Electronic Supplementary Material (ESI) for Chemical Communications

A Convenient Light Initiated Synthesis of Silver and Gold Nanoparticles using a Single Source Precursor

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Supporting Informations

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1 General

General conditions. All reactions involving phosphines were carried out under an atmosphere of dry nitrogen using standard Schlenk techniques. Dichloromethane was purified by distillation from calcium hydride. All preparations containing silver- or the light sensitive gold compound were carried out in the absence of light.

Reagents. Silver nitrate, *trans*-cinnamic acid, triphenylphosphine and triphenylphosphine gold(I)chloride were purchased from commercial suppliers and were used without further purification. *O,O'*-*trans*-(cinnamato)bis(triphenylphos phine) silver(I) was synthesised according the procedure published by Edwards *et al.*[1]

Instruments. ^1H NMR (500.3 MHz), $^{31}P\{^1H\}$ NMR (202.5 MHz) and $^{13}C\{^1H\}$ NMR (125.8 MHz) spectra were recorded with a Bruker Avance III 500 spectrometer at 298 K. Chemical shifts are reported in δ (parts per million) using undeuterated solvent residues as internal standard (chloroform-d_3: ^1H at 7.26 and $^{13}C\{^1H\}$ at 77.16 ppm). Infrared spectra were recorded using a FT-IR-Nicolet 200 spectrometer. The melting points of analytical purity samples (sealed off in nitrogen-purged capillaries) were determined using a Gallenkamp MFB 595 010 M melting point apparatus. Microanalyses were performed with a Thermo FLASHEA 1112 Series instrument. TEM Images were prepared with a 200kV-High Resolution Transmission Electron Microscope (HRTEM) Philips CM 20 FEG. For the size distribution 300 particles were counted and measured using Lince 2.4.2e. UV-Vis Spectra were obtained from a Varian Cary 5000 with standard quartz glas sample vials (d = 10 mm).

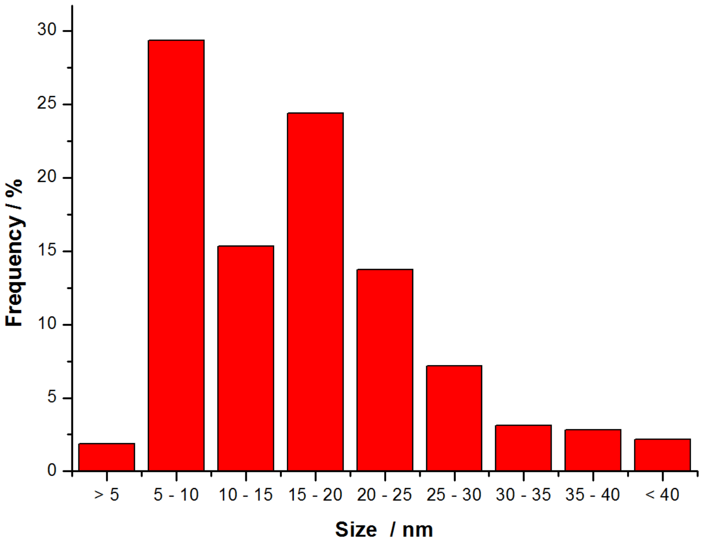
2 Experimental Section

Preparation of silver(I) *trans* cinnamate (1): 2.00 g (13 mmol) of *trans* cinnamic acid were dissolved in a mixture of 1.8 mL (13 mmol) triethyl amine, 40 mL of ethanol and 4 mL of acetonitrile. This solution was added dropwise at 0 °C under vigours stirring to a solution containing 2.31 g (13.7 mmol) of silver nitrate, 40 mL ethanol and 4 mL acetonitrile. The immediatly forming suspension was stirred at 0 °C for additional 2 h. The colorless precipitate was collected by filtration through a plug of celite and washed 3 times with 20 mL of cold ethanol followed by 30 mL of cold diethylether. The residue was dryed *in vacuo*. Yield: 3.02 g (99 %), colorless solid, insoluble in common organic solvents. Elemental Analysis calculated for $C_9H_7AgO_2$: C 42.39, H 2.77; found: C 42.13, H 2.72. IR data [KBr, cm^{-1}]: 1638 s [ν(C=C)], 1574 m [$\nu_{asym.}(CO_2)$], 1550 s [$\nu_{asym.}(CO_2)$], 1447 w [$\nu_{sym.}(CO_2)$].

