

Preparation and characterization of selected metal heavier chalcogenides

Student: Václav Kubát

Supervisor: doc. RNDr. Josef Novosad, CSc.

Metal selenides and tellurides are modern inorganic materials with a variety of applications, mainly in optical devices such as solar cells, but also in thermoelectrical devices and others [1]. There is considerable interest in studying various methods for the synthesis of these compounds, above all in a nanoscopic form.

One of the early methods uses a solvothermal reaction between Me_2Cd and trioctylphosphinechalcogenide (TOPE, E = S, Se or Te) to produce CdE [2]. This route is quite efficient, but requires high temperatures and utilization of toxic organometallic compounds. Another possibility is decomposition of single-source precursors (SSPs), i.e. coordination compounds that contain both metal (as a central atom) and chalcogen (as a donor atom of the ligand). The SSPs approach uses lower temperatures and less toxic chemicals compared to the solvothermal reactions, but it introduces a new difficulty. The ligand and thereafter the complex must be prepared and isolated, so the whole process of metal chalcogenide synthesis is rather complicated [3].

In our work we developed a new synthetic approach for the preparation of metal selenides and tellurides. Combining advantages of both mentioned methods we use a direct reaction between a metal source (metal carbonate or oxide), elemental chalcogen and a phosphine ($\text{Ph}_2\text{PNHPPPh}_2$ or $\text{Ph}_2\text{PCH}_2\text{CH}_2\text{COOH}$) to prepare the appropriate chalcogenide. The resulting chalcogenide is produced in one synthetic step as the whole process takes place in an autoclave.

As major part of the lecture the chemical nature of the process will be discussed. P-chalcogeno derivatives of $\text{Ph}_2\text{PCH}_2\text{CH}_2\text{COOH}$ were synthesized and structurally characterized as they most likely are key intermediates of the metal chalcogenide formation reactions. We presume that after a redox reaction between the phosphine and chalcogen a complex between metal cation and the P-chalcogen species is formed and it immediately decomposes forming a chalcogenide. We have demonstrated that our reaction system can be used for the preparation of cadmium, mercury and lead selenides and tellurides. The products are obtained in very good yields and according to PXRD and EDX analyses also in good purity. At last, reaction temperatures used for our process are below 200 °C.

References

- [1] M. Afzaal, P. O'Brien, J. Mater. Chem. 16 (2006) 1597 – 1602.
- [2] C.B. Murray, D.J. Norris, M.G. Bawendi, J. Am. Chem. Soc. 115 (1993) 8706 – 8715.
- [3] M. Afzaal, M.A. Malik, P. O'Brien, J. Mater. Chem. 20 (2010) 4031 – 4040.