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Background gradient suppression in pulsed gradient stimulated echo measurements

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Abstract

Pulsed gradient spin echo (PGSE) experiments can be used to measure the probability distribution of molecular displacements. In homogeneous samples this reports on the molecular diffusion coefficient, and in heterogeneous samples, such as porous media and biological tissue, such measurements provide information about the sample's morphology. In heterogeneous samples however background gradients are also present and prevent an accurate measurement of molecular displacements. The interference of time independent background gradients with the applied magnetic field gradients can be removed through the use of bipolar gradient pulses. However, when the background gradients are spatially non-uniform molecular diffusion introduces a temporal modulation of the background gradients. This defeats simple bipolar gradient suppression of background gradients in diffusion related measurements. Here we introduce a new method that requires the background gradients to be constant over coding intervals only. Since the coding intervals are typically at least an order of magnitude shorter than the storage time, this new method succeeds in suppressing cross-terms for a much wider range of heterogeneous samples.

Keywords: NMR; Diffusion; Heterogeneous media; Susceptibility fields; Background gradients

1. Introduction

The probability distribution function of molecular displacements is measured in a pulsed gradient spin echo (PGSE) experiment as a form of forced Rayleigh scattering [1]. The magnetic field gradient pulses encode a spatially varying phase grating into the spin magnetization and then molecular motion blurs this grating. A decoding gradient pulse can then measure the extent to which the original grating was preserved. By systematically varying the gradients, the wavenumber of the phase grating is changed and a series of reciprocal space measurements record the Fourier spectrum of the probability distribution of molecular displacement [2,3]. The accuracy of the measurement depends on having a linear magnetic field gradient so that the phase grating is accurately described by a single, well-characterized wavenumber $k(t)$, defined as

$$k(t) = \gamma \int_0^t g(t')\,\mathrm{d}t'. \tag{1}$$

The effects from background gradients are easily understood by following the complete wavenumber description [4] of the Pulsed Gradient STimulated Echo (PGSTE) experiment. As we will show, the path dependence of the echo signal on the wavenumber $k(t)$, places a set of constraints on the coding method.

The conditional displacement propagator $P(\vec{r} \mid \vec{r}', t)$ describes the probability for a spin initially at position $\vec{r}$ to be at position $\vec{r}'$ after time t. The NMR measurement is the Fourier Transform of an ensemble average of the initial spin density distribution and the probability propagator,

$$E(\vec{k}, t) = \int\int \rho(\vec{r}) P(\vec{r} \mid \vec{r}', t)\, \mathrm{e}^{-\mathrm{i}\vec{k}_\mathrm{e}\cdot\vec{r}}\, \mathrm{e}^{\mathrm{i}\vec{k}_\mathrm{d}\cdot\vec{r}'}\, \mathrm{d}r\, \mathrm{d}r', \tag{2}$$

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where $\vec{k}_e$ is the wavenumber of the encoding grating and $\vec{k}_d$ is the wavenumber of the decoding grating. In the most common case where $\vec{k}_e$ is equal to $\vec{k}_d$, it is customary to replace the variable $\vec{k}$ with $\vec{q}$ which is reciprocal to the displacement ($\vec{k}$ is reciprocal to the absolute position) and then Eq. (2) becomes

$$E(\vec{q},t) = \int\int \rho(\vec{r})P(\vec{r} \mid \vec{r}',t)\,\mathrm{e}^{\mathrm{i}\vec{q}\cdot(\vec{r}-\vec{r}')}\,\mathrm{d}r\,\mathrm{d}r'. \tag{3}$$

To understand the requirement for correctly characterizing $P(\vec{r} \mid \vec{r}',t)$ it is helpful to explore a few special cases. In the static case, the probability of displacement is a delta function,

$$P(\vec{r} \mid \vec{r}) = \delta(\vec{r}-\vec{r}') \quad \forall t \tag{4}$$

and

$$E(\vec{q},t) = \int\int \rho(\vec{r})\delta(\vec{r}-\vec{r}')\,\mathrm{e}^{\mathrm{i}\vec{q}(\vec{r}-\vec{r}')}\,\mathrm{d}r\,\mathrm{d}r' = \int \rho(\vec{r})\,\mathrm{d}r. \tag{5}$$

