

Sample Submission for XPS (ESCA) Analysis

Name:	Date (yyyy-mm-dd):
Email:	NetID or GuestID:
Affiliation:	PI:
Coral account (if you have more than one):	
List everything expected to be in your samples. CCMR needs to understand the associated hazards, and it will also help with analysis. Safety data sheets must be provided for hazardous materials.	
Sample type (film, powder, etc.):	
Nanoparticles?	Yes No Conductive? Yes No Unsure
Can the samples be cut, modified, or broken?	Yes No
CCMR will not provide your XPS data until you have retrieved your samples after analysis using one of the following options: You will retrieve your samples from D21 Clark Hall You will provide packaging and a prepaid UPS or FedEx shipping label	
Perform survey scan?	
List range (default is full 0-1100 eV range):	
Perform high-sensitivity composition scans?	
List elements:	
Perform high-resolution chemical bonding scans?	
List elements:	
Perform preliminary analysis?	

**SAFETY DATA SHEETS MUST BE PROVIDED
FOR HAZARDOUS MATERIALS**

XPS/ESCA Frequently Asked Questions

Q: How do I submit samples?

A: Submit samples and Safety Data Sheets for any hazardous materials along with this form. You may submit and retrieve samples in the desiccator cabinet in Clark D21 (send a note to ccmr-xps@cornell.edu when you do). Or, you may ship samples via UPS or FedEx, **but you must also provide a prepaid return shipping label and return packaging.** Ship samples to:

CCMR Clark Materials Facility
D21 Clark Hall, Cornell University
142 Sciences Drive
Ithaca, NY 14853

Q: I don't want my samples back. Can CCMR discard them after analysis?

A: No, CCMR will not dispose of your samples for you. You must retrieve your samples or arrange to have them shipped back to you at your expense before CCMR will provide you with your XPS data. **Samples left at the facility will be charged a recurring chemical inventory administration fee of \$10 per month per sample.**

Q: I don't have a CCMR Coral account. Can I still have XPS analysis done?

A: A Coral account is required for XPS analysis. You may request an account at <http://www.ccmr.cornell.edu/facilities/request>.

Q: Can users outside of Cornell have samples run?

A: Yes. A Coral account and purchase order number is required. Purchase orders should be faxed to 607-255-3967 or emailed to ccmr-coral-admin@cornell.edu.

Q: How long will it take to run my samples after I submit them?

A: It depends on the availability of the spectrometer. If you need more specificity, you can coordinate in advance with the XPS operator by sending email to ccmr-xps@cornell.edu.

Q: Do you offer XPS training?

A: We do not recommend infrequent users to run the XPS instrument unsupervised. Full training may be beneficial to frequent XPS users. Fully-trained users will learn how to independently prepare and install samples, prepare instrumentation, set up analysis scans, export and analyze data. Less frequent users can be trained to perform analysis, which will reduce charges associated with staff time.

Q: What is the recommended sample size?

A: Typical x-ray analysis spot is a 1x2 mm ellipse. A 1 cm² sample is easy to analyze, but samples can be smaller, and larger, up to 75 mm diameter. Standard mounting can accept as many samples as will fit on an 75 mm diameter area. Some techniques may limit the number of samples that can be mounted on one sample holder.

Q: What is the analysis depth?

A: Typically analysis depth is ~5 nm for metals and ~10 nm for polymers and is dependent on material and photoemission angle. The IMFP TPP2M program provides electron escape depths for various elements and can be downloaded at www.quases.com. The emission angle is typically 55°. An analysis depth worksheet is available at <http://www.ccmr.cornell.edu/instruments/x-ray-photoelectron-spectroscopy-xps/>.

Q: How do I prepare samples?

- *Label samples and/or containers well.* Containers should indicate the contents, your email, and the date. Identify samples with some kind of ID for analysis. Do not use pens, markers, or scotch tape on samples, as these will outgas in vacuum. If the front and back of your sample look identical, make sure to identify which surface is to be analyzed. If a particular region on your sample needs to be analyzed, include a drawing or picture.
- *Cleanliness is of utmost importance:* Do not touch the surface of your sample with anything (no fingers, gloves, tweezers, breath, acetone, alcohol, etc.). Do not put your samples in plastic bags or aluminum foil. XPS is exquisitely sensitive to anything that comes in contact with the surface of your sample (including adventitious carbon that deposits when surfaces are exposed to air).
- *Conductivity is good:* If possible, choose smooth, conductive substrates like doped silicon wafers. Conductive films on insulating substrates can be grounded with double-sided carbon tape.
- *Liquid samples:* may be drop cast onto silicon, gold film, or carbon tape, depending on the peaks of interest.
- *Transport:* The recommended way to prepare your samples is on aluminum SEM 3.2 mm pin-type mounts (e.g. http://www.tedpella.com/SEM_html/SEMPinmount.htm) using conductive double-stick tape (e.g. http://www.tedpella.com/SEMisc_html/SEMadhes.htm), and pack these mounts in an SEM pin-type storage box for transport (e.g. http://www.tedpella.com/storage-boxes-bags_html/SEM-mount-storage-boxes.htm). Alternatively, Fluoroware containers with curved bottom may protect flat, face-down samples. Glass containers are also good for powders, or larger individual samples that have been secured to protect the surface.

Q: How much does XPS analysis cost?

A: Academic and corporate rates are posted online <http://www.ccmr.cornell.edu/facilities/user-fees/>. Typically, a survey or high-resolution scan can take 20-60 minutes depending on sample quality, surface roughness, or surface contamination. A high-sensitivity scan typically takes about 5 minutes. There is an additional charge to cover staff time spent mounting samples, preparing the instrument, and performing preliminary analysis, which typically takes up to an hour. Monitored runs (such as depth profiling) and extensive analyses will incur additional staff labor charges depending on the time involved. **For estimation of cost, two hours of machine time per sample is a good starting point, but may be more or less depending on the types of scans required.** Users are encouraged to learn basic analysis methods, and other methods as needed (high resolution, angle-resolved, etc). CasaXPS is used for data analysis and instructions and a registration code is available to all Cornell users. Non-Cornell users can use the CasaXPS demo version with all capabilities except for saving and printing. See www.casaxps.com for licensing and other information.

Q: When I publish XPS data, how do I acknowledge CCMR?

A: Thank you for asking. All publications and patents (including applications) resulting from research supported by CCMR must acknowledge CCMR support and the appropriate grant number. See <http://www.ccmr.cornell.edu/research/acknowledging-ccmr-funding/> for details.

Any questions not covered here? Please direct them to ccmr-xps@cornell.edu.