Supporting Information

Fiber-supported Fe(III) complex catalyst in spinning basket reactor for cleaner ring-opening of epoxides with alcohols

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Experimental details

Materials

Commercially available PANF (93.0% acrylonitrile, 6.5% methyl acrylate, and 0.4-0.5% sodium styrene sulfonate) with a length of 10 cm and a diameter of 30 ± 0.5 μm (from the Fushun Petrochemical Corporation of China) was used after dried (In order to avoid the possible impacts of water absorption on the subsequent reactions and the consequent possible sources for experimental errors, all the fiber samples before use were dried fully at 60 °C under vacuum in our studies). Diethylenetriamine used in this study was brought from Aladdin (Shanghai), all other chemicals used were analytical grade and employed without further purification. Water was deionized.

Apparatus and instruments

The iron content of the fibers was measured by inductively coupled plasma (ICP) on a PE5300DV analyzer. Elemental analyses were performed on a thermo scientific flash 2000 auto-analyzer. Fourier transform infrared (FTIR) spectra were obtained with an AVATAR 360 FTIR spectrometer (Thermo Nicolet), KBr disc. The mechanical properties of different fiber samples were tested with an electronic single fiber strength tester (Laizhou Electronic Instrument Co., Ltd of China, model LLY-06E). A scanning electron microscope (Hitachi, model S-4800) was used to characterize the surface morphology of the fibers. ^{1}H NMR spectra were recorded on an AVANCE III (Bruker, 400 MHz) instrument using TMS as the internal standard. ^{13}C NMR spectra were recorded on an AVANCE III (Bruker, 101 MHz) instrument with complete proton decoupling.

The detection method of inductively coupled plasma (ICP) analysis

5 mg of fiber samples were digested in 5 mL of aqua regia. After digestion, the solution was diluted to 100 mL of de-ionized water in a measuring flask. Then the concentration was determined by ICP spectrometer.

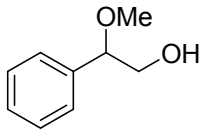
The method for testing the mechanical properties of fiber samples

The mechanical properties of the fiber samples were tested by an electronic single fiber strength tester. For each sample, 30 single fibers were selected randomly to test their breaking strength and tension, then took averages as the final breaking strength and tension of the special fiber sample, and the retention rate of breaking strength and tension were based on the original polyacrylonitrile fiber.

The experimental details of hot filtration test

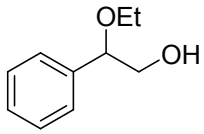
Styrene oxide (2 mmol) was dissolved in absolute ethanol (10 mL) in the dried spinning basket reactor (50 mL) with fiber catalyst $PANF_{DTA}$@Fe(III) (5 mol% Fe(III) content based on styrene oxide) in its impellers. The reaction mixture was stirred at room temperature for 15 min, and then the fiber catalyst was quickly removed form the system, next, the residue mixture was stirred for the subsequent time (1 h 15 min). After completion of the reaction, the mixture was let out through the discharge spout, and the reaction vessel was washed with ethanol (10 mL) which was combined to the mixture, then the combined solvents were evaporated, and the residue was purified by column chromatography over silica gel (eluent: petrol ether/ethyl acetate = 5/1) to afford the β-alkoxy alcohol with a very low yield of 29%.

^{1}H NMR and ^{13}C NMR spectra characterization data of compounds



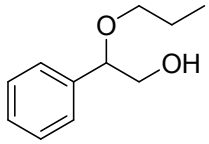
2-Methoxy-2-phenylethanol. (3a)[1]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36-7.24 (m, 5H), 4.20 (dd, *J* = 8.3, 3.9 Hz, 1H), 3.68-3.57 (m, 2H), 3.27 (s, 3H), 2.85 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.4, 128.8, 128.4, 127.2, 84.3, 67.3, 55.7.



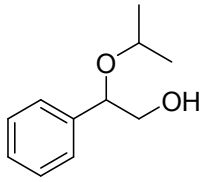
2-Ethoxy-2-phenylethanol. (3b)[2]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-7.26 (m, 5H), 4.43-3.65 (m, 2H), 3.59 (dd, *J* = 11.7, 3.9 Hz, 1H), 3.55-3.35 (m, 2H), 2.82 (s, 1H), 1.22 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 139.2, 129.1, 128.9, 126.2, 81.9, 67.5, 64.2, 15.0.



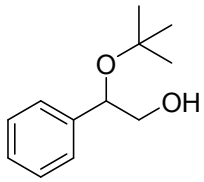
2-Phenyl-2-propoxyethanol. (3c)[2]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-7.26 (m, 5H), 4.43-3.67 (m, 2H), 3.57 (dd, *J* = 11.7, 4.2 Hz, 1H), 3.40-3.28 (m, 2H), 2.46 (s, 1H), 1.72-1.51 (m, 2H), 0.91 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 139.3, 129.0, 128.3, 126.2, 81.9, 70.5, 67.5, 23.4, 10.8.



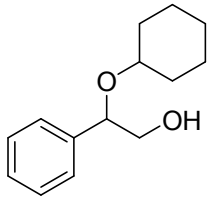
2-iso-Propoxy-2-phenylethanol. (3d)[1]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36-7.29 (m, 5H), 4.55 (dd, *J* = 8.1, 3.9 Hz, 1H), 3.70-3.59 (m, 3H), 2.39 (s, 1H), 1.29-1.11 (m, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 139.2, 129.1, 128.9, 126.7, 80.1, 68.1, 66.2, 22.9, 21.0.



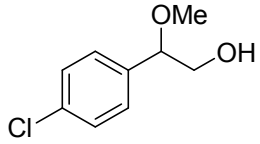
2-tert-Butoxy-2-phenylethanol. (3e)[1]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38-7.26 (m, 5H), 4.63 (dd, *J* = 8.4, 4.2 Hz, 1H), 3.54-3.46 (m, 2H), 2.42 (s, 1H), 1.18 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 142.3, 128.2, 127.4, 126.4, 75.2, 74.9, 68.0, 28.9.



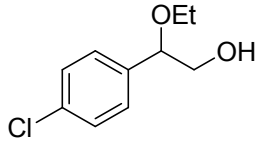
2-Cyclohexyloxy-2-phenylethanol. (3f)[3]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37-7.21 (m, 5H), 4.57-4.56 (m, 1H), 3.63-3.57 (m, 2H), 3.27-3.25 (m, 1H), 2.01-1.98 (m, 2H), 1.75-1.66 (m, 3H), 1.34-1.17 (m, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 139.9, 128.1, 127.8, 126.6, 79.5, 74.6, 67.6, 35.5, 31.6, 25.6, 24.2, 24.1, 24.0.



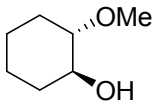
2-(4-Chlorophenyl)-2-methoxyethanol. (3g)[2]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.4 Hz, 2H), 7.27 (d, *J* = 8.4 Hz, 2H), 4.19 (dd, *J* = 6.4 Hz, 4.8 Hz, 1H), 3.52-3.40 (m, 2H), 3.20 (s, 3H), 2.42 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 135.9, 133.6, 128.4, 127.9, 83.3, 66.9, 55.6.



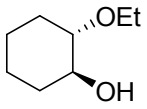
2-(4-Chlorophenyl)-2-ethoxyethanol. (3h)[2]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (d, *J* = 8.4 Hz, 2H), 7.29 (d, *J* = 8.4 Hz, 2H), 4.25 (dd, *J* = 11.5, 3.9 Hz, 1H), 3.63-3.32 (m, 4H), 2.59(s, 1H), 1.24 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.6, 133.8, 128.3, 127.9, 83.5, 67.2, 64.7, 15.5.



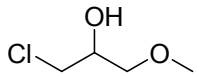
***Trans*-2-methoxycyclohexanol. (3i)[1]**

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.46-3.35 (m, 4H), 2.97-2.90 (m, 1H), 2.68 (s, 1H), 2.14-1.95 (m, 2H), 1.76-1.66 (m, 2H), 1.30-1.07 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 85.9, 73.5, 56.1, 31.9, 27.4, 21.3, 21.2.



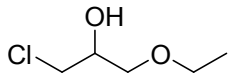
***Trans*-2-ethoxycyclohexanol. (3j)[2]**

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.73-3.62 (m, 1H), 3.45-3.34 (m, 2H), 3.03-2.96 (m, 1H), 2.71 (s, 1H), 2.12-1.93 (m, 2H), 1.77-1.60 (m, 2H), 1.46-1.09 (m, 7H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 83.5, 72.6, 64.1, 32.0, 29.1, 24.3, 24.1, 15.8.



