

Introducing Solid State Chemistry and Nanoscience with Colloidal Au–Sn Alloying

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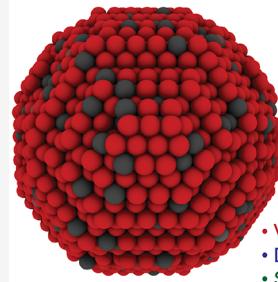
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Supporting Information

ABSTRACT: Integrating active research into educational laboratory experiences is critical for exposing students to the impact research can have. By using discoveries and experiments from ongoing active research, these laboratories create a crucial early career opportunity for students to directly engage in current scientific research. A summer laboratory program for incoming undergraduate chemistry majors was designed and implemented. Students with no required prior laboratory experience took part in experiments and discussions relating to Au–Sn alloy nanoparticles, focusing on concepts from nanoscience, solid state chemistry, and photonics. Societal impacts on sustainability and critical materials were also discussed. A strong emphasis is placed on teaching solid state chemistry and nanoscience, which can be challenging to incorporate into existing introductory laboratory experiences due to time, safety, and approachability limitations. This experiment is aqueous, can be performed in a single session, and requires no sophisticated equipment. The laboratory was reproduced solely using reagents and equipment available in our general chemistry stockroom. Instructor materials are also included that adapt this laboratory for teaching crystal structures and X-ray diffraction at an advanced level.

KEYWORDS: Nanoparticles, solid state chemistry, alloying, X-ray diffraction, plasmonics, optical properties, materials science, sustainability, critical materials

Colloidal Au–Sn Alloying: Nanoscience and Solid State Chemistry



- Visual and Quantitative
- Directly Adapted from Research
- Sustainability and Critical Materials

INTRODUCTION

A challenge in undergraduate laboratory education is to integrate ongoing research into curricula.¹ Typically, laboratory experiments are selected from a set of exercises that emphasize key content and skills from coursework. While important and well-developed, these exercises are often far removed from modern research activity.² This is particularly true in multidisciplinary areas such as solid state chemistry and nanoscience, which typically require distinct safety protocols, complex equipment, and skills that differ from those in most curricula. As such, laboratory experiments that integrate these emerging research concepts into the frameworks of both introductory and upper-level chemistry laboratory classes are needed.

Education research has consistently shown that providing students with early exposure to research themes and concepts effectively communicates the ongoing nature of scientific inquiry and continuous knowledge creation^{1,2} while improving outcomes and closing achievement gaps between groups.^{3,4} Highlighting the impact of modern research on societal challenges and interdisciplinary areas of science can also improve both learning outcomes and performance on assessments.^{5,6} While current literature examples can be used to bolster the relevance of curriculum in the classroom,

incorporating research into the laboratory is more difficult due to technical and logistical challenges. Specifically, experiments are needed that can be done within the framework of a periodic time-constrained course meeting, have easily achievable safety standards, remain cost-effective, and relate existing concepts with broader societal goals.

Here, we present a laboratory experiment that can be adopted for both introductory and upper-level courses to introduce concepts from solid state chemistry and nanoscience (Figure 1). The experiment describes a straightforward procedure for alloying Sn into Au nanoparticle seeds that can be completed in a single few-hour laboratory meeting. It is adapted from our recent work focused on the discovery and fundamental characterization of noble metal-post-transition metal alloy nanoparticles.^{7,8} While other aqueous metal alloy nanoparticle laboratory experiments have been developed, this work differs in two important ways.^{9,10} First, it introduces the

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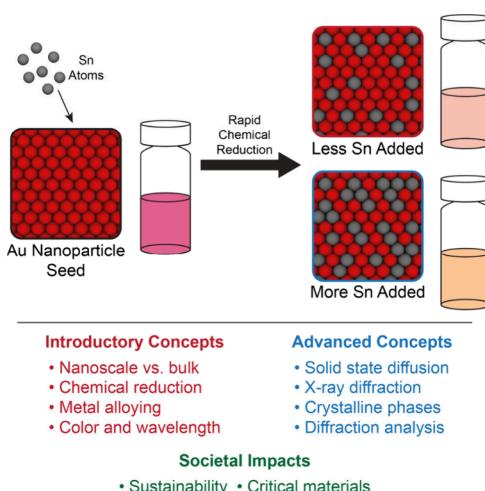


