1D NMR Common Acquisition Concepts and Problems

(1) Acquisition Parameters and Sampling the FID

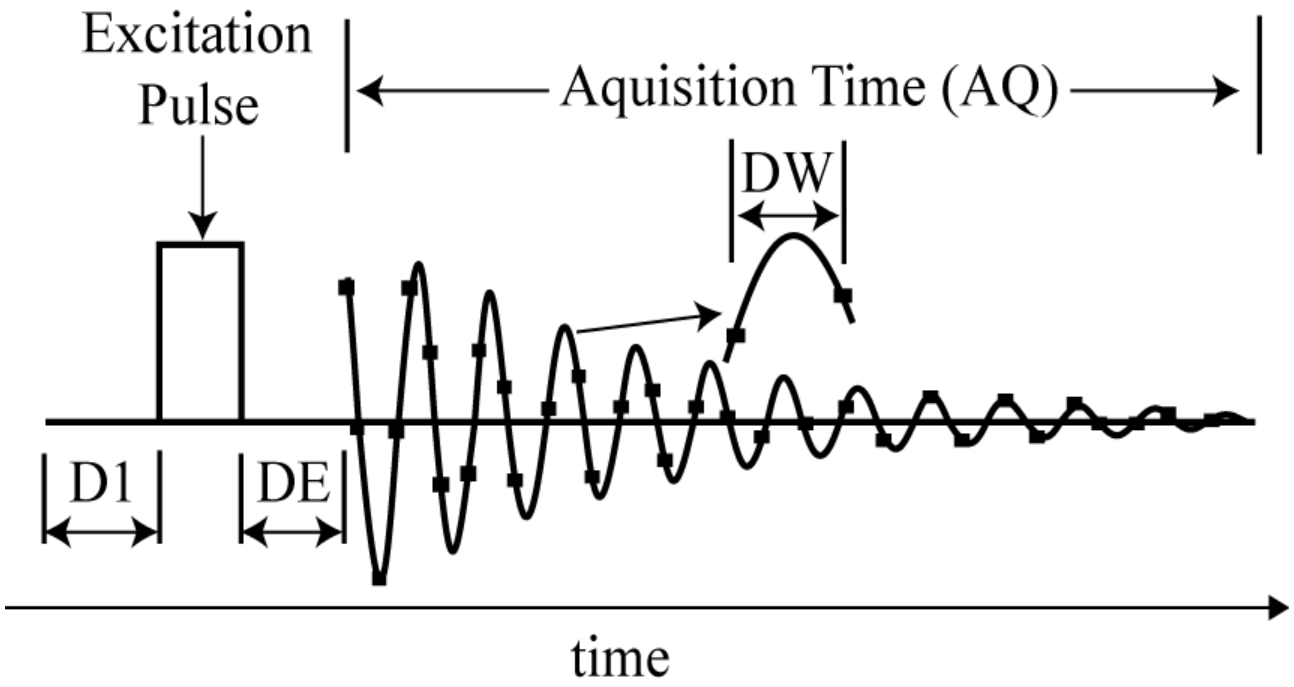
Acquisition Time (AQ) = NumPoints (TD) * DwellTime (DW)

DwellTime (DW) = 1 / SpectralWidth (SW)

Digital Resolutions (FIDRES) = 1 / [Acquisition Time (AQ)]

