

Our high-throughput measurements are possible through a unique system that can cycle samples through the X-ray beam in seconds per sample. In order to accomplish this, the sample must be loaded into a specially designed sample holder.

In this guide, we'll show you how to load your samples into the holder for best quality measurements! You can also watch our accompanying video.

## Contents of the Sample Preparation Kit

In addition to the sample holders, the kit contains several additional items to help you. Descriptions of all pieces included are listed below:



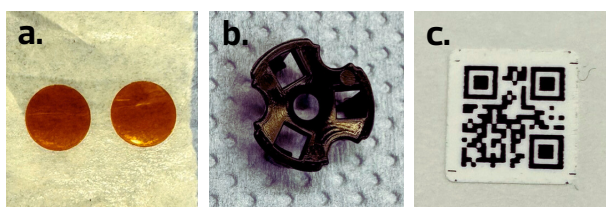
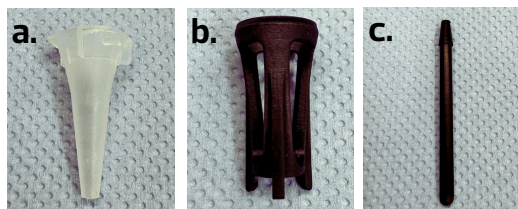
### Sample Holder

contains separate slots for up to 16 samples; each slot consist of a cylindrical window approximately 1.1 mm thick and 2.53 mm in diameter

### Powder Loading Tools

several tools are included to aid in loading the powders:

- funnel to direct powders into the sample window
- a frame to hold the funnel in place
- a plunger to pack the powder



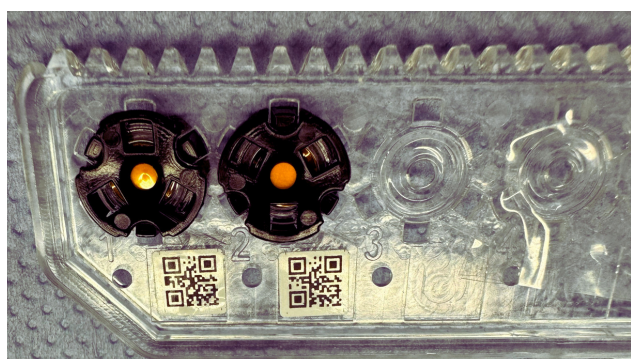
### Sample Windows and ID Sticker

several more pieces for sample containment and identification

- polyimide windows contain the sample
- clips to hold the windows tightly in place
- a sticker with QR code for sample identification

With these item, you will be able to completely fill and ID your samples to send to us for measurements!

## How It Works



### Two finished samples, let's do the next!

Positions 1 and 2 show two finished samples completed with QR codes. It is important that the QR codes are placed in the square positions and on the side shown in the picture (with the slanted notch on the left). The holes to the left

Please ensure a clean working surface! You'll also notice that at position 4 we have covered all sample slots not currently being filled with tape. This helps to avoid cross-contamination while filling the powder, in this case everything except slot 3 is covered.

**NOTE:** the plastic sample holders are somewhat brittle. Avoid applying excess or uneven pressure, torsion, or bending to the holder, as these could result in cracking. We cannot send cracked or broken sample holders

through the system since they can break and jam the sample changer.

### Step 1:

Cover the back side of an empty slot with a fresh polyimide window.



Ensure that the window is centered over the opening for best surface contact.

### Step 2:

Fix the window into place using one of the black clips.



The three prongs of the clip should snap into three of the six openings around the central window. Ensure that you feel a secure snap from each prong latching onto the opposite surface so that the window is held tightly. In rare cases, one or more of the prongs may be defective and not hold tightly – in that case, please toss the clip and use a new one.

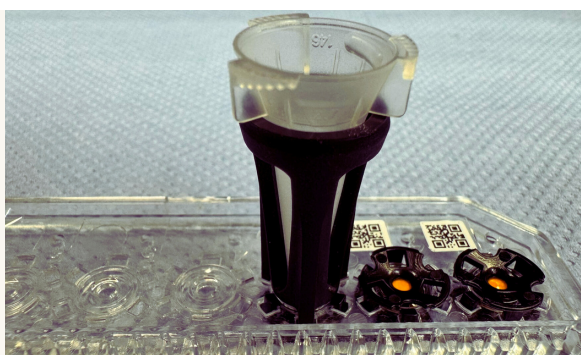
### Step 3:

Flip the sample holder to the other side, and ensure that the black prongs are gripped onto the edges.



### Step 4:

Place the three prongs of the black funnel holder into the three remaining open slots around the window, and insert the funnel.



