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An efficient $^{1}H/^{31}P$ double-resonance solid-state NMR probe that utilizes a scroll coil

Christopher V. Grant [a], Siu-Ling Sit [a], Anna A. De Angelis [a], Kelli S. Khuong [b], Chin H. Wu [a], Leigh A. Plesniak [b], Stanley J. Opella [a,*]

[a] Department of Chemistry and Biochemistry, University of California, San Diego, 9500 Gilman Drive, La Jolla, CA 92093-0307, USA
[b] Department of Chemistry and Biochemistry, University of San Diego, 5998 Alcala Park, San Diego, CA 92110, USA

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Abstract

The construction and performance of a scroll coil double-resonance probe for solid-state NMR on stationary samples is described. The advantages of the scroll coil at the high resonance frequencies of ^{1}H and ^{31}P include: high efficiency, minimal perturbations of tuning by a wide range of samples, minimal RF sample heating of high dielectric samples of biopolymers in aqueous solution, and excellent RF homogeneity. The incorporation of a cable tie cinch for mechanical stability of the scroll coil is described. Experimental results obtained on a Hunter Killer Peptide 1 (HKP1) interacting with phospholipid bilayers of varying lipid composition demonstrate the capabilities of this probe on lossy aqueous samples.

Keywords: Probe; Solid-state NMR; Scroll coil; Double-resonance; 31P NMR

1. Introduction

The combination of high resonance frequencies and lossy aqueous samples is problematic for probes that utilize conventional solenoid coils, especially those used to perform solid-state NMR experiments that require large B_1 fields. High magnetic fields, and correspondingly high resonance frequencies are essential in order to obtain the requisite sensitivity and resolution for studies of proteins and other biopolymers. Magnetically aligned phospholipid bilayers (bicelles) have recently been shown to be highly effective at providing a fully hydrated, planar bilayer environment for structural studies of membrane-associated peptides and proteins [1,2]; the only drawback to these samples is that they are very lossy at high frequencies due to the presence of high concentrations of lipids, proteins, and salts in aqueous solution. Similar problems are encountered with hydrated samples of mechanically aligned phospholipid bilayers [3] as well as with the unoriented protein samples used in magic angle sample spinning experiments [4,5]. The analysis of RF-induced sample heating has led to the development of alternative coil geometries, e.g., scroll and other specialized coils, which reduce RF sample heating by lowering the inductance of the coil and minimizing the electric field generated in the sample [4,6–8]. Furthermore, many of these designs have the added benefit of high B_1 homogeneity.

In this article, we describe the implementation of a scroll coil, which consists of a single sheet of copper wrapped concentrically with a sheet of a non-conducting material, in a $^{1}H/^{31}P$ double-resonance probe optimized for solid-state NMR experiments on stationary samples of magnetically aligned bilayers in aqueous solution. The previously reported applications of scroll coil NMR probes have been for $^{1}H/^{13}C/^{15}N$ triple-resonance magic angle sample spinning experiments [4,5]. The low inductance of a scroll coil limits its efficiency at the relatively low frequencies associated with ^{13}C and especially ^{15}N, limiting the impact of $^{1}H/^{15}N$ double-resonance probes for stationary samples;

* Corresponding author. Fax: +1 858 822 4821.
E-mail address: sopella@ucsd.edu (S.J. Opella).

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however, the low inductance is very well suited for the higher ^{1}H and ^{31}P resonance frequencies, even at magnetic field strengths as low as 11.7 T. Because of the relatively small electric field generated within the sample volume of a scroll coil, sample heating is minimized and probe tuning is not significantly perturbed by samples with quite different dielectric properties. As a result, the probe has sufficient flexibility to be used to test pulse sequences on single crystal samples, and then to be used to study aqueous protein samples without replacing fixed capacitors or other tuning elements.

