A protocol for accurately calibrating thermocouples at cryogenic temperatures

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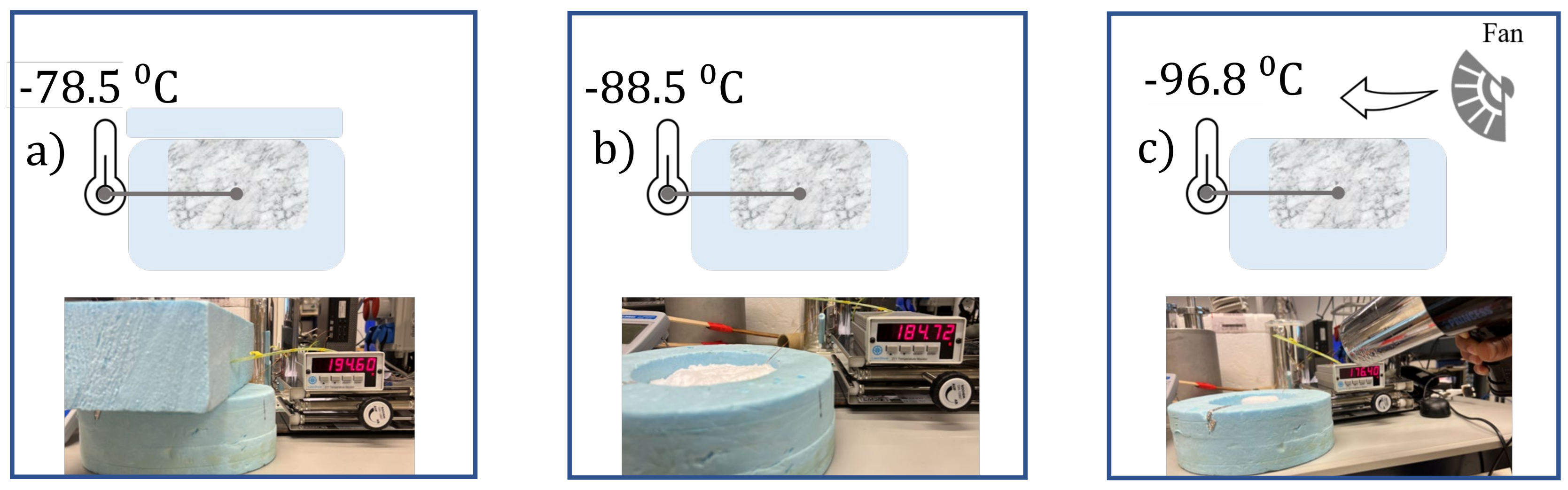
Fast response temperature measurements