In this case, the spins do not change position during the experiment, and there is no q dependence in the echo. The case most commonly dealt with in PGSE experiments is a stochastic motion where the conditional displacement propagator is both independent of initial position,

$$P(\vec{r} \mid \vec{r}',t) = P(\vec{r}-\vec{r}',t) = P(\Delta\vec{r},t) \tag{6}$$

and is constant over time

$$P(\Delta\vec{r},2t) = P(\Delta\vec{r},t) \otimes P(\Delta\vec{r},t). \tag{7}$$

The conditional displacement propagator can then be split into a coherent and a diffusion component,

$$P(\Delta\vec{r},t) = P_{\text{coherent}}(\Delta\vec{r},t) \otimes P_{\text{diffusion}}(\Delta\vec{r},t). \tag{8}$$

The coherent part describes a uniform displacement and leads to a phase shift of the echo which is linear in q, $\mathrm{e}^{\mathrm{i}\vec{q}\cdot\vec{r}}$. The diffusion propagator is a solution to the Fick's equation. In the case of a sample of infinite spatial extent with no boundaries, the diffusion propagator is a Gaussian,

$$P(\Delta\vec{r},t) = \frac{1}{\sqrt{4\pi Dt}}\,\mathrm{e}^{-\Delta r^2/4\pi Dt} \tag{9}$$

and,

$$E(\vec{q},t) = \int\int \rho(\vec{r})\frac{1}{\sqrt{4\pi Dt}}\,\mathrm{e}^{-\Delta r^2/4\pi Dt}\mathrm{e}^{\mathrm{i}\vec{q}(\vec{r}-\vec{r}')}\,\mathrm{d}r\,\mathrm{d}r' = E(0,t)\mathrm{e}^{-D\int_0^t q^2(t')\,\mathrm{d}t'}. \tag{10}$$

The echo is attenuated at a rate proportional to the so-called b factor

$$b(t) = \int_0^t q^2(t')\,\mathrm{d}t', \tag{11}$$

which gives

$$\frac{E(\vec{q},t)}{E(0,t)} = \mathrm{e}^{-b(t)D}. \tag{12}$$

In the case of finite volumes, as the evolution time increases, the spins start to feel the existence of the diffusion boundaries. The boundary condition depends on the local geometry such as the pore size and tortuosity, and surface properties such as the permeability and relaxitivity. While we can treat the short time and long time limit case analytically, exact solutions to the intermediate time scale are in general not known due to the complex behavior in heterogeneous samples. But the diffusion propagator can still generally be described as

$$P(\vec{r} \mid \vec{r}',t) = \sum_{k=1}^{\infty} \psi_k(\vec{r})\psi_k(\vec{r}')\,\mathrm{e}^{-t/(T_k)} \tag{13}$$

so that

$$E(q,t) = \frac{1}{\upsilon}\sum_{k=1}^{\infty} \mid \tilde{\psi}(k) \mid^2 \ \mathrm{e}^{-\mathrm{i}\vec{q}\cdot\vec{r}}, \tag{14}$$

where $\psi(k)$ is the normalized eigenfunction of diffusion equation,

$$D\nabla^2\psi_k = -\frac{\psi_k}{T_k}, \tag{15}$$

$$D\hat{n}\cdot\nabla\psi_k = 0, \tag{16}$$

and $\{T_k\}$ are the eigenvalues of the diffusion equation determined by its boundary conditions [5]. The complete behavior is usually studied via a cumulant expansion. If the measurement time is sufficiently short that few molecules see a boundary, the cumulant analysis results in the familiar quadratic dependence on $\vec{q}$,

$$\log\frac{E(\vec{q},t)}{E(0,t)} = -\frac{q^2\langle(r(0)-r'(t))^2\rangle}{2} = -Dq^2t. \tag{17}$$

In the more general case of $t \gg l^2/2D$, eigenmodes higher than zeroth order can be neglected because of its much higher attenuation from the temporal terms. Here the q dependence includes contributions from terms higher than q^2. Mitra and Sen [6] have shown in particular the importance of the q^4 term for simple geometries.

