IRmadillo™

KEIT spectrometers

An online and real-time process analyser for closed-loop control in pulp & paper manufacturing

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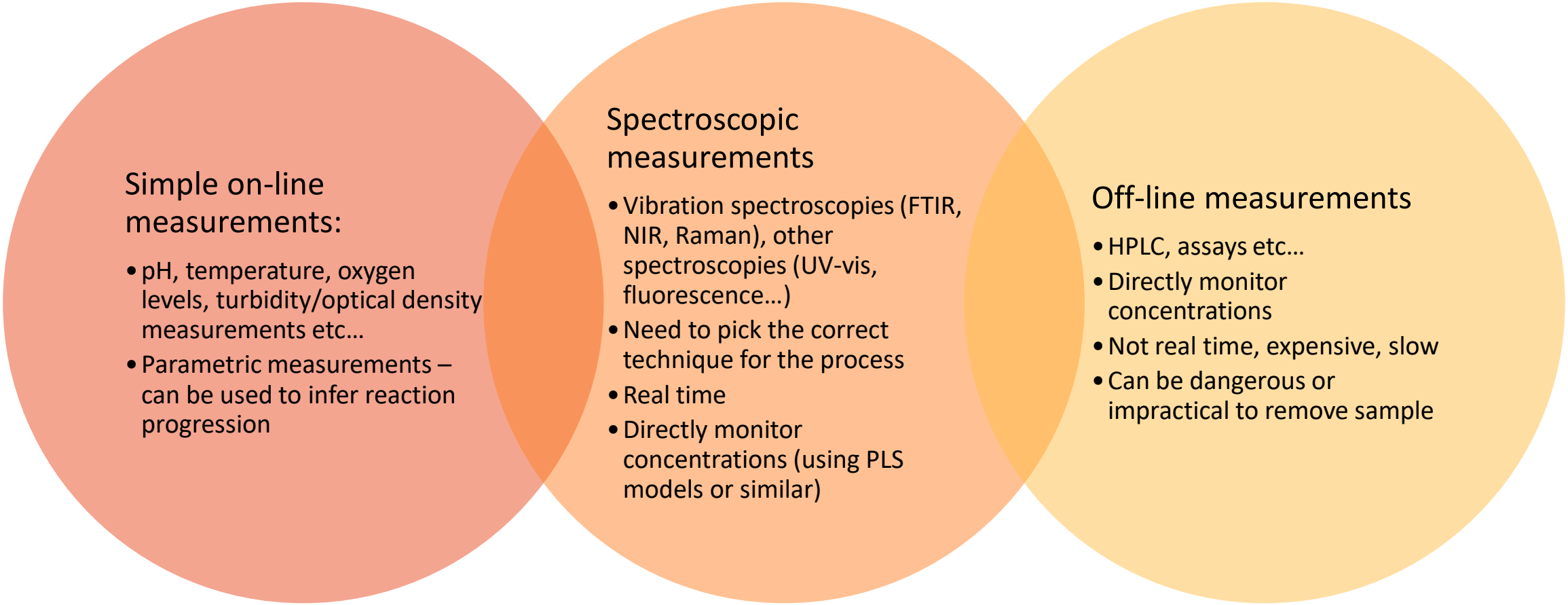
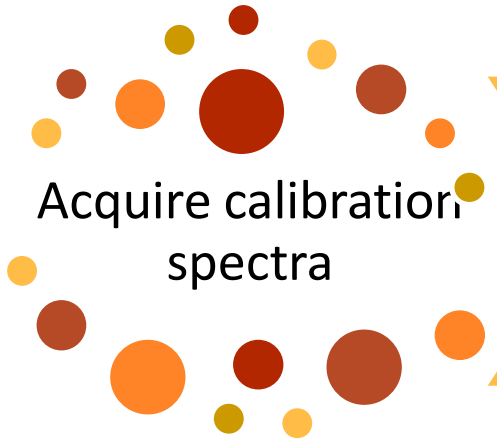
Why use spectroscopy to monitor processes?

On-line, real-time measurements | **Direct Concentration Measurements**



How to use spectroscopy for monitoring a process



- Specific calibration set (i.e. DoE)
- Acquired during normal process (i.e. piggy-backing on a fermentation)

Use set of reference concentrations

- For DoE this is known already
- For piggybacked processes gathered from extractive sampling (i.e. HPLC or assays)

Build model

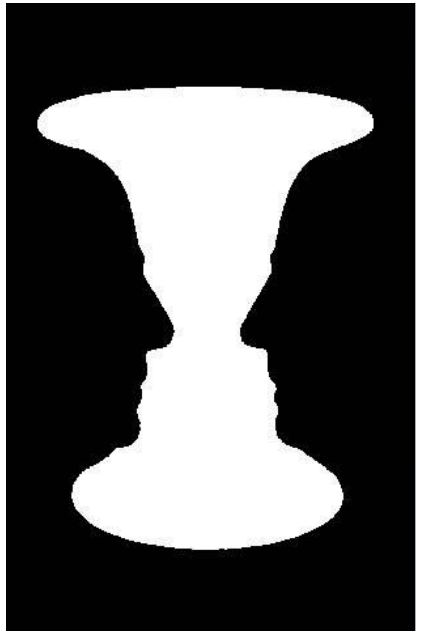
- Univariate model (i.e. Absorbance = (intensity x multiplier) + offset
- Chemometric model (i.e. PLS or SVR)

Validate model during use

Take off-line samples to confirm model performance

We know this! We tried NIR. It didn't work!

