

M1 Magnetic Resonance Hardware

NMR Hardware Mark Newton Andy Howes



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- Many of these slides are (altered or not) from this Module.



What's in a name?

- NMR or MRI or ESR?
 - What is the same?
 - Everything?
 - Really?
 - You have a sample-patient, rock,
 - Something to excite spins
 - Something to detect spins relaxing
 - Something to process data
 - You are there to control it all











What do you need?

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- You're going to excite some spins and observe what happens when they return to equilibrium.
- Zeeman splitting, magnet or internal field
- Transmitter, to excite the spins
- Receivers to detect the spins relaxing
- You or a computer to process the data



Spectrometer Overview





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You might have a lot of hardware







Magnets

- Magnets
 - Permanent
 - Electromagnet
 - Superconducting
- What do we want?
- A magnetic field, B₀
- High spatial homogeniety
- High temporal stability
- Low fringe / stray fields

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Permanent Magnets I

Materials

- Iron (Fe)
- Ferrite (Fe2O3)
- Samarium Cobalt (SmCo)
- Neodymium Iron Boron (NdFeB)

- Important Properties
 - Residual magnetization (B_r)
 - magnetization with no applied field (H)
 - Coercivity (H_c)
 - applied field needed to reduce magnetization to zero
 - Want high H_c for our magnet "hard magnet"
 - Saturation
 - Increasing applied field does not increase magnetization

 $B = \mu_0 (H + M)$





Permanent Magnets II

- Need to limit amount of high cost magnetic material
- Generally use iron "yoke" to channel flux
- Shaped "pole pieces", used to homogenise field
 - OpenGARField magnet

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Generates defined gradient in vertical direction for imagining





Permanent Magnets III



What and where is <u>B</u>?

 $\underline{\mathbf{m}} = -\frac{\mu_0}{4\pi} \left(\frac{\underline{\mathbf{m}}}{|\underline{\mathbf{r}}|^3} - \frac{3\underline{\mathbf{r}}(\underline{\mathbf{m}}.\underline{\mathbf{r}})}{|\underline{\mathbf{r}}|^5} \right)$

Magnetization, volume, $dV: \underline{m} = \underline{M} dV$

Magnet Pole Pieces: magnetization, M $B_0 = \mu_0 M$ ($\mu_0 M < 1.3 T$)



MR2: NMR Hardware – Lecture 1

Homogeneous field



Permanent Magnet IV







0.3 T



Active Spectrum, Inc.'s Micro-ESR Oil Condition Sensor



Electromagnets I









Electromagnets II





Solenoid: n turns per unit length, current, l $B_0 = \mu_0 n I$



Electromagnets III



For a solenoid *n* turns per unit length, current, *I* $B_0 = \mu_0 nI$

An example $B_0 = 1 \text{ T} \rightarrow n \times I \approx 8 \times 10^5 \text{ Am}^{-1}$ e.g. $I = 150 \text{ A}; n = 5300 \text{ m}^{-1}$

Copper wires ($\rho = 1.7 \text{ x}10^{-8} \Omega m$) in a 20 cm thick annulus and ~ 0.7 m radius

Current density, $J = 4 \times 10^{6} \text{ Am}^{-2}$ Power dissipation ~290 kWm⁻¹!

NEED COOLING WATER !!

Difficult to get above 2T because of power dissipation

In the 70s copper was expensive, there are a lot of magnets with aluminium windings

You need a BIG power supply, water cooled as well.





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Superconducting Magnets I

Problem with a resistive electromagnetic is it's resistive!

What if we got rid of the resistance?

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Superconducting Magnets II

- Niobium Titanium Alloy
 - Nb/Ti T_c= 9.2K
- Niobium Tin
 - $-Nb_{3}Sn T_{c} = 18.3K$
- Magnesium Di-Boride
 - $-MgB_{2}T_{C}=39K$
- Yittrium Barium Copper Oxide $-Y_1Ba_2Cu_3O_7$ T_C= 92K
- But you have to look at (critical) current density and ease of use.

