Evaluation of apparatus effects on calorimetric data as a basis for safe operating limits in thermal process safety

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Process safety in chemical industry acts within the area of conflict between economic and safety requirements. Sufficient safety measures are required in order to reduce risk to an acceptable level, protecting human life, the environment, and surrounding assets. But on the other hand, overly conservative safety measures can be adversely affect the economic aspects of the process. Therefore, the better we can characterize the safe operating window of a chemical process the better we are able to apply suitable measures to fulfill both safety and economic requirements.

The assessment of thermal process safety is essentially based on various calorimetric methods. When setting permissible safe operating limits, the measurement uncertainty, detection sensitivity and other factors that may influence the validity of the methods must always be identified and, if necessary, considered as an additional safety margin. In the case of simple screening methods, the required safety margin is correspondingly greater. If higher-quality methods with more precise knowledge of the boundary conditions and influencing factors are used, it is possible to get closer to the permissible limits while maintaining the same level of safety [1]. Using the simple example of evaluating the stability limit of reactants and reaction products, the differences can be shown:
The required limiting safety temperature can be estimated by a simple rule of thumb like the 100K-rule related to the DSC onset or derived more precisely by thermo kinetic modelling of DSC-curves or via adiabatic calorimetry. Here, high quality data is required. In general, experimental data is affected by several apparatus effects as mentioned before.
In many cases the usage of non-deconvoluted DSC-raw data for thermo kinetic modelling leads to conservative adiabatic induction periods resulting in a limited operation window. To enlarge the permissible safe operation windows and therefore increase the cost efficiency of a chemical process a fundamental knowledge of the apparatus effects on experimental data is essential. Furthermore, this knowledge is useful for efficient experiment design in adiabatic calorimetry within the context of an industrial laboratory environment. In the following presentation the influence of apparatus parameters on experimental data and derived safety limits by thermo kinetic modelling is evaluated in the context of thermal process safety. The authors are going to present results of an internal method sensitivity study with focus on DSC and adiabatic calorimetry.

Literature

[1] TRAS 410 – Erkennen und Beherrschen exothermer chemischer Reaktionen, Stand: Dezember 2020.