

Communication

Fractionation and Characterization of High Aspect Ratio Gold Nanorods Using Asymmetric-Flow Field Flow Fractionation and Single Particle Inductively Coupled Plasma Mass Spectrometry

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Supplemental Materials

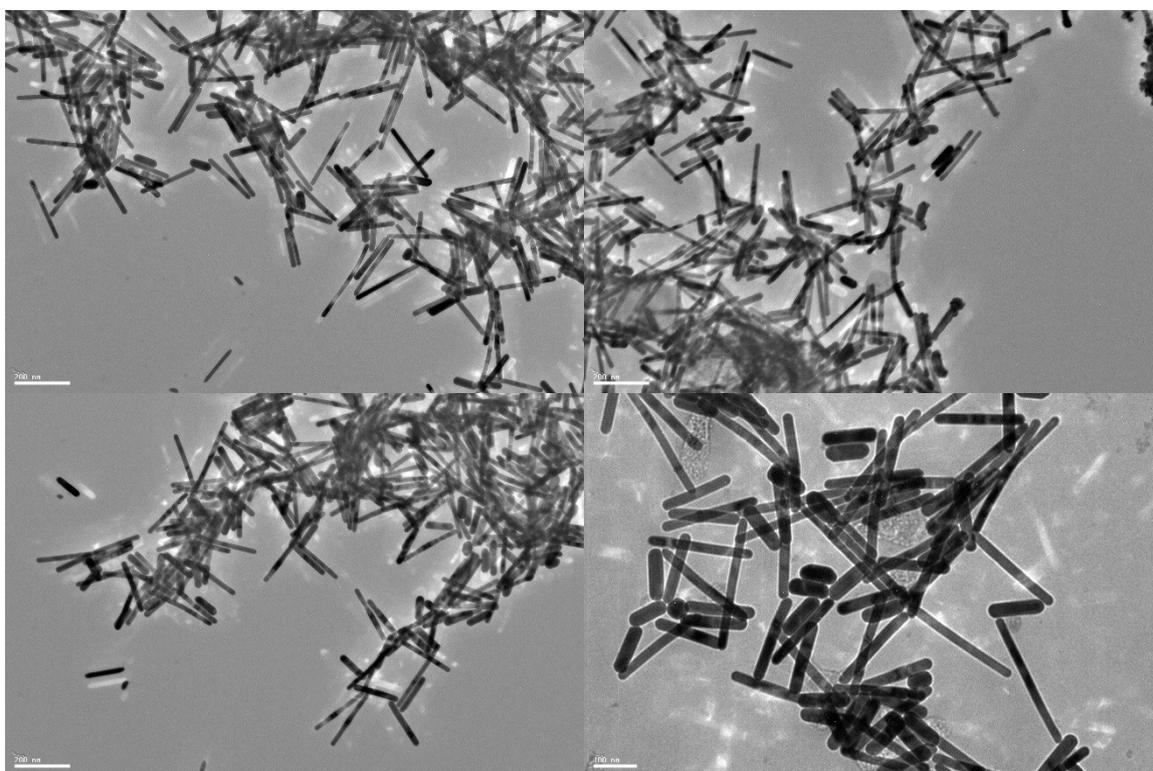


Figure S1. Additional TEM images of as received commercially sourced GNRs. To prepare the sample for analysis, 6 μ L of suspension was drop-cast on a formvar-coated copper grid. The sample was then air dried at room temperature.

Table S1. To estimate the length distribution from TEM imaging, 237 imaged particles were analyzed. Only single rods were counted (*i.e.*, no aggregates or overlapping rods). The TEM scale bar was the only reference of measurement; therefore intrinsic errors in the length measurements may exist.

Estimated Size (nm)	Number of Particles	% of total *
50	19	8
100	61	26
150	51	22
200	70	30
250	28	12
300	8	3
Total	237	

* Total does not add up to 100% due to rounding.

Determination of A4F Recovery

Sample recovery during A4F fractionation was estimated by replicating the experiment under identical conditions without applying cross-flow during elution. The integrated intensity of the UV-Vis absorbance, for example, can be used as a concentration metric. Recovery is estimated by difference between the integrated signals from the two measurements, assuming 100% recovery in the absence of cross-flow and a Beer–Lambert law relationship between Au mass and absorbance at an SPR peak. Given the presence of different morphologies, lengths and aspect ratios, the mass-absorbance relationship yields a rough estimate of recover in the present system. This is a commonly employed procedure in A4F.

Single Particle ICP-MS Data Acquisition and Processing

Single particle ICP-MS measurements were conducted on a Thermo Fisher X series^{II} quadrupole ICP-MS (The identification of any commercial product or trade name does not imply endorsement or recommendation by the National Institute of Standards and Technology). The ¹⁹⁷Au intensity was recorded in time-resolved analysis (TRA) mode using Thermo Fisher PlasmaLab software. A typical sample series consists of blanks (deionized water and 0.15 mmol L⁻¹ CTAB), a 27 ng L⁻¹ (1.5×10^4 particle mL⁻¹) AuNP standard (NIST RM 8013, nominal diameter 60 nm), ionic gold standards in the concentration range of (0.5 to 28) $\mu\text{g L}^{-1}$, and the diluted GNR sample suspensions in 0.15 mmol L⁻¹ CTAB. GNR samples were diluted in triplicate and measured for 600 s at a dwell time of 10 ms.

Each measurement of a sample results in 60,000 readings consisting of background signals (ions of the same element, particles smaller than the detection limit (10 nm for sphere AuNPs) and instrument noise) and pulse signals on top of the background produced by particles. Data in the unit of counts per second (cps) were converted to counts per event and then processed in Microsoft Excel to calculate GNR length and length distribution. The particle signals were first differentiated from the background

using a five times standard deviation (5σ) criterion [29,30]. To eliminate the bias of mean particle intensity by false positive signals (high background signals that are erroneously recognized as particle events), data points with intensity less than 5 counts per 10 ms were further removed [29]. After split pulse correction [29], the average particle intensity of 60 nm AuNP standard was used to calculate the transport efficiency by comparing with the intensity of soluble gold mass flow [35].

Then the mass of individual GNRs is calculated by:

$$m_{GNR} = I_{GNR} q_{liq} t_{dwell} \eta_n / S_{Au^+}$$

where m_{GNR} = GNR mass (μg), I_{GNR} = particle pulse intensity (counts), q_{liq} = sample uptake rate (L ms^{-1}), t_{dwell} = dwell time (10 ms), η_n = transport efficiency, and S_{Au^+} = slope of ionic Au standard calibration curve (counts per ($\mu\text{g L}^{-1}$)).

The GNRs used in this study have a relatively uniform diameter of approximately 20 nm. Assuming cylindrical geometry and fixed diameter of 20 nm, the lengths of GNRs were calculated as follows:

$$L = 4 \times 10^{15} \times m_{GNP} / \pi d^2 \rho$$

where L = length of GNRs (nm), d = diameter of cross section (20 nm), and ρ = density of Au (19.3 g cm^{-3}).