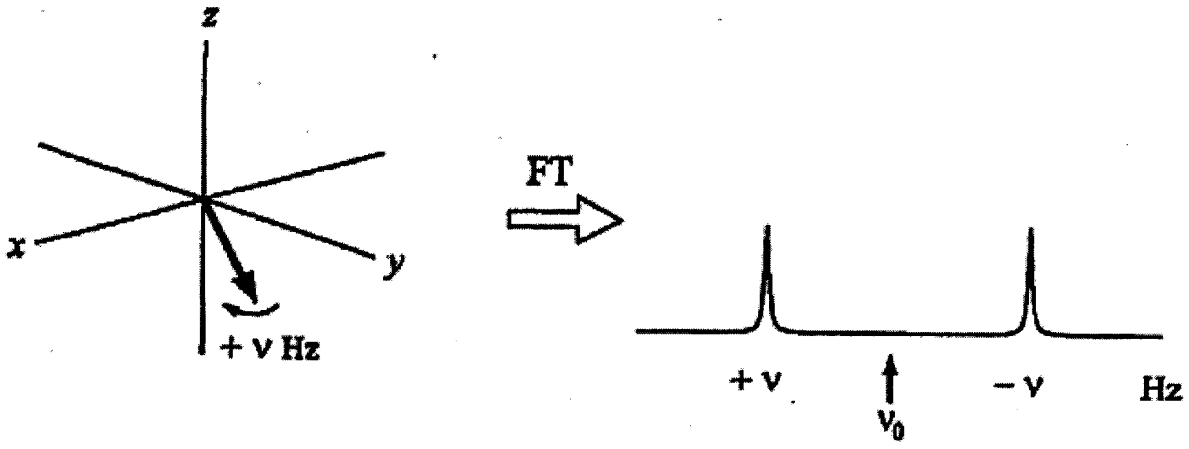
D1 = Recycle Delay DE = Pre-scan delay

NS = Number or scans DS = Number of dummy scans

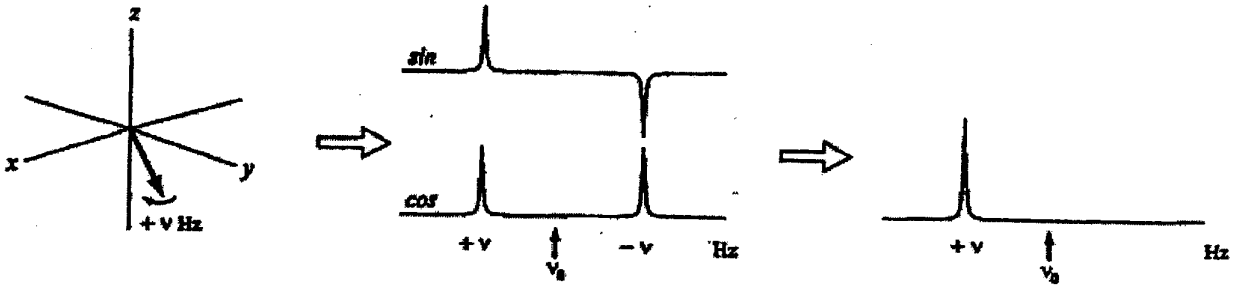


(2) Quadrature Detection

The spectrum corresponding to a positive precessing magnetization using single channel detection



Detection of a positive rotating magnetization using a two-channel scheme.



