Utilities of No-D NMR (No-Deuterium Proton NMR)

Product used : Nuclear Magnetic Resonance (NMR)

No-D NMR (No-Deuterium Proton NMR) technique is a measurement of high resolution ^{1}H NMR spectra without deuterium solvent. It suggests that any reaction mixture or reagent solution are directly available for No-D NMR measurement.
In conventional NMR measurements, ^{2}H signals of deuterium solvent are used for the shimming, however ^{1}H solvent signals are as well in No-D NMR measurements. Since suppression of strong ^{1}H solvent signals by WET eliminates ^{13}C satellite signals also, it is convenient approach to collect ^{1}H NMR spectra without deuterium solvent by No-D NMR measurement.

Merits of No-D NMR

" No-D NMR "is your solution!

- The better approach for unstable compound just after chemical reactions
- Skipping the laborious sample preparation (dissolving into a deuterated solvent after sample purification)
- Cost reduction for deuterated solvent in daily NMR measurement work

Automation script for No-D NMR

The following 5 steps are automated No-D NMR measurement procedure on NMR software Delta, and every tedious measurement parameters are automatically adjusted.

①Shimming
②Detecting solvent signals
③Suppression of solvent signals
④Processing FID data
⑤Chemical shift adjustment

On Delta software, chemical shift in the conventional ^{1}H measurement is not adjusted without NMR lock (Fig. 1a), but the automation of No-D NMR adjusts chemical shift automatically (Fig. 1b). Its result has the same chemical shift information as deuterated solvent sample spectrum (Fig. 1c).

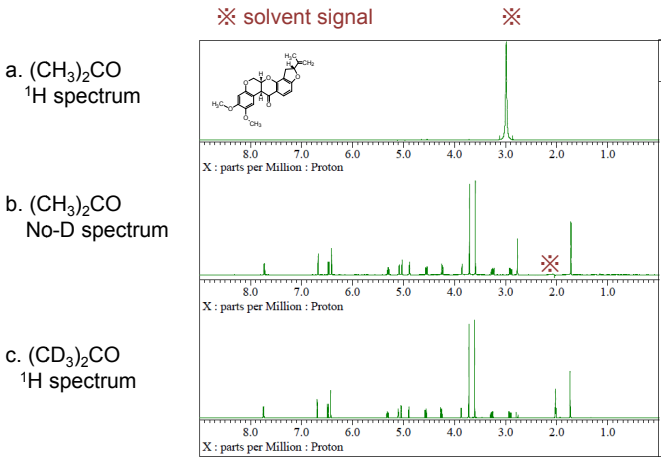

Fig.1 ^{1}H spectra of 5 mg rotenone measured with JNM-ECZ500R

For starting No-D NMR measurement, only simple 2 parameters are required (Fig.2).

1. Select solvent
2. Input the number of suppression signals

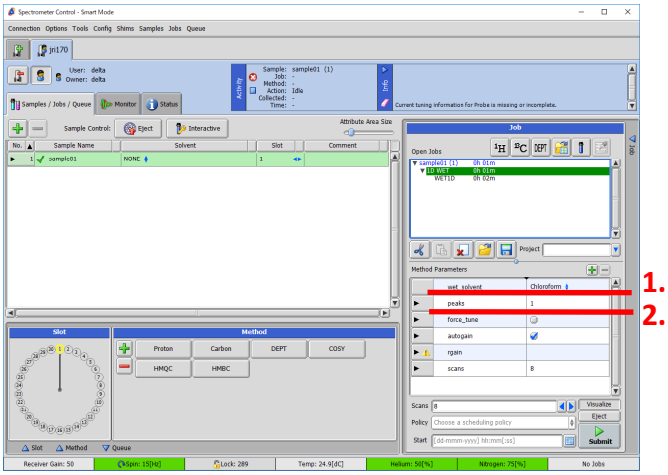

Fig.2 Operation window of No-D NMR (Delta V5)

Application of No-D NMR 1: Observation of exchangeable ^{1}H signals

When the compound with exchangeable proton is dissolved in exchangeable proton solvent (D2O, CD3OD, and etc.), proton atom is replaced with deuterium atom. As results, it is not possible to observe amino protons for the target molecule (Fig. 3 upper). On the other hand, the exchangeable proton can be observed in No-D NMR, since amino protons are kept under protonated solvent (Fig. 3 bottom).

