

21.-22.08.2017



SENSOR ROADMAP 4.0 – PROSPECTS TOWARDS A UNIFORM TOPOLOGY FOR PROCESS CONTROL AND SMART SENSOR NETWORKS

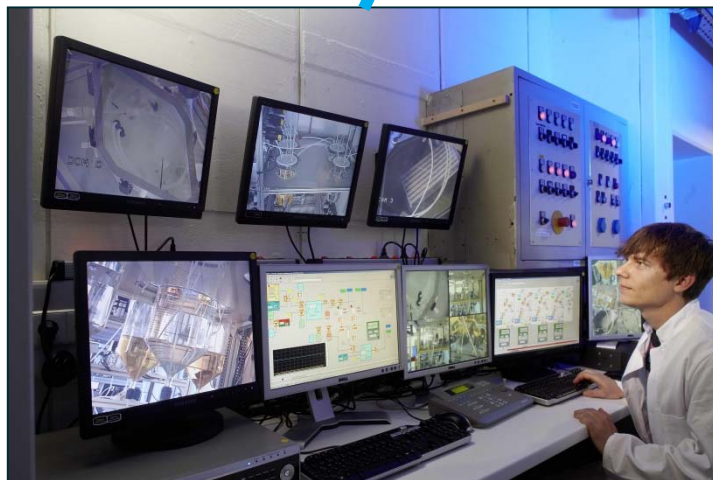
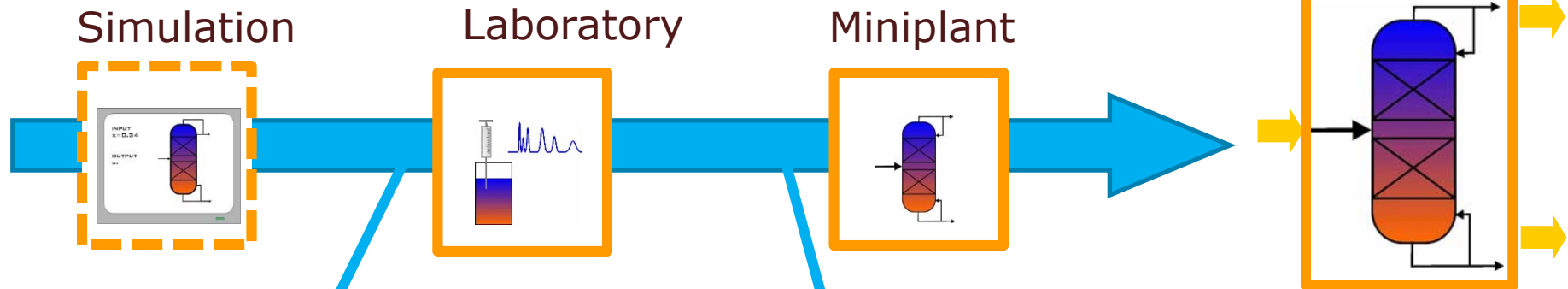
Michael Maiwald, Patrick Gräßer, Lukas Wander,
Svetlana Guhl, Klas Meyer, Simon Kern

Bundesanstalt für Materialforschung und -prüfung (BAM)
Division 1.4 Process Analytical Technology

Richard-Willstätter-Str. 11
D-12489 Berlin, Germany

Integrated Process Design and Intensified Processes

Current Trend: Hybrid Modular Plants



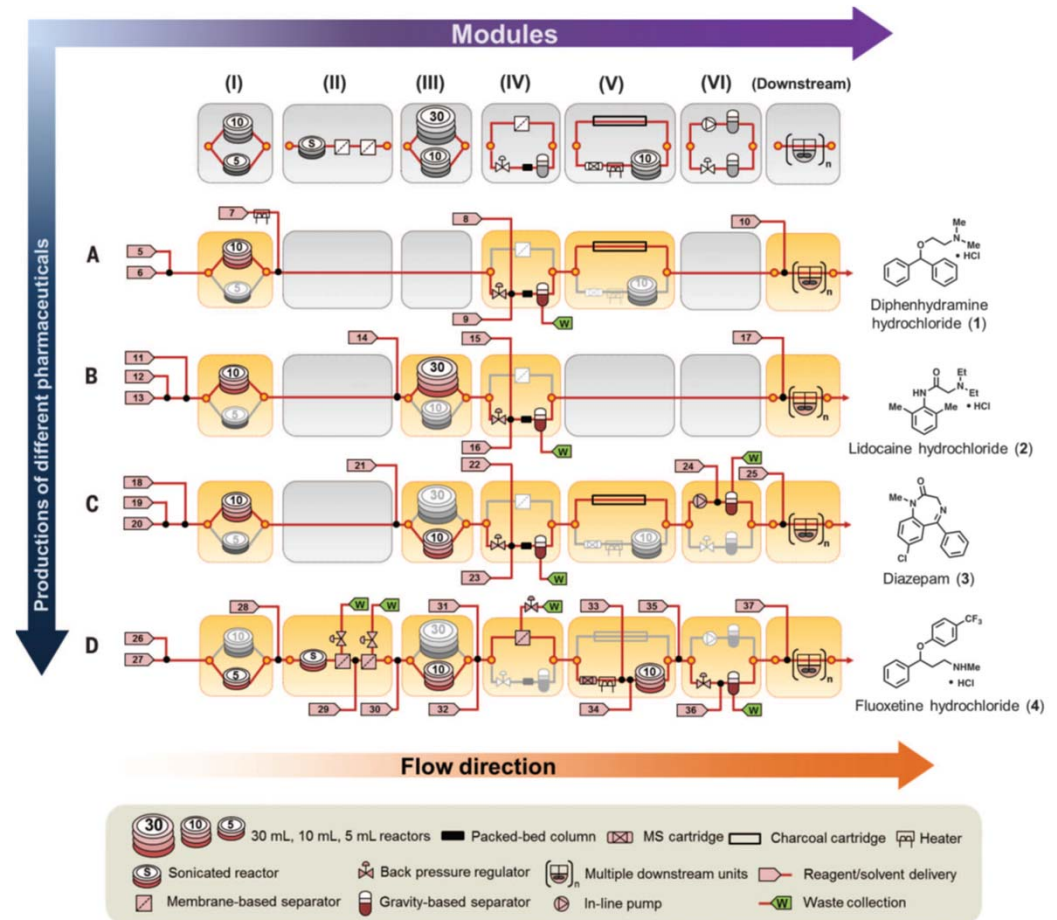
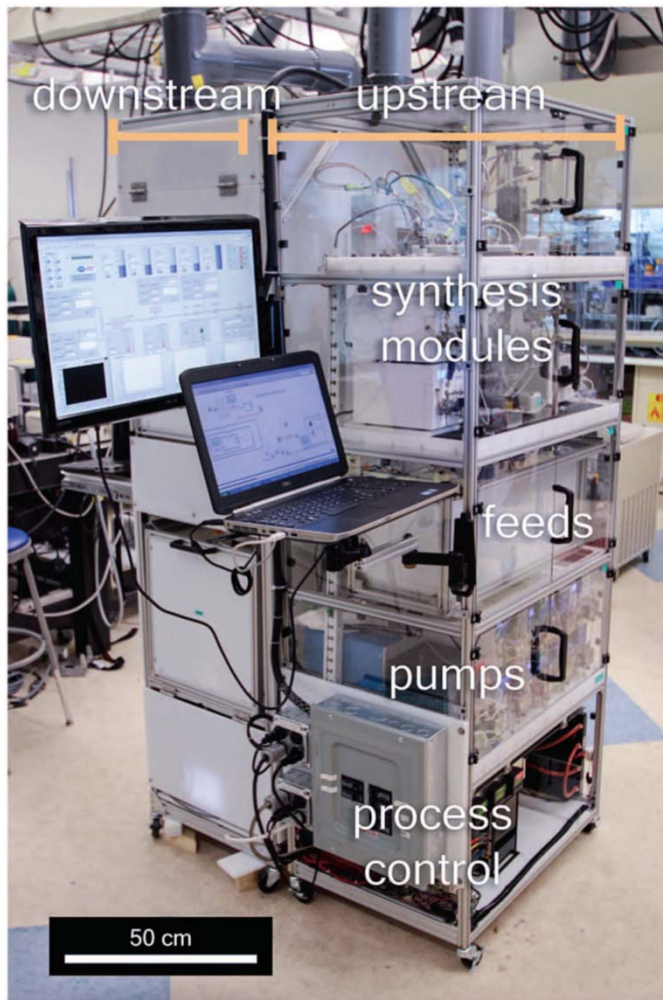
Micro Reaction Technology
(Example: Fraunhofer ICT, Pfinzta/D)



Modular Production
(Example: Foster Wheeler AG, Zug/CH)

Integrated Process Design and Intensified Processes

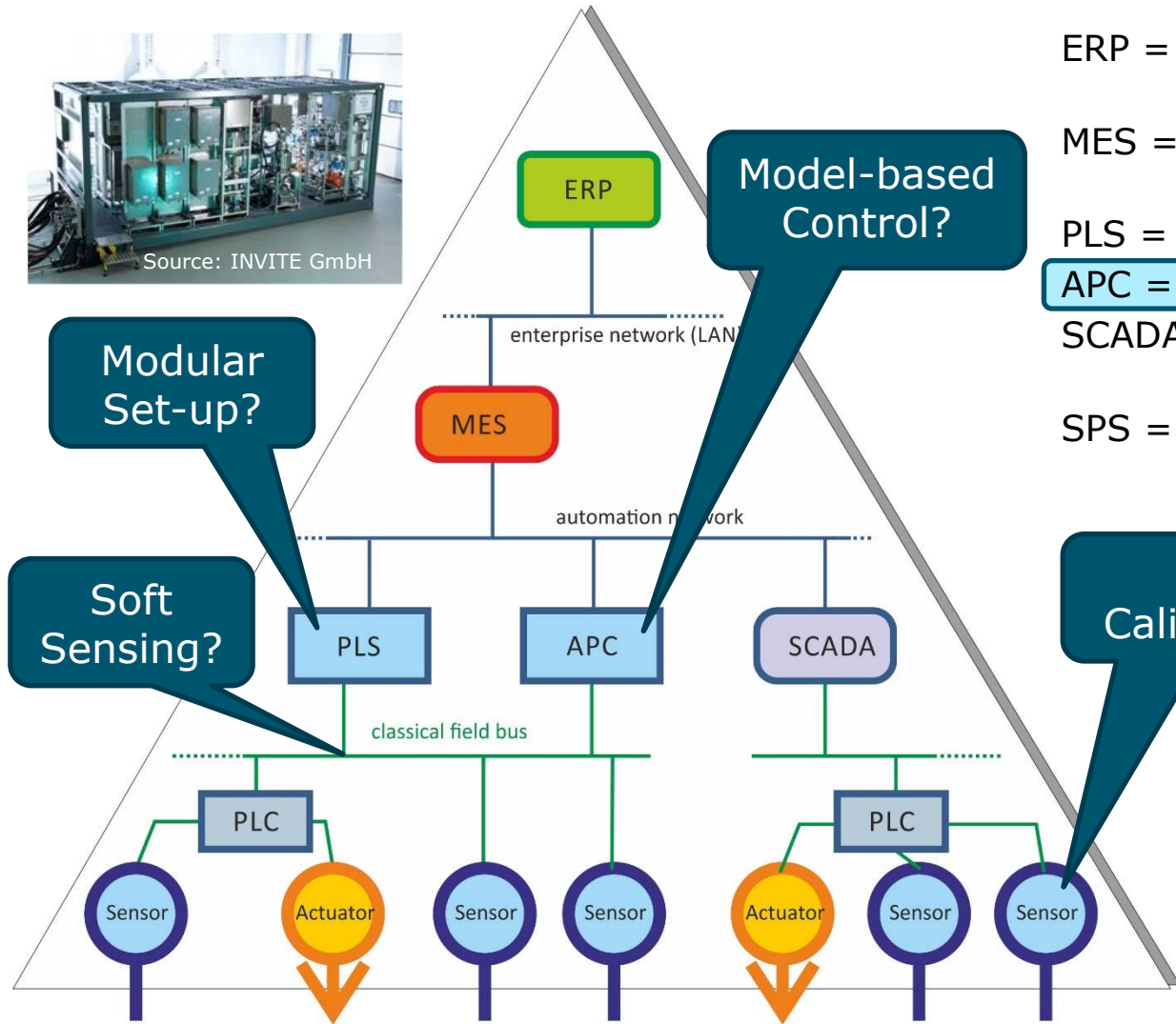
Example: Continuous Flow Production of Pharmaceuticals



A. Adamo, R. L. Beingessner, M. Behnam et al., On-demand continuous-flow production of pharmaceuticals in a compact, reconfigurable system, *Science* 352(6281), 2016, 61-67

Process Automation

The Classical Automation Pyramid → is Outdated!



- ERP = Enterprise Ressource Planning
- MES = Manufacturing Execution System
- PLS = Prozessleitsystem
- APC = Advanced Process Control**
- SCADA = Supervisory Control and Data Acquisition
- SPS = Speicherprogrammierbare Steuerung



Success of Technology Roadmaps

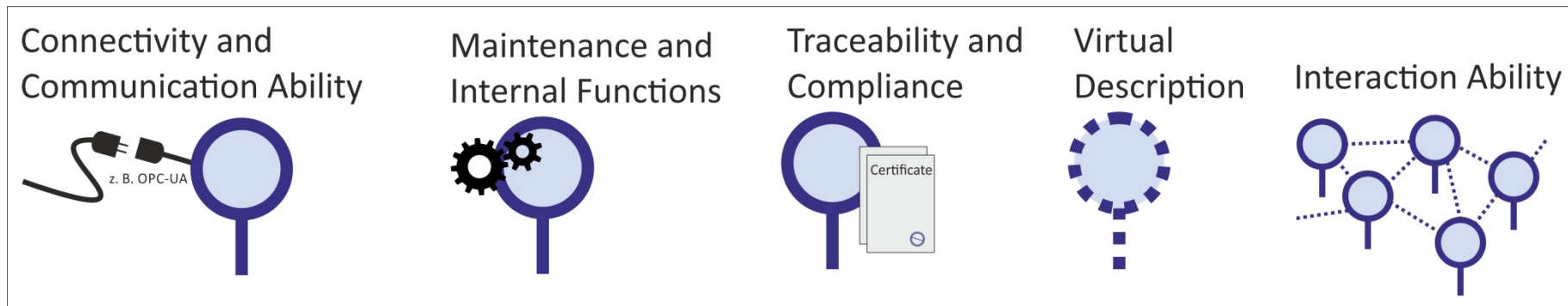


The former technology roadmaps „Prozess-Sensoren 2005–2015“ (2006) and „Prozess-Sensoren 2015+“ (2009) were a valuable basis for all enterprises in the process industry

Current and future requirements to process sensors

Sensors go *smart*

- Smart process sensors are important parts of cyber-physical production systems and generate new business cases for users, instrument manufacturers, and service providers.
- „Smart“ sensors provide services within networks and use information out of the network.