### Step 5:



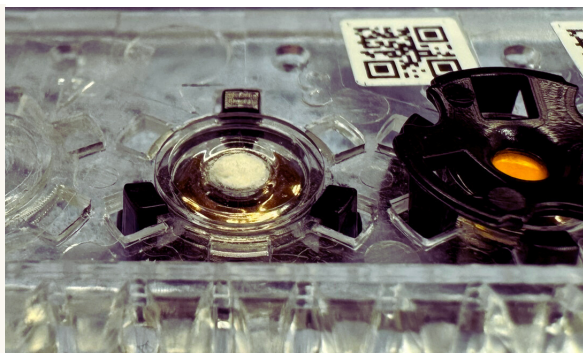
For best quality measurements, the powder should fill the window completely and densely. This ensures both that the maximum sample cross section is available for best possible data statistics, and maximizes the orientation distribution of the crystallites for acquisition of complete Debye–Scherrer rings.



We additionally scan the beam across the window to further improve the sampled volume, but this is not always sufficient if there is not enough sample present or if the sample contains very large crystallites.

### Step 6:

Ensure that the powder completely and densely fills the volume of the window.



That being said, avoid over-filling the slot. The polyimide windows should still lay flat against the powder and holder. Otherwise, the contribution of the background intensities will be slightly modified.

### Step 7:

Cover filled powder with another fresh polyimide window.



### Step 8:

Place another clip over the second polyimide window, clipping the three prongs into the remaining open holes as before.



### Step 9:

Place the QR code sticker in the square frame below the sample.



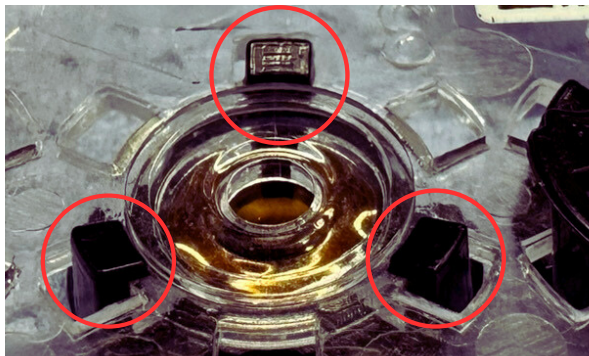
Don't forget to carefully record the assignment of QR codes to specific samples. You can easily double check the string associated with the QR code using any common QR reader, such as with most general phone camera apps, or e.g. Google Lens.

## What about reference and background measurements?

You don't need to worry about reference or background measurements. This is already part of our default experimental protocol; we automatically measure a set of NIST reference standards including LaB6, CeO2, TiO2, ZnO, and Si in addition to several duplicate measurements of the empty sample well with polyimide windows.

## Did I do it Right?

There are a few things to check to make sure that all is good.



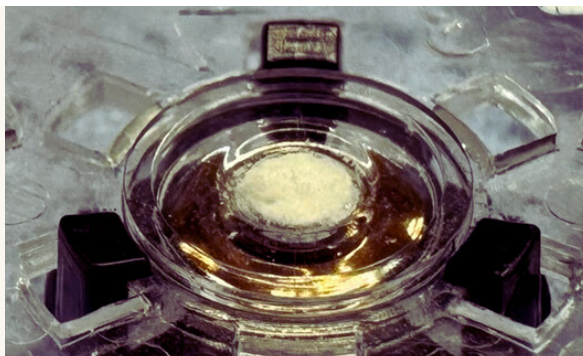
### Ensure the clips are secured.

This will ensure a tight fit of the polyimide windows and minimize any chance that the clips break off or leak material.

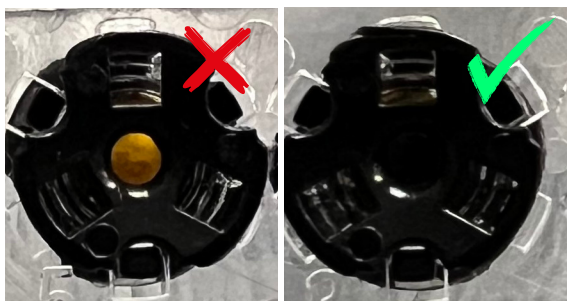
You should feel a secure click when the prong latches onto the sample holder. In the end, double check that the clips lie flat and do not have one side sitting higher up (an indication that the associated prong is still loose).

### Work cleanly.

Minimize spill-over of sample outside the well. This will help avoid safety hazards and further reduce chance of cross-contamination.



If a lot of powder does spill outside of the well, you can use a cotton swab wetted with some solvent to clean the excess around the edges or other surfaces of the sample holder.



### Fill the samples completely.

Check if light easily passes through the window. If so, you may have not sufficiently filled the well.

Maximizing the amount of sample in the scattering volume will improve the measurement statistics on both the scattering intensities and the crystallite orientations. This can be especially critical for multiphase samples, samples with large crystallites, e.g.  $\gg 10 \mu\text{m}$ , and weakly scattering materials (e.g. organics, low-Z, or minority phases/components). Densely packed samples will also give the best results for obtaining good scattering statistics at high angles for total scattering measurements.

## For Further Inquiries

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