Worldwide MIR Spectra Standardization

Bentley IR Cell and Spectra standardization
A new highly effective and simple approach (patented)

By Pierre Broutin
Managing Director/Senior Scientist
Bentley FTMIR Optical Deck

INTERFEROMETER

- Moving Mirror
- Flow Cell (milk)
- Detector
- IR Source

MILK SPECTRUM

- Fourier Transform

ICAR 2016 Congress Manufacturers Showcase, Puerto Varas, Chile
October 27, 2016
Bentley FTS/DairySpec
Milk Components Absorption wavebands
Bentley IR Cell and Spectra standardization

Why?

- Interferometer laser frequency can vary over time ➡ spectrum x axis shift
- Flow cell pathlength can increase over time ➡ spectrum y axis shift

Thus, spectra standardization is very important:

- For optimum calibration transfer between instruments
- For worldwide equivalence of analytical results
- For results/calibration stability (Slope/Bias)
- To reduce calibration development cost (centralized calibrations)
- For implementation of qualitative spectral analysis
Bentley IR Cell and Spectra standardization
A new highly effective and simple approach

Prerequisites to standardize the infrared spectra:

1 - Standardization of spectrum x axis (wavenumbers)
2 - Standardization of spectrum y axis (absorbance)
3 - Standardization of all operating conditions and sample preparation
4 - Removal of any moisture in optical deck that could affect the spectra
1 - Standardization of spectrum x axis with a polystyrene film (internationally recognized NIST standard) to calibrate optimally interferometer laser frequency

Example: Polystyrene Test

- Background Scan Completed
- Polystyrene Scan Completed
- Peak 3082.22 @:3082.18 cm⁻¹
- Peak 3060.14 @:3060.12 cm⁻¹
- Peak 1601.38 @:1601.37 cm⁻¹
- Peak 1583.04 @:1583.24 cm⁻¹
- Peak 1028.42 @:1028.59 cm⁻¹

[4:30 PM Central Daylight Time]  Polystyrene Test:  STARTED

[4:31 PM Central Daylight Time]  Polystyrene Test:  PASSED
1 - Standardization of spectrum x axis with a polystyrene film (internationally recognized NIST standard) to calibrate optimally interferometer laser frequency.
2 - Standardization of spectrum y axis (absorbance) by measuring very accurately and in real time the IR flow cell thickness (patented)
2 - Standardization of spectrum y axis (absorbance) by measuring very accurately and in real time the IR flow cell thickness (patented)

- The thickness of the sealed cells is one of the basic measurements required for quantitative analysis.

- The cell thickness should be verified from time to time frequency depending on the type of products analyzed, since it may change due to a gradual erosion of the cell internal surfaces (1% per month, up to 12% per year).

- The Beer-lambert law $A=\varepsilon bc$ establishes the linear relationship between absorbance and path length.
2 - Standardization of spectrum y axis (absorbance) by measuring very accurately and in real time the IR flow cell thickness (patented)

Summary Report

- Repeat 1 = 36.08468
- Repeat 2 = 36.08663
- Repeat 3 = 36.08227
- Repeat 4 = 36.08309
- Repeat 5 = 36.08712
- Repeat 6 = 36.08846
- Repeat 7 = 36.08889
- Repeat 8 = 36.09445
- Repeat 9 = 36.08202
- Repeat 10 = 36.09503

Average Cell Space calculated = 36.0873um
Standard deviation = 0.0046um
2 - Standardization of spectrum y axis (absorbance) by measuring very accurately and in real time the IR flow cell thickness (patented)
Why this way?

- Laser calibrated with NIST traceable films (polystyrene)
- Does not require any highly accurate liquid mixtures
- No consumable to package, store, maintain or process
- Automatic execution, no user dependent operations
- Can be performed as often as necessary
- A very reliable, easy to implement, and cost effective solution
3 - Standardization of all operating conditions and sample preparation
3 - Standardization of all operating conditions and sample preparation

Full standardization of all operating conditions:

- Samples mixing/shaking
- Sample temperature
- Sample heating time

Samples handling independent of operators and highly reproducible
4 - Removal of any moisture in optical deck that interfere with spectra

- The optical deck sealed to remove all potential interference
- The cell isolated outside the optical deck in a small elbow containing a dessicant
- The optical deck moisture level is also monitored in real time.
CECALAIT Calibration samples analyzed over 7 months – single calibration, no adjustment required
CECALAIT Milk BHB Calibration samples analyzed over 12 months – single calibration, no adjustment required

ICAR 2016 Congress Manufacturers Showcase, Puerto Varas, Chile
October 27, 2016
Bentley IR Cell and Spectra standardization
A new highly effective and simple approach (patented)

- Standardization of spectrum x axis with polystyrene NIST international standard
- Standardization of spectrum y axis (absorbance) - patented
- Standardization of all operating conditions and sample preparation
- Removal of any moisture in optical deck that could affect the spectra

Our unique approach leads to real time, optimum and cost effective:

- Spectra standardization
- Calibrations transfer, accuracy and stability
Thank you for your attention!

pbroutin@bentleyinstruments.com
www.bentleyinstruments.com