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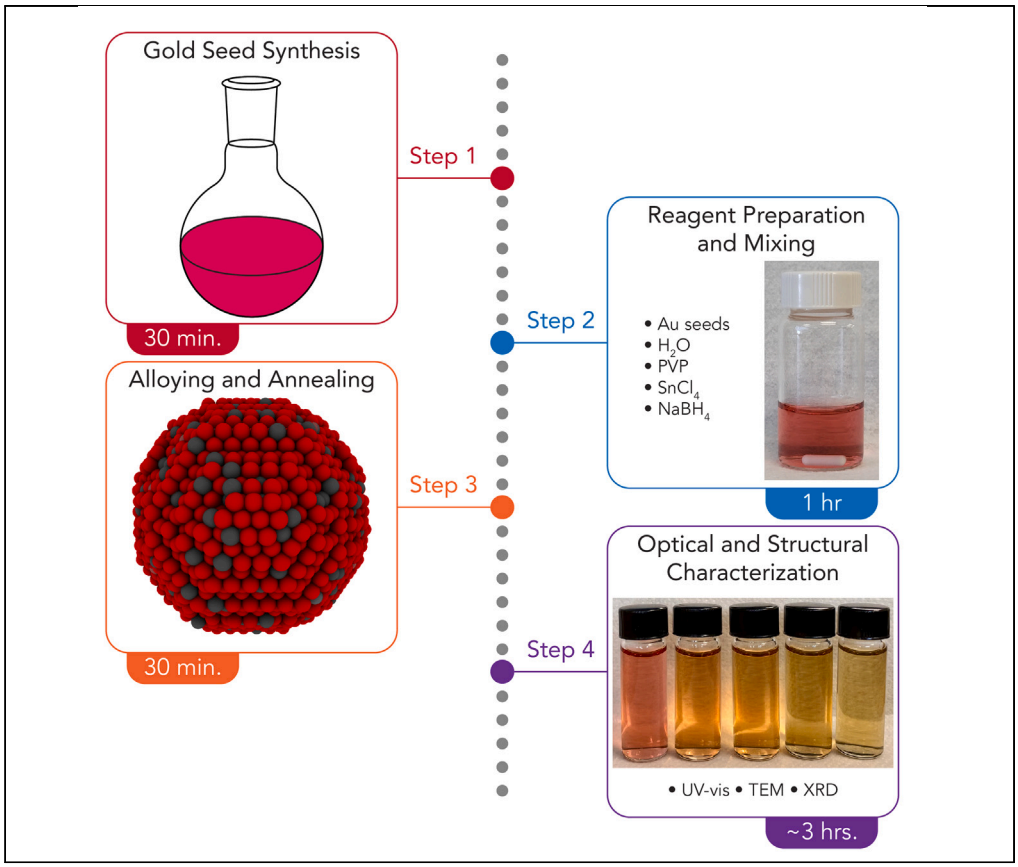
# Synthesis of gold–tin alloy nanoparticles with tunable plasmonic properties




Plasmonic nanoparticles and nanocrystalline materials have broad applicability in catalysis, optoelectronics, sensing, and sustainability. Below, we detail a robust protocol for the synthesis of bimetallic Au–Sn nanoparticles in mild, aqueous conditions. This protocol describes the steps for synthesizing gold nanoparticle seeds, diffusion Sn into the seeds by chemical reduction, and the optical and structural analysis by UV-visible spectroscopy, X-ray diffraction, and electron microscopy.

Publisher's note: Undertaking any experimental protocol requires adherence to local institutional guidelines for laboratory safety and ethics.

Anthony J. Branco, Sarah S. Dawes, Noah L. Mason, Maria V. Fonseca Guzman, Melissa E. King, Michael B. Ross

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## Highlights

Protocol for the synthesis of ~13 nm Au nanoparticle seeds

Protocol for the chemical reduction and diffusion of Sn into Au nanoparticle seeds

Optical characterization of bimetallic localized surface plasmon resonances

Powder X-ray diffraction and transmission electron microscopy characterization

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# Synthesis of gold–tin alloy nanoparticles with tunable plasmonic properties

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## SUMMARY

**Plasmonic nanoparticles and nanocrystalline materials have broad applicability in catalysis, optoelectronics, sensing, and sustainability. Below, we detail a robust protocol for the synthesis of bimetallic Au–Sn nanoparticles in mild, aqueous conditions. This protocol describes the steps for synthesizing gold nanoparticle seeds, diffusing Sn into the seeds by chemical reduction, and the optical and structural analysis by UV-visible spectroscopy, X-ray diffraction, and electron microscopy.**

**For complete details on the use and execution of this protocol, please refer to Fonseca Guzman et al.[1]**

## BEFORE YOU BEGIN

This protocol describes the synthesis of ~13 nm spherical Au seeds followed by the subsequent chemical reduction and diffusion of Sn into those seeds. Methods for their structural and optical characterization are included. Before beginning, one should ensure that the following procedures are implemented:

### Clean glassware

Timing: 2–3 h

1. Freshly clean all glassware and stir bars with aqua regia (1:3 mole ratio of $HNO_3$: HCl), rinse thoroughly with ultrapure water (18.2 MΩ), and allow to air dry.

