Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supplementary Information

Highly efficient nitrobenzene and alkyl/aryl azide reduction in stainless steel jars without catalyst addition

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General Information

All chemicals were purchased from Sigma-Aldrich (Milan, Italy) and used without further purification. Reactions were monitored by TLC on Merck 60 F254 (0.25 mm) plates (Milan, Italy), which were visualized by UV inspection and/or by heating after a spraying with 0,5% ninhydrin in ethanol or phosphomolybdic acid. Mechanochemical reactions were carried out in a Planetary Ball Mill (PM100 Retsch GmbH, Haan, Germany) using either 50 mL grinding jars and milling balls (both made in stainless steel). NMR spectra (300 MHz and 75 MHz for ^{1}H and ^{13}C, respectively) were recorded on a Bruker 300 Avance instrument (Milan, Italy) at 25 °C. Chemical shifts were calibrated to the residual proton and carbon resonances of the solvent; DMSO-d_6 (δH = 2.54, δC = 39.5), $CDCl_3$ (δH = 7.26, δC = 77.16), D_2O (δH = 4.79). Chemical shifts (δ) are given in ppm, and coupling constants (J) in Hz. GC-MS analyses were performed in a GC Agilent 6890 (Agilent Technologies, Santa Clara, CA, USA) that was fitted with a mass detector Agilent Network 5973, using a 30 m capillary column, i.d. of 0.25 mm and film thickness 0.25 µm. GC conditions were: injection split 1:20, injector temperature 250 °C, detector temperature 280 °C. Gas carrier: helium (1.2 mL/min), temperature program: from 70 °C (2 min) to 300 °C at 5 °C/min. The cations were determined with a Perkin Elmer Optima 7000 (Perkin Elmer, Norwalk, Connecticut, USA) inductively coupled plasma-optical emission spectrometer (ICP-OES).

General procedure

General Procedure for the nitrobenzene reduction reaction

The milling jar (50 mL; stainless steel) was equipped with 1500 milling balls (Ø = 2 mm, stainless steel) and 48 medium balls (Ø = 5 mm, stainless steel). Nitrobenzene (0.5 mmol), ammonium formate (15 mmol), KOH (1 mmol), and basic Al_2O_3 (1 g) were added in the given order. Milling was performed at 650 rpm for 30 min, 1, 1.5 and 2 hours. After the milling jar was cooled to room temperature, the crude products were transferred and the solid washed with CH_2Cl_2 (3 × 10 mL) and water (3 × 10 mL). The desired product was extracted in organic phase, washed with H_2O three times and finally dried (Na_2SO_4). When impure, products were purified by flash chromatography on silica gel (hexane–EtOAc). Products were analysed using ^{1}H NMR and ^{13}C NMR spectroscopy, MS and GC-MS chromatography.General Procedure for the aryl azide reduction reaction

The milling jar (50 mL; stainless steel) was equipped with 1500 milling balls (Ø = 2 mm, stainless steel) and 48 medium balls (Ø = 5 mm, stainless steel). The aryl azides (0.5 mmol), sodium formate (10 mmol), KOH (1 mmol), and basic Al_2O_3 (1 g) were added in the given order. Milling was accomplished at 650 rpm for 1 and 1.5 hours. After the milling jar was cooled to room temperature, the crude products were transferred and the solid washed with CH_2Cl_2 (3 × 10 mL) and water (3 × 10 mL). The desired product was extracted in organic phase, washed with H_2O three times and finally dried (Na_2SO_4). When impure, products were purified by flash chromatography on silica gel (hexane–EtOAc). Products were analysed using ^{1}H NMR and ^{13}C NMR spectroscopy, MS and GC-MS chromatography..

General Procedure for the alkyl azide reduction reaction

The milling jar (50 mL; stainless steel) was equipped with 1500 milling balls (Ø = 2 mm, stainless steel) and 48 medium balls (Ø = 5 mm, stainless steel). The alkyl azides (0.5 mmol), hydrazine (15 mmol), KOH (1 mmol) and basic Al_2O_3 (1 g) were added in the given order. Milling was

accomplished at 650 rpm for 1 hour. After the milling jar was cooled to room temperature, the crude products were transferred and the solid washed with CH_2Cl_2 (3 × 10 mL) and water (3 × 10 mL). The desired product was extracted in organic phase, washed with H_2O three times and finally dried (Na_2SO_4). When impure, products were purified by flash chromatography on silica gel (hexane–EtOAc). Products were analysed using ^{1}H NMR and ^{13}C NMR spectroscopy, MS and GC-MS chromatography.

Characterization of the products

Nitro aryl reduction

Aniline (Table 4, Entry 1), (Table 6, Entry 1)[1]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.26 (2H, t, J = 6 Hz), 6.86 (1H, t, J = 9 Hz), 6.76 (2H, t, J = 6 Hz), 3.63 (2H, s) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 146.57, 129.30, 118.40, 115.12 ppm.

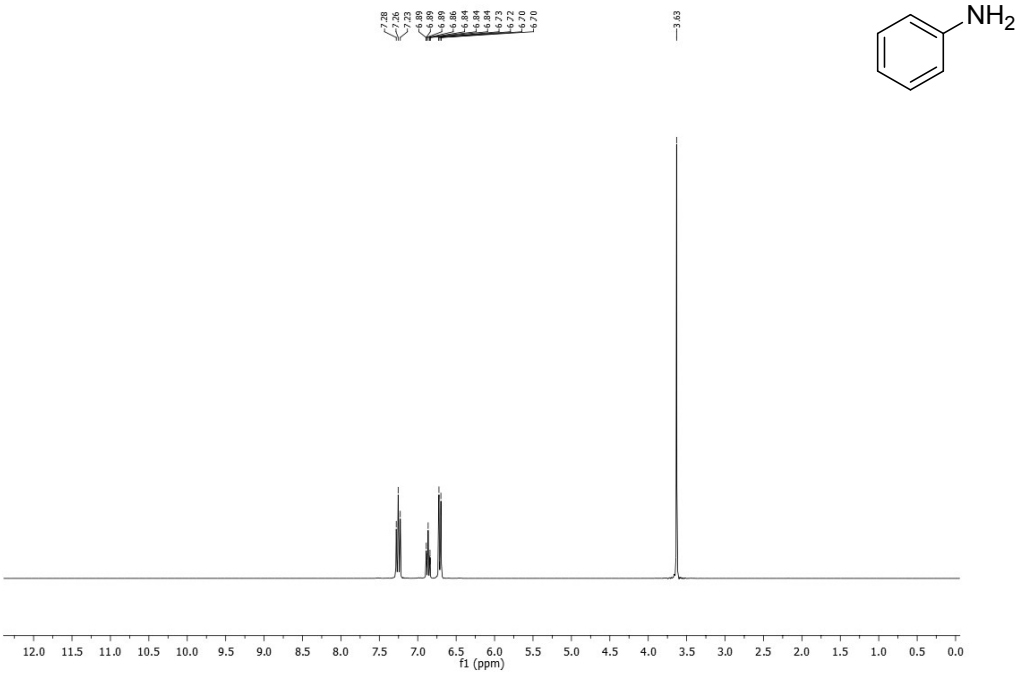


Figure 1. ^{1}H NMR (300 MHz, $CDCl_3$) of Aniline (Table 4, Entry 1), (Table 7, Entry 1).

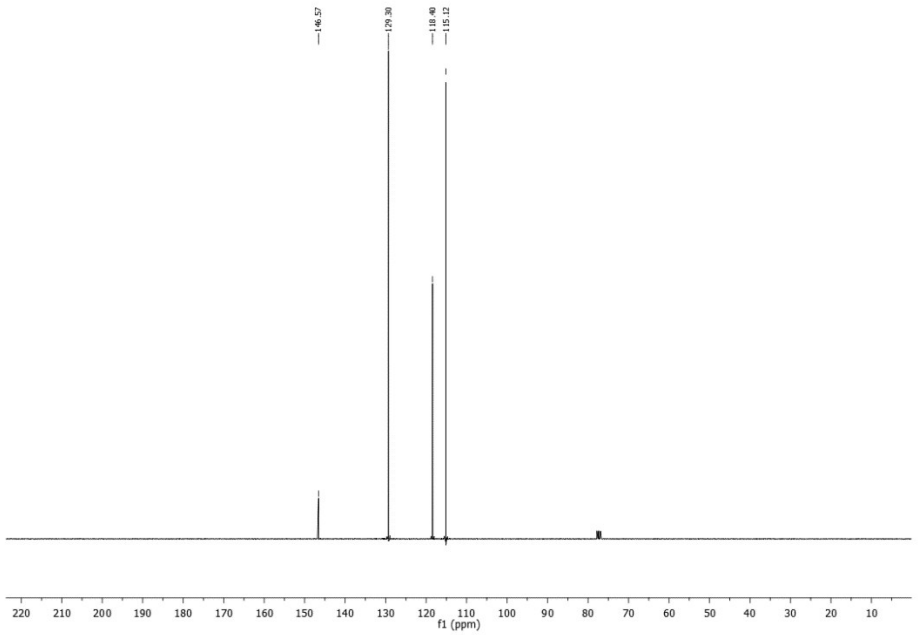


Figure 2. ^{13}C-NMR (75 MHz, $CDCl_3$) of Aniline (Table 4, Entry 1), (Table 7, Entry 1).

***p*-Bromoaniline (Table 4, Entry 2)[2]:** ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.17 (2H, d, J = 9 Hz), 6.49 (2H, d, J = 9 Hz) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.70, 132.27, 117.00, 110.44 ppm.

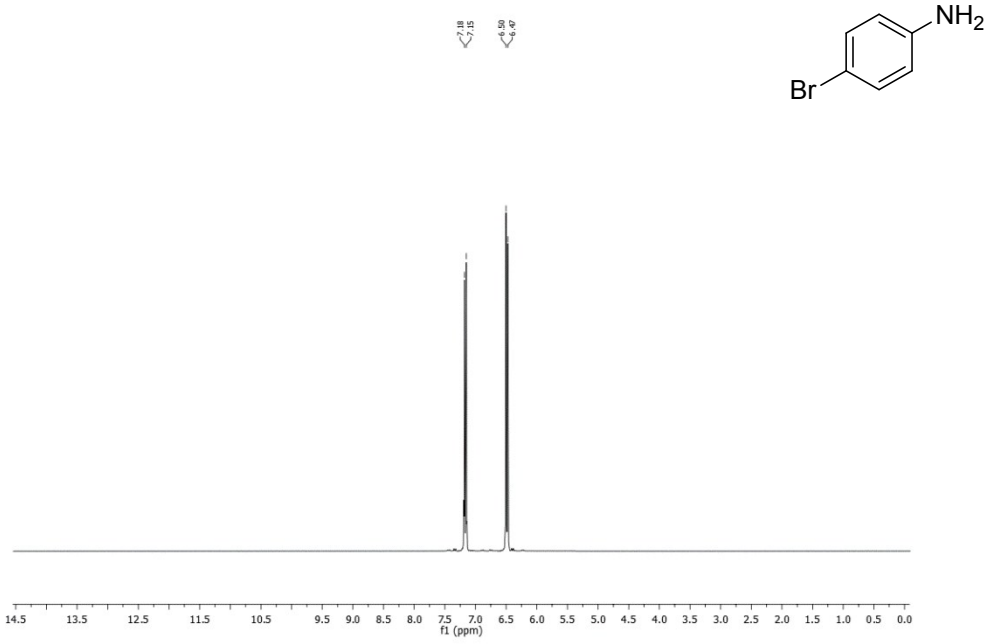


Figure 3. ^{1}H NMR (300 MHz, $CDCl_3$) of *p*-Bromoaniline (Table 4, Entry 2).

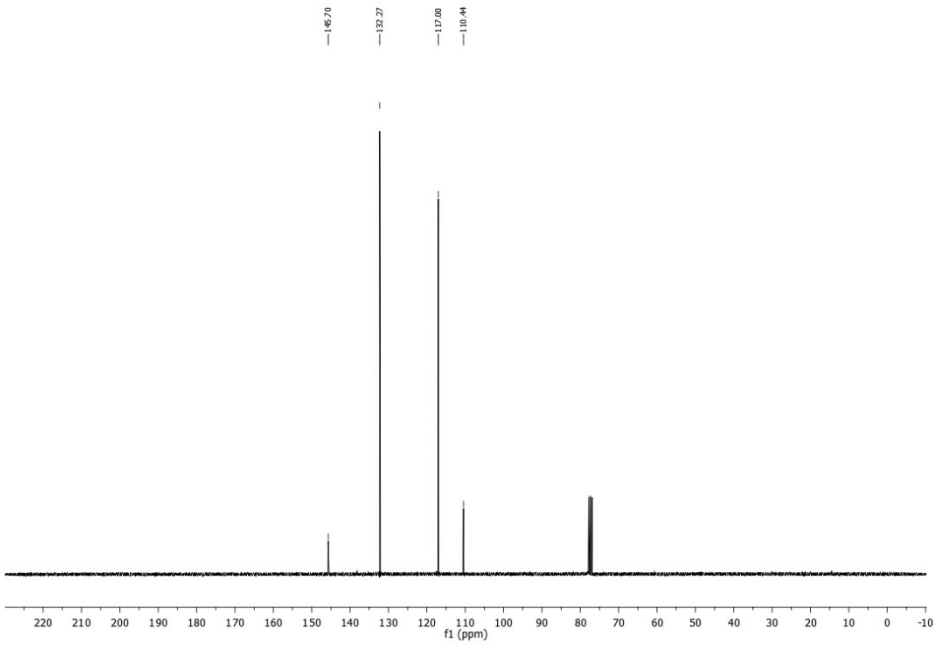


Figure 4.^{13}C-NMR (75 MHz, $CDCl_3$) of *p*-Bromoaniline (Table 4, Entry 2).

***p*-Iodooaniline (Table 4, Entry 3)**[2]**:** ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40 (2H, d, J = 9 Hz), 6.47(2H, d, J = 9 Hz) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 146.35, 138.20, 117.60, 79.68. ppm.

