Supplementary Materials

GitHub repository containing:
1. **Figure 1~5** in vector graphics (.pdf);
2. Python scripts for reading the XPCS results from APS Beamline 8-ID-I and plotting the figures in the manuscript;
3. Python scripts for driving the robotic pendant drop, built on the `urx` and `pyepics` library.

https://github.com/Dozgulbas/aps_droplet

Video S1: Side-by-side comparison of the robotic pendant drop operation in RoboDK simulation (left) and inside the chemistry laboratory adjacent to Beamline 8-ID-I (right): https://drive.google.com/file/d/1YE4AQPBVzHz24X1TUQH7GRGDcQU76MJB/view?usp=share_link

Video S2: Demonstration of the robotic pendant drop inside the chemistry laboratory. https://drive.google.com/file/d/1NuiexF5pUIwH6dr-h3qSj1FtVxNx_U7a/view?usp=share_link

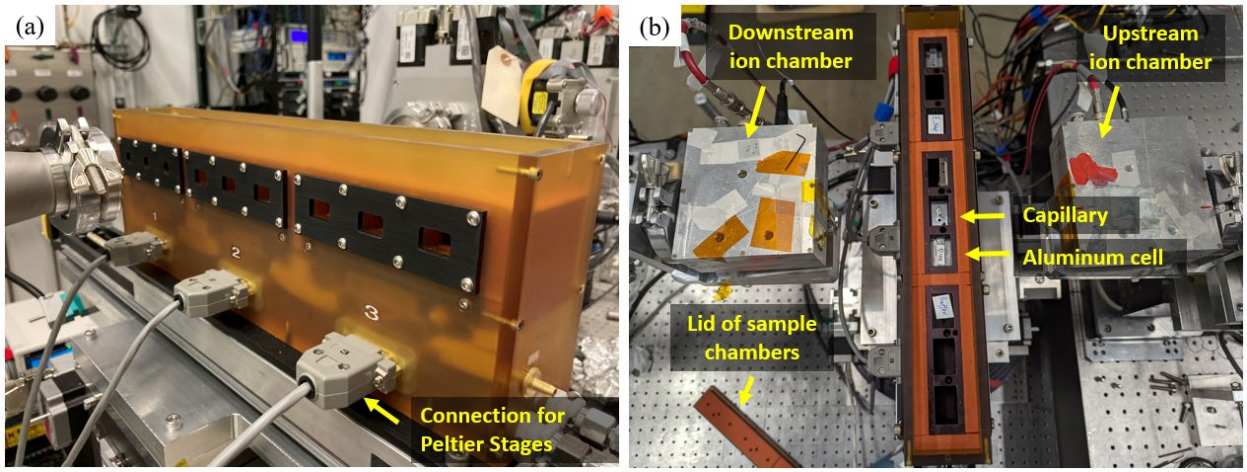

Figure S1 (a) Front view of Quantum Northwest setup (QNW 3×3) at APS Beamline 8-ID-I. The setup holds 9 cells in total. Every three cells share the same temperature environment and the sample temperature is driven by a Peltier stage. **(b)** Top view of the QNW setup. The cell slots are 35.6 mm (height) × 25.4 mm (width) × 15.7 mm (thickness in the x-ray direction). Each anodized aluminum holder (black grid) contains three cell slots and is connected with one Peltier stage. The spacing between the anodized aluminum holders is padded with thermally-insulating materials. Two ion chambers are placed before and after the sample to monitor the x-ray flux and sample transmission coefficient simultaneously during the XPCS measurements.

