Sample mounting for magnetic measurements

First, a note about cleanliness. I will motivate this with a simple calculation to answer this question: how much magnetic iron is required to give us 10^{-8} emu signal (the noise floor of the MPMS-3 system)?

Saturation magnetization density for α-Fe M_sat = 217 emu/g

Mass of α-Fe to create 10^{-8} emu: m = 10^{-8} emu / M_sat = **$4x10^{-11}$ g**

That could be just one grain of a powder sample! Please follow these procedures to encapsulate your samples properly BEFORE coming to the spintronics lab.

Please use only the tools in the drawer marked "MPMS-3 ONLY sample mounting": these are ceramic and to be kept clean so that we do not impart magnetic impurities in our samples. This article very nicely demonstrates how magnetic impurities can lead to spurious results and bogus conclusions about material properties– we don't want this to happen to you!!

An example of typical magnetic impurities is shown in graph below, which compares two long (10cm) pieces of Kapton tape, one clean and one which was drgged through dust to intentionally collect a lot of it. Note the almost reversible M(H) curve and saturation of the impurities around 5000 Oe: you will see them again!

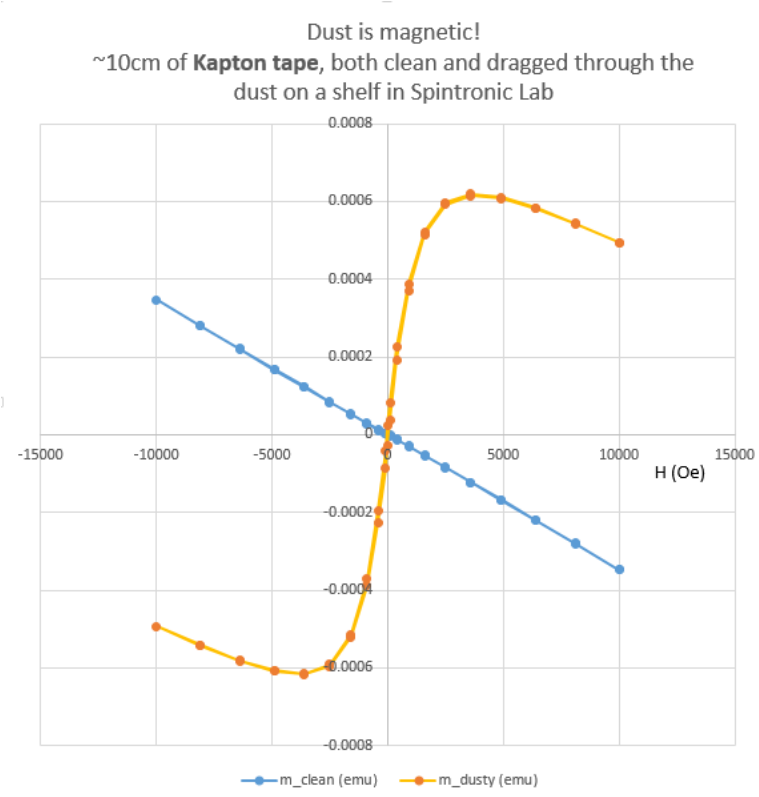
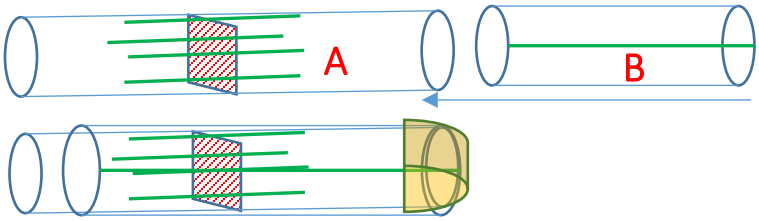


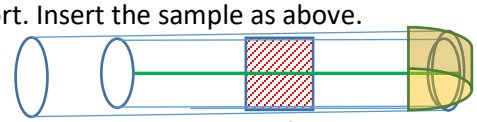
Figure 1: dust is magnetic

Thin Films or bulk samples: using straws

Straw Adapter (4084-814, yellow in picture above) and drinking straws (8000-001) are for use with MPMS-3, ACMS-II, and VSM Large Bore coils only (not for use with VSM standard 6mm bore coils). For use on the PPMS-VSM, cut the straw (**A** above) to **83mm length**, while on the MPMS-3 it should be **150mm long**. There is also a 2 part adapter version which works by pushing the straw over the barb of the main body (just like in older straw barb adapters from QD) and then screwing the collar down to retain the straw. Use straws for the following samples:



- Films perpendicular to field:
  - diagonal size = 5.8mm (e.g., 4.1 x 4.1mm square film); this is best done by placing the film near the recommended sample offset (66mm MPMS-3) for and using two clean applicators (wood handles of Q-tips, or clean glass rods stored in MPMS-3 kit) from each end of straw **A** to turn it sideways and jam it into the straw material so that it does not move.
  - (pictured above) If the film is larger (5x5mm), make 4 vertical slits (green lines above) of ~1 cm length in the main straw **A** (using the CLEAN ceramic blade stored in sample mounting drawer) which is centered at 66mm offset. Get another fresh straw out of the box, cut to 130mm, and slit this straw **B** along its entire length, then wrap it outside the main straw for support. Insert the sample as above.



