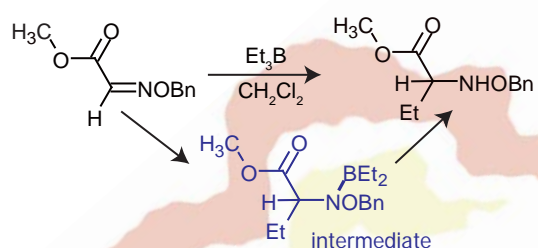


Application of MICCS-NMR #3

Reaction pathway analysis of radical addition by ^{11}B NMR

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.



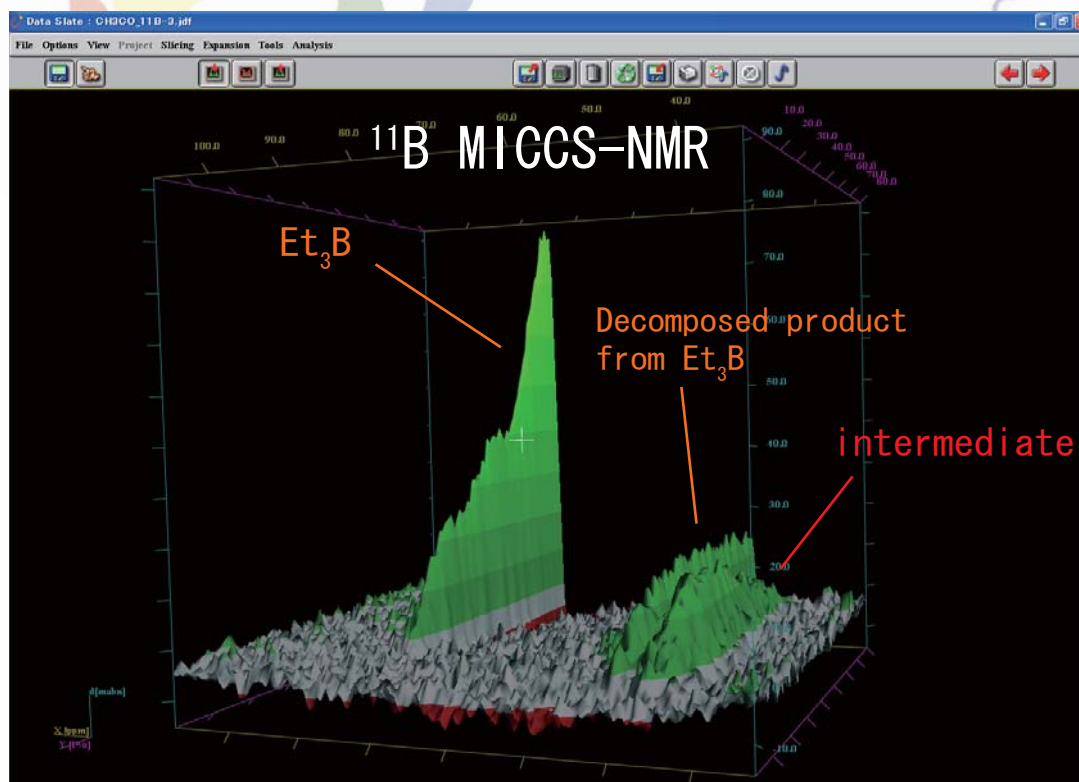
Sample is by courtesy of Prof. T. Naito, Prof. O. Miyata, and Dr. M. Ueda of Kobe Pharmaceutical University.

Protocol of NMR measurements:

^{11}B NMR measurements trace the chemical reaction while Et_3B is added to oxime ether. The figure shown below illustrates that the initial signals decrease when the ratio of oxime ether and Et_3B becomes 1 to 1 and that other signals appear eventually. This behavior permits direct assignment of reactants and intermediate.

Slice No.	0.5M oximeether	0.5M Et_3B
1-10	4.0	0.0
10-20	3.0	1.0
20-30	2.5	1.5
30-40	2.0	2.0
40-50	1.5	2.5
50-60	1.0	3.0
60-80	0.0	4.0

* Concentration of CH_2Cl_2 solution. unit : $\mu\text{L}/\text{min}$



Spectrometer: JNM-ECA400
Pulse sequence: Single pulse
Accumulation: 32scan



(1/1)