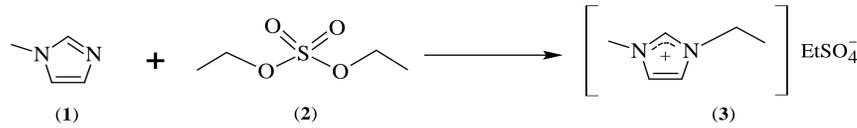


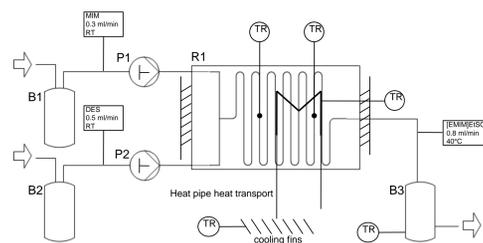
## Introduction



- Long latency period of 15 seconds,  $E_a = 89 \text{ kJ mol}^{-1}$  [2]
- Very fast and highly exothermic,  $\Delta H = 130 \text{ kJ mol}^{-1}$  [2]
- Second order kinetics [3]

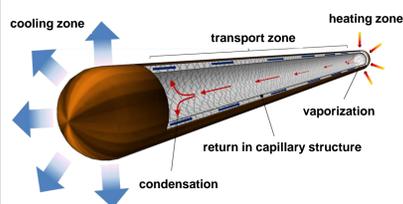
The synthesis of [EMIM]EtSO<sub>4</sub> (1-ethyl-3-methylimidazolium ethyl-sulfate) (3) from the respective reactants 1-methyl-imidazole (2) and diethylsulfate (3) suffers from the highly exothermic and self-accelerating reaction [1,2]. Recently we investigated the applicability of heat pipes for cooling highly exothermic reactions [4]. Heat pipes are advantageous due to their fast dynamic cooling and heating behavior. By heating the reactor via heat pipes connected to an external heat source (hot air stream), the reaction can be stabilized inside of the reactor. The reaction becomes self-stable (at 100°C), but due to the dynamic cooling of the heat pipes the temperature at the hot end is remarkably higher depending on the reaction heat release.

## Experimental setup

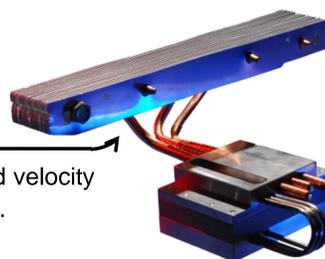


- Surface area: 970 mm<sup>2</sup>
- Volume: 290 mm<sup>3</sup>

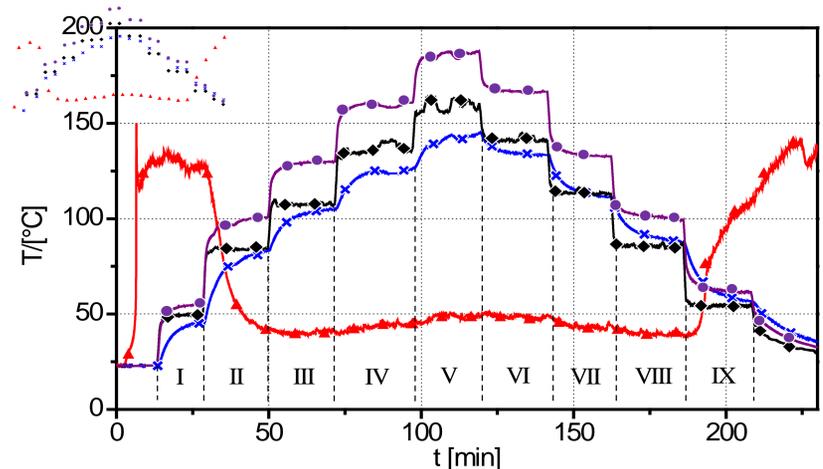
- Stepwise increasing of the reactor temperature by an external heat source yields to an optimal operating temperature.
- The best product quality results in section II, III, VII and VIII corresponding to the lowest temperature in the collecting basin. In section IV, V and VI coloration occurs. In I and IX impurities appear.



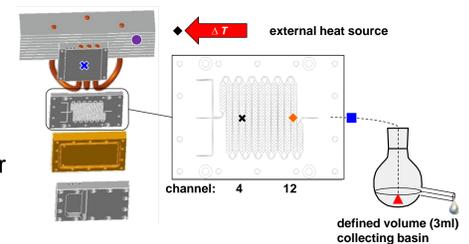
- High flexibility
- Heat transport in sonic sound velocity due to physical reasons (left).



