

Supporting Information

The first solvent-free method for the reduction of esters

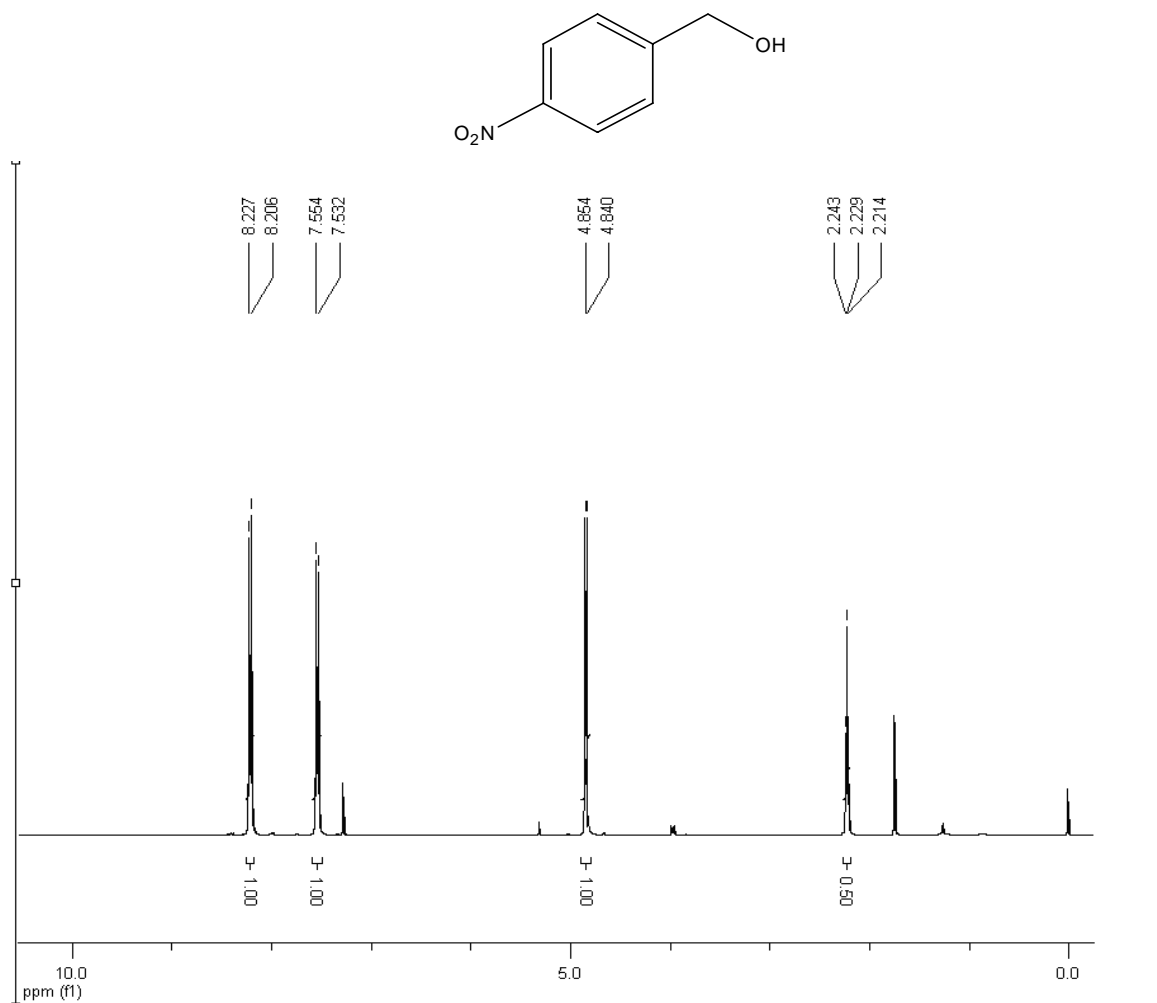
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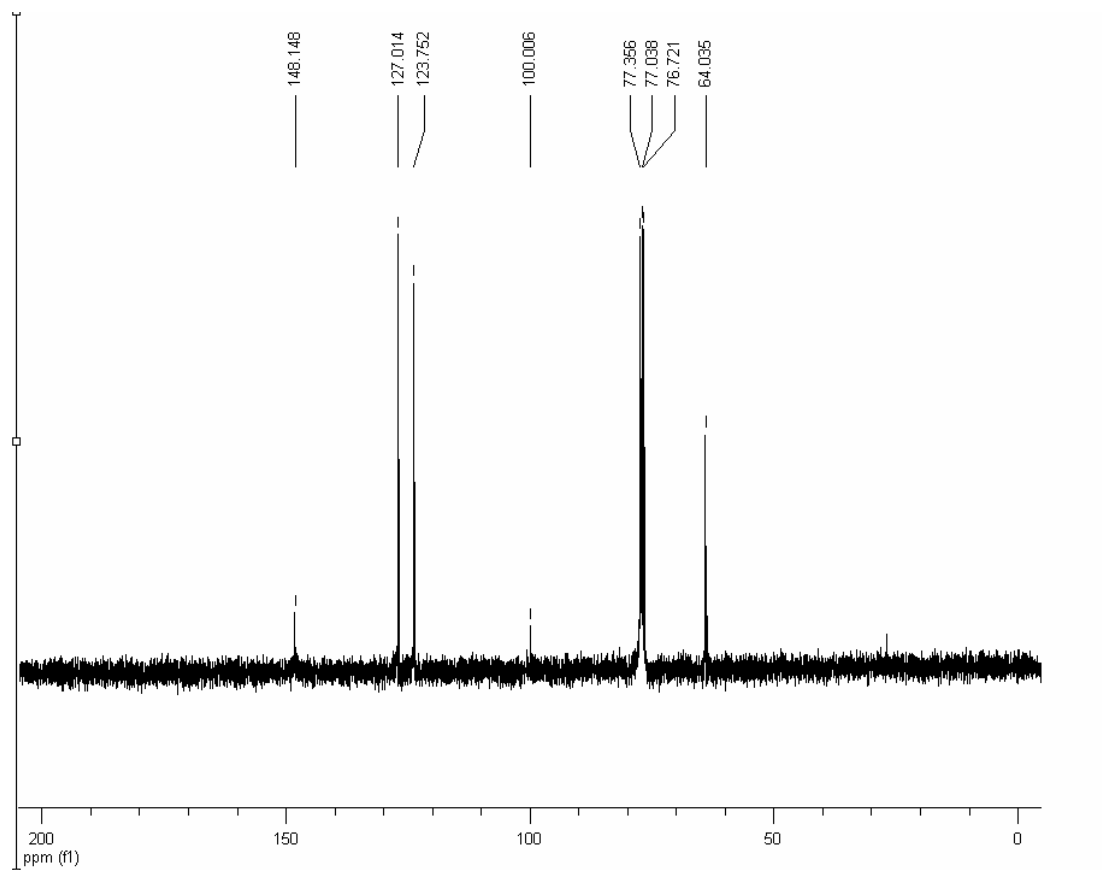
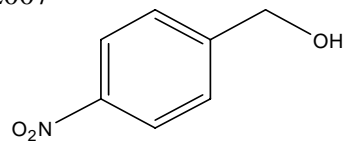
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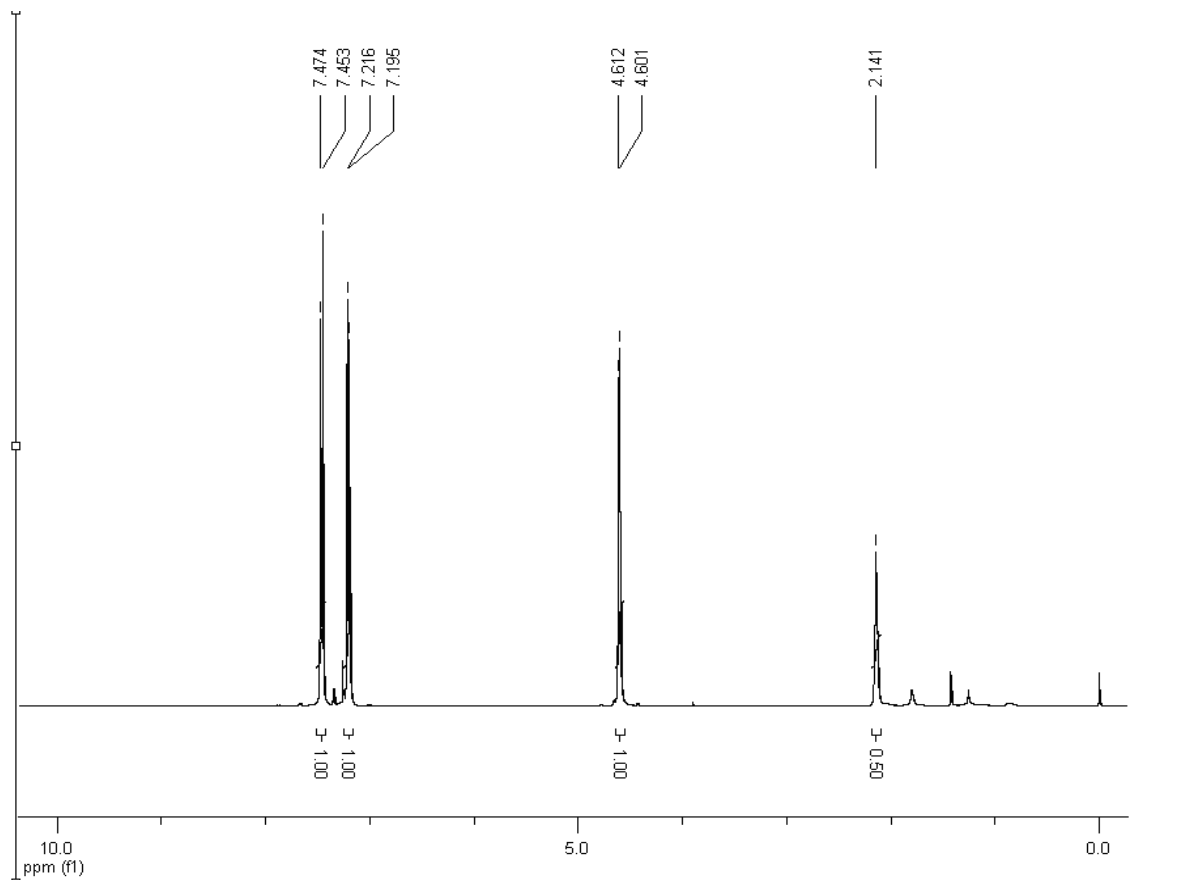
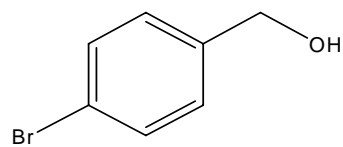
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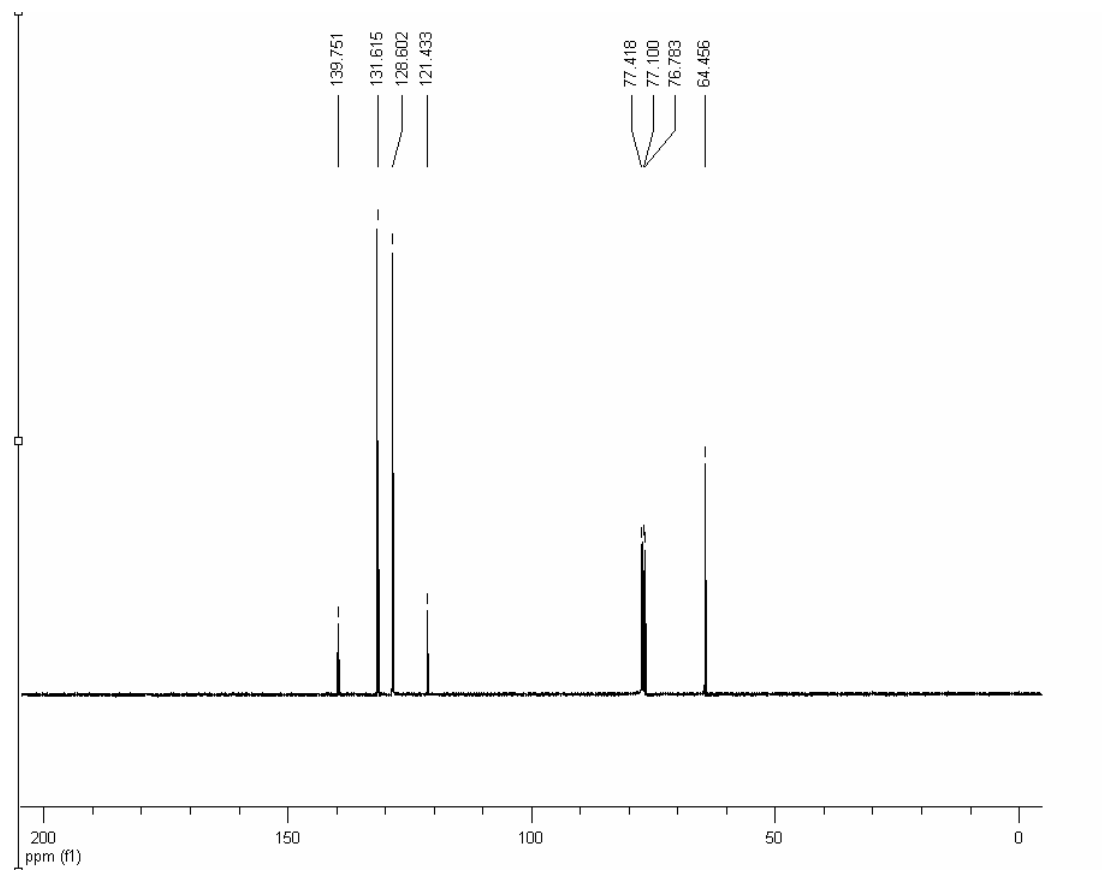
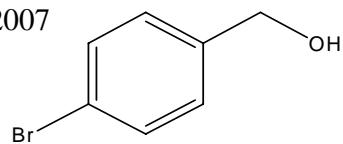
¹H NMR spectrum of (4-Nitro-phenyl)-methanol