The performance of the $^{1}H/^{31}P$ double-resonance probe is demonstrated with results obtained on a synthetic sample of a pro-apoptotic peptide that targets specific tissue for programmed cell death. These Hunter Killer Peptides (HKPs) bind to cell surface receptors, are subsequently internalized, and then initiate apoptosis by disrupting the mitochondrial membrane [9]. They consist of a protein homing motif connected to a pro-apoptotic sequence containing D-amino acids to confer resistance to proteolytic degradation. Examples have been designed to target tumor vasculature [9], adipose tissue, specifically white fat vasculature [10], prostate tissue [11], and collagen-induced arthritis in mice [12]. The specificity of HKPs toward mitochondrial membrane disruption is crucial for their application as therapeutics since plasma membrane disruption would lead to general toxicity. The conformation and interaction of two HKPs with micelles have been studied by solution-state NMR [13,14]. Because the mitochondrial membrane contains a relative high percentage of anionic phospholipids and HKPs show a preference for binding to vesicles containing anionic phospholipids, it is of interest to examine their interactions with bilayers containing anionic phospholipids. These studies are technically demanding because they require a solid-state NMR probe whose performance is not degraded by the lossy nature of the biologically relevant samples.

2. Results and discussion

Even with many years of experience building double- and triple-resonance probes with solenoid coils, our initial efforts to construct scroll coil probes were stymied by highly unstable tuning and self-resonance frequencies. The lack of stability was found to be mechanical rather than electrical in origin. There is a significant amount of capacitance in the scroll coil design and consequently, small changes in dimensions, such as those resulting from RF heating-induced expansion of components, give rise to significant changes in the observed self-resonance frequency. This necessitated the development of a construction protocol that produces mechanically stable scroll coils.

A scroll coil "blank" was cut from a 0.010 in. thick sheet of oxygen free copper (McMaster-Carr, www.mcmaster.com). The coil blank shown in Fig. 1A is 0.8 cm wide by 6.0 cm long measured between the inside edges of the leads. The polytetraflouroethylene (PTFE) dielectric used

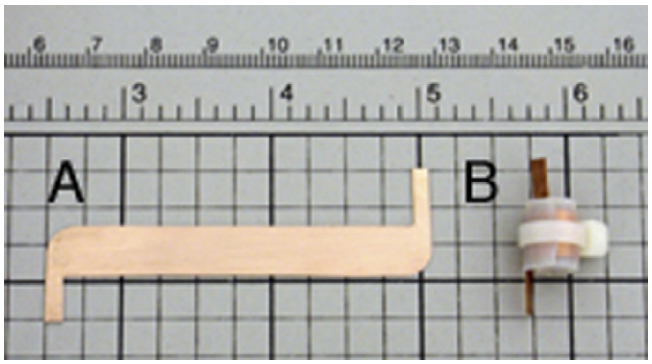

Fig. 1. (A) Copper blank for scroll coil. (B) Five millileter inner diameter scroll coil including the cable tie cinch that provides mechanical stability.

was in the form of adhesive backed tape 0.0115 in. thick by 0.75 in. wide (Saint-Gobain Performance Plastics, www.plastics.saint-gobain.com). The PTFE tape was adhered to one side of the copper, and trimmed to a width approximately 2 mm larger than that of the coil blank to prevent electrical shorting at the edges. This assembly was then wrapped around a cylindrical former with a 5 mm outer diameter; the relatively stiff mechanical properties of the copper sheet means that this process must be performed carefully in order to ensure uniform curvature throughout each turn. The resulting scroll coil (Fig. 1B) was cinched tightly using a nylon 0.141 in. wide 40 lb tinsel strength cable tie (Thomas & Betts, www.tnb.com), and was mechanically stable with a self resonance frequency of 629 MHz measured on a Hewlett-Packard 8753A network analyzer (Agilent, www.agilent.com).

