Lab-Tools Ltd.

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Lab-Tools are based at University of Kent's Canterbury Enterprise Hub, with research and analytic labs at Hersden:

We have in excess of 80 scientific publications in this field of porous media and liquids in confined geometry. For measuring pore size distributions, we find that NMR Cryoporometry is often the method of choice over other more established techniques.

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Lab-Tools
http://www.lab-tools.com

NMR Cryoporometry

an analytic service

from

Lab-Tools Ltd.

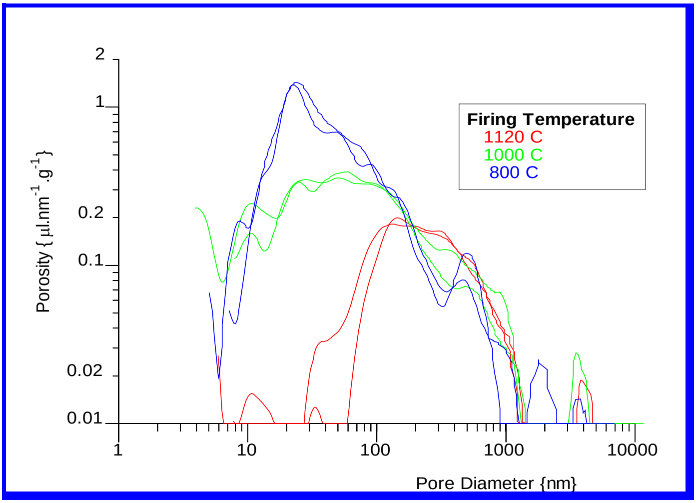
Science and Metrology
on the
nano- through meso- to micro-scale.

NMR Cryoporometry is a novel pore size distribution measurement technique that we have developed at the University of Kent and at Lab-Tools Ltd.
NMR Cryoporometry is well suited to characterising structured matter on a scale of about 2nm to 2 µm.
NMR Cryoporometry also measures particle size distribution and packing density, for densely packed particles.

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

NMR Cryoporometry offers the following advantages :

- The pore size calibration is in good co-linear agreement with gas adsorption measurements.
- NMR cryoporometry can measure over a wider pore size range than gas adsorption or thermoporosimetry.
- NMR cryoporometry can measure wet samples, and measures the true volume of pores for liquids.
- NMR cryoporometry measures a static signal; thus the measurement can be made arbitrarily slowly to improve signal-to-noise and resolution, whereas thermo-porosimetry senses a transient, dynamic signal, limiting the resolution.
- NMR cryoporometry has no 'preferred' pore distribution curve, such as the Gaussian distributions often assumed by inversion techniques.
- NMR cryoporometry may be combined with standard NMR imaging techniques to perform non-destructive 1, 2 or 3 dimensional macroscopic spatial imaging of pore size distributions.



NMR Cryoporometry pore-size distributions : A clay fired to three different temperatures -

Cryoporometry provides information that may be related to the durability and frost resistance of clays and clay mixtures.

NMR Cryoporometry measures pore volumes and pore size distributions on the 2nm to 2μm size range. It can also measure particle size distributions and packing, for densely packed particles.

Cryoporometry makes use of the fact that small crystals of a liquid in the pores melt at a lower temperature than the bulk liquid. This is the **Gibbs-Thomson effect: *the melting point depression is inversely proportional to the pore size*.**
A liquid is imbibed into the porous sample, the sample cooled until all the liquid is frozen, and then warmed slowly while measuring the quantity of the liquid that has melted.
Nuclear Magnetic Resonance (NMR) is used as a convenient method of measuring the quantity of liquid that has melted, deep inside the porous mass, as a function of temperature.

NMR Cryoporometry is, like gas adsorption, a thermodynamic method of measuring structure size. NMR Cryoporometry is closely related to thermoporosimetry, but has significant advantages. These techniques are all governed by the same set of Gibbs equations:

- Cryoporometry is the constant pressure case, and gas adsorption the constant temperature case;
- Cryoporometry uses the change between the solid and liquid phases of the imbibed liquid, and gas adsorption the change between the liquid and vapour phases.

NMR Cryoporometry pore-size distributions : porous silicas -

Normalised pore size distributions :

- **blue curves are sol-gel silicas,**
- **the red curve is for SBA-15,**
- **the green curves are CPG silicas.**