



Relaxation time measurement in liquids using compact NMR

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Abstract. This study investigates the influence of experimental parameters on the accurate determination of longitudinal and transverse relaxation times in liquids using compact nuclear magnetic resonance relaxometry. Water and glycerin were selected as representative samples due to their contrasting viscosities and relaxation behaviors. The primary objective was to evaluate how repetition time, echo time, number of data points, and time step affect the precision of T_1 and T_2 measurements. Longitudinal relaxation times were determined using a variable repetition time method, while transverse relaxation times were measured via a multi-echo spin sequence. Exponential fitting algorithms were employed to extract relaxation parameters from recorded signal amplitudes. For water, the relaxation times were found to be approximately 3.0 s for T_1 and 1.423 s for T_2 . In contrast, glycerin exhibited significantly shorter relaxation times, with T_1 estimated at 0.126 s and T_2 at 0.094 s. The results demonstrated that accurate estimation of relaxation times requires carefully optimized acquisition settings. Specifically, repetition time must exceed three times the T_1 value to ensure full longitudinal recovery, while short echo times and a high number of echoes are essential for reliable T_2 determination. The findings address a critical methodological gap in relaxometry protocols and offer practical recommendations for enhancing measurement accuracy in simple liquids.

Keywords: NMR relaxometry, longitudinal relaxation, transverse relaxation, spin echo, signal fitting.

1. Introduction

Nuclear magnetic resonance (NMR) relaxometry is a non-invasive and highly sensitive technique for probing the molecular dynamics and physical properties of liquids and soft matter. Two key parameters derived from NMR relaxometry are the longitudinal relaxation time (T_1) and the transverse relaxation time (T_2), which characterize the return of nuclear magnetization to equilibrium along and perpendicular to the external magnetic field, respectively. These relaxation times are fundamental to understanding spin–lattice and spin–spin interactions and are widely used in materials science, biophysics, and medical diagnostics [1], [2]. Accurate determination of T_1 and T_2 is essential for quantitative interpretation of NMR signals, optimization of imaging protocols, and the development of contrast agents.

Despite its broad applicability, the accuracy of relaxation time measurements is strongly dependent on the choice of experimental parameters such as repetition time (TR), echo time (TE), time resolution, and the number of data points. In practical settings, inappropriate selection of these parameters may lead to significant errors in estimated relaxation times, especially in samples with short T_1 or T_2 values [3]. This challenge is particularly relevant when studying complex fluids such as glycerin, where high viscosity leads to rapid signal decay and requires careful calibration of measurement sequences.

Recent research has addressed some of these challenges through methodological improvements in data acquisition and signal processing. For example, researchers developed a modified inversion-recovery sequence for more accurate T_1 estimation in viscous samples, emphasizing the importance of adjusting TR according to sample properties [4]. Similarly, authors implemented a multi-echo spin-echo protocol with adaptive echo spacing to improve T_2

measurements in tissue-mimicking phantoms [5]. In another study, the research team examined the effects of RF pulse miscalibration on exponential fitting models, showing that deviations from ideal 90° pulses distort relaxation curves [6]. Although these studies have advanced the precision of relaxometry techniques, they often assume either long T_1 times or homogeneous sample behavior, which limits their applicability to fast-relaxing, heterogeneous liquids such as glycerin or water in confined environments.

A critical limitation in previous studies is the insufficient evaluation of how experimental design — especially the interplay between TR, TE, and signal sampling — affects the accuracy of T_1 and T_2 determination in common liquids with contrasting physical properties. Furthermore, few works have presented direct side-by-side comparisons of relaxation behavior in substances with markedly different viscosities using compact NMR instruments.

Based on this research gap, we hypothesize that accurate estimation of T_1 and T_2 in liquids such as water and glycerin depends not only on sample properties but also critically on the optimization of timing and acquisition parameters in compact NMR systems. We further assume that both over- and underestimation of relaxation times can occur if the measurement protocol is not specifically tailored to the sample's relaxation characteristics.