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Superconducting Magnets III



Important Properties Critical Temperature, T_c Critical Current Density, J_c Critical Field, B_c

How easy is it to use? Cost?



MN Wilson, Superconducting Magnets, OUP, 1983



Superconductors IV



Niobium Titanium (Nb/Ti)

$$T_c \sim 10 \text{ K}$$

 $J_c \sim 10^{10} \text{ Am}^{-2}$ (4.2 K)
 $B_c \sim 15 \text{ T}$
Ductile

Nb/Ti filaments embedded in copper matrix and "drawn" from a billet Used in most superconducting magnets (< 9.4 T).





Superconductors V



Niobium Tin (Nb₃Sn) $T_c \sim 19 \text{ K}$ $J_c \sim 6 \times 10^{10} \text{ Am}^{-2}$ (4.2 K) $B_c \sim 28 \text{ T}$ Brittle

Nb filaments embedded in (Cu/Sn) bronze matrix and "drawn"from billet, heat treatment reaction produces Nb₃Sn.

Used in high field superconducting magnets (>9.4 T) and cryogen-free systems operating at > 4.2 K.





Superconductors V



Drawing



Superconductors VI



Destruction of magnet by JEOL



Warning: don't attempt this at home.



Superconductors VI



Finite Length Solenoid

n turns per unit length, current, *I* Field on axis $B_0 = \mu_0 n I (\cos\theta_1 + \cos\theta_2)/2$





Need correction coils or alternative approach to produce adequate homogeneity in a short magnet or have a long magnet



Shielded S/C Magnets

Inclusion of reversed turns at larger radius reduces fringe fields: Active shielding look at Bruker manual





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- Access must be restricted to area in which fringe field > 5 G (0.5 mT).
- There is a maximum operating field for many pieces of equipment
 - (e.g gradient / RF amplifiers < 50 G).
- Space required for the installation is dictated by the extent of the fringe field.



Summary

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- Permanent
 - Low field (< 0.3 T whole body)
 - Heavy.
 - Stability affected by temperature variation.
 - Low purchase and running costs.
 - Low fringe fields
 - Good for geology
- Hybrid
 - Combination used to give very high fields

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Short duration



- Low field (< 0.2 T whole body)
- Stability affected by temperature change and power supply.
- High power dissipation.
- Low purchase costs.
- Superconducting
 - High field (< 9.4 T whole body, +20 T small bore).
 - Highly stable.
 - High running costs.

Field Homogeneity

Bare magnet homogeneity over sample space may be only ~100ppm.

Heterogeneity of magnetic susceptibility introduces sample-specific variation of ~a few ppm, i.e. change probe and you have troubles

Shim systems used to cancel unwanted spatial field variation.

(i) superconducting shim coils

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- (ii) passive shims (magnetic material)
- (iii) "room-temperature" shim coils





Spherical Harmonics





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Shim Systems





OMT 4T Shim System (mid-80's)



Superconducting Shims

- Wound on outside of magnet windings.
 - Often low orders only (e.g. X,Y,Z1 and Z2)
 - More for smaller bore systems
- Map field and calculate or do a MR experiment and adjust current.
- Currents set when magnet energised.
- Hopefully remove local fluctuations
 - E.g. from other magnets





- Steel pieces positioned at locations around inside of magnet.
- Positions calculated from field map.
 - Steel is saturated above 2 T
- Good for generating high order harmonics.
- Used in most clinical magnets.



Room Temperature Shims

- Copper coils wound on a cylinder around (or integrated with) the gradient coils.
 - Currents adjusted for each probe/sample/subject.
- On imaging systems, X, Y, & Z generally set via gradient amplifiers.
- Number of coils depends on system: e.g.
 - Clinical 1.5 T Ω
 - Clinical 3T
 - Wide bore 7T
 - Wide bore 11.7T

5 (Z2,XZ,YZ,XY,X2-Y2)

- 9
- 18



Magnetic Field Gradients



Magnetic field, B_{z_i} varying linearly with position, r

 $B_z = Gr$

Used for spatial encoding of NMR signal in MRI frequency encoding, phase encoding, slice selection