1-Chloro-3-methoxypropan-2-ol. (3k)[4]

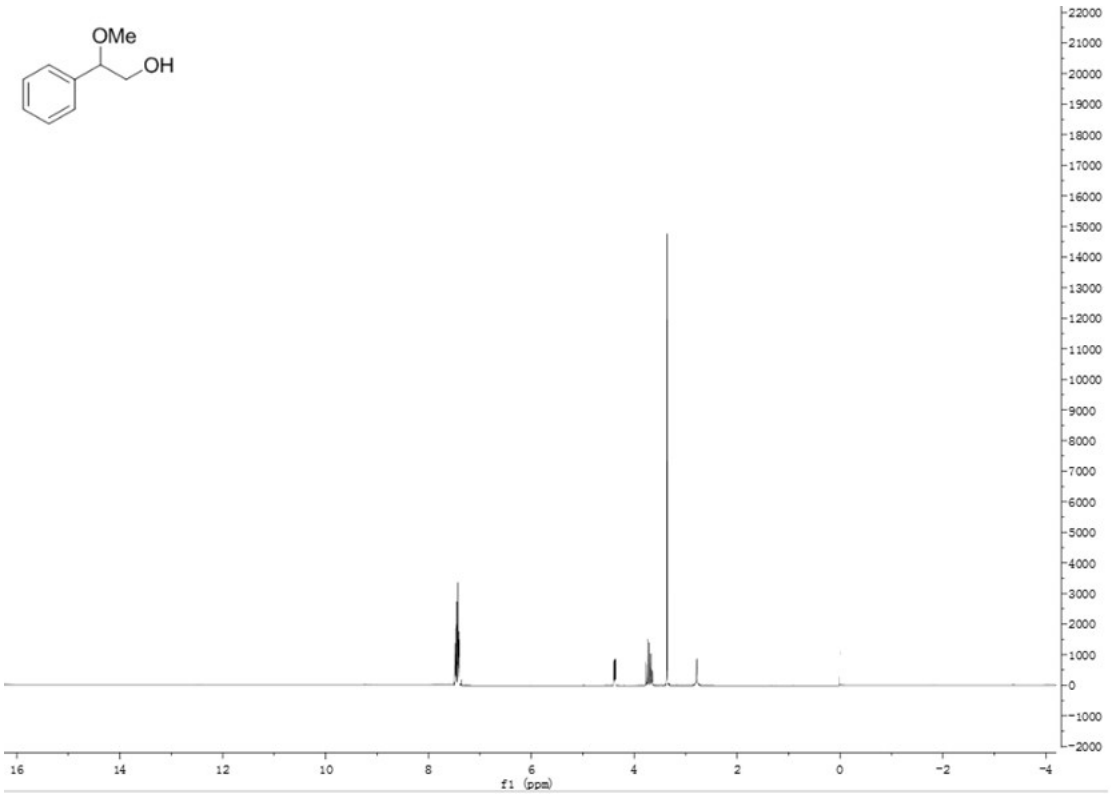
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.98-3.96 (m, 1H), 3.75-3.68 (m, 2H), 3.52-36 (m, 2H), 3.22-3.19 (s, 3H), 2.54 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 73.4, 70.5, 59.6, 46.3.



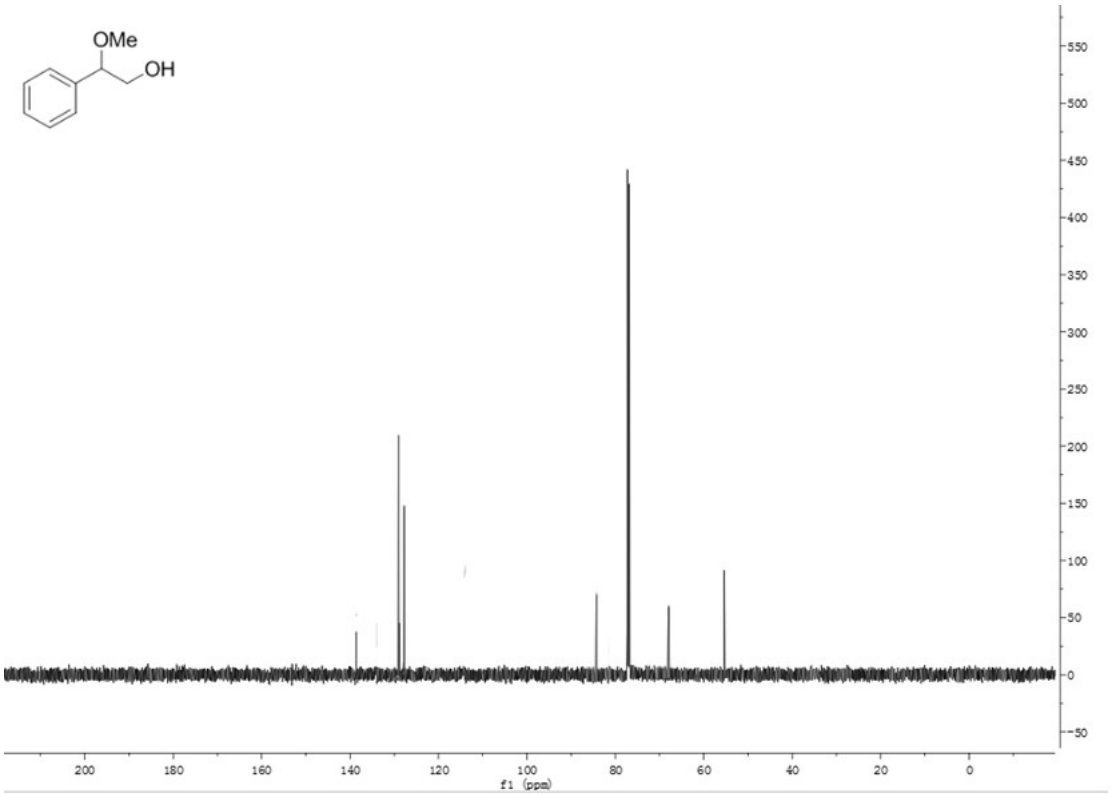
1-Chloro-3-ethoxypropan-2-ol. (3l)[2]

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.82-3.79 (m, 1H), 3.62-3.36 (m, 6H), 2.09 (s, 1H), 1.02 (t, J = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 71.2, 69.5, 63.9, 45.7, 14.7.

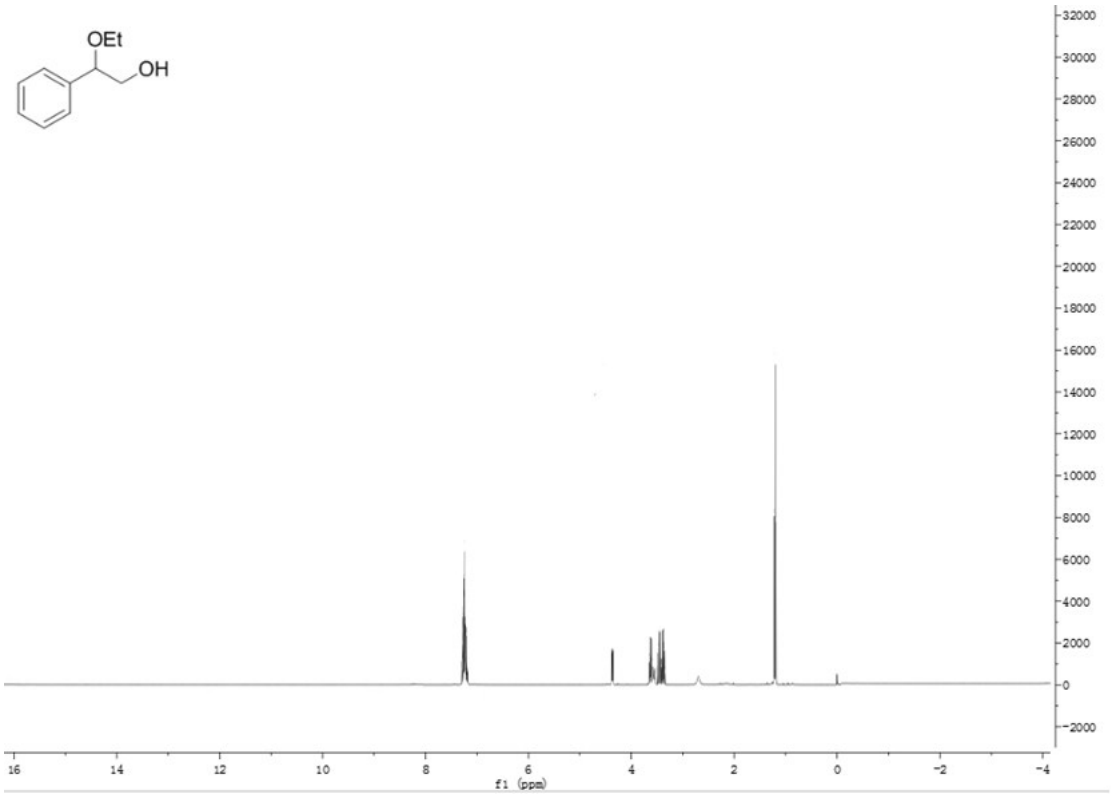
Copies of ^{1}H NMR and ^{13}C NMR spectra of compounds



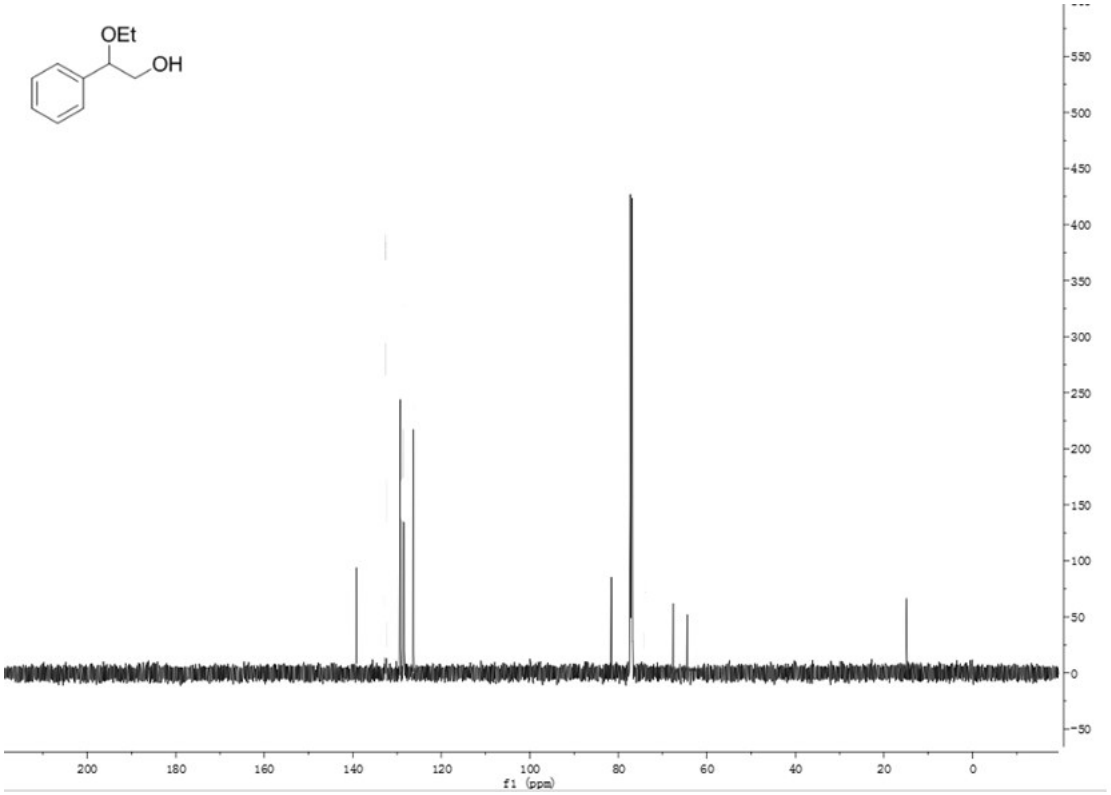
The ^{1}H NMR spectrum of 2-methoxy-2-phenylethanol (3a).



