

STK meeting, reaction calorimetry – case study

Abstract:

A batch reaction initiated thermally (temperature ramp) was studied using different calorimetric measurements (Reaction calorimetry, DSC and VSP). These measurements were used to assess the criticality of the reaction system and to estimate the consequences of several process deviations. The process showed a significant criticality. Indeed, a simple process deviation could lead to a pressure increase of above 16bar within less than 1 hour.

Different options were then studied to enhance the safety of the reaction system while maintaining all quality requirements. Several attempts like increasing the amount of solvent or dosing one of the reactive lead to an enhanced safety but the quality requirements of the final product could not be met. In this presentation, the modification done to the process to make it safer will be presented. The process modification was combined with the search of an inhibitor and with the dimensioning of the pressure relief system.

The newly developed system was re-evaluated with calorimetric measurements and showed a significantly enhanced robustness to process deviations. The quench with an inhibitor was also tested and optimized using calorimetric measurements.