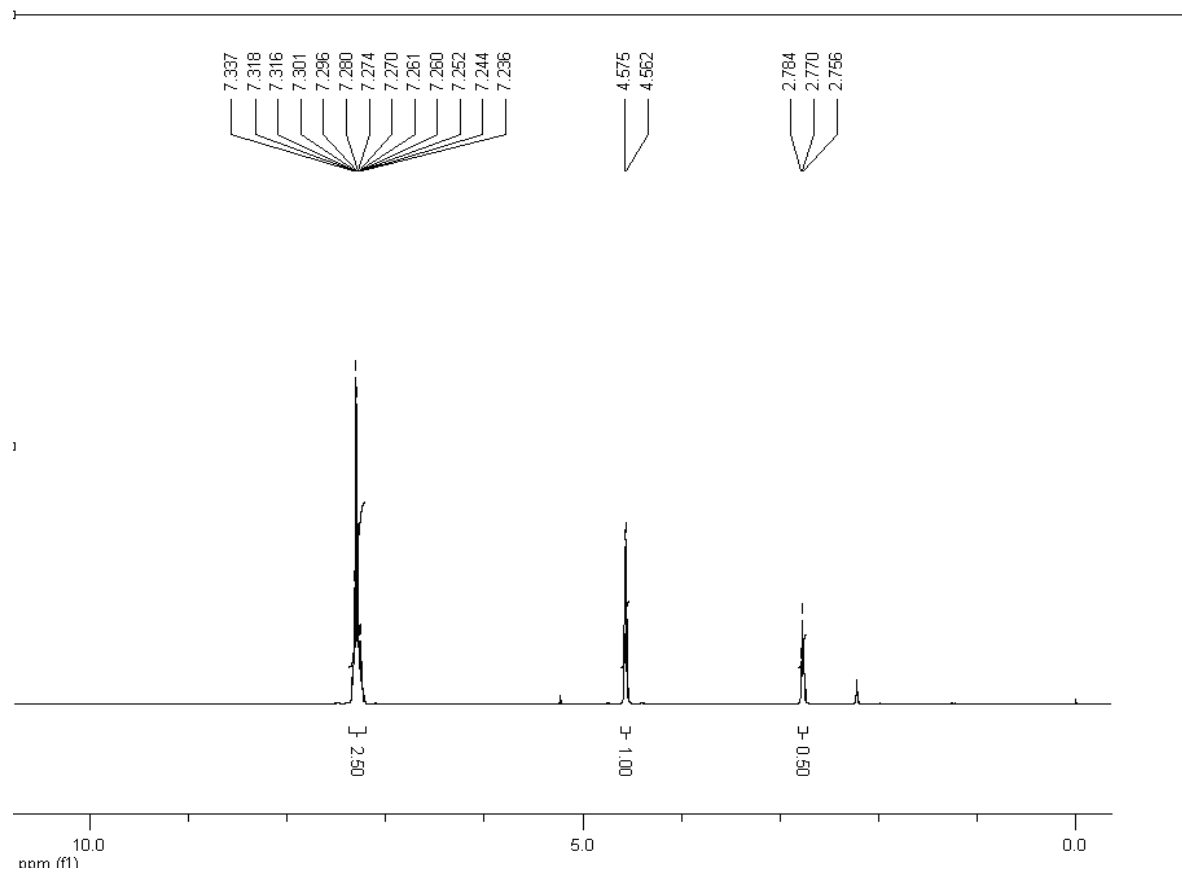
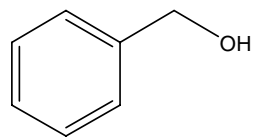
¹³C NMR spectrum of (4-Nitro-phenyl)-methanol



