

Choice of sample can - typical check list

- Is a sample can necessary?
- Many sample environments (e.g. cryostats, “displexes” (i.e. closed cycle refrigerators), furnaces) operate with a vacuum outside the sample can. This has several implications:
 - Is the sample can structurally strong enough to withstand differential pressure over the whole sample temperature range?
 - If the cell must be sealed (e.g. liquid sample, fine powders, outgassing samples etc.), what type of seal will support the temperature range (will it freeze, melt, disintegrate etc.)? Typical examples of seals are Viton (around room temperature), Indium (low temperature-400K), Lead (400K-570K), Copper gaskets and Gold wire for higher temperatures etc.
 - If the seal is in contact with the sample, is this an issue (chemical reaction etc.)?
- Is there any risk of pressure build up in the sample can (e.g. thermal expansion, anomalous expansion on freezing, outgassing of the sample)? Note too that cans overfilled with incompressible fluids can build large hydrostatic pressures when the seal is compressed.
- Is the sample can structurally resistant over the proposed temperature range, bearing in mind the melting point of the material, material embrittlement, pressure conditions?
- Is the sample can material chemically inert with respect to the sample (e.g. be careful with low or high pH samples)?
- Is the can material suitable for the experiment (low neutron absorption and scattering cross sections)?
- Can the sample can be filled easily? (e.g. thin annular cans used for liquid samples may be almost impossible to fill with powders, foils etc.).
- Will the sample can fit in the proposed sample environment?
- Is the heat conduction from the temperature control device to the sample sufficiently good? (Be careful with insulating seals and thermal contacts between sample cans and can lids etc.).
- Can the sample be handled in the facilities provided? (Hazardous samples, air-sensitive or moisture-sensitive samples, radioactive samples) - Usually, special conditions for handling the sample have to be addressed well in advance.

Choice of sample environment/ temperature control device

For most experiments, we use one of the following:

- Liquid He cryostats (1.4K-325K)
 - Very stable and uniform sample temperature
 - Labor-intensive: requires periodic filling with liquid cryogenes (nitrogen and helium)
 - Requires adjustment of helium flow rate for different temperature ranges
 - Requires a couple of hours' preparation time when starting afresh
 - Usually requires an experienced operator
- “Low temperature” ($\sim 10\text{K} - 325\text{K}$) closed-cycle refrigerators (“displexes”)
 - Low maintenance (no liquid cryogen filling) and easy to operate
 - Easy to control with appropriate temperature control parameters (PIDs).
 - Good response times
 - Heating/cooling occurs from one end of the sample. Usually there is more of a temperature gradient than with a cryostat (typically $\sim 1\text{-}2\text{K}$) along axis of sample but the gradient may be greater if there is poor heat conduction or poor thermal contact with the sample.
- “High temperature” (10/20K - 475/600K) displexes
 - Low maintenance (no liquid cryogen filling)
 - “Thermal switch” construction between “hot” and “cold” stages (conducting at low temperature, insulating at high temperature) - combination of large thermal inertia, long time constants ($\sim 1\text{hr!}$), possibility of delayed and highly non-linear heater power-sample temperature response. Notoriously difficult to control, high temperature displexes do not like abrupt changes of setpoint temperature but favor slow ramps. (This can be time consuming if many widely spaced sample temperatures are to be measured). PIDs usually have to be chosen carefully for steady state operation and adjusted when changing temperature.

Choice of sample environment/ temperature control device (continued)

More rarely - (usually requiring advance coordination with NCNR staff)

- Furnaces (>600K applications)
- Dilution refrigerators and ^3He cryostats (<1.4K applications)
- Cryomagnets
- High pressure devices
- Thermal baths etc.