

Exercise

Substitution reaction in semi-batch reactor (DSC)

Process

A reaction following the equation $A + B \rightarrow P$ is to be performed as semi-batch in a 4 m^3 reactor. The initial charge is 2000 kg of a solution of A at a concentration of $3 \text{ mol} \cdot \text{kg}^{-1}$. This solution is heated to 80°C , the intended reaction temperature. Then 1000 kg of reactant B (concentration $7.5 \text{ mol} \cdot \text{kg}^{-1}$) are added at a constant rate.

Potential

The reaction mass is studied by DSC. A sample of the reaction mass before addition of reactant B presents a flat thermogram. The thermogram of the final reaction mixture is represented in Figure 1.

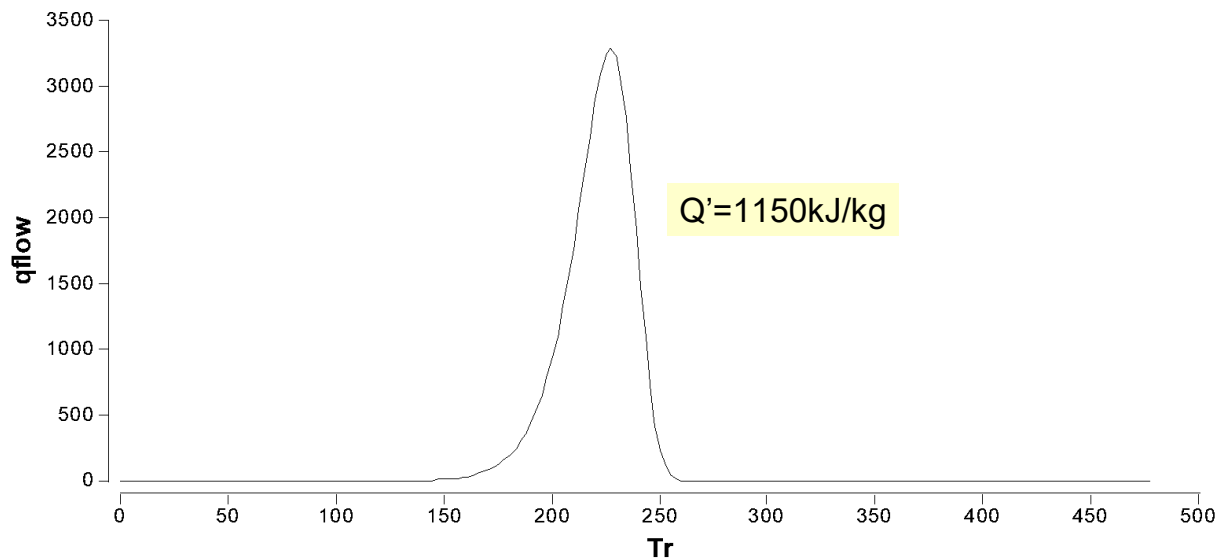


Figure 1: DSC Thermogramme of the final reaction mass recorded with a scan rate of 4 K/min in gold plated pressure resistant crucible. Heat release rate (qflow) in W/kg, temperature (Tr) in °C.

The specific heat capacity of the reaction masse is $c'_p = 1700 \text{ J} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$. The density is $1000 \text{ kg} \cdot \text{m}^{-3}$. The solvent is xylene with $T_b = 140^\circ\text{C}$.

Question 1:

Assess the severity in case of runaway of the decomposition of the final reaction mixture.

A thermogramme of a reaction mass with both reactants mixed at room temperature was also recorded. This thermogram is represented in Figure 2.

