IRmadillo™

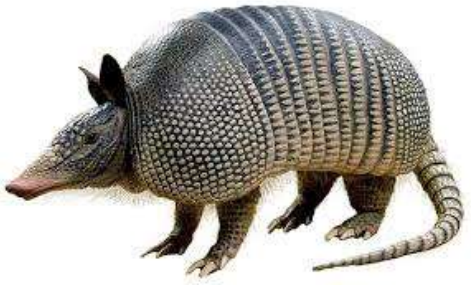
KEIT spectrometers

Robust FTIR for real-time process monitoring of liquids

Who are we?

Keit is a spin out from the UK government space research centre near Oxford, UK.
Our technology was originally designed for a Mars research project and has since been improved for use in industry.

What is the IRmadillo?

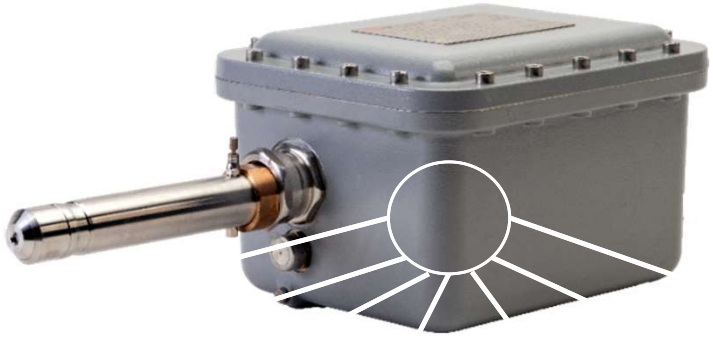


- Robust process FTIR spectrometer
- Real-time, multi-component analyser for the process industries
- ‘Concentration meter’ for liquids
- Enabler for Process Analytical Technology (PAT)

IRmadillo™

IRmadillo for Process Industries

IRmadillo™



Oil & Gas

Petrochemical

Biorenewables

Pharmaceuticals

Edible Oils

Alumina Refining

Pulp & Paper

Industry drivers

Efficiency

Process optimisation requires on-line measurement of key properties – payback in energy, yield and quality

Complexity

Complex chemistry requires insight into reaction conditions

Digitalisation

Real-time information contextualised with sensor, process and equipment data for agile manufacturing

Safety

Move away from manual sampling and laboratory analyses

IRmadillo: key features & benefits

Stability & Reliability

Years of continuous operation & minimal maintenance

Robust

Vibration tolerant, no moving parts, no fibre between probe and FTIR, no Stirling engine

Low cost of ownership

Low running costs (minimal maintenance)

Reduce down time, worry & cost

Key benefits: stability & robustness

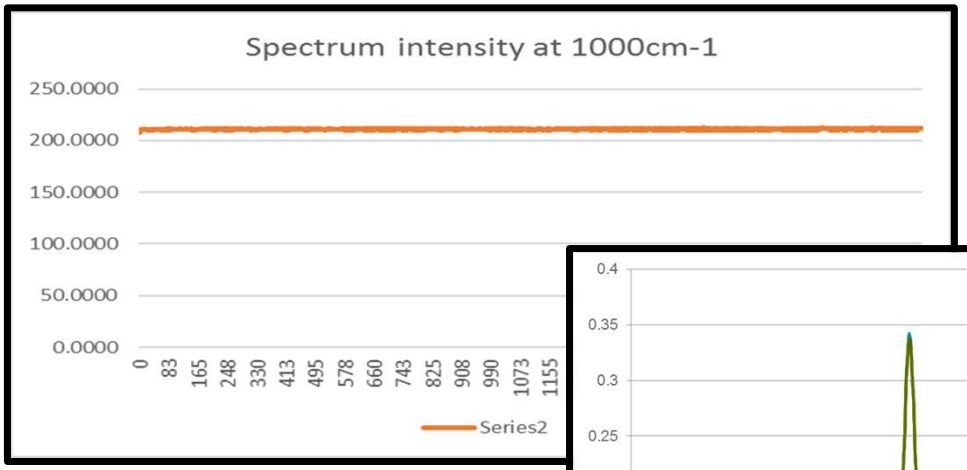
- Patented design with no moving parts – once aligned, it stays aligned
- Fixed mirrors and lenses that don't age
- Emitter lifetime is ~20 years
- Thermal control is at 28˚C - no Stirling Engine to break/replace
- As long as the probe material is compatible with the chemistry, customers are confirming that recalibration is not required - but we recommend a reference check (in air) every few months

Key benefits: stability & robustness

"We have been searching a long time for a rugged industrial infrared ATR spectrometer. We have found the IRmadillo to be compact, robust and easy to implement on-line…..The instrument is working like clockwork, demonstrating remarkable stability and precision on our application".

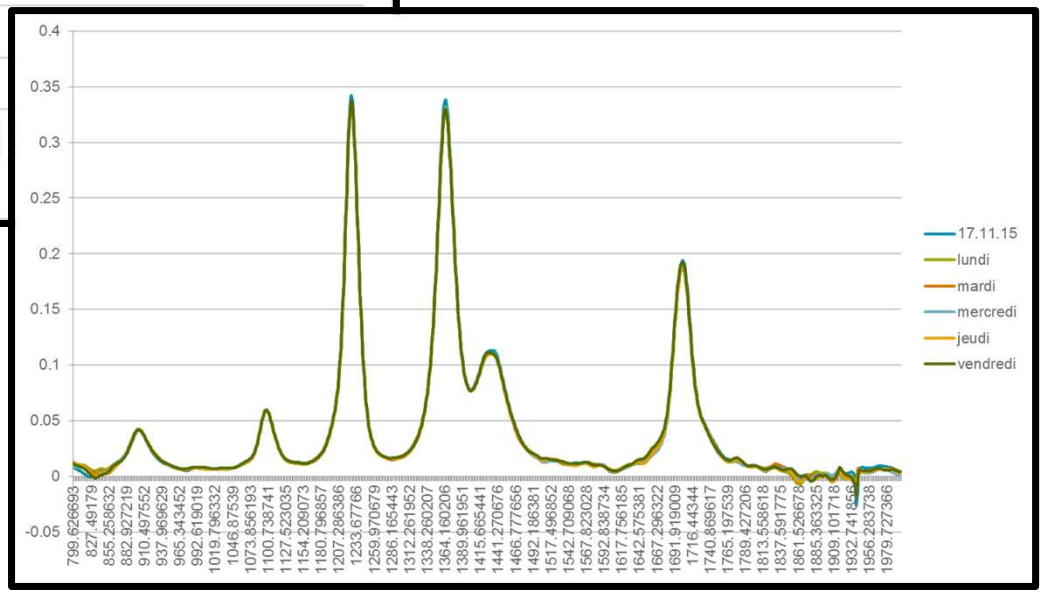
(Customer quote – major European chemical manufacturer)

Fixed optics -> no signal drift -> no re-calibrating
If it can't move, it can't go out of spec …

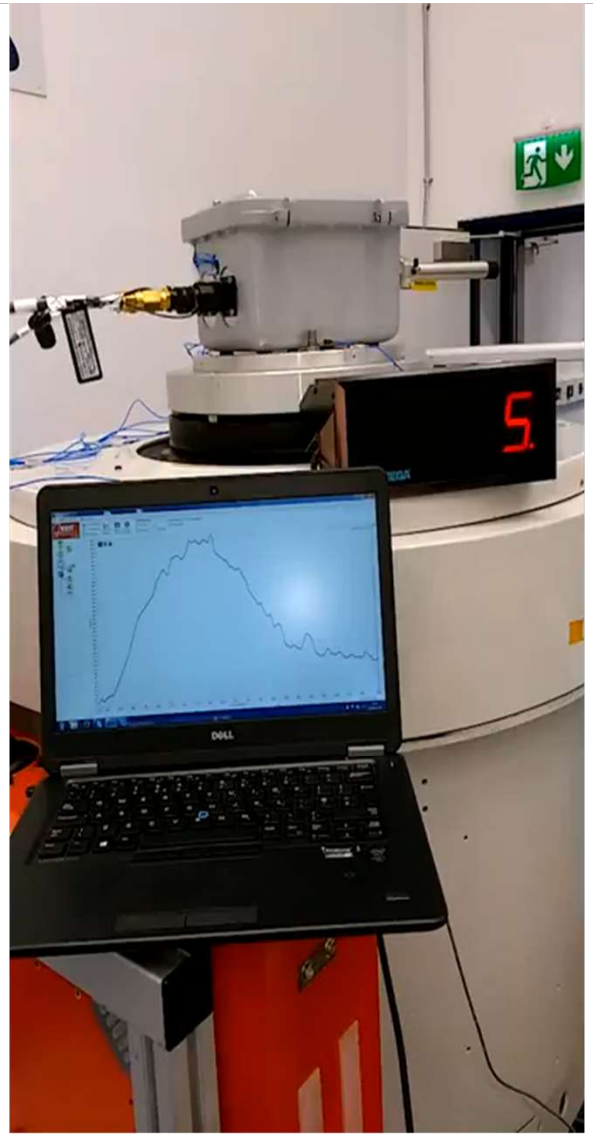


Absorbance at a given wavenumber over 10 months

Customer supplied data: mixture measured 6 times over 3 months, plots superimposed



