



Sample assemble manual

SPINNER: Kapton capillary





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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. Capillary Length

Some samples can be damaged by X-rays; if this is applied to your sample, discuss it with the local contact and make this information clear. The SPINNER is designed to change the incidence position of the beam on the capillary during the experiment. Therefore, radiation-sensitive samples need to be prepared with an appropriate length. See the

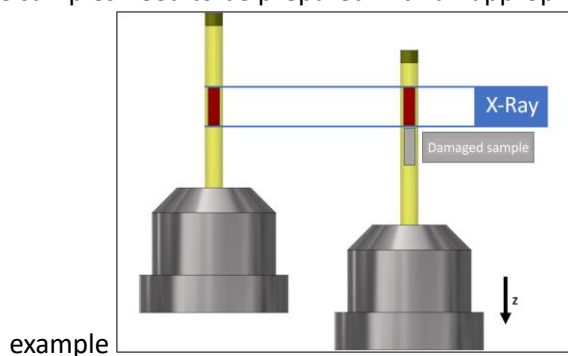


Figure 1.

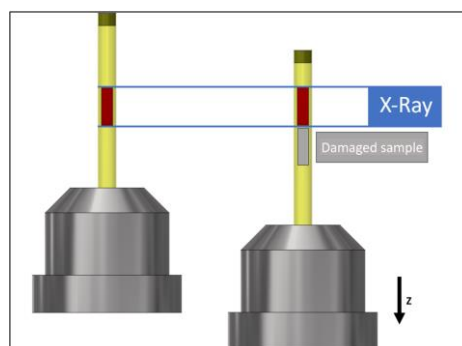


Figure 1 – Change in the beam incidence position

3. Beam Incidence Position

As shown in Figure 2, 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.



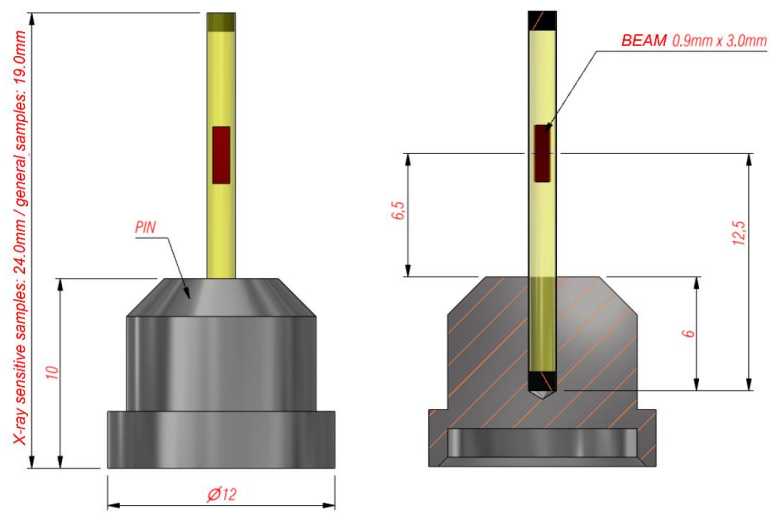


Figure 2 - Main dimensions and beam incidence position



Initial Preparations

1. Material List

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



Figure 3 - 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. Cutting the Kapton Capillary

Two lengths have been defined for the capillaries, one for general samples and the other for samples sensitive to X-ray.

- General samples: Cut capillaries to 15.0 mm in length
- X-ray sensitive samples: Cut capillaries to 20.0 mm in length

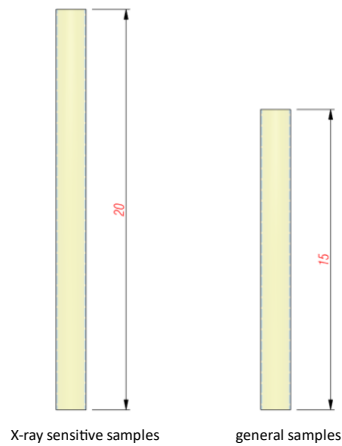


Figure 4 - Capillary length related to sample nature

- Adjust the pachymeter to the determined length and then use the marker pen to note the cutting position on the capillary, as shown in Figure 5.



Figure 5 - Marked capillary

- After marking the capillary, cut it with the scissors.



Figure 6 – Capillary in the final length



2. Sealing one capillary extremity

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.

Recommended procedure:

- Make a small DUREPOXI ball;
- Flatten the ball to form a disc;
- Gently press the capillary extremity into the mixture, rotating delicately;
- Pull it back while rotating gently;
- Ensure that DUREPOXI is not overflowing outside of the capillary, as shown in Figure 7.



Figure 7 - Capillary with one extremity sealed

Note: Do not place the capillary with wet DUREPOXI in the PIN. Wait for the complete cure.

3. Filling the Capillary with the Sample

- Deposit a small portion of the sample in the mortar sterilized with alcohol (Figure 8);



Figure 8 - Mortar with MgO sample

- 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 9 shows an example of a filled capillary.



Figure 9 - 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.

4. 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.

- 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 10.

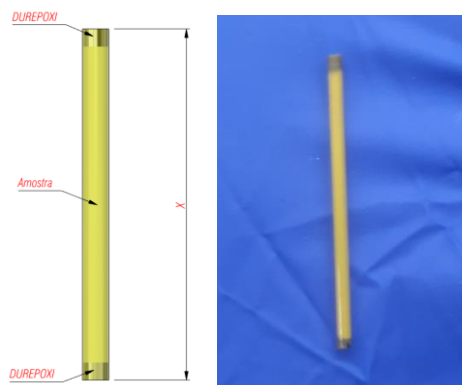


Figure 10 - Finished capillary



Inserting the Capillary into the PIN

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

Carefully insert the capillary into the corresponding PIN slot based on the capillary diameter, Figure 11.



Figure 11 - Capillary in the PIN and ready to start the experiment

