Instrumentation for X-ray emission spectroscopy at the KIT-INE radionuclide beamline stations at KARA

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KIT-INE operates two experimental stations dedicated to the investigation of radionuclide materials by X-ray based methods at the Karlsruhe research accelerator KARA (former ANKA synchrotron light source) - the INE-Beamline at a bending magnet port (fully operational since 2005) and the ACT laboratory at the CAT-ACT wiggler beamline (commissioned in 2016) [1,2]. Both experimental hutches are equipped and licensed for handling radioisotopes up to 1E+6 times their exemption limits and are operated as temporary controlled areas, enabling the investigation of 'hot' materials including genuine nuclear waste forms as well as in situ investigations (e.g., high p / high T) of radionuclide containing samples. While the focus at both beamlines was originally on XAFS-based speciation investigations in the context of the nuclear waste disposal safety case (encompassing processes during interim storage of spent nuclear fuel or nuclear waste glass and final deep geological disposal), another emphasis has been recently placed on fundamental studies employing the emerging high-resolution X-ray emission spectroscopy (HRXES) techniques.

The Johann-type X-ray emission spectrometer at the ACT laboratory is routinely applied for high-resolution/HERFD-XANES and RIXS experiments in a broad energy range encompassing the actinide M- and L-edges. In the tender X-ray region, scattering and absorption of X-rays is efficiently minimized by enclosing all beam paths - i.e., the impinging beam, the sample stage, five analyser crystals and the detector stage in Rowland circle geometry - in a He atmosphere. A new rigid He-flushed glove box with a flexible bag enclosing the spherical analyser crystals has been designed and will be permanently installed on the ACT experiment table at the beginning of 2020. The box is equipped with a spacious load lock for inserting sample cells (e.g., for in situ or combined UV-Vis/XES experiments) and various instrumentation feedthroughs (encompassing a LN_2 cryostat) and will allow for faster changes between standard transmission/fluorescence detection XAFS and HRXES experiments.

The capabilities for soft X-ray absorption and total yield fluorescence detection XAFS studies at INE-Beamline down to 2.1 keV - profiting from naturally narrow line widths - have been recently significantly improved by designing an advanced window-less 'He flow chamber'. The new setup has meanwhile allowed for recording the world's first Tc L_3-edge XANES (~2.68 keV) spectra of Tc species in aqueous solution.

References

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