

Communication

Fast and simultaneous measurement of longitudinal and transverse NMR relaxation times in a single continuous wave free precession experiment

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Abstract

The purpose of this communication is to describe a method for rapid and simultaneous determination of longitudinal (T_1) and transversal (T_2) relaxation times, based on a single continuous wave free precession (CWFP) experiment which employs RF pulses with a $\pi/2$ flip angle. We analyze several examples, involving nuclei such as ^1H , ^{31}P , and ^{19}F , where good agreement with T_1 and T_2 measurements obtained by traditional methods is apparent. We also compare with the more time-consuming steady-state free precession (SSFP) method of Kronenbitter and Schwenk where several experiments are needed to determine the optimum flip angle. The role of an inhomogeneous magnetic field on the observed decays and its effect upon the accuracy of relaxation times obtained by these methods is examined by comparing numerical simulations with experimental data. Possible sources of error and conditions to minimize its effects are described.

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1. Introduction

Kronenbitter and Schwenk [1] proposed, several years ago, a fast method for the determination of the longitudinal (T_1) and transverse (T_2) relaxation times in liquids using steady-state free precession (SSFP) [2–5]. This method, although quite general, is especially useful for systems with very long T_1 values. It has been successfully used, for example, in nuclei like Xe^{129} , where T_1 values may reach 30 s [6].

The method, as proposed in [1], consists of two steps. In the first “ T_1/T_2 ” step the ratio of these two relaxation times is obtained by measuring the steady-state signal amplitude as a function of the flip angle α of the radio-frequency (RF) pulses. The optimum value α_{opt} , which yields the maximum SSFP signal, is first determined by a sequence of measurements with different α values, a relatively time-consuming procedure. Once α_{opt} has been obtained, the expression $T_1/T_2 = (1 + \cos(\alpha_{\text{opt}}))/(1 - \cos(\alpha_{\text{opt}}))$ [1] yields the ratio of the two relaxation times. Next, the value α_{opt} is employed in a second “ $T_1 + T_2$ ” step, where, in a new experiment, the time constant T_{opt} for the evolution to the steady state is measured. For the optimum flip angle, this time

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constant can be shown [1] to be given by $T_{\text{opt}} = (T_1 + T_2)/2$, which together with the result of the first step permit the determination of both relaxation times.

The purpose of this communication is twofold. First we show that the time-consuming determination of α_{opt} can be bypassed permitting the simultaneous fast measurement of T_1 and T_2 in a single experiment. Second, the role of an inhomogeneous magnetic field and its effect on the measured relaxation times is examined pointing out possible sources of error. The effect of an inhomogeneous magnetic field can be especially critical when low-resolution spectrometers, quite common in routine analytical determinations [7,8], are employed.

2. Theory

The general response to a sequence of RF pulses in a liquid described by a single NMR line has been treated by several authors [9–12]. The general framework is provided by the Bloch equations with additional terms, which include the effect of diffusion in the presence of a magnetic field gradient.

For our specific case we consider, in a reference frame rotating at the frequency of the applied RF, a general isochromat with angular frequency ω . Let T_p denote the time interval between the RF pulses in the SSFP pulse sequence [2,3] and assume that the effect of each pulse can be considered as an instantaneous rotation about the x -axis with a fixed flip angle $\alpha = \pi/2$. Denoting by ϖ the average isochromat frequency, a detuning offset between the RF pulses and $\varpi/2\pi$ is introduced. As a result, the precession angle during T_p for the average isochromat becomes $\varpi T_p = (2m + 1)\pi$, where m is an integer. For a general isochromat, the precession angle can therefore be written as $\psi = \omega T_p = (2m + 1)\pi + \phi$, where the distribution of angles ϕ is governed by the inhomogeneity of the magnetic field. We first assume, for simplicity, a Lorentzian distribution of frequencies $\Omega = \phi/T_p = \omega - \varpi$ of form $g(\Omega) = (T_2^*/\pi)[1/(1 + (\Omega T_2^*)^2)]$, where the conditions $T_p \ll T_2^* \ll T_1$, T_2 are expected to be satisfied.