Figure 1. Using nanoscale diffusion of Sn into Au nanoparticles to introduce concepts in nanoscience, solid state chemistry, and sustainability.

use of a nonprecious metal (Sn) in a simple synthesis, providing an avenue to introduce and discuss sustainability and precious metals. Second, the synthesis and experiment is highly robust to common nanoscience pitfalls: the instructor notes detail how the laboratory experiment was reproduced using only reagents and materials available in our general chemistry stockroom.

While the primary scientific learning outcomes focus on nanoscience, alloying, crystals, and X-ray diffraction, there is a dual emphasis on societal goals relating to sustainable materials. At the introductory level, these experiments can be tailored to emphasize nanoscience and observable optical phenomena. At the upper level, they can be designed to focus on crystal structures and powder X-ray diffraction. These experiments have been successfully performed by students with no prior laboratory experience as part of a summer launch program, *CatalyzeUML*. Lab handouts, assessments, and instructor notes are included as *Supporting Information*.

EXPERIMENTAL SECTION

The laboratory experience was divided into four sections that can be spread over one or two laboratory sessions as needed (Table 1). The first three sections stand alone as an

Table 1. Timeline of Activities

Activity	Time Needed
Preparation of materials and reagents	2–3 h
Synthesis of Au nanoparticle seeds (optional)	30 min
Colloidal diffusion of Sn in Au seeds	1 h
Optical characterization by UV–visible spectroscopy	30 min
Structural analysis by XRD and TEM (Advanced, optional)	3 h

introductory nanoscience and sustainable materials laboratory experience while the last section introduces structural characterization and X-ray diffraction and is more suitable for an upper-level course. Each section introduced new and distinct concepts with discussion and visual observations throughout. The initial discussion revolved around how nanoparticles have distinct properties from the bulk and how their properties change with size.¹¹ Societal challenges in sustainability and materials scarcity were also included.

Students brainstormed and discussed what they knew about Au and Sn, material scarcity, how we reduce the use of precious metals, and how materials are important for sustainability. The instructor then led a short discussion on the applications and usage of precious metals, with a focus on Au nanoparticles and their applications in COVID-19 tests and CO₂-recycling catalysts.^{12–16} Finally, the experiment was introduced. Student handouts and instructor notes for facilitating discussion, introducing micropipette use (Figure S1) and nanoparticle light absorption (Figure S2) are included in the supporting documents.

Synthesis of Au Nanoparticle Seeds

This step involved the synthesis of 13 nm spherical Au nanoparticles to be used as seeds.¹⁷ Briefly, ultrapure water was massed into a round-bottom flask along with tetrachloroauric acid, a condenser was attached, and the solution was brought to reflux. Then, sodium citrate was rapidly injected. Due to time constraints and available equipment, this was prepared ahead of time with the synthesis shown by video, though it can also be performed as a group.⁷ Students noted color changes through the nucleation and growth process. Discussion of observed differences both over time and compared with bulk Au was led by the instructor. Further elaboration was done to explain how complementary colors are absorbed and/or reflected by materials and how it may hint at how the material is absorbing and/or transmitting light. The gold seeds are stable in solution over long periods (months-to-years); excess seeds can be set aside for future use.

Colloidal Diffusion of Sn into Au Seeds

The second experiment introduced metal reduction and solid state diffusion of Sn into Au seeds.¹⁸ Here, students synthesized Au–Sn alloy nanoparticles with varying compositions and noted changes in color and reactivity during the Sn reduction and diffusion process.¹⁹ First, students prepared a solution for Sn diffusion that included Au seeds, polyvinylpyrrolidone (PVP), and tin tetrachloride (SnCl₄, prepared fresh). This solution was stirred after each addition and heated in a water bath at 60°C for 10 min. After this, freshly prepared sodium borohydride (NaBH₄) was added to reduce the Sn⁴⁺ to metallic Sn⁰. After stirring for 1 min, the vial was placed back into a water bath at 60°C for 20 min. Students were instructed along the way to note any visual changes or bubbles. To generate a range of outcomes and results, students could either be individually instructed to add two different amounts of Sn in two separate vials (can be performed in parallel) or students could each be assigned a specific Sn-added amount, with their results combined for analysis (10–50% Sn-added). Representative data for these amounts can be found in our previous reports and open access protocol.^{7,8}