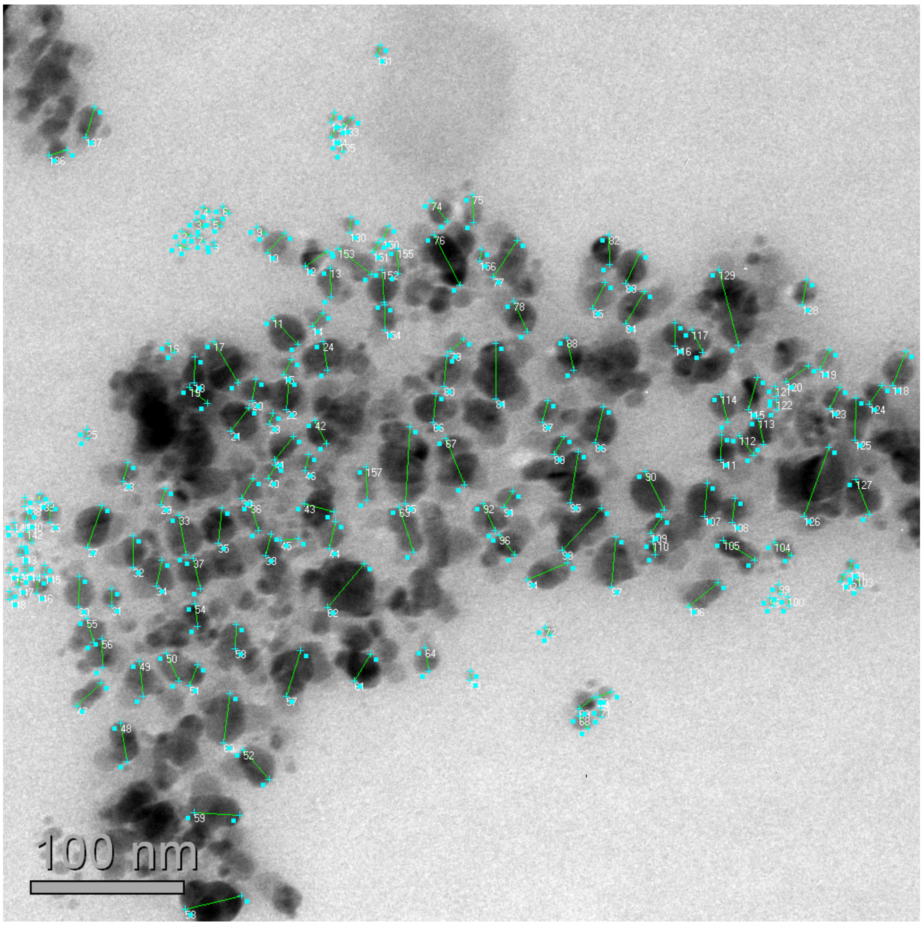
(*O*-*trans*-cinnamato)bis(triphenylphosphine) gold(I)(3): Compound **1** (0.17 g, 0.65 mmol) was suspended in 10 mL of abs. toluene. To this suspension 0.32 g of triphenylphosphine gold(I)chloride were added and the mixture was stirred overnight at ambient temperature. Precipitated silverchloride was filtered off and washed with an additional portion of toluene (10 mL). All volatiles were evaporated in *vacuo* and the resulting oil was redissolved in dichloromethan (5 mL) and precipitated with *n*-hexane (50 mL). The title compound can be isolated as an off-white solid. Yield: 0.96 g (97 %). Melting Point: 59 °C. Elemental Analysis calculated for $C_{27}H_{22}AuO_2P$: C 53.48, H 3.66; found: C 53.70, H 3.74. IR data [KBr, cm^{-1}]: 1636 s [ν(C=C)], 1573 m [$\nu_{asym.}(CO_2)$], 1554 s [$\nu_{asym.}(CO_2)$], 1477 w [$\nu_{sym.}(CO_2)$]. ^1H NMR [$\mathrm{CDCl_3}$, ppm]: 7.63 (d, $^3J_{HH}$ = 15,56 Hz, $C_6H_5\mathrm{C}\mathbf{H}$), 7.59 - 7.29 (m, 20 H, $\mathrm{P}C_6\mathbf{H}_5$ and $C_6\mathbf{H}_5$), 6.61 (d, $^3J_{HH}$ = 16.03 Hz, 1 H, $\mathbf{H}\mathrm{CCO_2Au}$. $^{13}C\{^1H\}$ NMR [$\mathrm{CDCl_3}$, ppm]: 172.95 (**C**OOH), 141.56 (Ph**C**C), 135.97 (C_6H_5), 134.33 ($^2J_{PC} = 13.65$ Hz, $\mathrm{P}(C_6H_5)_3$), 132.00 ($\mathrm{P}(C_6H_5)_3$), 134.33 ($^3J_{PC}$ = 12.18 Hz, $\mathrm{P}(C_6H_5)_3$ and C_6H_5), 128.73 (C_6H_5), 127.83 (C_6H_5), 124.09 ($\mathrm{Au_2OC}\mathbf{C}\mathrm{C}$). $^{31}P\{^1H\}$ NMR [$\mathrm{CDCl_3}$, ppm]: 27.99 (s, $\mathbf{P}\mathrm{Ph_3}$).