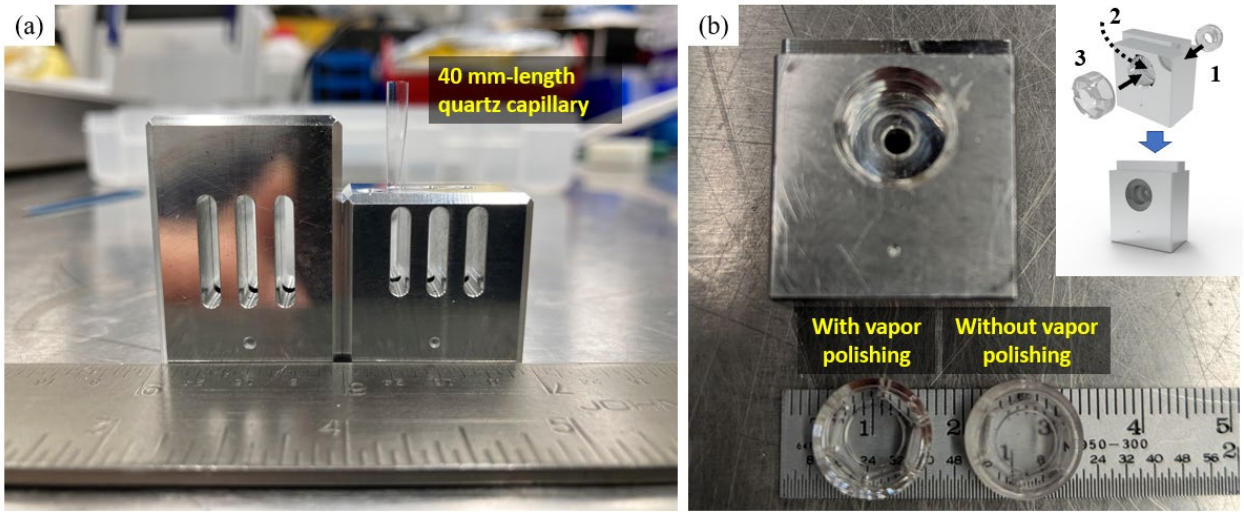

Figure S2: **(a)** The capillary setup. The customized thin-walled quartz capillary from Charles-Supper has a reduced total length of 40 mm including the funnel in order to fit inside the QNW setup. The capillary can slide into the aluminum blocks, and the aluminum blocks can be inserted into cell slots on the QNW holder as shown in **Figure S1b**. The taller aluminum block on the left is designed for 40 mm length capillaries without the loading funnel. The dents on the bottom of both blocks match the bumps inside the QNW holder and help lock the blocks in place during stage translation. **(b)** The Cap Cell setup. The sample reservoir has a diameter of 3 mm and the thickness of the sample along the x-ray beam can vary from 0.5 mm to 4 mm depending on the design of the cell. The threads of the tapped hole on the aluminum holder match the CNC-machined polycarbonate cap. The optical clarity of the polycarbonate cap can be improved via chemical vapor polishing. The Cap Cell assembly is illustrated in the inset figure: 1) Bolting down one cap; 2) Pipetting the sample into the reservoir if the sample is fluidic, or scooping out the sample and fill the reservoir if the sample is viscous or solid; 3) Bolting down the other cap to form a seal. Spacer window material (e.g. Kapton films) can be inserted between the sample and the polycarbonate caps if polycarbonate is not compatible with the sample.