Optical Characterization by UV–visible Spectroscopy

Students were then introduced to the importance of size and properties at the nanoscale during the synthesis of the Au nanoparticle seeds.⁸ Here, the emphasis was on solid state diffusion and changes in color based on composition. A discussion was led first on the visual differences between the starting Au seed nanoparticles and those with varying Sn amounts incorporated, as well as the diffusion mechanism to create Au–Sn alloy nanoparticles. Next, students measured UV–visible spectra and learn how to plot their data using graphical software (e.g., Microsoft Excel) to compare data with other students. Students then compared their visual observa-

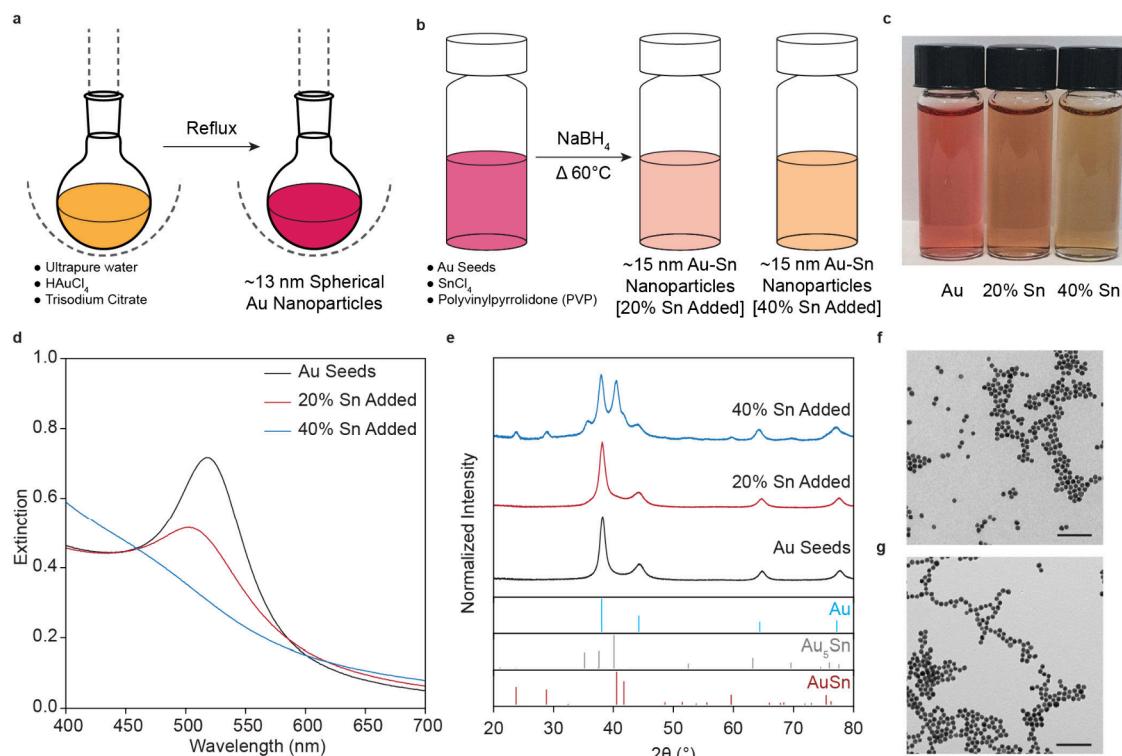


Figure 2. Experimental flow and typical results (a). Synthesis of spherical ~ 13 nm Au seed nanoparticles (b). Reduction and diffusion of Sn into Au seeds. Photographs (c), extinction spectra (d), and X-ray diffraction patterns (e) of Au, 20% and 40% Sn added colloidal nanoparticle solutions. Micrographs of Au seeds (f) and 20% added Sn nanoparticles (g). Scale bars are 100 nm.

tions with those from the UV-visible spectra, and the concepts of color absorbance and appearance were discussed within the context of the UV-visible spectra. Then, in smaller groups, students compared data and discussed how observations changed with respect to the amount of Sn added.