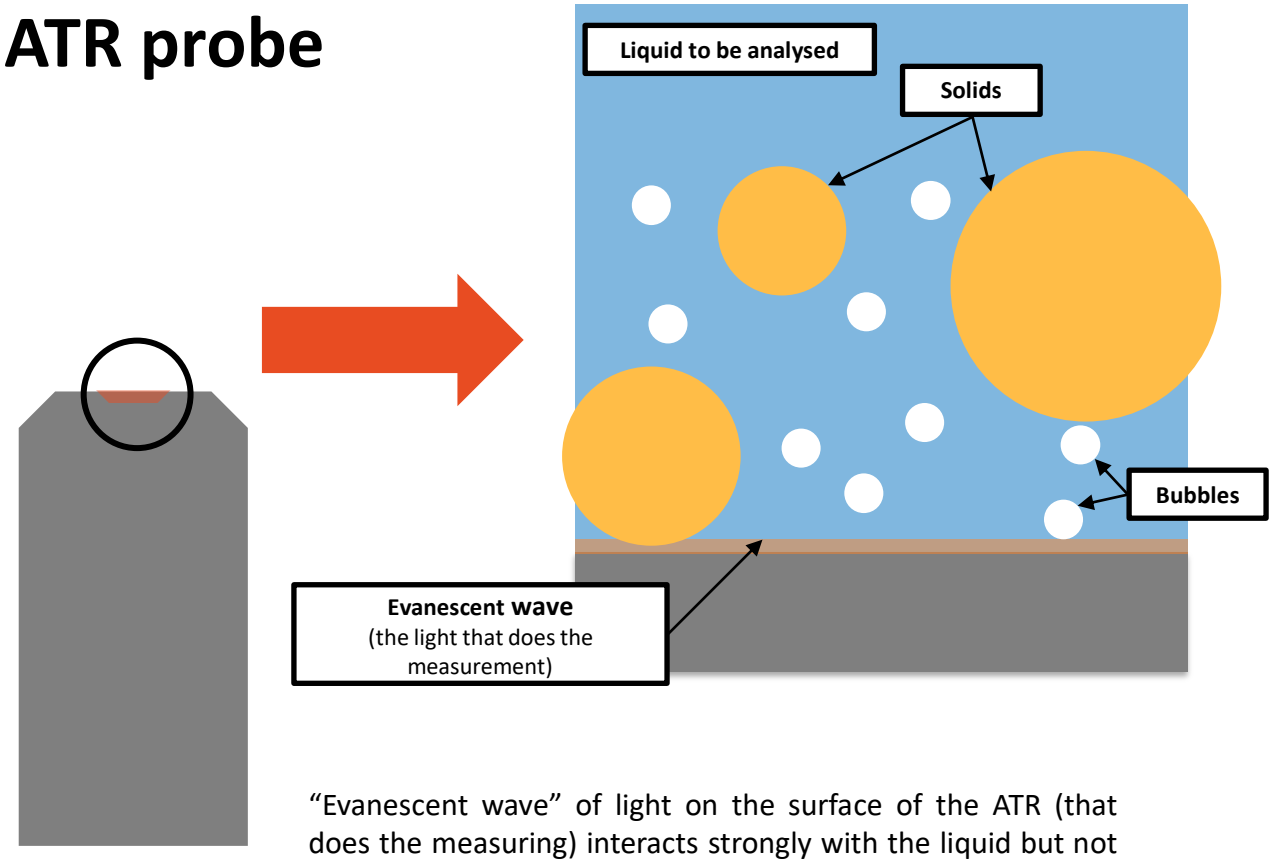
- NIR and FTIR give different information
  - NIR: looks at "combination bands" and "overtones" – hard to tell similar molecules apart
  - FTIR: looks at fundamental vibrations of molecules



NIR – is it a vase or two faces?

Benefits of ATR vs conventional NIR instruments

ATR probe

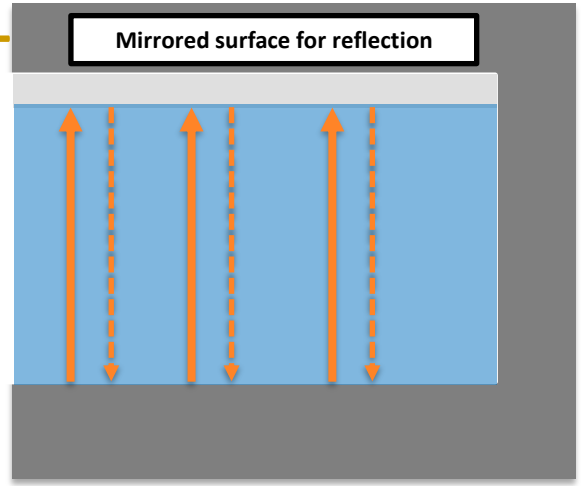


"Evanescent wave" of light on the surface of the ATR (that does the measuring) interacts strongly with the liquid but not the bubbles and/or solids on the analyte.

NIR reflectance probe – pure liquids

Outgoing and returned light for measurement

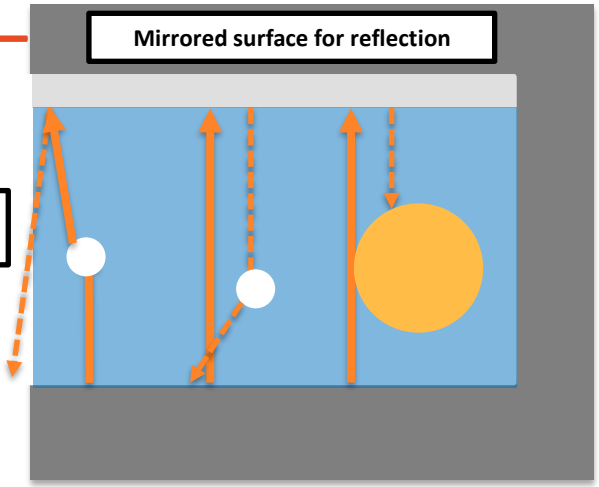
Light is sent out through the sample and reflected off the mirrored surface back into the probe for measurement. The long path length allows for lots of signal – critical to NIR performance.