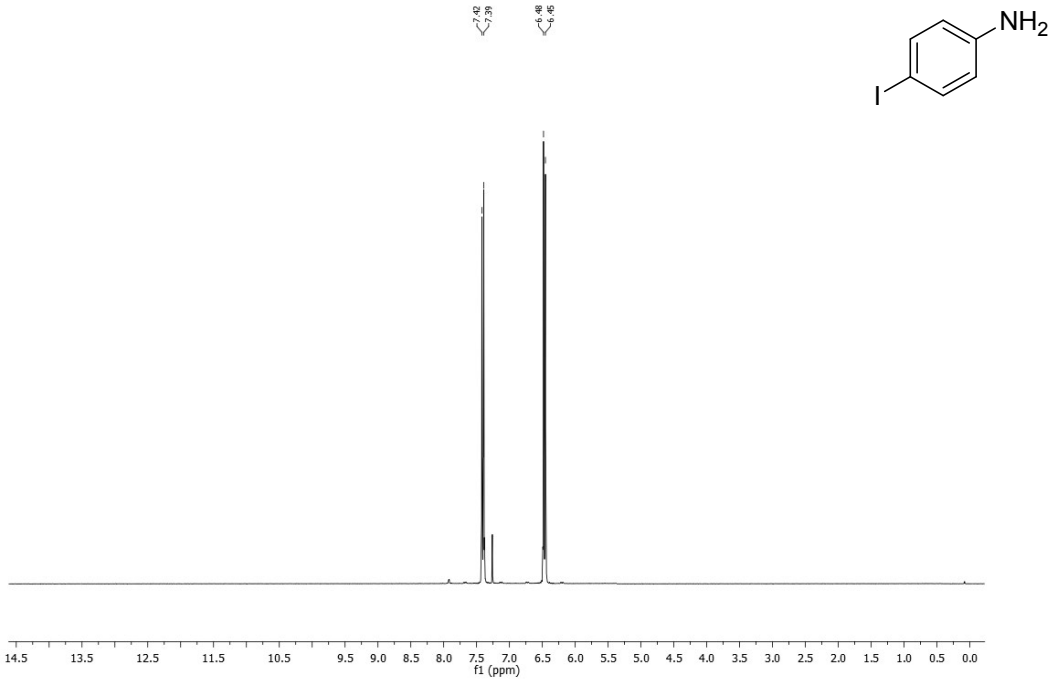


Figure 5. ^{1}H NMR (300 MHz, $CDCl_3$) of p-Iodoaniline (Table 4, Entry 3).

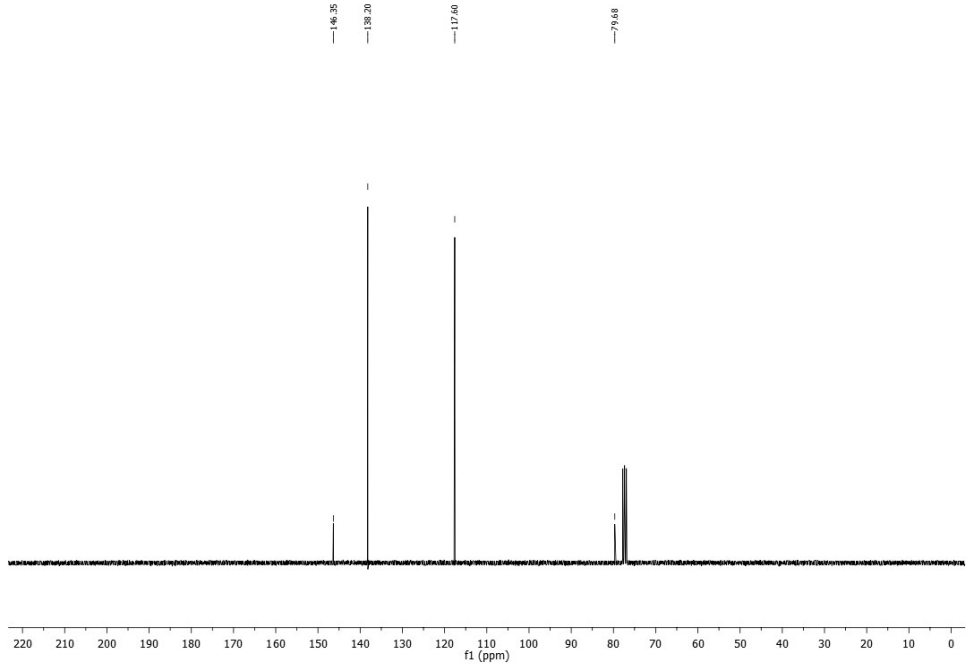


Figure 6. ^{13}C-NMR (75 MHz, $CDCl_3$) of p-Iodoaniline (Table 4, Entry 3).

***p*-Chloroaniline (Table 4, Entry 4)**[1]**:** ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.10 (2H, d, J = 9 Hz), 6.61 (2H, d, J = 9 Hz) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.17, 129.42, 123.51, 116.59 ppm.

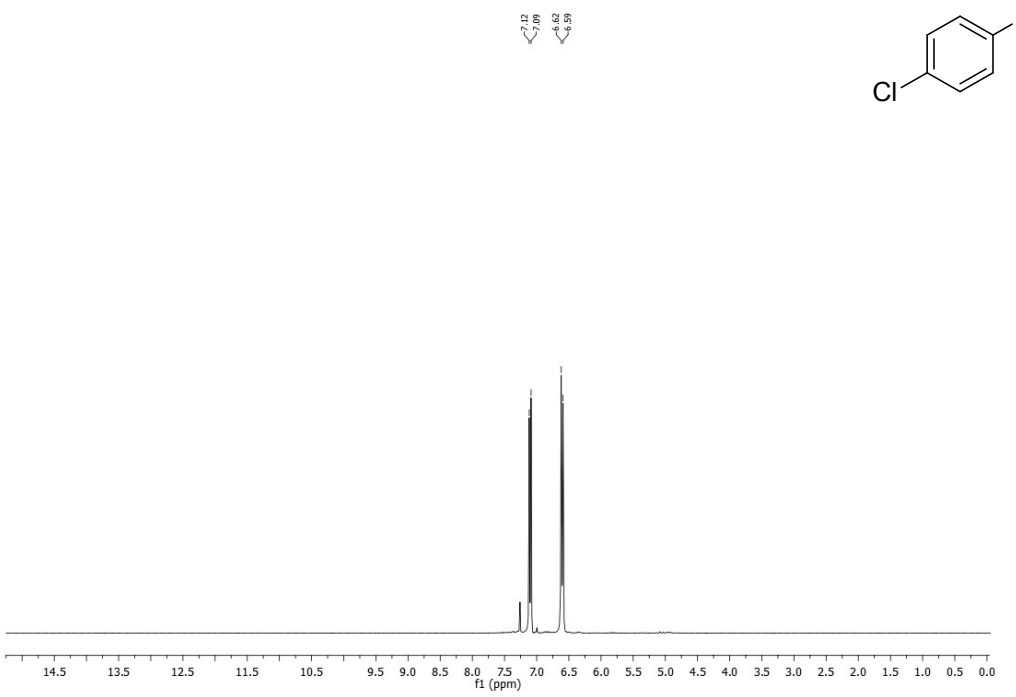


Figure 7. ^{1}H NMR (300 MHz, $CDCl_3$) of p-Chloroaniline (Table 4, Entry 4).

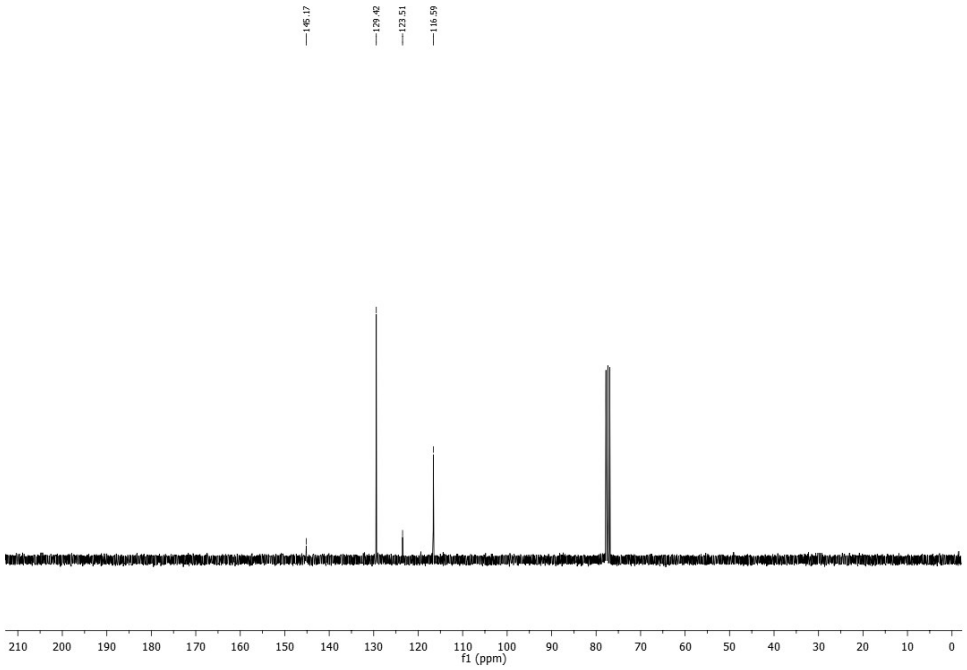


Figure 8. ^{13}C-NMR (75 MHz, $CDCl_3$) of p-Chloroaniline (Table 4, Entry 4).

***p*-Aminophenol (Table 4, Entry 5)[3]:** ^{1}H NMR (300 MHz, DMSO-d_6) δ 6.54- 6.45 (4H, m) ppm; ^{13}C NMR (75 MHz, DMSO-d_6) δ 149.47, 140.89, 116.48 ppm.

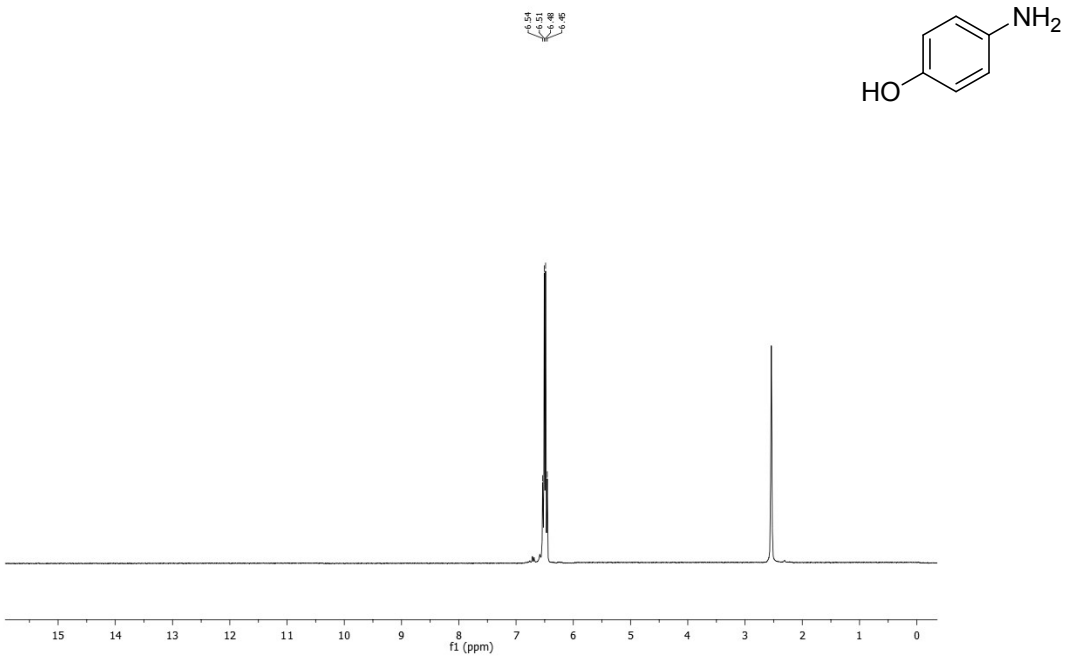


Figure 9. ^{1}H NMR (300 MHz, DMSO-d_6) of p-Aminophenol (Table 4, Entry 5).

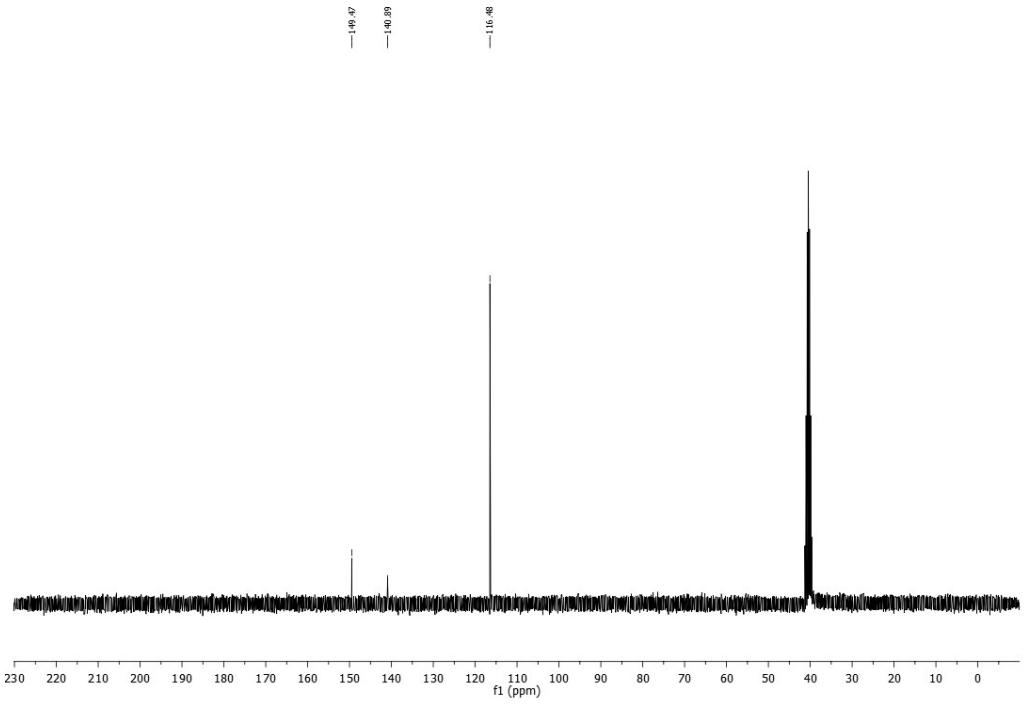


Figure 10. ^{13}C-NMR (75 MHz, DMSO-d_6) of p-Aminophenol (Table 4, Entry 5).