The scroll coil is incorporated into the circuit diagrammed in Fig. 2, which is an up-dated version of the original Cross et al. [15] circuit and is similar to those used in other applications [16–21] The circuit is double-tuned to 500 MHz (^{1}H) and 202 MHz (^{31}P). Two variable capacitors (Polyflon NRP/VC10-12-06A, www.polyflon.com) are used in both the ^{1}H and ^{31}P tuning networks, indicated as C2 and C3 for the ^{1}H channel and C6 and C7 for the ^{31}P channel. The ceramic chip capacitors are from American Technical Ceramics (www.atceramics.com) or Voltronics. (www.voltronicscorp.com) ^{31}P to ^{1}H isolation is

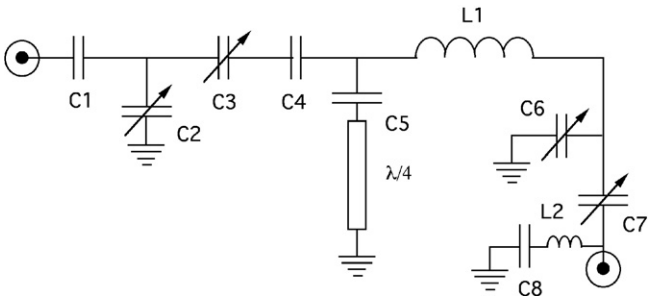

Fig. 2. Circuit diagram for the single-coil double-tuned probe. L1 represents the scroll coil. The values of the capacitors are: C1 = 4.7, C4 = 1.5, C5 = 10.0, and C8 = 8.2 pF.

accomplished with a grounded $\lambda/4$ line. The combination of the length of the $\lambda/4$ line and the C5 capacitor assembly are chosen to resonate the sample coil at the ^{1}H frequency. ^{1}H to ^{31}P isolation is achieved with an LC trap tuned to the ^{1}H frequency using the inductance of a curved lead approximately 1.5 cm in length (L2) in series with capacitor C8. The effectiveness of this trap was optimized by monitoring the transmission between the ^{1}H and ^{31}P ports at the ^{1}H tuning frequency using a network analyzer while varying the value of C8, an isolation of −32 db was achieved. Capacitor C5 was located on the opposite side of the sample inductor (L1) in the initial versions, however the placement shown in Fig. 2 results in superior isolation of the ^{31}P and ^{1}H channels. The ^{31}P to ^{1}H isolation was monitored using the same method as described above, while varying the value of capacitor C5 until a ^{31}P to ^{1}H isolation of −30 db was achieved. Temperature regulation of the sample is accomplished with a ceramic heating element (Doty Scientific, www.dotynmr.com) housed in a custom made glass dewar (New Era Enterprises, www.newera-spectro.com). A photograph of the completed probe is shown in Fig. 3.

The basic performance characteristics of the probe summarized in Table 1 demonstrate that it is very efficient on both channels. The results are reported for a 5 mm sample tube loaded with 160 μL of 85% phosphoric acid at room temperature. Both channels of the probe can be tuned and matched to samples with a wide range of dielectric constants, including air, crystalline solids, and aqueous samples containing significant salt concentrations, using only the variable capacitors. The RF fields generated at typical power levels are given in Table 1; the ^{1}H and ^{31}P channels were tested with continuous irradiation for 30 and 10 ms, respectively, and showed no evidence of arcing or instability. Table 1 also indicates that the RF homogeneity measured by comparing the amplitude of a signal following an 810° pulse to that of a 90° pulse and expressing this ratio as a percentage is >95% on both channels.

