User manual for the MMR Seebeck Instrument

!Important!

Check that the high-pressure gas is off (both main valve and regulator valve) before doing anything else. Keep the vacuum pump going until the gauge is at ambient pressure.

Never turn the high-pressure gas on without pumping the sample chamber. Failing to do this will result in the Joule-Thomson cooling stage breaking.

Mounting a sample.

Use silver paint to attach a bar to the sample holder. The reference wire (constantan) and sample should have a V shaped geometry. The closer to an ideal V shape the better. It is important that the ends are at the same "height" at the hot end (ideal V-shape).

Use the Ohm meter to check the resistance across the reference and your sample. This helps check that the silver paint is not connecting the two gold pads. Resistances up to about 20kOhm the instrument should work. For higher resistances, there is a high impedance board that can be mounted.

Let the Ag-paint dry (patience) 5-20 min.

Place the sample holder in the sample chamber make sure that the contacts are

Screw on the lid making sure there is a good seal (standard vacuum grease and a good O-ring).

Connect the vacuum pump and switch it on.

A quick test

After mounting the sample, the SB100 and K20 boxes can be turned on. (Always have them switched off when opening the sample chamber. Fuses break easily).

Open the "Seebeck" software (icon on desktop) and open the menu for manual control (holding the cursor on top of the icons reveals what they are for). Type "SM3" to initialize the measurement. Then "GV1" and "GV2" to check the sample and reference voltages. Values above 1000μV are too high for reliable measurement. This can be a sign that your sample is too resistive or that the contacts are not good.

Measurement set-up

Open the measurement set-up window. The standard settings usually work well. The things to change are the temperature range and step-size. To make sure the measurement is reliable it is advisable to collect 3 (at least 2) data points at the same temperature.

To make sure everything is working (before cooling down), it is wise to measure the Seebeck coefficient at room temperature or above (up to 380C for example). Input the temperature range and step-size and then press the play button on the menu bar (top of screen). You want the three readings at each temperature to agree with each other.

Cooling down

Cooling is based on the rapid expansion of UHP N_2 gas (Joule-Thomson cooling). Before being led through the cooler the gas is dried and purified using a filter inside the white box. In fact, there are two filters in parallel. One is used while the other is purged/cleaned simultaneously.

Between each cooling run, switch the valve on the box with the filters.

Make sure the **lid of the sample chamber is screwed down** and the **vacuum pump switched on**.

Make sure the **regulator valve is closed**.

Open the main cylinder valve.

Slowly open the regulator valve, and check for flow through the sample chamber using the flow meter. Increase the pressure to 1600 PSi (**never go above 1800!**). The flow through the chamber should be about 3.5 SCFH.

Now the instrument should be cooling. This can be monitored using the Seebeck measurement software. Using the manual control, a set-point can be set. (e.g. SP 100). The operating range of the instrument is between 85-400 K.

Problems with cooling

1. Poor vacuum in the sample chamber.
   Fix: close main valve on cylinder and regulator valve. Wait for pressure on gauge to reach ambient. Switch off vacuum pump. Open sample chamber and check O-rings and apply some vacuum grease.

2. Pressure in the N_2 cylinder is below 1600 Psi
   Fix: Throw cylinder out and buy a new one. Typically you can expect to get about 8 runs from a standard size cylinder (when collecting 3 datapoints at each temperature).

3. The filters are dirty. The MMR instruction manual has a section on how to clean the filters. This has not happened in the past year and if one switches regularly between the two filters there should be no problem.

Stopping the measurement

1. Set the temperature to 280 K (manual control, type SP 280)
2. Close the cylinder and regulator valve
3. Keep pumping till the regulator gauge is at ambient.
4. Switch off the pump
5. Switch off the SB100 and K20
6. Now you can open the sample chamber and take your sample out.
7. Save your data!!! Put it in your folder. This computer is not hooked up to the internet so you can use the floppy.

Good luck!

JWB and KH