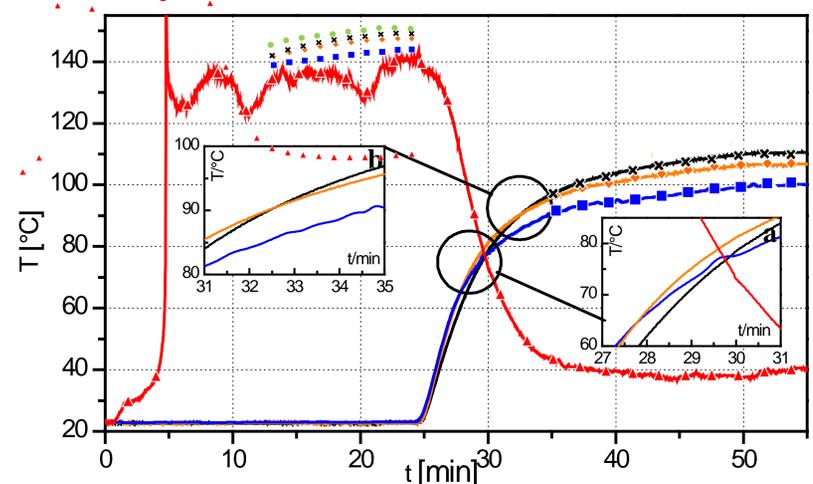
## Temperature measurements



- Explosion view (right) of the used heat pipe controlled micro reactor.
- Temperature measuring spots for the temperature curves are marked.

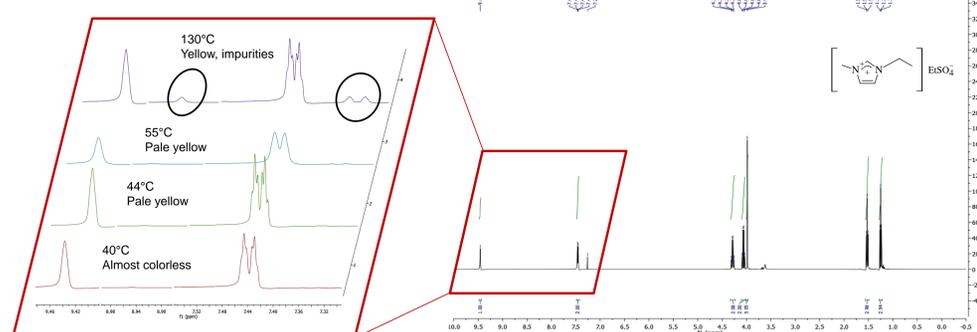
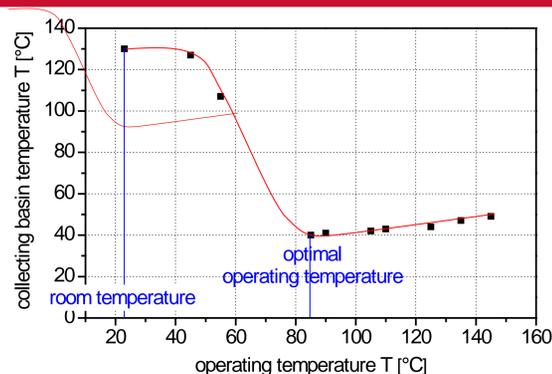


- An uncontrolled reaction in the collecting basin without external heating can be avoided.
- During heating via the heat pipe system the temperature curves show typical characteristics (right).
- The highest temperature is present in the withdrawal channel and falls below the temperatures in the micro channels during permanent heating (intercepts of blue line in a).
- Finally the temperature in channel 4 exceeds all temperatures. The reaction is shifted into the first micro channels (intercepts of orange line in b).



## Results

- After stepwise heating of the reactor, the optimal operating temperature can be found by plotting the outlet temperature as a function of the operating temperature.



## Summary

- The used set-up allows a self-mediated control of highly exothermic reactions.
- The dynamic cooling and heating behavior of the used heat pipes allows an independent increase of volume flow.
- Due to optimization of operating temperature of 85-110°C best product quality was achieved.
- The final product appears as a clear non-colored liquid indicating that no hot-spots occur inside the reactor.

## References

- [1] A. Renken, V. Hessel, P. Löb, R. Miszczuk, M. Uerdingen and L. Kiwi-Minsker, *Chem. Eng. Proc.*, 2007, **46**, 840-845.
- [2] H. Löwe, R. D. Axinte, D. Breuch, C. Hofmann, J. H. Petersen, R. Pommersheim and A. Wang, *Chem. Eng. J.*, 2010, **163**, 429-437.
- [3] A. Große Böwing and A. Jess, *Chem. Eng. Sci.*, 2007, **62**, 1760-1769.
- [4] H. Löwe, R. D. Axinte, D. Breuch, T. Hang and C. Hofmann, *Chem. Eng. Technol.*, 2010, **33**, 1153-1158.