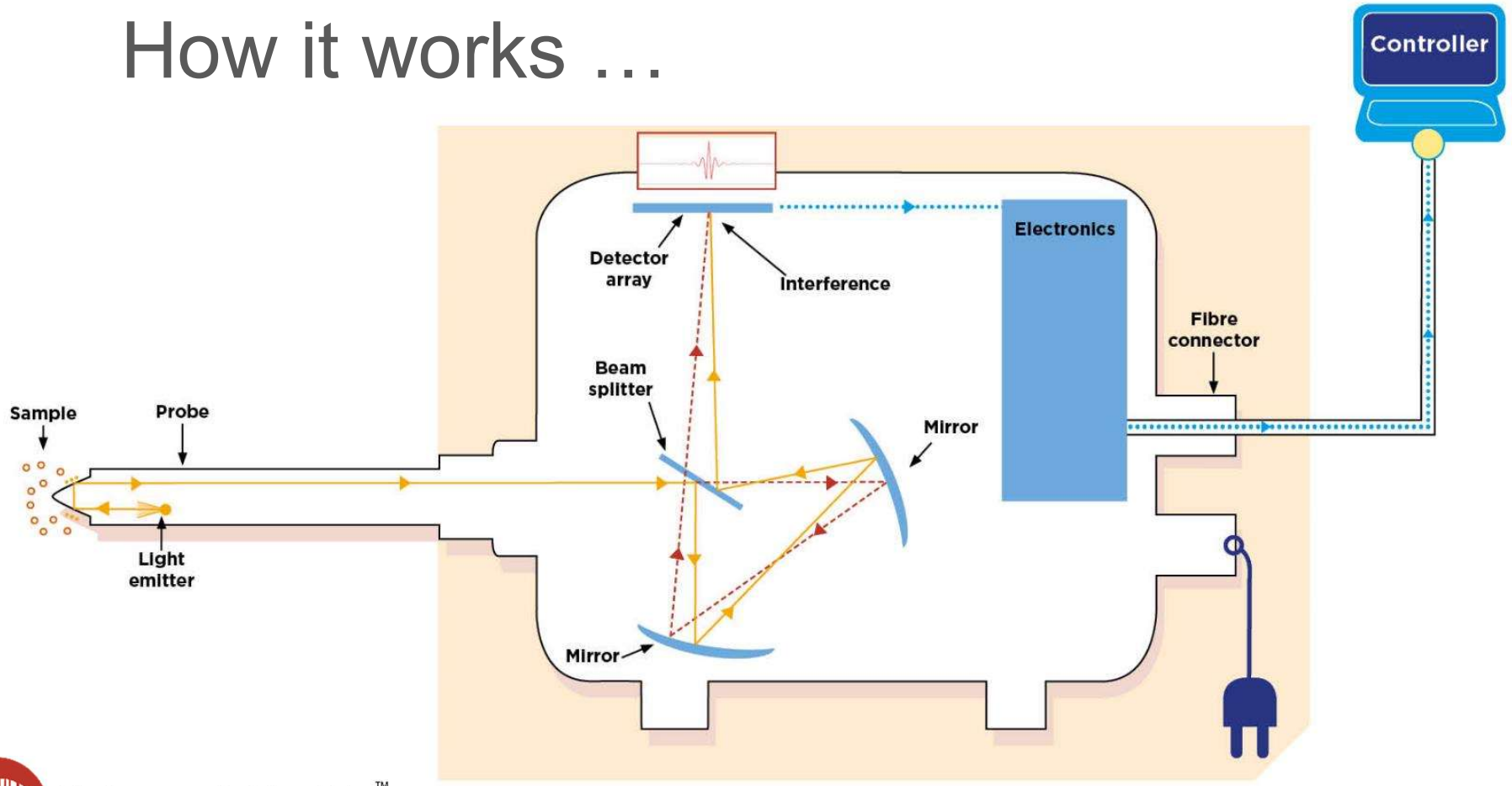
Simulated rocket launch vibration



Key benefit: sensitivity

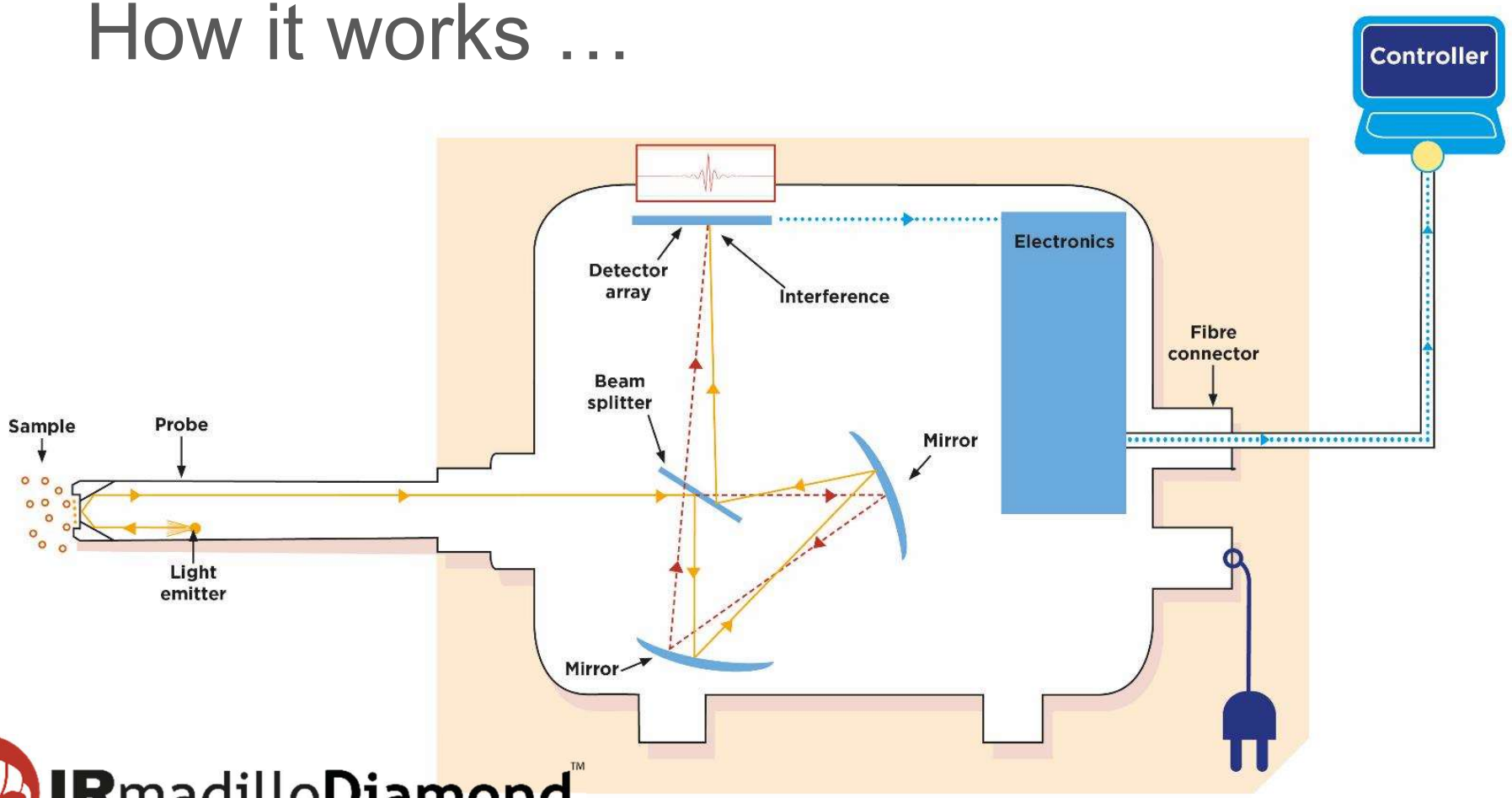
- Measurement capability: typically down to 10-500 ppm depending on chemistry
- Comparative studies by customers indicate equivalent sensitivity to other Michelson-based process FTIR spectrometers
- Operates at a resolution of 16cm^{-1} which is suitable for most liquid mixtures
- Multi-species analysis (simultaneous)

