Quantum Design Japan

High Pressure Cell User Manual

For use with the QD MPMS, MPMS3, PPMS VSM,
VersaLab VSM, and DynaCool VSM systems

CC-Spr-Φ8.5D-MC4
1.3 GPa model
Rev F
July, 2016

PREFACE

P.1 - Introduction

This preface contains the following information:

- Section P.2 discusses the overall scope of the manual
- Section P.3 summarizes the contents of the manual
- Section P.4 illustrates and describes conventions that appear in the manual

P.2 – Scope of manual

This manual discusses using the HMD high pressure cell with the Quantum Design magnetization measurement systems including the following:

- Mounting the sample in the high pressure cell
- Sample measurements
- Manometer measurements to determine the sample pressure
- Using the pressure cell with the QD MPMS, MPMS3, and VSM systems

This manual assumes the user is familiar with making magnetization measurements on QD systems.

P.3 – Contents of the manual

Chapter 1:	Introduction to the high pressure cell
Chapter 2:	Sample insertion, pressurization, and removal from the pressure cell
Chapter 3:	Determination of the sample pressure
Chapter 4:	Mounting pressure cell to various QD sample rods and measurement guidelines
Appendix A:	List of parts included with the HMD high pressure cell kit
Appendix B:	Preparation of the sample teflon tube and sample insertion
Appendix C:	Pressure cell assembly
Appendix D:	Pressurization process

P.4 – Conventions in the manual

Note of caution, warning, or other information to pay careful attention to.

CHAPTER 1 – HMD high pressure cell introduction

Introduction to the HMD high pressure cell

The HMD cell is an easy to use Beryllium Copper (BeCu) cylindrical cell for pressurizing samples for use in the Quantum Design MPMS, MPMS3, PPMS VSM, VersaLab VSM, and DynaCool systems. See Appendix A for a complete list of parts included in the high pressure cell kit.

⚠ If using the VSM transport in VSM mode for the PPMS, or VersaLab platforms, pay careful attention to the warnings on page 21 under the VSM transport section and repeated here.

- The maximum allowed VSM amplitude is 1.2 mm.
- The high vacuum mode must not be used if making VSM measurements with the pressure cell in the PPMS or VersaLab platforms.
- If not making measurements, stop the VSM motor to help limit the wear and tear on the VSM motor and other parts of the VSM system.

⚠ If using the MPMS3, the VSM measurement mode must not be used. The MPMS3 with pressure cell supports only DC and AC measurement modes.

Following are some of the features of the HMD cell:

- Maximum applied pressures up to 1.3 GPa
- Up to 2.6mm diameter high pressure region (2.2 mm actual sample space diameter)
- Novel pressurization system that requires no external press
- No copper rings to secure the pressure – ensures easy sample removal and low running costs
- Compatible with DC, RSO, and VSM sample transports

The pressure cell features an all BeCu design in and near the sample region, including the pistons and other parts for sealing the sample in the high pressure region. The use of non-BeCu parts is minimized throughout the pressure cell assembly. This ensures a more uniform magnetic background, making the HMD cell suitable for studying the magnetization of small samples.

The HMD cell comes with both 2.1 and 2.6 mm ID cylinder housings to allow for various sample sizes and configurations. Also included with the standard kit are sufficient consumables to perform about 60 experiments.

An important feature of the cell is an integrated bearing that is built into the pressurization nuts. This prevents the pressurization piston and other parts from rotating during the pressurization process, allowing for easier pressurization and for slightly higher applied pressures compared to traditional BeCu pressure cells.

A second important feature is the use of a teflon sample tube and teflon caps to form the high pressure seal. This insures that the sample removes relatively easily with the included sample removal rod with little risk of damage to any of the pressure cell components.

Additional notes:

- For VSM measurements, the large bore VSM coilset must be used.
- The pressure cell is not compatible with the ACMS II coil set.

CHAPTER 2 – Inserting, pressurizing, and removing the sample

Teflon sample tube preparation and sample insertion

(refer to Appendix B for a schematic outline)

1) After selecting the teflon tube (either 2.1 or 2.6 mm OD), use the included teflon tube cutting fixture to cut a desired length of tube for inserting the sample. The cell kit includes all necessary parts to accommodate either size teflon tube. The diameter of teflon tube depends primarily on the volume of sample to be measured (fig 1).

   A 4.5 to 5.0 mm length of teflon tube is recommended. Slightly longer teflon tubes, up to 7 mm, can be used if desired. However, the maximum pressure might decrease slightly.

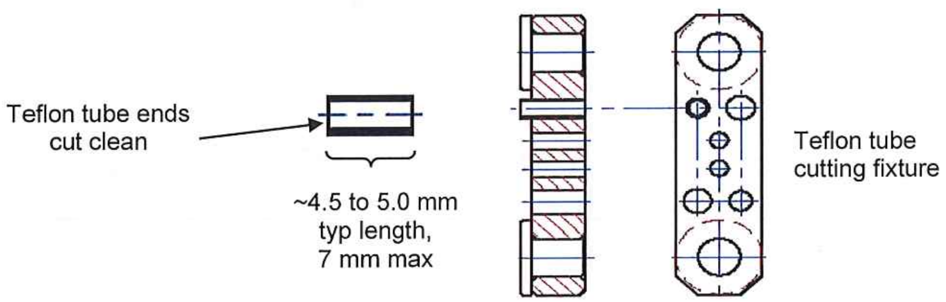


Fig 1 Cutting the teflon tube

⚠ The ends of the teflon tube must be cut clean and perpendicular to the length of the tube. If not, there will be increased chance of damage to the teflon caps and maximum pressure won't be achieved. Use the included teflon tube cutting fixture to help get a clean cut.

2) Seal one end of the teflon sample tube with a teflon cap (fig 2).

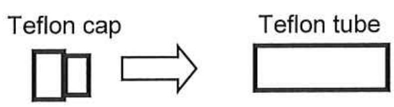


Fig 2 Setting the teflon cap to one end of teflon tube

Prior to using, carefully inspect the teflon caps to verify they are not cracked or otherwise damaged from previous use.

3) Slide the teflon sample tube/teflon cap combination into the center cylinder leading with the open end of the teflon sample tube. Push in with the appropriate diameter sample push rod until the end of the teflon sample tube extends slightly beyond the bevel of the center cylinder (fig 3).

The teflon caps can be difficult to insert into the center cylinder the first several times they are used. For smoother insertion of the teflon caps, pre-install the teflon cap into the center cylinder using the sample push rod. Work the teflon cap back and forth several times until the cap moves smoothly, then remove from the center cylinder.

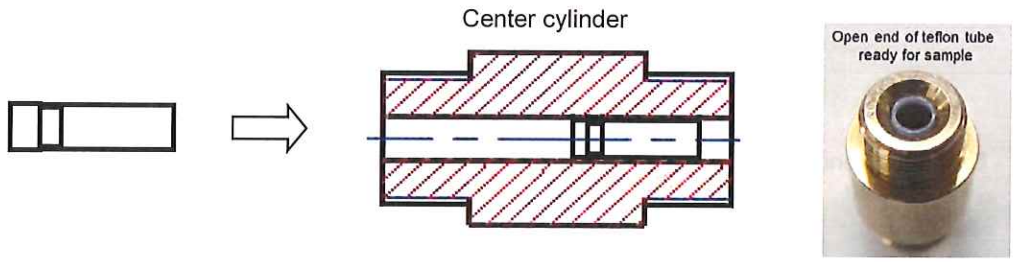


Fig 3 Inserting teflon tube + cap to center cylinder

4) Insert the sample, manometer, and pressure transmitting media into the open end of the teflon sample tube. If using the included Sn or Pb wire for the manometer, about 1mm length is sufficient. Leave enough space at the top of the teflon sample tube to allow the second teflon cap to be inserted (fig 4).

The teflon sample tube should be filled with as much sample as possible, with the amount of pressure transmitting media required to fill the teflon sample tube minimized.

If the filling factor of the manometer and sample is small (perhaps less than 60~70%), achieving 1 GPa will require significantly greater pressure cell compression.

The pressure cell kit includes Daphne 7373 oil as a pressure transmitting media. The included syringe can be used to help apply the transmitting media to the teflon sample tube.

