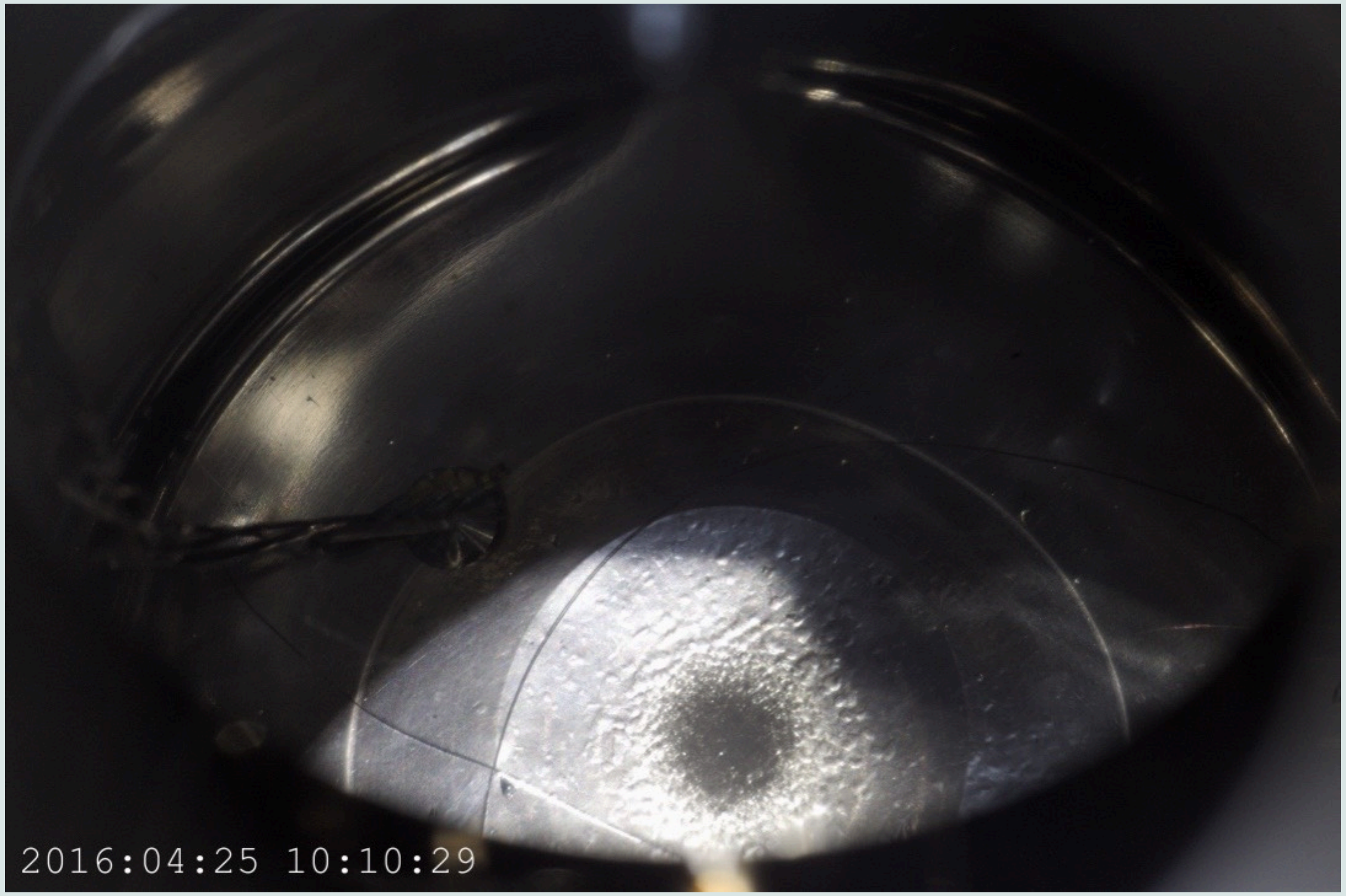
Growing solid deuterium crystal for Ultra Cold Neutron production

Ekaterina Korobkina



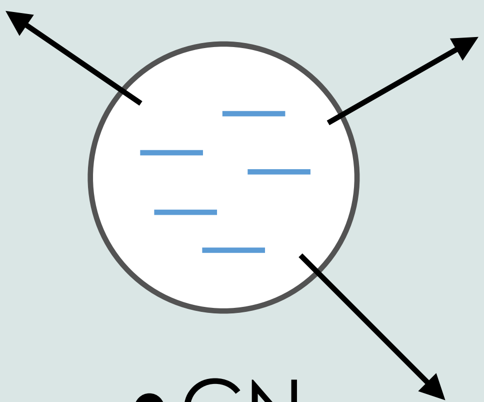
Outlines

- Solid Deuterium properties related to UCN production
- Growing small perfect sD2 crystals
- Growing large sD2 crystals in situ for UCN production
  - Overview of experimental setup
  - Study of SD2 growth
    - several steps condensation
    - temperature dependence of crystal shapes and quality
    - one step condensation
    - surface frost
    - freezing from a liquid
- Recommendations

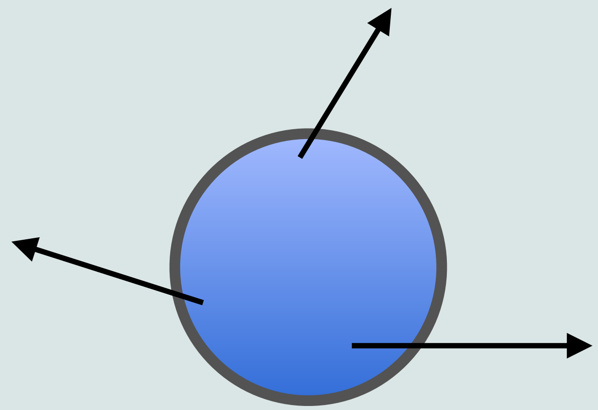
saturation density of Neutrons in the source $N = P\tau$

Liquid D2, $T_{source} \sim T_n$

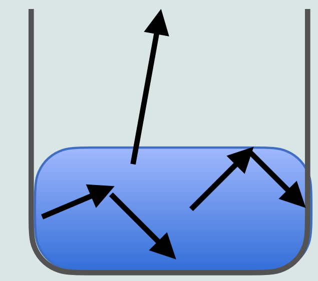
Solid D2, $T_{source} \gg T_n$



- CN



- VCN



- UCN

$L_{abs} = 500$ m $\gg L_{source}$

$L_{abs} = 25$ m $\gg L_{source}$

$L_{abs} \sim$ cm $\leq L_{source}$

UCN density in experiment $N = P \times \tau \times T$

Transmission $T = T_{Bulk} \times T_{top}$

elastic scattering is an important factor affecting both, UCN travel in the bulk and UCN extraction to the vacuum through the top of the crystal

Properties of solid deuterium crystal

- SD2 is a quantum molecular crystal with weak intermolecular interaction, hcp or/and fcc structure
- strongly temperature dependent thermal activated diffusion above 9K
- strongly temperature dependent anisotropic linear expansion coefficient above 10K
- triple-point-wetting

Properties of solid deuterium

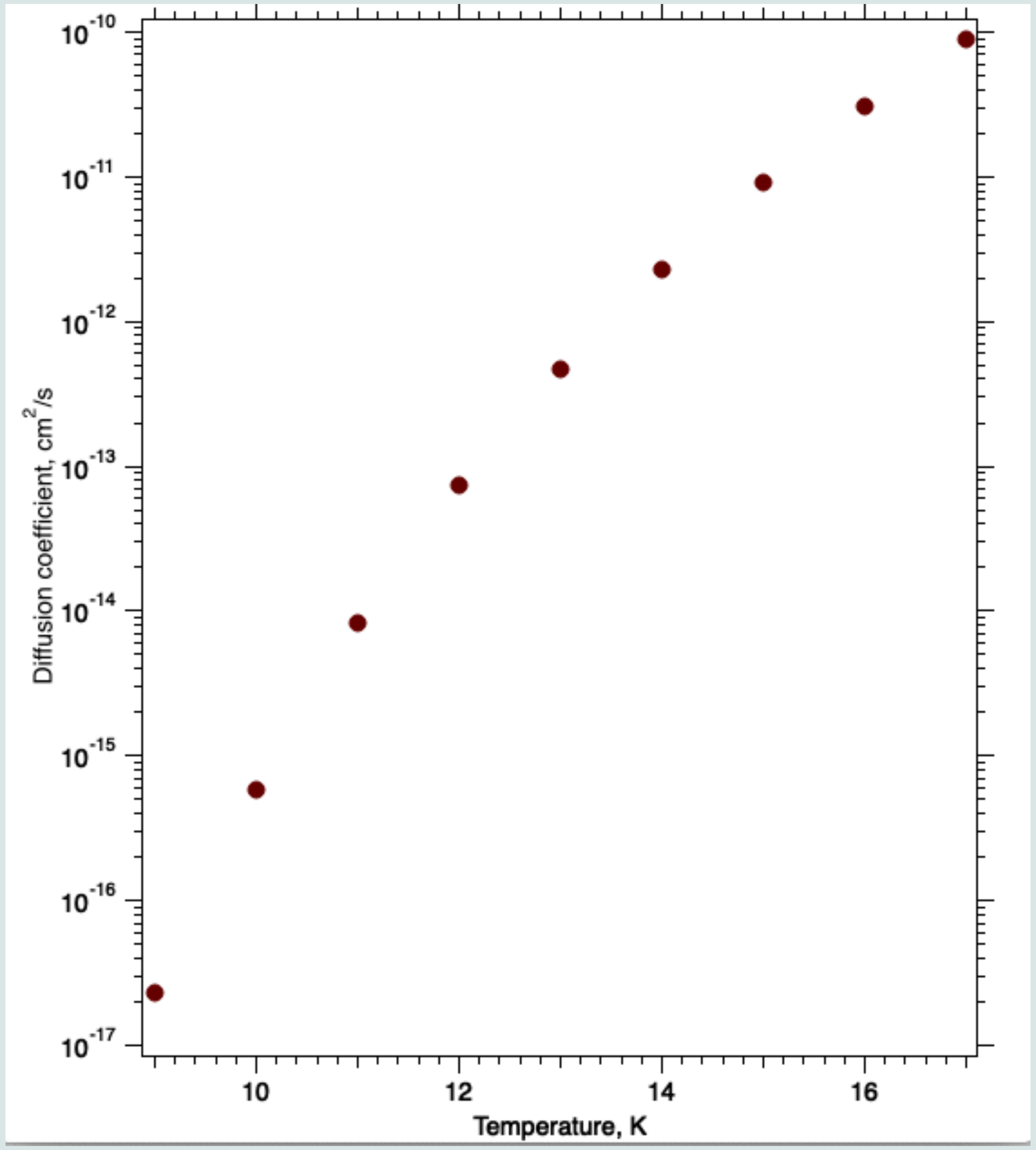
- strongly temperature dependent thermal activated diffusion

Time between jumps
$\tau = \tau_0 \exp(E/k_B/T)$

where diffusion activation energy
E/k_B=290K; $\tau_0 = 9.4 \times 10^{-14}$ s

Diffusion constant $D=D_0 \exp(-E/k_B/T)$

where $D_0 = R^2/6\tau_0$ for a random walk step R;
for sD_2 $D_0 = 2.3 \times 10^{-3}$ cm^2/s



Properties of solid deuterium

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- strongly temperature dependent molar volume and linear expansion
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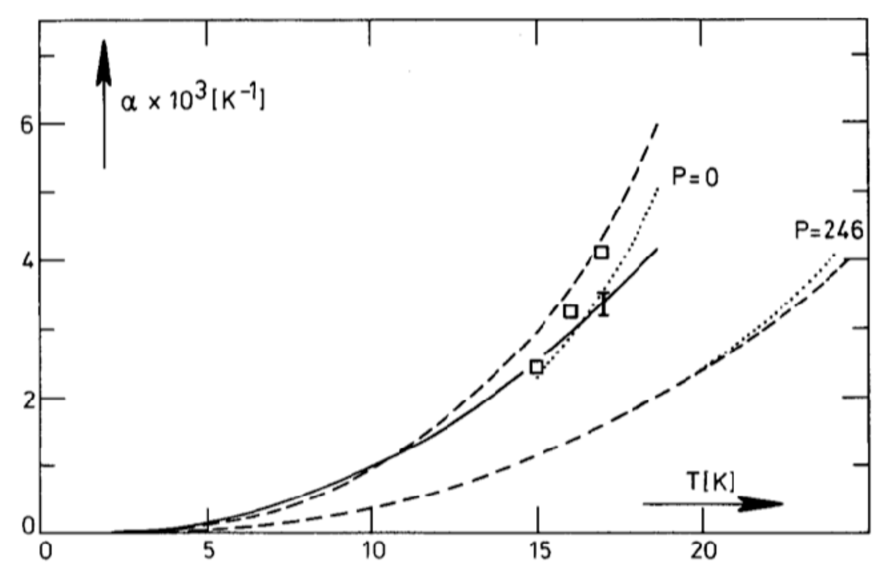

Fig. 3. The thermal expansion of o-D_2 as a function of temperature at zero pressure and 246 bar. Solid line: Nielson[35]; dashed lines from our EOS, given in the Appendix; dotted lines: Esel'son *et al.*[33]; squares: Krupskii *et al.*[34]

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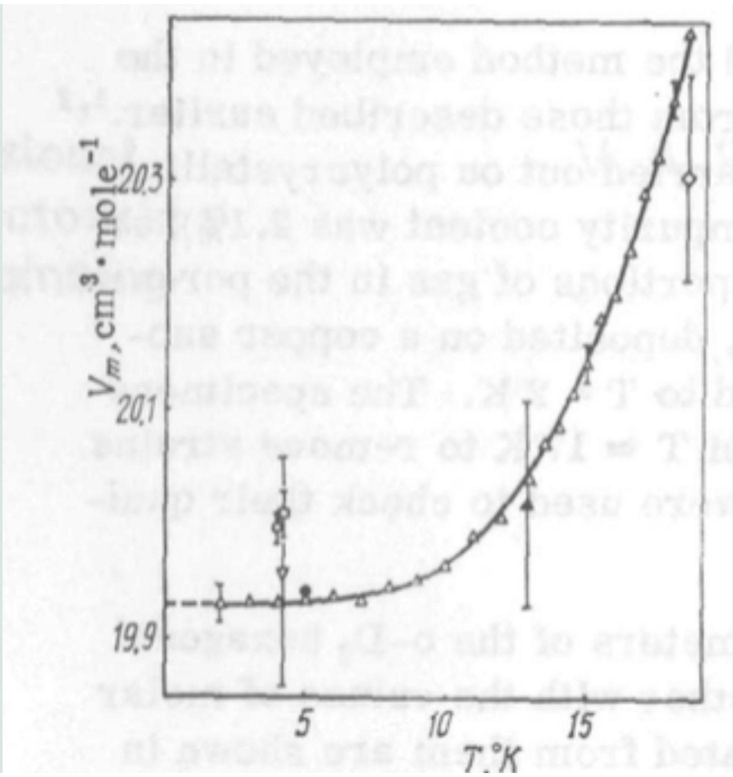

Sov. J. Low Temp. Phys. 10(1), January 1984

Properties of solid deuterium

- SD2 is a quantum molecular crystal with weak intermolecular interaction, hcp or fcc structure
- strongly temperature dependent thermal activated diffusion above 10K
- strongly temperature dependent lattice constant above 10K
- triple-point-wetting

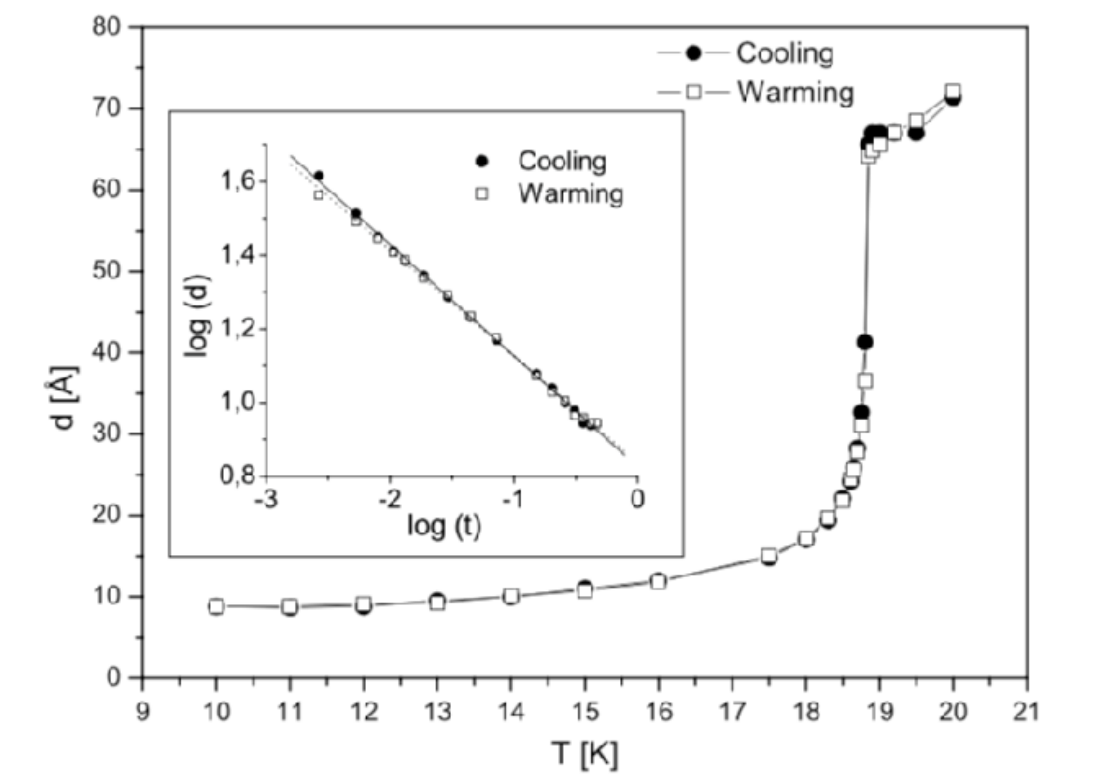

Figure 7. Wetting curves of D_2 on Au are shown. A similar behaviour has been observed for other kinds of adsorbates. In the inset, the same curves are plotted on a logarithmic scale.

