

Supplementary Material

Direct Synthesis of Nitriles from Aldehydes and Hydroxylamine Hydrochloride Catalyzed by HAP@AEPH₂-SO₃H Nanocatalyst

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Experimental

General

The purity determinations of the products were accomplished using TLC on silica gel polygram STL G/UV 254 plates. The melting points of products were determined with an Electrothermal type 9100 melting point apparatus. Fourier transform infrared (FT-IR) spectra were recorded on pressed KBr pellets using an AVATAR 370 FT-IR spectrometer (Thermo Nicolet spectrometer, USA) at room temperature in the range between 4000 and 400 cm^{-1} with a resolution of 4 cm^{-1} . NMR spectra were obtained with Bruker AMX 100, 300 and 400 MHz instruments in CDCl_3 . Elemental analyses were performed using a Thermo Finnegan Flash EA 1112 series instrument. Mass spectra were recorded with a CH7A Varianmat Bremem instrument at 70 eV electron impact ionization, in m/z (rel %). All of the products were known compounds and characterized using FT-IR spectroscopy, mass spectrometry and comparison of their melting points with known compounds. The structure of selected products was further confirmed using ^1H NMR spectroscopy.

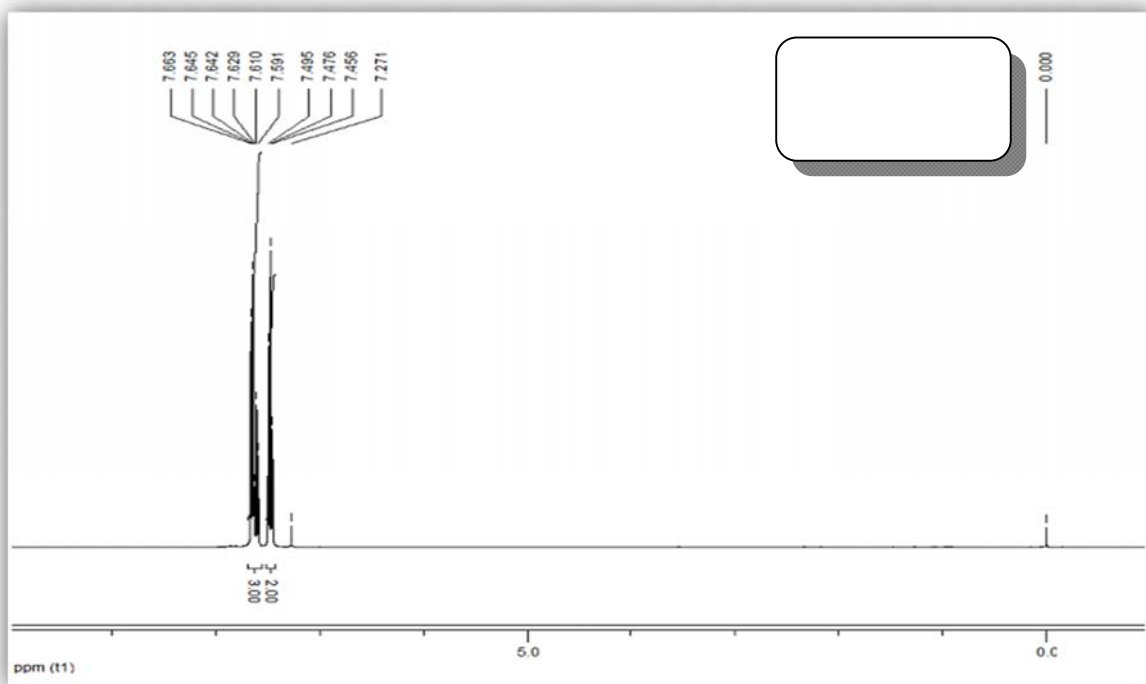


Fig 1a: ¹H NMR (400 MHz, CDCl₃) of Benzonitrile (Table 4, Entry 1)

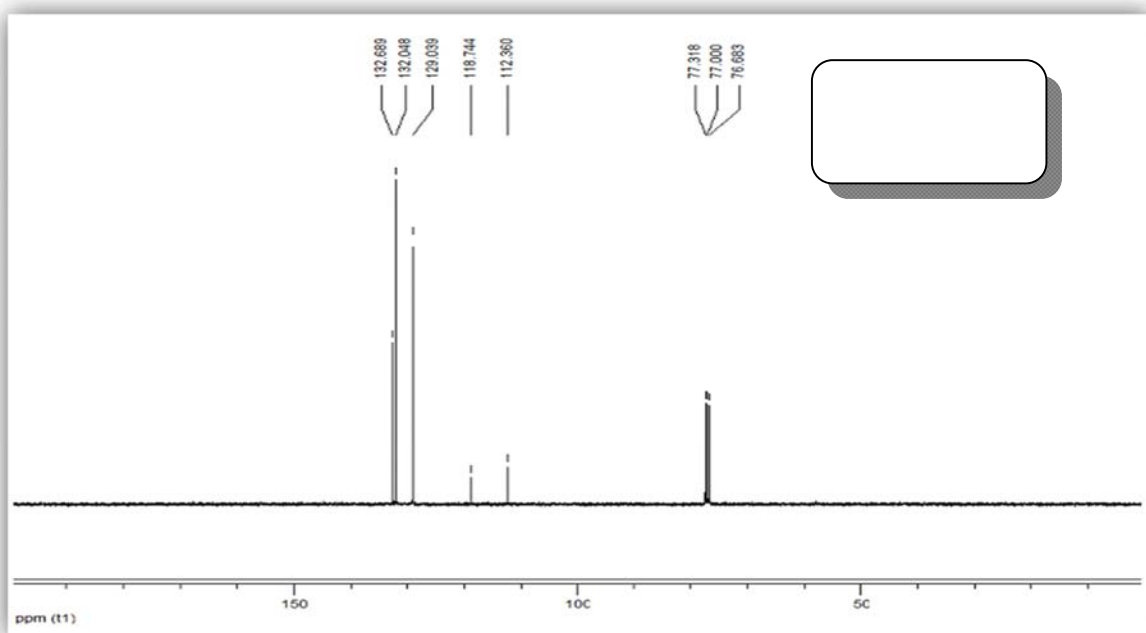


Fig 1b: ¹³C NMR (100 MHz, CDCl₃) of Benzonitrile (Table 4, Entry 1)

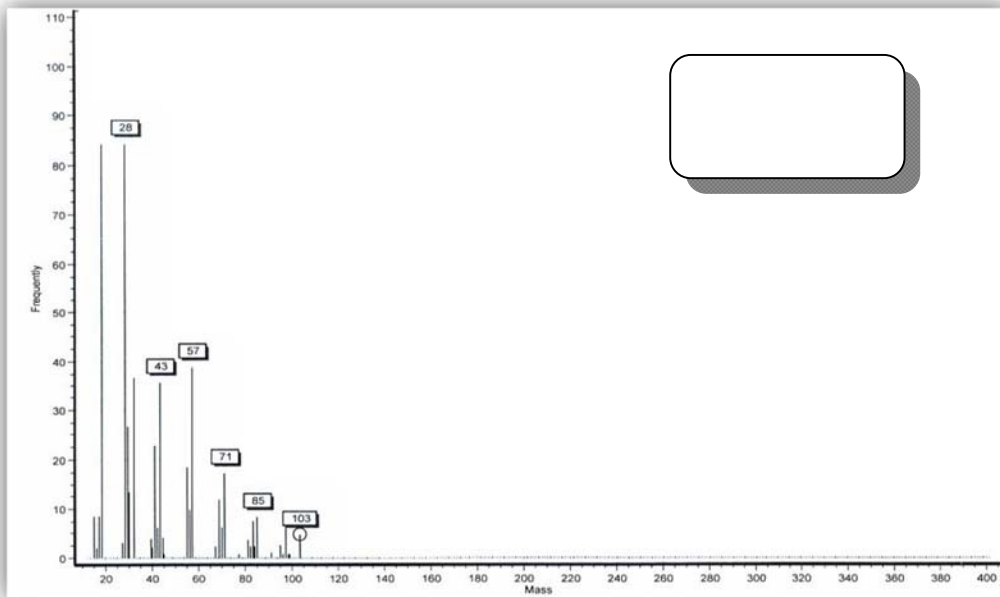


Fig 1c: Mass spectrum of Benzonitrile (Table 4, Entry 1)

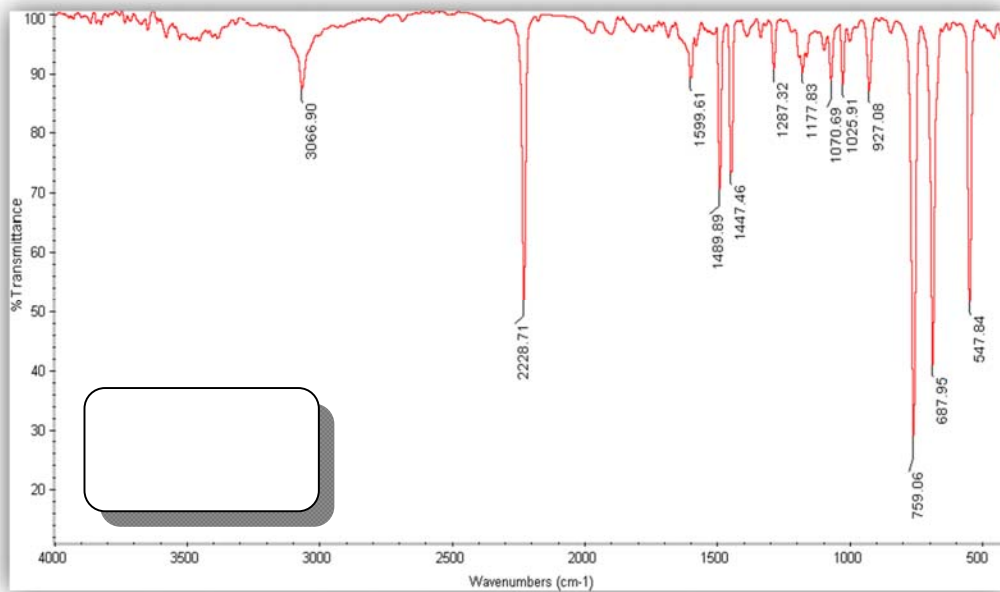


Fig 1d: FT-IR (neat) spectrum of Benzonitrile (Table 4, Entry 1)

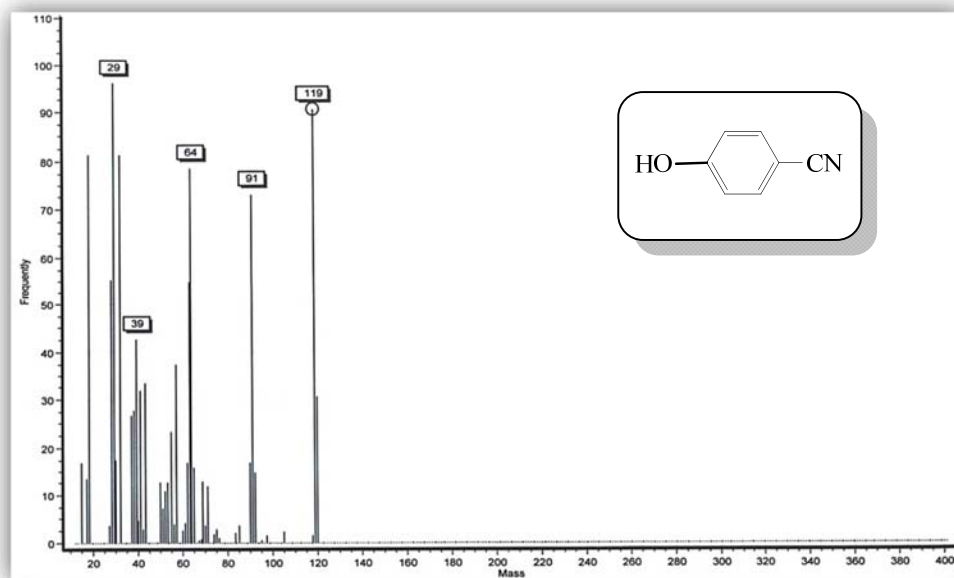


Fig 2a: Mass spectrum of 4-Hydroxybenzonitrile (Table 4, Entry 2)

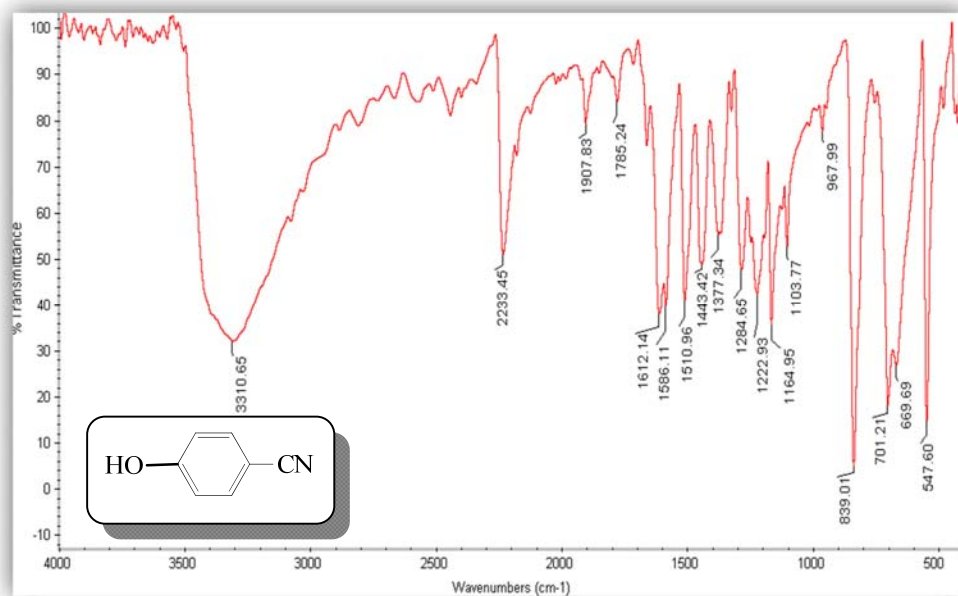


Fig 2b: FT-IR (KBr) spectrum of 4-Hydroxybenzonitrile (Table 4, Entry 2)

