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# Suppression of ring-down in noise spectroscopy

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**Abstract**

Suppression of systematic noise artifacts which complicate the understanding of broadline NMR spectra remains a difficult problem. Many strategies for canceling noise have evolved; many appear designed for very specific hardware implementations. In this paper we address the problem of artifact suppression in noise-based (stochastic) NMR, where low power pulses applied at high duty cycle are used to probe the spectral frequencies found in an NMR or NQR experiment. While typical peak powers are reduced by four to six orders of magnitude as compared to conventional NMR experiments, this power reduction corresponds only to an approximate halving of the idealized electronic ring-down in a tuned circuit; where other systematic sources of noise contribute, the recovery time advantage of noise spectroscopy may be larger or smaller. We suggest a simple experimental modification which exploits the linear response properties of nuclear spins in the presence of small rotations to demonstrate how ring-down—from whatever source—can be substantially eliminated as a problem in noise spectroscopy. This should provide substantial improvements in quantitation and lineshape measurements in NMR spectra of many solid state systems.


## 1. Introduction

Modern NMR spectrometers routinely exploit the many advantages of the Fourier transform (FT) method, where strong pulses are applied to a spin system and the evolving magnetization is sampled in a free induction decay (FID). Nonetheless, our recent experience suggests that there remain many applications throughout magnetic resonance where the *spectrum is faithfully reproduced* only where we forgo the many advantages of strong pulses which induce a nonlinear spin response and instead return to the linear-response limit wherein NMR spectra are derived from weak-field excitation—as was common prior to the triumph of modern FT methods. So as to retain the Fourier advantages in sensitivity, these weak fields are applied incoherently and in a continuously repeating fashion, with rf pulses rapidly alternating with short sampling windows over the seconds or hours required to attain the desired signal-to-noise ratios [1,2]. The advantages of one-dimensional noise-derived spectra are particularly desirable where the spectra of interest are broad, cover a broad range of shift dispersion, involve heterogeneous dynamical modes spanning a broad range of correlation times [3], or arise from quadrupolar nuclear spins [4], where strong, pulsed rf fields may not be available which can provide undistorted, quantitatively accurate spectra. In our recent experience, noise spectroscopy appears capable of solving many of the problems associated with accurate, quantitative NMR while maintaining the sensitivity advantages of time-domain methodologies. Nonetheless, much of the potential claimed for noise spectroscopy, and in particular the promise that arbitrarily broad bandwidths can be accessed, still cannot be routinely demonstrated. In this paper we suggest a simple rethinking of the excitation sequences used in stochastic NMR which provides a substantial improvement in the quality of noise-based NMR spectra by reducing the influence of ring-down on detected signals. These improvements are easily achieved and should therefore greatly extend the reach of these methods. We begin with a review of noise-based methods, continue by discussing the limitations on these methods encountered in typical implementations, and finally introduce and demonstrate our experimental approach to ring-down suppression.

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## 2. Discussion

### 2.1. Noise pulse sequences

Noise-based NMR experiments which operate in the weak-pulse limit [1–5] are characterized by a system response linear in the experimental input, so that the experiment yields the normal spectrum. Multidimensional NMR experiments can be derived from the more general and quite important set of applications where no such restriction is applied [6–10]. Our work in this paper primarily affects on the above and the question of how one might observe the spectrum of a nuclear spin species in a sample which faithfully reproduces, both in the resonance frequency and in intensity, the distribution of resonance frequencies found in the sample.

In noise spectroscopy, it is conventional to treat the system being probed as a black box responding to a stimulus [8]. Noise-based NMR experiments are most conveniently carried out where the input consists of a series of discrete, equally spaced pulses $P(l)$ applied at low power (typically $-10$ to 10 dBm, or to within a factor of 10 of 1 mW, at the probe). Correspondingly, the output response is conveniently represented by discrete samples of the magnitude and phase of the excited transverse magnetization, $M(l)$—ideally, at the instant each pulse ends, with successive points separated by the spacing between the beginnings of any two pulses, $\tau_{sp}$. For most one-dimensional spectroscopic applications, a family of broadband sequences known as maximal length binary sequences (MLBSs) is particularly robust [11–13] and faithfully reproduce the absorption spectrum over broad bandwidths determined not by the strength of the rf field but by the frequency with which pulses are applied. Under these conditions, all the coevolving components of transverse magnetization excited by any of the pulses in the sequence $P(l)$ can be separated, recorrelated, and rephased to yield the familiar free-induction-decay by cross-correlation of the known input pulse sequence $P(l)$ with the complex magnetization $M(l)$, which can be carried out mathematically via