## KEY RESOURCES TABLE

| REAGENT or RESOURCE | SOURCE | IDENTIFIER |
|---|---|---|
| Chemicals, peptides, and recombinant proteins | | |
| Hydrogen tetrachloroaurate (III) trihydrate ($HAuCl_4 \cdot 3H_2O$, 99.99%) | Alfa Aesar | CAS: 16961-25-4 |
| Trisodium citrate dihydrate ($C_6H_5Na_3O_7 \cdot 2H_2O$, 99%) | Alfa Aesar | CAS: 6132-04-3 |
| Tin(IV) chloride ($SnCl_4 \cdot 5H_2O$, 99.99%) | Alfa Aesar | CAS: 7646-78-8 |
| Poly(vinylpyrrolidone) (PVP; molecular weight [MW] = 40,000) | Alfa Aesar | CAS: 9003-39-8 |
| Sodium borohydride ($NaBH_4$, 97+%) | Alfa Aesar | CAS: 16940-66-2 |

(*Continued on next page*)

*Continued*

| REAGENT or RESOURCE | SOURCE | IDENTIFIER |
|---|---|---|
| Milli-Q® Ultrapure Water, (18.2 MΩ-cm) | Made by Direct-Q® 3 UV-R water purification system | N/A |
| Nitric acid ($HNO_3$, Certified ACS Plus) | Fisher Scientific | CAS: 7697-37-2, 7732-18-5 |
| Hydrochloric acid (HCl, NF/FCC) | Fisher Scientific | CAS: 7647-01-0, 7732-18-5 |
| Software and algorithms | | |
| Cary WinUV | Agilent Technologies | https://www.agilent.com/en/product/molecular-spectroscopy/uv-vis-uv-vis-nir-spectroscopy/uv-vis-uv-vis-nir-software/cary-winuv-software |
| ImageJ | National Institute of Health | RRID: SCR_003070; https://imagej.nih.gov/ij/download.html |
| SmartLab Studio II | Rigaku | https://www.rigaku.com/products/xrd/studio |
| Crystallography Open Database | CrystalEye | RRID: SCR_005874; http://www.crystallography.net/ |
| Other | | |
| Tacta® Mechanical Pipette (P10) | Sartorius | Cat#LH-729020 |
| Tacta® Mechanical Pipette (P20) | Sartorius | Cat#LH-729030 |
| Tacta® Mechanical Pipette (P200) | Sartorius | Cat#LH-729060 |
| Tacta® Mechanical Pipette (P1000) | Sartorius | Cat#LH-729070 |
| Tacta® Mechanical Pipette (P10000) | Sartorius | Cat#LH-729090 |
| SureOne™ Pipette Tips (0.1–10 µL) | Fisher Scientific | Cat#02-707-437 |
| Optifit Racked Pipette Tips (0.5–200 µL) | Sartorius | Cat#790200 |
| Optifit Racked Pipette Tips (10–1000 µL) | Sartorius | Cat#791000 |
| Pipette tips (1–10 mL) | USA Scientific | Cat#1051-0000 |
| Entris® Analytical Balance | Sartorius | Cat#ENTRIS64I-1SUS |
| Practum® Precision Balance | Sartorius | Cat# PRACTUM1102-1S |
| Glass round-bottom flask, (250 mL) | Fisher Scientific | Cat#FB201250 |
| Glass scintillation vials | Wheaton | Cat#986548 |
| PTFE Magnetic Stir Bar (12.7 mm) | Fisher Scientific | Cat#14-513-93 |
| PTFE Magnetic Stir Bar (25.4 mm) | Fisher Scientific | Cat#14-513-94 |
| Direct-Q® 3 UV-R Water Purification System | MilliporeSigma | Cat#ZRQSVR300 |
| Isotemp™ Hot Plate Stirrer | Fisher Scientific | Cat#SP88857200 |
| On/Off Temperature Controller for Heating Mantle | Fisher Scientific | Cat#11476289 |
| Round Bottom Heating Mantle 120V 250 mL | Fisher Scientific | Cat#11-476-004 |
| Isotemp GPD 10 Hot Water Bath | Fisher Scientific | Cat#FSGPD10 |
| Model 5418 Microcentrifuge | Eppendorf | Cat#022620304 |
| Basix™ Microcentrifuge Tubes | Fisher Scientific | Cat#02-682-004 |
| Quartz Cuvette (length × width × height: 10 mm × 12.5 mm × 45 mm) | Fisher Scientific | Cat#14-958-126 |
| Cary 100 UV-visible Spectrophotometer | Agilent Technologies | Cat#G9821A; RRID:SCR_019481 |
| Cu Carbon Type-B Grids, (200 mesh, 97 µm grid holes) | Ted Pella | Cat#01811 |
| Philips CM12 120 kV Transmission Electron Microscope | Philips | RRID:SCR_020411 |
| Miniflex X-Ray Diffractometer | Rigaku | RRID:SCR_020451; https://www.rigaku.com/products/xrd/miniflex |
| Zero-Background Si Sample Holder | Rigaku | N/A |

⚠ CRITICAL: Tin (IV) tetrachloride is a fuming, reactive liquid at ambient conditions; the reagent should be stored in the fume hood and wrapped with Parafilm between uses to ensure minimal exposure to ambient conditions.

