

Set-up of a novel Electrochemical IR Spectroscopy Apparatus with Liquid/Solid-Interface-Preparation: ELISA I&II

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Motivation

To investigate complex model catalysts' surfaces on single crystals under ultraclean conditions the latter must be prepared in the ultrahigh vacuum (UHV). To this end we present a UHV system that allows preparation and characterization of model electrocatalysts and subsequent transfer to an electrochemical cell without contact to ambient atmosphere. The system is equipped with all standard preparations tools, electron beam evaporators, thermal evaporator cells and a quartz microbalance. Structural and chemical analysis is possible by low energy electron diffraction (LEED), Auger electron spectroscopy (AES) and temperature programmed desorption (TPD). The sample single crystal is transferred through a differentially pumped stage into the electrolyte without breaking the UHV in the preparation system. Contamination-free transfer of the prepared single crystal samples are characterized by cyclic voltammetry (CV).

UHV System

Ion Source

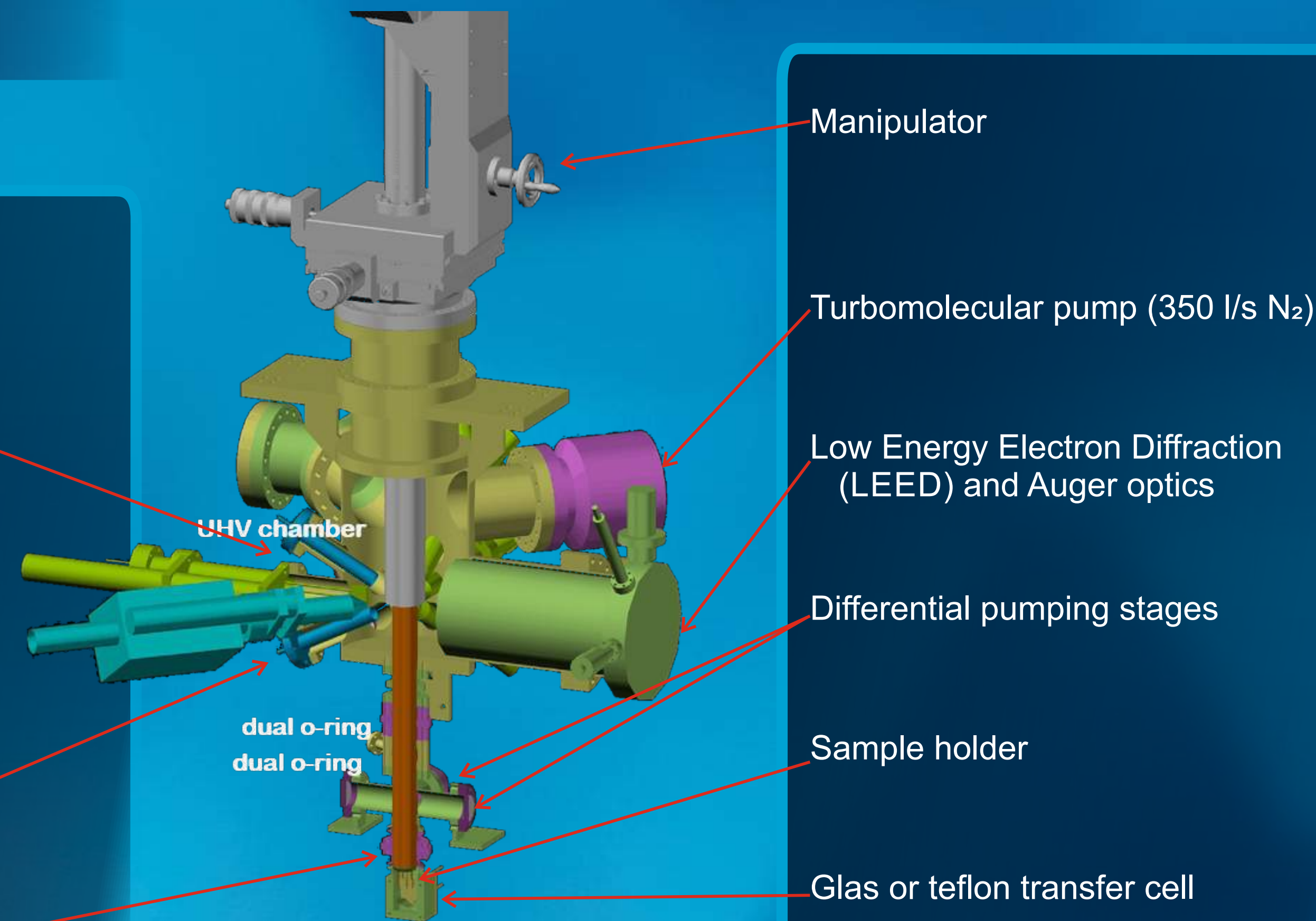
Electron Beam
Evaporator (2x)