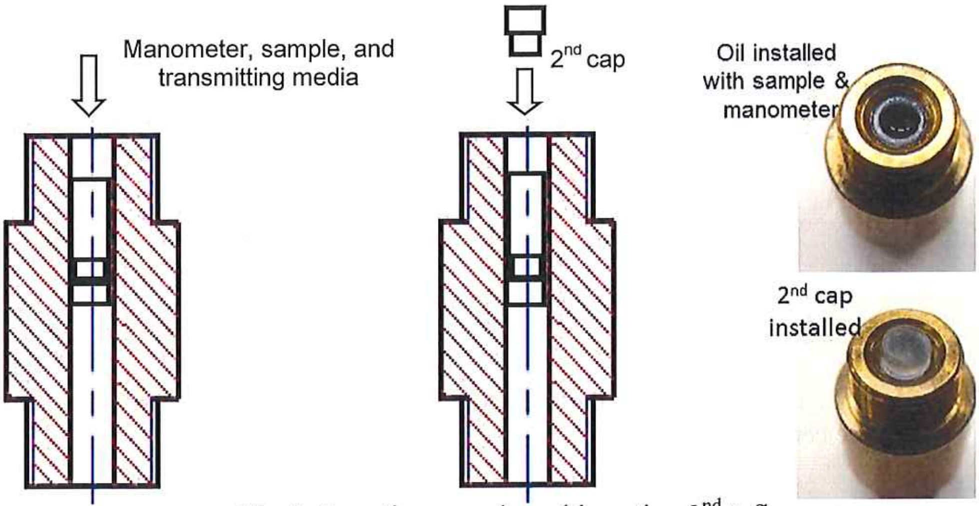


Fig 4 Inserting sample and inserting 2nd teflon cap

5) Seal the open end of the teflon sample tube with a teflon cap (fig 4).

⚠ Care should be taken to minimize any air bubbles in the teflon sample tube.

6) Using for example a Kimwipe, remove any excess oil or any other foreign particles from the threads of the center cylinder and around the teflon caps. Compressed air can also work well for removing any debris from the cell.

Pressure cell assembly

(Refer to Appendix C for a schematic outline of the cell assembly process)

1) Using the appropriate diameter sample push rod, carefully push the teflon sample tube so it is roughly centered within the center cylinder (fig 5).

⚠ The teflon sample tube and end caps form a very snug fit inside the center cylinder. Thus a bit of force may be necessary to push the teflon sample tube into the center cylinder. Gently tapping on the sample push rod can help push the sample to the center of the center cylinder.

2) Clean any excess grease or oil around the center cylinder.

3) Insert the two pistons into the center cylinder (fig 5).

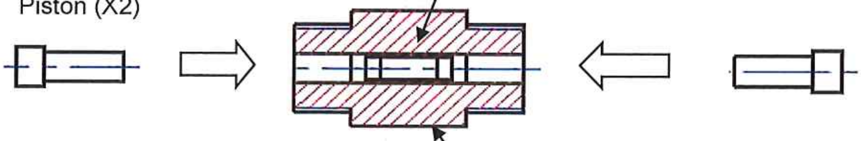


Fig 5 Teflon tube centered and setting the pistons

4) Using a toothpick or small wire, apply a thin coat of teflon powder to the threads of the center cylinder (fig 6).

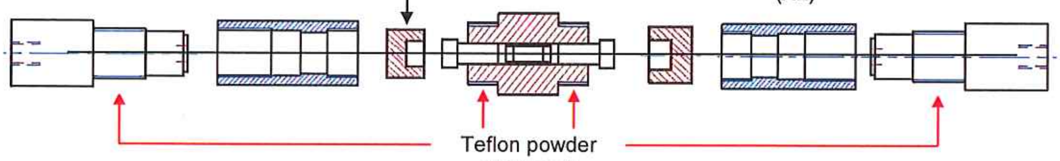


Fig 6 Applying teflon powder, piston backup, side cylinder, and pressurizing nut

5) With the center cylinder held horizontally, place a piston backup on each piston (fig 6).

6) Thread the side cylinders finger tight to the center cylinder. There should be no gap between the side and center cylinders (fig 7).

7) Coat the threads of the two cylinder pressurization nuts with teflon powder (fig 6).

8) Thread the cylinder pressurization nuts to the side cylinders finger tight (fig 6).

9) Loosen one of the pressurization nuts about two turns and tighten the opposite pressurization nut. The nut should tighten smoothly, indicating the pistons and teflon sample tube are moving smoothly.

   Next, loosen the tightened pressurization nut about 3 turns and tighten the opposite pressurization nut. Again, the pressurization nut should tighten smoothly. Work the pistons and teflon sample tube back and forth in this manner about 3 times. When finished, finger-tighten both pressurization nuts so the sample is centered within the center cylinder. This is indicated by noting the gap between the pressurization nut and side cylinder being roughly equal on both sides (fig 7).

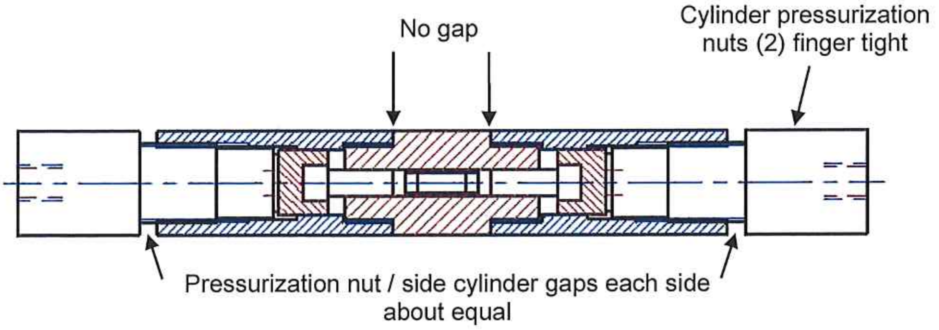


Fig 7 Final assembly before pressurizing

The sample is now inserted to the high pressure cell and is ready for pressurization and measurements. See appendix C for a detailed view of the final cell assembly.

Pressurizing the sample

(refer to Appendix D for a schematic outline of pressurizing the cell)

Prior to pressuring the sample, it is normally recommended to measure the manometer to determine a zero pressure manometer transition reference. See chapter 3 for guidelines on manometer measurements.

Perform the following steps to pressurize the sample.

1) Measure the pressure cell length. This will be used as a reference so the amount of compression can later be determined (fig 8 for the recommended dimension to measure).

2) Insert the pressure cell to the cell clamp. Cinch the pressure cell in place with the two hex screws. Note the third hex screw is used to slightly pry the cell clamp apart in case the cell does not easily slide into the cell clamp (fig 8).

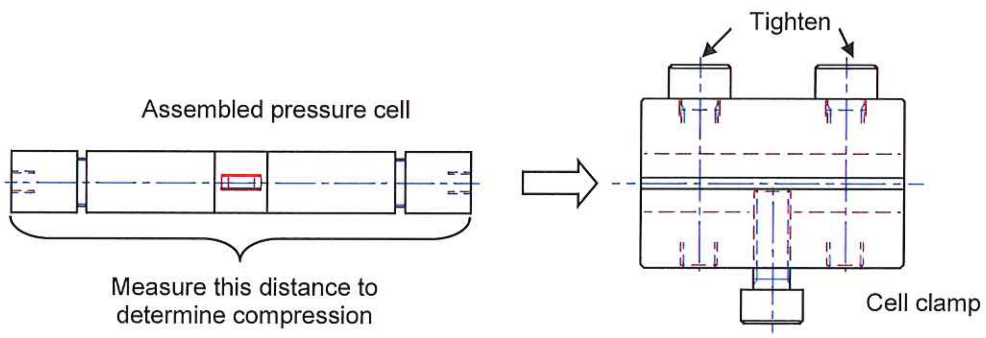


Fig 8 Measuring compression and inserting pressure cell to clamp

3) Attach the custom spanners to the pressurization nuts (fig 9).

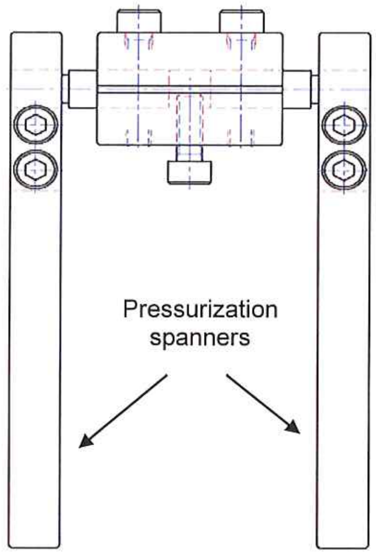


Fig 9 Setting the pressurization spanners

4) Use a vice or large adjustable wrench to hold the cell clamp stable.

5) Tighten one of the pressurization nuts 90 degrees, then the opposite pressurization nut 90 degrees.

⚠ To limit risk of damage to the pressurization nuts due to over torqueing, **slowly** tighten the pressurization nuts.

6) Continue tightening the pressurization nuts, alternating between the two pressurization nuts in 90 degree steps until the total cell compression is about 1 mm.

   For compression greater than about 1 mm, rotate the pressurization nuts in smaller steps, 30 to 45 degrees being recommended.

