Paineira Beamline

PAINEIRA Mail-in User Manual – PXRD (External Users)

Kapton Capillary - SPINNER (with High-throughput Automation)

Version: 1.3

Date: August 27, 2026

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Campinas, 2026

Sumário

1. Overview & Scope

Mail-in mode enables continuous submission and fast access to high-quality synchrotron-radiation powder X-ray diffraction (SR-PXRD) measurements using automated sample handling. In this initial phase, only users from Brazil and Latin American countries (including the Caribbean) are eligible. Only non-hazardous, inert samples (*e.g.*, minerals, oxides, hydroxides, metals) are accepted due to common courier transport. Measurements are performed automatically, preferentially at night/weekends.

Data will be made available in the ICAT system after measurement. The beamline team evaluates proposals; after acceptance, sample holders are shipped to the user (**Figure 1**), who prepares and registers samples in ICAT, then sends them back to CNPEM-LNLS. *A dedicated manual for the ICAT sample registration will be made available for users*.

2. End-to-End Workflow

• Proposal & eligibility check (region + non-hazardous materials).

• Kit shipment: Kapton capillaries (one diameter per proposal), magnetic pins, pin caps, IDs, support for mounted pins with lids, and instructions.

• Sample preparation in capillaries (Section 4) and ICAT registration.

• Packaging & tracked shipment back to CNPEM-LNLS.

• Intake & triage (damaged or non-compliant samples will not be measured).

• Automated measurement with PiMEGA 450D (fast) and/or MAC (high-resolution), including temperature control if scheduled.

• Data delivery *via* ICAT; instrument file for GSAS-II and NIST LaB_6 pattern sent by email (for high resolution measurements with MAC).

• Retention & disposal: immediate disposal after measurement unless retrieval is previously requested and samples are claimed within 30 days.

3. Paineira Beamline - Experimental Setup and Key Parameters

3.1 X-ray Source and Energy Range

• Energy range: 12 – 30 keV.

• Source: undulator-based insertion device with double crystal monochromator (DCM).

• Beam energy/wavelength is selected by the beamline team to optimize total transmission (~3–30%), mitigate fluorescence, and match operational conditions at measurement time. Users do not need to define the beam energy.

3.2 Experimental Geometry and Sample Position

• Main geometry: Debye–Scherrer (transmission) on magnetic PIN holders with sample rotation (50 Hz).

• Beam intercepts the capillary at 4.0 ± 0.1 mm above the PIN face; ensure homogeneous packing in this zone (**Figure 1**, left).

3.3 Beam Size at the Sample

• Millimetric beam at the sample position, defined by slits and optimized for capillary transmission: 0.4 mm vertical x 2.0 mm horizontal, when PIMEGA detector is used and the diameter of the capillary in the vertical director and 3.0 mm in horizontal direction, when MAC detector is used.

3.4 Detectors and Measurement Modes

• MAC (Multi Analyzer Crystal): ~0.005° FWHM at ~16 keV; typical ~4 h/sample; high-resolution studies.

• PiMEGA 450D (fast 2D): ~0.05° FWHM; typical ~5 min/sample; high-throughput screening.

3.5 Temperature Control

• Cryojet operation ~90–470 K; temperature steps increase total acquisition time and reduce sample counts. Each proposal must fit within ~24 h (three shifts of 8h).

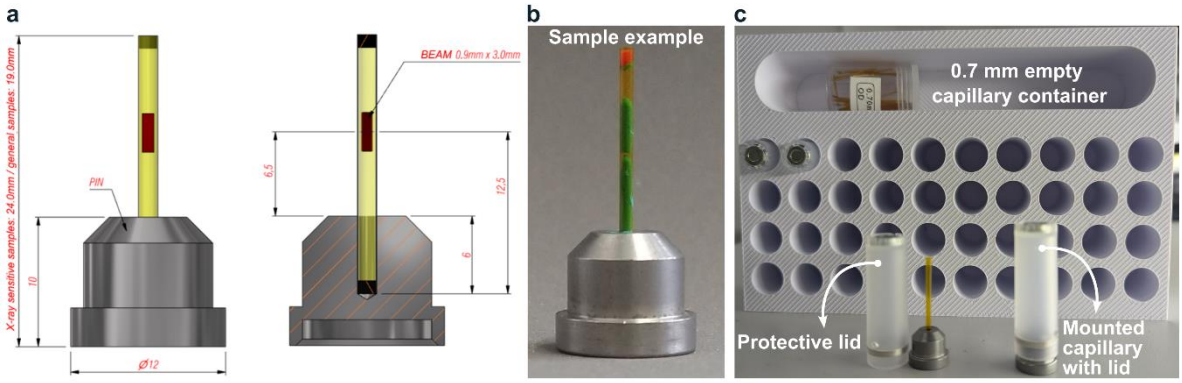

Figure 1 – **a)** Beam incidence position: 4.0 ± 0.1mm above the PIN face (keep sample homogeneous in this zone). **b)** Real image of the mounted capillary and the supporting pin with QR code at the bottom (DataMatrix) for registration. **c)** Sample kit mailed to the user comprised of a support for the mounted samples, magnetic pins with DataMatrix codes at the bottom, capillaries with specific diameter, and lids to protect mounted samples.