$$fid_{R,I}(j) = \Sigma\, P(i)\, M_{R,I}(i+j), \tag{1}$$

where $fid_{R,I}(j)$ is the $j$th complex data point in the free-induction-decay, $M_{R,I}(i+j)$ is the complex magnetization response measured $j$ sampling intervals after the $i$th pulse, and $P(i)$ is a record of one period of the continuously repeated sequence of applied pulses. For the MLBS sequences, $P(i) = 1$ or $-1$ for all $i$ (representing 0° and 180° phase shifts, respectively), though for other sequences it may be more involved. The index $(i+j)$ is cyclic with period $J$, corresponding to the number of the excitation pulses in one cycle through the pseudo-random sequence; thus, when $(i+j) \geqslant J$, $(i+j)$ is replaced by $(i+j)-J$.

In principle, this experimental scheme holds out the prospect of unlimited excitation bandwidths, as the spectral region excited without distortion is proportional to the inverse of the length of the excitation pulses, $\tau_{pul}$, and not the strength of the irradiation field, $\omega_1$. (Of course, the product $\omega_1\tau_{pul}$ corresponds to the flip-angle $\alpha$, so that the pulse strength does help determine the *sensitivity* of the experiment.) In practical applications, however, this exciting possibility is difficult to attain. Experimentally, a third time is relevant to the problem of the detectable bandwidth—a time which we will call $\tau_{rec}$, indicating the time it takes for the electronic circuitry to recover from the application of a pulse to the point where the signal can be detected. The ring-down may arise from any number of sources; ultimately, however, it can be no shorter than the time it takes for the tuned electronic circuit to "ring-down" from the pulse (though it is, more typically, somewhat longer). The ring-down time of the probe (defined as the $1/e$ time constant for the disappearance of the pulse in the tuned circuit) is ideally limited by $1/\Delta\omega$, where $\Delta\omega$ is the electrical bandwidth of the tuned circuit. In solid-state probes $\Delta\omega \sim 2\pi \times 1$ MHz is not unusual, so that the ring-down time constant is on the order of 160 ns. Where the pulses are applied at levels on the order of 1 V, however, and the signals to be detected are more typically detected in the $n$V–μV scales, 20 or more time constants may be required before the signal and noise are comparable in size. Due to the exponential fall-off of the ring-down voltages, while the rf power in each pulse is decreased by $10^{-4}$–$10^{-6}$ in noise experiments (as compared to conventional NMR), the ring-down is shortened by no more than a factor of two in noise experiments.

Of course, there are many other sources of ringing in the receiver which need not be associated with the tank circuit; in the probe itself, capacitors may "ring," acoustic modulations may be induced in the probe materials, and mechanical instabilities lasting many microseconds are also possible. Even once the signal has safely exited the probe, other receiver elements may introduce their own delays. This vast array of possible problem areas may account for the repeated discussions of methods to avoid its effects, particularly in applications to broadline NMR spectroscopy [14–22].

From whatever the source, however, the ring-down effect is problematical, as it obscures lineshapes and makes accurate quantitation impossible. Any delay in the accumulation of the signal as we wait to avoid the unwanted ringing introduces a delay between the pulse and the sampling of the magnetization; this delay is responsible for, at minimum, a first-order phase error in the spectrum which results after cross-correlation and Fourier transformation. Where the noise associated with the ring-down cannot be eliminated, it further contributes a rolling baseline in the spectrum. Finally, $\tau_{rec} + \tau_{pul}$

also would appear to provide a lower bound to $\tau_{sp}$, and therefore to provide an upper bound to the bandwidth in frequency space which can be faithfully sampled in the experiment. Though we have previously demonstrated a method for bandwidth extension which partially mitigates this problem, the short-time fidelity of the free-induction-decay is nonetheless ultimately determined by the level of systematic noise which is sampled and saved as part of the signal record $M(k)$. It is not our intention to discuss hardware strategies for minimizing these experimental blemishes; our aim, instead, is to discuss its impact on noise experiments and present a strategy which requires no hardware modifications and yet seems to systematically minimize its effects, whatever the source.

