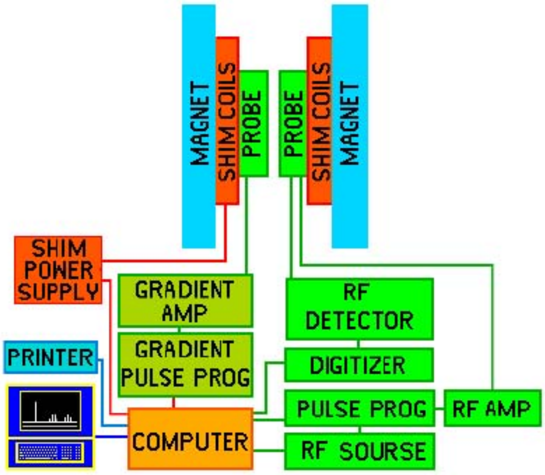
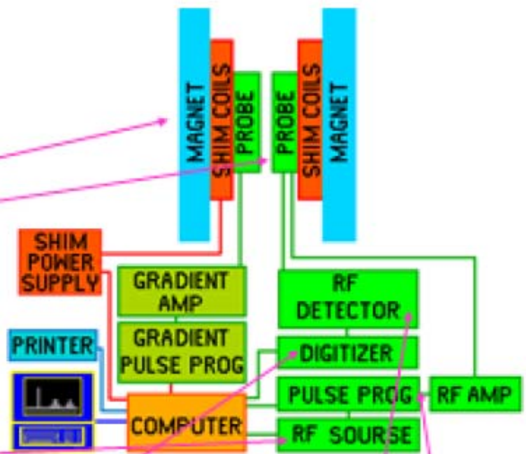
The Pulsed NMR Spectrometer

