Gold nanoparticles produced by low-temperature heating of the dry residue of a droplet of an HCl acidic solution of HAuCl₄·4H₂O in a low vacuum

Hiroki UMEDA, Yuya MEZAKI, Ayumi OSHIO, Yasunori KANEKO, Ryota OKAMOTO, Sakuya KUSUMOTO, and Shinsuke KUNIMURA†

Department of Industrial Chemistry, Tokyo University of Science, 1-3 Kagurazaka, Shinjuku-ku, Tokyo 162-8601, Japan

† To whom correspondence should be addressed.

E-mail: kunimura@rs.tus.ac.jp
Abstract

An easy method is presented for producing gold nanoparticles. We show that, by simultaneous low-temperature heating of a quartz glass substrate on which the dry residue of a 10 μL droplet of an HCl acidic solution of HAuCl₄·4H₂O is deposited and a counter substrate using Peltier devices in a low-vacuum condition produced by a rotary pump, gold nanoparticles with sizes ranging from about 20 to one hundred and several tens of nanometers are produced on the counter substrate. In this study, an application of a gold nanoparticle substrate produced by this method to the sample holder for surface-enhanced Raman scattering analysis is also shown.

Keywords Au nanoparticle, HAuCl₄, low-temperature heating, low vacuum, surface-enhanced Raman scattering
Introduction

Gold (Au) nanoparticles were applied to surface-enhanced Raman scattering (SERS) analysis.$^{1-8}$ The use of Au nanoparticles deposited on a substrate (Au nanoparticle substrate) is beneficial for easily performing SERS measurements. Therefore, a simple and inexpensive method for producing Au nanoparticle substrate can contribute to measuring a large amount of SERS spectra rapidly. Chemical synthesis methods for Au nanoparticles are classified into two methods: vapor phase methods and liquid phase methods.$^9$ As an example of a liquid phase method, a method to produce Au nanoparticles using the reduction of HAuCl₄ with a reducing agent is cited. When Au nanoparticles are produced by liquid phase methods, these particles produced in liquid are needed to be deposited on a substrate in order to prepare Au nanoparticle substrate.

HAuCl₄, which is usually used for producing Au nanoparticles using liquid phase methods, can be used as a precursor for producing Au nanoparticles using vapor phase methods as described below. When a vapor phase method is used, Au nanoparticles are directly produced on a substrate. Therefore, vapor phase methods make it possible to rapidly produce Au nanoparticle substrate. Shi et al.$^{10}$ produced Au nanoparticles by annealing monolithic porous silica containing HAuCl₄ in H₂ gas at 973 K (700 °C). Palgrave et al.$^{11}$ reported that Au films were produced on a substrate in a chemical vapor deposition reactor that was heated at 400 °C when aerosols produced from a methanol solution of HAuCl₄·3H₂O were transported to the reactor by nitrogen gas. Chen et al.$^{12}$ reported that Au nanoparticles were produced on a substrate placed away from an ethanol solution of HAuCl₄·4H₂O when the ethanol solution of HAuCl₄·4H₂O and the substrate were heated at 500 °C for 2h with nitrogen gas flow in a tube in which the pressure was 600 mTorr after the temperature was increased from 25 to 500 °C in 12.5
min, and they concluded that Au nanoparticles were produced by heating a film of a mixture of AuCl and AuCl$_3$ deposited on the substrate, which originated from gaseous these Au chloride species produced by heating HAuCl$_4$. According to their paper,$^{12}$ increasing the heating temperature was needed in order to reduce the amount of Au chloride species remained on the substrate, but in the supplementary information of that paper, Au nanoparticles were shown to be produced on a substrate even when heating was performed at 100 °C under atmospheric pressure. Yang et al.$^{13}$ reported that Au nanoparticles were produced on a substrate placed away from AuCl powder when heating was performed at 120 °C for 30 min with argon gas flow in a furnace in which the pressure was 0.7 Torr. In their report,$^{13}$ metallic Au and vaporized AuCl$_3$ were shown to be produced from solid AuCl by performing low-temperature heating.

In this study, we show that Au nanoparticles with high purity were produced on the counter substrate when the dry residue of a droplet of an HCl acidic solution of HAuCl$_4$$\cdot$4H$_2$O on a quartz glass substrate and the counter substrate were heated from room temperature to a hundred and several tens of degrees Celsius in 9 min in a low vacuum. In this method, Au nanoparticles with high purity are produced on the counter substrate by heating the dry residue at such a low temperature for a short time without flowing a carrier gas. An application of Au nanoparticles produced by using this method to SERS analysis is also presented.

