

Self-diffusion coefficient by single-sided NMR

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Abstract

It is presented a novel method for the measure of the self-diffusion coefficient. The method exploits the fixed gradient of an open magnet, as that used in single-sided NMR, and it does not use prior information on T_2 . The approach presented in this paper can be practiced also on the fringe field of superconducting magnets and it is based on the construction of the ratios between echoes taken at different interpulses separation in a Carr–Purcell–Meiboom–Gill pulse sequence. The determination of the self-diffusion coefficient facilitates the estimate of T_2 because the transverse relaxation results almost influenced by the molecular diffusion effect, also at the shorter interpulses time, when it is measured in field strongly inhomogeneous.

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1. Introduction

The single-sided NMR devices based on the NMR-MOUSE [1,2] find continuously new field of application [3] thanks to the extreme versatility of their use and because their mobility allows reaching unmovable sample as, for example, the great part of the works of art [4]. In this field, moreover, the non-invasive and the non-destructive character of single-sided NMR experiments add fundamental qualities for the safeguard and research about Cultural Heritage.

The price paid for this richness of possibilities is quite high and makes the single-sided NMR an “extreme condition” NMR. Besides the low intensity field of polarization in fact, the main characteristic of this device is a strong magnetic field inhomogeneity caused by the open geometry of the permanent magnet [5]. Therefore, chemical shift cannot be measured — the use of the so-called nutation echoes [6] is very difficult to carry out and a serious signal loss from translational diffusion is in practical inevitable, at least on liquid sample [7]. This feature seems to address the use of such surface high inhomogeneity probes (SHIP) on sample with minor or

absent translational diffusion, as materials characterized by solid-like behavior [3].

Actually to get the true T_2 from SHIP measurements it is necessary to match some special conditions, namely the self-diffusion coefficient should be very small and the transverse relaxation should have enough liquid-like character to refocus Hahn echoes. Or, to use multi-pulses sequence like the Carr–Purcell–Meiboom–Gill (CPMG) one, with interpulses delay as short as possible, to minimize the diffusion effect [8,9]. Not any of these conditions can be fully realized and, however, in most cases, this feature may limit the full employ of SHIP relaxometry.

In this paper we present a way to measure the self-diffusion coefficient D utilizing the fixed gradient of SHIP and without prior knowledge of T_2 . With this result we approach two objectives. The first is to utilize an inexpensive device, with fixed gradient, to measure D independently by the field inhomogeneity distribution, by making use of a simple method, which can be employed also on the fringe field of superconducting magnet [10]. The second objective concerns the measure of T_2 by SHIP: the determination of D would allow getting a realistic T_2 distribution by correcting the transverse relaxation from the diffusion effect. Even if this last aspect is in progress and it is outside the scope of the present work, it is quite intuitive to recognize that

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the measure of D can facilitate the quantification of the T_2 distribution in single-sided measurements: some examples of such a valuation of T_2 are however reported.

The method is based on the comparison, at the same evolution time, of CPMG echoes obtained at different interpulses delays. By this way the relaxation data become independent from T_2 and may be fitted with a function dependent on the time pulses density, with D as free parameter. The results obtained on different liquids with different D match very well with the data reported in literature and measured with the traditional methods. Moreover, some T_2 valued by SHIP, after the correction of the diffusive effect, match satisfactory well with those valued in homogeneous field.

2. Theory

The SHIP provides both the radiofrequency (B_1) and the polarizing (B_z) fields [1]. The strong inhomogeneity of the B_z field together with the use of a surface coil, which generates, as well, a high inhomogeneous B_1 , make the echo train produced by Carr–Purcell-like pulse sequences to result affected by superposition of Hahn and stimulated echoes as a result of the spatial distribution of pulse flip angles [5]. The CPMG is one of the pulse sequences that generates the most regular echo train in the SHIP measurement conditions, although it shows distortion effect mainly on the few first echoes [5,11]. Because, in what follow, we work on non-confined liquids ($T_1 \approx T_2$) using hundreds or thousands echoes, we can neglect this effect choosing to use the CPMG sequence in all experiments. On the other hand we are mainly interested in the D effect, which, for a high number of echoes, it is almost independent from imperfection related to T_2 evaluation by SHIP [11]. Nevertheless, the experimental values of T_2 that we get, after the self-diffusion correction effect, they are very close to the ones measured in homogeneous field.

