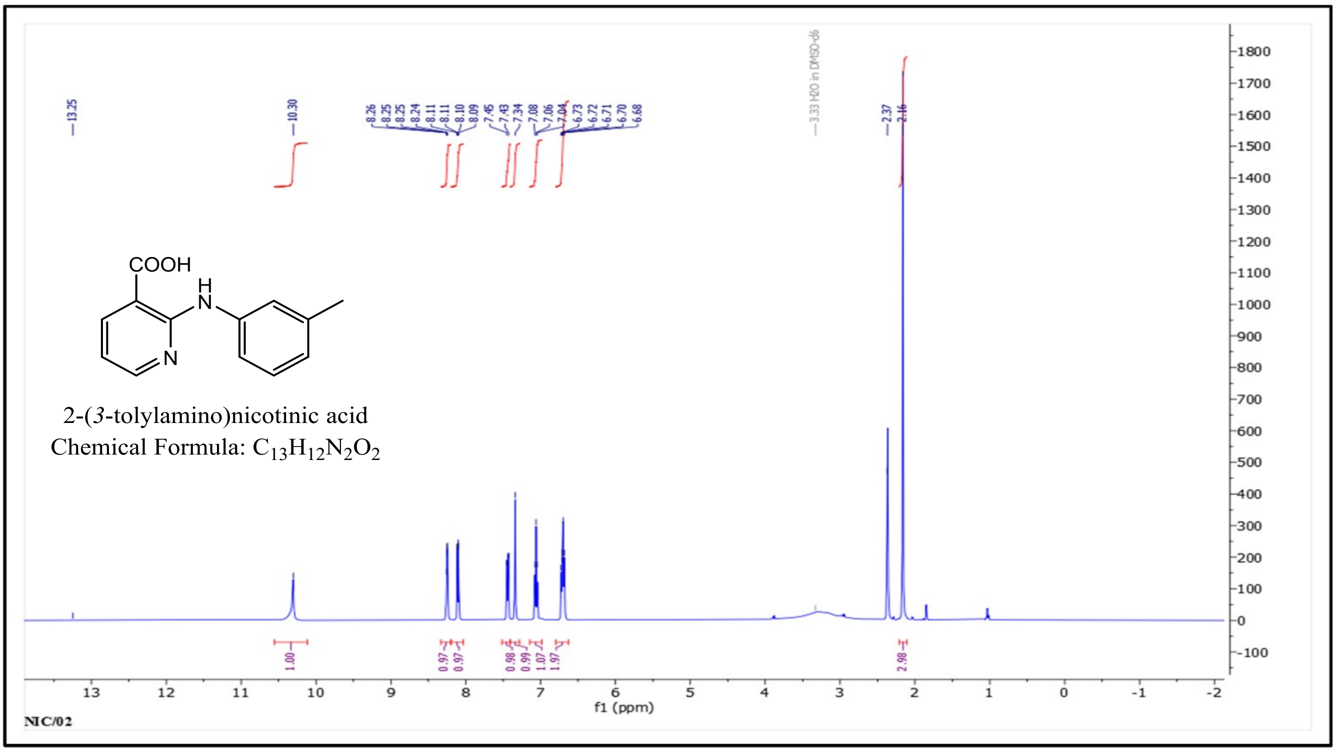
%T
Wavenumber [cm-1]
NIC02



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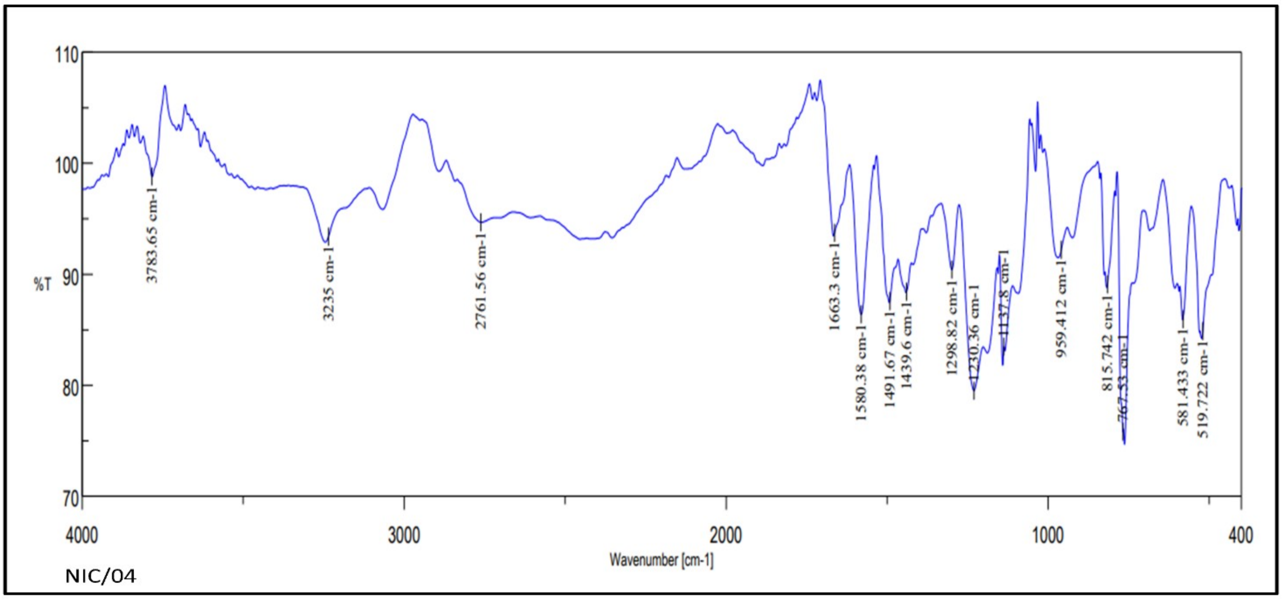
f1 (ppm)
NIC/02