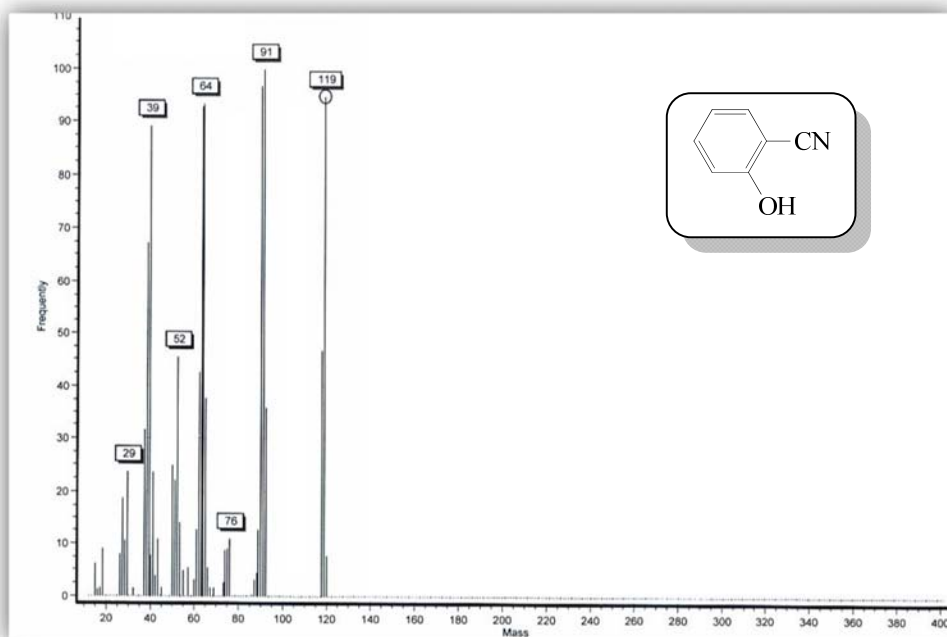


Fig 3a: Mass spectrum of 2-Hydroxybenzonitrile (Table 4, Entry 3)

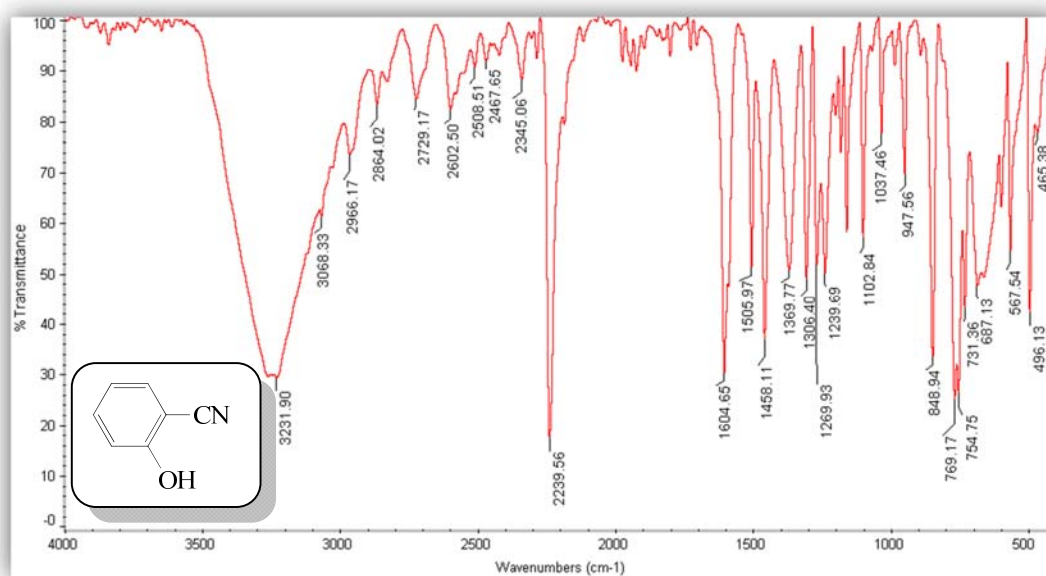


Fig 3b: FT-IR (KBr) spectrum of 2-Hydroxybenzonitrile (Table 4, Entry 3)

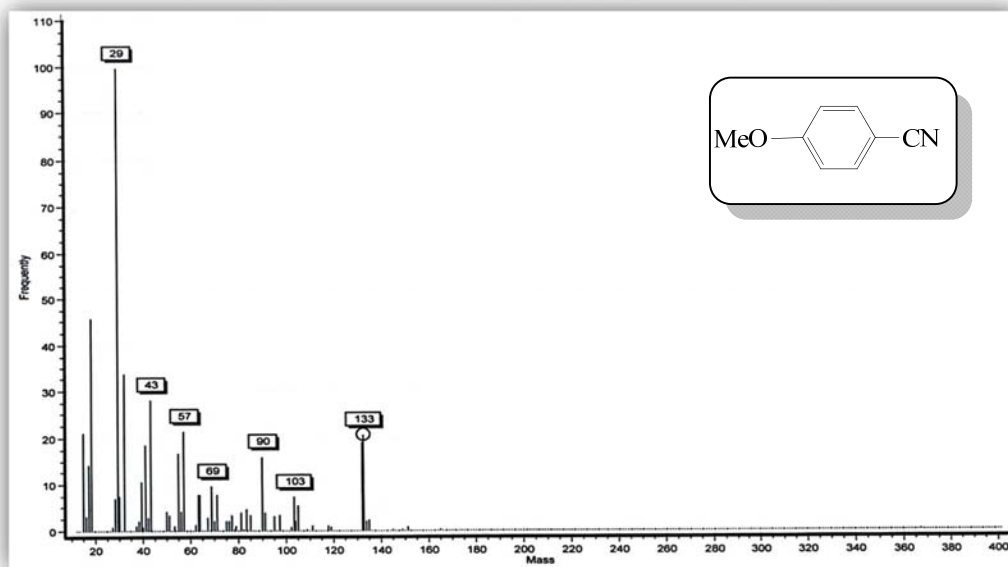


Fig 4a: Mass spectrum of 4-Methoxybenzonitrile (Table 4, Entry 4)

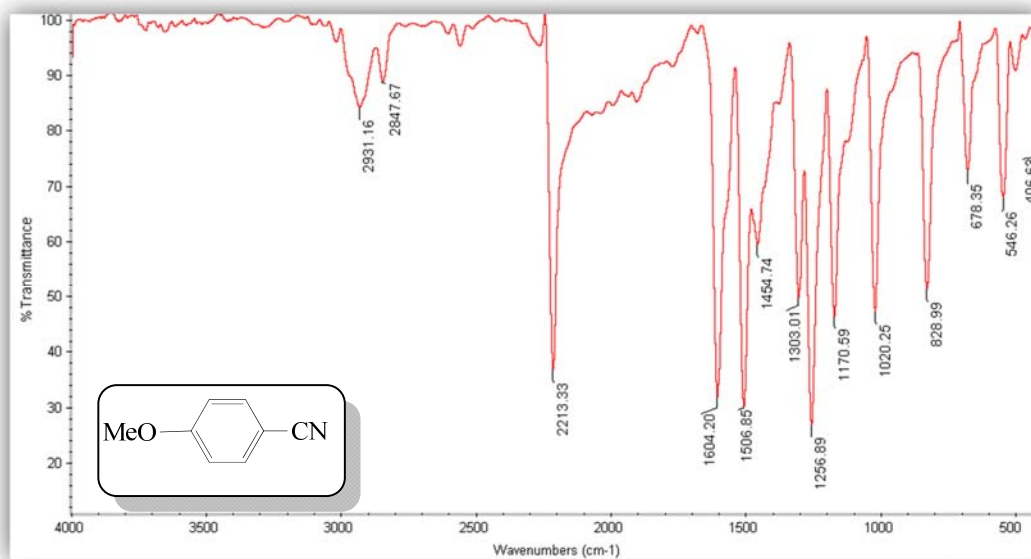


Fig 4b: FT-IR (KBr) spectrum of 4-Methoxybenzonitrile (Table 4, Entry 4)

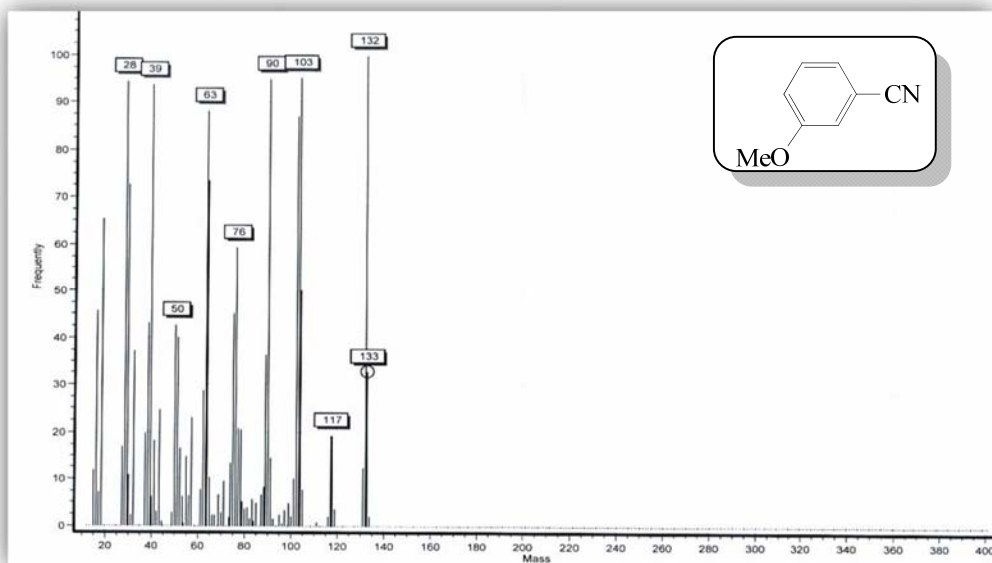


Fig 5a: Mass spectrum of 3-Methoxybenzonitrile (Table 4, Entry 5)

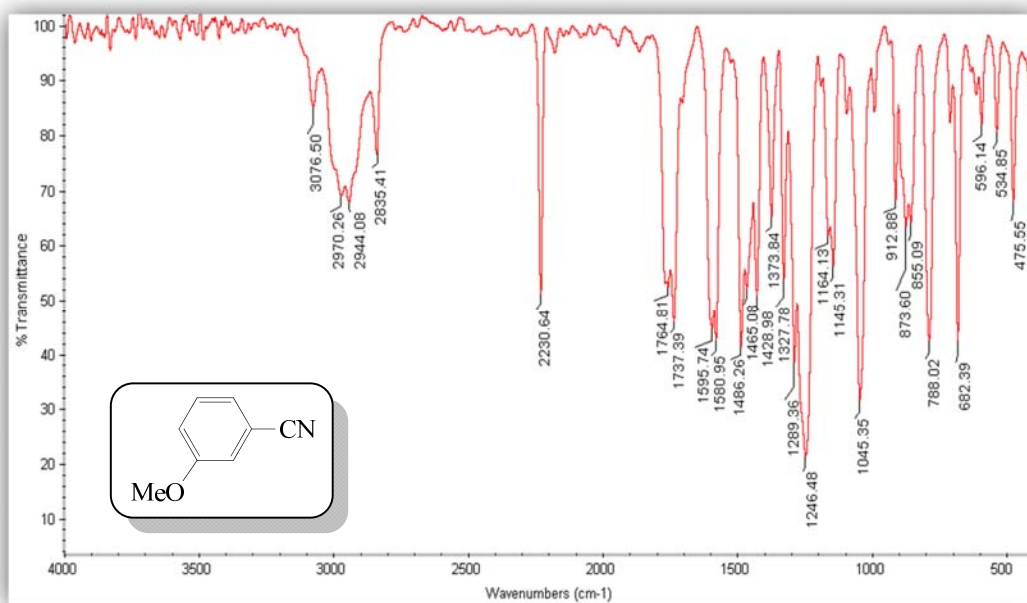


Fig 5b: FT-IR (neat) spectrum of 3-Methoxybenzonitrile (Table 4, Entry 5).

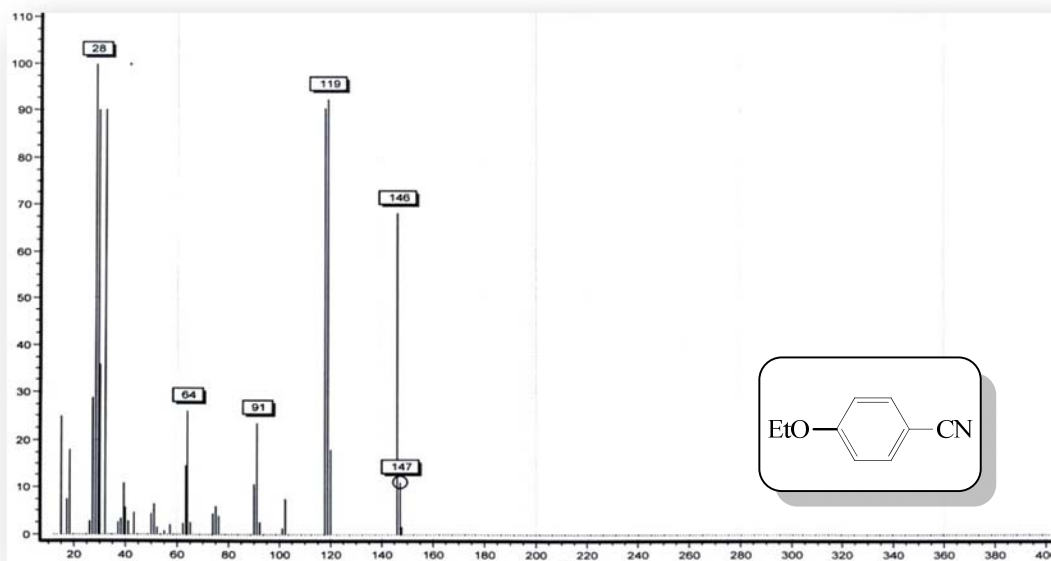


Fig 6a: Mass spectrum of 4-Ethoxybenzonitrile (Table 4, Entry 6).

