Sample assemble manual

Mail-In

SPINNER: Kapton capillary

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Sumário

Important Highlights

1. Experiment Category
   In this manual, you will find all the information and instructions to prepare samples inside Kapton capillaries. This type of capillary is used in experiments with the SPINNER sample holder on the Paineira beamline.

2. Beam Incidence Position
   As shown in Figure 1, by default, the beam strikes at 6.5 (±0.3) mm above the PIN face, so ensure that your sample is homogeneous, especially in this region. For X-ray sensitive samples, the interval can be determined after asking the local contact.

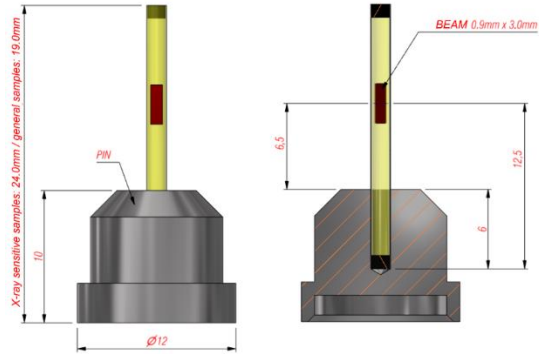

Figure 1 - Main dimensions and beam incidence position

Initial Preparations

1. Material List

   - ➢ Capillary of the desired diameter
   - ➢ Sample
   - ➢ Epoxy resin: Durepoxi
   - ➢ Pachymeter
   - ➢ Mortar
   - ➢ Spatula with a small spoon

Figure 2 – Materials needed for sample preparation.

2. IMPORTANT Precautions

Before starting to prepare the capillary, it is worth remembering some basic precautions that should be taken:

➢ The use of gloves is MANDATORY.

➢ To reduce the chance of contamination and facilitate cleaning, envelop the bench (by standard we encourage the use of aluminum foil);

➢ Select all the tools to be used and clean them with isopropanol.

Preparing the Capillary

1. Filling the Capillary with the Sample
    a. Deposit a small portion of the sample in the mortar sterilized with alcohol (Figure 3); **NOTE: If the beamline staff recommended dilution, add the diluent together with the sample to the mortar and triturate until the mixture becomes homogeneous.**

Figure 2 - Mortar with MgO sample

    b. Hold the capillary with your index finger and thumb or with tweezers;
    c. Dip the open side of the capillary into the sample and "fish" the sample;
    d. Gently tap the capillary against the table to make the sample settle and compact;
    e. Repeat steps c and d until the capillary is completely filled;

f. Gently tap the capillary against the table for at least 1 full minute. This will compact the sample;
g. If necessary, add more sample powder and repeat step (f);
h. Figure 4 shows an example of a filled capillary.

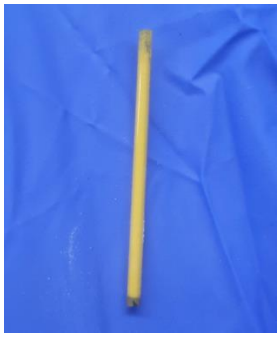

Figure 4 - Capillary filled with sample

Note: It is very important to completely fill the capillary so the beam does not pass through air gaps or areas without enough sample.

2. Final steps

Seal the other capillary extremity following the same procedure mentioned earlier, as follows:

Preparing the Durepoxi: Separate equal amounts of each component from the box and mix them to obtain a homogeneous modeling clay.

Before starting the sealing, pay attention to the following points:

- Do not bend the capillary to prevent folding or creasing, which may cause slight misalignment and affect rotation.
- Do not let DUREPOXI excess on the outside of the capillary; this excess mass can prevent the capillary from fitting into the PIN.

I. Make a small DUREPOXI ball;
II. Flatten the ball to form a disc;
III. Gently press the capillary extremity into the mixture, rotating delicately;
IV. Pull it back while rotating gently;
V. Ensure that DUREPOXI is not overflowing outside of the capillary, as shown in Figure 5.