Structural Analysis by X-ray Diffraction and Electron Microscopy

A final experiment focused on the structural changes that occur in the nanoparticles throughout the growth and alloying processes. For our introductory laboratory experience, this was introduced using representative data where the experiments were performed by other students as an instructive demonstration. However, a goal of this manuscript is to present sufficient detail that this could be adapted for an upper level course. For an upper level course, differences in crystal structure would first be discussed using unit cell models in free-to-use databases and software such as the Materials Project or VESTA.^{20,21} Nanoparticle size and shape would be discussed using transmission electron microscopy (TEM) micrographs. Students would then be instructed to collect X-ray diffraction (XRD) patterns from the Au nanoparticle seeds and for samples with 20 and 40% Sn added. After collection, the patterns would be plotted and discussed within the context of different unit cells that could form (these are provided to the students). As a conclusion, students would then discuss solid state diffusion and how it could lead to the formation of different crystalline phases.

HAZARDS

Safety goggles, nitrile gloves, and a laboratory coat should be worn during all parts of the experiment. Students should be instructed to review the safety behavior guidelines before the

beginning of the experiment. Specific noteworthy hazards include the use of nanomaterials, the use of tin tetrachloride (SnCl_4), the use of sodium borohydride (NaBH_4), and X-ray instrumentation use. The hot plate and water baths present both heating and electrical hazards and should be treated with caution. Colloidal metal nanomaterials should be treated as hazardous as their hazards are largely unknown, requiring the use of personal protective equipment (PPE). Tin tetrachloride fumes in moist air and presents an inhalation, ingestion, and dermal health hazard. It should only be used in a fume hood prior to dilution in water and, if necessary, the instructor can perform the initial dilution step or use alternative Sn sources as described in the instructor materials. Sodium borohydride is a strong reducing agent that can be an eye and skin irritant. While small (mg) amounts are used in this work, care should be taken prior to dilution. Tetrachloroauric acid should only be handled as needed using proper PPE as it can cause burns, serious eye damage, and may cause sensitization by skin contact. If aqua regia is used for cleaning glassware by the instructor, **extreme caution should be used as it is highly caustic**. The preparation should be done in small volumes (10 mL or less) due to the heating upon mixing, and disposal should be performed in a separate acid waste container, **which should never be capped tightly** to avoid any pressure buildup from the gaseous Cl_2 , NOCl , or NO_2 that may be evolved. All glassware cleaned with aqua regia should be triple rinsed prior to use and their waste should be discarded in the aqua regia waste container. Finally, in laboratories that allow students to perform X-ray diffraction, proper radiation training and precautions should be taken. All solids and solutions should be disposed of following proper local health and safety guidelines.

RESULTS AND DISCUSSION

The experiments were conducted by a total of 19 incoming undergraduate chemistry majors at University of Massachusetts Lowell as part of the *CatalyzeUML* onboarding and launch program. Two cohorts of students participated: nine in the first year and 10 in the second year. Cohorts of students were admitted to this elective program before the beginning of their freshman year. Over the course of two days, students worked either in pairs or groups of three under the supervision and guidance of experienced undergraduate students and by a faculty member. All experiments were performed in half-day increments, generally in a 3–4 h continuous period. These are designed such that the activities and modules can be substituted, prepared in advance, or fully completed by the students depending on their experience level and time constraints. For example, during *CatalyzeUML*, the Au nanoparticle seeds were prepared ahead of time and students were shown a demonstration video of the synthesis. On day one, a laboratory discussion introduced nanomaterials, their distinctiveness from bulk materials, and general observations relating to their properties and importance. Day two focused on characterization and instrumentation, and students were introduced to the importance of tools and measurements in chemistry. While these experiments can be modified to adapt to the students' experience, these were performed by students with *no required laboratory experience beyond the high school level*.

