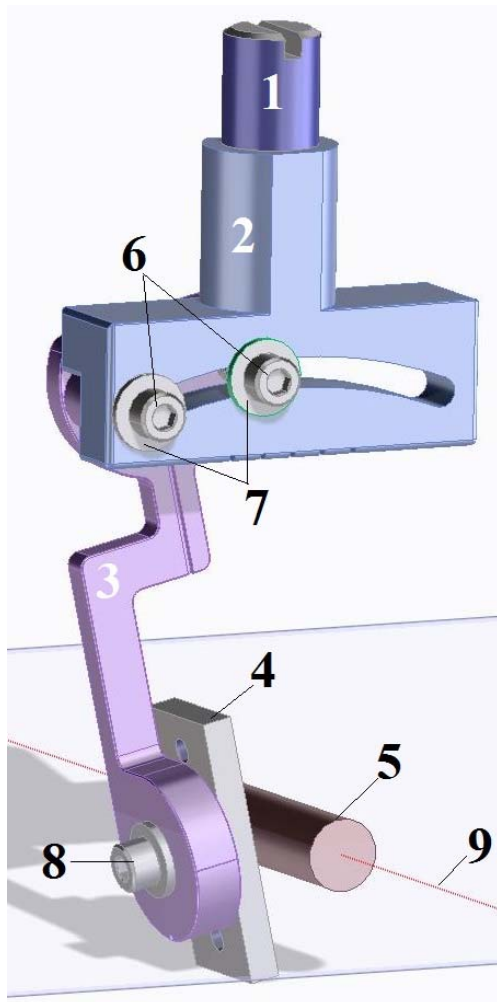


Adjustable Copper single crystal holder for Dil Fridge neutron experiments at NCNR

This sample holder is designed for and can be used with the ICE dil fridge and Oxford dil fridge inserts available for users at NCNR. The Holder allows single crystal sample alignment within two degrees of freedom (see [Sample aligning](#) paragraph) prior to mounting it into the dil fridge. The Holder is designed to provide good thermal conductivity between the sample and the mixing chamber of the dil fridge.

a)



b)

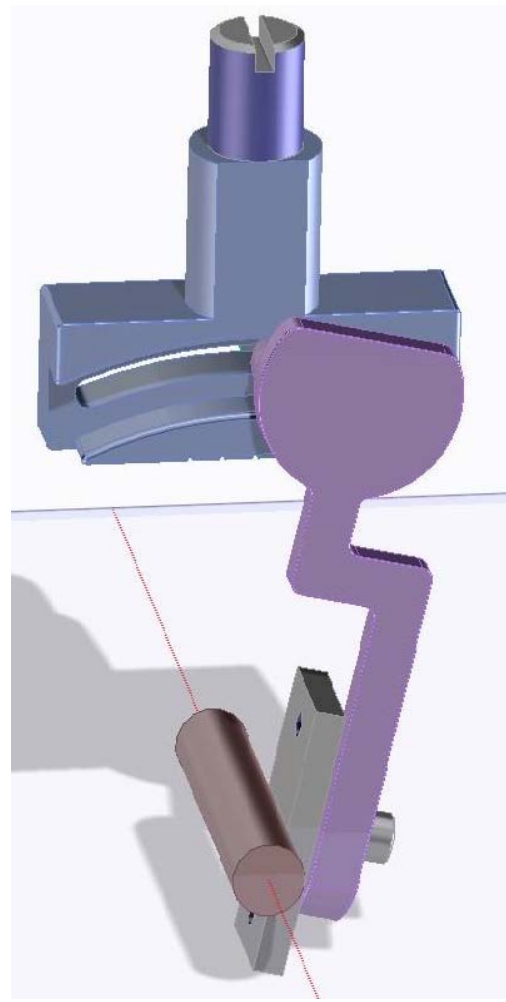


Figure 1 – Assembly of the Single crystal holder at the -12.5° position: (1) Brass M6 threaded stud for ICE dil fridge mount or M6 male to 5/16-18 male adapter for Oxford dil fridge mount; (2) Sample holder base; (3) Sample holder stud; (4) Sample holder substrate; (5) Sample; (6) M2.5 brass screws, length 8mm; (7) Brass washer for number 3 screw size; (8) M2.5 brass screw, length 5mm; (9) Rotation axis, R38mm, located 5.5mm apart from the Sample holder stud surface.

Sample holder material: Oxygen-free Copper body, brass screw and washers.

Maximum sample dimensions: cylinder with the length up to 30mm and the diameter up to 6mm. Other sample shapes possible – *contact Sample Environment for assistance*.

Sample attachment: Copper foil (not shown in Figs. 1-2) is recommended to attach a sample to the Sample substrate using two M2.5 brass screws. In addition, Hydrogen-free glue, Cytop M, can be used to improve thermal conductivity between a sample and the Sample holder substrate. Per well-timed user request (at least 3-4 weeks prior to the experiment), a custom-made Sample holder substrate can be designed and made to fit a particular sample shape and size – *contact Sample Environment for assistance*.

Sample aligning:

- (1) +/- 12.5° from the vertical position relative to the sample axis (as shown in Fig.1 and Fig.2);
- (2) 360° relative to the axis normal to the Sample substrate (Fig.1, #4). In other words, Sample substrate rotates 360° together with the sample attached.

