Per-6-Amino-β-cyclodextrin/CuI catalysed cyanation of aryl halides with $K_4[Fe(CN)_6]$

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Contents

1a. Materials and methods:

All chemicals are commercially available and were used without further purification. DMF was distilled at reduced pressure and stored in 4Å molecular sieves. New products were fully characterized after isolation by NMR and IR spectroscopy and ESI-MS). Column chromatography was performed with silica gel 60-120 mesh or aluminium oxide active, neutral activity I-II. Thin layer chromatography was carried out with Merck silica gel 60 F254 plates. Nuclear magnetic resonance (NMR) spectra were acquired on a Bruker DRX-300 (300 MHz) instrument using TMS as an internal standard. $CDCl_3$ was used as solvent unless otherwise noted. Coupling constants are reported in Hz and chemical shifts in ppm [relative to TMS for ^{1}H and ^{13}C (δ 77.00 ppm for the $CDCl_3$ signal)]. Electrospray ionisation mass spectrometry (ESI-MS) analysis was performed in the positive ion mode on a liquid chromatography-ion trap mass spectrometer (LCQ Fleet, Thermo Fisher Instruments Limited, US). The samples were introduced into the ion source by infusion method at flow rate 1μL/min. The capillary voltage of the mass spectrometer was 33 V, with source voltage 4.98 kV for the mass scale (m/z 50-300 and 500-2000). The percentage conversion of the products were carried out by using gas chromatography (Shimadzu GC-17A model, ZB-5 (10%) capillary column with FID detector using high purity nitrogen as carrier gas). Products were identified by their GC retention times and also by coinjection with authentic samples. IR spectral analyses were performed using JASCO FT/IR-410 instrument by KBr pellet technique. Elemental analyses were performed on Perkin Elmer 2400 series II Elemental CHNS analyser. Microwave irradiations were

performed on CEM-discover model No. 908010. Analytical HPLC (for purity determination) was performed with a Phenomenox-Gemini-NX 5µ C18 (50 x 4.6 mm) column using HPLC-grade solvents on a Thermo Finningon HPLC system with Surveyor plus Solvent degasser, Surveyor Autosampler plus, Thermostatic column housing. All samples were filtrated prior to injection.

1b. HPLC purity of aminocyclodextrins:

As aminocyclodextrins not absorb uv light, we have used LC-MS to identify the purity of the ligand from total ion chromatogram and its m/z value.

Table S1: HPLC purity of aminocyclodextrins

Aminocyclodextrin	**HPLC-MS purity (%)[a]**
per-6-NH_2-β-CD	98
per-6-MeNH-β-CD	97
per-6-*n*-BuNH-β-CD	97
mono-6-NH_2-β-CD	98

Method: 0.1% aqueous acetic acid (solvent A) and 0.1% acetic acid in acetonitrile (solvent B), (0.3 mL/min).

1c. General procedure for Microwave irradiation:

A mixture of per-6-amino-β-cyclodextrin (0.1 mmol), aryl halide (1mmol), CuI (0.1 mmol), $K_4[Fe(CN)_6]$ (0.2 mmol), Na_2CO_3 (0.2 mmol), KI (0.3 mmol) and DMF (3mL) are taken in 10 mL quartz vial was subjected to microwave irradiatin, programmed at 120 W, 130°C and 1 bar pressure. After a period of 1-2 min, the temperature reached a plateau, 130°C, and remained constant. After a period of 30

min After the reaction, the mixture is filterred, water is added to the filtrate then extracted with ethyl acetate and the organic phase is dried over Na_2SO_4. After evaporation of the solvents the mixture is analysed by GC.

Table. S2 Optimisation of reaction conditions in microwave irradiation[a]

S. No	Temp (°C)	Time (mins)	Conversion (%)[b]
1	80	30	0
2	110	30	0
3	130	30	19.1
4	130	60	27.5
5	130	90	42.9
6	130	120	61.9
7	130	150	93.1[c]

[a]**Reaction Conditions:** 1mmol iodobenzene, 0.1 mmol CuI, 0.1 mmol per-6-NH_2-β-CD, 0.2 mmol $K_4[Fe(CN)_6]$, 0.2 mmol Na_2CO_3, 2.5 mL DMF, [b] Analysed by GC; [c]6.9% byproduct is observed.

2. Spectral data of aryl nitriles:

a) Characterization of benzonitrile (Table 2, entry 1): Compound is prepared according to the general procedure. The resulting crude oily residue was purified by chromatography on silica gel (eluent: petroleum ether/ethyl acetate, 97:03) to provide the desired product as a colourless oil. Yield 95% (98 mg), *^{1}H NMR (300 MHz, $CDCl_3$):* 7.46-7.63 (m, 5H); *^{13}C NMR (75 MHz, CDCl3):* 111.9, 118.4, 128.7, 132.0, 132.8; *ESI-MS:* m/z. Calcd. for C_7H_5N: 103.04; found: 104.00 (M+H); FT-IR (KBr, cm^{-1}): 3097, 3050, 2231, 1502, 1401, 1276, 1198, 844, 561.

b) Characterization of 4-hydroxybenzonitrile (Table 2, entry 2): Compound is prepared according to the general procedure. The resulting crude oily residue was purified by chromatography on silica gel (eluent: petroleum ether/ethyl acetate, 95:05) to provide the desired product as a white powder. Yield 89% (110 mg), *^{1}H NMR (300 MHz, $CDCl_3$):* 5.31 (s, 1H), 6.72 (d, *J*=8.7Hz, 2H), 7.61 (d, J=8.7Hz, 2H); *^{13}C NMR (75 MHz, $CDCl_3$):* 110.11, 114.45, 117.82, 138.44, 155.36; *ESI-MS:* m/z. Calcd. for C_7H_5NO: 119.04; found: 118.00.

c) Characterization of 1,4-dicyanobenzene (Table 2, entries 3 and 9): Compound is prepared according to the general procedure. The resulting crude oily residue was purified by chromatography on silica gel (eluent: petroleum ether/ethyl acetate, 98:02) to provide the desired product as a white crystals. Yield 97% (124 mg). *^{1}H NMR (300 MHz, $CDCl_3$):* 7.81 (s, 4H); *^{13}C NMR (75 MHz, $CDCl_3$):* 116.7, 117.0, 132.8; *ESI-MS:* m/z. Calcd. for $C_8H_4N_2$: 128.04; found: 126.75 (M-H); *FT-IR (KBr, cm^{-1}):* 3048, 3097, 2227, 1941, 1693, 1496, 1276, 1195;

d) Characterization of ethyl 4-cyanobenzoate (Table 2, entry 4): Compound is prepared according to the general procedure. The resulting residue was purified by chromatography on silica gel (eluent: petroleum ether/ethyl acetate, 95:05) to provide the desired product as a white crystals. Yield 90% (158 mg). *^{1}H NMR (300 MHz, $CDCl_3$):* 1.44 (t, *J*=4.8 Hz, 3H), 4.43 (q, *J*=4.8Hz, 2H), 7.77 (d, *J*=7.2Hz, 2H), 8.17 (d, *J*=7.2Hz, 2H); *^{13}C NMR (75 MHz, $CDCl_3$):* 14.1, 61.6, 116.1, 117.8, 129.9, 132.0, 134.2, 164.7; *ESI-MS:* m/z. Calcd. for $C_{10}H_9NO_2$: 175.06; found: 174.65 (M-H); *FT-IR (KBr, cm^{-1}):* 3063, 2931, 2230, 1721, 1278, 1107, 1021;

e) Characterization of ethyl 3-cyanobenzoate (Table 2, entry 5): Compound is prepared according to the general procedure. The resulting residue was purified by chromatography on silica gel (eluent: petroleum ether/ethyl acetate, 95:05) to provide the desired product as a white powder. Yield 79% (138 mg). *^{1}H NMR (300 MHz, $CDCl_3$):* 1.39 (t, *J*=7.2 Hz, 3H), 4.44 (q, *J*=7.2Hz, 2H), 7.23, (t, *J*=5.1Hz, 1H), 7.83 (d, *J*=7.8Hz, 1H), 7.97 (d, *J*=6.6Hz, 1H), 8.34 (s, 1H) *^{13}C NMR (75 MHz, $CDCl_3$):* 14.2, 61.3, 116.1, 117.8, 128.6, 129.9, 132.3, 138.3, 141.5, 164.9; *ESI-MS:* m/z. Calcd. for $C_{10}H_9NO_2$: 175.06; found: 176.00 (M+H); *FT-IR (KBr, cm^{-1}):* 3056, 2927, 2225, 1722, 1278, 1106, 1018.

f) Characterization of 2-cyanobenzoic acid (Table 2, entry 6): Compound is prepared according to the general procedure. Yield 76% (112 mg). *^{1}H NMR (300 MHz, $CDCl_3$):* 7.16-7.27 (m, 1H), 7.44 (t, *J*=7.5Hz, 1H), 8.00-8.07 (m, 2H) *^{13}C NMR (75 MHz, $CDCl_3$):* 116.5, 117.9, 128.0, 132.0, 133.5, 141.9, 170.9; *ESI-MS:* m/z. Calcd. for $C_8H_5NO_2$: 147.03; found: 148.24 (M+H);

g) Characterization of 2,4-dimethoxybenzonitrile (Table 2, entry 7): Compound is prepared according to the general procedure. The resulting crude oily residue was purified by chromatography on silica gel (eluent: petroleum ether/ethyl acetate, 95:5) to provide the desired product as white powder. Yield 81% (132 mg). *^{1}H NMR (300 MHz, $CDCl_3$):* 3.86 (s, 3H), 3.90 (s, 3H), 6.46 (s, 1H), 6.52 (d, *J*=8.7Hz, 1H), 7.48 (d, *J*=8.7Hz, 1H); *^{13}C NMR (75 MHz, $CDCl_3$):*55.6, 55.9, 93.9, 98.4, 105.8, 116.9, 134.8, 162.8, 164.6; *ESI-MS:* m/z. Calcd. for $C_9H_9NO_2$: 163.03; found: 186.22 (M+Na); *FT-IR (KBr, cm^{-1}):* 3083, 2927, 2848, 2217, 1606, 1504, 1477, 1328, 1214;

h) Characterization of 3,4-dimethylbenzonitrile (Table 2, entry 8): Compound is prepared according to the general procedure. The resulting crude oily residue was purified by chromatography on silica gel (eluent: petroleum ether/ethyl acetate, 96:4) to provide the desired product as pale yellow crystal. Yield 83% (110 mg). *^{1}H NMR (300 MHz, $CDCl_3$):* 2.19 (s, 6H), 6.82 (d, *J*= 7.8Hz, 1H), 7.37 (d, *J*=7.8Hz, 1H), 7.44 (s, 1H); *^{13}C NMR (75 MHz, $CDCl_3$):* 19.2, 19.3, 105.9, 116.9, 131.4, 134.7, 136.0, 138.1, 138.9. *ESI-MS:* m/z. Calcd. for C_9H_9N: 131.07; found: 130.02 (M-H);

i) Characterization of 4-chlorobenzonitrile (Table 2, entry 10): Compound is prepared according to the general procedure. The resulting crude oily residue was purified by chromatography on silica gel (eluent: petroleum ether/ethyl acetate, 92:8) to provide the desired product as white powder. Yield 73% (100 mg). *^{1}H NMR (300 MHz, $CDCl_3$):* 7.46 (d, *J*=7.2Hz, 2H), 7.60 (d, *J*=7.2Hz, 2H) *^{13}C NMR (75 MHz,*

$CDCl_3$): 110.2, 119.8, 130.1, 133.8, 139.9; *ESI-MS:* m/z. Calcd. for C_7H_4ClN: 137.00; found: 136.00 (M-H);

j) Characterization of 4-nitrobenzonitrile (Table 2, entry 11): Compound is prepared according to the general procedure. The resulting crude oily residue was purified by chromatography on silica gel (eluent: petroleum ether/ethyl acetate, 97:03) to provide the desired product as a slight yellowish white powder. Yield 87% (129 mg).*^1H NMR (300 MHz, $CDCl_3$):* 7.91 (d, *J*=7.2Hz, 2H), 8.37 (d, *J*=7.2Hz, 2H); *^{13}C NMR (75 MHz, $CDCl_3$):* 116.7, 118.3, 124.2, 133.4, 150.0 *ESI-MS:* m/z. Calcd. for $C_7H_4N_2O_2$: 148.03; found: 189.1 (M+CH_3CN); *FT-IR (KBr, cm^{-1}):* 3108, 2227, 1602, 1527, 1348, 1290, 858, 680, 749, 565;

k) Characterization of 4-aminobenzonitrile (Table 2, entry 12): Compound is prepared according to the general procedure using BOC protected 4-bromoaniline. After the reaction BOC is deprotected using acid. The resulting crude oily residue was purified by chromatography on neutral aluminium oxide (eluent: petroleum ether/ethyl acetate, 70:30) to provide the desired product as a white solid. Yield 72% (85 mg). *^1H NMR (300 MHz, $CDCl_3$):*4.23 (s, 2H), 6.63 (d, *J*=8.4Hz, 2H), 7.47 (d, *J*=8.4Hz, 2H); *^{13}C NMR (75 MHz, $CDCl_3$):* 100.1, 114.4, 120.1, 133.7, 150.5; *ESI-MS:* m/z. Calcd. for $C_7H_6N_2$: 118.05; found: 116.92 (M-H); *FT-IR (KBr, cm^{-1}):* 3361, 3217, 2212, 1605, 1514, 1484, 1172, 1119, 694, 723, 542.

l) Characterization of 1-cyanonaphthalene (Table 2, entry 13): Compound is prepared according to the general procedure. The resulting crude oily residue was purified by chromatography on silica gel (eluent: petroleum ether/ethyl acetate, 98:02) to provide the desired product as oil. Yield 73% (112 mg). *^{1}H NMR (300 MHz, $CDCl_3$):* 7.50 (t, *J*= 7.8Hz, 1H), 7.56-7.70 (m, 2H), 7.90 (t, *J*=6Hz, 2H), 8.06 (d, *J*=8.6Hz, 1H), 8.22 (d, *J*=8.6Hz, 1H); *^{13}C NMR (75 MHz, $CDCl_3$):* 110.1, 117.7, 124.8, 125.0, 127.5, 128.5, 128.6, 132.3, 132.5, 132.8, 133.2; *ESI-MS:* m/z. Calcd. for $C_{11}H_7N$: 153.06; found: 154.10 (M+H); *FT-IR (KBr, cm^{-1}):* 3060, 2217, 1583, 1506, 1263, 1218;

m) Characterization of 4-(trifluoromethoxy)benzonitrile (Table 2, entry 14): Compound is prepared according to the general procedure. The resulting crude oily residue was purified by chromatography on silica gel (eluent: petroleum ether/ethyl acetate, 90:10) to provide the desired product as a white powder. Yield 93% (174 mg). *^{1}H NMR (300 MHz, $CDCl_3$):* 6.97 (d, *J*=7.1Hz, 2H), 7.70 (d, *J*=7.1Hz, 2H) *^{13}C NMR (75 MHz, $CDCl_3$):* 91.0, 110.1, 118.6, 122.0, 123.0, 138.9, 149.1; *ESI-MS:* m/z. Calcd. for $C_8H_4F_3NO$: 187.02; found: 185.92 (M+H); *FT-IR (KBr, cm^{-1}):* 3054, 2359, 1437, 1187, 1118, 1070;

n) Characterization of thiophene-2-carbonitrile (Table 2, entry 15): Compound is prepared according to the general procedure. The resulting crude oily residue was purified by chromatography on silica gel (eluent: petroleum ether/ethyl acetate, 95:5) to provide the desired product as a colourless oil. Yield 77% (84 mg). *^{1}H NMR (300 MHz, $CDCl_3$):* 7.90 (t, *J*=6.3Hz, 1H), 8.06 (d, *J*=8.4 Hz, 1H), 8.22 (d, *J*=9.9Hz, 1H);

^{13}C NMR (75 MHz, $CDCl_3$): 117.7, 124.8, 128.5, 132.5, 133.2**;** *ESI-MS:* m/z. Calcd. for C_5H_3NS: 108.92; found: 147.47 (M+K);

o) Characterization of 4-cyanopyridine(Table 2, entry 16): Compound is prepared according to the general procedure. The resulting crude oily residue was purified by chromatography on neutral alumina (eluent: petroleum ether/ethyl acetate, 95:5) to provide the desired product as a colourless solid. Yield 71% (74 mg). *^{1}H NMR (300 MHz, $CDCl_3$):* 7.53 (d, *J*=7.5Hz, 2H), 8.81 (d, *J*=7.5Hz, 2H); *^{13}C NMR (75 MHz, $CDCl_3$):* 116.5, 120.6, 125.3, 150.9; *ESI-MS:* m/z. Calcd. for $C_6H_4N_2$: 104.04; found: 103.38 (M-H); *FT-IR (KBr, cm^{-1}):* 3080, 3026, 2240, 1592, 1543, 1495, 1414, 1206;

p) Characterization of 3-cyanopyridine: (Table 2, entry 17): Compound is prepared according to the general procedure. The resulting crude oily residue was purified by chromatography on neutral alumina (eluent: petroleum ether/ethyl acetate, 95:5) to provide the desired product as a solid. Yield 69% (72 mg). *^{1}H NMR (300 MHz, $CDCl_3$):* 7.05 (s, 1H), 7.65-7.69 (m, 1H), 8.38-8.56 (m, 2H); *^{13}C NMR (75 MHz, $CDCl_3$):* 114.4, 120.6, 124.5, 138.3, 147.5, 150.6; *ESI-MS:* m/z. Calcd. for $C_6H_4N_2$: 104.04; found: 127.00 (M+Na);

FT-IR (KBr, cm^{-1}): 3089, 3023, 2237, 1972, 1722, 1589, 1542, 1490, 1413, 1201;

q) Characterization of 2-cyano-5-methylpyridine (Table 2, entry 18): Compound is prepared according to the general procedure. The resulting crude oily residue was

purified by chromatography on neutral alumina (eluent: petroleum ether/ethyl acetate, 93:7) to provide the desired product as a light yellow solid. Yield 83% (98 mg). *^{1}H NMR (300 MHz, $CDCl_3$):* 2.44 (s, 3H), 7.63 (s, 2H); 8.56 (s, 1H); *^{13}C NMR (75 MHz, $CDCl_3$):*18.7, 117.9, 127.4, 131.3, 137.3, 141.6, 151.7; *ESI-MS:* m/z. Calcd. for $C_7H_6N_2$: 118.05; found: 118.98 (M+H);

r) Characterization of 2-amino-4-(4-cyanophenyl)-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile (Table 2, entry 20): Compound is prepared according to the general procedure The resulting crude residue was purified by chromatography on neutral alumina (eluent: petroleum ether/ethyl acetate, 70:30) to provide the desired product as a solid. Yield 81% (237 mg). *^{1}H NMR (300 MHz, DMSO-d_6, $CDCl_3$):* 1.85 (m, 2H), 2.16 (m, 2H), 2.43 (m, 2H), 4.26 (s, 1H), 5.89 (s, 2H), 7.28 (d, 2H), 7.85 (d, 2H); *^{13}C NMR (75 MHz, $CDCl_3$):* 19.8, 26.9, 35.1, 36.4, 58.6, 113.5, 118.8, 124.5, 124.9, 131.5, 132.7, 144.4, 164.3, 195.8; *ESI-MS:* m/z. Calcd. for $C_{17}H_{13}N_3O_2$: 293.10; found: 294.08 (M+H);

s) Characterization of 4-(4-oxo-4*H*-chromen-2-yl)benzonitrile (Table 2, entry 21): Compound is prepared according to the general procedure. The resulting crude residue was purified by chromatography on neutral alumina (eluent: petroleum ether/ethyl acetate, 70:30) to provide the desired product as a yellow solid. Yield 73% (180 mg). *^{1}H NMR (300 MHz, $CDCl_3$):* 6.75 (s, 1H); 7.01 (d, J=7.2Hz, 2H); 7.44 (dd, *J*=6.6 Hz, *J*=1.5Hz, 1H); 7.61 (d, *J*=7.2Hz, 1H);7.71 (dd, *J*=6.6Hz, *J*=1.6Hz, 1H);7.93 (d, *J*=15.6Hz, 2H);8.23 (d, *J*=7.8Hz, 1H); *ESI-MS:* m/z. Calcd. for $C_{16}H_9NO_2$: 247.36; found: 245.99 (M-H);

3. NMR, ESI-MS and IR spectra of aryl nitriles:

3.1 NMR spectra of ligands:

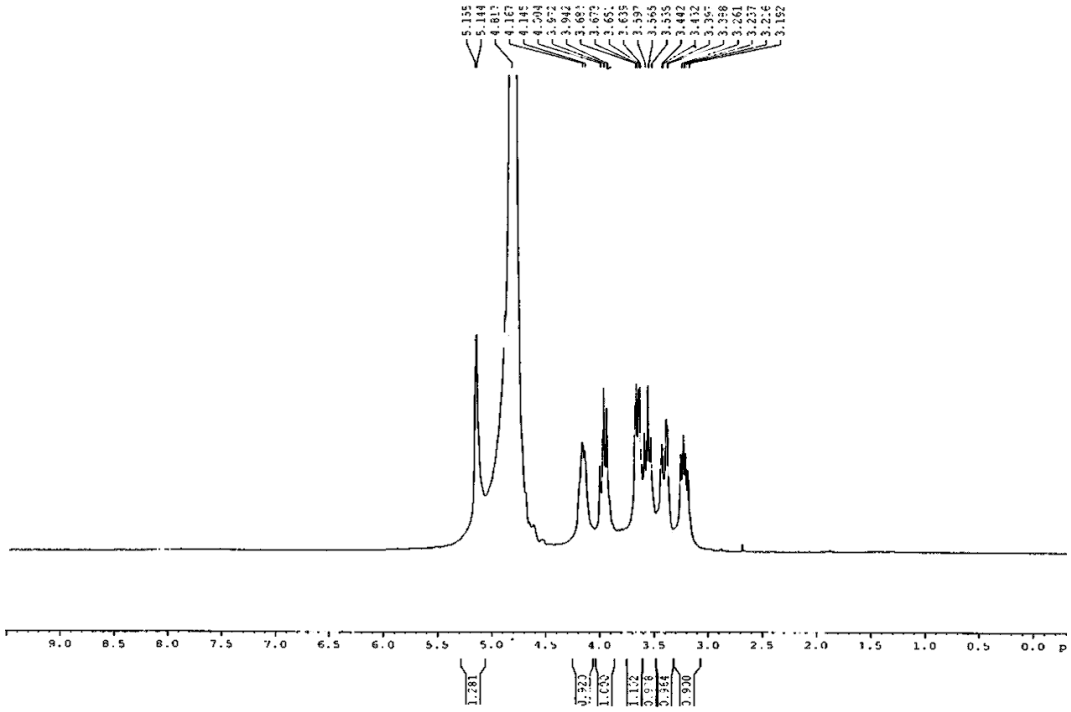

Fig.1 *^{1}H NMR of per-6-amino-β-cyclodextrin (*per-6-NH_2-β-CD*)*

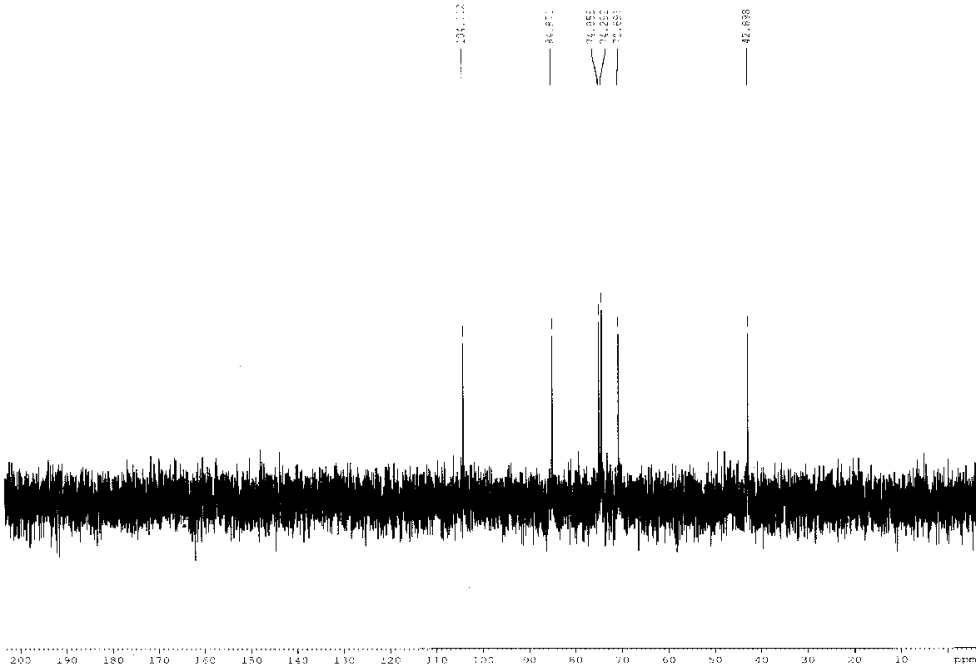

Fig.2 *^{13}C NMR of per-6-amino-β-cyclodextrin (*per-6-NH_2-β-CD*)*

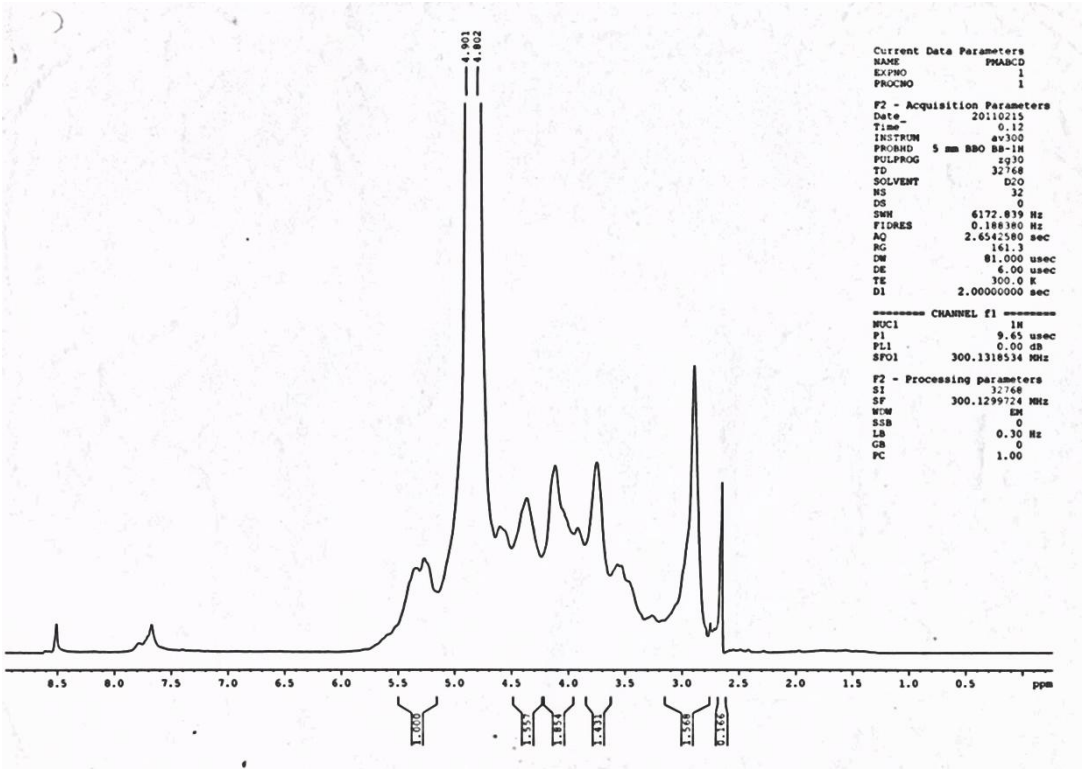

Fig. 3 *^{1}H NMR spectrum of per-6-methylamino-β-cyclodextrin (*per-6-MeNH-β-CD*)*

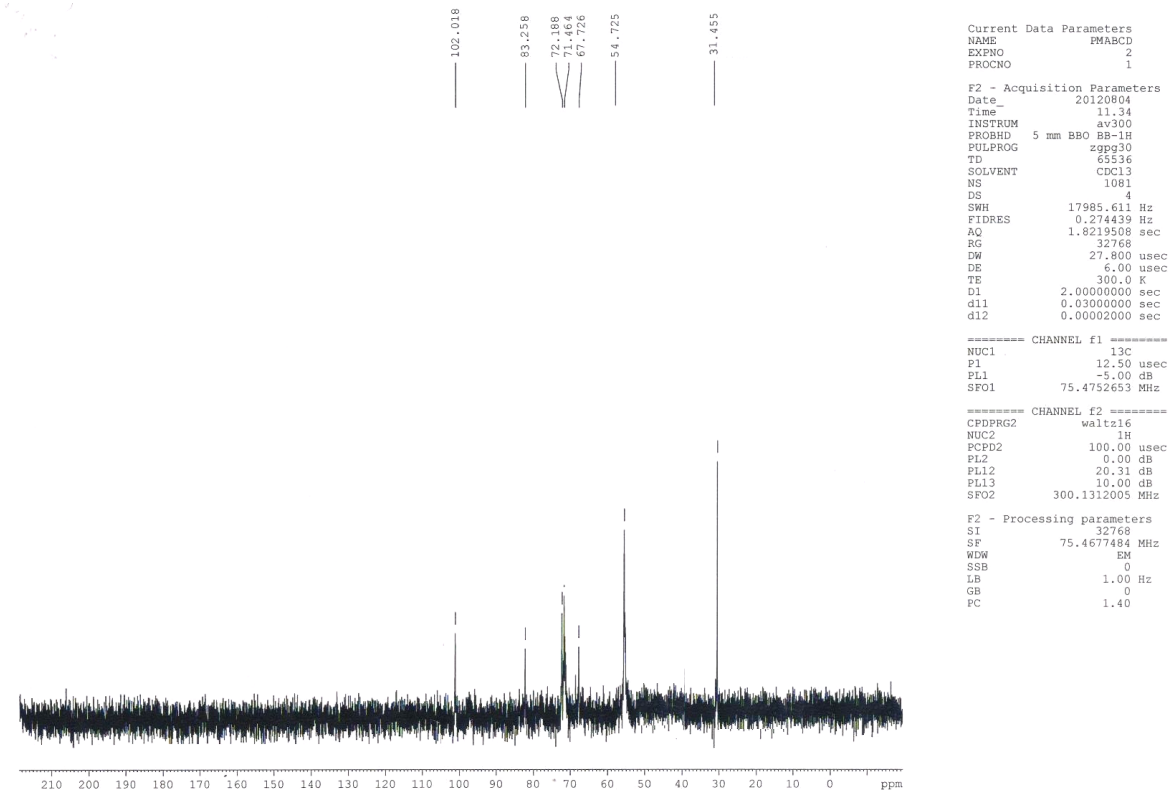

Fig. 4 *^{1}C NMR spectrum of per-6-methylamino-β-cyclodextrin (*per-6-MeNH-β-CD*)*