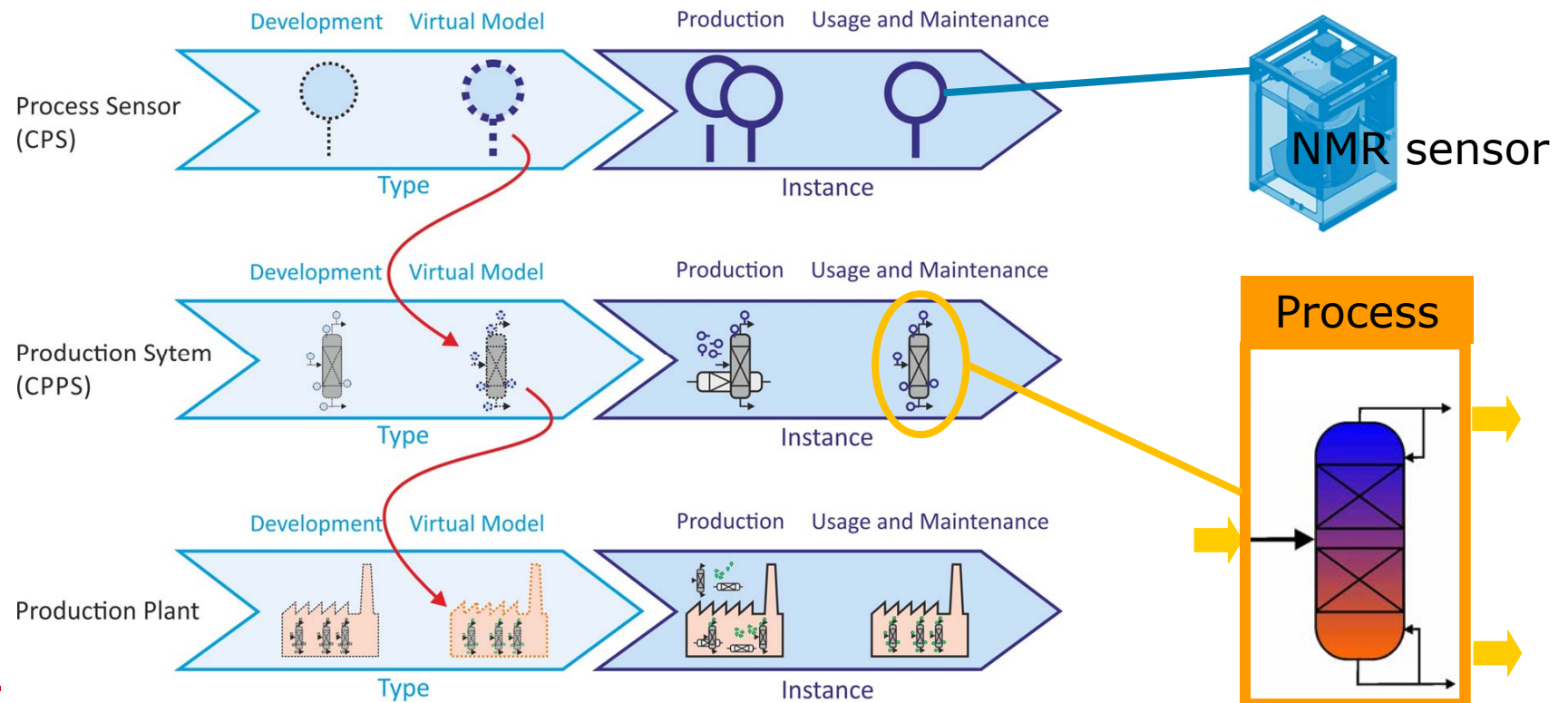
- Use of „complicated“ sensors has to be simplified towards „plug and play“

Continuous Engineering, Simulation

Towards Cyber-Physical Production Systems (CPPS)

“Cyber-Physical Systems (CPS) are integrations of computation with physical processes. Embedded computers and networks monitor and control the physical processes, usually with feedback loops where physical processes affect computations and vice versa”.

E. A. Lee, Cyber Physical Systems: Design Challenges, EECS Department, University of California, Berkeley, 2008



Maintenance and Internal Functions

Example: Revision Planning of Control Valves



Sensors in direct vicinity of valve

Revision schedule

Life time

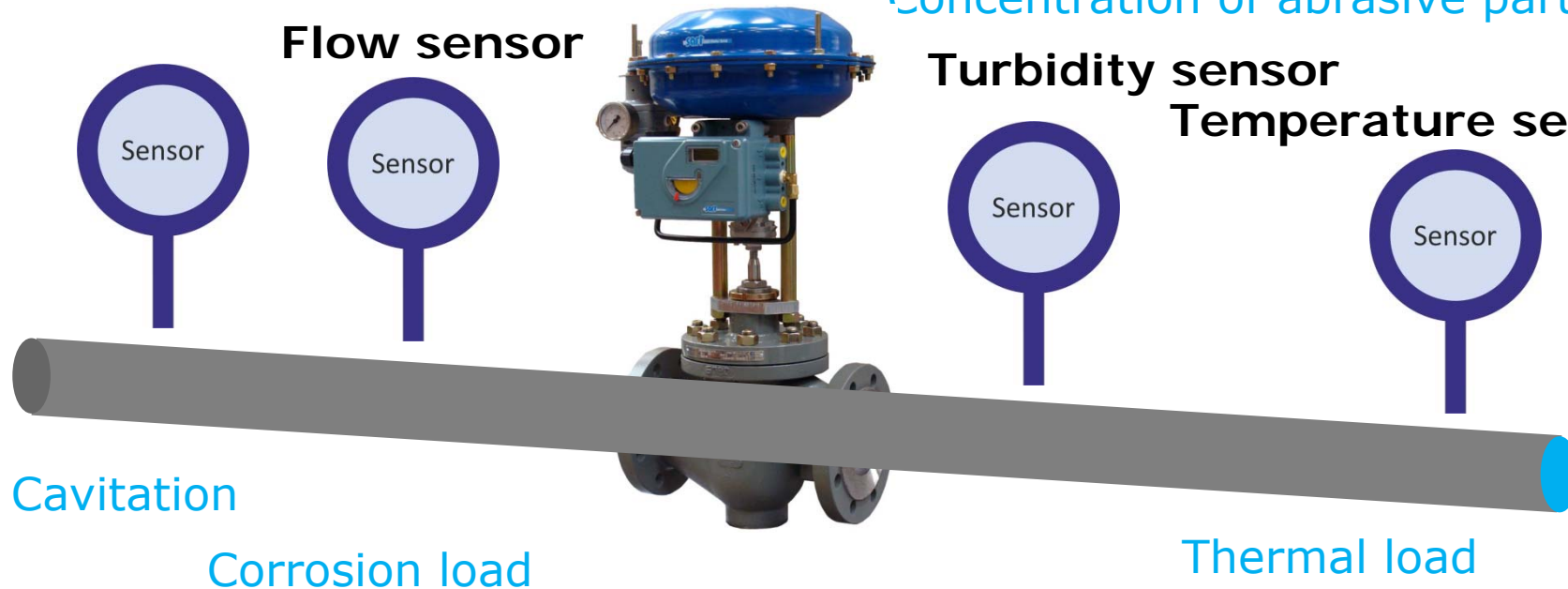
Pressure sensor

Flow sensor

Concentration of abrasive particles

Turbidity sensor

Temperature sensor

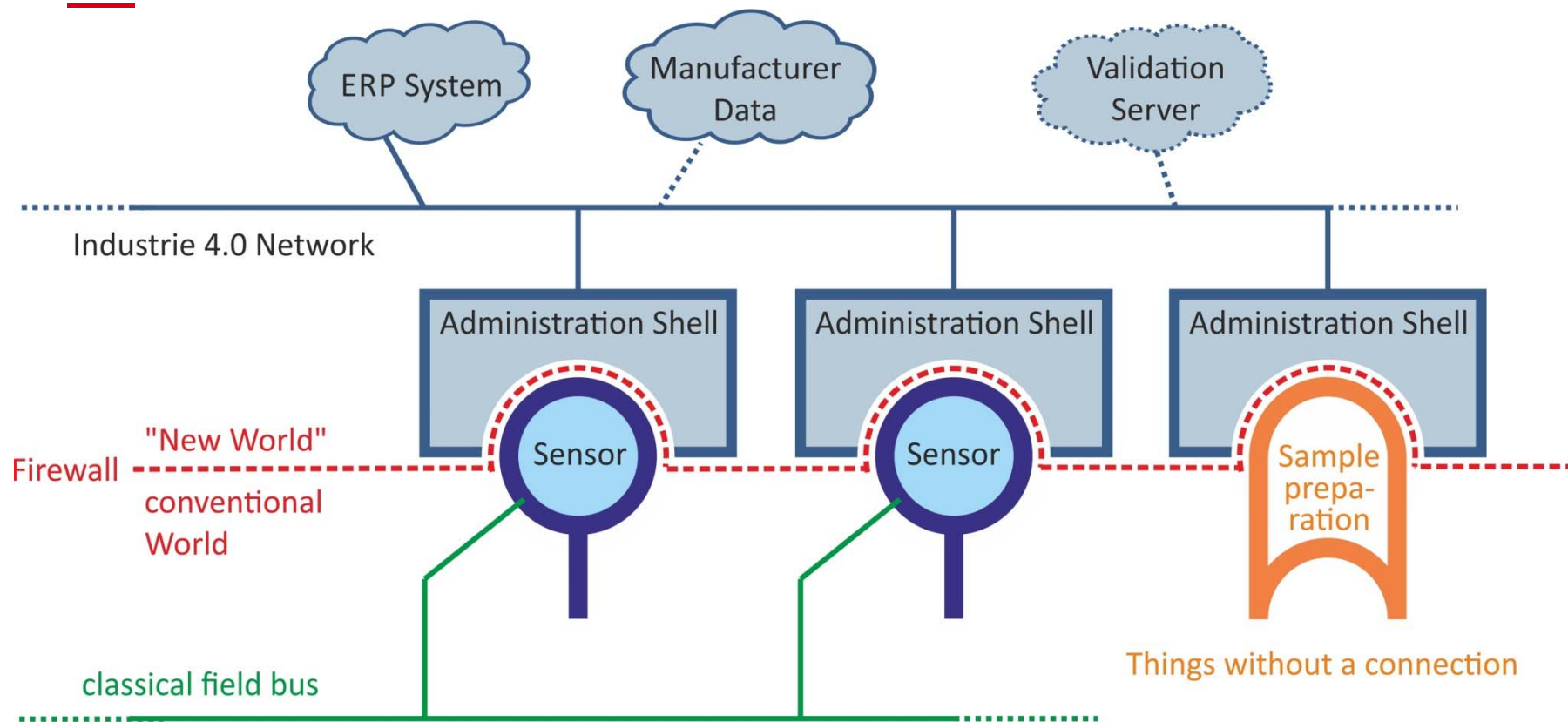


Maintenance actions are directly ordered by Enterprise Resource Planning (ERP) Software



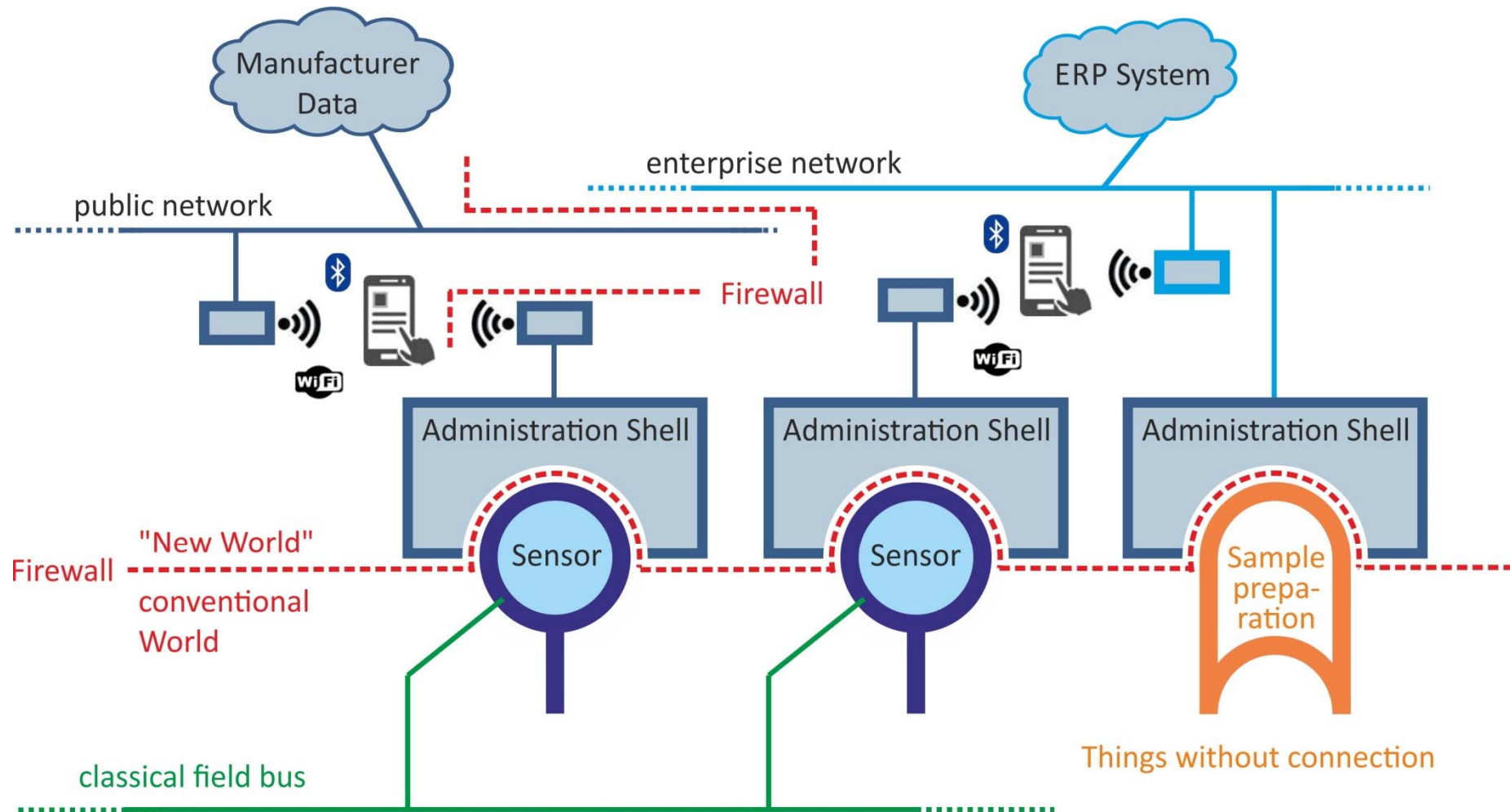
Perspectives

Sensor Image within Industrie 4.0 Network



Perspectives II

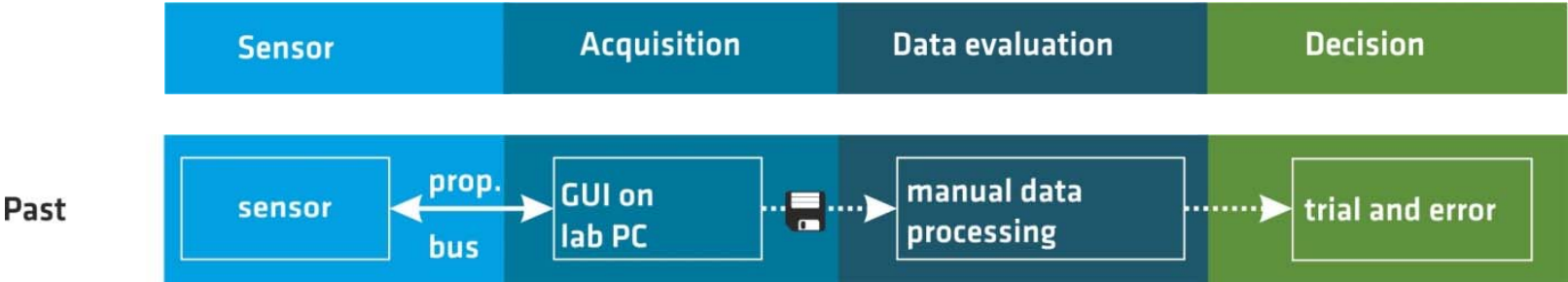
Simplified programming using mobile devices