⚠ CRITICAL: All other dry reagents (e.g., sodium borohydride, metal precursor salts) should be stored in a desiccator to ensure purity and stability of reagents remains consistent.

**Table 1. Required reagent volumes for citrate-capped Au nanoparticle seeds**

| | Required reagents | | |
|---|---|---|---|
| | Ultrapure $H_2O$ | 10 mM $HAuCl_4$ | 100 mM trisodium citrate |
| Au Nanoparticle Seeds | 58.56 mL | 1.20 mL | 480 μL |

## STEP-BY-STEP METHOD DETAILS

### Synthesis of citrate-capped Au nanoparticle seeds

Timing: 30 min

This preparation describes the synthesis of Au seed using the Turkevich method (Figure 1, Methods video S1 "Synthesis of Au Seeds").[2]

1. Preparation of 10 mM $HAuCl_4$ and 100 mM trisodium citrate precursor solutions:
   a. Carefully weigh 39.4 mg of $HAuCl_4$ trihydrate on an analytical balance into a 20 mL glass scintillation vial. Add 10 mL of ultrapure water to yield a 10 mM solution.
   b. Carefully weigh 58.8 mg of trisodium citrate dihydrate on an analytical balance into a glass scintillation vial. Add 2 mL of ultrapure water to yield a 100 mM solution.
2. Synthesis setup and Au seed synthesis (Figure 1A):
   a. See Table 1 for the exact volume of each reagent solution required.
   b. Transfer the ultrapure water to a clean 250 mL round bottom flask.
   c. Place a clean 25.4 mm PTFE stir bar into the flask and place on the 120 V 250 mL heating mantle resting on a stir plate.
   d. Set the heating mantle and attached heat controller to 138°C and initiate stirring at 640 r.p.m.
   e. Fasten a condenser to the neck of the round bottom flask, turn on water for circulation through the condenser, and ensure all components are securely attached to a ring stand with minimal motion of the reaction setup (Figure 1B).
   f. Once the water is boiling (100°C) and the reaction vessel is visibly at reflux, remove the condenser—leaving the round bottom flask on top of the heating mantle/hot plate.
   g. Directly pipette the 10 mM $HAuCl_4$ solution. Place the condenser back onto the reaction vessel.
   h. Once the reaction system returns to reflux (after approximately 1–2 min), remove the condenser.
   i. Rapidly pipette the 100 mM trisodium citrate solution in a single injection. Re-attach the condenser to the flask and heat/stir for 8 min.

*Note:* Visible color change should gradually occur within about 2 minutes of citrate injection to a dark purple-red solution; this is not a mistake. Visual inspection of the seed solution should yield a bright, transparent burgundy color, as shown in Figure 1C.

⚠ CRITICAL: Ensure the injection of the 100 mM citrate solution occurs as rapidly as possible; do not add slowly or in multiple injections.

   j. After 8 min, carefully remove the reaction vessel from the heating/stirring apparatus. Allow to return to room temperature before proceeding with further reactions or optical characterization.

### Synthesis of bimetallic Au–Sn nanoparticles

Timing: 1 h

This process details the preparation of precursor solutions and the chemical reduction and diffusion of Sn into the Au nanoparticle seeds (Figure 2, Methods video S2 "Reduction of Sn and diffusion into Au seeds"). Detailed below are the 10, 20, 30, and 40% molar equivalent additions of Sn.

