Radiation Damping Observed in Hyperpolarized 13C-1-Pyruvate at 14.1 T

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Introduction: Metabolic MR imaging of hyperpolarized substrates, such DNP- polarized [1-^{13}C] pyruvate, raises the question of whether radiation damping (*1*) is to be expected. Our analysis (given below) suggests it is unlikely to be noticed in metabolic studies at 3.0 tesla under normal conditions; we nonetheless report its observation at extremely high field – 14.1 tesla-- and with with a highly efficient probe.

Experimental Methods: Polarizer and compound Samples of 32µl [1-^{13}C] pyruvic acid (Isotec) and 15mM OX63 trityl were polarized and dissolved using a Hypersense DNP polarizer (Oxford Instruments), as described elsewhere (*2*).
NMR measurement: Measurements were performed on a Varian INOVA 14.1T spectrometer (Varian, Palo Alto, CA) equipped with an 8mm ^{13}C/^{31}P dual-tuned probe. Prior to sample dissolution, shimming was performed manually with a non-hyperpolarized [1-^{13}C] pyruvate solution of the same concentration. Upon dissolution, the hyperpolarized pyruvate solution was quickly injected manually via a syringe into an NMR tube and filled to a predetermined level (the same as that used in shimming); the NMR tube was then placed inside the spectrometer. The time between dissolution to the start of measurement was ~10s. NMR data were acquired using a pulse-acquire sequence at 3.5s interval for ~3 minutes by applying a small tip angle (5°) excitation pulse. WALTZ 16 decoupling of protons was performed continuously during the measurement. Data sampling duration was 2s. All measurements were performed at about 37 □. For calculation of the damping coefficient, the pulse power was measured with a spectrum analyzer (Agilent PSA 4400) (zero sweep width, center frequency at Larmor, video trigger) at the probe input; the probe return loss (Figure 1) was also measured by network analyzer (Agilent 5071)

Results and Discussion: Figure 2 shows the time course of the lineshape, at intervals of 10.5 sec, The linewidth is clearly seen to diminish; over this time interval the value drops from 5.8 Hz to 2.4 Hz. Figure 3 shows a parallel experiment in which all conditions were unchanged, except for the return loss of the probe, which was intentionally mis-tuned, from its customary value below - 20 dB to a value in the neighborhood of 0 dB. The horizontal scale of the spectra is expanded, to show the constancy of the linewidth over time, at a value of 1.7 Hz. Lacking an independent measure of polarization for this trial, we estimate it at 7% using the value of the radiation damping constant given in terms of B1 per square root of power absorbed by the probe (*3*), i.e.

$$k = \gamma \frac{\omega V M_0 B_{1x}(1)^2}{4R} \qquad [1.]$$

--where B_{1x} is the RF field strength per unit current in the lab frame, M_0 is the fractional polarization of the sample, *V* is the sample volume, *R* is the resistance of the coil with sample, but without source loading, and other symbols have their usual meaings. A more elaborate simulation, using the Kirchhoff-Bloch equations (*1*) gives an estimated value of 8%. An RF power of 165 W, applied to a the probe at near-perfect match gave a nutation of π/2 in 10 µs. In a separate experiment, with polarization measured at 5% by C2 doublet asymmetry(*4*) the Bloch-Kirchhoff simulation predicts a damping linewidth of 2.8 Hz, and we measure 2.7 Hz. Such good agreement may be fortuitous. Finally, given that a highly efficient volume coil (*2*) -- used for metabolic studies of small animals *in vivo*, at 3.0 T -- will typically require a pulse of width of ~ 1 ms for a nutation of π/2 at 100 to 200 W applied transmitter power, it is apparent from Eq. 1 that the linewidth due to to radiation damping in such a coil will not exceed about 0.2 Hz. Given the typical line broadening due to static field inhomogeneity, and relaxation, this contribution may be observed, but is unlikely to prove important in clinical or preclinical studies.

References:

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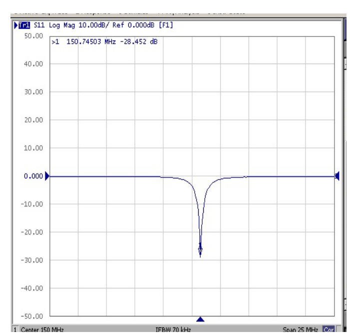
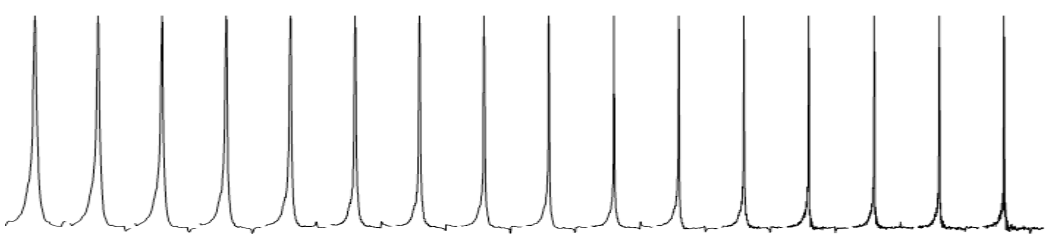


Figure 1: Reflection coefficient of carbon probe, showing return loss below -28 dB.



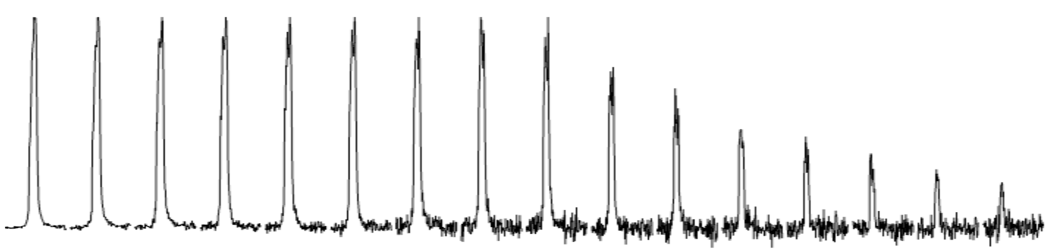


Figure 2: (above) Time course of proton decoupled lineshape of 1-^{13}C in series of 5 degree tips, showing gradual diminution of radiation damping over 3 minutes.
Figure 3 : (below) Same as Figure 2 but with probe detuned to ~ 0 dB return loss.