   Note the maximum sample compression is about 2.0 mm. 90 degree of pressurization nut rotation represents about 0.1mm of compression.

   Be careful about over pressurizing the sample region. If the sample region is compressed more than about 2.2mm, there is increased risk of damage to the pressure cell. Also refer to fig 13 - compressing the sample region more than about 2 mm should not result in significantly higher sample pressure.

   If the pressure is found to be below 1 GPa with 2 mm of compression, this indicates a problem with the cell. The most probable sources of this reduced pressure are:

   - Damaged teflon tube or caps, resulting in a leak
   - Too much pressure transmitting media / not enough sample

   For applied pressures above 1 GPa, the pistons will likely be deformed. For these higher pressures, the pistons are considered a single-use consumable. The pistons are reusable for applied pressures below 1 GPa.

7) If using the MPMS DC transport, once the desired compression is applied, thread on the pressure cell extensions (fig 10). If using the RSO, VSM (PPMS, VersaLab, or DynaCool), or MPMS3 sample transports, the cell extensions should not be attached. See chapter 4 for notes on using the RSO, PPMS VSM, and MPMS3 sample transports.

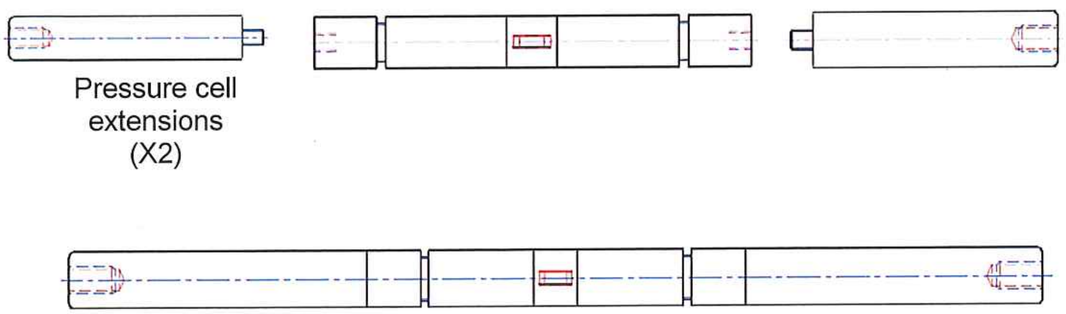


Fig 10 Applying cell extensions

Removing the sample

1) Insert the cell into the cell clamp and tighten the two hex bolts to cinch the cell in the cell clamp.

2) Attach the pressurization spanners to the pressurization nuts.

3) Slowly unscrew the pressurization nuts and remove from the cell.

4) Unthread the side cylinders. If they can't be loosened by hand, use the pressurization spanners to grip the side cylinders.

5) Remove the piston backups and pistons.

⚠ If the applied pressure was greater than about 1 GPa, the pistons may be deformed and wedged in the center cylinder. To remove the pistons in this case, with the center cylinder cinched in one of the pressurization spanners, use some pliers to grip the pistons. Slowly twist the pistons to remove them from the center cylinder.

If the pistons are deformed and do not smoothly insert into the center cylinder, they should be discarded.

6) Using the proper diameter sample push rod, push out the sample from the center cylinder. It may be necessary to gently tap the push rod with something hard to help push out the sample.

Chapter 3 – Determining applied sample pressure

There are two ways to determine the applied sample pressure:

1) Measure the transition temperature of a sample with a known pressure dependence. The pressure cell kit comes with both Pb and Sn wire for this purpose.

   Either AC or DC magnetization can be measured to determine the applied sample pressure. Low frequency (~1 Hz) AC susceptibility measurements will typically give a sharper T_c transition and is recommended if available. See below for details.

2) As the pressurization nuts are tightened, measure the compression of the cell.

Manometer measurements – DC magnetization

The following describes measurements on the MPMS / MPMS XL systems using the DC transport. If using the RSO, VSM, or MPMS3 systems, see chapter 4 for additional information about making measurements including guidelines for measuring the T_c of Pb and Sn.

The superconducting transition temperatures (T_c) for Pb and Sn are 7.19K and 3.72K respectively. However, the measured transition temperature might vary slightly depending on how the transition temperature is defined, for example at the onset of superconductivity or by checking the temperature where the maximum slope of the transition occurs. Note also T_c is suppressed by any applied magnetic field including residual fields.

Use the following guidelines to accurately determine T_c:

1) Use a consistent definition for T_c.

2) Use a constant DC field for all measurements that are used to determine pressure. This is important to get consistent T_c results. For example, a 5 gauss remnant field can result in a shift in the T_c of Sn by about 30 mK, enough to change the apparent sample pressure by about 0.08 GPa.

For Pb, once the shift in T_c has been determined, the corresponding change in sample pressure can be approximated by:

$$dT_c/dP \text{ (K/GPa)} = -0.379 \text{ K/GPa}$$

For Sn, the following can be used:

$$dT_c/dP \text{ (K/GPa)} = -0.489 \text{ K/GPa}$$

Measure the T_c as follows:

1) Apply about a one gauss DC field and center the sample. The best way to accurately set one gauss field is to reset the magnet prior to setting the field.

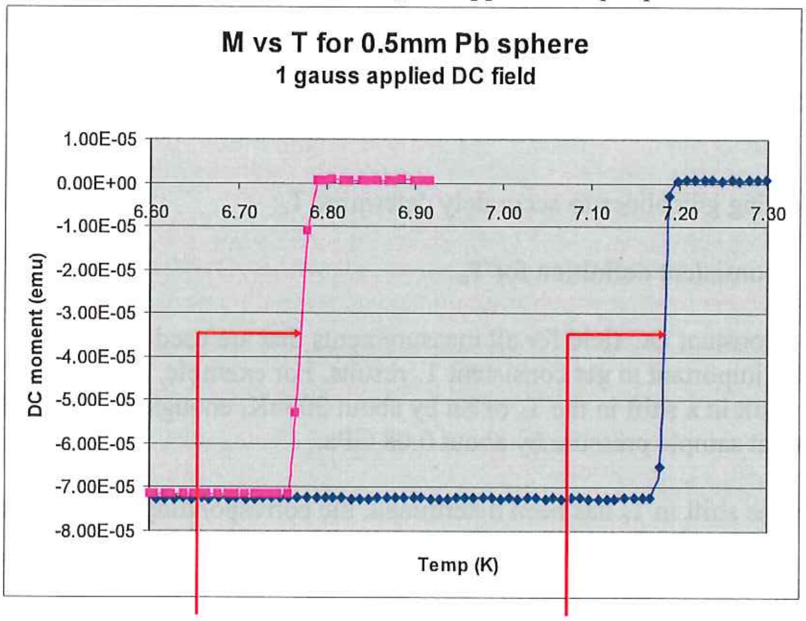
   If the magnet reset option is not available, the DC magnetic field may need to be adjusted until a desired emu is reached. With the supplied manometer in the superconducting state, adjust the magnetic field until the magnetization is in the $-1x10^{-4}$ to $-1x10^{-5}$ emu range.

   If using the MPMS or MPMS3 SQUID systems, look for a smooth symmetrical SQUID response indicated by regression fits over 0.95.

2) Measure M vs T:

   a. For Pb, 6.6 to 7.3 K to cover the entire temperature range of the expected T_c. For tin, measure from 3K to 3.8K.
   b. Temperature step size, 0.02 K
   c. 0.1 K/min
   d. Wait 10 seconds at each temperature prior to measuring

   The following Fig 11 shows typical m_{DC} vs T results for a Pb sample along with an example calculation for estimating the applied sample pressure.



T_c (1.9 mm cell compression) = 6.775 K

T_c (0 pressure) = 7.185K

dT (K) = 0.410 K
dP (GPa) = 0.410 (K) / 0.379 (K/GPa) = 1.081GPa

Fig 11 Determining the applied sample pressure

Manometer measurements – SQUID AC magnetization

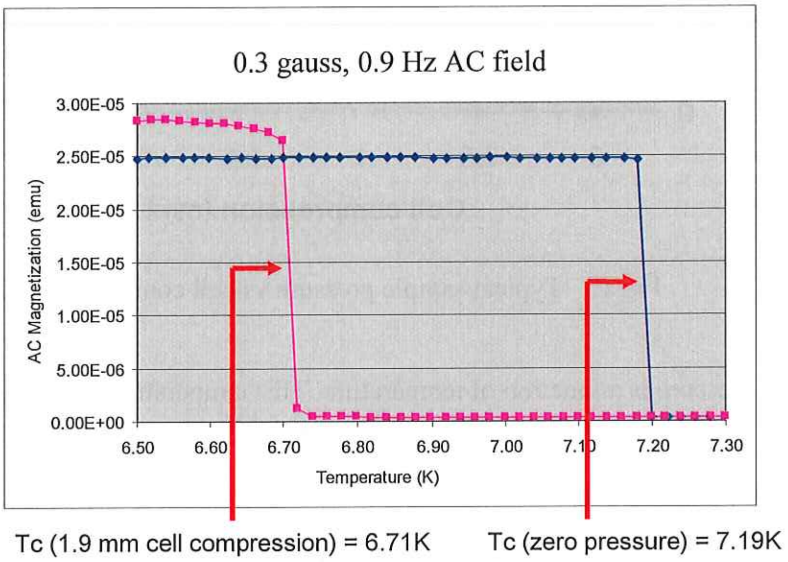
Measure the T_c as follows:

1) Set a small DC field - one gauss is enough, and DC center the sample.