Microbalance

Quadrupole Mass
Spectrometer

Knudsen Cell

Gate valve



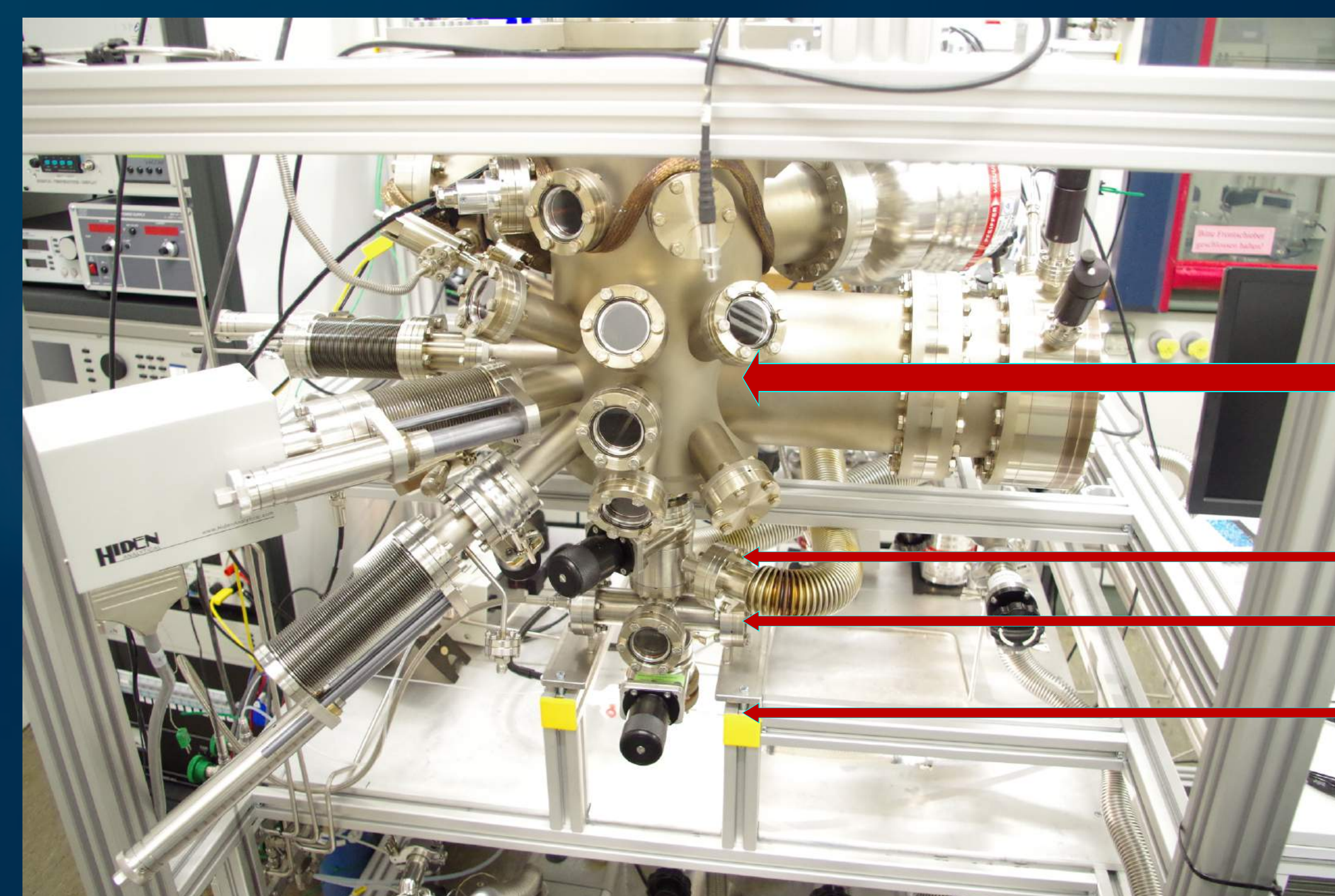
- Preparation and characterisation of complex model catalysts under UHV conditions
- Direct transfer in electrolyte into IR-spectroelectrochemistry cell without exposure to ambient conditions
- Back transfer to UHV and characterization by Auger and/or LEED optics

ELISA I



IR spectro-electrochemistry including vacuum FTIR spectrometer (Bruker Vertex 80v), gas inlet system, and new home build external reflection cell.

