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Gesellschaft für Anlagensicherheit
Höchst zuverlässig.

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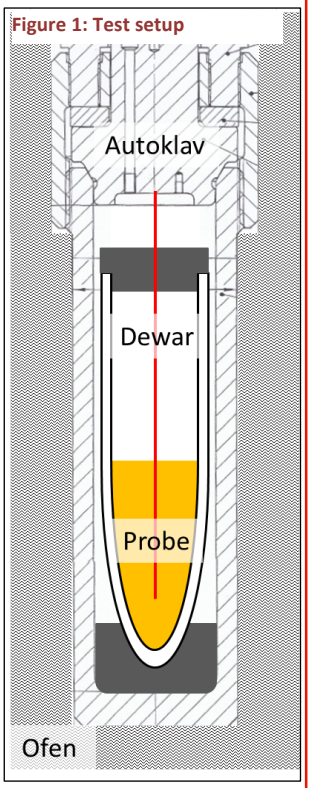
Thermal hazards: identify, assess, control...
Part II: Help! My process temperature exceeds the limit temperature

In our last consiLetter we showed how you can obtain a limit temperature for safe handling and use at an early stage in the development of a product or process and using a very small amount of substance in a screening test in the differential scanning calorimetry (DSC) cell. In some cases, however, the limit temperature specified with the DSC is very low and significantly below the desired process temperature. Because changing the process is often extremely difficult, we recommend in these cases more in-depth analysis of thermal stability. One of these additional assessments is the adiabatic heat-pressure accumulation test. Here, approximately 100 g of the substance is weighed out in a double-walled vacuum jacket (Dewar) with a reflective coating. This Dewar vessel is then inserted into a stainless steel autoclave, which is placed for the duration of the test into a massive aluminum block furnace, Fig. 1. This setup ensures that there is no exchange of heat or substances with the surrounding environment ("adiabatic conditions"). To replicate the actual process as closely as possible, stirring or dosing of additional components is possible. In addition to the temperature in the sample, the pressure in the headspace of the autoclave is also measured.

Figure 1: Test setup

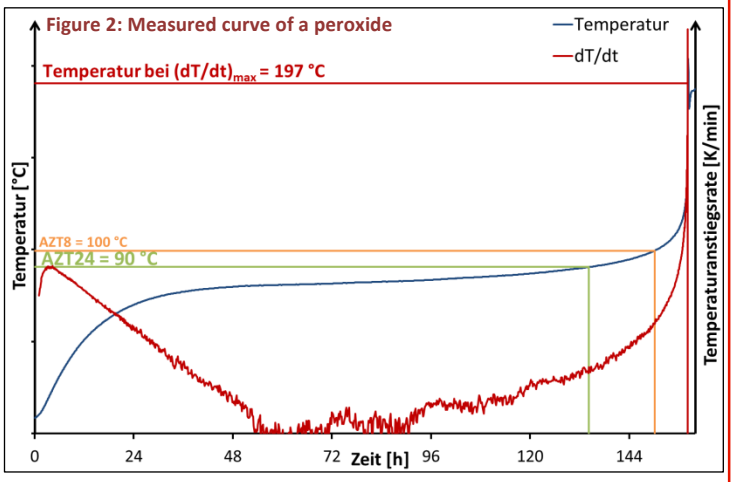


An adiabatic measurement of this type not only simulates the worst-case scenario of a malfunction, or failure of cooling, but the adiabatic temperature rise (ΔT_{ad}) can also be derived from the test. The adiabatic temperature rise indicates the temperature difference by which the sample may heat, independent of the volume or mass of the sample. Another parameter of interest for a thermally safe process, which is likewise obtained from the measured data, is the adiabatic induction time (TMR_{ad}). This shows the time span within which, under adiabatic conditions, the maximum rate of temperature rise is reached.

The adiabatic decomposition temperature for 24 hours ("**A**diabatische **Z**ersetzungs**T**emperatur" AZT24) describes the temperature at which the process requires 24 hours, under adiabatic conditions, to reach the maximum rate of temperature rise. In accordance with TRAS 410, the limit temperature for safe handling (T_{exo}) was selected at 10 K below the AZT24.

Figure 2 shows as an example of the temperature profile (blue), as well as the temperature rise rate (rot) from a test with a peroxide. First, the adjustment of temperature to the set oven temperature is observed. Afterward, an initially subtle temperature rise is observed. Due to the adiabatic test process, the heat produced cannot be dissipated and the product continues to become warmer, ultimately transitioning to an exponential temperature rise. The maximum temperature rise rate is detected at a temperature of 197 °C. The AZT24 is obtained by going back 24 hours from this point. For the decomposition of the peroxide, this is 90 °C. The T_{exo} is therefore arrived at by subtracting the safety margin of 10 K, for 80 °C. Consequently, the peroxide can be safely handled at a temperature of up to 80 °C.

Figure 2: Measured curve of a peroxide



The observed adiabatic decomposition of the peroxide not only leads to an adiabatic temperature rise of about 210 °C but also is accompanied by pressure buildup. In the next issue of the consiLetter we will show you what you can do when the pressure buildup identified in an adiabatic test exceeds the design limits of the plant.

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