The Importance of the CTAB Surfactant on the Colloidal Seed-Mediated Synthesis of Gold Nanorods

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Gold nanorods were synthesized by the colloidal seed-mediated, surfactant-assisted approach [Gou et al., *Chem. Mater*. **2005**, *17*, 3668−3672] using CTAB (hexadecylcetyltrimethylammonium bromide) obtained from ten different suppliers. The yield of gold nanorods depended strongly on the CTAB used: with the same recipe, three of the CTABs did not yield nanorods and produced only spherical gold particles, whereas the other CTABs yielded nanorods with nearly 100% yield. These results suggest that an impurity in the CTAB is very important for nanorod formation.

Gold nanorods have been explored for biological and medical use as optical contrast agents for dark field[1−5] and two-photon luminescence diagnostic imaging[6] and photothermal therapy of cancer cells.[1] They are attractive candidates for biomedical imaging because their optical response can be tuned to near-infrared wavelengths, which penetrate deep into cells and tissue;[7] furthermore, they do not photobleach or blink, and are chemically inert and biologically compatible.[8−11]

Gold nanorods have been produced in many laboratories[2,12−16] by a seed-mediated, surfactant-assisted approach that was first reported in 2001[17] and has been widely studied and improved.[18−20] Nonetheless, the reproducibility of the synthesis—i.e., the nanorod size, shape, and yield—has been a persistent challenge facing the technique.[14,21] Many studies have addressed this issue, attributing differences in reproducibility to a wide variety of factors including seed aging time,[22] the method of mixing the seed and growth solutions,[22] variations in salt concentration and temperature of the growth solution, as well as nanorod growth time.[14,21] Here, we report that one significant factor in nanorod reproducibility is the supplier of the CTAB, and that CTAB obtained from some suppliers did not yield gold nanorods. It appears that a very dilute impurity in the CTAB surfactant can greatly affect nanorod formation.

As sketched in Figure 1, the seed-mediated, surfactant-assisted gold nanorod synthesis relies on the initial preparation of ∼1.5 nm diameter gold nanoparticles formed by mixing aqueous solutions of hexadecylcetyltrimethylammonium bromide (CTAB), hydrogen tetrachloroaurate(III) hydrate, and sodium borohydride.[23] These gold nanoparticles are then added to a growth solution of concentrated CTAB, silver nitrate, hydrogen tetrachloroaurate(III) hydrate, and ascorbic acid.[19] Ascorbic acid is a weak reducing agent that induces heterogeneous gold deposition at the surface of the seed particles.[15] Anisotropic nanorod growth results from facet-selective gold deposition promoted by the silver ions, which adsorb to the gold surfaces by an underpotential deposition (UPD) mechanism as elucidated by Liu and Guyot-Sionnest.[24] The nanorod aspect ratio can be increased to a certain extent, up to 4.5, by increasing the silver concentration,[19] and the absence of Ag^+ from the reactions leads to only a very low yield of Au nanorods (see Supporting Information). CTAB coats the nanorod surface as a bilayer (as illustrated in Figure 2) that prevents aggregation;[25] it is also believed to aid nanorod growth by facet-sensitive surface adsorption.

In a recent paper,[6] we stated that we could only produce gold nanorods when CTAB from certain suppliers was used. Using the same recipe, CTAB from Acros, Sigma, and Aldrich did not yield nanorods, whereas CTAB from Fluka and MP Biomedicals did.[6] Figure 3 shows TEM and SEM images of gold colloids made using CTAB from these different suppliers.[26] In each preparation, the particles are monodisperse, but the shape is

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(26) Doubly-distilled deionized water was used in all preparations. Hydrogen tetrachloroaurate(III) hydrate, sodium borohydride, silver nitrate, and ascorbic acid were purchased from Sigma-Aldrich and used as received. Seed gold particles were formed by adding aqueous sodium borohydride (0.01 M, 600 μL) to a mixed aqueous solution of CTAB (0.10 M, 9.75 mL) and hydrogen tetrachloroaurate-(III) hydrate (0.01 M, 250 μL). The solution is stirred for 2 min after adding the sodium borohydride solution. 12 μL of the gold seed solution was then added to a growth solution of CTAB (0.10 M, 9.50 mL), silver nitrate (0.01 M, 75 μL), hydrogen tetrachloroaurate (III) hydrate (0.01 M, 500 μL), and ascorbic acid (0.1 M, 55 μL). The nanorods were allowed to grow overnight without stirring at 24 °C and their absorbance spectrum was measured the next day.

