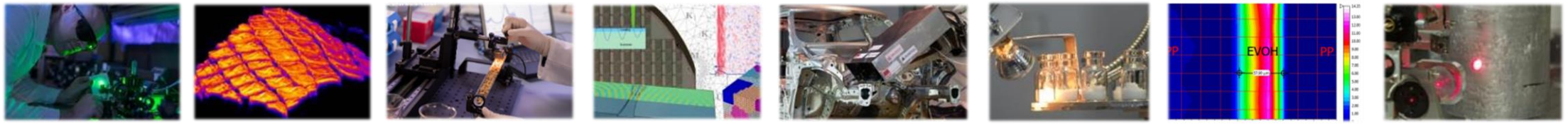
Infrared & Raman Spectroscopy for PAT applications

Infrared & Raman Spectroscopy Group

Paul Gattinger

RECENDT
RESEARCH CENTER NON DESTRUCTIVE TESTING



RECENDT - REsearch CEnter for Non-Destructive Testing

Science Park @ Johannes Kepler University
Linz, Austria

- Founded in 2009
- ~40 researchers
- Funded research
  (national, H2020, HEU, ITN, …)
- Contract research

upper austria

Member of UAR INNOVATION NETWORK

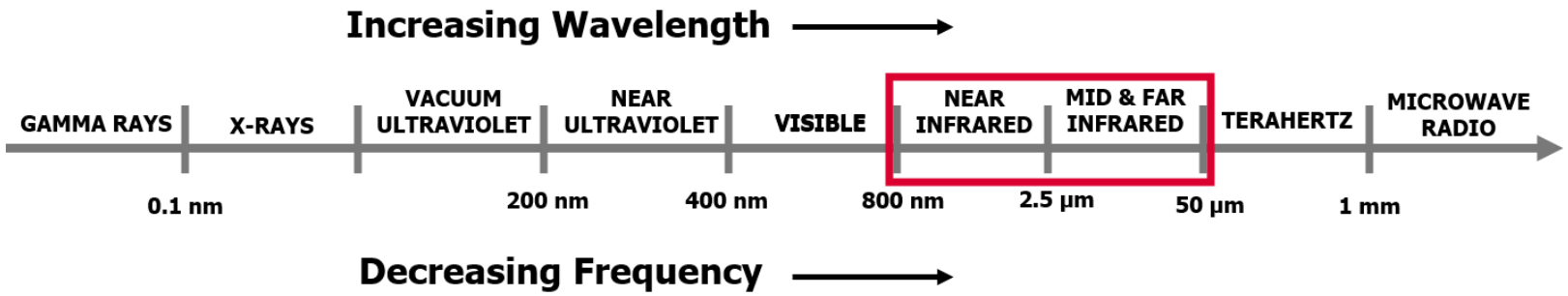
6 Research Groups:

Optics
- Infrared & Raman Spectroscopy
- Optical Coherence Tomography
- Terahertz Technology

Acoustics
- Laser-Ultrasound
- Photoacoustics
- Physical & Computational Acoustics

What is Infrared Spectroscopy?

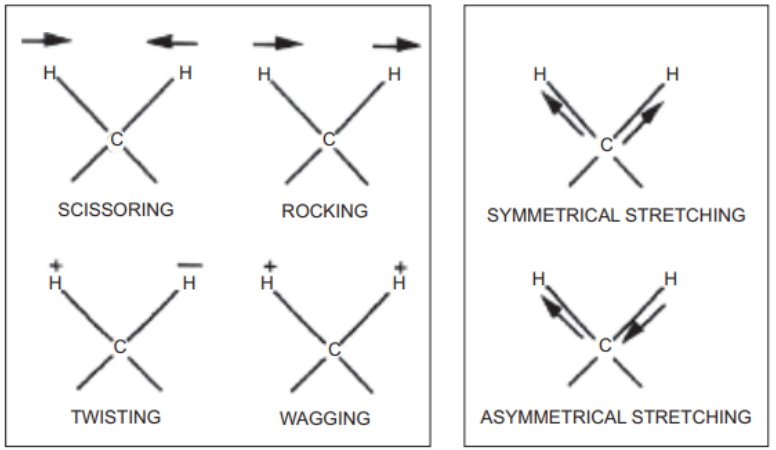
Infrared radiation:



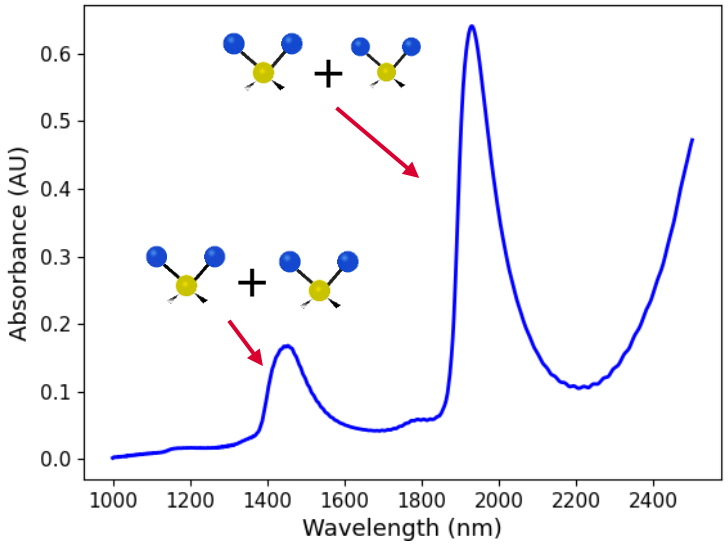
Infrared Spectroscopy:

- Measurement of molecular absorptions
- Molecules absorb specific parts of IR radiation
- Absorption of radiation induces molecular vibration
- Mid Infrared: fundamental vibrations
- Near Infrared: overtone and combination vibrations

Examples of fundamental vibrations



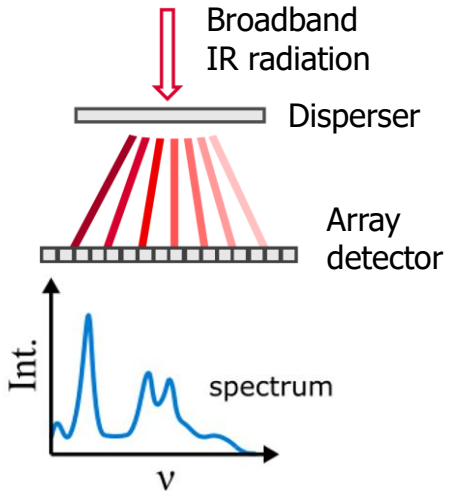
NIR Absorbance spectrum of water



How can we measure IR radiation?

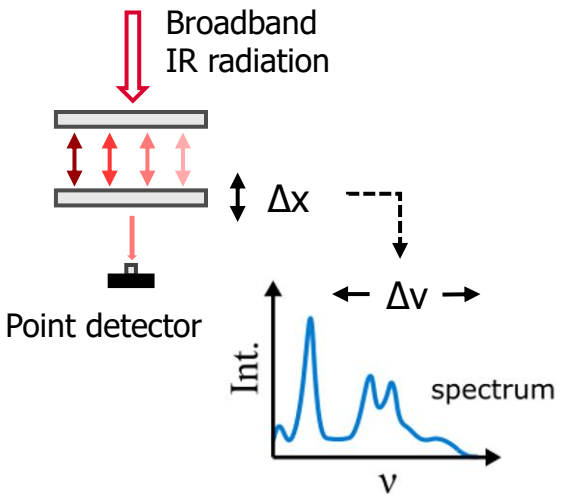
3 different basic types of IR spectrometers:

