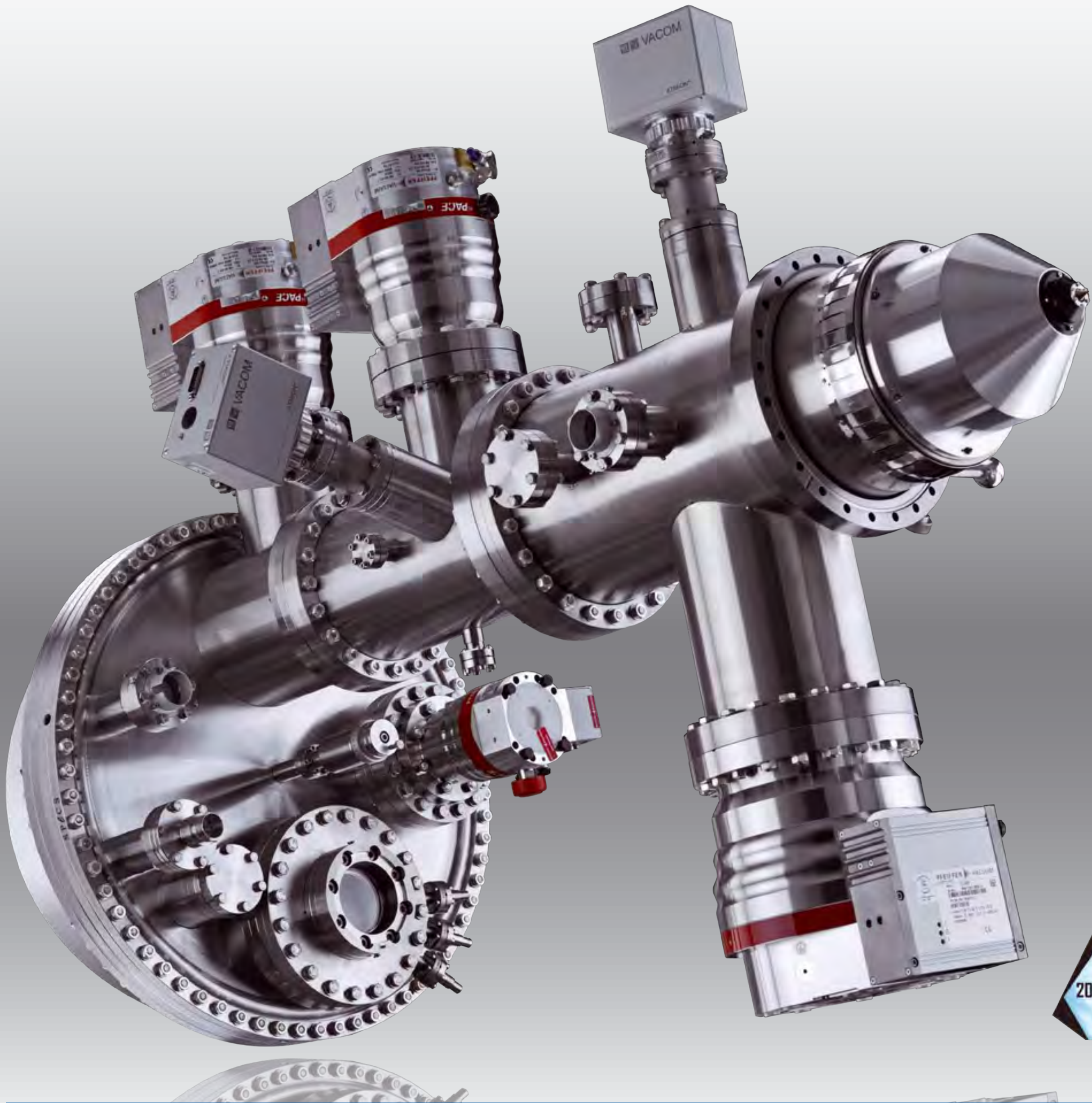
Components for Surface Analysis

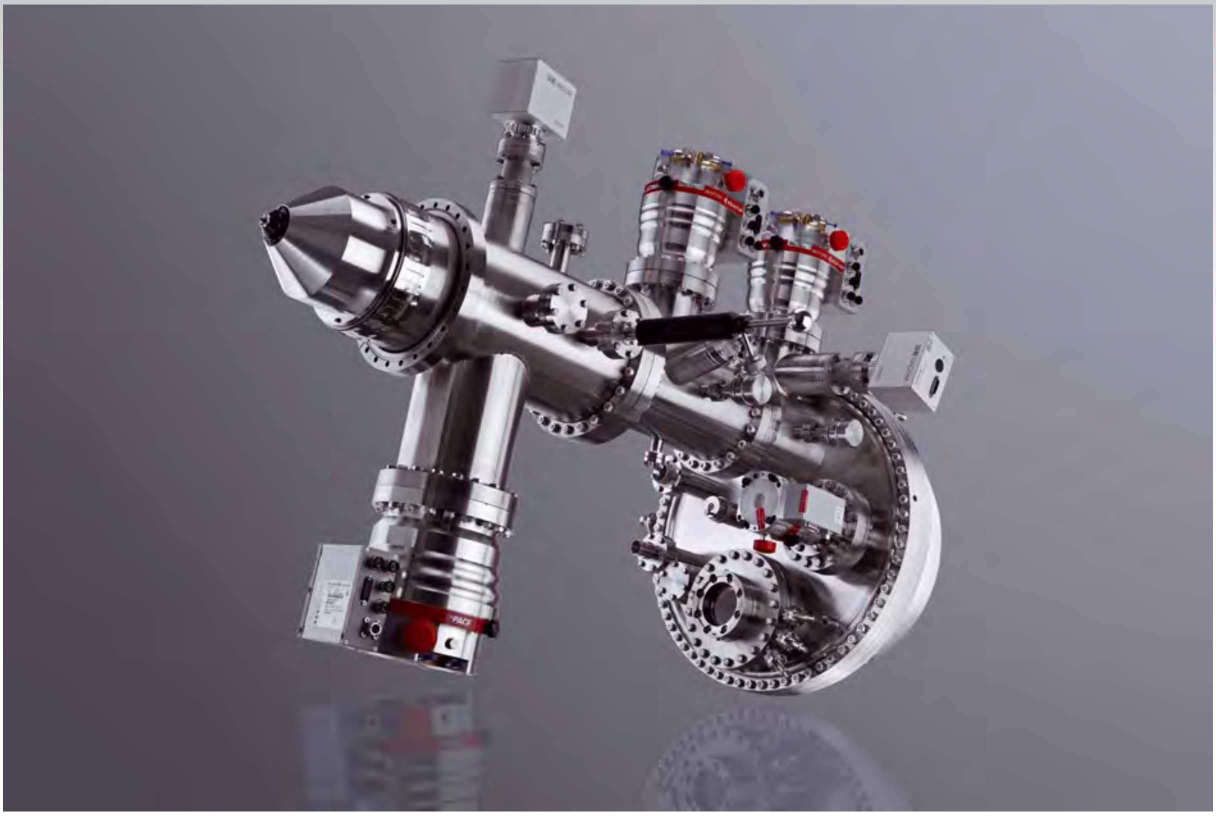
PHOIBOS 150 NAP

Near Ambient Pressure Hemispherical Energy Analyzer

- Wide Angle Pre-Lens with 44 ° Acceptance Angle
- Near Ambient Working Pressures up to 25 mbar
- Large Kinetic and Pass Energy Range
- High Energy and Angular Resolution



R&D 100 2010


SPECS has received an R&D 100 Award 2010 for the near ambient pressure photoelectron spectrometer PHOIBOS 150 NAP. Awarded by an independent judging panel and the editors of R&D Magazine, it recognizes the 100 most technologically significant products introduced during the past year.

Photoelectron Spectroscopy at Near Ambient Pressure Conditions

The increasing importance of surfaces and interfaces for all kinds of devices have helped X-ray Photoelectron Spectroscopy (XPS) to become a standard characterization method not only in basic research, but also in chemistry, materials science and analytical facilities. Nowadays XPS systems and components have reached such levels of performance, that most analytical questions can be answered within reasonable acquisition times. When analyzing properties of devices that function in ambient conditions, high pressures or liquid environments, the significance of the results has always been controversial due to the typical vacuum environment in XPS. For years this pressure gap has been an insurmountable barrier.