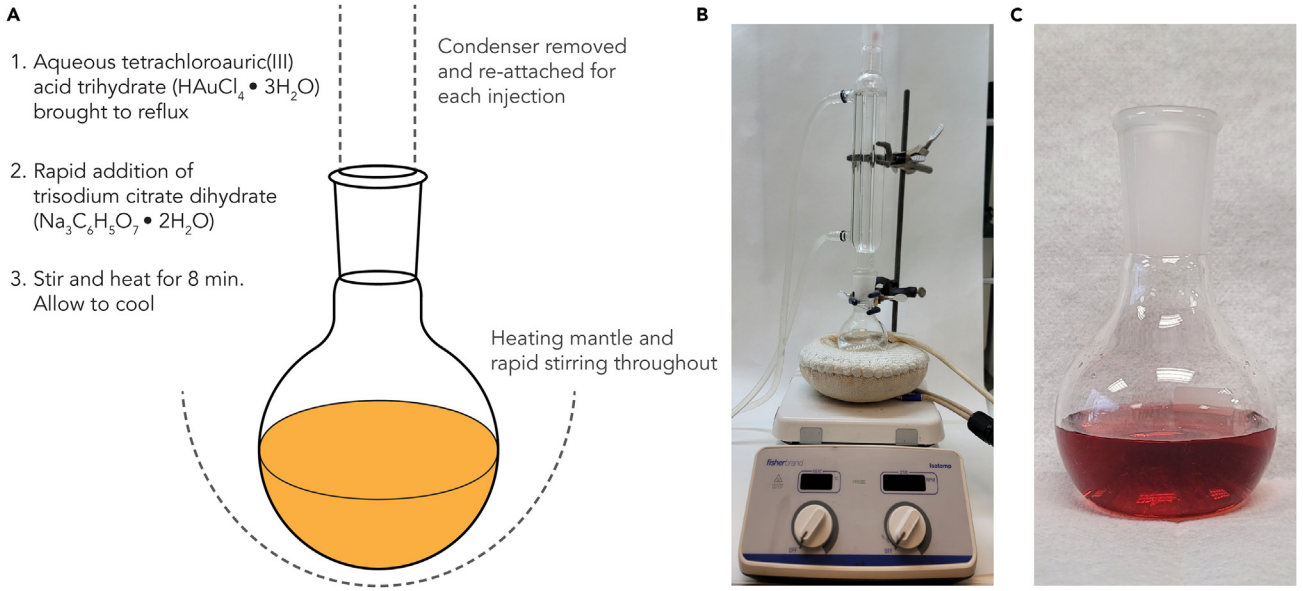


**Figure 1. Illustration of reaction steps, required apparatus features, and Au seed product**

(A) Scheme of sequential synthetic steps and necessary reagents.

(B and C) Photo images of reaction apparatus and expected visual outcomes of the Au seed suspension, respectively.

3. Preparation of 10 wt.% polyvinylpyrrolidone (PVP), 5 mM $SnCl_4$, and 260 mM $NaBH_4$ solutions.
   a. 10 wt.% PVP solution preparation:
      i. Carefully weigh 0.1 g of PVP into a 20 mL clean glass scintillation vial; add 1 mL of ultrapure water and swirl until fully dissolved.

   *Note:* If necessary, sonicate the vial containing the PVP and ultrapure water to expedite the solid dissolving into solution.

   b. 5 mM $SnCl_4$ solution preparation:
      i. Carefully pipette 7.5 mL of ultrapure water into a clean glass vial.
      ii. In a fume hood and using a micropipette, carefully and rapidly add 4.34 μL of $SnCl_4$ to the vial. Swirl until fully dissolved.
   c. 260 mM $NaBH_4$ solution preparation:

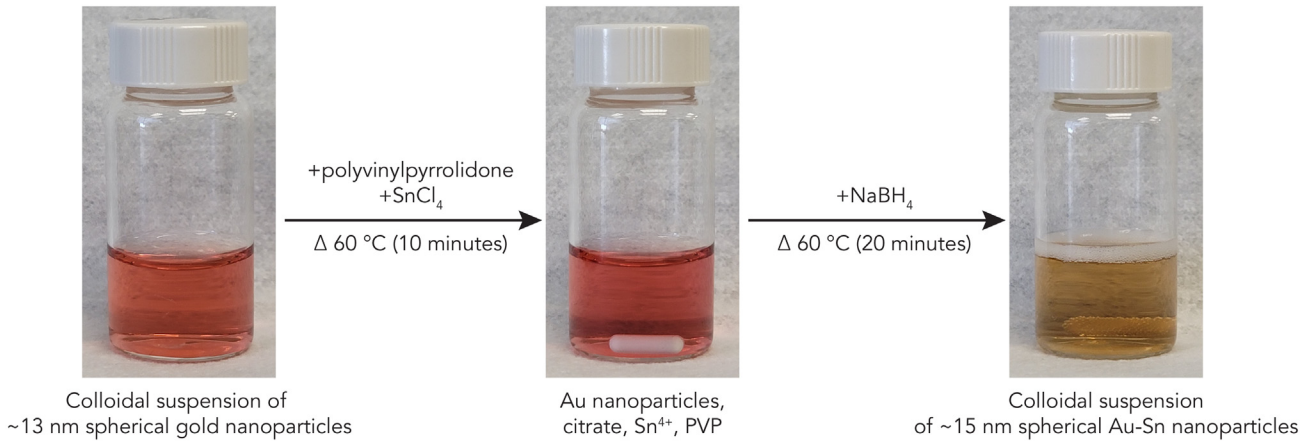


**Figure 2. Photographs of reaction steps and visually observable features**

Photographs of each sequential step of the tin reduction and diffusion into the gold seeds.

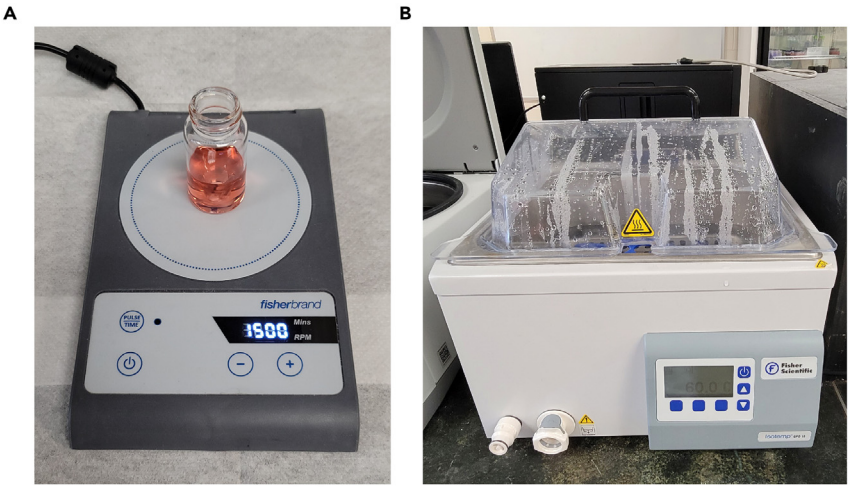


**Figure 3. Photographs of the necessary stirring and heating apparatus**
(A) Stir plate with variable stir controls.
(B) Standard hot water bath with precise temperature control. Scintillation vial holders are used to physically secure vials while heating.

i. Carefully weigh 20 mg of $NaBH_4$ into a clean glass vial.