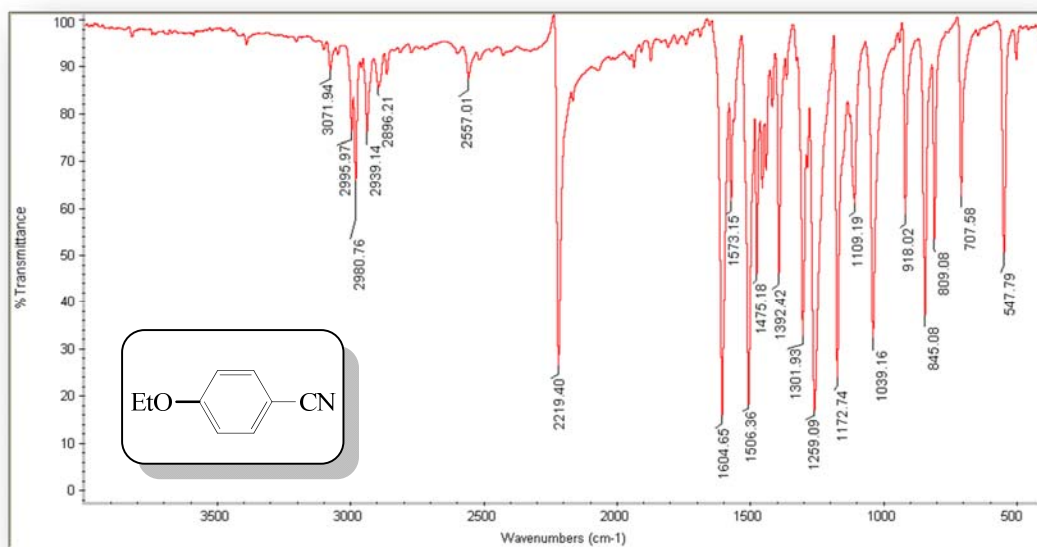


Fig 6b: FT-IR spectrum of 4-Ethoxybenzonitrile (Table 4, Entry 6).

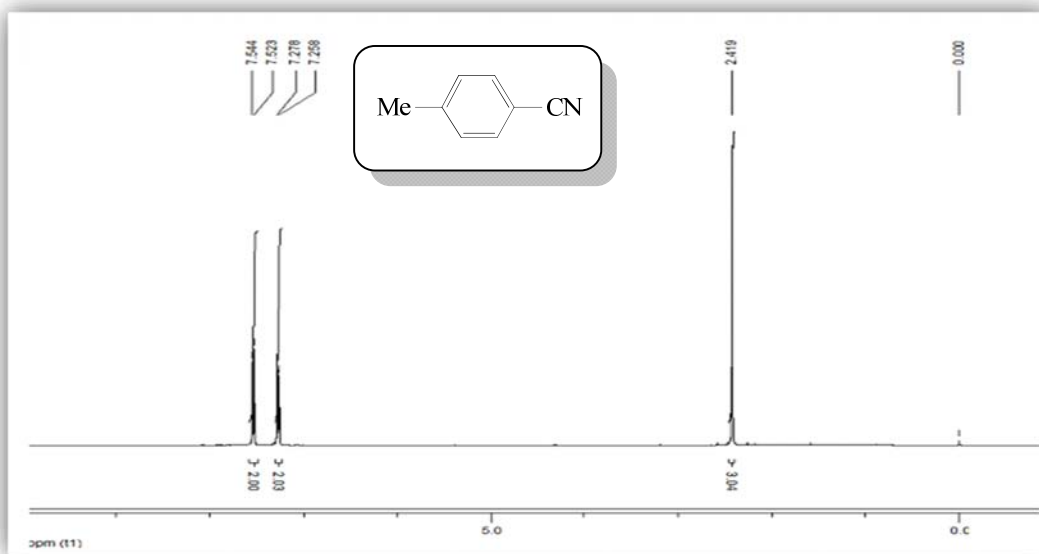


Fig 7a: ¹H NMR (400 MHz, CDCl₃) of 4-Methylbenzonitrile (Table 4, Entry 7).

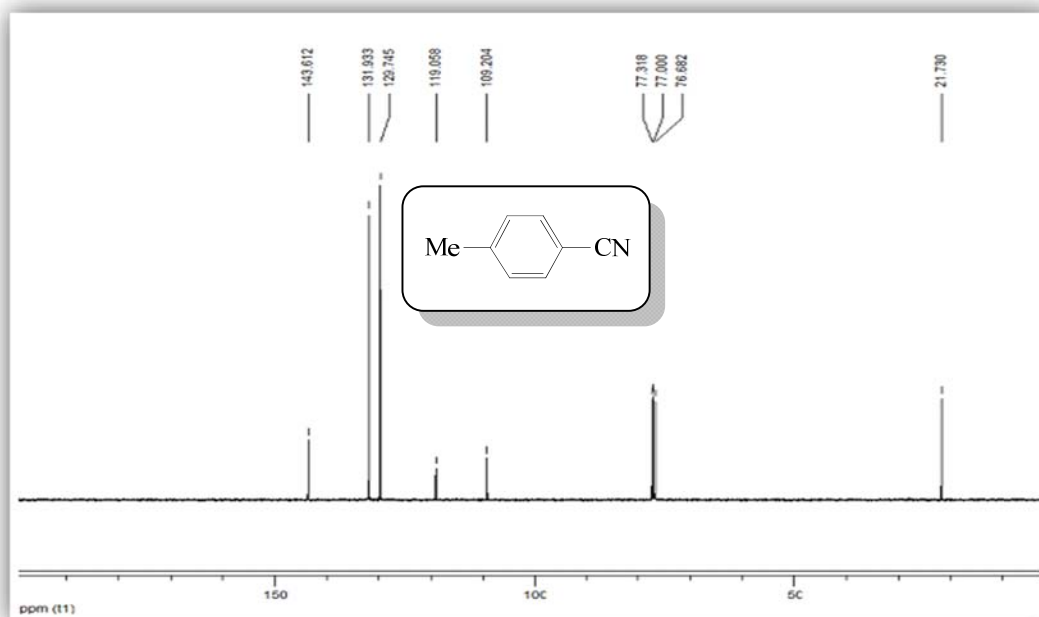


Fig 7b: ¹³C NMR (100 MHz, CDCl₃) of 4-Methylbenzonitrile (Table 4, Entry 7).

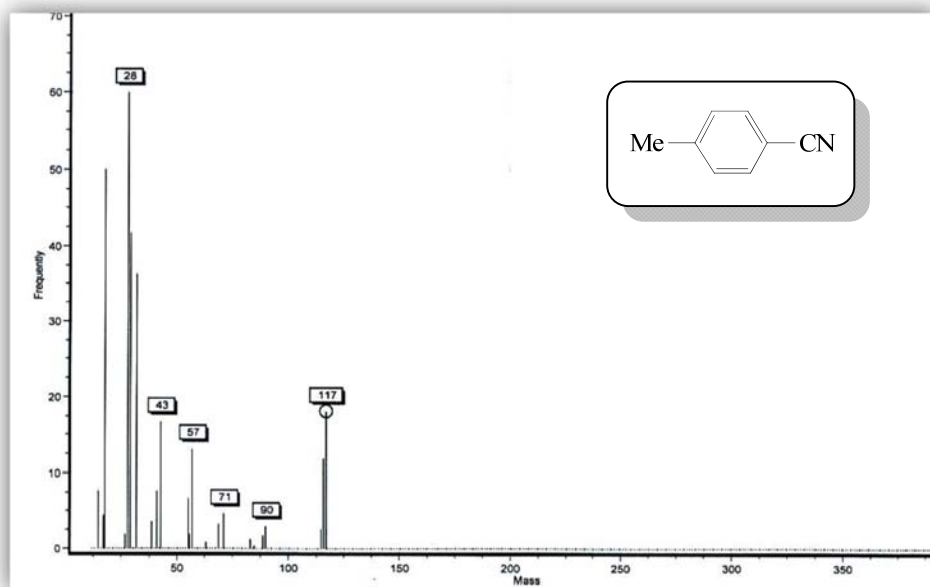


Fig 7c: Mass spectrum of 4-Methylbenzonitrile (Table 4, Entry 7).

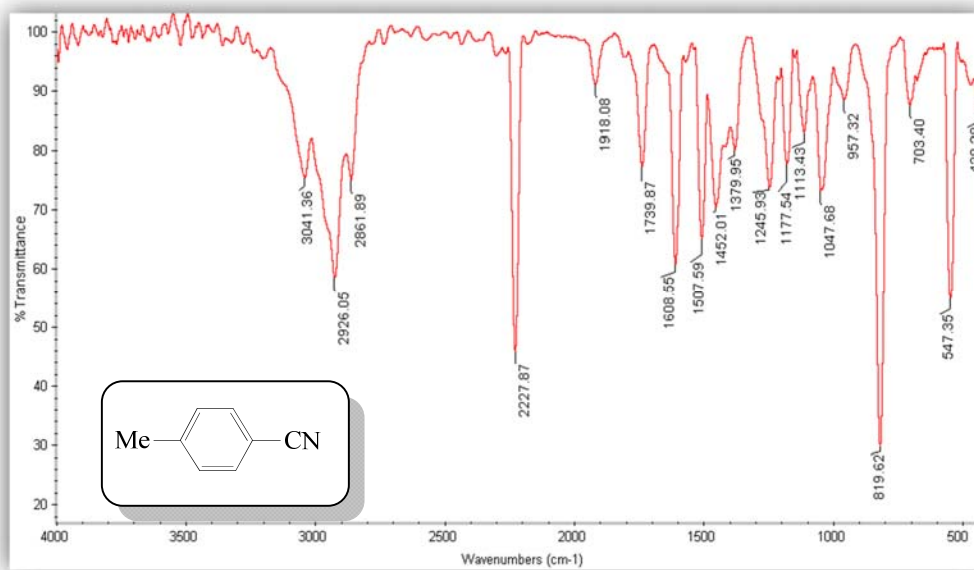


Fig 7d: FT-IR (neat) spectrum of 4-Methylbenzonitrile (Table 4, Entry 7).

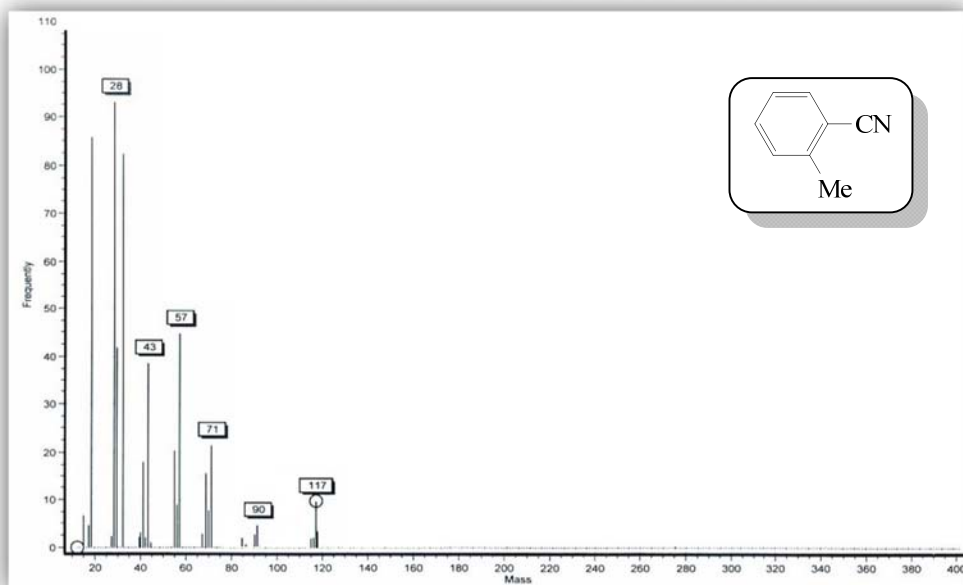


Fig 8a: Mass spectrum of 2-Methylbenzonitrile (Table 4, Entry 8).

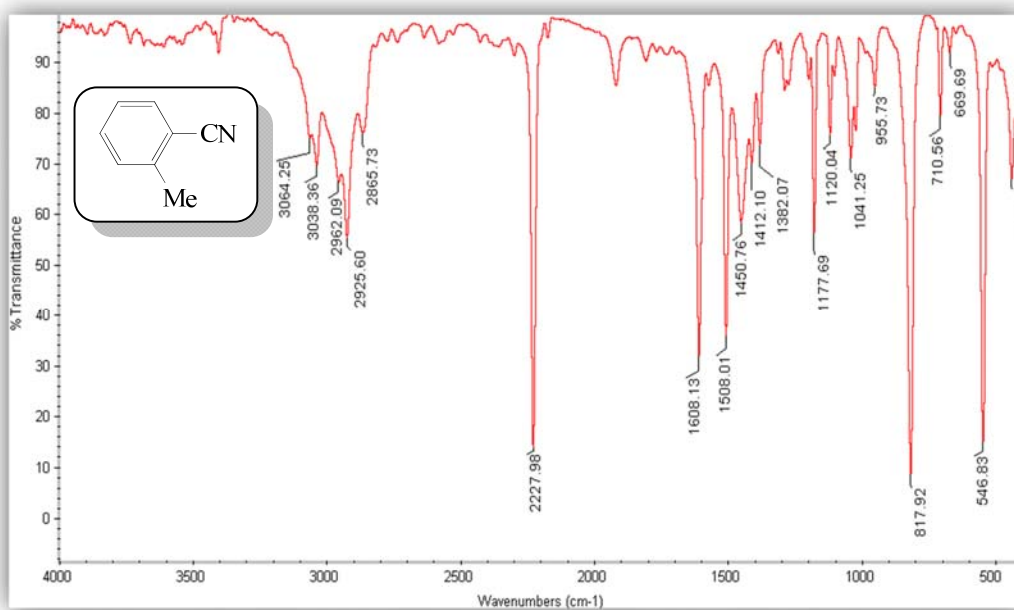


Fig 8b: FT-IR (neat) spectrum of 2-Methylbenzonitrile (Table 4, Entry 8).

