

Facile synthesis of hexagonal Sb_2Te_3 nanoplates using Ph_2SbTeR (R=Et, Te) single source precursors

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1. Precursor Synthesis

All manipulations are carried out using the standard Schlenk and glove-box techniques under argon atmosphere. Tetrahydrofuran (THF) and hexane were freshly distilled from sodium benzophenone ketyl. Tellurium powder (Aldrich 99.8%), Ethyllithium solution (Aldrich 0.5M in benzene:cyclohexane), Phenyllithium solution (Aldrich 1.8M in dibutyl ether) were used as received. Ph_2SbCl has been prepared by following literature procedure.¹ ^1H NMR (400 MHz) was recorded with Bruker Avance II spectrometer in CDCl_3 using TMS as an internal standard. Elemental analyses of the precursors were performed on a Perkin-Elmer 2400 CHN/S analyzer.

Synthesis of Ph_2SbTeEt .

205 mg (1.6 mmol) Tellurium powder was added to a mixture of 3.2 ml 0.5 M solution of Ethyl lithium in 10 ml THF under stirring. After approximately 10 min the tellurium powder started to react under the formation of a dark brown solution and stirring is continued for 30 mins at room temperature.

Ph_2SbCl (500 mg, 1.6 mmol) dissolved in 5 ml THF was added to the LiTeEt solution under stirring, which turned greenish yellow and after stirring for 3 h at room temperature the THF was removed under vacuum and 60 ml hexane was added. The solution was separated from precipitated LiCl by filtration. Removal of the solvent gives the desired product as red color liquid.

Yield: 40-45%. Elemental Anal. Calcd for $\text{C}_{14}\text{H}_{15}\text{SbTe}$ (%): C, 38.87; H, 3.49. Found (%): C, 39.11; H, 3.62. ^1H -NMR (400 MHz, CDCl_3): δ 1.5 (3H, CH_3 , t), 2.9 (2H, CH_2 , quad.), δ 7.2-7.7 (10H, Ph, m) .

Synthesis of Ph_2SbTePh .

1g Tellurium powder was added to a mixture of 4.35 ml 1.8 M solution of phenyl lithium in 15 ml THF under stirring. After approximately 10 min the tellurium powder started to react under the formation of a dark brown solution and stirring is continued for 30 mins at room temperature.

Ph_2SbCl (2.44 g) dissolved in 10 ml THF was added to the LiTePh solution under stirring, which turned greenish yellow and after stirring for 3 h at room temperature the THF was removed

under vacuum and 80 ml hexane was added. The solution was separated from precipitated LiCl by filtration. Removal of the solvent gives the desired product as dark brown color liquid.

Yield: 45-50%. Elemental Anal. Calcd for $C_{18}H_{15}SbTe$ (%): C, 44.98; H, 3.15. Found (%): C, 45.17; H, 3.27. 1H -NMR δ 6.9-7.7 (Ph, m).