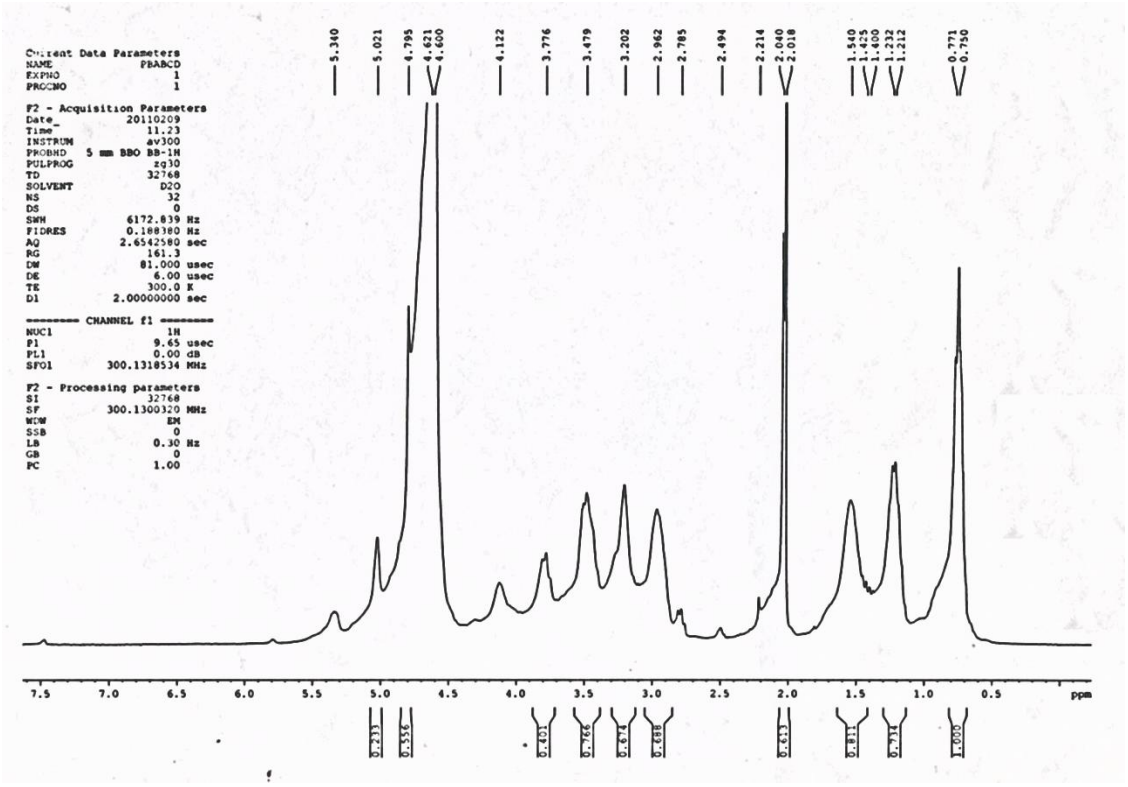

Fig. 5 *^{1}H NMR spectrum of per-6-butylamino-β-cyclodextrin (*per-6-*n*-BuNH-β-CD*)*

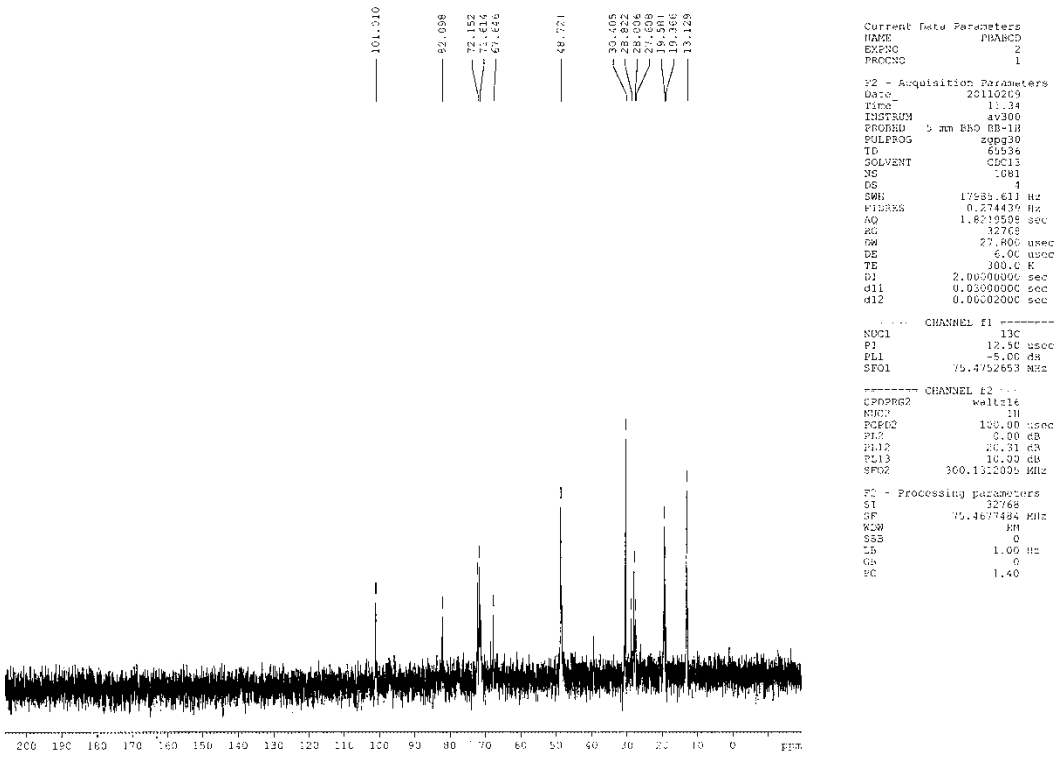

Fig. 6 *^{13}C NMR spectrum of per-6-butylamino-β-cyclodextrin (*per-6-*n*-BuNH-β-CD*)*