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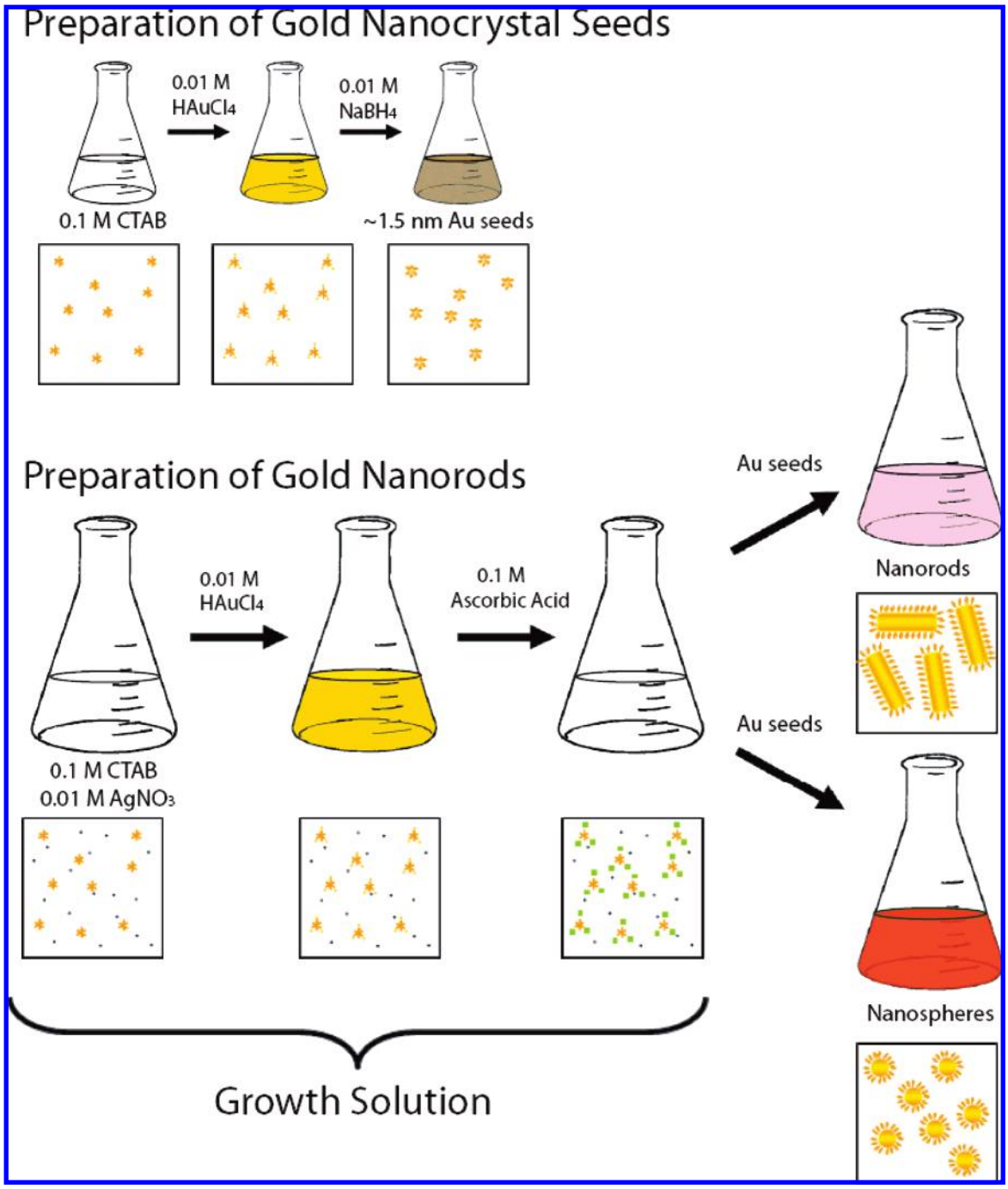

