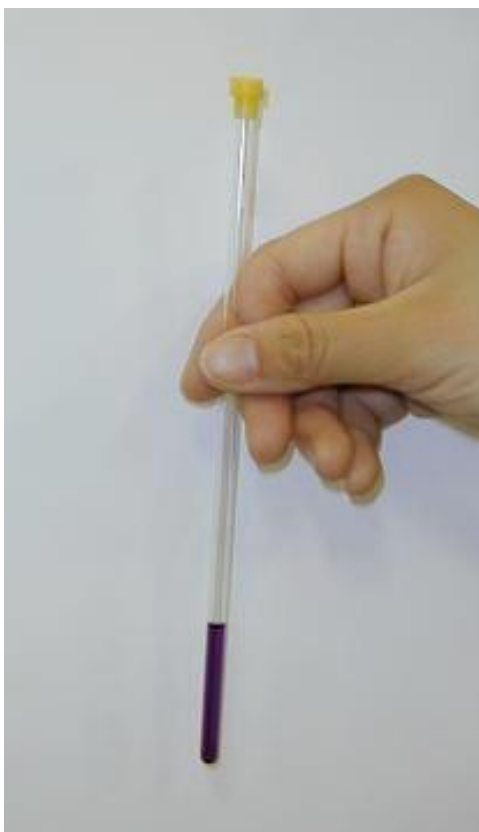




Solution NMR Sample Tube
($\varnothing$ 5 mm O.D.)

Volume: Ca. 0.5 ml

**Sample should be dissolved in
deuterated solvent in principle.**

***Common deuterated solvents**

Chloroform-*d* (CDCl_3)

Acetone-*d*₆

DMSO-*d*₆

Methyl Alcohol-*d*₄ (CD_3OD)

Deuterium Oxide (D_2O)

Sample Preparation

- **Dissolve Sample Homogeneously**
 - ✓ **A totally homogeneous concentration is required.**
 - ✓ **An appropriate concentration is required.**
- **Remove Insoluble**
 - ✓ **A floating materials in solution should not be exist.**
- **Remove (Beware) Impurities**
 - ✓ **Avoid signal line broadening due to paramagnetic impurities (organic radicals, metallic ions).**
 - ✓ **Avoid influence of water or acid in solvent.**
- **Select Purposeful Sample tube**

Important Points of NMR Sample Preparation

**“Homogeneous”
concentration**

**“Inhomogeneous”
concentration**



**An Inhomogeneous concentration
causes local magnetic susceptibility
distortion, and make shimming
impossible.**



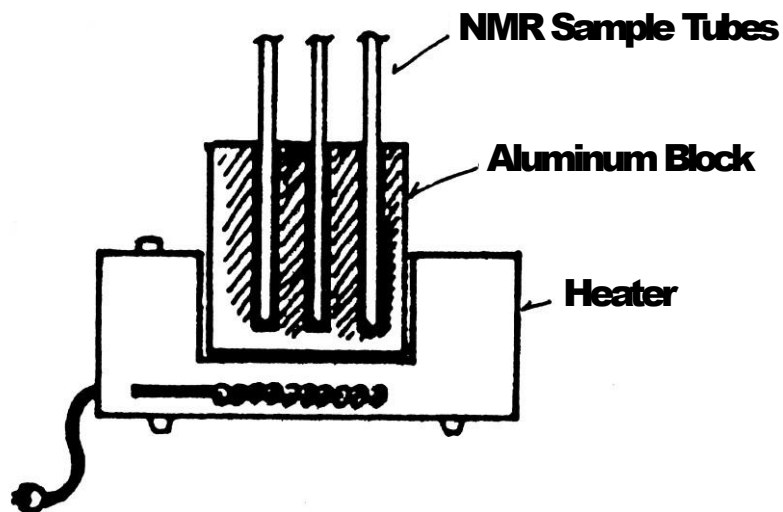
**Signal line broadening
Abnormal signal splitting**