How it works …



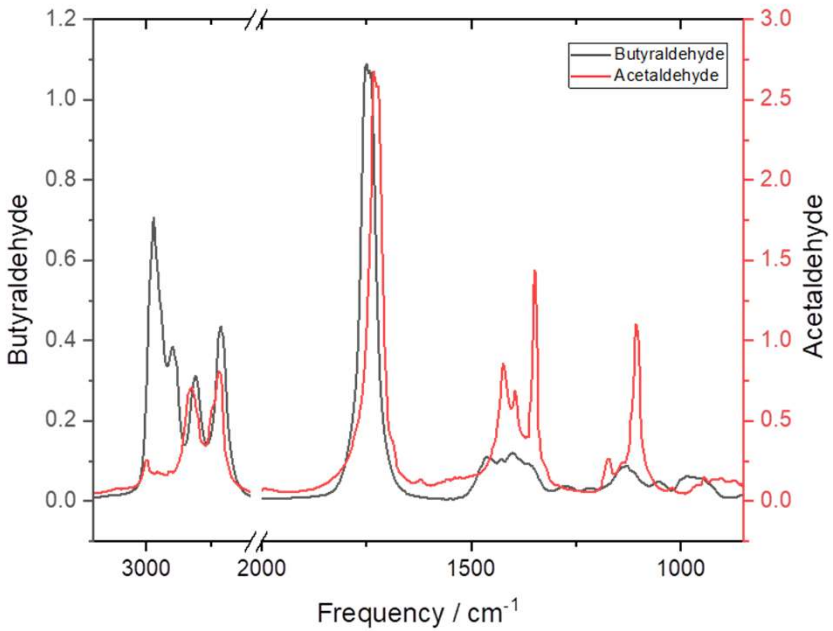
IRmadillo25™

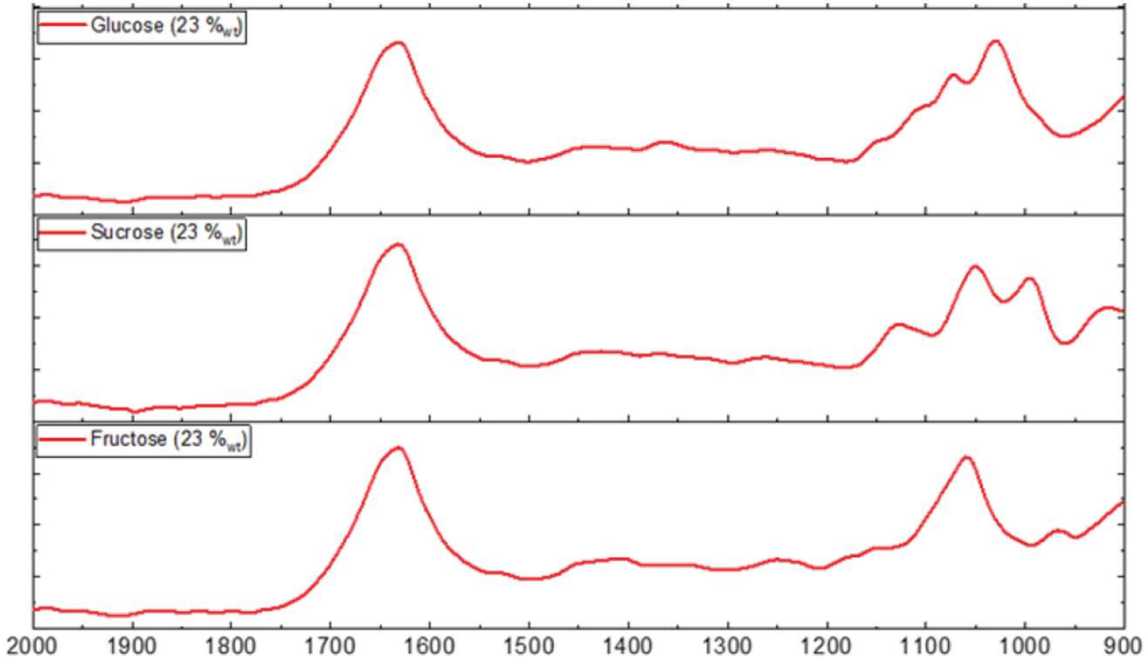
How it works …



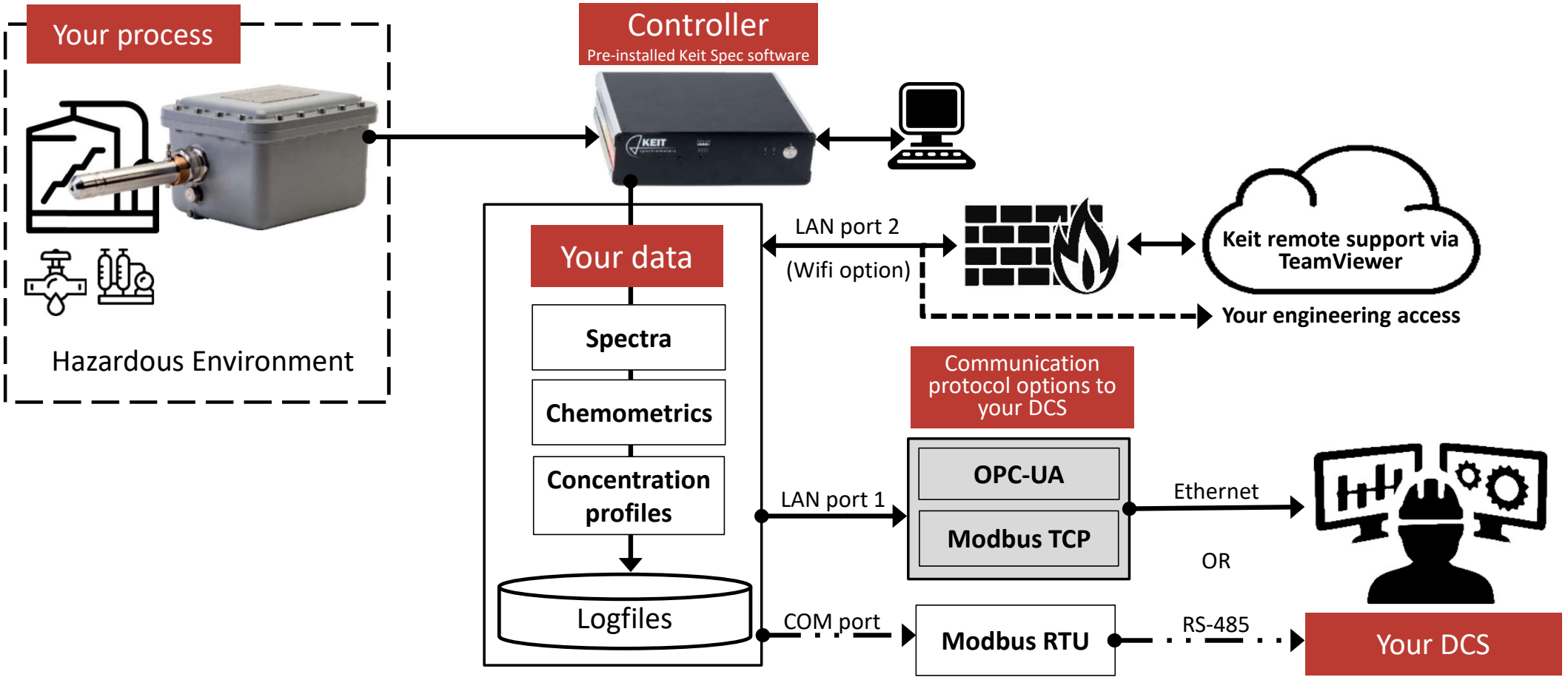
IRmadilloDiamond™

Example mid-infrared spectra



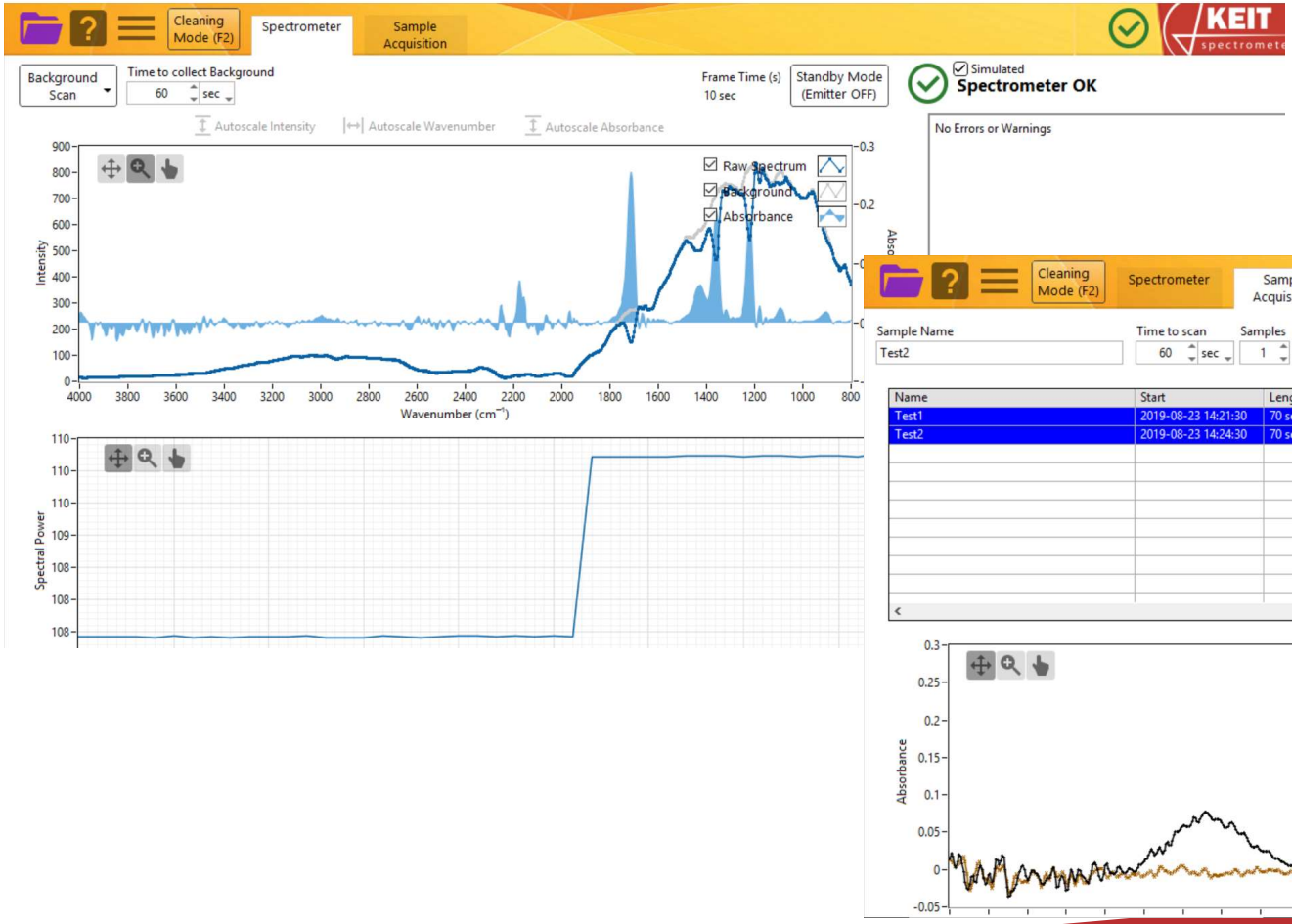


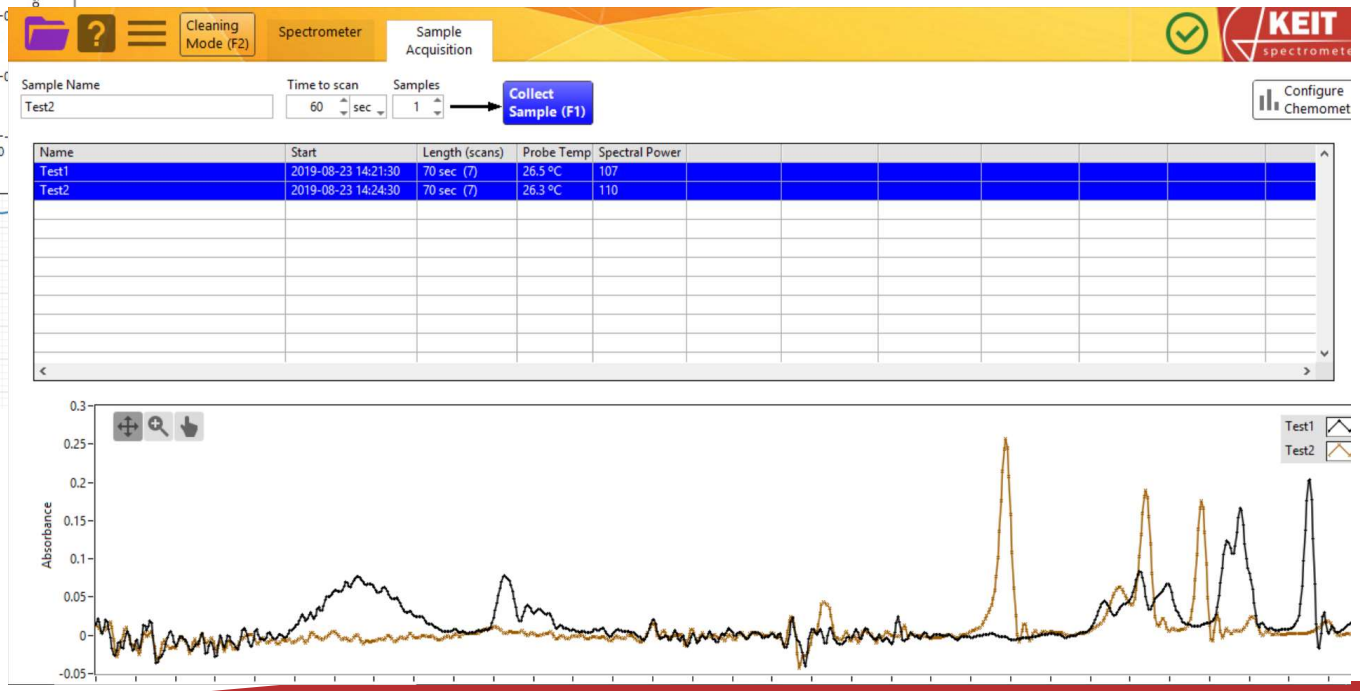
Data flow & systems communication



Field installation options

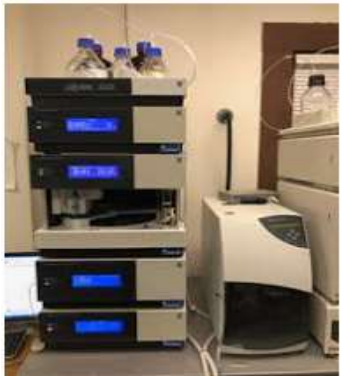
Keit Spec software





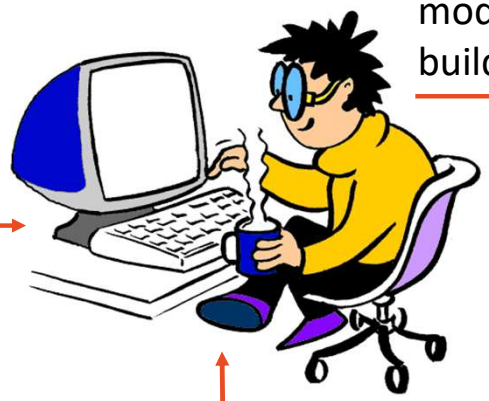
Building a calibration model

Process samples

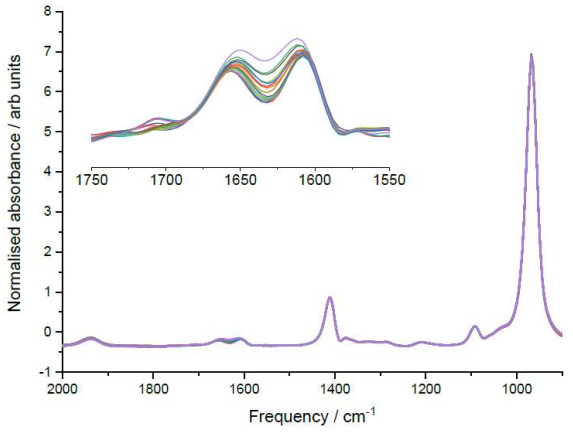


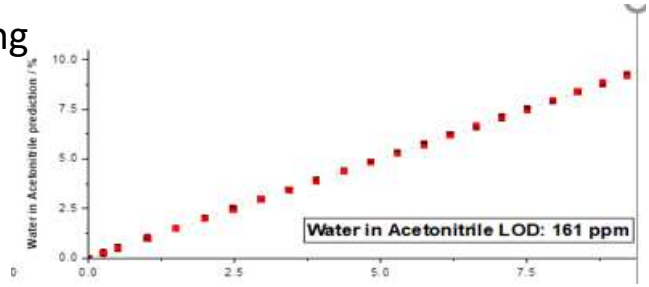
Reference concentrations by HPLC or similar

Spectra obtained from IRmadillo