It is well known that the echo attenuation due to molecular translational diffusion under the influence of field gradients may be reduced by multi-pulse sequence like the CPMG. The NMR response in this case is, supposing a homogeneous sample with single T_2 and ordinary diffusion, in the slow exchange limit

$$M(\tau) = \exp\left[-\frac{2n\tau}{T_2}\right] m_0 \int_V d\vec{r} P(\vec{r}) \times \exp\left[-\frac{2n\tau}{3} \gamma^2 D \left|\frac{\partial \vec{B}_z}{\partial \vec{r}}\right|^2 \tau^2\right], \quad (1)$$

where m_0 is the local equilibrium magnetization, $P(\vec{r})$ is a function which modulates the detection of m_0 according to the flip angles distribution, V is the sample volume or the sensitive volume of SHIP, τ is the time interval between pulses and n runs the number of 2τ

cycles. The time-independent gradient is $\partial \vec{B}_z / \partial \vec{r}$ where $\vec{B}_z = B_z(\vec{r})\hat{z}$ describes the polarizing field inside the sensitivity volume of SHIP.

In liquid-like spin system, the self-diffusion coefficient is normally measured by Hahn echo sequence utilizing field gradient of modifiable intensity and duration or, for fixed gradient, using a prior knowledge of T_2 [12,13]. For fixed field inhomogeneity and without prior knowledge of T_2 , the dependence of τ in Eq. (1) may be used to weight differently the self-diffusion effect on transverse relaxation in CPMG sequences. In the case of constant and known gradient $|\partial \vec{B}_z / \partial \vec{r}| = G$, by collecting different echo trains with different τ and building up their ratio R_n at time $t = 2n\tau_i = 2n'\tau' = 2n'm\tau_i$ one gets the ratio function

$$R_m(n\tau_i) = \exp\left[-\frac{2n\tau_i}{3} \gamma^2 D G^2 (m^2 - 1) \tau_i^2\right], \quad (2)$$

which does not depend on both $m_0 \int_V d\vec{r} P(\vec{r})$ and T_2 . The fits on the $n\tau_i$ variable, for all the i values and for the set of all possible m ratios, are able to furnish directly D .

Typically the whole inhomogeneity distribution produced by SHIP permanent magnet arrangement is quite complicate and has not an analytical form nor it can be known in great detail. Variation of the magnetic field occurs in any spatial direction with linear, quadratic, cubic, etc. terms that only a detailed analysis can broke down into spherical harmonics. For our SHIP both experimental field mapping and numerical simulation data [14] give a field distribution well described by $\vec{B}_z = B_0\hat{z} + cx^2\hat{z} + dy^2\hat{z} + bz^2\hat{z}$, where B_0 is a constant term and the xyz frame orientation, with respect to the SHIP geometry, is reported in Fig. 1. Nevertheless, being the sensitivity volume of SHIP roughly parallelepiped-shaped with sides x – y – z of about 2.0–0.2–0.8 cm, namely along y the size is very small, and because $c \ll d \approx b$, the field distribution, inside the sensitivity volume, can be simplified by $\vec{B}_z \cong B_0\hat{z} + ay\hat{z} + bz^2\hat{z}$, where the parameter $a \neq d$ allows to approximate the quadratic term to a linear term (the parameter a changes when the sensitivity volume changes).

For this field distribution $\partial \vec{B}_z / \partial \vec{r}$ is a function of the location and of the volume of the sample, if the sample is smaller than the sensitivity volume of SHIP; otherwise it depends only by the sensitivity volume [15]. Still in the slow exchange limit Eq. (1) becomes

$$M(\tau) = \exp\left[-\frac{2n\tau}{T_2}\right] m_0 \int_V d\vec{r} P(\vec{r}) \times \exp\left[-\frac{2n\tau}{3} \gamma^2 D [a^2 + (2bz)^2] \tau^2\right], \quad (3)$$

where V , as in Eq. (1), is the volume of the sample or that of SHIP. For the sake of simplicity and generality, by assuming a sample of side L_z along the z axis, the ratio function Eq. (2) in this case becomes

$$R_m(n\tau_i) = \exp \left[-\frac{2n\tau_i}{3} \gamma^2 D a^2 (m^2 - 1) \tau_i^2 \right] \frac{1}{m} \times \frac{\text{erf}(mQ_{n\tau_i}L_z)}{\text{erf}(Q_{n\tau_i}L_z)}, \quad (4)$$

where $Q_{n\tau_i} = \sqrt{\frac{4}{3}\gamma^2 D b^2 2n\tau_i^3}$, which, as in Eq. (2), it does not depend on T_2 . In Eq. (4) it has been supposed that along z , inside the sensitivity volume, the flip angle

