

12th NUCLEAR MAGNETIC
RESONANCE USERS MEETING
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MAY 4th - 08th, 2009 - HOTEL DO FRADE, ANGRA DOS REIS,
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EXTENDED
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IMPROVEMENTS IN THE CWFP METHOD FOR SOLVENT SUPPRESSION IN HIGH RESOLUTION NMR

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When the analyte is present in a very dilute solution, the solvent signal can cause serious problems in NMR spectrum. The large solvent signal reduces the quality of digitalization of analyte signals, causing a dynamic range problem. When it used pure deuterated solvent also can be a critical problem, for example in protein studies, because the chemical exchange that occurs in amide groups leads to lose information about amino acids sequencing. It is worse in "in vivo" experiments where is not possible to use deuterated solvents. Then, the solvent suppression is one of the most important techniques in high resolution NMR. There are several methods for solvent suppression based on saturation, magnetization destroying through gradient pulses, data post processing [1]. Nowadays, the most efficient methods for solvent suppression are those ones that use gradient pulses, but it could not be available in every machines. Recently, a simple technique based on a variation of the steady state free precession (SSFP), called continuous wave free precession (CWFP), was developed to perform the solvent suppression. It demonstrated to be useful, especially for "in vivo" measurements [2]. However, some problems need to be solved to improve the result obtained by using CWFP technique. These problems are mainly the intensity and phase anomalies. The purpose of this work is to try to solve these problems.

The experiments were performed in equipment consisted of a 2T Oxford magnet with a 30cm bore and electronic based on an Apollo-Tecmag spectrometer, a power amplifier AMT 2035 and a pre amplifier Miteq 1054. It was also employed a 1cm diameter home-made solenoidal coil, covered with an epoxy resin to keep it rigid. Neither plastic tube nor epoxy resin exhibit NMR signal in such conditions. The sample used is a 1:1 mixture of CHCl_3 : CH_3COOH , to guarantee a big distance between chemical shifts observed. The measurements were performed without shimming and locking. It was employed CWFP technique as in previous works [3], but now varying the phase cycling and pulse width of the train of the pulses. These modifications were based on a methodology proposed by Rudakov et al [4], and also in the excitation profile of the CWFP pulse sequence.

The ^1H NMR spectrum of this sample exhibits signals at 7.3ppm related to CHCl_3 and 2.0 and 11.7ppm related to CH_3COOH , as shown in fig.1 (a). When the CWFP pulse sequence is applied to suppress the CHCl_3 signal, at 7.3 ppm, without phase cycling it can be observed a phase anomaly in the spectrum. The CHCl_3 is not totally suppressed and the signals related to CH_3COOH are 180° inverted to each other, as can be observed in the fig.1 (b). The phase anomaly can be corrected when the CWFP pulse sequence is applied with a phase cycling, but another problem persists, which is related to intensity anomaly, as in the fig.1 (c). Based on the excitation profile, the pulse width of the pulse from the train was varied and the best result was obtained when it was used a pulse width of 15° . In the fig.1 (d) it was obtained a spectrum with phase and intensity anomalies corrected, and it must be observed a total suppression of the CHCl_3 signal in 7.3ppm.

