Controllable synthesis of gold nanoparticles with ultrasmall sizes and high monodispersity via continuous supplement of precursor

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Supporting Information

Details of estimating gold concentration in solution by XANES edge-jump

According to the fundamental principle of x-ray absorption, after a beam of x-ray (the initial intensity is $I_0$) passes through a cell of solution with thickness $d$ along the beam direction, the intensity of outcome x-ray is reduced to $I_1=I_0 \exp(-\mu_E d)$. Here $\mu_E$ is the linear x-ray absorption coefficient of the solution at x-ray energy $E$. The edge jump (J) is defined as the difference between $\mu d$ just above and below the absorption edge $E_0$, i.e.,

$$ J = (\mu_{E_0+\delta E} - \mu_{E_0-\delta E}) \cdot d \quad (1) $$

For a sample containing many different elements, the absorption coefficient is given by

$$ \mu = \rho \sum_i \frac{n_i}{N} \sigma_i \quad (2) $$

Here $\rho$ is mass density of the material as a whole, $n_i/N$ is the mole fraction of element $i$, and $\sigma_i$ is the absorption cross section of element $i$. Substituting Eq. (2) into (1), we have

$$ J = \left[ \rho \sum_i \frac{n_i}{N} \sigma_{i,E_0+\delta E} - \rho \sum_i \frac{n_i}{N} \sigma_{i,E_0-\delta E} \right] \cdot d \quad (3) $$

Here, $\sigma_{i,E_0+\delta E}$ and $\sigma_{i,E_0-\delta E}$ are the absorption cross section for element $i$ just above and below the absorption edge $E_0$ of element $j$ (here it is Au). In the energy range from $E_0-\delta E$ to $E_0+\delta E$, the changes of absorption cross section for other elements are very small and could be neglected, hence Eq. (3) could be simplified to

$$ J = \rho \left[ \frac{n_j}{N} [\sigma_{j,E_0+\delta E} - \sigma_{j,E_0-\delta E}] \right] \cdot d \quad (4) $$

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The above Eq. (4) shows that the edge-jump is proportional to the total content of element j dissolved in the solution. Based on it, we estimated the temporal evaluation of Au content in the solution, which is plotted as Figure 4(b) in the manuscript.

**Synthesizing gold nanoparticles in benzene:**

The precursor AuClPPh₃ (0.375 mmol) and the surfactant dodecanethiol (48 μl) were mixed in the solvent of benzene (21 ml). Then reducing agent tert-butylamine-borane (3.75 mmol) was injected to the solution. The reactions were carried out at room temperature under vigorous stirring (600-800 rpm).

![Figure S1](image1.png)

Figure S1. The TME image of gold nanocrystals prepared in the solvent of benzene. The mean diameter of these nanoparticles is 3.0 nm.

![Figure S2](image2.png)

Figure S2. The high energy-resolution TEM image of 3.3 nm gold nanoparticles.