

# Novel Flow Cell Designs for Process Monitoring with Compact NMR Spectroscopy

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## Motivation

3D printing of ceramics allows for the design of highly durable flow-through cells for use in process monitoring with NMR spectroscopy. Sophisticated shapes can be realized, yet the materials are compatible with the NMR experiments.

For instance, monitoring fast reactions at high temperatures and pressures requires a suitable mixing devices to be placed in close vicinity to the measuring volume.

In addition, compact NMR instruments pave the way for NMR spectroscopy and relaxometry to enter harsh industrial environments. Failsafe, temperature and pressure resistant flow-through cells are an essential cornerstone to enter industrial production plants and fulfill explosion safety requirements.

## Setups for Process Monitoring [1,2,3,4]

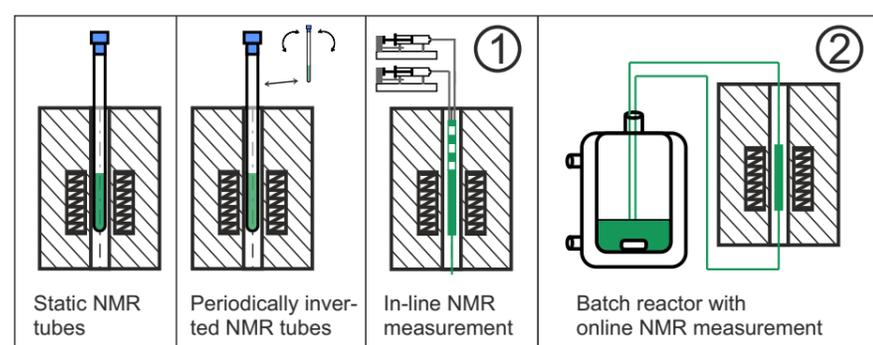


Fig. 1: Reaction monitoring with NMR spectroscopy based on different experimental setups.

## ① Design of an Integrated Mixer

A 3D printable flow-through cell design with an integrated static mixer for low field NMR instruments was investigated by Computational Fluid Dynamics.

With the use of a Lagrangian method, two preselected mixer designs were evaluated. Requirements for the design included fast mixing and minimal gap sizes of 0.8 mm to prevent blockages in the mixing region.

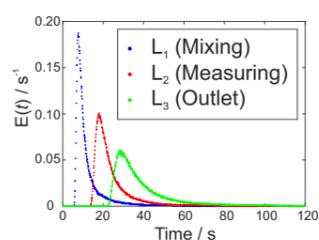


Fig. 2: Simulated residence time distributions  $E(t)$  at  $1.5 \text{ mL min}^{-1}$  mean flowrate at various lengths of flow cell with SMX mixer.

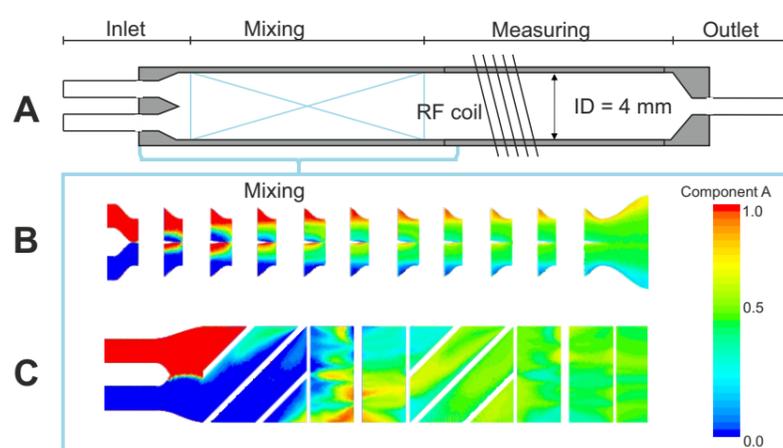


Fig. 3: Basic scheme of proposed NMR flow cell with integrated static mixer (A). Simulated concentration plots for mixing of two substances at  $1.5 \text{ mL min}^{-1}$  using the split and recombine principle (B) and chaotic advection principle (C, SMX mixer).