The objective of this project was to introduce real laboratory research to incoming students, providing them with hands-on discovery and inquiry experience distinct from typical introductory laboratories. There were three main conceptual and technical goals of these experiments, with an additional optional learning objective for advanced laboratory coursework. The first was to introduce the unique properties of nanoscale materials and their distinctiveness from the bulk. The second was the role of composition at the nanoscale and the observable differences that occur when it changes. The third was to highlight concepts relating to sustainability including precious metals as well as catalysis. For advanced students, a fourth concept that can be introduced is atomic ordering in crystal structures, the mechanism of solid state diffusion, and their characterization by X-ray diffraction (XRD).

The first experiment involved the synthesis of ~13 nm Au nanoparticles to be used as seeds (Figure 2a). These were synthesized using the well-established Turkevich method for synthesizing sodium citrate sols.¹⁷ These nanoparticles were synthesized before the students participated due to time constraints and a video of the synthesis was played and discussed.⁷ If time permits, students could perform this synthesis in one or two large groups. Detailed methods can be found in the *Supporting Information*. Students were instructed to watch and record the color changes over the course of approximately 10 min. Their observations were shared and the role of size at the nanoscale was discussed. The instructor then explained that the color change from the initial pale yellow to brown, purple, and finally burgundy is indicative of two steps. The initial change from yellow to brown was the reduction to Au^0 , while the subsequent changes from dark brown to burgundy was indicative of nanoparticle growth. It was then emphasized that these size-dependent properties are a key attribute of nanomaterials.

The second experiment emphasized the composition-dependent optical properties of the nanoparticles (Figure 2b). First, the idea that solids are mobile and can diffuse was introduced, within the context of liquid diffusion and then compared with solid solutions (alloys). The chemical reaction was written out and discussed, namely, the reduction of Sn^{4+} with NaBH_4 to Sn^0 (alloyed). To prepare for the reaction, students were instructed to prepare a scintillation vial with Au seeds, polyvinylpyrrolidone (PVP), and ultrapure water. Dilute solutions of tin tetrachloride in water should be prepared in the fume hood. Whether these should be prepared by students, teaching assistants, or the faculty is up to experience. Next, the tin solution was added to the reaction vessel and the vial was heated to 60°C. While heating, students individually massed sodium borohydride, such that it could immediately be diluted and added to the solution once it came to temperature. After the sodium borohydride injection, students were instructed to note any visual changes in the solution over time. Important observables include bubbling and color change. Students performed the reaction for two different amounts of added Sn (20 and 40%) in parallel to investigate how the optical response changed as a function of Sn content. To ensure consistency between syntheses and student groups, seeds were always used at ~0.7 O.D. (for a 1 cm optical path length) as detailed in the procedure.

The third experiment involved the characterization of the nanoparticles with a specific focus on relating observed color with measured optical properties (Figure 2c). First, a discussion of light, color, and wavelength was led. Then, students shared their observations from the synthesis, focusing on aspects such as “Did the color change?”, “When did the color change?”, “What do we hypothesize this means related to wavelength?” Students then used a UV–visible spectrometer to measure the spectrum of the as-synthesized nanoparticles. Their predictions were then compared with the measured UV–visible spectra (Figure 2d).

In an advanced class or as part of a learning demonstration, further characterization can also be included (Figure 2e–g). Powder X-ray diffraction (XRD) can also be included given sufficient facilities. The Au–Sn nanoparticles form intermetallic Au_5Sn and AuSn phases when alloyed with Sn. This provides a hands-on demonstration of how crystalline phases change and how this manifests in XRD patterns. In our demonstration, students were introduced to structure with visualization of the different unit cells. The peaks in the XRD were shown for the different phases (Figure 2e), and the presence of Au_5Sn in 20% Sn added nanoparticles and the presence of AuSn in the 40% Sn added nanoparticles was discussed. For an advanced laboratory, students would be instructed to tabulate the XRD reflections, compare them as a function of Sn composition, and form hypotheses relating to which intermetallic phases formed and why. During *CatalyzeUML*, trained graduate students demonstrated how the nanoparticles can be imaged using transmission electron microscopy (TEM) with a guided introduction to the facility (Figure 2f, g).