3.2 NMR spectra of aryl nitriles:

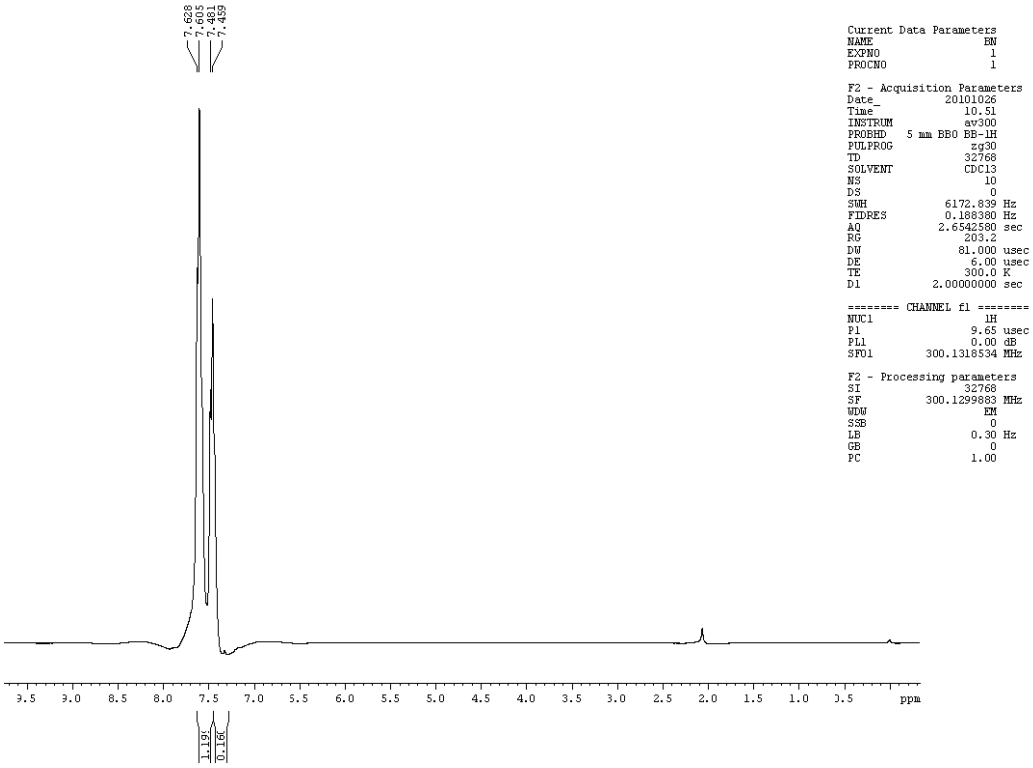

Fig: 7 ^{1}H-NMR of benzonitrile

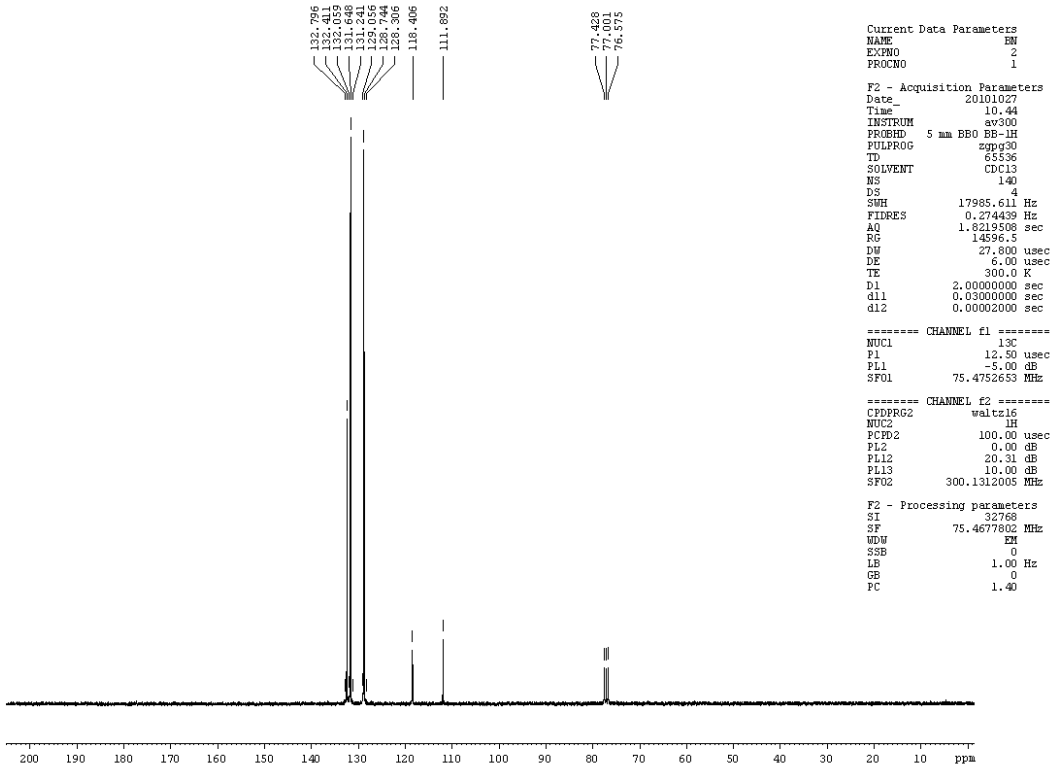

Fig: 8 ^{13}C-NMR of benzonitrile

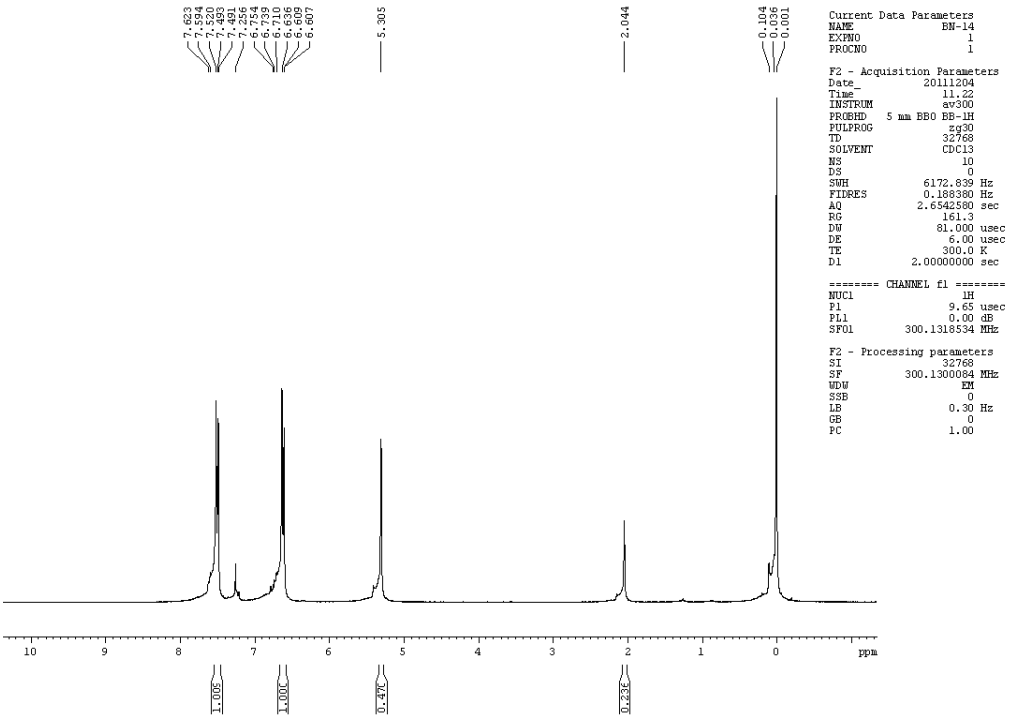

Fig: 9 ^{1}H NMR spectrum of 4-hydroxybenzonitrile

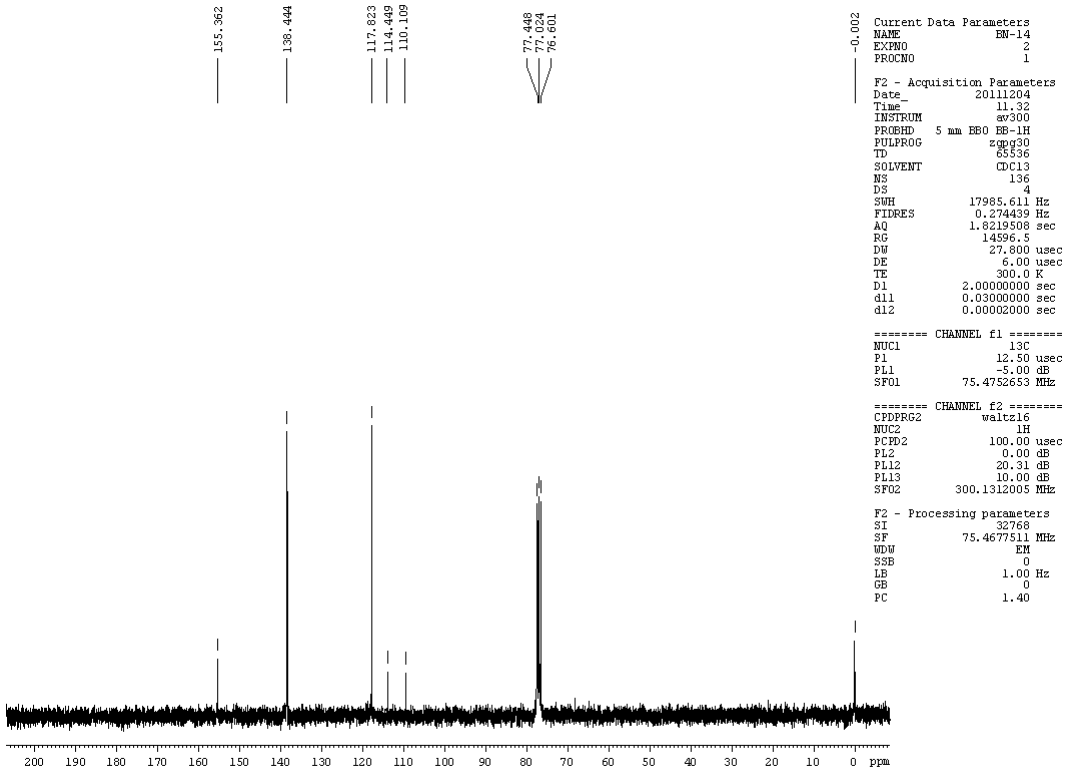

Fig: 10 ^{13}C NMR spectrum of 4-hydroxybenzonitrile

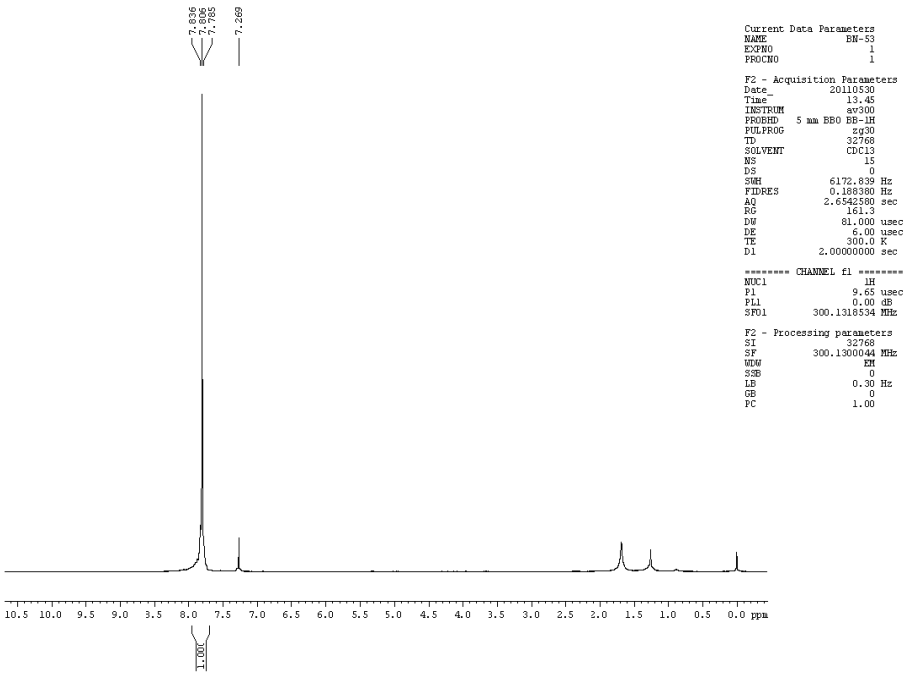

Fig: 11 ^{1}H-NMR of 1,4-dicyanobenzene

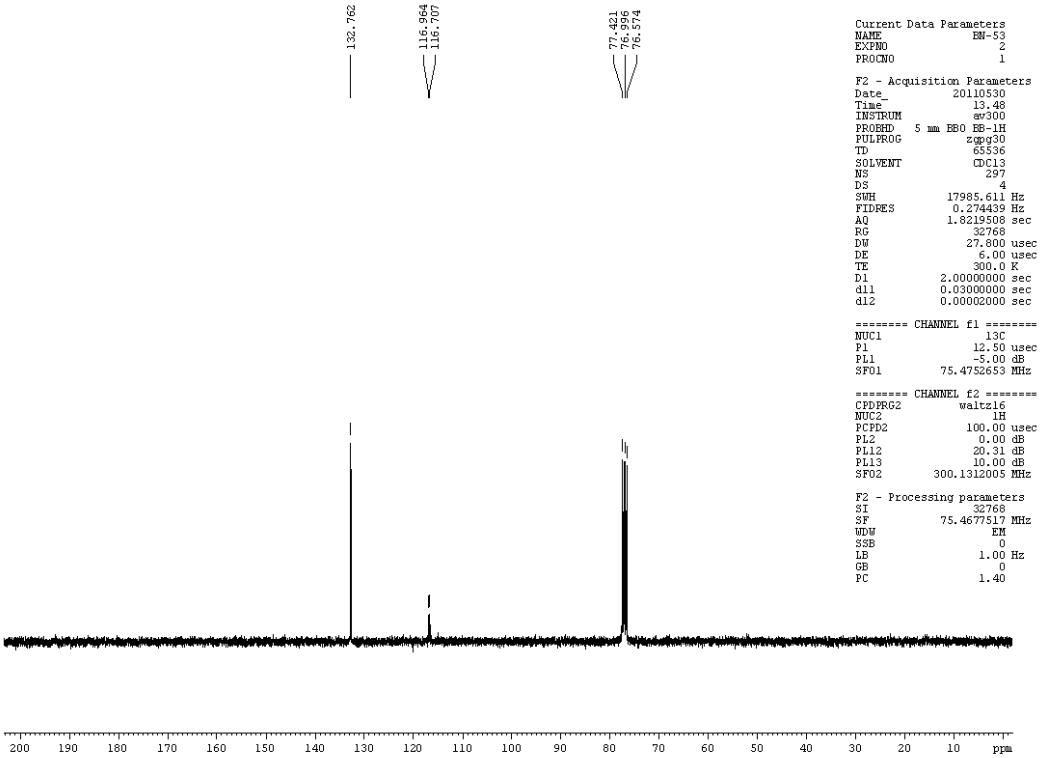

Fig:12 ^{13}C-NMR of 1,4-dicyanobenzene

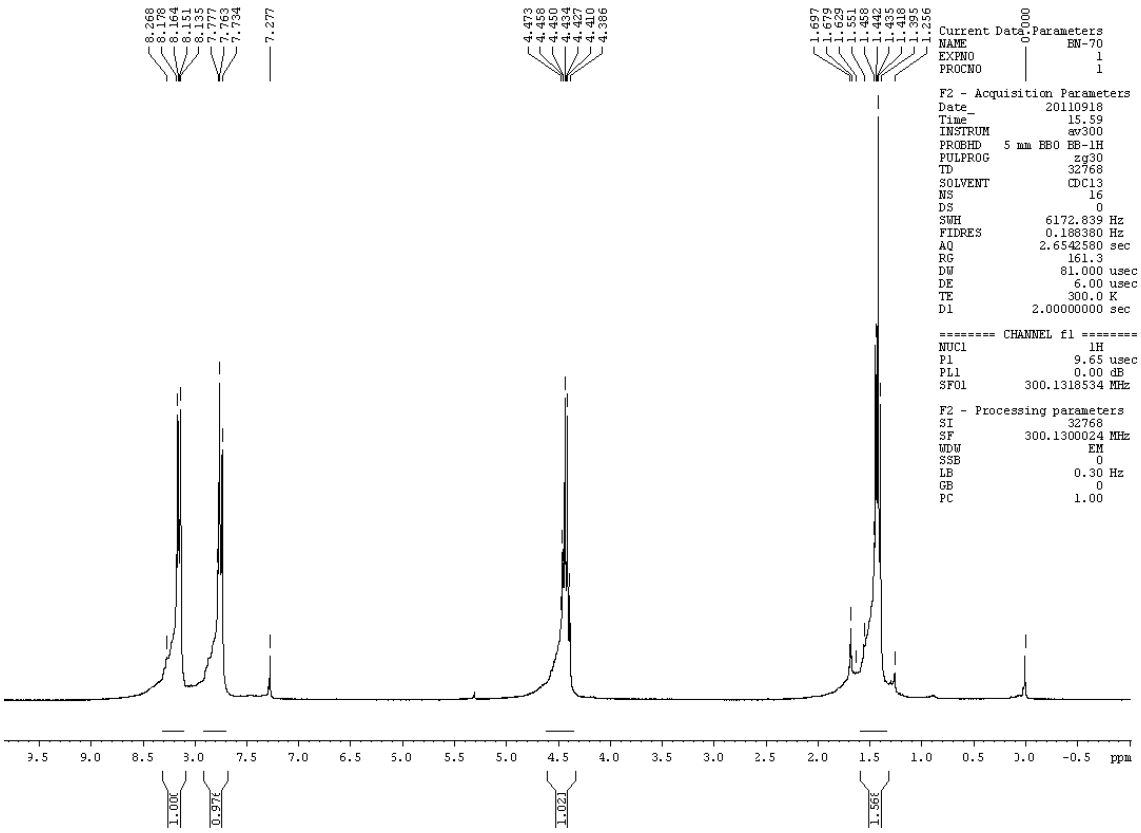

Fig: 13 ^{1}H-NMR of ethyl 4-cyanobenzoate

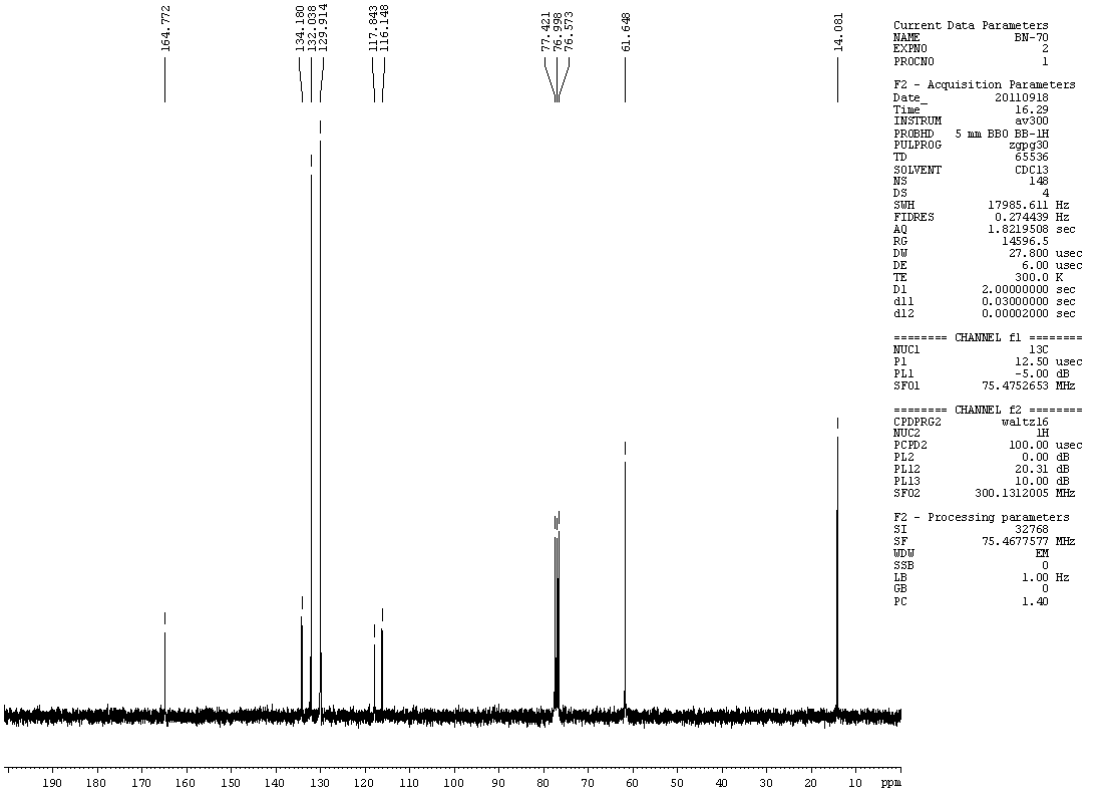

Fig:14 ^{13}C-NMR of ethyl 4-cyanobenzoate

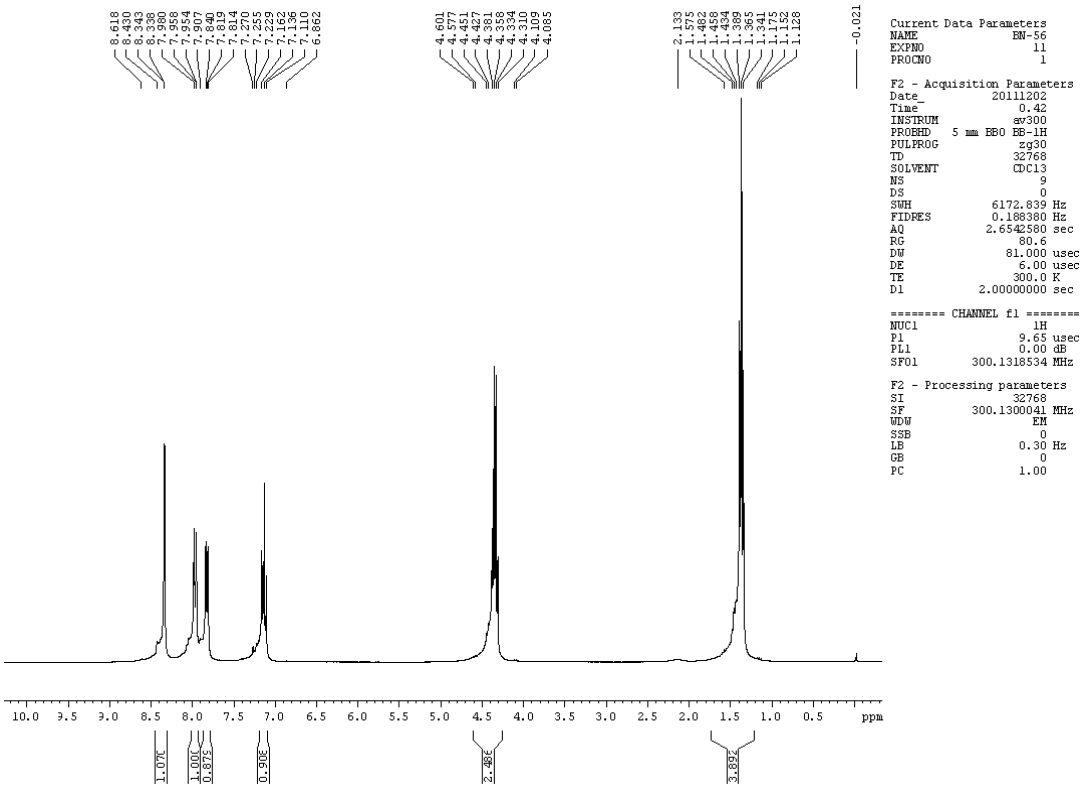

Fig: 15 ^{1}H-NMR of ethyl 3-cyanobenzoate

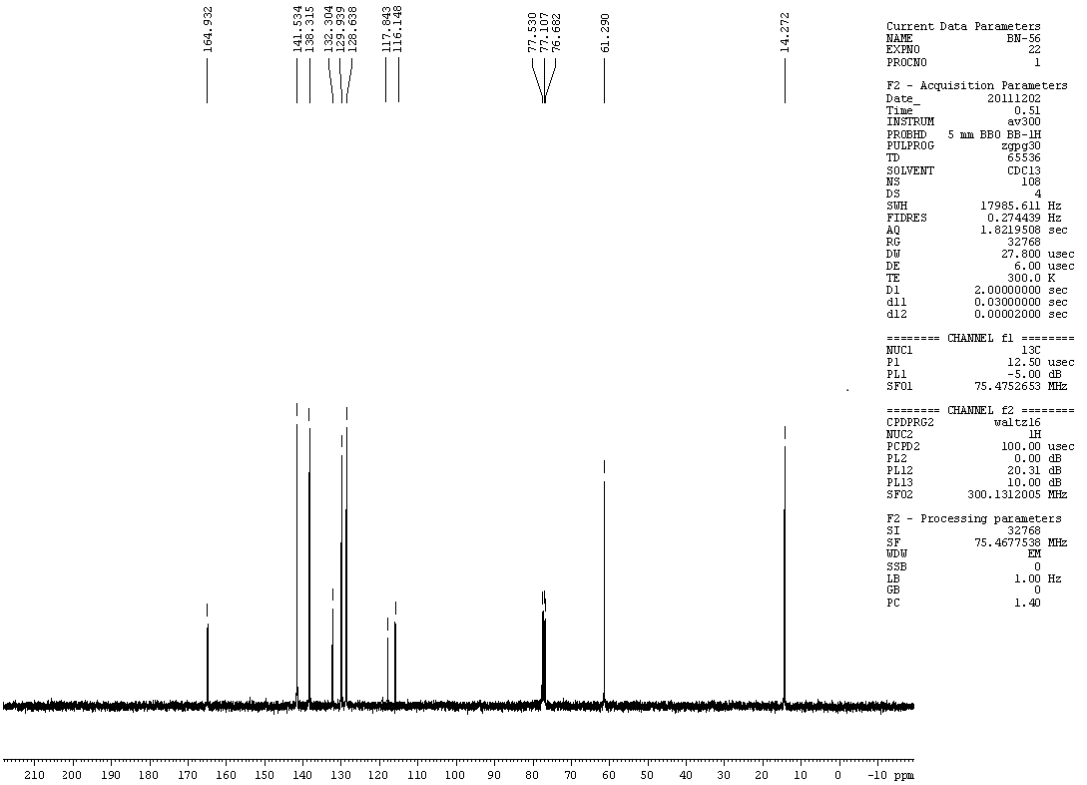

Fig: 16 ^{13}C-NMR of ethyl 3-cyanobenzoate

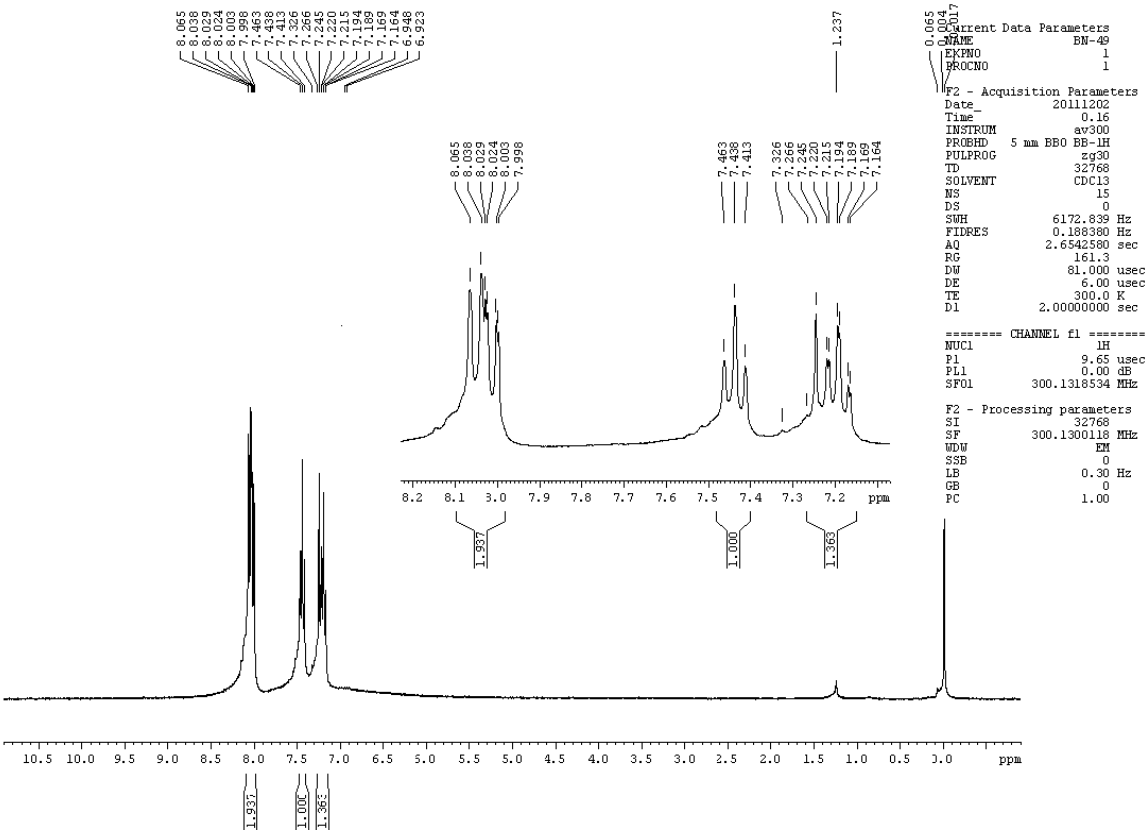

Fig: 17 ^{1}H-NMR of 2-cyanobenzoic acid

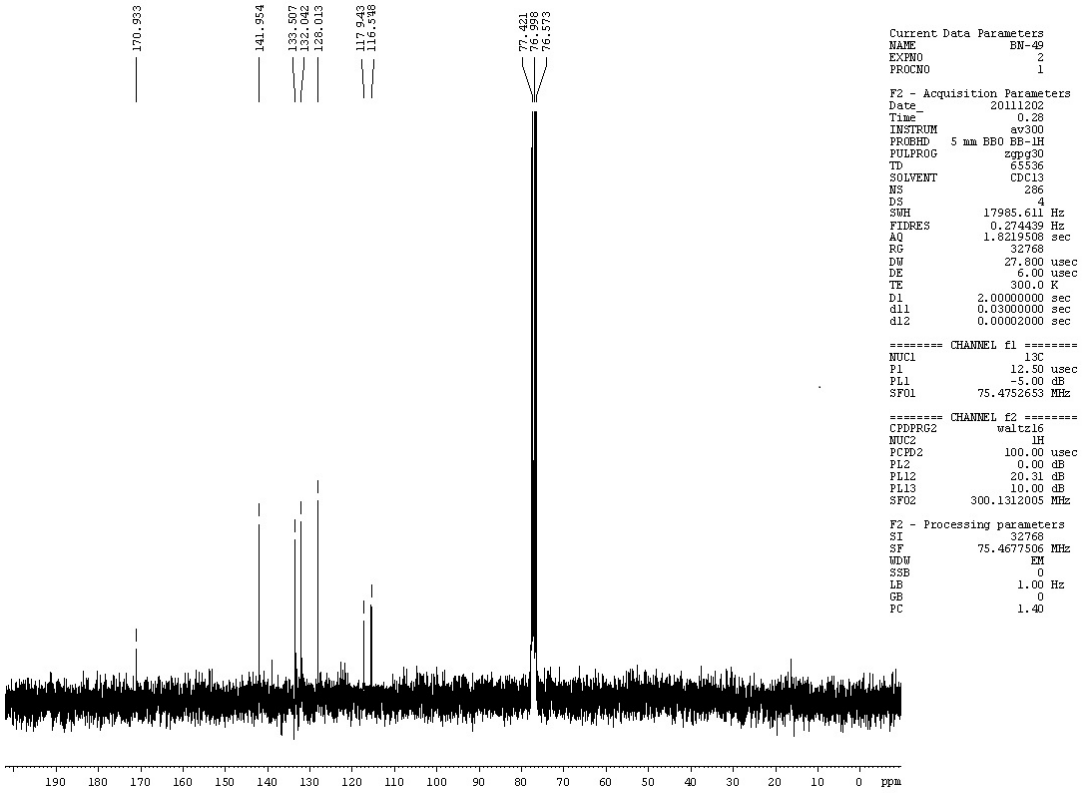

Fig: 18 ^{13}C-NMR of 2-cyanobenzoic acid

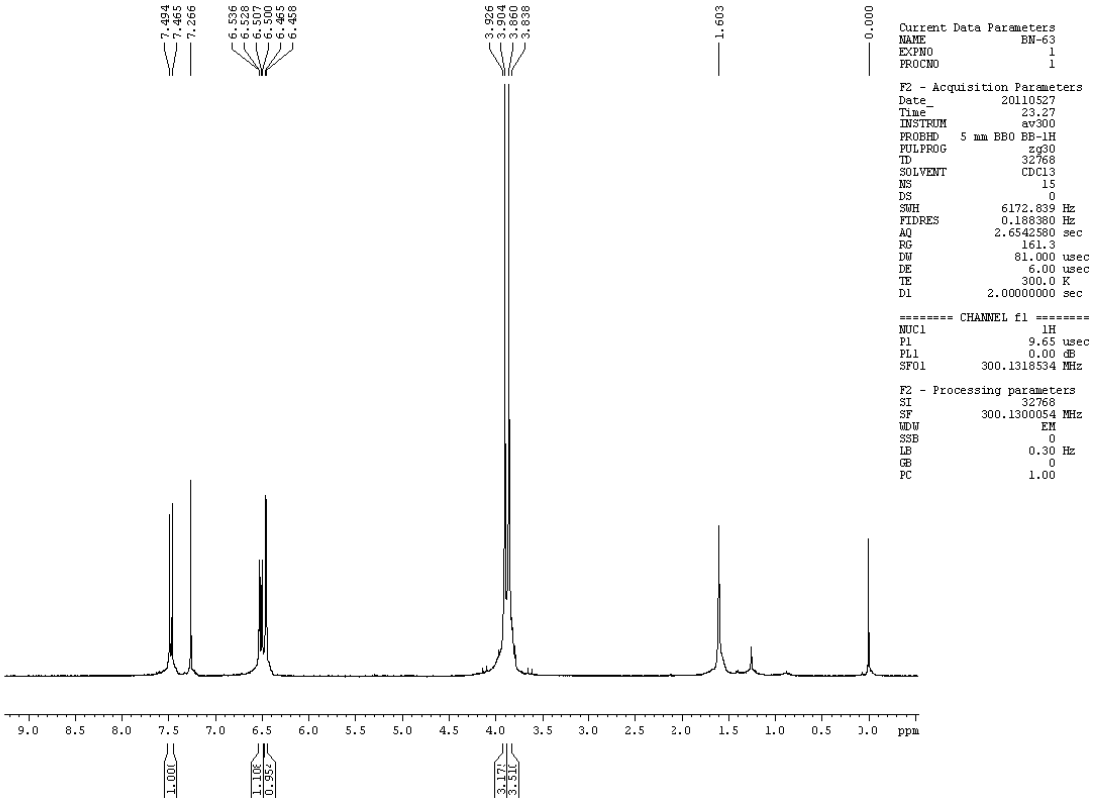

Fig: 19 ^{1}H-NMR of 2,4-dimethoxybenzonitrile

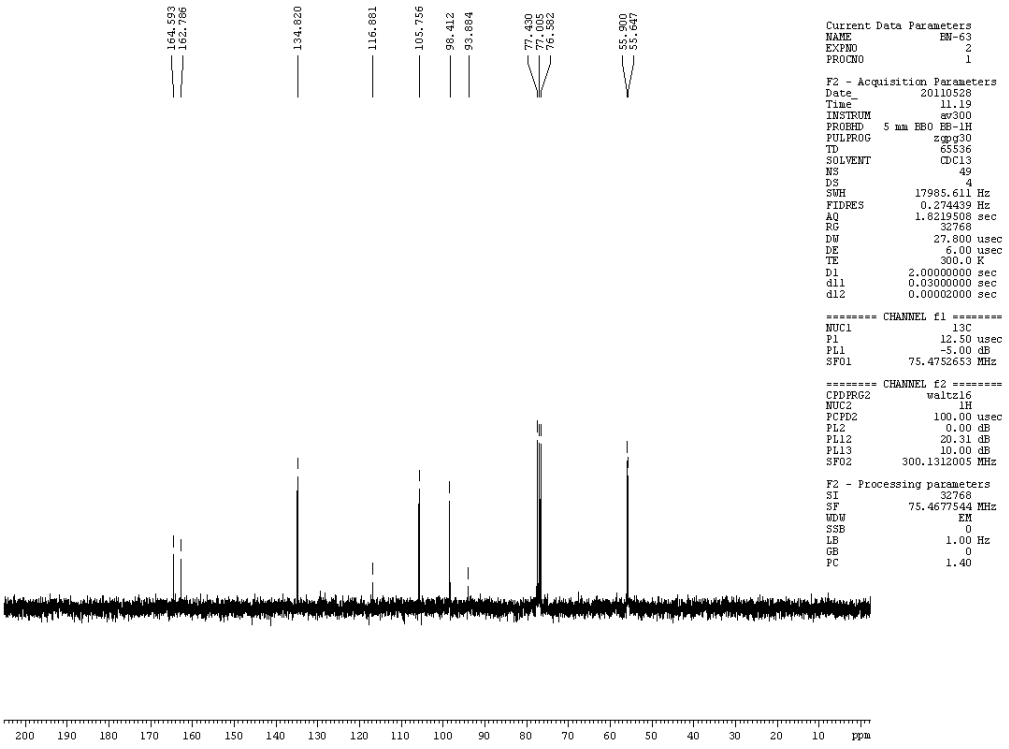

Fig: 20 ^{13}C-NMR of 2,4-dimethoxybenzonitrile

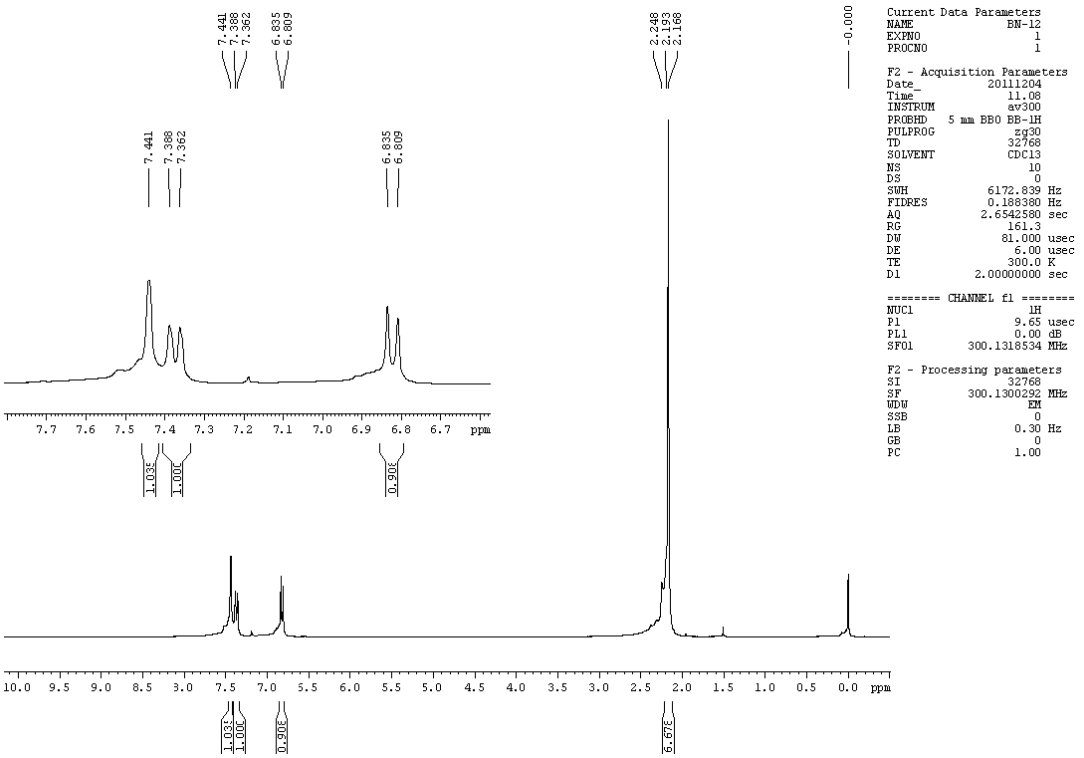

Fig: 21 ^{1}H-NMR of 3,4-dimethylbenzonitrile

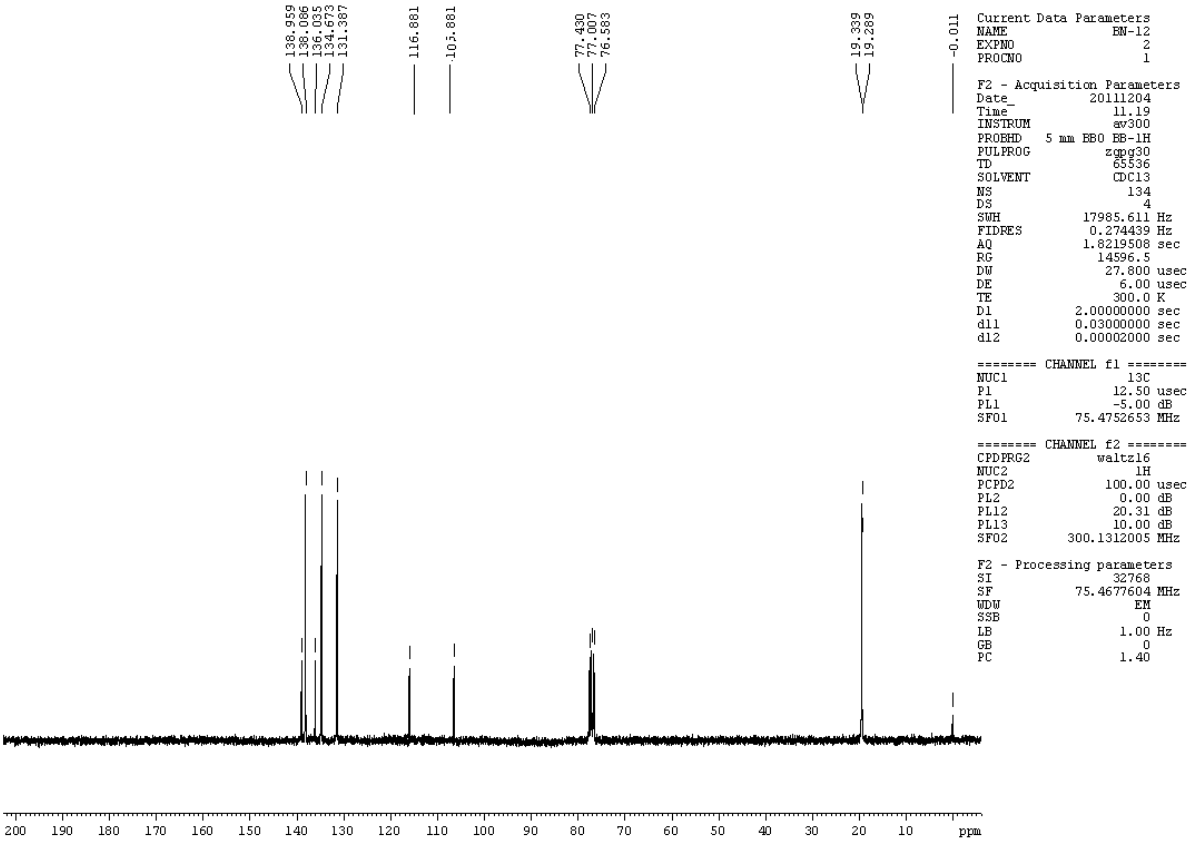