Used to encode motion information

diffusion, flow

Used for coherence selection

gradient selected spectroscopy



Magnetic Field Gradients

Need three, orthogonal gradients for imaging:

- *x*-Gradient $B_z = G_x x$
- y-Gradient $B_z = G_y y$
- *z*-Gradient $B_z = G_z z$

Maxwell's Equations: $\nabla \times \underline{B} = 0$ $\nabla . \underline{B} = 0$

Produced by passing current (*I*) through three gradient coils, usually wound on a cylindrical surface.

$$G = \eta I$$



Timing is everything

- NMR is sensitive to:
 - Timing errors
 - T1
 - K-space scanning
 - Phase coherence
 - Frequency jitter
- Need to be better than magnet
- Need stable and accurate frequency reference known as the CLOCK
 - Every RF output/input or timing comes from this one reference



The Crystal Oscillator



- Makes use of the piezoelectric property of a (quartz) crystal. The crystal behaves as a very stable LCR resonator with a very high Q.
- Very stable, which is what we want in MR.
 - Crystal oscillators are only an order of magnitude worse in accuracy and stability than an atomic clock.
- The oscillator is in an oven with an elevated temperature where the crystal will have a zero temperature co-efficient.
- Crystals also age due to relief of strain after manufacture. This settles very quickly.


Typical Circuit for a Crystal Oscillator





The output might be square, sinusoidal or something else



Horowitz and Hill: The Art of Electronics, Cambridge University Press 1980

Synthesiser



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Fourier Modulation Theorem

$$F(\omega) = \int_{-\infty}^{+\infty} f(t) e^{-i\omega t} dt$$

$$F(\omega - \omega_0) = \int_{-\infty}^{+\infty} f(t) e^{\omega_0 t} e^{-i\omega t} dt$$

$$\int_{-\infty}^{+\infty} f(t) \cos \omega_0 t dt = \int_{-\infty}^{+\infty} \left\{ f(t) \frac{\left(e^{i\omega_0 t} + e^{-i\omega_0 t}\right)}{2} \right\} e^{-i\omega t} dt$$
$$= \frac{1}{2} \left(F(\omega - \omega_0) + F(\omega + \omega_0) \right)$$



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$$\sin \omega_1 t \times \sin \omega_2 t = \frac{1}{2} \left(\cos(\omega_2 - \omega_1) + \cos(\omega_2 + \omega_1) \right)$$

- Notice this is a non-linear device
- $F_{out} = F_1 \times F_2$ not $F_{out} = F_1 + F_2$



Modulation



$$\sin \omega_1 t \times \sin \omega_2 t = \frac{1}{2} \left(\cos(\omega_2 - \omega_1) + \cos(\omega_2 + \omega_1) \right)$$





MR2: NMR Hardware - Lecture 3

Typical Mixer



Notice : 5-500MHz LO ,IF, RF Passive Device



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Why are we interested in mixing?



- One way of making higher frequencies than the oscillator (clock) frequency.
- Another way is Direct Digital Synthesis

 Because the frequencies we deal with are MHz/GHz and different for different gammas and different magnetic fields Wouldn't it be easy to bring this frequency range of signals down to something easier to look at (and then digitise and manipulate)



Why are we interested in modulation?

- We want to modulate:
 - Amplitude (as function of time) A(t)
 - To get sinc, secant or hard pulses
 - Phase (as function of time) $\phi(t)$
 - To get 0, 90, 180 pulse phase
 - E.g. 90_x, 90_y and 90_{-x}
 - Frequency (as function of time)
 - To select different slices
 - Chirp pulses

$$\omega = \frac{d\phi}{dt}$$









IF is a frequency called the intermediate frequency (fixed)

- **LO** is the local oscillator frequency (variable ω and ϕ)
- GATE is a switched DC voltage, maybe Transistor Transistor Logic, TTL

RF OUTPUT then goes to the RF power amplifier

The frequency of the out put will be the Transmitter frequency (and one other frequency).

