Spectral Separation of mixtures by solid state NMR

Product used : Nuclear Magnetic Resonance (NMR)

Fig.1 show ^{13}C NMR spectra of mixture of L-Histidine and L-Histidine Hydrochloride Monohydrate in solution and solid state.
Only a single state is observed in solution state NMR, as a contrast, individual crystal state can be distinguished in solid state NMR.
Solid state NMR is a powerful tool to obtain information in the crystalline state which would be lost in the solution state.

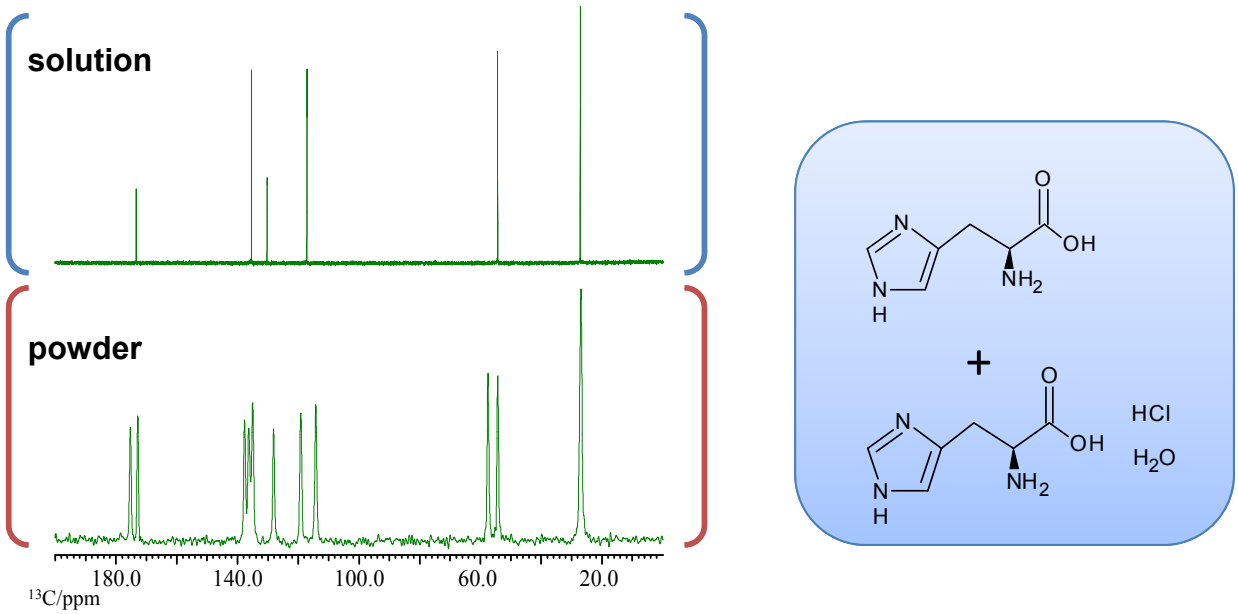

Fig.1 upper : ^{13}C solution NMR spectrum (10mg of each sample solved in D_2O)
lower : ^{13}C CPMAS spectrum of the mixture

ROSY is a method which provides a separate ^{13}C CPMAS spectra from the mixture. Fig.2 shows the spectral separation of each component by utilizing the different ^1H T_1 value of L-Histidine and L-Histidine Hydrochloride Monohydrate by ROSY method. Thus, ROSY is clearly useful tool for analysis of mixtures.

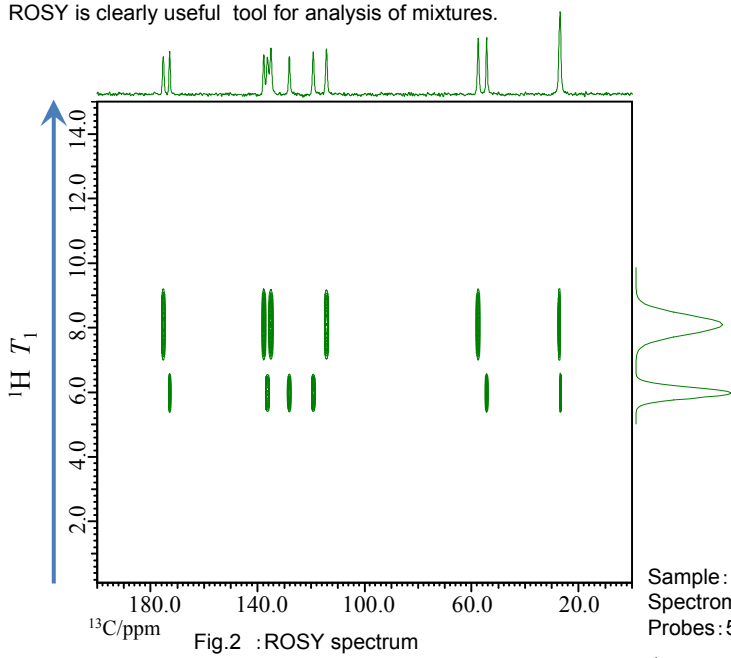

Fig.2 : ROSY spectrum

ROSY method

ROSY(**R**elaxation **O**rdered **S**pectroscop**Y**)[1] is a spectral separation method using the difference of ^1H T_1 value of each components in the mixture.
Ordinally, each compound has unique ^1H T_1 value in solid state NMR. Therefore when each compound has significantly different ^1H T_1 value, ROSY method can be applied.

Tips of ROSY

Sometimes, ROSY method fails to separate each component when ^1H T_1 value in a single component becomes not unique by ultra fast MAS. Therefore MAS speed around 10 kHz is recommended for ROSY, and TOSS is effective to suppress SSB.

Sample : mixture of L-Histidine and L-Histidine Hydrochloride Monohydrate
Spectrometer : JNM-ECZ500R
Probes : 5mmSuperCOOL(solution), 3.2mm HXMAS(solid)

[1] Y. Nishiyama, M.H. Frey, S. Mukasa, H. Utsumi, J. Magn. Reson. 202(2010) 135.

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