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The Design of a Single Coil Double
Resonance NMR Probe for Combined
Magic Angle Spinning Double
Resonance Experiments

by

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and
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MASTER

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Date Transmitted: March, 1978

PREPARED FOR THE DEPARTMENT OF ENERGY
UNDER CONTRACT W-7405-eng-82

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Printed in U. S. A.

Available from: National Technical Information Service
U. S. Department of Commerce
P.O. Box 1553
Springfield, VA 22161

Price: Microfiche	\$3.00
Paper Copy	3.50

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Abstract

A design of the r.f. circuitry for a single coil double resonance NMR probe is presented. The design is useful for NMR double resonance experiments at two widely separated frequencies. The design is characterized by relatively high power efficiency, high isolation between the two frequencies used, and excellent signal-to-noise ratios for the detected NMR frequency. Using this design, most single frequency probes can be easily modified for double resonance experiments.

I. Introduction

Cross polarization experiments which require the Hartmann-Hahn¹ condition to be satisfied over the entire sample imply the use of a single coil to produce homogeneous r.f. fields. Adequate high frequency isolation is also required since the rare spin is usually observed with abundant spin decoupling at the higher frequency. Thus the high frequency field present in the coil will overload the lower frequency receiving system if adequate isolation is not provided. Also, a large coil capable of handling a large sample is desirable to reduce coherent time averaging for low abundance nuclei - especially when a spinning rotor design is used. This report presents the design of a single coil spinning rotor double resonance probe with a 10 mm i.d. inductor. This compact design has been used to obtain high resolution data on natural abundance C-13 in coals using the Proton Enhanced Nuclear Induction Spectroscopy (PENIS¹) technique.

Other designs^{2, 3} which use 5 mm coils have not been found satisfactory when larger 10 mm inductors are used. The main problem with these designs is poor frequency isolation which results in high and/or low frequency power inefficiency.

II. Circuit Description

1. Design

Figure 1 shows a schematic layout of the probe circuit. The capacitor, C_{IL}, and transmission line, D₁, are used to tune the probe at the lower frequency f_L (14.1 MHz in the present design). Capacitors, C_{1H} and C_{2H}, are used to tune the probe at f_H (56 MHz in the present design). Isolation of f_H at the low frequency input is achieved by an open half wavelength of coaxial cable which produces a low impedance at point "a" and a high impedance at point "c" in Fig. 1. Both cables are of characteristic impedance, Z_0 .

Isolation of f_L at the high frequency input is obtained with capacitor C_{1H} which also serves as a tuning element at f_H .

Figure 1. The radio frequency circuitry of the spinning rotor probe.

2. Restrictions

The design will be most effective when the following conditions are met:

$$\text{at } f_H: R_{||} \gg X_L''' ,$$

$$\text{at } f_L: X_{C1H} \gg X_L' ,$$

and

$$\text{at } f_L: Z_{0/4} \cotan 2\pi D_\lambda' > X_L' .$$

$R_{||}$ is the maximum resistive impedance looking into the first $\lambda_{H/4}$ at point "c" in Fig. 1. X_L' and X_L''' are the inductive reactances to the coil at f_L and f_H respectively. D_λ' is the length of cable in wavelengths at f_L between points "a" and "c" of Fig. 1. This particular cable is adjusted so that the maximum resistive impedance, $R_{||}$, occurs at f_H . This impedance should be much larger than the inductive reactance of the coil at this frequency. As will be shown later, the high frequency quality factor, $Q_H \approx \frac{R_{||}}{X_L'''}$, is strongly dependent on this resistive impedance $R_{||}$. An inductor with $X_L''' = 98.7$ ohms at $f_H = 56$ MHz was used. $R_{||}$ was 2200 ohms, so $R_{||} = 22.3 X_L'''$.

The second restriction insures that the majority of the low frequency power will flow through the inductor. Since the value of the reactance, X_L' , is known, the value of the reactance, X_{C1H} at f_L can be calculated using the formulas presented in Fig. 2. Thus the validity of this criterion can be checked before construction of the probe. For the probe described here $X_L' = 22.7$ ohms and $X_{C1H} = 320.1$ ohms at $f_L = 14.1$ MHz thus $X_{C1H} = 14.1 X_L'$.