Figure 1. Seed-mediated, surfactant-assisted gold nanorod synthesis. Black dots represent Ag^+, the orange zig-zags are CTAB in the form of micelles, and the yellow circles and green squares are $AuCl_4^-$, and $AuCl_2^-$, respectively, complexed with CTAB micelles. The gold nanocrystal seeds are injected into the growth solution in the final step of the nanorod synthesis.

dramatically different—either spheres or rods—depending on the CTAB supplier. The only noticeable difference between the CTAB obtained from different suppliers seemed to be the purity. In our initial research, the CTAB that generated nanorods was relatively "impure" (~97%); therefore, we speculated[6] that an impurity in the CTAB was important for *inducing* nanorod formation.

In further experiments, however, we have found that relatively pure (>99%) CTAB from Sigma or Fluka also generates nanorods, and the role of impurities appears to be a more complicated issue.[27] Figure 4 shows UV−visible absorbance spectra of gold colloid dispersions made using CTAB from ten different suppliers. Dispersions of gold nanorods are easily

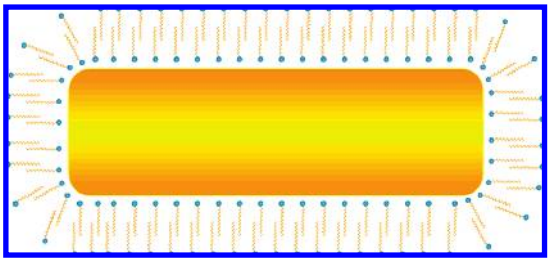

Figure 2. A gold nanorod created by a CTAB bilayer. The blue circles represent ammonium head groups and the orange zig-zags are the hydrocarbon tails of the CTAB.

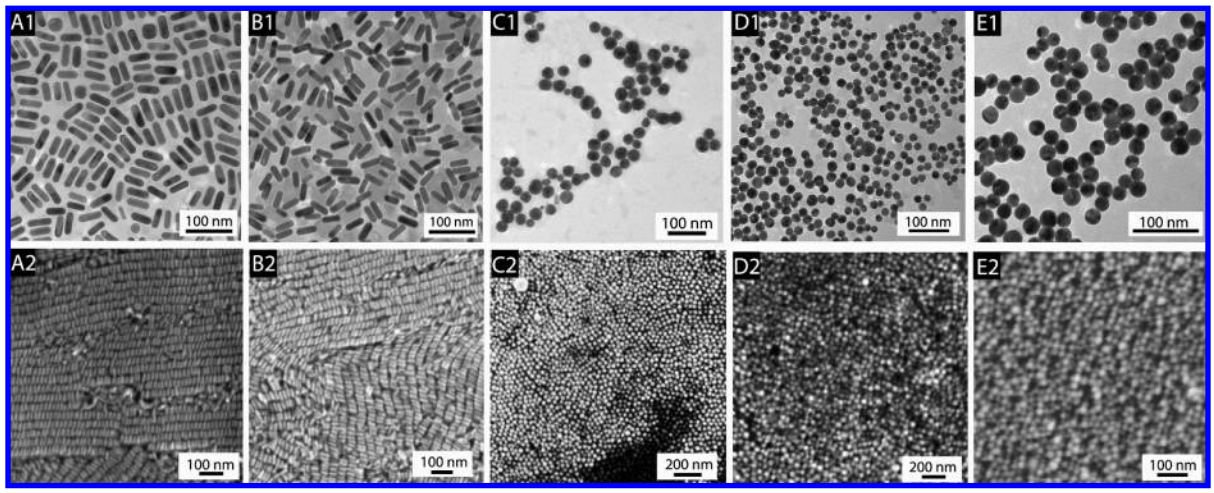

Figure 3. TEM (top images labeled with "1") and SEM (bottom images labeled with "2") images of gold colloids made using CTAB from five different suppliers (see Table 1): (A) Fluka (52370), (B) MP Biomedicals, (C) Acros, (D) Sigma (H5882), and (E) Aldrich. Of these, only CTAB supplied by Fluka and MP Biomedicals yielded nanorods, while the others yielded only spherical particles.