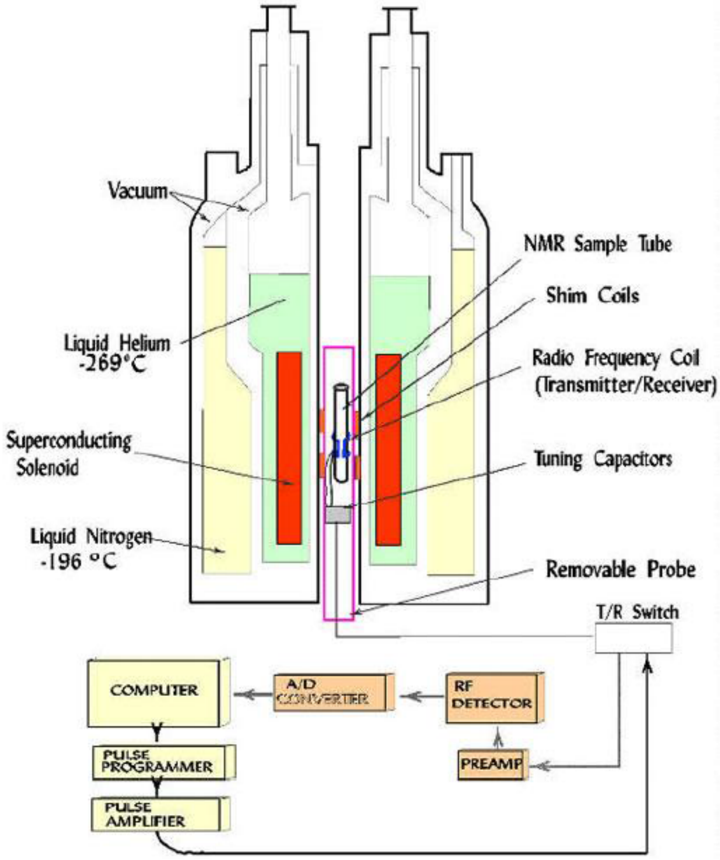


NMR Instrumentation

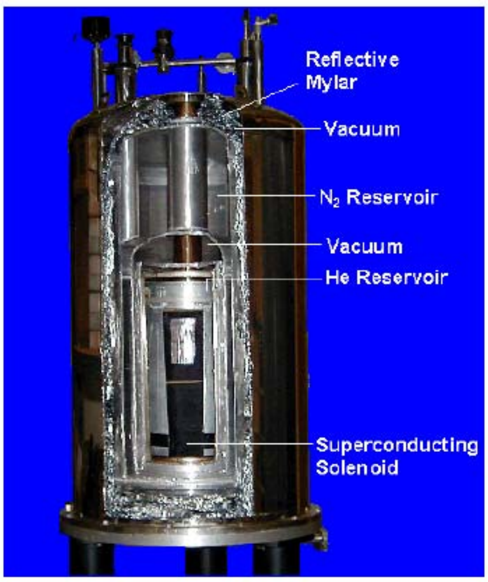


Components of a NMR spectrometer:

- An intense, homogeneous and stable magnetic field (magnet + shim)
- A "probe" which enables the coils used to excite and detect the signal to be placed close to the sample
- High-power RF transmitter/s capable of delivering short pulses (RF source + RF Amplifier)
- A sensitive receiver to amplify the NMR signals (RF Detector)
- A Digitizer to convert the NMR signals into a form which can be stored in computer memory
- A "pulse programmer" to produce precisely timed pulses and delays
- A computer to control everything and to process the data



Superconducting Magnet



- use **persistent** superconducting magnets to generate the B_0 field;
- at low temperatures (less than 6 K, typically) the **resistance goes to zero** – that is the wire(eg.Nb alloy) is ***superconducting***;
- To maintain the wire in its superconducting state the coil is immersed in a bath of liquid helium (**4 K, expensive**);
- "heat shield" kept at **77 K** by contact with a bath of liquid nitrogen (**cheap**) to reduces the amount of liquid helium boils off;
- vacuum flask so as to **further reduce the heat flow**.

Shim Coils

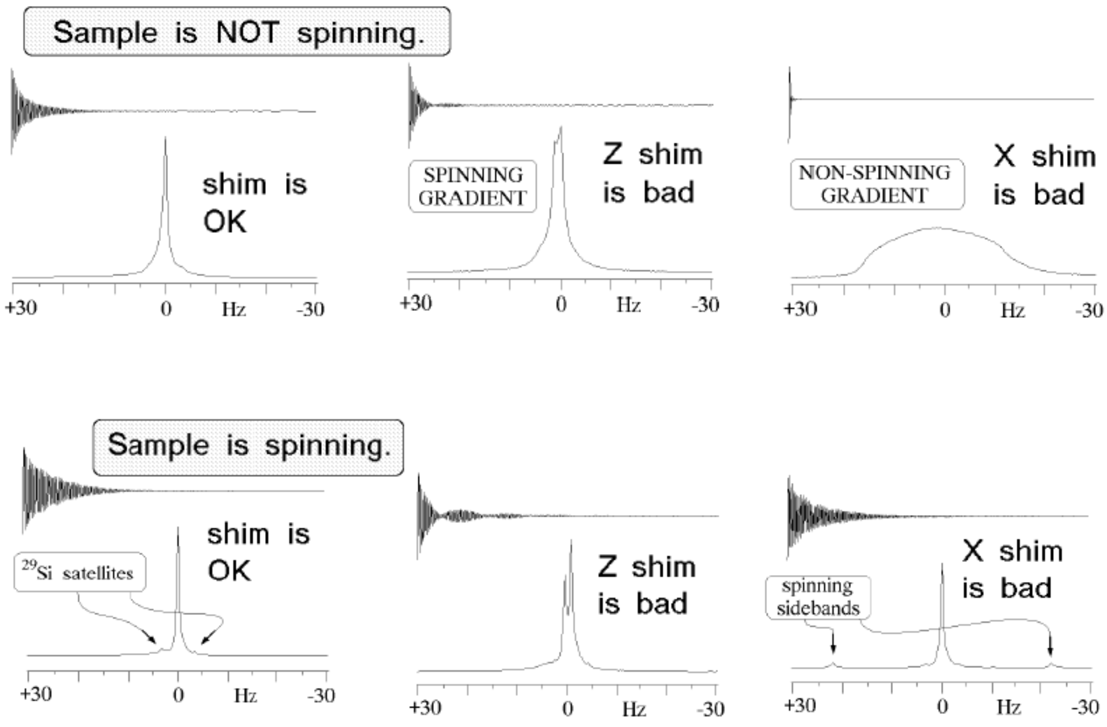
- High resolution NMR requires linewidths of 1 Hz or less

