Supplementary Information

Photolytic preparation of tellurium nanorods

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Experimental procedures

1.1. General procedures

All manipulations were conducted under a nitrogen atmosphere using standard Schlenk techniques. Deaerated solvents were used throughout.

Tellurium powder (99.8%; 200 mesh; Aldrich), dodecanethiol (98%; Aldrich), and polyoxyethylene(12) tridecyl ether (C_{12} - C_{14} isoalkyl isomers; Aldrich) were all used as received.

1.2. Synthesis of ${}^{t}Bu_{2}Te_{2}$

The synthesis of ${}^{t}Bu_{2}Te_{2}$ was adapted from a literature procedure.¹ In short, a solution of ${}^{t}BuLi$ (20 mL, 34 mmol) was added in 2 mL increments to a stirred suspension of tellurium (5.015 g, 40.01 mmol) in dry THF under a nitrogen atmosphere at -78 °C. After 12 min, 4 mL of deionized water was added dropwise to the stirred solution and the reaction was allowed to warm to room temperature and exposed to air. The organic solution was subsequently extracted with aqueous NH₄Cl and hexanes and the combined organic extracts were dried over anhydrous MgSO₄. Upon filtering off the MgSO₄, the organic solution was evacuated at room temperature to yield a crude brown solid. The crude product was purified by vacuum sublimation (0.05 mm Hg, 36 °C) to yield orange crystals (3.714 g, 59%). The product is stable in the solid-state for several months when stored at 8 °C in the absence of light. ¹H NMR (CDCl₃, 25 °C, 400 MHz): δ 1.65 (s). ¹³C{¹H} NMR (CDCl₃, 25 °C, 100.6 MHz): δ 23.0 (CMe₃), 36.5 (CH₃). ¹²⁵Te{¹H} NMR (CDCl₃, 25 °C, 104.7 MHz): δ 480. UV-vis (cyclohexane, nm): ca. 195 (ϵ = 54,000 M⁻¹ cm⁻¹), 262 (ϵ = 6,310 M⁻¹ cm⁻¹), 367 (ϵ = 630 M⁻¹ cm⁻¹). Mp = 39-40 °C.

1.3. Preparation of tellurium nanorods

^{*t*}Bu₂Te₂ (50.0 mg, 0.135 mmol), dodecanethiol (0.10 mL), and polyoxyethylene(12) tridecyl ether (5.0 mL) were stirred in a Schlenk flask and degassed by cycling between nitrogen and vacuum 3 times. Deaerated water (50 mL) was added with vigorous stirring to produce an optically transparent, light yellow solution which was cannula transferred into a nitrogen-filled quartz flask. The flask was sealed under nitrogen and subjected to photolysis at 254 nm (Luzchem ICH-2 photoreactor, 4/16 low-pressure 8 W Hg lamps were used) for 12 h with stirring to yield a dark brown suspension of the Te nanorods. Purification of the nanorods was accomplished by precipitation with acetone (acetone:water = 5:1 vol/vol) followed by centrifugation (6000 rpm, 15 min). The freshly purified nanorods can be easily redispersed in water with gentle agitation.

1.4. Characterization

Transmission electron microscopy (TEM) analysis was performed on a JEOL JEM-2100 microscope at an operating voltage of 200 kV, and equipped with a Gatan Orius CCD camera. Samples for TEM analysis were prepared from dilute purified samples of the nanorods deposited on 300 mesh carbon-coated copper grids (Ted Pella, Inc.). Powder X-ray diffraction (XRD) was performed on a Rigaku Ultima IV diffractometer using a Cu K α ($\lambda = 1.54$ Å) radiation source. X-ray photoelectron spectroscopy (XPS) was performed on a Surface Science M-Probe Spectrometer Model ESCA2703 using a monochromated Al anode. The binding energy of C 1s in all spectra was standardized to 285.0 eV. For both XRD and XPS analyses, suspensions of the nanorods were deposited on glass substrates and dried at room temperature. UV-vis spectra were acquired on a Cary 14 spectrophotometer in dual beam mode using quartz cuvettes with both 1 and 10 mm path-lengths. Solution NMR spectra were recorded at 400 MHz for ¹H, 100.6 MHz for ¹³C, and 104.7 MHz for ¹²⁵Te using a Varian Mercury 400 (¹H, ¹³C) or a Varian 400 MR (¹²⁵Te) spectrometer. Chemical shifts for ¹H NMR were referenced internally to the residual proton signal for CDCl₃ at 7.26 ppm. Chemical shifts for ¹³C NMR were referenced internally to CDCl₃ at 77.0 ppm. Chemical shifts for ¹²⁵Te NMR were referenced externally to diphenyl ditelluride in CDCl₃ at 422 ppm.²

- 1 L. Engman, M. P. Cava, *Synth. Commun.*, 1982, **12**, 163.
- 2 R. A. Gariani, F. Simonelli, A. R. M. Oliveira, A. Barison, J. V. Comasseto, *Tetrahedron: Asymmetry*, 2006, **17**, 2930.



Fig. S1 UV-vis spectrum of ^{*t*}Bu₂Te₂ in cyclohexane.



Fig. S2 TEM image of Te nanorods (left) and HRTEM image of a single Te nanorod showing the (001) and (003) lattice planes (d = 5.73 and 1.91 Å, respectively) (right).



Fig. S3 High resolution XPS spectrum of the Te 3d region for the Te nanorods.



Fig. S4 UV-vis spectra of Te nanorods and ellipsoidal nanocrystals taken in aqueous micellar solution. Control spectra of micellar solutions taken after removing the tellurium do not show appreciable absorption in this region (see Fig. S5).



Fig. S5 UV-vis spectra of aqueous micellar solution with nonionic surfactant and dodecanethiol after removal of the tellurium nanorods via precipitation. No appreciable absorption is observed in the region where ${}^{t}Bu_{2}Te_{2}$ or the Te nanostructures absorb.