In most applications of noise spectroscopy to date, it has been assumed that the input sequence $\mathscr{I}(t)$ is one-to-one with the pulse sequence $P(l)$. In fact, no such assumption is required. The input to the nuclear spins is a sequence of rotations, each characterized by a rotation angle ($\alpha$) and phase. For nuclear spins interacting with an rf field, the transformation between the pulse sequence and $\mathscr{I}(t)$ is therefore many-to-one, as there may be many ways of generating the same rotation which use different rf irradiation sequences. This is particularly obvious where the pulse sequence consists of infinitesimal rotations, because for small flip-angle pulses *each rotation commutes with all prior and subsequent rotations*, so that any arrangement of the same set of pulses generates the same input to the spins. In contrast, most efforts to eliminate ring-down proceed from an assumption of linearity, so that the decay of the electronic response in the tuned circuit, as well as other sources of ring-down in the receiver, depend primarily on the phase or amplitude of the *last pulse applied*. While this assumption is difficult to verify directly, it has been shown experimentally that a short pulse of inverted phase (of length approximately equal to the ring-down time constant of the tank circuit) can largely eliminate ring-down [15], and that the ring-down from longer pulses is primarily associated with the last pulse in the sequence. As a result, it should be possible to phase cycle the input pulse sequence $\mathscr{I}(t)$ to vary the relative phases and amplitudes of the signal with respect to the systematic noise due to ring-down, of whatever origin, in a predictable fashion—and thereby to design experiments to coherently average together the former while substantially suppressing the latter. Under these conditions the ring-down need not be eliminated entirely, but instead only reduced to a level sufficiently low so that the combination of the ring-down plus or minus the signal from the nuclear spins can be faithfully digitized in the NMR spectrometer. As the signal appears with effectively equal mean amplitude throughout the data set $M(j)$, the dynamic range requirements are substantially reduced from that of traditional NMR spectroscopy.

### 2.2. Using composite rotations

Eq. (1) describes the method by which we transform a noise interferogram (the output of our experiment, $M(j)$), into the more traditional free-induction-decay. Where each event in the excitation input $\mathscr{I}(t)$ corresponds to a series of pulses, our understanding of the input $P(l)$ against which we cross-correlate the output $M(j)$ must be reinterpreted. With respect to the nuclear spins, the input is the net propagator for the sequence of pulses. On the other hand, other system responses—in particular, the unwanted ring-down—respond differently to the input sequence. Thus our goal is to design inputs which enhance the desired spectrum, while eliminating systematically interferences which would normally result in increased noise in the final spectrum. In this paper we focus only on simple composite pulse sequences which demonstrate the efficacy of the method. In other applications more complex composites may also be imagined.

Consider a series of rotations $P(l)$ where each rotation consists of two discrete pulses of rotation angle $\alpha$, but where the first is applied along the $x$-axis of the rotating frame and the second appears along $y$. The net rotation achieved at the end of the sequence corresponds to the vector sum of the two rotations and corresponds to a flip-angle of magnitude $\sqrt{2}\alpha$, with rotation axis midway between the $x$- and the $y$-axes. Contrast this with the rotation corresponding to pulses of the same magnitude applied first along the $x$-axis, and next along the $\bar{y}$-axis. Under this pair of pulses, the net rotation is of identical magnitude—but about an axis midway between $x$- and $\bar{y}$-axes—and thus rotated by 90° with respect to the first pair of pulses, while the phase of the last pulse, which we assume determines the ringing, is shifted by 180°. Alternatively, one could apply the same pair of pulses but in alternating order—$y$ followed by $x$—and the resulting signal would be identical, while the ring-down effect would be rotated by 90°. Thus the different responses of the desired signal ($S$) and undesired noise ($N$) components of the detected response can be manipulated differently under a sequence of pulses. As a result, careful choice of the pulse sequence can isolate the signal while minimizing the noise; the strategy is similar to common phase-cycling methods applied in traditional Fourier methods, where the transient response can be improved by spin-temperature alternation (as in cross polarization) or phase-cycling of echo pulses, generating the same-phased echo from different pulse sequences. Where our efforts differ is in the expectation of improved spectral fidelity, as the noise-based sequences we describe involve no long delays for spin evolution—and thus are less impacted by, for example, distortions due to short $T_2$ times.