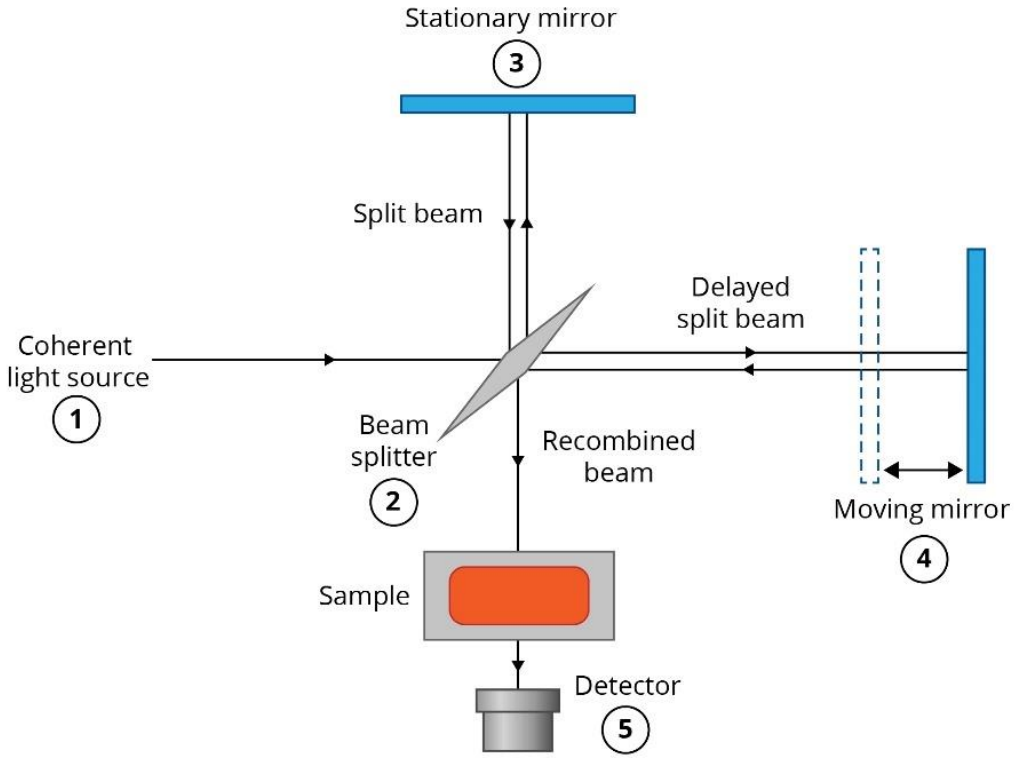
NIR reflectance probe – with solids or bubbles

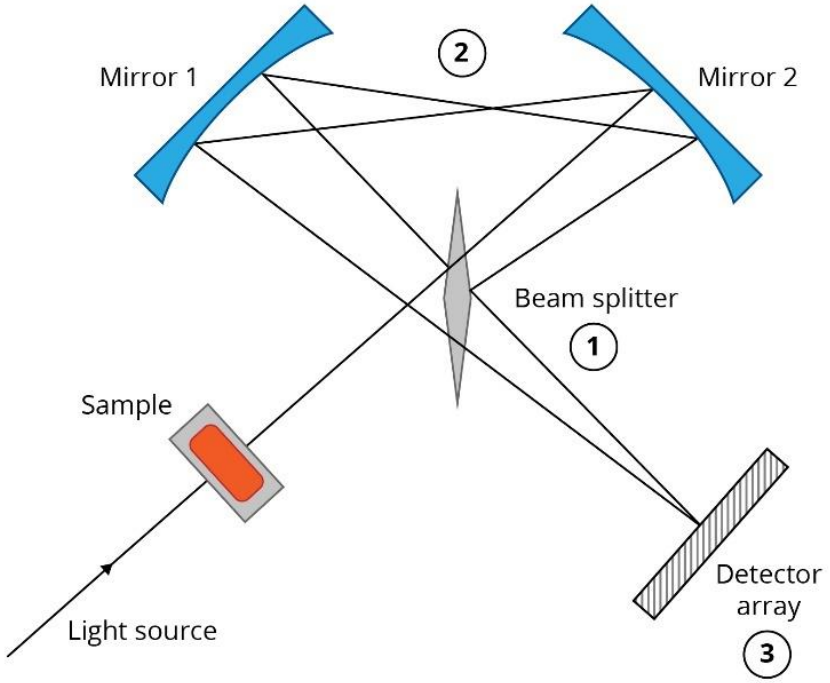
Outgoing and returned light for measurement

The bubbles and/or solids in the sample interfere with the light being returned back into the probe. This causes poor accuracy, precision and reliability. There is no way to remove this issue with conventional NIR instruments as they *require* long path lengths to give any signal.



So what makes this FTIR different?