ELISA II

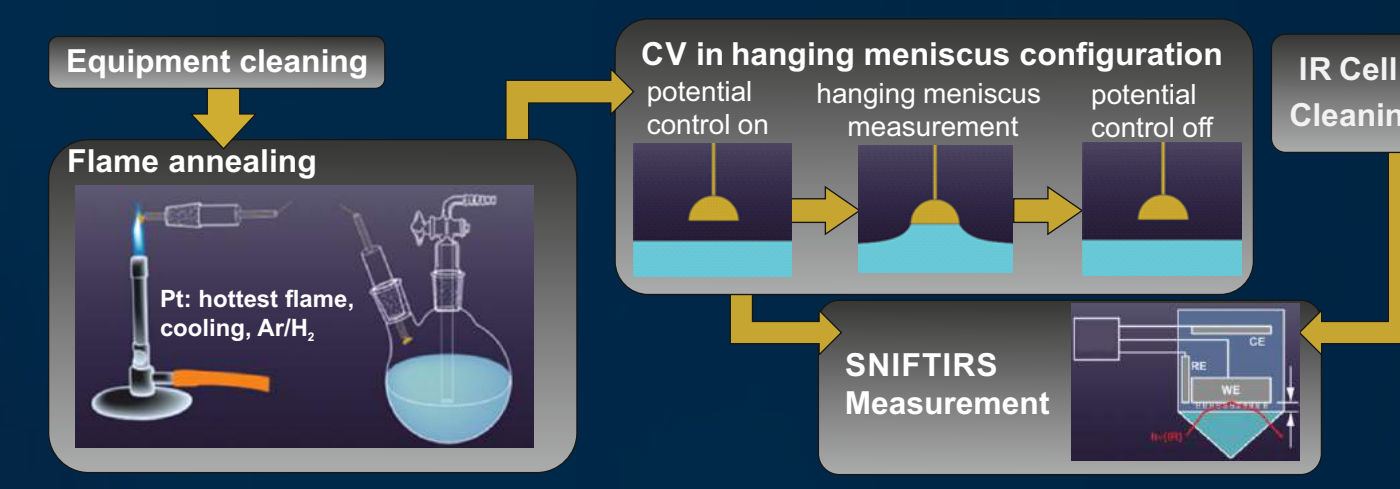
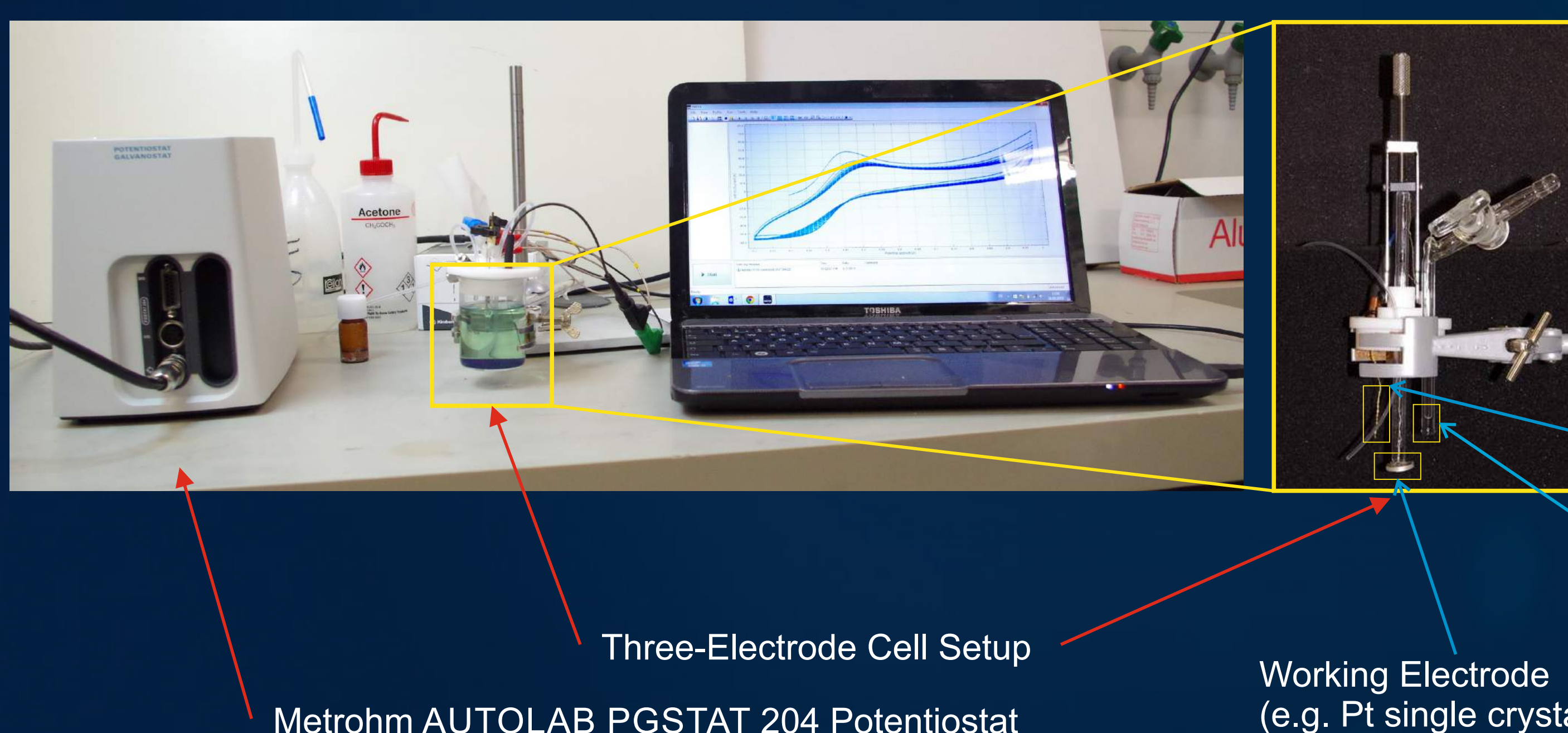