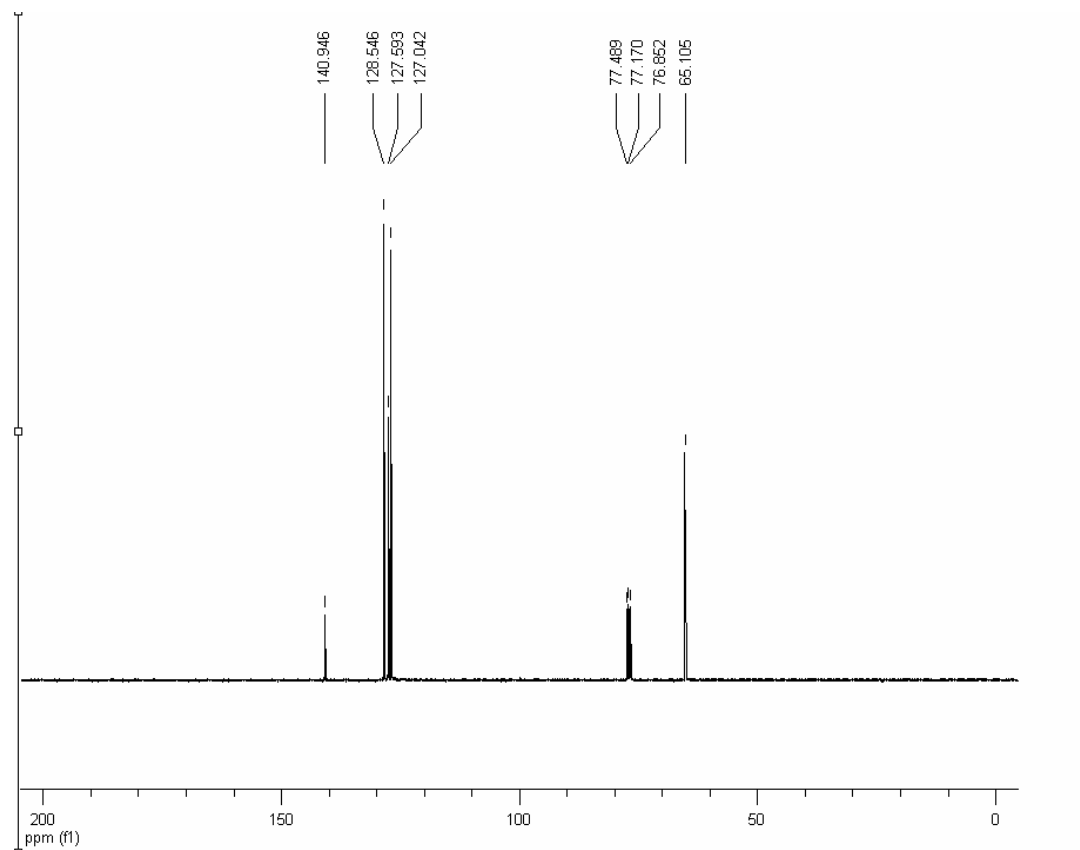
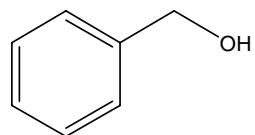
¹H NMR spectrum of (4-Bromo-phenyl)-methanol