The time evolution of the transverse components of magnetization for a general isochromat, denoted by $m_x(\phi)$ and $m_y(\phi)$, can be obtained from the matrix form of Bloch's equations. Provided T_p is sufficiently short, diffusion can be ignored [13–15] and the following recursion relationships can be shown to be obeyed:

$$m_{x,n+1}(\phi) = -m_{x,n}(\phi)E_2 \cos \phi + m_{y,n-1}(\phi)E_2 E_1 \sin \phi - E_2(1 - E_1) \sin \phi, \quad (1)$$

$$m_{y,n+1}(\phi) = m_{x,n}(\phi)E_2 \sin \phi + m_{y,n-1}(\phi)E_2 E_1 \cos \phi - E_2(1 - E_1) \cos \phi. \quad (2)$$

In Eqs. (1) and (2) $E_1 = \exp(-T_p/T_1)$ and $E_2 = \exp(-T_p/T_2)$ whereas $m_{x(y),n}$ represent the amplitudes of the transverse components of magnetization. The integers $n = 0, 1, 2, 3, \dots$ in $m_{x(y),n}$ denote the amplitudes of the precessing magnetization prior to the application of pulse n . Thus, the initial conditions necessary for the solution of the difference equations can be written as: $m_{x,0} = 0$, $m_{x,1} = -E_2 \sin \phi$, and $m_{y,1} = -E_2 \cos \phi$.

For homogeneous magnetic field, one can set $\phi \approx 0$ in Eqs. (1) and (2) and the difference equations can then be solved analytically. This can most easily be accomplished by noticing that Eq. (2) can be written, for this case, in the following manner:

$$\frac{m_{y,n+1} - m_{y,n-1}}{2T_p} = \frac{(E_2 E_1 - 1)m_{y,n-1}}{2T_p} - \frac{E_2(1 - E_1)}{2T_p}. \quad (3)$$

Since the value of T_p is assumed to be very short compared with the decay time of the continuous wave free precession CWFP [7] signal, the left-hand side of Eq. (3) becomes a good approximation for the time derivative of the decay, provided only pulses of the same parity are considered. Therefore for the signal prior the application of even pulses, for example, the left-hand side of Eq. (3) can be identified with $dm_y(t)/dt$, where $t = (n - 1)T_p$ and $n = 1, 3, 5, 7, \dots$. This leads to a simple differential equation which predicts an exponential decay to a constant steady-state value, as shown in [1]. To first order in the very small quantities T_p/T_1 and T_p/T_2 the time-constant of the decay is given by $T(\alpha = \pi/2) = 2T_1 T_2 / (T_1 + T_2)$, whereas the constant steady-state values are: $|m_{y,ss}| = M_0 T_2 / (T_1 + T_2)$ and $|m_x| = 0$. Here M_0 denotes the thermal equilibrium z -component of magnetization. Thus, from measurements of $|m_{y,ss}|/M_0$ and $T(\alpha = \pi/2)$, in a single experiment, the values of T_1 and T_2 could be determined.

In the presence of a distribution of values of $\phi = \Omega T_p$ the exponential decay to a constant steady-state value, mentioned above, can only be warranted under special circumstances and to a certain degree of accuracy. The measured signal amplitude in this case is a sum of contributions from all isochromats and can be written as the integral $M_{x(y),n} = \bar{m}_{x(y),n} = \int m_{x(y),n}(\Omega/T_p)g(\Omega) d\Omega$ over all values of Ω . Fig. 1 shows a numerical solution of Eqs. (1) and (2) as a function of n applicable to the ^1H resonance of phosphoric acid, where the measured relaxation times were $T_1 = 190$ ms and $T_2 = 140$ ms. The inhomogeneity of the magnetic field produced an approximately Lorentzian line of width (FWHM) $\Delta f = 90$ Hz, corresponding to $T_2^* = 1/\pi\Delta f = 3.54$ ms. Consequently, for each value of n , an integration over Ω was performed on each component of magnetization. Finally, the absolute value of the normalized integrated transverse magnetization $|M_{+,n}|/M_0 = |M_{x,n} + iM_{y,n}|/M_0$ was calculated and plotted together with the experimental data in Fig. 1A. The experimental results consist of absolute values of the CWFP [7] signal as a function