(3) Phasing

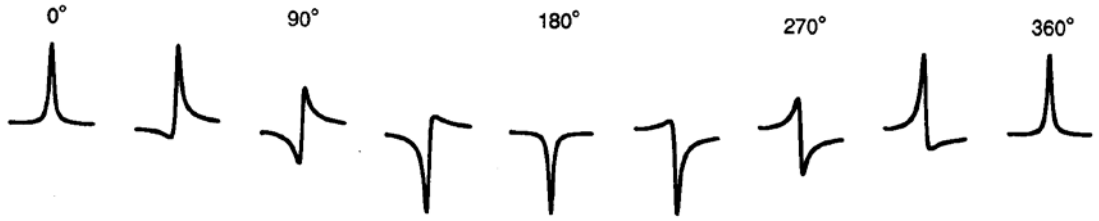
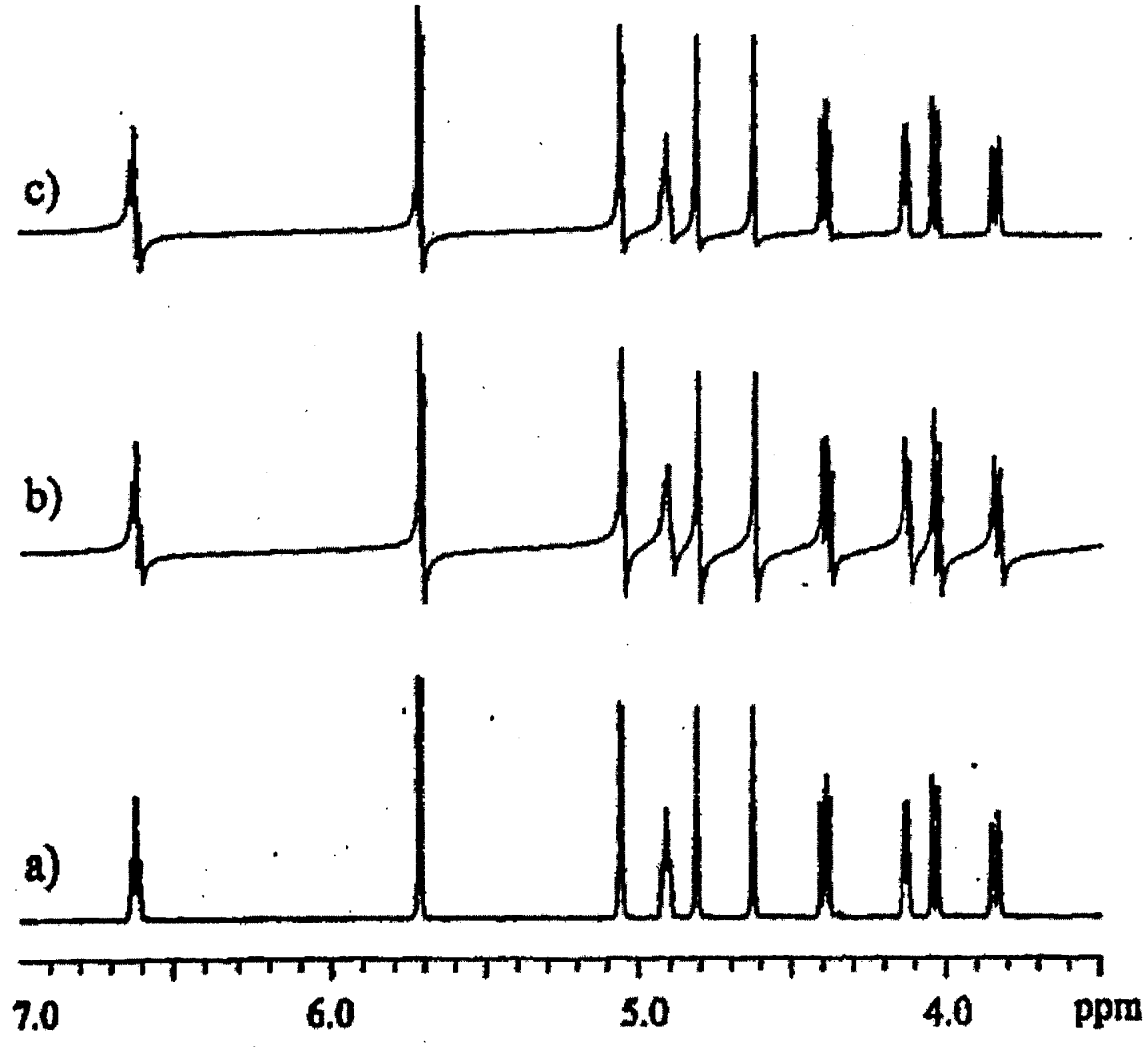
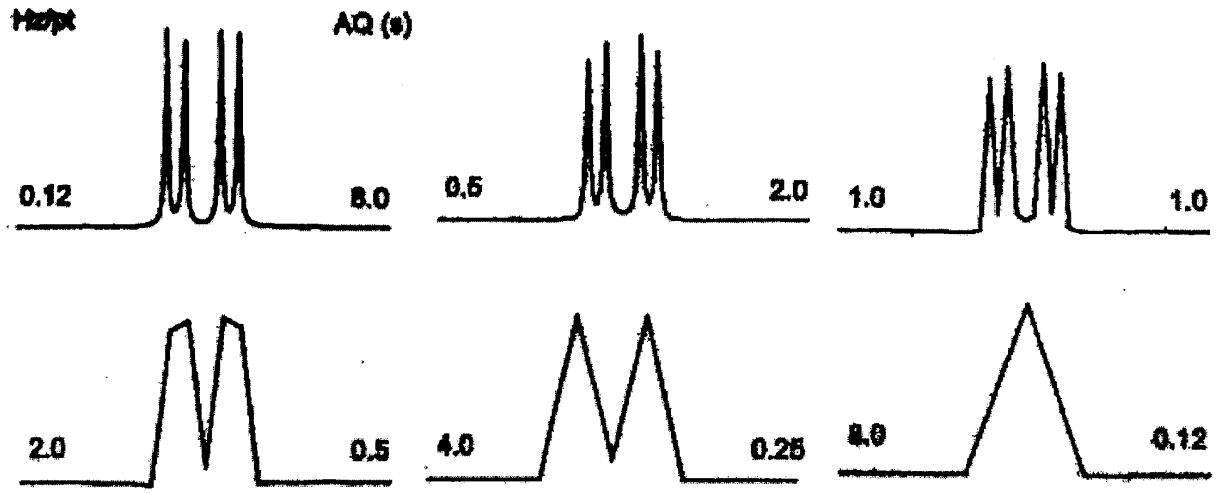


Fig. 1.15. The effect of a change in phase on the shape of a resonance signal; each step in phase is 45°.

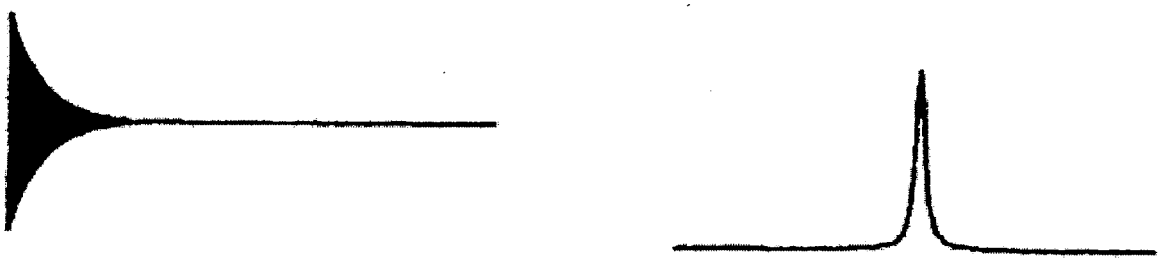
Phase Correcting a Spectrum, a) the initial spectrum, b) after zero-order correction, c) after first-order correction