Perspectives IV

From Lousy Interfaces to "Industrie 4.0"

Use of Runtime Environments

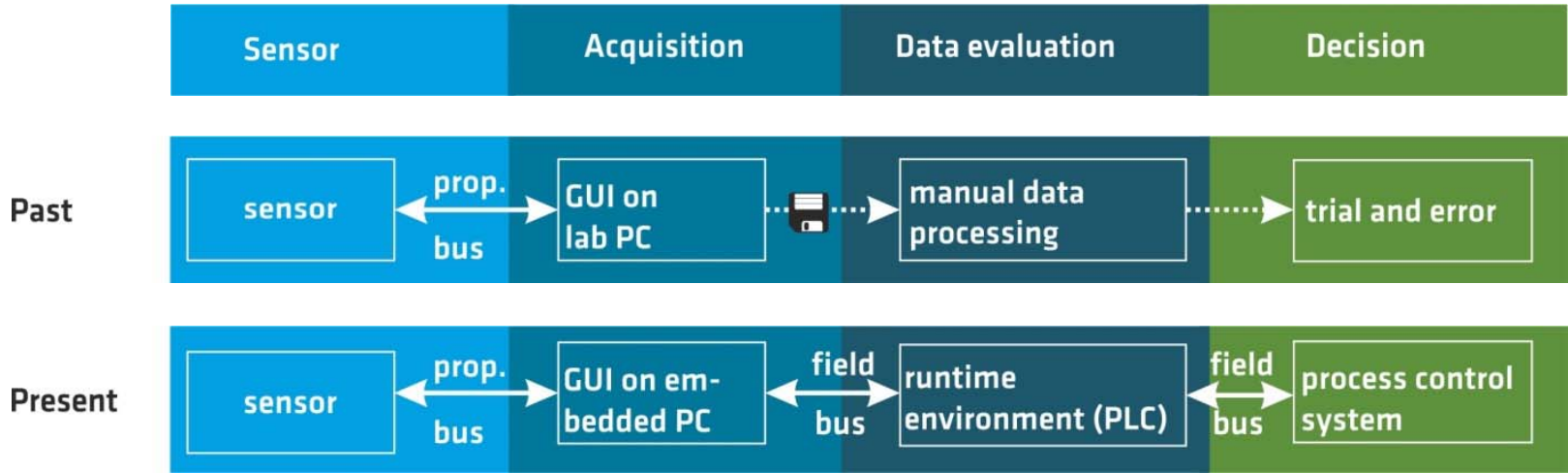


Perspectives IV



From Lousy Interfaces to "Industrie 4.0"

Use of Runtime Environments

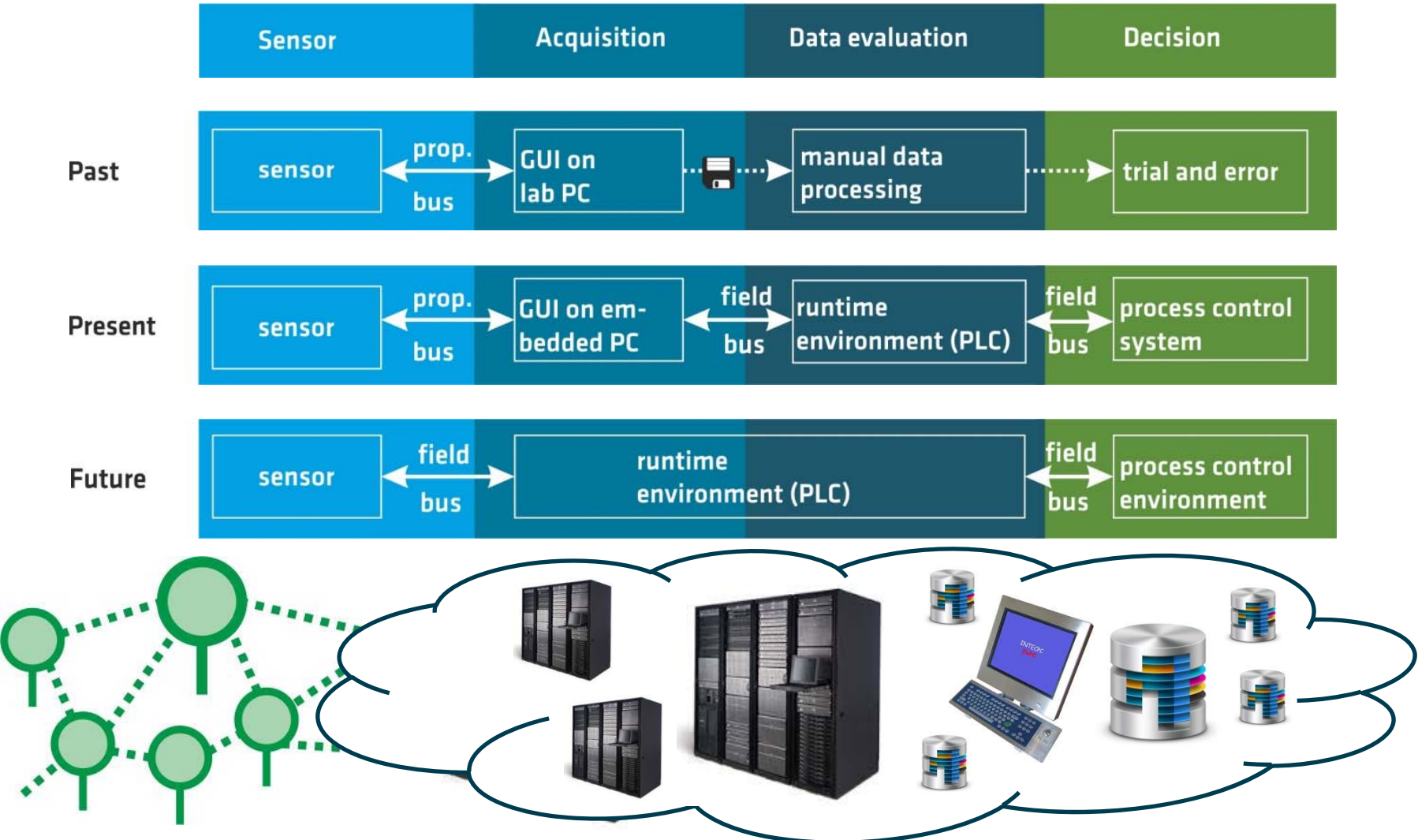


Perspectives IV



From Lousy Interfaces to "Industrie 4.0"

Use of Runtime Environments



Example

CONSENS: Smart Online NMR Sensor
for Advanced Process Control

Process Analytical Technology for Intensified Processes

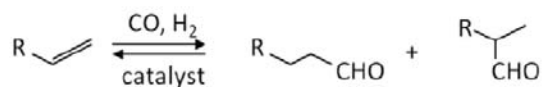


Examples from BAM, Berlin

Trends. Anal. Chem.
83 (2015) 39–52



Catalysis:
Hydroformylations of C12-alkenes
in micro emulsions



Alkene n-Aldehyde iso-Aldehyde

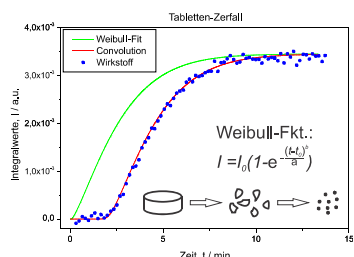
Meas. Sci. Technol. 28 (2017) 035501 + 035502



qNMR (Metrology):
Gases and
condensed phases
up to 1000 bar

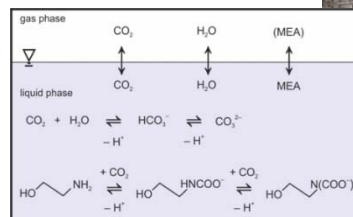
J. Magn. Reson.
275 (2017) 1–10

Pharmaceuticals:
Liberation kinetics



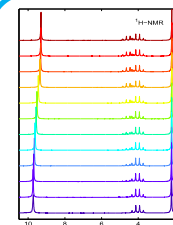
J. Mol. Structure. 1133 (2017) 18–23

CO₂ scrubbing

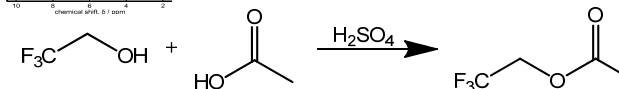


Klas Meyer, PhD
Humboldt University, Berlin, 2016

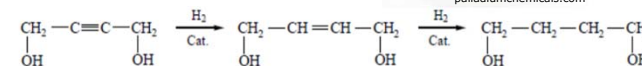
Method development:
Esterifications
(model reactions)



J. Magn. Reson. 249 (2014), 53–62
Magn. Reson. Chem. 54 (2016) 513–520

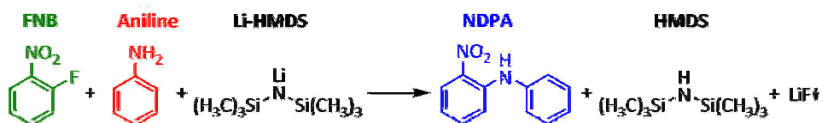
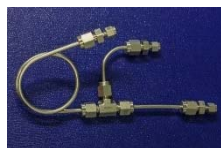


Catalysis:
Hydrogenations



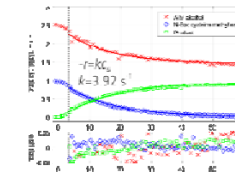
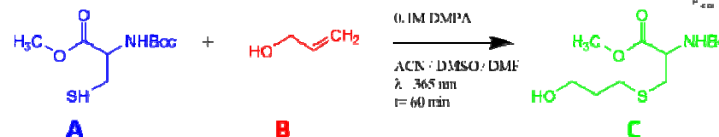
palladiumchemicals.com

Reaction Engineering:
Lithiation reactions



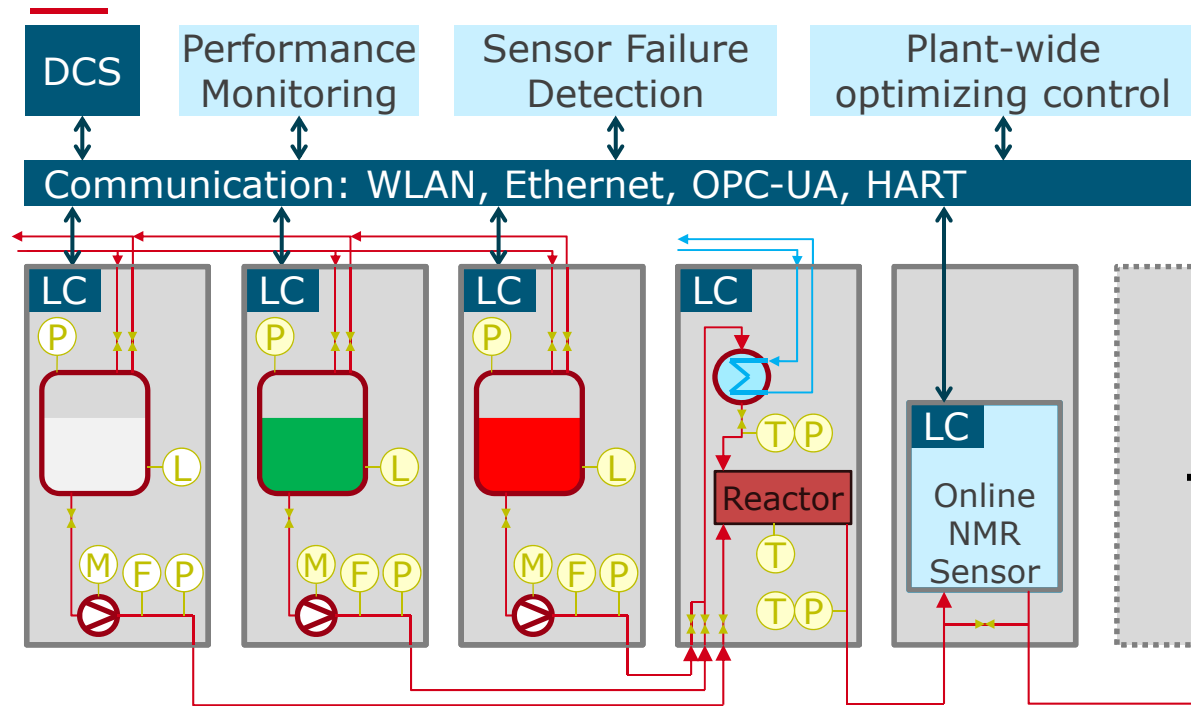
Reaction Engineering:
Photo reactions („click chemistry“)

J. Magn. Reson. 277 (2017) 154–161



Continuous Lithiation Reaction Set-up

Flexible Intensified Continuous Plant

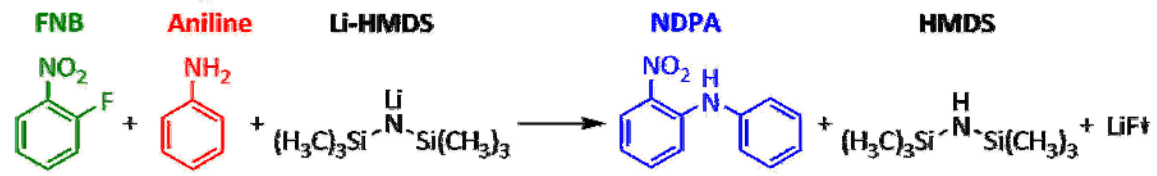


Containerized modular plant
Source: INVITE GmbH

- Standardized plant modules
- "Plug & Produce"
- Miniaturized flexible equipment
- Local control (LC)

- Fast and complex exothermic reaction
- Hazardous and inflammable substances
→ Explosion protection!
- Prone to fouling & clogging
- Multi-objective constrained optimization