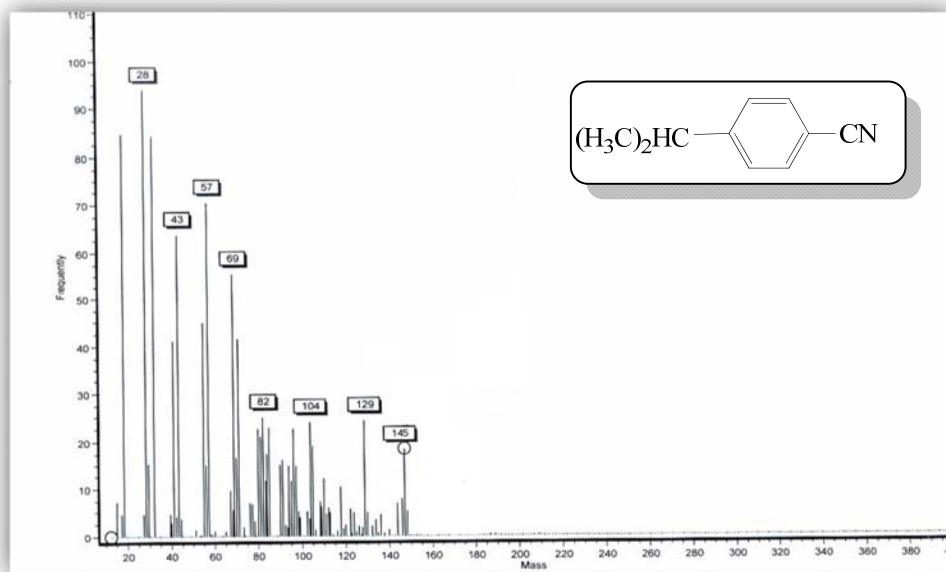


Fig 9a: Mass spectrum of 4-Isopropylbenzotrile (Table 4, Entry 9).

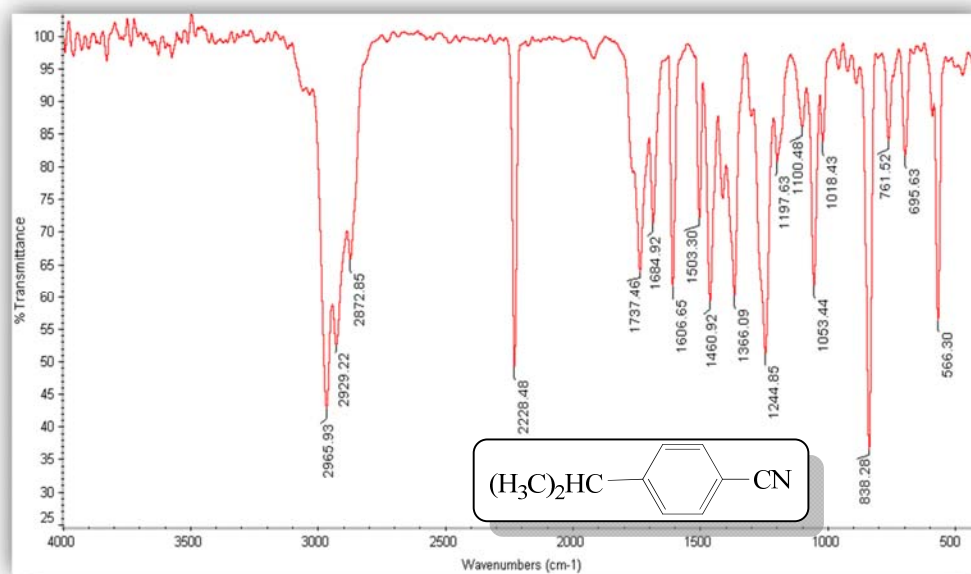


Fig 9b: FT-IR (neat) spectrum of 4-Isopropylbenzotrile (Table 4, Entry 9).

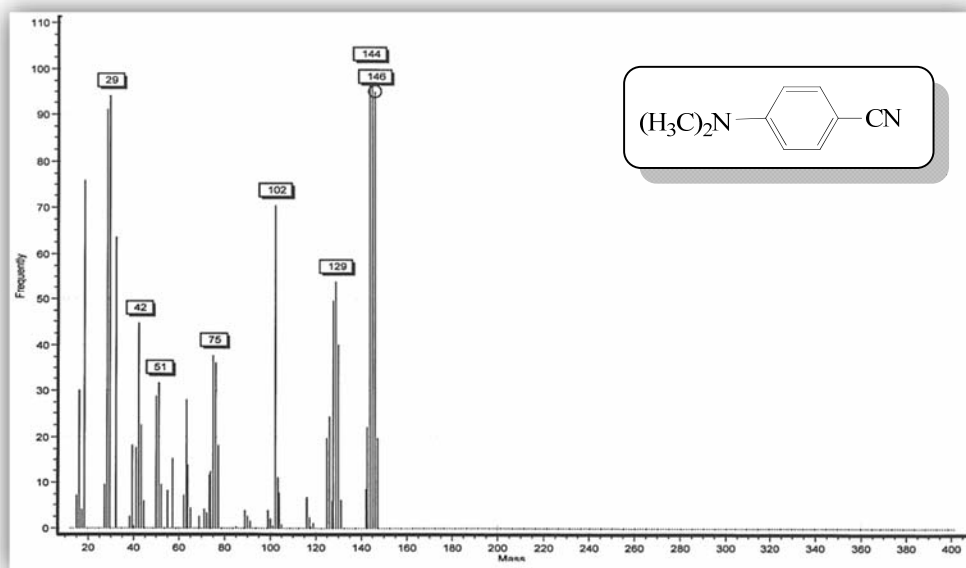


Fig 10a: Mass spectrum of 4-(Dimethylamino)benzonitrile (Table 4, Entry 10).

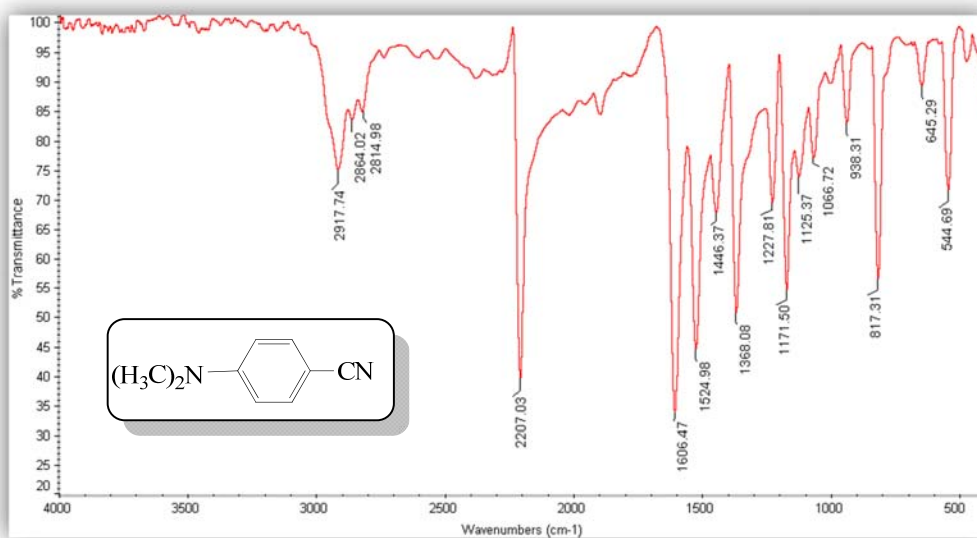


Fig 10b: FT-IR (neat) spectrum of 4-(Dimethylamino)benzonitrile (Table 4, Entry 10).

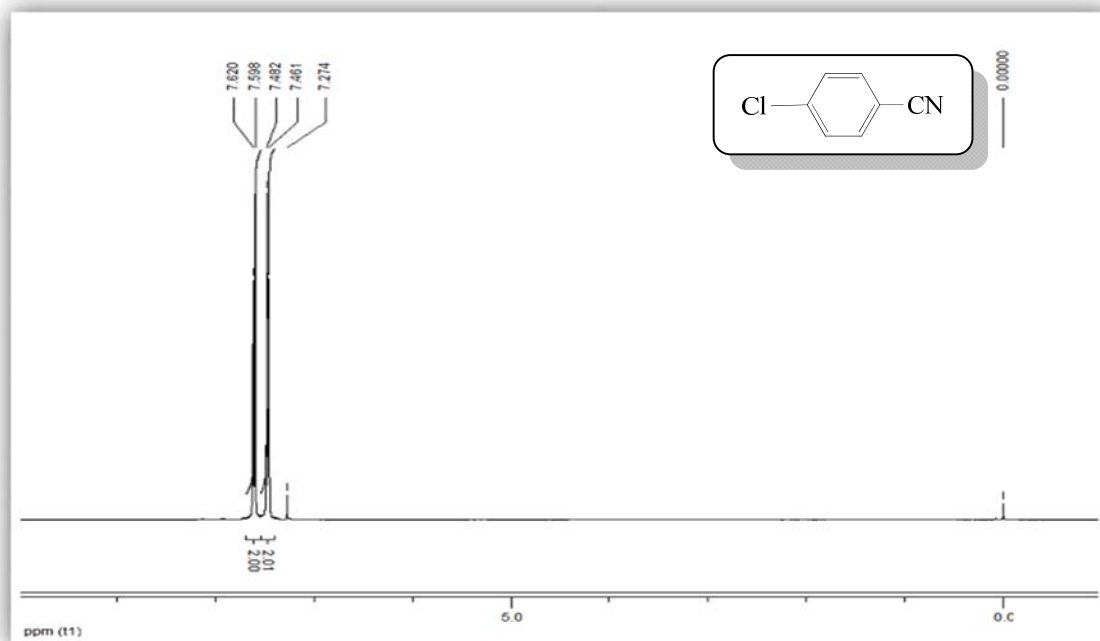


Fig 11a: ^1H NMR (400 MHz, CDCl_3) of 4-Chlorobenzonitrile (Table 4, Entry 11).

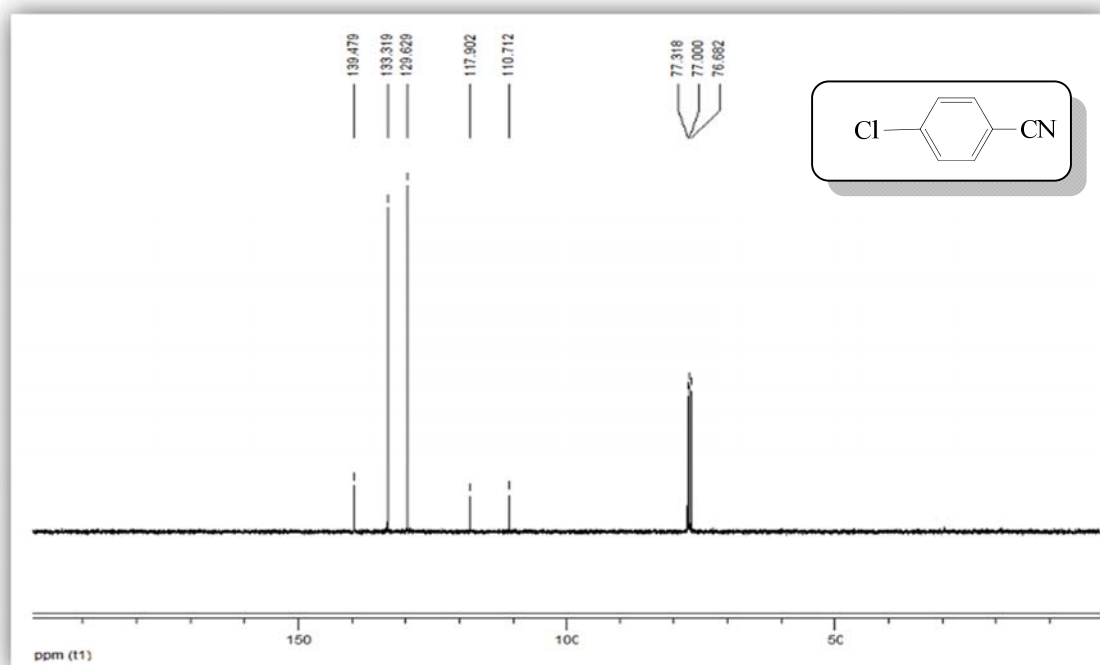


Fig 11b: ^{13}C NMR (100 MHz, CDCl_3) of 4-Chlorobenzonitrile (Table 4, Entry 11).

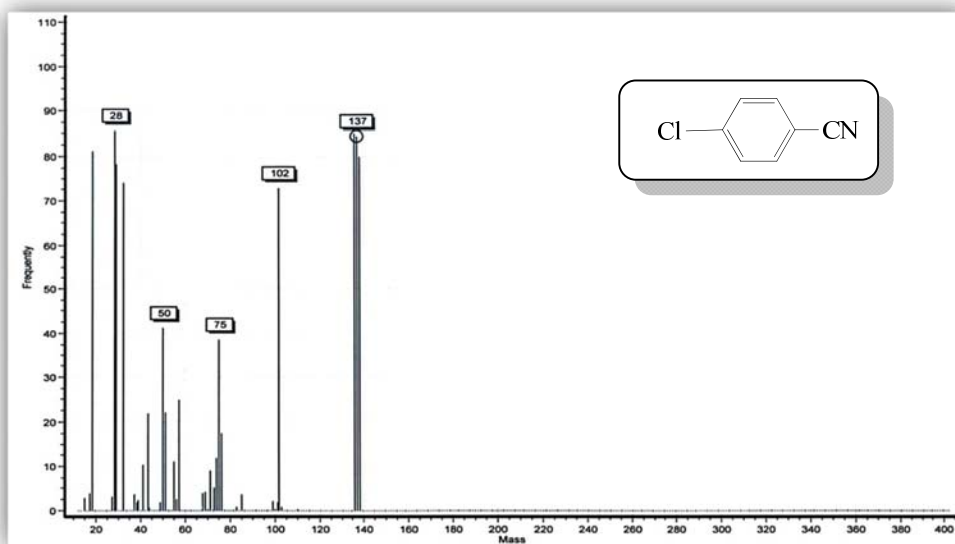


Fig 11c: Mass spectrum of 4-Chlorobenzonitrile (Table 4, Entry 11).