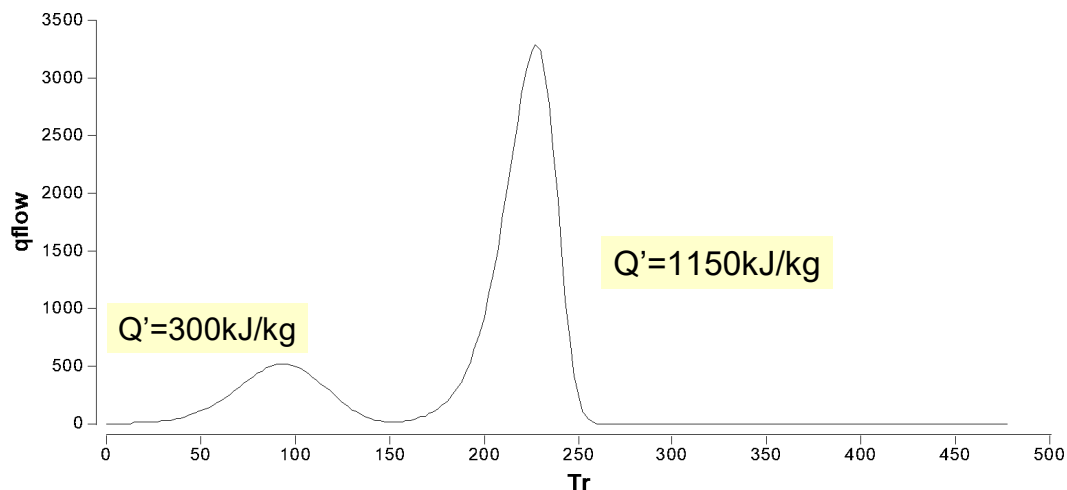


Figure 2: DSC Thermogram of initial reaction mixture recorded at 4 K/min in gold plated pressure resistant crucibles. Heat release rate (qflow) in W/kg, temperature (Tr) in °C.

Question 2

Calculate the adiabatic temperature rise and the MTSR.

Build a worst case cooling failure scenario. Do you think this reaction is feasible as batch reaction?

Triggering probability for the decomposition of the final reaction mass

The final reaction mass was studied in a series of isothermal DSC experiments in the temperature range between 175 and 195°C. The results are represented in Figure 3.

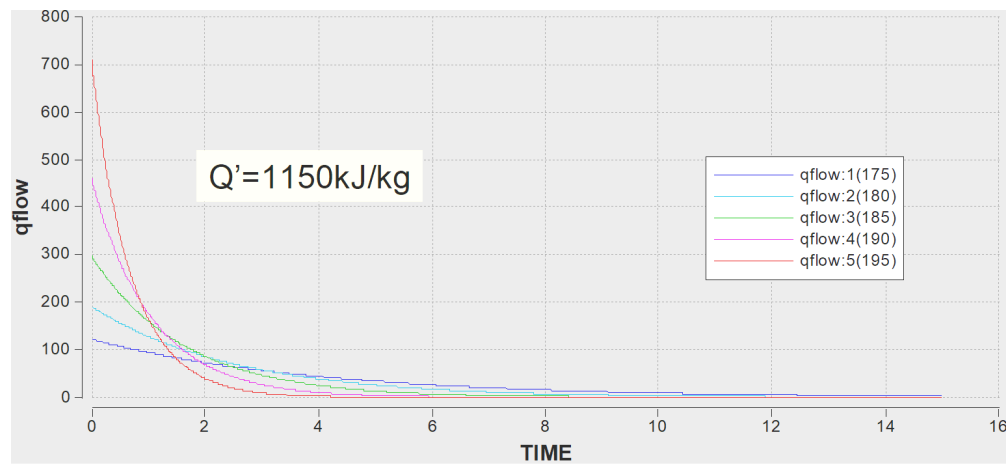


Figure 3 : DSC Thermograms of final reaction mass in isothermal mode at 175, 180, 185, 190 and 195°C recorder in gold plated pressure resistant crucibles.

The decomposition energy is always close to 1150 kJ/kg. the results are summarized in Table 1.

Table 1: Results of isothermal DSC measurements

Temperature (°C)	Maximum heat release rate (W/kg)
175	120
180	190
185	300
190	460
195	700

Questions 3:

Is the decomposition autocatalytic?

At which temperature is the TMR_{ad} 24 hours (T_{D24})?