SHUTTLE DEVICE FOR USE IN A SHARED COMMERCIAL NMR INSTRUMENT VERSION II

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This is a detailed description of our device as modified by early 2004. The first part of this report was mostly completed in the summer of 2004 and the second part was completed Dec. 2004. Some late news is summarized at the end. Work on this device was started in 2000, and described initially in a series of reports posted on my Brandeis website within months of developments. A fairly complete description of Version I was written in early 2003 and was kindly accepted as a contribution to a special issue of Magnetic Resonance in Chemistry devoted to relaxation, by the Editor of that issue, Professor Josef Kowalewski [Magnetic Resonance in Chemistry 41:753-768]. Subsequently we published 3 research articles: (with M. Roberts, C. Qui, C. J. Turner, & D. Case) Biochemistry 43:3637-3650 (2004); (with M. F. Roberts) JACS 126:13765-13777, and PNAS 101:17066-17071 (both 2004). The first of these showed the general utility, using a DNA duplex octamer, of field cycling ^{31}P relaxation to evaluate sizes of CSA vs dipolar interactions and correlation times; the last two are on membrane dynamics and structure by ^{31}P relaxatuion. We have also presented 2D ^{15}N T_1 relaxation data on cyclophilin A, down to 3.5 Tesla, at meetings (with Elan Eisenmesser and Dorothee Kern, unpublished). The latter are in good agreement with standard theory.

The figures are in the separate PDF file that is also posted, so that you can print some of them for study, and to avoid having this text file slow to read and print.

These reports are published on the web and in print in the hope that they will facilitate use of this important method in other labs. I will be happy to help non-profit labs reproduce this device by answering questions, within limits. However, this is not a stand-alone report, it is a continuation of, and supplement to, my article in Magnetic Res. In Chem ("MRC"). I will assume you have thoroughly read my article, as well as this report, and if you have not I may loose patience when you ask many questions already answered in them. If you ask intelligent questions I will try to answer as best I can, and would like to know what mistakes I have made or what is really unclear.

As usual, Frank Mello, the Director of the Brandeis Machine shop, made many useful suggestions and carefully built these components.

This report is posted on the web because version II described below is more straightforward to make than version I, and because MRC did not have drawings and other information useful for actually building and using the device. It is not much faster or otherwise better than version I. Version III is now being planned and will be described in about a year if it works well, and otherwise will be forgotten.

Besides reading the MRC article fairly carefully, you should skim the present report a few times. I do not provide a table of contents, but you should realize that the report starts from the smallest most inner part (the sample) and works out to the shuttle tube, then up the stop tube and over to the things on the remote table. So look at Fig. 2 of MRC carefully and then, after skimming this report, go back and study the assembly diagrams. These are Fig. SH1, the shuttle and sample holder, and Figs ST1 to ST3. The drawings are copied from working drawings, but certainly have errors. You must understand these drawings in any case, especially if you have to modify the device for a Bruker or other spectrometer. Please tell me about errors. Our system uses a standard Oxford magnet installed by Bruker about 10 years ago and retrofitted by Varian with their room temp. shim coils. You use these drawings and information at your own risk. Please supply me with drawings of modifications needed for other systems such as Bruker, or any other useful information.

I apologize for use of English units almost everywhere, but conversion to metric would probably have introduced errors. Some readers will dislike the quality of the drawings , and anyone who makes better ones could send them to me. Most of these drawings have been redrawn by me from working drawings used by Frank Mello and I think they are easily understood by someone familiar with the conventions of mechanical drawing.

Many scientists will have trouble understanding this report and I strongly advise them to find a physicist or chemical physicist as well as a good machinist, to help them after they have initially skim this report and tried hard to understand it. Then they could ask their physical mentor to quickly go over these drawings and explain what I intended to convey. The same applies to understanding the very trivial electronics that I describe in the second part, aided by an electronics professional.

The version I system and parts of version II were largely financed by a grant from the NSF Chem. Instrument Program. Some of the present design improvements were financed by my current grant from the Petroleum Research Fund. Early essential support came from my former NIH grant, and from the Rosensteil Center at Brandeis. I contributed much free labor supported by TIAA CREF. Therefore I will be extra appreciative if researchers will kindly acknowledge use of these designs in papers that first utilize them in a useful way.

The earlier reports that I posted will soon be removed or compressed into a brief summary.

Breakage. I have not broken a probe during a run except through stupidity. We probably have used the 10 mm probe about 40 days (24 hrs.) or more, and the 5 mm probe 20 days. Recently we have had 2 or 3 five mm tubes break at the extreme lower end without destroying the 10 mm probe. (that is an advantage of using this probe). Possible reasons are: something was not right in a complicated version of the stop assemblies and the shuttle was irregularly hit on some edge; or it was because I was not using any lower susceptibility plug, just filling the lower end with excess sample that we had, to save the small amount of work to put in a lower plug. We will now always pour in this plug with epoxy, and I have enlarged the lower hole (part (3) on fig ST 4B.
Through stupidity I broke a piece of plastic inside the 10 mm probe at the bottom without breaking the coil insert glasswear, costing about $2500 for Varian to repair. It was because I had the NMR tube much too low, and this is now nearly impossible. I broke the long glass shuttle tube once by not pulling the stop tube straight, when its end had a longer diameter machined into with tighter tolerance.

Of course I test off-line with no probe slid onto the bottom, and with both 5- and 8 mm tubes. I have not broken any tube during such tests (which are not lengthy), at the maximum pressure (about 0.2 bar vacuum, 0.5 bar higher pressure, 0.35 bar lower pressure with transit time nearly down to 100 msec transit time not counting bounces. I have had tubes break during non-tests (real runs) however, at the top where the tube enters the adapter, and in the middle also. By luck the probe did not break, perhaps because I then had a small hole, slightly smaller than the Varian 5 mm entry hole, at the bottom of the shuttle tube holder (see MRC). We stopped doing this because we have to remember to remove it when using 8 mm tubes in the 10 mm probe, but I should probably arrange to use it again. In one case I did not let the epoxy set long enough, that holds the tube to the adapter, and it failed. I now always wait overnight after applying epoxy.

Probes are as described in MRC.

Section SH Shuttle, NMR tube, and plugs

The shuttle etc design were changed from MRC to make the nmr tube and adapter shorter and the rest simpler to make. The main changes are: the nmr tube is shorter and the adapter is 1/2 in diameter instead of 3/8 (these things improve rigidity); it attaches to the shuttle body externally in a simpler way; the O-rings of the debounce system ride on the shuttle body; the shuttle is hollow so that the entire shuttle plus adapter plus tube now weighs 30 gm instead of 60. To allow the body to be hollow I dispensed with the extra assembly inside the shuttle body that acts as an extra shock absorber and find no tube breakage in several recent runs (8 mm). See fig SH-1.

This design has now (12/04) been used frequently with both 5 mm and 8 mm NMR tubes and I am confident that it is an improvement over version I.

The shuttle body consists of a simple tube, made as thin as is easy, about 3/4 OD and 5/8 ID drilled with a carbide drill, ends reasonably parallel and Id at the ends turned slightly larger to provide a fairly well aligned precise fit to be epoxied to the 2 end caps. The end caps carry the two precise bearing surfaces 0.798 OD that align the shuttle inside the 0.800 glass shuttle tube, and 1/2 inch extensions that hold the seven 1/2 x 3/4 inch Viton (fluorocarbon) O rings for shock absorption, on each end. The upper cap is drilled out to save weight and the bottom one (otherwise identical to the top one) mates to the NMR tube adapter. You need at least 2 shuttle bodies, the second one to use with the vee block while running if you want to seal a new sample.
I will return to the shuttle body and vee block later.

Sample loading See fig. SH-1.

The NMR tube design is similar to MRC but the tube is shorter, about 80 mm long. I plan to get 9 inch tubes from now on and have learned to cut them in two. Well, perhaps. (I will explain how later). I pipette about 0.1 ml of low-viscosity optical grade epoxy (Epotek 405 from Epoxy Technology, Billerica MA) into each 5 mm thin-wall tube a day or more before running, using a broken-off Wilmad long pipette. (about 0.25 ml of epoxy in the 8 mm tubes). I withdraw the pipette very rapidly to try to get as little epoxy on the walls. I spin the tubes in a small clinical centrifuge to get most of the epoxy off the walls and cure at room temperature for at least 3-4 hours before putting in the sample. I expect to use the upper half of each cut off tube to make a second tube by plugging the upper half with an epoxy plug-- by sealing off the good upper end with parawax and pouring epoxy as above in from the other end to form a sealed bottom about 5 mm or a little more long. I hope this works, the 8 mm tubes are expensive. (This is easy but I have not yet had the courage to use thse home-made tubes). I then cut off the NMR tube to the correct length son that it can later be epoxied into the adapter, assuming that the nmr sample will be 15 mm long and that the center of the sample will be 65 mm below the bottom end of the adapter and that about 12 mm is needed for the epoxy coupling to the adapter. Thus the NMR tube will be about 5+(15/2)+65+10=87.5 mm long, but this is varied depending on how long the poured epoxy plug at the bottom is, and it is longer for the 8 mm tubes because the bottom epoxy plug has to be longer. The main point is to try to make the center of the sample space (which is 15 mm long) be 65 mm below the lower end of the NMR tube adapter (Fig. SH-1). I designed the adapter (Fig. SH-5) so that there is excess length for the NMR tube to poke into, and so that it is not required to be very precise in cutting it off.

It is rather tedious, and risky to the tube, to clean off the epoxy as just described, and I have gone back to using a shield tube (see MRC) to avoid getting epoxy on the upper walls. *Details:* After the NMR tube has been shortened to the correct length (above), break off a long Wilmad NMR pipette so that its end reaches to the bottom of the tube at least. Cut off (or re-use) a piece of shield tube that slides over the end of the pipette, far enough to leave about 1 cm of the lower end of the pipette exposed. This tube has to be small enough in diameter to fit into the 5 mm tube. The only kind of tubing that I have found suitable is Intramedic-Clay-Adams Beckton Dickinson number 42745 polyethylene tube sold by several large chemistry supply houses, ID 3.17 mm, OD 3.99 mm. You can prepare at least two, probably four, tubes at a time using EPOTEK 305 (Epoxy Technology, Billerica MA) resin which sets very slowly (hours) and has low viscosity. Set up an ordinary 1 ml pipettor vertically, with its standard plastic pipette tube attached in a standard lab stand and clamp. Couple the glass pipette to the plastic pipette tip with a short piece of rubber tube. You will soon develop your own variations, but it seems best to have the end of the glass pipette about 3 cm above the base. Mix about 1 ml of epoxy per two NMR tubes in a short dispo test tube. The shield tube should now be slid up over the bottom of the glass pipette, with 1-2 cm of the glass end uncovered at the bottom. Suck 1 ml of epoxy into the glass tube in the usual way, but keep the tube containing the epoxy low enough so that epoxy does not touch or cover the shield tube. Release the top button of the pipettor slowly while sucking in the epoxy, to avoid excess vacuum. Do not worry about small bubbles that will appear in the epoxy. Now try to have a short (mm’s) length of air space at the bottom of the glass pipette, and wipe off the pipette and the shield tube thoroughly to remove excess epoxy. Now insert the glass pipette with shield into the NMR tube and push the pipettor button slowly to release the desired amount of epoxy, releasing close to the bottom of the epoxy but again trying not to have the epoxy touch the shield. I usually put in epoxy to a depth equal to the nmr tube diameter. Finally, grasp the top of the NMR tube in such a way as to keep the shield tube fixed relative to it, and pull up the glass tube so that its tip is just within the shield. Then shift to grasp the shield and glass pipette and pull the NMR tube off of both. This usually works; if not, little blobs of epoxy will be left on the upper part of the NMR tube. (Use a wooden stick or a small NMR tube inverted to try later to scrape them off so that the upper hollow susceptibility plug will slide in without excess force.) Now do another tube by first pushing the shield tube back up and wiping carefully.

I have previously prepared the hollow upper susceptibility plug by scraping the epoxy off its upper end, left over from the previous use, with a new single-edge razor blade, and perhaps also filing or sanding it gently. I rely on the fact that PEEK is much harder than epoxy so that it is only slightly worn by scraping; in fact I have yet to throw away any of these plastic pieces because they were too worn. I test it in the course of this cleaning to see that it will fit in an old NMR tube of the same size and then test that it will fit in the tube I plan to use. If not, because of epoxy left on the inside of the new tube, I clean this off first with a Fisher brand wooden stick and then with a smaller NMR tube stuck inside the larger one, i.e a 4 mm, scraping inverted using the top as a chisel, inside a 5 mm tube. And then wash out the epoxy pieces with water and ethanol).