Calibration model building

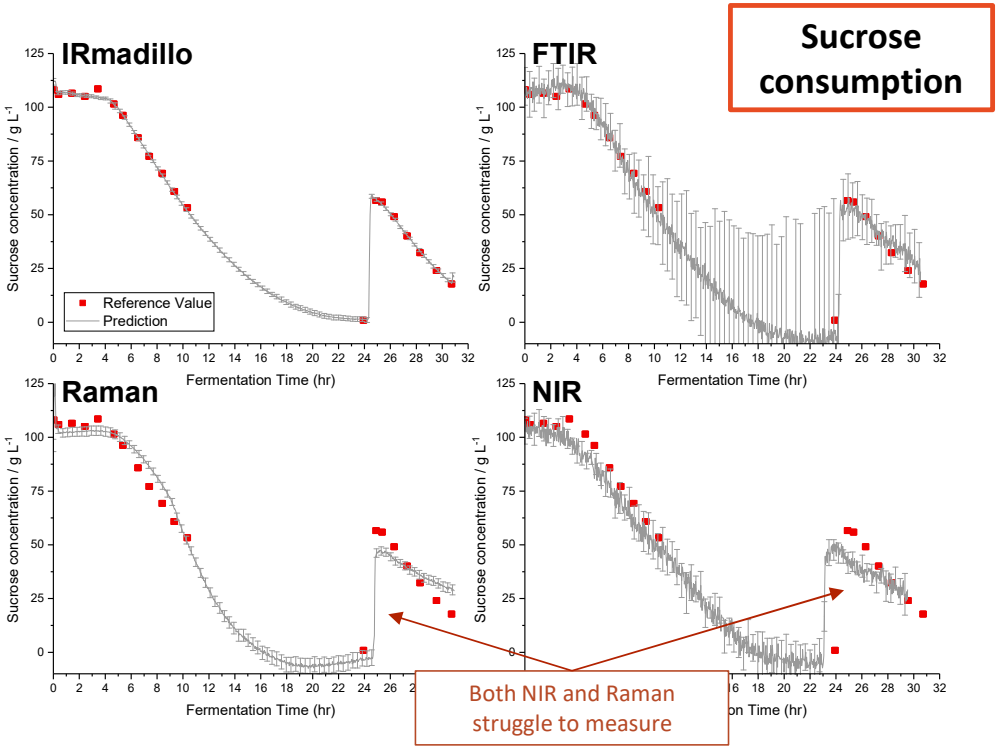


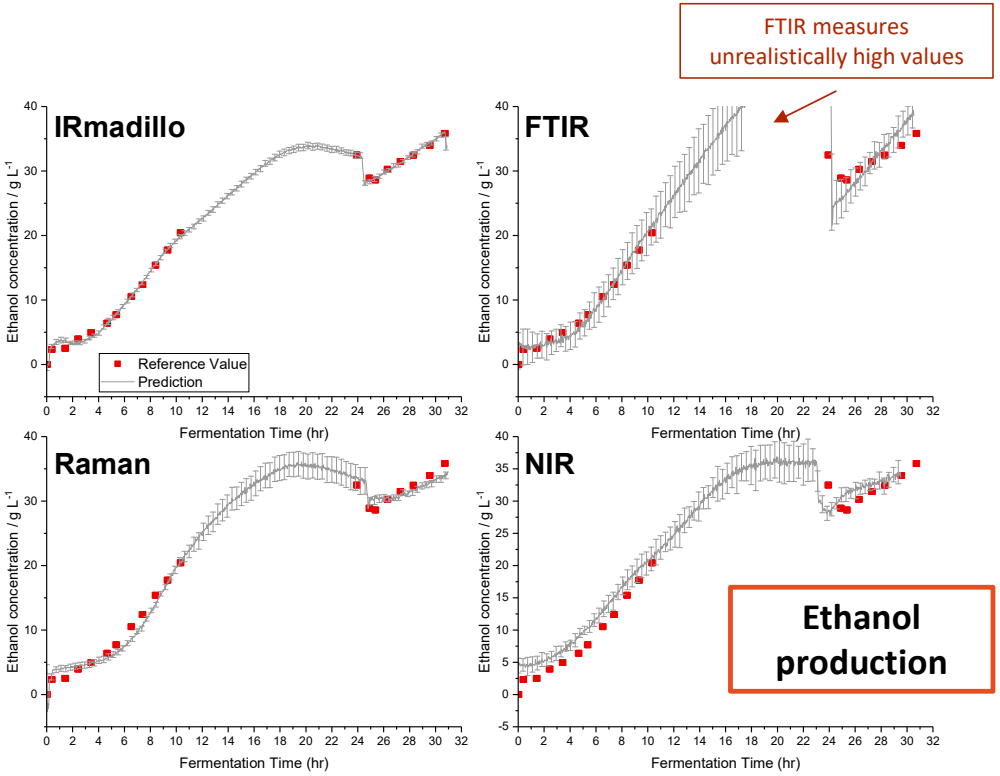


Model exported to IRmadillo Controller

- CAMO Unscrambler
- Eigenvector Solo

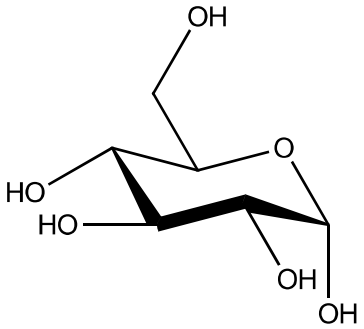
Technology comparison



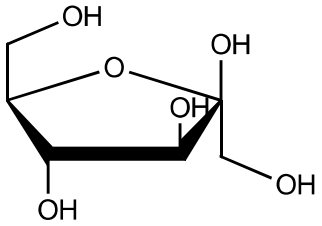


Conventional FTIR really struggles over periods with no reference showing the instability of the instrument – even over only 12 hrs.
Raman and NIR both fail with high solid loadings, which is also seen when bubbles are present.
Only the IRmadillo successfully performs throughout the entire process

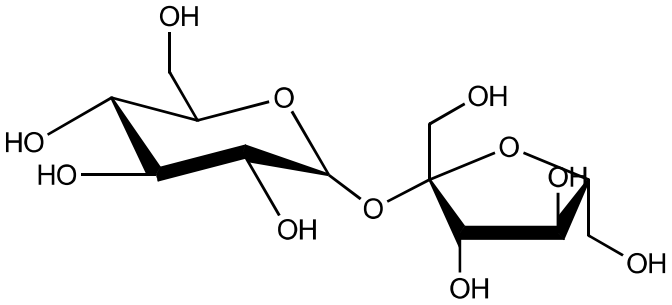
Why does NIR struggle?



