"Design of a simple and robust NMR-tube cleaner"

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There are two basic designs for NMR-tube cleaners, classic and reverse flow (since the flow of washing solvent is opposite along the NMR tube with respect to the former). In the classic design, the NMR tube is (part) enclosed in a space that is to be evacuated and solvent is delivered into the tube upon evacuation *via* an insert which is fed by a solvent reservoir. In the reverse-flow design, the insert is attached to a vacuum source and the opening of the inverted NMR tube dips directly into the solvent reservoir, hence engaging the vacuum source draws the solvent up into the NMR tube which then exits *via* the insert. Both designs have their advantages and inherent flaws.

For the classic design, spilling of the NMR-tube contents into the apparatus when inverting the tube is not problematic as the contents are moving in their intended direction (a limited amount entering the insert is inconsequential as it is subsequently easily flushed). In the reverse-flow design, the contents are spilling into the solvent reservoir thus contaminating the wash solvent. Similarly, contamination occurs if there is material on the outside of the NMR tube, which is normally the case at the top where the NMR-tube cap has been in contact with the NMR tube. This is one drawback of the reverse-flow design and some care is thus required during use though spillage can be reasonably well flushed away if the NMR tube reaches close to the bottom of the reservoir. However, this then demands that the lengths of the NMR tubes are all consistent. An adjustable length system for the apparatus brings its own problems and generates extra burden in any case for its use and, furthermore, makes no allowance for chipped–but perfectly good otherwise–NMR tubes. The unavoidable residue of solvent in the reservoir of the reverse-flow design means that switching of wash solvents is also problematic (*e.g.* polar solvent $\leftrightarrow$ organic, rough washing solvent $\rightarrow$ final rinse with high grade solvent).

In the usual layout of the classic design, the evacuation route is typically longitudinal whilst the insert feed is lateral. The bend in the insert line at the base where it penetrates the sleeve wall is a weak point excessively prone to breaking when made of glass (most commonly the case). The experience has been that domestically manufactured tube cleaners are repaired every 2–3 years or less. (It is worth nothing that some constructions have the orientation of the evacuation and feed lines switched.)

Some aspects are equal between the two designs. Both usually require one hand to be in contact with the apparatus during operation: in the classic design this is to seal the system airtight during washing by placing one's finger over the end of the glass sleeve (though this is redundant if using an attachment or

cap, as in the current apparatus, to effect a seal); in the reverse-flow design it may be required to lift the NMR tube up somewhat so that solvent may flow into the insert unimpeded. For equal diameters of the insert tubes, from casual observation it appears that the vigorousness of the solvent flow, *i.e.* the available mechanical agitation, is essentially the same for the two designs.

The considerable advantage of the reverse-flow design is the ease of its construction and the ready use of materials other than glass. Thus, to maintain the beneficial features of the classic design but to diminish its limitations, we have altered the design to enable a moderately easy method of fabrication from simple materials. For example, from a cylindrical block of Teflon only five separate drillings are required, and the whole process is self-evident from the schematic diagram of the apparatus presented below.
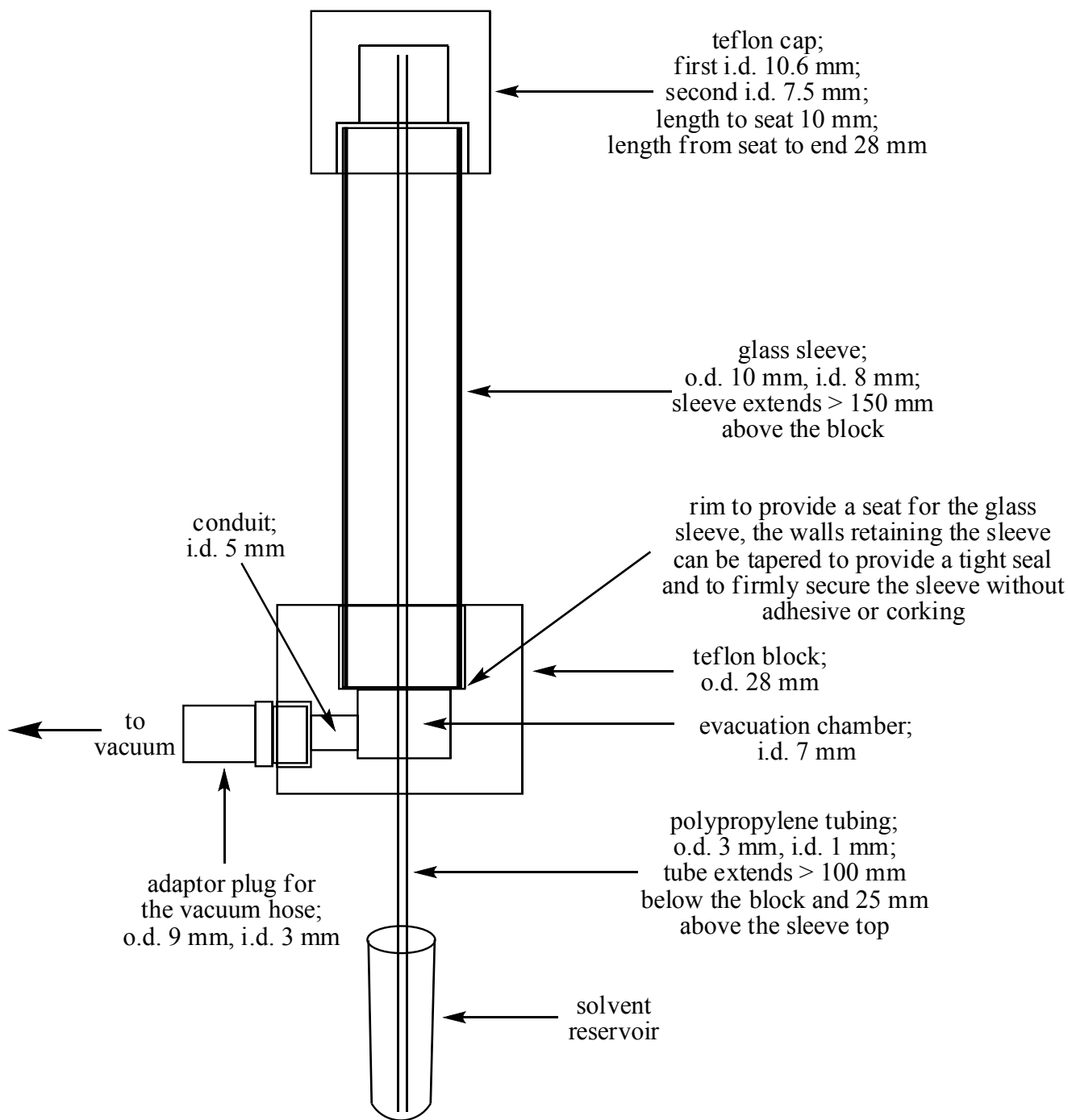
Notes and options:

1) One consideration was to have the sleeve close-ended so that it could also function as a cap, but this would preclude the ability to rinse the outside of the NMR tube by the introduction of solvent at the sleeve top opening whilst it is on the apparatus. The requirement for an a cap or not, in the latter case the insert does not extend past the end of the sleeve and sealing is effected by one's finger or by an attachment (*e.g.* an NMR-tube cap is often used) to the end of the NMR tube which both effects a seal and enables withdrawal of the NMR tube after washing is completed, is arbitrary as both accommodate rinsing of the outside of the NMR tube. To retrieve the NMR tube when the end of the insert lies below the top of sleeve and an attachment has not been used, a simple extractor is used consisting of a hollow or part hollow Teflon piece (i.d. 5 mm, wall thickness 0.6 mm) which grips the NMR tube when pressed gently against the end and thus enables it to be drawn back out of the apparatus.

2) Stainless steel tubing can be substituted for the polypropylene tubing (*e.g.* see the reverse-flow design in the pictures below), though this runs the risk of scratching of the inside of the NMR tube.

3) The glass sleeve can be made of other materials if desired, *e.g.* stainless steel or Teflon, but our preference was to be able to view the progress of the washing.

4) Attachment of longer tubing below the apparatus leading off to the reservoir provides a means to quickly change between vessels containing different wash solvents, possible also by the insertion of a directional tap for switching between a number of reservoirs, though we still prefer the use of wash bottles for this purpose. Dispensing the solvent into the reservoir by wash bottle during the washing process also permits the introduction of air which seems to add to the turbulence hence heightening the mixing and mechanical agitation.

5) There is a lot of latitude in the listed measurements of the schematic and commonsense mandates which values need to follow which. A longer rather than shorter glass sleeve is recommended, not only to accommodate very long tubes but also to limit the amount of backsplash from the bottom of the tube cleaner onto the outside of the NMR tube.

teflon cap;
first i.d. 10.6 mm;
second i.d. 7.5 mm;
length to seat 10 mm;
length from seat to end 28 mm

glass sleeve;
o.d. 10 mm, i.d. 8 mm;
sleeve extends > 150 mm
above the block

conduit;
i.d. 5 mm

rim to provide a seat for the glass
sleeve, the walls retaining the sleeve
can be tapered to provide a tight seal
and to firmly secure the sleeve without
adhesive or corking

teflon block;
o.d. 28 mm

to
vacuum

evacuation chamber;
i.d. 7 mm

adaptor plug for
the vacuum hose;
o.d. 9 mm, i.d. 3 mm

polypropylene tubing;
o.d. 3 mm, i.d. 1 mm;
tube extends > 100 mm
below the block and 25 mm
above the sleeve top

solvent
reservoir

Schematic of the design.

Below, pictures of the NMR-tube cleaner. Present in the left of the pictures is a reverse-flow NMR-tube cleaner.