COMPARISON OF SAMPLE HEATING IN SOLENOIDAL AND LOW-E NMR PROBES

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Introduction

Solid state NMR spectroscopy experiments expose the sample to high levels of high frequency electromagnetic field. Just as in a microwave oven, these fields will heat the sample. Excessive heating may distort the NMR spectrum or even damage the sample. Recently introduced Low-E coil designs may reduce the level of heating [1]. A convenient way to quantify the improvement is to compare the values of RF input power required to produce identical 90° pulse lengths in a lossy biological sample (P_{bio}) and in a non-lossy reference (P_{nl}). The difference in input power $P_{heat} = P_{bio} - P_{nl}$ must then end up heating the biological sample. The same measurement can be done more precisely by comparing 90° pulse lengths (or RF fields $f_1 = \omega_1/2\pi$) measured with a lossy sample (τ_{90bio}) and with a non-lossy reference (τ_{90nl}) at the same power level P_{in}. Holding P_{in} constant eliminates the effect of errors that often accompany measurements of pulsed RF power. In the latter case, the fraction of RF input power diverted to sample heating can be expressed as

$$\alpha_{heat} = \frac{P_e + P_m}{P_{bio}} = 1 - \frac{\tau_{90nl}^2}{\tau_{90bio}^2} = 1 - \frac{f_{1bio}^2}{f_{1nl}^2}$$

The loss factor α_{heat} alone should not be used to compare sample heating in different probes with different coils, or at different frequencies. A less efficient probe circuit with the same loss factor will deposit more power into the sample while achieving the same RF field f_1. For a heat sensitivity sample, a spectroscopist should choose a probe with minimal heat deposition $q_{heat} = P_{heat} / f_{1bio}^2$, which is the cost of producing the required RF field f_1 in terms of power P_{heat} deposited in the sample. The heat deposition coefficient q_{heat} combines sample loss factor α_{heat} with probe circuit power efficiency η in the absence of sample loss:

$$\eta = \frac{f_{1nl}^2}{P_{in}} \qquad q_{heat} = \frac{\alpha_{heat} P_{in}}{f_{1bio}^2} = \frac{\alpha_{heat}}{1-\alpha_{heat}} \frac{P_{in}}{f_{1nl}^2} = \frac{\alpha_{heat}}{(1-\alpha_{heat})\eta}$$

Experimental

Tests of sample heating by the ^{1}H field were conducted in five different probes designed for the same sample size, but operating in magnets at three different fields. The results are summarized in Table 1. An applied ^{1}H power of 65 W was used in all cases. The 90° pulse length was measured for a non-lossy sample of pump oil and a typical biological sample described in [2]. Low E resonators [1] using gaps of 6.4 (coil I) and 9.9 mm (coil II) were both tested, because the larger gap can produce a somewhat larger f_1 without arcing.

Field (MHz)	Probe	τ_{90nl} (μs)	τ_{90bio} (μs)	α_{heat}	η (kHz^2/W)	q_{heat} (mW/kHz^2)
400	4't solenoid	4.75	7.10	55 %	42.6	**28.7**
600	4't solenoid	5.05	9.55	72 %	37.7	**68.2**
600	low-E coil I	5.10	5.48	13 %	37.0	**4.0**
600	low-E coil II	5.75	6.23	15 %	29.1	**6.1**
900	low-E coil II	6.88	8.45	34 %	20.3	**25.4**

Table 1. Sample heating comparison between ^{1}H–^{15}N probes with low-E resonator and double-tuned 4-turn solenoid.

Discussion and Conclusions

The reduction in sample heating is illustrated by comparing the low-E probe to a conventional 4-turn solenoid at 600 MHz. RF power deposition in the biological sample has been reduced by 17 times in low-E coil I and by 11 times in low-E coil II. Coil I, with its smaller gap, is more efficient and produces the least sample heating. One might expect the ^{1}H power efficiency η of the low-E probe to be much worse than that of the solenoid due to the 3.3 times larger volume of the low-E resonator. However, the superior efficiency of the single resonance ^{1}H circuit in the low-E probe makes up for the difference in filling factor. As shown in Table 1, the low-E probe with coil I has the same ^{1}H efficiency as the double-tuned solenoid. The low-E probe with coil II is somewhat less efficient, which can be attributed to using more chip capacitors in the gap of a resonator. The amount of ^{1}H power dissipated in the bilayer sample in a 900 MHz low-E probe is 4 times larger than in its 600 MHz counterpart, yet it is still just 37% of that dissipated in the 4-turn solenoid at 600 MHz. To date, the aligned samples studied in the 900 MHz low-E probe have exhibited only minimal damage after demanding experiments. Yet because of the large rise of RF absorption at 900 MHz, it is important to invest in further refinements to the low-E coil for use at ultra-high NMR fields.

References

[1] Gor'kov, P.L. *et al.*, J. Magn. Reson. **in press**.
[2] Gor'kov, P.L. *et al.*, J. Magn. Reson. **181** (2006) 9-20.

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