Dispersive spectrometers



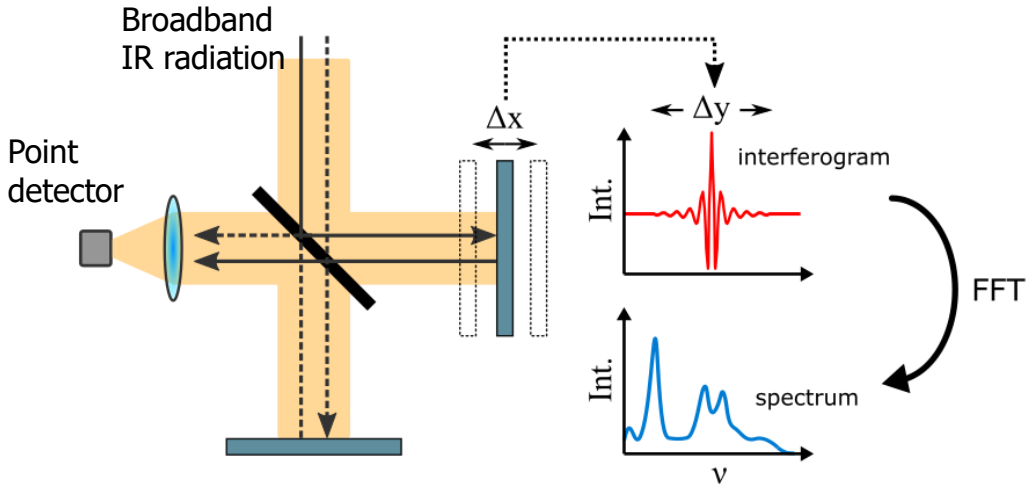
- **Entire spectrum measured at the same time**
- **Fast measurements possible**
- **Narrow entrance slit limits strength of incoming signal**

Tunable Fabry-Pérot filters



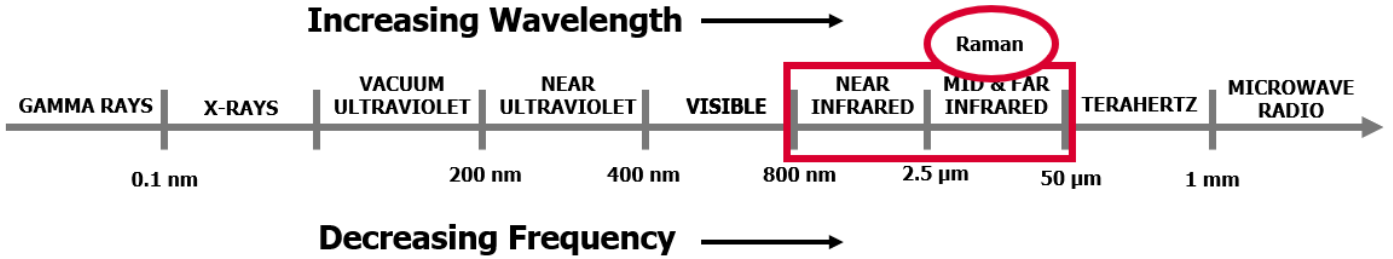
- **Cost efficient**
- **Flexible spectral range**
- **Compact form factor**
- **Narrow spectral range**
- **Low spectral resolution**

Fourier Transform Spectrometer



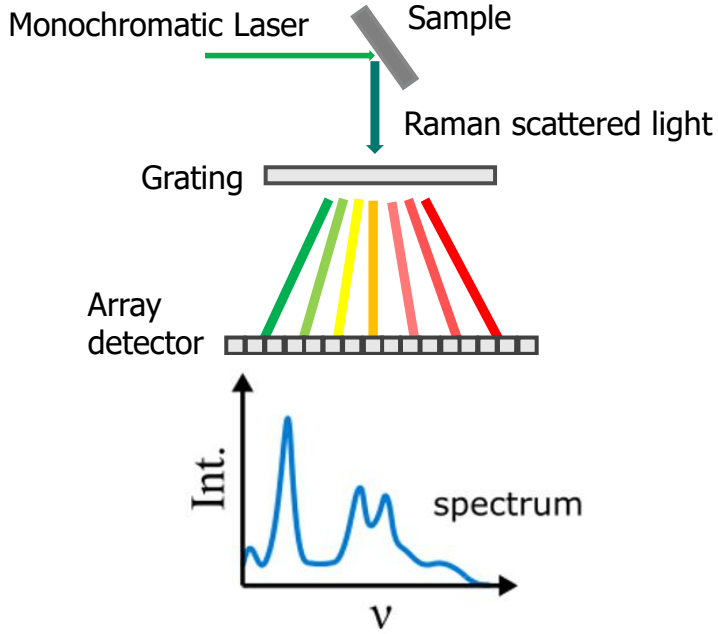
- **Very broad spectral range**
- **Spectral multiplexing advantage**
- **High spectral resolution**
- **Inflexible (fixed spectral range)**
- **Relatively low measurement speed**