^{13}C NMR spectrum of (4-Bromo-phenyl)-methanol



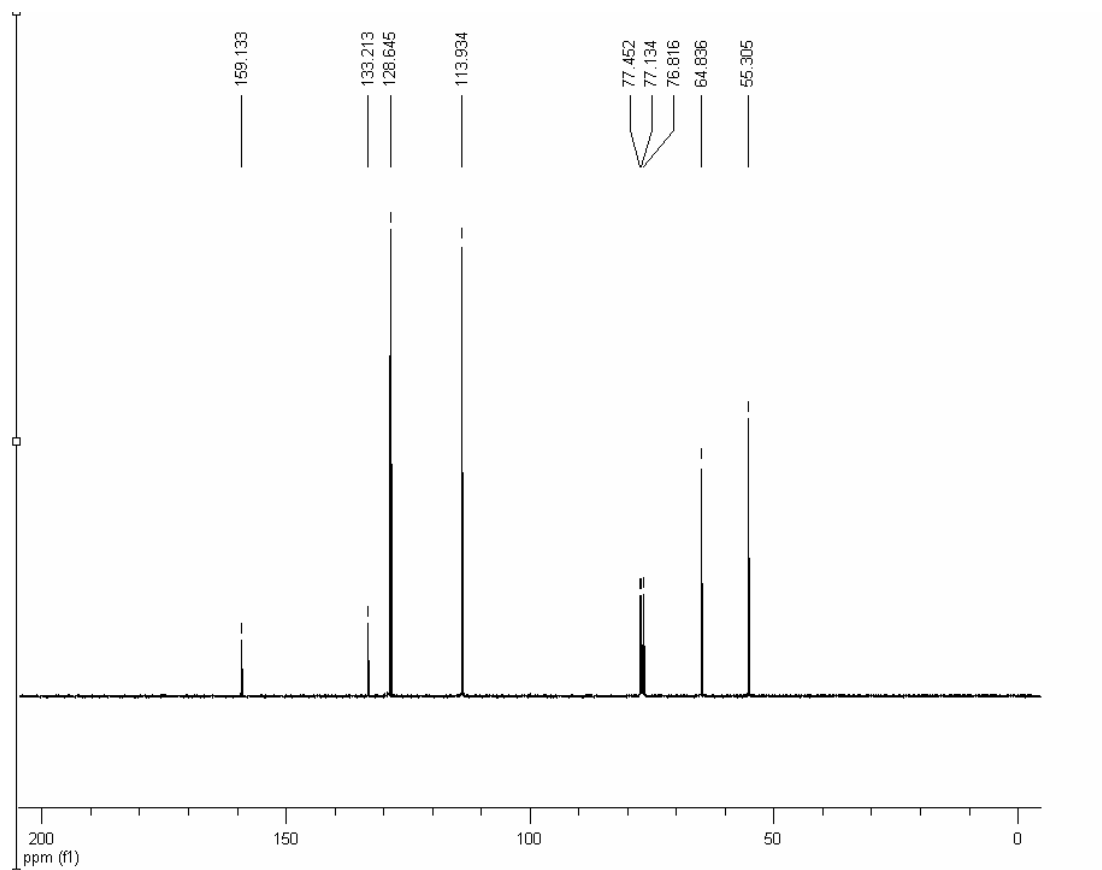
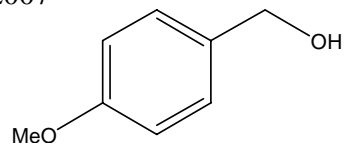
^1H NMR spectrum of Phenyl-methanol



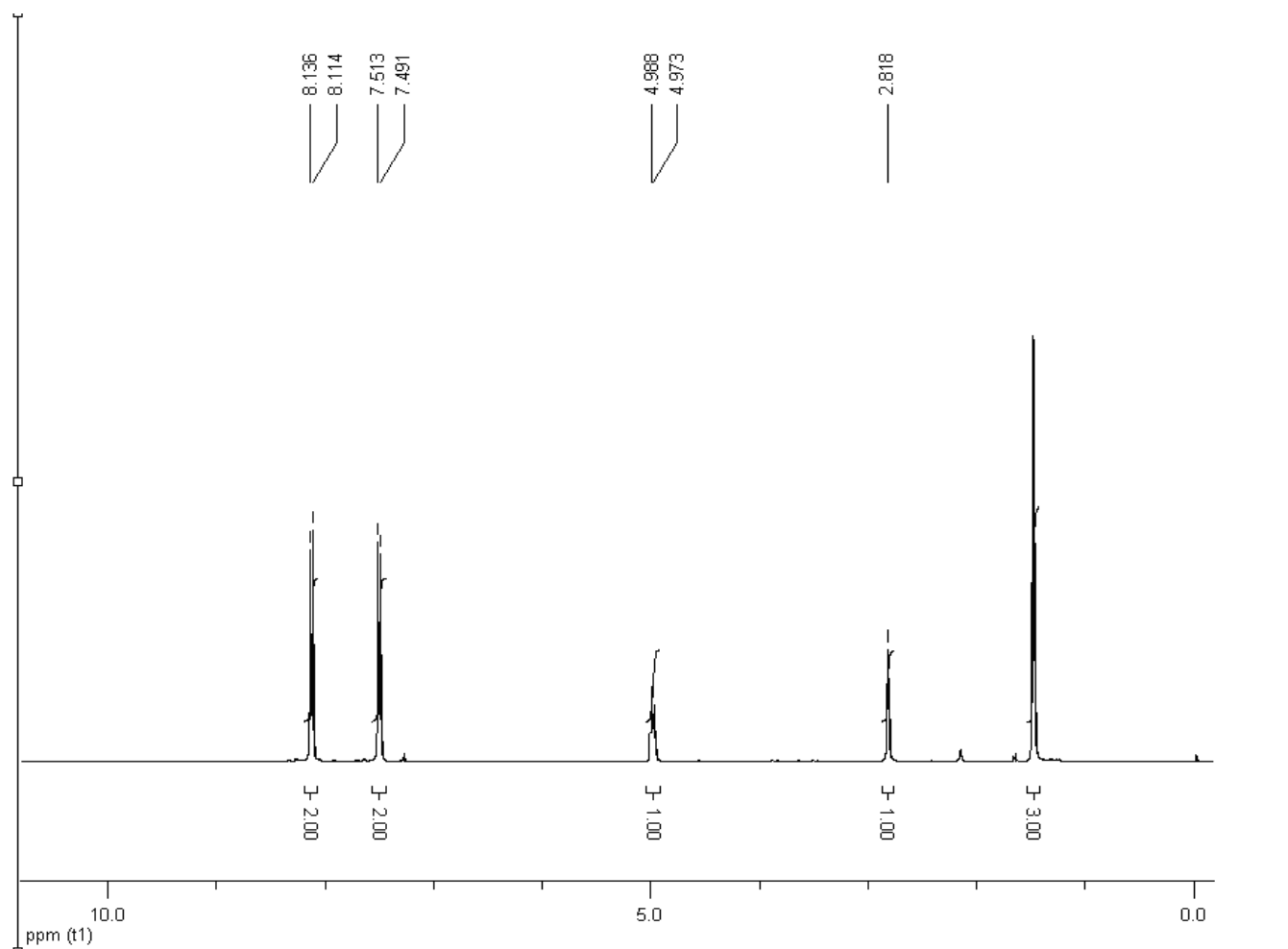
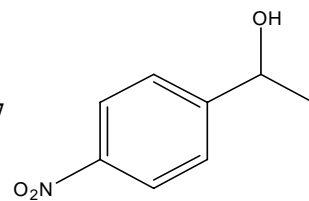
^{13}C NMR spectrum of Phenyl-methanol