Based on an earlier instrument a new conceptual design of a near ambient pressure analyzer has developed in a collaboration between Advanced Light Source (Berkley, USA), Fritz-Haber Institut (Berlin, Germany) and SPECS. The new instrument, PHOIBOS 150 NAP, uses a differentially pumped electrostatic pre-lens based on the design of the wide-angle lens of the new PHOIBOS WAL for highest transmission.

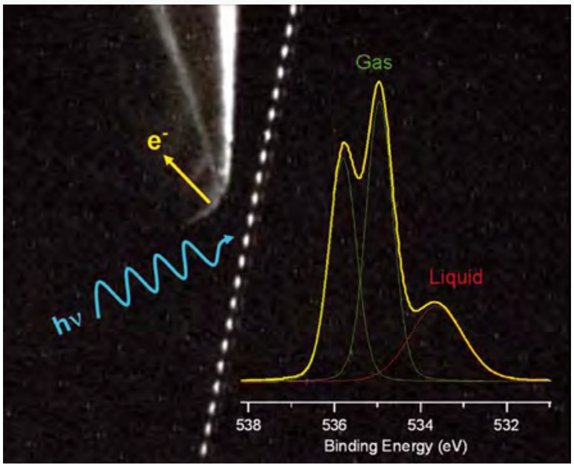
Simpler operation and new system concepts for synchrotron or laboratory based system solutions open the research fields to a wider group of researchers.

"In order to understand real world chemical processes, we need to analyze them as they occur in the real world."
Miquel Salmeron, Berkeley Lab

Applications

Driven by the challenge of modern energy production, chemistry and environmental research, the upcoming fields for in situ analyses are catalysis, electrochemistry and the study of liquids and their interfaces to solid materials. Although relatively new, surface analysis in near ambient pressure conditions has already led to groundbreaking discoveries in the fields of climate change, atmospheric science, nanotechnology and industrial processes like heterogeneous catalysis. Researchers worldwide have started to study the impact of chemicals on agricultural plants and human health as well as the pollution of air, water and other environmental systems.

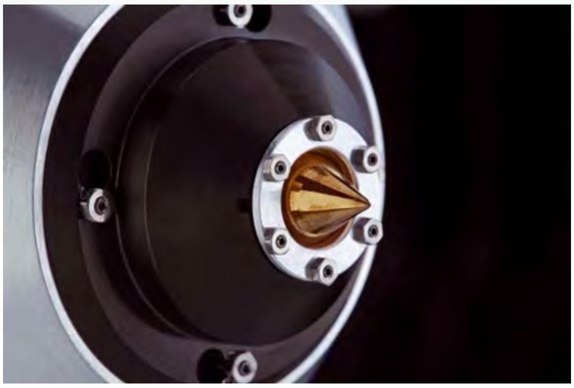
As a key component for these studies, the PHOIBOS 150 NAP makes it possible to use XPS under pressures and humidities similar to those encountered in natural environments. In this way, it is possible to observe chemical interactions on the atomic level for gas/liquid and liquid/solid interfaces at near ambient pressures. In the following, two results are presented, showing the application of near ambient pressure XPS with PHOIBOS 150 NAP to a pure liquid sample and a liquid/solid interface.



Stroboscopic photograph of a droplet jet prepared from a 40% ethanol solution in front of the entrance aperture (diameter 0.3 mm) of the differentially pumped lens system. The O 1s XP spectrum of the methanol solution droplets is recorded with incident photon energy of 938 eV. Both gas phase and liquid phase photoemission peaks are visible in the spectrum. Data courtesy of David E. Starr et al., published in Phys. Chem. Chem. Phys. 2008.

