Sample Submission for XPS (ESCA) Analysis

Name: Date (yyyy-mm-dd):

Email: NetID or GuestID:

Affiliation: PI:

Coral project/account to use, if 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 may 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 0-1200 eV range):

Perform any individual high-sensitivity composition scans ☐ (*e.g.* for trace elements not seen in wide surveys) List elements of interest:

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 during work hours to/from the desiccator cabinet in Clark D21 (send a note to ccmr-xps@cornell.edu when you do). If you need after-hours access to D21, complete the access form online. 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: Can users outside of Cornell have samples run?
A: Yes. You may request an account at http://www.ccmr.cornell.edu/facilities/request. Accounting questions can be sent to ccmr-coral-admin@cornell.edu.

Q: I don't have a CCMR Coral account. Can I still have XPS analysis done?
A: A Coral billing account is required for each individual researcher (no shared accounts).

Q: How long will it take to run my samples after I submit them?
A. Turnaround time is typically 2-4 days for standard measurements but will depend on the availability of the spectrometer(s). 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: Yes, but we do not recommend infrequent users to run the XPS instrument unsupervised. Full training will be beneficial to frequent XPS users. Fully-trained users will learn how to independently prepare and load samples, set up analysis parameters/scans, export and analyze data. Less frequent users can be trained to perform data acquisition**,** which may reduce charges associated with staff time.

Q: What is the recommended sample size?
A: A 1 cm^2 sample is usually perfect, but samples can be somewhat smaller or larger. Powders will typically be spread onto a 5x5mm piece of carbon tape. Some techniques may limit the number of samples that can be mounted onto one sample holder.

Q: What is the analysis depth?
A: Typically analysis depth is ~3 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. Emission angles are typically 0-60°. 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. **Number your samples (1,2,3,...).** Do not use pens, markers, or scotch tape directly on samples, as these can 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.
- Powder samples: Powder should be sent in glass or plastic vials. The quantity of powder consumed will fit onto the tip of a metal spatula.
- *Liquid samples:* may be drop cast onto silicon, gold film, or carbon tape, depending on the peaks of interest.
- *Transport:* Fluoroware containers with curved bottom may protect flat, face-down samples. It may be possible to analyze samples on SEM stubs.

Q: How much does XPS analysis cost?

A: Academic and corporate rates are posted online http://www.ccmr.cornell.edu/facilities/user-fees/. For samples being analyzed as a service there is a fixed labor charge (typically ~1 hour) with actual instrument time added. A typical survey or high-resolution scan can take 2-10 minutes depending on sample quality, surface roughness, surface contamination and desired signal quality. Monitored runs (such as depth profiling, UPS, etc.) and extensive analyses will incur additional staff labor charges depending on the time involved. For estimation of cost, 20-60 minutes 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/current grant number. See http://www.ccmr.cornell.edu/research/acknowledging-ccmr-funding/ for suggested wording.

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