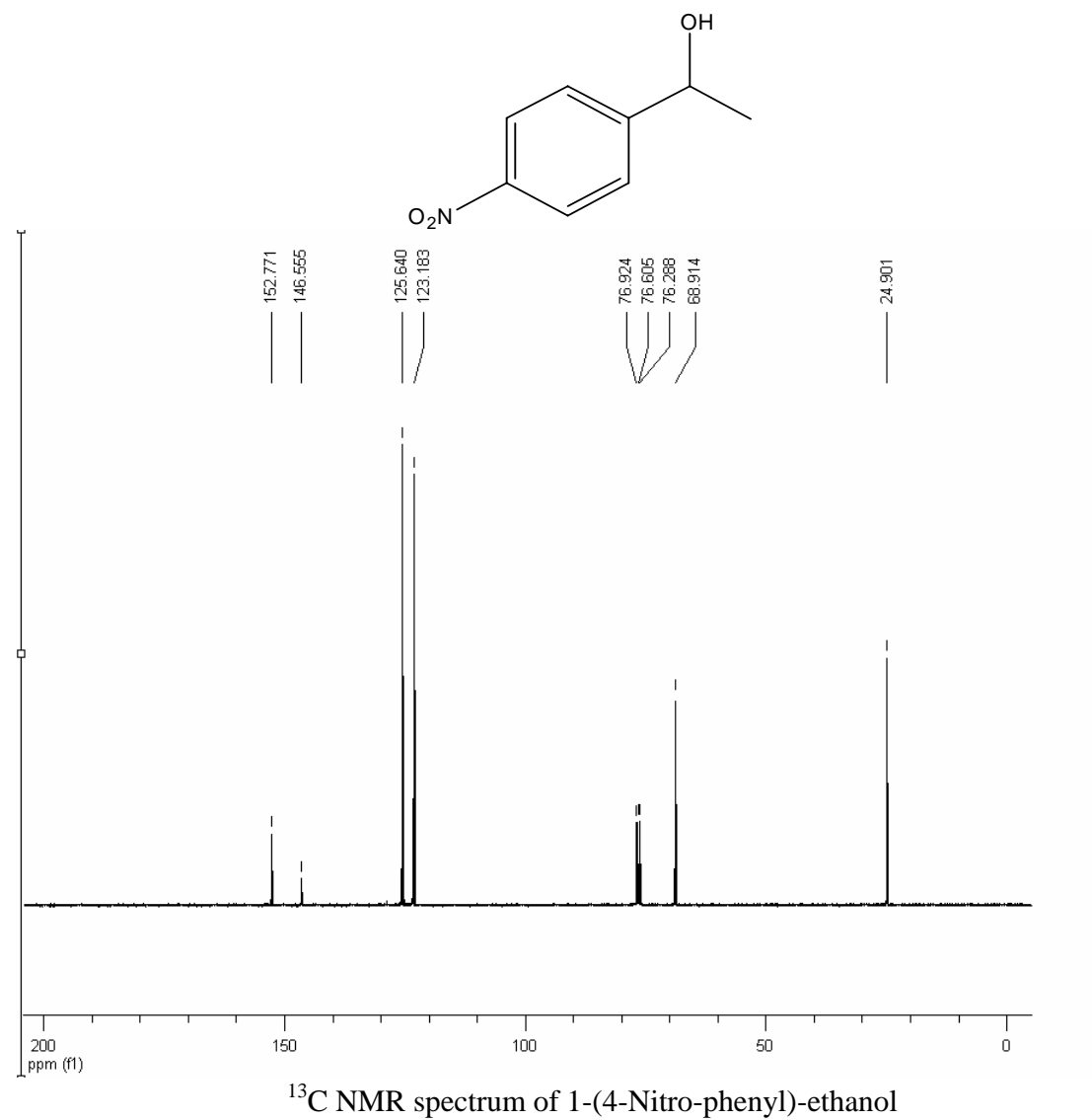
¹H NMR spectrum of (4-methoxy-phenyl)-methanol

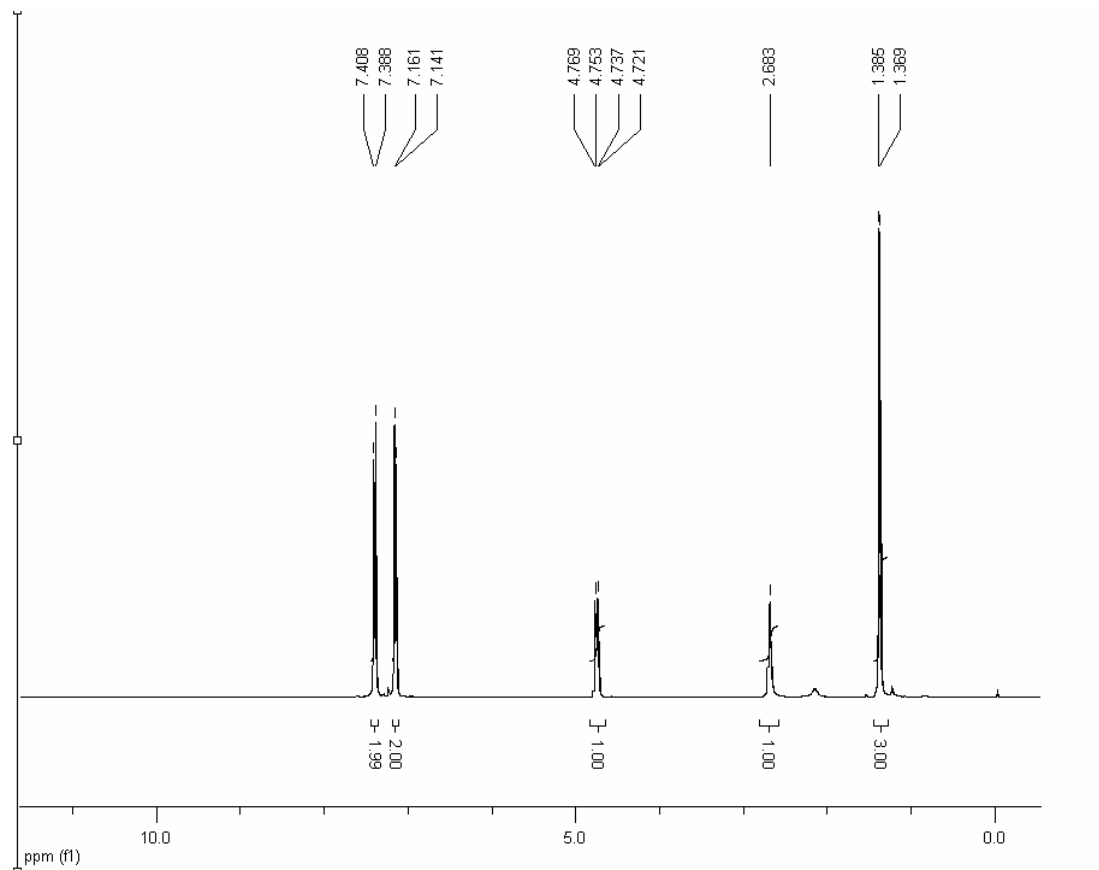
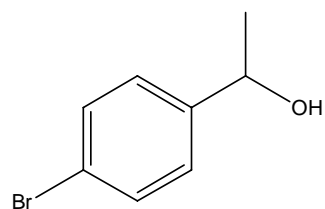