⚠ CRITICAL: Do not add ultrapure water to the solid $NaBH_4$ until immediately prior to the injection of the 260 mM $NaBH_4$ in the reduction step. Once water is added, the solution should be used as quickly as possible and should not be stored for future use.

ii. Add 2.03 mL of ultrapure water to the vial and swirl until dissolved.

4. Addition of Sn to Au seeds to form bimetallic nanoparticles (Figure 2).
   a. To a clean glass vial, pipette 6 mL of as-synthesized Au seeds, water, and add a 12.7 mm PTFE stir bar.
   b. Add vials back to the stir plates (Figure 3A) and begin stirring at 1,500 rpm. See Table 2 for the exact volume of each precursor solution required.
   c. While solutions are stirring, pipette the 10 wt.% PVP solution into the vial.
   d. Add the 5 mM $SnCl_4$ solution to the vial; one can inject this solution immediately after addition of PVP to the vials.
   e. Remove the reaction vials from the stir plates and cap.
   f. Place the entire capped vial into a hot water bath at 60°C for 10 min (Figure 3B). The stir bar does not need to be removed from the reaction setup during this step.
   g. After 10 min, uncover the vials and place back on to the stir plates and resume stirring.
   h. To the vials, pipette the freshly prepared 260 mM $NaBH_4$. Upon dispensing of the reduction agent, the solution should visually change to a translucent, homogeneous yellow-orange color with residual bubble formation on the top of the suspensions. Allow this solution to stir for 30 s.
   i. Again, remove the entire reaction vial from the stir plates, cover, and place them into the hot water bath at 60°C for 20 min.

*Note:* Leave the reaction vials with slightly loosened caps during the 20-minute heating step (in the hot water bath) to allow for gas evolution from the reduction process.

   j. Remove the vials from the 60°C hot water bath.
   k. Remove the 12.7 mm PTFE stir bars from the sample solutions, with the assistance of another stir bar, upon removal from the hot water bath.

**Table 2. Required Reagent Volumes for synthesis of bimetallic Au–Sn nanoparticles**

| | Required reagents | | | | |
|---|---|---|---|---|---|
| | Au seeds | Ultrapure $H_2O$ | 10 wt.% PVP | 5 mM $SnCl_4$ | 260 mM $NaBH_4$ |
| 10% Au–Sn Addition | 6 mL | 1.82 mL | 31.89 μL | 26.56 μL | 153.23 μL |
| 20% Au–Sn Addition | 6 mL | 1.77 mL | 35.87 μL | 59.76 μL | 172.39 μL |
| 30% Au–Sn Addition | 6 mL | 1.70 mL | 41.00 μL | 102.45 μL | 197.01 μL |
| 40% Au–Sn Addition | 6 mL | 1.61 mL | 47.83 μL | 159.36 μL | 229.85 μL |

l. Allow colloidal suspensions to return to room temperature before characterizing.

⚠ CRITICAL: A minimal color change occurs during the second heating, but importantly they should remain fully homogeneous colloidal suspensions with no visible flakes or precipitation.

## Optical characterization of plasmonic bimetallic nanoparticles

Timing: 30 min

This process describes the process required to optically characterize the synthesized bimetallic nanocrystals to effectively understand localized surface plasmon resonance (LSPR). Specifically, multichannel UV-visible spectroscopy will be used.

5. Perform background and blank correction using ultrapure water pipetted directly into the quartz cuvette.
6. Samples prepared in Step 4, can be pipetted directly into a clean 1 cm path-length quartz cuvette without dilution or concentration of the colloid to obtain the UV-visible spectrum.
7. Measure the UV-visible spectrum over a range of 200–700 nm and ensure data is appropriately saved on your device.

## Structural characterization of bimetallic Au–Sn nanoparticles

Timing: 3 h

Characterization of crystalline features, size distribution, and nanoparticle morphology will be described through the use of XRD and TEM. This protocol describes the sample preparation, instrumentation settings, and steps required for the analysis using a powder X-ray diffractometer and transmission electron microscope.

