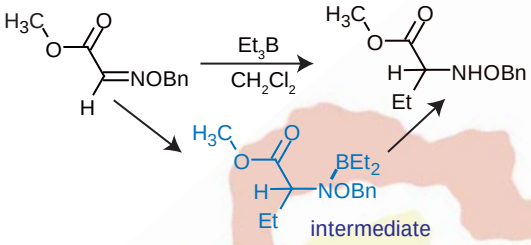
Application of MICCS-NMR #2

Kinetics analysis of radical addition

Reaction:

This note treats triethylborane (Et_3B)-mediated radical addition to oxime ether, where a borane complex is proposed as a key intermediate. However, its isolation is very difficult using conventional methods.



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

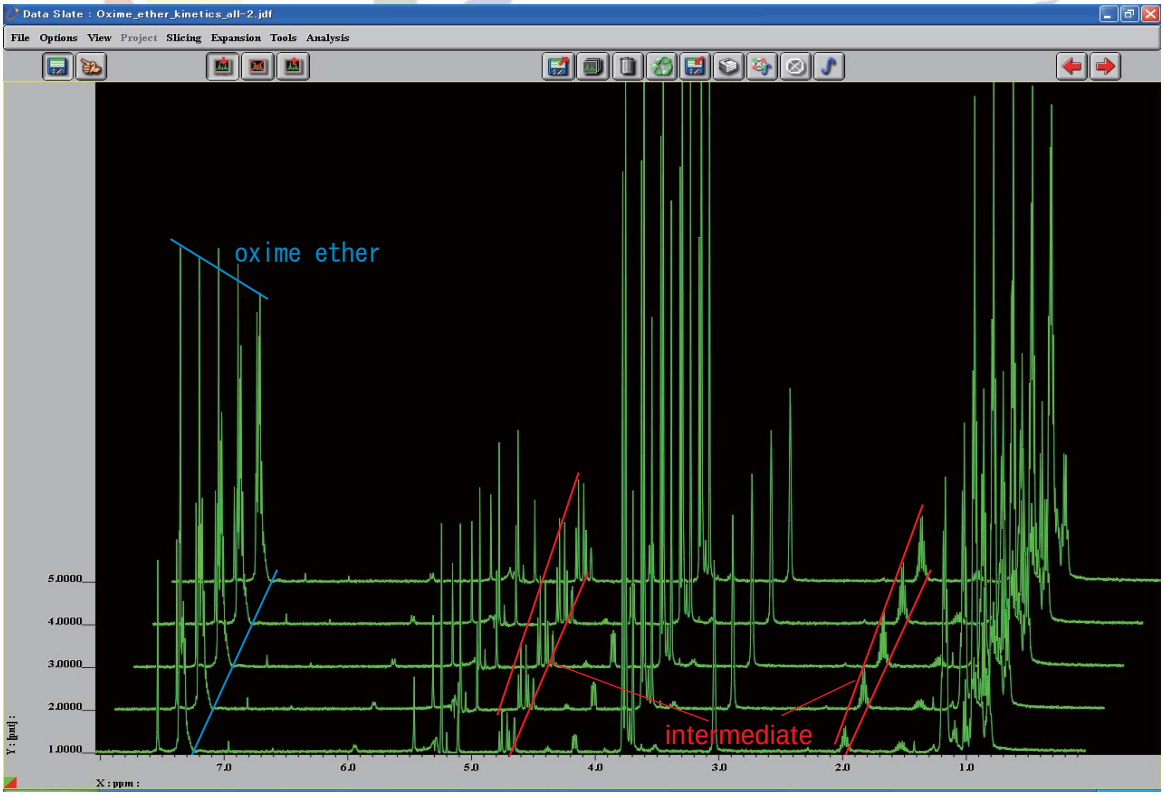
Protocol of NMR measurements:

Information on reaction rate can be obtained by changing time required for the sample in MICCS to move from the reaction point to the detection point. The example shown below demonstrates that, by lowering the entire flow rate with keeping flow ratio of oxime ether and Et_3B to be 1 to 1, NMR signals of reactants gradually decrease while those of intermediate increase.

0.5M oxime ether	0.5M Et_3B
20.0	20.0
10.0	10.0
5.0	5.0
2.0	2.0
1.0	1.0

* Concentration of CH_2Cl_2 solution.

unit : μL/min



Spectrometer: JNM-ECA500
Accumulation: 16scan
Recycle delay: 10s

* CH_2Cl_2 signal is suppressed.