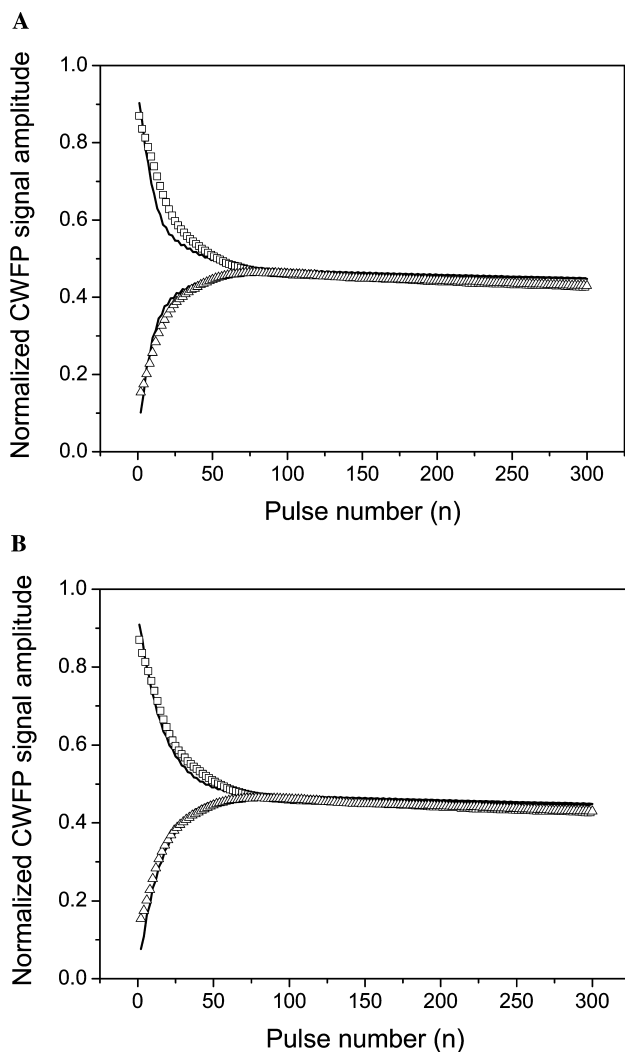


Fig. 1. The solid lines represent numerical solutions of Eqs. (1) and (2) as a function of n for $\alpha = \pi/2$ pulses with $T_p = 0.3$ ms, $\varpi T_p = 3\pi$, $T_1 = 190$ ms, and $T_2 = 140$ ms. (B) A Lorentzian distribution of isochromats with FWHM $\Delta f = 90$ Hz is assumed. (A) An asymmetric two-Lorentzian distribution with FWHM $\Delta f = 90$ Hz is assumed. The experimental data (\square odd pulses and \triangle even pulses) correspond to the ^1H resonance of phosphoric acid.

of n for phosphoric acid, with $T_p = 0.3$ ms and $\varpi T_p = 3\pi$.

Since magnetic field inhomogeneities quite often produce asymmetric lines the simulations were also performed using a normalized distribution function consisting of a linear combination of two shifted Lorentzians. One Lorentzian curve was centered at $\Omega = 0$ with a FWHM of 77.6 Hz whereas the other, shifted by 92.7 Hz and with half the weight of the first one, had FWHM of 155.3 Hz. The overall FWHM of the unresolved doublet was maintained at 90 Hz. The effect of such an asymmetry upon the CWFP decay is shown in Fig. 1A.