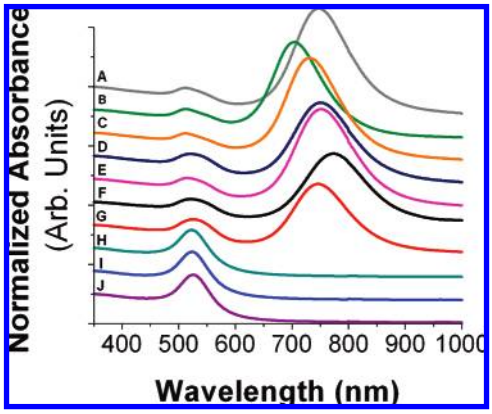

Figure 4. Absorbance spectra of gold nanorods synthesized using CTAB from ten different suppliers, as listed in Table 1: (A) Sigma (H6269), (B) Sigma (H9151), (C) Fluka (52365), (D) Fluka (52367), (E) Fluka (52369), (F) Fluka (52370), (G) MP Biomedicals, (H) Acros, (I) Sigma (H5882), (J) Aldrich. The longer wavelength peak in samples A−G indicates that nanorods formed. The peak shifts to longer wavelength with increasing nanorod length.

distinguished from gold spheres because nanorods exhibit two absorbance peaks, one at ∼520 nm and the other at ∼700−1300 nm, depending on the aspect ratio[28]—as opposed to only one at ∼520 nm for spheres. These peaks correspond to plasmon resonances: the shorter-wavelength peak at 520 nm to plasmon oscillations in the shorter transverse direction, and the longer wavelength peak (between 700 and 1300 nm) to longitudinal oscillations. Table 1 summarizes our findings. Of the ten different CTABs sampled, three do not generate rods (the spectra labeled H, I, and J in Figure 4 with only one absorbance peak indicate that nanorods did not form in these reactions).

These results are rather dramatic. If the "wrong" CTAB is used, then nanorods do not form. When we began to synthesize gold nanorods in our laboratory, we were unfortunate enough to have ordered the three CTABs that do not produce nanorods. We were about to abandon the research because we could not make

Table 1. Purity Noted in the Catalog and the Actual Lot Purity of CTAB from Several Different Suppliers That Were Used to Synthesize Gold Nanorods

sample	supplier	product #	nanorods?[a]	catalog purity	actual lot purity
A	Sigma	H6269	yes	∼99%	100.0%
B	Sigma	H9151	yes	∼99%	100.3%
C	Fluka	52365	yes	≥99%	99.4%
D	Fluka	52367	yes	≥99%	99.7%
E	Fluka	52369	yes	≥99%	99.7%
F	Fluka	52370	yes	≥96%	97.1%
G	MP Biomedicals	194004	yes	>98%	98.9%
H	Acros	22716V	no	≥99%	99.0%
I	Sigma	H5882	no	≥99%	100%
J	Aldrich	855820	no	95%	100.3%

[a] Nanorod formation is revealed by the color of the solution (spheres are red; rods are light purple), the appearance of the low-energy plasmon peak (at 700−800 nm) in the absorbance spectra, and confirmed by TEM imaging.

rods, when we made a last effort with one other CTAB from the chemical shelf of a neighboring lab, and it worked. We did not find any mention in the literature about the importance of the CTAB supplier on the synthesis and this seemed completely unexpected. Thus, we sought to identify the difference between the CTABs. We tried to determine whether an impurity in the CTABs was giving these results and tried several different analytical techniques, including size exclusion chromatography, XRD, NMR, and mass spectrometry (see Supporting Information), but did not observe any noticeable difference between the CTABs that generate nanorods and those that did not. Additionally, we tried adding different "impurities", including NaBr, KBr, cetyldimethylamine,[29] and surfactants with differing head groups (benzyldimethylammonium chloride (BDAC) and cetyltrimethylammonium chloride (CTAC)) in small amounts to the reactant solutions that did not yield nanorods (i.e., reactions with CTAB purchased from Sigma (H5882), Aldrich, and Acros), but we could not induce nanorod formation using any of these additives.