Fig: 22 ^{13}C-NMR of 3,4-dimethylbenzonitrile

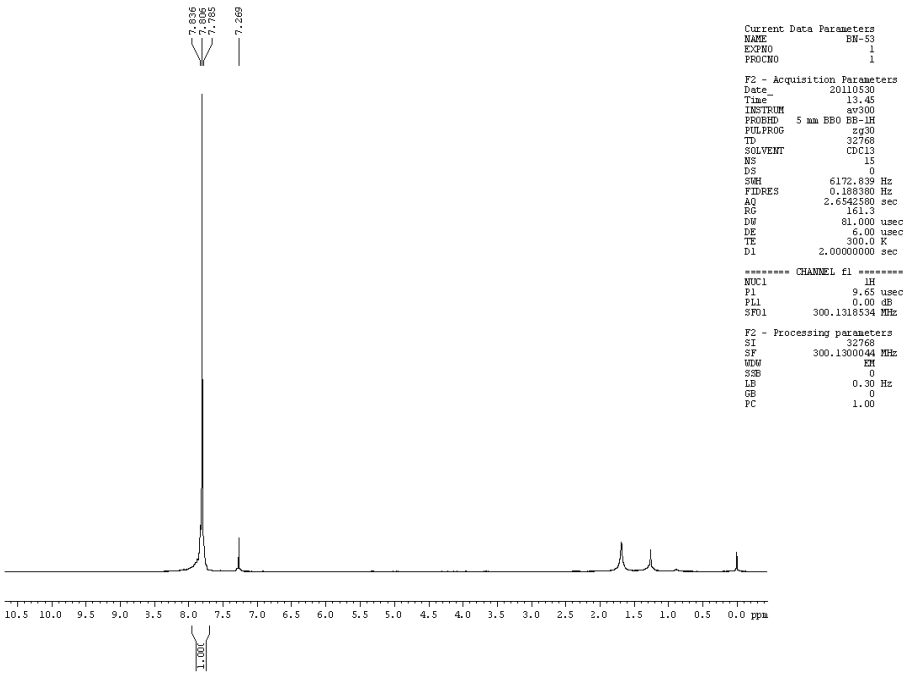

Fig: 23 ^{1}H-NMR of 1,4-dicyanobenzene

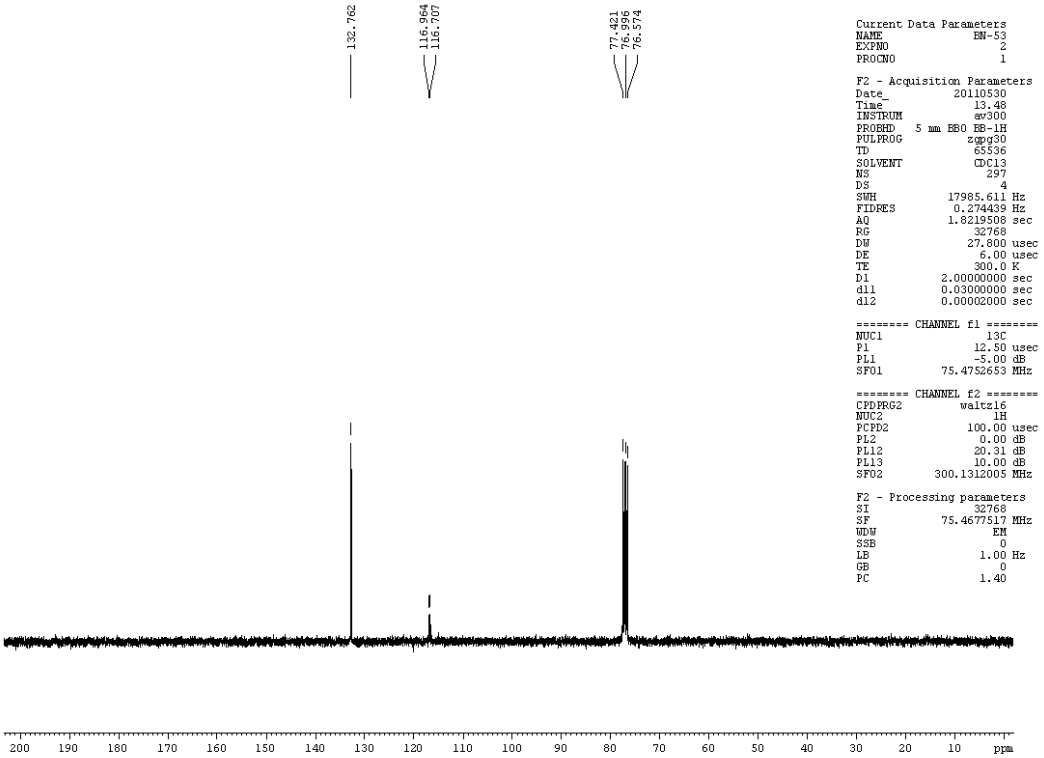

Fig: 24 ^{13}C-NMR of 1,4-dicyanobenzene

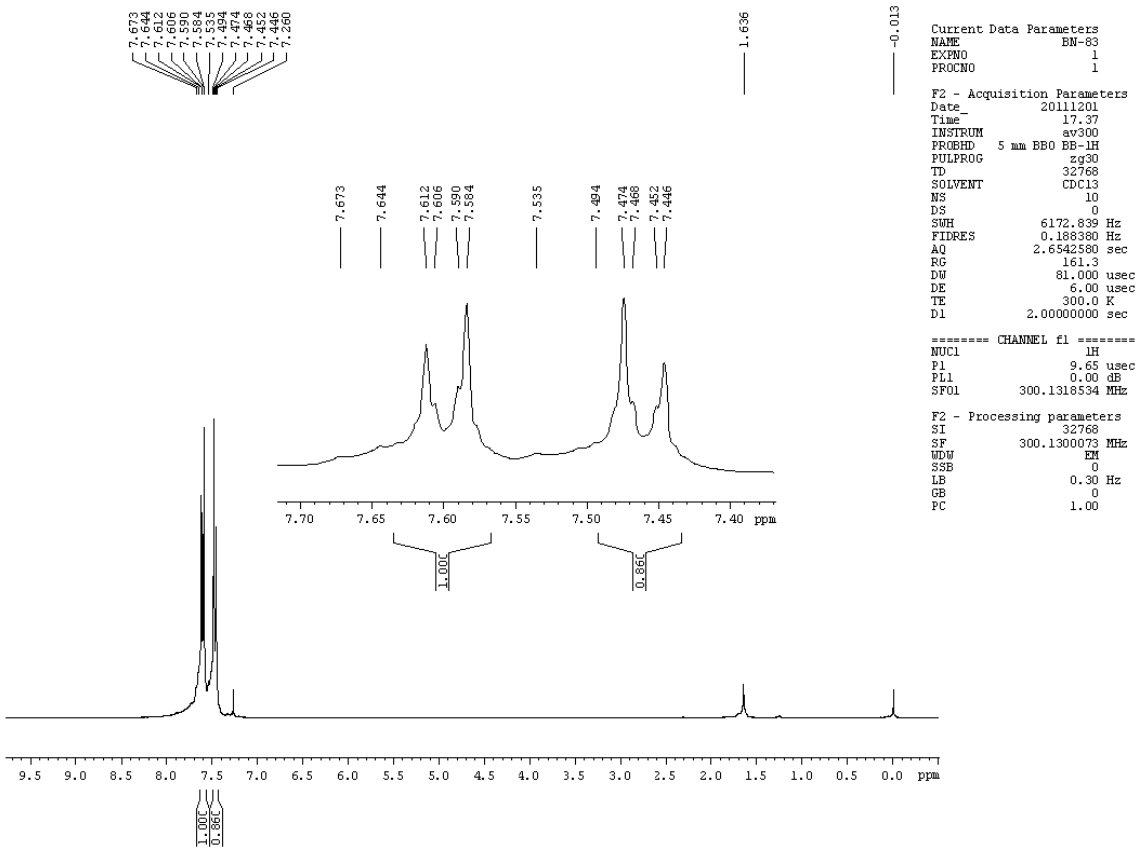

Fig: 25 ^{1}H-NMR of 4-chlorobenzonitrile

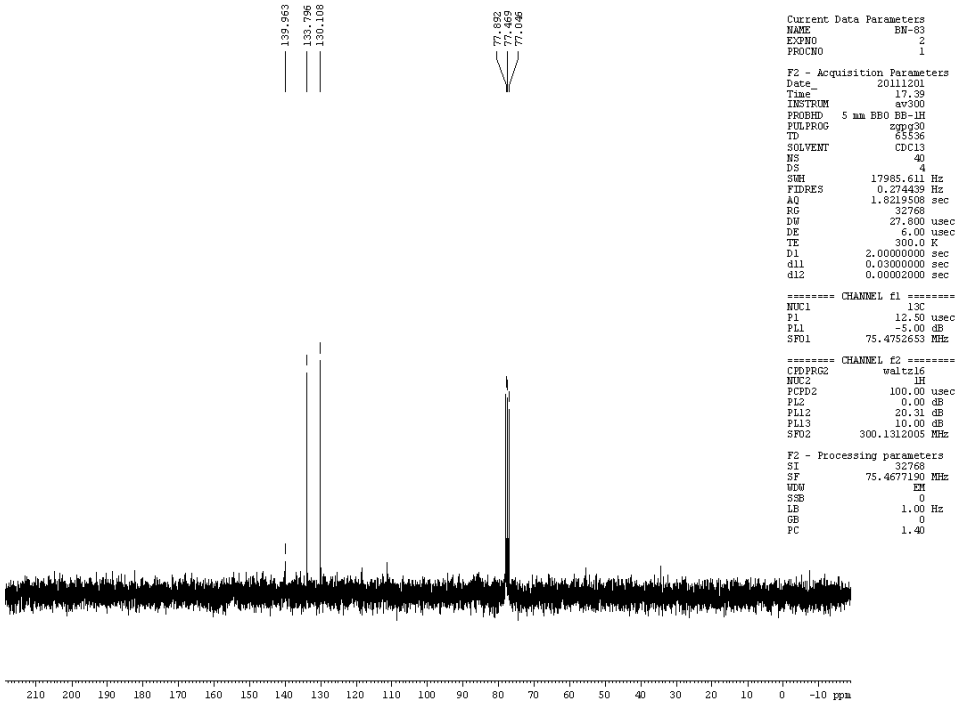

Fig: 26 ^{13}C-NMR of 4-chlorobenzonitrile

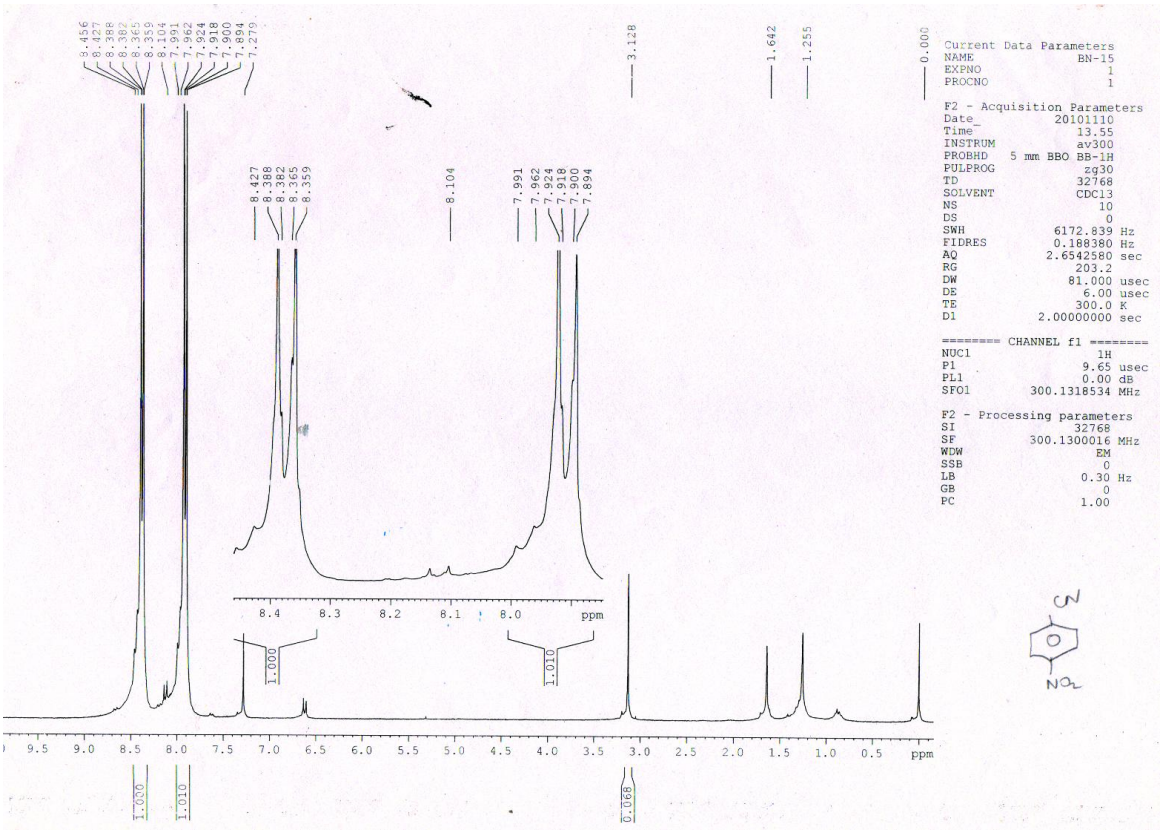

Fig: 27 ^{1}H-NMR of 4-nitrobenzonitrile

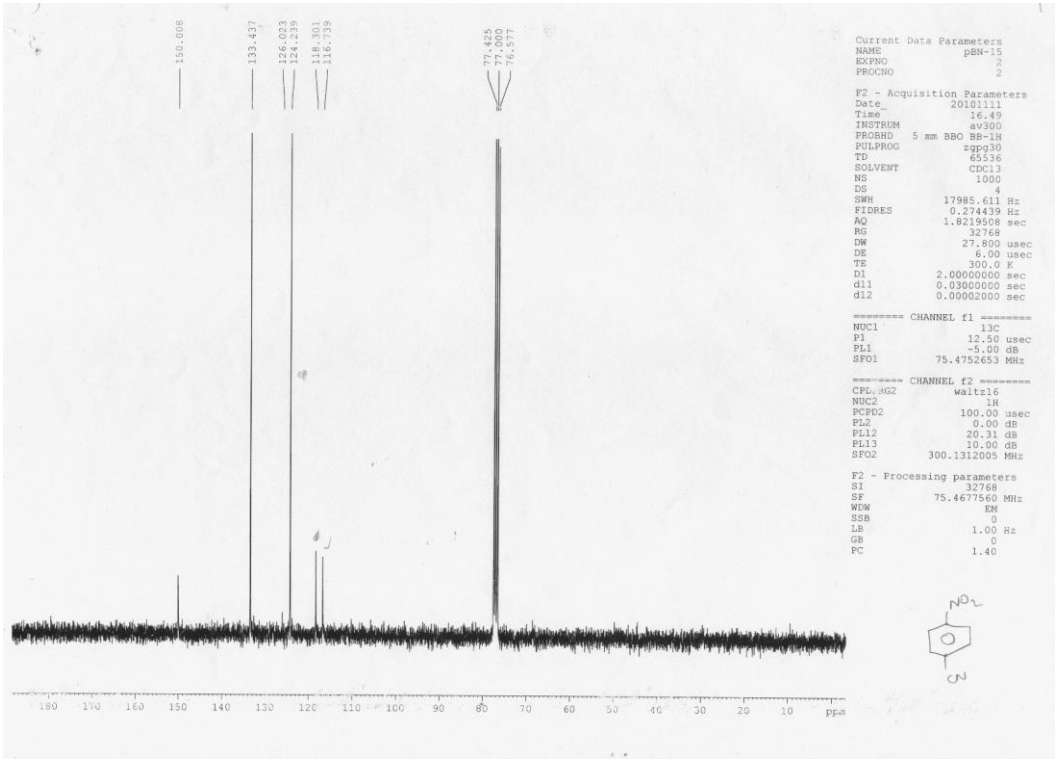

Fig: 28 ^{13}C-NMR of 4-nitrobenzonitrile

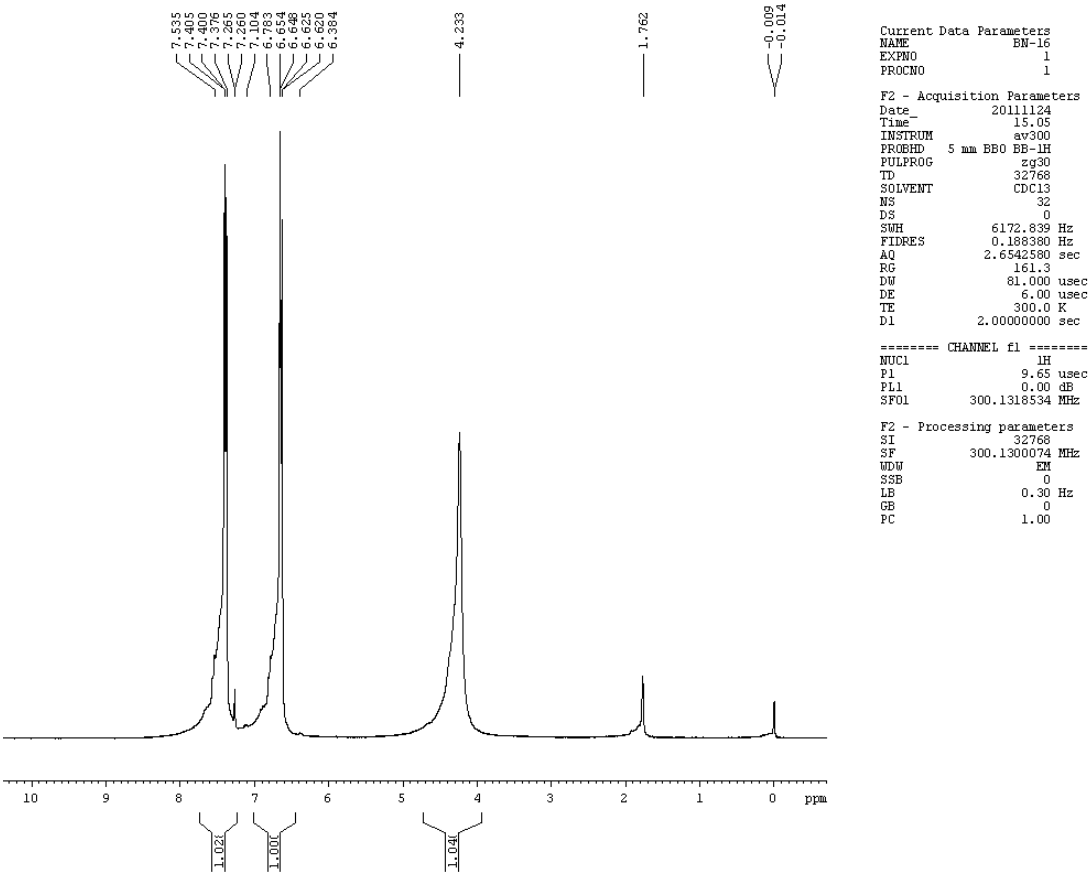

Fig: 29 ^{1}H NMR spectrum of 4-aminobenzonitrile

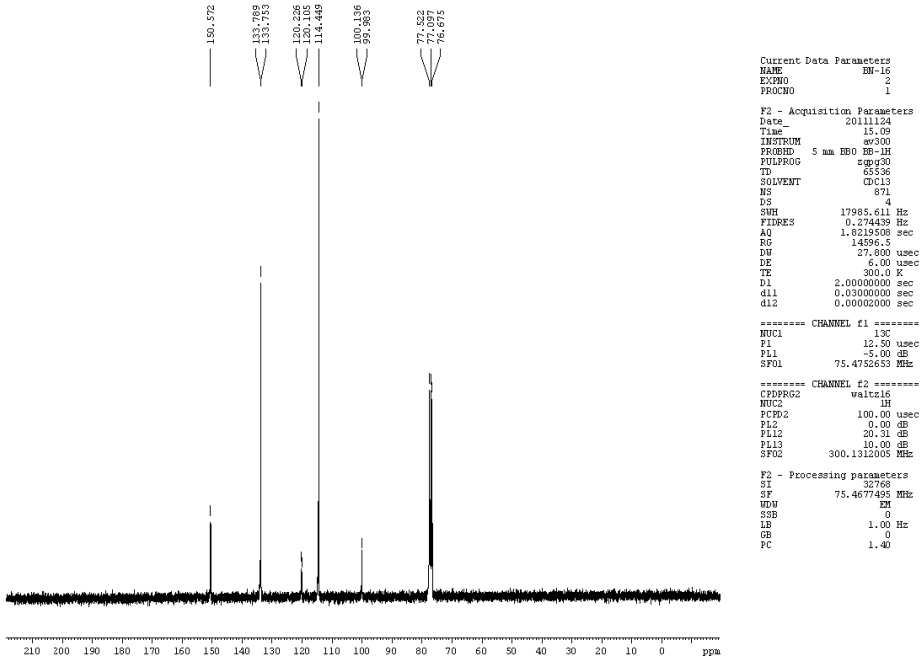

Fig: 30 ^{13}C NMR spectrum of 4-aminobenzonitrile

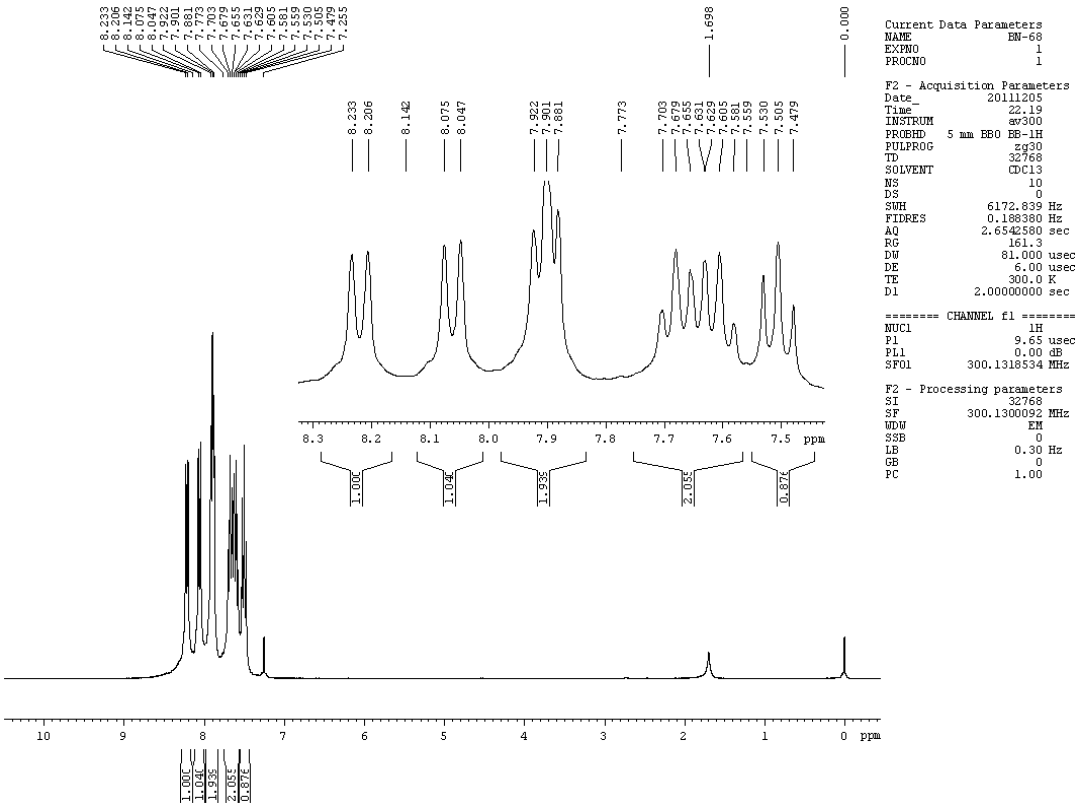

Fig: 31 ^{1}H-NMR of 1-cyanonaphthalene

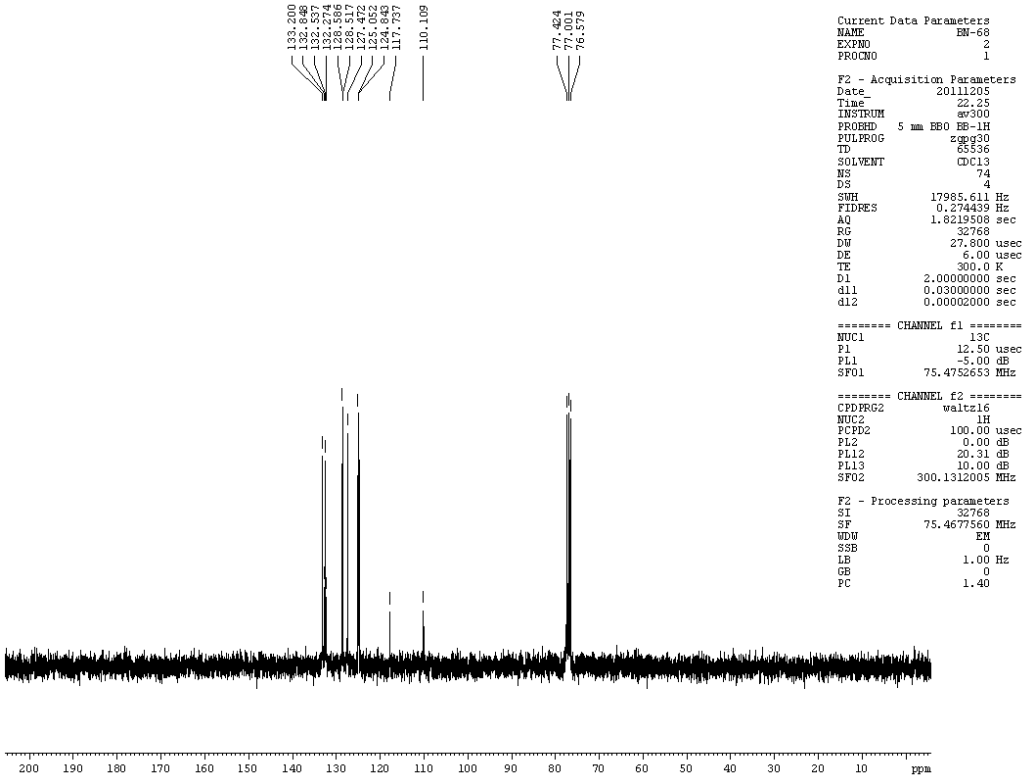

Fig: 32 ^{13}C-NMR of 1-cyanonaphthalene

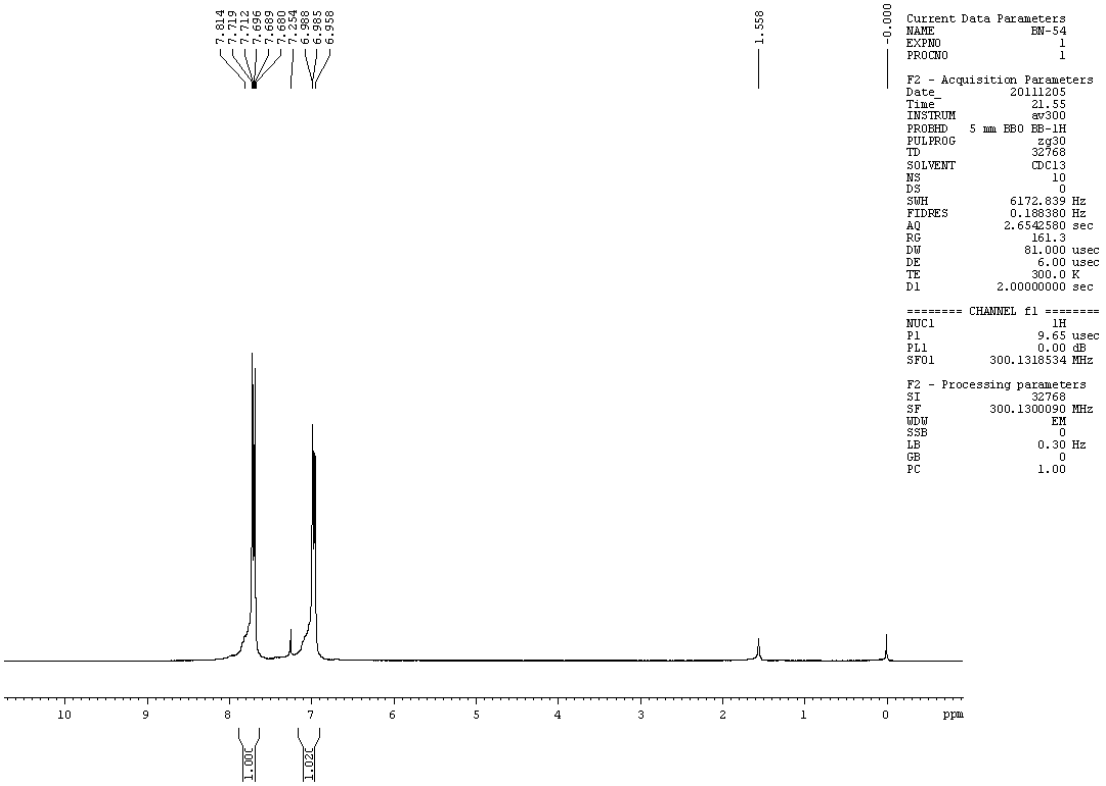

Fig: 33 ^{1}H-NMR of 4-(trifluoromethoxy)cyanobenzene

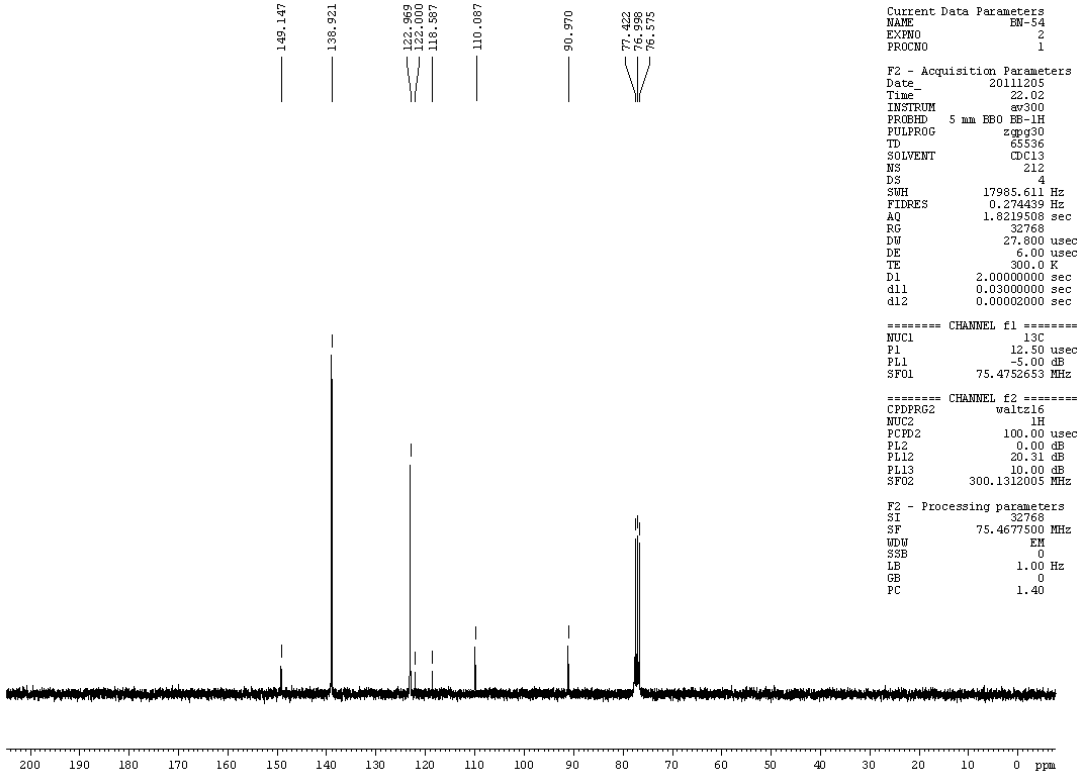

Fig: 34 ^{13}C-NMR of 4-(trifluoromethoxy)cyanobenzene

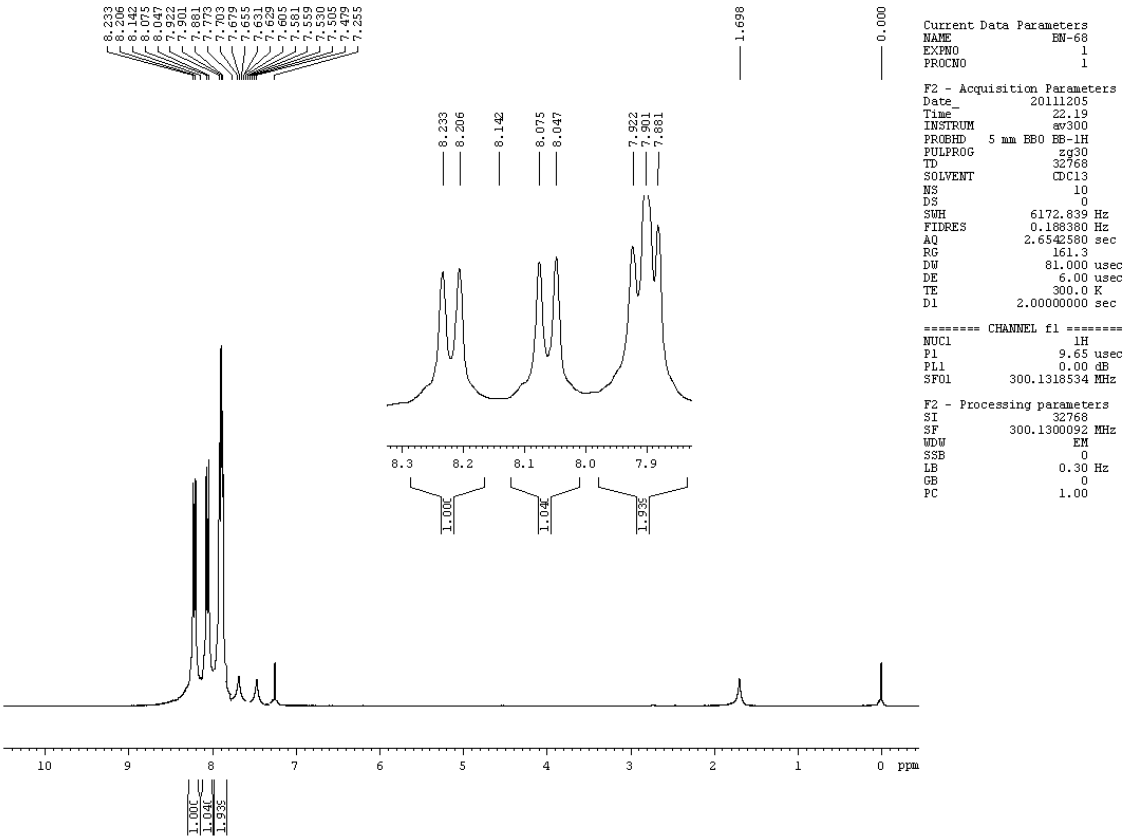

Fig: 35 ^{1}H-NMR of thiophene-2-carbonitrile

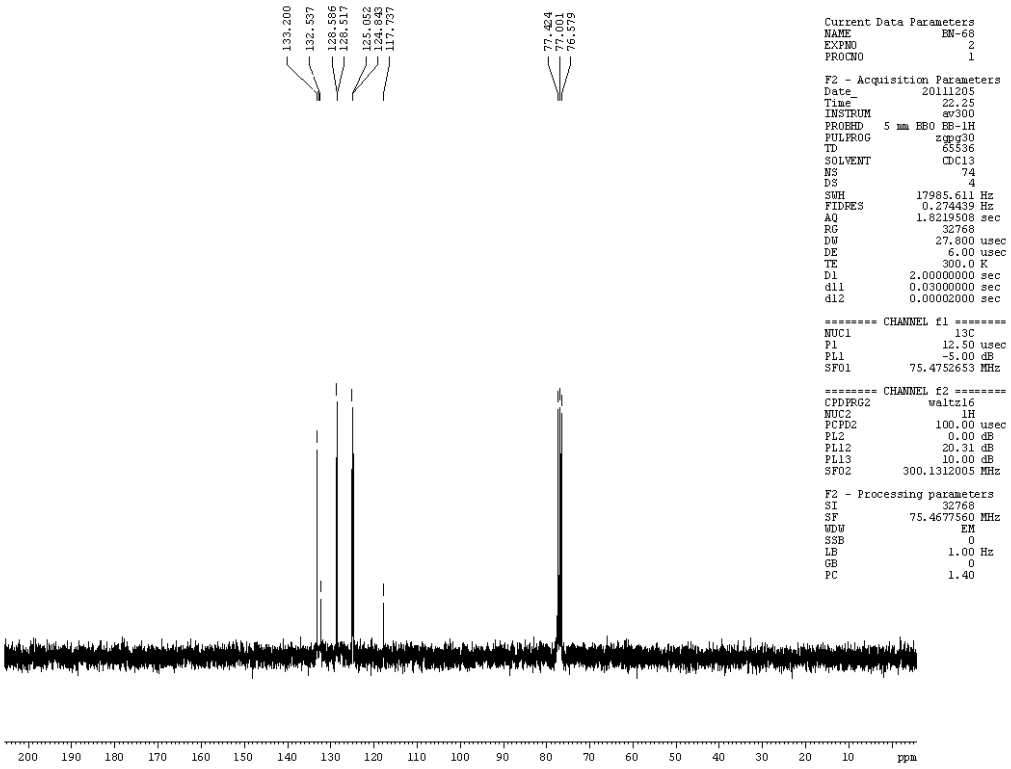

Fig: 36 ^{13}C-NMR of thiophene-2-carbonitrile

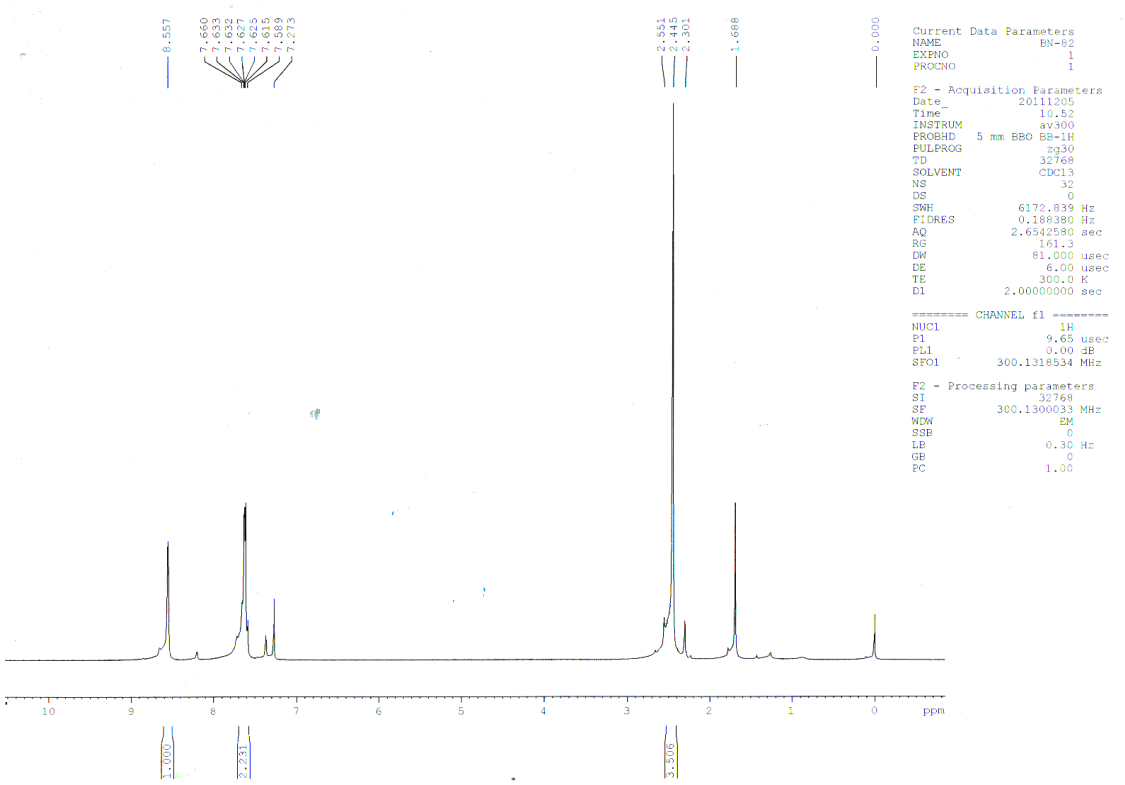

Fig: 37 ^{1}H-NMR of 2-cyano-5-methylpyridine

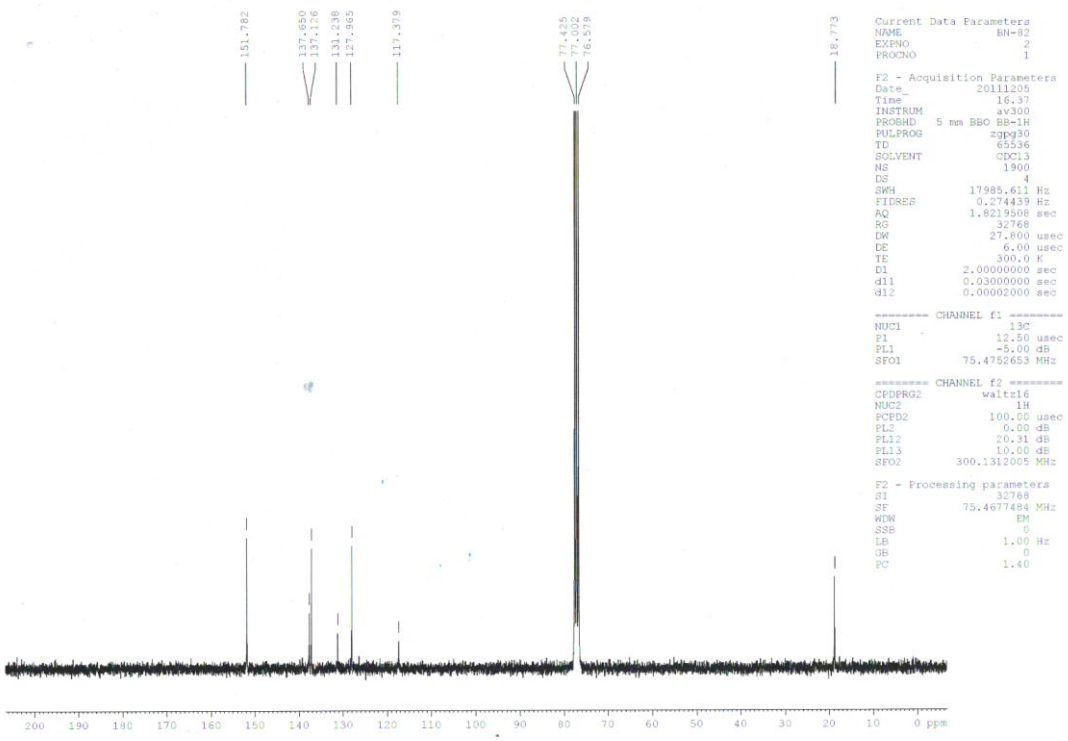