- Films parallel to the field:
  - width = 6mm; this width will ensure that the film does not slip, but note that if precise vertical alignment of the film is required, the quartz paddle may be preferred.
  - (pictured above) Narrower films or bulk samples: using the same slitted straw **B** mentioned in perpendicular sample mounting, insert in inside main straw, providing a smaller inner diameter for sample mounting. To immobilize the inner straw after it is inserted in the main straw, poke several holes with a pin near the bottom of the assembly so that the holes pass through both the outer and inner straws.

ALWAYS WRAP A ~2cm PIECE OF KAPTON TAPE (yellow piece below) AT BOTTOM OF THE STRAW AS A "SAFETY NET" IN CASE SAMPLE FALLS. Leave a vent to let air escape during purging of chamber.

Films or bulk samples: quartz paddle holder

To adhere samples to the quartz paddle, GE 7031 varnish and tapes are both on-hand in the lab. Measure a background of the holder and adhesive (without sample) – this is especially important in the case of tape which can pick up magnetic dust particles.

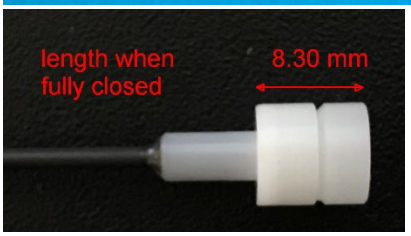
Oven sample mounting (300 – 1000 K)

For samples of thickness 0.5mm and thinner: use the Dry Mount technique described in the placard stored in the VSM oven user kit and also online here .

For samples thicker than 0.5mm or irregular shape which can tolerate water exposure and heat gun treatment: The "wet" technique is described in the VSM oven manual, as well as in these training videos (you'll need a Quantum Design Pharos account for the latter).

Powders can be measured if carefully packed in a separate Cu foil pouch (25um thick foil, cut to 8x10mm, folded and tucked down to 4x4mm), but **WORK WITH NEIL** before doing this since release of powder is a major research disruption. Only use DC Scan Mode due to possibly loose powder inside the Cu pouch.

Liquid samples



We have a couple sealed Delrin (acetal copolymer) buckets from Quantum Design for containing liquids (part # C130D). Work with Neil before using this, as any misuse of the bucket or release of liquid material will be a real mess. Procedure:

1) Sample (red in picture above) is placed in the bucket, some silicone vacuum grease is put on the threads, and the bucket is screwed in TIGHT to seal.
2) To test that it **will not leak** in the MPMS-3 sample chamber: we have a dessicator and vacuum line in the lab which we use as a test bell jar, so put in there on a Kimwipe and observe it does not leak while pumping down (the syringe in there expands at low pressure and is thus a crude vacuum gauge).

3) Due to air gap above sample, we don't move this holder quickly. So, **only DC Scan mode** or **AC measurements** are possible (not the VSM mode!).
4) The background signal from this holder will be significant (expect >10e-5 emu), so you are strongly encouraged to measure the blank holder background before and after your sample.
5) Unscrewing of the slippery bucket may be helped by clamping with a non-magnetic (plastic jawed) tools.
6) If you cannot center your sample by scanning (i.e., if the background signal competes with your sample), then use 56.4mm for the sample offset if the sample is a flat plate sitting on the bottom of the bucket. A taller sample would have a larger offset.
7) Lid to base length is 8.30 mm when fully closed, and can be measured with a clean set of calipers.

Powder samples

These must always be prepared in your own lab so that no powder release happens in the Spin Lab.