Example: Lithiation reaction



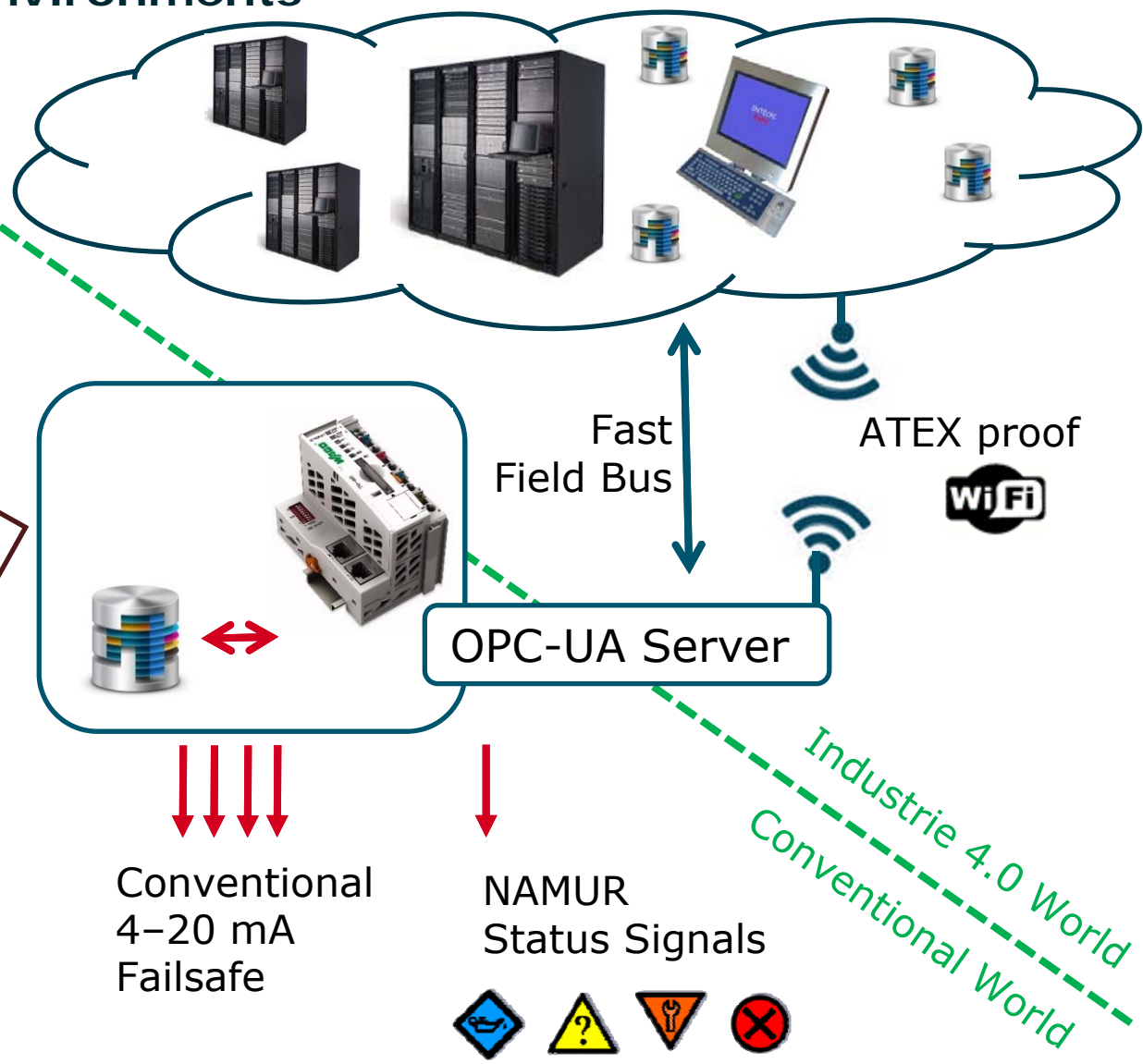
Conventional and Smart Functions



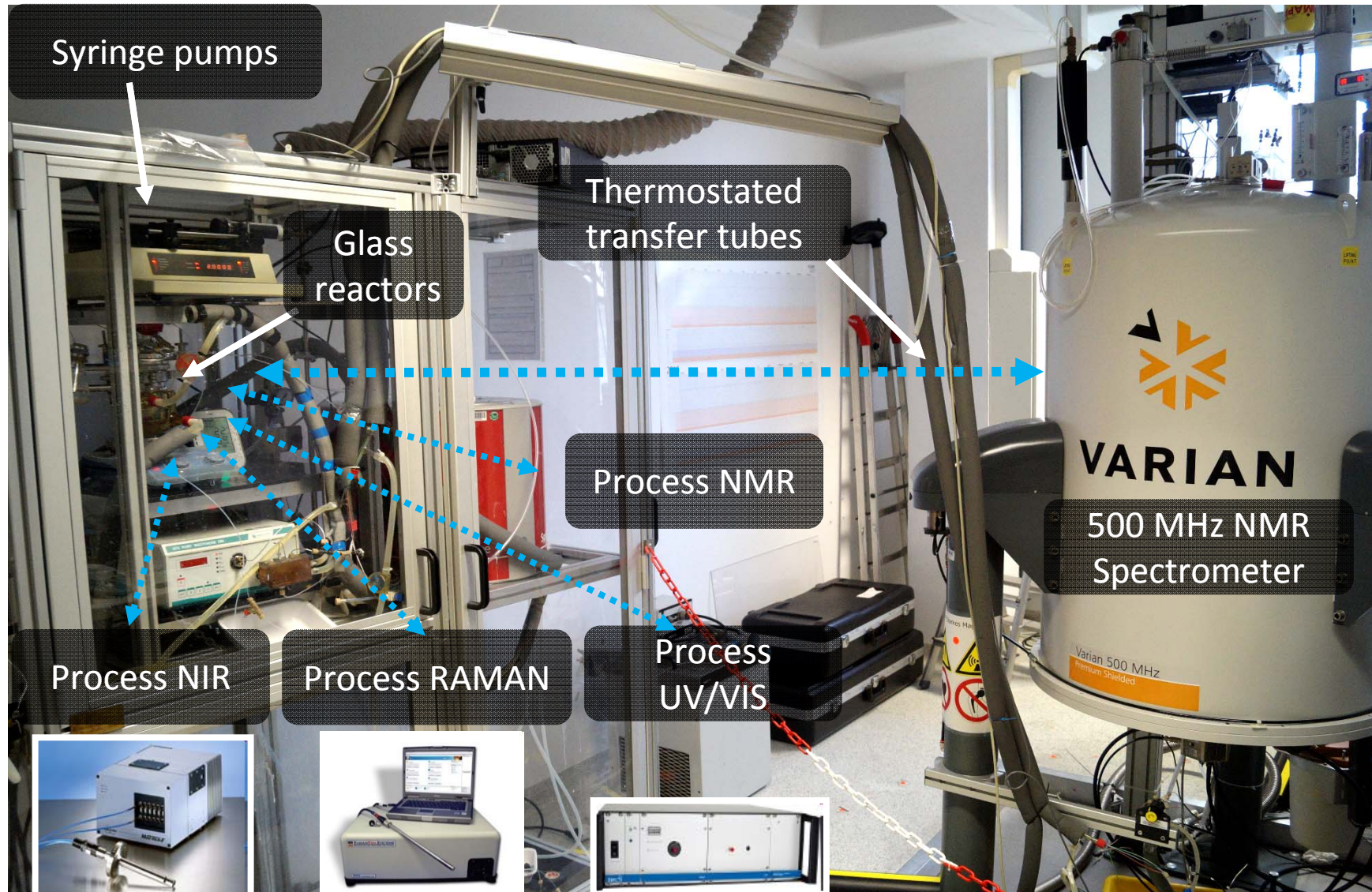
Enabling Runtime Environments



- Fail-safe Functions:**
- Leakage control
 - Temperature
 - Sample Pressure

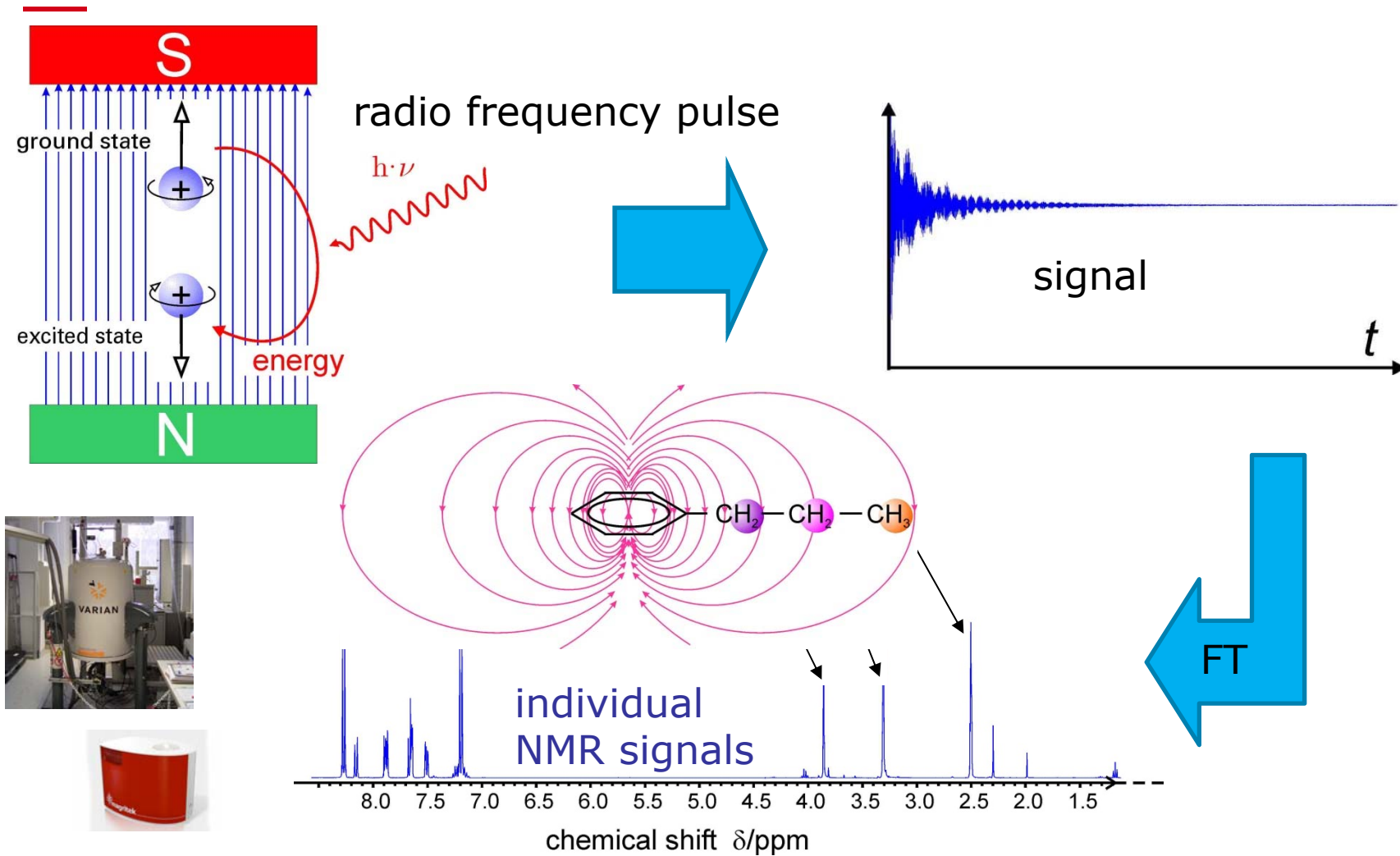


Reaction Monitoring in Lab-scale at BAM



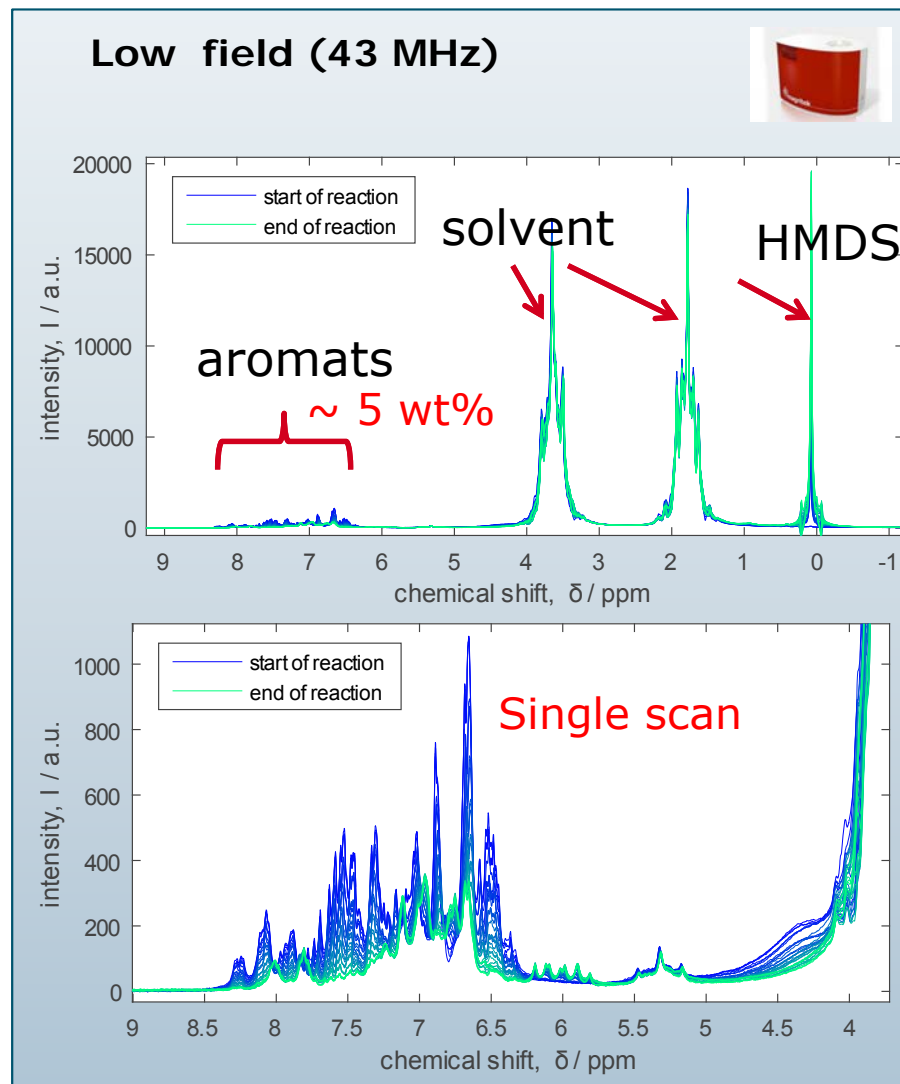
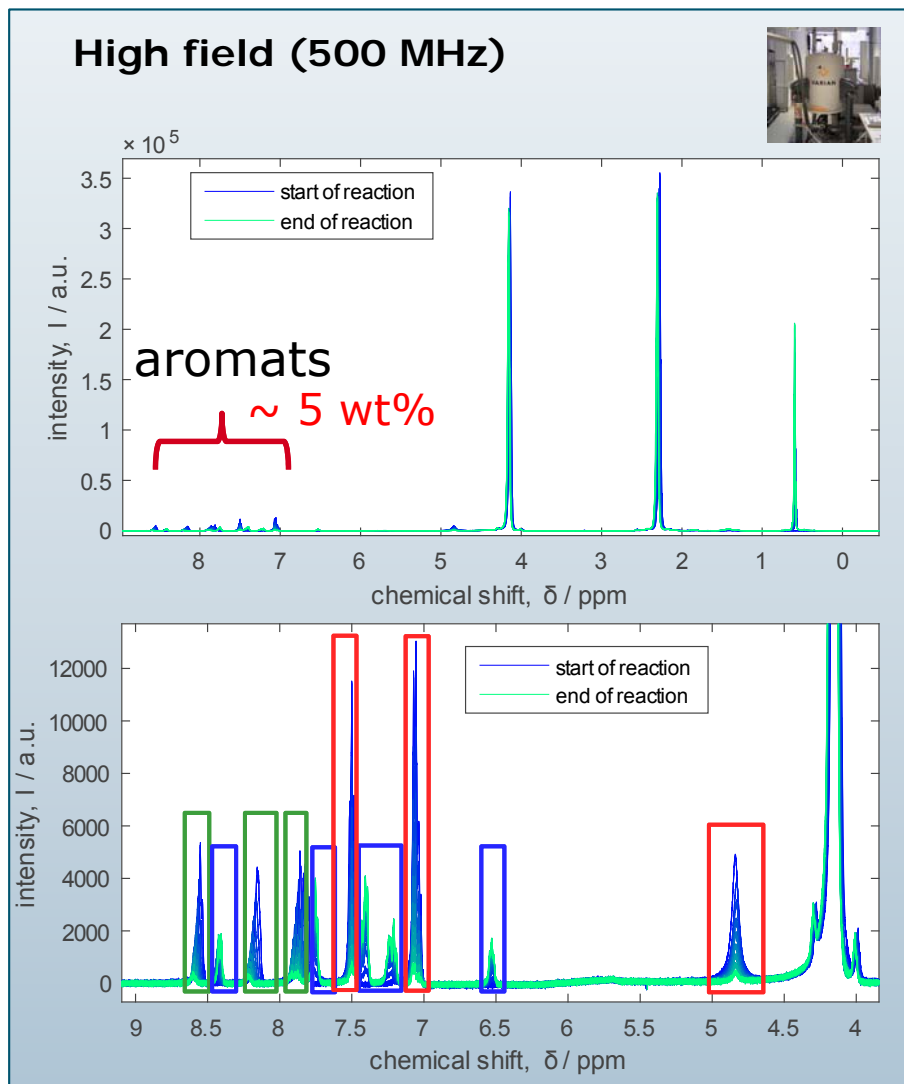
Nuclear Magnetic Resonance (NMR)

Direct Comparison Method → Calibration Free!