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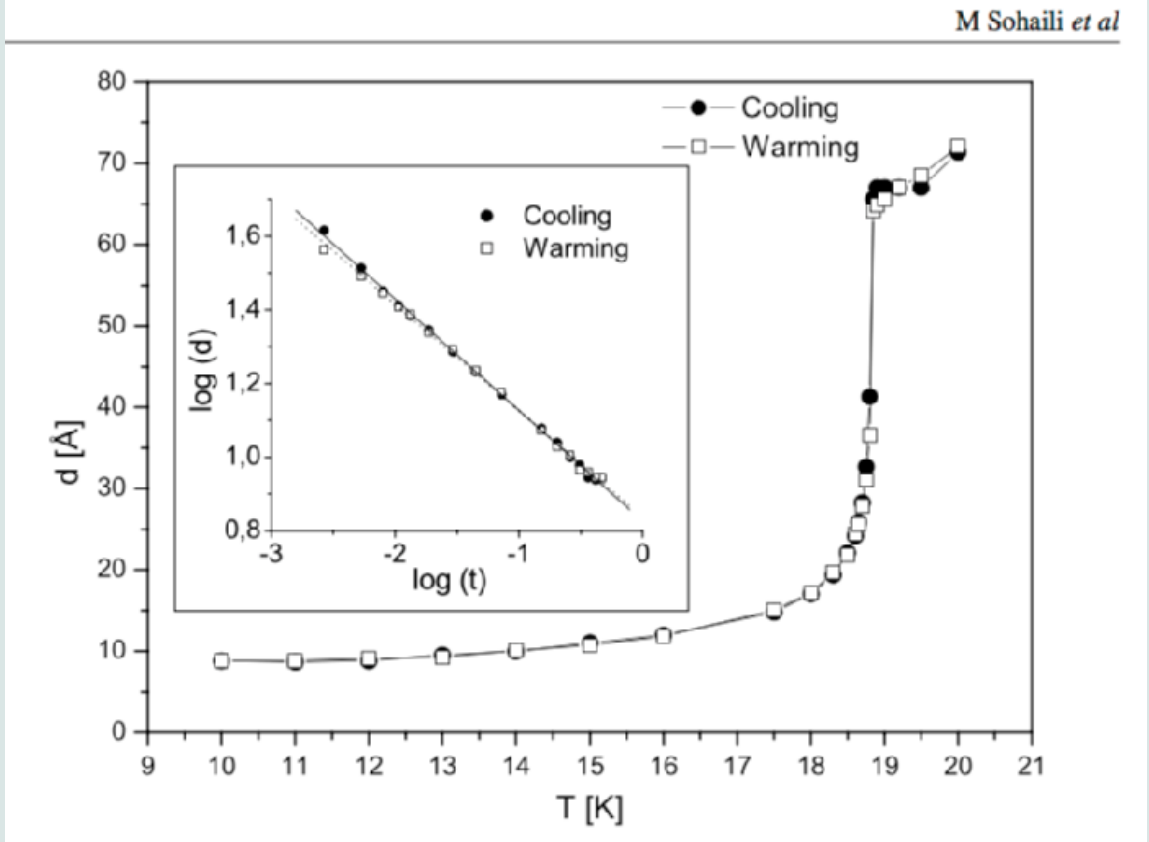

Figure 7. Wetting curves of D_2 on Au are shown. A similar behaviour has been observed for other kinds of adsorbates. In the inset, the same curves are plotted on a logarithmic scale.

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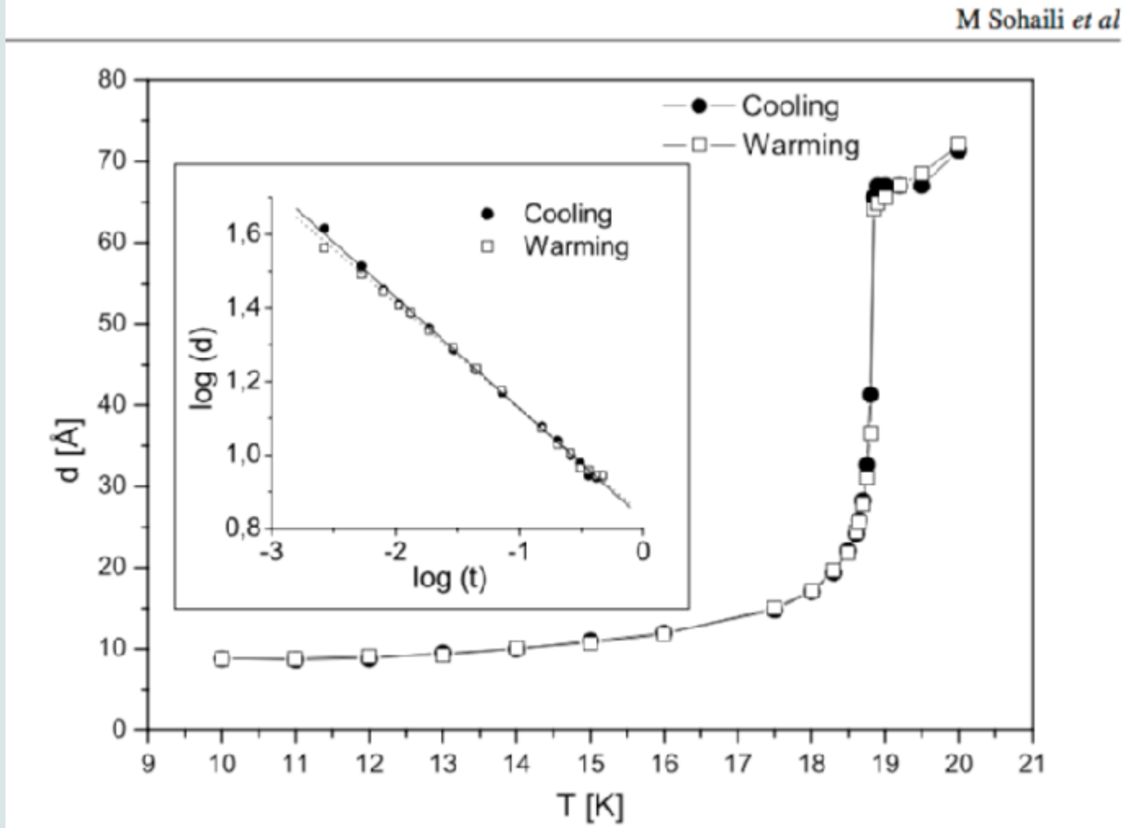

Figure 7. Wetting curves of D_2 on Au are shown. A similar behaviour has been observed for other kinds of adsorbates. In the inset, the same curves are plotted on a logarithmic scale.

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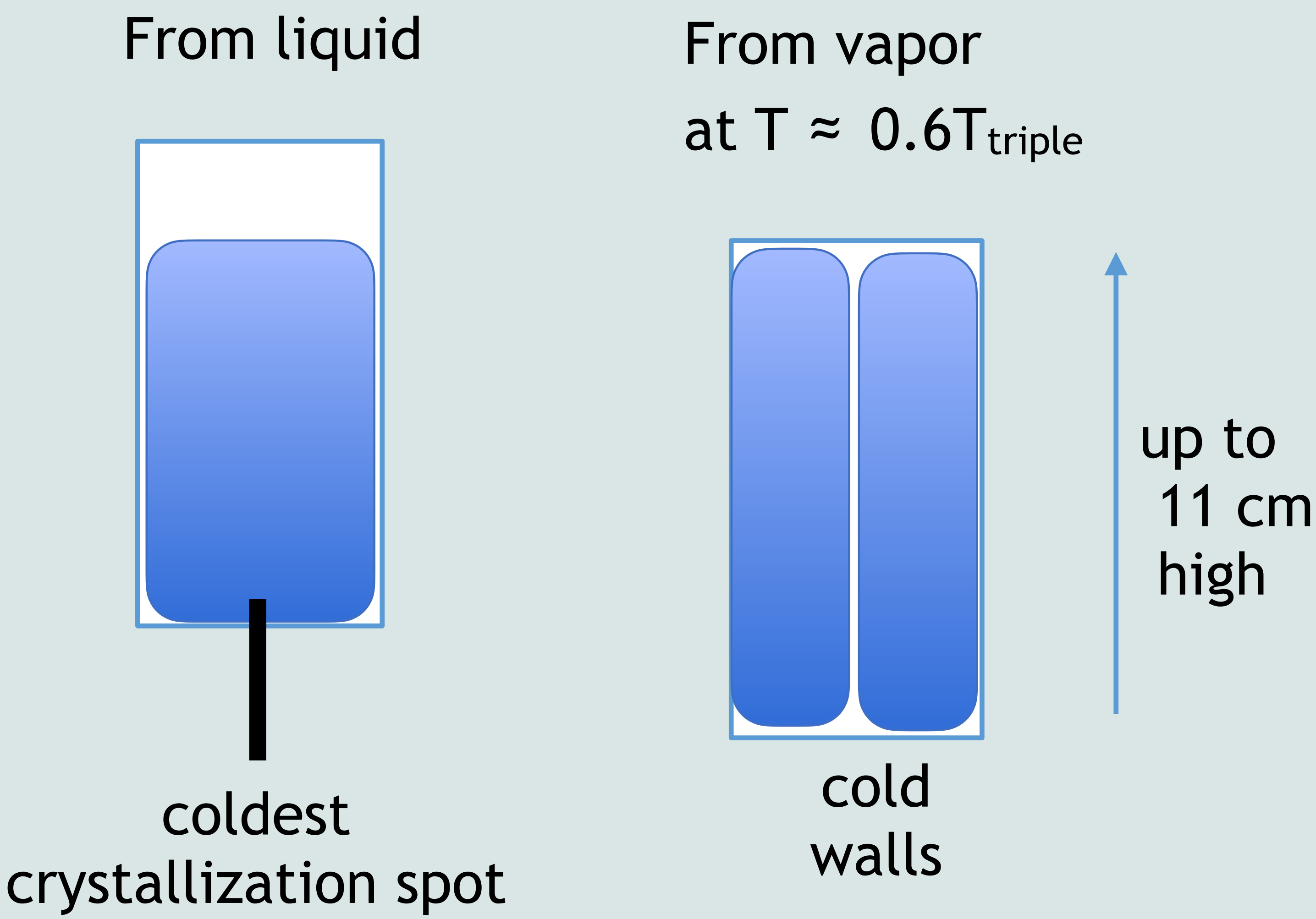
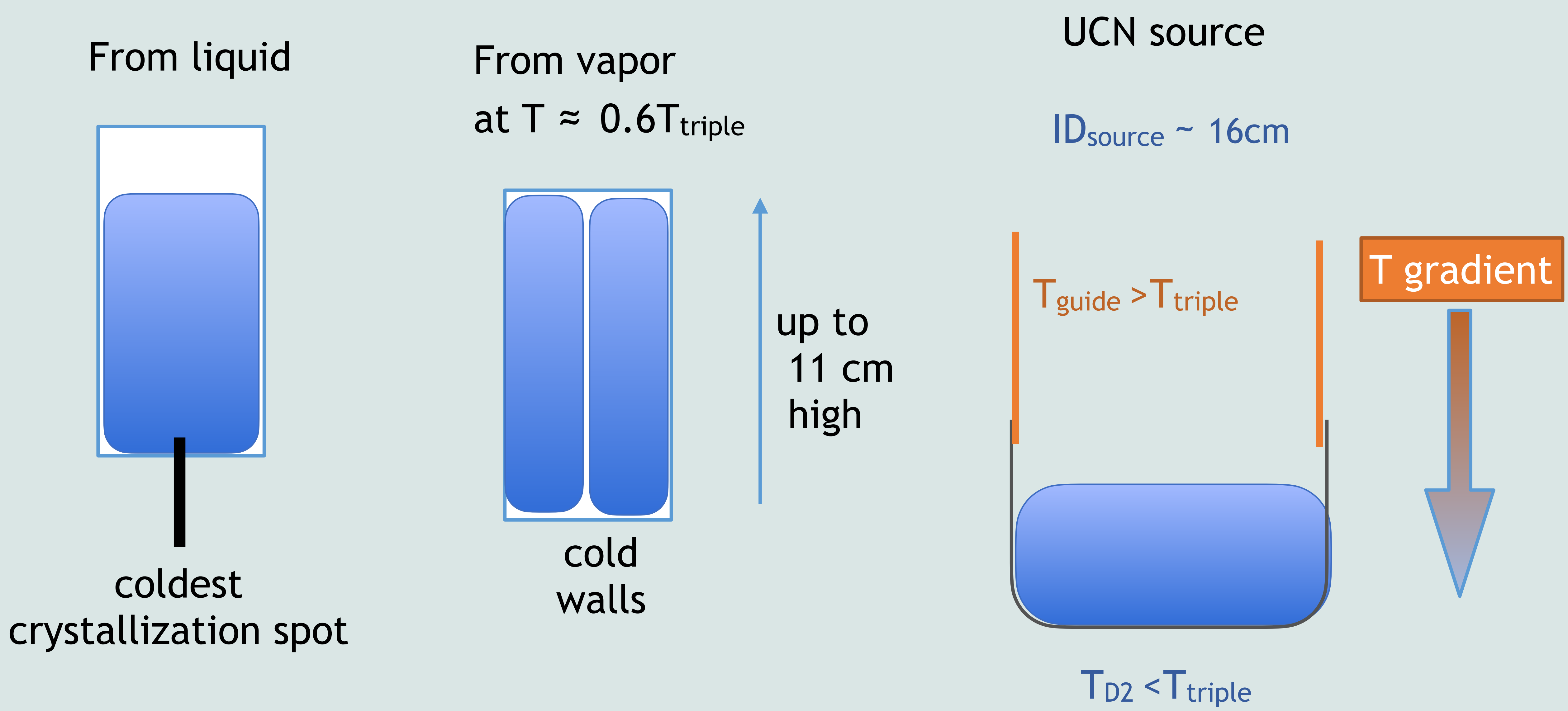
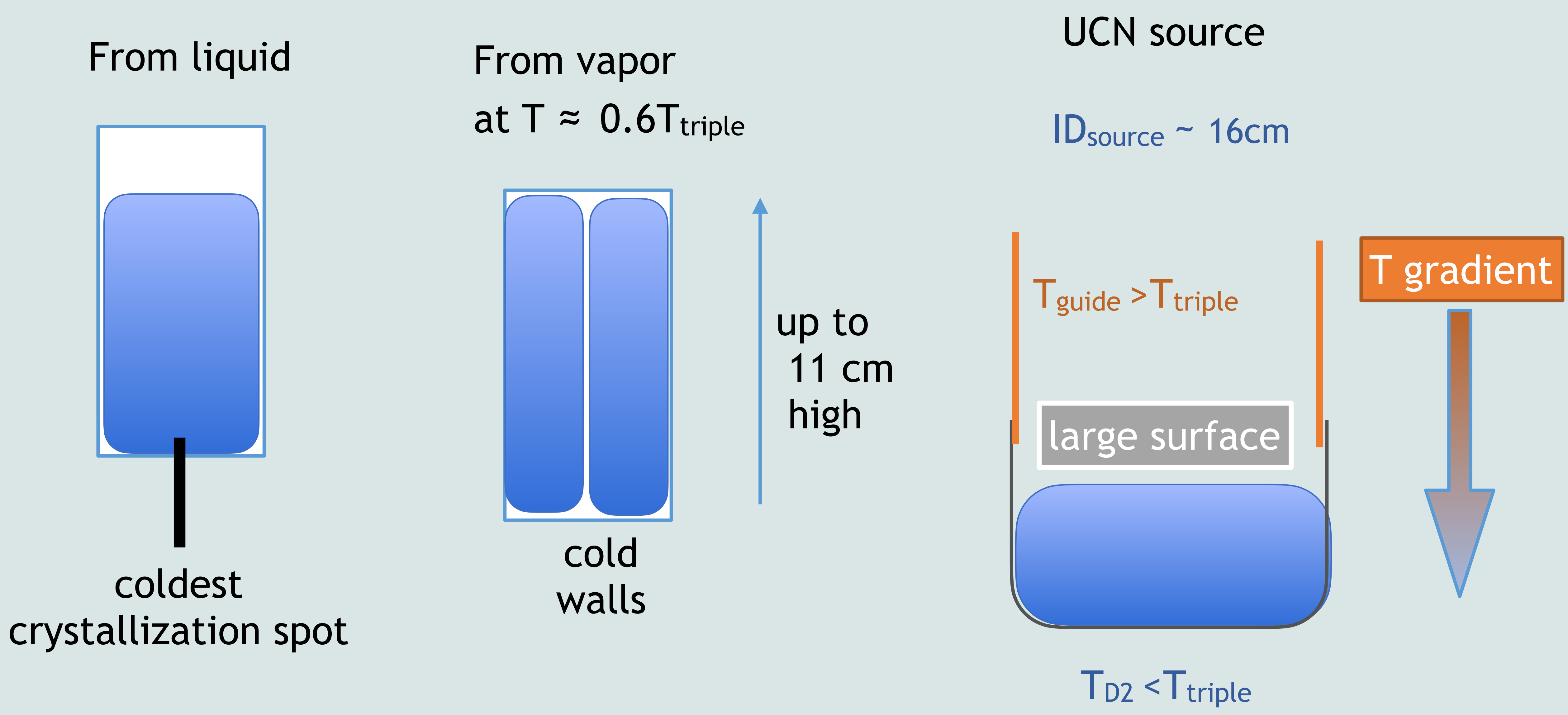
Growing cm size perfect deuterium crystals