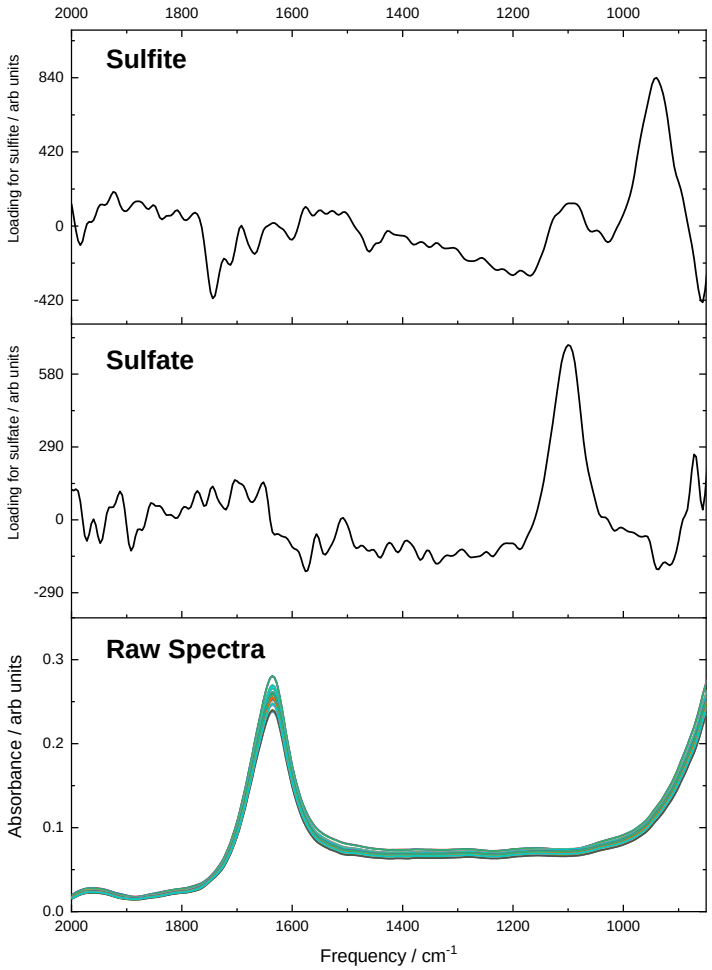


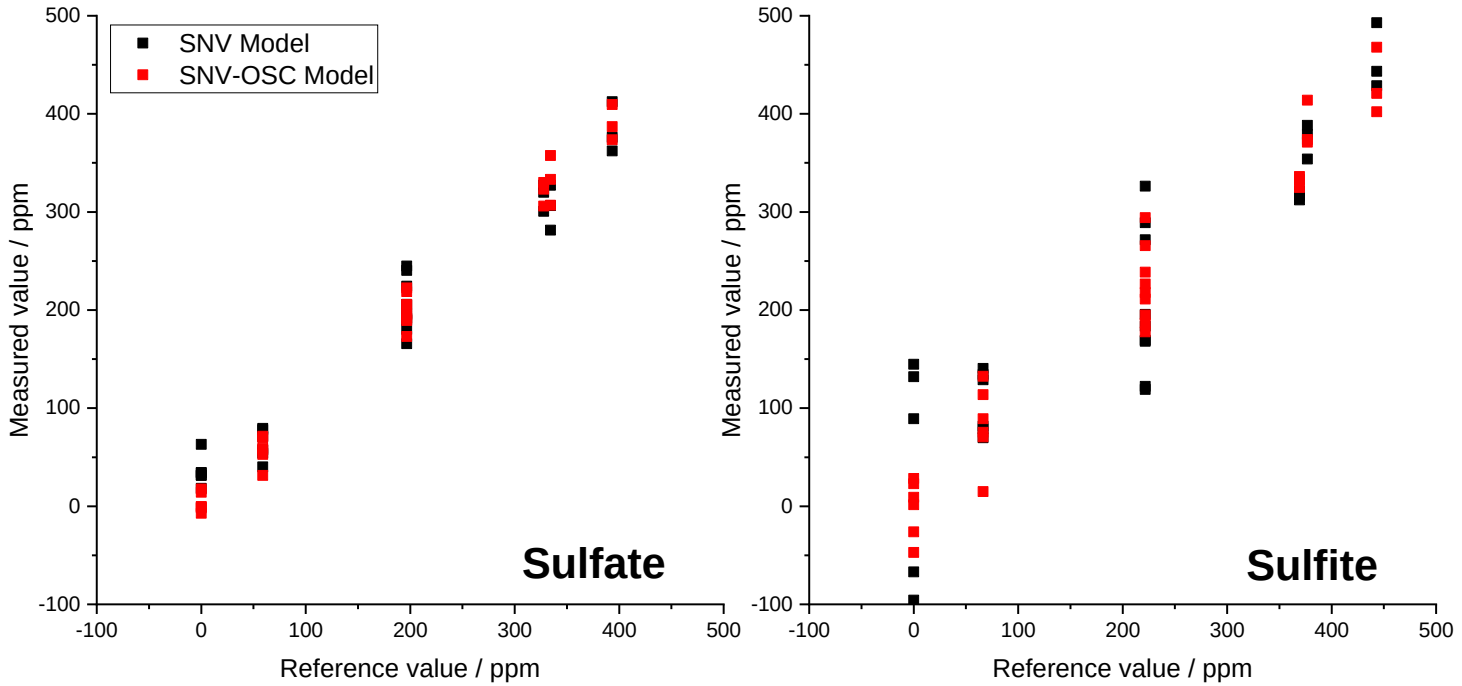


Classical Michelson interferometers
Moving mirrors, fragile optics, needing a fibre probe and unsuitable for manufacturing environments

Static Optics Sagnac interferometer
Static mirrors, robust optics and no need for a fibre probe: suitable for manufacturing environments