### 2.3. Removing the effect of probe ring-down: combining signals

Even where the desired signals and undesired noise are excited with multipulse rotations as described above, the distinction between the two is unusable in any given experiment. Only by combining the results of multiple experiments is it possible to distinguish between the two. In this section we describe how appropriate combinations might be found. We assume that $\varphi_1$ is the phase of the desired noise signal and $\varphi_2$ that of the ring-down effect generated after a pair of pulses with phase $x$ followed by $y$ (in shorthand, $x(y)$), and consider the predicted phases of the desired initial response signal $S$ and undesired systematic noise $N$ after pulses applied in the two pulse pairs $x(y)$ and $x(\bar{y})$:

| Pulse sequence | Real channel | Imaginary channel |
|---|---|---|
| $x(y)$ | $S\cos\varphi_1 + N$ | $S\sin\varphi_1 - N$ |
| | $\cos\varphi_2 \equiv R_1$ | $\sin\varphi_2 \equiv I_1$ |
| $x(\bar{y})$ | $-S\sin\varphi_1 - N$ | $S\cos\varphi_1 - N$ |
| | $\cos\varphi_2 \equiv R_2$ | $\sin\varphi_2 \equiv I_2$ |

where $R_i$ and $I_i$ represent the real and imaginary data at zero time after the applied pulse. (Of course, each signal as it evolves will generate both real and imaginary components, all of which are predicted by the initial phase described above and a knowledge of the Hamiltonian.) Where we simply combine the signals observed in the two experiments by direct addition of the separately accumulated real and imaginary data sets we find

$$R = R_1 + R_2 = S\cos\varphi_1 - S\sin\varphi_1$$

$$I = I_1 + I_2 = S\sin\varphi_1 + S\cos\varphi_1.$$

Note that the contribution from the ring-down effect is absent, while the signal is peculiarly phased. This latter can be easily corrected, so that the correctly phased signal is observed via

$$S\cos\varphi_1 = 1/2(R + I) = 1/2(R_1 + R_2 + I_1 + I_2)$$

$$S\sin\varphi_1 = 1/2(-R + I) = 1/2(-R_1 - R_2 + I_1 + I_2).$$

Of course, other choices of pulse pairs appropriately combined should generate similar results.

## 3. Experimental

We demonstrate the procedure used to remove the contribution of ring-down to noise spectroscopy in Figs. 1 and 2. Fig. 1 demonstrates how, at least under our experimental conditions, the ringing depends only on the phase of the last pulse applied. In Fig. 1 we show the signal observed in our receiver after application of a pair of pulses with phases $x$ and $y$, each lasting 1 μs and with $\tau_{sp} = 20\,\mu s$. Our homemade probe had $Q > 100$ and operated at a resonance frequency $\omega_0 \approx 2\pi \times 120\,\mathrm{MHz}$. As executed in our lab, most noise experiments consist of pulses applied to the probe at powers $\sim 20\,\mathrm{mW}$ (13 dBm), which are sufficiently gentle so that the job of directing pulses to the probe and signals to the receiver can be carried out by fast SPDT GaAs switches rather than the more standard duplexing circuitry associated with high power rf probes. The switch's transition time is ~10 ns, which is considerably shorter than the rise and fall times of the pulses (usually 50–100 ns). With no particular effort to detune the probe, or otherwise optimize the remaining receiver electronics, the systematic noise caused saturation of our receiver for several microseconds, and its amplitude far exceeded the signal observed throughout our 18 μs long sampling window. We typically chose to observe the signal as soon as possible after the receiver recovered from the pulse—generally, 3 μs after it was gated off. This relatively short delay minimized phase errors associated with sampling long after the pulses end. When the experiment is re-

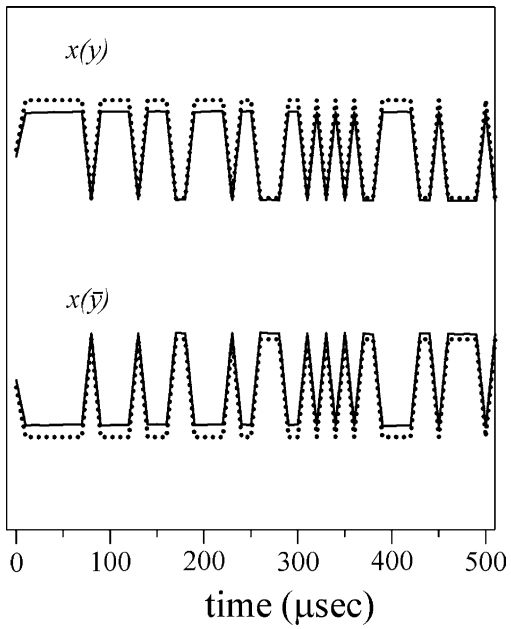


Fig. 1. Noise response data demonstrating the phase shift in the ring-down signal. Using a high-$Q$ probe, the receiver required more than 20 μs to completely recover after a pulse. So as to sample only the ring-down, the receiver was ungated 3 μs after termination of the 2 μs long pulses (4 μs/pulse pair both real (solid) and imaginary (dotted) channels are displayed). (Top) signal response observed after a pair of pulses with phases $x(y)$. (Bottom) signal response observed after a pair of pulses with phases $x(\bar{y})$. In these two data sets the signals are essentially inverted, demonstrating that under these experimental conditions the systematic noise depends primarily on the phase of the last pulse applied.