High Frequency

$$R_H = R_{RH} + R_{RC} \quad Q_H = \frac{X_L'''}{R_H}$$

$$C_{CH} = [2\pi f_H (X_L''' - R_H \sqrt{\frac{Z_{IN}}{R_H} - 1}]^{-1} \quad (1)$$

$$C_{C2H} = \frac{\sqrt{\frac{Z_{IN}}{R_H} - 1}}{2\pi f_H Z_{IN}} \quad (2)$$

Low Frequency

$$R_a = Z_a \cos \phi_a, \quad X_a = Z_a \sin \phi_a, \quad Z_{IN} = Z_0, \quad Q_L = \frac{X_L'}{R_{RL}}$$

$$B = \frac{Z_0 X_a - \sqrt{Z_0 R_a (R_a^2 + X_a^2 + Z_0^2 - 2Z_0 R_a)}}{X_a^2 + R_a^2 - Z_0 R_a}$$

$$D_{D1} = (1/2\pi) \tan^{-1} B \quad (3)$$

$$X_{C1} = Z_0 \frac{[X_a Z_0 + (Z_0^2 - X_a^2 - R_a^2) \tan 2\pi D_{D1} - X_a Z_0 \tan^2 2\pi D_{D1}]}{(Z_0 - X_a \tan 2\pi D_{D1})^2 + (R_a \tan 2\pi D_{D1})^2}$$

$$C_{C1} = [2\pi f_L X_{C1}]^{-1} \quad (4)$$

Figure 2. The design equations for the spinning rotor probe.

The third restriction insures that the low frequency impedance is inductive at point "a" and will be tunable with capacitor C1L and cable D1.

$$D'_{\lambda} = .0592 \text{ for } f_L = 14.1 \text{ MHz}$$

and thus with $Z_0 = 50$ ohms

$$Z_0/4 \cotan 2\pi D'_{\lambda} = 1.41 X'_L.$$

3. Isolation

Isolation of the high frequency at the low frequency input is obtained by using an open half wavelength of coaxial cable in the low frequency side of the probe, as shown in Fig. 1. This open half wavelength will have a relatively high impedance at point "c" in Fig. 1. This impedance can be estimated by first considering the impedance at point "a" produced by just the open quarter wave.

At $f_H = 56$ MHz for RG 58 c/u cable, this impedance is roughly 1 ohm. This 1 ohm will be ideally transformed ($Z_{out} = \frac{Z_0^2}{Z_{in}}$) to 2500 ohms at point "c". The measured value of $R_{||}$ was 2200 ohms. This lower value is attributed to the non-ideal behavior of the cable.

An estimate of the isolation can be obtained by considering the ratio of the high frequency voltage at point "c" to that at point "a". This ratio is just the ratio of the impedances at the two points. The high frequency input voltage is amplified by Q_H at point "c" for small values of R_H , ($= R_{RH} + R_{RC}$) in Fig. 3. For this probe $Q_H = 24.5$, thus an isolation of $\frac{2200}{(24.5)^2}$ equivalent to 39.1 dB is expected. An actual value of 33 dB was measured at the low frequency input port. This lower isolation is attributed to the effects of cable D1,

EQUIVALENT CIRCUITS

LOW FREQUENCY

$$C_{\lambda_H/4} = \frac{\tan 2\pi D_{\lambda_H/4}}{2\pi f_L Z_0} \quad 1.$$

HIGH FREQUENCY

$$R_{RC} \approx \frac{(X_L''')^2}{R_{II}} \quad 2.$$

the length of which is close to $\lambda_H/4$.

Low frequency isolation is achieved by the large reactance, X_{C1H} , of capacitor, $C1H$, at f_L . As a first approximation this reactance will form an r.f. voltage divider along with X_{C2H} at f_L . The low frequency input voltage will be amplified by $|1+K_L|$ at the load, Z_L , which consists of the inductor and resistor, R_L . K_L is the voltage reflection coefficient which may be complex and ranges in magnitude from 0 to 1. Z_0 is the characteristic impedance of the cable.

$$K_L = \frac{(Z_L/Z_0 - 1)}{(Z_L/Z_0 + 1)} \cdot 4$$

For

$$X'_L \gg R_{RL}, \quad Z_L \cong jX'_L,$$

and for $X'_L = 22.7$ ohms, $|1+K_L| \approx 2$.

