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Selective NMR excitation in strongly inhomogeneous magnetic fields

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Abstract

The NMR-MOUSE is a unilateral and mobile NMR sensor which operates with highly inhomogeneous magnetic fields. To produce a mobile NMR unit, RF excitation is sought, which can be produced with the most simple equipment, in particular nonlinear, low-power amplifiers, and to observe a free induction decay in strongly inhomogeneous fields, the excitation needs to be selective. The possibility to produce selective excitation by sequences of hard low-power radiofrequency pulses in the strongly inhomogeneous magnetic fields of the NMR-MOUSE is explored. The use of the DANTE sequence for selection of magnetization from parts of the sensitive volume was investigated for longitudinal and transverse magnetization by computer simulations and experiments. The spectra of the recorded FIDs and echo signals are in good agreement with those simulated for the excitation, which verifies the concept of the DANTE excitation. The results obtained are an important step towards a low-power operation of the NMR-MOUSE to improve its mobility.

Keywords: Selective excitation; DANTE pulse sequence; Strongly inhomogeneous magnetic fields; NMR-MOUSE

1. Introduction

Methods to improve the spatial resolution of NMR imaging or the selectivity in high resolution NMR spectroscopy are topics of continuing interest in the last decades. Selective excitation is one of the directions in this endeavor that provides new applications of NMR, e.g., in biomolecular studies and materials investigations [1–3]. Many techniques for suppression of water and other resonance signals, decoupling techniques, selective excitation of a single resonance or multiplet within a spectrum in high resolution NMR spectroscopy, and slice selection in magnetic resonance imaging are based on selective excitation [4–6].

The most simple way to generate selective excitation is to extend the duration of a rectangular radiofrequency (RF) pulse, for which the frequency spectrum is the sinc function. The selectivity is poor because only a narrow part of the main excitation region is sufficiently flat to produce a uniform perturbation of the selected spectral region, and the response spectrum contains undesired signal contributions from the frequency sidebands of the excitation pulse. Improving the selectivity of the RF excitation is possible by the use of pulse trains, for example the DANTE sequence [7], colored noise excitation [8], and by the use of shaped pulses like the Gauss, Digger, sinc, or other pulses [3], for which the overall frequency sidebands of the spectrum are more reduced compared to those of rectangular pulses [9,10]. Previously, experiments in this direction were performed using standard NMR spectrometers, with homogeneous magnetic field B_0 or linear magnetic gradient fields [10,11].

In most pulse sequences of NMR spectroscopy and imaging only one or a few RF pulses are used for excitation, which requires relatively high RF power. Reducing the RF power is possible by spreading the pulse energy over a large time [12]. Undesired consequences in this case are the modification of the excitation spectrum and possibly the undesired apparition of correlations between the nuclear spins during the pulse. Technical problems arise also from the stability of the RF electronics during long shaped pulses and distortions of the

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RF signal at high amplitude, caused by nonlinear operation of the transmitter. The use of such pulses requires the availability of reliable equipment to provide strong and reproducibly shaped RF pulses. Such hardware is standard on today's high performance NMR spectrometers and imaging systems.

New applications of NMR in industry, geophysics, and environmental studies, request the use of mobile and often simple equipment, which may not provide the features requested for the production of shaped high-power pluses, and bulky high-voltage power supplies and capacitors are an obstacle to the mobility of the instrument. For reasons of mobility and nondestructiveness the NMR-MOUSE (Mobile Universal Surface Explorer) has been developed [13–17]. Here, the static magnetic field $\boldsymbol{B}_0$ is generally produced by one or more small permanent magnets with a surface induction less than 1 T and a strong magnetic field gradient on the order of 10–20 T/m [18,19]. Previous work has analyzed the effect of conventional hard pulses on the behavior of the nuclear spins in the strongly inhomogeneous magnetic fields of the NMR-MOUSE and demonstrated the possibility to measure parameters of nuclear relaxation like T_1, T_2, $T_1\rho$, and mass transport like the diffusion coefficient [20–22].

The goal of this paper is to investigate the possibility to reduce the peak RF excitation power to a minimum, for use with mobile NMR sensors, and to demonstrate the possibility to obtain a spin–echo in strongly inhomogeneous magnetic fields at low voltage. To this end the standard short and strong RF pulses are replaced by sequences of short small flip-angle pulses having an effect on the nuclear spins similar to hard high-power pulses. Alternatively, adiabatic pulses [23] can be explored, because they are selective, insensitive to miscalibration, and can be generated at arbitrarily low power. But their generation requires more sophisticated hardware, and the goal in mobile NMR at this stage is to employ the most simple instrumentation possible. This means, that pulse shaping should be avoided as well as high-peak power which demands high-voltage capacitors and a suitable power supply.