NIC/04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959.412 cm-1
815.742 cm-1
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2000
1000
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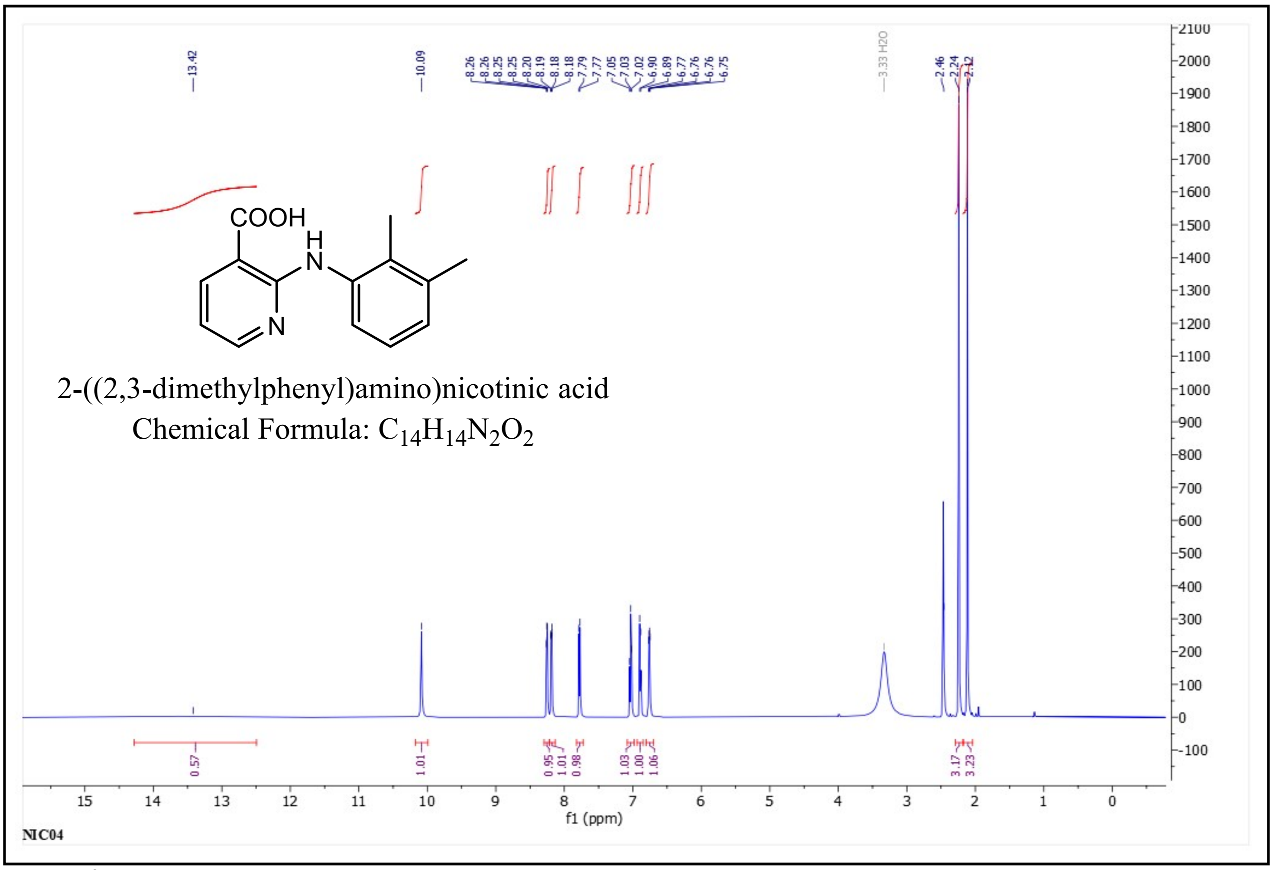
Wavenumber [cm-1]
NIC/04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10.09
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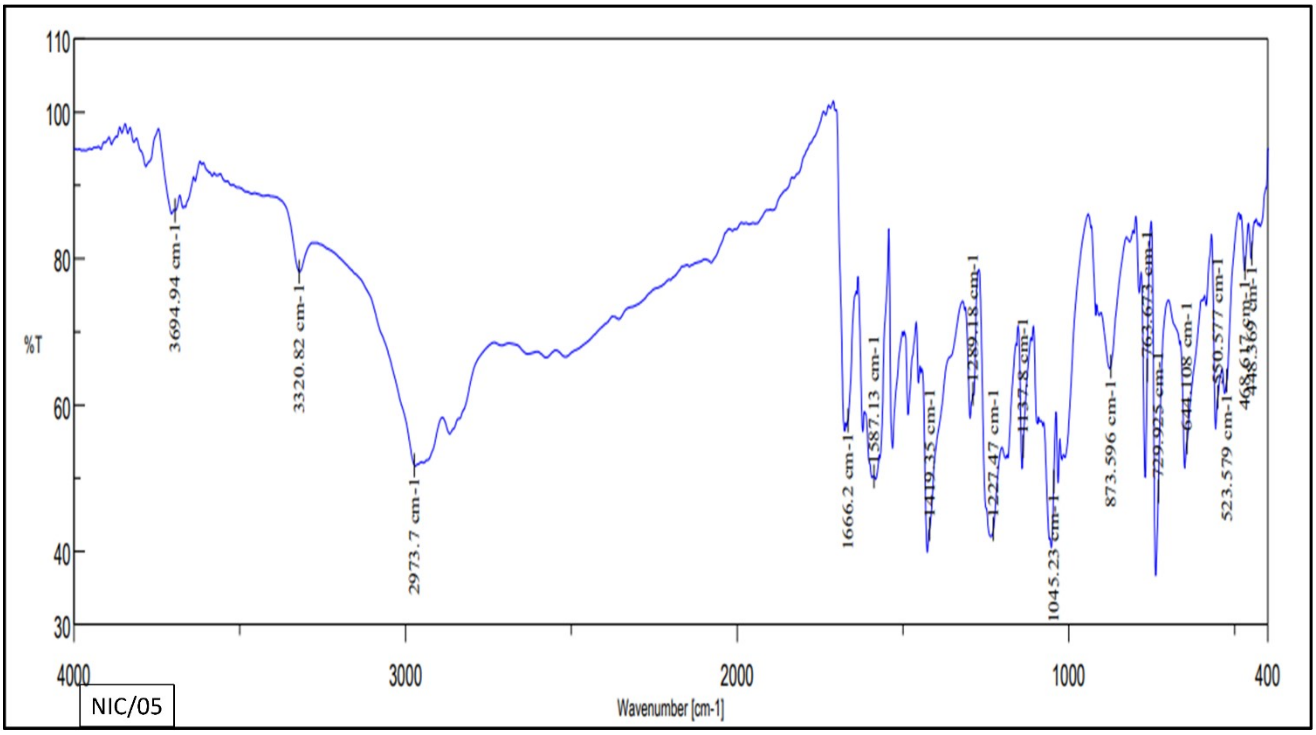
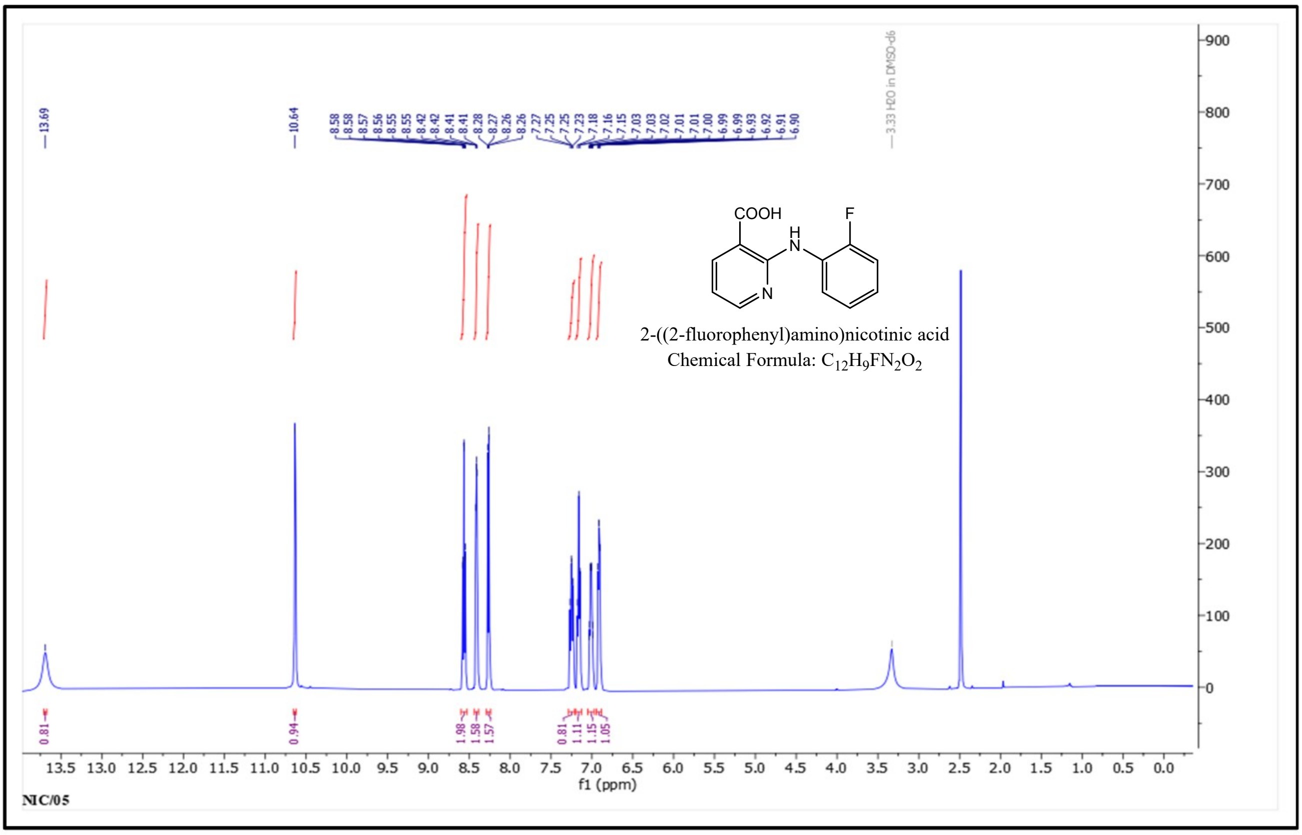
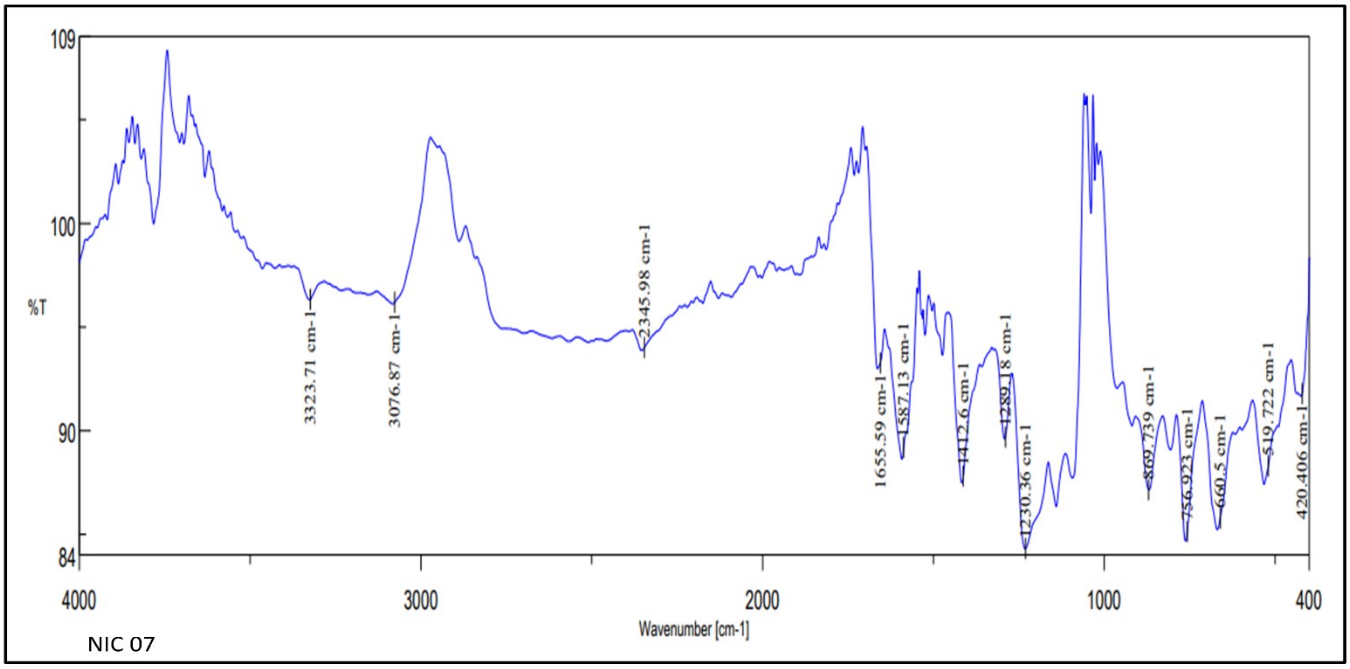
3.33 H2O
2.46 2.24 2.12
COOH
H
N
N
2-((2,3-dimethylphenyl)amino)nicotinic acid
Chemical Formula: $C_{14}H_{14}N_2O_2$
0.57
1.01
0.95 1.01 0.98
1.03 1.00 1.06
3.17 3.23
f1 (ppm)
NIC04