Fig:38 ^{13}C-NMR of 2-cyano-5-methylpyridine

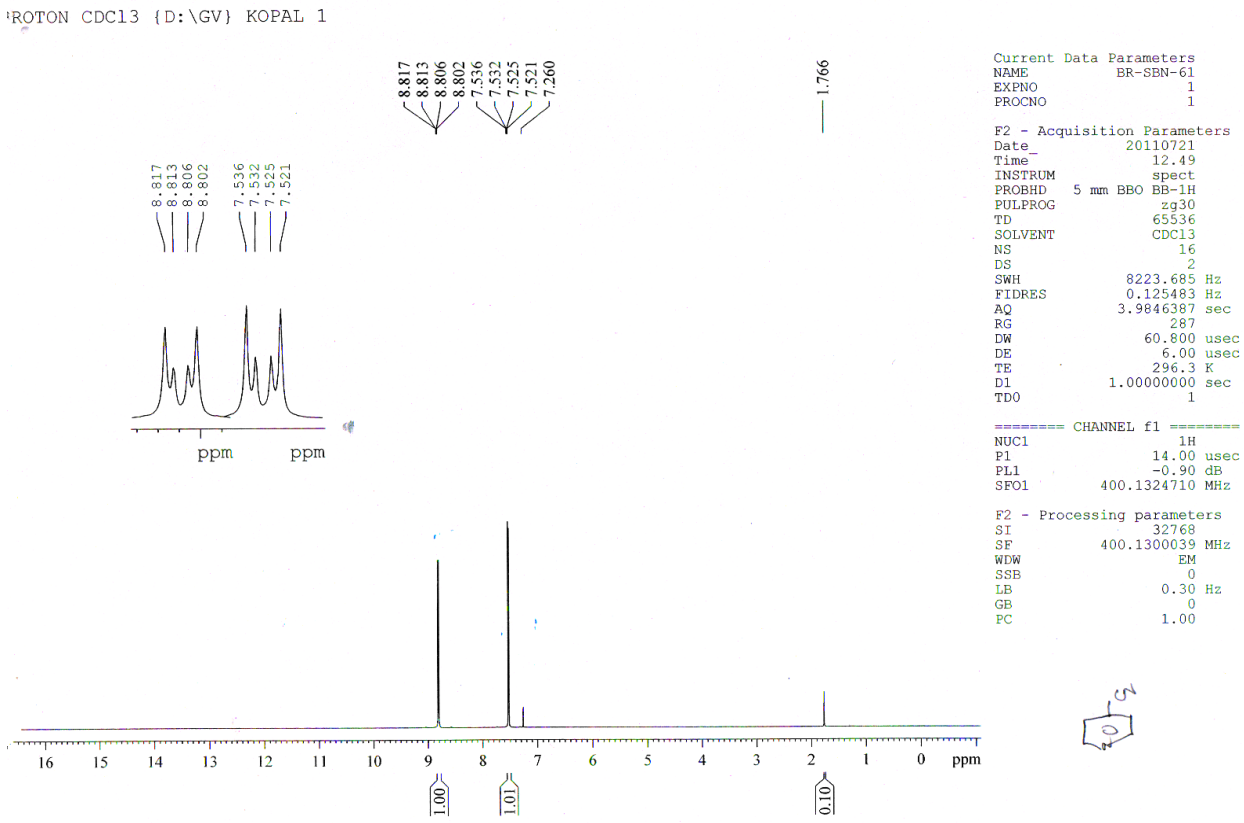

Fig: 39 ^{1}H-NMR of 4-cyanopyridine

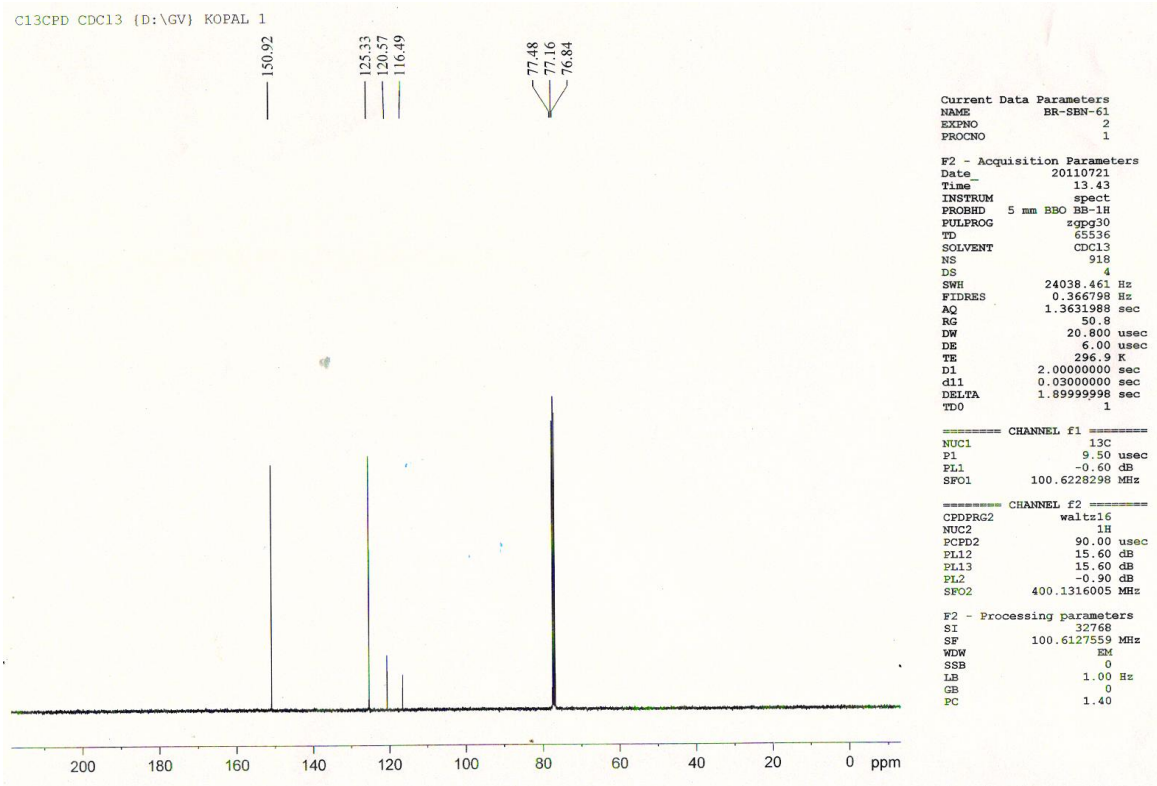

Fig: 40 ^{13}C-NMR of 4-cyanopyridine

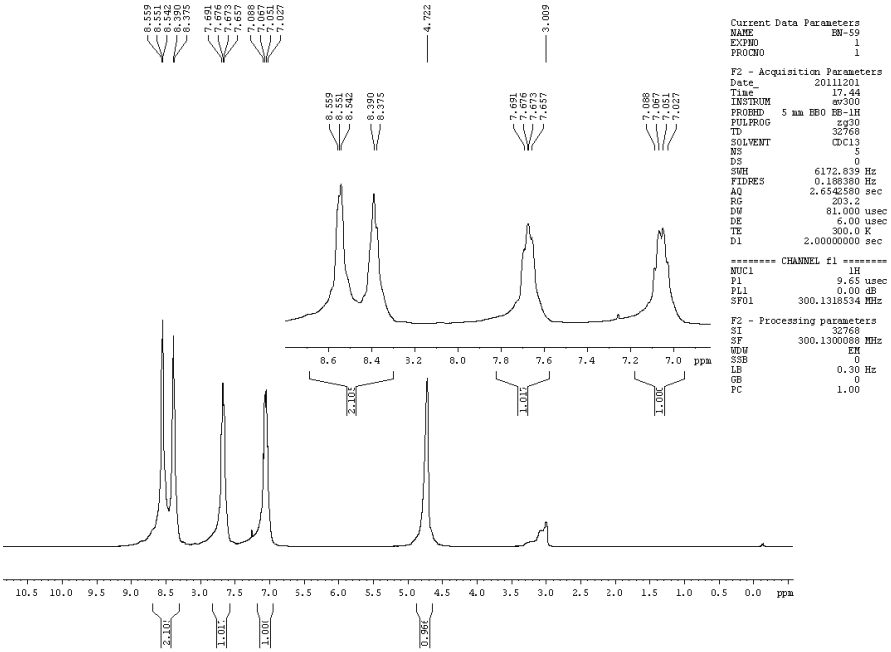

Fig: 41 ^{1}H-NMR of 3-cyanopyridine

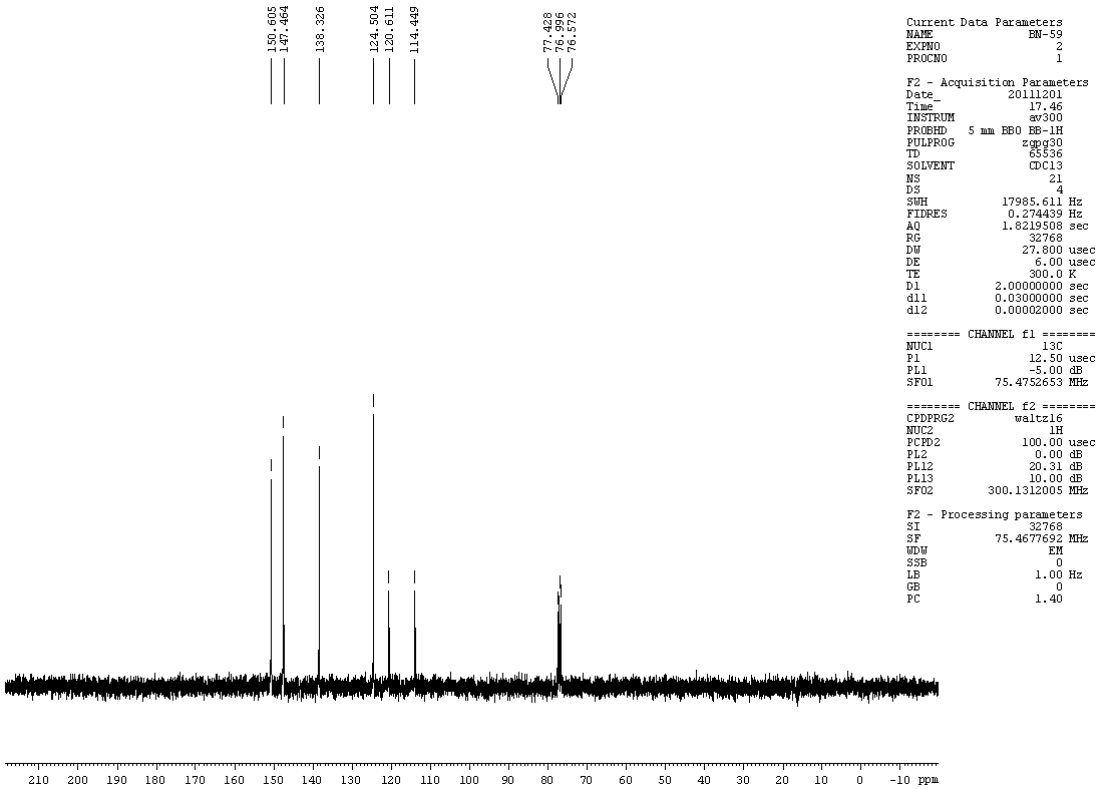

Fig: 42 ^{13}C-NMR of 3-cyanopyridine

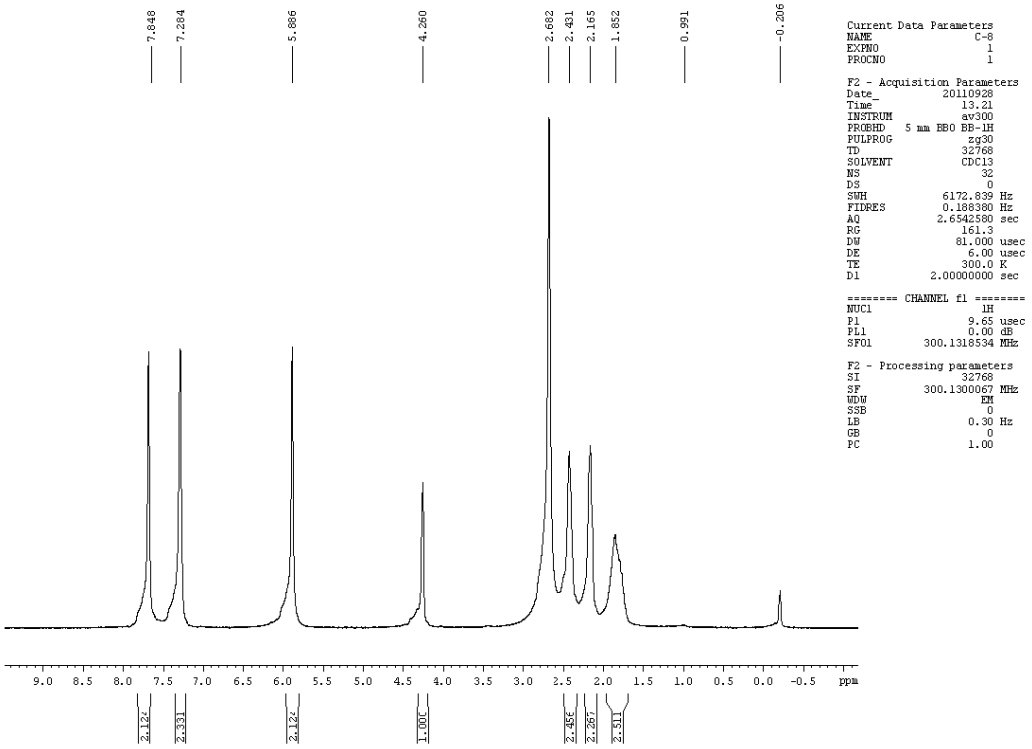

Fig: 43 ^{1}H-NMR spectrum of 2-amino-4-(4-cyanophenyl)-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile

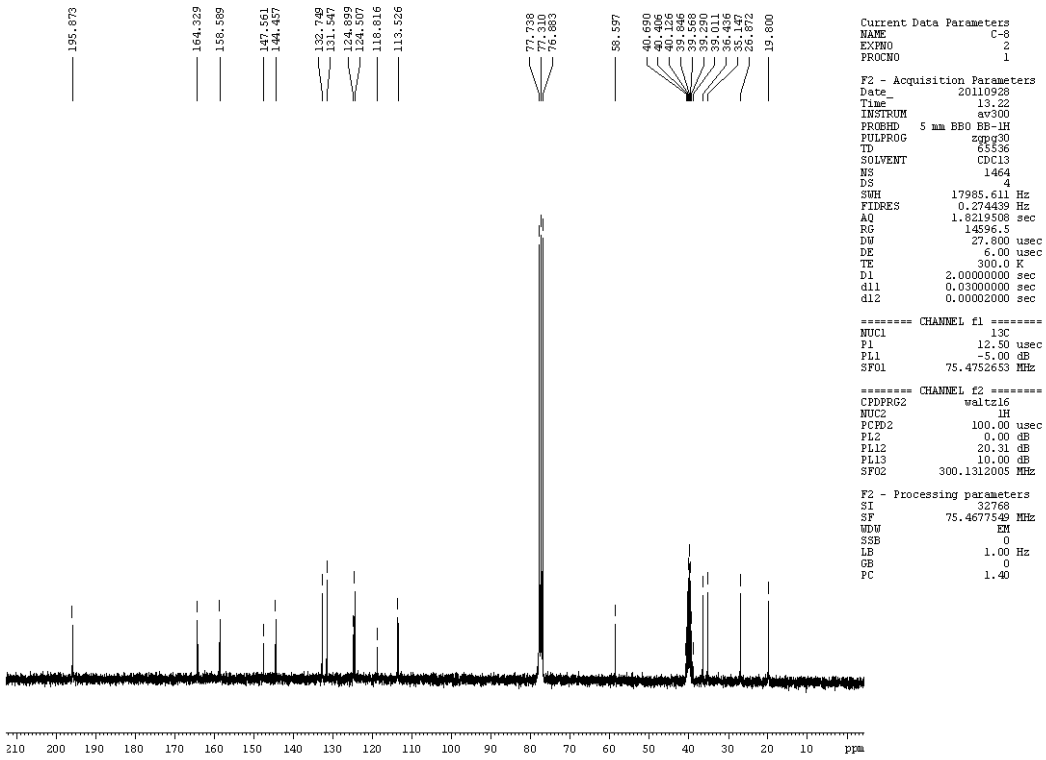

Fig: 44 ^{13}C-NMR spectrum of 2-amino-4-(4-cyanophenyl)-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile

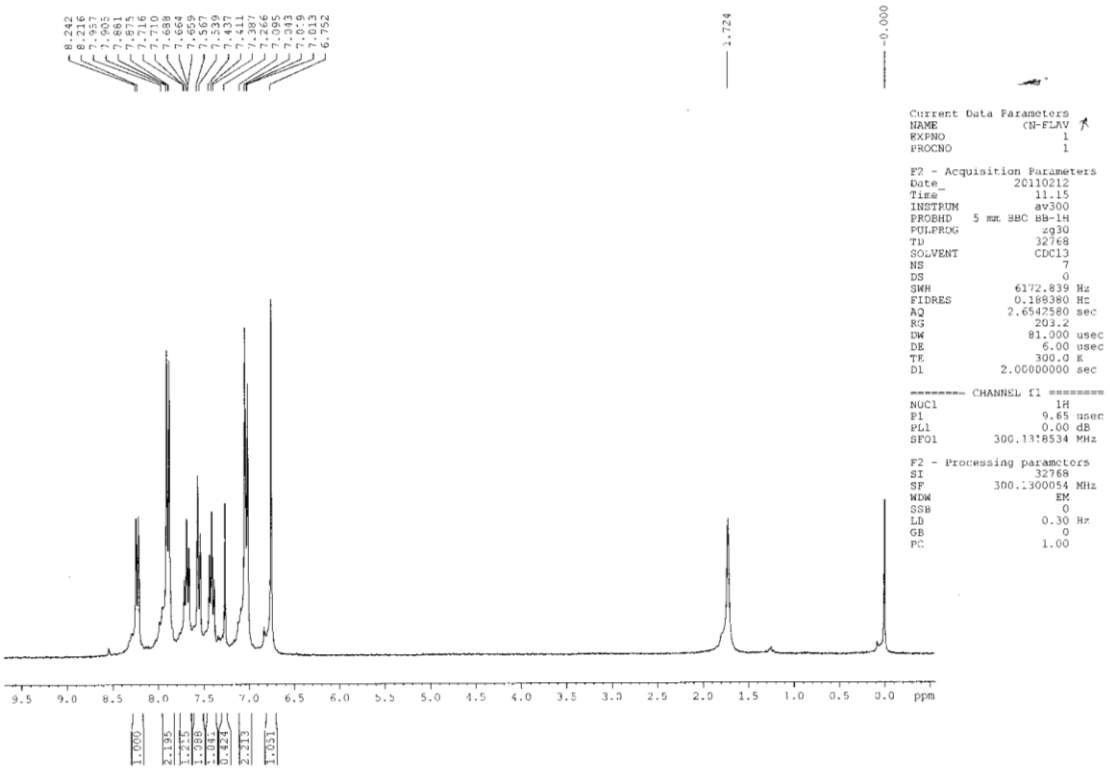

Fig: 45 ^{1}H-NMR of 4-(4-oxo-4H-chromen-2-yl)benzonitrile

3.3 ESI-MS spectra

3.3a Mass spectra of ligands:

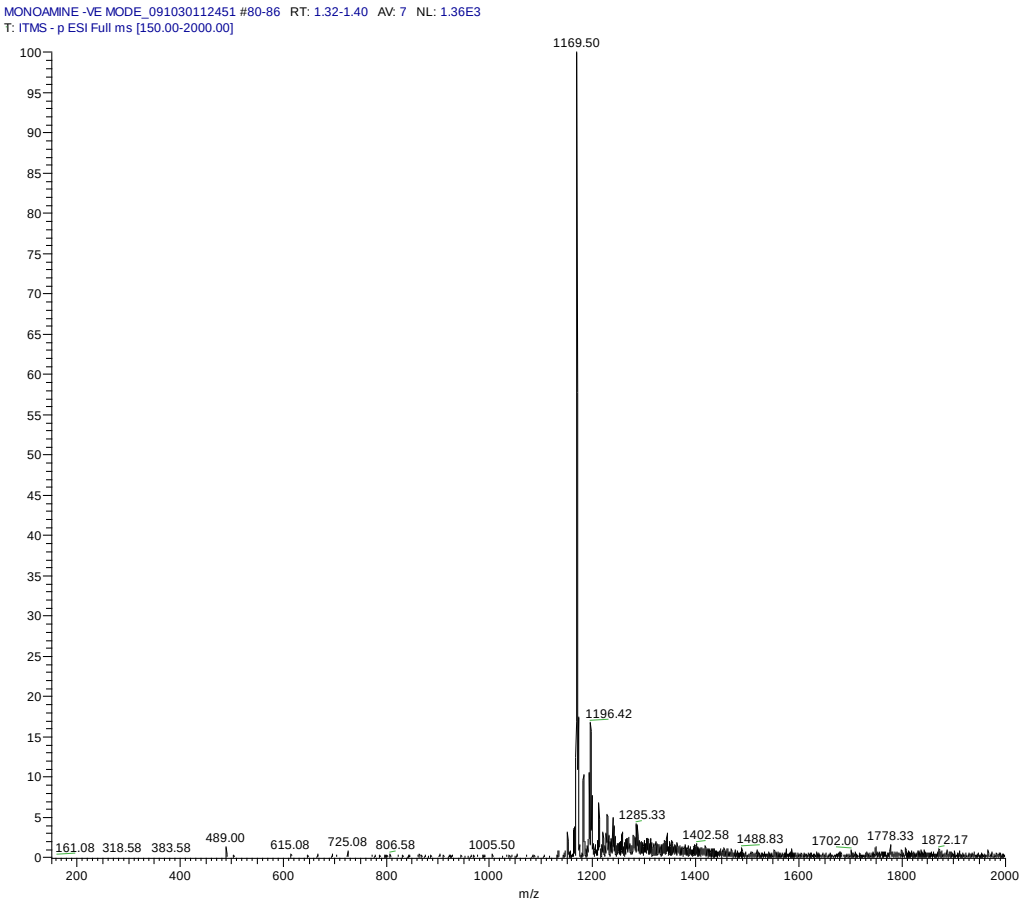

Fig: 46 ESI-MS spectrum of mono-6-amino-β-cyclodextrin ($M+Cl^-$ in negative mode)

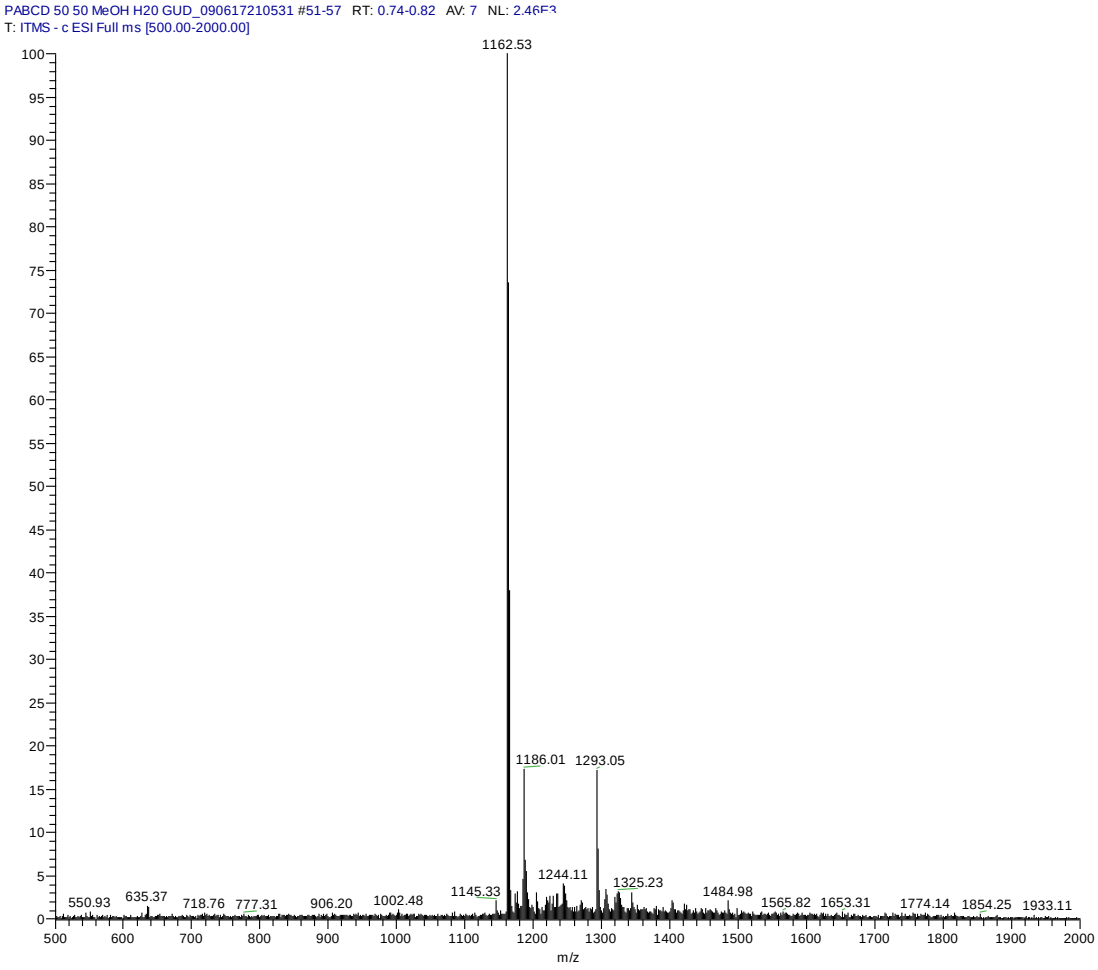

Fig: 47 ESI-MS spectrum of per-6-amino-β-cyclodextrin ($M+Cl^-$ in negative mode)

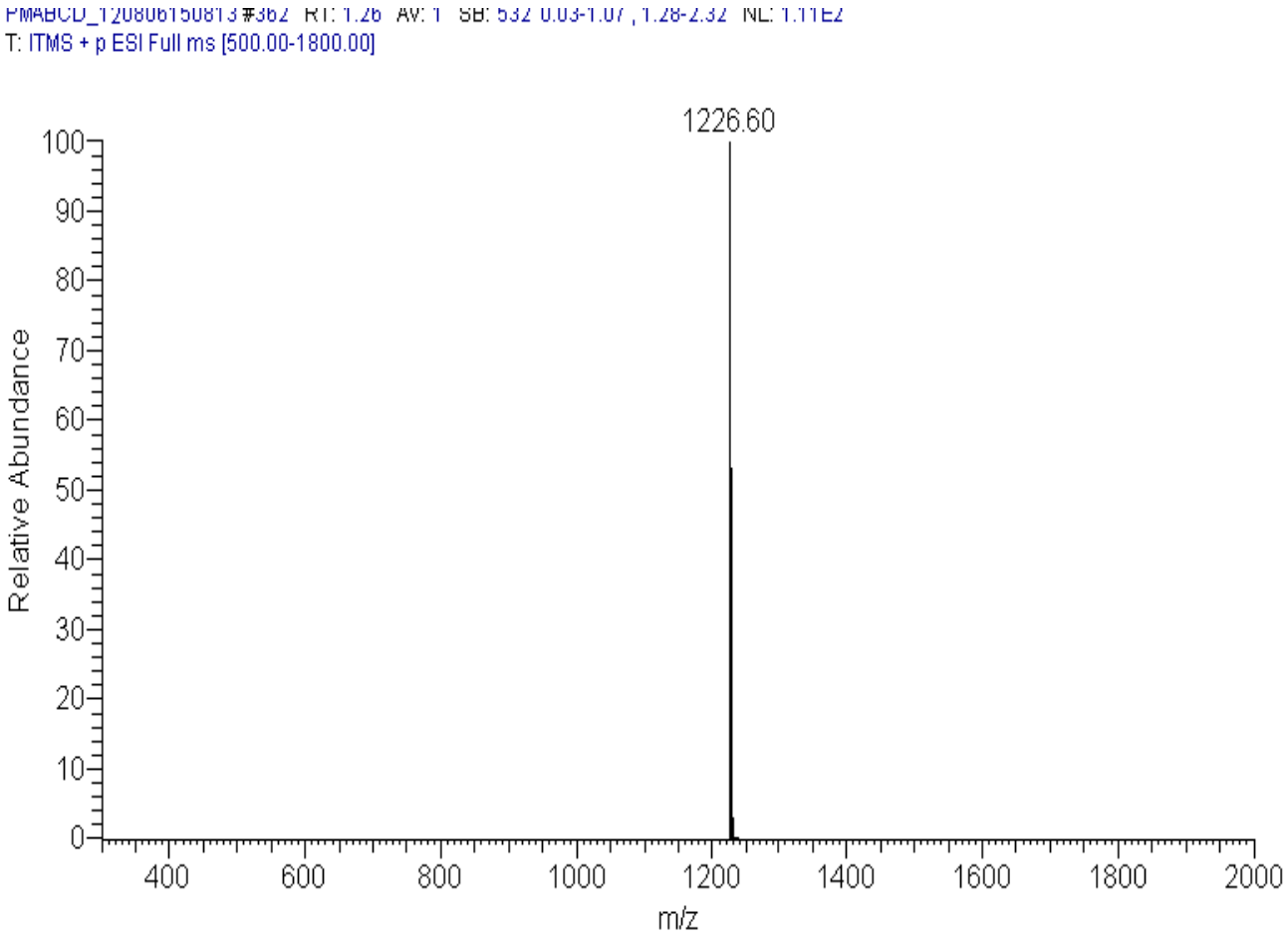

Fig: 48 ESI-MS spectrum of per-6-methylamino-β-cyclodextrin ($M+H^+$ in positive mode)

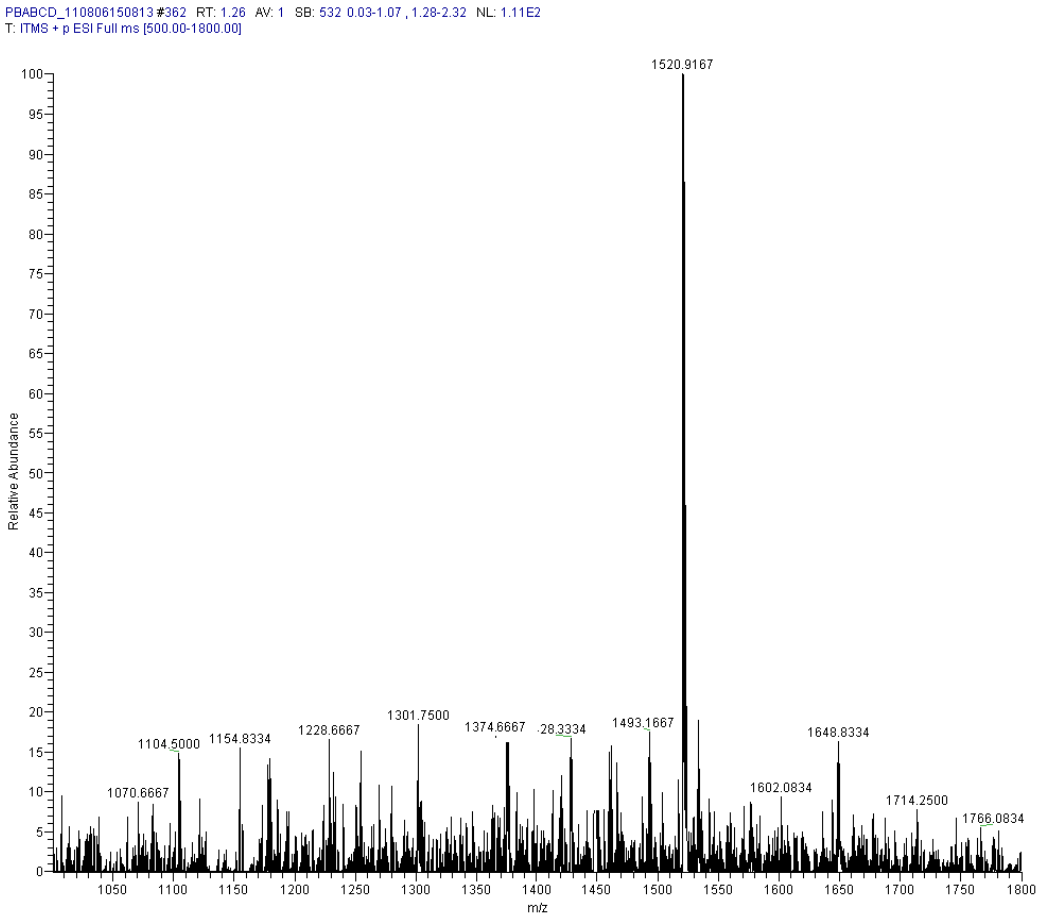

Fig: 49 ESI-MS spectrum of per-6-butylamino-β-cyclodextrin ($M+H^+$ in positive mode)

2.3b Mass spectra of aromatic nitriles:

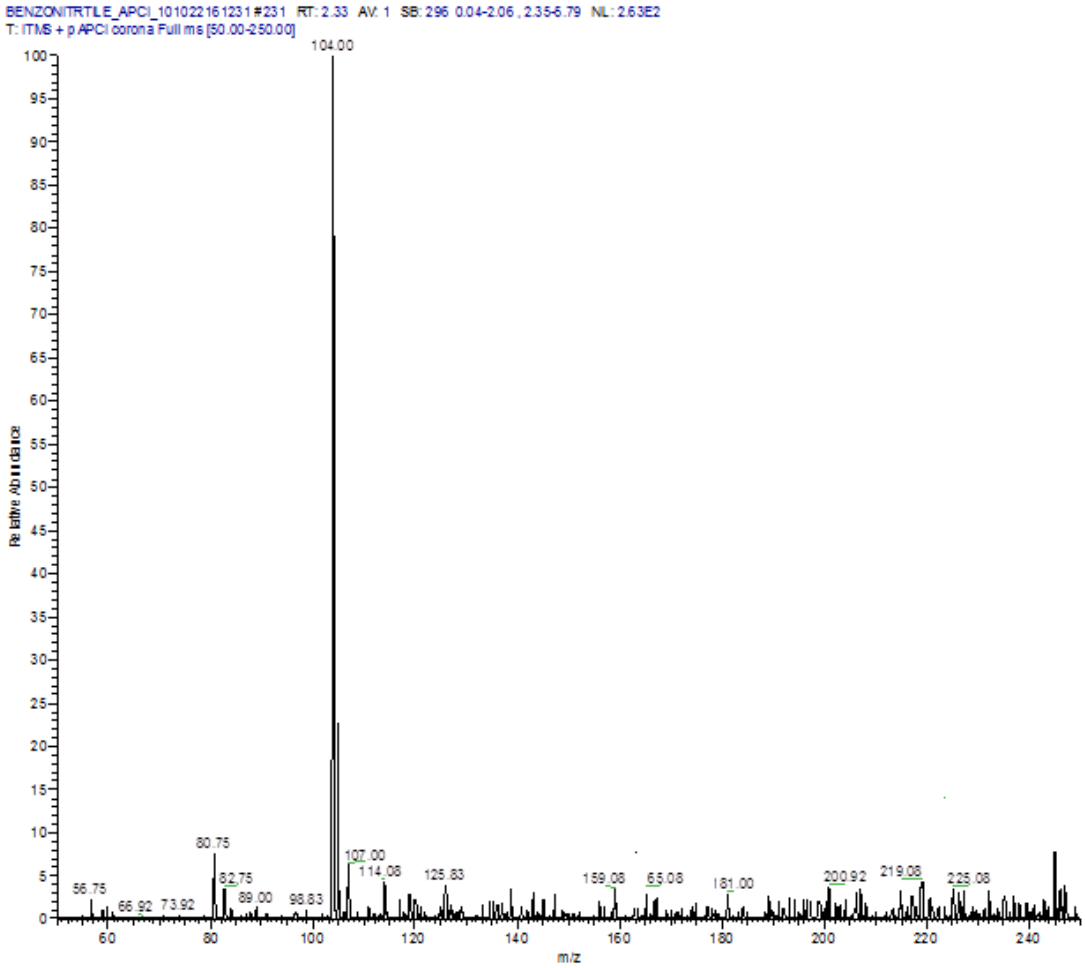

Fig: 50 ESI-MS spectrum of benzonitrile

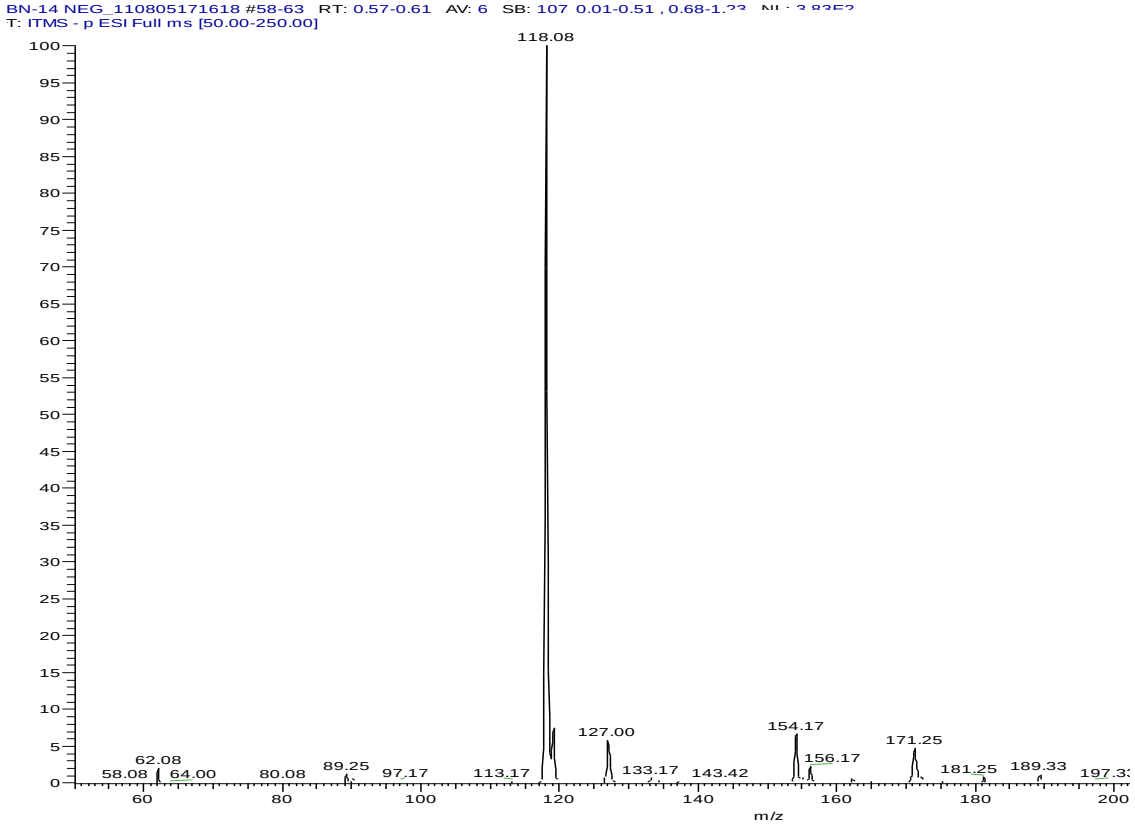

Fig: 51ESI-MS spectrum of 4-hydroxybenzonitrile

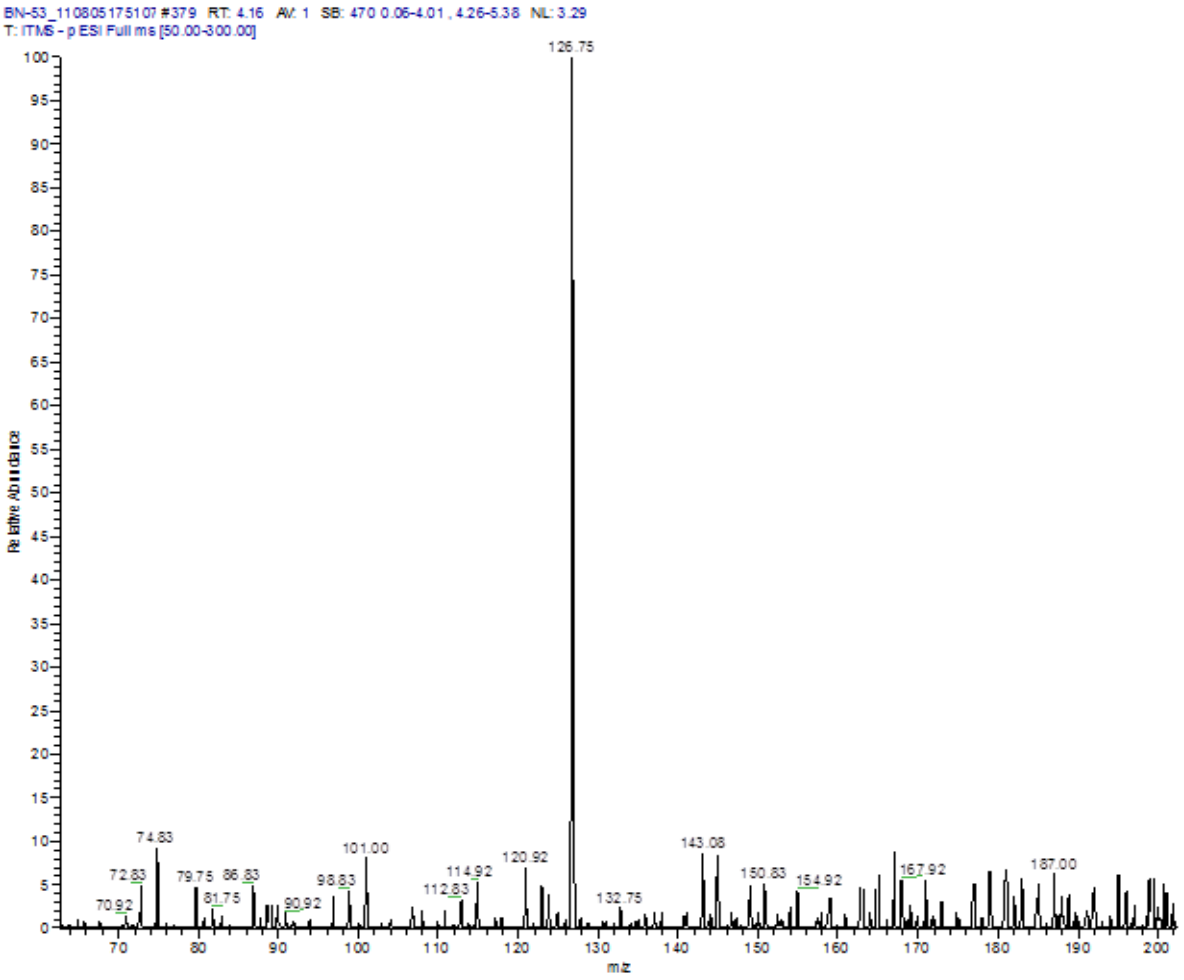

Fig: 52ESI-MS spectrum of 1,4-dicyanobenzene

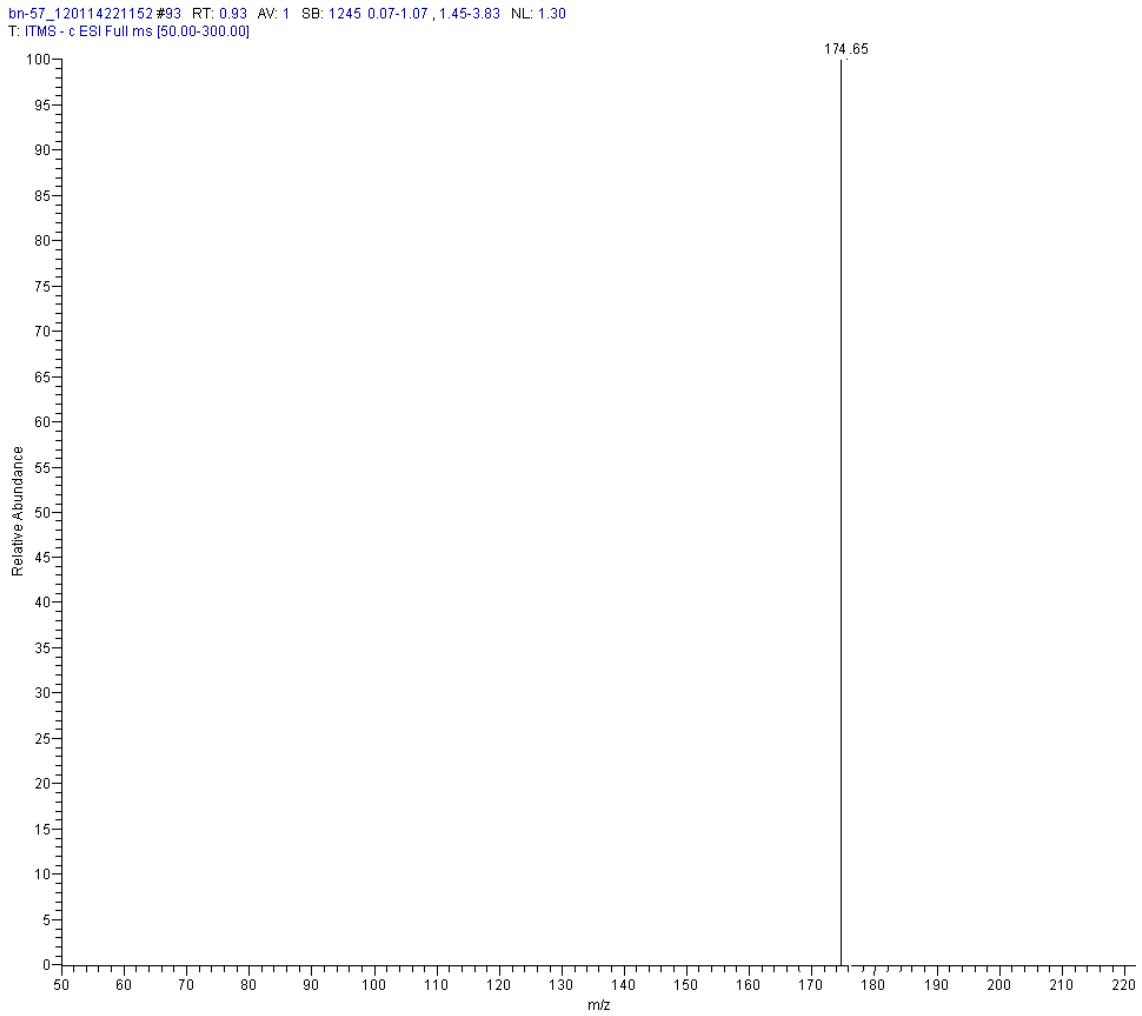

Fig: 53 ESI-MS spectrum of ethyl 4-cyanobenzoate

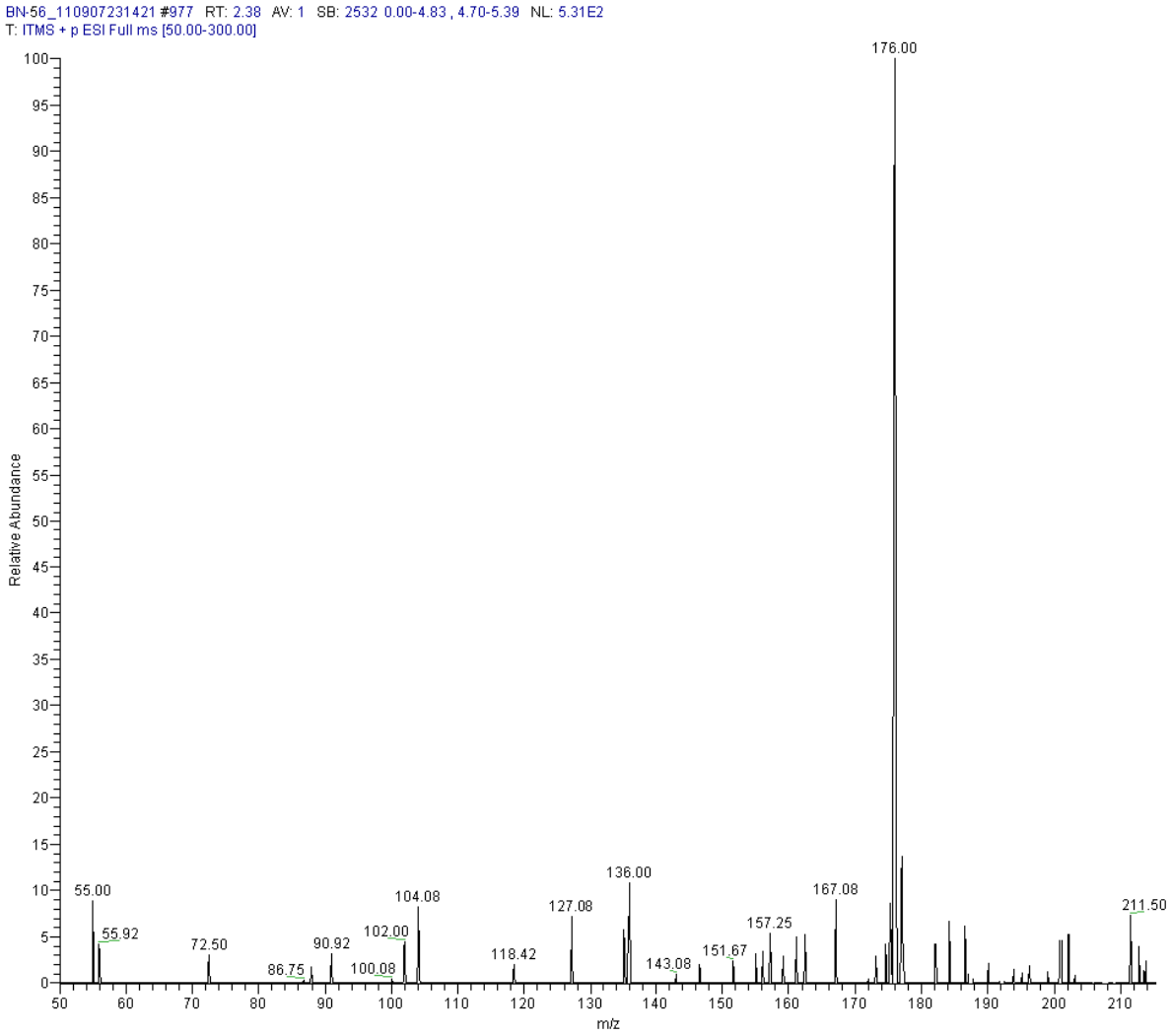

Fig: 54 ESI-MS spectrum of ethyl 3-cyanobenzoate

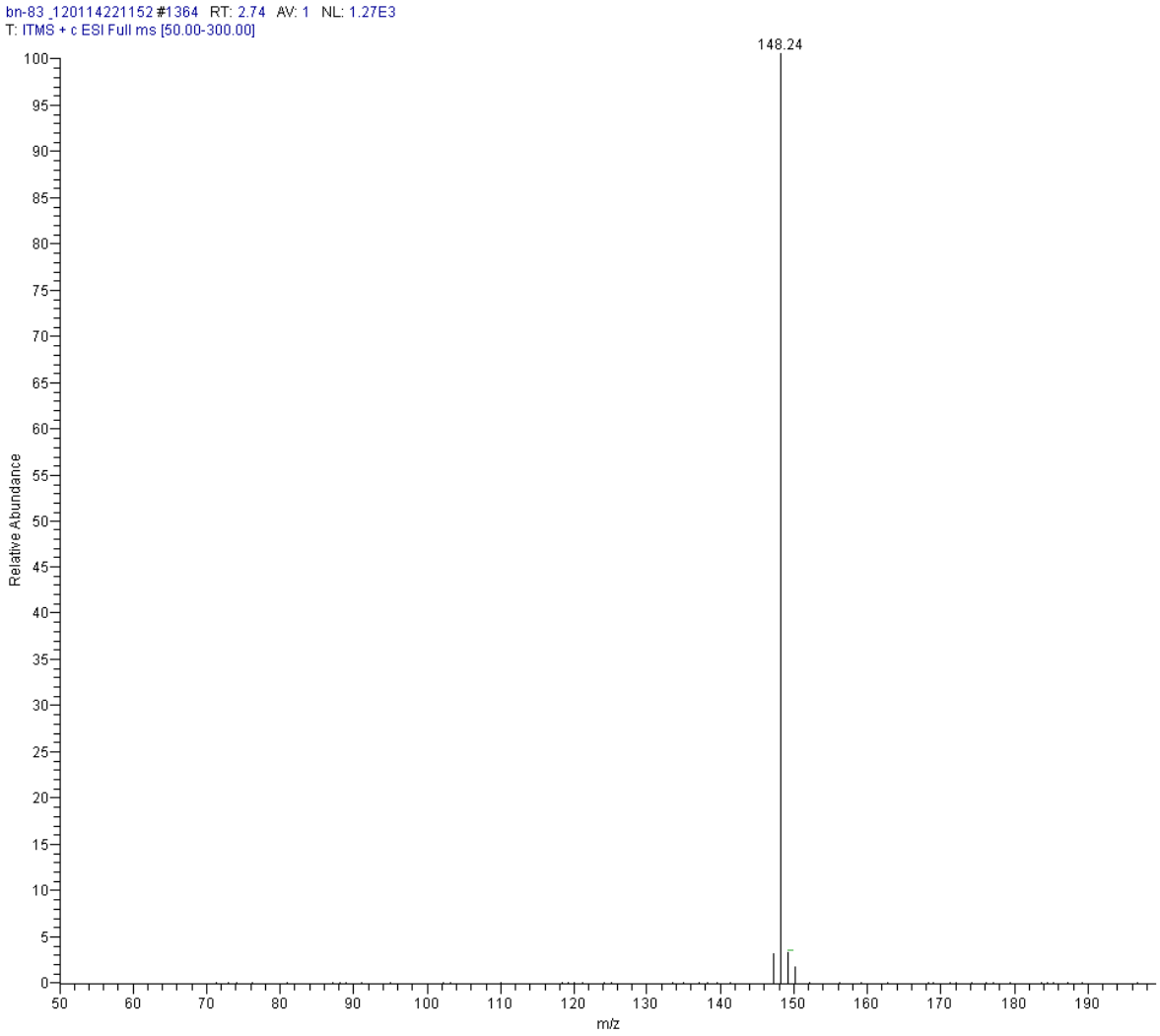

Fig: 55 ESI-MS spectrum of 2-cyanobenzoic acid

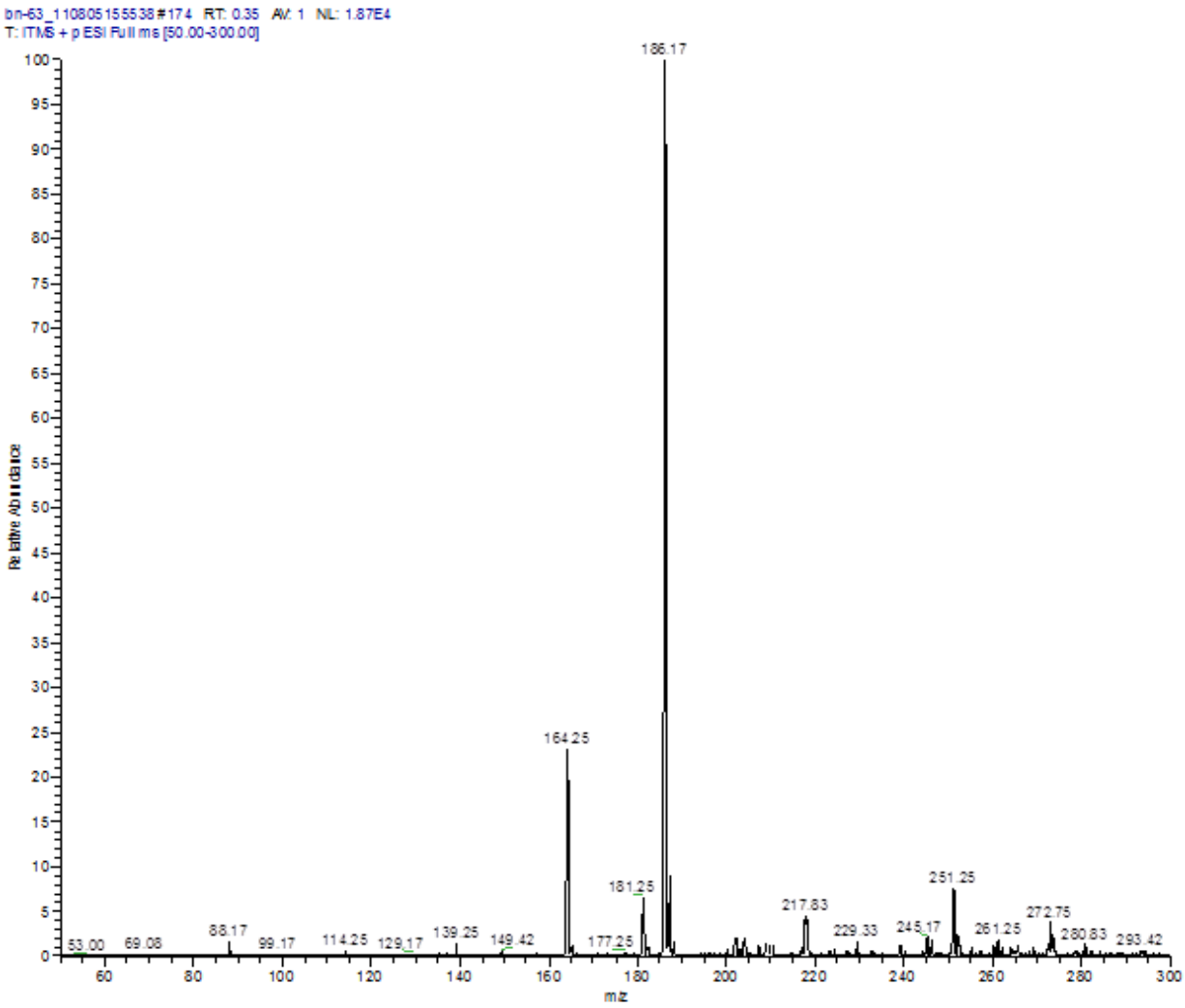

Fig: 56 ESI-MS spectrum of 2,4-dimethoxybenzonitrile

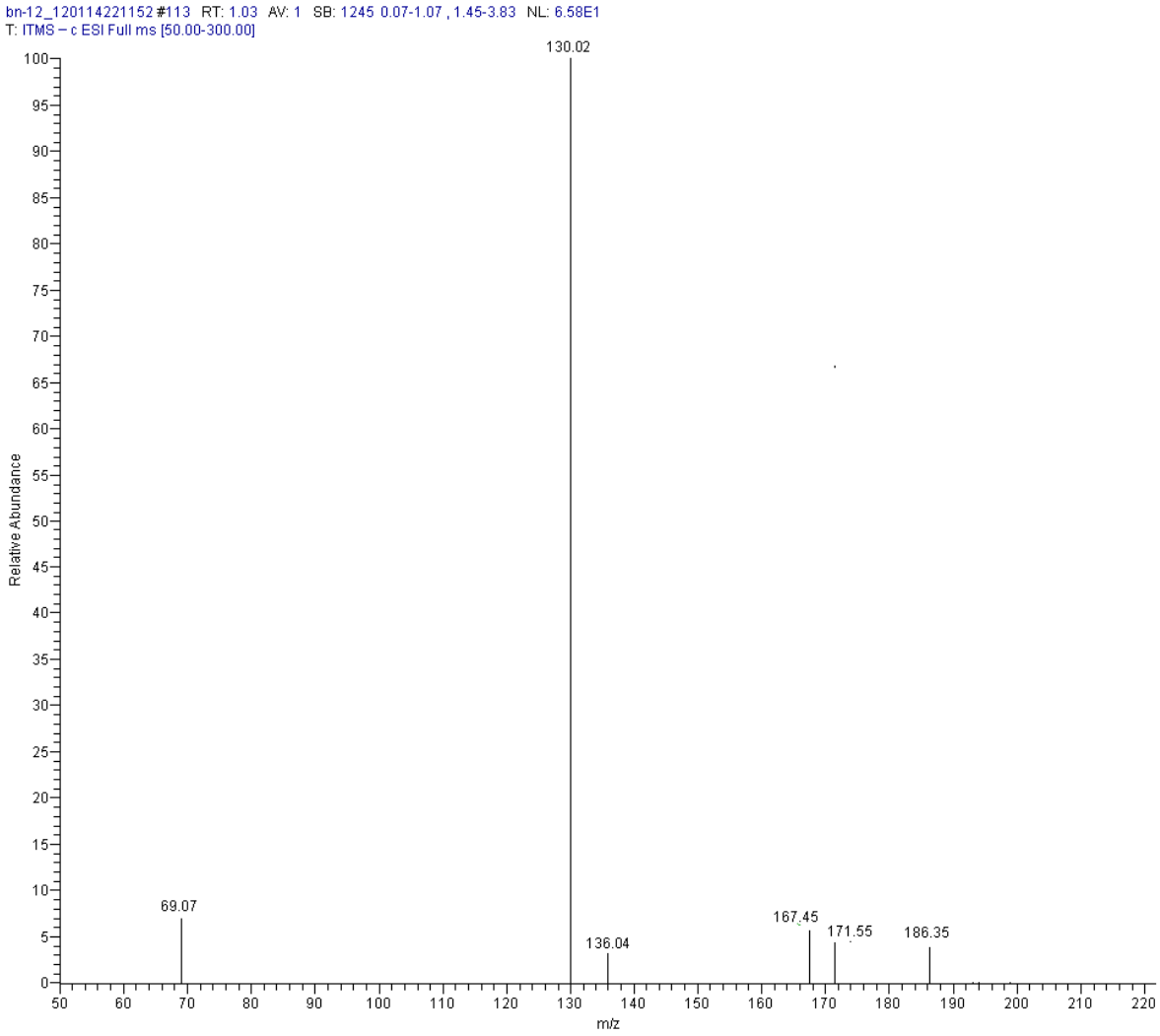

Fig: 57 ESI-MS spectrum of 3,4-dimethylbenzonitrile

Fig. 58 ESI-MS spectrum of 4-chlorobenzonitrile

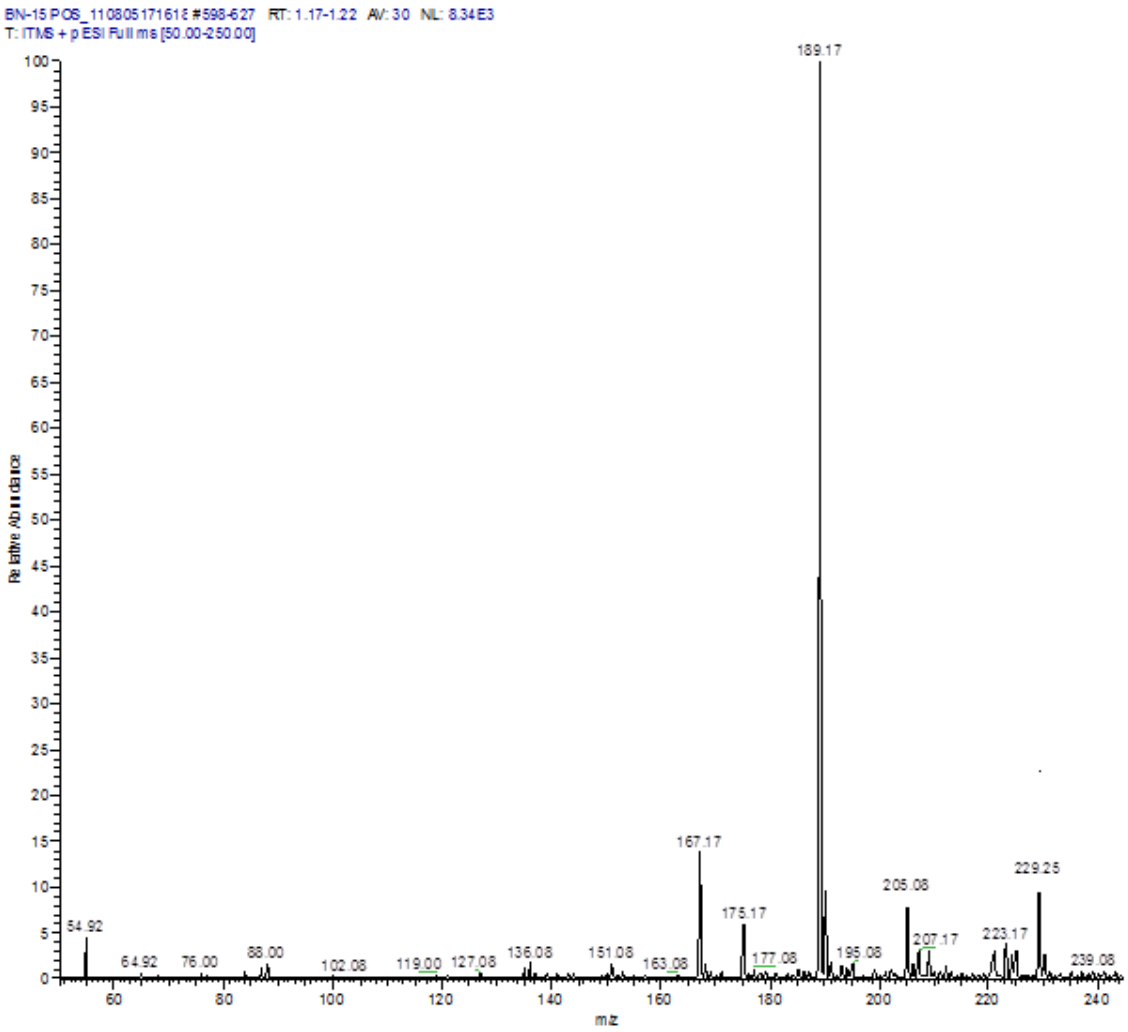

Fig. 59 ESI-MS spectrum of 4-nitrobenzonitrile (M+ACN adduct)

