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# Digital quadrature detection in nuclear magnetic resonance spectroscopy

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A digital quadrature detection technique for the acquisition of the nuclear magnetic resonance signal is proposed. It is demonstrated with experimental results that the suggested method is superior to the conventional quadrature detection (QD) technique. The superiority arises mainly from the fact that only one channel of analog circuit is utilized and no spectral distortion due to unbalance between two channels used by the QD occurs. As a result, no phase cycling for canceling the distortion mentioned above is necessary. © *1999 American Institute of Physics.* [S0034-6748(99)04102-7]

## I. INTRODUCTION

Nuclear magnetic resonance (NMR) spectroscopy has developed into a major spectroscopic technique over the last 20 years or so. This development has benefited largely from both instrumental and experimental aspects which seem to keep NMR still expanding.[1]

The NMR receiver design is extremely important since it directly determines the final spectrum quality.[2] A typical radio-frequency (rf) receiver design is schematically shown in Fig. 1. The NMR signal is first amplified to a level of perhaps several hundreds of millivolts. Then it is detected using a phase-sensitive detector (PSD). For quadrature detection (QD), now universally utilized, two channels are needed. The reference (rf) signal is split into two components that are 90° phase shifted with respect to each other. The advantage of QD is that positive and negative frequencies can be discriminated due to the phase difference of 90°. The rf signal can thus be placed in the center of the spectrum. This arrangement reduces the necessary bandwidth of the receiver as well as the data processing and rf transmitter output power requirements. However, since the NMR signal is split into two components, a slight difference in gain between two channels and small phase error in the quadrature reference signals will lead to image peaks. This spectral distortion may be eliminated by using the scheme[1] which involves accumulation of signals obtained with a series of rf pulses whose phase is successively shifted by 90°. Obviously, it will take a longer time for complete NMR data acquisition when the signal-to-noise ratio (S/N) is sufficiently good and signal accumulation is unnecessary. In order to speed up signal data acquisition, one can use the PSD of a single channel. However, in this mode of detection, the rf frequency must be placed out of the spectrum width to avoid the reflection spectral lines. Since positive and negative frequencies are not distinguished, noise from the outside is folded onto the spectrum. Moreover, the rf transmitter output power must be increased to ensure uniform excitation.

In this article, we suggest a detection method, namely digital quadrature detection (DQD). The present detection technique acquires a single channel of the NMR signal and performs quadrature detection digitally. Therefore, no unbalance and error between the real and imaginary components appear. The NMR signal may be samples in only one scan if the S/N is adequately high, resulting in a spectrum without any distortion caused by the detection procedure. In the following, we will discuss the principle of the DQD technique and the hardware requirements. Finally, an experimental demonstration will be given.

## II. PRINCIPLE

Unlike the normal PSD method, which places both the irradiation and detection (reference) frequencies outside the spectrum width, the suggested DQD technique employs two different frequencies for irradiation and detection, as schematically displayed in Fig. 2.

Under the irradiation of the rf pulse with frequency $f_i$ placed in the middle of the spectrum, the nuclear spins will give a free-induction decay (FID) signal written as

$$S_0(t)=\cos[2\pi(f_i+\delta)t+\varphi_0]\exp(-t/T_2), \tag{1}$$

where $\delta$ is the resonance offset caused by the interactions of the nuclear spin system, $\varphi_0$ is the phase shift, and $T_2$ is the transverse relaxation time. In order to sample the signal, we must lower the frequency by multiplying an additional signal with a frequency of $f_r$,

$$\begin{aligned} S_1(t)&=S_0(t)\cos(2\pi f_r t+\varphi_1)\\ &=1/2\{\cos[2\pi(f_i+f_r+\delta)t+(\varphi_0+\varphi_1)]\\ &\quad+\cos[2\pi(f_i-f_r+\delta)t+(\varphi_0-\varphi_1)]\}\exp(-t/T_2), \end{aligned} \tag{2}$$

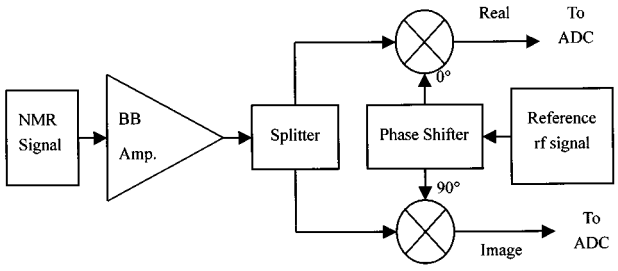


FIG. 1. The rf receiver with quadrature detection.