NIC/05

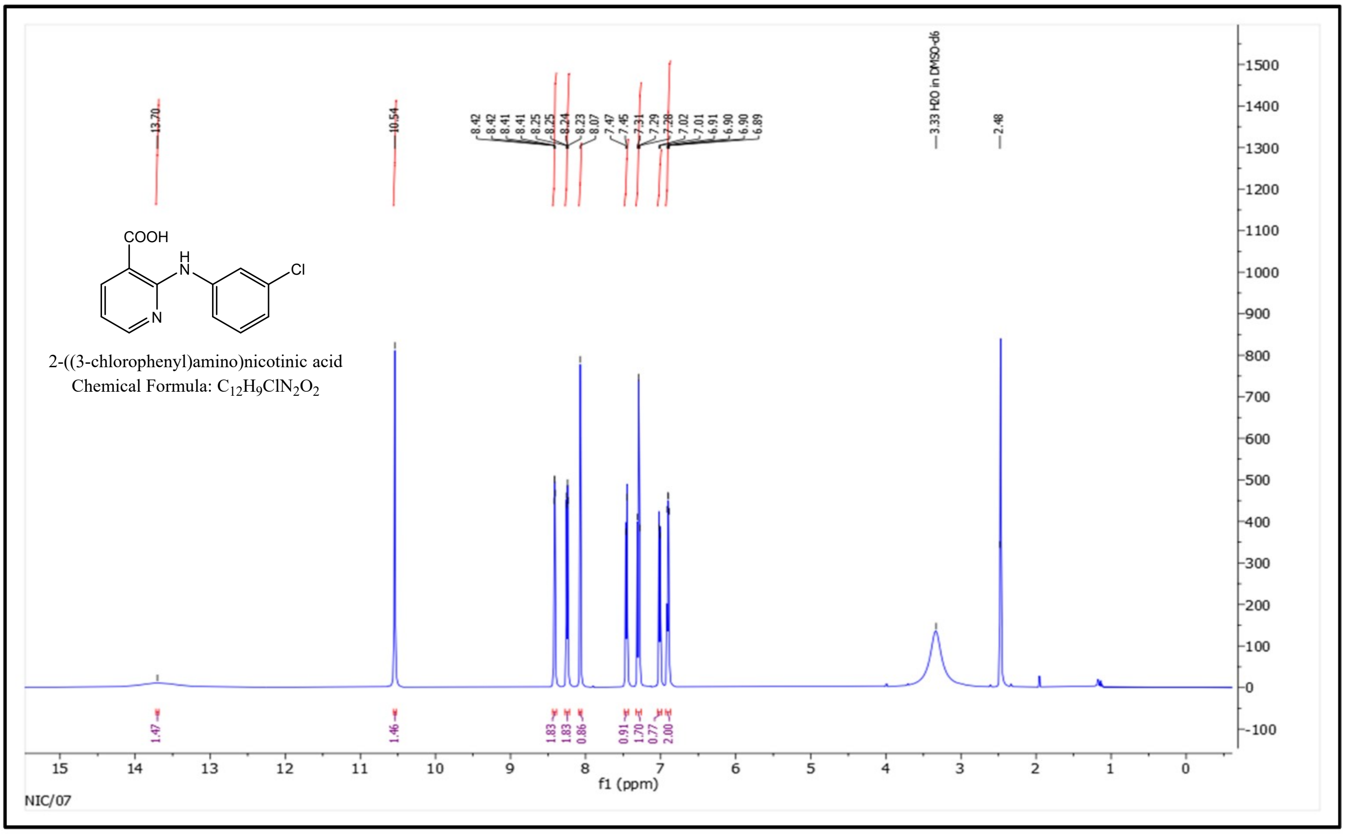

%T
3694.94 cm-1
3320.82 cm-1
2973.7 cm-1
1666.2 cm-1
1587.13 cm-1
1419.35 cm-1
1289.18 cm-1
1227.47 cm-1
1137.8 cm-1
1045.23 cm-1
873.596 cm-1
763.673 cm-1
729.925 cm-1
644.108 cm-1
550.577 cm-1
523.579 cm-1
468.617 cm-1
448.369 cm-1
Wavenumber [cm-1]
NIC/05