The piece of wire at the upper end of the upper plug is now used as a spring to retard the plug from falling to the bottom during a spin. I bend it sideways carefully so as to do this but not break the tube, and test by putting it slowly and inverted in the junk NMR tube part way upside down. About 20 gram or more should be needed to move it. This is measured (not for every, tube but occasionally) by putting the plug with wire into the top of a spare NMR tube, and invert it with a wooden or glass rod stuck up into the top. Then I press the rod on to an electronic top-loader balance and push until the plug moves; the force it indicates when the plug moves should be around 100 times the weight of the plug. I bend a little hook on the end of this wire so that a curved surface slides along the glass.

One adapter (new design, fig. SH-5) has to be prepared for each sample to be sealed. If newly made it has to be de-greased with ethanol. If previously used the remains of the previous NMR tube is broken off crudely and as much removed from the lower end of the adapter with miniature pliers. Then I crudely break out the glass from the hole with the largest size drill that almost fits this hole, holding the drill bit with pliers and stopping when the drilling feels smoother, or using a good quality electric drill to turn it at low speed. I repeat this with a larger drill bit or two, and finally apply a 5 mm reamer. (I do not have an 8 mm reamer and use a drill in what follows for the 8 mm size.) I insert the reamer or final drill and turn it while exerting lateral force to scrape off the epoxy from the sides of the hole. I test by seeing if the bottom end of an nmr tube slides easily into the hole and is not pushed away from concentric by remaining pieces of epoxy in the hole. It should be possible to wiggle it enough to have the end of the NMR tube slide easily into the Vee-block adapter suitable for the NMR tube, when the tube inserted into the adaptor and connected to the shuttle are in the V-block flat on the bench.

The above steps take only ten minutes or less per sample, after practice. Before the next step you also have to locate the vee-block adapters and vee block (see below, and fig SH 2) and mentally go over what follows, which has to be done with moderate speed before the epoxy solidifies. The vee block should be ready with rubber bands to hold the shuttle assembly with the correct spacer held on in place with paper tape. Buy a bag of wide rubber bands. You have to set up the gas bag if needed (see MRC) and figure out how to use it (a pain). The upper plug should be washed in distilled water and dried with ethanol and in some cases soaked in buffer of some type suggested by the biochemistry (i. e. containing EDTA and/or DTT) and finally dried and placed ready to be loaded on a new kimwipe. You have to find a #16 syringe tip (Fisher, BD 16G 1 1/2) and a 5 or 10 cc syringe body and assemble them tightly ready on the bench, for transferring epoxy (a smaller syringe tip is inconvenient though possible. Order a box of the #16). Cut off the very sharp end of the syringe with small diagonal cutters for your safety. Find and set up the epoxy ready to mix (below).

If bubbles are of great concern I degas the sample as described in MRC in the gas bag, requiring about 1/2 hour using my slow shaker (MRC) under helium. Usually I do not do so for phospholipid vesicles for which the line-width is 50 Hz or more. I did try to degas epoxy but gave it up, though it could be done. See MRC for more on this.

I then pipette in the sample with a Wilmad long NMR pipette, in a gas bag if required, or after being chelexed if needed (for ^{31}P, if EDTA can't be used). About 270 microliters for the 5 mm tubes, 750 for the 8 mm. It is important not to put in too much sample. If you do, pipette enough out so that the sample length is now about 17-8 mm. The extra length (over the final 15 mm length) fills the space between the upper plug and the inner wall of the NMR tube. After this the sample is centrifuged ("spun") for a few minutes to avoid excess moisture on the walls of the glass especially at the top. A new pipe cleaner can also be used to dry the tube. Then check the height of the sample and adjust the volume as mentioned, if needed.

Now move to a window or other strong light and put in the upper plug in the tube with the wire up. It may be worth practicing what follows with an inexpensive colored sample like cytochrome C. Shove the plug down carefully til it hits the surface of the sample and then a very little further. There will be a large bubble at the top of the sample. In this and what follows the object is to get the top meniscus of the bubble-free aqueous sample a few millimeters below the groove at the top of the plug. (see MRC, the groove blocks the flow of aqueous solution and buffer across it so they do not mix) and *never* into the groove or especially into the space above the groove which is where the epoxy is supposed to go. (If this happens I generally spin to tube at the highest speed possible to get the aqueous sample back down). So look at the upper end of the plug to see that the meniscus of the sample does not get very close to this groove. Now spin the sample at high speed (typically position 3 on a clinical centrifuge) and the bubble will disappear. Now push the upper plug down a tiny bit more and spin again and repeat this zero to 2 more times. If you are especially worried about bubbles or have an oxygen-sensitive sample, do the pushing in the gas bag under helium, then put on the usual cap and spin outside. Finally spin several minutes at high speed until preferably the sample meniscus is a few mm below the groove on the plug. When this is achieved as well as seems possible, store the sample spinning at the lowest speed until the epoxy is ready.

I use a 1/2 to 1 hour slow setting household epoxy that comes in a double syringe, but throw away the stupid storage plugs they supply. I use small dispo-pipette tips instead, points shoved into the double-syringe holes. We store the double epoxy syringe with its tip up on the flat end of the dual syringe, and the syringe should be withdrawn after each use so that there is a small bubble on each side before the pipette tips are put in. When mixing, put a ml or so of epoxy in a small plastic weigh-boat. Be sure to put the pipe tips back in the same side that they were when you removed them, using their color or just keeping track or using new ones every time. I generally have not degassed the epoxy before use but that could be done. If bubbles may be a problem (for samples with small line-width) I stir the epoxy in the gas bag under helium.

I use the #16 syringe instead of the complicated procedure in MRC to transfer the epoxy to the top of the upper plug. I forcefully push the plunger up while the end of the tip is immersed in the newly mixed epoxy and get some epoxy to appear as a blob above the top end of the syringe tip, inside the syringe body. Then I apply it to the top of the upper plug, distributing it around the circumference to trap gas in the groove space at the top of the plug. Once this happens (normally very rapidly) I do not add much more epoxy. I used to spin momentarily to bring down the epoxy on the walls, usually by turning on the centrifuge at the lowest position for 15-30 sec only. If spun too long it might figure out how to sneak down into the groove. This is no longer required with the new short tubes, because you can get the end if the syringe right to the top of the plug. Finally, inspect carefully to verify that the epoxy is above the groove and the aqueous sample is below. If not, you could pull out the plug and throw away the NMR tube and start over (but I seldom do. In the rare cases where there seems to be a possibility of mixing, I usually just spin (see above) and charge ahead, hoping for the best.).

Now smear the remaining epoxy on the upper 10 mm or so of the outside of the glass tube and the inside of the adapter. You need enough to fill the space between the glass and the adapter, but excess will have to be wiped off and also it could get into the upper end of the hole and corrupt the thread there (this has not yet happened). Shove them together holding the right side up (tube to the bottom) until the center of the sample is 65 mm below the lower end of the adapter (or whatever distance you find is needed). Try to hold it there as you screw the adapter firmly (but only hand-tight) into the shuttle body. Doing this seals the top fairly tightly and if you get the sample in the wrong place, after screwing it on the body, the trapped gas resists correcting the position and you have to unscrew it partly to be able to move it to the desired position without force, and then re-tighten.

Finally I place the assembled parts in the vee-block, usually doing this with the vee block momentarily horizontal to facilitate putting on the rubber bands and getting the glass tube and body in the correct place approximately and not dropping anything on the floor (usually). The lower end of the glass tube should be even with the end of the vee block, and the 65 mm distance from sample center to the adapter bottom should be set using a ruler. Then I tip the V block on end and recheck these things and make sure that the above adjustments are correct, and that the glass, the vee block adapter, the nmr tube adapter, and the vee block are all pressed together by the rubber bands. A folded-up piece of paper towel can be placed between the rubber band and the NMR tube to push the tube in place more forcefully. Check again later that all is well. If my lab is cold, in winter, I do this in a warmer room. The assembly can be remove in about 3 hours if you need to seal another tube, but when we shuttled once the same day the epoxy failed. So we wait a day before using it. Also, after removing it from the rubber bands, you can check by feeling with fingers, or with a microscope (see MRC) to see if it fits in the vee-block + vee block adapter without excess space and independent of the angular position of the shuttle body. (for the 8 mm tube or a 5 mm tube to be used in the 10 mm probe this is not so necessary because of the > 1 mm clearance. In this case I use an old-model spare shuttle to get good enough alignment.).

Bubbles can form in the sample after running or after storage in the refrigerator. During running we keep the down-travel faster than the up travel (down-pressure greater than the up-suction) to inhibit bubble formation. This is not so serious a restriction as you may think, because a delay in the initial part of the up travel occurs where the sample is in high field and the T_1 is longer. Usually we keep the vacuum half as large as the pressure (both referenced to atmospheric pressure). We have to remember that the vacuum gauge dial is marked in inches of Hg, and divide by two.

Breaking the NMR tubes (?cleanly). I have a small rotary grinder (Dremel) mounted by a small vise, that turns a small circular diamond saw, with a piece of large aluminum I-beam that has a saw cut in it. These are mounted together to resemble a minature circular wood saw. You cannot use this to entirely cut the glass without a jagged result, but it is very useful for making a series of usually 3 cuts that do not go through the glass. Then with practice you can get a break that is only slightly jagged. These do no great harm since the adapter is drilled out with excess length, and the tube is pushed in to get the sample itself in the same place, as described. As long as there is more than a cm of un-cracked tube at the bottom end of the adapter to cement the tube in place, the jagtged ends gives no problem.

Storage. Bubbles tend to appear in the sample region during the night before we can run or after the sample is stored in the refrigerator. I do not know what gas they are or how they get there. I get rid of them by spinning the sample in either our small clinical centrifuge or if that does not work, in a large benchtop swinging bucket centrifuge of the type made by major manufacturers. Whatever you do, it should be a swinging bucket. The new short NMR tube and adapter will probably allow you to spin by just putting the tube+adapter in a small-hole centrifuge tube carrier with a counterbalance. If not, I have a thin-wall brass tube that fits into the centrifuge carrier and around the tube with the adapter. It is long enough so that when spinning it would swing down on the center part of the rotor. But to start the centrifuge I have to close the top on this brass tube. So I tie it down loosely with a cable tie (or any old piece of wire, but the tie is better, and can be left there permanently, un-noticed by the members of the owner's lab). (I did get her permission to do this however). The length of the brass tube has to be just right for this to work.

Vee block and vee block adapter. The vee block differs from the MRC version to make it easy to align various diameters. It is now a single block of (surplus) aluminum 3" square by 14" long (see fig SH-2). Great precautions were taken to mill it precisely and straight by clamping it directly on the movable base of the milling machine and using a precision 45 degree cutter in good condition. It was outside-milled square first and the outer edges were rounded to reduce breakage of the rubber bands. A narrower rough square mill in the center (where the tip of V-mill was going to be) allowed us to avoid having the tip of the V-mill do any cutting that could impair its accuracy. Since the block is always used with objects that are 0.8 inch in diameter it is only the part that would touch this diameter that matters. I worry that the aluminum will easily scratch. A brass or steel block, perhaps thinner and permanently screwed to a thicker block or I-beam to avoid bending, might be worth considering.

The vee block *adapter* is a simple precision-turned brass block that is precisely the same diameter as the glass tube inside (0.800 inch), and has a highly concentric inside hole reamed no more than .001 inch larger than the 5 mm NMR tube (fig. SH 2). It is then then cut in half in a plane parallel to its axis and offset by about .01 inch from it, and the slightly smaller half is carefully trimmed and used. (or in another model we milled a 5 mm wide groove from the outer surface toward the center so that a tube can be pushed in from the side. It is roughly held to the vee block with tape for cementing the NMR tube in place (rubber band are later used to hold it more firmly in place after very thing is assembled), Test alignment by feeling with your finger for play (to sense ~ 0.1 mm play) or use with a microscope (see MRC). Different adapters can be easily made and used to align-mount different round objects as precisely as possible. For an 8 mm NMR tube that fits in a 10 mm probe alignment is not much of a problem and we made the vee block adapter using the next larger available drill (US 21/64 inch) Note that an 8.00 mm reamer may be too small for an 8 mm Wilmad tube whose minimum dimension is 8 mm.

Design and fabrication. We do not use a separate lower susceptibility plug, use of epoxy (above) seems satisfactory but we do not know its magnetic susceptibility. NMR linewidths look ok for macromolecules. Contamination of the sample by something from the epoxy (including oxygen) is certainly a concern but we have never had any evidence of it. We have made solid PEEK lower plugs for the 8 mm tubes but these little plugs have to be exoxy-cemented into the tube, and are hard to clean for re-use.

You need as many upper plugs (fig. SH 3 and 4) as the number of samples that you expect to keep stored at any time, perhaps 4 to 10.