Thus an isolation of $\frac{321 + 58.7}{(2) 58.7}$ which is equivalent to 10.2 dB is expected.

An isolation of 11.4 dB was measured.

A shorted $\lambda_H/4$ and/or a bandpass filter between the transmitter and probe will lessen the low frequency power which enters the high frequency transmitter. All losses from this isolation will be accounted for in R_{RL} and Q_L as discussed below.

4. Matching

Both the low and high frequency equivalent circuits are shown in Fig. 3. The high frequency losses of the quarter wavelengths, have been accounted for by an equivalent resistance, R_{RC} which is in series with the inductor

at f_H . If $R_{||} > X_L'''$ then $R_{RC} \approx \frac{(X_L''')^2}{R_{||}}$ and $Q_H \approx \frac{R_{||}}{X_L'''}$.

The open quarter wave is approximated by its equivalent capacitance at f_L . Lossless cable of characteristic impedance Z_0 is assumed. All distances are in wavelengths at f_L . All low frequency losses are accounted for by resistor R_L .

The equations given in Fig.2 allow the values of capacitors C_{1H} , C_{2H} , C_{1L} and cable D1 to be estimated with reasonable accuracy. In these equations Z_{IN} is the input impedance at zero phase angle - normally 50 ohms, Z_0 is the characteristic impedance of cable D1. X_L''' is the inductive reactance of the coil at f_H . Z_a and ϕ_a are the impedance and phase angle measured at point "a" after all components except C_{1L} and D1 are installed and properly tuned. R_a and X_a are the resistive and reactive components of this impedance. Q_L and Q_H are the quality factors at f_L and f_H . At f_L , the circuit is tuned to the characteristic impedance of the cable, D1. With proper circuit design the addition of cable D1 and capacitor C_{1L} will not affect the high frequency tuning provided D1 is not an odd multiple of a quarter wavelength at f_H . All capacitors, inductors and cables are considered ideal. Losses are accounted for by R_{RL} , R_{RH} and R_{RC} .

In the probe designed below, $f_L = 14.1$ MHz, $f_H = 56$ MHz, $Z_{IN} = 50$ ohms, $Z_0 = 50$ ohms, $D_{D1} = .063$, $R_{||} = 2200$ ohms, $X_L' = 22.7$ ohms, $X_L''' = 98.7$ ohms, $Z_a = 108$ ohms, $\phi_a = 89.3^\circ$. No lumped resistors were used i. e., The equivalent resistances, R_{RL} , R_{RH} , and R_{RC} result from distributed

losses only. For these data $Q_H = \frac{R_{||}}{X_L} = 22.3$ has been assumed. The calculated values using the equations of Fig. 2 are given in Table I.

Table I. A comparison of the actual component values to the estimated values from the equations of Fig. 2. These values are for the spinning rotor circuitry described in the report.

	Estimated Values	Actual Value
C_{C1H}	33.6	35.2
C_{C2H}	183.0	192.0
Q_H	22.3	24.5
D_{D1}	75.6	73.0
C_{C1L}	24.6	28.0
Q_L	—	36.2
capacitance in pf.	cable length in cm	

Care must be taken to compensate for the residual inductive readings of the vector impedance meter probe when measuring reactances. For our probe this residual impedance is 8.3 ohms $< 90^\circ$ at 56 MHz and 2.3 ohms $< 90^\circ$ at 14.1 MHz. Failure to compensate for these will normally result in very inaccurate measurements of inductance and/or capacitance. The calculated and actual values given in Table I agree relatively well. Capacitance, C_{C2H} , is extremely sensitive to the Q_H as will be discussed in the section. Its larger value indicates that the losses in R_{RC} are less than those in a circuit with $Q_H = 22.3$. Procedures for accurately calculating the Q 's based on experimentally measured values will be discussed in the next section.

