

11-BM High-Resolution Powder Diffraction Mail-In Instructions:

Carefully read these instructions to ensure a successful mail-in experiment.

Please contact beamline staff (11BM@aps.anl.gov) with questions or comments at any time.

For more information about the 11-BM, visit our webpage: <http://11bm.xray.aps.anl.gov/>

SAMPLE BASE KIT:

The requested mail-in sample base kit(s) and pre-printed return-shipping label are enclosed.

Each sample kit contains the following:

- **Mounting Base:** a unique barcode number (e.g. ANLOAA1234) printed on the side & bottom (as a 2D barcode). This number is needed to register samples, and is used to track the sample in all steps of the mail-in service. **Do not use solvents to clean the base; this will damage the printed barcode and number.**
- **Magnetic Cap:** to cover and protect samples during shipment & handling. **Do not write on the caps; they are re-used and cannot be used to identify your sample.** All samples must fit completely inside the caps; the 11-BM robotic sample loader will destroy longer samples.
- **Kapton Capillary Tubes:** to contain samples. The inner diameter of the supplied tube is 0.80 mm. The outside diameter fits snugly inside the mounting base. **All samples (powders, foils, nested capillaries, etc.) must be fully contained and secured within straight Kapton tubes.** Samples are spun at high speed during data collection. Non-secured samples will shift or move out of the beam position when spun. We cannot mount unsecured or bend samples. Kapton tubes are provided for your convenience. Kapton is stable over a wide temperature range and adds a negligible contribution to the diffraction scan background.



PROHIBITED SAMPLE HAZARDS:

11-BM does **NOT** accept any of the following sample hazards for mail-in work:

- Biohazard or Human-Derived Materials
- Non-Sterilized Regulated Soils
- Radioactive Materials
- Explosives or Unstable Materials

We do not accept samples **combining** the following hazards: *flammable*, *corrosive* or *oxidizer*.

Regulated metals ("RCRA" class: Ag, As, Ba, Cd, Cr, Hg, Pb, Se) are accepted, but must be identified when registered.

All samples shipped to the APS must qualify for small quantity exceptions under U.S. Dept. of Transportation (DOT) regulations (CFR Title 49 §173.4). In general, most samples (except those listed above) are acceptable under these regulations except materials that are pyrophoric (ignite in air) (§173.124b) or dangerous when wet (§173.124b). **Full safety details and definitions are available on the 11-BM website.**

Please take special care to ensure that samples potentially hazardous to beamline staff (Nano, Toxic, Carcinogenic, Flammable, etc) are securely contained and permanently sealed in the Kapton tubes.

REGISTRATION & SCAN REQUESTS:

Sample registration is required for APS safety forms, and allows mail-in users to request scan parameters.

You must register and receive approval from 11-BM staff BEFORE mailing any samples to the APS.

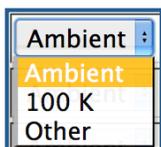
Register your sample(s) using the provided email links or on the 11-BM web page at:

<http://11bm.xray.aps.anl.gov/user.php?step=2>

For each sample, provide a chemical name & formula. A sample ID for your records is optional. Do **not** use non-element symbols to stand for classes of elements (RE for rare-earth, etc). Compositions may be symbolic or approximate [e.g. Na(x)Li(1-x)O]. Also specify the appropriate hazard(s) information.

Check all the hazard boxes that apply. Hazard definitions are available on the 11-BM webpage

Select scan parameters for each sample using the pull-down menus. Select a standard (\approx 1 hour) scan at room temperature **Ambient** or low temperature **100 K**. Alternatively, select **Other** from the menu to request non-standard scan types or temperatures (100 - 450 K), or to select multiple scans (i.e. two temperatures) for a single sample. Note custom scan requests are added on a subsequent webpage (this page appears only after clicking on the "Register Sample Info" button to proceed). See 11-BM webpages for custom scan options & restrictions.



When prompted by email, you must **take action to confirm** the registration information. This "e-signature" is necessary to comply with APS and DOE safety regulations.

It is important that samples are registered, confirmed, and approved before shipment. **Unregistered samples arriving at the APS may be destroyed.** Repeat offenders may lose mail-in access to 11-BM and the APS.

SHIPPING:

Users are responsible for shipping samples to Argonne in full compliance with DOT rules and regulations (see Safety Notice above). You may use the US Mail or a commercial shipping company (FedEx, UPS, DHL, etc). We recommend protecting samples in bubble wrap (provided) or some other padding during shipment. Do not add a signature requirement for delivery of your package.

