EnviroESCA™

ELECTRON SPECTROSCOPY FOR CHEMICAL ANALYSIS
UNDER ENVIRONMENTAL CONDITIONS

KEY FEATURES

- Fast Quality Control
- High Throughput Analysis
- Controllable Atmosphere
- Revolutionary Technology
- Ergonomic all-in-one Design
- Fully Software Controlled

SPECS™

SPECS™

INNOVATION IN SURFACE SPECTROSCOPY AND MICROSCOPY SYSTEMS

SPECS leads the way in developing cutting-edge components and systems for groundbreaking new surface analysis tools.

SPECS Surface Nano Analysis GmbH

SPECS Surface Nano Analysis GmbH headquarters is situated in the center of Germany's capital Berlin with subsidiaries in Switzerland, USA and China. SPECS has attracted a talented team of scientists and engineers who have dedicated their knowledge and experience to the development, design, and production of instruments for surface science, materials research, and nanotechnology

Mounting of EnviroESCA component for final testing

SPECS engineer during system assembly

for almost 30 years. In order to continuously improve performance and keep abreast of the latest developments, we are in contact with numerous scientists, users and customers from all over the world. Reliable quality control (ISO 9001 certified) and excellent fast service, both remote and on-site, ensures maximum uptime and long-term operation and reliability of SPECS instruments over many years.

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A new dimension in chemical surface analysis

Electron Spectroscopy for Chemical Analysis – Past to Present

In 1905 Albert Einstein received the Nobel Prize in Physics for his quantum mechanical interpretation of the photoelectric effect. Based on the results of Heinrich Hertz and Max Planck about the nature of light being an electromagnetic wave and about the general existence of discrete energy portions, nowadays named “quantum”, this has been a big step for basic science. At this time nobody knew, that this will evolve into the most important method for non-destructive surface chemical analysis. To reach this understanding the development of energy dispersive electron analyzers had been necessary.

Thus it took several decades until Kai Siegbahn developed and experimentally realized the first experiment of this kind in the late 1960s, again resulting in a Nobel Prize in Physics. By excitation of electrons from solid samples using characteristic X-rays and detecting the number of photoelectrons in dependence of their kinetic energies it became possible to use the element-specific electron energies to derive the chemical composition of sample surfaces without destroying them. He named the method Electron Spectroscopy for Chemical Analysis, or in short ESCA.

The global success of X-ray Photoelectron Spectroscopy (XPS) is a result of the development of methods for reliable and precise quantification of ESCA data with an elemental detection limit of <1% in the uppermost surface layers. Already in the early 1970s Kai Siegbahn realized, that the Ultra-High Vacuum (UHV) environment necessary in conventional ESCA machines is limiting the applications of this method to solid sample surfaces. So he suggested applying ESCA to liquids, using a differential pumping setup for the analyzer and X-ray source. He was able to reach a maximum pressure of 10^{-2} mbar at that time.

Again it took almost three decades in experimental development to reach pressures of up to 1 mbar in synchrotron experiments. Near Ambient Pressure XPS (NAP-XPS) was born, yielding fundamental insight in the operation of catalysts and the analysis of liquids and liquid-solid interfaces. State-of-the-art instrumentation for NAP-XPS allows for purely laboratory-based NAP-XPS systems as the use of synchrotron radiation is not mandatory anymore.

It is time for the next step in evolution.

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The Beginning of a New Era

Building on our pioneering developments of recent years SPECS proudly presents EnviroESCA. This novel and smart analysis tool overcomes the barriers of standard XPS systems by enabling analyses at pressures far above UHV. EnviroESCA is designed for high-throughput analysis and opens up new applications in the fields of medical technology, biotechnology and the life sciences. It offers the shortest loading-to-measurement time on samples of all types including liquids, tissue, plastics and foils, powders, soil, zeolites, rocks, minerals and ceramics.