In all the cases discussed above, the presence of background gradients will cause $q(t)$, and thus higher order terms of q, to be unknown. If the background gradient is spatially uniform and time-independent however, bipolar gradients can be combined with spin echo methods to refocus their effects [7,8], and thus remove the distortions to the grating.

The attenuation of the phase grating depends on the path of $q(t)$. If the spins are moving through a non-uniform distribution of background gradients, as in the case of porous media and biological tissue [9–13], refocusing the gradient in the coding intervals is not sufficient to completely remove the effects of the background gradients [14]. In this paper we analyze the influence of background gradients where the motion during the encoding time can be neglected compared to the storage time, and show that in this case the effects of non-uniform background gradients can be suppressed.

2. Method

In order to faithfully measure a diffusion constant both $q(t)$ and $b(t)$ must be correctly encoded. It is the presence of background gradients that prevent this. As shown in Fig. 1, in the stimulated echo measurement there are two relevant temporal scales, the coding time and the storage time. $q(t)$ is determined by the encoding, and it remains constant during the storage time. However $b(t)$ depends on the storage time, and in order to suppress the background gradient effects, it is necessary to remove this dependency.

To determine the effect of magnetic gradients on the echo attenuation, we calculate the value of $b(t)$ using Eq. (11). For a uniform background gradient g_b, and an applied gradient, g_a, the echo attenuation for the PGSTE sequence is [15]

$$\ln\frac{E(\vec{q})}{E(0)} = -\gamma^2 D\left[\delta^2(\Delta+\tau-\delta/3)g_a^2 + \delta\left(2\tau\Delta + 2\tau^2 - \frac{2\delta^2}{3} - \delta(\delta_1+\delta_2) - \left(\delta_1^2 + \delta_2^2\right)\right)g_a g_b + \tau^2\left(\Delta + \frac{2\tau}{3}\right)g_b^2\right], \quad (18)$$

where $b(t)$ depends both on the applied magnetic gradient g_a, and the background gradient g_b. The integral $\int_0^\tau q_{g_b}(t)\,dt \neq 0$, and it contributes to the integrand of $b(t)$ in the following intervals, including the storage time Δ. The cross-term $g_a g_b$ evolves through the whole pulse sequence, which introduces systematic errors in the PGSTE measurement.

Karlicek and Lowe [7] showed that the application of bipolar gradients strongly reduces the effect of the cross-term. Cotts et al. [8] and Latour et al. [16] introduced modified PGSTE sequences where pairs of bipolar gradients separated by π-pulses were applied in the coding intervals. The 13-interval pulse sequence suggested in [8]

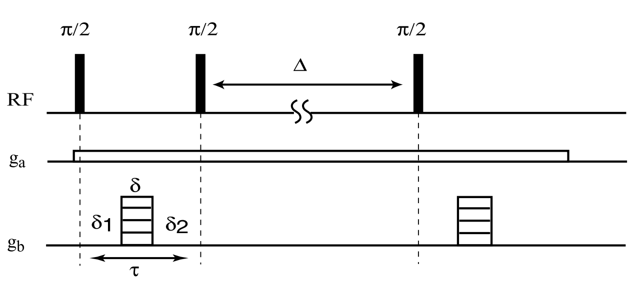

Fig. 1. Schematic diagram of the PGSTE pulse sequence [15]. Gradients of strength g_a are applied in the encoding and decoding intervals, which are separated by the storage time.

is shown in Fig. 2. The π-pulse inverts the sign of $q(t)$, so the value $\int_0^{2\tau} q_{g_b}(t)\,dt = 0$. The background gradient will therefore not contribute to terms in first order of $q(t)$. However, the value of $b(t)$ at the end of the encoding and decoding intervals still contains a τ-dependent cross-term. The condition for the suppression of this term is that $\int_0^{2\tau} q(t)\,dt - \int_{2\tau+\Delta}^{4\tau+\Delta} q(t)\,dt = 0$, denoted as condition I in [8]. This sequence is shown in scheme (a) of Fig. 2. The cross-terms during the two coding intervals cancel each other, provided that there is a spatially uniform background gradient. However, the encoding and decoding intervals are separated by the storage time, which can be hundreds of milliseconds. Thus in general spins may experience different background gradients in these two coding intervals. Assuming g_{b1} and g_{b2} are the values of the background gradient in the encoding and decoding intervals, respectively, the echo attenuation is given by