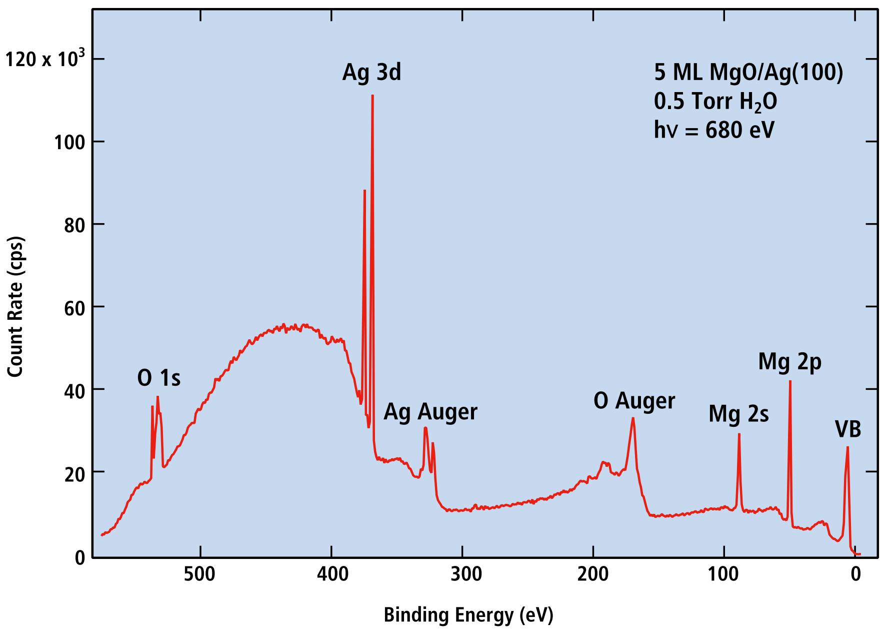
Electron Spectrometer Design

The transfer optics are a crucial part of the NAP electron spectrometer design. The first aperture, or nozzle, is shaped conically with a half angle of 35 °. This aperture, with a diameter of down to 0.3 mm, maximizes the differential pumping and brings the pressure down to the 10^{-3} mbar range a few millimeter, from the sample. This cone is detachable and can be exchanged by similar cones with different aperture diameters.



Exchangeable conic aperture separating the high pressure chamber from the first pumping stage. The diameter is 1 mm or less.

Behind the nozzle, a wide-angle deceleration lens using a focussing mesh with high transparency creates images of the sample region that is analyzed at the entrance aperture of the second lens stage with negligible spherical aberration. A quadrupole lens guides the beam to the entrance of a PHOIBOS 150 hemispherical analyzer connected the focussing lens system.



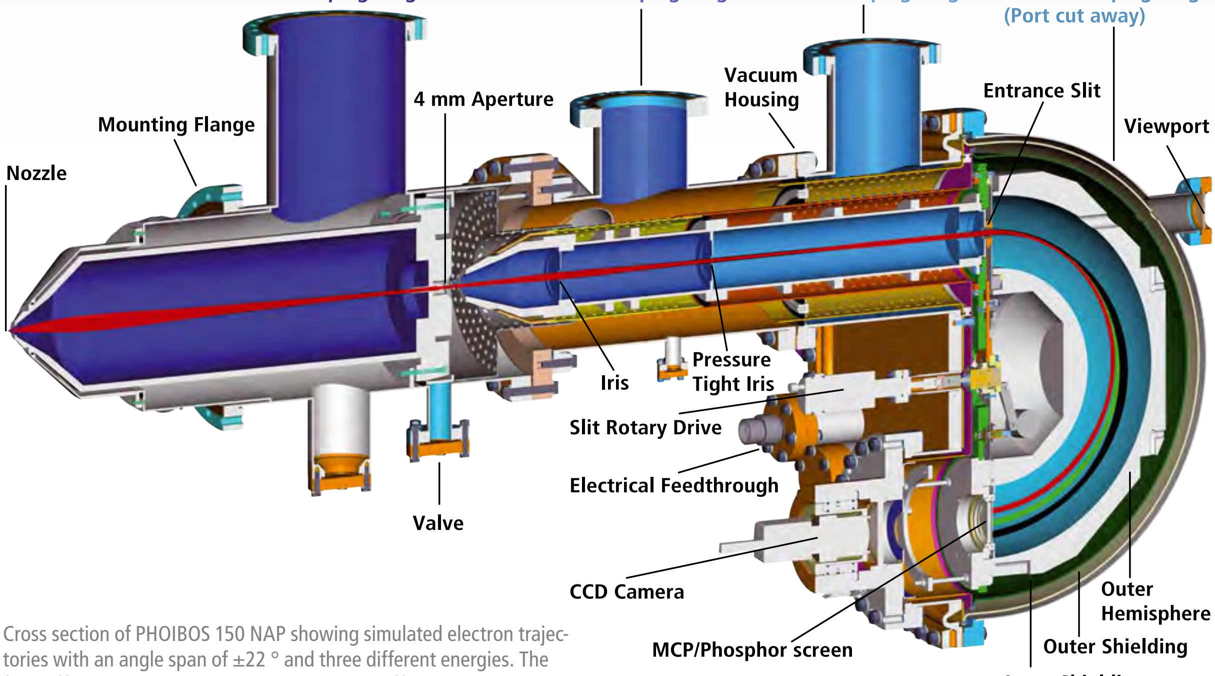
Formation of hydroxyl and water layers on MgO films studied with ambient pressure XPS. Data courtesy of J.T. Newberg et al., accepted for publication in Surf. Sci. (2010).