small vs UCN source deuterium crystals



small vs UCN source deuterium crystals



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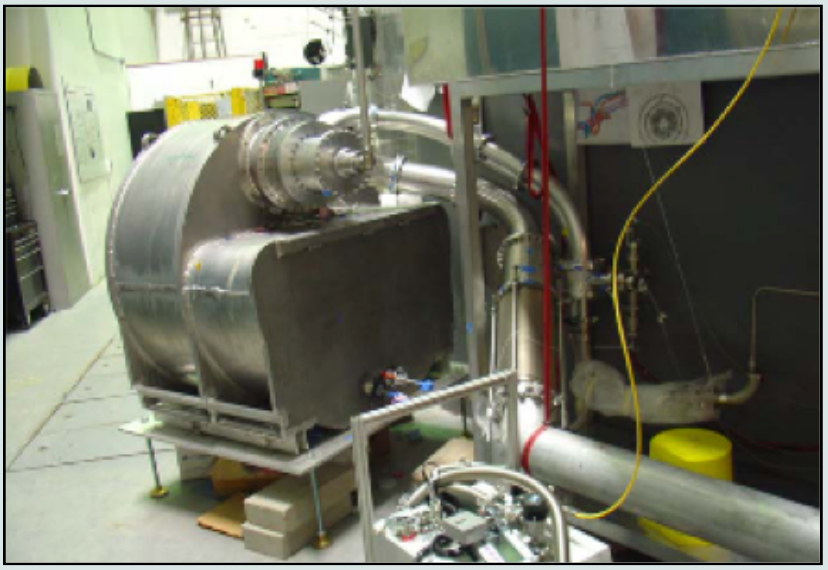
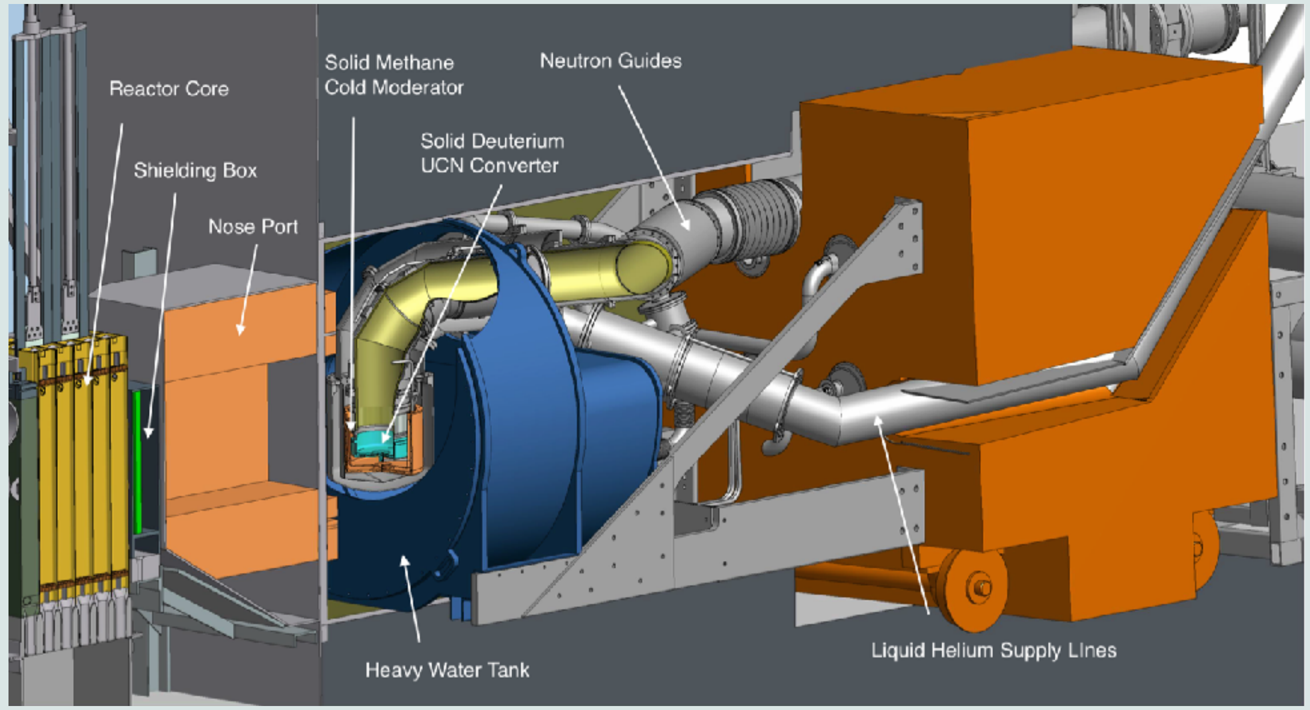
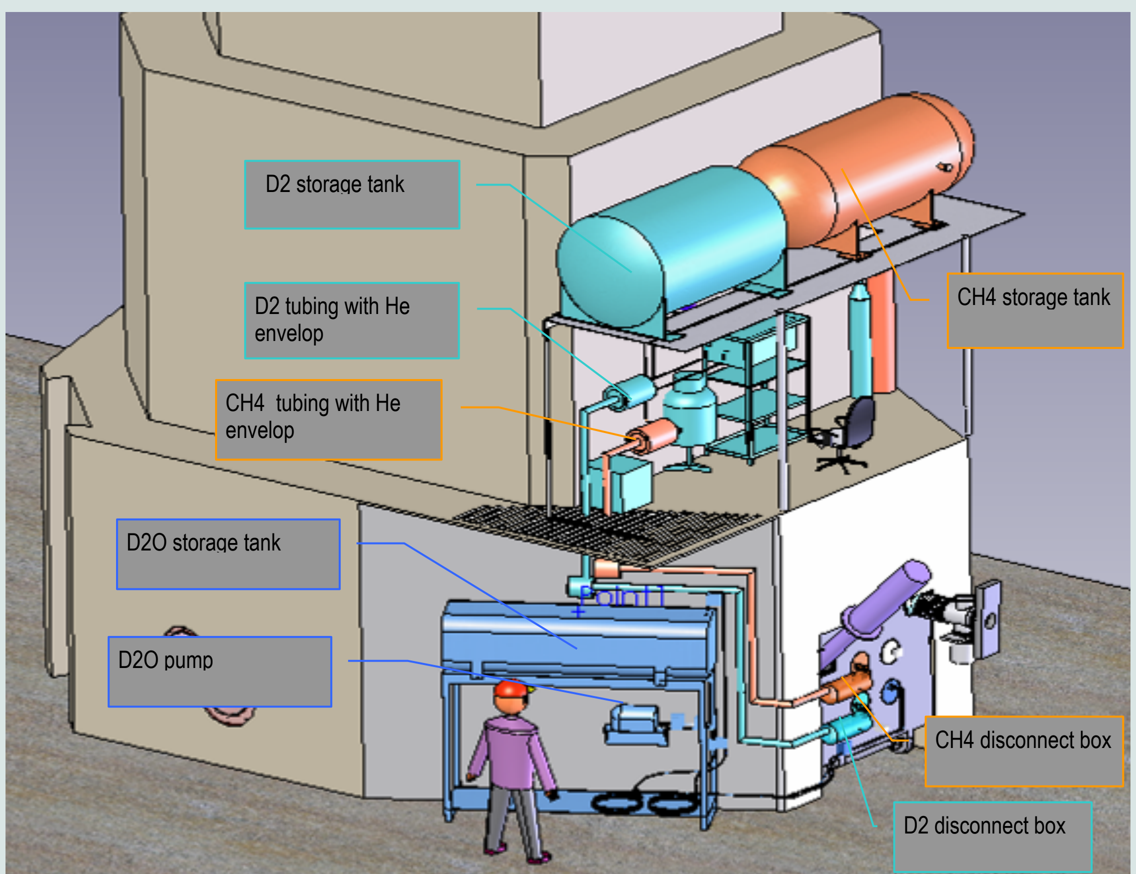
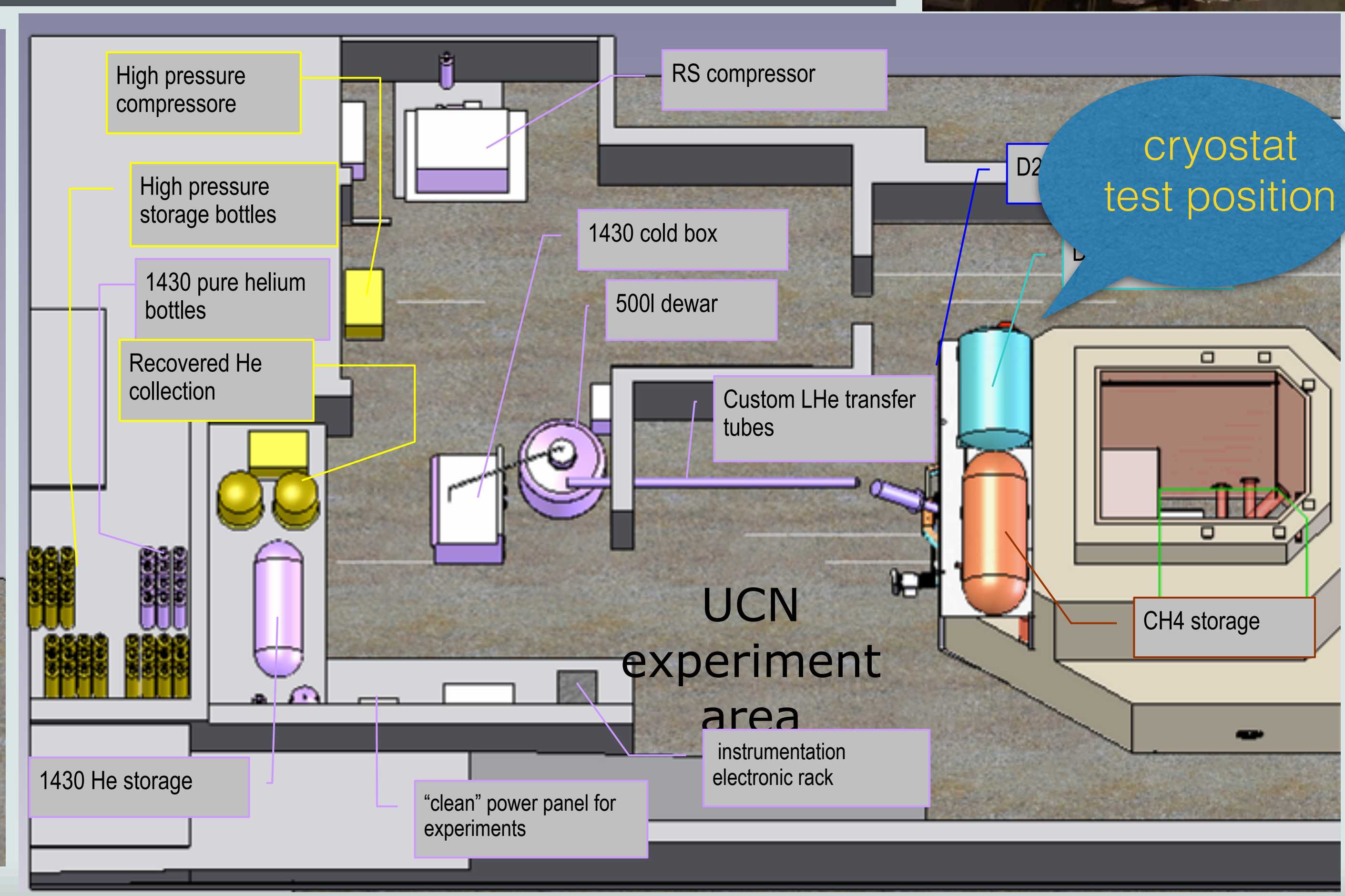
Engineering PULSTART source design

