

## MS49 How to...: crystallization for small and large molecules

Chairs: Aurelien Crochet, Andrew Maloney

### MS49-P1 Growing single crystals using thermal recrystallization

Philipp P. Nievergelt<sup>1</sup>, Bernhard Spingler<sup>1</sup>

<sup>1</sup> Department of Chemistry, University of Zurich, Switzerland

email: philipp.nievergelt@chem.uzh.ch

Growing of suitable single crystals is fundamental for the determination of the X-ray structure. Quite often, only an X-ray analysis reveals the composition of a compound. We have written a tutorial about the growth of single crystals, however this publication only discusses isothermal methods such as vapour diffusion or layering of different solvents.[1] There are very few reports about molecular single crystals that were grown by slow cooling.[2, 3]

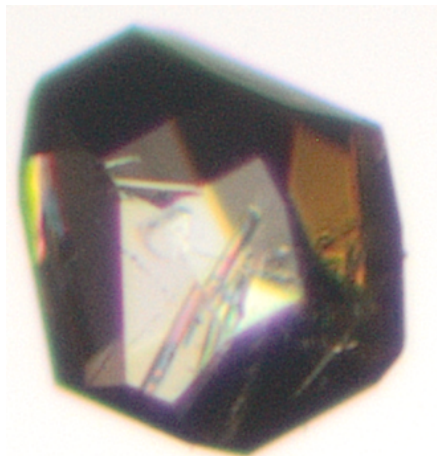
We have now studied for a series of very different compounds the growth of single crystals by thermal crystallisation with the help of the CrystalBreeder. For thermal recrystallization, it is essential to know the solubility in the chosen solvent at a selected temperature in order to obtain supersaturation at the cooling step. We observed that thermal recrystallization can be performed very fast – suitable carbamazepine and *p*-amino benzoic acid single crystals could be obtained only within one day – more importantly the obtained crystal quality for these compounds was excellent. Furthermore, we only used small amounts (as little as 1 mg) of material which is of course very advantageous.

[1] B. Spingler, S. Schnidrig, T. Todorova, F. Wild, *CrystEngComm* **2012**, *14*, 751.

[2] R. W. Seidel, J. Graf, R. Goddard, I. M. Opperl, *Acta Cryst.* **2011**, *E67*, m236.

[3] T. Notake, K. Nawata, H. Kawamata, T. Matsukawa, H. Minamide, *Opt. Mater. Express* **2012**, *2*, 119.

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200  $\mu\text{m}$

Figure 1. Carbamazepine crystal grown in isopropyl alcohol within six hours

**Keywords:** Crystal growth, carbamazepine, *p*-amino benzoic acid, polymorphs, single crystals, thermal recrystallization