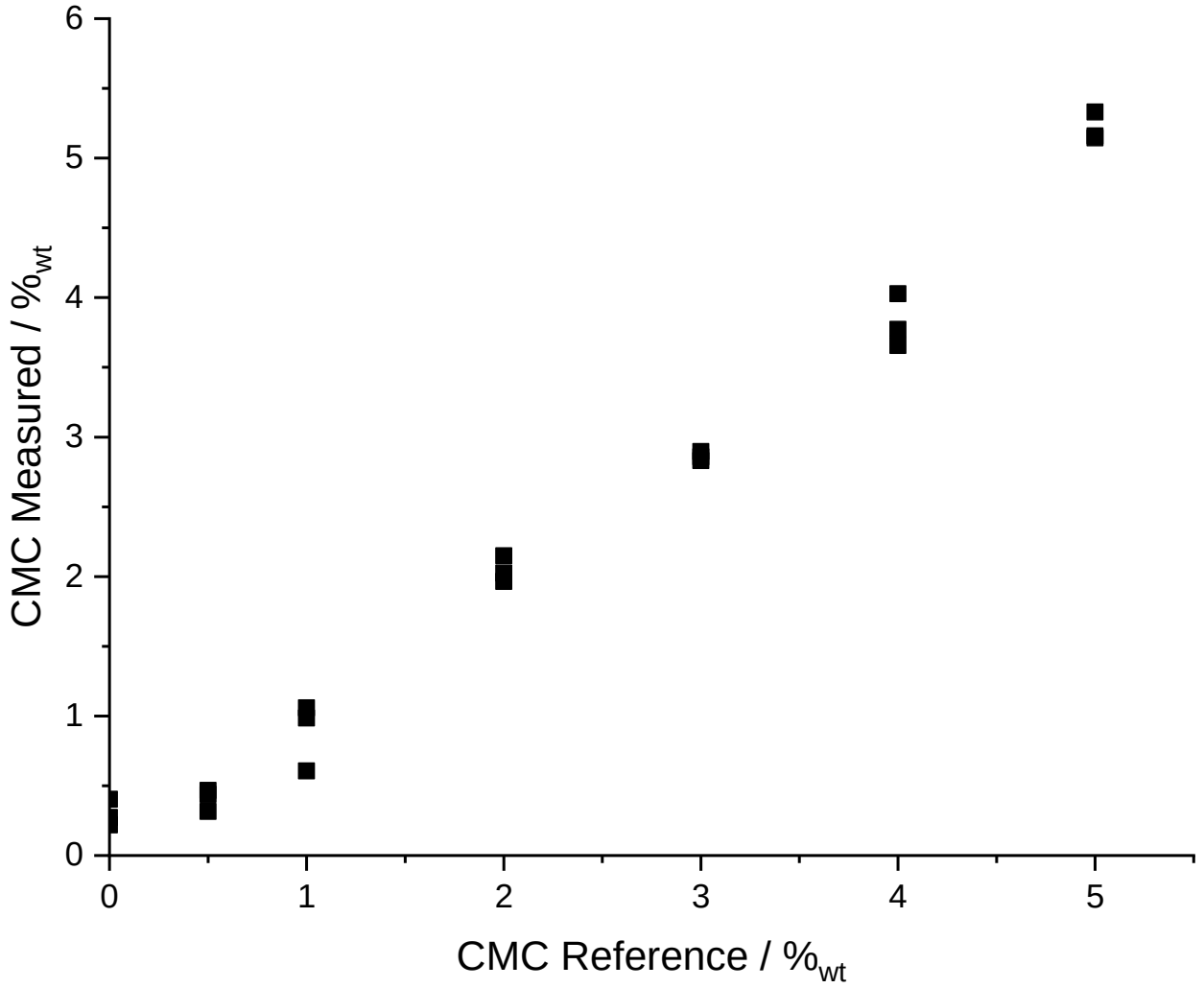
Measuring sulfite and sulfate levels in aqueous mixtures





Left: loadings for sulfite and sulfate from PLS models, and the raw spectra
Above: predicted vs actual plots for sulfate and sulfite calibration models

Just sulfite and sulfate?



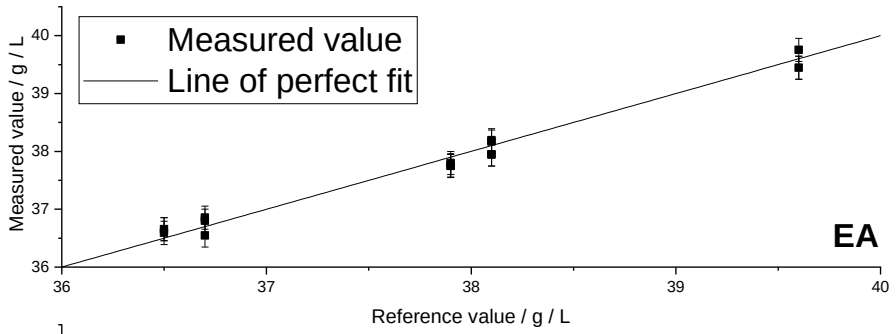
Left: measured vs reference plot for CMC calibration (RMSECV of 0.5% obtained from this dataset).

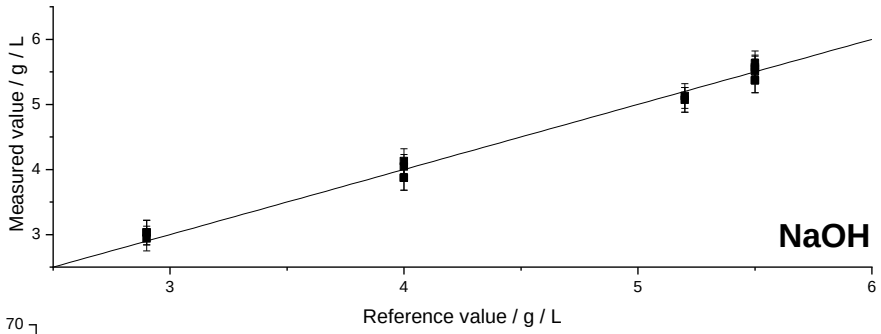
As long as the molecules of interest either contain covalent bonds (such as CO_3^{2-} and SO_4^{2-} ions, or organic molecules like sugars, additives, lignin etc…) or have an effect on covalent bonds in other molecules (such a S^{2-} ions) then FTIR can observe and quantify the chemical of interest.