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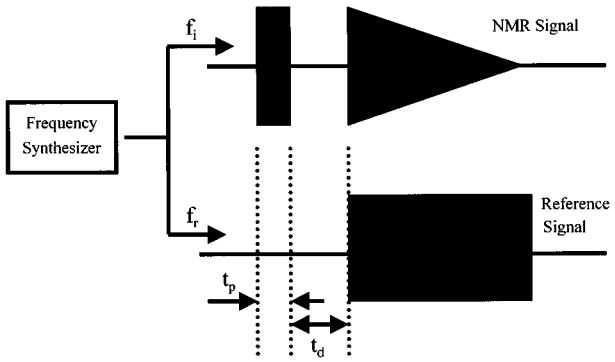


FIG. 2. Representation of the DQD method, where $f_i$ and $f_r$ are irradiation and reference frequencies, respectively. $t_p$ is the width of the excitation rf pulse and $t_d$ is the dead time of the spectrometer.

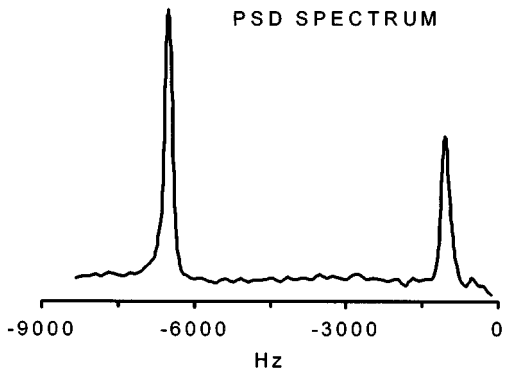


FIG. 3. $^{14}$N NQR spectra obtained with the conventional single-channel PSD method. The experimental parameters are as follows: $f_i = f_r = 3605$ kHz, $t_p = 100.0$, $t_d = 200$ $\mu$s, number of data sampling nd=128, sampling cycle $dw = 60.0$ $\mu$s, number of scans ns=256. Note that the spectral intensity depends strongly on the resonance offset since $t_p = 100.0$ $\mu$s and the excitation bandwidth will be confined to about $\pm 5$ kHz.

where $\varphi_1$ is the phase of the reference signal. After applying low-pass filtering, Eq. (2) becomes

$$S_2(t) = 1/2 \cos[2\pi(\Delta f + \delta)t + \Delta\varphi]\exp(-t/T_2). \quad (3)$$

In Eq. (3), $\Delta f = f_i - f_r$ and $\Delta\varphi = \varphi_0 - \varphi_1$. Generally, the spectrum width (SW) under extreme limit (solid-state NMR) is slightly above 1 MHz, approximately. Hence, $\Delta f$ can be made several times larger than the desired SW for the currently available analog-to-digital converter (ADC) to easily sample $S_2(t)$ into the computer. After digitization, we multiply $S_2(t)$ with two digital sine signals in quadrature. Then, the resultant complex FID signal may be expressed as

$$\begin{aligned} S_3(t) &= S_2(t)\exp(i2\pi\Delta f t + \varphi) \\ &= S_r(t) + iS_i(t), \end{aligned}$$

with

$$\begin{aligned} S_r(t) &= A\{\cos[2\pi(2\Delta f + \delta)t + \Delta\varphi + \varphi] \\ &\quad + \cos(2\pi\delta t + \Delta\varphi - \varphi)\}, \\ S_i(t) &= A\{\sin[2\pi(2\Delta f + \delta)t + \Delta\varphi + \varphi] \\ &\quad + \sin(2\pi\delta t + \Delta\varphi - \varphi)\}, \end{aligned} \quad (4)$$

where $A$ can be considered as the amplitude of the detected FID signal. Now we can perform a digital filtration upon Eq. (4). The cutoff frequency of the digital filter can be chosen such that it matches the expected SW (or $\delta$). The final normalized FID signal then becomes

$$S(t) = [\cos(2\pi\delta t + \Phi) + i\sin(2\pi\delta t + \Phi)]\exp(-t/T_2), \quad (5)$$

where $\Phi = \Delta\varphi - \varphi$ is the total phase difference. Having obtained Eq. (5), one can further perform normal data handling procedures, such as application of windowed and weighted functions to the original FID data, fast Fourier transform, phasing of the spectrum, etc., as usual.

## III. HARDWARE REQUIREMENT

In most cases the NMR signal is so weak that one has to perform data accumulation to increase the S/N to a reasonable level. It can be seen from Eq. (5) that data accumulation from different scans can be successfully performed only if the phase factor $\Phi$ remains constant. If this crucial requirement is not met, cancellation of signals from different scans will occur. Keeping the phase factor $\Phi$ time independent may present problems when using a conventional frequency synthesizer in the NMR spectrometer, even if $f_i$ and $f_r$ are derived from one source of the oscillator. Since, for two signals with different frequency, the phase difference is $(2\pi\Delta f t + \Delta\phi_0)$, where $\Delta\phi_0$ is the difference at the initial state, for the signals from the same oscillator, $\Delta\phi_0 = 0$, but the phase shift is still time dependent. Therefore, signal accumulation can only be done under the condition of $f_i = f_r$.

