

An *In-situ* Variable-temperature Surface-enhanced Raman Spectroscopic Study of the Plasmon-mediated Selective Oxidation of *p*-Aminothiophenol

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Chemicals and Materials. $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ and sodium citrate were purchased from Aladdin and Sinopharm Chemical Reagent Co., Ltd., respectively. Ultrapure water ($18.2 \text{ M}\Omega \cdot \text{cm}$ at 300 K) was produced by using Milli-Q system and used to make all solutions.

Preparations of Au Nanoparticles. The Au nanoparticles were synthesized through Au-seeds-growth, according to Frens' recipe.^[1] Firstly, 1.5 mL 1 wt% sodium citrate solution was rapidly ejected into boiling 50 mL 1 wt% HAuCl_4 aqueous solution under vigorously stirring. After that, the mixture was boiling for 60 minutes and cooled naturally, which is a sol of 16 nm Au nanoparticles. Secondly, 3 mL Au sol was diluted by using 18.2 mL ultrapure water. Under vigorous stirring, 0.1 mL 1 wt% sodium citrate solution and 2.5 mL 1 wt% ascorbic acid solution were ejected into the diluted Au sol at 4 °C. Eventually, 1.2 mL 0.825 wt% HAuCl_4 solution was added into the mixture of Au sol, which eventually gives a sol of 50 nm Au nanoparticles.

The sol of 50 nm Au nanoparticles was cleaned and concentrated, and then dropped onto a clean Si wafer which was the substrate in VT-SERS measurements.

n REFERENCES

(1) Fang, P.-P.; Li, J.-F.; Yang, Z.-L.; Li, L.-M.; Ren, B.; Tian, Z.-Q. Optimization of SERS activities of gold nanoparticles and gold-core-palladium-shell nanoparticles by controlling size and shell thickness. *J. Raman Spectrosc.* **2008**, 39, 1679-1687.