What is Raman Spectroscopy?



Raman scattering:

- Inelastic scattering effect
- **Monochromatic** (Visible-NIR) **light** used to excite Raman scattering
- Raman scattered **light** gets **shifted** towards lower frequencies
- **Shift corresponds** to wavelengths (frequencies) of **fundamental molecular vibrations**



Difference to Infrared Spectroscopy:

- Shifted wavelength still in the Visible-NIR light regime
- **Silicon detectors** can be used → spectrometers are simple and sensitive
- IR- and Raman spectroscopy are **complementary techniques**
- Raman spectrum is less influenced by water (often preferable for aqueous samples)
- Raman: **sensitive** to vibrations of **homonuclear bonds**
- Infrared: sensitive to **polar bonds**

NIR Spectroscopy in PAT (Process Analytical Technology)

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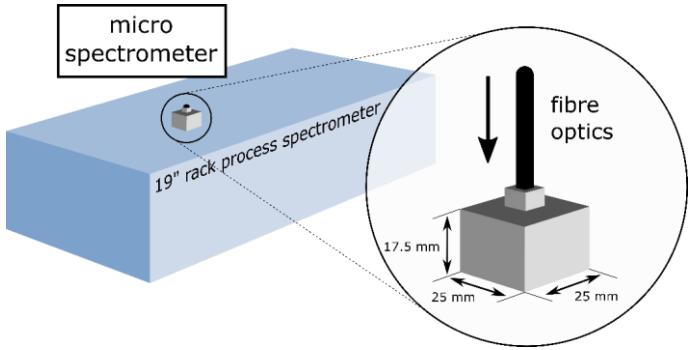
NIR is an established technique in PAT

- In-process measurements of **many different materials/chemicals**
- Effective **chemical analysis** (multivariate data analysis/chemometrics)
- **Real time** measurements
- **Contact-less** measurements possible
- Highly compatible with **fiber-optics**

Recent development: **Miniaturization of spectrometers**

- Higher cost efficiency / lower price
- Compact hardware
- High ruggedness / low maintenance