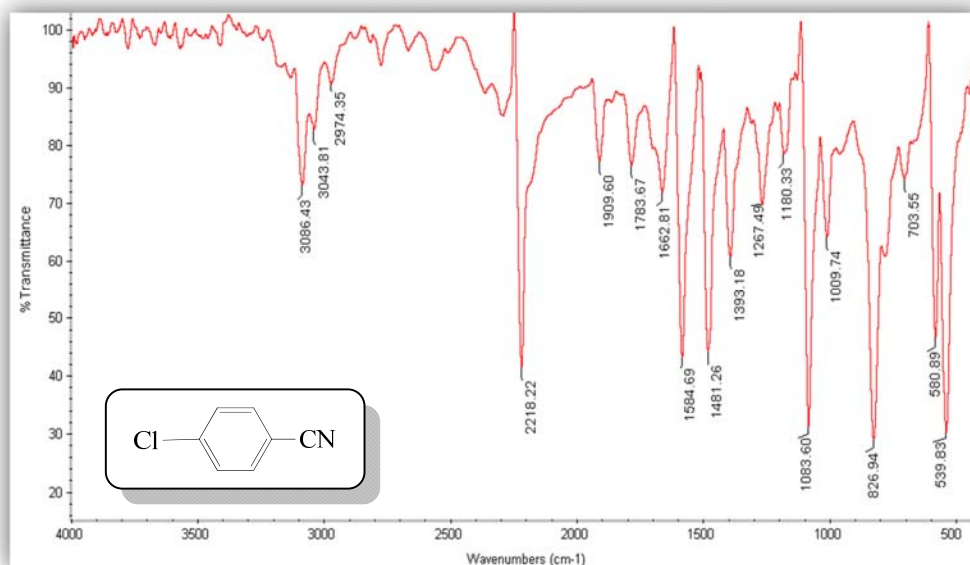


Fig 11d: FT-IR (KBr) spectrum of 4-Chlorobenzonitrile (Table 4, Entry 11).

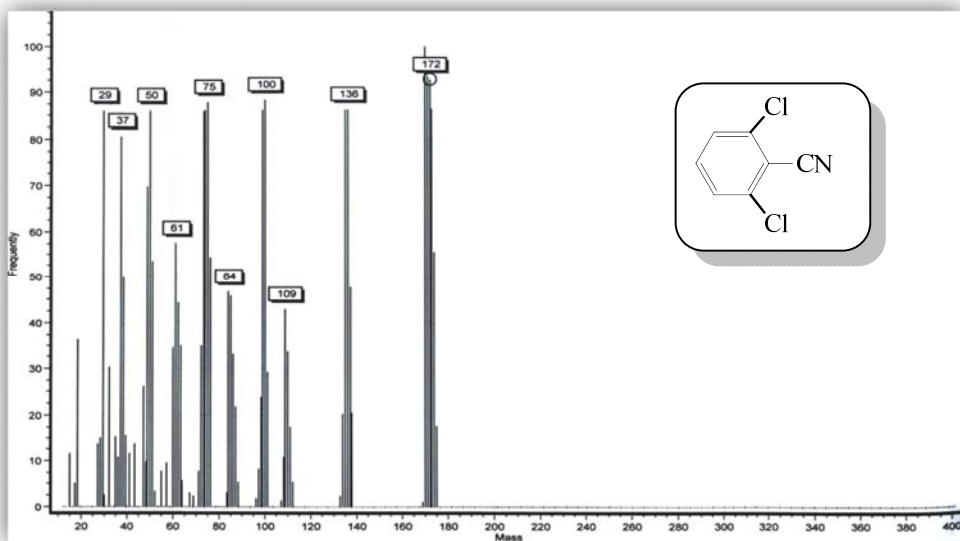


Fig 12a: Mass spectrum of 2,6-Dichlorobenzonitrile (Table 4, Entry 12).

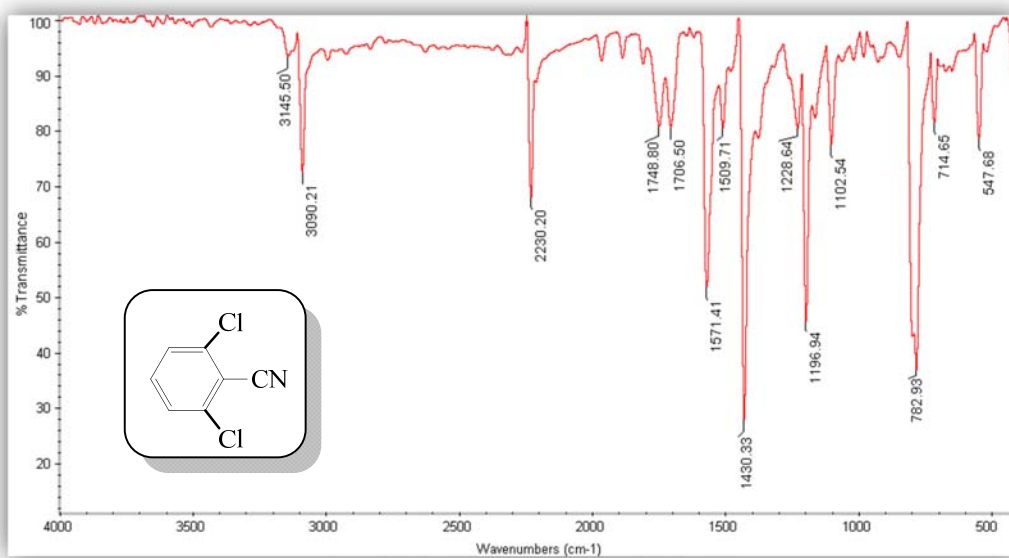


Fig 12b: FT-IR (KBr) spectrum of 2,6-Dichlorobenzonitrile (Table 4, Entry 12).

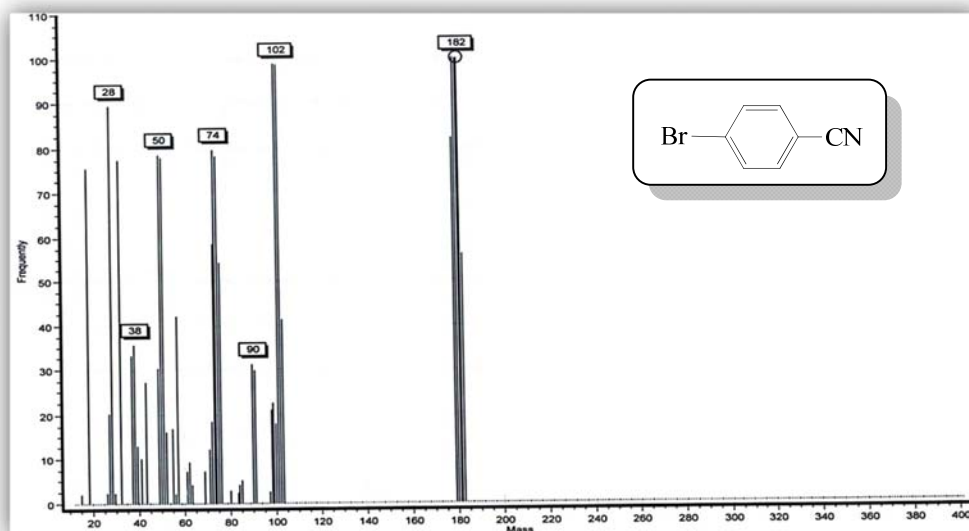


Fig 13a: Mass spectrum of 4-Bromobenzonitrile (Table 4, Entry 13).

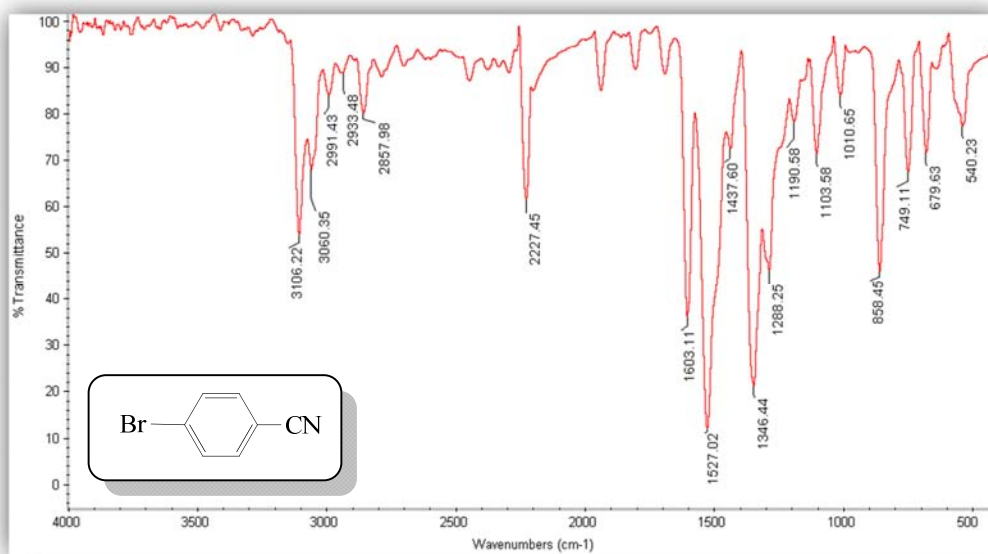


Fig 13b: FT-IR (KBr) spectrum of 4-Bromobenzonitrile (Table 4, Entry 13).

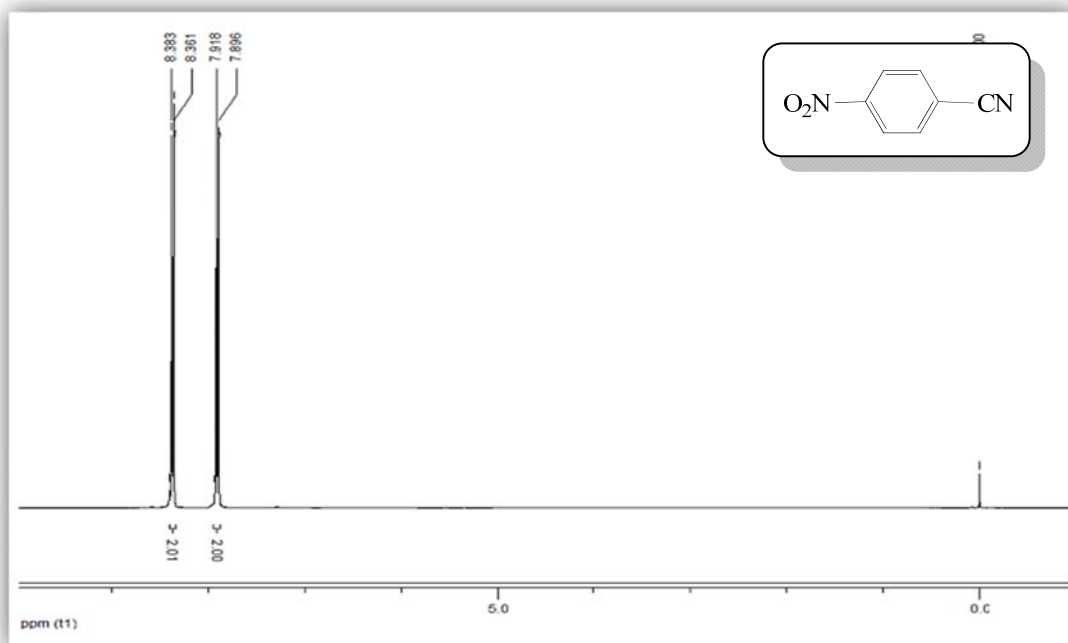


Fig 14a: ¹H NMR (400 MHz, CDCl₃) of 4-Nitrobenzonitrile (Table 4, Entry 14).

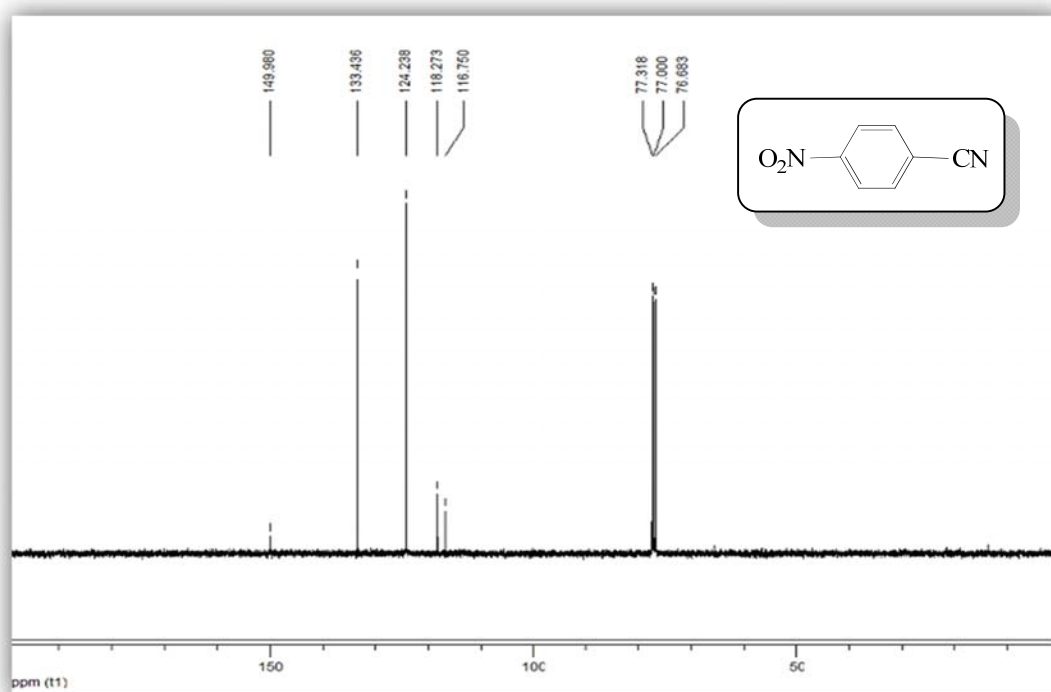


Fig 14b: ¹³C NMR (100 MHz, CDCl₃) of 4-Nitrobenzonitrile (Table 4, Entry 14).