2) Measure m_{ac} vs T:

   a. For Pb, 6.6 to 7.3 K to cover the entire temperature range of the expected T_c
   b. Temperature step size, 0.02 K
   c. 0.1 K/min
   d. Wait 10 seconds at each temperature prior to measuring
   e. AC measurement parameters:
      i. 0.9 Hz wave frequency
      ii. 0.3 gauss AC drive amplitude
      iii. 5 blocks, 2 scans to average

Fig 12 shows a typical m_{ac} vs T result using the supplied manometer. Note the graph shows the magnitude of the AC moment. In some cases depending on the measurement parameters, a bit sharper transition might be seen by plotting the out of phase magnetization (m”) as a function of temperature.



dT (K) = 0.48 K

dP(GPa) = 0.48 (K) / 0.379 (K/GPa) = 1.27 GPa

Fig 12 M_{ac} vs T for Pb and pressure determination

Measuring the pressure cell compression to approximate the pressure

The following fig 13 shows a typical compression vs sample pressure chart for the pressure cell. This will change depending on the length of sample teflon tube and the filling factor of the sample. Assuming these are kept constant, it is possible to approximate the applied pressure by measuring the cell compression.

In the case of fig 13, the pressure was determined by measuring the T_c of a Pb manometer. A 5 mm length of teflon sample tube was used.

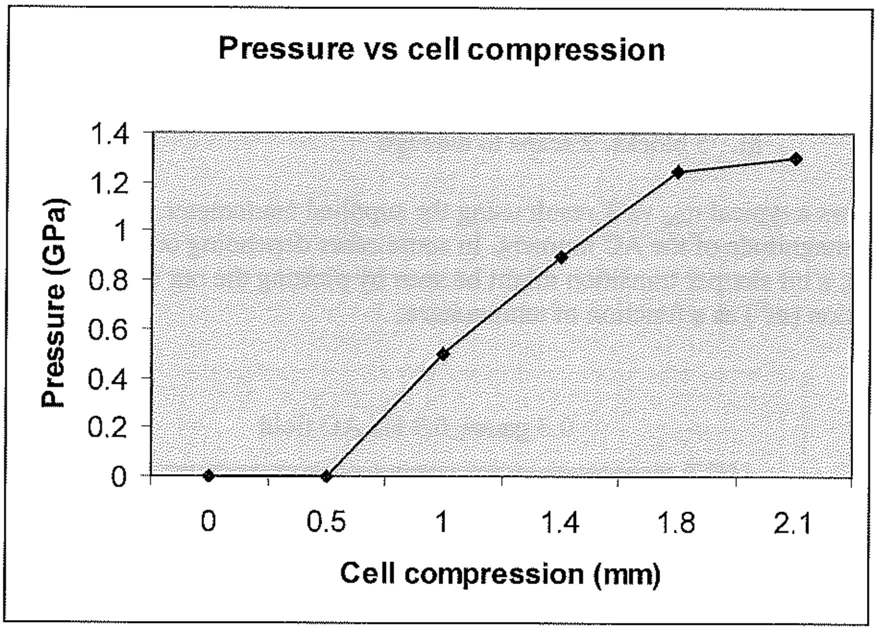


Fig 13 Typical sample pressure vs cell compression, 7K

The sample pressure is a function of temperature. This temperature dependence is negligible for temperatures less than about 70K (liquid N_2 temperature).

For room temperature, samples that show ferromagnetic ordering can be used as a manometer. For example, Gadolinium shows ferromagnetic ordering near 300K and with a pressure dependence on the ordering temperature of 12.2 K/GPa[1].

[1] M. Mito, et al.,J. Phys. Chem. Solids (2009), doi :10.1016/j.jpcs.2009.07.013

Chapter 4 - Mounting pressure cell to various QD sample rods and measurement guidelines

The HMD pressure cell kit includes adaptors for using the DC, RSO, and VSM sample rods per fig 14 below.

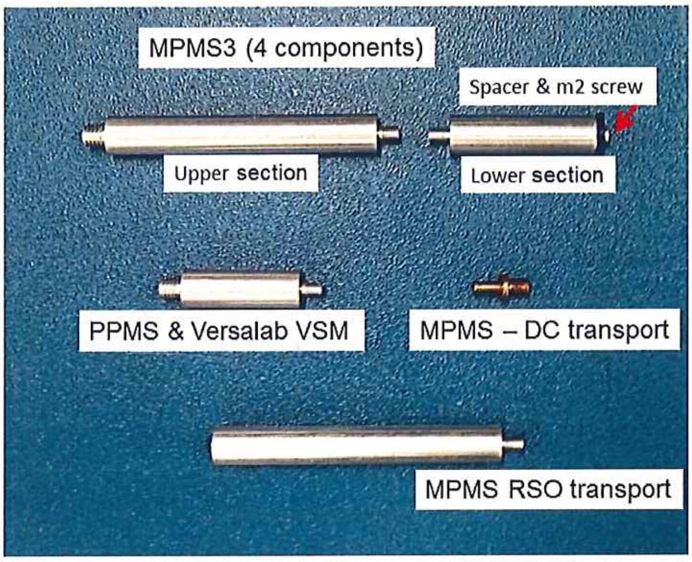


Fig 14 Sample rod adaptors

MPMS - DC transport

The DC transport adaptor is made of BeCu to minimize magnetic background contribution. It may be attached to the bottom of the standard DC sample rod using a piece of copper wire or may be brazed. The pressure cell can then be threaded onto this adaptor. See fig 15a, next page.

The pressure cell diameter is too large to fit within the quartz tube that is part of the slide seal assembly. In this case, the pressure cell should be installed into the MPMS with the airlock valve open as follows:

1) Set the MPMS temperature > 100K.
2) Open the airlock valve.
3) Open the vent valve (Utilities/Diagnostics/Chamber, Vent valve open).
4) Remove the blue sample space plug.
5) Install the pressure cell + sample rod into the MPMS and attach the sample rod to the DC transport as usual.
6) Purge sample space.

The sample should now be ready for centering and measurements. If the slide seal assembly does not have the quartz tube installed, the pressure cell can be installed into the MPMS with the airlock valve closed as usual.

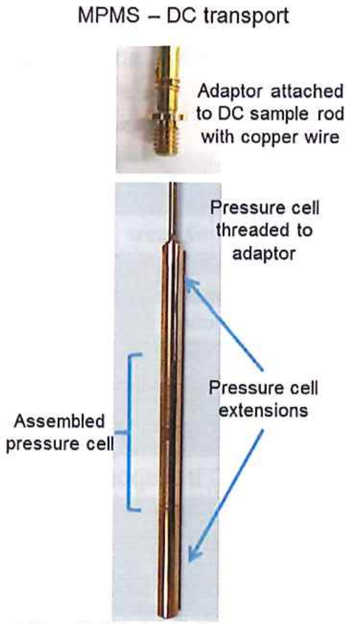


Fig 15a MPMS DC transport

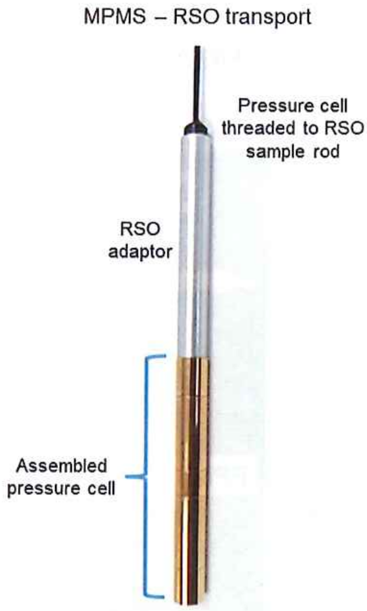


Fig 15b MPMS RSO transport

Fig 15c VSM

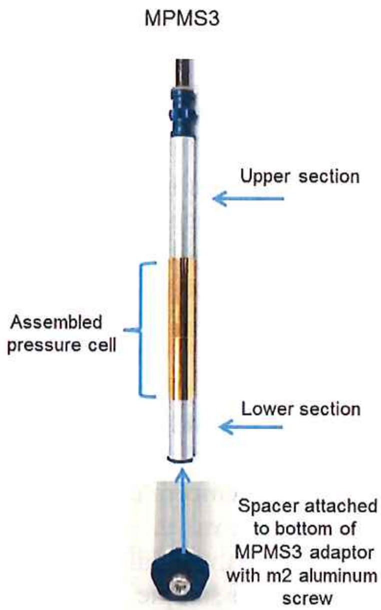


Fig 15d MPMS3

Fig 15 Pressure cell mounted to various sample rods

MPMS - RSO transport

The RSO adaptor is made of aluminum to minimize magnetic background signal as well as minimize the mass of the sample holder. It simply threads onto the standard RSO sample rod. See fig 15b.