- Magnetic field across the sample must be homogeneous so that the corresponding variation in the Larmor frequency is small

- Surround the sample with a set of *shim coils*, each of which produces a tiny magnetic field with a particular spatial profile to canceling out the small residual inhomogeneities in the main magnetic field.

- Modern spectrometers might have up to 40 different shim coils labeled according to the field profiles they generate, such as *x*, *y*, *z*, z^2, z^3, z^4, z^5, xy, xz, yz, x^2-y^2, etc…

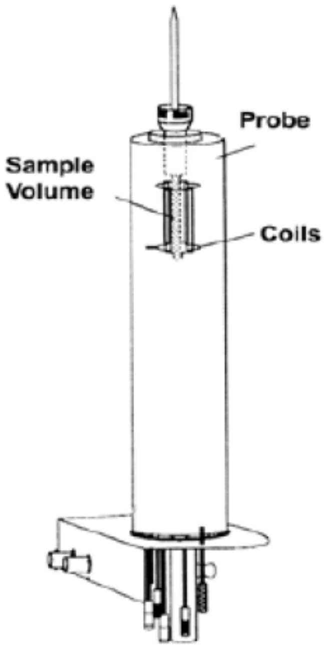
- Shimming, the process to optimize the shims, requires skill and experience because various shims will interact with each other.



The Probe

• The key part of the probe is the small coil used to excite and detect the magnetization in radio-frequency.

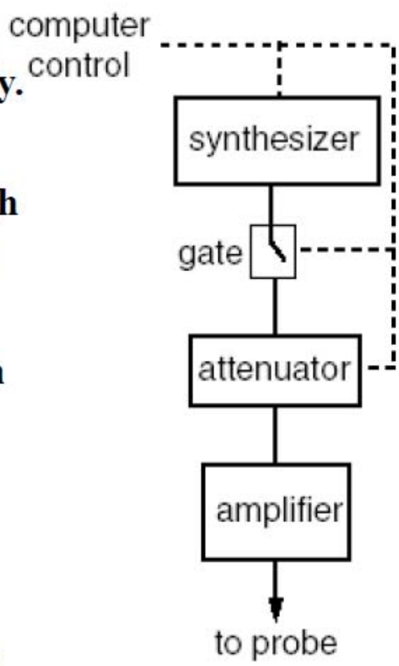
• To optimize the sensitivity this coil needs to be (1) as close possible to the sample; (2) tuned to resonant at the Larmor frequency of the nuclei being detected and (3) matched to maximize power transfer between the probe and the transmitter and receiver.



• Usually multi-coils for different nucleus: e.g. ^{1}H, ^{2}H (for locking), ^{13}C, ^{15}N, etc… with observe coil at inner-most position.

The transmitter: Channel

Synthesizer: RF source which produces a stable frequency which can be set precisely.



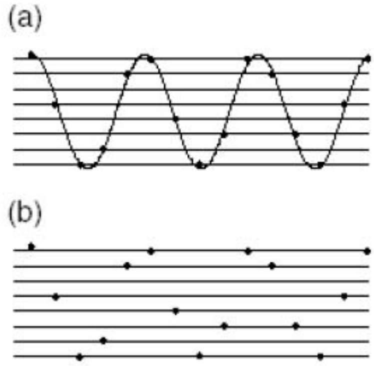
RF amplifier: boost this small signal to a power of 100 W or more to provide enough energy to excite the NMR active nuclei in the sample.