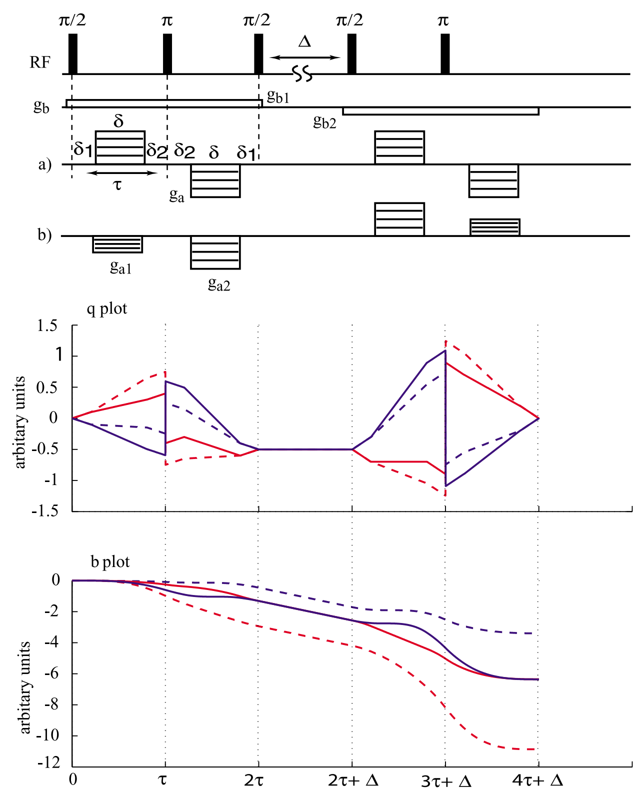

Fig. 2. Bipolar PGSTE pulse sequences. g_{b1} and g_{b2} denote the strength of the background gradients in the encoding and decoding intervals. Two schemes of applying gradients are shown. Scheme (a) with equal magnitude of the bipolar gradients is the 13 intervals sequence with Cotts' condition I [8]. Scheme (b) with asymmetric gradients is the new method suggested in the present work. Simulated q-path and b factor resulting from the two schemes of applying the gradients with different background gradients in the encoding and decoding intervals are shown. For the red lines a positive background gradient was applied in the encoding interval, followed by a negative background gradient of double size in the decoding interval, while for the blue lines the polarity of the background gradients was switched.

$$\ln\frac{E(\vec{q})}{E(0)} = -\gamma^2 D\Big[2g_a(g_{b1}-g_{b2})\delta\tau^2 - \frac{2}{3}(g_{b1}^2 + g_{b2}^2)\tau^3 - \frac{2}{3}g_a^2\delta^2(4\delta + 3(\Delta+\tau+\delta_1))\Big]. \quad (19)$$

Since the criteria that $\int_0^{2\tau} q_{g_b}(t)\,dt = 0$ is fulfilled, the Δ-dependent cross-term is still suppressed, but the τ-dependent cross-term remains. The same result was obtained in [14].

From the evaluations above we can conclude that in order to measure the true diffusion rate at storage times longer than the correlation time of the background gradient, the cross-term needs to be suppressed during the encoding and decoding intervals independently. The criteria for this is that the value of $b(t)$ at the end of these intervals should not depend on $g_a g_b$, implying that the cross-terms in the two halves of the coding intervals have to cancel each other. Assuming a constant background gradient during coding intervals such a cancellation can be achieved through an intensity modulation of the applied gradient. In order to derive this modulation, we introduce a parameter η, the ratio between the intensities of the two bipolar gradients, g_{a1} and g_{a2}, respectively. η is then determined by solving Eq. (11) under the criteria that the cross-term is zero. The diffusion attenuation at the end of the encoding interval is then given by

$$\ln\frac{E(\vec{q})}{E(0)} = -\frac{1}{3}\gamma^2 D\big[2g_{b1}^2\tau^3 + g_{a2}^2\delta^2\big(\eta^2(3\tau+\delta+3\delta_2) + 3\eta(\delta+2\delta_1) + (\delta+3\delta_1)\big) + g_{a2}g_{b1}\delta\big(\eta(5\delta^2+9\delta\delta_1+3\delta_1^2+12(\delta+\delta_1)\delta^2 + 6\delta_2^2) + (\delta^2+3\delta\delta_1+3\delta_1^2)\big)\big]. \quad (20)$$