¹³C NMR spectrum of (4-Methoxy-phenyl)-methanol

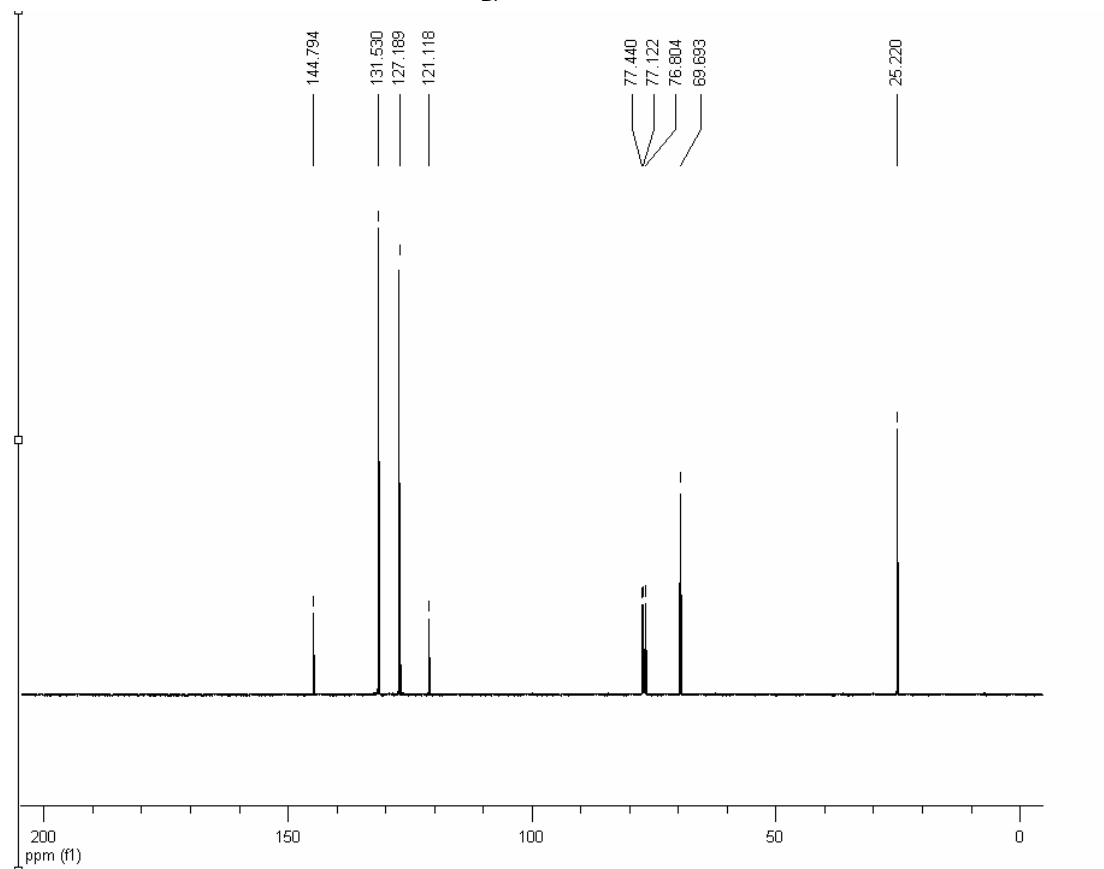
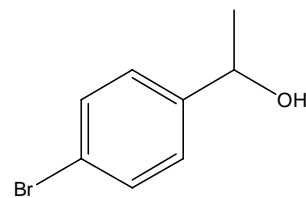


¹H NMR spectrum of 1-(4-Nitro-phenyl)-ethanol

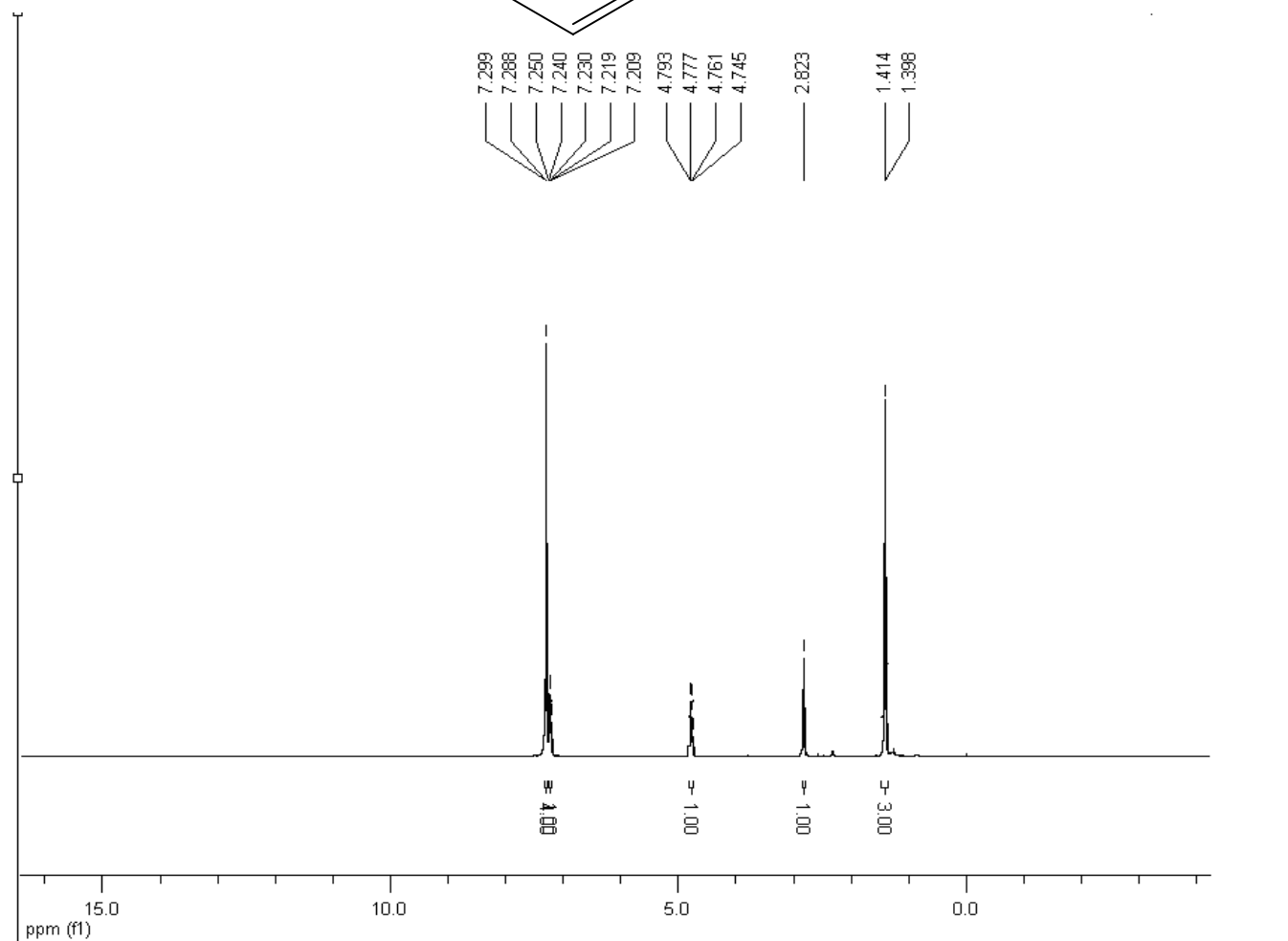
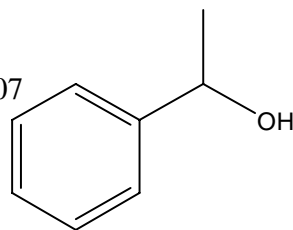