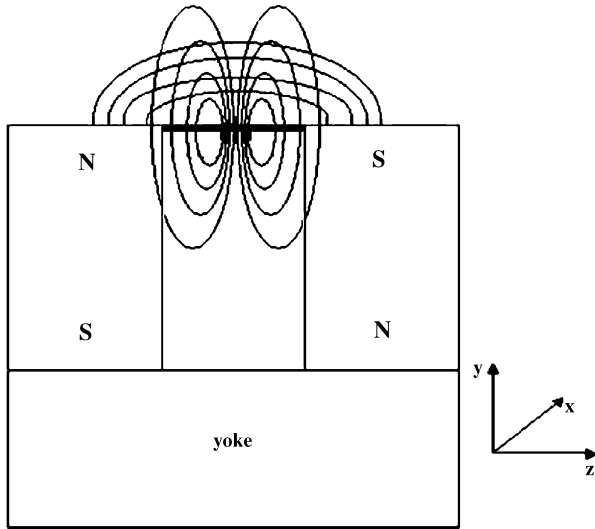


Fig. 1. The reference frame axis for SHIP is reported. The origin of the reference frame is supposed to be in the geometrical center of the gap, on the magnet faces plane. The tube sample was placed along the x -axis between the two magnets. The dimensions of Bruker SHIP are about $8 \times 8 \times 8$ cm. The gap between the two magnets is about 2 cm. The radiofrequency surface coil is positioned into the gap (thick line) with its magnetic axes oriented along y [1].

distribution is almost constant. This is quite well verified in surface coil along directions parallel to the coil plane (the xz plane in Fig. 1) within the coil dimension (moving along y , the xz homogeneity interval becomes narrower) while the larger inhomogeneity effect is along the direction perpendicular to the coil plane (the y direction in Fig. 1) [16]. In our SHIP the variation of the B_1 field along z can be valued in few percent within the coil dimension, which is however slightly larger the sensitivity volume along this direction. This approximation makes Eq. (4) independent from the equilibrium magnetization. When the flip angle distribution cannot be considered constant, the erf function should be replaced by an integral like $m_{0z} \int_{L_z} dz P(z) \exp[-(2n\tau/3)\gamma^2 D (2bz)^2 \tau^2]$, where $P(z)$ takes in account the distribution of the pulse flip angle along z and m_{0z} is the term which allows to integrate the constant local equilibrium magnetization along the same axes (it is still supposed a homogeneous sample). The other two integrals, like $m_{0x} \int_{L_x} dx P(x)$, along x and y , are canceled in the ratio function. The appearance of $P(z)$ in the erf integral does not change appreciably the fitting procedure when the B_1 spatial distribution is known, also because the m_{0z} term is canceled in the ratio function. By fixing the a and b parameters, the fit of Eq. (4) on data taken at different m and τ_i is able to furnish directly the value of D .

3. Results and comments

The schematic of SHIP utilized in our measurements is reported in Fig. 1. The apparatus, called Bruker

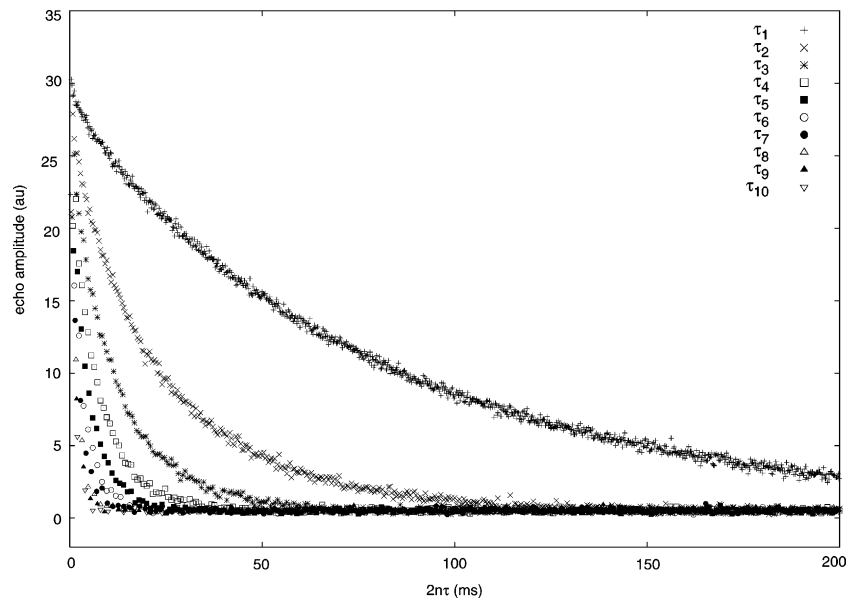


Fig. 2. The CPMG results at 14.2 MHz taken with different τ_i on octan-1-ol. The 2τ values utilized in this measurement were $2\tau_1 = 200 \mu\text{s}$, $2\tau_2 = 400 \mu\text{s}$, $2\tau_3 = 600 \mu\text{s}$, $2\tau_4 = 800 \mu\text{s}$, $2\tau_5 = 1000 \mu\text{s}$, $2\tau_6 = 1200 \mu\text{s}$, $2\tau_7 = 1400 \mu\text{s}$, $2\tau_8 = 1600 \mu\text{s}$, $2\tau_9 = 1800 \mu\text{s}$, and $2\tau_{10} = 2000 \mu\text{s}$. For each τ_i measurement 512 scans have been used. In this figure, as well as in all the others below, the density of experimental points as been reduced to make the different symbols distinguishable.

