

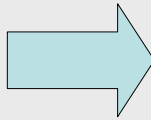
Safety Aspects of the Process Control of Grignard Reactions

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Motivation and objectives

Hazard potentials of Grignard Reactions:

- very high reaction enthalpy
- spontaneous heat release during initiation stage
- delayed reaction start due to traces of impurities
- exothermic consecutive reactions of Grignard reagents with impurities

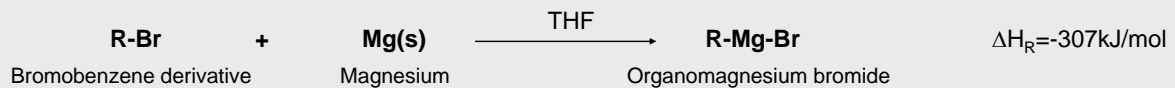


Industrially applicable on-line methods for:

- objective detection of reaction start-up
- monitoring of educt accumulation during the semi-batch process

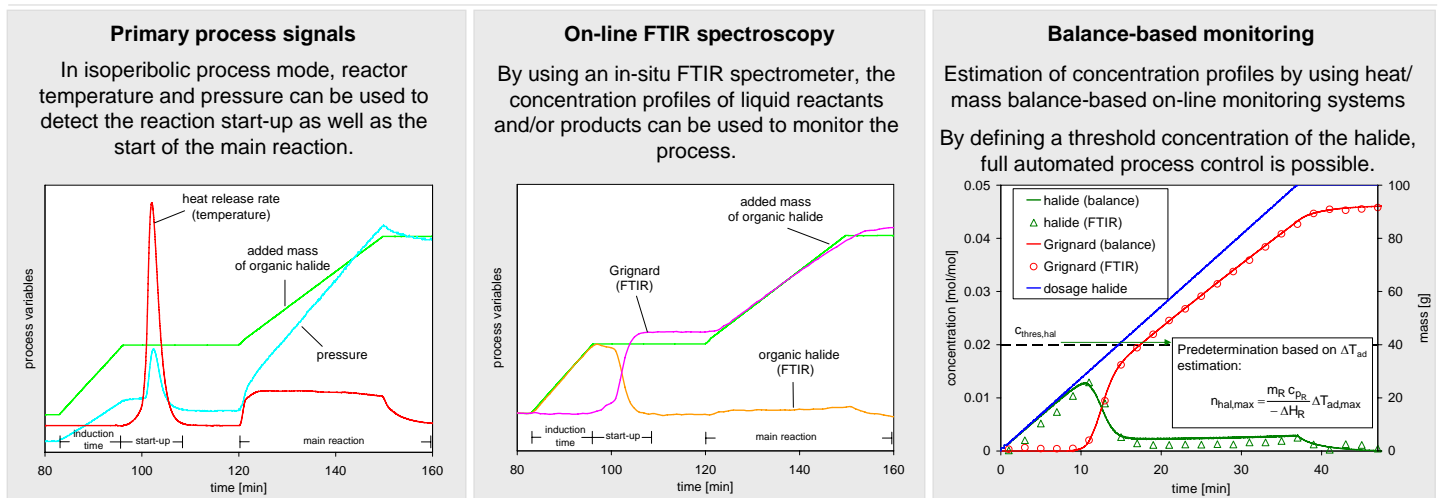
Experiments

Development and test of monitoring methods by Grignard Reaction experiments in a **closed** reaction calorimeter (RC1/HP60, **without** reflux equipment)



In-situ concentration measurements using FTIR-spectrometer ReactIR1000

On-line detection of Grignard Reactions in pressurised STR



method	primary process signals (p_R, T_R)	on-line FTIR-spectroscopy (qualitative)	on-line FTIR-spectroscopy (quantitative)	balance-based monitoring
start-up detection	+	+	+	+
quantitative monitoring	-	-	+	+
pros	easy to use cost-efficient	independent of process control	independent of process control	cost-efficient virtually independent of process control (pressurised STR recommended)
cons	dependent on process control misinterpretations possible	high investment cost high operating costs local measurement	high investment cost high operating costs high calibration effort local measurement	advanced knowledge of process and plant parameters required

Conclusions

- on-line monitoring of Grignard processes in closed STR based on primary process signals, FTIR spectroscopy and heat/mass balance-based monitoring systems possible
- advanced quantitative on-line monitoring methods opens up new perspectives towards full automated process control of Grignard reactions
- Recommendation:**
 - process control in closed (pressurised) STR without reflux cooling system
 - on-line monitoring using heat/mass balance-based systems (i.e. MoSys[®])

* MoSys: On-line Monitoring System using adaptive heat/mass balances developed by FZD/ Fraunhofer Institut Umsicht/ Degussa AG