13.69
10.64
8.58 8.58 8.57 8.56 8.55 8.55 8.42 8.42 8.41 8.41 8.28 8.27 8.26 8.26 7.27 7.25 7.25 7.23 7.18 7.16 7.15 7.03 7.03 7.02 7.01 7.01 7.00 6.99 6.99 6.93 6.92 6.91 6.90
3.33 H2O in DMSO-d6
COOH
H
N
F
N
2-((2-fluorophenyl)amino)nicotinic acid
Chemical Formula: $C_{12}H_9FN_2O_2$
0.81
0.94
1.98 1.58 1.57
0.81 1.11 1.15 1.05
f1 (ppm)
NIC/05

NIC/07

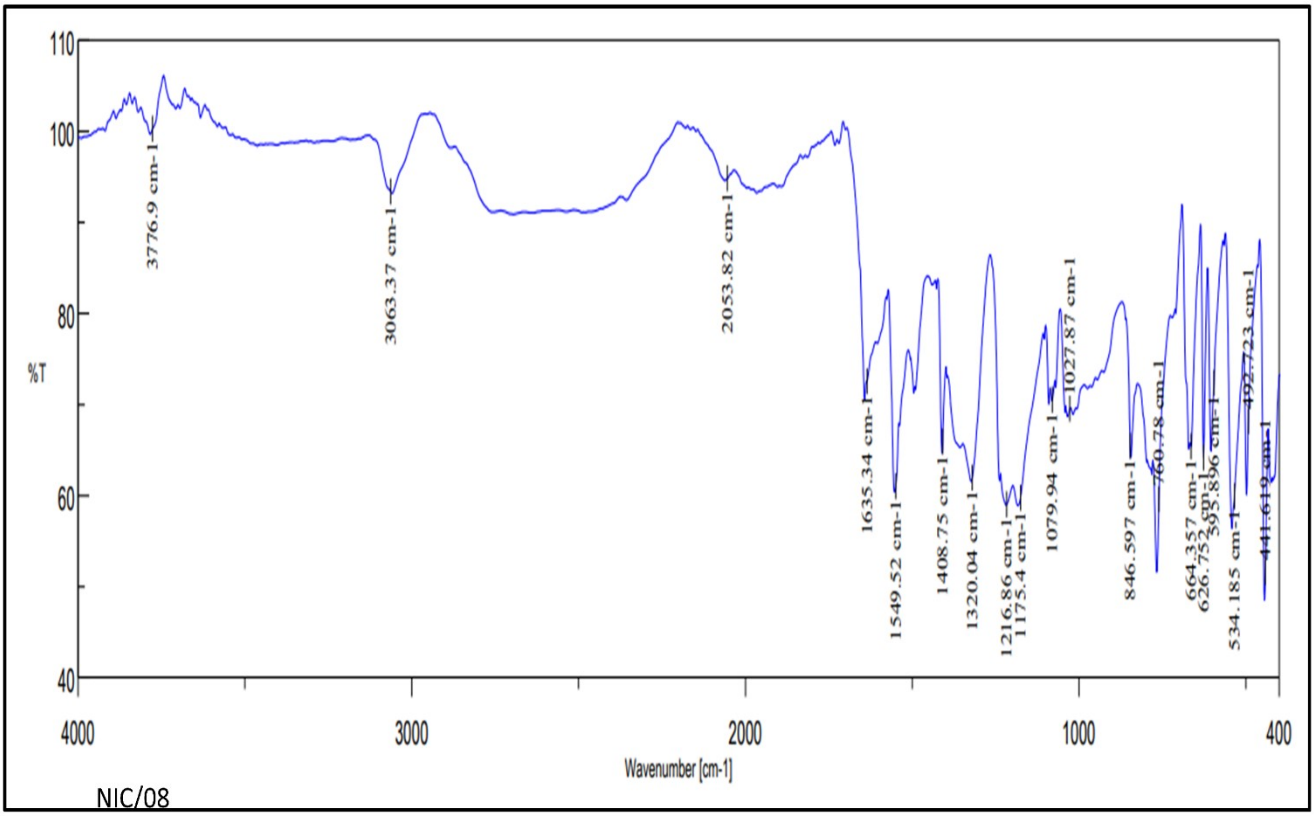


109
100
%T
90
84
3323.71 cm-1
3076.87 cm-1
2345.98 cm-1
1655.59 cm-1
1587.13 cm-1
1412.6 cm-1
1289.18 cm-1
1230.36 cm-1
869.739 cm-1
756.923 cm-1
660.5 cm-1
519.722 cm-1
420.406 cm-1
4000
3000
2000
1000
400
Wavenumber [cm-1]
NIC 07