In all instances, students were successfully able to synthesize Au–Sn alloy nanoparticles that showed the anticipated properties. Specifically, the LSPR of the nanoparticles shows a blue-shift upon the reduction of Sn that is consistent and measurable. Importantly, when students added different amounts of Sn (20 and 40%), there was a systematic change in the LSPR such that more Sn added leads to a larger blue-

shift. This is consistent with the original report of the synthesis and is seen in the average LSPR maxima (Table 2).⁸

Table 2. Average Experimental Results^a

Sample	LSPR Maximum (nm)	Nanoparticle Diameter (nm)
Au Seed ^a	518	13.8 ± 1.5
20% Sn Added	501 ± 4.2	14.8 ± 1.8
40% Sn Added	458 ± 13.7	14.6 ± 2.0

^a*n* = 10. Synthesized before the experiment. *n* = 19 for the 20 and 40% Sn-added samples.

Representative XRD showed the composition-dependent formation of intermetallic phases. The Au seeds exhibit a characteristic face-centered cubic (fcc) Au diffraction pattern with a primary (111) reflection at $\sim 38^\circ 2\theta$. For 20% Sn added, some broadening and shifting of the primary diffraction peak is observed. For 40% Sn added, new peaks emerge on both sides of the (111) fcc peak indicative of the formation of trigonal Au_5Sn intermetallic. Size analysis performed on TEM micrographs show a reasonable 10–15% polydispersity (Table 2).

A post-laboratory assessment asks students to compile and analyze their data, including UV–visible plots, a table of visual observations, and scientific justification for the laboratory. The visual nature of the experiments reinforces the use of observation in integrating hypothesis formation and quantitative analysis. In post-lab discussion, the student cohort of *CatalyzeUML* performed group presentations describing their experimental experiences. Overall, the goal of this laboratory was to introduce new scientific concepts such as nanoscience and solid state chemistry through real research experience on modern materials. By introducing these experiments as part of ongoing research, this laboratory experience provided insight into the scientific method and the process of discovery.

■ TECHNICAL LEARNING OUTCOMES

The specific learning outcomes of this experiment include new conceptual areas, specific technical skills, and insights into the research and discovery process.

1. Students will be able to describe how the physical and chemical properties of nanomaterials are distinct from those of bulk materials.
2. Students will be able to explain the role of size at the nanoscale and its influence on physical properties, including light absorption.
3. Students will be able to relate the visual appearance of a material to its UV–visible spectrum, relying on detailed observations over the course of an experiment.
4. Students will be able to articulate how modern research is performed and the importance of concepts in sustainability, nanoscience, and catalysis.
5. Students will be able to describe how diffusion can occur in solids [upper level].
6. Students will be able to describe differences in unit cells and crystalline structure [upper level].
7. Students will be able to recognize differences in X-ray diffraction patterns and describe their physical importance [upper level].

■ CONCLUSION

These experiments allow students to experience research without laboratory experience beyond the high school level.

Specifically, they introduce concepts relating to nanoscience, light, and sustainability through highly visual experiments that can be completed in one laboratory session. The experiments are performed in water, with few hazardous materials, and with limited required equipment. Pre- and post-lab discussion and data analysis highlight how students were able to generate consistent data. These data introduce multiple solid state and nanoscience concepts and rely on relating visual observations with quantitative analysis. While these experiments were implemented during a summer learning experience, methods for translating them both to an introductory and upper-level laboratory class are discussed. This laboratory will enable the integration of nanoscience and solid state chemistry into curricula using existing instrumentation. Future work will investigate the specific effectiveness of the experimental design in achieving the desired learning outcomes while also assessing how the introduction of modern research into the curriculum can positively impact attitudes and conceptions toward scientific inquiry.

■ ASSOCIATED CONTENT

SI Supporting Information

The Supporting Information is available at <https://pubs.acs.org/doi/10.1021/acs.jchemed.4c00173>.

Detailed materials and method ([PDF](#), [DOCX](#))

Laboratory handouts ([PDF](#), [DOCX](#))

Instructor materials ([PDF](#), [DOCX](#))

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Notes

The authors declare no competing financial interest.

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