

**INDIAN INSTITUTE OF TECHNOLOGY BOMBAY**  
**Central Surface Analytical Facility (ESCA Lab)**

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**External User Registration Process**

Bonafide user of (a) academic institutions, (b) national R & D laboratories and (c) industries can avail this facility. Registration form along with a letter from the Guide/ HoD/authorized person from any of the above institutions can be submitted in person or through registered post / courier along with the administered charges to Convener, ESCA Lab, Room No. 020, Physics Dept., IIT Bombay, Mumbai - 400076.

You are requested to mention in your request letter that *"We agree to acknowledge the Central Surface Analytical Facility of IIT Bombay when the data from the ESCA lab are used in our papers/reports/thesis"*.

The information on such acknowledgements with appropriate reference should be communicated to ESCA lab vide email [esca@iitb.ac.in](mailto:esca@iitb.ac.in). Kindly send the complete publication reference (Journal name/volume number/names of the authors/date of issue of the publication etc).

**The following charges\* are applicable for External Users:**

Description	Academic	National R & D Lab	Industry/Non-Govt. Agency
Per sample/per mode <sup>#</sup>	Rs. 3000/-	Rs. 8000/-	Rs. 12000/-

<sup>#</sup>Modes: XPS, UPS, AES

\*GST extra as on 1.8.2017:

- 1) If the recipient of the report is from Maharashtra: 9% SGST and 9% CGST
- 2) If the recipient of the report is from outside Maharashtra: 18% IGST

Please provide your GSTIN, if available

For samples requiring more than two hours of analysis, there will be 30% extra charge per hour.

- **Payment** should be made in advance by a Demand Draft (DD) drawn in favor of "The Registrar, IIT Bombay, P and C Account". The same should be sent to Convener, ESCA Lab, Room No. 020, Physics Dept., IIT Bombay, Mumbai - 400076, along with the Request Letter and Registration Form.
- **Appointment:** The users will be informed about their date and time-slot by email for submission of sample/s. The analysis is typically done on the next day. If the day

and time-slot is not suitable for you, an email request should be sent immediately for an alternate slot.

- **Sample Preparation:** Please see the details below
- **Sample Submission:** Samples are to be brought along on the date of your appointment for submission (Between 9:30 to 11 AM). The samples will be analyzed on the next day of submission.
- **Analysis results:** After the sample analysis is complete the results will be sent through email.

### **GENERAL INSTRUCTIONS TO THE USERS**

1. The experimental data provided is only for research / development purposes. These cannot be used as certificates in legal disputes.
2. A maximum 3 samples will be analyzed against a single Registration Form.
3. MSDS (Material Safety Data Sheet) should be given along with samples to ensure that the samples are not toxic or hazardous. Samples will not be accepted unless accompanied by MSDS.

#### **Lab Address:**

**ESCA Lab**  
#020, Dept. of Physics  
Indian Institute of Technology Bombay  
Powai, MUMBAI 400076

21-08-2017

## About the facility

### Working Principle:

Electron spectroscopy for chemical analysis (ESCA) instrument comprises of X-ray photoelectron spectroscopy (XPS), Ultra-violet photoelectron spectroscopy (UPS) and Auger electron spectroscopy (AES) / Scanning Auger Microscopy (SAM).

The surface to be analyzed is placed in a vacuum environment and then irradiated with photons (X-ray or Ultra-violet). The incident photons release electrons (called photoelectrons) from their electronic structure (core-levels and valence band) and ionize the atoms. The kinetic energy and the number of photoelectrons are precisely measured and counted respectively using an analyzer-detector based on which the binding energy and the intensity of the photoelectron are determined. AES/SAM is based on the Auger effect (series of internal relaxation events upon ionization of core level) leading to emission of electrons called Auger electrons. It uses primary electron beam (3 to 10 keV) and the possibility to focus and scan this primary electron beam in nm and  $\mu\text{m}$  range. The Auger electrons are part of the secondary electron spectrum with characteristic energy allowing one to identify the emitting element. The experimental setup is somewhat similar to that of SEM with a difference, that is, the electrons are not only used for imaging but also for chemical identification of the surface atoms.

Limitations: ESCA can detect all the elements except H and He. The detection limit is 0.1%

### Reference Books:

1. An Introduction to Surface Analysis by XPS & AES, John F. Watts & John Wolstenholme, Wiley 2003.
2. Surface Analysis – The Principle Techniques, Edited by John Vickerman & Ian Gilmore, Wiley 2009.