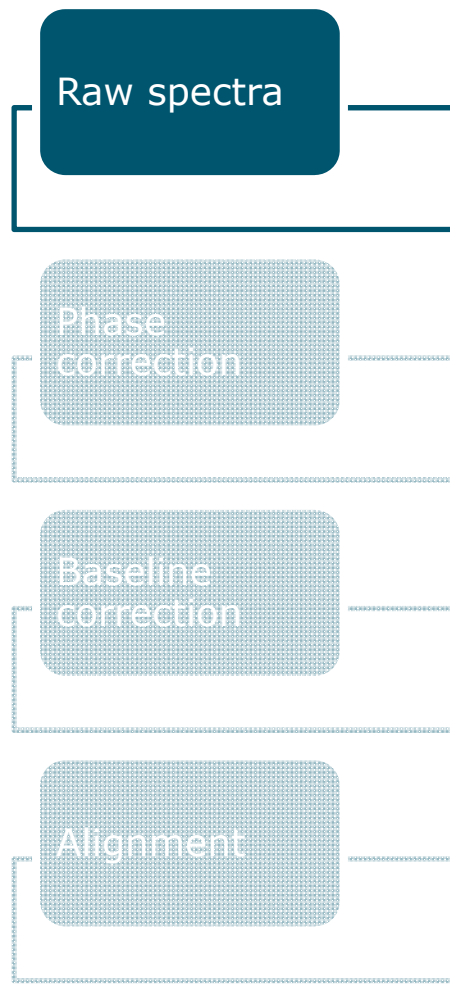


Proton NMR Spectra 500 MHz vs 43 MHz

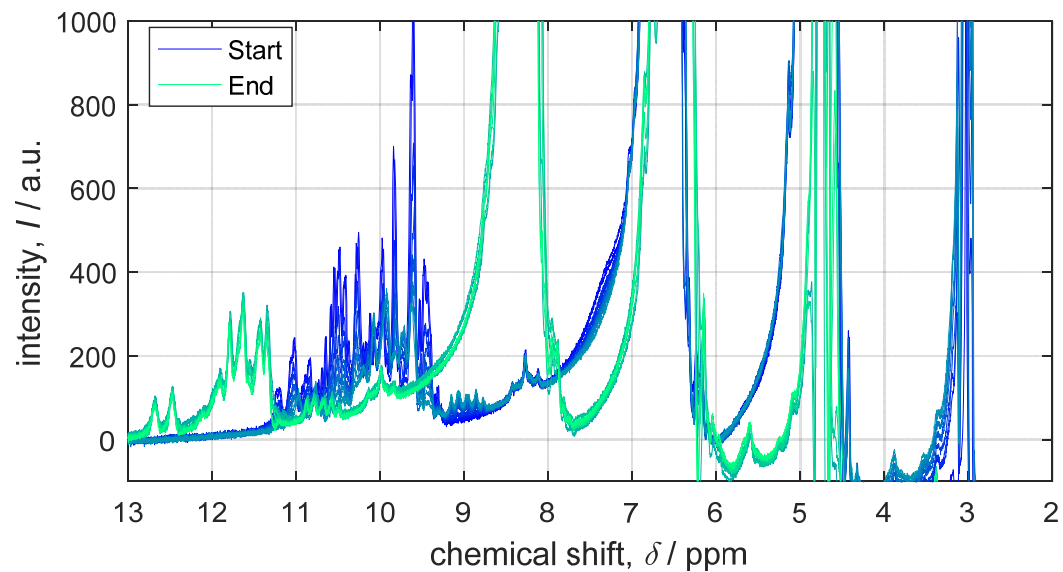
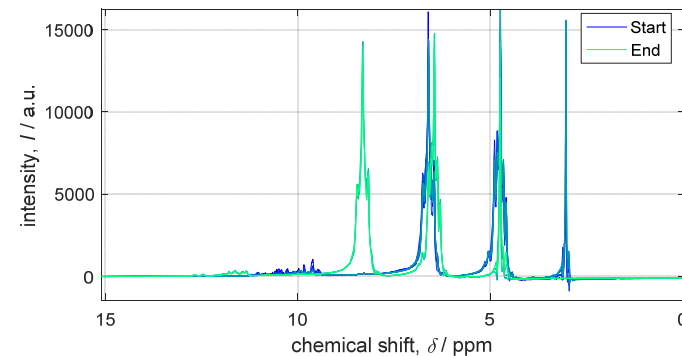
Lab Experiments in Batch Along Reaction Co-ordinate



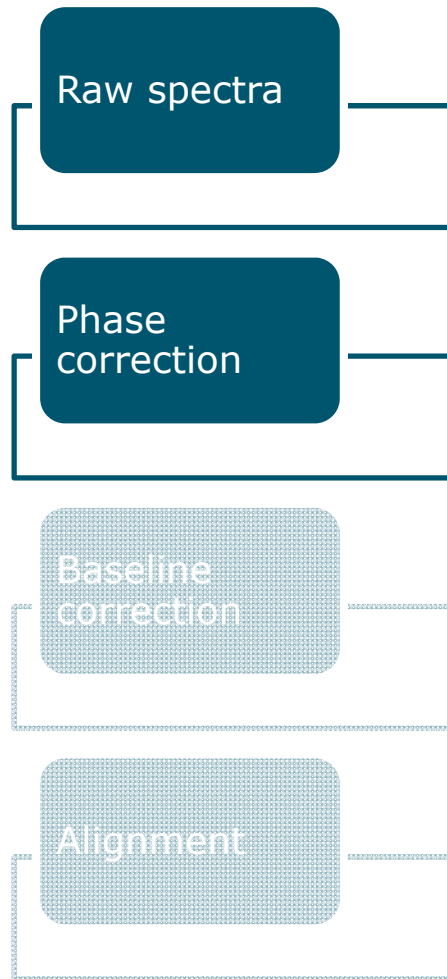
Data Processing for Low-field NMR in Matlab



single scan
each 15s
in flow

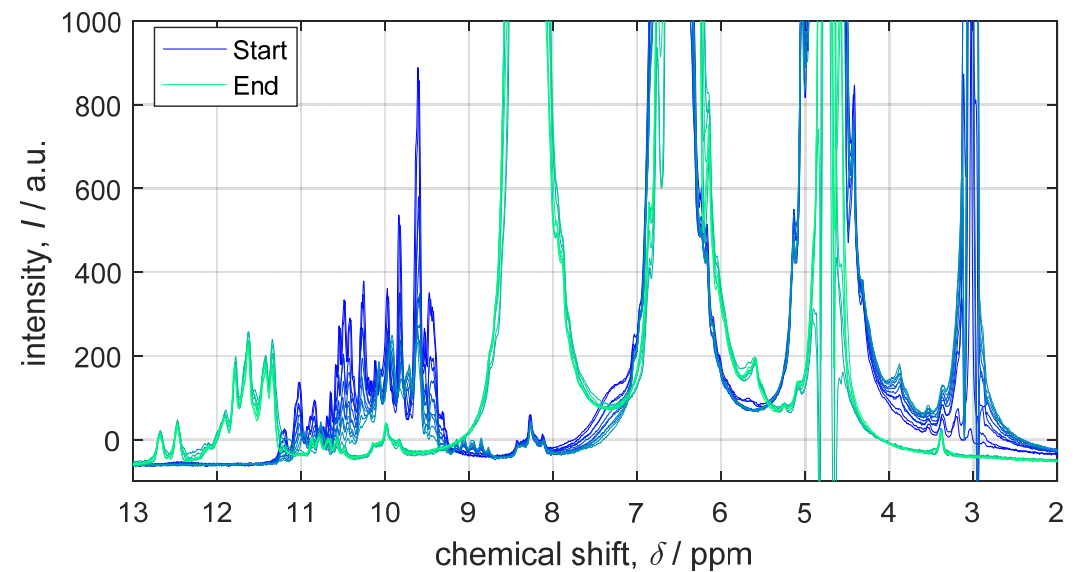
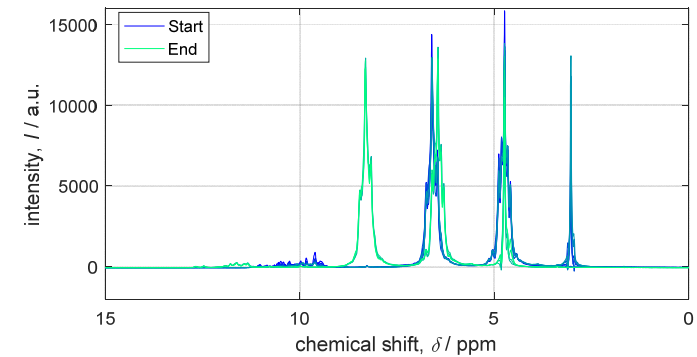


Data Processing for Low-field NMR in Matlab

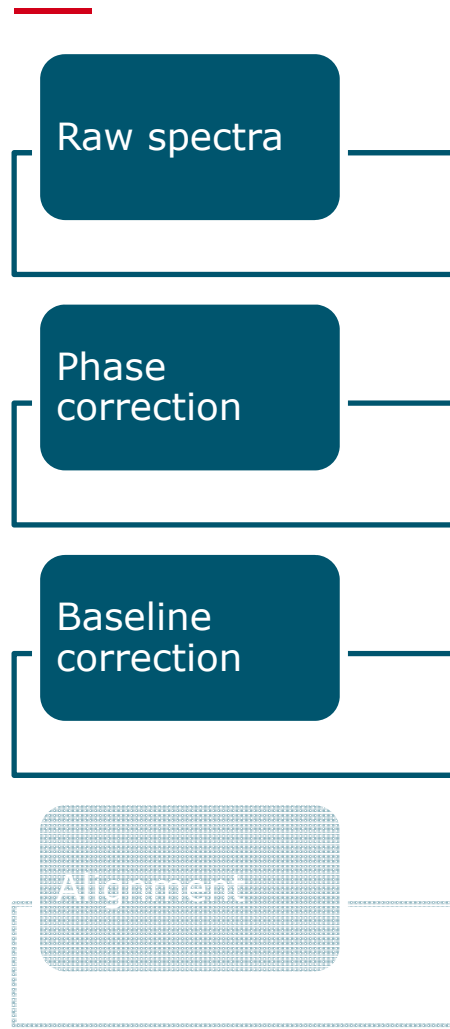


Entropy minimization method:

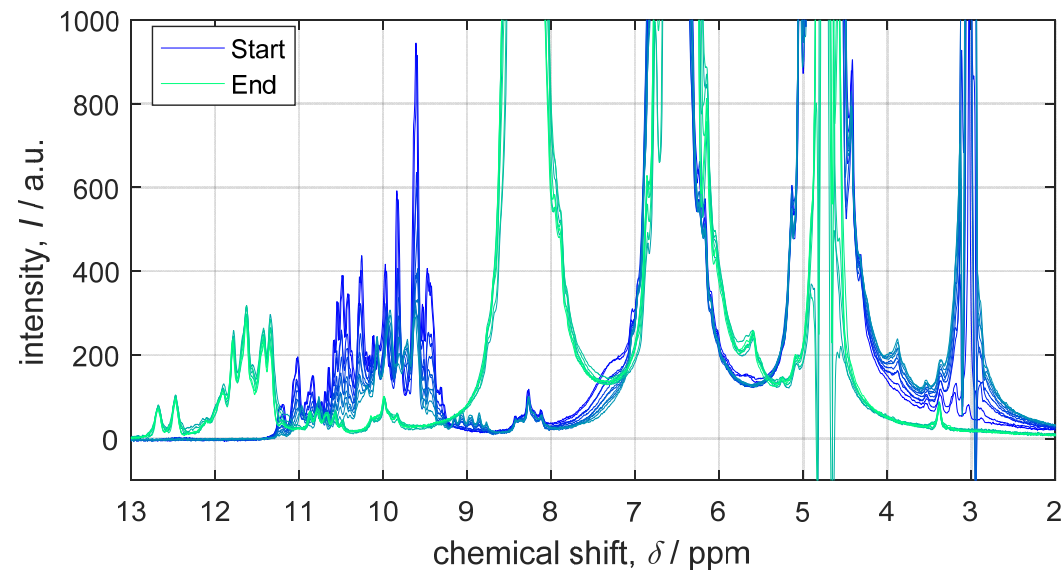
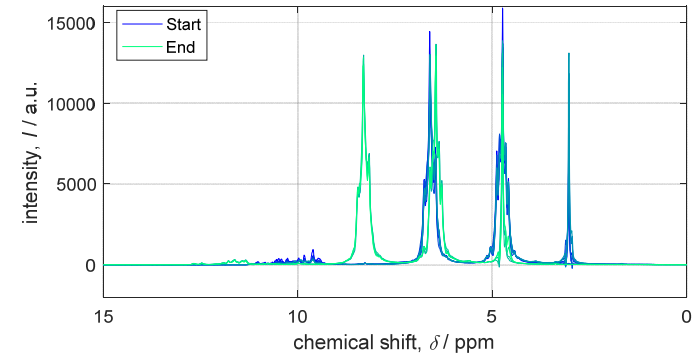
Chen et al., J Magn Reson. 2002, 158, 164-168



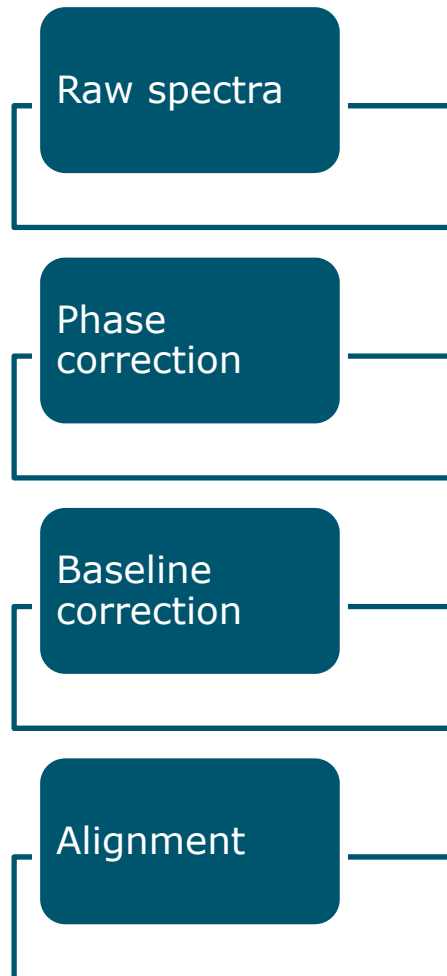
Data Processing for Low-field NMR in Matlab



Low-order Polynomial fit:
Mazet et al., Chemometrics
Intell. Lab. Sys. **2005**, *76*,
121-133.