The analyzer is a true 180 ° hemispherical energy analyzer with 150 mm mean radius. The analyzer, the lens system, and the detector are surrounded by two layers of 2 mm thick μ-metal to screen external magnetic fields down to an uncritical level. For ultimate performance, the analyzer and the lens system are constructed entirely from non magnetic materials inside the μ-metal shielding.

Differential Pumping System

The PHOIBOS 150 NAP consists of a differentially pumped electrostatic pre-lens, with a three-stage differentially pumped PHOIBOS 150 analyzer. Thus, the design concept provides four separate pressure stages separated by apertures. The first pumping stage (pre-lens) is separated from the analytic chamber by a nozzle with an 0.3 mm hole at the tip. By using a turbopump with a pump speed of 500 l/s on the pre-lens stage, a pressure difference of four orders of magnitude (compared to the analytic chamber) is possible.

The first and second stages are separated by an aperture with a 4 mm diameter. A valve allows a high-vacuum seal between the stages. This second stage represents the first part of the electrostatic lenses of the PHOIBOS. The front and rear parts of the electrostatic lenses are separated by an iris aperture. The third pumping stage is situated behind the iris. The fourth and final pumping stage contains the hemispheres and detector.



Cross section of PHOIBOS 150 NAP showing simulated electron trajectories with an angle span of ±22 ° and three different energies. The four differential pumping zones are colored in different blue shades.

Detectors

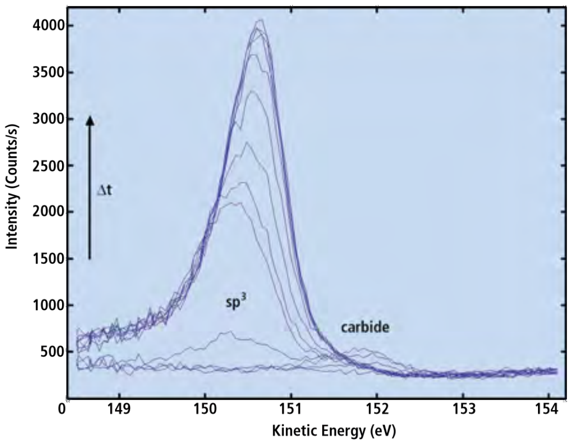
The analyzer with its modular detector concept can be equipped with different detectors according to the customers' needs. The standard detector assembly consists of nine channeltron detectors (MCD-9) arranged as a single block which provides both compactness and durability. Channel electron multipliers with an extended dynamic range for extremely high count rate applications are used as standard. The design of the detection electronics takes into account the need for reliable counting results even in difficult environments and for extended dynamic ranges.

MCD-9 Detector.

The one dimensional delay line detector 1D-DLD for the PHOIBOS NAP combines a convenient number of parallel channels for fast snapshot operations (100 or 200). It features highest count rates at excellent energy resolution in scanned mode, true count rate detection, and a high dynamic range, all available at an attractive price.

1D-DLD Detector.

Real time monitoring of processes becomes possible. This is documented in the following results of an in situ growing C film on $Ni/SiO_2/Si$ substrate in 1mbar C_2H_2 for a sample temperature of 500 °C.



