

Instructions for 11-BM Mail-In Service:

Thank you for your interest in the APS high resolution synchrotron powder diffraction mail-in service at beamline 11-BM. **Please carefully read and follow the instructions below to ensure a successful mail-in experiment.** Contact beamline staff (11BM@aps.anl.gov) with questions or comments at any time.

In this package, you will find your requested mail-in sample base kit(s) and a pre-printed return shipping label.

SAMPLE BASE KIT:

For each requested sample base, this shipment contains: one sample mounting base, one magnetic cap, and Kapton sample capillary tubes (inside a plastic vial). Additional Kapton tubes are provided for your convenience.

- **Mounting Base:** Holds one sample capillary tube and is integrated with the robotic arm used for automated data collection. The unique 10 digit barcode (e.g. ANL0AA1234) printed on the side, also encoded in a 2D barcode on the bottom, tracks the sample at all steps of our mail-in service.
Note: The barcode number is required when registering each sample online. Do this BEFORE returning samples to the APS.
- **Magnetic Cap:** A magnetic ring secures caps on mounting bases, and provides some protection for sample capillary tubes during shipment. Please avoid writing on the caps, as they are re-used and cannot be used to identify your sample.
Note: all samples must fit completely inside the caps when placed on mounting bases; longer samples may be destroyed by the 11-BM robotic sample loader.
- **Capillary Tubes:** The supplied Kapton tubes have an inner diameter of 0.80 mm, and an outer diameter that fits snugly inside 11-BM mounting bases. Kapton is stable over a wide temperature range and adds a negligible contribution to the diffraction scan background. **Note:** All sample powders must be fully contained and secured within these capillary tubes.



SAFETY NOTICE:

The mail-in service at 11-BM **cannot** accept any of the following sample types:

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

Note: We do not accept materials that possess > 1 of the following hazards: *flammable, corrosive and oxidizer*.

RCRA regulated metals (Ag, As, Ba, Cd, Cr, Hg, Pb and Se) can be accepted, but samples containing them must be identified (see "Register Sample Base Numbers" below).

Additionally, all registered 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:

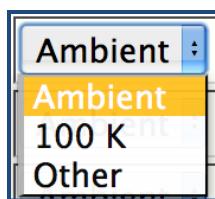
You must register and receive email approval from 11-BM staff BEFORE mailing any samples to the APS.

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

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

For each sample, provide a complete chemical name, a chemical formula that specifies all elements present in the material by the one or two-letter elemental symbol, and an optional sample ID for your records. 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. After submitting this information online, you will be sent an email to which you MUST reply and electronically verify the provided information. This "e-signature" is necessary to comply with APS and DOE safety regulations.

Check all the hazard boxes that apply. Eight metals (Ag, As, Ba, Cd, Cr, Hg, Pb and Se) are regulated as "heavy metals". If your sample contains any of these, please note that by selecting "Other, specify:" in addition to the other hazards (they would be "Toxic" as well, for example) and note "RCRA Metals" in the text-entry box next to the "Other, specify:" check-box.



You will receive a confirmation email when your sample registration is reviewed and approved; only then may you send samples to the APS (see SHIPPING below). The provided sample information (once approved) is used to generate an Experimental Safety Approval Form (ESAF) for your experiment. For 11-BM mail-in users, an ESAF is automatically submitted and posted on your behalf by beamline staff.

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

SELECTING SCAN PARAMETERS:

Mail-in users select scan parameters for their samples during sample registration (see above). Scan types are selected by the pull-down menu on the registration webpage. Most users select a standard (\approx 1 hour) scan at room temperature (**Ambient**) or low temperature (**100 K**). Alternatively, after first providing sample information, users may select "**Other**" to request custom scan parameters for one or more samples on a subsequent webpage (this page appears only after clicking on the "Register Sample Info" button to proceed).

Custom scan options include select temperatures between 100 K (-175 °C) and 400 K (+125 °C), and a choice of more specialized scan parameters (2θ range, step size, etc). The total estimated experiment time is tracked and limited to 1 shift (8 hours) per rapid access mail-in proposal. Note: we cannot guarantee the run order of mail-in samples – they will be run in the most efficient sequence for data collection, not necessarily the order you specify. Please consult the 11-BM website for full details or email beamline staff with questions.

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.

Email notification will be sent when samples are received at the beamline. Note: we do not track sample base kits sent out, and cannot help track samples shipped back to the APS. Also, do not add a signature requirement for delivery of your package; these samples may be delayed or returned by the laboratory receiving office.

Samples should be shipped to this address:

Beamline 11-BM
9700 South Cass Ave, Building 433/D002
Argonne National Laboratory
Argonne, IL 60439-4856

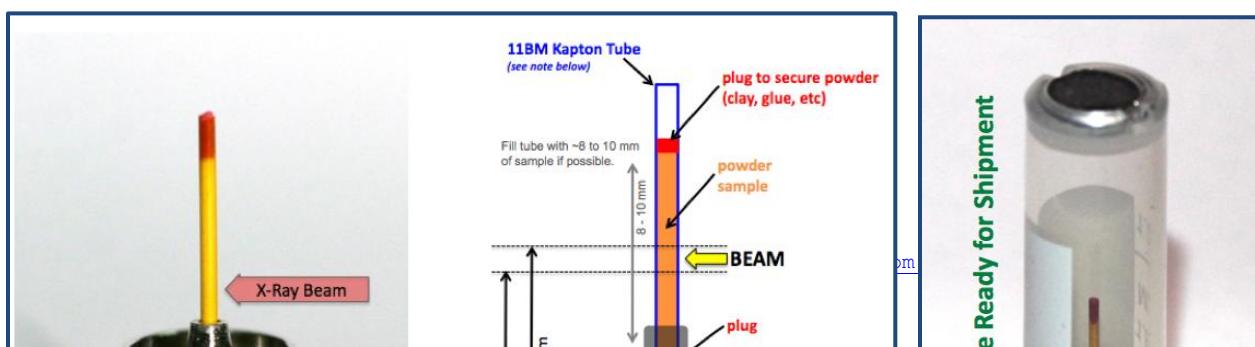
International users shipping from outside the U.S. are responsible for all regulations covering international shipment of chemicals. Ensure paperwork required for entry of samples to the U.S. is done in advance and is filed with the shipment. Please note if samples are assigned a value, US Customs may request duty payment. 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 should fit snugly inside 11-BM sample bases without a need for glue. 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 damage 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. However, this inner capillary MUST also be contained 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 ≈ 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.xor.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.xor.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?

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

Thank you for reading all the way to the end, and good luck with your mail-in synchrotron powder diffraction experiment at 11-BM! Please feel free to contact 11-BM beamline staff at anytime (11BM@aps.anl.gov) with your questions, comments, or suggestions about how we can improve this service.