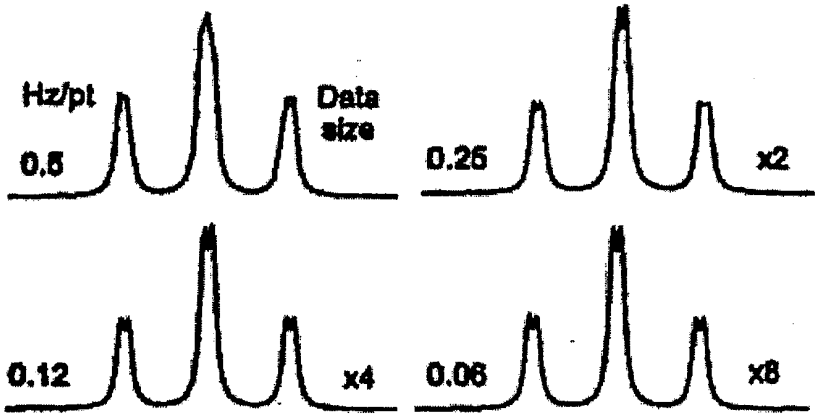
(4) Digital resolution



(5) Zero filling the FID



Zero filling may reveal fine structure that would otherwise be overlooked



(6) Exponential Multiplication

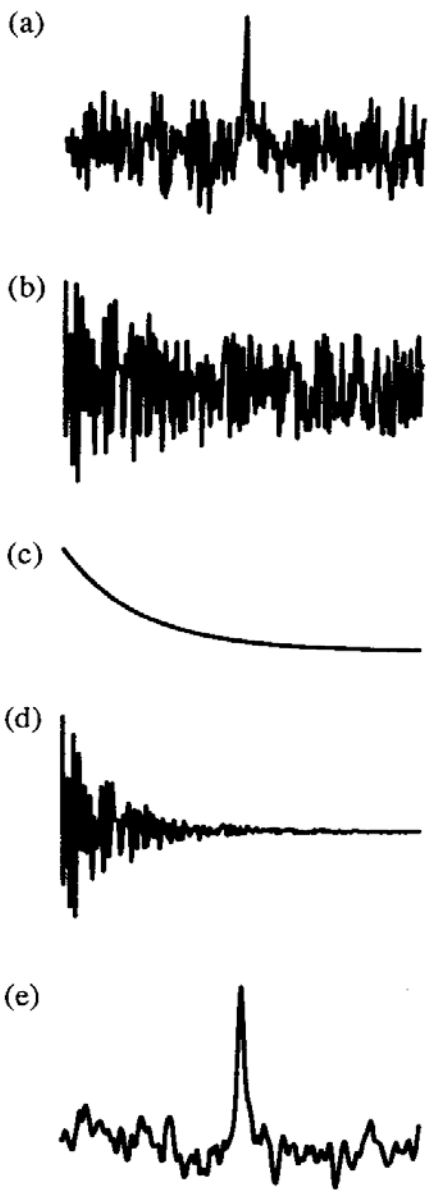


Fig. 1.23. Computer simulation of the effect of exponential multiplication: (a) signal from Fourier transformation of (b); (b) the 'untreated' FID; (c) the exponential decay function; (d) the FID after exponential multiplication; (e) signal from Fourier transformation of (d).