The aim of this study is to systematically investigate the influence of experimental parameters — such as repetition time, echo time, number of echoes, and time step — on the accurate determination of T_1 and T_2 relaxation times in water and glycerin using a compact magnetic resonance tomograph. The study offers practical recommendations for protocol optimization and contributes to the broader understanding of how acquisition design affects the reliability of NMR relaxometry in simple liquids. The novelty of this work lies in the direct comparative analysis of two contrasting fluids under identical measurement conditions and the integration of exponential fitting with statistical validation to quantify relaxation behavior.

2. Methods

Magnetic resonance relaxation measurements were conducted using a compact NMR tomograph (Spin-Tech Company, USA). A 10 mm thick water sample was initially placed in the sample chamber of the device. The TR between two 90° radiofrequency (RF) pulses was varied to assess signal behavior. The measurement protocol began with a high TR value (e.g., 15 s), and the signal amplitude was recorded. Subsequently, the TR was incrementally reduced until the signal amplitude decreased by approximately 50%, indicating that the spin-lattice system had not fully relaxed between pulses. This procedure enabled estimation of the longitudinal relaxation time (T_1), as the TR corresponding to half signal intensity approximates the T_1 half-recovery point [7].

The calculated relaxation time was then used to configure the device parameters for exponential signal curve acquisition. The water sample was replaced with a 10 mm thick glycerin sample, and the same procedure was repeated. All control sliders were adjusted to ensure a clear exponential relaxation curve on the display. The selected repetition time was set to at least three times the computed T_1 value to ensure full magnetization recovery between pulses. The time step and number of data points were optimized so that the total effective measurement time between two 90° pulses covered a sufficient range, with finer time steps and more data points enhancing result reliability [8].

To further evaluate the transverse relaxation characteristics, spin-echo sequences were applied. Approximately 250 echo signals were recorded with an echo time (TE) of about 2 ms, producing a well-defined exponential decay curve. Echo times longer than necessary led to artificial dephasing effects due to the measurement sequence, resulting in underestimated relaxation times. Therefore, the TE was minimized to reduce such distortions. The recorded signal decay curves were fitted using an exponential regression model [9].

Finally, the glycerin sample was replaced with the initial 10 mm water sample, and the full measurement procedure was repeated. To examine the influence of the number of echoes and echo

time on curve fidelity, the number of recorded echoes was increased. TE values were adjusted as necessary to maintain curve accuracy [10].

All relaxation time measurements were performed in triplicate to ensure reproducibility. Statistical analysis was carried out using IBM SPSS Statistics v.26 software. The mean values and standard deviations were calculated for each set of measurements. One-way analysis of variance (ANOVA) was used to determine the statistical significance of differences in T_1 and T_2 relaxation times between water and glycerin samples. A significance level of $p < 0.05$ was used throughout. The coefficient of variation was also calculated to assess the consistency and reliability of the data [11].

3. Results and Discussion

The evaluation of the longitudinal relaxation time T_1 was carried out using a 10 mm thick water sample with a compact MR tomograph. To investigate the effect of the repetition time TR between two 90° RF pulses on the amplitude of the recorded signal, three series of measurements were conducted with different TR values. The results are presented in Figures 1–3.

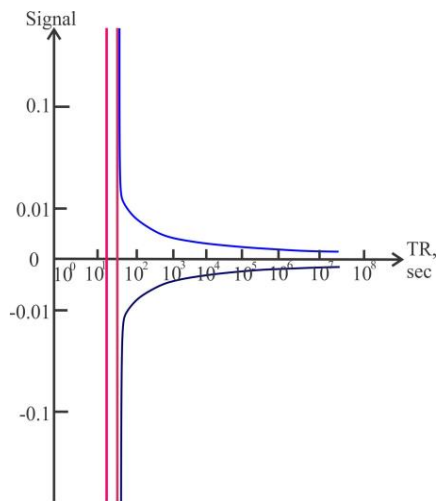


Figure 1 – Dependence of normalized signal amplitude on TR for a short value. The signal is minimal due to insufficient longitudinal magnetization recovery