Eq. (20) has one unique real solution for η which eliminates the τ-dependent cross-terms,

$$\eta = -\frac{\delta^2+3\delta\delta_1+3\delta_1^2}{5\delta^2+9\delta\delta_1+3\delta_1^2+12(\delta+\delta_1)\delta_2+6\delta_2^2}. \quad (21)$$

A negative coefficient η means that the two applied gradients have the same polarity. The value of η depends on the time constants δ, δ_1, and δ_2. With a proper choice of η, the evolution of the cross-term during τ is completely suppressed by the following evolution of this term from τ to 2τ. The same condition is also valid in the decoding interval. Because of the symmetry in q-space the two gradients applied in the decoding interval have the same ratio as in the encoding interval, but have opposite polarity. The total diffusion attenuation is now given by

$$\ln\frac{E(\vec{q})}{E(0)} = -\frac{1}{3}\gamma^2 D\Big[2(g_{b1}^2+g_{b2}^2)\tau^3 + g_{a1}^2\delta^2 \Big(3(1+\eta)^2(\Delta+2\delta_1) + \eta^2(3\tau+\delta+3\delta_2) + 3\eta(\delta+2\delta_1) + (\delta+3\delta_1)\Big)\Big]. \quad (22)$$

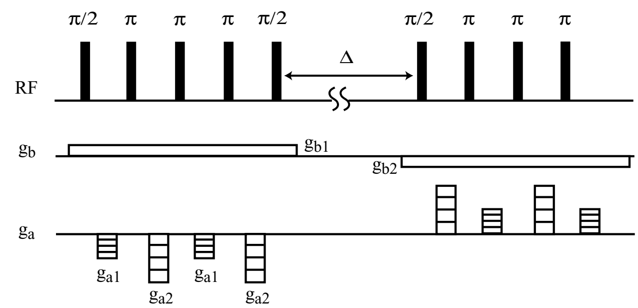

Fig. 3. An asymmetric bipolar PGSTE pulse sequence having four gradient pulses in the encoding and decoding intervals. The magnitude of the first and third gradients have max strength g_{a1}, and the second and fourth gradients have max strength g_{a2}.

This new gradient sequence is shown as scheme (b) in Fig. 2, where two negative gradients are applied in the encoding interval and two positive gradients are applied in the decoding interval.

The method presented above can be generalised to sequences having any number of π-pulses, as long as the encoding and decoding time is short compared to the correlation time of the spins moving through the susceptibility field. By adjusting the applied gradient strengths, full control of the cross-terms is possible. An example of a pulse sequence having three π-pulses in the coding intervals is shown in Fig. 3. The cross-terms are eliminated by adjusting the first and third applied gradients relative to the second and fourth gradients. The value of η which results in zero cross-term for this sequence is given by

$$\eta = -\frac{1}{3}\left(1 + \frac{4(\delta^2+3\delta\delta_1+3\delta_1^2)}{8\delta^2+15\delta\delta_1+6\delta_1^2+18(\delta+\delta_1)\delta_2+9\delta_2^2}\right). \quad (23)$$

3. Experimental

The experiments were carried out on a Bruker 14.1 T system using a homebuilt high resolution imaging probe with maximum gradient strength of 1200 G/cm. A 1.8 mm ID capillary tube containing distilled water doped with D_2O in a ratio of 1:3 was used in all of the experiments. The experiments were performed at a temperature of 18 °C, using $\delta = 3$ ms, $\delta_1 = \delta_2 = 1$ ms, $\Delta = 10$ ms, and with a maximum applied gradient strength of 16.3 G/cm.