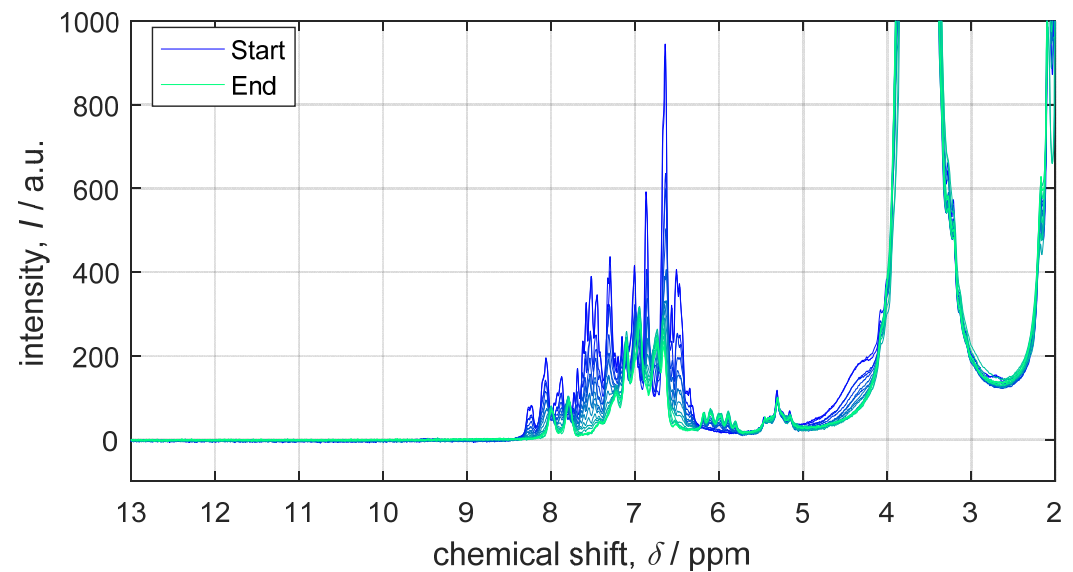
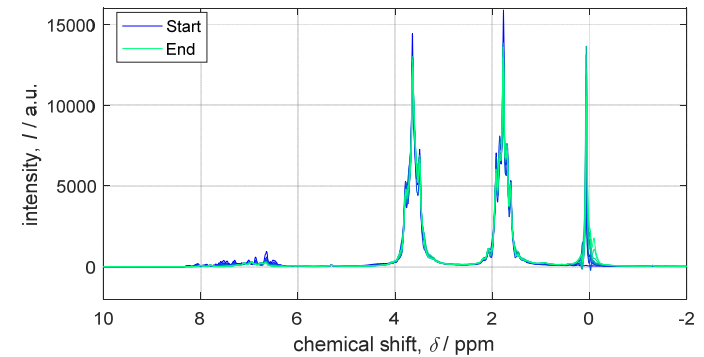


Data Processing for Low-field NMR in Matlab



Alignment to reference signal (THF) using *icoshift*:

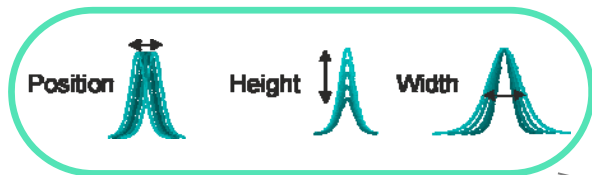
Savorani et al., J Magn Reson. 2010, 2, 190-202



Indirect Hard Modeling (IHM)

- Modeling → peak fitting of pure component spectra → mixture model
- Analysis → “component fitting” → calculation of component area

- Group of peaks represent pure component spectra

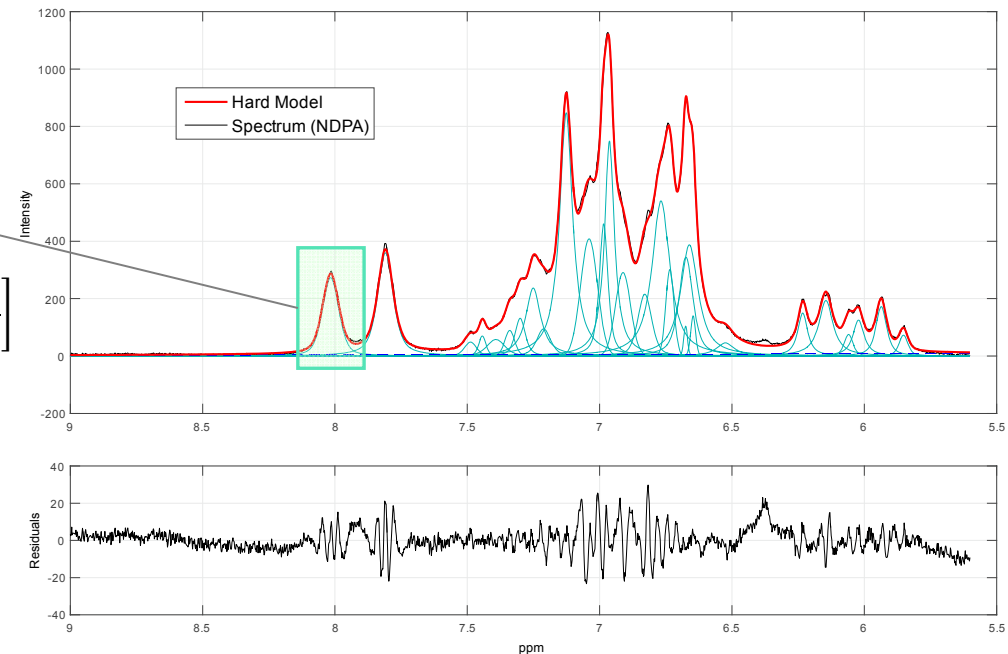


- Pseudo-Voigt function

$$V = \alpha \left[\beta \cdot \exp\left(-\ln(2) \frac{(x - \delta)^2}{\gamma^2}\right) + (1 - \beta) \frac{\gamma^2}{(x - \delta)^2 + \gamma^2} \right]$$

- α = peak maximum
- β = Gaussian-Lorentzian-ratio
- γ = half width
- δ = center position

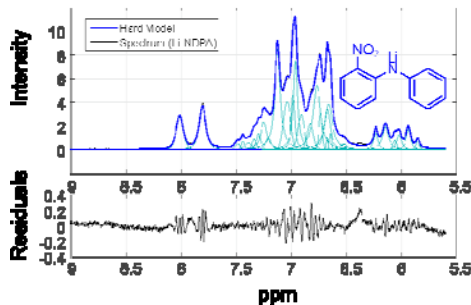
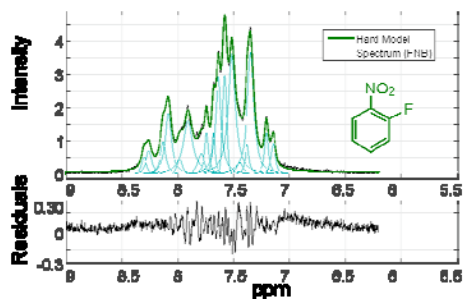
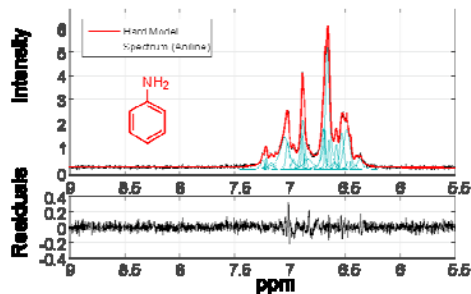
Hard model for pure components



IHM – „Model Building“

Assignment of NMR Signals to Pure Components

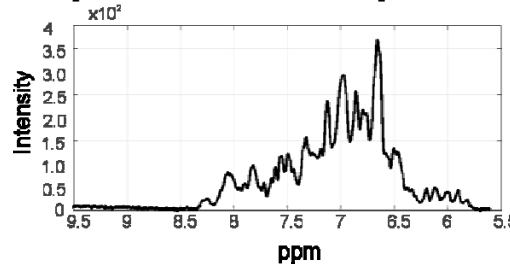
Pure component models



Mixture model

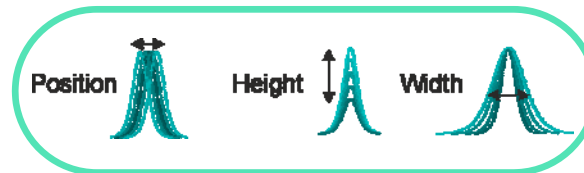
Component fitting

Experimental Spectrum



Modeling → Mixture model

No. components	3
Peaks per component	22, 21, 28
Degree of freedom (total free parameters)	292
Considered interacting peaks	25
Max. peak shift	0.01 ppm
...	...

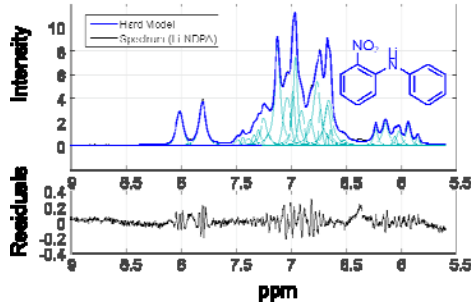
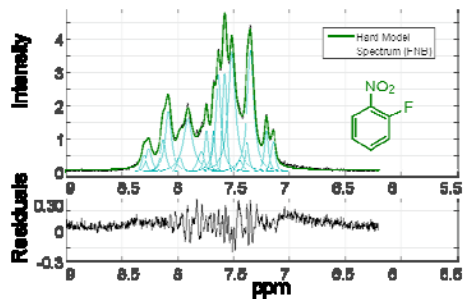
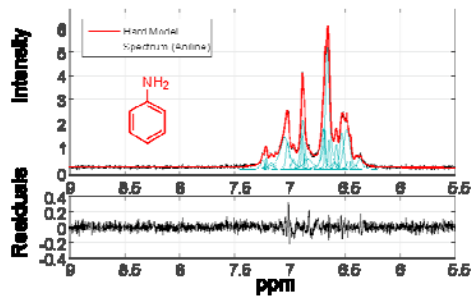


Peak area ratio for each pure component model is kept constant!

IHM – „Component Fitting“

Interpretation of Experimental Data with Pure Components

Pure component models

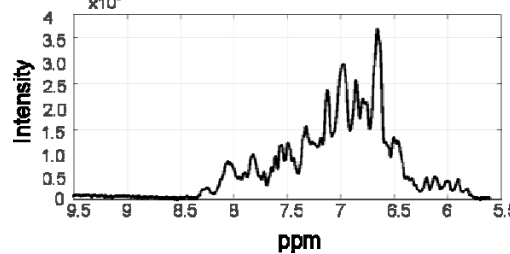


Analysis "component fitting"

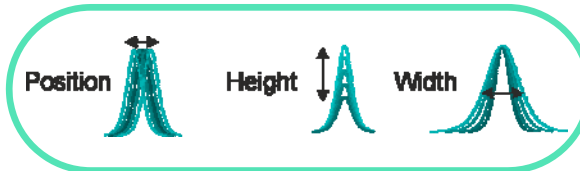
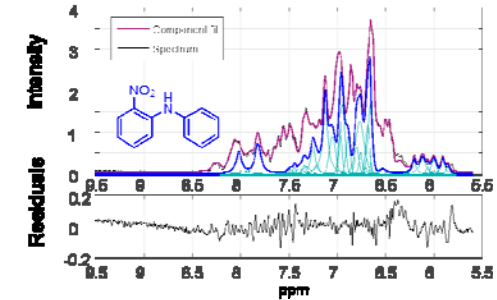
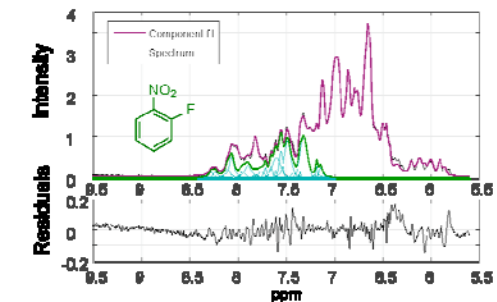
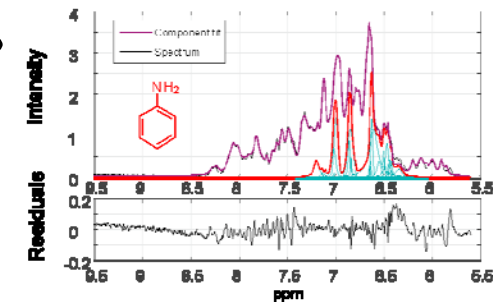
Mixture model