This section describes all the parts of the system made of PEEK, which is very expensive. It is used because it is very strong and hard. It is easy to clean hardened epoxy from it, and the important interface surface, between it and the precision glass tube that it slides inside of, never wears. It is not very hard to machine, but all work has to be done with carbide-tipped steel.

The upper plug (Fig. SH3-4) was redesigned with moderately tight clearance (.06 mm nominal) for the body (for the central tube, Fig. SH-4), and with end caps turned to as close a fit as possible to still allow the plug to be pushed freely into the tube. Four flats were index-milled on the lower cap to allow the sample to pass when the plug is pushed in, to the same diameter (Fig. SH-3). Our theory (see MRC)(which is not well verified) is that the ends keep the central tube aligned so that sloshing, produced by the >1000 G deceleration at the end of each travel, and partly in cooperation with un-evenness in the thickness of the plastic-to-glass clearance, will be inhibited. Originally I filed the flats by hand, using a magic-marker as well as a micrometer to keep track of where to file.

The upper plug also carries the groove at its top that is shallower than that described in MRC (now 0.13 mm deep, 1 mm long). We went back to using this groove because we were worried about water-epoxy mixing problems. The smaller-volume groove reduces the compressibility that the fluid sample sees. This compressibility may help the sloshing to occur. We may try an even smaller groove; the current groove nearly always keeps the setting epoxy away from the solution.

The central tube of the upper plug (Fig SH-4) is made from 1/4" PEEK stock. We bought a 3-fluted carbide steel drill to make the hole, and the hole is drilled half-way from each end with a thick section in the middle to provide extra stiffness. Great care is needed to avoid variations in the outside diameter of the plug, that might come from machining too fast.

The caps are glued with household 1/2 hour epoxy. It is desirable to machine the joints between the caps and the central tube to fit well, but not so tightly that they expand the outer diameter of the central tube. The wire at the top is usually epoxied at the same time. Do not be alarmed if the wire and/or the caps come apart during re-cycling of the plug when it is broken out of the glass tube and cleaned of old epoxy, simply re-epoxy them (but clean the cap a day in advance to allow time for this). Excess epoxy is wiped of with a tissue (Kimwipe) before it is set, and scraped off with a razor blade afterward (see above) and tested in an a NMR tube to see that it slide easily. The wire is bent and tested to give moderate resistance to sliding exery time it is used.

The upper plug is now (as of 5/2004) rather long (~35 mm), in order that the assembled plug would float during spinning, before we used the wire at the top as a spring to keep the plug in place. A length shorter than this may be desirable and the floatation feature may not be needed, we do not know at this point and we will try shorter plugs (1.5 cm?). The reason why we want to try this (aside from the fact that the plug will be easier to make) is that when assembled to the adapter (Fig. SH1) the upper end of the plug must be a few millimeters or more below the end of the adapter. Otherwise it is hard to disassemble the plug from the adapter without destroying it. If the nmr tube were shorter by about a cm, then the distance from the sample center to the adapter bottom could be reduced to about 55 mm instead of the present 65 mm and the tube will be shorter and stiffer. The distance of 55 mm was our original design, and would allow the bottom of the adapter to be about 5 mm above the recessed plastic top surface of the probe. The bottom of the adapter is now even with the bottom of the shuttle tube holder assembly (below) when it is at the bottom of its travel (Fig. ST2).

Other variations of the plug design will be tried, perhaps a nearly solid short design. It is useful but not essential to be able to be able to reach in and pull out the upper plug in case of some error as we now can, but it is not essential. We even connected a piece of thread to the wire at one point to be able to do this but gave it up since we never used it.

You need as many NMR tube adapters (Fig SH5, not to be confused with vee-block adapters above) as you do upper plugs. They are designed to be made easily and are made from 1/2" PEEK stock turned down to slightly smaller diameter so that the upper ends can be machined to get good and defined alignment to mate with the lower end of the shuttle body. Note that the upper central shoulder (0.3 inch diameter, at the top of the adapter) is less high than the depth of the corresponding cylindrical hole that it mates with(see Fig SH-7), so that it does not touch this surface of the shuttle body when it and the adapter are screwed together, and only the outer annular surfaces of the adapter and the body ends are used for alignment to perpendicularity, and the diameters around the inside of these surfaces are used for radial alignment. The mate should be rather snug, and the fit should be such that there is no play before screwing together, and no big crack visible where the two pieces come together, afterward. The edge of the connection surface marked (Fig. SH5) should be rounded slightly to allow this. Although the assembly of the NMR tube using the vee block etc. is the most important aspect of our system to get good alignment, we also think it desirable that the adapters and their connection to the shuttle body should be excellent.

The stud connecting the adapter to the shuttle body is made without special care from a cut-off brass or stainless steel screw and trimmed. It is designed to be screwed moderately tightly into the shuttle body, where it will live forever. If it is not brass, check it with a stir-bar or other magnet for non-attraction.

The long hollow tube of the shuttle body (Fig SH6) does not require great care in fabrication for alignment. The design shows an optional thick section in the middle if you wish to drill from each end (we did not do this the first time). It is now shorter than that in MRC but could be longer than shown. We bought a carbide-tipped drill of an inexpensive type (Koolcar durapoint, MSC catalog p.95) to drill this hole from 1" stock, after finishing the outside and predrilling with a ½" carbide tipped drill. Then we turned a 1/2 " long section in the inside diameter at each end of the tube for the epoxy joint with the two end pieces, slightly larger than the drill hole. It is a good idea to score these surfaces and the mating ones lightly after this, to make the epoxy permanent Then we turned the outside diameter to ~3/4 inch, to reduce the weight.

The two end caps (fig. SH7) carry the 0.798 nominal dia. bearing surfaces that have .001" (.03 mm) nominal clearance with the precision glass shuttle tube. This precision surface should be made, for the bottom cap, at the same time as the lower end of the lower cap that mates with the NMR adapter, to make them as well aligned as is easily possible. The 1/2 " outer surfaces that carry the seven O-rings are not so critical. I worried that the seven O-rings would fall off but they do not seem to, they are actually slightly less than ½ " inside diameter. Be sure to get Viton (Fluorocarbon) O rings for this.

You can cement (with 1/2 hour epoxy) the lower cap while the tube is in a collet in the lathe, setting up an indicator to test for alignment. Then after it has set, cement the other end using a jaw holder in the same way. It is probably better to epoxy the top cap first, without special precautions, wait for it to set, then epoxy the bottom cap using the vee block. First make a vee-block adapter with the correct diameter (slightly less than 1/2") to fit the outside of the NMR adapters. Then screw the adapter and bottom cap together before epoxying the bottom cap. Do this epoxy-ing in the same way as we seal the NMR tube, with the vee-block

We test the vee-block procedure as described in MRC.

The shuttle should drop freely though the open glass tube in about 1 sec but, with an adapter screwed on, it falls remakably slowly, abou1 cm/sec.

Temperature. See MRC. We have only done a few longer runs up to around 50 ^{o} C. I expect from various experiences that cracking of the epoxy, or the glass at the epoxied points, will occur at some temperature not much above this. I think it is better to use as little epoxy as possible. Possibly the nmr tube seals could be set in a warm room or incubator at the mid-point of the desired temperature range. Possibly a supplier like Epoxy Technology could suggest a good epoxy. (However, the high viscosity of the household epoxy we now use is good for cementing the upper plug in the glass tube. Don't substitute a lower-viscosity epoxy here.)

Section ST. Shuttle tube and stop tube.

Cleanliness, The shuttle tube does not have to be super clean but if there is any chance that dirt has gotten in, the assembly should be taken apart and the inside of the glass washed out with good water and then ethanol, and dried by pushing a dry paper towel through it with a clean rod. The shuttle and O rings on it should be likewise cleaned. This is especially important if there has been glass breakage. The symptom of such dirt is: the shuttle gets stuck. It only takes 1/2 hour to clean things but it is annoying after a run is set up. Perhaps as a matter of policy we should disassemble and clean before every block of running.

The shuttle tube components are made simpler than before because they no longer carry the O-rings of the debounce system, these are now on the shuttle body; and I based the support system on the smallest diameter heavy wall brass tube that would clear the glass tube (1.25 outside dia., 1/16" wall). I chose a heavy tube because I thought it would be less prone to vibrations after the sample lands back in the center, and that they would be better damped. Perhaps so.

The current "new" design (6/2004) looks, on the other hand, more complicated than before because there are two window sections on it that expose the glass directly (see Fig ST-1), the support in these sections being provided by 1/4" dia brass rods (lower window inside the magnet) or 5/8 " dia. brass rods (upper window, above the magnet). These sections will allow me to put saddle coils around the glass and run a coax up to the top if needed, to apply rf to the sample at any desired stop position.

The saddle coil will be crudely made to slide up and down the glass by hand, when the support assembly is removed from the magnet. The lower window will allow me to map the magnetic field by nmr, by allowing me to try to saturate a thin water sample, mostly deuterated (to get a long proton T_1) in a field cycling manner, during a time of 1 sec or so when it is at the upper stop position. Loss of NMR intensity after the cycle will tell me that the sample was saturated, and was is in resonance with the rf frequency, and provide a point on a field vs height determination.

I have not done this yet and it will be laborious. When it is done I can dispense with the lower window. The upper window allows the same thing above the magnet. I could also do T_2 measurements at up to 1000 gauss there, by shimming the field there moderately well, and I could even do coherence transfer between ^{13}C and protons for example, at low field. I can see a potential use for this but have lots else to do. Optical and microwave irradiation could also be applied through the window to do various DNP experiments (not planned as of 12/04).

The windows also allowed me to put an optical sensor temporarily on the glass tube to investigate the speed and bounce of the system. It is an ultra-bright LED facing an inexpensive visual light phototransistor. They were mounted with tape on a light metal ring that I put around either window by taking the support rods apart. I established that the system can now move the sample up to 1 cm/msec but that it bounces a single bounce at both the top and bottom lasting around 30 msec (more at higher speeds), the height of the bounce being about 1.5 cm. I can't now say how serious will be protein denaturation problems at the highest speeds. I could not break either 5 or 8 mm tubes at the highest speed. But there is not much improvement in overall speeds for vacuum above about 2 psi (.133 bar) or pressure above 4 psi (.266 bar). Perhaps the flow of air is limited by turbulence in the solenoid valves (an estimate of the Reynold’s number in the valves supports this idea) and greater speed could be attained with 3 or 4 valves in parallel in place of the 2 I now use. We may work on improving this flow in the distant future.

At the end of this section I will also talk about the tube extension and field bucking modification to do T_1 experiments down to 20 Gauss (reported in a poster, ASBMB meeting Boston '04) to get sensational new results on membranes (see PNAS Dcember ’04).

Alol the PEEK machining has now been described and we now proceed to discuss, very briefly, the individual parts of the assembly shown if Figs. ST-1 to ST-3 which are the main parts of the shuttle tube.

Figs. ST1-3 are schematic/assembly drawings of the shuttle tube support system, and ST4 A, B, etc are mechanical drawings of the parts from bottom up. Several parts have longitudinal grooves cut in them after being turned on the lathe, indicated on end views. The grooves are intended to provide a low-resistance return path for air flow. These do not have to be very precise as long as the remaining sections of the outer diameters are not touched. As in MRC, Fig. ST-2 and others may be confusing since the two sides are not identical: the cross section on the right side is through the connecting screws, and on the left it goes through the grooves, with dotted lines where material is cut to make them. (the assembly diagrams are very useful to check dimensions).

The window sections held together with brass rods were straightforward to design and I will not say much about them. I was afraid that the construction with brass rods would be flimsy, and the glass tube would break, but it seems good.

As before the support is pretty trivial: it supports the glass tube at the bottom on a Buna-N O-ring (in a well at the top of part (3), fig ST4-B), and it supports the glass tube at the top with very light force downward via a plastic ring (part (9) fig. ST4H) that compresses the bottom O ring very slightly. The bottom end piece(part 1), fig.ST 4B) has to center the bottom end of the glass tube on the probe correctly, and for a Varian probe the top end of the probe also has to be centered by part (1), fig ST4 A. The top end of the support system has to be centered on the magnet bore and this is now done with a plastic ring (part (5B), fig. ST4D) that is taped on the brass tube just below the point where the tube comes out of the magnet. The entire support tube, windows, etc are still supported from the top of the magnet, and simple adjustment screws at the top allow us to change its height to center the sample in the probe vertically. We are trying to avoid having to change this adjustment very much, by standardizing the length of the NMR tube etc. (above, a big success). These screws are now located at the very top of the shuttle tube support assembly, and they are, for the time being, sitting on top of a Helmholz coil system that we place there even when we don't expect to use it.(see Fig ST-1 for its approximate location). An extension tube can be added at the top of the system to allow us to move the sample up to 20 cm above the magnet for very low field work (see also Fig.ST 5A and B)

Details.