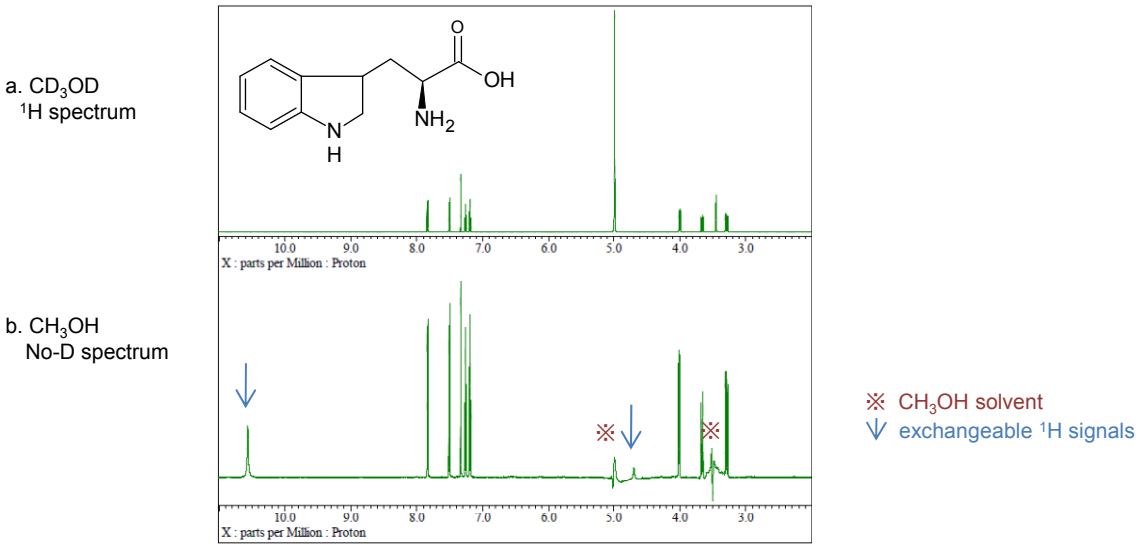

Fig.3 2.5mg *L*-Tryptophan measured with JNM-ECZ600R

Application of No-D NMR 2: Apply to mixed solvent

Because No-D NMR can suppress multi site signals, it is also effective for multi signal solvents such as CH_3OH and mixed solvent sample. Furthermore, ^{13}C decoupling is useful, when solvent ^{13}C satellite signal overlap with sample signal. Fig.5 indicates how sample signal can be distinguished from ^{13}C satellite by ^{13}C decoupling. Chemical shifts in Fig.4 and Fig.5 are intentionally adjusted for better clarity.

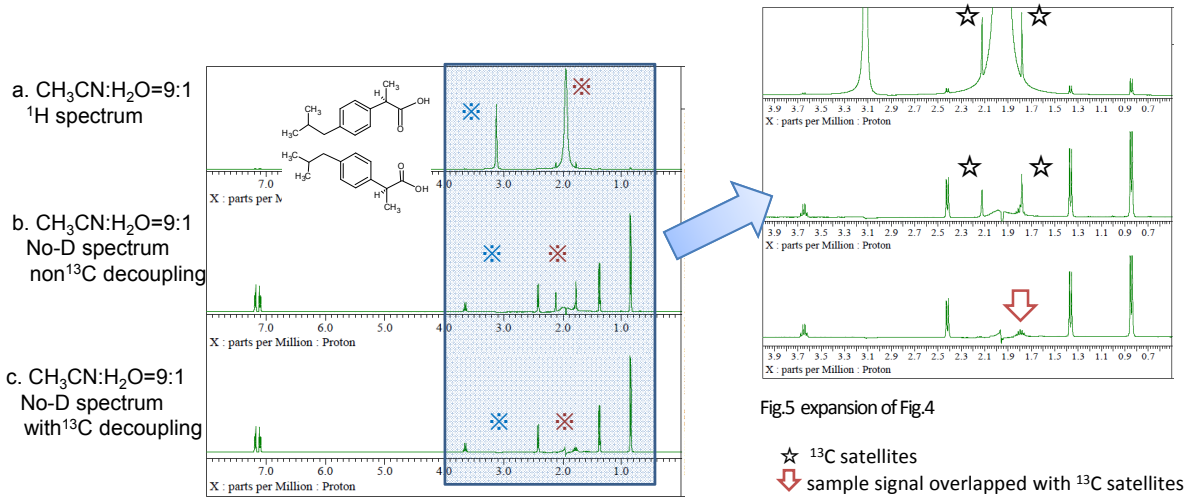

Fig.4 2.5mg ibuprofen (racemic form)
Measured with JNM-ECZ400S

Fig.5 expansion of Fig.4

☆ ^{13}C satellites
⇩ sample signal overlapped with ^{13}C satellites

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