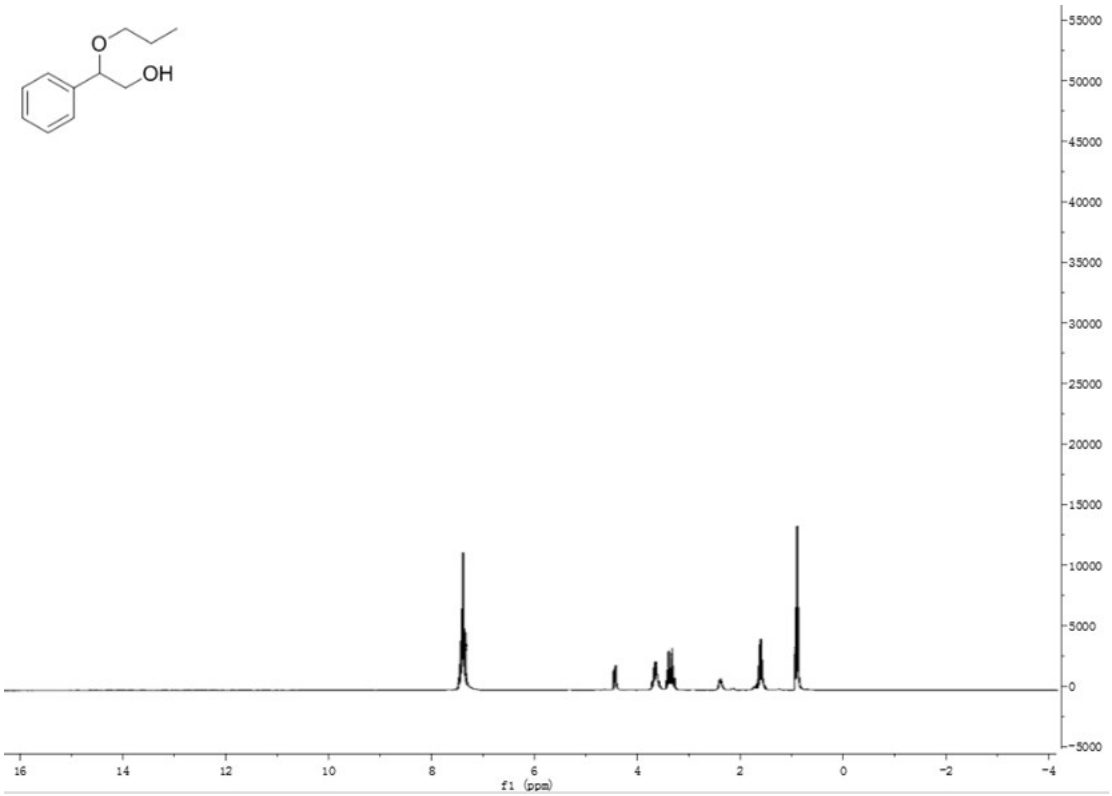
The ^{13}C NMR spectrum of 2-methoxy-2-phenylethanol (3a).



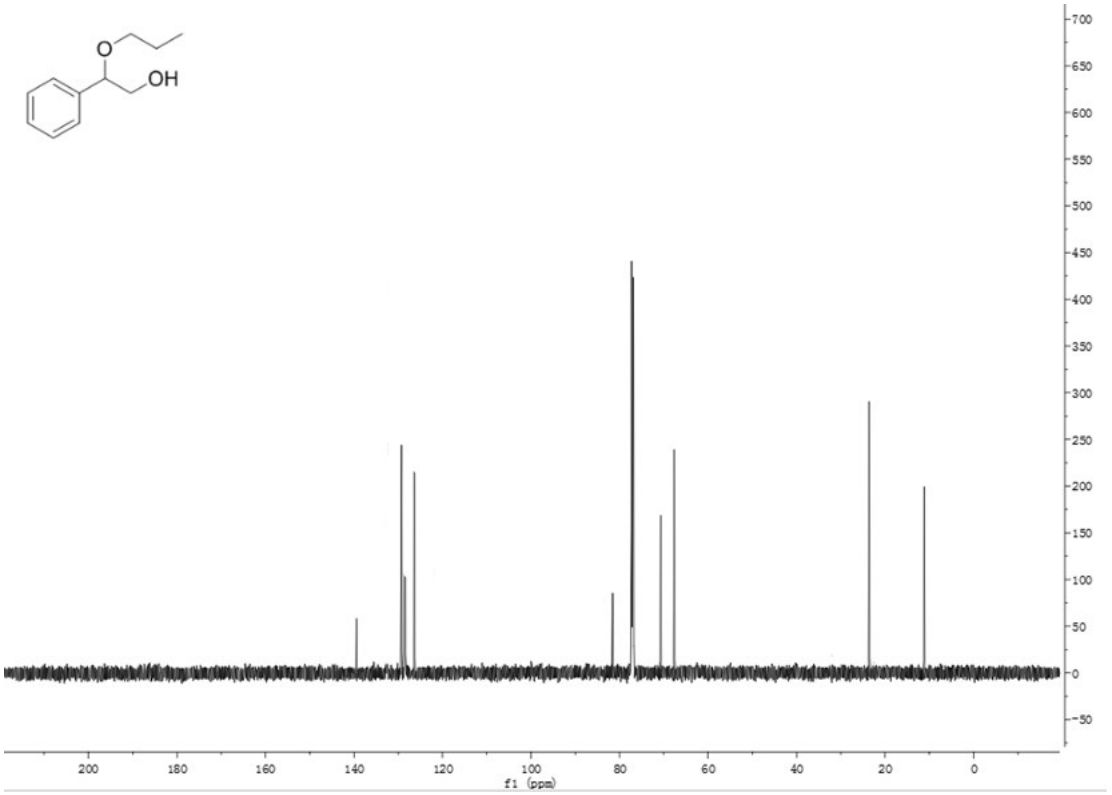
The ^{1}H NMR spectrum of 2-ethoxy-2-phenylethanol (3b).



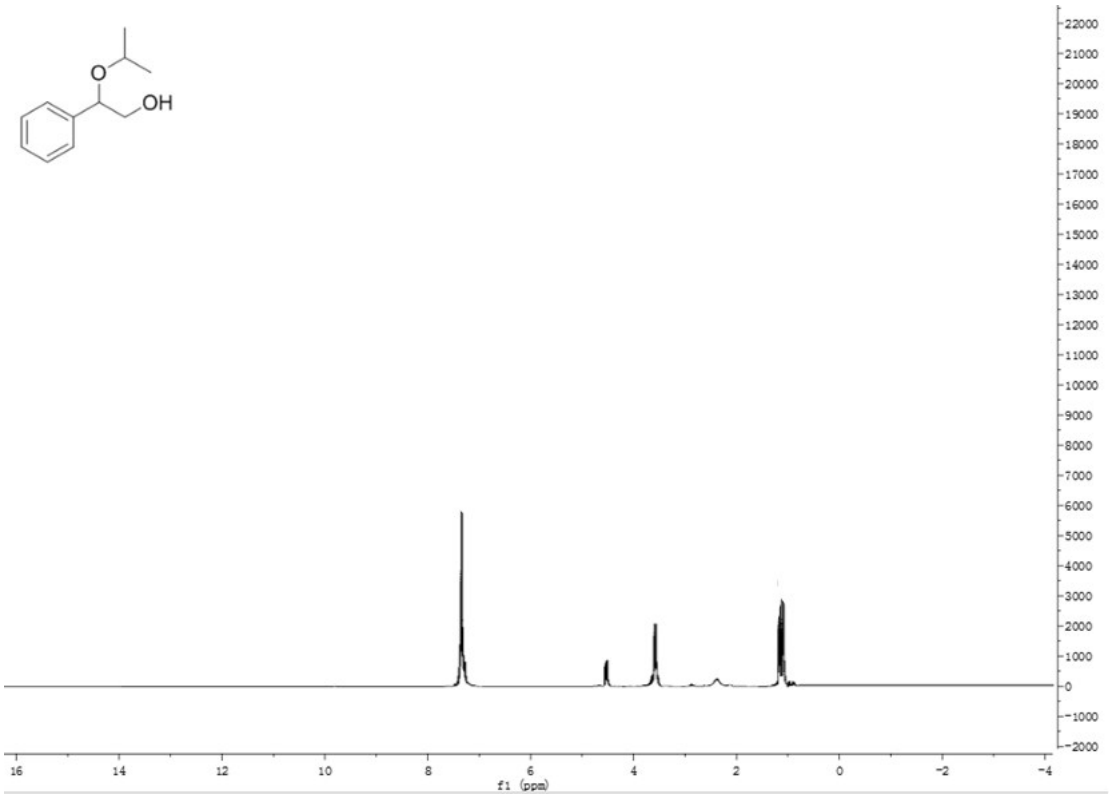
The ^{13}C NMR spectrum of 2-ethoxy-2-phenylethanol (3b).