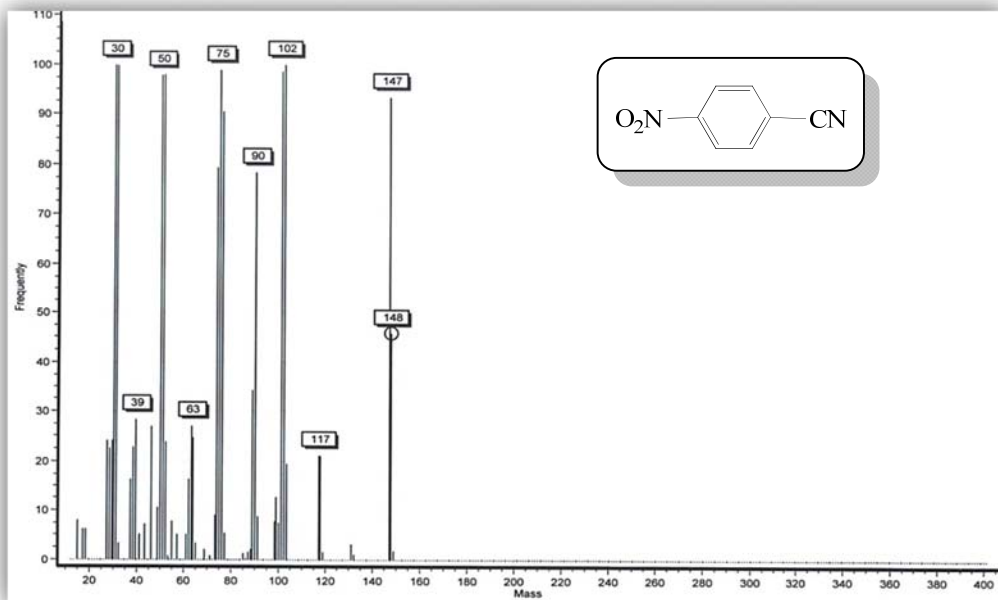


Fig 14c: Mass spectrum of 4-Nitrobenzonitrile (Table 4, Entry 14).

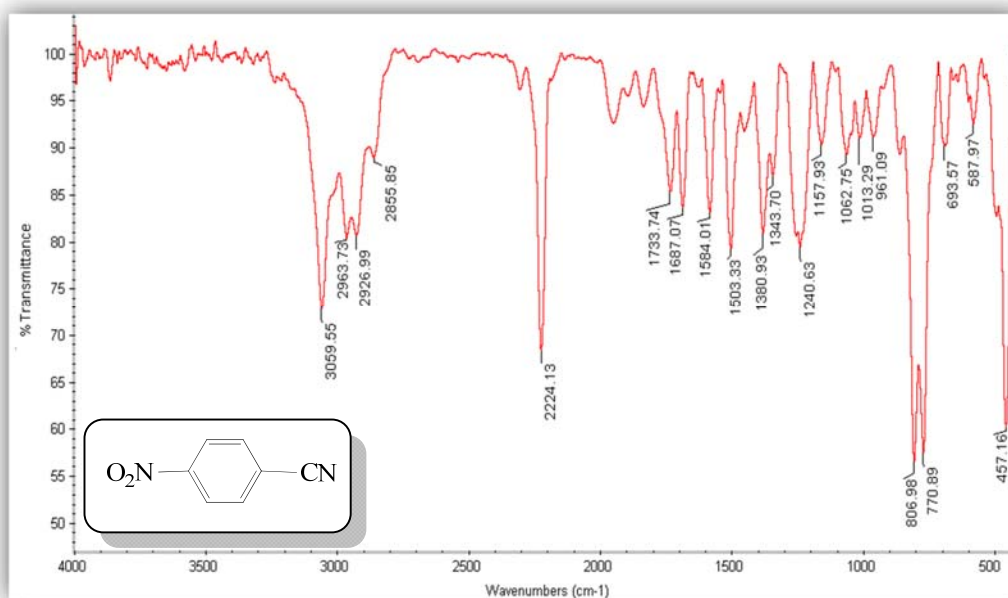


Fig 14d: FT-IR (KBr) spectrum of 4-Nitrobenzonitrile (Table 4, Entry 14).

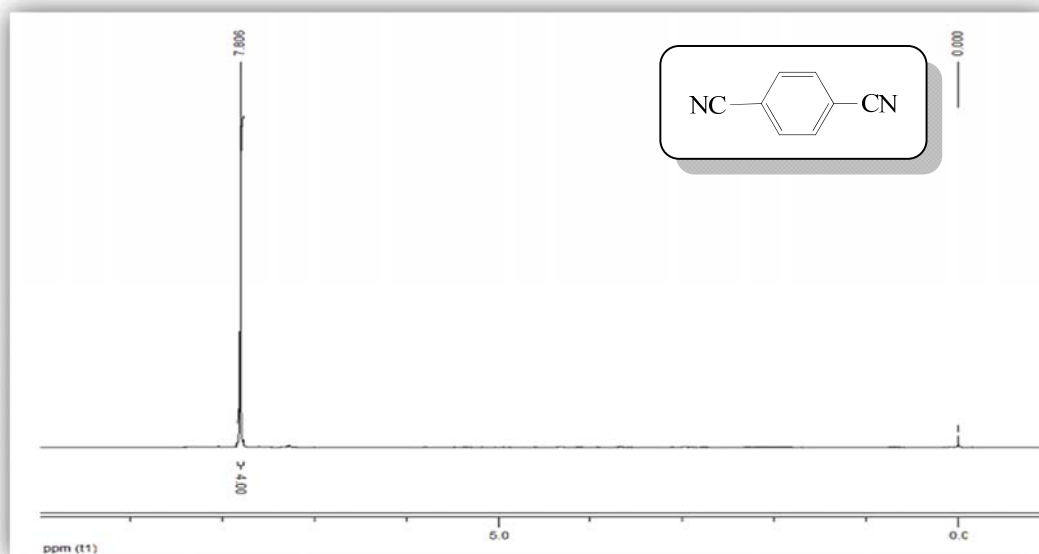


Fig 15a: ^1H NMR (400 MHz, CDCl_3) of Terephthalonitrile (Table 4, Entry 15).

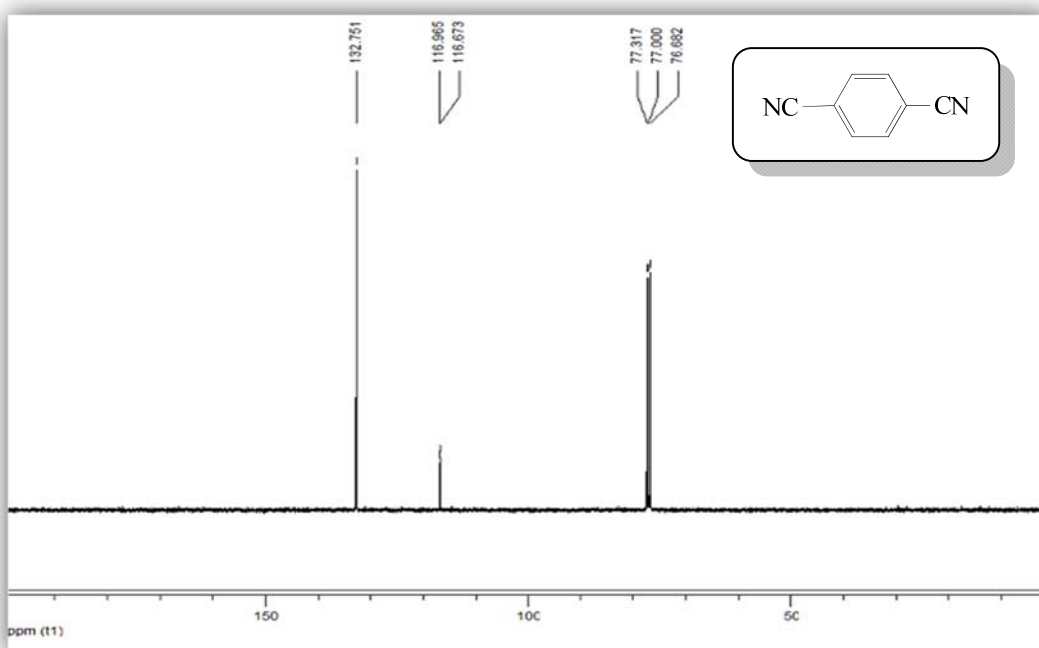


Fig 15b: ^{13}C NMR (100 MHz, CDCl_3) of Terephthalonitrile (Table 4, Entry 15).

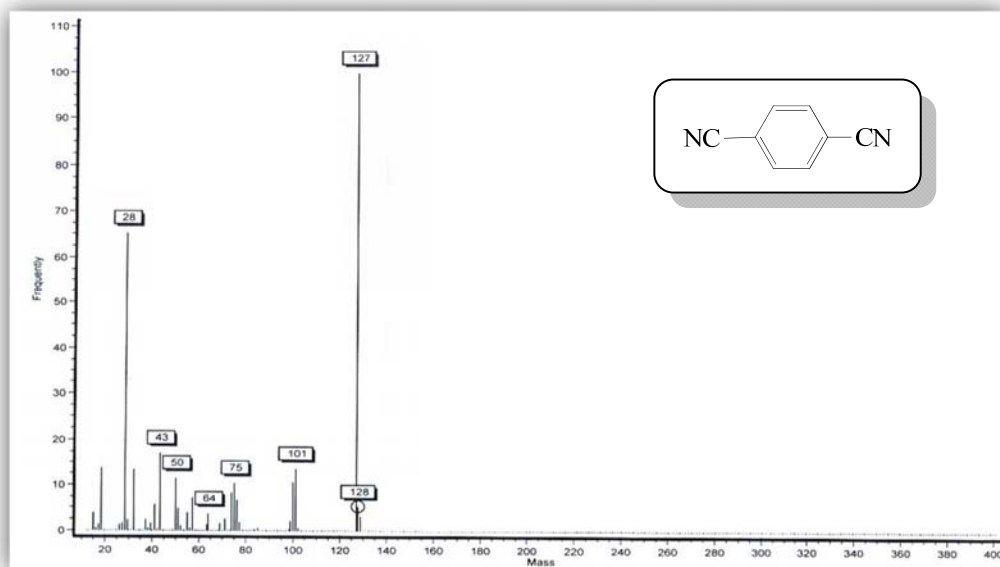


Fig 15c: Mass spectrum of Terephthalonitrile (Table 4, Entry 15).

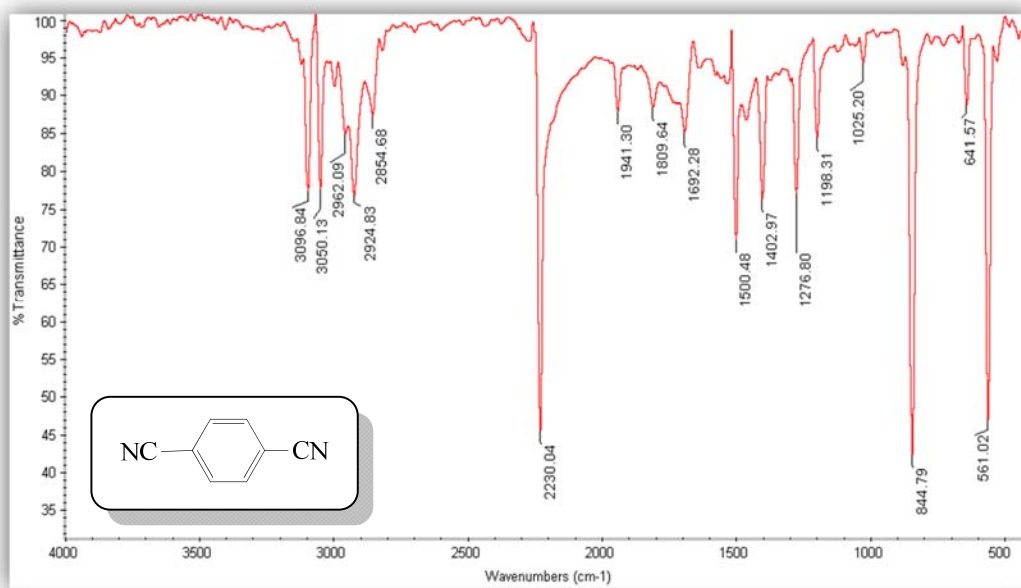


Fig 15d: FT-IR (KBr) spectrum of Terephthalonitrile (Table 4, Entry 15).

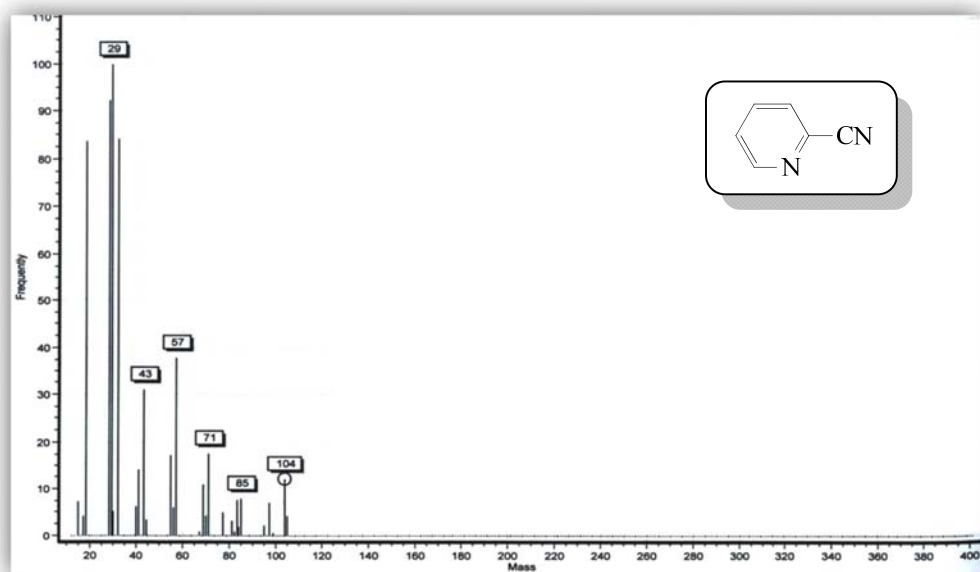


Fig 16a: Mass spectrum of 2-Cyanopyridine (Table 4, Entry 16).

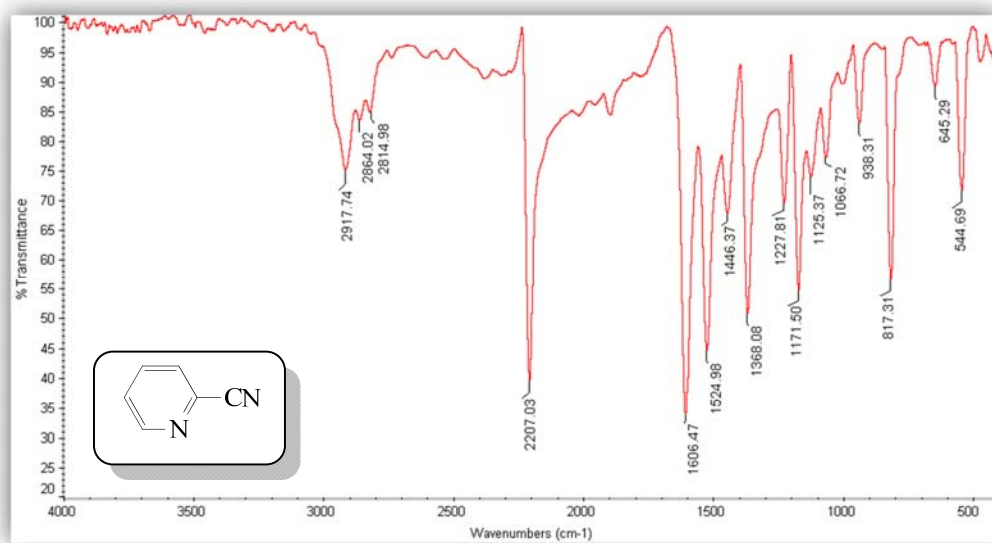


Fig 16b: FT-IR (neat) spectrum of 2-Cyanopyridine (Table 4, Entry 16).

