Developments in MR Diffusion and Microscopy

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High-resolution Diffusion Tensor Imaging on ex-vivo mouse brain

The study of the underlying microstructure of living tissues is essential in biomedicine as it is directly related to neurodegenerative diseases such as Alzheimer's. Diffusion MR is an excellent tool for studying the dynamics of water in tissues, but it suffers from poor signal-to-noise ratio (SNR) and long scan times. Here we report on a super-high resolution protocol for DTI acquired with a CryoProbe, designed to explore the limits of the hardware.

Methods

A mouse brain was extracted and stored in PBS. Prior to the experiments, the sample surface was dried from PBS and transferred to a 10 mm sample tube filled with fluorinated oil.

Experiments were performed on a 400 MHz WB Bruker AV-NEO system equipped with a three-axis Micro2.5 gradient set and a CryoProbe. A standard diffusion tensor imaging sequence was used with a segmented EPI readout (10 segments) and three b-values as shown in Fig.1. Other acquisition parameters were Resolution: 35x35x35 μm^3, TE = 18.3 ms and TR = 1000 ms. The total scan time was approximately 90 h. In addition, six low b value data sets were acquired to stabilize the diffusion tensor fit.

Results

With only two scans (Fig. 1), ultra-high resolution ex vivo parametric DTI data was achieved with an adequate signal-to-noise ratio. This allows for optimal fibre tracking with isotropic resolution (Fig. 2).