The experimental data and the simulations of Fig. 1 demonstrate that, for $T_2^* \ll T(\alpha = \pi/2) = 2T_1T_2/$

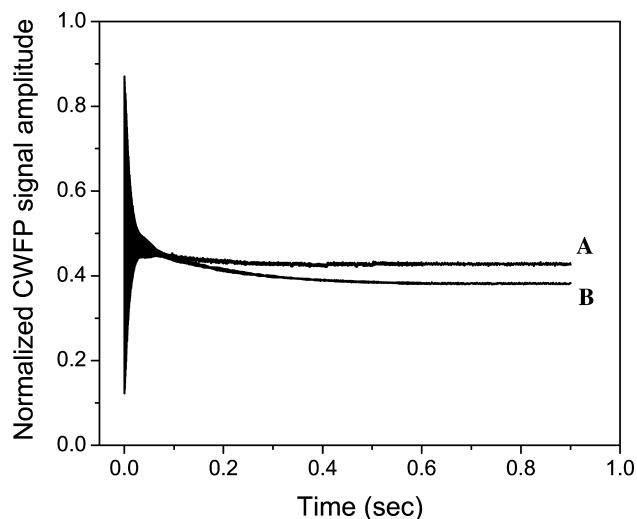


Fig. 2. Normalized ^1H CWFP signal amplitude in phosphoric acid for RF pulses with flip angle $\alpha = \alpha_{\text{opt}}$ (A) and for $\alpha = \pi/2$ (B), $T_p = 0.3$ ms and $\varpi T_p = 3\pi$.

$(T_1 + T_2)$, two transient regimes can be identified before a constant steady state is reached. The first one is characterized by an alternation of signal amplitude between even and odd values of n and by a time constant related to T_2^* . This leads to a quasi-stationary state [7] where these alternations disappear. Finally, the decay of the quasi-stationary state, controlled by T_1 and T_2 , leads to a truly constant stationary regime. All three regimes are clearly displayed by the experimental data of Figs. 2A and B, where the initial part ($t < 0.09$ s) of Fig. 2B corresponds to the data shown earlier in greater detail in Fig. 1.

For the quasi-stationary regime to accurately represent an exponential decay with time-constant $T = 2T_1T_2/(T_1 + T_2)$ and stationary amplitude $|M_{+,s}|/M_0 = T_2/(T_1 + T_2)$ some conditions must be met. After integration of both sides of Eq. (2) with the distribution function $g(\Omega)$, the ratio $\rho_n = |\overline{m_{x,n}}(\phi)E_2 \sin \phi|/|\overline{m_{y,n-1}}(\phi)E_2E_1 \cos \phi|$ must become sufficiently small for all values of n above some threshold value n_{th} . This ratio was calculated from the numerical solution of the recursion relationships (Eqs. (1) and (2)) and found to be dependent only upon T_p/T_2^* , provided $T_2^* \ll T_1, T_2$. For $n > n_{\text{th}} \approx 5T_2^*/T_p \approx 60$ and $T_p/T_2^* = 0.085$, corresponding to a Lorentzian line width (FWHM) $\Delta f = 90$ Hz, we obtained $\rho_n \approx 0.05$. As expected, the asymmetric distribution of Fig. 1B only causes some changes in the region $n < n_{\text{th}}$. The value of ρ_n was also calculated for this distribution function yielding a value $\rho_n \approx 0.06$, which does not significantly differ from the symmetrical case. Hence, within an accuracy of approximately 5%, one could neglect the term in $\overline{m_{x,n}}(\phi) \sin \phi$ in Eq. (2). Furthermore, for $n > n_{\text{th}}$, the value of the ratio $\sigma_n = |\overline{m_{y,n}}(\phi) \cos \phi|/|\overline{m_{y,n}}(\phi)| |\cos \phi|$ for the single-Lorentzian distribution was found to be $\sigma_n \approx 1.03$ with $|\cos \phi| = 0.92$, whereas for the asymmetric distribution