Starting from the bottom, we no longer use separate adapter rings rings stuck in each probe top to center the probe (as in MRC). Part (1) Fig. ST-4A has two surfaces (Outside diameter (OD)1.430 and 1.563 inch) that mate respectively with the upper inside of the probe, and the inside of the shim coils, and thereby center the probe in the magnet. This piece is made of Kynar, which is cheaper than PEEK but harder than Delrin. We like to use plastic to mate with the 500 magnet to avoid damaging it. We hope that this piece is the only one that would have to be modified for a Bruker, because their probe is already centered in their room temp shim coils assembly, supposedly, and only a single bearing surface may be needed.

Part (3), fig ST4B carries part (1) and on the other end it mates with the glass shuttle tube (see Fig ST2) and centers it precisely on the outside of a pedestal that pokes in to the precision-ground inside of the bottom of the glass shuttle tube. It has a 0.560 inch hole drilled through it, through which the nmr tube adapter can easily pass. The horizontal annular area between the glass tube and this hole is where the O-rings on the lower end of the shuttle-body land (see fig ST2).

These parts might be modified as follows: Decrease the length of Part (4) and the nmr tube adapter to get a shorter, more rigid adapter. Decrease part (2) and increase the length of part (4) to allow space for more O-rings on the shock absorber. Increase the length of either part (3) or part (4) and open up the top of part

(4) to be able to use a larger diameter shuttle tube. The current glass shuttle tube size is the largest that could extend below the room temp. shim coils.

Parts (4) and (6), Figs. ST 4C and E, are connected by the ¼ “ dia. brass rods (RCA), stiffened by the well-fitting countersink holes. The brass tube (5A) fig ST4D is soft-soldered into part (6), after it is hard soldered into part (7), fig. ST4F, and after the alignment ring (5B) is slid on to it. You should cut the tube and rods last after you understand what follows, and you may want to change their lengths. Also, the face of part (7) (fig. ST4E) should be refaced in the lathe after the hard soldering step. This seemed likely to be the most-strained joint so we hard-soldered it, but that may not be necessary. In any case all solder joints and rods must be made with as much care as possible to preserve alignment.

Finally, rods (RCB), fig ST4E connect part (7) to the top, part (8) fig. ST4G. The top should not be screwed to the 3/8 " rods, except for preliminary tests, until the cap is assembled on the glass tube (below) and the rods (RCB) are cut to length. Don’t make these rods until you understand the following. And don't attempt to understand what follows without a copy of Figs. St-1 to ST-3 in front of you1!!

In the first place, early on you should put whatever probe of yours that you will use for field cycling in the magnet, in the normal way required by Varian (which is weird) and adjust the probe height with Varian’s upper stack (their mysterious name for a nearly useless tube. “push up, push down”). (If you have two probes as we do it should not matter which probe you use, they are standardized (I hope). Then tighten the Allen screw tightly at the bottom, that clamps onto the probe to determine its height, and NEVER CHANGE IT AGAIN. Now the probe is in a good place we hope. Now remove the “upper stack", of course first REMOVING THE SAMPLE that perhaps was in there.

Now assemble the shuttle tube support up to part (7), and be sure you PUT THE O RING in on the top of part (3) to cushion the glass tube when you put it in. It is an ordinary Buna-N ring, ¾ "

I.D. x 1/16 " thick. Put the new assembly, carefully and gently, in the magnet hole from the top, leaving your probe in the magnet, (with no glass tube or shuttle etc) as far as it will go. It will probably go until part (7) hits the top of the magnet (that is, the top of the smaller aluminum flange the stays there always), or almost does. This means that the assembly is not too long. If it does not go in that far, make sure nothing unexpected is too big in diameter; if not, it is to be hoped that the bottom of the assembly, Part (1), is resting on the plastic part of the probe at the top, inside the top of the probe shell. (That is why you have to be careful!) Normally the bottom of part (1) is designed to be at least ½ cm above this point, and there should then be a few mm between the bottom of part 1 and the top of the magnet for air to get out and for the cap screw caps that connect to rods (RCB) fig. ST4 F. If the support seems too long at this point you have to shorten the ¼" rods (STA) or the 1.25" dia. brass tube. In any case you probably want to measure the distance from the inside plastic surface of the probe to the magnet top carefully using a clean plastic or aluminum rod as a probe, and a marker or piece of tape to mark the top. Then if it seems that the assembly is too short by the above criterion you have to throw away something, perhaps the ¼" rods and make new ones etc.

Now we come a diversion, namely the **plastic cap** (part (9), fig. ST4H**) for the top of the glass tube.** It protects the tube end from chipping, and holds the tube in place. It is a good idea to order 2 or more glass shuttle tubes from Wilmad, and when you do, ask them to make them all with the same length. They will probably do so for free and then it is likely that, if you make identical plastic caps for the glass tubes, they will be interchangeable after being assembled.

What follows is needed especially if you want to connect glass tubes to other tubes (in our case, a shorter brass tube, see below, but in other cases such as for a larger magnet, another glass tube.) The outsides of their tubes are not precision ground, at least not the ones I could afford. So if you try to butt-join them it will be hard to avoid a shoulder on either side that would hang up the shuttle tube. My solution is to have a hole of slightly larger (.06 mm) diameter perfectly centered on the precision inside diameter of the glass tube. I have not yet designed a coupling between glass tubes, but the following was done to allow me to join a glass tube to a short brass extension tube (see below) to allow me to get the sample a few cm above the top of the magnet (to be described later). The cap (9) fig. ST4 H was made with a precision *inner upper* surface on a lathe, concentric with its precision *outer* reference surface. The *inner lower* surface is made a very small amount larger in inside dimension (perhaps 0.75 mm= .003 inch in radius) larger than the glass tube. Then I devised a way to fix this cap so that this surface is almost perfectly aligned with the inner precision surface of the glass (below). As a result, the outer surface of the plastic is perfectly aligned with the inner surface of the glass. It could then be coupled to another glass tube capped in the same way with ordinary couplings made on a lathe, to align the two caps.

In the design here, the upper inner surface of the plastic cap is specified to be very slightly larger than the inner diameter of the precision glass tube. The diameters of the shuttle body's bearing surfaces are conically reduced slightly at each end, to be able to negotiate the small shoulders (0.02 mm or 0.0007 inch) between the glass and the plastic. [In fact this is the *next* design. Currently the plastic inside surface and the glass are the same nominal diameter and I have had trouble with the shuttle sticking. I had to hand-sand the plastic with very fine sandpaper. I hope, and am pretty sure, that the new procedure described here will work better. Let me know.]

I assemble this by having made a Teflon “epoxy tool” fig. ST 4F (bottom), that mates the inside of the glass and the plastic together. The upper and lower outside diameters of the epoxy tool are very slightly smaller than the inside diameters, respectively, of the plastic cap part (9), and the glass tube, and the epoxy tool mates perfectly with these surfaces to align them The inside diameter of the lower hole in the plastic cap (Fig. ST4H) has to be made sufficiently larger than the outside diameter of the glass tube so that this is possible with as small a clearance as possible, as needed because as already explained the glass thickness is not precisely uniform. Then the glass tube, with the plastic cap stuck on it via the epoxy tool, is inverted and spot-epoxy (household, high viscosity)is applied to 3 spots in the space around the outside of the glass where it exits the plastic, while the parts are held firmly but gently together in a standard lab holder. After this sets I *remove the "epoxy tool"* and apply Epotek low viscosity epoxy which fills the remaining small space between the plastic and the outside of the glass to make a strong joint. I do not do this assembly in one step of cementing, because the low viscosity epoxy might flow around and interfere with removal of the epoxy tool. See Fig. ST4H. The household epoxy is too viscous to do so, but it establishes the alignment, and the immediately-following low-viscosity epoxy gives strength.

Back to the main assembly: Support the assembly made so far in a secure clamp to hold it vertically. Now you insert the heavy glass shuttle tube with its cap attached (see above) and put it in all the way, seated on the O-ring and surrounding the pedastle at the top of part (3).   G*ently* hold on the top piece (8) and estimate how long the 3/8 spacer rods RCB should be. Cut them carefully to equal length with a little length to spare. Then screw them in place loosely, connecting parts 7 and 8. If the parts don't fit you made the rods too short; toss them out and make new ones.

Now measure how much extra space there is above the plastic cap which is on the glass tube. Now the hard part comes: you cut down the 3/8 brass rods until there is no excess space between the top of the plastic cap and the shoulder (0.6 " above the bottom of part (8)) to which it mates, but almost no force on the glass tube transmitted down to the O-ring on the top of part (3). **This completes the shuttle tube assembly.**

Now you have to make a support for the shuttle tube to hold it at the correct height in the magnet. If you did not make a Helmholz coil for work at very low fields you could make a “dummy” thing that resembles it; or you could have redesigned the part (7) so that it has a flange like the one at the top of part (8), with screws through it to adjust the height of the shuttle, which would rest on the top of the magnet. [In fact we used to do this (see MRC) but we always put down a thin sheet of aluminum to protect the magnet flange from being scratched.]

[Sections like the following one that refer to “very low fields” or “Helmholz coils” do not describe anything related to MRC. They are new capability used to generate the data in our last paper in PNAS, Dec. 2004]

The Helmholz coils are each about 16 cm in average diameter, about 2 cm thick radially and each 4 cm longitudinally. They were made by Dmitri Ivanov for his quadrupole resonance experiment (J. Magn. Reson. 166:19-27 (2004)), and each has about 345 turns of #16 wire. They were would on a lathe using a wooden coil form 4 cm long and about 12 cm outside diameter, having gaps and screwed together to >20 cm diameter end pieces of wood so that the parts of the form could be pulled off the coil without sliding. The wood was first treated with wax to resist epoxy bonding. Wire came from a spool on an improvised stand next to the lathe, and it was tensioned while winding by a rag held in the hand, or pieces of Delrin clamped lightly on the wire. After each layer was wound epoxy resin was generously smeared on the entire layer, before winding the next. The result is rigid and conducts heat well so that an air fan could increase its power capability, and we did point a fan rather crudely at the coils when Dmitri used them.

The coils are supported by 3 plates outlined on fig. ST 8 (top), separated by spacers which are shown informally in the sketch at the bottom of the same figure. The coils rest on the two lower plates, contacting the four wings on each plate only, for vertical support, and held in place radialy by the four rods. Other than the wings, the coil surfaces are exposed for the sake of cooling. A small muffin fan hung in mid-air helped cooling for Dmitri's setup but we have not used it yet in this device. The rods are ¾ inch in diameter and the central one is threaded at each end for 10-32 screws. The upper rod is drilled out with a ¼ inch hole and the purpose of the upper plate was simply to provide a convenient surface to support the shuttle tube assembly. The coils are held in place by several plastic cabling ties. The supporting plates and rods have pieces of Scotch-brand thermoplastic glass electrical tape attached to them using a soldering iron briefly applied to melt their adhesive, so that the coil does not contact the support structure directly. . The inside diameter of the coils was only slightly smaller than the outside diameter of the top aluminum flange of the Oxford 500 magnet, so I only needed to offset the holes in the upper two sets of rod by 1/8 inch to do it as shown. Alternatively separate sets of holes could be used for the four feet. Probably you will redesign the coils so I do not give detailed designs.

The lower support rods (fig. ST 8, between the magnet and the coil) were Delrin drilled out to ¼ at the top, and hollow, with holes large enough for a cap screw head at the top. They fit the outside of the aluminum plate at the top of the Oxford 500 magnet snugly, and thereby align the magnet radialy as well as supporting it vertically, resting on a black plastic (?anodized aluminum?) surface to which the top plate appears to be screwed. **Important!** The lengths of these rods should probably be determined once the shuttle-tube assembly is complete. Their length should be such that the shuttle tube assembly sits at the correct height for maximum sensitivity of NMR detection (that is, with the sample at the sensitive region of the probe) when the 10-32 screws with larges knurled tops (McMaster Carr) were screwed in with ½ to 1 cm projecting out the bottom of the flange of part (8) (“HEIGHT ADJ SCREW”, fig. ST-1). These screws would have to be adjusted if the NMR tube is not epoxied into the NMR tube adapter at the usual heath that you establish. The height adjustment is again discussed in a few pages.

For our setup we have designed the top cap (8) to have the adjustment screws as high as possible, so that the Helmholz coil can be rather high, and so that its center is about 14 cm above the top of the top aluminum plate of the magnet where the field is slightly less than 0.03 Tesla, dictated by the fact that the Helmholz coils and our 50 V 10 A supply could reach this field, and buck it to allow us to get to nearly zero field. [Do you wonder why not zero? If we went close to zero we would have to verify that there is no transverse field component at the magnet center.]