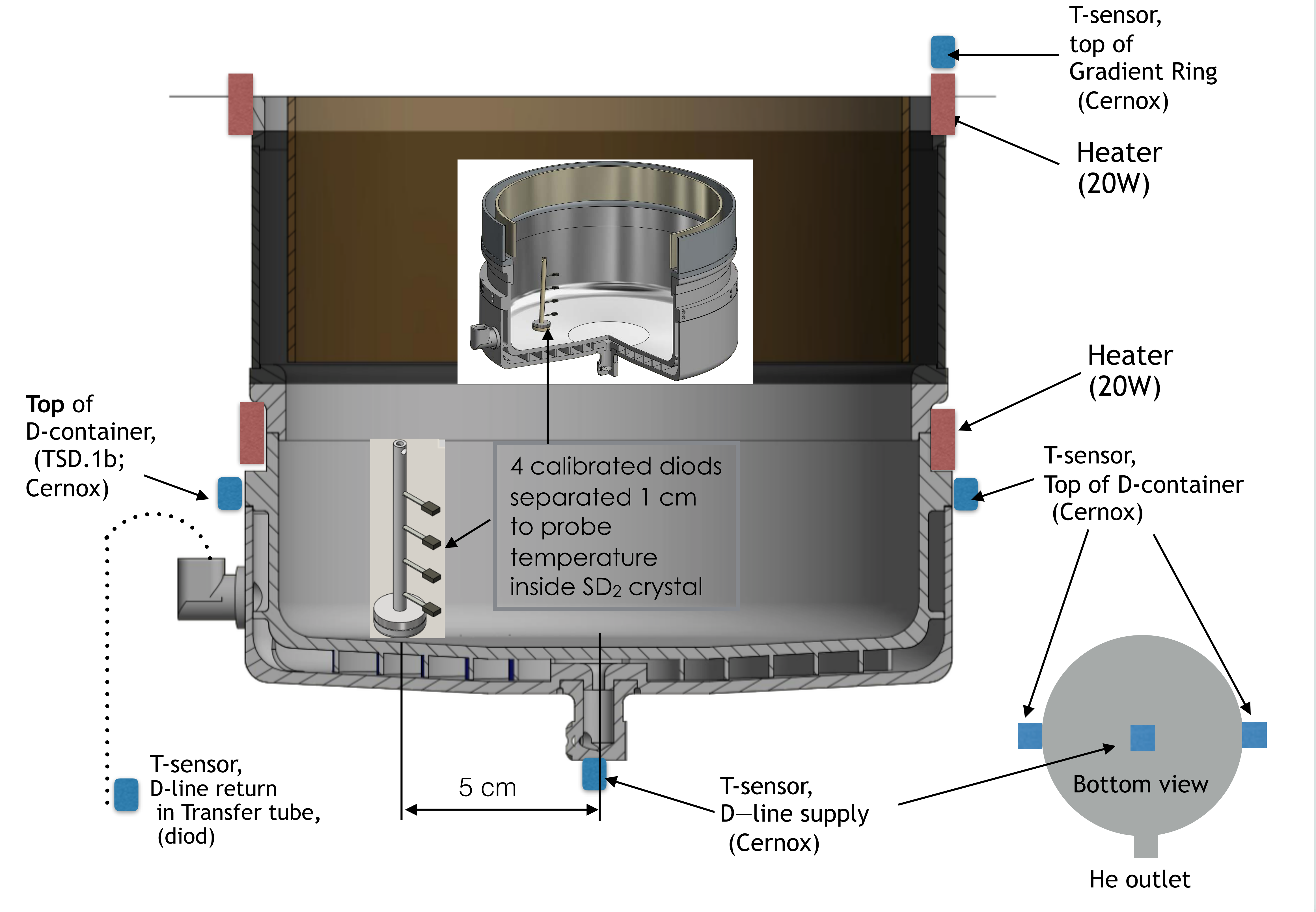






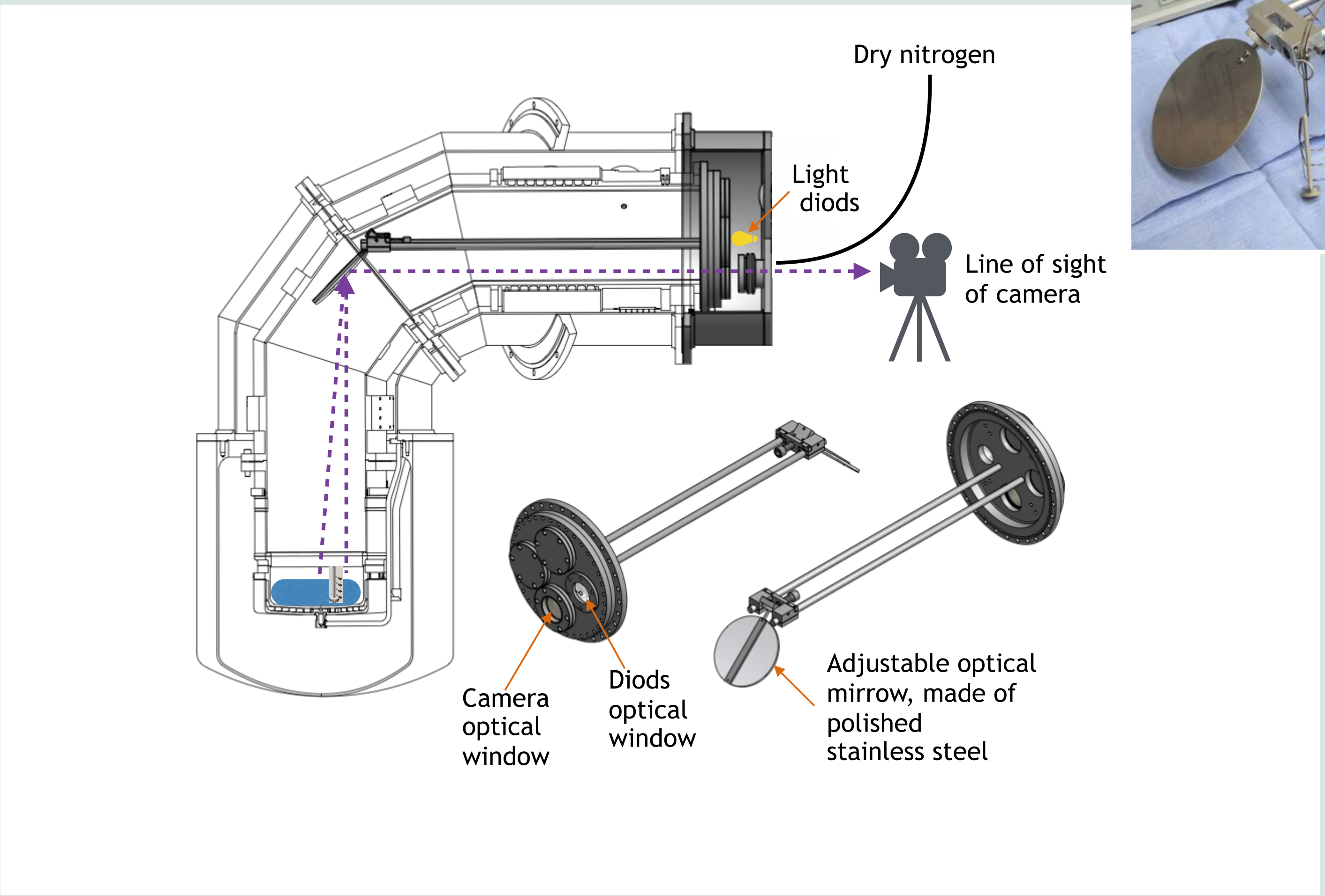


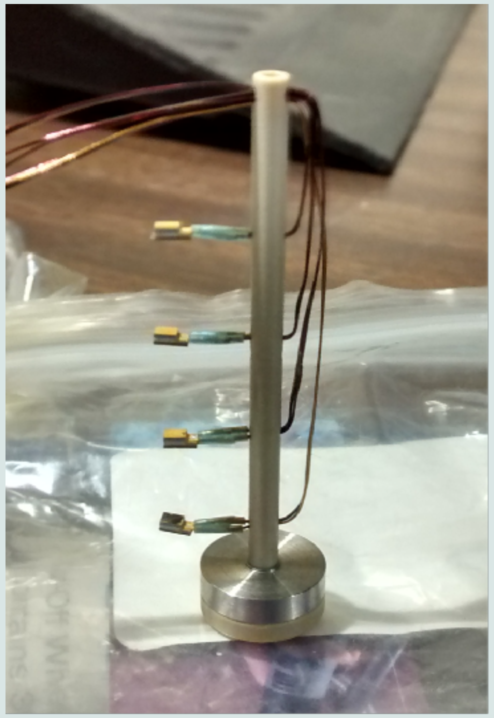
SD2 growing tests : instrumentation details

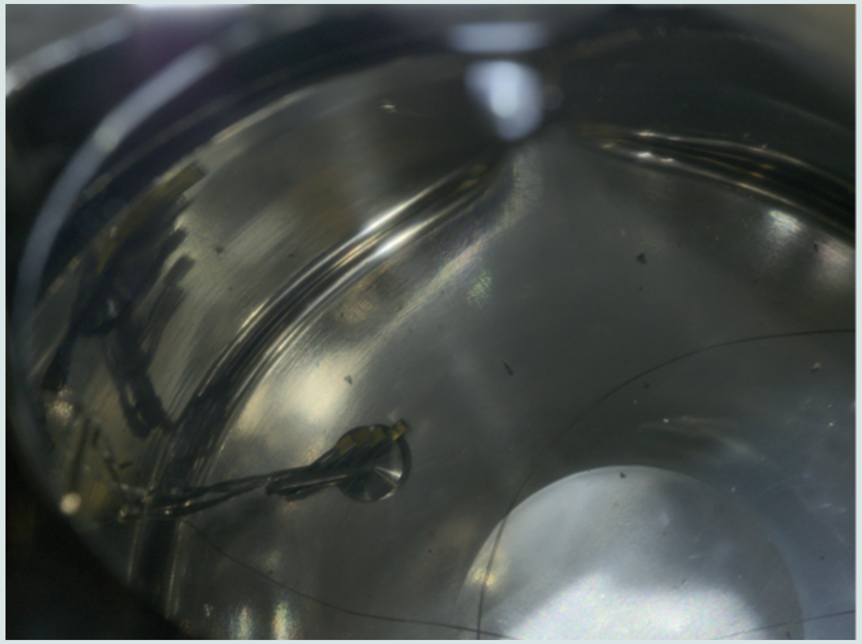


SD2 monitoring system for visual control

- Graham Medlin designed and implemented a monitoring system which allows to observe D2-container by using camera outside cryostat







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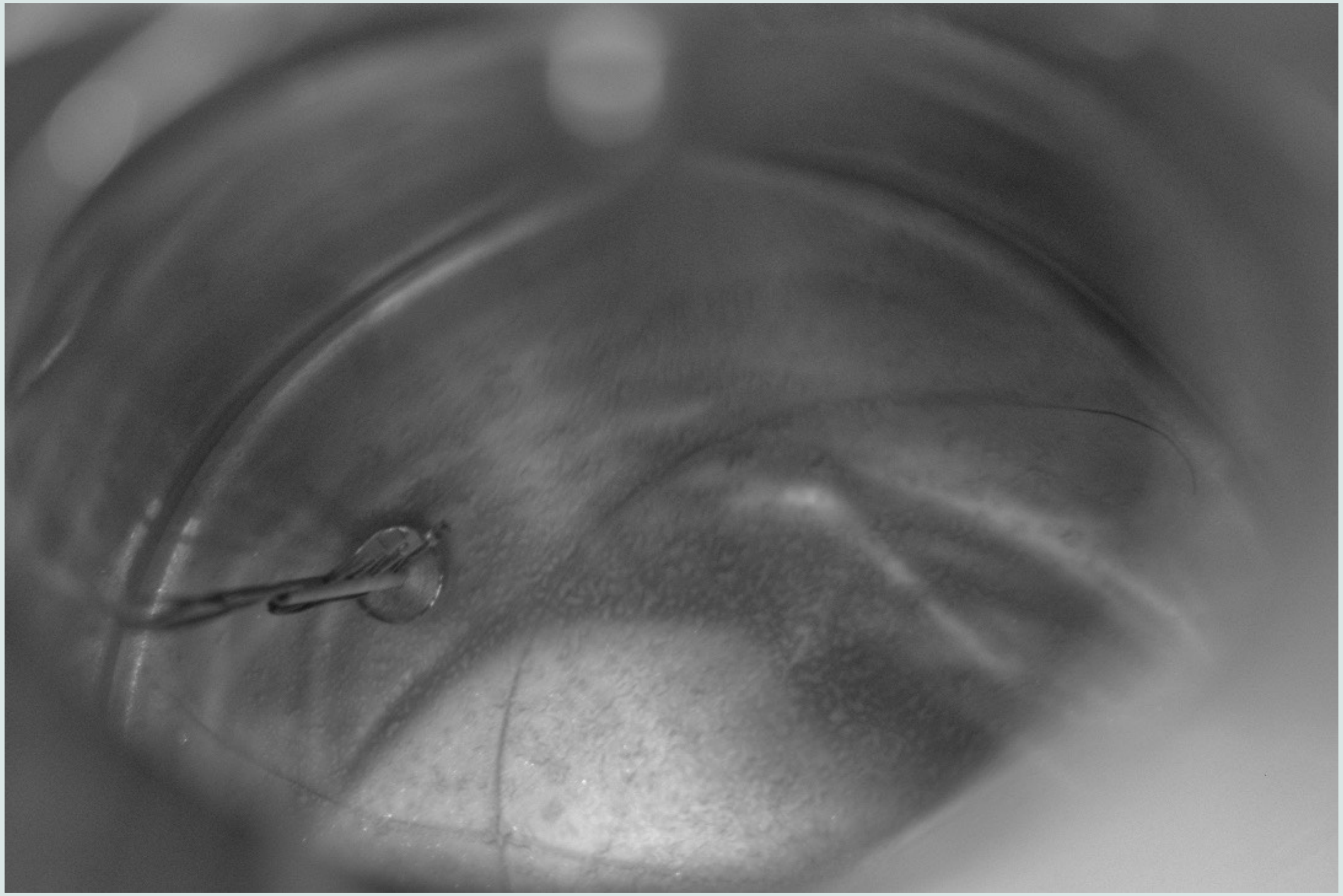
Condensation at **5.4K (inlet)/ 8.2K(top)/ 7.2K(outlet)**:

- this run was to simulated Mainz UCN source condensation of SD2 with cold (6K) bottom of container and slow D2 flow rate



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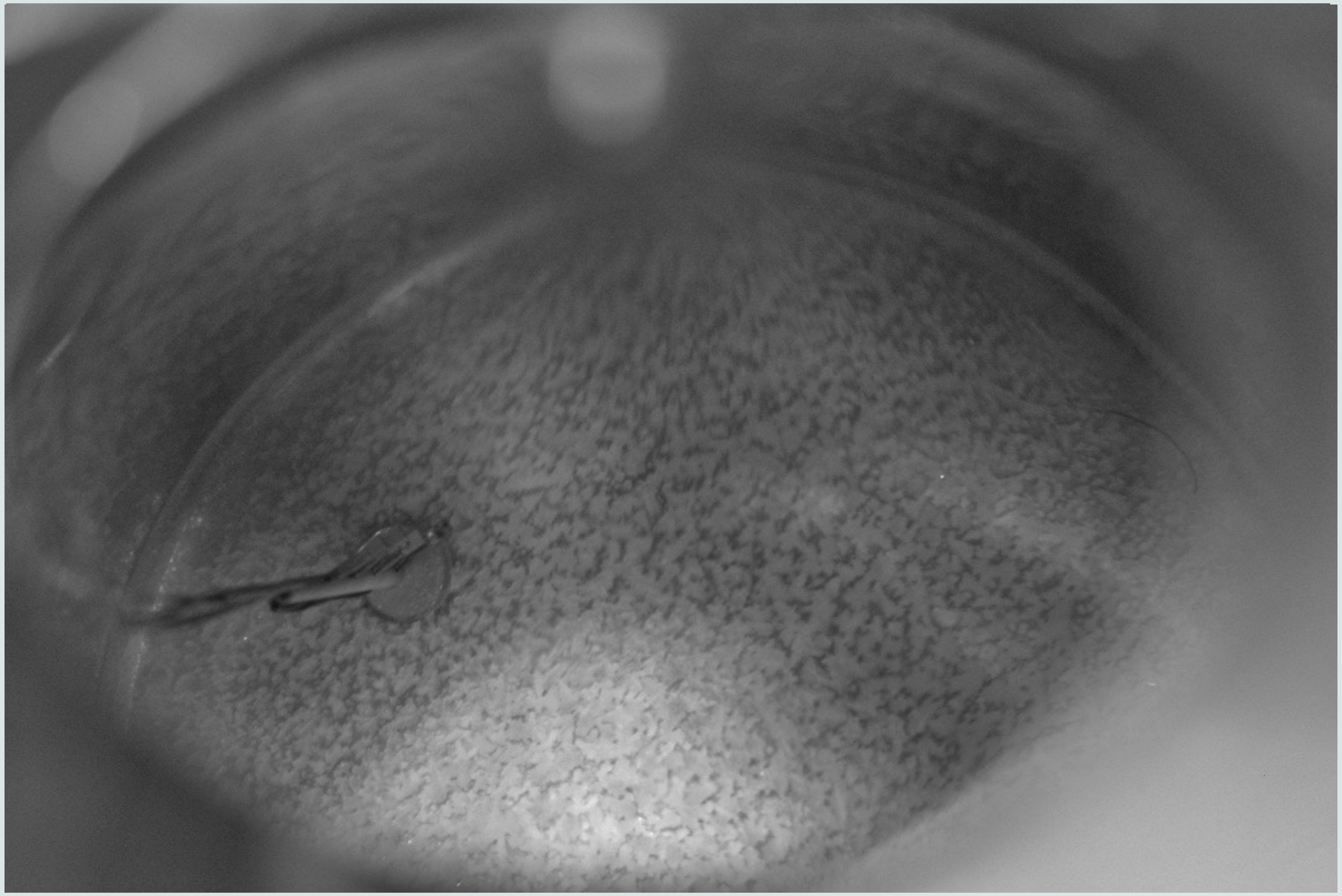

Condensation at **5.4K (inlet)/ 8.2K(top)/ 7.2K(outlet)**:

- this run was to simulated Mainz UCN source condensation of SD2 with cold (6K) bottom of container and slow D2 flow rate

- Small flow (0.3 l/m) produced dense multicrystall, optically opaque

Condensation at **5.4K (inlet)/ 8.2K(top)/ 7.2K(outlet)**:

- Higher D2 flow >1 l/m produced snow-flake-like mass

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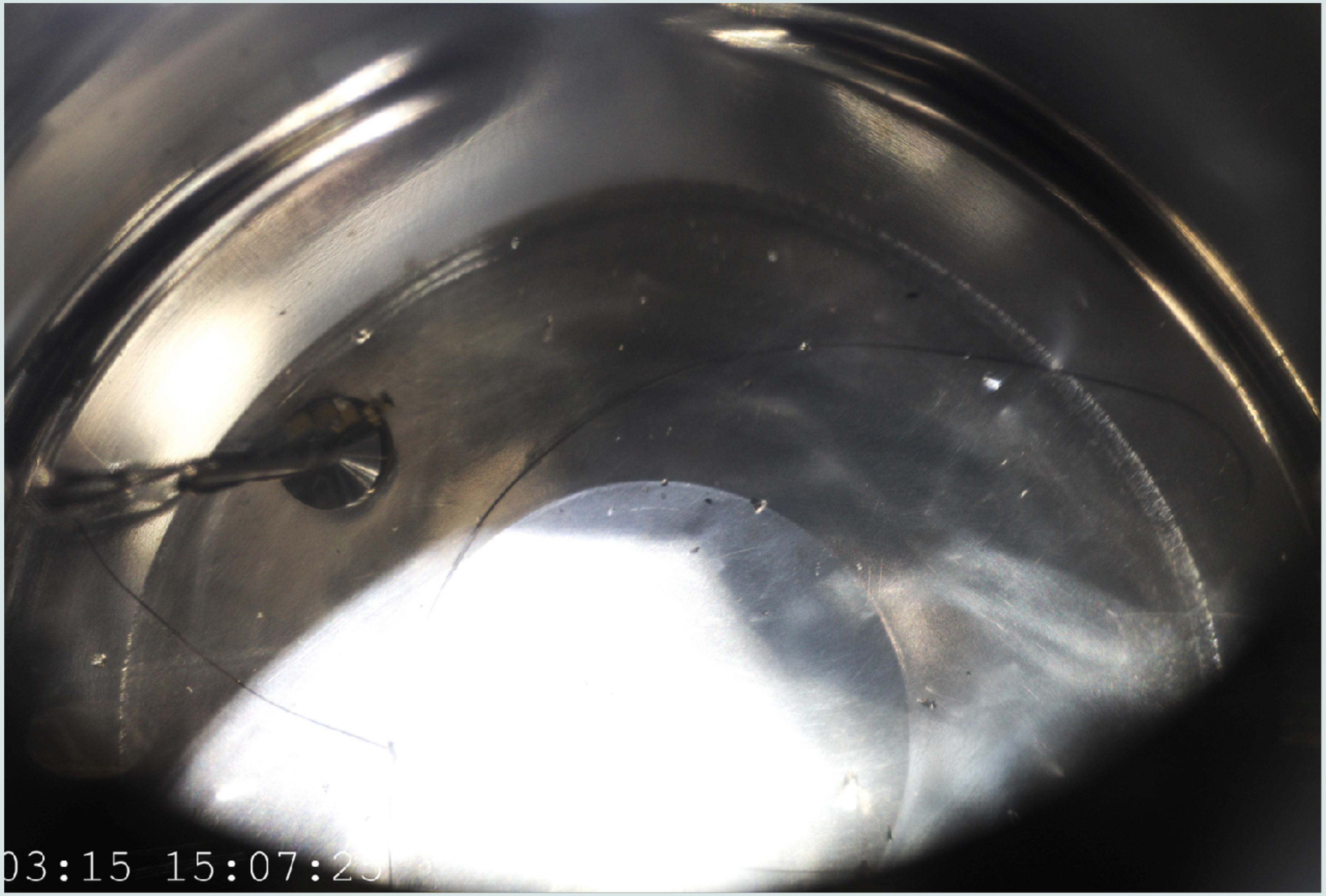
condensation at **9/ 17.5/ 17.5, D2 flow 0.8 l/m**



