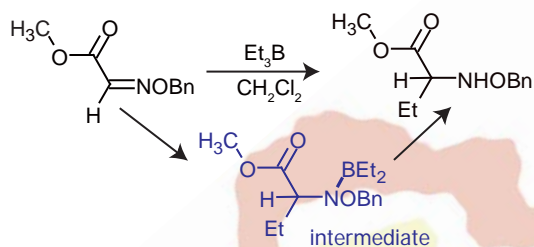


Application of MICCS-NMR #1

Reaction pathway analysis of radical addition

Reaction:

This note treats triethylborane (Et₃B)-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.

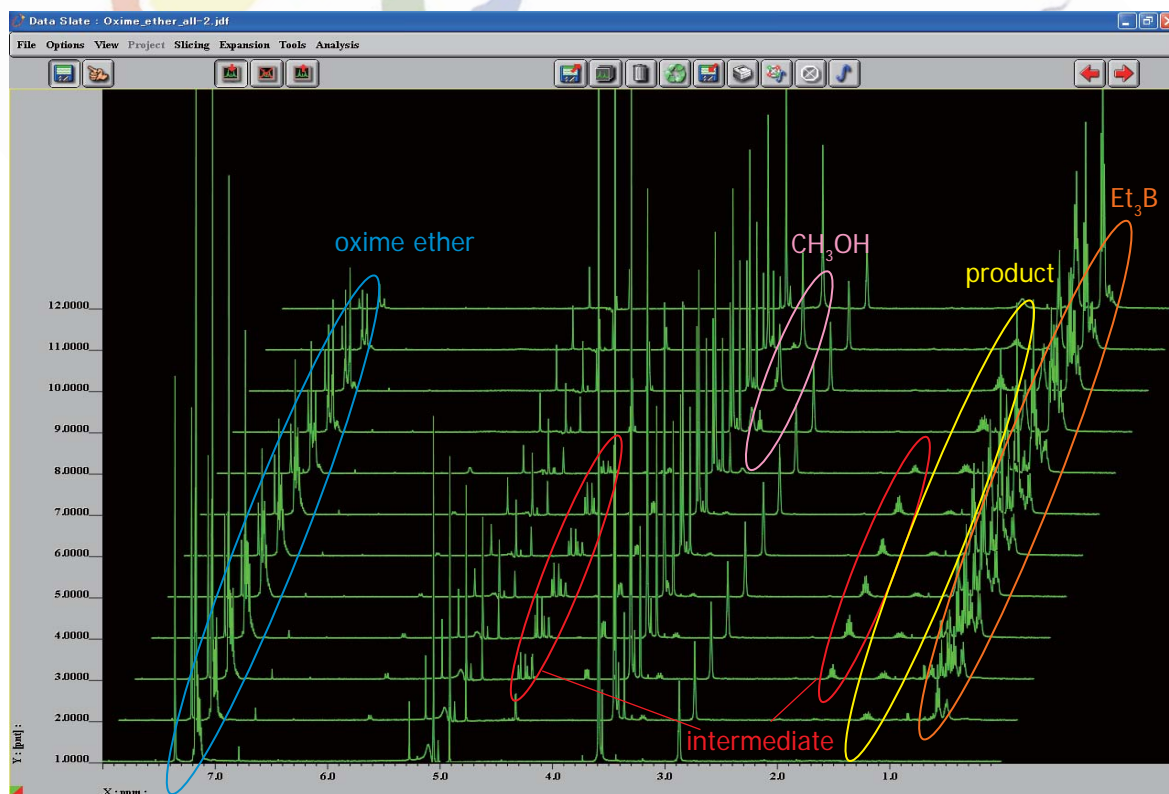
Protocol of NMR measurements:

NMR spectra are observed throughout the reaction, which is initiated by controlling the flow rate of the individual reactants. When the concentration ratio of oxime ether and Et₃B becomes 1 to 1, CH₃OH is added. Thereby, spectra yield signals of reactants, intermediate, and product, sequentially.

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

* Concentration of CH₂Cl₂ solution.

unit : μL/min



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

* CH₂Cl₂ signal is suppressed.



(1/1)