The solid wires of each coil are connected to separate short 2-wire cables that are tied down with tape to the support assembly. Then these cables go to a temporary screw terminal barrier strip where the are connected in series, and from there via a few feet of multiple stranded wire cable to a 24 pin AMP "circular plastic connector" type 23-24 which hangs on the cable (more trouble to order than to make. Do not bother to get a crimping tool, solder the wires.). The solid wires must not flex in ordinary use! This makes a compact unit that can be lifted on and off the magnet when moving in or out. This magnet produces more than 320 G using a 50 v 10 amp Kepco supply which is left turned on, and whose output connection is on and off with a solid state relay. A reversed power diode across the coils protects everything. See Fig. EC. This is the fringe field 13.5 cm above the magnet. The gradient from the main magnet is about 10 G/cm there. For Dmitri's work we could get the field homogeneous to about 1 Gauss over 1 cm by running unequal currents through the two coils. We did this by connecting two cheap Tektronix supplies carrying opposing currents to the two coils. You can connect several current- regulating supplies to the same place but you have to put in series resistors or series fixed voltage supplies in series in order that the supplies can operate on a positive voltage within their range. We will do this if we want to measure R2 or do polarization transfer at low field. However, it is unlikely that we will do this except perhaps for R2 of ^{31}P. We probably cannot get much above 1000 Gauss with this magnet, and in most cases T1 is too short at this field to allow us to do any experiments.

It would be nice to have a temperature sensor that will turn off these coils. Otherwise in case of an electronics failure the 500 magnet might be damaged when the magnet goes up in flames.

The extender tube (fig. ST5A) can be put on the top when we need it, to get to very low fields by raising the sample to 14 cm above the top of the magnet and using the field of the coils to buck the remaining ~300 gauss there to nearly zero. Because it is hard to machine with precision we made it slightly larger in diameter than the glass. Probably 0.804 inside diameter would work better to avoid problems. It is very straightforward, mating at the bottom part (8) in a precision manner, and shaped at the top like the top of part (7), so that the stop-tube-clamp can be put at the top of the extender tube instead of on the top of part (7). It is awkward to have this tube attached if it is not needed, and I designed it with trapped screws where it attaches to the top of the shuttle tube, which can easily be tightened by hand (Fig. ST-5B). Only the lower half of the inside of the brass tube has to be well machined because the upper half does not actually contact the shuttle. It would be hard to machine an extender tube much longer than this one unless you have an unusual lathe, and instead a glass tube coupled with plastic caps as described above would be needed.

The stop tube, Fig. ST6, is very simple. We have 3 of different length, overall 20, 38, 81 cm. This is a good assortment to cover the full range of fields. The shortest is for use at very low field with the extender tube. A short shoulder around the outside of the lower end piece (Fig. ST5A) with small clearance to the glass centers the stop tube so that the end of the shuttle tube that holds the O-rings will not hit the shoulder of the stop tube that contacts the O-rings when the shuttle tube hits the stop. This shoulder should be short, as shown, so that the glass cannot be easily broken when the stop tube is not lifted out absolutely vertically.

The upper end of the stop tube is simple cut off with a small bevel, at the top. It is coupled to a rubber tube that goes to the solenoid manifold by a vacuum quick-disconnect as described in a later section. It happens that the tube slides into this fitting too far, so we have a stainless steel collar semi-permanently attached about ¾ inch from the top of each tube to prevent this from happening. This has to be put on the end after the clamp (just below) is put on the tube.

The clamp assembly, fig.ST7, holds the stop tube in place to determine the lower field-value. There is an identical one on each stop tube. It gave a lot of trouble because it must be easy to move the stop tube when the clamp is released, but it must hold it tightly enough to resist the thousands of poundings during cycling. No matter what the design, it is good to put a piece of paper tape on the stop tube just above the clamp when you start a new run so that you can look in on the spectrometer every so often and see if the tube is moving upward during the run, at least if you are not confident about this point.

We list and explain the components of the clamp starting from the bottom, in the order in which they have to be placed on the stop tube. The first component is, when in place, just above part part (8), fig. ST4G (and above the plastic cap (9), fig ST4H, which it MUST not touch), is a ring (Delrin or aluminum) about 1.2 " OD, 5/8 " ID, ½" long (not shown)) that sits in the small well at the to of part (8) and centers the stop tube. It is machined to slide easily on the stop tube and fit easily into the well. It is essential that this end of the stop tube be moderately centered so that the shuttle's upper end will enter the stop tube smoothly. After this is an optional 1/16 thick O-ring 5/8 or ¾ inside diameter, as a cushion. Next is the part (A) fig. ST7 which is in fact two nearly identical pieces of aluminum or brass that fit around the 5/8" stop tube with the central hole illustrated made large enough to slide on the tube without much resistance, as already mentioned. The gap between pieces allows the clamp to be closed tightly on the tube. One side of the clamp is closed by a 10-32 screw with a tight lock nut and a spacer chosen to hold the clamp slightly open (all not shown). The spacer consists of washers or thin shim stock, and they are threaded onto the screw after it is inserted through the un-threaded hole of one side of a clamp, and before the screw is threaded into the other side of the clamp. The screw on the other side is closed with the wrench (S2) that can be tightened without great effort. It has to be long enough to clear the various components nearby. Next on top is another O-ring for a cushion, and finally a top plate (B) that holds two knurled screws (S1) in two small tubes that extend below it. The part (B) is put in with these tubes pointing downward and is oriented as shown in fig. ST7 so that the flat plat is in contact with the O-ring. The length of the small tubes is such that the lower ends of the tubes almost touch the upper surface of part (8), and can be screwed into holes tapped into that plate to secure the clamp system. The idea is that these screws can be tightened to slightly compress the O-rings and firmly capture the clamp, without preventing the clamp from being loosened. The screws S1 and plate assembly trap the screws so that they do not get lost. Finally, a small slot was cut in one of the clamp pieces to make it easier to close while permitting a looser fit when unclamped.

The sample-sucker for pulling out samples is shown on fig. ST8. It consists of a plexiglass fitting cemented to a standard US ¾ size sweat-solder Tee. To the side arm of the Tee is permanently attached a small valve, and a rubber tube is permanently attached from the other side of the valve to the vacuum reservoir on the Tower (below). The other arm of the Tee is not used and you can probably use an elbow instead of a Tee. The valve is normally closed. To remove a sample the stop tube has to be removed by unscrewing the knurled-knob screws that go through part (B) page ST 7, and pulling the stop tube out being careful not to break the shuttle tube. Then the bottom of the sucker assembly is pressed into the 1.2” diameter shallow well normally occupied by the lowest part of the clamp assembly, in the top of the shuttle assembly (see part (8) fig ST 4G), and the valve is opened. The sample rises and you may see the top of the shuttle enter the plexiglass piece. You then gently lift the sucker, with the sample stuck to its bottom (leaving the valve open, of course). Once it is raised about one inch you grab the shuttle with your fingers and lift it and the sucker out, and eventually close the valve on the sucker. That's it.

Installation. Generally I install the shuttle tube assembly before the probe and, obviously, after the Helmholz coils are in place. Then insert the probe, as usual taking things very slowly. For the 10 mm probe there is no problem. For the 5 mm probe you have to worry about a vestigial ridge that previously aligned the probe's gradient connector. [these lines were re-routed by Varian to come out the bottom of the probe on a connector, at no cost, after the probe was delivered. Be sure to ask them to do this, apparently it is easy except there was some indication that they did not do a great job of by passing these leads against RF interference. Almost certainly they could omit the ridge I am talking about, it appears to be made of epoxy]. For use with this probe I put in a matching groove (on part (10), fig. ST 4A, and by rotating the entire shuttle tube assembly the ridge and shoulder can be lined up. Varian's ridge can also fit into one of the slots in part (1) that is put in to let air pass. In either case you have to be very careful not to force the shuttle assembly to rotate and break something or deform the thin upper aluminum tube on the probe. [Better get Varian to leave out the ridge! Bruker does not have such a problem].

Remember, do not loosen the probe at the bottom the way you normally do for a Varian probe. Set the probe height up once, then do not change it (assuming you have your own probes). By not having the shuttle tube and probe in direct vertical contact, as Varian does, I hoped to avoid transmission of vibration to the probe when the shuttle lands at the bottom.

Adjustment. The only adjustment is to have the height of the shuttle tube such that the sample is centered in the sensitive region of the probe, when it is not being sucked up. By sealing the NMR tube in in a reproducible way I hope to avoid making this adjustment all the time, but it will be necessary initially and occasionally thereafter. The height is changed by screwing in/out the three knurled screws at the extreme top. Adust one, then adjust the other two to equalize the force on them, changing height about 2-3 mm at a time. Keep track of what you do by measuring the distance between the top surface of the Helmholz coil assembly and the bottom of the flange that holds these screws. Lock on the sample, optimize the shim carefully including the phase, keep track of the lock gain, and keep to lock power as low as possible while the lock gain is near maximum. Note the lock signal size, change the height, optimize shim settings and lock phase, try again. Very tedious, find the highest lock signal. (It might be better to use the proton or phosphorous signal instead but we have not done so.)

If you keep track of the height as described below, then if you do not get the sample position just right, as determined by measuring the distance from the sample center to the bottom of the nmr tube adapter, you can compensate for this error by changing the height of the tube at the top. Be sure to remember that you did so, and change it back for the next sample. Then the tedious adjustment can be avoided or at least speeded up. (This works for us).

You may find that the vertical screws that go through the top of part (8), to adjust the height of the shuttle tube as just mentioned, end up at an inconvenient heigh, so that the screws have to be screwed all the way in, or all the way out, or nearly so. (In the former case spare blocks of metal can be used as extra spacers). If so, you can change the wat you cut the NMR tubes to length and cement them together, or else shorten or lengthen (re-make) the rods RCB. Sorry.

Clamping the stop tube at the correct height. The following applies when not using the Helmholz coil to get to very low field. Before you run, measure the overall lengths of the three stop tubes or refer to your notes if you have already done so. Pick a likely stop tube and call the overall length **S**. Before dropping each sample+shuttle in, measure the length **N** from the liquid sample center to the top of the upper O rings on the shuttle. Measure the height **C** of the Helmholz coil assembly from the magnet top flange to the support assebmbly top (note that this is 9mm less than the actual height of the coil support in our design because the legs doe not sit on this flange.

For each R_1 run, first decide what Tesla you want to get. Refer to the graph that tells you what the depth **D** is below the top of the top plate of the magnet, to get this field. You want to calculate the height **H** to set the top of the stop tube above the Helholz coil assembly top. By drawing a diagram on a piece of paper you will find the relation **D=N+S-C-H,** or **H=(N+S-C)-D.** So you calculate (**N+S-C)** when you put the sample in, and write it down, and subtract **D** to get the height **H** for each Tesla value**.** Now with the meter stick and a drafting square you try to clamp it at this height, as tightly as possible. The put a piece of paper adhesive tape on the stop tube to easily see, during a run, if it is moving as a result of the shuttling. If the height **H** is calculated to be negative, or positive but too small to get two cm or so that is needed for the clamp assembly, then you have to use another stop tube of a different length, or you may not be able to reach the desired field. If **H** is calculated to be too high for convenience, or the stop tube will not pull out that far, you may have to use a longer or shorter one.

Running at very low field (below about 300 G.) Use the shortest tube, which will permanently live on the upper end of the brass extender tube. It has a commercial stainless collar for a clamp. It is supposed to be set so that the sample is sucked up to be centered half way between the two helmholz coils. We set this by eye, in the magnet, by unplugging the electronics from the wall power (which opens the normally-open vacuum solenoid valves) and connecting the vacuum line to the wall, and opening the suction supply valve on the wall (and setting the vacuum regulator, if necessary to a couple of inches vacuum). If the sample is not in the right position half way between the Helmholz coils, the clamp has to be removed from the brass extender tube, and the clamp loosened with a hex wrench and reset to make the position correct.

To set the field, a parameter "cur" is set in the sequence FCLtonep, for example, to a number 1 to 255 according to a table that I supply, of cur vs field. Not that setting cur to zero turns off the field cycling entirely. You can do experiments with several values of cur, and one at 11.7 T, in que'd experiments.

This completes description of the parts that were made by a professional machinist. Most of the rest was assembled by A. R. with only hand tools and a drill press.

Part II, upstream from the stop tube

Most of what follows is likely to be modified by your circumstances. In our case the small size and low ceiling of the 500's room dictated that our system be very portable, whereas some labs could have permanent setups at or near the instrument. This is especially likely for shuttlers to be used with physically larger magnets (600 and higher) as will eventually be desirable.

Furthermore, I recently concluded that it is not needed to have the pressure/vacuum introduced at the top of the stop tube, requiring, or nearly so, that a set of heavy valves by lifted up and down each time the stop tube is used. Instead, the air I/O can enter at the side into annular space just above the top of the main glass shuttle tube, and transmitted to the inside of the stop tube by many small transverse holes near its bottom. The upper end of the stop tube might be a solid rod of around 1/4 inch diameter, and would not be completely straightforward to design. This is worth trying because it is annoying and fatiguing to deal with the air I/O as it is now done.