Table 1
Summary of probe performance

Channel	Nutation frequency, power	Homogeneity $(A_{810}/A_{90}) \times 100\%$
^{1}H	89 kHz, 44 Watts	>95%
^{31}P	68 kHz, 125 Watts	>95%

The RF heating effects of the scroll coil have been analyzed previously [4]. We assessed sample heating by monitoring the ^{1}H chemical shift of the H6 resonance of Na_5[TmDOTP] (Macrocyclics, www.macrocyclics.com), the sodium salt of the complex between the thulium ion and the macrocyclic chelate 1,4,7,10-tetraazacyclodecane-1,4,7,10-tetrakis(methylene phosphonate) [22,23]. The Na_5[TmDOTP] sample included an additional 70 mM of NaCl so that its dielectric properties are comparable to a "worst-case" lossy aqueous sample, as judged by the power required to achieve a 50 kHz ^{1}H B_1 field in a solenoid coil probe. This allows for a meaningful comparison between the RF heating measurements made using the Na_5[TmDOTP] sample and the RF heating in representative biological samples. The RF heating data are presented in Fig. 4, where the sample temperature determined from the Na_5[TmDOTP] H6 chemical shift is plotted as a function of the average RF field deposition, calculated as the product of B_1^2 and a duty cycle factor. The duty cycle factor is the ratio of the time that the RF irradiation is on to the total duration of the pulse sequence. In this case, B_1 is

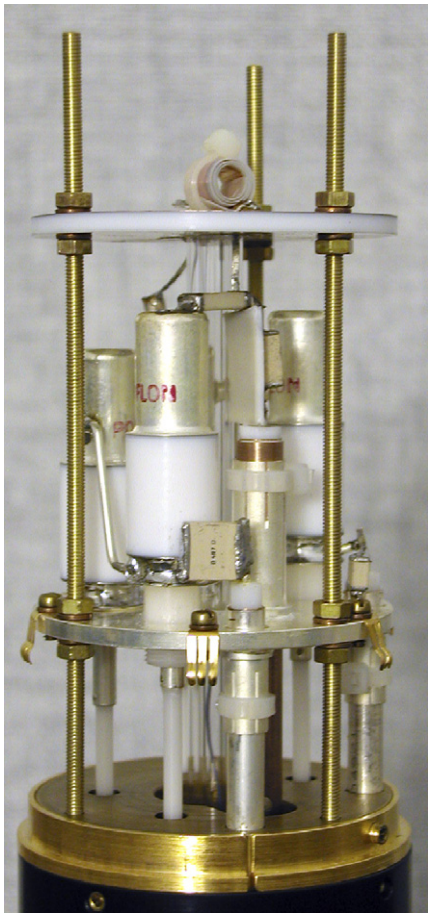

Fig. 3. A photograph of the complete probe assembly.

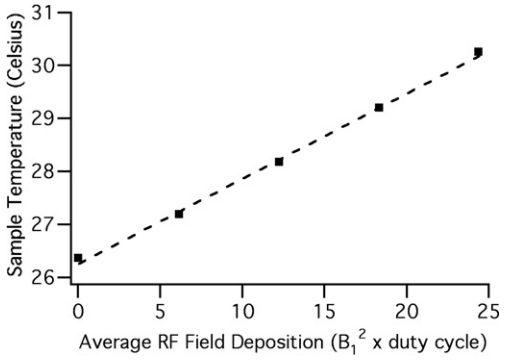

Fig. 4. Sample temperature measured by chemical shifts as a function of the average RF field deposition for a B_1 field of 50 kHz. The dashed line is a linear fit to the experimental data with a slope of 0.16(°C/kHz^2).

set to 50 kHz and four data points were obtained each using an 8 minute pulse train (to ensure thermal equilibrium) consisting of ^{1}H pulses of 2.5, 5.0, 7.5, or 10.0 ms duration with a 1 s delay between pulses, followed by a 90° pulse and a 16 ms acquisition time. As an example, an NMR experiment utilizing 50 ms of ^{1}H irradiation and a 5 s recycle delay with a 50 kHz B_1 would give rise to an average RF field deposition of 25; typical experiments on proteins have RF field deposition values between 4 and 10, and the value is 4.3 for the spectra displayed in Fig. 5. Using the scroll coil, the temperature of the sample is increased by 3.9 °C at an average RF field deposition of 24.4. For comparison, a probe of similar construction, but utilizing a conventional 6 turn 5 mm solenoid coil resulted in a 25 °C temperature increase at the same average RF field deposition (data not shown).