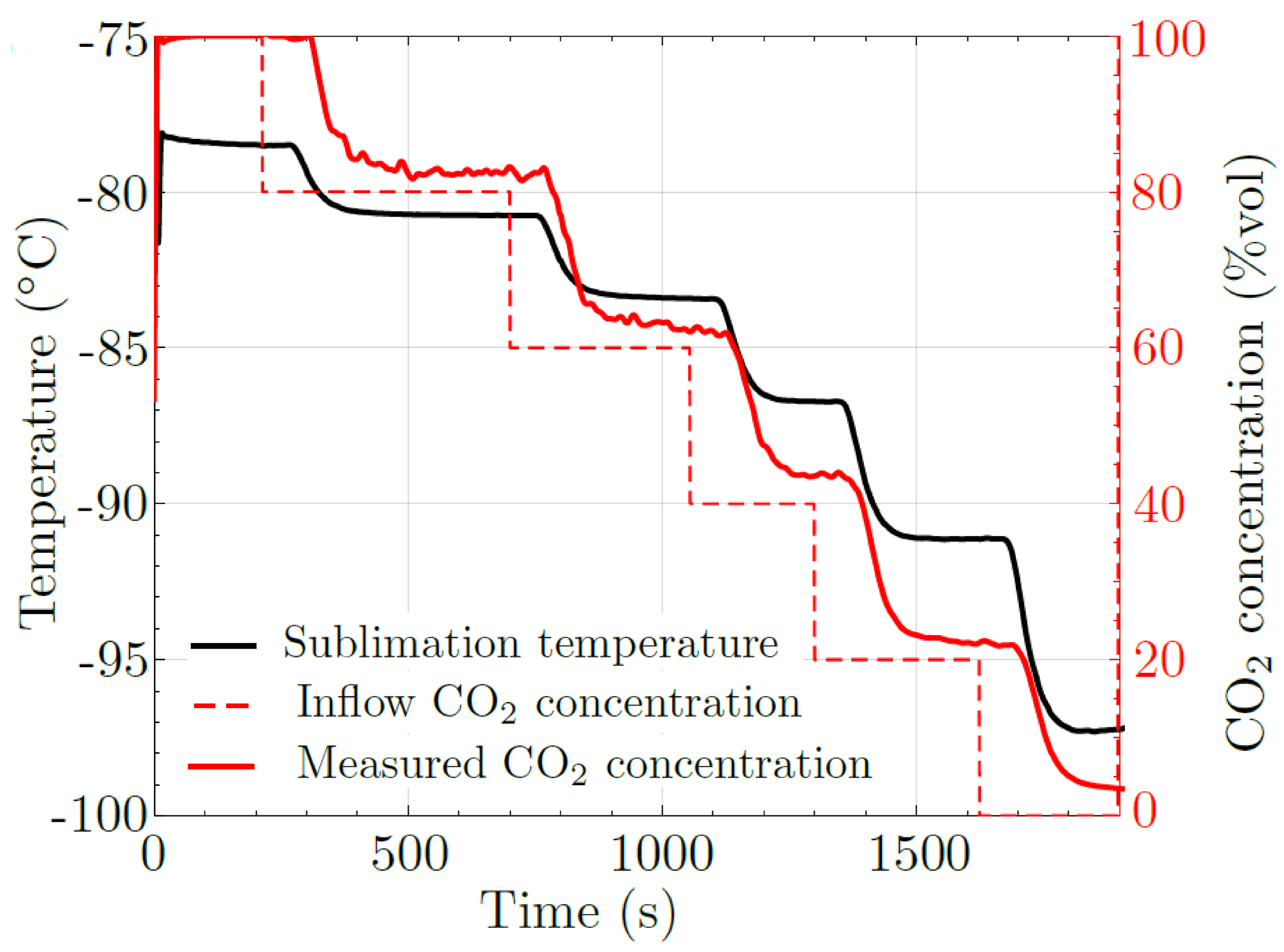
Thermocouples are affordable temperature sensors with low self-heat capacity and a quick response time, making them well-suited for investigating the cooling behavior of materials in cryogenic liquids and performing fast and precise transient measurements. To ensure precise temperature measurements, it is essential to calibrate newly acquired thermocouples or recalibrate existing ones. For this purpose, a common practice is the use of fixed-point temperatures such as the ice-water slush (0 °C), the normal sublimation temperature of dry ice (-78.5 °C), and the normal boiling temperature of liquid nitrogen (-196 °C). However, uncertainties are associated with the fixed-point temperatures of dry ice sublimation and liquid nitrogen boiling due to the inherent heat and mass transport phenomena occurring at the phase-changing interface and within the bulk medium. Therefore, it is important to address these uncertainties and define a protocol for calibrating thermocouples using these fixed-point temperatures.

Fixed point: Dry ice

The dry ice (solid CO_2) sublimation temperature **depends on the far field CO_2 concentration**