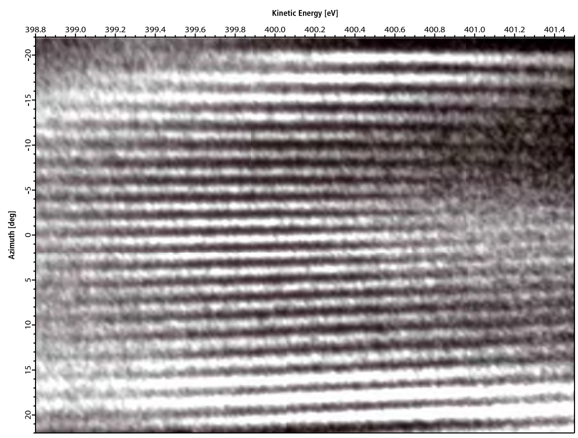
In situ growth of C film on $Ni/SiO_2/Si$. Data recorded at ISISS beamline at BESSY II, photon energy 435 eV. Total acquisition time per spectrum $\Delta t = 2.4$ s. Data courtesy of Stephan Hoffmann, Univ. of Cambridge, UK.

The 2D detector system (CCD or delay line detector) uses both the energy and angular resolution for angular resolved measurements. The CCD system features a 12-bit digital CCD camera with high dynamic range. The detector design is especially optimized for the detection of low kinetic energy electrons. A 3D (one time and two lateral dimensions) delay line detector system can be installed. The design combines high count rates with extremely high temporal resolution in one device.

3D-DLD Detector.

2D-CCD Detector.

The following result shows the angular resolving power of the PHOIBOS 150 NAP analyzer. Angle resolved near ambient pressure UPS and XPS open the possibility of directly mapping the electronic structure of catalysts under relevant operating conditions.



Detector image at 400 eV kinetic energy and 30 eV pass energy. The nozzle has been removed and a slotted aperture installed instead. This aperture has a slot every 2 °. The angle resolution at the center 30% of the detector is better than 1 °.

Power Supply

The HSA 3500 plus is a versatile high voltage power supply. The modular design of the unit allows independent setting of all voltages via high-precision 24-bit digital-to-analog converters with an overall maximum settle time of 3 ms. Each module is equipped with a micro controller allowing independent setting of all voltages, including diagnosis and error localization. The complete electronic package is contained in a double 19" standard rack housing with removable cables. The power supply can be operated in FAT (Fixed Analyzer Transmission) or FRR (Fixed Retarding Ratio) mode. Both pass energy and retarding ratio can be continuously adjusted to fine-tune resolution and intensity.

With an energy span of ±3500 eV, the power supply of the PHOIBOS analyzer provides a wider energy range than most other instruments and gives access to high kinetic energy lines. For ultra high energy resolution applications, the unit can be operated in a 400 V bipolar range and a 100 V unipolar range with extremely low ripple. Step widths down to 0.125 mV are possible. These ranges guarantee extraordinary stability and low noise, allowing ultra high resolution measurements. The power supply uses the fast and reliable CAN Bus interface and an internal microprocessor for fast and reliable processing and remote control. The temperature stability of the analyzer voltage modules are better than 1.5 ppm of the voltage span per °C.

HSA 3500 plus upper: analyzer, lower: pre-lens.

Software

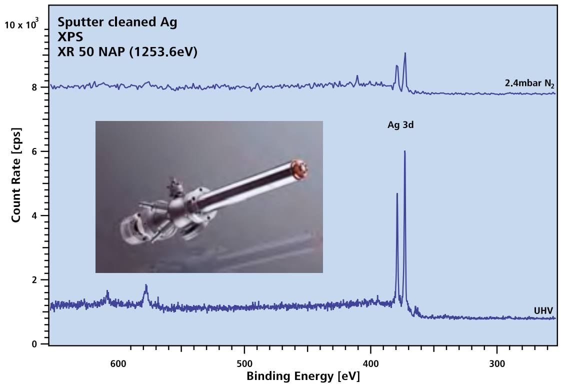
SpecsLab2 is the user friendly data acquisition software package which accompanies the PHOIBOS analyzer series. It enables data acquisition using a wide variety of operational modes. Some important features are:

- Angle integrated measurements
- Angular resolved measurements
- Lateral resolved measurements
- 2D-measurements in sweep or snapshot mode
- Batch processing of spectra
- Source manipulator and monochromator remote control for automated depth profiling

SpecsLab2 itself can be triggered from external programs via TCP/IP interface. Discuss your software integration and operation with SPECS for efficient experiments realization.