the value was $\sigma_n \approx 1.05$ with $|\overline{\cos \phi}| = 0.90$. Thus, in both cases, the values of the relaxation times obtained from the reasonably accurate exponential decays are expected to be within approximately 8% of the values obtained by setting $\phi = 0$ in Eq. (2). It is also worth mentioning that for $n > n_{th}$ the ratio $(M_{x,n})^2 / (M_{y,n})^2$ is negligible for both distributions and therefore $|M_{+,n}| \approx |M_{y,n}|$.

We conclude that, as far as errors caused by magnetic field inhomogeneity are concerned, reasonably accurate relaxation times could be obtained provided $T_p / T_2^* < 0.085$.

A second possible source of error, inherent to these techniques when an inhomogeneous magnet is employed, arises from the actual values T_1 and T_2 to be measured. As the ratio T_1 / T_2 approaches unity, and the steady-state amplitude $|M_{+,s}| / M_0$ approaches 0.5, the decay from the quasi-stationary state to the steady state, may involve a too narrow range of data for a precise determination of the time-constant, especially if signal-to-noise ratio is relatively poor. Furthermore, if T_2 is very short and approaches T_2^* , considerable decay would occur before the quasi-stationary regime of Fig. 1 is attained. This could also introduce errors in the determination of $T(\alpha = \pi/2)$.

Finally, the effect of deviations of the flip angle from $\alpha = \pi/2$ caused by an inhomogeneous RF field or by errors in setting the flip angle must also be considered. The effect upon the CWFP decay is expected to be more pronounced when the ratio T_1 / T_2 is large, since in this case $\alpha = \pi/2$ differs considerably from the value α_{opt} , which yields a maximum in the normalized stationary CWFP signal amplitude. Simulations for four different flip angles were performed adopting a value $T_1 / T_2 = 10$, which yields $\alpha_{opt} = 35.12^\circ$. Fig. 3 shows the results for

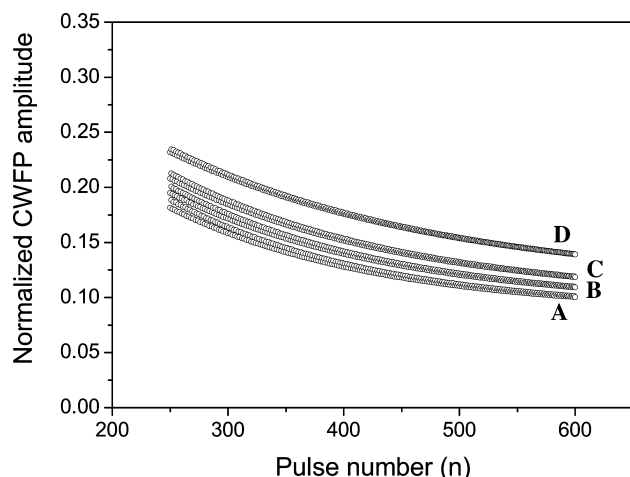


Fig. 3. Calculated CWFP decays for various flip angles α and $T_1 / T_2 = 10$. A single-Lorentzian distribution of isochromats with FWHM $\Delta f = 90$ Hz was assumed, with $T_1 = 300$ ms and $T_2 = 30$ ms. (A) $\alpha = 90^\circ$, (B) $\alpha = 85.5^\circ$, (C) $\alpha = 81^\circ$, and (D) $\alpha = 76.5^\circ$.

$T_1 = 300$ ms, $T_2 = 30$ ms, and $T_p = 0.3$ ms assuming a Lorentzian distribution of static magnetic field inhomogeneity with $\Delta f = 90$ Hz. Both even and odd pulses are shown in the region $n \geq 250$.