Benzene-1,4-diamine (Table 4, Entry 6)[3]**:** ^{1}H NMR (300 MHz, $CDCl_3$) δ (4H, s) 6.56 ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 138.98, 117.11 ppm.

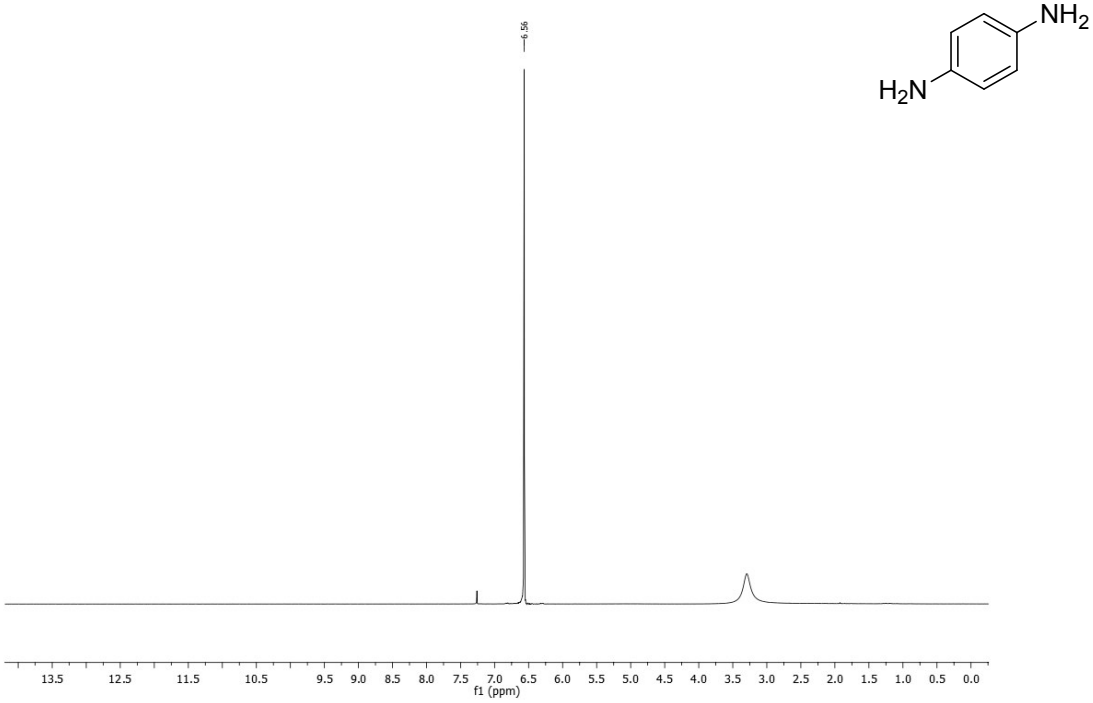


Figure 11. ^{1}H NMR (300 MHz, $CDCl_3$) of benzene-1,4-diamine (Table 4, Entry 6).

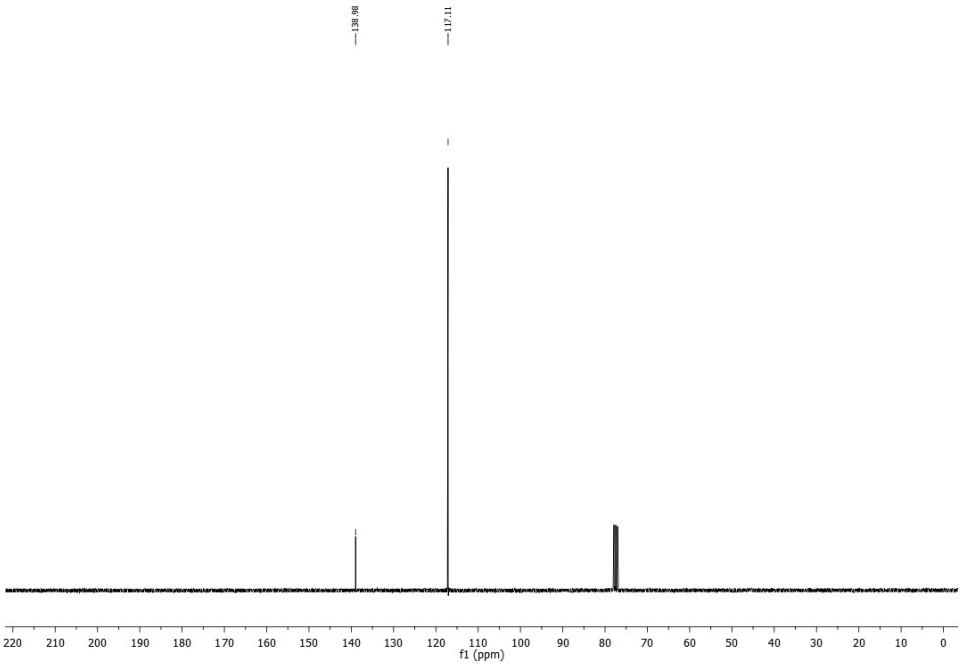
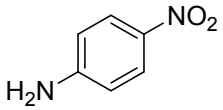


Figure 12. ^{13}C-NMR (75 MHz, $CDCl_3$) of benzene-1,4-diamine (Table 4, Entry 6).

***p*-Nitroaniline (Table 4, Entry 7)[4]:** ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.98 (2H, d, J = 9 Hz), 6.64 (2H, d, J = 9 Hz) ppm; ^{13}C NMR (75 MHz, DMSO-d_6) δ 156.67, 136.63, 127.37, 113.35 ppm.



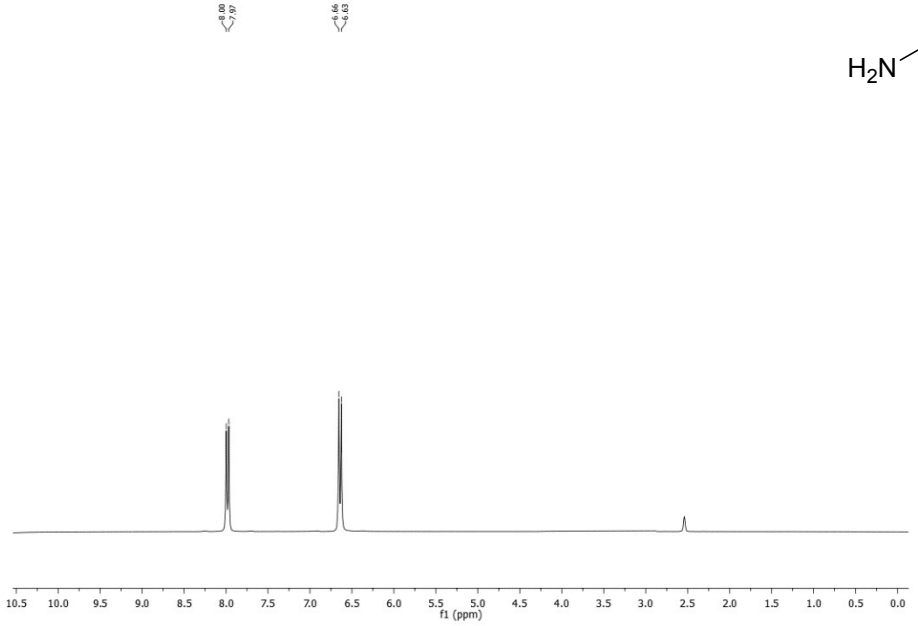


Figure 13. ^{1}H NMR (300 MHz, DMSO-d_6) of *p*-Nitroaniline (Table 4, Entry 7).

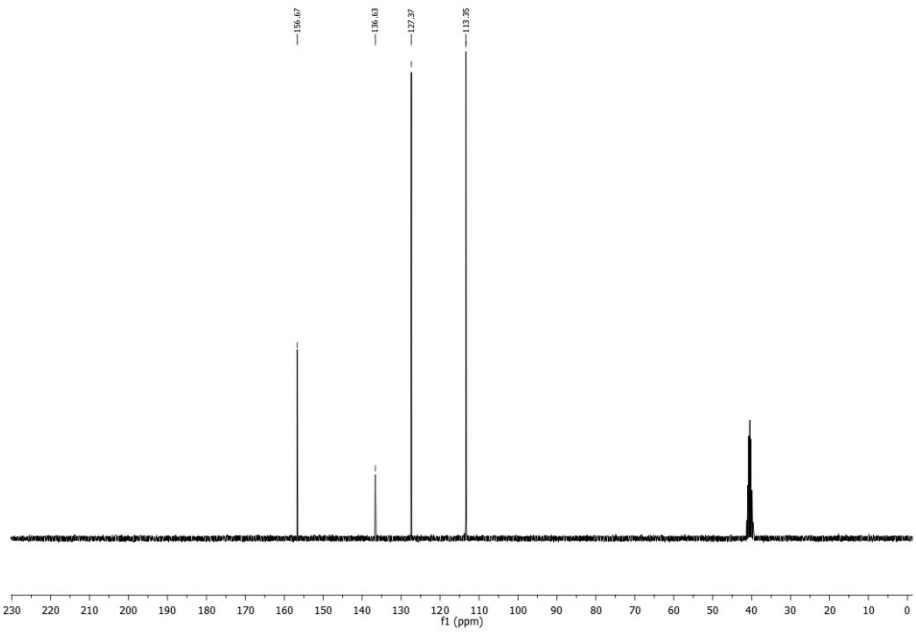
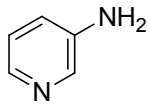


Figure 14. ^{13}C-NMR (75 MHz, DMSO-d_6) of *p*-Nitroaniline (Table 4, Entry 7).

3-Aminopiridine (Table 4, Entry 8)[5]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.10- 8.01 (2H, m), 7.10- 7.06 (1H,m), 6.99-6.96 (1H,m) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.88, 140.14, 137.60, 124.13, 121.94 ppm.



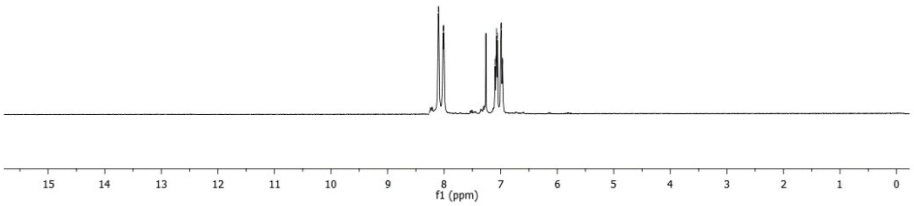


Figure 15. ^{1}H NMR (300 MHz, $CDCl_3$) of 3-Aminopiridine (Table 4, Entry 8).

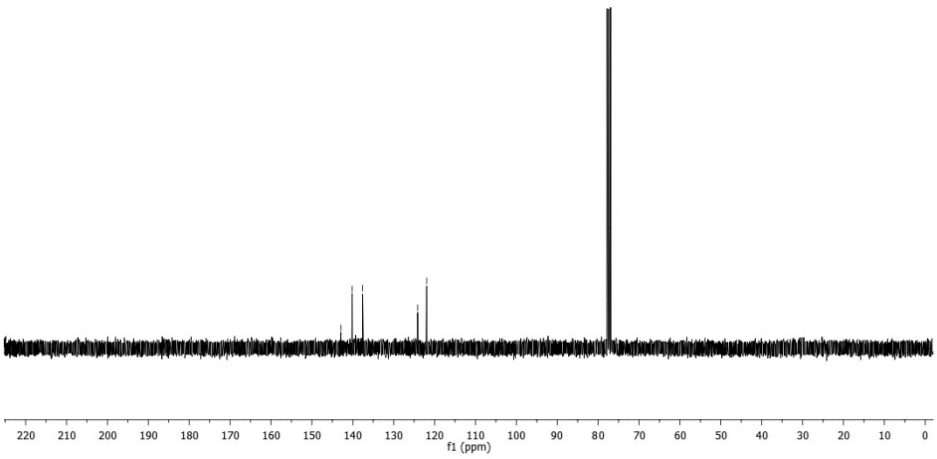


Figure 16.^{13}C-NMR (75 MHz, $CDCl_3$) of 3-Aminopiridine (Table 4, Entry 8).

***p*-Aminoacetophenone (Table 4, Entry 9)[6]:** ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.80 (2H, d, J = 9 Hz), 6.64(2H, d, J = 9 Hz), 2.50 (3H, s) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 196.85, 151.38, 131.13, 129.76, 114.07, 26.44 ppm.

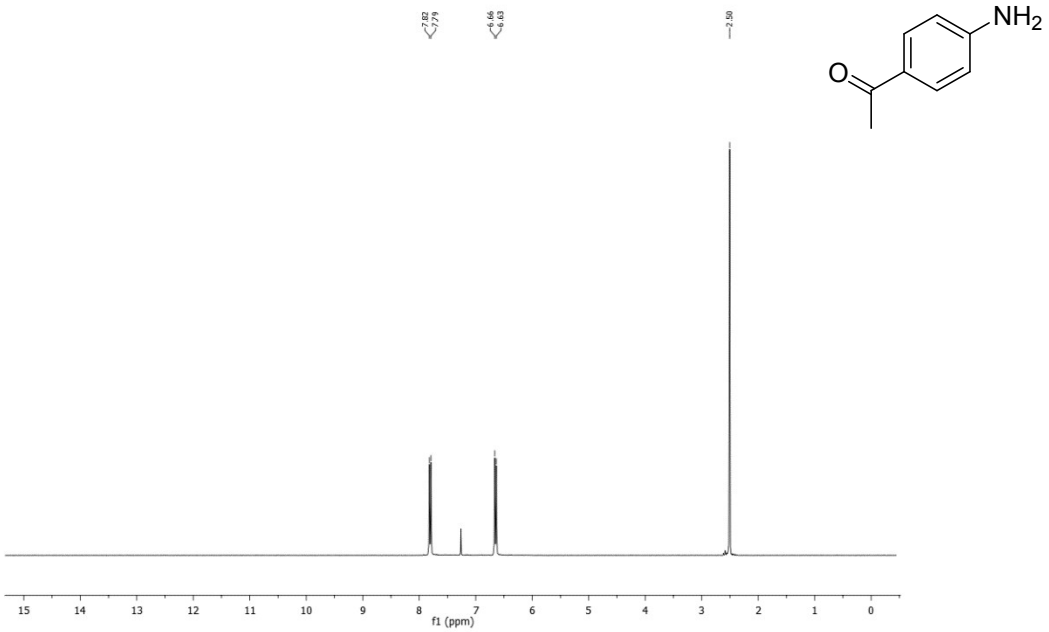


Figure 17.^{1}H NMR (300 MHz, $CDCl_3$) of *p*-Aminoacetophenone (Table 4, Entry 9).