Component fitting

Experimental spectrum



Deconvoluted spectrum

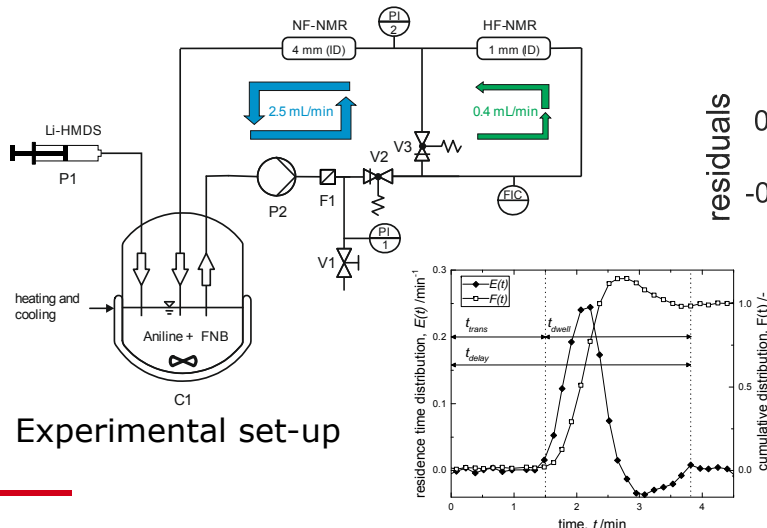
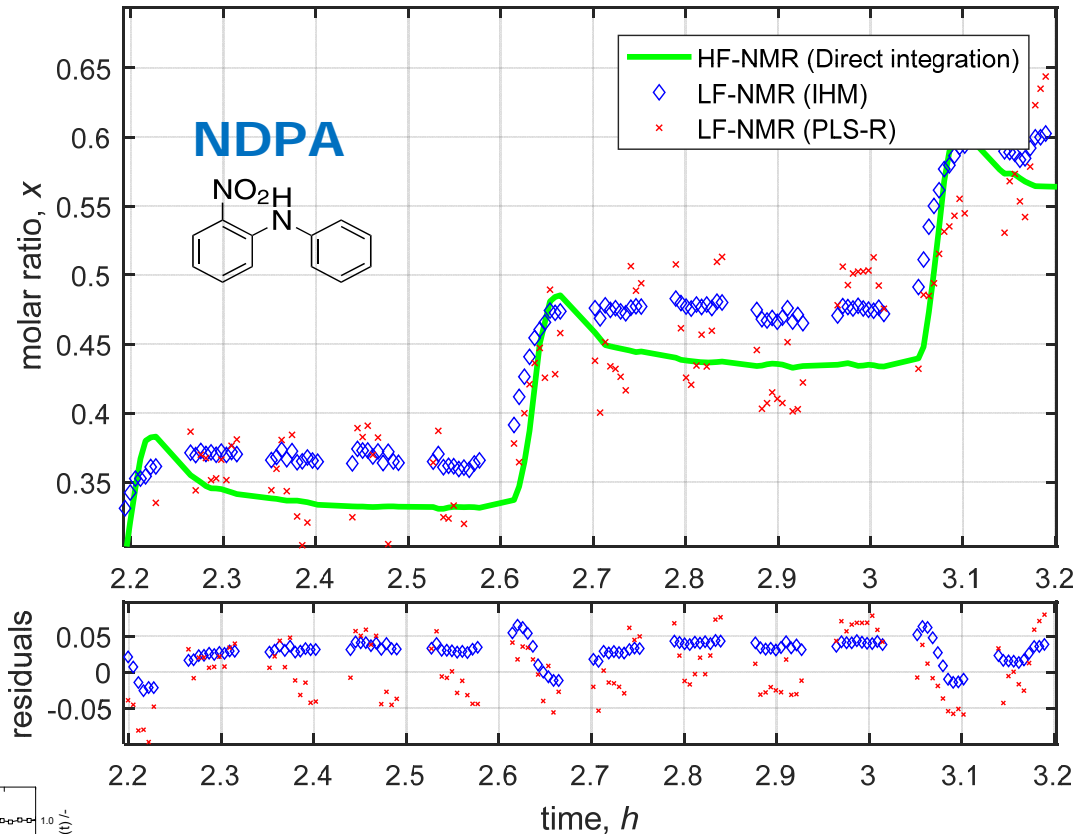
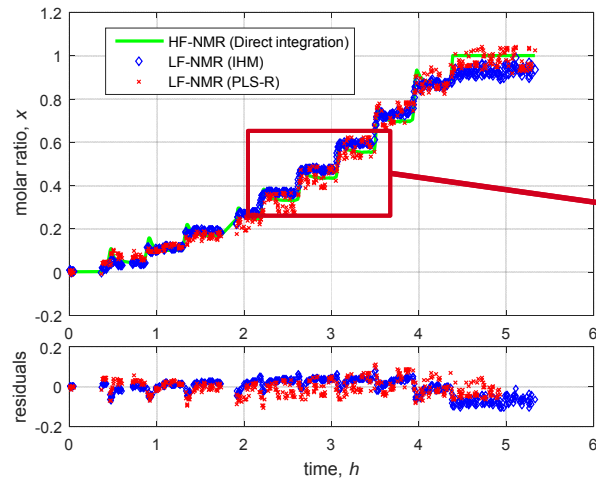


Model Building: IHM and PLS-R

Lab Experiments in Batch Along Reaction Co-ordinate

Validation with HF-NMR

IHM = Indirect Hard Modeling; PLS-R = Partial Least Squares Regression



Experimental set-up

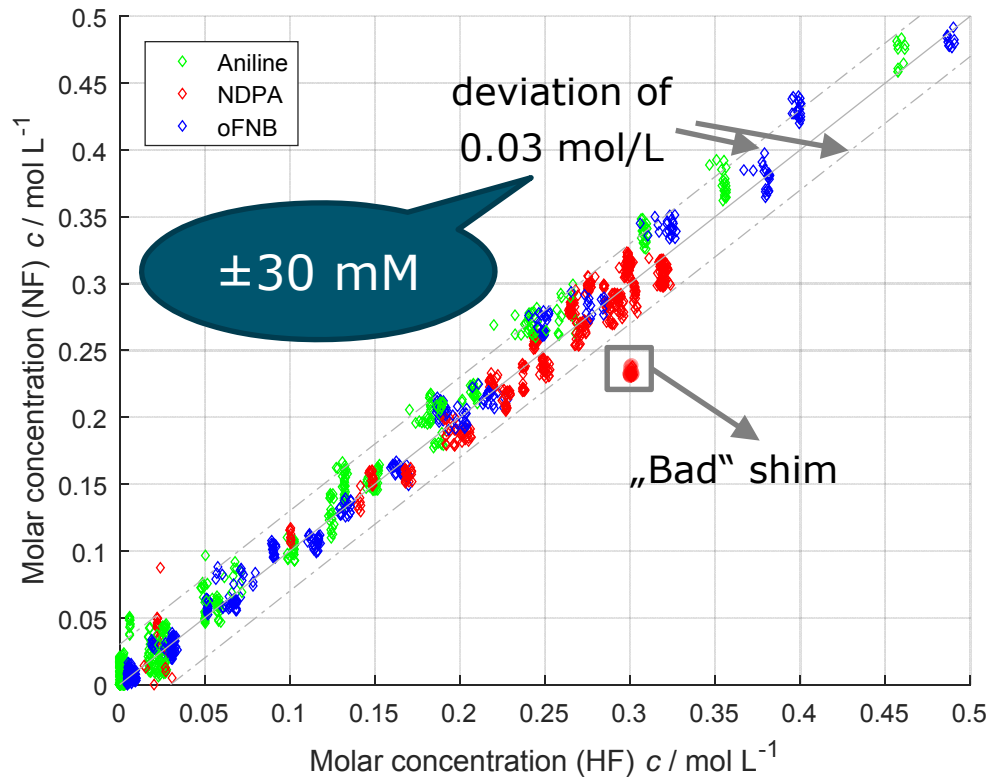
RTD of high field NMR
(Back-mixing)

Results: Reaction Characterization

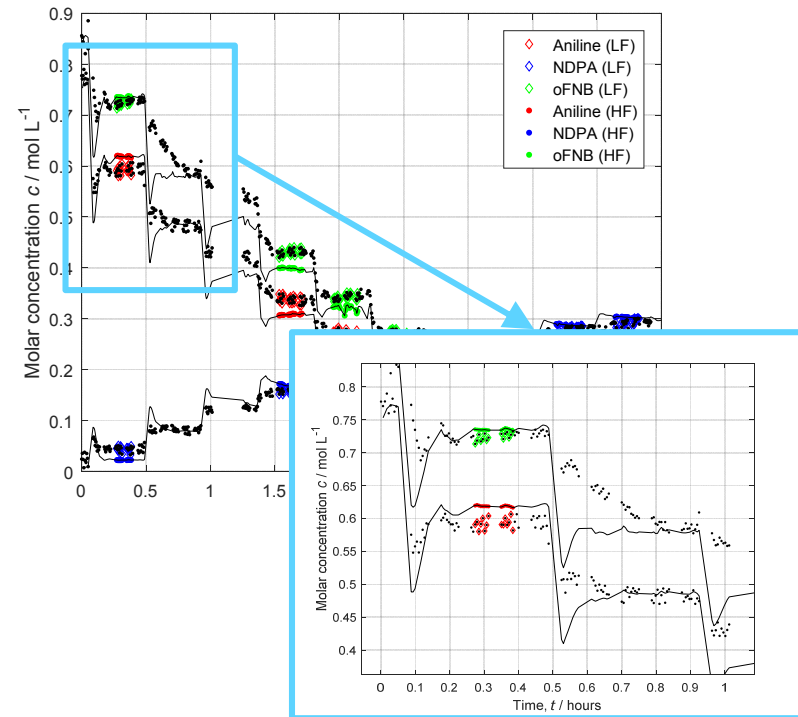
Lab Experiments in Batch along Reaction Co-ordinate

3 semi-batch reaction with varying starting concentrations

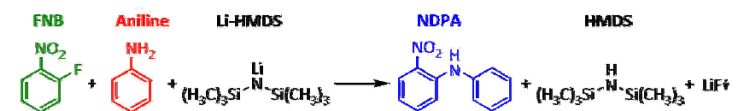
1500 Spectra for each HF- and NF-NMR



Classification of steady states

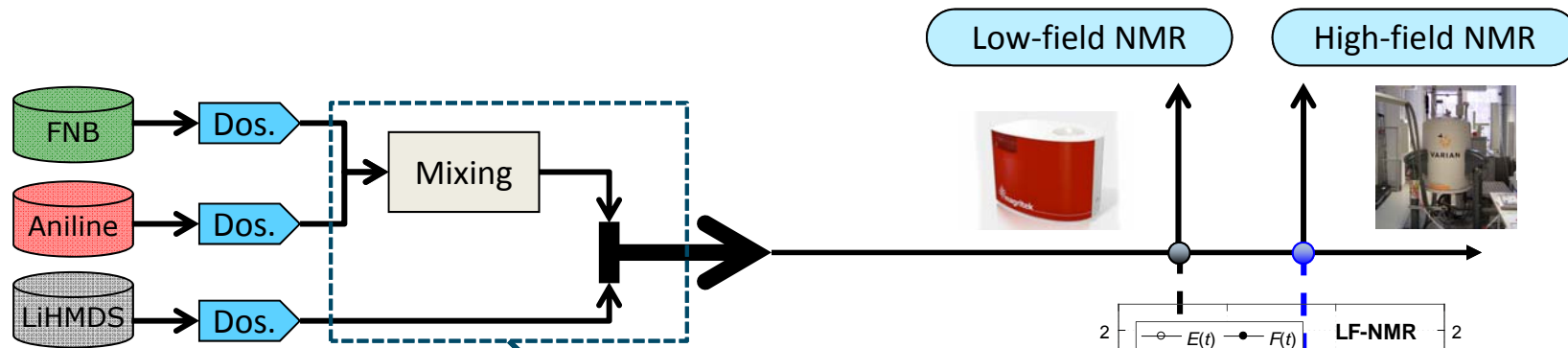


Reaction Scheme:

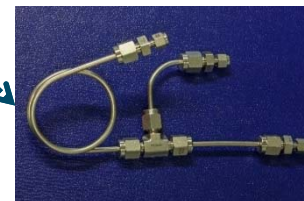


Validation of Lab Experiments in Tube Reactor

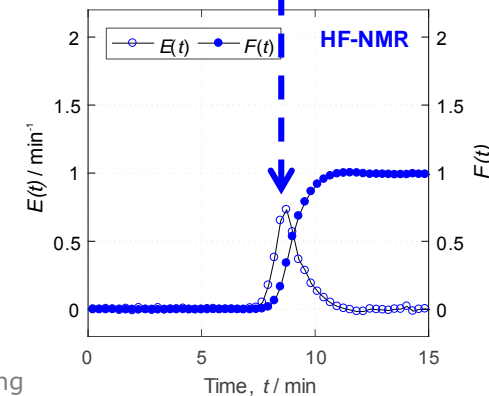
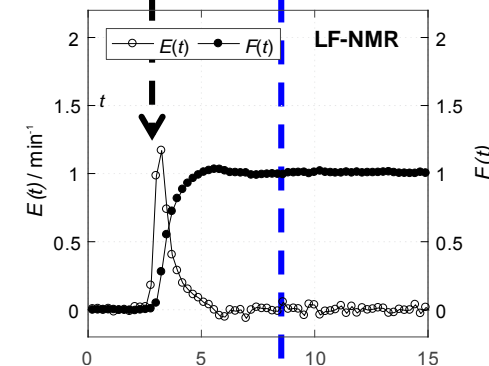
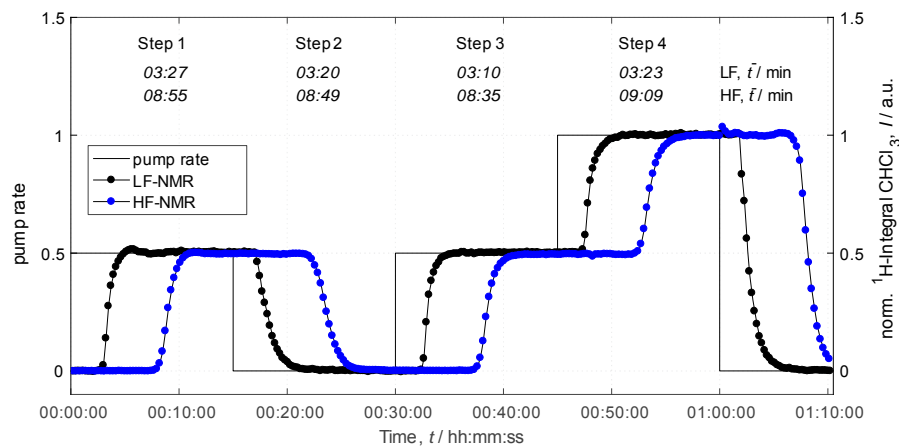
Continuous Lithiation Reaction Set-up at BAM



1/8" tube reactor



Step tracer experiments

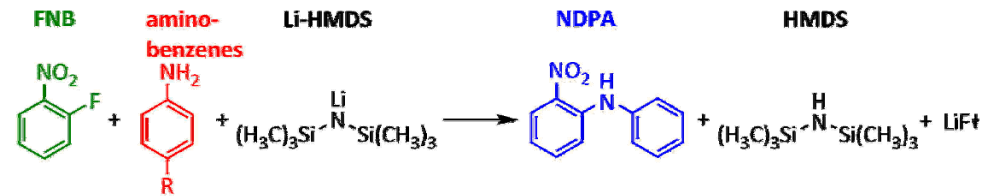


Experimental Design

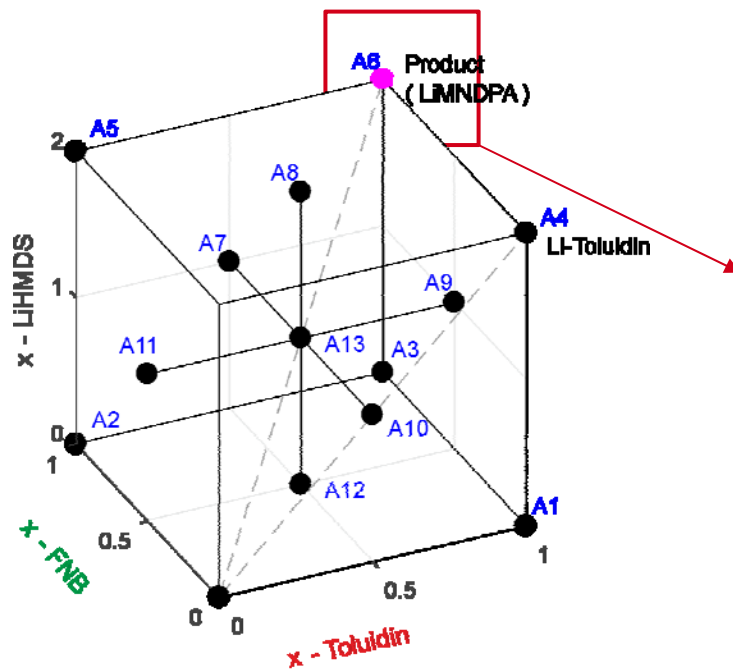
Continuous Lithiation Reaction Set-up at BAM

