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Communication

Stray field measurements of flow displacement distributions without pulsed field gradients

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Abstract

The probability distribution $P(\zeta)$ of diffusive and advective molecular displacements is determined using a fixed field gradient (FFG) pulse sequence, on fluid flow through a Bentheimer sandstone, in the grossly inhomogeneous stray field of a super-conducting magnet. Two decades of q-space are scanned with stimulated echoes, using the gradient of the stray field and variable encoding times δ. The strength of the gradient permits the use of short encoding times, which is desirable for limiting the distorting effects produced by flow displacements through susceptibility induced field inhomogeneities. CPMG and CP echo trains are used to refocus separately the real and imaginary parts of the stimulated echo, for experimental efficiency.

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Keywords: Stray field; Propagator; Flow; Rocks

1. Introduction

Flow in porous media is commonly studied using pulsed field gradient (PFG) NMR techniques [1–3], where pulsed external field gradients are applied to encode the displacement of spins during some evolution time Δ in the phase θ of their magnetization. The displacement distribution, also referred to as the volume averaged propagator, can be extracted from such data by Fourier transform. This paper presents an analogous fixed field gradient (FFG) method suitable for flow measurements in samples located outside of an NMR apparatus, say in the stray field of an unshielded super-conducting magnet, a well logging tool, or an NMR mouse [4], where a static field gradient of several mT/m can be present. The viability of the method discussed here is demonstrated with measurements of the volume averaged displacement probability distribution of molecular displacements in Stokes flow through a rock core.

NMR in stray fields has found many applications where the sample is located outside the NMR apparatus, for example in well logging [5], materials testing and quality control [6], stray field imaging [4], and stray field measurement of velocity distributions [7]. The CPMG-experiment $((\pi/2)_x–[\tau–\pi_y–\tau–]^n)$ is the prototypical sequence for stray field work because it offers advantages in the signal-to-noise ratio (SNR), and because it can be used to probe relaxation and diffusion properties of the sample. Its spin dynamics in grossly inhomogeneous fields are well understood [8,9], and it is used in logging applications for the measurement of T_2-relaxation time distributions. CPMG-like sequences [10] have been introduced in two-dimensional stray field experiments probing diffusion–relaxation (D–T_2) correlations and relaxation–relaxation (T_1–T_2) correlations, where 'editing' sequences sensitive to diffusion or T_1-relaxation

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replace the standard π/2 excitation pulse of the CPMG-sequence. More recently PFG-CPMG/CP stray field imaging experiments [4] have been developed, where the 'editing' sequence consists of the standard PFG spatial encoding, followed by CPMG and CP echo trains measuring real and imaginary parts of the NMR signal. In this work, we employ a pulse sequence belonging to the 'editing' family of stray field sequences, sharing salient features with the (D–T_2)-editing sequence and the stray field imaging sequences referred to above. Below we discuss our experimental setup, the pulse sequence, and the data acquired with it. Our experiments demonstrate how to measure volume averaged molecular displacement distributions efficiently and without pulsed field gradients, in the extremely inhomogeneous fields of a stray field NMR geometry. We present results obtained on water in Bentheimer rock core, with and without flow, and discuss some limitations of the technique, comparing data obtained in a stray field with the equivalent data obtained using standard PFG methods in a homogeneous field at 83 MHz.

2. Experimental

A cylindrical Bentheimer sandstone core was sealed on its circumference with epoxy, and fluid distributor caps were attached to the ends of the core and then connected to a flow system. Axial flow of water through the core was driven by a ISCO-1000D piston pump. Experiments were conducted in the absence of flow and for a flow rate of $\langle v\rangle = 0.5$ mm/s. The core was placed within a 1.765 MHz solenoid NMR probe, located in the fringe field of a super-conducting magnet, where the field $\|\mathbf{B}_0\| = 41.4$ mT and the gradient $\|\mathbf{g}\| = 13.8$ mT/m at the center of solenoid. The axes of solenoid, rock core, and gradient were co-linear, and hence the direction of flow and of the field gradient were co-linear as well.