8. Powder XRD sample preparation and characterization:
   a. Pipette samples into 1.5 mL microcentrifuge tubes and place into centrifuge.
   b. Centrifuge samples at 8,000 r.p.m. (5,510 r.c.f.) for 8 min.
   c. After centrifugation carefully remove supernatant and leave the pellet.
   d. Re-suspend the pellet into solution by adding 1.5 mL ultrapure water. Ensure sample is evenly distributed by inverting the microcentrifuge tubes.
   e. Place the washed samples back into the centrifuge for a second centrifugation at 8,000 r.p.m. (5,510 r.c.f.) for 8 min.
   f. Carefully remove the supernatant again, leaving only the concentrated sample in each microcentrifuge tube.
   g. Pipette the concentrated sample onto a zero-background silicon background holder for the Rigaku Miniflex X-ray diffractometer.
   h. Allow samples to visibly dry by placing in a desiccator uncovered.

**Table 3. Nanoparticle size distributions**

| Sample | Diameter (nm) | Standard deviation (nm) | Coefficient of variation (%) |
|---|---|---|---|
| Au Seed | 13.3 | ±1.1 | 8.3 |
| 10% Sn Added | 13.3 | ±1.0 | 7.6 |
| 20% Sn Added | 13.6 | ±1.4 | 10.6 |
| 30% Sn Added | 14.4 | ±1.0 | 7.1 |
| 40% Sn Added | 15.8 | ±1.4 | 9.0 |

i. Place samples onto the holders in the Rigaku Miniflex (utilizing Cu $K_\alpha$ X-ray source with 1.54 Å wavelength) and measure the diffraction pattern at a scan rate of 1° $min^{-1}$ in the 2θ range 10–90°.
j. Ensure all data is saved for future analysis.

9. TEM sample preparation and characterization:
   a. Pipette samples into 1.5 mL microcentrifuge tubes and place into centrifuge.
   b. Centrifuge samples at 8,000 r.p.m. (5,510 r.c.f.) for 8 min.
   c. After centrifugation, carefully remove the supernatant and leave the concentrated sample at the bottom.
   d. Re-suspend the concentrated sample back to 1.5 mL by adding ultrapure water. Ensure sample is evenly distributed by inverting the microcentrifuge tubes.
   e. Place the washed samples back into the centrifuge for another centrifugation at 8,000 r.p.m. (5,510 r.c.f.) for 8 min.
   f. Carefully remove the supernatant again, leaving only the concentrated sample in each microcentrifuge tube.
   g. Pipette 10 μL of the concentrated sample onto a Ted Pella Cu Carbon Type-B TEM grid.
   h. Allow samples to dry completely in a desiccator uncovered for approximately 2 h.
   i. Bimetallic nanoparticles were imaged using a 100 kV accelerating voltage and ImageJ software for size and polydispersity analysis.
   j. Ensure all imaging and data is saved appropriately for future analysis.

## EXPECTED OUTCOMES

The optical and structural characterization methods provided in this protocol describe the quantification and analysis of post-transition metal diffusion into the prepared Au seeds.

With the Au seed synthesis, spherical, monodisperse Au nanoparticles of ~13 nm are expected. See Table 3 for more detailed information about sample particle size information. Upon addition of Sn, nanoparticles should remain spherical but grow slightly in diameter to ~15 nm (Figure 4A). As-synthesized, the Au seed colloid should show a distinct, asymmetric absorption peak at ~520 nm with an extinction maximum of ~0.7 that corresponds with the LSPR. Upon the addition of Sn, this peak will blue-shift towards shorter wavelengths, resulting in a color change from red to orange to tan-brown (Figure 4B). As more Sn is added, continued blue-shifting is observed with broadening of the LSPR. With additions of 10, 20, and 30% Sn, the expected LSPR maxima are ~514, 502, and 475 nm. For 40% Sn added, the LSPR is almost entirely damped, with a faint peak ~470 nm (Figure 4B). The as-synthesized Au–Sn colloids have lower intensity extinction with increasing amounts of Sn added because they have lower extinction coefficients compared to the pure Au nanoparticles.[1]

To understand the crystal structure of the nanoparticles, XRD is used. It is important to note that XRD of nanomaterials can be difficult to interpret compared to bulk crystallographic analysis. For example, due to their small diameter significant broadening in the diffraction peaks will be observed which can make peak differentiation challenging and decreased signal to noise is also expected for nanomaterials.[3] To index the observed peaks, the Open Crystallographic Database is used to generate reference stick patterns for face-centered cubic (FCC) gold (9013036), trigonal $Au_5Sn$

