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Adding a lens Improves spinning speed characterization

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ABSTRACT

Highly stable sample rotation is important in many solid-state NMR experiments. Whether the necessary stability is achieved is not always clear. Typically only an average frequency over some time interval (often relatively long and unknown) is available from the spinning speed controller readout, which is not representative of the short-term variations of instantaneous rotation frequency. The necessity of the relatively slow measurement of spinning speed is a consequence of phase noise in the tachometer, which prevents speed measurement to be both rapid and precise at the same time. We show that adding a lens to the tachometer, without any other changes in the probe, reduces phase noise by nearly an order of magnitude and allows improved measurement of the spinning speed.

1. Introduction

Magic-angle sample spinning (MAS) NMR has become routine, especially since Schaefer and Stejskal combined MAS with cross-polarization [1–3]. Commercial probes and spinning speed controllers make sample spinning easy, and at moderate speeds MAS requires no more attention than properly setting the stator angle and dialing in the desired speed into the spinning speed controller. However, some rotor synchronous pulse sequences, including specialized variations of REDOR [4–6], demand extremely precise synchronization between sample rotation and RF pulse trains, and without good spinning speed control the observed signal intensity can be greatly compromised [7,8]. In these cases it is particularly important to understand the details of measurement and control of sample spinning.

Although theory and practice of frequency and phase measurement are extremely well developed in electrical engineering and metrology [9,10], the topic is seldom discussed systematically in the NMR context. The stability of spinning speed is most often reported in the literature as a range within which the readout of the spinning speed controller remained while the experimenter cared to monitor it, as this is usually the only data available to the user. This is analogous to specifying the frequency of a peak in an NMR spectrum without saying anything about its width or shape. Controllers do not provide much information about the quality of sample spinning to the user. Usually the only information that is provided is the average spinning speed that is determined over a relatively long measurement interval. Although the dynamics of feedback loops in the controller is typically faster than the display update rate, neither is specified and even controllers with a faster feedback loop do not display the short-term deviations of the spinning speed. This makes comparison between reported spinning speeds rather less meaningful, while the actual dynamics of instantaneous rotation frequency remains unknown.

Evidently, experiments for which standard stability of the spinning speed is not sufficient remain, as indicated by suggestions for improving sample spinning stability [11,12]. At the same time, even when lack of spinning speed stability is suspected of causing problems, rigorous analysis of spinning speed stability is hardly ever attempted. Due to lack of diagnostic data on the instantaneous spinning speed, it is generally presumed and hoped, but seldom known with certainty, that any changes in the speed are sufficiently slow to be unimportant for a particular experiment.

The instantaneous rotation frequency can be determined as a reciprocal of the rotation period, and it can be measured, in principle, for each revolution. Unfortunately, this is impossible to achieve in practice using current NMR hardware. A typical MAS probe tachometer reliably produces a pulse per rotor revolution, sufficient for the majority of MAS NMR experiments. However, from turn to turn the tachometer pulses correspond to slightly different angular orientations of the rotor. While of little importance when the rotational frequency is measured as the number of pulses per second, this effect becomes crippling when spinning speed estimates are attempted by accurately timing a small number of revolutions.

When synchronization of elements of the pulse sequence to the tachometer signal is employed [13], the jitter in tachometer pulse timing is also a concern. In principle, the synchronization between sample spinning and the NMR pulse programmer reduces the problem of accurately maintaining the spinning speed for rotor-

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synchronous pulse sequences by preventing accumulation of phase error across multiple rotor periods despite small deviations of rotation frequency from its set point. However, the jitter (phase noise) in the tachometer signal limits the accuracy with which angular orientation of the rotating sample is known. This fact has prompted development of various techniques to “clean” the tachometer output and reduce the amount of phase noise. These methods often include averaging tachometer phase by using a phase locked loop [14–16] or implementation of Kalman filtering techniques [17,18] to optimally combine several sources of information and produce the best possible estimate of the rotation frequency under existing measurement noise and uncertain dynamics of the system [19].

Here, we investigate the root cause of the problem of why the tachometer output does not accurately represent angular position of the spinning rotor. Then we show that adding a focusing lens to the tachometer, without any additional changes to the probe, provides a simple way to reduce the phase noise by nearly an order of magnitude. To be clear, we note that this modification by itself will not improve spinning speed stability but what it will do is provide a means to more accurately determine rotational periods over a short time frame.

2. Probe tachometer

Typical solid-state MAS probes contain an optical tachometer to measure the rotation frequency. It illuminates the sample rotor and measures the intensity of light scattered back by a small area of the rotor, on which one or more regions are painted or darkened by laser etching. During the course of rotation of the rotor this mark passes through the view field of the tachometer and thus modulates the intensity of backscattered light collected by the tachometer. The optical system is usually trivially simple and consists of two optical fibers, one for illumination and one for detection, with their ends a short distance away and facing the rotor (style A), or a single, larger, bifurcated optical fiber bundle which both illuminates and collects back scattered light (style B). In both cases, the backscattered light is then converted to an electrical signal by a photodiode. A threshold crossing detection circuit then converts the amplified photocurrent into a train of digital pulses corresponding to changes in intensity of the backscattered light due to rotation of the rotor. These pulses are the output signal of the tachometer, and they are used by the electronics in the spinning speed controller to monitor and control sample rotation.