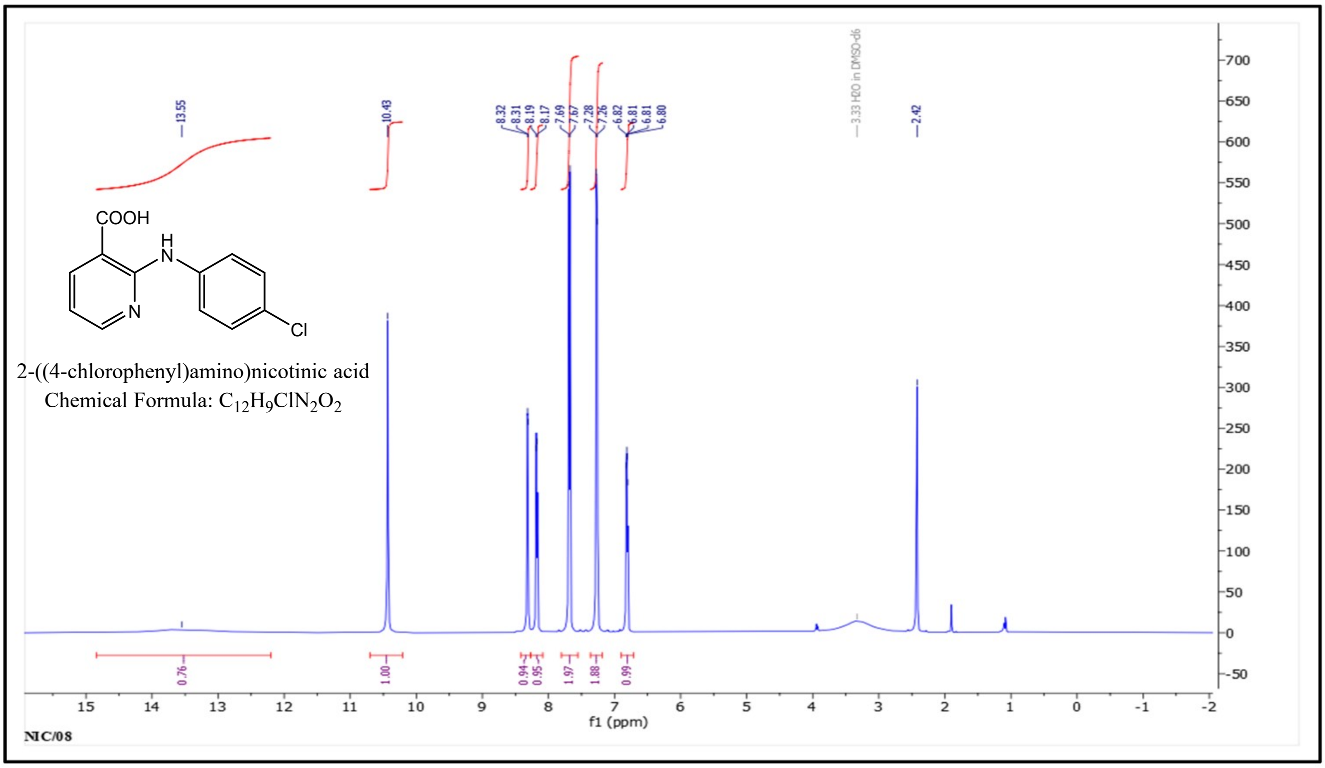


13.70
10.54
8.42 8.42 8.41 8.41 8.25 8.25 8.24 8.23 8.07 7.47 7.45 7.31 7.29 7.28 7.02 7.01 6.91 6.90 6.90 6.89
3.33 H2O in DMSO-d6
2.48
COOH
H
N
Cl
N
2-((3-chlorophenyl)amino)nicotinic acid
Chemical Formula: $C_{12}H_9ClN_2O_2$
1.47
1.46
1.83
1.83
0.86
0.91
1.70
0.77
2.00
f1 (ppm)
NIC/07

NIC/08

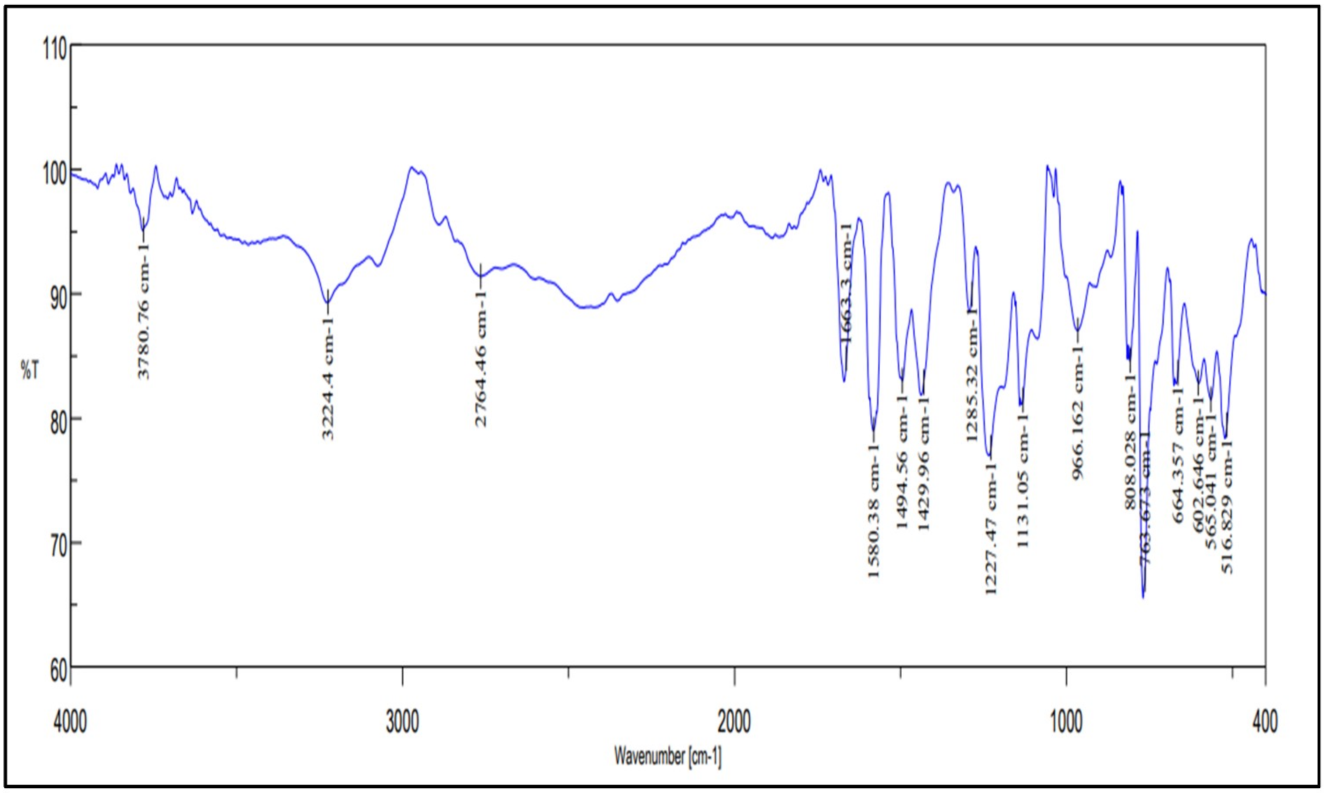


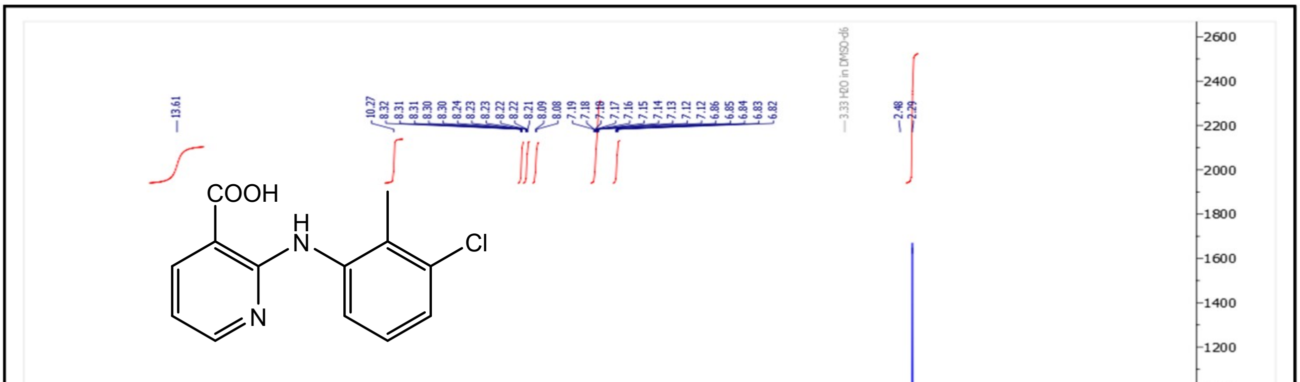
110
100
80
60
40
%T
3776.9 cm-1
3063.37 cm-1
2053.82 cm-1
1635.34 cm-1
1549.52 cm-1
1408.75 cm-1
1320.04 cm-1
1216.86 cm-1
1175.4 cm-1
1079.94 cm-1
1027.87 cm-1
846.597 cm-1
760.78 cm-1
664.357 cm-1
626.752 cm-1
595.896 cm-1
534.185 cm-1
492.723 cm-1
441.619 cm-1
4000
3000
2000
1000
400
Wavenumber [cm-1]
NIC/08



