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HR EXCELLENCE IN RESEARCH

Quote for X-ray photoelectron spectroscopy (XPS) measurements

1. Introduction

The Surface and Nanostructures Laboratory at the Institute of Catalysis and Surface Chemistry (IKiFP) is equipped with a multi-chamber analytical system designed for comprehensive studies of the surfaces of solid and powdered materials under ultra-high vacuum (UHV) conditions. Thanks to a specialized distribution system, it is possible to transfer samples between the individual chambers of the system. As a result, we can create and modify the surfaces of samples and nanostructures in dedicated chambers and analyze them using spectroscopic, microscopic, and diffraction methods over a wide temperature range (from -170°C to 1700°C). The vacuum level during measurements is typically better than 5×10^{-10} mbar. The most important spectroscopic technique available in this system is X-ray photoelectron spectroscopy (XPS).

2. Technical specifications (XPS)

A hemispherical electron energy analyzer (SES R4000 by Gammadata Scienta) with high energy resolution (< 2 meV) enables high-precision XPS measurements. A non-monochromatic dual Mg/Al X-ray tube is used to generate X-rays. To optimize measurements, a 40-mm MCP (multi-channel plate) is used as the detector, together with a CCD camera. The system's energy resolution, measured as the full width at half maximum (FWHM) of the Ag $3d_{5/2}$ line, is 0.9 eV for an analyzer pass energy of 100 eV. The sample analysis area can be varied from a fraction of a mm² to over 10 mm². The sampling depth typically ranges from 7 to 12 nm, depending on the lamp used and the density of the layers being analyzed. The spectrometer is calibrated in accordance with ISO 15472:2001. Numerical analysis of the obtained XPS spectra using the latest version of CasaXPS software allows for the determination of the chemical composition and a quasi-quantitative picture of the chemical bonds on the analyzed surface with good accuracy (< 3%). The assignment of appropriate oxidation states of elements and the identification of chemical bonds are performed based on the SpecMaster Max Database and the NIST online database.

3. Scope of work:

a) Analysis of the surface layer composition of the supplied samples using a survey spectrum (spectral step no greater than 0.25 eV, spectral energy resolution no worse than 1.1 eV for the Ag $3d_{5/2}$ line, spectral energy range on the electron binding energy scale not less than 0–1100 eV, number of survey scans not less than 5). Determination of the content of elements from Li to Pu at surface concentrations >0.1% at.

b) Generation of high-resolution spectra for elements present on the analyzed surfaces (spectral step no greater than 0.05 eV, spectral energy resolution no worse than 0.9 eV for the Ag $3d_{5/2}$ line, number of spectral scans not less than 20, signal-to-background intensity ratio in the spectrum not worse than 50:1 for surface concentrations of not less than 0.1% at.).

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c) Decomposition of high-resolution spectra into components and their interpretation, specifying the bonds, functional groups, and environments of the analyzed elements in accordance with established best practices confirmed by publications. Note: If the amount of the element being tested on the surface is negligible, the spectral decomposition into individual components may be subject to significant error and prevent an unambiguous interpretation of the results. In such a case, upon agreement with the client, it may be decided to extend the measurement time, which may result in an increase in the measurement price.

d) The average time for measuring and analyzing 10 samples is 2 weeks.

4. Data delivery:

a) Final report
b) Raw spectral data and fitting data in .vms, .pxt, and/or .txt formats
c) Transfer of copyright to the interpretation of results

5. Total price for standard measurements:

net PLN 1,800/sample (1–10 samples)
net PLN 1,500/sample (more than 10 samples)

6. Notes

a. The price of a single measurement, including analysis, may increase depending on the complexity and duration of the measurement. This will be agreed upon after a pilot measurement is performed.

b. Solid samples for XPS analysis should not exceed dimensions of 20×20×5 mm^3. The optimal sample size is 10×6×1 mm^3.

c. To avoid the accumulation of large amounts of atmospheric carbon deposits on the samples, it is recommended to deliver the samples in tightly sealed (and secured with Scotch tape) glass (or plastic) containers under an inert gas atmosphere.

d. It is recommended that a series of samples be prepared for XPS measurements on the same substrate, under similar conditions, and within a short time frame to avoid the influence of undesirable environmental factors. Ideally, the delivered samples should be prepared after each stage of the surface modification process, starting with the unmodified substrate.

This proposal was prepared by Dr. Jacek Gurgul

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