III. Circuit Losses

At this point inductors, capacitors and cables have been considered ideal; losses have been accounted for by R_{RL} , R_{RH} and R_{RC} . This treatment is very desirable since all losses can be referred to these components and the actual values of R_{RL} and $R_H (= R_{RH} + R_{RC})$ can be determined after the probe is assembled and tuned.

A quick study of equations 1 and 2 in Fig. 2 will convince one that capacitance, C_{C2H} , is solely determined by circuit losses whereas capacitance, C_{C1H} , is relatively insensitive to these losses.

Rearrangement of equation 2 with $Q_H = X_L'''/R_H$, ($R_H = R_{RH} + R_{RC}$) yields $Q_H = 4\pi^2 f_H^2 X_L''' Z_{IN} C_{C2H}^2 + \frac{X_L'''}{Z_{IN}}$.

Thus an accurate measurement of the values of X_L''' and C_{C2H} will allow an accurate calculation of the actual Q_H after the probe has been assembled and tuned. This treatment refers all losses from the capacitors and inductor to the resistance $R_H (= R_{RH} + R_{RL})$. For example, the measured value of C_{C2H} was 192 pf for $X_L''' = 98.7$ ohms; thus the actual value of Q_H is 24.5.

A similar treatment for Q_L is not as straightforward since the low frequency tuning circuit is bisected at point "a" in Fig. 1 where the open quarter wave is attached. However a segmental approximation can be made which allows the equivalent resistance, R_{RL} (and thus Q_L) to be determined if an accurate measurement of the length of the two cables, D_1 and the quarterwavelength between points "a" and "c" of Fig. 1 is known. This treatment refers all losses from cables, capacitor, inductors, etc. to the resistance, R_{RL} , in series with the NMR coil. If each cable segment, $D_i \ll \lambda_L/2$ and the $\sin^2 \theta_a \approx 1.0$, then the following relationship is valid:

$$Q_L = \frac{X_L' Z_0 (1 + \tan^2 2\pi D_{D1}) (1 + \tan^2 2\pi D_{\lambda_H/4}) [Z_a \left(\tan \frac{2\pi f_L}{4f_H} \right) + Z_0]^2}{(Z_0 - Z_a \tan 2\pi D_{D1})^2 (Z_0 - X_L' \tan 2\pi D_{\lambda_H/4})^2} .$$

D_{D1} and $D_{\lambda_H/4}$ (the cable between points "a" and "c" of Fig. 1) are the actual lengths in wavelengths at f_L that result in the input being tuned to Z_0 ohms. Coaxial cable of characteristic impedance, Z_0 , is also assumed. Z_a is the magnitude of the impedance measured at point "a" of Fig. 1 with D_1 and C_{iL} not attached. X_L' is the inductive reactance of the coil at f_L .

In the probe designed in this report $D_{D1} = .0521 (73 \text{ cm}) D_{\lambda_H/4} = .0592 (83 \text{ cm})$, $Z_0 = 50 \text{ ohms}$, $Z_a = 104.7 \text{ ohms}$, $\phi_a = 88.3$, $X_L = 22.7 \text{ ohms}$. The above formula yields $Q_L = 36.2$.

III. Field Strength, Homogeneity and Signal to Noise

In this section the actual performance of the probe will be discussed. The inductor consisted of 6 turns of No. 14 wire with 10 mm inside diameter and length 12 mm. The inductance was $0.26 \mu\text{H}$ at 14.1 MHz and $0.28 \mu\text{H}$ at 56 MHz.