References

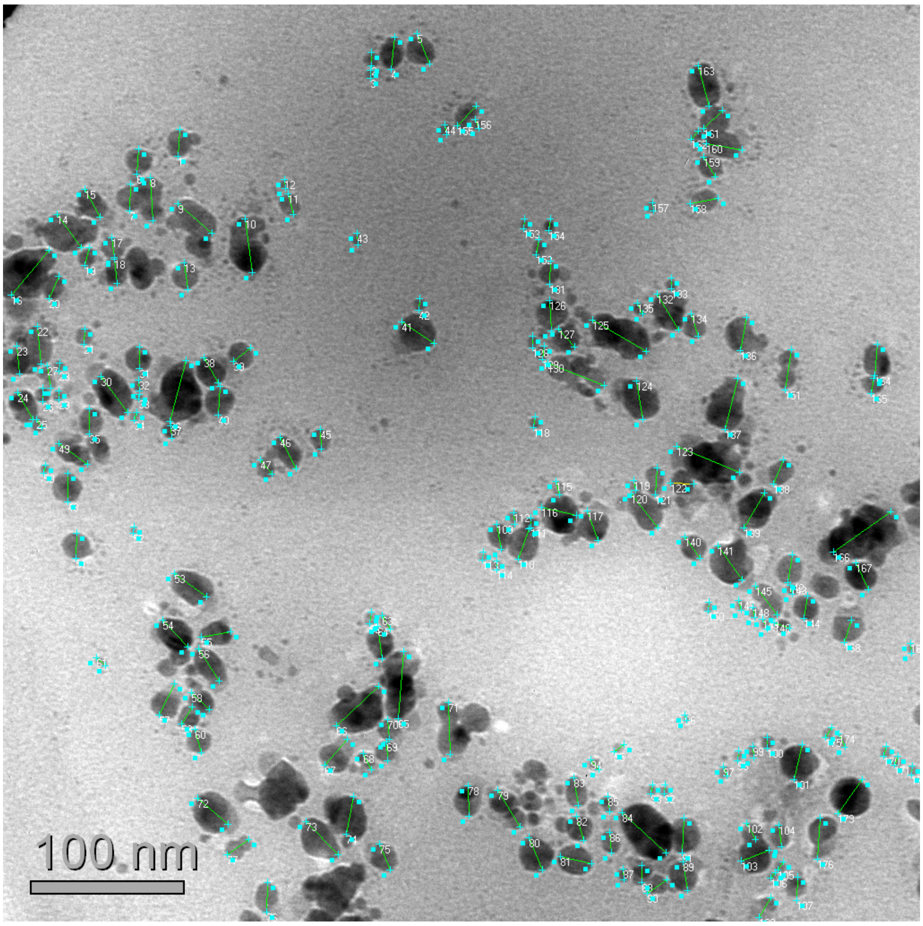
[1] D. A. Edwards, M. F. Mahon, K. C. Molloy and V. Ogrodnik, *J. Mater. Chem.*, 2003, **13**, 563–570.