Since we could not identify analytically any impurity of particular importance, we performed a series of tests to examine the influence of CTAB from different suppliers on seed growth and nanorod growth to determine if there were any noticeable differences. Figure 5 shows a picture of seed particle solutions

(27) We performed these tests after talking to Jason Hafner, who pointed out that in their lab they successfully use CTAB from Sigma to generate nanorods. As it turns out, Sigma sells several different kinds of CTAB. We were using product number H5882, and Jason Hafner's lab was using product number H9151. We had not yet tried the particular CTAB that they were using. Upon testing, we found that different "high-purity" CTABs from Sigma did in fact yield nanorods.

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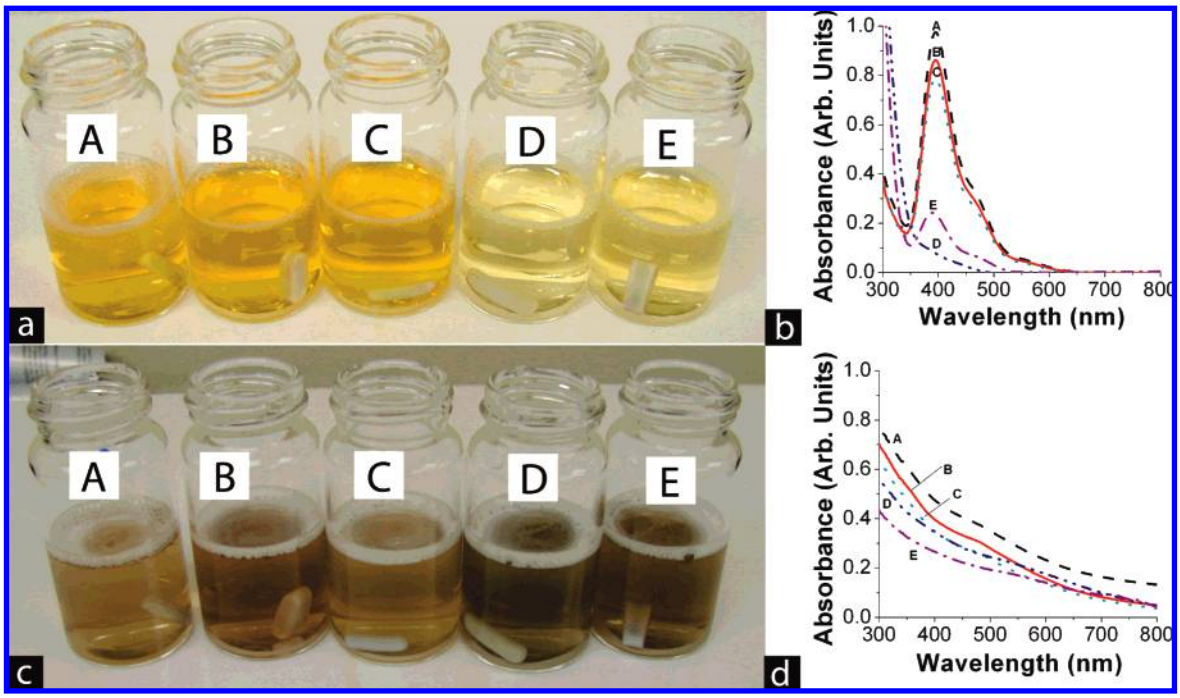

Figure 5. (a) Photographs of the reactant solutions for the gold seed particles (0.1 M CTAB and 0.01 M hydrogen tetrachloroaurate(III) hydrate) prior to sodium borohydride addition. Each solution was made using CTAB from a different supplier: (A) Fluka (52370), (B) MP Biomedicals, (C) Acros, (D) Sigma (H5882), and (E) Aldrich. (b) The corresponding absorbance spectra of the solutions in (a). (c) Reactant solutions after adding sodium borohydride (600 μL, 0.01 M) to induce particle formation, and (d) their corresponding absorbance spectra.