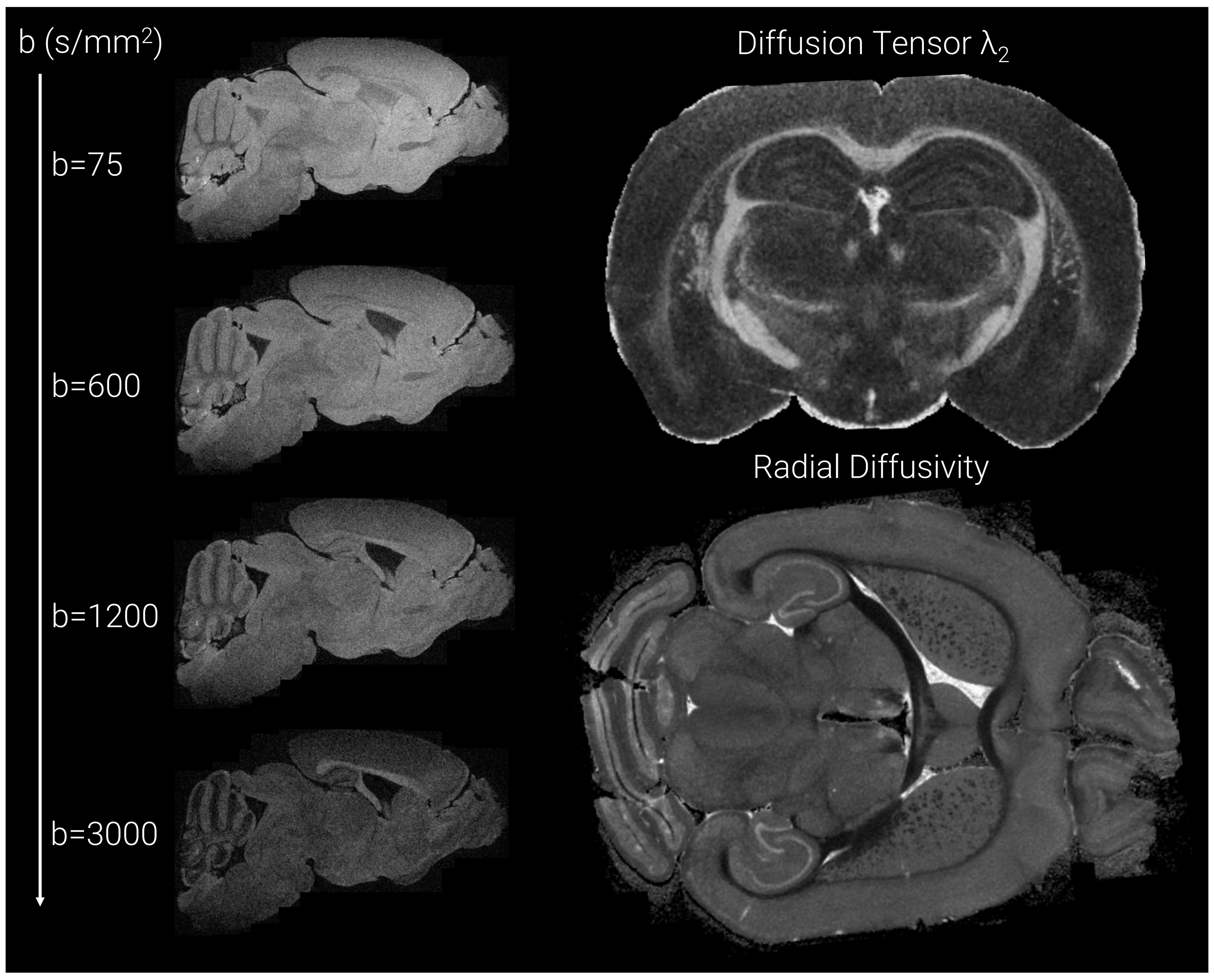

Fig. 1: Diffusion weighted images at various b-values. The different structures are apparent. Eigenvalue component map in an axial orientation. Radial diffusivity as derived from the diffusion tensor (sagittal)