Glucose/dextrose



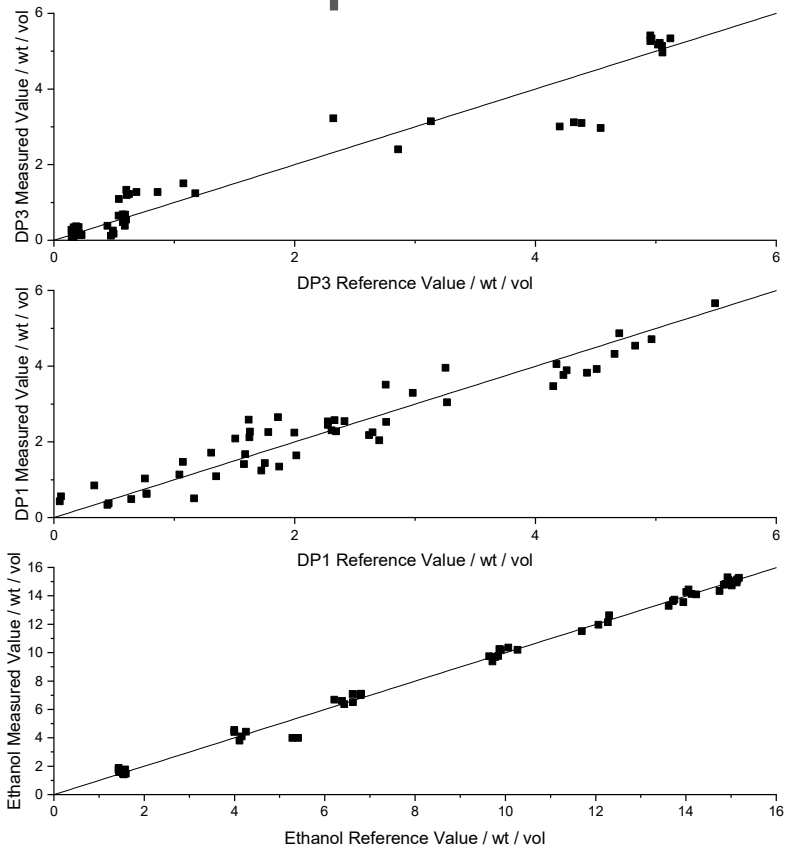
Fructose

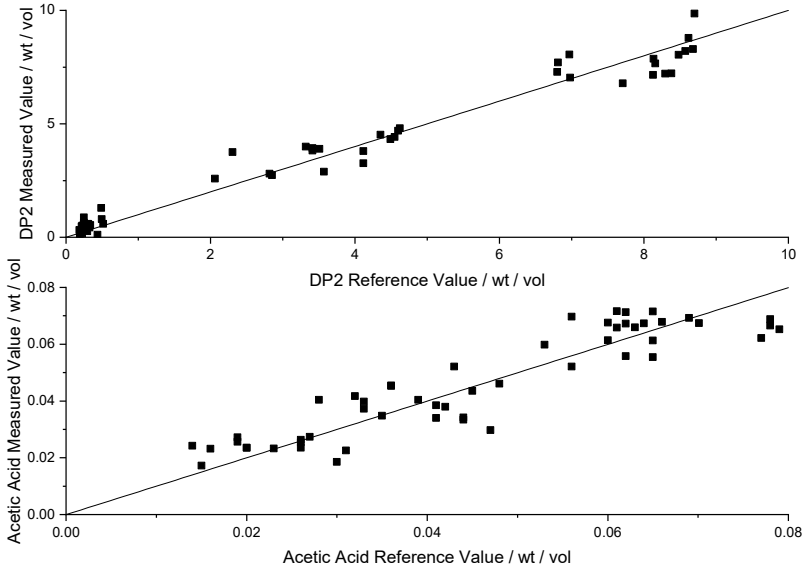


Sucrose

- All sugars are made up of C-C bonds, C-O bonds, C-H bonds and O-H bonds.
- The arrangement is fairly similar in all cases.
- NIR looks at combinations and overtones of fundamental vibrations, so struggles to detect these differences
- Raman and FTIR both look at fundamental vibrations, so can detect these changes and hence discriminate between similar molecules, even isomers

Example: Bioethanol fermenation

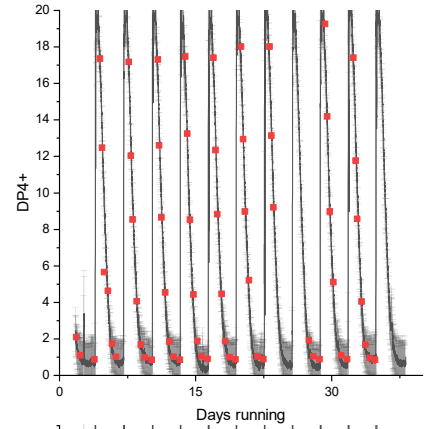


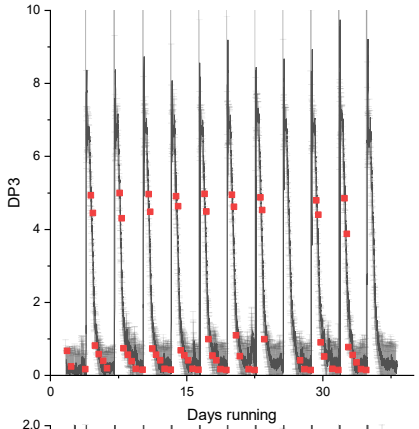


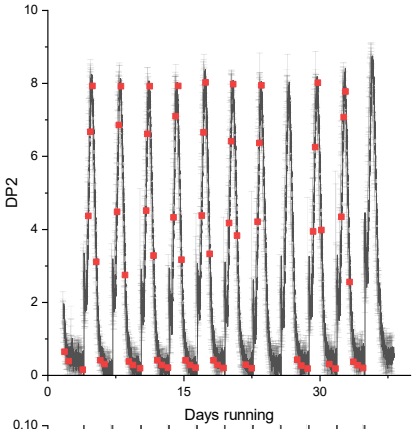
Calibration plots for the PLS models used for calibration. The results show excellent measurement ability for ethanol, good measurement for DP2 and DP1 and reasonable measurement for DP3 and acetic acid (see later for potential improvements).

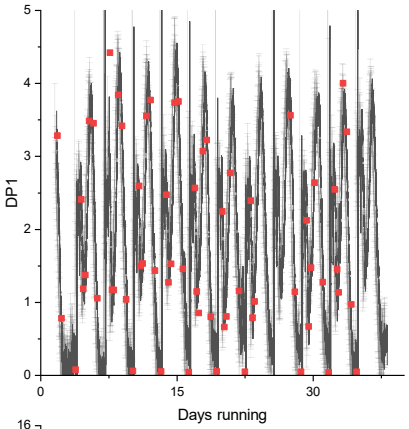
Average error values:
DP3 – 0.51
DP2 – 0.68
DP1 – 0.55
Acetic Acid – 0.009
Ethanol – 0.4