4. Sample Preparation in Kapton Capillaries (SPINNER)

4.1 Safety & Bench Setup

Wear gloves. Cover the bench (aluminum foil recommended). Clean tools with isopropyl alcohol before starting. Prepare: capillary, sample, epoxy (Durepoxi), caliper (or ruler), mortar, and spatula.

4.2 Filling & Compaction

• Place a small portion of sample into a clean mortar (if dilution is recommended, triturate with amorphous carbon or silica until homogeneous).

• Hold the capillary and dip the open end into the powder, tapping gently to get the sample inside.

• Tap the other end of the capillary gently against the bench to compact; repeat until completely filled. You may use the pin to mount and tap the capillary on the benchtop.

• Compact by tapping for at least 1 minute; add more powder if needed. Avoid voids to prevent the beam from crossing air gaps.

4.3 Sealing with Epoxy - minimal and away from the beam window

• Mix equal parts of epoxy to a homogeneous mass; make a small sphere and flatten into a disk.

• Gently touch and rotate the capillary end to seal, then withdraw while rotating; ensure no overflow on the outside.

• Do not bend or crease the capillary; misalignment affects 50 Hz rotation.

• After curing, remove any external excess. The beam window must be entirely clean (no epoxy residue). The epoxy resin shows diffractions in the diffractogram; make sure to clean any epoxy residue from the capillary surface. See **Figure 2** for reference.

4.4 Special case: 0.5 mm inside 0.7 mm

If 0.5 mm capillaries are required, insert the filled 0.5 mm tube into a 0.7 mm Kapton tube and seal the top with epoxy to improve mechanical stability. You may trim the 0.5 mm top to fit before sealing (see **Figure 2**).

Figure 2 – Correct sample preparation and sealing (no epoxy overflow; beam window must be clean). Rightmost figure: special case of samples mounted in 0.5 mm capillary within a 0.7 mm capillary.

4.5 Insert into the PIN & Register

• Wait for full epoxy cure on the end that contacts the PIN.

• Clean the PIN base (DataMatrix) with alcohol before use. **NEVER** use acetone; it can erase the DataMatrix.

• Insert the capillary into the slot based on the capillary diameter; keep the assembly straight/coaxial.

• Register the sample-to-PIN mapping (DataMatrix) in the sheet/ICAT (see ICAT manual). The DataMatrix can be visualized with the help of a smartphone camera, in case it is too small to read (after “HM…”, the next four characters are numbers, **Figure 3**).

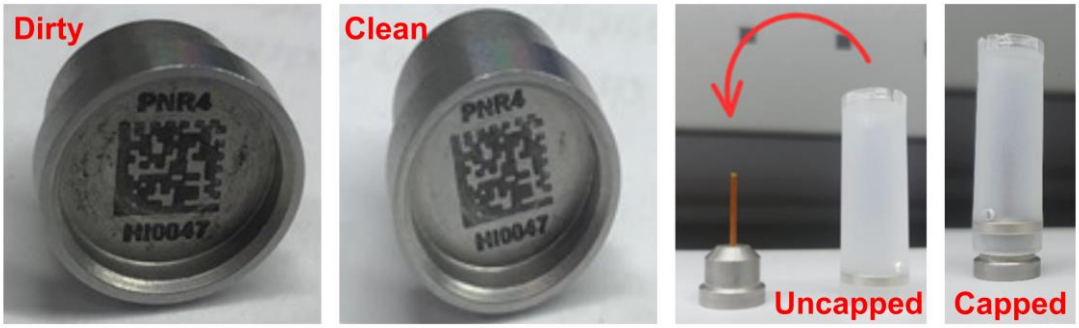

Figure 3 – Clean the PIN DataMatrix with isopropyl alcohol. **DO NOT** use acetone. Place the protective cap for transportation.

• Make sure you register the sample with the correct pin code on the printed table sent in the mail-in. For instance, Al_2O_3 powder → PNR4HM0168, so that samples are traceable and labels are not misplaced.

• Place the protective cap on each PIN for transport.

5. Packaging & Shipment

• Immobilize pins/capillaries in the plastic support grid provided, ensuring the samples will not move during transportation (**Figure 4**).

• Use an inner cushioning to accommodate the sample holder, if necessary; mark as FRAGILE and keep it dry.

• Include the printed list mapping the PIN IDs to samples (*e.g.*, Al_2O_3 powder → PNR4HM0168).