Professional Mouse, is produced by Bruker Biospin and works at a Larmor frequency of 15.8 MHz, within a layer of about 0.2 cm from the magnet surface along the y direction (it is in such a case that the sensitivity volume x - y - z is about 2.0–0.2–0.8 cm). Two other different rf coils, resonant at 14.2 and 13.8 MHz, allow reaching layers centered, respectively, at a deep of 0.3 and 0.5 cm along the y direction with sensitivity volumes slightly

different relating to the one at 15.8 MHz. In this work we use only the 15.8 and 14.2 MHz coils. The dead time is about 20 μ s and the $\pi/2$ pulse is about 3 μ s long. The indicative dimensions of the magnet are reported in the caption of the figure.

Six different liquids have been used to test the efficacy of our approach. They are acetone ($D = 4.57 \times 10^{-5}$ cm²/s), water ($D = 2.30 \times 10^{-5}$ cm²/s), ethanol ($D =$

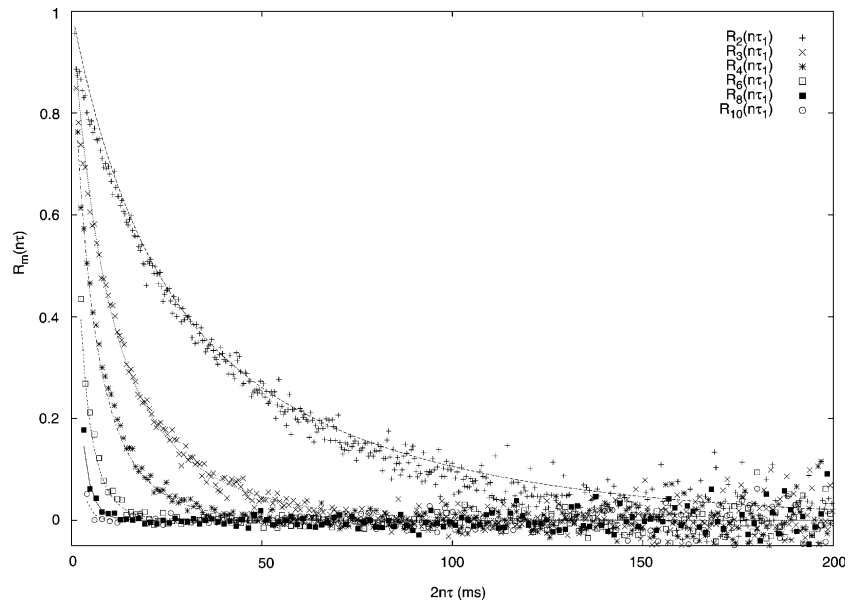


Fig. 3. In this figure few ratio functions ($R_2(n\tau_1)$, $R_3(n\tau_1)$, $R_4(n\tau_1)$, $R_6(n\tau_1)$, $R_8(n\tau_1)$, $R_{10}(n\tau_1)$) with $2\tau_1 = 200 \mu$ s on octan-1-ol are reported. The fits made by Eq. (4) are reported too.

