Facile Preparation of Metal Telluride Nanocrystals Using Di-n-octylphosphine Oxide (DOPO) as an Air-stable and Less Toxic Alternative to the Common Tri-alkylphosphines

Huaibin Shen,[‡] Xin-Dong Jiang,[‡] Sujuan Wang, Yantao Fu, Changhua Zhou, and Lin

Song Li*

Key Laboratory for Special Functional Materials of the Ministry of

Education, Henan University, Kaifeng, 475004, P. R. China

E-mail:lsli@henu.edu.cn,

Source of Reagents. *n*-octylmagnesium bromide (as 2.0 M diethyl ether solution, Aldrich), di-nbutylphosphite (96%, Aldrich), sulfuric acid, and potassium carbonate were obtained from Beijing Chemical Reagent Ltd., China. All chemicals were used as received without any further purification.

Synthesis of DOPO. The synthesis of DOPO was adapted from a literature method (Williams, R. H.; Hamilton, L. A. J. Am. Chem. Soc. 1952, 74, 5418–5420). Di-nbutylphosphite (110 g, 0.57 mol) was added dropwise under a nitrogen atmosphere, with stirring, to *n*-octylmagnesium bromide (1.6 mol, 800 mL of diethyl ether solution) over the course of 110 min. The reaction temperature was maintained

at ~10-15 °C during addition and was subsequently raised to the reflux point to complete the reaction. The reaction mixture was then cooled to 5-10 °C and an aqueous sulfuric acid solution (25 w/w %, 600 g) was added during one hour period. The ether phase was separated and washed successively with deionized water (200 mL) three times, potassium carbonate solution (15w/w %, 200 mL) three times, and deionized water (500 mL) five times. The ether was removed by distillation at 65 °C. Recrystallization of the crude product from n-hexane (100 mL) yielded white crystals (144 g, 0.53 mol, 92%).



Figure S1. Photographs of as-prepared Te precursors at different temperature: (a) 100 °C, (b) 320 °C.



Figure S2. HRTEM images of CdTe nanocrystals that are corresponding with Figure1

b-d.



Figure S3. XRD pattern of as-synthesized dot-shaped CdTe nanocrystals.



Figure S4. TEM and HRTEM images of as-synthesized tetrapod-shaped CdTe nanocrystals.



Figure S5. Typical photoluminescence and absorption spectra of a CdS, CdSe, and CdTe nanocrystals synthesized by using S, Se, and Te dissolved in DOPO as precursor, respectively.



Figure S6. XPS spectra of tetrapod-shaped CdTe (a), dot-shaped ZnTe (b), cubic-shaped PbTe (c), cubic-shaped $Cu_{2-x}Te$ (d), dot-shaped Ag_2Te (e), ternary-alloyed CuInTe₂ (f), quaternary-alloyed Cu₂ZnSnTe₄ (g). All the XPS spectra reveal the successfully synthesized metal telluride nanocrystals.