Attenuator: altering the RF power level in units of decibels (dB) (Bruker: 120 to –6 dB)

All under computer control

Each nucleus type required one set of transmitter channel => usually more than one channels

The receiver

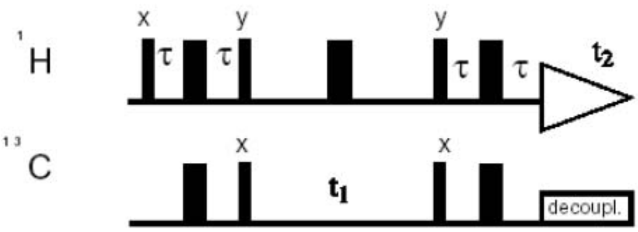


The NMR signal from the probe is detected by a digitizer receiver at regular time intervals (dwell time).

A device known as an *analogue to digital converter* or ADC is used to convert the NMR signal from a voltage to a binary number which can be stored in computer memory. Dynamic range of ADC digitizer is measured by bits (e.g. 16-bit, i.e. 0 to 2^{16}-1 or 65535).

Pulse programmer

to produce precisely timed pulses and delays required by the NMR pulse experiment



Computer system

Control all electronics
Date acquisition and processing (Bruker software-XwinNMR)
Plotting Spectrum (Bruker software-Xwinplot)

Third party software may be used for processing or analysis e.g. nmrPipe, Felix, nmrView.

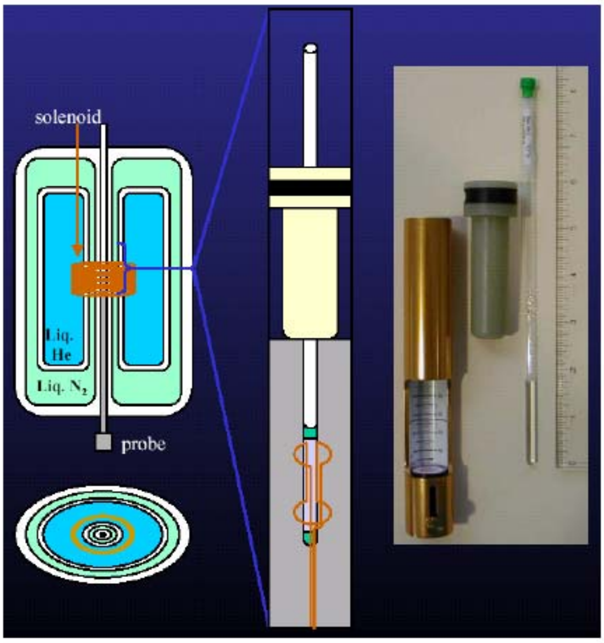
Additional Instrumentation

Sample spinner:

Spinning equalize xy magnetic field homogeneity, i.e. better resolution

Eject/Insert system:

using air stream to eject and insert sample tube along the long bore tube



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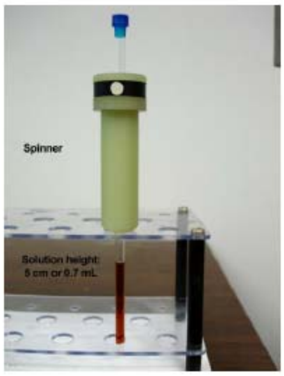

Additional Instrumentation: Locking (separate ^2H channel)

•Even in the best spectrometers the field strength varies to some extent over time

•The position of the deuterium peak is monitored

• To counteract the field drift a lock field is applied to maintain a constant deuterium resonance position

Deuteriated solvent is usually used to provide the Deuterium Lock signal e.g.

$CDCl_3$, D_2O, CD_3OD