- Independent variation of flowrates / stoichiometric conditions

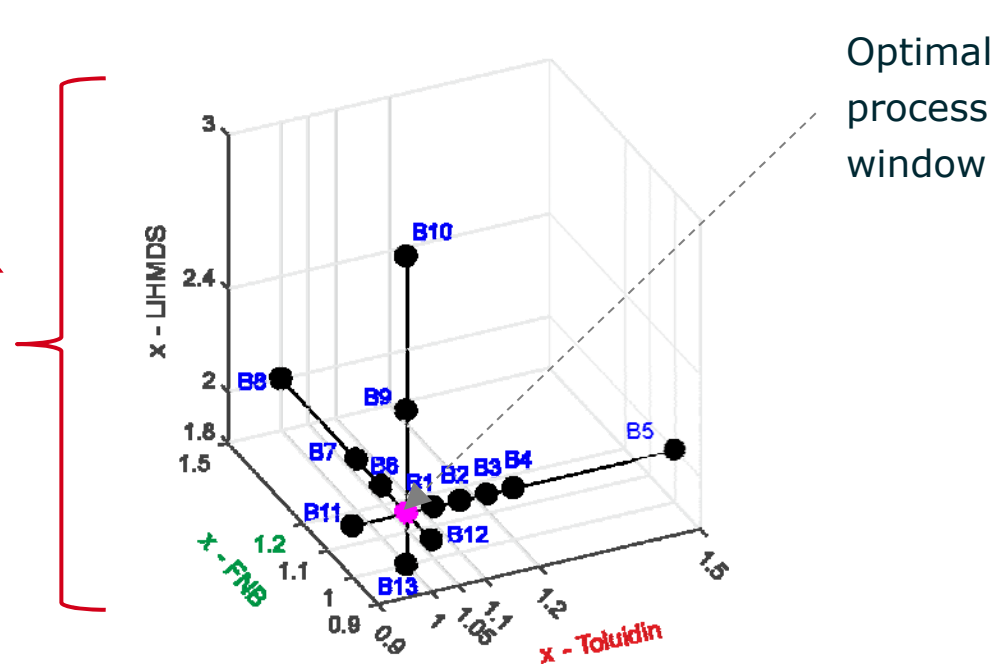
Reaction Scheme:



Coarse variation:



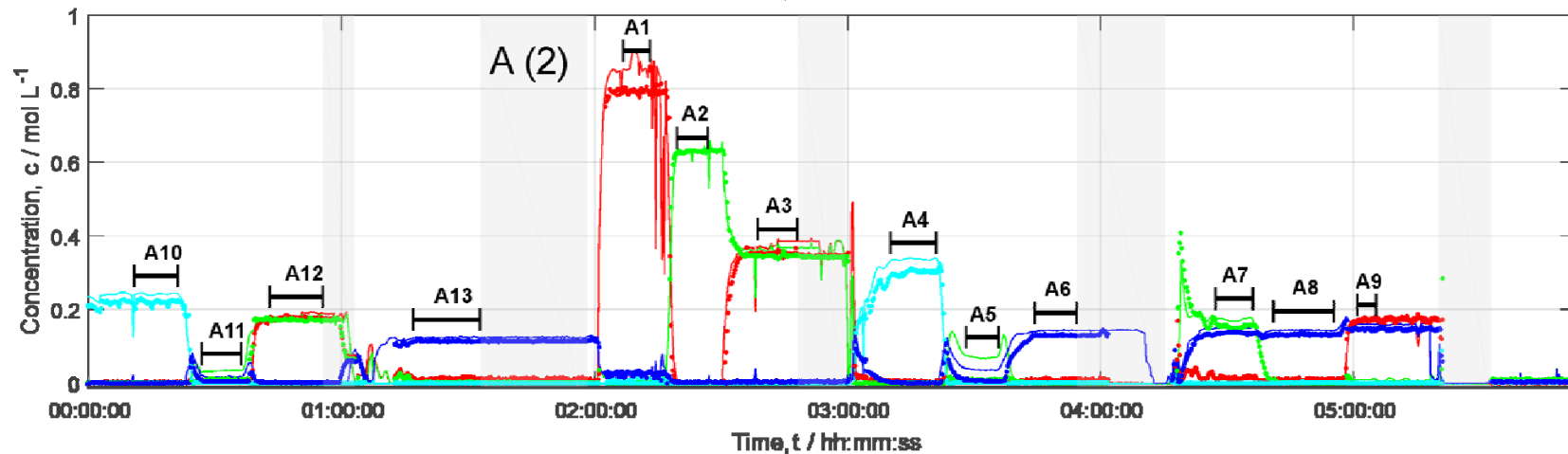
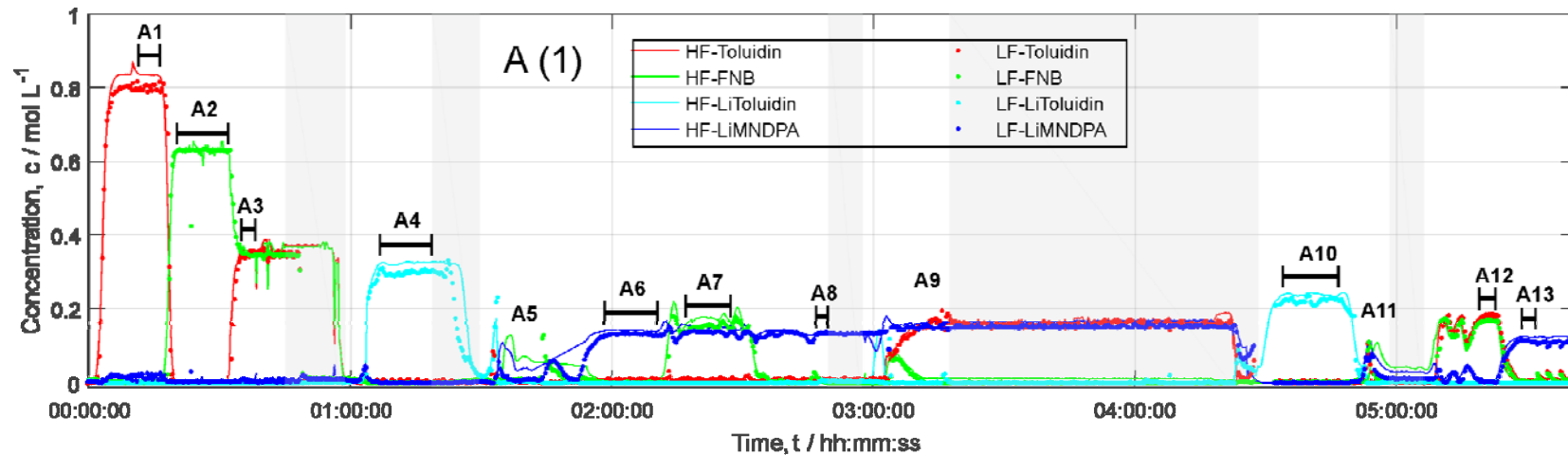
Fine variation:



Validation Instead of Calibration

Variation of Set-Points according to Experimental Design

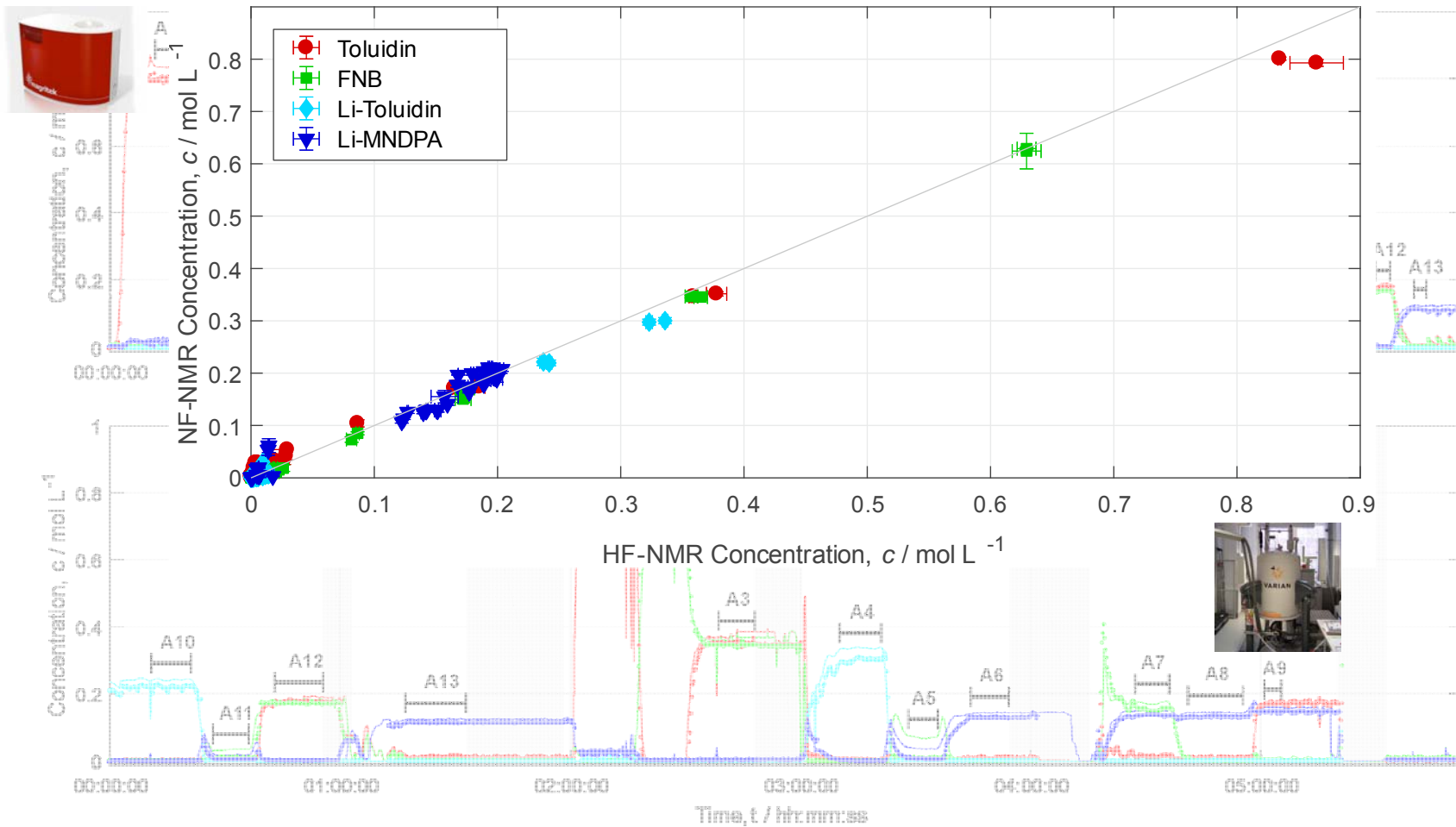
Low-Field versus High-Field NMR Spectroscopy



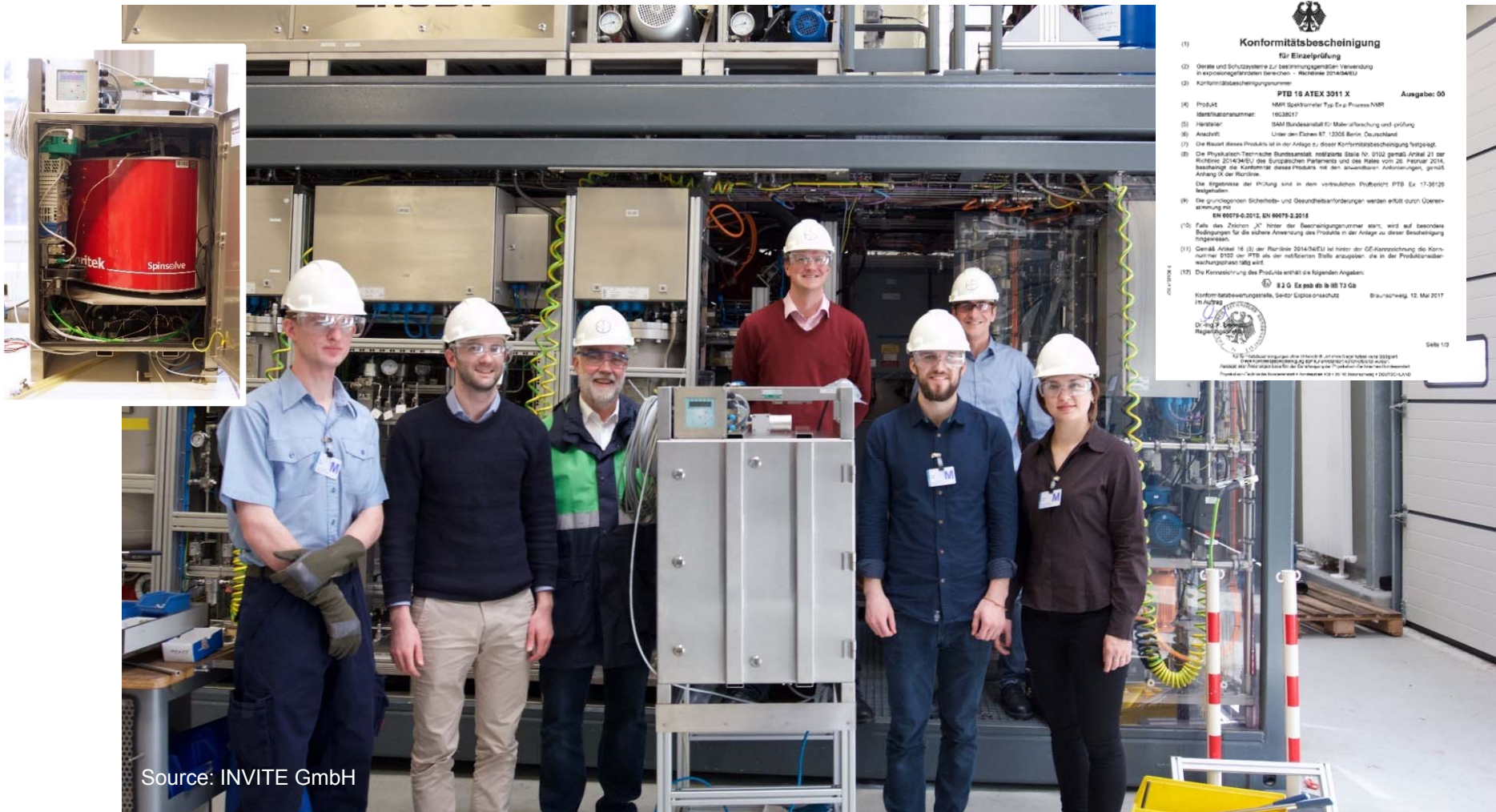
Validation Instead of Calibration

Variation of Set-Points according to Experimental Design

Low-Field versus High-Field NMR Spectroscopy



Example: Smart Online NMR Sensor Validation at Bayer/INVITE in Leverkusen



Source: INVITE GmbH



VDE

VDI/VDE-Gesellschaft
Mess- und Automatisierungstechnik



Thank You!

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S-Pact

S-Pact

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13. Kolloquium

21. und 22. November 2017 · Esslingen
mit Opening-Session am 20.11.2017

Themenschwerpunkt

**Prozessführung in der modernen
Produktion**

Brückenschläge von der Fertigungstechnik
zur Industrie 4.0



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GESELLSCHAFT DEUTSCHER CHEMIKER



20.-22. November 2017
Esslingen bei Stuttgart

Themenschwerpunkt

**„Prozessführung in der
modernen Produktion –
Brückenschläge von der
Fertigungstechnik
zur Industrie 4.0“**

<http://arbeitskreis-prozessanalytik.de>