Once the pressure cell is threaded onto the RSO adaptor, insert the RSO sample rod into the MPMS as with straw-mounted samples.

Use the following guidelines to measure the T_c of lead or tin:

1) For Pb, measure 6.6 to 7.3 K to cover the entire temperature range of the expected T_c. For tin, measure 3K to 3.8K.
2) Temperature step size, 0.02 K
3) 0.05K/min sweep
4) Measure every 0.02K
5) RSO parameters:

Amplitude	3 cm
Number of Cycles	2
Scans per Measurement	1
Position	Center
Frequency	0.5 Hz

⚠ The mass of the pressure cell adds wear to the RSO transport. The lower RSO drive frequency (0.5 Hz) is thus strongly recommended for all RSO measurements.

VSM transport (PPMS, DynaCool, and VersaLab)

⚠ The large diameter VSM coil is required to use the HMD pressure cell with the QD VSM system.

⚠ The software does not recognize the presence of the pressure cell. Thus it is the user responsibility to take the following precautions to limit the load and stress on the VSM motor in order to reduce the chance of damage to the VSM transport.

For the VSM amplitude parameter, never use an amplitude greater than 1.2mm with the pressure cell installed. Higher amplitudes puts additional stress on the VSM motor which increases the risk of damage.

Do not run the PPMS, Dynacool, or VersaLab in the high vacuum mode if using the VSM with pressure cells. The helium exchange gas is important to help prevent the VSM motor from overheating, limiting the chance of damage to the VSM motor.

⚠ When not collecting emu data, stop the VSM transport vibration to help limit long-term wear and tear on the VSM transport and other mechanical parts of the VSM measurement system.

The same adaptor can be used for the PPMS, VersaLab, and DynoCool systems. Figure 15c shows the pressure cell mounted to the VSM sample rod.

Using the PPMS VSM, measure the T_c of lead as follows:

1) Set PPMS (or DynaCool) temperature to 6K.
2) Once the temperature is stable, set magnetic field to 100 gauss.
3) Set the VSM parameters as follows:

VSM amplitude	1.2 mm
VSM frequency	40 Hz

4) Locate sample.
5) Set the magnetic field so the magnetization of the lead is about $-1x10^{-4}$ to $-1x10^{-5}$ emu. Due to remnant fields in the PPMS magnet, it may be necessary to adjust the magnetic field to get a suitably small signal from lead. Measure M vs. H of lead as follows (see fig 16a for an example of M vs. H for lead for determining remnant field).
   a. 20 to -20 gauss
   b. 2 gauss steps
   c. Magnet charging mode driven
   d. From the resulting M vs. H curve, set the magnetic field such that the magnetization is about $-1x10^{-4}$ to $-1x10^{-5}$ emu.
6) Set 7.5K, 0.1 K/min and plot m vs. T. See figure 16b for example M vs. T for lead.

⚠ In the lower temperature range of VersaLab, there is not a suitable manometer for determining the pressure.

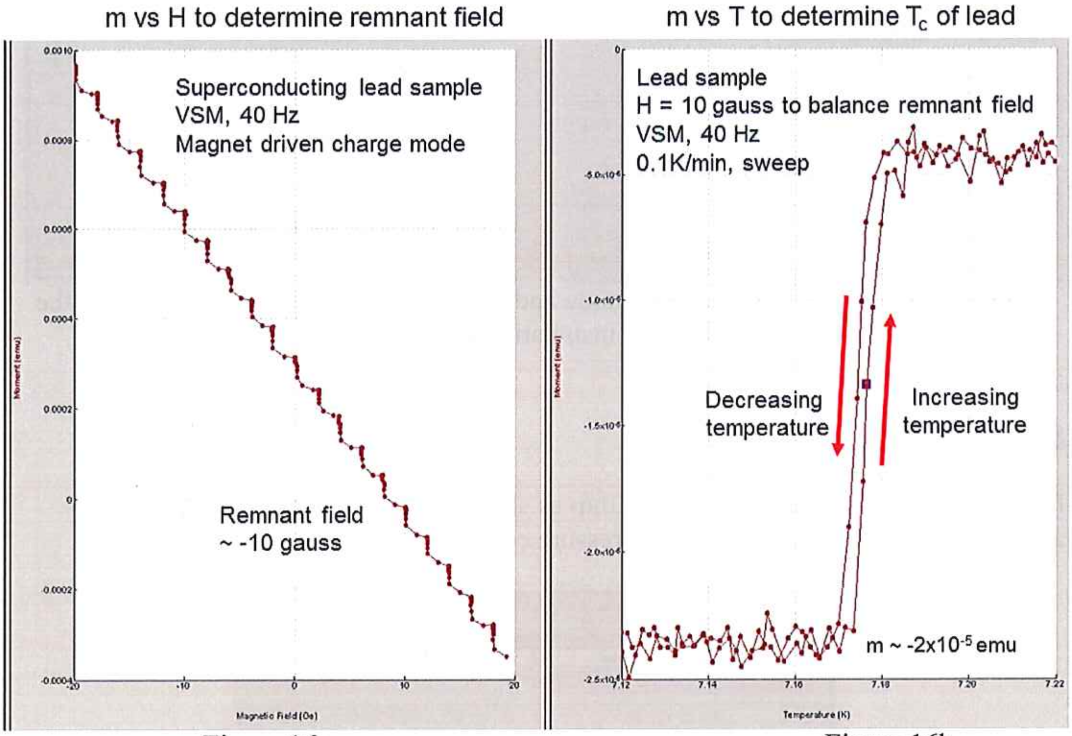


Figure 16a

Figure 16b

Fig 16 Measuring lead to determine remnant field and T_c

VSM measurement guidelines

⚠ The following describes guidelines when using the pressure cell with the VSM transport. It is important to follow these guidelines to limit the risk of damage to the VSM transport and other parts of the VSM system.

Sample Installation:

During the sample installation process, chose "Standard" VSM mode and "Extended Purge" as shown in the below fig 17.

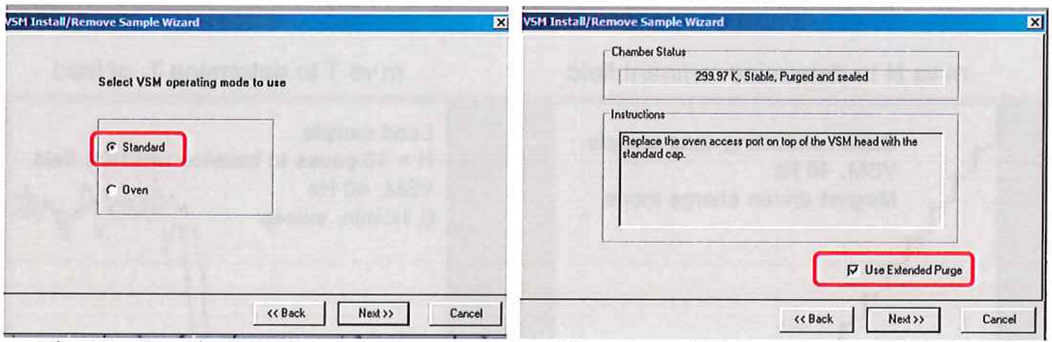


Fig 17 Standard VSM operating mode and Use Extended Purge selected during the sample installation procedure

Sample Centering:

If entering the offset manually, use 35mm as shown below – this will be close to the actual sample offset when using the pressure cell.