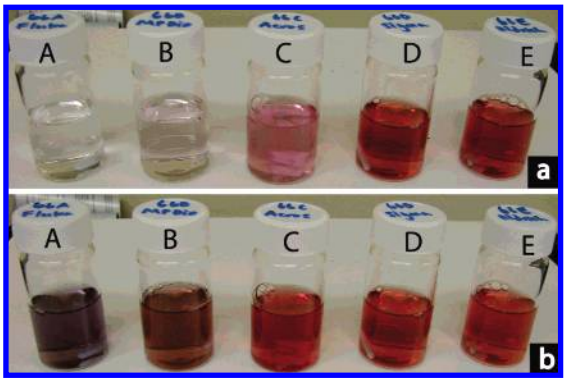

Figure 6. The growth solutions (a) 6 min and (b) 30 min after seed addition. The reactions were carried out using the same reactant concentrations and addition procedures with CTAB from five different suppliers: (A) Fluka (52370), (B) MP Biomedicals, (C) Acros, (D) Sigma (H5882), and (E) Aldrich. The red solution color is indicative of spherical particles, and the blue/purple color is characteristic of nanorods. Note that the formation of larger-diameter spherical gold particles has occurred in vials C, D, and E after only 6 min.

made using the last five CTABs listed in Table 1, along with their absorbance spectra before and after adding sodium borohydride. Prior to adding the reducing agent, the CTAB/$HAuCl_4$ solutions (Figure 5a) have distinctly different colors (note: aqueous solutions of all five CTABs prior to the addition of hydrogen tetrachloroaurate are clear and have identical absorbance spectra.) However, after reduction, all of the solutions turned similar shades of brown, indicating that the seed particle sizes made during this step were roughly equal, as also confirmed by TEM (see Supporting Information).

Figure 6 shows five vials of the growth solution 6 min and 30 min after adding 12 μL of the seed solution to the growth solution. The three vials on the right contain CTAB from Acros, Sigma (H5882), and Aldrich. CTAB from the different suppliers induced noticeably different gold colloid growth rates. After 6 min, the solutions with Fluka (52370) and MP Biomedicals CTAB were still clear, indicating that gold colloid growth had not yet occurred. In contrast, the solutions with CTABs from Acros, Sigma (H5882), and Aldrich were red 6 min after injection, indicating that larger-diameter spherical gold particles had already formed. Thirty minutes after seed injection, the three vials on the right were still red, indicating that the gold colloids remained spherical. The two vials on the left became blue/purple after 30 min, indicating that nanorods had formed. TEM and SEM images of the final products (shown in Figure 3) confirmed that the two vials on the left contained nanorods and the three vials on the right had only spherical particles. These experiments show that the CTABs that do not yield nanorods exhibit a much faster rate of gold colloid growth during the "growth" step. The CTAB can influence the growth rate by either increasing (or decreasing) the rate of gold cation reduction in solution, or by enhancing (or decreasing) the bonding strength of the adsorbed surfactant layer, which can also change the growth rate of the particles.

Additional experiments were performed to determine if the CTAB in the seed solution or growth solution was most important to nanorod growth (see Figure 7). When CTAB supplied by Acros, Sigma (H5882), or Aldrich was used in the growth solution, the final product always consisted entirely of spherical particles, regardless of the CTAB used to make the gold nanocrystal seeds. On the other hand, nanorods always formed when CTAB supplied by Acros, Sigma (H5882), or Aldrich was used to make the seeds but CTAB from Fluka (52370) or MP Biomedicals was used in the growth solution. The nanorods formed in these reactions, however, made up only a small percentage of the total product, and the lengths of the nanorods that formed were relatively short. This indicates that CTAB is primarily important for nanorod formation but is also important in the production of the seed particles as well. It is possible that these data reveal that different CTABs have slightly different binding strengths to the seed particles, which can influence the growth rates of the gold nanorods during the growth step.