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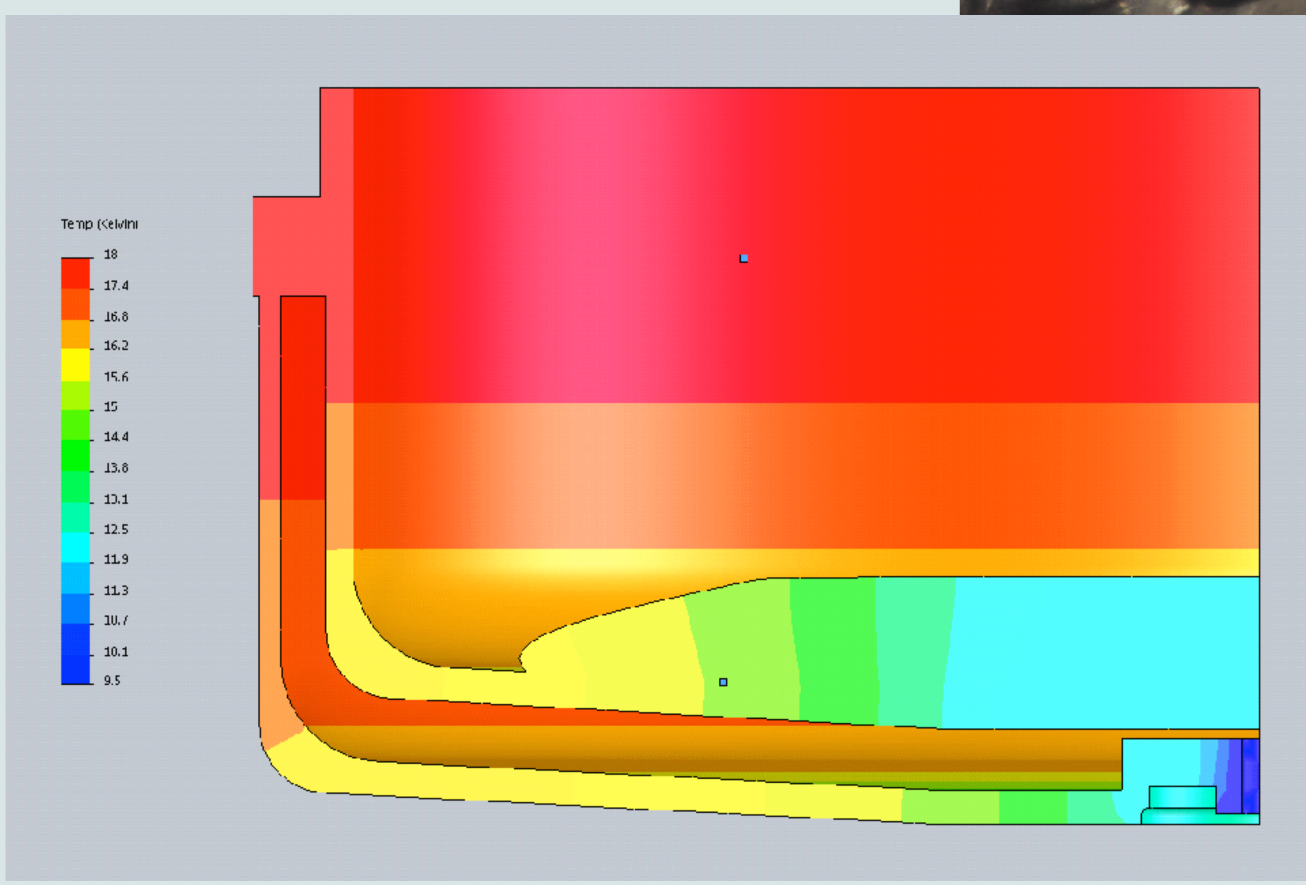


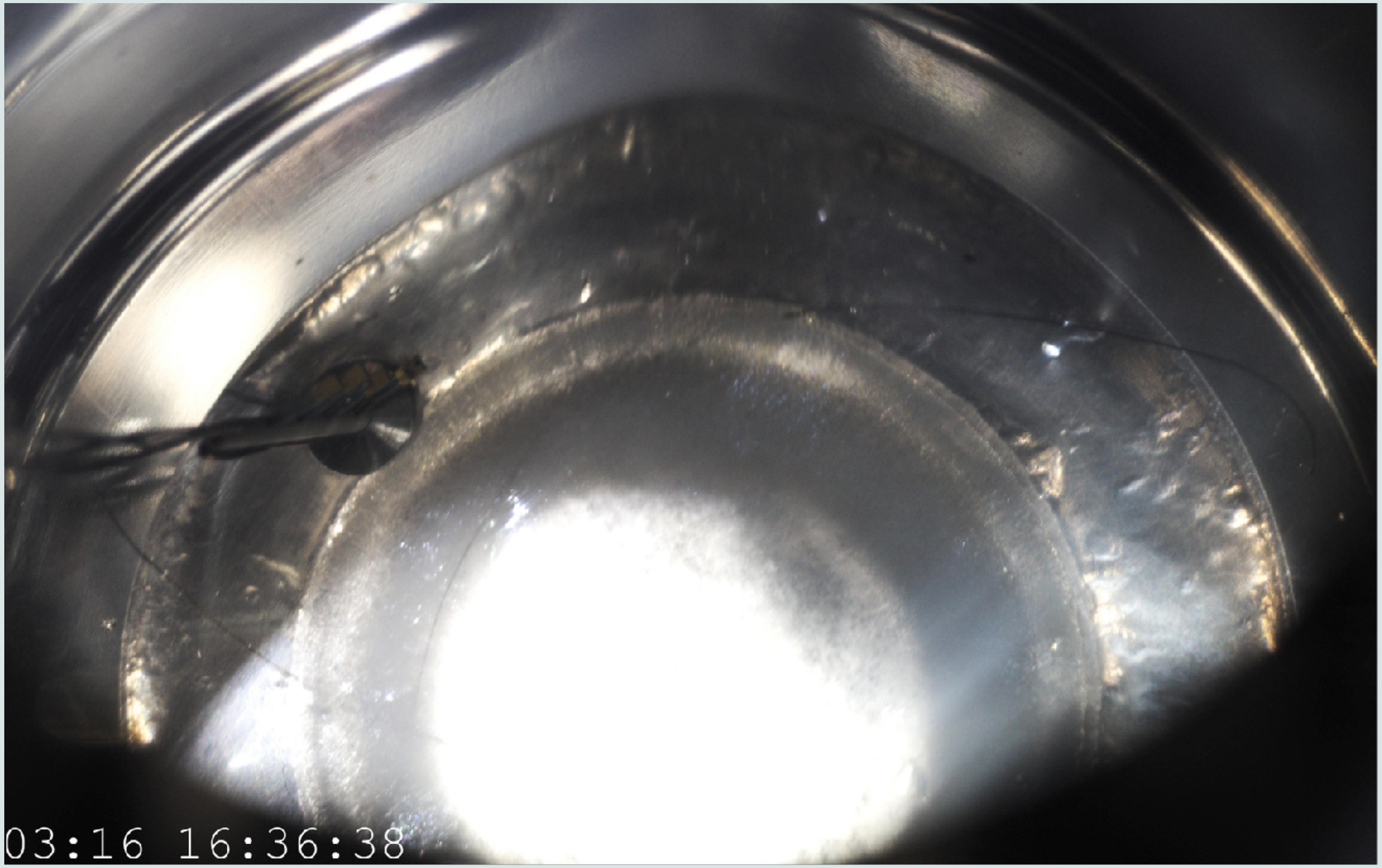
shapes

- Mar 15-16-18, evolution to blob at 11.5K

Shapes

- Mar 15-16, evolution to blob at 11.5K

- Mar 16 condensation, with heaters on, 8.5/ 18/ 15K, 1 l/m

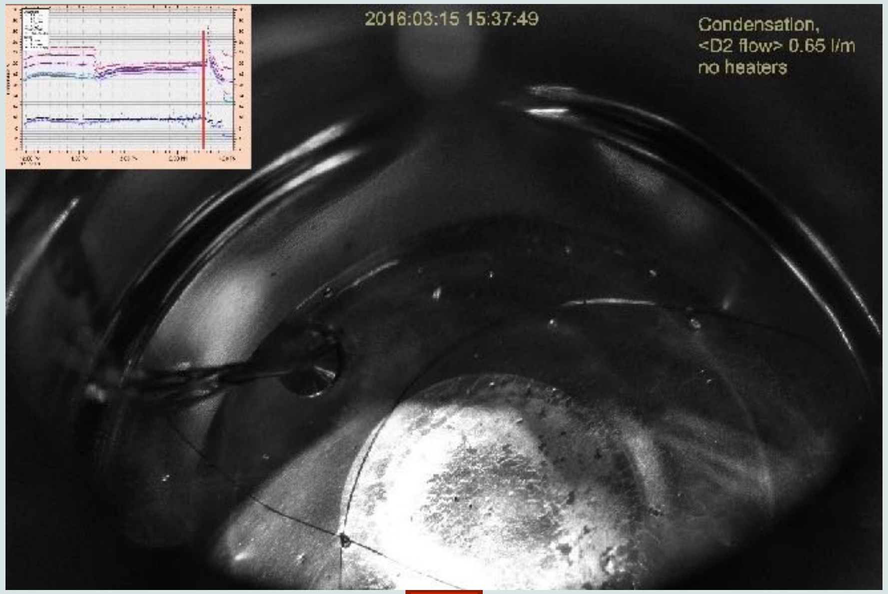


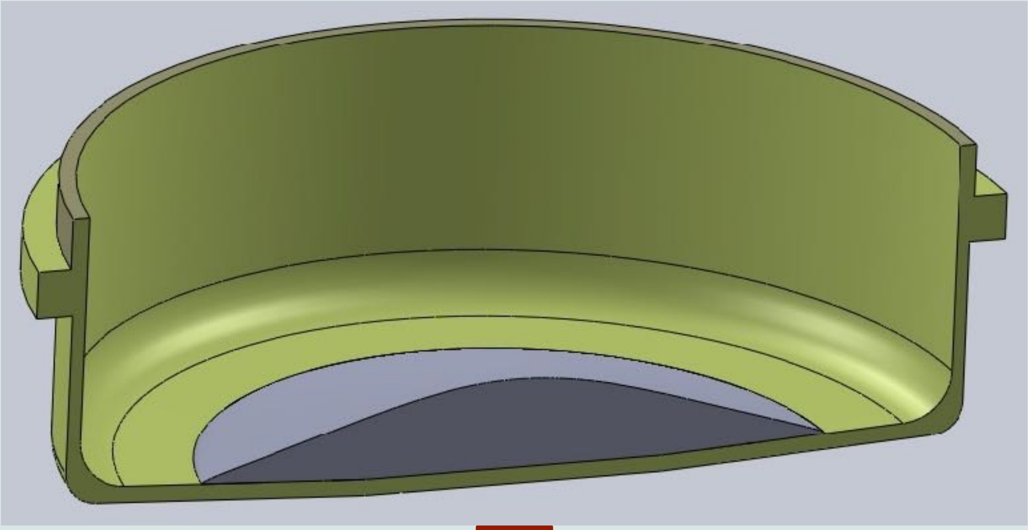
Shapes

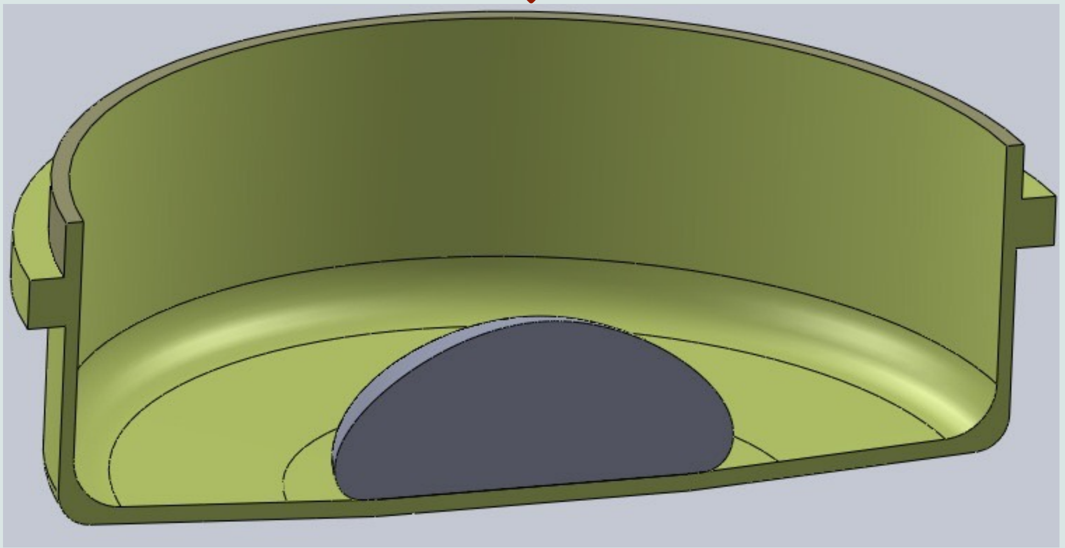
- Mar 17-18, evolution to blob at 11.5K
- tendency for avoiding warmer surfaces

Shape reconstruction

Mar 15, Annealed overnight at 12K. High mobility







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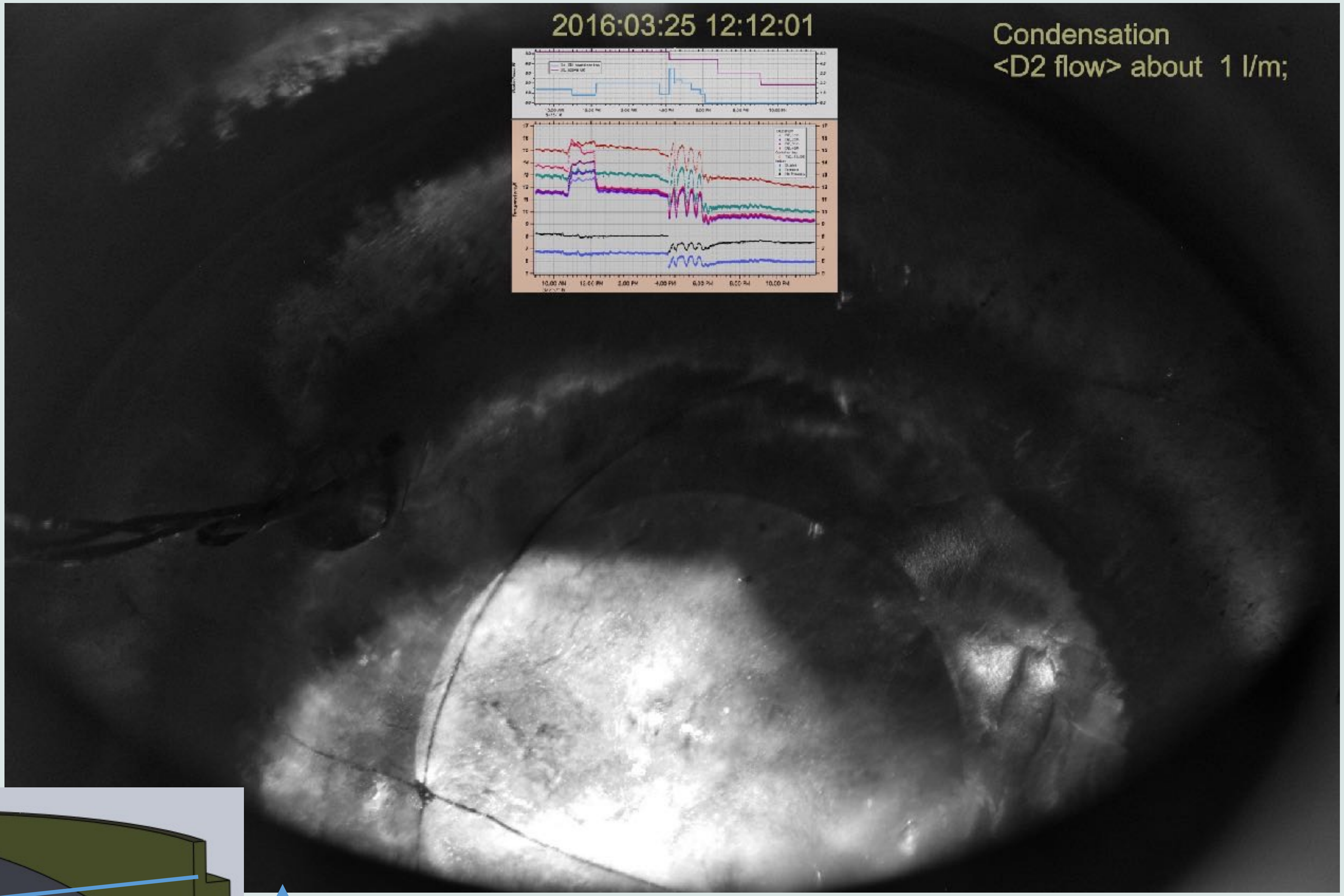

