

Process Modification

Improves Safety and Shortens Downtime

- Production restarted after 1 month
- Considerable savings in time and money achieved
- Process safety improved

The challenge

A running production from a major company in the dyestuffs and chemicals sector proved to be problematical. Solvent losses during work-up resulted not only in increased costs, but they also represent an undesirable load on the environment.

The reaction calorimeter was used in an attempt to develop an improved process in a short time span. Initial investigations showed the existence of a more serious safety problem in addition to the ecological problem, which necessitated immediate shutdown of production. A quick solution to this problem became extremely important as product could no longer be delivered to customers.

The process

The reaction, a Friedel-Crafts acylation, is started in the melt at 65°C. The reaction mixture is then kept for one hour each at 70° and 90°C. After cooling to 65°C the reaction mass is dissolved in chlorobenzene for subsequent processing.



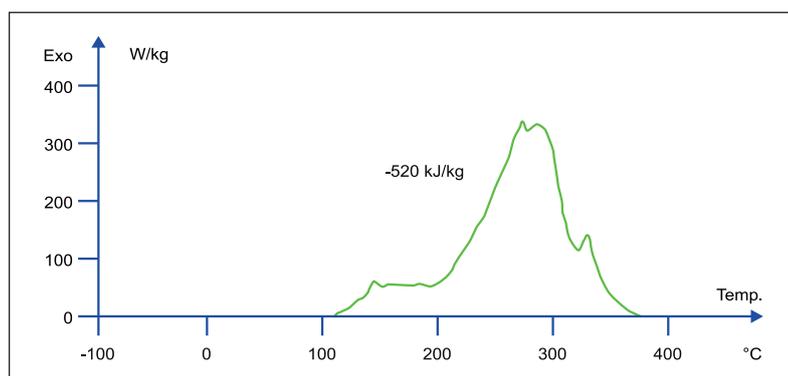
Safety problems

The viscosity of the melt may increase to such an extent that the reaction heat will mostly be accumulated in case of a stirrer breakdown. As can be seen from the DSC measurement (Fig. 1), severe decomposition sets in above 200°C. A thermal potential of over 500 kJ/kg is present in the reaction



mass. Since it is virtually impossible to dissipate the heat if there is a malfunction, a thermal explosion must be anticipated within hours. As there are hardly any possibilities for emergency action, production had to be stopped when these facts became known.

Figure 1
DSC measurement
of the reaction
mixture after
1 hour at 70°C



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Modification of the procedure

Chlorobenzene was replaced by a solvent that has a higher boiling point and a lower solubility in water. To reduce the viscosity of the reaction mass, the reaction is now run, right from the start, in solution and not in the melt.

Thanks to the highly reproducible performance of the RC1_e only few experiments were needed for optimization and testing of the new procedure (Fig. 2).

The quality of the product was checked by HPLC in a duplicate RC1_e experiment with the following results:

	Exp. 1	Exp. 2
Desired product	97.8%	98.3%
Undesired isomers	<0.1%	<0.1%
Sum of elutable unknown substances	<0.1%	<0.1%

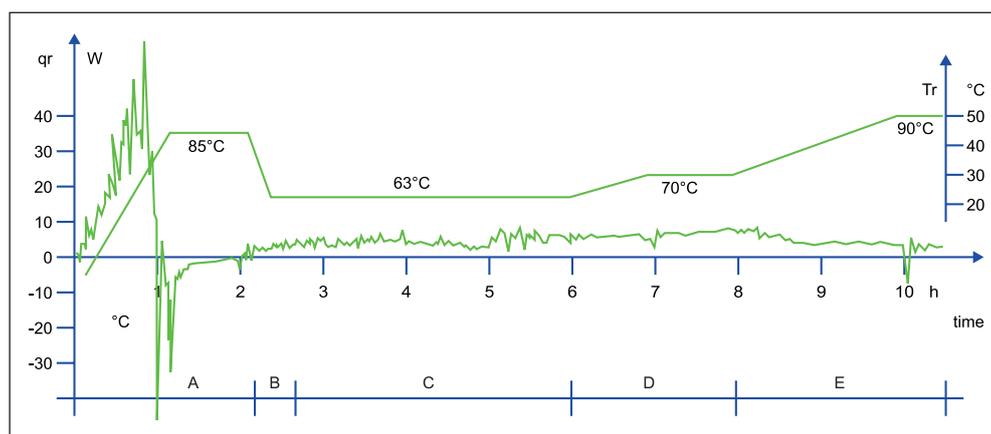


Figure 2

Automatic RC1_e experiment with the optimized procedure where:

- A: Addition of AlCl₃ with simultaneous heating and holding at 85°C
- B: Cooling to 63°C and holding for 15 minutes
- C: Dosing of the arene at 63°C
- D: Heating up to 70°C and holding for 1 hour
- E: Heating up to 90°C and holding for 0.5 hour

Benefits achieved

Thanks to precise and comprehensive process data it was possible to restart operations with the modified procedure after an interruption of only 1 month. The company was thus able to resume deliveries within a short time. Without the availability of an RC1_e the same task would have taken

at least 6 months: A large number of lab experiments would have been necessary; and for reasons of safety as well as for final optimization a pilot study would have been essential. Moreover, the product shortage would have resulted in considerable commercial losses.

Because no pilot study was needed considerable savings in time and money were achieved. The safety of the process is now assured, even if a malfunction occurs, as the viscosity of the reaction mass is appreciably lower.

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