NEW GLASS RULE !!! The use of glass vials, ampoules or other containers for shipping 11-BM samples to the APS is now **prohibited**. (excluding nested sample quartz capillaries). Instead, simply use bubble wrap or plastic containers. Most of the time, glass containers are broken on arrival at the APS, which is dangerous for beamline staff and does nothing to protect your samples



Email notification is sent when samples are received. We are unable track shipped samples or sample base kits.

Samples should be shipped to this address:

Beamline 11-BM
Advanced Photon Source
Argonne National Laboratory
9700 South Cass Ave, Building 433/D002
Argonne, IL 60439
USA

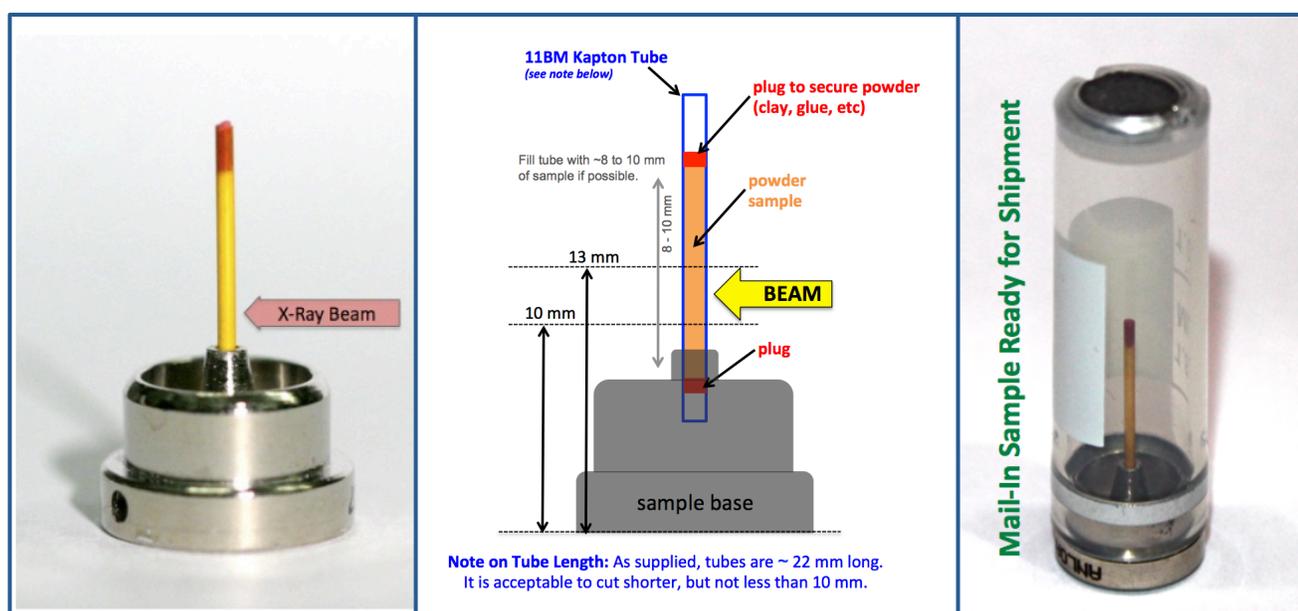
International users shipping from outside the U.S. are responsible for all appropriate international chemical shipment regulations and paperwork. We cannot assist with international shipping paperwork or provide documentation or payment for samples.

SAMPLE PREPARATION:

Sample preparation is critical and can dramatically impact the quality of your collected data. Consult the 11-BM webpage or contact beamline staff with any questions on sample preparation **before** shipping your sample(s). For each powder sample or composition; consider the following:

- **Sample Position & Containment:** Sample powder must be located in the beam position when mounted by the 11-BM robot (see diagram at bottom). Other materials (glue, wax, clay) in the beam position will add peaks to the diffraction pattern. **Powders must be fully contained & sealed in the capillary, and must not shift during shipment or when spun (> 60 Hz) during data collection.** Supplied capillaries normally fit snugly inside 11-BM sample bases without a need for glue, if necessary use only a small dot of adhesive *inside* the mounting hole. A capillary is sufficiently secure if you can hold a base upside down without the capillary falling.
- **Particle Size & Morphology:** Ideally, all sample powders would have a small (≈ 5 micron) and homogeneous particle size and shape distribution. In reality, this is not always possible or practical. However, please do consider how non-ideal powder size or shape distributions impact your data. Large crystallites tend to reduce particle counting statistics, while very small (or strained) particles will result in peak broadening. Nano-materials may have VERY broad peaks.
- **X-ray Absorption:** In general, heavy elements (high Z) absorb X-rays more strongly than light elements (Pb vs Be). 11-BM uses transmission (Debye-Scherrer) geometry; therefore sample X-ray absorption must be considered. Absorption is not normally an issue for most mail-in users; the high energy beam (≈ 30 keV) easily penetrates these sample powders. However, for samples containing a large fraction of high-Z elements, absorption can be problematic unless special care is taken. See our webpage for more information and a web based X-ray absorption calculation tool.
- **Radiation Damage:** Ionizing radiation from the intense synchrotron beam can induce damage in samples via several mechanisms. While oxide or metallic samples are largely unaffected, samples containing organic materials may suffer radiation damage (e.g. proteins, MOFs, pharmaceuticals, etc). Cooling samples during data collection can help to minimize this effect, but the extent of damage at any temperature is hard to predict in advance. If obvious beam damaged is observed during data collection you will be notified. Collection strategies are available at 11-BM to reduce the impact of radiation damage; contact beamline staff for more details.
- **Atmosphere & Temperature Stability:** Ensure that sample powders and containment materials (wax, glue, clay, etc) are compatible with the scan temperature(s) requested. Air or moisture sensitive sample may be sealed inside the Kapton tubes using epoxy; however, the gas permeability of Kapton is not guaranteed. Alternatively, first seal sensitive powders in a glass or quartz capillary. **This inner capillary MUST be contained and secured, and fit completely inside the supplied Kapton tubes.** These samples should be clearly marked for special handling and will be rejected if the glass/quartz capillary is not enclosed in Kapton. Contact beamline staff for more details.

Figures below illustrate a properly prepared sample for the 11-BM mail-in service, including the position of the X-ray beam on the sample capillary tube. Red material visible at the capillary tip is modeling clay used to secure powder inside the Kapton tube. See the 11-BM webpage for larger images and advice on sample preparation.



OBTAINING DATA:

Email notification will be sent to users once data have been collected on your sample(s). Our goal is to collect data on registered samples within \approx 3 weeks of receipt at 11-BM. Often data is sent much sooner, sometimes within just a few days, but depending on beamline scheduling and the APS operations calendar (check the APS webpage for dates) there may be an unavoidable delay. Once collected, beamline staff reviews each data set and may include comments in the notification email(s) when appropriate.

Data may be obtained by following the provided email links or on the 11-BM web page at:

<http://11bm.xray.aps.anl.gov/user.php?step=4>

Users may request data be emailed directly (sent as an archived zip file), or posted to an ftp site.

Additional information about 11-BM data and file formats is available on the website.

PUBLISHING:

The free mail-in powder diffraction service at 11-BM is made possible by funding from the U. S. Department of Energy. By reporting your publications that include 11-BM data, you demonstrate the value of this beamline to your research and help ensure that funding for the program is continued.

Please report all citations to beamline staff (11BM@aps.anl.gov). We will also periodically inquire on the status of unreported mail-in user data via automated emails.

It is APS policy is that publications using data collected at the APS contain the following acknowledgement:

Use of the Advanced Photon Source at Argonne National Laboratory was supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.

Thanking beamline staff is always welcome, but co-authorships are only expected where we have been involved in experiment design or data analysis.

DISPOSAL:

By default, all mail-in samples sent to 11-BM will be saved for a minimum of two weeks after data collection before being disposed. Under special circumstances, samples may be returned if requested in advance.

All requests for sample return must be made via the 11-BM user web pages at the time of sample registration.

<http://11bm.xray.aps.anl.gov/user.php?step=3>

Users requesting sample return should arrange for payment of shipping costs (e.g. APS cost code, FedEx #, etc) and MSDS sheets are required for all compositions. Requests will be processed when beamline operations allow; no specific delivery dates can be guaranteed. Note: sample return is time consuming and could impact the number of accommodated samples; please request return only where samples are of significant value.

Due to the complexities of overseas sample shipping, we regret that samples cannot be shipped to addresses outside of the U.S. If needed, please find a U.S. based collaborator to assist with international sample return.

QUESTIONS / COMMENTS?

Contact 11-BM beamline staff at anytime (11BM@aps.anl.gov) with your questions, comments, or suggestions.