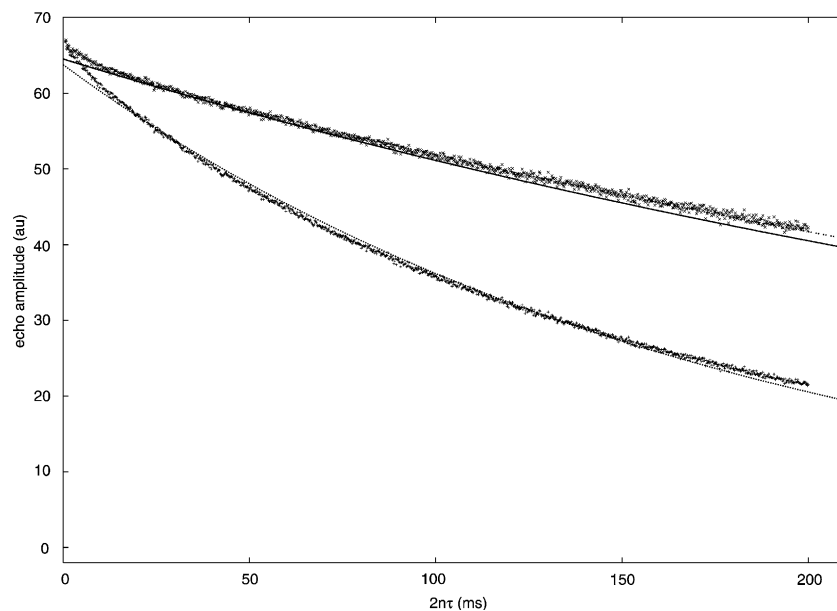


Fig. 4. The 15.8 MHz CPMG echoes on octan-1-ol at $2\tau = 200 \mu$ s, (lowest curve) with the data rectified by the diffusion effect (highest curve), are reported. Superimposed to each curve there is the fit by which T_2 has been estimated. The full line reports the decay of octan-1-ol measured at 20 MHz in homogeneous field.

$1.08 \times 10^{-5} \text{ cm}^2/\text{s}$), formamide ($D = 0.55 \times 10^{-5} \text{ cm}^2/\text{s}$), octan-1-ol ($D = 0.14 \times 10^{-5} \text{ cm}^2/\text{s}$), and glycerol ($D = 0.0022 \times 10^{-5} \text{ cm}^2/\text{s}$). The self-diffusion coefficient values reported in brackets are those at a temperature of 25°C [17,18]. The choice of these solvents has been made to explore a very large range of D . The liquids have been inserted in an NMR tube of 1 cm diameter and filled up to 10 cm. The measurements have been made with the NMR tubes set along the magnet gap (the x direction of Fig. 1). Being the samples along the y and the z directions of smaller sizes and of different shapes with respect to those of the sensitivity volume of SHIP, the integral in Eq. (4) is made on the sample volume which falls into

the sensitivity volume of SHIP. Although the sensitivity volume of Bruker SHIP at 14.2 MHz is slightly bigger with respect to that at 15.8 MHz, it result however smaller than the sample volume and therefore the integral of Eq. (4) is made on the sensitivity volume of SHIP. All measurements have been performed at a temperature of $25 \pm 2^\circ\text{C}$.

In Fig. 2 some CPMG results at a Larmor frequency of 14.2 MHz (in the follow, to reduce the number of figures, are reported example of data either at 15.8 or at 14.2 MHz), taken with different τ_i on octan-1-ol, are reported. All the 2τ values utilized in this measurement were $2\tau_1 = 200$, $2\tau_2 = 400$, $2\tau_3 = 600$, $2\tau_4 = 800$, $2\tau_5 =$