Environmental

- Controllable atmosphere from sample loading to analysis
- Adaptable process gas dosing systems
- Specialized sample environments
- Compatible with all kinds of samples and sizes up to Ø 60 mm and 40 mm in height

Integrated

- Ergonomic all-in-one design
- Quick installation and setup
- Minimized downtime
- Cost and time efficient servicing
- Easy consumable replacement

Networking

- SampleExplorer
- SmartDock
- AutoLoader
- GloveBox

Reliable

- SampleJournal for complete documentation
- Reproducible analysis recipes
- Comprehensive system parameter logging
- Uptime focused user support

Versatile

- Revolutionary analyzer technology
- µ-Focus X-ray source
- High resolution XPS
- Automated charge compensation
- Sputter depth profiling

Optimized

- Application oriented software package
- Fully remote operation
- Automated vacuum system
- Simple sample loading
- Optical 3D sample navigation

Environmental

EnviroESCA - CHEMICAL SURFACE ANALYSIS UNDER ENVIRONMENTAL CONDITIONS

Key Applications for EnviroESCA

XPS is established as a powerful and wide-ranging non-destructive analytical method. In particular, the precise and reproducible quantification of trace signals which it affords, has helped to answer important questions in fundamental and applied science. EnviroESCA enhances the significance of the results in many conventional applications and expands the technique's horizons.

Astrochemistry and Astrobiology

The interaction of organic molecules with water and ice surfaces in atmospheres that can be found on distant planets is a vital field of research. EnviroESCA can create sample environments that realistically simulate conditions in planetary atmospheres such as on Mars, where the pressure ranges from 10^{-6} mbar to 7 mbar.

Liquids

Water and aqueous reagents are essential in any biological process or system. But apart from a few special low vapor-pressure cases, liquids have not been accessible to any technique requiring UHV conditions. EnviroESCA opens up this exciting field of applications.

Biological Materials

With the capability of operating in the near ambient pressure regime EnviroESCA offers an entirely new opportunity to investigate biological materials and processes, making ESCA more versatile than ever before.

Gaseous and Liquid Environments

The interaction of gases and liquids with surfaces plays a key role in many different fields ranging from biological and catalytic systems to construction materials. EnviroESCA offers the possibility of investigating surfaces in contact with gases and liquids, such as salt water, acidic rain, wastewater, or gaseous atmospheres with high humidity.

Soils and Minerals

XPS analysis is widely used in soil and mineral research for characterizing surface organic films, mineral decomposition and redox transformations. Until now these studies were limited to UHV compatible samples. EnviroESCA overcomes this constraint and offers new exciting possibilities.

Fabrics

The performance of highly sophisticated fabrics is governed by the interaction of the interface with the surrounding atmosphere. By studying the surface properties of the fibers in wet air, deeper insights into relevant processes under more realistic conditions can be gained.

Medical and Biomaterials

Medical implants are devices or tissues that are placed inside or on the surface of the body. A widely used material for surgical implants is Titanium. It is important to be able to analyze the Ti surface to achieve optimized interactions with the surrounding tissue.

Energy Materials and Devices

Batteries and fuel cells are devices that use chemical reactions to store and convert energy. Fuel cells for instance consume fuel and oxidants during operation so that their investigation is not possible in traditional XPS spectrometers. With EnviroESCA the fundamental steps in such devices can be investigated *in operando*.

Catalysts

The function and efficiency of a catalyst is principally determined by its surface properties. XPS and NAP-XPS are proven and powerful tools for investigating catalytic behaviour in studies ranging from model systems to real world materials.

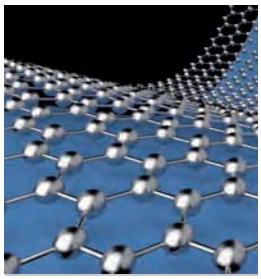
Polymers and Plastics

Polymers and plastics are used in many fields such as food grade packaging and medical technology. Their composition is especially important when the polymers get in direct contact with food or humans. With EnviroESCA the concentration of hazardous contaminations can be quantified regardless of their vacuum compatibility.

Coatings and Thin Films

Coatings and platings are widely used in optics manufacturing and metal refinement as they optimize the surface properties of materials to make them harder, stronger and more durable. The coatings are thin films which interact with the substrate on one side and the gaseous or liquid environment on the other.

Nanomaterials



Nanomaterials have attracted a lot of attention from research and industry in the past decades. Questions about the influence of the surrounding atmosphere on the chemical composition and potential core-shell structure are ideally addressed by EnviroESCA.

Microelectronics and Semiconductors

The quality of conductive platings on printed circuit boards is crucial for the operation of any microelectronic device. With the AutoLoader this high performance tool is ideally suited for unattended and automated analysis of microelectronic devices.

Corrosion

The reliability of mechanical or electrical connections depends strongly on their chemical composition and degree of corrosion. EnviroESCA facilitates the investigation of the metal surface in interaction with its surrounding gas or liquid phase environment to gain a detailed understanding of processes governing corrosion.