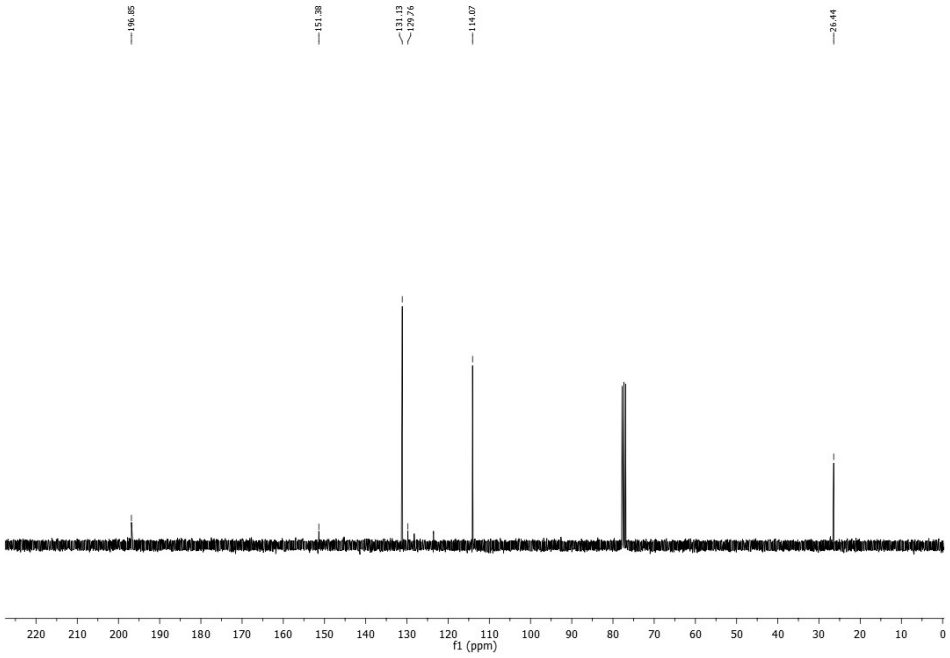
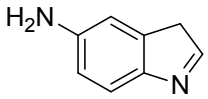


Figure 18.^{13}C-NMR (75 MHz, $CDCl_3$) of *p*-Aminoacetophenone (Table 4, Entry 9).

5-Aminoindole (Table 4, Entry 10): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.20 (1H, d, J = 6 Hz) 7.12 (1H, t, J =3 Hz), 6.96 (1H, m), 6.68 (1H, dd, J = 9 Hz), 6.38 (1H, m) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.87, 131.08, 129.16, 125.15, 113.36, 111.94, 105.96, 101.93 ppm.



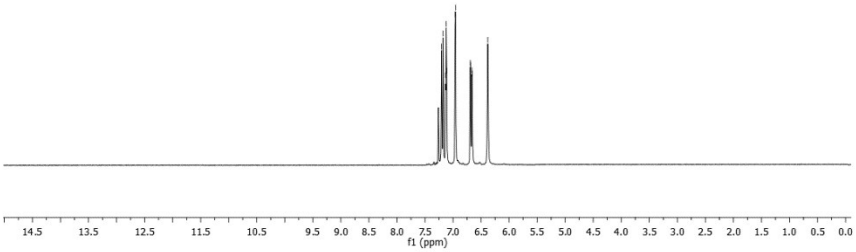


Figure 19. ^{1}H NMR (300 MHz, $CDCl_3$) of 5-Aminoindole (Table 4, Entry 11).

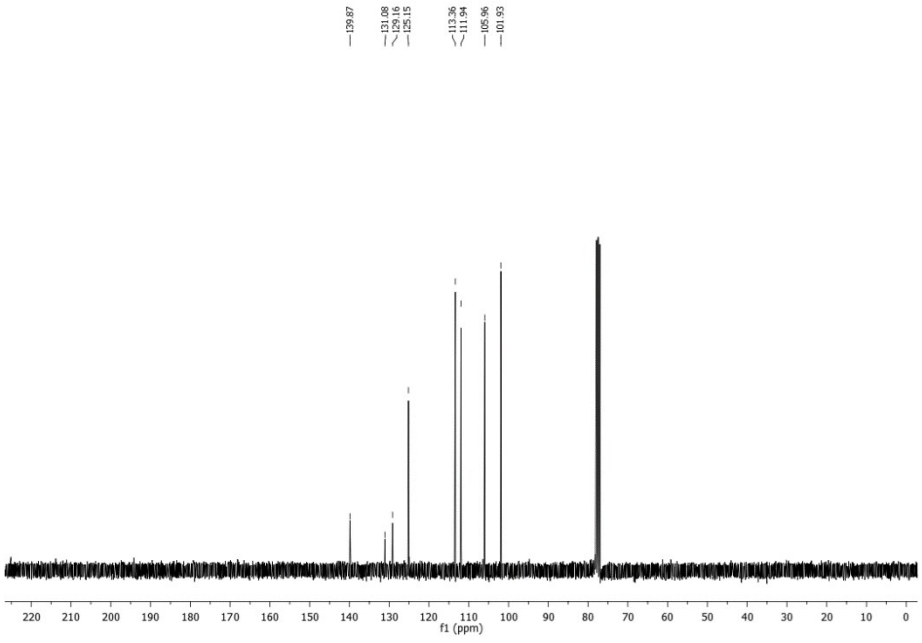
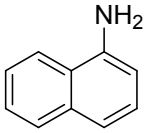


Figure 20. ^{13}C-NMR (75 MHz, $CDCl_3$) of 5-Aminoidole (Table 4, Entry 11).

1-Naphthylamine (Table 4, Entry 11), (Table 6, Entry 4) [7]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.66, 7.77, 7.73, 7.70, 7.39, 7.38, 7.37, 7.23, 7.21, 6.66, 6.62 ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.43, 134.68, 128.95, 126.71, 126.14, 125.26, 121.92, 121.05, 119.67, 111.06 ppm.



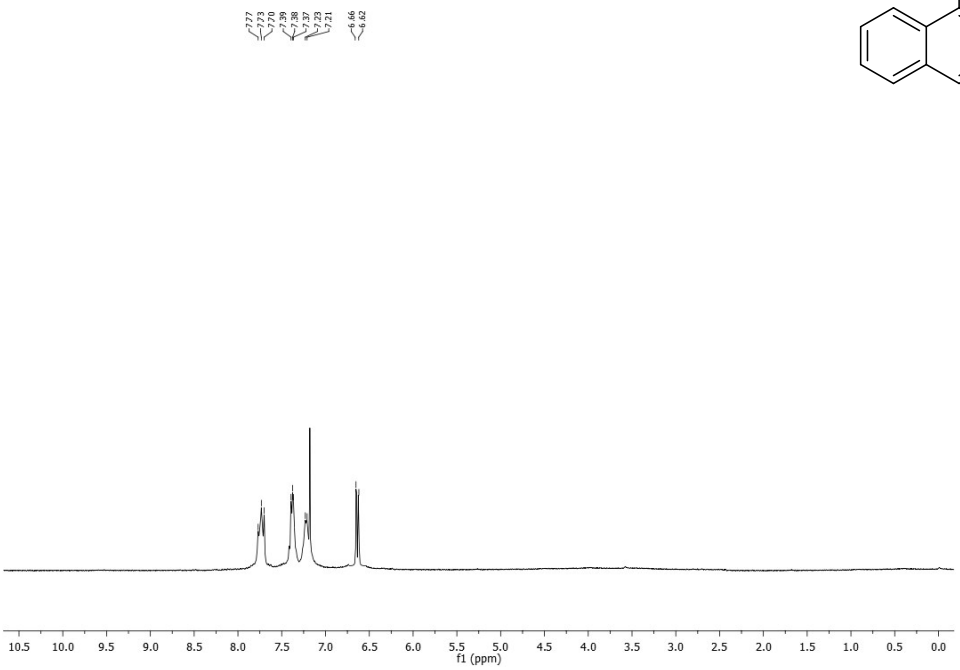


Figure 21. ^{1}H NMR (300 MHz, $CDCl_3$) of 1-Naphthylamine (Table 4, Entry 12), (Table 6, Entry 4).

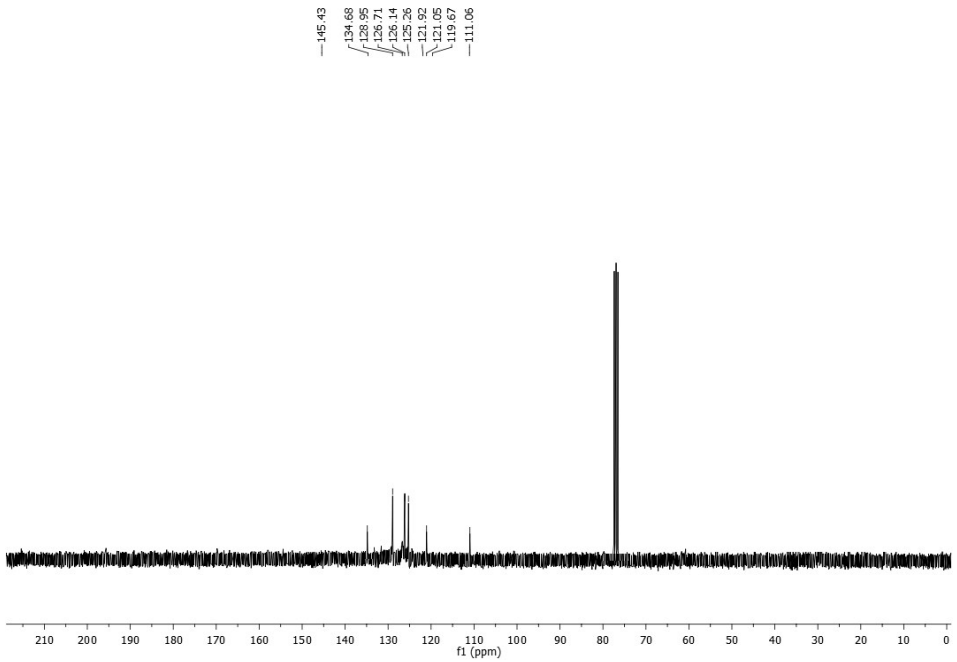


Figure 22. ^{13}C-NMR (75 MHz, $CDCl_3$) of 1-Naphthylamine (Table 4, Entry 12), (Table 6, Entry 4).

Aryl azides reduction reactions:

***p*-Methoxyaniline (Table 6, Entry 2)[7]:** ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.74 (2H, d, J = 9 Hz), 6.64 (2H, d, J = 9 Hz), 3.74 (3H, s) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.10, 140.23, 116.72, 115.10, 56.04 ppm.

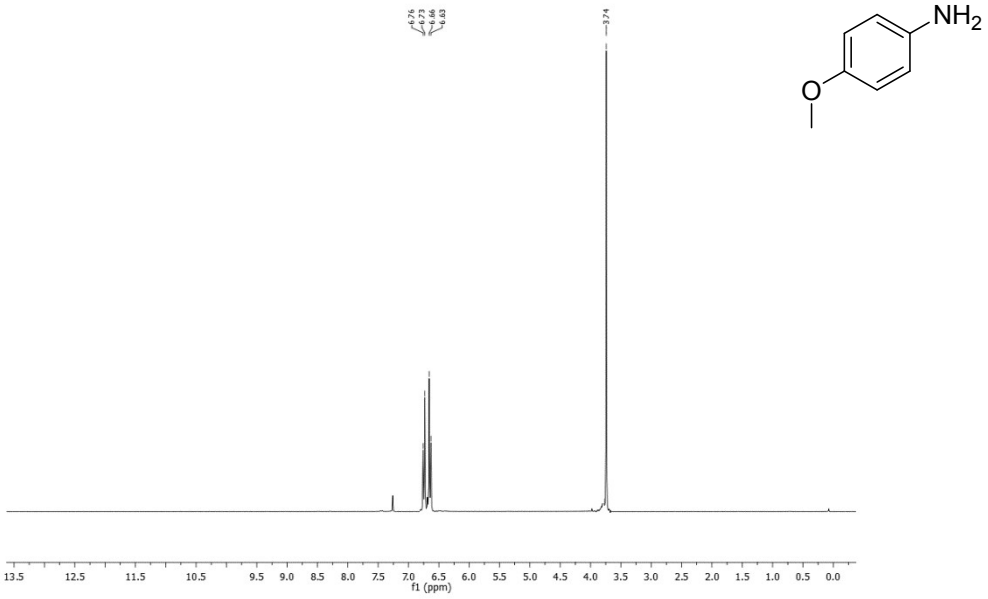


Figure 23. ^{1}H NMR (300 MHz, $CDCl_3$) of *p*-Methoxyaniline (Table 6, Entry 2).