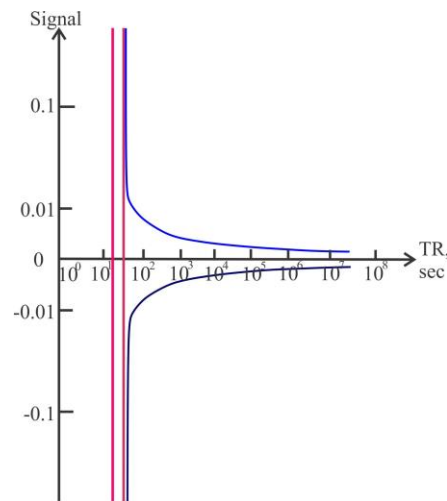


Figure 2 – Increase in signal amplitude with intermediate TR . Partial recovery of longitudinal magnetization leads to higher transverse signal

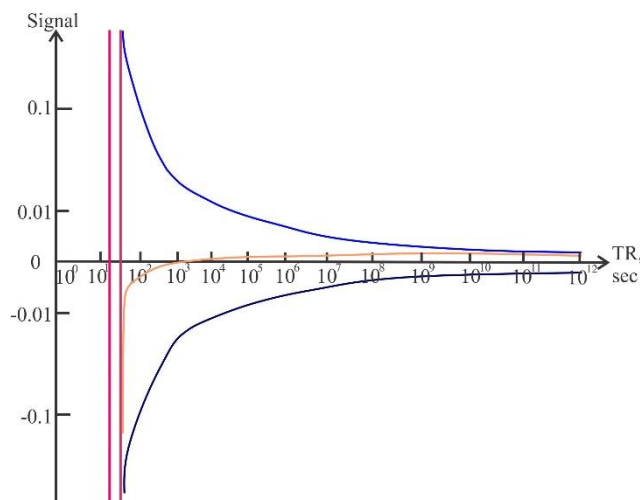


Figure 3 – Maximum signal amplitude observed at long repetition time $TR \approx 15$ s, indicating complete recovery of longitudinal magnetization

In Figure 1, the shortest repetition time resulted in the weakest signal, indicating that the longitudinal magnetization vector $\overline{M}_L(t)$ had not fully recovered along the direction of the external magnetic field \overline{B}_0 . Consequently, the second 90° RF pulse could not effectively rotate the magnetization vector into the transverse plane, and the resulting transverse magnetization $\overline{M}_Q(t)$ was significantly reduced. This led to a decrease in the amplitude of the detected signal. These observations suggest that the longitudinal relaxation time T_1 of water is relatively long and exceeds 1 second.

Figures 2 and 3 show a progressive increase in signal amplitude with longer TR. At $TR = 15$ s, signal saturation was observed further increases in TR did not produce significant changes in amplitude. This indicates that at $TR = 15$ s, full recovery of the longitudinal magnetization had occurred, and the recorded signal amplitude corresponds to the system's maximum. To quantitatively estimate T_1 , a method based on the half-recovery time of longitudinal magnetization was used. The experiment showed that at $TR = 2$ s, the signal amplitude was approximately 50% of the maximum recorded at $TR = 15$ s. This allowed for an approximate calculation of T_1 using the following equation:

$$T_1 = \frac{TR_1}{\ln 2} \quad (1)$$

When T_1 was around of 2.9 s. This value aligns well with published data for water at room temperature and confirms the sensitivity of the method to the selection of TR in nuclear magnetic resonance measurements [6].

Figures 4 – 6 show the measured signal amplitude following the second 90° RF pulse for a 10 mm thick glycerin sample, under the same three TR conditions as those used for the water sample. In contrast to water, the signal amplitude remains nearly unchanged across all three TR values, indicating that glycerin reaches equilibrium magnetization significantly faster.