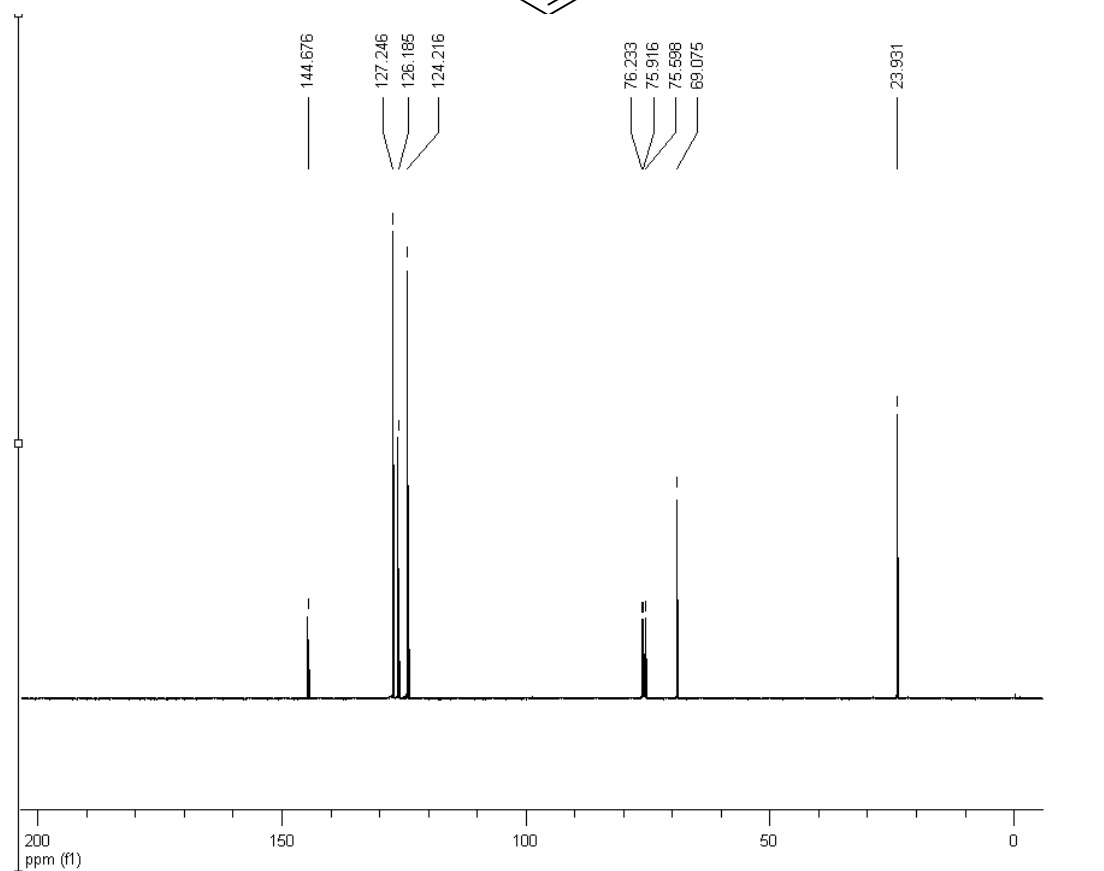
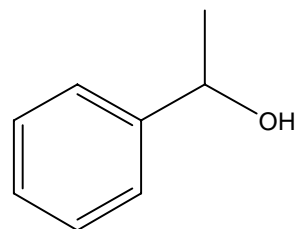
¹H NMR spectrum of 1-(4-Bromo-phenyl)-ethanol



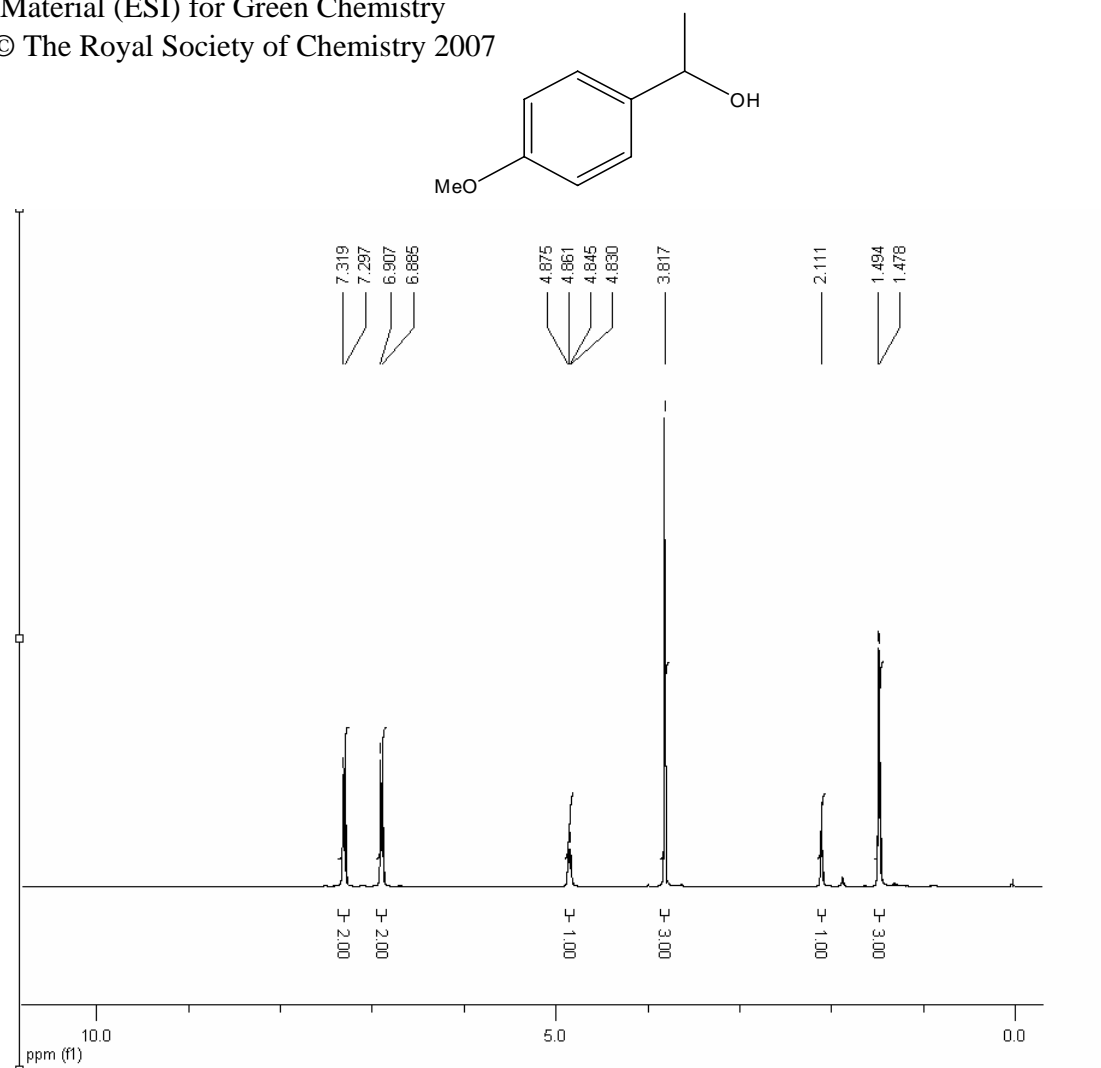
^{13}C NMR spectrum of 1-(4-Bromo-phenyl)-ethanol