For this relatively large T_1 / T_2 ratio it is apparent that a flip angle $\alpha = 85.5^\circ$, 5% smaller than $\pi/2$, causes the steady-state amplitude to differ from the $\alpha = \pi/2$ case by approximately 8%. The difference becomes quite appreciable for larger deviations. Hence, good homogeneity of the RF field within the sample volume is needed in order to minimize errors, especially when $T_1 / T_2 \gg 1$.

3. T_1 and T_2 measurements

The experimental CWFP data were acquired in an Apollo Transceiver (Tecmag), using a AMT 2035 power amplifier, and a Miteq 1054 pre-amplifier. A single solenoidal coil containing a relatively small amount of sample provided adequate RF homogeneity and a $\pi/2$ pulse of 10 μ s. The receiver blocking time was 22 μ s and the separation between pulses was 300 μ s. A 2 T Oxford Magnet with a 30 cm bore was employed.

As discussed earlier, T_1 and T_2 can be obtained from the time constant $T(\alpha = \pi/2)$ and the normalized magnetization amplitude in the continuous wave regime $|M_{+,s}| / M_0$. The final result is, $T_1 = (T(\alpha = \pi/2)/2) / (|M_{+,s}| / M_0)$ and $T_2 = (T(\alpha = \pi/2)/2) / [1 - (|M_{+,s}| / M_0)]$, where $T(\alpha = \pi/2)$ and $|M_{+,s}| / M_0$ are obtained from a single CWFP experiment as shown in Fig. 2B. The amplitudes are normalized with respect to the initial signal amplitude following a $\pi/2$ pulse.

Fig. 4 shows a semi-logarithmic plot of $[|M_+(t)| / |M_{+,s}|] - 1$ for the quasi-stationary regime of the data of Fig. 2B, which exhibits a good exponential decay and permits to determine $T(\alpha = \pi/2)$ for ^1H in phosphoric acid.

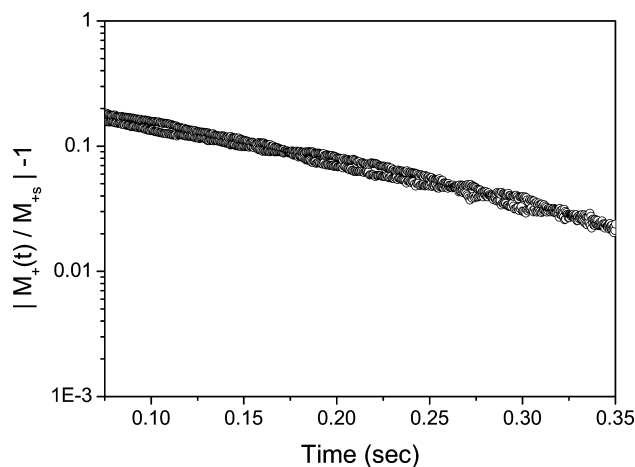


Fig. 4. Semi-logarithmic plot of $1 - [|M_+(t)| / |M_{+,s}|] - 1$ as a function of time for ^1H in phosphoric acid.

Table 1

 T_1 relaxation times measured by; IR, optimum flip angle SSFP- α_{opt} , and single CWFP- $\pi/2$ experiments

Sample	T_1 (IR) (ms)	T_1 (CWFP- $\pi/2$) (ms)	T_1 (SSFP- α_{opt}) (ms)
Deionized water (^1H)	2430 \pm 10	2460 \pm 15	2450 \pm 6
FeSO ₄ NH ₄ doped water (^1H)	24.31 \pm 0.01	22.65 \pm 0.01	22.45 \pm 0.02
Acetone (^1H)	4520 \pm 30	4260 \pm 30	4452 \pm 10
Vegetable oil (^1H)	199.60 \pm 0.01	214.60 \pm 0.02	209.70 \pm 0.01
Phosphoric acid (^1H)	191.00 \pm 0.03	190.00 \pm 0.05	203.00 \pm 0.05
Phosphoric acid (^{31}P)	746 \pm 44	741 \pm 44	655 \pm 40
2,2,2-Trifluoroethanol (^{19}F)	2008 \pm 13	2120 \pm 20	2030 \pm 20
Trifluoralin (^{19}F)	272.00 \pm 0.02	272.00 \pm 0.02	249.00 \pm 0.02