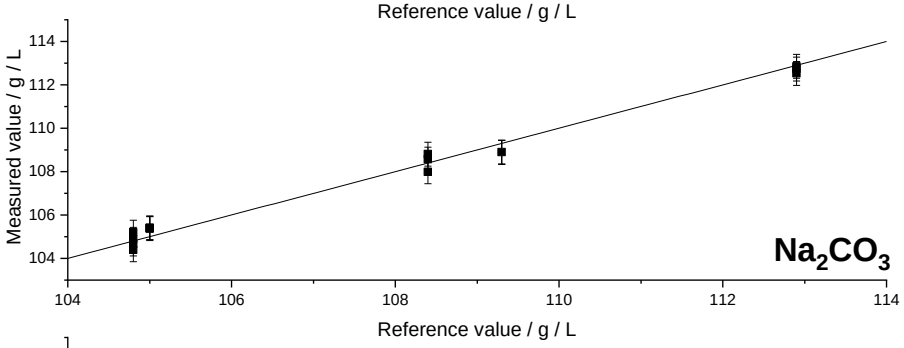
What about real-life samples?

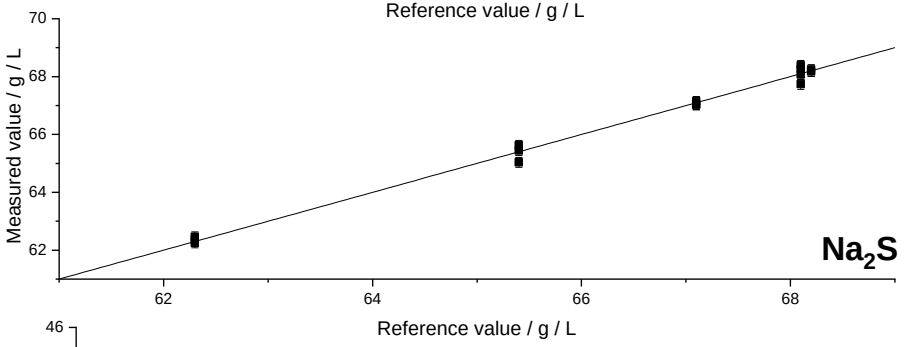
- Green liquor samples received directly from customer
- Indicative calibration models built using samples are received:
  - Effective alkali
  - NaOH
  - Na_2CO_3
  - Na_2S
  - Na_2SO_4
  - Total sulfur