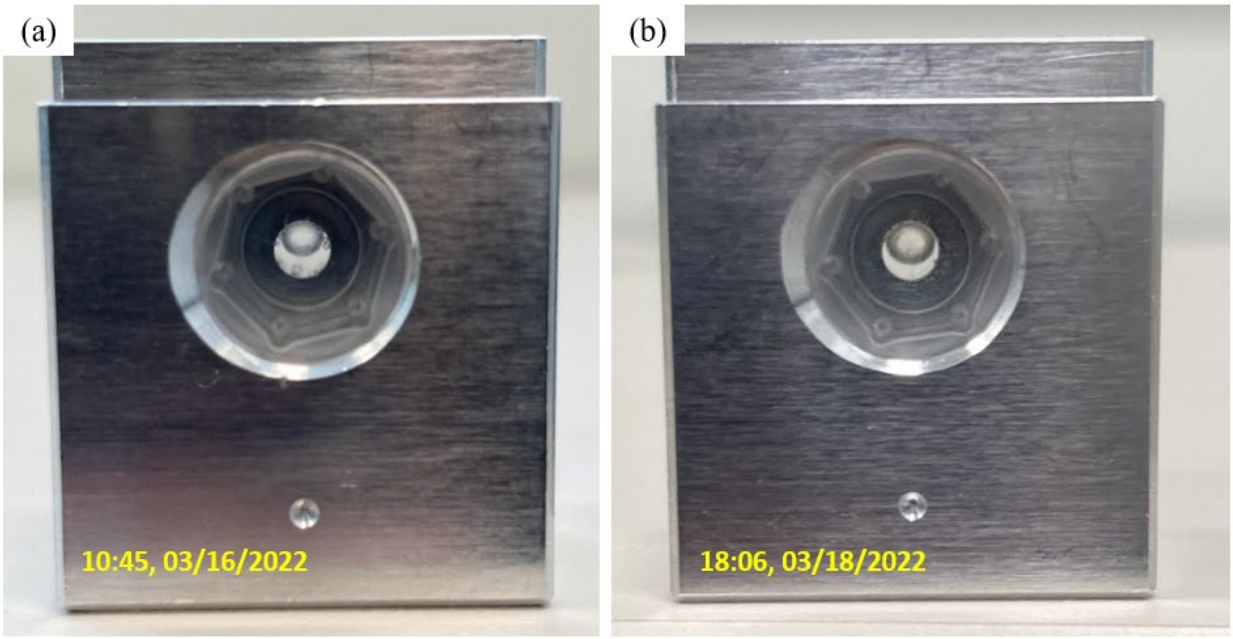

Figure S3: Leak test of the Cap Cell setup. The Cap Cell can hold water up to 40 hours at ambient environment without significant leakage.

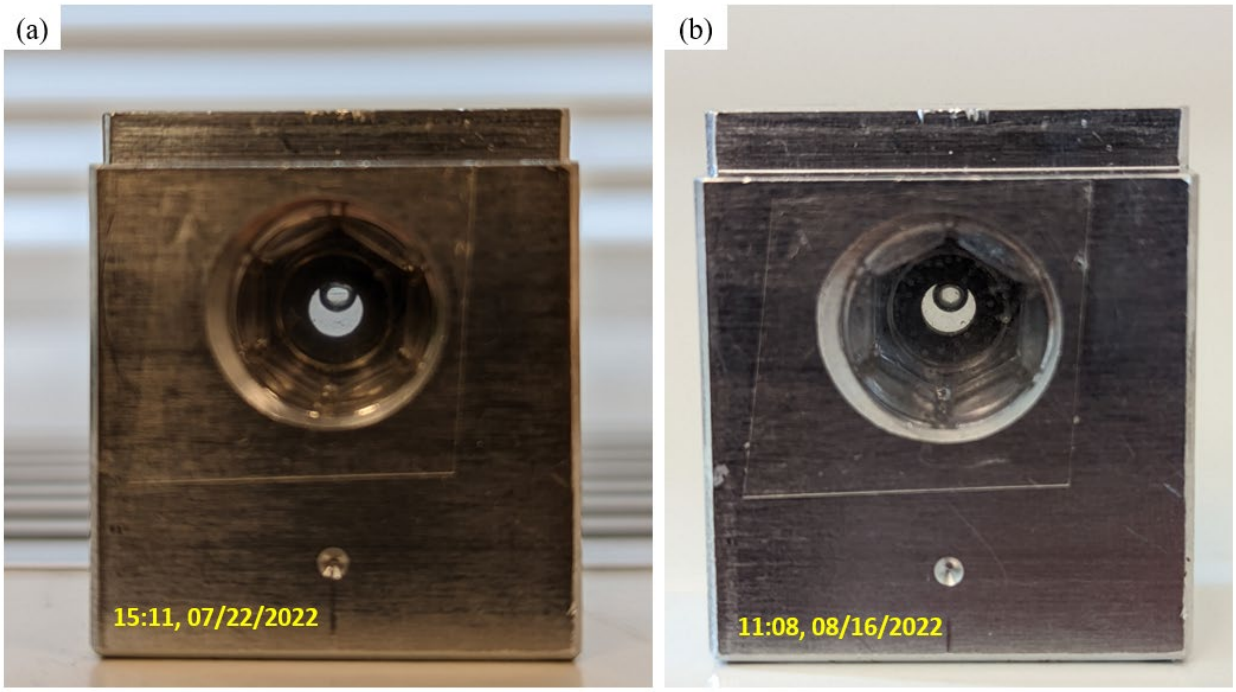

Figure S4: Leak test of the reinfornced Cap Cell setup. The Cap Cell can hold water at ambient environment for as long as 4 weeks if the seal is reinforced with adhesives, e.g., VIEWSeal pressure-activated silicone/polyolefin sealing film from Greiner.