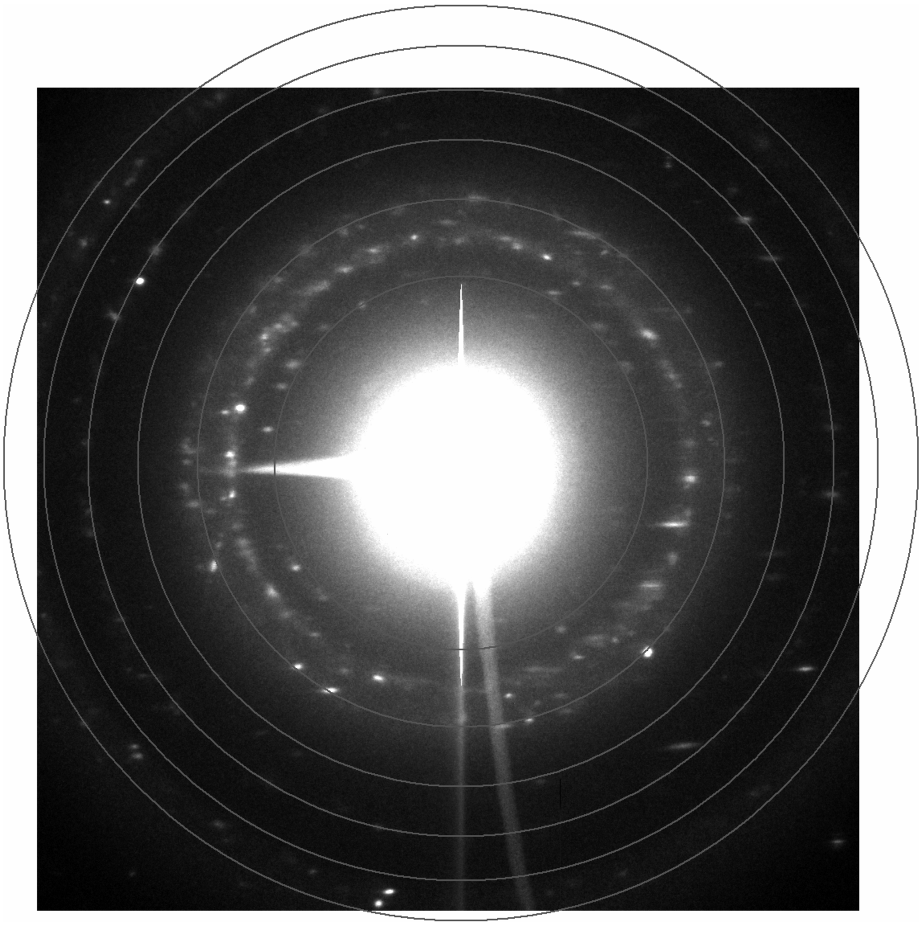
SI 1: Particle size distribution resulting from **2**.



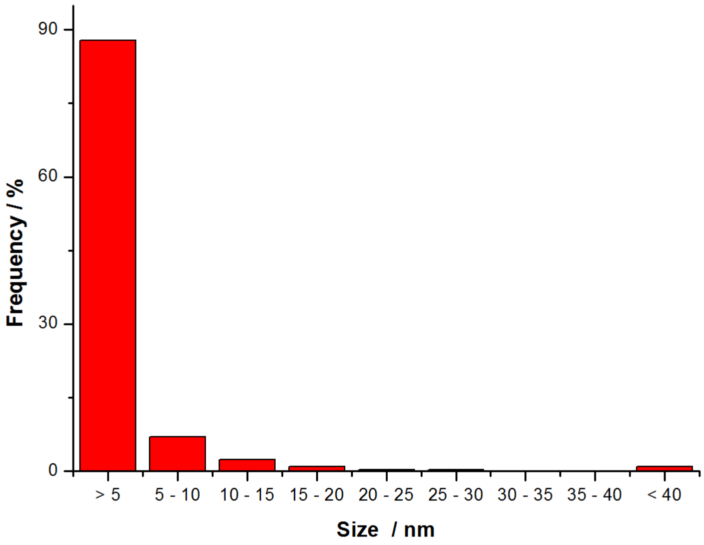
SI 2: TEM image of the resulting silver nanoparticles from **2**.