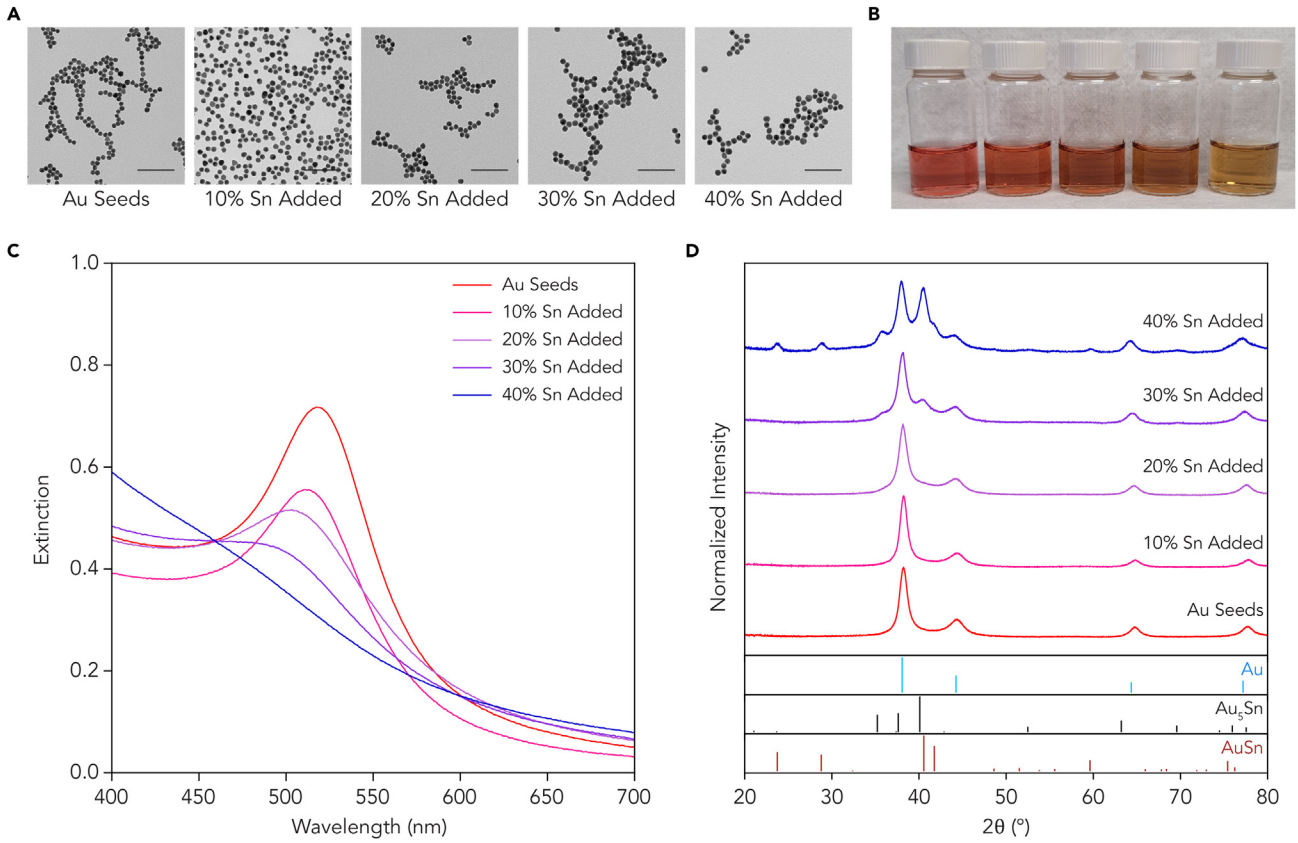


**Figure 4. UV-visible, powder X-ray diffraction, and transition electron microscopy characterization**

(A) TEM imaging of Au–Sn bimetallic nanoparticles compared to Turkevich Au seeds.

(B–D) (B) Optical image of Au–Sn nanoparticles in scintillation vials following Sn reduction (C) UV-visible spectroscopy of Au-Sn nanoparticle solutions. (D) Powder XRD diffraction of Au–Sn nanoparticles. Scale bars are 100 nm.

(1510571), and hexagonal AuSn (1510301) at the bottom of Figure 4D. The XRD pattern for Au seeds should yield peaks that correspond only to FCC Au, with a primary peak at ~38° that corresponds to the (111) reflection. With 10% Sn added, no additional peaks are observed, though shifting of the (111) is observed which is indicative of substitutional alloying.[1,3] This shifting is expected to continue linearly with increasing incorporated Sn amounts, approximately following Vegard's Law.[1,3] At 20% Sn added, a faint reflection at ~40° can be seen which corresponds to $Au_5Sn$ intermetallic. More $Au_5Sn$ peaks can be seen at 30% and 40% Sn added, with AuSn intermetallic peaks appearing at 40% Sn added.

## LIMITATIONS

The primary limitation of this protocol is in the scalability of the synthesis for high reaction yield. In a typical synthesis, 8 mL of Au–Sn nanoparticles can be prepared easily with stability longer than 2–3 weeks at room temperature.[1] Importantly, this synthesis has been optimized for 4–8 mL solution preparations and has not been systematically investigated for larger volumes. Increasing the reaction volume will begin to alter how steps like heating and rapid dispersion of reducing agent impact the final Au–Sn products and their stability. In addition, cleaning of glassware or reaction vessels requires the use of aqua regia which entails the handling of strong, oxidizing acids; this is necessary for the dissolution of gold and extreme caution must be exercised. Seed size can also greatly influence the outcomes of Sn incorporation, and subsequent LSPR position and breadth. The seed size can be most accurately confirmed with the samples using TEM, where average seed and sample size distributions can be measured to ensure expected LSPR and phase behavior. The extent of Sn

incorporation has not been systematically studied above the amounts described herein, which may limit the upper thresholds of UV LSPR tunability and functionality.[4,5] Lastly, it can be difficult to quantify the metal content in each nanoparticle, as added metal precursor amounts are only an estimate of the true incorporated amount. Detailed quantification of metal content is a difficult and laborious process that can be carried out via energy dispersive X-ray spectroscopy (EDS) or inductively coupled plasma mass spectrometry / optical emission spectroscopy (ICP-MS/OES). Detailed methodologies and approximate incorporation amounts for added metal content are reported in the associated manuscript.[1]

## TROUBLESHOOTING

### Problem 1

Formation of non-uniform, inconsistent or large Au seeds during the synthesis.