## ② Comparison of Various Flow Cell Designs

A ceramic ( $\text{ZrO}_2$ ) flow cell was produced via additive manufacturing (Fig 4.1). The geometry was optimized by expanding the diameter in the active region [5]. A continuous flow setup was used for testing.

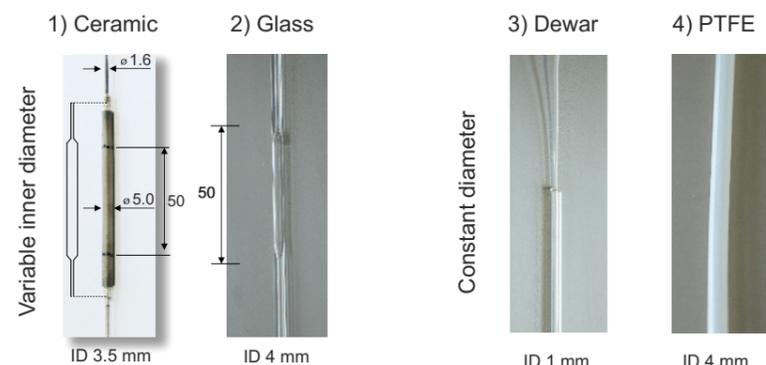


Fig. 4: Comparison of new 3D printed flow-through cell (1) with cells made from common materials (2, 3, 4). Materials: Zirconium dioxide, glass, perfluoroalkoxy (PFA) and polytetrafluoroethylene (PTFE) with varying geometry.

## Measurements in Continuous Flow [6]

- Neat Ethanol as example
- Automated flow setup using Coriolis mass flow controllers
- Randomized flow rates with equilibration time for each setpoint

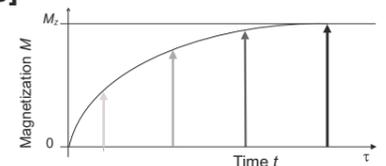


Fig. 5: Magnetization ( $M$ ) of samples is dependent on the longitudinal relaxation time  $T_1$ . In flow measurements,  $M$  is influenced by the hold-up time in the magnetic field.

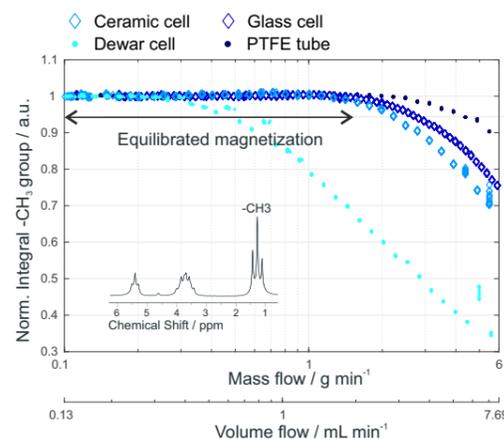


Fig. 6:  $-\text{CH}_3$  Integrals measured with a low-field NMR spectrometer (43 MHz) at varying flow rates of ethanol.

Full magnetization was reached for the ceramic, glass and PTFE flow cells up to  $1.5 \text{ mL min}^{-1}$  (Fig. 6). By contrast, the magnetization within the dewar cell dropped above  $0.3 \text{ mL min}^{-1}$  due to the decreased inner diameter, and thus, increased flow velocity.

## Results and Outlook

- The application of 3D printed  $\text{ZrO}_2$  offers new possibilities for flow cell and static mixer designs
- An integrated design of a static mixer into a flow cell was studied using Computational Fluid Dynamics
- The rough surface of 3D printed materials is challenging due to the adhesion of bubbles and magnetic field inhomogeneities

## Literature

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