Engineers will hate our electronics system based on monostable (one-shot) timing and be tempted to use a modern system based on an embedded computer/timing system. But the present system is completely adequate. A problem might seem to be that the timing settings cannot be stored and reproduced as are parameters on commercial instruments. Perhaps, but air pressure parameters could not be documented so easily, and because of subtle differences between instruments they could not be easily ported.

For these reasons we will not present such details as we did above.

Section TW TOWER and components on it. The tower is useful probably in any case. We bought a heavy aluminum 84x19" full-size rack made by Bud (they used to make aluminum passenger railroad cars and "Budliners"), strongly recommended, with many 12-24 tapped holes on both sides of its vertical aluminum beams (the electronic supplier that sells it does not sell these screws which are unusual in the US; you get them from McMaster Carr or equivalent.) It has two very heavy (2 1/2 x 6 x 1/4") tranverse aluminum angle base pieces which I mounted *upside down* and on both sides of the rack's vertical channel pieces (The *front* of the tower is defined as the side of the rack that will be closest to the magnet). Then to hold the front wheels I cut two pieces of 1 1/2 by 3/16 angle aluminum 25 inches long and fastened them with at least 4 1/4-20 screws to the top (what would have been the bottom) of the base piece, extending out 11 1/4 inches from the front of the rack's vertical channels It thus extends 11 3/4 inches out from the back surface of these channels. The 1 1/2 " vertical surfaces of these angle pieces touch the inner edges of the vertical channels. Two 11" x 1 1/2 x 3/16" aluminum angles 11 long are screwed to the top of the front base piece angle, next to the long 1 1/2 " angles, so that they extend out exactly as far as the longer ones beyond the front. The bottoms of each pair of angles form a 3" wide surface to which I attached, at the most frontward points, stainless steel front casters. To mount the back casters I screwed a pair of 11" long 1 1/2 " aluminum angles to the base piece, next to the longest angle, toward the outside of the rack and ending in back, exactly even with the end of the longest piece. These are also screwwd sideways to the back ends of the long angle pieces To the bottoms, at the back end, of each set of these angles I attached a rear caster. The latter were swivel casters, but the front casters were not but were oriented toward the front; this is desirable because the front of the tower is less stable because the casters have to be close together to fit under the magnet The result is a rack which fits through all doors in the building, rolls freely, does not tip over very easily, and to which all kinds of things can be fastened.

An old piece of heavy plywood was hand-cut to fit in the space between the horizontal angle pieces, to form a wood base, and heavy cider-blocks were placed on it, leaving room for the reservoirs (below), to increase stability. The reservoirs are made inexpensively from polyvinyl chloride (PVC) 3" diameter tubes normally used for sewer piping in houses, and standard fittings, cut with a wood saw and cemented with standard cement for this purpose. Each is about 6 ft high. Heave plywood boards about 4" wide are screwed onto the outsides of the rack, extending backward, and connected at the back with a cross board. This U-shaped assembly is vertically a few inches below the top of the sewar pipes, and its back board is even horizontally with the back of the base of the rack assembly (with the bottom casters). The three reservoirs are attached to this assembly with elastic "bungie" cords available at hardware stores. Small strips of 3/4" plywood nailed into the base wood constrain the bases of the reservoirs, forming a 3" wall at the back and extending a few inches along the side, to constrain the bottoms of the reservoir tubes..

The bottom of each reservoir is closed with a standard cap. At the top, each tube has standard fittings (that can be found only in big stores or catalogs like that of McMaster Carr (with great care)) to reduce the size, and couple to a brass T of nominal 1/2" size (inside diameter ~1/2 "). The long side of each T is vertical, and the upper vertical end is coupled to a large quick-disconnect fitting, to go via a large flexible tube to the valve manifold (below). The side connection of each T goes to a smaller diameter quick disconnect and flexible tube that crosses via an overhead trough to the rolling table. This plumbing will be discussed more later.

A wooden trough that is instantly removable connects the tower to another smaller (49x19") Bud aluminum rack that sits on a relatively inexpensive white plastic and aluminum rolling lab table. The trough is made by nailing (with stainless steel nails) two "one by fours" with their four inch surfaces vertical, to a same-size bottom with the four-inch surface horizontal (and no top cover). The length of the trough is determined by the size of the laboratory and the need to keep powers supplies away from the magnet; ours is about six feet long. It is convenient (for moving in and out) to have the sides longer than the bottom by about 1 foot so that the ends form forks that can snag the wires and tubes that the trough must carry, during installation. The ends of the tops of the side wall pieces of the trough are tastefully cut off with a 45 degree angle cut. The trough is supported at each end by horizontal ~8" light aluminum angle pieces (1x1x1/8) bolted at convenient places on the sides of the two racks, with short (3") pieces of the same angle stock, at their outer ends, to keep the trough in place but allow it to be in stalled and removed easily. The bottom of the trough is thereby about 76" off the floor when it is installed. No wires or tubes run across the floor to the magnet or tower. The trough is vastly superior to commercial metal or plastic wire troughs.

The heavy (1 1/2 by 3/16) pieces of aluminum angle stock that are supplied with the tower's rack, for its top, are not used there. Instead these and lighter 1/8 thick pieces of aluminum angle stock about 20" long are bolted horizontally across the front or back of the tower at various heights to support anything you want. Most important, to support the valve manifold, two of these are mounted across the front of the rack about 3 feet apart vertically, with 7/8' diameter holes in their centers. A long (7-8 foot) 3/4" diameter rod passes vertically through these two holes to support the valve manifold and let us raise and lower it . I made Delrin bearings for the rod out of 1 1/4 x 2 pieces of Delrin 1/2" thick with 3/4 holes that were screwed in above the 7/8" holes in the aluminum with small screws. The 3/4 rod had the valve manifold attached at its upper end, and the entire rod was raised by a rope attached to a short foot at its bottom. The rope went through each wheel of a pair of double pulleys, one attached to the foot of the rod and the other attached high on the frame. In this way we have a 4-times mechanical advantage in lifting the heavy manifold. A nautical cleat fastened to the tower at a convenient level was used to fasten the end of the rope. Thus the valve manifold and rod, total weight around 15 lb, could be easily raised and lowered next to the magnet, and lowered to get through the door.

The valve manifold will be described shortly. It is attached rather loosely to a platform formed from the top of two short horizontal (~6" long) pieces of angle stock running roughly forward to back. The vertical sides of these pieces point downward and are attached to an assembly of aluminum pieces that I will not describe in detail. They form a rectangular box about 5" wide on both dimensions and 6" high. Near the lower end there is a horizontal 1/8 thick aluminum plate with a 3/4 " dia. hole, for the long 3/4 rod, described just above, to pass through loosely. Another similar plate and hole is located inside the box about 5" above the first, for the 3/4" rod to pass though loosely also. The two holes are about 4 1/2 and 10 inches from the top surface of the box, on which the manifold will sit. To get through doors, the 3/4" rod passes through this box, extending about 3" above the top of the box, even with the tops of the valves.

When moving the tower, a commercial collar (whenever possible use these, stainless steel ones, not the cheap set screw type but the ones which squeeze the rod with a single cap screw, it is always cheaper to use them than using some machining) fixes the position of the valve manifold vertically on the rod, and also the rod is limited in how far it can go down by the height of the highest fixed hole (3/4" bearing) fixed on the tower's front, through which the rod passes. The heights of the collar and of the crosspiece that holds the bearing are set so that the 3/4" support rod is about even with the tops of the solenoid valves, as mentioned, and both are just below the top of the lowest door through which the tower has to pass when the rope is released and the manifold is as low as possible.

Once the tower is in the 500's NMR room, we can insert a 6" wood spacer between the collar and the box to eventually get the valves very high. The spacer is made from two 6" long pieces of 3/4" plywood attached to a third narrower one so that they form a groove 6" long, and can surround the 3/4" rod snugly but not tightly. The (vertical) groove between the outside pieces is about 3" deep horizontally, and there are hook-like 1/2" knobs cut on the outer edges of these pieces, at both the upper and lower ends, that tend to keep the spacer in place. When it is, the valve manifold is pushed upward 6" relative to the rod, compared to when the spacer is not there, so that the end of the rod is 3" below the bottom of the valve manifold, just barely going through the top one of the two 3/4" holes in the support box for the manifold.

Section PL, plumbing. This covers all the plumbing from the top of the stop tube back all the way to the sources of pressure and vacuum. Very tedious. The purpose is, first, to provide regulated pressures (low and high) and suction, and then to connect these via electric solenoid valves, in the correct order and time, to the common output connected at the top of the stop tube. At this point you must READ PARTS of the MRC article which I will not repeat here. It even gives the valve manufacturer's part numbers but there was a misprint: the valves are Asco numbers 8262G212 for pressure and 8262G264 (thanks to Boris Itin for finding this)..

Later I will give an equally tedious description of the electrical system that mainly turns these valves off and on.

General remarks: it is good not to have leaks in the system before the solenoid valves (though very small ones can be tolerated). Downstream (toward the magnet) of these valves small leaks are not at all a problem because they are not likely to be large compared to the flow through the shuttle tube (however, note that this latter flow is nearly shut off as soon as the shuttle is at either end of its travel), and the valves are open usually only for a small fraction of the cycle.

The valve manufacturer recommends that the valves be mounted with their magnetic axes vertical, to aid in clearing particle dirt, but this is not a big problem because the air is well filtered, as is the vacuum (joke). I have had problems after modifications or glass breakage. In the latter case the shuttle itself often sticks in the glass tube and it has to be taken out and washed with water and dried with paper towels pushed through the shuttle tube, after any NMR tube brakage. We recently had one valve stick for no obvious reason (it was the last one to open, to let the pressure equilibrate to ambient pressure) but this fixed itself as soon as I had time to identify the valve, and to determine that it was not an electric problem. I followed this recommendation, and also arranged the valves so their bottoms were also on the same plane so they could sit on the platform formed from two pieces of angle stock, mentioned above. It is also desirable to make the manifold as compact as easily possible, and clustered around the 3/4" aluminum tube that supports it (not in a straight line, for example) and with space for this rod to pass through between the valves, vertically, close to their center of gravity. It is also a good idea to be able to add more valves later, easily. It was hard work to design the manifold. In general it was a poor idea to use plastic plumbing which breaks, so brass was used everywhere except as noted.

The entire manifold can be disconnected mechanically and electrically in a few minutes, and lifted off for inspection and maintenance. Unfortunately a strong person has to get on a ladder to do so. Getting the manifold back on is especially strenuous. The valves seldom are expected to need replacement (although I have one spare of each type), but their internal parts have to be inspected for wear probably ever half million cycles and this is easy if they are vertical. The Asco Company seems to think you will know how to take them apart. You have to slide off the top nameplate horizontally with the help of a screwdriver, and you can eventually figure this out. Then the solenoid coil slides up off the top of the valve. A spring then has to be removed to get access to the large hex nut that has to be unscrewed. I invested in a large wrench of the "box" type that encloses the nut like a socket wrench, but it is a single assembly. I took apart each valve, and put it together again, before assembling them, so that they would be easy to take apart later. Then you can take out the guts. BE SURE NOT TO GET CONFUSED ABOUT HOW PARTS GO TOGETHER, ESPECIALLY FOR THE NORMALLY OPE VALVES, AND INCLUDING THE NAME PLATES. ONLY DISASSEMBLE ONE VALVE AT A TIME.

Asco does not tell you what to look for when inspecting their valves, so I can't either. I have not had to replace one yet. I have a repair kit for both of the types of valves I used.

We have an electronic counter (Veeder-Root) that has a battery that will last 7 years and counts the number of cycles, to help us decide when to opent the valves up for inspection

Ordering the parts for the manifold (from McMaster-Carr) was a major job. Read what follows carefully when you do are ready to build it,, and not before, you will forget what you read.

I do not use Teflon tape for plumbing even though it looks good and does not soil your hands; I use standard plumber's compound to save time and frustration. Some of the details may be important, and changing them could complicate easy construction of the manifold.

Tour through the plumbing: I now describe the gas plumbing in tedious detail, going upstream, starting from the top of the stop tube. The stop tube itself ends at the top simply cut off on a lathe and then beveled slightly on the outside ("chamfered") to facilitate connecting the to the manifold. Upstream of the stop tube, everything remains connected together forever. The first thing upstream is a "vacuum quick disconnect" that adapts any 5/8 tube via a compressed O-ring to a brass solderable tube. They can be bought cheaply from vacuum supply houses. It is adapted to nominal (US size) 1/2 plumbing by drilling through a commercial brass plug of this size and soldering it in. This is screwed into one side arm (oriented vertically) of an aluminum 1/2" US nominal Tee (aluminum to save weight, this is a big Tee). The opposite vertical side arm has a similar plug screwed into it to which is adapted a silicon pressure gauge (Honeywell XCA505DN, marketed by Newark Electronics, 5 volt supply, +- 5 pounds per square inch). This is adapted to the brass 1/2 nominal plug with a plate that pushes it onto a questionable O-ring seal with the gauge's surface. These gauges contain parts that break if you drop the gauge. After you receive and study the gauge you will figure out how to seal it. It is a permanent part of the manifold assembly so it will not be likely to be dropped.