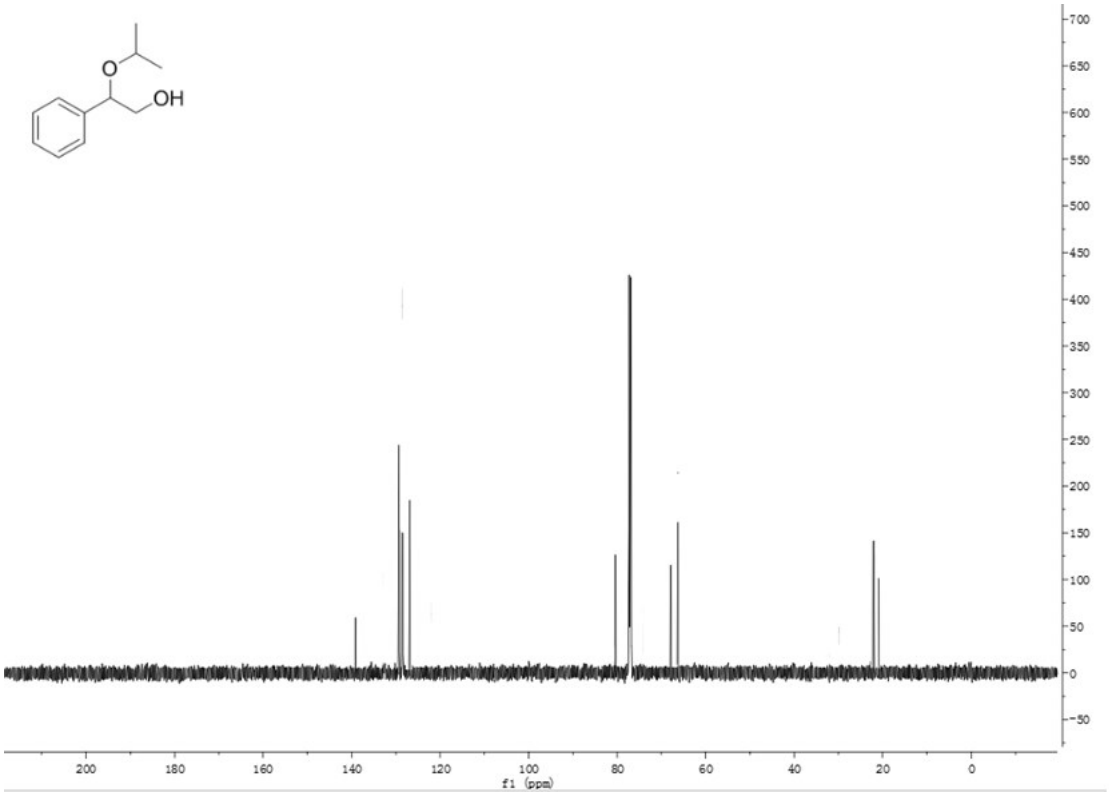
The ^{1}H NMR spectrum of 2-phenyl-2-propoxyethanol (3c).



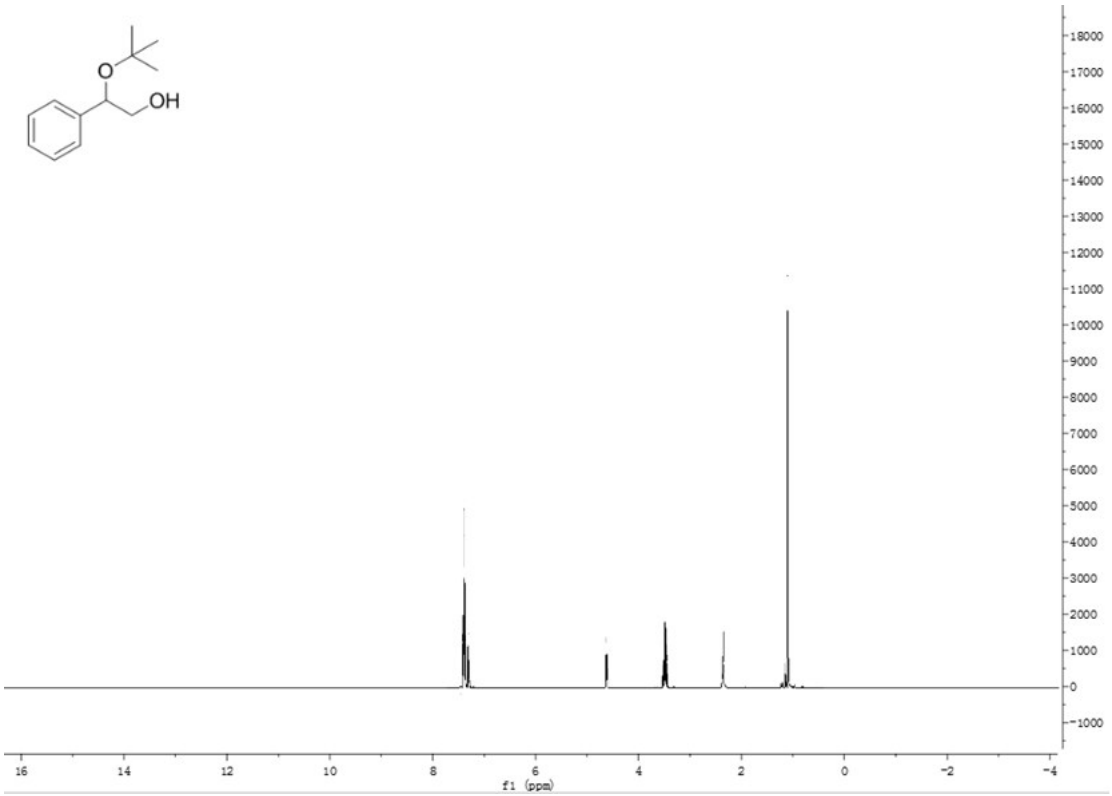
The ^{13}C NMR spectrum of 2-phenyl-2-propoxyethanol (3c).



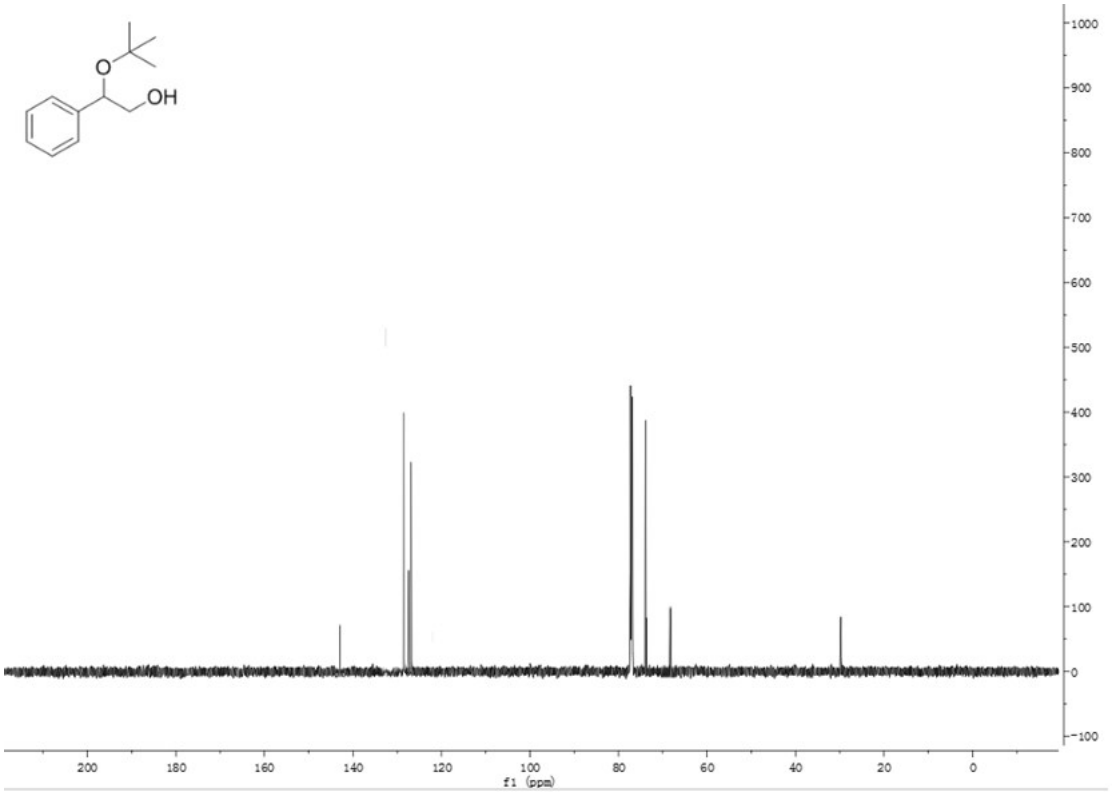
The ^{1}H NMR spectrum of 2-iso-propoxy-2-phenylethanol (3d).



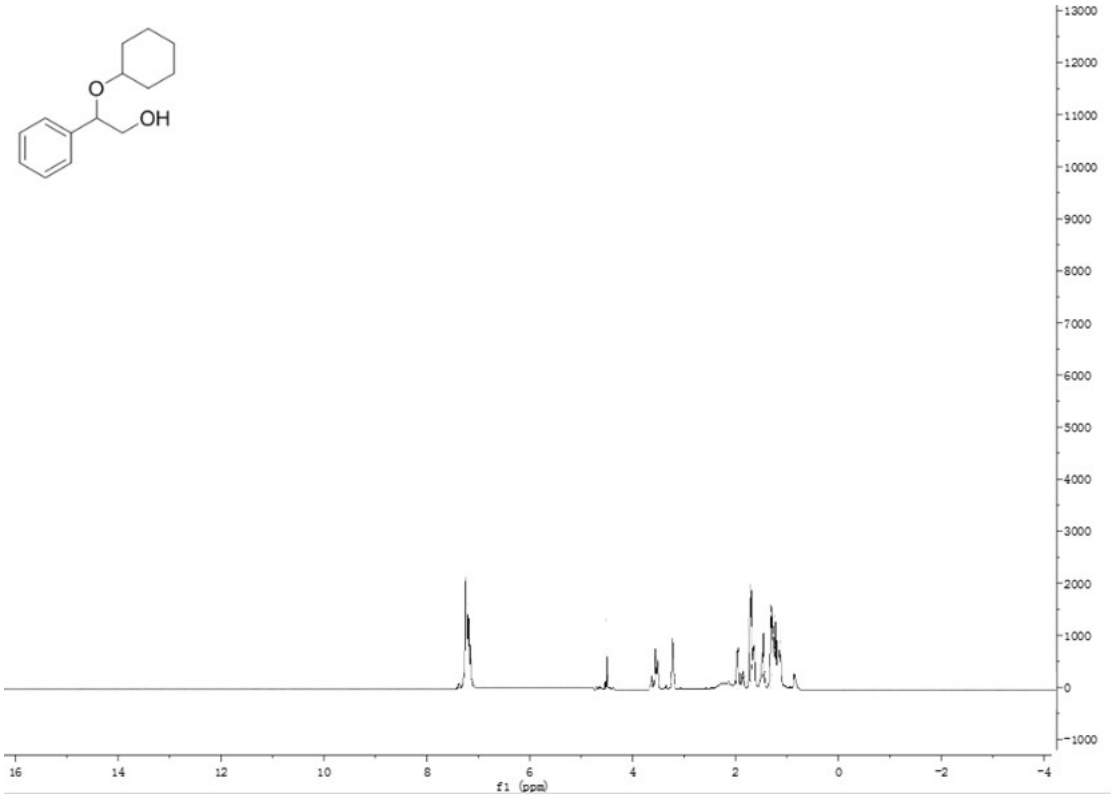
The ^{13}C NMR spectrum of 2-iso-propoxy-2-phenylethanol (3d).