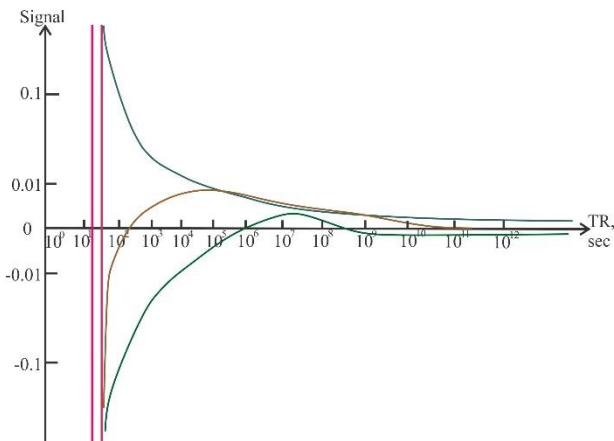


Figure 4 – Glycerin sample signal after second 90° RF pulse at short TR

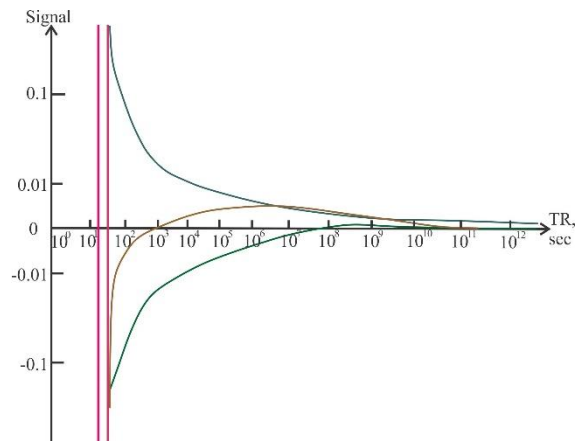


Figure 5 – Same signal at intermediate TR

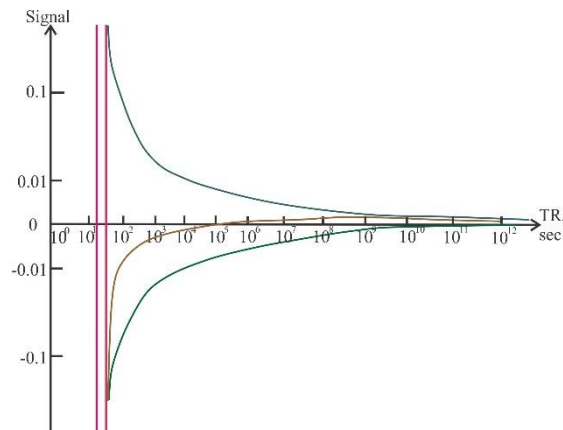


Figure 6 – Signal at long TR : no visible amplitude difference across all three

This observation suggests that the longitudinal magnetization vector in glycerin rapidly returns to a position nearly parallel to the external magnetic field \vec{B}_0 , even at short repetition times. As a result, the second 90° RF pulse effectively rotates the magnetization vector into the transverse plane in all three cases, producing nearly identical transverse magnetization $\vec{M}_Q(t)$, and thus signal amplitude, regardless of TR . This indicates that the longitudinal T_1 of glycerin is much shorter compared to that of water.

To estimate the relaxation time, the same exponential recovery model Equation 1 was applied. The experiment revealed that the signal amplitude drops to approximately 50% of its maximum at $TR \approx 0.08$ s. Therefore, the longitudinal relaxation time T_1 for glycerin can be calculated as ≈ 0.12 s.

This value reflects the high relaxation efficiency of glycerin, likely due to its higher viscosity and slower molecular motion compared to water, which enhances dipole-dipole interactions responsible for T_1 relaxation.