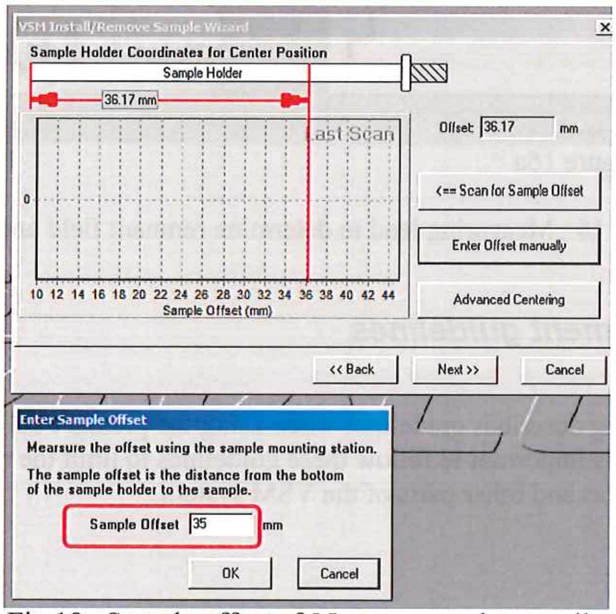


Fig 18 Sample offset of 35 mm entered manually

Scan for Sample Offset centering can also be done.

EMU Measurements:

For immediate measurements performed directly from the VSM Measurement window, set the VSM Peak Amplitude to 1.2mm as shown in fig 19 before selecting the "Start" button.

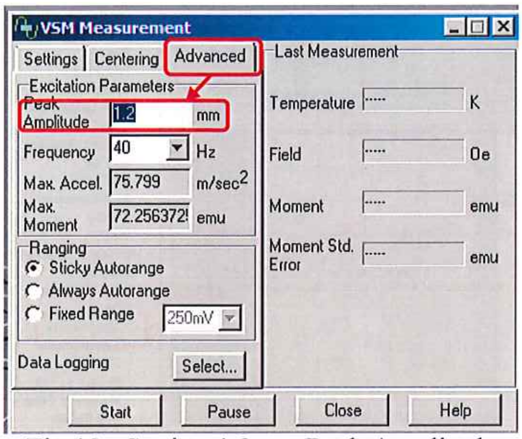


Fig 19 Setting 1.2mm Peak Amplitude

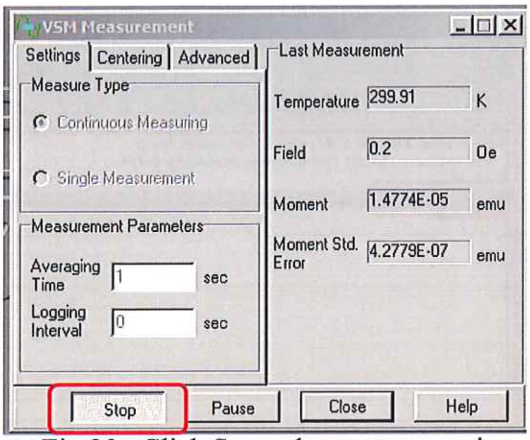


Fig 20 Click Stop when not measuring

When not making measurements, stop the VSM transport vibration by clicking the "Stop" button per fig 20. This helps prevent extended wear on the VSM motor and other parts of the VSM system.

M vs T and M vs H sequence measurements can be done similar to measurements without the pressure cell. As in immediate measurements described above, a maximum 1.2 mm peak amplitude should be used. The example shown in fig 21a & 21b shows a basic M vs T measurement. Note the following:

- Peak Amplitude of 1.2 mm specified in the VSM measure command (fig 21a).
- VSM Measure Stop command at the end of the sequence (fig 21b).

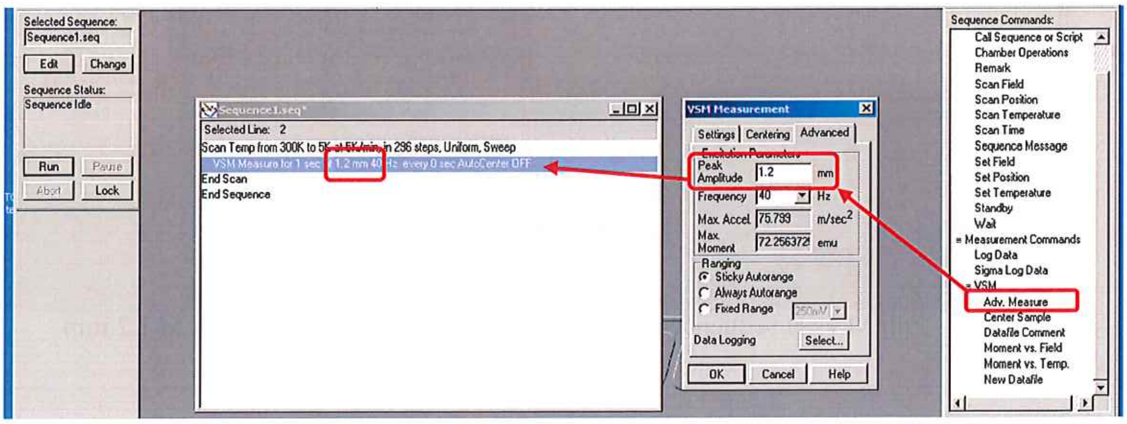


Fig 21a VSM measure command with 1.2 mm Peak Amplitude

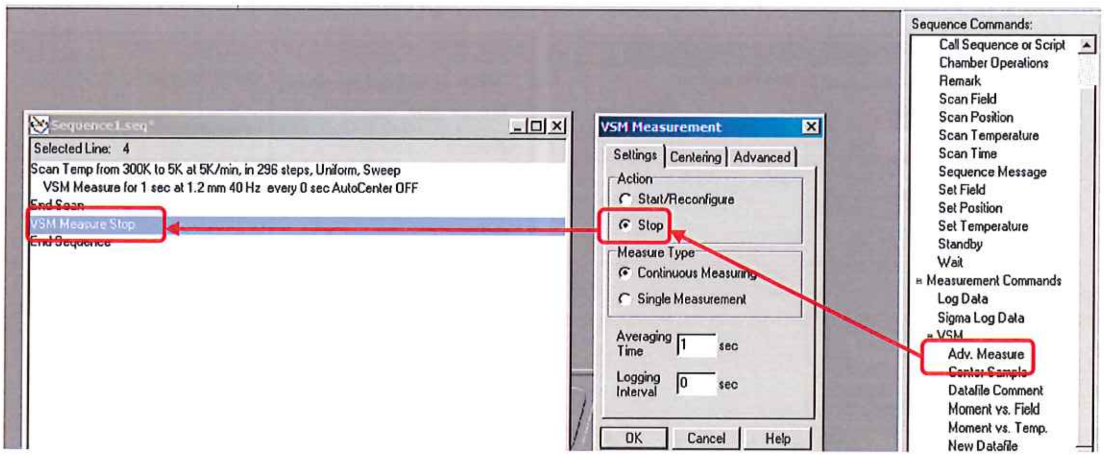


Fig 21b Simple M vs T scan temperature sequence terminated by VSM Measure Stop command

Following in fig 22 is an example of a full M vs H measurement. Note in this case, the VSM motor automatically shuts down after the full M vs H command is completed.

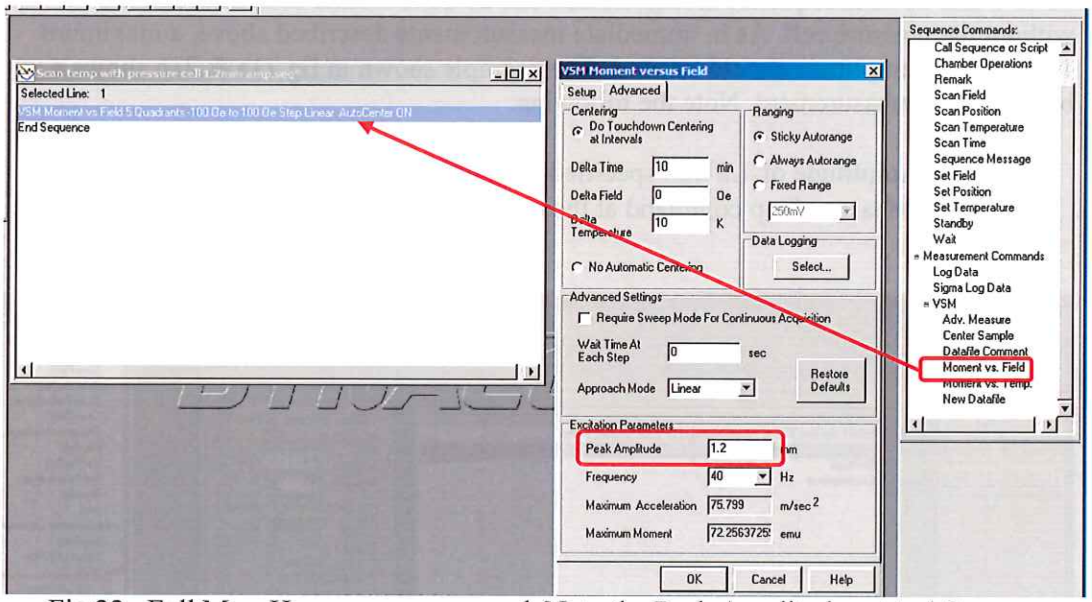


Fig 22 Full M vs H sequence command. Note the Peak Amplitude set to 1.2 mm.