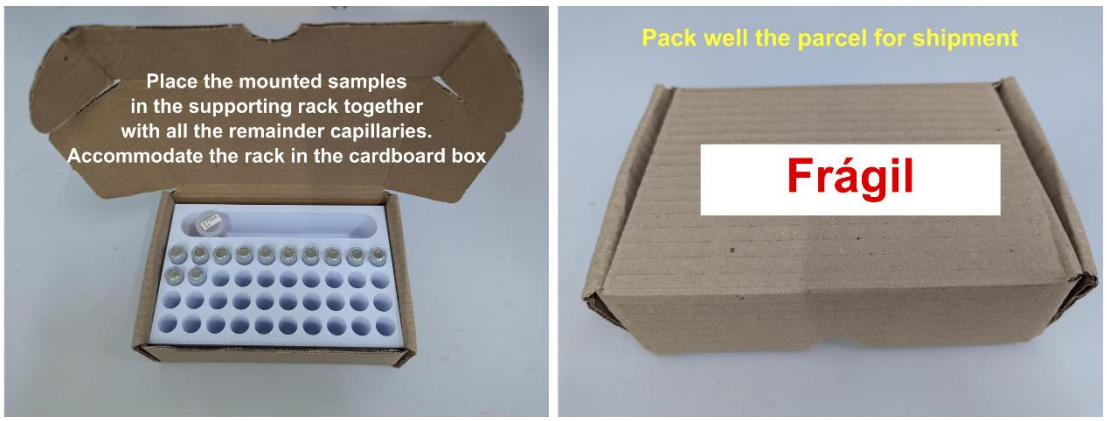

Figure 4 - Example showing how to prepare a parcel to ship samples back to Sirius after preparation and registration.

6. Acceptance / Rejection Criteria

Samples will be measured when: capillary straight and intact; no epoxy on the beam window; powder uniform and compacted; PIN ID legible; metadata complete.

Sample will NOT be measured when: cracked/creased capillary; epoxy overflow preventing seating or contaminating the beam window; loose powder/voids; missing identification. Damaged samples will not be measured.

7. Data Delivery & Access

PXRD data will be available in ICAT after measurement. The instrumental parameter file for GSAS-II and the LaB_6 NIST pattern for high resolution measurements will be sent by email along with the user data.

8. Data Access and Management (ICAT and Ibirá)

All Mail-in PXRD data are managed through Sirius data systems. ICAT manages proposals, samples and metadata; Ibirá stores raw and processed datasets. Access to Ibirá is linked to the user's ICAT account and proposal.

8.1 ICAT - Experiment and Metadata Management

- Register sample names for each PIN DataMatrix code (Mail-in kit).
- Track parcel status and shipment; view links to datasets and metadata.
- Download experimental metadata after measurements (automatically ingested).

8.2 Ibirá - Data Repository

- Download raw diffraction files and processed datasets.
- Retrieve auxiliary files (instrumental parameter files, NIST LaB_6, logs).
- Access is granted based on ICAT credentials and proposal association.

8.3 Data Formats

- HDF5
- ASCII (.dat)
- GSAS-II (.fxye / .xye)

9. Retention & Disposal Policy

Capillaries are disposed of immediately after measurements. If retrieval is desired, the user must inform the Paineira Team within 30 days after notification. LNLS-CNPEM is not responsible for sending back measured capillaries; users must arrange reverse logistics.

10. Safety & Compliance

Only inert samples are accepted in Mail-in mode due to courier transport constraints. For non-standard conditions (*e.g.*, reactive samples, *in-situ*/*operando* beyond Mail-in capabilities), submit a standard proposal during the open call and coordinate with the beamline team.

11. Quick Checklists

Before sealing

- Capillary fully filled and compacted (≥ 1 min tapping).
- No voids in the beam window zone (~6.5 ± 0.3 mm above PIN face).
- Minimal epoxy, away from beam window; no external overflow.

Before inserting into the PIN

- Epoxy fully cured on the PIN-contact end.
- PIN base cleaned with alcohol (**never acetone**).
- Insert capillary straight/coaxial to the pin.
- Register PIN DataMatrix → sample mapping in ICAT.
- Add protective cap to the PIN.

Packaging

- Immobilize pins in the supporting rack and stabilize it in the shipping package.
- Add FRAGILE labels.
- Add printed list of PIN IDs linked to sample names.

Before shipping your samples

- All PINs have protective caps installed
- PIN DataMatrix codes are clean, legible, and correctly registered in ICAT
- Sample names in ICAT match exactly the samples mounted in each PIN
- Only non-hazardous materials are included (Mail-in safety requirement)
- Samples are homogeneous in the beam incidence region (6.5 ± 0.3 mm above the PIN face)
- Printed PIN-sample list is included inside the shipping box

After measurement - Data access checklist

- Receive the notification that measurements are completed
- Log in to ICAT and see your Mail-in proposal and samples

- Dataset links are available within your proposal in ICAT
- Access and download raw and/or processed data from Ibirá
- Receive (or download) the GSAS-II instrumental parameter file and LaB_6 NIST reference data
- Confirm the data format required for your analysis (HDF5, .dat, .xye/.fxye)
- Download all files before the retention period expires.