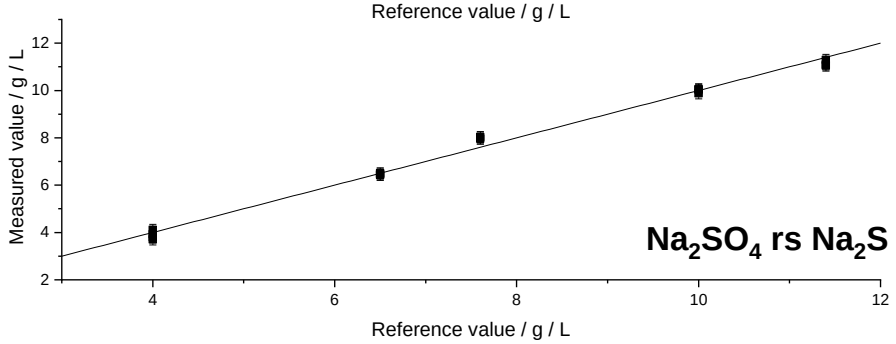
Results from calibration

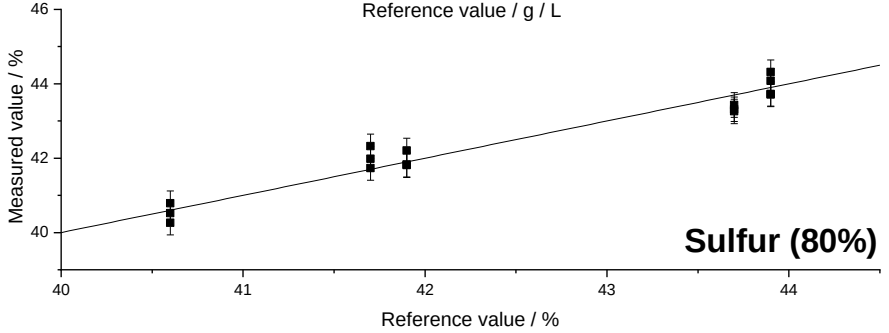








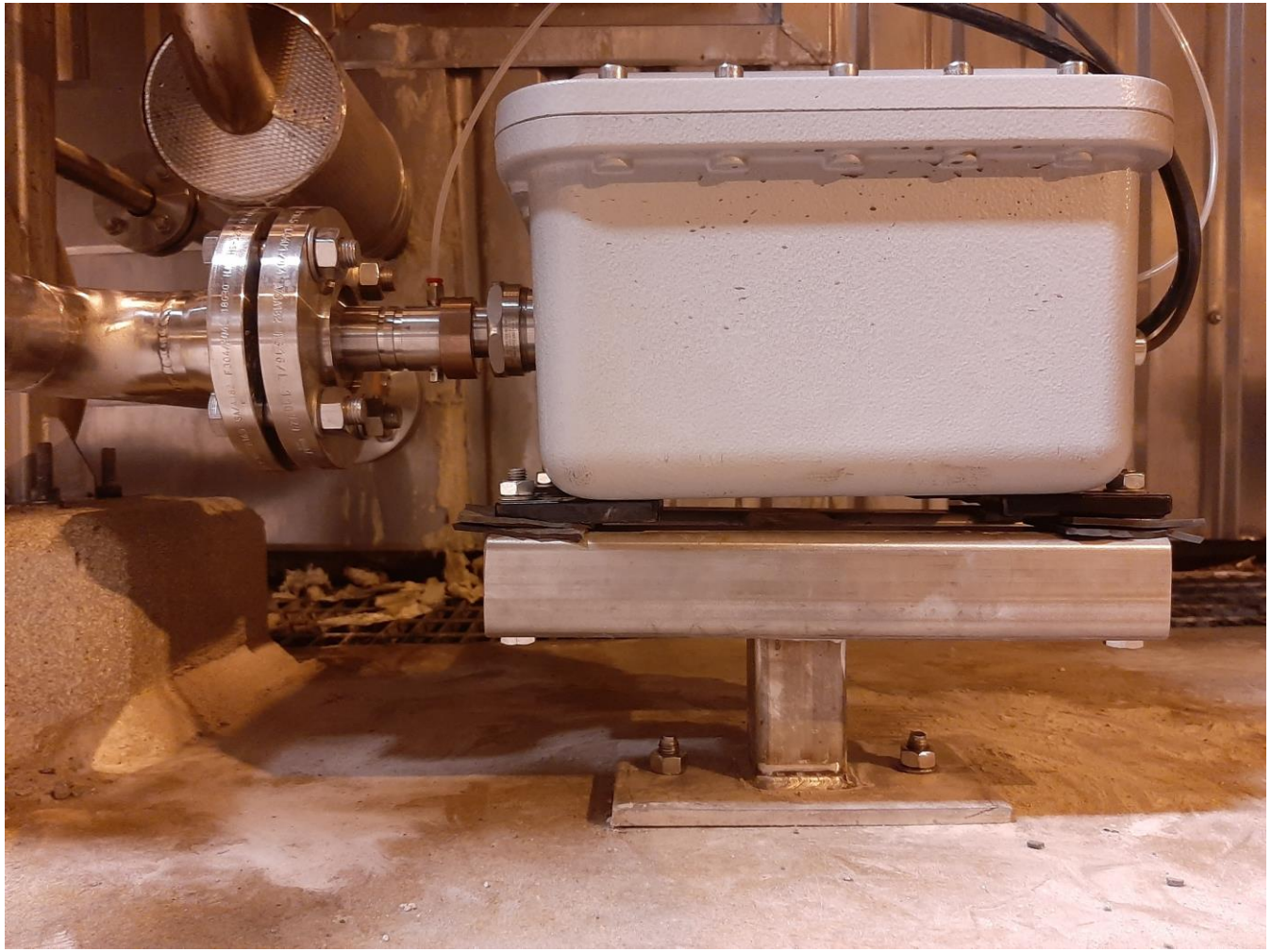


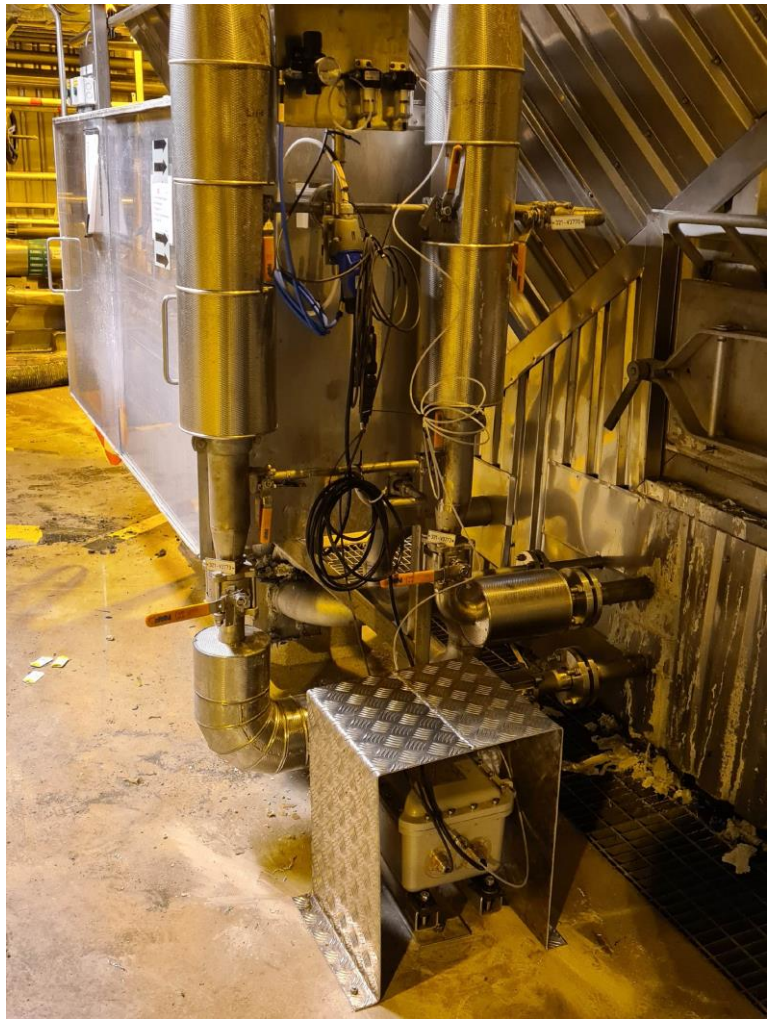


Excellent calibration for every chemical and property studied.

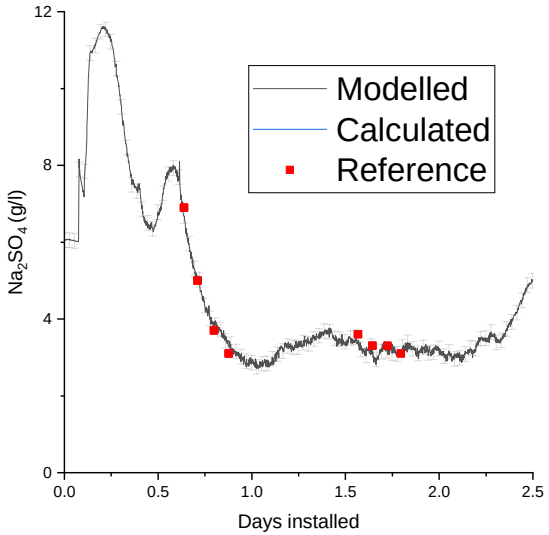
EA: +/- 0.2 g / L
NaOH: +/- 0.19 g / L
Na_2CO_3: +/- 0.55 g / L
Na_2S: +/- 0.17 g / L
Na_2SO_4:+/- 0.23 g / L
Sulfur: +/- 0.33 %