MPMS3

⚠ Regardless if the VSM measurement option is available on your MPMS3 system, never use the pressure cell with the MPMS3 VSM option. First, the temperature will be very unstable due to the rapid moving of the sample, especially at temperatures less than 10K. Second, continuous vibrations of the sample can wear out the inside surface of the MPMS3 sample space, possibly leading to reduced SQUID sensitivity for future measurements.

The MPMS3 adaptor consists of 4 parts (refer to figure 15d):

- Upper section
- Lower section
- Spacer
- Aluminum m2 screw for holding the spacer to the lower section.

Once the pressure cell is assembled and mounted on the sample rod, the sample rod can be installed to the MPMS3 same as if using the QD supplied sample holders. Use the following guidelines to measure the lead manometer:

⚠ It is usually easier to precisely locate the sample with the manometer in the superconducting state. To specify the sample location in the sample install/remove wizard, choose Enter Offset manually and specify 67 mm.

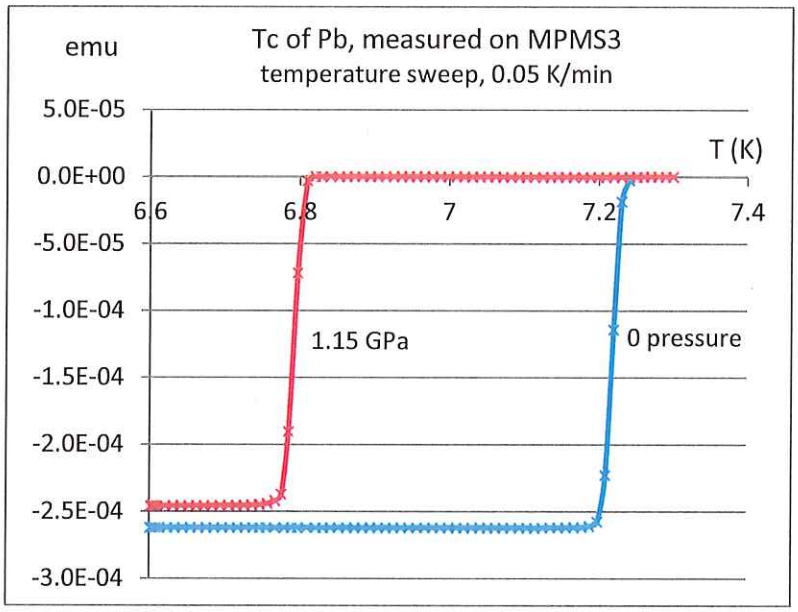
With the MPMS3 adaptors attached to the pressure cell, 67 mm will be close to the true sample location.

1) Set the MPMS3 temperature to 6.5K.
2) Set 1 gauss DC field.
3) Locate sample. For the centering method, use DC and 5 seconds. Verify a clean, symmetrical SQUID voltage response is observed.
4) Set the MPMS3 measurement parameters as follows:

Measure Mode	Continuous Measuring
Ranging	Sticky Autorange
Scan Length	30 mm
Scans per Meas.	1
Scan Time	5 s

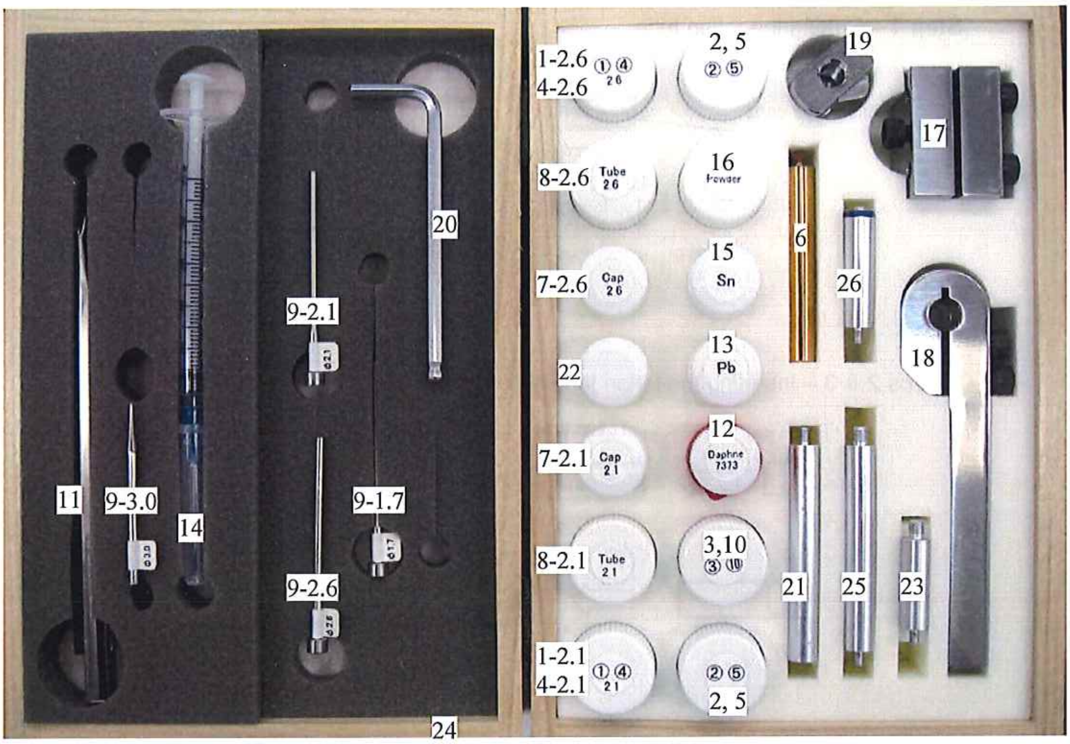
⚠ If the MPMS3 system includes VSM measuring mode, be sure VSM measurement is NOT accidently selected. Perform only DC or AC measurements with the pressure cell installed.

5) Begin measuring.
6) Set 7.5 K, 0.05 K/min sweep rate. Following is an example of lead data using the above guidelines.



Appendix A – Parts of the CC-Spr-Φ8.5D-MC4 kit

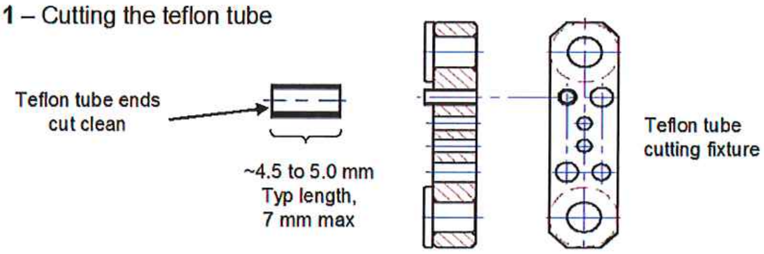
CC-Spr-08.5D-MC4 kit and list of parts



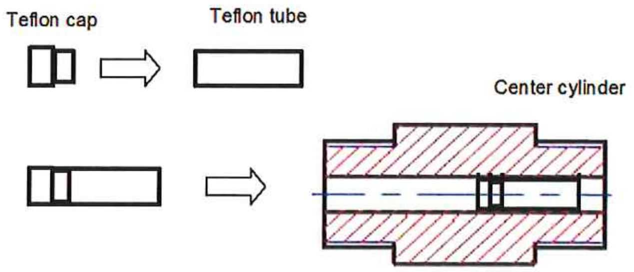
Designator/ Location	Part Name	Quan.	Designator/ Location	Part Name	Quan.
1-2.1	Center cylinder, 2.1mm ID	1	12	Oil, Daphne 7373 5 ml	1
1-2.6	Center cylinder, 2.6mm ID	1	13	Manometer Pb, Φ0.5mm x 100 length	1
2	Cylinder pressurization nut	2	14	Syringe, 1 ml	1
3	Piston backup	2	15	Manometer, Sn, Φ0.5mm x 100 length	1
4-2.1	Piston, 2.1 mm diameter	10	16	Teflon power, 5 ml	1
4-2.6	Piston, 2.6 mm diameter	10	17	Cell clamp	1
5	Side cylinder	2	18	Pressurization spanner	2
6	Pressure cell extensions	2	19	Oil pan	1
7-2.1	Teflon cap, 2.1mm	10	19	Cylinder support, 7mm	1
7-2.6	Teflon cap, 2.6mm	10	19	Cylinder support, 9mm	1
8-2.1	Teflon tube, 2.1mm 30mm length	15	20	Hex wrench, 4mm	1
8-2.6	Teflon tube, 2.6mm 30mm length	15	21	Adaptor, RSO sample rod	2
9-1.7	Sample push rod, 1.7mm	1	22	Adaptor, DC sample rod	2
9-2.1	Sample push rod, 2.1mm	1	23	Adaptor, VSM sample rod	2
9-2.6	Sample push rod, 2.6mm	1	24	Case	1
9-3.0	Pin, 3.0mm	1	25	MPMS3 adaptor – upper section	2
10	Teflon tube cutting fixture	1	26	MPMS3 adaptor - lower section, spacer, & m2 aluminum screw	2
11	Knife	1			