**Experimental**

Two different procedures, which are referred to as procedure A and procedure B in this paper, were conducted for producing Au nanoparticles. Procedure A was performed as follows:
1. A 10 μL droplet of a 1000 mg/L Au standard solution (an HCl acidic solution of HAuCl₄·4H₂O) (FUJIFILM Wako Pure Chemical Co., Osaka, Japan), which was placed on a quartz glass substrate, was naturally dried in air at room temperature.

2. The dry residue of the 1000 mg/L Au standard solution on the quartz glass substrate, which faced to the surface of a counter substrate with a distance of about 2 mm in a vacuum chamber, was heated for 9 min using a Peltier device in a low-vacuum condition.

After this procedure, products were produced on the counter substrate. Procedure B was similar to procedure A, but simultaneous heating of a quartz glass substrate on which the dry residue of a 10 μL droplet of the 1000 mg/L Au standard solution was placed and a counter substrate was performed for 9 min in a low vacuum. Figure 1 shows a schematic view of experimental setup. The air was evacuated from the vacuum chamber by using a rotary pump, and the pressure value was set to 5 Pa when starting procedures A and B. However, the pressure value was not constant during experiments. The pressure value increased to over 10 Pa after starting heating, and then it gradually decreased. A quartz glass substrate was usually used as a counter substrate, but a carbon sheet was attached on a quartz glass substrate when preparing an analyte for acquiring secondary electron images and energy-dispersive X-ray (EDX) spectra. Quartz glass substrates used in this study had a diameter of 10 mm and a thickness of 1 mm. The temperature of the surface of the Peltier device increased by applying a DC current of 1.2 A to the Peltier device using a DC power supply. A quartz glass substrate where the dry residue of the 1000 mg/L Au standard solution was placed and the counter substrate were attached on Peltier devices as shown in Fig. 1.
Temperatures of the surfaces of two Peltier devices facing each other were measured using chromel-alumel thermocouples, but a quartz glass substrate on which the dry residue of a droplet of the 1000 mg/L Au standard solution was placed and another quartz glass substrate as the counter substrate were not placed on the Peltier devices. When starting the measurement of temperatures, the pressure value was set to 5 Pa. In these measurements, the temperatures of the surfaces of the Peltier devices increased from room temperature to about 160 °C and about 170 °C 9 min after starting the application of a DC current of 1.2 A to the Peltier devices. The temperature of the surface of a quartz glass substrate attached on a Peltier device 9 min after applying a DC current of 1.2 A to the Peltier device when procedures A and B were performed would be lower than that of the surface of the Peltier device.

Total reflection X-ray fluorescence (TXRF) spectra of products on counter substrates produced by procedures A and B were measured by using a portable TXRF spectrometer. The setup of the portable TXRF spectrometer used in this study was described in ref. 15. Three products were prepared by procedure A, and the same number of products was prepared by procedure B. All products were measured by the portable TXRF spectrometer. A counter substrate on which products were deposited was tilted at 0.05° to the horizontal for the measurement. An analytic was irradiated with X-rays from an X-ray tube 50 kV Magnum (Moxtek Inc., Orem, UT, USA) with a tantalum anode that was operated at 25 kV and 0.2 mA. An X-ray detector VITUS-SDD (Ketek GmbH, Munich, Germany) was used for measuring X-ray intensities at each X-ray energy. TXRF spectra of dry residues of 1 μL droplets of the 1000 mg/L Au standard solution and a 1000 mg/L K standard solution that is a KCl aqueous solution (FUJIFILM Wako Pure Chemical, Co.) were also measured. A diamond-like carbon (DLC) coated quartz glass substrate was used as the sample.
holder for these measurements. In this study, the thickness of a DLC film formed on a square quartz glass substrate with a length of 30 mm, a width of 30 mm, and a height of 5 mm were set to 1 μm, and the film formation was performed by Nanotec Co. (Kashiwa, Japan). Experimental conditions for measurements of these dry residues were the same as those for measurements of products produced by procedures A and B. All TXRF spectra were measured in air for 600 s. By using a scanning electron microscope (SEM) JSM-7200F (JEOL Ltd., Akishima, Japan) equipped with an energy-dispersive X-ray (EDX) spectrometer JED-2300F (JEOL Ltd.), secondary electron images and EDX spectra of products produced by procedures A and B were obtained. For acquiring secondary electron images and EDX spectra, the accelerate voltage of electron beam was set to 5 kV. Raman spectra of 1 μL droplets of 1×10^{-2} mol/L nicotinamide solution were measured by using a portable Raman spectrometer C12710 (Hamamatsu Photonics K. K., Hamamatsu, Japan). One μL droplets of the nicotinamide solution on a quartz glass substrate and the products on the counter substrates produced by procedures A and B were irradiated with 785 nm laser beam. A Raman spectrum of the nicotinamide solution was acquired by averaging 5 spectra that were measured for 10 s. The power of the 785 nm laser beam was set to 55.5 mW for the measurements.