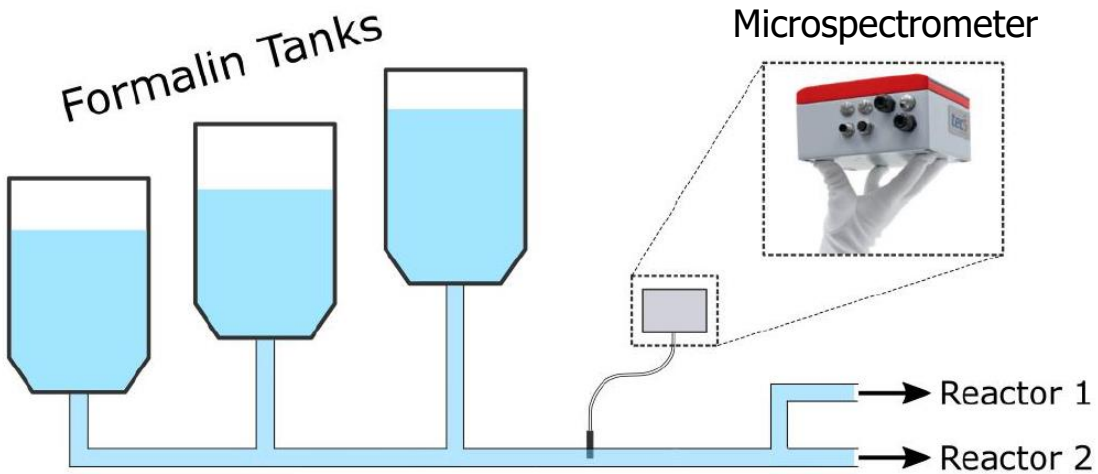
<3 k€

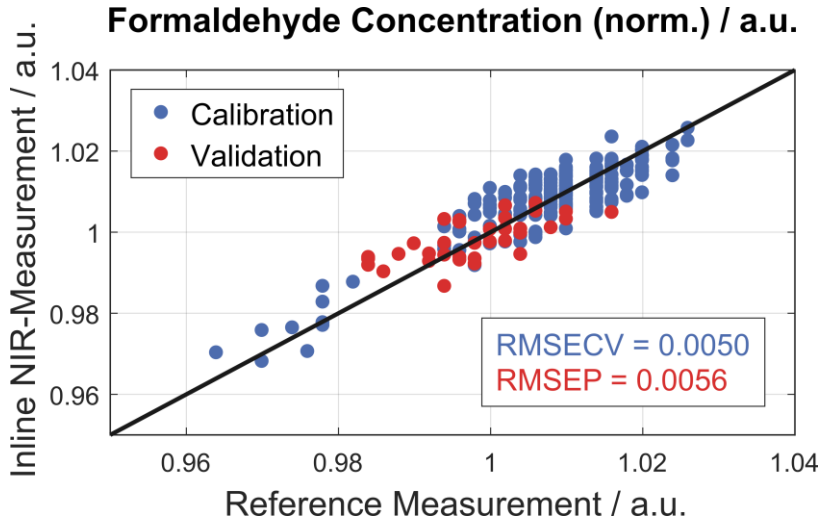
Application Example – Formalin Measurement

NIR-based in-line process control in a **melamine (MF)** and **phenol formaldehyde (PF) resin** batch production

- Implementation of miniaturized spectrometer for **in-line monitoring** of **formaldehyde concentration** in **formalin** via immersion probe
- Real-time NIR-measurement utilizing miniaturized NIR spectrometer