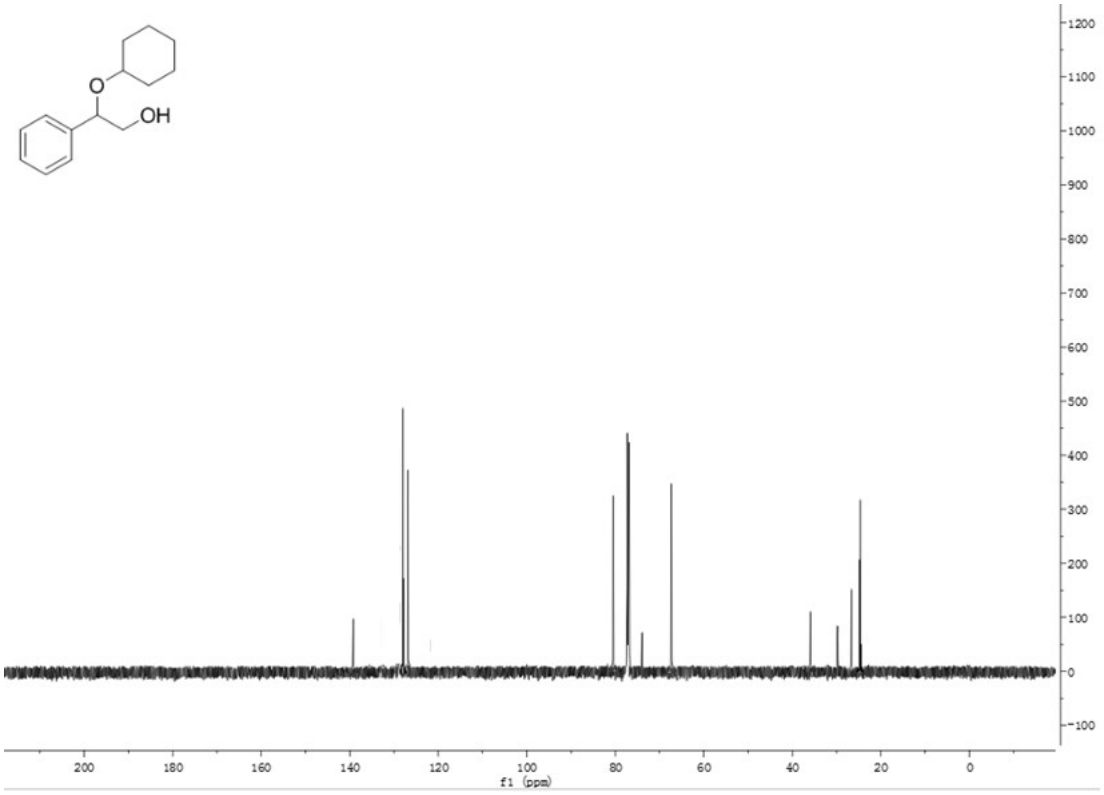
The ^{1}H NMR spectrum of 2-tert-butoxy-2-phenylethanol (3e).



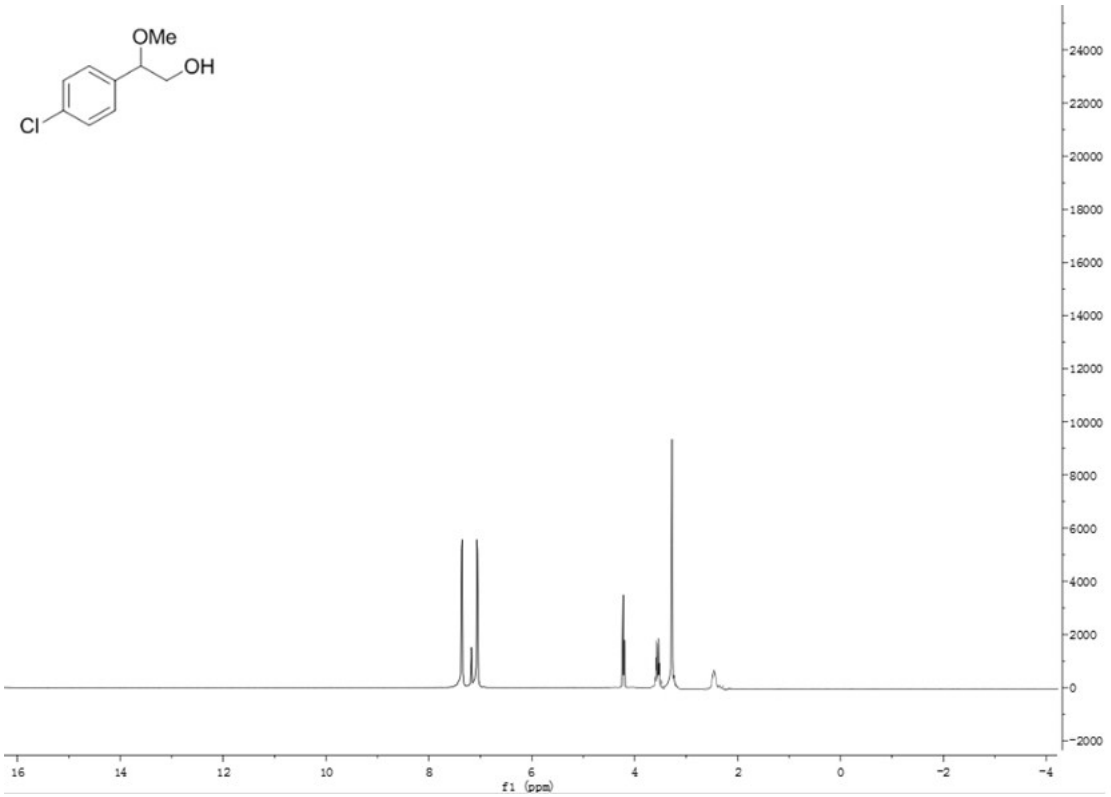
The ^{13}C NMR spectrum of 2-tert-butoxy-2-phenylethanol (3e).



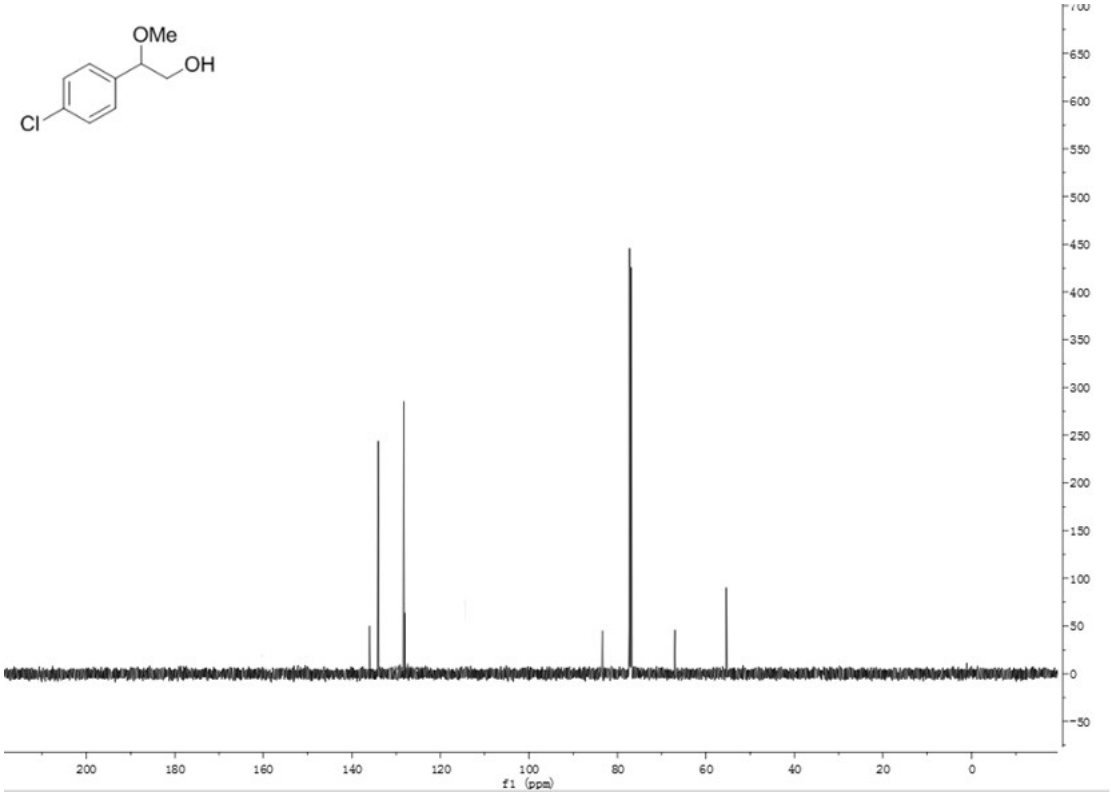
The ^{1}H NMR spectrum of 2-cyclohexyloxy-2-phenylethanol (3f).