13.55
10.43
8.32
8.31
8.19
8.17
7.69
7.67
7.28
7.26
6.82
6.81
6.81
6.80
3.33 H2O in DMSO-d6
2.42
COOH
H
N
N
Cl
2-((4-chlorophenyl)amino)nicotinic acid
Chemical Formula: C12H9ClN2O2
0.76
1.00
0.94
0.95
1.97
1.88
0.99
f1 (ppm)
NIC/08

NIC/09

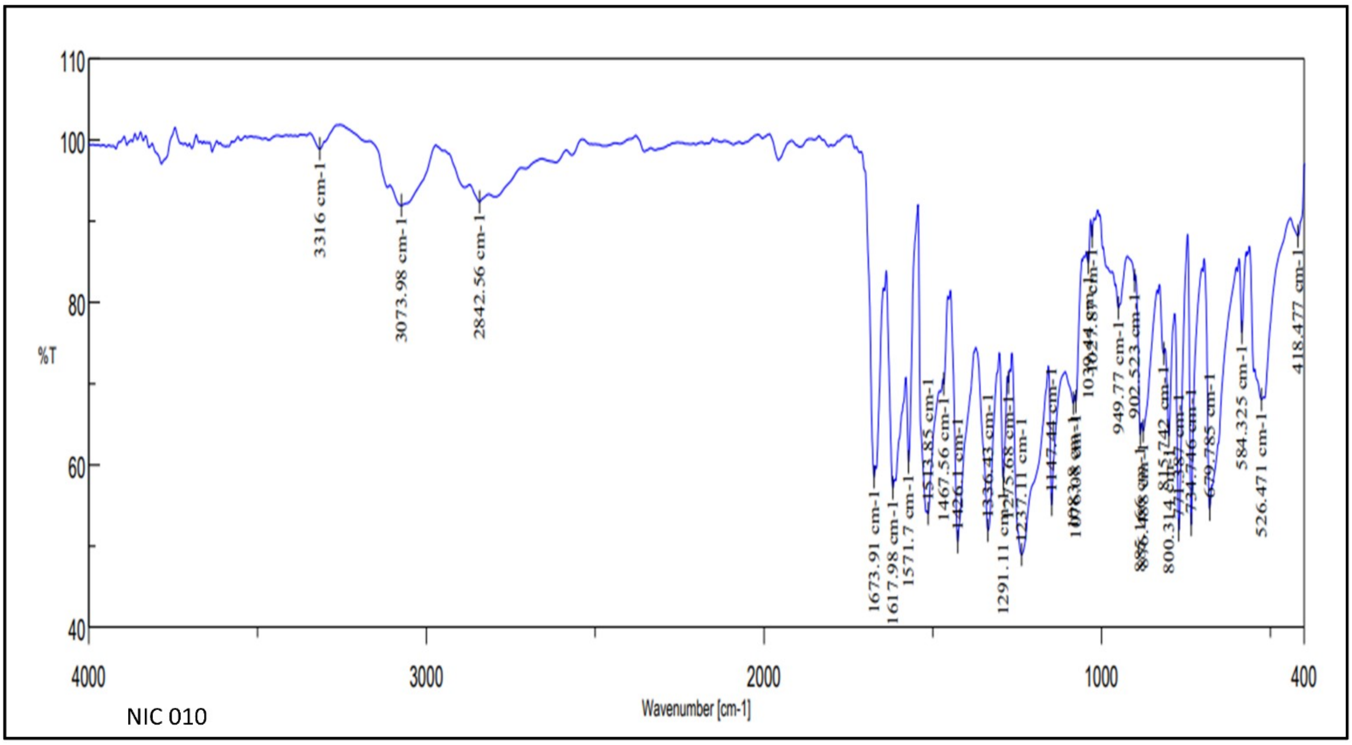




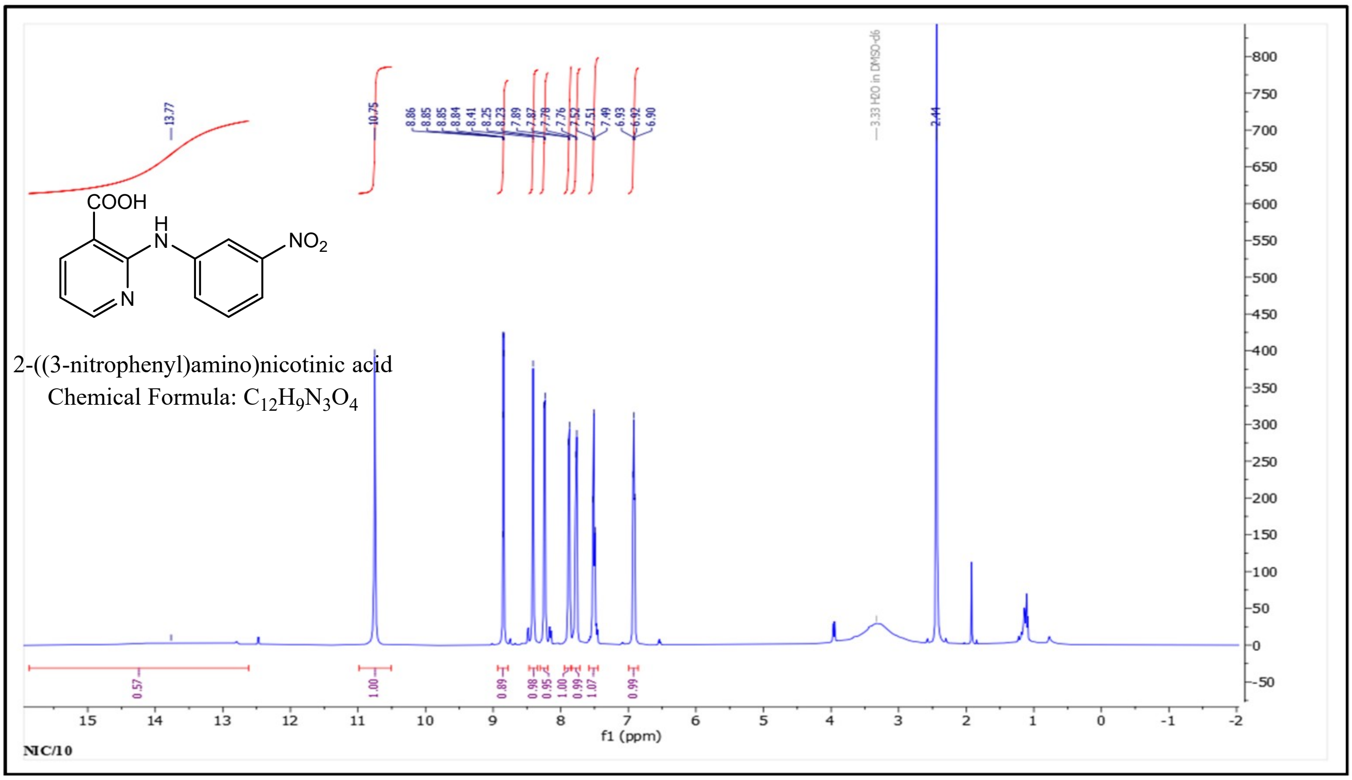
2-((3-chloro-2-methylphenyl)amino)nicotinic acid