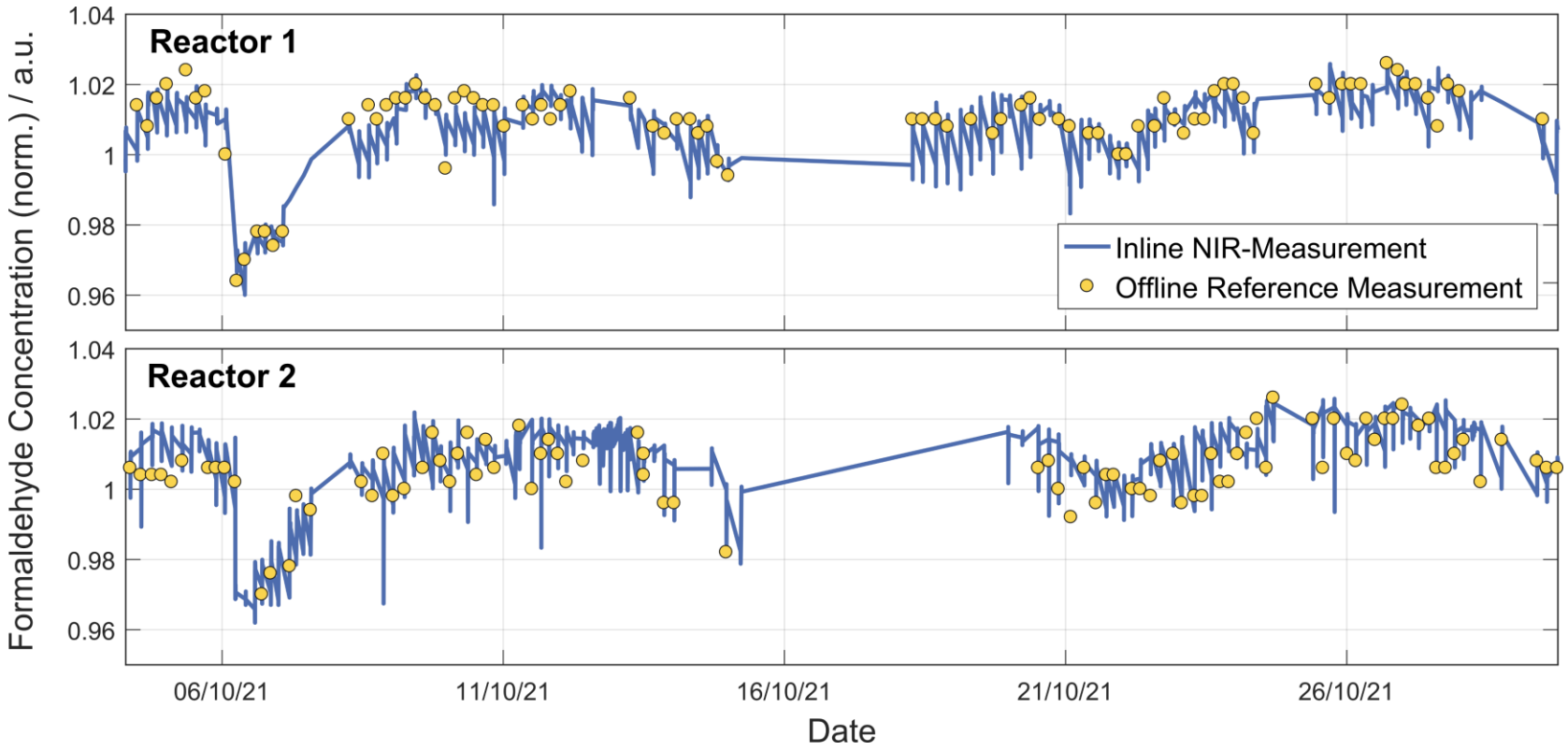
Immersion probe





Application Example – Formalin Measurement

Comparison of **in-line acquired data** to **off-line reference measurements**



→ Reduction of wet chemical offline reference measurements by >90%

→ **Savings of ~200k€ per year** due to reduced efforts

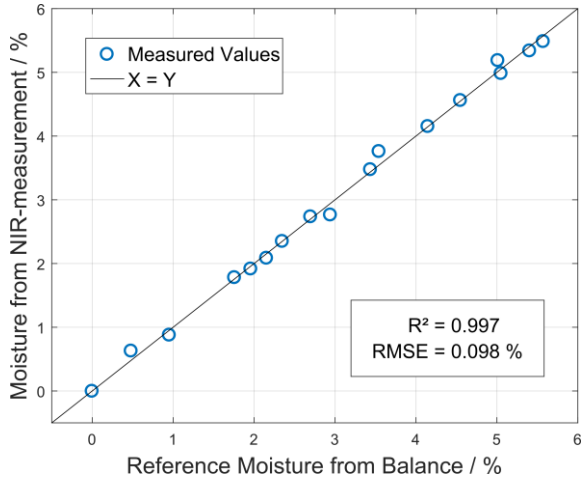
→ **Better working conditions** (reduced exposure to hazardous formaldehyde)

→ **Additional revenue** due to more efficient use of available reactor space

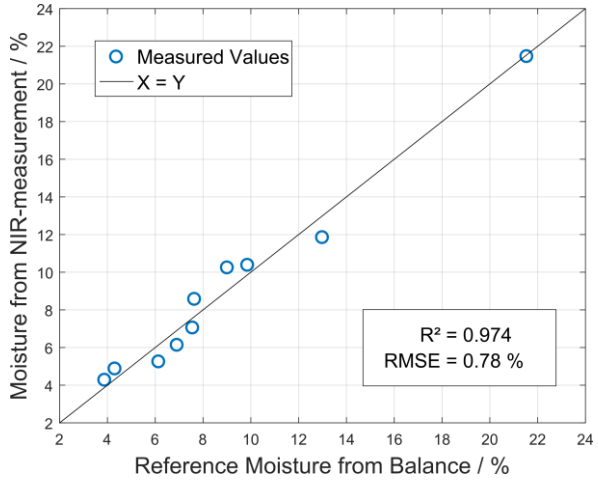
Application Example – Moisture Monitoring