Magnetic Media

The chemical composition of materials from magnetic media can precisely be determined despite their magnetization. Due to the abscence of artificial magnetic fields in the measurement position magnetic samples can be measured without affecting their properties.

Metals

Metallic parts can rapidly be analyzed without pre-cleaning or surface preparation. With the unrivaled short sample loading time and the robotic AutoLoader they can be taken directly from the production line for quality control.

Versatile

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EnviroESCA features a unique modular approach to the system design, combining the advantages of several specialized ESCA systems in a single entity. It is adaptable to suit different experimental conditions through the use of dedicated interchangeable sample environment modules. As in any conventional ESCA instrument the electron energy analyzer and the X-ray source are the key components of EnviroESCA. But combined with SPECS advanced, automated vacuum and gas-handling systems, reliable working conditions in pressure ranges from ultra-high vacuum to hundreds of mbar are now possible. This combination enables the user to investigate even the most challenging samples of a class that was incompatible with traditional ESCA equipment.

Electron Energy Analyzer

- Hemispherical analyzer
- Specialized for environmental applications
- Delayline electron detector with up to 400 channels

Ion Source

- Scannable small spot ion source or optional gas cluster ion source

Charge Compensation

- Low energy electron source
- Specialized for environmental applications

Microscopes

- Digital microscopes for easy sample positioning and documentation.

X-ray Source

- Al K_α micro-focused monochromator
- Variable spot sizes tailored to experimental requirements

Analysis Compartment

- Minimized chamber volume
- stainless steel

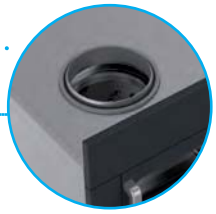
The unique sample environment in EnviroESCA makes it ideally suited for characterization of materials that may not be UHV compatible: The stringent UHV requirement of conventional XPS is dispensed with in EnviroESCA. Dedicated sample environment modules are provided as smart units for different classes of samples and applications. The modules are equipped with all relevant components such as sample stage, plasma cleaning and gas handling. Their exchange can be readily accomplished in just a few minutes.

Digital Microscope

- Video recordings and still photos of the sample during pump down or pretreatment cycles

Quick Connector

- Gas tight seal between sample environment and analysis compartment

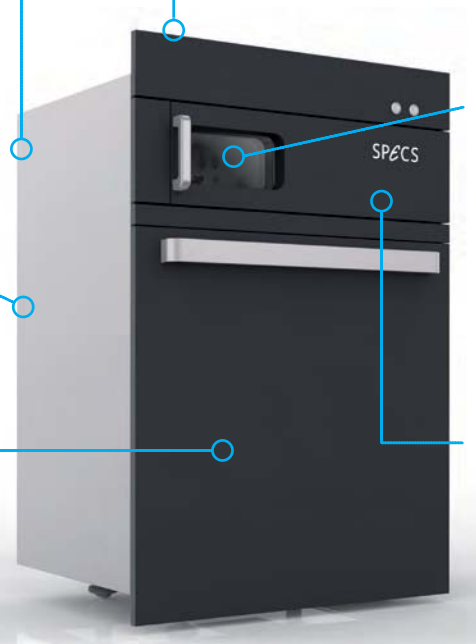


Pumping System

- Fully computer controlled integrated pumping stages

Gauges

- Full range gauges from ambient to ultra high vacuum conditions
- Gas type independent capicitance gauges



SmartDock

- Sliding door mechanism for manual sample introduction
- Allows mounting of sample containers under vacuum or with inert gas filling

Plasma Cleaner

- RF plasma cleaner for sample and equipment cleaning

Sample Stage

- Fully motorized sample stage for precise positioning of the sample in front of the analyzer and X-ray source
- Samples can be of any shape within a volume of Ø 60 mm and 40 mm in height

Sample Connections

- Spare ports for electrical, gas or liquid feedthroughs to connect the sample with the outside
- Examples are thermocouple contacts, multipin feedthroughs for Peltier cooling or heating stages, USB, fiber optics and many more

Integrated

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The compact all-in-one design of EnviroESCA includes all the necessary equipment, from power supplies to closed-cycle water cooling, within a single small-footprint unit. Located centrally on the front face is the sample loading bay where samples are introduced into the Sample Environment manually or automatically via the robotic AutoLoader.