**Results and Discussion**

Figure 2 shows representative TXRF spectra of the dry residues of the 1000 mg/L Au standard solution and the 1000 mg/L K standard solution and the products produced by procedures A and B. The sum of net intensities of Au Mα and Mβ lines in Fig. 2a were 20828 counts / 600 s. Because the mass of Au in the dry residue of a 1 μL droplet of
the 1000 mg/L Au standard solution was calculated to be 1 μg, the sum of net intensities of Au Mα and Mβ lines per 1 μg of Au was calculated to be 20828 counts /μg. The net intensity of the Cl Kα line in Fig. 2b was 135684 counts / 600 s. Because the 1000 mg/L K standard solution was a KCl aqueous solution, the mass of Cl in the dry residue of a 1 μL droplet of this solution was calculated to be 0.9 μg. Therefore, the net intensity of the Cl Kα line per 1μg of Cl was calculated to be 150760 counts / μg. The Au and Cl peaks in Figs. 2c and 2d originated from the products produced by procedures A and B. The sum of net intensities of Au Mα and Mβ lines and the net intensity of the Cl Kα line, which were shown in Fig. 2c, were 134149 and 143127 counts, respectively. Because the sum of net intensities of Au Mα and Mβ lines per 1μg of Au was about 0.14 times as high as the net intensity of the Cl Kα line per 1 μg of Cl, the mass of Au in the product produced by procedure A was estimated to be about 7 times as high as that of Cl. Therefore, the number of moles of Cl in this product was estimated to be comparable to that of Au. The ratio of the net intensity of the Cl Kα line to the sum of net intensities of Au Mα and Mβ lines in Fig. 2c was 1.1, and that in Fig. 2d was 0.14. Table 1 shows maximum values, minimum values, mean values, and relative standard deviations of the ratio of the net intensity of the Cl Kα line to the sum of net intensities of Au Mα and Mβ lines. As shown in Table 1, the ratio of the net intensity of the Cl Kα line to the sum of net intensities of Au Mα and Mβ lines was significantly reduced when procedure B was performed, but the relative standard deviation increased.

Figure 3 shows a representative secondary electron image of the product produced by procedure A. Figure 3 also shows EDX spectra of the product produced by procedure A and an SEM image indicating points where the EDX spectra were measured. Particles with sizes of several hundred nanometers were observed in Fig. 3a.
As shown in Figs. 3c and 3d, strong Au M lines were observed in the spectra of points 001 and 003 on particles in Fig. 3b, and weak Cl Kα lines appeared in these spectra. The Au peak, whose intensity was lower than those in the spectra of points 001 and 003, was detected in the spectrum of point 002 in a relatively dark region where particles were not observed, but was near point 001 on the particle, and the intensity of the Cl peak was much lower than that of the Au peak in this region. The Au and Cl peaks were observed in the spectrum of point 004 in the region where particles were not present, and the intensity of the Cl peak was comparable to that of the Au peak. Figure 4 shows representative secondary electron images of products produced by procedure B. Figure 4 also shows EDX spectra of the products produced by procedure B and SEM images indicating points and areas where the spectra were measured. As shown in Fig. 4a, spherical, triangular, and hexagonal particles were observed, and the size of particles ranged from about 20 to one hundred and several tens of nanometers. Particles look like rod-shaped particles in Fig. 4a may be triangular and hexagonal particles tilted at about 90° to the surface of the carbon sheet. Although the same procedure was used, spherical particles, which were larger than those in Fig. 4a, were observed in Fig. 4d. Small spherical particles in Fig. 4d tended to be densely distributed. As shown in Fig. 4c, strong Au peaks were detected in the spectra of points 001 and 002 on particles in Fig. 4b, and Cl peaks were not observed. A weak Au peak was detected in the spectrum of point 003 in the region where particles were not observed, and the Cl Kα line was hardly observed. As shown in Fig. 4f, Au peaks appeared in the spectra of point 001 on a particle and area 002 in the region where small particles were distributed in Fig. 4e, and weak Cl peaks were observed in these spectra. When the spectrum of area 003 in the region where particles were not observed in Fig. 4e was measured, the net intensity of the Cl peak was as high as that of the Au peak. Although the products
shown in Fig. 4 were produced by the same procedure, the purity of Au nanoparticles in
the product shown in Figs. 4d were worse than that of the product shown in Figs. 4a.