The pulse sequence is shown in Fig. 1. It is based on the STE-CPMG diffusion editing sequence introduced in [10]. Three π/2 pulses produce a stimulated echo $S(q)$ at time $T_e = \Delta + 2\delta$. Signals associated with unwanted coherence pathways are eliminated by phase cycling

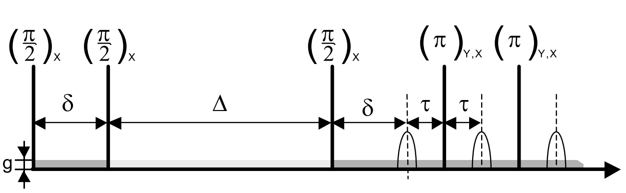

Fig. 1. A stimulated echo at $T_e = \Delta + 2\delta$ encodes displacement. With CPMG phase cycling the $(\pi)_y$ pulses re-focus the real part of the stimulated echo. With CP phase cycling $(\pi)_x$ pulses refocus the imaginary part of the stimulated echo. The fixed background gradient is indicated in dark grey, for intervals where magnetization is in the xy-plane.

[10] or were reduced below detection level by diffusion in the external gradient, or both.The ensemble average $S(q) = \langle e^{iq\zeta_j}\rangle$ is taken over all magnetized spins j displaced by ζ_j in the interval $\Delta + O(\delta)$ between the first and third π/2-pulse, and therefore it is recognized as the qth Fourier component of the displacement distribution, if the effects of relaxation and internal fields can be ignored [11]. $S(q)$ is measured, as in the classic PFG experiments [1–3], over a suitable range $\{q\}$ of q-space, where $q = \gamma\delta g$, γ is the proton gyromagnetic ratio, δ is the duration of the gradient encoding period, and g is the amplitude of the stray field gradient.

For experimental efficiency the signal is refocussed and measured with 2000 CPMG/CP spin echoes. It has been shown [8] that asymptotically in grossly inhomogeneous fields only a y-component of the initial magnetization is re-focussed by the π_y-pulses of the CPMG sequence, while the orthogonal x-component of the magnetization, and hence all phase information, is lost. Therefore, the complex stimulated echo signal $S(q)$ is measured with echo trains using CPMG and CP phase cycles, where for the CP phase cycle the phase of the refocussing π-pulse was rotated by 90° with respect to that of the CPMG echo train. A complex echo train $S'(q,t)$ is then obtained by adding the real part of the CPMG echo train to imaginary part of the CP echo train, for each choice of $q = \gamma\delta g$. The echo trains are summed to determine the stimulated echo $S(q)$. Measurements are performed for 30 logarithmically spaced values of δ between 20 μs and 3 ms, with $\Delta = 280$ ms, and the ensemble averaged displacement distribution $P(\zeta)$ is then determined by Fourier transform of the measured set $\{S(q)\}$.

Standard PFG experiments employing the 13-interval alternating pulsed field gradient sequence [3] (APGSTE) were performed to compare with our stray field results, using the same sample, flow rate, and evolution time Δ.

3. Results

Fig. 2 shows the real and imaginary parts of the magnetization decay $S'(q,t)$ acquired with CPMG and CP echo trains, in the presence and absence of flow, for three representative choices of q. The time of the first recorded echo defines $t = 0$ for these plots. We first focus on the stagnant case shown in the panels on the left. The imaginary part of the echo trains shown in Fig. 2C is zero because we have used the average phase of measurements performed in the absence of flow to define the single reference phase used for all measurements. The phase is independent of q, as expected for diffusive displacements. In Fig. 2A the signal for the largest choice of q is reduced below the noise level because the stimulated echo $S(q) \propto \exp\{-q^2 D(\Delta + \frac{2}{3}\delta)\} \ll 0.01$. Such diffusive attenuation is absent for the two smaller