IF and LO

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- Intermediate frequency is a fixed frequency for the spectrometer.
 Normally chosen to be an "odd" frequency.
- For instance 283MHz or 443MHz this is to avoid spurious signals in your FID. Remember you have to make frequencies in a spectrometer from the clock frequency.
- Local Oscillator is a very stable frequency from a very stable frequency synthesiser. The frequency of LO is chosen so that the sum or difference of this and the IF is the NMR frequency you want. It is also referenced from the clock.
- The **GATE** is OFF or ON, this way the output is the frequency from the first mixer times ZERO or the output is the frequency times a DC voltage.



Intermediate Frequency







RF Power Amplifiers

- For NMR we require
 - Stability + Repeatability
 - Low phase noise
 - Linearity
 - High power in short pulses
 - Lower power long pulses
 - Low noise when not pulsing (i.e. for receive)

Mainly Solid State Devices, The odd valve amplifier for protons









MR2: NMR Hardware – Lecture 3

Receiving the signal

- Signals are nV to microvolts
- Need order of 1000000 times gain (120 dB)
- Need good linearity and high dynamic range (SFDR is spurious free dynamic range >90 dB)
- Noise figure:
 - Signal to noise $(SNR)_{out} (SNR)_{in}$ [in dB]
 - -0.5 dB typical





 'Process of recovering information added to carrier frequency' [telecoms definition]

- Process of recovering spectrum from frequencies at Larmor frequency
 - Spectrum is shifted down to the "zero" frequencies by modulating with LO and IF frequencies
 - Why two stages? (I'll tell you later)



Demodulation

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- A problem:
 - How do we discriminate positive and negative frequencies?





Why not demodulate on one side?

- We do sometimes
 - this would lead to extra noise
 - and higher sampling frequency
 - Thanks to mobile phones this is now sometimes done





Quadrature Detection



 Is our detected frequency higher or lower than the transmitter frequency?



If we can split the frequency into two components $\pi/2$ apart and look at how They evolve with time we can learn which why it is rotating.





Bandwidth of Receiver





Early receivers came from the radio and had an audio bandwidth, 20KHz

Later on video bandwidths, 8MHz

Now 20MHz thanks to modern communication technology.



MR2: NMR Hardware - Lecture 1

Analogue to Digital Convertors

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- Converts voltage to digital value suitable for sampling and processing by computer
 - Require number of bits to cover dynamic range
 - 16 bits > 90 dB SFDR
 - Need to sample at at-least twice the highest frequency to be discriminated
 - MRI need 500 KHz bandwidth
 - NMR may need few MHz bandwidth



In the (good) old days!



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Digital Spectrometer

NARIAN I

 Signals are digitised immediately after amplification





MR2: NMR Hardware - Lecture 3

Computing resources

- Front panel interface
- Sequence design, control and management
- Data acquisition
- Data processing
- Data archive and storage
- NMR, ESR one PC
- MRI up to four computers!!









Apply oscillating voltage --- oscillating current

-////-

- $V(t) = V_0 cos \omega t$
- $I(t) = V(t)/R = V_0 \cos \omega t/R$
- So voltage and current are in phase



Electronics Refresher II



Capacitor

|--|

- Apply oscillating voltage --- oscillating current
- Q = CV C capacitance
- dQ/dt = CdV/dt = I
- $I(t) = -C\omega V_0 sin\omega t$
- So, current leads voltage by $\pi/2$ and there is an ωC term
- $Z_c = 1/j\omega C$, reactance
- j (or i) gives you phase information

Electronics Refresher III





- dl/dt = V/L L inductance
- integrate
- $I(t) = (V_0/\omega L) sin \omega t$
- So, current lags voltage by $\pi/2$ and there is an $1/\omega L$ term
- $Z_L = j\omega L$



Complex number Refresher



- Invent a complex voltage,
- $V(t) = V_0 e^{j\omega t} = V_0 cos\omega t + jV_0 sin\omega t$
- Compare with original term for V(t), at the end we are interested in real component.
- With a capacitor, I = CdV/dt, so
- $I(t) = j\omega CV_0 e^{j\omega t} = j\omega CV_0 cos\omega t \omega CV_0 sin\omega t$
- Real part, $I(t) = -\omega CV_0 \sin \omega t = \omega CV_0 \cos(\omega t + \pi/2)$
- Compare with V = IZ where Z is the impedance
- Z_c looks like 1/ ω C but with a phase change of $\pi/2$