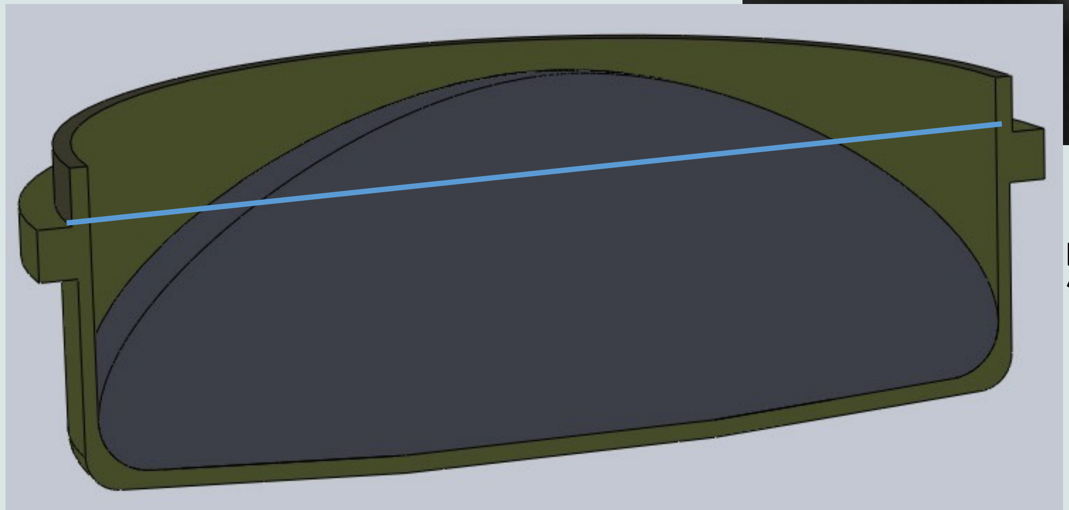

5 cm

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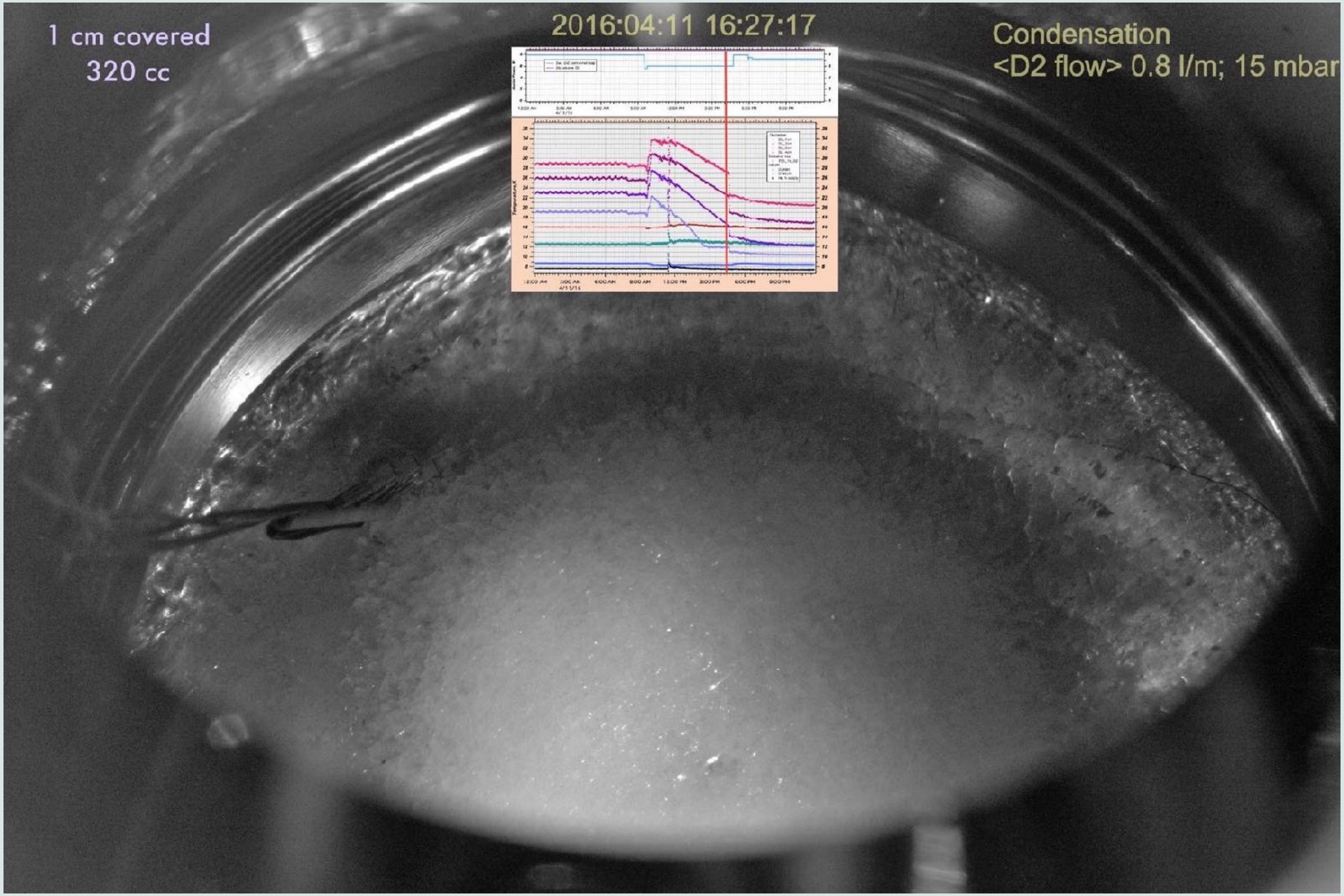
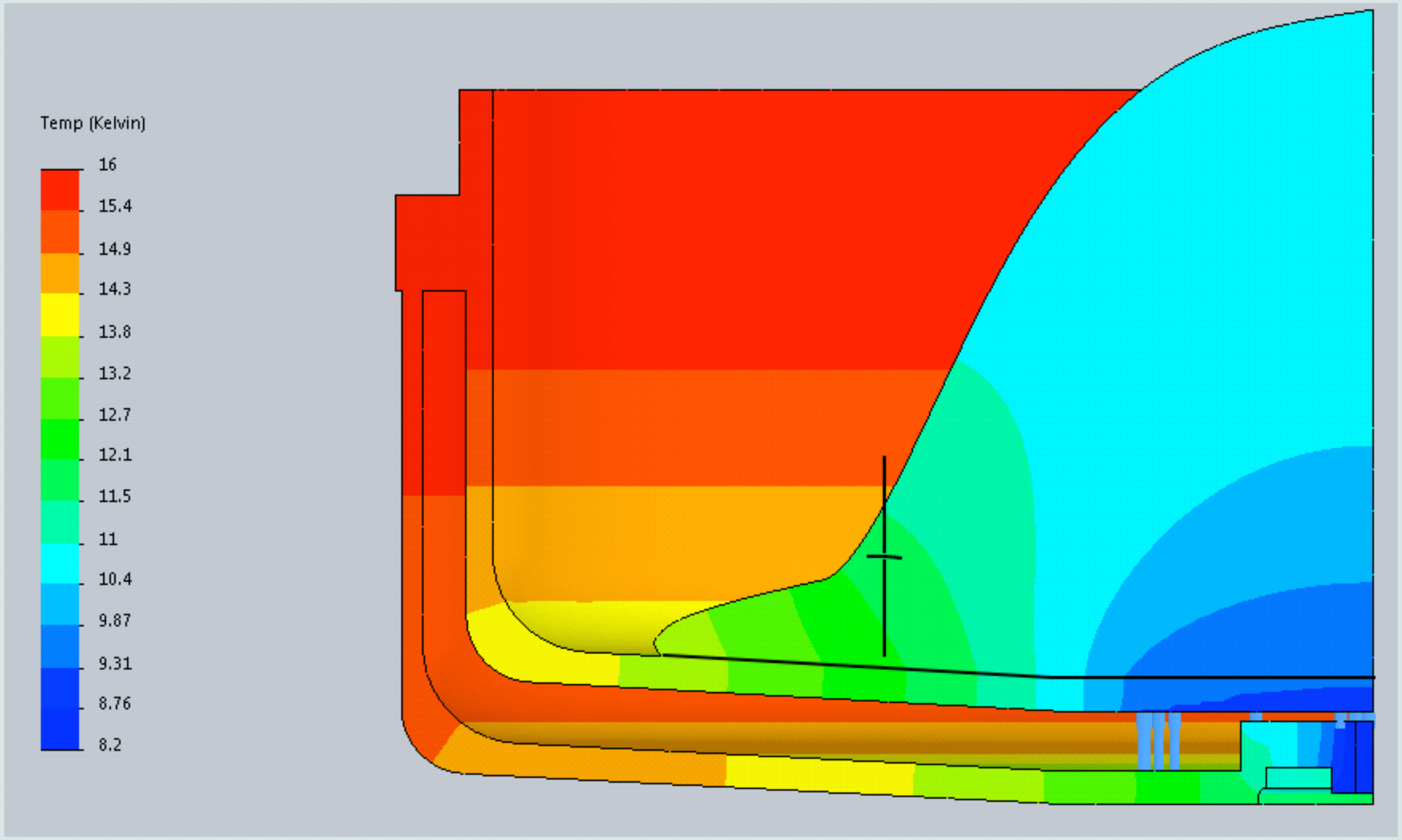
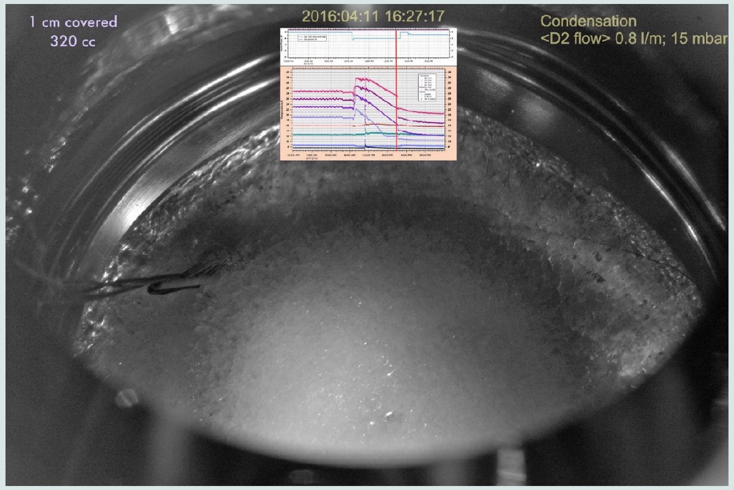
Condensation at 8.3/ 16.2/ 13, 0.8 l/m



April 11 simulation, D2 flow on and off

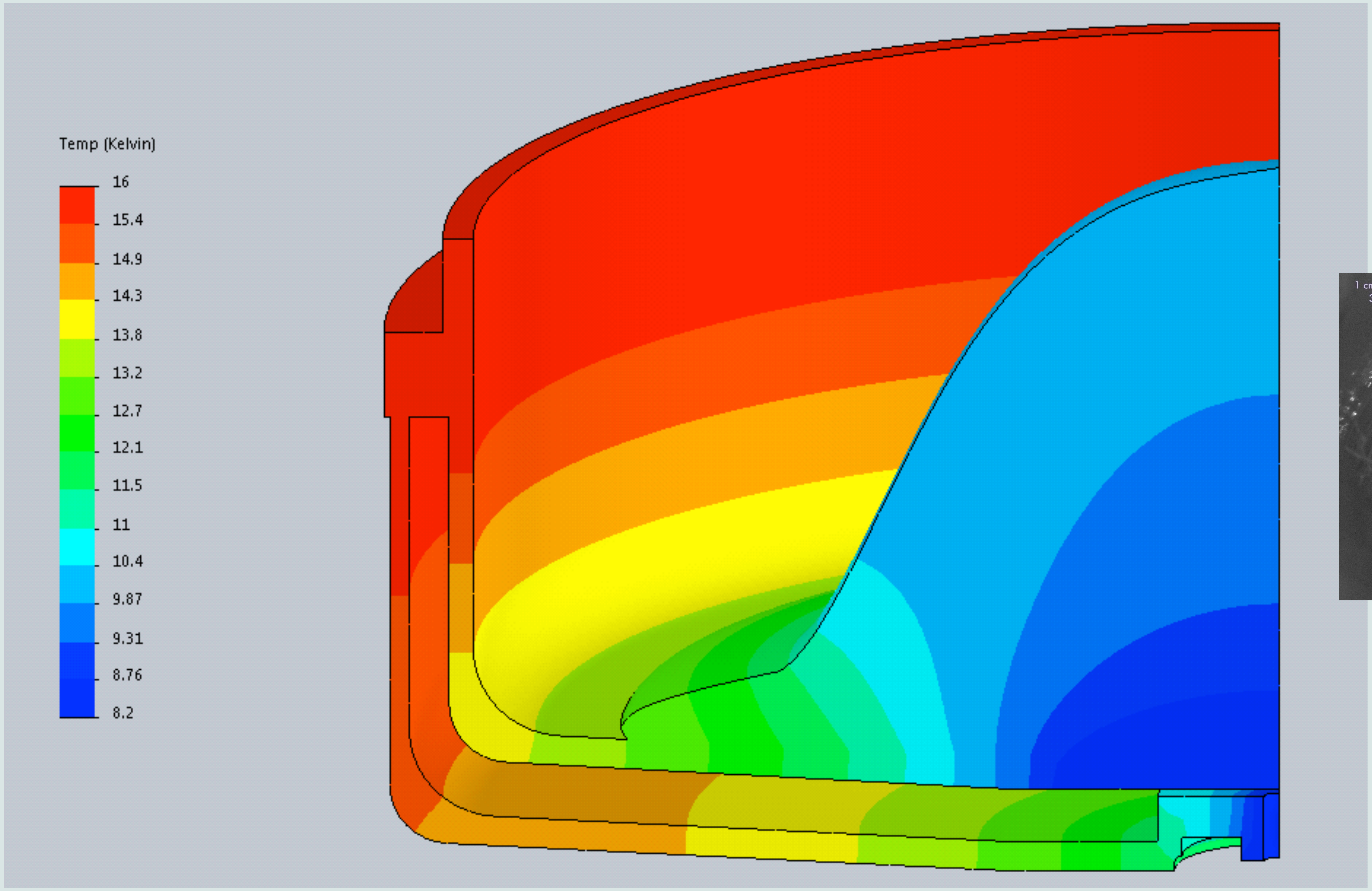
- 1cm temperature can be reproduced only when assuming that bottom circle is at 9.3K
- Transparent region start from above 12K

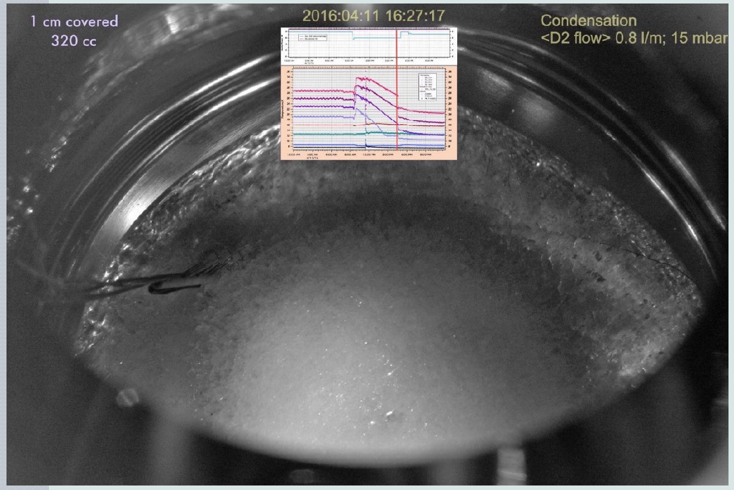




April 11 simulation, D2 flow on and off

- after D2 flow was off, 1 cm dropped to 11K, while the container has not changed, it consistent with Bottom circle at the same T as D-inlet





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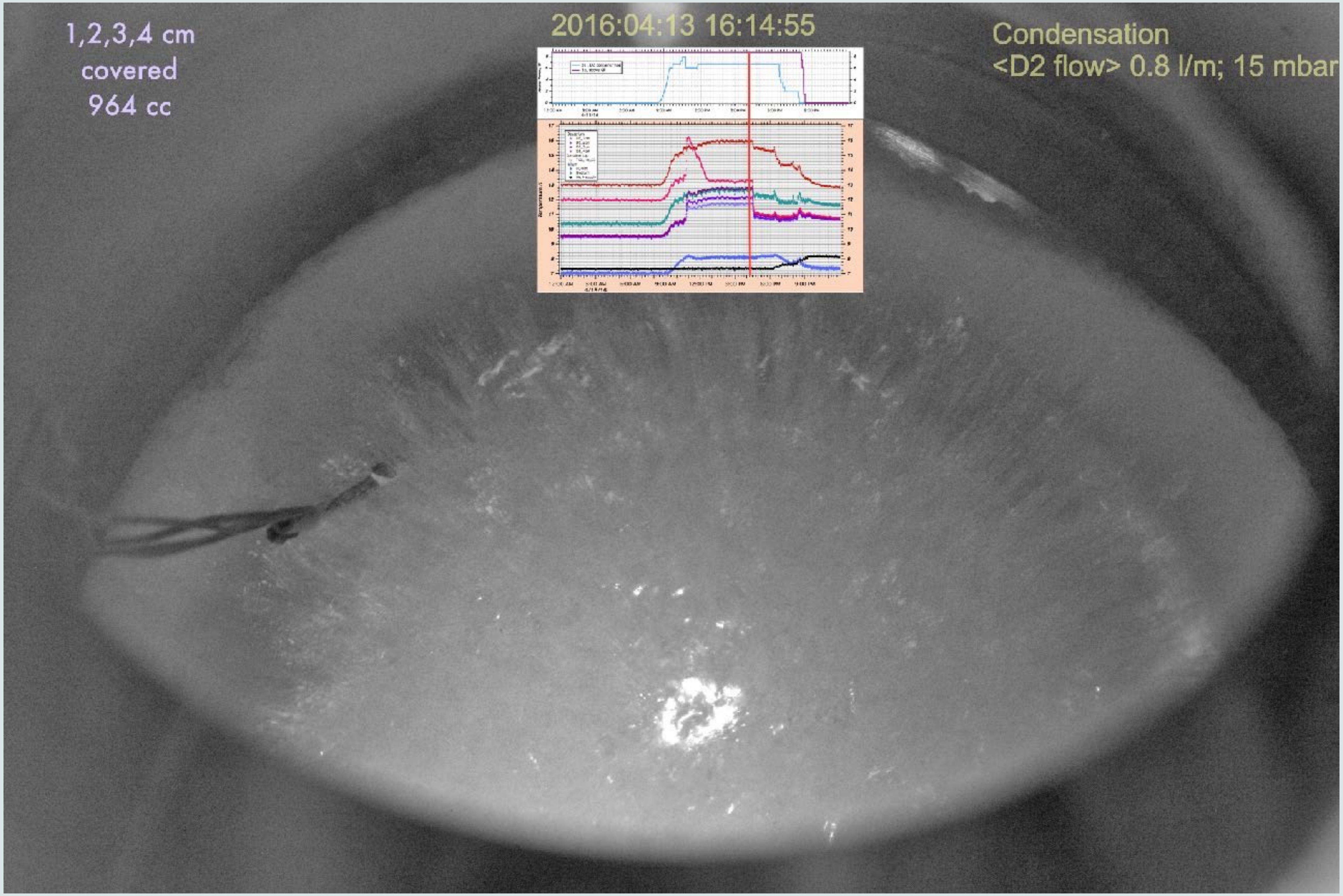