The ^{1}H/^{31}P double-resonance probe was used to study the interaction of the Hunter Killer Peptide HKP1 with magnetically aligned phospholipid bilayers in two different preparations. The first sample contains the zwitterionic lipids 1, 2-di-*O*-hexyl-*sn*-glycero-3-phosphocholine (6-*O*-PC) and 1, 2-dimyristoyl-*sn*-glycero-3-phosphocholine (DMPC). The second sample includes about 20% anionic long-chain lipids prepared with a 20:80 molar ratio of 1, 2-dimyristoyl-*sn*-glycero-3-[phospho-rac-(1-glycerol)] sodium salt (DMPG) and DMPC (Avanti Polar Lipids, www.avantilipids.com). Fig. 5A contains the one-dimensional ^{31}P NMR spectrum obtained for the DMPC/6-*O*-PC sample at its optimal alignment temperature of 40 °C. Fig. 5B contains the spectrum obtained following the addition of approximately 2 mg of HKP1 to the sample. Similarly, Fig. 5C and D compare the DMPC/DMPG/6-*O*-PC samples in the absence and presence of 2 mg of HKP1, respectively, at a temperature of 34 °C, the experimentally determined optimal alignment temperature for this mixture of lipids. These initial results demonstrate that HKP1 disrupts bilayer formation for both lipid compositions. The differences in the ^{31}P NMR spectra (Fig. 5B and D) obtained in the presence of HKP1 indicate that the resulting samples may be significantly different from one another. Included here to demonstrate the probe performance on actual samples, further studies are being pursued to characterize the interactions of these peptides with phospholipid bilayers.

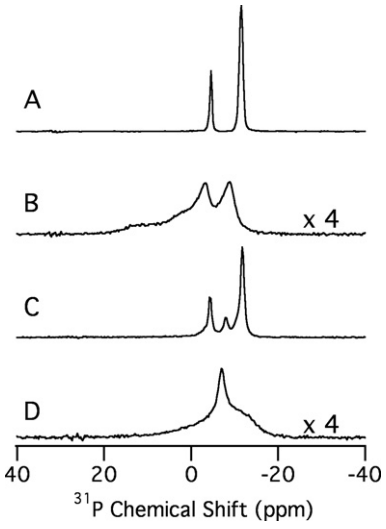

Fig. 5. Experimental ^{31}P NMR spectra of the magnetically-aligned phospholipid bilayer samples. (A) DMPC/6-*O*-PC. (B) DMPC/6-*O*-PC with HKP1. (C) DMPC/DMPG/6-*O*-PC. (D) DMPC/DMPG/6-*O*-PC with HKP1. The vertical scale of the spectra in (B and D) has been increased by a factor of 4 compared to that in (A and C).

3. Conclusion

A ^{1}H–^{31}P double tuned probe was built based on a scroll coil sample inductor. A protocol was established to construct a stable scroll coil using a teflon dielectric and a cable tie tensioning device, or cinch, to stress the structure thus improving dimensional and mechanical stability. A well-established tuning circuit was used and optimized for the constructed coil resulting in a highly efficient, and thus highly sensitive [24], probe. The scroll coil offers the significant advantage of reduced sample heating because of the low electric field component developed within the coil. A useful side effect of this is that the ^{1}H resonance frequency shift and Q factor reduction upon the introduction of a high dielectric sample, such as a bicelle sample, are minimized. This allows the probe to easily tune to a variety of samples without a substantial reduction in probe performance. Furthermore, the scroll coil allows for a substantial reduction in sample heating by RF power deposition. The coil presented in this work is expected to elevate the sample temperature by less than 3.9 °C even for high duty cycle solid-state NMR experiments with samples containing a significant amount of salt.