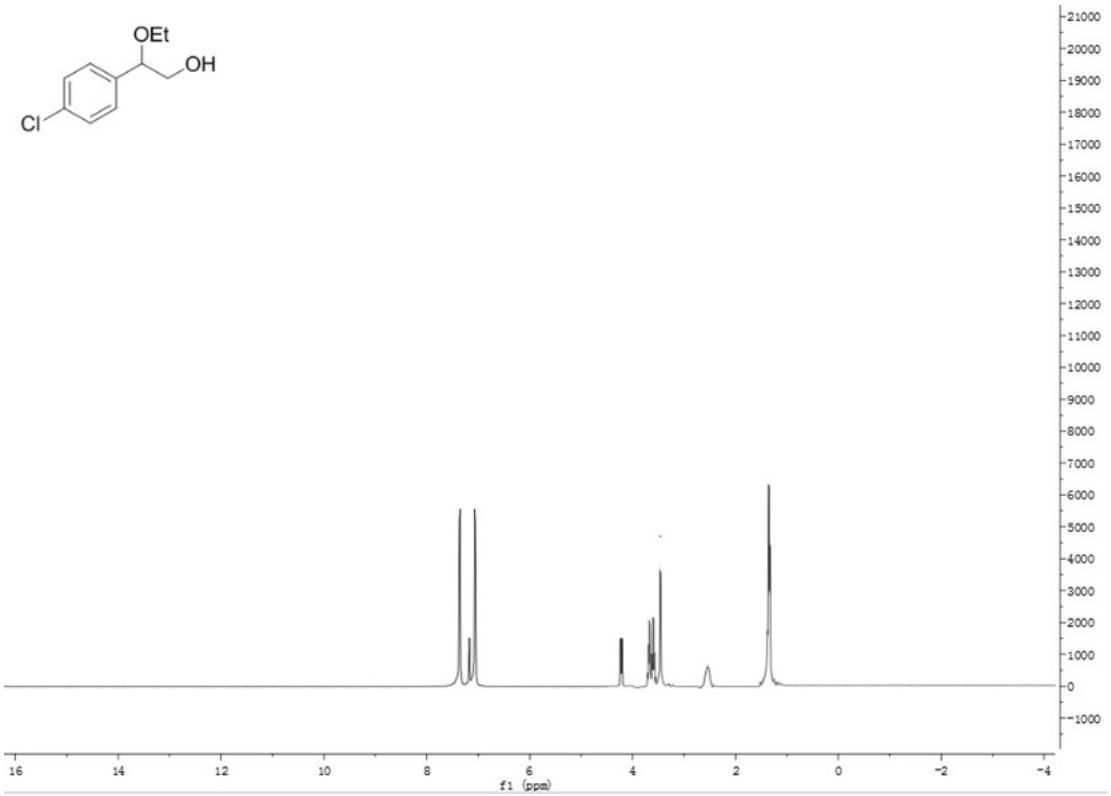
The ^{13}C NMR spectrum of 2-cyclohexyloxy-2-phenylethanol (3f).



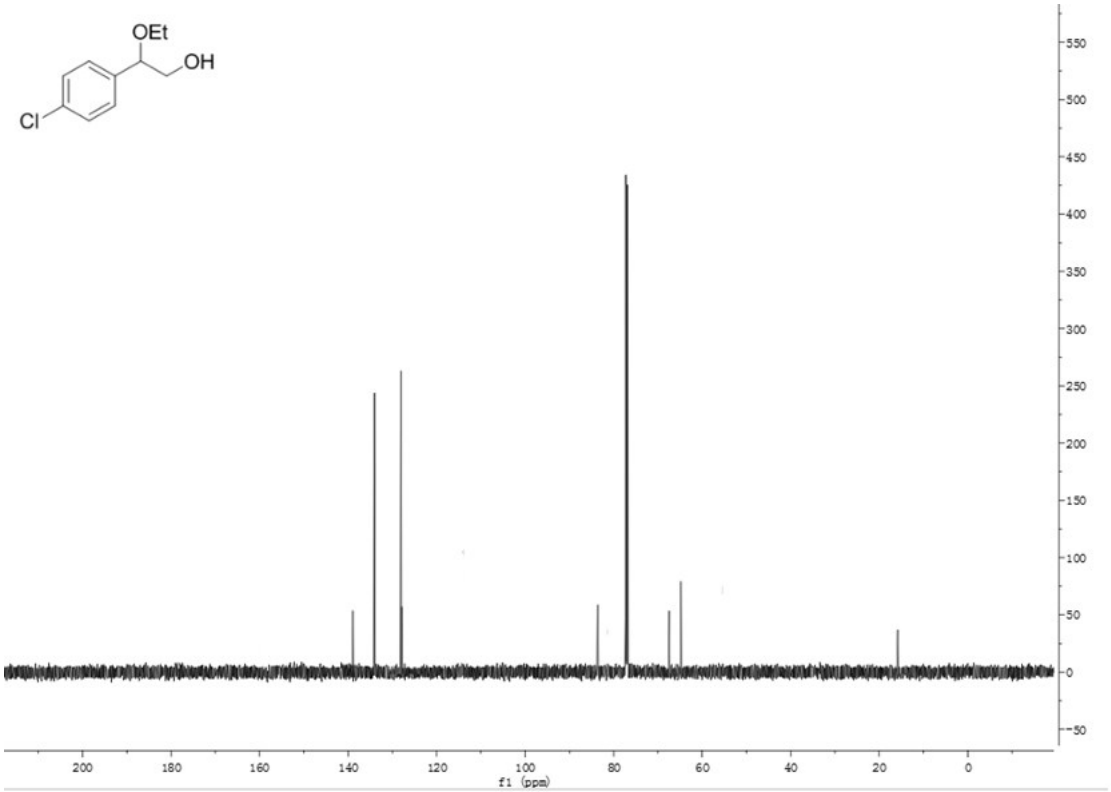
The ^{1}H NMR spectrum of 2-(4-chlorophenyl)-2-methoxyethanol (3g).



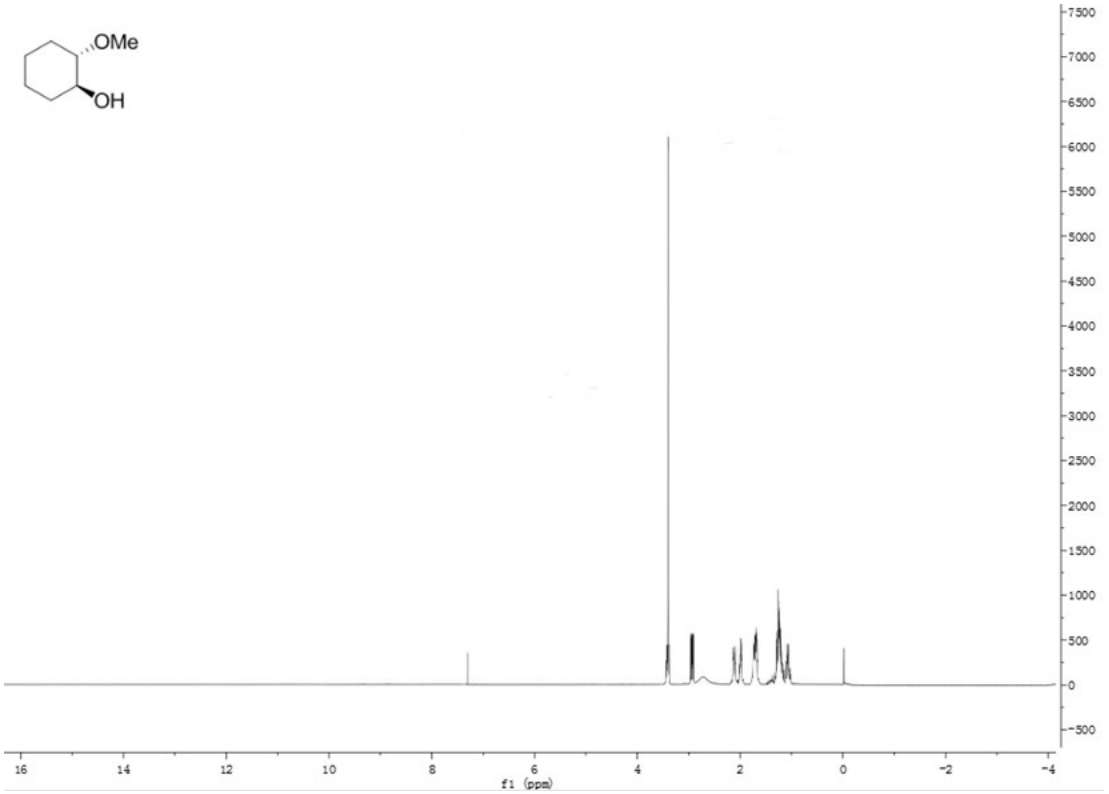
The ^{13}C NMR spectrum of 2-(4-chlorophenyl)-2-methoxyethanol (3g).



