Aqueous Synthesis of Thiol-Capped CdTe Nanocrystals: State-of-the-Art

Andrey L. Rogach,* Thomas Franzl, Thomas A. Klar, and Jochen Feldmann Photonics and Optoelectronics Group, Physics Department and Center for NanoScience (CeNS), Ludwig-Maximilians-Universität München, Amalienstr. 54, D-80799 Munich, Germany

Nikolai Gaponik,* Vladimir Lesnyak, Alexey Shavel, and Alexander Eychmüller Physical Chemistry, TU Dresden, Bergstr. 66b, D-01062 Dresden, Germany,

Yuri P. Rakovich and John F. Donegan Semiconductor Photonics Group, Department of Physics, and CRANN Research Institute, Trinity College Dublin, Dublin 2, Ireland

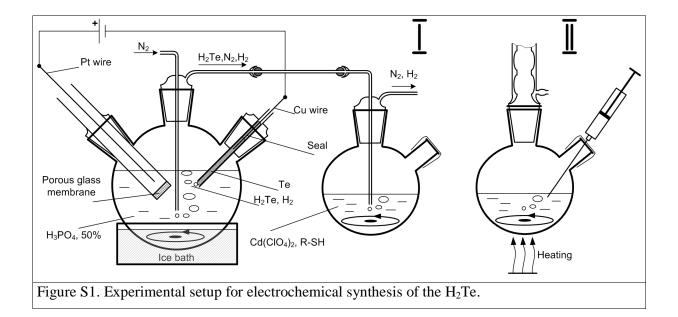
Supporting Information

Electrochemical generation of H₂Te gas used for the synthesis of CdTe nanocrystals

In our experiments the hydrogen telluride was produced by electrochemical reduction of a tellurium electrode in the electrochemical cell at room temperature according to the following reaction:

$$Te^{0} + 2H^{+} + 2e^{-} \rightarrow H_{2}Te^{\uparrow}$$
 $E_{0} = -0.51V [1]$

The electrochemical cell was equipped with a Te cathode and a Pt anode. The anode compartment was separated from the cell by a porous glass membrane. 50 wt % solution of the H_3PO_4 was used as the electrolyte. Figure S1 shows the details of the experimental setup.



The Te electrode was prepared as follows: Tellurium lumps (powder, Fluka) were placed in the glass tube and melted. A Pt contact wire was placed in the liquid tellurium. After cooling to room temperature, the empty part of the tube was sealed by epoxy resin and the glass end of tube was carefully broken providing a "naked" Te-surface.

Refs. [2,3,4] use this reaction at low temperature (-5C° to -10C°) in order to avoid decomposition of the H₂Te gas. At the current density used (380 mA/cm²) and at room temperature we did not find this effect to be significant.

In order to calculate the amount of H₂Te one may use following equation:

 $v = I t \eta/2 F$

(I - current strength, F = 96484.56: Faraday constant, η - electrochemical efficiency, t - duration of the experiment).

In order to estimate the effectiveness of our cell the followed experiment was carried out: 100 ml of cadmium nitrate solution (0.04 M) was placed in a three-necked flask fitted with a septum and valves and was de-aerated by Ar bubbling for 1.0 hours. The desired amount of H₂Te gas was passed through the solution, controlled by the duration of electrolysis (at 380 mA/cm² current strength). In order to remove all dissolved H₂Te gas, the flow of Ar was maintained for 30 additional minutes. The precipitate of CdTe was filtered out, washed with bi-distilled water, dried and weighed. The efficiency of our cell was 35% which is less than was published by Hodes for 50% H₂SO₄ electrolyte [4]. Altering the electrochemical efficiency during long term

use of the Te electrode can not be excluded [4]. Thus, periodic calibration of the electrochemical efficiency of the cell is necessary.

References

- 1. Lur'e, Y.Y., *Handbook on Analytical Chemistry*. 6th ed. 1989, Moscow, USSR: Khimiya. 446 pp.
- 2. Kovalenko, M.V., et al., *Spectral, Optical, and Photocatalytic Characteristics of Quantum-Sized Particles of CdTe.* Theoretical and Experimental Chemistry (Translation of Teoreticheskaya i Eksperimental'naya Khimiya), 2004. **40**(4): p. 220-225.
- 3. Kovalenko, M.V., et al., *Colloidal HgTe Nanocrystals with Widely Tunable Narrow Band Gap Energies: From Telecommunications to Molecular Vibrations*. J. Am. Chem. Soc., 2006. **128**(11): p. 3516-3517.
- 4. Bastide, S., et al., *Electrochemical preparation of* H_2S and H_2Se . J. Electrochem. Soc. 2005. **152**(3): p. D35-D41.