Two minor drawbacks were identified while using this scroll coil probe for experiments. The first of which is that magnet shimming is somewhat more difficult with a scroll coil probe compared to an equivalent solenoid coil probe. However, we found that acceptable shimming (0.1–0.2 ppm H_2O line width using a standard room temperature shim coils) could always be achieved for the types of samples presented here. Second, the tuning frequency of the scroll coil probe shifts slightly more as a function of temperature than for a solenoid coil, the likely result of a capacitance change in the coil as thermally induced dimensional changes take place. This makes it necessary to retune the probe when the sample (and thus coil) temperature is changed by as few as 2–3 °C. Long-term stability of probe tuning was easily accomplished with the use of a standard variable temperature control system.

Preliminary results were presented for the interaction of HKP1 with bicelles consisting of both zwitterionic lipids and a mixture of zwitterionic lipids and lipids with negatively charged head groups. Regardless of bicelle composition, the HKP1 (in concentrations suitable for solid-state NMR studies of the peptide) disrupted bicelle formation. Although the nature of the resulting peptide lipid

complexes that are formed can not be determined by ^{31}P NMR analysis alone, the data indicate that the two different bicelle compositions gave rise to different interactions, as judged by the significant differences in the spectra.

4. Experimental

The ^{31}P NMR experiments were performed on a spectrometer that consisted of a Chemagnetics console interfaced to a Magnex 500/89 wide-bore magnet. The ^{1}H frequency is 499.9 MHz. All samples were equilibrated in the magnetic field at constant temperature for at least 30 min prior to the NMR measurements. One-dimensional ^{31}P NMR chemical shift spectra were obtaining by direct excitation with a single pulse with radio-frequency field of 28 kHz. All ^{31}P NMR spectra were obtained with a 6 s recycle delay, 128 scans with a 10.24 ms acquisition time. Continuous wave ^{1}H decoupling was accomplished using a B_1 field strength of 50 kHz during the acquisition time. Temperatures of 40 and 34 °C corresponded to the optimal alignment of the liquid crystalline phase in the absence of peptide for the DMPC/6-*O*-PC and DMPC/DMPG/6-*O*-PC bicelles [25,26], respectively. All chemical shifts are referenced to the ^{31}P resonance of external 85% phosphoric acid, which is assigned to 0 ppm at room temperature. NMR spectra were processed using nmrPipe [27] and visualized using IGOR pro (WaveMetrics, www.wavemetrics.com).

The HKP1 peptide was synthesized as described previously [13]. The samples used in the NMR experiments without peptide and samples containing HKP1 were prepared for a total lipid concentration cL = 28% (w/v) and lipid molar ratios: $q = [\text{DMPC}]/[6\text{-}O\text{-PC}] = 3.2$; and $q = [\text{DMPC}] + [\text{DMPG}]/[6\text{-}O\text{-PC}] = 3.2$, [DMPG]:[DMPC] = 20:80. The DMPC/6-*O*-PC and DMPC/DMPG/6-*O*-PC bicelles were prepared as previously described [1,2] by preparing an aqueous solution containing the short-chain lipid (6-*O*-PC) and then adding this solution either to a dispersion of the long-chain lipid (DMPC), or to a dispersion of the long-chain lipids (DMPC) and (DMPG). The resulting solution was vortexed and freeze/heated (0/40 °C) a few times and then allowed to equilibrate to room temperature. Samples of HKP1 in bicelles were prepared by adding a 160 μl volume of the bicelles directly to about 2 mg of the purified, lyophilized polypeptide. Sodium hydroxide was used to adjust to pH 6. In all cases, the bicelles were non-viscous between 0 and 10 °C. A 1.5 cm long, flat-bottomed NMR tube with 5 mm outer diameter (New Era Enterprises, www.newera-spectro.com) was filled with the sample solution, using a pre-cooled glass pipette, at 4 °C. The NMR tube was sealed with a tight-fitting rubber cap, pierced with a thin syringe to remove residual air from the sample and create a tight seal.

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