

### 1. Introduction

Figure 1a shows the simplest pulse sequence for an NMR experiment: spins are excited by a hard pulse at the Larmor frequency, and the receiver is turned on to acquire the Free Induction Decay (FID). The FID is at maximum when the spin flip angle equals  $90^\circ$ . At a given power level, the pulse width causing spins to flip  $90^\circ$  is called "90° pulse width". It is one of the most important parameters for setting up NMR experiments. To calibrate the 90° pulse width,  $\tau_{90^\circ}$ , one can acquire a set of 1D spectra with series of pulse widths,  $\tau$ . The signal reaches the first maximum when  $\tau$  equals  $\tau_{90^\circ}$  and zero when  $\tau$  equals  $\tau_{180^\circ}$ . Looking for the first node is a common way to calibrate  $\tau_{90^\circ}$ . In this example, we demonstrate how to establish a one pulse experiment including  $\tau_{90^\circ}$  calibration, 1D data acquisition and processing on Tecmag spectrometers.

### 2. Set-up a Pulse sequence

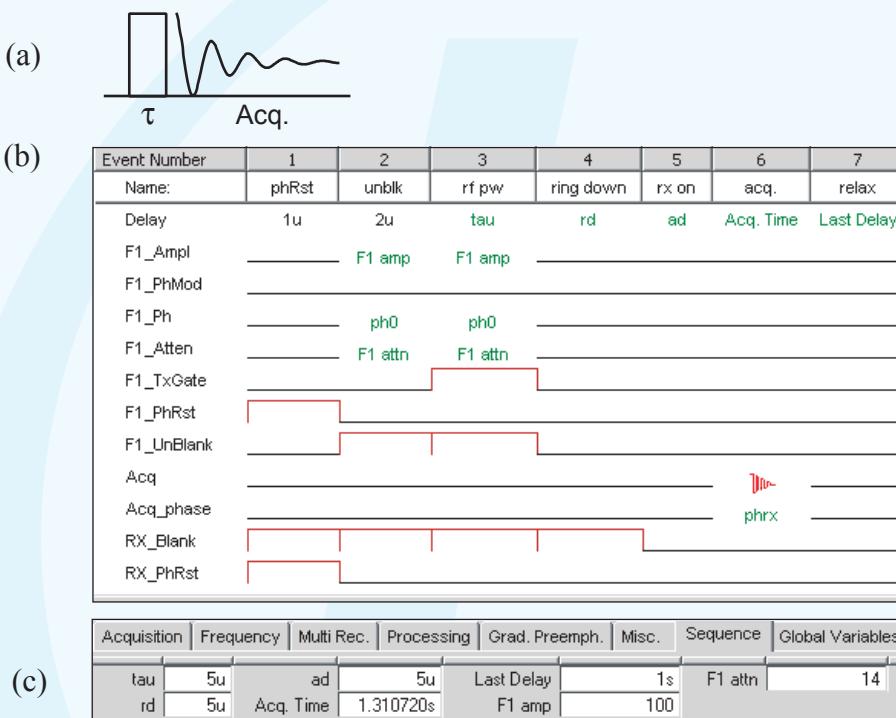


Fig. 1. (a) The diagram for the one pulse sequence. (b) The sequence realized in the NTNMR sequence editor. The pulse is generated in the F1 channel. (c) The sequence variable dashboard.

### Events in the Sequence Editor:

Figure 1 shows the implementation of the one pulse sequence in the NTNMR sequence editor. Typically 7 events are necessary:

- Event 1: Set the phase reset for the transmitter (F1 channel) and receiver ("F1\_PhRst", "RX\_PhRst").
- Event 2: Turn on the F1 high power amplifier ("F1\_UnBlank").
- Event 3: Turn on the RF pulse ("F1\_TxGate"). "F1\_Ampl" and "F1\_Attn" control the amplitude. The variables "F1 amp" and "F1 attn" are used to set their respective values. "F1\_PhMod" and "F1\_Ph" control the phase. The 1D phase table "ph0" is used. Entries are 0, 2, 1, 3 for a phase cycle through x, -x, y, -y. Starting phase and amplitude controls in Event 2 avoids glitches.
- Event 4: Set a delay for the probe ring down time. It is represented here by the variable "rd".
- Event 5: Turn on the receiver before data acquisition ("RX\_Blk"). The turn-on time is defined using the variable "ad". The receiver is blanked from Event 1 to 4.
- Event 6: Acquire data. The receiver phase is set on "Acq\_phase". The 1D phase table "phrx" with entries, 0, 2, 1, 3, is used.
- Event 7: Set the recycling time according to the sample's T1.