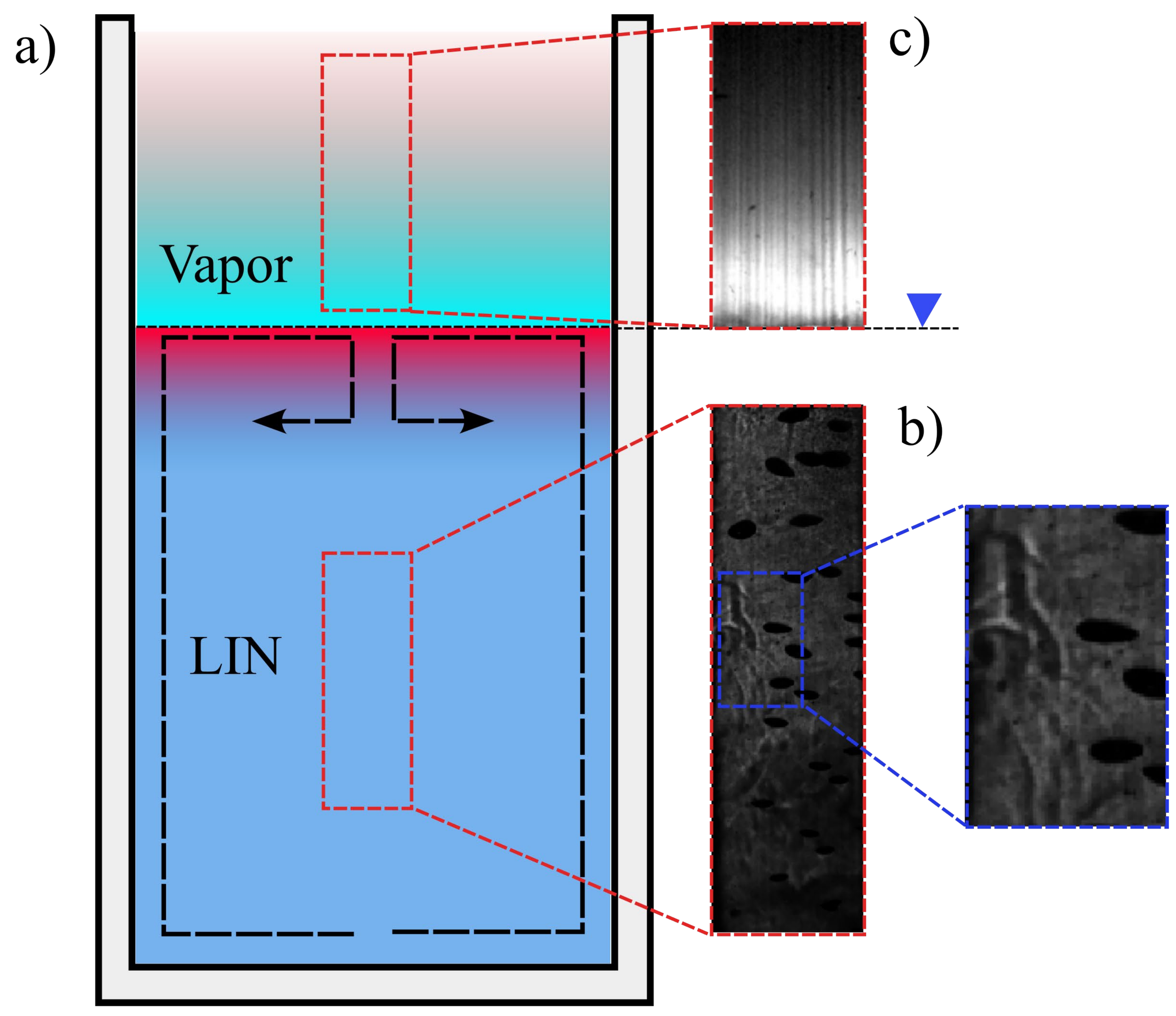
Measurement of the dry ice sublimation temperature under various conditions; a) a closed environment with tiny vents to overcome pressure built-up, b) an open lab environment, c) Flushed with a fan



Measured values of the sublimation temperature of dry ice for different far-field CO_2 concentrations in a controlled test rig [2].

Fixed point: Liquid nitrogen

Thermal stratification in a liquid nitrogen bath due to low thermal conductivity of the liquid.