Complex number refresher

- Or just $V(t) = V_0 e^{j\omega t}$
- $I = CdV/dt = j\omega CV_0 e^{j\omega t}$
- Remember $e^{j\pi/2} = j$
- $I = \omega C V_0 e^{j(\omega t + \pi/2)}$
- This gives you a $\pi/2$ phase shift
- cf, V = IR
- So we can write, $Z_c = 1/j\omega C$
- Can do the same thing for an inductor
- and can write, $Z_L = j\omega L$





Complex number refresher

- VARIAN
- So complex numbers gives us phase information for currents and voltages
- Real means in phase with applied voltage (or current)
- Imaginary means $\pi/2$ with applied voltage
- Why do you care about this?
- Because only Real impedances (resistive stuff) absorb power
- Think integrating terms like (cosωt)(sinωt) wrt time



Electronics Refresher

- Why do you care about this?
- Because NMR signals are small
- You want to maximise the signal you see
- The heart of an NMR probe is the (work) coil, an inductor

 This has to make B₁ and pick up the emf from spins relaxing (this can be nano volts)





A first example of a NMR circuit

- Lets analyse this circuit,
- $1/Z_T = 1/Z_1 + 1/Z_2$ etc
- $Z_T = j\omega L/(1-\omega^2 LC)$
- Purely imaginary reactance, no real components
- Can write, $V(t) = V_0 e^{j\omega t} = I(t)j\omega L/(1-\omega^2 LC)$
- No power is dissipated by the circuit, no resistive heating
- BUT circuit has an unique frequency, $1/\omega C = \omega L$
- Lets not call this resonance (yet)





Maximise power transferred into circuit

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In reality inductors have a small resistance

A better circuit

- $1/Z_{T} = 1/(j\omega L + r) + j\omega C$
- A bit of complex algebra

Real $Z_T = r / ((1 - \omega^2 LC)^2 + (\omega rC)^2)$

Imag $Z_T = (\omega L(1 - \omega^2 LC) - \omega r^2 C) / ((1 - \omega^2 LC)^2 + (\omega rC)^2)$

- Reconance is defined as when the circuit is purely resistive
- Resonance is defined as when the circuit is purely resistive
- What's so important, voltage and current are in phase
- Why do you care?

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Resonance condition

- The circuit is resistive when,
- $(1-\omega^2 LC) r^2 C/L = 0$
- i.e. when the imaginary part is equal to zero.
- Often the term $r^2C/L << 0$ so at resonance $1 \approx \omega^2 LC$
- Resistive part r / $((1-\omega^2 LC)^2 + (\omega rC)^2)$
- We can make this circuit resonant at a chosen frequency ω by changing L and C
- We can also make this circuit have a particular impedance by changing $\omega,\,L$ and C
- In reality this is difficult with only two components



Tuning and Matching

- What we want to do is TUNE a probe to a particular frequency and match to a particular real impedance
- Why do you want to?
- Because the frequency will be the Lamor frequency for your nuclei so you can manipulate the spins
- Because you need to impedance MATCH the probe circuit to the spectrometer power amplifier and the spectrometer receiver.
- This is to maximise the transfer of power to make B₁ and to maximise the transfer of NMR signal to the receiver





Impedance standards



- You need some standard values for input and output impedances of devices
- 50 Ω , 75 Ω , 600 Ω are all different standards
- Values chosen for material characteristics and historical reasons
- We have to consider the impedance characteristics of all devices, amplifiers, receivers, individual components, the cables that connect them
- You have to chose a standard
- Most popular for magnetic resonance is 50Ω