Fig. 60 ESI-MS spectrum of 4-aminobenzonitrile

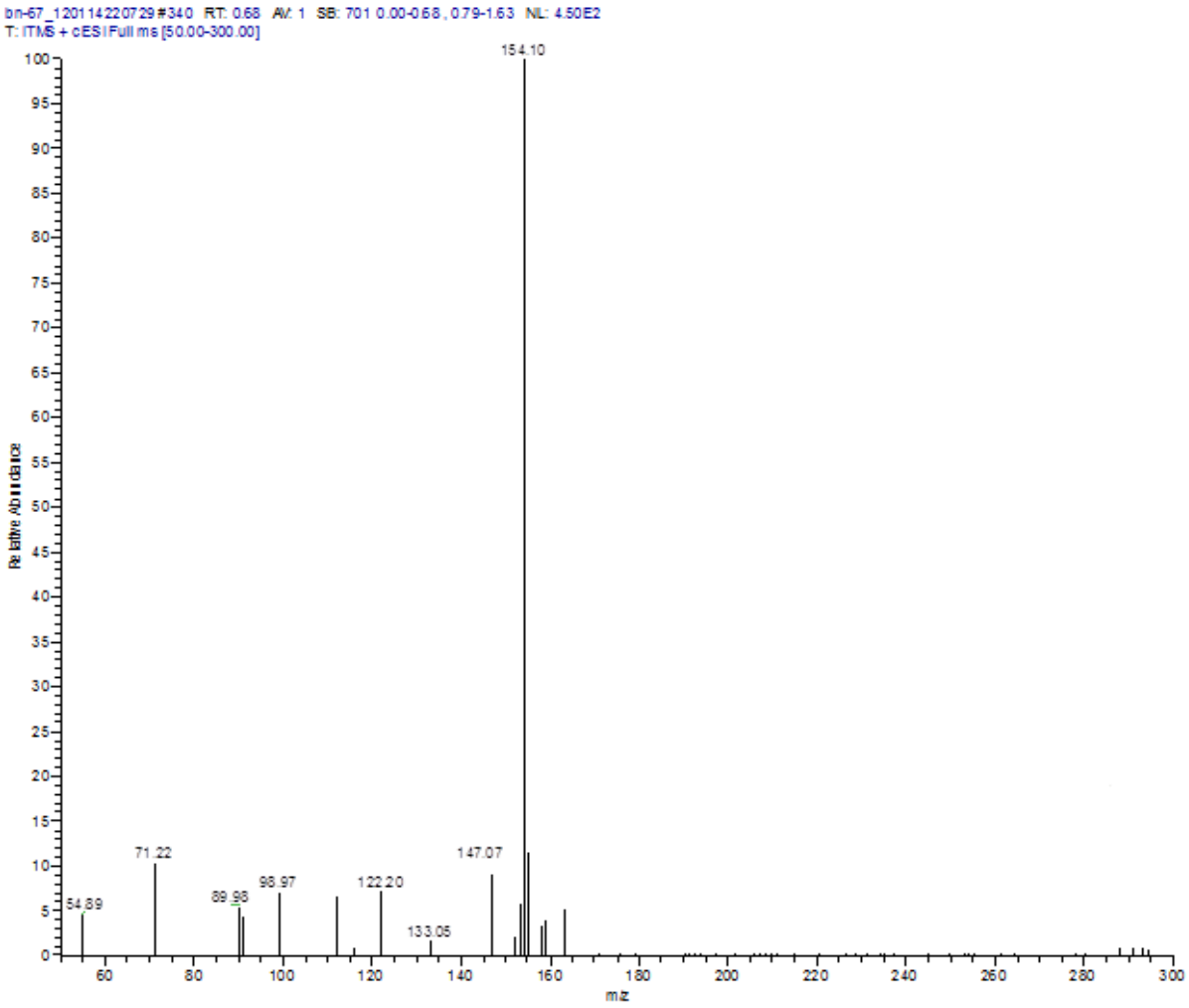

Fig.61 ESI-MS spectrum of 1-naphthonitrile

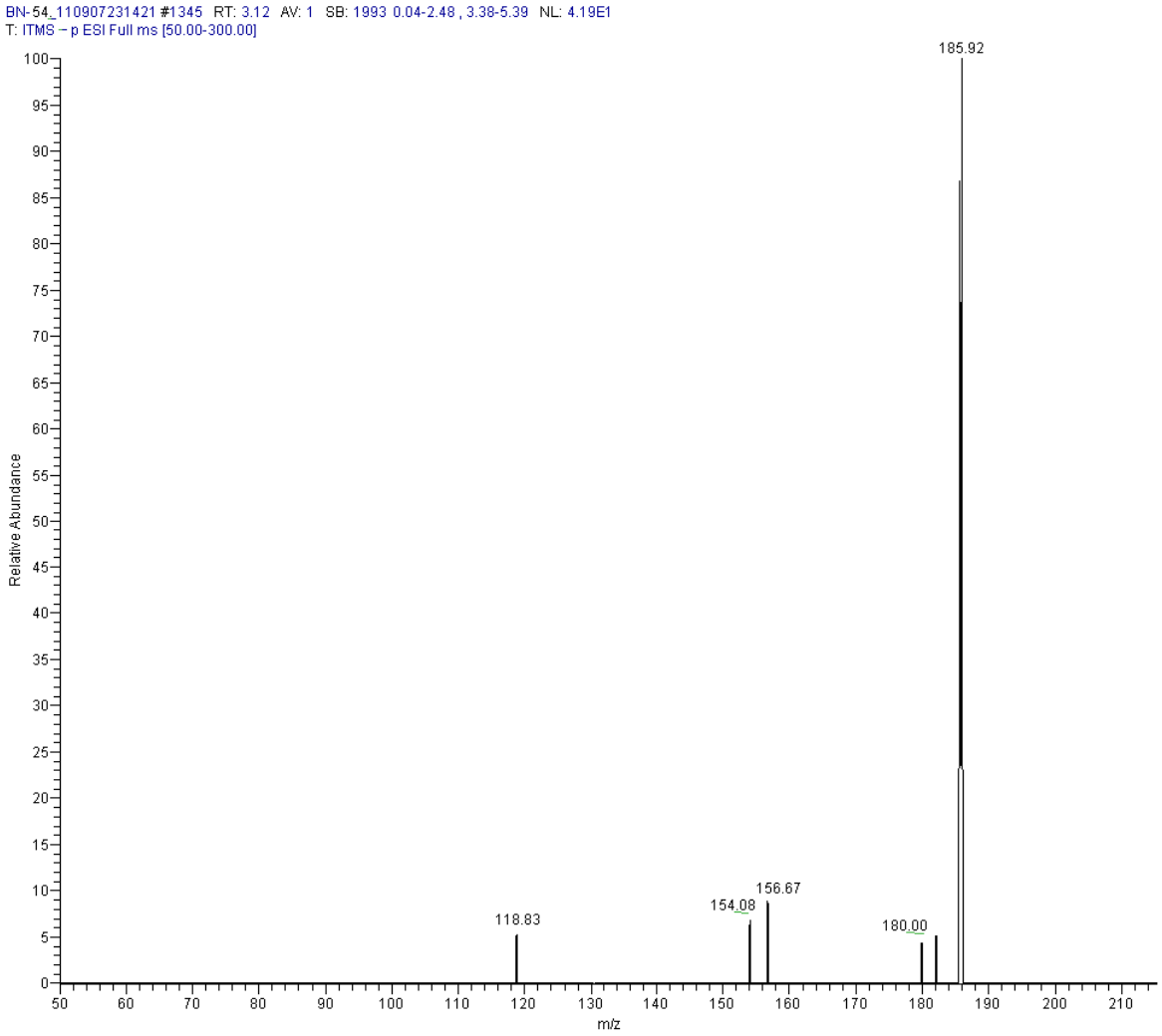

Fig. 62 ESI-MS spectrum of 4-(trifluoromethoxy)benzonitrile

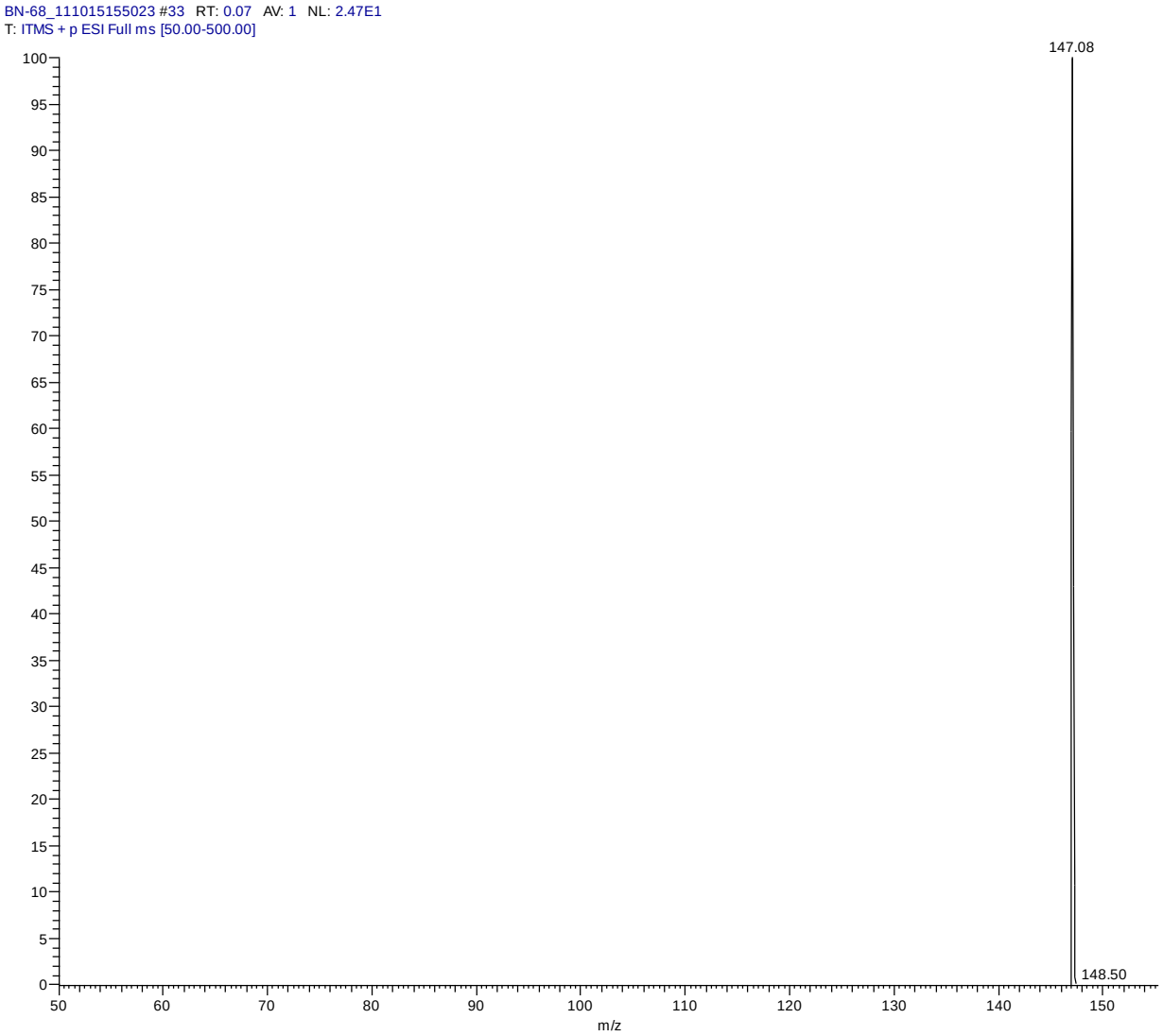

Fig. 63 ESI-MS spectrum of thiophene-2-carbonitrile

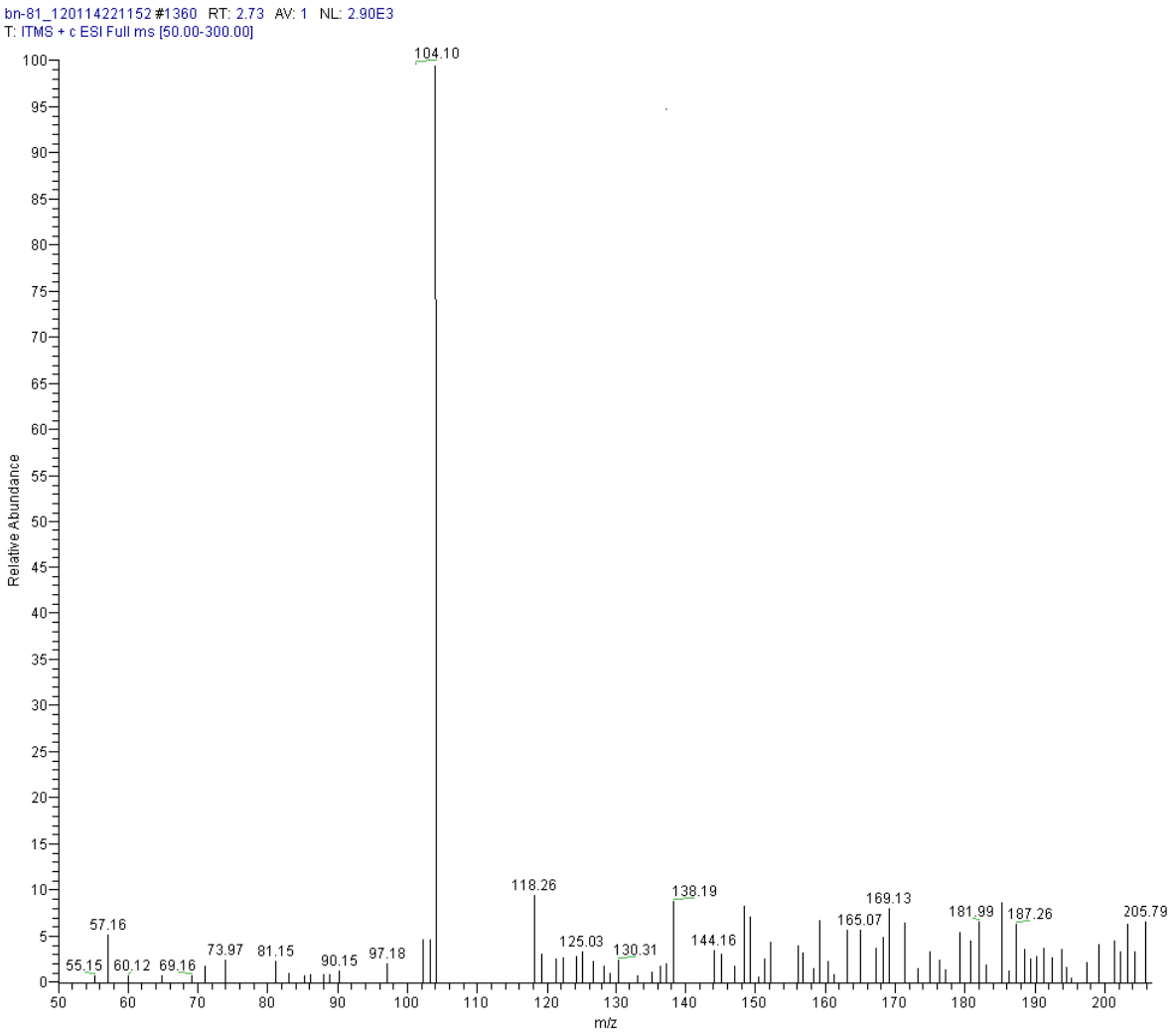

Fig. 64 ESI-MS spectrum of pyrimidine-5-carbonitrile

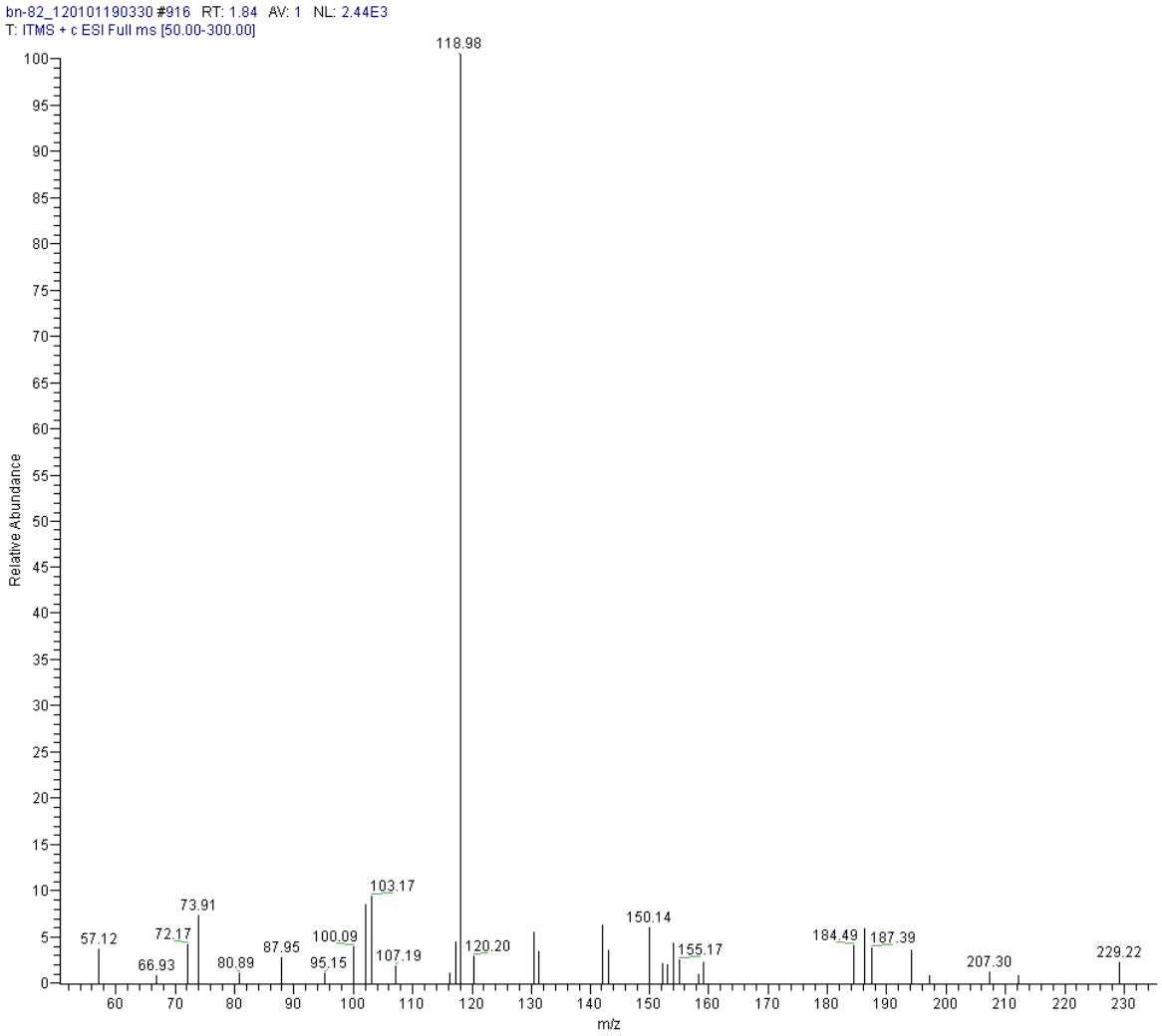

Fig. 65 ESI-MS spectrum of 5-methylpicolinonitrile

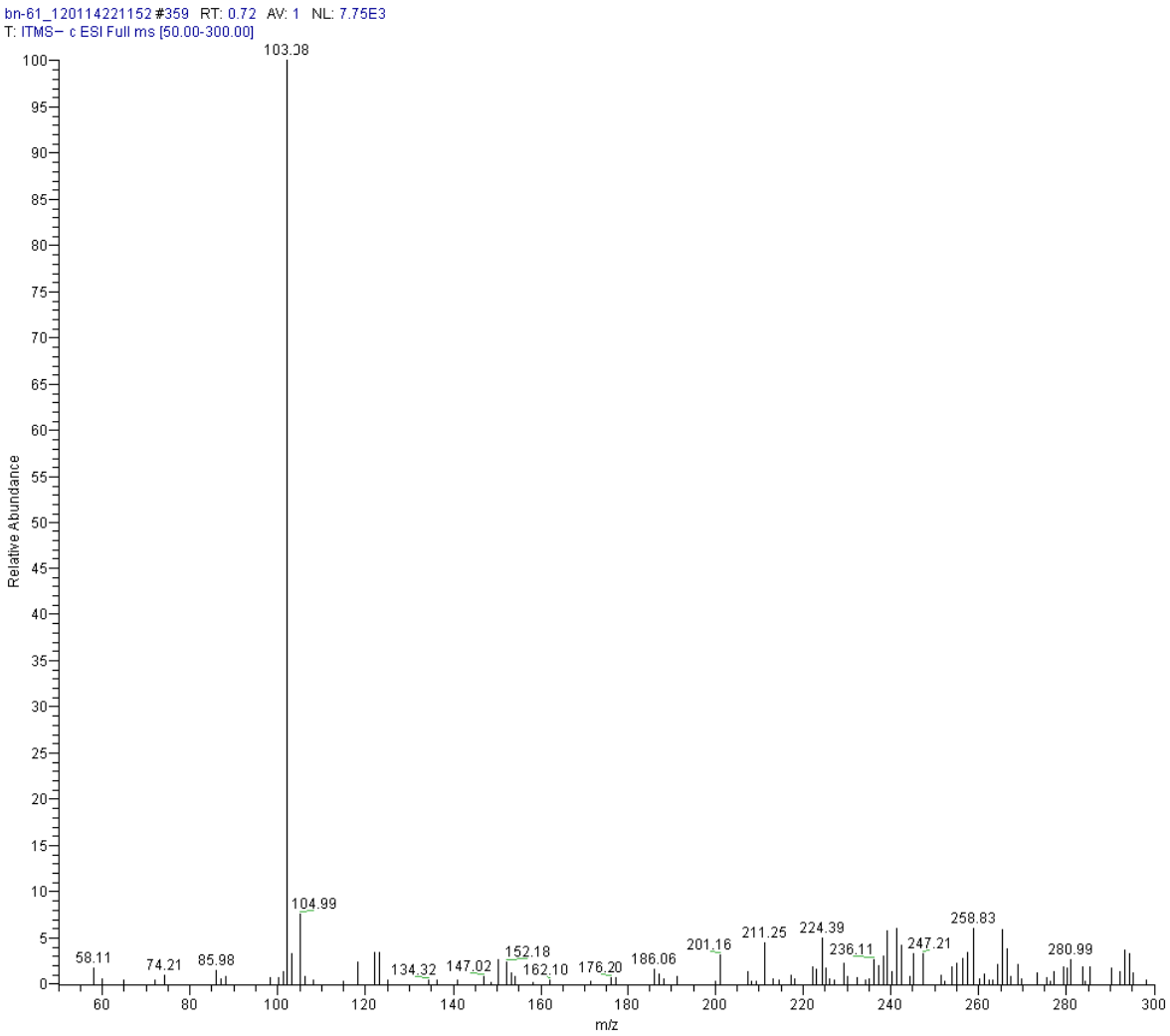

Fig. 66 ESI-MS spectrum of 4-cyanopyridine

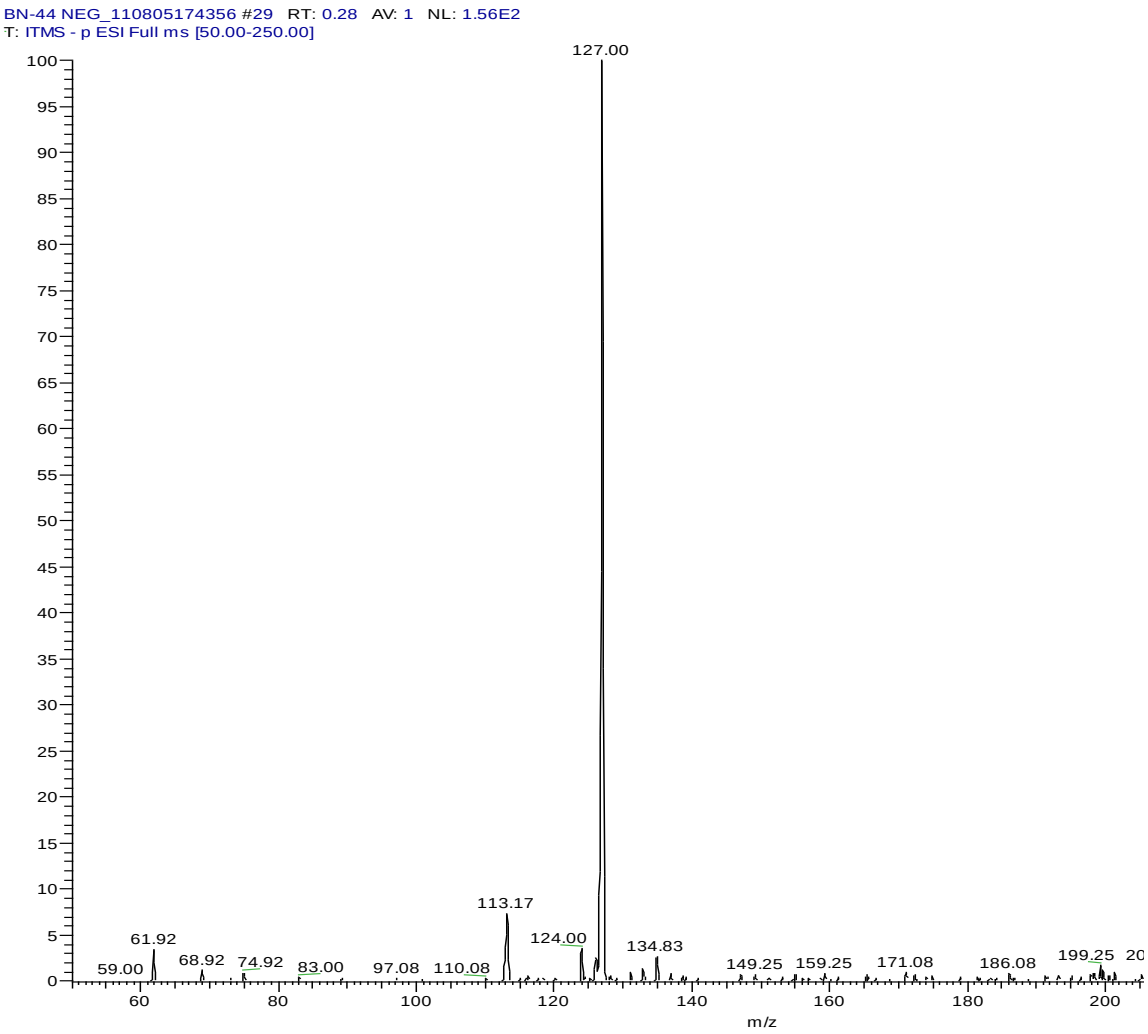

Fig. 67ESI-MS spectrum of 3-cyanopyridine

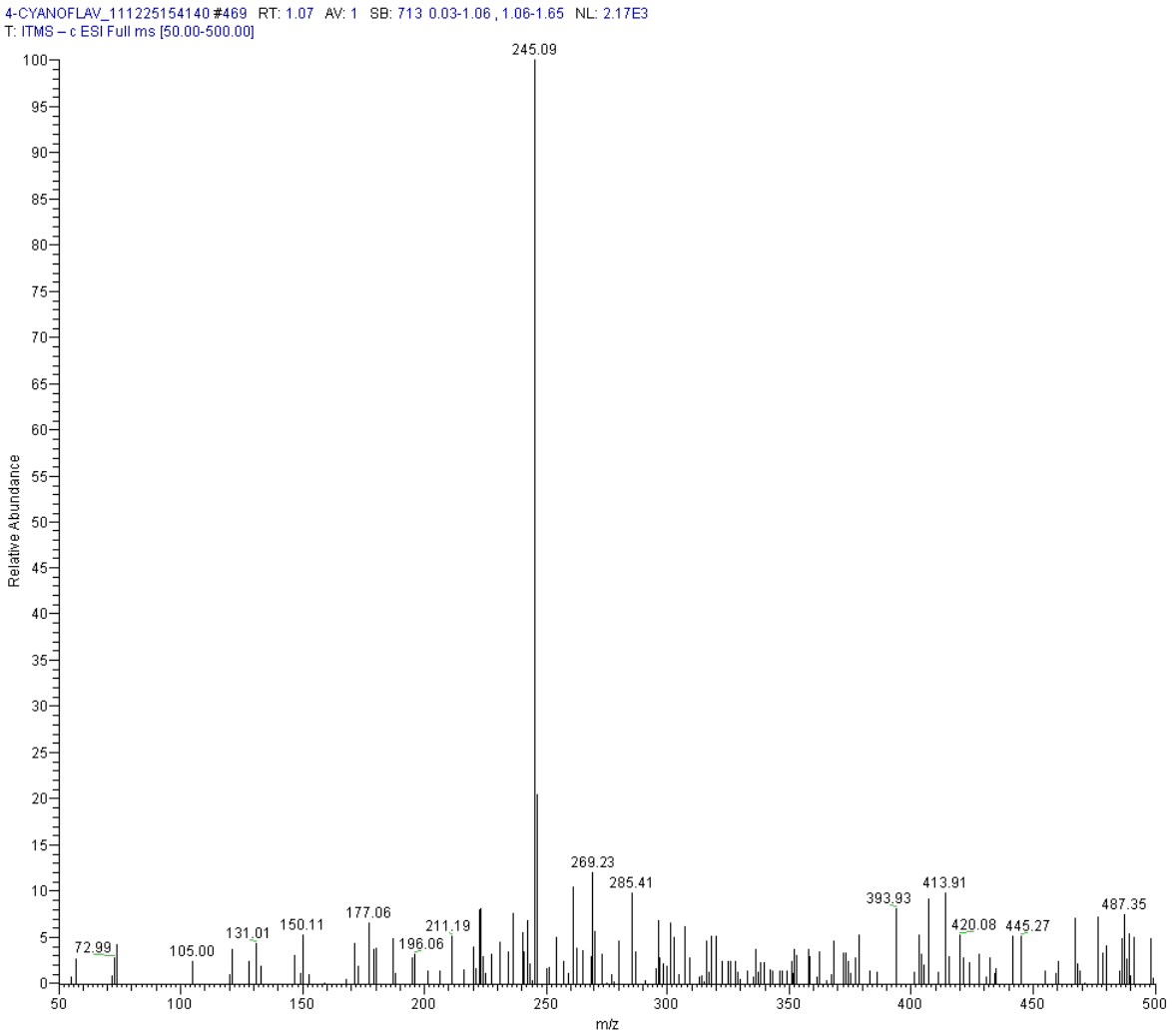

Fig. 68ESI-MS spectrum of 4-(4-oxo-4H-chromen-2-yl)benzonitrile

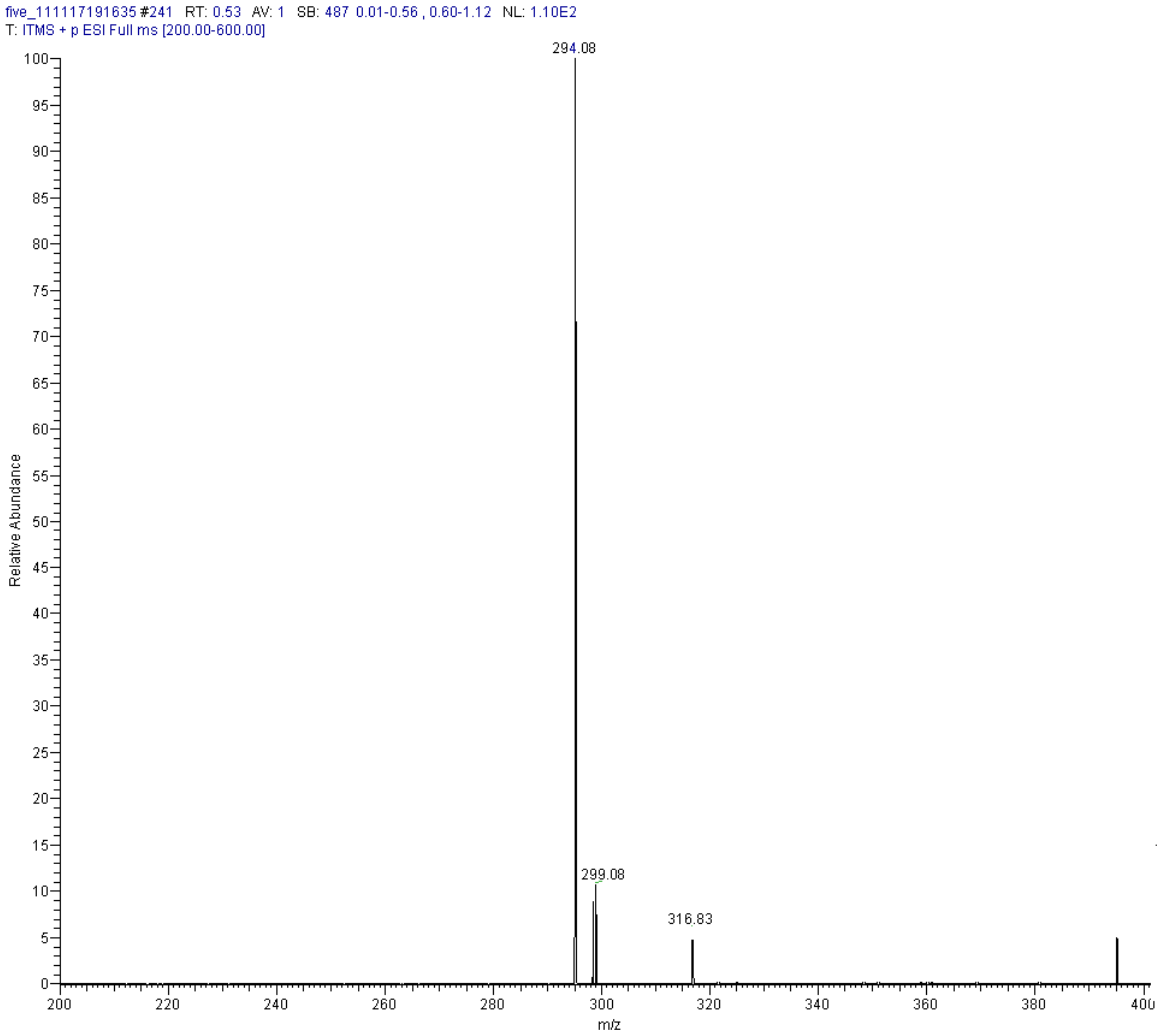

Fig.69 ESI-MS spectrum of 2-amino-4-(4-cyanophenyl)-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile

3.4. IR spectra of aryl nitriles:

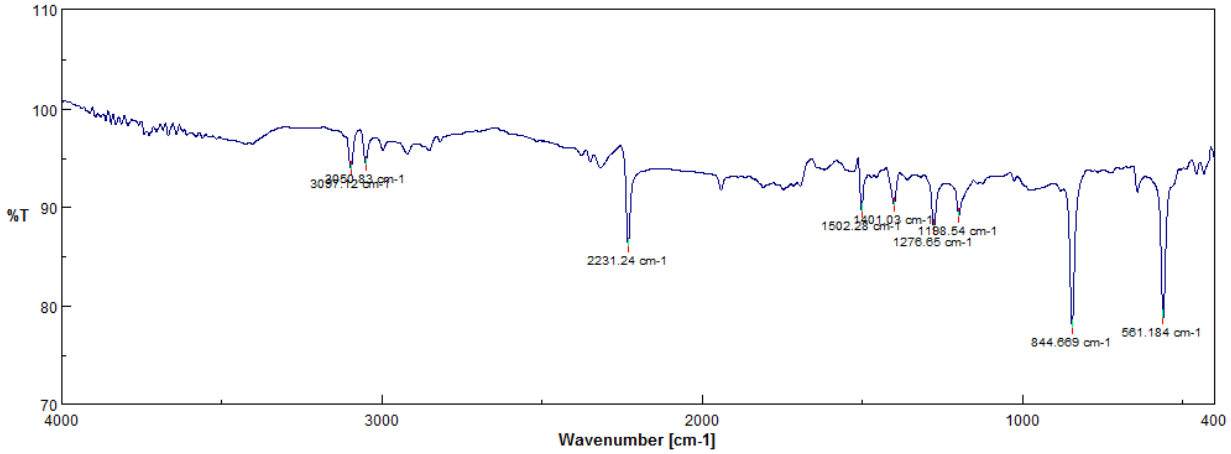

Fig. 70 FT-IR spectrum of benzonitrile

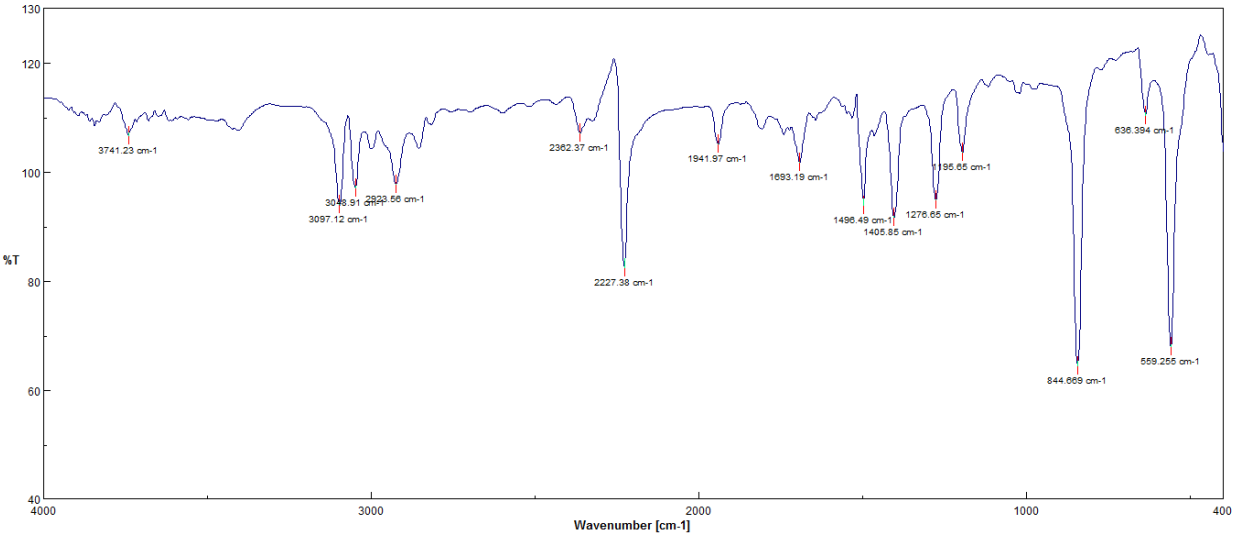

Fig. 71 FT-IR spectrum of 4-dicyanobenzene

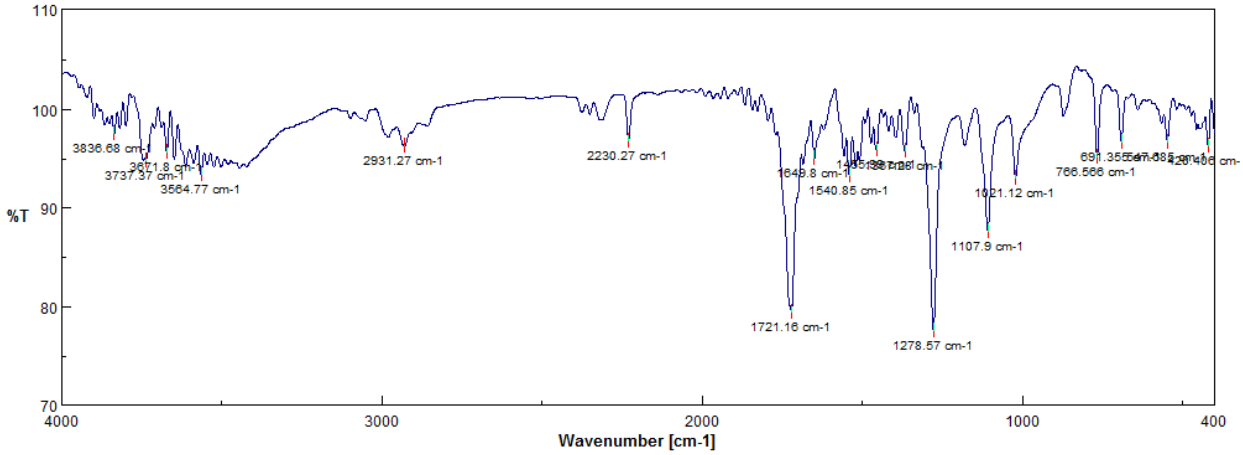

Fig.72 FT-IR spectrum of ethyl 4-cyanobenzoate

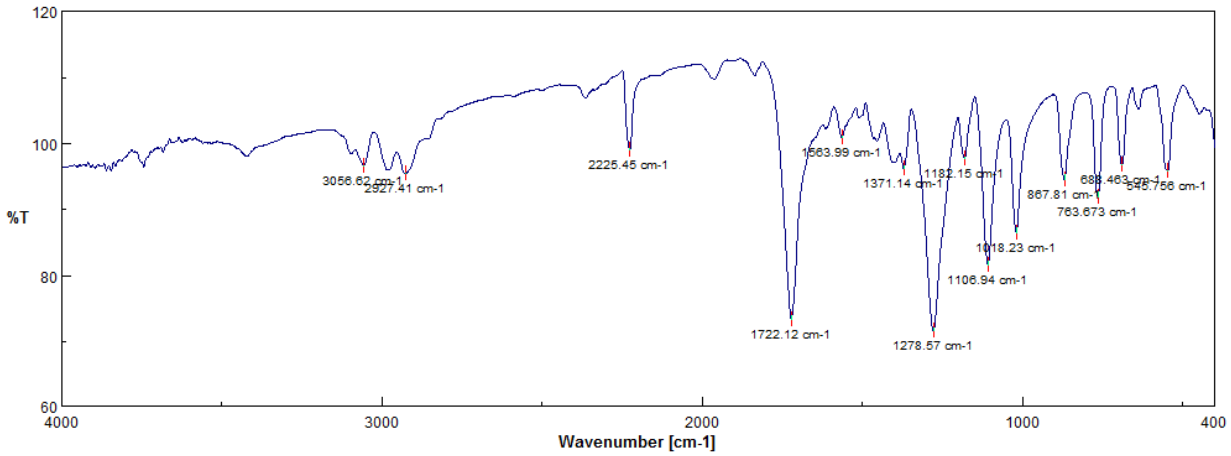

Fig. 73 FT-IR spectrum of ethyl 3-cyanobenzoate

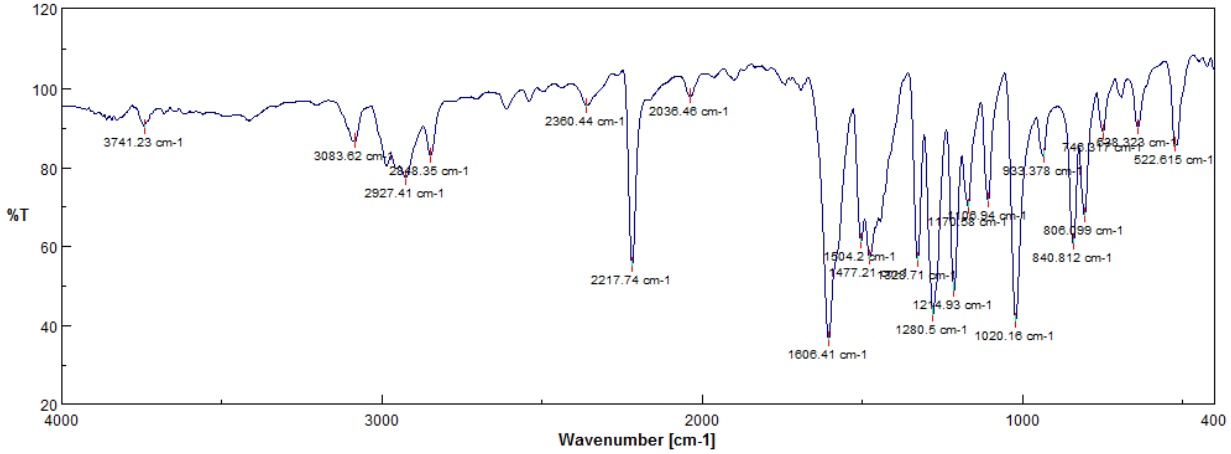

Fig. 74 FT-IR spectrum of 2,4-dimethoxycyanobenzene

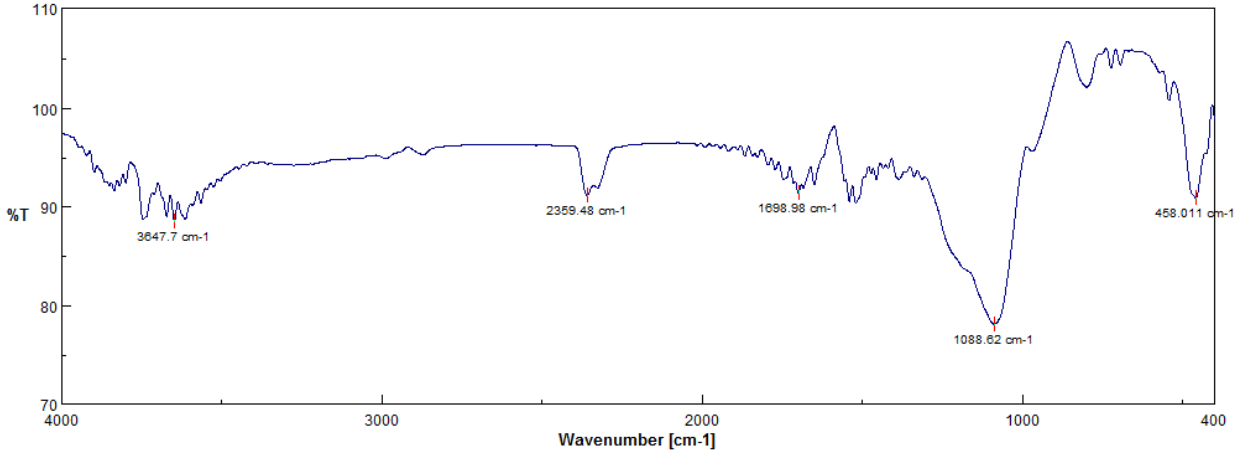

Fig. 75 FT-IR spectrum of 2,4-dimethylcyanobenzene

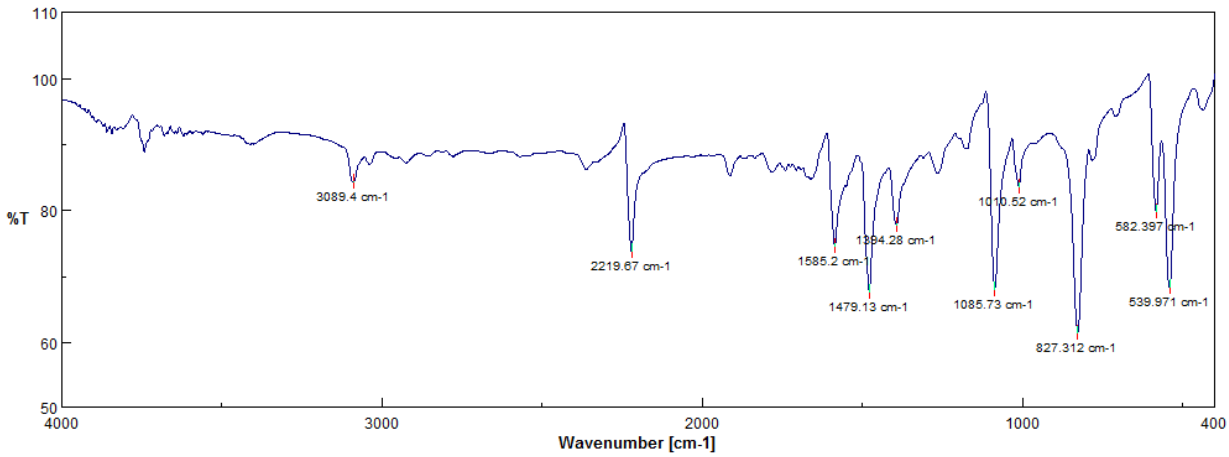

Fig. 76 FT-IR spectrum of 4-chlorobenzonitrile

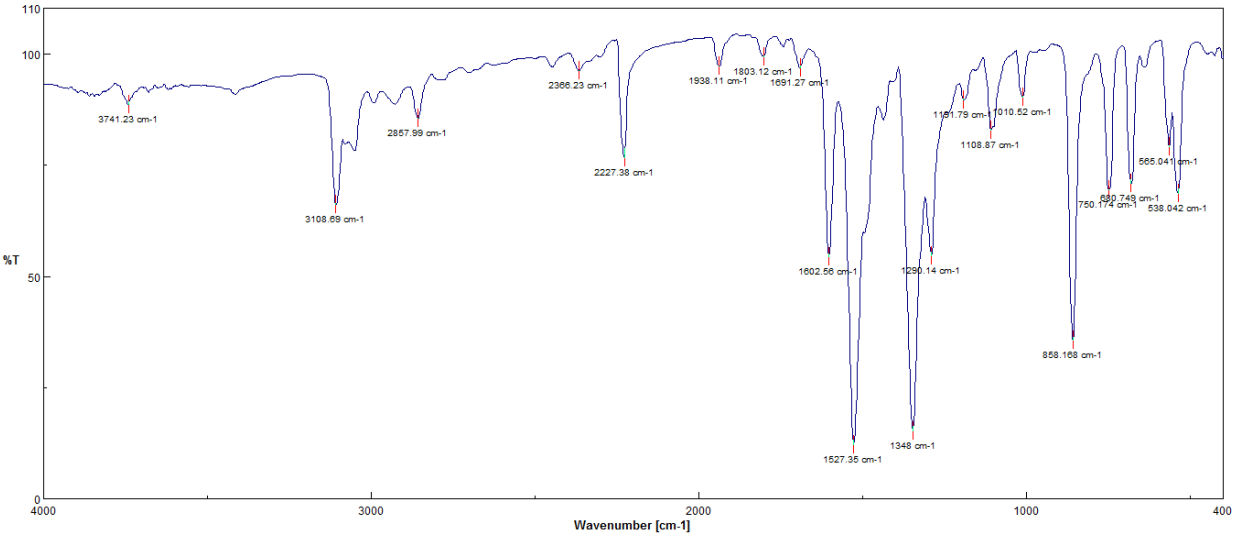

Fig. 77 FT-IR spectrum of 4-nitrobenzonitrile

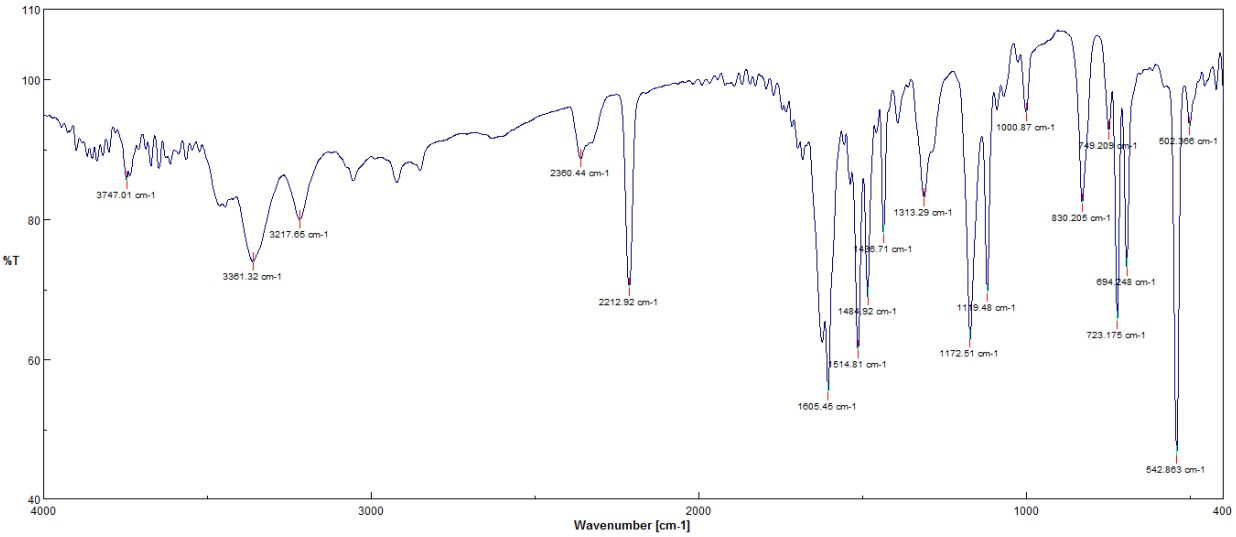

Fig. 78 FT-IR spectrum of 4-aminobenzonitrile

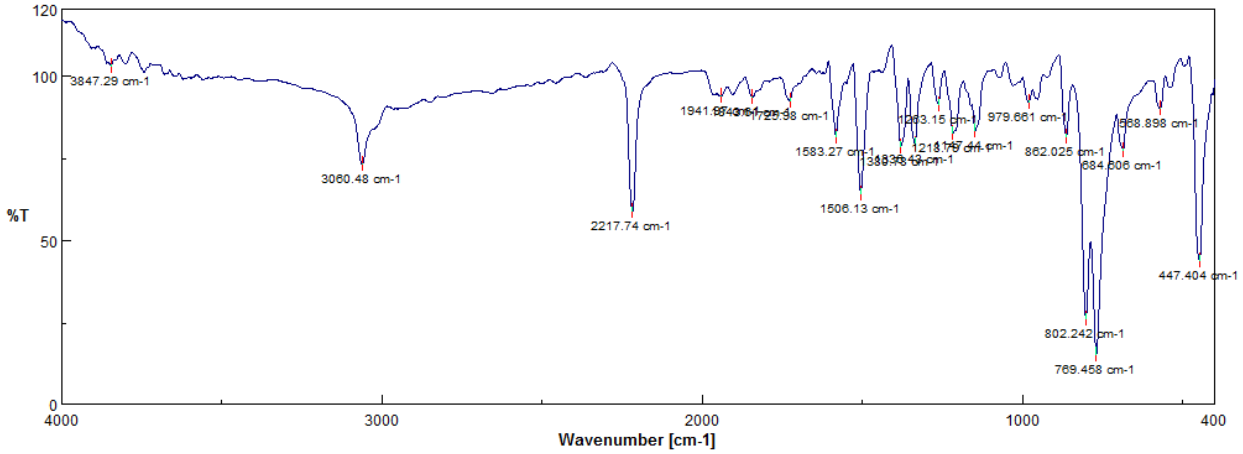

Fig. 79 FT-IR spectrum of 1-cyanonaphthalene

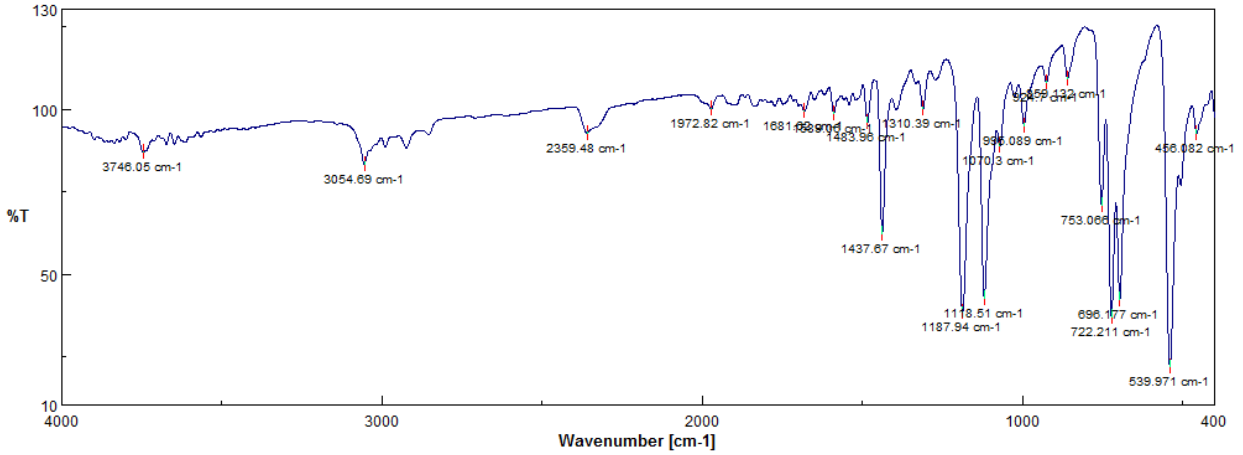

Fig. 80 FT-IR spectrum of 4-(trifluoromethoxy)cyanobenzene

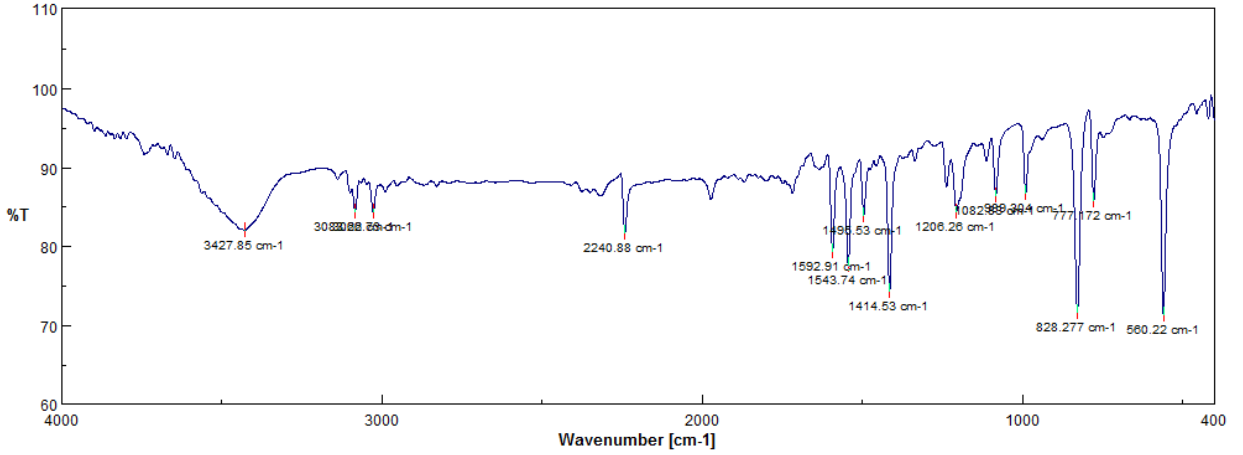

Fig. 81 FT-IR spectrum of 4-cyanopyridine

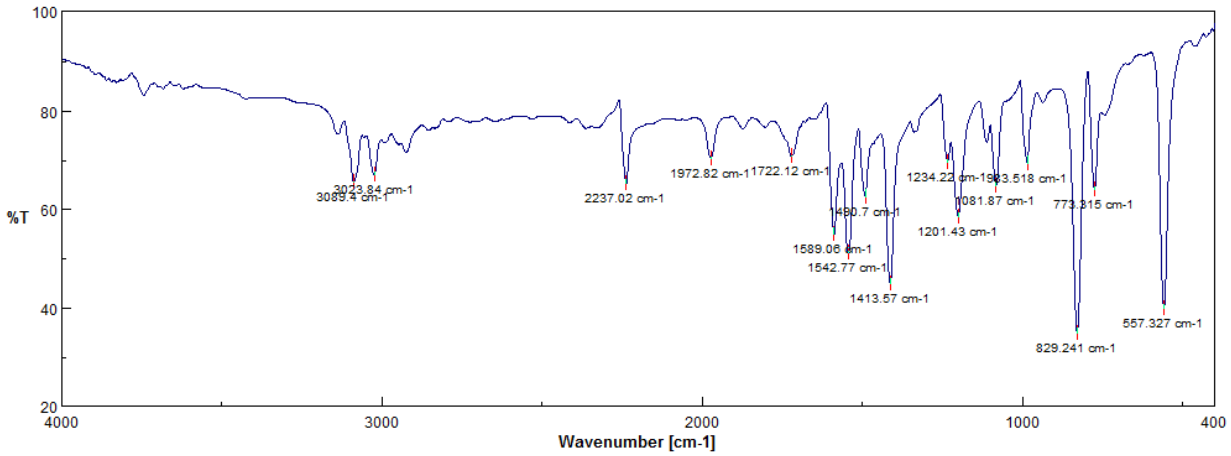

Fig. 82 FT-IR spectrum of 3-cyanopyridine

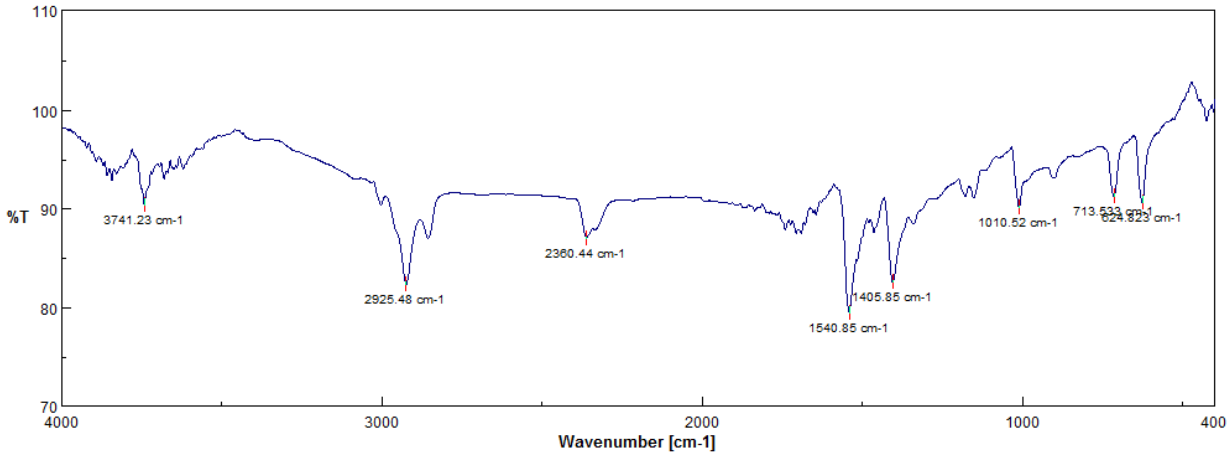

Fig. 83 FT-IR spectrum of 5-cyanopyrimidine

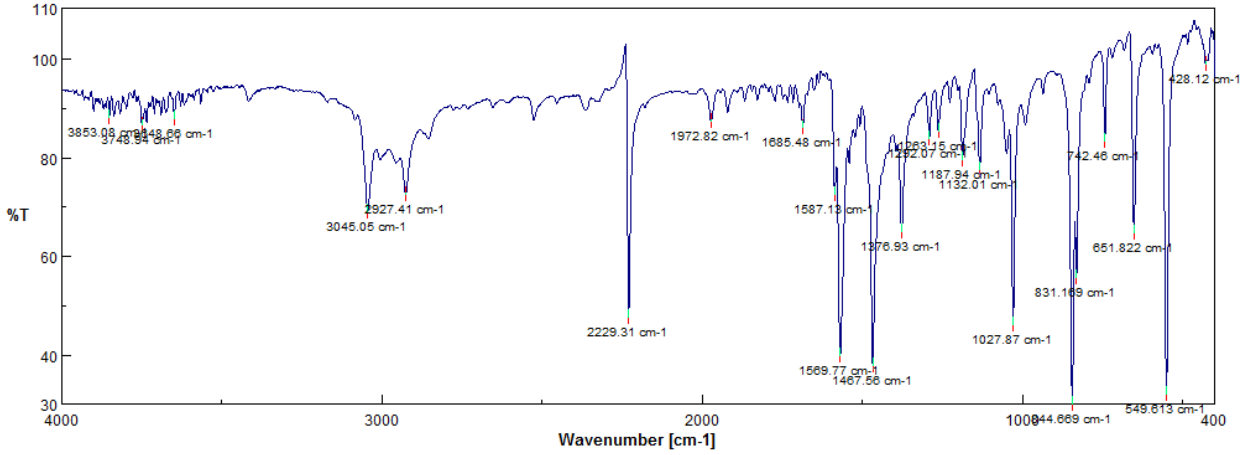

Fig.84FT-IR spectrum of 5-methyl-2-cyanopyridine