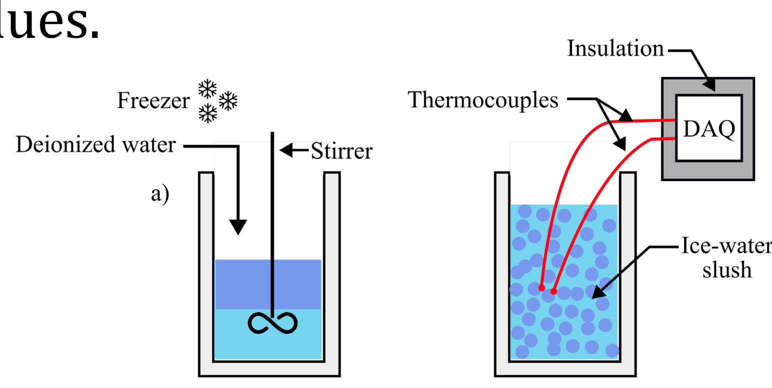


a) Schematic of the thermal stratification in pool of liquid nitrogen and in vapor above the pool; b) Schlieren image depicting density gradients (bright-dark contrast) inside pool of liquid nitrogen; c) Schlieren image depicting density gradients (bright-dark contrast) in vapor above the pool.

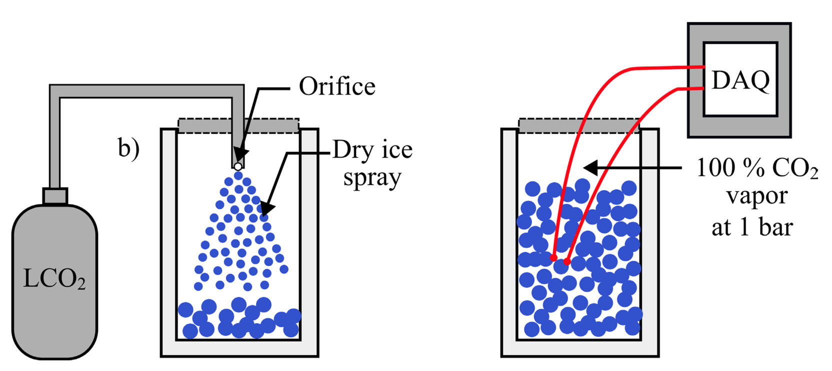
Oxygen enrichment of the liquid pool due to condensation when open to air causes the boiling temperature of the mixture to deviate from that of pure liquid nitrogen.

Thermocouple calibration protocol

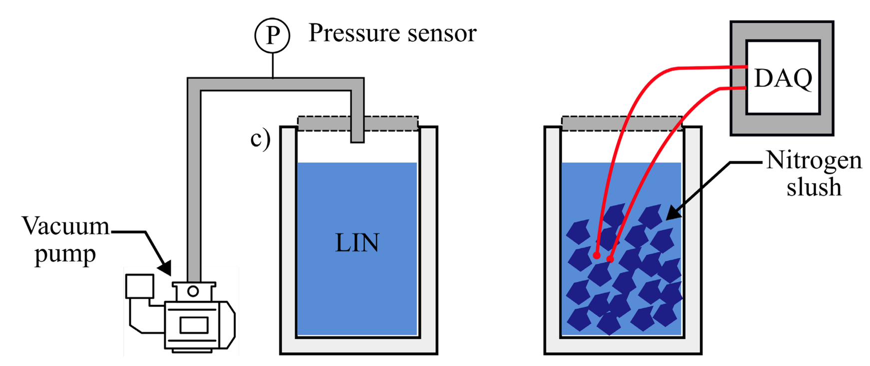
- Ice cubes are mixed in de-ionized water in a beaker to obtain a slush of ice and water.
- Dry ice can be procured or produced in a laboratory by expanding high-pressure liquid CO_2 through an orifice, and then transferred into a container with a lid. The CO_2 gas should not build pressure in the container and must be vented out.
- Nitrogen slush is created by reducing the vapor pressure of the liquid nitrogen below 0.125 bar. After solid is produced, the system is vented to atmospheric pressure.
- The saturation temperature **corresponding to the atmospheric (lab) pressure** for the above phase change processes will be the fixed-point values.



Ice-water slush



Saturated dry ice



Nitrogen slush

1. Scott R B 1940 Calibration of thermocouples at low temperatures J. Res. Natl. Bur. Stand. 25 459-474
2. Purandare A, Verbruggen W, and Vanapalli S 2023 Experimental and theoretical investigation of the dry ice sublimation temperature for varying far-field pressure and CO2 concentration Preprint 10.2139/ssrn.4462700
3. Kang M, Kim J, You H, and Chang D 2018 Experimental investigation of thermal stratification in cryogenic tanks Exp. Therm. Fluid Sci. 96 371-382

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