The RF-coil of the homebuilt probe has a $\pi/2$ pulse length of 5 μs at a power of 12.5 W. The effect of a background gradient on the RF pulses is therefore negligible. In addition, an eight step phase cycling, as shown in Table 1, was applied in order to cancel the echoes formed by imperfect RF pulses and signals from

Table 1
Phase-cycling for the bipolar-PGSTE

No.	$\pi/2$	π	$\pi/2$	$\pi/2$	π	Rec.
1	$+x$	$+y$	$+x$	$+x$	$+y$	$+x$
2	$+x$	$+y$	$-x$	$+x$	$+y$	$-x$
3	$+x$	$+y$	$+x$	$-x$	$+y$	$-x$
4	$+x$	$+y$	$-x$	$-x$	$+y$	$+x$
5	$+x$	$+y$	$+y$	$+y$	$+y$	$+x$
6	$+x$	$+y$	$-y$	$+y$	$+y$	$-x$
7	$+x$	$+y$	$+y$	$-y$	$+y$	$-x$
8	$+x$	$+y$	$-y$	$-y$	$+y$	$+x$

unwanted q-paths. This phase cycling is a modification of the procedure presented in [17].

4. Results and discussion

The new method is designed to provide an undistorted measure of molecular diffusion in the presence of background gradients that vary slowly on the time scale of the encoding, but are free to vary over the storage time. This robust action is achieved by arranging that both $q(t)$ and $b(t)$ are correctly implemented at the start of the storage time regardless of the presence of background gradients. The pulse sequence in Fig. 2 achieves this with the asymmetric gradient pulse pairs calculated according to Eq. (21). This is shown in the q plots and b plots of Fig. 2. The value of $b(t)$ for the new pulse sequence at the end the encoding and decoding intervals are independent of a change of the strength of the background gradient during the storage time, and the cross-term is suppressed. This is not the case for the scheme (a), where the value of $b(t)$ at the end of the coding intervals strongly depends on the relative values of the background gradient during the encoding and decoding intervals.

To illustrate the difference between Cotts' and our solution of suppressing background gradients we ran a series of measurements where the "background" gradient was applied with a gradient coil. This extra gradient was kept static during the coding times, but varied from encoding to decoding. The experimentally obtained diffusion attenuations for water are shown in Fig. 4. The curves obtained using Cotts' sequence with condition I are roughly independent of the background gradients if they have the same strength. However, when the strength of the background gradient changes during the storage time, a clear deviation is observed. The diffusion attenuations obtained from the new method do not depend on the background gradient, provided it is constant over the coding intervals.

The apparent diffusion coefficients using different pulse sequences were estimated from the obtained attenuations. In the cases where a nonlinear behaviour of the attenuation was observed, the initial slope was taken

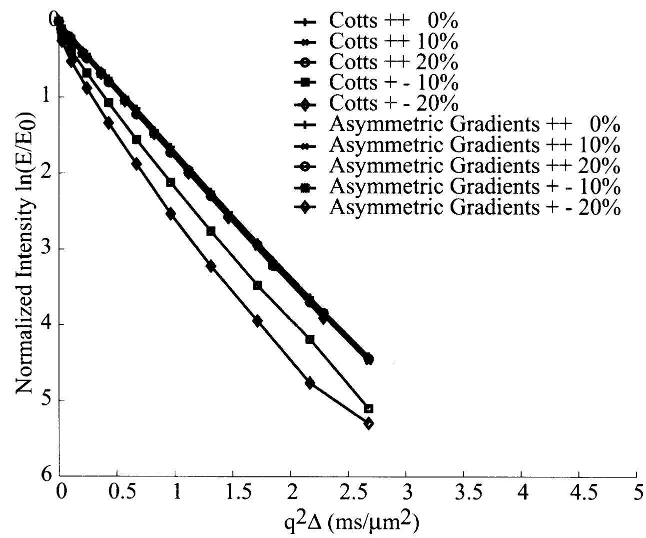

Fig. 4. Results from diffusion measurements in a sample of distilled water doped with D_2O. The measurements were performed using different pulse sequences, and with different values and polarities of the external background gradients in the encoding and decoding intervals. The strength of background gradients was varied between ±20% of the applied gradients.

as the value of the diffusion coefficient. The results are plotted in Fig. 5. The diffusion coefficients obtained using the new method have a constant value of $1.7\,\mu m^2/ms$, which is the expected value for water self diffusion in this sample at 18 °C. The results obtained with the Cotts' sequence show a clear deviation from this value.