With 81 watts input at 56 MHz an H_1 field of 10 gauss was obtained, as determined by the time required for a $\pi/2$ pulse on ^1H in $\text{H}_2\text{O}(d)$. With 256 watts input at 14.1 MHz an H_1 field of 40 gauss was obtained, measured similarly by the time required for a $\pi/2$ pulse on ^{13}C in CH_3COOH . These fields satisfy the Hartman Hahn condition for $^1\text{H} - ^{13}\text{C}$ double resonance. The ratio of the H_1 fields in the same inductor can be written⁵ as

$$\frac{H_{1L}}{H_{1H}} \approx \left(\frac{P_L Q_L f_H}{P_H Q_H f_L} \right)^{1/2}$$

P_L and P_H are the input powers at f_L and f_H respectively. With $Q_H = 24.5$ and $Q_L = 36.2$ and the above values $\frac{H_{1L}}{H_{1H}} \approx 4.32$. This ratio is reasonably close to the actual ratio of 3.98. Thus the circuit losses are reasonably accounted for and the Q's are fairly accurate. Conversely, an accurate knowledge of the Q's can allow the Hartman-Hahn condition to be set fairly accurately by simply adjusting the low frequency power level.

(a)

(b)

Figure 4. a) One scan on a .43 g (2×10^{20} spin) sample of natural abundance adamantane using the PENIS experiment without spinning and the probe described in this report.

b) A typical fourier spectrum obtain from coherent averaging of 25,000 PENIS experiments with magic angle spinning at 1.5 KHz. The sample consist of a 0.2 g sample of coal vitrain enclosed in a spinning rotor. Radio frequency fields of 10 and 40 gauss and a cross polarization time of 2 msec were used.

Figure 5. A photograph of the actual probe. Shown in this figure are:

- a) the spinning rotor, b) the probe cover, c) the main probe body and d) the capacitor box containing capacitor C_{1L} . The open $\lambda_H/4$, not shown in this figure, is connected to the cable between the probe and box.

Figure 4 illustrates the typical signal-to-noise obtainable with this probe. The figure shows a natural abundance C^{13} -F.I.D. for one scan on a .43 g ($2 \times 10^{20} \text{ }^{13}\text{C}$ spins) sample of adamantane (no spinning) and a typical coal fourier transform spectrum for a 0.15 g sample with magic angle spinning at 1.5 KHz (25,000 scans). Fields of 10 and 40 gauss are used at $f_L = 14.1 \text{ MHz}$ and $f_H = 56.0 \text{ MHz}$. A commercial 14.1 bandpass filter (down 80 dB at 56 MHz) rendered the 56 MHz signal undetectable at the receiver input. R.F. inhomogeneity was measured at 1% over a 5 mm spherical sample.

IV. Construction

The probe circuitry and inductor were mounted in a spinning rotor probe previously described.⁶ The first quarter wavelength consisted of 71 cm of RG58 c/u cable and a 12 cm, 0.114 inch O.D. copper tube enclosing a teflon coated No. 18 wire (total length of $\lambda/4$ at 56 MHz). The teflon portion was connected to the coil. All other cable lengths were made from RG58 c/u. Capacitors were small Centralab ceramic 7-45 pf trimmers. Capacitors C1L and C2H were made from two such capacitors in series for high voltage protection. Impedances were measured on a HP-4815A vector impedance meter. No eddy current transients⁷ resulting from the long cross polarizing pulse were observed with this probe. Figure 5 shows the actual spinning rotor probe described in this report.

V. Modification of Existing Probes

Probes, whose tuning circuit is similar to the high frequency equivalent circuit of Figure 3, can be simply modified to accept the lower frequency tuning elements. The base of the coil should be grounded and any Q_H -limiting resistor connected in series with the top of the coil. Any Q_L -limiting resistor and the first quarter wave are connected to the coil as shown in Fig. 1.

Since the remaining low frequency tuning elements, i.e., open $\lambda_H/4$, D1 and C1 L, are located a maximum of $\lambda_H/4$ (80-90 cm for $f_H = 56$ MHz), their positions can be far enough from the coil to negligibly disturb the existing geometry of the probe. This feature allows for easy modification of existing low temperature probes, spinning rotor probes and others.

VI. Summary

A design procedure has been presented which explains the design of the circuitry for a spinning rotor double resonance probe. This design has been found useful for large (10 mm i.d.) inductors. Methods have been presented which allow the actual Q's to be determined from experimentally measured parameters. This design allows easy modification of existing probes for operation under double resonance conditions.

Acknowledgement

This work was supported by the U.S. Department of Energy, Office of Basic Energy Sciences, Materials Sciences Division.

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