XPS Frequently Asked Questions

**What is XPS?**

X-ray photoelectron spectroscopy (XPS) is used to identify and quantify surface elements via detection of core and valence electrons with binding energies characteristic of those elements. The high resolution of this technique enables the user to distinguish minute variations in the emissions to, for example, identify functional groups and oxidation states.

**What kinds of materials can be analysed with this technique?**

XPS can be used to characterise organic, inorganic and metallic thin films and semi-conductor surfaces. The in-built charge neutraliser allows insulating materials and rough surfaces to be measured by compensating for spectral shift caused by charge build up on the surface. Owing to this, small granules and powders can be measured by being spread into a thin layer on an adhesive.

All elements can be detected by XPS, the only exceptions being hydrogen and helium. The ability to distinguish between oxidation states and chemical environments will vary – some elements show larger chemical shifts than others.

**What is the make/model of the XPS instrument?**

It is a Kratos AXIS Supra+ (<https://www.kratos.com/product/axis-supra-xps-surface-analysis-instrument/>)

**What experiments are possible with the Axis Supra+?**

The Kratos AXIS Supra+ is equipped a variety of excitation sources and experimental capabilities. Please note that powder samples can *only* be submitted for XPS, and samples *must* be at least partially conductive to be submitted for UPS or REELS.

The gas cluster ion source (GCIS) produces monatomic argon ions or 1000+ cluster argon ions at variable power for surface etching and depth profiling. Monatomic ions will etch faster but may damage softer materials. Cluster etching is gentler but also causes localised heating that can affect emissions. An iterative process may be necessary to find the correct settings for your material. Any literature you can find that indicates an acceptable procedure would be helpful.

The ultraviolet light source and helium gas supply are fitted for ultraviolet photoelectron spectroscopy (UPS) to probe low binding energy valence electrons with improved sensitivity and resolution over XPS. This technique can be used to determine the valence band edge of a material.

An electron source power supply is used for reflection electron energy loss spectroscopy (REELS), to determine electronic band gaps or energy levels of unoccupied molecular orbitals.

**Are there any additional capabilities?**

It has a small N2 purged glovebox and a transfer arm that can be used separately or in tandem to introduce air-sensitive samples.

The heated stage can be raised up to 800°C and the cooling accessory can bring samples below -100°C using liquid nitrogen.

There are a variety of sample holders for in-situ experiments.

**How should I prepare my samples?**

Single piece samples should be ideally between 5 and 15 mm in width and length and no thicker than 6 mm. Measurement of larger samples can be arranged on a case-by-case basis.

Samples should be dry, and powders degassed if possible. The department has access to dedicated degassing instruments if needed.

To ensure reliable data, it is strongly encouraged to provide reference materials in addition to your samples.

If the surface layer or substrate of a sample is conductive, then specific efforts should be made to ground samples to the instrument to align the fermi levels of both for the most accurate measurements possible. Strategies and experimental methods for this will be discussed with users in the first instance.

**How do I arrange XPS analysis?**

Contact Chris Kelly (Christopher.Kelly.3@glasgow.ac.uk) in the first instance, and consult the ‘Accessing the Kratos AXIS Supra’ document for details

**How much does XPS analysis cost and how do I pay?**

Presently, XPS is charged at £25 per sample. Depth profiles are charges at £100 *pro rata*. Work done for users external to the University of Glasgow will be subject to VAT as well.

For internal users, payment will be handled twice per year – usually in November and May. All charges will be sent to your PI, along with cost breakdowns and budget codes.

For external users, following the completion of your work, a quote will be sent to you so that you can raise a purchase order and send it on to me. An invoice will then be sent on to you by our finance team.