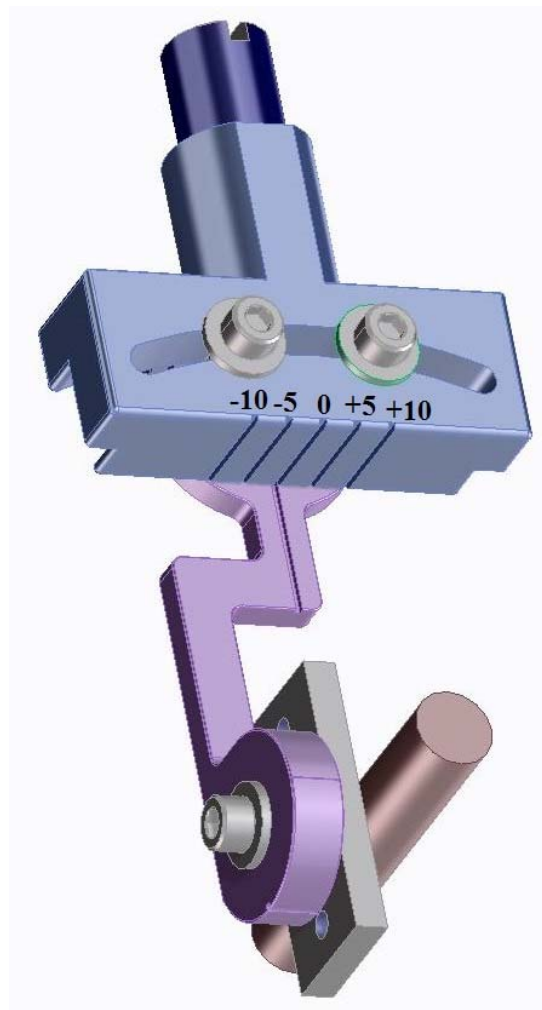


Figure 2 – Assembly of the Single crystal holder at the vertical position (0°, in this case the notch on the Sample holder stud is aligned with the middle notch on the Sample holder base. Each next notch on the Sample holder base, counting from the middle one, when aligned with the notch on the Sample holder stud, adds 5° to sample rotation.

Sample thermalization. Most NCNR users do care about knowing the real temperature their samples can be cooled to. When a dil fridge is used on high flux instruments, like the Multiple Axis Crystal Spectrometer (MACS) at NCNR, one should expect noticeable sample overheat. To evaluate sample overheat, this Sample holder has been tested to estimate the temperature at the position where a sample attaches to the Sample holder substrate. Offline (no neutrons) measurements have been performed with a RuO₂ calibrated sensor attached to the Sample holder substrate replacing a real sample. A wire heater was wrapped around the Sample holder stud close to the temperature sensor to mimic the heat generated by a neutron flux. The heater and sensor, in turn, were used to stabilize the temperature while the mixing chamber sensor readings were recorded. It should be noted, the mixing chamber sensor is the only one that can be used for sample temperature estimation during a neutron experiment under 1.5K. For better thermal conductivity, it is very important to use Apiezon N vacuum grease in between all matching surfaces of the Holder except those occurred in the neutron beam.

Figure 3 shows how much warmer the sample position is, compared to the mixing chamber of the dil fridge at its base temperature. No heat is applied directly to the mixing chamber heater. The neutron beam on the MACS instrument often overheats the mixing chamber of the ICE dil fridge insert up to 100 mK. The cooling power of this fridge is about 20 μW at 100 mK. This means the temperature at the sample position is 100 + 40 = 140 mK. The sample temperature, in turn, could be even higher and can be estimated using, for instance, temperature dependent sample properties measured by neutrons.

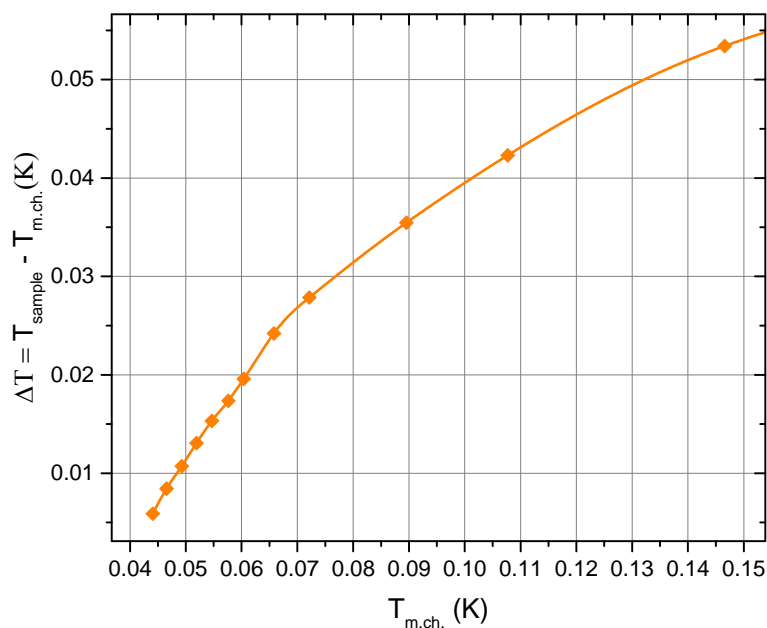


Figure 3 – Measured (offline, no neutrons) overheat, ΔT , as a function of mixing chamber temperature measured during neutron experiment. This is a steady-state overheat at the Sample position for the Sample holder attached to the ICE dil fridge insert. Apiezon N vacuum grease was used (and should be always used) in between mixing chamber and the Sample holder base and in between the Sample holder base and the Sample holder stud.