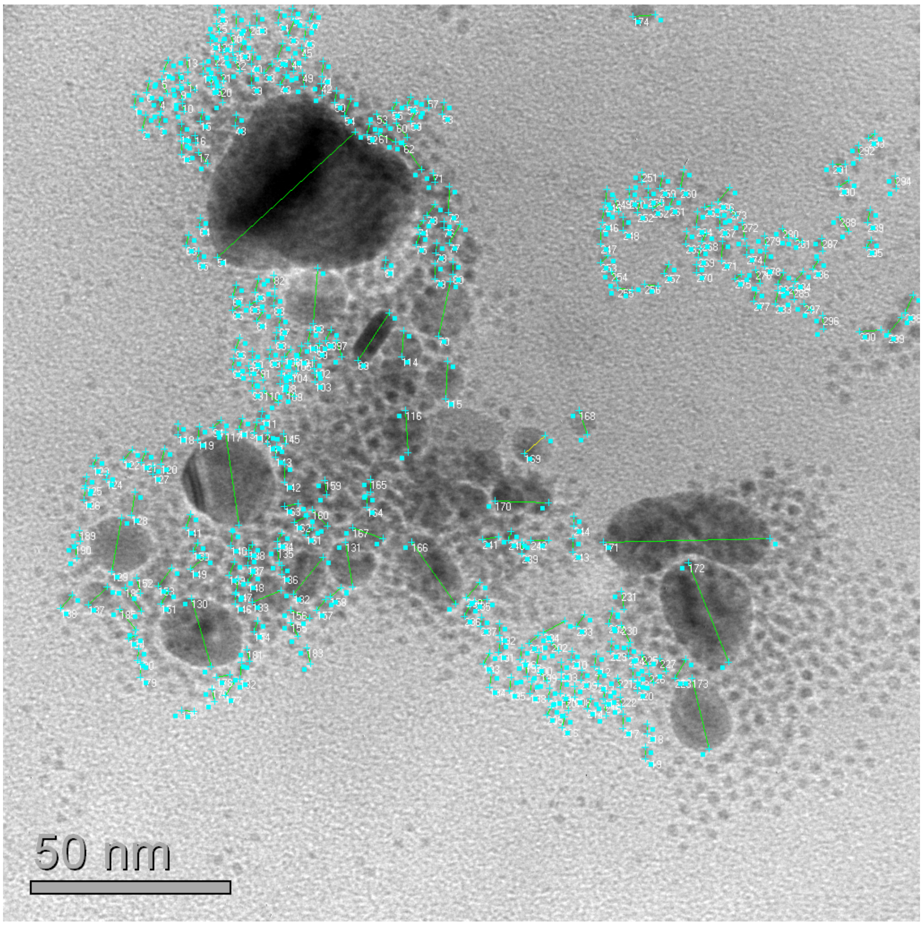
SI 3: TEM image of the resulting silver nanoparticles from **2**.



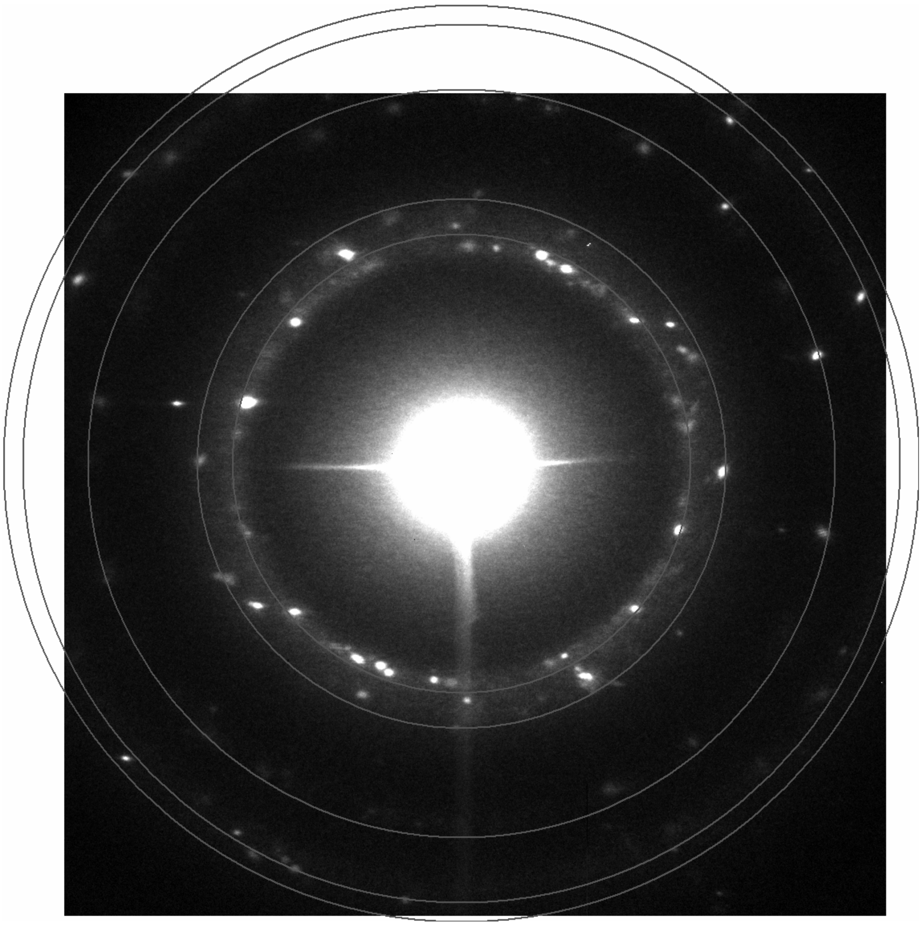
SI 4: Diffraction pattern showing the typical reflexes of silver fcc.



SI 5: Particle size distribution resulting from **3**.



SI 6: TEM image of the resulting gold nanoparticles from **3**.



SI 7: Diffraction pattern showing the typical reflexes of gold fcc