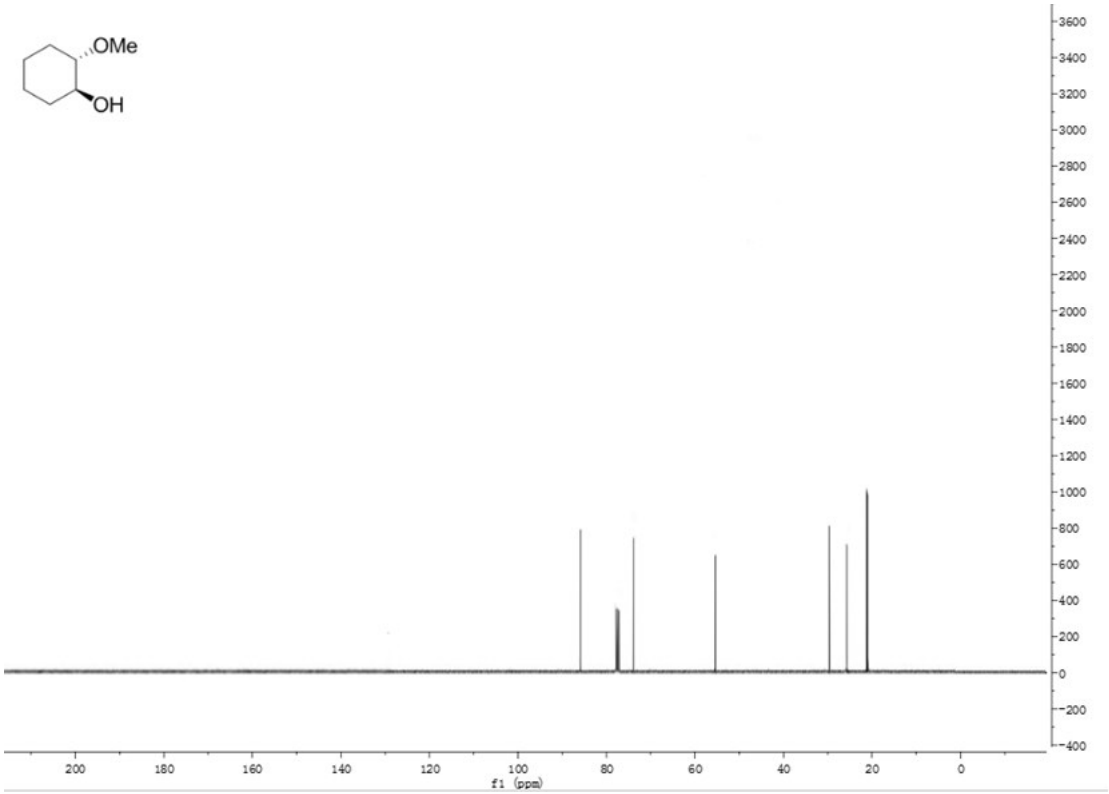
The ^{1}H NMR spectrum of 2-(4-chlorophenyl)-2-ethoxyethanol (3h).



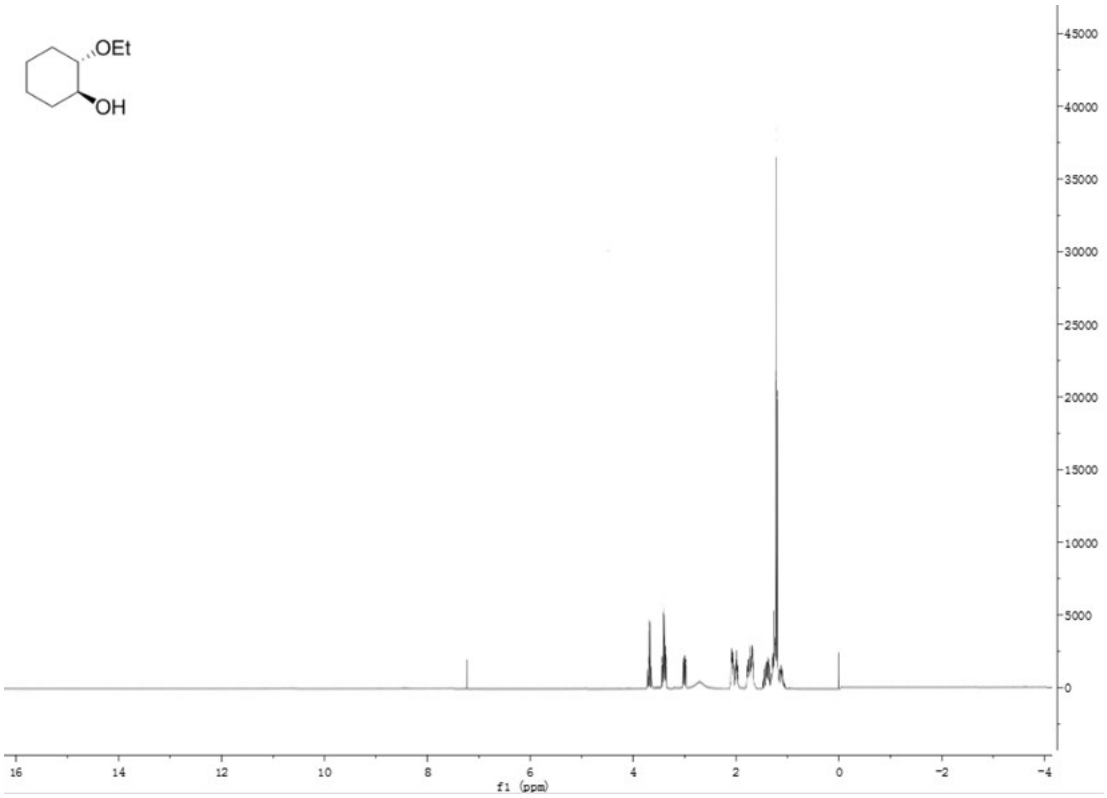
The ^{13}C NMR spectrum of 2-(4-chlorophenyl)-2-ethoxyethanol (3h).



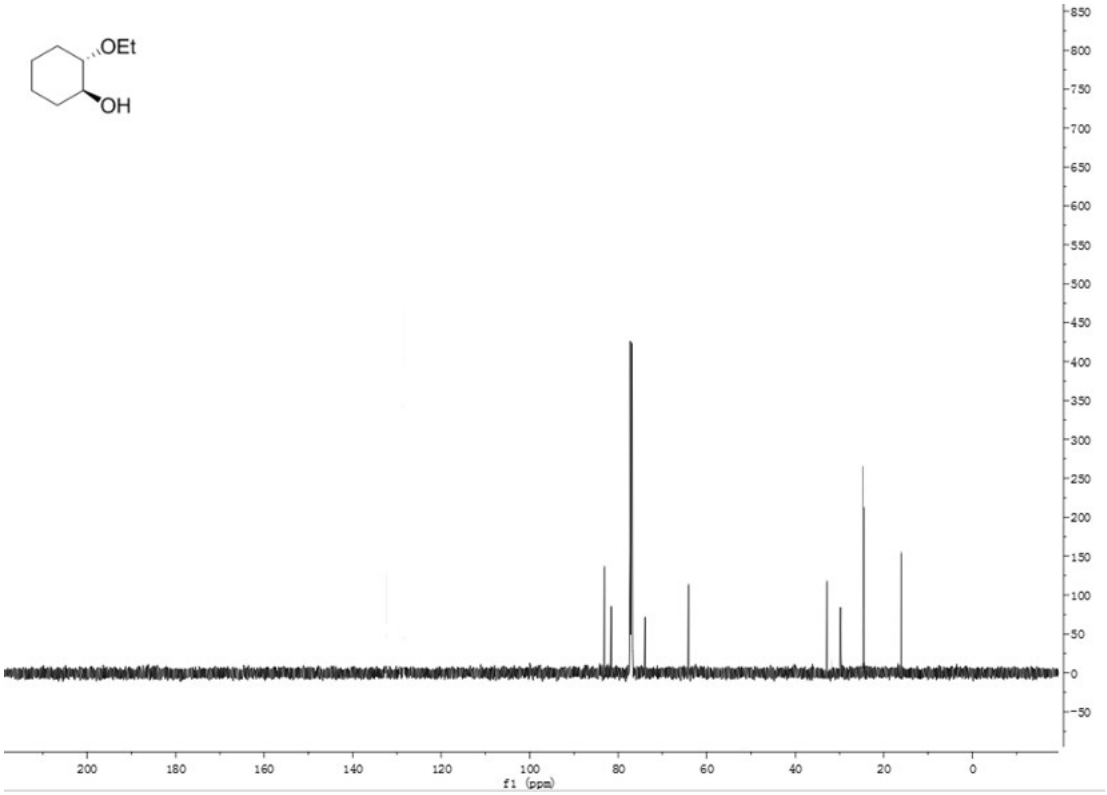
The ^{1}H NMR spectrum of *trans*-2-methoxycyclohexanol (3i).



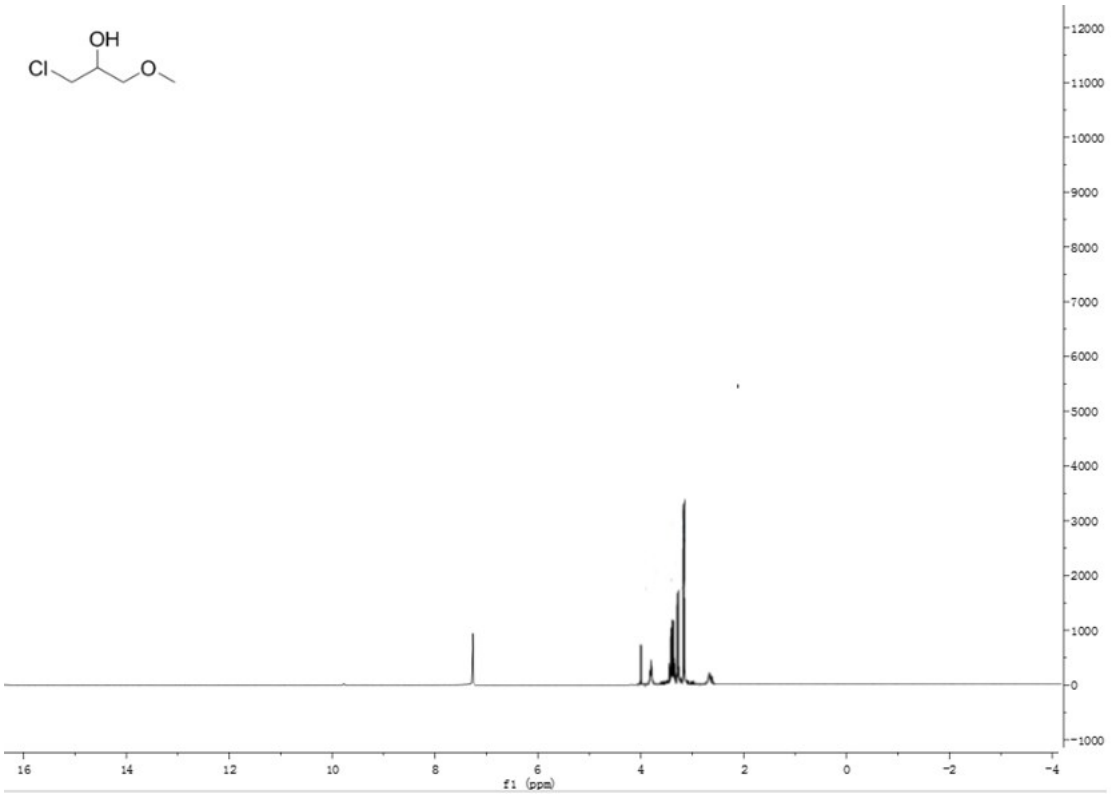
The ^{13}C NMR spectrum of *trans*-2-methoxycyclohexanol (3i).