Transmission Lines

- Often hear that the cable between a spectrometer and probe is referred to as a 50Ω cable (or even a 50Ω co-axial cable)
- What is at 50Ω?
- $50 = (L/C)^{1/2}$
- L is the inductance/unit length
- C is the capacitance/unit length
- Why do you care?
- Because this is a lossless transmission line, no energy lost in the cable whatever the frequency (with a proviso)
- Solutions to I and V look like simple wave equations



An Experiment

- put V₀cosωt into one end of a length of transmission line
- Terminate the other end with a resistance of (L/C)^{1/2}
- Then make $V(x,t) = V_0(\cos\omega t kx)$, (we'll just look L to R)
- The wave number k gives us the velocity of 1/ (L/C)^{1/2} from that differential equation
- i.e. the wave moves away from our input end at 1/ (L/C)^{1/2} independent of frequency
- Current at terminating resistor? = $V_0(\cos\omega t kI)/(L/C)^{1/2}$
- But this will work whatever the length of line, so current at any x = V₀(cosωt – kx)/ (L/C)^{1/2}



More transmission line



- What do we learn from terminating the line with the characteristic impedance
- Because if we terminate the line with the characteristic impedance then looking from the other end of the line we see the characteristic impedance
- i.e. $V(0,t)/I(0,t) = (L/C)^{1/2}$
- The transmission line is Ohmic and independent of frequency
- This means V and I are in phase for any frequency
- Why does all this matter?
- We can match input and output impedances



Co-axial cable

Lots of transmission lines we use are co-axial cable with a 50Ω impedance

- Inner and outer copper conductors with a material between them of a particular relative permittivity ε
- If a is radius of inner conductor and b is overall radius
- Then, after a bit of fiddling and some honest approximations

$$Z_0 = \frac{60}{\sqrt{\varepsilon}} \ln\left(\frac{b}{a}\right)$$

• You can also have parallel and strip transmission lines









More reflections

- Open circuit --- no current at termination
- Short circuit --- no voltage at termination
- No power transmitted, standing waves set up
- In general a phase difference for voltage and current between forward and reflected waves.
- The cable is pretty lossless but you lose with,
 - Long cables
 - Higher frequencies
 - High power
 - Any junction, connection



Quarter lambda lines

- Useful trick
- At home tonight you can work out that

$$Z_{in} = Z_0 \left\{ \frac{Z_t \cos kl + jZ_0 \sin kl}{Z_0 \cos kl + jZ_t \sin kl} \right\}$$

- Here I is the length from the termination (work from left to right so this would be –I) Z_t is the termination impedance
- K is the wave number $=2\pi/\lambda$ for a particular frequency
- What if we make $kl = \pi/2$, i.e. l = a/4

$$Z_{in} = Z_0 \left\{ \frac{0 + jZ_0}{0 + jZ_t} \right\} = \frac{Z_0^2}{Z_t}$$





Quarter lambda line

- Can be used to match mismatched lines
- More interestingly can be used to protect the receiver
- Remember we have calculated the impedance back down the line at –I not at the junction, current and voltage will be out of phase at the junction unless Z_t = Z₀
- Since $Z_{in} = Z_o^2/Z_t$ where Z_t is how we have terminated, then if we short the end ($Z_t = 0$) then back at the other end of the line Z_{in} will be infinite



Protecting a receiver

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Lots of volts across a receiver will kill it



- Use the above circuit
- The diodes will conduct if potential across them is a few mV
- So diode end of quarter lambda line looks like a short if transmitter is on
- At other end towards probe looks like 50Ω
- Receiver direction looks like infinite impedance!!!



Cross diodes

- Normally more than just two diodes
- But diodes add capacitance
- Another use of cross diodes is to kill noise from the receiver when it's waiting to pulse. Just put the cross diodes in series (in between) with the receiver and probe this also makes the transmitter look like a huge impedance for a small signal looking from the probe. (i.e. the voltage is too small to switch on the diode)
- Remember we used a cable but you can replicate a quarter lambda line with a simple LC circuit





Double resonance

- We have seen that with some L and C we can make a resonant circuit. Some probes really are that simple
- One coil one resonance, what about double or even triple resonance?
- With our simple circuit we inject RF in at the LHS and ground the RHS



