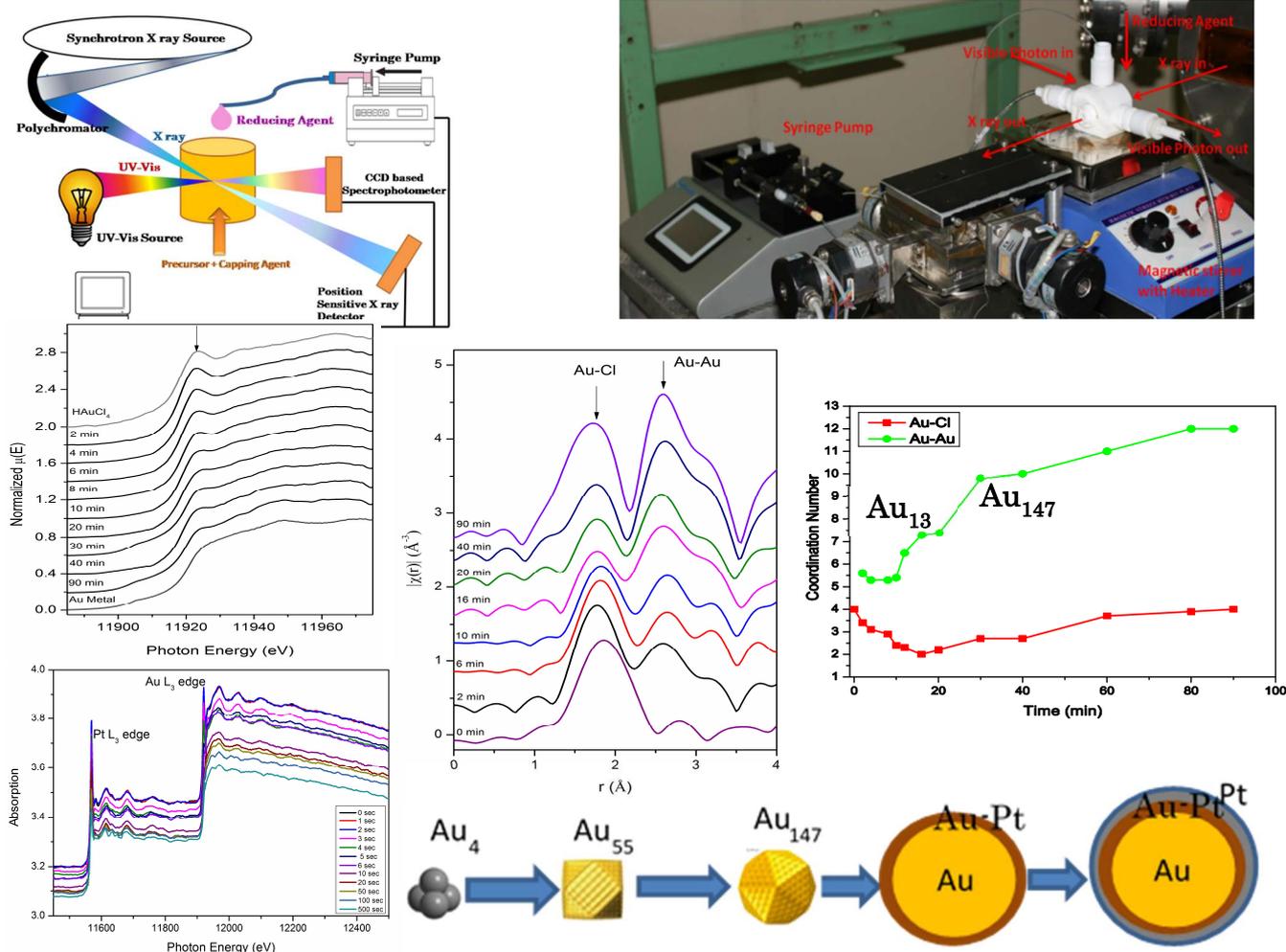


- Facility for *in-situ* XAS measurements on growth of nanoparticles (2016)



A facility has been developed at Indus-2 SRS to probe structural evolution of nanoparticles from a solution based precursor by simultaneously using time resolved XAS and UV-Vis absorption spectrophotometry. The schematic (top left panel) and the actual photograph (top right panel) of the set-up are shown above. The synthesis-cum-measurement process has been carried out in a specially designed teflon reaction cell having paths for both X-rays and UV-Vis radiation in mutually perpendicular directions. X-rays is transmitted through Kapton windows while the optical light is passed using optical fibres which are capped with teflon ferrules and are directly immersed into the solution. The growth of Au nanoparticles from its chloride based solution precursors using block copolymer based reducing agent-cum-stabilizer has been studied by *in-situ* XAS using the above set-up. The XANES measurements (middle left panel) show continuous reduction of white line peak with time depicting conversion of chloride precursors to metallic phase while EXAFS measurements (middle centre panel) show reduction of Au-Cl coordinations and increase in Au-Au coordinations manifesting formation of Au nanoparticles. From the coordination numbers obtained by EXAFS data analysis, the growth stages have been simulated (middle right panel). Au-Pt bimetallic nanoparticles have also been synthesized through using the above one-pot synthesis route from their respective chloride precursors using block copolymer as a stabilizer. Simultaneous *in-situ* XAS measurements at Au and Pt edges (bottom panel left) at BL-08 Energy Dispersive EXAFS beamline of Indus-2 SRS gives a detail microscopic account of the growth phenomena (bottom panel right) where Au<sub>4</sub> clusters are first formed which act like nuclei for the further growth and the Pt reduction occurs slowly on the surface of the Au nuclei.