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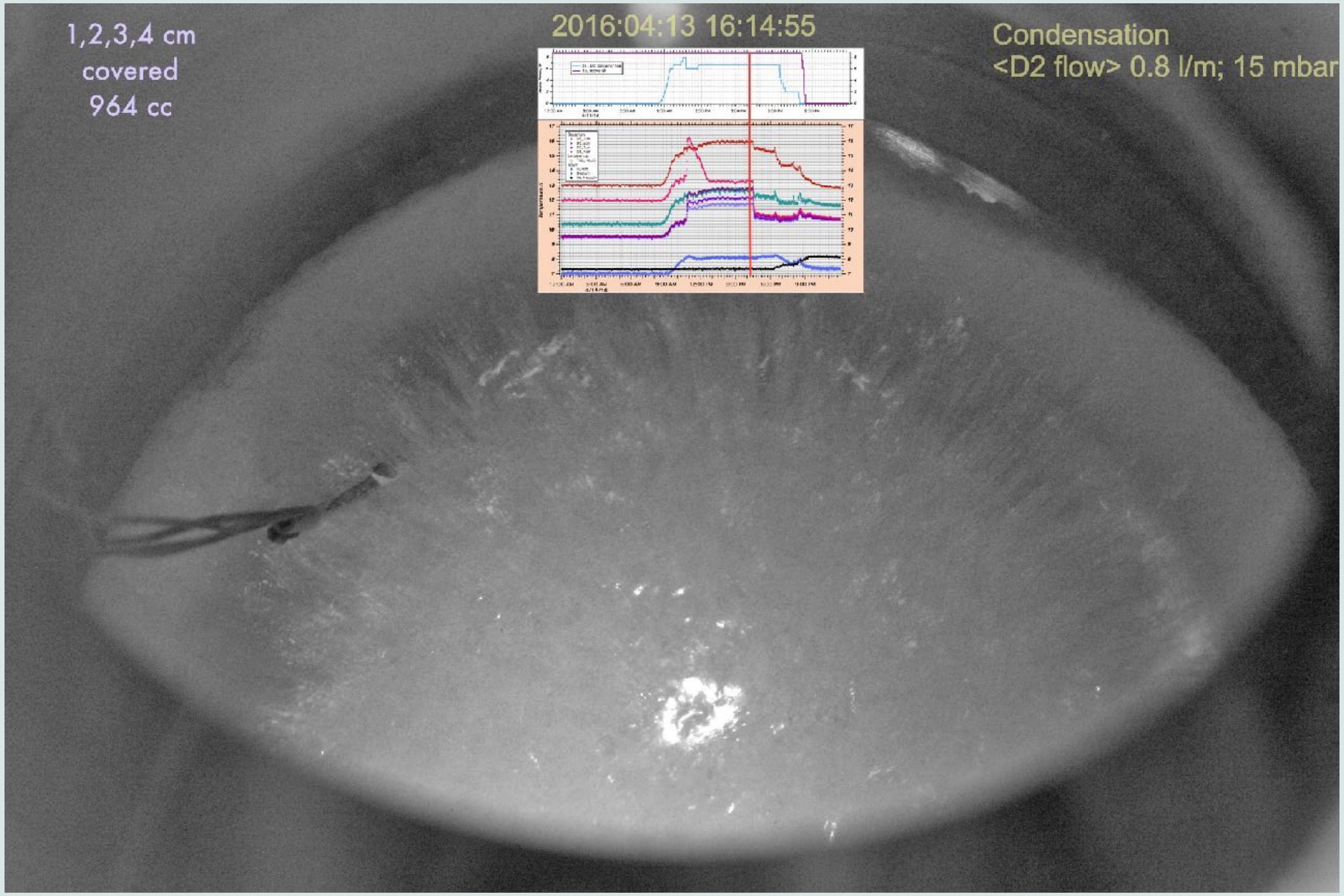

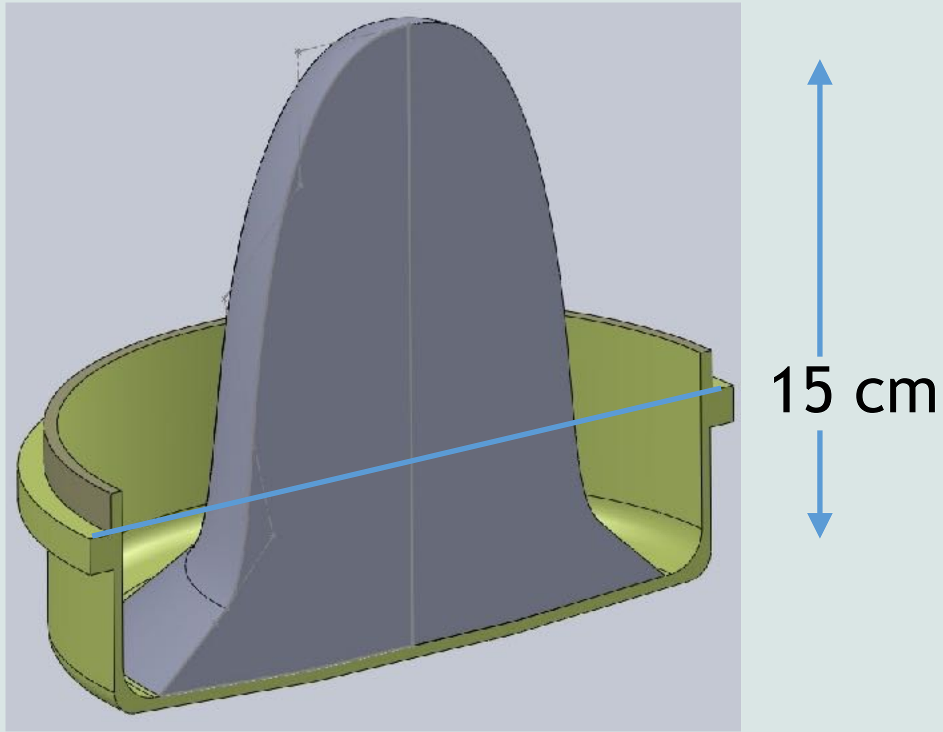

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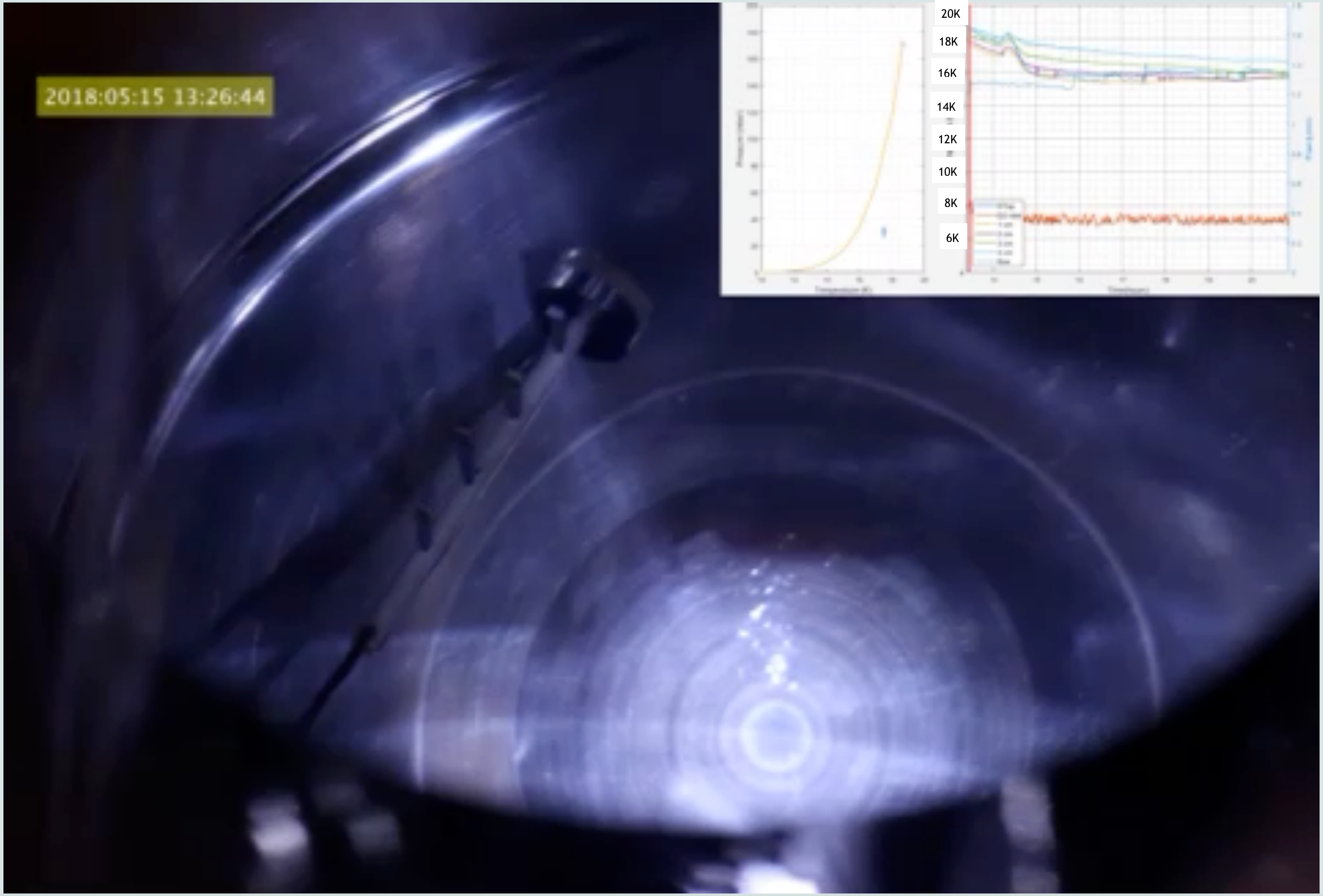
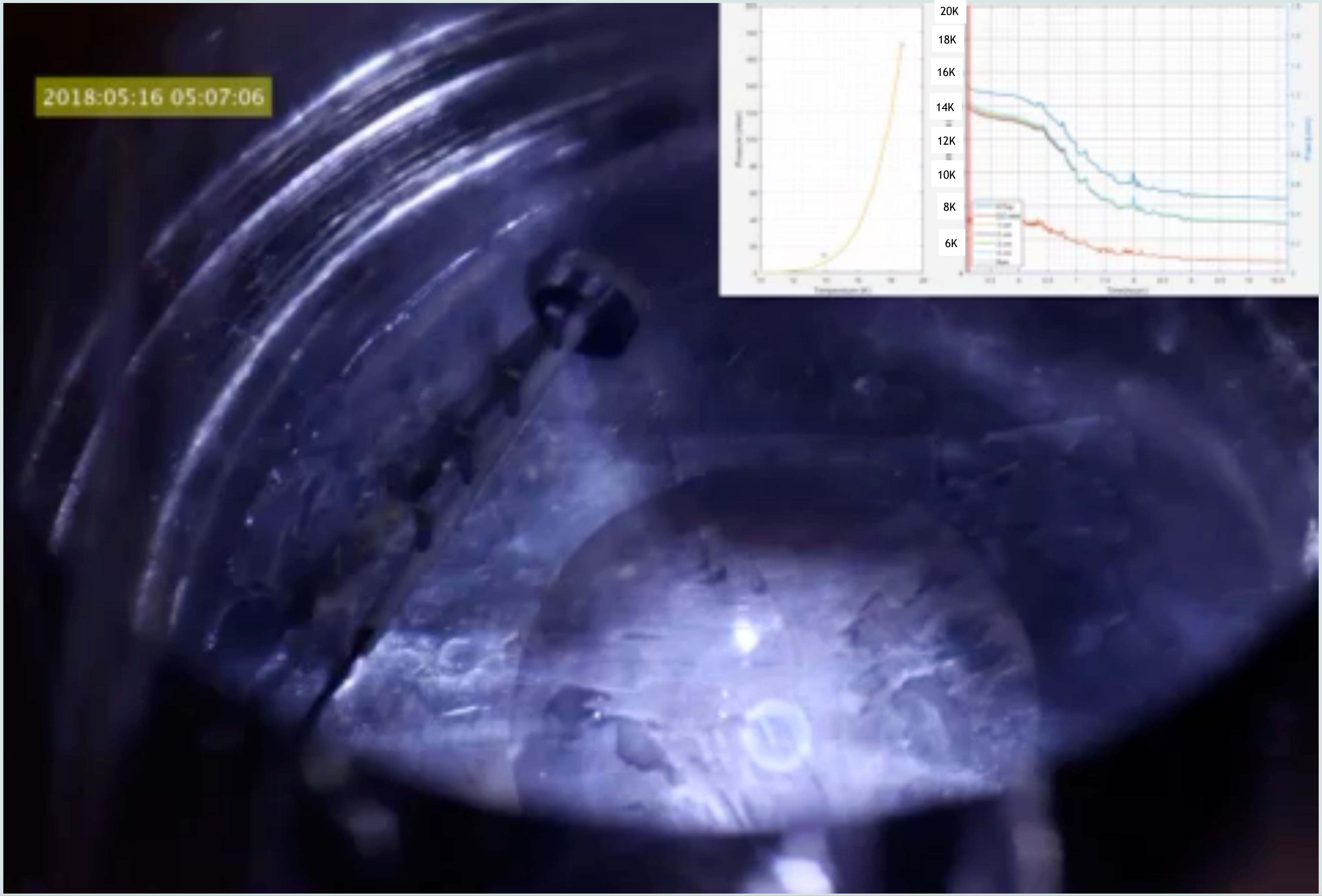
One step condensation

- initial Ts: **8K/ 18K/ 18K;** Ts were controlled by adjusting LHe flow and continuously decreasing flow of D2.



cooling

- final Ts: **7K/ 15K/ 15K**, D2 probes at **14K**, gradient only **1K**



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Surface Frost formation

- We observed Surface frost during:
  - cooling down after condensation

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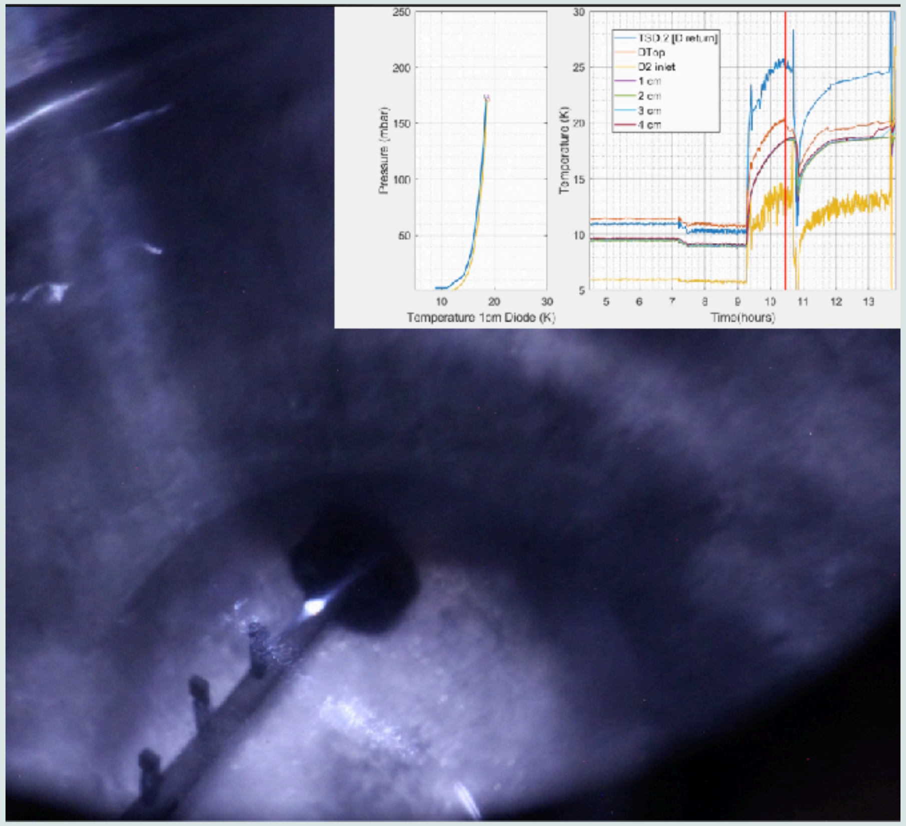
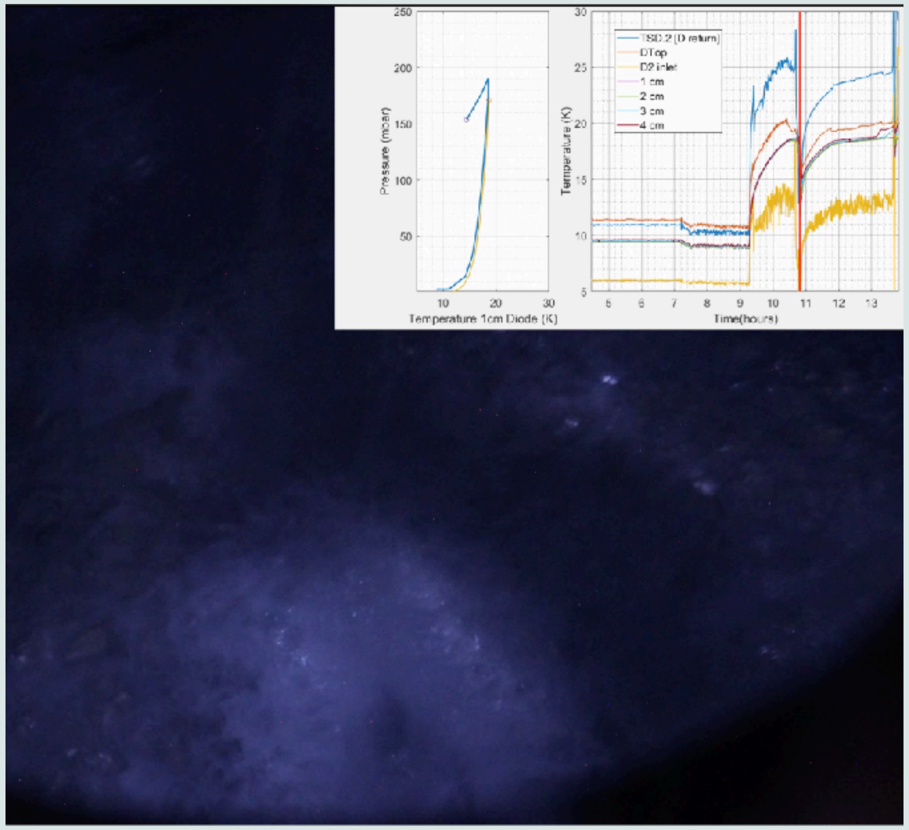
- especially bad is fast cooling from T_{triple}

