NMR Using Solution Flow Circuit and Inductively-Coupled Microcoils

Patrick Berthault, Guillaume Carret, Celine Boutin

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Investigation of systems in evolution such as chemical reactions via NMR can be rendered comfortable if the intrinsic lack of sensitivity of this technique is circumvented and provided that quantitative data are obtained.

Here we present an integrated device based on a 3D-printed mini bubble-pump associated with fluidics and micro-detection that overcomes the sensitivity problems inherent to slow return of magnetization to equilibrium in liquid-state NMR. The use of a closed-loop circuit of the solution near the NMR magnetic center presents two main advantages: pre-polarization is achieved for the whole solution volume, this volume can be reduced to tens of microliters.

This device is installable into every commercial liquid probehead without modification; it is easily inserted from the top of the NMR magnet. A gas flow driven by a programmable syringe pump actuates a mini bubble-pump which leads to circulation of the liquid sample. A part of the solution circuit crosses the NMR detection region consisting of a micro-solenoid inductively coupled to the coil of the commercial probehead. In order to optimize this coupling a rod fixed on the upper part of the insert and ended by a Vernier placed on top of the magnet enables angular positioning of the micro-coil.

The two resonance frequencies created by the coupling allow one to observe nuclei inaccessible with the host probe alone, or to study two different nuclei with the optimized detection allowed by the microcoil. To further increase the signal-to-noise ratio, this system can also be used to efficiently dispense gaseous species such as hyperpolarized xenon and parahydrogen to the solution. The performances of this device, in particular with cryoprobes, will be presented.

Reference. G. Carret, T. Berthelot, P. Berthault, Analytical Chemistry 90 (2018) 11169–11173, DOI: 10.1021/acs.analchem.8b01775

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Primary author(s) : Dr BERTHAULT, Patrick (CEA); Dr CARRET, Guillaume (Cortecnet); Dr BOUTIN, Céline (CEA)

Presenter(s) : Dr BERTHAULT, Patrick (CEA)

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