It should be mentioned that in the new asymmetric pulse sequence the maximum value of q is significantly lower than that which is obtainable with Cotts' sequence, for a given maximum gradient strength. However, since modern NMR probes are usually equipped with strong gradients, we do not expect this to pose a serious problem to the proposed method. In addition more gradient pulses in the coding intervals as shown in Fig. 3, can increase the maximum q value achievable. This will increase the coding intervals, but the suppression of cross-terms is still fulfilled provided that the coding intervals are less than the background gradient correlation time.

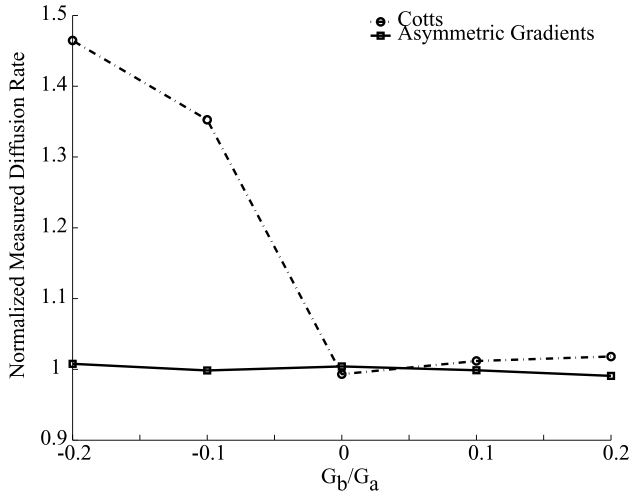

Fig. 5. Normalised apparent diffusion coefficients obtained from the attenuations presented in Fig. 4.

It is well known that the eddy currents introduced by the pulsed gradients influence the signal acquisition. Wu et al. [17] showed that using bipolar gradients can reduce the eddy current effects. In the new asymmetrical gradient sequence presented in this work, the eddy currents can not self compensate as in the case of Cotts' sequence because the pair of gradients have the same polarity. However, the smaller strength of the last pulsed gradient may reduce the eddy current effects. Since the time constant for the gradient is less than 300 μs in our system, no eddy current effects were observed in our diffusion experiments. In systems with gradient coils having long ring down time, a further comparison of the eddy current effects between Cotts' sequence and the new asymmetrical sequence is worthwhile.

In this study, diffusion in a homogeneous sample with an artificially varying background gradient was used to model molecular displacement in heterogeneous systems. The results clearly showed that the new pulse sequence produces more reliable diffusion attenuations in this extreme case. However, the higher apparent diffusivity is in contrast to what has been observed in heterogeneous systems in the presence of a spatially distribution of background gradients, where a lower apparent diffusivity is obtained [13,14,18,19]. In our study all the molecules in the sample experience the same background gradient, which attenuates all the spins to the same degree. In the presence of a spatial distribution of background gradients different pools of spins are attenuated to different degrees. Clearly, the structure and properties of heterogeneous samples introduce complex effects such as restricted diffusion, surface relaxation, as well as the above mentioned temporal dependent g_b. Further analysis of these effects on the asymmetric sequence for flow and diffusion experiments in heterogeneous samples is under investigation.

5. Conclusions

In heterogeneous samples spatially non-uniform background gradients are present. Molecular displacement may introduce time-dependent background gradients. Experiments have shown that application of bipolar gradients then fails to suppress all the cross-terms between the applied and background gradients. We have presented a new asymmetrical gradient pulse sequence, which can suppress cross-terms caused by molecular displacement during the storage interval. The method is based on an independent suppression of the cross-terms in the encoding and decoding intervals of the stimulated echo pulse sequence. This new sequence removes the effects of background gradient up to q^2, providing very reliable diffusion measurements even when the sample was exposed to a time varying background gradient. The method presented in this study can therefore be applied in diffusion related measurements in systems with strong susceptibility fields.

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