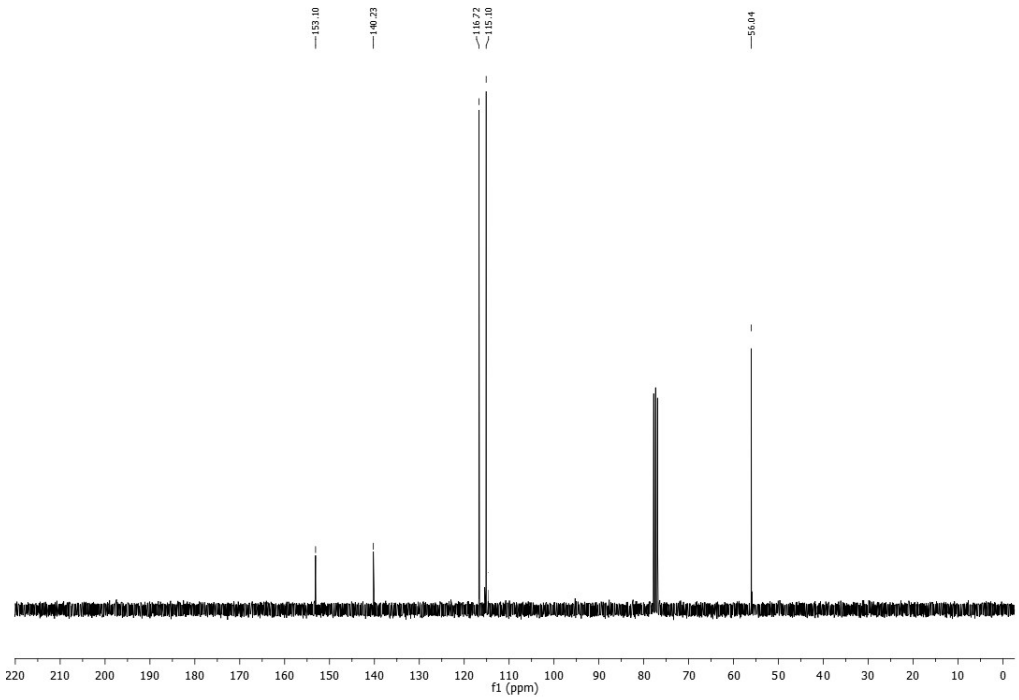


Figure 24. ^{13}C-NMR (75 MHz, $CDCl_3$) of *p*-Methoxyaniline (Table 6, Entry 2).

***o*-Toluidine (Table 6, Entry 3)**[16]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.07-7.02 (2H, m), 6.77- 6.67 (2H, m), 2.18 (3H, s) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 144.85, 130.76, 127.28, 122.64, 118.94, 115.23, 17.68 ppm.

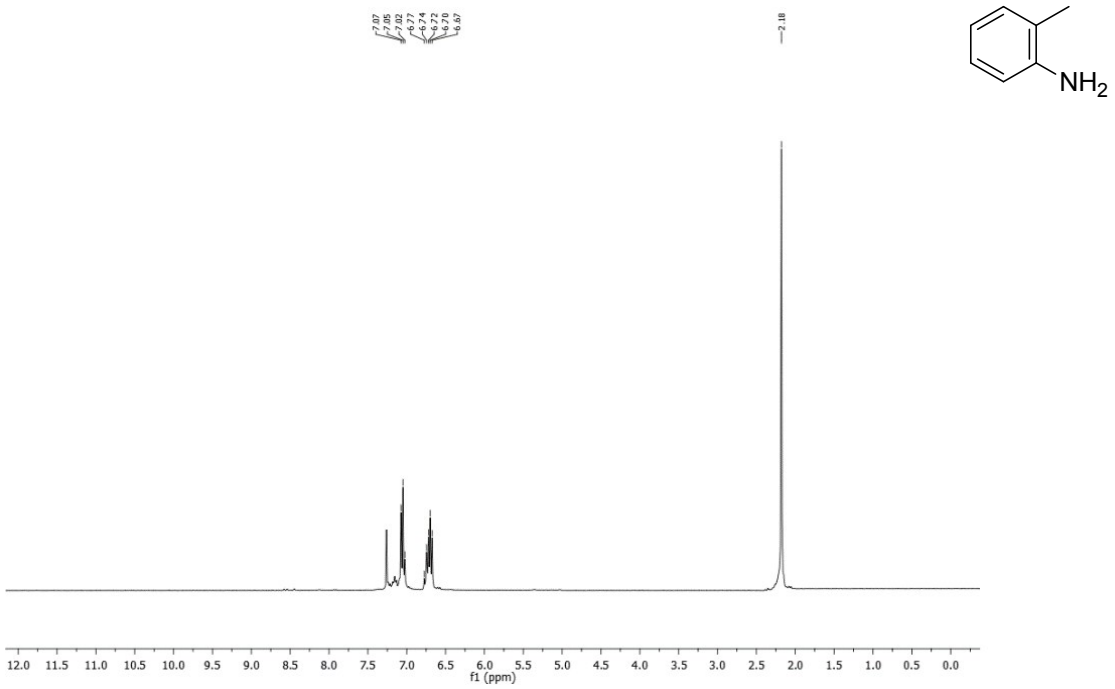


Figure 25. ^{1}H NMR (300 MHz, $CDCl_3$) of *o*-Toluidine (Table 6, Entry 3).

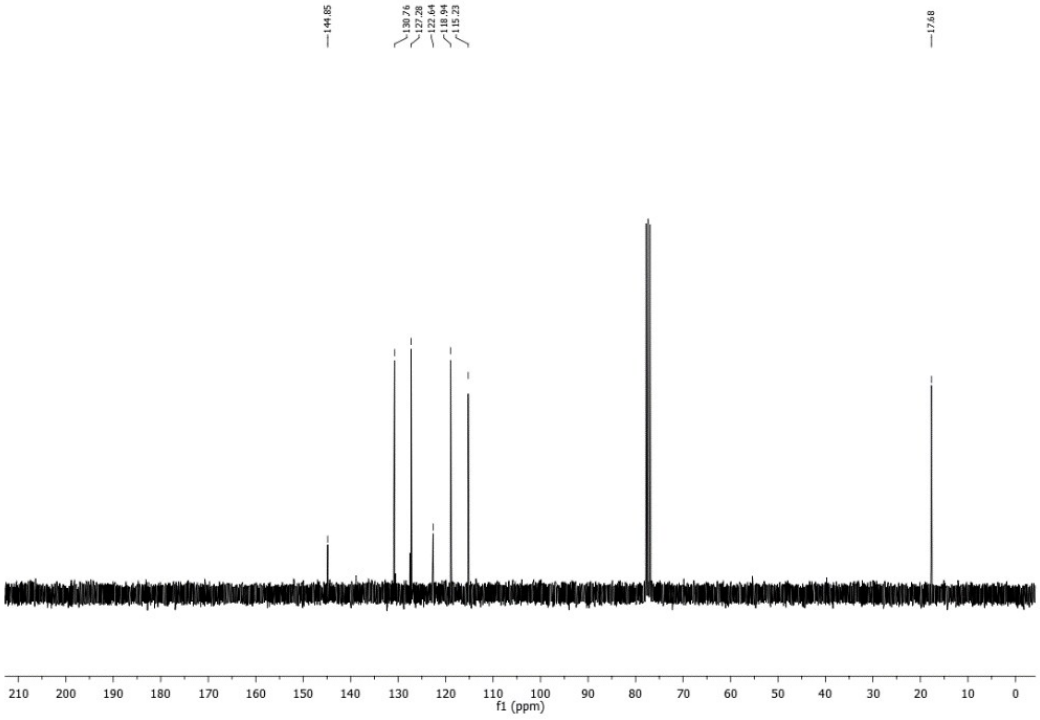
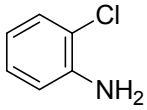


Figure 26. ^{13}C-NMR (75 MHz, $CDCl_3$) of *o*-Toluidine (Table 6, Entry 3).

2-Chloroaniline (Table 6, Entry 5)[16]**:** ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.25-22 (1H, m), 7.06 (1H, t, J = 6 Hz), 6.76 (1H, d, J = 6 Hz), 6.68 (1H, t, J = 6 Hz) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.11, 129.64, 127.85, 119.25, 116.09 ppm.



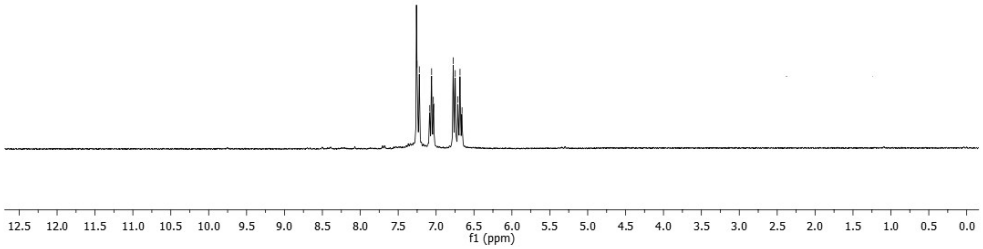


Figure 27. ^{1}H NMR (300 MHz, $CDCl_3$) of 2-Chloroaniline (Table 6, Entry 5).

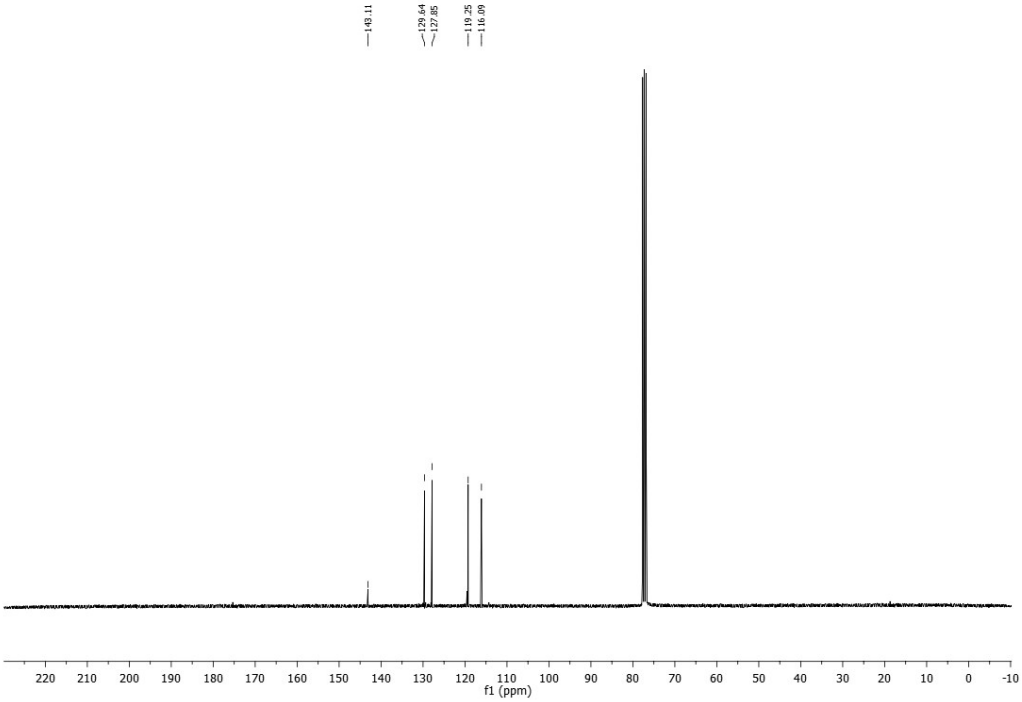


Figure 28. ^{13}C-NMR (75 MHz, $CDCl_3$) of 2-Chloroaniline (Table 6, Entry 5).

Alkyl azides reduction reactions:

Benzylamine (Table 7, Entry 1)[8]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.24-7.10 (5H, m), 3.70 (2H, s) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.16, 128.29, 126.87, 126.53, 46.23.

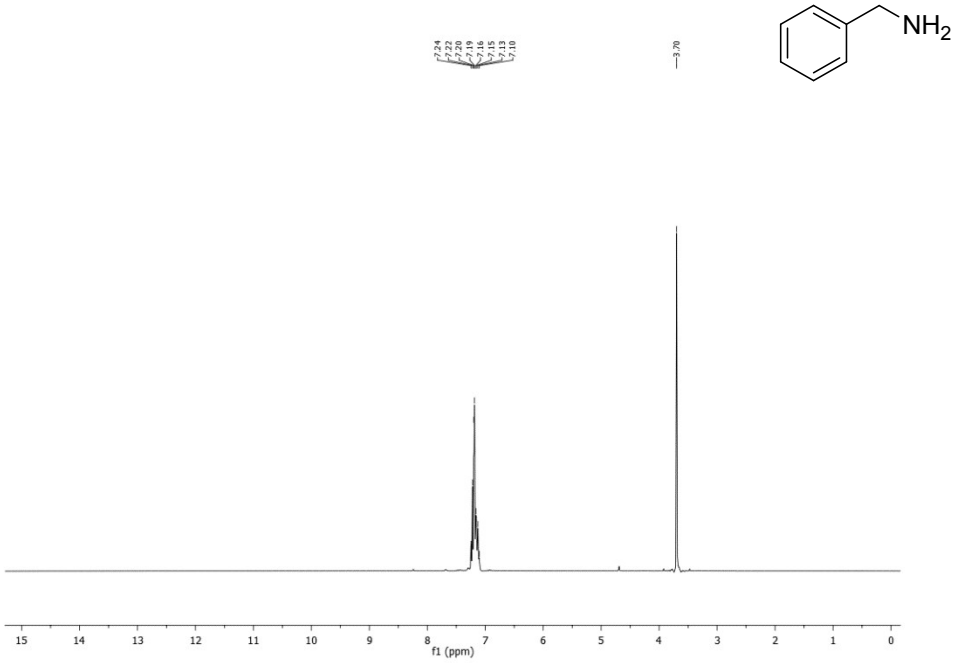


Figure 29. ^{1}H NMR (300 MHz, $CDCl_3$) of Benzylamine (Table 7, Entry 1).

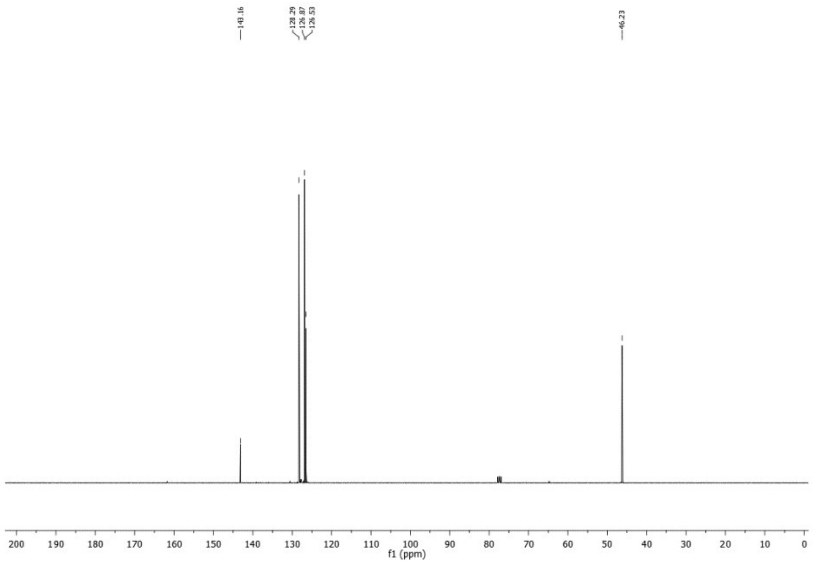


Figure30. ^{13}C-NMR (75 MHz, $CDCl_3$) of Benzylamine (Table 7, Entry 1).