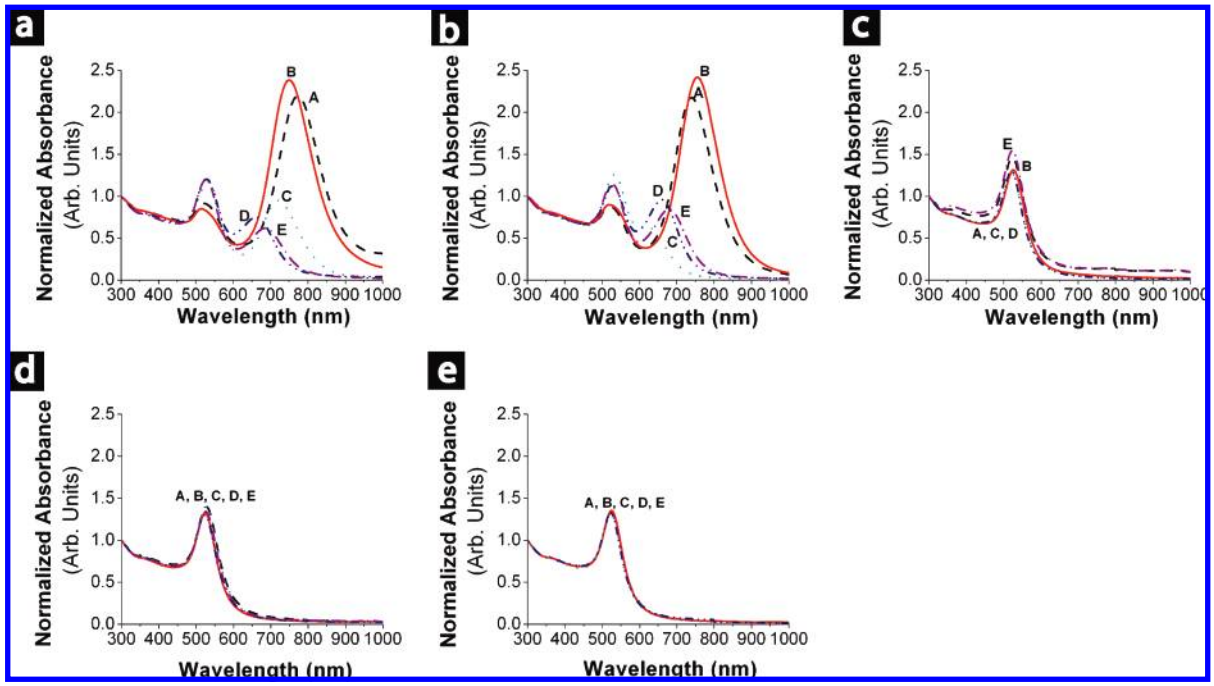

Figure 7. Absorbance spectra of gold nanorods produced in growth solutions with CTAB from five different suppliers: (a) Fluka (52370), (b) MP Biomedicals, (c) Acros, (d) Sigma (H5882), and (e) Aldrich. The curves labeled A−E in each plot correspond to spectra from nanorods that were made with gold seeds capped with CTAB from five different suppliers: (A) Fluka (52370), (B) MP Biomedicals, (C) Acros, (D) Sigma (H5882), (E) Aldrich.

In summary, we note that the literature contains reports of gold nanorod synthesis using CTAB purchased from Acros[30] and Aldrich,[13,14,20−22,31−40] which we were never able to use to synthesize nanorods. This suggests that the presence of impurities in the CTAB from a particular supplier can vary from lot to lot. Only three papers report the synthesis of nanorods using Fluka CTAB,[19,24,41] while several use Sigma[2,23,42−49] or other suppliers.[29,50] It is presently unclear to us whether the impurity in the CTAB either *induces* or *disrupts* nanorod formation, but we speculate at this point that it is most likely a very dilute impurity in >99% pure CTABs from Acros, Sigma (H5882), and Aldrich that in fact *disrupts* nanorod formation by enhancing the growth rate during the "growth step", which leads to spherical particles. The CTAB could increase the particle growth rate either by increasing the reduction rate of gold cations in solution or by allowing faster Au deposition on the colloid surface with weaker surface passivation. Interestingly, there is an analogous story for colloidal CdSe nanorods. Peng et al.[51] found that, in the case of CdSe nanorods grown by high temperature arrested precipitation in the coordinating solvent, trioctylphosphine oxide (TOPO), that an impurity was needed to induce their formation. Without this impurity in the TOPO—which turns out to be a phosphonic acid—the particle growth rates were much faster, and only spherical CdSe nanocrystals would form. The impurity in this case, slowed down nanocrystal growth and helped along the formation of the nanorods. Similarly, in the case of Au nanorods, "slow" controlled colloid growth is observed when nanorods form, and an impurity in the CTAB surfactant can dramatically influence the colloid growth rate and thus nanorod formation.

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Supporting Information Available: Size exclusion chromatography, mass spectrometry, XRD, ^{1}H NMR, and ^{13}C NMR spectroscopy data obtained from CTABs from different suppliers. This material is available free of charge via the Internet at http://pubs.acs.org.

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