The table below should help you decide which method to use for powder containment:

Issue	Al foil pouch	VSM capsules	comments
Moment <1e-4 emu			
Need T < 100 K			
AC susceptibility			Mount capsules in a straw: no metal!
M(T) fitting			VSM capsules M(T) is hard to fit at low T
Need VSM mode			VSM mode has best sensitivity at high fields
Oven mode			Only the Al foils are oven heater compatible

METHOD 1 : aluminum foil pouch, DC Scan measurements

Unless indicated by the table above, I highly recommend to use clean aluminum foil pouches and to measure using DC Scan mode. The reasons are that:

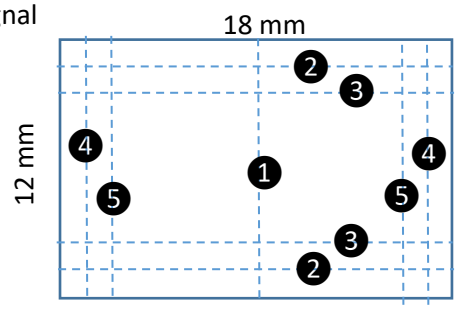
1) background from Al foil is very reproducible: although it is paramagnetic (chi > 0), the M(H) is linear so it is easy to subtract from the data.
2) it is only weakly dependent on temperature, changing only ~18% between 300 K and 2 K. See graph on next page.
3) it is at the same location as the sample so that the total magnetic material signal still looks like a dipole and can be fit by the DC Scan method nicely.

Important note! VSM mode will NOT be safe since the powder will move around and can get out, so you MUST use DC Scan mode.

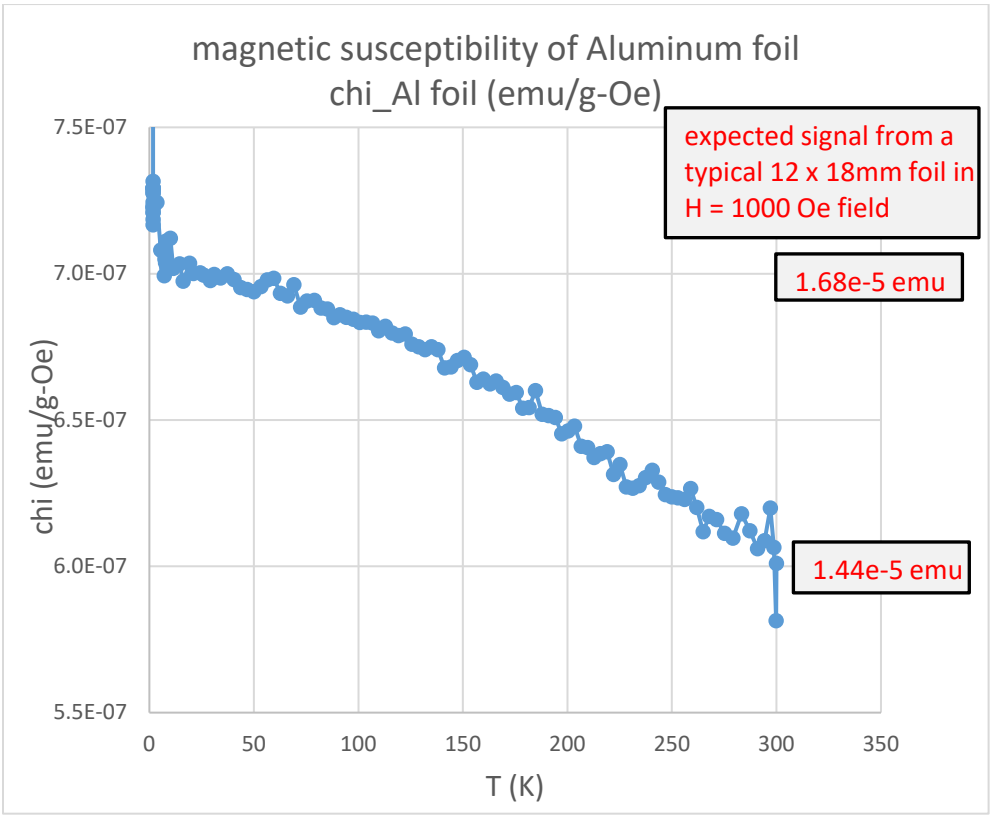
How to make the Al foil pouch for standard mode (T<400 K – see below for oven mode):

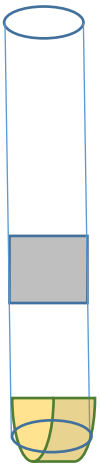
- cut a piece of clean aluminum foil (we use UHV foil in Birck) to approx. 12 x 18 mm using NON-FERROUS tools like ceramic scissors or ceramic blade.
- Clean the surfaces and edges of foil with IPA swab.

- Weigh the pouch so you can normalize the background signal to a previously measured blank Al foil. You still need to regularly make blank Al foil pouch measurements to check your background signals, but I have found that the foil can be cleaned and show consistent susceptibility that matches the literature very well.