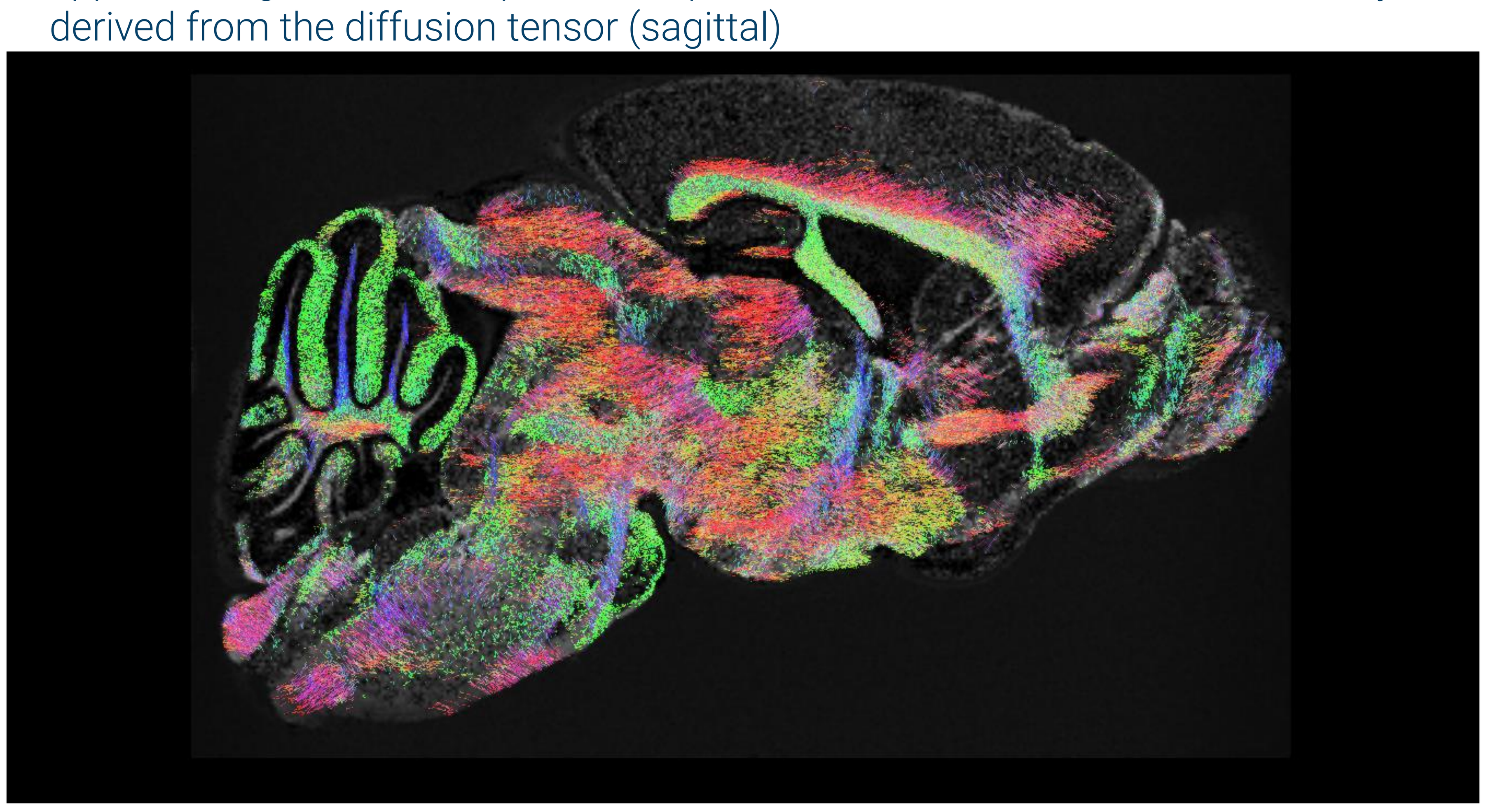

Fig. 2: White matter fiber tracking based on the diffusion tensor.