Let us consider that if we can reset the phase of the frequency synthesizer to a known absolute value, say, immediately before the excitation of the rf pulse (see Fig. 2), then the phase difference between the excitation and detection signals becomes $2\pi\Delta f(t_p + t_d)$, independent on time. So, the focus of the DQD technique is to find a device which meets the need mentioned above. Fortunately, the direct digital synthesis (DDS) chip, newly developed, is an ideal candidate for the proposed DQD technique.

In the present article, we employ a DDS, AD7008 from Analog Device, for this purpose. A clock rate up to 50 MHz is supported. A reset pin enables user to clear the registers to zero and the output will go to sin(0) after additional clock cycles. Frequency accuracy can be controlled to one part in 4 billion, and a frequency update can be done at a rate $>10$ MHz, much less than the dead-time $t_d$. By using the frequency mixer and/or multiple DDS chips, one can easily convert the output frequency of the DDS to a higher range where NMR experiments are performed.

In summation, equipped with a DDS on the spectrometer, the DQD method can be accomplished in the following steps. First, immediately before rf pulsing, reset DDS and then set the output frequency to $f_i$ at once. Second, immediately before detection (at the end of the $t_d$ interval in Fig. 2) switch the output frequency of the DDS from $f_i$ to $f_r$. Finally, after acquisition of FID data, multiply it with a set of quadrature digital sine data $\exp(i\Delta f t + \varphi)$, do digital filtering with cutoff frequency which matches the desired SW, and down-sample the FID data according to the desired SW. The final step can be done during the recovery time interval of

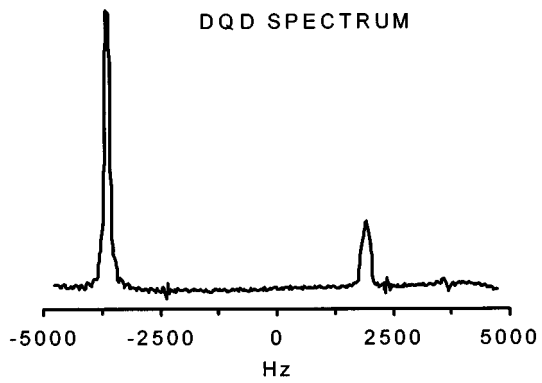


FIG. 4. $^{14}$N NQR spectra obtained with the suggested DQD method. The parameters employed in the experiments are as follows: $f_i = 3600$ kHz, $f_r = 3607$ kHz, $t_p = 100.0$, $t_d = 200$ $\mu$s, number of data sampling nd=512, sampling cycle dw=30.0 $\mu$s, and number of scans ns=256.

each scan or after the total acquisition of the over-sampled FID data is complete.

## IV. EXPERIMENT AND DISCUSSION

The experiments were carried out on a home-built remote $^{14}$N nuclear quadrupole resonance (NQR) spectrometer[3] around room temperature. The main computer of the spectrometer is a PC of PII/233. We examined the DQD method by employing a single-pulse sequence, as displayed in Fig. 2. The sample is a mixture of sodium nitrite ($NaNO_2$) powder and quartz with a resonant frequency of about 3596 kHz. Figure 3 shows $^{14}$N NQR spectra obtained with the conventional PSD method. Because the rf pulse width is relatively large, uniform excitation is limited to a small frequency range. As a result, the spectral intensity arising from the quarts is heavily distorted (weakened). This distortion may be reduced simply by decrease of the rf pulse width $t_p$. However, the necessary rf output power will increase accordingly.

The spectrum displayed in Fig. 4 is obtained with the DQD method. the desired spectrum width SW for the sample is $\pm 5.0$ kHz, and the parameter $\Delta f$ we chose is 7.0 kHz. In this case, the highest-frequency component within the detected FID signal is $(\Delta f + 0.5\ \mathrm{SW}) = 12.0$ kHz. Naturally, the better the digital filter, the smaller the $\Delta f$. Under certain spectrum width, it is advantageous to use smaller $\Delta f$ because this can ease the demands for the speed of the ADC and reduce the data size to be handled. But, it relies upon the characteristics of the digital filter employed.

The DQD spectrum shown in Fig. 4 was taken at relatively low frequency. In order to extend the technique to higher frequencies, one can simply choose a faster DDS device. Now DDS chips capable of producing frequencies higher than 300 MHz are commercially available. The other method is to mix a low-frequency DDS output with a fixed high-frequency signal. For experiments performed in hundreds of MHz, we believe that the latter method will give a better result, since errors caused by the intrinsic jitters in the DDS reset could be very important and significant at higher frequencies.

We have demonstrated theoretically and experimentally the digital quadrature detection of the NMR signal. Comparing PSD, QD, and DQD, we believe that the proposed DQD technique may be widely employed in modern NMR instruments in the near future.

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