All plumbing of what follows is 3/8" US nominal plumbing and 3/8" (~11 mm) inside dia. rubber, copper, or plastic tubing except the solenoid valves which are only available as 1/4" nominal, and the 3 flexible tubes that are about 60 cm long and connect the movable manifold to the fixed tops of the reservoirs. The long (~8ft) plastic or rubber (for vacuum) tubes that go from the reservoirs to the table, via the trough, are also 3/8" inside diameter reinforced Tygon or thick rubber, not because pumping speed is critical for them but to avoid kinks that could pinch off the air flow.

Returning to our tour, the side arm of the aluminum Tee is adapted connected with a male (M) to female (F) aluminum elbow, (known as a "street elbow"). This elbow is important: it does not affect the speed of gas flow much but it allows a higher degree of flexibility of the connection between the tower and the stop tube.

(Note also that the entire manifold can swivel about the 3/4" aluminum rod, on around a vertical axis, which we arrange for the same reason.)

The other connection of the 1/2" aluminum elbow is adapted with standard brass fittings to a 6" length of rubber tubing whose other end is adapted to the central Tee of the manifold (which is female gender). This is the side-arm hole of a FFF Tee of the type which looks as if it were milled from a square block. (Tees have three connected holes and are sold in three forms, FFF (all female) and FFM (or is it MFF?) and FMF which have one out of the three connections being male and with FFM having this male being one of the two end connections, and FMF in the middle.)

The overall shape of the manifold is more or less like a two-pronged fork whose handle is the rubber hose already described, oriented towards the magnet, then a perpendicular cross piece made of three block-type Tee's, and two arms pointing away from the magnet. (sewe Fig. PA). The six valves are connected, three on each side, to the outer sides or ends of the arms. The cross piece consists of a central FFF Tee, already mentioned, screwed directly into FFM Tees via their M connections, and all in the same orientation. The two arms each consist essentially of a single standard (non-block) FFF Tee with its long part pointed back away from the magnet coupled to the middle F connection of one of the two cross-piece Tee's. All five Tee's are oriented so that the plane defined by the 3 connections is horizontal. The side-arm Tees and the two cross-piece Tees are not directly coupled, they are connected by a short (but not he shortest available, you need some space here) M-M connector (a "stud"). Now if you put it together, there are 6 female connections always connected to the rubber tube via the Tees: two at each end of the arms, and four pointing out though the relay. The current is off on any relay when its control line from the timing card is up (+5) so the strobe for sideways away from the manifold. The solenoid valves will be screwed into these holes via short MM 1/4 nominal to 3/8 nominal adapters. But wait, don't put it together now, first you have to screw the valves onto the T's, at least the side ones. And before you do that you have to read the next three paragraphs.

First you have to carefully read what I wrote in MRC to determine which wire driving the valves go to + voltage and which to -voltage!!! These wires are both red and you can't tell which is which, each valve has to be tested!!!. (a green wire on each valve is supposed to go to ground for safety reasons but is not so important for these DC voltage valves). IMPORTANT!! LABEL the wires after determining the one that is +.

Second you have to read the obscure paper sent with the valve to understand which way the gas is supposed to flow in the valve, indicated only by letter on the two ports. This may not be so important if you use the valves I suggest that are rated for considerably higher pressure than the pressure you are likely to use; but you never know. Obviously, for the three normally closed valves used for pressure the flow direction will be toward the rubber hose; but for the other three, that is two normally open for vacuum at the start of a cycle, and one normally closed for atmosphere relief at the end, the flow is away from the rubber tube. Mark these valves before assembling the manifold.

OK, after several hour you got it together. Plan now how to attach it loosely to the aluminum platform mentioned above, with a transverse scraps of aluminum angle stock and two long screws. Perhaps don't build the platform until now, after further thought. Before, this plan and install the plumbing to/from the three reservoirs. The valve that releases to atmosphere at the end has nothing connected to its female connection hole, not even a plug. The low pressure valve is connected to medium wall 1/2" inside diameter tygon tubing with standard fittings and this and the remaining two large flexible tubes are clamped semi-permanently with screw-type hose clamps. The remaining two lines from the reservoirs, vacuum and higher pressure, each go to a pair of electrically and functionally parallel solenoid valves. I used copper tubing to connect the upstream ends to the hoses, to get a pleasing compact connection rather than a multitude of bulky Tee's, at the cost of a slightly complex connection procedure (and a similar reverse disconnection, if the valve bodies ever have to be disconnected which is unlikely). First I assemble copper adapter elbows, 1/2" nominal pipe thread to 1/2" nominal copper "sweat" (solder) connections by soldering 4 of them onto 4 pieces carefully cut to be equal in length, about 3" long. These were then screwed into the four valves (with pipe compound) on the side that was to be further from the rubber tube to the magnet. Then, with the four tubes pointing downward, the four solenoid valves were coupled to the Tee's as shown in Fig. PA. Finally the Tees were all connected together as already described. *Then* I soldered the lower ends of the two pairs of the nearby copper tubes together and each pair to a short horizontal output tube, using two short pairs of copper tubing and two each of an elbow and a Tee for the entire solder part of the job. The long flexible tubes that go down to high-pressure and vacuum reservoirs are clamped directly onto the two open ends of the short copper tubes with screw-on tubing clamps.

These three large diameter Tygon or rubber tubes that go to the reservoir have quick-disconnect pressure connectors (McMaster Carr) (not to be confused with the vacuum quick disconnect used above at the top of the stop tube). They could be eliminated in favor of screw-type hose clamps, but use of quick-disconnects encourages removal of the manifold for inspection. (Here and below, "quick-disconnect pressure connectors" refers to a type of connector that usually has a one-way valve inside one side which is normally the high-pressure female side; but in our case we used so-called fast-flow types in which this valve is not included. For the particular variety we ordered, the female's were brass but the males were brass-plated magnetic steel, and we had to re-order stainless steel ones.) The quick disconnects used here were 1/2" nominal size. For these connecters in general we used an orientation for gas flow from male to female; as a result only one of the two pressure pairs of connectors has to be color-coded to assure proper connection because a vacuum line cannot be connected to a pressure line. Each of these three connections are standard, using hose clamps and barbed tubing connections where needed. The corresponding smaller lines to the relay rack on the table, via the trough, are nominal 3/8" size and entirely similar otherwise.

The vacuum reservoir also has a small diameter side-connection to a 4 foot small-diameter hose, going to the sample sucker. (an annoying feature of our present system is that the vacuum lines must all be connected and the electronics plugged in to use the sucker. This could to a considerable extent be alleviated with a small reservoir in this line having a one-way valve at its downstream end.).

The three long supply hoses are semi-permanently connected at the table, using hose-clamps to the three vacuum/pressure gauges (see MRC for the types). Pressure of about 20 PSI comes from a regulator on the lab wall, fed from the spectrometer's carefully filtered 80 PSI air, with a gate valve in front. A small tube connected reversibly to a taper fitting at the wall regulator goes to the higher 0-10 PSI regulator for the higher pressure, and feeds the lower pressure 0-5 PSI regulator, as well as the line to the tower. Manual pressure-relief valves at the output of both regulators are useful for setting the pressures.. All these plumbing and the vacuum connections are straight-forward. Unfortunately all vacuum gauges sold in the US seem to cover only 0-30 PSI vacuum and are marked in inches of mercury. These valves and gauges are mounted on recycled pieces of rack-panel, on the upper part of the relay rack that sits on the rolling table.

The vacuum originates in an oil-free pump (type given in MRC). This is a vane pump and can be used either for low vacuum or pressure, apparently. It has been trouble free but I have a repair kit anyway. It is in a sound-reducing box in the lab down the hall where I do off-line testing. The box is made of 3/4 roofing plywood to whose inside is cemented the fake anechoic chamber lining made of cheap foam (left over from the treatment of our 500 MHz NMR room). This is not a simple box but its internal top extends not quite all the way across, and an outer top covers all but a small section on the other end, and is about 4" above the inner top, so they form together a duct. This duct is lined with the anechoic foam. The box has a cooling fan mounted on a hole in the lower part of one wall to draw air into the top duct, very important, and should have a temperature turn-off switch to avoid a fire in case the fan stops and the pump motor blows up.

We had a vacuum line installed for this pump down the hall to the NMR room, with gate valves at each end. The vacuum is sensed by a standard electromechanical regulator at the pump, which turns the pump on and off to keep the vacuum around 1/2 atm. This switch tends to stick in the on-position so we installed a solid-state relay to run the pump, controlled by the regulator switch. We also have a pressure line installed, to ship the dried air pressure back from the NMR room to the development lab. It is not connected yet so we use standard nitrogen gas tanks to supply the brief tests we usually do in this room.

SEECTION EL, electronics. *Tower.* The only electronics in it is a +5 volt regulator chip that converts the 12 volt relay supply voltage to 5 volts for the silicon pressure sensor . All lines go to two ten-position terminal strips which also hold the 5 v. regulator. These terminal strips are bolted to the vertical arn of a piece of 1x1 aluminum angle stock, that also holds down the valves on their platform by means of two screws through holes in the platform. The 12 wires from the relays are screwed to these strips and then all the relay + wires are connected together to the input 12 volt supply. (see Fig. (EA), lower section. The two pairs of minus wires from the valves that operate in parallel, for the vacuum and the high pressure, are screwed into the same terminals of the blocks. Back-biased power diodes are installed here between the four - control wires and the +12 volts, with the cathode side to +12, to protect the solid-state relays. All wires but the output of the pressure gauge go through a 6 conductor cable about 70 cm long, to an archaic 8-pin connector (Cinch Jones) which will be replaced by a type that is easier to connect and disconnect. (We will standardize on a low cost 24 pin round plastic connector made by AMP, CPC series.) The signal from the silicon pressure gauge also goes through the same connector but is otherwise carried by a coax cable taped to the 6-wire cable to reduce noise. The upper end of the coax and 6-wire cables are firmly taped to tubes of the manifold, to avoid breakage.The 8-pin connector is disconnected when the tower is removed from the NMR room. The pressure signal is again routed through a small coax line, taped onto a six-wire cable after this connector. These lines then all go via the trough to the table, where they are semi-permanently connected to a circuit board.

Table. Solenoid valve control. Most of the electronics in general is on circuit boards that live in a Vector type CCA13C/90 cage. The circuit boards are mostly standard Vector type 3677 proto-boards with 0.1" hole spacing and convenient layout of connectors and pads. The cage has its own small +5, +-15 V supply module. Interconnections are via soldered wires between board connectors or 26-wire ribbon via standard IDC connectors or barrier strips, to the cage, to a few BNC cable connectors, or to the wires mentioned above.

The four minus end wires from the valves, and the ground and +15 go to a connector-less circuit board via a barrier strip, that slides into this cage and normally lives there (Fig (EA), top section) This card has four nearly identical solid-state relays on it controlled via a ribbon cable from the timing card in a conventional way. The +inputs from the solid-state relays all go to a +5 supply voltage, and the -inputs go to the ribbon.

The timing control card (Fig. EB) runs these four control lines and another that turns the Helmholtz coil on & off that is used for very low field operation (below). These are controlled by 7407 open collector drivers. The inputs of this card are only 3 wires: most important, a positive gate from the Varian console that coincides the "raisetime" of the sequence. It comes from spare (coax) output 1 on the back of the Varian console to the end of a long coax cable that dangles off the magnet in back. To this is connected another long coax line that travels via the trough to a coax connector on the back of the cage. The other input lines to this card select either this strobe, or an internal test strobe generated on the card. There are also three potentiometers on the front panel connected to the timing card via the ribbon connector, one of which determines the length of this test strobe, and another which determines the time between strobes. A third one controlls the time of the high pressure . These are connected via the same (and only) ribbon cable as the solenoid control lines. There are also two wires that transmit the position of the "Varian-stop-test switch on the front panel.

