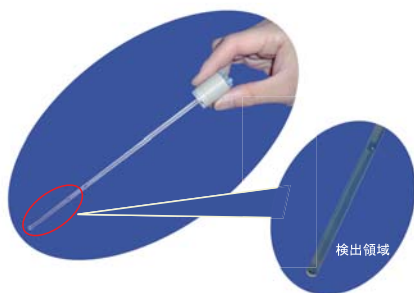


MICCS - NMR Real-time monitoring of chemical reaction

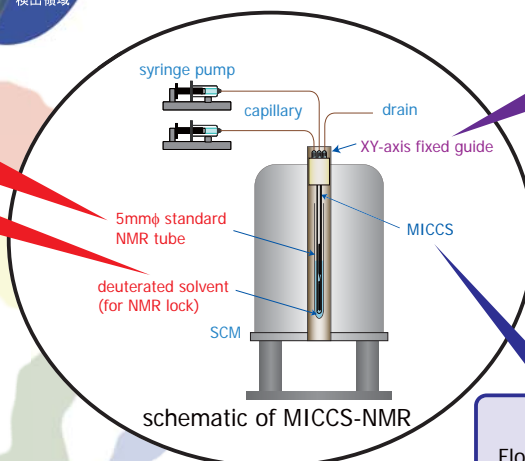


MICCS (Micro Channel Cell for Synthesis monitoring) is an interface microchip designed for standard NMR systems. In MICCS-NMR experiments, chemical reaction is initiated in the cell, by introducing reactants from outside of SCM with syringe pumps through separate inlets. Thereby, MICCS-NMR enables real-time monitoring of chemical reaction.

5mmφ standard NMR tube & deuterated solvents

MICCS is connected with a 5mmφ sample tube. Therefore,

- Standard 5mmφ probe can be used without any modification.
- External lock system permits long-time measurements even when normal solvent for internal precursor is used.



XY-axis fixed guide

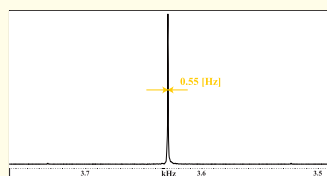
MICCS is loaded along the lane of XY-axis fixed guide attached to SCM. Thereby, the orientation of MICCS is fixed when it is taken in and out, reproducing spectral resolution.



schematic of MICCS-NMR

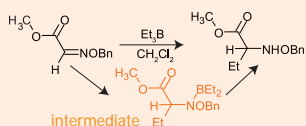
Resolution in MICCS-NMR

Full width at half height of chloroform signal in MICCS-NMR is 0.55Hz, comparable with that in conventional experiments.



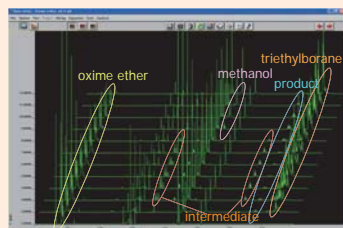
Reaction pathway analysis

MICCS-NMR was applied to monitor addition reaction of ethyl radical to oxime ether, revealing unstable intermediate.



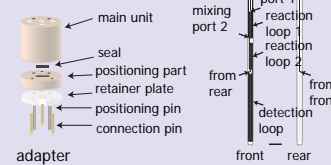
Sample is by courtesy of Prof. T. Naito, Prof. O. Miyata, and Dr. M. Ueda of Kobe Pharmaceutical University.

unit : μL/min		
1M Oxime ether	1M Et ₃ B	1M CH ₃ OH
10.0	0.5	0.0
9.0	1.0	0.0
8.0	2.0	0.0
7.0	3.0	0.0
6.0	4.0	0.0
5.0	5.0	0.0
5.0	5.0	0.5
4.5	4.5	1.0
4.0	4.0	2.0
3.5	3.5	3.5
2.0	4.0	2.0
1.0	5.0	5.0



MICCS

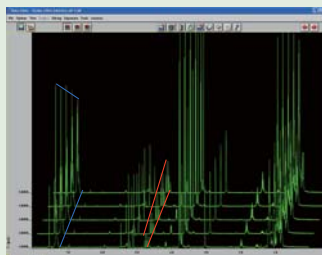
Flow path consists of 3 inlets, 2 reaction loops, 1 detection loop, and 1 drain, enabling 2-step 3-fluid reaction. At fastest, detection can be monitored after 2 seconds of reaction.



Reaction rate analysis

Reaction rate of addition reaction of ethyl radical to oxime ether was analysed by MICCS-NMR.

unit : μL/min	
1M Oxime ether	1M Et ₃ B
20.0	20.0
10.0	10.0
5.0	5.0
2.0	2.0
1.0	1.0



Note

MICCS-NMR is applicable to search for optimal condition of chemical reaction. In contrast, it is not appropriate for the reaction where deposit/gas is generated.

MICCS was developed in "Microanalysis-product system project" supported by NEDO.