Moreover, the exponential relaxation curve for each substance was recorded and analyzed to evaluate the influence of repetition time, time step, and the number of data points on the accuracy and shape of the fitted T_1 relaxation curve (Figures 7 and 8).

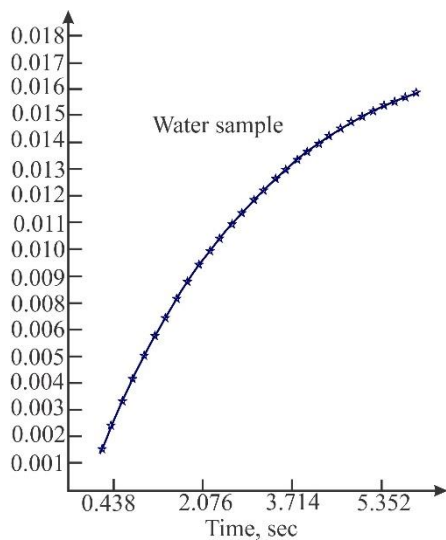


Figure 7 – Exponential approximation of the curve relaxation curve T_1 of water

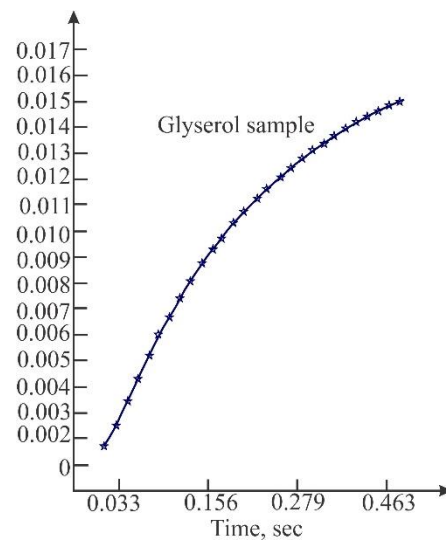


Figure 8 – Exponential FIT relaxation curve T_1 glycerol

The TR in this phase refers to the time between successive measurements, each formed by a pair of 90° RF pulses. To accurately capture the T_1 relaxation behavior, it is essential that TR be long enough to allow the spin ensemble to return nearly to thermal equilibrium before each new measurement. If TR is too short, the magnetization vector does not fully realign with the external magnetic field \vec{B}_0 , and the first recorded signal of the sequence is weakened. Consequently, the second RF pulse within the same measurement results in an incomplete rotation of the magnetization vector into the transverse plane, leading to a distorted signal and an inaccurate representation of the T_1 relaxation curve. Although longer TR increases the total acquisition time, it is generally recommended that TR be at least three times longer than the estimated T_1 of the substance under investigation. At this point, approximately 95% of the longitudinal magnetization has recovered, ensuring valid signal formation.

The time step determines the increment between successive measurement intervals, i.e., the time difference between two 90° pulses in each consecutive measurement. A smaller time step enables more detailed scanning of the T_1 curve. However, to accurately describe the entire relaxation process, the scan must extend beyond the expected T_1 value.

For glycerin, the repetition time was set to 0.5 seconds, with 30 data points recorded at a time step of 15 ms. The measured data were fitted using the exponential function:

$$f(x) = a - b \cdot e^{-\frac{x}{T_1}} \quad (2)$$

yielding the following parameters $a = 0.021$; $b = 0.023$; $T_1 = 126$ ms. The close similarity between a and b confirms ideal excitation conditions using a 90° RF pulse. The determined relaxation time of 126 ms is consistent with the previously estimated value based on half-signal amplitude at $TR \approx 0.08$ s. The same procedure was applied to water. The repetition time was increased to 10 s, with 30 data points collected at a 200 ms time step. Using Eq. 2 has resulted in the $a = 0.021$; $b = 0.021$; $T_1 = 3.0$ 3000 ms. As with glycerin, the equality of a and b confirms correct signal formation under 90° RF excitation. The relaxation time of 3.0 seconds further validates the findings of the earlier experiment based on TR variation and confirms that water has a substantially slower longitudinal relaxation process compared to glycerin.