- Fold the 12x18 mm foil in half (fold 1 below) so it's now 12 x 9 mm.
- Double-fold (folds 2 and 3) the sides using 1-1.5mm in each fold, using ceramic tweezers. Very important to have good double folds to contain the powder!
- Now you have a cup which you can position in a fixture to hold it upright while you load powder.
- After loading, make sure the powder is not at the top of the opening.
- Double-fold the top down (folds 4 and 5) to contain the powder and gently press the pouch to compress the space a bit, but watch out for punctures in the foil which can result.
- Weigh the full pouch to get sample mass.
- The final size of the pouch should be ~ 6mm x 6mm and should only be able to fit into the straw by compressing the straw to an oval at the opening, and can be pushed up to ~66mm sample offset with a clean swab.
- Test firmness of the grip from the straw by doing a "drop test" of the straw vertically a few inches above the bench: the impact should not move the sample offset any measurable amount.
- Last IMPORTANT step: wrap 2cm length of Kapton tape around the bottom of the straw to catch a falling sample! See diagram below.





How to make the Al foil pouch for OVEN mode (T= 300 to 1000 K):

- Same process as for standard mode, except that the Al foil is 8 x 10 mm, still folded in same manner such that it is <4mm wide when finished.
- Thickness must remain less than ~0.5mm to work with the dry mount ceramic plate.
- As always, use ONLY DC SCAN MODE when using Al foil pouches.
- Special note for oven mode: use scan time = 3.35 sec
    - This is to minimize aliasing effects from the AC heater currents in the oven stick.
    - There is an example sequence in the usual examples folder for this.

METHOD 2: VSM powder capsules, VSM mode

If you need to use VSM mode or AC susceptibility, then powder should be loaded into single-use powder sample holders which we will provide to you (QD part # 4096-388). Currently these are the only containers approved for holding powders. They are two identical parts placed end-to-end to make a captured area. See procedure below. For AC susceptibility, mount this holder inside a straw by bracing it with sideways jammed cut pieces of straws or other non-conducting braces (we don't want to generate eddy currents from conductors in AC field). Ask Neil if you need.

The distance, end-to-end, of two holders pressed together with zero gap (no sample) is **37.50mm**. This can be useful in using calipers to set a blank with a specific gap, as is needed for background subtraction (see below).

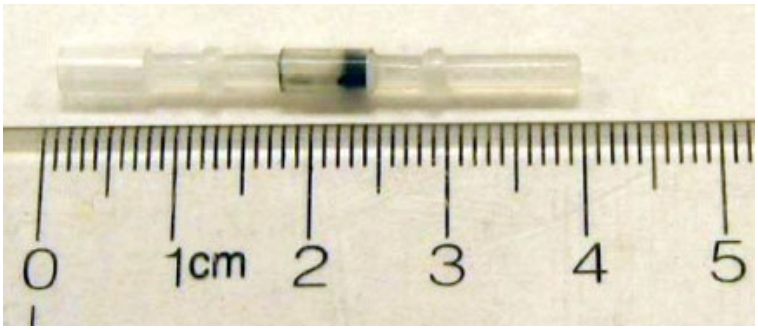


Figure 2: powder holders with powder sample (black) loaded.

Figure 3: powder holder mounted in the brass half-tube. Showing the shorter PPMS VSM brass version here.

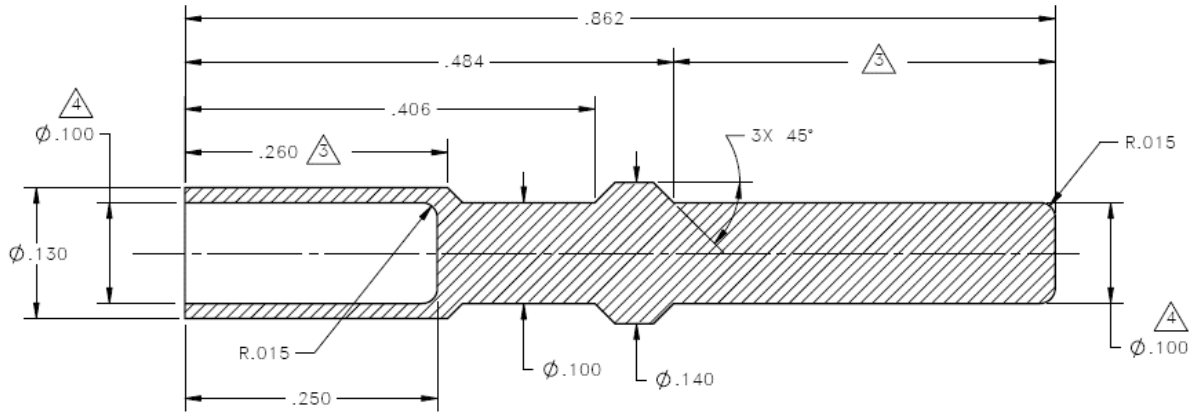


Figure 4: dimensions (in inches) of one half of a powder holder.

