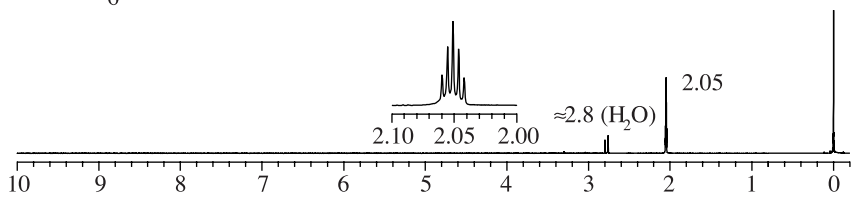
5.14 Spectra of Solvents and Reference Compounds

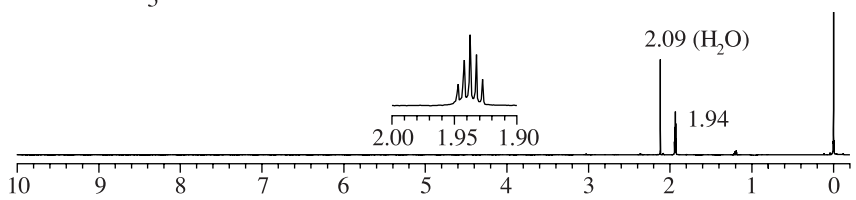
5.14.1 ^{1}H NMR Spectra of Common Deuterated Solvents

