



Application Note 1085-157

3D Heat Capacity Puck for PPMS DynaCool

Background

The PPMS DynaCool provides a variable temperature (1.8 – 400 K) and magnetic field (up to 14 T, depending on the system) environment, just as in the original PPMS, but now as a ‘dry’ cryogen-free system. DynaCool also leverages the same puck interface and sample chamber design as the original PPMS, so much of the PPMS option hardware is trivially compatible; one interesting exception, however, is the case of the Heat Capacity Option when run in conjunction with the Dilution Refrigerator Option.

The underlying cooling circuit in the DynaCool, driven by a two-stage pulse tube cooler, is fundamentally different from the original ‘wet’ PPMS. The convenience of a cryogen-free system comes at the cost of some added sources of mechanical vibration: these include the pulse tube cryocooler, rotary valve motor, and high pressure gas lines which circulate the helium to and from the external compressor. When measuring heat capacity, these vibrations can induce a physical oscillation of the suspended calorimeter platform, the mechanical action of which generates a small amount of heat. For standard (400 – 1.8 K) heat capacity measurements, and even down to the base temperature of the Helium-3 Refrigerator Option (400 mK), this additional heat load does not present a problem.

When measuring heat capacity down to the base temperature of 50 mK in the DR, however, this heat can be significant. The vibration-induced heat manifests as noise in the measurement of the platform temperature and occurs to an extent that is prohibitive to extracting accurate calorimetry data. The physical oscillation of the platform in the original ‘2D’ puck (4085-264) design for the Dilution Refrigerator Option insert owes to the slight amount of slack in the supporting structures keeping the calorimeter platform in place (the primary degree of freedom is indicated by the arrow in Figure 1). Initially, these supports were thin tubes of Kapton (all DR heat capacity pucks through serial number 126), but were eventually replaced with a more rigid fiberglass thread meant to dampen the oscillations (DR heat capacity pucks numbered 127 and above) as well as to resolve the issue of Helium condensing within the tubes (which modified the effective wire conductance).

Ultimately, it was not possible to manufacture the 2D-style pucks to within the tolerances necessary to consistently eliminate the low temperature vibration-driven heating effect, and thus a new design, the 3D heat capacity puck (4085-280, serial number 3010 and above), was engineered to address this concern.

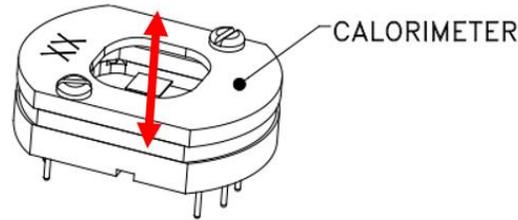


Fig. 1: Schematic drawing of the original 2D Puck; axis of travel of the calorimeter platform when driven by vibrations from the DynaCool pulse tube shown by the red arrow.

Sample Mounting

The construction of the 3D puck is such that it is incompatible with the sample mounting station (4085-240) and adapter (4092-625) used for installing samples on any of the 2D-style pucks. The fiberglass threads which secure the platform, along not only the x and y directions but also the z axis, actually interfere with the mounting station tools, so they are not to be used with the 3D puck.

The sample mounting station was designed to mitigate the strain on the wires and joints stabilizing the platform against the forces experienced when adding or removing a sample- in the case of the 3D puck, these accessories end up being unnecessary. Here, the epoxy joints and fiberglass threads are sufficiently robust that grease and samples may be added and removed, with the same care and attention as before, but without additional hardware. Now, the latticework supports the platform against modest forces applied in virtually any direction, eliminating the concern of damaging the delicate wires providing the thermal and electrical connections. Access to the bare side of the platform is by the large aperture on the pin-side of the puck assembly, shown in Figure 2.

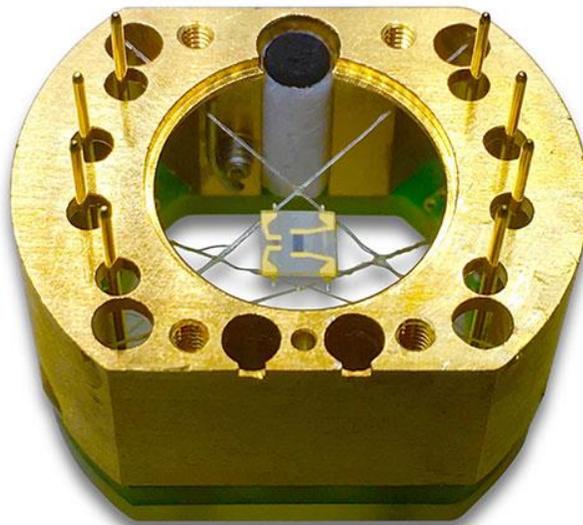


Fig. 2: View of the underside of a 3D puck. This is the proper orientation for adding grease or samples to the platform, as well as for cleaning when the user's experiment is complete.

Field Calibrations

Additionally, the gold-plated silver calibration fixture (4091-624) for the 2D puck is also not compatible with the 3D puck. The user kit for the 3D puck includes its own calibration plug (4085-283) which has a small length of a woven copper braid. Installing this for field calibrations is not trivial, as care needs to be taken to ensure adequate thermal contact. First, using a thin wooden toothpick, pull the loop of copper braid away from the post so as to create a small loop, as depicted in Fig. 3.



Fig. 3: 3D puck calibration fixture, showing the copper braid extended to optimize the contact area made with the calorimeter platform.

Next, add a small amount of N-Grease to the rim of the large aperture on the pin-side of the 3D puck- spread it around the small lip to create a uniform layer. This will provide the thermal link between the calibration post and the sample stage of the dilution refrigerator. A drop should also be added at the center of the platform: a sufficient amount should be used such that the drop extends roughly 1 mm vertically upward from the surface of the platform. This ensures when the calibration plug is installed the copper braid has enough grease to establish a good thermal link with the platform.

Puck Cleaning

As with any heat capacity puck particular care and attention must be taken when cleaning the platform at the end of an experiment. The first step should always be to remove as much sample as possible using a good pair of tweezers. The remaining sample fragments and grease should then be removed using a fine cotton swab by starting at the edge of the sample holder and moving inward, lifting off the swab and material before the opposite edge is reached. This is to avoid pushing grease and sample material over the edge of the platform, where it is much harder to remove.

Once the bulk of sample and grease has been removed, wet a clean swab with a small amount of toluene and again wipe from and edge towards the center. Do not attempt this step before removing as much material with the dry swab as possible- the toluene dissolves the N-Grease, and if excess remains it can flow over the edge to the platform back, along the wires, etc. When all of the grease is removed, the platform should have a matte-looking finish. Inspect the platform edges for any remaining beads of grease or sample material. If something has worked its way to the *back* of the platform, please contact apps@qdusa.com for further instructions.