## Technical Specifications:



**Make: Kratos Analytical, UK (SHIMADZU group)**

**Model: AXIS Supra**

- **Two chamber ultra high vacuum system: Analysis chamber ( $< 2.0 \times 10^{-9}$  Torr) and Sample load-lock chamber ( $< 5.0 \times 10^{-8}$  Torr)**
- **Automated sample transfer mechanism and five-axis sample manipulator.**
- **XPS source:** Monochromatic (AlK $\alpha$ ) 600 W X-ray source; 1486.6 eV
- **UPS source:** High photon flux He gas discharge lamp (He-I 21.2 eV and He-II at 40.8 eV)
- **Auger:** Field emission gun for Auger electron spectroscopy and Scanning Auger microscopy. Source energy range up to 10 kV
- **Electron energy analyser and detector:** Concentric hemispherical analyzer (CHA) for spectroscopy, and Spherical mirror analyzer for parallel imaging. Multichannel plates stack with delay-line detector for counting. Spectroscopy, snapshot and imaging modes with option of large area and small area analysis.
- **Differentially pumped ion gun:** High performance gun for precision depth profiling, Energy range 500 eV to 4 keV for sample etching (cleaning) and depth profiling
- **Sample cleaning ion gun:** Broad beam sputter ion gun in sample entry chamber. Energy ranges 300 eV to 3 keV.
- **Charge neutralizer:** For non-conducting samples
- **Sample heating & cooling:** 500 °C to -100 °C
- **Software system:** Windows based software (ESCApe) for automated sample transfer, data acquisition and data processing. Site license for data processing.

- **Capabilities:** Under ideal sample preparation conditions the following can be achieved  
XPS: energy resolution is ~0.5 eV, & 0.7 eV in case of non-conducting samples, XPS-imaging spatial resolution is ~ 1  $\mu\text{m}$   
UPS: energy resolution is ~ 0.12 eV.  
Auger elemental mapping spatial resolution: ~ 0.1  $\mu\text{m}$
- Typically analysis depth is ~ 5 nm for metals/semiconductors and ~ 10 nm for polymers and is dependent on the material and photoemission angle. Typical emission angle is 0 degree (Analyzer is normal to sample surface)

#### Special Features:

- Parallel 2D- XPS imaging and mapping (Spatial resolution ~ 1  $\mu\text{m}$ )
- Depth profiling (Zalar rotation is also available)
- Angle dependent spectroscopy (Compucentric)
- Scanning Auger microscopy

#### Applications:

- Elemental composition of surface and quantification of their relative concentration
- Chemical states of elements
- Relative quantification of each chemical state of each element
- Thickness of thin films
- Depth profiling
- Spatial distribution of material and chemical state

### Sample Preparation and Submission:

#### Instructions for sample preparation/submission

- Maximum size of samples should be (10 x 10 x 5) mm. Smaller samples of the size 8 mm x 5 mm or 5 mm diameter are more suitable. Bigger or smaller samples than the size mentioned here may be possible and user is required to check with lab staff.
- Label the samples only on the containers. Sample labels/codes should be brief (less than three letters/numbers) or may use just single digits (1,2,3..)
- Keep the samples clean. Try not to touch the surface of your sample with anything (fingers, gloves, breath etc.)
- The sample should be completely dry and it should be stable in ultra high vacuum (no out-gassing allowed).
- Powders should be made into pellet or form a thin coating on a clean conducting substrate like silicon, aluminum or copper foil. Pressing powder in to indium foil is also another method. User should clearly mention if the sample needs etching (surface cleaning) before recording the data. It may be noted that the etching may alter the chemical composition and chemical state to some extent.

- Samples containing organic molecules /polymers and some high vapour pressure elements such as Na, K, S, P, Zn, Se, As, I, Te or Hg are not suitable for depth profile analysis.
- UPS and Auger is possible only on sufficiently conducting samples. Insulating samples in the form of very thin films on the conducting substrate may work.

#### **Instructions for Users**

- Registered user will be allotted time as per queue.
- User should submit the samples to the ESCA lab between 9:30 AM and 11:00 AM on the scheduled day.
- Samples will be loaded in to the system on the submitted day and analysis will be carried out after 4 to 6 hours or on the next day. Data will be sent through email.

#### **Instructions for registration**

- Internal users can register online.
- External users need to down load registration form and submit it to lab along with the requisite demand draft. The samples should be submitted personally on the day of allotted time slot. Analysis will be performed on the next day and results will be sent through email.
- Getting Back the samples: Users desiring to get back the samples need to mark on the form. Collect them from the lab between 9:30 AM to 11:00 AM after two-three days of completion of the work (intimated through mail) and uncollected samples will be disposed-off after ten days of intimation of the completion of the work and no reminders will be sent.