Chemical Formula: $C_{13}H_{11}ClN_2O_2$

NIC/010

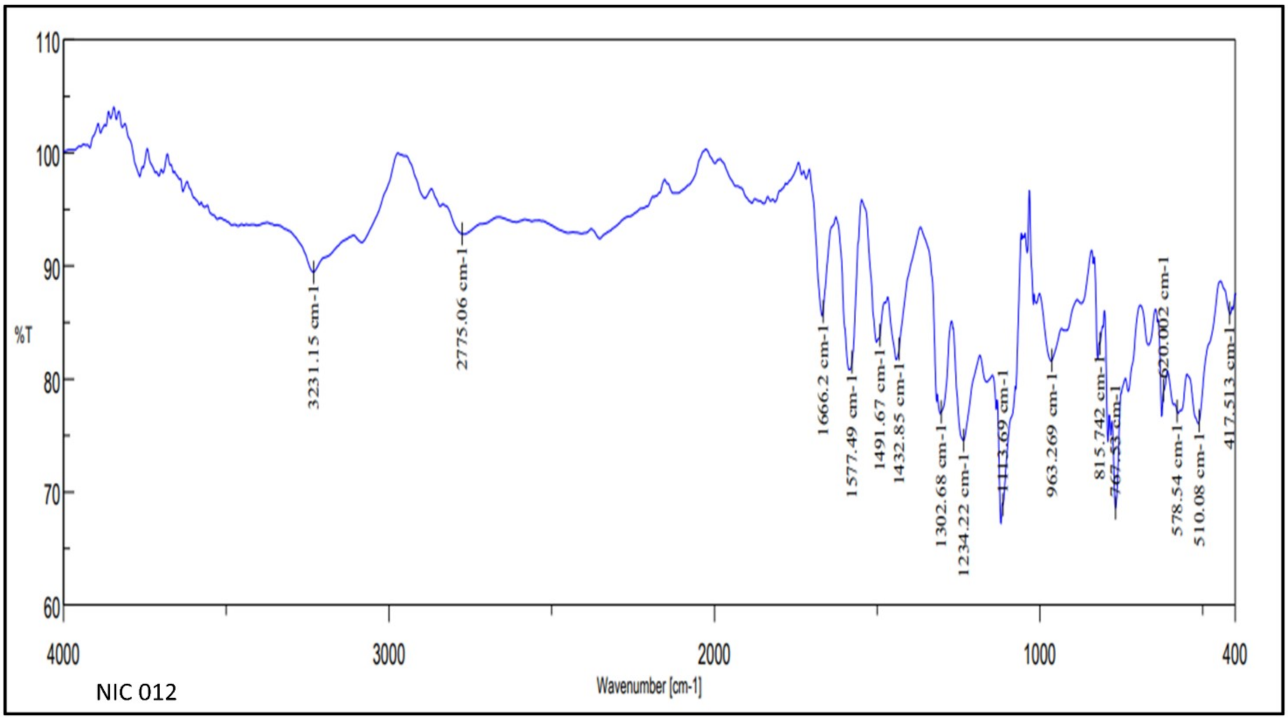


%T
Wavenumber [cm-1]
NIC 010
3316 cm-1
3073.98 cm-1
2842.56 cm-1
1673.91 cm-1
1617.98 cm-1
1571.7 cm-1
1513.85 cm-1
1467.56 cm-1
1426.1 cm-1
1336.43 cm-1
1291.11 cm-1
1237.11 cm-1
1147.44 cm-1
949.77 cm-1
902.523 cm-1
584.325 cm-1
526.471 cm-1
418.477 cm-1

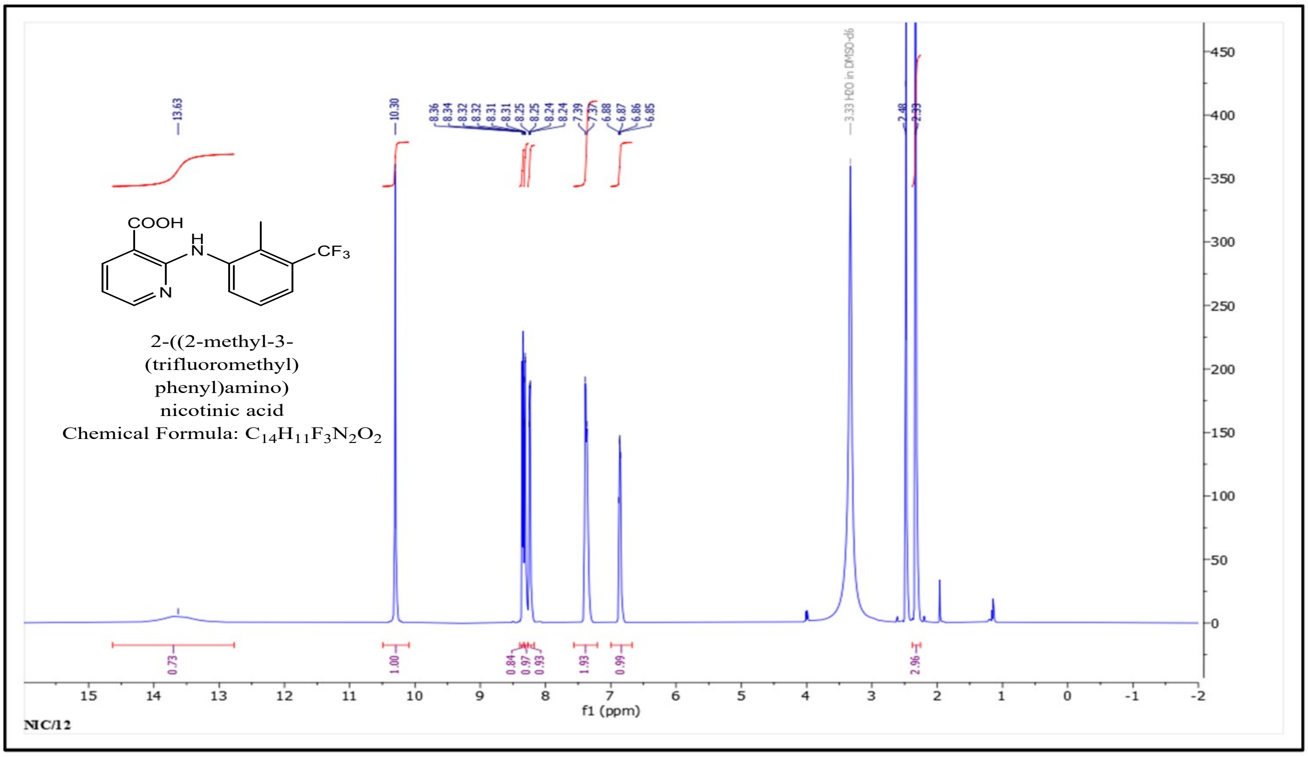


COOH
H
N
NO2
N
2-((3-nitrophenyl)amino)nicotinic acid
Chemical Formula: C12H9N3O4
NIC/10
f1 (ppm)
13.77
10.75
3.33 H2O in DMSO-d6
2.44