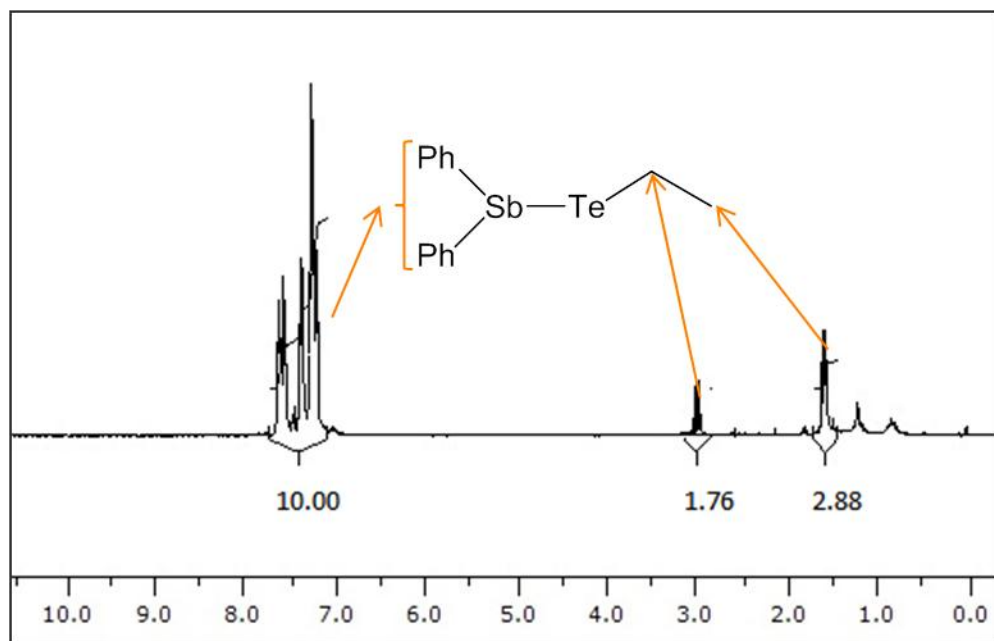


Fig. S1. 1H NMR spectrum of $Ph_2SbTeEt$ in $CDCl_3$

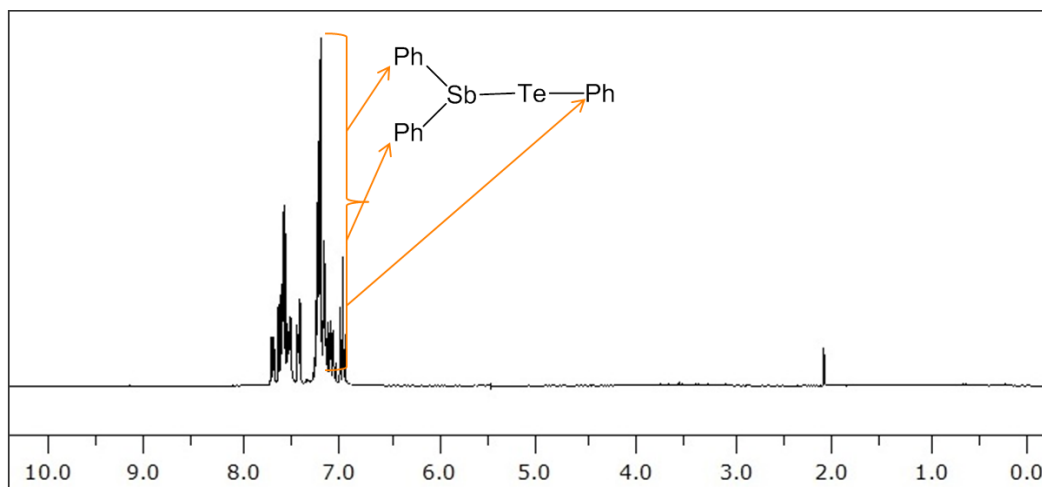


Fig. S2. 1H NMR spectrum of $Ph_2SbTePh$ in $CDCl_3$

2. Characterization

Powder X-ray diffraction (XRD) data were obtained from a Rigaku DMAX 2000 X-ray diffractometer. Transmission electron microscopy (TEM) images and energy-dispersive X-ray spectroscopy (EDS) data were obtained from a Tecnai G2F30 field emission transmission electron microscope. Scanning electron microscope (SEM) images were obtained from an Ultra-high Resolution FE-SEM S-4800 (Hitachi) microscope. The X-ray photoelectron spectra (XPS) were recorded on a MultiLab ESCA 2000 machine.

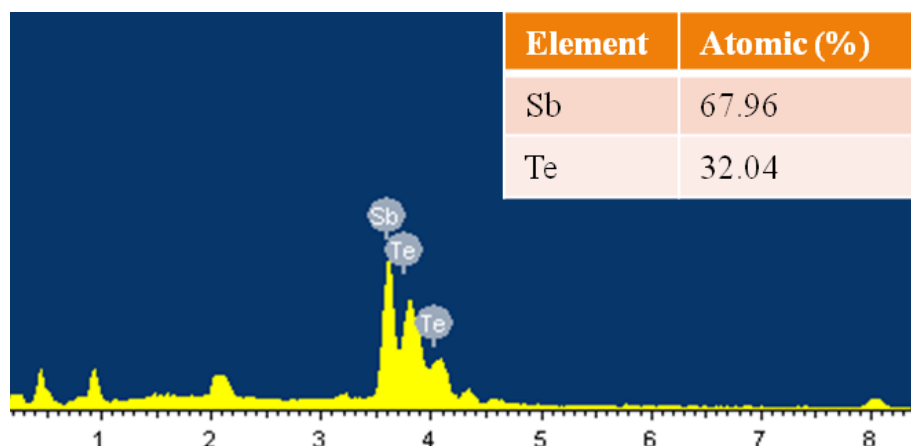


Figure S3. Energy-dispersive X-ray spectroscopy pattern of Sb₂Te nanocrystals obtained at 1 h reaction time using Ph₂SbTeEt SSP