Analysis Module

- Excitation sources
- Electron analyzer
- Gas dosing

Status Indicator

- Integrated design

Service Accesses

- Ergonomically positioned
- Monitored access

Sample Loading Bay

- Quick access door
- SmartDock
- Safety glass

Dimensions

- Footprint 2.1 m x 1.7 m
- Height 2.7 m
- Weight 1800 kg

Sample Environment

- Precision sample stage
- RF plasma cleaner

Noise-damped compartments enclose the high-specification vacuum system ensuring that EnviroESCA can be installed with ease in sensitive and space-constrained laboratories. Convenient service access facilitates fast and simple maintenance and consumable replacement. All connections to the laboratory infrastructure are located at a central position.

Thermal Management

- Forced air and convection cooling

Vacuum System

- Integrated design
- Fully computer controlled
- Integrated pumping stages

Electronics

- Integrated cabinets
- Measurement and automation PC

Gas Handling

- Specialized gas dosing units

Cooling System

- Closed-cycle water cooling

Connectivity

- Mains
- Purge and venting gas
- Process gases
- Pressurized air

Service Access

- Accessible machine interior
- Monitored access

Reliable & Optimized

EnviroESCA - CHEMICAL SURFACE ANALYSIS UNDER ENVIRONMENTAL CONDITIONS

Automation

The advanced software control system enables fully automated sample analysis to be carried out. It controls the vacuum system, the gas handling system and all analysis components. Even operation from mobile devices is possible.

Interactive remote control from mobile devices

In addition, the optional robotic AutoLoader can be used for completely unattended introduction and processing of sample batches. Thus high-throughput analysis of stored samples or parts taken from production lines can be realized in a unique way. Manual access to all procedures is nevertheless available to experienced users.

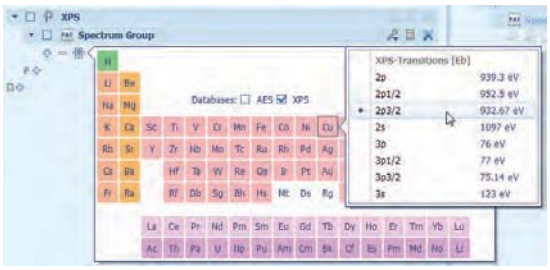
Vacuum Control Expert View

Data Logging

All system parameters including temperature of system components, pressure readings, and equipment health status are permanently recorded. Availability of this database makes for time-efficient service and maintenance.

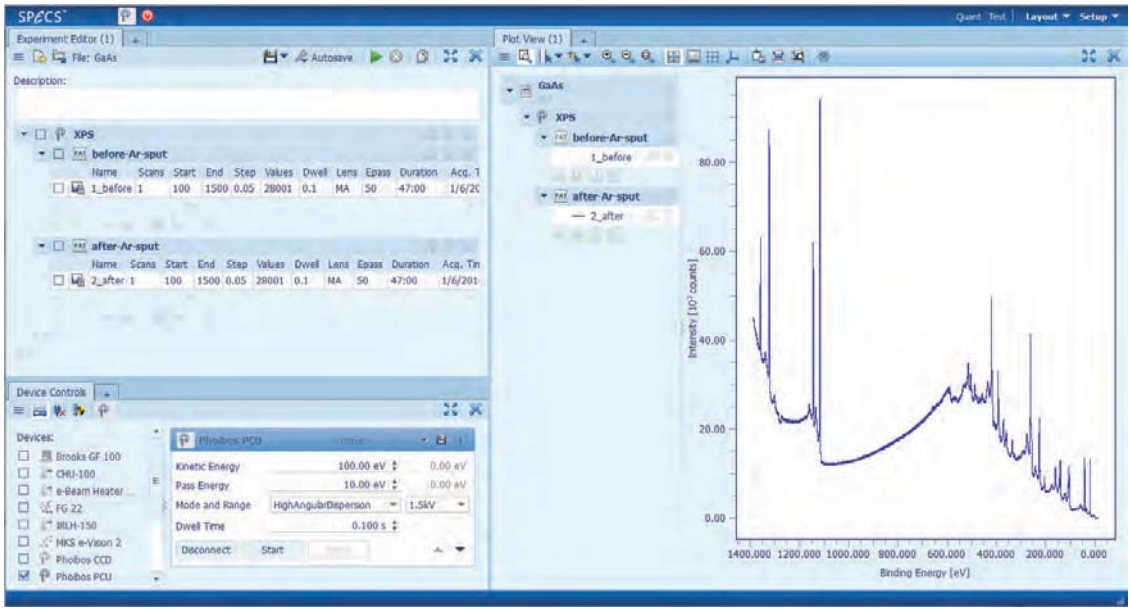
Experiment Recipes

Customized measurement recipes simplify the workflow of complex experimental procedures. The SampleExplorer is a fully integrated but stand-alone tool to define experiments in advance for most efficient use of the measurement time. Software tools such as a periodic library are of course included to assist less experienced operators.