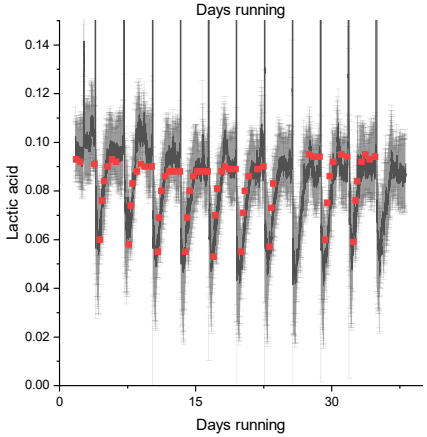
Bioethanol: real-time production data

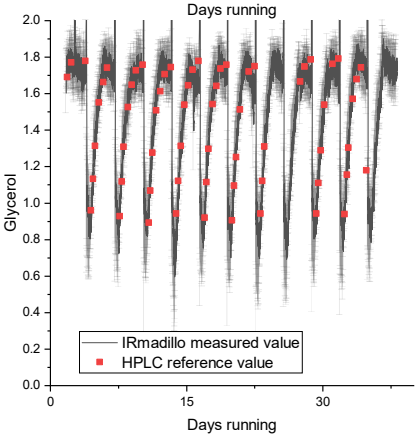


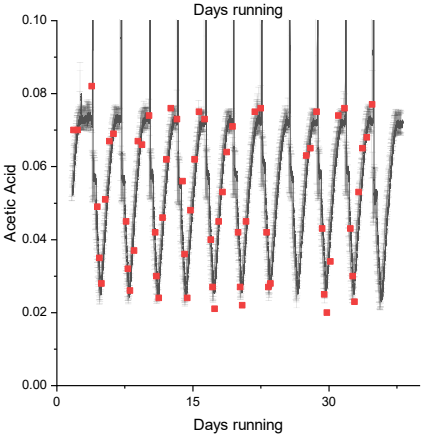


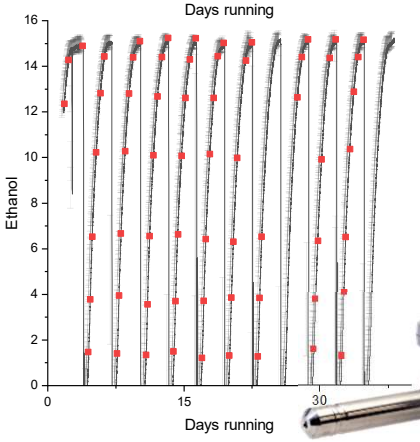








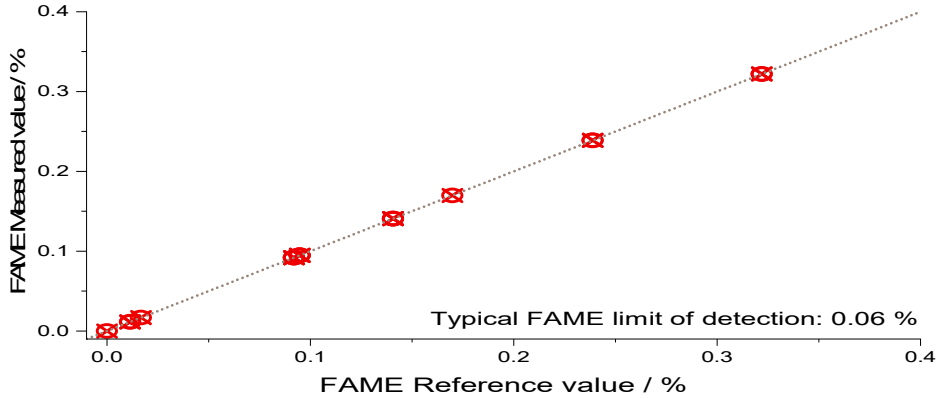


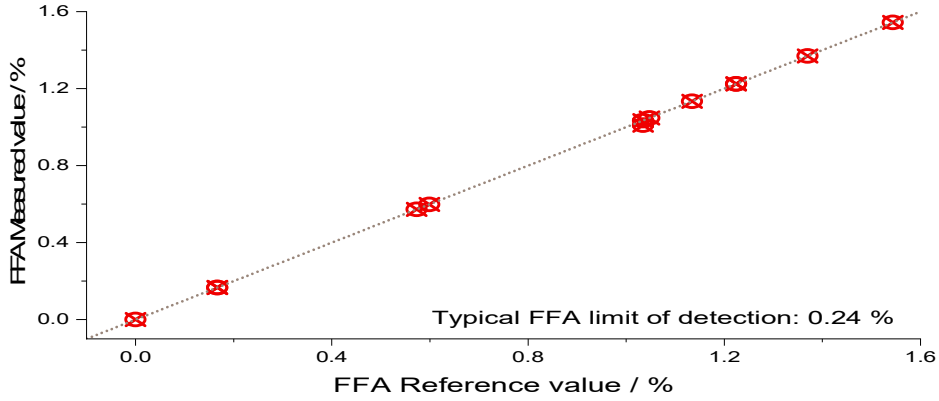


Example: Edible oil refining

Typical Measurements

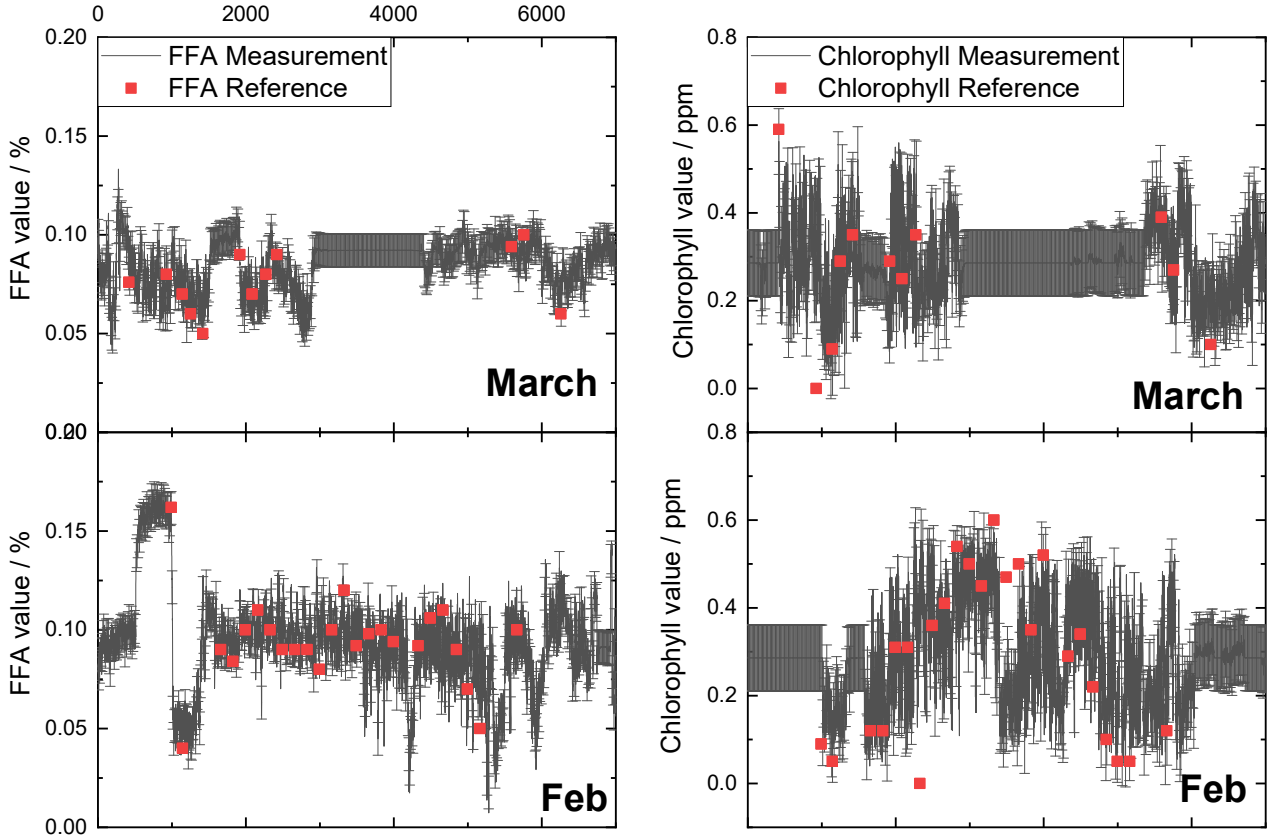
- Water
- Free glycerol
- Monoglycerides
- Diglycerides
- Fatty acids
- Fatty acid methyl esters (FAME)
- Soaps (as fatty acid sodium salts)
- Phosphorus (as phospholipids – differentiating between hydratable and non-hydratable)
- Thermal degradation (oxidation)





FAME and FFA calibrations

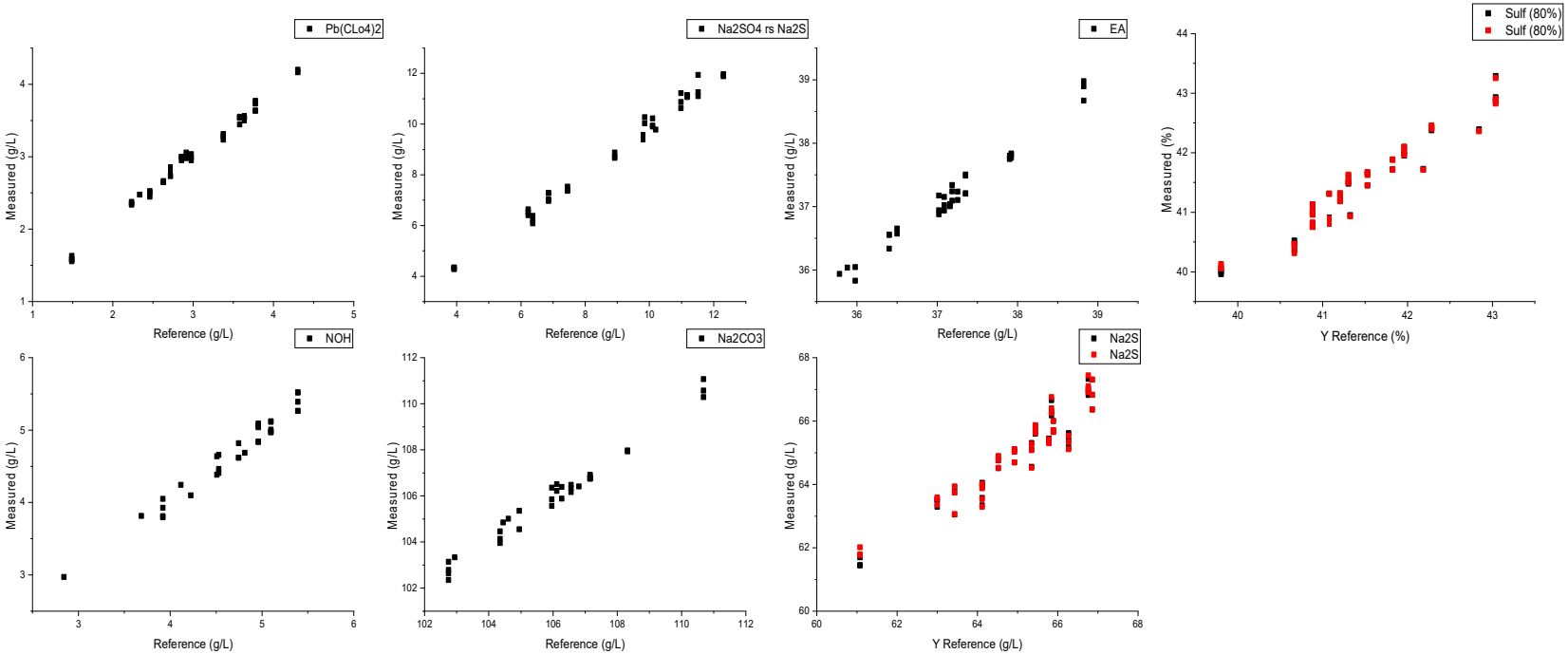
Edible oil refining: real-time production data



Results show ability to monitor process for timings when we have reference values. We can spot deviation from desired process control, as is shown in FFA for February measurements.