Procedure for mounting powder samples

Follow these steps to fully contain the powder material in your lab before coming to the Spin Lab.

- See detailed instructions below if you need more help.
- use personal protective equipment like mask, gloves and eyewear and place a disposable wipe down under your work area.
- Weigh the empty lower half of the powder sample holder.
- Load powder into the bucket of one holder by holding it vertically. Waxy weigh paper, folded to a sharp valley, can be a good way to funnel the powder, and a clean toothpick can push it along.
- Weigh the sample + holder to obtain sample mass.
- Before pressing in the peg, spread a very thin layer of **silicone vacuum grease** on the peg (you can use another grease if it is clear, magnetically clean and low vapor pressure).
- Press in the peg, and then carefully wipe the lip to remove any excess grease and loose powder.
- Put the holder in a container for transport.

Do in the spintronics lab:

- Get the MPMS-3 (“SQUID VSM”) user kit out and locate the specially modified brass holder which has been widened at the top.
- Put it in the mounting station, and insert the powder holder at the widened section.
- Once inserted, push the holder down so the sample is near 66mm offset. Push from the top portion of the holder.
- CAREFUL! Do not let the two sections of the powder holder separate in this process.
- There should be some resistance to movement from the brass holder; this is important to keep them in place while moving and changing temperature in the system.
- Mount on sample rod and insert as usual.
- When done, put back in the mounting station and push up from the bottom portion until easily removable in the widened section.

PLEASE INFORM NEIL OR MIKE IMMEDIATELY IF THERE IS ANY RELEASE OF MATERIAL. The sooner we deal with it, the better for everyone’s research.

Detailed instructions on powder sample mounting

Supplies needed:

- Clean 1109 tissue or similar, to work on
- Scissors
- Scotch tape
- Waxy weigh paper (Glassine or similar)
- Toothpick, or a straw cut to a narrow spatula at end (see pic later)
- Clean and clear vacuum grease (Dow silicone)

Figure 5: supplies needed (left); Glassine sheet (middle); cut a ~5x5cm square out of sheet (right)

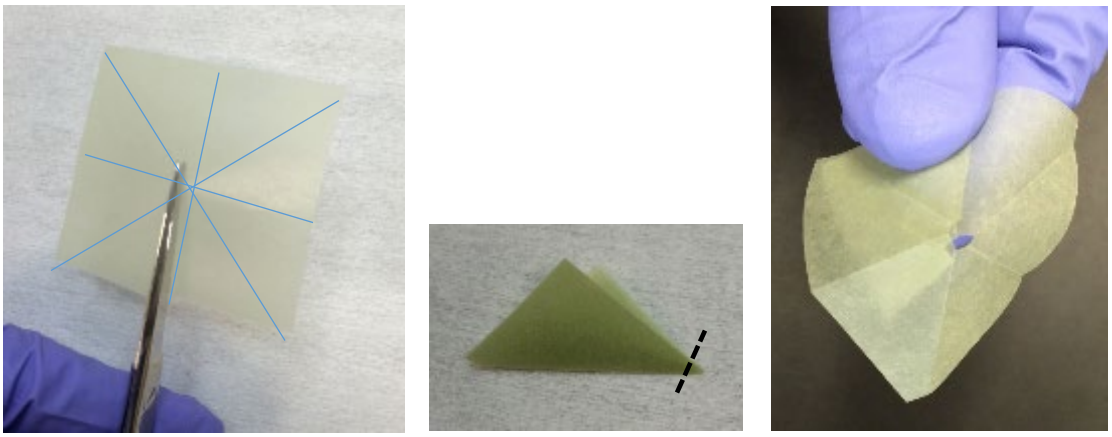


Figure 6: (LEFT) make ONE cut to the middle of sheet; (MIDDLE) fold into 1/8 sections along blue lines shown in left, then cut a SMALL part off the inside tip ~0.5mm (dashed line)to make a hole in center; (RIGHT) unfold to see small hole, and overlap cut edges by 90 deg to make cone. Acknowledgements to Socrates Gomez for this concept.