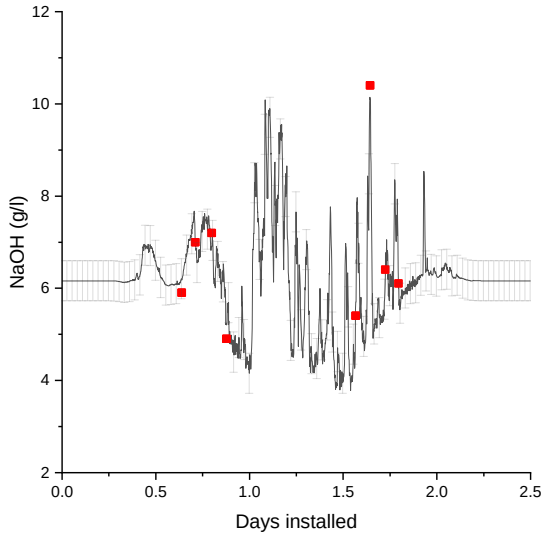
Green liquor installation

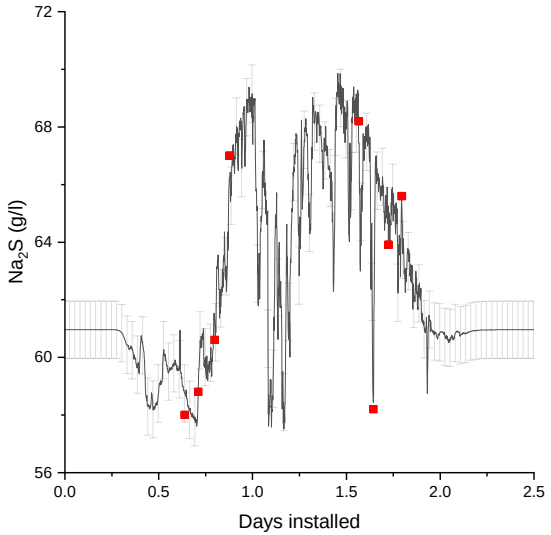


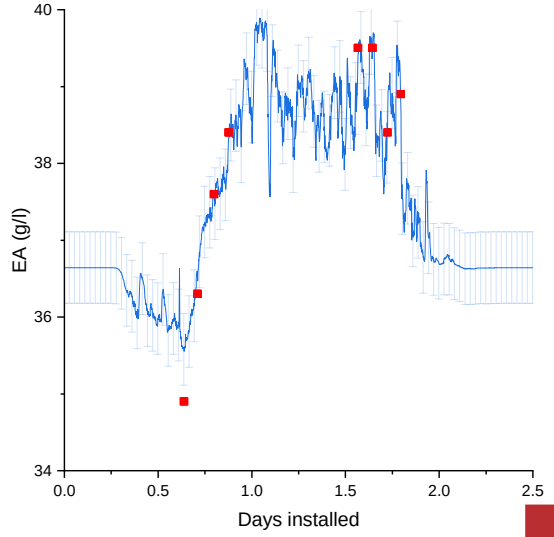


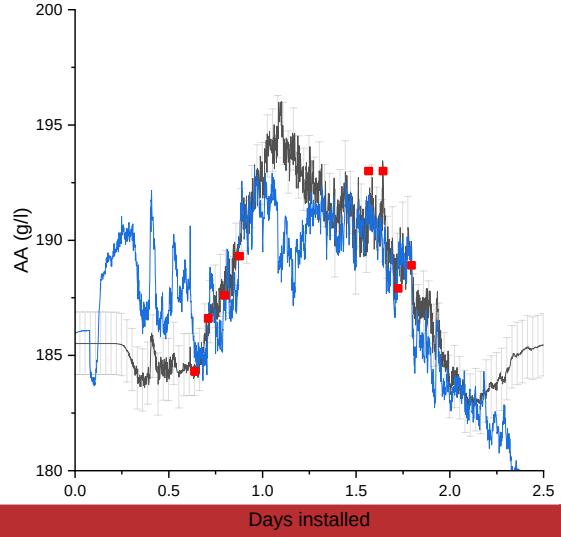
Preliminary results from green liquor application pulp and paper mill in Sweden

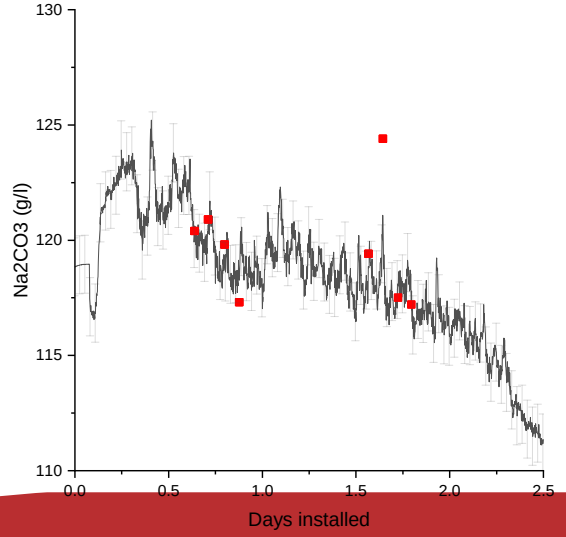












Process data from installed instrument on green liquor line measuring a range of different components in real time.

Some features – such as AA – have been calibrated using online data and also calculated from raw chemical concentrations. Both are shown if applicable.

Conclusions

- Spectroscopy as a technique can and should be an important facet of modern manufacturing principals and Industry 4.0
- Classical spectroscopic techniques (such as NIR) are difficult to use for real-world problems
  - They require intensive model maintenance and lots of calibration samples
  - The probes themselves aren't suitable for many turbid or heterogenous liquids
- Static optics based FTIR spectrometers can be used for monitoring and controlling pulp and paper process right from wood processing through to additive control on the final product