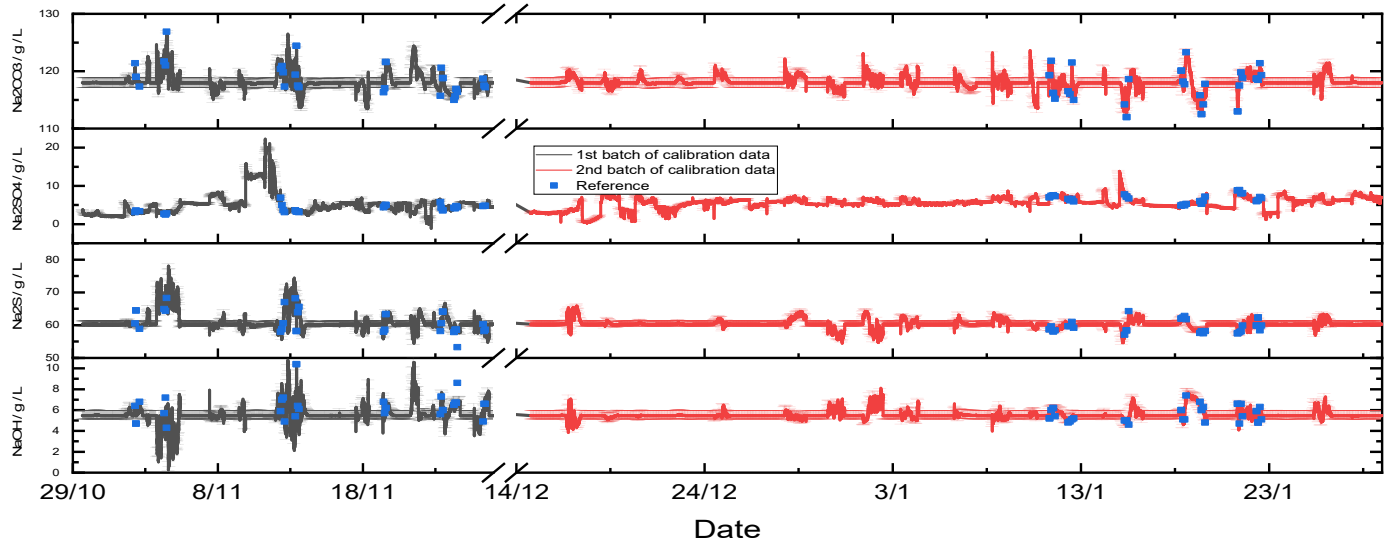
Customer was happy with these results, and decided to move the instrument further upstream to calibrate for feed-forward control of alkali dosing.

Example: Pulp Mill - Green Liquor



Compound	LoD
PbClO4	0.12 g/L
NaOH	0.3 g/L
Na2SO4	0.4 g/L
Na2CO3	0.8 g/l
EA	0.4 g/L
Na2S	0.5 g/L
Sulfur 80%	0.22%

Green liquor: real-time production data



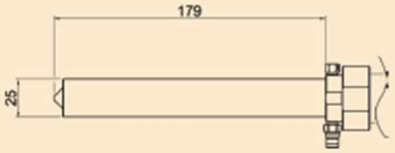
Compound	LoD
NaOH	0.4 g/L
Na2SO4	0.2 g/L
Na2CO3	0.8 g/l
Na2S	0.8 g/L

Probe options

IRmadillo25

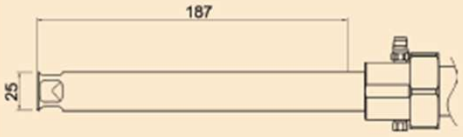
Infrared-glass ATR probe tip with option for high-temperature compatibility

Ex Certified for North America



- Probe diameter: 25 mm
- Length (insertable): 179 mm
- ATR material: Infrared transparent glass
- Probe material: Hastelloy or stainless steel
- Spectral range: 800 to 3800 cm^{-1}
- Temperature (ambient): -15°C to +40°C
- Temperature (analyte): 0°C to +80°C
- Pressure (analyte): 0 to 19 barg
- pH range: 0 to 9
- Ex certificates: marked C1D2

Ex Certified: ATEX, IECEx High-Temperature Probe

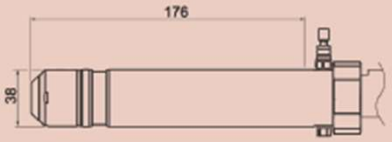


- Probe diameter: 25 mm
- Length (insertable): 187 mm
- ATR material: Infrared transparent glass
- Probe material: Hastelloy
- Spectral range: 800 to 3800 cm^{-1}
- Temperature (ambient): -15°C to +60°C
- Temperature (analyte): -15°C to +220°C
- Pressure (analyte): 0.3 to 42.37 bara
- pH range: 0 to 9
- Ex certificates: ATEX, IECEx

IRmadilloDiamond

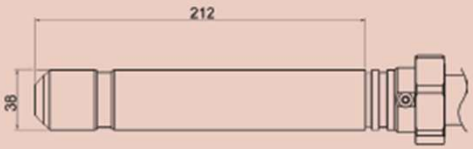
Diamond ATR probe tip for wide range of chemical compatibility

Ex Certified: ATEX, IECEx



- Probe diameter: 38 mm
- Length (insertable): 176 mm
- ATR material: Diamond
- Probe material: Hastelloy
- Spectral range: 650 to 1900 cm^{-1} & 2700 to 3800 cm^{-1}
- Temperature (ambient): -20°C to +60°C
- Temperature (analyte): -20°C to +80°C
- Pressure (analyte): 0 to 20 barg
- pH range: 0 to 14
- Ex certificates: ATEX, IECEx

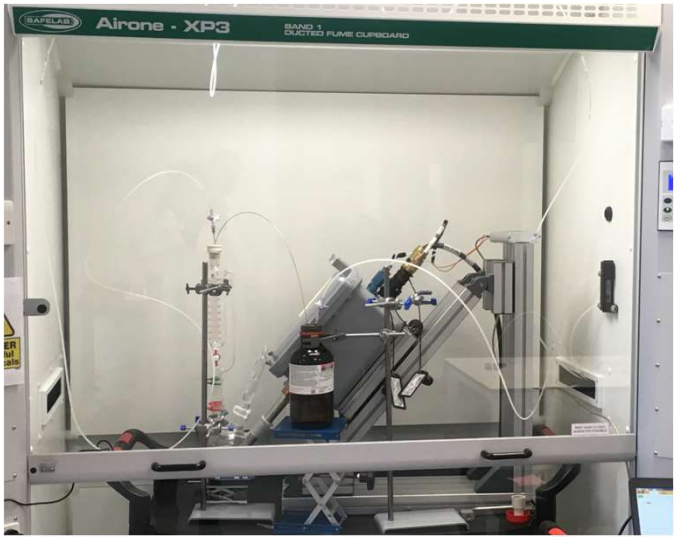
CE Marked



- Probe diameter: 38 mm
- Length (insertable): 212 mm
- ATR material: Diamond
- Probe material: Hastelloy
- Spectral range: 648 to 1800 cm^{-1} & 2700 to 3800 cm^{-1}
- Temperature (ambient): -20°C to +60°C
- Temperature (analyte): -20°C to +130°C
- Pressure (analyte): 0 to 20 barg
- pH range: 0 to 14
- Clean in place (CIP): Yes
- Sterilise in place (SIP): Yes

We continue to develop lab. accessories to help customers test the device and build models …

Sample cell – 5ml



Lab stand

Glass Reaction Vessel

Where has IRmadillo been deployed?

Industry	Location	Measurement/Process
Chemical	Eastern Europe	Silicates
Metals & Minerals	Australia, Germany	Bayer Process Chemistry
Biotechnology	Denmark	Fermentation species (x 2)
Pulping	Nordics	Cellulose Hydrolysis
Bioethanol	USA	Sugars, ethanol
Chemical	Switzerland	Crystallisation (x2)
Petrochemical	USA	(not disclosed)
Bioethanol	Eastern Europe	Sugars, ethanol
Edible Oil Refining	UK	Free Fatty Acids, others
Pulp & Paper	Scandinavia	Green Liquor
Petrochemicals	UK	(not disclosed)
Bioethanol	Belgium	Sugars, ethanol

Many more applications in discussion, trial and test….

I’m interested. What’s next?

- Lab trial (for novel chemistry)
- Production trial
- Installation & training
- Calibration services
- Ongoing support

