Ultra-Low Field NMR Relaxometry: Calibration Method and T1-Dispersion below 1000 Hz
Vasileios Zampetoulas\textsuperscript{1}, Lionel M. Broche\textsuperscript{1}, and David J. Lurie\textsuperscript{1}
\textsuperscript{1}Aberdeen Biomedical Imaging Centre, School of Medicine & Dentistry, University of Aberdeen, Foresterhill, AB25 2ZD, Aberdeen, United Kingdom

TARGET AUDIENCE
This work is targeted at scientists working on techniques and applications of NMR relaxometry, who wish to extend their work to ultra-low magnetic fields in order to expand the range of biomedical applications and the information obtained from these measurements.

PURPOSE
Fast Field-Cycling (FFC) NMR is a readily applicable technique that is used for the investigation of properties of a range of materials including polymers, liquid crystals, and biological samples, with applications in chemistry, oil industry and medicine to cite a few. A graph of $T_1$ versus the magnetic field (known as a $T_1$-dispersion curve) is obtained via FFC NMR measurements and can be developed into a new diagnostic tool thanks to the information about molecular dynamics which it provides.

Without correction for external magnetic fields a FFC relaxometer can measure $T_1$ down to magnetic fields as low as 10 kHz proton Larmor frequency. However, attempts to make measurements below that range may be corrupted by the presence of stray magnetic fields arising from the Earth, ferromagnetic structures or other sources of interference. Therefore it is necessary to compensate for these external sources of magnetic field and to properly and robustly calibrate the compensation system. When the corrections are implemented successfully the $T_1$-dispersion curve can extend to magnetic fields in the region of $\mu$T, allowing for the investigation of much slower molecular motions.

METHODS
The calibration is achieved with the implementation of FFC measurements in a range of fields close to zero Tesla along with correction magnetic fields of varying magnitude and orientation applied by the relaxometer. During this process, the magnetisation precesses around a resultant field of unknown direction composed of the correction and stray magnetic field, with the frequency of precession determined by its magnitude. Since the correction magnetic field varies, the direction and magnitude of the resultant field changes, leading to variations in the frequency of precession. During this exposure to low fields the varying precession frequency is measured and plotted, with the minimum frequency indicating the correction magnetic fields that lead to a resultant field of minimum magnitude and thus a correct calibration.

RESULTS
As indicated by the calibration, the stray magnetic fields applied in the bore of our FFC magnet are compensated with the application of a longitudinal correction magnetic field of 23.5 $\mu$T and a transverse correction magnetic field of 7.2 $\mu$T oriented at 43° from the position of the device. This leads to the extension of the $T_1$-dispersion curves to the region of $\mu$T, as shown from the FFC measurements on samples of the polymer Polydimethylsiloxane (PDMS, Figure 1) and tendons obtained from bovine leg (Figure 2).

DISCUSSION
In Figure 1, the continuous slope shown in the dispersion curve acquired after the calibration extends down to 400 Hz in terms of $^1$H Larmor frequencies measurable and is similar to the one found in the literature\textsuperscript{1}, indicating a correct calibration for the stray magnetic fields that is achieved within 1 hour. In the dispersion curves of bovine tendons (Figure 2) the 3 quadrupole peaks shown between the magnetic fields of 3.5 MHz to 1.5 MHz and 0.9 MHz to 0.4 MHz are developed due to known interactions between $^1$H and $^{13}$N nuclei.\textsuperscript{2} Additionally, the differences in the slope between the 2 curves of 45° and 90° at the segments below 10$^3$Hz, and the offset of the curve of 0° can be related to tissue anisotropy in tendons.

CONCLUSION
This work shows that it is possible to calibrate a FFC NMR relaxometer in a reasonable time frame so that the $\mu$T range is accessible for experimentations. This is expected to provide clinically relevant information on slow dynamic processes in tissues and should allow measuring the local magnetic fields generated within the tissues. Future work will include the translation of this technique to our FFC MRI device as well as a series of clinical experimentation to explore how this portion of the dispersion curve can be exploited in medicine.

REFERENCES
\begin{thebibliography}{9}
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\begin{figure}[h]
\centering
\includegraphics[width=0.45\textwidth]{Figure_1.png}
\caption{$R_1$-dispersion curves of a sample of PDMS acquired before (grey circles) and after (red triangles) the calibration.}
\end{figure}

\begin{figure}[h]
\centering
\includegraphics[width=0.45\textwidth]{Figure_2.png}
\caption{$R_1$-dispersion curves of 3 samples of bovine tendons with the tendon fibres oriented at about 90° (blue circles), 45° (green rectangles), and 0° (red triangles) with respect to $B_0$ acquired after the calibration.}
\end{figure}