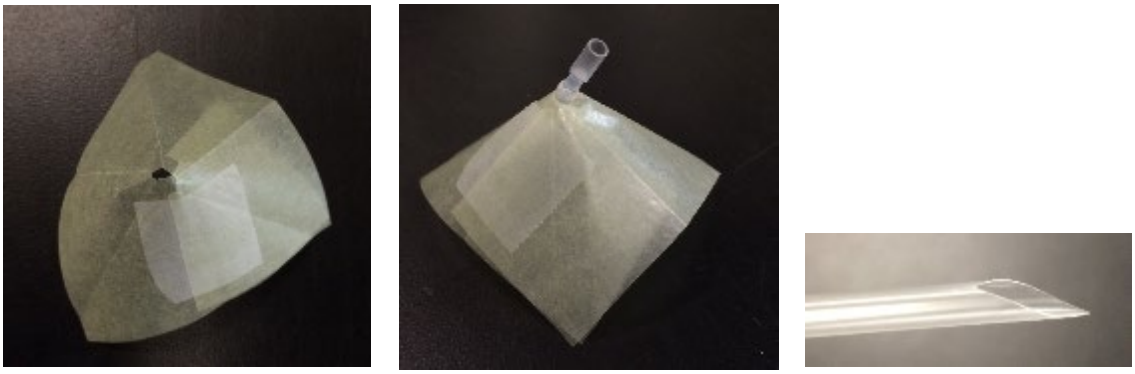


Figure 7: (LEFT) tape the overlapped cut edges; (MIDDLE) a capsule should sit in the hole and not fall; (RIGHT) example of cut straw for spatula. Obtain a tare measurement of capsule + paper using a 0.1mg (or better) sensitivity balance.

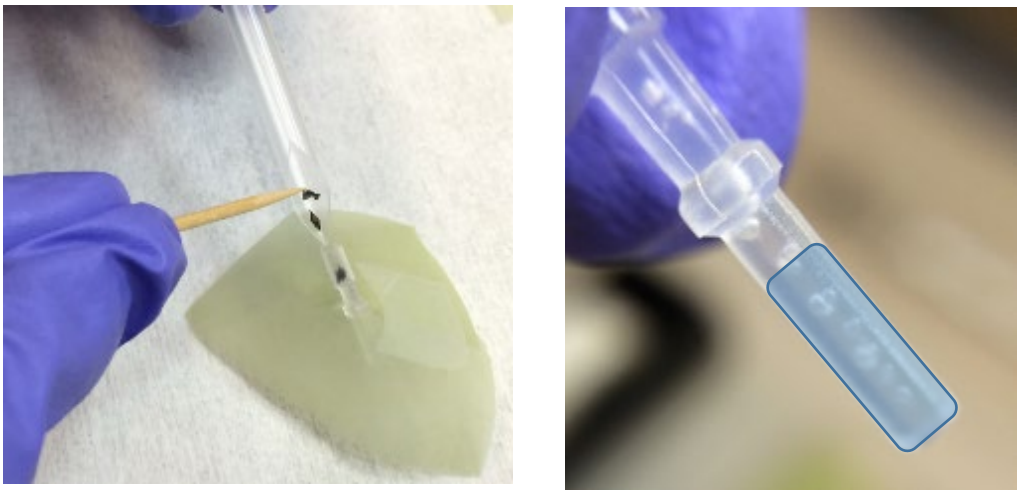


Figure 8: (LEFT) load material into capsule, with help of toothpick. Weigh sample + holder + paper to get sample mass; (RIGHT) coat top capsule with THIN layer of a clean vacuum grease, shown in blue shaded area.

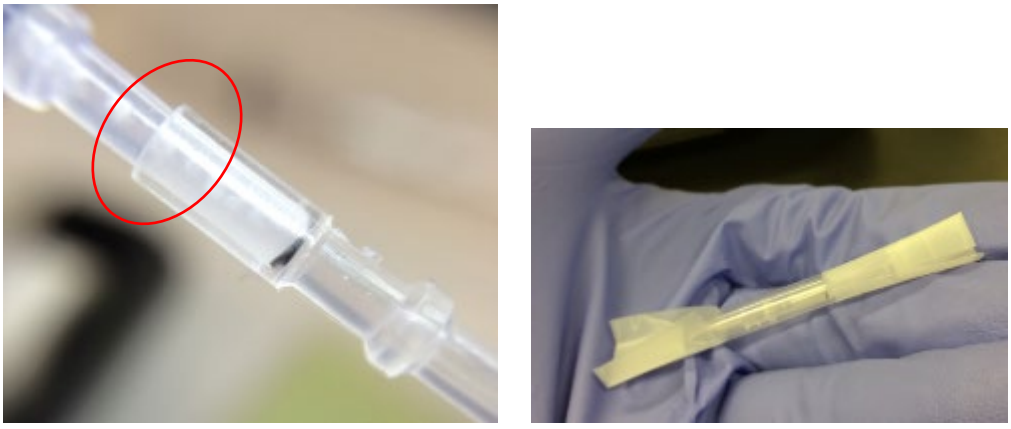


Figure 9: (LEFT) press the two capsules together tightly and wipe up ALL excess grease/powder (red area); (RIGHT) for transporting, put in a clean straw section and tape ends.