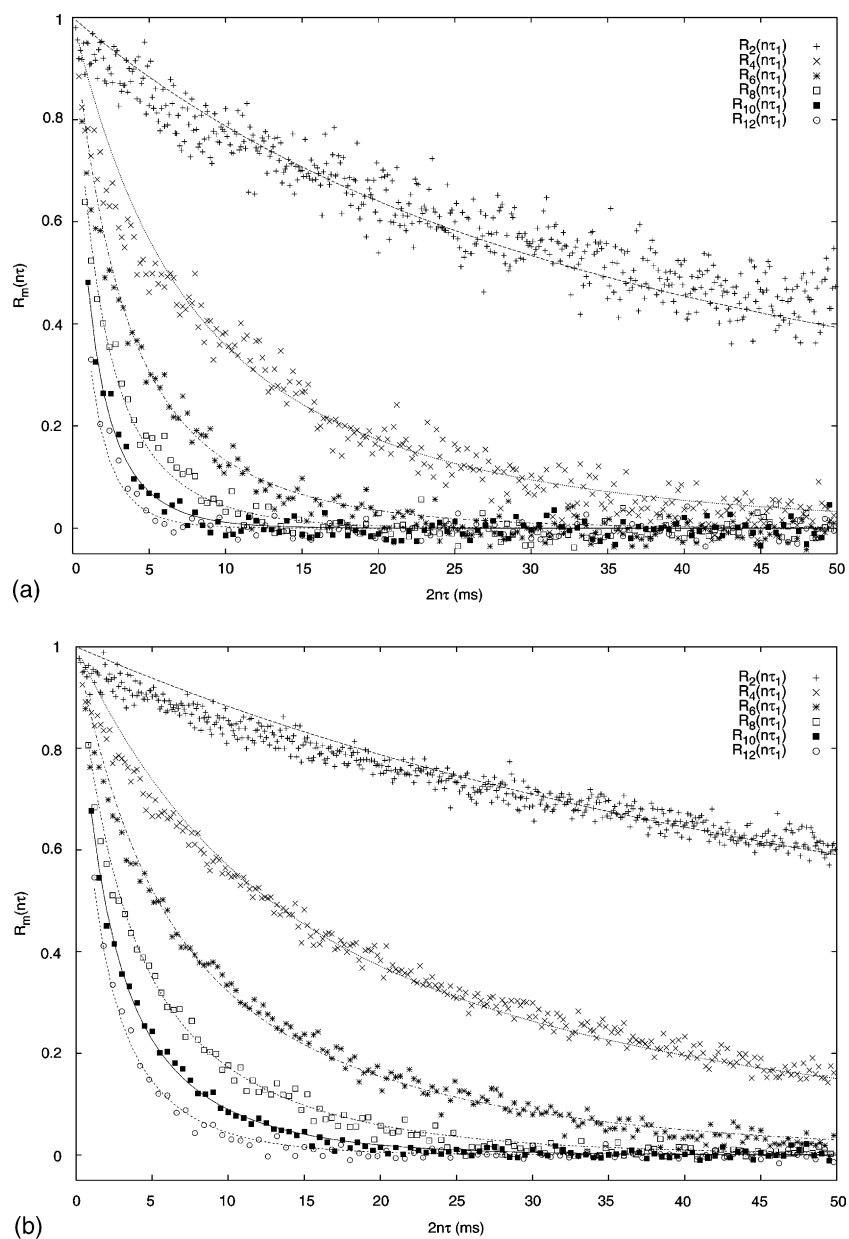


Fig. 5. The ratios $R_2(n\tau_1)$, $R_4(n\tau_1)$, $R_6(n\tau_1)$, $R_8(n\tau_1)$, $R_{10}(n\tau_1)$, $R_{12}(n\tau_1)$, where $2\tau_1 = 50 \mu\text{s}$, are reported for acetone (a), water (b), ethanol (c) and formamide (d). The fits made by Eq. (4) are also reported.

1000, $2\tau_6 = 1200$, $2\tau_7 = 1400$, $2\tau_8 = 1600$, $2\tau_9 = 1800$, and $2\tau_{10} = 2000 \mu\text{s}$. For each τ_i measurement 512 scans have been used. The same numbers of scans have been used also for all the samples utilized in this work.

The ratio functions Eq. (4), which can be realized with these choice of τ_i , are $R_2(n\tau_1), \dots, R_{10}(n\tau_1), R_2(n\tau_2), R_3(n\tau_2), R_4(n\tau_2), R_5(n\tau_2), R_2(n\tau_3), R_3(n\tau_3), R_2(n\tau_4)$, and $R_2(n\tau_5)$. In Fig. 3 some of these ratio functions, are reported. In the same figure the fits made by Eq. (4) are also reported. All parameters have been evaluated by fitting the weighted sums of square deviations up to convergence. We used the MINUIT program, from CERN library, to find the minimum of χ^2 with respect to each parameter. We considered the χ^2 of all the 16 ratio

functions reported above. The iteration procedures continued up to a convergence limit of 10^{-13} . The same data handling has been used for all the samples.

By utilizing $a = 6.5 \text{ T/m}$ and $b = 1.76 \text{ kT/m}^2$ at 15.8 MHz and $a = 16.8 \text{ T/m}$ and $b = 0.93 \text{ kT/m}^2$ at 14.2 MHz [14], the results of the fit for octan-1-ol give $D = 0.13 \times 10^{-5} \text{ cm}^2/\text{s}$ at 15.8 MHz and $D = 0.13 \times 10^{-5} \text{ cm}^2/\text{s}$ at the deep of 0.3 cm. The error on D is estimated to be about 2%.

In Fig. 4, the 15.8 MHz CPMG echoes of octan-1-ol at $2\tau = 200 \mu\text{s}$, namely at the shorter interpulse time, are compared with the data rectified by the diffusion effect. The transverse relaxation time estimates from the rectified data is about $T_2 = 460 \text{ ms}$ while that valuated in