### The sequence variable dashboard:

Click on the Sequence tab of the dashboard, all the sequence variables defined in the sequence editor appear (Fig.1d). Their values are directly entered into the corresponding boxes.

### 3. Experiment and Results

Sample:  $\text{H}_2\text{O}$   
 Spectrometer: 7 Tesla Magnet with Tecmag HF3 discovery  
 Probe: Naloac D300-5 OWB 5mm  $^1\text{H}/^{13}\text{C}$  Switchable probe  
 $^1\text{H}$  hard pulse: 13.9 kHz ( $90^\circ = 19 \mu\text{s} @ 5\text{W}$ )

#### 3.1 One Pulse Experiment

##### The Acquisition dashboard parameters:

Observed freq.: 299.90142 MHz

Observed Ch.: 1

Acq. points: 4096

SW +/-: 500

Dwell Time: 100  $\mu\text{s}$

Scans 1D: 1

Last Delay: 3 s

Scans 2D: 1

##### 1D Data Processing:

In the NDFT window, click the "1D" tab and select: "Use 1D settings", "Fourier Transform", "Exponential, LB = 5", click "Do it".

Click Option|Phase Adjustment, and adjust the spectrum to pure absorption.

#### 3.2 Pulse Calibration

Left-click on "tau" in the sequence editor of the one pulse experiment (Fig.1b), a pop-up window appears. Select "2D Table". Set the table as: Auto, Increment Value = 5  $\mu\text{s}$ . In the dashboard, set "tau" = 5  $\mu\text{s}$  (the "tau" value serves as the first entry in the 2D delay table), and "Scans 2D" = 20. The rest of the dashboard parameters are the same as in the experiment described in 3.1. Click the ZG button. A 2D spectrum will be acquired with 20 records.

Process the data as a 1D spectrum (see 3.1). Adjust the phase of the first record for pure absorption. Use the same phase correction for the rest of the records by select "Apply to all" in the Phase Adjustment.

To display all 20 spectra as shown in Fig. 3, click Commands|Data Manipulation|Separate Records|Joint (20). Since the first node is at  $\sim 38 \mu\text{s}$  the  $90^\circ$  pulse width is  $\sim 19 \mu\text{s}$ .

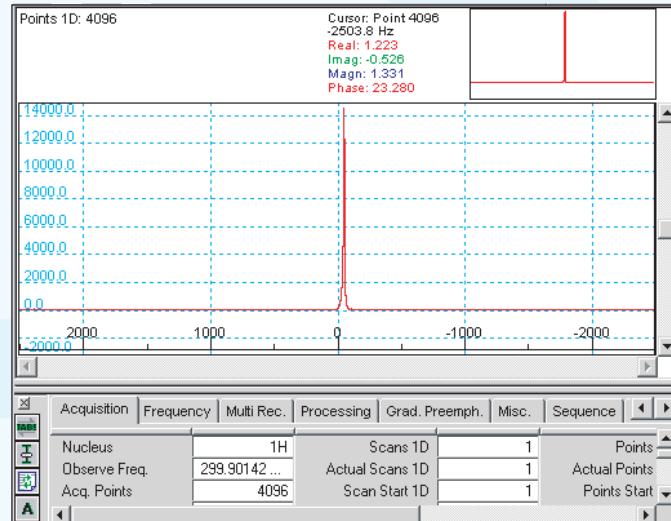


Fig. 2. The  $^1\text{H}$  spectrum of  $\text{H}_2\text{O}$  acquired with the one sequence shown in Fig.1. The pulse width tau is set to 17  $\mu\text{s}$ .

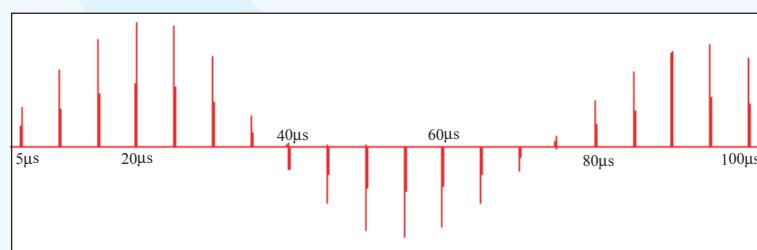


Fig. 3. A set of  $^1\text{H}$  spectra of  $\text{H}_2\text{O}$  acquired using the one sequence with different pulse widths.