

Design and Validation of a Compact Online NMR Module

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Motivation

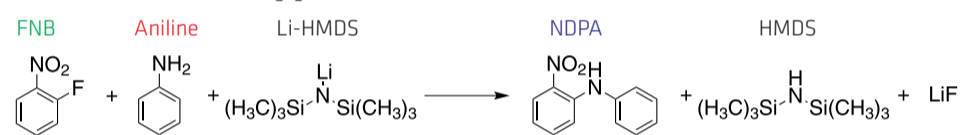
- New "plug and produce" concept
 - Modular and standardized chemical production elements
 - Novel online sensors
 - Closed-loop control methods
- More efficient and flexible low to medium scale production for specialty chemicals.
- Specific challenges are
- Higher quality levels: more accurate sensors
 - Innovative products: fast design, engineering and optimization
 - short set-up times



Fig. 1: Containerized modular plant (source: INVITE GmbH)

Model Reaction and Specific Challenge

In CONSENS, two aromatic substances (Eq. 1) are coupled by a lithiation reaction [1]:



Eq. 1: Reaction scheme, FNB: 1-Fluoro-2-nitrobenzene, Li-HMDS: Lithium bis(trimethylsilyl)amide, NDPA: 2-Nitrodiphenylamine

Plug and Produce Concept for a Lithiation Reaction

The laboratory set-up at BAM exhibits same reactor residence times as plant using controlled syringe pumps and NMR analyzers. [3, 4]

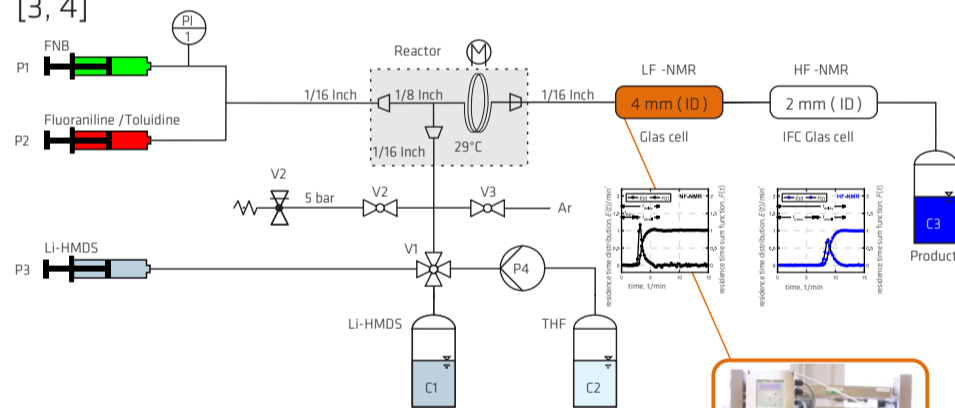


Fig. 2 (top): Concept and flow scheme of the lithiation reaction in continuous mode at BAM as a scale-down of the real modular plant

Fig. 3 (right): Field demonstrator featuring ATEX housing for spectrometer and electronics, embedded PC for automated data preparation and analysis, programmable logic controller (PLC) connected to plant process control system (PLS)



Current and Future Requirements to Process Sensors [2, 3]

- Use of „complicated“ sensors has to be simplified towards „plug and play“, i.e., they exhibit short set-up times.
- 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.

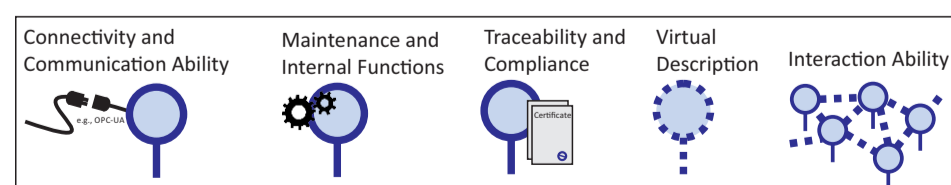


Fig 4: Most important general requirements to smart process sensors

Concept of the Compact Online NMR Module

The developed NMR analyzer is provided in an explosion proof housing (Fig. 3, right) involves a compact 43.5 MHz NMR spectrometer together with an acquisition unit and a programmable logic controller for fully automated data preparation (phasing, baseline correction) and evaluation [3, 4]. A spectral modeling procedure based on Indirect Hard Modeling (IHM) was developed and validated for data analysis of the low-field NMR spectra. Results are shared with process control systems via an integrated OPC-UA server over intrinsically safe Ethernet cable or Wi-Fi connection.

Conventional Functions

- Failsafe functions, e.g., leakage and *T* control, sample pressure
- Conventional 4-20 mA failsafe signals
- NAMUR status signals

Smart Functions [2, 3]

- OPC-UA server
- Fast failsafe field bus
- ATEX proof WiFi
- Cloud computing
- Use of runtime environments

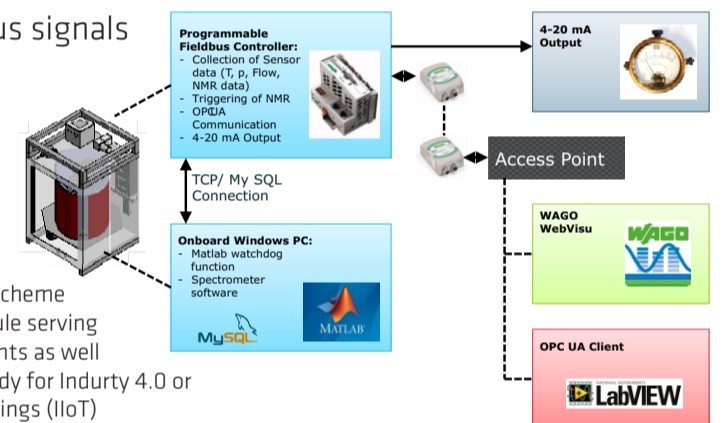


Fig. 5: Communication scheme of the smart NMR Module serving conventional requirements as well as Smart Functions, ready for Industry 4.0 or Industrial Internet of Things (IIoT)

Validation Runs According to Design of Experiments

A considerable number of continuous experiments were performed for validation according to a DoE (Fig. 6b) taking account for various reaction conditions by individually adjusting the flow rates of the reagents aniline, FNB, and Li-HMDS. Figure 6a depicts amount of substance fractions observed with the NMR sensor and the IHM methods over an observation period of six hours.

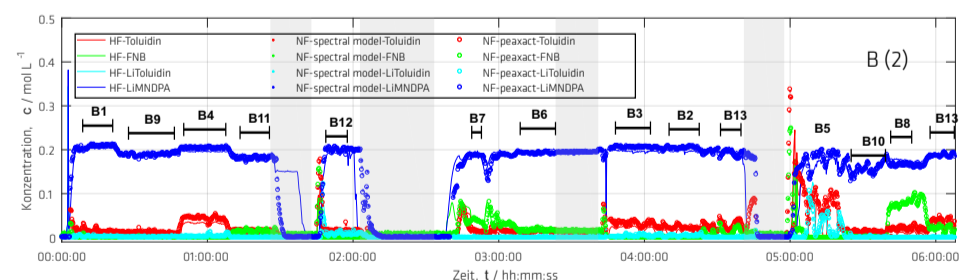


Fig. 6a (top): The largest prediction uncertainties in mole fractions were found for aniline to be 5-7 % relative, i.e., 0.25-0.35 % absolute.

Fig. 6a (right): Design of Experiments for validation run.

References and Acknowledgements

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