Complete System Solution

In recent years, most of near ambient pressure XPS research has been performed on synchrotron sources like BESSY II and ALS, because of their superior photon flux densities. The number of laboratory studies were limited mainly by the lack of suitable system concepts, near ambient pressure excitation sources and high transmission analyzers. Test results at 2.4 mbar demonstrate that operation with the near ambient pressure source XR 50 NAP is possible up to at least 1 mbar.

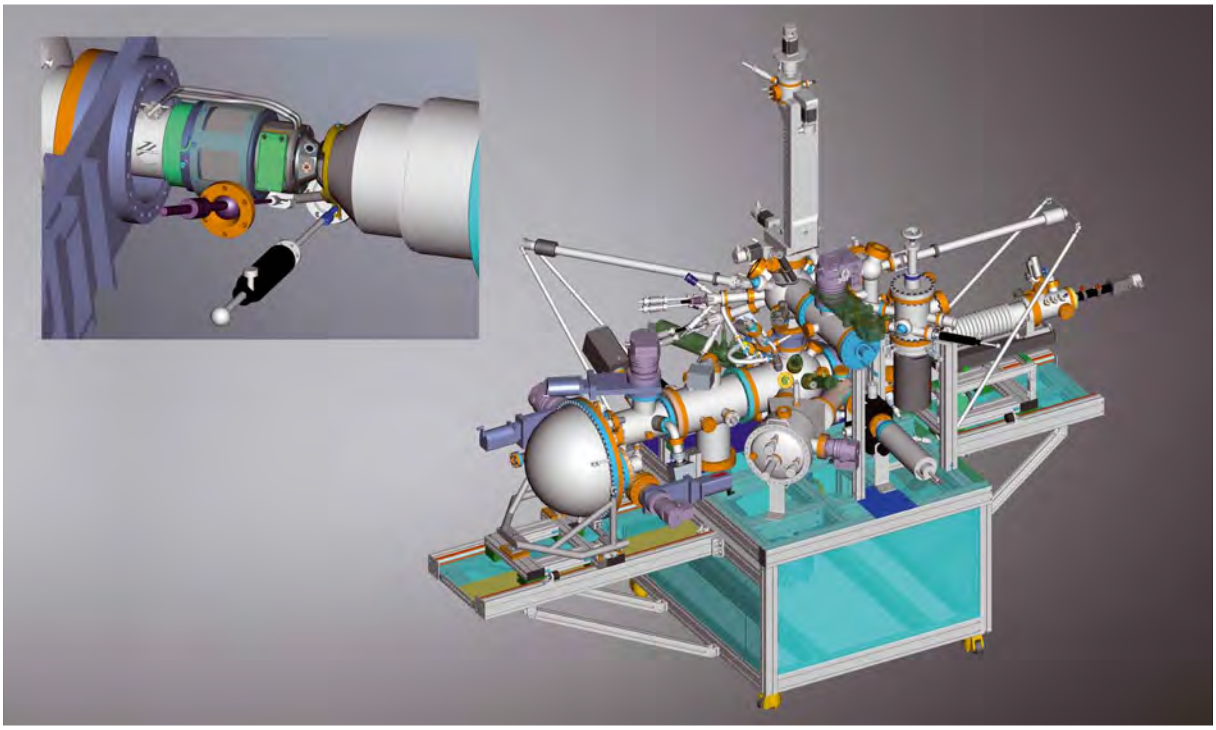


Ag XPS spectrum in UHV and 2.4 mbar N_2 pressure using a differentially pumped x-ray source XR 50 NAP.

Besides synchrotron solutions and hybrid systems SPECS now offers complete system solutions for pure laboratory applications up to 10 mbar. They mainly consist of the PHOIBOS 150 NAP analyzer, special near ambient pressure in situ cells and suitable near ambient pressure X-ray sources.

Complete hybrid analysis system concept for sychrotron and laboratory use.

