Crystal seed growths and the possibilities of a controlled crystallization from solution

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Various sophisticated techniques exist for crystallization, mostly of proteins. However, nothing was reported for simple automated crystallization devices in the field of routine crystallography of small molecules.

For a control of crystallization processes in solution it is indispensable to detect the size and number of crystal seeds. Light scattering represents one of the methods for particle detection, the analysis of MIE-Scattering allows for empirical determination of particle sizes in the range of one to approximately 100 u m. The intensity of a scattered laser beam in a given angular range can be used as a measure of particle sizes, the number of particles (counts) is a measure of the number of events in a given volume. We decided to apply a commercial available 'low-cost' instrument (SPECTREX particle counter SX10S, Fig. 1) which enables us to use corvettes of our own design to be used as crystallization vessels.

For a temperature control of the crystallization it was required to develop a computer control (ASCOT) which runs together with the particle counter software in order to link the temperature with the particle counting process on the same time base (clock).

First experiments with this device clearly demonstrate that it is possible to detect the crystal seeds which appear when cooling a saturated solution of anthracite in toluene (Fig. 2). Although a stirring process was continued during the experiment the crystal seeds would settle at the bottom of the vessel. Further detection of the crystal growths was not possible.

The continuation of a crystal growing process may be observed at the bottom of the vessel by means of a video camera. The pictures may be grabbed by a computer at time intervals and compared to give an idea of the increasing (possibly decreasing) size of the crystals.

The experiments show that there exist simple possibilities for an automated detection of crystal seeds at a given temperature to be analyzed by a computer and for a further automated control of a crystallization process by analyzing video pictures.

Other possibilities of crystal seed detection and control of crystallization processes for routine work shall be discussed.

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