Sample Submission for XPS Analysis

Name: Email:	Date (yyyy-mm-dd): NetID or GuestID:
Affiliation: FOM account (if you have more than one):	PI:
Provide sample description (film, powder, nanoparticles, etc.) List everything expected to be in your samples. CCMR must understand all material safety risks. **Safety Data Sheets must be provided for hazardous materials.** Is your sample conductive?	
CCMR will not dispose of any samples. Please choose a retrieval method. ☐ You will retrieve your samples from D21 Clark Hall ☐ You will provide packaging and prepaid UPS or FedEx shipping label	
Describe what you are trying to learn about your samples below:	
Survey spectra are taken by default. List elements below for high-resolution scans:	
A tutorial for data processing and analysis in CasaXPS is available at: https://fom.ccmr.cornell.edu/fom/documents It covers file handling, atomic percent calculations, spectral peak fitting for chemical bonding information, and data exportation. CCMR is available to answer questions, but any in-depth analysis may incur charges for staff hours.	

XPS 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@email.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 FOM account. Can I still have XPS analysis done?
A: A FOM account is required for use of any CCMR resources. Please help us track number of individual researchers we impact, by requesting your individual account at https://www.ccmr.cornell.edu/facilities/becoming-a-ccmr-facility-user/

Q: Can users outside of Cornell have samples run?
A: Yes. A FOM account with billing contact information is required. Invoices will be sent out soon after the work is completed. Email any billing updates to ccmr-fom-admin@email.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@email.cornell.edu.

Q: Do you offer XPS training?
A: 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 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: XPS spot sizes range from 10-400 μm. A 1 cm^2 sample is easy to analyze, but samples can be smaller and larger up to 60 mm. Standard mounting can accept as many samples as will fit on an 60 mm square 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 QUASES and NIST programs provide electron escape depths for various elements and materials and can be downloaded at www.quases.com or https://www.nist.gov/srd/nist-standard-reference-database-7. The emission angle is typically 90° relative to the sample surface.

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 (e.g. A/B/C/D or 1/2/3/4). **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 directly in plastic bags, packing material, 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 to secure them with double sided conductive carbon tape in Fluoroware containers. Fluoroware can be cleaned with 2-propanol and reused. Glass containers are good for powders or larger individual samples that have been secured to protect their 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 single point where a survey and ~5 high-resolution scans are run takes 10-30 minutes depending on sample quality, surface roughness, or surface contamination. Surfaces are rarely homogeneous, so 2 points minimum are recommended per sample surface. 3 points are ideal for statistical analysis. 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 unless spectral fitting is requested. 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 data analysis methods and other measurement methods as needed (angle-resolved, UPS, 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@email.cornell.edu.