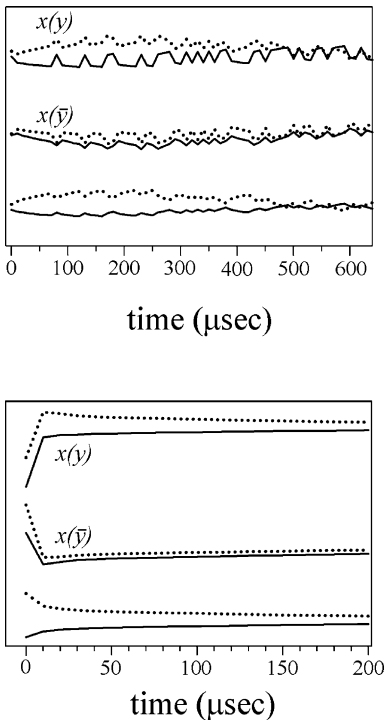


Fig. 2. $^{7}$Li NMR noise response data and the associated free-induction-decays observed after cross-correlation with the input sequence of rotations, for lithium sites in Li-monmorillonite. Noise response is observed after application of an MLBS containing 511 composite pulses; $\tau_{samp} = 20\,\mu s$, and in each case pulses lasted for $\tau_p = 2\,\mu s$. Data were acquired at 3 μs after termination of the pulse, so that the receiver might come out from saturation. Both real (solid) and imaginary (dotted) channels are displayed. (a) Noise response data from top, after $x(y)$-based MLBS pulse sequence; middle, after $x(\bar{y})$ sequence; bottom, after the two data sets are combined as described in the text, to eliminate ring-down. (b) Free-induction-decays derived from cross-correlation of noise response data of (a) with input MLBS rotation sequence; top, $x(y)$ data set; middle, $x(\bar{y})$ data set; bottom, the ring-down-suppressed combination of the two experiments. In either of the uncorrected data sets the ring-down signal dominates the early portions of the free-induction-decays.

peated with pulses applied along the $x$- and $-y$-axes, the voltage observed in the coil shifts phase by 180°; in contrast, where only a single pulse of similar amplitude and phase $y$ is applied, the phase and amplitude of the noise appear the same as in the $x(y)$ experiment. Presumably, for second pulses sufficiently short the ringing would have to depend on the first pulse, as well. We expect, however, that "short enough" refers to pulses which are short as compared to the apparent time constant of the coil—which for the relatively low $Q$'s and high operating frequencies found in our systems are consistently shorter than the minimum 300 ns pulse allowed by our hardware. As a result, we see no evidence for contributions to the systematic noise from the first pulse.

In Fig. 2 we present $^{7}$Li NMR noise response data acquired in the same fashion and the free-induction-decay which arises after cross-correlation of the noise response with the input sequence of rotations, obtained from a sample of Li-montmorillonite. In this sample, the galleries separating two-dimensional silicate platelets are filled with polyethylene oxide. The ring-down signal dominates the response in this sample, and we estimate that the magnitude of the systematic noise $N$ is no less than four times as large as the signal $S$ associated with the nuclear-spin magnetization. A pair of experiments is carried out; in the first, an MLBS-derived noise sequence is applied to the sample where all pulses are of the form $x(y)$ or $(-x)(-y)$; in the second experiment, all pulses are of the form $x(\bar{y})$ or $\bar{x}(y)$. (In addition, each pulse sequence is phase-cycled using the CYCLOPS procedure, to compensate for receiver artifacts.) Thus in the two experiments the noise $N$ is shifted by 180°, while the signal $S$ is shifted by 90°. After cross-correlation, the two data sets are combined as suggested above. Of course, the artifact suppression might equally well be accomplished with a variety of other pulse substitutions (e.g., $x(y) \rightarrow y(x)$ yielding identical signal $S$ but phase-shifted noise $N$), or even conceivably within a single MLBS excitation sequence—at the cost of somewhat more complex postexperimental processing.