- What if we want two resonances and the sample to see two different frequencies for CP, decoupling etc
- Can use two coils but there will be mutual induction between the two. BUT there is an easier way





Filters

• Notch Filter

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- Lets every frequency through apart from one narrow band.
- $Z = j\omega L/(1-\omega^2 LC)$. Blocks one frequency.
- Band pass



- Lets through one (range) frequency. $Z = (1-\omega^2 LC)/j\omega C$
- Chose L and C to pass the frequency you want



Double resonance





- Can add more components
- Becomes more difficult as freq A and B get closer
- Sometimes a $\lambda/4$ line is used (to block protons) and ground the broad band channel instead of the parallel L and C (RHS)
- Triple resonance? Add second broad band channel and second coil parallel to work coil to couple wit the work coil. Always less efficient than double resonance
- Be careful RF wavelengths can be the size of probes/ components.



Real Probes



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Some Real MR probes



- You can make a probe from a few capacitors and an inductor yourself. It costs a few pounds
- You can spend 20K or more on a commercial probe
- It all depends what you want
- Some of our prototypes have come from CAD drawings, some from components stuck on a sheet of cardboard



Static, Single Resonance

- •One coil, notice it's a flat ribbon
- Interchangeable, change inductance





Variable capacitor -tune
Fixed value, interchangeable capacitor- tune
Variable inductor-match



Skin Depth

For RF there is a problem of penetration into the conductor. RF travels on (or close) to the surface. Skin depth,

$$\delta = \sqrt{\frac{2}{\mu\mu_0\sigma\omega}}$$

- μ_0 , permeability of free space μ , relative permeability (~1) σ conductivity
- Copper with RF at 100MHz, skin depth ~0.1mm
- This all comes from oscillating electric fields set up currents in conductors, work is done to drive the current, wave is attenuated in the conductor, (and you get resistive heating)



Flat Ribbon Cable/Wire/Tape



- At high frequencies all current is carried in a thin outer layer, this is the skin effect
- Ribbon can give you better B₁ homogeneity
- Can be caught out resistance goes up with frequency. RF resistance of ribbon can be vary different from that at d.c.



Magic Angle Spinning



- As you know from MR1, many NMR interactions have a (3cos²θ –1) term. So if you rotate the sample at the magic angle and the spinning speed is faster than the interaction you get narrowing as you have removed the interaction. Better resolution.
- Tricky on anything but a powder.
- Lots of static single crystal studies done as a function of angle-goniometer probe head
- Solution state always do a bit of rotation to average any B₀ inhomogeneity



MAS probe head





•The angle

Angle adjustment (set this with an NMR experiment)Optics



Inside the probe head





MR2: NMR Hardware



Rotors

- Choose spinning speed for experiment
- ~100Hz to +70KHz
- Watch out for overall sensitivity. You can't spin large rotors fast
- Rotor diameters vary from 1.1mm to 17mm
- Volumes vary from ~1ul to ~1ml
- Careful packing

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Watch density of sample, worry if >3g/cc

Can have micro-coils and samples nl volumes

MR2: NMR Hardware



Rotors





MR2: NMR Hardware

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Spinning Speeds



•A,B,C&D are different wall thicknesses and materials
•Doty Inc data
•1/R² dependency



MR2: NMR Hardware

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How is it spun?

- Compressed air, typically supplied at 6 bar
- Rotor floats on air bearing and is rotated by turbine





• Different drive and bearing pressure for different probes



More spinning

- For high and low temperature experiments high pressure nitrogen gas is used
- No oxidation of probe
- No CO₂
- No icing





Spinning Speed?

Need to know



- sometimes because of synchronisation with experiment
- So you don't spin to fast and break it.
- Measured
 - Acoustically
 - Tribo electric effect
 - Optically
 - "To thine own ears be true"Shakespeare, Taming of the ShrewWhat does it sound like?