$T=T_{triple}$

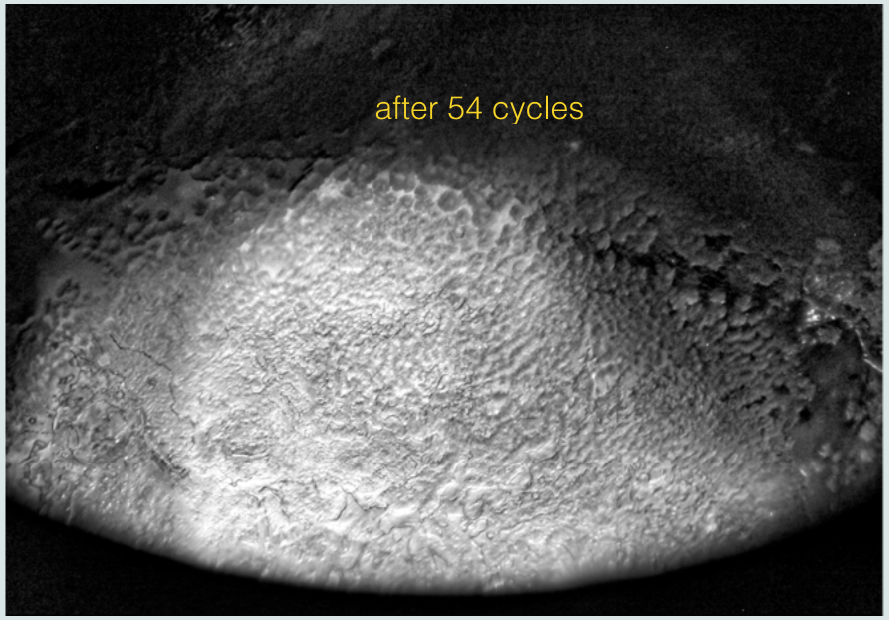




T=12K

Surface Frost formation

- We observed Surface frost due to temperature oscillations:
  - LHe flow accidental oscillations
  - Intentional heat pulsing using heaters, if crystal T rises above 8.4K



T_{base}= 8.5K, T_{max}=9.7K; $T_{top\ max}$=14K

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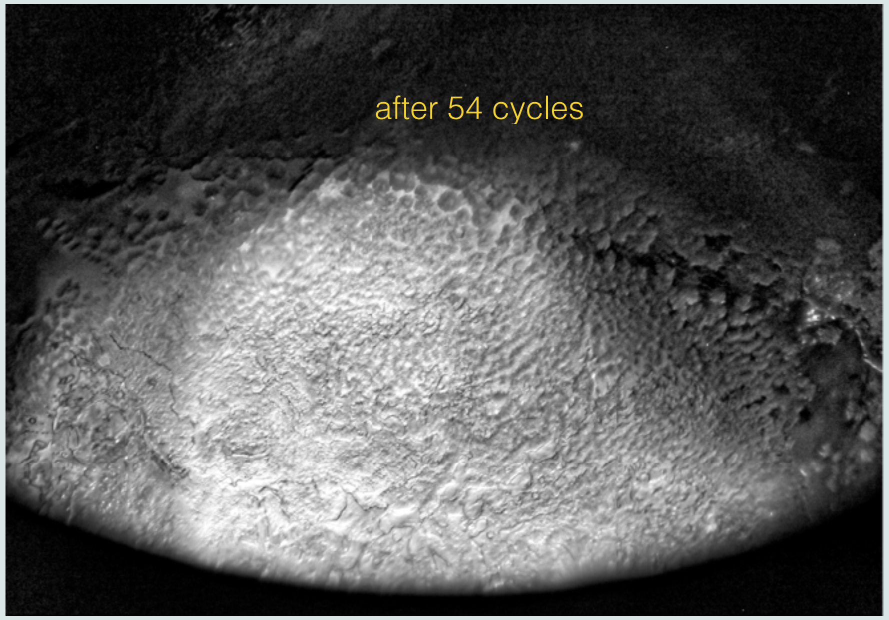
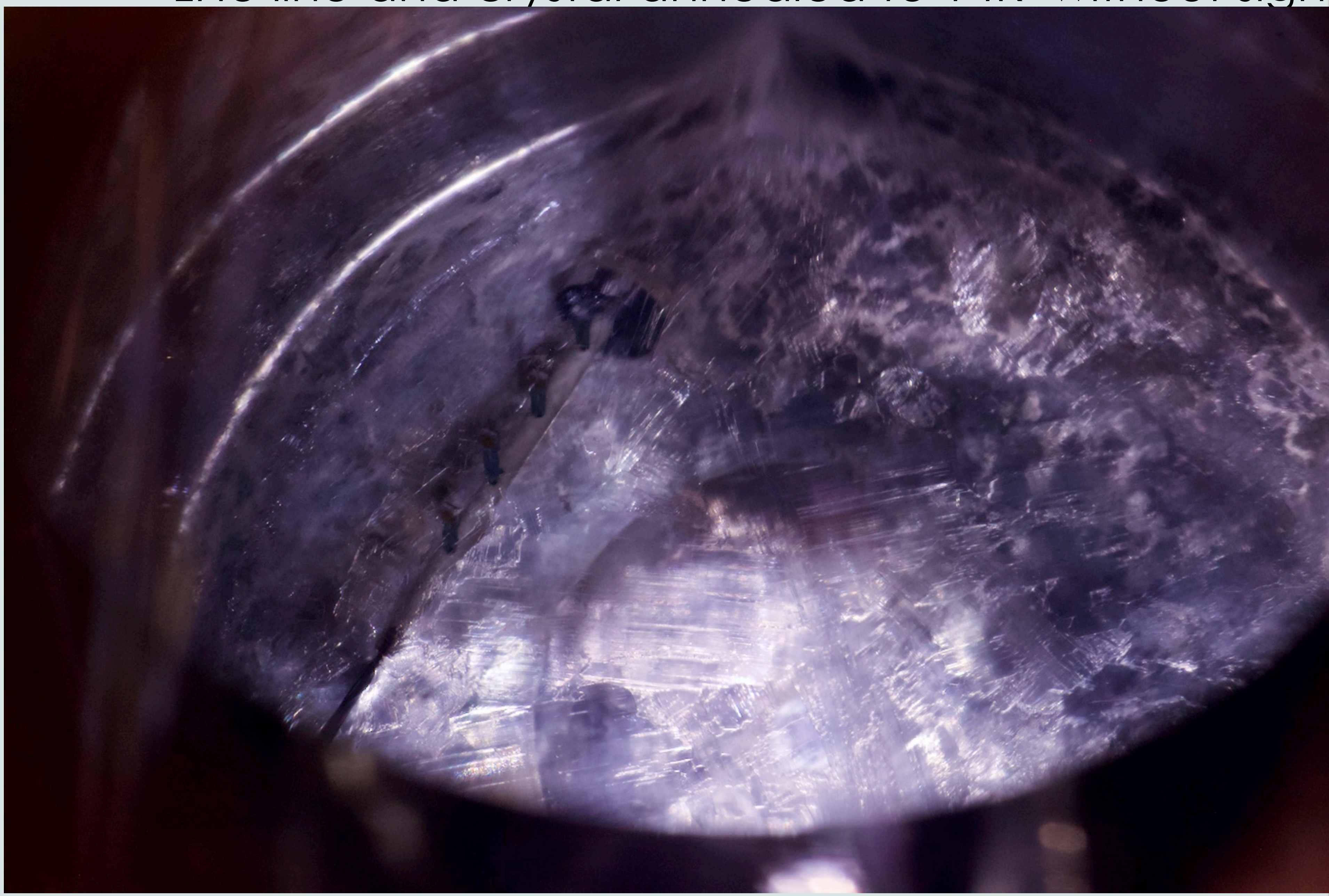


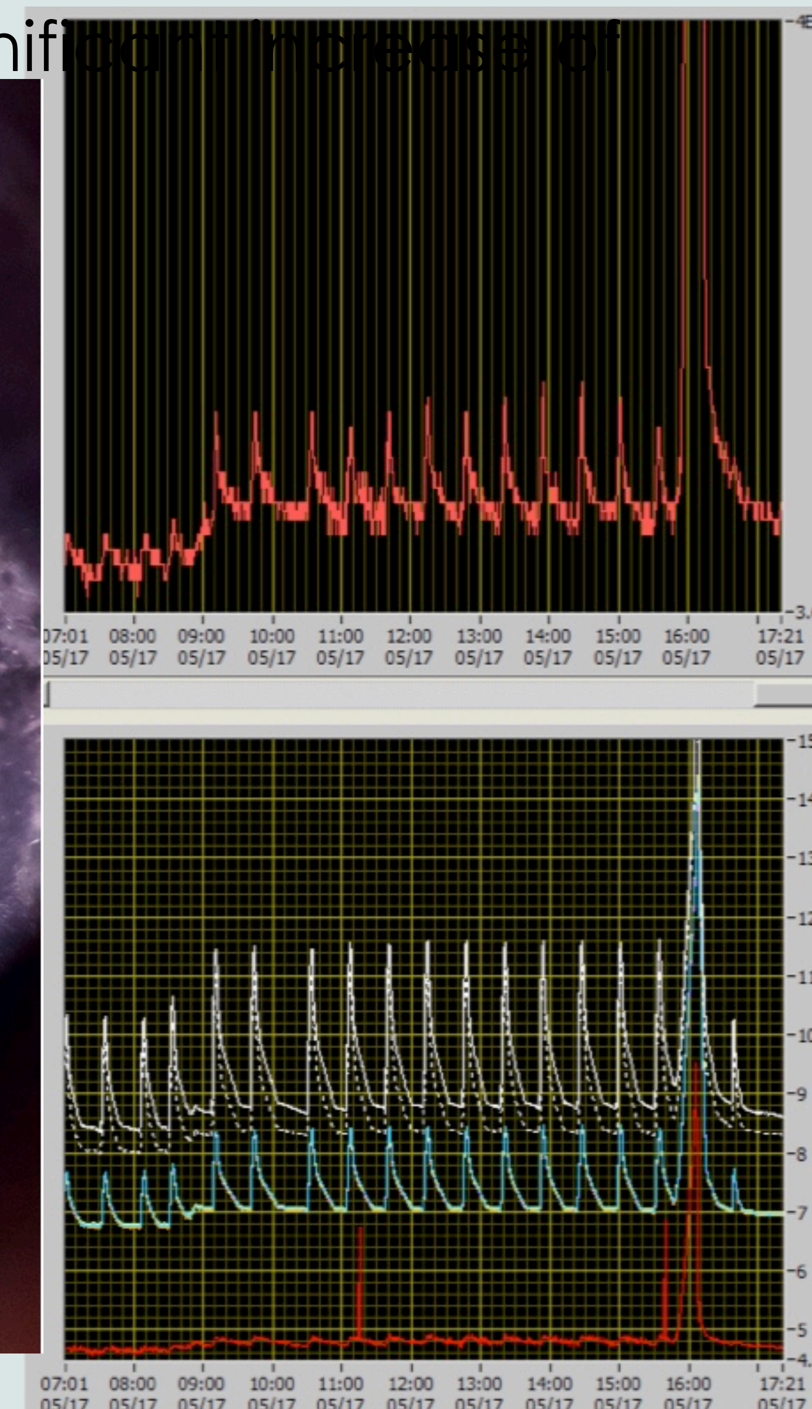
T_{base}= 8.5K, T_{max}=9.7K; $T_{top\ max}$=14K

T_{base}= 7K, T_{max}=8.4K; $T_{top\ max}$=11.5K

Surface Frost formation during cooling down

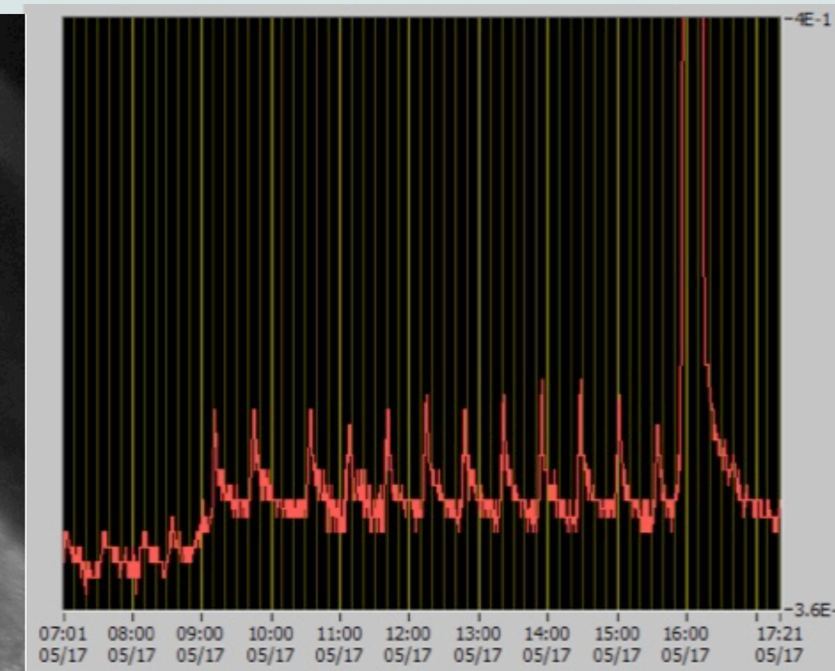
- I set up heat shooting, as result at one point there was a bible in LHe line and crystal annealed to 14K without significant increase of

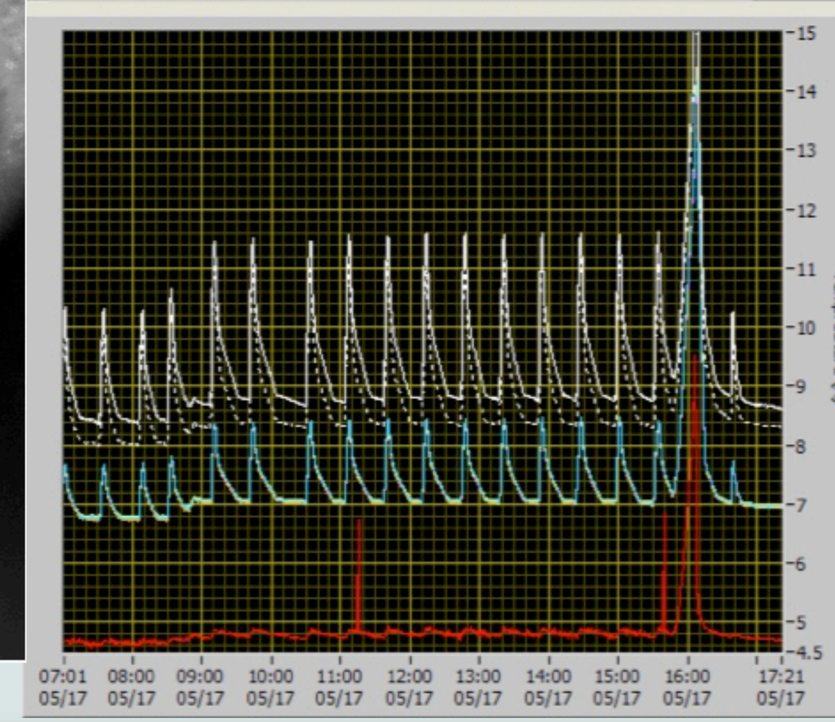




Surface frost can be removed: accidental annealing

- I set up heat shooting, as result at one point there was a bible in LHe line and crystal annealed to 14K without significant increase of pressure





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- Solid Deuterium properties related to UCN production
- Growing small perfect sD2 crystals
- Growing large sD2 crystals in situ for UCN production
  - Overview of experimental setup
  - Study of SD2 growth
    - growing at operational temperature - low T range
    - several steps condensation at high T-range (above 12K)
    - condensation at intermediate T range (below and above 10K)
    - crystal shapes
    - one step condensation
    - freezing from a liquid
  - Study of SD2 surface degradation under heat pulsing
- Conclusions

Freezing from a liquid phase

Conclusions and Recommendations for UCN SD2 source design

- It is possible to grow SD2 crystal of a good quality for UCN production
- Quality and shape of the crystal is defined by temperature of cryostat walls
- Special care must be taken about Surface frost formation
- When designing UCN source think about:
  - modeling thermal performance of the cryostat
  - Positioning temperature sensors to characterize temperature profile of the walls
  - Good cooling power to keep SD2 temperature as close to 5K as possible
  - Design LHe system to avoid flow instability and allow growing crystal above operational T
  - Testing of crystal growth and surface annealing before installations