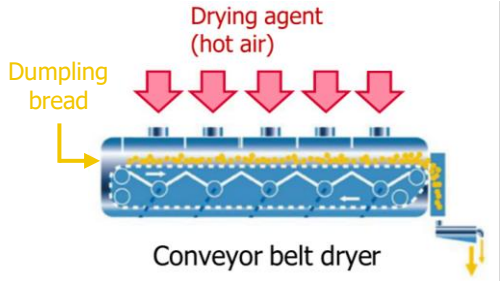
NIR-based moisture sensor for in-line moisture measurement of **products in the food industry**

- **Starch derived products**



- **Dumpling bread**





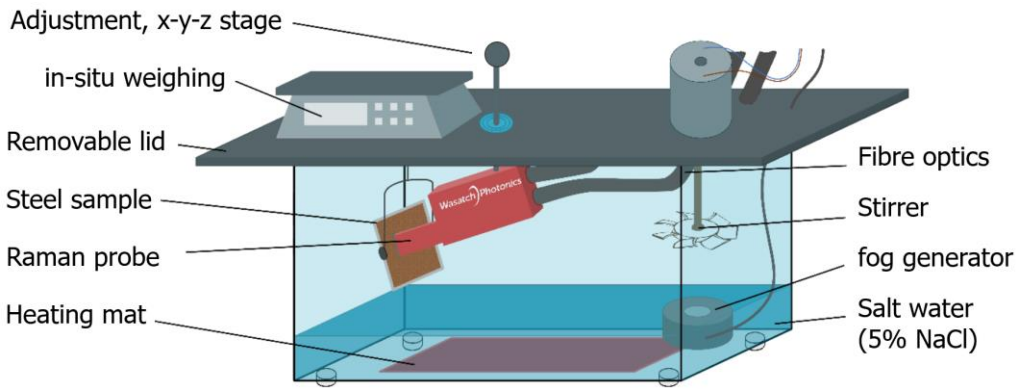
- Calibration curves for both products demonstrate **applicability for in-line moisture measurement** using miniaturized NIR-spectrometers

- Development of the final customer specific inline sensors is currently in progress

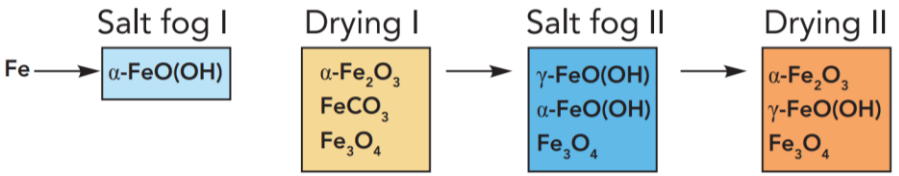
Application Example – In-Situ Raman Measurements

