



	Experiment title: XAS investigation of of intermediates in the synthesis of Au and Cu nanoparticles in aqueous solutions	Experiment number: CH-4866
Beamline:	Date of experiment: from: 01/09/2016 to: 06/09/2016	Date of report:
Shifts: 15	Local contact(s): Debora Motta-Meira	<i>Received at ESRF:</i>
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Report:

In frame of the beamtime we have studied three previously planned chemical processes along with some sulfide mineral samples. The first one is a well-known citrate-assisted synthesis of gold nanoparticles. A predetermined volume of sodium hydroxide solution (initial molar concentration is of 0.1 M) and then a portion of trisodium citrate solution were added to the reaction flask containing HAuCl_4 solution (1 mM – 10 mM, up to 200 mL) pre-heated to 70 °C using a water bath to set the molar ratio $\text{Na}_3\text{Citrate}/\text{HAuCl}_4$ of 3:1. After this the flask was put into a flow of cooled water to quench the reaction. Then, a portion of the reaction solution was loaded into a liquid cell for XAFS measurement.

The second process is a reduction of chloroauric acid with sodium sulfide. A pre-determined volume of sodium sulfide solution (concentration is of 0.1 M) was added to chloroauric acid solution (1-5 mM).

The third process is a reaction between copper sulfate and sodium sulfide in aqueous solution yielding copper sulfide nanoparticles. In this case, we are going to add a pre-determined portion of sodium sulfide solution to copper sulfate solution (1 mM). In addition, XAFS of the reactant solutions and solid metal sulphides and oxides samples as references were measured.

The treatment and analysis of experimental data are in progress. After the background subtraction and normalization procedure, the oscillating parts of the XAS spectra were extracted using Athena program of IFEFFIT program suite. After this, spectra in k-space (with appropriate k-weight) were Fourier-transformed. The spectra (in R-space) will be fitted using Artemis program. For the sake of illustration, the most representative XAS spectra obtained in solution during the second process along with corresponding EXAFS spectra in k- and R-space are shown in fig. 1.

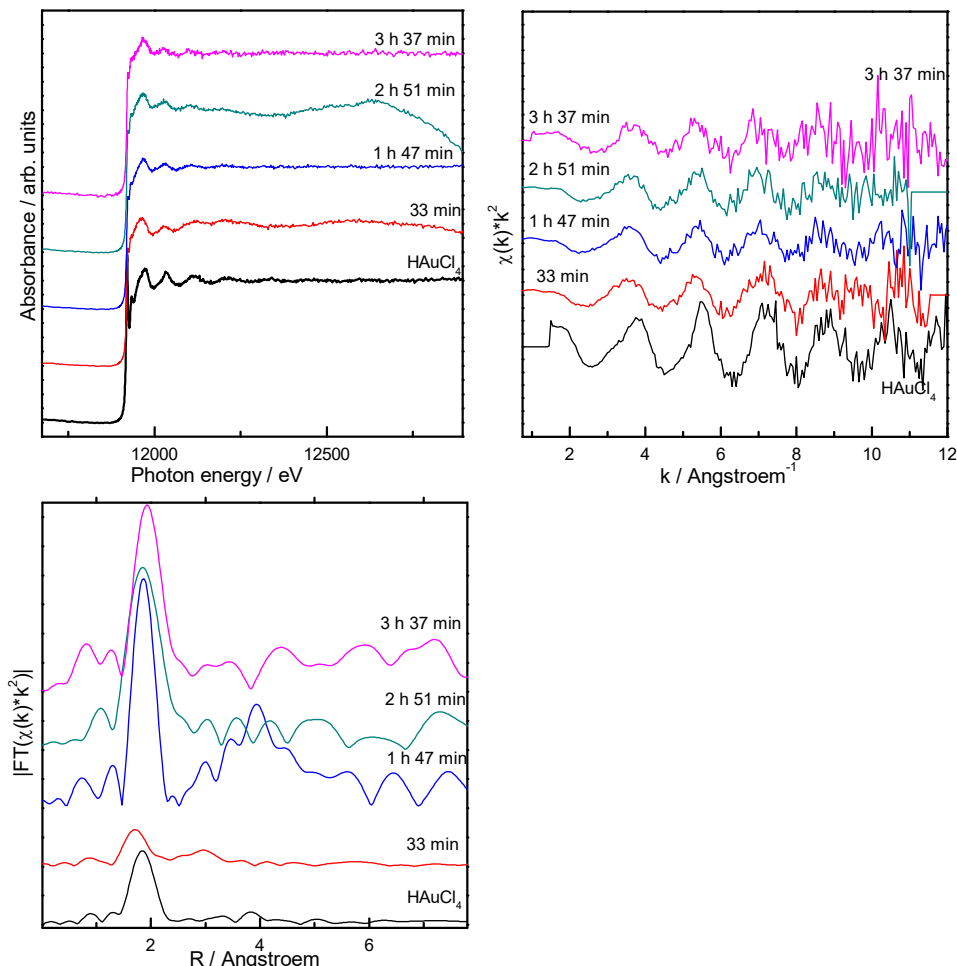


Figure 1. XAS spectra, EXAFS-spectra in k- and R-space of 1 mM HAuCl₄ solution and those of solutions obtained through mixing the tetrachloroauric acid with sodium sulfide in molar ratio [Na₂S]/[HAuCl₄] of 3:1 labeled with the time from start of the reaction.