Locally distorted magnetic susceptibility

Homogenous magnetic susceptibility

Dissolve Sample Homogeneously

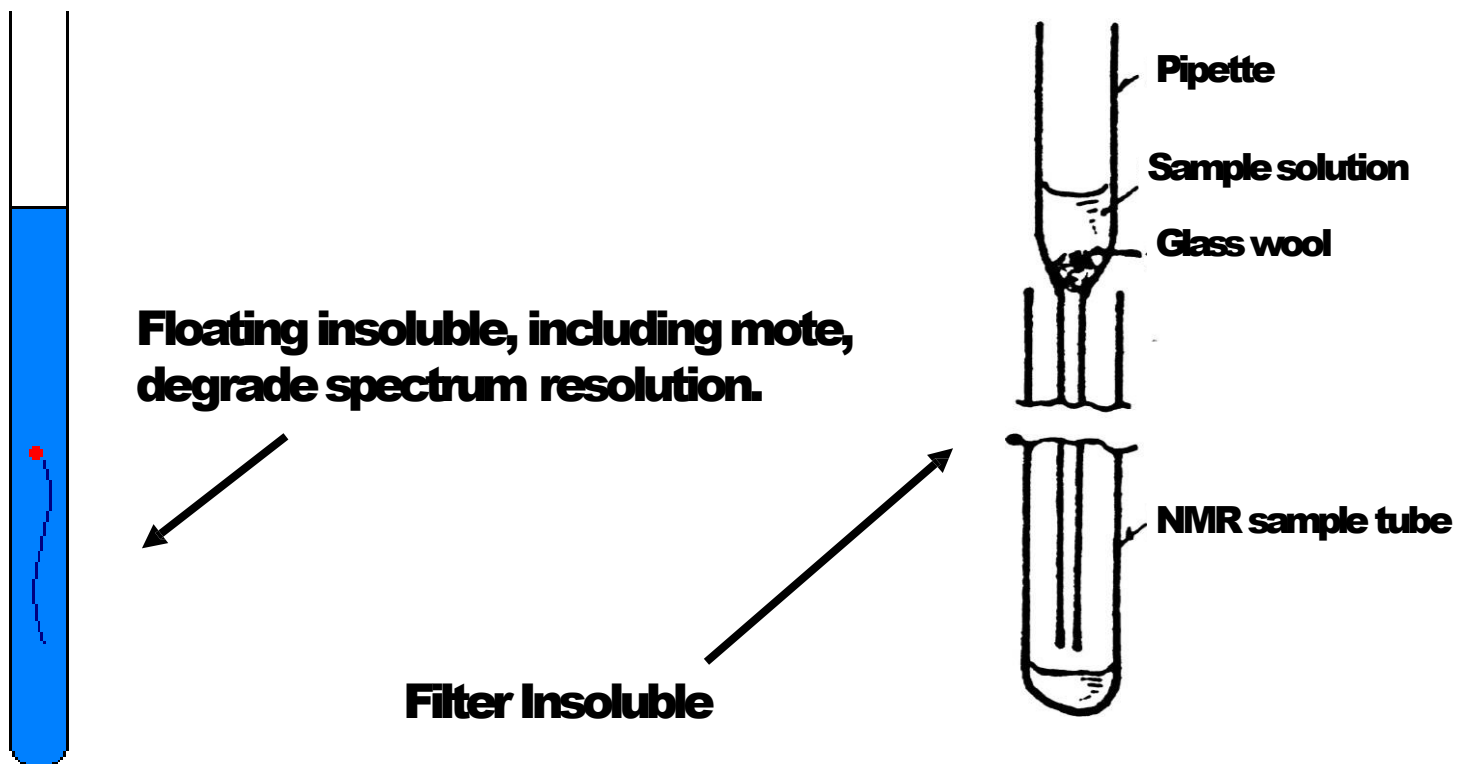
**It is difficult to homogenize concentration due to high viscosity usually.
In case of high resolution required, homogenize concentration carefully
using long term diffusion.**



Convection flow with long term heating is effective.

***Replacing Nitrogen gas to avoid oxidation is recommended.**

Notification for Polymer Solution



Remove Insoluble

Paramagnetic Impurities (* dissolved oxygen is paramagnetic!)

- ✓ **Paramagnetic component such as metallic ion or organic radicals broaden NMR signals, let alone high concentration of those causes signal disappear and make NMR lock impossible.**

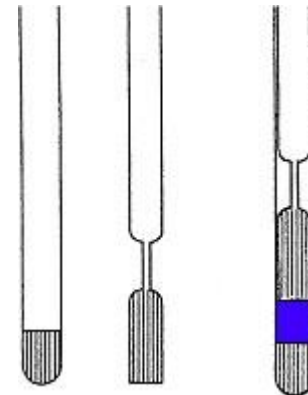
Impurities of solvent

- ✓ **A chloroform produces a hydrochloric acid as a photodecomposition product during storage. Thus it should be stored in a cool, dark place. A hydrochloric acid has to be removed before dissolving especially for an acid sensitive sample.**
- ✓ **Carefully handle pipetting to avoid contamination of solvent.**
- ✓ **Trace amount of water easily contaminate from used pipette.**

Remove (Beware) Impurities

Special Sample Tubes

- **Quartz Tube, PTFE Tube**
Background signals of ^{29}Si or ^{11}B from sample tube will not be obtained.
(*Materials of a probe may be obtained)
- **Shigemi Symmetrical NMR Microtube**
Reducing resolution degradation due to insufficient sample solution volume.
- **Special Use**
High pressure sapphire cell, etc..



The magnetic susceptibility matched symmetrical NMR microtube assembly by Shigemi Co., Ltd

*** Disposable Tubes**

“Cheap” NMR tube may have distortion, and degrade spectrum resolution.

NMR sample tubes

Clean away factors to degrade resolution.

Scratch

!! Do not scrub tubes with a brush.

Distortion

!! Do not dry tubes in a laboratory oven.

Dust

!! Cap it to keep out dusts.

Cleaning, Drying, and Storing of Tubes