1-Phenylethylamine (Table 7, Entry 2)[9]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37-7.24 (5H, m), 4.13 (1H, q, J = 6 Hz), 1.40 (3H, d, J = 9 HZ) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.66, 128.85, 127.24, 126.07, 51.69, 25.79 ppm.

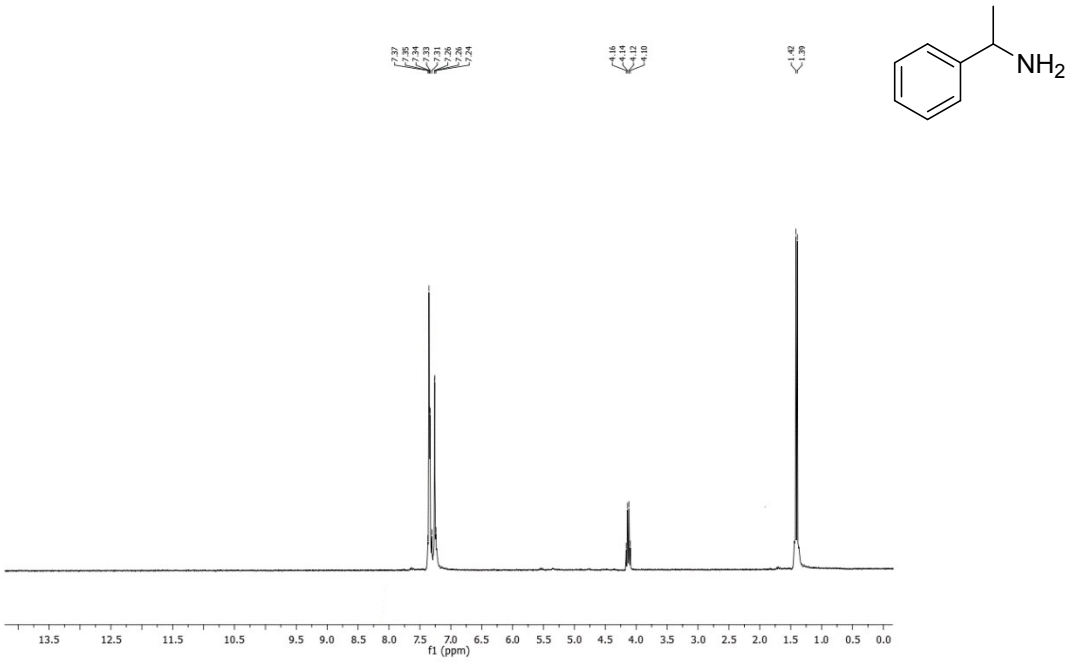


Figure 31. ^{1}H NMR (300 MHz, $CDCl_3$) of 1-Phenylethylamine (Table 7, Entry 2).

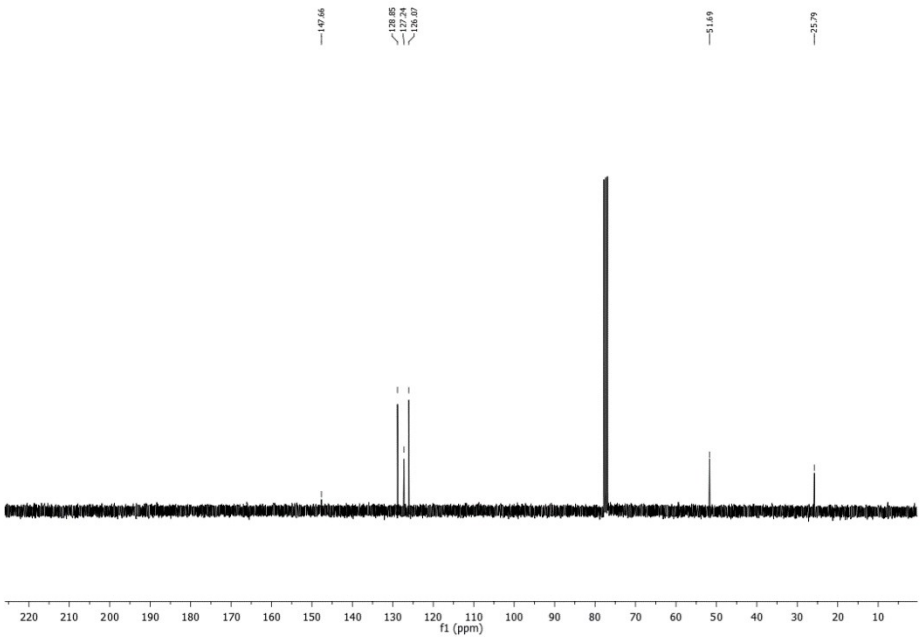


Figure 32. ^{13}C-NMR (75 MHz, $CDCl_3$) of 1-Phenylethylamine (Table 7, Entry 2).

3-Chlorobenzylamine (Table 7, Entry 3)[10]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33-7.30 (1H, m), 7.28- 7.19 (3H, m), 3.87 (2H, s) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.57, 134.64, 130.08, 127.52, 127.20, 125.48, 77.36, 46.25 ppm.

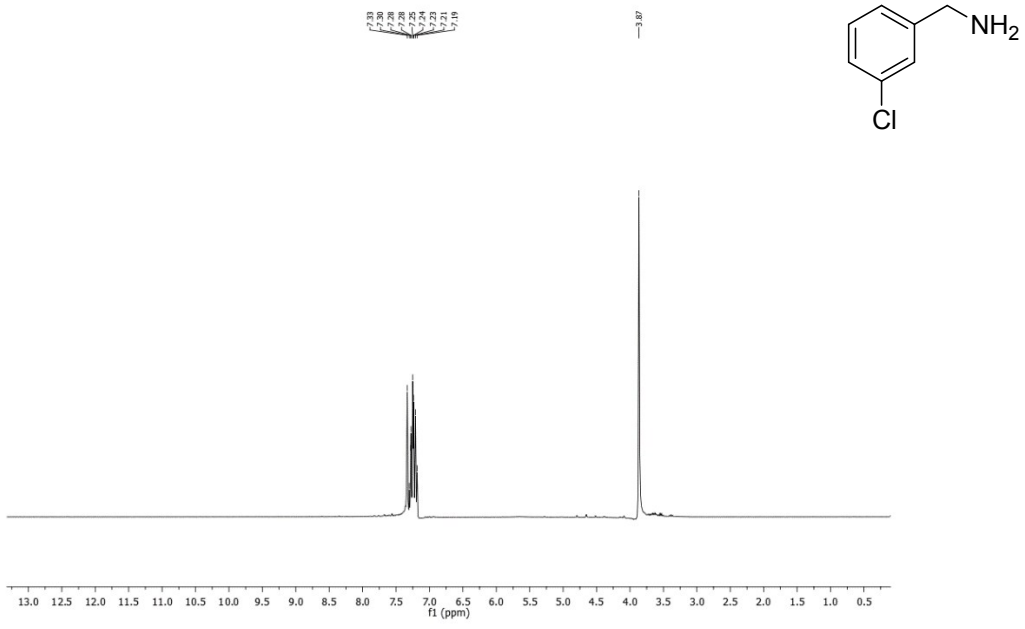


Figure 33. ^{1}H NMR (300 MHz, $CDCl_3$) of 3-Chlorobenzylamine (Table 7, Entry 3).

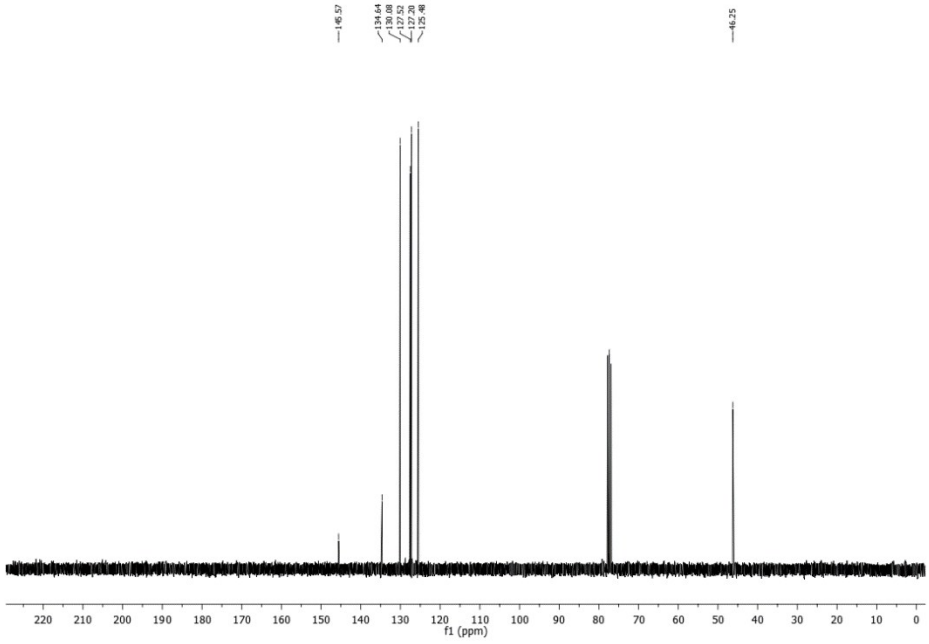
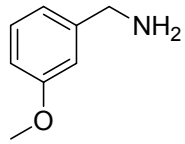


Figure 34. ^{13}C-NMR (75 MHz, $CDCl_3$) of 3-Chlorobenzylamine (Table 7, Entry 3).

3-Methoxybenzylamine (Table 7, Entry 5)[11]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.16 (1H, t, J =6 Hz), 6.81-6.79 (2H, m), 6.72- 6.68 (1H, m), 3.96 (2H, s), 3.72 (3H, s) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.24, 145.38, 129.96, 119.73, 112.98, 112.64, 55.60, 46.84 ppm.



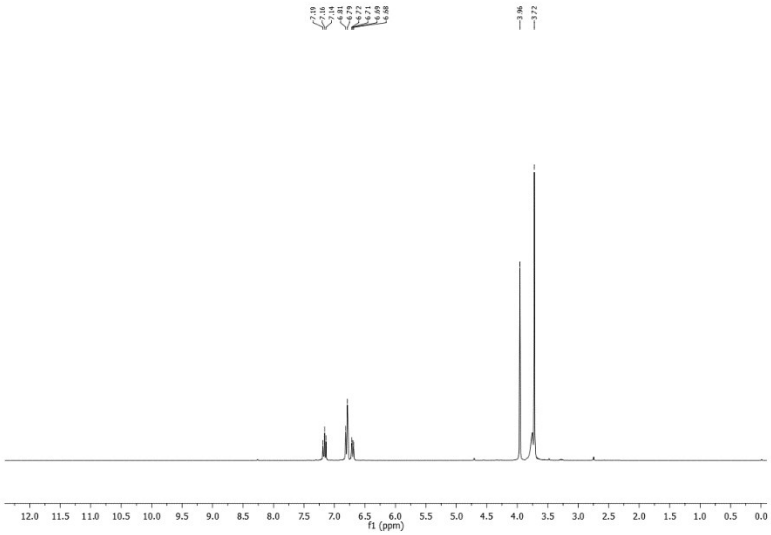


Figure 35. ^{1}H NMR (300 MHz, $CDCl_3$) of 3-Methoxybenzylamine (Table 7, Entry 5).

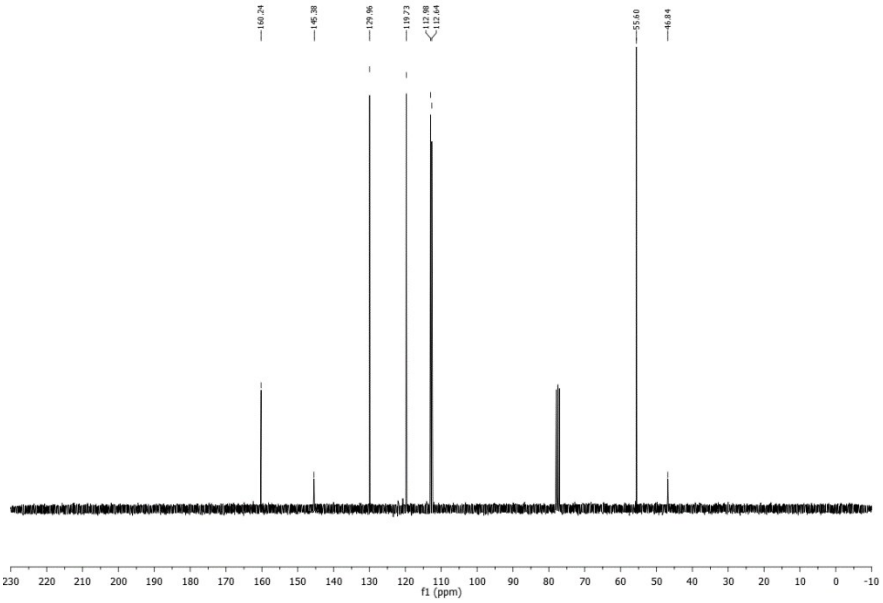


Figure 36. ^{13}C-NMR (75 MHz, $CDCl_3$) of 3-Methoxybenzylamine (Table 7, Entry 5).

3-Benzyloxypropylamine (Table 7, Entry 6)[12]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30-7.19 (5H, m), 4.43 (2H, s), 3.48 (2H, t, J = 6 Hz), 2.68 (2H, t, J = 6 Hz), 1.70 (2H, q, J = 6 Hz) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 138.68, 128.69, 127.93, 127.88, 73.29, 68.60, 39.72, 33.16.

