

# 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

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

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

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

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  $\text{AuClPPh}_3$  (0.375 mmol) and the surfactant dodecanethiol (48  $\mu\text{l}$ ) 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).

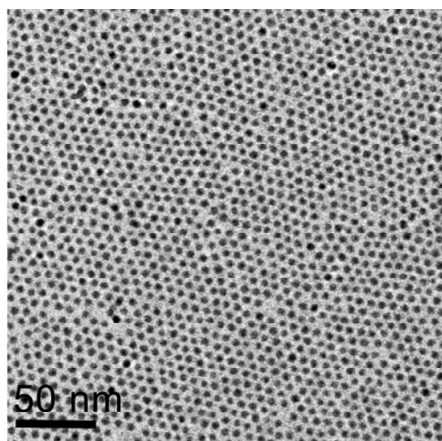


Figure S1. The TEM image of gold nanocrystals prepared in the solvent of benzene. The mean diameter of these nanoparticles is 3.0 nm.

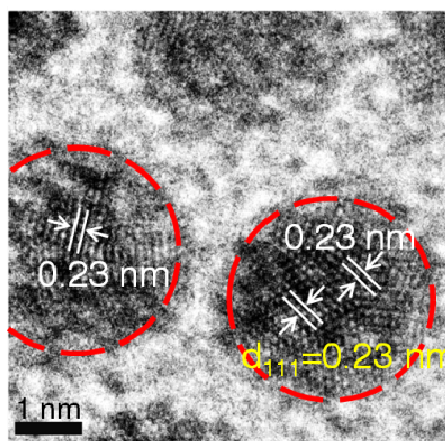


Figure S2. The high energy-resolution TEM image of 3.3 nm gold nanoparticles.