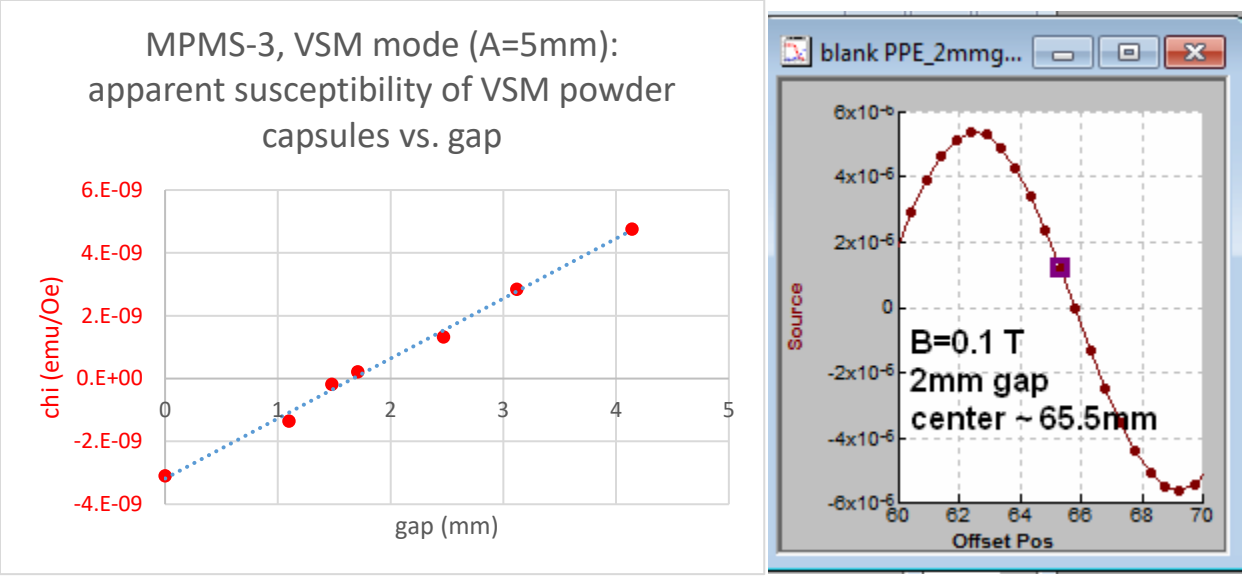


Figure 10: the plastic (Formolene Polypropylene) of the powder holders is diamagnetic but due to the distribution of material, it can have an apparent positive or negative susceptibility as measured using 5mm peak amplitude (A) (LEFT). Note that this means that the moment is simply linear in field. RIGHT: reported moment vs. position varies strongly, here A=5mm and the 2mm gap center was at 65.5mm. This means that an error in identifying sample center results in large error in reported moment.

Loading powder capsules in brass holders, and typical background signals seen

A recently (Nov. 2018) modified "bottom loader" brass holder is slitted at the bottom which allows us to push the powder capsules in from below (instead of the other "widened" brass holders which were spread open near the top). Use a quartz brace (pictured) to hold the shape of the brass holder and also be a catch to things that may slip down.

Background signal of the bare brass holder (using a 66mm sample offset) is shown below to be weak and diamagnetic, with a slight paramagnetic M(T) tail at low T which looks inverted because the impurity is away from the sample location. The saturation moment is ~1e-6 emu in the case we studied. These signals will be holder-dependent, so a "null" measurement with the powder capsules a non-magnetic sample (or no sample) is always advised. As shown in Fig. 10, the apparent signal from the powder capsules is dependent on the gap, so use the same gap in blank and sample measurements.

The powder capsules alone have their plastic diamagnetism which (see Fig. 10) can appear as either a negative or positive susceptibility depending on the gap. In addition, there are paramagnetic impurities present which cause a tail in M(T) but is much larger, m_sat ~ 1e-4 emu evident at low T. Again, measure a blank powder capsule from the same batch to verify the background signal.

