In situ X-ray for monitoring crystallization in solution : In-situX®

S. Clevers[1], **M.Sanselme**[1], **Y. Cartigny**[1], **G. Coquerel**[1]

[1]Laboratoire Sciences et Méthodes Séparative EA3233, Normandie Université, Université de Rouen, Mont Saint Aignan, 76821 cedex France

Introduction Crystal growth from solution is widely used in many fields for industrial crystallizations (e.g. pharmaceuticals, food chemistry) as well as for academic purposes (e.g. solvent-assisted phase transitions). To fully understand crystallization processes, it is essential to know all of the solid phases that may appear in suspension. In this context, a new laboratory prototype based on X-ray diffraction to perform in-situ analyses directly in the crystallization reactor was developed: In-situX® technology [1]. In-situX® is designed to monitor crystallizations directly inside a reactor by means of XRPD. The sample (e.g. mixture of solid and liquid) is placed in a double-jacketed reactor and analyzed in suspension without any sampling (Figure 1). The reactor is mounted in a Bruker® D8 diffractometer inverted geometry ($-\theta$ /$-\theta$) goniometer. The temperature of the reactor can be accurately controlled in a wide temperature range (-70degC/+70degC) without any icing. The sample is analyzed in real-time at the bottom of the reactor (thin membrane transparent to X-rays, mechanically and chemically resistant) all along crystallization processes. Applications of the In-situX® technology In-situX® has already proved its efficiency to deal with characterization of new solid forms, for polymorphic identifications, polymorphic screening, solvent assisted solid-solid transitions, crystallization of hydrates, solvates or co-crystals, for purification and resolution processes and during maturation of solid phases.[1] In-situX® can be also used for studying solid/vapor equilibria in order to analyze the stability of solvates (hydrates). Through selected examples among possibilities cited above, this presentation aims at proving the efficiency of In-situX® technology to characterize suspensions during a crystallization process.

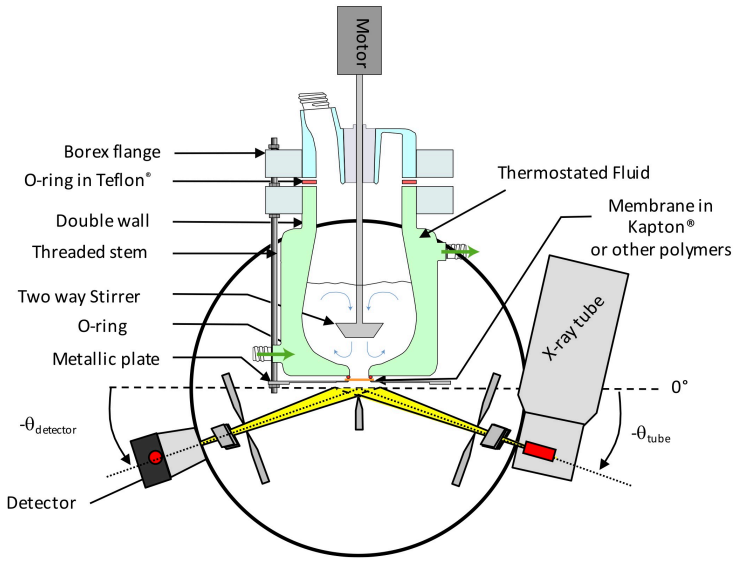

Figure 1: Schematic view of the reactor and the goniometer geometry in the In-situX® technology

References

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[2] H. Takahashi, S. Iwama, S. Clevers, S. Veesler, G. coquerel, H. Tsue, and R. Tamura, Crystal Growth Design, vol. 17, no. 2, pp. 671–676, 2016.