UHV preparation system including the transfer cell

- Ultra High Vacuum 10^{-8} mbar
- High Vacuum $\sim 10^{-5}$ mbar
- Low Vacuum ~ 1 mbar
- Atmosphere ~ 1000 mbar

transfer direction
pressure

Electrochemical set-up

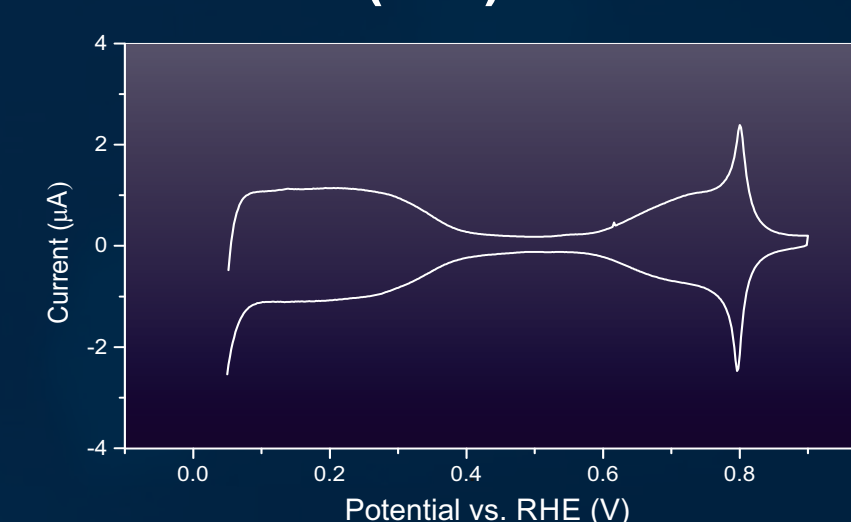


Gold Counter Electrode

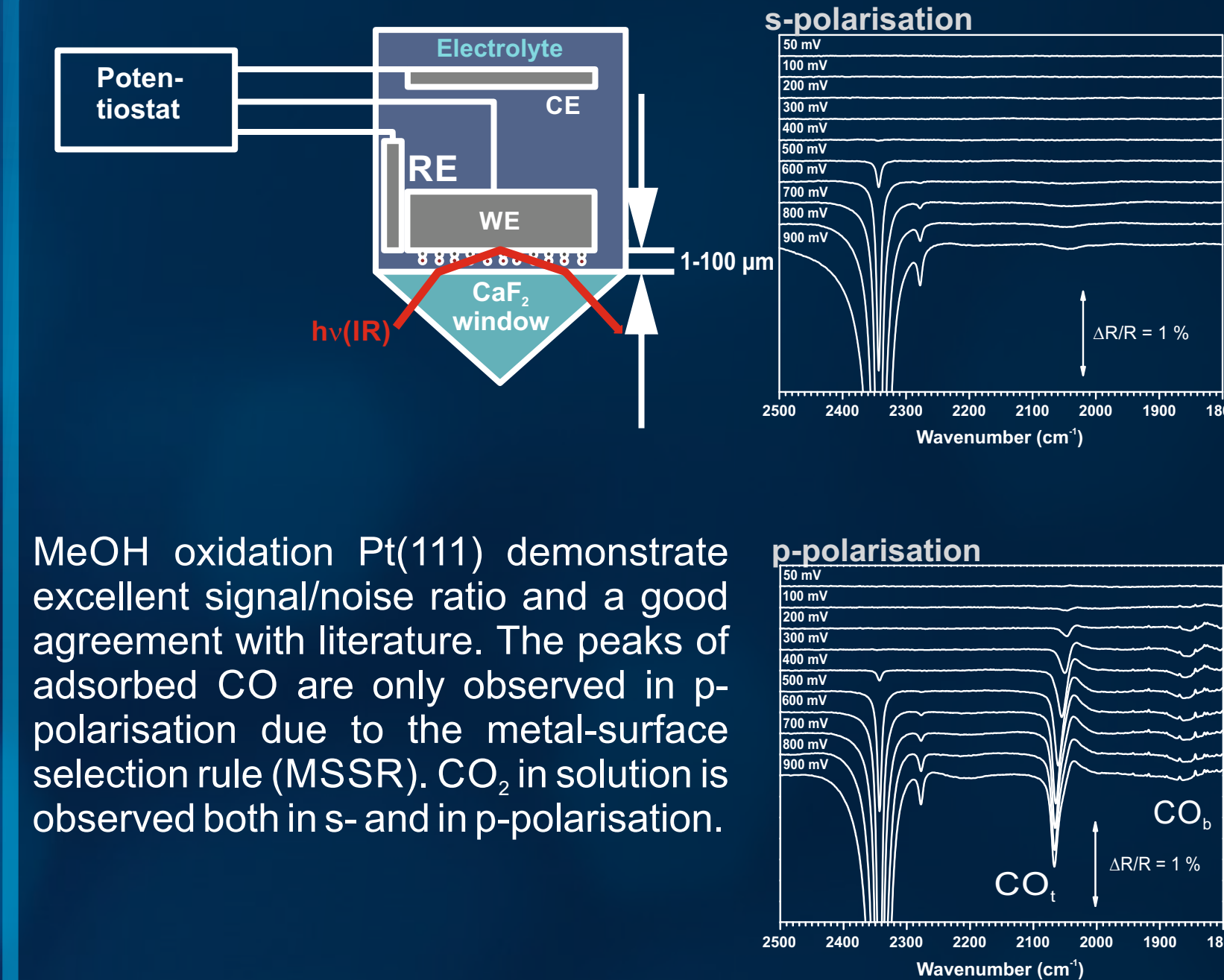
Normal Hydrogen
Reference Electrode
(NHE)

Working Electrode
(e.g. Pt single crystal)

Cyclic voltammogram of
clean Pt (111) in HClO_4



IR characterisation



MeOH oxidation Pt(111) demonstrate excellent signal/noise ratio and a good agreement with literature. The peaks of adsorbed CO are only observed in p-polarisation due to the metal-surface selection rule (MSSR). CO_2 in solution is observed both in s- and in p-polarisation.

[1] T. Iwasita, *Electrochimica Acta* 2002, 47, 3663-3674.
[2] G. Garcia, P. Rodriguez, V. Rosca, M. T. M. Koper, *Langmuir* 2009, 25, 13661-13666.

Acknowledgements