Restricting the attention to the linear response [24] for the sake of simplicity, our approach is the following: The response spectrum $Y(\omega)$ is the complex product of the Fourier conjugate $X^*(\omega)$ of the excitation spectrum $X(\omega)$ with the distribution $S(\omega)$ of frequencies ω of the system under investigation $Y(\omega) = S(\omega) \times X^*(\omega)$. $S(\omega)$ is broad in unilateral NMR because of the spread of frequencies induced by the field gradient and due to the fact that the object is larger than the sensor. In this case, $S(\omega)$ may be considered to be independent of (ω), $S(\omega) = S$. Any RF pulse applied, therefore is selective and the response spectrum is proportional to the excitation spectrum, $Y(\omega) \propto X(\omega)$, and following the convolution theorem, no FID can be observed in the time domain. Our final goal is to introduce a narrow structure into the response spectrum $Y(\omega)$ of a short pulse by making S dependant on frequency within the sensitive volume of the NMR-MOUSE, i.e., within a frequency window narrow compared to that of a short pulse, so an FID can be observed following selective excitation of parts of the sensitive volume. The work reported below is an important step towards reaching this goal.

In principle a single long pulse or a shaped pulse would be suitable to achieve this goal. But given that the FID should have maximum amplitude and duration and be generated with simple low-voltage hardware, we opted for the DANTE pulse train (Delays Alternating with Nutations for Tailored Excitation) first proposed by Morris and Freeman [7]. It consists of several short, small flip-angle pulses. Each pulse excites the entire sensitive volume, but the succession of pulses leads to destructive interference in the excitation spectrum of the pulse train, so that a magnetization grating is excited within the sensitive volume. We used this sequence for selective excitation with the NMR-MOUSE. Different RF pulse sequences are proposed, experimentally tested, and their selectivity is analyzed in terms of the response spectrum $Y(\omega)$. The frequency characteristics of the RF pulse train were carefully adjusted to obtain the desired excitation spectrum. The first tests were made on elastomers.

2. Experimental

The NMR-MOUSE used was a bar shaped permanent magnet with the RF coil positioned on the top of the magnet [19]. The device was operated at a resonance frequency of 19.2 MHz corresponding to the surface frequency of the NMR-MOUSE. Here, the volume averaged-field gradient was approximately 20 T/m. The NMR experiments were controlled by a Bruker Minispec PC spectrometer for excitation and data acquisition. The transmitter and receiver system are characterized by a bandwidth of 0.5 MHz. We used a sample of natural rubber with the size of 2 cm × 2 cm and a thickness of 2 mm for testing. Typically a recycling delay of 0.5 s was used, and 100 scans were averaged for improvement of the signal-to-noise ratio.

3. Results and discussion

The implementation of the DANTE pulse sequence involved several steps: adjustment of the sequence parameters, evaluation of the excitation spectrum, evaluation of the RF characteristics of the transmitter and receiver system, and evaluation of methods to explore the selective excitation effect.

3.1. Design of the pulse sequence

The DANTE pulse sequence consists on n equidistant RF pulses, each pulse with the same amplitude B_1 and length δ, repeated with a period Δ (Fig. 1a). The length and the amplitude of the component pulses are chosen so that each pulse produces a rotation of the magnetization in the rotating frame with an angle $\theta \ll 90°$, but the net flip angle of the entire sequence corresponds 90°, i.e., $\theta = \gamma B_1 \delta$ and $n\theta = 90°$ [7].

The excitation spectrum $X(\omega)$ is given by the Fourier transform of the pulse sequence drawn in Fig. 1a. It consists of a comb of sidebands spaced by Δ^{-1}, each spike in the comb having the form of a sinc function of width $(n\Delta)^{-1}$ (Fig. 1b). The characteristics of the spectrum depend on the values of the parameters δ and Δ. The length δ determines the total comb width and the pulse spacing Δ determines the distance between the spikes [9]. The total number of pulses used in a DANTE sequence determines its selectivity. By modifying δ, Δ, and n the excitation spectrum is adjusted. The amplitude B_1 of the RF pulses can be reduced by increasing the length δ of each pulse or increasing the total number n of the pulses. In this way, it is possible to reduce the excitation power. However, the delay time Δ and the total duration of the sequence must have values less than the relaxation times T_2 and T_1 in order to minimize the effects of relaxation during the sequence. Usually, the parameters δ and Δ are of the order of few microseconds and the total number of pulses is in the range 50–100. Pulse widths δ less than 1 μs could not be used because the limitations in the available transmitter to provide accurate pulses.

We used the minimum pulse length $\delta = 1$ μs possible to generate on the spectrometer. Each sequence consisted of $n = 50$ pulses with a flip angle of $\theta = 1.8°$. The transmitter output was attenuated by 20 dB. Many sequences were tested with pulse periods varying from 3 to 11 μs. For each sequence, the excitation spectrum $X(\omega)$ was computed and compared with the experimental response spectrum $Y(\omega)$.

3.2. Spectral domains

When setting the DANTE sequence parameters, different points need to be accounted for: (1) the total bandwidth to be excited; (2) the bandwidths of the RF transmitter and receiver circuits of the spectrometer; and (3) the spectral bandwidths of the probing $\pi/2$ and π pulses of an echo detection pulse, possibly following the DANTE sequence. The excitation frequency ω_{RF} of DANTE sequence is considered to be on resonance in the center of the sensitive volume. The position of the selective volume can be shifted by changing ω_{RF}. Suppose that the excitation bandwidth of interest is a region of width SW_{RF} centered at frequency ω_{RF}, which is schematically shown in Fig. 1b. The spectrum of the DANTE sequence consists of many narrow excitation bands of width $\delta\omega_{\mathrm{RF}}$, centered on frequencies $\omega_{i,\mathrm{RF}}$ and separated by the interval $\Delta\omega_{\mathrm{RF}}$. We select the parameters δ and Δ of the DANTE sequence so that some narrow excitation bands are included in the sensitive volume of the sample (Fig. 1b).

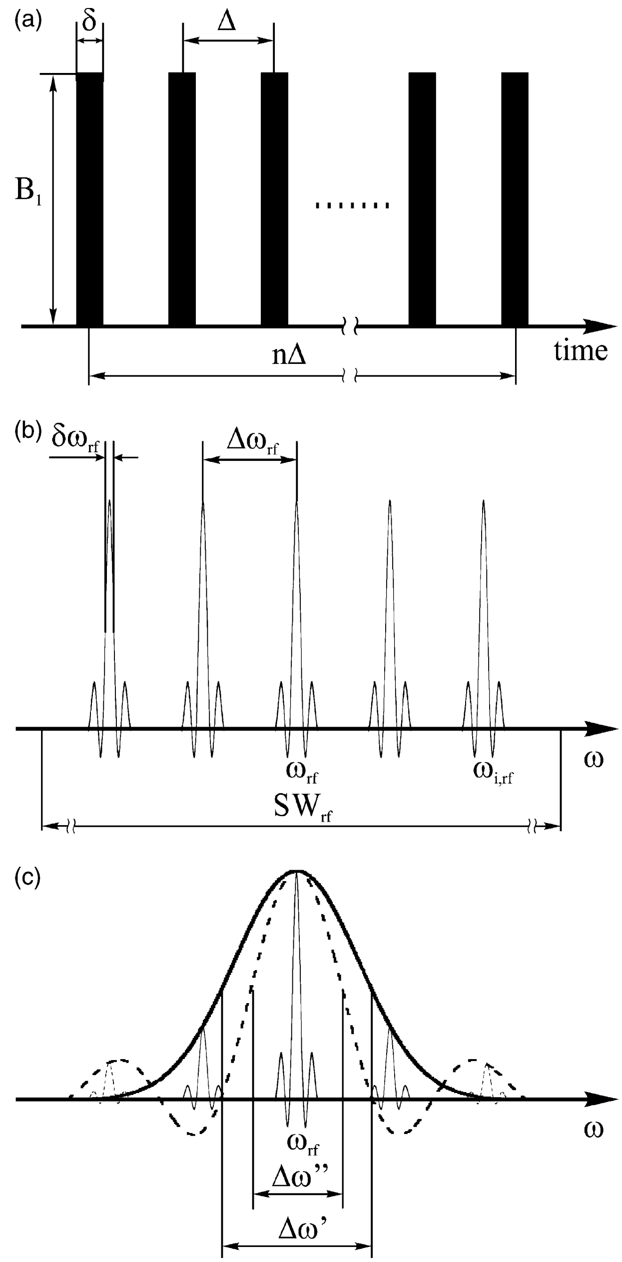

Fig. 1. (a) Schematic representation of the DANTE pulse sequence. (b) Excitation spectrum obtained by Fourier transformation of the DANTE sequence. The spectrum consists of many excitation bands of width $\delta\omega_{\mathrm{RF}}$, centered at frequencies $\omega_{i,\mathrm{RF}}$ and separated by the frequency intervals $\Delta\omega_{\mathrm{RF}}$. SW_{RF} is the spectral width of the excitation. (c) The effective excitation. The solid line is the transfer function of the spectrometer; the dashed line represents the spectrum of the probing 90° and 180° pulses. The effective excitation is obtained by multiplication of the DANTE spectrum with the transfer function and possibly the spectra of the probe pulses.

The RF transmitter and receiver circuits of the spectrometer are tuned to the central frequency ω_{RF}. The transfer function of the transmitter and receiver circuits is a Lorentz function with the bandwidth $\Delta\omega' = 0.5$ MHz which is determined only by the electronic components of the spectrometer (Fig. 1c). The DANTE sequence produces excitation bands at many frequencies $\omega_{i,RF}$ but only those within the bandwidth of the RF circuits are effective. The effective excitation spectrum is the product of the DANTE excitation spectrum and spectrometer transfer function.

When the effect of the DANTE sequence is probed by a single high-power 90° pulse (FID) or 180° pulse (echo), the excitation spectra of the probe pulses has to be considered as well. For a rectangular probe pulse, the excitation spectrum is the sinc function (Fig. 1c). The probe pulse produces a continuous excitation spectrum with a width $\Delta\omega''$ proportional to the inverse pulse duration t_p. It is necessary that $1/t_p$ is larger than the width $\delta\omega_{RF}$ one narrow DANTE excitation band. Only the frequencies bands of the DANTE spectrum contained within the excitation spectra of the probe pulses will give a contribution to the detected signal. The probe pulse should be as short as possible for a wide excitation spectrum. We used rectangular 90° probe pulses with a length $t_p = 2.5\,\mu$s corresponding to an excitation bandwidth of 0.4 MHz. This bandwidth is sufficient to probe the excitation slices selected by DANTE sequence, and it is comparable to the width of the spectrometer transfer function.

3.3. Test of the selectivity of the DANTE pulse sequence

When arguing with a homogeneous B_1 field the effect of the DANTE pulse sequence is equivalent to a 90° pulse on the spins with resonance frequencies within the excitation bands. The longitudinal magnetization of these spins is flipped by 90° into the transverse plane. Only these spins can give rise to a FID or an echo. Other spins remain unaffected and give not contribution to the resonance signal. There are different possibilities to test the effect of the sequence.

3.3.1. DANTE FID

Following the DANTE sequence, the transverse magnetization contains only contributions of nuclear spins at Larmor frequencies $\omega_{i,RF}$. The Fourier transform $Y(\omega)$ of the FID following DANTE excitation will show signal only in the vicinity of the excitation frequencies $\omega_{i,RF}$. We recorded the FID for many DANTE sequences with different values of the period Δ. The spectra of the recorded FIDs were compared with the spectra simulated for the sequence. Although the response spectrum is noisy, the response signal arises at the correct frequencies within the frequency range of the receiver (Fig. 2).

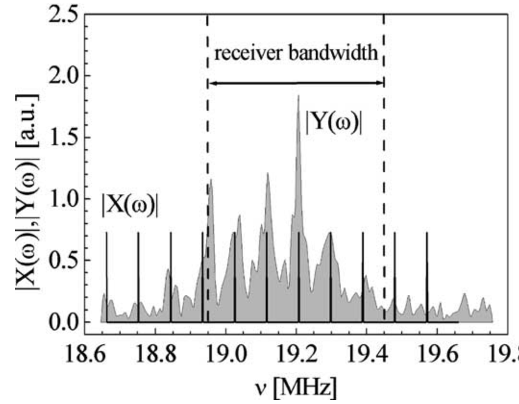

Fig. 2. The spectrum $|Y(\omega)|$ of the recorded FID, and the calculated spectrum $|X(\omega)|$ of DANTE sequence. The sequence contains $n = 50$ RF pulses of length $\delta = 1\,\mu$s repeated with a period of $\Delta = 11\,\mu$s. The number of scans was 100. The broken lines indicate the receiver bandwidth.

3.3.2. DANTE echo

Due to the strong gradient of the NMR-MOUSE, the decay of the FID is fast and the recorded FID is short. To shift the detectable signal further beyond the receiver dead time an echo was generated by a 180° pulse, $(\text{DANTE})_x$–τ–$(\pi)_y$–τ–echo (Fig. 3a). In this DANTE echo pulse sequence, τ is about half of the echo time.

We recorded DANTE echoes for different values of Δ. Like the DANTE-FID the spectrum of the DANTE echo contains the excitation frequencies $\omega_{i,RF}$ of the excitation sequence. The agreement between the spectrum of the recorded echo and the calculated DANTE excitation sidebands is good (Fig. 3b). Fig. 3c shows a similar echo spectrum for a smaller value of Δ. As expected, the response sidebands are further apart than those in Fig. 3b.

A supplementary test for verifying the selectivity of the DANTE excitation was conducted by comparing the spectrum of the DANTE echo with that of standard Hahn echo [25]. The Hahn echo was recorded with the same sequence parameters except for replacing the DANTE sequence by a 90° pulse, $(\pi/2)_x$–τ–$(\pi)_y$–τ–echo (Fig. 4a). The excitation spectrum of the Hahn echo is given by the square of the sinc function multiplied by the spectrometer transfer function. The spectrum of the echo is broad and without structure. The clear difference between the spectra of the Hahn and the DANTE echoes is observed from Fig. 4b. The narrow response bands generated by the DANTE excitation are largely included in the response spectrum of the Hahn echo. This clearly demonstrates the selectivity of DANTE pulse sequence produced within the sensitive volume of the NMR-MOUSE. Consistent results are obtained when changing Δ in the DANTE excitation (Fig. 4c).

3.3.3. DANTE selection of longitudinal magnetization

The DANTE sequence can also be employed for signal suppression of some spectral regions and exploration of

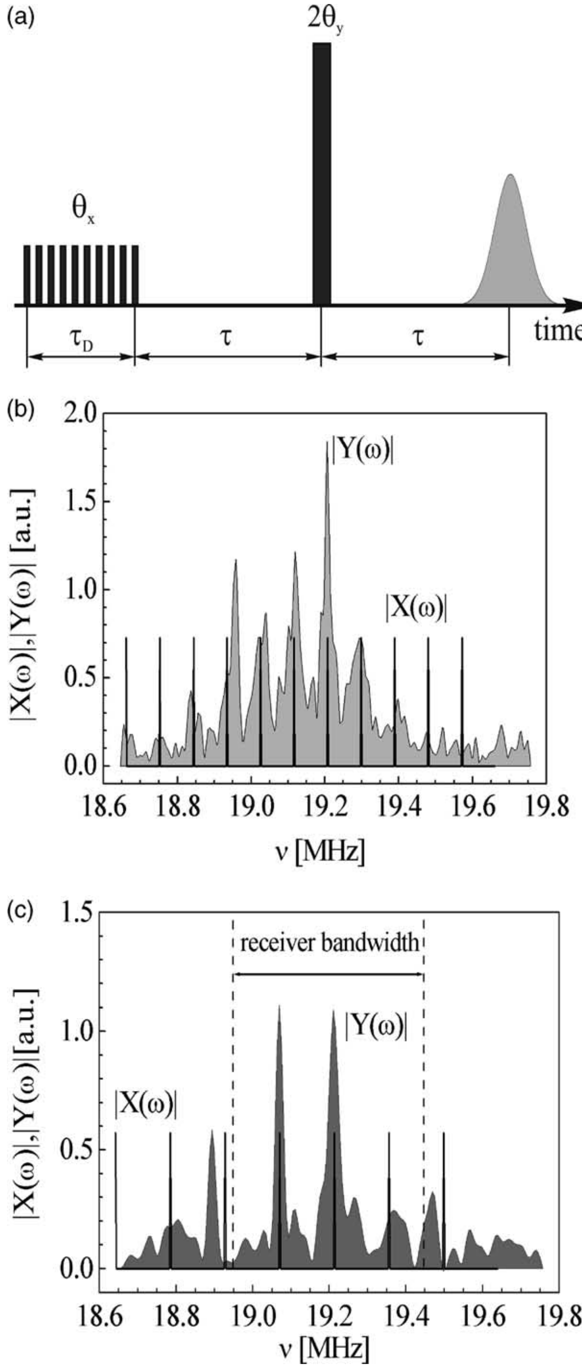

Fig. 3. (a) Schematic representation of the DANTE echo pulse sequence. (b) The response spectrum $|Y(\omega)|$ and the calculated excitation spectrum $|X(\omega)|$. The sequence contains $n = 50$ RF pulses of length $\delta = 1\,\mu s$ repeated with a period of $\Delta = 11\,\mu s$. (c) Modification of the spectrum of the recorded echo induced by decreasing the pulse period Δ of the DANTE sequence. The sequence contains $n = 50$ RF pulses of length $\delta = 1\,\mu s$ repeated with a period of $\Delta = 7\,\mu s$.

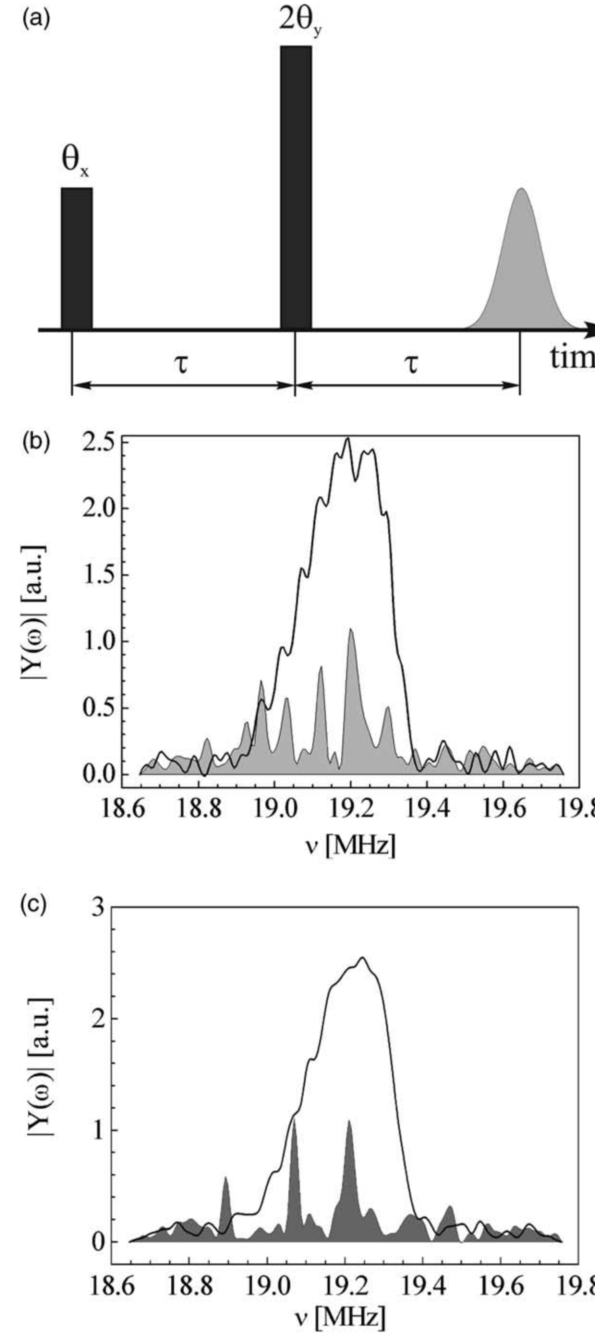

Fig. 4. (a) Schematic representation of Hahn echo sequence. (b) Comparison of response spectra for the Hahn echo (solid line) and the DANTE echo (shaded area). The DANTE sequence contains $n = 50$ RF pulses of length $\delta = 1\,\mu s$ repeated with a period of $\Delta = 11\,\mu s$. (c) Same as (b) but for $\Delta = 7\,\mu s$.

the remaining longitudinal magnetization. We call this approach *selection of longitudinal magnetization* to stress the point, that the magnetization state prepared in this way exists on the time scale of T_1, which in soft matter is often much longer than that of T_2 and can subsequently be interrogated by generating transverse magnetization at any time shorter than T_1. The pulse sequence is: $(\mathrm{DANTE})_x$–$(\pi/2)_x$–τ–$(\pi)_y$–τ–echo (Fig. 5a). The longitudinal magnetization corresponding to the excitation bands of the DANTE sequence is converted into transverse magnetization, the longitudinal magnetization corresponding to the gaps in the excitation spectrum is left as such. The 90° pulse applied immediately after the