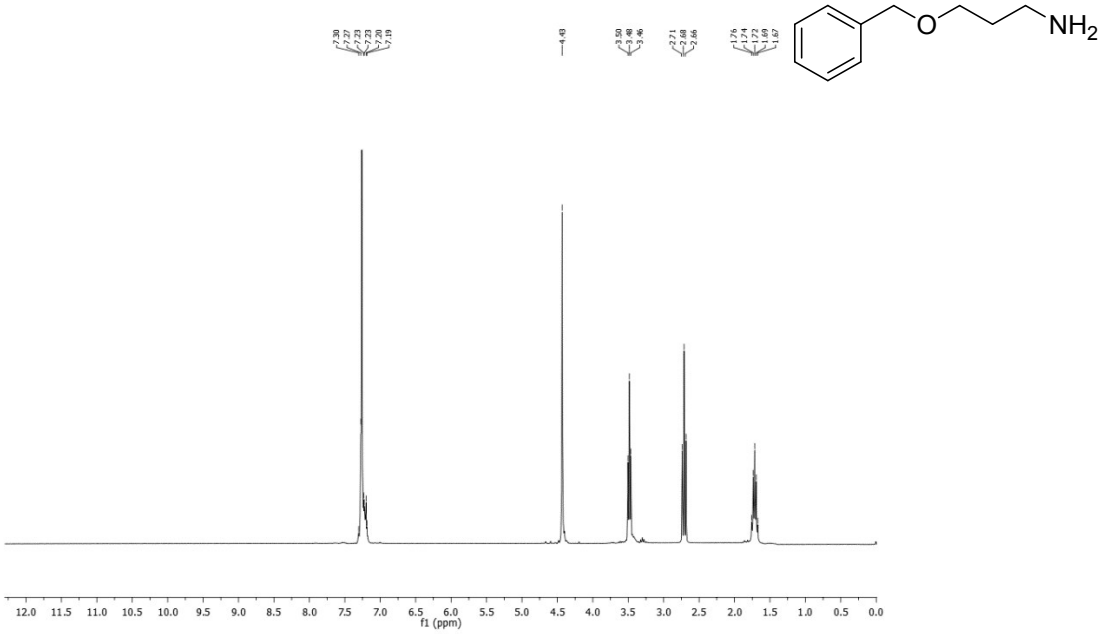


Figure 37. ^{1}H NMR (300 MHz, $CDCl_3$) of 3-Benzyloxypropylamine (Table 7, Entry 6).

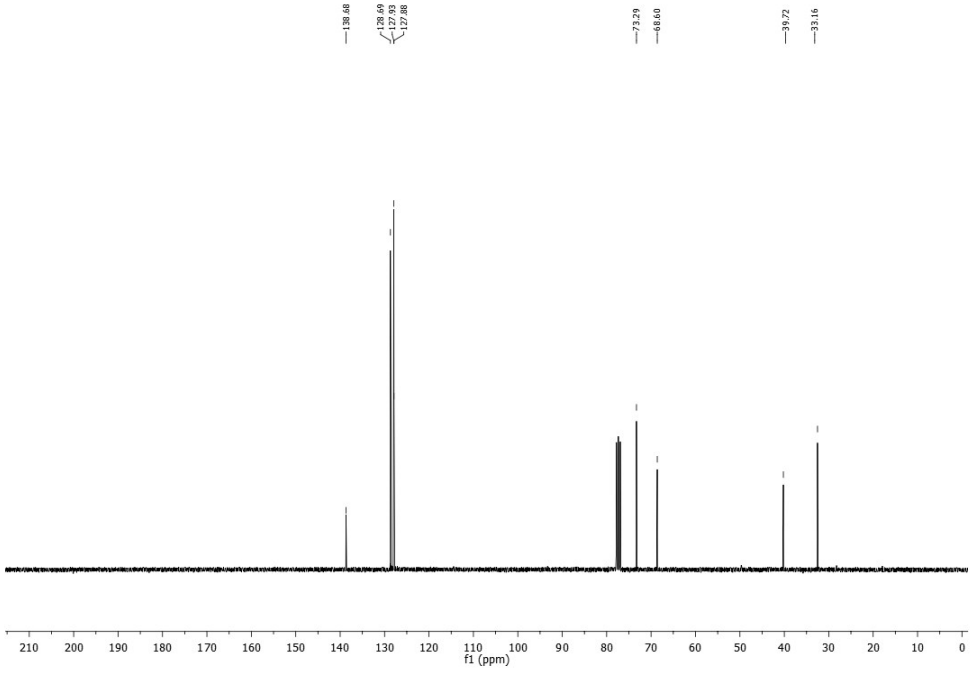


Figure 38. ^{13}C-NMR (75 MHz, $CDCl_3$) of 3-Benzyloxypropylamine (Table 7, Entry 6).

1-Octanamine (Table 7, Entry 7)[10]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.67 (2H, t, J = 9Hz), 1.43-1.40 (2H, m), 1.27 (10H, m), 0.87 (3H, t, J = 6 Hz) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 42.57, 34.13, 32.18, 29.80, 29.64, 27.24, 23.00, 14.44 ppm.

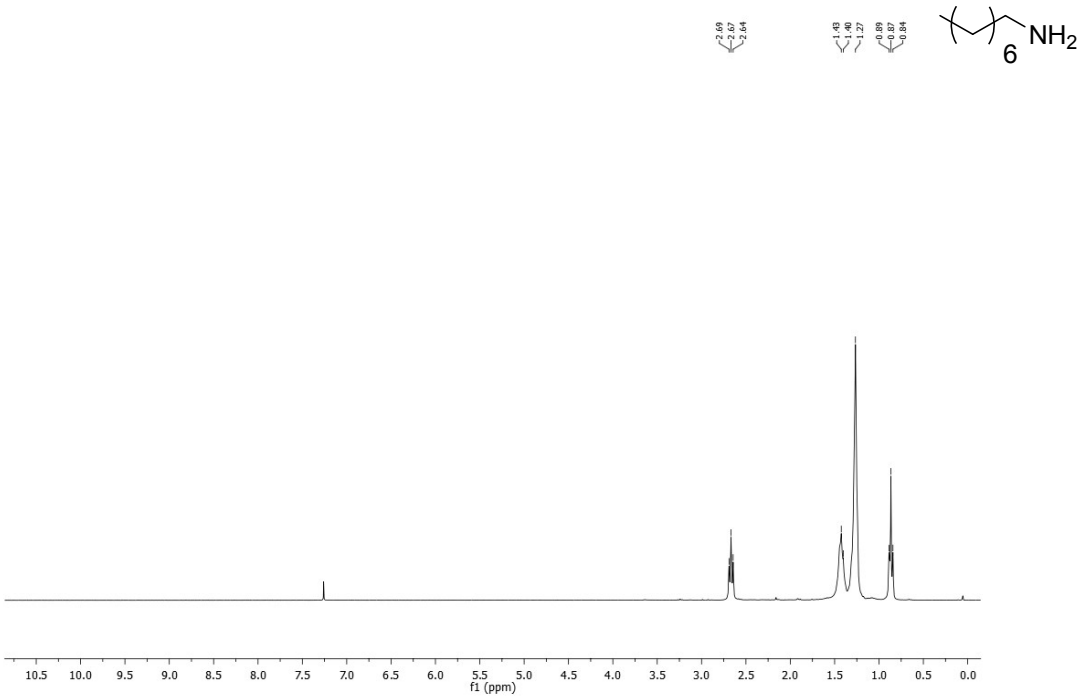


Figure 39. ^{1}H NMR (300 MHz, $CDCl_3$) of 1-Octanamine (Table 7, Entry 7).

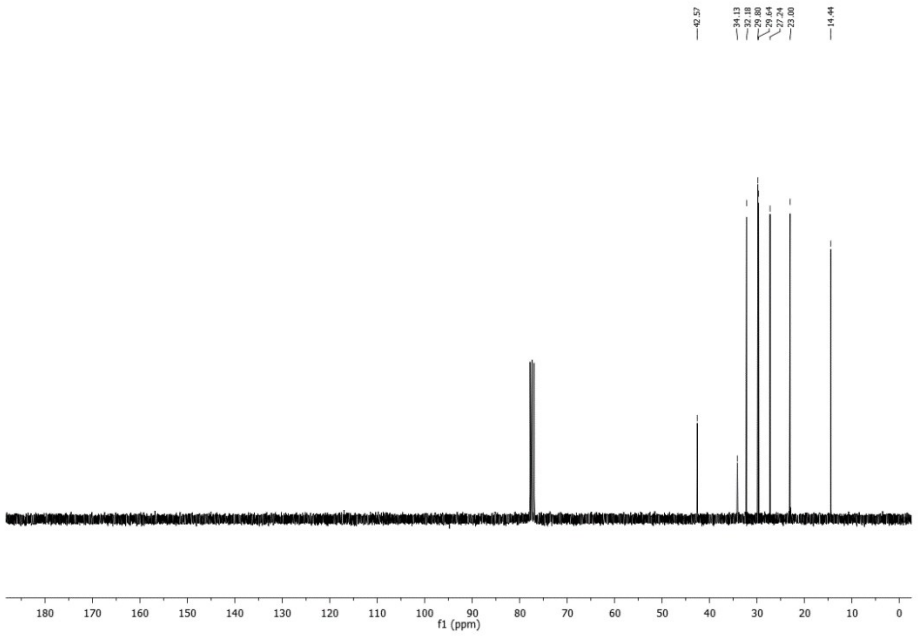
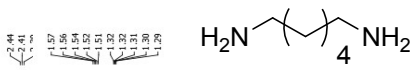


Figure 40. ^{13}C-NMR (75 MHz, $CDCl_3$) of 1-Octanamine (Table 7, Entry 7).

1,6-Diaminohexane (Table 7, Entry 8)[13]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.41 (4H, t, J = 6 Hz), 1.54 (4H, q, J = 3 Hz), 1.31 (4H, q, J = 3 Hz) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 42.35, 33.98, 27.00 ppm.



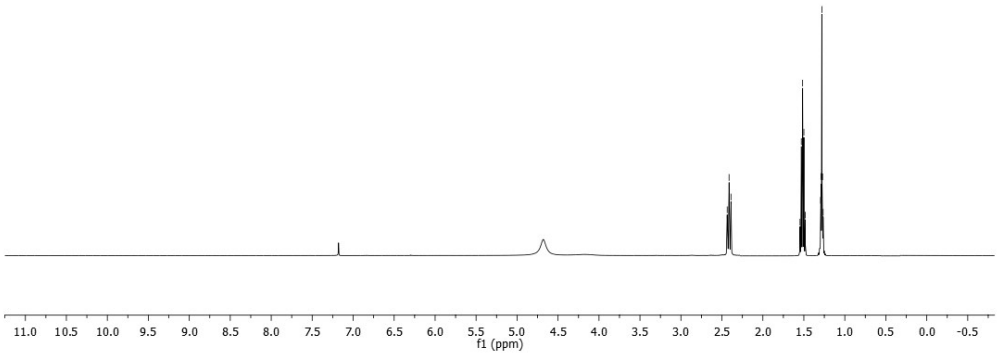


Figure 41. ^{1}H NMR (300 MHz, $CDCl_3$) of 1,6-Diaminohexane (Table 7, Entry 8).

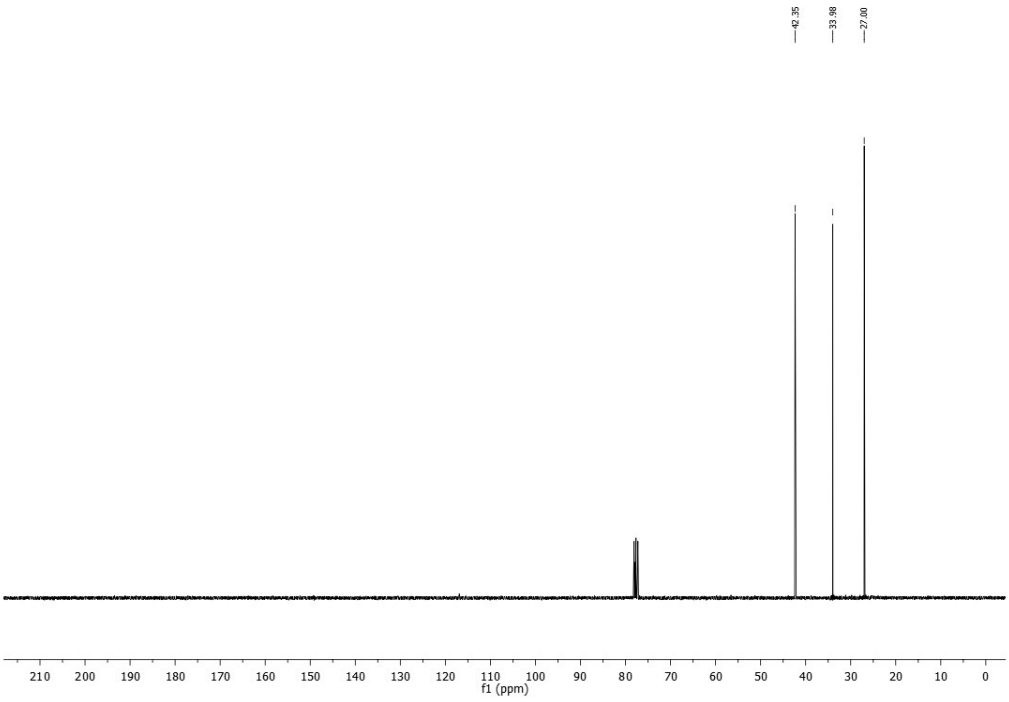
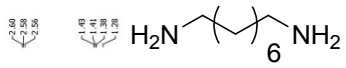


Figure 42.^{13}C-NMR (75 MHz, $CDCl_3$) of 1,6-Diaminohexane (Table 7, Entry 8).

1,8-Diaminooctane (Table 7, Entry 9)[14]: ^{1}H NMR (300 MHz, D_2O) δ 2.58 (4H, t, J = 6 Hz), 1.43- 1.38 (4H, m), 1.28 (8H, m) ppm; ^{13}C NMR (75 MHz, D_2O) δ 40.84, 31.77, 28.88, 26.37 ppm.