In-situ Raman measurements of **mild steel corrosion**

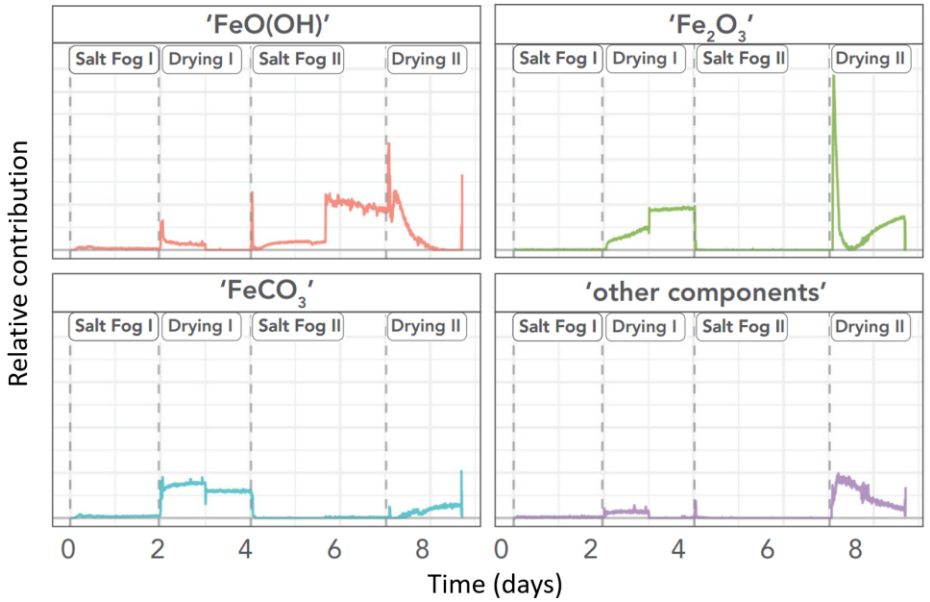
- Sped up corrosion in salt-fog chamber
- 8 days of corrosion cycles (salt-fog & drying phases)
- **Chemical transitions** during corrosion cycles could be **monitored**



➢ **Raman Spectroscopy is a powerful technique for in-process measurements even under adverse conditions (here: salt-fog)**



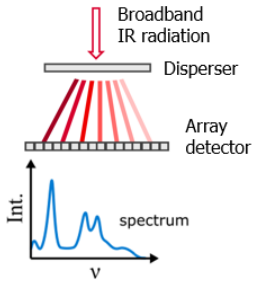
Representation of the chemical transitions during the corrosion cycles

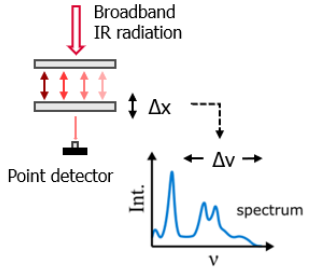


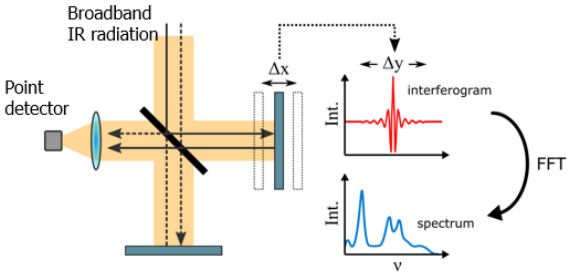
Dynamics and time profile of the contributions of four components to the experimental spectra set, as determined with MCR-ALS analysis, here tentatively assigned to four chemical species as indicated.

Summary

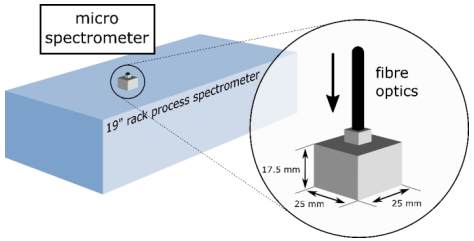
- Infrared & Raman spectroscopy are complementary **non-destructive** methods for **chemical analysis**
- **3** basic **different types** of IR-spectrometers







- **Contactless** as well as in-contact real-time **measurements** are possible
- Highly compatible with **PAT applications**
- Miniaturized spectrometers allow for **strong cost reduction without significant cutbacks** in measurement performance



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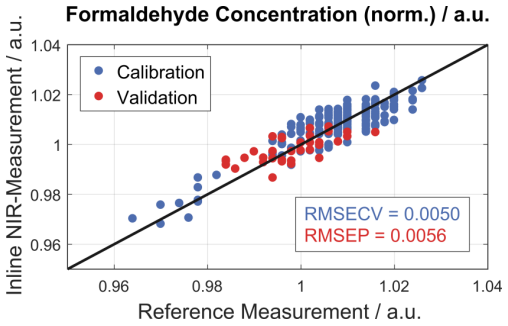

Acknowledgements

Infrared and Raman Group @ RECENDT

External Partners:

Funding:

Europäische Union Investitionen in Wachstum & Beschäftigung. Österreich.