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Technical manual for pressure cell operation for µSR experiments in GPD

1. <u>Preparation</u>

- -Keep all the tools clean
- -Check that all the pieces fit together
- -Check for any cracks in the solid or rubber parts of the system. Discard any parts that show wear.
- -Do not mix used liquid with pure liquid
- -Ensure that the right materials are used for the appropriate cell: MP35N double cells needs WC pistons and MP35N mushrooms CuBe/MP35 cells can be used with ceramic pistons and CuBe mushrooms
- -Ensure that the mushrooms are the correct length: for MP35 short mushrooms are enough (6mm) for any cell that has CuBe, longer mushrooms need to be used (7mm)
- -Ensure that there are enough Teflon rings, short mushroom needs 1, long one needs 2.
- -Put all the parts on a white sheet of paper and double check that all the parts are present as shown for example in the Figure below:



Figure 1: Pressure cell parts before the assembly. Left side corresponds to top.

2. <u>Assembly of the cell</u>

- assemble the mushrooms: Mushroom + Teflon ring + O-ring

First assemble bottom side

- Place one of the assembled mushrooms from the bottom side of the cell.
- Push the mushroom inside by about a few mm.

- Put the sealing ring + disc on top of the mushroom. (sharp edge of the ring towards the center)
- Add one drop of lubricant (molybdenium disulfite+daphne oil mixture)
- Put the piston (the shorter one) in
- To prevent cold-welding between the piston and the pressing pad, put a piece of paper with lubricant between them
- Combine the bottom bolt with the pressing pad.
- Apply some molycote grease on the threads of the bottom bolt
- Close the bottom bolt.

Now flip the cell and assemble the top part.

- Place pressure gauge (Indium disc)
- Place sample (Ideally, a few cylinders of combined 10-12 mm height and the required diameter)
- Fill the remaining of the volume with Daphne 7373 (or Daphne 7575) oil.
- Remove some Daphne oil (about 6 mm, using the pre-gauged syringe)
- Place the second mushroom.
- Put the sealing ring + disc on top of the mushroom. (sharp edge of the ring towards the center)
- Add one drop of lubricant (molybdenium disulfite +daphne oil mixture)
- Put the piston (the longer one) in
- To prevent cold-welding add a piece of paper with lubricant
- Combine the top bolt with the pressing pad.
- Apply some molycote on the threads of the top bolt
- Close the top bolt

The cell is now assembled but not sealed yet.

- To seal the cell, fix the cell body to the table and tighten the bottom bolt.
- Once the bottom bolt is tight, then switch to the top bolt and tighten it by hand.
- The sealing procedure, depending on how strong one closes it already gives about 5 kbar at room temperature, so a few kbar at low temperatures.

The cell is now sealed and ready for high pressure application.

3. Application of pressure

- Place the cell into a cylinder (#1) as shown in the figure on the right:
- The cylinder already centers the cell.
- Put a pressure application piston (#2) into the hole inside the top bolt

Now everything is assembled, the press will soon be turned on

- Check the conversion factor to obtain the right value of pressure in the cell from the manometer.
- Close all the potentially dangerous solid angle by plexi glass.
- Turn on the press by pressing green "ON" button
- Set the 15 Bar oil pressure value and 10Bar/min ramping speed.
- Wait until the end part of the Load Cell (Force measuring device) attaches to the compression rod (i.e. Piece #4 attaches to Piece#2)
- Fix the distance measurement device (#3).
- Start the recording
- Make sure that the needle valve is closed.
- Set the reliable oil pressure according to the conversation table. Start to apply pressure.
- Monitor both the pressure on the screen and on the manometer.
- Once the desired pressure is reached, wait for 10 more minutes before entering with hands to the pressure area.
- Now use a wrench to transfer the force from the press to the top bolt. Be careful and wear gloves.
- Once the pressure cell is tightened, set the oil pressure to "zero" and allow the pressure to be slowly removed from the press.
- After the pressure has dropped to zero, the gap between Piece #4 and Piece #2 appears. Wait until it reaches at least 1-2 cm.

Now the cell is pressurized. Leave it for 10 minutes for moderate pressures and 30 minutes for pressures larger than 75% of the maximum pressure.



4. Further manipulation

Depending on what needs to be done next (load to the spectrometer or to the ACS measurement), different steps will be taken. But in all occasions, try to minimize the exposure of the cell to outside. Namely – use specialized ammo-boxes to transport the cell and perform all the handling behind the plexiglass shields.