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Short Communication

Time-Domain benchtop and mobile NMR for liquid & solid material science, pore-size measurement and process monitoring

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Abstract

We discus Time-Domain NMR, as a method of measuring the physical properties of liquid and solid materials. Time-Domain NMR is also a good technique for measuring pore-size distributions from the nano-meter to microns.

When an NMR measurement is made on a sample, the perturbed magnetisation of the nuclei creates a measurable signal that evolves with time. This signal is often then Fourier-Transformed to give spectral information, but for physical information there are many advantages in studying the captured time-domain signal.

Time-Domain NMR is excellent for quantified monitoring of physical change, particularly as a function of some changing parameter such as time or sample temperature. Thus it is a superb tool for material science studies on both liquids and solids and also hence for process monitoring and control.

NMR time-domain relaxation (NMRR) is most useful for quantitative material science measurements of both the mass and what is described in various fields as the mobility / dynamics / stiffness / viscosity / rigidity of the sample, particularly of solid hydrocarbons, rubbers and other polymers. These properties may be measured both in the bulk and in nano-meter and upward sized pores. These properties may be determined from the time-evolution of the NMR signal from the sample.

There are various methods for studying porous materials by time-domain NMR; NMR is an excellent method for studying pore structure, as it can 'see inside' the pores. Many of these methods work by filling the pores with a material, such as water, that then gives a measurable NMR signal modified by the pore structure.

NMR Relaxation (NMRR)

NMR Diffusion and Percolation

NMR Imaging

NMR Relaxometry

NMR Cryoporometry (NMRC)

NMRC works by freezing a liquid in the pores, and then slowly warming the sample. One just simply uses NMRR as a robust method to determine the quantity of liquid that has melted at a particular temperature.

Lab-Tools Ltd. have now extended NMRC for measuring distributions of pore sizes, to cover measurements over 3 orders of magnitude in pore dimensions. Examples will be given of some of the measurements that have been performed. As part of the evolution of applying time-domain NMRR for making NMRC measurements, Lab-Tools have developed a highly compact precision NMR time-domain relaxation spectrometer, suitable for use on the laboratory benchtop and in the field. The R.F. is processed digitally, on a single chip Field Programmable Gate array (FPGA) which gives it the long-term stability necessary for process control. There is an associated Peltier thermo-electrically cooled variable temperature probe, which together make a high-performance NMR Cryoporometry instrument. This Peltier cooled NMR probe is also highly useful for other temperature dependant material science NMRR measurements. A range of international companies, universities and research institutes are now using NMRC as part of their arsenal of research tools to study their samples.

Note: This work is partly presented at 34th International Conference on Nanotechnology & Expo at November 15-16, 2019 | Madrid, Spain