

Introduction of Solid-state NMR

Solid-state NMR is useful to ...

- 1) Determine chemical structures for insoluble materials or samples which want avoiding to dissolve in a solvent.
- 2) Evaluate molecular mobility in the solid state.
- 3) Obtain the detail chemical information for observed element selectively.

Measurable elements

^{13}C , ^{29}Si , ^{19}F , ^7Li are nuclei measured frequently, and detailed structural analysis is possible from our abundant databases. From ^{31}P , ^{27}Al , ^{11}B , ^6Li , ^{15}N , ^2H nuclei, it is also possible to obtain useful information. For rubber or gel samples, useful information can also be acquired from ^1H nucleus.

Available information

1) About Chemical Structure

Chemical species, coordination number, primary structure, secondary structure (conformation), high-order structure (crystalline and amorphous).

2) About Molecular Mobility

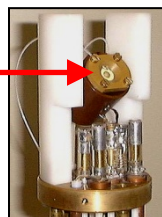
Crystallinity, orientation, crosslinking, domain size of polymer alloy (the order of several tens nm or several nm), interaction of active pharmaceutical ingredients or polymers with water or solvents.

How do measurements?

The sample tube filled up with a sample is installed into a probe, and carried out high speed spinning in the superconducting magnet for measurements.

Sample (powder, fiber, cutting film, gel, etc.)

Sample tube

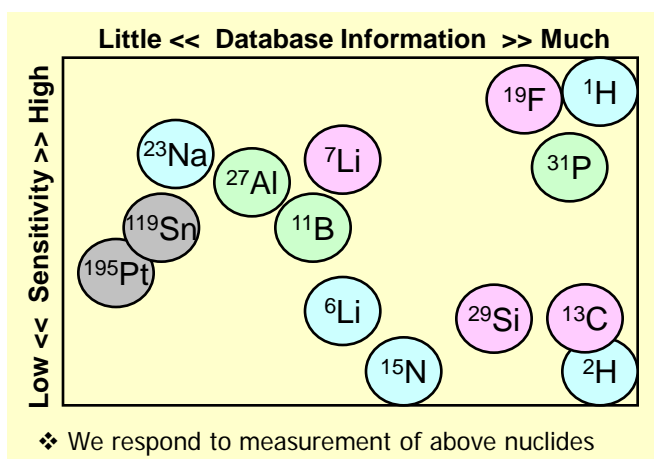


Probe inside



Superconducting magnet

- ❖ Required sample volume is several mg - several hundred mg.
(When the sample amount is sufficient, quantify measurement is possible.)

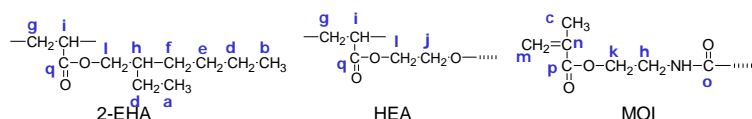


Determining component ratio of polymers.

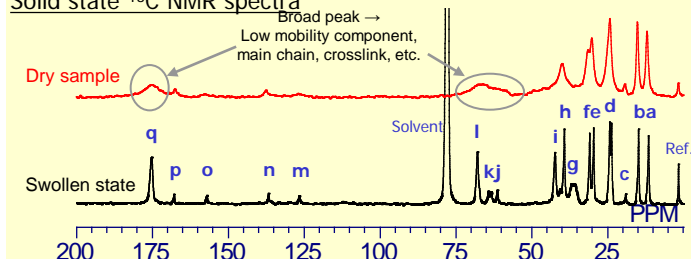
Chemical structure information can be acquired from the peak position (chemical shift value), and the quantity the ratio of each component in the observed elements from the peak area.

Component ratio of adhesive

To determine the composition ratio of three main components of the UV curable adhesive, solid-state ^{13}C NMR measurements were performed.



Solid state ^{13}C NMR spectra



component	2-EHA	HEA	MOI
ratio	71 mol%	19 mol%	10 mol%

In the case of the resin of which monomer composition is unknown, it is effective to carry out IR, pyrolysis GC/MS, and organic analysis of the soluble component in conjunction.

With measuring at high temperature near the melting point or in the swollen state, it is possible to obtain higher resolution spectrum and the accuracy of analysis is improved. we have been carrying out a lot of such high temperature or swelling measurement.

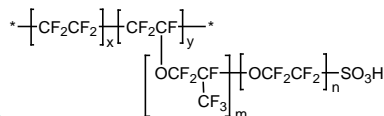
Chemical structure of electrolyte membranes

In order to determine the chain structure for the fluorinated electrolyte membrane in the polymer electrolyte fuel cell, solid-state ^{19}F NMR measurements was effectual.

A difference of the chemical structure of the side chain was captured by ^{19}F NMR spectrum for two samples. It is possible to calculate the EW (Equivalent Weight) from the estimated unit ratio summarized in the following table.

	x	y	m	n
NRE212	6.6	1.0	1.1	1.3
Short side chain type	5.1	1.0	—	0.9

【Nafion® NRE212】



【Short side chain type】