A microfocus monochromator or a dual anode source is available. Such a system is shown in the next picture including an ex situ high pressure cell for operational up to 20 bar and 800 °C. The in situ near ambient pressure cell is mounted directly onto the analyzer lens, including the nozzle. X-rays are guided to the sample through a window. The maximum working pressure is about 10 mbar. Samples can be introduced into the small near ambient pressure volume through a door. The complete cell can be retracted into a dedicated load lock chamber for service and cleaning purposes. This arrangement additionally allows UHV to be performed.



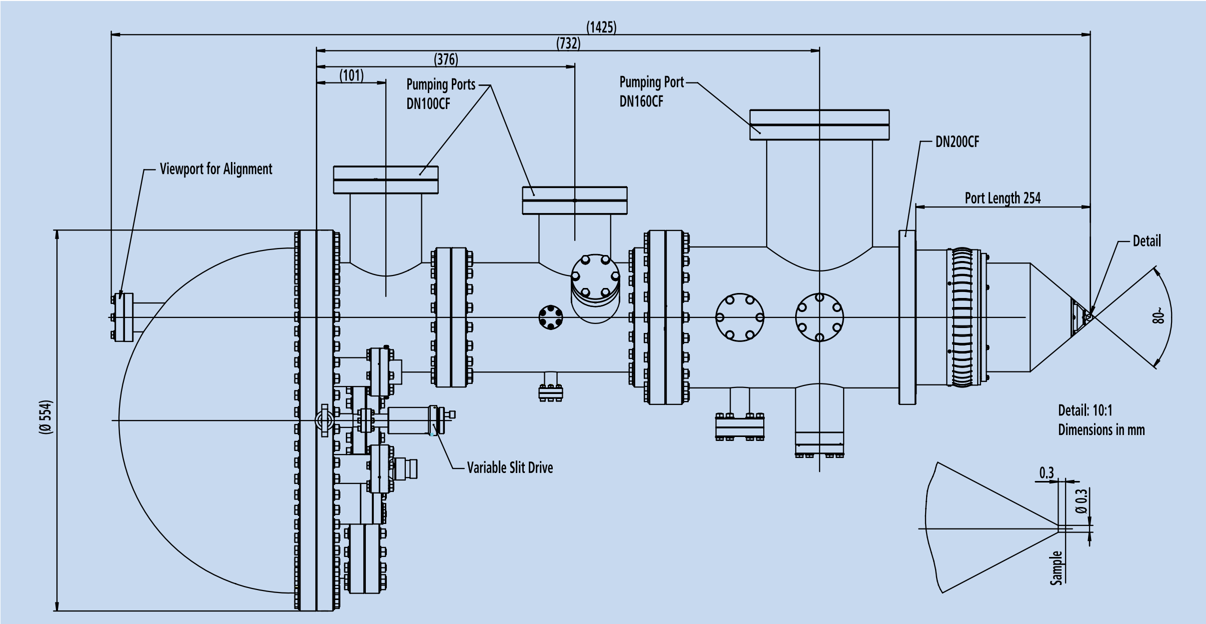
Laboratory near ambient pressure XPS system example with additional ex situ high pressure cell.

SPECS™

Technical Data

PHOIBOS 150 NAP	
Acceptance Angle	±22 °
Maximal Sample Pressure	25 mbar
Kinetic Energy Range	5 - 3500 eV
Pass Energy Range	1 - 200 eV
Ultimate Energy Resolution	< 2.5 meV
Working Distance	0.3 mm
Entrance Slits	8
Maximum Bakeout Temperature	180 °C
Mounting Flange	DN 200 CF
Shielding	Double μ-metal
Weight	ca. 200 kg

Power Supply HSA 3500 plus	
Voltage Ranges	100 V, 400 V, 1500 V, 3500 V
DAC Resolution	24 bit
Voltage Setting Accurancy	20 bit
Voltage Ripple and Noise	< 250 μV @ 400 V
Warm-up Time	30 min
Weight	Approx. 15 kg
Size	45 cm (W) x 32 cm (H) x 50 cm (D)



Printed in November 2010

SPECS Surface Nano Analysis GmbH
Voltastrasse 5
13355 Berlin
Germany

Tel. +49 30 46 78 24-0
Fax +49 30 46 42 08-3
Email support@specs.com
Web www.specs.com

ISO 9001 Certificate

Your Local Representative: