



28 years of nano-pore analysis by NMR Cryoporometry : background, capabilities, comparison with other techniques, instrumentation & protocols, and recent developments.

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Abstract:

NMR Cryoporometry (NMRC) is a powerful technique for the measurement of pore-size distributions and total porosities on a pore length scale from sub 1nm to over 1micron. This technique is suitable for measuring pore-sizes in a wide range of polymers and porous materials, including porous glass, rock, clays and porous carbons including biochar. It offers various advantages over other techniques, including the ability to study wet samples. By swelling rubbers and polymers with added organic liquids cross-link density and nano- to micro-porous properties of the polymer may be obtained. In biochar, progressive changes to the quantity and mobility of hydrocarbons, as well as changes in pore-blocking, as a function of preparation temperature, have been demonstrated. The capabilities of NMRC have in recent years been extended in a number of directions, to greater sensitivity, to sub-nanometric pore sizes (lower temperatures) and to above micron sized pore-sizes (tiny melting point depressions). NMRC has been used to probe the effect of using different probe liquids on measured pore volume. In the nearly 30 years that Lab-Tools have been developing NMRC, the protocols have evolved. The data formats have been formalised, and the programs to access and display the NMRC control variables and results data have been optimised. Some of these protocols are now encoded into Graphical User Interface (GUIs) and their associated process and graph windows.

Biography:

Beau Webber gained his PhD at the University of Kent, UK (thesis : "Characterising Porous Media"). He has published 50 papers in refereed journals, and is called upon referee. He makes use of a wide range of measurement techniques for studying porous materials and liquids contained in them and has made extensive use of Central Facilities neutron scattering instrumentation at Grenoble, Paris and Abingdon, for these studies. He is director of Lab-Tools (nano-science) Ltd. a small UK spin-off research laboratory that performs academic and commercial con-



tract nano- to meso- materials-science research, studying the structure, dynamics and phases of liquids and their solids (and also gas hydrates) in confined geometry and at and near surfaces. Lab-Tools also designs, implements and sells the cutting-edge NMR instrumentation needed to carry out this research, including NMR relaxation spectrometers and NMR cryoporometers.

Publication of speakers:

1. Nuclear Magnetic Resonance Cryoporometry J. Mitchell, J. Beau W. Webber and J.H. Strange. Physics Reports, 461, 1-36, 2008. DOI: 10.1016/j.physrep.2008.02.001
2. An evaluation of NMR Cryoporometry, Density Measurement and Neutron Scattering methods of pore characterisation. J.B.W. Webber, J.H. Strange and J.C. Dore. Mag. Res. Imag., 19, 3-4, 395-399, 2001.
3. Studies of nano-structured liquids in confined geometry and at surfaces. J. Beau W. Webber. Progress in NMR Spectroscopy, 56, 1, 78-93, 2010. DOI: 10.1016/j.pnmrs.2009.09.001
4. An NMR study of porous rock and biochar containing organic material. J. Beau W. Webber, Patrick Corbett, Kirk T. Semple, Uchenna Ogbonnaya, Wayne S. Teel, Carrie A. Masiello, Quentin J. Fisher, John J Valenza II, Yi-Qiao Song, Qinhong Hu. Proceedings of the 11th International Bologna Conference on Magnetic Resonance in Porous Media (MRPM11), University of Surrey, 2012. Microporous and Mesoporous Materials, 178, 94-98, 2013. DOI: 10.1016/j.micromeso.2013.04.004

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