MR2: NMR Hardware

Variable Temperature

- Static, from 10⁻⁴ K to +10⁺³ K
- Spinning, from 5K to ~10⁺³ K
- Static very low temperatures are reserved for He-3 experiments
- Conventional He-4 cryostats get you down to a few K
- High temperatures can be achieved by using
 - Furnace (wire wound)
 - Laser
 - Light bulb

• Always have the problem of cooling and noise





Variable Temperature

- Spin on helium gas down to 4K, nitrogen down to 80K
- Spinning close to1000K can be done with very special probes
- Lot of conventional spinning probes have a VT range of ~150K to ~400K
- Drive and bearing gas is heated cooled, or separate VT gas supply
- Problems
 - Oxidisation
 - Water, CO₂

luate School

- Noise introduced by thermocouple
- Magnet, electronics etc getting too hot or cold



4K static probe





NDS

CF

GraduateSchool



MR2: NMR Hardware

Tuning and Matching a probe

- On the bench, the network analyser
- Output/input impedance 50Ω
- Sweep frequency range
- See what is and what isn't reflected back







3dB point, What is a dB?

• It is a relative power measurement,

Relative power (dB) = $10Log_{10}$ (P/P₀)

Relative voltage (dB) = $20Log_{10}$ (P/P₀)

- Introduce the dBm where you define P₀ as 1 mW
 - so 0 dB, no attenuation, 20 dB, 100 fold attenuation
 - 10 dBm is 10mW, 60 dBm is 1KW
 - If it's 50Ω impedance you also have voltage



Quality factor

- Back to the two resonances from the two probes
- 4MHz width and 1MHz width
- At -3dB point, this is half power, (1/2 to probe, 1/2 reflected)
- Quality factor is: width at -3dB point / freq. at resonance
 - Both at 115MHz, so Q is ~25 and ~100
 - Different probes for different jobs
 - Low Q, wide line static, irradiate a broad range but low sensitivity
 - High Q, narrow line (MAS) high sensitivity

•Can Q spoil with a few Ω s in series with coil







Tuning at the spectrometer

- Directional coupler
 - Clever device that taps off a fraction of power going to or from the probe from the power amplifier (maybe a 50dB worth)
 - Common to use second transmission line coupled to main line
 - See if the frequency you want is going to the probe or not
- This can be built into the spectrometer "Q tune or RP tune" Varian or "wobb" Bruker







Directional coupler, $\lambda/4$ set up







MR2: NMR Hardware

Directional coupler

- Can sweep frequency and look for resonance
- Fix frequency for finer tuning and matching
 - Look at reflected signal from directional coupler on oscilloscope and minimise it
 - This gives you the best tune and match




Little bit more on match and tune



- You are using variable capacitors and variable inductors
- Swapping fixed capacitors and inductors (for different ranges)
- Most of the time these are "off the shelf" standard items that look like capacitors and inductors.





Bruker probe

More Photographs







Slightly different capacitors

- You have the problem of RF arcing across components
- You can win with space
- Probes can only be so big, so components are only so big, but sometimes you see something very clever
- Have big surface to distribute volts/current



Tune tube technology, Varian Inc, now Agilent









Field strength of work coil



 $B = \mu_0 N I$

•B is the magnetic field, N is turns/unit length, I is the current. μ_0 is the permeability of free space

So to double the field strength you double the current
But the probe looks purely resistive so double the volts you double the current

•But this is four time the power, V^2

•Take care you can end up arcing across the coil



RF arcing

- VARIAN
- Can be across coil or to other parts of probe after air is ionised.
- Long pulses and/or high power can be a problem
- Low temperature experiments with helium and partial vacuum also causes problems
- If coil or another component is arcing, the NMR circuit is no longer tuned or matched and power is reflected back
- Sometimes this problem is not obvious until you take the probe apart. (apart from the lack of signal)



More arcing







And more arcing





MR2: NMR Hardware

VARIAN

And more arcing







Electron Spin / Paramagnetic Resonance

• Unpaired electron. EPR spectra can be complicated

$$\hbar\omega = g\mu_B B$$

- G-factor can be very different from 2, μ_{B} Bohr magneton
- 4 Tesla gives you 100 GHz
- The world of microwaves







ESR probe





MR2: NMR Hardware

VARIAN

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