Fig. 2 also demonstrates how probe ring-down in noise experiments, as in normal NMR experiments, most significantly impacts on the early data points of the FID. In either of the versions of the individual noise experiments, the interferograms (top) show similar signal amplitudes at all points in the experiment. After cross-correlation with the input sequence, we find that the ring-down—corresponding to a rapid decrease in the initial amplitude of the free-induction signal—vastly exceeds the desired nuclear magnetic resonance. Yet after recombining the individual experiments as suggested above, elimination of the ringing is nearly complete. Fig. 3 provides a more sensitive test of the technique, where we show raw FIDs as well as the ring-down-suppressed free-induction-decay, and the resulting Fourier transform, of $^{2}$H NMR spectra at 320 K of deuterated polyethylene oxide, d-PEO, intercalated into the two-dimensional galleries found in montmorillonite, a naturally occurring clay. In this system we estimate the probe ring-down $N$ to be about 100 times larger than the desired response signal, $S$, and the former entirely obscures the broad signals expected from the polymer in the individual experiments. Even at a temperature close to the melting point of PEO (approximately 60 °C), where substantial molecular dynamics would ordinarily average away the powder pattern, in the ring-down-suppressed noise spectrum we clearly observe the broad powder pattern representing a small number of PEO units with limited mobility—a result of the confinement-induced densification of the polymer near the montmorillonite surface.

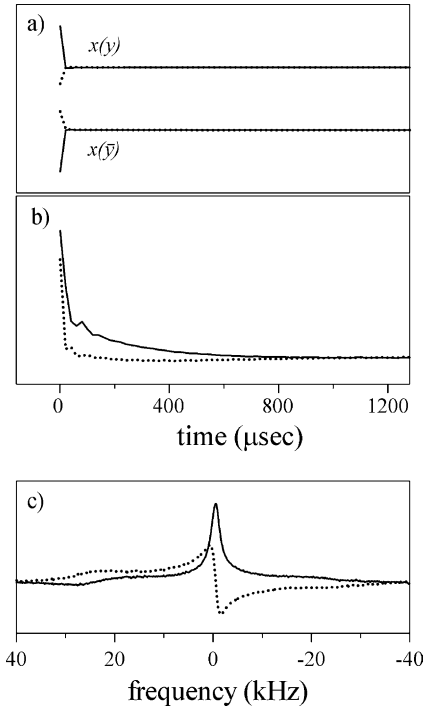


Fig. 3. $^2$H NMR of d-PEO at 320 K, derived from the set of noise experiments with $x(y)$ and $x(\bar{y})$-composite MLBS pulse sequences. Each pulse was applied for 1 μs; data were sampled 8 μs after termination of the second pulse, and $\tau_{samp} = 12.5$ μs. Both real (solid) and imaginary (dotted) channels are displayed. (a) Interferograms derived from MLBS sequence based on $x(y)$ and $x(\bar{y})$ composite pulse sequences, as indicated. The ring-down signal vastly exceeds the desired NMR signal amplitude. (b) Interferogram derived from ring-down-suppressed combination of two MLBS sequences as described in the text. Vertical scale is expanded 100-fold as compared to (a). (c) Fourier transform NMR spectrum of (b), showing the motionally averaged $^2$H sites in the sample.

## 4. Conclusions

We have demonstrated experimentally how probe ring-down can be removed by the simple use of composite pulses in noise-excitation NMR experiments. This approach requires no significant modification of the experimental conditions in the design of either the probe or the spectrometer; in fact, the only cost is a small increase in the average rf field required (as the excited signal increases in amplitude by $\sqrt{2}$ while twice as many pulses are applied). As the applied rf field is already reduced by 100–1000 with respect to ordinary NMR experiments, this increase in rf field will rarely prove costly. This work demonstrates that even where the ring-down effect is quite large it can be reliably removed, and, of course, even greater suppression of ring-down might be achieved where this experimental technique might be combined with other methods of ring-down suppression (e.g., active $Q$-damping or overcoupling), or with oversampling methods which provide signal sampling on timescales much shorter than $\tau_{sp}$, and thus open up the possibility of substantially broadened spectral bandwidths. We expect that the ring-down suppression—which can be improved by longer composite sequences where necessary—will ultimately be determined by the stability of the probe and spectrometer, and eventually by the digital limitations imposed by the process of analog-to-digital conversion in the receiver.

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