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Low magnetic fields for flow propagators in permeable rocks

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Abstract

Pulsed field gradient NMR flow propagators for water flow in Bentheimer sandstone are measured at low fields (¹H resonance 2 MHz), using both unipolar and bipolar variants of the pulsed gradient method. We compare with propagators measured at high fields ¹H resonance 85 MHz). We show that (i) measured flow propagators appear to be equivalent, in this rock, and (ii) the lower signal to noise ratio at low fields is not a serious limitation. By comparing different pulse sequences, we study the effects of the internal gradients on the propagator measurement at 2 MHz, which for certain rocks may persist even at low fields. $© 2006 Elsevier Inc. All rights reserved.$

Keywords: Low magnetic fields; Flow propagators; Internal gradients; Signal to noise ratio

1. Introduction

Increasingly, laboratory nuclear magnetic resonance (NMR) measurements are being made at ever-higher magnetic fields to enhance resolution and signal to noise ratio $(S/N$ or SNR). In a different context, NMR at low magnetic fields (¹H resonance frequency $\omega_0/2\pi \leq 2$ MHz), and with low-resolution magnets, is now widely used as a borehole measurement in petroleum and other geophysical exploration [\[1,2\].](#page--1-0) With the growing demand for hydrocarbons there is an immense and imminent need for developing new NMR laboratory techniques, for application to sedimentary rocks and other porous media, at these lower frequencies.

A laboratory technique receiving much current attention is the measurement of the NMR flow propagator [\[3–15\]](#page--1-0) and the NMR time-of-flight technique [\[16–18\]](#page--1-0). This is mainly because the largest length scales that can be probed by diffusion and relaxation measurements [\[19–21\]](#page--1-0) are \sim 100 μ m, which can be less than the size of the largest pores in some rocks, especially carbonates (limestones and dolomites). Furthermore the actual physics of flow in complex porous media is of interest in a wide range of processes in chemical, geological, and biological systems. Dispersion, the transport of molecules or tracers due to combined effects of diffusion and fluid flow at low Reynolds number, is an important problem both in the fundamentals of hydrodynamics [\[22–24\]](#page--1-0) and in its application in diverse fields including biological perfusion, chemical reactors, soil remediation and oil recovery. These flow processes are controlled by the nature of the interconnections, and the topology of the pore space over length scales equivalent to many pores. Although for highly heterogenous samples, multiple length scales can be important [\[7,8,12,24–27\]](#page--1-0) and not all are accessible by NMR methods, the NMR flow propagators can nevertheless probe displacements of the order of \sim 5 mm. This is almost two orders of magnitude larger than those achievable by the diffusion and relaxation methods currently used, in petroleum industry practice, as probes of the rock pore space.

Hitherto, most laboratory pulsed-field-gradient (PFG-NMR) experiments measuring flow propagators and dispersion in porous media have used high (typically 85 MHz 1 H resonance) magnetic fields [3-9,11-16,18]. The advantage of high fields in SNR is well-known; their draw-back is a strong increase in the deleterious effects of

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induced internal field gradients. Contrast in magnetic susceptibility $\Delta \chi$ between fluid components and solids gives rise to induced internal field gradients g_{int} which increase with static field $B_0(=\omega_0/\gamma)$. These may be reduced by reductions in B_0 . The reduction is at least linear; in [\[28\]](#page--1-0) it is shown that diffusion in the larger pores imposes a maximum effective gradient g_{max} , which scales super-linearly as $B_0^{3/2}$. For the first time, we compare the propagators measured at low fields (2 MHz present work) to those determined at high fields (85 MHz [\[14\]\)](#page--1-0) using adjacent core-plugs and using similar pulse sequences. We conclude that internal gradient effects on the flow propagator can be satisfactorily ameliorated at high fields if (but only if) the correct pulse sequence is used. At low fields, internal gradients are greatly reduced, and more protocol options are available in PFG experiments [\[29\].](#page--1-0) In particular, we show that the unipolar PFG archetypes [\[30,31\]](#page--1-0) are usable when internal gradients are sufficiently small. Even when they persist, as in our study rock, the unipolar protocols remain marginally acceptable, subject to a small penalty on SNR and larger systematic errors on the moments of the propagator.

Flow propagators may also be measured at low fields with fixed-field-gradients (FFG) in the fringing field of a superconducting magnet [\[32\].](#page--1-0) However, unlike the PFG experiments used here, only a more limited number of FFG pulse sequences can be used in fringe fields. In particular there are no analogues of the bipolar variants available in PFG experiments. Our data implies that in the case of our study rock (a sandstone), FFG experiments would be subject to quantitative errors in the propagator measurement even at low fields, whilst in many carbonates they would not.