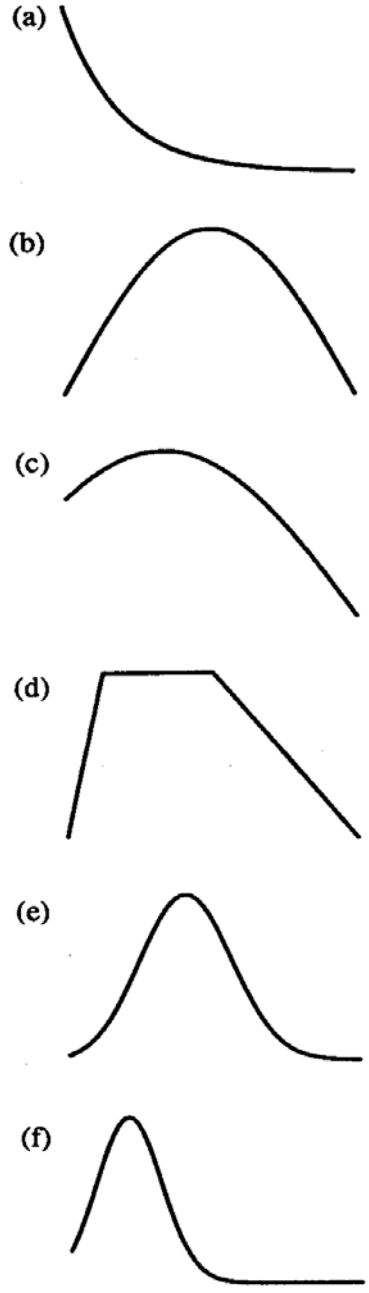
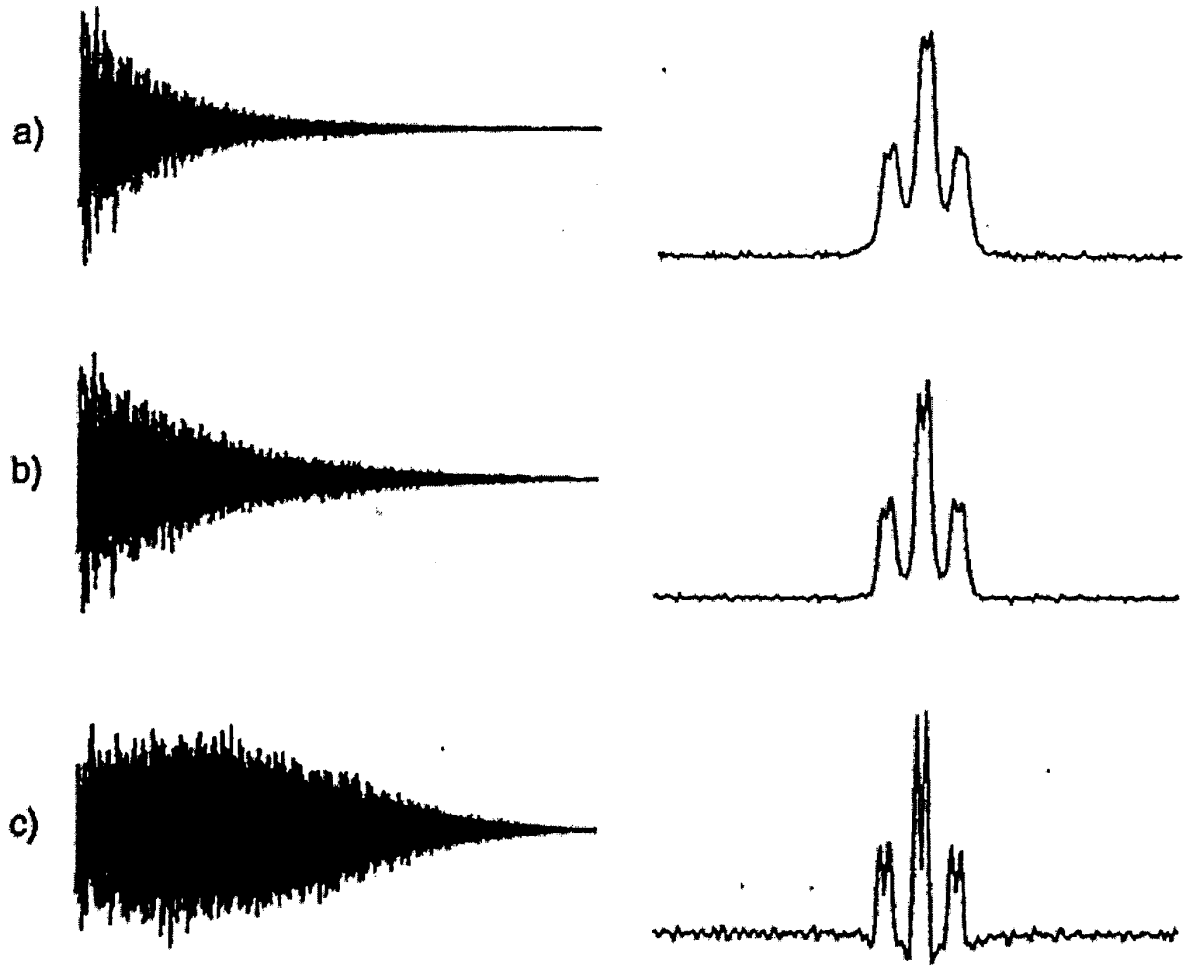


Fig. 1.24. Selected apodization window functions: (a) exponential multiplication; (b) sine-bell; (c) phase-shifted sine-bell; (d) trapezoidal multiplication; (e) Gaussian multiplication; (f) Gaussian multiplication with different parameters.