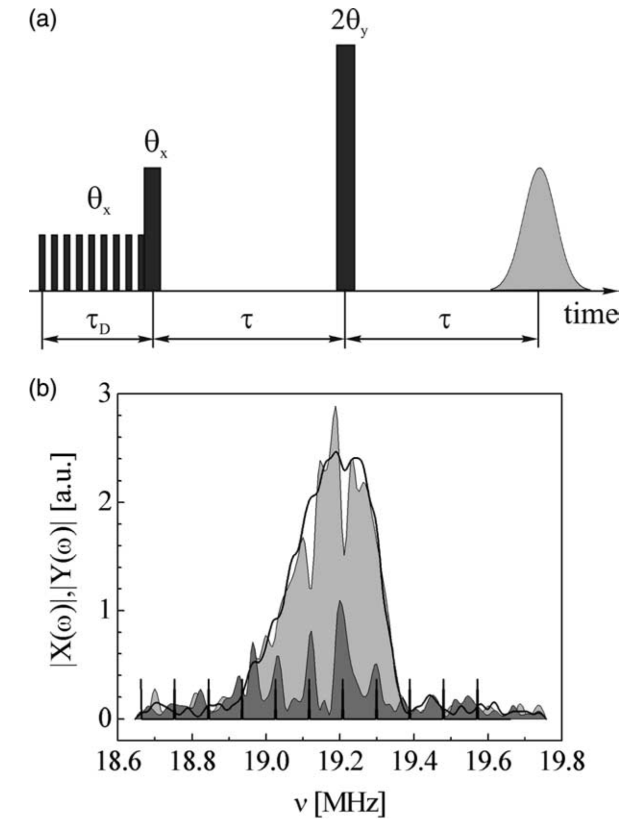

Fig. 5. (a) Schematic representation of the pulse sequence for DANTE selection of longitudinal magnetization. (b) Comparison of response spectra for the Hahn echo (solid line), DANTE selection of longitudinal magnetization (shaded area) and the spectrum for DANTE echo (black area). The solid line represents the calculated DANTE excitation spectrum for $n = 50$ RF pulses of length $\delta = 1\,\mu s$ repeated with a period of $\Delta = 11\,\mu s$.

DANTE sequence, rotates the entire distribution of magnetization by 90° around the x-axis. As the result the former longitudinal magnetization components now process in the transverse plane and can be refocused into an echo for detection. The spectrum of this echo contains all the frequencies that were not contained in the DANTE excitation spectrum (Fig. 5b). If we compare the spectrum of the DANTE echo and the complementary spectrum we observe that the regions excited in the first one correspond to the ones suppressed in the second one. The suppressed regions in the complementary DANTE spectrum correspond to the excitation bands of the simulated DANTE spectrum (Fig. 5b). This demonstrates the effect of selection of longitudinal magnetization and the associated signal suppression in the transverse magnetization by the DANTE sequence.

3.3.4. DANTE–DANTE echo

Apart from the DANTE-FID, the pulse sequence used to test the excitation effect of the DANTE sequence contains high-power pulses with flip angles 90° or 180°. Our interest is to reduce the peak RF power required for signal generation with the NMR-MOUSE to a minimum. In addition to monitoring the FID, the refocusing pulse of the DANTE echo may also be replaced by a DANTE sequence. The resultant sequence is called the DANTE–DANTE echo (Fig. 6a). The parameters δ and Δ of both DANTE sequences are the same, except that the second sequence has $2n$ pulses. A better, but more difficult solution could be to keep the number of DANTE pulses equal in both sequences and to double the pulse amplitude in the refocusing sequence. The first DANTE sequence achieves selective excitation, and the second one achieves selective refocusing of the transverse magnetization, at the same frequencies as the initial excitation. The refocusing DANTE sequence functions as a supplementary frequency filter and increases the selectivity of the entire pulse sequence. This effect can be clearly seen from the Fig. 6b. Despite low signal-to-noise ratio, the spectrum of DANTE–DANTE echo can be seen to be consistent with the excitation spectrum. This result demonstrates the possibility of replacing all the hard pulses of an echo sequence by DANTE pulse sequences.

In all our experiments, the amplitude of the individual DANTE pulses is greatly reduced in comparison to those of the non-selective 90° and 180° pulses. The attenuation of the DANTE pulses is 20 dB with reference to the maximum amplitude possible on the spectrometer. The standard rectangular 90° and 180° pulses have the maximum amplitudes with zero attenuation, but durations of 2.5 and 5 μs. Compared to rectangular pulses of length 1 μs and maximum amplitude, the B_1 field strength

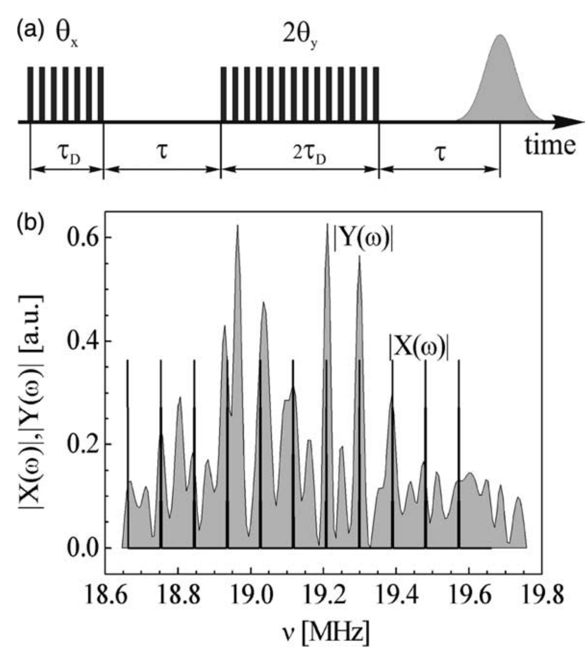

Fig. 6. (a) Schematic representation of the DANTE–DANTE echo pulse sequence. (b) Comparison of spectra for the DANTE–DANTE echo (shaded area) and the spectrum of the excitation (solid line). The sequence contains $n = 50$ RF pulses of length $\delta = 1\,\mu s$ repeated with a period of $\Delta = 11\,\mu s$.

of the DANTE pulses is 50 times less, corresponding to a saving in peak RF power by a factor of 2500.

4. Conclusions

Our results demonstrate the possibility to produce selective excitation with the DANTE pulse sequences in the strongly inhomogeneous magnetic fields of the NMR-MOUSE. The excitation is produced in equally spaced narrow frequency regions of the sensitive volume of the NMR-MOUSE and can be adjusted with the parameters of the sequence. The FID following the DANTE excitation can be observed even in the strongly inhomogeneous field of the NMR-MOUSE. The selectivity of the excitation was tested by the DANTE echo sequence, which denotes the detection of the DANTE FID via a Hahn echo generated with a non-selective 180° pulse. The spectrum of the DANTE echo contains signal only at the frequencies excited by the pulse sequence. The experimental and the simulated spectra are in good agreement.

The spectra of the DANTE echo and the excitation spectrum were compared to the spectrum of Hahn echo recorded under similar conditions. The spectrum of the Hahn echo contains all the resonance frequencies of the sensitive volume, but filtered by the spectrometer transfer function. The difference between these spectra clearly demonstrates the excitation selectivity of the DANTE pulse sequence.

The DANTE sequence has also been shown to be useful for selective elimination of longitudinal magnetization. The spectrum generated by a $90°–\tau–180°–\tau$–echo sequence detected following DANTE excitation contains signal from frequencies complementary to those of the DANTE sequence. The frequencies of the suppressed signal bands correspond to the excitation bands of the DANTE sequence.

Finally the refocusing pulse of the DANTE echo sequence can be replaced by another DANTE sequence, resulting in the DANTE–DANTE echo sequence. The use of two DANTE sequences generates echoes, which can be employed to measure transverse relaxation with relaxation times longer than the DANTE sequence. The advantage of using the DANTE FID and DANTE–DANTE echo with the NMR-MOUSE is a considerable savings in peak excitation power. This is an important step towards the development of small, mobile, and simple NMR instruments, which can be operated by batteries. To reach this goal the signal-to-noise ratio must be improved by sequence optimization. The inherent shortcoming of this approach is the restriction of the signal bearing volume due to the selectivity of the excitation. Furthermore, the effect of the flip-angle distribution has been ignored in this study and needs to be investigated by numerical simulations.

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