### Potential solution

Here, if experimenters are not consistently injecting the trisodium citrate rapidly, aggregation or inconsistent size and/or dispersity of nanoparticles in suspension may occur. Also, ensure the use of fresh reagents, particularly trisodium citrate, which should be prepared as needed and not in advance. Additionally, we recommend thoroughly re-cleaning any glassware or stir bars with aqua regia and rinsing all cleaned glassware with ultrapure water prior to use, as trace contaminants can alter the structure of the gold seed nanoparticles.

### Problem 2

Inconsistency in the blue-shifting of Au–Sn nanoparticle solutions compared to expected values.

### Potential solution

The freshness of the $SnCl_4$ reagent can significantly modify the rates of tin integration into the bimetallic nanoparticles. To limit variability in the synthesis of Au–Sn nanoparticles, ensure that after opening the bottle of $SnCl_4$ reagent, the bottle is capped. Furthermore, to ensure the quality of the reagent for future use, one should wrap the outside of the cap with Parafilm and store in the desiccator to prevent any possible degradation of the reagent.

### Problem 3

Formation of nanoparticle aggregates during Au or Sn reduction, which can be seen as visible precipitation or flakes in solution, a purple solution color with a significantly red-shifted and broad LSPR for the Au seeds (> 525 nm), or as a darkened, gray solution color with similarly red-shifted and broad LSPR and/or disappearance of the LSPR for Au–Sn nanoparticles.

### Potential solution

To remedy aggregation, we recommend re-cleaning glassware and stir bars for Au Seed preparation and cleaning stir bars for Au–Sn nanoparticle preparation with aqua regia. Care should be taken to thoroughly rinse any aqua regia cleaned equipment with ultrapure water to remove any acid contamination. If aggregation persists during Au Seed synthesis, another source of contamination could be a dirty / contaminated condenser column that is dripping back into the solution. Similar cleaning and rinsing precautions should be taken for this apparatus.

### Problem 4

The formation of a broad and red-shifted LSPR peak (> 520 nm), instead of an LSPR peak between 475-520 nm, is observed in the extinction spectrum.

### Potential solution

The observation of an LSPR red-shifted beyond 520 nm is indicative of a population of multi-nanoparticle aggregates that formed in solution. These can be seen at higher Sn contents, when stir bars are contaminated, and/or an old $SnCl_4$ stock solution is being used. To avoid this, stir bars should be

cleaned with aqua regia and thoroughly washed with ultrapure water prior to use. Additionally, the $SnCl_4$ precursor should be relatively fresh (< 1 year old) and after use should be capped, wrapped in Parafilm, and stored in an appropriate desiccator.

### Problem 5

Low signal is seen by XRD and/or new diffraction peaks are seen in the XRD pattern that are not described in the expected outcomes.

### Potential solution

It is important to note that resulting XRD pattern will vary for identical samples if a different X-ray source is being utilized. For our analysis, a Cu $K_\alpha$ X-ray source with 1.54 Å wavelength was used which determines the location for specific diffraction peaks; a different XRD source will have different expected diffraction peaks. If unexpected peaks are observed despite identical sources and settings, a possible cause is a scratch in the sample holder which is diffracting. This can be tested by measuring XRD on the blank sample holder to see if the same peak is observed. If the sample holder is scratched, it may be able to be cleaned or polished, though typically a new sample holder must be used. In the case that diffraction patterns have very low signal, this can be attributed to the low signal-to-noise achievable in nanomaterial diffraction.[3] To increase the signal, the loading of the sample can be increased from 8 mL to 16 mL of concentrated sample onto a sample holder. Additionally, the scan rate can also be slowed from $1^\circ$ $min^{-1}$ to decrease noise, though this will increase the collection time.

### Problem 6

Phase compositions are observed in the XRD patterns that differ from expected outcomes.

### Potential solution

If unexpected intermetallic peaks are observed at a given Sn content, it is most likely due to a slight variation in the temperature of the synthesis. It is critical that solutions heat in a water bath at a set temperature to minimize variation in temperature, as small temperature differences can greatly impact the resulting phase composition. Additionally, if $NaBH_4$ is not rapidly injected into the solution, varying phase compositions can be expected due to slower reduction of $Sn^{4+}$.

## RESOURCE AVAILABILITY

### Lead contact

Further information and requests for resources and reagents should be directed to and will be fulfilled by the lead contact, Michael Ross (Michael_Ross@uml.edu).

### Materials availability

This study did not generate new unique reagents.

### Data and code availability

All experimental data are available upon reasonable request.

## SUPPLEMENTAL INFORMATION

Supplemental information can be found online at https://doi.org/10.1016/j.xpro.2023.102410.

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## AUTHOR CONTRIBUTIONS

All authors contributed to the writing of this manuscript.

## DECLARATION OF INTERESTS

The authors declare no competing interests.

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