We consider the signal to noise ratio SNR of the low field experiments in more detail. According to [\[33\]](#page--1-0) this scales as $\omega_0^{7/4}$ for equivalent resonator geometries. A mitigating factor is the solenoid resonator geometry used in our low-field system. This yields an advantage (relative to a saddle coil) of a factor \sim 3.1 [\[33\]](#page--1-0) additional to the frequency scaling. At 2 MHz, relative to 85 MHz, we estimate a reduction in SNR of \simeq 245 (see Appendix C). Nevertheless, our practical results show that this is not a serious limitation, and dispel previous misconceptions about SNR limitations for flow propagator measurements at low fields.

2. Experimental

PFG-NMR propagator experiments measure the statistical distribution of fluid displacements (ζ) for a chosen mean flow velocity (v) during a chosen flow evolution time (Δ) . The (tunable) length scale in PFG-NMR experiments is given by $\langle \zeta \rangle_0 = v \Delta$, where $\langle \zeta \rangle_0$ is the mean displacement during time Δ along the mean flow direction (\hat{y}), i.e. $\langle \mathbf{v} \rangle = [0, v, 0]$. v is a volume-average "interstitial" or capillary flow velocity given by $v = V/A\phi$, where V is the imposed volumetric flow rate, and A and ϕ are the crosssectional area and porosity of the rock, respectively. For the flow rates \dot{V} used here, the maximum capillary flow velocity was $v = 1.10$ mm s⁻¹. From the measured permeability (\sim 2 μ m²), one can estimate a mean capillary diameter $d \sim 30 \text{ µm}$ [\[34\],](#page--1-0) implying a low Reynolds number $Re \approx 0.03$ and a large Péclet number $Pe \approx 15$ regime.

By incrementing values of $q = \gamma \delta g_{v}$, the magnetization wave vector set up by the pulsed gradients, the entire probability distribution function of these displacements $P(\zeta|\Delta, \zeta)$ v) may be determined. This is the NMR ''Flow Propagator", for chosen Δ and v. Alternatively, a more limited measurement of the moments of the distribution of $P(\zeta|\Delta, v)$ may be attempted. Both approaches have been applied to flow through mono-disperse bead packs and rocks [\[3–9,11–14,16,18\]](#page--1-0). Here, we demonstrate the feasibility of both methods at 2 MHz.

2.1. Rock, core-holder and NMR system

The flow experiments were performed on Bentheimer sandstone, a German building sandstone. The aeolian origin is reflected in a low proportion of clays, and a high hydraulic (Darcy) permeability. Typically about $2 \mu m^2$, this would be regarded as unusually high for a reservoir rock. In Section [4](#page--1-0) we also compare the Bentheimer data to Indiana Limestone for the case of no flow.

Porosity as determined by Boyle's Law helium pycnometry was $\phi = 23$ p.u. ("p.u." means "porosity units", where 1 p.u. $\equiv 1\%$). The rock was saturated in a NaCl brine of conductivity $5 S m^{-1}$. Porosity measured with NMR using a CPMG sequence [\[35\]](#page--1-0), was $\phi = 22.2$ p.u. The small discrepancy in measures of porosity is common, and can be due to several reasons such as imperfect saturation of the rock (residual air), surface or corner flaws causing shape deviations from a right cylinder, and volume changes by swelling of water-sensitive clays. Because of a degraded SNR, the original brine was replaced by deionized water; the NMR porosity was further reduced to $\phi = 21.5$ p.u. Clay swelling under reduced salinity is a plausible explanation. During the multiple months of flowing deionized water and NMR data acquisition, the porosity $\phi =$ 21.5 p.u. was stable within ± 0.2 p.u.

The rock was a cylinder of diameter 1.5 in. nominal (238 mm) and length 62 mm. The diameter 1.5 in. is a standard size in petrophysics laboratory where accurate porosity measurements are critical; measurement accuracy (for any method) rapidly deteriorates in practice for smaller diameter cores. This necessitates both a core holder, and a magnet, probe and gradient system based on such physical sample sizes. The NMR system [Oxford Instruments, Abingdon, UK; model ''Big-2''] with ''Maran-DRX'' console] consists of a 50 mT (nominal) permanent magnet thermostated to 30 °C, equipped with a 53 mm solenoid resonator and ''slab'' format field gradient coils.

A custom-made core holder [ErgoTech Ltd, Conwy, UK] [\(Fig. 1\)](#page--1-0), was used for mounting the rock sample. A glass-fibre/PEEK (poly(ether-ether-ketone)) composite pressure tube confines all pressurized fluids radially, but

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