Created for Mars: application on Earth

IRmadillo™

KEIT spectrometers

Thank You

Richard Salliss
Sales Director
richard.salliss@keit.co.uk

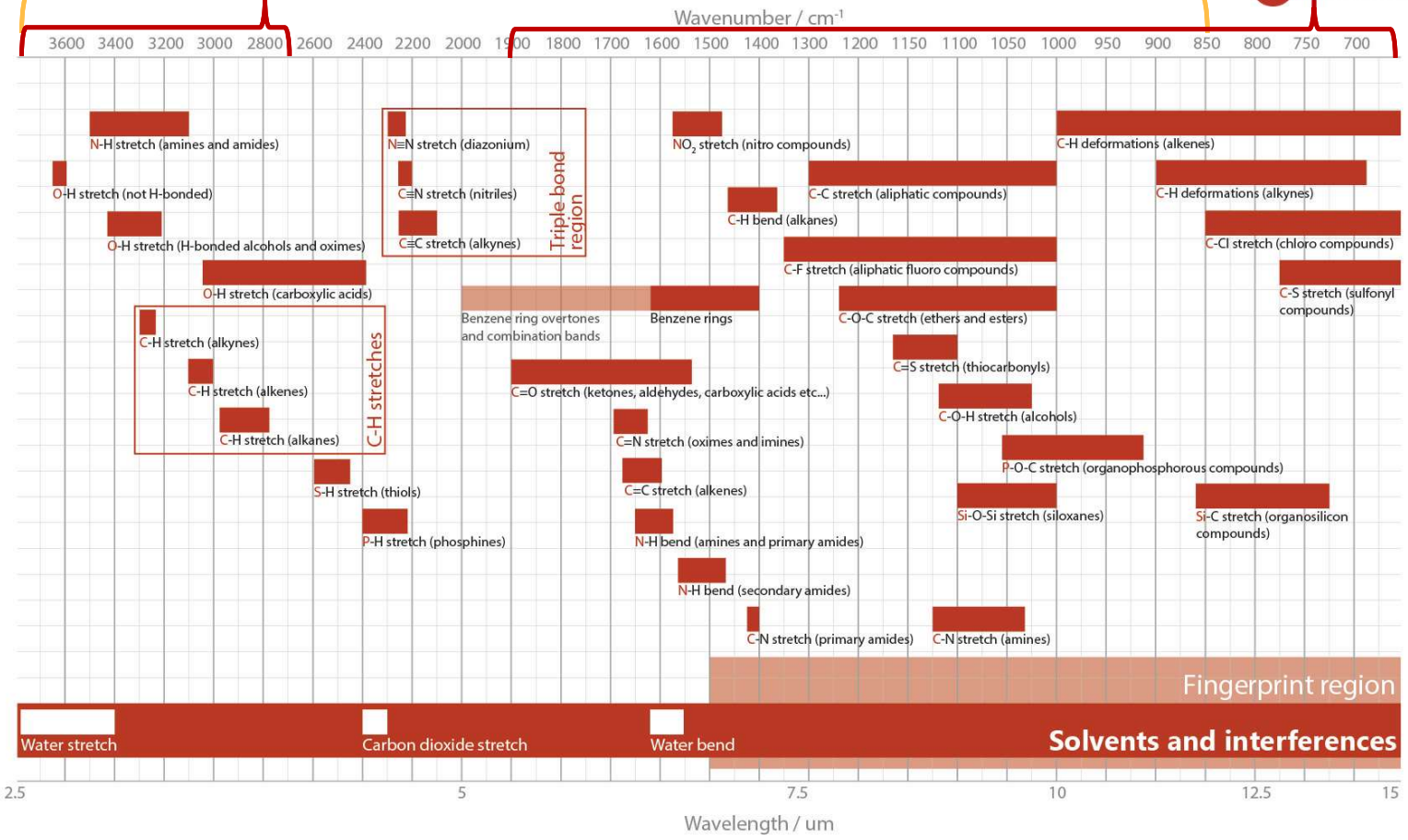
Rugged FTIR for real-time process monitoring of liquids

Back-up slides

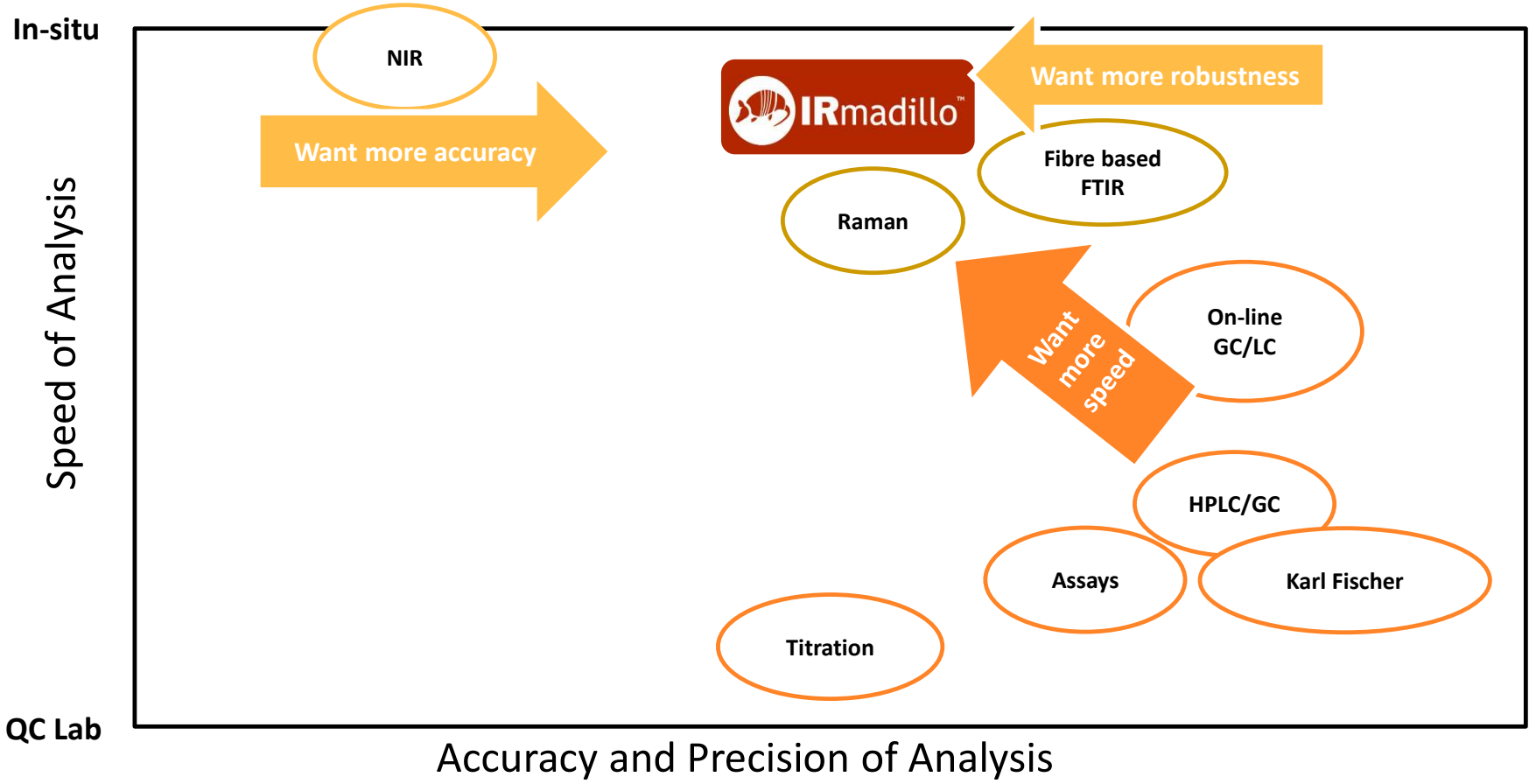
The Power of Mid-Infrared

850–3800 cm^{-1} / 12 – 2.6 µm
IRmadillo25™

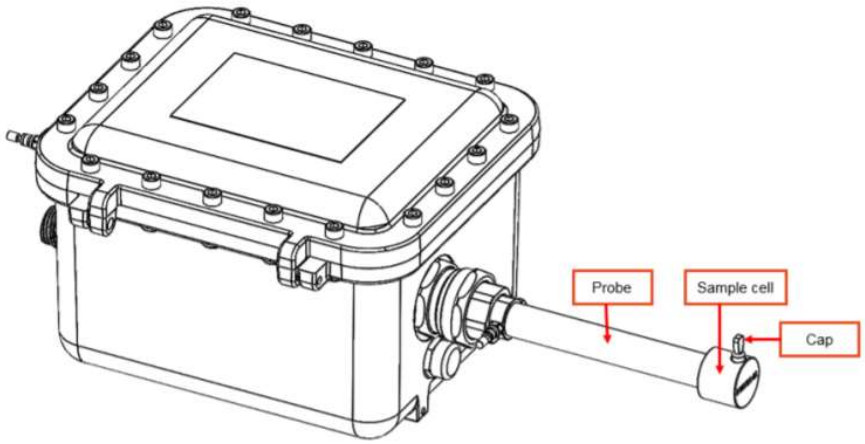
650 – 1900 cm^{-1} & 2700 – 3800 cm^{-1}
15.4 – 5.3 µm & 3.7 – 2.6 µm
IRmadilloDiamond™



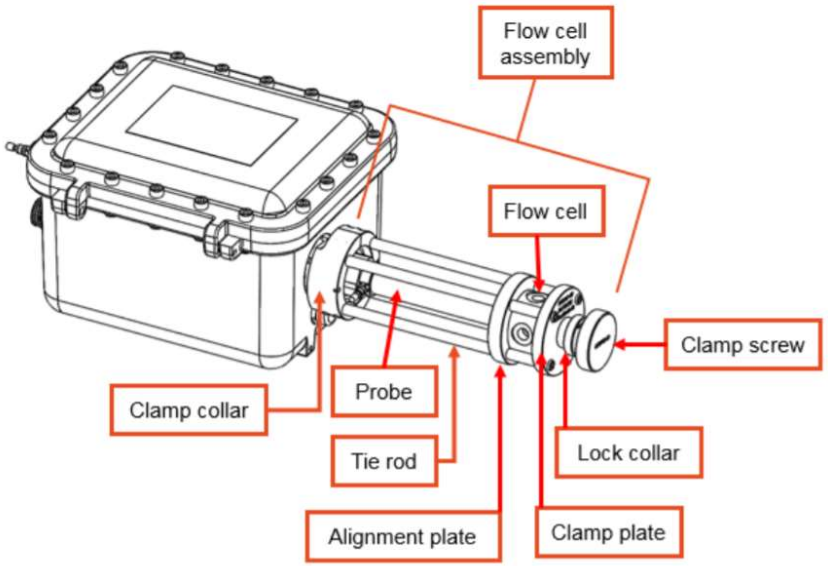
Competitive technologies

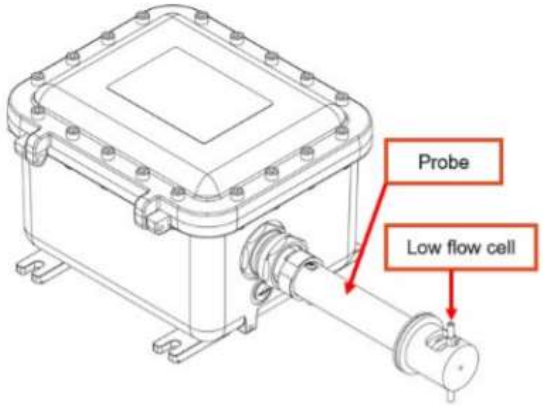


マニュアルサンプリング



フローセル


Flow cell assembly
Flow cell
Clamp screw
Clamp collar
Probe
Tie rod
Lock collar
Alignment plate
Clamp plate


Probe
Low flow cell