Appendix B – Teflon sample tube preparation and sample insertion

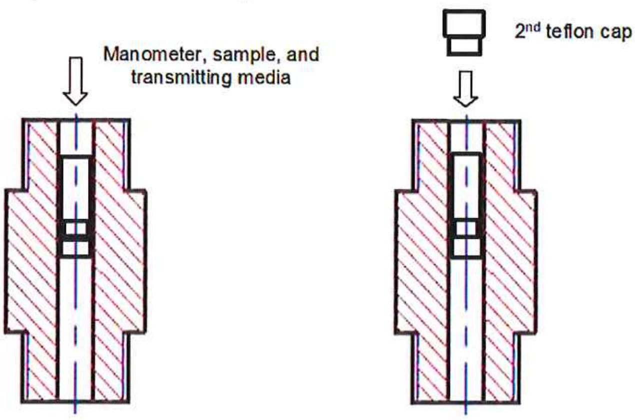
Step 1 – Cutting the teflon tube



Steps 2 & 3 – Inserting the teflon tube and cap into the center cylinder

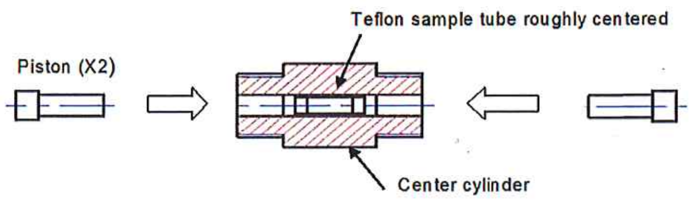


Steps 4 & 5 – Inserting sample and 2nd teflon cap

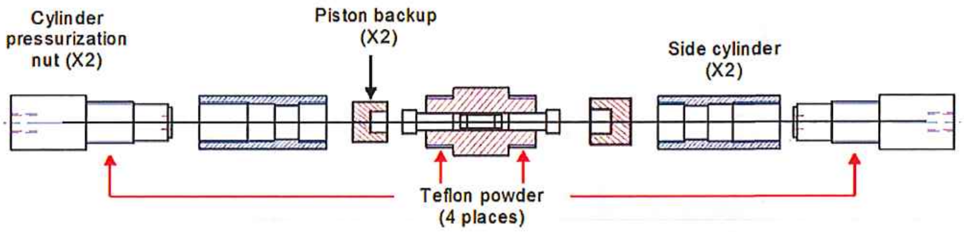


Appendix C – Pressure cell assembly

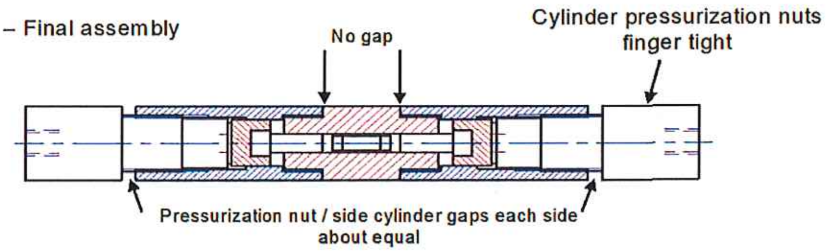
Steps 1 & 3 – Teflon tube centered, setting the pistons



Steps 4 - 8 – Applying teflon powder and assembling the cell

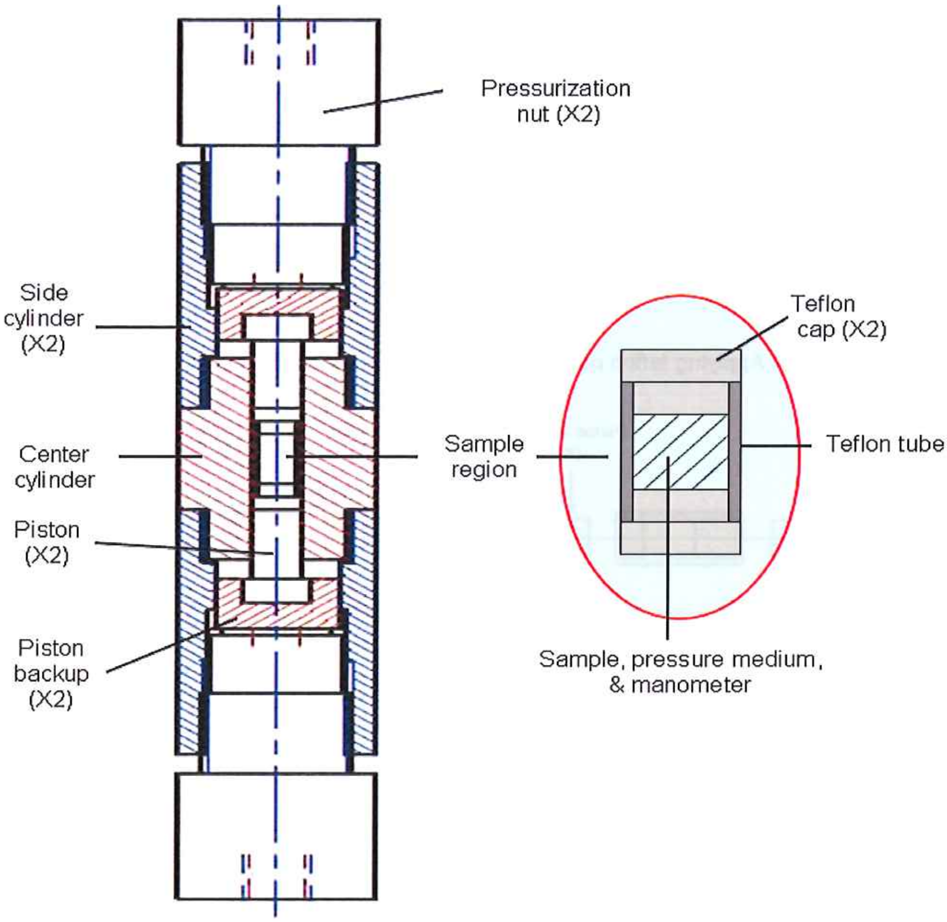


Step 9 – Final assembly



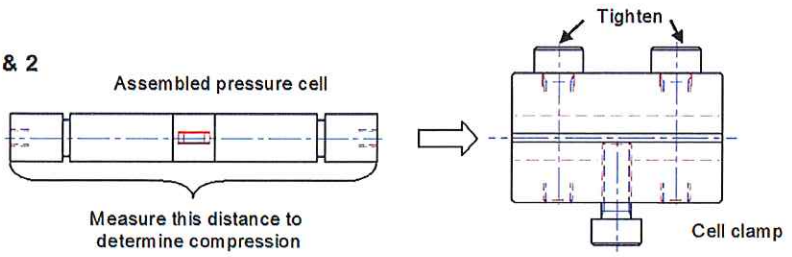
Assembled pressure cell before pressurization

Assembled pressure cell

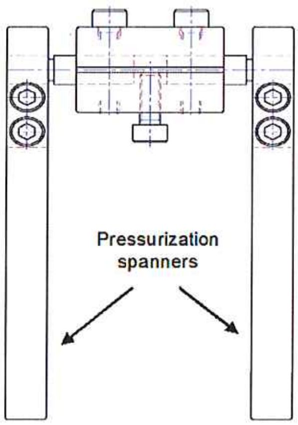


Appendix D – Sample pressurization

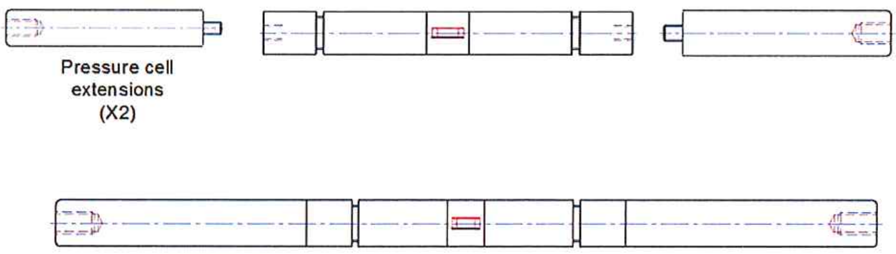
Steps 1 & 2



Step 3 – Setting the pressurization spanners



Step 7 – Applying the pressure cell extensions



Assembled pressure cell with cell extensions