Water Suppression in High Resolution ^{1}H NMR Spectroscopy Using Continuous Wave Free Precession Sequence

R. B. V. Azeredo
Instituto de Química de São Carlos – USP, Brazil

T. Venâncio, L. A. Colnago*
Embrapa Instrumentação Agropecuária, Brazil
colnago@cnpdia.embrapa.br

M. Engelsberg
Departamento de Física – UFPE, Brazil

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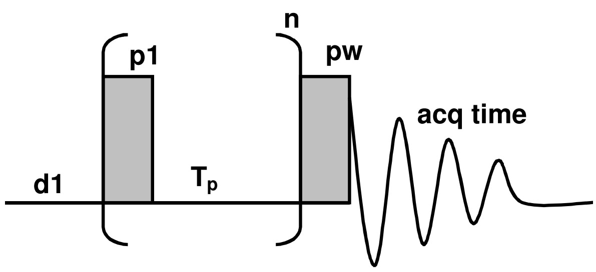
Abstract: *The area of solvent suppression has a long history and is an indispensable part of almost every NMR experiment conducted in a solvent containing nuclei of the type to be detected. Here a new method for solvent signal suppression has been proposed. It consists of a pulse sequence named CWFP (continuous wave free precession). In this technique the spin system is submitted to a train of pulses with same phase, intensity, and duration, and separated by time (T_p) << T_2. The experiments were performed in a 2T Inova80 Varian spectrometer using ripped kaki fruits. The excitation profile can be calculated from the Bloch equations and depends on the relaxation times T_1 and T_2, tip angle α and T_p. The signal is null in the zero frequency. We are testing this sequence to suppress the water signal in fresh fruits to measure their sugar content. The sugar signal is a shoulder in the water peak and is difficult to be measured without water suppression. The CWFP was tested in kaki fruit and compared with the jump return sequence. Both sequences reduced the water signal by about the same magnitude, but the jump-return sequence introduced more artifact in the spectrum than CWFP.*

The area of solvent suppression has a long history and is an indispensable part of almost every NMR experiment conducted in a solvent containing nuclei of the type to be detected[1]. As the solvent concentration is much higher than that of the solute, it composes most of the NMR signal. There are several methods for solvent signal suppression. Some are simple, like "presaturation" and "jump-return", and others include gradient sequences and shaped pulses, which are very efficient but not available in all spectrometers.[1]

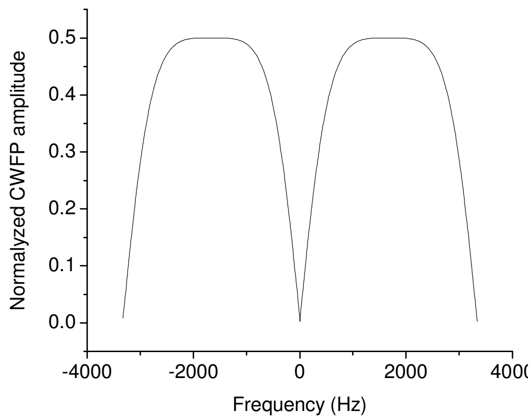
Here a new method for solvent signal suppression is proposed. It consists of a new pulse sequence named CWFP (continuous wave free precession)[2], which has been successfully used in analytical applications[2a,2b] and flow measurements.[2c] In this technique, the spin system is submitted to a train of pulses with same phase, intensity and duration, and separated by time (T_p) << T_2. This sequence produces an excitation profile similar to Jump-return and binomial pulse sequences[1], with a null in the zero frequency offset.

The experiments were performed in a 2T Inova80 Varian spectrometer (30 cm bore), using a surface coil probe. The sample used was ripped kaki fruits. The pulse sequence was written in the programming module of the VNMR-Varian software. The CWFP pulse sequence (Figure 1a) uses a train of *n* pulses (p1), separated by a time (T_p), centered at the frequency of the signal whose suppression is desired. The signal is acquired after the pw pulse. D1 is the recycle delay between sequences. The number of p1 pulses (*n*) depends on the intensity of the signal to be suppressed and relaxation times.

The excitation profile can be calculated from Bloch equations[2] and depends on the relaxation times T_1 and T_2, tip angle α and T_p. Figure 1b shows the excitation profile in absolute values for $\alpha=\pi/2$ pulses and T_p= 0.3ms.



(a)



(b)

Figure 1. (a) CWFP pulse sequence; **(b)** excitation profile in absolute values for CWFP sequence for $\alpha=\pi/2$ pulses and T_p= 0.3ms.

The signal is null at zero frequency and at frequencies equal to $1/T_p$. We have tested this sequence to suppress water signal in fresh fruits to measure their sugar content. The sugar signal is a shoulder in the water peak and is difficult to be measured without efficient water suppression. Figure 2 shows the ^{1}H NMR spectra in absolute values of a ripped kaki fruit using jump-return (a) and CWFP pulse sequences (b) to suppress the water signal in δ 4.7.

The sucrose signal is in δ 3.5 ppm. As can be seen in the results shown in Figure 2, both sequences reduced the water signal by about the same magnitude, but the jump-return sequence introduced more artifact in the spectrum than that of CWFP.

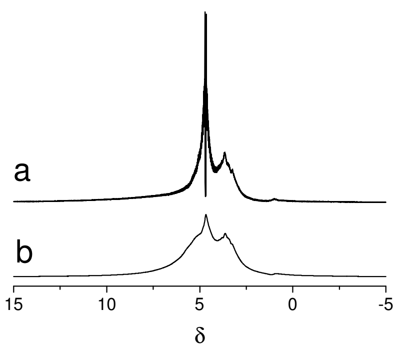


Figure 2. ^{1}H-NMR spectra in absolute values of an intact kaki fruit with water suppression by Jump-return **(a)** CWFP sequences **(b)**. In CWFP sequence n=1024, T_p=0.3ms, p1=pw=$\pi/2$ pulses.

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