Title: NMR Manual for Paramagnetic Susceptibility Measurement (Evans Method)

Purpose

This Evans Method manual will help NMR users understand paramagnetic susceptibility measurement by using NMR. Users can simply follow the procedures to conduct their experiments with little or even no NMR staff assistance

Introduction

Evans Method was established in 1959 for magnetic susceptibility measurement [Ref 1]. It was developed by many other researchers in the past half century. Now, the most common equation is

$\chi_{mass} = 3\Delta f/4\pi fm + \chi_0 + \chi_0(d_0 - d_s)/m$ (1) [Ref 2]

- χ_{mass} is mass Susceptibility in cm^3g^{-1}
- Δf is obs freq diff in Hz
- f is spectrometer freq, e.g. $399.732x10^6$ Hz on Hg400
- m is mass of paramagnetic substance in $g{\cdot}cm^{-3}$ i.e. concentration in g/mL
- χ_0 is mass susceptibility of solvent in cm^3g^{-1}
- d_0 is density of solvent in $g{\cdot}cm^{-3}$
- d_s is density of solution in $g{\cdot}cm^{-3}$

There are some other related concepts: [Ref 3]

volume Susceptibility (χ_v) $\chi_v = \rho\chi_{mass}$ here ρ is the density in $kg{\cdot}cm^{-3}$ or $g{\cdot}cm^{-3}$ and

molar Susceptibility (χ_{mol}) $\chi_{mol} = M\chi_{mass}$ here M is molecular weight

Also, understand Conversion of SI and cgs units: $\chi^{SI} = 4\pi\chi^{cgs}$

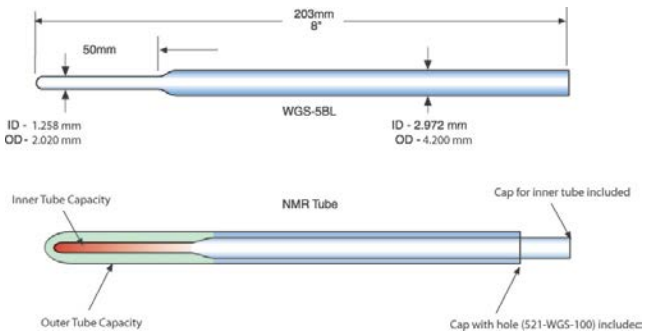
In this manual, we use cgs unit through-out. For instance, χ_{mol} in cm^3mol^{-1}

Preparation

Prerequisite: users have done the basic NMR training

Spectrometer: Any, but NMR-Hg400 recommended

NMR tube: coaxial inserts (see the picture below) or capillary inserts [Ref 4]



NMR Sample Prep: (using FeSO4, t-butanol and D2O as example [Ref 5])

- Solution A: dissolve 7.0 mg FeSO4.7H2O in 0.5 mL D2O, add 30 uL t-butanol, and adjust total volume to 1 mL with D2O
- Solution B: 30 uL t-butanol + 970 uL D2O
- Transfer A into the coaxial insert and B into a 5mm tube

Reading: please see the **References** section at the end of this manual

Experiment Setup

1) Login on FOM system, then, login on spectrometer computer by netid
2) Insert NMR sample into magnet and do locking and shimming as usual
3) Tune H1 (optional)
4) Run a regular 1D H1 (will see two methyl peaks caused by paramagnetic reagent)
5) Save data in a correct folder

Ending Work

1) Switch a dummy sample into the magnet
2) Lock on CDCl3
3) Write a note on Log Book
4) Keep desk top clean
5) Logout from spectrometer computer and FOM

Data analysis

1) measure distance between two methyl peaks (Δf 510 Hz)
2) use a simplified equation $\chi_{mass} = 3\Delta f/4\pi fm + \chi_0$ (2)
   Here, f = 399.732 on Hg400
   m = 0.0072 g (actual weight) in 1.00 mL (solution A)
   χ_0 is approximately equal to water mass susceptibility, -0.72×10^{-6} cm^3/g
3) molar susceptibility $\chi_{mol} = M\chi_{mass}$
   Here, molar mass M is 278 for FeSO4, and $\chi_{mol} \sim 11200 \times 10^{-6}$ [Ref. 6]
4) Actual experimental result is $\chi_{mass} = 41.58 \times 10^{-6}$ cm^3/g and $\chi_{mol} = 11600 \times 10^{-6}$ cm^3/mol
5) If the χ_{mol} value is small, you may consider the diamagnetic susceptibility correction term $\chi_{mol\text{-}para} = \chi_{mol} + \chi_{mol\text{-}dia}$ [ref 7]

References

1) D.F. Evans, J. Chem. Soc. 2003 (1959)
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3) http://en.wikipedia.org/wiki/Magnetic_susceptibility
4) http://www.wilmad-labglass.com/Support/NMR-and-EPR-Technical-Reports/NMR-007--Coaxial-Inserts-in-NMR-Studies/
5) 200 and More NMR Experiments. Stefan Berger and Siegmar Braun (2004)
6) http://www-d0.fnal.gov/hardware/cal/lvps_info/engineering/elementmagn.pdf
7) http://pubs.acs.org/doi/pdf/10.1021/ed085p532