Figure 5 shows representative Raman spectra of 1 μL droplets of 1×10⁻² mol/L
nicotinamide solution on a quartz glass substrate and the products on the counter
substrates produced by procedures A and B. A Raman peak clearly appeared at 1029
\( \text{cm}^{-1} \) in Fig. 5 when using the product produced by procedure B. A Raman peak
appeared at 1032 cm⁻¹ in a SERS spectrum of nicotinamide,\(^\text{17}\) and the Raman peak at
1029 cm⁻¹ in Fig. 5 would originate from nicotinamide. On the other hand, the Raman
peak at 1029 cm⁻¹ was not observed when the quartz glass substrate was used as a
sample holder. From the above results, Au nanoparticles produced by procedure B led
to an enhancement in the intensity of Raman scattering due to SERS effect. The
Raman peak at 1029 cm⁻¹ did not appear in the spectrum of the nicotinamide solution
on the product produced by procedure A as shown in Fig. 5. There was a possibility
that SERS effect was not obtained because of the low purity of Au nanoparticles
produced by procedure A.

Evaporable products can be produced even when heating HAuCl₄ at 100 °C.\(^\text{12}\)
Therefore, by heating the dry residue of the 1000 mg/L Au standard solution (an HCl
acidic solution of HAuCl₄·4H₂O) at one hundred and several tens of degrees Celsius in
a low vacuum, evaporable products can be produced from the dry residue. As shown
in Fig. 2c, strong Cl Kα line and Au M lines were observed when the TXRF spectrum of
the product produced by procedure A was measured. This result indicated that Au
chloride species were deposited on the counter substrate because gaseous Au chloride
species, which were produced by heating the dry residue of the 1000 mg/L Au standard
solution, reached the counter substrate. Furthermore, this result showed that an
adequate amount of Au chloride species originating from gaseous Au chloride species
can be deposited on the counter substrate without flowing a carrier gas. As shown in Figs. 3c and 3d, strong Au M lines were observed in the EDX spectra of the points on particles in the product produced by procedure A, and the intensities of Cl peaks were much lower than those of the Au peaks in these spectra. As shown in Fig. 3d, the intensity of the Cl Kα line was comparable to that of the Au peak when the spectrum of the point in the region where particles were not observed was measured. These results showed the following points:

1. A thin film of Au chloride species was formed on the counter substrate.
2. The weak Cl peaks in the spectra of points on particles observed in the SEM image originated from remaining Au chloride species, and these particles were metallic Au.

Therefore, we have concluded that the product produced by procedure A consisted of metallic Au and remaining Au chloride species.

As shown in Fig. 2d, the intensity of the Cl Kα line in the TXRF spectrum of the product produced by procedure B was weak, and this result showed that increasing the heating temperature of the counter substrate accelerated the production of metallic Au from Au chloride species. As shown in Figs. 4c and 4f, the Au M lines were detected in the EDX spectra of points on particles in the product produced by procedure B, but Cl peaks were not observed or weakly detected. Therefore, these particles were shown to be metallic Au. The Au M lines were detected in the spectrum of a point in the region where particles were not observed as shown in Fig. 4c, and the Cl Kα line scarcely appeared in this spectrum. This would be because ultra-small Au seeds were produced from Au chloride species, and these ultra-small Au seeds would grow to Au nanoparticles. Our study in the present paper showed that the production of metallic
Au from Au chloride species deposited on the counter substrate was almost completed by performing the heating from room temperature to one hundred and several tens of degrees Celsius in 9 min. Therefore, we have found that Au nanoparticles with high purity can be produced by performing low-temperature heating of the dry residue of a solution of H\text{AuCl}_4\cdot4\text{H}_2\text{O} for a short time. As shown in Figs. 3 and 4, the size of Au nanoparticles on the counter substrate produced by procedure B was smaller than that produced by procedure A. This result indicated that increasing the heating temperature of Au chloride species deposited on the counter substrate led to the reduction in the size of Au nanoparticles. When procedure B was conducted, the variation in the ratio of the net intensity of the Cl K\(\alpha\) line to the sum of net intensities of Au M\(\alpha\) and M\(\beta\) lines became large as shown in Table 1. Although the same procedure was performed, the Cl K\(\alpha\) line was detected as shown in Fig. 4f when the EDX spectrum of the area of the region where particles were not observed was measured, but it was hardly detected in the spectrum of the point in the region where particles were not observed as shown in Fig. 4c. Furthermore, spherical Au nanoparticles, which were larger than those in Fig. 4a, appeared in Fig. 4d. These reasons remain unclear, but there is a possibility that the difference in the amount of remaining Au chloride species and the size of spherical Au nanoparticles was caused by the variation in the temperature of the counter substrate heated by the Peltier device during procedure B.