Data Processing

Advanced curve-fitting routines used for automated peak identification and quantification are just one example of the feature-packed software package. Specific data sets are easily retrieved from the central database by using advanced search routines. Each entry can be tagged and grouped for batch data analysis.



XPS survey spectrum shown in experiment recipes editor

SampleJournal

In the SampleJournal all relevant information regarding an individual specimen is collected chronologically including high resolution sample and survey platter images, measurement data and recipes, experimental parameters and operator notes. The database entries can easily be exported for reporting, further analysis, or archiving.

Service

All system components were designed or selected for extended lifetime operation and highest reliability ensuring low cost-of-ownership. EnviroESCA offers the possibility of full remote control of the entire system. Rapid and easy maintenance reduces down-time and service costs to a minimum.

Quality control of spare parts

Networking

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SampleExplorer

Measurement time can be saved by planning experiments in advance with the stand-alone SampleExplorer. An unlimited number of measurement positions and tasks can be defined for batch processing before inserting the sample into EnviroESCA. A high precision sample stage is embedded in a geometrical mockup of the analysis setting. Equipped with three high resolution cameras it enables documentation of the analysis area. For each measurement position a “through the lens” view with an optical resolution well below 10 µm and a wide angle view of the analysis position are recorded in conjunction with a survey view of the platter.

SmartDock

All samples are introduced to the sample environment modules via SmartDock. It consists of a sliding door mechanism for manual sample loading with a connection system for easy docking of sample containers. The sample containers allow transportation of samples prepared *ex situ* under vacuum or gas atmospheres. While docked, sample containers are supplied with power, gases and pressurized air for valves or actuators. The containers are fully integrated into the EnviroESCA device network for remote control or automation. The SmartDock can also be exchanged with a glove box docking system.

AutoLoader

The AutoLoader uses a collaborative industrial robot in combination with a sample storage unit enabling high throughput sample analysis. Multiple sample storage units can be used and each of them can be replaced on the fly without interfering with the robot’s movements.

Technical Data

Summary

EnviroESCA	
Electron Spectrometer	• Hemispherical electron analyzer with 150 mm mean radius • Differentially pumped lens system • Delayline detector with up to 400 channels
X-ray Source	• Al K_α micro-focused monochromator • Rowland circle diameter of 600 mm • Spot sizes of 200 µm – 1 mm optimized to analysis area
Charge Neutralization	• Differentially pumped low energy electron source
Ion Source	• Scannable small spot ion source (200 eV – 5 keV) • Gas cluster ion source
Pumping System	• Turbomolecular pumps • Oil-free backing pumps • Chemical resistant pumping system on request
Pressure Range	• Defined by analyzer aperture (up to 100 mbar with an aperture of 300 µm; other aperture sizes on request)
Gas Dosing System	• Two separated gas dosers at analysis position • Mass flow controllers
Cameras	• 3 digital microscopes for sample navigation and documentation
Automation and Software	• Fully automated vacuum and gas dosing system • Advanced software package

Sample Environment (standard, others on request)	
Sample Stage	• High precision 3-axis stage
Sample Size	• Up to Ø 60 mm and 40 mm in height
Gas Dosing	• Connections for process and purge gases
Cleaning	• Downstream RF plasma cleaner
Camera	• Digital microscope for sample observation and documentation
Spare Ports	• 2 x DN40CF (2,75") e.g. for electric or liquid feedthroughs

Dimensions

SPECS Surface Nano Analysis GmbH
Voltastrasse 5
13355 Berlin / Germany
www.EnviroESCA.com

T +49 30 46 78 24-0
F +49 30 46 42 083
E info@specs.com

Distributor

株式会社 東京インスツルメンツ
TOKYO INSTRUMENTS, INC.
MUTUAL SATISFACTION

Head Office 6-18-14 Nishikasai Edogawa-ku, Tokyo 134-0088
Phone +81-3-3686-4711 Fax +81-3-3686-0831

Osaka Office 4-1-46 Miyahara Yodogawa-ku, Osaka 532-0003
Phone +81-6-6393-7411 Fax +81-6-6393-7055

http://www.tokyoinst.co.jp/

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