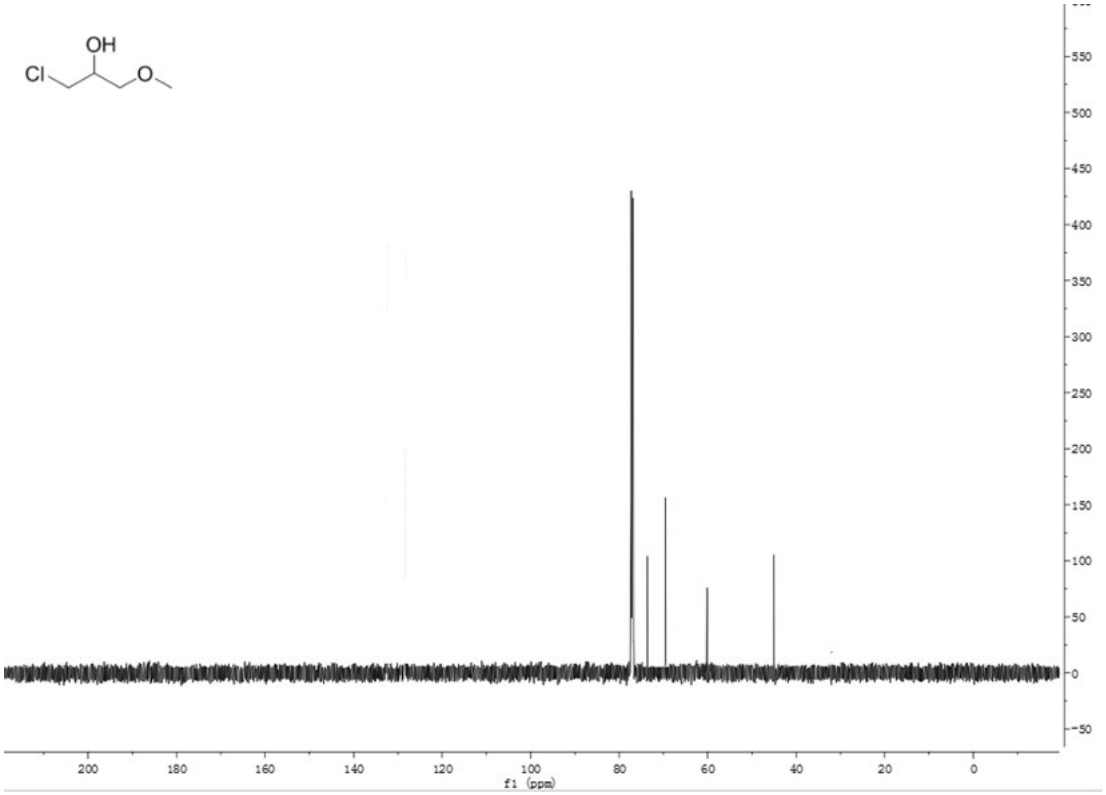
The ^{1}H NMR spectrum of *trans*-2-ethoxycyclohexanol (3j).



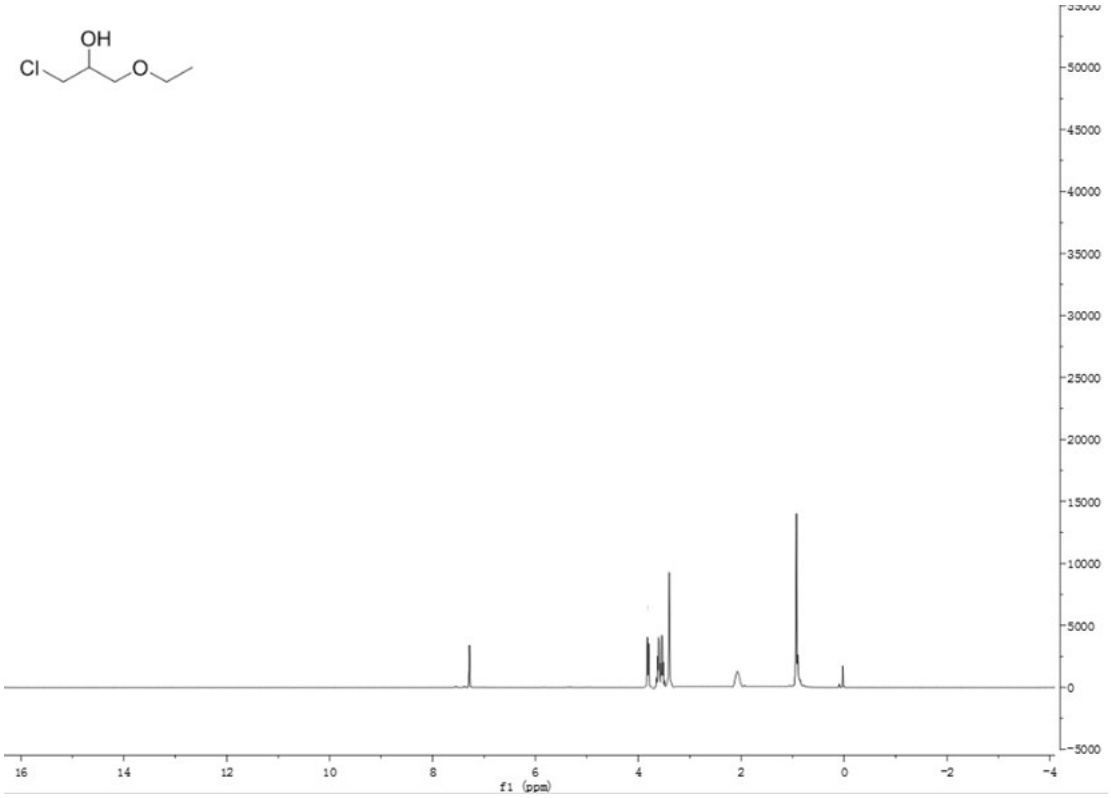
The ^{13}C NMR spectrum of *trans*-2-ethoxycyclohexanol (3j).



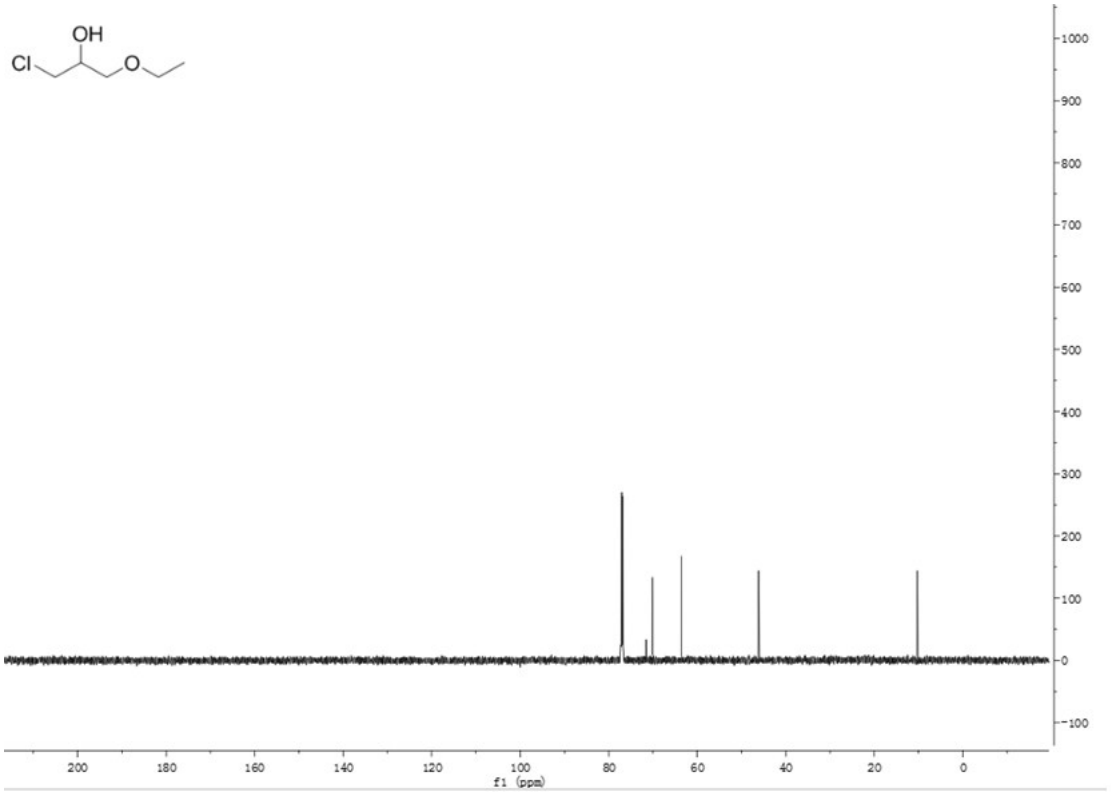
The ^{1}H NMR spectrum of 1-chloro-3-methoxypropan-2-ol (3k).



The ^{13}C NMR spectrum of 1-chloro-3-methoxypropan-2-ol (3k).



The ^{1}H NMR spectrum of 1-chloro-3-ethoxypropan-2-ol (3l).



The ^{13}C NMR spectrum of 1-chloro-3-ethoxypropan-2-ol (3l).

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