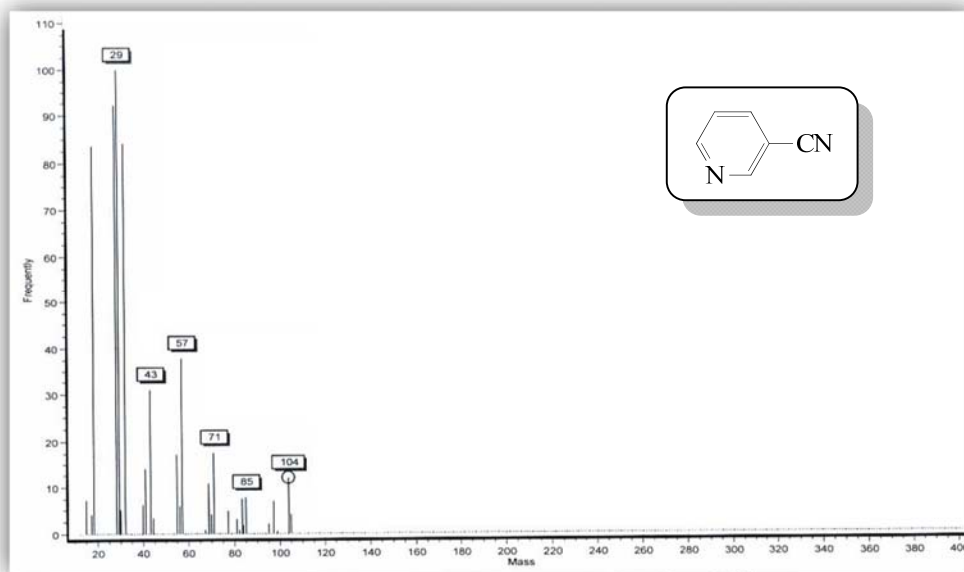


Fig 17a: Mass spectrum of 3-Cyanopyridine (Table 4, Entry 17).

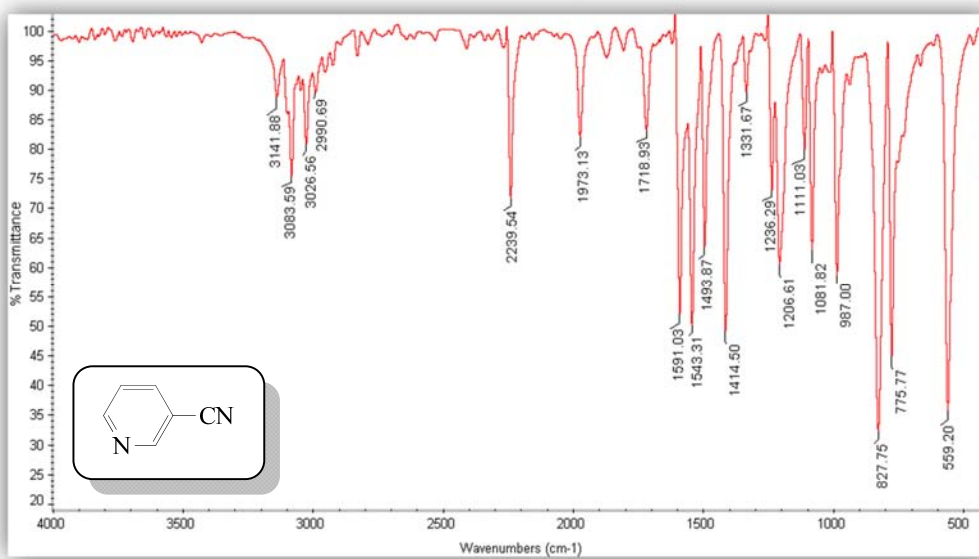


Fig 17b: FT-IR (KBr) spectrum of 3-Cyanopyridine (Table 4, Entry 17).

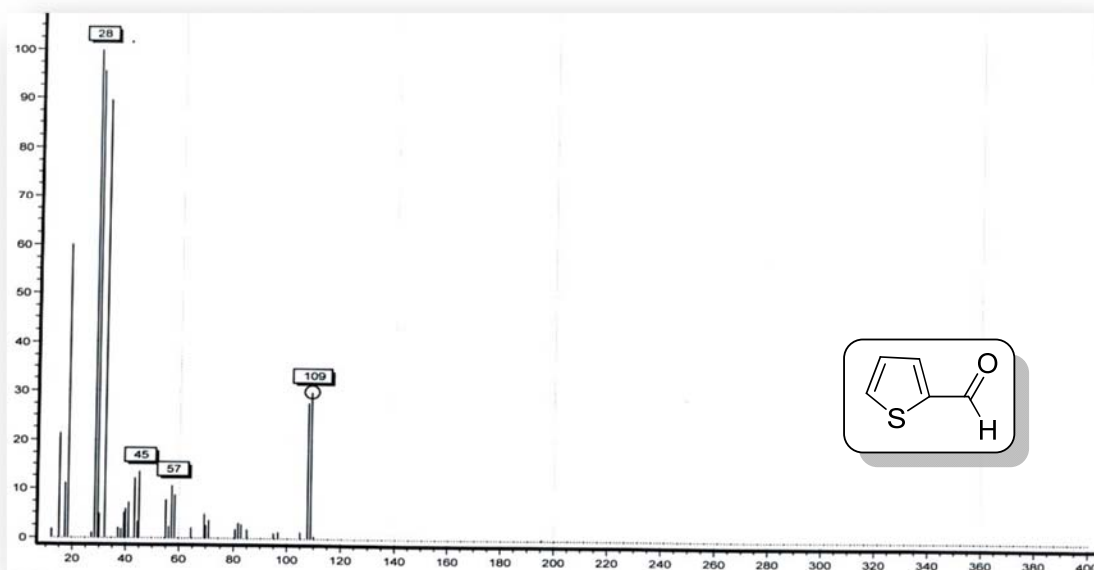


Fig 18a: Mass spectrum of Thiophen-2-carbonitrile (Table 4, Entry 18).

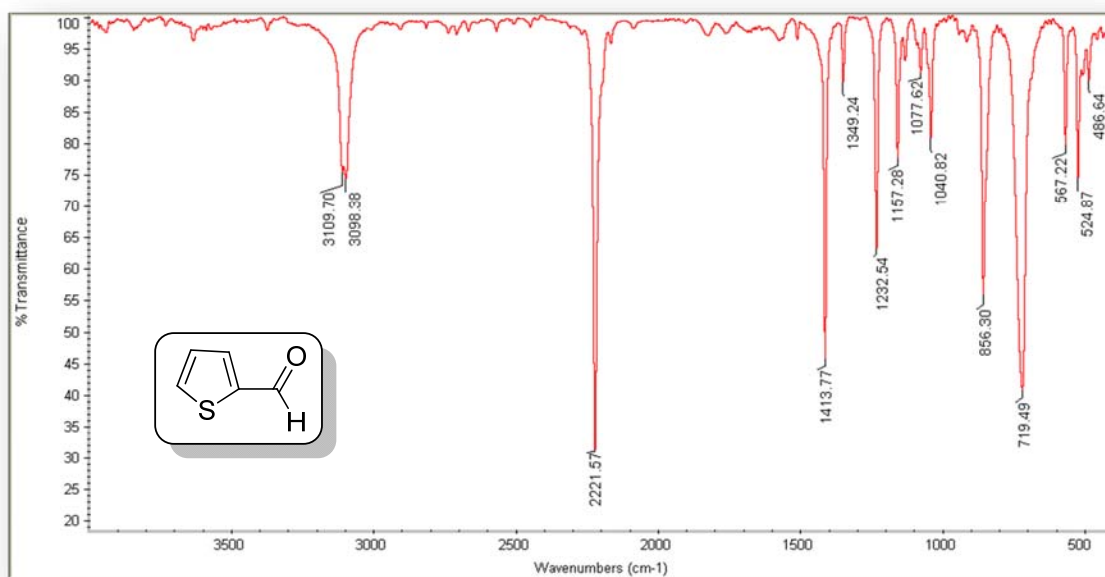


Fig 18b: FT-IR (KBr) spectrum of Thiophen-2-carbonitrile (Table 4, Entry 18).

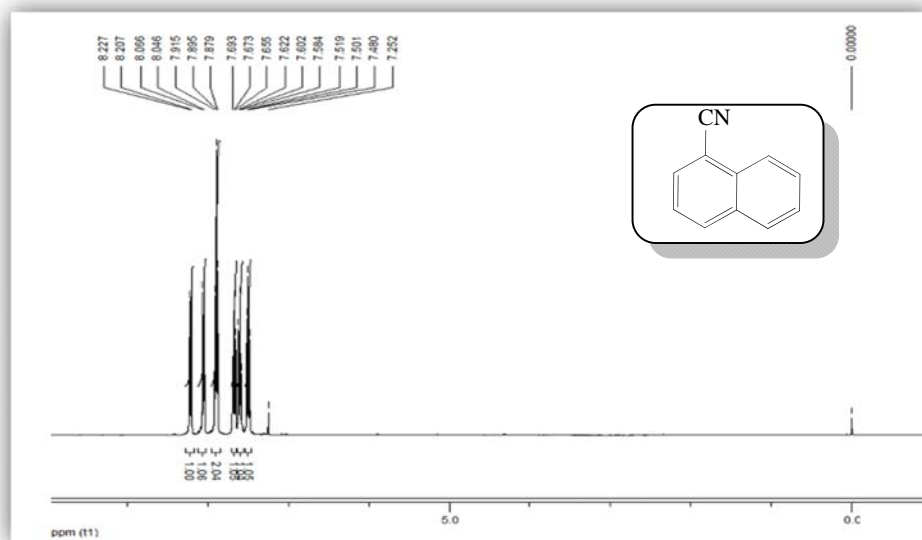


Fig 19a: ^1H NMR (400 MHz, CDCl_3) of 1-Naphthonitrile (Table 4, Entry 19).

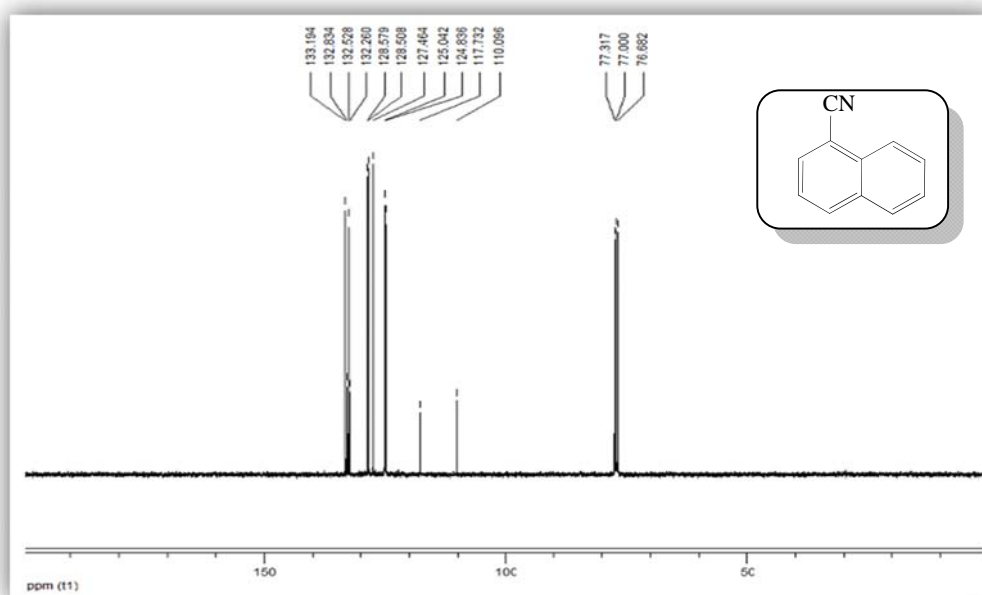


Fig 19b: ^{13}C NMR (100 MHz, CDCl_3) of 1-Naphthonitrile (Table 4, Entry 19).

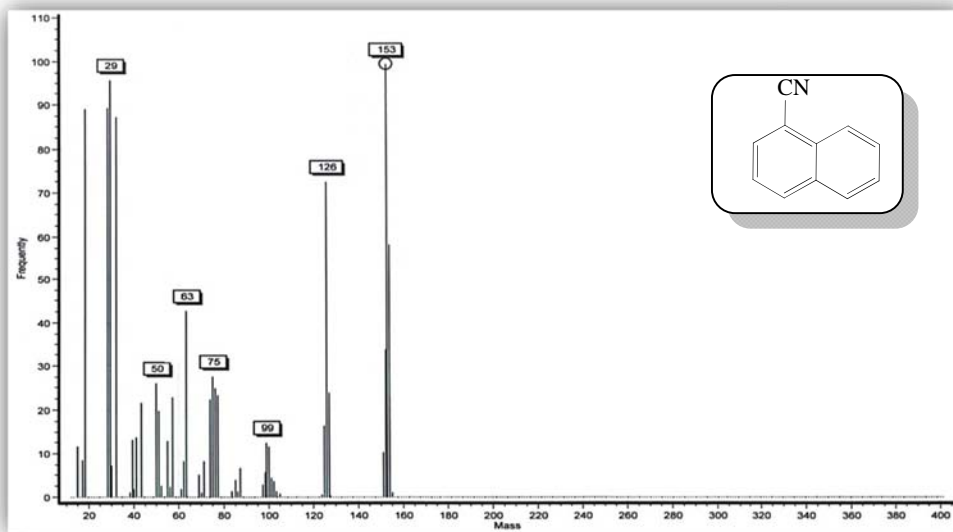


Fig 19c: Mass spectrum of 1-Naphthonitrile (Table 4, Entry 19).