For each revolution, the start of the electric pulse ideally coincides with specific orientation of the sample rotor. In practice, noise in the tachometer electronics makes the timing of the pulse to deviate slightly from the ideal position as illustrated in Fig. 1. The noise e_n causing the uncertainty in the time of threshold crossing Δt is exacerbated by the relatively slow transition between the “dark” and “bright” conditions caused by a relatively large size of the area from which the back scattered light is collected. As the blackened mark on the rotor enters the illuminated area, the backscattered light intensity gradually diminishes with rotation of the rotor and then returns to its previous high intensity level as the blackened sector leaves the illuminated area. The magnitude of the jitter Δt is equal to the magnitude of the amplifier noise e_n divided by the slope of the signal edge. If the observed dark-bright transition could be made more rapid, the time variation would be reduced correspondingly.

3. Phase noise and jitter

NMR practitioners are familiar with the Fourier transform and the connection that it provides between the representation of a signal as a function of time and a representation of the same signal in terms of spectral density as a function of frequency. Deviation of any physical process from ideal periodicity appears in both domains; it is reflected as the repetitions of the signal being non-identical from one time to the next and as a spectral line shape having finite width [9,20]. For nearly periodic processes, the short-term non-ideal behavior is described as phase noise (as opposed to arbitrarily longer term random or systematic frequency drift). The phase noise corresponds to “jitter” in the time domain, where jitter is commonly viewed as the signal transitions deviating from the expected positions. In the frequency domain the same phase noise is described as signal power density at frequencies slightly offset from the mean value of fundamental frequency thereby giving rise to the spectral line shape. An adequate description of the phase noise requires either the specification of the jitter as a function of time delay or the power density as a function of frequency offset.

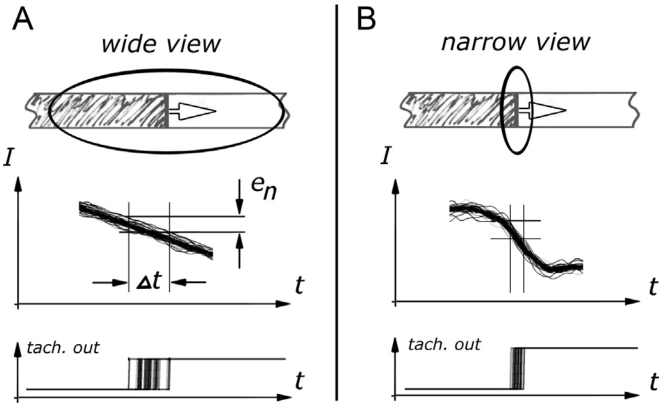

Fig. 1. Jitter in the tachometer output. The top of the sketch schematically shows the timing mark on the rotor crossing the indicated view field of the tachometer. Below it is shown the corresponding change in the backscattered light intensity as a function of time. At the bottom is the corresponding digital output of the tachometer. Panel (b) shows the effect of reducing the tachometer view field with respect to that in panel (a). For a given amount of electrical and optical noise (e_n) making the point of threshold crossing uncertain, the corresponding jitter Δt is inversely proportional to the slope of the light intensity signal as a function of time.

Magic-angle sample spinning is an approximately periodic process and, as such, is only partially specified by its mean frequency. A more accurate description of the sample rotation requires knowledge of the properties of its phase noise and frequency drift. Although it is in principle possible to monitor the “line shape” of the sample spinning and frequency drift using a high-resolution audio-frequency spectrum analyzer [21], a more simple and convenient diagnostic procedure is to directly observe the tachometer signal on an oscilloscope. The oscilloscope trace is triggered by the rising (or falling) edge of the tachometer signal crossing the trigger threshold, which should be set at a fixed level at the steepest part of the edge. By design of the oscilloscope, the position of the trigger point and the signal trace in its vicinity will appear stationary on the screen. Portions of the same signal that are further away from the trigger point will however not retrace each other on successive repetitions, generally diverging further apart at longer time delays if the process that generates the signal exhibits random phase and frequency variations. However, we note that if the frequency undergoes periodic changes of any kind, no matter how large the amplitude of the changes is, the average over any integral number of periods would remain the same as long as all the periods are identical. For example, if the drive pressure oscillates at 30 Hz, then the rotational frequency measured over any multiple of 33.3 ms will remain constant, despite large periodic variations of instantaneous frequency. Many oscilloscopes have a useful feature that allows observing the signal at a