Simultaneous low-temperature heating of the dry residue of an HCl acidic solution of H\text{AuCl}_4\cdot4\text{H}_2\text{O} and the counter substrate in a low vacuum for a short time is beneficial for producing Au nanoparticles on the counter substrate although the mechanism of this method remains unclear. The concentration of remaining Au chloride species on the counter substrate is low after the production of Au nanoparticles. Although the mass of the dry residue of a 10 \(\mu\)L droplet of dry residue of the 1000 mg/L
Au standard solution, which was used as a material for producing Au nanoparticles, was only about 20 μg, the Au nanoparticle substrate produced by this method was applied to SERS analysis. This method makes it possible to produce Au nanoparticle substrates for SERS measurements in a short time.

Conclusions

This paper described a method for producing Au nanoparticles using low-temperature heating of the dry residue of a droplet of an HCl acidic solution of HAuCl₄⋅4H₂O in a low-vacuum condition. By using this method, Au nanoparticles with high purity were produced, and they were applied to the sample holder for SERS analysis. Advantages of this method are as follows: 1) low cost; 2) no waste liquid arising; 3) simple procedure; and 4) short production time. Therefore, conventional methods for producing Au nanoparticles deposited on a substrate can be replaced with this method.

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Table 1 Maximum values, minimum values, mean values, and relative standard deviations of the ratio of the net intensity of the Cl Kα line to the sum of net intensities of Au Mα and Mβ lines.

| Procedure | n  | Maximum | Minimum | Mean  | Relative standard deviation, % |
|-----------|----|---------|---------|-------|-------------------------------|
| A         | 3  | 1.07    | 0.73    | 0.92  | 19                            |
| B         | 3  | 0.15    | 0.04    | 0.11  | 55                            |

Figure captions

Fig. 1 Schematic view of experimental setup.

Fig. 2 TXRF spectra of the dry residues of (a) a 1000 mg/L Au standard solution and (b) a 1000 mg/L K standard solution and products produced by procedures (c) A and (d) B.

Fig. 3 (a) Secondary electron image of a product produced by procedure A, (b) an SEM image indicating points where the EDX spectra of a product produced by procedure A were measured, (c) EDX spectra of points 001 (black line) and 002 (light grey line) in Fig. 3b, and (d) EDX spectra of points 003 (black line) and 004 (light grey line) in Fig. 3b.

Fig. 4 (a) Secondary electron image and (b) a SEM image of a product produced by procedure B indicating points where the EDX spectra were measured, (c) EDX spectra of points 001 (black line), 002 (light grey line), and 003 (grey line) in Fig. 4b, (d) a secondary electron image and (e) a SEM image of another product produced by procedure B indicating a point and areas where the EDX spectra were measured, and (f) EDX spectra of point 001 (black line) and areas 002 (light grey line) and 003 (grey line) in Fig. 4e.

Fig. 5 Raman spectra of 1 μL droplets of 1×10⁻² mol/L nicotinamide solution on a quartz glass substrate (grey line) and products on quartz glass substrates produced by procedures A (light grey line) and B (black line).
Dry residue of a droplet of an HCl acidic solution of HAuCl₄·4H₂O

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Fig. 5 Raman spectra of 1 μL droplets of $1 \times 10^{-2}$ mol/L nicotinamide solution on a quartz glass substrate (grey line) and products on quartz glass substrates produced by procedures A (light grey line) and B (black line).
Simple method for producing Au nanoparticles

**Graphical Index**

Dry residue of a droplet of an HCl acidic solution of HAuCl₄·4H₂O

- Rotary pump
- Peltier device
- Quartz glass substrate
- Counter substrate (Quartz glass substrate)

Pirani gauge

DC power supply