500 MHz; ≈1 000 data points per 1 ppm; δ in ppm relative to TMS

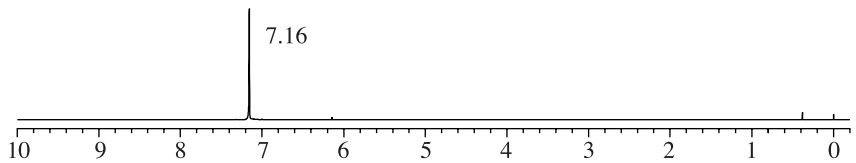
Acetone-d_6



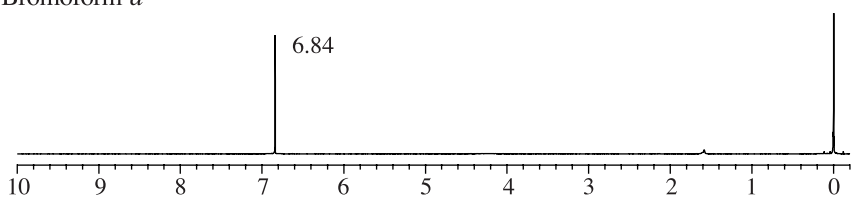
Acetonitrile-d_3



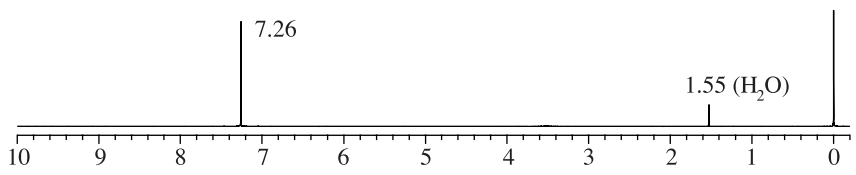
Benzene-d_6



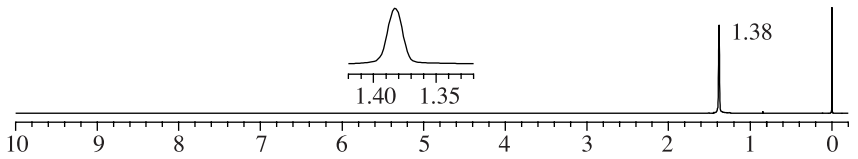
Bromoform-*d*



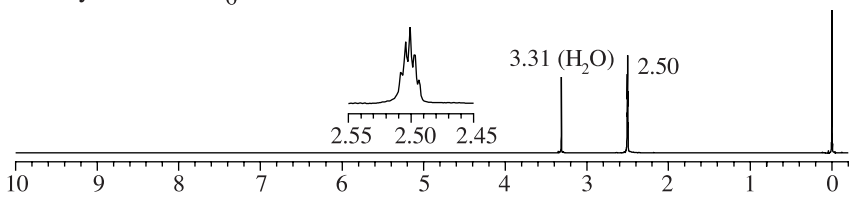
Chloroform-*d*



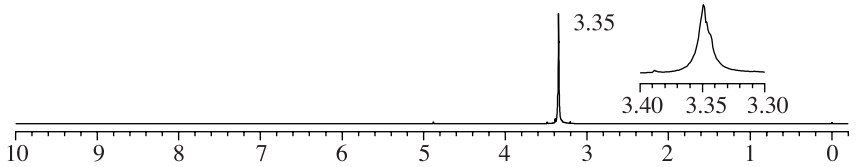
Cyclohexane-d_{12}



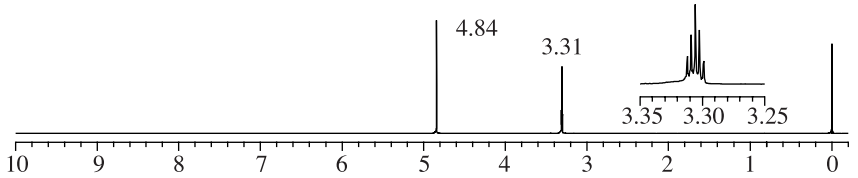
Dimethyl sulfoxide-d_6



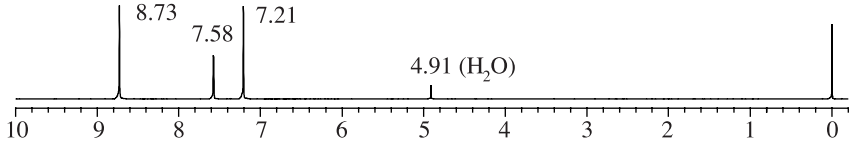
Methanol-d_1



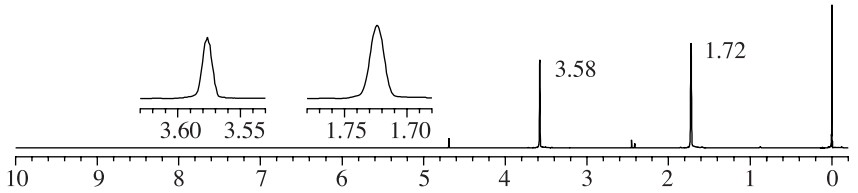
Methanol-d_4



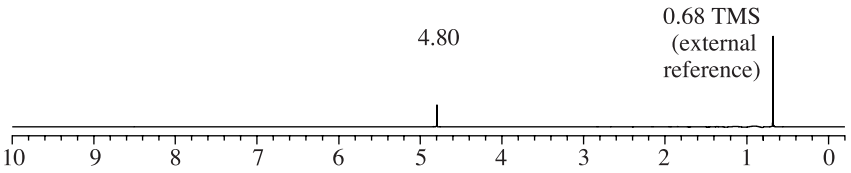
Pyridine-d_5



Tetrahydrofuran-d_8



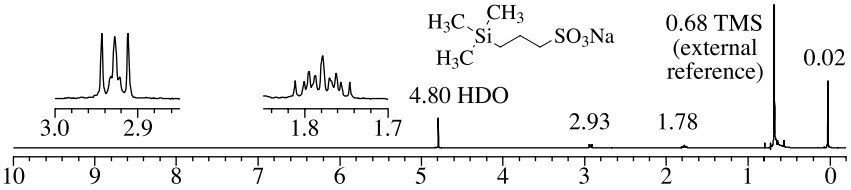
Water-d_2



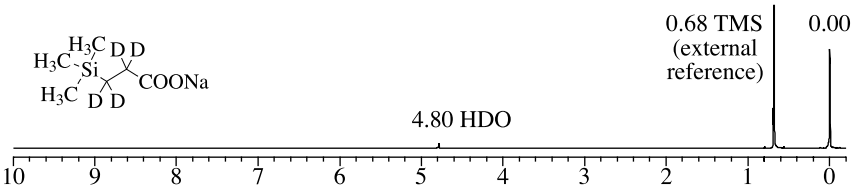
5.14.2 ^{1}H NMR Spectra of Secondary Reference Compounds

Chemical shifts in ^{1}H NMR spectra are usually reported relative to the peak position of tetramethylsilane (TMS) added to the sample as an internal reference. If TMS is not sufficiently soluble, a capillary with TMS may be used as external reference. In this case, owing to the different volume susceptibilities, the local magnetic fields in the sample and reference differ, and the peak position of the reference must be corrected. For a D_2O solution in a cylindrical sample and neat TMS in a capillary, the correction amounts to +0.68 and -0.34 ppm for superconducting and electromagnets, respectively. These values must be subtracted from the chemical shifts relative to the external TMS signal if its position is set to 0.00 ppm. Alternatively, secondary references with $(CH_3)_3SiCH_2$ groups may be used. The following spectra of two such secondary reference compounds in D_2O were measured at 500 MHz with TMS as external reference. Chemical shifts are reported in ppm relative to TMS upon correction for the difference in the volume susceptibilities of D_2O and TMS. As a result, the peak for the external TMS appears at 0.68 ppm.

3-(Trimethylsilyl)-1-propanesulfonic acid sodium salt (sodium 4,4-dimethyl-4-silapentane-1-sulfonate; DSS)



2,2,3,3-D_4-3-(Trimethylsilyl)propionic acid sodium salt



5.14.3 ^{1}H NMR Spectrum of a Mixture of Common Nondeuterated Solvents

The following ^{1}H NMR spectrum (500 MHz, δ in ppm relative to TMS) of $CDCl_3$ containing 18 common solvents (0.05–0.4 vol%) is shown as a guide for the identification of possible impurities. Where the signals of several solvents overlap, insets show signals for the individual compounds from separate spectra. Peaks in these insets are labeled with the corresponding chemical shifts from their main spectrum but their values may differ by up to 0.03 ppm. Signals that are particularly prone to vary in their position are marked with *. THF: tetrahydrofuran; EGDME: ethylene glycol dimethyl ether.