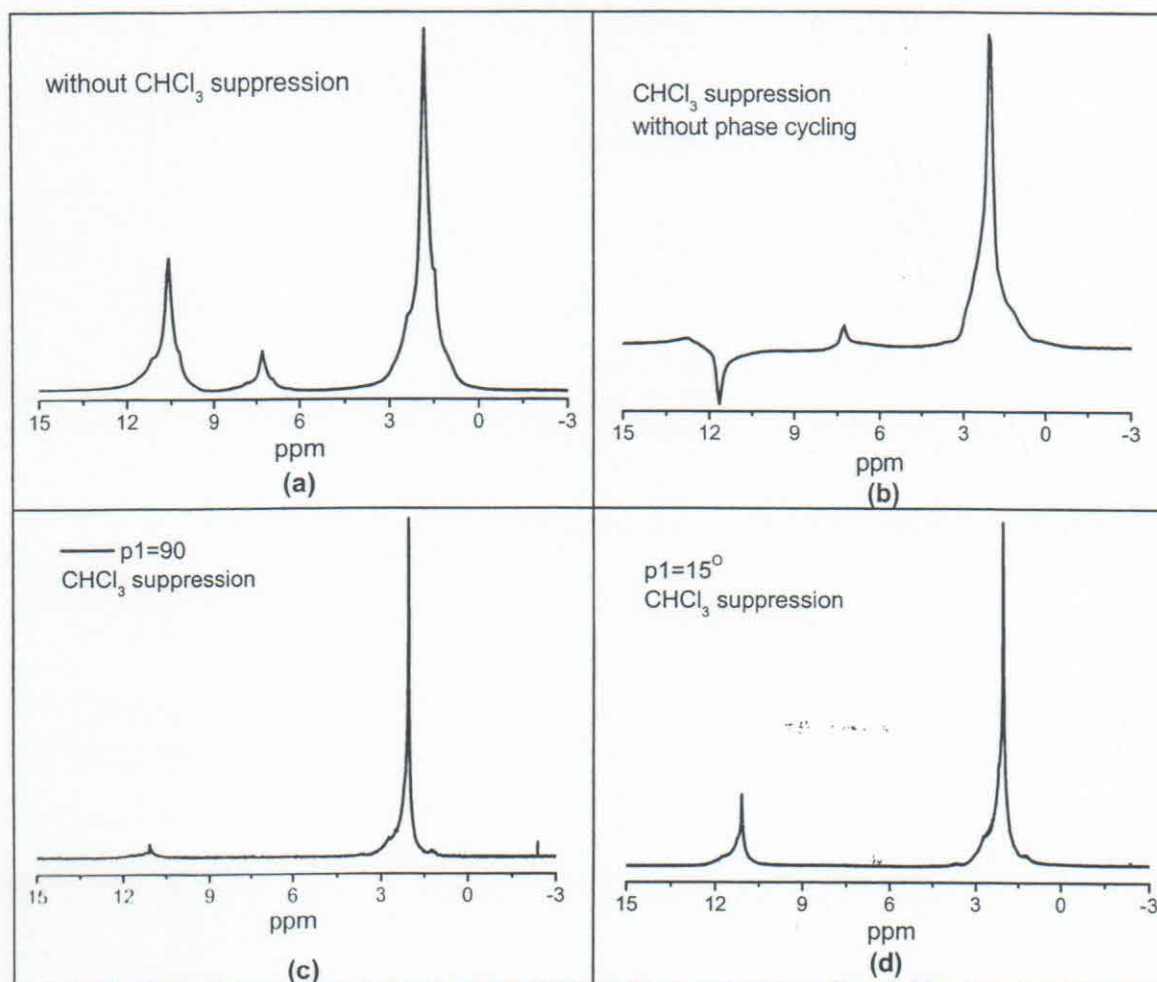


Figure 1: ^1H NMR spectrum of a mixture $\text{CHCl}_3/\text{CH}_3\text{COOH}$: **(a)** without CHCl_3 suppression, using FID; **(b)** CHCl_3 suppressed using CWFP without phase cycling - $(90^\circ_x - T_p)_n 90^\circ_x$; **(c)** CHCl_3 suppressed with 90° pulse width CWFP and phase cycling - $(90^\circ_x - T_p - 90^\circ_y - T_p - 90^\circ_x - T_p - 90^\circ_y - T_p)_n 90^\circ_y$; **(d)** CHCl_3 suppressed with 15° pulse width CWFP and phase cycling - $(15^\circ_x - T_p - 15^\circ_y - T_p - 15^\circ_x - T_p - 15^\circ_y - T_p)_n 90^\circ_y$. $T_p = 300\mu\text{s}$ for all the experiments.

From the obtained results, it could be concluded that the CWFP can be used as a good technique for solvent suppression, taking account some adjustments to improve its efficiency. These adjustments must be done in its phase cycling and also in the pulse width. The advantage of the CWFP method is that it is not necessary to use gradient pulses and it is easy to be implemented. Another possible interference, such chemical exchange, is being investigated.

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FAPESP, CNPq and CAPES