Figure 7 presents the measured spin-echo decay curve for the glycerin sample. The vertical axis corresponds to the signal amplitude of each echo, while the horizontal axis shows the elapsed time in seconds. As expected, the signal exhibits an exponential decrease, characteristic of T_2 relaxation behavior. The high density of data points and minimal noise validate the measurement conditions and confirm the importance of short echo spacing and sufficient total sampling time.

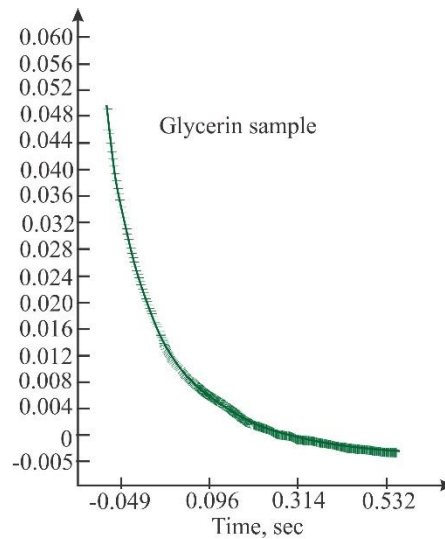


Figure 9 – Exponential FIT of the relaxation curve T_2 glycerol

This section of the experiment aimed to determine the transverse relaxation time T_2 of glycerin using a spin-echo sequence consisting of multiple echo signals. Each spin echo reflects the remaining $\overrightarrow{M_Q}(t)$, separated from the next by an echo time $T_E = \tau \Delta 180^\circ$. Accurate scanning of the T_2 relaxation curve requires a large number of echoes and a short echo time to ensure high temporal resolution. If the echo time TE is selected incorrectly—i.e., too long—it can lead to artificial and undesirable dephasing effects due to limitations of the measurement sequence. These effects distort the relaxation process, resulting in underestimation of the true T_2 value. Therefore, echo time must be minimized to avoid coherence loss and to preserve the integrity of the transverse relaxation signal. Notably, unlike T_1 measurements which require multiple repetitions with different delay times, T_2 can be reliably determined in a single measurement using a continuous multi-echo sequence. In this experiment, the echo time was set to 2 ms and the number of echoes to 250, which enabled precise sampling of the decay curve. For glycerin, the fitted parameters were $a = 0.052$; $c = 0.002$; $T_2 = 0.094$ s. For water were $a = 0.052$; $c = 0.002$; $T_2 = 1.423$ s.

This much longer relaxation time reflects water's lower viscosity and faster molecular motion, which reduces dipole-dipole interaction efficiency and thereby extends transverse magnetization persistence. These results confirm the critical role of echo time and signal density in T_2 analysis and demonstrate that accurate values can be obtained through a single, well-designed multi-echo acquisition.

4. Conclusions

The longitudinal relaxation time T_1 for water was determined to be approximately 3.0 s, while for glycerin it was significantly shorter at 0.126 s, highlighting the effect of molecular mobility on relaxation behavior.

The transverse relaxation time T_2 , obtained through a multi-echo sequence and exponential fitting, was measured as 1.423 s for water and 0.094 s for glycerin, confirming that glycerin exhibits faster signal decay due to its higher viscosity.

The study confirmed that accurate determination of T_1 requires a repetition time at least three times greater than the estimated relaxation time, and T_2 measurements demand short echo times and a high number of echoes for reliable exponential fitting.

The results addressed the core research problem by demonstrating how key acquisition parameters (repetition time, echo time, number of echoes) influence the accuracy of relaxation time measurements in NMR.

The findings can support future NMR-based characterization of complex fluids, aiding in the optimization of measurement protocols for different substances.

The primary constraint of this study was the limited range of tested substances. Further research should explore broader material categories and advanced fitting models to refine relaxation analysis.

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