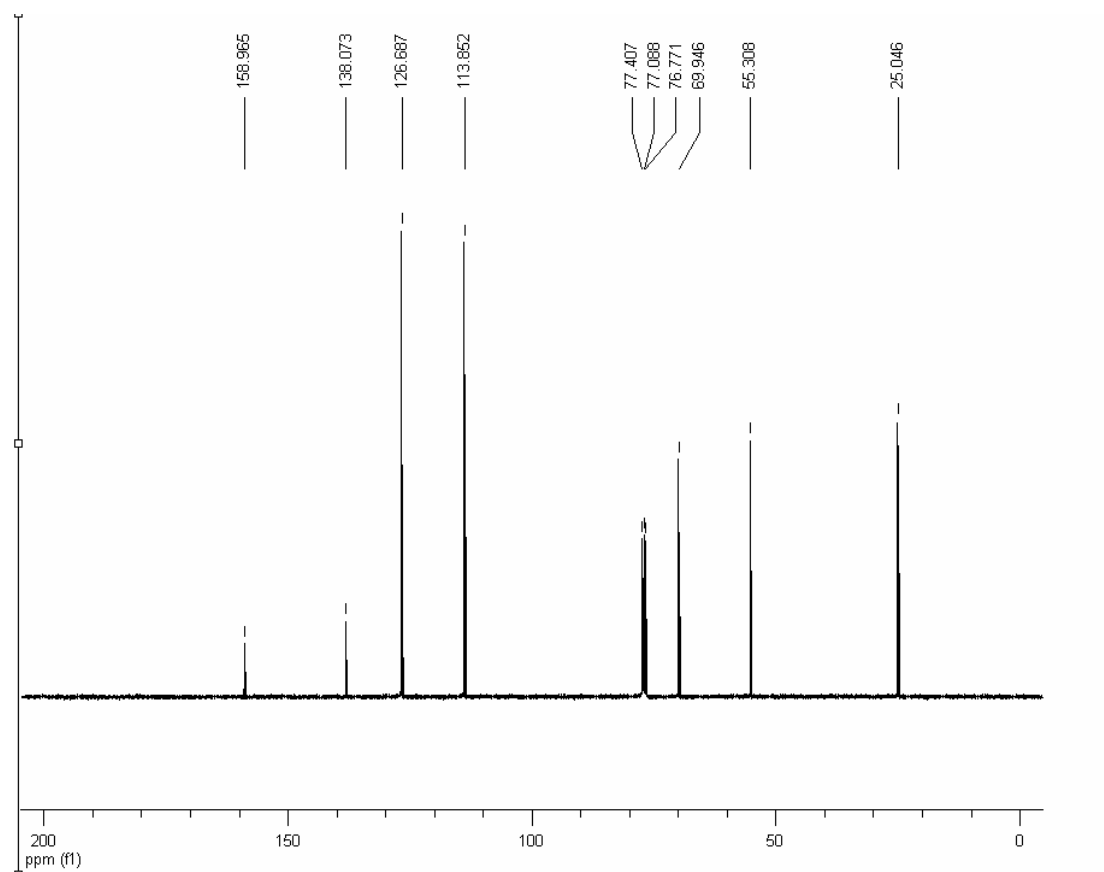
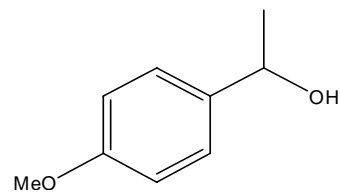
^1H NMR spectrum of 1-Phenyl-ethanol



^{13}C NMR spectrum of 1-Phenyl-ethanol



¹H NMR spectrum of 1-(4-Methoxy-phenyl)-ethanol



¹³C NMR spectrum of 1-(4-Methoxy-phenyl)-ethanol

Experimental Section

¹H NMR spectra were recorded on a Bruker Avance 400 spectrometer. Deuterated NMR solvents were obtained from Cambridge Isotope Laboratories, Inc., Andover MA, and used without further purification., *p*-nitrobenzaldehyde, *p*-bromobenzaldehyde, anisaldehyde, benzaldehyde, *p*-nitroacetophenone, *p*-bromoacetophenone, *p*-methoxyacetophenone, acetophenone, methyl-*p*-nitrobenzoate, methyl-*p*-bromobenzoate, methyl-*p*-methoxybenzoate, methyl benzoate, and sodium borohydride were purchased from Acros Organics and used without further purification. Ball bearings were purchased from Small Parts inc. Ball milling was carried out in an 8000M SpexCertiprep Mixer/Mill purchased from Spex certiprep.

Typical procedure for the reduction of aldehydes: *p*-bromobenzaldehyde (0.281 g, 1.52 mmol) and sodium borohydride (0.057 g, 1.52 mmol) were added to a custom-made 2.0 inch by 0.5 inch screw capped stainless steel vial along with a 0.250 inch aluminum oxide ball bearing. The vial was placed in an 8000M Spex Certiprep mixer/mill and the contents were ball milled for 1 h. The resulting mixture was quenched with a 10% HCl (50 mL). The solid was washed with water and filtered to dryness. For further purification the solid was recrystallation in methanol to give a yellowish solid (0.200 g, 1.07 mmol) in 70% yield.

Typical procedure for the reduction of ketones: *p*-bromoacetophenone (0.236 g, 1.19 mmol) and sodium borohydride (0.045 g, 1.19 mmol) were added to a custom-made 2.0 inch by 0.5 inch screw capped stainless steel vial along with a 0.250 inch aluminum oxide ball bearing. The vial was placed in an 8000M Spex Certiprep mixer/mill and the contents were ball milled for 6 h. The

resulting mixture was quenched with a 10% HCl (50 mL). The solid was washed with water and filtered to dryness. For further purification the solid was recrystallized in methanol to give a yellowish solid (0.174 g, 0.87 mmol) in 73% yield.

Typical procedure for the reduction of esters: methyl-*p*-bromobenzoate (0.211 g, 0.98 mmol) sodium borohydride (0.037 g, 0.98 mmol) and lithium chloride (0.208 g, 4.9 mmol) were added to a custom-made 2.0 inch by 0.5 inch screw capped stainless steel vial along with a 0.250 inch aluminum oxide ball bearing. The vial was placed in an 8000M Spex Certiprep mixer/mill and the contents were ball milled for 17 h. The resulting mixture was quenched with a 10 % HCl (50 mL). The solid was washed with water and filtered to dryness. For further purification the solid was recrystallized in methanol to give a yellowish solid (0.155 g, 0.84 mmol) in 85 % yield.