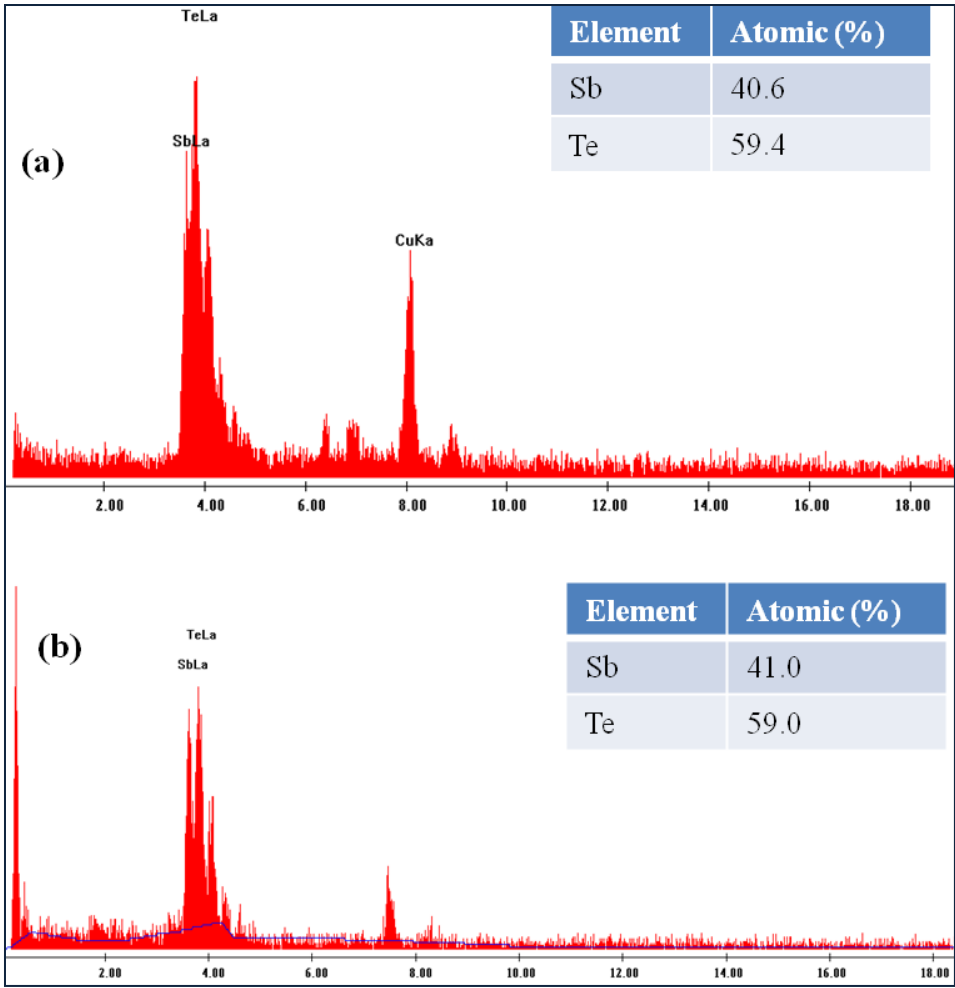


Figure S4: Energy-dispersive X-ray spectroscopy pattern indicating that the plates are composed of Sb_2Te_3 obtained from (a) Ph_2SbTeEt and (b) Ph_2SbTePh SSP.

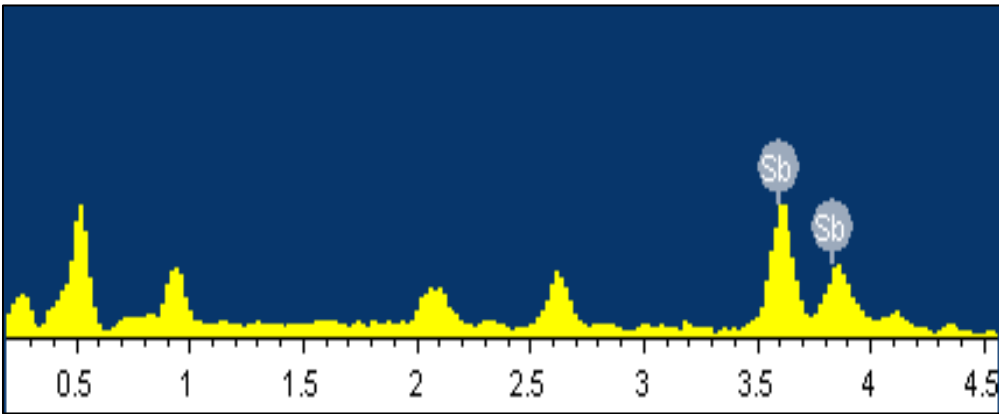


Figure S5: Energy-dispersive X-ray spectroscopy pattern of Sb nanocrystals obtained from Ph₂SbTePh SSP in the absence of solvent.

References:

- 1 M. Nunn, D. B. Sowerby, D. M. Wesolek, *J. Organomet. Chem.* 1983, **251**, C45-C46.