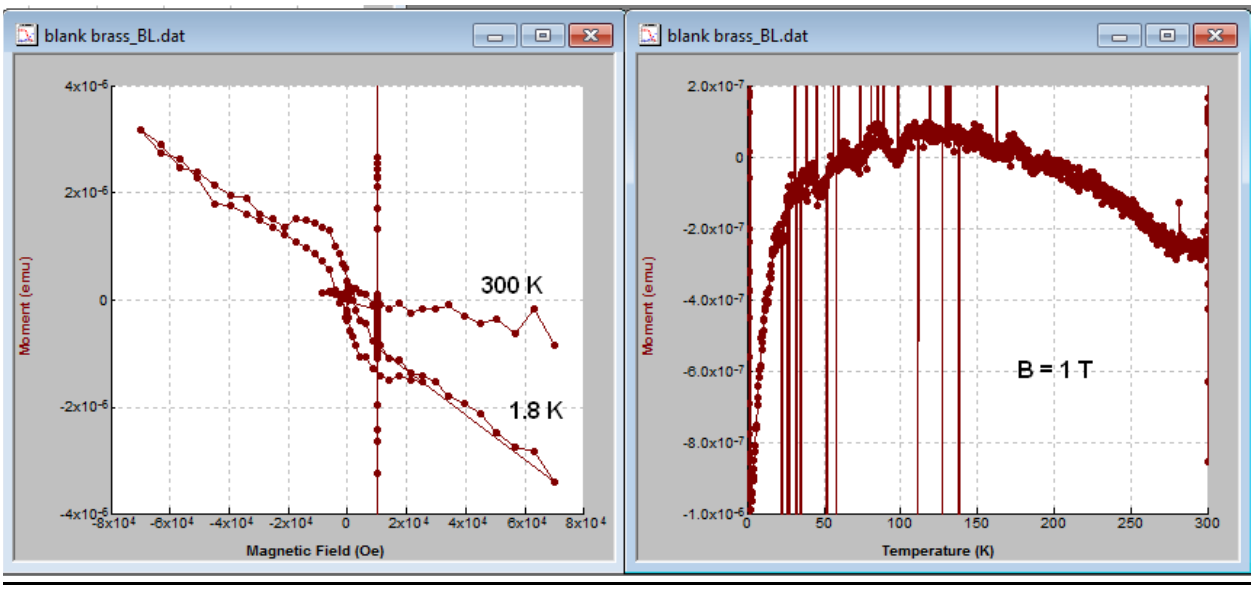


Figure 11: backgruond signal from a blank brass sample holder (the first bottom loader, Nov 2018).

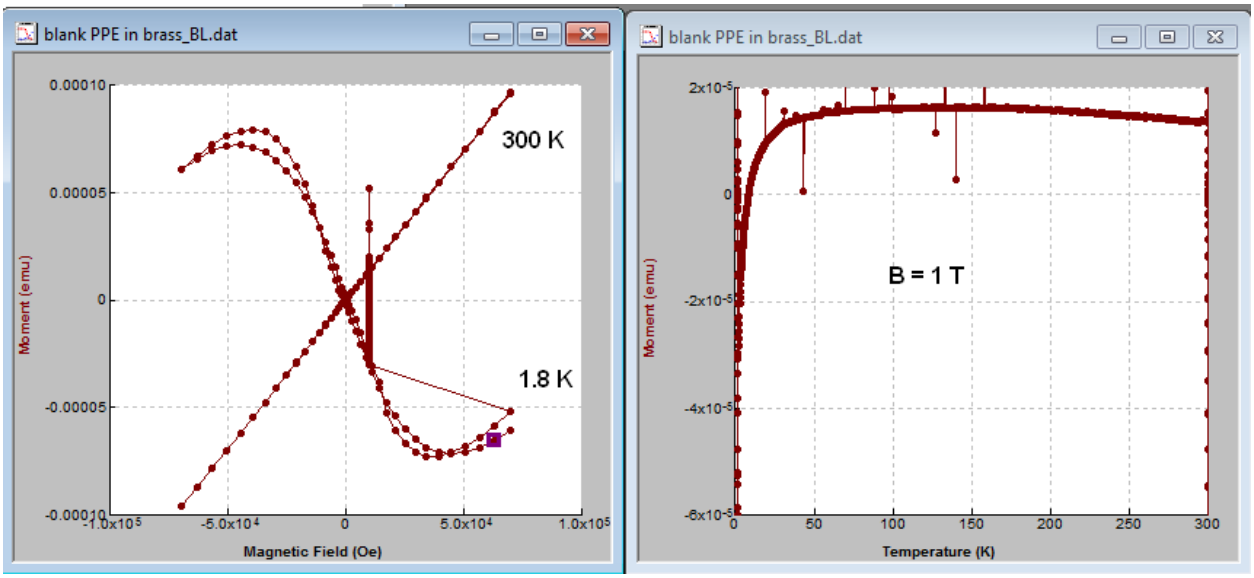


Figure 12: background signal from the same brass holder with two new powder capsules (Nov 2018). Note the large paramagnetic signal at low T. Spikes in M(T) data occur when fast temperature sweep rate (5 K/min) is used.

Further reading

See the VSM sample mounting techniques Application Note here:
http://www.qdusa.com/sitedocs/appNotes/ppms/1096-306.pdf