(7) Lorentz-Gauss Transformation

a) Unadulterated FID b) Exponential Multiplication of FID
c) Lorentz-Gauss Transformation of FID



(8) Folding

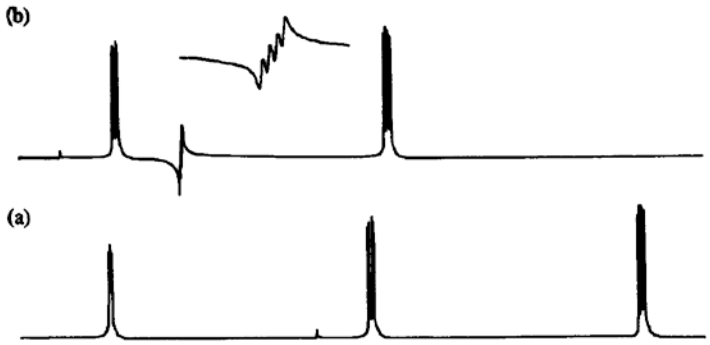
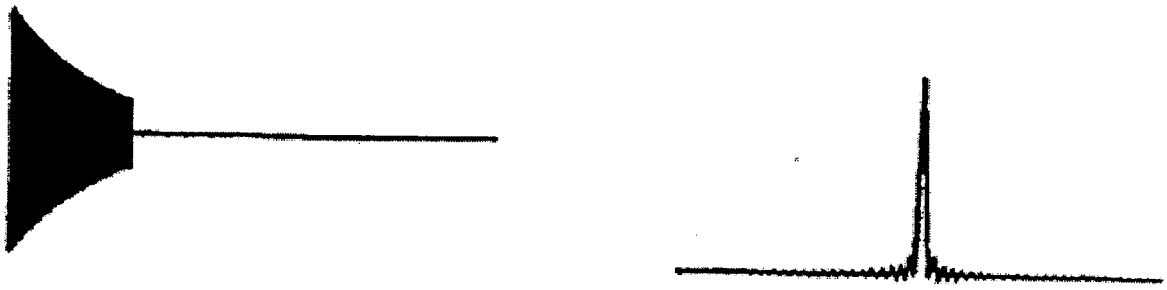


Fig. 1.19. ^1H spectra of furoic acid-d_1, **1.1**. (a) Acquired with the spectral width and reference frequency set correctly; (b) acquired with the reference incorrectly set. The inset shows the 'folded' multiplet.

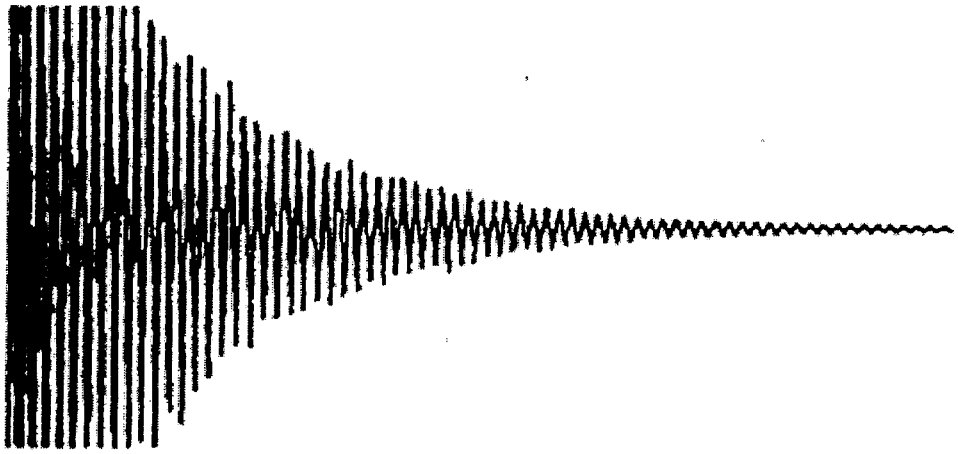
(9) Truncation

Truncation with zero filling

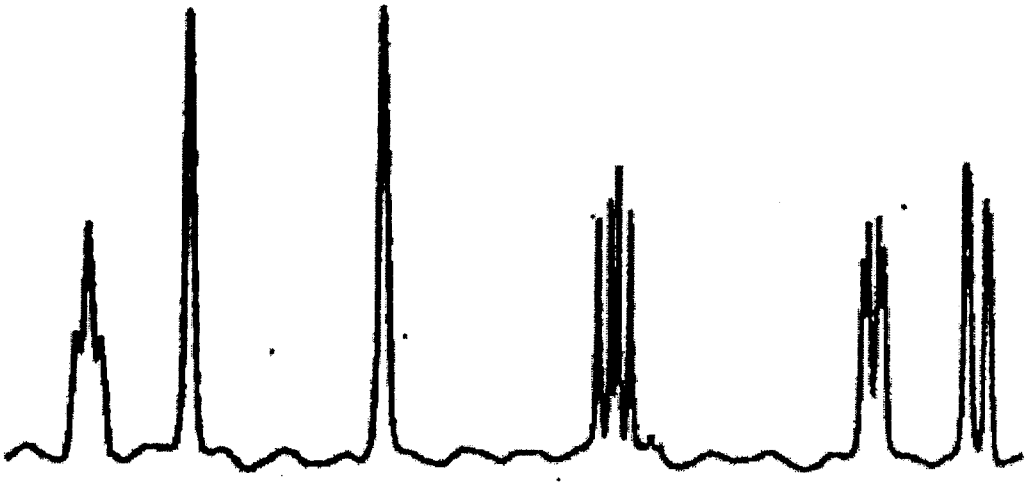


(10) Clipping

When the signal is too large to digitize the resulting FID is clipped.