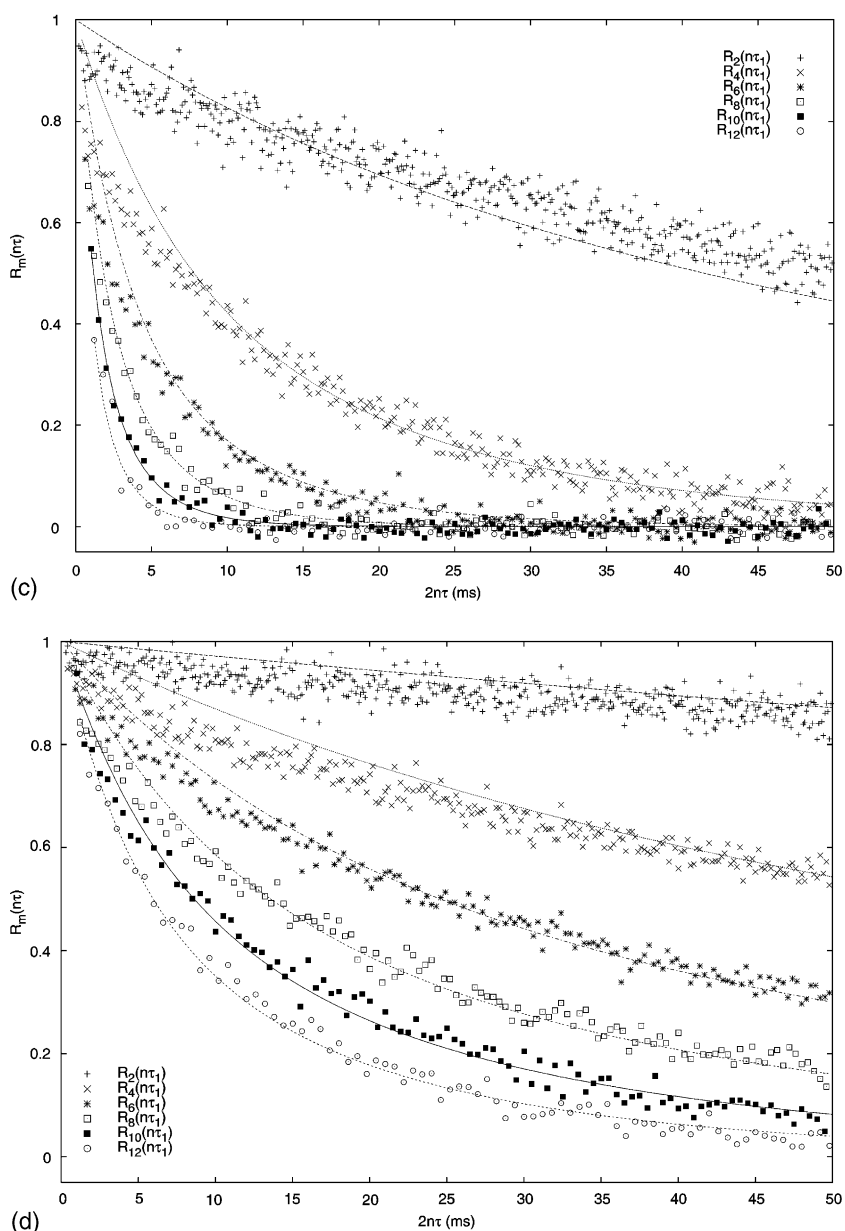


Fig. 5. (continued)

homogeneous field, at 20 MHz, is about $T_2 = 430$ ms. The T_2 valuated without the correction of the self-diffusion effect was about $T_2 = 100$ ms. For glycerol (see below) we estimate about $T_2 = 23$ ms from the rectified data while T_2 valuated in homogeneous field, at 20 MHz, is about 24 ms. Even if the Larmor frequencies are slightly different (we have not a closer frequency experimental point), the results are very similar to that measured in homogeneous field and it is very promising for the utilization of SHIP for transverse relaxation measurements also in liquid-like spin systems.

In Fig. 5a some ratio functions at 15.8 MHz on acetone are reported. The 2τ values utilized in this measurement were $2\tau_1 = 50$, $2\tau_2 = 100$, $2\tau_3 = 200$, $2\tau_4 = 300$, $2\tau_5 = 400$, $2\tau_6 = 500$, and $2\tau_7 = 600$ μ s. The ratio functions realized with these choice of τ_i , are $R_2(n\tau_1)$, $R_4(n\tau_1)$, $R_6(n\tau_1)$, $R_8(n\tau_1)$, $R_{10}(n\tau_1)$, $R_{12}(n\tau_1)$, $R_2(n\tau_2)$, \dots , $R_6(n\tau_2)$, $R_2(n\tau_3)$, $R_3(n\tau_3)$, and $R_2(n\tau_4)$. The results give $D = 4.72 \times 10^{-5}$ cm²/s at 15.8 MHz and $D = 5.16 \times 10^{-5}$ cm²/s at 14.2 MHz within an error of about 2%.

In Fig. 5b some ratio functions at a Larmor frequency of 15.8 MHz on water are reported. The 2τ values utilized in this case were as those employed for acetone. The results of the fit give $D = 2.16 \times 10^{-5}$ cm²/s at 15.8 MHz and $D = 2.36 \times 10^{-5}$ cm²/s at 14.2 MHz. The errors are as in acetone.