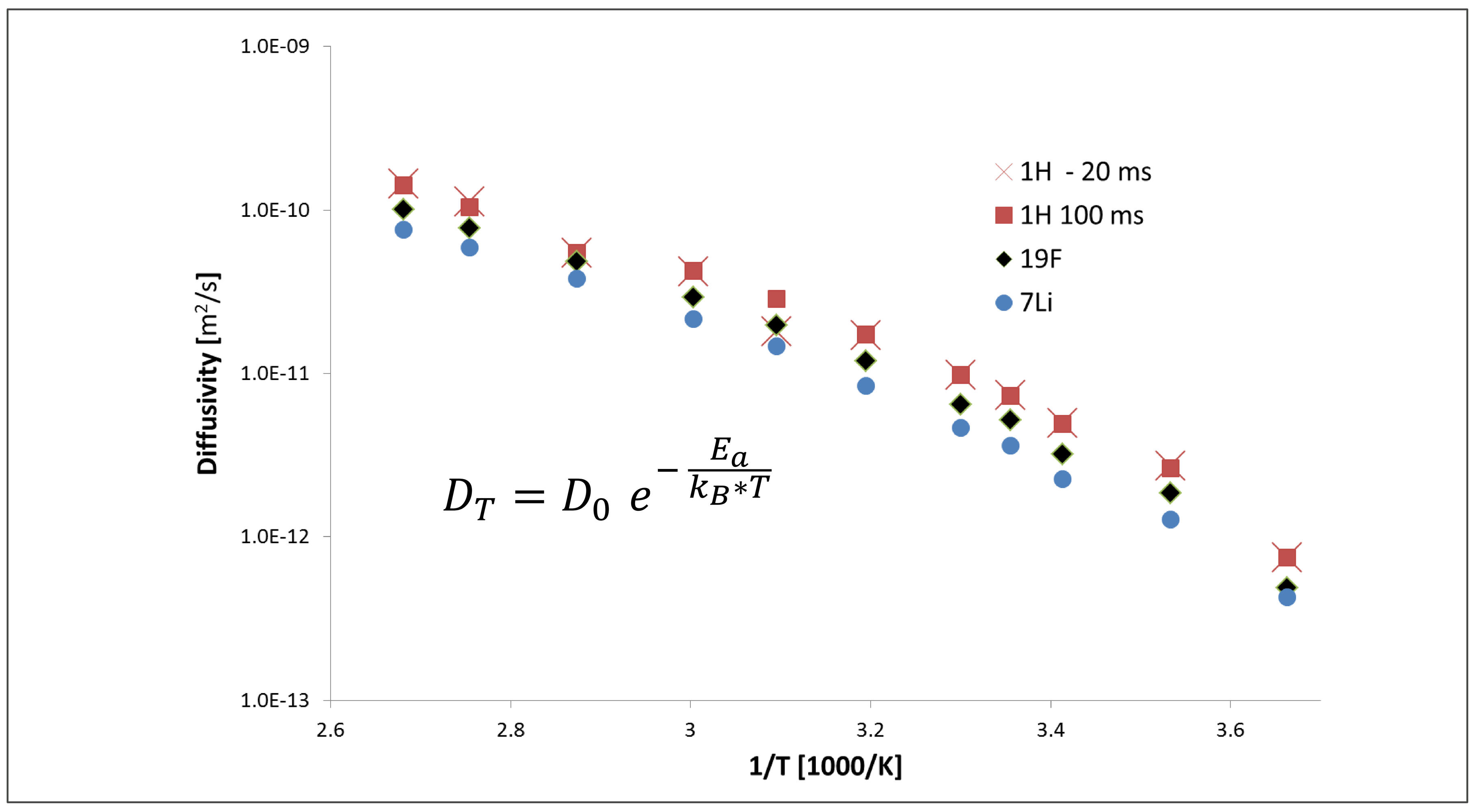

Fig. 3: Arrhenius plot of the diffusion coefficients of 1H, 19F and 7Li with respect to temperature. The activation energy is approximately the same for all ions (0.43 eV). Temperature range 0°C to 100°C

Ion-Mobility by Multi-Nuclear PGSE NMR

The internal resistance of a lithium-ion battery is strongly dependent on the mobility of the ions involved. Improving the transport properties of the target ions, e.g., by molecular additives, is imperative in battery design. Pulsed gradient spin echo (PGSE) NMR can measure the mobility of different ions independently and in situ by measuring the diffusivity of different nuclei, such as ^{1}H, ^{19}F, ^{23}Na and ^{7}Li. PGSE NMR is therefore the ideal tool for measuring the mobility of ions.

Experimental

As an example of a realistic battery electrolyte, we used an ionic-liquid 0.5 molar LiTNf2, often called LiTFSI, dissolved in [bmpy][NTf_2][1)]. To avoid convection perturbations, the sample was filled into a 2 mm capillary tube (id about 1.3 mm), which was placed axially centered at the bottom of a regular 5 mm NMR tube.

The DiffBB SB probe is equipped with a proton channel tunable to 19F with a lock channel attached to the same coil. The X-channel is tunable from ^{31}P down to ^{15}N.

As the probe has automatic tuning and matching (ATMA) for both channels experiments on three nuclei, here ^{1}H, ^{19}F, and ^{7}Li, could be performed at different temperatures in full automation.

The pulse sequence in use was a PGSTE[2)] sequence, preceded by a spoiler recovery sequence[3)] with recovery delay.

Summary

These experiments were designed to demonstrate the potential of PGSE diffusion in lithium-ion battery research. In particular, the use of the SB-DiffBB probe greatly facilitates automated measurements of such materials. The accessible diffusion coefficients for 7Li were further reduced by increasing the diffusion time.

1) Paul M. Bayley et al., J. Phys. Chem. C 2010, 114, 20569–20576

2) Tanner, J.E., J. Chem. Phys., 52, 2523, 1970.

3) G.H. Sørland et al., Diffusion-Fundamentals.org 15 6, 1 (2011)

Conclusion

- Ultra-high resolution ex-vivo imaging
- Optimal fibre-tracking at isotropic resolution
- Independent detection of the mobility of different ions in a battery electrolyte.
- Absolute measurement of diffusion coefficients
- Wide temperature range

Technology