NMR Instrumentation

BCMB/CHEM 8190

Biomolecular NMR
Instrumental Considerations - Block Diagram of an NMR Spectrometer
Modern NMR Magnets are Super Conducting Solenoids

- Materials: NbTi < 10T, NbSn > 10T
- Max Field (2000): 23.5 T (900 MHz)
- Advantages: high field, stability, homogeneity > 1 : 10^9
Shim Coils

Inherent field profile

\[ B_0(z) = B_0 + a_1 z + a_2 z^2 + \ldots \]

Design coils to produce \(-a_1 z, -a_2 z^2, \text{etc}\)
\(a_i\) are set by adjusting current in each of 16-40 coil sets – x, y, z, powers and cross terms

a linear z coil
Room Temperature Shim Assemblies

From Modern NMR Techniques for Chemistry Research, Andrew E. Derome
Shimming

Origin: old electromagnets used shims under pole pieces

Interactive shimming

Auto shim: based on amplitude of lock signal
Gradient shim: observing effect of imposed field gradient – can deconvolute field inhomogeneity

Adjust $z^2$
A Deuterium Lock Stabilizes the Field

Drift of field will produce positive and negative signals
Depending on direction when dispersive signal is observed
Detection of NMR Signals: Probe Coils

\[ E \propto \frac{dB'}{dt} \propto \frac{dM}{dt} \propto \gamma B_0 M_0 = \gamma^3 B_0^2 h^2 I(I+1) / (12\pi^2 kT) \]

\[ S/N \propto \left( \frac{\gamma_C}{\gamma_H} \right)^3 = 64^{-1} \]
NMR Probe

coops: \[ \text{to } B_0 \]

dual role:
- generate \( B_1 \)
- detect \( dM_y/dt \)

solenoid coil:
specialized applications

Helmoltz coil:
normal applications
**Coil Design: Needs/Implications**

Requirements:  
1. Maximize $B_1$ from applied rf current  
2. Maximize signal from $\bar{M}$ of sample

--- look at 2 ---

$$S \propto \frac{dB'}{dt} \times s$$

$$B' \propto \frac{M}{r^3}$$

$$S \propto \frac{M}{r}$$

So:

- minimize coil size to increase $S$
- maximize sample volume to increase $M$
- these demands conflict one another
  (compromise)
Tuned Radiofrequency (RF) Circuits Improve Efficiency

Example: parallel LC (inductance/capacitance) circuit

\[ \nu_0 = \frac{1}{2\pi\sqrt{LC}} \]

-the circuit is resonant (tuned) when the impedance is purely resistive \((\chi_L = \chi_C)\)
-the tuned resonance frequency is \(\nu_0\)
-Quality factors (Q) measure how much current is stored vs dissipated
– cryoprobes reduce resistance and loss \(Q \sim 1000\)
What does an NMR Probe Look Like?

This is for a 7T magnet – $^{13}$C observe at 75 MHz
Probes are delicate – glass, teflon, ceramic
Receiver Functions

1. Amplify signals: 10 microV -> 1 V  
   Three stages 30dB/stage  
   \[ dB = 10 \times \log(\frac{P_{out}}{P_{in}}) = 20 \times \log(\frac{V_{out}}{V_{in}}) \]

2. Shift Frequency: 100 MHz -> 1 KHz  
   convenient digitization in the “rotating” frame  
   often uses a “double-balanced mixer”

\[ V_{out} = a_0 V_{in} + a_1 V_{in}^2 + \ldots \]
\[ 2\cos(\omega_s)\cos(\omega_r) = \cos(\omega_s-\omega_r) + \cos(\omega_s+\omega_r) \]
Quadrature Detection
Allows distinction of +/- Frequencies

One detector
\[ y = My \]

Two detectors
\[ \text{Ref + 90} \]
\[ \text{Sig} \]
\[ \text{Ref} \]
\[ \text{Real out} \]
\[ \text{Imaginary out} \]

Actually split signal
And use two reference
Frequency phases
Why use Quadrature Detection?
To put $\omega_{\text{ref}}$ in middle of spectrum.

Reduced band width of audio filter increases S/N by $1/\sqrt{2}$