Solvents, NMR Tubes, and Susceptibility Matched Plugs Sets

Courtesy of Charlie Fry, U Wisc-Madison and adapted for our facility

Solvents

The deuterated stockroom solvents are fine for all nominal experiments. See Aldrich or CIL for specialized deuterated solvents.

Achieving optimal line shapes in an NMR experiment depends critically on the amount of solvent used. The recommendation is:

Varian 5mm probes	0.7 ml solvent
Bruker 5mm probes	0.5 ml solvent

In general, what is needed is for the solvent column to extend the ***rf coil length*** both above and below the coil (i.e., total height = 3 times the coil length). Different probes have different coil lengths, so there is no universal guideline to apply. The above-recommended volumes are derived as follows:

Varian 5mm coil length = 16 mm $\rightarrow$ 48 mm recommended solvent length = 650 µl.
Bruker 5mm coil length = 12 mm $\rightarrow$ 36 mm recommended solvent length = 500 µl.

For variable-temperature experiments, when using expensive solvents, and to maximize concentration, minimum solvent volume is preferred. Empirically, we find that using less solvent than the above-recommended volumes can safely be done, but only within certain limitations, and with a price to be paid of increasing the shimming effort needed to achieve a desired linewidth. Going less than 0.45 ml without susceptibility plugs (see below) on a Varian 5mm probe is almost certain to be fatal to achieving reasonable line shapes. Similarly, going to less than 0.35 ml on a Bruker 5mm will almost certainly prevent optimal line shapes from being achieved. [*Note: Facility experiments find very similar sensitivities with both vendor's probes for identical sample amount—e.g., ^{13}C of 10mg sucrose in $CDCl_3$. Thus, Varian probes are not less sensitive because the concentration is lower; the longer coil makes up for the lower concentration.*] Since sensitivity decreases with degraded line shape, ***the user should never push solvent volume too low for sensitivity reasons (i.e., to maximize concentration)***.

Shigemi tubes allow the solvent volume to be reduced to 1/3 that stated above by removing the susceptibility gradients normally occurring at the solvent-air interface. Shigemi tubes have been successfully used in our facility for ^{1}H and ^{13}C experiments. In these experiments, we have always seen the expected factor of 3^2 (=9) improvement in experiment time for identical S/N. Thus, an overnight experiment in a normal tube gives the same S/N as a 1.5 h experiment with a Shigemi tube! **USE SHIGEMI TUBES WHEN SAMPLE AMOUNT IS LIMITED!!!**

Note that when using Shigemi tubes, the solvent volume should equal the length of the rf coil. The facility has Shigemi tubes available for loan to members of the chemistry department.

Wilmad NMR tubes (800-229-5171 or www.wilmad.com):

for 200/300 MHz work:

recommended	507-PP-8	$6.64	(current stock room tubes)

for 300 MHz work:

recommended	507-PP-8	$6.64	(current stock room tubes)

for 500 MHz work:

routine work	507-PP-8	$6.64	(do see some Z^3/Z^4 trouble)
recommended	528-PP-7	$9.17	(current stock room tubes)
best	535-PP-8	$16.65	(for solvent suppression)
best(er)	541-PP-8	$27.50	(for 800 work?)
best(erest)	542-PP-8	$37.50	(presume highest field only)

The tubes come in 7", 8" and 9" versions. 8" and 9" versions allow tubes to be flame sealed and still be long enough in spinners.

See Wilmad's NMR-010 technical note at their web site for suggestions on how to clean and dry tubes.

Other vendors sell similar products; I cannot comment on relative quality of the other vendors, but know the Wilmad tubes give consistently good results.

Susceptibility Inserts (Shigemi):

Shigemi Susceptibility Tubes Sets (724-444-3011) or www.geocities.com/~shigemi

Shigemi tube sets are generally regarded as optimum for precision/best quality work (e.g., when needing water suppression). The disadvantages are slightly higher cost (glass is more chemically durable, however), and susceptibility matching to the solvents shown below.

$CDCl_3$:	CMS-005V	$75.00 per set
CD_3OD:	MMS-005V	$75.00 per set
DMSO:	DMS-005V	$85.00 per set
D_2O:	BMS-005V	$75.00 per set

Susceptibility Inserts (Wilmad):

Doty Susceptibility Plugs from Wilmad (http://www.wilmad.com/html/nf/DotyPlugs.html)

These inserts are polymeric (except Zirconia), and thus chemical compatibility with solvent/solute combination must be taken into account for their use. The 1st line following the price indicates the susceptibility match to the solvent; the 2nd line indicates the chemical compatibility with the solvent and solute combination.

5mm kel-f positioning rod	$24.00
5mm sealing clamp	$62.00

Aurum plug set $72.00
susceptibility match to D2O/water (as solvents)
excellent chem compatibility with alcohols, aliphatics, aromatic H-C, esters, ketones

Glass filled PEEK set $40.00
susceptibility match to methanol, MEK, ethyl ether (as solvents)
excellent chem compatibility with alcohols, aliphatics, aromatic H-C, esters, ketones

G-10 plug set $30.00
susceptibility match to acetone, MEK, methanol (as solvents)
excellent chem compatibility with strong bases, alcohols, aliphatics, aromatic H-C, esters, ketones

Kel-f plug set $28.00
susceptibility match to glycerol (as solvents)
excellent chem compatibility with strong acids and bases, alcohols, aliphatics, aromatic H-C, esters, ketones

PPS plug set $50.00
susceptibility match to $CDCl_3$, water (as solvents)
good chem. compatibility with strong acids and excellent compatibility with strong bases, alcohols, aliphatics, aromatic H-C, esters, ketones

Ultem plug set $24.00
susceptibility match to D_2O, water (as solvents)
excellent chem. compatibility with alcohols, aliphatics, esters, ketones

Zirconia plug set $240.00
susceptibility match to D_2O, CCl_4, DMSO, benzene (as solvents)
excellent chem. compatibility with strong acids and bases, alcohols, aliphatics, aromatic H-C, esters, ketones