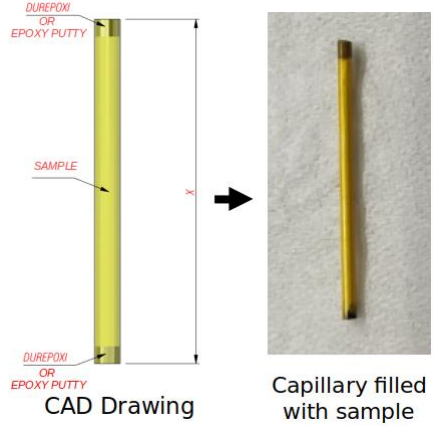

Figure 5 - Finished capillary

3. **NOTE: ONLY FOLLOW THIS STEP IF YOU ARE USING 0.5 MM CAPILLARIES.**

If your sample requires the 0.5 mm capillaries, once you have completed the preparations of the capillary, as already explained, you should insert it inside one of the 0.7 mm capillaries also sent inside your Mail-in kit. Seal the other end with DUREPOXI to make the assembly tight. This step is important to give mechanical stability to the capillary inside the PIN. The figure below illustrates how the final assembly should be.

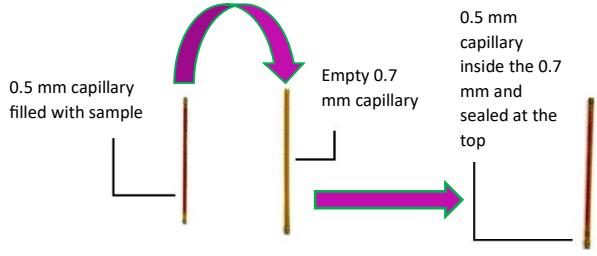

Figure 6 – Correct assembly for the 0.5 mm capillary. It's necessary to insert it inside a 0.7 mm one.

It may be necessary to trim the top of the 0.5 mm capillary so that it fits inside the 0.7 mm one. Just remember to always seal the top with DUREPOXI.

Inserting the Capillary into the PIN

Important: Ensure the end of the capillary in contact with the PIN has fully cured Durepoxi.

First, make sure to clean the bottom of the PIN, where the DataMatrix code is located, with a cotton swab and some cleaning alcohol. This will facilitate the reading of your PIN, and therefore your sample. **NOTE: DO NOT USE ACETONE UNDER ANY CIRCUMSTANCES. Acetone can erase the DataMatrix, rendering the PIN unusable.**

a) b)

Figure 6 - PIN before (a) and after (b) cleanup.

Carefully insert the capillary into the corresponding PIN slot based on the capillary diameter, Figure 7.

Figure 7 - Capillary in the PIN

Register your samples

Remember to register every sample to the code associated with the **back** of the PIN that you put your sample, for example, if you have the sample of Al_2O_3 and you put this sample inside the PIN that have the code PNR4HM0168 as show in Figure 8:

Figure 8 – Code of the PIN

Then it is necessary to register in the paper sheet table that is sent together with the mail-in box, that the sample Al_2O_3 is inside the PIN with the code PNR4HM0168, this is important to us to allocate the correct experiment to the correct sample.

- It's possible to read the DataMatrix code with the camera of your cell phone and get the correct code.
- After the letters "HM...." the next four characters are ONLY NUMBERS.

Protect your samples

To protect your sample is necessary to cover the pin with the cap that came together with your kit:

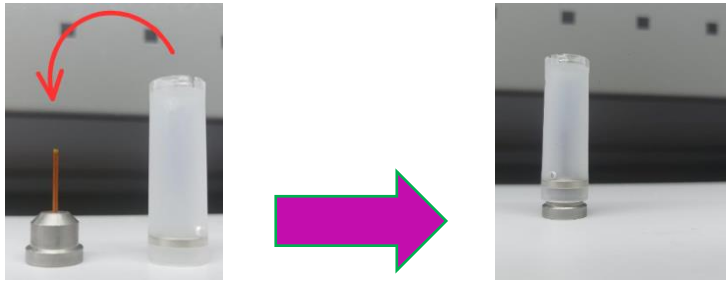

Figure 3 – Putting the cap on the PIN with the Capillary and ready to deploy

Now you are ready to prepare all the samples that you need in your experiment and then put them inside the box that you receive and send us back.