NIC/12

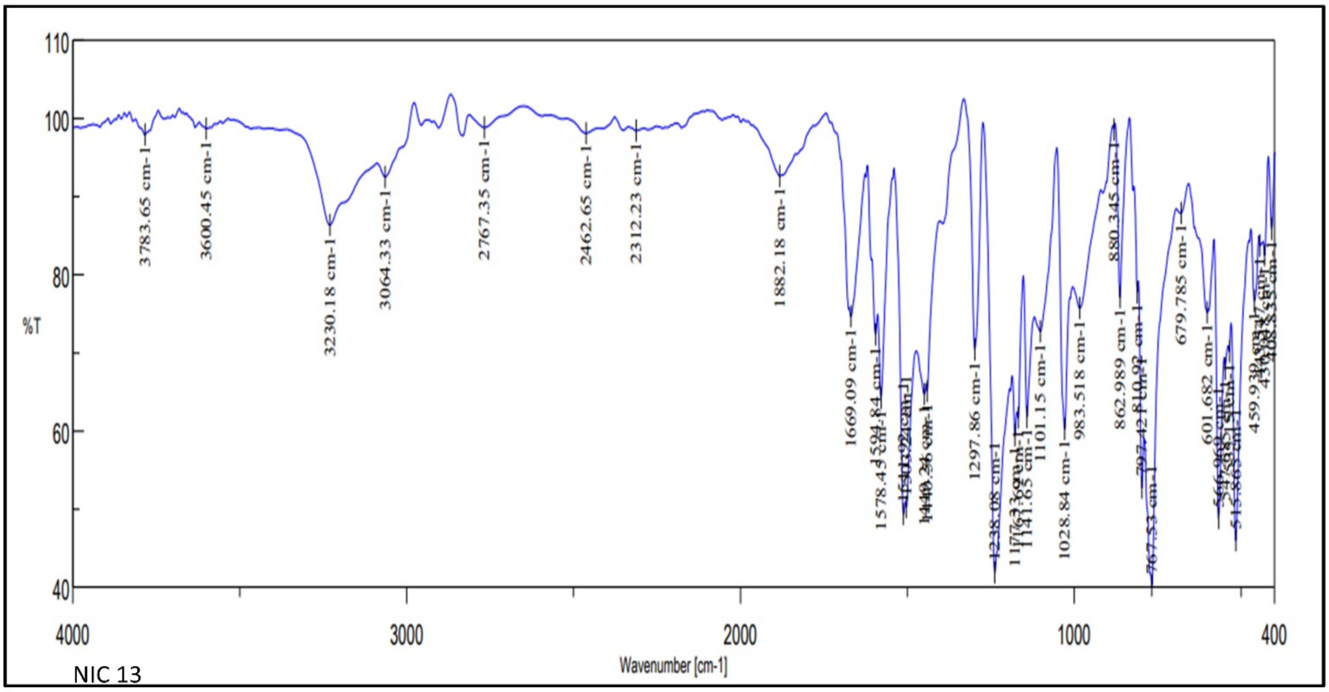


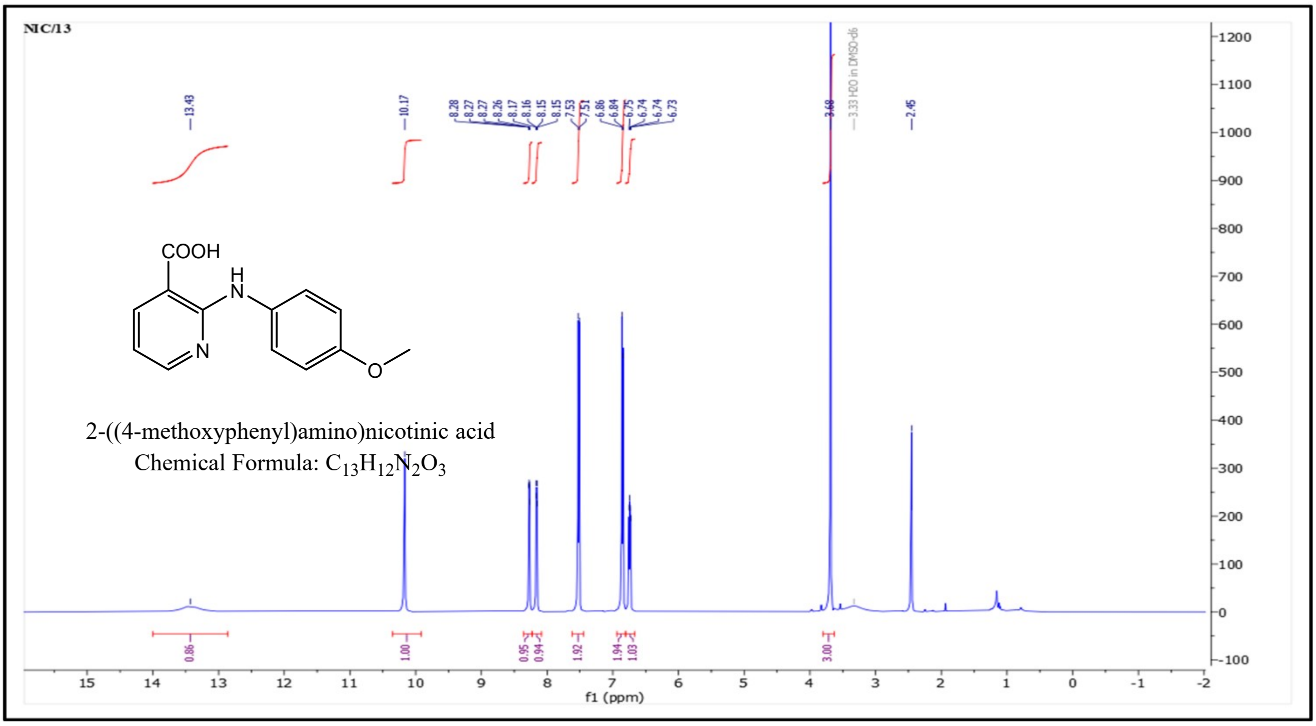
%T
3231.15 cm-1
2775.06 cm-1
1666.2 cm-1
1577.49 cm-1
1491.67 cm-1
1432.85 cm-1
1302.68 cm-1
1234.22 cm-1
1113.69 cm-1
963.269 cm-1
815.742 cm-1
767.53 cm-1
620.002 cm-1
578.54 cm-1
510.08 cm-1
417.513 cm-1
Wavenumber [cm-1]
NIC 012



13.63
10.30
8.36 8.34 8.32 8.32 8.31 8.31 8.25 8.25 8.24 8.24
7.39 7.37
6.88 6.87 6.86 6.85
3.33 H2O in DMSO-d6
2.48 2.33
COOH
N
H
CF3
2-((2-methyl-3-(trifluoromethyl)phenyl)amino)nicotinic acid
Chemical Formula: $C_{14}H_{11}F_3N_2O_2$
0.73
1.00
0.84 0.97 0.93
1.93
0.99
2.96
f1 (ppm)
NIC/12

NIC/013


NIC 13
%T
Wavenumber [cm-1]
3783.65 cm-1
3600.45 cm-1
3230.18 cm-1
3064.33 cm-1
2767.35 cm-1
2462.65 cm-1
2312.23 cm-1
1882.18 cm-1
1669.09 cm-1
1297.86 cm-1
1238.08 cm-1
1101.15 cm-1
1028.84 cm-1
983.518 cm-1
880.345 cm-1
862.989 cm-1
767.53 cm-1
679.785 cm-1
601.682 cm-1


NIC/13
13.43
10.17
8.28
8.27
8.27
8.26
8.17
8.16
8.15
8.15
7.53
7.51
6.86
6.84
6.75
6.74
6.73
3.68
3.33 H2O in DMSO-d6
2.45
COOH
H
N
N
O
2-((4-methoxyphenyl)amino)nicotinic acid
Chemical Formula: $C_{13}H_{12}N_2O_3$
0.86
1.00
0.95
0.94
1.92
1.94
1.03
3.00
f1 (ppm)