I will not describe how the 555 chip that generates the strobe works (bottom left of Fig. (EB)), you will have to get help from a local electronics technician, nor how the "one shots" (officially called monstable multivibrators) type 74221 work. Basically the latter convert a TTL negative or positive edge into a pulse whose length is determined by a soildered-in capacitor and a resistor. They are about the simplest thing you find in digital electronics. The circuit diagram EB uses conventional symbols for some logic gates, and box symbols for the timer and one-shot units. The one-shots are in dual packages and each section is represented by a single box. Generally the pin numbers are written near the wire-lines and often the function is written just inside the box near the wire; if not, the positions of the wires are similar for similar units. Letters like (HY) near each box or gate indicates to me where the unit is located, as row H, column Y on the circuit board, to help me trouble-shoot. Generally I leave out of the diagram a 0.01 microfarad capacitor which I ALWAYS solder across the 5 volt power line to ground., right at every chip. These cards are intended for standard logic with three long lands that are for ground, and four others for +5 V. Lines going to square boxes with letters in them or numbers up to 22 denote edge connections, I use A for +5, and B and Z for ground. It is good practice to connect the distant ends of ground and + 5 together with cross-wires, and connect them with .01 microfarad capacitors, and ground the grounds B and Z to the cage, and I always do. Lines going to circles with numbers inside them denote connections to the ribbon connector.

The 555 chip generates pulses at a rate determined by a variable resistor on the front panel and the falling edge of this triggers the one-shot (EX), whose length is determined by another variable resistor. The output of this is the test stroe. It, and the Varian strobe input via a BNC connector go to a 74253 data selector (lower right part of figure) that selects which strobe to use as selected by the panel switch.

The selected +strobe (Varian strobe or on-board generated test strobe) is convert via logic to a strobe which drives the vacuum control line and turns on the vacuum (upper left, to ribbon line (8). Note however that the vacuum relays are normally open so this strobe. In fact only these vacuum, normally open, solenoid relays, of the four, have current running through them most of the time, to keep them closed (because they are "normally open"; normal means, in this case, with no current running through them strobe must turn *off* the current these relays is up during the selected). MRC explains why I do this.

A minor point is that this current heats up these relays, which could shorten the life of their coils or internal parts. For this reason I inserted a 3 ohm resistor between the "plus" line of the solid state relay R_1 and the + 12V supply, to decrease the steady current through these solenoid valves; the valves only need about 1/2 of their stated current to stay closed once they are closed. But to assure that the valves actually close, it is desirable to apply the full voltage to them just at the beginning of closed period, and doing so results in faster closing at this time which is the most crucial of the sequence. I do this by use of the 0.0048 farad capacitor connected from the plus terminal of the solid-state relay R_1 to ground, so that the capacitor charges during the time (~0,1 sec or more) that the valve is open (and the solid state relay is off, and the vacuum is raising the sample or keeping it up), and then when the solid-state relay closes full voltage (12 volts) is applied to the solenoid, and then decays to about 8 volts in about xx milliseconds. The resistor and capacitor are mounted on a spare location in back of one of the pressure regulators, because the capacitor is too large to fit in the Vector cage.

Now going forward in time, the negative edge of the strobe goes to the "A" input of the next one-shot and turns it on for a time controlled by a front panel potentiometer mentioned above. This applies a negative strobe to the high-pressure control line and turns on the high pressure for only a short time (~15 msec) depending on the fron t panel potentiometer setting.

The end of this strobe turns on another one shot that controls the low pressure time, set by a screwdriver-adjusted potentiometer typically to about 0.3 sec. It has to be longer than the "doptime" (see MRC).

Finally the end of this strobe turns on the last solenoid relay that opens the manifold to atmosphere for about 0.3 sec. Three very similar steps.

This board also generates two other important signals:

1.The "rear scope trigger" goes to an edge terminal (11) and then to a BNC coax connector on the rear, to trigger the storage oscilloscope sweep. It is identical to whichever strobe is being used, the internal test strobe ore the Varian-generated strobe. A TTL amplifier isolates this output. See below under "pressure monitor". This signal also goes to the Veeder Root counter that tells the number of cycles that the system has done.

2. The Helmholz coil used for very low field measurements, and located on top of the magnet, can get hot if it is run all the time. So we turn it off during most of the time that the selected strobe is off (down) and the sample is at the center of the magnet. This can be done by using the selected strobe directly (passing through the TTL inverter at (FY) and edge pin (22) to a coax connector on the rear of the Vector cage because the Helmholz circuit is turned on by a negative strobe. However, there is a delay between the time the vacuum is turned off, and the same time that

the high pressure is actually applied to the sample because of delays in turning on the solenoids, then a delay in actually opening the solenoid valves, and finally a delay in filling the shuttle tube with high pressure. We do not want the field to start turning off before the sample starts to leave the upper position at the stop tube. So the time at which the Helmholz coil is turned off is delayed by "stretching" the strobe that controls the coil, by about 0.1 sec. One-shot (HY) does this; it is triggered by the falling edge of the selected strobe and turns on for aboput 0.1 sec. Its positive output is "Nor'd" by use of the second inverter connected to the output to lengthen the output at the rear connector. It is necessary to check with an oscilloscope to see if the delay in turn-off is as long as needed, and also that the current through the Helmholz coil is on by the time that the sample is at the stop tube.

Helmholz coil. Fig (EC) shows the entire electronics of the coil. Basically, a solid state relay turns it on under control of the output just described. It is in series with the connection of the Helmholz coil to a large power supply which is set to regulate current. The size of the current is determined by the Varian computer under control of the NMR operator.

Staring at the right side of Fig. (EC), 8 lines of a binary representation of the user-entered parameter "cur" are presented by use of simple commands (see Varian user programming manual, which does not tell you that the most significant bit of the number that appears is on pin 1 and not pin 8 as I initially assume!). This is a number from 1 to 255 (if cur is zero the program is written to turn off the shuttle strobe entirely, and a high field R_1 run results). The current in the Helmholz coil, if it is on, is supposed to be proportional to this number, and the system is adjusted so that if cur is nearly 255, the Helmholz coil exactly cancels the main magnet's fringe field. We determine exactly what this number is by using a Hall magnetic field probe and seeing at what number of "cur" the field in the center of the coil is zero. The number cur is fed directly into the 8 most significant digit inputs of a digital-toanalog converter, while the four least significant inputs are set to zero. The output of the converter is then proportional to Cur and equals about 1 volt when cur = 255. This voltage is fed via a cable to the input of the power supply that controls its output current when it is in the voltage-to-current operating mode. The power supply is an excellent 0-10A 0-50V Kepco supply. We mounted a toggle switch on the top back of the supply to switch from the external-voltage-control, constant current mode, to the normal mode using the front panel controls. THE FRONT PANEL CONTROLS HAVE TO BE TURNEDTO THEIR MAXIMUM VALUES when the supply is externally controlled (at least for this power supply). More precisely, the current control should be all the way on (clockwise) but the voltage control should be set very slightly below 50 v or the supply mat turns itself off and will have to be turned on.). You will have to consult the manual of the particular power supply that you have, to learn how to wire this constant current mode; and be sure that you can get or buy a power supply that regulates current; high speed is not needed.

In theory the output circuit (left side of Fig. (EC)) does not have to be grounded anywhere but it probably works best if the + output of the supply is grounded, as it is. A back-biased diode is connected across the Helmholz coil which conducts no current and normally does nothing, but when the magnet is switched off suddenly by the solid state relay, a larger reverse voltage could appear on it and destroy the relay. (This diode may not be needed, but it is hard to get practical information on these relays. Aside from this, the main thing is to connect the input and output voltages with the right polarity This must be a DC-to DC relay, be sure not to get one for AC output. Also, one with plenty of voltage and current capabitiy, and mount it on a heat sink or panel). A series "shunt" resistor connected to a digital voltmeter ("DVM") monitors the current through the coil and is not used much in running; it was mainly used initially, to verify that the current output was proportional to the parameter "cur".

The control voltage (off, or zero volts, to turn on the current) comes from a coax connector on the back of the Vector cage, as described already at the end of the last section. The pull-up voltage is supplied by a little modular supply of the type used to supply portable electronic consumer devices. I had some trouble with this and had to put a resistor to the + voltage in order to turn off the current completely; perhaps the 7407 driver does not turn off completely.

Slightly exotic circuitry and mechanical design is used to have the negative output of the A/D converter floated at the ground of the Kepco supply, to reduce "ground loop" problems. This is suggested by the "earth" symbols, a horizontal line with cross hatches /// below it. Consult an engineer who understands about ground loops.

Yet a third smaller Bud aluminum rack sits on a dolly made of heavy plywood and casters, and holds the Kepco supply and a medium-size rack panel on which is mounted the other things shown in fig. (EC). This rack is not wheeled into the lab except if very low field measurements are planned.

Pressure monitor. The time-course of the pressure during the field cycle is monitored using an excellent and inexpensive 60 MHz Tektronix digital storage oscilloscope. Its horizontal sweep is triggered from the selected strobe ("rear scope trigger", usually its trailing edge so we can see the key part of the sequence at the end of the sample's visit to low field. We have described how the output of the silicon pressure sensor is shipped via the trough to the table's relay rack. This signal could be fed directly into the oscilloscope's vertical input, but this is inconvenient when looking at small pressure changes because zero voltage directly from the silicon pressure gauge is indicated by +3.5 volts. This is avoided by connecting three forward-biased small diodes to this point, whose other (base) end goes via a 22K resistor to -15 volts supplied by the Vector cage's supply. The a BNC connector on a panel that goes to the oscilloscope vertical input is connected between the diodes and the resistor to give about zero volts with zero pressure.

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Power supply. The +-15v, +5 V supply is a small unit inside the Vector cage. A 12 V 5A open supply sits of the bottom shelf of the lab cart on the end furthest from the 500 magnet. This shelf carries the Helmholz coil in transit also. The middle shelf carries the shuttle tube and all other long pieces and miscellaneous junk. The trough and a professional-grade 6 foot ladder have to be hand carried. The voltage of 12 volts was selected for invalid reasons; 24 volts is probably better; 24 volt valves are more standard and easier to get.

Fringe-field calibration. See MRC. Fig. (ED) shows the operational amplifier circuit used. It is powered by two 9 volt batteries in the same box. Probably the coil wire and number of turns are not critical. It was wound onto a small brass spool formed by soldering 3 annular brass cylinders, all 1” diameter with a hole through which passed a 3/8” dia.brass tube about 1.3 meters long. Two of the discs were soldered at the bottom about 1 cm apart to form a spool for the wire; the third was about halfway up the rod as a guide. The outside of the coil of wire is about 20 mm dia and it is probably #30 wire.

After tests using stir bars, we (Dan Miner and I) inserted the coil in the magnet (with the Varian upper stack removed and no probe) and the reset button was pushed to zero the output voltage, and subsequently the integrators drift control was adjusted continuously. Then the coil was moved to different heights at different speeds and a good speed was found, fast enough to allow us to estimate the voltage change, but not too fast to give non-reproducible results. The latter may occur if the induced voltage is so high that the op-amp output is overloaded. Then we recorded a series of voltages with displacements from the center of the magnet going up, and also from the top of the magnet (at the upper edge of the uppermost flange that is permanently part of the magnet) and especially between these points. The latter voltage should correspond very accurately to 11.75 T, to allow us to convert the voltages to gauss intervals. Dan Miner then took the deflections and fitted them with a spline-fitting program to generate the graphs (Figs EF and EG) which we use to set the height of the upper stop (see part I).

This calibration has been used for our three papers published so far, and I am not worried about the calibration, because the relaxation rates do not vary strongly except at fields below about 2 Tesla, where errors in the calibration may be small. However, as this is being written I am trying to check this calibration by field-cycling NMR at about 4 Tesla, and at lower fields by use of Hall magnetometers calibrated using a teaching NMR setup (Teachspin).

Future sections. I did not write a sections on the modifications we make to standard Varian library sequences. There is some information in an earlier report. But hey, no one else has this machine so there's no hurry, and it is easy. I will also write more on measurement of the fringe field when I have done it better.

I have been trying variations of the upper plug, making it nearly half as long, and eliminating the groove at the top. It is now made with only a cap at the bottom (where the sample is). It is still hollow but we no longer make it with such a thin wall (drilling the inside with a smaller drill), and this makes it easier to make. We n o longer rely on the fact that the plug will float; the wire at the top makes this unnecessary, although it is good to have the plug as light as possible. The end sections are made to fit the NMR tubes as closely as possible and are slightly longer. (Frank can machine the clearance with better than 1/2 mil clearance, that's nearly 100 mkicrons.) The clearance in the middle is also a little less. The flats at the bottom are still used. Probably we will go back to a longer plug, or some compromise length. All this is to reduce sloshing and bubbles still more. All the above is being tried with 5 mm tubes. I am also experimenting with 5-minute epoxy for cementing in the plug, and also for cementing the tube in the adapter. This may allow a lead of only 4 hours between sealing and running, rather than 18 hours. This epoxy turns out to be less viscous as well as setting fast. The theory is that if the epoxy and sample meet only in the close clearance at the top, they will not mix much before the epoxy sets in 5 minutes. When I have more experience I will revise the relevant sections above.