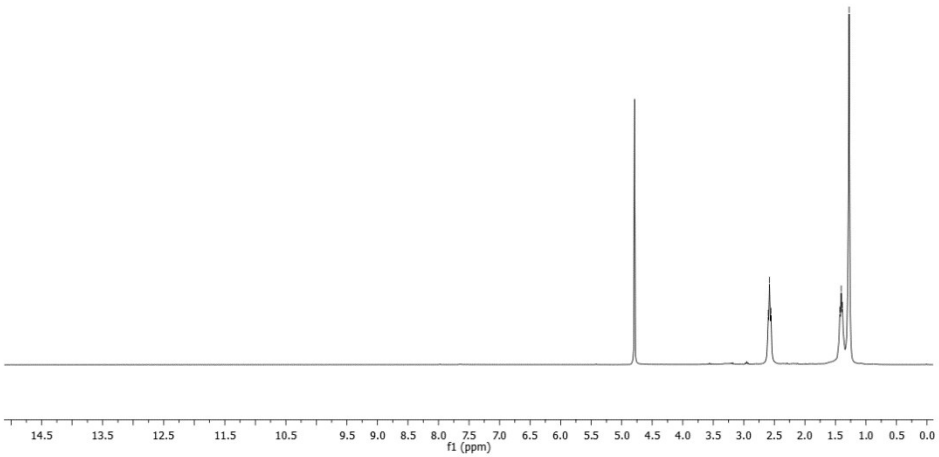


Figure 43. ^{1}H NMR (300 MHz, D_2O) of 1,8-Diaminooctane (Table 7, Entry 9).

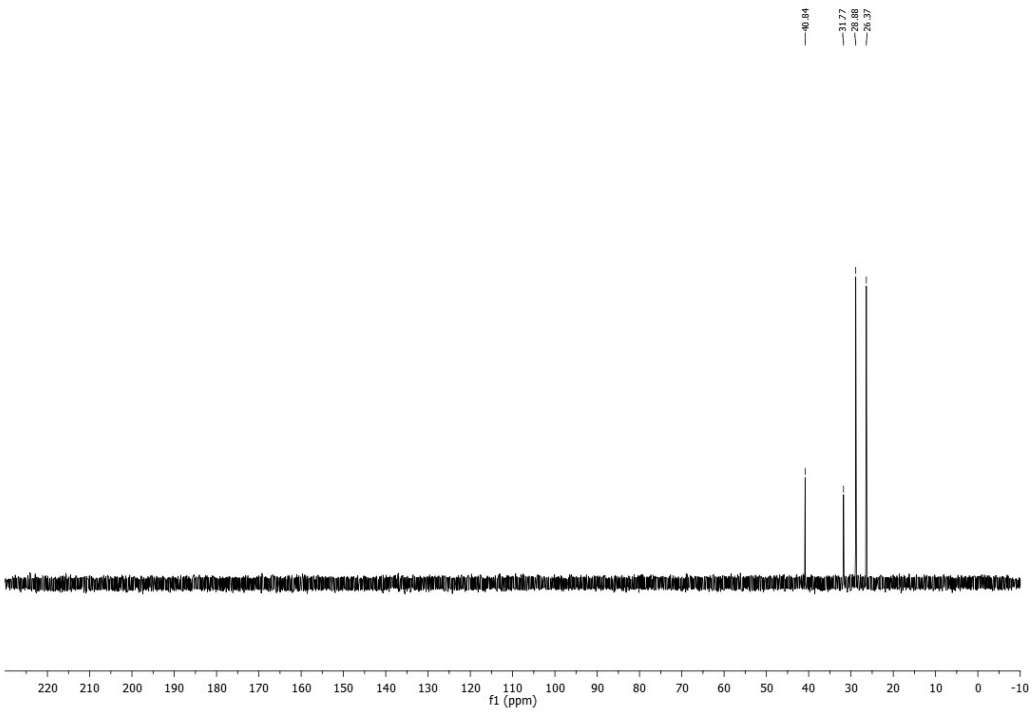


Figure 44. ^{13}C-NMR (75 MHz, D_2O) of 1,8-Diaminooctane (Table 7, Entry 9).

6-Amino-1-hexanol (Table 7, Entry 10)[15]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.54 (2H, t, J = 6 Hz), 2.68 (2H, t, J = 6 Hz), 1.53-1.30 (8H, m) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 62.58, 42.32, 33.17, 33.12, 27.00, 26.03 ppm.

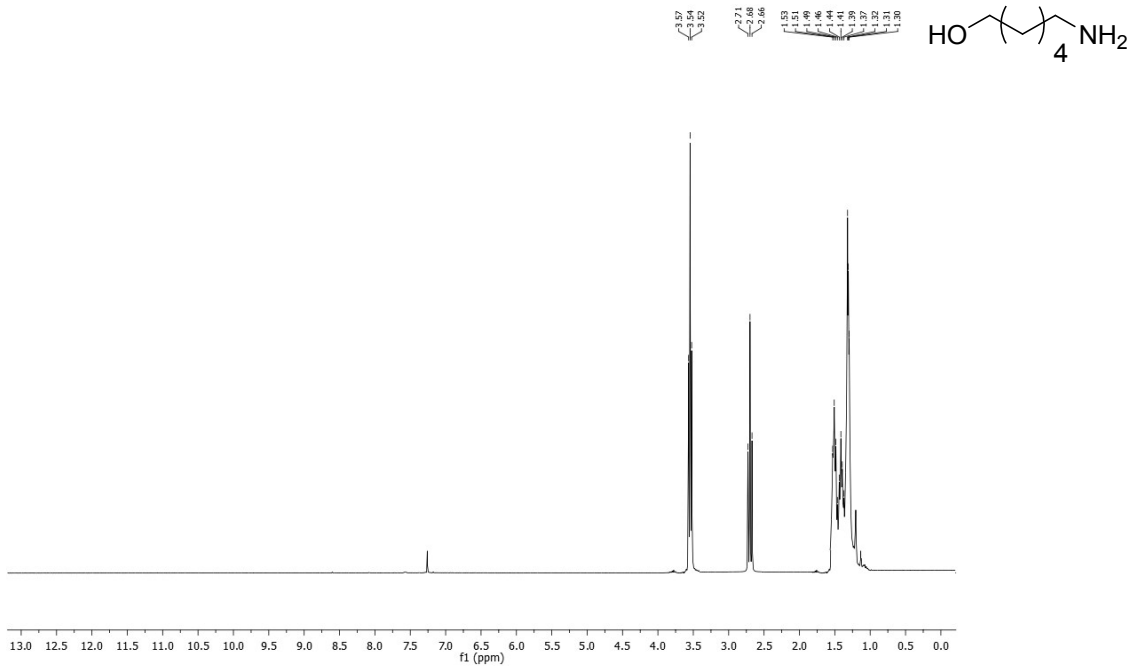


Figure 45. ^{1}H NMR (300 MHz, $CDCl_3$) of 6-Amino-1-hexanol (Table 7, Entry 10).

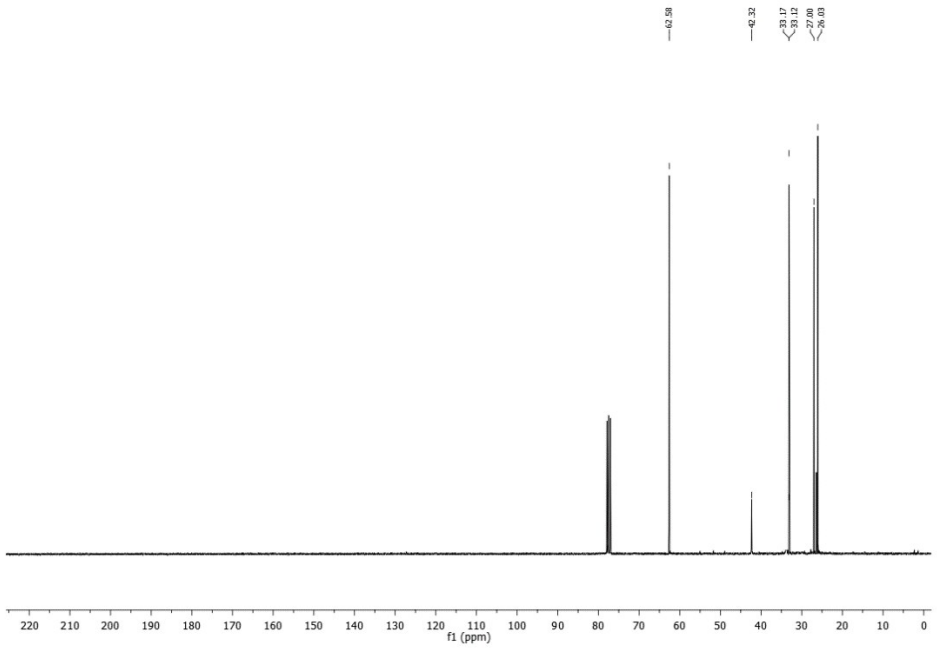


Figure 46. ^{13}C-NMR (75 MHz, $CDCl_3$) of 6-Amino-1-hexanol (Table 7, Entry 10).

10-Undecen-1-amine (Table 7, Entry 11)[16]: ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.86- 5.73 (1H, m), 5.00-4.89 (2H, m), 2.66 (2H, t, J = 6Hz), 2.02 (2H, q, J = 6 Hz), 1.82- 1.60 (2H, m), 1.44-1.24 (12H, m) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.54, 114.41, 42.50, 34.12, 32.23, 29.89, 29.44, 29.24, 27.20 ppm.

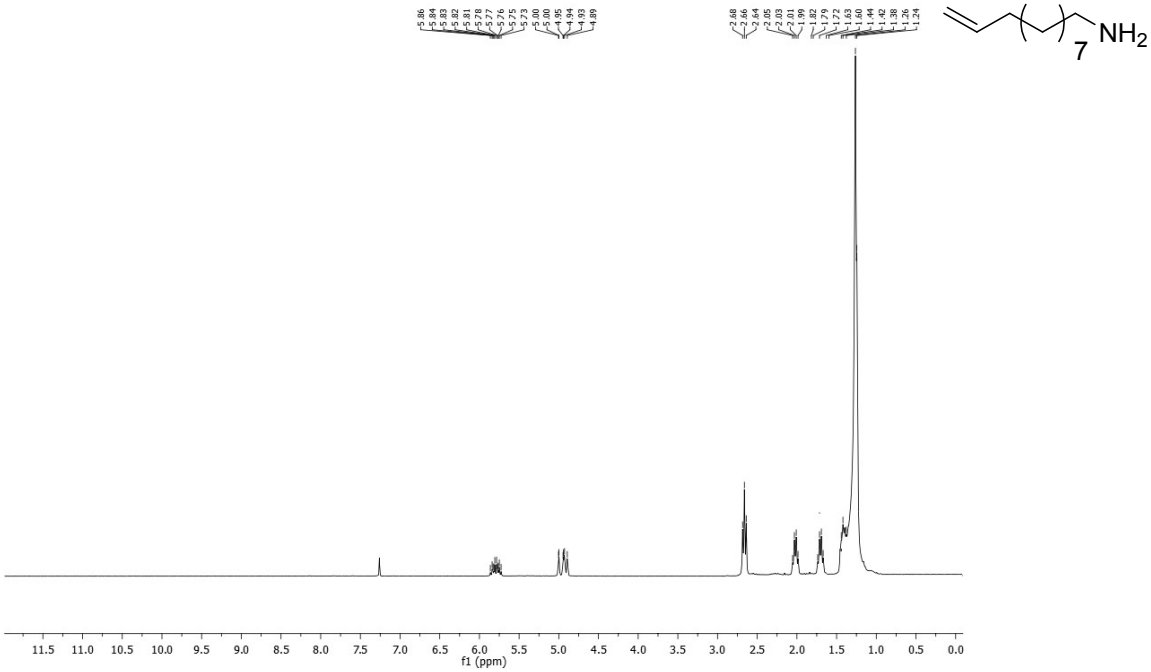


Figure 47. ^{1}H NMR (300 MHz, $CDCl_3$) of 10-Undecen-1-amine (Table 7, Entry 11).

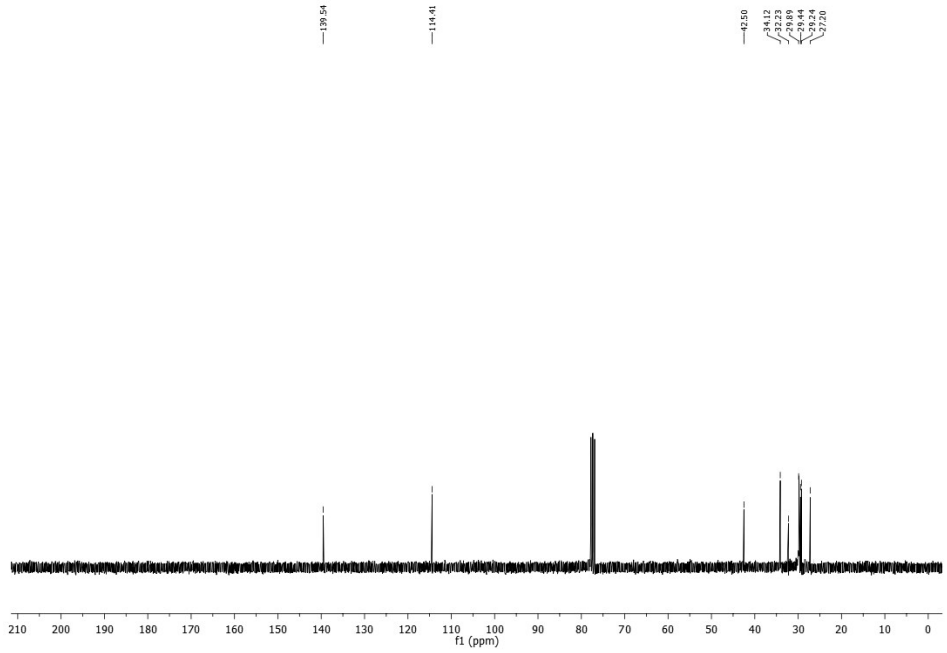


Figure 48. ^{13}C-NMR (75 MHz, $CDCl_3$) of 10-Undecen-1-amine (Table 7, Entry 11).

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