Baseline Distortions caused by Clipping the FID



(11) 2D NMR

All of the NMR experiments that you have seen up to this point are one-dimensional (1D) techniques where the detected signal is a function of only one time variable. This results in a spectrum that gives the signal intensity as a function of only one frequency axis as seen in figure 29.

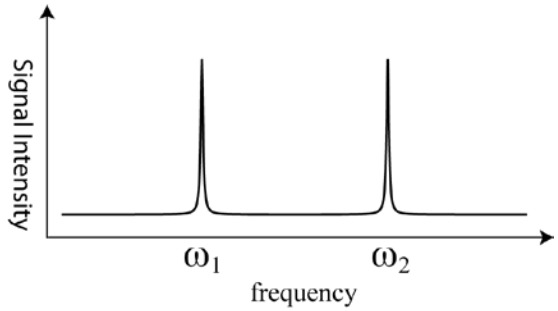


Figure 1A 1D NMR spectra containing to isotropic lines.

In complex molecules a 1D spectrum can become very crowded, due to multiple chemical shifts, and difficult to interpret; therefore, it is difficult to extract information about the connectivity, or interactions, of the various spin networks. In multi-dimensional NMR spectroscopy the detected signal is a function of several time variables and this provides a tool for analyzing the connectivity of these complex systems.

In two-dimensional (2D) NMR spectroscopy the signal is detected as a function of two time variables, thus providing a way to make observations of the spin-spin interactions directly. The general pulse sequence for a 2D NMR experiment is shown in figure 30 and is composed of four time periods known as preparation, evolution, mixing, and detection.

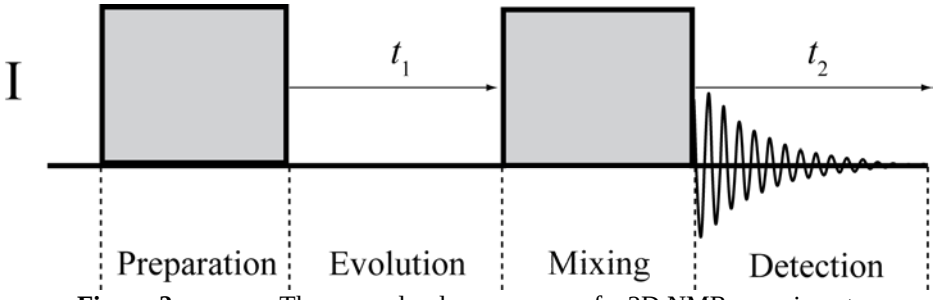


Figure 2 The general pulse sequence of a 2D NMR experiment.

The preparation period uses RF pulses to carefully manipulate the spin system and create a desired state, or coherence order, of the spin system. This state then evolves freely for the duration of the evolution period (t_1). It is important to note that the magnetization is not directly detected during the evolution period. The mixing period then uses RF pulses to re-convert the state of the spin system to a form in which it can be detected during the detection period (t_2). The resulting FID is now a function of both the evolution (t_1) and detection (t_2) time periods. The application of a Fourier transform in both time variables results in a 2D spectrum (figure 31) that contains the signal as a function of frequency along two axes, F_1 and F_2, and these axes correspond to evolution during the time periods of t_1 and t_2, respectively. The cross-peaks in figure 31 indicate correlations between spins due to coupling.