Table 2

 T_2 relaxation times measured by; CPMG, optimum flip angle SSFP- α_{opt} , and single CWFP- $\pi/2$ experiments

Sample	T_2 (CPMG) (ms)	T_2 (CWFP- $\pi/2$) (ms)	T_2 (SSFP- α_{opt}) (ms)
Deionized water (^1H)	2120 \pm 4	2400 \pm 20	2180 \pm 8
FeSO ₄ NH ₄ doped water (^1H)	15.40 \pm 0.01	15.20 \pm 0.03	17.75 \pm 0.02
Acetone (^1H)	4260 \pm 2	4030 \pm 10	4408 \pm 6
Vegetable oil (^1H)	127.00 \pm 0.01	119.70 \pm 0.04	117.20 \pm 0.01
Phosphoric acid (^1H)	140.0 \pm 0.3	160.0 \pm 0.3	170.0 \pm 0.3
Phosphoric acid (^{31}P)	178.0 \pm 0.3	145.73 \pm 0.03	128.0 \pm 0.3
2,2,2-Trifluoroethanol (^{19}F)	1832 \pm 0	2080 \pm 7	1990 \pm 5
Trifluoralin (^{19}F)	127.00 \pm 0.01	127.00 \pm 0.02	115.00 \pm 0.01

T_1 and T_2 measurements using a single CWFP experiment with a $\pi/2$ flip angle were performed on different samples and different nuclei. In all cases an exponential decay from the quasi-stationary state to a constant steady state was observed. J -couplings could, in principle, modulate the CWFP decay. However, since only monotonic decays were observed, the modulation depth can be concluded to be quite small for all the cases examined. The results are presented in Tables 1 and 2. These data are compared with inversion-recovery (IR) T_1 -measurements, Carr–Purcell–Meiboom–Gill (CPMG) T_2 -measurements [16], and optimum angle-SSFP methods [1,6]. The results show that the present method yields T_1 and T_2 values, which are in agreement with those obtained with the more time-consuming optimum flip angle method and also with those obtained by more traditional methods. Given the parameters employed in the experiments, the discrepancies cannot be attributed only to systematic errors caused by magnetic field inhomogeneity but also to other instrumental errors, which are estimated in Tables 1 and 2.

We conclude that, since for the CWFP technique a rapid pulsing regime always prevails for a wide range of experimental parameters, it can be used for the measurement of T_1 and T_2 in a single experiment without any appreciable effect caused by diffusion. This requires $1/T_p \gg \gamma G \sqrt{DT} (\alpha = \pi/2)$ where D denotes the diffusivity, G is a small magnetic field gradient caused by magnet inhomogeneities, and γ is the magnetogyric ratio. Moreover, magnetic fields of only moderate homogeneity can be tolerated. For measurements with approximately 8% accuracy, for example, the condition $T_p/T_2^* < 0.085$

appears to be sufficient. The main advantage of this method is its ability to drastically reduce the time required for the measurements of longitudinal relaxation times in liquids consisting of a single NMR line. Under appropriate conditions of the parameters involved, this could be potentially useful for the monitoring of dynamical processes involving a time evolution of the relaxation rates. To the extent changes in molecular mobility caused by these processes are not large enough to invalidate the applicability of Bloch's equations, possible spin-locking effects could be ignored and the present model could be used to interpret the decays. Moreover, transverse relaxation times are obtained in the same experiment with accuracy comparable to that provided by a CPMG sequence, provided $T(\alpha = \pi/2) \gg T_2^*$.

As with various other applications of CWFP [7,8,17], the present method should be especially useful for fast routine analytical determinations using low-resolution NMR spectrometers, as well as in several other applications [18–23].

Acknowledgments

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