SMALL SAMPLE LOW-E PROBE FOR SOLID STATE NMR OF PEPTIDES AVAILABLE IN LIMITED QUANTITIES

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Introduction

A solid-state probe for the study of small quantities of oriented membrane samples was developed at the NHMFL for use at the Forschungszentrum Karlsruhe (FZK) (Fig. 1). The low-E ^{15}N-^{1}H probe is designed for PISEMA studies in a 600 MHz field of peptides that are not available in small quantities, and so would not be efficiently observed in a larger coil. The smaller 8.5×3×9 mm flat-coil (Fig. 2) also allows for the use of larger B_1 fields with less applied power. The basic characteristics of the probe have been compared to two probes supplied by Bruker Biospin: a single solenoid "BioPE" probe and an "Efree" probe based on the same low-E cross-coil design that was developed at NHMFL. Heat deposition is similar in the two cross-coil probes, and is about 30% that of the solenoid.

Fig. 1. Probe head showing tuning network.

Experimental

The probe was designed, constructed and tested at the NHMFL in a collaborative effort between NHMFL and FZK. The probe was tested to a ^{1}H B_1 of 100 kHz for 50 ms, and a ^{15}N B_1 of 90 kHz for 3 ms. It has a sample temperature range of –40°C to +100°C. Samples of DMPC bilayers with varying salt concentration were created at FZK. All NMR experiments were conducted at the FZK. To test sample heating, 70 W of ^{1}H power was applied to the probe and the 90° pulse time was measured. For a lossless sample, the pulse time for both Bruker probes was measured to be 5.1 µs, while the NHMFL probe time was a slightly shorter 4.6 µs. For the 80 and 240 mM samples, the pulse length increased for all three probes, indicating that RF power was deposited into the sample. RF heat deposition was determined from the increase in pulse time using the method described in [1].

Results and Conclusion

The solenoidal probe deposits approximately 3 times more heat into the sample than either the NHMFL or Bruker low-E probes, as indicated in Table 1. Both low-E probes have approximately the same heat deposition coefficient, which is not surprising since both probes were based on the same design principles. It is clear that low-E design is a better choice for dielectrically lossy biological samples, especially for the heat-sensitive peptide preparations.

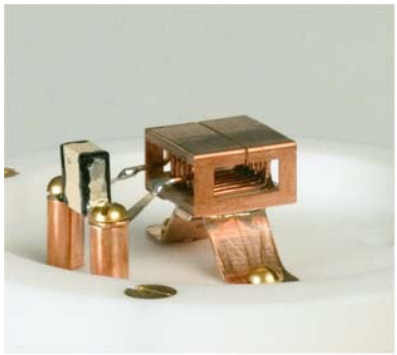

Fig. 2. Expanded view of coil.

Table 1. Comparison of heat deposition into DMPC sample at 70 W of ^{1}H.

Conc. NaCl (mM)	**Sample heat deposition (mW/kHz^2)**		
	NHMFL low-E probe	Bruker Efree™ probe	Bruker BioPE™ solenoid probe
80	3.2	3.5	9.9
240	7.8	7.3	22.7

Acknowledgements

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References

[1] Gor'kov, P.L., *et al.*, J. Magn. Reson. **185**, 77-93 (2007).