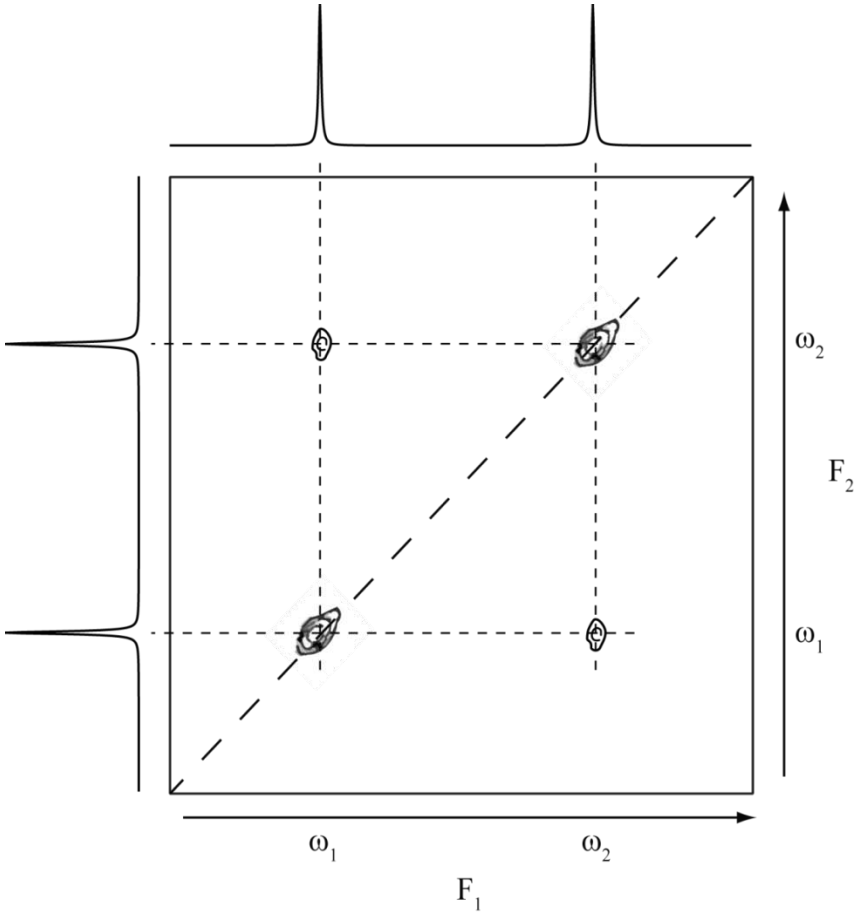


Figure 3A general 2D NMR spectra.

(12) T1 Evolution

The value of t_1 is incremented, and the sequence is repeated for each point in the indirect time dimension, thereby creating an array of FID's that constitute a data set that is two-dimensional in time S (t_1, t_2). This signal is then converted from time domains to the corresponding frequency domains, F_1 and F_2, via double Fourier transformation, as illustrated in figure 32. Finally, the 2D spectrum is displayed as a contour map plot with frequency axes labeled F_1 and F_2, correlations between the spins shown as a vertical projection of signal intensities, and the peak coordinates reflecting respective frequencies.

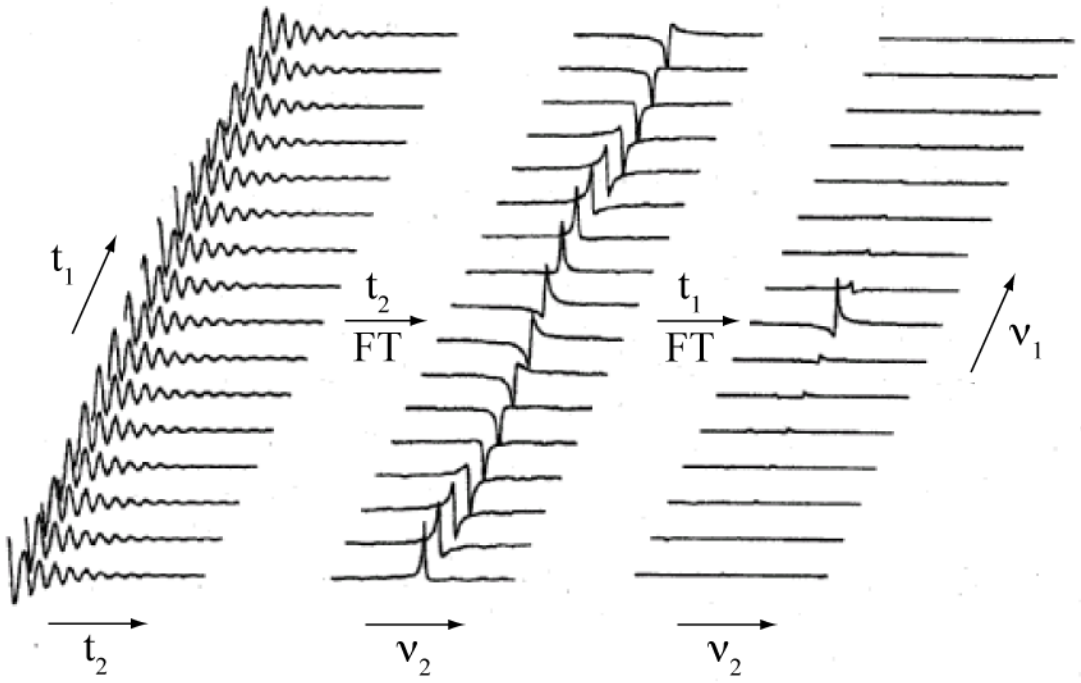


Figure 32 Structure of a two-dimensional NMR experiment.