Momentum Transfer

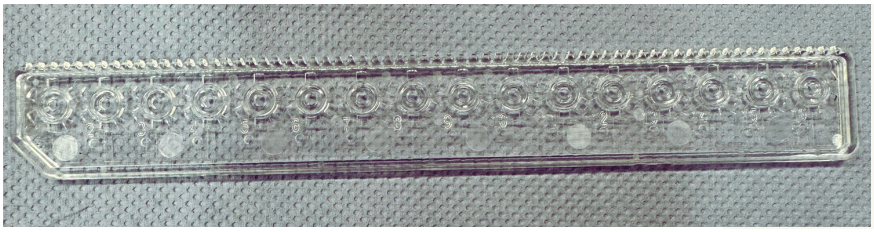
A Venture of Chemovator

Our high-throughput measurements are possible through a unique system that can cycle samples through the X-ray beam in seconds per measurement. In order to accomplish this, we prepare them in specially designed sample holders.

Here we demonstrate how the samples are loaded into the holder for best quality measurements.

Sample Preparation Tools

Descriptions of the sample holder components and tools used are listed below:



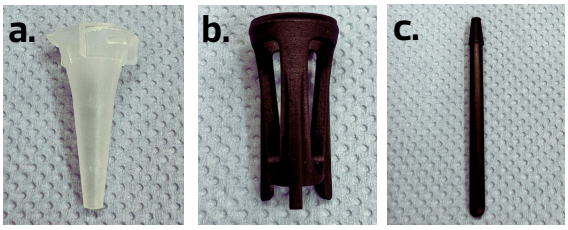
Sample Holder

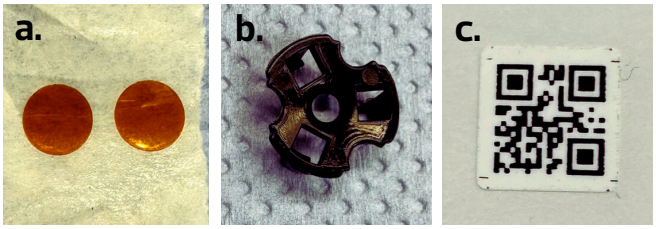
contains separate slots for up to 16 samples; each slot consist of a cylindrical window approximately 1.1 mm thick and 2.53 mm in diameter

Powder Loading Tools

several tools are included to aid in loading the powders:

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



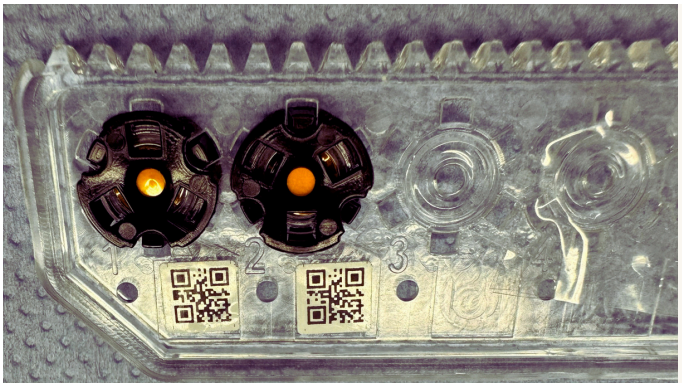


Sample Windows and ID Sticker

several more pieces for sample containment and indentification

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

How We Prepare the Samples



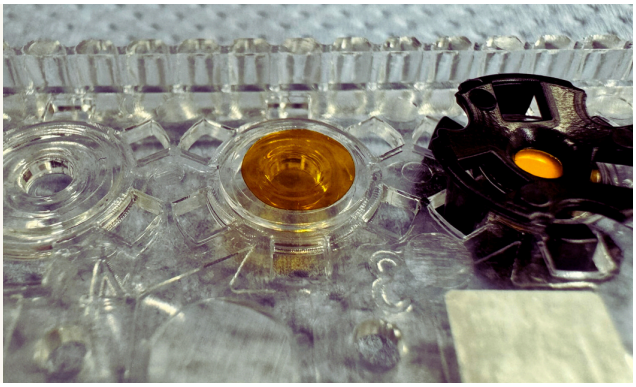
Two finished samples, let's do the next!

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 of the QR code are for laser alignment and should not be covered over.

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

Step 1:

The back side of an empty well is covered with a fresh polyimide window.



The window is centered over the opening for best surface contact.

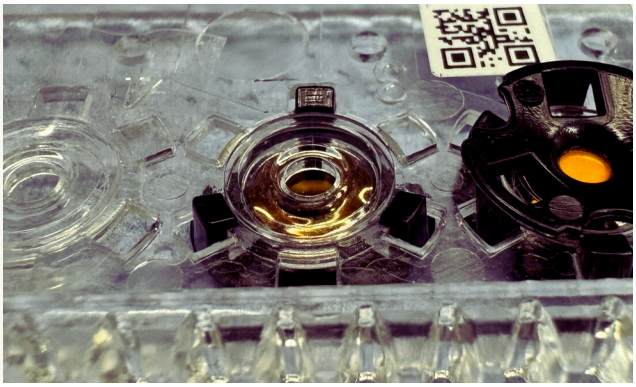
Step 2:

The window is fixed into place using one of the black clips.

The three prongs of the clip snap into three of the six openings around the central window holding the window tightly in place. Though tightly fitted, it is not perfectly sealed against ambient air. For air sensitive materials, preparations can be performed in a glove box and stored under inert gas until the measurements are performed.