In Fig. 5c some ratio functions at 14.2 MHz on ethanol are reported. The 2τ values utilized in this measurement, as well as the ratio functions, were as in acetone. The results give $D = 0.98 \times 10^{-5}$ cm²/s at 15.8 MHz and $D = 1.08 \times 10^{-5}$ cm²/s at 14.2 MHz. In Fig. 5d some ratio functions at a Larmor frequency of 15.8 MHz on formamide are reported. The 2τ values utilized in this measurement, as well as the ratio func-

tions, were as in acetone. The results give $D = 0.52 \times 10^{-5}$ cm²/s at 15.8 MHz and $D = 0.59 \times 10^{-5}$ cm²/s at 14.2 MHz. Also for this sample the errors are as in acetone.

In Fig. 6 some ratio functions at a Larmor frequency of 14.2 MHz on glycerol are reported. The 2τ values utilized in this measurement were $2\tau_1 = 1$, $2\tau_2 = 2$, $2\tau_3 = 3$, $2\tau_4 = 4$, $2\tau_5 = 5$, $2\tau_6 = 6$, $2\tau_7 = 7$, $2\tau_8 = 8$, $2\tau_9 = 9$, and $2\tau_{10} = 10$ ms. The ratio functions, which can be realized with these choice of τ_i , are $R_2(n\tau_1)$, $R_4(n\tau_1)$, $R_6(n\tau_1)$, $R_8(n\tau_1)$, $R_{10}(n\tau_1)$, $R_2(n\tau_2)$, \dots , $R_5(n\tau_2)$, and $R_2(n\tau_3)$. The results give $D = 0.0027 \times 10^{-5}$ cm²/s at 15.8 MHz and $D = 0.0019 \times 10^{-5}$ cm²/s at 14.2 MHz. Also for this sample the errors are as above.

The method presented in this paper allows evaluating the self-diffusion coefficient and to improve the determination of the transverse relaxation in magnetic field with strong and not modifiable inhomogeneity. Besides to be utilized with device like SHIP, the method can be used also on the fringe field of superconducting magnet and, potentially, also in high-resolution spectrometers exploiting the field generated by the shimming coils. Established the effectiveness of the method, the setting of the field inhomogeneity terms can, of course, easily be made utilizing a liquid with known D .

The study of porous media, particularly for what concerns the materials of Cultural Heritage, could acquire advantages by the use of our approach. It is well established, in fact, that by Laplace transform of the relaxation distribution it is possible to get the pore size distribution in many porous system [19,20]. By the ratio function method, it becomes possible to eliminate the self-diffusion effect on transverse relaxation of liquid filling pores, not only when the polarization field is

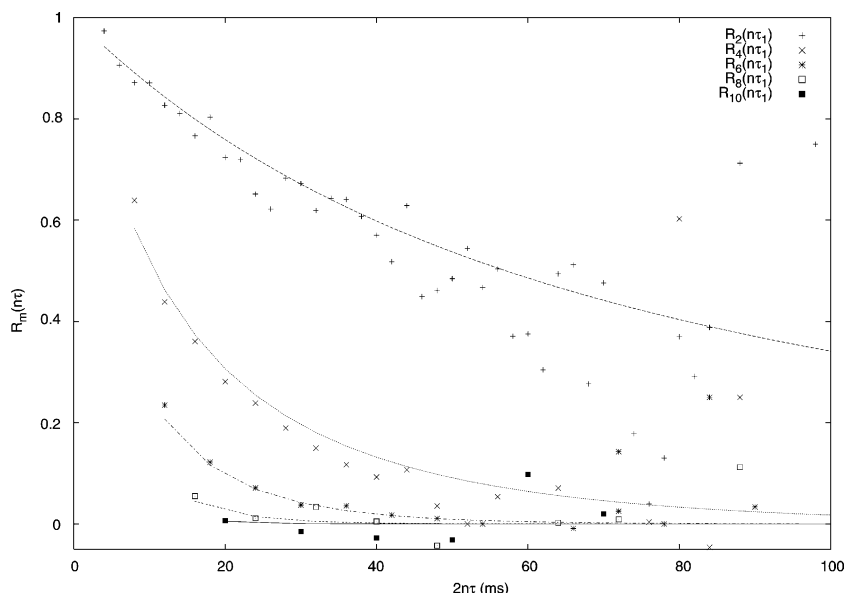


Fig. 6. The $R_2(n\tau_1)$, $R_4(n\tau_1)$, $R_6(n\tau_1)$, $R_8(n\tau_1)$, $R_{10}(n\tau_1)$ functions for glycerol are reported with $2\tau_1 = 1$ ms. The fits made by Eq. (4) are also reported.

inhomogeneous, as for SHIP measurements, but also when the inhomogeneity is produced by internal structure to the sample, as for the susceptibility effect, although in this case the presence of multiexponential decay [21,22] will make the algebra of the correction procedure more complicated. A work which takes in account the susceptibility effect in porous systems is currently in progress.

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