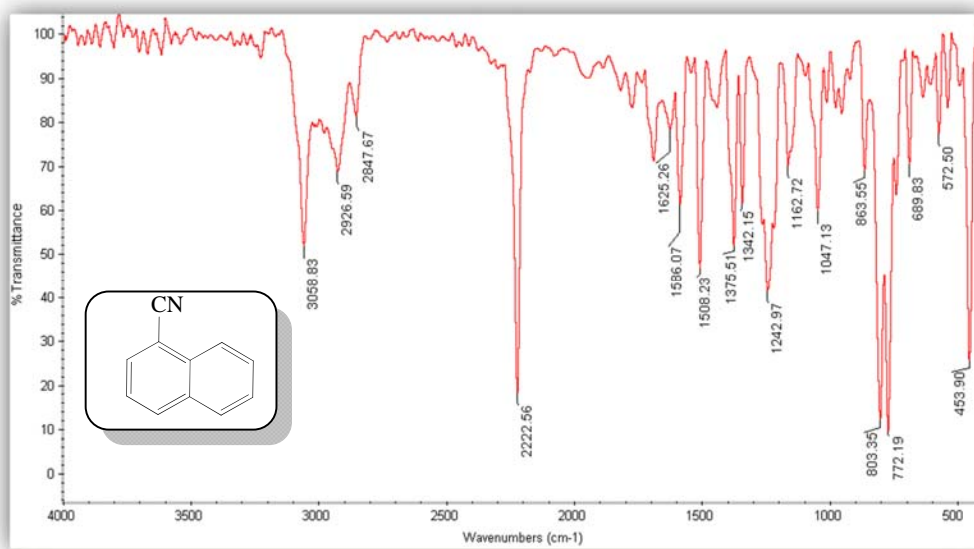


Fig 19d: FT-IR (neat) spectrum of 1-Naphthonitrile (Table 4, Entry 19).

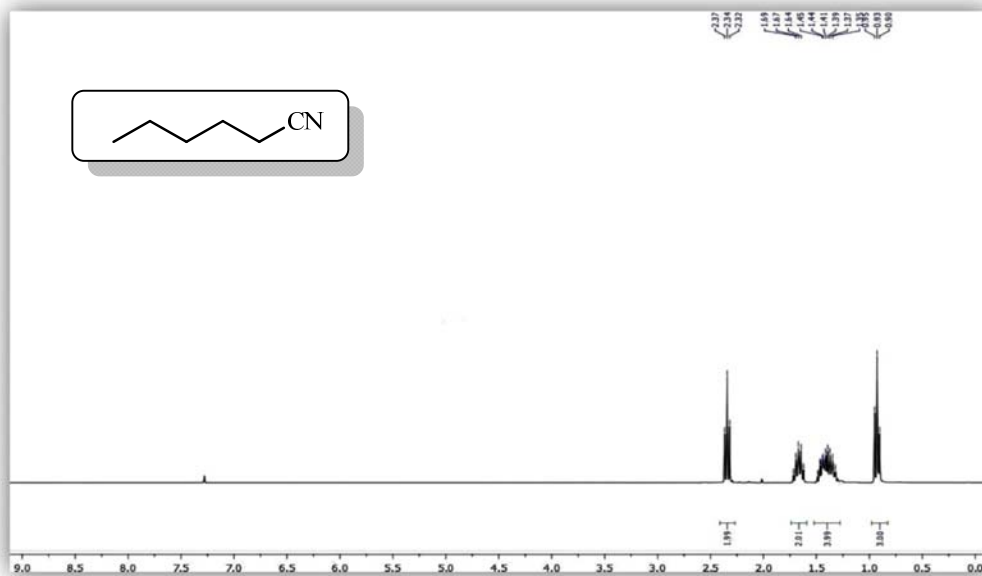


Fig 20a: ¹H NMR (300 MHz, CDCl₃) of Hexanenitrile (Table 4, Entry 20).

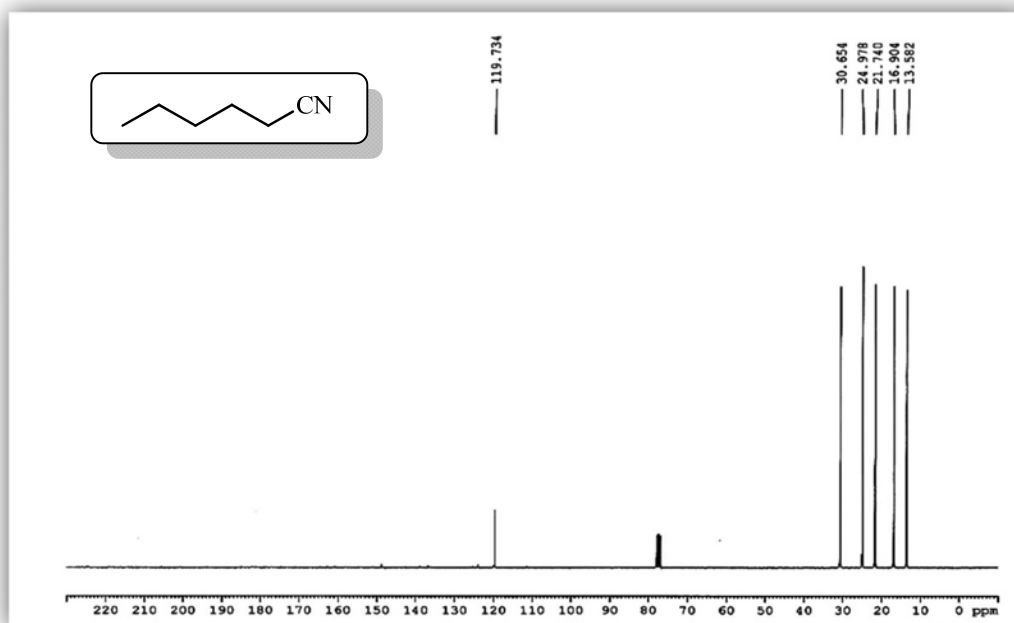


Fig 20b: ¹³C NMR (75 MHz, CDCl₃) of Hexanenitrile (Table 4, Entry 20).

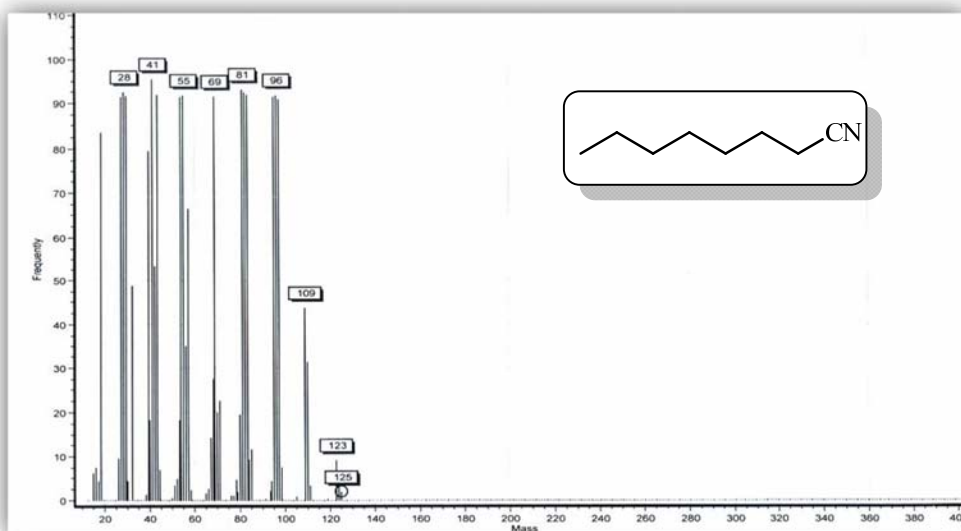


Fig 21c: Mass spectrum of Octanenitrile (Table 4, Entry 21).

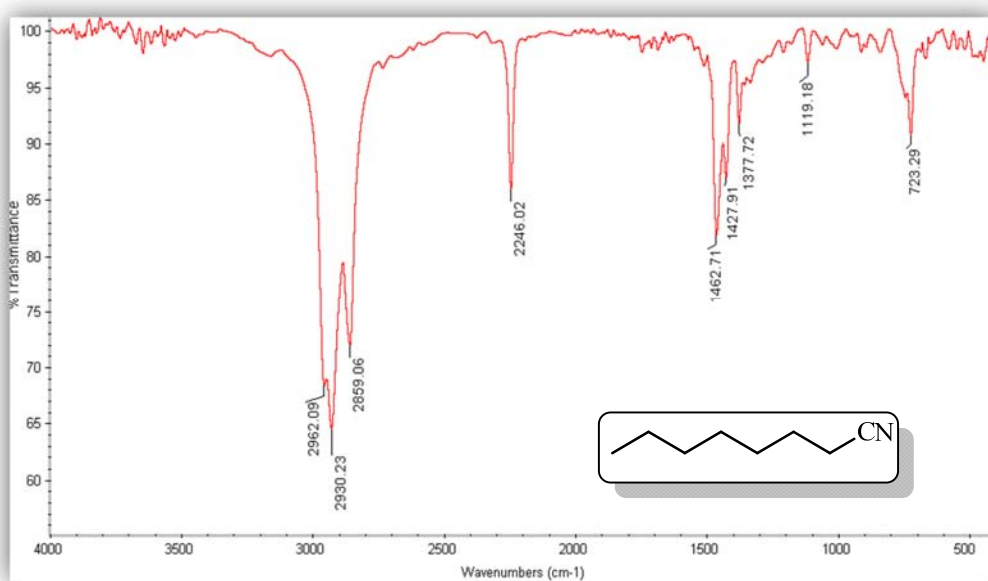


Fig 21d: FT-IR (neat) spectrum of Octanenitrile (Table 4, Entry 21).

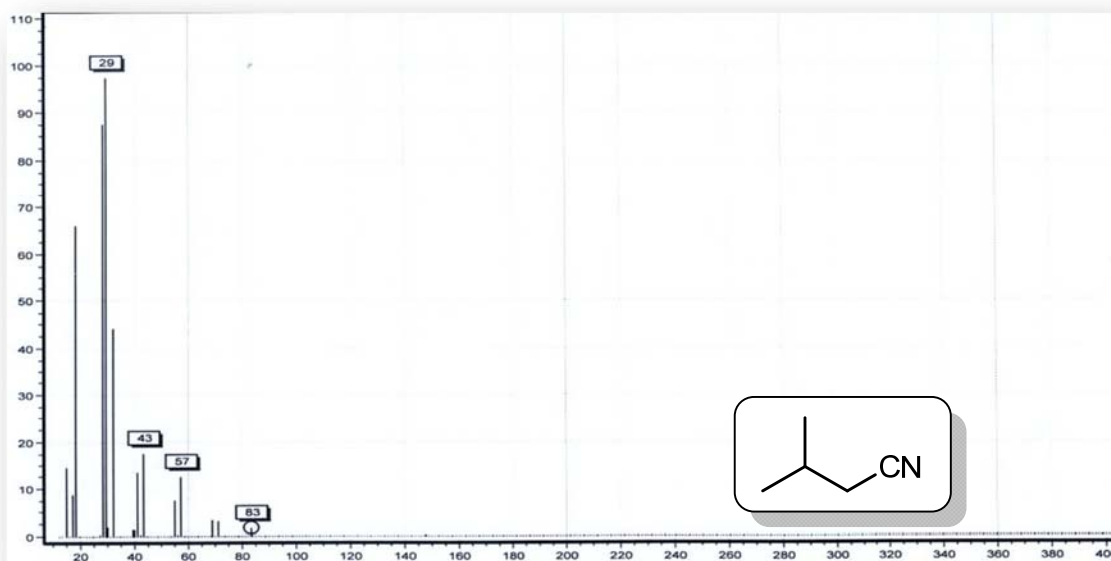


Fig 22c: Mass spectrum of 3-Methylbutanenitrile (Table 4, Entry 22).

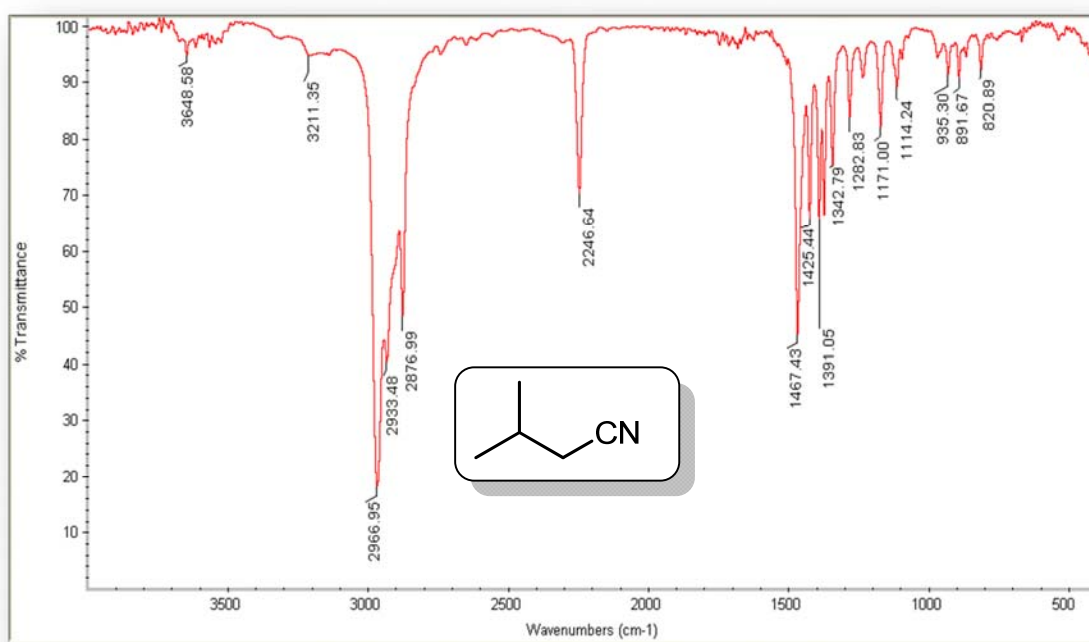


Fig 22d: FT-IR (neat) spectrum of 3-Methylbutanenitrile (Table 4, Entry 22).