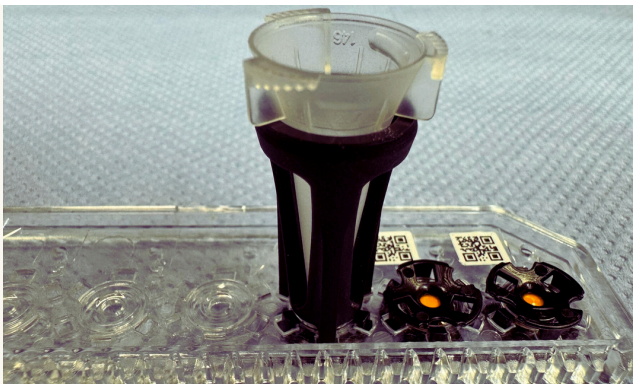
Step 3:

The sample holder is flipped over, ensuring that the black prongs are gripped onto the edges.



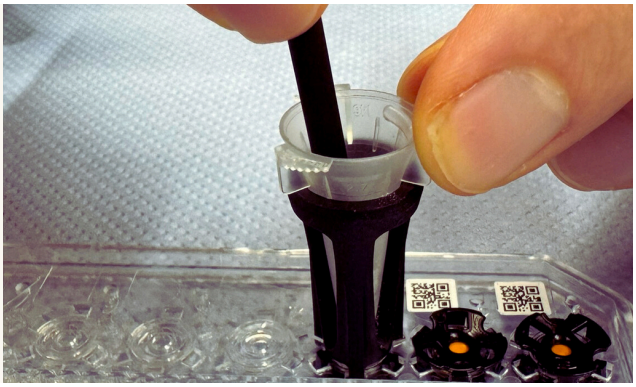
Step 4:

A funnel holder is positioned into the three remaining open slots around the window, and a clean funnel is inserted.



Step 5:

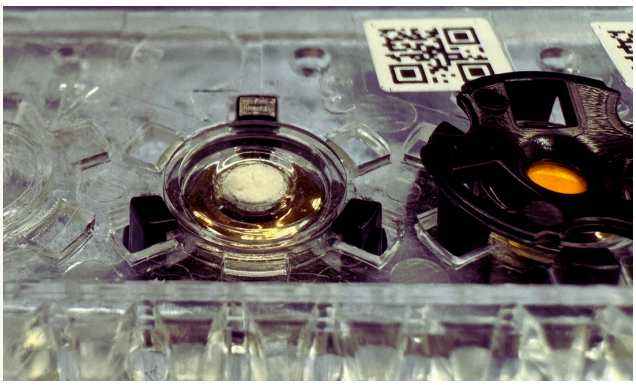
The sample is poured into the funnel and packed into place using the plunger.



For best quality measurements, the powder is filled completely and densely into the well. This ensures that the maximum sample cross section is available for best possible data statistics, and it maximizes the orientation distribution of the crystallites for acquisition of complete Debye–Scherrer rings. During measurements, we additionally shake the sample holder and scan the beam across the window to further improve the sampled volume.

Step 6:

Powder is filled completely and densely into the sample holder well.



Over-filling the well is avoided to keep the polyimide windows flat and maintain a constant background signal.

Step 7:

The open side of the filled well is fitted with another fresh polyimide window.

Step 8:

A second clip is placed, clipping the three prongs into the remaining open holes as before.

Step 9:

The sample is labelled with a QR code sticker in the square frame below the sample.

Sample QR codes are registered in our app https://app.momentum-transfer.com for robust sample tracking.

What about air sensitive materials?

The handling of samples during storage, shipment, and measurement should be carefully considered depending on the possibility for sample degradation under ambient conditions. The sample holders discussed above as-is are not specifically designed to be sealed against oxygen or moisture from the air. Nevertheless, we have tested some protocols for working with hygroscopic materials that are suitable for minimizing moisture uptake.

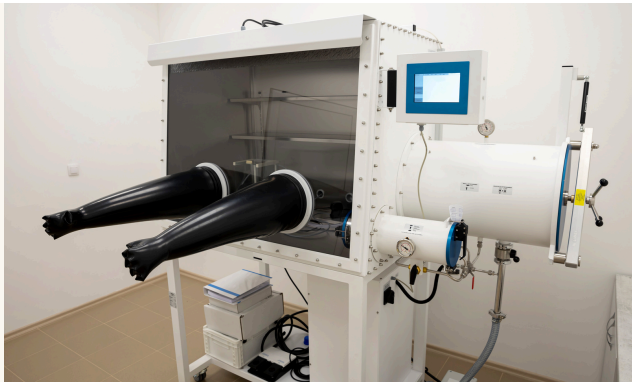
Shipping / Storage:

Samples sent in sealed containers under inert gas to prevent exposure during shipment.

Note: if samples should be loaded in the **glovebox**, then they should be placed in a properly sealed container that can withstand medium vacuum conditions during the purging of the antechamber.

Loading in glovebox:

Samples are then unpackaged and loaded under argon in a glovebox, and sealed with an extra layer of polyimide tape.



Sealing:

An extra layer of polyimide tape is used to cover the primary windows with a high amount of contact between tape and sample holder around the window edges.

Loaded sample holders are kept under **argon** until just before measurement.

If multiple measurements are to be performed during the same beamtime, the sample holders are additionally stored in a **desiccator** between measurements.

Although this has been successful for very hygroscopic materials like LiZrCl-type solid state electrolytes, we cannot guarantee that it will work for everything. Note, this is not suitable for materials that react violently or produce harmful products when exposed to air.

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 incuding LaB6, CeO2, TiO2, ZnO, and Si in addition to several duplicate measurements of the empty sample well with polyimide windows.

For air sensitive materials, additional background measurements should be prepared and measured with the samples to account for the additional layers added to seal the windows.

Further Inquiries

For questions please contact: support@momentum-transfer.com